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Zinc, Cadmium and Mercury 1,1-Dithiolates

A Thesis Submitted for the Degree of

Doctor of Philosophy

by

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ADDENDUM

Page 27: The structures reported in this thesis were solved by utilising the PATTY option in the DIRDIF package.

Table 5.III.1: The choice of space group was determined by the successful refinement.

The compound in Table 6.III.1 is $[Cd(S_2CN(CH_2CH(CH_3)_2)_2)_2]$.

Appendices: The temperatures at which data were collected are in K.

Figure 1.6: The compound is $[Cd(S_2COCH_2CH_2OMe)_2]$.

N.B. Values reported in Tables of bond lengths and angles in the text were calculated using reflections that satisfied the $I \geq 3.0\sigma(I)$ criterion of observability.

DECLARATION

This thesis contains no material which has been accepted for any other degree or diploma in any university or other tertiary institution and, to the best of my knowledge and belief contains no material published or written by another person, except where due reference is made.

I consent to this thesis being made available for photocopying and loan.

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PUBLICATIONS

(1) Cox, M. J.; Tiekink, E. R. T.: Crystal and molecular structures of bis(*O*-*n*-propyldithiocarbonato)nickel(II) and bis(*O*-*i*-propyldithiocarbonato)nickel(II). Z. Kristallogr. **211** (1996) 111 – 113.

(2) Cox, M. J.; Tiekink, E. R. T.: The crystal and molecular structures of $[Te(S_2COR)_2]$, R=nPr and $CH_2CH_2CMe_3$. Z. Kristallogr **212** (1997) 811 – 814.

(3) Cox, M. J.; Tiekink, E. R. T.: Crystal structure of bis(*O*-n-butyldithiocarbonato)nickel(II), $C_{10}H_{18}O_2NiS_4$. Z. Kristallogr. **212** (1997) 350.

(4) Cox, M. J.; Tiekink, E. R. T.: Crystal structure of bis(*O*-3,3-dimethylbutyldithiocarbonato)nickel(II), $[Ni(S_2COC_6H_{13})_2]$. Z. Kristallogr. **211** (1996) 575.

(5) Cox, M. J.; Tiekink, E. R. T.: Crystal structure of bis(*O*-3-methylbutyldithiocarbonato)nickel(II), $C_{12}H_{22}NiO_2S_4$. Z. Kristallogr. **211** (1996) 750.

(6) Cox, M. J.; Tiekink, E. R. T.: Structural variations in the mercury(II) bis(1,1-dithiolate)s: the crystal and molecular structure of $[Hg(S_2CNMe_2)_2]$. Z. Kristallogr. **212** (1997) 542 - 544.

(7) Cox, M. J.; Tiekink, E. R. T.: The crystal and molecular structures of some nickel(II) bis(O-alkyldithiocarbonate)s and nickel(II) bis(N,N-dialkyldithiocarbamate)s: an evaluation of the coordination potential of 1,1-dithiolate ligands in their nickel(II) complexes. Z. Kristallogr. in press.

(8) Cox, M. J.; Tiekink, E. R. T.: Structural systematics of tellurium(II) bis(O-alkyldithiocarbonate) and tellurium(II) bis(N,N-dialkyldithiocarbamate) compounds. Z. Kristallogr. submitted.

(9) Cox, M. J.; Tiekink, E. R. T.: The diverse coordination patterns in the structures of zinc, cadmium and mercury bis (1,1-dithiolates). Rev. Inorg. Chem. **17** (1997) 1 - 23.

(10) Cox, M. J.; Tiekink, E. R. T.: Structural characteristics of cadmium(II) bis(*N*,*N*-dialkyldithiocarbamate) and cadmium(II) bis(*O*-alkyldithiocarbonate) compounds. Z Kristallogr. accepted.

(11) Cox, M. J.; Tiekink, E. R. T.: Structural diversity in mercury(II) bis(O-alkyldithiocarbonate) compounds. Z. Kristallogr. accepted.

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(13) Cox, M. J.; Tiekink, E. R. T.: Structural features of zinc(II) bis(O-alkyldithiocarbonate) and zinc(II) bis(N,N-dialkyldithiocarbamate) compounds. Z. Kristallogr. in press.

ABSTRACT

A fascinating aspect of the solid state chemistry of the main group element 1,1-dithiolates is their structural diversity. It is known that a small change in the organic substituent of the 1,1-dithiolate ligand may result in a significant change in the coordination geometry about the metal atom. The reasons for these variations have yet to be delineated. Thus, a systematic investigation of the coordination ability of 1,1-dithiolate ligands, namely xanthates and dithiocarbamates, has been undertaken.

The electronic characteristics of the xanthate and dithiocarbamate ligands were examined by the determination of the solid state structures of the ligands coordinated to the transition metal element, nickel. This revealed that the coordinating ability of both the xanthates and dithiocarbamates are independent of the nature of the organic substituent on the ligand. This was further substantiated by an analogous study with the main group element, tellurium, where again the coordination ability of the ligands was shown to be independent of the organic substituent of the ligand.

Computational work on the free xanthate and dithiocarbamate ligands was conducted to gain an understanding of the electronic distribution over the ligands and the effect of the various organic substituents on this electronic distribution. The results revealed that a relatively large excess of electron density resides on the respective S_2C moieties and that the substitution of a particular organic substituent has little overall effect.

The determination of a number of solid state structures of the zinc, cadmium and mercury 1,1-dithiolates has allowed for a systematic study of their structural chemistry. This study revealed that a large number of structural motifs exist for these systems. The systematic analysis has enabled a rationalisation of the appearance of the different motifs for the first time. It was found that the ultimate structure adopted in the solid state is determined by a balance between the need to satisfy the valency of the central element (intramolecular bonding) and the requirements for efficient crystal packing (intermolecular interactions). This indicates that the stability of the molecular structure is influenced significantly by intermolecular interactions.

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Chapter 1

INTRODUCTION

The 1,1-dithiolates are a class of uninegative ligands that includes, dithiocarbonates [$^{-}S_2COR$, commonly known as xanthates; Zeise coined this term in 1815 after the bright yellow colour of their lead complexes (Winter, 1980)], dithiocarbamates [$^{-}S_2CNR_2$], dithiophosphates [$^{-}S_2P(OR)_2$] and dithiophosphinates [$^{-}S_2PR_2$]. The coordination chemistry of metal complexes of these ligands represents a field of continuous interest owing to their various applications (Winter, 1980; Tiekink, Winter, 1992; Haiduc, Sowerby, Lu, 1995; Eisenberg, 1970; Coucouvanis, 1979; Coucouvanis, 1970).

Metal 1,1-dithiolates have been used as agents in metallurgy (flotation), as accelerators in rubber vulcanisation, as fungicides and pesticides in agricultural applications, and as high pressure lubricants. More recently, 1,1-dithiolates have been used for the removal of heavy metals and organometallic cations from mammalian systems, and their anti-microbial activity has been explored. In addition, the zinc, cadmium and mercury dithiolates find uses in contemporary applications. For instance, zinc dithiolates are used in metal organic chemical vapour deposition (MOCVD), dithiolates can be used to remove cadmium from contaminated soils, and similarly, dithiolates are used to remove mercury and mercury vapour from waste products.

In order to fully understand and rationalise the chemical and physical properties of metal dithiolates, a thorough knowledge of their chemical structures is required. A pivotal technique in structure determination is single crystal X-ray diffraction. This technique stands alone in providing an unambiguous geometry, as well as precise interatomic parameters and details of supramolecular interactions. While the question remains whether the solid state structures thus determined have relevance to their solution state structures, another question posed is related to the influence that crystal packing effects has on molecular geometry. Thus, recent reviews of main group element

structures have revealed that very different structural motifs may be observed even though only small changes in chemistry have been made (Tiekink, Winter, 1992; Tiekink, 1991, 1992, 1994, 1995). In consideration of this question, and given the inherent interest in zinc, cadmium and mercury 1,1-dithiolate compounds, it is timely to survey the structural chemistry of these compounds.

In this introduction, the structures of zinc, cadmium and mercury binary 1,1-dithiolate complexes have been categorised. This survey reveals a rich variety of coordination patterns including monomeric, dimeric and polymeric structures, which often arise as a result of a diverse range of supramolecular M···S interactions. Of the crystal structures available, there are at least twelve distinct structural motifs in existence. This diversity can occur as a result of both the ability of the dithiolate ligands to exhibit several different modes of coordination, involving one or both sulfur atoms, and the propensity for the zinc, cadmium and mercury to have different coordination numbers. Of particular interest is the observation that chemically similar ligands (*i.e.* different R group/s) can produce dramatic changes in the structures, including coordination numbers and geometries. This occurs despite the fact that normally the organic substituent is remote from the coordination centre.

This introduction will summarise the structural characteristics of the zinc, cadmium and mercury 1,1-dithiolates by describing each element in turn, and considering the dithiolates in the following order i) xanthates, ii) dithiocarbamates, iii) dithiophosphates, and iv) dithiophosphinates. Diagrams have been drawn with the ORTEP program using arbitrary thermal ellipsoids (Johnson, 1976). The aim of the introduction is to give an overview of the structural motifs that may be adopted for these compounds in the solid state and readers are referred to the original literature for particular details.

1.1 ZINC BIS 1,1-DITHIOLATES

Molecular structures, as determined by X-ray diffraction techniques, have been reported for a relatively large number of zinc bis(1,1-dithiolates). As will be demonstrated, even for the same ligand type (*i.e.* xanthate, dithiocarbamate, *etc.*), changes in the nature of the organic substituent can result in a major modification to the structure adopted in the solid state. In almost all of the zinc(II) derivatives, the coordination around the zinc atom can be considered as distorted tetrahedral with four covalent Zn-S bonds. However, if secondary Zn···S interactions, *i.e.* supramolecular associations, are considered, the coordination polyhedron may be thought as being expanded to a distorted trigonal bipyramid or tetragonal prism.

1.1.1 *Xanthate structures*

A distorted tetrahedral geometry exists around the zinc(II) atom in each of $[Zn(S_2COEt)_2]$ and $[Zn(S_2COiPr)_2]$, however, the two structures adopt different coordination patterns.

The structure of $[Zn(S_2COiPr)_2]$ (Ito, 1972), illustrated in Fig. 1.1, comprises isolated centrosymmetric sixteen-membered rings. The structure can be thought of as been constructed about a square array of zinc atoms, with each edge being bridged by a bidentate isopropylxanthate ligand. The remaining positions in the distorted tetrahedral geometry about each zinc atom are occupied by two sulfur atoms derived from a chelating ligand. This structure will hereby be labelled as structural motif A. The bridging xanthate ligands form slightly asymmetric Zn-S distances, *i.e.* the Zn(1)-S(1) and Zn(2)-S(6)' distances of 2.343(8) Å and 2.342(8) Å, respectively are longer than the Zn(1)-S(5) and Zn(2)-S(2) distances of 2.311(7) Å and 2.307(8) Å, respectively. Whereas the S(7), S(8) xanthate chelates with equivalent Zn-S distances (2.370(8) Å and 2.357(7) Å), there is some asymmetry in the Zn-S(3) and Zn-S(4) distances of 2.351(7) Å and 2.408(7) Å, respectively. Nevertheless, the range of Zn-S distances is relatively small at

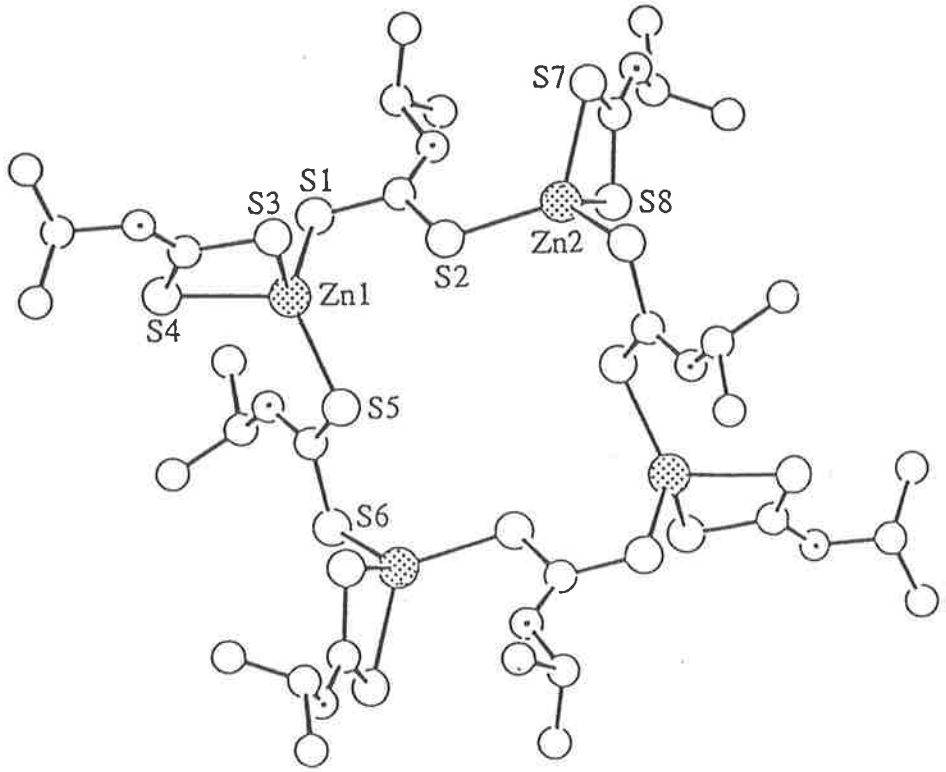


Fig. 1.1 Structural motif A, as represented by $[Zn(S_2COiPr)_2]$

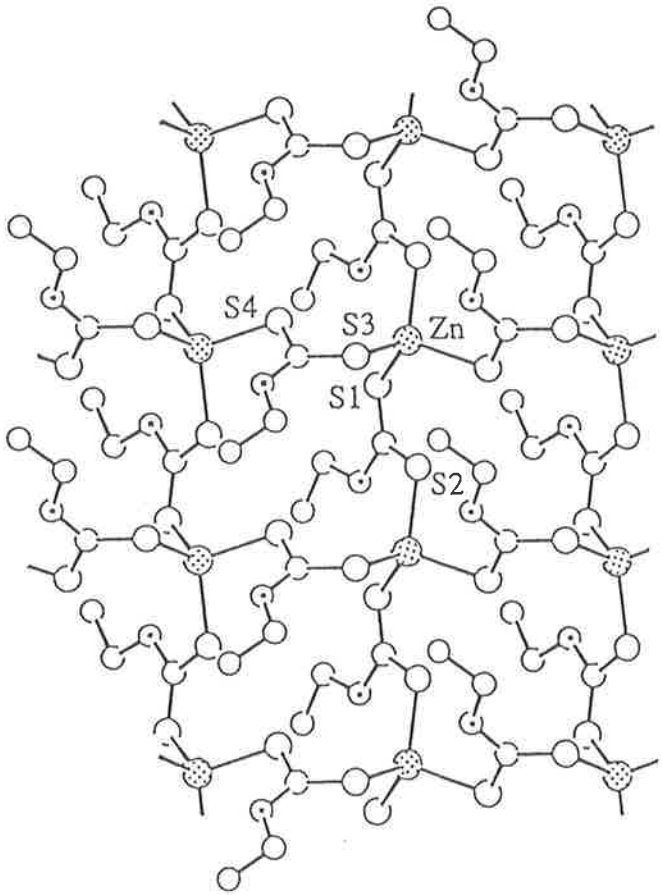


Fig. 1.2 Structural motif B, as represented by $[Zn(S_2COEt)_2]$

2.307(8) Å to 2.408(7) Å. The range of tetrahedral angles is wide owing to the restricted bite distance of the xanthate ligand (*i.e.* 76.7(2)° and 77.1(2)°), if one excludes these angles the range then becomes 106.6(3)° to 122.1(2)°.

The simple substitution of the isopropyl groups in $[Zn(S_2COiPr)_2]$ for ethyl groups, leading to $[Zn(S_2COEt)_2]$, results in a major structural change, *i.e.* structural motif B (Ikeda, Hagihara, 1966). The basic sixteen-membered ring framework as found in $[Zn(S_2COiPr)_2]$ is retained in the structure of $[Zn(S_2COEt)_2]$. The difference between the structures arises in that the chelating xanthate ligands of $[Zn(S_2COiPr)_2]$ are now bridging, so that the $[Zn(S_2COEt)]_4$ tetramers are interconnected to form a two-dimensional sheet, as shown in Fig. 1.2. The Zn-S bond lengths are equal within experimental error and have an average value of 2.35(1) Å. Relatively small distortions from the ideal tetrahedral geometry are observed with the S-Zn-S bond angles ranging from 102.6(3)° to 115.2(3)°.

1.1.2 *Dithiocarbamate structures*

By contrast to the tetrameric and polymeric structures found for $Zn(S_2COR)_2$, the $Zn(S_2CNR_2)_2$ structures are dimeric and fall into two distinct classes. The first (structural type C), shown in Fig. 1.3, is found for $[Zn(S_2CNMe_2)_2]$ (Klug, 1966) and features two zinc atoms that are bridged by two bidentate dithiocarbamate ligands; the dimer has crystallographic two-fold symmetry. The coordination about each zinc atom is completed by a chelating ligand. Structural type D is very similar to that of C, as exemplified in Fig. 1.4 for $[Zn(S_2CNEt_2)_2]$ (this structure has been the subject of two independent investigations (Bonamico, Mazzone, Vaciago, Zambonelli, 1965; Zvonkova, Khvatkina, Ivanova, 1968); parameters from the first study are cited here). There are a further five structures that adopt this motif and these are invariably centrosymmetric. The major difference between structural types C and D relates to the relative disposition of the bridging dithiocarbamate ligands, *i.e.* in C they lie to the same side of the dimer but for D they lie on opposite sides. The eight-membered ring in structural motif C can be

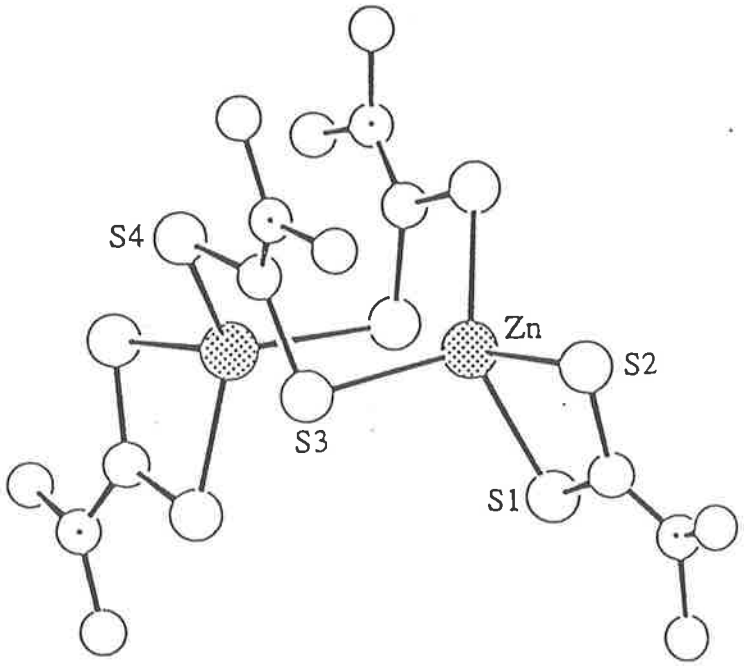


Fig. 1.3 Structural motif C, as represented by $[Zn(S_2CNMe_2)_2]$

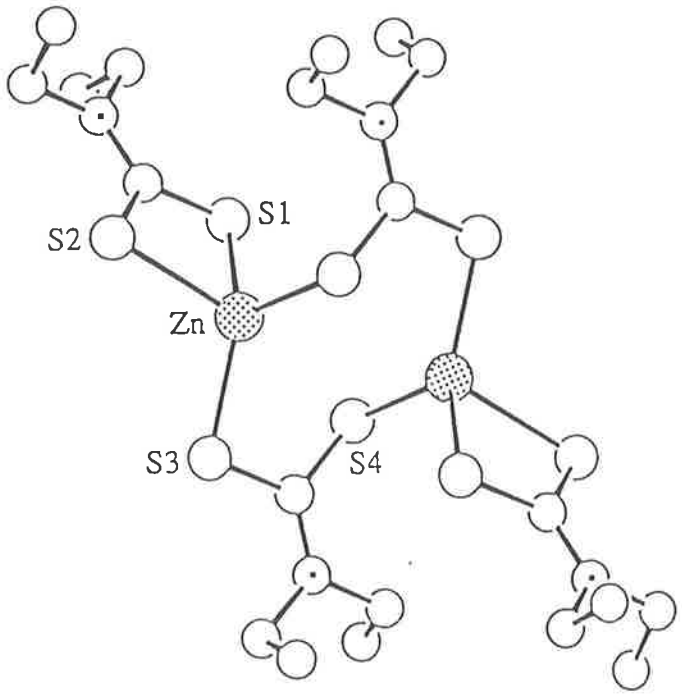


Fig. 1.4 Structural motif D, as represented by $[Zn(S_2CNEt_2)_2]$

described as a boat or saddle conformation and that of D as a twisted-chair conformation. In each of the structures, there are close intra-dimer Zn···S separations. As a result of the different orientation of the dithiocarbamate ligands, the close interaction involves the S(3) atom in structure C and the S(4) atom in structural type D. Table 1.1 collects important interatomic parameters for six of the seven structures as one of these is the subject of a space group ambiguity.

From Table 1.1 it can be noted that there is a remarkable constancy (in the context of this review) in the parameters about the zinc atoms in these structures. Typically, there is a slight asymmetry of approximately 0.1 Å in the Zn-S bonds formed by the chelating ligands and the Zn-S(4) bond distance is invariably longer than the Zn-S(3) bond (interchange the S(3) and S(4) atoms for the $[Zn(S_2CNMe_2)_2]$ structure, as above). The latter trend can be traced to the close intra-dimer Zn···S(4) interactions which lie in the range 2.815(1) Å to 3.036(6) Å. These distances are less than the sum of the van der Waals radii (3.1 Å) for these two atoms. If the Zn···S interactions were considered significant, the zinc atom could be described to exist in a distorted trigonal bipyramidal environment. The range of S-Zn-S angles about the zinc atoms (excluding the chelate angle and those involving the weakly associated S(4)' atom) shows that there is significant distortion from the ideal tetrahedral geometry, consistent with the close approach of the fifth sulfur atom. Whether the geometry is described as tetrahedral or trigonal bipyramidal is controversial, what is clear is that zinc has the ability to increase its coordination number in these systems.

1.1.3 *Dithiophosphate structures*

There are two zinc(II) bis(dithiophosphate) structures available in the literature. The structure of $[Zn(S_2P(OiPr)_2)_2]$ (Lawton, Kokotailo, 1969) adopts the structural motif C (Fig. 1.3), described above for $[Zn(S_2CNMe_2)_2]$, and has crystallographic two-fold symmetry. The chelating ligand forms Zn-S bond distances of 2.351(5) Å and 2.409(5) Å and the bridging distances are experimentally equivalent at 2.306(5) Å and

Table 1.1 Selected interatomic parameters (Å, deg.) for $Zn(S_2CNR_2)_2$

Compound	Zn-S(1)	Zn-S(2)	Zn-S(3)	Zn-S(4)	Zn...S [a]	Zn...Zn	S-Zn-S [b]	S-Zn-S [c]	Ref.
$[Zn(S_2CNMe_2)_2]$	2.333(6)	2.429(6)	2.373(6)	2.312(6)	3.036(6)	3.973(6)	105.8(2) - 136.5(2)	76.4(2) - 136.5(2)	g
$[Zn(S_2CNEt_2)_2]$	2.355(3)	2.443(3)	2.331(3)	2.383(3)	2.815(2)	3.545(4)	105.2(2) - 137.7(2)	75.8(2) - 160.0(2)	h
$[Zn(S_2CNiPr_2)_2]$	2.342(1)	2.454(1)	2.335(1)	2.377(1)	2.815(1)	3.545(1)	106.41(3) - 136.40(3)	68.89(3) - 155.31(2)	i
$[Zn(S_2CNR^1{}_2)_2]$ [d]	2.346(2)	2.468(2)	2.339(3)	2.387(2)	2.846(3)	3.580(3)	105.5(1) - 135.6(1)	68.1(1) - 156.6(1)	j
$[Zn(S_2CNR^2)_2]$ [e]	2.343(3)	2.493(3)	2.318(2)	2.380(3)	2.910(2)	3.701(1)	106.2(1) - 135.2(1)	75.6(1) - 157.0(1)	k
$[Zn(S_2CNR^3)_2]$ [f]	2.340(7)	2.445(7)	2.335(7)	2.343(7)	2.957(7)	3.630(7)	104.0(2) - 127.5(2)	67.5(2) - 153.3(2)	l

a S = S(3) for $[Zn(S_2CNMe_2)_2]$ and S(4) for the remaining structures; b S-Zn-S range excludes chelate angles; c all S-Zn-S angles; d R^1 = $-CH_2C(H)CH_2$; e $R^2 = (CH_2)_4$; f $R^3 = (CH_2)_6$; g Klug, 1966; h Bonamico, Mazzone, Vaciago, Zambonelli, 1965; i Miyamae, Ito, Iwasaki, 1979; j Kellö, Vrábel, Kettmann, Garaj, 1983; k Francetič , Leban, 1979; l Agre, Shugam, 1972b.

2.302(6) Å. The similarity of the Zn-S(bridging) distances may be traced to the absence of a significant intra-dimer interaction involving the S(3) atom (Zn···S(3) is 3.347(6) Å) and hence there is no elongation of the Zn-S(3) bond. The absence of a significant intramolecular interaction may be explained simply by the increased S-Y distances in the Y = P compound compared with the Y = C compound which has the effect of placing the S(3) atom further away from zinc. There is a profound change in structure when the isopropyl group is substituted for an ethyl group leading to $[Zn(S_2P(OEt)_2)_2]$ (Ito, Igarashi, Hagihara, 1969); structural motif E and Fig. 1.5. The zinc atom is tetrahedrally coordinated (range of S-Zn-S angles 85.9(2)° to 122.1(2)°), as above, but with two sites being occupied by a chelating dithiophosphate ligand (Zn-S(3) 2.351(6) Å and 2.401(7) Å). The two remaining sites are occupied by two sulfur atoms derived from two bidentate bridging ligands; Zn-S(1) 2.345(4) Å and Zn-S(2)' 2.337(7) Å. This arrangement leads to the formation of zig-zag chains (propagated by a crystallographic glide plane) throughout the lattice.

1.1.4 *Dithiophosphinate structures*

$[Zn(S_2PEt_2)_2]$ crystallises with two molecules in the asymmetric unit, each of which adopt structural motif C, having approximate two-fold symmetry (Calligaris, Nardin, Ripamonti, 1970). The average Zn-S bond distance in the tetrahedral geometry (S-Zn-S range: 85.3(3)° to 124.8(3)°) was calculated to be 2.342 Å with a range of 2.302(7) Å to 2.382(8) Å; no significant intra-dimer Zn···S interaction was observed. The centrosymmetric structure of $[Zn(S_2PnPr_2)_2]$ (Wunderlich, 1982) adopts motif D: the Zn-S(1, 2, 3, 4) distances are 2.344(2) Å, 2.450(2) Å, 2.306(2) Å and 2.321(2) Å, respectively (range of S-Zn-S: 84.1(1)° to 124.3(1)°). Again, no significant Zn···S interactions were found.

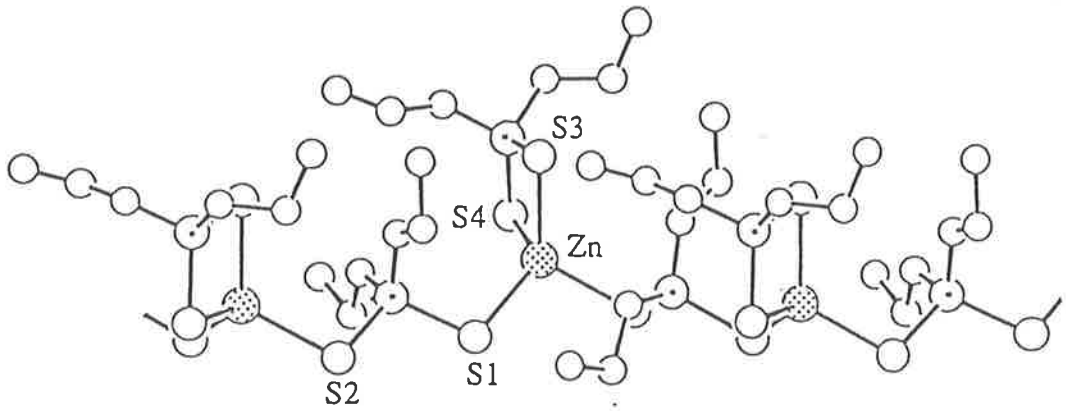


Fig. 1.5 Structural motif E, as represented by $[Zn(S_2P(OEt)_2)_2]$

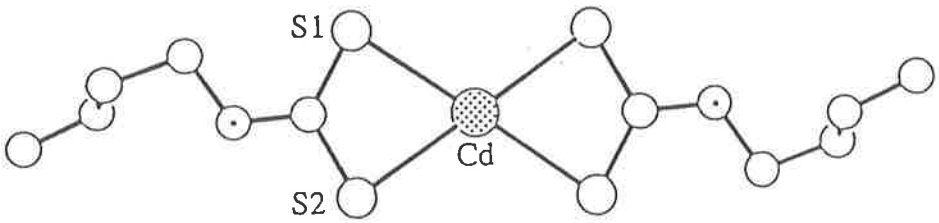


Fig. 1.6 Structural motif F, as represented by $[Cd(S_2CCOCH_2CH_2OMe)_2]$

1.2 CADMIUM BIS 1,1-DITHIOLATES

A number of cadmium bis (1,1-dithiolate) structures have been determined by X-ray crystallographic methods. In the main, similar structures to those observed previously for the zinc bis (1,1-dithiolates) are adopted by the cadmium dithiolates with the expected differences in the derived interatomic parameters. There is, however, one unique coordination pattern for these compounds.

1.2.1 *Xanthate structures*

Four cadmium bis (xanthate) structures, $[Cd(S_2COR)_2]$, are available with three, R = Et, iPr and nBu being isostructural with $[Zn(S_2COEt)_2]$, i.e. adopting structural type B (Iimura, Ito, Hagihara, 1972; Iimura, 1973; Rietveld, Maslen, 1965). The coordination polyhedron around the cadmium atoms is distorted tetrahedral with bond lengths and angles as listed in Table 1.2; these show the expected variations owing to the increased size of the cadmium atom compared with that of zinc. Whereas the three structures cited have a precedent, there is one other motif for $[Cd(S_2COR)_2]$.

The centrosymmetric structure of $[Cd(S_2COCH_2CH_2OMe)_2]$ (Abrahams, Hoskins, Tiekink, Winter, 1988) is shown in Fig. 1.6 (motif F) and features a square planar cadmium centre with symmetrically chelating xanthate ligands; Cd-S(1) 2.6265(5) Å and Cd-S(2) 2.6397(7) Å. There are weak intermolecular Cd...S interactions above and below the CdS_4 square plane of 3.0225(8) Å which, although less than the sum of the van der Waals radii of cadmium and sulfur (3.4 Å), are not in the immediate coordination environment of the cadmium atom; if considered significant, the cadmium geometry would be described as tetragonally distorted octahedral, $Cd(S_4)S_2$. A striking feature of structural type F is the different coordination geometry about the cadmium, which is not repeated often in the other structures cited in this introduction.

Table 1.2 Selected parameters (Å, deg.) for cadmium bis (1,1-dithiolates)

Compound	Cd-S	S-Cd-S	Ref.
$[Cd(S_2COEt)_2]$	2.544(2) - 2.590(3)	94.47(5) - 121.29(5)	b
$[Cd(S_2COiPr)_2]$	2.536(3) - 2.586(2)	90.78(8) - 110.90(7)	c
$[Cd(S_2COnBu)_2]$	2.560(9) - 2.619(8)	95.3(3) - 121.5(3)	d
$[Cd(S_2CNEt_2)_2]$	2.536(4) - 2.644(3), 2.800(3)	70.9(1) - 158.9(1)	e
$[Cd(S_2CNnBu_2)_2]$	2.513(5) - 2.601(7), 2.888(5)	66.4(2) - 160.1(2)	f
$[Cd(S_2CNR_2)_2]$ [a]	2.54(1) - 2.63(1), 2.873(1)	67.0(4) - 160.0(4)	g
$[Cd(S_2P(OiPr)_2)_2]$	2.486(7) - 2.590(8), 3.194(8)	79.1(3) - 121.6(2)	h
$[Cd(S_2P(OcHex)_2)_2]$	2.517(5) - 2.660(8), 3.34(1)	78.3(2) - 131.4(2)	i
$[Cd(S_2PEt_2)_2]$	2.543(2) - 2.653(3), 3.1(1)	79.0(1) - 127.6(1)	j
$[Cd(S_2PPh_2)_2]$	2.520(1) - 2.641(1), 3.078(1)	79.85(4) - 137.35(4)	k

a R_2 = cyclo-$(CH_2)_6$, b Iimura, Ito, Hagihara, 1972; c Iimura, 1973); d Rietveld, Maslen, 1965; e Domenicano, Torelli, Vaciago, Zambonelli, 1968; f Casas, Sánchez, Bravo, García-Fontán, Castellano, Jones, 1989; g Agre, Shugam, 1972a; h Lawton, Kokotailo, 1969; i Casas, Castiñeiras, García -Tasende, Sánchez, Sordo, Vázquez-López, 1995; j Svensson, Albertsson, 1991; k Casas, García-Tasende, Sánchez, Sordo, Vázquez-López, Castellano, Zukerman-Schpector, 1994.

1.2.2 *Dithiocarbamate structures*

One of the three available $Cd(S_2CNR_2)_2$ structures, *i.e.* R = nBu (Casas, Sánchez, Bravo, García-Fontán, Castellano, Jones, 1989), resembles structural motif C, as found for $[Zn(S_2CNMe_2)_2]$, whereas R = Et (two determinations (Domenicano, Torelli, Vaciago, Zambonelli, 1968; Shugam, Agre, 1968) and R_2 = cyclo-$(CH_2)_6$ (Agre, Shugam 1972a), are similar to the structure found for $[Zn(S_2CNEt_2)_2]$ (structural type D). Selected geometric parameters are collected in Table 1.2. As was the case with the zinc structures, there are close Cd···S interactions of approximately 2.8 Å across the central eight-membered ring. These represent significant interactions in $Cd(S_2CNR_2)_2$ and thus, each cadmium atom must be considered as five-coordinate, distorted trigonal bipyramidal; the structures are illustrated in Figs 1.7 and 1.8 for R = nBu (the axial angle S(2)-Cd-S(4)' is 160.0(4)°) and R_2 = $(CH_2)_6$ (S(2)-Cd-S(3)' angle is 160.1(2)°), respectively. Given the close similarity of these structures to the structural types C and D described above for the zinc compounds, the cadmium structures are assigned motifs C' and D', respectively.

1.2.3 *Dithiophosphate structures*

There are two cadmium bis (dithiophosphate) structures reported in the literature, namely $[Cd(S_2P(OiPr)_2)_2]$ (Lawton, Kokotailo, 1969) and $[Cd(S_2P(OCy)_2)_2]$ (Casas, Castiñeiras, García-Tasende, Sánchez, Sordo, Vázquez-López, 1995). The first of these is isomorphous to its zinc analogue, $[Zn(S_2P(OiPr)_2)_2]$ (Lawton, Kokotailo, 1969), but adopts structural type C', *i.e.* with tetrahedral cadmium centres, owing to the fact that the intramolecular Cd...S(3)' interaction is long at 3.194(8) Å (see above); other parameters are listed in Table 1.2. The centrosymmetric $[Cd(S_2P(OCy)_2)_2]$ structure adopts the dimeric structural motif D', details are given in Table 1.2.

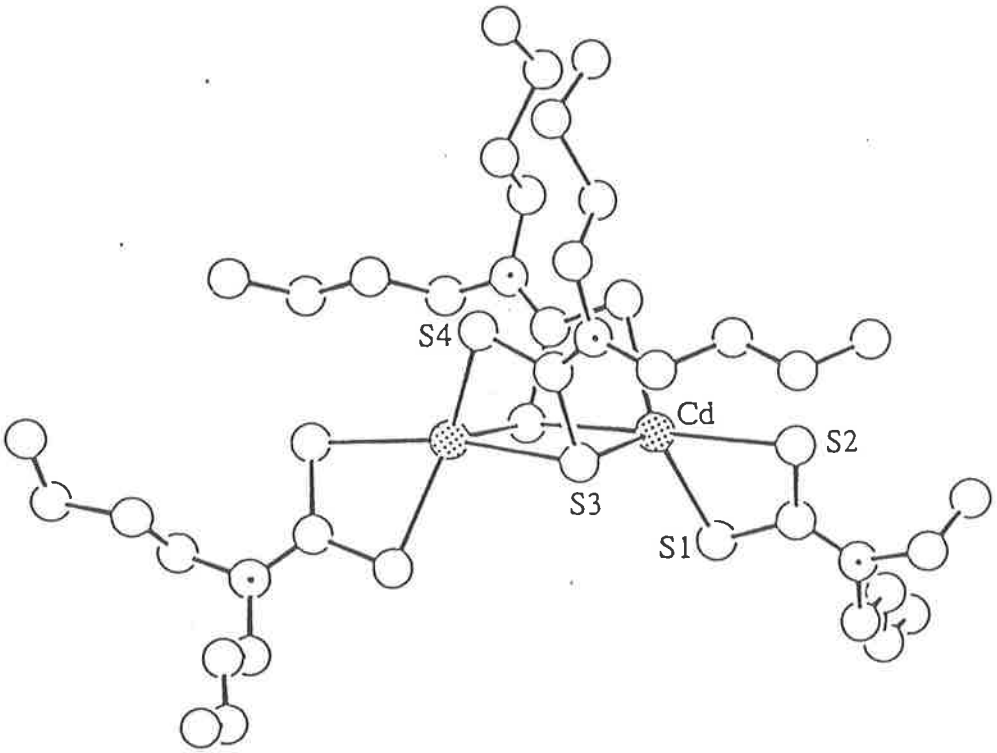


Fig. 1.7 Structural motif C', as represented by [Cd(S_2CNnBu_2)2]

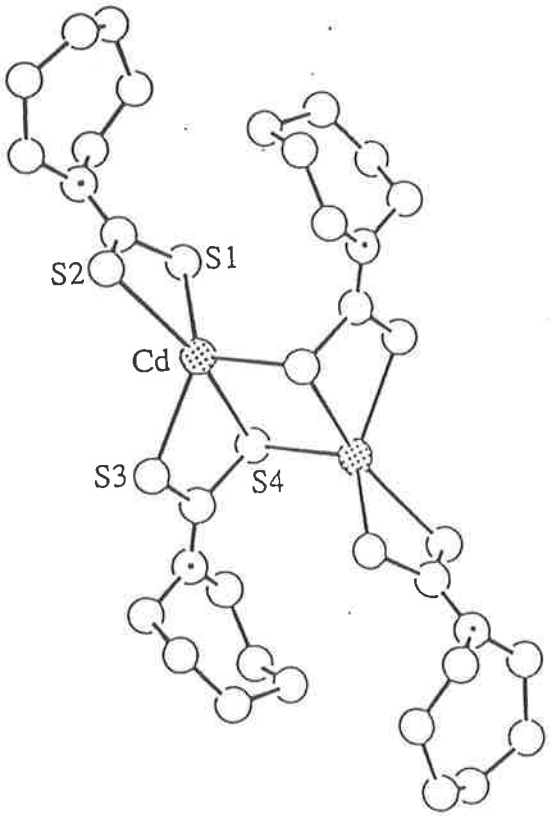


Fig. 1.8 Structural motif D', as represented by [Cd(S_2CNR)2], where R = (CH_2)6

1.2.4 *Dithiophosphinate structures*

Each of centrosymmetric $[Cd(S_2PEt_2)_2]$ (two independent studies (Svensson, Albertsson, 1991; Wunderlich, 1986)) and $[Cd(S_2PPh_2)_2]$ (Casas, García-Tasende, Sánchez, Sordo, Vázquez-López, Castellano, Zukerman-Schpector, 1994) adopts structural motif D' as illustrated in Fig. 1.8; selected parameters are given in Table 1.2.

1.3 MERCURY BIS 1,1-DITHIOLATES

There are six different coordination patterns observed for the mercury bis (1,1-dithiolates), three of these motifs have yet to be described thus far. Generally, mercury tends to form four coordinate, distorted tetrahedral geometries owing to disparity in the Hg-S separations.

1.3.1 *Xanthate structures*

There are five structures available with the general formula $Hg(S_2COR)_2$. The compound with R = Me (Tiekink, 1987) defines structural motif G as illustrated in Fig. 1.9. The xanthate ligands adopt different coordination modes with one being bidentate bridging and the other being monodentate. A polymeric chain arises as a result of the bridging ligands and the three-coordinate, T-shaped geometry about each mercury atom is completed by the monodentate ligand. The bridging Hg-S(3) and Hg-S(4)' distances are 2.383(3) Å and 2.924(3) Å, respectively and the Hg-S(1) bond distance is 2.365(3) Å. The Hg-S(4)' separation is well within the sum of the van der Waals radii of these atoms of 3.3 Å and hence must be considered an interaction, if not, the coordination geometry would be linear HgS_2. It should be noted that both xanthate ligands are orientated so that their oxygen atoms are relatively close to the mercury atom. However, the Hg···O separations of 2.926(8) Å and 2.932(8) Å, while less than the sum of the van der Waals

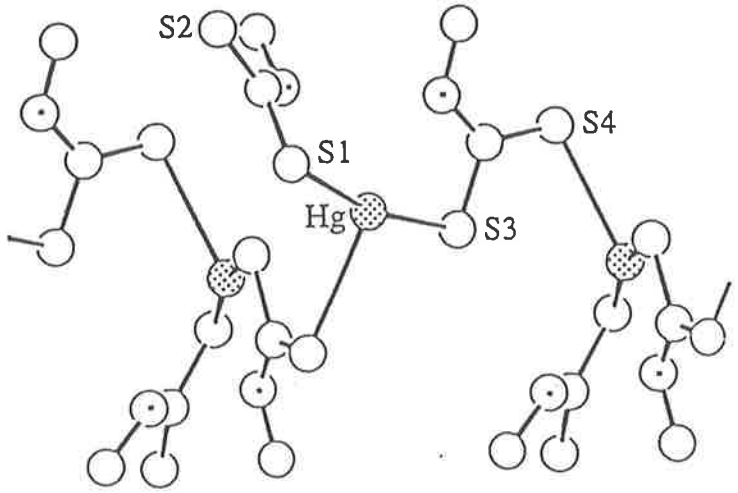


Fig. 1.9 Structural motif G, as represented by [Hg(S_2COMe)$_2$]

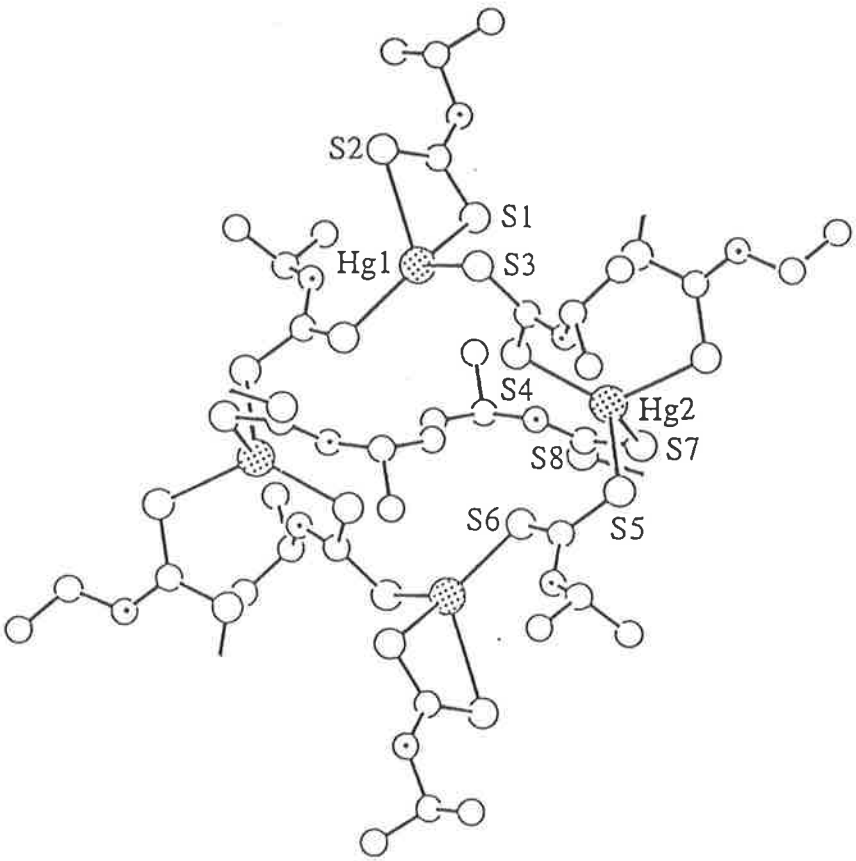


Fig. 1.10 Structural motif H, as represented by [Hg(S_2COiPr)$_2$]

radii for mercury and oxygen of 3.0 Å, are not indicative of significant bonding interactions. The remaining structures in this category feature distorted tetrahedral HgS_4 units and sixteen-membered rings.

Structural motif B (see Fig. 1.2) is adopted for the $Hg(S_2COR)_2$ compounds with R = Et (two polymorphs, labelled α (Watanabe, 1977) and β (Chieh, Moynihan, 1980) and nPr (with crystallographic two-fold symmetry (Hounslow, Tiekink, 1991)). Geometric parameters are collected in Table 1.3. The difference between the two polymorphs of $[Hg(S_2COEt)_2]$ rests in the relative orientation of the layers of sixteen-membered rings. In the α-form, which crystallises in the non-centrosymmetric space group $P2_1$, successive layers are in the same orientation, whereas in the β-polymorph (space group $P2_1/c$) the layers are related by a centre of inversion. The coordination geometries about the mercury atoms are based on distorted tetrahedra. A comparison of the derived parameters for the zinc, cadmium and mercury structures of this motif reveals that the distortions from the ideal tetrahedral geometry progressively increase. In the $[Hg(S_2COEt)_2]$ structures, the Hg-S bond distances fall into two classes, with two being shorter than the other two (Table 1.3). The angle subtended at the mercury centre by the two sulfur atoms that form the stronger interactions approaches 150°, a result which indicates that the geometry is approaching linearity (*cf.* $[Hg(S_2COMe)_2]$ described above), with the weaker Hg-S bonds lying on either side.

The remaining structure in this category is that found for $[Hg(S_2COiPr)_2]$ (Watanabe, 1981) as illustrated in Fig. 1.10; this represents motif H. This structure is based on the familiar (for the xanthate structures) sixteen-membered rings. In this case, two of the mercury atoms, opposite each other, have their distorted tetrahedral coordination environment completed by a chelating xanthate ligand. In this respect, this motif resembles that of motif A. The remaining mercury atoms are surrounded by four sulfur atoms, derived from four bridging atoms, *i.e.* similar to that found in motif B. A key difference concerns the association between the tetramers. In motif B, there are no xanthate bridges between successive layers, however, in H, there are. The chelating

ligand about the Hg(1) atom forms asymmetric distances of 2.48(1) Å and 2.88(1) Å and the bridging distances are 2.62(1) Å and 2.442(9) Å. In addition, the Hg(1) atom forms a weak contact of 3.112(9) Å to the S(4) atom. The range of angles subtended at this atom is 66.8(3)° - 143.2(3) °. The range of Hg(2)-S distances is 2.477(9) Å - 2.74(1) Å and S-Hg(2)-S is 91.0(3) ° - 123.7(3) °.

The three motifs observed for $[Hg(S_2COR)_2]$ are remarkably different from each other, ranging from a linear chain for R = Me, to a layer structure for R = Et, to a network structure for R = iPr. Hence, the simple variation of R substituents from R = Me to Et to iPr results in a change from an one-, to a two- to a three-dimensional structure.

Table 1.3 Selected parameters (Å, deg.) for mercury bis (xanthates)

Compound	Hg-S	S-Hg-S	Ref.
α-$[Hg(S_2COEt)_2]$	2.313(8) - 2.94(1)	81.8(3) - 148.8(3)	a
β-$[Hg(S_2COEt)_2]$	2.417(4) - 2.854(4)	84.3(1) - 147.7(1)	b
$[Hg(S_2CO^nPr)_2]$	2.418(3) - 2.835(4)	82.0(3) - 148.9(3)	c

a Watanabe, 1977; b Cheih, Moynihan, 1980; c Hounslow, Tiekink, 1991.

1.3.2 *Dithiocarbamate structures*

There are five $[Hg(S_2CNR_2)_2]$ structures in the literature and these adopt no less than four different motifs.

The structure of $[Hg(S_2CNH_2)_2]$ (Chieh, Cheung, 1981) is represented in Fig. 1.11 and is labelled motif I. The layer structure is made up of interconnected eight-membered $[-Hg-S-C-S-]_2$ rings, comprising two mercury atoms that are bridged by two dithiocarbamate ligands. These rings are connected to symmetry related rings via bridging dithiocarbamates. The larger rings in the structure thus constructed, contain six

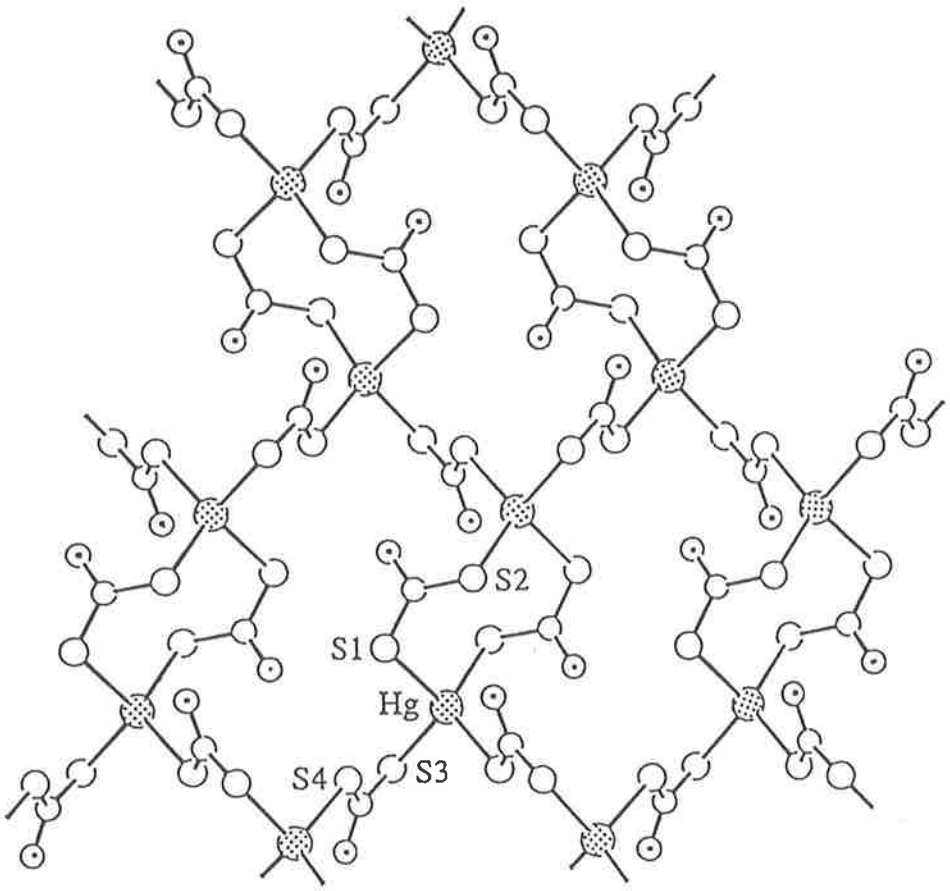


Fig. 1.11 Structural motif I, as represented by $[Hg(S_2CNH_2)_2]$

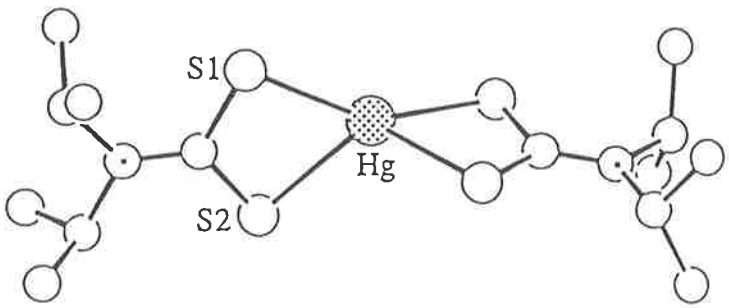


Fig. 1.12 Structural motif J, as represented by $[Hg(S_2CNiPr_2)_2]$

mercury atoms and eight bridging dithiocarbamate ligands. The range of Hg-S bond distances is relatively narrow at 2.499(4) Å - 2.629(4) Å and the range of angles subtended at the mercury atom is 100.9(1)° - 119.9(1)°. Clearly, the geometry about the mercury atom is distorted tetrahedral.

There are two polymorphs of $[Hg(S_2CNEt_2)_2]$, labelled α (Iwasaki, 1973) and β (Iwasaki, 1973; Healy, White, 1973), each of which adopts a distinct structure in the solid state. The α- form of $[Hg(S_2CNEt_2)_2]$ adopts structural type D' as illustrated in Fig. 1.8. The four Hg-S interactions lie in the range 2.418(7) Å - 2.698(6) Å and the intramolecular Hg···S(4)' interaction is 3.137(6) Å and indicate a distorted tetrahedral geometry about mercury (S-Hg-S 70.0(2)° - 146.0(2)°). By contrast to the dimeric structure of α-$[Hg(S_2CNEt_2)_2]$, the structure of the other polymorph is monomeric (see below). The structure of $[Hg(S_2CNR)_2]$, where HNR is the secondary amine 4-methylpiperidine (Benedetti, Fabretti, Preti, 1988), adopts the same motif with Hg-S distances in the range 2.450(2) Å - 2.669(1) Å and S-Hg-S angles in the range 69.53(4)° - 139.16(6)°. The intramolecular Hg···S(4) contact is 3.127(2) Å.

The structure of β-$[Hg(S_2CNEt_2)_2]$ has been subjected to two studies (Iwasaki, 1973; Healy, White, 1973) and is closely related to the structure of $[Cd(S_2COCH_2CH_2OMe)_2]$, motif F (Fig. 1.6). The mercury atom is situated on a centre of inversion and the two independent Hg-S distances are 2.398(4) Å and 2.965(4) Å. A trend to a linear HgS_2 geometry is thus apparent with the S(1)-Hg-S(1)' angle being constrained crystallographically to 180°. In the lattice, there are additional interactions between the mercury atoms and symmetry related S(2) atoms of 3.292(4) Å. The observation of two distinct motifs for $[Hg(S_2CNEt_2)_2]$ indicates that there is relatively little energy difference between them.

The final mercury dithiocarbamate structure is that of $[Hg(S_2CNiPr_2)_2]$ (Ito, Iwasaki, 1979), as shown in Fig. 1.12; this is structural motif J. The monomeric structure has crystallographic two-fold symmetry and features a distorted tetrahedral

geometry: Hg-S 2.445(4) Å - 2.645(4) Å and S-Hg-S 70.7(2)° - 149.7(2)°. The closest intermolecular Hg···S contact is 3.408(6) Å.

1.3.3 *Dithiophosphinate structure*

The chain structure of [Hg(S_2P(OiPr)$_2$)$_2$] (Lawton, 1971), with both chelating and bridging ligands, resembles that of [Zn(S_2P(OEt)$_2$)$_2$], *i.e.* motif E as illustrated in Fig. 1.5. Distortions from the ideal tetrahedral geometry are significant however, as seen in the Hg-S distances of 2.391(6) Å and 2.888(5) Å for the chelating ligand, and 2.388(5) Å and 2.748(5) Å for the bridging ligand; the range of S-Hg-S angles is 76.3(2)° - 149.7(2)°.

1.4 SUMMARY

The above survey demonstrates the rich diversity of structural motifs for the binary 1,1-dithiolates of zinc, cadmium and mercury. A summary of the coordination patterns adopted by these compounds is presented in Fig. 1.13 (after Ito, 1972). The two monomeric motifs, *i.e.* F and J, which differ only in the detail of the coordination geometry about the central element (*i.e.* square planar and tetrahedral, respectively), are represented by one schematic in this diagram. Similarly, motifs C and D are combined as the difference between them relates to a different orientation of the bridging ligands. Further, the motifs C' and D' are combined with these as the differences between C and C' (and D and D') relate to the magnitude of the intramolecular M···S interactions (see text). Having combined closely related motifs, there are eight distinct coordination patterns for these compounds. The assignment of particular structures to motifs is given in Table 1.4.

Some broad observations can be made concerning the adoption of particular motifs. Generally, xanthate structures tend to be based on sixteen-membered rings with varying associations between them. Also, dithiocarbamate structures are often dimeric.

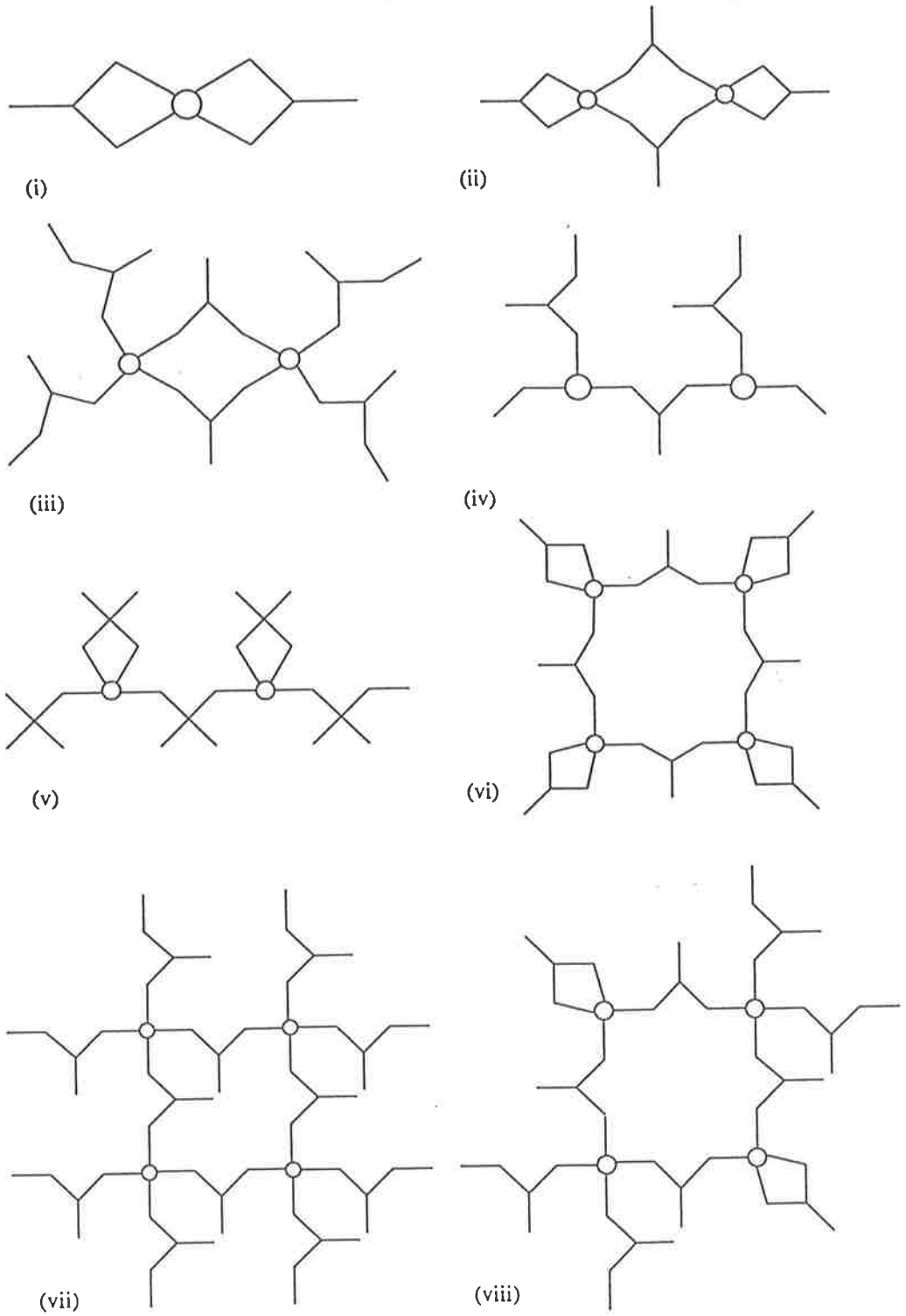


Fig. 1.13 Schematic diagrams of the major structural motifs found in the solid state for zinc, cadmium, and mercury bis (1,1-dithiolates): (i) motifs F and J, (ii) motifs C and D (C' and D'), (iii) motif I, (iv) motif G, (v) motif E, (vi) motif A, (vii) motif B and (viii) motif H.

Table 1.4 Summary of structural motifs for zinc, cadmium and mercury bis (1,1-dithiolates) *

Motif	Compound
A	$[Zn(S_2COiPr)_2]$
B	$[Zn(S_2COEt)_2]$; $[Cd(S_2COEt)_2]$; $[Cd(S_2COiPr)_2]$; $[Cd(S_2COnBu)_2]$; α-and β-$[Hg(S_2COEt)_2]$; $[Hg(S_2COnPr)_2]$
C	$[Zn(S_2CNMe_2)_2]$; $[Zn(S_2P(OiPr)_2)_2]$; $[Zn(S_2PEt_2)_2]$
C'	$[Cd(S_2CNnBu_2)_2]$; $[Cd(S_2P(OiPr)_2)_2]$
D	$[Zn(S_2CNEt_2)_2]$; $[Zn(S_2CNnPr_2)_2]$; $[Zn(S_2CNiPr_2)_2]$; $[Zn(S_2CNR^1{}_2)_2]$; $[Zn(S_2CNR^2)_2]$; $[Zn(S_2CNR^3)_2]$; $[Zn(S_2PnPr_2)_2]$
D'	$[Cd(S_2CNEt_2)_2]$; $[Cd(S_2CNR^3)_2]$; $[Cd(S_2P(OCy)_2)_2]$; $[Cd(S_2PEt_2)_2]$; $[Cd(S_2PPh_2)_2]$; α-$[Hg(S_2CNEt_2)_2]$
E	$[Zn(S_2P(OEt)_2)_2]$; $[Hg(S_2P(OiPr)_2)_2]$
F	$[Cd(S_2COCH_2CH_2OMe)_2]$; β-$[Hg(S_2CNEt_2)_2]$
G	$[Hg(S_2COMe)_2]$
H	$[Hg(S_2COiPr)_2]$
I	$[Hg(S_2CNH_2)_2]$
J	$[Hg(S_2CNiPr_2)_2]$

* $R^1 = -CH_2C(H)CH_2$; $R^2 = (CH_2)_4$; $R^3 = (CH_2)_6$

Although there is only a small number of dithiophosphate/dithiophosphinate structures available, most are dimeric. The reasons for the rich structural diversity in these compounds, and indeed in related systems (Haiduc, Sowerby, Lu, 1995; Tiekink, Winter, 1992; Tiekink, 1991, 1992, 1994, 1995), is not clear.

In the present case, the demands of the central element clearly influences the ultimate structure adopted in the solid state. Further, the ability of the 1,1-dithiolate ligands themselves to adopt different coordination modes, ranging from monodentate to bidentate, as well as weaker associations, is manifest. Over and above these considerations, one needs to take into account i) electronic and ii) steric factors associated with the 1,1-dithiolate ligands, and more difficult to quantify, iii) crystal packing effects. This is particularly true for instances where very different structures are observed for only minor changes in the composition of the ligands, *e.g.* the $[Hg(S_2COR)_2]$ compounds.

In order to ascertain the relative importance of these factors in xanthate and dithiocarbamate (^-S_2COR and $^-S_2CNR_2$, R=alkyl, aryl) structures, a systematic investigation of their solid state structures has been undertaken. To begin, data of nickel(II) bis(xanthates) and nickel(II) bis(dithiocarbamates) was collated with several additional related structures so as to ascertain whether there are any systematic features in the nickel-atom geometries that may be correlated with the electronic and/or steric profiles of the ligands.

The decision to systematically examine the structures of nickel(II) bis xanthates and nickel(II) bis(dithiocarbamates) is motivated by two primary reasons. The first relates to the fact that there is a characteristic mode of coordination of the 1,1-dithiolate ligands in these nickel(II) complexes. Without exception all of the nickel(II) complexes studied thus far crystallise with a square planar geometry of sulfur atoms around the nickel centre. This affords the possibility of systematically investigating the coordinating ability of the 1,1-dithiolate ligands in a consistent coordination environment. The square planar geometry adopted suggests that the steric profiles of the ligands do not influence the coordination geometry and hence, the electronic effects of the ligands may be

monitored. A second reason is the availability of a relatively large number of X-ray crystallographic studies thereby making possible a thorough and comprehensive analysis of the effect on the nickel atom geometry of changing the R substituent in each series of complexes.

Following the investigation of the nickel 1,1-dithiolate compounds a similar investigation involving a main group metal is undertaken. Tellurium was chosen as the structural chemistry of the tellurium bis(xanthates), $Te(S_2COR)_2$, and bis(dithiocarbamates), $Te(S_2CNR_2)_2$, is remarkable in that a common structural motif is adopted for these compounds in the solid state. In this respect, the tellurium compounds resemble the situation found in many transition metal bis xanthate complexes (Tiekink, Winter, 1992).

Given that a consistent motif is found for the tellurium bis(1,1-dithiolates) and nickel bis(1,1-dithiolates), the opportunity exists to systematically analyse their structures with the view of determining the influence on the nickel/tellurium atom geometry of moderating the nature (electronic/steric) of the R substituents of the ligands, *i.e.* to discover whether a correlation exists between the Ni-S/Te-S distances and R. In this way, it may be possible to ascertain the magnitude of the 'electronic' effect of R and hence, provide information on the coordinating ability of the ligands as R is varied.

Following the above investigation, the structural investigation into the coordination of 1,1-dithiolates to the metals zinc, cadmium and mercury, in that order is undertaken. Several new structures for each metal is reported and a comparison with the literature analogs results. The structures were analysed in order to determine the reason(s) for their observed structural diversity. Finally, in order to investigate the electronic profile of the xanthate and dithiocarbamate anions further, their theoretical structures were calculated employing *ab initio* molecular orbital theory.

Chapter 2

EXPERIMENTAL

This chapter is divided into two parts, firstly a discussion of the instrumentation used to characterise the compounds and a general procedure for solving their crystal structures, with specifics for a particular compound reported in the Appendix. Secondly the synthesis of the ligands and the compounds is reported with the characterisation of the compounds discussed. All experimental procedures were undertaken by the author.

2.1 INSTRUMENTATION

2.1.1 *Infrared spectroscopy (IR)*

Infrared spectra for all compounds were recorded as KBr discs on a Perkin-Elmer 1720X FT spectrometer in the range of 4000 - 400 cm^{-1} and calibrated with the polystyrene absorption at 1601 cm^{-1}.

2.1.2 *Nuclear magnetic resonance spectroscopy (NMR)*

Proton (^{1}H) and carbon-13 proton decoupled (^{13}C) NMR spectra were recorded on a either a Bruker ACP-300 or Gemini 200 NMR spectrometer with deuterated chloroform, $CDCl_3$, as the solvent. The recording frequencies used were 300.145 (199.953) MHz for ^{1}H NMR and 75.479 (50.283) MHz for ^{13}C NMR. The internal reference used was $SiMe_4$ (TMS). Selected samples were recorded in deuterated DMSO due to the lack of solubility in $CDCl_3$.

2.1.3 *Melting points*

All melting points were determined using a Gallenkamp melting point apparatus calibrated with benzil (m. pt 94-95°C).

2.1.4 *Crystallography*

Crystal data and refinement details for all structures are listed in the Appendix of this thesis. This section outlines the general procedures employed for each analysis. Crystals were mounted on glass fibers using super glue or silicon grease. Collection temperatures were either room temperature, 20°C or colder at -70°C or -160°C as noted in the crystallographic parameter tables.

Intensity data were measured on a Rigaku AFC6R diffractometer fitted with Mo$K\alpha$ (graphite monochromator) radiation, λ = 0.71073 Å. The ω:2θ scan technique was employed to measure data up to a maximum Bragg angle of 25.0-27.5°. The net intensity values of three standard reflections, remeasured after every 400 intensity measurements, allowed for the determination of crystal stability. The data sets were corrected for Lorentz and polarization effects (teXsan, 1993) and for absorption effects employing an empirical correction (Walker, Stuart, 1983). DIFABS was typically applied twice. The first correction was applied after the inclusion of all atoms and refinement of the non-hydrogen atoms isotropically. The second application was after anisotropic refinement. Accordingly, two sets of transmission factors were calculated and those reported in the appendices are the final ranges. Thus, given the empirical nature of the correction and its mode of application, the transmission factor range is arbitrary and does not necessarily have physical meaning. All of the unique reflections measured were used in the subsequent analysis. Values reported in the Tables of bond lengths and angles in the text were calculated using reflections that satisfied the $I \geq 3.0\sigma(I)$ criterion of observability.

The unit cell dimensions were determined from the least squares refinement of 25 carefully-centred reflections in all cases.

All the structures were solved by direct-methods (Beurskens, Admiraal, Beuskens, Bosman, García-Granda, Smits, Smykalla, 1992) and each refined by a full-

matrix least-squares procedure based on F and $\underline{F}^2$ (all data) (teXsan, 1993). The function minimised was

$$\sum_{i=1}^{n} w_i (|F_{obs}|_i - |F_{calc}|_i)^2$$

where n is the number of reflections, and

$$w = \frac{1}{\sigma^2(F_{obs})}.$$

Non-hydrogen atoms were refined with anisotropic displacement parameters (where possible) and hydrogen atoms were included in their calculated positions (C-H 0.97 Å). A sigma weighting scheme was applied and the refinement continued until convergence in each case. At convergence, the values of R and R_w were obtained, where

$$R = \frac{\sum_{i=1}^{n} (|F_{obs}|_i - |F_{calc}|_i)}{\sum_{i=1}^{n} |F_{obs}|_i} \text{ and } R_w = \left\{ \frac{\sum_{i=1}^{n} w_i (|F_{obs}|_i - |F_{calc}|_i)^2}{\sum_{i=1}^{n} w_i |F_{obs}|^2} \right\}.$$

The analysis of variance for each refinement revealed no special features indicating that an appropriate weighting scheme was applied in each case.

Scattering factors for all the atoms were those incorporated in the teXsan software package (teXsan, 1993) which was installed on a Silicon Graphics Iris Indigo computer system. Tables of bond distances and bond angles, fractional atomic coordinates, anisotropic thermal and hydrogen atom parameters, and structure factors for all determinations are located in the Appendices. The expression for the anisotropic thermal parameter is

$$T_{aniso} = \exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2hka^*b^*U_{12} + 2hla^*c^*U_{13} + 2klb^*c^*U_{23})]$$

and the expression for *B*eq is

$$B_{eq} = \frac{8\pi^2(U_{11} + U_{22} + U_{33})}{3}.$$

All diagrams were drawn utilising the ORTEP program (Johnson, 1976) at 50% probability thermal ellipsoids unless otherwise specified, with unit cell diagrams drawn at 25% probability thermal ellipsoids.

When appropriate, the absolute configuration of a structure was determined by refining each hand and comparing the final values of R_w. As a matter of course, each chiral structure was examined by the 'Look for Centres of Symmetry' routine in teXsan (teXsan, 1993).

2.2 SYNTHESIS

2.2.1 *Chemicals*

A number of 1,1-dithiolate ligands were synthesised and then coordinated to the appropriate metal atom. The alcohols used in the syntheses of the xanthate ligands were: 3,3-dimethyl-n-butanol, 3-methyl-n-butanol, 1-propanol, 2-propanol, 1-butanol, hexyl alcohol, cyclohexanol, 1-pentanol, benzyl alcohol and were used as obtained.

The amines used in the synthesis of the dithiocarbamate ligands include: dimethylamine, diisopropylamine, dibutylamine, dicyclohexylamine, diisobutylamine, cyclohexylisopropylamine, cyclohexylethylamine and were used without further purification.

The metal atoms came from the following compounds, nickel chloride, cadmium chloride, mercury(II)chloride, zinc chloride and tellurium oxide and were used without purification.

Carbon disulphide and sodium/potassium hydroxide were used as supplied. All the solvents employed for recrystallisation were of analytical grade and were used as obtained.

2.2.2 *Preparation of starting materials.*

Xanthates

The preparation of the xanthates followed the established literature procedure (Coucouvanis, 1970). This required dissolving NaOH/KOH (usually 5 grams) in an excess of the appropriate alcohol. Once dissolved CS_2 was added dropwise in excess. A pale yellow to orange precipitate would develop on addition of CS_2 and the solution was stirred for 1 hour to ensure completion of the reaction before being left to dry.

Dithiocarbamates

A similar procedure to that outlined above was employed for the synthesis of the dithiocarbamates as described in the literature (Coucouvanis, 1970). NaOH/KOH was dissolved in an excess of the required amine and excess CS_2 was added dropwise. The solution developed a pale yellow to orange precipitate on the addition of CS_2 and was stirred for a further hour before it was left to dry. The dithiocarbamates were then recrystallised from acetone or an acetone/petroleum spirit mixture before being used.

2.2.3 *Preparation of bis(xanthate/dithiocarbamate)M(II) compounds, M = Ni, Zn, Cd, Hg*

The procedure to coordinate the 1,1-dithiolate ligand to the metal atom involved the 1:2 reaction between $NiCl_2.6H_2O$ ($ZnCl_2$, $CdCl_2$, $HgCl_2$) and the respective

Table 2.1 Physical characteristics of the compounds obtained.

Compound	colour	m. pt. (°C)	recrystallisation solvent
[Ni(S_2COMe)2]	Brown	136-137	$CHCl_3$/iPrOH [a]
[Ni(S_2COnPr)2]	Brown	105-106	$CHCl_3$/Pet Spirit 120-160 [b]
[Ni(S_2COiPr)2]	Brown	116-117	$CHCl_3$/Pet Spirit 120-160 [b]
[Ni(S_2COnBu)2]	Brown	95	Acetonitrile/ether [b]
[Ni(S_2CO3-MeBu)2]	Brown	91	Acetonitrile /ether [b]
[Ni(S_2CO3,3-DiMeBu)2]	Brown	143	Acetonitrile /ether [b]
[Ni(S_2COiBu)2]	Brown	138-139	$CHCl_3$/iPrOH [a]
[Ni(S_2COHex)2]	Brown	52-53	Acetone/ Acetonitrile [a]
[Ni(S_2COCy)2]	Brown	168-169	CH_2Cl_2/ Acetonitrile [a]
[Ni(S_2CN(Cy)2)2]	Olive green	180-181 dec.	CH_2Cl_2/EtOH [a]
[Ni(S_2CN(Cy)Et)2]	Olive green	218	Acetone/iPrOH [a]
[Te(S_2COnPr)2]	Red	80	CH_2Cl_2/EtOH [a]
[Te(S_2COiBu)2]	Red	88-89	Acetone/iPrOH [a]
[Te(S_2CO-3,3-Me_2Bu)2]	Red	125-126	$CHCl_3$/EtOH [a]
[Te(S_2COCH_2Ph)2]	Red	115-116	CH_2Cl_2/EtOH [a]
[Te(S_2CNCy2)2]	Red	225-227 dec.	$CHCl_3$/EtOH [a]
[Zn(S_2COnBu)2]	Colourless	119-120	Benzene/Pet Spirit 100/120[a]
[Zn(S_2CN(Cy)2)2]	Colourless	219-221	CH_2Cl_2/ Acetonitrile [a]
[Zn(S_2CN(Et)Cy)2]	Colourless	175-177	$CHCl_3$/EtOH [a]
[Cd(S_2CN(iPr)2)2]	Colourless	218 dec.	DMF
[Cd(S_2CN(iBu)2)2]	Colourless	167	CH_2Cl_2/iPrOH [a]
[Cd(S_2CN(Cy)2)2]	Colourless	>350	CH_2Cl_2/iPrOH [a]
[Cd(S_2CN(Et)Cy)2]	Colourless	236-238	$CHCl_3$/EtOH [a]

Table 2.1 Cont.

Compound	colour	m. pt. (°C)	recrystallisation solvent*
[Hg(S_2COnBu)2]	Pale yellow	75-76	Actone [c]
[Hg(S_2CO-3-MeBu)2]	Pale yellow	65-66	Acetonitrile /ether [b]
[Hg(S_2CO-3,3-Me_2Bu)2]	Pale yellow	118-119	CHCl3/Acetone [a]
[Hg(S_2COnPent)2]	Pale yellow	63-64	CH_2Cl_2/ Acetonitrile [a]
[Hg(S_2CN(Me)2)2]	Pale yellow	262-263	CH_2Cl_2
[Hg(S_2CN(nBu)2)2]	Pale yellow	59-60	Acetonitrile
[Hg(S_2CN(iBu)2)2]	Pale yellow	87-88	DMF
[Hg(S_2CN(Et)Cy)2]	Pale yellow	160-162	Acetone/iPrOH [a]
[Hg(S_2CN(iPr)Cy)2]	Pale yellow	172-173	Acetone/iPrOH [a]

[a] Mixture of the two solvents was in a ratio of (1/1).

[b] Vapour diffusion method used with compound dissolved in first solvent and vapour of second solvent diffused into mixture.

[c] The solution was left in the fridge.

dec. The compound decomposed.

dithiolate salt in aqueous solution. The recovered precipitate was recrystallised from solutions of the complexes, listed in Table 2.1. Although a large number of compounds were synthesized, only those resulting in crystals suitable for X-ray diffraction are reported, with physical characteristics collected in Table 2.1.

2.2.4 *Preparation of bis (xanthate/dithiocarbamate)Te(II) compounds*

The compounds were prepared by employing a literature method (Hoskins, Tiekink, Winter, 1985). This method involved the 1:2 reaction between TeO_2 and the respective dithiolate salt in aqueous solution. The TeO_2 was first dissolved in a minimum quantity of warm HCl before the dithiolate salt was added to the stirred solution. The complex was separated as soon as possible from the acidified solution using either $CHCl_3$ or CH_2Cl_2. This solution was evaporated which resulted in a red oil or solid which was then recystallised. As with other metal complexes only those compounds that afforded crystals suitable for X-ray diffraction are reported, and the physical characteristics are listed in Table 2.1.

2.3 SPECTROSCOPIC CHARACTERISATION

Despite the fact that several of these ligands and complexes have been reported previously, characterisation of crystals was conducted prior to X-ray crystal structure analysis, to ensure the correct compound was synthesised, and to explore the possibility of a comparison between the solution phase and the solid state. To characterize the compound infrared (IR) data (section 2.3.1) and ^{1}H, ^{13}C nuclear magnetic resonance (NMR) data (section 2.3.2) was collected. The spectroscopic data ensures a pure compound and identifies the respective parts of the compound.

2.3.1 *IR spectroscopy*

Xanthates

The spectroscopic data of numerous xanthate ligands and complexes is available in the literature. Table 2.2 summarizes the IR spectroscopic data of the xanthate complexes with two strong absorptions which are characteristic of xanthate ligands, found in the regions 1296 – 1182 cm^{-1} and 1083 – 1010 cm^{-1} and these are assigned to υ(C-O) and υ(C=S), respectively (Winter, 1980).

Dithiocarbamates

The dithiocarbamate ligand is similar to the xanthate ligand in that it contains two strong absorption bands that are sensitive to the nature of the metal to ligand coordination and these are in the regions 1503 – 1441 cm^{-1} and 1036 – 950 cm^{-1}. These bands are assigned as υ(C-N) stretch and the υ(C-S) stretch, respectively (Brown, Glass, Burke, 1976). All complexes possess absorption bands in these regions and are listed in Table 2.3.

In summary the IR spectroscopic data identifies the presence of the functional group S_2CO or S_2CN and the complexation of the ligand to the metal atom due to the shift in their characteristic absorption bands.

2.3.2 *NMR spectroscopy*

The ^{13}C NMR spectra of the xanthate and dithiocarbamate compounds are summarised in Tables 2.6 and 2.7, respectively and similar to the proton NMR spectra the carbon atoms resonate in the expected positions. The chemical shifts of the quaternary carbon (Cq) are moved upfield relative to the free ligand, suggesting a decrease in the delocalisation of the electrons in the S_2CO or S_2CN chromophore. The

Table 2.2 IR spectroscopic data for the xanthate complexes.

Compound	υ(C - O)	υ(C - S)
[Ni(S_2COMe)$_2$]	1236(s)	1041(s)
[Ni(S_2COiPr)$_2$]	1278(s)	1083(s)
[Ni(S_2COnPr)$_2$]	1280(m) 1267(s)	1043(s)
[Ni(S_2COnBu)$_2$]	1277(s) 1264(s)	1034(s) 1017(s)
[Ni(S_2COiBu)$_2$]	1268(s)	1043(s)
[Ni(S_2CO-3-MeBu)$_2$]	1280(s) 1271(s)	1028(m)
[Ni(S_2CO-3,3-Me_2Bu)$_2$]	1281(s) 1253(s)	1025(m)
[Ni(S_2COHex)$_2$]	1296(m) 1268(s)	1044(m) 1023(s)
[Ni(S_2COCy)$_2$]	1272(s) 1263(s) 1243(s)	1041(s)
[Te(S_2COnPr)$_2$]	1220(s)	1035(s)
[Te(S_2COiBu)$_2$]	1223(s)	1038(s)
[Te(S_2CO-3,3-Me_2Bu)$_2$]	1214(s)	1032(s) 1040(m)
[Te(S_2COCH$_2$Ph)$_2$]	1232(s) 1224(s) 1206(s)	1041(s)
[Zn(S_2COnBu)$_2$]	1206(s)	1043(s)
[Hg(S_2COnBu)$_2$]	1198(s)	1033(s) 1062(m)
[Hg(S_2CO-3-MeBu)$_2$]	1202(m)	1027(s)
[Hg(S_2CO-3,3-Me_2Bu)$_2$]	1197(s) 1182(s)	1027(s)
[Hg(S_2COnPent)$_2$]	1200(s)	1010(s)

s strong; m medium; w weak.

Table 2.3 IR spectroscopic data for the dithiocarbamate compounds.

Compound	υ(C - N)	υ(C - S)
[Ni(S_2CNCy_2)2]	1479(s)	1036(m)
[Ni($S_2CN(Et)Cy$)2]	1486(s)	1007(w)
[Te(S_2CNCy_2)2]	1449(s) 1468(m)	998(m)
[Zn(S_2CNCy_2)2]	1476(s)	998(w)
[Zn($S_2CN(Et)Cy$)2]	1484(s)	997(m)
[Cd(S_2CNiPr_2)2]	1480(m) 1463(m)	1035(m)
[Cd(S_2CNiBu_2)2]	1488(s) 1466(s)	979(m)
[Cd(S_2CNCy_2)2]	1473(m) 1442(s)	998(m)
[Cd($S_2CN(Et)Cy$)2]	1485(s)	1011(m)
[Hg(S_2CNMe_2)2]	1503(s)	964(s)
[Hg(S_2CNiBu_2)2]	1490(s)	974(m)
[Hg(S_2CNnBu_2)2]	1491(s)	950(m)
[Hg($S_2CN(Et)Cy$)2]	1479(s)	1011(m)
[Hg($S_2CN(iPr)Cy$)2]	1478(s) 1441(s)	1002(m)

s strong; m medium; w weak.

Table 2.4 ^{1}H NMR data for $M(S_2COR)_2$ M = Ni, Te, Zn, Cd and Hg[a].

Compound	H1	H2	H3	H4	H5	H6
$[Ni(S_2COMe)_2]$	4.13, s					
$[Ni(S_2COnPr)_2]$	4.43, t (6.58)	1.81, m (7.04)	0.99, t (7.40)			
$[Ni(S_2COiPr)_2]$	5.43, m (6.15)	1.44, d (6.40)				
$[Ni(S_2COnBu)_2]$	4.50, t (6.54)	1.80, m (7.00)	1.45, m (7.42)	0.96, t (7.36)		
$[Ni(S_2COiBu)_2]$	4.28, d (6.60)	2.15, m (6.60)	1.01, d (6.60)			
$[Ni(S_2CO\text{-}3\text{-}MeBu)_2]$	4.53, t (6.35)	1.81 - 1.56, m		0.95, d (5.96)		
$[Ni(S_2CO\text{-}3,3\text{-}Me_2Bu)_2]$	4.56, t (7.31)	1.75, t (7.28)	0.96, s			
$[Ni(S_2COCy)_2]$	5.19, m (6.30)		1.97 - 1.25, m			
$[Ni(S_2COHex)_2]$	4.47, t (6.40)	1.78, m (7.20)		1.5 - 1.2, m		0.88, t (6.60)
$[Te(S_2COnPr)_2]$	4.55, t (6.61)	1.88, m (7.10)	1.03, t (7.38)			
$[Te(S_2COiBu)_2]$	4.37, d (6.60)	2.19, m (6.70)	1.03, d (6.70)			
$[Te(S_2CO\text{-}3,3\text{-}Me_2Bu)_2]$	4.66, t (7.40)	1.78, t (7.40)	0.98, s			
$[Te(S_2COCH_2Ph)_2]$	5.61, s		7.23 - 7.41, m			

Table 2.4 Cont.

Compound	H1	H2	H3	H4	H5	H6
[Zn(S_2COnBu)$_2$]	4.51, t (6.60)	1.83, m (7.05)	1.47, m (7.50)	0.97, t (7.35)		
[Hg(S_2COnBu)$_2$]	4.47, t (6.45)	1.80, m (7.05)	1.45, m (7.50)	0.97, t (7.35)		
[HgS_2CO (nPent)$_2$]	4.46, t (6.60)	1.82, m (6.80)	1.43 - 1.36, m		0.93, t (4.60)	
[Hg(S_2CO-3-MeBu)$_2$]	4.50, t (6.45)	1.82 - 1.67, m		0.96, d (6.60)		
[Hg(S_2CO-3,3-Me$_2$Bu)$_2$]	4.53, t (7.20)	1.76, t (7.35)	0.97, s			

a coupling constants are in parentheses: ^{3}J(HH)

br = broad, d = doublet, m = multiplet, s = singlet, t = triplet.

H1 corresponds to the hydrogen atoms nearest the oxygen atom with numbering of the hydrogen atoms increasing further away from the oxygen atom.

Table 2.5 ^{1}H NMR data for $M(S_2CNR_2)_2$ M = Ni, Te, Zn, Cd, and Hg[a].

Compound	H1	H2	H3	H4	H_α	H_β:H_δ
$[Ni(S_2CNCy_2)_2]$					5.19, br	1.83 - 1.07
$[Ni(S_2CN(Et)Cy)_2]$	3.55, q (7.20)	1.26, t (6.90)			4.37, br	1.85 - 1.09
$[Te(S_2CNCy_2)_2]$					5.01, br	1.85 - 1.20
$[Zn(S_2CNCy_2)_2]$					4.98, br	1.85 - 1.06
$[Zn(S_2CN(Et)Cy)_2]$	3.73, q (6.90)	1.20, t (7.20)			4.83, br	1.74 - 1.06
$[Cd(S_2CNiPr_2)_2]$	3.38, m (6.30)	1.43, d (6.30)				
$[Cd(S_2CNiBu_2)_2]$	3.73, d (7.60)	2.49, m (6.90)	0.96, d (6.80)			
$[Cd(S_2CNCy_2)_2]$					5.12, br	1.87 - 0.99
$[Cd(S_2CN(Et)Cy)_2]$	3.75, br	1.20, br			4.88, br	1.79 - 1.20
$[Hg(S_2CNMe_2)_2]$	3.48, s					
$[Hg(S_2CNnBu_2)_2]$	3.72, t (7.65)	1.78, m (7.50)	1.35, m (7.50)	0.95, t (7.35)		
$[Hg(S_2CNiBu_2)_2]$	3.61, d (7.60)	2.45, m (6.95)	0.97, s (6.60)			

Table 2.5 Cont.

Compound	H1	H2	H3	H4	H_α	H_β - H_δ
$[Hg(S_2CN(Et)Cy)_2]$	3.79, q (7.07)	1.35, t (7.20)			4.67, br	1.99 - 1.25
$[Hg(S_2CN(iPr)Cy)_2]$	3.92, br	1.60, br			4.76, br	1.86 - 1.14

a coupling constants are in parentheses: $^3J(HH)$

br = broad, d = doublet, m = multiplet, s = singlet, t = triplet.

H1 corresponds to the hydrogen atoms nearest the nitrogen atom with the numbering of the hydrogen atoms increasing further away from the nitrogen atom.

H atoms with greek letters identify the Cy group.

Table 2.6 ^{13}C NMR data for $M(S_2COR)_2$, M=Ni, Te, Zn, Cd, Hg[a].

Compound	C_q	C1	C2	C3	C4	C5	C6
$[Ni(S_2COMe)_2]$	232.43	57.96					
$[Ni(S_2COnPr)_2]$	231.48	73.56	21.59	10.16			
$[Ni(S_2COiPr)_2]$	230.36	77.97	21.54				
$[Ni(S_2COnBu)_2]$	231.37	72.25	30.05	18.87	13.54		
$[Ni(S_2COiBu)_2]$	231.54	78.04	27.62	18.76			
$[Ni(S_2CO\text{-}3\text{-}MeBu)_2]$	230.93	71.08	36.61	24.79	22.29		
$[Ni(S_2CO\text{-}3,3\text{-}Me_2Bu)_2]$	231.26	70.32	41.05	29.82	29.46		
$[Ni(S_2COHex)_2]$	231.54	72.56	31.21	28.05	25.24	22.40	13.89
$[Ni(S_2COCy)_2]$	230.36	82.65	31.11	24.86	23.30		
$[Te(S_2COnPr)_2]$	215.76	77.42	21.83	10.31			
$[Te(S_2COiBu)_2]$	215.71	81.79	27.84	18.93			
$[Te(S_2CO\text{-}3,3\text{-}Me_2Bu)_2]$	215.69	73.65	41.37	29.85	29.54		
$[Te(S_2COCH_2Ph)_2]$	n.o.	77.18	137.39	128.69	127.42	129.40	

Table 2.6 Cont.

Compound	C_q	C1	C2	C3	C4	C5	C6
[Zn(S_2COnBu)$_2$]	230.75	78.43	30.34	18.93	13.53		
[Hg(S_2COnBu)$_2$]	222.66	77.82	30.29	19.04	13.60		
[Hg(S_2COnPent)$_2$]	222.45	78.09	28.02	27.94	22.25	13.85	
[Hg(S_2CO-3-MeBu)$_2$]	222.34	76.62	36.90	25.02	22.37		
[Hg(S_2CO-3,3-Me$_2$Bu)$_2$]	222.33	75.90	41.26	29.87	29.56		

a Cq is the quaternary carbon. The carbon atoms are numbered with C1 coordinated to the oxygen atom and the numbering of the carbon atoms increasing further away from the oxygen atom.

n.o. not observed.

Table 2.7 ^{13}C NMR data for $M(S_2CNR_2)_2$, M = Ni, Te, Zn, Cd, Hg[a].

Compound	C_q	C1	C2	C3	C4	C_α	C_β
$[Ni(S_2CNCy_2)_2]$	206.14	60.23	29.59	25.95	25.03		
$[Ni(S_2CN(Et)Cy)_2]$	206.38	58.87	30.24	25.46	25.19	40.53	14.33
$[Te(S_2CNCy_2)_2]$	193.74	53.89	29.27	25.13	24.73		
$[Zn(S_2CNCy_2)_2]$	201.98	53.22	29.57	25.80	25.11		
$[Zn(S_2CN(Et)Cy)_2]$ *	203.17	43.29	29.84	25.33	24.68	63.58	13.97
$[Cd(S_2CNiPr_2)_2]$ *	n.o.	45.81	19.28				
$[Cd(S_2CNiBu_2)_2]$ *	204.88	64.43	27.00	20.34			
$[Cd(S_2CNCy_2)_2]$ *	n.o.	52.15	32.69	25.48	24.40		
$[Cd(S_2CN(Et)Cy)_2]$ *	204.14	43.83	29.89	25.35	24.71	64.49	13.99
$[Hg(S_2CNMe_2)_2]$ *	n.o.	46.66					
$[Hg(S_2CNnBu_2)_2]$	203.50	56.88	28.89	20.01	13.69		
$[Hg(S_2CNiBu_2)_2]$	205.26	64.52	27.03	20.21			

Table 2.7 Cont.

Compound	C_q	C1	C2	C3	C4	C_α	C_β
$[Hg(S_2CN(Et)Cy)_2]$	201.92	45.55	30.65	25.53	25.22	67.31	13.94
$[Hg(S_2CN(iPr)Cy)_2]$	203.48	52.41	29.67	25.64	25.15	60.61	19.86

* spectra recorded in deuterated DMSO.

a Cq is the quaternary carbon. The carbon atoms are numbered with C1 coordinated to the nitogen atom and the numbering of carbons increases further away from the nitogen atom. Carbon atoms with greek letters correspond to the Et/iPr carbons in the ligand.

spectra suggest fluxionality of the ligands on the time scale of the NMR experiment with equivalent carbon atoms resonating together resulting in clean spectra.

Similar to the IR spectroscopy, NMR spectroscopy identifies the presence of the ligands by characteristic resonances, and the complexation of the ligand to the metal atom is identified due to the movement of these resonances upfield with respect to the free ligand. The spectra display equivalence of carbons and protons of the ligands with equivalent carbons and protons resonating together, implying that on the time scale of the NMR experiment the ligands are free to rotate in the solution phase.

Chapter 3

NICKEL(II) BIS 1,1-DITHIOLATES

In this chapter the crystal and molecular structures of nine new $Ni(S_2COR)_2$ and two new $Ni(S_2CNR_2)_2$ complexes are reported and their structural characteristics described. The derived interatomic parameters of the new structures are then compared with literature structures in order to make an assessment of the coordinating ability of the xanthate and dithiocarbamate ligands in their respective nickel(II) complexes.

3.1 XANTHATE STRUCTURES

The molecular structure of $[Ni(S_2COMe)_2]$ is shown in Fig. 3.1 and selected interatomic parameters for this structure and the other xanthate complexes described herein are given in Table 3.1. The four sulfur atoms adopt an essentially square planar geometry around the central nickel atom with the nickel atom lying 0.091(1) Å above the least-squares plane through the S_4 donor set (mean deviation: 0.042 Å). The bidentate coordination of the methylxanthate ligands lead to the formation of two four membered rings with the nickel atom at the apex of each. Distortions from the ideal geometry may be traced to the restricted bite angle of the xanthate ligand which leads to acute S-Ni-S chelate angles of 79.24(7)° and 79.34(7)°, respectively. The two ligands may be considered as forming essentially equivalent Ni-S bonds with the Ni-S separations being experimentally equivalent. There is a close intermolecular contact in the lattice that occurs between the nickel and symmetry related S(1) atom of 3.167(2) Å (symmetry operation 1-*x*, -*y*, 1-*z*). This contact is within the sum of the van der Waals radii for nickel and sulfur of 3.4 Å (Bondi, 1964), however it does not appear to influence significantly the length of intramolecular Ni-S interactions. Reflecting the equivalence in the Ni-S distances, the C-S bond distances are equal within experimental error at 1.685(7) Å and 1.712(6) Å, respectively. The C(1)-O(1) and C(3)-O(2) separations of 1.308(7) Å and 1.297(7) Å, respectively are shorter than the O(1)-C(2) and O(2)-C(4) separations of 1.444(8) Å and

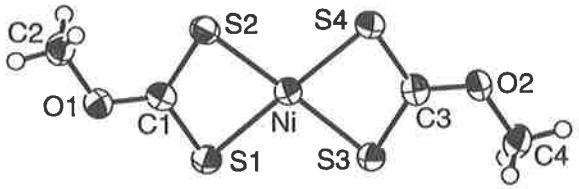


Fig. 3.1 Molecular structure of $[Ni(S_2COMe)_2]$ showing the crystallographic numbering scheme.

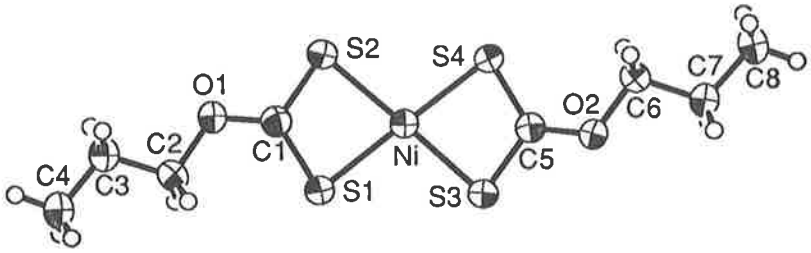


Fig. 3.2 Molecular structure of $[Ni(S_2COnPr)_2]$ showing the crystallographic numbering scheme.

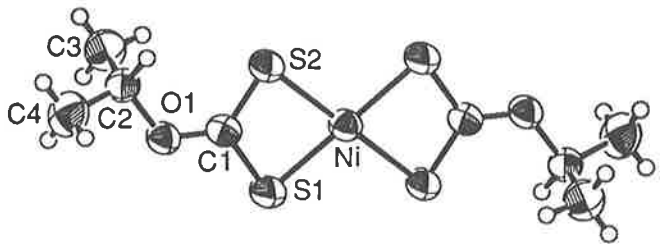


Fig. 3.3 Molecular structure of $[Ni(S_2COiPr)_2]$ showing the crystallographic numbering scheme.

Table 3.1 Selected bond distances (Å) and bond angles (deg.) for $Ni(S_2COR)_2$

R	Ni-S(1)	Ni-S(2)	av. Ni-S	Ni···S[a]	S(1)-C	S(2)-C	C-O	S-Ni-S	S-C-S
Me [b]	2.209(2)	2.221(2)	2.218(1)	3.167(2)	1.702(6)	1.678(6)	1.308(7)	79.24(7)	113.4(3)
	2.220(2)	2.221(2)			1.712(6)	1.685(7)	1.297(7)	79.34(7)	113.1(3)
Et [c]	2.210(2)	2.212(2)	2.211(1)	3.448(2)	1.696(5)	1.679(5)	1.306(6)	79.4(6)	113.6(3)
CH_2CF_3 [d]	2.218(4)	2.223(5)	2.214(2)	3.42(1)	1.71(2)	1.68(2)	1.29(2)	79.0(2)	114(1)
	2.203(5)	2.211(4)			1.70(1)	1.69(1)	1.31(2)	79.2(2)	113(1)
nPr [b]	2.204(1)	2.223(1)	2.214(1)	3.275(1)	1.694(3)	1.682(3)	1.308(3)	79.09(3)	113.2(2)
	2.215(1)	2.215(1)			1.689(3)	1.688(3)	1.303(3)	79.20(3)	113.5(2)
iPr [b]	2.199(2)	2.212(2)	2.206(1)	3.563(9)	1.697(6)	1.683(6)	1.307(6)	79.28(6)	112.7(3)
CH_2CH_2OMe [e]	2.2128(7)	2.2146(8)	2.2136(5)	3.4975(6)	1.685(3)	1.698(3)	1.302(3)	79.42(3)	113.5(1)

Table 3.1 Cont.

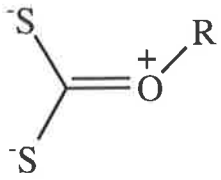
R	Ni-S(1)	Ni-S(2)	av. Ni-S	Ni···S[a]	S(1)-C	S(2)-C	C-O	S-Ni-S	S-C-S
nBu [b]	2.1998(9)	2.2136(9)	2.2067(6)	4.5246(9)	1.687(3)	1.688(3)	1.307(3)	79.48(3)	113.5(2)
iBu [b]	2.202(1)	2.215(1)	2.2079(7)	4.667(1)	1.688(3)	1.694(3)	1.306(4)	76.69(3)	113.6(2)
3-MeBu [b]	2.203(1)	2.205(2)	2.2034(9)	4.890(1)	1.663(6)	1.688(6)	1.309(5)	79.44(6)	114.4(3)
3,3-Me_2Bu [b]	2.2026(8)	2.2155(8)	2.2091(6)	4.7439(6)	1.697(3)	1.687(3)	1.308(3)	79.46(3)	113.1(2)
Hex [b]	2.204(1)	2.2169(9)	2.2111(6)	5.0616(8)	1.693(4)	1.689(4)	1.304(4)	79.52(4)	113.4(2)
Cy [b]	2.2101(8)	2.2241(8)	2.2160(4)	3.7884(2)	1.697(3)	1.690(3)	1.311(3)	79.22(3)	113.2(2)
	2.2118(8)	2.2182(8)			1.704(3)	1.689(3)	1.305(3)	79.09(3)	112.4(2)
CH_2Ph [f]	2.2035(5)	2.2201(7)	2.2091(4)	3.401(2)	1.693(2)	1.687(3)	1.305(5)	79.39(2)	113.5(2)
4-MeCy [g]	2.205(1)	2.205(1)	2.2050(7)	4.8091(6)	1.692(3)	1.696(3)	1.296(4)	79.46(4)	112.7(2)

Table 3.1 Cont.

R	Ni-S(1)	Ni-S(2)	av. Ni-S	Ni···S[a]	S(1)-C	S(2)-C	C-O	S-Ni-S	S-C-S
Ph-4-tBu [h]	2.213(6)	2.219(4)	2.214(6)	4.128(2)	1.699(19)	1.670(27)	1.318(19)	79.1(2)	113.7(10)
	2.205(5)	2.222(7)			1.687(30)	1.682(26)	1.323(18)	79.4(2)	114.4(11)

a shortest intermolecular Ni···S contact; b this work; c orthorhombic form (Hoskins, Tiekink, Winter, 1985b); d Romanenko, Podberezskaya, Bakakin, 1987; e Abrahams, Hoskins, Tiekink, Winter, 1988; f Edwards, Hoskins, Winter, 1986; g Xiong, Liu, Li, You, Huang, 1996; h Chen, Fackler, 1978.

1.442(8) Å, respectively and are consistent with the significant contribution of canonical structure (I) in the planar portions of the xanthate ligands in $[Ni(S_2COMe)_2]$.



(I)

The remaining xanthate structures adopt the same motif as that found for $[Ni(S_2COMe)_2]$ and hence, their description will be brief.

The molecular structure of $[Ni(S_2COnPr)_2]$ is illustrated in Fig. 3.2 depicting the molecular structure as adopting the same square planar arrangement as observed for the above mentioned methyl derivative. Similarly to the methyl derivative there exists a close intermolecular contact between the nickel and S(1) atoms (symmetry operation $-x$, $1-y$, $-z$) of 3.275(1) Å. This results in an elongation of one of the Ni-S bonds and as a consequence the ligand coordinates to the nickel atom in an asymmetric fashion. However it is worth noting that the average Ni-S separations of the asymmetric ligand (caused by the close intermolecular contact) of 2.214(1) Å is equivalent to the average Ni-S distances of the second xanthate ligand. The features associated with xanthate ligands are similar to those described for the methyl derivative.

The structure of $[Ni(S_2COiPr)_2]$ is manifested in Fig. 3.3. The structure is molecular with the closest non-hydrogen contact of 3.563(9) Å occurring between the O(1) and C(3)" atoms (symmetry operation: $-1-x$, $-0.5+y$, $-0.5-z$); there are no close intermolecular Ni···S interactions in the lattice. The molecule is centrosymmetric with the nickel atom situated on a crystallographic centre of inversion and the xanthate ligand forms Ni-S distances of 2.199(2) Å and 2.212(2) Å which are equivalent to the 4σ level. The features associated with the xanthate ligand are similar to those described for the methyl structure.

The molecular structure of [Ni(S_2COnBu)$_2$] is illustrated in Fig. 3.4. The molecule is centrosymmetric with the nickel atom situated on a crystallographic centre of inversion and the xanthate ligand forms Ni-S distances of 2.1998(9) Å and 2.2136(9) Å. The Ni-S separations are significantly different and unlike previous structures that displayed disparity in bond lengths *i.e.* R = methyl and n-propyl, there exists no significant intermolecular interactions with the closest non-hydrogen contact between atoms S(1) and C(4) being 3.660(4) Å (symmetry operation: $+x$, $+y$, $+z$). The features associated with the xanthate ligand are similar to those described in previous structures.

The molecular structure of [Ni(S_2COiBu)$_2$] is illustrated in Fig. 3.5 which depicts a centrosymmetric square planar arrangement of four sulfur atoms about the nickel atom. The Ni-S bond distances of 2.202(1) Å and 2.215(1) Å are marginally different although the general features of the xanthate ligand remain similar to those described above. There are no significant intermolecular contacts in the lattice with the closest non-hydrogen distance of 3.552(6) Å occurring between the O(1) and C(5) atoms (symmetry operation: $-x$, $-1-y$, $1-z$).

Fig. 3.6 shows the molecular structure of [Ni(S_2CO-3-MeBu)$_2$] in which the nickel atom is situated on a crystallographic inversion centre. The Ni-S separations are equivalent within experimental error, 2.203(1) Å and 2.205(2) Å and the xanthate ligand exhibits similar features as described in previous structures. The closest non-hydrogen contact of 3.58(1) Å (symmetry operation; $+x$, $+y$, $+z$) is between atoms O(1) and C(6) with no significant Ni-S interactions.

The structure of [Ni(S_2CO-3,3-Me$_2$Bu)$_2$] is depicted in Fig 3.7 and the centrosymmetric molecule has nickel at a crystallographic centre of inversion. The xanthate ligand features are similar to those described for previous structures and the two Ni-S bond distances are marginally inequivalent being 2.204(1) Å and 2.2168(8) Å. There are no significant intermolecular interactions existing in the molecule, the closest non-hydrogen contact being 3.546(3) Å that occurs between the atoms S(2) and C(2)' (symmetry operation; $-x$, $-y$, $-z$).

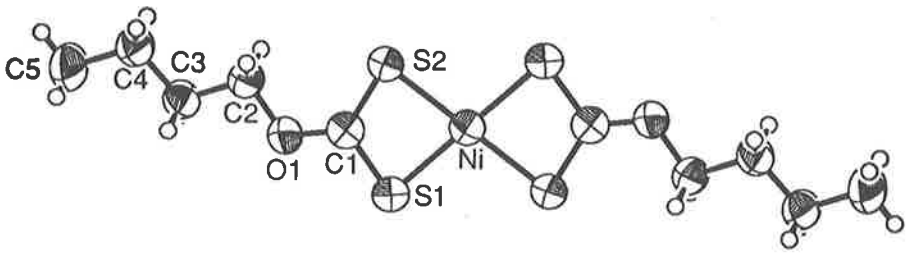


Fig. 3.4 Molecular structure of [Ni(S_2COnBu)$_2$] showing the crystallographic numbering scheme.

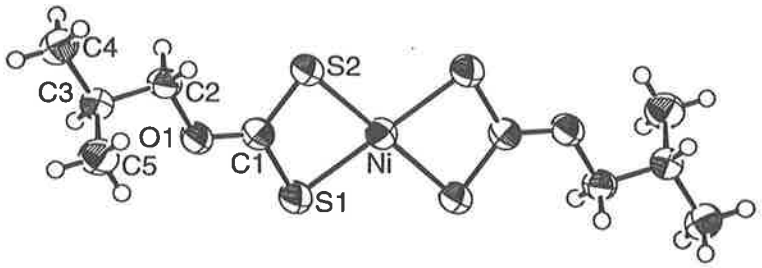


Fig. 3.5 Molecular structure of [Ni(S_2COiBu)$_2$] showing the crystallographic numbering scheme.

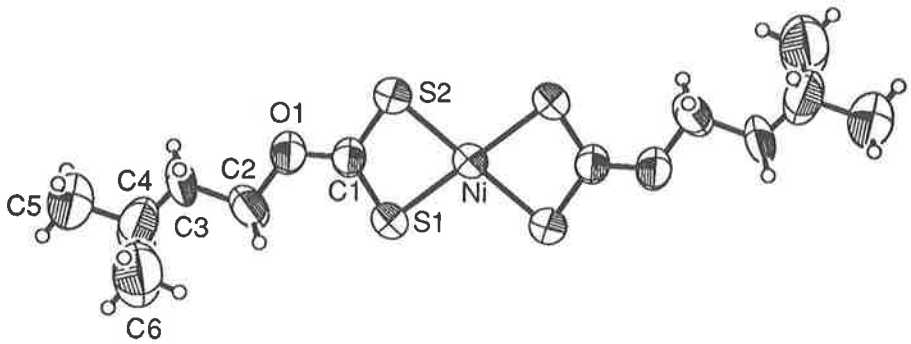


Fig. 3.6 Molecular structure of [Ni(S_2CO-3-MeBu)$_2$] showing the crystallographic numbering scheme.

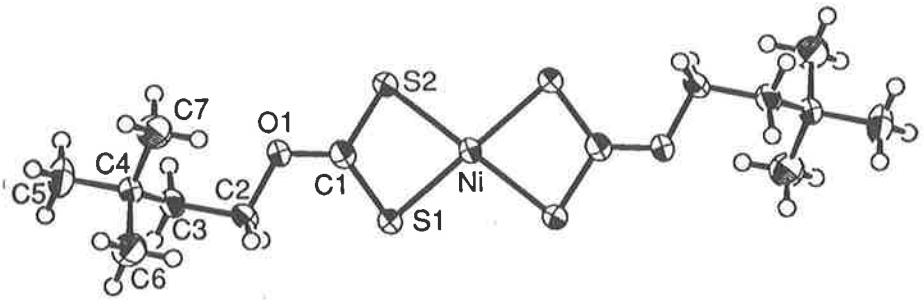


Fig. 3.7 Molecular structure of [Ni(S_2CO-3,3-Me_2Bu)$_2$] showing the crystallographic numbering scheme.

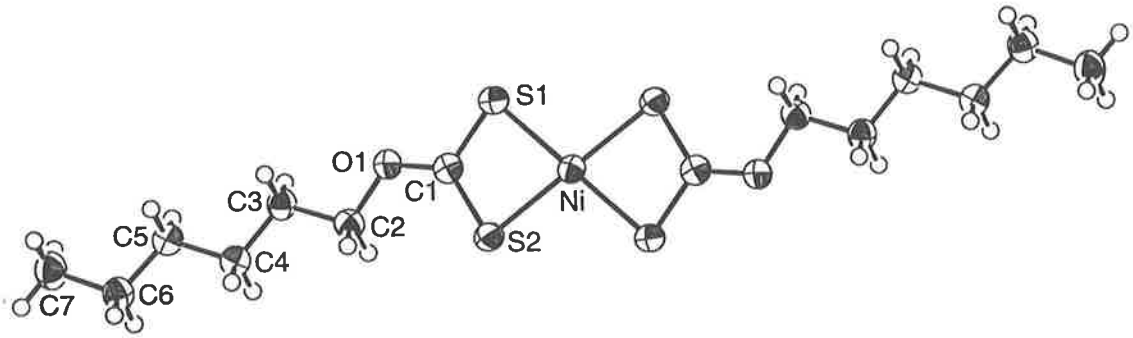


Fig. 3.8 Molecular structure of [Ni(S_2COHex)$_2$] showing the crystallographic numbering scheme.

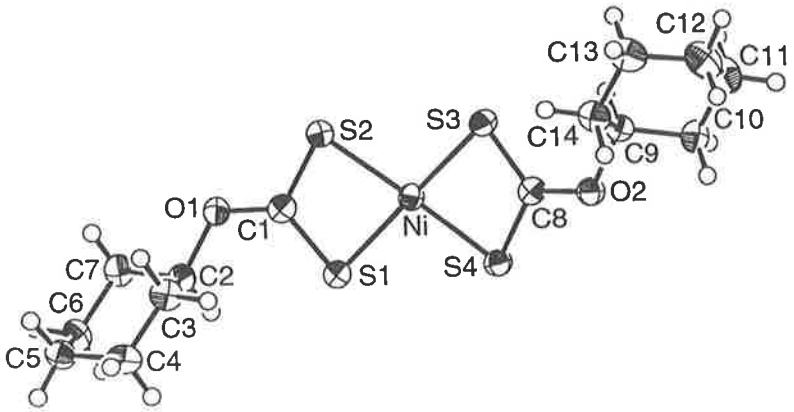


Fig. 3.9 Molecular structure of [Ni(S_2COCy)$_2$] showing the crystallographic numbering scheme.

Fig. 3.8 depicts the molecular structure of [Ni(S_2COHex)$_2$]. The nickel atom is situated at a crystallographic centre of inversion with the independent Ni-S separations displaying a slight variance with values of 2.204(1) Å and 2.2169(9) Å. There are no significant intermolecular interactions involving the non-H atoms less than 3.6 Å.

The molecular structure of [Ni(S_2COCy)$_2$] is illustrated in Fig. 3.9. The nickel atom lies 0.1410(4) Å above the least-squares plane (mean deviation: 0.015 Å) through the S_4 donor set. There exists a slight disparity in the Ni-S separations, *i.e.* Ni-S(1, 4) 2.2182(8) Å, 2.2241(8) Å, 2.2118(8) Å and 2.2180(9) Å, respectively. An intermolecular contact between the Ni and S(3)' atoms of 3.076(3) Å is noted (symmetry operation: $-x$, $1-y$, $-z$).

3.2 DITHIOCARBAMATE STRUCTURES

The molecular structures of the two independent centrosymmetric molecules of [Ni(S_2CNCy$_2$)$_2$] are illustrated in Fig. 3.10. As with the nickel xanthates described above, the four sulfur atoms adopt a square planar geometry around the central nickel atom. The Ni-S bond separations of 2.182(1) Å, 2.203(1) Å and 2.184(2) Å and 2.196(1) Å for the two independent molecules, respectively indicate a negligible difference between the coordination geometries, the S-Ni-S chelate angles are 79.06(5)° and 79.06(6)°, respectively. There are no intermolecular contacts involving non-H atoms in the lattice less than 3.6 Å. The dithiocarbamate ligands have features similar to the xanthate ligands described above with the C-S bond lengths being equal within experimental error (1.734(5) Å, 1.721(5) Å, and 1.717(5) Å, 1.724(5) Å). Further, the experimentally equivalent C(1)-N(1) and C(14)-N(2) separations of 1.320(6) Å and 1.324(6) Å, respectively are shorter than the N(1)-C(2), C(8) and N(2)-C(15), C(21) distances of 1.489(6) Å, 1.488(6) Å, and 1.509(7) Å, 1.439(7) Å, respectively, and are consistent with the significant contribution of canonical form (II) to the overall structure.

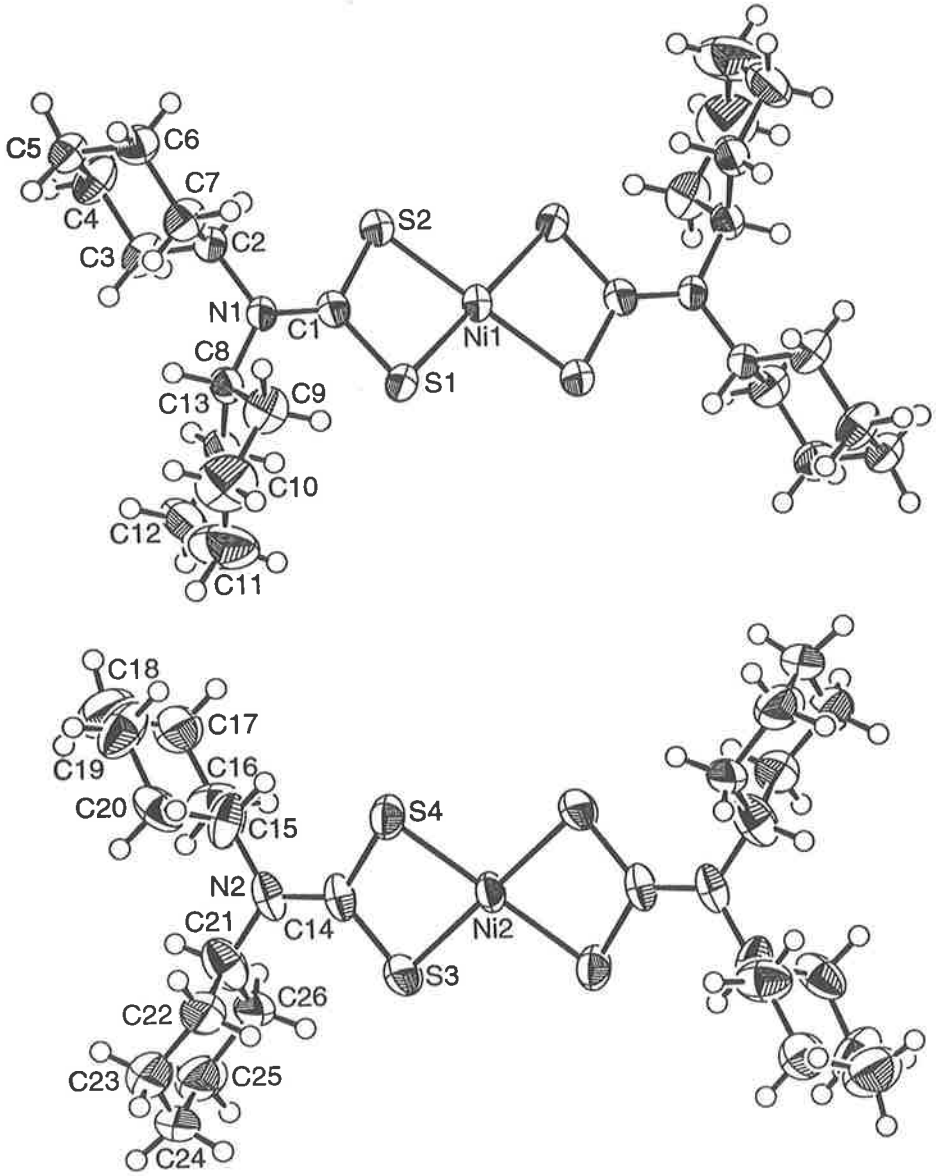


Fig. 3.10 Molecular structure of [Ni(S_2CNCy_2)2] showing the crystallographic numbering scheme.

Table 3.2 Selected bond distances (Å) and bond angles (deg.) for $Ni(S_2CNR_2)_2$

R	Ni-S(1)	Ni-S(2)	Ni-S	S(1)-C	S(2)-C	C-N	S-Ni-S	S-C-S
H [a]	2.207(7)	2.224(15)	2.211(4)	1.70(3)	1.68(2)	1.37(3)	78.6(3)	111.9(11)
	2.210(7)	2.218(15)		1.70(3)	1.68(2)	1.38(3)	78.4(2)	111.6(12)
Me [b]	2.198(2)	2.217(2)	2.208(1)	1.750(8)	1.691(8)	1.349(9)	79.29(7)	109.9(4)
Et [c]	2.196(1)	2.202(1)	2.1990(7)	1.715(3)	1.714(3)	1.324(3)	79.4(1)	110.1(1)
Et [d]	2.192(1)	2.204(1)	2.1980(7)	1.711(3)	1.725(3)	1.319(4)	79.6(1)	110.0(2)
CH_2CF_3 [e]	2.197(2)	2.200(1)	2.1990(9)	1.699(6)	1.699(6)	1.340(7)	79.06(6)	110.9(3)
CH_2CH_2OH [f]	2.193(1)	2.209(1)	2.2010(7)	1.713(4)	1.728(4)	1.319(5)	79.3(1)	109.4(2)
nPr [g]	2.197(2)	2.209(2)	2.203(1)	1.693(8)	1.722(8)	1.330(9)	79.2(1)	110.6(4)
iPr [h]	2.179(3)	2.183(3)	2.181(2)	1.714(12)	1.711(11)	1.329(15)	79.6(1)	109.2(6)
	2.181(3)	2.182(3)		1.720(12)	1.673(12)	1.337(17)	78.9(1)	109.5(7)

Table 3.2 Cont.

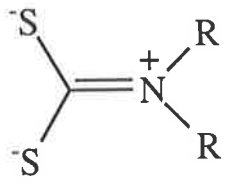
R	Ni-S(1)	Ni-S(2)	Ni-S	S(1)-C	S(2)-C	C-N	S-Ni-S	S-C-S
C_3H_5 [i]	2.198(2)	2.210(2)	2.204(1)	1.712(8)	1.731(8)	1.29(1)	79.2(1)	109.3(4)
nBu [j]	2.201(3)	2.211(3)	2.206(2)	1.720(11)	1.742(11)	1.329(13)	79.4(1)	109.0(6)
iBu [k]	2.193(3)	2.205(3)	2.201(2)	1.718(8)	1.721(9)	1.31(1)	79.3(1)	109.4(5)
	2.203(3)	2.204(7)		1.717(9)	1.720(9)	1.33(1)	79.4(1)	110.0(5)
R_1 [l]	2.198(1)	2.214(1)	2.2060(7)	1.707(4)	1.717(4)	1.311(4)	79.33(5)	110.7(2)
R_2 [m]	2.2063(7)	2.2107(6)	2.2088(5)	1.714(2)	1.712(2)	1.328(3)	79.16(2)	110.4(1)
R_3 [n]	2.196(1)	2.207(1)	2.2015(7)	1.717(4)	1.718(3)	1.327(5)	79.4(1)	110.0(2)
Cy [o]	2.182(1)	2.203(1)	2.1925(7)	1.734(5)	1.721(5)	1.320(6)	79.06(5)	107.8(3)
	2.184(1)	2.196(1)		1.717(5)	1.724(5)	1.324(6)	79.06(6)	108.2(3)
R_4 [p]	2.201(4)	2.211(4)	2.206(3)	1.697(14)	1.718(13)	1.32(1)	79.0(1)	110.5(6)

Table 3.2 Cont.

R	Ni-S(1)	Ni-S(2)	Ni-S	S(1)-C	S(2)-C	C-N	S-Ni-S	S-C-S
R_5 [q]	2.19(1)	2.21(1)	2.200(5)	1.72(3)	1.72(3)	1.35(4)	81.0(4)	111(2)
	2.20(1)	2.20(1)		1.72(3)	1.71(3)	1.35(4)	80.0(4)	112(2)
H, Me [r]	2.196(9)	2.203(9)	2.200(6)	1.72(1)	1.70(1)	1.30(2)	79.2(1)	109.8(7)
H, nPr [s]	2.193(1)	2.202(1)	2.1975(7)	1.711(4)	1.719(4)	1.295(7)	79.92(4)	110.7(3)
Me, Ph [t]	2.198(3)	2.208(3)	2.203(2)	1.70(1)	1.74(1)	1.30(1)	79.3(1)	109.3(6)
R_6, R_7 [u]	2.198(2)	2.199(2)	2.200(1)	1.695(8)	1.694(8)	1.37(1)	78.82(8)	110.9(4)
	2.195(2)	2.206(2)		1.691(9)	1.698(9)	1.38(1)	78.60(8)	110.7(5)
Cy, Et [o]	2.1965(7)	2.197(1)	2.1967(6)	1.725(3)	1.723(3)	1.314(3)	79.58(3)	109.3(1)

Table 3.2 Cont.

a Gasparri, Nardelli, Villa, 1967; b Lokaj, Garaj, Kettmann, Vrábel, 1980; c Selvaraju, Panchanatheswaran, Thiruvalluvar, Parthasarathi, 1995; d beta form (Khan, Fackler, Murray, Heinrich, Campana, 1987); e Sundberg, Riekkola, 1987; f Ramalingam, Radha, Aravamudan, Mahadevan, Subramanyam, Seshasayee, 1984; g Peyronel, Pignedoli, 1967; h Newman, White, 1972a; i Lokaj, Pavelč ík, Kettmann, Masaryk, Vrábel, Garaj, 1981; j Lokaj, Vrábel, Kellö, 1984; k Raston, White, 1976; l NR_1 is pyrrolidine (Engelhardt, Patrick, White, 1985); m NR_2 is morpholine (Herring, Park, Rettig, Trotter, 1979); n NR_3 is 1-piperidine (Radha, Seshasayee, Aravamudan, 1988); o this work; p R_4 is $(CH_2)_5$ (Kettmann, Garaj, Kúdela, 1978); q R_5 is $(CH_2)_6$ (Starikova, Shugam, Agre, Oboznenko, 1972); r Newman, White, 1972b; s Kameníč ek, Pastorek, Bř ezina, Kratochvíl, Trávníč ek, 1990; t Martin, Newman, Robinson, White, 1972; u R_6 is acetyl, R_7 is CH_2Ph (Christidis, 1986).



(II)

The molecular structure of centrosymmetric [Ni(S_2CN(Et)Cy)$_2$], featuring an unsymmetrical dithiocarbamate ligand, is illustrated in Fig. 3.11 which shows the familiar square planar NiS_4 motif. The Ni-S separations are equivalent within experimental error at 2.1969(6) Å and 2.1975(9) Å. As with [Ni(S_2CNCy$_2$)$_2$] there are no significant intermolecular contacts in the lattice less than 3.6 Å.

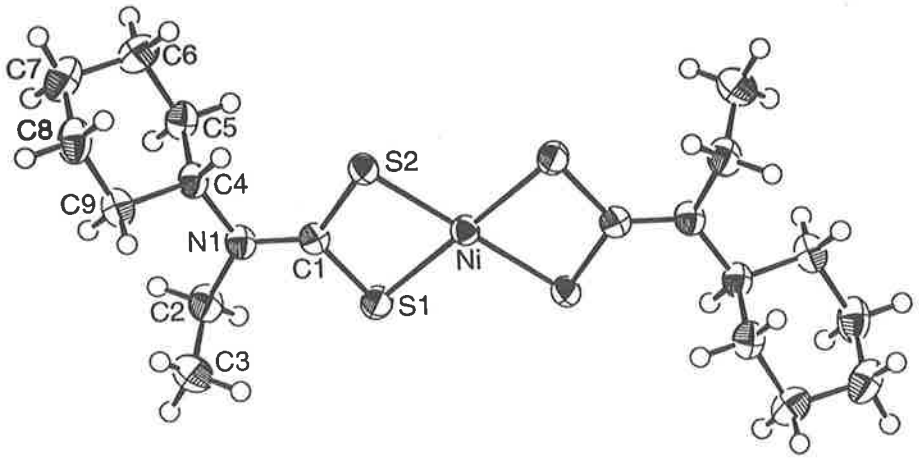


Fig. 3.11 Molecular structure of [Ni(S_2CN(Et)Cy)$_2$] showing the crystallographic numbering scheme.

3.3 SYSTEMATIC TRENDS FROM CRYSTALLOGRAPHY

Selected interatomic parameters for the fifteen Ni(S_2COR)$_2$ structures, including the nine new structures reported herein, are collected in Table 3.1. Invariably, the

structures feature a square planar NiS_4 motif as detailed above. In some of the structures, notably those containing smaller R substituents, there are close intermolecular Ni···S interactions with the shortest such contact of 3.167(2) Å occurring in the structure of $[Ni(S_2COMe)_2]$; see above. The intermolecular Ni···S contacts often do not lead to systematic perturbations in the Ni-S bond distances, *e.g.* $[Ni(S_2COMe)_2]$ *cf.* and $[Ni(S_2COnPr)_2]$. A plot of the Ni-S(1) and Ni-S(2) distances, showing their 3σ error bars, versus the R group is presented in Fig. 3.12; data for two structures, *i.e.* $[Ni(S_2COCH_2CF_3)_2]$ and $[Ni(S_2COPh\text{-}4\text{-}tBu)_2]$, are not included in the plot owing to their high standard uncertainties but their data were used in the calculation of average parameters. The Ni-S distances are plotted in order of increasing Ni-S(1) distance. Several interesting conclusions may be drawn from the data plotted in Fig. 3.12.

Of the fifteen known $Ni(S_2COR)_2$ structures, ten have the nickel atom located at a crystallographic center of inversion. In nine structures, the Ni-S(1) and Ni-S(2) distances are equivalent within experimental error. The Ni-S distances range from 2.199(2) Å in the R = iPr structure to 2.2241(8) Å in the R = Cy structure. This result indicates that the absolute difference between the shortest and longest Ni-S separations in the fifteen structures is only 0.025 Å indicating that the range of Ni-S distances is narrow (see below). Further, most of the individual Ni-S distances are equal within experimental error to the calculated average Ni-S distance for all structures of 2.2107(2) Å and certainly at the 5σ limit. The reason for the small disparity in the Ni-S distances within individual structures can be explained in terms of the intramolecular interactions between the O-bound R groups and the sulfur atoms. Thus, at the resolution of the X-ray experiment, electronic effects due to the different R substituents are not manifested in the mode of coordination of the xanthate ligands.

Given the range of R groups found in the $Ni(S_2COR)_2$ structures, an attempt was made to detect whether any systematic influence on the derived interatomic parameters could be correlated with an electronic effect associated with R. As can be seen from Table 3.1 and Fig. 3.12 there are no obvious trends in the parameters with R as i) the

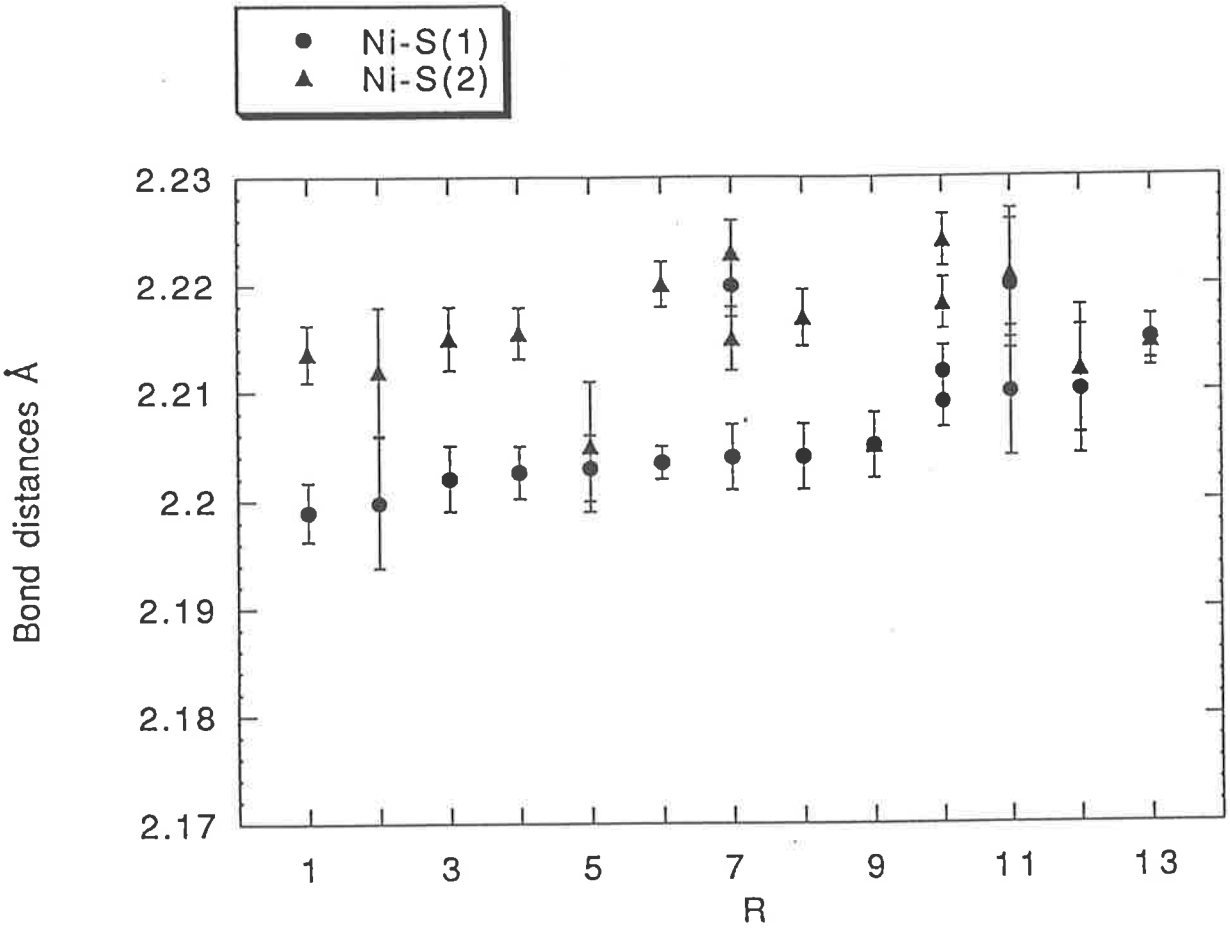


Fig. 3.12 Plot of Ni-S distances (Å) in $Ni(S_2COR)_2$ versus R: **1**, R = 3-MeBu; **2**, R = 4-MeCy; **3**, R = iPr; **4**, R = nBu; **5**, R = iBu; **6**, R = 3,3-Me_2Bu; **7**, R = CH_2Ph; **8**, R = Et; **9**, R = Hex; **10**, R = nPr; **11**, R = CH_2CH_2OMe; **12**, R = Cy; **13**, R = Me.

chain length is varied (*e.g.* R = Me, Et, nPr, nBu), ii) the degree of branching is varied (*e.g.* nPr, iPr; nBu, iBu), and iii) the steric bulk is varied (*e.g.* nBu, 3-MeBu, 3,3-Me_2Bu).

Another parameter worth commenting on is the ligand bite angle. From the data presented in Table 3.1 it can be seen that the S-C-S angles range from 112.4(2)° to 114.4(3)° with an average of 113.35(4)°. Accordingly, the S-Ni-S angles also lie in a narrow range 79.0(2)° to 79.69(3)° with an average value of 79.371(6)°.

The small range of Ni-S bond distances in the $Ni(S_2COR)_2$ structures and the lack of systematic trends indicate that the variations in the electronic/steric profiles of the ligands have little or no effect on the Ni-S distances as resolved by X-ray crystallography. In this context, it is worth mentioning that in addition to the thus far unexplained structural diversity alluded to in the *Introduction*, systematic analysis of certain transition metal complexes characterised in different crystalline environments show that variations in metal-ligand parameters can exceed the normally accepted experimental errors (Martín, Orpen, 1996; Cotton, Yokochi, 1997). Further, combined crystallographic and theoretical studies demonstrate that unexpected variations in Sn-ligand parameters may be explained by the influence of intermolecular forces, *i.e.* crystal packing effects, on molecular geometry (*e.g.* Buntine, Hall, Kosovel, Tiekink, 1998; Tiekink, Hall, Hook, Buntine, 1998, Buntine, Hall, Tiekink, 1998). As for the $Ni(S_2COR)_2$ complexes, a similar systematic examination of coordination geometry was conducted for the related $Ni(S_2CNR_2)_2$ complexes.

Selected interatomic parameters for the $Ni(S_2CNR_2)_2$ structures are collected in Table 3.2. Reflecting the trend in molecular symmetry found for the xanthate structures, of the twenty two $Ni(S_2CNR_2)_2$ structures listed, sixteen have crystallographic $\bar{1}$ symmetry. By contrast to several $Ni(S_2COR)_2$ structures, no Ni···S intermolecular contacts less than 3.6 Å are observed in the dithiocarbamate analogs. Fig. 3.13 presents a plot of the Ni-S(1) and Ni-S(2) distances against R as for Fig. 3.12; four structures (*i.e.* $[Ni(S_2CNH_2)_2]$, $[Ni(S_2CN(CH_2)_5)_2]$, $[Ni(S_2CN(CH_2)_6)_2]$ and $[Ni(S_2CN(H)Me)_2]$)

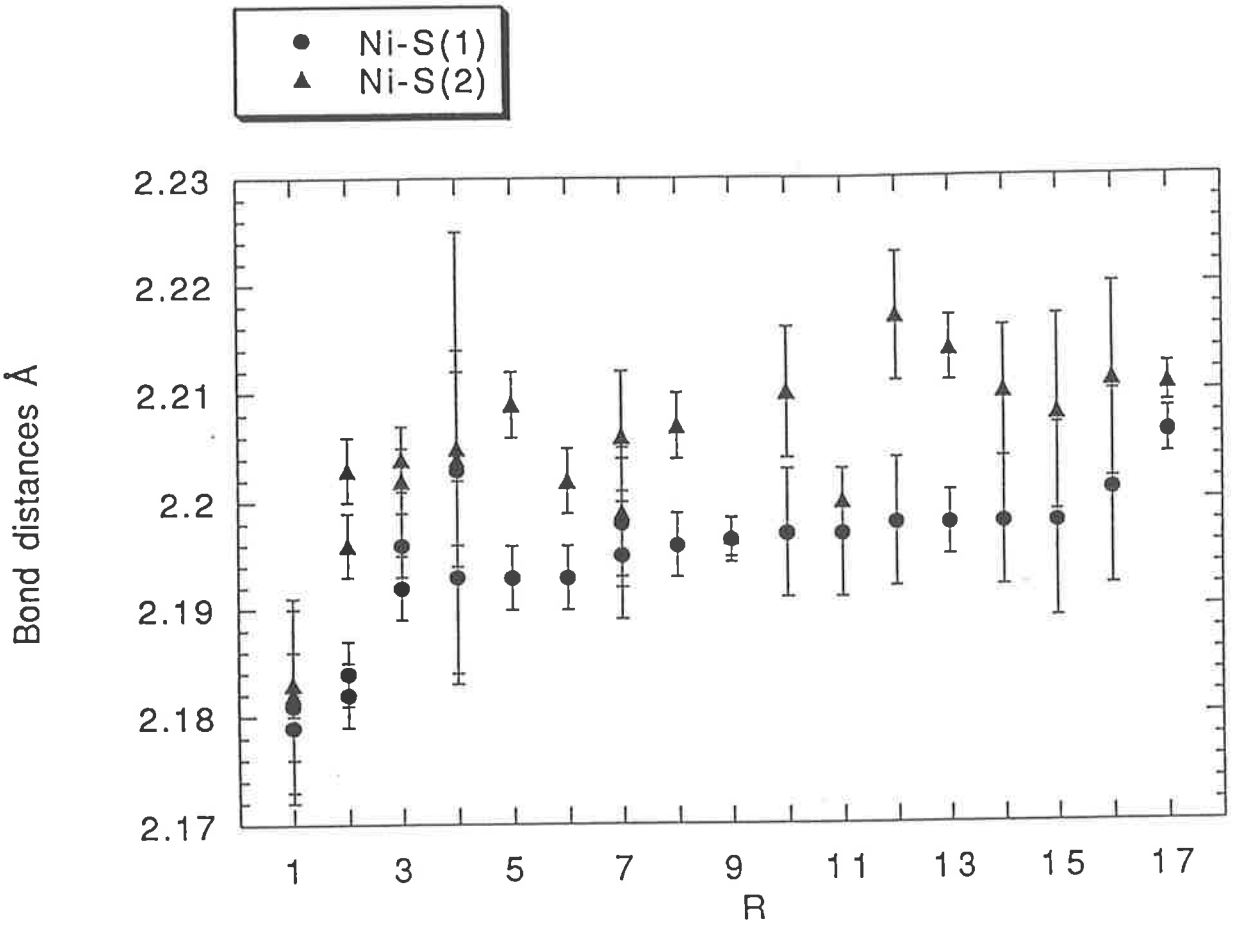


Fig. 3.13 Plot of Ni-S distances (Å) in $Ni(S_2CNR_2)_2$ versus R: **1**, R = iPr; **2**, R = Cy; **3**, R = Et; **4**, R = iBu; **5**, R = CH_2CH_2OH; **6**, R_2 = H, nPr; **7**, R_2 = acetyl, CH_2Ph; **8**, NR_2 = 1-piperidine; **9**, R_2 = Et, Cy; **10**, R = nPr; **11**, R = CH_2CF_3; **12**, R = Me; **13**, NR_2 = pyrrolidine; **14**, R = C_3H_5; **15**, R_2 = Me, Ph; **16**, R = nBu; **17**, NR_2 = morpholine.

are omitted from the plot owing to high standard deviations but, as for the xanthate structures above, their parameters are included in the calculation of average parameters. Two entries are included for $Ni(S_2CNR_2)_2$ representing the two polymorphs for this complex (Khan, Fackler, Murray, Heinrich, Campana, 1987; Selvaraju, Panchanatheswaran, Thiruvalluvar, Parthasarathi, 1995).

The Ni-S distances in the $Ni(S_2CNR_2)_2$ complexes range from 2.179(3) Å (R = iPr) to 2.224(15) Å (R = H), corresponding to a spread of 0.045 Å and the average calculated for all structures is 2.2011(2) Å. Disparities between Ni-S(1) and Ni-S(2) distances are also evident in the $Ni(S_2CNR_2)_2$ structures. As can be seen from Fig. 3.13, where the Ni-S distances has been determined at a relatively high level of precision, the difference in the Ni-S distances is greater than the 3σ limit. It is likely that the disparities arise, in part, as a result of intramolecular interactions between the sulfur atoms and R substituents. The comparatively large spread of Ni-S distances suggests that there may be some correlation between their magnitude and R. There does not appear to be a correlation between the chain length of R and the Ni-S distances, however, there is a suggestion that increasing the branching of the R groups results in a decrease in Ni-S distance, *e.g.* av(Ni-S) for R = nPr is 2.203(1) Å *cf.* 2.181(2) Å for R = iPr. Indeed, as a general trend, it would appear that the structures with the largest R groups (*e.g.* iPr, Cy) tend to form the shortest Ni-S bonds. This observation is consistent with the increased steric hindrance forcing the larger R groups further away from the coordinated sulfur atoms and hence, reducing any interaction between the sulfur and R groups. In terms of bite angles, the S-C-S angles range from 107.8(3)° to 112(2)° with an average value of 109.95(3)° and the S-Ni-S chelate angles range from 78.4(2)° to 81.0(4)° (average value is 79.316(9)°). These parameters may be compared to those obtained for the xanthates.

The average Ni-S bond distance in the xanthate complexes of 2.2107(2) Å is longer than the equivalent parameter (2.2011(2) Å) for the dithiocarbamate structures confirming that the dithiocarbamate ligand is a better coordinating ligand than the xanthate ligand (Hoskins, Tiekink, Winter, 1985a). The S-C-S angle is wider in the xanthate

complexes compared with the dithiocarbamate complexes, *i.e.* 113.35(4)° *cf.* 109.95(3)°, however, owing to the longer Ni-S distances in the xanthate structures, the S-Ni-S chelate angles are effectively equivalent at 79.371(6)° and 79.316(9)°, respectively.

3.4 CONCLUSIONS

The results of this study indicate that there is no impedance for both xanthate and dithiocarbamate ligands to coordinate in a symmetric, bidentate fashion in their bis complexes of nickel(II). The relatively small ranges of Ni-S distances in both series of complexes and a lack of systematic variation indicate that no definitive trend exists between the Ni-S separations and the electronic/steric profiles of the respective ligands as moderated by the nature of R. Instead, the small variations in Ni-S parameters may be rationalised in terms of intramolecular interactions between the sulfur atoms and the R residues and intermolecular forces.

Chapter 4

TELLURIUM(II) BIS 1,1-DITHIOLATES

In this chapter, four new $Te(S_2COR')_2$ structures and one new $Te(S_2CNR_2)_2$ structure have been determined and their structural characteristics are described below. As with chapter 3, the derived interatomic parameters of these new structures are compared with their literature analogues to assess the coordinating ability of the xanthate and dithiocarbamate ligands in their respective tellurium(II) complexes.

4.1 XANTHATE STRUCTURES

The molecular structure of $[Te(S_2COnPr)_2]$ is shown in Fig. 4.1 and selected interatomic parameters are listed in Table 4.1. The immediate coordination geometry about the tellurium atom is defined by four essentially coplanar sulfur atoms derived from two asymmetrically chelating xanthate ligands. The Te–S distances fall into two classes, *i.e.* short (2.5 Å) and long (2.9 Å) and these are arranged such that the shorter interactions lie on the same side of the molecule. Hence, the S_4 donor set defines a trapezoidal planar geometry. The two lone pairs of electrons of the tellurium(II) centre are thought to lie above and below the TeS_4 plane and to be orientated over the weaker Te–S bonds. The mean deviation of the sulfur atoms from the least-squares plane through them is 0.012 Å and the tellurium atom lies 0.0123(6) Å above this plane. Other parameters associated with the xanthate ligands are as expected, in particular the disparity in the C–S bond distances with the C–S(1), S(3) bonds of 1.70(1) Å and 1.71(1) Å being considerably longer than the C–S(2), S(4) bonds of 1.61(1) Å and 1.64(1) Å. The longer C–S bonds are associated with the shorter of the Te–S interactions, this implies that canonical structure I makes a significant contribution to the structure of the ligand. The C–O bond lengths of 1.37(5) Å and 1.33(1) Å are significantly shorter than the O-C bond distances of 1.47(1) Å and 1.46(1) Å indicating that canonical structure II also

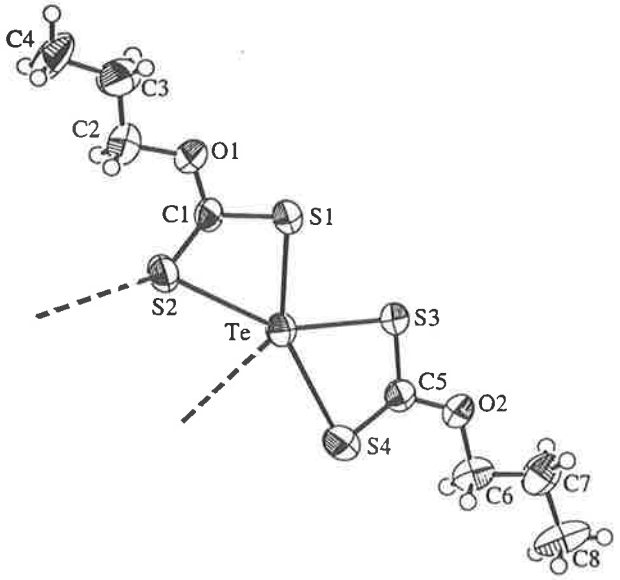


Fig. 4.1 Molecular structure of [Te(S2COnPr)2] showing the crystallographic numbering scheme.

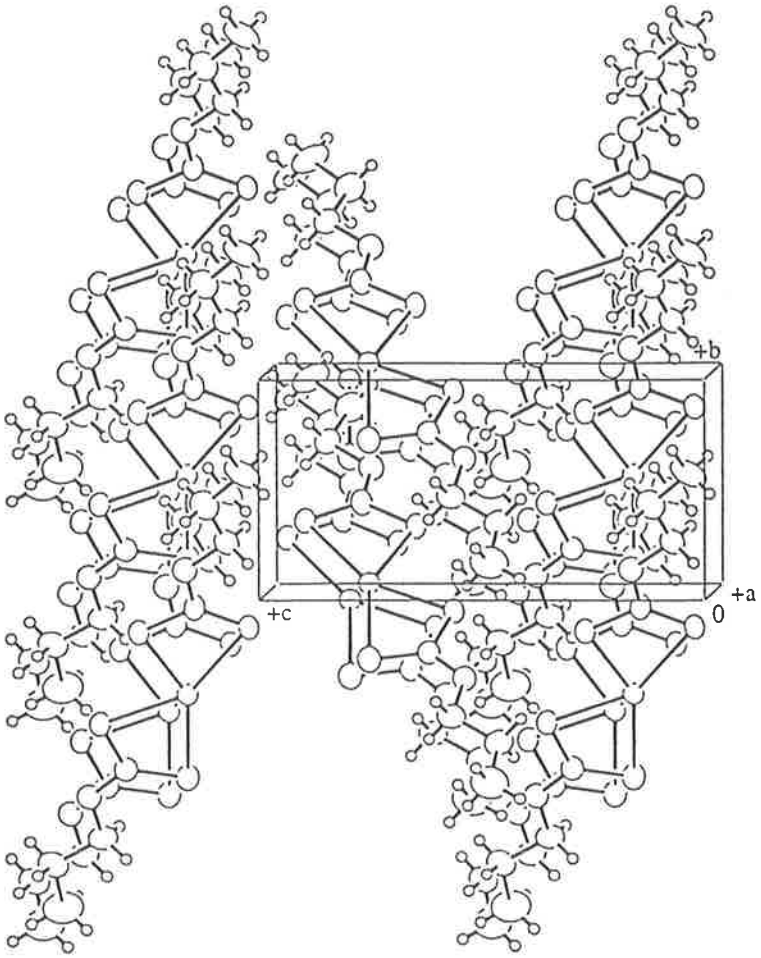


Fig. 4.2 Unit cell contents for [Te(S2COnPr)2].

Table 4.1 Selected intramolecular parameters for $Te(S_2COR)_2$

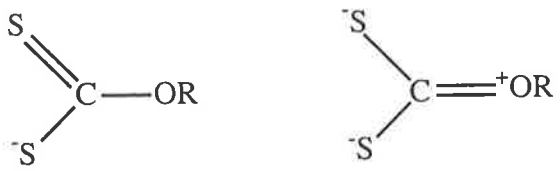
R	Te-S(1)	Te-S(2)	Te···S	S(1)-C	S(2)-C	C-O	S1-Te-S2	S1-C-S2	S2-Te-S4	S1-Te-S3
Me[a]	2.499(1)	2.846(1)	3.513(1)	1.717(4)	1.650(4)	1.314(5)	66.33(4)	121.6(2)	142.29(3)	85.18(4)
	2.510(1)	2.841(1)		1.709(4)	1.638(4)	1.335(5)	66.20(4)	122.6(2)		
Et[b]	2.486(11)	2.897(17)	3.61(1)	1.67(5)	1.69(3)	1.30(5)	65.5(4)	121.7(30)	144.5(3)	83.6(4)
	2.480(13)	2.857(14)		1.79(4)	1.63(4)	1.30(6)	66.4(3)	118.4(33)		
Et[c,d]	2.491(3)	2.948(4)	3.509(3)	1.734(12)	1.650(13)	1.293(16)	65.1(1)	121.4(8)	144.4(1)	83.3(1)
	2.481(3)	2.852(3)		1.716(13)	1.653(13)	1.334(16)	66.5(1)	123.6(8)		
	2.499(3)	2.958(4)	3.490(3)	1.703(12)	1.678(11)	1.281(15)	67.3(1)	121.0(8)	145.6(1)	83.2(1)
	2.483(4)	2.850(4)		1.716(13)	1.653(13)	1.307(16)	64.6(1)	121.8(8)		
nPr[e]	2.497(4)	2.881(4)	3.414(3)	1.70(1)	1.61(1)	1.36(2)	65.7(1)	124.5(9)	144.9(1)	83.2(1)
	2.495(3)	2.867(4)		1.71(1)	1.64(1)	1.33(1)	66.2(1)	123.6(8)		

Table 4.1 Cont.

R	Te-S(1)	Te-S(2)	Te···S	S(1)-C	S(2)-C	C-O	S-Te-S	S-C-S [c]	S-Te-S	S-Te-S
iPr[d,f]	2.483(4)	2.877(5)	4.516(4)	1.74(1)	1.64(1)	1.32(2)	66.2(1)	121.5(9)	144.1(1)	83.7(1)
	2.497(4)	2.881(5)		1.72(1)	1.64(1)	1.33(2)	65.9(1)	122.4(8)		
	2.481(3)	2.852(3)	4.657(4)	1.70(1)	1.64(1)	1.35(2)	65.5(1)	123.4(9)	145.2(1)	83.9(1)
	2.487(4)	2.920(6)		1.72(1)	1.66(1)	1.33(2)	65.3(1)	121.2(9)		
3,3-Me_2Bu[e]	2.489(6)	2.844(7)	3.701(6)	1.65(2)	1.73(2)	1.31(2)	66.0(2)	119(1)	142.0(1)	85.4(2)
	2.510(6)	2.885(7)	3.819(5)	1.78(2)	1.63(2)	1.31(2)	66.6(2)	121(1)		
i-Bu[e]	2.494(1)	2.839(1)	3.473(1)	1.721(5)	1.661(5)	1.324(6)	66.92(4)	121.7(3)	145.51(4)	82.14(4)
	2.510(1)	2.935(1)		1.723(5)	1.665(5)	1.323(5)	65.44(4)	122.1(3)		
CH_2Ph[e]	2.518(2)	2.814(1)	4.083(6)	1.713(5)	1.667(5)	1.324(5)	66.87(4)	121.4(3)	145.26(4)	81.54(4)
	2.535(1)	2.866(2)		1.719(5)	1.665(5)	1.328(5)	66.22(5)	122.2(3)		

a Graver, Husebye, 1975; b α-form (Husebye, 1967); c β-form (Tiekink, 1986); d two molecules in asymmetric unit; e this work; f Hoskins, Tiekink, Winter, 1985c.

makes a significant contribution to the overall structure of the ligand. As indicated in Fig. 4.1 there are additional Te···S interactions of note.



(I) (II)

Weak Te···S(2)' contacts of 3.414(3) Å (symmetry operation: $-x$, $-0.5 + y$, $2 - z$) exist in the lattice of [Te(S2COnPr)2] such that each molecule forms one 'donor' and one 'acceptor' contact; a view of the unit cell contents is provided in Fig. 4.2. The Te···S(2) separations are within 3.90 Å, being the sum of the van der Waals radii for these atoms. These contacts are aligned along the crystallographic *b*-axis leading to the formation of loosely associated molecules arranged as helical ribbons.

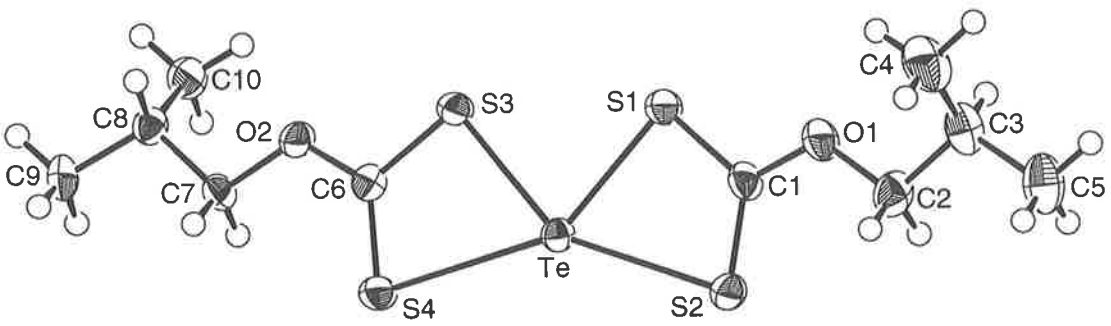


Fig. 4.3 Molecular structure of [Te(S2COiBu)2] showing the crystallographic numbering scheme.

Fig. 4.3 depicts the molecular geometry of [Te(S2COiBu)2] and selected interatomic contacts are listed in Table 4.1. The coordination geometry is essentially identical to the [Te(S2COnPr)2] described above with the characteristic short Te-S bond lengths (2.494(1) Å and 2.510(1) Å) situated on one side of the molecule and the longer

Te-S bond lengths (2.839(1) Å and 2.935(1) Å) on the opposite side, thus the four coordinating sulfur atoms adopt a trapezoidal geometry around the tellurium atom. The general features of the iBu xanthate ligand are similar to those described for the nPr derivative.

There is a significant intermolecular contact of 3.473(1) Å between Te and the S(3) atoms, symmetry operation (*+x*, -1/2-*y*, -1/2+*z*). This leads to similar lattice interactions as described for the [Te(S_2COnPr)$_2$] compound, *i.e.* loosely associated molecules arranged as helical ribbons.

The molecular geometry of [Te(S_2CO-3,3-Me_2Bu)$_2$] is illustrated in Fig. 4.4 and associated intramolecular contacts are listed in Table 4.1. This molecule exhibits similar intramolecular coordination to that reported previously. The asymmetric mode of coordination of the two ligands (2.487(4) Å, 2.850(4) Å and 2.509(4) Å, 2.883(4) Å) lead to the trapezoidal geometry of the sulfur atoms around the tellurium atom. The features associated with the 3,3-Me_2Bu xanthate ligand are similar to those described for the nPr derivative. A difference between this structure and the two previously reported ones occurs in the nature of the intermolecular Te···S interactions.

A view of the unit cell contents for [Te(S_2CO-3,3-Me_2Bu)$_2$] is shown in Fig. 4.5. In this case each molecule forms two donor and two acceptor contacts as indicated in Fig. 4.4. The structure can be described as being comprised of layers of molecules lying in the *bc*-plane that are sandwiched by alkyl-rich regions. The Te···S contacts occur along the *b*-direction where pairs of Te···S contacts occur between centrosymmetrically related molecules such that each molecule forms associations with two other molecules. Thus, Te···S(2)' is 3.819(5) Å (symmetry operation: -*x*, -*y*, 2-*z*) and Te···S(4)'' is 3.701(6) Å (symmetry operation: -*x*, 1-*y*, 2-*z*). These distances are significantly longer than the comparable distance in the structure of [Te(S_2COnPr)$_2$] and are only slightly less than the sum of the van der Waals radii for these atoms. The lengthening of the Te···S interactions may reflect the fact that each tellurium now forms two weak intermolecular contacts rather than one as in the structure of [Te(S_2COnPr)$_2$].

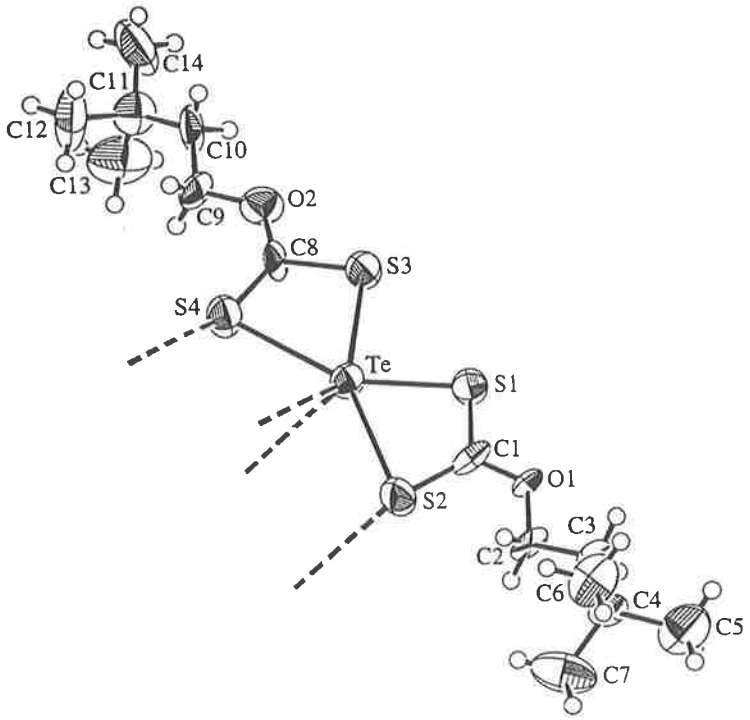


Fig. 4.4 Molecular structure of [Te(S2CO-3,3-Me2Bu)2] showing the crystallographic numbering scheme.

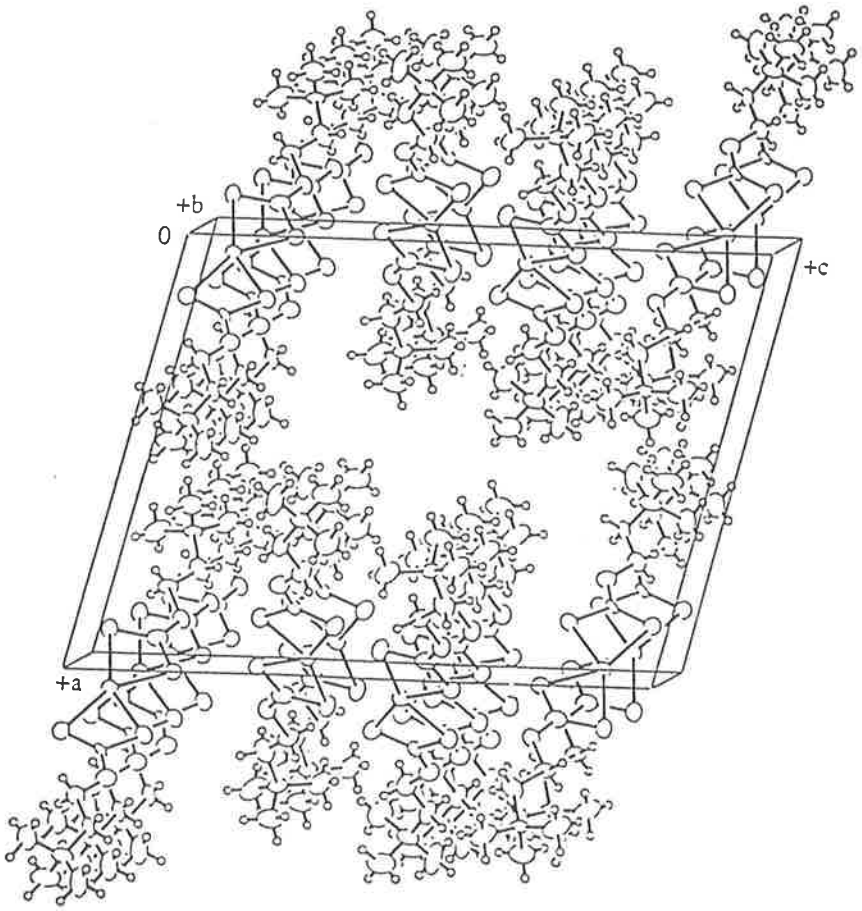


Fig. 4.5 Unit cell contents for [Te(S2CO-3,3-Me2Bu)2].

Fig. 4.6 illustrates the molecular geometry of [Te(S_2COBenzyl)$_2$] and selected parameters are listed in Table 4.1. The mode of coordination of the xanthate ligands to the tellurium atom is asymmetric (2.518(2) Å, 2.814(1) Å and 2.535(1) Å, 2.866(2) Å) with the shorter of the two Te-S bonds situated on the same side of the molecule forming a part of the trapezoidal planar geometry of sulfur atoms. The general features of the benzylxanthate ligand are similar to those observed for the nPr derivative. There is a difference between this structure and those reported above, that occurs in the crystal geometry of the compound.

The unit cell of [Te(S_2COBenzyl)$_2$] illustrated in Fig 4.7 depicts the molecule with no significant interactions between molecules. Unlike the structures reported above the compound contains no significant interactions less than the sum of van der Waals radii.

4.2 DITHIOCARBAMATE STRUCTURE

The molecular structure of [Te(S_2CNCy$_2$)$_2$] is shown in Fig. 4.8 and selected geometric parameters given in Table 4.2. The geometry in this complex is essentially as described above for the xanthate structures with the tellurium atom lying 0.0288(6) Å out of the S_4 least-squares plane (mean deviation: 0.017 Å). In the lattice, the closest intermolecular contact of 3.55(2) Å occurs between the C(11) and C(17)^i atoms (symmetry operation *i*: 0.5-*x*, -0.5+*y*, 0.5-*z*).

4.3 SYSTEMATIC TRENDS FROM CRYSTALLOGRAPHY

Table 4.1 lists some selected intramolecular parameters of the known Te(S_2COR)$_2$ structures and Table 4.3 summarises some salient geometric parameters, namely the Te-S distances, Δ(Te-S) [*i.e.* Te-S_{long} – Te-S_{short}], and the intermolecular Te···S contacts, when present. Two features of the Te(S_2COR)$_2$ structures merit particular attention, firstly the degree of asymmetry in the mode of

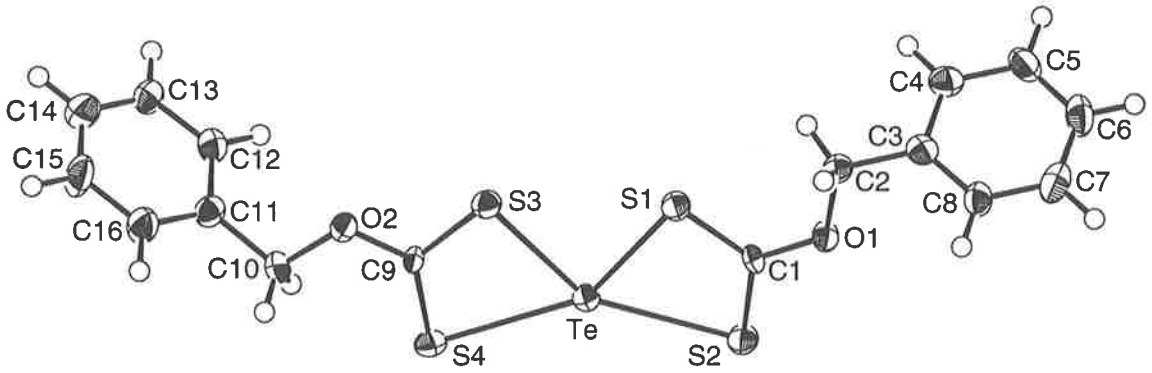


Fig. 4.6 Molecular structure of [Te($S_2COCH_2Ph)_2$] showing the crystallographic numbering scheme.

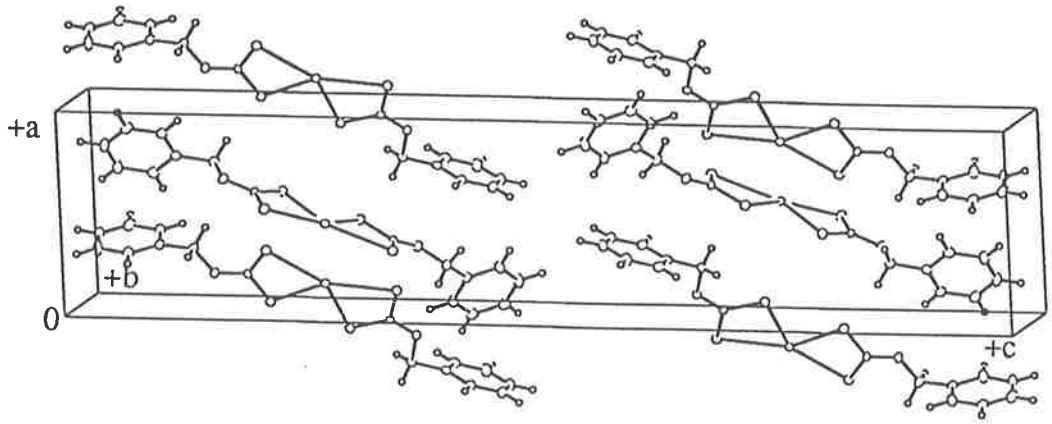


Fig. 4.7 Unit cell contents for [Te($S_2COBenzyl)_2$].

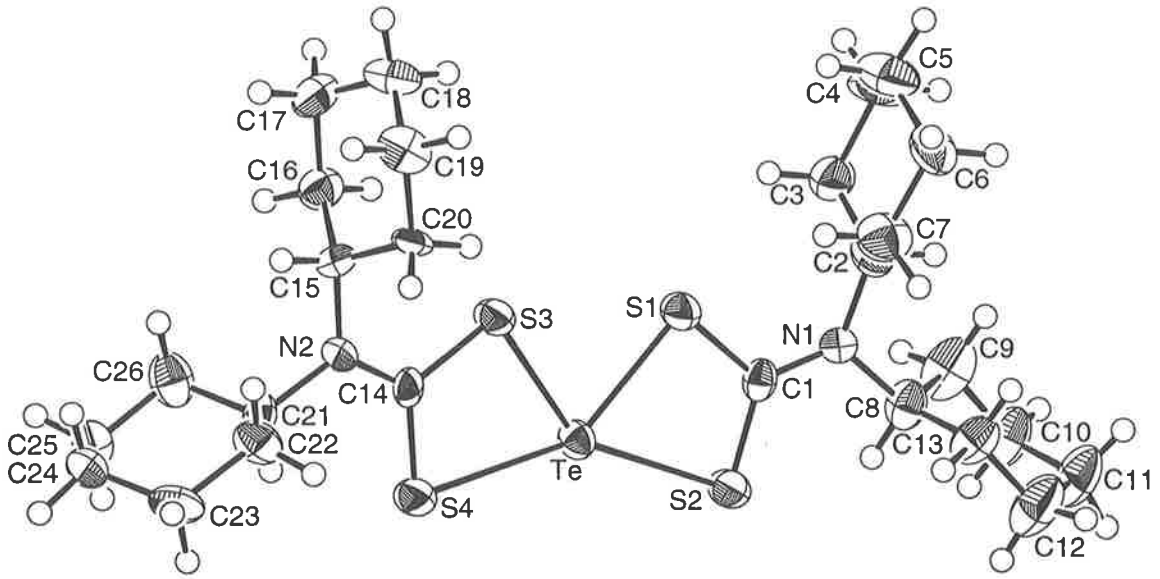


Fig. 4.8 Molecular structure of [Te(S_2CNCy_2)2] showing the crystallographic numbering scheme.

Table 4.2 Selected intramolecular parameters for $Te(S_2CNR_2)_2$

R	Te-S(1)	Te-S(2)	Te···S	S(1)-C	S(2)-C	C-N	S1-Te-S2	S1-C-S2	S2-Te-S4	S1-Te-S3
Et[a]	2.518(4)	2.893(4)	3.579(5)	1.73(2)	1.71(2)	1.33(2)	65.8(1)	118.6(7)	147.8(1)	80.1(1)
	2.519(4)	2.830(3)		1.74(2)	1.69(2)	1.36(2)	66.5(1)	117.9(9)		
NR_2[b]	2.527(7)	2.793(8)	3.296(7)	1.73(3)	1.71(3)	1.36(4)	67.4(3)	119(2)	146.8(2)	80.3(3)
	2.562(8)	2.788(6)		1.71(3)	1.64(3)	1.39(3)	65.6(2)	120(1)		
n-Pr[c]	2.511(1)	2.853(1)	3.499(1)	1.74(5)	1.686(5)	1.323(6)	66.0(1)	118.1(3)	147.0(1)	80.5(1)
	2.516(1)	2.821(2)		1.732(5)	1.691(5)	1.321(7)	66.5(1)	118.1(3)		
i-Pr[d]	2.520(2)	2.821(2)	3.628(1)	1.740(6)	1.687(6)	1.321(8)	65.8(1)	116.3(3)	148.1(1)	79.9(1)
	2.530(2)	2.821(2)		1.747(7)	1.693(6)	1.337(8)	66.0(1)	116.4(3)		
NR_2[e]	2.498(7)	2.856(8)	3.676(7)	1.76(3)	1.69(2)	1.33(4)	66.8(2)	117.9(16)	145.7(2)	80.9(2)
	2.530(6)	2.834(9)		1.74(3)	1.67(2)	1.33(4)	66.6(2)	119.8(17)		

Table 4.2 Cont.

R	Te-S(1)	Te-S(2)	Te···S	S(1)-C	S(2)-C	C-N	S1-Te-S2	S1-C-S2	S2-Te-S4	S1-Te-S3
Cy[f]	2.506(3)	2.835(3)	5.959(3)	1.734(9)	1.70(1)	1.34(1)	65.85(8)	116.4(5)	147.41(8)	81.34(8)
	2.516(3)	2.854(3)		1.735(9)	1.70(1)	1.34(1)	65.38(8)	116.2(5)		

a Fabianič , Spagna, Vaciago, Zambonelli, 1971; b NR2 = CH_2CH_2OH (Rout, Seshasayee, Radha, Aravamudan, 1983); c Ganesh, Seshasayee, Chidambaram, Aravamudan, Goubitz, Schenk, 1989; d Kumar, Aravamudan, Seshasayee, Selvam, Yvon, 1990; e NR_2 = morpholine (Husebye, 1970); f this work.

Table 4.3 Summary of tellurium to sulfur bond distances (Å) for $Te(S_2COR)_2$

R	Te-S_{short}	Te-S_{long}	ΔTe-S	av. ΔTe-S	Te···S
Me[a]	2.510(1)	2.841(1)	0.331(1)		3.513(1)
	2.499(1)	2.846(1)	0.347(1)	0.3390(7)	
Et[b]	2.486(11)	2.897(17)	0.411(20)		3.61(1)
	2.480(13)	2.857(14)	0.377(19)	0.393(14)	
Et[c,d]	2.491(3)	2.948(4)	0.457(5)		3.509(3)
	2.481(3)	2.852(3)	0.371(4)		
	2.499(3)	2.958(4)	0.459(5)		3.490(3)
	2.483(4)	2.850(4)	0.367(6)	0.411(2)	
nPr[e]	2.497(4)	2.881(4)	0.384(6)		3.414(3)
	2.495(3)	2.867(4)	0.372(5)	0.377(4)	
iPr[d,f]	2.483(4)	2.877(5)	0.394(6)		n/a
	2.497(4)	2.881(5)	0.384(6)		
	2.487(4)	2.906(5)	0.419(6)		
	2.487(4)	2.920(6)	0.433(7)	0.406(3)	
3,3-Me_2Bu[e]	2.489(6)	2.844(7)	0.355(9)		3.701(6)
	2.510(6)	2.885(7)	0.375(9)	0.365(10)	3.819(5)
iBu[e]	2.494(1)	2.839(1)	0.345(1)		3.473(1)
	2.510(1)	2.935(1)	0.425(1)	0.3850(7)	
CH_2Ph[e]	2.517(2)	2.814(1)	0.297(3)		n/a
	2.535(1)	2.866(2)	0.331(3)	0.314(2)	

a Graver, Husebye, 1975; b α-form (Husebye, 1967); c β-form (Tiekink, 1986); d two molecules in asymmetric unit; e this work; f Hoskins, Tiekink, Winter, 1985c.

coordination of the xanthate ligand as measured by Δ(Te-S) and secondly, the influence of intermolecular Te···S interactions on the Te-S distances. The significance of supramolecular Te···S interactions in the structural chemistry of tellurium/sulfur compounds has been highlighted in a recent review (Haiduc, King, Newton, 1994).

In all of the $Te(S_2COR)_2$ structures the xanthate ligands chelate the tellurium atoms forming disparate Te-S interactions, *i.e ca* 2.5 Å and 2.9 Å. The $Te\text{-}S_{short}$ distances lie in the range 2.480(13) Å to 2.535(1) Å with an average value of 2.5071(4) Å and the range for the $Te\text{-}S_{long}$ distances is 2.814(1) Å to 2.958(4) Å with an average distance of 2.8589(4) Å. These parameters are reflected in the values of Δ(Te-S) for each ligand which lie in the range 0.314(2) Å to 0.411(2) Å, however, there is no obvious correlation between the nature of R and Δ(Te-S). The small range of Δ(Te-S) indicates that there is no significant dependence of R on the coordinating ability of the xanthate ligands. The consistently observed asymmetric mode of coordination leads to a constant coordination geometry for $Te(S_2COR)_2$. Thus, in the trapezoidal planar geometry the S-Te-S chelate angles lie in the range 64.6(1)° to 67.3(1)° (average 66.23(1)°), the S_{short}-Te-S_{short} angles lie in the range 81.54(4)° to 85.4(2)° (average 83.09(2)°) and the range of S_{long}-Te-S_{long} is 142.0(1)° to 145.6(1)° (average 144.02(2)°). As there is no obvious correlation between the coordinating ability of the xanthate ligand and R, *i.e.* intramolecular effects, intermolecular contacts for these systems are now considered.

A systematic examination of the Te···S interactions, listed in Table 4.3, was undertaken in order to ascertain whether the variations in Δ(Te-S) could be related to intermolecular interactions of this type. In two structures, *i.e.* R = iPr and CH_2Ph, there were no significant Te···S interactions and yet the Δ(Te-S) varied from 0.297(3) Å to 0.433(7) Å. Conversely, in the R = Me and R = nPr structures where intermolecular Te···S interactions of 3.513(1) Å and 3.414(3) Å, respectively occur, no significant differences in Δ(Te-S) were noted. From the above, it is clear that there is no direct relationship between the mode of coordination of the xanthate ligand and the presence of Te···S interactions in the lattice. The lack of a systematic influence by the Te···S contacts

on the Te-S distances suggests that these contacts should be considered as only very weak interactions. This point is borne out by the different associations, when present, seen in the lattices indicating that the Te···S interactions are not dominant in forcing a common packing motif. Thus, in the R = Me and Et (two polymorphs) structures associations occur between centrosymmetrically related pairs. By contrast extended Te···S interactions leading to chains (R = iBu and R = 3,3-Me_2Bu) and helical ribbons (R = nPr) are found in other examples. Finally, there is no correlation between the presence of Te···S and the bulk of the R substituent; see Table 4.3. An analysis of the derived interatomic parameters, similar to that described above for the $Te(S_2COR)_2$ compounds, was conducted for the related dithiocarbamate structures.

Table 4.2 collates some selected intramolecular parameters for the $Te(S_2CNR_2)_2$ compounds and Table 4.4 lists pertinent structural information. The $Te\text{-}S_{short}$ distances lie in the range 2.498(7) Å to 2.562(8) Å with an average of 2.5158(6) Å and the range of $Te\text{-}S_{long}$ is 2.788(6) Å to 2.893(4) Å with an average of 2.8398(7) Å. The range of Δ(Te-S) is 0.226(10) Å to 0.375(6) Å. As for the xanthate structures, there is no obvious correlation between the magnitude of Te-S and the nature of R nor is there a correlation with the extent of Te···S interactions in the lattice. That the trapezoidal planar motif is consistently found in the $Te(S_2CNR_2)_2$ structures is reflected in the ranges of S-Te-S chelate (65.38(8)° to 67.4(3)°, average 65.98(3)°), $S_{short}\text{-}Te\text{-}S_{short}$ (79.9(1)° to 81.34(8)°, average 80.58(5)°) and $S_{long}\text{-}Te\text{-}S_{long}$ (145.7(2)° to 148.1(1)°, average 147.43(4)°) angles.

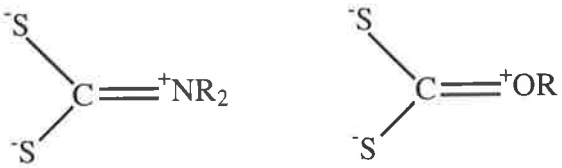
The difference between the average $Te\text{-}S_{short}$ and $Te\text{-}S_{long}$ distances for the xanthate structures of 0.352 Å is longer than the equivalent value for the dithiocarbamate complexes of 0.324 Å and clearly indicates that the dithiocarbamate ligand is a better chelating ligand than the xanthate ligand. This confirms the results of the crystal structure determination of a mixed xanthate/dithiocarbamate tellurium(II) compound, *i.e.* $[Te(S_2COEt)(S_2CNEt_2)]$ (Hoskins, Tiekink, Winter, 1985a) where the dithiocarbamate ligand binds in a more symmetric fashion. These observations may be rationalised in

Table 4.4 Summary of tellurium to sulfur bond distances (Å) for $Te(S_2CNR_2)_2$

R	Te-S_{short}	Te-Slong	ΔTe-S	av. ΔTe-S	Te···S
Et[a]	2.518(4)	2.893(4)	0.375(6)		3.579(5)
	2.519(4)	2.830(3)	0.311(5)	0.337(4)	
CH_2CH_2OH[b]	2.527(7)	2.793(8)	0.266(11)		3.296(7)
	2.562(8)	2.788(6)	0.226(10)	0.244(7)	
nPr[c]	2.511(1)	2.853(1)	0.342(1)		3.499(1)
	2.516(1)	2.821(1)	0.305(2)	0.3346(9)	
iPr[d]	2.520(2)	2.821(2)	0.301(3)		3.628(2)
	2.530(2)	2.821(2)	0.291(3)	0.296(2)	
NR_2[e]	2.498(7)	2.856(8)	0.358(11)		3.676(8)
	2.530(6)	2.834(9)	0.304(11)	0.331(8)	
Cy[f]	2.505(3)	2.835(3)	0.330(4)		n/a
	2.516(3)	2.851(3)	0.335(4)	0.333(3)	

a Fabianič , Spagna, Vaciago, Zambonelli, 1971; b Rout, Seshasayee, Radha, Aravamudan, 1983; c Ganesh, Seshasayee, Chidambaram, Aravamudan, Goubitz, Schenk, 1989; d Kumar, Aravamudan, Seshasayee, Selvam, Yvon, 1990; e NR_2 = morpholine (Husebye, 1970); f this work.

terms of the greater relative contribution of canonical structure (I) to the overall structure of the dithiocarbamate ligand compared with the analogous canonical structure (II) for the xanthate ligand.



(I) (II)

4.4 CONCLUSIONS

The foregoing discussion shows that significant variations in the Te-S bond distances may be found in individual bis(xanthate) and bis(dithiocarbamate) compounds, all of which display a common structural motif based on a trapezoidal planar geometry, and that the differences in Te-S can not be correlated with the nature of R nor with intermolecular Te···S interactions. The coordinating abilities of the xanthate and dithiocarbamate ligands were found to be independent of the nature of R. These results confirm those found in chapter 3 with the nickel(II) complexes. It would appear that intermolecular forces, *i.e.* crystal packing forces, influence the geometric parameters about the tellurium atom in these systems in a manner similar to that described for related organotin systems (Buntine, Hall, Kosovel, Tiekink, 1998; Tiekink, Hall, Buntine, 1998) and transition metal complexes (Martín, Orpen, 1996; Cotton, Yokochi, 1997).

Chapter 5

ZINC(II) BIS 1,1-DITHIOLATES

In this chapter, one new $Zn(S_2COR')_2$ structure and two new $Zn(S_2CNR_2)_2$ structures are described. The derived interatomic parameters of these new structures are compared with the zinc 1,1-dithiolate structures discussed previously in the *Introduction* and an attempt to explain the structural diversity of these compounds pursues.

5.1 XANTHATE STRUCTURES

The molecular structure of $[Zn(S_2COnBu)_2]$ is shown in Fig. 5.1 and selected interatomic parameters are listed in Table 5.1. The asymmetric unit comprises two formula units of $Zn(S_2COnBu)_2$ and 1.5 molecules of benzene solvent, one of which is disposed about a center of inversion. The structure is cyclotetrameric being comprised of an approximate square of four zinc atoms with each edge bridged by a xanthate ligand leading to the formation of a 16-membered, $[-Zn-S-C-S-]_4$ ring; the ring is centrosymmetric. Each of the remaining four xanthate ligands chelates a zinc atom. The bridging xanthate ligands form almost symmetrical bridges with S(1)-Zn(1) and S(2)-Zn(2) 2.298(3) Å and 2.318(3) Å, respectively and S(5)-Zn(2) and S(6)-$Zn(1)^i$ of 2.324(3) Å and 2.355(3) Å (symmetry operation *i*: -*x*, -*y*, -*z*). There is slight asymmetry in the Zn(1)-S bonds formed by the chelating S(3), S(4) xanthate ligand (2.360(3) Å *cf.* 2.397(3) Å) compared with the other chelating ligand which forms symmetric Zn(2)-S(7), S(8) bond distances of 2.375(3) Å and 2.383(3) Å. Thus, each zinc atom exists in a distorted tetrahedral geometry defined by two bridging and two chelating sulfur atoms. Owing to the presence in each coordination geometry of the chelating xanthate ligand, with its associated restricted bite angle, the distortions from the ideal tetrahedral geometry are significant. Thus, the S-Zn-S angles range from 76.8(1)° to 128.7(1)° for the Zn(1) center and 76.5(1)° to 116.9(1)° for the Zn(2) atom with the smaller angles being due to the chelating ligands.

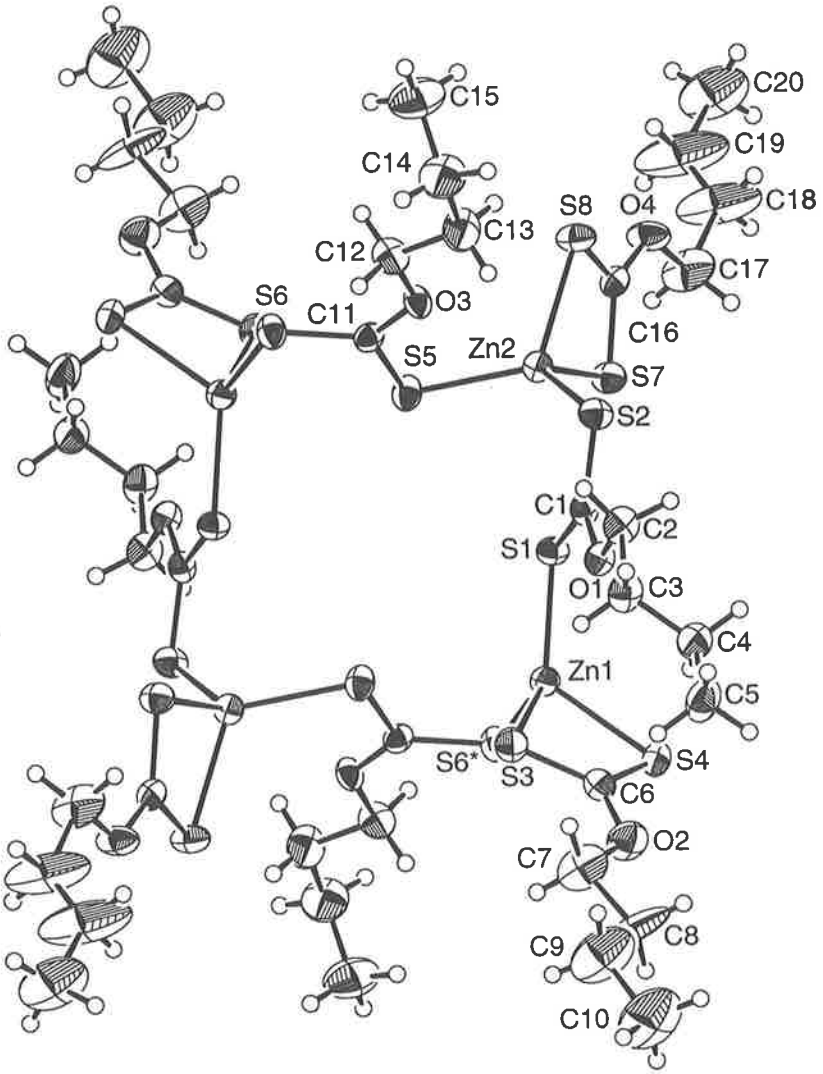


Fig. 5.1 Molecular structure of [$Zn(S_2COnBu)_2$] drawn with 35% probability ellipsiods showing the crystallographic numbering scheme.

Table 5.1 Selected interatomic parameters (Å, deg.) for $[Zn(S_2COnBu)_2]^a$

Zn(1)-S(1)	2.298(3)	Zn(2)-S(2)	2.318(3)
Zn(1)-S(3)	2.360(3)	Zn(2)-S(5)	2.324(3)
Zn(1)-S(4)	2.397(3)	Zn(2)-S(7)	2.375(3)
Zn(1)-S(6)^i	2.355(3)	Zn(2)-S(8)	2.383(3)
S(1)-C(1)	1.713(9)	S(5)-C(11)	1.701(9)
S(2)-C(1)	1.67(1)	S(6)-C(11)	1.70(1)
S(3)-C(6)	1.70(1)	S(7)-C(16)	1.68(1)
S(4)-C(6)	1.66(1)	S(8)-C(16)	1.70(1)
C(1)-O(1)	1.32(1)	C(11)-O(3)	1.29(1)
C(6)-O(2)	1.35(1)	C(y)-O(4)	1.32(1)
S(1)-Zn(1)-S(3)	128.7(1)	S(2)-Zn(2)-S(5)	116.8(1)
S(1)-Zn(1)-S(4)	122.6(1)	S(2)-Zn(2)-S(7)	116.9(1)
S(1)-Zn(1)-S(6)^i	108.9(1)	S(2)-Zn(2)-S(8)	111.5(1)
S(3)-Zn(1)-S(4)	76.8(1)	S(5)-Zn(2)-S(7)	113.0(1)
S(3)-Zn(1)-S(6)^i	112.5(1)	S(5)-Zn(2)-S(8)	115.7(1)
S(4)-Zn(1)-S(6)^i	101.5(1)	S(7)-Zn(2)-S(8)	76.5(1)
Zn(1)-S(1)-C(1)	105.7(4)	Zn(2)-S(2)-C(1)	101.9(3)
Zn(1)-S(3)-C(6)	80.4(4)	Zn(2)-S(5)-C(11)	100.3(3)
Zn(1)-S(4)-C(6)	79.9(4)	Zn(2)-S(7)-C(16)	81.5(4)
Zn(1)-S(6)^i-C(11)^i	102.5(3)	Zn(2)-S(8)-C(16)	80.7(4)
C(1)-O(1)-C(2)	117.2(8)	C(11)-O(3)-C(12)	120.5(8)
C(6)-O(2)-C(7)	120(1)	C(16)-O(4)-C(17)	118.8(9)
S(1)-C(1)-S(2)	122.4(6)	S(5)-C(11)-S(6)	121.4(6)
S(1)-C(1)-O(1)	115.4(7)	S(5)-C(11)-O(3)	116.7(7)
S(2)-C(1)-O(1)	122.2(7)	S(6)-C(11)-O(3)	121.9(7)
S(3)-C(6)-S(4)	122.9(7)	S(7)-C(16)-S(8)	121.2(6)

Table 5.1 Cont.

S(3)-C(6)-O(2)	121.5(8)	S(7)-C(16)-O(4)	123.2(8)
S(4)-C(6)-O(2)	115.6(8)	S(8)-C(16)-O(4)	115.6(8)

a symmetry operations i: *-x*, *-y*, *-z*

Clearly, the range of S-Zn-S angles about the Zn(1) atom is greater than that for the Zn(2) atom. This difference in the degree of distortion may be rationalised in terms of the influence of intramolecular Zn···O interactions, *i.e.* Zn(1)···O(1) is 3.074(6) Å and Zn(2)···O(3) is 2.927(6) Å. The O(1) atom is orientated towards the Zn(1) atom so as to lie over the chelating xanthate ligand and thereby pushing the S(3) and S(4) atoms away from the S(1) atom and expanding the associated angles. By contrast, the O(3) atom approaches the Zn(2) atom on a tetrahedral face defined through the S(2), S(5) and S(8) atoms, thereby expanding these angles subtended at zinc. As mentioned above, this structure crystallises with benzene solvent molecules in the lattice.

A view of the unit cell contents for $[Zn(S_2COnBu)_2].0.75C_6H_6$ is shown in Fig. 5.2 from which it can be seen that the crystallographically independent benzene molecules occupy distinct regions of the crystal lattice. An interesting feature of the crystal packing is the occupancy of the 16-membered rings. The centrosymmetric benzene molecules occupy channels defined by the rings aligned in the *c*-direction. The benzene molecules that occupy general positions stack in the *c*-direction but in hydrophobic regions defined by the n-butyl groups. Other xanthate complexes, notably immine adducts of nickel(II) bis xanthates (*e.g.* Gable, Hoskins, Winter, 1985), are known to form clathrates.

The structural motif reported here for $[Zn(S_2COnBu)_2]$ has a precedent in the literature. The $[Zn(S_2COiPr)_2]$ structure (Ito, 1972) also adopts a motif, labeled A in Fig. 5.3 (adapted from Fig 1.13 (vi)), based on isolated 16-membered rings. A difficulty of systematic studies of the type reported herein, is the difficulty in obtaining suitable single crystals for analysis and thus far, attempts to grow crystals of other $Zn(S_2COR)_2$ derivatives has proved elusive. We have, however, obtained crystals of the $[Zn(S_2COiBu)_2]$ compound and a preliminary X-ray analysis shows that motif A is also found. Severe disorder associated with the alkyl groups has precluded satisfactory modelling and hence, full details of the structure are not reported here. The only other $Zn(S_2COR)_2$ structure available for comparison is that of $[Zn(S_2COEt)_2]$ (Ikeda, Hagihara, 1966). This structure, shown as motif B in Fig. 5.3 (adapted from Fig 1.13

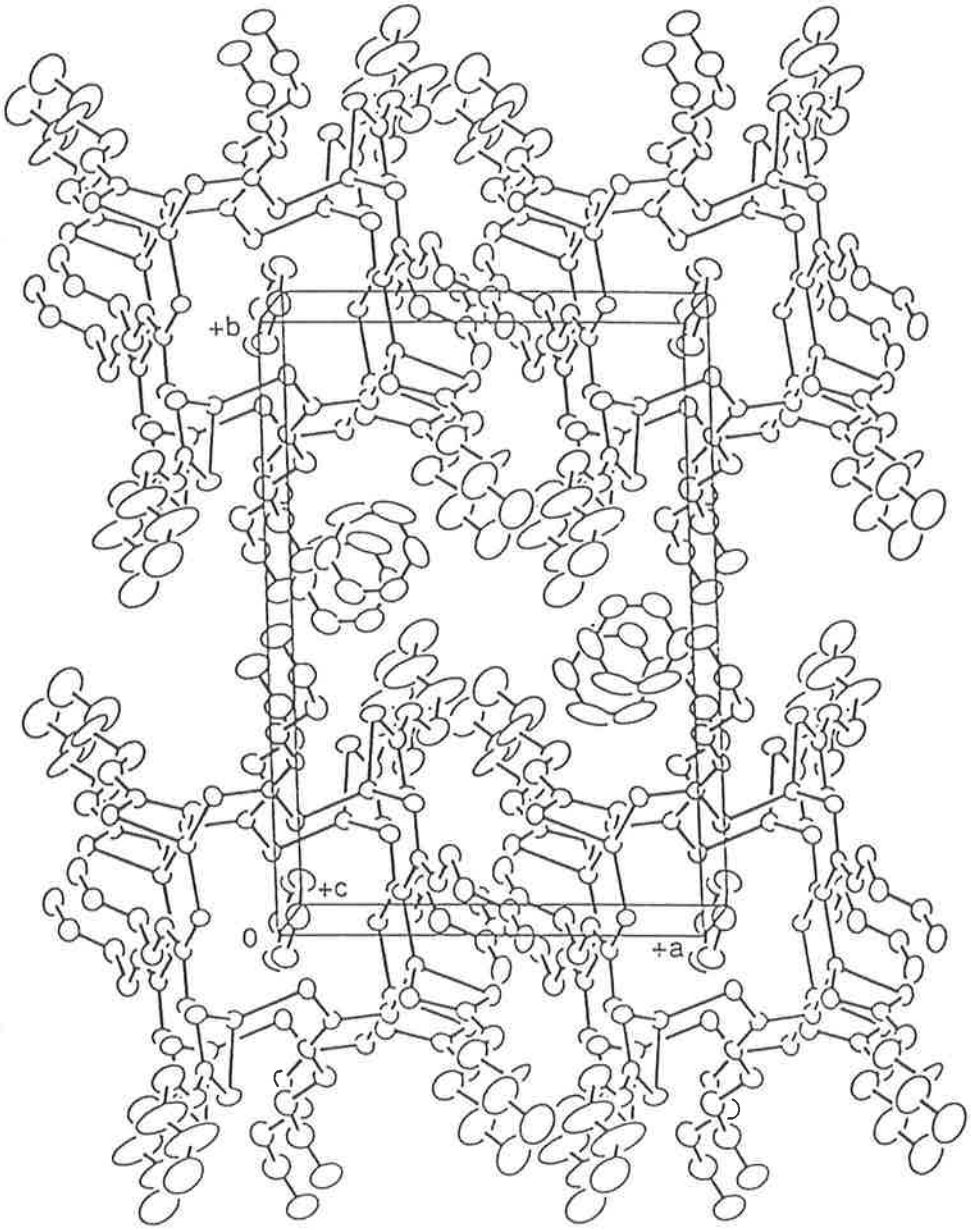


Fig. 5.2 Unit cell contents for $[Zn(S_2COnBu)_2]$.

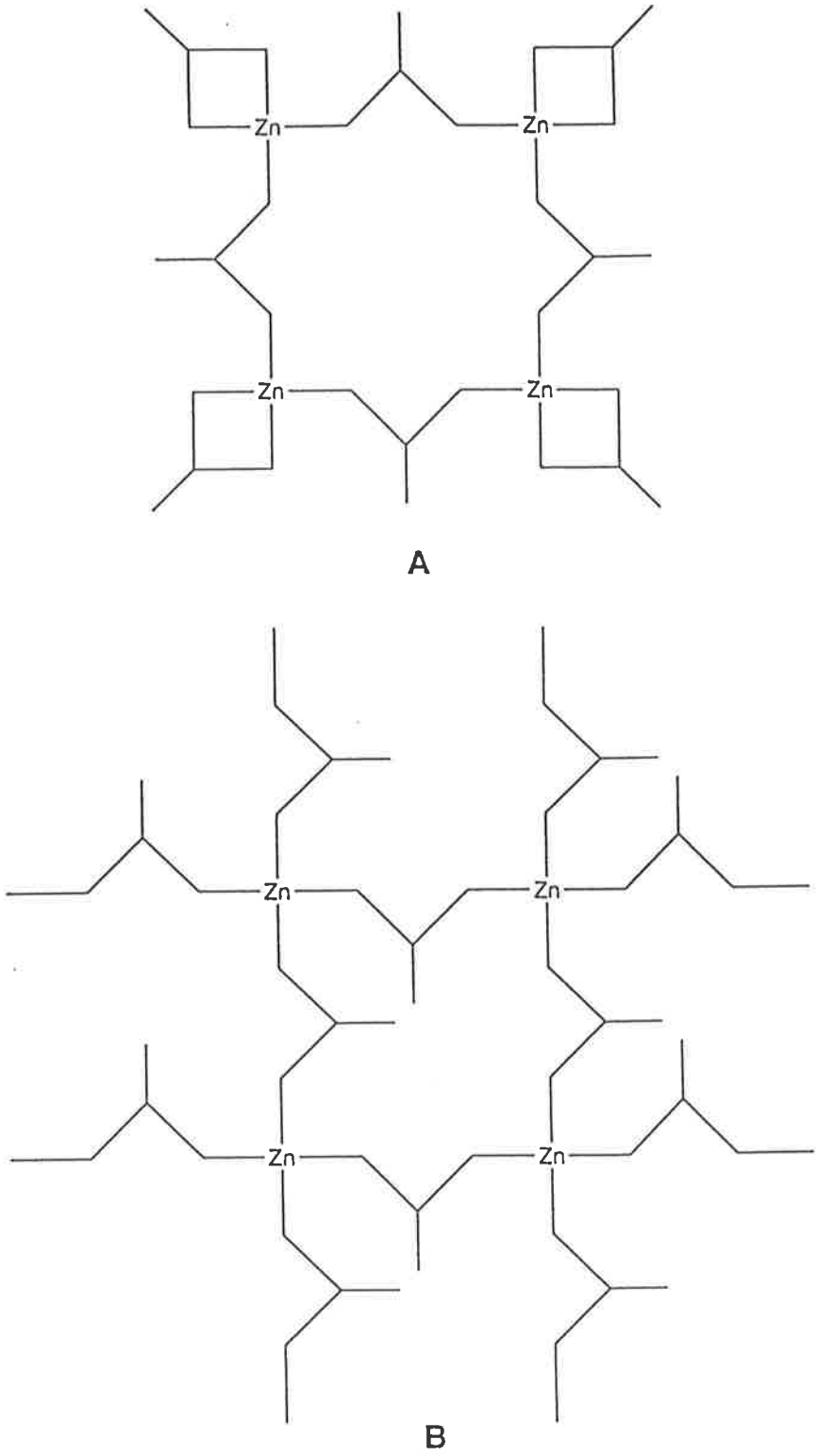


Fig. 5.3 Structural motifs for $Zn(S_2COR)_2$. Motif A is found for R = nBu, iBu and iPr. Motif B is found for R = Et.

(vii)), is also based on 16-membered rings, however, in this case the chelating xanthate ligands of motif A are now bridging adjacent zinc atoms of neighbouring 16-membered rings giving rise to 2-dimensional sheets but retaining tetrahedral ZnS_4 coordination. The absence of chelating ligands in motif B results in a more regular tetrahedral geometry as seen in the narrower range of S-Zn-S angles, *i.e.* 102.6(3)° to 115.2(3)°.

5.2 DITHIOCARBAMATE STRUCTURES

The molecular structure of $[Zn(S_2CN(Et)Cy)_2]$, containing a disymmetric dithiocarbamate ligand, is illustrated in Fig. 5.4 and selected interatomic parameters are collected in Table 5.2. Molecules associate via bridging dithiocarbamate ligands about a crystallographic center of inversion to generate 8-membered $[-Zn-S-C-S-]_2$ rings. The coordination geometries about the zinc atoms are defined by four sulfur atoms, two bridging and two chelating. The Zn-S bridging distances are similar at 2.341(1) Å and 2.366(1) Å but there is some asymmetry in the mode of coordination of the chelating ligand, Zn-S(1) 2.332(1) Å *cf.* Zn-S(2) 2.491(1) Å. The range of tetrahedral angles is 74.66(3)° to 135.62(4)° with the acute angle being associated with the chelating dithiocarbamate ligand. The wider S-Zn-S angles may be traced directly to the presence of an intramolecular $Zn\cdots S(4)^i$ contact of 2.838(1) Å (symmetry operation *i*: 1-*x*, -*y*, -*z*). This distance is less than the sum of the van der Waals radii for these atoms of 3.1 Å (Bondi, 1964) and it is significant that the Zn-S(2) bond, *i.e.* with the S(2) atom *trans* to the $S(4)^i$, is the longest of the four nearest Zn-S interactions. If $Zn\cdots S(4)^i$ was considered to represent a significant bonding interaction, a distortion towards a trigonal bipyramidal geometry would be indicated. In this description, the S(2) and $S(4)^i$ atoms would occupy the axial positions and define an axial angle of 158.23(3)°; the trigonal angles range from 102.78(5)° to 135.62(4)°. The $Zn\cdots Zn^i$ separation of 3.6827(9) Å is not indicative of a bonding interaction. As a consequence of the presence of a center of inversion in the 8-membered ring, the disposition of the alkyl substituents is such as to place them on either side of the molecule. Substitution of the

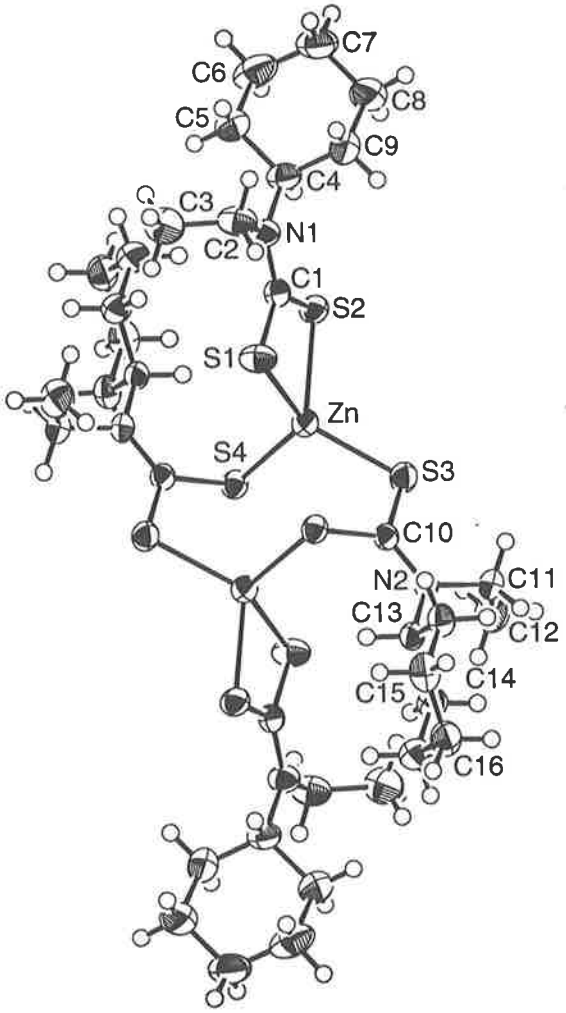


Fig. 5.4 Molecular structure of $[Zn(S_2CN(Et)Cy)_2]$ showing the crystallographic numbering scheme.

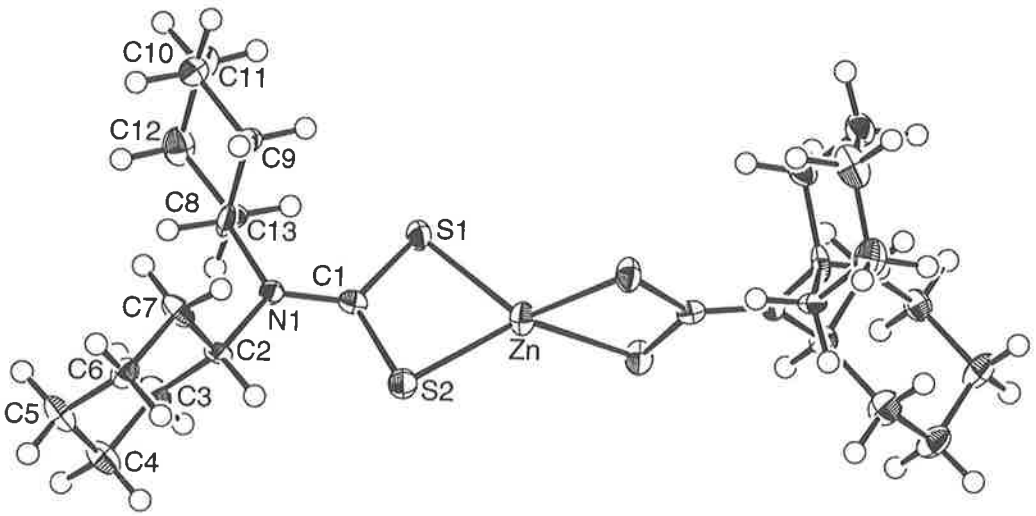


Fig. 5.5 Molecular structure of $[Zn(S_2CNCy_2)_2]$ showing the crystallographic numbering scheme.

Table 5.2 Selected interatomic parameters (Å, deg.) for $Zn(S_2CNR_2)_2$, R_2 = (Et, Cy), Cy_2[a]

bond	y = 4	y = 8
Zn-S(1)	2.332(1)	2.321(3)
Zn-S(2)	2.491(1)	2.349(3)
Zn-S(3)	2.341(1)	
Zn-S(4)	2.366(1)	
S(1)-C(1)	1.719(4)	1.731(7)
S(2)-C(1)	1.721(4)	1.746(9)
S(3)-C(10)	1.716(3)	
S(4)-C(10)	1.754(4)	
C(1)-N(1)	1.336(4)	1.33(1)
C(10)-N(2)	1.323(4)	
S(1)-Zn-S(2)	74.66(3)	77.83(7)
S(1)-Zn-S(3)	135.62(4)	125.9(1)[b]
S(1)-Zn-S(4)	117.06(4)	122.97(7)[b]
S(2)-Zn-S(3)	109.99(4)	122.97(7)[b]
S(2)-Zn-S(4)	110.55(4)	137.0(2)[b]
S(3)-Zn-S(4)	102.78(5)	77.83(7)[b]
Zn-S(1)-C(1)	86.8(1)	84.1(3)
Zn-S(2)-C(1)	81.8(1)	82.9(2)
Zn-S(3)-C(10)	94.6(1)	
Zn-S(4)-C(10)[i]	99.7(1)	
C(1)-N(1)-C(2)	120.6(3)	120.0(7)
C(1)-N(1)-C(y)	121.4(3)	124.9(6)
C(2)-N(1)-C(y)	117.5(3)	114.9(6)
C(10)-N(2)-C(11)	119.6(3)	

Table 5.2 Cont.

C(10)-N(2)-C(13)	121.9(3)	
C(11)-N(2)-C(13)	118.2(3)	
S(1)-C(1)-S(2)	116.6(2)	115.1(4)
S(1)-C(1)-N(1)	120.8(3)	122.6(6)
S(2)-C(1)-N(1)	122.6(3)	122.3(5)
S(3)-C(10)-S(4)^{i}	117.8(2)	
S(3)-C(10)-N(2)	121.1(3)	
S(4)^{i}-C(10)-N(2)	121.0(3)	

a symmetry operations i: 1-*x*, -*y*, -*z* and ii: *x*-*y*, -*y*, 1/3-*z*; b S(3)≡S(1)^{ii} and S(4)≡S(2)^{ii}

ethyl groups in [Zn(S_2CN(Et)Cy)$_2$] for cyclohexyl groups results in the adoption of another structural motif.

The molecular structure of [Zn(S_2CNCy$_2$)$_2$] is shown in Fig. 5.5 and selected geometric parameters are listed in Table 5.2. The molecule has crystallographically imposed 2-fold symmetry with the zinc atom situated on this axis. The molecular geometry is based on a tetrahedron defined by four sulfur atoms derived from two symmetrically chelating dithiocarbamate ligands, Zn-S(1) is 2.321(3) Å and Zn-S(2) is 2.349(3) Å. The range of S-Zn-S angles is 77.83(7)° to 137.0(2)° with the wider angle being defined by the two less tightly held S(2) atoms; this distortion probably reflects the restricted bite distance of each ligand. There are no intermolecular contacts involving non-H atoms in the lattice less than 3.6 Å.

The structural motif reported here for [Zn(S_2CN(Et)Cy)$_2$], *i.e.* motif C (see Fig. 5.6), has several precedents in the literature. Thus, the structures of Zn(S_2CNR$_2$)$_2$ where R = Et (Bonamico, Mazzone, Vaciago, Zambonelli, 1965; Zvonkova, Khvatkina, Ivanova, 1968), nPr (Sreehari, Varghese, Monoharan, 1990); -CH_2CH=CH_2 (Kellö, Vrábel, Kettmann, Garaj, 1983), and iPr (Miyamae, Ito, Iwasaki, 1979); where R_2 = $(CH_2)_4$ (Francetič , Leban, 1979) and $(CH_2)_6$ (Agre, Shugam, 1972b); and where R_2 = (Me, Et), (Me, nPr), (Me, iPr) and (Me, nBu) (Motevalli, O'Brien, Walsh, Watson, 1996) adopt this motif. A related structural motif, motif C' (Fig. 5.6), is found for [Zn(S_2CNMe$_2$)$_2$] where the 8-membered ring has crystallographic 2-fold symmetry with the consequence that the methyl groups lie to one side of the molecule (Klug, 1966; Ramalingam, bin Shawkataly, Fun, Abdul-Razak, 1998). The C motif may be described as the chair form and the C' motif as the boat form of Zn(S_2CNR$_2$)$_2$. The structure of [Zn(S_2CNCy$_2$)$_2$] represents a third motif, *i.e.* D, for Zn(S_2CNR$_2$)$_2$ structures. It is noteworthy that the motifs adopted by the zinc bis dithiocarbamates are quite distinct from those adopted by the zinc bis xanthates. Despite the appearance of the different motifs, A - D, the zinc centers invariably adopt distorted tetrahedral geometries with distortions

arising as a result of weaker Zn···O or Zn···S interactions as well as the restricted bite distances of the respective 1,1-dithiolate ligands.

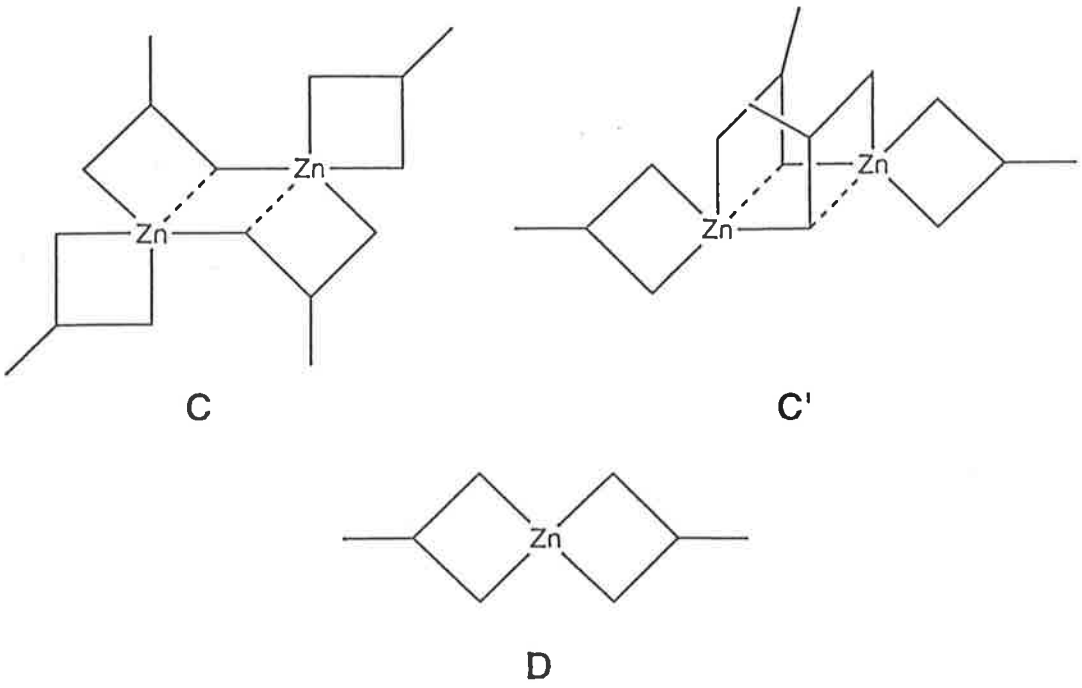


Fig. 5.6 Structural motifs for $Zn(S_2CNR_2)_2$. Motif C is found for R = Et, nPr, -$CH_2CH{=}CH_2$, and iPr; R_2 = $(CH_2)_4$ and $(CH_2)_6$; and R_2 = (Me, Et), (Me, nPr), (Me, iPr), (Me, nBu) and (Et, Cy). Motif C' is found for R = Me. Motif D is found for R = Cy. (Adapted from Fig. 1.13)

5.3 SYSTEMATIC TRENDS FROM CRYSTALLOGRAPHY

It is interesting to speculate on the origin of the different motifs found for the $Zn(S_2COR)_2$ and $Zn(S_2CNR_2)_2$ structures. For the zinc bis xanthates, only four structures are known and of these, three, with R = iPr, nBu and iBu, adopt the isolated 16-membered ring motif (A). The presence of the smaller ethyl substituent in $[Zn(S_2COEt)_2]$ results in association between the rings to form a layer structure (motif

B). Thus, a simple argument, based on the size of the R substituents, allows for a graduation from a cyclotetrameric motif to a 2-dimensional lattice as the size of R is reduced. In this context it is appropriate to consider the analogous mercury bis xanthates where a chain structure is found for R = Me (Tiekink, 1987), a 2-dimensional structure is found for R = Et (Watanabe, 1977; Chieh, Moynihan, 1980) and nPr (Hounslow, Tiekink, 1991), and a 3-dimensional structure is found for R = iPr (Watanabe, 1981), *i.e.* the greater the steric bulk, the greater the aggregation in the solid state, *i.e.* contrary to the general trend in the zinc bis xanthates. Clearly, extreme caution must be made when justifying the appearance of one motif in preference to another based on steric reasons alone.

With the above caveat in mind, it is nevertheless almost compelling to rationalise the appearance of the different motifs for the three $Zn(S_2CNR_2)_2$ structures, *i.e.* R = Et, R_2 = (Et, Cy) and R = Cy based on steric arguments. Thus, as the less sterically demanding ethyl groups are replaced by the larger cyclohexyl groups reduced association is observed. Also relevant is the observation that the Lewis basicity of the dithiocarbamate ligand is increased as ethyl is substituted for cyclohexyl (*e.g.* Hall, Tiekink, 1998; Tiekink, Hall, Buntine, 1998). Hence, the observed variation in structural motifs is consistent with variations in electronic effects as well.

5.4 CONCLUSIONS

Several motifs may be found in the solid state for the bis xanthates and bis dithiocarbamates of zinc(II). The steric profiles of the O- and N- bound groups seem to play a significant role in determining the ultimate motif adopted in the solid state for these systems, however, comparison with other Zn-triad structures suggests that other factors may also need to be taken into account.

Chapter 6

CADMIUM(II) BIS 1,1-DITHIOLATES

In this chapter, the crystal and molecular structures of four new $Cd(S_2CNR_2)_2$ complexes have been determined and their structural characteristics described below. Crystals of new $Cd(S_2COR')_2$ compounds suitable for X-ray analysis proved to be elusive, however a discussion of the cadmium 1,1-thiolate structures previously mentioned in the *Introduction* and the derived interatomic parameters of the four new $Cd(S_2CNR_2)_2$ pursues.

6.1 DITHIOCARBAMATE STRUCTURES

All of the new $Cd(S_2CNR_2)_2$ structures described herein adopt the same motif in the solid state and selected interatomic parameters for these are presented in Table 6.1. The molecular structure of $[Cd(S_2CNiPr_2)_2]$ is illustrated in Fig. 6.1. Two centrosymmetrically related molecules associate in the solid state via bridging dithiocarbamate ligands to form dinuclear units. The independent cadmium atom is chelated by two dithiocarbamate ligands one of which also bridges the neighbouring cadmium atom. The bridging S(2)-Cd^i distance of 2.601(2) Å is shorter than the intramolecular Cd-S(2) distance of 2.814(2) Å, an observation that is repeated in all four new $Cd(S_2CNR_2)_2$ structures (see Table 6.1); symmetry operation *i*: - *x*, - *y*, 1 – *z*. As a consequence of the bridging, the asymmetry in the Cd-S bond distances formed by the bridging dithiocarbamate ligand is greater than that found for the chelating ligand which does not participate in further associations with cadmium; ΔCd-S (*i.e.* $Cd\text{-}S_{long}$ - $Cd\text{-}S_{short}$) is 0.268(3) Å for the bridging ligand *cf.* 0.048(3) Å for the chelating ligand. The coordination geometry is best described as distorted trigonal bipyramidal with the S(2) and S(4) atoms (that form the longer of the Cd-S bonds) defining the axial positions. In this description, the cadmium atom lies 0.1950(6) Å out of the trigonal plane defined

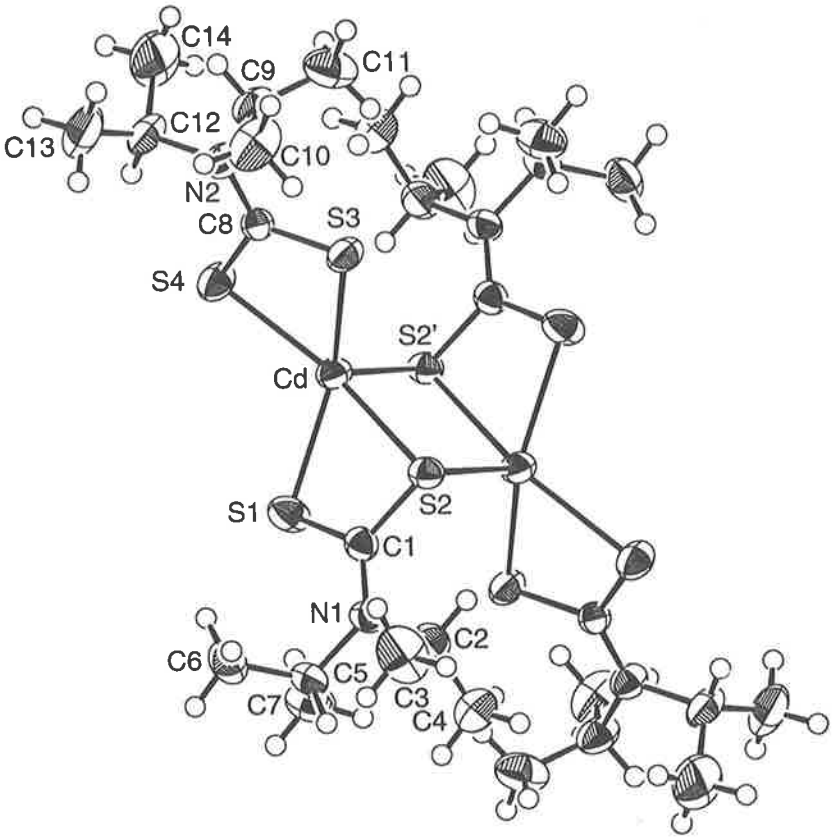


Fig. 6.1 Molecular structure of $[Cd(S_2CNiPr_2)_2]$ showing the crystallographic numbering scheme.

Table 6.1 Selected interatomic parameters (Å, deg.) for $Cd(S_2CNR_2)_2$, R = iPr, R_2 = (Et, Cy), R = Cy, R = iBu

	R = iPr[a]	R_2 = (Et, Cy)[b]	R = Cy[c]	R = iBu[d] molecule *a*	R = iBu[e] molecule *b*
	y = 2	y = 4	y = 2	y = 4	y = 4
	z = 8	z = 10	z = 14	z = 10	z = 10
Cd—S(1)	2.546(2)	2.549(2)	2.539(4)	2.566(4)	2.539(3)
Cd—S(2)	2.814(2)	2.626(2)	2.897(3)	2.592(3)	2.653(3)
Cd—S(3)	2.555(2)	2.550(2)	2.560(3)	2.591(3)	2.550(4)
Cd—S(4)	2.603(2)	2.812(2)	2.596(3)	2.780(3)	2.780(4)
Cd—S(y)[i]	2.601(2)	2.602(2)	2.551(3)	2.590(3)	2.617(3)
S(1)—C(1)	1.710(8)	1.741(8)	1.73(1)	1.74(1)	1.72(1)
S(2)—C(1)	1.753(7)	1.711(8)	1.76(1)	1.72(1)	1.72(1)
S(3)—C(z)	1.737(7)	1.735(8)	1.72(1)	1.718(9)	1.70(1)
S(4)—C(z)	1.729(7)	1.735(7)	1.74(1)	1.731(9)	1.74(1)
C(1)—N(1)	1.331(9)	1.329(9)	1.32(1)	1.34(2)	1.32(1)

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Table 6.1 Cont.

C(z)—N(2)	1.314(8)	1.303(9)	1.34(1)	1.35(1)	1.35(1)
S(1)—Cd—S(2)	66.71(6)	69.96(7)	65.4(1)	70.2(1)	69.4(1)
S(1)—Cd—S(3)	143.32(8)	143.30(8)	139.4(1)	151.69(9)	141.0(1)
S(1)—Cd—S(4)	109.96(8)	94.77(6)	108.0(1)	100.28(8)	111.4(1)
S(1)—Cd—S(y)^{i}	101.63(8)	111.42(7)	102.0(1)	108.9(1)	114.2(1)
S(2)—Cd—S(3)	97.02(6)	113.10(7)	93.6(1)	105.25(9)	98.4(1)
S(2)—Cd—S(4)	153.87(7)	155.18(7)	146.1(1)	145.60(8)	158.7(1)
S(2)—Cd—S(y)^{i}	96.50(6)	111.26(7)	92.4(1)	116.35(9)	109.3(1)
S(3)—Cd—S(4)	69.92(6)	67.42(6)	69.7(1)	67.07(7)	67.1(1)
S(3)—Cd—S(y)^{i}	113.11(7)	101.49(8)	113.8(1)	98.2(1)	104.8(1)
S(4)—Cd—S(y)^{i}	109.37(7)	92.29(6)	121.2(1)	98.04(7)	90.1(1)
Cd—S(y)—Cd^{i}	83.50(6)	87.71(6)	87.6(1)	81.96(7)	89.9(1)
Cd—S(1)—C(1)	92.9(2)	86.7(3)	95.5(4)	86.0(4)	87.8(4)
Cd—S(2)—C(1)	83.3(3)	84.8(3)	83.2(4)	85.7(4)	84.1(3)
Cd—S(3)—C(z)	87.2(2)	91.0(2)	87.3(4)	89.5(4)	91.3(4)
Cd—S(4)—C(z)	85.8(2)	82.6(3)	85.8(4)	100.9(3)	83.1(3)

Table 6.1 Cont.

Cd—S(y)—C	99.3(2)	97.6(2)	99.6(4)	83.2(4)	100.9(4)
S(1)—C(1)—S(2)	117.2(4)	118.5(5)	115.8(6)	118.0(8)	118.7(5)
S(1)—C(1)—N(1)	122.0(5)	119.8(6)	122.5(9)	119.3(7)	119.8(9)
S(2)—C(1)—N(1)	120.8(6)	121.6(6)	121.7(9)	122.7(7)	121.4(9)
S(3)—C(z)—S(4)	117.0(4)	118.6(4)	117.2(6)	119.0(6)	118.0(5)
S(3)—C(z)—N(2)	121.7(5)	119.5(5)	122.3(8)	119.8(6)	120.9(8)
S(4)—C(z)—N(2)	121.3(5)	121.8(6)	120.5(8)	121.2(6)	121.1(8)

a symmetry operation *i*: - *x*, - *y*, 1 - *z*

b symmetry operation *i*: 1 - *x*, - *y*, - *z*

c symmetry operation *i*: - *x*, - *y*, - *z*

d symmetry operation *i*: - *x*, - *y*, -1 - *z*

e symmetry operation *i*: -0.5 - *x*, -0.5 - *y*, -1 - *z*

by the S(1), S(3) and S(2)^{i} atoms in the direction of the more tightly bound axial atom, *i.e.* S(4). An alternate description of the coordination geometry about the cadmium atom would be one based on a square pyramidal geometry. The basal plane would be defined by the S(1)-S(4) atoms that lie 0.107(3) Å, -0.074(2) Å, 0.086(2) Å and -0.095(2) Å, respectively out of the S_4 least-squares plane and the axial position is occupied by the bridging S(2)' atom; the cadmium atom lies 0.6824(6) Å above this plane in the direction of the S(2)' atom. Within the "square" face, two edges are approximately 3 Å and the other two are slightly greater than 4 Å. The independent distances within the centrosymmetric rhombus are 2.601(2) Å and 2.814(2) Å and the two angles are 96.50(6)° (at cadmium) and 83.50(6)° (at the bridging sulfur atom) indicating an almost regular geometry. The disposition of the bridging dithiocarbamate ligands is such that they lie on either side of the rhombus. The motif described here for $[Cd(S_2CNiPr_2)_2]$ is the common motif for the $Cd(S_2CNR_2)_2$ structures (see later) and the observed conformation is repeated in other new structures reported herein.

The molecular structure of $[Cd(S_2CN(Et)Cy_2)_2]$, *i.e.* containing a disymmetric dithiocarbamate ligand, is shown in Fig. 6.2. The cadmium atom lies 0.2724(6) Å out of the plane through the S(1), S(3) and S(4)^{i} atoms in the direction of the S(2) atom; symmetry operation *i*: 1 - *x*, - *y*, - *z*. The dimensions within the rhombus are almost identical to those found in the structure of $[Cd(S_2CNiPr_2)_2]$, however, the angle subtended at the bridging sulfur atom has expanded by *ca* 3° resulting in a concomitant reduction in the angle subtended at cadmium. An interesting observation is that when the molecule is viewed along the Cd···Cd vector the four cyclohexyl groups are directed away from the remaining part of the dinuclear molecule, presumably to minimise steric repulsions.

The molecular structure of $[Cd(S_2CNCy_2)_2]$ is shown in Fig. 6.3. The cadmium atom lies 0.313(1) Å out of the plane defined by the S(1), S(3) and S(2)^{i} atoms in the direction of the S(4) atom (symmetry operation *i*: - *x*, - *y*, - *z*). A comparison of the geometric parameters defining the central rhombus shows that while the angles are similar

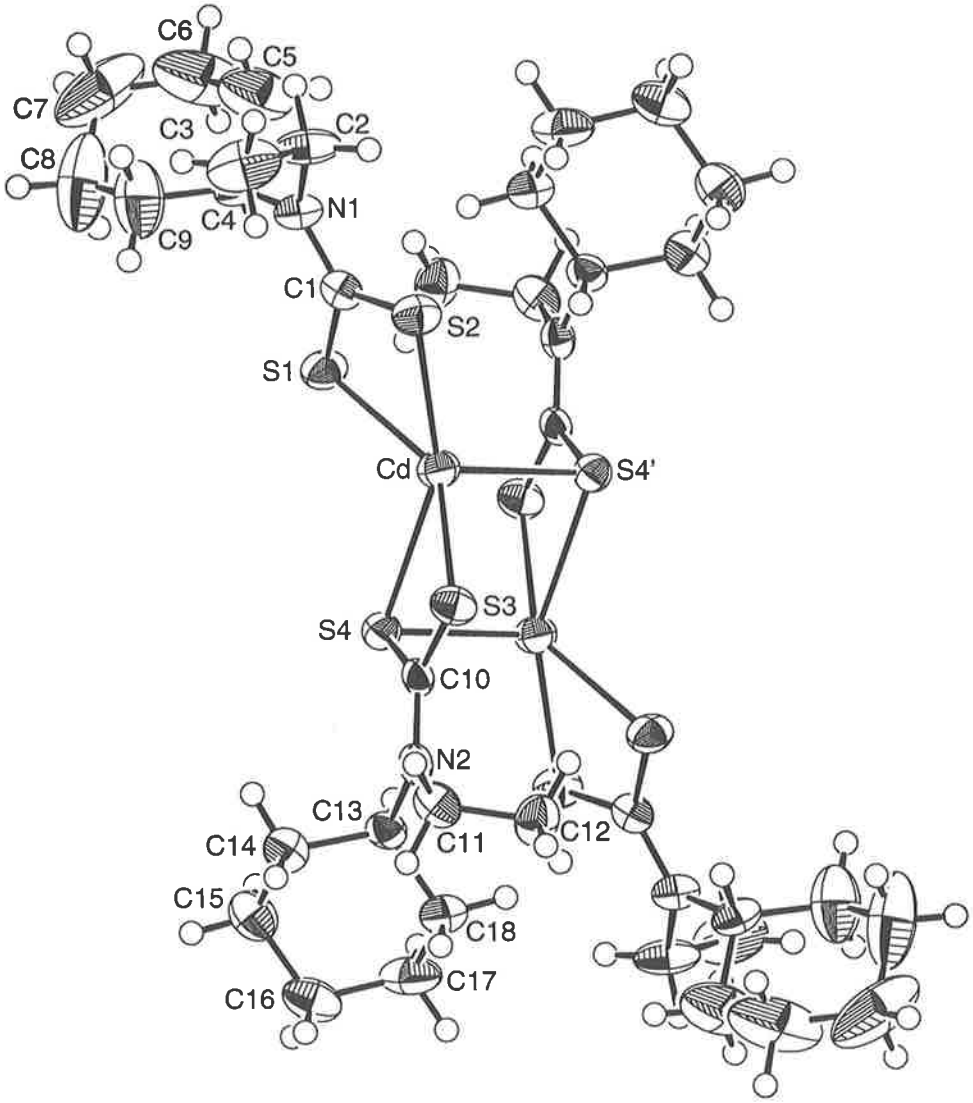

Fig. 6.2 Molecular structure of $[Cd(S_2CN(Et)Cy)_2]$ showing the crystallographic numbering scheme.

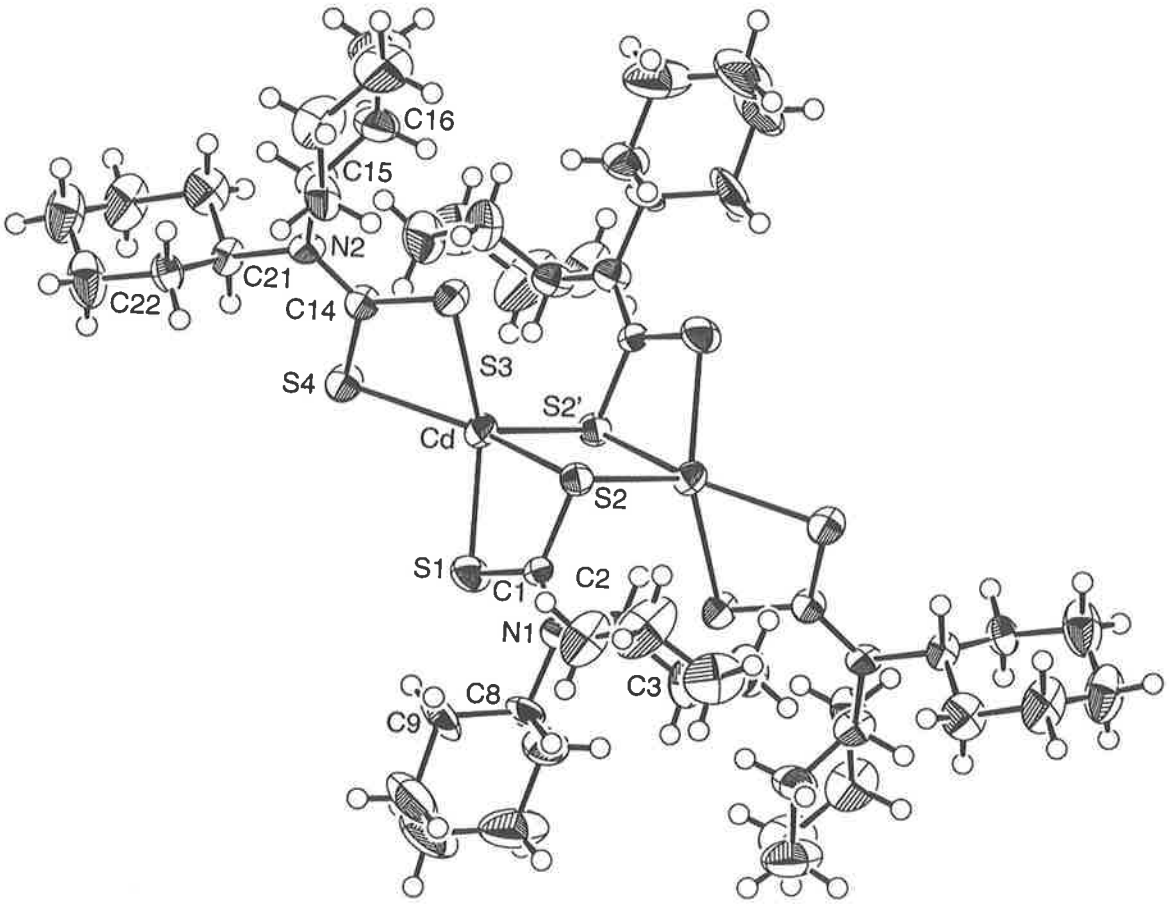

Fig. 6.3 Molecular structure of $[Cd(S_2CNCy_2)_2]$ showing the crystallographic numbering scheme.

to those found above, the disparity in the distances is significantly greater reflecting the stronger Cd-S bridge (2.551(3) Å) and the greater asymmetry in the chelating distances formed by the bridging dithiocarbamate ligand (ΔCd-S = 0.358(5) Å). The crystal chosen for analysis was found to contain solvent CH_2Cl_2 such that the ratio of dimer to solvent was 1:2, however, no significant interactions exist between them. In the lattice the solvent molecules reside in channels, parallel to the *c*-direction, defined by the cyclohexyl residues.

Two independent centrosymmetric molecules, labeled *a* and *b*, comprise the crystallographic asymmetric unit in the structure of [Cd(S_2CNiBu2)2], both of which are disposed about a center of inversion; the molecules are illustrated in Fig. 6.4. There are no chemically significant differences between the two molecules, however, minor conformational as well as crystallographic differences do exist. For example, the range of Cd-S distances of 2.566(4) Å to 2.780(3) Å involving the Cd(1) atom is smaller than that involving Cd(2) of 2.539(3) Å to 2.780(4) Å. The Cd(1) atom lies 0.0099(7) Å above the plane through the S(2)a, S(4)a and S(4)a^{i} atoms in the direction of the S(1) atom and similarly, the Cd(2) atom lies 0.001(1) Å above the S(1)b, S(3)b and S(4)b^{ii} trigonal plane in the direction of the S(2) atom (symmetry operation *i*: - *x*, - *y*, -1 - *z*; *ii*: -0.5 - *x*, - 0.5 - *y*, -1 - *z*). Of the structures described herein, the disparity in the distances within the rhombus is smallest in the two independent molecules of [Cd(S_2CNiBu2)2] with the disparity being least in molecule *b*, which reflects the greater strength of the Cd(1)-S(4)^{i} bridge (2.590(3) Å) *cf.* the Cd(2)-S(7) bridge (2.617(3) Å); the bridging sulfur atoms form experimentally equivalent distances when completing the chelate to their respective cadmium atom of 2.780(3) Å and 2.780(4) Å, respectively. A result of the strong bridge in molecule *a* is that the Cd···Cd separation of 3.525(2) Å is less than the equivalent separation in molecule *b* of 3.815(1) Å; neither interaction represents a significant bonding contact.

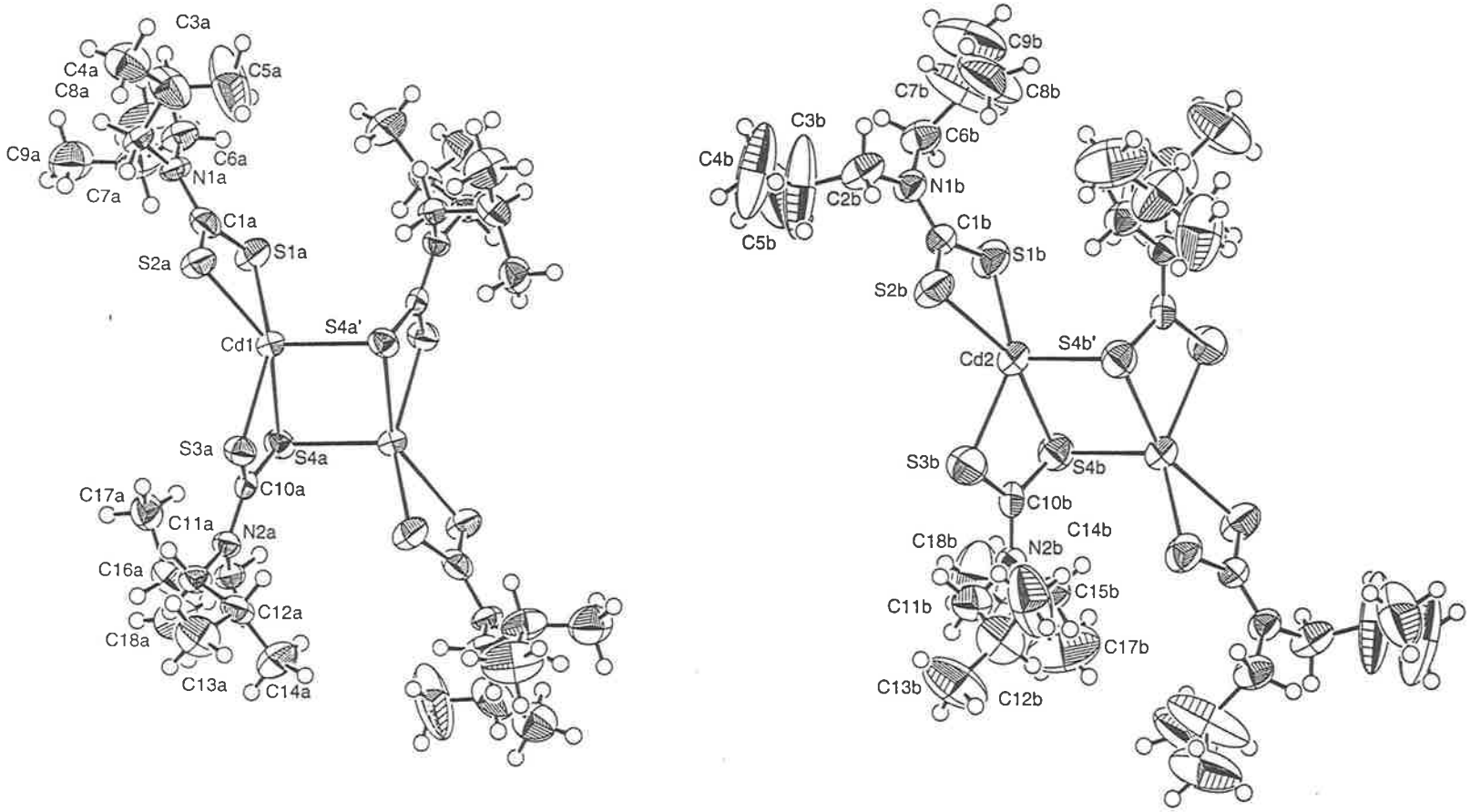

Fig. 6.4 Molecular structure of [Cd(S2CNiBu2)2] showing the crystallographic numbering scheme for the two independent dinuclear units.

6.2 SYSTEMATIC TRENDS FROM CRYSTALLOGRAPHY

The structures reported here are in essential agreement with two of those reported previously for $Cd(S_2CNR_2)_2$. Thus, when R = Et (Shugam, Agre, 1968; Domenicano, Torelli, Vaciago, Zambonelli, 1968) and $R_2 = (CH_2)_6$ (Agre, Shugam, 1972a), motif A is found (Fig. 6.5). Motif A' is found when R = nBu (Casas, Sánchez, Bravo, García-Fontán, Castellano, Jones, 1989), this being the only example. The major difference between the two motifs is seen in the relative disposition of the bridging dithiocarbamate ligands. In motif A, by virtue of the centre of inversion, the two bridging ligands lie on opposite sides of the central rhombus whereas in motif A', with crystallographic 2-fold asymmetry, the bridging dithiocarbamate ligands are on the same side of the molecule. In chair/boat notation, motif A represents the chair form of $Cd(S_2CNR_2)_2$ and motif A' represents the boat form.

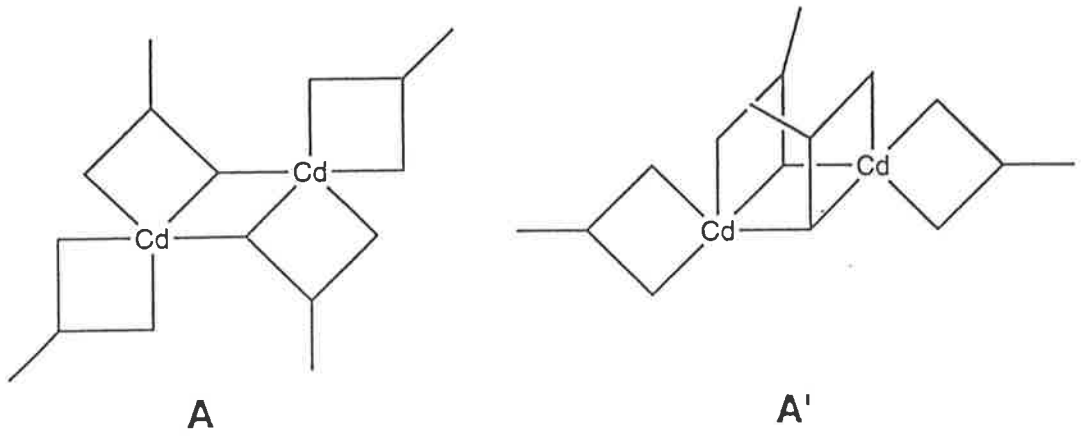

Fig. 6.5 Structural motifs for $Cd(S_2CNR_2)_2$: (A) R = Et, iPr, Cy, iBu and R_2 = (Et, Cy), $(CH_2)_6$; (A') R = Me

It is interesting to compare the three $Cd(S_2CNR_2)_2$ structures where R = Et, Cy and R_2 = (Et, Cy) with the respective zinc analogues (chapter 5). As described above, in the cadmium series all three structures adopt the same motif. A similar motif is found in the zinc structures with R = Et (Bonamico, Mazzone, Vaciago, Zambonelli, 1965;

Zvonkova, Khvatkina, Ivanova, 1968) and with R_2 = (Et, Cy) (chapter 5). The difference between these structures and those of the cadmium structures is found in the intramolecular bond formed by the bridging dithiocarbamate ligand which is not thought to represent a significant bonding interaction in $Zn(S_2CNR_2)_2$ and hence, the zinc atoms are considered four-coordinate, distorted tetrahedral. This difference between the structures is entirely consistent with the propensity of the larger cadmium atom to increase its coordination number. It is noteworthy that for $Zn(S_2CNCy_2)_2$ a new monomeric motif is formed. It was argued that the increasing bulk of the dithiocarbamate ligand, *i.e.* as ethyl groups were replaced by cyclohexyl groups, precluded association in the solid state. No such change in motif is evident in the cadmium series. It is likely that as the Cd-S distances are longer in the latter series of compounds, the increased bulk of the N-bound substituents may be accommodated more readily in the dinuclear structures Consistent with this argument is the fact that most of the $Cd(S_2CNR_2)_2$ compounds adopt the same motif for a wide range of R substituents. Although new $Cd(S_2COR)_2$ structures are not reported herein, for completeness a brief description of their structural motifs is given.

6.3 XANTHATE STRUCTURES

The molecular structures of $[Cd(S_2COiBu)_2]$ and $[Cd(S_2CO\text{-}3,3\text{-}Me_2Bu)_2]$ were determined, however, the refinements were not optimal and hence, full details are not reported. The structures were found to be similar to those reported previously for R = Et (Iimura, Ito, Hagihara, 1972) and R = nBu (Rietveld, Maslen, 1965). As shown for motif B in Fig. 6.6, the $Cd(S_2COR)_2$ structures are based on 2-dimensional sheets comprised of interconnected 16-membered $[Cd\text{-}S\text{-}C\text{-}S]_4$ rings as a result of all xanthate ligands being bridging. The coordination geometry about the cadmium atom in each structure is distorted tetrahedral. The other motif found for the $Cd(S_2COR)_2$ compounds is represented as motif C in Fig. 6.6 and is found for R = CH_2CH_2OMe (Abrahams, Hoskins, Winter, Tiekink, 1988). The structure is essentially monomeric and features a

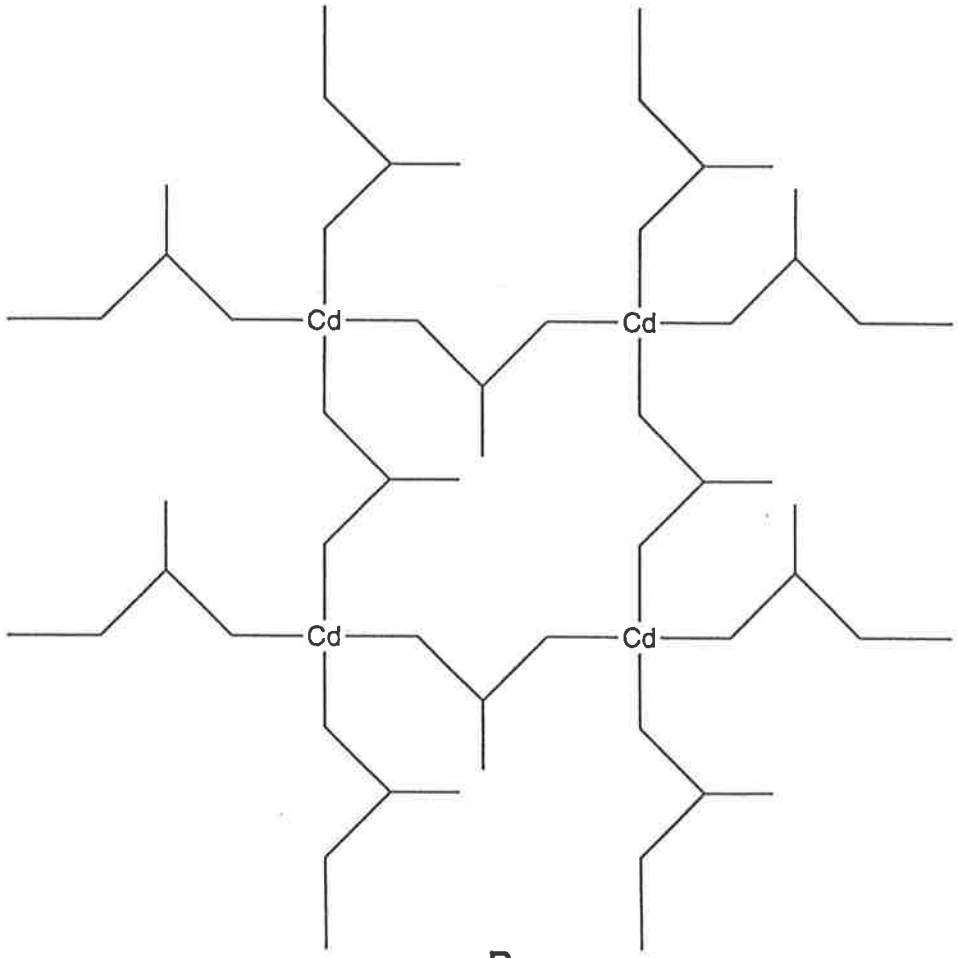

Fig. 6.6 Structural motifs for $Cd(S_2COR)_2$: (B) R = Et and iBu; (C) R = CH_2CH_2OMe.

distorted square planar geometry about the cadmium centre.

6.4 CONCLUSIONS

In the context of the structural diversity of the $M(S_2CNR_2)_2$ and $M(S_2COR)_2$ compounds where M = Zn, Cd and Hg (chapter 1) the small number of motifs found for the cadmium structures is remarkable. There are two very closely related motifs for $Cd(S_2CNR_2)_2$ and the xanthates generally adopt one motif only. This observation lends weight to the suggestion that there is no inherent electronic/steric reason to account for the structural diversity in these systems and that an analysis of crystal structure is often essential to rationalise molecular structure.

Chapter 7

MERCURY(II) BIS 1,1-DITHIOLATES

This chapter details the crystal and molecular structures of four new $Hg(S_2COR')_2$ and five new $Hg(S_2CNR_2)_2$ complexes. The structures are compared to literature analogues and an attempt is undertaken to explain their observed structural diversity.

7.1 XANTHATE STRUCTURES

The molecular structure of $[Hg(S_2COnBu)_2]$ showing the immediate geometry about the mercury atom is given in Fig. 7.1. Selected interatomic parameters are listed in Table 7.1. As with some of the other structures described in this chapter, the crystallographic analysis is not optimal owing to the difficulty in obtaining crystals. It is important to emphasise however, that the crystal and molecular structures have been determined unambiguously even if the standard deviations associated with some of the derived interatomic parameters are relatively high.

In $[Hg(S_2COnBu)_2]$ the mercury atom is situated on a crystallographic 2-fold axis and thus, the asymmetric unit comprises half a mercury atom and one xanthate ligand. The immediate environment about the mercury atom is defined by four sulfur atoms derived from four bridging xanthate ligands. The coordination geometry is distorted tetrahedral with the independent Hg-S distances being 2.413(5) Å and 2.842(5) Å. The range of S-Hg-S angles is 81.8(2)° to 150.8(3)° with the wider angle involving the more tightly bound S(1) atoms. Thus, to a first approximation the coordination geometry may be described as approximately linear. The weaker Hg-S(2) bonds are well within the sum of the van der Waals radii for these atoms of 3.3 Å (Bondi, 1964) and must be considered to represent significant bonding interactions. As can be seen from Fig. 7.1 the orientation of the xanthate ligand is such as to place the O(1) atom in close proximity to the mercury atom. The Hg···O(1) separation of 2.99(1) Å is approximately equal to

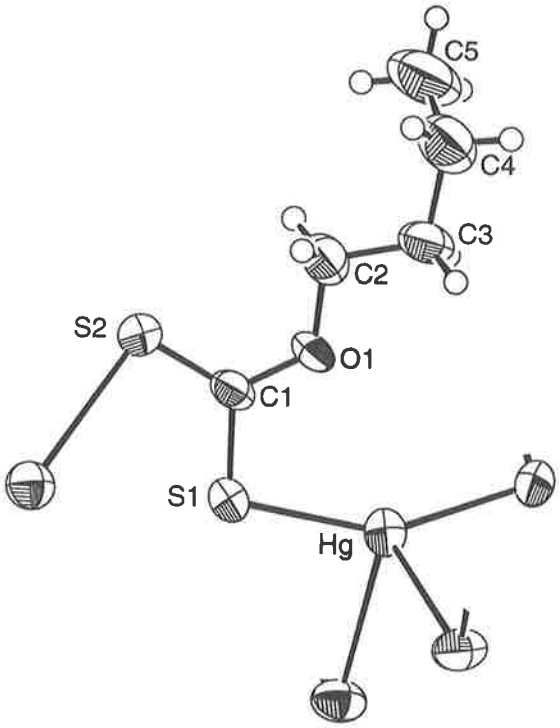

Fig. 7.1 Molecular structure of [Hg(S2COnBu)2] showing the mercury atom geometry and crystallographic numbering scheme.

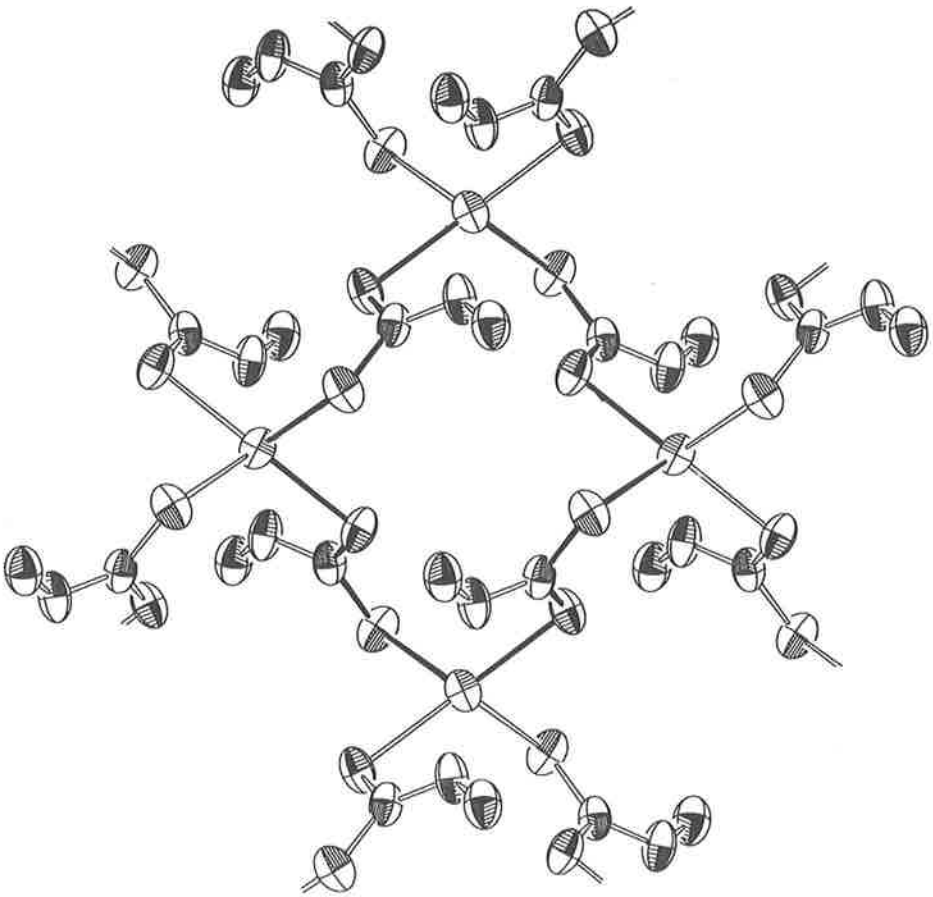

Fig. 7.2 The tetrameric unit in [Hg(S2COnBu)2]. For reasons of clarity only the α-carbon atom of the n-butyl group are shown.

Table 7.1 Selected interatomic parameters (Å, deg.) for $Hg(S_2COR)_2$, R = nBu, 3-MeBu, 3,3-Me_2Bu, and nPent

Parameter	R = nBu[a]	R = 3-MeBu[b]	R = 3,3-Me_2Bu[c]	R = nPent[d]
	y = 1'	y = 7	y = 8	y = 7
Hg—S(1)	2.413(5)	2.498(7)	2.505(3)	2.524(3)
Hg—S(2)	2.842(5)	2.507(7)	2.527(3)	2.534(3)
Hg—S(3)	2.413(5)	2.568(9)	2.531(3)	2.556(2)
Hg—S(4)	2.842(5)	2.546(6)	2.586(3)	2.572(3)
S(1)—C(1)	1.69(2)	1.70(2)	1.67(1)	1.69(1)
S(2)—C(1)	1.69(2)	1.69(2)	1.69(1)	1.68(1)
S(3)—C(y)	1.69(2)	1.66(2)	1.68(1)	1.71(1)
S(4)—C(y)	1.69(2)	1.72(3)	1.678(9)	1.69(1)
C(1)—O(1)	1.32(2)	1.35(3)	1.33(1)	1.34(1)
C(y)—O(2)	1.32(2)	1.30(3)	1.34(1)	1.31(1)

Table 7.1 Cont.

S(1)—Hg—S(2)	90.8(2)	116.6(3)	120.0(1)	116.62(9)
S(1)—Hg—S(3)	150.8(3)	115.5(3)	104.02(9)	120.51(9)
S(1)—Hg—S(4)	111.5(2)	98.5(3)	111.6(1)	99.1(1)
S(2)—Hg—S(3)	111.5(2)	99.4(3)	115.28(9)	97.24(8)
S(2)—Hg—S(4)	81.8(2)	121.1(3)	99.9(1)	117.5(1)
S(3)—Hg—S(4)	90.8(2)	106.2(3)	105.24(9)	106.68(8)
Hg—S(1)—C(1)	101.9(6)	107(1)	108.3(4)	103.3(4)
Hg—S(2)—C(1)	99.6(6)	104.3(7)	106.0(3)	107.1(4)
Hg—S(3)—C(y)	101.9(6)	105(1)	103.3(3)	99.2(4)
Hg—S(4)—C(y)	99.6(6)	100.5(9)	103.8(4)	103.8(4)
C(1)—O(1)—C	120(1)	117(2)	120.3(8)	120.0(8)
C(y)—O(2)—C	120(1)	121(2)	118.4(8)	120.0(9)
S(1)—C(1)—S(2)	121(1)	122(1)	124.0(6)	123.8(6)
S(1)—C(1)—O(1)	115(1)	116(1)	116.3(8)	119.7(8)
S(2)—C(1)—O(1)	122(1)	120(1)	119.7(7)	116.5(7)
S(3)—C(y)—S(4)	121(1)	122(1)	124.0(6)	124.3(6)

Table 7.1 Cont.

S(3)—C(y)—O(2)	115(1)	123(2)	114.8(7)	114.6(7)
S(4)—C(y)—O(2)	122(1)	114(1)	121.0(8)	121.0(8)

a S(2) ≡ S(2)^{i}, S(3) ≡ S(1)^{ii}, S(4) ≡ S(2)iii, symmetry operation *i* = 0.5 - *x*, 0.5 - y, 0.5 + *z*, ii = - *x*, *y*, 0.5 - z, *iii* -0.5 + *x*, 0.5 - *y*, - *z*

b S(2) ≡ S(2)^{i}, S(4) ≡ S(4)^{ii}, *i*: *x*, 1 + *y*, *z*, *ii*: *x*, - *y*, 0.5 + *z*

c S(2) ≡ S(2)^{i}, S(4) ≡ S(4)^{ii}, *i*: *x*, 1 + *y*, *z*, *ii*: *x*, 0.5 - *y*, 0.5 + *z*

d S(2) ≡ S(2)^{i}, S(4) ≡ S(4)^{ii}, *i*: *x*, -1 + *y*, *z*, *ii*: *x*, - *y*, 0.5 + *z*

the sum of the van der Waals radii of these atoms of 3.0 Å (Bondi, 1964) and while this may be responsible for some distortion in the mercury atom geometry it is not considered to represent significant bonding interactions. The structure may be thought of as being comprised of tetrameric 16-membered rings as illustrated in Fig. 7.2. Thus, each edge of a square of mercury atoms is bridged by a bidentate bridging xanthate ligand giving rise to a [-Hg-S-C-S-]$_4$ tetramer. The tetrameric units are linked to adjacent units in the same plane via the remaining bridging xanthate ligands leading to a 2-dimensional layer structure. As shown in Fig. 7.3 the layers are separated by hydrophobic interactions along the crystallographic *b*-axis. The structures of three other Hg(S_2COR)$_2$ compounds reported herein also conform to this motif; Table 7.1 gives selected interatomic parameters for these structures.

The coordination geometry in [Hg(S_2CO-3-MeBu)$_2$] is illustrated in Fig. 7.4. This structure may be related to that of [Hg(S_2COnBu)$_2$] by the addition of a methyl group at the 4-position of the n-butyl group. The mercury atom exists in a distorted tetrahedral geometry with the Hg-S bonds in the range of 2.498(7) Å to 2.568(9) Å and the S-Hg-S angles in the range of 98.5(3)° to 121.1(3)°. It is noteworthy that the range of Hg-S distances is smaller than that found in the structure of [Hg(S_2COnBu)$_2$] described above and as a consequence the range of tetrahedral angles is also narrower. In the context of the Hg···O contacts described for [Hg(S_2COnBu)$_2$], the Hg···O(1) and O(2)···Hg^i separations are 3.29(2) Å and 3.10(2) Å, respectively; symmetry operation *i*: 0.5 + *x*, 0.5 - *y*, 0.5 + *z*. Clearly, the weaker the Hg···O interactions, as in [Hg(S_2COCH$_2$CH$_2$C(H)Me$_2$)$_2$], the less pronounced the distortion from the ideal tetrahedral geometry. The addition of a second methyl group at the 4-position of the n-butyl group in [Hg(S_2COnBu)$_2$] does not result in a change in structural motif.

The coordination geometry about the mercury atom in [Hg(S_2CO-3,3-Me$_2$Bu)$_2$] is shown in Fig. 7.5. The range of Hg-S distances in this structure is 2.505(3) Å to 2.586(3) Å and the range of tetrahedral angles is 99.9(1)°

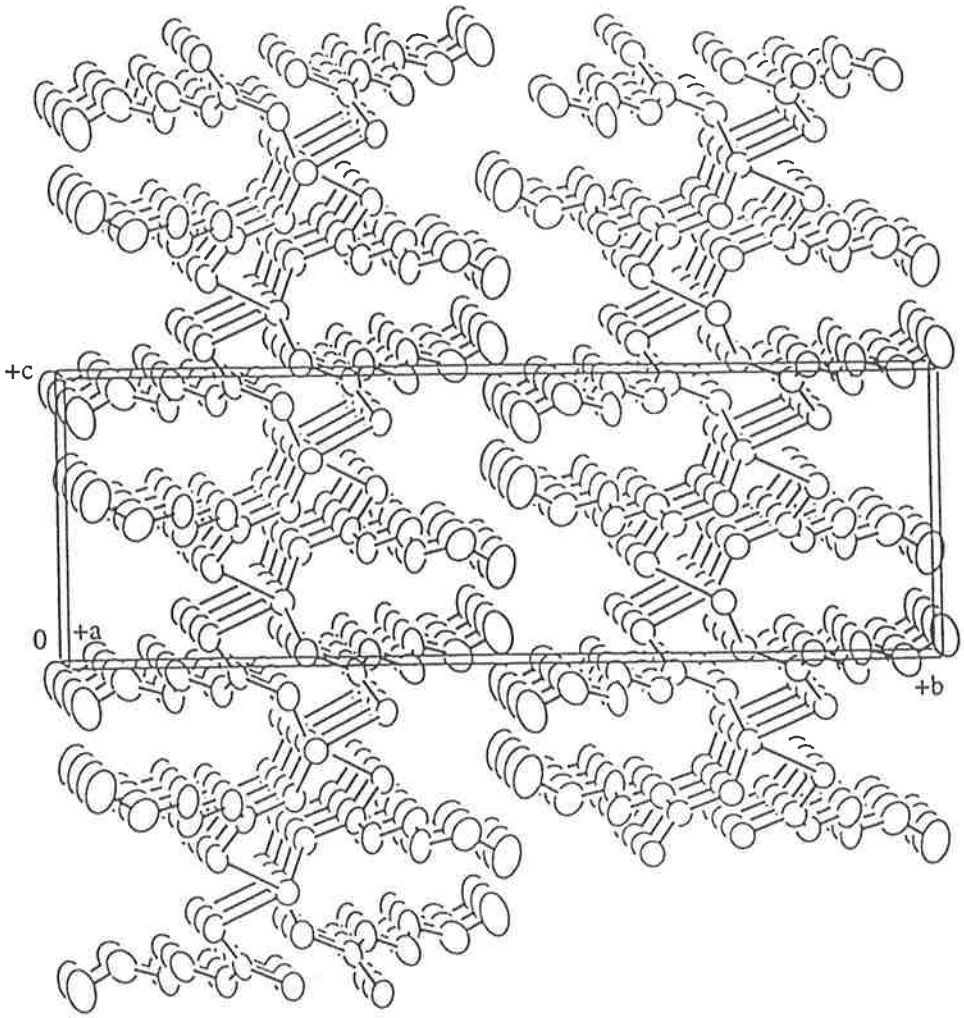

Fig. 7.3 The unit cell contents for $[Hg(S_2COnBu)_2]$.

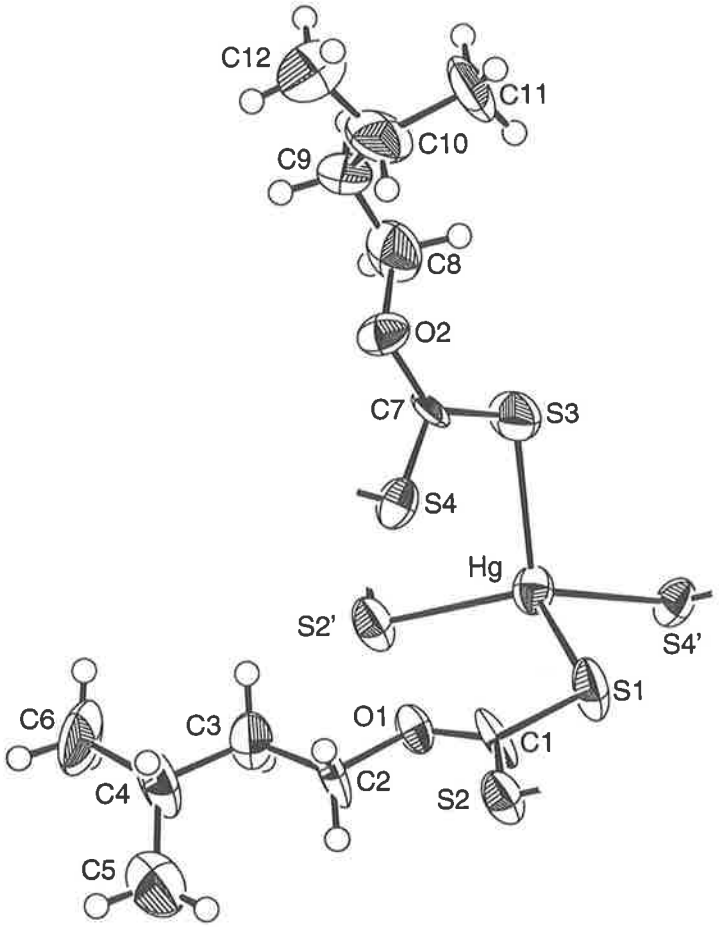

Fig. 7.4 Molecular structure of [Hg(S_2CO-3-MeBu)$_2$] showing the mercury atom geometry and crystallographic numbering scheme.

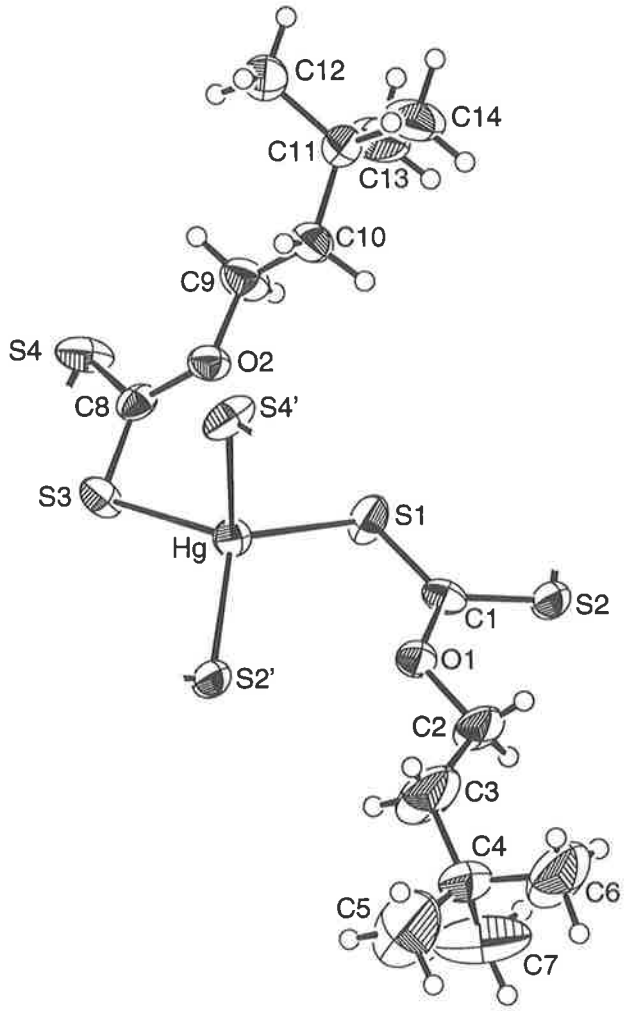

Fig. 7.5 Molecular structure of [Hg(S_2CO-3,3-Me_2Bu)$_2$] showing the mercury atom geometry and crystallographic numbering scheme.

to 120.0(1)°; the Hg···O(1) and Hg···O(2) separations are 3.277(7) Å and 3.116(7) Å, respectively. The immediate geometry about the mercury atom in the structure of the n-pentyl derivative, [Hg(S_2COnPent)$_2$], is shown in Fig. 7.6. This structure is isomorphous with that of [Hg(S_2CO-3-MeBu)$_2$]. As described above, a distorted tetrahedral geometry is found with Hg-S in the range 2.524(3) Å to 2.572(3) Å and S-Hg-S in the range 97.24(8)° to 120.51(9)°; Hg···O(1)^{i} and Hg···O(2) are 3.278(7) Å and 3.088(7) Å, respectively (symmetry operation *i*: *x*, -1 + *y*, *z*).

7.2 DITHIOBARBAMATE STRUCTURES

The molecular structure of [Hg(S_2CNMe$_2$)$_2$] is shown in Fig. 7.7. The mercury atom is situated about a crystallographic centre of inversion in the monoclinic space group C2/c and is coordinated by two dimethyldithiocarbamate ligands. The dithiocarbamate ligands each form disparate Hg-S interactions of 2.374(3) Å and 2.988(3) Å, respectively, indicating an asymmetric coordination mode of the dithiolate ligands. To a first approximation it could be argued that the mercury atom exists in a linear geometry (crystallographically imposed) defined by the two S(1) atoms. The Hg-S(2) separation of 2.988(3) Å is, however, well within the sum of the van der Waals radii of mercury and sulfur of 3.3 Å (Bondi, 1964) and must be considered a bonding interaction; the equivalence of the associated C-S bonds supports this interpretation. Thus, the mercury atom exists in a planar S_4 geometry based on a diamond shape. In addition to the immediate geometry about the mercury atom there are intermolecular Hg···S(1)' contacts of 3.404(5) Å (symmetry operation: -*x*, *y*, -0.5-*z*). These contacts, which occur parallel to the *c*-axis, are too long to be considered significant bonding interactions; see Fig. 7.8. The remaining parameters associated with the ligand are as expected. Similar to [Hg(S_2CNMe$_2$)$_2$] the following two complexes are monomeric but instead of a square planar arrangement around the mercury atom a tetrahedral coordination geometry is adopted.

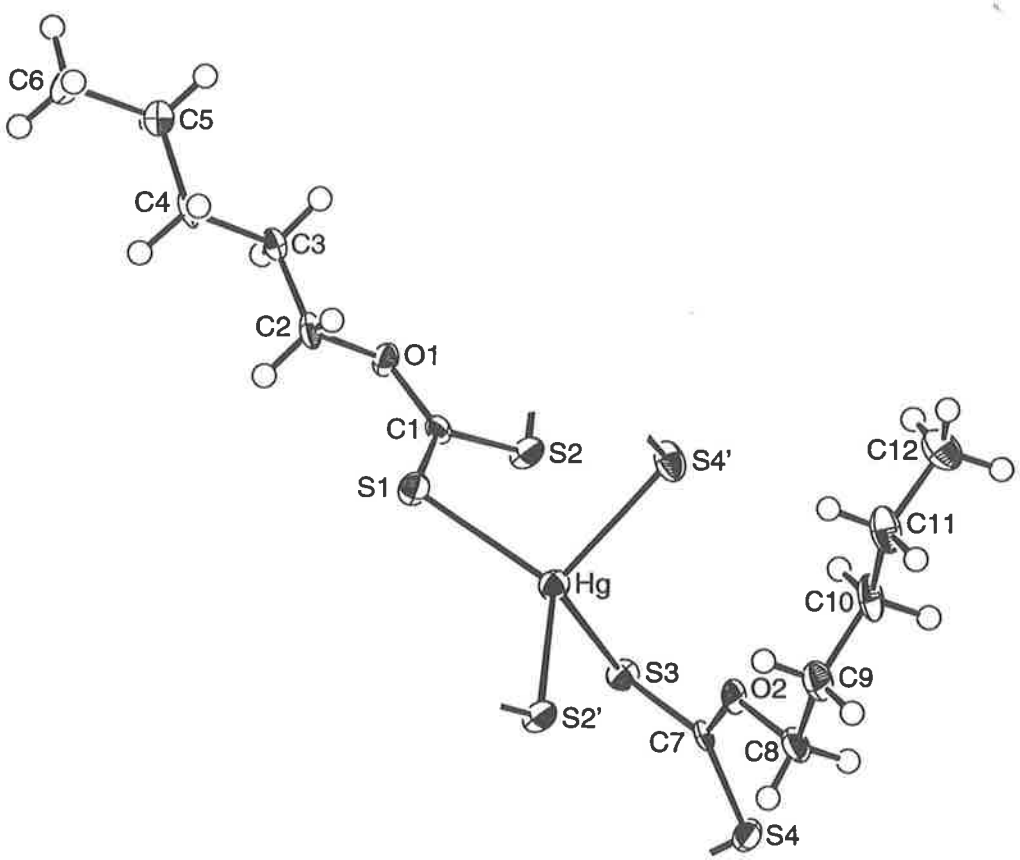

Fig. 7.6 Molecular structure of [Hg(S_2COnPent)2] showing the mercury atom geometry and crystallographic numbering scheme.

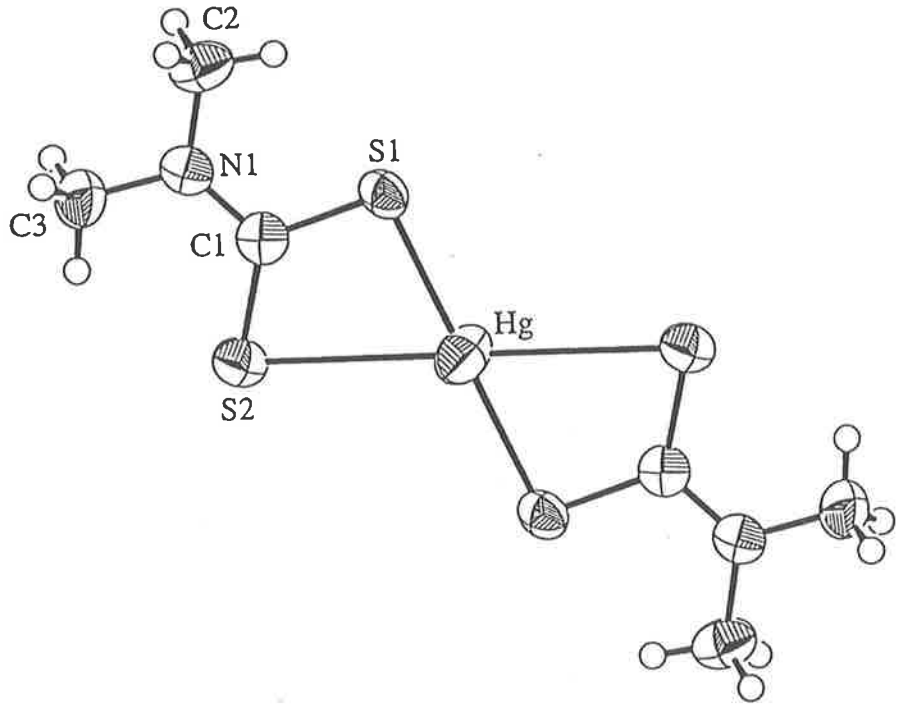

Fig. 7.7 Molecular structure of $[Hg(S_2CNMe_2)_2]$ showing the mercury atom geometry and crystallographic numbering scheme.

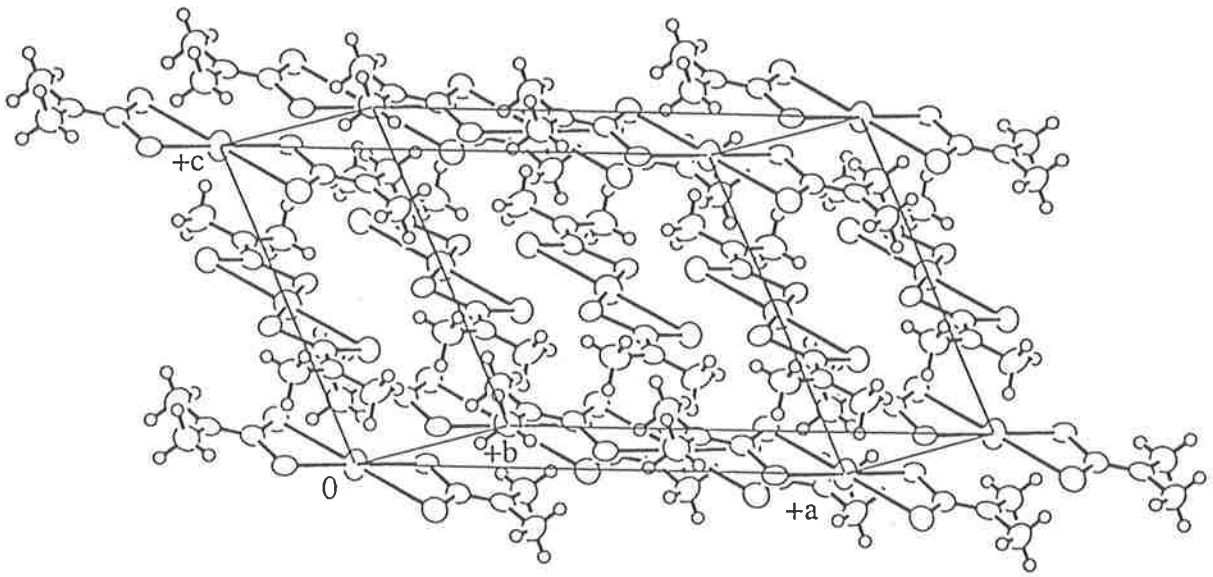

Fig. 7.8 Unit cell contents of $[Hg(S_2CNMe_2)_2]$.

The molecular structure of [Hg(S_2CNiBu$_2$)$_2$] is shown in Fig. 7.9 and selected interatomic parameters are collected in Table 7.2. The structure is monomeric and features a four coordinate mercury centre defined by four sulfur atoms derived from two asymmetrically coordinating dithiocarbamate ligands. To a first approximation the mercury atom geometry may be considered to be linear with the two more tightly bound sulfur atoms, Hg-S(1) is 2.416(2) Å and Hg-S(3) is 2.437(2) Å, subtending an angle of 153.92(8)°. However, the two less tightly held sulfur atoms, Hg-S(2) is 2.714(2) Å and Hg-S(4) is 2.710(3) Å, are at distances well within the sum of the van der Waals radii for these atoms of 3.3 Å (Bondi, 1964). Thus, the coordination geometry is best described as distorted tetrahedral with the distortions arising, in part, owing to the restricted bite angles of the dithiocarbamate ligands; the S(1)-Hg-S(2) and S(3)-Hg-S(4) chelate angles are 70.58(7)° and 70.44(7)°, respectively. The dihedral angle between the least-squares planes through the two independent HgS_2C moieties is 97.8°. Other parameters within the molecule are as expected. In the lattice there are no intermolecular contacts involving non-hydrogen atoms less than 3.6 Å, however, as can be seen from the unit cell contents illustrated in Fig. 7.10, centrosymmetrically related molecules are aligned so as to facilitate Hg···S(2)^{i} contacts of 3.728(3) Å; symmetry operation *i*: 0.5 - *x*, 0.5 - *y*, 0.5 - *z*. These contacts are longer than the sum of the van der Waals radii for these atoms and thus, can not be considered bonding interactions. However, the mode of packing gives a clue as to why different motifs may be adopted in the solid state as the orientation of the molecules is such as to *potentially* dimerise to yield a dimeric motif (see later). The monomeric motif found for [Hg(S_2CNiBu$_2$)$_2$] is repeated in the structure of [Hg(S_2CN(iPr)Cy)$_2$].

The crystallographic numbering scheme for [Hg(S_2CN(iPr)Cy)$_2$] is shown in Fig. 7.11 and geometric parameters are listed in Table 7.2. The Hg-S bond distances involving the S(1), S(2) dithiocarbamate ligand are equal within experimental error to the equivalent values found for [Hg(S_2CNiBu$_2$)$_2$]. By contrast, the Hg-S(3) and Hg-S(4) distances are longer (2.464(2) Å *cf.* 2.437(2) Å) and shorter (2.669(2) Å *cf.* 2.710(3)

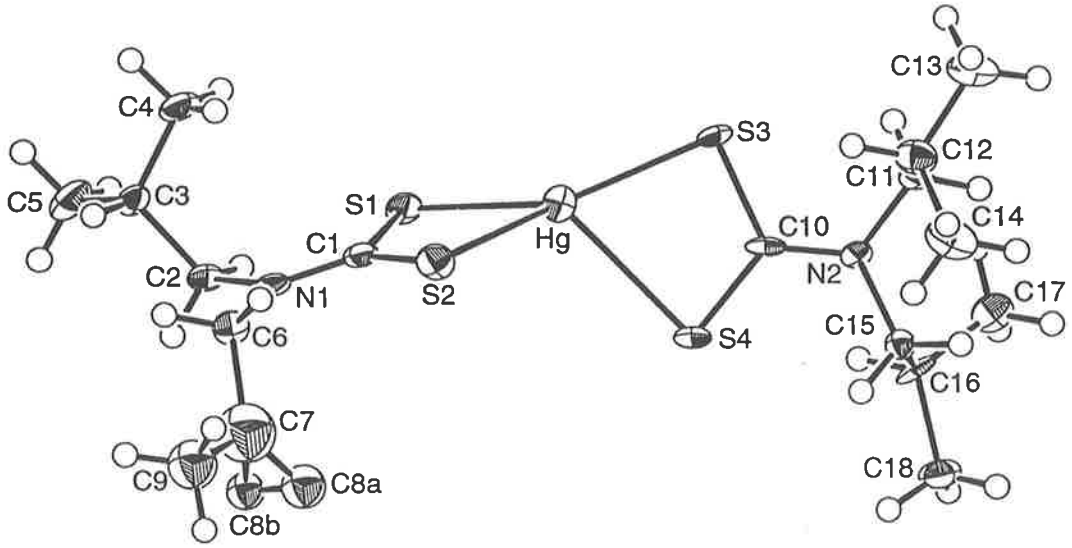

Fig. 7.9 Molecular structure of [Hg(S_2CNiBu_2)2] showing the crystallographic numbering scheme.

Table 7.2 Selected bond distances (Å) and angles (deg.) for $Hg(S_2CNR_2)_2$, R = iBu and R_2 = (iPr, Cy)

Parameter	R = iBu y = 10	R_2 = (iPr, Cy) y = 11
Hg-S(1)	2.416(2)	2.413(2)
Hg-S(2)	2.714(2)	2.724(2)
Hg-S(3)	2.437(2)	2.464(2)
Hg-S(4)	2.710(3)	2.669(2)
S(1)-C(1)	1.742(9)	1.751(7)
S(2)-C(1)	1.712(9)	1.706(7)
S(3)-C(y)	1.768(8)	1.749(7)
S(4)-C(y)	1.719(8)	1.709(7)
C(1)-N(1)	1.33(1)	1.329(8)
C(y)-N(2)	1.31(1)	1.326(8)
S(1)-Hg-S(2)	70.58(7)	69.99(6)
S(1)-Hg-S(3)	153.92(8)	159.99(6)
S(1)-Hg-S(4)	126.56(7)	129.10(6)
S(2)-Hg-S(3)	127.92(8)	112.30(6)
S(2)-Hg-S(4)	105.96(8)	110.77(6)
S(3)-Hg-S(4)	70.44(7)	69.80(6)
Hg-S(1)-C(1)	89.4(3)	90.7(2)
Hg-S(2)-C(1)	80.6(3)	81.7(2)
Hg-S(3)-C(y)	90.0(3)	89.0(2)
Hg-S(4)-C(y)	82.4(3)	83.3(2)
S(1)-C(1)-S(2)	118.9(5)	117.6(4)
S(1)-C(1)-N(1)	118.9(6)	119.5(5)
S(2)-C(1)-N(1)	122.3(6)	122.9(5)

Table 7.2 Cont.

S(3)-C(y)-S(4)	117.2(5)	116.5(4)
S(3)-C(y)-N(2)	119.1(6)	119.4(5)
S(4)-C(y)-N(2)	123.7(6)	124.1(5)

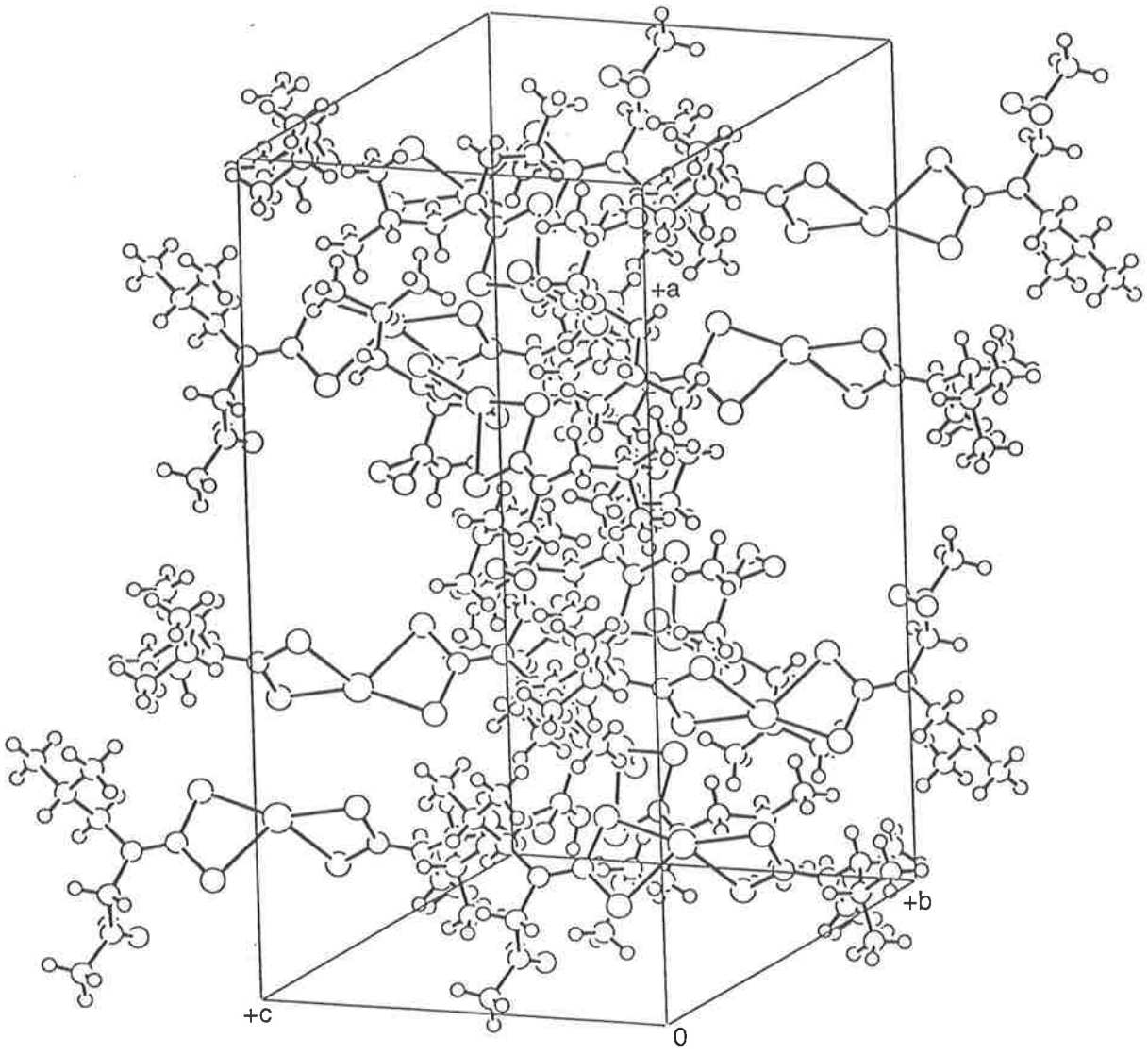

Fig. 7.10 Unit cell contents for [Hg(S_2CNiBu_2)2].

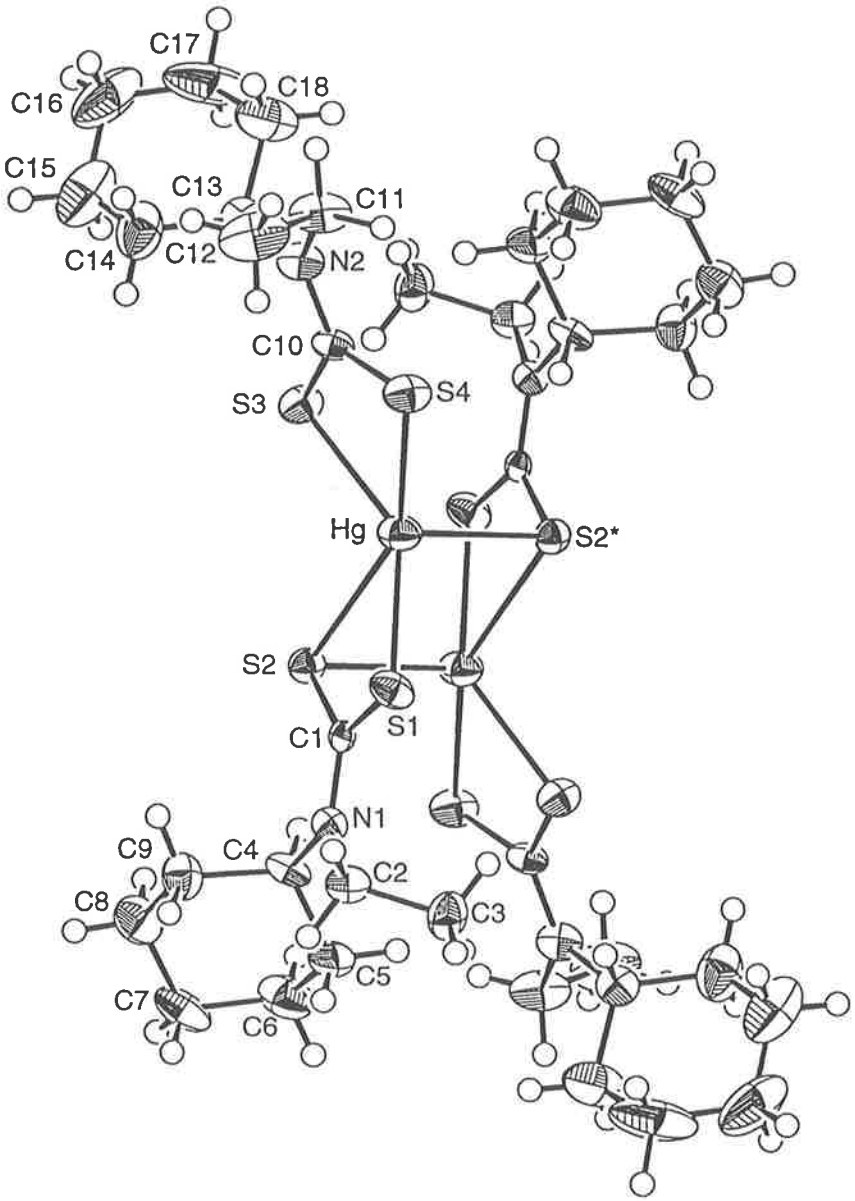

Fig. 7.11 Molecular structure of $[Hg(S_2CN(iPr)Cy)_2]$ showing the crystallographic numbering scheme.

Å), respectively. The difference in asymmetry also influences the angles about the mercury atom so that the S(2)-Hg-S(3) angle of 112.30(6)° is approximately 15° more acute compared with that found in [Hg(S_2CNiBu_2)$_2$]. The dihedral angle between the least-squares planes through each of the HgS_2C moieties was calculated to be 84.0°. In the lattice there is an intermolecular interaction between the S(1) and symmetry related C(11)^i atom of 3.484(7) Å; symmetry operation *i*: - *x*, - *y*, - *z*. As can be seen from the unit cell contents illustrated in Fig. 7.12, the lattice comprises *ac*-layers of [Hg(S_2CN(iPr)Cy)$_2$] molecules separated by hydrophobic interactions. Rows of molecules run parallel to the *c*-axis and are linked to adjacent rows (in the *a*-direction) via the contact mentioned above as well as Hg···S(4)^i and Hg···S(3)^i contacts of 3.753(2) Å and 3.927(2) Å. It is likely that the steric pressure exerted by the intermolecular interactions is responsible for the changes in the molecule mentioned above. Further, the nature of the Hg···S interactions implies a potential for significant association via Hg···S contacts. Whereas the structures of [Hg(S_2CNiBu_2)$_2$] and [Hg(S_2CN(iPr)Cy)$_2$] are essentially monomeric in the solid state, those of [Hg(S_2CN(Et)Cy)$_2$] and [Hg(S_2CNnBu_2)$_2$] are dinuclear.

The molecular structure of [Hg(S_2CN(Et)Cy)$_2$] is shown in Fig. 7.13 and selected interatomic parameters are listed in Table 7.3. The structure is dimeric, being centered about a crystallographic center of inversion. The mercury atom is coordinated by two chelating dithiocarbamate ligands, one of which forms relatively symmetric Hg-S bond distances, *i.e.* Hg-S(3) 2.508(2) Å and Hg-S(4) 2.707(2) Å, and the other which forms grossly disymmetric bonds, *i.e.* Hg-S(1) 2.452(3) Å and Hg-S(2) 3.047(2) Å. The second mentioned dithiocarbamate ligand also bridges the symmetry related Hg^i atom, via the S(2) atom, forming a bond distance of 2.640(3) Å; symmetry operation *i*: - *x*, - *y*, 1 - *z*. The mercury atom is best described as existing in a distorted trigonal bipyramidal geometry with the trigonal plane being defined by the S(1), S(2)^i and S(3) atoms and the axial positions by the less tightly bound S(2) and S(4) atoms. The mercury atom lies 0.3656(4) Å out of the plane in the direction of the S(4) atom which forms the

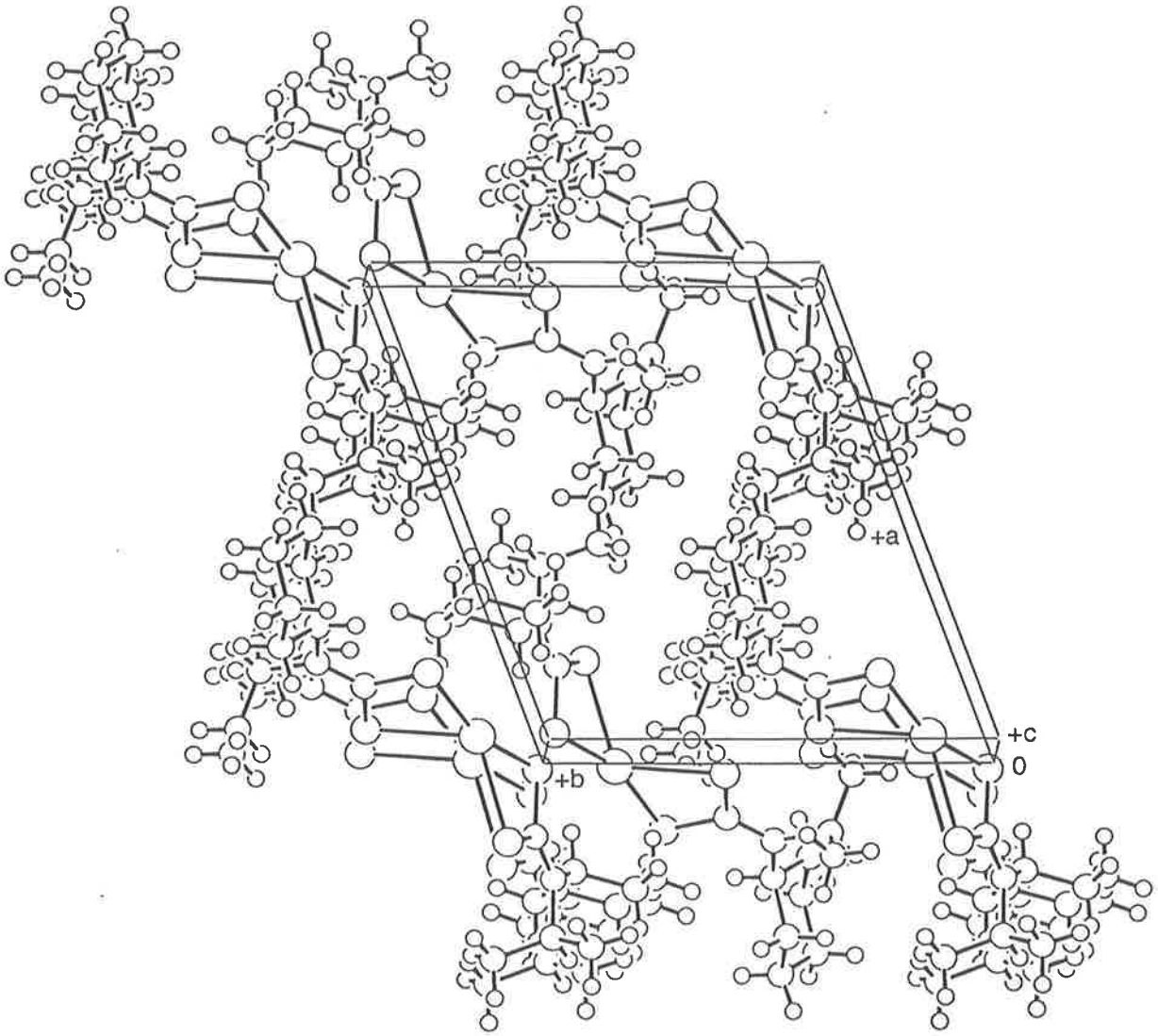

Fig. 7.12 Unit cell contents for $[Hg(S_2CN(iPr)Cy)_2]$.

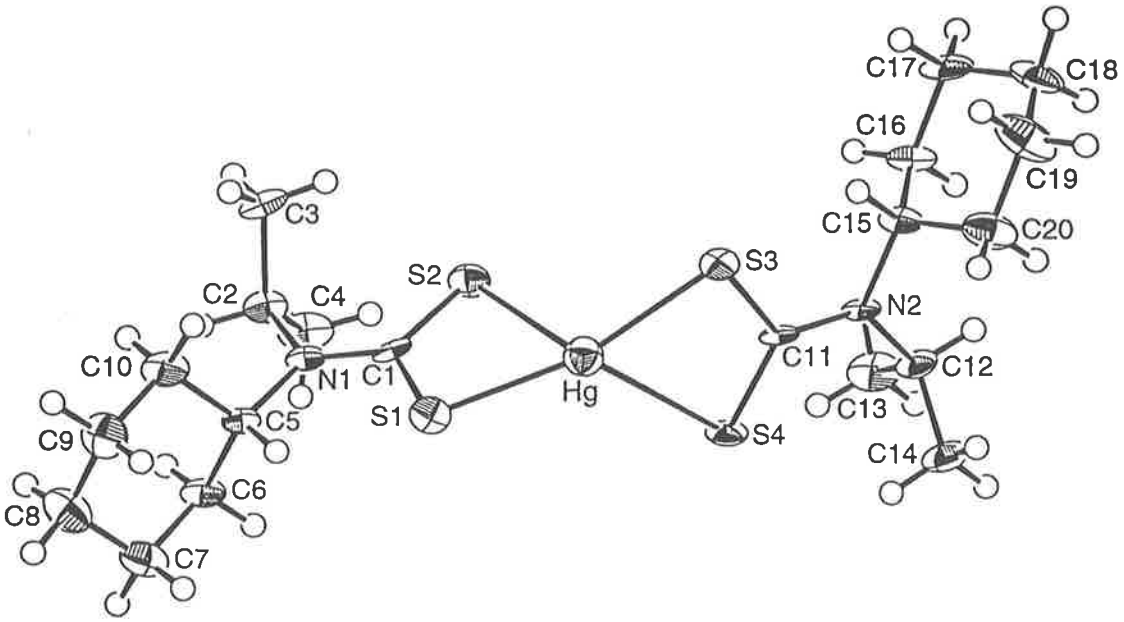

Fig. 7.13 Molecular structure of [Hg(S_2CN(Et)Cy)2] showing the crystallographic numbering scheme.

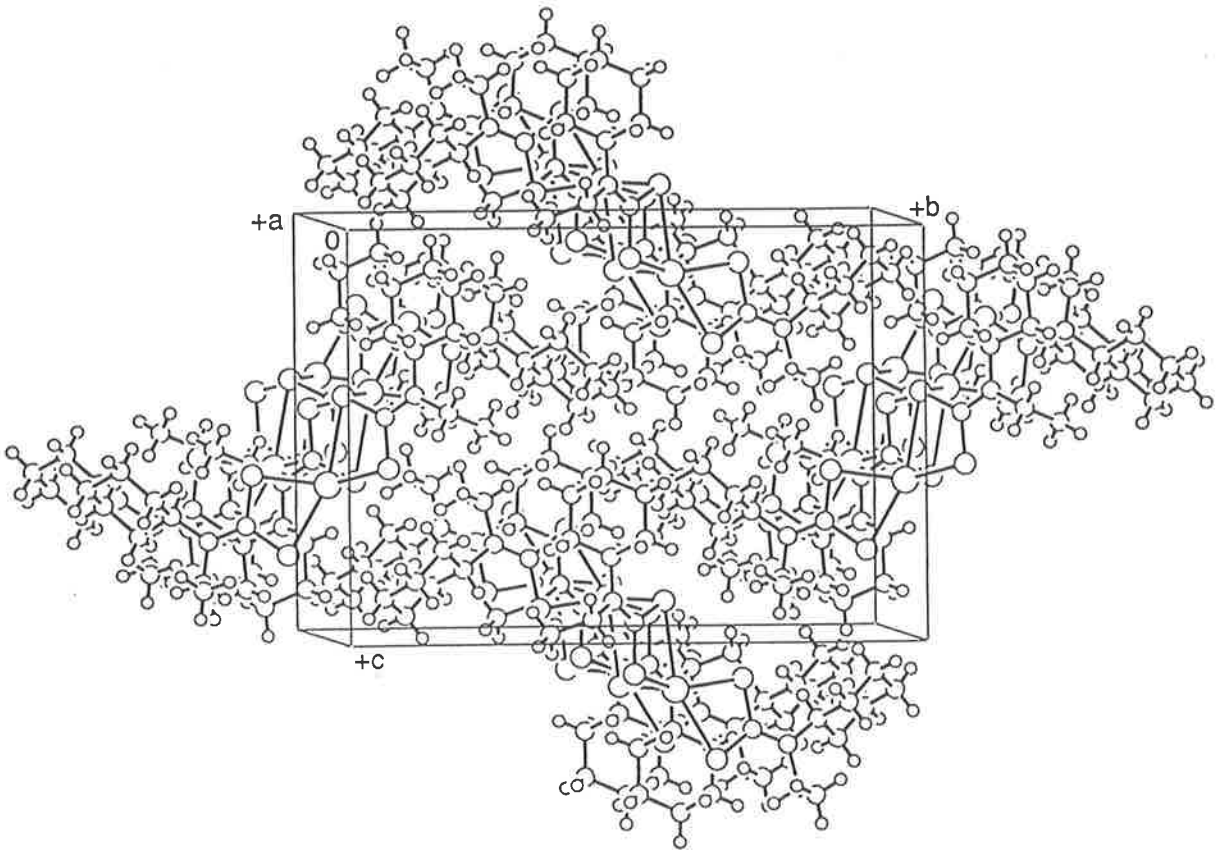

Fig. 7.14 Unit cell contents for [Hg(S_2CN(Et)Cy)2].

Table 7.3 Selected bond distances (Å) and angles (deg.) for $Hg(S_2CNR_2)_2$, R_2 = (Et, Cy) and R = nBu

Parameter	R_2 = (Et, Cy)	R = nBu
	y = 10	y = 11
Hg-S(1)	2.452(3)	2.457(4)
Hg-S(2)	3.047(2)	3.160(4)
Hg-S(2)'	2.640(3)[a]	2.601(4)[b]
Hg-S(3)	2.508(2)	2.543(4)
Hg-S(4)	2.707(2)	2.632(5)
S(1)-C(1)	1.747(8)	1.72(2)
S(2)-C(1)	1.724(8)	1.74(2)
S(3)-C(y)	1.737(8)	1.70(2)
S(4)-C(y)	1.708(9)	1.72(2)
C(1)-N(1)	1.31(1)	1.32(2)
C(y)-N(2)	1.34(1)	1.35(2)
S(1)-Hg-S(2)	64.99(7)	63.2(1)
S(1)-Hg-S(2)'	101.51(9)	109.7(1)
S(1)-Hg-S(3)	142.32(8)	127.7(1)
S(1)-Hg-S(4)	120.83(7)	121.3(2)
S(2)-Hg-S(2)'	91.44(6)	94.3(1)
S(2)-Hg-S(3)	92.33(7)	89.2(1)
S(2)-Hg-S(4)	155.70(7)	156.4(1)
S(2)'-Hg-S(3)	109.11(8)	116.2(1)
S(2)'-Hg-S(4)	109.04(7)	104.4(1)
S(3)-Hg-S(4)	69.06(7)	69.8(1)
Hg-S(2)-Hg'	88.56(6)	84.9(1)
Hg-S(1)-C(1)	97.0(3)	99.9(5)

Table 7.3 Cont.

Hg-S(2)-C(1)	78.2(3)	76.4(6)
Hg-S(2)'-C(1)'	98.1(2)	99.1(5)
Hg-S(3)-C(y)	89.1(3)	86.7(6)
Hg-S(4)-C(y)	83.3(3)	83.5(6)
S(1)-C(1)-S(2)	119.4(4)	120.4(9)
S(1)-C(1)-N(1)	118.0(6)	118(1)
S(2)-C(1)-N(1)	122.6(6)	120(1)
S(3)-C(y)-S(4)	118.5(5)	120(1)
S(3)-C(y)-N(2)	119.5(7)	121(1)
S(4)-C(y)-N(2)	121.9(6)	118(1)

a symmetry operation: - *x*, - *y*, 1 - *z*

b symmetry operation: - *x*, *y*, 0.5 - *z*

stronger of the Hg-S_{axial} bonds. An alternate description of the coordination geometry would be one based on a square pyramid where the S(1), S(2), S(3) and S(4) atoms define the basal plane (deviations from the least-squares plane 0.092(2) Å, -0.087(2) Å, 0.120(2) Å and -0.110(3) Å, respectively) with the mercury atom lying 0.6047(4) Å out of the plane in the direction of the axial $S(2)^i$ atom. If the weaker of the Hg-S bonds, *i.e.* Hg-S(2), is not considered a bonding interaction, the coordination geometry could be described as distorted tetrahedral in a way similar to that described for two of the above monomeric structures. In the low coordinate description the range of tetrahedral angles would be 69.06(7)° (the chelate angle) to 142.32(8)° (the angle involving the two more tightly bound sulfur atoms), again indicating a tendency for linear coordination. A view of the unit cell contents is given in Fig. 7.14 from which it can be seen that the lattice comprises columns of dimeric units aligned in the *a*-direction which are separated from each other by hydrophobic interactions. Within the columns there are no significant Hg···S interactions.

The structure of $[Hg(S_2CNnBu_2)_2]$, illustrated in Fig. 7.15, is also dimeric, however it differs from that of $[Hg(S_2CN(Et)Cy)_2]$ in the orientation of the bridging dithiocarbamate ligands. Selected interatomic parameters are listed in Table 7.3. Compared with the structure of $[Hg(S_2CN(Et)Cy)_2]$, the Hg-$S(2)^i$ and Hg-S(4) bond distances in $[Hg(S_2CNnBu_2)_2]$ contract as does the S(1)-Hg-S(3) angle; symmetry operation *i*: - *x*, *y*, 0.5 - *z*. The coordination geometry is best described as being based on a trigonal bipyramid so that the mercury atoms lies 0.3680(6)Å out of the trigonal plane defined by the S(1), $S(2)^i$ and S(3) atoms in the direction of the S(4) atom. The major difference between the structure of $[Hg(S_2CNnBu_2)_2]$ and that of $[Hg(S_2CN(Et)Cy)_2]$ rests with the relative disposition of the two bridging dithiocarbamate ligands. In $[Hg(S_2CN(Et)Cy)_2]$, by virtue of the center of inversion, the bridging dithiocarbamate ligands lie on opposite sides of the Hg_2S_2 rhombus which is constrained by symmetry to be planar. In the structure of $[Hg(S_2CNnBu_2)_2]$, the two halves of $[Hg(S_2CNnBu_2)_2]_2$ are related by a crystallographic 2-fold axis so that the

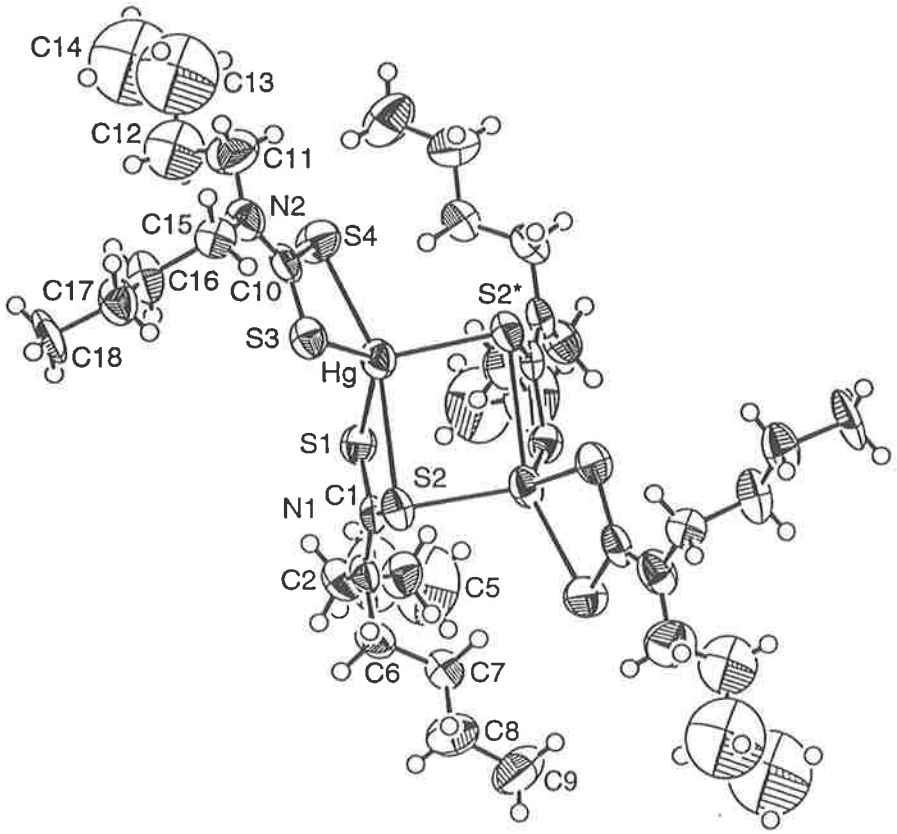

Fig. 7.15 Molecular structure of [Hg(S_2CNnBu_2)2] showing the crystallographic numbering scheme.

bridging dithiocarbamate ligands lie on the same side of the molecule. Further, the Hg_2S_2 rhombus is not planar as seen in the dihedral angle of 165.6° formed between the two HgS_2 triangles. There are no contacts involving non-hydrogen atoms in the lattice less than 3.6 Å.

7.3 SYSTEMATIC TRENDS FROM CRYSTALLOGRAPHY

In summary, the $Hg(S_2COR)_2$ structures adopt three distinct motifs in the solid state as illustrated in Fig. 7.16 These range from (A) helical polymeric chains, (B) 2-dimensional layers structures, and (C) 3-dimensional network structures. Only one example is known to adopt motif A, namely $[Hg(S_2COMe)_2]$ (Tiekink, 1987). In motif A, neighbouring mercury atoms are linked by bidentate bridging xanthate ligands leading to helical chains. The 3-coordinate geometry is completed by a monodentate xanthate ligand with the pendent sulfur atom of this ligand not participating in coordination to mercury. The geometry about the mercury atom in $[Hg(S_2COMe)_2]$ is best described as T-shaped. The bridging Hg-S distance of 2.924(3) Å is significantly longer than the other Hg-S bond distances of 2.365(3) Å and 2.383(3) Å and hence, to a first approximation, the geometry about the mercury atom may be considered linear with a S_{short}-Hg-S_{short} angle of 164.9(1)°. Motif B is the predominant motif for compounds of the general formula $Hg(S_2COR)_2$ being found for R = Et (two polymorphs, Watanabe, 1977; Chieh, Moynihan, 1980), nPr (Hounslow, Tiekink, 1991), nBu, 3-MeBu, 3,3-Me_2Bu, and nPent. It should be noted that the tendency for linear coordination by the two most strongly bound sulfur atoms as described above for $[Hg(S_2COnBu)_2]$ are also found in the R = Et and nPr structures. Finally one example is known for motif C, namely that of $[Hg(S_2COiPr)_2]$ (Watanabe, 1981). In this structure, the 16-membered rings of motif B are retained. However, changes in the mode of association of the remaining xanthate ligands are found. For two of the mercury centres, the coordination geometry is completed by chelating xanthate ligands. The other mercury atoms are linked to neighbouring 16-membered rings above and below the original 16-

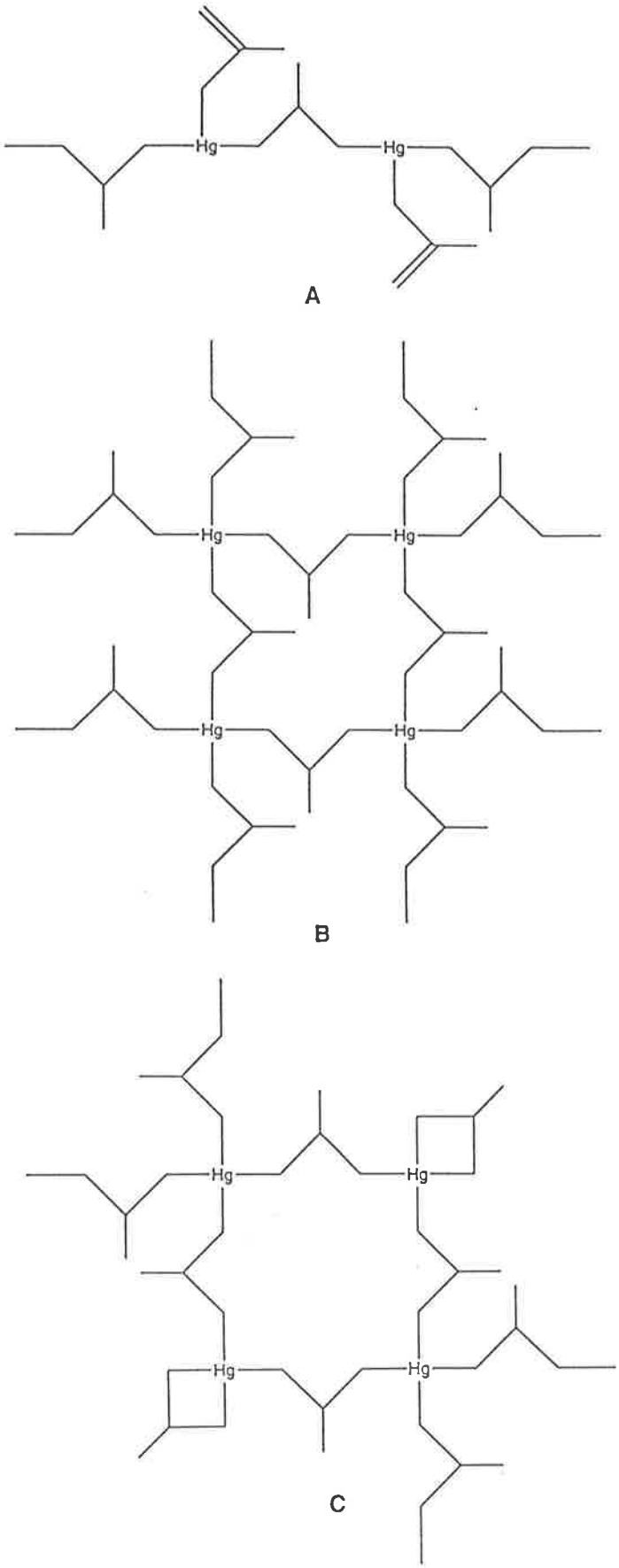

Fig. 7.16 Structural motifs for $Hg(S_2COR)_2$. (A) R = Me. (B) R = Et, nPr, nBu, $CH_2CH_2C(H)Me_2$, $CH_2CH_2CMe_3$, nPent. (C) R = iPr.

membered ring, *i.e.* not laterally leading to a layer structure as in motif B, but leading to a 3-dimensional network structure.

In previous systematic analyses of the $Ni(S_2COR)_2$ (chapter 3) and $Te(S_2COR)_2$ (chapter 4) structures it was demonstrated that no electronic property associated with the nature of R influenced the geometric parameters about the central atom. Nor was there any evidence of a steric effect operating in these systems as a consistent structural motif was found for each series. Given the above, it is clear that the possible role of crystal packing effects must be taken into consideration when rationalising the different motifs found for $Hg(S_2COR)_2$.

To a first approximation the $Hg(S_2COR)_2$ species may be considered as essentially monomeric with linear S-Hg-S arrangements. That is, the valency about the central mercury atom is essentially satisfied by the formation of two strong Hg-S bonds. The appearance of different motifs occur as a result of the different modes of association of the second sulfur atom of each xanthate ligand. In the R = Me structure, *i.e.* motif A, association in the solid state is effected by the weak Hg-S intermolecular bridge leading to the formation of helical chains. As the R substituent becomes larger, the packing arrangement of stacked rods as for R = Me no longer maximises the intermolecular forces and hence, a new arrangement is found, *i.e.* motif B. The intermolecular associations are maximised by the presence of bidentate bridging xanthate ligands leading to 2-dimensional sheets held together by hydrophobic interactions. The presence of four near-neighbour sulfur atoms, albeit at longer Hg-S separations, results in significant distortions from linear geometries. When the R group becomes too bulky as for R = iPr, with branching at the alpha carbon atom, the layer structure is disrupted into 16-membered rings that are now interconnected above and below leading to a 3-dimensional structure, *i.e.* motif C. A simple steric argument along the lines: greatest bulk is equated with minimal association, is not valid to explain the appearance of the different motifs for $Hg(S_2COR)_2$ as the reverse is true. However, a consideration of the packing requirements gives a qualitative model for the observed structural diversity.

For the $Hg(S_2CNR_2)_2$ complexes it is clear that five quite distinct motifs are found. A diagram representing the different motifs for the $Hg(S_2CNR_2)_2$ compounds is presented in Fig. 7.17. Motif A is monomeric and features a distorted tetrahedral geometry about the mercury atom as described for $Hg(S_2CNR_2)_2$, R = iBu, iPr (Ito, Iwasaki, 1979) and R_2 = (iPr, Cy). Further, although full details are not reported owing to a less than optimal refinement, the structure $[Hg(S_2CNCy_2)_2]$ has been determined in our laboratories and shown to adopt motif A; the structure is isomorphous with the zinc analogue (chapter 5). A second monomeric motif, B, is known which also features a four-coordinate geometry but this one is square planar. This has been found for R = Et (β-form, two determinations: Iwasakai, 1973; Healy, White, 1973) and R = Me. To a first approximation motif B may be considered to comprise a linear mercury atom geometry (the structures are centrosymmetric) defined by two tightly held sulfur atoms (Hg-S(1) is 2.398(4) Å and 2.374(3) Å for R = Et (β-form) and Me, respectively). These are significantly shorter than the weaker Hg-S(2) bonds of 2.965(4) Å and 2.988(3) Å, respectively. In both of the structures there are also intermolecular Hg···S interactions, above and below the HgS_4 plane, of 3.292(5) Å for R = Et and 3.404(5) Å for R = Me, distances considered too long to represent significant bonding interactions. Two dimeric motifs are also known. The first, C, is centrosymmetric and is found for R = Et (α-form, Iwasaki, 1973), for R_2 = Et, Cy and for NR_2 = 4-methyl-piperidine (Benedetti, Fabretti, Preti, 1988). The second, D, with crystallographic 2-fold symmetry, is found for R = nBu. The final motif, E, is found for the parent dithiocarbamate, *i.e.* $[Hg(S_2CNH_2)_2]$ (Chieh, Cheung, 1981). This structure exists as 2-dimensional sheets where 8-membered $[Hg\text{-}S\text{-}C\text{-}S]_2$ rings are connected to neighbouring rings via bridging dithiocarbamate ligands. From the foregoing it is clear that remarkable structural diversity exists for the $Hg(S_2CNR_2)_2$ compounds, the challenge remains to rationalise the appearance of the different motifs.

Firstly, and as mentioned for the xanthate structures above, it is important to recall the results of earlier systematic studies/analyses of the bis(dithiocarbamate) structures of nickel(II) and tellurium(II) where changes in molecular geometry and geometric

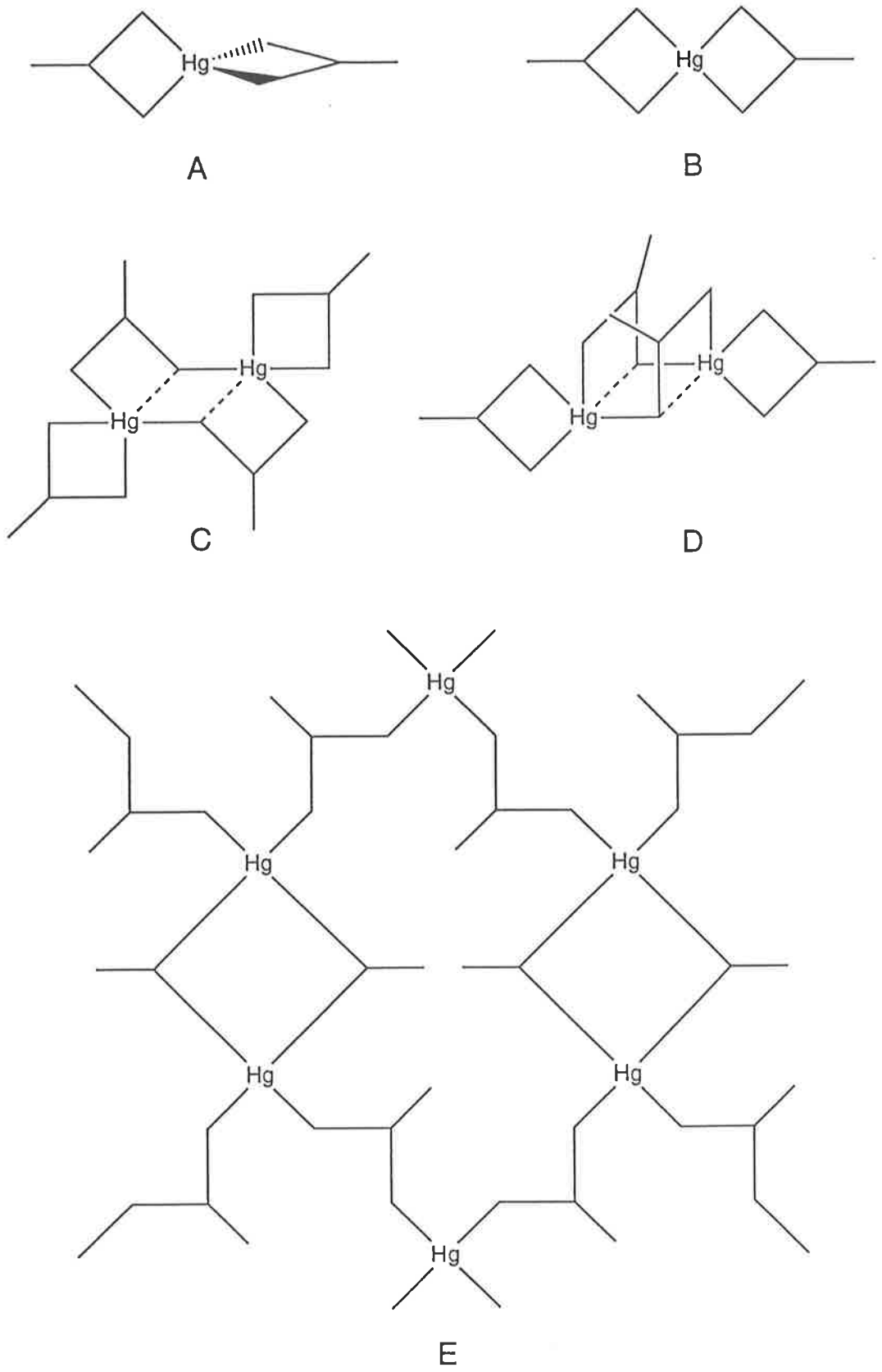

Fig. 7.17 Structural motifs for $Hg(S_2CNR_2)_2$. (A) R = iBu, iPr, R_2 = (iPr, Cy).(B) R = Et (β-form), Me. (C) R = Et (α-form), R_2 = (Et, Cy), NR_2 = 4-methyl-piperidine. (D) R = nBu. (E) R = H.

parameters about the central atoms were monitored as the nature of the R substituents were altered (chapters 3 and 4, respectively). It was found that any differences in the electronic profiles of the ligands are not manifested in systematic variations in the Ni-S or Te-S bond distances. Further, there is no change in structural motif for either of the nickel(II) or tellurium(II) series. The key result of these studies is the demonstration that little or no influence on molecular geometry or geometric parameters is found as R is varied. Given the above, the question is whether crystal packing effects play a significant role in determining molecular structure.

It is argued that given there are no obvious electronic or molecular structure-determining effects associated with the dithiocarbamate ligands, crystal packing effects *must* be taken into consideration. Evidence for this proposal is based on the experimentally determined structures for $Hg(S_2CNR_2)_2$, however, at this stage the arguments are entirely qualitative. In the crystallisation process there will be an energy associated with the molecular geometry as well as an energy of crystal packing. The ultimate structure adopted in the solid state will be the one in which a balance between the two energy considerations has been achieved. It is suggested that in the $Hg(S_2CNR_2)_2$ systems both energy contributions are significant. By contrast, for transition metal complexes it is normally assumed that the energy associated with the formation of the molecular entity (intramolecular association) far exceeds any energy contribution associated with the packing of the molecules (intermolecular association). Applying this proposal to the $Hg(S_2CNR_2)_2$ structures it is suggested that the mercury atom valencies are essentially satisfied by the formation of two primary Hg-S bonds and that the other interactions, giving rise to changes in coordination geometry, are (weak) intramolecular or intermolecular contacts.

Consider first the two structures that adopt motif B. In both examples, the two primary Hg-S bonds define a linear geometry and the additional Hg-S interactions, giving rise to a square planar geometry, are significant weaker. Indeed, the stronger Hg-S bonds in motif B are the strongest of all the Hg-S bonds found in the $Hg(S_2CNR_2)_2$

structures consistent with i) the weak secondary intramolecular associations and ii) the weak intermolecular associations; see Table 7.4. Stability in the packing of the planar molecules, with small R groups, is facilitated by weak intermolecular Hg···S interactions. As the R groups become larger, as in motif A, evidence for linear geometries is present as discussed above, however, the coordination environments are best described as distorted tetrahedral. Further, in these structures, evidence for potential intermolecular association via Hg···S interactions is also found in the respective unit cells. Reducing the size and/or branching in the R groups results in the formation of dimeric units as seen in motifs C and D. Here, the valency about the mercury atom is again satisfied by two stronger Hg-S bonds and a set of weaker Hg-S interactions. When crystallising from solution, the molecules of $Hg(S_2CNR_2)_2$ associate via intermolecular Hg-S interactions, *i.e.* maximising stability of the lattice, giving rise to dimeric units. Thus, in the absence of bulky R substituents it is possible for molecules to align in the solid state giving rise to different motifs. Finally, in the structure of $[Hg(S_2CNH_2)_2]$ where steric interactions are minimised, the valency about the mercury is attained by four *weaker* Hg-S interactions while at the same time maximising intermolecular Hg-S interactions given rise to the 2-dimensional motif, E (Chieh, Cheung, 1981). That the effects described above are subtle is seen in the fact that at least one compound, namely $[Hg(S_2CNEt_2)_2]$, crystallises in two polymorphs, adopting structural motifs B and C.

7.4 CONCLUSION

The adoption of the various structural motifs for the $Hg(S_2COR)_2$ and $Hg(S_2CNR_2)_2$ compounds has been explained, albeit qualitatively, in terms of a balance between the requirements of attaining an appropriate valency about the mercury atom on the one hand and the maximising of intermolecular forces to attain the most stable crystal structure on the other. Given that the phenomenon of structural diversity is often encountered in main group element chemistry (see *Introduction*), it is clear that a close examination of crystal structure is required in order to possibly rationalise the appearance

Table 7.4 Hg-S bond distances (Å) for $Hg(S_2CNR_2)_2$

R	Motif	Hg-S(1), S(2)	Hg-S(3), S(4)
iBu	A	2.416(2), 2.714(2)	2.437(2), 2.710(3)
iPr[a]	A	2.445(4), 2.645(4)	2.445(4), 2.645(4)
(iPr, Cy)	A	2.413(2), 2.724(2)	2.464(2), 2.669(2)
Et (β-form)[b]	B	2.398(4), 2.965(4)	2.398(4), 2.965(4)
Me	B	2.374(3), 2.988(3)	2.374(3), 2.988(3)
Et (α-form)	C	2.418(7), 3.137(6), 2.698(6)	2.520(6), 2.663(6)
(Et, Cy)	C	2.452(3), 3.047(2), 2.640(3)	2.508(2), 2.707(2)
NR_2[c]	C	2.450(2), 3.127(2), 2.625(2)	2.519(1), 2.669(1)
nBu	D	2.457(4), 3.160(4), 2.601(4)	2.543(4), 2.632(5)
H	E	2.508(4), 2.629(4)	2.499(4), 2.533(4)

a molecule has 2-fold symmetry

b molecule is centrosymmetric

c NR_2 is 4-methyl piperidine

of different motifs. Clearly, in the process of obtaining a stable crystalline lattice for these systems, perturbations in molecular geometry may sometimes arise.

Chapter 8

AB INITIO MOLECULAR ORBITAL CALCULATIONS OF SELECTED *O*-ALKYLDITHIOCARBONATES AND *N,N*-DIALKYLDITHIOCARBAMATES

To complement the crystallographic results of this thesis it was thought of interest to investigate the electronic properties the 1,1-dithiolate ligands studied, by molecular orbital theory, to determine the theoretical electronic profiles of these ligands. *Ab initio* molecular orbital theory allows the calculation of an approximate electronic wave function by numerically solving the electronic Schrödinger equation. Knowledge of the electronic wave function allows the calculation of many of the observables that are of interest to the chemist. Such observables include the potential energy of a molecule at any fixed nuclear geometry, the dipole moment of a molecule and most relevant to this study, the electron density in certain regions of a molecule.

Electronic structural programs such as the Gaussian suite (Gaussian 94, 1995) use sophisticated algorithms that allow optimisation of the molecular potential energy by varying the nuclear geometry. Such calculations are known as geometry optimisations, and at high levels of theory these can predict the equilibrium geometries of molecular systems to within experimental accuracy.

It is appropriate to mention the various levels of theory used. As mentioned, *ab initio* techniques approximately solve the electronic wave function. There exists a diverse range of theoretical implementations of varying sophistications and computational expense. The lowest level is Hartree-Fock (HF) theory. This technique for solving the wave function suffers from its lack of accounting for electron correlation. Stated simply, HF theory treats an electron's motion as if it moves in an average field of stationary electrons. In reality, the motion between electrons is "correlated", that is, they "move to avoid each other". Thus, HF energies are higher than experimental energies and HF theory often gives inaccurate reaction barriers and equilibrium geometries.

The molecular wave function is essentially a product of molecular orbitals (MOs). Each MO describes the motion, energy and distribution of two electrons (for singlet electronic states). A MO is built from atomic centered functions known as basis functions. The basis set is a well defined set of basis functions given to each type of atom; the nomenclature of basis sets is not described here. It has been shown that larger basis sets (more functions on each atom) are required to yield accurate equilibrium geometries and reaction energetics. This is intuitive, as the more basis functions that are given to the description of the electronic distribution, the more flexible it can be. A given level of theory then is the statement of and the use of an electron correlation technique and a given basis set. For example: HF/ 6-311 ++G denotes the use of Hartree-Fock theory in combination with the 6-311++G basis set.

Thus, using molecular orbital theory it is possible to investigate the electronic distribution over the 1,1-dithiolate molecules with the longer term goal of the theoretical study being to investigate the coordination of the molecules as ligands to metal centres and thereby enabling a comparison between solid state crystallographic results and gaseous state calculations. This approach has been employed with some success in Adelaide (Buntine, Hall, Tiekink, 1998; Buntine, Hall, Kosovel, Tiekink, 1998; Stewart, Tiekink, Buntine, 1997; Tiekink, Hall, Buntine, 1999) and other laboratories (Cotton, Yokochi, 1997; Wierzbicki, Salter, Hoffman, Stevens, Van Do, VanLoock, Madura, 1996). To study the electronic distribution of the selected 1,1-dithiolate ligands, the Hartree-Fock level of theory was used and the basis set employed was 6-311++G**. In addition the LANL2DZ basis set was also used to enable eventual comparisons with the complex (Buntine, Hall, Tiekink, 1998; Buntine, Hall, Kosovel, Tiekink, 1998). At the completion of the optimisations of the anions, Mulliken and natural population analysis (NPA) were used to calculate the electronic density distribution of the anions.

All geometry optimisations were performed using the GAUSSIAN 94 suite of programs (Gaussian 94, 1995) run on Silicon Graphics Indigo^{2}xZ Workstation and Silicon Graphics Power Challenge computers. All calculations were performed at the

Hartree-Fock SCF level of theory using both the 6-311++G** and LanL2DZ basis set (Gaussian 94, 1995). The LanL2DZ basis set employs the Dunning/Huzinaga double zeta descriptor (Dunning, Hay, 1976) for all first-row elements and replaces the core electrons of sulphur (up to 2*p*) chlorine (up to 2*p*),and tin (up to 4*p*) with the effective core potentials (Hay, Wadt, 1985a, 1985b; Wadt, Hay, 1985).

Thus, using the above protocols the geometry optimisations for a series of *O*-alkyldithicarbonate anions [$^{-}S_2COR$] where R = H, Me, Et, nPr, iPr, nBu, iBu, tBu, 1-MePr, nPent, 3-MeBu, 3,3-Me_2Bu, Benzyl, Hex, Cy, and *N*,*N*-dialkyldithiocarbamate anions [$^{-}S_2CNR_2$] where R_2 = H, Me, Et, nPr, iPr, iBu, nBu, Cy and R/R' = Et/Cy, iPr/Cy have been conducted. Results for the xanthate anions are presented in Tables 8.1 to 8.15 and Figs 8.1 to 8.15 and those for the dithiocarbamate anions are in Tables 8.16 to 8.25 and Figs 8.16 to 8.25.

8.1 XANTHATE ANIONS

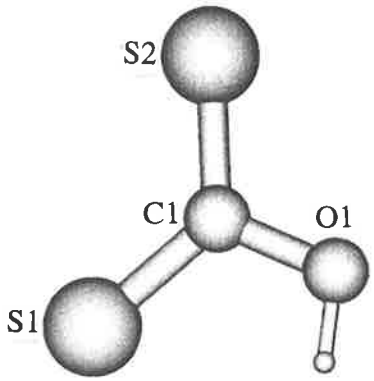

Fig. 8.1 The optimised geometry of [$^{-}S_2COH$] with the adopted numbering scheme.

Table 8.1 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2COH$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.704	1.748	C1-O1	1.337	1.382
S2-C1	1.679	1.712			
S1-C1-S2	128.6	129.8	S2-C1-O1	116.0	115.7
S1-C1-O1	115.4	114.5			
S1-C1-O1-H	0.0	0.0			

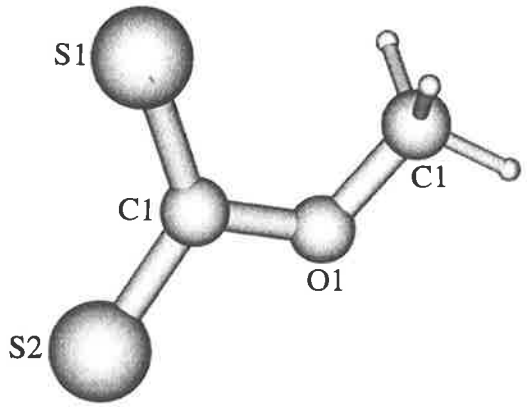

Fig. 8.2 The optimised geometry of [$^{-}S_2COMe$] with the adopted numbering scheme.

Table 8.2 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2COMe$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.698	1.745	C1-O1	1.336	1.376
S2-C1	1.691	1.725	O1-C2	1.403	1.436
S1-C1-S2	127.7	127.7	S2-C1-O1	116.0	114.0
S1-C1-O1	115.4	118.4	C1-O1-C2	106.8	110.9
S1-C1-O1-C2	0.0000	0.0			

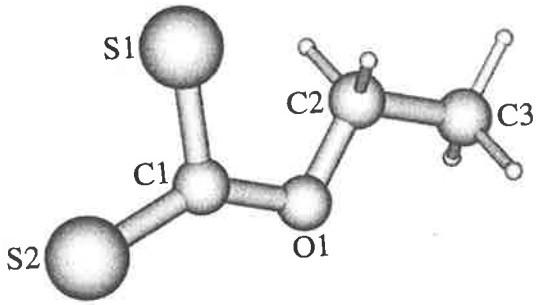

Fig. 8.3 The optimised geometry of [$^{-}S_2COEt$] with the adopted numbering scheme.

Table 8.3 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2COEt$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.698	1.745	O1-C2	1.407	1.442
S2-C1	1.692	1.726	C2-C3	1.517	1.522
C1-O1	1.335	1.375			
S1-C1-S2	126.5	127.6	C1-O1-C2	122.9	123.6
S1-C1-O1	119.8	118.4	O1-C2-C3	106.8	106.3
S2-C1-O1	113.7	114.0			
S1-C1-O1-C2	0.0	0.0	C1-O1-C2-C3	180.0	180.0

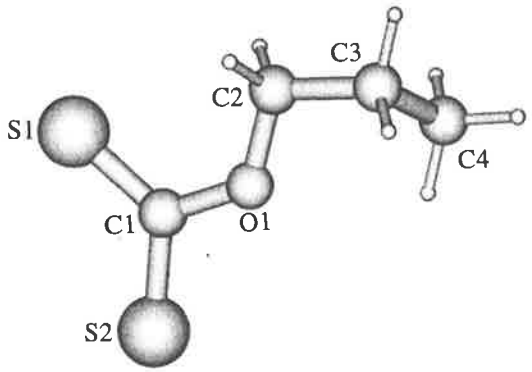

Fig. 8.4 The optimised geometry of [$^{-}S_2$COnPr] with the adopted numbering scheme.

Table 8.4 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2$COnPr].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.697	1.744	O1-C2	1.407	1.4
S2-C1	1.692	1.726	C2-C3	1.520	1.525
C1-O1	1.335	1.375	C3-C4	1.528	1.536
S1-C1-S2	126.6	127.7	C1-O1-C2	123.1	123.8
S1-C1-O1	119.8	118.4	O1-C2-C3	107.2	106.6
S2-C1-O1	113.6	113.9	C2-C3-C4	113.7	113.0
S1-C1-O1-C2	-0.8	-0.9	O1-C2-C3-C4	62.9	62.9
C1-O1-C2-C3	-179.1	-178.9			

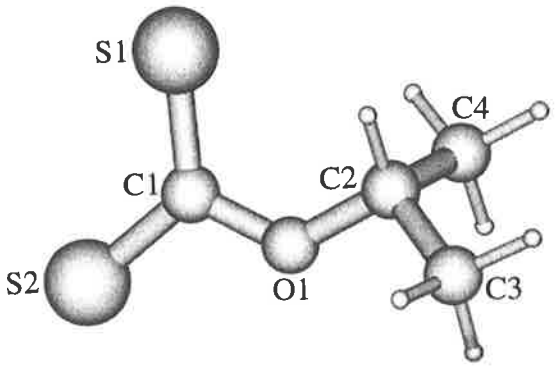

Fig. 8.5 The optimised geometry of [$^{-}S_2COiPr$] with the adopted numbering scheme.

Table 8.5 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2COiPr$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.702	1.749	O1-C2	1.417	1.450
S2-C1	1.691	1.725	C2-C3	1.522	1.529
C1-O1	1.335	1.375	C3-C4	1.521	1.526
S1-C1-S2	125.9	127.1	O1-C2-C3	109.7	109.3
S1-C1-O1	120.6	119.1	O1-C2-C4	105.7	105.2
S2-C1-O1	113.5	113.8	C3-C2-C4	112.5	112.9
C1-O1-C2	124.7	125.4			
S1-C1-O1-C2	-2.2	-1.5	C1-O1-C2-C4	-90.7	-88.4
C1-O1-C2-C3	-147.7	-150.1			

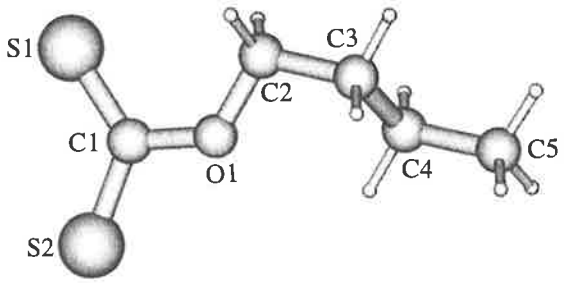

Fig. 8.6 The optimised geometry of [$^{-}S_2$COnBu] with the adopted numbering scheme.

Table 8.6 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2$COnBu].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.697	1.744	C2-C3	1.520	1.525
S2-C1	1.692	1.726	C3-C4	1.528	1.537
C1-O1	1.335	1.375	C4-C5	1.528	1.538
O1-C2	1.408	1.441			
S1-C1-S2	126.6	127.7	O1-C2-C3	107.2	106.6
S1-C1-O1	119.8	118.4	C2-C3-C4	113.9	113.4
S2-C1-O1	113.6	113.9	C3-C4-C5	112.9	112.5
C1-O1-C2	123.1	123.7			
S1-C1-O1-C2	-0.8	-0.8	O1-C2-C3-C4	63.4	63.1
C1-O1-C2-C3	-179.3	-179.2	C2-C3-C4-C5	180.0	-179.6

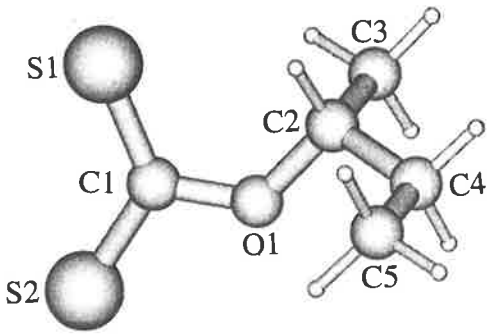

Fig. 8.7 The optimised geometry of [$^{-}S_2CO1$-MePr] with the adopted numbering scheme.

Table 8.7 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2CO1$-MePr].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.701	1.748	C2-C3	1.522	1.529
S2-C1	1.692	1.726	C3-C4	1.521	1.530
C1-O1	1.335	1.375	C4-C5	1.527	1.536
O1-C2	1.417	1.450			
S1-C1-S2	126.0	127.2	O1-C2-C3	109.6	109.2
S1-C1-O1	120.6	119.1	O1-C2-C3	106.2	105.6
S2-C1-O1	113.4	113.7	C2-C3-C4	113.5	113.0
C1-O1-C2	124.9	125.6	C3-C4-C5	112.4	112.7
S1-C1-O1-C2	-0.3	-0.6	C1-O1-C2-C4	146.9	149.3
C1-O1-C2-C3	-91.4	-89.2	O1-C3-C4-C5	-62.8	-62.9

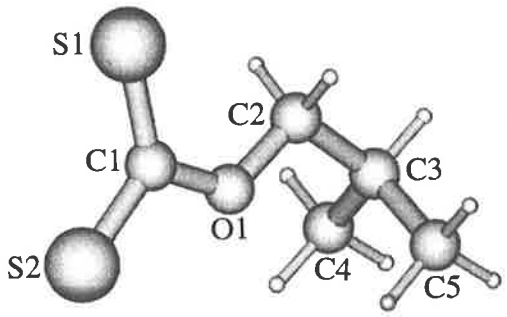

Fig. 8.8 The optimised geometry of [$^{-}S_2COiBu$] with the adopted numbering scheme.

Table 8.8 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2COiBu$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.697	1.744	C2-C3	1.526	1.530
S2-C1	1.692	1.726	C3-C4	1.531	1.539
C1-O1	1.335	1.375	C3-C5	1.531	1.539
O1-C2	1.407	1.440			
S1-C1-S2	126.6	127.8	O1-C2-C3	107.7	107.2
S1-C1-O1	119.8	118.4	C2-C3-C4	111.8	111.4
S2-C1-O1	113.5	113.8	C2-C3-C5	111.8	111.4
C1-O1-C2	123.2	124.0	C4-C3-C5	111.4	111.1
S1-C1-O1-C2	-0.1	0.0	O1-C2-C3-C4	-62.8	-62.4
C1-O1-C2-C3	-179.8	180.0	O1-C2-C3-C5	62.8	62.4

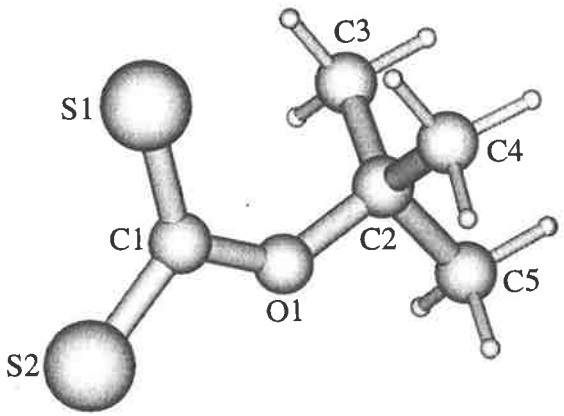

Fig. 8.9 The optimised geometry of [$^{-}S_2COtBu$] with the adopted numbering scheme.

Table 8.9 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2COtBu$].

	6-311++ G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.700	1.744	C2-C3	1.529	1.530
S2-C1	1.695	1.726	C2-C4	1.529	1.539
C1-O1	1.335	1.375	C2-C5	1.531	1.539
O1-C2	1.432	1.440			
S1-C1-S2	124.9	125.9	O1-C2-C4	112.0	111.3
S1-C1-O1	122.5	121.2	O1-C2-C5	101.5	101.3
S2-C1-O1	112.6	113.0	C3-C2-C4	112.3	112.4
C1-O1-C2	131.2	131.8	C3-C2-C5	109.2	110.0
O1-C2-C3	112.0	111.3	C4-C2-C5	109.2	110.0
S1-C1-O1-C2	0.000	0.0	C1-O1-C2-C4	63.6	63.1
C1-O1-C2-C3	-63.567	-63.2	C1-O1-C2-C5	180.0	180.0

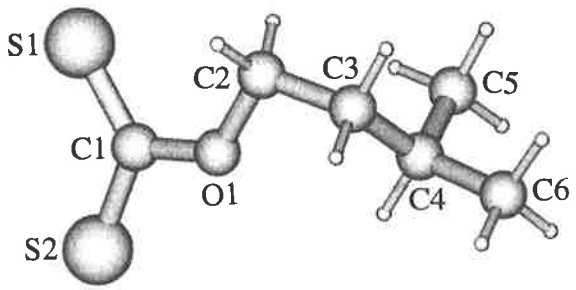

Fig. 8.10 The optimised geometry of [$^{-}S_2CO$-3-MeBu] with the adopted numbering scheme.

Table 8.10 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2CO$-3-MeBu].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.697	1.743	C2-C3	1.521	1.526
S2-C1	1.692	1.726	C3-C4	1.536	1.547
C1-O1	1.335	1.376	C4-C5	1.531	1.541
O1-C2	1.408	1.440	C4-C6	1.532	1.543
S1-C1-S2	126.6	127.8	C2-C3-C4	115.8	115.8
S1-C1-O1	119.8	118.5	C3-C4-C5	112.6	112.6
S2-C1-O1	113.7	113.7	C3-C4-C6	110.2	110.2
C1-O1-C2	123.1	123.7	C5-C4-C6	110.4	110.2
O1-C2-C3	107.2	108.2			
S1-C1-O1-C2	-0.6	-1.4	C2-C3-C4-C5	61.6	-80.7
C1-O1-C2-C3	-179.3	-179.7	C2-C3-C4-C6	-174.6	155.8
O1-C2-C3-C4	58.8	65.8			

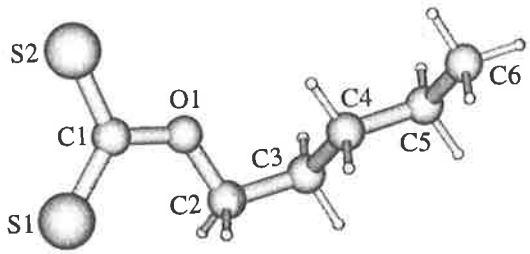

Fig. 8.11 The optimised geometry of [$^{-}S_2$COnPent] with the adopted numbering scheme.

Table 8.11 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2$COnPent].

Bonds	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.697	1.744	C2-C3	1.520	1.525
S2-C1	1.692	1.726	C3-C4	1.528	1.536
C1-O1	1.335	1.375	C4-C5	1.528	1.538
O1-C2	1.408	1.441	C5-C6	1.450	1.538
S1-C1-S2	126.5	127.7	O1-C2-C3	107.2	106.6
S1-C1-O1	119.8	118.4	C2-C3-C4	113.9	113.4
S2-C1-O1	113.6	113.9	C3-C4-C5	112.9	112.8
C1-O1-C2	123.1	123.8	C4-C5-C6	119.5	112.8
S1-C1-O1-C2	-0.8	-0.8	C2-C3-C4-C5	-179.6	-179.0
C1-O1-C2-C3	-179.4	-179.2	C3-C4-C5-C6	-179.8	-179.9
O1-C2-C3-C4	63.5	63.1			

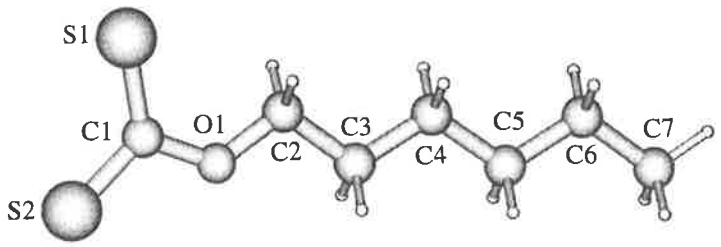

Fig. 8.12 The optimised geometry of [$^{-}S_2$COHex] with the adopted numbering scheme.

Table 8.12 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2$COHex].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.698	1.745	C3-C4	1.529	1.538
S2-C1	1.692	1.725	C4-C5	1.529	1.538
C1-O1	1.336	1.376	C5-C6	1.529	1.538
O1-C2	1.406	1.440	C6-C7	1.528	1.538
C2-C3	1.519	1.524			
S1-C1-S2	126.5	127.6	C2-C3-C4	112.3	112.0
S1-C1-O1	119.8	118.4	C3-C4-C5	113.2	112.9
S2-C1-O1	113.7	114.0	C4-C5-C6	113.6	113.3
C1-O1-C2	122.9	123.5	C5-C6-C7	113.3	112.9
O1-C2-C3	106.8	106.5			
S1-C1-O1-C2	0.0	0.0	C2-C3-C4-C5	180.0	180.0
C1-O1-C2-C3	180.0	180.0	C3-C4-C5-C6	180.0	180.0
O1-C2-C3-C4	180.0	180.0	C4-C5-C6-C7	180.0	180.0

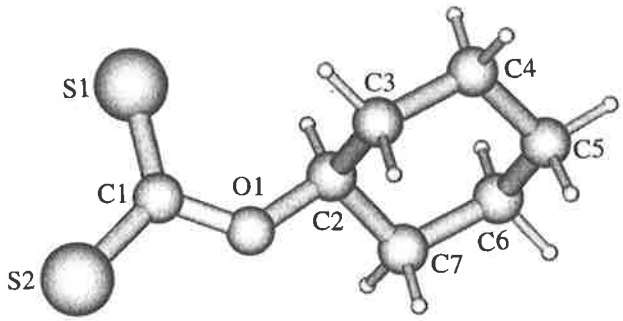

Fig. 8.13 The optimised geometry of [$^{-}S_2COCy$] with the adopted numbering scheme.

Table 8.13 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2COCy$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.702	1.749	C3-C4	1.531	1.540
S2-C1	1.691	1.724	C4-C5	1.531	1.541
C1-O1	1.336	1.376	C5-C6	1.531	1.541
O1-C2	1.413	1.444	C6-C7	1.531	1.540
C2-C3	1.525	1.532	C7-C2	1.524	1.529
S1-C1-S2	125.9	127.1	C2-C3-C4	111.0	110.7
S1-C1-O1	120.6	119.1	C3-C4-C5	111.6	111.4
S2-C1-O1	113.5	113.8	C4-C5-C6	111.2	111.1
C1-O1-C2	124.6	125.3	C5-C6-C7	111.6	111.4
O1-C2-C3	110.3	110.1	C6-C7-C2	111.1	110.9
O1-C2-C7	106.6	106.1	C7-C2-C3	111.3	111.5
S1-C1-O1-C2	1.8	0.7	O1-C2-C3-C4	174.375	174.1
C1-O1-C2-C3	91.5	89.5	O1-C2-C7-C6	-176.490	-176.4
C1-O1-C2-C7	-147.5	-149.8	C2-C3-C4-C5	-55.392	-55.4

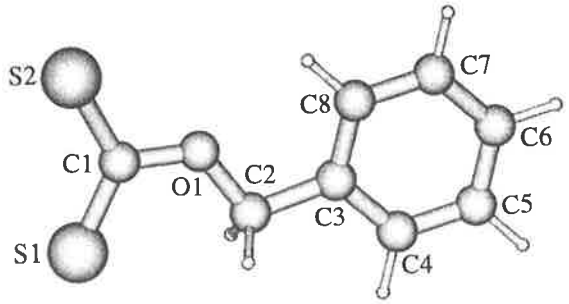

Fig. 8.14 The optimised geometry of [$^{-}S_2COCH_2Ph$] with the adopted numbering scheme.

Table 8.14 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2COCH_2Ph$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.695	1.741	C4-C5	1.388	1.397
S2-C1	1.689	1.722	C5-C6	1.384	1.395
C1-O1	1.341	1.380	C6-C7	1.388	1.397
O1-C2	1.398	1.430	C7-C8	1.383	1.394
C2-C3	1.513	1.514	C8-C3	1.392	1.400
C3-C4	1.388	1.397			
S1-C1-S2	127.0	128.2	C4-C5-C6	120.7	120.7
S1-C1-O1	119.7	118.2	C5-C6-C7	119.3	119.3
S2-C1-O1	113.3	113.6	C6-C7-C8	120.0	120.0
C1-O1-C2	122.7	123.5	C7-C8-C3	121.0	120.9
O1-C2-C3	108.7	108.2	C8-C3-C4	118.7	118.9
C2-C3-C4	122.7	122.1	C8-C3-C2	118.6	119.0
C3-C4-C5	120.3	120.2			
S1-C1-O1-C2	0.1	0.0	O1-C2-C3-C4	-0.7	180.0
C1-O1-C2-C3	179.9	180.0	O1-C2-C3-C8	179.4	180.0

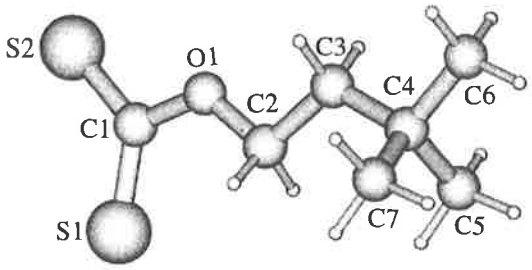

Fig. 8.15 The optimised geometry of [$^{-}S_2CO$-3,3-Me_2Bu] with the adopted numbering scheme.

Table 8.15 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2CO$-3,3-Me_2Bu].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.698	1.745	C3-C4	1.544	1.552
S2-C1	1.692	1.724	C4-C5	1.538	1.546
C1-O1	1.335	1.376	C4-C6	1.537	1.546
O1-C2	1.408	1.441	C4-C7	1.537	1.546
C2-C3	1.522	1.527			
S1-C1-S2	126.5	127.6	C3-C4-C5	111.2	111.2
S1-C1-O1	119.8	118.4	C3-C4-C6	107.8	107.8
S2-C1-O1	113.7	114.0	C3-C4-C7	111.2	111.2
C1-O1-C2	122.7	123.4	C5-C4-C6	108.6	108.7
O1-C2-C3	105.8	105.4	C6-C4-C7	108.6	108.7
C2-C3-C4	116.4	116.0	C5-C4-C7	109.3	109.3
S1-C1-O1-C2	-0.0	-0.0	C2-C3-C4-C5	-61.0	-61.0
C1-O1-C2-C3	180.0	180.0	C2-C3-C4-C6	180.0	180.0
O1-C2-C3-C4	180.0	180.0	C2-C3-C4-C7	61.0	61.0

8.1.1 *Summary of the optimised geometries of the xanthate anions.*

To obtain the optimised geometry of a particular [^-S_2COR] several alternative starting conformations were used. The resulting optimised geometry with the lowest energy is reported. A general comparison of the parameters of the optimised geometries between the two different basis sets (LANL2DZ and 6311++G**) reveals that the bond lengths for the LANL2DZ basis set are longer than the bond lengths in the 6311++G** basis set. The angles involved in each basis set are approximately the same. An investigation of the influence of the R group on the geometry of [^-S_2COR] follows.

The optimised geometries of the xanthate anions have a consistent characteristic, the ^-S_2CO moiety. There is asymmetry in the C-S bond distances with the S(1)-C(1) bond (the bond closest to the coordinated R group) slightly longer than the S(2)-C(1) bonds throughout the range of xanthate anions, this is ascribed to S...H interactions. The S(1)-C(1) bond lengths fall in the narrow range of 1.697 Å to 1.704 Å (1.743 Å - 1.749 Å for the LANL2DZ basis set) and the S(2)-C(1) bonds lie in the narrow range of 1.689 Å to 1.695 Å (1.722 Å to 1.726 Å) excluding [^-S_2COH] which displays a slightly shorter bond length (1.679 Å (1.712 Å)). The bite angle of the ^-S_2CO moiety lies in the narrow range of 124.9° to 128.6° (125.9° - 128.2°). The C(1)-O(1) and O(1)-C(2) bond lengths are very consistent throughout the series remaining in the narrow range of 1.335 Å to 1.341 Å (1.375 Å to 1.382 Å) and 1.398 Å to 1.432 Å (1.430 Å to 1.450 Å) respectively. The relatively narrow ranges observed for the S-C and C-O bond distances, and associated angles, support the hypothesis that the influence exerted by the O-bound H and organic substituents is minimal. The other geometric result of interest is the torsion angle, which for every ^-S_2COC moiety is 180°, indicating that the ^-S_2COC moiety is planar.

The organic substituents coordinated to the ^-S_2CO moiety display characteristic C-C bond lengths and angles. There are some interesting features of the anions as the R group increases in size. In those anions containing methyl and ethyl groups, are co-planar with the ^-S_2CO moiety. When the n-propyl group is substituted, rather than being

co-planar, the torsion angle involving the third carbon atom in the chain is 62.9°. This third carbon atom in the chain remains at this torsion angle as the carbon chain increases in length to butyl and pentyl with the additional carbon atoms *i.e.* the fourth and fifth carbon atoms displaying a torsion angle of 180°. When the carbon chain increases to hexyl all the carbon atoms in the chain are co-planar with the $^{-}S_2CO$ moiety.

8.2 DITHIOCARBAMATE ANIONS

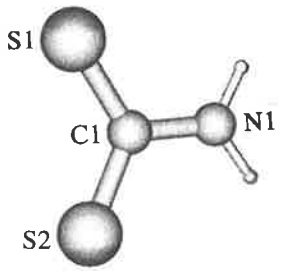

Fig. 8.16 The optimised geometry of $[^{-}S_2CNH_2]$ with the adopted numbering scheme.

Table 8.16 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of $[^{-}S_2CNH_2]$.

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.711	1.756	C1-N1	1.348	1.367
S2-C1	1.711	1.756			
S1-C1-S2	127.1	128.0	S2-C1-N1	116.4	116.0
S1-C1-N1	116.4	116.0			

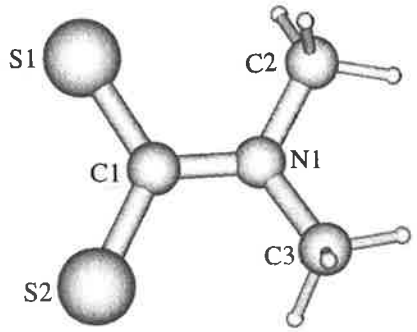

Fig. 8.17 The optimised geometry of [$^{-}S_2CNMe_2$] with the adopted numbering scheme.

Table 8.17 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2CNMe_2$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.718	1.767	N1-C2	1.452	1.467
S2-C1	1.718	1.767	N1-C3	1.452	1.467
C1-N1	1.360	1.370			
S1-C1-S2	122.106	123.344	C1-N1-C2	121.248	121.063
S1-C1-N1	118.793	118.329	C1-N1-C3	121.589	121.064
S2-C1-N1	119.100	118.327			
S1-C1-N1-C2	8.0	0.0	S1-C1-N1-C3	172.4	180.0

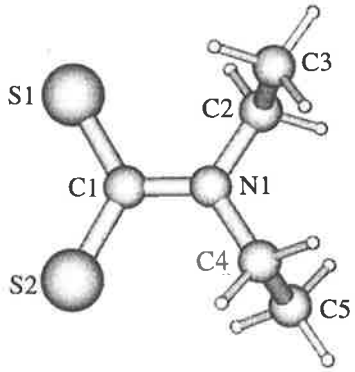

Fig. 8.18 The optimised geometry of [$^{-}S_2CNEt_2$] with the adopted numbering scheme.

Table 8.18 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2CNEt_2$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.722	1.771	C2-C3	1.527	1.537
S2-C1	1.722	1.771	N1-C4	1.458	1.471
C1-N1	1.358	1.371	C4-C5	1.527	1.537
N1-C2	1.458	1.471			
S1-C1-S2	121.4	122.1	C1-N1-C4	122.9	122.7
S1-C1-N1	119.3	119.0	N1-C2-C3	113.3	112.8
S2-C1-N1	119.3	119.0	N1-C4-C5	113.3	112.8
C1-N1-C2	122.9	122.7			
S1-C1-N1-C2	-1.8	-1.9	C1-N1-C2-C3	91.4	90.3

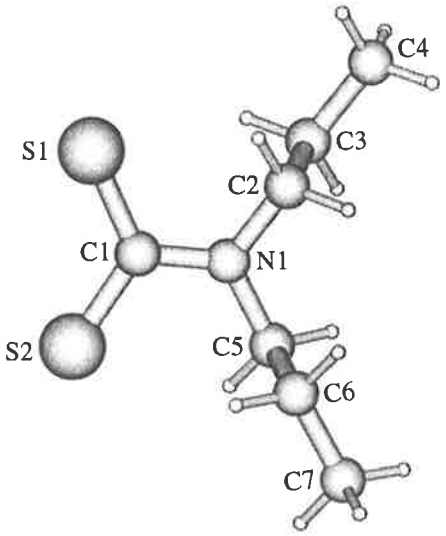

Fig. 8.19 The optimised geometry of [$^{-}S_2CNnPr_2$] with the adopted numbering scheme.

Table 8.19 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2CNnPr_2$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.722	1.771	C3-C4	1.528	1.538
S2-C1	1.722	1.771	N1-C5	1.457	1.469
C1-N1	1.358	1.371	C5-C6	1.529	1.537
N1-C2	1.457	1.469	C6-C7	1.528	1.538
C2-C3	1.529	1.537			
S1-C1-S2	121.4	122.1	C2-C3-C4	111.9	111.7
S1-C1-N1	119.1	119.0	C1-N1-C5	122.8	122.6
S2-C1-N1	119.3	119.0	N1-C5-C6	113.5	113.1
C1-N1-C2	122.8	122.6	C5-C6-C7	111.9	111.7
N1-C2-C3	113.5	113.1			
S1-C1-N1-C2	1.7	1.8	N1-C2-C3-C4	-179.5	-179.7
C1-N1-C2-C3	-90.7	-89.7			

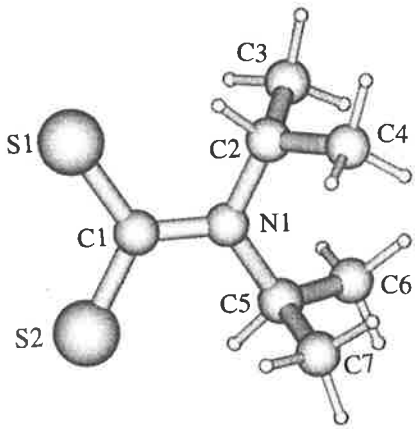

Fig. 8.20 The optimised geometry of [$^{-}S_2CNiPr_2$] with the adopted numbering scheme.

Table 8.20 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2CNiPr_2$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.725	1.775	C2-C4	1.532	1.542
S2-C1	1.725	1.775	N1-C5	1.475	1.488
C1-N1	1.371	1.386	C5-C6	1.534	1.540
N1-C2	1.475	1.488	C5-C7	1.532	1.542
C2-C3	1.534	1.540			
S1-C1-S2	118.9	119.7	C3-C2-C4	111.9	112.0
S1-C1-N1	120.5	120.2	C1-N1-C5	119.3	119.3
S2-C1-N1	120.5	120.2	N1-C5-C6	113.7	113.3
C1-N1-C2	119.3	119.3	N1-C5-C7	114.7	114.5
N1-C2-C3	113.7	113.3	C6-C5-C7	111.9	112.0
N1-C2-C4	114.7	114.5			
S1-C1-N1-C2	5.6	7.2	C1-N1-C2-C4	-96.1	-95.7
C1-N1-C2-C3	133.4	134.2			

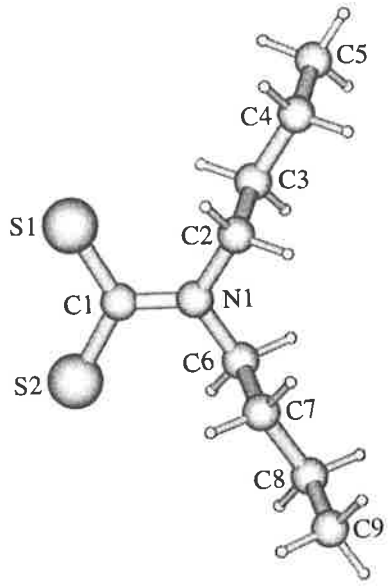

Fig. 8.21 The optimised geometry of [$^{-}S_2CNnBu_2$] with the adopted numbering scheme.

Table 8.21 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2CNnBu_2$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.722	1.771	C4-C5	1.528	1.538
S2-C1	1.722	1.771	N1-C6	1.457	1.470
C1-N1	1.358	1.371	C6-C7	1.529	1.537
N1-C2	1.457	1.470	C7-C8	1.528	1.538
C2-C3	1.529	1.537	C8-C9	1.528	1.538
C3-C4	1.528	1.538			
S1-C1-S2	121.4	122.1	C3-C4-C5	113.1	112.7
S1-C1-N1	119.3	119.0	C1-N1-C6	122.8	122.6
S2-C1-N1	119.3	119.0	N1-C6-C7	113.4	113.0
C1-N1-C2	122.8	122.6	C6-C7-C8	112.2	112.1
N1-C2-C3	113.4	113.0	C7-C8-C9	113.1	112.7
C2-C3-C4	112.2	112.1			
S1-C1-N1-C2	1.6	1.8	N1-C2-C3-C4	-180.0	179.4
C1-N1-C2-C3	-90.6	-89.6	C2-C3-C4-C5	-180.0	180.0

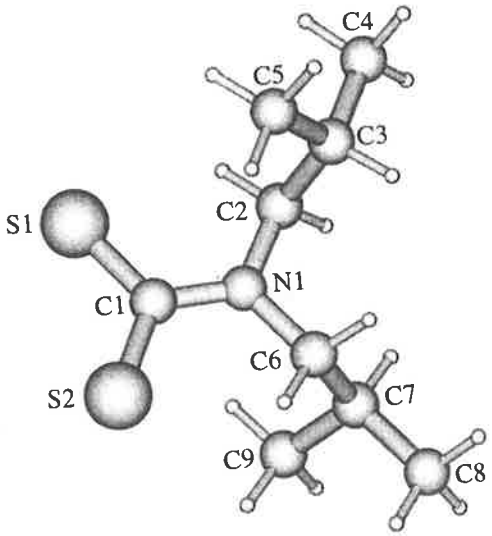

Fig. 8.22 The optimised geometry of [$^{-}S_2CNiBu_2$] with the adopted numbering scheme.

Table 8.22 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2CNiBu_2$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.722	1.772	C3-C5	1.530	1.542
S2-C1	1.722	1.772	N1-C6	1.458	1.471
C1-N1	1.366	1.378	C6-C7	1.540	1.547
N1-C2	1.458	1.471	C7-C8	1.532	1.542
C2-C3	1.540	1.547	C7-C9	1.530	1.538
C3-C4	1.532	1.538			
S1-C1-S2	120.1	120.8	C2-C3-C5	112.9	112.8
S1-C1-N1	120.0	119.6	C4-C3-C5	111.0	111.0
S2-C1-N1	120.0	119.6	N1-C6-C7	115.2	114.7
C1-N1-C2	122.7	122.8	C6-C7-C8	108.8	108.8
N1-C2-C3	115.2	114.7	C6-C7-C9	112.9	112.8
C2-C3-C4	108.8	108.8	C8-C7-C9	111.0	111.0
S1-C1-N1-C2	4.8	4.2	N1-C1-C2-C3	179.6	178.7
C1-N1-C2-C3	107.3	105.3	N1-C1-C2-C4	-56.7	-57.8

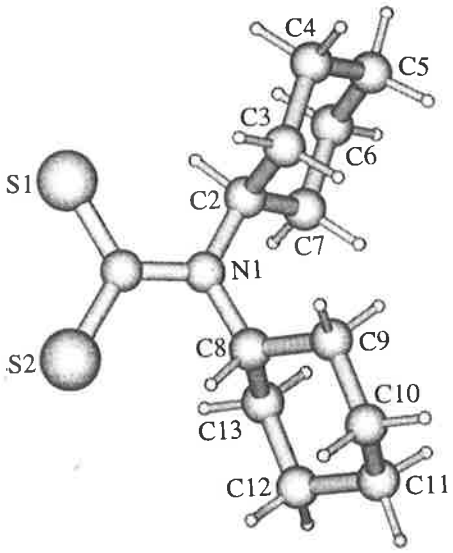

Fig. 8.23 The optimised geometry of [$^{-}S_2CNCy_2$] with the adopted numbering scheme.

Table 8.23 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2CNCy_2$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.725	1.775	C7-C2	1.535	1.543
S2-C1	1.725	1.775	N1-C8	1.474	1.485
C1-N1	1.372	1.386	C8-C9	1.535	1.543
N1-C2	1.474	1.485	C9-C10	1.534	1.543
C2-C3	1.537	1.543	C10-C11	1.530	1.540
C3-C4	1.532	1.540	C11-C12	1.530	1.540
C4-C5	1.530	1.540	C12-C13	1.532	1.540
C5-C6	1.530	1.540	C13-C8	1.537	1.544
C6-C7	1.534	1.543			
S1-C1-S2	118.8	119.6	C7-C2-C3	111.3	111.3
S1-C1-N1	120.6	120.2	C1-N1-C8	119.1	119.1
S2-C1-N1	120.6	120.2	C8-C9-C10	109.9	109.6
C1-N1-C2	119.1	119.1	C9-C10-C11	112.1	112.0
C2-C3-C4	110.4	110.2	C10-C11-C12	111.1	111.1
C3-C4-C5	111.4	111.2	C11-C12-C13	111.4	111.2
C4-C5-C6	111.1	111.1	C12-C13-C8	110.4	110.2
C5-C6-C7	112.1	112.0	C13-C8-C9	111.3	111.3
C6-C7-C2	109.9	109.6			
S1-C1-N1-C2	-5.5	-7.4	C2-C3-C4-C5	-56.1	-56.3
C1-N1-C2-C3	95.9	-78.9	C1-N1-C2-C7	-133.4	-134.3
N1-C2-C3-C4	-169.9	-169.7	N1-C2-C7-C6	171.3	171.3

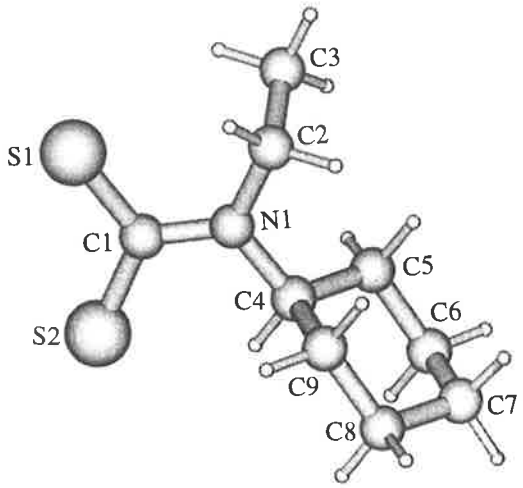

Fig. 8.24 The optimised geometry of [$^{-}S_2CN(Et)Cy$] with the adopted numbering scheme.

Table 8.24 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2CN(Et)Cy$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.720	1.770	C4-C5	1.534	1.543
S2-C1	1.725	1.775	C5-C6	1.533	1.540
C1-N1	1.364	1.377	C6-C7	1.530	1.540
N1-C2	1.463	1.475	C7-C8	1.530	1.540
C2-C3	1.528	1.537	C8-C9	1.532	1.541
N1-C4	1.466	1.478	C9-C4	1.535	1.541
S1-C1-S2	120.3	121.0	C4-C5-C6	110.5	111.0
S1-C1-N1	119.4	119.1	C5-C6-C7	111.7	111.5
S2-C1-N1	120.3	119.9	C6-C7-C8	111.1	111.1
C1-N1-C2	121.3	121.2	C7-C8-C9	111.6	111.6
N1-C2-C3	114.6	114.2	C8-C9-C4	111.1	110.2
C1-N1-C4	121.5	121.5	C9-C4-C5	111.2	111.1
S1-C1-N1-C2	4.1	3.8	C1-N1-C2-C3	-86.5	-85.3
S1-C1-N1-C4	174.5	176.3	C4-C5-C6-C7	-56.0	-55.1
C1-N1-C4-C5	135.6	135.8	C1-N1-C4-C9	-97.3	-97.7
N1-C4-C5-C6	-176.3	-176.5	N1-C4-C9-C8	175.9	175.9

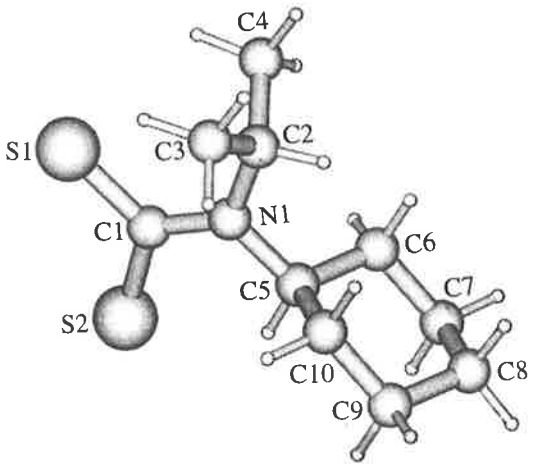

Fig. 8.25 The optimised geometry of [$^{-}S_2CN(iPr)Cy$] with the adopted numbering scheme.

Table 8.25 Selected bond lengths (Å), angles (deg) and dihedral angles (deg) of [$^{-}S_2CN(iPr)Cy$].

	6-311 ++G**	LANL2DZ		6-311 ++G**	LANL2DZ
S1-C1	1.718	1.770	C5-C6	1.535	1.542
S2-C1	1.729	1.780	C6-C7	1.532	1.541
C1-N1	1.365	1.378	C7-C8	1.530	1.540
N1-C2	1.479	1.492	C8-C9	1.530	1.540
C2-C3	1.533	1.541	C9-C10	1.532	1.541
C2-C4	1.533	1.541	C10-C5	1.535	1.542
N1-C5	1.470	1.483			
S1-C1-S2	118.9	119.2	C1-N1-C5	120.6	120.7
S1-C1-N1	120.8	120.9	C5-C6-C7	110.6	110.5
S2-C1-N1	120.4	120.0	C6-C7-C8	111.7	111.5
C1-N1-C2	125.2	125.1	C7-C8-C9	111.1	111.1
N1-C2-C3	114.8	114.3	C8-C9-C10	111.7	111.5
N1-C2-C4	114.8	114.3	C9-C10-C5	110.6	110.5
C4-C2-C3	112.5	112.2	C10-C5-C6	111.6	111.6
S1-C1-N1-C2	-0.1	-0.1	N1-C5-C6-C7	-175.3	-175.4
C1-N1-C2-C3	-66.3	-65.6	C5-C6-C7-C8	-55.3	-55.6
C1-N1-C2-C4	66.4	65.7	C1-N1-C5-C10	-116.0	-116.2
S1-C1-N1-C4	179.9	179.9	N1-C5-C10-C9	175.3	175.4
C1-N1-C5-C6	115.8	116.1			

8.2.1 *Summary of the optimised geometries of the dithiocarbamate anions.*

To obtain the optimised geometry of a particular [$^{-}S_2CNR_2$] species the method used for the xanthate anions was employed that is, several alternative starting conformations were used and the resulting optimised geometry with the lowest energy reported. Similarly, a general comparison of the parameters of the optimised geometries between the two different basis sets (LANL2DZ and 6311++G**) reveals that the bond lengths for the LANL2DZ basis set are longer than the bond lengths in the 6311++G** basis set. The angles involved in each basis set are approximately the same. An investigation of the influence of the R group on the geometry of [$^{-}S_2CNR_2$] follows.

The dithiocarbamates display a similar feature to the xanthates in that the geometry of the $^{-}S_2CN$ moiety remains consistent irrespective of the N-bound R group. The S(1)-C(1) and S(2)-C(1) bond lengths are equal in dithiocarbamate anions where the R and R' groups are identical. The S-C bond lengths lie in the narrow range of 1.718 Å to 1.729 Å (1.767 Å to 1.780 Å for the LANL2DZ basis set) excluding the dithiocarbamate anion itself which displays a marginally shorter length of 1.711 Å (1.756 Å). Unlike the xanthate anions, the bite angle of the dithiocarbamate anions vary slightly with a change in the N-bound substituent. The largest R group of cyclohexyl results in the smallest S(1)-C(1)-S(2) angle of 118.8° (119.6°), with the smallest R group, the hydrogen atom displaying the largest S(1)-C(1)-S(2) angle of 127.1° (128.0°). This reflects the different steric profiles of the R groups. The size of the R group does exert a small but discernable influence on the S(1)-C(1)-S(2) angle. The C-N bond length remains relatively constant as the R and R' groups are varied. These distances are observed in a narrow range of 1.348 Å to 1.372 Å (1.367 Å to 1.386 Å). This same feature is seen for the N(1)-C(2)/C(x) (x varies depending on the R group) with a narrow range observed 1.452 Å to 1.479 Å (1.467 Å to 1.492 Å). All of the dithiocarbamate anions adopt a torsion angle for the $^{-}S_2CNC$ moiety of 180° demonstrating the planar nature of the moiety. Similar to that observed for the xanthate anions, the narrow ranges of the geometric parameters of the $^{-}S_2CN$ moiety of the dithiocarbamate anions supports the suggestion that the nature of

the N-bound organic substituent has little effect on the geometry of the $^{-}S_2CN$ moiety of the anion.

The organic substituents coordinated to the $^{-}S_2CN$ moiety display characteristic C-C bond lengths and angles. There are some interesting features of the anions when the R and R' groups are identical. As the R and R' group increases in size from methyl to ethyl the second carbon atom is found at a torsion angle of 90° (*i.e.* C(1)-N(1)-C(2)-C(3) and C(1)-N(1)-C(4)-C(5)). This second carbon in the chain remains at a torsion angle of 90° as the length of the carbon change is increased to n-propyl and n-butyl with the subsequent carbon atoms in the chain (*i.e.* the third and fourth carbon atoms) linear with a torsion angle of 180°.

8.3 ELECTRON DENSITY DISTRIBUTION OF [$^{-}S_2COR$] AND [$^{-}S_2CNR_2$].

The electronic density distribution of [$^{-}S_2COR$] and [$^{-}S_2CNR_2$] was calculated using the Mulliken population analysis and the Natural Population Analysis (NPA) for the geometry optimised structures calculated using both the 6-311++G** and LANL2DZ basis sets. The results are summarised in Tables 8.27 to 8.30 for the xanthate anions and Tables 8.31 to 8.34 for the dithiocarbamate anions; these are presented at the end of the chapter. It should be noted that Mulliken population analysis is an arbitrary method for the assignment of atomic charges. Mulliken population analysis assigns atomic charges by dividing molecular orbital overlap evenly between each pair of atoms involved in a chemical bond. Usually, differences in Mulliken population analysis are a more useful indicator of changes in electronic distribution. To check whether there are artifacts in the Mulliken population analysis, a natural population analysis (Glendening, Reed, Carpenter, Weinhold, NBO, Version 3.1) was also performed. Thus, the values reported in Tables 8.27 to 8.34 do not represent absolute charges but give an indication of excess or deficiency of electron density on particular atoms.

8.3.1 *Xanthate anions.*

The electron density distribution of the *O*-alkyldithiocarbonate anions is such that electron distribution of the $^{-}S_2CO$ moiety remains consistent regardless of the R group coordinated to the oxygen atom. The electron distribution of the $^{-}S_2CO$ moiety is summarised in Table 8.35. All calculations reveal, as expected, a significant electron density on the S(1) and S(2) atoms and consistently, a small excess of electron density on the S(1) atom compared to the S(2) atom for each $^{-}S_2COR$ anion. This result is consistent with the small elongation of the S(1)-C bonds over the S(2)-C bonds mentioned in 8.1.1.

Table 8.35 General summary of the electron distribution over the [$^{-}S_2CO$] moiety.

Mulliken population analysis		
Atom	6-311++G**	LANL2DZ
S(1)	-0.70047 to -0.83731	-0.28856 to -0.32189
S(2)	-0.66550 to -0.713861	-0.25922 to -0.28494
C(1)	0.24091 to 0.63339	-0.24055 to -0.26299
O(1)	-0.06963 to -0.327543	-0.42715 to -0.54345

Natural population analysis		
Atom	6-311++G**	LANL2DZ
S(1)	-0.46101 to -0.49216	-0.46641 to -0.49185
S(2)	-0.43889 to -0.44825	-0.43358 to -0.44284
C(1)	0.09917 to 0.10696	0.09123 to 0.11396
O(1)	-0.28534 to -0.35978	-0.29118 to -0.36776

8.3.2 *Dithiocarbamate anions*

As with the *O*-alkyldithiocarbonate anions the *N,N*-dialkyldithiocarbamate anions reveal that the electron density of the $^{-}S_2CN$ moiety remains constant regardless of the R group coordinated to the nitrogen atom. The electron distribution of the $^{-}S_2CN$ moiety is summarised in Table 8.36. Similar to the *O*-alkyldithiocarbonate anions all calculations reveal a significant electron density on the S(1) and S(2) atoms. Unlike that observed for the analogous $^{-}S_2COR$ anions, the differences in charge residing on the two sulphur atoms is much smaller in the $^{-}S_2CNR_2$ anions, a result consistent with geometric parameters which showed a near equivalence of the C - S bond distances.

Table 8.36 General summary of the electron distribution over the [$^{-}S_2CN$] moiety.

Mulliken population analysis		
Atom	6-311++G**	LANL2DZ
S(1)	-0.77246 to -0.97430	-0.32122 to -0.34529
S(2)	-0.80149 to -0.97430	-0.32168 to -0.34529
C(1)	0.60509 to 1.21378	-0.31354 to -0.44724
N(1)	-0.461648 to 1.11088	-0.26441 to -0.68255

Natural population analysis		
Atom	6-311++G**	LANL2DZ
S(1)	-0.46526 to -0.49138	-0.47330 to -0.49546
S(2)	-0.46725 to -0.49138	-0.47330 to -0.49546
C(1)	0.06990 to 0.09839	0.08090 to 0.11196
N(1)	-0.27244 to -0.44213	-0.27654 to -0.44819

8.4 CONCLUSION

The *ab initio* molecular orbital calculations for both the *O*-alkyldithiocarbonate anions and *N,N*-dialkyldithiocarbamate anions reveal that the nature of R has little influence on

(i) the geometry of the respective $^{-}S_2CO$ and $^{-}S_2CN$ moieties, and

(ii) the electronic profiles of the respective $^{-}S_2CO$ and $^{-}S_2CN$ moieties.

Table 8.27 The mulliken electronic density distribution of [$^{-}S_2COR$] optimised using the 6-311++G** basis set[a].

Atom	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
S1	-0.70047	-0.72984	-0.71473	-0.73906	-0.75400	-0.75830	-0.76722	-0.77797	-0.73951	-0.77147	-0.75423	-0.77362	-0.77300	-0.76420	-0.83731
S2	-0.67940	-0.68387	-0.71038	-0.70410	-0.69914	-0.69552	-0.68159	-0.68743	-0.71386	-0.69563	-0.71336	-0.68864	-0.69447	-0.66549	-0.68126
C1	0.24091	0.32402	0.36538	0.42855	0.44025	0.45998	0.49058	0.48441	0.46442	0.47568	0.50125	0.48457	0.60000	0.51299	0.63339
O1	-0.06963	0.02065	0.12302	0.16866	0.22265	0.19310	0.23728	0.26033	0.32754	0.20065	0.21989	0.22620	0.25235	0.23921	0.22780
C2		-0.27240	-0.16135	-0.29608	-0.18393	-0.51962	-0.51549	-0.57544	-0.22493	-0.63845	-0.60028	-0.64643	-0.17267	-0.86398	-1.79228
C3			-0.54624	-0.32010	-0.47247	-0.31380	0.15374	-0.12427	-0.38750	-0.29422	-0.40145	-0.31467	-0.38939	-0.43798	-0.11462
C4				-0.42957	-0.43521	0.10709	-0.55325	-0.16975	-0.48887	-0.67880	0.51851	-0.08631	-0.31662	-0.71259	-0.54573
C5						-0.64250	-0.55359	-0.54753	-0.48887	0.11311	-0.64273	-0.15118	-0.36975	-0.66974	0.02895
C6										-0.11715	-0.63668	-0.06575	-0.12052	1.13682	-0.64630
C7												-0.69983	-0.60394	-0.57468	1.91482
C8															-0.55585
H	0.20860	0.08952	0.13502	0.10902	0.09999	0.14557	0.14736	0.10727	0.08778	0.09631	0.12318	0.12086	0.13797	0.10904	0.17395
H		0.12595	0.13502	0.09924	0.13991	0.11336	0.14730	0.12439	0.17557	0.13131	0.17104	0.15790	0.17553	0.14915	0.18765
H		0.12595	0.10528	0.15470	0.13901	0.16641	0.11143	0.15623	0.15945	0.12030	0.16799	0.12733	0.15563	0.11287	0.16427
H			0.13449	0.10612	0.08312	0.16169	0.11710	0.08816	0.10628	0.17631	0.13248	0.16889	0.14847	0.12275	0.20596
H			0.13449	0.13974	0.14376	0.12559	0.16189	0.15174	0.14984	0.15930	0.11613	0.15731	0.13265	0.12722	0.23046
H				0.15696	0.13437	0.11541	0.11139	0.12917	0.14984	0.12483	0.14834	0.12370	0.10380	0.11340	0.20303
H				0.12589	0.14166	0.11195	0.16184	0.15128	0.08778	0.10619	0.11366	0.13029	0.14385	0.12823	0.20303
H						0.12509	0.11712	0.13107	0.15945	0.11161	0.11874	0.10109	0.14564	0.12052	
H						0.10448	0.11407	0.09833	0.17557	0.14013	0.11885	0.11953	0.13180	0.14905	
H										0.12145	0.12408	0.12211	0.17521	0.14001	
H										0.11857	0.17455	0.11897	0.13745	0.14688	
H												0.12263		0.18887	
H												0.14501		0.19160	

a, 1=H, 2=Me, 3=Et, 4=nPr, 5=iPr,6=nBu, 7=iBu, 8=1-MePr, 9=tBu, 10=nPen, 11=3-MeBu, 12=Hex, 13=Cy, 14=3,3-Me_2Bu, 15=Benzyl

Table 8.28 The Mulliken electronic density distribution of [$^{-}S_2COR$] optimised using the LANL2DZ basis set[a].

Atom	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
S1	-0.33598	-0.31219	-0.31111	-0.30754	-0.32189	-0.30627	-0.30474	-0.31780	-0.32056	-0.30564	-0.30048	-0.30587	-0.31787	-0.30608	-0.28856
S2	-0.25922	-0.28229	-0.28343	-0.28412	-0.27942	-0.28435	-0.28427	-0.28038	-0.28193	-0.28449	-0.28494	-0.28128	-0.27636	-0.28047	-0.27232
C1	-0.25323	-0.29175	-0.24055	-0.24163	-0.24296	-0.24225	-0.24224	-0.24551	-0.23818	-0.24243	-0.24079	-0.25366	-0.26299	-0.25373	-0.25229
O1	-0.54345	-0.42715	-0.44149	-0.45145	-0.46522	-0.45123	-0.45933	-0.47782	-0.50386	-0.45137	-0.45987	-0.44124	-0.48130	-0.44540	-0.47083
C2		-0.23804	-0.02587	-0.04698	-0.56033	-0.07715	-0.06730	0.21417	-0.59082	-0.08604	-0.08007	-0.07261	-0.33065	-0.09860	-0.13175
C3			-0.54743	-0.27156	0.20389	-0.26656	-0.57605	-0.58370	0.48649	-0.28145	-0.29786	-0.27786	-0.31612	-0.29697	-0.23930
C4				-0.55994	-0.55740	-0.26256	-0.02226	-0.26168	-0.56364	-0.26282	-0.58258	-0.28556	-0.32905	-0.59902	-0.21484
C5						-0.57209	-0.57605	-0.58008	-0.59082	-0.26047	0.01142	-0.26238	-0.35576	-0.59902	-0.34470
C6										-0.57183	-0.58967	-0.26573	0.19837	0.25724	0.28439
C7												-0.57195	-0.35307	-0.58973	-0.34927
C8															-0.20312
H	0.39188	0.16314	0.17697	0.14791	0.1329	0.13085	0.18094	0.14265	0.12927	0.12360	0.13399	0.16508	0.20969	0.15080	0.20240
H		0.19413	0.17697	0.14292	0.16619	0.21728	0.18094	0.15419	0.24091	0.21108	0.16024	0.16432	0.15957	0.18554	0.19079
H		0.19413	0.13354	0.23099	0.22534	0.12057	0.14016	0.23278	0.17980	0.12277	0.17064	0.16432	0.18601	0.17030	0.19469
H			0.18119	0.12377	0.13157	0.15821	0.15603	0.13412	0.12529	0.15845	0.18482	0.14792	0.15467	0.15080	0.19703
H			0.18119	0.16364	0.19052	0.17047	0.22440	0.16509	0.18903	0.17108	0.13404	0.14792	0.14553	0.17030	0.29897
H				0.17068	0.17666	0.18603	0.14016	0.22605	0.18903	0.18622	0.13509	0.14354	0.13741	0.18554	0.19935
H				0.18328	0.20008	0.16575	0.22440	0.19780	0.12927	0.13074	0.28218	0.14354	0.15396	0.15513	0.19935
H						0.14749	0.15603	0.16009	0.17980	0.15667	0.16296	0.14053	0.15534	0.17003	
H						0.16581	0.12920	0.12000	0.24091	0.17629	0.15937	0.14053	0.15171	0.17003	
H										0.16019	0.15648	0.15925	0.14801	0.16818	
H										0.14944	0.14499	0.15925	0.22288	0.16818	
H												0.12096		0.18347	
H												0.12096		0.18347	

a, 1=H, 2=Me, 3=Et, 4=nPr, 5=iPr,6=nBu, 7=iBu, 8=1-MePr, 9=tBu, 10=nPen, 11=3-MeBu, 12=Hex, 13=Cy, 14=3,3-Me_2Bu, 15=Benzyl

Table 8.29 The natural population analysis electronic density distribution of [$^{-}S_2COR$] optimised using the 6311++G** basis set[a].

Atom	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
S1	-0.49216	-0.46907	-0.46974	-0.46866	-0.47313	-0.46842	-0.46784	-0.47213	-0.47257	-0.46830	-0.46614	-0.46887	-0.47233	-0.46959	-0.46101
S2	-0.44166	-0.44602	-0.44723	-0.44792	-0.44404	-0.44768	-0.44825	-0.44493	-0.44475	-0.44768	-0.44763	-0.44643	-0.44256	-0.44624	-0.43889
C1	0.10254	0.09917	0.09966	0.09990	0.10253	0.09974	0.10022	0.10256	0.10696	0.09973	0.09922	0.09968	0.10264	0.09944	0.09790
O1	-0.35978	-0.28534	-0.29486	-0.29663	-0.30373	-0.29667	-0.29817	-0.30527	-0.31525	-0.29669	-0.29737	-0.29489	-0.30304	-0.29439	-0.29376
C2		0.03615	0.10808	0.10105	-0.14927	0.10077	0.09587	0.16396	0.25484	0.10100	0.10062	0.10208	-0.05589	0.09663	0.08204
C3			-0.13858	-0.05799	0.17112	-0.06437	-0.1303	-0.06167	-0.16122	-0.06451	-0.07147	-0.06951	-0.05575	-0.07855	-0.05469
C4				-0.12511	-0.14256	-0.05015	0.01821	-0.14941	-0.16122	-0.05698	-0.13681	-0.05376	-0.05608	-0.13689	-0.04283
C5						-0.12382	-0.13031	-0.12638	-0.14372	-0.04602	0.03136	-0.05434	-0.07742	-0.13689	-0.05661
C6										-0.12569	-0.12856	-0.04797	0.15468	0.09968	0.03190
C7												-0.12597	-0.0839	-0.13301	-0.04583
C8															-0.03875
H	0.19107	0.01423	0.0191	0.03139	0.02327	0.02476	0.01778	0.0226	0.02051	0.02312	0.01677	0.03342	0.02702	0.02593	0.02703
H		0.02544	0.0191	0.02327	0.04076	0.05037	0.01776	0.03406	0.06201	0.04892	0.02937	0.03578	0.02902	0.04308	0.02208
H		0.02544	0.02533	0.05612	0.03658	0.01529	0.02178	0.05695	0.03758	0.01533	0.01756	0.03578	0.03323	0.03477	0.02178
H			0.03957	0.01646	0.02273	0.03014	0.03224	0.02295	0.0196	0.03020	0.02003	0.02733	0.01866	0.02593	0.02556
H			0.03957	0.0313	0.03312	0.01802	0.05387	0.03299	0.03857	0.01804	0.02293	0.02733	0.02402	0.03477	0.06236
H				0.01803	0.05307	0.01936	0.02178	0.05361	0.03857	0.01945	0.02845	0.02543	0.02207	0.04308	0.03086
H				0.01879	0.02954	0.0306	0.05386	0.02782	0.02051	0.02047	0.07439	0.02543	0.01821	0.02580	0.03086
H						0.02805	0.03223	0.02814	0.03758	0.02929	0.02811	0.02506	0.01951	0.03314	
H						0.034	0.00928	0.01414	0.06201	0.03972	0.02872	0.02506	0.02621	0.03314	
H										0.03400	0.02962	0.03199	0.02606	0.03106	
H										0.02659	0.02084	0.03199	0.04564	0.03106	
H												0.01769		0.01903	
H												0.01769		0.01903	

a, 1=H, 2=Me, 3=Et, 4=nPr, 5=iPr,6=nBu, 7=iBu, 8=1-MePr, 9=tBu, 10=nPen, 11=3-MeBu, 12=Hex, 13=Cy, 14=3,3-Me_2Bu, 15=Benzyl

Table 8.30 The natural population analysis electronic density distribution of [$^{-}S_2COR$] optimised using the LANL2DZ basis set[a].

Atom	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
S1	-0.49185	-0.47405	-0.47503	-0.47444	-0.47833	-0.47368	-0.47299	-0.47718	-0.47838	-0.47354	-0.47105	-0.47411	-0.47728	-0.47468	-0.46641
S2	-0.43636	-0.44081	-0.44189	-0.44136	-0.43899	-0.44228	-0.44284	-0.43988	-0.43959	-0.44230	-0.44228	-0.44073	-0.43757	-0.44050	-0.43358
C1	0.11396	0.10979	0.11005	0.11032	0.11256	0.11012	0.11072	0.11264	0.11767	0.11011	0.10981	0.11005	0.11294	0.09123	0.10893
O1	-0.36776	-0.29118	-0.30105	-0.30158	-0.31091	-0.30315	-0.30494	-0.31270	-0.32358	-0.30316	-0.30423	-0.30127	-0.31067	-0.30097	-0.30039
C2		0.02939	0.10317	0.09676	-0.15105	0.09518	0.08981	0.16283	0.25367	0.09543	0.09550	0.09646	-0.05476	-0.13830	0.07418
C3			-0.14014	-0.06232	0.17033	-0.06450	-0.13111	-0.15123	-0.16279	-0.06464	-0.13809	-0.06959	-0.05464	-0.13830	-0.04162
C4				-0.12381	-0.14479	-0.04808	0.02057	-0.12623	-0.16279	-0.05561	-0.07231	-0.05223	-0.07792	-0.07995	-0.05593
C5						-0.12394	-0.13111	-0.06199	-0.14724	-0.04429	0.03486	-0.05338	0.15326	0.10456	0.03261
C6										-0.12575	-0.12942	-0.04619	-0.08393	-0.13450	-0.04412
C7												-0.12600	-0.05466	0.10456	-0.03761
															-0.05321
H	0.18200	0.01509	0.01895	0.02713	0.02273	0.02403	0.01782	0.02264	0.02056	0.02253	0.01694	0.03323	0.02392	0.02614	0.02590
H		0.02588	0.01895	0.03368	0.03373	0.05054	0.01782	0.03328	0.06260	0.04928	0.02974	0.03571	0.02970	0.04301	0.02092
H		0.02588	0.02602	0.03368	0.05483	0.01541	0.02188	0.05796	0.03815	0.01544	0.01762	0.03571	0.03340	0.03492	0.02057
H			0.04048	0.03348	0.02401	0.03062	0.03197	0.02295	0.02080	0.03066	0.01980	0.02666	0.01815	0.02614	0.02453
H			0.04048	0.03348	0.04169	0.01811	0.05502	0.03358	0.03982	0.01815	0.02313	0.02666	0.02346	0.03492	0.06283
H				0.01749	0.03783	0.01933	0.02187	0.05540	0.03982	0.01939	0.02717	0.02497	0.02172	0.04300	0.03121
H				0.01749	0.02639	0.03039	0.05502	0.02472	0.02056	0.01972	0.07635	0.02497	0.01760	0.02596	0.03121
H						0.02791	0.03197	0.02897	0.03815	0.02904	0.02815	0.02457	0.01895	0.03318	
H						0.03397	0.00852	0.01423	0.06260	0.03973	0.02851	0.02457	0.02556	0.03318	
H										0.03382	0.02940	0.03229	0.02601	0.03160	
H										0.02597	0.02045	0.03229	0.04678	0.03160	
H												0.01767		0.01895	
H												0.01767		0.01895	

a, 1=H, 2=Me, 3=Et, 4=nPr, 5=iPr,6=nBu, 7=iBu, 8=1-MePr, 9=tBu, 10=nPen, 11=3-MeBu, 12=Hex, 13=Cy, 14=3,3-Me_2Bu, 15=Benzyl

Table 8.31 The Mulliken electronic density distribution of [$^{-}S_2CNR_2$] optimised using the 6-311++G** basis set[a].

Atom	1	2	3	4	5	6	7	8
S1	-0.97429	-0.89450	-0.88251	-0.92212	-0.82239	-0.93244	-0.89851	-0.85426
S2	-0.97432	-0.89027	-0.88249	-0.92225	-0.82241	-0.93262	-0.89854	-0.85429
C1	0.88143	0.60508	0.61031	0.75484	0.77267	0.82942	0.83635	1.21377
N1	-0.46164	0.16450	0.41735	0.58592	0.64778	0.67862	0.74801	1.11088
C1		-0.33414	-0.43675	-0.76533	-0.13641	-0.79127	-1.08766	0.08459
C2		-0.33269	-0.26916	0.38355	-0.64884	0.32638	0.96348	-0.55062
C3			-0.26917	-0.73431	-0.48003	-0.26428	-0.71796	-0.75399
C4			-0.43674	-0.73430	-0.13664	-0.70534	-0.76672	0.01963
C5				0.38359	-0.64868	-0.79141	-1.08753	-0.70895
C6				-0.76536	-0.48005	0.32649	0.96348	-0.40572
C7						-0.26428	-0.71796	0.08459
C8						-0.70545	-0.76671	-0.55065
C9								-0.75401
C10								0.01964
C11								-0.70897
C12								-0.40569
C13								0.01493
H	0.26441	0.05572	0.11182	0.11868	0.10358	0.08899	0.11509	0.14198
H	0.26441	0.13461	0.08014	0.13477	0.11228	0.18509	0.18110	0.18795
H		0.15041	0.15437	0.10832	0.14477	0.08485	0.14773	0.14344
H		0.06655	0.08014	0.11868	0.12653	0.18509	0.12699	0.16274
H		0.14577	0.15436	0.13478	0.15880	0.08487	0.11071	0.10322
H		0.12895	0.11182	0.10832	0.12628	0.08902	0.13988	0.15786
H			0.07242	0.09647	0.10357	0.13873	0.12699	0.14749
H			0.15582	0.18057	0.11227	0.13876	0.11071	0.17571
H			0.07241	0.08294	0.14475	0.11375	0.13987	0.12222
H			0.15583	0.18061	0.12655	0.11873	0.11507	0.14945
H				0.08293	0.15881	0.13957	0.18111	0.01494
H				0.09647	0.12629	0.11374	0.14773	0.14198
H				0.14609	0.10529	0.11874	0.10067	0.18795
H				0.14610	0.10521	0.13958	0.10066	0.14344
H						0.10913	0.12931	0.16273
H						0.13414	0.16367	0.10322
H						0.13418	0.12929	0.15786
H						0.10914	0.16373	0.14749
H								0.17571
H								0.12222
H								0.14946

a, 1=H, 2=Me, 3=Et, 4=nPr, 5=iPr, 6=nBu, 7=iBu, 8=Cy, 9= Cy, Et, 10=Cy, iPr.

Table 8.31 Cont.

Atom	9	10
S1	-0.77245	-0.79394
S2	-0.89289	-0.80148
C1	0.80822	0.76967
N1	0.84898	0.99294
C1	-0.05384	0.25711
C2	-0.52803	-0.64344
C3	-1.25276	-0.64313
C4	-0.42011	-1.08550
C5	0.05314	0.11815
C6	0.28869	-0.55177
C7	-0.64556	-0.46431
C8	-0.46647	-0.55102
C9		0.11983
C10		
C11		
C12		
C13		
H	-0.04789	0.08151
H	0.13623	0.13980
H	0.16172	0.09095
H	0.14817	0.18943
H	0.15681	0.09088
H	0.10461	0.13983
H	0.14583	0.18944
H	0.14484	-0.15564
H	0.15695	0.17723
H	0.12631	0.13760
H	0.19170	0.16838
H	0.16960	0.14577
H	0.08535	0.14758
H	0.09161	0.10518
H	0.11153	0.14578
H	0.14968	0.16838
H		0.13765
H		0.17709
H		
H		
H		

Table 8.32 The Mulliken electronic density distribution of [$^{-}S_2CNR_2$] optimised using the LANL2DZ basis set[a].

Atom	1	2	3	4	5	6	7	8
S1	-0.34529	-0.33055	-0.33436	-0.33175	-0.32681	-0.33064	-0.32168	-0.32168
S2	-0.34529	-0.33055	-0.33436	-0.33174	-0.32681	-0.33063	-0.32168	-0.32168
C1	-0.31354	-0.35992	-0.37735	-0.40212	-0.39261	-0.41057	-0.40587	-0.44724
N1	-0.68254	-0.26441	-0.28912	-0.29965	-0.39659	-0.29261	-0.32569	-0.44321
C2		-0.41527	-0.18369	-0.19419	0.13210	-0.22613	-0.21680	0.15711
C3		-0.41527	-0.53187	-0.23970	-0.58820	-0.23502	0.01529	-0.33442
C4			-0.18370	-0.57997	-0.57099	-0.26760	-0.59182	-0.32043
C5			-0.53187	-0.19427	0.13211	-0.57652	-0.56725	-0.35017
C6				-0.57999	-0.58822	-0.22623	-0.21679	-0.34285
C7				-0.23966	-0.57098	-0.23497	0.01528	-0.34425
C8						-0.26761	-0.56726	0.15711
C9						-0.57653	-0.59182	-0.33443
C10								-0.32043
C11								-0.35017
C12								-0.34285
C13								-0.34425
H	0.34333	0.12895	0.14502	0.16080	0.14338	0.12043	0.24775	0.21667
H	0.34333	0.21449	0.13705	0.18084	0.15038	0.23817	0.14384	0.22331
H		0.21455	0.22892	0.14643	0.22528	0.13331	0.13059	0.13482
H		0.12894	0.1370	0.16080	0.15629	0.23818	0.15753	0.15871
H		0.21440	0.22890	0.18084	0.19171	0.13332	0.15804	0.17131
H		0.21463	0.14502	0.14644	0.15226	0.12044	0.17593	0.14750
H			0.13646	0.12741	0.14338	0.20986	0.21460	0.15291
H			0.2357	0.23425	0.15036	0.20992	0.19084	0.13313
H			0.13645	0.13193	0.22528	0.15623	0.12890	0.14670
H			0.23572	0.23424	0.15630	0.15721	0.14384	0.18511
H				0.13193	0.19172	0.17730	0.24775	0.13173
H				0.12739	0.15226	0.15624	0.13059	0.21666
H				0.21489	0.22918	0.15721	0.21460	0.22331
H				0.21483	0.22918	0.17729	0.12891	0.17131
H						0.12975	0.19083	0.14750
H						0.16522	0.15753	0.15291
H						0.16523	0.15804	0.13313
H						0.12973	0.17593	0.15871
H								0.14670
H								0.18511
H								0.13173
H								0.13481

a 1=H, 2=Me, 3=Et, 4=nPr, 5=iPr, 6=nBu, 7=iBu, 8=Cy, 9= Cy, Et, 10=Cy, iPr.

Table 8.32 Cont.

Atom	9	10
S1	-0.32648	-0.32121
S2	-0.33147	-0.33286
C1	-0.41692	-0.42434
N1	-0.35530	-0.39234
C2	-0.17993	0.17404
C3	-0.54962	-0.57700
C4	0.11721	-0.57702
C5	-0.32942	0.11450
C6	-0.33264	-0.33040
C7	-0.32021	-0.33642
C8	-0.33932	-0.32006
C9	-0.32955	-0.33641
C10		-0.33037
C11		
C12		
C13		
H	0.13484	0.13033
H	0.23161	0.12259
H	0.15584	0.24116
H	0.23621	0.16806
H	0.13379	0.24115
H	0.23871	0.12260
H	0.21758	0.16808
H	0.12323	0.23561
H	0.16368	0.19889
H	0.14851	0.12753
H	0.13269	0.16189
H	0.15496	0.14774
H	0.14782	0.13248
H	0.15663	0.15555
H	0.13039	0.14774
H	0.18712	0.16193
H		0.19903
H		0.12749
H		
H		
H		

Table 8.33 The natural population analysis electronic density distribution of $[^{-}S_2CNR_2]$ optimised using the 6-311++G** basis set[a].

Atom	1	2	3	4	5	6	7	8
S1	-0.49138	-0.47588	-0.47682	-0.47586	-0.46724	-0.47556	-0.46901	-0.46526
S2	-0.49137	-0.47498	-0.47682	-0.47586	-0.46725	-0.47556	-0.46900	-0.46526
C1	0.09839	0.06990	0.07318	0.07270	0.07337	0.07257	0.07587	0.07341
N1	-0.44213	-0.27244	-0.28646	-0.28650	-0.30197	-0.28599	-0.28863	-0.30065
C2		-0.00772	0.06252	0.05677	0.11870	0.05663	0.03911	0.10110
C3		-0.00920	-0.13546	-0.06098	-0.14026	-0.06764	0.02038	-0.07514
C4			0.06252	-0.12344	-0.13889	-0.04650	-0.12890	-0.05585
C5			-0.13546	0.05677	0.11867	-0.12463	-0.13360	-0.05717
C6				-0.06098	-0.14025	0.05663	0.03912	-0.05585
C7				-0.12344	-0.13889	-0.06764	0.02038	-0.07202
C8						-0.04651	-0.13360	0.10110
C9						-0.12463	-0.12890	-0.07515
C10								-0.05585
C11								-0.05717
C12								-0.05558
C13								-0.07202
H	0.16325	0.01443	0.02555	0.02626	0.05702	0.01770	0.05787	0.05415
H	0.16325	0.04955	0.02072	0.03719	0.02179	0.04881	0.00543	0.04367
H		0.02087	0.05323	0.02558	0.02303	0.00556	0.01067	0.01637
H		0.01386	0.02072	0.02626	0.04987	0.04881	0.02747	0.02753
H		0.01748	0.05322	0.03719	0.02512	0.00555	0.02376	0.03135
H		0.05415	0.02555	0.02558	0.02603	0.01770	0.03541	0.01720
H			0.00638	0.01915	0.03915	0.04681	0.05341	0.01819
H			0.05052	0.04878	0.05703	0.04679	0.04600	0.01875
H			0.00637	0.00535	0.03916	0.02743	0.01836	0.01631
H			0.05052	0.04876	0.02603	0.03108	0.00543	0.03243
H				0.00536	0.02512	0.03813	0.05786	0.01759
H				0.01915	0.04986	0.02743	0.01068	0.05415
H				0.04810	0.02300	0.03108	0.05342	0.04367
H				0.04811	0.02180	0.03814	0.01837	0.03134
H						0.01844	0.04599	0.01719
H						0.03045	0.02748	0.01819
H						0.03046	0.02376	0.01876
H						0.01844	0.03541	0.02753
H								0.01631
H								0.03243
H								0.01759
H								0.01638

a 1=H, 2=Me, 3=Et, 4=nPr, 5=iPr, 6=nBu, 7=iBu, 8=Cy, 9= Cy, Et, 10=Cy, iPr.

Table 8.33 Cont.

Atom	9	10
S1	-0.46923	-0.46622
S2	-0.47294	-0.46995
C1	0.07267	0.07435
N1	-0.29286	-0.29810
C1	0.06236	0.14522
C2	-0.13666	-0.14339
C3	0.10251	-0.14340
C4	-0.07333	0.09618
C5	-0.05723	-0.07216
C6	-0.05569	-0.05664
C7	-0.05609	-0.05560
C8	-0.07150	-0.05665
C9		-0.07217
C10		
C11		
C12		
C13	0.00851	0.00628
H	0.04493	0.01684
H	0.02647	0.06117
H	0.05669	0.03306
H	0.02023	0.06117
H	0.05519	0.01685
H	0.04155	0.03308
H	0.01491	0.05927
H	0.02915	0.03532
H	0.01792	0.01561
H	0.01944	0.02849
H	0.01836	0.01718
H	0.01707	0.01921
H	0.02708	0.01833
H	0.01796	0.01719
H	0.0325	0.0285
H		0.03538
H		0.01559
H		
H		
H		
H		

Table 8.34 The natural population analysis electronic density distribution of $[^{-}S_2CNR_2]$ optimised using the LANL2DZ basis set[a].

Atom	1	2	3	4	5	6	7	8
S1	-0.49546	-0.48552	-0.48323	-0.48268	-0.47497	-0.48254	-0.47688	-0.47330
S2	-0.49546	-0.48551	-0.48323	-0.48268	-0.47497	-0.48254	-0.47688	-0.47330
C1	0.11196	-0.00493	0.08486	0.08488	0.08568	0.08472	0.08773	0.08581
N1	-0.44819	-0.27654	-0.29029	-0.29079	-0.30825	-0.29014	-0.29257	-0.30686
C2		0.08090	-0.13590	-0.12378	0.12358	0.05731	0.04057	0.10505
C3		-0.00492	0.06397	-0.05906	-0.14157	-0.06646	0.02365	-0.07477
C4			0.06397	0.05747	-0.14054	-0.04479	-0.12993	-0.05433
C5			-0.13590	0.05747	0.12358	-0.12477	-0.13478	-0.05597
C6				-0.05905	-0.14157	0.05731	0.04056	-0.05482
C7				-0.12378	-0.14054	-0.06646	0.02365	-0.07207
C8						-0.04479	-0.13478	0.10505
C9						-0.12477	-0.12993	-0.07477
C10								-0.05433
C11								-0.05597
C12								-0.05482
C13								-0.07207
H	0.16357	0.01607	0.02589	0.02553	0.05477	0.01732	0.00621	0.05207
H	0.16357	0.03608	0.02101	0.02649	0.02255	0.04847	0.05666	0.03223
H		0.03611	0.05451	0.03743	0.05135	0.00600	0.00997	0.01795
H		0.01607	0.02101	0.02649	0.02338	0.04847	0.02727	0.01603
H		0.03603	0.05450	0.03743	0.02600	0.00602	0.02427	0.02726
H		0.03616	0.02590	0.02554	0.02706	0.01733	0.03556	0.01777
H			0.00664	0.01873	0.03969	0.04748	0.05469	0.01837
H			0.04983	0.04839	0.05478	0.04752	0.04662	0.01695
H			0.00664	0.00583	0.05135	0.02737	0.01853	0.03136
H			0.04983	0.04839	0.02339	0.03103	0.05666	0.01607
H				0.00582	0.02254	0.03837	0.00621	0.04467
H				0.01871	0.02705	0.02738	0.00997	0.05207
H				0.04862	0.02600	0.03104	0.04662	0.01606
H				0.04858	0.03968	0.03836	0.01853	0.04467
H						0.01773	0.05469	0.01696
H						0.03016	0.03556	0.03137
H						0.03016	0.02427	0.01777
H						0.01772	0.02727	0.01837
H								0.01603
H								0.02726
H								0.03223
H								0.01795

a 1=H, 2=Me, 3=Et, 4=nPr, 5=iPr, 6=nBu, 7=iBu, 8=Cy, 9= Cy, Et, 10=Cy, iPr.

Table 8.34 Cont.

Atom	9	10
S1	-0.47707	-0.47736
S2	-0.48026	-0.47363
C1	0.08532	0.08709
N1	-0.29787	-0.30342
C2	0.06369	0.14736
C3	-0.13758	-0.14529
C4	0.10589	-0.14528
C5	-0.07268	0.09965
C6	-0.05621	-0.07202
C7	-0.05464	-0.05548
C8	-0.05482	-0.05454
C9	-0.07148	-0.05548
C10		-0.07201
C11		
C12		
C13		
H	0.00844	0.00497
H	0.04510	0.01739
H	0.02643	0.06311
H	0.05824	0.03305
H	0.02083	0.03306
H	0.05361	0.06311
H	0.01491	0.01739
H	0.04209	0.05800
H	0.01770	0.03542
H	0.02900	0.01599
H	0.01912	0.02833
H	0.01796	0.01700
H	0.01682	0.01797
H	0.02685	0.01893
H	0.03223	0.02832
H	0.01838	0.01700
H		0.03537
H		0.01601
H		
H		
H		
H		

CONCLUSIONS

The systematic investigation of the crystal and molecular structures of a series of nickel(II) and tellurium(II) bis(xanthates and bis(dithiocarbamates) showed, conclusively, that no significant influence on molecular structure and on the geometric parameters describing the central atom geometry was exerted by the ligand-bound R substituents. This result was supported by *ab initio* molecular orbital calculations performed on the uncoordinated xanthate and dithiocarbamate anions.

Thus, neither the steric bulk nor the electronic profile of the respective R groups in both xanthate and dithiocarbamate ligands have little influence on the coordination ability of these ligands. Despite the above structural diversity was observed in the bis(xanthate) and bis(dithiocarbamate) compounds of zinc(II), cadmium(II) and mercury(II).

Of the three systems studied, the cadmium bis(1,1-dithiolates) displayed the least structural diversity. There was one motif only for $Cd(S_2COR)_2$ and two, closely related, for $Cd(S_2CNR_2)_2$.

The zinc(II) compounds displayed several motifs. It is apparent that the steric profiles of the ligands play a significant role in determining the ultimate structure adopted in the solid state. This is supported by the results of the related mercury(II) compounds for which more structures are available for examination.

The mercury(II) bis(1,1-dithiolate) compounds showed the greatest structural diversity. The different structures in each series may be explained in terms of a trade

off between the requirements of satisfying the valency of the mercury(II) centre and the need to maximise intermolecular forces, *i.e.* packing efficiency.

The systematic analysis of structures for which no structural diversity is observed and those where this phenomenon is found illustrates the importance of the careful consideration of intermolecular forces/crystal packing effects when describing molecular structure.

Crystal packing effects may exert a significant influence on molecular structure in heavy element systems such as those of zinc(II), cadmium(II) and mercury(II).

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APPENDIX

APPENDIX 3.I

$[Ni(S_2COMe)_2]$

Table 3.I.1: Crystallographic Parameters for $[Ni(S_2COCH_3)_2]$

Formula	$C_4H_6NiO_2S_4$
Formula weight	273.03
Crystal morphology / colour	Block / brown
Crystal dimensions (mm)	0.16 x 0.16 x 0.32
Crystal system	monoclinic
Space group	$P2_1/c$ (#14)
a (Å)	6.357(4)
b (Å)	13.76(1)
c (Å)	10.566(3)
α (°)	90
β (°)	97.20(3)
γ (°)	90
V (Å^3)	916.5(7)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.979
$F(000)$	552
Temperature (°K)	293
μ (cm^{-1})	29.51
θ limits, cell (°)	24.6 – 27.3
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 8, 0 – 17, -13 – 13
Range of transmission factors	0.952 – 1
Scan technique	ω:2θ
No. of data measured	2391
No. of unique data	2214
R_{amal}	0.168
No. of parameters	100
R (I≥0σ(I))	0.090
R_w (I≥0σ(I))	0.122
R (I≥3σ(I))	0.047
R_w (I≥3σ(I))	0.058
Maximum and minimum residual electron density (e Å^{-3})	1.12, -1.19

Table 3.I.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Ni(S_2COCH_3)_2]$

Atom	*x*	*y*	*z*
Ni	0.4097(1)	0.11150(5)	0.39753(5)
S(1)	0.5854(2)	0.1185(1)	0.5915(1)
S(2)	0.1434(2)	0.1213(1)	0.5144(1)
S(3)	0.6764(2)	0.1206(1)	0.2810(1)
S(4)	0.2341(2)	0.1111(1)	0.2023(1)
O(1)	0.3180(5)	0.1353(3)	0.7583(3)
O(2)	0.5011(5)	0.1193(3)	0.0358(3)
C(1)	0.3387(8)	0.1266(4)	0.6373(4)
C(2)	0.1061(9)	0.1379(5)	0.7947(5)
C(3)	0.4787(8)	0.1182(4)	0.1563(4)
C(4)	0.7104(8)	0.1269(5)	-0.0028(5)

Table 3.I.3: Anisotropic thermal parameters (Å^2) for the non-hydrogen atoms in [Ni(S_2COCH_3)$_2$]

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	0.0341(3)	0.0287(5)	0.0323(3)	-0.0002(4)	0.0036(2)	-0.0002(4)
S(1)	0.0335(6)	0.042(1)	0.0380(6)	0.0001(9)	0.0023(5)	0.0009(8)
S(2)	0.0342(7)	0.043(1)	0.0366(6)	0.0004(7)	0.0028(5)	-0.0037(7)
S(3)	0.0335(6)	0.040(1)	0.0361(6)	-0.0011(7)	0.0030(5)	0.0029(7)
S(4)	0.0329(6)	0.043(1)	0.0380(6)	-0.0003(8)	0.0010(4)	-0.0006(8)
O(1)	0.040(2)	0.041(3)	0.036(2)	-0.002(2)	0.005(1)	-0.003(2)
O(2)	0.041(2)	0.056(3)	0.031(2)	-0.001(2)	0.005(1)	-0.005(2)
C(1)	0.037(3)	0.019(4)	0.038(2)	-0.002(2)	0.004(2)	-0.003(2)
C(2)	0.048(3)	0.049(5)	0.043(3)	-0.003(3)	0.014(2)	-0.004(3)
C(3)	0.045(3)	0.020(3)	0.034(2)	0.003(3)	0.004(2)	-0.004(3)
C(4)	0.043(3)	0.071(6)	0.039(3)	0.002(3)	0.012(2)	-0.002(3)

Table 3.I.4: Fractional atomic coordinates and B_{eq} values (Å^2) for the hydrogen atoms in [Ni(S_2COCH_3)$_2$]*

Atom	x	y	z	Beq
H(2a)	0.1142	0.1458	0.8846	4.3553
H(2b)	0.0302	0.1909	0.7534	4.3553
H(2c)	0.0349	0.0789	0.7702	4.3553
H(4a)	0.7975	0.0735	0.0337	4.7681
H(4b)	0.7002	0.1242	-0.0951	4.7681
H(4c)	0.7741	0.1881	0.0271	4.7681

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table: 3.I.5: Bond distances (Å) for $[Ni(S_2COCH_3)_2]$

Ni — S(1)	2.210(1)	S(3) — C(3)	1.704(5)
Ni — S(2)	2.221(2)	S(4) — C(3)	1.689(5)
Ni — S(3)	2.220(2)	O(1) — C(1)	1.306(5)
Ni — S(4)	2.219(1)	O(1) — C(2)	1.447(6)
S(1) — C(1)	1.701(5)	O(2) — C(3)	1.299(5)
S(2) — C(1)	1.682(5)	O(2) — C(4)	1.443(6)

Table: 3.I.6: Bond angles (deg.) for $[Ni(S_2COCH_3)_2]$

S(1) — Ni — S(2)	79.23(6)	Ni — S(4) — C(3)	83.9(2)
S(1) — Ni — S(3)	100.34(6)	C(1) — O(1) — C(2)	118.3(4)
S(1) — Ni — S(4)	177.63(7)	C(3) — O(2) — C(4)	119.7(4)
S(2) — Ni — S(3)	173.31(7)	S(1) — C(1) — S(2)	113.3(3)
S(2) — Ni — S(4)	100.80(6)	S(1) — C(1) — O(1)	119.6(3)
S(3) — Ni — S(4)	79.34(6)	S(2) — C(1) — O(1)	127.2(4)
Ni — S(1) — C(1)	83.7(2)	S(3) — C(3) — S(4)	113.3(3)
Ni — S(2) — C(1)	83.8(2)	S(3) — C(3) — O(2)	126.7(4)
Ni — S(3) — C(3)	83.5(2)	S(4) — C(3) — O(2)	120.1(3)

Table 3.I.7: Observed and calculated structure factors for $[Ni(S_2COCH_3)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	2	30	12	23	0	4	11	88	82	99	0	9	2	107	92	99	0	14	3	95	109	99	1	1	5	479	474	99
0	0	4	199	192	99	0	4	12	-31	4	99	0	9	3	53	17	99	0	14	4	177	175	99	1	1	6	-10	15	95
0	0	6	449	468	99	0	4	13	-28	5	99	0	9	4	226	209	99	0	14	5	-132	11	99	1	1	7	205	206	99
0	0	8	285	308	99	0	5	1	926	916	99	0	9	5	639	579	99	0	14	6	134	135	99	1	1	8	94	103	78
0	0	10	596	655	99	0	5	2	46	40	95	0	9	6	146	137	99	0	14	7	88	14	99	1	1	9	649	637	99
0	0	12	-53	8	99	0	5	3	12	15	99	0	9	7	99	71	99	0	14	8	-111	11	99	1	1	10	248	238	99
0	1	-9	534	457	99	0	5	4	193	186	99	0	9	8	151	145	99	0	15	1	59	22	99	1	1	11	66	67	85
0	1	2	56	45	34	0	5	5	859	835	99	0	9	9	411	396	99	0	15	2	-93	22	99	1	1	12	62	74	83
0	1	3	26	11	74	0	5	6	-49	16	99	0	9	10	-51	27	99	0	15	3	47	4	99	1	1	13	97	85	93
0	1	4	141	118	99	0	5	7	65	62	99	0	9	11	140	149	99	0	15	4	-110	43	99	1	2	-13	91	93	99
0	1	5	1025	888	99	0	5	8	298	301	99	0	10	0	684	696	99	0	15	5	129	137	99	1	2	-12	25	22	99
0	1	6	259	247	99	0	5	9	467	481	99	0	10	1	320	323	99	0	15	6	85	112	99	1	2	-11	-30	75	99
0	1	7	8	43	99	0	5	10	71	55	99	0	10	2	57	80	99	0	15	7	104	9	99	1	2	-10	-58	29	99
0	1	8	444	453	99	0	5	11	177	191	99	0	10	3	64	103	99	0	16	0	362	396	99	1	2	-9	91	80	99
0	1	10	72	76	84	0	5	12	-17	54	99	0	10	4	252	235	99	0	16	1	244	275	99	1	2	-8	45	5	99
0	1	11	183	187	99	0	5	13	56	45	97	0	10	5	-33	38	99	0	16	2	82	19	99	1	2	-7	798	783	99
0	1	12	41	42	69	0	6	0	670	700	99	0	10	6	204	179	99	0	16	3	-91	31	99	1	2	-6	214	203	99
0	1	13	57	48	73	0	6	1	607	613	99	0	10	7	104	71	99	0	16	4	64	22	99	1	2	-5	704	677	99
0	2	1	821	866	99	0	6	2	96	98	99	0	10	8	-28	8	99	0	16	5	-25	8	99	1	2	-4	30	13	99
0	2	2	52	53	47	0	6	3	39	54	99	0	10	9	152	135	99	0	16	6	-140	5	99	1	2	-3	619	591	99
0	2	3	34	33	50	0	6	4	252	236	99	0	10	10	226	221	99	0	17	1	215	214	99	1	2	-2	62	57	41
0	2	4	118	114	76	0	6	5	76	75	99	0	10	11	94	93	99	0	17	2	107	100	99	1	2	-1	65	37	50
0	2	5	114	113	77	0	6	6	155	164	99	0	11	1	50	79	99	0	17	3	-75	5	99	1	2	0	220	223	99
0	2	6	77	66	66	0	6	7	127	136	99	0	11	2	58	74	99	0	17	4	-77	121	99	1	2	1	1242	1290	99
0	2	7	145	149	99	0	6	8	-35	5	99	0	11	3	22	5	99	1	0	-12	148	143	99	1	2	2	64	4	57
0	2	8	-43	5	99	0	6	9	243	239	99	0	11	4	110	91	99	1	0	-10	50	44	99	1	2	3	1189	1237	99
0	2	9	341	317	99	0	6	10	193	200	99	0	11	5	43	75	99	1	0	-8	77	68	99	1	2	4	227	237	99
0	2	10	97	83	85	0	6	11	180	182	99	0	11	6	249	237	99	1	0	-6	1571	1500	99	1	2	5	433	452	99
0	2	11	238	253	99	0	6	12	55	16	99	0	11	7	69	2	99	1	0	-4	90	77	67	1	2	6	21	29	99
0	2	12	14	8	78	0	7	1	333	302	99	0	11	8	297	275	99	1	0	-2	371	308	99	1	2	7	686	700	99
0	2	13	105	110	93	0	7	2	111	114	99	0	11	9	36	51	99	1	0	0	1804	1757	99	1	2	8	-27	24	99
0	3	1	600	619	99	0	7	3	-38	11	99	0	11	10	66	51	99	1	0	2	94	50	51	1	2	9	88	85	85
0	3	2	-19	44	99	0	7	4	135	123	99	0	12	0	940	973	99	1	0	4	1841	1678	99	1	2	10	70	62	83
0	3	3	11	6	99	0	7	5	178	158	99	0	12	1	448	435	99	1	0	6	17	11	81	1	2	11	418	406	99
0	3	4	39	42	65	0	7	6	395	362	99	0	12	2	46	34	99	1	0	8	284	284	99	1	2	12	-63	9	99
0	3	5	592	571	99	0	7	7	44	15	99	0	12	3	-49	44	99	1	0	10	428	431	99	1	2	13	74	59	90
0	3	6	391	390	99	0	7	8	399	400	99	0	12	4	77	25	99	1	0	12	36	40	99	1	3	-13	39	12	99
0	3	7	-9	28	99	0	7	9	173	172	99	0	12	5	-74	3	99	1	1	-13	-22	12	99	1	3	-12	161	166	99
0	3	8	484	486	99	0	7	10	63	71	99	0	12	6	-117	85	99	1	1	-12	114	117	99	1	3	-11	128	121	99
0	3	9	337	330	99	0	7	11	52	55	99	0	12	7	185	153	99	1	1	-11	218	221	99	1	3	-10	16	15	99
0	3	10	85	82	98	0	7	12	35	13	99	0	12	8	133	118	99	1	1	-10	24	24	99	1	3	-9	-27	38	99
0	3	11	127	130	99	0	8	0	1968	2034	99	0	12	9	111	96	99	1	1	-9	37	29	99	1	3	-8	357	341	99
0	3	12	9	24	99	0	8	1	542	555	99	0	12	10	221	188	99	1	1	-8	326	298	99	1	3	-7	448	427	99
0	3	13	-23	41	99	0	8	2	20	45	99	0	13	1	413	424	99	1	1	-7	607	588	99	1	3	-6	216	212	99
0	4	0	3815	3940	99	0	8	3	61	54	99	0	13	2	-110	117	99	1	1	-6	158	146	99	1	3	-5	777	730	99
0	4	1	497	498	99	0	8	4	48	21	99	0	13	3	-111	9	99	1	1	-5	1031	988	99	1	3	-4	188	178	99
0	4	2	31	28	99	0	8	5	-76	20	99	0	13	4	192	182	99	1	1	-4	101	98	78	1	3	-3	-28	35	99
0	4	3	15	14	99	0	8	6	257	225	99	0	13	5	341	317	99	1	1	-3	103	98	66	1	3	-2	214	214	99
0	4	4	95	82	92	0	8	7	177	170	99	0	13	6	192	171	99	1	1	-2	207	166	99	1	3	-1	68	46	66
0	4	5	23	29	99	0	8	8	208	189	99	0	13	7	-51	63	99	1	1	-1	472	455	99	1	3	0	772	792	99
0	4	6	381	387	99	0	8	9	-30	110	99	0	13	8	70	44	99	1	1	0	755	741	99	1	3	1	371	388	99
0	4	7	120	124	99	0	8	10	377	384	99	0	13	9	296	265	99	1	1	1	341	334	99	1	3	2	176	172	99
0	4	8	262	266	99	0	8	11	118	120	99	0	14	0	498	507	99	1	1	2	64	82	46	1	3	3	1031	1024	99
0	4	9	70	75	99	0	8	12	22	4	99	0	14	1	108	117	99	1	1	3	1264	1259	99	1	3	4	34	32	99
0	4	10	576	575	99	0	9	1	674	691	99	0	14	2	60	50	99	1	1	4	9	2	90	1	3	5	277	284	99
1	3	6	-18	25	99	1	5	8	-54	8	99	1	8	-12	94	87	99	1	10	-3	235	209	99	1	12	9	51	41	99
1	3	7	196	203	99	1	5	9	634	646	99	1	8	-11	-64	23	99	1	10	-2	194	178	99	1	13	-9	62	9	99
1	3	8	178	183	99	1	5	10	118	109	99	1	8	-10	52	63	99	1	10	-1	56	7	99	1	13	-8	10	14	99
1	3	9	450	450	99	1	5	11	59	66	99	1	8	-9	-54	16	99	1	10	0	396	414	99	1	13	-7	272	292	99
1	3	10	313	316	99	1	5	12	52	46	99	1	8	-8	60	53	99	1	10	1	380	377	99	1	13	-6	-44	25	99
1	3	11	58	58	96	1	6	-12	49	55	99	1	8	-7	269	245	99	1	10	2	-53	11	99	1	13	-5	388	395	99
1	3	12	79	89	99	1	6	-11	-33	57	99	1	8	-6	904	866	99	1	10	3	400	392	99	1	13	-4	120	95	99
1	3	13	67	67	99	1	6	-10	79	72	99	1	8	-5	249	250	99	1	10	4	571	552	99	1	13	-3	-53	14	99
1	4	-13	46	47	99	1	6	-9	72	72	99	1	8	-4	29	51	99	1	10	5	155	154	99	1	13	-2	161	176	99
1	4	-12	134	127	99	1	6	-8	36	7	99	1	8	-3	141	126	99	1	10	6	10	73	99	1	13	-1	314	320	99
1	4	-11	18	17	99	1	6	-7	625	572	99	1	8	-2	55	62	99	1	10	7	410	380	99	1	13	0	-116	44	99
1	4	-10	-46	53	99	1	6	-6	492	462	99	1	8	-1	84	84	99	1	10	8	66	65	99	1	13	1	60	62	99
1	4	-9	-28	11	99	1	6	-5	492	461	99	1	8	0	811	824	99	1	10	9	64	51	99	1	13	2	-36	99	99
1	4	-8	69	64	99	1	6	-4	-39	9	99	1	8	1	292	307	99	1	10	10	187	170	99	1	13	3	374	376	99
1	4	-7	187	168	99	1	6	-3	318	297	99	1	8	2	50	57	99	1	10	11	184	187	99	1	13	4	68	0	99
1	4	-6	1380	1312	99	1	6	-2	166	169	99	1	8	3	314	319	99	1	11	-10	-75	8	99	1	13	5	268	263	99
1	4	-5	194	188	99	1	6	-1	-74	15	99	1	8	4	864	850	99	1	11	-9	-65	34	99	1	13	6	87	127	99
1	4	-4	73	64	99	1	6	0	405	432	99	1	8	5	242	227	99	1	11	-8	209	210	99	1	13	7	-87	57	99
1	4	-3	100	102	82	1	6	1	695	743	99	1	8	6	50	7	99	1	11	-7	23	68	99	1	13	8	-77	100	99
1	4	-2	204	202	99	1	6	2	51	8	99	1	8	7	31	35	99	1	11	-6	103	131	99	1	13	9	340	316	99
1	4	-1	59	59	86	1	6	3	781	810	99	1	8	8	166	166	99	1	11	-5	74	84	99	1	14	-8	-80	4	99
1	4	0	1371	1420	99	1	6	4	543	541	99	1	8	9	55	43	99	1	11	-4	146	140	99	1	14	-7	114	144	99
1	4	1	215	211	99	1	6	5	330	332	99	1	8	10	276	270	99	1	11	-3	-48	10	99	1	14	-6	359	367	99
1	4	2	48	17	81	1	6	6	69	69	99	1	8	11	-15	90	99	1	11	-2	82	64	99	1	14	-5	84	75	99
1	4	3	231	243	99	1	6	7	543	563	99	1	9	-11	181	198	99	1	11	-1	292	287	99	1	14	-4	-59	11	99
1	4	4	1401	1365	99	1	6	8	72	56	99	1	9	-10	-3	87	99	1	11	0	337	313	99	1	14	-3	150	151	99
1	4	5	155	170	99	1	6	9	77	70	99	1	9	-9	-74	8	99	1	11	1	171	161	99	1	14	-2	-30	123	99
1	4	6	-31	10	99	1	6	10	145	152	99	1	9	-8	91	79	99	1	11	2	287	273	99	1	14	-1	-93	19	99
1	4	7	33	24	99	1	6	11	308	315	99	1	9	-7	480	454	99	1	11	3	221	215	99	1	14	0	322	303	99
1	4	8	237	252	99	1	6	12	49	23	99	1	9	-6	-54	2	99	1	11	4	69	50	99	1	14	1	164	162	99
1	4	9	65	27	99	1	7	-12	145	161	99	1	9	-5	637	662	99	1	11	5	71	38	99	1	14	2	93	23	99
1	4	10	380	384	99	1	7	-11	-67	7	99	1	9	-4	94	96	99	1	11	6	44	60	99	1	14	3	130	134	99
1	4	11	65	60	99	1	7	-10	42	20	99	1	9	-3	41	39	99	1	11	7	56	93	99	1	14	4	382	404	99
1	4	12	-40	38	99	1	7	-9	36	36	99	1	9	-2	154	151	99	1	11	8	145	131	99	1	14	5	-28	35	99
1	5	-13	20	13	99	1	7	-8	314	297	99	1	9	-1	533	545	99	1	11	9	23	25	99	1	14	6	47	54	99
1	5	-12	-44	39	99	1	7	-7	237	217	99	1	9	0	81	98	99	1	11	10	248	217	99	1	14	7	256	225	99
1	5	-11	262	248	99	1	7	-6	210	195	99	1	9	1	81	93	99	1	12	-10	-22	63	99	1	14	8	62	58	99
1	5	-10	41	68	99	1	7	-5	366	345	99	1	9	2	98	86	99	1	12	-9	-39	16	99	1	15	-7	-84	13	99
1	5	-9	-72	15	99	1	7	-4	177	184	99	1	9	3	668	679	99	1	12	-8	-52	39	99	1	15	-6	-79	71	99
1	5	-8	208	188	99	1	7	-3	42	14	99	1	9	4	44	15	99	1	12	-7	202	214	99	1	15	-5	60	35	99
1	5	-7	609	582	99	1	7	-2	164	156	99	1	9	5	432	425	99	1	12	-6	399	427	99	1	15	-4	112	98	99
1	5	-6	92	62	99	1	7	-1	206	203	99	1	9	6	120	129	99	1	12	-5	177	200	99	1	15	-3	-50	4	99
1	5	-5	1009	914	99	1	7	0	530	562	99	1	9	7	103	104	99	1	12	-4	-49	39	99	1	15	-2	24	16	99
1	5	-4	54	35	99	1	7	1	246	267	99	1	9	8	87	83	99	1	12	-3	44	102	99	1	15	-1	241	256	99
1	5	-3	87	74	99	1	7	2	328	339	99	1	9	9	513	505	99	1	12	-2	-49	32	99	1	15	0	151	141	99
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5	8	6	215	208	99
5	8	7	118	81	99
5	9	-9	121	116	99
5	9	-8	30	40	99
5	9	-7	34	24	99
5	9	-6	84	90	99
5	9	-5	480	464	99
5	9	-4	51	10	99
5	9	-3	55	45	99
5	9	-2	144	143	99
5	9	-1	56	23	99
5	9	0	99	95	99
5	9	1	589	585	99
5	9	2	-70	48	99
5	9	3	-94	12	99
5	9	4	49	48	99
5	9	5	304	295	99
5	9	6	10	9	99
5	9	7	-55	10	99
5	10	-8	-26	51	99
5	10	-7	154	167	99
5	10	-6	79	87	99
5	10	-5	58	115	99
5	10	-4	263	258	99
5	10	-3	45	12	99
5	10	-2	-53	20	99
5	10	-1	133	120	99
5	10	0	241	246	99
5	10	1	164	149	99
5	10	2	13	44	99
5	10	3	147	104	99
5	10	4	-48	27	99
5	10	5	-32	14	99
5	10	6	167	164	99
5	11	-7	-12	0	99
5	11	-6	199	224	99
5	11	-5	78	9	99
5	11	-4	113	108	99
5	11	-3	-53	18	99
5	11	-2	129	113	99
5	11	-1	-68	52	99
5	11	0	-79	78	99
5	11	1	-104	76	99
5	11	2	329	301	99
5	11	3	27	5	99
5	11	4	165	141	99
5	11	5	-69	29	99
5	12	-6	131	126	99
5	12	-5	142	142	99
5	12	-4	254	254	99
5	12	-3	75	141	99
5	12	-2	29	6	99
5	12	-1	-139	27	99
5	12	0	-117	28	99
5	12	1	93	66	99
5	12	2	142	160	99
5	12	3	96	94	99
5	12	4	105	106	99
5	13	-5	284	291	99
5	13	-4	62	21	99
5	13	-3	64	42	99
5	13	-2	72	93	99
5	13	-1	93	16	99
5	13	0	70	97	99
5	13	1	343	358	99
5	13	2	84	30	99
5	13	3	70	10	99
5	14	-3	76	20	99
5	14	-2	-65	9	99
5	14	-1	-126	107	99
5	14	0	198	185	99
6	0	-10	297	332	99
6	0	-8	50	49	99
6	0	-6	329	342	99
6	0	-4	341	338	99
6	0	-2	161	155	99
6	0	0	212	212	99
6	0	2	-88	5	99
6	0	4	540	531	99
6	0	6	208	206	99
6	0	8	106	94	99
6	1	-10	36	17	99
6	1	-9	18	23	99
6	1	-8	159	160	99
6	1	-7	-62	6	99
6	1	-6	125	116	99
6	1	-5	398	406	99
6	1	-4	241	238	99
6	1	-3	13	14	99
6	1	-2	165	160	99
6	1	-1	352	347	99
6	1	0	40	34	99
6	1	1	-30	10	99
6	1	2	-51	43	99
6	1	3	12	29	99
6	1	4	112	108	99
6	1	5	489	490	99
6	1	6	197	202	99
6	1	7	-17	6	99
6	1	8	84	71	99
6	2	-10	70	41	99
6	2	-9	-71	32	99
6	2	-8	38	2	99
6	2	-7	38	14	99
6	2	-6	42	49	99
6	2	-5	105	88	99
6	2	-4	19	32	99
6	2	-3	318	320	99
6	2	-2	54	10	99
6	2	-1	188	185	99
6	2	0	70	68	99
6	2	1	-87	14	99
6	2	2	-35	13	99
6	2	3	-31	41	99
6	2	4	83	53	99
6	2	5	242	236	99
6	2	6	70	22	99
6	2	7	101	106	99
6	2	8	31	3	99
6	3	-10	79	29	99
6	3	-9	-76	31	99
6	3	-8	169	161	99
6	3	-7	-43	6	99
6	3	-6	138	146	99
6	3	-5	294	282	99
6	3	-4	287	286	99
6	3	-3	-16	21	99
6	3	-2	212	212	99
6	3	-1	247	238	99
6	3	0	-13	71	99
6	3	1	36	29	99
6	3	2	-58	47	99
6	3	3	-66	36	99
6	3	4	163	152	99
6	3	5	372	367	99
6	3	6	250	251	99
6	3	7	46	1	99
6	4	-10	313	292	99
6	4	-9	35	41	99
6	4	-8	54	46	99
6	4	-7	-45	40	99
6	4	-6	317	303	99
6	4	-5	17	28	99
6	4	-4	315	303	99
6	4	-3	83	47	99
6	4	-2	145	142	99
6	4	-1	67	60	99
6	4	0	180	173	99
6	4	1	37	59	99
6	4	2	-44	2	99
6	4	3	53	43	99
6	4	4	461	467	99
6	4	5	100	59	99
6	4	6	190	188	99
6	4	7	28	7	99
6	4	8	13	99	
6	5	-9	-12	13	99
6	5	-8	151	136	99
6	5	-7	45	6	99
6	5	-6	30	61	99
6	5	-5	434	420	99
6	5	-4	136	137	99
6	5	-3	30	8	99
6	5	-2	95	71	99
6	5	-1	360	358	99
6	5	0	54	14	99
6	5	1	56	32	99
6	5	2	-18	33	99
6	5	3	38	15	99
6	5	4	-27	37	99
6	5	5	515	497	99
6	5	6	114	113	99
6	5	7	-27	7	99
6	6	-9	31	36	99
6	6	-8	27	6	99
6	6	-7	12	19	99
6	6	-6	121	120	99
6	6	-5	51	62	99
6	6	-4	87	80	99
6	6	-3	249	262	99
6	6	-2	51	29	99
6	6	-1	117	130	99
6	6	0	169	166	99
6	6	1	23	25	99
6	6	2	68	28	99
6	6	3	61	40	99
6	6	4	148	130	99
6	6	5	192	186	99
6	6	6	-14	55	99
6	7	-8	122	135	99
6	7	-7	-32	8	99
6	7	-6	119	132	99
6	7	-5	124	120	99
6	7	-4	233	252	99
6	7	-3	-20	21	99
6	7	-2	196	204	99
6	7	-1	54	97	99
6	7	0	53	76	99
6	7	1	-81	50	99
6	7	2	56	45	99
6	7	3	51	34	99
6	7	4	153	144	99
6	7	5	204	194	99
6	7	6	254	238	99
6	8	-8	56	36	99
6	8	-7	-56	60	99
6	8	-6	197	207	99
6	8	-5	59	42	99
6	8	-4	210	217	99
6	8	-3	50	74	99
6	8	-2	113	113	99
6	8	-1	33	84	99
6	8	0	-65	84	99
6	8	1	41	85	99
6	8	2	-12	6	99
6	8	3	-67	63	99
6	8	4	325	318	99
6	8	5	104	85	99
6	9	-7	-16	8	99
6	9	-6	-80	10	99
6	9	-5	311	333	99
6	9	-4	55	40	99
6	9	-3	66	6	99
6	9	-2	-68	2	99
6	9	-1	267	275	99
6	9	0	68	44	99
6	9	1	17	41	99
6	9	2	64	28	99
6	9	3	-43	10	99
6	9	4	-87	20	99
6	9	5	408	394	99
6	10	-6	138	136	99
6	10	-5	-28	28	99
6	10	-4	103	94	99
6	10	-3	159	174	99
6	10	-2	-98	40	99
6	10	-1	-63	58	99
6	10	0	199	184	99
6	10	1	59	42	99
6	10	2	62	28	99
6	10	3	45	36	99
6	10	4	175	147	99
6	11	-5	-12	4	99
6	11	-4	156	170	99
6	11	-3	-65	14	99
6	11	-2	179	154	99
6	11	-1	70	10	99
6	11	0	53	55	99
6	11	1	56	52	99
6	11	2	-134	33	99
6	11	3	81	28	99
6	12	-3	38	79	99
6	12	-2	66	79	99
6	12	-1	65	69	99
6	12	0	-81	8	99
7	4	-3	73	42	99
7	4	-2	475	466	99
7	4	-1	102	33	99
7	4	0	216	209	99
7	4	1	55	19	99
7	4	2	41	18	99
7	4	3	49	24	99
7	4	4	127	129	99
7	4	5	53	52	99
7	5	-7	187	182	99
7	5	-6	-22	15	99
7	5	-5	-86	12	99
7	5	-4	71	72	99
7	5	-3	-55	36	99
7	5	-2	109	90	99
7	5	-1	537	521	99
7	5	0	37	77	99
7	5	1	-89	5	99
7	5	2	-101	36	99
7	5	3	25	57	99
7	5	4	63	8	99
7	6	-7	-17	95	99
7	6	-6	106	112	99
7	6	-5	58	48	99
7	6	-4	-74	3	99
7	6	-3	69	67	99
7	6	-2	129	116	99
7	6	-1	68	88	99
7	6	0	72	52	99
7	6	1	-72	25	99
7	6	2	59	6	99
7	6	3	42	4	99
7	6	4	132	110	99
7	7	-6	68	30	99
7	7	-5	-99	35	99
7	7	-4	-54	72	99
7	7	-3	-96	24	99
7	7	-2	187	190	99
7	7	-1	196	195	99
7	7	0	190	194	99
7	7	1	16	6	99
7	7	2	-98	12	99
7	7	3	87	36	99
7	8	-5	-37	48	99
7	8	-4	41	15	99
7	8	-3	-35	64	99
7	8	-2	338	336	99
7	8	-1	-102	50	99
7	8	0	132	148	99
7	8	1	47	32	99
7	8	2	64	8	99
7	9	-3	34	34	99
7	9	-2	40	21	99
7	9	-1	406	414	99
7	9	0	-30	1	99
7	9	1	61	1	99
8	0	-4	48	27	99
8	0	-2	214	202	99
8	0	0	57	19	99
8	1	-5	-61	17	99
8	1	-4	56	55	99
8	1	-3	44	34	99
8	1	-2	-51	14	99
8	1	-1	130	133	99
8	1	0	-52	35	99
8	1	1	16	49	99
8	2	-4	25	7	99
8	2	-3	65	16	99
8	2	-2	42	41	99
8	2	-1	166	162	99
8	2	0	51	5	99
8	2	1	125	131	99
8	3	-4	-98	50	99
8	3	-3	59	6	99
8	3	-2	48	15	99
8	3	-1	126	107	99
8	3	0	74	52	99
8	3	1	65	50	99
8	4	-3	52	30	99
8	4	-2	177	177	99
8	4	-1	57	43	99
8	4	0	-74	19	99

APPENDIX 3.II

$[Ni(S_2COnPr)_2]$

Table 3.II.1: Crystallographic Parameters for $[Ni(S_2COCH_2CH_2CH_3)_2]$

Formula	$C_8H_{14}NiO_2S_4$
Formula weight	329.10
Crystal morphology / colour	block / brown
Crystal dimensions (mm)	0.09 x 0.31 x 0.39
Crystal system	triclinic
Space group	$P\bar{1}$ (#2)
a (Å)	9.482(2)
b (Å)	9.972(1)
c (Å)	7.701(1)
α (°)	93.62(1)
β (°)	105.96(1)
γ (°)	73.11(1)
V (Å^3)	669.8(2)
Z	2
$\rho_{calc.}$ (g cm^{-3})	1.631
$F(000)$	340
Temperature (°K)	293
μ (cm^{-1})	20.33
θ limits, cell (°)	24.2 – 25
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 12, -13 – 13, -10 – 10
Range of transmission factors	0.967 – 1
Scan technique	ω:2θ
No. of data measured	3270
No. of unique data	3084
R_{amal}	0.023
No. of parameters	136
R (I≥0σ(I))	0.057
R_w (I≥0σ(I))	0.071
R (I≥3σ(I))	0.037
R_w (I≥3σ(I))	0.034
Maximum and minimum residual electron density (e Å^{-3})	0.54, -0.48

Table 3.II.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Ni(S_2COCH_2CH_2CH_3)_2]$

Atom	*x*	*y*	*z*
Ni	0.19422(4)	0.45878(4)	0.20818(6)
S(1)	0.11540(9)	0.67941(8)	0.2838(1)
S(2)	-0.04000(8)	0.47180(7)	0.2238(1)
S(3)	0.43395(8)	0.44479(8)	0.2192(1)
S(4)	0.28322(8)	0.23321(7)	0.1545(1)
O(1)	-0.1573(2)	0.7334(2)	0.3264(3)
O(2)	0.5729(2)	0.1752(2)	0.1751(3)
C(1)	-0.0454(3)	0.6383(3)	0.2835(4)
C(2)	-0.2998(3)	0.7005(3)	0.3187(4)
C(3)	-0.4168(3)	0.8372(3)	0.3263(4)
C(4)	-0.5684(3)	0.8158(3)	0.3238(5)
C(5)	0.4488(3)	0.2738(3)	0.1830(4)
C(6)	0.7181(3)	0.2102(3)	0.2073(4)
C(7)	0.8395(3)	0.0754(3)	0.2110(5)
C(8)	0.9920(4)	0.0977(3)	0.2324(6)

Table 3.II.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Ni(S_2COCH_2CH_2CH_3)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	0.0436(2)	0.0443(2)	0.0469(3)	-0.0105(2)	0.0123(2)	0.0025(2)
S(1)	0.0500(4)	0.0491(4)	0.0751(7)	-0.0184(4)	0.0206(4)	-0.0084(4)
S(2)	0.0489(4)	0.0438(4)	0.0568(6)	-0.0139(3)	0.0171(4)	-0.0011(4)
S(3)	0.0477(4)	0.0464(4)	0.0737(6)	-0.0140(3)	0.0179(4)	-0.0012(4)
S(4)	0.0469(4)	0.0467(4)	0.0590(6)	-0.0143(3)	0.0130(4)	0.0009(4)
O(1)	0.048(1)	0.047(1)	0.053(1)	-0.0128(9)	0.017(1)	-0.0059(9)
O(2)	0.045(1)	0.049(1)	0.069(2)	-0.0091(9)	0.015(1)	-0.003(1)
C(1)	0.045(2)	0.045(2)	0.034(2)	-0.011(1)	0.008(1)	0.003(1)
C(2)	0.046(2)	0.050(2)	0.046(2)	-0.014(1)	0.014(1)	0.002(1)
C(3)	0.051(2)	0.051(2)	0.064(2)	-0.009(1)	0.019(2)	-0.004(2)
C(4)	0.052(2)	0.068(2)	0.070(3)	-0.007(2)	0.020(2)	-0.003(2)
C(5)	0.045(2)	0.048(2)	0.032(2)	-0.009(1)	0.007(1)	0.003(1)
C(6)	0.043(2)	0.055(2)	0.063(2)	-0.015(1)	0.010(2)	-0.003(2)
C(7)	0.052(2)	0.053(2)	0.108(3)	-0.009(2)	0.021(2)	-0.002(2)
C(8)	0.054(2)	0.060(2)	0.125(4)	-0.007(2)	0.027(2)	-0.007(2)

Table 3.II.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Ni(S_2COCH_2CH_2CH_3)_2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	-0.2852	0.6408	0.4206	4.4111
H(2b)	-0.3322	0.6534	0.2071	4.4111
H(3a)	-0.3807	0.8843	0.4365	5.2642
H(3b)	-0.4307	0.8950	0.2228	5.2642
H(4a)	-0.6417	0.9059	0.3292	6.0992
H(4b)	-0.6056	0.7694	0.2135	6.0992
H(4c)	-0.5554	0.7582	0.4272	6.0992
H(6a)	0.7131	0.2710	0.1109	5.1547
H(6b)	0.7394	0.2568	0.3219	5.1547
H(7a)	0.8124	0.0274	0.0987	6.8370
H(7b)	0.8457	0.0179	0.3114	6.8370
H(8a)	1.0207	0.1448	0.3451	7.6446
H(8b)	1.0675	0.0079	0.2335	7.6446
H(8c)	0.9869	0.1551	0.1325	7.6446

* H atoms were included in their calculated positions, C-H 0.97Å.

Table 3.II.5: Bond distances (Å) for [Ni($S_2COCH_2CH_2CH_3$)2]

Ni — S(1)	2.2048(9)	O(1) — C(1)	1.306(3)
Ni — S(2)	2.2230(9)	O(1) — C(2)	1.465(3)
Ni — S(3)	2.2147(9)	O(2) — C(5)	1.309(3)
Ni — S(4)	2.2167(8)	O(2) — C(6)	1.469(3)
S(1) — C(1)	1.689(3)	C(2) — C(3)	1.497(4)
S(2) — C(1)	1.684(3)	C(3) — C(4)	1.509(4)
S(3) — C(5)	1.682(3)	C(6) — C(7)	1.490(4)
S(4) — C(5)	1.687(3)	C(7) — C(8)	1.489(4)

Table 3.II.6: Bond angles (deg.) for [Ni($S_2COCH_2CH_2CH_3$)2]

S(1) — Ni — S(2)	79.10(3)	C(5) — O(2) — C(6)	119.5(2)
S(1) — Ni — S(3)	99.74(3)	S(1) — C(1) — S(2)	113.4(2)
S(1) — Ni — S(4)	175.55(4)	S(2) — C(1) — O(1)	127.2(2)
S(2) — Ni — S(3)	174.90(4)	S(1) — C(1) — O(1)	119.4(2)
S(2) — Ni — S(4)	101.63(3)	O(1) — C(2) — C(3)	106.7(2)
S(3) — Ni — S(4)	79.15(3)	C(2) — C(3) — C(4)	111.4(2)
Ni — S(1) — C(1)	83.97(9)	S(3) — C(5) — S(4)	113.9(1)
Ni — S(2) — C(1)	83.51(9)	S(3) — C(5) — O(2)	126.3(2)
Ni — S(3) — C(5)	83.52(9)	S(4) — C(5) — O(2)	119.8(2)
Ni — S(4) — C(5)	83.35(9)	O(2) — C(6) — C(7)	106.8(2)
C(1) — O(1) — C(2)	119.7(2)	C(6) — C(7) — C(8)	112.0(3)

Table 3.II.7: Observed and calculated structure factors for $[Ni(S_2COCH_2CH_2CH_3)_2]$

h	k	l	Fo	Fc	sigF
0	0	2	1541	1816	9
0	0	3	431	455	3
0	0	4	302	313	2
0	0	5	256	264	2
0	0	6	78	73	3
0	0	7	101	99	3
0	0	8	66	69	4
0	0	9	40	34	7
0	1	-9	49	34	6
0	1	-8	21	13	11
0	1	-7	138	137	3
0	1	-6	219	219	2
0	1	-5	296	309	2
0	1	-4	868	878	5
0	1	-3	275	284	2
0	1	-2	1483	1614	8
0	1	-1	60	57	1
0	1	1	319	322	2
0	1	2	196	206	2
0	1	3	269	283	2
0	1	4	191	194	2
0	1	5	186	190	2
0	1	6	70	74	3
0	1	7	120	121	3
0	1	8	18	34	12
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10	158	0	0	8	12
10	158	1	0	8	12
10	159	-1	0	8	12
10	159	0	0	8	12
10	159	1	0

APPENDIX 3.III

$[Ni(S_2COiPr)_2]$

Table 3.III.1: Crystallographic Parameters for $[Ni(S_2COCH(CH_3)_2)_2]$

Formula	$C_8H_{14}NiO_2S_4$
Formula weight	329.10
Crystal morphology / colour	block / brown
Crystal dimensions (mm)	0.13 x 0.23 x 0.32
Crystal system	monoclinic
Space group	$P2_1/c$ (#14)
a (Å)	8.869(2)
b (Å)	6.084(1)
c (Å)	13.2029(9)
α (°)	90
β (°)	94.671(9)
γ (°)	90
V (Å^3)	710.1(2)
Z	2
$\rho_{calc.}$ (g cm^{-3})	1.539
$F(000)$	340
Temperature (°K)	293
μ (cm^{-1})	19.18
θ limits, cell (°)	21.3 – 25.0
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 11, 0 – 7, -16 – 16.
Range of transmission factors	0.962 – 1
Scan technique	ω:2θ
No. of data measured	1906
No. of unique data	1796
R_{amal}	0.072
No. of parameters	70
R (I≥0σ(I))	0.056
R_w (I≥0σ(I))	0.077
R (I≥3σ(I))	0.044
R_w (I≥3σ(I))	0.034
Maximum and minimum residual electron density (e Å^{-3})	0.58, -0.65

Table 3.III.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Ni(S_2COCH(CH_3)_2)_2]$

Atom	*x*	*y*	*z*
Ni	0	0	0
S(1)	-0.1927(2)	-0.1637(2)	-0.08437(9)
S(2)	-0.0316(1)	0.2113(2)	-0.13700(9)
O(1)	-0.2717(3)	0.0468(5)	-0.2499(2)
C(1)	-0.1767(5)	0.0398(7)	-0.1714(3)
C(2)	-0.2624(5)	0.2273(8)	-0.3221(3)
C(3)	-0.3488(6)	0.4202(8)	-0.2895(4)
C(4)	-0.3266(6)	0.1311(9)	-0.4187(3)

Table 3.III.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Ni(S_2COCH(CH_3)_2)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	0.0531(6)	0.0551(6)	0.0537(5)	-0.0086(5)	0.0059(4)	-0.0003(5)
S(1)	0.076(1)	0.0691(9)	0.0666(8)	-0.0263(8)	-0.0043(7)	0.0099(7)
S(2)	0.0578(8)	0.0651(9)	0.0633(8)	-0.0174(7)	0.0004(6)	0.0086(7)
O(1)	0.057(2)	0.064(2)	0.052(2)	-0.013(2)	0.002(2)	0.005(2)
C(1)	0.052(3)	0.054(3)	0.048(2)	-0.007(3)	0.015(2)	-0.002(2)
C(2)	0.054(3)	0.068(3)	0.046(2)	-0.007(3)	0.008(2)	0.001(3)
C(3)	0.087(4)	0.070(4)	0.082(4)	0.017(3)	-0.002(3)	-0.012(3)
C(4)	0.107(5)	0.089(4)	0.068(3)	0.006(4)	0.014(3)	0.004(3)

Table 3.III.4: Fractional atomic coordinates and B_{eq} values (Å^2) for the hydrogen atoms in [Ni($S_2CO(CH(CH_3)_2)_2$]*

Atom	*x*	*y*	*z*	B(eq)
H(2a)	-0.1570	0.2662	-0.3310	5.2955
H(3a)	-0.3055	0.4668	-0.2274	7.6586
H(3b)	-0.3431	0.5346	-0.3399	7.6586
H(3c)	-0.4507	0.3784	-0.2872	7.6586
H(4a)	-0.4303	0.0924	-0.4181	8.4157
H(4b)	-0.3208	0.2342	-0.4767	8.4157
H(4c)	-0.2723	0.0014	-0.4388	8.4157

* H atoms were included in their calculated positions, C-H 0.97Å.

Table 3.III.5: Bond distances (Å) for [Ni($S_2COCH(CH_3)_2$)$_2$]

Ni — S(1)	2.201(1)	O(1) — C(1)	1.282(4)
Ni — S(2)	2.218(1)	O(1) — C(2)	1.462(5)
S(1) — C(1)	1.703(4)	C(2) — C(3)	1.485(6)
S(2) — C(1)	1.689(4)	C(2) — C(4)	1.474(6)

Table 3.III.6: Bond Angles (deg.) for [Ni($S_2COCH(CH_3)_2)_2$]

S(1) — Ni — S(1)*	180(-)	S(1) — C(1) — S(2)	111.9(2)
S(1) — Ni — S(2)	78.96(4)	S(1) — C(1) — O(1)	118.9(3)
S(1) — Ni — S(2)*	101.04(4)	S(2) — C(1) — O(1)	129.1(3)
S(2) — Ni — S(2)*	180(-)	O(1) — C(2) — C(3)	110.2(3)
Ni — S(1) — C(1)	84.7(1)	O(1) — C(2) — C(4)	103.3(4)
Ni — S(2) — C(1)	84.4(1)	C(3) — C(2) — C(4)	113.1(4)
C(1) — O(1) — C(2)	119.0(3)		

* atom related by a centre of symmetry

Table 3.III.7: Observed and calculated structure factors for [Ni($S_2COCH(CH_3)_2)_2$]

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	2	12	82	12	0	3	15	25	16	66	1	0	-4	166	165	52	1	2	-5	517	513	99	1	4	-12	37	27	83
0	0	4	67	66	34	0	4	0	282	287	99	1	0	-2	821	821	99	1	2	-4	372	372	99	1	4	-11	74	70	90
0	0	6	100	99	46	0	4	1	-20	9	49	1	0	2	335	334	97	1	2	-3	20	33	30	1	4	-10	124	119	99
0	0	8	258	273	99	0	4	2	78	77	59	1	0	4	443	447	99	1	2	-2	386	384	99	1	4	-9	108	117	99
0	0	10	97	119	75	0	4	3	29	28	48	1	0	6	652	648	99	1	2	-1	490	471	99	1	4	-8	209	209	99
0	0	12	264	257	99	0	4	4	138	148	77	1	0	8	495	474	99	1	2	0	50	49	32	1	4	-7	116	115	85
0	0	14	140	111	99	0	4	5	207	204	99	1	0	10	147	148	84	1	2	1	204	207	64	1	4	-6	176	162	99
0	0	16	64	59	89	0	4	6	312	325	99	1	0	12	110	112	81	1	2	2	496	502	99	1	4	-5	167	157	98
0	1	1	838	816	99	0	4	7	82	76	75	1	0	14	75	75	83	1	2	3	217	210	74	1	4	-4	51	50	64
0	1	2	190	199	50	0	4	8	229	215	99	1	0	16	36	25	75	1	2	4	833	824	99	1	4	-3	41	39	54
0	1	3	81	83	34	0	4	9	115	104	80	1	1	-17	-11	50	99	1	2	5	109	121	51	1	4	-2	142	140	78
0	1	4	149	346	99	0	4	10	49	33	67	1	1	-16	8	3	96	1	2	6	213	198	90	1	4	-1	222	211	99
0	1	5	405	417	99	0	4	11	39	50	72	1	1	-15	73	56	99	1	2	7	233	228	99	1	4	0	358	350	99
0	1	6	362	347	99	0	4	12	20	28	73	1	1	-14	41	11	87	1	2	8	65	57	60	1	4	1	101	96	64
0	1	7	274	263	99	0	4	13	81	86	82	1	1	-13	200	205	99	1	2	9	147	132	88	1	4	2	217	217	99
0	1	8	294	313	99	0	4	14	-12	13	71	1	1	-12	-6	40	84	1	2	10	228	243	99	1	4	3	85	86	61
0	1	9	188	187	94	0	5	1	268	273	99	1	1	-11	34	24	70	1	2	11	65	81	71	1	4	4	153	156	83
0	1	10	271	261	99	0	5	2	148	157	90	1	1	-10	224	212	99	1	2	12	119	112	87	1	4	5	86	88	67
0	1	11	164	165	99	0	5	3	57	54	62	1	1	-9	143	137	85	1	2	13	104	97	84	1	4	6	224	219	99
0	1	12	77	104	78	0	5	4	99	101	70	1	1	-8	101	106	62	1	2	14	26	16	68	1	4	7	69	80	71
0	1	13	125	128	99	0	5	5	-25	10	60	1	1	-7	199	200	86	1	2	15	34	1	71	1	4	8	154	143	97
0	1	14	88	84	89	0	5	6	36	46	63	1	1	-6	492	485	99	1	2	16	58	65	72	1	4	9	131	121	88
0	1	15	66	56	82	0	5	7	143	167	96	1	1	-5	423	413	99	1	3	-15	23	27	87	1	4	10	31	16	67
0	1	16	-33	13	76	0	5	8	39	43	68	1	1	-4	304	325	99	1	3	-14	36	39	96	1	4	11	70	82	79
0	2	0	114	114	41	0	5	9	85	78	77	1	1	-3	585	566	99	1	3	-13	30	32	90	1	4	12	29	40	74
0	2	1	119	117	42	0	5	10	-16	32	67	1	1	-2	837	846	99	1	3	-12	21	18	82	1	4	13	67	63	76
0	2	2	131	135	47	0	5	11	-9	34	68	1	1	-1	1161	1192	99	1	3	-11	159	140	99	1	4	14	55	61	76
0	2	3	134	134	48	0	5	12	27	11	65	1	1	0	265	260	72	1	3	-10	86	66	86	1	5	-13	53	62	91
0	2	4	851	869	99	0	5	13	58	53	64	1	1	1	283	278	79	1	3	-9	290	288	99	1	5	-12	49	52	89
0	2	5	200	208	77	0	6	0	118	128	85	1	1	2	59	38	29	1	3	-8	101	118	84	1	5	-11	22	19	80
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8	0	6	200	201	99	8	3	-7	54	21	99	9	0	8	28	21	53	9	3	5	19	25	70	10	2	6	40	39	59
8	0	8	49	51	63	8	3	-6	58	36	99	9	1	-11	42	31	99	9	3	6	47	43	68	10	3	-6	21	31	99
8	0	10	-16	27	54	8	3	-5	92	95	99	9	1	-10	-44	6	99	9	3	7	42	48	62	10	3	-5	74	86	99
8	1	-13	69	39	99	8	3	-4	88	71	99	9	1	-9	90	107	99	9	4	-7	16	20	99	10	3	-4	49	40	99
8	1	-12	65	3	99	8	3	-3	81	82	99	9	1	-8	-24	27	99	9	4	-6	26	22	99	10	3	-3	48	54	99
8	1	-11	21	13	99	8	3	-2	-26	12	90	9	1	-7	28	52	99	9	4	-5	39	58	99	10	3	-2	11	2	98
8	1	-10	-19	26	99	8	3	-1	111	109	99	9	1	-6	-50	8	99	9	4	-4	94	95	99	10	3	-1	20	16	89
8	1	-9	123	133	99	8	3	0	-38	23	87	9	1	-5	64	54	99	9	4	-3	52	46	99	10	3	0	49	42	96
8	1	-8	94	104	99	8	3	1	141	131	99	9	1	-4	88	83	99	9	4	-2	79	90	99	10	3	1	57	53	83
8	1	-7	107	102	99	8	3	2	39	41	77	9	1	-3	68	71	99	9	4	-1	-25	8	92	10	3	2	35	39	81
8	1	-6	34	56	99	8	3	3	110	112	94	9	1	-2	8	11	93	9	4	0	15	14	85	10	3	3	34	38	78
8	1	-5	86	92	99	8	3	4	66	59	73	9	1	-1	85	80	97	9	4	1	71	74	94	10	3	4	20	15	63
8	1	-4	178	180	99	8	3	5	48	31	66	9	1	0	145	148	99	9	4	2	4	16	78	11	0	-6	-20	11	99
8	1	-3	77	80	99	8	3	6	49	51	70	9	1	1	86	84	88	9	4	3	36	41	79	11	0	-4	52	60	99
8	1	-2	40	61	92	8	3	7	25	10	62	9	1	2	-37	5	75	9	4	4	54	49	74	11	0	-2	56	80	99
8	1	-1	157	157	99	8	3	8	19	5	64	9	1	3	105	104	93	9	4	5	44	32	64	11	0	0	68	79	81
8	1	0	148	148	99	8	3	9	75	69	66	9	1	4	87	79	79	10	0	-8	51	54	99	11	0	2	27	27	68
8	1	1	12	14	71	8	4	-9	64	28	99	9	1	5	71	75	78	10	0	-6	-21	20	99	11	1	-6	-14	9	99
8	1	2	28	17	69	8	4	-8	65	29	99	9	1	6	-6	12	62	10	0	-4	-14	38	99	11	1	-5	4	42	99
8	1	3	172	168	99	8	4	-7	21	19	99	9	1	7	38	45	64	10	0	-2	126	130	99	11	1	-4	-30	18	99
8	1	4	15	3	68	8	4	-6	29	12	99	9	1	8	-28	13	59	10	0	0	49	50	80	11	1	-3	40	45	97
8	1	5	103	109	87	8	4	-5	-24	8	99	9	1	9	20	12	49	10	0	2	56	63	75	11	1	-2	49	38	92
8	1	6	21	29	64	8	4	-4	83	94	99	9	2	-10	31	24	99	10	0	4	58	76	75	11	1	-1	63	64	97
8	1	7	100	92	72	8	4	-3	103	105	99	9	2	-9	41	20	99	10	0	6	34	37	58	11	1	0	-11	13	82
8	1	8	-32	0	61	8	4	-2	140	131	99	9	2	-8	-33	18	99	10	1	-9	49	37	99	11	1	1	42	40	76
8	1	9	15	27	62	8	4	-1	42	30	85	9	2	-7	12	1	99	10	1	-8	27	23	99	11	1	2	45	44	76
8	1	10	48	48	58	8	4	0	75	76	97	9	2	-6	114	112	99	10	1	-7	36	36	99	11	1	3	-3	13	79
8	1	11	30	21	50	8	4	1	58	60	90	9	2	-5	-37	30	99	10	1	-6	11	29	99	11	2	-4	38	44	99
8	2	-12	53	66	99	8	4	2	31	17	78	9	2	-4	74	63	99	10	1	-5	-34	26	99	11	2	-3	-32	6	96
8	2	-11	-35	6	99	8	4	3	21	1	71	9	2	-3	13	39	99	10	1	-4	-18	24	99	11	2	-2	-35	14	89
8	2	-10	-51	12	99	8	4	4	61	47	75	9	2	-2	60	56	95	10	1	-3	79	67	99	11	2	-1	-28	3	86
8	2	-9	10	9	99	8	4	5	39	30	66	9	2	-1	85	92	99	10	1	-2	61	57	98	11	2	0	61	59	88
8	2	-8	92	91	99	8	4	6	43	44	70	9	2	0	91	100	99	10	1	-1	38	58	96	11	2	1	-15	5	77
8	2	-7	57	58	99	8	4	7	11	20	58	9	2	1	51	52	84	10	1	0	46	56	92						
8	2	-6	84	90	99	8	5	-6	22	13	99	9	2	2	129	127	99	10	1	1	102	97	94						
8	2	-5	49	50	99	8	5	-5	32	14	98	9	2	3	16	4	73	10	1	2	19	16	76						
8	2	-4	76	76	99	8	5	-4	-27	15	94	9	2	4	-11	7	71	10	1	3	36	29	74						
8	2	-3	79	80	99	8	5	-3	92	97	99	9	2	5	48	51	76	10	1	4	71	77	84						
8	2	-2	69	64	93	8	5	-2	44	49	92	9	2	6	-22	15	64	10	1	5	35	43	79						
8	2	-1	7	33	84	8	5	-1	69	71	92	9	2	7	-9	4	62	10	1	6	24	21	68						
8	2	0	121	134	99	8	5	0	49	30	81	9	2	8	62	60	66	10	1	7	19	25	56						
8	2	1	159	157	94	8	5	1	17	17	81	9	3	-9	31	18	99	10	1	-8	37	20	99						
8	2	2	185	189	99	8	5	2	67	72	85	9	3	-8	22	22	99	10	1	-7	27	14	99						
8	2	3	30	34	77	8	5	3	-12	6	71	9	3	-7	23	5	99	10	1	-6	30	35	99						
8	2	4	93	91	86	8	5	4	13	2	65	9	3	-6	29	7	99	10	1	-5	80	3	99						
8	2	5	-7	19	70	8	5	5	73	71	73	9	3	-5	127	115	99	10	1	-4	76	75	99						
8	2	6	63	62	89	9	1	-11	125	136	99	9	3	-4	66	61	99	10	1	-3	24	22	99						
8	2	7	28	4	68	9	1	-6	94	93	99	9	3	-3	39	35	99	10	1	-2	-28	5	96						
8	2	8	92	88	74	9	1	-5	11	16	99	9	3	-2	42	33	99	10	1	-1	13	38	98						

APPENDIX 3.IV

$[Ni(S_2COnBu)_2]$

Table 3.IV.1: Crystallographic Parameters for [Ni($S_2COCH_2CH_2CH_2CH_3$)2]

Formula	$C_{10}H_{18}NiO_2S_4$
Formula weight	357.19
Crystal morphology / colour	plate / brown
Crystal dimensions (mm)	0.08 x 0.25 x 0.40
Crystal system	triclinic
Space group	$P\bar{1}$(#2)
a (Å)	6.410(2)
b (Å)	11.073(2)
c (Å)	5.972(1)
α (°)	104.55(2)
β (°)	100.46(2)
γ (°)	76.67(2)
V (Å^3)	395.7(1)
Z	1
$\rho_{calc.}$ (g cm^{-3})	1.499
F(000)	186
Temperature (°K)	293
μ (cm^{-1})	17.41
θ limits, cell (°)	10.0 – 12.0
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 8, -14 – 14, -7 – 7.
Range of transmission factors	0.961 – 1
Scan technique	ω:2θ
No. of data measured	1999
No. of unique data	1835
R_{amal}	0.023
No. of parameters	79
R (I≥0σ(I))	0.057
R_w (I≥0σ(I))	0.070
R (I≥3σ(I))	0.038
R_w (I≥3σ(I))	0.031
Maximum and minimum residual electron density (e Å^{-3})	0.61, -0.99

Table 3.IV.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Ni(S_2COCH_2CH_2CH_2CH_3)_2]$

Atom	*x*	*y*	*z*
Ni	0	0	0
S(1)	0.1041(2)	0.17750(10)	0.1949(2)
S(2)	0.2205(2)	0.01764(9)	-0.2305(2)
O(1)	0.3691(4)	0.2322(2)	-0.0286(4)
C(1)	0.2503(5)	0.1513(3)	-0.0267(6)
C(2)	0.4949(6)	0.2086(3)	-0.2230(6)
C(3)	0.5823(6)	0.3264(3)	-0.1952(6)
C(4)	0.7192(6)	0.3150(3)	-0.3816(6)
C(5)	0.8206(8)	0.4301(4)	-0.3455(9)

Table 3.IV.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Ni(S_2COCH_2CH_2CH_2CH_3)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	0.0555(5)	0.0619(5)	0.0570(4)	-0.0100(4)	0.0029(3)	0.0162(3)
S(1)	0.0810(7)	0.0758(7)	0.0654(6)	-0.0231(6)	0.0184(5)	0.0037(5)
S(2)	0.0720(7)	0.0641(6)	0.0615(6)	-0.0181(5)	0.0119(5)	0.0076(5)
O(1)	0.076(2)	0.063(2)	0.067(2)	-0.021(1)	0.011(1)	0.014(1)
C(1)	0.057(2)	0.059(2)	0.063(2)	-0.005(2)	0.002(2)	0.019(2)
C(2)	0.075(3)	0.067(2)	0.065(2)	-0.017(2)	0.012(2)	0.017(2)
C(3)	0.077(3)	0.056(2)	0.084(3)	-0.012(2)	0.019(2)	0.011(2)
C(4)	0.090(3)	0.065(3)	0.087(3)	-0.017(2)	0.027(2)	0.013(2)
C(5)	0.156(5)	0.083(3)	0.154(4)	-0.051(3)	0.074(4)	0.001(3)

Table 3.IV.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in [Ni($S_2COCH_2CH_2CH_2CH_3)_2$]*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.6101	0.1375	-0.2160	6.4
H(2b)	0.4046	0.1930	-0.3679	6.4
H(3a)	0.6680	0.3415	-0.0473	6.8
H(3b)	0.4645	0.3960	-0.2030	6.8
H(4a)	0.8312	0.2419	-0.3807	7.5
H(4b)	0.6309	0.3056	-0.5286	7.5
H(5a)	0.9095	0.4405	-0.1990	11.7
H(5b)	0.9060	0.4184	-0.4665	11.7
H(5c)	0.7099	0.5038	-0.3484	11.7

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 3.IV.5: Bond distances (Å) for [Ni($S_2COCH_2CH_2CH_2CH_3)_2$]

Ni(1) — S(1)	2.1998(9)	O(1) — C(2)	1.468(4)
Ni(1) — S(2)	2.2136(9)	C(3) — C(4)	1.503(4)
S(1) — C(1)	1.687(3)	C(2) — C(3)	1.496(4)
S(2) — C(1)	1.688(3)	C(4) — C(5)	1.513(5)
O(1) — C(1)	1.307(3)		

Table 3.IV.6: Bond angles (deg.) for $[Ni(S_2COCH_2CH_2CH_2CH_3)_2]$

S(1) — Ni(1) — S(1)*	180(-)	C(1) — O(1) — C(2)	119.2(3)
S(1) — Ni(1) — S(2)	79.48(3)	C(2) — C(3) — C(4)	112.2(3)
S(1) — Ni(1) — S(2)*	100.52(3)	C(3) — C(4) — C(5)	112.5(3)
S(2) — Ni(1) — S(2)*	180(-)	S(1) — C(1) — S(2)	113.5(2)
Ni(1) — S(1) — C(1)	83.3(1)	S(1) — C(1) — O(1)	126.8(3)
Ni(1) — S(2) — C(1)	83.8(1)	S(2) — C(1) — O(1)	119.7(3)
O(1) — C(2) — C(3)	106.2(3)		

* atom related by a centre of symmetry

Table 3.IV.7: Observed and calculated structure factors for $[Ni(S_2COCH_2CH_2CH_2CH_3)_2]$

h	k	l	Fo	Fc	sigF
0	0	1	512	515	4
0	0	2	123	123	2
0	0	3	64	63	2
0	0	4	243	244	2
0	0	5	210	208	3
0	0	6	27	34	7
0	0	7	8	11	9
0	1	-7	0	10	10
0	1	-6	8	10	10
0	1	-5	95	90	3
0	1	-4	150	147	2
0	1	-3	11	19	7
0	1	-2	87	87	2
0	1	-1	323	318	3
0	1	1	571	601	4
0	1	2	81	80	2
0	1	3	122	124	2
0	1	4	283	283	3
0	1	5	152	152	2
0	1	6	28	22	6
0	1	7	0	7	8
0	2	-7	31	28	6
0	2	-6	0	14	10
0	2	-5	29	16	6
0	2	-4	49	50	4
0	2	-3	113	115	2
0	2	-2	57	61	2
0	2	-1	305	300	2
0	2	0	36	28	2
0	2	1	163	161	2
0	2	2	27	23	3
0	2	3	139	141	2
0	2	4	189	187	2
0	2	5	60	58	3
0	2	6	25	17	6
0	2	7	20	4	6
0	3	-7	24	22	8
0	3	-6	14	3	10
0	3	-5	46	48	5
0	3	-4	148	150	2
0	3	-3	212	212	2
0	3	-2	105	107	2
0	3	-1	45	41	2
0	3	0	174	170	2
0	3	1	136	134	2
0	3	2	70	78	2
0	3	3	75	73	2
0	3	4	64	62	3
0	3	5	23	23	6
0	3	6	16	7	8
0	4	-7	13	10	10
0	4	-6	22	17	10
0	4	-5	97	100	3
0	4	-4	293	289	3
0	4	-3	240	237	2
0	4	-2	14	12	7
0	4	-1	18	16	5
0	4	0	353	344	3
0	4	1	387	384	3
0	4	2	147	148	2
0	4	3	20	18	7
0	4	4	33	27	4
0	4	5	47	46	3
0	4	6	25	25	5
0	5	-7	0	4	10
0	5	-6	22	5	10
0	5	-5	103	101	3
0	5	-4	236	236	3
0	5	-3	125	125	2
0	5	-2	77	77	2
0	5	-1	113	111	2
0	5	0	455	446	4
0	5	1	384	378	3
0	5	2	0	6	8
0	5	3	44	47	4
0	5	4	78	83	3
0	5	5	86	89	3
0	5	6	26	28	5
0	6	-7	0	14	11
0	6	-6	19	23	11
0	6	-5	85	83	3
0	6	-4	133	129	2
0	6	-3	57	55	3
0	6	-2	72	75	3
0	6	-1	137	141	2
0	6	0	358	357	3
0	6	1	192	183	2
0	6	2	43	41	4
0	6	3	57	63	3
0	6	4	74	79	3
0	6	5	89	95	3
0	6	6	10	14	7
0	7	-7	46	33	5
0	7	-6	0	22	12
0	7	-5	42	40	5
0	7	-4	78	84	3
0	7	-3	102	110	3
0	7	-2	102	102	2
0	7	-1	65	61	3
0	7	0	177	175	2
0	7	1	94	96	3
0	7	2	0	14	10
0	7	3	29	32	6
0	7	4	52	57	4
0	7	5	36	40	4
0	8	-7	17	29	11
0	8	-6	43	17	5
0	8	-5	31	8	6
0	8	-4	73	81	4
0	8	-3	124	132	3
0	8	-2	136	143	3
0	8	-1	104	112	3
0	8	0	38	36	5
0	8	1	93	93	3
0	8	2	55	64	4
0	8	3	56	50	4
0	8	4	52	54	4
0	8	5	13	14	7
0	9	-6	24	5	10
0	9	-5	0	14	11
0	9	-4	67	78	4
0	9	-3	95	110	3
0	9	-2	23	5	9
0	9	-1	15	11	10
0	9	0	105	102	3
0	9	1	97	96	3
0	9	2	61	68	5
0	9	3	9	12	10
0	9	4	20	14	7
0	10	-6	21	17	10
0	10	-5	25	17	10
0	10	-4	66	77	5
0	10	-3	58	61	5
0	10	-2	43	33	5
0	10	-1	81	80	3
0	10	0	62	66	5
0	10	1	124	121	3
0	10	2	35	21	6
0	10	3	25	25	8
0	10	4	0	7	9
0	11	-5	15	11	11
0	11	-4	46	55	6
0	11	-3	33	41	8
0	11	-2	19	9	11
0	11	-1	15	5	11
0	11	0	31	37	8
0	11	1	54	60	5
0	11	2	18	3	10
0	11	3	16	27	10
0	12	-5	0	21	11
0	12	-4	22	30	11
0	12	-3	39	43	7
0	12	-2	15	25	12
0	12	-1	28	33	9
0	12	0	55	52	5
0	12	1	37	31	6
0	12	2	22	1	9
0	13	-4	26	23	9
0	13	-3	30	39	9
0	13	-2	33	36	8
0	13	-1	0	19	11
0	13	0	15	25	12
1	-13	0	15	25	12
1	-13	1	27	26	12
1	-13	2	46	10	6
1	-13	3	22	9	12
1	-12	-2	21	19	11
1	-12	-1	0	17	12
1	-12	0	43	50	9
1	-12	1	0	10	13
1	-12	2	34	19	9
1	-12	3	27	3	12
1	-12	4	30	27	10
1	-11	-3	0	4	10
1	-11	-2	19	3	11
1	-11	-1	42	33	7
1	-11	0	88	81	4
1	-11	1	10	37	13
1	-11	2	15	35	13
1	-11	3	0	3	12
1	-11	4	29	39	12
1	-11	5	36	31	8
1	-10	-3	38	27	6
1	-10	-2	32	27	8
1	-10	-1	57	54	5
1	-10	0	47	48	6
1	-10	1	60	59	5
1	-10	2	34	37	9
1	-10	3	20	35	13
1	-10	4	52	56	6
1	-10	5	25	28	12
1	-10	6	34	7	8
1	-9	-4	23	23	7
1	-9	-3	68	59	4
1	-9	-2	26	33	10
1	-9	-1	31	33	8
1	-9	0	73	67	4
1	-9	1	34	40	8
1	-9	2	69	72	4
1	-9	3	71	77	5
1	-9	4	57	62	6
1	-9	5	39	35	8
1	-9	6	39	9	7
1	-8	-5	15	20	8
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5	-3	-5	49	47	3	5	1	3	7	18	11	5	6	1	103	102	3	5	13	-2	14	2	9	6	0	-3	87	87	3
5	-3	-4	62	67	3	5	1	4	0	21	11	5	6	2	71	72	3	5	13	-1	0	4	8	6	0	-2	52	48	4
5	-3	-3	17	4	9	5	1	5	22	30	10	5	6	3	16	23	9	6	-7	-1	0	4	12	6	0	-1	31	34	7
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5	-3	5	35	37	8	5	2	1	144	138	3	5	7	0	62	61	3	6	-5	-3	38	37	6	6	1	-3	68	65	3
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5	-2	-4	41	43	4	5	2	3	23	14	9	5	7	2	33	39	6	6	-5	-1	0	8	12	6	1	-1	30	28	7
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5	-2	-2	0	4	10	5	2	5	36	35	5	5	8	-6	19	3	6	6	-5	1	49	51	7	6	1	1	90	88	4
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5	-2	0	161	156	3	5	3	-5	53	53	3	5	8	-4	77	74	3	6	-5	3	0	5	13	6	1	3	31	12	8
5	-2	1	76	75	4	5	3	-4	97	94	2	5	8	-3	55	54	3	6	-4	-4	29	34	4	6	1	4	13	4	11
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5	-2	3	7	21	12	5	3	-2	46	44	3	5	8	-1	16	25	9	6	-4	-2	20	2	11	6	2	-5	27	27	4
5	-2	4	55	56	5	5	3	-1	59	59	3	5	8	0	63	61	3	6	-4	-1	22	5	12	6	2	-4	42	44	4
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5	1	-5	11	22	8	5	5	4	37	29	5	5	11	0	25	6	8	6	-1	-1	22	11	10	6	4	1	45	36	5
5	1	-4	114	117	2	5	6	-6	17	13	7	5	11	1	14	17	9	6	-1	0	29	18	9	6	4	2	69	62	4

h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF
6	4	3	35	43	6	6	11	-3	37	45	4	7	3	-1	52	57	4	8	2	-3	4	11	8						
6	5	-6	17	3	6	6	11	-2	35	35	4	7	3	0	31	23	7	8	2	-2	0	16	9						
6	5	-5	0	22	8	6	11	-1	4	9	8	7	3	1	20	4	11	8	2	-1	30	33	7						
6	5	-4	0	3	8	6	11	0	15	9	8	7	3	2	8	26	11	8	2	0	0	17	10						
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6	5	1	45	41	5	7	-4	0	0	16	12	7	4	-1	0	22	9	8	4	-3	7	12	7						
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6	5	3	25	26	8	7	-3	-3	18	7	9	7	4	1	21	3	10	8	4	-1	7	28	9						
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6	8	1	25	30	6	7	1	-3	31	24	4	7	8	-2	34	33	4												
6	8	2	20	28	8	7	1	-2	51	53	4	7	8	-1	52	54	3												
6	9	-5	0	6	8	7	1	-1	47	41	5	7	8	0	24	18	6												
6	9	-4	0	17	8	7	1	0	32	26	8	7	8	1	11	5	8												
6	9	-3	18	21	8	7	1	1	37	36	7	7	9	-4	0	5	7												
6	9	-2	68	64	3	7	1	2	23	38	12	7	9	-3	0	7	8												
6	9	-1	51	42	3	7	2	-5	20	25	5	7	9	-2	39	38	4												
6	9	0	0	6	9	7	2	-4	0	2	8	7	9	-1	39	44	4												
6	9	1	16	3	8	7	2	-3	0	3	8	7	9	0	9	8	8												
6	9	2	21	21	6	7	2	-2	52	55	3	7	10	-3	15	21	7												
6	10	-5	8	4	7	7	2	-1	78	86	4	7	10	-2	37	40	4												
6	10	-4	23	10	5	7	2	0	40	42	6	7	10	-1	0	22	8												
6	10	-3	33	27	4	7	2	1	0	19	12	8	0	-2	0	13	9												
6	10	-2	72	65	3	7	2	2	26	30	10	8	0	-1	27	34	9												
6	10	-1	24	24	6	7	3	-5	13	12	7	8	0	0	17	30	11												
6	10	0	21	16	7	7	3	-4	21	24	6	8	1	-2	10	7	9												
6	10	1	0	3	9	7	3	-3	13	7	8	8	1	-1	34	35	7												
6	11	-4	6	15	8	7	3	-2	52	55	4	8	1	0	37	39	6												

APPENDIX 3.V

$[Ni(S_2COiBu)_2]$

Table 3.V.1: Crystallographic Parameters for $[Ni(S_2COCH_2CH(CH_3)_2)_2]$

Formula	$C_{10}H_{18}NiO_2S_4$
Formula weight	357.19
Crystal morphology / colour	block / brown
Crystal dimensions (mm)	0.08 x 0.10 x 0.35
Crystal system	triclinic
Space group	$P\bar{1}$ (#2)
a (Å)	8.224(7)
b (Å)	8.722(3)
c (Å)	6.139(4)
α (°)	98.99(4)
β (°)	105.36(5)
γ (°)	109.10(4)
V (Å^3)	386.7(5)
Z	1
$\rho_{calc.}$ (g cm^{-3})	1.534
$F(000)$	186
Temperature (°K)	293
μ (cm^{-1})	17.82
θ limits, cell (°)	20.5 – 24.2
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 10, -11 – 9, -7 – 6
Range of transmission factors	0.937 – 1
Scan technique	ω:2θ
No. of data measured	1898
No. of unique data	1775
R_{amal}	0.048
No. of parameters	79
R (I≥0σ(I))	0.075
R_w (I≥0σ(I))	0.095
R (I≥3σ(I))	0.040
R_w (I≥3σ(I))	0.044
Maximum and minimum residual electron density (e Å^{-3})	0.55, -1.05

Table 3.V.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Ni(S_2COCH_2CH(CH_3)_2)_2]$

Atom	*x*	*y*	*z*
Ni	0	0	0
S(1)	-0.0932(1)	-0.2596(1)	0.0336(2)
S(2)	0.1913(1)	0.0364(1)	0.3521(2)
O(1)	0.1077(3)	-0.2686(3)	0.4332(4)
C(1)	0.0758(4)	-0.1739(4)	0.2962(6)
C(2)	0.2545(5)	-0.1934(4)	0.6599(6)
C(3)	0.2738(4)	-0.3341(4)	0.7646(6)
C(4)	0.4092(5)	-0.2600(5)	1.0124(7)
C(5)	0.3317(6)	-0.4471(5)	0.6161(7)

Table 3.V.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Ni(S_2COCH_2CH(CH_3)_2)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	0.0443(4)	0.0432(3)	0.0472(4)	0.0171(3)	0.0172(3)	0.0128(3)
S(1)	0.0540(5)	0.0446(4)	0.0539(6)	0.0102(4)	0.0071(4)	0.0122(4)
S(2)	0.0526(5)	0.0406(4)	0.0521(5)	0.0128(4)	0.0129(4)	0.0108(4)
O(1)	0.052(1)	0.040(1)	0.052(1)	0.012(1)	0.006(1)	0.013(1)
C(1)	0.043(2)	0.042(2)	0.051(2)	0.017(1)	0.017(2)	0.013(1)
C(2)	0.049(2)	0.042(2)	0.048(2)	0.015(1)	0.011(2)	0.007(1)
C(3)	0.041(2)	0.044(2)	0.048(2)	0.015(1)	0.015(1)	0.011(1)
C(4)	0.065(2)	0.065(2)	0.054(2)	0.030(2)	0.017(2)	0.012(2)
C(5)	0.079(3)	0.061(2)	0.066(3)	0.038(2)	0.026(2)	0.011(2)

Table 3.V.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Ni(S_2COCH_2CH(CH_3)_2)_2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.3681	-0.1303	0.6389	4.6573
H(2b)	0.2243	-0.1187	0.7622	4.6573
H(3a)	0.1559	-0.4011	0.7713	4.2352
H(4a)	0.4212	-0.3484	1.0797	5.7990
H(4b)	0.5247	-0.1912	1.0094	5.7990
H(4c)	0.3665	-0.1940	1.1034	5.7990
H(5a)	0.3421	-0.5350	0.6854	6.2270
H(5b)	0.2429	-0.4940	0.4633	6.2270
H(5c)	0.4465	-0.3832	0.6063	6.2270

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 3.V.5: Bond distances (Å) for $[Ni(S_2COCH_2CH(CH_3)_2)_2]$

Ni — S(1)	2.2021(9)	O(1) — C(2)	1.460(4)
Ni — S(2)	2.215(1)	C(2) — C(3)	1.505(4)
S(1) — C(1)	1.687(3)	C(3) — C(4)	1.515(5)
S(2) — C(1)	1.695(3)	C(3) — C(5)	1.511(5)
O(1) — C(1)	1.305(4)		

Table 3.V.6: Bond angles (deg.) for $[Ni(S_2COCH_2CH(CH_3)_2)_2]$

S(1) — Ni — S(1)*	180(-)	S(1) — C(1) — S(2)	113.6(2)
S(1) — Ni — S(2)	79.69(3)	S(1) — C(1) — O(1)	120.1(2)
S(1) — Ni — S(2)*	100.31(3)	S(2) — C(1) — O(1)	126.4(2)
S(2) — Ni — S(2)*	180(-)	O(1) — C(2) — C(3)	107.7(2)
Ni — S(1) — C(1)	83.7(1)	C(2) — C(3) — C(4)	109.1(3)
Ni — S(2) — C(1)	83.1(1)	C(2) — C(3) — C(5)	111.2(3)
C(1) — O(1) — C(2)	119.7(2)	C(4) — C(3) — C(5)	111.3(3)

* atom related by a centre of symmetry

Table 3.V.7: Observed and calculated structure factors for $[Ni(S_2COCH_2CH(CH_3)_2)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	1	178	188	77	0	4	-1	336	333	99	0	9	-3	27	22	23	1	-6	1	-5	10	30	1	-2	0	176	171	79
0	0	2	309	331	99	0	4	1	113	113	58	0	9	-2	34	32	24	1	-6	2	51	53	20	1	-2	1	41	39	11
0	0	3	509	528	99	0	4	3	268	259	99	0	9	-1	66	64	28	1	-6	3	156	154	98	1	-2	2	74	73	25
0	0	4	110	109	54	0	4	4	43	38	31	0	9	0	22	15	19	1	-6	4	59	58	24	1	-2	3	161	161	86
0	0	5	133	136	76	0	4	5	100	95	58	0	-4	0	494	493	99	1	-6	5	20	41	45	1	-2	4	93	93	43
0	0	6	91	90	48	0	4	6	55	44	41	0	-4	-2	309	315	99	1	-6	6	59	58	26	1	-2	5	118	115	63
0	0	7	20	11	67	0	5	-7	14	8	41	0	9	1	-22	17	63	1	-6	7	3	3	26	1	-2	6	-9	19	57
0	1	-7	32	30	36	0	5	-6	58	56	27	0	9	2	42	38	35	1	-5	-5	63	60	42	1	-2	7	-20	21	64
0	1	-6	84	87	46	0	5	-5	102	106	48	0	10	-4	41	41	28	1	-5	-4	56	53	36	1	-1	-7	24	32	93
0	1	-5	64	62	30	0	5	-4	43	44	19	0	10	-3	19	10	24	1	-5	-3	218	221	99	1	-1	-6	128	120	89
0	1	-4	15	19	40	0	5	-3	138	139	78	0	10	-2	27	21	23	1	-5	-2	61	60	26	1	-1	-5	84	85	42
0	1	-3	127	148	69	0	5	-2	120	117	65	0	10	-1	57	64	30	1	-5	-1	145	146	82	1	-1	-4	157	156	99
0	1	-2	19	17	10	0	5	-1	123	121	63	0	10	0	-7	29	49	1	-5	0	318	302	99	1	-1	-3	363	380	99
0	1	-1	183	190	81	0	5	0	151	155	80	1	-11	2	-12	4	46	1	-5	1	144	137	75	1	-1	-2	30	25	12
0	1	1	95	96	35	0	5	1	-7	9	21	1	-10	-1	-23	44	63	1	-5	2	53	50	18	1	-1	-1	811	781	99
0	1	2	212	221	99	0	5	2	148	146	99	1	-10	0	22	27	25	1	-5	3	177	179	99	1	-1	1	85	85	29
0	1	3	414	418	99	0	5	3	131	127	78	1	-10	1	15	26	52	1	-5	4	43	44	19	1	-1	2	449	492	99
0	1	4	70	66	30	0	5	4	-16	5	63	1	-10	2	50	45	29	1	-5	5	23	19	16	1	-1	3	127	133	72
0	1	5	142	142	86	0	5	5	90	84	54	1	-10	3	53	53	30	1	-5	6	42	44	22	1	-1	4	-20	6	45
0	1	6	95	91	51	0	6	-7	29	31	22	1	-10	4	-18	14	48	1	-5	7	21	19	21	1	-1	5	68	59	34
0	1	7	40	34	37	0	6	-6	66	69	29	1	-9	-2	59	66	39	1	-4	-6	65	59	49	1	-1	6	-24	21	77
0	2	-7	-3	6	71	0	6	-5	29	28	18	1	-9	-1	21	8	43	1	-4	-5	59	55	40	1	-1	7	-30	7	65
0	2	-6	85	85	44	0	6	-4	80	80	32	1	-9	0	66	62	30	1	-4	-4	35	41	28	1	0	-7	44	34	40
0	2	-5	-23	6	53	0	6	-3	114	119	69	1	-9	1	94	95	46	1	-4	-3	278	273	99	1	0	-6	39	41	37
0	2	-4	48	40	21	0	6	-2	173	174	99	1	-9	2	38	37	24	1	-4	-2	161	159	95	1	0	-5	15	9	48
0	2	-3	209	209	99	0	6	-1	116	111	64	1	-9	3	62	62	30	1	-4	-1	117	115	56	1	0	-4	135	138	89
0	2	-2	161	160	75	0	6	0	37	38	16	1	-9	4	2	4	41	1	-4	0	564	554	99	1	0	-3	316	332	99
0	2	-1	165	166	72	0	6	1	84	86	36	1	-9	5	17	13	20	1	-4	1	56	62	16	1	0	-2	113	113	47
0	2	0	80	85	27	0	6	2	21	19	22	1	-8	-3	76	79	46	1	-4	2	311	298	99	1	0	-1	221	213	99
0	2	1	113	114	47	0	6	3	-32	10	59	1	-8	-2	58	61	34	1	-4	3	328	327	99	1	0	1	210	231	99
0	2	2	-13	10	26	0	6	4	-8	18	63	1	-8	-1	63	66	30	1	-4	4	87	84	39	1	0	2	382	414	99
0	2	3	26	26	19	0	6	5	28	17	35	1	-8	0	185	181	99	1	-4	5	91	91	45	1	0	3	176	185	99
0	2	4	87	91	38	0	7	-6	93	94	43	1	-8	1	85	84	35	1	-4	6	91	93	42	1	0	4	124	125	71
0	2	5	65	61	36	0	7	-5	62	64	26	1	-8	2	106	99	53	1	-4	7	-14	12	51	1	0	5	143	135	93
0	2	6	18	31	42	0	7	-4	127	124	64	1	-8	3	135	131	92	1	-3	-6	60	53	42	1	0	6	67	66	46
0	3	-7	13	39	67	0	7	-3	187	185	99	1	-8	4	62	66	28	1	-3	-5	43	39	31	1	0	7	-29	25	77
0	3	-6	118	121	63	0	7	-2	70	68	27	1	-8	5	42	43	24	1	-3	-4	-9	4	50	1	1	-7	24	6	63
0	3	-5	162	166	99	0	7	-1	205	204	99	1	-8	6	55	58	26	1	-3	-3	172	179	99	1	1	-6	74	71	40
0	3	-4	169	161	99	0	7	0	114	124	54	1	-7	-4	11	12	63	1	-3	-2	13	14	25	1	1	-5	60	57	30
0	3	-3	372	372	99	0	7	1	-14	13	44	1	-7	-3	9	16	58	1	-3	-1	-8	16	20	1	1	-4	89	93	37
0	3	-2	91	95	40	0	7	2	149	144	99	1	-7	-2	61	58	29	1	-3	0	370	359	99	1	1	-3	151	155	82
0	3	-1	444	445	99	0	7	3	-29	3	68	1	-7	-1	-35	12	43	1	-3	1	33	29	10	1	1	-2	142	137	62
0	3	0	328	324	99	0	7	4	11	6	50	1	-7	0	133	137	84	1	-3	2	391	376	99	1	1	-1	81	76	25
0	3	1	6	16	21	0	8	-6	34	34	22	1	-7	1	32	27	18	1	-3	3	202	192	99	1	1	0	598	621	99
0	3	2	238	244	99	0	8	-5	-1	3	27	1	-7	2	108	109	54	1	-3	4	-16	6	29	1	1	1	139	146	62
0	3	3	-9	7	37	0	8	-4	108	113	60	1	-7	3	187	183	99	1	-3	5	211	209	99	1	1	2	145	160	78
0	3	4	-24	14	52	0	8	-3	67	64	30	1	-7	4	53	48	23	1	-3	6	75	76	36	1	1	3	195	188	99
0	3	5	47	46	36	0	8	-2	105	105	52	1	-7	5	68	67	28	1	-3	7	12	23	59	1	1	4	45	42	28
0	3	6	22	8	34	0	8	-1	150	147	99	1	-7	6	83	85	35	1	-2	-7	19	18	76	1	1	5	152	147	99
0	4	-7	20	23	28	0	8	0	67	73	27	1	-6	-5	20	23	75	1	-2	-6	78	78	49	1	1	6	15	18	74
0	4	-6	90	87	40	0	8	1	102	101	50	1	-6	-4	10	7	58	1	-2	-5	37	38	33	1	2	-7	49	53	40
0	4	-5	86	88	38	0	8	2	138	130	88	1	-6	-3	77	77	38	1	-2	-4	147	144	99	1	2	-6	161	159	99
0	4	-4	143	152	83	0	8	3	24	26	65	1	-6	-2	38	41	23	1	-2	-3	92	94	41	1	2	-5	17	16	44
0	4	-3	221	232	99	0	9	-5	22	20	22	1	-6	-1	72	78	28	1	-2	-2	-10	8	22	1	2	-4	191	187	99
0	4	-2	334	340	99	0	9	-4	36	36	25	1	-6	0	147	143	84	1	-2	-1	298	290	99	1	2	-3	210	200	99
1	2	-2	13	10	9	1	6	-1	158	167	96	2	-8	-3	101	105	52	2	-4	1	265	260	99	2	0	-3	115	113	60
1	2	-1	431	419	99	1	6	0	45	44	20	2	-8	-2	37	35	24	2	-4	2	323	324	99	2	0	-2	29	26	10
1	2	0	83	83	33	1	6	1	68	63	28	2	-8	-1	77	80	33	2	-4	3	181	180	99	2	0	-1	772	787	99
1	2	1	51	56	15	1	6	2	40	33	25	2	-8	0	173	169	99	2	-4	4	246	242	99	2	0	0	46	42	11
1	2	2	132	136	74	1	6	3	34	38	34	2	-8	1	108	107	57	2	-4	5	130	132	87	2	0	1	217	223	99
1	2	3	27	23	22	1	6	4	18	17	76	2	-8	2	155	149	99	2	-4	6	57	58	29	2	0	2	591	626	99
1	2	4	-22	29	63	1	7	-6	32	33	23	2	-8	3	143	144	99	2	-4	7	48	49	29	2	0	3	68	74	29
1	2	5	46	49	37	1	7	-5	69	68	30	2	-8	4	54	56	26	2	-3	-7	-29	9	94	2	0	4	202	212	99
1	2	6	-28	5	70	1	7	-4	141	142	99	2	-8	5	92	94	44	2	-3	-6	43	37	41	2	0	5	138	123	91
1	3	-7	73	70	41	1	7	-3	111	106	56	2	-8	6	72	75	29	2	-3	-5	8	25	67	2	0	6	49	43	42
1	3	-6	96	93	45	1	7	-2	96	97	42	2	-7	-5	23	9	55	2	-3	-4	43	43	25	2	1	-7	71	70	48
1	3	-5	86	82	39	1	7	-1	180	188	99	2	-7	-4	7	10	68	2	-3	-3	48	50	21	2	1	-6	35	31	30
1	3	-4	230	235	99	1	7	0	87	91	39	2	-7	-3	52	47	30	2	-3	-2	9	6	26	2	1	-5	23	29	24
1	3	-3	346	335	99	1	7	1	99	93	49	2	-7	-2	91	93	41	2	-3	-1	98	92	44	2	1	-4	152	148	94
1	3	-2	96	93	42	1	7	2	86	81	44	2	-7	-1	50	53	23	2	-3	0	99	99	41	2	1	-3	123	124	65
1	3	-1	520	515	99	1	7	3	-26	4	72	2	-7	0	134	131	85	2	-3	1	235	233	99	2	1	-2	34	36	10
1	3	0	175	175	89	1	8	-6	25	2	22	2	-7	1	143	146	91	2	-3	2	265	257	99	2	1	-1	194	197	96
1	3	1	128	129	66	1	8	-5	-19	11	45	2	-7	2	154	153	99	2	-3	3	27	25	14	2	1	0	77	80	31
1	3	2	353	357	99	1	8	-4	63	59	30	2	-7	3	66	67	27	2	-3	4	10	9	33	2	1	1	62	65	20
1	3	3	27	32	23	1	8	-3	24	14	21	2	-7	4	-25	4	40	2	-3	5	119	118	66	2	1	2	42	43	18
1	3	4	35	30	29	1	8	-2	90	89	42	2	-7	5	104	98	51	2	-3	6	-6	12	57	2	1	3	71	75	32
1	3	5	91	87	52	1	8	-1	99	101	45	2	-7	6	39	37	21	2	-3	7	30	41	36	2	1	4	74	77	38
1	3	6	18	11	76	1	8	0	28	14	22	2	-6	-5	26	6	33	2	-2	-7	60	55	51	2	1	5	49	52	39
1	4	-7	37	38	29	1	8	1	80	70	41	2	-6	-4	75	78	45	2	-2	-6	97	94	57	2	1	6	23	9	79
1	4	-6	10	5	43	1	8	2	101	94	53	2	-6	-3	52	53	29	2	-2	-5	-20	9	55	2	2	-7	75	69	43
1	4	-5	30	29	19	1	9	-5	-13	5	46	2	-6	-2	13	5	19	2	-2	-4	255	255	99	2	2	-6	111	106	65
1	4	-4	205	202	99	1	9	-4	54	52	32	2	-6	-1	105	104	46	2	-2	-3	119	121	70	2	2	-5	94	95	45
1	4	-3	14	17	28	1	9	-3	63	60	31	2	-6	0	62	63	22	2	-2	-2	11	11	13	2	2	-4	278	272	99
1	4	-2	92	96	45	1	9	-2	45	45	26	2	-6	1	130	125	75	2	-2	-1	361	329	99	2	2	-3	99	100	51
1	4	-1	419	415	99	1	9	-1	20	20	21	2	-6	2	89	87	40	2	-2	0	104	104	43	2	2	-2	195	200	99
1	4	0	152	151	81	1	9	0	60	64	33	2	-6	3	61	60	23	2	-2	1	13	12	23	2	2	-1	287	289	99
1	4	1	107	111	48	1	9	1	53	56	35	2	-6	4	116	119	55	2	-2	2	157	161	79	2	2	0	18	17	10
1	4	2	316	319	99	1	10	-3	11	3	36	2	-6	5	44	45	21	2	-2	3	89	94	40	2	2	1	168	175	94
1	4	3	35	33	27	1	10	-2	33	38	28	2	-6	6	-2	6	25	2	-2	4	84	85	36	2	2	2	2	16	35
1	4	4	118	112	68	1	10	-1	49	49	27	2	-5	-6	73	65	54	2	-2	5	85	89	42	2	2	3	42	42	26
1	4	5	100	94	59	2	-11	1	-16	5	48	2	-5	-5	27	25	78	2	-2	6	49	41	35	2	2	4	24	46	68
1	5	-7	42	42	29	2	-11	2	24	25	26	2	-5	-4	164	158	99	2	-2	7	20	13	63	2	2	5	-31	4	72
1	5	-6	-15	15	47	2	-11	3	39	38	26	2	-5	-3	251	248	99	2	-1	-7	63	64	51	2	2	6	48	40	40
1	5	-5	16	22	40	2	-10	-2	18	9	36	2	-5	-2	71	71	25	2	-1	-6	89	83	50	2	3	-7	75	72	41
1	5	-4	118	118	72	2	-10	-1	34	28	23	2	-5	-1	325	327	99	2	-1	-5	108	106	56	2	3	-6	83	81	38
1	5	-3	91	90	39	2	-10	0	65	62	33	2	-5	0	299	294	99	2	-1	-4	369	359	99	2	3	-5	127	126	67
1	5	-2	58	55	19	2	-10	1	34	31	26	2	-5	1	207	203	99	2	-1	-3	233	230	99	2	3	-4	234	230	99
1	5	-1	131	133	70	2	-10	2	39	31	26	2	-5	2	259	260	99	2	-1	-2	255	247	99	2	3	-3	210	205	99
1	5	0	39	44	16	2	-10	3	15	37	40	2	-5	3	133	133	76	2	-1	-1	1090	1041	99	2	3	-2	383	380	99
1	5	1	28	23	19	2	-10	4	-15	21	47	2	-5	4	35	35	18	2	-1	0	347	360	99	2	3	-1	489	492	99
1	5	2	109	100	62	2	-9	-3	75	83	47	2	-5	5	110	111	54	2	-1	1	312	303	99	2	3	0	86	94	34
1	5	3	41	40	32	2	-9	-2	61	63	33	2	-5	6	-24	12	38	2	-1	2	415	451	99	2	3	1	386	396	99
1	5	4	34	26	35	2	-9	-1	57	59	30	2	-5	7	-9	2	40	2	-1	3	148	158	99	2	3	2	231	236	99
1	5	5	58	44	41	2	-9	0	105	102	59	2	-4	-6	48	37	43	2	-1	4	108	103	63	2	3	3	69	68	34
1	6	-7	61	66	30	2	-9	1	63	62	29	2	-4	-5	28	9	31	2	-1	5	124	117	32	2	3	4	129	124	35
1	6	-6	67	67	31	2	-9	2	51	52	28	2	-4	-4	96	87	50	2	-1	6	-7	5	67	2	3	5	49	34	41
1	6	-5	11	11	40	2	-9	3	96	91	44	2	-4	-3	173	166	99	2	0	-7	31	53	98	2	4	-7	42	40	29
1	6	-4	172	166	99	2	-9	4	18	5	42	2	-4	-2	22	21	14	2	0	-6	19	6	58	2	4	-6	50	53	29
1	6	-3	106	105	50	2	-9	5	3	25	45	2	-4	-1	418	401	99	2	0	-5	21	12	46	2	4	-5	46	47	24
1	6	-2	-13	6	30	2	-8	-4	-18	6	66	2	-4	0	311	306	99	2	0	-4	178	168	99	2	4	-4	34	30	44

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
2	4	-3	3	6	25	2	9	-1	26	29	25	3	-6	6	16	31	40	3	-1	-7	48	43	49	3	3	-7	4	3	67
2	4	-2	119	115	61	2	9	0	28	29	29	3	-5	-6	16	29	93	3	-1	-6	27	13	30	3	3	-6	41	36	27
2	4	-1	192	195	99	3	-11	0	36	29	26	3	-5	-5	-26	6	70	3	-1	-5	76	72	37	3	3	-5	140	142	99
2	4	0	51	47	18	3	-11	1	45	41	29	3	-5	-4	175	174	99	3	-1	-4	285	271	99	3	3	-4	72	76	27
2	4	1	171	175	99	3	-11	2	51	52	30	3	-5	-3	72	68	29	3	-1	-3	45	42	17	3	3	-3	95	100	40
2	4	2	224	217	99	3	-11	3	26	25	24	3	-5	-2	147	151	86	3	-1	-2	298	299	99	3	3	-2	304	313	99
2	4	3	11	7	28	3	-10	-2	5	11	44	3	-5	-1	356	342	99	3	-1	-1	509	483	99	3	3	-1	194	195	99
2	4	4	118	111	72	3	-10	-1	36	44	29	3	-5	0	93	92	39	3	-1	0	43	41	13	3	3	0	113	113	65
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9	-4	-1	67	71	47	10	-4	-3	26	37	88																		
9	-4	0	28	32	78	10	-4	-2	67	77	52																		
9	-4	1	-27	33	85	10	-4	-1	-42	24	85																		
9	-4	2	4	27	63	10	-4	0	43	49	41																		
9	-3	-5	-7	25	79	10	-3	-4	-29	14	88																		

APPENDIX 3.VI

$[Ni(S_2CO\text{-}3\text{-}MeBu)_2]$

Table 3.VI.1: Crystallographic Parameters for [Ni($S_2COCH_2CH_2CH(CH_3)2)2$]

Formula	$C_{12}H_{22}NiO_2S_4$
Formula weight	385.24
Crystal morphology / colour	block / brown
Crystal dimensions (mm)	0.10 x 0.27 x 0.36
Crystal system	triclinic
Space group	$P\bar{1}$(#2)
a (Å)	6.585(3)
b (Å)	11.35(1)
c (Å)	6.315(2)
α (°)	102.42(5)
β (°)	94.96(3)
γ (°)	87.98(6)
V (Å^3)	459.1(6)
Z	1
$\rho_{calc.}$ (g cm^{-3})	1.393
F(000)	202
Temperature (°K)	293
μ (cm^{-1})	15.06
θ limits, cell (°)	17.0 – 18.5
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 8, -14 – 14, -8 – 8.
Range of transmission factors	0.908 – 1
Scan technique	ω:2θ
No. of data measured	2316
No. of unique data	2129
R_{amal}	0.081
No. of parameters	88
R (I≥0σ(I))	0.088
R_w (I≥0σ(I))	0.134
R (I≥3σ(I))	0.055
R_w (I≥3σ(I))	0.057
Maximum and minimum residual electron density (e Å^{-3})	0.62, -0.44

Table 3.VI.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Ni(S_2COCH_2CH_2CH(CH_3)_2)_2]$

Atom	*x*	*y*	*z*
Ni	0	0	0
S(1)	0.2153(2)	0.0228(2)	-0.2361(2)
S(2)	0.1865(3)	0.1481(2)	0.1971(3)
O(1)	0.4566(6)	0.2032(4)	-0.0393(6)
C(1)	0.3076(8)	0.1309(6)	-0.0329(9)
C(2)	0.563(1)	0.1899(7)	-0.241(1)
C(3)	0.709(1)	0.300(1)	-0.179(1)
C(4)	0.820(1)	0.3058(8)	-0.342(2)
C(5)	1.004(1)	0.3879(9)	-0.286(1)
C(6)	0.696(2)	0.349(1)	-0.524(2)

Table 3.VI.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Ni(S_2COCH_2CH_2CH(CH_3)_2)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	0.0969(7)	0.116(1)	0.0823(7)	-0.0198(7)	0.0110(5)	0.0219(7)
S(1)	0.122(1)	0.139(2)	0.092(1)	-0.032(1)	0.0259(9)	0.018(1)
S(2)	0.133(1)	0.159(2)	0.093(1)	-0.048(1)	0.0156(9)	0.011(1)
O(1)	0.120(3)	0.140(4)	0.129(3)	-0.042(3)	-0.003(2)	0.045(3)
C(1)	0.100(4)	0.123(6)	0.109(5)	-0.030(4)	-0.003(3)	0.039(4)
C(2)	0.122(5)	0.223(9)	0.121(5)	-0.030(5)	0.022(4)	0.073(6)
C(3)	0.170(7)	0.38(1)	0.154(7)	-0.167(8)	0.008(5)	0.096(8)
C(4)	0.177(8)	0.21(1)	0.212(10)	-0.096(7)	-0.059(7)	0.091(8)
C(5)	0.202(8)	0.22(1)	0.223(9)	-0.074(8)	0.002(7)	0.052(7)
C(6)	0.23(1)	0.39(2)	0.28(1)	-0.12(1)	-0.063(9)	0.19(1)

Table 3.VI.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in [Ni($S_2COCH_2CH_2CH(CH_3)_2)_2$]*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.6377	0.1140	-0.2717	14.1
H(2b)	0.4677	0.1960	-0.3644	14.1
H(3a)	0.6292	0.3740	-0.1437	21.1
H(3b)	0.7999	0.2917	-0.0531	21.1
H(4a)	0.8700	0.2246	-0.4005	18.3
H(5a)	1.0944	0.3624	-0.1734	20.2
H(5b)	1.0776	0.3832	-0.4145	20.2
H(5c)	0.9589	0.4705	-0.2337	20.2
H(6a)	0.6542	0.4324	-0.4747	26.7
H(6b)	0.7790	0.3432	-0.6472	26.7
H(6c)	0.5770	0.2995	-0.5706	26.7

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 3.VI.5: Bond distances (Å) for [Ni($S_2COCH_2CH_2CH(CH_3)_2)_2$]

Ni — S(1)	2.203(1)	O(1) — C(2)	1.479(6)
Ni — S(2)	2.205(2)	C(2) — C(3)	1.571(9)
S(1) — C(1)	1.663(6)	C(3) — C(4)	1.33(1)
S(2) — C(1)	1.688(6)	C(4) — C(5)	1.522(9)
O(1) — C(1)	1.309(5)	C(4) — C(6)	1.51(1)

Table 3.VI.6: Bond angles (deg.) for [Ni($S_2COCH_2CH_2CH(CH_3)_2)_2$]

S(1) — Ni — S(1)*	180(-)	S(1) — C(1) — O(1)	126.3(4)
S(1) — Ni — S(2)	79.44(6)	S(2) — C(1) — O(1)	119.2(5)
S(1) — Ni — S(2)*	100.56(6)	O(1) — C(2) — C(3)	101.3(6)
S(2) — Ni — S(2)*	180(-)	C(2) — C(3) — C(4)	111.2(8)
Ni — S(1) — C(1)	83.4(2)	C(3) — C(4) — C(5)	116.1(8)
Ni — S(2) — C(1)	82.7(2)	C(3) — C(4) — C(6)	112.0(9)
C(1) — O(1) — C(2)	119.7(5)	C(5) — C(4) — C(6)	105.2(8)
S(1) — C(1) — S(2)	114.4(3)		

* atom related by a centre of symmetry

Table 3.VI.7: Observed and calculated structure factors for $[Ni(S_2COCH_2CH_2CH(CH_3)_2)_2]$

h	k	l	Fo	Fc	sigF2
0	0	1	406	448	99
0	0	2	150	142	57
0	0	3	59	54	23
0	0	4	246	230	99
0	0	5	154	133	76
0	0	6	21	18	28
0	0	7	-13	3	22
0	0	8	6	6	7
0	1	-8	17	7	9
0	1	-7	5	8	22
0	1	-6	11	19	26
0	1	-5	99	83	43
0	1	-4	110	115	43
0	1	-3	45	46	18
0	1	-2	15	8	11
0	1	-1	280	298	99
0	1	1	677	675	99
0	1	2	55	55	17
0	1	3	108	111	40
0	1	4	275	256	99
0	1	5	127	122	61
0	1	6	17	3	25
0	1	7	-13	4	21
0	2	-8	7	11	10
0	2	-7	20	22	22
0	2	-6	13	16	24
0	2	-5	-13	9	24
0	2	-4	71	58	27
0	2	-3	36	44	18
0	2	-2	101	96	30
0	2	-1	374	374	99
0	2	1	42	43	14
0	2	2	33	21	14
0	2	3	143	148	60
0	2	4	194	187	99
0	2	5	75	68	39
0	2	6	-12	7	27
0	2	7	-9	1	18
0	3	-8	12	16	9
0	3	-7	8	25	23
0	3	-6	22	13	22
0	3	-5	-17	8	22
0	3	-4	80	80	31
0	3	-3	168	167	73
0	3	-2	129	126	45
0	3	-1	49	44	14
0	3	0	191	203	87
0	3	1	25	9	14
0	3	2	43	38	19
0	3	3	85	92	35
0	3	4	89	89	43
0	3	5	22	25	32
0	3	6	-9	1	27
0	3	7	-8	6	14
0	4	-8	11	18	8
0	4	-7	11	15	18
0	4	-6	-13	6	22
0	4	-5	51	51	27
0	4	-4	164	166	74
0	4	-3	236	240	99
0	4	-2	71	71	23
0	4	-1	35	21	15
0	4	0	102	98	36
0	4	1	248	241	99
0	4	2	116	122	47
0	4	3	47	40	28
0	4	4	29	30	30
0	4	5	12	19	29
0	4	6	-7	8	27
0	4	7	-14	7	13
0	5	-8	5	16	7
0	5	-7	5	10	15
0	5	-6	-13	12	20
0	5	-5	35	42	22
0	5	-4	214	208	99
0	5	-3	182	182	87
0	5	-2	20	6	16
0	5	-1	26	27	18
0	5	0	274	268	99
0	5	1	295	285	99
0	5	2	91	88	41
0	5	3	15	6	28
0	5	4	21	21	32
0	5	5	32	33	36
0	5	6	28	15	24
0	6	-8	7	10	4
0	6	-7	6	10	13
0	6	-6	17	3	18
0	6	-5	33	36	26
0	6	-4	124	129	56
0	6	-3	117	109	51
0	6	-2	29	35	23
0	6	-1	-18	12	23
0	6	0	263	250	99
0	6	1	209	223	99
0	6	2	34	32	37
0	6	3	41	26	38
0	6	4	18	27	38
0	6	5	41	39	41
0	6	6	25	13	22
0	7	-7	11	13	12
0	7	-6	16	14	22
0	7	-5	44	35	30
0	7	-4	59	62	38
0	7	-3	61	66	36
0	7	-2	-12	4	28
0	7	-1	49	42	35
0	7	0	153	147	82
0	7	1	108	111	61
0	7	2	23	2	39
0	7	3	13	7	18
0	7	4	23	29	46
0	7	5	-18	29	41
0	7	6	-3	6	15
0	8	-7	-9	13	14
0	8	-6	-13	17	27
0	8	-5	23	19	32
0	8	-4	47	43	41
0	8	-3	52	53	42
0	8	-2	57	54	40
0	8	-1	50	53	41
0	8	0	71	76	49
0	8	1	64	55	49
0	8	2	17	5	47
0	8	3	-23	6	53
0	8	4	1	24	53
0	8	5	11	16	30
0	9	-7	1	10	13
0	9	-6	-9	10	28
0	9	-5	17	8	33
0	9	-4	26	29	40
0	9	-3	59	55	49
0	9	-2	60	53	49
0	9	-1	36	42	49
0	9	0	37	38	51
0	-2	0	73	62	17
0	9	1	23	35	53
0	9	2	-23	21	58
0	9	3	33	13	56
0	9	4	28	13	46
0	9	5	-14	7	22
0	10	-7	-7	5	11
0	10	-6	-9	2	26
0	10	-5	14	1	37
0	10	-4	-18	17	46
0	10	-3	46	42	49
0	10	-2	24	27	47
0	10	-1	19	9	49
0	10	0	20	20	54
0	10	1	13	29	63
0	10	2	31	21	61
0	10	3	-17	9	57
0	10	4	-17	5	39
0	11	-6	21	0	20
0	11	-5	-24	1	38
0	11	-4	-11	11	48
0	11	-3	11	21	53
0	11	-2	16	10	49
0	11	-1	23	6	53
0	11	0	-21	7	58
0	11	1	3	21	67
0	11	2	-14	13	65
0	11	3	17	2	50
0	11	4	-17	1	24
0	12	-6	10	2	14
0	12	-5	8	2	30
0	12	-4	-9	9	43
0	12	-3	21	12	48
0	12	-2	24	3	49
0	12	-1	-21	3	55
0	12	0	-40	5	61
0	12	1	25	12	58
0	12	2	24	6	55
0	12	3	28	0	29
0	13	-5	3	4	19
0	13	-4	18	6	30
0	13	-3	5	9	39
0	13	-2	-29	4	46
0	13	-1	-34	1	48
0	13	0	28	5	49
0	13	1	-37	6	50
0	13	2	6	2	32
0	14	-4	-11	4	19
0	14	-3	-12	6	28
0	14	-2	27	5	30
0	14	-1	-12	3	34
0	14	0	10	3	31
1	-14	0	3	3	19
1	-14	1	21	1	21
1	-14	2	4	0	23
1	-14	3	14	2	19
1	-13	-2	9	2	13
1	-13	-1	-11	5	29
1	-13	0	18	6	37
1	-13	1	-1	2	37
1	-13	2	13	1	37
1	-13	3	-3	4	34
1	-13	4	-12	4	23
1	-13	5	12	2	13
1	-12	-3	-8	2	12
1	-12	-2	1	4	29
1	-12	-1	28	8	46
1	-12	0	-17	10	52
1	-12	1	6	6	50
1	-12	2	-25	5	47
1	-12	3	-17	9	44
1	-12	4	16	9	31
1	-12	5	-3	3	23
1	-11	-3	13	6	26
1	-11	-2	11	5	45
1	-11	-1	-10	10	56
1	-11	0	25	16	53
1	-11	1	-22	15	56
1	-11	2	-22	14	47
1	-11	3	19	16	44
1	-11	4	-9	14	40
1	-11	5	-16	7	31
1	-11	6	-5	3	17
1	-10	-4	-8	10	19
1	-10	-3	-18	9	40
1	-10	-2	-19	1	55
1	-10	-1	23	11	58
1	-10	0	9	29	64
1	-10	1	21	27	54
1	-10	2	28	17	43
1	-10	3	-11	17	44
1	-10	4	18	18	42
1	-10	5	-10	12	37
1	-10	6	-6	6	25
1	-9	-5	-11	7	13
1	-9	-4	-8	18	32
1	-9	-3	12	10	40
1	-9	-2	-11	6	50
1	-9	-1	19	19	54
1	-9	0	64	60	68
1	-9	1	55	46	52
1	-9	2	-12	8	39
1	-9	3	-13	15	40
1	-9	4	29	26	39
1	-9	5	16	20	40
1	-9	6	23	8	27
1	-9	7	8	5	14
1	-8	-5	16	12	23
1	-8	-4	-22	24	40
1	-8	-3	15	8	41
1	-8	-2	24	8	46
1	-8	-1	42	45	57
1	-8	0	120	112	83
1	-8	1	78	72	54
1	-8	2	-19	3	35
1	-8	3	13	12	34
1	-8	4	56	53	44
1	-8	5	32	34	40
1	-8	6	-24	8	33
1	-8	7	-11	3	19
1	-7	-6	10	4	12
1	-7	-5	11	13	28
1	-7	-4	17	23	41
1	-7	-3	17	11	42
1	-7	-2	18	9	40
1	-7	-1	83	77	61
1	-7	0	123	138	77
1	-7	1	84	87	47
1	-7	2	17	12	26
1	-7	3	22	28	31
1	-7	4	95	82	55
1	-7	5	54	53	45
1	-7	6	-19	8	35
1	-7	7	-15	1	18
1	-6	-6	19	5	16
1	-6	-5	-13	7	32
1	-6	-4	22	22	36
1	-6	-3	29	31	42
1	-6	-2	47	35	39
1	-6	-1	88	78	51
1	-6	0	130	114	66
1	-6	1	52	57	32
1	-6	2	21	16	23
1	-6	3	77	79	39
1	-6	4	71	79	46
1	-6	5	57	52	39
1	-6	6	-22	10	30
1	-6	7	3	3	19
1	-5	-6	13	1	22
1	-5	-5	17	1	30
1	-5	-4	48	44	42
1	-5	-3	87	89	53
1	-5	-2	64	57	40
1	-5	-1	23	5	28
1	-5	0	59	58	31
1	-5	1	75	70	32
1	-5	2	46	36	25
1	-5	3	62	56	31
1	-5	4	70	72	37
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1	-5	6	5	8	25
1	-5	7	-9	9	19
1	-4	-7	-7	10	12
1	-4	-6	14	7	23
1	-4	-5	-17	6	28
1	-4	-4	110	107	58
1	-4	-3	181	170	98
1	-4	-2	69	66	35
1	-4	-1	22	6	21
1	-4	0	53	47	25
1	-4	1	189	201	91
1	-4	2	146	146	61
1	-4	3	-10	9	17
1	-4	4	44	39	25
1	-4	5	-15	26	27
1	-4	6	18	9	24
1	-4	7	9	9	19
1	-4	8	3	8	6
1	-3	-7	-19	7	16
1	-3	-6	-4	11	24
1	-3	-5	31	29	29
1	-3	-4	179	178	94
1	-3	-3	231	232	99
1	-3	-2	14	9	20
1	-3	-1	54	57	23
1	-3	0	500	518	99
1	-3	1	502	482	99
1	-3	2	118	116	41
1	-3	3	36	38	18
1	-3	4	34	37	23
1	-3	5	88	80	41
1	-3	6	29	31	26
1	-3	7	17	6	16
1	-3	8	-8	4	6
1	-2	-7	-13	5	17
1	-2	-6	-6	3	25
1	-2	-5	61	49	32
1	-2	-4	182	183	92
1	-2	-3	196	202	98
1	-2	-2	15	10	16
1	-2	-1	105	115	35
1	-2	0	876	875	99
1	-2	1	900	930	99
1	-2	2	193	179	89
1	-2	3	68	69	24
1	-2	4	113	114	45
1	-2	5	143	134	68
1	-2	6	58	53	32
1	-2	7	-15	3	18
1	-2	8	-4	1	6
1	-1	-7	-10	12	21
1	-1	-6	18	15	24
1	-1	-5	43	41	28
1	-1	-4	114	109	45
1	-1	-3	98	99	34
1	-1	-2	23	14	14
1	-1	-1	146	160	52
1	-1	0	247	218	99
1	-1	1	688	650	99
1	-1	2	232	238	99
1	-1	3	25	21	14
1	-1	4	149	138	65
1	-1	5	149	149	71
1	-1	6	50	45	28
1	-1	7	-10	2	17
1	0	-8	-7	7	9
1	0	-7	19	28	22
1	0	-6	32	35	27
1	0	-5	6	8	21
1	0	-4	27	18	17
1	0	-3	85	90	28
1	0	-2	79	72	22
1	0	-1	232	225	99
1	0	1	26	13	8
1	0	2	57	64	16
1	0	3	75	73	25
1	0	4	133	134	56
1	0	5	113	109	51
1	0	6	14	22	23
1	0	7	-8	3	16
1	1	-8	10	13	10
1	1	-7	35	39	24
1	1	-6	36	36	24
1	1	-5	-8	3	21
1	1	-4	28	30	19
1	1	-3	168	168	71
1	1	-2	332	337	99
1	1	-1	37	44	10
1	1	0	196	175	86
1	1	1	36	29	10
1	1	2	57	58	18
1	1	3	48	52	20
1	1	4	84	83	34
1	1	5	52	48	29
1	1	6	13	9	23
1	1	7	15	2	15
1	2	-8	13	19	10
1	2	-7	43	41	25
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8	1	0	9	4	6
8	1	1	8	2	5
8	1	2	-3	5	3
8	2	-3	-2	3	3
8	2	-2	5	5	4
8	2	-1	10	11	5
8	2	0	7	10	6
8	2	1	9	6	5
8	3	-3	-1	1	3
8	3	-2	9	4	4
8	3	-1	11	13	6
8	3	0	11	12	6
8	3	1	5	6	5
8	4	-3	-6	3	3
8	4	-2	1	5	5
8	4	-1	7	16	6
8	4	0	13	13	6
8	4	1	1	2	4
8	5	-2	8	6	5
8	5	-1	10	14	6
8	5	0	10	10	5

APPENDIX 3.VII

$[Ni(S_2CO\text{-}3,3\text{-}Me_2Bu)_2]$

Table 3.VII.1: Crystallographic Parameters for $[Ni(S_2COCH_2CH_2C(CH_3)_3)_2]$

Formula	$C_{14}H_{26}NiO_2S_4$
Formula weight	413.30
Crystal morphology / colour	plate / brown
Crystal dimensions (mm)	0.05 x 0.15 x 0.24
Crystal system	triclinic
Space group	$P\bar{1}$ (#2)
a (Å)	6.653(2)
b (Å)	12.173(4)
c (Å)	6.156(1)
α (°)	98.52(2)
β (°)	94.55(1)
γ (°)	91.17(3)
V (Å^3)	491.2(2)
Z	1
$\rho_{calc.}$ (g cm^{-3})	1.397
$F(000)$	218
Temperature (°K)	293
μ (cm^{-1})	14.13
θ limits, cell (°)	21.8 – 24.2
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 8, -15 – 15, -7 – 7
Range of transmission factors	0.960 – 1
Scan technique	ω:2θ
No. of data measured	2458
No. of unique data	2267
R_{amal}	0.029
No. of parameters	97
R ($I \geq 0\sigma(I)$)	0.065
R_w ($I \geq 0\sigma(I)$)	0.067
R ($I \geq 3\sigma(I)$)	0.038
R_w ($I \geq 3\sigma(I)$)	0.029
Maximum and minimum residual electron density (e Å^{-3})	0.58, -0.54

Table 3.VII.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Ni(S_2COCH_2CH_2C(CH_3)_3)_2]$

Atom	*x*	*y*	*z*
Ni	0	0	0
S(1)	0.2210(1)	0.00814(7)	-0.2458(1)
S(2)	0.1947(1)	0.14273(7)	0.1655(1)
O(1)	0.4745(3)	0.1809(2)	-0.0916(3)
C(1)	0.3201(4)	0.1174(3)	-0.0654(5)
C(2)	0.5819(5)	0.1497(3)	-0.2923(5)
C(3)	0.7776(4)	0.2182(3)	-0.2661(5)
C(4)	0.7730(4)	0.3354(3)	-0.3257(5)
C(5)	0.9889(5)	0.3841(3)	-0.2795(6)
C(6)	0.7074(5)	0.3330(3)	-0.5691(6)
C(7)	0.6338(5)	0.4089(3)	-0.1863(6)

Table 3.VII.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Ni(S_2COCH_2CH_2C(CH_3)_3)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	0.0259(3)	0.0357(4)	0.0284(4)	-0.0051(3)	0.0067(3)	0.0017(3)
S(1)	0.0365(5)	0.0411(5)	0.0337(5)	-0.0101(4)	0.0128(4)	-0.0047(4)
S(2)	0.0326(5)	0.0442(5)	0.0277(5)	-0.0086(4)	0.0073(4)	-0.0031(4)
O(1)	0.032(1)	0.039(1)	0.032(1)	-0.009(1)	0.009(1)	0.002(1)
C(1)	0.025(2)	0.038(2)	0.027(2)	0.003(1)	0.000(1)	0.010(1)
C(2)	0.039(2)	0.036(2)	0.030(2)	-0.003(2)	0.013(1)	0.004(1)
C(3)	0.026(2)	0.043(2)	0.034(2)	-0.001(1)	0.005(1)	0.005(2)
C(4)	0.032(2)	0.035(2)	0.030(2)	-0.007(1)	0.007(1)	0.004(1)
C(5)	0.051(2)	0.061(3)	0.068(3)	-0.026(2)	0.007(2)	0.010(2)
C(6)	0.073(3)	0.055(2)	0.036(2)	-0.015(2)	-0.002(2)	0.015(2)
C(7)	0.064(3)	0.039(2)	0.057(2)	0.001(2)	0.013(2)	0.006(2)

Table 3.VII.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Ni(S_2COCH_2CH_2C(CH_3)_3)_2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.6101	0.0729	-0.3088	3.3
H(2b)	0.5013	0.1647	-0.4182	3.3
H(3a)	0.8281	0.2245	-0.1159	3.2
H(3b)	0.8691	0.1779	-0.3562	3.2
H(5a)	1.0328	0.3853	-0.1252	5.7
H(5b)	1.0778	0.3382	-0.3699	5.7
H(5c)	0.9932	0.4590	-0.3149	5.7
H(6a)	0.7117	0.4080	-0.6044	5.1
H(6b)	0.7977	0.2875	-0.6574	5.1
H(6c)	0.5709	0.3019	-0.6004	5.1
H(7a)	0.6777	0.4117	-0.0313	5.0
H(7b)	0.6387	0.4835	-0.2243	5.0
H(7c)	0.4967	0.3786	-0.2142	5.0

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 3.VII.5: Bond distances (Å) for $[Ni(S_2COCH_2CH_2C(CH_3)_3)_2]$

Ni — S(1)	2.2026(8)	C(3) — C(4)	1.525(4)
Ni — S(2)	2.2155(8)	C(4) — C(5)	1.532(4)
S(2) — C(1)	1.697(3)	C(4) — C(6)	1.523(4)
S(1) — C(1)	1.687(3)	C(4) — C(7)	1.525(4)
O(1) — C(1)	1.308(3)	C(2) — C(3)	1.516(4)
O(1) — C(2)	1.481(3)		

Table 3.VII.6: Bond angles (deg.) for $[Ni(S_2COCH_2CH_2C(CH_3)_3)_2]$

S(1) — Ni — S(1)*	180(-)	S(1) — C(1) — O(1)	126.5(2)
S(1) — Ni — S(2)	79.46(3)	O(1) — C(2) — C(3)	107.9(2)
S(1) — Ni — S(2)*	100.54(3)	C(2) — C(3) — C(4)	118.5(3)
S(2) — Ni — S(2)*	180(-)	C(3) — C(4) — C(5)	106.7(3)
Ni — S(2) — C(1)	83.4(1)	C(3) — C(4) — C(6)	111.0(3)
Ni — S(1) — C(1)	84.0(1)	C(3) — C(4) — C(7)	111.7(2)
C(1) — O(1) — C(2)	117.0(2)	C(5) — C(4) — C(6)	108.8(3)
S(1) — C(1) — S(2)	113.1(2)	C(5) — C(4) — C(7)	109.0(3)
S(2) — C(1) — O(1)	120.4(2)	C(6) — C(4) — C(7)	109.6(3)

* atom related by a centre of symmetry

Table 3.VII.7: Observed and calculated structure factors for $[Ni(S_2COCH_2CH_2C(CH_3)_3)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	1	450	439	99	0	4	-3	306	318	99	0	8	-6	63	52	98	0	12	0	120	130	99	1	-12	4	85	81	99
0	0	2	304	290	99	0	4	-2	114	117	65	0	8	-5	30	19	84	0	12	1	112	106	99	1	-12	5	64	62	99
0	0	3	93	90	67	0	4	-1	187	185	89	0	8	-4	165	165	99	0	12	2	92	82	99	1	-11	-4	135	140	99
0	0	4	194	193	99	0	4	0	79	80	61	0	8	-3	77	77	82	0	12	3	81	83	99	1	-11	-3	148	158	99
0	0	5	87	87	89	0	4	1	27	28	58	0	8	-2	28	13	68	0	12	4	66	35	99	1	-11	-2	-41	26	99
0	0	6	40	41	89	0	4	2	101	104	75	0	8	-1	197	195	99	0	13	-5	60	67	99	1	-11	-1	71	79	99
0	0	7	75	88	99	0	4	3	169	164	99	0	8	0	299	294	99	0	13	-4	99	97	99	1	-11	0	123	116	99
0	1	-7	95	101	99	0	4	4	251	253	99	0	8	1	156	154	99	0	13	-3	-16	44	99	1	-11	1	135	136	99
0	1	-6	34	47	91	0	4	5	-41	26	99	0	8	2	96	93	99	0	13	-2	77	84	99	1	-11	2	106	102	99
0	1	-5	-22	8	76	0	4	6	57	47	98	0	8	3	43	11	89	0	13	-1	18	24	99	1	-11	3	76	79	99
0	1	-4	42	33	61	0	4	7	39	44	99	0	8	4	172	170	99	0	13	0	116	113	99	1	-11	4	78	71	99
0	1	-3	143	147	78	0	5	-7	30	25	88	0	8	5	157	164	99	0	13	1	119	124	99	1	-11	5	45	34	99
0	1	-2	78	75	50	0	5	-6	30	25	86	0	8	6	45	32	99	0	13	2	32	17	99	1	-11	6	-36	16	99
0	1	-1	196	192	69	0	5	-5	122	117	99	0	9	-7	58	57	99	0	13	3	-33	3	99	1	-10	-5	51	54	99
0	1	1	764	787	99	0	5	-4	461	457	99	0	9	-6	9	4	99	0	14	-4	54	72	99	1	-10	-4	140	136	99
0	1	2	16	18	51	0	5	-3	167	164	97	0	9	-5	-32	10	92	0	14	-3	61	42	99	1	-10	-3	140	139	99
0	1	3	31	35	59	0	5	-2	193	196	99	0	9	-4	72	72	99	0	14	-2	42	10	99	1	-10	-2	-25	15	89
0	1	4	459	456	99	0	5	-1	175	170	91	0	9	-3	179	186	99	0	14	-1	35	31	99	1	-10	-1	35	38	86
0	1	5	201	201	99	0	5	0	478	481	99	0	9	-2	190	183	99	0	14	0	108	102	99	1	-10	0	122	109	99
0	1	6	-42	8	91	0	5	1	335	342	99	0	9	-1	111	118	99	0	14	1	79	69	99	1	-10	1	227	228	99
0	1	7	11	9	96	0	5	2	72	80	77	0	9	0	195	199	99	0	14	2	59	42	99	1	-10	2	94	90	99
0	2	-7	114	104	99	0	5	3	83	76	90	0	9	1	43	28	81	0	15	-3	73	69	99	1	-10	3	12	21	86
0	2	-6	-7	33	83	0	5	4	49	51	95	0	9	2	83	88	99	0	15	-2	36	36	99	1	-10	4	68	87	99
0	2	-5	28	22	71	0	5	5	50	23	87	0	9	3	60	55	99	0	15	-1	13	24	99	1	-10	5	-19	31	99
0	2	-4	132	134	85	0	5	6	-7	21	99	0	9	4	216	212	99	0	15	0	106	101	99	1	-10	6	19	31	99
0	2	-3	-23	11	47	0	5	7	57	55	99	0	9	5	134	134	99	0	15	1	69	52	99	1	-9	-5	53	42	99
0	2	-2	193	197	89	0	6	-7	58	70	99	0	10	-6	26	51	99	1	-15	-1	43	32	99	1	-9	-4	117	117	99
0	2	-1	446	463	99	0	6	-6	62	57	92	0	10	-5	26	15	96	1	-15	0	68	57	99	1	-9	-3	49	45	99
0	2	0	219	214	84	0	6	-5	104	111	99	0	10	-4	104	101	99	1	-15	1	63	48	99	1	-9	-2	20	38	97
0	2	1	89	86	57	0	6	-4	201	207	99	0	10	-3	173	174	99	1	-15	2	-35	12	99	1	-9	-1	76	76	99
0	2	2	124	115	72	0	6	-3	176	177	99	0	10	-2	92	89	98	1	-15	3	40	22	99	1	-9	0	315	296	99
0	2	3	277	280	99	0	6	-2	77	82	67	0	10	-1	95	85	97	1	-14	-2	19	50	99	1	-9	1	269	262	99
0	2	4	441	442	99	0	6	-1	86	86	65	0	10	0	106	113	99	1	-14	-1	79	74	99	1	-9	2	12	33	86
0	2	5	318	313	99	0	6	0	534	542	99	0	10	1	113	119	99	1	-14	0	103	101	99	1	-9	3	37	25	82
0	2	6	-32	6	92	0	6	1	391	397	99	0	10	2	-30	15	97	1	-14	1	11	26	99	1	-9	4	36	20	88
0	2	7	43	52	99	0	6	2	103	99	87	0	10	3	41	37	99	1	-14	2	45	26	99	1	-9	5	122	122	99
0	3	-7	45	38	98	0	6	3	35	22	82	0	10	4	169	168	99	1	-14	3	14	12	99	1	-9	6	51	67	99
0	3	-6	35	38	84	0	6	4	62	49	99	0	10	5	81	79	99	1	-14	4	24	40	99	1	-9	7	57	41	99
0	3	-5	82	83	86	0	6	5	44	35	99	0	11	-6	45	31	98	1	-13	-3	90	78	99	1	-8	-6	40	44	99
0	3	-4	256	253	99	0	6	6	-13	19	99	0	11	-5	26	19	98	1	-13	-2	73	45	99	1	-8	-5	33	7	89
0	3	-3	338	342	99	0	7	-7	74	70	99	0	11	-4	140	131	99	1	-13	-1	55	59	99	1	-8	-4	35	31	93
0	3	-2	127	132	66	0	7	-6	96	92	99	0	11	-3	132	122	99	1	-13	0	54	64	99	1	-8	-3	91	90	99
0	3	-1	214	211	94	0	7	-5	140	145	99	0	11	-2	35	36	90	1	-13	1	71	63	99	1	-8	-2	31	9	82
0	3	0	195	190	86	0	7	-4	81	76	83	0	11	-1	35	7	91	1	-13	2	38	17	98	1	-8	-1	-6	37	79
0	3	1	19	14	53	0	7	-3	-13	20	68	0	11	0	47	44	93	1	-13	3	-19	29	99	1	-8	0	378	373	99
0	3	2	42	35	58	0	7	-2	-18	13	61	0	11	1	141	132	99	1	-13	4	80	81	99	1	-8	1	285	282	99
0	3	3	260	260	99	0	7	-1	219	221	99	0	11	2	126	118	99	1	-13	5	41	28	99	1	-8	2	31	6	69
0	3	4	352	352	99	0	7	0	398	405	99	0	11	3	93	82	99	1	-12	-4	99	106	99	1	-8	3	31	38	84
0	3	5	173	172	99	0	7	1	292	286	99	0	11	4	44	68	99	1	-12	-3	121	118	99	1	-8	4	119	124	99
0	3	6	-24	17	93	0	7	2	29	35	78	0	12	-6	26	8	99	1	-12	-2	-18	32	99	1	-8	5	134	131	99
0	3	7	-17	12	94	0	7	3	49	41	90	0	12	-5	72	60	99	1	-12	-1	38	20	98	1	-8	6	69	58	99
0	4	-7	16	15	93	0	7	4	150	153	99	0	12	-4	96	94	99	1	-12	0	71	57	99	1	-8	7	57	47	99
0	4	-6	79	76	91	0	7	5	115	113	99	0	12	-3	47	34	96	1	-12	1	63	64	99	1	-7	-6	39	46	99
0	4	-5	167	169	99	0	7	6	59	54	99	0	12	-2	58	50	99	1	-12	2	44	55	99	1	-7	-5	34	32	96
0	4	-4	323	336	99	0	8	-7	67	78	99	0	12	-1	46	17	91	1	-12	3	81	94	99	1	-7	-4	31	18	83
1	-7	-3	112	106	99	1	-3	-7	36	52	99	1	0	3	182	177	99	1	4	-4	178	177	99	1	8	-6	35	7	92
1	-7	-2	136	134	99	1	-3	-6	25	11	94	1	0	4	216	221	99	1	4	-3	294	294	99	1	8	-5	72	64	94
1	-7	-1	119	123	94	1	-3	-5	35	12	81	1	0	5	354	353	99	1	4	-2	72	74	56	1	8	-4	26	7	83
1	-7	0	136	135	91	1	-3	-4	332	348	99	1	0	6	156	147	99	1	4	-1	315	308	99	1	8	-3	219	218	99
1	-7	1	264	270	99	1	-3	-3	371	372	99	1	0	7	39	30	99	1	4	0	305	304	99	1	8	-2	254	251	99
1	-7	2	-18	6	62	1	-3	-2	13	18	54	1	1	-7	147	152	99	1	4	1	615	621	99	1	8	-1	181	184	99
1	-7	3	64	68	83	1	-3	-1	107	109	67	1	1	-6	-43	10	91	1	4	2	419	415	99	1	8	0	61	67	76
1	-7	4	247	249	99	1	-3	0	613	593	99	1	1	-5	43	29	78	1	4	3	25	23	73	1	8	1	149	146	99
1	-7	5	224	233	99	1	-3	1	784	816	99	1	1	-4	156	156	99	1	4	4	-22	31	92	1	8	2	100	105	99
1	-7	6	42	49	99	1	-3	2	239	249	99	1	1	-3	469	477	99	1	4	5	58	46	99	1	8	3	32	25	95
1	-7	7	53	36	99	1	-3	3	67	61	55	1	1	-2	317	314	99	1	4	6	56	62	99	1	8	4	129	126	99
1	-6	-6	45	7	96	1	-3	4	37	35	60	1	1	-1	92	91	43	1	4	7	51	53	99	1	8	5	144	142	99
1	-6	-5	38	38	96	1	-3	5	49	45	76	1	1	0	319	319	99	1	5	-7	92	96	99	1	8	6	47	44	99
1	-6	-4	170	170	99	1	-3	6	-25	7	82	1	1	1	22	27	47	1	5	-6	120	129	99	1	9	-7	69	61	99
1	-6	-3	219	209	99	1	-3	7	49	39	87	1	1	2	66	58	59	1	5	-5	139	124	99	1	9	-6	16	6	99
1	-6	-2	134	134	99	1	-2	-7	100	97	99	1	1	3	30	25	65	1	5	-4	133	125	99	1	9	-5	79	74	99
1	-6	-1	73	78	73	1	-2	-6	128	138	99	1	1	4	272	269	99	1	5	-3	161	166	98	1	9	-4	124	117	99
1	-6	0	42	10	55	1	-2	-5	31	45	88	1	1	5	273	269	99	1	5	-2	35	8	47	1	9	-3	202	194	99
1	-6	1	51	52	62	1	-2	-4	11	15	70	1	1	6	87	80	99	1	5	-1	93	88	60	1	9	-2	115	118	99
1	-6	2	106	97	78	1	-2	-3	200	194	99	1	1	7	-40	13	99	1	5	0	275	281	99	1	9	-1	30	21	76
1	-6	3	119	127	90	1	-2	-2	119	117	68	1	2	-8	101	101	99	1	5	1	480	494	99	1	9	0	-11	12	74
1	-6	4	116	123	99	1	-2	-1	217	214	99	1	2	-7	89	97	99	1	5	2	183	174	99	1	9	1	147	146	99
1	-6	5	228	237	99	1	-2	0	844	877	99	1	2	-6	56	62	98	1	5	3	80	80	89	1	9	2	197	204	99
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7	1	-4	14	21	99
7	1	-3	106	105	99
7	1	-2	150	148	99
7	1	-1	25	43	99
7	1	0	97	100	99
7	1	1	39	17	98
7	1	2	137	139	99
7	1	3	117	114	99
7	2	-5	122	115	99
7	2	-4	83	83	99
7	2	-3	91	98	99
7	2	-2	105	105	99
7	2	-1	32	11	99
7	2	0	54	49	98
7	2	1	32	39	99
7	2	2	120	117	99
7	2	3	111	100	99
7	3	-5	131	129	99
7	3	-4	44	54	99
7	3	-3	48	38	99
7	3	-2	104	109	99
7	3	-1	56	48	99
7	3	0	47	31	99
7	3	1	109	99	99
7	3	2	111	107	99
7	3	3	-26	18	99
7	4	-4	17	9	99
7	4	-3	11	24	99
7	4	-2	84	84	99
7	4	-1	140	143	99
7	4	0	35	70	99
7	4	1	77	93	99
7	4	2	124	119	99
7	4	3	-22	10	99
7	5	-4	33	44	99
7	5	-3	-10	26	99
7	5	-2	110	115	99
7	5	-1	178	169	99
7	5	0	84	79	99
7	5	1	32	29	99
7	5	2	42	54	99
7	6	-4	47	35	99
7	6	-3	-22	6	99
7	6	-2	162	164	99
7	6	-1	144	150	99
7	6	0	42	32	99
7	6	1	29	17	99
7	6	2	36	34	99
7	6	3	30	34	99
7	7	-2	136	138	99
7	7	-1	119	124	99
7	7	0	38	19	99

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
7	7	1	54	50	99	8	3	-2	50	9	99																		
7	8	-3	96	114	99	8	3	-1	104	103	99																		
7	8	-2	130	132	99	8	3	0	143	145	99																		
7	8	-1	38	42	99	8	3	1	47	53	99																		
7	8	0	-20	13	99	8	4	-3	31	23	99																		
7	8	1	-31	13	99	8	4	-2	76	75	99																		
7	9	-1	34	23	99	8	4	-1	136	138	99																		
8	-6	-1	20	57	99	8	4	0	86	90	99																		
8	-6	0	30	36	99	8	4	1	52	10	99																		
8	-5	-2	29	17	99	8	5	-2	61	83	99																		
8	-5	-1	84	79	99	8	5	-1	132	143	99																		
8	-5	0	86	94	99	8	5	0	57	63	99																		
8	-5	1	78	62	99																								
8	-4	-2	55	26	99																								
8	-4	-1	85	101	99																								
8	-4	0	100	100	99																								
8	-4	1	55	62	99																								
8	-4	2	62	51	99																								
8	-3	-3	-25	50	99																								
8	-3	-2	70	64	99																								
8	-3	-1	119	120	99																								
8	-3	0	86	74	99																								
8	-3	1	-40	22	99																								
8	-3	2	32	20	99																								
8	-2	-3	19	21	99																								
8	-2	-2	74	79	99																								
8	-2	-1	116	107	99																								
8	-2	0	16	17	99																								
8	-2	1	8	33	99																								
8	-2	2	31	23	99																								
8	-1	-3	62	51	99																								
8	-1	-2	82	78	99																								
8	-1	-1	95	97	99																								
8	-1	0	19	12	99																								
8	-1	1	50	67	99																								
8	-1	2	19	26	99																								
8	0	-3	57	68	99																								
8	0	-2	37	67	99																								
8	0	-1	57	51	99																								
8	0	0	-7	14	99																								
8	0	1	-22	4	99																								
8	0	2	57	31	99																								
8	1	-3	-22	19	99																								
8	1	-2	60	51	99																								
8	1	-1	75	67	99																								
8	1	0	53	31	99																								
8	1	1	51	45	99																								
8	1	2	92	89	99																								
8	2	-3	12	40	99																								
8	2	-2	53	26	99																								
8	2	-1	124	126	99																								
8	2	0	117	113	99																								
8	2	1	25	36	99																								
8	2	2	59	67	99																								
8	3	-3	43	34	99																								

APPENDIX 3.VIII

$[Ni(S_2COHex)_2]$

Table 3.VIII.1: Crystallographic Parameters for $[Ni(S_2COCH_2CH_2CH_2CH_2CH_2CH_3)_2]$

Formula	$C_{14}H_{26}NiO_2S_4$
Formula weight	413.30
Crystal morphology / colour	block / brown
Crystal dimensions (mm)	0.05 x 0.19 x 0.35
Crystal system	triclinic
Space group	$P\bar{1}$ (#2)
a (Å)	7.566(2)
b (Å)	10.713(4)
c (Å)	6.779(2)
α (°)	97.73(3)
β (°)	111.22(2)
γ (°)	103.19(2)
V (Å^3)	484.2(3)
Z	1
$\rho_{calc.}$ (g cm^{-3})	1.417
$F(000)$	218
Temperature (°K)	200
μ (cm^{-1})	14.21
θ limits, cell (°)	27.1 – 27.5
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 9, -13 – 12, -8 – 7
Range of transmission factors	0.906 – 1
Scan technique	ω:2θ
No. of data measured	2416
No. of unique data	2245
R_{amal}	0.078
No. of parameters	97
R (I≥0σ(I))	0.091
R_w (I≥0σ(I))	0.121
R (I≥3σ(I))	0.049
R_w (I≥3σ(I))	0.058
Maximum and minimum residual electron density (e Å^{-3})	0.75, -0.95

Table 3.VIII.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Ni(S_2COCH_2CH_2CH_2CH_2CH_2CH_3)_2]$

Atom	*x*	*y*	*z*
Ni	0	0	0
S(1)	-0.2605(1)	-0.1730(1)	-0.1946(1)
S(2)	-0.1706(1)	-0.00024(9)	0.2057(1)
O(1)	-0.4945(4)	-0.2108(2)	0.0085(4)
C(1)	-0.3308(5)	-0.1361(3)	0.0112(5)
C(2)	-0.5514(5)	-0.1766(4)	0.1887(5)
C(3)	-0.7437(5)	-0.2789(4)	0.1393(6)
C(4)	-0.8252(5)	-0.2558(3)	0.3105(6)
C(5)	-1.0173(5)	-0.3610(4)	0.2589(6)
C(6)	-1.1080(5)	-0.3433(4)	0.4220(6)
C(7)	-1.3020(6)	-0.4489(4)	0.3627(7)

Table 3.VIII.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Ni(S_2CO(CH_2CH_2CH_2CH_2CH_2CH_3)2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	0.0325(3)	0.0350(4)	0.0324(3)	0.0102(3)	0.0112(3)	0.0113(3)
S(1)	0.0416(5)	0.0452(6)	0.0368(5)	0.0055(4)	0.0165(4)	0.0026(4)
S(2)	0.0388(5)	0.0379(6)	0.0362(5)	0.0066(4)	0.0148(4)	0.0063(4)
O(1)	0.038(1)	0.041(2)	0.041(1)	0.006(1)	0.018(1)	0.006(1)
C(1)	0.036(2)	0.035(2)	0.035(2)	0.016(2)	0.012(1)	0.014(2)
C(2)	0.039(2)	0.042(2)	0.037(2)	0.014(2)	0.016(2)	0.012(2)
C(3)	0.039(2)	0.037(2)	0.045(2)	0.011(2)	0.018(2)	0.011(2)
C(4)	0.039(2)	0.033(2)	0.042(2)	0.012(2)	0.016(2)	0.011(2)
C(5)	0.037(2)	0.036(2)	0.041(2)	0.012(2)	0.014(2)	0.012(2)
C(6)	0.044(2)	0.038(2)	0.052(2)	0.012(2)	0.023(2)	0.015(2)
C(7)	0.051(2)	0.047(3)	0.075(3)	0.008(2)	0.034(2)	0.012(2)

Table 3.VIII.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Ni(S_2CO(CH_2CH_2CH_2CH_2CH_2CH_3)_2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	-0.5692	-0.0895	0.1979	3.6457
H(2b)	-0.4502	-0.1783	0.3252	3.6457
H(3a)	-0.7220	-0.3649	0.1309	3.8186
H(3b)	-0.8409	-0.2773	-0.0002	3.8186
H(4a)	-0.8492	-0.1705	0.3176	3.5674
H(4b)	-0.7278	-0.2563	0.4505	3.5674
H(5a)	-0.9914	-0.4457	0.2526	3.6383
H(5b)	-1.1128	-0.3606	0.1176	3.6383
H(6a)	-1.1321	-0.2581	0.4308	4.1090
H(6b)	-1.0144	-0.3457	0.5631	4.1090
H(7a)	-1.3975	-0.4474	0.2222	5.3755
H(7b)	-1.3532	-0.4322	0.4726	5.3755
H(7c)	-1.2798	-0.5348	0.3554	5.3755

* H atoms were included in their calculated positions, C-H 0.97Å.

Table 3.VIII.5: Bond distances (Å) for [Ni($S_2COCH_2CH_2CH_2CH_2CH_2CH_3$)2]

Ni — S(1)	2.204(1)	C(2) — C(3)	1.500(5)
Ni — S(2)	2.2168(8)	C(3) — C(4)	1.516(4)
S(1) — C(1)	1.694(3)	C(4) — C(5)	1.511(5)
S(2) — C(1)	1.688(4)	C(5) — C(6)	1.511(5)
O(1) — C(1)	1.304(4)	C(6) — C(7)	1.513(5)
O(1) — C(2)	1.464(4)		

Table 3.VIII.6: Bond angles (deg.) for [Ni($S_2COCH_2CH_2CH_2CH_2CH_2CH_3$)2]

S(1) — Ni — S(1)*	180(-)	S(1) — C(1) — O(1)	120.0(3)
S(1) — Ni — S(2)*	100.48(4)	S(2) — C(1) — O(1)	126.6(2)
S(1) — Ni — S(2)	79.52(4)	O(1) — C(2) — C(3)	106.3(3)
S(2) — Ni — S(2)*	180(-)	C(2) — C(3) — C(4)	112.9(3)
Ni — S(1) — C(1)	83.6(1)	C(3) — C(4) — C(5)	111.9(3)
Ni — S(2) — C(1)	83.4(1)	C(4) — C(5) — C(6)	114.7(3)
C(1) — O(1) — C(2)	118.8(3)	C(5) — C(6) — C(7)	113.2(3)
S(1) — C(1) — S(2)	113.4(2)		

* atom related by a centre of symmetry

Table 3.VIII.7: Observed and calculated structure factors for $[Ni(S_2COCH_2CH_2CH_2CH_2CH_3)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	1	574	556	99	0	4	-7	-28	8	53	0	7	3	56	36	50	0	12	2	59	47	93	1	-8	-4	240	220	99
0	0	2	121	124	64	0	4	-6	90	96	44	0	7	4	102	91	83	0	13	-4	18	40	95	1	-8	-3	100	88	64
0	0	3	28	25	15	0	4	-5	130	135	79	0	7	5	121	99	99	0	13	-3	20	14	99	1	-8	-2	116	116	76
0	0	4	265	277	99	0	4	-4	251	253	99	0	8	-7	43	47	38	0	13	-2	23	36	99	1	-8	-1	18	10	65
0	0	5	358	361	99	0	4	-3	174	176	99	0	8	-6	67	77	46	0	13	-1	75	80	98	1	-8	0	238	240	99
0	0	6	157	160	99	0	4	-2	36	34	15	0	8	-5	100	103	55	0	13	0	50	48	91	1	-8	1	313	318	99
0	0	7	-21	13	69	0	4	-1	102	114	59	0	8	-4	101	103	55	1	-13	-1	27	11	99	1	-8	2	123	117	79
0	1	-8	35	31	26	0	4	0	297	332	99	0	8	-3	96	101	54	1	-13	0	69	53	99	1	-8	3	46	41	38
0	1	-7	13	9	51	0	4	1	318	341	99	0	8	-2	54	57	37	1	-13	1	118	116	99	1	-8	4	21	15	72
0	1	-6	186	201	99	0	4	2	167	171	99	0	8	-1	162	180	99	1	-13	2	-3	36	99	1	-8	5	183	178	99
0	1	-5	252	261	99	0	4	3	52	50	34	0	8	0	140	151	99	1	-13	3	62	66	99	1	-8	6	157	153	99
0	1	-4	154	157	99	0	4	4	11	8	37	0	8	1	-36	47	99	1	-13	4	32	7	99	1	-8	7	29	27	99
0	1	-3	101	101	47	0	4	5	101	85	70	0	8	2	30	51	99	1	-12	-2	-23	14	99	1	-7	-6	43	33	45
0	1	-2	173	159	99	0	4	6	108	85	82	0	8	3	97	89	81	1	-12	-1	30	50	99	1	-7	-5	117	110	78
0	1	-1	405	375	99	0	4	7	44	22	60	0	8	4	133	112	99	1	-12	0	88	90	97	1	-7	-4	109	105	67
0	1	1	782	825	99	0	5	-8	10	15	38	0	8	5	20	16	78	1	-12	1	-47	31	99	1	-7	-3	80	83	52
0	1	2	86	85	39	0	5	-7	44	51	32	0	9	-7	-26	28	86	1	-12	2	77	75	93	1	-7	-2	15	7	55
0	1	3	83	85	38	0	5	-6	90	91	45	0	9	-6	38	34	38	1	-12	3	18	53	99	1	-7	-1	104	108	62
0	1	4	363	341	99	0	5	-5	328	334	99	0	9	-5	78	89	52	1	-12	4	21	25	99	1	-7	0	324	332	99
0	1	5	278	273	99	0	5	-4	271	274	99	0	9	-4	110	113	67	1	-12	5	73	66	88	1	-7	1	300	301	99
0	1	6	64	63	40	0	5	-3	51	49	21	0	9	-3	145	157	99	1	-11	-3	82	72	86	1	-7	2	61	60	35
0	1	7	19	27	75	0	5	-2	61	63	23	0	9	-2	125	144	86	1	-11	-2	81	70	79	1	-7	3	32	25	30
0	2	-8	24	29	25	0	5	-1	175	192	99	0	9	-1	-24	8	90	1	-11	-1	59	50	67	1	-7	4	86	85	49
0	2	-7	-7	15	48	0	5	0	459	504	99	0	9	0	116	115	80	1	-11	0	80	73	75	1	-7	5	171	165	99
0	2	-6	42	48	28	0	5	1	364	387	99	0	9	1	115	117	85	1	-11	1	88	88	84	1	-7	6	169	175	99
0	2	-5	132	136	80	0	5	2	18	7	53	0	9	2	77	77	77	1	-11	2	-16	21	99	1	-7	7	41	35	48
0	2	-4	33	37	19	0	5	3	43	38	37	0	9	3	88	70	79	1	-11	3	103	99	87	1	-6	-6	16	29	82
0	2	-3	58	56	20	0	5	4	157	133	99	0	9	4	69	60	83	1	-11	4	97	95	89	1	-6	-5	73	65	48
0	2	-2	90	84	45	0	5	5	171	147	99	0	10	-6	-28	22	80	1	-11	5	56	58	71	1	-6	-4	65	57	40
0	2	-1	434	421	99	0	5	6	118	91	92	0	10	-5	89	93	62	1	-11	6	51	49	74	1	-6	-3	-19	9	56
0	2	0	13	17	10	0	6	-8	-20	10	57	0	10	-4	120	125	77	1	-10	-4	37	35	99	1	-6	-2	67	72	34
0	2	1	198	210	99	0	6	-7	-13	4	64	0	10	-3	16	8	75	1	-10	-3	122	109	94	1	-6	-1	124	133	73
0	2	2	38	40	18	0	6	-6	86	94	47	0	10	-2	41	32	46	1	-10	-2	55	47	59	1	-6	0	244	249	99
0	2	3	137	137	99	0	6	-5	239	252	99	0	10	-1	117	119	85	1	-10	-1	23	13	94	1	-6	1	150	155	99
0	2	4	248	222	99	0	6	-4	183	187	99	0	10	0	76	79	70	1	-10	0	30	51	99	1	-6	2	71	69	32
0	2	5	128	111	84	0	6	-3	55	53	26	0	10	1	134	128	99	1	-10	1	157	153	99	1	-6	3	42	40	27
0	2	6	-11	5	70	0	6	-2	-8	33	42	0	10	2	45	40	68	1	-10	2	142	137	99	1	-6	4	128	127	85
0	2	7	32	14	44	0	6	-1	246	281	99	0	10	3	15	13	99	1	-10	3	21	11	55	1	-6	5	122	115	76
0	3	-8	46	54	32	0	6	0	412	450	99	0	11	-6	27	26	98	1	-10	4	-30	43	99	1	-6	6	28	30	38
0	3	-7	60	64	31	0	6	1	176	184	99	0	11	-5	88	105	71	1	-10	5	55	67	67	1	-6	7	50	48	46
0	3	-6	53	49	27	0	6	2	71	70	44	0	11	-4	71	76	57	1	-10	6	75	79	70	1	-5	-7	36	36	42
0	3	-5	14	26	46	0	6	3	98	94	64	0	11	-3	30	27	99	1	-9	-5	78	74	76	1	-5	-6	-30	4	67
0	3	-4	153	155	99	0	6	4	160	139	99	0	11	-2	114	123	85	1	-9	-4	145	133	99	1	-5	-5	14	10	42
0	3	-3	119	116	71	0	6	5	258	217	99	0	11	-1	56	68	72	1	-9	-3	50	52	58	1	-5	-4	84	79	43
0	3	-2	156	148	95	0	6	6	56	46	75	0	11	0	190	190	99	1	-9	-2	26	22	97	1	-5	-3	132	131	82
0	3	-1	36	39	13	0	7	-7	34	29	33	0	11	1	69	60	84	1	-9	-1	47	57	51	1	-5	-2	98	94	52
0	3	0	137	151	82	0	7	-6	120	126	74	0	11	2	32	24	99	1	-9	0	126	125	92	1	-5	-1	51	44	23
0	3	1	50	53	21	0	7	-5	148	153	99	0	11	3	56	45	86	1	-9	1	233	237	99	1	-5	0	70	67	28
0	3	2	75	83	31	0	7	-4	76	77	39	0	12	-5	77	85	76	1	-9	2	174	166	99	1	-5	1	154	154	99
0	3	3	71	65	36	0	7	-3	45	52	29	0	12	-4	38	51	60	1	-9	3	-28	6	99	1	-5	2	106	109	54
0	3	4	78	71	47	0	7	-2	65	67	33	0	12	-3	29	32	99	1	-9	4	-18	24	99	1	-5	3	105	105	59
0	3	5	69	48	49	0	7	-1	251	274	99	0	12	-2	27	24	95	1	-9	5	89	89	66	1	-5	4	63	59	33
0	3	6	-27	9	99	0	7	0	239	259	99	0	12	-1	9	16	99	1	-9	6	106	103	78	1	-5	5	74	76	40
0	3	7	-51	23	99	0	7	1	75	74	46	0	12	0	97	110	99	1	-9	7	70	65	61	1	-5	6	31	18	33
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7	-5	-4	84	85	39	7	0	-6	47	47	20	7	6	-6	-23	13	59	8	-4	-1	107	110	60	8	2	-2	99	99	51
7	-5	-3	27	14	23	7	0	-5	105	98	54	7	6	-5	75	77	43	8	-4	0	14	15	62	8	2	-1	-17	14	59
7	-5	-2	24	41	60	7	0	-4	157	152	99	7	6	-4	81	84	46	8	-4	1	27	40	47	8	2	0	26	25	33
7	-5	-1	22	6	57	7	0	-3	76	74	34	7	6	-3	79	70	44	8	-4	2	91	91	66	8	3	-6	28	26	19
7	-5	0	129	130	89	7	0	-2	131	126	79	7	6	-2	17	26	74	8	-3	-7	52	60	20	8	3	-5	17	25	21
7	-5	1	107	114	72	7	0	-1	53	58	31	7	6	-1	23	6	65	8	-3	-6	36	37	17	8	3	-4	51	51	28
7	-5	2	38	18	45	7	0	0	122	126	74	7	7	-4	89	86	51	8	-3	-5	31	31	19	8	3	-3	148	142	99
7	-5	3	67	64	64	7	0	1	127	130	86	7	7	-3	-21	14	73	8	-3	-4	53	55	26	8	3	-2	63	62	37
7	-4	-7	-4	12	23	7	0	2	40	45	41	8	-10	-3	71	67	75	8	-3	-3	146	150	99	8	3	-1	28	27	30
7	-4	-6	47	48	21	7	1	-8	29	30	16	8	-10	-2	69	64	82	8	-3	-2	47	43	30	8	4	-6	1	15	32
7	-4	-5	98	110	53	7	1	-7	28	29	17	8	-10	-1	-21	25	99	8	-3	-1	-25	26	63	8	4	-5	53	51	29
7	-4	-4	106	116	61	7	1	-6	28	28	18	8	-10	0	-30	4	99	8	-3	0	18	26	74	8	4	-4	104	104	57
7	-4	-3	35	39	24	7	1	-5	120	117	68	8	-9	-4	48	37	48	8	-3	1	51	56	43	8	4	-3	57	61	33
7	-4	-2	21	12	48	7	1	-4	152	147	99	8	-9	-3	95	91	75	8	-3	2	78	86	65	8	4	-2	41	33	29
7	-4	-1	88	88	46	7	1	-3	29	12	21	8	-9	-2	82	61	70	8	-2	-7	6	3	27	8	4	-1	11	19	65
7	-4	0	116	118	73	7	1	-2	55	55	30	8	-9	-1	32	5	99	8	-2	-6	18	35	36	8	5	-5	23	34	55
7	-4	1	101	103	63	7	1	-1	-35	13	57	8	-9	0	-20	10	99	8	-2	-5	5	7	31	8	5	-4	115	107	76
7	-4	2	14	20	62	7	1	0	108	111	61	8	-9	1	21	38	99	8	-2	-4	117	116	67	8	5	-3	62	68	39
7	-4	3	-40	4	99	7	1	1	93	98	56	8	-8	-5	19	11	55	8	-2	-3	109	106	59	9	-8	-3	50	53	42
7	-3	-7	53	61	23	7	1	2	-14	8	52	8	-8	-4	67	65	43	8	-2	-2	15	12	48	9	-8	-2	79	77	57
7	-3	-6	51	58	22	7	2	-7	-4	7	31	8	-8	-3	94	85	68	8	-2	-1	92	95	49	9	-8	-1	-16	16	98
7	-3	-5	54	56	23	7	2	-6	57	58	25	8	-8	-2	39	35	54	8	-2	0	13	9	44	9	-7	-4	20	12	25
7	-3	-4	128	135	93	7	2	-5	104	100	57	8	-8	-1	16	34	94	8	-2	1	94	98	58	9	-7	-3	28	34	35
7	-3	-3	180	187	99	7	2	-4	92	89	45	8	-8	0	0	7	99	8	-1	-7	34	34	16	9	-7	-2	91	99	59
7	-3	-2	38	45	28	7	2	-3	57	54	28	8	-8	1	58	61	85	8	-1	-6	-24	6	30	9	-7	-1	115	120	77
7	-3	-1	76	75	39	7	2	-2	33	34	25	8	-7	-5	7	28	33	8	-1	-5	61	60	25	9	-6	-5	-18	5	43
7	-3	0	105	110	59	7	2	-1	76	74	40	8	-7	-4	78	82	42	8	-1	-4	102	103	57	9	-6	-4	29	23	22
7	-3	1	80	86	52	7	2	0	102	105	59	8	-7	-3	40	33	33	8	-1	-3	94	93	47	9	-6	-3	44	56	34
7	-3	2	80	76	56	7	2	1	69	71	46	8	-7	-2	-27	11	71	8	-1	-2	-26	5	51	9	-6	-2	77	82	46
7	-3	3	59	53	54	7	2	2	27	8	73	8	-7	-1	-42	4	92	8	-1	-1	16	10	54	9	-6	-1	66	74	49
7	-2	-7	57	63	22	7	3	-7	85	82	39	8	-7	0	32	29	99	8	-1	0	21	29	65	9	-6	0	53	50	49

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
9	-5	-5	40	41	20																								
9	-5	-4	-20	3	41																								
9	-5	-3	61	64	35																								
9	-5	-2	86	88	49																								
9	-5	-1	21	9	64																								
9	-5	0	32	29	40																								
9	-4	-5	15	13	35																								
9	-4	-4	14	26	22																								
9	-4	-3	87	91	42																								
9	-4	-2	50	54	34																								
9	-4	-1	-4	8	71																								
9	-4	0	43	45	40																								
9	-3	-6	7	17	30																								
9	-3	-5	14	18	24																								
9	-3	-4	56	59	27																								
9	-3	-3	69	67	34																								
9	-3	-2	50	53	31																								
9	-3	-1	-20	14	60																								
9	-3	0	33	18	31																								
9	-2	-6	87	90	41																								
9	-2	-5	15	25	33																								
9	-2	-4	15	30	42																								
9	-2	-3	40	47	26																								
9	-2	-2	61	63	33																								
9	-2	-1	59	59	37																								
9	-2	0	-3	39	72																								
9	-1	-6	77	82	33																								
9	-1	-5	39	37	19																								
9	-1	-4	20	18	19																								
9	-1	-3	38	38	25																								
9	-1	-2	92	91	46																								
9	-1	-1	75	78	40																								
9	0	-6	38	40	18																								
9	0	-5	28	26	18																								
9	0	-4	28	27	20																								
9	0	-3	68	66	33																								
9	0	-2	104	110	62																								
9	0	-1	46	52	36																								
9	1	-5	13	22	41																								
9	1	-4	14	12	29																								
9	1	-3	65	67	35																								
9	1	-2	101	101	57																								
9	2	-5	-18	3	37																								
9	2	-4	75	74	36																								
9	2	-3	96	97	52																								

APPENDIX 3.IX

$[Ni(S_2COCy)_2]$

Table 3.IX.1: Crystallographic Parameters for [Ni($S_2COCH(CH_2)5)2$]

Formula	$C_{14}H_{22}NiO_2S_4$
Formula weight	409.27
Crystal morphology / colour	block / brown
Crystal dimensions (mm)	0.08 x 0.19 x 0.24
Crystal system	triclinic
Space group	$P\bar{1}$ (#2)
a (Å)	9.89(1)
b (Å)	10.102(7)
c (Å)	9.850(7)
α (°)	110.83(5)
β (°)	107.19(7)
γ (°)	87.28(9)
V (Å^3)	876(1)
Z	2
$\rho_{calc.}$ (g cm^{-3})	1.550
$F(000)$	428
Temperature (°K)	200
μ (cm^{-1})	15.69
θ limits, cell (°)	26.6 – 27.5
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 12, -13 – 13, -12 – 11
Range of transmission factors	0.938 – 1
Scan technique	ω:2θ
No. of data measured	4259
No. of unique data	4025
R_{amal}	0.036
No. of parameters	190
R (I≥0σ(I))	0.058
R_w (I≥0σ(I))	0.096
R (I≥3σ(I))	0.035
R_w (I≥3σ(I))	0.045
Maximum and minimum residual electron density (e Å^{-3})	0.56, -0.68

Table 3.IX.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Ni(S_2COCH(CH_2)5)2]$

Atom	*x*	*y*	*z*
Ni	0.14562(4)	0.53707(4)	0.18615(4)
S(1)	0.14166(8)	0.55710(7)	0.41576(8)
S(2)	0.12374(8)	0.76902(7)	0.28682(7)
S(3)	0.17611(8)	0.52098(7)	-0.03263(7)
S(4)	0.20004(8)	0.31290(7)	0.10125(8)
O(1)	0.1422(2)	0.8365(2)	0.5733(2)
O(2)	0.2631(2)	0.2591(2)	-0.1513(2)
C(1)	0.1369(3)	0.7332(3)	0.4450(3)
C(2)	0.1826(3)	0.8079(3)	0.7173(3)
C(3)	0.3429(3)	0.8203(3)	0.7781(3)
C(4)	0.3907(3)	0.8060(3)	0.9340(3)
C(5)	0.3229(3)	0.9123(3)	1.0442(3)
C(6)	0.1627(3)	0.9021(3)	0.9792(3)
C(7)	0.1154(3)	0.9171(3)	0.8234(3)
C(8)	0.2197(3)	0.3530(3)	-0.0447(3)
C(9)	0.2808(3)	0.2967(3)	-0.2784(3)
C(10)	0.2530(3)	0.1598(3)	-0.4146(3)
C(11)	0.2762(4)	0.1879(4)	-0.5484(3)
C(12)	0.4240(4)	0.2557(4)	-0.5074(4)
C(13)	0.4534(3)	0.3900(4)	-0.3661(4)
C(14)	0.4285(3)	0.3618(3)	-0.2332(3)

Table 3.IX.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Ni(S_2CO(CH(CH_2)5)2)2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	0.0273(2)	0.0270(2)	0.0284(2)	0.0002(1)	0.0079(1)	0.0082(1)
S(1)	0.0432(4)	0.0291(3)	0.0320(3)	0.0023(3)	0.0113(3)	0.0104(3)
S(2)	0.0396(4)	0.0298(3)	0.0307(3)	0.0024(3)	0.0079(3)	0.0112(3)
S(3)	0.0323(4)	0.0276(3)	0.0348(3)	0.0022(3)	0.0123(3)	0.0122(3)
S(4)	0.0390(4)	0.0300(3)	0.0350(3)	0.0050(3)	0.0144(3)	0.0139(3)
O(1)	0.043(1)	0.030(1)	0.0296(9)	0.0053(9)	0.0075(9)	0.0083(8)
O(2)	0.034(1)	0.033(1)	0.0333(9)	0.0071(8)	0.0154(8)	0.0129(8)
C(1)	0.022(1)	0.031(1)	0.036(1)	0.002(1)	0.006(1)	0.012(1)
C(2)	0.026(1)	0.033(1)	0.028(1)	0.001(1)	0.007(1)	0.010(1)
C(3)	0.024(1)	0.044(2)	0.042(1)	0.005(1)	0.014(1)	0.018(1)
C(4)	0.021(1)	0.047(2)	0.045(2)	0.005(1)	0.010(1)	0.023(1)
C(5)	0.029(2)	0.043(2)	0.035(1)	0.002(1)	0.004(1)	0.017(1)
C(6)	0.029(2)	0.044(2)	0.030(1)	0.007(1)	0.013(1)	0.012(1)
C(7)	0.024(1)	0.039(2)	0.034(1)	0.009(1)	0.005(1)	0.010(1)
C(8)	0.020(1)	0.029(1)	0.033(1)	0.000(1)	0.006(1)	0.010(1)
C(9)	0.024(1)	0.031(1)	0.031(1)	0.004(1)	0.008(1)	0.013(1)
C(10)	0.037(2)	0.036(2)	0.038(1)	-0.001(1)	0.012(1)	0.008(1)
C(11)	0.048(2)	0.052(2)	0.034(1)	-0.003(2)	0.014(1)	0.009(1)
C(12)	0.040(2)	0.077(3)	0.048(2)	0.005(2)	0.022(2)	0.025(2)
C(13)	0.024(2)	0.063(2)	0.051(2)	-0.006(1)	0.008(1)	0.027(2)
C(14)	0.024(1)	0.045(2)	0.040(1)	-0.001(1)	0.003(1)	0.016(1)

Table 3.IX.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Ni(S_2COCH(CH_2)_5)_2]$*

Atom	*x*	*y*	*z*	*B*eq
H(2a)	0.1469	0.7133	0.6985	2.7805
H(3a)	0.3811	0.7458	0.7083	3.3095
H(3b)	0.3778	0.9121	0.7871	3.3095
H(4a)	0.3638	0.7107	0.9229	3.3594
H(4b)	0.4929	0.8225	0.9747	3.3594
H(5a)	0.3477	0.8935	1.1383	3.4270
H(5b)	0.3588	1.0074	1.0649	3.4270
H(6a)	0.1244	0.9770	1.0486	3.2542
H(6b)	0.1261	0.8106	0.9692	3.2542
H(7a)	0.1446	1.0114	0.8337	3.2604
H(7b)	0.0130	0.9026	0.7825	3.2604
H(9a)	0.2119	0.3637	-0.3001	2.7158
H(10a)	0.1559	0.1233	-0.4416	3.5981
H(10b)	0.3174	0.0906	-0.3898	3.5981
H(11a)	0.2064	0.2513	-0.5777	4.3390
H(11b)	0.2647	0.0987	-0.6332	4.3390
H(12a)	0.4935	0.1884	-0.4885	4.9448
H(12b)	0.4318	0.2796	-0.5920	4.9448
H(13a)	0.3911	0.4613	-0.3887	4.2266
H(13b)	0.5513	0.4246	-0.3379	4.2266
H(14a)	0.4402	0.4507	-0.1478	3.5164
H(14b)	0.4967	0.2973	-0.2038	3.5164

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 3.IX.5: Bond distances (Å) for [Ni($S_2COCH(CH_2)_5)_2$]

Ni — S(1)	2.2099(7)	C(2) — C(3)	1.514(4)
Ni — S(2)	2.2241(8)	C(2) — C(7)	1.507(4)
Ni — S(3)	2.2116(7)	C(3) — C(4)	1.525(4)
Ni — S(4)	2.2182(8)	C(4) — C(5)	1.522(4)
S(1) — C(1)	1.699(3)	C(5) — C(6)	1.516(4)
S(2) — C(1)	1.689(3)	C(6) — C(7)	1.527(4)
S(3) — C(8)	1.702(3)	C(9) — C(10)	1.512(4)
S(4) — C(8)	1.690(3)	C(9) — C(14)	1.505(4)
O(1) — C(1)	1.310(3)	C(10) — C(11)	1.524(4)
O(1) — C(2)	1.484(3)	C(11) — C(12)	1.523(5)
O(2) — C(8)	1.306(3)	C(12) — C(13)	1.522(5)
O(2) — C(9)	1.488(3)	C(13) — C(14)	1.522(4)

Table 3.IX.6: Bond angles (deg.) for [Ni($S_2COCH(CH_2)_5)_2$]

S(1) — Ni — S(2)	79.22(3)	C(3) — C(2) — C(7)	112.2(2)
S(1) — Ni — S(3)	173.48(3)	C(2) — C(3) — C(4)	110.3(2)
S(1) — Ni — S(4)	99.98(3)	C(3) — C(4) — C(5)	111.4(2)
S(2) — Ni — S(3)	100.79(3)	C(4) — C(5) — C(6)	111.5(2)
S(2) — Ni — S(4)	171.92(3)	C(5) — C(6) — C(7)	112.1(2)
S(3) — Ni — S(4)	79.08(3)	C(2) — C(7) — C(6)	109.5(2)
Ni — S(1) — C(1)	83.78(9)	S(3) — C(8) — S(4)	112.5(1)
Ni — S(2) — C(1)	83.56(9)	S(3) — C(8) — O(2)	127.1(2)
Ni — S(3) — C(8)	84.09(9)	S(4) — C(8) — O(2)	120.4(2)
Ni — S(4) — C(8)	84.16(9)	O(2) — C(9) — C(10)	106.1(2)
C(1) — O(1) — C(2)	119.7(2)	O(2) — C(9) — C(14)	109.0(2)
C(8) — O(2) — C(9)	119.7(2)	C(10) — C(9) — C(14)	112.4(2)
S(1) — C(1) — S(2)	113.1(2)	C(9) — C(10) — C(11)	109.4(2)
S(1) — C(1) — O(1)	126.6(2)	C(10) — C(11) — C(12)	111.4(3)
S(2) — C(1) — O(1)	120.3(2)	C(11) — C(12) — C(13)	111.5(3)
O(1) — C(2) — C(3)	107.9(2)	C(12) — C(13) — C(14)	111.4(3)
O(1) — C(2) — C(7)	106.5(2)	C(9) — C(14) — C(13)	110.1(2)

Table 3.IX.7: Observed and calculated structure factors for $[Ni(S_2COCH(CH_2)_5)_2]$

h	k	l	Fo	Fc	sigF2
0	0	2	10	9	5
0	0	3	23	22	22
0	0	4	13	12	9
0	0	5	36	35	50
0	0	6	8	7	5
0	0	7	20	20	23
0	0	8	29	29	40
0	0	9	13	14	10
0	0	10	12	12	9
0	0	11	6	7	6
0	1	-9	6	8	6
0	1	-8	7	9	6
0	1	-7	4	5	4
0	1	-6	7	8	4
0	1	-5	25	29	28
0	1	-4	20	22	19
0	1	-3	54	54	99
0	1	-2	49	46	83
0	1	1	14	13	9
0	1	2	19	17	16
0	1	3	12	11	8
0	1	5	39	40	61
0	1	6	11	10	7
0	1	7	13	13	10
0	1	8	35	35	55
0	1	9	4	3	5
0	1	10	13	13	10
0	2	-11	5	8	5
0	2	-9	10	12	7
0	2	-8	9	10	6
0	2	-6	48	50	86
0	2	-5	90	90	99
0	2	-4	57	56	99
0	2	-3	20	19	18
0	2	-2	5	5	3
0	2	-1	112	116	99
0	2	0	24	24	22
0	2	1	18	18	15
0	2	2	24	24	24
0	2	4	35	35	48
0	2	5	59	58	99
0	2	6	10	9	7
0	2	7	25	25	32
0	2	8	21	21	22
0	3	-11	4	4	4
0	3	-9	26	30	34
0	3	-8	38	42	60
0	3	-7	9	10	7
0	3	-6	40	40	64
0	3	-5	55	54	99
0	3	-4	10	10	5
0	3	-3	30	28	35
0	3	-2	7	6	3
0	3	-1	5	5	3
0	3	0	75	75	99
0	3	1	29	30	34
0	3	3	33	33	43
0	3	4	43	44	72
0	3	5	41	42	67
0	3	6	9	9	6
0	3	7	13	13	9
0	3	8	11	11	8
0	4	-11	12	13	9
0	4	-9	42	45	74
0	4	-8	15	16	13
0	4	-7	6	7	5
0	4	-6	22	21	26
0	4	-5	43	41	70
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APPENDIX 3.X

$[Ni(S_2CNCy_2)_2]$

Table 3.X.1: Crystallographic Parameters for [Ni($S_2CN(CH(CH_2)_5)_2)_2$]

Formula	$C_{26}H_{44}N_2NiS_4$
Formula weight	571.59
Crystal morphology / colour	block / brown
Crystal dimensions (mm)	0.13 x 0.13 x 0.19
Crystal system	triclinic
Space group	$P\bar{1}$ (#2)
a (Å)	11.551(5)
b (Å)	14.651(7)
c (Å)	9.872(5)
α (°)	102.46(5)
β (°)	114.78(3)
γ (°)	89.65(4)
V (Å^3)	1474(1)
Z	2
$\rho_{calc.}$ (g cm^{-3})	1.287
F(000)	612
Temperature (°K)	200
μ (cm^{-1})	9.58
θ limits, cell (°)	20.6 – 22.4
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 15, -18 – 18, -12 – 12
Range of transmission factors	0.946 – 1
Scan technique	ω:2θ
No. of data measured	7115
No. of unique data	6780
R_{amal}	0.083
No. of parameters	301
R (I≥0σ(I))	0.113
R_w (I≥0σ(I))	0.133
R (I≥3σ(I))	0.055
R_w (I≥3σ(I))	0.058
Maximum and minimum residual electron density (e Å^{-3})	1.21, -1.91

Table 3.X.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Ni(S_2CN(CH(CH_2)5)2)2]$

Atom	*x*	*y*	*z*
Ni(1)	0	0.5	0.5
Ni(2)	-0.5	0	-0.5
S(1)	0.1535(1)	0.61360(8)	0.6277(1)
S(2)	0.0939(1)	0.49001(8)	0.3445(1)
S(3)	-0.3152(1)	-0.02838(9)	-0.3336(1)
S(4)	-0.4491(1)	-0.11355(8)	-0.6448(1)
N(1)	0.2761(3)	0.6363(2)	0.4513(3)
N(2)	-0.2100(3)	-0.1559(2)	-0.4849(5)
C(1)	0.1904(4)	0.5884(3)	0.4712(5)
C(2)	0.2951(4)	0.6058(3)	0.3085(4)
C(3)	0.2560(4)	0.6763(3)	0.2098(5)
C(4)	0.2687(4)	0.6385(3)	0.0600(5)
C(5)	0.4028(4)	0.6115(3)	0.0900(5)
C(6)	0.4419(4)	0.5423(3)	0.1915(5)
C(7)	0.4290(4)	0.5802(3)	0.3415(5)
C(8)	0.3558(4)	0.7220(3)	0.5631(5)
C(9)	0.4489(5)	0.6999(3)	0.7135(5)
C(10)	0.5327(6)	0.7898(5)	0.8246(7)
C(11)	0.4529(7)	0.8669(5)	0.8484(9)
C(12)	0.3642(6)	0.8873(3)	0.6988(8)
C(13)	0.2772(4)	0.8018(3)	0.5868(5)
C(14)	-0.3074(4)	-0.1076(3)	-0.4845(5)
C(15)	-0.2107(5)	-0.2106(3)	-0.6333(6)
C(16)	-0.1161(6)	-0.1699(4)	-0.6694(6)

Table 3.X.2: Cont.

Atom	*x*	*y*	*z*
C(17)	-0.1178(5)	-0.2216(4)	-0.8177(6)
C(18)	-0.1253(7)	-0.3266(5)	-0.8435(7)
C(19)	-0.2250(5)	-0.3626(4)	-0.8078(7)
C(20)	-0.2216(5)	-0.3151(3)	-0.6519(6)
C(21)	-0.0984(5)	-0.1622(4)	-0.3488(6)
C(22)	-0.1260(6)	-0.2140(4)	-0.2483(6)
C(23)	-0.0075(6)	-0.2293(4)	-0.1243(7)
C(24)	0.0752(5)	-0.1455(4)	-0.0307(6)
C(25)	0.1130(6)	-0.0863(5)	-0.1252(7)
C(26)	-0.0113(6)	-0.0739(3)	-0.2645(6)

Table 3.X.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Ni(S_2CN(CH(CH_2)_5)_2)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni(1)	0.0613(6)	0.0402(4)	0.0492(5)	-0.0066(4)	0.0324(5)	0.0100(4)
Ni(2)	0.0369(5)	0.0414(4)	0.0527(5)	0.0143(4)	0.0151(4)	0.0118(4)
S(1)	0.0708(9)	0.0524(7)	0.0461(7)	-0.0133(6)	0.0351(7)	0.0047(5)
S(2)	0.0670(8)	0.0458(6)	0.0498(7)	-0.0128(6)	0.0335(7)	0.0028(5)
S(3)	0.0444(7)	0.0781(9)	0.0533(8)	0.0238(6)	0.0160(6)	0.0168(6)
S(4)	0.0430(7)	0.0478(6)	0.0648(8)	0.0121(5)	0.0185(6)	0.0050(6)
N(1)	0.045(2)	0.036(2)	0.035(2)	-0.003(2)	0.018(2)	0.009(1)
N(2)	0.039(2)	0.054(2)	0.074(3)	0.017(2)	0.026(2)	0.034(2)
C(1)	0.050(3)	0.037(2)	0.045(3)	0.001(2)	0.022(2)	0.012(2)
C(2)	0.047(3)	0.040(2)	0.036(2)	-0.003(2)	0.020(2)	0.008(2)
C(3)	0.040(3)	0.072(3)	0.041(3)	0.019(2)	0.019(2)	0.025(2)
C(4)	0.054(3)	0.086(3)	0.043(3)	0.020(3)	0.022(3)	0.029(3)
C(5)	0.051(3)	0.057(3)	0.046(3)	0.011(2)	0.027(2)	0.014(2)
C(6)	0.054(3)	0.052(3)	0.054(3)	0.013(2)	0.025(3)	0.013(2)
C(7)	0.059(3)	0.056(3)	0.051(3)	0.025(2)	0.026(3)	0.026(2)
C(8)	0.040(2)	0.038(2)	0.042(3)	-0.001(2)	0.017(2)	0.009(2)
C(9)	0.049(3)	0.075(3)	0.048(3)	0.008(2)	0.013(3)	0.021(3)
C(10)	0.057(4)	0.129(6)	0.069(4)	-0.014(4)	0.000(3)	0.008(4)
C(11)	0.099(6)	0.101(5)	0.105(6)	-0.028(4)	0.036(5)	-0.043(4)
C(12)	0.100(5)	0.043(3)	0.138(6)	-0.010(3)	0.082(5)	-0.004(3)
C(13)	0.054(3)	0.042(2)	0.064(3)	0.001(2)	0.033(3)	0.012(2)
C(14)	0.035(2)	0.044(2)	0.071(3)	0.013(2)	0.024(2)	0.025(2)
C(15)	0.065(3)	0.064(3)	0.097(4)	0.022(3)	0.055(3)	0.013(3)
C(16)	0.101(5)	0.072(4)	0.055(3)	-0.016(3)	0.031(3)	0.016(3)

Table 3.X.3: Cont.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(17)	0.063(4)	0.085(4)	0.071(4)	0.011(3)	0.024(3)	0.013(3)
C(18)	0.118(6)	0.115(5)	0.082(5)	0.015(4)	0.055(4)	0.004(4)
C(19)	0.056(4)	0.086(4)	0.106(5)	0.004(3)	0.039(4)	-0.025(3)
C(20)	0.059(3)	0.058(3)	0.068(4)	-0.012(3)	-0.003(3)	0.027(3)
C(21)	0.050(3)	0.086(4)	0.083(4)	0.016(3)	0.030(3)	0.045(3)
C(22)	0.108(5)	0.069(3)	0.057(3)	0.028(3)	0.036(4)	0.024(3)
C(23)	0.086(5)	0.095(4)	0.068(4)	0.033(4)	0.035(4)	0.011(4)
C(24)	0.080(4)	0.091(4)	0.040(3)	0.028(3)	0.010(3)	0.007(3)
C(25)	0.073(4)	0.126(5)	0.069(4)	0.029(4)	0.032(4)	-0.002(4)
C(26)	0.107(5)	0.054(3)	0.041(3)	0.027(3)	0.002(3)	0.002(2)

Table 3.X.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Ni(S_2CN(CH(CH_2)_5)_2)_2]$*

Atom	*x*	*y*	*z*	*B*eq
H(2a)	0.2384	0.5489	0.2492	3.8106
H(3a)	0.1677	0.6881	0.1863	4.6302
H(3b)	0.3108	0.7345	0.2650	4.6302
H(4a)	0.2081	0.5835	0.0010	5.5468
H(4b)	0.2495	0.6866	0.0018	5.5468
H(5a)	0.4626	0.6676	0.1402	4.6384
H(5b)	0.4051	0.5832	-0.0071	4.6384
H(6a)	0.5303	0.5308	0.2156	5.0220
H(6b)	0.3874	0.4840	0.1370	5.0220
H(7a)	0.4889	0.6355	0.4006	4.9340
H(7b)	0.4483	0.5323	0.4001	4.9340
H(8a)	0.4080	0.7434	0.5184	3.8076
H(9a)	0.5034	0.6536	0.6928	5.6155
H(9b)	0.4009	0.6750	0.7602	5.6155
H(10a)	0.5855	0.7759	0.9227	9.2105
H(10b)	0.5873	0.8106	0.7823	9.2105
H(11a)	0.4028	0.8480	0.8975	10.9484
H(11b)	0.5087	0.9232	0.9139	10.9484
H(12a)	0.4150	0.9096	0.6532	8.2462
H(12b)	0.3118	0.9357	0.7182	8.2462
H(13a)	0.2203	0.7825	0.6272	4.7846
H(13b)	0.2270	0.8174	0.4893	4.7846
H(15a)	-0.2917	-0.1993	-0.7109	6.5836
H(16a)	-0.1330	-0.1056	-0.6750	7.2539

Table 3.X.4: Cont.

Atom	*x*	*y*	*z*	*B*eq
H(16b)	-0.0318	-0.1705	-0.5880	7.2539
H(17a)	-0.0401	-0.2007	-0.8218	7.1726
H(17b)	-0.1916	-0.2057	-0.9001	7.1726
H(18a)	-0.0432	-0.3445	-0.7782	9.8675
H(18b)	-0.1447	-0.3539	-0.9502	9.8675
H(19a)	-0.2165	-0.4289	-0.8109	8.4337
H(19b)	-0.3076	-0.3551	-0.8867	8.4337
H(20a)	-0.2997	-0.3347	-0.6473	6.6410
H(20b)	-0.1484	-0.3325	-0.5706	6.6410
H(21a)	-0.0479	-0.2034	-0.3882	6.4747
H(22a)	-0.1776	-0.1775	-0.2045	7.2473
H(22b)	-0.1728	-0.2743	-0.3099	7.2473
H(23a)	-0.0299	-0.2592	-0.0587	7.9396
H(23b)	0.0392	-0.2709	-0.1694	7.9396
H(24a)	0.1530	-0.1631	0.0438	7.3692
H(24b)	0.0318	-0.1063	0.0224	7.3692
H(25a)	0.1713	-0.1191	-0.1619	8.8147
H(25b)	0.1541	-0.0252	-0.0602	8.8147
H(26a)	0.0132	-0.0519	-0.3355	7.5026
H(26b)	-0.0576	-0.0273	-0.2270	7.5026

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 3.X.5: Bond distances (Å) for [Ni($S_2CN(CH(CH_2)_5)_2)_2$]

Ni(1) — S(1)	2.182(1)	C(6) — C(7)	1.534(6)
Ni(1) — S(2)	2.203(1)	C(8) — C(9)	1.527(6)
Ni(2) — S(3)	2.185(1)	C(8) — C(13)	1.515(6)
Ni(2) — S(4)	2.196(1)	C(9) — C(10)	1.542(7)
S(1) — C(1)	1.735(4)	C(10) — C(11)	1.495(9)
S(2) — C(1)	1.720(4)	C(11) — C(12)	1.496(9)
S(3) — C(14)	1.714(4)	C(12) — C(13)	1.512(7)
S(4) — C(14)	1.721(4)	C(15) — C(16)	1.450(6)
N(1) — C(1)	1.320(4)	C(15) — C(20)	1.503(6)
N(1) — C(2)	1.490(4)	C(16) — C(17)	1.489(7)
N(1) — C(8)	1.485(5)	C(17) — C(18)	1.500(8)
N(2) — C(14)	1.327(5)	C(18) — C(19)	1.469(8)
N(2) — C(15)	1.508(6)	C(19) — C(20)	1.530(7)
N(2) — C(21)	1.440(6)	C(21) — C(22)	1.503(7)
C(2) — C(3)	1.511(5)	C(21) — C(26)	1.492(7)
C(2) — C(7)	1.505(6)	C(22) — C(23)	1.460(7)
C(3) — C(4)	1.530(6)	C(23) — C(24)	1.435(7)
C(4) — C(5)	1.517(6)	C(24) — C(25)	1.584(8)
C(5) — C(6)	1.513(6)	C(25) — C(26)	1.560(7)

Table 3.X.6: Bond angles (deg.) for $[Ni(S_2CN(CH(CH_2)5)2)2]$

S(1) — Ni(1) — S(1)*	180(-)	C(18) — C(19) — C(20)	116.0(5)
S(1) — Ni(1) — S(2)	79.07(4)	C(15) — C(20) — C(19)	108.6(4)
S(1) — Ni(1) — S(2)*	100.93(4)	C(22) — C(23) — C(24)	114.6(5)
S(2) — Ni(1) — S(2)*	180(-)	C(23) — C(24) — C(25)	113.5(4)
S(3) — Ni(1) — S(3)*	180(-)	C(24) — C(25) — C(26)	108.6(5)
S(3) — Ni(2) — S(4)	79.05(5)	C(21) — C(26) — C(25)	113.6(5)
S(3) — Ni(2) — S(4)*	100.95(5)	N(1) — C(2) — C(7)	112.1(3)
S(4) — Ni(1) — S(4)*	180(-)	C(3) — C(2) — C(7)	112.0(3)
Ni(1) — S(1) — C(1)	86.5(1)	C(2) — C(3) — C(4)	110.2(4)
Ni(1) — S(2) — C(1)	86.2(1)	C(3) — C(4) — C(5)	111.6(4)
Ni(2) — S(3) — C(14)	86.4(1)	C(4) — C(5) — C(6)	111.2(3)
Ni(2) — S(4) — C(14)	85.8(1)	C(5) — C(6) — C(7)	111.3(3)
C(1) — N(1) — C(2)	119.8(3)	C(2) — C(7) — C(6)	110.5(4)
C(1) — N(1) — C(8)	123.8(3)	N(1) — C(8) — C(9)	111.6(3)
C(2) — N(1) — C(8)	116.4(3)	N(1) — C(8) — C(13)	113.0(3)
C(14) — N(2) — C(15)	120.8(4)	C(9) — C(8) — C(13)	112.8(4)
C(14) — N(2) — C(21)	124.4(4)	C(8) — C(9) — C(10)	110.1(4)
C(15) — N(2) — C(21)	114.9(4)	C(9) — C(10) — C(11)	111.5(5)
S(1) — C(1) — S(2)	107.8(2)	C(10) — C(11) — C(12)	111.0(5)
S(1) — C(1) — N(1)	126.5(3)	C(11) — C(12) — C(13)	112.6(5)
S(2) — C(1) — N(1)	125.7(3)	C(8) — C(13) — C(12)	110.2(4)
N(1) — C(2) — C(3)	112.5(3)	S(3) — C(14) — S(4)	108.5(2)
N(2) — C(21) — C(22)	113.7(4)	S(3) — C(14) — N(2)	127.8(4)
N(2) — C(21) — C(26)	115.7(4)	S(4) — C(14) — N(2)	123.7(4)
C(22) — C(21) — C(26)	114.6(5)	N(2) — C(15) — C(16)	112.3(4)
C(21) — C(22) — C(23)	111.0(5)	N(2) — C(15) — C(20)	113.5(4)

Table 3.X.6: Cont.

C(16) — C(17) — C(18)	115.4(5)	C(16) — C(15) — C(20)	118.1(4)
C(17) — C(18) — C(19)	111.6(5)	C(15) — C(16) — C(17)	112.9(5)

* atom related by a centre of symmetry

Table 3.X.7: Observed and calculated structure factors for [Ni($S_2CN(CH(CH_2)_5)_2)_2$]

h	k	l	Fo	Fc	sigF2
0	0	2	11	13	7
0	0	3	16	15	12
0	0	4	19	19	20
0	0	5	35	33	63
0	0	6	65	62	99
0	0	7	49	44	99
0	0	8	16	13	25
0	0	9	-4	1	47
0	0	10	-2	4	52
0	0	11	1	0	47
0	1	-11	-4	2	34
0	1	-10	-7	1	36
0	1	-9	6	6	16
0	1	-8	13	13	19
0	1	-7	7	4	13
0	1	-6	6	3	11
0	1	-5	1	6	18
0	1	-4	6	6	8
0	1	-3	9	11	7
0	1	-2	10	11	6
0	1	1	2	1	6
0	1	2	3	1	5
0	1	3	-4	5	17
0	1	4	9	9	10
0	1	5	23	22	26
0	1	6	9	11	17
0	1	7	19	18	27
0	1	8	8	8	21
0	1	9	-6	3	44
0	1	10	-7	0	51
0	1	11	-8	5	59
0	2	-11	6	5	16
0	2	-10	-3	7	35
0	2	-9	-1	6	39
0	2	-8	12	12	18
0	2	-7	13	16	17
0	2	-6	22	22	24
0	2	-5	14	15	14
0	2	-4	6	7	8
0	2	-3	14	15	11
0	2	-2	104	103	99
0	2	-1	7	6	4
0	2	1	142	145	99
0	2	2	31	30	36
0	2	3	7	7	8
0	2	4	30	31	48
0	2	5	67	64	99
0	2	6	60	58	99
0	2	7	45	43	82
0	2	8	-5	12	53
0	2	9	-7	3	51
0	2	10	5	3	31
0	3	-11	-3	2	36
0	3	-10	4	0	30
0	3	-9	6	6	16
0	3	-8	-2	5	26
0	3	-7	4	1	24
0	3	-6	6	5	11
0	3	-5	20	20	20
0	3	-4	16	17	12
0	3	-3	14	14	10
0	3	-2	14	14	11
0	3	-1	12	11	7
0	3	0	25	25	24
0	3	1	18	20	14
0	3	2	3	3	13
0	3	3	6	5	8
0	3	4	6	3	9
0	3	5	13	12	16
0	3	6	21	20	27
0	3	7	14	11	23
0	3	8	13	8	22
0	3	9	-3	5	48
0	3	10	8	3	24
0	4	-11	-9	9	44
0	4	-10	-2	7	23
0	4	-9	1	6	28
0	4	-8	-3	4	28
0	4	-7	0	1	23
0	4	-6	3	6	22
0	4	-5	24	23	27
0	4	-4	55	56	93
0	4	-3	66	62	99
0	4	-2	7	7	6
0	4	-1	20	19	17
0	4	0	26	25	27
0	4	1	42	41	57
0	4	2	20	22	19
0	4	3	21	21	22
0	4	4	16	17	15
0	4	5	30	27	41
0	4	6	35	32	59
0	4	7	14	12	24
0	4	8	4	4	20
0	4	9	7	3	49
0	4	10	-9	4	51
0	5	-11	-8	7	40
0	5	-10	8	7	17
0	5	-9	4	4	14
0	5	-8	10	10	16
0	5	-7	-9	4	29
0	5	-6	14	14	16
0	5	-5	3	1	9
0	5	-4	7	8	9
0	5	-3	5	7	7
0	5	-2	4	6	6
0	5	-1	38	36	49
0	5	0	11	11	8
0	5	1	38	37	52
0	5	2	22	23	23
0	14	-7	3	1	28
0	14	-6	2	3	14
0	14	-5	13	14	19
0	14	-4	24	24	32
0	14	-3	20	20	27
0	14	-2	15	14	21
0	14	-1	10	7	15
0	14	0	6	6	15
0	14	1	9	8	15
0	14	2	16	15	21
0	14	3	16	15	21
0	14	4	11	10	19
0	14	5	5	4	14
0	15	-9	5	3	23
0	15	-8	3	3	32
0	15	-7	4	7	14
0	15	-6	5	7	22
0	15	-5	5	2	30
0	15	-4	6	7	39
0	15	-3	8	7	18
0	15	-2	5	3	31
0	15	-1	13	12	19
0	15	0	4	5	35
0	15	1	5	3	16
0	15	2	3	3	34
0	15	3	6	3	15
0	15	4	5	3	27
0	16	-8	-3	6	32
0	16	-7	1	8	31
0	16	-6	5	8	17
0	16	-5	-9	5	41
0	16	-4	-1	2	39
0	16	-3	-6	2	38
0	16	-2	-1	1	34
0	16	-1	9	8	18
0	16	0	10	9	19
0	16	1	9	10	18
0	16	2	9	10	17
0	16	3	7	6	16
0	17	-7	5	1	28
0	17	-6	-3	2	33
0	17	-5	8	7	22
0	17	-4	8	8	21
0	17	-3	-5	6	41
0	17	-2	-3	1	37
0	17	-1	1	4	30
0	17	0	3	3	31
0	17	1	-6	3	30
0	17	2	5	2	27
0	18	-6	4	7	34
0	18	-5	6	4	21
0	18	-4	6	2	19
0	18	-3	-3	5	40
0	18	-2	8	8	21
0	18	-1	13	12	21
0	18	0	-4	12	48
0	18	1	4	5	24
0	19	-3	-8	1	45
0	19	-2	4	0	18
1	-18	-1	5	1	29
1	-18	0	-8	4	35
1	-18	1	2	4	33
1	-18	2	3	3	28
1	-18	3	-3	3	35
1	-18	4	4	4	25
1	-18	5	1	3	40
1	-17	-2	5	6	32
1	-17	-1	10	9	16
1	-17	0	6	13	44
1	-17	1	13	14	21
1	-17	2	7	10	21
1	-17	3	-2	3	39
1	-17	4	5	4	37
1	-17	5	-4	5	51
1	-17	6	6	9	44
1	-17	7	4	10	43
1	-16	-4	-2	3	20
1	-16	-3	2	1	26
1	-16	-2	5	1	27
1	-16	-1	-1	1	31
1	-16	0	4	6	32
1	-16	1	-2	3	24
1	-16	2	-4	2	42
1	-16	3	4	3	34
1	-16	4	1	2	41
1	-16	5	-3	3	44
1	-16	6	-4	4	29
1	-16	7	5	3	17
1	-16	8	-5	2	35
1	-15	-5	4	5	17
1	-15	-4	7	9	16
1	-15	-3	11	12	18
1	-15	-2	8	10	16
1	-15	-1	5	7	30
1	-15	0	8	5	14
1	-15	1	9	9	19
1	-15	2	22	21	31
1	-15	3	18	18	30
1	-15	4	3	9	47
1	-15	5	10	11	23
1	-15	6	10	10	20
1	-15	7	3	8	40
1	-15	8	6	5	17
1	-14	-5	5	9	16
1	-14	-4	9	9	18
1	-14	-3	5	4	25
1	-14	-2	14	15	20
1	-14	-1	2	2	34
1	-14	0	11	12	19
1	-14	1	8	10	18
1	-14	2	8	9	21
1	-14	3	4	0	25
1	-14	4	7	6	21
1	-14	5	-4	3	40
1	-14	6	4	0	25
1	-14	7	4	3	40
1	-14	8	-10	2	47
1	-14	9	-4	3	37
1	-13	-6	6	8	16
1	-13	-5	17	16	22
1	-13	-4	19	19	26
1	-13	-3	17	16	22
1	-13	-2	-5	2	30
1	-13	-1	7	6	15
1	-13	0	-2	4	23
1	-13	1	10	8	18
1	-13	2	23	21	32
1	-13	3	33	33	53
1	-13	4	17	17	27
1	-13	5	6	1	18
1	-13	6	-8	2	41
1	-13	7	5	6	40
1	-13	8	6	9	44
1	-13	9	10	11	23
1	-12	-7	7	5	13
1	-12	-6	13	11	18
1	-12	-5	4	6	34
1	-12	-4	6	3	14
1	-12	-3	15	14	20
1	-12	-2	22	21	26
1	-12	-1	8	8	15
1	-12	0	22	21	26
1	-12	1	18	18	24
1	-12	2	-9	4	42
1	-12	3	2	5	40
1	-12	4	7	8	21
1	-12	5	-7	1	40
1	-12	6	5	6	36
1	-12	7	5	4	17
1	-12	8	1	2	38
1	-12	9	-3	1	41
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12	-1	-6	6	11	18	12	3	-7	5	4	30	12	7	-1	5	4	18	13	-6	-2	-6	1	36	13	-1	0	6	2	43
12	-1	-5	18	17	23	12	3	-6	-5	8	40	12	7	0	-7	2	42	13	-6	-1	-3	2	43	13	-1	1	4	1	34
12	-1	-4	2	5	36	12	3	-5	-4	0	29	12	8	-10	2	1	29	13	-6	0	5	1	41	13	0	-10	-4	2	28
12	-1	-3	7	6	17	12	3	-4	-4	2	31	12	8	-9	5	1	14	13	-5	-8	3	2	11	13	0	-9	-3	1	24
12	-1	-2	15	15	24	12	3	-3	4	7	38	12	8	-8	10	10	18	13	-5	-7	-3	2	24	13	0	-8	-5	1	25
12	-1	-1	13	11	24	12	3	-2	3	1	37	12	8	-7	12	11	19	13	-5	-6	-2	4	32	13	0	-7	7	5	14
12	-1	0	5	6	46	12	3	-1	7	2	19	12	8	-6	14	13	21	13	-5	-5	14	15	22	13	0	-6	12	13	20

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
13	0	-5	10	10	20	13	5	-5	8	9	19	14	-2	-8	0	5	28	14	5	-8	5	6	15						
13	0	-4	-2	0	32	13	5	-4	12	10	18	14	-2	-7	8	7	14	14	5	-7	1	3	31						
13	0	-3	14	13	21	13	5	-3	7	6	17	14	-2	-6	10	10	18	14	5	-6	4	4	23						
13	0	-2	12	14	24	13	5	-2	-3	2	36	14	-2	-5	-2	7	36	14	5	-5	2	5	35						
13	0	-1	8	8	21	13	5	-1	4	5	42	14	-2	-4	2	6	33	14	5	-4	3	2	32						
13	0	0	-2	3	27	13	6	-9	-7	5	29	14	-2	-3	7	4	16	14	5	-3	-5	2	35						
13	0	1	3	7	49	13	6	-8	9	7	16	14	-2	-2	-8	1	43	14	6	-8	8	8	16						
13	1	-10	10	10	16	13	6	-7	-9	3	39	14	-2	-1	-7	2	44	14	6	-7	9	11	19						
13	1	-9	10	10	16	13	6	-6	7	7	17	14	-1	-8	-1	0	24	14	6	-6	7	12	21						
13	1	-8	3	6	31	13	6	-5	12	10	20	14	-1	-7	-7	2	30	14	6	-5	9	10	19						
13	1	-7	-2	2	30	13	6	-4	-2	6	38	14	-1	-6	-9	4	26	14	6	-4	7	6	16						
13	1	-6	-4	1	29	13	6	-3	-6	0	33	14	-1	-5	5	0	14	14	7	-6	-8	2	34						
13	1	-5	-4	2	32	13	6	-2	-3	7	38	14	-1	-4	-4	4	34	15	-1	-5	6	6	16						
13	1	-4	6	5	16	13	7	-9	-7	2	30	14	-1	-3	-5	5	37	15	0	-6	7	7	17						
13	1	-3	-9	7	44	13	7	-8	3	6	32	14	-1	-2	6	6	20	15	0	-5	1	0	32						
13	1	-2	8	7	20	13	7	-7	13	14	22	14	-1	-1	6	2	42												
13	1	-1	6	7	43	13	7	-6	14	14	21	14	0	-9	9	10	15												
13	1	0	-9	1	43	13	7	-5	5	7	36	14	0	-8	6	6	16												
13	2	-10	5	3	13	13	7	-4	-5	1	31	14	0	-7	4	1	28												
13	2	-9	4	1	26	13	7	-3	2	1	37	14	0	-6	5	0	16												
13	2	-8	5	2	33	13	7	-2	-3	3	37	14	0	-5	6	6	17												
13	2	-7	-6	2	30	13	8	-8	1	1	27	14	0	-4	5	8	40												
13	2	-6	3	1	28	13	8	-7	6	7	15	14	0	-3	9	10	21												
13	2	-5	-10	2	40	13	8	-6	6	8	40	14	0	-2	6	9	19												
13	2	-4	-4	2	31	13	8	-5	7	8	18	14	0	-1	5	7	38												
13	2	-3	-3	2	32	13	8	-4	5	4	22	14	1	-9	2	4	27												
13	2	-2	-9	0	45	13	8	-3	-3	1	35	14	1	-8	4	2	13												
13	2	-1	5	7	41	13	9	-7	8	9	19	14	1	-7	4	1	14												
13	2	0	-11	4	51	13	9	-6	7	8	18	14	1	-6	-5	4	30												
13	3	-10	-3	7	29	13	9	-5	6	3	15	14	1	-5	8	6	17												
13	3	-9	6	4	14	13	9	-4	-6	0	32	14	1	-4	-3	1	40												
13	3	-8	6	4	14	14	-6	-6	5	3	29	14	1	-3	3	2	40												
13	3	-7	-7	6	34	14	-6	-5	12	13	21	14	1	-2	5	3	38												
13	3	-6	9	10	17	14	-6	-4	13	14	22	14	2	-9	7	7	15												
13	3	-5	15	15	21	14	-6	-3	9	10	20	14	2	-8	-3	1	29												
13	3	-4	16	15	23	14	-5	-7	-7	0	29	14	2	-7	4	4	20												
13	3	-3	9	11	19	14	-5	-6	5	6	34	14	2	-6	5	1	14												
13	3	-2	4	7	27	14	-5	-5	6	9	39	14	2	-5	6	6	16												
13	3	-1	5	4	19	14	-5	-4	3	6	44	14	2	-4	15	15	23												
13	3	0	2	3	38	14	-5	-3	5	2	18	14	2	-3	14	15	24												
13	4	-10	-6	4	31	14	-5	-2	-9	3	40	14	2	-2	-4	3	36												
13	4	-9	5	4	31	14	-4	-7	3	4	14	14	3	-9	5	1	12												
13	4	-8	-2	6	26	14	-4	-6	3	6	15	14	3	-8	-8	3	30												
13	4	-7	3	4	20	14	-4	-5	8	9	19	14	3	-7	5	0	29												
13	4	-6	4	1	28	14	-4	-4	9	8	22	14	3	-6	-7	2	28												
13	4	-5	4	1	28	14	-4	-3	-7	3	39	14	3	-5	5	3	28												
13	4	-4	-8	2	35	14	-4	-2	5	1	38	14	3	-4	4	0	24												
13	4	-3	-4	0	33	14	-3	-8	3	2	23	14	3	-3	5	2	22												
13	4	-2	4	6	41	14	-3	-7	-6	0	30	14	3	-2	5	1	32												
13	4	-1	2	3	36	14	-3	-6	-8	1	36	14	4	-8	2	1	30												
13	5	-10	3	4	28	14	-3	-5	6	4	15	14	4	-7	3	1	27												
13	5	-9	-1	1	31	14	-3	-4	4	1	39	14	4	-6	4	7	34												
13	5	-8	-4	2	31	14	-3	-3	2	2	36	14	4	-5	10	11	21												
13	5	-7	4	8	18	14	-3	-2	-2	1	34	14	4	-4	-3	8	39												
13	5	-6	11	11	20	14	-3	-1	5	3	37	14	4	-3	-2	4	34												

APPENDIX 3.XI

$[Ni(S_2CN(Et)Cy)_2]$

Table 3.XI.1: Crystallographic Parameters for [Ni($S_2CN(CH_2CH_3)(CH(CH_2)_5)$)2]

Formula	$C_{18}H_{32}N_2NiS_4$
Formula weight	463.40
Crystal morphology / colour	block / olive green
Crystal dimensions (mm)	0.13 x 0.15 x 0.29
Crystal system	monoclinic
Space group	$P2_1/c$ (#14)
a (Å)	8.534(4)
b (Å)	12.087(4)
c (Å)	11.109(4)
α (°)	90
β (°)	95.54(3)
γ (°)	90
V (Å^3)	1140.5(7)
Z	2
$\rho_{calc.}$ (g cm^{-3})	1.349
$F(000)$	492
Temperature (°K)	200
μ (cm^{-1})	12.10
θ limits, cell (°)	22.5 – 24.5
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 10, -14 – 15, -14 – 14
Range of transmission factors	0.953 – 1
Scan technique	ω:2θ
No. of data measured	2928
No. of unique data	2752
R_{amal}	0.029
No. of parameters	115
R (I≥0σ(I))	0.053
R_w (I≥0σ(I))	0.081
R (I≥3σ(I))	0.031
R_w (I≥3σ(I))	0.037
Maximum and minimum residual electron density (e Å^{-3})	0.49, -0.59

Table 3.XI.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Ni(S_2CN(CH_2CH_3)(CH(CH_2)_5))_2]$

Atom	*x*	*y*	*z*
Ni	0	0	0
S(1)	-0.07461(7)	0.15686(6)	0.41245(6)
S(2)	0.22317(7)	0.05510(5)	0.43641(6)
N(1)	0.1733(2)	0.2490(2)	0.3161(2)
C(1)	0.1177(3)	0.1678(2)	0.3781(2)
C(2)	0.0676(3)	0.3384(2)	0.2669(2)
C(3)	0.0500(4)	0.4308(3)	0.3566(3)
C(4)	0.3389(3)	0.2447(2)	0.2849(2)
C(5)	0.3488(3)	0.1850(2)	0.1650(2)
C(6)	0.5219(3)	0.1728(3)	0.1407(2)
C(7)	0.6027(3)	0.2844(3)	0.1406(3)
C(8)	0.5890(3)	0.3449(2)	0.2597(3)
C(9)	0.4165(3)	0.3581(2)	0.2850(2)

Table 3.XI.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Ni(S_2CN(CH_2CH_3)(CH(CH_2)_5))_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ni	0.0323(2)	0.0298(2)	0.0275(2)	0.0000(2)	0.0032(2)	0.0023(2)
S(1)	0.0331(3)	0.0409(4)	0.0445(4)	0.0052(3)	0.0066(3)	0.0115(3)
S(2)	0.0328(3)	0.0306(3)	0.0357(3)	0.0021(2)	0.0035(2)	0.0048(3)
N(1)	0.032(1)	0.035(1)	0.032(1)	0.0008(8)	-0.0023(8)	0.0068(9)
C(1)	0.032(1)	0.032(1)	0.026(1)	0.000(1)	-0.0006(9)	0.001(1)
C(2)	0.038(1)	0.049(2)	0.044(2)	0.007(1)	-0.001(1)	0.019(1)
C(3)	0.064(2)	0.046(2)	0.073(2)	0.017(1)	0.013(2)	0.014(2)
C(4)	0.030(1)	0.036(1)	0.031(1)	-0.002(1)	-0.0015(9)	0.008(1)
C(5)	0.042(1)	0.042(1)	0.035(1)	-0.007(1)	0.003(1)	0.001(1)
C(6)	0.046(2)	0.064(2)	0.042(2)	-0.001(1)	0.010(1)	-0.005(1)
C(7)	0.038(1)	0.072(2)	0.048(2)	-0.007(1)	0.007(1)	0.012(2)
C(8)	0.040(1)	0.053(2)	0.055(2)	-0.014(1)	-0.003(1)	0.012(1)
C(9)	0.042(1)	0.037(1)	0.043(2)	-0.005(1)	-0.004(1)	0.005(1)

Table 3.XI.4: Fractional atomic coordinates and B_{eq} values (Å^2) for the hydrogen atoms in [Ni($S_2CN(CH_2CH_3)(CH(CH_2)_5)$)2]*

Atom	*x*	*y*	*z*	*B*eq
H(2a)	0.1098	0.3692	0.1962	4.1552
H(2b)	-0.0356	0.3070	0.2437	4.1552
H(3a)	0.1523	0.4631	0.3804	5.7497
H(3b)	-0.0199	0.4870	0.3194	5.7497
H(3c)	0.0062	0.4013	0.4274	5.7497
H(4a)	0.3980	0.2010	0.3469	3.1163
H(5a)	0.3013	0.1123	0.1687	3.7789
H(5b)	0.2929	0.2274	0.1003	3.7789
H(6a)	0.5270	0.1379	0.0626	4.7754
H(6b)	0.5757	0.1268	0.2033	4.7754
H(7a)	0.7131	0.2736	0.1298	5.0124
H(7b)	0.5537	0.3286	0.0745	5.0124
H(8a)	0.6445	0.3029	0.3250	4.7148
H(8b)	0.6363	0.4175	0.2556	4.7148
H(9a)	0.3618	0.4039	0.2227	3.9340
H(9b)	0.4117	0.3928	0.3633	3.9340

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 3.XI.5: Bond distances (Å) for [Ni($S_2CN(CH_2CH_3)(CH(CH_2)_5)$)2]

Ni — S(1)	2.1969(6)	C(2) — C(3)	1.513(4)
Ni — S(2)	2.1975(9)	C(4) — C(5)	1.524(3)
S(1) — C(1)	1.726(2)	C(4) — C(9)	1.522(3)
S(2) — C(1)	1.724(2)	C(5) — C(6)	1.534(3)
N(1) — C(1)	1.314(3)	C(6) — C(7)	1.515(4)
N(1) — C(2)	1.478(3)	C(7) — C(8)	1.526(4)
N(1) — C(4)	1.489(3)	C(8) — C(9)	1.533(3)

Table 3.XI.6: Bond angles (deg.) for [Ni($S_2CN(CH_2CH_3)(CH(CH_2)_5)$)2]

S(1) — Ni — S(1)*	180(-)	S(2) — C(1) — N(1)	125.9(2)
S(1) — Ni — S(2)	79.59(3)	N(1) — C(2) — C(3)	113.1(2)
S(1) — Ni — S(2)*	100.41(3)	N(1) — C(4) — C(5)	110.7(2)
S(2) — Ni — S(2)*	180(-)	N(1) — C(4) — C(9)	113.0(2)
Ni — S(1) — C(1)	85.57(8)	C(5) — C(4) — C(9)	111.5(2)
Ni — S(2) — C(1)	85.59(8)	C(4) — C(5) — C(6)	109.5(2)
C(1) — N(1) — C(2)	120.1(2)	C(5) — C(6) — C(7)	111.1(2)
C(1) — N(1) — C(4)	119.7(2)	C(6) — C(7) — C(8)	110.7(2)
C(2) — N(1) — C(4)	119.9(2)	C(7) — C(8) — C(9)	111.3(2)
S(1) — C(1) — S(2)	109.2(1)	C(4) — C(9) — C(8)	109.3(2)
S(1) — C(1) — N(1)	124.9(2)		

* atom related by a centre of symmetry

Table 3.XI.7: Observed and calculated structure factors for $[Ni(S_2CN(CH_2CH_3)(CH(CH_2)_5))_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	2	128	138	82	0	4	6	574	564	99	0	8	8	219	219	99	0	13	8	51	27	99	1	2	-13	-27	2	99
0	0	4	66	55	61	0	4	7	115	108	99	0	8	9	118	111	99	0	14	0	120	115	99	1	2	-12	173	177	99
0	0	6	85	71	87	0	4	8	113	111	99	0	8	10	303	308	99	0	14	1	46	9	99	1	2	-11	-52	64	99
0	0	8	78	75	99	0	4	9	29	6	99	0	8	11	48	57	99	0	14	2	136	137	99	1	2	-10	237	244	99
0	0	10	110	115	99	0	4	10	136	127	99	0	8	12	-51	43	99	0	14	3	26	9	99	1	2	-9	262	267	99
0	0	12	184	191	99	0	4	11	89	112	99	0	9	1	96	91	99	0	14	4	107	93	99	1	2	-8	271	284	99
0	0	14	189	188	99	0	4	12	-18	74	99	0	9	2	236	227	99	0	14	5	86	85	99	1	2	-7	294	285	99
0	1	2	613	601	99	0	4	13	93	97	99	0	9	3	27	52	99	0	14	6	-29	41	99	1	2	-6	429	420	99
0	1	3	967	958	99	0	5	1	514	505	99	0	9	4	109	108	99	0	15	1	58	67	99	1	2	-5	230	235	99
0	1	4	156	156	99	0	5	2	121	114	99	0	9	5	470	458	99	0	15	2	54	13	99	1	2	-4	284	276	99
0	1	5	83	71	77	0	5	3	287	300	99	0	9	6	71	74	99	0	15	3	122	131	99	1	2	-3	534	540	99
0	1	6	340	329	99	0	5	4	71	77	88	0	9	7	403	404	99	0	15	4	16	45	99	1	2	-2	234	237	99
0	1	7	54	31	88	0	5	5	208	220	99	0	9	8	129	141	99	1	0	-14	177	177	99	1	2	-1	260	253	99
0	1	8	409	398	99	0	5	6	237	234	99	0	9	9	239	227	99	1	0	-12	352	358	99	1	2	0	867	851	99
0	1	9	77	80	99	0	5	7	237	229	99	0	9	10	-52	17	99	1	0	-10	311	298	99	1	2	1	126	128	84
0	1	10	-45	41	99	0	5	8	251	255	99	0	9	11	-46	23	99	1	0	-8	147	149	99	1	2	2	430	438	99
0	1	11	213	217	99	0	5	9	255	250	99	0	10	0	347	342	99	1	0	-6	251	246	99	1	2	3	135	59	99
0	1	12	46	5	99	0	5	10	138	142	99	0	10	1	86	90	99	1	0	-4	119	117	78	1	2	4	835	842	99
0	1	13	133	132	99	0	5	11	145	130	99	0	10	2	132	145	99	1	0	-2	406	401	99	1	2	5	291	283	99
0	1	14	-85	8	99	0	5	12	98	103	99	0	10	3	213	210	99	1	0	2	1646	1646	99	1	2	6	523	518	99
0	2	0	399	405	99	0	5	13	93	96	99	0	10	4	206	200	99	1	0	4	211	208	99	1	2	7	125	117	99
0	2	1	46	50	41	0	6	0	650	648	99	0	10	5	38	5	99	1	0	6	274	266	99	1	2	8	317	293	99
0	2	2	1205	1236	99	0	6	1	151	154	99	0	10	6	442	424	99	1	0	8	158	150	99	1	2	9	65	79	99
0	2	3	1150	1175	99	0	6	2	296	304	99	0	10	7	-72	40	99	1	0	10	297	300	99	1	2	10	82	70	99
0	2	4	542	542	99	0	6	3	-46	2	99	0	10	8	264	255	99	1	0	12	74	80	99	1	2	11	93	102	99
0	2	5	27	7	65	0	6	4	164	174	99	0	10	9	-53	10	99	1	0	14	63	61	99	1	2	12	17	12	99
0	2	6	180	181	99	0	6	5	286	288	99	0	10	10	-15	58	99	1	1	-14	-42	34	99	1	2	13	20	44	99
0	2	7	-41	3	99	0	6	6	41	33	99	0	10	11	31	17	99	1	1	-13	211	214	99	1	2	14	123	129	99
0	2	8	96	106	99	0	6	7	117	120	99	0	11	1	20	19	99	1	1	-12	47	54	99	1	3	-14	52	74	99
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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6	7	-11	46	42	99	6	9	6	23	38	99	7	0	6	349	368	99	7	3	-3	213	212	99	7	6	-10	61	46	99
6	7	-10	-19	16	99	6	9	7	66	73	99	7	0	8	211	213	99	7	3	-2	120	113	99	7	6	-9	-14	31	99

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
6	7	-9	27	16	99	6	9	8	-58	75	99	7	0	10	101	98	99	7	3	-1	186	180	99	7	6	-8	101	100	99
6	7	-8	35	4	99	6	10	-8	68	68	99	7	1	-11	59	30	97	7	3	0	159	159	99	7	6	-7	26	26	99
6	7	-7	110	116	99	6	10	-7	-18	26	99	7	1	-10	89	77	99	7	3	1	249	252	99	7	6	-6	177	177	99
6	7	-6	69	77	99	6	10	-6	-53	33	99	7	1	-9	216	215	99	7	3	2	176	171	99	7	6	-5	53	49	99
6	7	-5	376	379	99	6	10	-5	43	27	99	7	1	-8	112	115	99	7	3	3	158	160	99	7	6	-4	138	142	99
6	7	-4	12	19	99	6	10	-4	143	136	99	7	1	-7	380	372	99	7	3	4	128	134	99	7	6	-3	107	108	99
6	7	-3	296	292	99	6	10	-3	-52	2	99	7	1	-6	32	15	99	7	3	5	43	16	99	7	6	-2	161	157	99
6	7	-2	182	181	99	6	10	-2	245	240	99	7	1	-5	323	323	99	7	3	6	-58	26	99	7	6	-1	79	73	99
6	7	-1	230	228	99	6	10	-1	68	54	99	7	1	-4	175	181	99	7	3	7	-40	10	99	7	6	0	100	93	99
6	7	0	43	52	99	6	10	0	309	301	99	7	1	-3	183	192	99	7	3	8	85	90	99	7	6	1	55	43	99
6	7	1	76	73	99	6	10	1	-64	11	99	7	1	-2	248	248	99	7	3	9	220	222	99	7	6	2	101	88	99
6	7	2	265	263	99	6	10	2	136	132	99	7	1	-1	37	40	74	7	4	-11	35	51	99	7	6	3	84	64	99
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6	8	-7	49	28	99	6	11	-2	97	97	99	7	1	10	-42	26	99	7	4	0	227	225	99	7	7	-5	153	157	99
6	8	-6	139	142	99	6	11	-1	175	174	99	7	2	-11	84	90	99	7	4	1	132	135	99	7	7	-4	135	130	99
6	8	-5	-67	4	99	6	11	0	51	47	99	7	2	-10	118	123	99	7	4	2	239	241	99	7	7	-3	137	136	99
6	8	-4	306	296	99	6	11	1	103	95	99	7	2	-9	143	147	99	7	4	3	53	27	99	7	7	-2	100	96	99
6	8	-3	191	189	99	6	11	2	124	104	99	7	2	-8	151	151	99	7	4	4	44	27	99	7	7	-1	100	92	99
6	8	-2	228	227	99	6	11	3	42	64	99	7	2	-7	2	43	99	7	4	5	130	137	99	7	7	0	114	118	99
6	8	-1	-6	38	99	6	11	4	138	142	99	7	2	-6	312	311	99	7	4	6	149	147	99	7	7	1	168	167	99
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6	8	1	-25	10	99	6	12	-5	-4	19	99	7	2	-4	186	179	99	7	4	8	84	76	99	7	7	3	272	266	99
6	8	2	148	143	99	6	12	-4	162	160	99	7	2	-3	286	275	99	7	4	9	81	57	99	7	7	4	199	208	99
6	8	3	249	250	99	6	12	-3	106	99	99	7	2	-2	135	133	99	7	5	-11	56	66	99	7	7	5	169	156	99
6	8	4	42	36	99	6	12	-2	121	140	99	7	2	-1	211	219	99	7	5	-10	25	7	99	7	7	6	59	40	99
7	7	7	90	90	99	7	11	3	80	81	99	8	3	-8	129	127	99	8	6	-7	35	66	99	8	10	-3	22	21	99
7	7	8	-41	18	99	7	12	-2	-36	64	99	8	3	-7	27	10	99	8	6	-6	172	167	99	8	10	-2	23	17	99
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7	8	-4	97	93	99	8	0	-6	351	352	99	8	3	-1	348	346	99	8	6	0	65	54	99	9	0	-6	100	104	99
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7	8	-2	110	111	99	8	0	-2	183	185	99	8	3	1	156	154	99	8	6	2	132	131	99	9	0	-2	111	112	99
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7	8	1	69	75	99	8	0	4	-29	20	99	8	3	4	-40	3	99	8	6	5	73	80	99	9	0	4	224	237	99
7	8	2	258	255	99	8	0	6	101	98	99	8	3	5	40	68	99	8	6	6	264	268	99	9	0	6	-68	8	99
7	8	3	152	149	99	8	0	8	125	133	99	8	3	6	39	15	99	8	6	7	80	78	99	9	1	-9	67	77	99
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7	9	-3	163	159	99	8	1	-1	186	186	99	8	4	-3	71	71	98	8	7	1	95	103	99	9	1	1	259	267	99
7	9	-2	40	77	99	8	1	0	-25	38	99	8	4	-2	231	226	99	8	7	2	-28	12	99	9	1	2	25	41	99
7	9	-1	209	207	99	8	1	1	108	114	99	8	4	-1	162	159	99	8	7	3	208	204	99	9	1	3	177	183	99
7	9	0	34	45	99	8	1	2	162	165	99	8	4	0	215	211	99	8	7	4	21	53	99	9	1	4	71	81	99
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7	10	-2	232	235	99	8	2	-5	59	49	89	8	5	-6	186	186	99	8	8	2	196	187	99	9	2	-1	181	184	99
7	10	-1	79	62	99	8	2	-4	297	303	99	8	5	-5	125	115	99	8	8	3	68	60	99	9	2	0	257	256	99
7	10	0	280	276	99	8	2	-3	118	111	99	8	5	-4	174	165	99	8	8	4	194	193	99	9	2	1	58	44	99
7	10	1	-23	42	99	8	2	-2	295	299	99	8	5	-3	92	91	99	8	8	5	-24	3	99	9	2	2	168	165	99
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7	10	3	107	105	99	8	2	0	237	244	99	8	5	-1	83	73	99	8	9	-5	-39	13	99	9	2	4	-26	40	99
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7	11	-3	-47	48	99	8	2	5	16	2	99	8	5	4	70	64	99	8	9	0	36	40	99	9	3	-8	-10	7	99
7	11	-2	157	160	99	8	2	6	-42	15	99	8	5	5	123	121	99	8	9	1	121	126	99	9	3	-7	39	24	99
7	11	-1	209	206	99	8	2	7	19	0	99	8	5	6	90	91	99	8	9	2	91	65	99	9	3	-6	71	59	98
7	11	0	30	10	99	8	2	8	31	9	99	8	5	7	119	109	99	8	9	3	167	166	99	9	3	-5	95	91	99
7	11	1	151	152	99	8	3	-10	16	25	99	8	6	-9	-38	15	99	8	9	4	-22	41	99	9	3	-4	83	81	99
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9	3	-2	78	75	99	9	7	-2	45	52	99	10	3	0	95	107	99												
9	3	-1	289	291	99	9	7	-1	58	43	99	10	3	1	31	12	99												
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9	4	-6	21	27	99	9	8	-1	76	74	99	10	4	0	-38	61	99												
9	4	-5	189	186	99	9	8	0	36	11	99	10	4	1	29	16	99												
9	4	-4	200	199	99	9	8	1	67	63	99	10	4	2	47	44	99												

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
9	4	-3	192	192	99	9	8	2	36	10	99	10	4	3	-39	29	99												
9	4	-2	215	210	99	9	8	3	-72	7	99	10	5	-5	98	96	99												
9	4	-1	66	58	99	9	9	-2	-27	18	99	10	5	-4	138	133	99												
9	4	0	164	154	99	9	9	-1	-92	38	99	10	5	-3	-41	44	99												
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9	5	-4	203	205	99	10	1	-4	85	80	99	10	6	1	60	52	99												
9	5	-3	110	108	99	10	1	-3	72	73	99	11	0	-2	49	62	99												
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9	5	2	127	137	99	10	1	2	94	82	99	11	1	0	21	2	99												
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9	5	4	-64	63	99	10	1	4	93	83	99	11	2	-1	136	126	99												
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9	6	-6	231	239	99	10	2	-5	118	115	99																		
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9	6	-4	12	28	99	10	2	-3	41	42	99																		
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9	7	-5	124	127	99	10	3	-3	167	174	99																		
9	7	-4	-33	11	99	10	3	-2	93	91	99																		
9	7	-3	-44	22	99	10	3	-1	166	157	99																		

APPENDIX 4.I

$[Te(S_2COnPr)_2]$

Table 4.I.1: Crystallographic Parameters for $[Te(S_2COCH_2CH_2CH_3)_2]$

Formula	$C_8H_{14}O_2S_4Te$
Formula weight	398.04
Crystal morphology / colour	needle / red
Crystal dimensions (mm)	0.08 x 0.08 x 0.65
Crystal system	monoclinic
Space group	$P2_1$ (#4)
a (Å)	10.501(6)
b (Å)	5.973(3)
c (Å)	12.0551(9)
α (°)	90
β (°)	102.39(2)
γ (°)	90
V (Å^3)	738.6(4)
Z	2
$\rho_{calc.}$ (g cm^{-3})	1.790
$F(000)$	388
Temperature (°K)	293
μ (cm^{-1})	25.43
θ limits, cell (°)	16.2 – 20.8
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 13, 0 – 7, -15 – 14
Range of transmission factors	0.904 – 1
Scan technique	ω:2θ
No. of data measured	1974
No. of unique data	1878
R_{amal}	0.036
No. of parameters	135
R (I≥0σ(I))	0.077
R_w (I≥0σ(I))	0.077
R (I≥3σ(I))	0.046
R_w (I≥3σ(I))	0.037
Maximum and minimum residual electron density (e Å^{-3})	1.41, -1.62

Table 4.I.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Te(S_2COCH_2CH_2CH_3)_2]$

Atom	*x*	*y*	*z*
Te	0.02604(5)	0	0.79763(4)
S(1)	-0.1295(2)	0.2725(4)	0.6843(1)
S(2)	-0.0977(2)	0.2946(4)	0.9318(2)
S(3)	0.0506(2)	-0.1283(4)	0.6064(1)
S(4)	0.2069(2)	-0.3625(4)	0.8051(2)
O(1)	-0.2527(5)	0.547(1)	0.7806(4)
O(2)	0.2024(5)	-0.446(1)	0.5894(4)
C(1)	-0.1618(8)	0.381(2)	0.8055(6)
C(2)	-0.2920(9)	0.670(1)	0.8740(7)
C(3)	-0.3930(10)	0.830(2)	0.8194(8)
C(4)	-0.4478(9)	0.966(2)	0.9031(8)
C(5)	0.1614(7)	-0.326(1)	0.6677(6)
C(6)	0.2961(8)	-0.626(2)	0.6235(8)
C(7)	0.3100(9)	-0.745(2)	0.5150(7)
C(8)	0.4069(9)	-0.934(2)	0.5413(8)

Table 4.I.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Te(S_2COCH_2CH_2CH_3)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Te	0.0507(3)	0.0481(2)	0.0464(2)	-0.0064(5)	0.0145(2)	-0.0016(4)
S(1)	0.072(2)	0.068(2)	0.042(1)	0.013(1)	0.013(1)	0.0004(10)
S(2)	0.086(2)	0.066(2)	0.044(1)	0.011(2)	0.017(1)	-0.003(1)
S(3)	0.056(2)	0.063(1)	0.0449(10)	0.009(1)	0.015(1)	0.0026(10)
S(4)	0.068(2)	0.074(2)	0.055(1)	0.004(2)	0.004(1)	0.002(1)
O(1)	0.071(4)	0.069(6)	0.064(3)	0.023(4)	0.023(3)	0.004(3)
O(2)	0.050(4)	0.064(6)	0.058(3)	0.008(3)	0.020(3)	0.002(3)
C(1)	0.038(6)	0.074(5)	0.046(2)	-0.003(4)	0.019(3)	0.003(3)
C(2)	0.080(7)	0.067(6)	0.071(6)	0.001(5)	0.033(5)	-0.011(4)
C(3)	0.087(8)	0.096(8)	0.091(7)	0.027(5)	0.011(6)	-0.008(5)
C(4)	0.112(8)	0.08(1)	0.145(9)	0.030(9)	0.054(6)	-0.042(8)
C(5)	0.035(5)	0.059(5)	0.054(2)	-0.007(3)	0.012(3)	-0.001(3)
C(6)	0.049(6)	0.059(5)	0.093(6)	0.002(4)	0.014(6)	0.000(5)
C(7)	0.063(7)	0.070(6)	0.098(6)	-0.007(5)	0.034(6)	-0.009(5)
C(8)	0.065(7)	0.12(1)	0.164(9)	0.044(7)	0.021(7)	0.016(9)

Table 4.I.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Te(S_2COCH_2CH_2CH_3)_2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	-0.2195	0.7467	0.9182	6.6166
H(2b)	-0.3264	0.5688	0.9210	6.6166
H(3a)	-0.4623	0.7486	0.7730	8.7670
H(3b)	-0.3558	0.9288	0.7735	8.7670
H(4a)	-0.4852	0.8695	0.9499	10.3925
H(4b)	-0.5129	1.0649	0.8633	10.3925
H(4c)	-0.3799	1.0513	0.9491	10.3925
H(6a)	0.2652	-0.7275	0.6724	6.3551
H(6b)	0.3777	-0.5666	0.6614	6.3551
H(7a)	0.3391	-0.6408	0.4663	7.0705
H(7b)	0.2277	-0.8037	0.4782	7.0705
H(8a)	0.4900	-0.8756	0.5764	11.0198
H(8b)	0.4129	-1.0079	0.4728	11.0198
H(8c)	0.3791	-1.0371	0.5911	11.0198

* H atoms were included in their calculated positions, C-H 0.97Å.

Table 4.I.5: Bond distances (Å) for [Te($S_2COCH_2CH_2CH_3$)2]

Te — S(1)	2.495(3)	O(1) — C(1)	1.37(1)
Te — S(2)	2.881(3)	O(1) — C(2)	1.47(1)
Te — S(3)	2.495(3)	O(2) — C(5)	1.33(1)
Te — S(4)	2.869(4)	O(2) — C(6)	1.46(1)
S(1) — C(1)	1.70(1)	C(2) — C(3)	1.48(2)
S(2) — C(1)	1.61(1)	C(3) — C(4)	1.51(2)
S(3) — C(5)	1.71(1)	C(6) — C(7)	1.52(2)
S(4) — C(5)	1.64(1)	C(7) — C(8)	1.50(2)

Table 4.I.6: Bond angles (deg.) for [Te($S_2COCH_2CH_2CH_3$)2]

S(1) — Te — S(2)	65.66(9)	C(5) — O(2) — C(6)	120(1)
S(1) — Te — S(3)	83.1(1)	S(1) — C(1) — S(2)	124.9(8)
S(1) — Te — S(4)	149.41(9)	S(1) — C(1) — O(1)	110.3(8)
S(2) — Te — S(3)	148.8(1)	S(2) — C(1) — O(1)	124.8(9)
S(2) — Te — S(4)	144.93(9)	O(1) — C(2) — C(3)	106(1)
S(3) — Te — S(4)	66.29(9)	C(2) — C(3) — C(4)	114(1)
Te — S(1) — C(1)	90.3(4)	S(3) — C(5) — S(4)	123.7(7)
Te — S(2) — C(1)	79.2(5)	S(3) — C(5) — O(2)	111.1(8)
Te — S(3) — C(5)	90.4(4)	S(4) — C(5) — O(2)	125.2(9)
Te — S(4) — C(5)	79.6(4)	O(2) — C(6) — C(7)	107(1)
C(1) — O(1) — C(2)	119.1(9)	C(6) — C(7) — C(8)	111(1)

Table 4.I.7: Observed and calculated structure factors for [Te($S_2COCH_2CH_2CH_3$)$_2$]

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	2	962	935	99	0	3	12	119	119	99	1	0	-4	607	621	99	1	2	-7	449	440	99	1	4	-8	234	257	99
0	0	3	765	767	99	0	3	13	112	119	99	1	0	-3	284	284	99	1	2	-6	148	134	99	1	4	-7	206	222	99
0	0	4	35	29	94	0	3	14	91	103	99	1	0	-2	422	423	99	1	2	-5	752	748	99	1	4	-6	134	129	99
0	0	5	905	888	99	0	4	0	420	412	99	1	0	2	1229	1302	99	1	2	-4	307	311	99	1	4	-5	182	187	99
0	0	6	117	97	99	0	4	1	180	168	99	1	0	3	169	176	99	1	2	-3	821	828	99	1	4	-4	276	277	99
0	0	7	764	715	99	0	4	2	294	299	99	1	0	4	437	441	99	1	2	-2	670	671	99	1	4	-3	231	224	99
0	0	8	639	601	99	0	4	3	225	232	99	1	0	5	635	630	99	1	2	-1	138	135	87	1	4	-2	439	435	99
0	0	9	79	84	99	0	4	4	217	231	99	1	0	6	284	265	99	1	2	0	450	457	99	1	4	-1	331	329	99
0	0	10	548	509	99	0	4	5	90	97	99	1	0	7	553	530	99	1	2	1	454	465	99	1	4	0	411	399	99
0	0	11	65	56	99	0	4	6	112	119	99	1	0	8	444	413	99	1	2	2	637	626	99	1	4	1	114	103	99
0	0	12	212	180	99	0	4	7	220	220	99	1	0	9	11	16	99	1	2	3	588	595	99	1	4	2	266	276	99
0	0	13	57	56	99	0	4	8	232	235	99	1	0	10	346	336	99	1	2	4	335	333	99	1	4	3	299	299	99
0	0	14	45	24	99	0	4	9	144	142	99	1	0	11	94	61	99	1	2	5	698	695	99	1	4	4	132	133	99
0	0	15	208	183	99	0	4	10	110	112	99	1	0	12	82	63	99	1	2	6	233	227	99	1	4	5	239	236	99
0	1	1	539	530	99	0	4	11	-18	42	99	1	0	13	-10	34	99	1	2	7	283	276	99	1	4	6	142	136	99
0	1	2	361	362	99	0	4	12	65	74	99	1	0	14	-32	23	99	1	2	8	162	148	99	1	4	7	259	256	99
0	1	3	300	281	99	0	4	13	73	53	99	1	0	15	162	155	99	1	2	9	70	39	99	1	4	8	242	237	99
0	1	4	843	806	99	0	5	1	279	260	99	1	1	-15	68	55	94	1	2	10	248	258	99	1	4	9	150	152	99
0	1	5	167	158	99	0	5	2	230	214	99	1	1	-14	85	95	99	1	2	11	38	76	99	1	4	10	131	121	99
0	1	6	446	456	99	0	5	3	269	262	99	1	1	-13	87	101	99	1	2	12	209	207	99	1	4	11	12	20	99
0	1	7	208	221	99	0	5	4	363	358	99	1	1	-12	76	63	99	1	2	13	162	146	99	1	4	12	66	58	99
0	1	8	200	211	99	0	5	5	37	39	99	1	1	-11	274	278	99	1	2	14	67	44	99	1	5	-11	113	118	99
0	1	9	240	230	99	0	5	6	210	216	99	1	1	-10	83	95	99	1	3	-14	89	86	99	1	5	-10	52	60	99
0	1	10	-23	6	99	0	5	7	107	111	99	1	1	-9	260	256	99	1	3	-13	174	165	99	1	5	-9	78	104	99
0	1	11	293	273	99	0	5	8	130	123	99	1	1	-8	259	271	99	1	3	-12	83	88	99	1	5	-8	180	183	99
0	1	12	121	103	99	0	5	9	122	117	99	1	1	-7	270	266	99	1	3	-11	184	179	99	1	5	-7	76	86	99
0	1	13	97	92	99	0	5	10	50	25	99	1	1	-6	608	597	99	1	3	-10	92	75	99	1	5	-6	209	203	99
0	1	14	100	86	99	0	5	11	120	129	99	1	1	-5	32	42	69	1	3	-9	264	265	99	1	5	-5	40	52	99
0	1	15	18	16	99	0	6	0	222	211	99	1	1	-4	693	685	99	1	3	-8	354	357	99	1	5	-4	318	308	99
0	2	0	481	476	99	0	6	1	134	124	99	1	1	-3	717	718	99	1	3	-7	231	221	99	1	5	-3	288	279	99
0	2	1	175	168	99	0	6	2	168	173	99	1	1	-2	665	643	99	1	3	-6	516	520	99	1	5	-2	172	168	99
0	2	2	607	604	99	0	6	3	159	172	99	1	1	-1	865	836	99	1	3	-5	309	312	99	1	5	-1	288	281	99
0	2	3	776	782	99	0	6	4	-17	50	99	1	1	0	377	366	99	1	3	-4	246	246	99	1	5	0	120	115	99
0	2	4	324	317	99	0	6	5	191	204	99	1	1	1	637	622	99	1	3	-3	598	599	99	1	5	1	181	179	99
0	2	5	829	827	99	0	6	6	65	38	99	1	1	2	333	326	99	1	3	-2	251	243	99	1	5	2	233	231	99
0	2	6	140	123	99	0	6	7	118	124	99	1	1	3	372	367	99	1	3	-1	637	629	99	1	5	3	239	244	99
0	2	7	308	303	99	0	6	8	93	102	99	1	1	4	860	835	99	1	3	0	112	120	99	1	5	4	305	320	99
0	2	8	130	117	99	0	6	9	46	43	99	1	1	5	140	126	99	1	3	1	647	646	99	1	5	5	64	70	99
0	2	9	229	230	99	0	7	1	167	142	99	1	1	6	560	593	99	1	3	2	697	697	99	1	5	6	173	182	99
0	2	10	259	258	99	0	7	2	52	48	99	1	1	7	206	210	99	1	3	3	392	378	99	1	5	7	153	150	99
0	2	11	83	81	99	0	7	3	47	42	99	1	1	8	347	369	99	1	3	4	454	459	99	1	5	8	72	62	99
0	2	12	182	192	99	0	7	4	58	73	99	1	1	9	303	304	99	1	3	5	200	200	99	1	5	9	86	110	99
0	2	13	171	168	99	0	7	5	-37	24	99	1	1	10	96	84	99	1	3	6	267	264	99	1	5	10	40	15	99
0	2	14	79	72	99	0	7	6	88	97	99	1	1	11	317	298	99	1	3	7	412	413	99	1	5	11	102	108	99
0	3	1	801	802	99	1	0	-15	109	151	86	1	1	12	96	88	99	1	3	8	225	215	99	1	6	-9	70	72	99
0	3	2	549	545	99	1	0	-14	94	96	85	1	1	13	132	112	99	1	3	9	322	315	99	1	6	-8	39	68	99
0	3	3	363	364	99	1	0	-13	53	50	83	1	1	14	118	103	99	1	3	10	68	56	99	1	6	-7	175	171	99
0	3	4	300	313	99	1	0	-12	259	268	99	1	2	-15	88	90	99	1	3	11	84	84	99	1	6	-6	55	14	99
0	3	5	36	44	83	1	0	-11	154	156	99	1	2	-14	105	94	99	1	3	12	91	111	99	1	6	-5	191	194	99
0	3	6	387	387	99	1	0	-10	448	463	99	1	2	-13	157	149	99	1	3	13	83	80	99	1	6	-4	125	120	99
0	3	7	347	337	99	1	0	-9	93	95	99	1	2	-12	166	151	99	1	4	-13	22	50	99	1	6	-3	143	136	99
0	3	8	323	323	99	1	0	-8	545	533	99	1	2	-11	97	86	99	1	4	-12	124	131	99	1	6	-2	150	156	99
0	3	9	335	331	99	1	0	-7	550	524	99	1	2	-10	259	249	99	1	4	-11	-27	38	99	1	6	-1	68	47	99
0	3	10	-15	20	99	1	0	-6	214	197	99	1	2	-9	363	362	99	1	4	-10	122	127	99	1	6	0	202	199	99
0	3	11	149	150	99	1	0	-5	859	873	99	1	2	-8	162	163	99	1	4	-9	117	113	99	1	6	1	116	120	99
1	6	2	142	150	99	2	1	-11	341	342	99	2	2	14	52	16	99	2	5	-12	70	53	99	3	0	-15	30	61	87
1	6	3	194	202	99	2	1	-10	188	190	99	2	3	-14	61	53	99	2	5	-11	80	101	99	3	0	-14	90	111	99
1	6	4	63	66	99	2	1	-9	403	419	99	2	3	-13	206	189	99	2	5	-10	68	82	99	3	0	-13	-18	45	99
1	6	5	155	166	99	2	1	-8	385	399	99	2	3	-12	76	54	99	2	5	-9	81	90	99	3	0	-12	266	266	99
1	6	6	59	64	99	2	1	-7	251	259	99	2	3	-11	179	165	99	2	5	-8	241	243	99	3	0	-11	163	167	99
1	6	7	63	81	99	2	1	-6	731	733	99	2	3	-10	108	105	99	2	5	-7	72	74	99	3	0	-10	162	153	99
1	6	8	120	112	99	2	1	-5	288	273	99	2	3	-9	96	101	99	2	5	-6	171	169	99	3	0	-9	25	26	99
1	6	9	-12	7	99	2	1	-4	960	937	99	2	3	-8	298	306	99	2	5	-5	91	100	99	3	0	-8	88	87	99
1	7	-6	89	100	99	2	1	-3	483	460	99	2	3	-7	123	115	99	2	5	-4	173	185	99	3	0	-7	213	206	99
1	7	-5	34	40	99	2	1	-2	467	465	99	2	3	-6	428	426	99	2	5	-3	251	256	99	3	0	-6	261	244	99
1	7	-4	88	83	99	2	1	-1	1503	1535	99	2	3	-5	337	345	99	2	5	-2	142	148	99	3	0	-5	637	630	99
1	7	-3	49	62	99	2	1	0	1126	1127	99	2	3	-4	241	236	99	2	5	-1	317	319	99	3	0	-4	785	776	99
1	7	-2	35	52	99	2	1	1	1078	1070	99	2	3	-3	544	538	99	2	5	0	167	155	99	3	0	-3	336	339	99
1	7	-1	124	139	99	2	1	2	614	629	99	2	3	-2	130	135	99	2	5	1	130	129	99	3	0	-2	191	204	99
1	7	0	-32	17	99	2	1	3	236	224	99	2	3	-1	507	499	99	2	5	2	221	237	99	3	0	-1	75	75	99
1	7	1	156	153	99	2	1	4	812	827	99	2	3	0	36	21	79	2	5	3	148	159	99	3	0	0	398	410	99
1	7	2	47	55	99	2	1	5	153	155	99	2	3	1	275	273	99	2	5	4	235	244	99	3	0	1	131	147	97
1	7	3	29	32	99	2	1	6	595	624	99	2	3	2	552	545	99	2	5	5	42	52	99	3	0	2	272	286	99
1	7	4	92	84	99	2	1	7	278	302	99	2	3	3	279	281	99	2	5	6	117	136	99	3	0	3	706	736	99
1	7	5	28	8	99	2	1	8	286	317	99	2	3	4	406	409	99	2	5	7	165	168	99	3	0	4	303	318	99
1	7	6	84	106	99	2	1	9	333	329	99	2	3	5	240	245	99	2	5	8	36	9	99	3	0	5	481	486	99
2	0	-15	70	89	75	2	1	10	131	99	99	2	3	6	178	172	99	2	5	9	95	107	99	3	0	6	191	162	99
2	0	-14	92	120	99	2	1	11	284	264	99	2	3	7	239	219	99	2	5	10	-11	5	99	3	0	7	373	367	99
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9	5	-7	93	109	99
9	5	-6	-55	53	99
9	5	-5	92	109	99
9	5	-4	35	42	99
9	5	-3	73	100	99
9	5	-2	63	122	99
9	5	-1	-68	36	99
9	5	0	95	104	99
9	5	1	36	22	99
9	5	2	101	79	99
9	5	3	50	77	99
10	0	-12	25	64	99
10	0	-11	129	130	99
10	0	-10	66	33	99
10	0	-9	136	131	99
10	0	-8	120	101	99
10	0	-7	86	89	99
10	0	-6	268	257	99
10	0	-5	105	93	99
10	0	-4	316	325	99
10	0	-3	49	88	99
10	0	-2	140	145	99
10	0	-1	169	162	99
10	0	0	74	9	99
10	0	1	228	220	99
10	0	2	171	192	99
10	0	3	138	144	99
10	0	4	157	175	99
10	0	5	-32	16	99
10	0	6	164	129	99
10	0	7	-110	28	99
10	0	8	14	71	99
10	1	-12	114	93	99
10	1	-11	-46	20	99
10	1	-10	171	159	99
10	1	-9	96	117	99
10	1	-8	172	171	99
10	1	-7	139	136	99

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
10	1	-6	-47	26	99	10	4	-6	157	159	99	11	2	-10	73	47	99	12	1	-9	41	97	99	13	2	-4	78	56	99
10	1	-5	195	209	99	10	4	-5	36	10	99	11	2	-9	134	132	99	12	1	-8	53	52	99	13	2	-3	50	84	99
10	1	-4	126	127	99	10	4	-4	88	111	99	11	2	-8	114	117	99	12	1	-7	79	126	99	13	2	-2	85	27	99
10	1	-3	208	219	99	10	4	-3	67	99	99	11	2	-7	-14	40	99	12	1	-6	81	54	99	13	2	-1	89	71	99
10	1	-2	157	150	99	10	4	-2	74	29	99	11	2	-6	136	128	99	12	1	-5	49	108	99						
10	1	-1	73	14	99	10	4	-1	135	124	99	11	2	-5	25	64	99	12	1	-4	78	124	99						
10	1	0	179	194	99	10	4	0	72	46	99	11	2	-4	97	117	99	12	1	-3	43	70	99						
10	1	1	99	91	99	10	4	1	94	67	99	11	2	-3	109	125	99	12	1	-2	115	136	99						
10	1	2	162	171	99	10	4	2	95	89	99	11	2	-2	99	75	99	12	1	-1	61	2	99						
10	1	3	95	106	99	10	4	3	44	42	99	11	2	-1	224	211	99	12	1	0	108	93	99						
10	1	4	-4	30	99	10	4	4	78	96	99	11	2	0	-70	29	99	12	1	1	109	90	99						
10	1	5	78	99	99	10	5	-6	27	40	99	11	2	1	113	119	99	12	1	2	69	76	99						
10	1	6	58	66	99	10	5	-5	33	91	99	11	2	2	-30	64	99	12	1	3	101	83	99						
10	1	7	65	89	99	10	5	-4	-36	32	99	11	2	3	40	33	99	12	1	4	40	33	99						
10	2	-12	-46	25	99	10	5	-3	76	83	99	11	2	4	81	96	99	12	2	-9	40	76	99						
10	2	-11	140	132	99	10	5	-2	119	136	99	11	2	5	45	33	99	12	2	-8	-76	95	99						
10	2	-10	-8	77	99	10	5	-1	43	26	99	11	3	-9	73	51	99	12	2	-7	-33	22	99						
10	2	-9	160	171	99	10	5	0	54	102	99	11	3	-8	-20	52	99	12	2	-6	148	111	99						
10	2	-8	129	130	99	10	5	1	19	18	99	11	3	-7	167	156	99	12	2	-5	44	73	99						
10	2	-7	30	66	99	11	0	-11	109	113	99	11	3	-6	84	23	99	12	2	-4	125	91	99						
10	2	-6	121	158	99	11	0	-10	-35	80	99	11	3	-5	158	165	99	12	2	-3	106	97	99						
10	2	-5	104	99	99	11	0	-9	95	150	99	11	3	-4	-31	92	99	12	2	-2	48	49	99						
10	2	-4	107	130	99	11	0	-8	132	121	99	11	3	-3	22	81	99	12	2	-1	128	128	99						
10	2	-3	198	198	99	11	0	-7	77	72	99	11	3	-2	140	137	99	12	2	0	103	34	99						
10	2	-2	77	83	99	11	0	-6	248	246	99	11	3	-1	70	46	99	12	2	1	-70	72	99						
10	2	-1	257	268	99	11	0	-5	108	99	99	11	3	0	87	125	99	12	2	2	-25	69	99						
10	2	0	91	76	99	11	0	-4	226	260	99	11	3	1	67	60	99	12	2	3	35	41	99						
10	2	1	124	143	99	11	0	-3	138	149	99	11	3	2	117	67	99	12	3	-7	-59	107	99						
10	2	2	106	74	99	11	0	-2	51	85	99	11	3	3	111	138	99	12	3	-6	53	4	99						
10	2	3	88	84	99	11	0	-1	83	123	99	11	3	4	47	13	99	12	3	-5	128	114	99						
10	2	4	75	109	99	11	0	0	107	89	99	11	4	-7	92	13	99	12	3	-4	-66	81	99						
10	2	5	41	65	99	11	0	1	171	211	99	11	4	-6	126	120	99	12	3	-3	99	33	99						
10	2	6	80	89	99	11	0	2	129	135	99	11	4	-5	54	16	99	12	3	-2	-49	126	99						
10	2	7	64	78	99	11	0	3	-31	87	99	11	4	-4	25	69	99	12	3	-1	36	43	99						
10	3	-11	-50	42	99	11	0	4	148	152	99	11	4	-3	-3	69	99	12	3	0	-84	79	99						
10	3	-10	91	97	99	11	0	5	82	19	99	11	4	-2	71	9	99	12	3	1	42	61	99						
10	3	-9	75	45	99	11	0	6	88	91	99	11	4	-1	65	92	99	13	0	-7	64	42	99						
10	3	-8	93	90	99	11	1	-11	33	21	99	11	4	0	-72	16	99	13	0	-6	122	11	99						
10	3	-7	145	166	99	11	1	-10	122	96	99	11	4	1	49	66	99	13	0	-5	75	67	99						
10	3	-6	99	59	99	11	1	-9	114	112	99	12	0	-10	109	114	99	13	0	-4	19	32	99						
10	3	-5	196	193	99	11	1	-8	115	103	99	12	0	-9	114	109	99	13	0	-3	64	111	99						
10	3	-4	35	66	99	11	1	-7	105	126	99	12	0	-8	111	94	99	13	0	-2	6	4	99						
10	3	-3	93	125	99	11	1	-6	-87	79	99	12	0	-7	84	65	99	13	0	-1	-51	62	99						
10	3	-2	154	153	99	11	1	-5	132	87	99	12	0	-6	123	108	99	13	0	0	24	52	99						
10	3	-1	98	99	99	11	1	-4	128	133	99	12	0	-5	41	78	99	13	0	1	-27	39	99						
10	3	0	145	145	99	11	1	-3	127	120	99	12	0	-4	164	155	99	13	1	-7	21	110	99						
10	3	1	38	51	99	11	1	-2	111	116	99	12	0	-3	155	145	99	13	1	-6	-74	50	99						
10	3	2	92	100	99	11	1	-1	63	7	99	12	0	-2	77	39	99	13	1	-5	109	104	99						
10	3	3	140	146	99	11	1	0	47	103	99	12	0	-1	76	90	99	13	1	-4	-80	101	99						
10	3	4	-9	50	99	11	1	1	111	103	99	12	0	0	67	93	99	13	1	-3	78	60	99						
10	3	5	92	102	99	11	1	2	96	96	99	12	0	1	101	107	99	13	1	-2	69	101	99						
10	3	6	-46	6	99	11	1	3	92	68	99	12	0	2	96	107	99	13	1	-1	-41	24	99						
10	4	-9	-40	29	99	11	1	4	-60	15	99	12	0	3	58	59	99	13	1	0	12	91	99						
10	4	-8	63	98	99	11	1	5	-31	53	99	12	0	4	86	67	99	13	1	1	82	76	99						
10	4	-7	55	38	99	11	1	6	53	57	99	12	1	-10	-88	76	99	13	2	-5	113	69	99						

APPENDIX 4.II

$[Te(S_2COiBu)_2]$

Table 4.II.1: Crystallographic Parameters for $[Te(S_2COCH_2CH(CH_3)_2)_2]$

Formula	$C_{10}H_{18}O_2S_4Te$
Formula weight	426.09
Crystal morphology / colour	needle / red
Crystal dimensions (mm)	0.07 x 0.08 x 0.40
Crystal system	monoclinic
Space group	$P2_1/c$ (#14)
a (Å)	15.535(10)
b (Å)	10.402(4)
c (Å)	10.832(4)
α (°)	90
β (°)	109.43(5)
γ (°)	90
V (Å^3)	1650(1)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.714
$F(000)$	840
Temperature (°K)	200
μ (cm^{-1})	22.97
θ limits, cell (°)	25.8 – 27.5
θ limits, data (°)	3.0 – 27.5
hkl range	-20 – 20, -13 – 0, 0 – 13
Range of transmission factors	0.931 – 1
Scan technique	ω:2θ
No. of data measured	4222
No. of unique data	4019
R_{amal}	0.047
No. of parameters	154
R (I≥0σ(I))	0.071
R_w (I≥0σ(I))	0.094
R (I≥3σ(I))	0.035
R_w (I≥3σ(I))	0.042
Maximum and minimum residual electron density (e Å^{-3})	0.98, -1.13

Table 4.II.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Te(S_2COCH_2CH(CH_3)_2)_2]$

Atom	*x*	*y*	*z*
Te	0.22985(2)	-0.17392(3)	0.13447(3)
S(1)	0.2986(1)	-0.0287(1)	0.3251(1)
S(2)	0.27509(10)	0.0723(1)	0.0610(1)
S(3)	0.22501(10)	-0.3264(1)	0.3116(1)
S(4)	0.15189(10)	-0.4271(1)	0.0405(1)
O(1)	0.3486(3)	0.1965(3)	0.2858(4)
O(2)	0.1563(2)	-0.5490(3)	0.2591(3)
C(1)	0.3097(4)	0.0916(5)	0.2224(5)
C(2)	0.3693(4)	0.3018(5)	0.2114(6)
C(3)	0.4236(4)	0.3996(5)	0.3088(6)
C(4)	0.5109(5)	0.3428(6)	0.4006(7)
C(5)	0.4420(5)	0.5144(6)	0.2334(7)
C(6)	0.1731(3)	-0.4449(5)	0.2006(5)
C(7)	0.1097(4)	-0.6569(5)	0.1792(5)
C(8)	0.0941(4)	-0.7575(5)	0.2707(5)
C(9)	0.0530(4)	-0.8773(5)	0.1914(6)
C(10)	0.0317(4)	-0.7078(5)	0.3421(6)

Table 4.II.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Te(S_2COCH_2CH(CH_3)_2)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Te	0.0252(2)	0.0259(2)	0.0266(2)	-0.0027(1)	0.0087(1)	0.0006(1)
S(1)	0.0451(8)	0.0333(7)	0.0277(7)	-0.0115(6)	0.0103(6)	-0.0010(5)
S(2)	0.0373(8)	0.0307(7)	0.0305(7)	-0.0059(6)	0.0103(6)	0.0021(5)
S(3)	0.0427(8)	0.0315(6)	0.0242(6)	-0.0108(6)	0.0086(6)	-0.0010(5)
S(4)	0.0403(8)	0.0344(7)	0.0258(6)	-0.0076(6)	0.0111(6)	-0.0004(5)
O(1)	0.046(2)	0.032(2)	0.040(2)	-0.014(2)	0.012(2)	-0.006(2)
O(2)	0.040(2)	0.029(2)	0.029(2)	-0.012(2)	0.008(2)	0.003(1)
C(1)	0.033(3)	0.028(3)	0.034(3)	-0.008(2)	0.010(2)	-0.004(2)
C(2)	0.041(3)	0.036(3)	0.049(3)	-0.011(3)	0.015(3)	0.005(3)
C(3)	0.036(3)	0.036(3)	0.062(4)	-0.007(3)	0.026(3)	-0.009(3)
C(4)	0.042(4)	0.057(4)	0.071(5)	-0.013(3)	0.002(3)	-0.017(4)
C(5)	0.054(4)	0.041(3)	0.085(5)	-0.014(3)	0.029(4)	-0.005(3)
C(6)	0.023(3)	0.028(2)	0.031(3)	-0.002(2)	0.009(2)	0.002(2)
C(7)	0.031(3)	0.026(3)	0.030(3)	-0.003(2)	0.006(2)	-0.004(2)
C(8)	0.028(3)	0.028(3)	0.033(3)	-0.002(2)	0.008(2)	0.002(2)
C(9)	0.052(4)	0.022(2)	0.049(3)	-0.006(3)	0.017(3)	-0.002(2)
C(10)	0.043(3)	0.037(3)	0.040(3)	-0.005(3)	0.017(3)	0.000(2)

Table 4.II.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in [Te($S_2COCH_2CH(CH_3)_2)_2$]*

Atom	*x*	*y*	*z*	*B*eq
H(2a)	0.4046	0.2704	0.1589	3.9516
H(2b)	0.3131	0.3402	0.1547	3.9516
H(3a)	0.3869	0.4288	0.3604	4.0070
H(4a)	0.5433	0.4079	0.4632	5.7574
H(4b)	0.4966	0.2707	0.4468	5.7574
H(4c)	0.5488	0.3142	0.3507	5.7574
H(5a)	0.4770	0.4863	0.1791	5.5415
H(5b)	0.3843	0.5503	0.1785	5.5415
H(5c)	0.4760	0.5790	0.2947	5.5415
H(7a)	0.1475	-0.6927	0.1318	2.8510
H(7b)	0.0519	-0.6293	0.1174	2.8510
H(8a)	0.1523	-0.7801	0.3350	2.8630
H(9a)	0.0438	-0.9431	0.2497	3.8707
H(9b)	-0.0054	-0.8559	0.1265	3.8707
H(9c)	0.0940	-0.9096	0.1481	3.8707
H(10a)	0.0585	-0.6321	0.3926	3.7318
H(10b)	-0.0272	-0.6854	0.2789	3.7318
H(10c)	0.0234	-0.7738	0.4004	3.7318

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 4.II.5: Bond distances (Å) for [Te($S_2COCH_2CH(CH_3)_2)_2$]

Te — S(1)	2.494(1)	O(1) — C(2)	1.459(6)
Te — S(2)	2.839(1)	O(2) — C(6)	1.323(5)
Te — S(3)	2.510(1)	O(2) — C(7)	1.455(6)
Te — S(4)	2.935(1)	C(2) — C(3)	1.506(8)
S(1) — C(1)	1.721(5)	C(3) — C(4)	1.510(9)
S(2) — C(1)	1.661(5)	C(3) — C(5)	1.527(8)
S(3) — C(6)	1.723(5)	C(7) — C(8)	1.516(7)
S(4) — C(6)	1.665(5)	C(8) — C(9)	1.527(7)
O(1) — C(1)	1.324(6)	C(8) — C(10)	1.519(8)

Table 4.II.6: Bond angles (deg.) for [Te($S_2COCH_2CH(CH_3)_2)_2$]

S(1) — Te — S(2)	66.92(4)	S(1) — C(1) — O(1)	113.0(4)
S(1) — Te — S(3)	82.14(4)	S(2) — C(1) — O(1)	125.3(4)
S(1) — Te — S(4)	147.57(4)	O(1) — C(2) — C(3)	107.1(5)
S(2) — Te — S(3)	149.05(4)	C(2) — C(3) — C(4)	111.6(5)
S(2) — Te — S(4)	145.51(4)	C(2) — C(3) — C(5)	108.3(5)
S(3) — Te — S(4)	65.44(4)	C(4) — C(3) — C(5)	111.7(5)
Te — S(1) — C(1)	90.7(2)	S(3) — C(6) — S(4)	122.1(3)
Te — S(2) — C(1)	80.6(2)	S(3) — C(6) — O(2)	111.8(3)
Te — S(3) — C(6)	92.6(2)	S(4) — C(6) — O(2)	126.0(4)
Te — S(4) — C(6)	79.8(2)	O(2) — C(7) — C(8)	107.4(4)
C(1) — O(1) — C(2)	118.6(4)	C(7) — C(8) — C(9)	108.8(4)
C(6) — O(2) — C(7)	119.0(4)	C(7) — C(8) — C(10)	111.5(4)
S(1) — C(1) — S(2)	121.7(3)	C(9) — C(8) — C(10)	110.3(4)

Table 4.II.7: Observed and calculated structure factors for $[Te(S_2COCH_2CH(CH_3)_2)_2]$

h	k	l	Fo	Fc	sigF
0	-1	2	1496	1430	21
0	-1	3	565	539	12
0	-1	5	670	640	15
0	-1	6	1097	1119	19
0	-1	7	205	198	10
0	-1	8	364	360	10
0	-1	9	211	215	13
0	-1	10	756	768	17
0	-1	11	267	276	16
0	-1	12	408	396	14
0	-2	13	165	159	22
0	-2	12	203	220	16
0	-2	9	661	696	15
0	-2	8	607	622	14
0	-2	7	708	711	17
0	-2	6	317	309	9
0	-2	5	1532	1541	23
0	-2	4	1075	1061	17
0	-2	3	618	596	12
0	-2	2	495	460	11
0	0	2	1979	2071	27
0	0	4	889	930	15
0	0	6	637	665	16
0	0	10	474	444	13
0	0	12	445	420	15
0	-2	1	785	745	13
0	-2	0	497	447	11
0	-3	12	175	182	19
0	-3	11	280	260	14
0	-3	7	911	923	19
0	-3	6	188	175	10
0	-3	5	200	194	8
0	-3	4	206	197	7
0	-3	3	1854	1907	26
0	-3	2	573	556	12
0	-3	1	1581	1614	22
0	-4	12	209	206	17
0	-4	9	831	837	21
0	-4	8	119	100	17
0	-4	7	203	225	11
0	-4	6	119	107	13
0	-4	5	372	376	10
0	-4	4	524	515	14
0	-4	3	1543	1581	23
0	-4	2	297	304	8
0	-4	1	957	950	16
0	-4	0	625	593	14
0	-5	12	268	294	16
0	-5	11	318	315	14
0	-5	10	158	161	19
0	-5	8	286	268	11
0	-5	7	756	763	18
0	-5	6	907	941	20
0	-5	5	512	496	12
0	-5	4	131	130	12
1	-10	0	146	167	14
1	-10	1	408	416	11
1	-10	2	186	230	17
1	-10	3	925	963	22
1	-10	5	155	166	19
1	-10	7	495	479	13
1	-9	0	135	94	15
1	-9	1	304	316	11
1	-9	2	150	180	16
1	-9	3	640	652	15
1	-9	5	442	448	12
1	-9	7	347	361	13
1	-9	8	360	342	13
1	-9	9	348	356	14
1	-8	1	581	611	14
1	-8	2	683	733	19
1	-8	3	316	321	10
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-11	0	4	1047	1102	21	-11	-6	8	288	288	12	-12	-2	2	613	631	16	-13	0	8	635	619	16	-13	-7	9	211	205	15
-11	0	2	115	89	17	-11	-6	5	149	144	17	-12	-2	1	547	543	15	-13	0	6	485	462	13	-13	-7	6	422	412	13
-11	-1	12	311	298	14	-11	-6	4	939	942	21	-12	-3	13	388	398	15	-13	0	4	906	896	22	-13	-7	5	119	94	20
-13	-7	2	368	356	13	-14	-5	8	438	431	13	-15	-4	5	516	506	13	-16	-4	2	227	240	18	-18	0	6	438	424	15
-13	-7	1	237	243	16	-14	-5	6	241	228	14	-15	-4	4	267	243	14	-16	-4	1	204	225	20	-18	0	4	448	445	14
-13	-8	9	349	370	14	-14	-5	5	353	341	13	-15	-4	3	185	186	19	-16	-5	10	180	202	20	-18	0	2	266	238	17
-13	-8	8	238	209	14	-14	-5	4	302	328	14	-15	-4	2	144	115	17	-16	-5	9	241	239	15	-18	-1	10	424	383	15
-13	-8	6	286	272	14	-14	-5	3	475	468	13	-15	-4	1	609	648	15	-16	-5	8	278	296	15	-18	-1	6	485	470	15
-13	-8	5	350	337	14	-14	-5	2	448	439	13	-15	-5	10	163	187	21	-16	-5	6	322	314	14	-18	-1	4	308	297	15
-13	-8	4	467	466	13	-14	-5	1	295	297	14	-15	-5	7	319	303	13	-16	-5	5	218	237	18	-18	-1	2	323	315	17
-13	-8	1	305	297	14	-14	-6	10	404	409	14	-15	-5	6	460	467	13	-16	-5	4	361	364	14	-18	-1	1	365	360	15
-13	-9	8	206	194	17	-14	-6	8	236	268	17	-15	-5	5	226	225	15	-16	-5	1	397	421	14	-18	-2	9	328	320	16
-13	-9	7	379	390	14	-14	-6	7	145	171	18	-15	-5	4	245	281	18	-16	-6	8	350	359	15	-18	-2	8	327	293	16
-13	-9	5	159	143	18	-14	-6	6	455	468	13	-15	-5	3	233	256	15	-16	-6	7	222	204	16	-18	-2	7	417	421	15
-13	-9	3	533	528	15	-14	-6	2	238	223	14	-15	-5	2	164	177	21	-16	-6	6	163	147	20	-18	-2	5	151	145	20
-13	-9	2	312	306	15	-14	-7	9	156	147	19	-15	-6	10	337	356	15	-16	-6	4	190	196	19	-18	-2	4	207	220	20
-14	0	12	446	437	14	-14	-7	8	290	274	14	-15	-6	8	274	263	14	-16	-6	2	211	254	19	-18	-2	3	328	331	17
-14	0	10	630	607	17	-14	-7	7	170	177	19	-15	-6	6	524	542	14	-16	-7	6	447	462	15	-18	-2	2	160	185	25
-14	0	6	691	649	17	-14	-7	6	452	464	13	-15	-6	5	157	132	18	-16	-7	4	383	394	15	-18	-3	9	388	409	18
-14	0	2	575	579	15	-14	-7	4	541	545	14	-15	-6	4	419	412	14	-16	-7	2	222	270	19	-18	-3	5	364	389	15
-14	-1	12	266	268	16	-14	-7	3	142	160	22	-15	-7	9	184	154	18	-16	-8	4	198	196	21	-18	-3	1	348	383	16
-14	-1	10	373	367	13	-14	-7	1	178	170	19	-15	-7	6	338	348	15	-16	-8	3	330	311	15	-18	-4	7	403	375	15
-14	-1	9	192	219	16	-14	-8	7	253	249	14	-15	-7	4	137	146	23	-16	-8	2	244	299	21	-18	-4	4	217	231	19
-14	-1	8	178	195	18	-14	-8	6	334	325	14	-15	-7	2	307	295	14	-17	0	6	582	572	15	-18	-4	3	602	607	15
-14	-1	7	174	179	17	-14	-8	3	345	323	14	-15	-8	5	342	332	15	-17	0	4	641	640	17	-18	-5	6	356	352	15
-14	-1	6	474	450	13	-14	-8	2	406	391	13	-15	-8	4	458	438	14	-17	0	2	620	605	17	-18	-5	5	228	225	17
-14	-1	5	687	676	17	-14	-9	5	398	412	15	-15	-8	2	238	238	17	-17	-1	11	206	189	20	-18	-5	4	378	356	15
-14	-1	4	678	686	17	-14	-9	1	279	300	17	-15	-8	1	298	308	16	-17	-1	10	286	237	16	-18	-5	1	271	292	17
-14	-1	1	210	203	16	-15	0	10	296	327	17	-15	-9	3	406	416	15	-17	-1	8	289	259	14	-19	0	8	162	172	26
-14	-2	12	256	254	17	-15	0	8	132	94	18	-16	0	10	233	198	17	-17	-1	7	294	278	15	-19	0	6	428	420	17
-14	-2	11	341	319	13	-15	0	6	687	692	18	-16	0	8	151	158	20	-17	-1	6	240	246	17	-19	0	4	295	289	18
-14	-2	10	225	258	18	-15	0	4	1020	1064	25	-16	0	6	593	592	15	-17	-1	4	255	279	16	-19	0	2	424	446	16
-14	-2	8	263	269	13	-15	0	2	610	619	16	-16	0	4	297	309	16	-17	-1	2	257	245	17	-19	-1	8	330	327	19
-14	-2	7	623	613	18	-15	-1	12	425	424	14	-16	0	2	469	458	14	-17	-2	10	318	321	17	-19	-1	7	237	235	19
-14	-2	6	367	372	13	-15	-1	10	337	337	15	-16	-1	10	474	463	15	-17	-2	9	447	448	15	-19	-1	6	152	119	24
-14	-2	4	210	191	14	-15	-1	9	217	217	18	-16	-1	9	264	263	16	-17	-2	7	159	200	23	-19	-1	4	372	379	16
-14	-2	3	491	484	12	-15	-1	8	327	311	13	-16	-1	8	266	259	16	-17	-2	6	256	289	16	-19	-1	3	293	274	15
-14	-2	2	348	350	13	-15	-1	7	432	432	13	-16	-1	6	351	328	14	-17	-2	5	183	206	18	-19	-1	2	163	131	20
-14	-2	1	348	358	14	-15	-1	6	371	384	15	-16	-1	5	338	346	14	-17	-2	4	190	171	16	-19	-2	7	216	224	23
-14	-3	9	634	636	17	-15	-1	3	290	321	16	-16	-1	4	431	441	14	-17	-2	1	324	325	15	-19	-2	6	258	246	19
-14	-3	8	396	383	12	-15	-1	2	536	551	15	-16	-1	2	277	269	15	-17	-3	9	270	252	16	-19	-2	4	160	152	25
-14	-3	7	236	260	15	-15	-2	12	175	159	20	-16	-1	1	304	275	14	-17	-3	7	488	495	15	-19	-2	1	318	319	17
-14	-3	5	407	388	12	-15	-2	10	186	239	20	-16	-2	11	324	335	16	-17	-3	3	566	567	16	-19	-3	7	402	384	15
-14	-3	4	166	164	18	-15	-2	9	567	580	14	-16	-2	10	174	166	19	-17	-4	9	235	249	18	-19	-3	5	262	223	20
-14	-3	3	198	217	17	-15	-2	8	302	304	14	-16	-2	9	201	202	19	-17	-4	5	511	499	14	-19	-3	3	482	500	16
-14	-3	1	472	467	13	-15	-2	6	305	296	13	-16	-2	8	354	345	15	-17	-4	2	237	226	17	-19	-3	2	156	149	24
-14	-4	11	300	311	15	-15	-2	5	442	440	12	-16	-2	7	613	582	15	-17	-4	1	428	461	15	-19	-4	5	391	405	16
-14	-4	10	153	106	15	-15	-2	4	229	253	17	-16	-2	4	193	226	20	-17	-5	8	275	274	16	-20	0	4	363	355	17
-14	-4	9	126	115	21	-15	-2	1	405	408	13	-16	-2	3	363	396	14	-17	-5	7	346	355	16	-20	-1	6	409	421	17
-14	-4	8	175	172	17	-15	-3	11	322	306	14	-16	-2	1	363	389	14	-17	-5	6	167	197	23						
-14	-4	7	581	583	14	-15	-3	9	267	255	15	-16	-3	9	466	469	14	-17	-5	4	198	205	19						

h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF
-14	-4	6	227	211	14	-15	-3	7	663	662	18	-16	-3	8	200	208	18	-17	-5	1	192	218	19						
-14	-4	5	157	158	19	-15	-3	6	234	226	14	-16	-3	5	397	411	14	-17	-6	6	447	474	15						
-14	-4	4	170	161	16	-15	-3	4	237	230	15	-16	-3	1	397	420	14	-17	-6	5	157	109	22						
-14	-4	3	952	988	25	-15	-3	3	789	810	19	-16	-4	8	138	117	19	-17	-6	4	170	196	23						
-14	-4	2	251	264	15	-15	-3	1	391	399	14	-16	-4	7	327	326	14	-17	-7	4	238	220	16						
-14	-4	1	506	522	13	-15	-4	9	408	406	14	-16	-4	4	198	235	19	-17	-7	2	237	246	16						
-14	-5	9	368	389	14	-15	-4	7	270	266	14	-16	-4	3	980	1028	27	-18	0	8	201	204	22						

APPENDIX 4.III

$[Te(S_2CO\text{-}3,3\text{-}Me_2Bu)_2]$

Table 4.III.1: Crystallographic Parameters for [Te($S_2COCH_2CH_2C(CH_3)_3)_2$]

Formula	$C_{14}H_{26}O_2S_4Te$
Formula weight	482.20
Crystal morphology / colour	needle / red
Crystal dimensions (mm)	0.06 x 0.16 x 0.32
Crystal system	monoclinic
Space group	$P2_1/c$ (#14)
a (Å)	16.744(6)
b (Å)	6.098(4)
c (Å)	21.334(3)
α (°)	90
β (°)	104.89(2)
γ (°)	90
V (Å^3)	2105(1)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.521
F(000)	968
Temperature (°K)	293
μ (cm^{-1})	18.11
θ limits, cell (°)	10.3 – 13.3
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 21, 0 – 7, -27 – 26
Range of transmission factors	0.917 – 1
Scan technique	ω:2θ
No. of data measured	5469
No. of unique data	5299
R_{amal}	0.135
No. of parameters	190
R (I≥0σ(I))	0.259
R_w (I≥0σ(I))	0.158
R (I≥3σ(I))	0.061
R_w (I≥3σ(I))	0.058
Maximum and minimum residual electron density (e Å^{-3})	4.70, -6.91 (located near the Te atom)

Table 4.III.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Te(S_2COCH_2CH_2C(CH_3)_3)_2]$

Atom	*x*	*y*	*z*
Te	-0.03061(6)	0.2439(2)	0.90036(3)
S(1)	0.0007(2)	0.0236(7)	0.8115(1)
S(2)	0.0900(3)	-0.0899(8)	0.9439(1)
S(3)	-0.1260(3)	0.4579(7)	0.8114(1)
S(4)	-0.1183(3)	0.5882(8)	0.9471(1)
O(1)	0.1081(6)	-0.275(2)	0.8363(3)
O(2)	-0.2136(6)	0.762(2)	0.8404(4)
C(1)	0.0688(10)	-0.127(2)	0.8615(6)
C(2)	0.1757(10)	-0.402(3)	0.8804(6)
C(3)	0.211(1)	-0.541(3)	0.8408(6)
C(4)	0.295(1)	-0.649(3)	0.8760(6)
C(5)	0.310(1)	-0.830(3)	0.8282(7)
C(6)	0.362(1)	-0.483(3)	0.8857(7)
C(7)	0.289(1)	-0.754(4)	0.9427(6)
C(8)	-0.1568(8)	0.616(3)	0.8691(5)
C(9)	-0.251(1)	0.898(3)	0.8766(6)
C(10)	-0.316(1)	1.037(3)	0.8348(5)
C(11)	-0.378(1)	1.142(3)	0.8592(8)
C(12)	-0.335(1)	1.281(4)	0.9237(6)
C(13)	-0.434(1)	0.984(4)	0.8842(8)
C(14)	-0.431(1)	1.290(4)	0.8130(6)

Table 4.III.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Te(S_2CO(CH_2CH_2C(CH_3)_3)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Te	0.0502(6)	0.0588(6)	0.0569(4)	0.009(1)	0.0148(3)	-0.0006(7)
S(1)	0.057(3)	0.071(3)	0.055(2)	0.015(3)	0.007(2)	0.001(2)
S(2)	0.083(4)	0.090(4)	0.052(2)	0.033(3)	0.013(2)	0.008(2)
S(3)	0.073(3)	0.081(4)	0.058(2)	0.028(3)	0.025(2)	0.006(2)
S(4)	0.070(4)	0.091(4)	0.059(2)	0.029(3)	0.010(2)	-0.008(2)
O(1)	0.071(8)	0.050(8)	0.060(4)	0.036(8)	0.019(5)	0.008(6)
O(2)	0.064(7)	0.084(8)	0.079(5)	0.03(1)	0.019(5)	0.010(8)
C(1)	0.06(1)	0.032(10)	0.084(9)	0.013(10)	0.027(9)	-0.006(8)
C(2)	0.07(1)	0.06(1)	0.074(8)	0.05(1)	0.021(8)	0.004(9)
C(3)	0.10(2)	0.06(1)	0.10(1)	0.02(1)	0.04(1)	0.02(1)
C(4)	0.04(1)	0.07(1)	0.076(9)	0.03(1)	0.004(9)	-0.002(9)
C(5)	0.10(2)	0.10(2)	0.12(1)	0.01(1)	-0.03(1)	0.00(1)
C(6)	0.07(1)	0.08(2)	0.14(1)	-0.01(1)	0.02(1)	0.00(1)
C(7)	0.11(2)	0.16(2)	0.085(9)	0.02(2)	-0.017(9)	0.04(1)
C(8)	0.03(1)	0.09(1)	0.047(7)	0.03(1)	0.013(7)	-0.004(8)
C(9)	0.08(1)	0.08(1)	0.068(8)	0.06(1)	0.008(9)	-0.006(9)
C(10)	0.10(2)	0.16(2)	0.046(7)	0.07(2)	0.021(8)	-0.014(10)
C(11)	0.05(1)	0.08(2)	0.11(1)	0.01(1)	0.02(1)	-0.01(1)
C(12)	0.13(2)	0.17(2)	0.094(9)	0.03(2)	0.02(1)	-0.08(1)
C(13)	0.08(2)	0.17(2)	0.18(2)	0.03(2)	0.01(1)	0.08(2)
C(14)	0.19(2)	0.24(3)	0.054(8)	0.13(2)	0.04(1)	0.03(1)

Table 4.III.4: Fractional atomic coordinates and B_{eq} values (Å^2) for the hydrogen atoms in [Te($S_2CO(CH_2CH_2C(CH_3)_3)_2$]*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.2172	-0.3031	0.9053	6.4014
H(2b)	0.1536	-0.4907	0.9098	6.4014
H(3a)	0.2205	-0.4531	0.8053	8.1774
H(3b)	0.1722	-0.6567	0.8236	8.1774
H(5a)	0.3113	-0.7628	0.7873	11.1472
H(5b)	0.3624	-0.9020	0.8469	11.1472
H(5c)	0.2658	-0.9373	0.8209	11.1472
H(6a)	0.3637	-0.4209	0.8442	9.1337
H(6b)	0.3528	-0.3681	0.9142	9.1337
H(6c)	0.4149	-0.5540	0.9051	9.1337
H(7a)	0.2448	-0.8598	0.9349	11.9927
H(7b)	0.3412	-0.8269	0.9627	11.9927
H(7c)	0.2794	-0.6396	0.9713	11.9927
H(9a)	-0.2754	0.8084	0.9041	7.4357
H(9b)	-0.2087	0.9919	0.9034	7.4357
H(10a)	-0.2870	1.1534	0.8184	9.5016
H(10b)	-0.3445	0.9451	0.7991	9.5016
H(12a)	-0.3773	1.3568	0.9390	12.5346
H(12b)	-0.3048	1.1828	0.9571	12.5346
H(12c)	-0.2972	1.3879	0.9135	12.5346
H(13a)	-0.4005	0.8929	0.9182	13.6826
H(13b)	-0.4728	1.0677	0.9013	13.6826
H(13c)	-0.4639	0.8922	0.8488	13.6826

Table 4.III.4: Cont.

Atom	x	y	z	B_{eq}
H(14a)	-0.4721	1.3527	0.8324	15.3756
H(14b)	-0.3976	1.4066	0.8018	15.3756
H(14c)	-0.4581	1.2094	0.7742	15.3756

* H atoms were included in their calculated positions, C-H 0.97Å.

Table 4.III.5: Bond distances (Å) for $[Te(S_2COCH_2CH_2C(CH_3)_3)_2]$

Te — S(1)	2.487(4)	O(2) — C(9)	1.38(2)
Te — S(2)	2.850(4)	C(2) — C(3)	1.43(2)
Te — S(3)	2.509(4)	C(3) — C(4)	1.55(2)
Te — S(4)	2.883(4)	C(4) — C(5)	1.57(2)
S(1) — C(1)	1.63(1)	C(4) — C(6)	1.49(2)
S(2) — C(1)	1.72(1)	C(4) — C(7)	1.58(2)
S(3) — C(8)	1.74(1)	C(9) — C(10)	1.48(2)
S(4) — C(8)	1.63(1)	C(10) — C(11)	1.43(2)
O(1) — C(1)	1.31(1)	C(11) — C(12)	1.62(2)
O(1) — C(2)	1.49(1)	C(11) — C(13)	1.53(2)
O(2) — C(8)	1.33(1)	C(11) — C(14)	1.46(2)

Table 4.III.6: Bond angles (deg.) for [$Te(S_2COCH_2CH_2C(CH_3)_3)_2$]

S(1) — Te — S(2)	65.9(1)	C(3) — C(4) — C(5)	105(1)
S(1) — Te — S(3)	85.5(1)	C(3) — C(4) — C(6)	110(2)
S(1) — Te — S(4)	152.0(1)	C(3) — C(4) — C(7)	111(1)
S(2) — Te — S(3)	151.4(1)	C(5) — C(4) — C(6)	109(1)
S(2) — Te — S(4)	142.10(9)	C(5) — C(4) — C(7)	111(1)
S(3) — Te — S(4)	66.5(1)	C(6) — C(4) — C(7)	111(1)
Te — S(1) — C(1)	93.1(5)	S(3) — C(8) — S(4)	123.5(9)
Te — S(2) — C(1)	79.5(5)	S(3) — C(8) — O(2)	110.6(8)
Te — S(3) — C(8)	90.0(5)	S(4) — C(8) — O(2)	126(1)
Te — S(4) — C(8)	79.9(5)	O(2) — C(9) — C(10)	112(1)
C(1) — O(1) — C(2)	118.6(9)	C(9) — C(10) — C(11)	122(1)
C(8) — O(2) — C(9)	121(1)	C(10) — C(11) — C(12)	110(1)
S(1) — C(1) — S(2)	121.5(9)	C(10) — C(11) — C(13)	115(2)
S(1) — C(1) — O(1)	117.3(9)	C(10) — C(11) — C(14)	114(2)
S(2) — C(1) — O(1)	121(1)	C(12) — C(11) — C(13)	102(1)
O(1) — C(2) — C(3)	107(1)	C(12) — C(11) — C(14)	108(2)
C(2) — C(3) — C(4)	115(1)	C(13) — C(11) — C(14)	108(2)

Table 4.III.7: Observed and calculated structure factors for $[Te(S_2COCH_2CH_2C(CH_3)_3)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	4	3450	3570	99	0	2	17	49	110	99	0	4	22	-85	45	99	1	0	-20	396	419	99	1	1	8	1693	1579	99
0	0	6	347	360	99	0	2	18	193	185	99	0	4	23	45	31	99	1	0	-18	85	132	99	1	1	9	329	299	99
0	0	8	950	980	99	0	2	19	-128	12	99	0	5	1	84	72	99	1	0	-16	136	135	99	1	1	10	494	482	99
0	0	10	671	728	99	0	2	20	232	245	99	0	5	2	570	544	99	1	0	-14	666	779	99	1	1	11	-80	7	99
0	0	12	47	71	99	0	2	21	100	7	99	0	5	3	-55	32	99	1	0	-12	400	411	99	1	1	12	1335	1285	99
0	0	14	897	888	99	0	2	22	-112	19	99	0	5	4	-78	127	99	1	0	-10	681	742	99	1	1	13	253	257	99
0	0	16	-118	98	99	0	2	23	-73	4	99	0	5	5	-201	31	99	1	0	-8	907	894	99	1	1	14	486	439	99
0	0	18	242	268	99	0	2	24	88	60	99	0	5	6	232	178	99	1	0	-6	867	825	99	1	1	15	157	154	99
0	0	20	582	502	99	0	2	25	44	28	99	0	5	7	-60	83	99	1	0	-4	1554	1393	99	1	1	16	332	324	99
0	0	22	302	213	99	0	3	1	38	124	99	0	5	8	535	487	99	1	0	4	916	993	99	1	1	17	-205	100	99
0	0	24	309	268	99	0	3	2	1234	1209	99	0	5	9	-29	81	99	1	0	6	793	890	99	1	1	18	497	468	99
0	0	26	90	138	99	0	3	3	139	44	99	0	5	10	-139	98	99	1	0	8	347	373	99	1	1	19	141	89	99
0	1	1	66	4	99	0	3	4	1775	1699	99	0	5	11	113	90	99	1	0	10	952	1014	99	1	1	20	90	24	99
0	1	2	1405	1508	99	0	3	5	156	162	99	0	5	12	428	435	99	1	0	12	230	209	99	1	1	21	28	81	99
0	1	3	-99	117	99	0	3	6	486	457	99	0	5	13	-17	87	99	1	0	14	740	733	99	1	1	22	124	221	99
0	1	4	555	608	99	0	3	7	121	57	99	0	5	14	-136	109	99	1	0	16	180	96	99	1	1	23	-38	95	99
0	1	5	78	61	99	0	3	8	859	888	99	0	5	15	-65	33	99	1	0	18	152	229	99	1	1	24	-120	22	99
0	1	6	533	504	99	0	3	9	-119	37	99	0	5	16	71	69	99	1	0	20	707	622	99	1	1	25	-33	34	99
0	1	7	-103	2	99	0	3	10	-187	248	99	0	5	17	106	11	99	1	0	22	169	130	99	1	1	26	47	106	99
0	1	8	1655	1621	99	0	3	11	-209	35	99	0	5	18	106	111	99	1	0	24	359	286	99	1	2	-26	76	173	99
0	1	9	92	111	99	0	3	12	168	202	99	0	5	19	-134	13	99	1	0	26	16	122	99	1	2	-25	-150	61	99
0	1	10	109	169	99	0	3	13	98	70	99	0	5	20	59	63	99	1	1	-26	-68	96	99	1	2	-24	-46	32	99
0	1	11	60	40	99	0	3	14	129	25	99	0	6	0	399	424	99	1	1	-25	78	43	99	1	2	-23	-92	29	99
0	1	12	1325	1264	99	0	3	15	108	44	99	0	6	1	-96	72	99	1	1	-24	105	143	99	1	2	-22	147	14	99
0	1	13	-35	100	99	0	3	16	276	334	99	0	6	2	107	204	99	1	1	-23	97	188	99	1	2	-21	-220	78	99
0	1	14	547	498	99	0	3	17	-100	48	99	0	6	3	79	92	99	1	1	-22	68	216	99	1	2	-20	243	302	99
0	1	15	-166	11	99	0	3	18	260	317	99	0	6	4	242	252	99	1	1	-21	-124	32	99	1	2	-19	127	142	99
0	1	16	207	198	99	0	3	19	116	56	99	0	6	5	-135	18	99	1	1	-20	73	166	99	1	2	-18	-68	120	99
0	1	17	117	2	99	0	3	20	-127	83	99	0	6	6	304	295	99	1	1	-19	-92	50	99	1	2	-17	78	132	99
0	1	18	378	378	99	0	3	21	30	24	99	0	6	7	143	66	99	1	1	-18	352	438	99	1	2	-16	729	794	99
0	1	19	56	33	99	0	3	22	147	234	99	0	6	8	107	34	99	1	1	-17	-135	78	99	1	2	-15	-213	102	99
0	1	20	169	160	99	0	3	23	-140	21	99	0	6	9	-354	28	99	1	1	-16	179	152	99	1	2	-14	173	191	99
0	1	21	123	3	99	0	3	24	116	178	99	0	6	10	36	133	99	1	1	-15	227	299	99	1	2	-13	-121	78	99
0	1	22	227	240	99	0	4	0	837	861	99	0	6	11	47	25	99	1	1	-14	611	707	99	1	2	-12	442	466	99
0	1	23	26	42	99	0	4	1	155	164	99	0	6	12	7	41	99	1	1	-13	-126	162	99	1	2	-11	487	518	99
0	1	24	107	81	99	0	4	2	81	93	99	0	6	13	-121	43	99	1	1	-12	971	1030	99	1	2	-10	843	856	99
0	1	25	-163	28	99	0	4	3	164	51	99	0	6	14	-124	119	99	1	1	-11	201	257	99	1	2	-9	114	187	99
0	1	26	90	130	99	0	4	4	581	555	99	0	6	15	-32	2	99	1	1	-10	276	326	99	1	2	-8	874	817	99
0	2	0	648	676	99	0	4	5	-94	59	99	0	6	16	88	112	99	1	1	-9	274	320	99	1	2	-7	151	122	99
0	2	1	-74	31	99	0	4	6	314	297	99	0	6	17	45	30	99	1	1	-8	1493	1566	99	1	2	-6	1577	1590	99
0	2	2	668	669	99	0	4	7	-226	24	99	0	7	1	-65	16	99	1	1	-7	924	929	99	1	2	-5	223	182	99
0	2	3	-55	78	99	0	4	8	177	47	99	0	7	2	-47	56	99	1	1	-6	715	663	99	1	2	-4	1065	1093	99
0	2	4	1071	1074	99	0	4	9	95	102	99	0	7	3	18	43	99	1	1	-5	855	776	99	1	2	-3	279	254	99
0	2	5	-51	20	99	0	4	10	747	785	99	0	7	4	-53	180	99	1	1	-4	976	925	99	1	2	-2	793	827	99
0	2	6	1109	1145	99	0	4	11	-149	24	99	0	7	5	-45	90	99	1	1	-3	239	237	99	1	2	-1	230	257	99
0	2	7	-90	19	99	0	4	12	312	366	99	0	7	6	-135	134	99	1	1	-2	579	604	99	1	2	0	729	738	99
0	2	8	666	716	99	0	4	13	-126	76	99	0	7	7	-273	26	99	1	1	-1	77	145	99	1	2	1	312	287	99
0	2	9	-91	34	99	0	4	14	411	453	99	0	7	8	92	200	99	1	1	0	66	78	99	1	2	2	494	473	99
0	2	10	607	666	99	0	4	15	135	7	99	0	7	9	-78	59	99	1	1	1	60	162	99	1	2	3	401	439	99
0	2	11	85	9	99	0	4	16	265	316	99	0	7	10	122	34	99	1	1	2	605	632	99	1	2	4	1633	1687	99
0	2	12	536	558	99	0	4	17	-40	35	99	0	7	11	-121	18	99	1	1	3	144	96	99	1	2	5	152	122	99
0	2	13	73	100	99	0	4	18	103	2	99	0	7	12	89	115	99	1	1	4	607	614	99	1	2	6	721	759	99
0	2	14	259	285	99	0	4	19	-89	11	99	1	0	-26	111	159	99	1	1	5	845	830	99	1	2	7	-86	159	99
0	2	15	-56	9	99	0	4	20	140	197	99	1	0	-24	205	251	99	1	1	6	1319	1298	99	1	2	8	1163	1231	99
0	2	16	742	764	99	0	4	21	85	43	99	1	0	-22	309	286	99	1	1	7	438	427	99	1	2	9	293	318	99
1	2	10	644	686	99	1	3	14	40	121	99	1	4	21	72	35	99	1	6	-6	306	331	99	2	0	-14	765	824	99
1	2	11	305	235	99	1	3	15	-126	8	99	1	4	22	49	43	99	1	6	-5	170	11	99	2	0	-12	854	861	99
1	2	12	391	406	99	1	3	16	386	418	99	1	5	-21	-97	87	99	1	6	-4	291	246	99	2	0	-10	1595	1509	99
1	2	13	149	150	99	1	3	17	101	51	99	1	5	-20	-202	80	99	1	6	-3	-73	18	99	2	0	-8	838	805	99
1	2	14	548	551	99	1	3	18	140	328	99	1	5	-19	-85	22	99	1	6	-2	156	274	99	2	0	-6	1158	1197	99
1	2	15	-116	23	99	1	3	19	-168	66	99	1	5	-18	123	120	99	1	6	-1	-58	120	99	2	0	-4	301	301	99
1	2	16	699	742	99	1	3	20	-144	95	99	1	5	-17	210	75	99	1	6	0	403	415	99	2	0	2	63	118	99
1	2	17	70	32	99	1	3	21	-81	8	99	1	5	-16	143	99	99	1	6	1	72	66	99	2	0	4	853	863	99
1	2	18	318	336	99	1	3	22	61	219	99	1	5	-15	-160	38	99	1	6	2	152	100	99	2	0	6	1119	1171	99
1	2	19	145	99	99	1	3	23	48	9	99	1	5	-14	-218	181	99	1	6	3	126	138	99	2	0	8	367	399	99
1	2	20	189	210	99	1	3	24	67	174	99	1	5	-13	-208	132	99	1	6	4	207	310	99	2	0	10	1218	1209	99
1	2	21	89	3	99	1	4	-23	-136	39	99	1	5	-12	440	408	99	1	6	5	133	81	99	2	0	12	395	395	99
1	2	22	150	49	99	1	4	-22	-133	108	99	1	5	-11	64	26	99	1	6	6	-187	244	99	2	0	14	560	548	99
1	2	23	-230	18	99	1	4	-21	131	9	99	1	5	-10	164	183	99	1	6	7	189	34	99	2	0	16	151	111	99
1	2	24	-179	43	99	1	4	-20	-112	182	99	1	5	-9	49	123	99	1	6	8	-208	118	99	2	0	18	64	116	99
1	2	25	-71	27	99	1	4	-19	120	72	99	1	5	-8	419	418	99	1	6	9	158	2	99	2	0	20	620	546	99
1	3	-25	-36	30	99	1	4	-18	-30	42	99	1	5	-7	106	13	99	1	6	10	119	139	99	2	0	22	166	39	99
1	3	-24	137	160	99	1	4	-17	-181	22	99	1	5	-6	-29	209	99	1	6	11	186	6	99	2	0	24	278	309	99
1	3	-23	156	30	99	1	4	-16	380	313	99	1	5	-5	172	10	99	1	6	12	-172	7	99	2	0	26	-72	57	99
1	3	-22	209	207	99	1	4	-15	-216	31	99	1	5	-4	313	196	99	1	6	13	-137	45	99	2	1	-27	-83	7	99
1	3	-21	-88	6	99	1	4	-14	146	388	99	1	5	-3	167	62	99	1	6	14	145	128	99	2	1	-26	-108	38	99
1	3	-20	-306	55	99	1	4	-13	139	212	99	1	5	-2	673	643	99	1	6	15	-222	32	99	2	1	-25	22	68	99
1	3	-19	125	17	99	1	4	-12	442	405	99	1	5	-1	-63	155	99	1	6	16	64	109	99	2	1	-24	178	178	99
1	3	-18	475	420	99	1	4	-11	216	104	99	1	5	0	158	164	99	1	6	17	-49	23	99	2	1	-23	172	203	99
1	3	-17	129	14	99	1	4	-10	766	700	99	1	5	1	112	21	99	1	7	-12	59	100	99	2	1	-22	183	245	99
1	3	-16	223	245	99	1	4	-9	347	346	99	1	5	2	615	604	99	1	7	-11	-213	19	99	2	1	-21	53	56	99
1	3	-15	140	157	99	1	4	-8	-216	50	99	1	5	3	130	170	99	1	7	-10	-272	18	99	2	1	-20	121	213	99
1	3	-14	151	227	99	1	4	-7	181	110	99	1	5	4	-138	32	99	1	7	-9	-164	42	99	2	1	-19	-166	59	99
1	3	-13	106	66	99	1	4	-6	434	341	99	1	5	5	-243	124	99	1	7	-8	-106	207	99	2	1	-18	514	563	99
1	3	-12	263	265	99	1	4	-5	-101	0	99	1	5	6	343	314	99	1	7	-7	70	44	99	2	1	-17	-151	17	99
1	3	-11	108	132	99	1	4	-4	561	586	99	1	5	7	268	122	99	1	7	-6	131	116	99	2	1	-16	90	123	99
1	3	-10	-90	83	99	1	4	-3	233	253	99	1	5	8	439	445	99	1	7	-5	78	109	99	2	1	-15	522	572	99
1	3	-9	-54	87	99	1	4	-2	229	102	99	1	5	9	-270	79	99	1	7	-4	133	194	99	2	1	-14	751	814	99
1	3	-8	936	927	99	1	4	-1	91	198	99	1	5	10	149	108	99	1	7	-3	-157	33	99	2	1	-13	122	153	99
1	3	-7	99	10	99	1	4	0	825	827	99	1	5	11	214	158	99	1	7	-2	116	79	99	2	1	-12	855	904	99
1	3	-6	327	310	99	1	4	1	188	3	99	1	5	12	363	400	99	1	7	-1	-173	23	99	2	1	-11	125	196	99
1	3	-5	289	284	99	1	4	2	-128	237	99	1	5	13	145	38	99	1	7	0	129	35	99	2	1	-10	419	414	99
1	3	-4	1539	1496	99	1	4	3	-290	8	99	1	5	14	-131	29	99	1	7	1	-100	6	99	2	1	-9	32	7	99
1	3	-3	123	40	99	1	4	4	586	587	99	1	5	15	172	10	99	1	7	2	129	111	99	2	1	-8	1642	1433	99
1	3	-2	970	922	99	1	4	5	166	1	99	1	5	16	-111	56	99	1	7	3	-135	41	99	2	1	-7	832	729	99
1	3	-1	88	112	99	1	4	6	127	241	99	1	5	17	-108	29	99	1	7	4	-133	107	99	2	1	-6	805	766	99
1	3	0	214	102	99	1	4	7	-280	14	99	1	5	18	-82	120	99	1	7	5	163	32	99	2	1	-5	164	205	99
1	3	1	72	24	99	1	4	8	146	194	99	1	5	19	78	1	99	1	7	6	170	161	99	2	1	-4	1063	1047	99
1	3	2	1309	1283	99	1	4	9	174	145	99	1	5	20	39	53	99	1	7	7	61	12	99	2	1	-3	1409	1359	99
1	3	3	246	144	99	1	4	10	819	783	99	1	6	-17	72	12	99	1	7	8	151	146	99	2	1	-2	1365	1350	99
1	3	4	1257	1125	99	1	4	11	68	87	99	1	6	-16	160	142	99	1	7	9	87	38	99	2	1	-1	452	440	99
1	3	5	53	215	99	1	4	12	129	124	99	1	6	-15	180	55	99	1	7	10	65	32	99	2	1	0	813	788	99
1	3	6	810	748	99	1	4	13	-150	29	99	1	6	-14	148	126	99	1	7	11	123	26	99	2	1	1	1579	1629	99
1	3	7	128	42	99	1	4	14	368	455	99	1	6	-13	-219	35	99	1	7	12	-122	125	99	2	1	2	1419	1454	99
1	3	8	545	569	99	1	4	15	113	55	99	1	6	-12	172	81	99	2	0	-26	178	161	99	2	1	3	265	251	99
1	3	9	-170	127	99	1	4	16	-28	232	99	1	6	-11	-76	11	99	2	0	-24	204	225	99	2	1	4	50	62	99
1	3	10	149	208	99	1	4	17	-78	80	99	1	6	-10	146	175	99	2	0	-22	400	375	99	2	1	5	497	381	99
1	3	11	-158	108	99	1	4	18	-156	9	99	1	6	-9	173	66	99	2	0	-20	315	312	99	2	1	6	1398	1268	99
1	3	12	271	276	99	1	4	19	58	86	99	1	6	-8	-116	31	99	2	0	-18	117	142	99	2	1	7	401	381	99
1	3	13	43	38	99	1	4	20	164	174	99	1	6	-7	76	34	99	2	0	-16	425	434	99	2	1	8	1196	1070	99

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
2	1	9	-113	48	99	2	2	12	193	170	99	2	3	16	443	476	99	2	5	-21	44	73	99	2	6	-5	35	49	99
2	1	10	503	448	99	2	2	13	102	144	99	2	3	17	-166	15	99	2	5	-20	-36	110	99	2	6	-4	-47	139	99
2	1	11	-120	41	99	2	2	14	643	601	99	2	3	18	247	238	99	2	5	-19	-124	20	99	2	6	-3	94	17	99
2	1	12	1156	1044	99	2	2	15	-91	69	99	2	3	19	37	80	99	2	5	-18	282	142	99	2	6	-2	177	237	99
2	1	13	657	586	99	2	2	16	411	394	99	2	3	20	-142	124	99	2	5	-17	-212	65	99	2	6	-1	115	152	99
2	1	14	256	195	99	2	2	17	155	136	99	2	3	21	-79	15	99	2	5	-16	124	72	99	2	6	0	200	260	99
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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19	1	-1	104	25	99	20	0	-2	-121	182	99	21	1	-11	65	4	99												
19	1	0	-205	116	99	20	0	0	130	145	99	21	1	-10	-69	150	99												
19	1	1	147	60	99	20	0	2	-243	22	99	21	1	-9	-107	50	99												
19	1	2	-50	269	99	20	0	4	34	111	99	21	1	-8	80	184	99												
19	1	3	150	33	99	20	1	-16	-133	6	99	21	1	-7	-198	5	99												
19	1	4	96	48	99	20	1	-15	-31	23	99	21	1	-6	51	67	99												
19	1	5	-151	60	99	20	1	-14	-64	188	99	21	1	-5	46	33	99												
19	1	6	81	181	99	20	1	-13	-134	72	99	21	1	-4	-227	218	99												
19	2	-17	91	51	99	20	1	-12	75	117	99	21	1	-3	112	61	99												
19	2	-16	159	167	99	20	1	-11	-103	43	99	21	1	-2	144	18	99												
19	2	-15	163	69	99	20	1	-10	-192	123	99	21	1	-1	-57	68	99												
19	2	-14	140	135	99	20	1	-9	90	62	99	21	2	-9	92	10	99												
19	2	-13	42	62	99	20	1	-8	198	161	99	21	2	-8	111	127	99												
19	2	-12	102	151	99	20	1	-7	43	52	99	21	2	-7	-55	51	99												
19	2	-11	-288	75	99	20	1	-6	-149	9	99	21	2	-6	34	180	99												
19	2	-10	168	218	99	20	1	-5	-245	88	99	21	2	-5	-63	55	99												

APPENDIX 4.IV

$[Te(S_2COCH_2Ph)_2]$

Table 4.IV.1: Crystallographic Parameters for $[Te(S_2COCH_2C_6H_5)_2]$

Formula	$C_{16}H_{14}O_2S_4Te$
Formula weight	494.13
Crystal morphology / colour	block / red
Crystal dimensions (mm)	0.10 x 0.19 x 0.48
Crystal system	monoclinic
Space group	$P2_1/n$ (#14)
a (Å)	8.28(1)
b (Å)	5.66(1)
c (Å)	38.19(1)
α (°)	90
β (°)	91.7(1)
γ (°)	90
V (Å^3)	1789(3)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.834
$F(000)$	968
Temperature (°K)	200
μ (cm^{-1})	21.19
θ limits, cell (°)	26.0 — 27.5
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 10, 0 – 6, -35 – 35
Range of transmission factors	0.962 – 1
Scan technique	ω:2θ
No. of data measured	4260
No. of unique data	4012
R_{amal}	0.048
No. of parameters	208
R (I≥0σ(I))	0.068
R_w (I≥0σ(I))	0.104
R (I≥3σ(I))	0.037
R_w (I≥3σ(I))	0.054
Maximum and minimum residual electron density (e Å^{-3})	0.79, -0.90

Table 4.IV.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Te(S_2COCH_2C_6H_5)_2]$

Atom	*x*	*y*	*z*
Te	0.57513(3)	0.53630(5)	0.739625(8)
S(1)	0.3893(1)	0.8018(2)	0.77269(3)
S(2)	0.5275(2)	0.3842(2)	0.80870(4)
S(3)	0.5163(1)	0.8374(2)	0.69179(3)
S(4)	0.7151(1)	0.4216(2)	0.67406(4)
O(1)	0.3475(4)	0.6858(6)	0.83903(9)
O(2)	0.6458(4)	0.7897(6)	0.63275(9)
C(1)	0.4149(5)	0.6283(8)	0.8092(1)
C(2)	0.2404(6)	0.8905(9)	0.8400(1)
C(3)	0.2094(6)	0.9346(8)	0.8779(1)
C(4)	0.2704(6)	1.1380(9)	0.8944(1)
C(5)	0.2431(7)	1.1765(10)	0.9297(1)
C(6)	0.1571(7)	1.013(1)	0.9483(1)
C(7)	0.0956(7)	0.812(1)	0.9321(2)
C(8)	0.1208(6)	0.7753(9)	0.8972(1)
C(9)	0.6316(5)	0.6801(8)	0.6633(1)
C(10)	0.7474(6)	0.6786(9)	0.6073(1)
C(11)	0.7855(6)	0.8580(9)	0.5796(1)
C(12)	0.8799(7)	1.0553(10)	0.5881(1)
C(13)	0.9212(7)	1.2145(10)	0.5625(2)
C(14)	0.8712(7)	1.181(1)	0.5287(2)
C(15)	0.7782(8)	0.987(1)	0.5199(2)
C(16)	0.7348(7)	0.8260(10)	0.5454(1)

Table 4.IV.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Te(S_2COCH_2C_6H_5)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Te	0.0219(2)	0.0229(2)	0.0233(2)	-0.0008(1)	-0.0001(1)	-0.0002(1)
S(1)	0.0311(6)	0.0265(6)	0.0256(7)	0.0073(5)	0.0000(5)	0.0021(5)
S(2)	0.0326(7)	0.0274(6)	0.0303(8)	0.0107(5)	0.0030(5)	0.0044(5)
S(3)	0.0295(6)	0.0236(6)	0.0303(8)	0.0029(5)	0.0023(5)	0.0019(5)
S(4)	0.0278(6)	0.0218(6)	0.0326(8)	0.0022(4)	0.0012(5)	0.0030(5)
O(1)	0.029(2)	0.029(2)	0.025(2)	0.011(1)	0.004(1)	0.003(1)
O(2)	0.033(2)	0.027(2)	0.023(2)	0.004(1)	0.006(1)	0.004(1)
C(1)	0.021(2)	0.025(2)	0.019(3)	0.002(2)	0.003(2)	-0.003(2)
C(2)	0.029(2)	0.026(2)	0.030(3)	0.010(2)	0.000(2)	0.004(2)
C(3)	0.024(2)	0.028(2)	0.031(3)	0.009(2)	-0.003(2)	-0.001(2)
C(4)	0.033(3)	0.029(3)	0.042(4)	0.004(2)	0.000(2)	-0.001(2)
C(5)	0.053(4)	0.037(3)	0.030(4)	0.007(3)	-0.003(3)	-0.009(2)
C(6)	0.050(3)	0.054(4)	0.022(3)	0.015(3)	0.002(2)	0.001(3)
C(7)	0.039(3)	0.050(3)	0.034(4)	0.006(3)	0.007(2)	0.013(3)
C(8)	0.031(3)	0.035(3)	0.032(3)	0.001(2)	0.004(2)	0.001(2)
C(9)	0.025(2)	0.022(2)	0.017(3)	-0.003(2)	0.001(2)	0.002(2)
C(10)	0.040(3)	0.029(3)	0.028(3)	0.000(2)	0.010(2)	-0.002(2)
C(11)	0.029(3)	0.030(3)	0.027(3)	0.001(2)	0.006(2)	0.003(2)
C(12)	0.041(3)	0.037(3)	0.027(3)	-0.003(2)	-0.002(2)	-0.001(2)
C(13)	0.040(3)	0.034(3)	0.041(4)	-0.008(2)	0.008(2)	0.001(2)
C(14)	0.046(3)	0.040(3)	0.042(4)	-0.004(3)	0.012(3)	0.006(3)
C(15)	0.068(4)	0.055(4)	0.022(3)	-0.016(3)	-0.003(3)	0.007(3)
C(16)	0.049(3)	0.041(3)	0.028(3)	-0.012(3)	-0.002(2)	0.000(2)

Table 4.IV.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Te(S_2COCH_2C_6H_5)2]$*

Atom	*x*	*y*	*z*	*B*eq
H(2a)	0.2918	1.0270	0.8298	2.6770
H(2b)	0.1398	0.8573	0.8273	2.6770
H(4a)	0.3316	1.2520	0.8812	3.2669
H(5a)	0.2844	1.3179	0.9412	3.8070
H(6a)	0.1395	1.0391	0.9731	3.9554
H(7a)	0.0349	0.6981	0.9453	3.8681
H(8a)	0.0762	0.6356	0.8859	3.0730
H(10a)	0.8467	0.6240	0.6187	3.0587
H(10b)	0.6909	0.5454	0.5966	3.0587
H(12a)	0.9167	1.0807	0.6122	3.3102
H(13a)	0.9864	1.3514	0.5688	3.6168
H(14a)	0.9010	1.2932	0.5108	4.0069
H(15a)	0.7425	0.9631	0.4958	4.5960
H(16a)	0.6686	0.6904	0.5390	3.7344

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 4.IV.5: Bond distances (Å) for $[Te(S_2COCH_2C_6H_5)_2]$

Te — S(1)	2.518(2)	C(3) — C(4)	1.399(7)
Te — S(2)	2.814(1)	C(3) — C(8)	1.389(7)
Te — S(3)	2.535(1)	C(4) — C(5)	1.392(8)
Te — S(4)	2.866(2)	C(5) — C(6)	1.378(8)
S(1) — C(1)	1.713(5)	C(6) — C(7)	1.386(9)
S(2) — C(1)	1.667(5)	C(7) — C(8)	1.369(8)
S(3) — C(9)	1.719(5)	C(10) — C(11)	1.506(7)
S(4) — C(9)	1.665(5)	C(11) — C(12)	1.396(7)
O(1) — C(1)	1.324(5)	C(11) — C(16)	1.371(8)
O(1) — C(2)	1.460(5)	C(12) — C(13)	1.380(8)
O(2) — C(9)	1.328(5)	C(13) — C(14)	1.358(8)
O(2) — C(10)	1.449(6)	C(14) — C(15)	1.376(8)
C(2) — C(3)	1.499(7)	C(15) — C(16)	1.390(8)

Table 4.IV.6: Bond angles (deg.) for [Te($S_2COCH_2C_6H_5$)2]

S(1) — Te — S(2)	66.87(4)	C(4) — C(3) — C(8)	119.0(5)
S(1) — Te — S(3)	81.54(4)	C(3) — C(4) — C(5)	119.8(5)
S(1) — Te — S(4)	147.47(4)	C(4) — C(5) — C(6)	119.9(5)
S(2) — Te — S(3)	148.41(4)	C(5) — C(6) — C(7)	120.5(5)
S(2) — Te — S(4)	145.26(4)	C(6) — C(7) — C(8)	119.7(5)
S(3) — Te — S(4)	66.22(5)	C(3) — C(8) — C(7)	121.1(5)
Te — S(1) — C(1)	90.2(2)	S(3) — C(9) — S(4)	122.2(3)
Te — S(2) — C(1)	81.5(2)	S(3) — C(9) — O(2)	112.3(3)
Te — S(3) — C(9)	90.6(2)	S(4) — C(9) — O(2)	125.5(3)
Te — S(4) — C(9)	80.9(2)	O(2) — C(10) — C(11)	108.5(4)
C(1) — O(1) — C(2)	119.3(4)	C(10) — C(11) — C(12)	120.2(5)
C(9) — O(2) — C(10)	117.0(4)	C(10) — C(11) — C(16)	120.9(5)
S(1) — C(1) — S(2)	121.4(3)	C(12) — C(11) — C(16)	118.7(5)
S(1) — C(1) — O(1)	120.9(3)	C(11) — C(12) — C(13)	120.4(5)
S(2) — C(1) — O(1)	117.6(3)	C(12) — C(13) — C(14)	120.5(5)
O(1) — C(2) — C(3)	106.2(4)	C(13) — C(14) — C(15)	119.6(5)
C(2) — C(3) — C(4)	120.1(5)	C(14) — C(15) — C(16)	120.5(5)
C(2) — C(3) — C(8)	120.9(5)	C(11) — C(16) — C(15)	120.1(5)

Table 4.IV.7: Observed and calculated structure factors for $[Te(S_2COCH_2C_6H_5)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	6	1307	1222	99	0	2	5	871	866	99	0	3	25	933	954	99	0	5	9	541	534	99	0	6	28	163	151	99
0	0	8	190	147	99	0	2	6	816	826	99	0	3	26	291	285	99	0	5	10	724	731	99	0	7	1	67	97	99
0	0	10	966	903	99	0	2	7	1331	1386	99	0	3	27	648	676	99	0	5	11	278	288	99	0	7	2	139	133	99
0	0	12	1446	1406	99	0	2	8	1102	1143	99	0	3	28	205	207	99	0	5	12	835	837	99	0	7	3	280	259	99
0	0	14	1796	1788	99	0	2	9	1122	1132	99	0	3	29	282	265	99	0	5	13	316	321	99	0	7	4	133	127	99
0	0	16	1284	1272	99	0	2	10	860	863	99	0	3	30	46	132	99	0	5	14	711	701	99	0	7	5	287	287	99
0	0	18	362	346	99	0	2	11	562	593	99	0	3	31	399	404	99	0	5	15	228	215	99	0	7	6	103	126	99
0	0	20	66	71	99	0	2	12	1098	1115	99	0	3	32	56	120	99	0	5	16	496	496	99	0	7	7	135	141	99
0	0	22	-57	6	99	0	2	13	546	510	99	0	3	33	502	513	99	0	5	17	292	289	99	0	7	8	123	116	99
0	0	24	649	631	99	0	2	14	1069	1070	99	0	3	34	269	270	99	0	5	18	478	464	99	0	7	9	64	2	99
0	0	26	649	647	99	0	2	15	116	123	99	0	3	35	481	486	99	0	5	19	327	318	99	0	7	10	167	143	99
0	0	28	795	808	99	0	2	16	637	658	99	0	4	0	128	121	99	0	5	20	434	426	99	0	7	11	143	150	99
0	0	30	671	662	99	0	2	17	359	354	99	0	4	1	854	867	99	0	5	21	501	497	99	0	7	12	213	231	99
0	0	32	327	314	99	0	2	18	568	570	99	0	4	2	208	212	99	0	5	22	465	455	99	0	7	13	63	59	99
0	0	34	124	135	99	0	2	19	163	150	99	0	4	3	1024	1064	99	0	5	23	446	451	99	0	7	14	283	288	99
0	1	-17	1990	2039	99	0	2	20	307	295	99	0	4	4	723	758	99	0	5	24	473	473	99	0	7	15	91	76	99
0	1	-15	1880	1976	99	0	2	21	630	620	99	0	4	5	1233	1273	99	0	5	25	246	234	99	1	0	-35	524	494	99
0	1	-13	1637	1657	99	0	2	22	117	108	99	0	4	6	971	995	99	0	5	26	520	533	99	1	0	-33	124	74	99
0	1	-11	1551	1541	99	0	2	23	449	452	99	0	4	7	1080	1112	99	0	5	27	78	51	99	1	0	-31	666	682	99
0	1	1	133	124	94	0	2	24	390	383	99	0	4	8	1085	1141	99	0	5	28	565	573	99	1	0	-29	1215	1232	99
0	1	2	316	283	99	0	2	25	156	165	99	0	4	9	573	582	99	0	5	29	-35	13	99	1	0	-27	1379	1413	99
0	1	3	701	669	99	0	2	26	303	299	99	0	4	10	826	886	99	0	5	30	417	416	99	1	0	-25	1393	1451	99
0	1	4	185	167	99	0	2	27	318	302	99	0	4	11	319	320	99	0	5	31	-58	15	99	1	0	-23	962	968	99
0	1	5	92	80	91	0	2	28	145	146	99	0	4	12	320	325	99	0	5	32	465	458	99	1	0	-21	673	700	99
0	1	6	85	71	99	0	2	29	71	69	99	0	4	13	-31	53	99	0	5	33	20	10	99	1	0	-19	1344	1362	99
0	1	7	406	399	99	0	2	30	128	142	99	0	4	14	50	44	99	0	5	34	368	387	99	1	0	-17	1916	1983	99
0	1	8	308	288	99	0	2	31	142	147	99	0	4	15	253	241	99	0	5	35	94	114	99	1	0	-15	1362	1364	99
0	1	9	716	696	99	0	2	32	490	476	99	0	4	16	-56	37	99	0	6	0	296	305	99	1	0	-13	1771	1773	99
0	1	10	70	10	99	0	2	33	268	299	99	0	4	17	463	485	99	0	6	1	587	595	99	1	0	-11	1235	1175	99
0	1	12	612	602	99	0	2	34	768	780	99	0	4	18	99	83	99	0	6	2	70	86	99	1	0	-9	1128	1067	99
0	1	14	972	978	99	0	2	35	368	385	99	0	4	19	556	537	99	0	6	3	510	513	99	1	0	-7	101	85	99
0	1	16	409	397	99	0	3	1	291	286	99	0	4	20	-62	10	99	0	6	4	251	258	99	1	0	-5	705	617	99
0	1	18	197	174	99	0	3	2	34	76	99	0	4	21	-27	94	99	0	6	5	485	496	99	1	0	5	746	630	99
0	1	19	679	668	99	0	3	3	598	594	99	0	4	22	-59	25	99	0	6	6	227	230	99	1	0	7	299	244	99
0	1	20	914	870	99	0	3	4	53	18	99	0	4	23	403	410	99	0	6	7	246	258	99	1	0	9	164	137	99
0	1	21	544	539	99	0	3	5	866	881	99	0	4	24	130	108	99	0	6	8	-16	53	99	1	0	11	593	623	99
0	1	22	686	669	99	0	3	6	770	804	99	0	4	25	265	265	99	0	6	9	267	272	99	1	0	13	453	430	99
0	1	23	693	660	99	0	3	7	427	451	99	0	4	26	304	304	99	0	6	10	76	63	99	1	0	15	307	294	99
0	1	24	462	473	99	0	3	8	874	883	99	0	4	27	460	444	99	0	6	11	530	517	99	1	0	17	427	433	99
0	1	25	835	832	99	0	3	9	126	110	99	0	4	28	36	30	99	0	6	12	52	59	99	1	0	19	418	454	99
0	1	26	389	416	99	0	3	10	659	701	99	0	4	29	246	233	99	0	6	13	449	447	99	1	0	21	1355	1385	99
0	1	27	1090	1087	99	0	3	11	317	328	99	0	4	30	158	151	99	0	6	14	70	36	99	1	0	23	796	815	99
0	1	28	-204	26	99	0	3	12	584	591	99	0	4	31	190	189	99	0	6	15	313	328	99	1	0	25	494	523	99
0	1	29	1409	1395	99	0	3	13	192	174	99	0	4	32	303	295	99	0	6	16	47	28	99	1	0	27	827	851	99
0	1	30	156	279	99	0	3	14	491	504	99	0	4	33	123	128	99	0	6	17	375	360	99	1	0	29	1103	1131	99
0	1	31	1050	1044	99	0	3	15	73	75	99	0	4	34	427	421	99	0	6	18	161	138	99	1	0	31	1159	1182	99
0	1	32	-99	46	99	0	3	16	439	431	99	0	4	35	67	122	99	0	6	19	355	371	99	1	0	33	1161	1181	99
0	1	33	677	693	99	0	3	17	531	552	99	0	5	1	34	60	99	0	6	20	180	169	99	1	0	35	867	915	99
0	1	34	168	161	99	0	3	18	482	504	99	0	5	2	369	368	99	0	6	21	297	285	99	1	1	-35	209	127	99
0	1	35	331	357	99	0	3	19	1059	1064	99	0	5	3	-68	34	99	0	6	22	121	110	99	1	1	-34	705	697	99
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
8	2	8	414	442	99	8	3	6	215	207	99	8	4	15	363	362	99	9	1	-3	76	104	99	9	2	2	176	168	99
8	2	9	339	338	99	8	3	7	86	83	99	8	4	16	292	280	99	9	1	-2	75	60	99	9	2	3	495	499	99
8	2	10	269	272	99	8	3	8	246	249	99	8	4	17	225	222	99	9	1	-1	143	118	99	9	2	4	123	136	99
8	2	11	246	255	99	8	3	9	66	75	99	8	4	18	198	220	99	9	1	0	144	170	99	9	2	5	382	399	99
8	2	12	439	436	99	8	3	10	14	11	99	9	0	-27	244	240	99	9	1	1	70	102	99	9	2	6	145	133	99
8	2	13	65	98	99	8	3	11	80	83	99	9	0	-25	336	349	99	9	1	2	493	498	99	9	2	7	210	244	99
8	2	14	489	481	99	8	3	12	266	280	99	9	0	-23	355	340	99	9	1	3	393	384	99	9	2	8	83	108	99
8	2	15	138	116	99	8	3	13	268	284	99	9	0	-21	382	395	99	9	1	4	673	689	99	9	2	9	141	330	99
8	2	16	457	468	99	8	3	14	131	140	99	9	0	-19	208	204	99	9	1	5	207	219	99	9	2	10	143	116	99
8	2	17	155	152	99	8	3	15	227	230	99	9	0	-17	-88	28	99	9	1	6	663	689	99	9	2	11	248	241	99
8	2	18	389	406	99	8	3	16	85	13	99	9	0	-15	317	321	99	9	1	7	-80	102	99	9	2	12	159	134	99
8	2	19	284	272	99	8	3	17	208	212	99	9	0	-13	744	763	99	9	1	8	464	488	99	9	2	13	-96	192	99
8	2	20	335	324	99	8	3	18	37	21	99	9	0	-11	1123	1118	99	9	1	9	170	187	99	9	2	14	168	128	99
8	2	21	194	194	99	8	3	19	97	69	99	9	0	-9	1055	1061	99	9	1	10	343	355	99	9	2	15	186	183	99
8	2	22	262	266	99	8	3	20	55	93	99	9	0	-7	873	878	99	9	1	11	288	294	99	9	2	16	161	142	99
8	2	23	64	114	99	8	3	21	85	51	99	9	0	-5	556	567	99	9	1	12	199	201	99	9	2	17	147	142	99
8	2	24	165	143	99	8	3	22	246	220	99	9	0	-3	273	263	99	9	1	13	302	328	99	9	2	18	128	113	99
8	2	25	24	57	99	8	3	23	248	220	99	9	0	-1	259	271	99	9	1	14	328	341	99	9	2	19	126	70	99
8	2	26	165	124	99	8	3	24	339	310	99	9	0	1	366	371	99	9	1	15	165	170	99	9	2	20	58	10	99
8	2	27	61	64	99	8	3	25	166	174	99	9	0	3	607	605	99	9	1	16	445	469	99	9	2	21	166	170	99
8	2	28	193	189	99	8	4	-20	36	27	99	9	0	5	575	575	99	9	1	17	105	83	99	9	2	22	110	114	99
8	2	29	43	25	99	8	4	-19	-152	11	99	9	0	7	347	348	99	9	1	18	528	533	99	9	3	-19	304	306	99
8	3	-27	269	276	99	8	4	-18	86	45	99	9	0	9	127	155	99	9	1	19	147	116	99	9	3	-18	111	106	99
8	3	-26	122	119	99	8	4	-17	136	124	99	9	0	11	251	238	99	9	1	20	714	745	99	9	3	-17	243	233	99
8	3	-25	275	272	99	8	4	-16	99	39	99	9	0	13	67	19	99	9	1	21	91	90	99	9	3	-16	281	276	99
8	3	-24	151	156	99	8	4	-15	182	156	99	9	0	15	41	142	99	9	1	22	519	527	99	9	3	-15	-73	109	99
8	3	-23	274	299	99	8	4	-14	113	63	99	9	0	17	168	183	99	9	1	23	155	165	99	9	3	-14	392	386	99
8	3	-22	240	219	99	8	4	-13	-186	76	99	9	0	19	308	328	99	9	1	24	318	293	99	9	3	-13	122	88	99
8	3	-21	287	289	99	8	4	-12	114	107	99	9	0	21	199	172	99	9	1	25	182	188	99	9	3	-12	274	274	99
8	3	-20	381	371	99	8	4	-11	68	83	99	9	0	23	40	80	99	9	2	-24	9	80	99	9	3	-11	159	125	99
8	3	-19	308	299	99	8	4	-10	-88	18	99	9	0	25	89	13	99	9	2	-23	290	291	99	9	3	-10	47	48	99
8	3	-18	362	380	99	8	4	-9	39	96	99	9	1	-27	69	96	99	9	2	-22	-118	91	99	9	3	-9	77	121	99
8	3	-17	530	521	99	8	4	-8	48	24	99	9	1	-26	504	491	99	9	2	-21	289	262	99	9	3	-8	-86	21	99
8	3	-16	299	262	99	8	4	-7	206	192	99	9	1	-25	31	77	99	9	2	-20	235	238	99	9	3	-7	-77	33	99
8	3	-15	682	644	99	8	4	-6	148	139	99	9	1	-24	527	531	99	9	2	-19	231	216	99	9	3	-6	153	152	99
8	3	-14	266	245	99	8	4	-5	258	259	99	9	1	-23	143	108	99	9	2	-18	357	338	99	9	3	-5	184	162	99
8	3	-13	699	675	99	8	4	-4	310	286	99	9	1	-22	518	527	99	9	2	-17	268	258	99	9	3	-4	165	153	99
8	3	-12	184	173	99	8	4	-3	327	317	99	9	1	-21	149	122	99	9	2	-16	473	471	99	9	3	-3	233	226	99
8	3	-11	547	522	99	8	4	-2	321	316	99	9	1	-20	352	367	99	9	2	-15	388	358	99	9	3	-2	99	63	99
8	3	-10	208	191	99	8	4	-1	205	212	99	9	1	-19	21	135	99	9	2	-14	105	67	99	9	3	-1	247	263	99
8	3	-9	489	488	99	8	4	0	241	250	99	9	1	-18	364	361	99	9	2	-13	364	368	99	9	3	0	-130	36	99
8	3	-8	386	384	99	8	4	1	-50	118	99	9	1	-17	66	16	99	9	2	-12	178	154	99	9	3	1	327	337	99
8	3	-7	308	298	99	8	4	2	173	173	99	9	1	-16	234	258	99	9	2	-11	362	339	99	9	3	2	-50	11	99
8	3	-6	385	364	99	8	4	3	54	129	99	9	1	-15	-17	37	99	9	2	-10	-111	31	99	9	3	3	176	185	99
8	3	-5	238	263	99	8	4	4	190	169	99	9	1	-14	190	169	99	9	2	-9	432	417	99	9	3	4	81	25	99
8	3	-4	230	234	99	8	4	5	252	241	99	9	1	-13	64	77	99	9	2	-8	-37	24	99	9	3	5	-76	32	99
8	3	-3	280	279	99	8	4	6	222	194	99	9	1	-12	139	118	99	9	2	-7	391	397	99	9	3	6	140	162	99
8	3	-2	269	263	99	8	4	7	390	383	99	9	1	-11	-28	40	99	9	2	-6	240	246	99	9	3	7	125	150	99
8	3	-1	235	219	99	8	4	8	227	217	99	9	1	-10	61	31	99	9	2	-5	358	356	99	9	3	8	279	264	99
8	3	0	217	214	99	8	4	9	589	609	99	9	1	-9	150	130	99	9	2	-4	363	359	99	9	3	9	380	384	99
8	3	1	84	99	99	8	4	10	319	292	99	9	1	-8	62	23	99	9	2	-3	401	396	99	9	3	10	401	397	99
8	3	2	231	217	99	8	4	11	676	716	99	9	1	-7	-103	25	99	9	2	-2	464	453	99	9	3	11	423	448	99
8	3	3	174	153	99	8	4	12	402	417	99	9	1	-6	87	43	99	9	2	-1	515	517	99	9	3	12	534	555	99
8	3	4	248	239	99	8	4	13	559	559	99	9	1	-5	56	23	99	9	2	0	339	349	99	9	3	13	389	402	99
8	3	5	297	307	99	8	4	14	358	362	99	9	1	-4	63	26	99	9	2	1	480	473	99	9	3	14	509	495	99

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
9	3	15	326	332	99	10	1	5	489	520	99
9	3	16	472	461	99	10	1	6	194	150	99
9	3	17	194	195	99	10	1	7	475	496	99
9	4	-6	426	410	99	10	1	8	79	10	99
9	4	-5	375	378	99	10	1	9	456	484	99
9	4	-4	452	456	99	10	1	10	-96	34	99
9	4	-3	417	395	99	10	1	11	339	349	99
9	4	-2	425	420	99	10	1	12	-21	31	99
9	4	-1	406	413	99	10	1	13	172	189	99
9	4	0	232	195	99	10	1	14	68	40	99
9	4	1	351	352	99	10	1	15	255	263	99
9	4	2	91	102	99	10	2	-14	336	329	99
9	4	3	257	243	99	10	2	-13	135	76	99
9	4	4	136	119	99	10	2	-12	388	374	99
10	0	-18	334	350	99	10	2	-11	53	94	99
10	0	-16	269	280	99	10	2	-10	342	343	99
10	0	-14	326	335	99	10	2	-9	116	114	99
10	0	-12	266	252	99	10	2	-8	289	318	99
10	0	-10	222	207	99	10	2	-7	-111	119	99
10	0	-8	207	195	99	10	2	-6	135	141	99
10	0	-6	165	173	99	10	2	-5	201	171	99
10	0	-4	-77	118	99	10	2	-4	73	24	99
10	0	-2	142	150	99	10	2	-3	81	72	99
10	0	0	142	132	99	10	2	-2	25	52	99
10	0	2	159	122	99	10	2	-1	164	135	99
10	0	4	65	80	99	10	2	0	24	7	99
10	0	6	84	75	99	10	2	1	188	170	99
10	0	8	338	348	99	10	2	2	149	139	99
10	0	10	388	412	99	10	2	3	94	84	99
10	0	12	221	231	99	10	2	4	234	238	99
10	0	14	273	286	99	10	2	5	191	188	99
10	0	16	123	132	99	10	2	6	271	272	99
10	1	-18	84	80	99	10	2	7	76	108	99
10	1	-17	88	99	99	10	2	8	205	216	99
10	1	-16	170	171	99	10	2	9	-33	26	99
10	1	-15	170	166	99	10	2	10	218	266	99
10	1	-14	-122	14	99	10	2	11	119	137	99
10	1	-13	139	143	99						
10	1	-12	217	200	99						
10	1	-11	290	292	99						
10	1	-10	183	233	99						
10	1	-9	491	471	99						
10	1	-8	106	152	99						
10	1	-7	550	556	99						
10	1	-6	153	172	99						
10	1	-5	458	451	99						
10	1	-4	155	171	99						
10	1	-3	377	396	99						
10	1	-2	226	243	99						
10	1	-1	327	334	99						
10	1	0	75	101	99						
10	1	1	325	347	99						
10	1	2	79	81	99						
10	1	3	495	508	99						
10	1	4	213	212	99						

APPENDIX 4.V

$[Te(S_2CNCy_2)_2]$

Table 4.V.1: Crystallographic Parameters for [Te($S_2CN(CH(CH_2)_5)_2)_2$]

Formula	$C_{26}H_{44}N_2S_4Te$
Formula weight	640.49
Crystal morphology / colour	needle / red
Crystal dimensions (mm)	0.10 x 0.11 x 0.42
Crystal system	monoclinic
Space group	$P2_1/n$ (#14)
a (Å)	9.417(3)
b (Å)	27.11(1)
c (Å)	11.745(3)
α (°)	90
β (°)	102.10(2)
γ (°)	90
V (Å^3)	2932(1)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.451
F(000)	1320
Temperature (°K)	200
μ (cm^{-1})	13.17
θ limits, cell (°)	16.4 – 18.6
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 11, 0 – 35, -13 – 13
Range of transmission factors	0.935 – 1
Scan technique	ω:2θ
No. of data measured	5612
No. of unique data	5279
R_{amal}	0.208
No. of parameters	298
R (I≥0σ(I))	0.138
R_w (I≥0σ(I))	0.153
R (I≥3σ(I))	0.054
R_w (I≥3σ(I))	0.052
Maximum and minimum residual electron density (e Å^{-3})	1.66, -2.69

Table 4.V.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Te(S_2CN(CH(CH_2)_5)_2)_2]$

Atom	*x*	*y*	*z*
Te	0.01453(7)	0.12251(2)	0.39911(6)
S(1)	0.2019(3)	0.06822(10)	0.3399(2)
S(2)	-0.1022(3)	0.0418(1)	0.2563(2)
S(3)	0.2387(3)	0.16611(10)	0.5065(2)
S(4)	-0.0406(3)	0.2069(1)	0.5285(2)
N(1)	0.1180(9)	-0.0158(3)	0.2275(7)
N(2)	0.2137(8)	0.2472(3)	0.6309(7)
C(1)	0.075(1)	0.0258(3)	0.2706(8)
C(2)	0.271(1)	-0.0339(4)	0.2524(9)
C(3)	0.365(1)	-0.0088(4)	0.1830(9)
C(4)	0.514(1)	-0.0346(5)	0.201(1)
C(5)	0.577(1)	-0.0412(5)	0.327(1)
C(6)	0.481(1)	-0.0688(4)	0.393(1)
C(7)	0.336(1)	-0.0424(5)	0.379(1)
C(8)	0.007(1)	-0.0514(4)	0.1669(9)
C(9)	0.029(1)	-0.0707(4)	0.0527(9)
C(10)	-0.095(1)	-0.1018(4)	-0.0052(9)
C(11)	-0.127(1)	-0.1444(5)	0.073(1)
C(12)	-0.141(1)	-0.1244(5)	0.189(1)
C(13)	-0.021(1)	-0.0907(4)	0.2477(9)
C(14)	0.143(1)	0.2112(3)	0.5639(8)
C(15)	0.375(1)	0.2488(4)	0.6765(9)
C(16)	0.459(1)	0.2601(4)	0.5841(9)
C(17)	0.623(1)	0.2631(4)	0.637(1)

Table 4.V.2: Cont.

Atom	*x*	*y*	*z*
C(18)	0.677(1)	0.2189(5)	0.7064(10)
C(19)	0.592(1)	0.2097(4)	0.8023(9)
C(20)	0.432(1)	0.2052(4)	0.7531(8)
C(21)	0.127(1)	0.2889(3)	0.6690(8)
C(22)	0.124(1)	0.2860(4)	0.7947(8)
C(23)	0.035(1)	0.3282(5)	0.830(1)
C(24)	0.078(1)	0.3784(4)	0.7933(10)
C(25)	0.074(1)	0.3793(4)	0.6593(10)
C(26)	0.168(1)	0.3380(4)	0.6284(9)

Table 4.V.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Te(S_2CN(CH(CH_2)_5)_2)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Te	0.0361(4)	0.0341(3)	0.0339(3)	0.0016(4)	0.0071(3)	-0.0042(4)
S(1)	0.032(2)	0.038(2)	0.053(2)	0.001(1)	0.005(1)	-0.012(1)
S(2)	0.033(2)	0.045(2)	0.055(2)	0.001(1)	0.007(1)	-0.015(1)
S(3)	0.031(2)	0.042(2)	0.047(2)	0.002(1)	0.009(1)	-0.014(1)
S(4)	0.036(2)	0.043(2)	0.053(2)	-0.001(1)	0.014(1)	-0.014(1)
N(1)	0.033(5)	0.031(5)	0.047(5)	-0.001(4)	0.010(4)	-0.002(4)
N(2)	0.034(5)	0.028(4)	0.043(5)	0.001(4)	0.014(4)	-0.004(4)
C(1)	0.038(7)	0.030(5)	0.034(6)	-0.005(5)	0.009(5)	0.000(4)
C(2)	0.052(8)	0.046(7)	0.042(7)	0.013(6)	0.012(6)	-0.002(5)
C(3)	0.045(8)	0.056(7)	0.055(7)	0.000(6)	0.012(6)	0.002(6)
C(4)	0.039(8)	0.10(1)	0.10(1)	0.009(8)	0.031(8)	-0.020(9)
C(5)	0.041(8)	0.09(1)	0.09(1)	0.018(8)	0.014(8)	0.001(9)
C(6)	0.047(8)	0.056(7)	0.070(8)	0.015(7)	-0.007(7)	0.003(7)
C(7)	0.045(8)	0.069(8)	0.066(8)	0.005(7)	0.000(6)	0.017(7)
C(8)	0.059(8)	0.034(6)	0.038(6)	-0.011(6)	0.015(5)	-0.008(5)
C(9)	0.095(10)	0.055(8)	0.035(6)	-0.023(8)	0.007(6)	0.000(6)
C(10)	0.090(10)	0.049(7)	0.041(7)	-0.017(7)	0.003(6)	-0.001(6)
C(11)	0.09(1)	0.065(8)	0.063(9)	-0.032(8)	0.015(7)	-0.024(7)
C(12)	0.10(1)	0.079(9)	0.069(8)	-0.037(9)	0.038(8)	-0.024(8)
C(13)	0.055(8)	0.058(7)	0.032(6)	-0.018(6)	0.011(5)	-0.006(5)
C(14)	0.040(7)	0.018(5)	0.042(6)	-0.001(5)	0.020(5)	0.000(4)
C(15)	0.026(7)	0.039(6)	0.064(7)	-0.005(5)	0.008(5)	0.001(6)
C(16)	0.043(8)	0.048(6)	0.044(7)	0.001(6)	0.013(6)	0.003(5)
C(17)	0.052(8)	0.049(7)	0.079(9)	-0.015(7)	0.028(7)	-0.005(7)

Table 4.V.3: Cont.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(18)	0.037(7)	0.083(9)	0.056(8)	0.011(7)	0.013(6)	0.005(7)
C(19)	0.050(8)	0.068(8)	0.043(7)	0.011(7)	0.007(6)	-0.001(6)
C(20)	0.027(6)	0.038(6)	0.048(6)	0.003(5)	0.012(5)	-0.007(5)
C(21)	0.042(7)	0.025(5)	0.047(6)	-0.004(5)	0.002(5)	-0.012(5)
C(22)	0.051(7)	0.051(7)	0.035(6)	0.006(6)	0.020(5)	-0.004(5)
C(23)	0.054(8)	0.064(8)	0.091(10)	0.002(7)	0.046(7)	-0.013(7)
C(24)	0.043(7)	0.050(7)	0.079(8)	0.002(7)	0.031(6)	-0.024(7)
C(25)	0.047(7)	0.042(6)	0.072(8)	0.000(6)	0.008(6)	-0.007(7)
C(26)	0.067(9)	0.056(8)	0.040(6)	0.013(7)	0.012(6)	-0.002(6)

Table 4.V.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Te(S_2CN(CH(CH_2)_5)_2)_2]$*

Atom	x	*y*	*z*	*B*eq
H(2a)	0.2617	-0.0670	0.2204	4.3836
H(3a)	0.3189	-0.0099	0.1012	4.9471
H(3b)	0.3796	0.0252	0.2080	4.9471
H(4a)	0.5018	-0.0666	0.1630	7.2815
H(4b)	0.5791	-0.0146	0.1658	7.2815
H(5a)	0.5974	-0.0089	0.3614	6.9402
H(5b)	0.6678	-0.0594	0.3338	6.9402
H(6a)	0.4659	-0.1021	0.3631	5.7196
H(6b)	0.5279	-0.0699	0.4749	5.7196
H(7a)	0.3502	-0.0109	0.4186	5.8330
H(7b)	0.2702	-0.0625	0.4126	5.8330
H(8a)	-0.0823	-0.0326	0.1482	4.0794
H(9a)	0.0385	-0.0430	0.0025	5.9477
H(9b)	0.1170	-0.0903	0.0657	5.9477
H(10a)	-0.0722	-0.1157	-0.0754	5.8184
H(10b)	-0.1806	-0.0812	-0.0257	5.8184
H(11a)	-0.0486	-0.1682	0.0834	6.7927
H(11b)	-0.2174	-0.1603	0.0361	6.7927
H(12a)	-0.1438	-0.1522	0.2406	7.4608
H(12b)	-0.2314	-0.1063	0.1785	7.4608
H(13a)	-0.0492	-0.0754	0.3144	4.5724
H(13b)	0.0666	-0.1098	0.2736	4.5724
H(15a)	0.3911	0.2771	0.7281	4.0973
H(16a)	0.4259	0.2914	0.5480	4.2025

Table 4.V.4: Cont.

Atom	x	*y*	*z*	*B*eq
H(16b)	0.4414	0.2342	0.5258	4.2025
H(17a)	0.6408	0.2920	0.6865	5.4992
H(17b)	0.6744	0.2663	0.5739	5.4992
H(18a)	0.7784	0.2237	0.7418	5.4757
H(18b)	0.6663	0.1904	0.6553	5.4757
H(19a)	0.6269	0.1794	0.8426	5.1061
H(19b)	0.6090	0.2370	0.8569	5.1061
H(20a)	0.4141	0.1752	0.7072	3.5074
H(20b)	0.3812	0.2035	0.8168	3.5074
H(21a)	0.0274	0.2835	0.6280	3.6907
H(22a)	0.2221	0.2877	0.8400	4.1801
H(22b)	0.0801	0.2549	0.8099	4.1801
H(23a)	-0.0663	0.3227	0.7955	6.1466
H(23b)	0.0484	0.3280	0.9145	6.1466
H(24a)	0.0107	0.4029	0.8113	5.1659
H(24b)	0.1754	0.3862	0.8352	5.1659
H(25a)	0.1097	0.4108	0.6382	5.1582
H(25b)	-0.0256	0.3747	0.6169	5.1582
H(26a)	0.2686	0.3447	0.6647	5.1563
H(26b)	0.1578	0.3370	0.5445	5.1563

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 4.V.5: Bond distances (Å) for [Te($S_2CN(CH(CH_2)_5)_2)_2$]

Te — S(1)	2.506(3)	C(6) — C(7)	1.52(1)
Te — S(2)	2.835(3)	C(8) — C(9)	1.49(1)
Te — S(3)	2.516(3)	C(8) — C(13)	1.49(1)
Te — S(4)	2.854(3)	C(9) — C(10)	1.48(1)
S(1) — C(1)	1.734(9)	C(10) — C(11)	1.54(2)
S(2) — C(1)	1.70(1)	C(11) — C(12)	1.50(2)
S(3) — C(14)	1.735(9)	C(12) — C(13)	1.50(2)
S(4) — C(14)	1.70(1)	C(15) — C(16)	1.50(1)
N(1) — C(1)	1.34(1)	C(15) — C(20)	1.51(1)
N(1) — C(2)	1.49(1)	C(16) — C(17)	1.54(1)
N(1) — C(8)	1.49(1)	C(17) — C(18)	1.48(2)
N(2) — C(14)	1.34(1)	C(18) — C(19)	1.53(1)
N(2) — C(15)	1.50(1)	C(19) — C(20)	1.51(1)
N(2) — C(21)	1.52(1)	C(21) — C(22)	1.49(1)
C(2) — C(3)	1.49(1)	C(21) — C(26)	1.49(1)
C(2) — C(7)	1.50(1)	C(22) — C(23)	1.52(1)
C(3) — C(4)	1.54(2)	C(23) — C(24)	1.51(2)
C(4) — C(5)	1.49(2)	C(24) — C(25)	1.57(1)
C(5) — C(6)	1.51(2)	C(25) — C(26)	1.52(1)

Table 4.V.6: Bond angles (deg.) for [Te($S_2CN(CH(CH_2)_5)_2)_2$]

S(1) — Te — S(2)	65.85(8)	N(1) — C(8) — C(9)	116.2(9)
S(1) — Te — S(3)	81.34(8)	N(1) — C(8) — C(13)	111.1(8)
S(1) — Te — S(4)	146.72(8)	C(9) — C(8) — C(13)	113.6(8)
S(2) — Te — S(3)	147.14(8)	C(8) — C(9) — C(10)	112(1)
S(2) — Te — S(4)	147.41(8)	C(9) — C(10) — C(11)	112.7(9)
S(3) — Te — S(4)	65.38(8)	C(10) — C(11) — C(12)	110(1)
Te — S(1) — C(1)	93.6(3)	C(11) — C(12) — C(13)	116(1)
Te — S(2) — C(1)	83.5(3)	C(8) — C(13) — C(12)	111.2(9)
Te — S(3) — C(14)	94.4(3)	S(3) — C(14) — S(4)	116.2(5)
Te — S(4) — C(14)	84.0(3)	S(3) — C(14) — N(2)	120.5(7)
C(1) — N(1) — C(2)	124.2(8)	S(4) — C(14) — N(2)	123.3(7)
C(1) — N(1) — C(8)	119.4(8)	N(2) — C(15) — C(16)	113.2(8)
C(2) — N(1) — C(8)	115.6(8)	N(2) — C(15) — C(20)	113.1(8)
C(14) — N(2) — C(15)	124.8(8)	C(16) — C(15) — C(20)	114.6(8)
C(14) — N(2) — C(21)	119.0(7)	C(15) — C(16) — C(17)	110.7(9)
C(15) — N(2) — C(21)	116.1(7)	C(16) — C(17) — C(18)	112.2(9)
S(1) — C(1) — S(2)	116.4(5)	C(17) — C(18) — C(19)	111.6(9)
S(1) — C(1) — N(1)	120.3(7)	C(18) — C(19) — C(20)	111.4(9)
S(2) — C(1) — N(1)	123.3(7)	C(15) — C(20) — C(19)	111.4(9)
N(1) — C(2) — C(3)	114.0(9)	N(2) — C(21) — C(22)	112.3(8)
N(1) — C(2) — C(7)	115.0(8)	N(2) — C(21) — C(26)	112.2(8)
C(3) — C(2) — C(7)	116(1)	C(22) — C(21) — C(26)	115.1(8)
C(2) — C(3) — C(4)	111(1)	C(21) — C(22) — C(23)	111.1(9)
C(3) — C(4) — C(5)	110.7(9)	C(22) — C(23) — C(24)	113.9(8)
C(4) — C(5) — C(6)	115(1)	C(23) — C(24) — C(25)	110.5(9)
C(5) — C(6) — C(7)	109(1)	C(24) — C(25) — C(26)	109.6(9)

Table 4.V.6: Cont.

C(2) — C(7) — C(6)	111(1)	C(21) — C(26) — C(25)	112.0(9)

Table 4.V.7: Observed and calculated structure factors for $[Te(S_2CN(CH(CH_2)_5)_2)_2]$

h	k	l	Fo	Fc	sigF2
0	0	2	1498	1426	99
0	0	4	565	549	99
0	0	6	421	399	99
0	0	8	645	615	99
0	0	10	525	535	99
0	0	12	379	375	99
0	0	14	324	333	99
0	1	2	392	374	99
0	1	3	490	474	99
0	1	4	184	168	99
0	1	5	970	981	99
0	1	6	368	374	99
0	1	7	-114	10	99
0	1	8	976	956	99
0	1	9	651	630	99
0	1	10	266	213	99
0	1	11	294	299	99
0	1	12	77	239	99
0	1	13	-213	25	99
0	2	2	1532	1523	99
0	2	3	1300	1317	99
0	2	4	143	167	99
0	2	5	802	813	99
0	2	6	514	487	99
0	2	7	1124	1140	99
0	2	8	217	180	99
0	2	9	437	438	99
0	2	10	168	270	99
0	2	11	267	239	99
0	2	12	28	184	99
0	2	13	352	351	99
0	3	3	-412	513	99
0	3	4	-274	382	99
0	3	5	-256	90	99
0	3	6	-82	146	99
0	3	7	-106	112	99
0	3	8	-205	362	99
0	3	9	196	575	99
0	3	10	17	30	99
0	3	11	150	426	99
0	3	12	-90	241	99
0	3	13	-130	138	99
0	4	+10	570	810	99
0	4	0	-145	382	99
0	4	1	-164	130	99
0	4	3	-199	261	99
0	4	5	-414	667	99
0	4	6	-355	626	99
0	4	7	-126	109	99
0	4	8	-89	327	99
0	4	9	-57	269	99
0	4	11	-232	9	99
0	4	12	157	180	99
0	4	13	117	141	99
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0	5	5	-211	700	99
0	5	7	163	292	99
0	5	8	223	733	99
0	5	9	252	351	99
0	5	10	-152	46	99
0	5	11	165	397	99
0	5	12	173	414	99
0	5	13	-166	122	99
0	6	2	-270	254	99
0	6	4	-661	844	99
0	6	5	-116	56	99
0	6	6	130	78	99
0	6	7	181	632	99
0	6	8	-69	154	99
0	6	9	160	413	99
0	6	10	160	158	99
0	6	11	381	576	99
0	6	12	-20	51	99
0	6	13	203	352	99
0	7	-9	434	550	99
0	7	1	82	107	99
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0	7	5	-375	854	99
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0	7	8	334	528	99
0	7	10	-45	42	99
0	7	11	240	345	99
0	7	12	357	498	99
0	7	13	-182	70	99
0	8	-6	-212	1084	99
0	8	0	-450	846	99
0	8	1	-123	78	99
0	8	2	97	200	99
0	8	3	129	534	99
0	8	5	165	677	99
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0	9	10	106	209	99
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0	10	6	151	94	99
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0	10	8	214	250	99
0	10	9	287	385	99
0	10	10	209	193	99
0	10	11	322	363	99
0	10	12	-67	84	99
0	11	-2	-547	1069	99
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0	11	3	63	256	99
0	11	4	-120	197	99
0	11	5	519	575	99
0	11	6	486	541	99
0	11	7	234	249	99
0	11	8	1058	1144	99
0	11	9	470	490	99
0	11	10	96	228	99
0	11	11	-153	61	99
0	11	12	355	355	99
0	12	0	1976	1954	99
0	12	1	187	211	99
0	12	2	441	441	99
0	12	3	408	414	99
0	12	4	382	394	99
0	12	5	239	282	99
0	12	6	1109	1113	99
0	12	7	101	142	99
0	12	8	186	141	99
0	12	9	206	175	99
0	12	10	371	386	99
0	12	11	-22	155	99
0	12	12	-123	28	99
0	13	1	916	901	99
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0	13	4	176	99	99
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0	13	9	542	530	99
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0	13	11	98	123	99
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0	14	9	271	237	99
0	14	10	176	150	99
0	14	11	275	218	99
0	14	12	200	157	99
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0	15	2	798	760	99
0	15	3	335	317	99
0	15	4	732	705	99
0	15	5	842	816	99
0	15	6	320	317	99
0	15	7	101	59	99
0	15	8	437	372	99
0	15	9	-46	78	99
0	15	10	-156	47	99
0	15	11	-197	113	99
0	15	12	352	383	99
0	16	0	793	790	99
0	16	1	581	576	99
0	16	2	272	275	99
0	16	3	277	254	99
0	16	4	1660	1580	99
0	16	5	710	682	99
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0	16	8	105	34	99
0	16	9	167	35	99
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0	16	11	-160	37	99
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0	17	7	-142	15	99
0	17	8	167	118	99
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0	17	10	-117	82	99
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0	18	7	354	319	99
0	18	8	-45	103	99
0	18	9	164	180	99
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0	18	11	305	246	99
0	19	1	129	64	99
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0	19	4	238	219	99
0	19	5	432	374	99
0	19	6	372	338	99
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0	19	8	-214	259	99
0	19	9	164	171	99
0	19	10	147	52	99
0	19	11	184	146	99
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0	20	1	116	132	99
0	20	2	-176	125	99
0	20	3	307	260	99
0	20	4	612	652	99
0	20	5	469	478	99
0	20	6	371	348	99
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0	21	5	372	333	99
0	21	6	-138	46	99
0	21	7	217	53	99
0	21	8	-75	111	99
0	21	9	469	415	99
0	21	10	-58	174	99
0	22	0	946	925	99
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0	24	-8	533	524	99
0	24	-7	665	662	99
0	24	-6	729	716	99
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0	24	4	388	399	99
0	24	5	364	329	99
0	24	6	820	780	99
0	24	7	267	250	99
0	24	8	128	79	99
0	24	9	354	342	99
0	24	10	562	578	99
0	24	11	439	415	99
0	24	12	161	8	99
0	24	13	147	132	99
0	24	-13	208	214	99
0	24	-12	-166	114	99
0	24	-11	404	371	99
0	24	-10	552	534	99
0	24	-9	647	635	99
0	24	-8	-141	78	99
0	24	-7	178	171	99
0	24	-6	856	836	99
0	24	-5	2063	1965	99
0	24	-4	365	329	99
0	24	-3	443	426	99
0	24	-2	794	791	99
0	24	-1	1402	1382	99
0	24	0	85	16	99
0	24	1	719	736	99
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0	24	3	888	821	99
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0	24	7	545	501	99
0	24	8	-90	169	99
0	24	9	346	324	99
0	24	10	342	285	99
0	24	11	305	246	99
0	24	12	129	64	99
0	24	13	557	611	99
0	24	14	238	219	99
0	24	15	432	374	99
0	24	16	372	338	99
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0	24	18	-214	259	99
0	24	19	164	171	99
0	24	20	147	52	99
0	24	21	184	146	99
0	24	22	908	833	99
0	24	23	116	132	99
0	24	24	-176	125	99
0	24	25	307	260	99
0	24	26	612	652	99
0	24	27	469	478	99
0	24	28	371	348	99
0	24	29	117	150	99
0	24	30	122	110	99
0	24	31	118	175	99
0	24	32	527	500	99
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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8	2	6	261	165	99	8	5	4	494	482	99	8	8	3	-60	25	99	8	12	-10	89	80	99	8	15	0	435	360	99
8	2	7	-105	165	99	8	5	5	334	291	99	8	8	4	292	290	99	8	12	-9	137	42	99	8	15	1	-17	111	99
8	3	-11	185	39	99	8	5	6	-267	172	99	8	8	5	100	99	99	8	12	-8	77	148	99	8	15	2	249	234	99
8	3	-10	223	151	99	8	5	7	370	342	99	8	8	6	411	422	99	8	12	-7	-136	124	99	8	15	3	157	134	99
8	3	-9	315	354	99	8	6	-11	200	242	99	8	9	-10	236	46	99	8	12	-6	256	294	99	8	15	4	-275	225	99
8	3	-8	189	27	99	8	6	-10	103	98	99	8	9	-9	175	111	99	8	12	-5	-47	81	99	8	15	5	315	217	99
8	3	-7	-49	36	99	8	6	-9	157	30	99	8	9	-8	212	147	99	8	12	-4	873	846	99	8	16	-8	310	399	99
8	3	-6	631	649	99	8	6	-8	220	85	99	8	9	-7	-177	22	99	8	12	-3	237	152	99	8	16	-7	163	158	99
8	3	-5	-159	17	99	8	6	-7	341	319	99	8	9	-6	284	280	99	8	12	-2	380	379	99	8	16	-6	174	71	99
8	3	-4	-101	19	99	8	6	-6	-187	98	99	8	9	-5	372	377	99	8	12	-1	93	115	99	8	16	-5	-24	41	99
8	3	-3	639	642	99	8	6	-5	527	516	99	8	9	-4	-159	32	99	8	12	0	263	259	99	8	16	-4	516	498	99
8	3	-2	463	491	99	8	6	-4	264	310	99	8	9	-3	907	850	99	8	12	1	17	28	99	8	16	-3	-149	15	99
8	3	-1	258	290	99	8	6	-3	548	615	99	8	9	-2	527	507	99	8	12	2	334	237	99	8	16	-2	267	213	99
8	3	0	477	486	99	8	6	-2	-139	52	99	8	9	-1	-115	142	99	8	12	3	298	282	99	8	16	-1	204	275	99
8	3	1	401	567	99	8	6	-1	561	591	99	8	9	0	406	394	99	8	12	4	96	186	99	8	16	0	199	194	99
8	3	2	253	192	99	8	6	0	150	189	99	8	9	1	249	212	99	8	12	5	-282	64	99	8	16	1	124	10	99
8	3	3	347	334	99	8	6	1	225	255	99	8	9	2	284	266	99	8	13	-9	263	178	99	8	16	2	374	386	99
8	3	4	287	256	99	8	6	2	136	127	99	8	9	3	160	100	99	8	13	-8	-265	57	99	8	16	3	261	109	99
8	3	5	309	197	99	8	6	3	436	434	99	8	9	4	165	122	99	8	13	-7	332	234	99	8	16	4	30	94	99
8	3	6	-173	151	99	8	6	4	151	37	99	8	9	5	617	527	99	8	13	-6	306	333	99	8	17	-8	199	52	99
8	3	7	194	23	99	8	6	5	186	162	99	8	9	6	211	104	99	8	13	-5	427	497	99	8	17	-7	344	274	99
8	4	-11	-139	16	99	8	6	6	284	223	99	8	10	-10	175	35	99	8	13	-4	140	1	99	8	17	-6	189	102	99
8	4	-10	402	450	99	8	6	7	250	309	99	8	10	-9	179	184	99	8	13	-3	405	429	99	8	17	-5	143	196	99
8	4	-9	128	117	99	8	7	-11	298	163	99	8	10	-8	251	217	99	8	13	-2	266	267	99	8	17	-4	-30	193	99
8	4	-8	621	589	99	8	7	-10	-18	298	99	8	10	-7	369	311	99	8	13	-1	122	129	99	8	17	-3	164	158	99
8	4	-7	-135	52	99	8	7	-9	312	270	99	8	10	-6	218	86	99	8	13	0	235	177	99	8	17	-2	285	313	99
8	4	-6	253	174	99	8	7	-8	57	237	99	8	10	-5	535	478	99	8	13	1	697	572	99	8	17	-1	267	254	99
8	17	0	47	10	99	9	1	-10	-93	47	99	9	4	-3	436	407	99	9	7	5	-192	126	99	9	11	2	173	143	99
8	17	1	336	372	99	9	1	-9	279	229	99	9	4	-2	-57	2	99	9	8	-9	493	395	99	9	11	3	582	588	99
8	17	2	64	184	99	9	1	-8	540	561	99	9	4	-1	-86	131	99	9	8	-8	-113	38	99	9	11	4	207	80	99
8	17	3	284	226	99	9	1	-7	192	232	99	9	4	0	215	126	99	9	8	-7	-209	94	99	9	12	-8	221	115	99
8	17	4	218	222	99	9	1	-6	86	80	99	9	4	1	475	527	99	9	8	-6	263	184	99	9	12	-7	-223	19	99
8	18	-7	221	333	99	9	1	-5	222	272	99	9	4	2	266	120	99	9	8	-5	254	171	99	9	12	-6	182	64	99
8	18	-6	-320	31	99	9	1	-4	370	413	99	9	4	3	321	304	99	9	8	-4	-96	44	99	9	12	-5	566	451	99
8	18	-5	335	327	99	9	1	-3	316	342	99	9	4	4	-143	177	99	9	8	-3	179	382	99	9	12	-4	-67	115	99
8	18	-4	199	31	99	9	1	-2	195	249	99	9	4	5	127	226	99	9	8	-2	86	83	99	9	12	-3	565	551	99
8	18	-3	544	531	99	9	1	-1	390	410	99	9	5	-10	252	192	99	9	8	-1	-144	53	99	9	12	-2	241	38	99
8	18	-2	-130	145	99	9	1	0	488	547	99	9	5	-9	198	73	99	9	8	0	-188	155	99	9	12	-1	237	240	99
8	18	-1	232	211	99	9	1	1	-60	47	99	9	5	-8	402	372	99	9	8	1	561	547	99	9	12	0	-199	117	99
8	18	0	214	186	99	9	1	2	212	112	99	9	5	-7	208	59	99	9	8	2	203	226	99	9	12	1	374	401	99
8	18	1	356	306	99	9	1	3	234	203	99	9	5	-6	-102	42	99	9	8	3	265	273	99	9	12	2	122	27	99
8	18	2	230	137	99	9	1	4	163	111	99	9	5	-5	308	272	99	9	8	4	178	217	99	9	12	3	-285	18	99
8	18	3	216	239	99	9	1	5	169	164	99	9	5	-4	408	409	99	9	8	5	616	491	99	9	13	-8	288	209	99
8	19	-7	191	198	99	9	2	-10	346	372	99	9	5	-3	311	282	99	9	9	-9	150	21	99	9	13	-7	327	327	99
8	19	-6	371	307	99	9	2	-9	178	132	99	9	5	-2	-82	62	99	9	9	-8	206	75	99	9	13	-6	366	320	99
8	19	-5	73	89	99	9	2	-8	191	51	99	9	5	-1	445	430	99	9	9	-7	310	306	99	9	13	-5	340	251	99
8	19	-4	-219	164	99	9	2	-7	-250	27	99	9	5	0	380	331	99	9	9	-6	268	226	99	9	13	-4	205	218	99
8	19	-3	236	176	99	9	2	-6	534	553	99	9	5	1	305	322	99	9	9	-5	94	85	99	9	13	-3	108	44	99
8	19	-2	344	379	99	9	2	-5	68	10	99	9	5	2	359	325	99	9	9	-4	427	466	99	9	13	-2	456	443	99
8	19	-1	157	91	99	9	2	-4	131	79	99	9	5	3	323	361	99	9	9	-3	209	133	99	9	13	-1	-99	75	99
8	19	0	75	107	99	9	2	-3	-34	37	99	9	5	4	68	66	99	9	9	-2	368	384	99	9	13	0	307	236	99
8	19	1	-83	74	99	9	2	-2	592	582	99	9	5	5	-147	14	99	9	9	-1	477	463	99	9	13	1	174	158	99
8	19	2	-118	10	99	9	2	-1	111	41	99	9	6	-10	285	208	99	9	9	0	104	14	99	9	13	2	-202	298	99
8	19	3	-207	47	99	9	2	0	374	388	99	9	6	-9	160	34	99	9	9	1	-98	87	99	9	13	3	64	281	99
8	20	-6	188	18	99	9	2	1	208	141	99	9	6	-8	296	314	99	9	9	2	317	340	99	9	14	-7	64	27	99
8	20	-5	117	126	99	9	2	2	148	204	99	9	6	-7	-120	26	99	9	9	3	240	80	99	9	14	-6	429	489	99
8	20	-4	340	285	99	9	2	3	112	65	99	9	6	-6	424	366	99	9	9	4	176	124	99	9	14	-5	-162	33	99
8	20	-3	167	21	99	9	2	4	319	276	99	9	6	-5	219	231	99	9	10	-9	108	73	99	9	14	-4	-234	138	99
8	20	-2	168	69	99	9	2	5	-110	34	99	9	6	-4	282	231	99	9	10	-8	279	355	99	9	14	-3	92	80	99
8	20	-1	53	111	99	9	3	-10	-152	214	99	9	6	-3	297	235	99	9	10	-7	-106	174	99	9	14	-2	301	262	99
8	20	0	265	213	99	9	3	-9	258	200	99	9	6	-2	414	434	99	9	10	-6	416	370	99	9	14	-1	126	8	99
8	20	1	-159	57	99	9	3	-8	333	304	99	9	6	-1	135	88	99	9	10	-5	176	161	99	9	14	0	10	226	99
8	20	2	271	347	99	9	3	-7	372	457	99	9	6	0	588	658	99	9	10	-4	190	221	99	9	14	1	269	317	99
8	21	-5	292	278	99	9	3	-6	-156	21	99	9	6	1	139	100	99	9	10	-3	309	269	99	9	14	2	377	349	99
8	21	-4	141	73	99	9	3	-5	236	237	99	9	6	2	248	148	99	9	10	-2	-67	159	99	9	14	3	185	96	99
8	21	-3	280	276	99	9	3	-4	429	369	99	9	6	3	144	54	99	9	10	-1	100	112	99	9	15	-7	347	379	99
8	21	-2	192	224	99	9	3	-3	330	283	99	9	6	4	406	401	99	9	10	0	462	410	99	9	15	-6	130	114	99
8	21	-1	-151	271	99	9	3	-2	225	140	99	9	6	5	120	32	99	9	10	1	217	80	99	9	15	-5	259	60	99
8	21	0	298	310	99	9	3	-1	484	550	99	9	7	-9	56	103	99	9	10	2	305	280	99	9	15	-4	434	434	99
8	21	1	262	285	99	9	3	0	-192	213	99	9	7	-8	-69	259	99	9	10	3	215	17	99	9	15	-3	-134	17	99
8	22	-4	280	234	99	9	3	1	172	152	99	9	7	-7	300	331	99	9	10	4	615	612	99	9	15	-2	137	185	99
8	22	-3	30	21	99	9	3	2	433	433	99	9	7	-6	-79	112	99	9	11	-9	-156	5	99	9	15	-1	605	502	99
8	22	-2	92	113	99	9	3	3	99	21	99	9	7	-5	433	421	99	9	11	-8	248	253	99	9	15	0	112	18	99
8	22	-1	376	346	99	9	3	4	36	67	99	9	7	-4	-90	112	99	9	11	-7	321	249	99	9	15	1	66	34	99
9	0	-9	295	277	99	9	3	5	201	193	99	9	7	-3	164	107	99	9	11	-6	202	149	99	9	15	2	111	12	99
9	0	-7	478	448	99	9	4	-10	252	41	99	9	7	-2	195	23	99	9	11	-5	262	306	99	9	16	-6	199	29	99
9	0	-5	382	477	99	9	4	-9	548	570	99	9	7	-1	337	281	99	9	11	-4	299	223	99	9	16	-5	402	395	99
9	0	-3	586	599	99	9	4	-8	-96	45	99	9	7	0	246	297	99	9	11	-3	-194	13	99	9	16	-4	289	171	99
9	0	-1	90	176	99	9	4	-7	294	296	99	9	7	1	132	129	99	9	11	-2	439	353	99	9	16	-3	290	359	99
9	0	1	555	520	99	9	4	-6	338	274	99	9	7	2	548	485	99	9	11	-1	230	222	99	9	16	-2	82	18	99
9	0	3	-126	131	99	9	4	-5	246	185	99	9	7	3	448	466	99	9	11	0	196	233	99	9	16	-1	-153	38	99
9	0	5	144	113	99	9	4	-4	178	0	99	9	7	4	238	55	99	9	11	1	-40	48	99	9	16	0	292	316	99

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
9	16	1	452	383	99	10	3	-1	264	226	99	10	8	-3	154	104	99	11	1	-2	380	395	99						
9	16	2	-195	40	99	10	3	0	253	248	99	10	8	-2	-93	62	99	11	1	-1	269	258	99						
9	17	-6	158	115	99	10	3	1	109	107	99	10	8	-1	-94	99	99	11	2	-5	340	300	99						
9	17	-5	330	317	99	10	3	2	115	140	99	10	8	0	229	186	99	11	2	-4	279	260	99						
9	17	-4	371	382	99	10	3	3	188	13	99	10	8	1	47	64	99	11	2	-3	-102	55	99						
9	17	-3	141	131	99	10	4	-8	164	267	99	10	8	2	462	518	99	11	2	-2	73	255	99						
9	17	-2	-107	11	99	10	4	-7	129	161	99	10	9	-7	198	159	99	11	2	-1	-97	87	99						
9	17	-1	176	21	99	10	4	-6	111	91	99	10	9	-6	-109	178	99	11	3	-5	-173	217	99						
9	17	0	452	363	99	10	4	-5	-183	166	99	10	9	-5	129	190	99	11	3	-4	520	487	99						
9	17	1	-174	49	99	10	4	-4	421	450	99	10	9	-4	248	125	99	11	3	-3	-94	2	99						
9	18	-5	-278	7	99	10	4	-3	180	192	99	10	9	-3	226	251	99	11	3	-2	300	211	99						
9	18	-4	-162	82	99	10	4	-2	320	198	99	10	9	-2	276	280	99	11	3	-1	347	330	99						
9	18	-3	81	54	99	10	4	-1	166	229	99	10	9	-1	-238	20	99	11	4	-4	-156	97	99						
9	18	-2	309	438	99	10	4	0	217	202	99	10	9	0	-15	99	99	11	4	-3	391	344	99						
9	18	-1	144	63	99	10	4	1	271	162	99	10	9	1	292	305	99	11	4	-2	-91	83	99						
9	18	0	-243	169	99	10	4	2	488	463	99	10	9	2	289	140	99	11	4	-1	46	110	99						
9	19	-3	400	442	99	10	4	3	-138	29	99	10	10	-7	343	429	99	11	5	-4	187	42	99						
9	19	-2	-237	53	99	10	5	-8	172	99	99	10	10	-6	200	39	99	11	5	-3	-87	174	99						
10	0	-8	314	299	99	10	5	-7	260	344	99	10	10	-5	196	270	99	11	5	-2	-347	61	99						
10	0	-6	180	102	99	10	5	-6	279	271	99	10	10	-4	120	131	99	11	6	-3	178	13	99						
10	0	-4	661	585	99	10	5	-5	248	78	99	10	10	-3	163	78	99												
10	0	-2	756	718	99	10	5	-4	175	189	99	10	10	-2	151	45	99												
10	0	0	343	325	99	10	5	-3	194	264	99	10	10	-1	324	238	99												
10	0	2	-137	24	99	10	5	-2	245	163	99	10	10	0	230	150	99												
10	1	-8	45	128	99	10	5	-1	295	304	99	10	10	1	-112	22	99												
10	1	-7	145	104	99	10	5	0	315	307	99	10	11	-6	314	280	99												
10	1	-6	-221	127	99	10	5	1	321	402	99	10	11	-5	-91	61	99												
10	1	-5	292	171	99	10	5	2	64	90	99	10	11	-4	186	80	99												
10	1	-4	-112	27	99	10	5	3	159	176	99	10	11	-3	165	187	99												
10	1	-3	577	558	99	10	6	-8	-135	147	99	10	11	-2	83	144	99												
10	1	-2	343	393	99	10	6	-7	124	170	99	10	11	-1	82	26	99												
10	1	-1	144	51	99	10	6	-6	154	107	99	10	11	0	139	77	99												
10	1	0	308	289	99	10	6	-5	424	521	99	10	11	1	168	222	99												
10	1	1	177	199	99	10	6	-4	-143	10	99	10	12	-6	230	6	99												
10	1	2	160	183	99	10	6	-3	138	248	99	10	12	-5	82	46	99												
10	1	3	256	29	99	10	6	-2	24	61	99	10	12	-4	329	354	99												
10	2	-8	218	10	99	10	6	-1	345	321	99	10	12	-3	-34	34	99												
10	2	-7	188	163	99	10	6	0	339	266	99	10	12	-2	375	296	99												
10	2	-6	206	212	99	10	6	1	285	312	99	10	12	-1	95	30	99												
10	2	-5	251	240	99	10	6	2	-166	44	99	10	12	0	289	267	99												
10	2	-4	237	120	99	10	7	-8	120	72	99	10	13	-5	164	171	99												
10	2	-3	-131	152	99	10	7	-7	212	241	99	10	13	-4	248	156	99												
10	2	-2	-107	44	99	10	7	-6	439	489	99	10	13	-3	326	298	99												
10	2	-1	590	679	99	10	7	-5	-32	185	99	10	13	-2	-151	146	99												
10	2	0	358	377	99	10	7	-4	9	124	99	10	13	-1	-136	32	99												
10	2	1	173	59	99	10	7	-3	197	89	99	10	13	0	249	121	99												
10	2	2	124	33	99	10	7	-2	63	151	99	10	14	-4	181	47	99												
10	2	3	307	288	99	10	7	-1	110	92	99	10	14	-3	-160	17	99												
10	3	-8	111	95	99	10	7	0	98	200	99	10	14	-2	186	31	99												
10	3	-7	123	17	99	10	7	1	-98	155	99	11	0	-5	208	214	99												
10	3	-6	321	389	99	10	7	2	129	83	99	11	0	-3	653	671	99												
10	3	-5	395	320	99	10	8	-7	-266	11	99	11	0	-1	142	211	99												
10	3	-4	247	264	99	10	8	-6	106	14	99	11	1	-5	69	171	99												
10	3	-3	332	405	99	10	8	-5	316	249	99	11	1	-4	247	187	99												
10	3	-2	429	444	99	10	8	-4	303	278	99	11	1	-3	115	76	99												

APPENDIX 5.I

$[Zn(S_2COnBu)_2]\cdot 75C_6H_6$

Table 5.I.1: Crystallographic Parameters for $[Zn(S_2COCH_2CH_2CH_2CH_3)2]\cdot 75C_6H_6$

Formula	$C_{10}H_{18}O_2S_4Zn\ 0.75C_6H_6$
Formula weight	363.88
Crystal morphology / colour	block / colourless
Crystal dimensions (mm)	0.13 x 0.16 x 0.32
Crystal system	triclinic
Space group	$P\bar{1}$ (#2)
a (Å)	12.885(5)
b (Å)	18.72(1)
c (Å)	8.564(3)
α (°)	96.54(4)
β (°)	102.48(3)
γ (°)	87.33(4)
V (Å^3)	2002(1)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.645
$F(000)$	878
Temperature (°K)	200
μ (cm^{-1})	16.45
θ limits, cell (°)	12.2 – 16.0
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 16, -24 – 24, -10 – 10
Range of transmission factors	0.975 – 1
Scan technique	ω:2θ
No. of data measured	9642
No. of unique data	9237
R_{amal}	0.096
No. of parameters	388
R (I≥0σ(I))	0.193
R_w (I≥0σ(I))	0.148
R (I≥3σ(I))	0.058
R_w (I≥3σ(I))	0.055
Maximum and minimum residual electron density (e Å^{-3})	1.95, -4.19

Table 5.I.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Zn(S_2COCH_2CH_2CH_2CH_3)_2]\cdot 75C_6H_6$

Atom	*x*	*y*	*z*
Zn(1)	0.25557(7)	-0.10520(5)	0.1382(1)
Zn(2)	0.16308(7)	0.18132(5)	0.0959(1)
S(1)	0.2317(2)	0.0156(1)	0.1111(2)
S(2)	0.2539(2)	0.1478(1)	0.3419(2)
S(3)	0.2265(2)	-0.1684(1)	0.3473(2)
S(4)	0.4264(2)	-0.1585(1)	0.2361(3)
S(5)	-0.0064(2)	0.1364(1)	-0.0022(2)
S(6)	-0.2049(2)	0.1705(1)	0.1168(2)
S(7)	0.2622(2)	0.1827(1)	-0.1074(2)
S(8)	0.1803(2)	0.3063(1)	0.0794(3)
O(1)	0.2973(4)	0.0162(3)	0.4166(5)
O(2)	0.4091(5)	-0.2381(3)	0.4516(7)
O(3)	-0.0162(4)	0.2211(3)	0.2500(6)
O(4)	0.2784(5)	0.3196(3)	-0.1464(7)
C(1)	0.2635(6)	0.0583(4)	0.3023(9)
C(2)	0.3183(6)	0.0498(4)	0.5850(8)
C(3)	0.3412(7)	-0.0106(5)	0.6960(9)
C(4)	0.4471(7)	-0.0445(5)	0.701(1)
C(5)	0.4742(7)	-0.0973(5)	0.834(1)
C(6)	0.3559(6)	-0.1906(4)	0.3517(9)
C(7)	0.3552(9)	-0.2697(6)	0.555(1)
C(8)	0.443(1)	-0.3166(6)	0.661(1)
C(9)	0.405(1)	-0.3471(8)	0.778(2)
C(10)	0.486(1)	-0.3979(8)	0.868(2)

Table 5.I.2: Cont.

Atom	*x*	*y*	*z*
C(11)	-0.0723(6)	0.1808(4)	0.1325(8)
C(12)	-0.0653(7)	0.2594(4)	0.3744(9)
C(13)	0.0236(7)	0.2959(5)	0.499(1)
C(14)	-0.0167(9)	0.3420(6)	0.633(1)
C(15)	-0.0725(9)	0.4090(6)	0.585(1)
C(16)	0.2440(6)	0.2717(4)	-0.0670(9)
C(17)	0.3398(8)	0.2938(6)	-0.267(1)
C(18)	0.365(1)	0.3599(8)	-0.340(2)
C(19)	0.298(1)	0.3989(9)	-0.419(2)
C(20)	0.327(1)	0.4610(7)	-0.496(1)
C(21)	0.759(1)	-0.5111(6)	0.959(2)
C(22)	0.856(1)	-0.5128(7)	1.046(2)
C(23)	0.9061(10)	-0.578(1)	1.062(2)
C(24)	0.856(2)	-0.6373(8)	0.987(3)
C(25)	0.757(2)	-0.6350(9)	0.903(2)
C(26)	0.7076(10)	-0.568(1)	0.885(2)
C(27)	0.9729(7)	-0.0603(5)	0.397(1)
C(28)	0.9896(7)	0.0037(6)	0.3395(10)
C(29)	0.9816(7)	-0.0655(5)	0.557(1)

Table 5.I.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Zn(S_2COCH_2CH_2CH_2CH_3)_2]\cdot 75C_6H_6$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn(1)	0.0413(6)	0.0411(6)	0.0435(6)	-0.0002(5)	0.0048(4)	0.0044(5)
Zn(2)	0.0414(6)	0.0445(6)	0.0400(6)	-0.0001(5)	0.0093(4)	0.0062(5)
S(1)	0.050(1)	0.038(1)	0.037(1)	0.0021(10)	0.0065(9)	0.0033(10)
S(2)	0.058(1)	0.043(1)	0.038(1)	-0.001(1)	0.000(1)	0.0000(10)
S(3)	0.049(1)	0.055(1)	0.054(1)	-0.001(1)	0.009(1)	0.016(1)
S(4)	0.038(1)	0.055(1)	0.073(2)	0.002(1)	0.007(1)	0.013(1)
S(5)	0.038(1)	0.059(1)	0.042(1)	-0.001(1)	0.0102(10)	-0.004(1)
S(6)	0.041(1)	0.057(1)	0.047(1)	-0.007(1)	0.014(1)	-0.008(1)
S(7)	0.057(1)	0.043(1)	0.055(1)	0.003(1)	0.026(1)	0.005(1)
S(8)	0.073(2)	0.038(1)	0.062(1)	0.008(1)	0.030(1)	0.007(1)
O(1)	0.044(3)	0.052(4)	0.033(3)	0.003(3)	-0.001(2)	0.010(3)
O(2)	0.075(5)	0.078(5)	0.074(5)	0.002(4)	0.020(4)	0.022(4)
O(3)	0.040(3)	0.046(3)	0.043(3)	-0.012(3)	0.011(3)	-0.013(3)
O(4)	0.082(5)	0.050(4)	0.080(4)	0.012(3)	0.050(4)	0.023(3)
C(1)	0.038(5)	0.049(5)	0.040(5)	-0.005(4)	0.002(4)	0.013(4)
C(2)	0.060(6)	0.063(6)	0.025(4)	0.000(5)	-0.004(4)	0.006(4)
C(3)	0.052(6)	0.079(7)	0.049(5)	-0.005(5)	0.001(4)	0.020(5)
C(4)	0.056(6)	0.059(6)	0.083(7)	0.005(5)	0.014(5)	0.031(5)
C(5)	0.047(6)	0.096(8)	0.104(8)	0.007(5)	0.014(5)	0.039(7)
C(6)	0.057(6)	0.044(5)	0.054(5)	0.004(4)	0.002(4)	0.014(4)
C(7)	0.110(9)	0.110(9)	0.067(7)	0.030(7)	0.046(7)	0.033(6)
C(8)	0.19(1)	0.12(1)	0.120(10)	0.114(9)	0.100(10)	0.095(8)
C(9)	0.16(1)	0.15(1)	0.13(1)	0.06(1)	0.03(1)	0.06(1)
C(10)	0.16(1)	0.19(2)	0.14(1)	0.06(1)	0.01(1)	0.09(1)

Table 5.I.3: Cont.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(11)	0.043(5)	0.043(5)	0.037(4)	0.005(4)	0.005(4)	0.007(4)
C(12)	0.064(6)	0.064(6)	0.048(5)	-0.010(5)	0.017(5)	-0.013(5)
C(13)	0.067(7)	0.091(8)	0.065(6)	0.000(6)	0.009(5)	-0.032(6)
C(14)	0.099(9)	0.110(10)	0.074(7)	-0.012(7)	0.022(6)	-0.033(7)
C(15)	0.14(1)	0.071(8)	0.095(8)	0.040(7)	0.024(7)	-0.003(7)
C(16)	0.040(5)	0.063(6)	0.045(5)	0.003(4)	0.011(4)	0.013(4)
C(17)	0.123(10)	0.082(8)	0.118(9)	0.024(7)	0.087(8)	0.033(7)
C(18)	0.25(2)	0.14(1)	0.21(2)	0.11(1)	0.20(2)	0.12(1)
C(19)	0.30(2)	0.14(1)	0.19(2)	0.12(1)	0.17(2)	0.11(1)
C(20)	0.20(2)	0.14(1)	0.13(1)	0.05(1)	0.06(1)	0.092(10)
C(21)	0.10(1)	0.064(9)	0.14(1)	0.009(8)	0.019(9)	0.010(8)
C(22)	0.12(1)	0.09(1)	0.12(1)	-0.022(9)	0.016(9)	-0.011(8)
C(23)	0.072(9)	0.18(2)	0.14(1)	0.03(1)	0.002(8)	0.07(1)
C(24)	0.22(2)	0.050(9)	0.18(2)	0.04(1)	0.14(2)	0.06(1)
C(25)	0.19(2)	0.09(1)	0.12(1)	-0.07(1)	0.07(1)	-0.05(1)
C(26)	0.077(10)	0.13(1)	0.16(1)	-0.03(1)	0.008(8)	0.01(1)
C(27)	0.090(7)	0.070(7)	0.043(6)	-0.025(6)	0.017(5)	-0.014(5)
C(28)	0.069(7)	0.096(8)	0.044(5)	0.000(6)	0.017(5)	0.029(6)
C(29)	0.083(7)	0.070(7)	0.051(6)	-0.004(5)	0.020(5)	-0.010(5)

Table 5.I.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Zn(S_2COCH_2CH_2CH_2CH_3)_2]\cdot 75C_6H_6$

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.2579	0.0776	0.6046	4.8473
H(2b)	0.3780	0.0800	0.6043	4.8473
H(3a)	0.2890	-0.0463	0.6584	5.7254
H(3b)	0.3370	0.0091	0.8018	5.7254
H(4a)	0.4989	-0.0082	0.7233	6.0802
H(4b)	0.4483	-0.0706	0.5995	6.0802
H(5a)	0.5445	-0.1185	0.8359	7.6224
H(5b)	0.4222	-0.1350	0.8111	7.6224
H(5c)	0.4727	-0.0710	0.9381	7.6224
H(7a)	0.2997	-0.2993	0.4936	8.6132
H(7b)	0.3265	-0.2334	0.6216	8.6132
H(8a)	0.5011	-0.2866	0.7124	12.3400
H(8b)	0.4677	-0.3541	0.5918	12.3400
H(9a)	0.3433	-0.3733	0.7269	13.8677
H(9b)	0.3874	-0.3096	0.8525	13.8677
H(10a)	0.4550	-0.4191	0.9448	15.3380
H(10b)	0.5484	-0.3712	0.9246	15.3380
H(10c)	0.5067	-0.4356	0.7923	15.3380
H(12a)	-0.0997	0.2266	0.4221	5.5878
H(12b)	-0.1156	0.2943	0.3297	5.5878
H(13a)	0.0714	0.2600	0.5446	7.4179
H(13b)	0.0601	0.3258	0.4468	7.4179
H(14a)	0.0425	0.3543	0.7181	9.1615
H(14b)	-0.0643	0.3143	0.6708	9.1615

Table 5.I.4: Cont.

Atom	x	y	z	B_{eq}
H(15a)	-0.0245	0.4388	0.5495	9.9803
H(15b)	-0.0969	0.4346	0.6759	9.9803
H(15c)	-0.1331	0.3975	0.4977	9.9803
H(17a)	0.2984	0.2605	-0.3498	9.2014
H(17b)	0.4051	0.2700	-0.2174	9.2014
H(18a)	0.4124	0.3425	-0.4113	16.5383
H(18b)	0.4036	0.3914	-0.2508	16.5383
H(19a)	0.2535	0.4189	-0.3460	17.6953
H(19b)	0.2569	0.3673	-0.5049	17.6953
H(20a)	0.3481	0.5010	-0.4141	14.1524
H(20b)	0.2659	0.4754	-0.5746	14.1524
H(20c)	0.3853	0.4468	-0.5482	14.1524
H(21)	0.7238	-0.4645	0.9481	9.6380
H(22)	0.8914	-0.4688	1.0966	10.3154
H(23)	0.9773	-0.5816	1.1280	12.1297
H(24)	0.8933	-0.6835	0.9949	12.6752
H(25)	0.7196	-0.6787	0.8553	12.4112
H(26)	0.6366	-0.5633	0.8196	11.6586
H(27)	0.9545	-0.1026	0.3210	6.4366
H(28)	0.9835	0.0074	0.2258	6.3743
H(29)	0.9754	-0.0618	0.4435	6.4544

Table 5.I.5: Bond distances (Å) for $[Zn(S_2COCH_2CH_2CH_2CH_3)_2]\cdot 75C_6H_6$

Zn(1) — S(1)	2.299(2)	O(4) — C(17)	1.46(1)
Zn(1) — S(3)	2.358(2)	C(2) — C(3)	1.53(1)
Zn(1) — S(4)	2.395(2)	C(3) — C(4)	1.47(1)
Zn(1) — S(6)*	2.354(2)	C(4) — C(5)	1.56(1)
Zn(2) — S(2)	2.317(2)	C(7) — C(8)	1.59(1)
Zn(2) — S(5)	2.323(2)	C(8) — C(9)	1.39(1)
Zn(2) — S(7)	2.375(2)	C(9) — C(10)	1.52(1)
Zn(2) — S(8)	2.383(2)	C(12) — C(13)	1.51(1)
S(1) — C(1)	1.711(7)	C(13) — C(14)	1.53(1)
S(2) — C(1)	1.673(8)	C(14) — C(15)	1.47(1)
S(3) — C(6)	1.694(8)	C(17) — C(18)	1.53(1)
S(4) — C(6)	1.656(8)	C(18) — C(19)	1.24(2)
S(5) — C(11)	1.698(7)	C(19) — C(20)	1.50(2)
S(6) — C(11)	1.702(7)	C(21) — C(22)	1.31(1)
S(7) — C(16)	1.676(8)	C(21) — C(26)	1.31(2)
S(8) — C(16)	1.696(8)	C(22) — C(23)	1.36(2)
O(1) — C(1)	1.318(7)	C(23) — C(24)	1.33(2)
O(1) — C(2)	1.480(8)	C(24) — C(25)	1.33(2)
O(2) — C(6)	1.362(8)	C(25) — C(26)	1.39(2)
O(2) — C(7)	1.43(1)	C(27) — C(28)	1.37(1)
O(3) — C(11)	1.292(7)	C(27) — C(29)	1.35(1)
O(3) — C(12)	1.457(8)	C(28) — C(29)	1.38(1)
O(4) — C(16)	1.329(8)		

* symmetry operation: *-x*, *-y*, *-z*

Table 5.I.6: Bond angles (deg.) for $[Zn(S_2COCH_2CH_2CH_2CH_3)_2]\cdot 75C_6H_6$

S(1) — Zn(1) — S(3)	128.72(8)	C(3) — C(4) — C(5)	111.0(7)
S(1) — Zn(1) — S(4)	122.51(8)	S(3) — C(6) — S(4)	123.7(5)
S(1) — Zn(1) — S(6)*	108.93(8)	S(3) — C(6) — O(2)	121.1(6)
S(3) — Zn(1) — S(4)	76.83(8)	S(4) — C(6) — O(2)	115.1(6)
S(3) — Zn(1) — S(6)*	112.58(8)	O(2) — C(7) — C(8)	105.2(8)
S(4) — Zn(1) — S(6)*	101.44(8)	C(7) — C(8) — C(9)	113(1)
S(2) — Zn(2) — S(5)	116.84(8)	C(8) — C(9) — C(10)	113(1)
S(2) — Zn(2) — S(7)	117.00(9)	S(5) — C(11) — S(6)	121.7(4)
S(2) — Zn(2) — S(8)	111.52(8)	S(5) — C(11) — O(3)	116.6(5)
S(5) — Zn(2) — S(7)	112.99(8)	S(6) — C(11) — O(3)	121.8(5)
S(5) — Zn(2) — S(8)	115.62(8)	O(3) — C(12) — C(13)	106.6(7)
S(7) — Zn(2) — S(8)	76.54(7)	C(12) — C(13) — C(14)	112.8(8)
Zn(1) — S(1) — C(1)	105.7(3)	C(13) — C(14) — C(15)	114.4(9)
Zn(2) — S(2) — C(1)	101.7(3)	S(7) — C(16) — S(8)	121.8(5)
Zn(1) — S(3) — C(6)	79.9(3)	S(7) — C(16) — O(4)	122.7(6)
Zn(1) — S(4) — C(6)	79.4(3)	S(8) — C(16) — O(4)	115.5(6)
Zn(2) — S(5) — C(11)	100.5(3)	O(4) — C(17) — C(18)	106.6(8)
Zn(1) — S(6)* — C(11)*	102.3(3)	C(17) — C(18) — C(19)	125(2)
Zn(2) — S(7) — C(16)	81.1(3)	C(18) — C(19) — C(20)	123(2)
Zn(2) — S(8) — C(16)	80.5(3)	C(22) — C(21) — C(26)	124(1)
C(1) — O(1) — C(2)	117.6(6)	C(21) — C(22) — C(23)	118(1)
C(6) — O(2) — C(7)	119.6(7)	C(22) — C(23) — C(24)	119(1)
C(11) — O(3) — C(12)	120.5(6)	C(23) — C(24) — C(25)	122(2)
C(16) — O(4) — C(17)	118.5(7)	C(24) — C(25) — C(26)	118(1)
S(1) — C(1) — S(2)	122.7(4)	C(21) — C(26) — C(25)	118(1)
S(1) — C(1) — O(1)	115.4(6)	C(28) — C(27) — C(29)	120.3(8)

Table 5.I.6: Cont.

S(2) — C(1) — O(1)	121.9(5)	C(27) — C(28) — C(29)	119.4(8)
O(1) — C(2) — C(3)	108.0(6)	C(27) — C(29) — C(28)	120.3(8)
C(2) — C(3) — C(4)	112.6(7)		

* symmetry operation: -*x*, -*y*, -*z*

Table 5.I.7: Observed and calculated structure factors for $[Zn(S_2COCH_2CH_2CH_2CH_3)_2]\cdot 75C_6H_6$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	2	450	448	99	0	3	-2	360	346	99	0	6	-10	-54	160	99	0	8	3	481	466	99	0	11	-2	141	139	99
0	0	3	52	87	99	0	3	-1	1408	1404	99	0	6	-9	295	341	99	0	8	4	218	213	99	0	11	-1	503	488	99
0	0	4	1400	1347	99	0	3	0	770	746	99	0	6	-8	128	208	99	0	8	5	824	797	99	0	11	0	528	505	99
0	0	5	741	757	99	0	3	1	631	686	99	0	6	-7	507	485	99	0	8	6	693	634	99	0	11	1	433	414	99
0	0	6	198	189	99	0	3	2	257	261	99	0	6	-6	482	489	99	0	8	7	277	252	99	0	11	2	140	158	99
0	0	7	64	68	99	0	3	3	384	389	99	0	6	-5	517	541	99	0	8	8	-180	91	99	0	11	3	386	364	99
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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16	7	-3	126	17	99
16	7	-2	132	39	99
16	7	-1	-126	43	99
16	8	-3	44	95	99
16	8	-2	184	172	99

APPENDIX 5.II

$[Zn(S_2CN(Et)Cy)_2]$

Table 5.II.1: Crystallographic Parameters for [Zn($S_2CN(CH_2CH_3)(CH(CH_2)_5)$)2]

Formula	$C_{18}H_{22}N_2S_4Zn$
Formula weight	470.08
Crystal morphology / colour	block / colourless
Crystal dimensions (mm)	0.08 x 0.16 x 0.34
Crystal system	monoclinic
Space group	$P2_1/c$ (#14)
a (Å)	8.618(5)
b (Å)	16.500(4)
c (Å)	16.242(2)
α (°)	90
β (°)	97.70(2)
γ (°)	90
V (Å^3)	2288(1)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.364
F(000)	992
Temperature (°K)	200
μ (cm^{-1})	14.43
θ limits, cell (°)	23.2 – 27.1
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 11, 0 – 21, -21 – 21
Range of transmission factors	0.935 – 1
Scan technique	ω:2θ
No. of data measured	5835
No. of unique data	5490
R_{amal}	0.072
No. of parameters	226
R (I≥0σ(I))	0.068
R_w (I≥0σ(I))	0.079
R (I≥3σ(I))	0.037
R_w (I≥3σ(I))	0.038
Maximum and minimum residual electron density (e Å^{-3})	0.70, -0.73

Table 5.II.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Zn(S_2CN(CH_2CH_3)(CH(CH_2)5))2]$

Atom	*x*	*y*	*z*
Zn	0.43506(4)	0.01132(2)	0.10287(2)
S(1)	0.4779(1)	0.14572(5)	0.14430(5)
S(2)	0.44108(9)	0.01037(5)	0.25663(5)
S(3)	0.22091(9)	-0.06387(5)	0.04022(5)
S(4)	0.6513(1)	-0.06083(4)	0.06431(5)
N(1)	0.4784(3)	0.1653(1)	0.3071(1)
N(2)	0.1209(3)	-0.0445(1)	-0.1203(1)
C(1)	0.4675(3)	0.1126(2)	0.2438(2)
C(2)	0.4898(4)	0.2536(2)	0.2923(2)
C(3)	0.6544(5)	0.2828(2)	0.2880(2)
C(4)	0.4608(4)	0.1381(2)	0.3923(2)
C(5)	0.5829(4)	0.1730(2)	0.4593(2)
C(6)	0.5613(5)	0.1369(3)	0.5437(2)
C(7)	0.3997(6)	0.1494(2)	0.5644(2)
C(8)	0.2766(5)	0.1195(3)	0.4966(2)
C(9)	0.2987(4)	0.1560(2)	0.4126(2)
C(10)	0.2196(3)	-0.0200(2)	-0.0556(2)
C(11)	-0.0034(3)	-0.1043(2)	-0.1082(2)
C(12)	0.0484(4)	-0.1917(2)	-0.1120(2)
C(13)	0.1191(3)	-0.0079(2)	-0.2038(2)
C(14)	-0.0007(4)	0.0610(2)	-0.2164(2)
C(15)	0.0012(4)	0.1004(2)	-0.3010(2)
C(16)	-0.0269(4)	0.0385(2)	-0.3703(2)
C(17)	0.0944(4)	-0.0288(2)	-0.3574(2)
C(18)	0.0936(4)	-0.0701(2)	-0.2733(2)

Table 5.II.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Zn(S_2CN(CH_2CH_3)(CH(CH_2)_5))_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn	0.0331(2)	0.0376(2)	0.0377(2)	-0.0024(2)	0.0051(1)	-0.0061(2)
S(1)	0.0712(6)	0.0336(5)	0.0361(4)	0.0005(4)	0.0104(4)	-0.0002(4)
S(2)	0.0444(5)	0.0340(4)	0.0366(4)	-0.0080(4)	0.0062(3)	-0.0033(3)
S(3)	0.0358(5)	0.0453(5)	0.0354(4)	-0.0065(4)	0.0025(3)	0.0045(4)
S(4)	0.0321(4)	0.0305(4)	0.0373(4)	-0.0014(3)	0.0035(3)	-0.0012(3)
N(1)	0.050(2)	0.034(1)	0.037(1)	-0.004(1)	0.009(1)	-0.004(1)
N(2)	0.026(1)	0.033(1)	0.038(1)	0.000(1)	0.003(1)	0.000(1)
C(1)	0.030(2)	0.038(2)	0.036(2)	0.001(1)	0.004(1)	-0.003(1)
C(2)	0.074(3)	0.033(2)	0.047(2)	0.004(2)	0.014(2)	-0.006(2)
C(3)	0.088(3)	0.037(2)	0.071(3)	-0.019(2)	0.026(2)	-0.007(2)
C(4)	0.060(2)	0.039(2)	0.034(2)	-0.007(2)	0.008(2)	-0.006(1)
C(5)	0.055(2)	0.074(3)	0.044(2)	-0.008(2)	0.000(2)	-0.003(2)
C(6)	0.100(4)	0.079(3)	0.043(2)	-0.020(3)	-0.009(2)	0.000(2)
C(7)	0.108(4)	0.069(3)	0.048(2)	-0.036(3)	0.026(2)	-0.017(2)
C(8)	0.088(3)	0.098(3)	0.056(3)	-0.040(3)	0.027(2)	-0.016(2)
C(9)	0.052(2)	0.089(3)	0.054(2)	-0.023(2)	0.007(2)	-0.007(2)
C(10)	0.026(1)	0.031(2)	0.035(1)	0.002(1)	0.004(1)	-0.003(1)
C(11)	0.030(2)	0.047(2)	0.043(2)	-0.009(1)	0.004(1)	-0.004(2)
C(12)	0.061(3)	0.041(2)	0.073(3)	-0.011(2)	0.014(2)	-0.006(2)
C(13)	0.027(2)	0.044(2)	0.031(1)	-0.002(1)	0.002(1)	0.003(1)
C(14)	0.033(2)	0.042(2)	0.047(2)	0.000(1)	0.005(1)	0.006(2)
C(15)	0.041(2)	0.054(2)	0.059(2)	0.002(2)	0.005(2)	0.017(2)
C(16)	0.042(2)	0.076(3)	0.039(2)	-0.003(2)	-0.001(2)	0.013(2)
C(17)	0.046(2)	0.071(3)	0.041(2)	0.001(2)	0.001(2)	-0.006(2)
C(18)	0.038(2)	0.054(2)	0.038(2)	0.003(2)	0.001(1)	-0.004(2)

Table 5.II.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Zn(S_2CN(CH_2CH_3)(CH(CH_2)5))2]^*$

Atom	x	y	z	B_{eq}
H(2a)	0.4274	0.2661	0.2411	4.8194
H(2b)	0.4501	0.2814	0.3362	4.8194
H(3a)	0.7184	0.2708	0.3388	6.0710
H(3b)	0.6952	0.2563	0.2436	6.0710
H(3c)	0.6533	0.3397	0.2789	6.0710
H(4a)	0.4733	0.0809	0.3936	4.2172
H(5a)	0.6846	0.1603	0.4467	5.4876
H(5b)	0.5710	0.2302	0.4612	5.4876
H(6a)	0.5819	0.0803	0.5427	7.2010
H(6b)	0.6335	0.1619	0.5854	7.2010
H(7a)	0.3892	0.1209	0.6142	6.9691
H(7b)	0.3840	0.2057	0.5725	6.9691
H(8a)	0.2834	0.0621	0.4932	7.4732
H(8b)	0.1762	0.1342	0.5097	7.4732
H(9a)	0.2844	0.2130	0.4146	6.1436
H(9b)	0.2236	0.1334	0.3708	6.1436
H(11a)	-0.0356	-0.0950	-0.0553	3.8016
H(11b)	-0.0896	-0.0960	-0.1503	3.8016
H(12a)	-0.0359	-0.2264	-0.1035	5.5153
H(12b)	0.0794	-0.2023	-0.1649	5.5153
H(12c)	0.1343	-0.2012	-0.0700	5.5153
H(13a)	0.2193	0.0155	-0.2055	3.2209
H(14a)	-0.1022	0.0399	-0.2131	3.8333

Table 5.II.4: Cont.

Atom	x	y	z	B_{eq}
H(14b)	0.0243	0.1005	-0.1742	3.8333
H(15a)	-0.0786	0.1405	-0.3090	4.8649
H(15b)	0.1003	0.1252	-0.3025	4.8649
H(16a)	-0.1284	0.0159	-0.3709	5.0196
H(16b)	-0.0202	0.0645	-0.4219	5.0196
H(17a)	0.1952	-0.0064	-0.3599	5.0233
H(17b)	0.0717	-0.0680	-0.4002	5.0233
H(18a)	0.1751	-0.1092	-0.2656	4.1626
H(18b)	-0.0044	-0.0961	-0.2721	4.1626

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 5.II.5: Bond distances (Å) for [Zn($S_2CN(CH_2CH_3)(CH(CH_2)_5)$)2]

Zn — S(1)	2.3324(9)	C(2) — C(3)	1.509(5)
Zn — S(2)	2.4910(8)	C(4) — C(5)	1.522(4)
Zn — S(3)	2.340(1)	C(4) — C(9)	1.507(5)
Zn — S(4)	2.366(1)	C(5) — C(6)	1.529(5)
S(1) — C(1)	1.720(3)	C(6) — C(7)	1.490(6)
S(2) — C(1)	1.718(3)	C(7) — C(8)	1.507(5)
S(3) — C(10)	1.715(3)	C(8) — C(9)	1.526(5)
S(4) — C(10)	1.755(3)	C(11) — C(12)	1.513(4)
N(1) — C(1)	1.340(3)	C(13) — C(14)	1.531(4)
N(1) — C(2)	1.483(4)	C(13) — C(18)	1.520(4)
N(1) — C(4)	1.482(4)	C(14) — C(15)	1.522(4)
N(2) — C(10)	1.324(3)	C(15) — C(16)	1.515(5)
N(2) — C(11)	1.489(4)	C(16) — C(17)	1.521(5)
N(2) — C(13)	1.482(3)	C(17) — C(18)	1.526(4)

Table 5.II.6: Bond angles (deg.) for [Zn($S_2CN(CH_2CH_3)(CH(CH_2)_5)$)2]

S(1) — Zn — S(2)	74.67(3)	N(1) — C(4) — C(5)	113.9(3)
S(1) — Zn — S(3)	135.62(4)	N(1) — C(4) — C(9)	111.0(3)
S(1) — Zn — S(4)	117.05(4)	C(5) — C(4) — C(9)	110.0(3)
S(2) — Zn — S(3)	109.99(3)	C(4) — C(5) — C(6)	109.7(3)
S(2) — Zn — S(4)	110.55(3)	C(5) — C(6) — C(7)	112.2(3)
S(3) — Zn — S(4)	102.80(4)	C(6) — C(7) — C(8)	112.2(3)
Zn — S(1) — C(1)	86.7(1)	C(7) — C(8) — C(9)	111.3(3)
Zn — S(2) — C(1)	81.8(1)	C(4) — C(9) — C(8)	110.4(3)
Zn — S(3) — C(10)	94.6(1)	S(3) — C(10) — S(4)*	117.8(2)
Zn — S(4) — C(10)*	99.7(1)	S(3) — C(10) — N(2)	121.2(2)
C(1) — N(1) — C(2)	120.8(3)	S(4)* — C(10) — N(2)	121.0(2)
C(1) — N(1) — C(4)	121.0(3)	N(2) — C(11) — C(12)	113.9(3)
C(2) — N(1) — C(4)	117.8(2)	N(2) — C(13) — C(14)	110.4(2)
C(10) — N(2) — C(11)	119.6(2)	N(2) — C(13) — C(18)	112.8(3)
C(10) — N(2) — C(13)	121.9(2)	C(14) — C(13) — C(18)	112.0(2)
C(11) — N(2) — C(13)	118.2(2)	C(13) — C(14) — C(15)	110.5(3)
S(1) — C(1) — S(2)	116.8(2)	C(14) — C(15) — C(16)	111.2(3)
S(1) — C(1) — N(1)	120.5(2)	C(15) — C(16) — C(17)	110.6(3)
S(2) — C(1) — N(1)	122.7(2)	C(16) — C(17) — C(18)	111.3(3)
N(1) — C(2) — C(3)	113.9(3)	C(13) — C(18) — C(17)	110.2(3)

* symmetry operation: 1-*x*, -*y*, -*z*.

Table 5.II.7: Observed and calculated structure factors for $[Zn(S_2CN(CH_2CH_3)(CH(CH_2)_5))_2]$

h	k	l	Fo	Fc	sigF2
0	0	4	594	638	99
0	0	6	1490	1589	99
0	0	8	372	377	99
0	0	10	46	44	99
0	0	12	381	387	99
0	0	14	323	312	99
0	0	16	-92	15	99
0	0	18	40	43	99
0	0	20	441	354	99
0	1	-5	1151	1200	99
0	1	3	214	218	99
0	1	4	76	86	91
0	1	6	920	991	99
0	1	7	382	427	99
0	1	8	226	244	99
0	1	9	96	100	99
0	1	10	-39	33	99
0	1	11	76	63	99
0	1	12	87	121	99
0	1	13	388	401	99
0	1	14	14	38	99
0	1	15	163	169	99
0	1	16	63	62	99
0	1	17	66	70	99
0	1	18	-7	7	99
0	1	19	359	326	99
0	1	20	-117	26	99
0	2	-8	515	529	99
0	2	-5	1005	1024	99
0	2	2	219	230	99
0	2	3	141	130	91
0	2	4	355	368	99
0	2	6	1253	1244	99
0	2	7	1043	1012	99
0	2	9	293	309	99
0	2	10	49	49	99
0	2	11	47	94	99
0	2	12	-26	13	99
0	2	13	254	279	99
0	2	14	439	450	99
0	2	15	81	104	99
0	2	16	-60	54	99
0	2	17	-62	29	99
0	2	18	119	119	99
0	2	19	130	122	99
0	2	20	214	173	99
0	3	1	1927	1935	99
0	3	2	310	309	99
0	3	3	694	700	99
0	3	4	735	725	99
0	3	5	618	637	99
0	3	6	680	676	99
0	3	7	1144	1106	99
0	3	8	206	216	99
0	3	9	253	252	99
0	3	10	107	92	99
0	3	11	588	609	99
0	3	12	265	261	99
0	3	13	627	642	99
0	3	14	-44	6	99
0	3	15	201	201	99
0	3	16	120	116	99
0	3	17	75	62	99
0	3	18	-77	60	99
0	3	19	347	319	99
0	3	20	-42	5	99
0	4	-7	934	912	99
0	4	0	573	559	99
0	4	1	246	226	99
0	4	2	586	603	99
0	4	3	869	856	99
0	4	4	222	226	99
0	4	5	348	347	99
0	4	6	955	955	99
0	4	8	344	366	99
0	4	9	51	60	99
0	4	10	444	453	99
0	4	11	115	97	99
0	4	12	4	47	99
0	4	13	374	348	99
0	4	14	402	401	99
0	4	15	56	19	99
0	4	16	56	24	99
0	4	17	66	40	99
0	4	18	136	111	99
0	4	19	37	62	99
0	4	20	125	105	99
0	5	-12	727	765	99
0	5	-9	702	724	99
0	5	1	595	617	99
0	5	2	416	412	99
0	5	3	491	476	99
0	5	4	24	5	99
0	5	5	488	493	99
0	5	6	925	894	99
0	5	7	223	204	99
0	5	8	544	558	99
0	5	10	89	83	99
0	5	11	551	555	99
0	5	13	259	270	99
0	5	14	55	70	99
0	5	15	127	119	99
0	5	16	99	115	99
0	5	17	-102	53	99
0	5	18	39	38	99
0	5	19	285	273	99
0	5	20	27	47	99
0	6	-4	962	971	99
0	6	0	1116	1141	99
0	6	1	151	146	99
0	6	2	163	143	99
0	6	3	-11	31	99
0	6	5	119	110	99
0	6	6	446	450	99
0	6	7	413	412	99
0	6	8	598	631	99
0	6	9	216	223	99
0	6	10	271	272	99
0	6	11	212	209	99
0	6	12	204	218	99
0	6	13	315	308	99
0	6	14	290	306	99
0	6	15	85	104	99
0	6	16	33	34	99
0	6	17	-18	56	99
0	6	18	73	67	99
0	6	19	63	8	99
0	6	20	241	233	99
0	7	1	387	365	99
0	7	2	267	265	99
0	7	3	718	737	99
0	7	4	54	95	99
0	7	5	580	581	99
0	7	6	685	674	99
0	7	7	532	550	99
0	7	8	264	277	99
0	7	9	532	526	99
0	7	10	98	93	99
0	7	11	348	349	99
0	7	12	239	246	99
0	7	13	-58	20	99
0	7	14	36	31	99
0	7	15	95	94	99
0	7	16	-36	10	99
0	7	17	186	194	99
0	7	18	66	68	99
0	7	19	125	135	99
0	8	0	647	646	99
0	8	1	924	925	99
0	8	2	659	663	99
0	8	3	301	298	99
0	8	4	348	366	99
0	8	5	244	233	99
0	8	6	337	323	99
0	8	7	225	216	99
0	8	8	778	774	99
0	8	9	58	28	99
0	8	10	423	438	99
0	8	11	120	131	99
0	8	12	315	320	99
0	8	13	61	93	99
0	8	14	252	249	99
0	8	15	16	51	99
0	8	16	67	54	99
0	8	17	43	32	99
0	8	18	63	45	99
0	8	19	171	189	99
0	9	1	-84	53	99
0	9	2	681	723	99
0	9	3	488	483	99
0	9	4	438	432	99
0	9	5	406	408	99
0	9	6	47	68	99
0	9	7	66	53	99
0	9	8	-65	78	99
0	9	9	528	524	99
0	9	10	81	86	99
0	9	11	500	485	99
0	9	12	21	94	99
0	9	13	233	234	99
0	9	14	-92	29	99
0	9	15	178	182	99
0	9	16	40	29	99
0	9	17	57	71	99
0	9	18	123	135	99
0	9	19	119	129	99
0	10	0	123	115	99
0	10	1	505	515	99
0	10	2	369	400	99
0	10	3	624	636	99
0	10	4	32	65	99
0	10	5	367	353	99
0	10	6	460	457	99
0	10	7	337	333	99
0	10	8	466	464	99
0	10	9	124	125	99
0	10	10	528	518	99
0	10	11	87	99	99
0	10	12	264	272	99
0	10	13	169	153	99
0	10	14	429	434	99
0	10	15	100	122	99
0	10	16	58	17	99
0	10	17	46	73	99
0	10	18	45	53	99
0	11	-18	127	117	99
0	11	1	126	139	99
0	11	2	788	782	99
0	11	3	52	30	99
0	11	4	600	604	99
0	11	5	517	505	99
0	11	6	376	365	99
0	11	7	-33	24	99
0	11	8	405	403	99
0	11	9	341	344	99
0	11	10	-81	5	99
0	11	11	393	390	99
0	11	12	267	265	99
0	11	13	235	238	99
0	11	14	36	115	99
0	11	15	143	159	99
0	11	16	169	184	99
0	11	17	-16	9	99
0	12	0	482	465	99
0	12	1	524	535	99
0	12	2	147	177	99
0	12	3	478	491	99
0	12	4	58	43	99
0	12	5	854	855	99
0	12	6	271	263	99
0	12	7	209	184	99
0	12	8	38	31	99
0	12	9	66	108	99
0	12	10	174	173	99
0	12	11	62	16	99
0	12	12	55	33	99
0	12	13	110	119	99
0	12	14	205	202	99
0	12	15	268	256	99
0	12	16	54	43	99
0	12	17	121	124	99
0	13	1	317	326	99
0	13	2	474	473	99
0	13	3	127	133	99
0	13	4	588	588	99
0	13	5	125	122	99
0	13	6	145	147	99
0	13	7	158	146	99
0	13	8	318	321	99
0	13	9	57	14	99
0	13	10	49	54	99
0	13	11	58	23	99
0	13	12	189	176	99
0	13	13	47	67	99
0	13	14	-70	51	99
0	13	15	-40	45	99
0	13	16	156	172	99
0	14	0	685	689	99
0	14	1	474	498	99
0	14	2	68	99	99
0	14	3	394	384	99
0	14	4	69	102	99
0	14	5	340	352	99
0	14	6	222	220	99
0	14	7	139	139	99
0	14	8	56	41	99
0	14	9	62	22	99
0	14	10	167	155	99
0	14	11	102	76	99
0	14	12	-65	10	99
0	14	13	35	56	99
0	14	14	72	24	99
0	14	15	46	88	99
0	15	-15	84	69	99
0	15	1	425	431	99
0	15	2	464	452	99
0	15	3	-34	44	99
0	15	4	267	260	99
0	15	5	-42	86	99
0	15	6	61	41	99
0	15	7	58	90	99
0	15	8	49	20	99
0	15	9	115	124	99
0	15	10	75	90	99
0	15	11	61	45	99
0	15	12	25	16	99
0	15	13	137	126	99
0	15	14	40	41	99
0	16	-14	104	105	99
0	16	0	410	401	99
0	16	1	398	408	99
0	16	2	130	143	99
0	16	3	178	182	99
0	16	4	163	155	99
0	16	5	55	8	99
0	16	6	325	304	99
0	16	7	319	314	99
0	16	8	-22	45	99
0	16	9	61	35	99
0	16	10	-13	25	99
0	16	11	50	30	99
0	16	12	-40	41	99
0	16	13	64	68	99
0	17	1	227	229	99
0	17	2	173	169	99
0	17	3	142	133	99
0	17	4	93	77	99
0	17	5	122	124	99
0	17	6	153	149	99
0	17	7	241	240	99
0	17	8	126	127	99
0	17	9	-60	23	99
0	17	10	79	88	99
0	17	11	-67	26	99
0	17	12	144	151	99
0	18	0	219	226	99
0	18	1	180	155	99
0	18	2	-78	10	99
0	18	3	111	89	99
0	18	4	156	162	99
0	18	5	108	101	99
0	18	6	172	184	99
0	18	7	296	302	99
0	18	8	-10	83	99
0	18	9	107	85	99
0	18	10	21	15	99
0	18	11	80	59	99
0	19	1	134	129	99
0	19	2	132	91	99
0	19	3	-57	23	99
0	19	4	-62	27	99
0	19	5	73	99	99
0	19	6	169	178	99
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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10	8	-6	133	131	99																								
10	8	-5	50	18	99																								
10	8	-4	-74	22	99																								
10	8	-3	-102	19	99																								
10	8	-2	172	155	99																								
10	8	-1	67	48	99																								
10	8	0	-55	72	99																								
10	8	1	-78	6	99																								
10	8	2	-45	41	99																								
10	9	-6	83	98	99																								
10	9	-5	-51	80	99																								
10	9	-4	-90	57	99																								
10	9	-3	47	125	99																								
10	9	-2	-51	91	99																								
10	9	-1	-78	71	99																								
10	9	0	-75	37	99																								
10	9	1	70	46	99																								
11	0	-6	114	53	99																								
11	0	-4	-27	12	99																								
11	0	-2	236	257	99																								
11	0	0	85	84	99																								
11	1	-6	-46	2	99																								
11	1	-5	80	45	99																								
11	1	-4	-118	9	99																								
11	1	-3	45	48	99																								
11	1	-2	94	47	99																								
11	1	-1	136	131	99																								
11	1	0	58	5	99																								

APPENDIX 5.III

$[Zn(S_2CNCy_2)_2]$

Table 5.III.1: Crystallographic Parameters for $[Zn(S_2CN(CH(CH_2)_5)_2)_2]$

Formula	$C_{26}H_{44}N_2S_4Zn$
Formula weight	578.27
Crystal morphology / colour	cube / colourless
Crystal dimensions (mm)	0.13 x 0.13 x 0.13
Crystal system	trigonal
Space group	$P3_221$ (#154)
a (Å)	10.264(10)
b (Å)	10.264(10)
c (Å)	23.630(9)
α (°)	90
β (°)	90
γ (°)	90
V (Å^3)	2154(5)
Z	3
$\rho_{calc.}$ (g cm^{-3})	1.343
$F(000)$	924
Temperature (°K)	110
μ (cm^{-1})	11.68
θ limits, cell (°)	10.6 – 15.0
θ limits, data (°)	3.0 – 25.0
hkl range	0 – 10, 0 – 7, 0 – 28
Range of transmission factors	0.899 – 1
Scan technique	ω:2θ
No. of data measured	2847
No. of unique data	1516
R_{amal}	0.122
No. of parameters	150
R (I≥0σ(I))	0.101
R_w (I≥0σ(I))	0.100
R (I≥3σ(I))	0.041
R_w (I≥3σ(I))	0.037
Maximum and minimum residual electron density (e Å^{-3})	1.16, -2.09

Table 5.III.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Zn(S_2CN(CH(CH_2)5)2)2]$

Atom	*x*	*y*	*z*
Zn	0.3474(1)	0	1/3
S(1)	0.3005(2)	0.1118(2)	0.24337(9)
S(2)	0.5542(2)	0.2458(2)	0.16401(8)
N(1)	0.5181(7)	0.4020(6)	0.2483(2)
C(1)	0.4645(9)	0.2707(8)	0.2223(3)
C(2)	0.6607(9)	0.5358(9)	0.2278(3)
C(3)	0.782(1)	0.6009(9)	0.2742(3)
C(4)	0.9266(9)	0.7336(9)	0.2505(3)
C(5)	0.8936(9)	0.8541(9)	0.2278(3)
C(6)	0.7751(9)	0.7889(9)	0.1814(3)
C(7)	0.6296(8)	0.6542(9)	0.2028(3)
C(8)	0.4401(9)	0.4342(9)	0.2953(3)
C(9)	0.2923(9)	0.4193(9)	0.2761(3)
C(10)	0.2256(9)	0.4690(9)	0.3235(3)
C(11)	0.208(1)	0.3823(10)	0.3768(3)
C(12)	0.3566(10)	0.3974(10)	0.3957(3)
C(13)	0.4246(8)	0.3448(9)	0.3499(3)

Table 5.III.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Zn(S_2CN(CH(CH_2)_5)_2)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn	0.0283(7)	0.0218(8)	0.0242(6)	0.0109(-)	-0.0029(3)	-0.0057(6)
S(1)	0.022(1)	0.019(1)	0.030(1)	0.005(1)	0.0047(9)	-0.0048(10)
S(2)	0.024(1)	0.022(1)	0.022(1)	0.008(1)	0.0058(10)	-0.001(1)
N(1)	0.016(4)	0.015(3)	0.018(3)	0.007(3)	0.004(3)	0.001(3)
C(1)	0.015(4)	0.022(4)	0.020(4)	0.010(4)	-0.001(4)	0.001(3)
C(2)	0.011(4)	0.016(4)	0.023(4)	0.003(4)	0.011(3)	0.005(4)
C(3)	0.032(6)	0.023(5)	0.033(4)	0.009(5)	-0.004(4)	0.006(4)
C(4)	0.019(5)	0.024(5)	0.033(5)	0.003(4)	-0.004(4)	0.006(4)
C(5)	0.020(5)	0.027(5)	0.032(5)	0.001(4)	-0.006(4)	0.000(4)
C(6)	0.028(5)	0.020(4)	0.021(4)	0.013(4)	0.004(4)	0.001(4)
C(7)	0.017(4)	0.020(5)	0.026(4)	0.000(4)	0.003(3)	0.011(4)
C(8)	0.030(5)	0.013(4)	0.024(4)	0.008(4)	0.006(4)	-0.007(4)
C(9)	0.021(5)	0.022(5)	0.023(4)	0.012(4)	0.004(4)	0.000(4)
C(10)	0.025(5)	0.026(5)	0.043(5)	0.012(4)	0.004(4)	-0.007(4)
C(11)	0.048(6)	0.029(5)	0.028(5)	0.019(5)	0.017(5)	0.007(4)
C(12)	0.037(6)	0.030(5)	0.023(4)	0.012(5)	0.001(4)	-0.001(4)
C(13)	0.018(5)	0.026(5)	0.024(4)	0.015(4)	-0.005(4)	-0.004(4)

Table 5.III.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Zn(S_2CN(CH(CH_2)_5)_2)_2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.6995	0.5011	0.1975	1.7771
H(3a)	0.8026	0.5236	0.2873	2.9238
H(3b)	0.7459	0.6353	0.3056	2.9238
H(4a)	1.0012	0.7764	0.2804	2.7329
H(4b)	0.9647	0.6985	0.2201	2.7329
H(5a)	0.9854	0.9371	0.2125	2.9154
H(5b)	0.8571	0.8902	0.2585	2.9154
H(6a)	0.7537	0.8659	0.1684	2.1413
H(6b)	0.8136	0.7568	0.1501	2.1413
H(7a)	0.5858	0.6877	0.2317	2.3436
H(7b)	0.5596	0.6104	0.1715	2.3436
H(8a)	0.5047	0.5394	0.3049	2.2256
H(9a)	0.2225	0.3152	0.2664	1.9927
H(9b)	0.3097	0.4821	0.2431	1.9927
H(10a)	0.2920	0.5752	0.3308	2.9594
H(10b)	0.1278	0.4522	0.3118	2.9594
H(11a)	0.1704	0.4202	0.4065	3.3134
H(11b)	0.1359	0.2768	0.3701	3.3134
H(12a)	0.3396	0.3370	0.4294	3.0546
H(12b)	0.4266	0.5021	0.4043	3.0546
H(13a)	0.3593	0.2381	0.3431	1.9960
H(13b)	0.5228	0.3633	0.3619	1.9960

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 5.III.5: Bond distances (Å) for [Zn($S_2CN(CH(CH_2)_5)_2)_2$]

Zn — S(1)	2.321(2)	C(4) — C(5)	1.53(1)
Zn — S(2)	2.349(2)	C(5) — C(6)	1.52(1)
S(1) — C(1)	1.731(7)	C(6) — C(7)	1.53(1)
S(2) — C(1)	1.746(7)	C(8) — C(9)	1.52(1)
N(1) — C(1)	1.325(8)	C(8) — C(13)	1.54(1)
N(1) — C(2)	1.501(9)	C(9) — C(10)	1.53(1)
N(1) — C(8)	1.499(9)	C(10) — C(11)	1.50(1)
C(2) — C(3)	1.54(1)	C(11) — C(12)	1.52(1)
C(2) — C(7)	1.52(1)	C(12) — C(13)	1.52(1)
C(3) — C(4)	1.53(1)		

Table 5.III.6: Bond angles (deg.) for $[Zn(S_2CN(CH(CH_2)5)2)2]$

S(1) — Zn — S(1)*	125.9(1)	C(3) — C(2) — C(7)	112.1(6)
S(1) — Zn — S(2)	77.83(8)	C(2) — C(3) — C(4)	109.9(6)
S(1) — Zn — S(2)*	122.97(7)	C(3) — C(4) — C(5)	109.7(7)
S(2) — Zn — S(2)*	137.0(1)	C(4) — C(5) — C(6)	110.4(6)
Zn — S(1) — C(1)	84.1(3)	C(5) — C(6) — C(7)	111.3(6)
Zn — S(2) — C(1)	82.9(3)	C(2) — C(7) — C(6)	110.6(7)
C(1) — N(1) — C(2)	120.0(6)	N(1) — C(8) — C(9)	111.8(6)
C(1) — N(1) — C(8)	124.9(6)	N(1) — C(8) — C(13)	113.3(7)
C(2) — N(1) — C(8)	114.9(6)	C(9) — C(8) — C(13)	113.0(6)
S(1) — C(1) — S(2)	115.1(4)	C(8) — C(9) — C(10)	109.8(6)
S(1) — C(1) — N(1)	122.6(6)	C(9) — C(10) — C(11)	111.1(7)
S(2) — C(1) — N(1)	122.3(6)	C(10) — C(11) — C(12)	111.5(7)
N(1) — C(2) — C(3)	112.0(6)	C(11) — C(12) — C(13)	111.7(6)
N(1) — C(2) — C(7)	111.0(7)	C(8) — C(13) — C(12)	108.1(6)

* symmetry operation: *x-y*, *-y*, 1/3-*z*

Table 5.III.7: Observed and calculated structure factors for $[Zn(S_2CN(CH(CH_2)_5)_2)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	6	80	44	99	0	2	21	165	199	99	0	4	21	-82	53	99	0	7	0	112	67	99	0	9	14	-114	48	99
0	0	9	1163	1157	99	0	2	22	-89	116	99	0	4	22	445	434	99	0	7	1	609	605	99	0	10	0	73	71	99
0	0	12	1508	1539	99	0	2	23	112	176	99	0	4	23	-58	44	99	0	7	2	92	95	99	0	10	1	91	60	99
0	0	15	1274	1255	99	0	2	24	-153	8	99	0	4	24	161	101	99	0	7	3	-180	66	99	0	10	2	78	58	99
0	0	18	720	716	99	0	2	25	-92	47	99	0	4	25	363	361	99	0	7	4	219	231	99	0	10	3	174	168	99
0	0	21	366	356	99	0	2	26	285	286	99	0	4	26	122	100	99	0	7	5	262	243	99	0	10	4	-203	59	99
0	0	24	534	531	99	0	2	27	173	194	99	0	5	0	-17	164	99	0	7	6	168	170	99	0	10	5	-123	51	99
0	0	27	572	566	99	0	3	0	408	423	99	0	5	1	221	240	99	0	7	7	51	37	99	0	10	6	306	326	99
0	1	3	619	612	99	0	3	1	928	921	99	0	5	2	282	298	99	0	7	8	-157	134	99	0	10	7	207	170	99
0	1	4	2104	2109	99	0	3	2	258	248	99	0	5	3	118	103	99	0	7	9	-52	27	99	0	10	8	173	180	99
0	1	5	395	383	99	0	3	3	518	532	99	0	5	4	125	130	99	0	7	10	175	145	99	0	10	9	-125	24	99
0	1	6	727	736	99	0	3	4	656	647	99	0	5	5	383	402	99	0	7	11	265	274	99	1	0	1	834	824	99
0	1	7	596	621	99	0	3	5	129	135	99	0	5	6	527	521	99	0	7	12	122	83	99	1	0	4	314	322	99
0	1	8	689	705	99	0	3	6	840	850	99	0	5	7	269	236	99	0	7	13	193	203	99	1	0	5	58	88	99
0	1	9	125	117	99	0	3	7	335	346	99	0	5	8	860	896	99	0	7	14	-85	17	99	1	0	6	448	431	99
0	1	10	877	867	99	0	3	8	177	178	99	0	5	9	392	395	99	0	7	15	245	277	99	1	0	7	608	593	99
0	1	11	190	183	99	0	3	9	1070	1091	99	0	5	10	392	384	99	0	7	16	233	243	99	1	0	8	255	282	99
0	1	12	65	31	99	0	3	10	171	180	99	0	5	11	939	964	99	0	7	17	200	156	99	1	0	9	801	786	99
0	1	13	1113	1110	99	0	3	11	-91	83	99	0	5	12	246	256	99	0	7	18	141	157	99	1	0	10	107	131	99
0	1	14	-101	35	99	0	3	12	514	523	99	0	5	13	318	312	99	0	7	19	230	173	99	1	0	11	1172	1157	99
0	1	15	100	70	99	0	3	13	567	558	99	0	5	14	541	524	99	0	7	20	87	30	99	1	0	12	205	201	99
0	1	16	761	748	99	0	3	14	76	32	99	0	5	15	-104	18	99	0	7	21	140	100	99	1	0	13	257	261	99
0	1	17	88	73	99	0	3	15	175	125	99	0	5	16	324	314	99	0	8	0	70	45	99	1	0	14	1029	1038	99
0	1	18	305	308	99	0	3	16	303	320	99	0	5	17	355	360	99	0	8	1	405	404	99	1	0	15	230	242	99
0	1	19	315	311	99	0	3	17	211	203	99	0	5	18	270	257	99	0	8	2	280	265	99	1	0	16	79	39	99
0	1	20	313	280	99	0	3	18	342	327	99	0	5	19	-109	103	99	0	8	3	320	332	99	1	0	17	375	375	99
0	1	21	79	57	99	0	3	19	192	192	99	0	5	20	337	317	99	0	8	4	-134	50	99	1	0	18	113	147	99
0	1	22	447	445	99	0	3	20	150	185	99	0	5	21	63	93	99	0	8	5	381	386	99	1	0	19	230	224	99
0	1	23	111	120	99	0	3	21	515	520	99	0	5	22	178	158	99	0	8	6	101	151	99	1	0	20	345	314	99
0	1	24	103	67	99	0	3	22	106	168	99	0	5	23	418	415	99	0	8	7	125	209	99	1	0	21	363	377	99
0	1	25	494	464	99	0	3	23	63	46	99	0	5	24	-125	48	99	0	8	8	210	219	99	1	0	22	-91	134	99
0	1	26	48	3	99	0	3	24	330	325	99	0	6	0	283	298	99	0	8	9	181	164	99	1	0	23	385	393	99
0	1	27	118	24	99	0	3	25	150	126	99	0	6	1	206	189	99	0	8	10	184	164	99	1	0	24	94	167	99
0	1	28	286	317	99	0	3	26	-98	10	99	0	6	2	97	130	99	0	8	11	300	284	99	1	0	25	122	128	99
0	2	0	483	474	99	0	4	0	62	13	99	0	6	3	167	185	99	0	8	12	111	118	99	1	0	26	473	459	99
0	2	1	551	553	99	0	4	1	589	591	99	0	6	4	476	462	99	0	8	13	298	313	99	1	0	27	-217	155	99
0	2	2	566	581	99	0	4	2	124	103	99	0	6	5	-58	56	99	0	8	14	439	428	99	1	0	28	82	53	99
0	2	3	1278	1276	99	0	4	3	624	629	99	0	6	6	97	177	99	0	8	15	-73	41	99	1	1	0	1082	1092	99
0	2	4	951	933	99	0	4	4	715	749	99	0	6	7	131	171	99	0	8	16	233	261	99	1	1	1	392	397	99
0	2	5	234	244	99	0	4	5	260	250	99	0	6	8	179	181	99	0	8	17	-161	144	99	1	1	2	857	869	99
0	2	6	17	56	99	0	4	6	327	302	99	0	6	9	101	311	99	0	8	18	107	160	99	1	1	3	1601	1596	99
0	2	7	457	465	99	0	4	7	1437	1419	99	0	6	10	-177	44	99	0	9	0	510	503	99	1	1	4	537	533	99
0	2	8	508	534	99	0	4	8	172	151	99	0	6	11	342	349	99	0	9	1	122	171	99	1	1	5	525	514	99
0	2	9	373	372	99	0	4	9	54	55	99	0	6	12	81	90	99	0	9	2	151	176	99	1	1	6	922	914	99
0	2	10	161	145	99	0	4	10	1209	1216	99	0	6	13	-132	9	99	0	9	3	407	409	99	1	1	7	185	179	99
0	2	11	129	126	99	0	4	11	-76	94	99	0	6	14	245	268	99	0	9	4	222	230	99	1	1	8	340	333	99
0	2	12	191	191	99	0	4	12	70	75	99	0	6	15	318	322	99	0	9	5	92	66	99	1	1	9	351	369	99
0	2	13	-81	114	99	0	4	13	501	492	99	0	6	16	207	188	99	0	9	6	394	395	99	1	1	10	586	597	99
0	2	14	287	304	99	0	4	14	98	160	99	0	6	17	-141	8	99	0	9	7	103	47	99	1	1	11	282	295	99
0	2	15	225	215	99	0	4	15	321	321	99	0	6	18	-61	50	99	0	9	8	196	196	99	1	1	12	566	564	99
0	2	16	239	200	99	0	4	16	538	510	99	0	6	19	263	236	99	0	9	9	279	274	99	1	1	13	229	228	99
0	2	17	334	358	99	0	4	17	51	6	99	0	6	20	91	65	99	0	9	10	23	12	99	1	1	14	201	236	99
0	2	18	351	367	99	0	4	18	164	155	99	0	6	21	-74	42	99	0	9	11	173	154	99	1	1	15	520	517	99
0	2	19	157	148	99	0	4	19	554	563	99	0	6	22	71	14	99	0	9	12	307	282	99	1	1	16	407	408	99
0	2	20	137	95	99	0	4	20	-66	161	99	0	6	23	168	148	99	0	9	13	-140	79	99	1	1	17	213	198	99
1	1	18	273	263	99	1	3	17	398	417	99	1	5	19	217	226	99	1	8	7	392	385	99	2	1	3	241	220	99
1	1	19	370	343	99	1	3	18	240	219	99	1	5	20	235	240	99	1	8	8	205	201	99	2	1	4	611	597	99
1	1	20	297	306	99	1	3	19	172	189	99	1	5	21	192	193	99	1	8	9	-96	71	99	2	1	5	197	213	99
1	1	21	216	208	99	1	3	20	726	714	99	1	5	22	242	249	99	1	8	10	465	473	99	2	1	6	325	322	99
1	1	22	160	164	99	1	3	21	187	187	99	1	5	23	86	215	99	1	8	11	209	169	99	2	1	7	378	363	99
1	1	23	130	198	99	1	3	22	183	196	99	1	6	0	-111	115	99	1	8	12	271	283	99	2	1	8	757	737	99
1	1	24	253	285	99	1	3	23	349	340	99	1	6	1	293	286	99	1	8	13	269	231	99	2	1	9	39	106	99
1	1	25	101	23	99	1	3	24	65	12	99	1	6	2	465	458	99	1	8	14	124	99	99	2	1	10	489	490	99
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4	2	13	243	281	99
4	2	14	254	267	99
4	2	15	217	208	99
4	2	16	368	363	99
4	2	17	232	268	99
4	2	18	154	111	99
4	2	19	75	77	99
4	2	20	62	102	99
4	2	21	83	40	99
4	2	22	136	64	99
4	2	23	-78	44	99
4	2	24	235	262	99
4	3	1	173	194	99
4	3	2	493	476	99
4	3	3	88	30	99
4	3	4	183	183	99
4	3	5	294	275	99
4	3	6	270	269	99
4	3	7	96	68	99
4	3	8	353	354	99
4	3	9	251	247	99
4	3	10	366	367	99
4	3	11	262	250	99
4	3	12	85	104	99
4	3	13	173	164	99
4	3	14	372	369	99
4	3	15	45	128	99
4	3	16	193	185	99
4	3	17	344	318	99
4	3	18	212	244	99
4	3	19	-120	79	99
4	3	20	214	173	99
4	3	21	232	220	99
4	3	22	151	144	99
4	3	23	108	129	99
4	4	0	502	493	99
4	4	1	67	99	99
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4	4	4	461	468	99
4	4	5	229	261	99
4	4	6	548	571	99
4	4	7	45	63	99
4	4	8	320	315	99
4	4	9	586	561	99
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4	4	17	-137	24	99
4	4	18	324	316	99
4	4	19	-145	58	99
4	4	20	55	122	99
4	4	21	283	267	99
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4	5	1	541	557	99
4	5	2	142	106	99
4	5	3	221	238	99
4	5	4	497	494	99
4	5	5	88	121	99
4	5	6	160	114	99
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4	5	8	73	22	99
4	5	9	210	177	99
4	5	10	383	389	99
4	5	11	226	240	99
4	5	12	128	51	99
4	5	13	298	294	99
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4	5	15	272	261	99
4	5	16	351	360	99
4	5	17	-109	86	99
4	5	18	132	83	99
4	6	0	136	126	99
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4	6	4	265	281	99
4	6	5	241	229	99
4	6	6	98	126	99
4	6	7	98	152	99
4	6	8	242	234	99
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4	6	12	103	106	99
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4	7	1	-193	92	99
4	7	2	117	124	99
4	7	3	220	220	99
4	7	4	100	99	99
4	7	5	204	208	99
4	7	6	183	177	99
4	7	7	114	118	99
4	7	8	119	149	99
4	7	9	129	157	99
4	7	10	99	159	99
4	7	11	-83	33	99
4	8	0	-115	66	99
4	8	1	230	203	99
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5	0	2	522	526	99
5	0	3	200	169	99
5	0	4	1322	1324	99
5	0	5	518	509	99
5	0	6	167	165	99
5	0	7	690	699	99
5	0	8	243	260	99
5	0	9	485	502	99
5	0	10	729	713	99
5	0	11	52	84	99
5	0	12	89	183	99
5	0	13	588	562	99
5	0	14	449	467	99
5	0	15	233	221	99
5	0	16	485	480	99
5	0	17	323	312	99
5	0	18	-108	42	99
5	0	19	414	434	99
5	0	20	-168	46	99
5	0	21	-100	24	99
5	0	22	273	272	99
5	0	23	-68	53	99
5	0	24	108	35	99
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5	1	3	610	614	99
5	1	4	108	177	99
5	1	5	637	618	99
5	1	6	556	555	99
5	1	7	203	192	99
5	1	8	247	210	99
5	1	9	251	259	99
5	1	10	351	352	99
5	1	11	267	272	99
5	1	12	180	176	99
5	1	13	218	226	99
5	1	14	435	425	99
5	1	15	391	386	99
5	1	16	28	128	99
5	1	17	390	368	99
5	1	18	173	215	99
5	1	19	146	46	99
5	1	20	263	238	99
5	1	21	-96	126	99
5	1	22	104	100	99
5	1	23	131	133	99
5	2	1	137	177	99
5	2	2	213	226	99
5	2	3	337	342	99
5	2	4	137	133	99
5	2	5	204	240	99
5	2	6	227	209	99
5	2	7	473	472	99

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
5	2	8	322	325	99	5	5	1	-50	135	99	6	0	22	116	117	99	6	3	12	141	341	99	7	0	16	105	114	99
5	2	9	367	339	99	5	5	2	221	224	99	6	0	23	-23	46	99	6	3	13	142	346	99	7	0	17	216	267	99
5	2	10	396	394	99	5	5	3	241	224	99	6	1	1	231	237	99	6	3	14	-98	140	99	7	0	18	32	58	99
5	2	11	248	244	99	5	5	4	41	88	99	6	1	2	497	474	99	6	3	15	229	225	99	7	0	19	-141	41	99
5	2	12	370	341	99	5	5	5	189	216	99	6	1	3	348	311	99	6	3	16	39	79	99	7	0	20	28	106	99
5	2	13	384	364	99	5	5	6	213	237	99	6	1	4	126	128	99	6	3	17	126	149	99	7	0	21	-110	11	99
5	2	14	109	36	99	5	5	7	61	63	99	6	1	5	92	102	99	6	3	18	270	290	99	7	1	1	288	304	99
5	2	15	174	158	99	5	5	8	91	60	99	6	1	6	176	222	99	6	4	1	-13	202	99	7	1	2	78	59	99
5	2	16	206	215	99	5	5	9	48	152	99	6	1	7	639	622	99	6	4	2	204	187	99	7	1	3	436	432	99
5	2	17	189	191	99	5	5	10	365	379	99	6	1	8	-35	52	99	6	4	3	313	291	99	7	1	4	-22	179	99
5	2	18	208	145	99	5	5	11	-68	82	99	6	1	9	629	629	99	6	4	4	143	162	99	7	1	5	351	361	99
5	2	19	199	189	99	5	5	12	111	165	99	6	1	10	254	236	99	6	4	5	285	315	99	7	1	6	216	181	99
5	2	20	-75	149	99	5	5	13	265	225	99	6	1	11	-70	18	99	6	4	6	-144	94	99	7	1	7	-145	65	99
5	2	21	193	150	99	5	5	14	275	260	99	6	1	12	187	183	99	6	4	7	364	333	99	7	1	8	359	351	99
5	2	22	-137	129	99	5	5	15	103	179	99	6	1	13	329	304	99	6	4	8	-77	117	99	7	1	9	210	206	99
5	3	1	437	426	99	5	5	16	-219	119	99	6	1	14	238	194	99	6	4	9	105	44	99	7	1	10	-60	62	99
5	3	2	127	131	99	5	6	0	325	336	99	6	1	15	220	205	99	6	4	10	210	198	99	7	1	11	79	181	99
5	3	3	334	336	99	5	6	1	137	145	99	6	1	16	246	264	99	6	4	11	283	263	99	7	1	12	352	365	99
5	3	4	280	287	99	5	6	2	241	262	99	6	1	17	254	220	99	6	4	12	-83	94	99	7	1	13	-130	123	99
5	3	5	277	252	99	5	6	3	45	160	99	6	1	18	65	142	99	6	4	13	170	156	99	7	1	14	126	76	99
5	3	6	130	176	99	5	6	4	36	107	99	6	1	19	71	111	99	6	4	14	121	169	99	7	1	15	331	325	99
5	3	7	372	386	99	5	6	5	26	37	99	6	1	20	75	112	99	6	4	15	99	90	99	7	1	16	193	175	99
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5	3	14	131	146	99	5	6	12	226	193	99	6	2	5	311	298	99	6	5	7	-127	117	99	7	2	4	326	318	99
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5	3	19	153	136	99	5	7	4	117	76	99	6	2	10	249	245	99	6	5	12	272	271	99	7	2	9	71	79	99
5	3	20	101	80	99	6	0	1	371	382	99	6	2	11	545	531	99	6	6	0	202	192	99	7	2	10	305	286	99
5	3	21	168	181	99	6	0	2	590	557	99	6	2	12	322	301	99	6	6	1	207	205	99	7	2	11	191	205	99
5	4	1	67	32	99	6	0	3	753	755	99	6	2	13	-47	168	99	6	6	2	-182	146	99	7	2	12	89	135	99
5	4	2	350	338	99	6	0	4	358	359	99	6	2	14	277	253	99	6	6	3	114	121	99	7	2	13	263	226	99
5	4	3	119	130	99	6	0	5	241	228	99	6	2	15	63	121	99	6	6	4	157	161	99	7	2	14	159	172	99
5	4	4	202	165	99	6	0	6	287	239	99	6	2	16	103	113	99	6	6	5	158	129	99	7	2	15	87	222	99
5	4	5	540	519	99	6	0	7	-69	16	99	6	2	17	97	61	99	7	0	1	156	126	99	7	2	16	212	237	99
5	4	6	114	105	99	6	0	8	116	16	99	6	2	18	120	15	99	7	0	2	309	330	99	7	2	17	99	147	99
5	4	7	162	155	99	6	0	9	84	36	99	6	2	19	268	280	99	7	0	3	108	94	99	7	3	1	258	241	99
5	4	8	297	308	99	6	0	10	291	239	99	6	2	20	-273	62	99	7	0	4	79	88	99	7	3	2	281	259	99
5	4	9	214	159	99	6	0	11	53	75	99	6	3	1	335	328	99	7	0	5	233	263	99	7	3	3	248	249	99
5	4	10	-118	69	99	6	0	12	220	199	99	6	3	2	244	234	99	7	0	6	416	414	99	7	3	4	277	264	99
5	4	11	429	411	99	6	0	13	472	479	99	6	3	3	405	408	99	7	0	7	-93	65	99	7	3	5	267	239	99
5	4	12	186	210	99	6	0	14	-142	136	99	6	3	4	218	192	99	7	0	8	176	198	99	7	3	6	263	284	99
5	4	13	103	54	99	6	0	15	167	224	99	6	3	5	-148	65	99	7	0	9	-72	97	99	7	3	7	217	244	99
5	4	14	380	378	99	6	0	16	218	213	99	6	3	6	332	322	99	7	0	10	381	366	99	7	3	8	314	300	99
5	4	15	20	89	99	6	0	17	170	155	99	6	3	7	420	429	99	7	0	11	298	290	99	7	3	9	348	369	99
5	4	16	-73	100	99	6	0	18	131	231	99	6	3	8	-21	104	99	7	0	12	-135	74	99	7	3	10	168	151	99
5	4	17	280	312	99	6	0	19	-45	189	99	6	3	9	404	394	99	7	0	13	134	141	99	7	3	11	-75	209	99
5	4	18	20	93	99	6	0	20	23	52	99	6	3	10	366	393	99	7	0	14	307	295	99	7	3	12	98	137	99
5	5	0	285	281	99	6	0	21	-164	132	99	6	3	11	172	188	99	7	0	15	120	52	99	7	3	13	206	172	99

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
7	3	14	-21	82	99	8	2	5	218	197	99	-3	3	27	372	354	99
7	3	15	94	40	99	8	2	6	193	220	99	-4	9	19	268	302	99
7	4	1	83	64	99	8	2	7	134	196	99	-5	9	19	139	143	99
7	4	2	122	110	99	8	2	8	212	198	99	10	0	1	93	125	99
7	4	3	149	139	99	8	2	9	231	220	99	10	0	2	81	136	99
7	4	4	120	155	99	8	2	10	-47	122	99	10	0	3	-105	70	99
7	4	5	-90	121	99	8	2	11	244	228	99	10	0	4	179	159	99
7	4	6	76	194	99	8	2	12	221	197	99	10	0	5	128	116	99
7	4	7	-108	97	99	8	2	13	-154	87	99	10	0	6	372	410	99
7	4	8	216	161	99	8	2	14	206	215	99	10	0	7	98	10	99
7	4	9	285	281	99	8	3	1	187	138	99	10	0	8	133	50	99
7	4	10	139	149	99	8	3	2	92	48	99	10	0	9	-13	100	99
7	4	11	78	63	99	8	3	3	309	315	99	10	1	1	107	138	99
7	5	1	101	103	99	8	3	4	109	144	99	10	1	2	192	170	99
7	5	2	168	129	99	8	3	5	-64	38	99						
7	5	3	-71	117	99	8	3	6	186	153	99						
7	5	4	-236	90	99	8	3	7	102	164	99						
8	0	1	520	536	99	8	3	8	101	21	99						
8	0	2	325	314	99	8	3	9	-67	85	99						
8	0	3	-199	68	99	8	3	10	-121	220	99						
8	0	4	569	565	99	8	4	1	45	133	99						
8	0	5	158	139	99	9	0	1	176	179	99						
8	0	6	117	118	99	9	0	2	-71	25	99						
8	0	7	111	121	99	9	0	3	241	252	99						
8	0	8	36	125	99	9	0	4	-78	157	99						
8	0	9	235	277	99	9	0	5	110	18	99						
8	0	10	508	483	99	9	0	6	362	377	99						
8	0	11	285	359	99	9	0	7	197	155	99						
8	0	12	52	69	99	9	0	8	127	110	99						
8	0	13	467	467	99	9	0	9	224	200	99						
8	0	14	150	274	99	9	0	10	-24	30	99						
8	0	15	113	90	99	9	0	11	127	70	99						
8	0	16	253	243	99	9	0	12	320	316	99						
8	0	17	-61	42	99	9	0	13	11	145	99						
8	0	18	-189	12	99	9	0	14	-40	14	99						
8	1	1	88	80	99	9	1	1	254	220	99						
8	1	2	343	314	99	9	1	2	204	201	99						
8	1	3	-85	28	99	9	1	3	75	117	99						
8	1	4	350	323	99	9	1	4	159	205	99						
8	1	5	269	261	99	9	1	5	321	337	99						
8	1	6	196	217	99	9	1	6	-112	128	99						
8	1	7	272	266	99	9	1	7	189	193	99						
8	1	8	330	296	99	9	1	8	249	256	99						
8	1	9	-65	178	99	9	1	9	-98	83	99						
8	1	10	75	208	99	9	1	10	255	257	99						
8	1	11	310	324	99	9	1	11	-113	54	99						
8	1	12	66	104	99	9	1	12	37	61	99						
8	1	13	-138	38	99	9	2	1	273	270	99						
8	1	14	269	251	99	9	2	2	-71	156	99						
8	1	15	122	74	99	9	2	3	275	242	99						
8	1	16	119	74	99	9	2	4	161	167	99						
8	2	1	139	136	99	9	2	5	-73	139	99						
8	2	2	274	271	99	9	2	6	224	208	99						
8	2	3	317	307	99	9	2	7	147	155	99						
8	2	4	-167	139	99	-3	4	10	628	611	99						

APPENDIX 6.I

$[Cd(S_2CNiPr_2)_2]$

Table 6.I.1: Crystallographic Parameters for $[Cd(S_2CN(CH(CH_3)_2)_2)_2]$

Formula	$C_{14}H_{28}CdN_2S_4$
Formula weight	465.04
Crystal morphology / colour	block / colourless
Crystal dimensions (mm)	0.16 x 0.16 x 0.32
Crystal system	monoclinic
Space group	$P2_1/n$ (#14)
a (Å)	11.611(5)
b (Å)	11.228(9)
c (Å)	16.767(2)
α (°)	90
β (°)	108.90(2)
γ (°)	90
V (Å^3)	2067(1)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.494
$F(000)$	952
Temperature (°K)	293
μ (cm^{-1})	14.57
θ limits, cell (°)	25.2 – 26.4
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 15, 0 – 13, -21 – 21
Range of transmission factors	0.933 – 1
Scan technique	ω:2θ
No. of data measured	5291
No. of unique data	5053
R_{amal}	0.104
No. of parameters	190
R (I≥0σ(I))	0.108
R_w (I≥0σ(I))	0.157
R (I≥3σ(I))	0.059
R_w (I≥3σ(I))	0.074
Maximum and minimum residual electron density (e Å^{-3})	2.71, -2.41

Table 6.I.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Cd(S_2CN(CH(CH_3)_2)_2)_2]$

Atom	*x*	*y*	*z*
Cd	0.09580(5)	0.07078(5)	0.44802(3)
S(1)	0.2577(2)	-0.0426(2)	0.5620(1)
S(2)	0.0306(2)	-0.1667(2)	0.4658(1)
S(3)	-0.0081(2)	0.0859(2)	0.2888(1)
S(4)	0.1971(2)	0.2381(2)	0.3862(1)
N(1)	0.2150(5)	-0.2711(5)	0.5862(3)
N(2)	0.0980(5)	0.2492(5)	0.2195(3)
C(1)	0.1737(6)	-0.1699(6)	0.5444(4)
C(2)	0.1401(7)	-0.3819(7)	0.5699(5)
C(3)	0.1847(9)	-0.4688(8)	0.5217(7)
C(4)	0.1277(9)	-0.431(1)	0.6531(8)
C(5)	0.3411(6)	-0.2855(7)	0.6482(5)
C(6)	0.4414(7)	-0.2681(8)	0.6077(5)
C(7)	0.3607(8)	-0.2154(8)	0.7289(5)
C(8)	0.0959(5)	0.1977(6)	0.2893(4)
C(9)	0.0126(7)	0.2214(8)	0.1329(4)
C(10)	0.0340(9)	0.0947(9)	0.1055(5)
C(11)	-0.1195(8)	0.2506(8)	0.1230(5)
C(12)	0.1888(7)	0.3468(7)	0.2209(4)
C(13)	0.2717(9)	0.310(1)	0.1714(6)
C(14)	0.126(1)	0.4665(9)	0.1937(8)

Table 6.I.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Cd(S_2CN(CH(CH_3)_2)_2)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cd	0.0420(3)	0.0441(3)	0.0361(3)	0.0072(2)	0.0164(2)	0.0101(2)
S(1)	0.045(1)	0.033(1)	0.070(1)	-0.0021(8)	-0.0027(9)	0.0071(9)
S(2)	0.0377(8)	0.040(1)	0.0311(8)	0.0031(7)	0.0095(6)	0.0033(7)
S(3)	0.047(1)	0.041(1)	0.0388(9)	-0.0105(8)	0.0112(7)	0.0047(8)
S(4)	0.052(1)	0.060(1)	0.0338(9)	-0.020(1)	0.0078(8)	0.0031(8)
N(1)	0.033(3)	0.034(3)	0.046(3)	0.005(2)	0.007(2)	0.008(3)
N(2)	0.047(3)	0.042(4)	0.034(3)	-0.008(3)	0.014(2)	0.000(3)
C(1)	0.035(3)	0.046(4)	0.032(3)	0.002(3)	0.012(3)	0.000(3)
C(2)	0.041(4)	0.041(4)	0.077(6)	-0.002(4)	0.006(4)	0.015(4)
C(3)	0.076(7)	0.038(5)	0.135(9)	-0.005(5)	0.016(6)	-0.021(6)
C(4)	0.069(6)	0.100(9)	0.124(9)	-0.010(6)	0.023(6)	0.061(7)
C(5)	0.034(4)	0.050(5)	0.053(4)	0.007(3)	0.005(3)	0.016(4)
C(6)	0.039(4)	0.052(5)	0.078(6)	0.007(4)	0.015(4)	-0.002(4)
C(7)	0.068(5)	0.075(6)	0.042(4)	0.008(5)	0.006(4)	0.006(4)
C(8)	0.034(3)	0.036(4)	0.034(3)	0.004(3)	0.014(3)	0.002(3)
C(9)	0.066(5)	0.058(5)	0.031(4)	-0.013(4)	0.011(3)	0.004(3)
C(10)	0.100(7)	0.071(7)	0.053(5)	-0.021(6)	0.036(5)	-0.019(5)
C(11)	0.072(6)	0.065(6)	0.056(5)	0.001(5)	-0.008(4)	0.006(4)
C(12)	0.062(5)	0.050(5)	0.045(4)	-0.021(4)	0.021(4)	0.003(4)
C(13)	0.075(6)	0.094(8)	0.085(7)	-0.024(6)	0.040(5)	0.019(6)
C(14)	0.088(8)	0.052(6)	0.16(1)	-0.015(6)	0.038(7)	0.017(7)

Table 6.I.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in [Cd($S_2CN(CH(CH_3)2)2)2$]*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.0606	-0.3597	0.5354	5.3107
H(3a)	0.1307	-0.5371	0.5087	8.2717
H(3b)	0.1871	-0.4325	0.4698	8.2717
H(3c)	0.2660	-0.4943	0.5549	8.2717
H(4a)	0.0954	-0.3689	0.6803	9.4320
H(4b)	0.0728	-0.4984	0.6408	9.4320
H(4c)	0.2070	-0.4554	0.6902	9.4320
H(5a)	0.3466	-0.3668	0.6647	4.5565
H(6a)	0.5198	-0.2882	0.6483	5.4118
H(6b)	0.4255	-0.3195	0.5588	5.4118
H(6c)	0.4422	-0.1857	0.5905	5.4118
H(7a)	0.4407	-0.2332	0.7682	6.1429
H(7b)	0.3548	-0.1309	0.7163	6.1429
H(7c)	0.2991	-0.2375	0.7538	6.1429
H(9a)	0.0346	0.2737	0.0957	4.9822
H(10a)	0.0121	0.0370	0.1411	6.8390
H(10b)	-0.0159	0.0822	0.0473	6.8390
H(10c)	0.1191	0.0852	0.1107	6.8390
H(11a)	-0.1256	0.3333	0.1378	6.7333
H(11b)	-0.1697	0.2373	0.0650	6.7333
H(11c)	-0.1471	0.1998	0.1599	6.7333
H(12a)	0.2390	0.3555	0.2779	4.8698
H(13a)	0.3118	0.2355	0.1939	7.7094
H(13b)	0.2238	0.2991	0.1126	7.7094
H(13c)	0.3325	0.3711	0.1762	7.7094

Table 6.I.4: Cont.

Atom	x	y	z	B_{eq}
H(14a)	0.1876	0.5276	0.1990	9.3803
H(14b)	0.0741	0.4617	0.1355	9.3803
H(14c)	0.0780	0.4865	0.2294	9.3803

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 6.I.5: Bond distances (Å) for [Cd($S_2CN(CH(CH_3)_2)_2)_2$]

Cd — S(1)	2.545(2)	N(2) — C(8)	1.313(8)
Cd — S(2)	2.814(2)	N(2) — C(9)	1.502(8)
Cd — S(2)*	2.603(2)	N(2) — C(12)	1.516(9)
Cd — S(3)	2.554(2)	C(2) — C(3)	1.47(1)
Cd — S(4)	2.604(2)	C(2) — C(4)	1.55(1)
S(1) — C(1)	1.701(7)	C(5) — C(6)	1.54(1)
S(2) — C(1)	1.755(6)	C(5) — C(7)	1.52(1)
S(3) — C(8)	1.739(7)	C(9) — C(10)	1.54(1)
S(4) — C(8)	1.730(6)	C(9) — C(11)	1.52(1)
N(1) — C(1)	1.340(8)	C(12) — C(13)	1.52(1)
N(1) — C(2)	1.491(9)	C(12) — C(14)	1.52(1)
N(1) — C(5)	1.505(8)		

* Symmetry operation: -*x*, -*y*, 1-*z*

Table 6.I.6: Bond angles (deg.) for [Cd($S_2CN(CH(CH_3)_2)_2)_2$]

S(1) — Cd — S(2)	66.75(6)	C(8) — N(2) — C(12)	121.2(5)
S(1) — Cd — S(2)*	101.67(7)	C(9) — N(2) — C(12)	113.8(5)
S(1) — Cd — S(3)	143.31(7)	S(1) — C(1) — S(2)	117.6(4)
S(1) — Cd — S(4)	109.92(7)	S(1) — C(1) — N(1)	122.3(5)
S(2) — Cd — S(2)*	96.54(5)	S(2) — C(1) — N(1)	120.1(5)
S(2) — Cd — S(3)	96.99(5)	N(1) — C(2) — C(3)	111.5(7)
S(2) — Cd — S(4)	153.86(6)	N(1) — C(2) — C(4)	110.5(7)
S(2)* — Cd — S(3)	113.11(6)	C(3) — C(2) — C(4)	114.4(8)
S(2)* — Cd — S(4)	109.34(7)	N(1) — C(5) — C(6)	112.9(6)
S(3) — Cd — S(4)	69.95(6)	N(1) — C(5) — C(7)	113.0(6)
Cd — S(1) — C(1)	92.7(2)	C(6) — C(5) — C(7)	114.3(7)
Cd — S(2) — Cd*	83.46(5)	S(3) — C(8) — S(4)	116.9(4)
Cd — S(2) — C(1)	82.9(2)	S(3) — C(8) — N(2)	121.9(5)
Cd — S(2) — C(1)	99.1(2)	S(4) — C(8) — N(2)	121.2(5)
Cd — S(3) — C(8)	87.2(2)	N(2) — C(9) — C(10)	111.2(7)
Cd — S(4) — C(8)	85.8(2)	N(2) — C(9) — C(11)	112.7(6)
C(1) — N(1) — C(2)	121.6(5)	C(10) — C(9) — C(11)	114.9(7)
C(1) — N(1) — C(5)	123.7(6)	N(2) — C(12) — C(13)	110.7(7)
C(2) — N(1) — C(5)	114.6(5)	N(2) — C(12) — C(14)	111.8(7)
C(8) — N(2) — C(9)	124.9(6)	C(13) — C(12) — C(14)	113.7(7)

* symmetry operation: *-x*, *-y*, 1-*z*

Table 6.I.7: Observed and calculated structure factors for $[Cd(S_2CN(CH(CH_3)_2)_2)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	4	146	141	99	0	3	7	294	320	99	0	6	2	258	224	99	0	9	3	441	418	99	0	13	1	208	209	99
0	0	6	1140	1094	99	0	3	8	641	624	99	0	6	3	617	556	99	0	9	4	71	66	99	0	13	2	138	127	99
0	0	8	1063	1097	99	0	3	9	417	402	99	0	6	4	309	280	99	0	9	5	94	84	99	0	13	3	157	147	99
0	0	10	842	836	99	0	3	10	145	145	99	0	6	5	817	792	99	0	9	6	689	672	99	0	13	4	201	203	99
0	0	12	1276	1275	99	0	3	11	132	120	99	0	6	6	608	574	99	0	9	7	424	406	99	0	13	5	75	101	99
0	0	14	54	74	99	0	3	12	851	852	99	0	6	7	194	193	99	0	9	8	123	89	99	0	13	6	177	154	99
0	0	16	47	10	99	0	3	13	263	266	99	0	6	8	344	322	99	0	9	9	532	545	99	0	13	7	42	76	99
0	0	18	470	463	99	0	3	14	616	623	99	0	6	9	24	27	99	0	9	10	184	187	99	0	13	8	75	89	99
0	0	20	193	187	99	0	3	15	328	334	99	0	6	10	180	165	99	0	9	11	330	326	99	0	13	9	69	62	99
0	1	2	1226	1178	99	0	3	16	142	152	99	0	6	11	245	223	99	0	9	12	260	249	99	0	14	0	-41	71	99
0	1	3	722	704	99	0	3	17	206	205	99	0	6	12	440	432	99	0	9	13	54	72	99	0	14	1	-81	14	99
0	1	4	871	895	99	0	3	18	177	177	99	0	6	13	408	405	99	0	9	14	186	188	99	0	14	2	49	40	99
0	1	5	632	595	99	0	3	19	84	83	99	0	6	14	97	92	99	0	9	15	37	21	99	0	14	3	76	101	99
0	1	6	342	355	99	0	3	20	74	66	99	0	6	15	233	231	99	0	9	16	90	76	99	0	14	4	45	35	99
0	1	7	1604	1584	99	0	4	0	614	605	99	0	6	16	177	187	99	0	10	0	421	413	99	0	14	5	128	113	99
0	1	8	348	344	99	0	4	1	860	834	99	0	6	17	39	38	99	0	10	1	218	226	99	0	14	6	-89	28	99
0	1	9	941	930	99	0	4	2	285	274	99	0	6	18	355	372	99	0	10	2	383	369	99	1	0	-21	-80	21	99
0	1	10	138	149	99	0	4	3	1560	1509	99	0	7	1	811	750	99	0	10	3	254	234	99	1	0	-19	279	261	99
0	1	11	969	984	99	0	4	4	888	839	99	0	7	2	137	122	99	0	10	4	41	36	99	1	0	-17	330	314	99
0	1	12	240	234	99	0	4	5	1760	1677	99	0	7	3	399	362	99	0	10	5	463	429	99	1	0	-15	429	409	99
0	1	13	157	154	99	0	4	6	247	250	99	0	7	4	218	208	99	0	10	6	215	200	99	1	0	-13	290	302	99
0	1	14	368	347	99	0	4	7	1085	1073	99	0	7	5	113	99	99	0	10	7	279	280	99	1	0	-11	1206	1174	99
0	1	15	151	132	99	0	4	8	535	527	99	0	7	6	-60	12	99	0	10	8	-21	110	99	1	0	-9	1189	1166	99
0	1	16	220	213	99	0	4	9	87	95	99	0	7	7	803	757	99	0	10	9	46	73	99	1	0	-7	1346	1312	99
0	1	17	221	196	99	0	4	10	271	270	99	0	7	8	39	42	99	0	10	10	172	169	99	1	0	-5	671	656	99
0	1	18	74	81	99	0	4	11	298	264	99	0	7	9	432	441	99	0	10	11	133	125	99	1	0	-3	1829	1904	99
0	1	19	354	341	99	0	4	12	161	153	99	0	7	10	286	265	99	0	10	12	-24	39	99	1	0	3	1246	1252	99
0	1	20	216	196	99	0	4	13	582	591	99	0	7	11	474	478	99	0	10	13	269	270	99	1	0	5	889	894	99
0	2	0	388	361	99	0	4	14	47	66	99	0	7	12	332	336	99	0	10	14	14	27	99	1	0	7	838	821	99
0	2	1	607	569	99	0	4	15	302	331	99	0	7	13	198	191	99	0	10	15	122	123	99	1	0	9	759	766	99
0	2	2	1166	1173	99	0	4	16	171	168	99	0	7	14	45	56	99	0	11	1	113	90	99	1	0	11	359	383	99
0	2	3	776	729	99	0	4	17	287	301	99	0	7	15	142	152	99	0	11	2	219	210	99	1	0	13	651	680	99
0	2	4	990	967	99	0	4	18	69	75	99	0	7	16	86	76	99	0	11	3	72	11	99	1	0	15	371	369	99
0	2	5	1297	1260	99	0	4	19	36	59	99	0	7	17	237	260	99	0	11	4	331	289	99	1	0	17	232	219	99
0	2	6	95	93	94	0	5	1	388	384	99	0	7	18	94	128	99	0	11	5	228	231	99	1	0	19	369	350	99
0	2	7	350	333	99	0	5	2	1298	1263	99	0	8	0	817	779	99	0	11	6	153	139	99	1	1	-21	88	94	99
0	2	8	1319	1269	99	0	5	3	258	231	99	0	8	1	423	436	99	0	11	7	-69	11	99	1	1	-20	192	190	99
0	2	9	165	165	99	0	5	4	1427	1373	99	0	8	2	644	619	99	0	11	8	56	74	99	1	1	-19	253	232	99
0	2	10	706	705	99	0	5	5	110	118	99	0	8	3	92	99	99	0	11	9	58	38	99	1	1	-18	378	345	99
0	2	11	128	126	99	0	5	6	964	953	99	0	8	4	490	446	99	0	11	10	99	107	99	1	1	-17	92	99	99
0	2	12	-44	59	99	0	5	7	298	291	99	0	8	5	355	332	99	0	11	11	126	103	99	1	1	-16	227	213	99
0	2	13	770	744	99	0	5	8	321	295	99	0	8	6	103	94	99	0	11	12	137	144	99	1	1	-15	42	53	99
0	2	14	248	235	99	0	5	9	336	309	99	0	8	7	282	254	99	0	11	13	59	91	99	1	1	-14	191	162	99
0	2	15	396	382	99	0	5	10	45	50	99	0	8	8	723	704	99	0	12	0	321	303	99	1	1	-13	944	899	99
0	2	16	269	262	99	0	5	11	388	364	99	0	8	9	257	258	99	0	12	1	59	67	99	1	1	-12	635	602	99
0	2	17	254	245	99	0	5	12	286	268	99	0	8	10	638	633	99	0	12	2	120	84	99	1	1	-11	-77	45	99
0	2	18	183	167	99	0	5	13	96	69	99	0	8	11	443	453	99	0	12	3	282	272	99	1	1	-10	693	645	99
0	2	19	47	42	99	0	5	14	374	379	99	0	8	12	304	321	99	0	12	4	199	168	99	1	1	-9	160	125	99
0	2	20	239	221	99	0	5	15	165	158	99	0	8	13	130	140	99	0	12	5	249	240	99	1	1	-8	1147	1182	99
0	3	1	40	16	62	0	5	16	313	326	99	0	8	14	37	36	99	0	12	6	57	12	99	1	1	-7	176	179	99
0	3	2	924	880	99	0	5	17	-65	20	99	0	8	15	63	88	99	0	12	7	157	165	99	1	1	-6	1055	1072	99
0	3	3	151	138	99	0	5	18	98	110	99	0	8	16	104	90	99	0	12	8	31	35	99	1	1	-5	264	278	99
0	3	4	984	976	99	0	5	19	159	175	99	0	8	17	193	195	99	0	12	9	79	79	99	1	1	-4	346	351	99
0	3	5	780	712	99	0	6	0	1446	1454	99	0	9	1	894	846	99	0	12	10	-72	62	99	1	1	-3	496	582	99
0	3	6	2009	2002	99	0	6	1	331	341	99	0	9	2	186	174	99	0	12	11	105	104	99	1	1	-2	185	214	99
1	1	1	1302	1296	99	1	2	16	216	224	99	1	4	-7	455	524	99	1	5	11	310	324	99	1	7	-9	66	78	99
1	1	2	1661	1675	99	1	2	17	130	122	99	1	4	-5	15	44	99	1	5	12	459	448	99	1	7	-8	603	644	99
1	1	3	124	103	76	1	2	18	242	231	99	1	4	-3	276	299	99	1	5	13	-15	32	99	1	7	-7	122	128	99
1	1	4	632	616	99	1	2	19	-136	38	99	1	4	-2	1096	1134	99	1	5	14	105	91	99	1	7	-6	438	460	99
1	1	5	745	727	99	1	3	-20	-54	73	99	1	4	-1	679	644	99	1	5	15	155	183	99	1	7	-5	103	84	99
1	1	6	79	78	87	1	3	-19	31	44	99	1	4	0	692	640	99	1	5	16	85	99	99	1	7	-4	315	331	99
1	1	7	326	309	99	1	3	-18	223	188	99	1	4	1	746	683	99	1	5	17	312	325	99	1	7	-3	227	217	99
1	1	8	396	417	99	1	3	-17	357	302	99	1	4	2	314	272	99	1	5	18	130	136	99	1	7	-2	202	219	99
1	1	9	352	344	99	1	3	-16	302	265	99	1	4	3	180	153	99	1	6	-19	338	267	99	1	7	-1	743	731	99
1	1	10	441	448	99	1	3	-15	325	303	99	1	4	4	452	431	99	1	6	-18	-59	36	99	1	7	0	1119	1036	99
1	1	11	129	132	99	1	3	-14	293	263	99	1	4	5	791	762	99	1	6	-17	418	341	99	1	7	1	239	226	99
1	1	12	749	771	99	1	3	-13	508	464	99	1	4	6	1272	1248	99	1	6	-16	-28	94	99	1	7	2	776	710	99
1	1	13	104	111	99	1	3	-12	233	233	99	1	4	7	-6	10	99	1	6	-15	579	542	99	1	7	3	-60	23	99
1	1	14	217	227	99	1	3	-11	555	550	99	1	4	8	702	696	99	1	6	-14	197	182	99	1	7	4	509	475	99
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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12	1	-15	358	351	99	12	3	-8	256	286	99	12	5	2	84	76	99	13	0	-3	345	346	99	13	3	-6	48	33	99
12	1	-14	68	62	99	12	3	-7	39	4	99	12	5	3	88	81	99	13	0	-1	216	244	99	13	3	-5	65	71	99
12	1	-13	108	120	99	12	3	-6	171	185	99	12	5	4	69	59	99	13	0	1	-33	33	99	13	3	-4	-108	4	99
12	1	-12	128	140	99	12	3	-5	-77	34	99	12	6	-14	233	222	99	13	0	3	90	113	99	13	3	-3	159	138	99
12	1	-11	-37	127	99	12	3	-4	284	296	99	12	6	-13	159	156	99	13	1	-16	220	207	99	13	3	-2	48	54	99
12	1	-10	-9	11	99	12	3	-3	136	113	99	12	6	-12	62	59	99	13	1	-15	28	99	99	13	3	-1	274	291	99
12	1	-9	343	348	99	12	3	-2	480	491	99	12	6	-11	127	142	99	13	1	-14	289	299	99	13	3	0	-46	58	99
12	1	-8	78	87	99	12	3	-1	173	168	99	12	6	-10	65	54	99	13	1	-13	-85	8	99	13	3	1	72	92	99
12	1	-7	512	521	99	12	3	0	83	118	99	12	6	-9	115	101	99	13	1	-12	143	139	99	13	3	2	91	95	99
12	1	-6	184	174	99	12	3	1	133	153	99	12	6	-8	251	253	99	13	1	-11	57	14	99	13	3	3	118	126	99
12	1	-5	240	250	99	12	3	2	57	4	99	12	6	-7	-31	57	99	13	1	-10	68	63	99	13	4	-15	-45	130	99
12	1	-4	-52	28	99	12	3	3	-78	27	99	12	6	-6	-77	89	99	13	1	-9	113	107	99	13	4	-14	149	171	99
12	1	-3	170	168	99	12	3	4	113	119	99	12	6	-5	182	195	99	13	1	-8	202	203	99	13	4	-13	-61	8	99
12	1	-2	59	76	99	12	3	5	-27	25	99	12	6	-4	27	62	99	13	1	-7	-95	2	99	13	4	-12	212	222	99
12	1	-1	220	225	99	12	3	6	166	180	99	12	6	-3	81	109	99	13	1	-6	218	213	99	13	4	-11	90	65	99
12	1	0	64	101	99	12	4	-16	70	60	99	12	6	-2	69	57	99	13	1	-5	32	45	99	13	4	-10	306	302	99
12	1	1	226	242	99	12	4	-15	154	166	99	12	6	-1	99	122	99	13	1	-4	228	237	99	13	4	-9	44	33	99
12	1	2	31	38	99	12	4	-14	65	4	99	12	6	0	140	156	99	13	1	-3	-13	6	99	13	4	-8	332	323	99
12	1	3	227	217	99	12	4	-13	428	416	99	12	6	1	42	10	99	13	1	-2	190	198	99	13	4	-7	103	105	99
13	4	-6	106	111	99	14	1	-11	257	258	99	14	5	-7	64	38	99												
13	4	-5	128	113	99	14	1	-10	31	68	99	14	5	-6	162	153	99												
13	4	-4	51	50	99	14	1	-9	154	168	99	14	5	-5	-58	13	99												
13	4	-3	216	199	99	14	1	-8	159	163	99	14	5	-4	42	52	99												
13	4	-2	277	288	99	14	1	-7	101	83	99	15	0	-9	178	193	99												
13	4	-1	17	35	99	14	1	-6	56	14	99	15	0	-7	-63	6	99												
13	4	0	202	217	99	14	1	-5	191	193	99	15	0	-5	149	158	99												
13	4	1	60	10	99	14	1	-4	50	10	99	15	1	-9	72	10	99												
13	4	2	238	250	99	14	1	-3	327	341	99	15	1	-8	-78	79	99												
13	5	-13	188	183	99	14	1	-2	-25	53	99	15	1	-7	-41	54	99												
13	5	-12	54	20	99	14	1	-1	204	226	99	15	1	-6	-63	33	99												
13	5	-11	220	230	99	14	1	0	-11	16	99	15	1	-5	97	74	99												
13	5	-10	64	89	99	14	1	1	131	128	99																		
13	5	-9	294	307	99	14	2	-13	32	47	99																		
13	5	-8	117	102	99	14	2	-12	-97	42	99																		
13	5	-7	101	115	99	14	2	11	13	29	99																		
13	5	-6	62	54	99	14	2	-10	29	88	99																		
13	5	-5	16	19	99	14	2	-9	158	153	99																		
13	5	-4	168	170	99	14	2	-8	56	45	99																		
13	5	-3	-61	18	99	14	2	-7	129	155	99																		
13	5	-2	116	121	99	14	2	-6	-88	16	99																		
13	5	-1	139	160	99	14	2	-5	151	151	99																		
13	5	0	-85	25	99	14	2	-4	146	146	99																		
13	5	1	185	188	99	14	2	-3	-9	24	99																		
13	6	-12	132	119	99	14	2	-2	224	236	99																		
13	6	-11	47	105	99	14	2	-1	57	84	99																		
13	6	-10	54	76	99	14	2	0	102	62	99																		
13	6	-9	71	42	99	14	3	-13	-24	16	99																		
13	6	-8	128	99	99	14	3	-12	-39	21	99																		
13	6	-7	141	170	99	14	3	-11	-30	27	99																		
13	6	-6	-31	27	99	14	3	-10	171	163	99																		
13	6	-5	46	106	99	14	3	-9	-102	17	99																		
13	6	-4	89	82	99	14	3	-8	220	235	99																		
13	6	-3	121	147	99	14	3	-7	-48	17	99																		
13	6	-2	34	8	99	14	3	-6	201	210	99																		
13	6	-1	-76	23	99	14	3	-5	-66	13	99																		
13	6	0	63	62	99	14	3	-4	201	219	99																		
13	7	-9	174	174	99	14	3	-3	-76	18	99																		
13	7	-8	170	147	99	14	3	-2	113	141	99																		
13	7	-7	-41	15	99	14	3	-1	-76	37	99																		
13	7	-6	178	185	99	14	3	0	181	182	99																		
13	7	-5	21	86	99	14	4	-12	67	5	99																		
13	7	-4	154	154	99	14	4	-11	137	145	99																		
13	7	-3	22	31	99	14	4	-10	-26	15	99																		
14	0	-14	267	273	99	14	4	-9	353	359	99																		
14	0	-12	358	383	99	14	4	-8	-9	9	99																		
14	0	-10	331	341	99	14	4	-7	214	227	99																		
14	0	-8	-43	79	99	14	4	-6	-57	36	99																		
14	0	-6	272	271	99	14	4	-5	202	209	99																		
14	0	-4	370	355	99	14	4	-4	101	68	99																		
14	0	-2	291	324	99	14	4	-3	-74	33	99																		
14	0	0	167	173	99	14	4	-2	126	124	99																		
14	1	-14	192	185	99	14	5	-10	221	228	99																		
14	1	-13	227	230	99	14	5	-9	-44	30	99																		
14	1	-12	54	51	99	14	5	-8	313	317	99																		

APPENDIX 6.II

$[Cd(S_2CNCy_2)_2]\cdot CH_2Cl_2$

Table 6.II.1: Crystallographic Parameters for $[Cd(S_2CN(CH(CH_2)_5)_2)_2]\cdot CH_2Cl_2$

Formula	$C_{26}H_{44}CdN_2S_4.CH_2Cl_2$
Formula weight	710.23
Crystal morphology / colour	needle / colourless
Crystal dimensions (mm)	0.07 x 0.08 x 0.44
Crystal system	monoclinic
Space group	$P\bar{1}$ (#2)
a (Å)	13.557(7)
b (Å)	14.49(2)
c (Å)	9.604(2)
α (°)	100.70(6)
β (°)	91.55(3)
γ (°)	115.58(6)
V (Å^3)	1659(2)
Z	2
$\rho_{calc.}$ (g cm^{-3})	1.421
$F(000)$	736
Temperature (°K)	293
μ (cm^{-1})	10.91
θ limits, cell (°)	12.2 – 17.0
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 17, -17–17, -10–10
Range of transmission factors	0.905 – 1
Scan technique	ω:2θ
No. of data measured	7967
No. of unique data	7647
R_{amal}	0.079
No. of parameters	325
R (I≥0σ(I))	0.254
R_w (I≥0σ(I))	0.184
R (I≥3σ(I))	0.079
R_w (I≥3σ(I))	0.079
Maximum and minimum residual electron density (e Å^{-3})	3.45, -5.74 (located near Cd atom)

Table 6.II.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Cd(S_2CN(CH(CH_2)_5)_2)_2]\cdot CH_2Cl_2$

Atom	*x*	*y*	*z*
Cd	0.05497(8)	-0.00027(8)	0.18282(10)
Cl(1)	0.7086(4)	0.1621(5)	0.7077(5)
Cl(2)	0.7420(5)	0.1987(5)	1.0123(6)
S(1)	-0.0658(3)	-0.1953(3)	0.0896(3)
S(2)	0.0696(2)	-0.0786(2)	-0.1123(3)
S(3)	0.2595(2)	0.1302(3)	0.2332(3)
S(4)	0.1271(2)	0.0287(3)	0.4482(3)
N(1)	-0.0743(8)	-0.2834(8)	-0.1844(10)
N(2)	0.3439(7)	0.1557(7)	0.5019(9)
C(1)	-0.0307(8)	-0.1976(9)	-0.082(1)
C(2)	-0.043(1)	-0.2820(10)	-0.332(1)
C(3)	-0.140(1)	-0.321(1)	-0.447(1)
C(4)	-0.100(1)	-0.310(1)	-0.592(1)
C(5)	-0.027(1)	-0.365(1)	-0.630(2)
C(6)	0.066(1)	-0.329(2)	-0.515(2)
C(7)	0.027(1)	-0.339(1)	-0.370(1)
C(8)	-0.155(1)	-0.3874(9)	-0.162(1)
C(9)	-0.107(1)	-0.432(1)	-0.064(2)
C(10)	-0.186(2)	-0.542(2)	-0.060(2)
C(11)	-0.297(3)	-0.549(2)	-0.021(2)
C(12)	-0.344(2)	-0.505(2)	-0.122(2)
C(13)	-0.266(1)	-0.394(1)	-0.132(1)

Table 6.II.2: Cont.

Atom	*x*	*y*	*z*
C(14)	0.2540(9)	0.1116(10)	0.405(1)
C(15)	0.4555(9)	0.224(1)	0.473(1)
C(16)	0.468(1)	0.325(1)	0.441(1)
C(17)	0.588(1)	0.399(1)	0.427(2)
C(18)	0.635(1)	0.342(2)	0.325(2)
C(19)	0.626(1)	0.243(2)	0.363(2)
C(20)	0.506(1)	0.170(1)	0.367(1)
C(21)	0.3390(9)	0.131(1)	0.646(1)
C(22)	0.396(1)	0.067(1)	0.665(1)
C(23)	0.390(1)	0.045(1)	0.814(2)
C(24)	0.428(1)	0.140(2)	0.931(2)
C(25)	0.365(1)	0.199(1)	0.910(1)
C(26)	0.373(1)	0.228(1)	0.765(1)
C(27)	0.760(2)	0.142(2)	0.852(3)

Table 6.II.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Cd(S_2CN(CH(CH_2)5)2)2]\cdot CH_2Cl_2$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cd	0.0291(5)	0.0399(6)	0.0379(5)	0.0146(5)	-0.0008(4)	0.0077(4)
Cl(1)	0.103(4)	0.177(6)	0.084(3)	0.058(4)	0.015(3)	0.008(4)
Cl(2)	0.173(6)	0.145(5)	0.094(4)	0.108(5)	0.035(4)	0.049(4)
S(1)	0.059(2)	0.042(2)	0.036(2)	0.011(2)	0.013(2)	0.013(2)
S(2)	0.027(2)	0.037(2)	0.039(2)	0.012(2)	0.006(1)	0.013(2)
S(3)	0.030(2)	0.058(2)	0.028(2)	0.009(2)	0.008(1)	0.014(2)
S(4)	0.024(2)	0.066(3)	0.039(2)	0.006(2)	0.010(1)	0.022(2)
N(1)	0.047(7)	0.032(7)	0.030(6)	0.013(6)	0.011(5)	0.002(5)
N(2)	0.017(5)	0.041(7)	0.031(6)	0.005(5)	0.012(4)	0.010(5)
C(1)	0.023(6)	0.022(7)	0.035(6)	0.014(6)	-0.003(5)	0.002(6)
C(2)	0.049(9)	0.033(8)	0.038(8)	0.012(7)	0.014(7)	0.003(6)
C(3)	0.07(1)	0.07(1)	0.044(8)	0.047(9)	0.012(7)	0.014(8)
C(4)	0.11(1)	0.08(1)	0.031(8)	0.03(1)	0.016(9)	0.019(8)
C(5)	0.08(1)	0.09(1)	0.047(10)	0.03(1)	0.042(9)	-0.002(9)
C(6)	0.05(1)	0.17(2)	0.06(1)	0.04(1)	0.019(9)	0.00(1)
C(7)	0.06(1)	0.11(1)	0.047(9)	0.05(1)	0.011(8)	-0.002(9)
C(8)	0.06(1)	0.015(8)	0.050(8)	0.006(7)	0.001(7)	0.007(6)
C(9)	0.12(1)	0.04(1)	0.08(1)	0.04(1)	0.02(1)	0.035(9)
C(10)	0.20(3)	0.05(1)	0.10(2)	0.02(2)	-0.01(2)	0.04(1)
C(11)	0.19(3)	0.08(2)	0.07(1)	-0.01(2)	0.02(2)	0.05(1)
C(12)	0.10(2)	0.08(1)	0.07(1)	-0.03(1)	0.03(1)	0.00(1)
C(13)	0.049(10)	0.06(1)	0.057(9)	0.004(8)	0.016(7)	0.020(8)

Table 6.II.3: Cont.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(14)	0.023(7)	0.051(9)	0.032(7)	0.008(6)	0.011(5)	0.016(6)
C(15)	0.021(7)	0.056(10)	0.032(7)	0.012(7)	0.020(5)	0.008(7)
C(16)	0.056(10)	0.043(10)	0.060(9)	0.004(8)	0.009(7)	0.016(8)
C(17)	0.07(1)	0.07(1)	0.08(1)	-0.01(1)	0.012(10)	0.00(1)
C(18)	0.05(1)	0.12(2)	0.07(1)	-0.01(1)	0.029(9)	0.04(1)
C(19)	0.029(9)	0.13(2)	0.056(10)	0.016(10)	0.036(7)	0.03(1)
C(20)	0.035(8)	0.08(1)	0.050(8)	0.026(8)	0.025(7)	0.041(8)
C(21)	0.017(6)	0.064(10)	0.026(7)	0.010(7)	0.016(5)	0.022(7)
C(22)	0.054(9)	0.051(9)	0.032(7)	0.018(8)	0.009(6)	0.020(7)
C(23)	0.05(1)	0.12(2)	0.08(1)	0.04(1)	0.026(9)	0.06(1)
C(24)	0.07(1)	0.12(2)	0.039(9)	0.05(1)	0.019(8)	0.03(1)
C(25)	0.07(1)	0.13(2)	0.027(8)	0.04(1)	0.029(7)	0.026(9)
C(26)	0.07(1)	0.07(1)	0.042(8)	0.034(9)	0.022(7)	0.011(8)
C(27)	0.16(2)	0.18(3)	0.16(2)	0.15(2)	-0.04(2)	-0.04(2)

Table 6.II.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Cd(S_2CN(CH(CH_2)_5)_2)_2]\cdot CH_2Cl_2$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.0038	-0.2091	-0.3337	3.6481
H(3a)	-0.1876	-0.3920	-0.4493	5.0126
H(3b)	-0.1791	-0.2797	-0.4240	5.0126
H(4a)	-0.0580	-0.2373	-0.5903	6.5641
H(4b)	-0.1619	-0.3389	-0.6621	6.5641
H(5a)	-0.0718	-0.4387	-0.6423	7.1174
H(5b)	0.0003	-0.3505	-0.7172	7.1174
H(6a)	0.1039	-0.3722	-0.5393	8.9045
H(6b)	0.1155	-0.2588	-0.5132	8.9045
H(7a)	0.0893	-0.3114	-0.3003	6.0304
H(7b)	-0.0160	-0.4120	-0.3710	6.0304
H(8a)	-0.1701	-0.4341	-0.2556	4.2311
H(9a)	-0.0906	-0.3898	0.0292	6.9818
H(9b)	-0.0415	-0.4317	-0.0973	6.9818
H(10a)	-0.1959	-0.5874	-0.1505	11.4592
H(10b)	-0.1555	-0.5652	0.0104	11.4592
H(11a)	-0.2871	-0.5090	0.0728	11.8333
H(11b)	-0.3471	-0.6197	-0.0266	11.8333
H(12a)	-0.4098	-0.5047	-0.0916	9.9754
H(12b)	-0.3605	-0.5485	-0.2160	9.9754
H(13a)	-0.2939	-0.3732	-0.2040	5.3503
H(13b)	-0.2559	-0.3478	-0.0419	5.3503

Table 6.II.4: Cont.

Atom	*x*	*y*	*z*	B_{eq}
H(15a)	0.5017	0.2439	0.5626	3.1849
H(16a)	0.4231	0.3104	0.3538	5.2248
H(16b)	0.4425	0.3592	0.5161	5.2248
H(17a)	0.5902	0.4567	0.3930	7.7430
H(17b)	0.6301	0.4225	0.5184	7.7430
H(18a)	0.7110	0.3863	0.3240	8.5006
H(18b)	0.5971	0.3232	0.2323	8.5006
H(19a)	0.6669	0.2590	0.4551	6.6636
H(19b)	0.6561	0.2084	0.2948	6.6636
H(20a)	0.4664	0.1515	0.2756	4.1951
H(20b)	0.4995	0.1084	0.3958	4.1951
H(21a)	0.2619	0.0877	0.6512	3.0030
H(22a)	0.4717	0.1043	0.6512	3.8958
H(22b)	0.3632	0.0029	0.5960	3.8958
H(23a)	0.4348	0.0104	0.8250	6.8387
H(23b)	0.3159	-0.0003	0.8209	6.8387
H(24a)	0.4172	0.1200	1.0195	6.8135
H(24b)	0.5045	0.1833	0.9292	6.8135
H(25a)	0.2899	0.1581	0.9191	6.5148
H(25b)	0.3935	0.2626	0.9821	6.5148
H(26a)	0.3260	0.2596	0.7531	4.8625
H(26b)	0.4469	0.2756	0.7597	4.8625
H(27a)	0.8389	0.1640	0.8471	13.9996
H(27b)	0.7256	0.0644	0.8440	13.9996

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 6.II.5: Bond distances (Å) for $[Cd(S_2CN(CH(CH_2)5)2)2]\cdot CH_2Cl_2$

Cd — S(1)	2.540(4)	C(5) — C(6)	1.49(2)
Cd — S(2)	2.897(3)	C(6) — C(7)	1.52(2)
Cd — S(2)*	2.551(3)	C(8) — C(9)	1.52(2)
Cd — S(3)	2.560(3)	C(8) — C(13)	1.50(2)
Cd — S(4)	2.596(3)	C(9) — C(10)	1.50(2)
Cl(1) — C(27)	1.66(2)	C(10) — C(11)	1.53(3)
Cl(2) — C(27)	1.69(2)	C(11) — C(12)	1.51(3)
S(1) — C(1)	1.73(1)	C(12) — C(13)	1.52(2)
S(2) — C(1)	1.76(1)	C(15) — C(16)	1.48(2)
S(3) — C(14)	1.72(1)	C(15) — C(20)	1.52(2)
S(4) — C(14)	1.74(1)	C(16) — C(17)	1.54(2)
N(1) — C(1)	1.32(1)	C(17) — C(18)	1.50(2)
N(1) — C(2)	1.49(1)	C(18) — C(19)	1.51(3)
N(1) — C(8)	1.49(1)	C(19) — C(20)	1.52(2)
N(2) — C(14)	1.34(1)	C(21) — C(22)	1.46(2)
N(2) — C(15)	1.48(1)	C(21) — C(26)	1.52(2)
N(2) — C(21)	1.49(1)	C(22) — C(23)	1.52(2)
C(2) — C(3)	1.53(2)	C(23) — C(24)	1.48(2)
C(2) — C(7)	1.51(2)	C(24) — C(25)	1.48(2)
C(3) — C(4)	1.52(2)	C(25) — C(26)	1.51(2)
C(4) — C(5)	1.52(2)		

* symmetry operation: *-x, -y, -z*

Table 6.II.6: Bond angles (deg.) for $[Cd(S_2CN(CH(CH_2)_5)_2)_2]\cdot CH_2Cl_2$

S(1) — Cd — S(2)	65.4(1)	N(1) — C(2) — C(7)	113(1)
S(1) — Cd — S(2)*	102.0(1)	C(3) — C(2) — C(7)	111(1)
S(1) — Cd — S(3)	139.4(1)	C(2) — C(3) — C(4)	110(1)
S(1) — Cd — S(4)	108.0(1)	C(3) — C(4) — C(5)	113(1)
S(2) — Cd — S(2)*	92.4(1)	C(4) — C(5) — C(6)	112(1)
S(2) — Cd — S(3)	93.6(1)	C(5) — C(6) — C(7)	112(1)
S(2) — Cd — S(4)	146.1(1)	C(2) — C(7) — C(6)	112(1)
S(2)* — Cd — S(3)	113.8(1)	N(1) — C(8) — C(9)	113(1)
S(2)* — Cd — S(4)	121.2(1)	N(1) — C(8) — C(13)	115(1)
S(3) — Cd — S(4)	69.7(1)	C(9) — C(8) — C(13)	114(1)
Cd — S(1) — C(1)	95.5(4)	C(8) — C(9) — C(10)	111(1)
Cd — S(2) — Cd*	87.6(1)	C(9) — C(10) — C(11)	111(2)
Cd — S(2) — C(1)	83.2(4)	C(10) — C(11) — C(12)	111(2)
Cd — S(2)* — C(1)	99.6(4)	C(11) — C(12) — C(13)	114(2)
Cd — S(3) — C(14)	87.3(4)	C(8) — C(13) — C(12)	110(1)
Cd — S(4) — C(14)	85.8(4)	S(3) — C(14) — S(4)	117.2(6)
C(1) — N(1) — C(2)	121(1)	S(3) — C(14) — N(2)	122.3(8)
C(1) — N(1) — C(8)	124(1)	S(4) — C(14) — N(2)	120.5(8)
C(2) — N(1) — C(8)	115.3(9)	N(2) — C(15) — C(16)	114(1)
C(14) — N(2) — C(15)	124.1(9)	N(2) — C(15) — C(20)	115(1)
C(14) — N(2) — C(21)	121.3(9)	C(16) — C(15) — C(20)	112(1)
C(15) — N(2) — C(21)	114.4(8)	C(15) — C(16) — C(17)	113(1)
S(1) — C(1) — S(2)	115.8(6)	C(16) — C(17) — C(18)	110(1)
S(1) — C(1) — N(1)	122.5(9)	C(17) — C(18) — C(19)	113(1)
S(2) — C(1) — N(1)	121.7(9)	C(18) — C(19) — C(20)	109(1)
N(1) — C(2) — C(3)	114(1)	C(15) — C(20) — C(19)	110(1)

Table 6.II.6: Cont.

N(2) — C(21) — C(22)	113(1)	C(23) — C(24) — C(25)	109(1)
N(2) — C(21) — C(26)	113(1)	C(24) — C(25) — C(26)	113(1)
C(22) — C(21) — C(26)	114(1)	C(21) — C(26) — C(25)	111(1)
C(21) — C(22) — C(23)	111(1)	Cl(1) — C(27) — Cl(2)	117(1)
C(22) — C(23) — C(24)	114(1)		

* symmetry operation: -*x*, -*y*, -*z*

Table 6.II.7: Observed and calculated structure factors for $[Cd(S_2CN(CH(CH_2)_5)_2)_2]\cdot CH_2Cl_2$

h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF
0	0	3	670	670	12	0	3	8	278	329	11	0	7	2	216	211	12	1	-14	5	236	195	24	1	-8	11	156	59	25
0	0	4	298	303	8	0	4	-11	262	310	17	0	7	3	392	393	11	1	-14	8	220	157	34	1	-7	-8	378	350	20
0	0	5	251	264	8	0	4	-9	239	239	15	0	7	4	266	263	12	1	-13	-3	273	215	24	1	-7	-6	393	350	16
0	0	6	174	192	10	0	4	-8	615	664	15	0	7	5	369	392	12	1	-13	-1	240	162	23	1	-7	-5	362	283	15
0	0	7	183	213	13	0	4	-5	651	668	15	0	7	6	269	273	14	1	-13	2	239	196	22	1	-7	-3	579	550	16
0	0	8	228	253	12	0	4	-4	136	169	14	0	7	8	166	217	20	1	-13	3	180	210	27	1	-7	-1	387	389	11
0	0	11	168	225	17	0	4	-3	411	371	10	0	8	-9	180	152	23	1	-13	5	316	313	19	1	-7	0	277	251	12
0	1	-10	217	206	18	0	4	-1	902	867	15	0	8	-8	235	217	18	1	-12	-3	241	186	23	1	-7	1	949	816	19
0	1	-9	205	186	17	0	4	0	267	274	7	0	8	-6	349	361	13	1	-12	2	291	271	20	1	-7	2	699	654	16
0	1	-8	252	293	14	0	4	2	399	387	10	0	8	-5	645	659	15	1	-12	3	273	244	18	1	-7	3	682	603	16
0	1	-6	260	294	10	0	4	3	899	896	16	0	8	-3	509	527	12	1	-12	4	196	153	22	1	-7	5	460	428	12
0	1	-5	258	295	9	0	4	4	109	99	15	0	8	-2	614	661	16	1	-12	5	323	294	17	1	-7	6	215	225	17
0	1	-4	472	450	13	0	4	5	295	299	9	0	8	0	275	272	10	1	-12	8	225	154	23	1	-7	8	387	431	14
0	1	-3	791	753	14	0	4	6	548	555	14	0	8	2	463	485	13	1	-12	10	202	194	29	1	-6	-9	180	164	29
0	1	-2	1191	1138	18	0	4	8	212	257	13	0	8	3	404	412	11	1	-11	-4	218	122	24	1	-6	-8	384	343	18
0	1	2	459	465	10	0	5	-11	269	362	19	0	8	5	330	367	12	1	-11	-3	326	279	19	1	-6	-6	230	221	20
0	1	3	880	926	15	0	5	-9	198	232	19	0	8	7	149	84	18	1	-11	0	732	645	20	1	-6	-3	628	599	15
0	1	4	388	389	11	0	5	-8	402	412	12	0	9	-8	346	330	18	1	-11	1	195	154	24	1	-6	-1	472	486	12
0	1	5	299	317	8	0	5	-5	260	299	10	0	9	-5	526	544	14	1	-11	2	454	407	14	1	-6	0	429	448	12
0	1	6	104	135	15	0	5	-4	206	204	10	0	9	-3	341	349	12	1	-11	3	516	498	14	1	-6	1	384	361	10
0	1	7	215	207	10	0	5	-3	155	165	11	0	9	-2	270	279	13	1	-11	4	291	307	17	1	-6	2	294	320	9
0	1	8	205	247	12	0	5	-1	584	598	13	0	9	-1	144	129	18	1	-11	5	195	206	26	1	-6	3	643	601	17
0	1	9	114	115	18	0	5	0	878	926	15	0	9	0	375	390	11	1	-11	8	237	243	23	1	-6	4	162	112	15
0	1	11	166	166	17	0	5	1	466	523	13	0	9	3	170	178	18	1	-10	-3	410	342	16	1	-6	5	381	374	11
0	2	-10	152	131	22	0	5	3	728	745	15	0	9	5	190	155	17	1	-10	-1	409	372	15	1	-6	6	271	272	12
0	2	-8	377	380	12	0	5	5	241	274	11	0	10	-10	204	195	31	1	-10	0	850	728	23	1	-6	7	150	107	21
0	2	-6	486	505	12	0	5	6	432	416	12	0	10	-8	262	268	24	1	-10	2	373	358	13	1	-6	8	406	409	13
0	2	-5	765	769	15	0	5	8	244	265	14	0	10	-5	511	504	13	1	-10	3	303	300	13	1	-6	10	186	112	18
0	2	-4	581	540	13	0	5	10	140	129	19	0	10	-4	290	295	15	1	-10	4	202	167	18	1	-6	11	232	261	17
0	2	-3	1186	1150	19	0	6	-11	203	242	22	0	10	-3	380	385	13	1	-10	5	562	519	16	1	-5	-8	255	266	21
0	2	-2	583	571	12	0	6	-8	213	207	17	0	10	-2	333	345	14	1	-10	7	177	213	26	1	-5	-7	196	178	21
0	2	-1	74	69	11	0	6	-7	147	134	20	0	10	-1	345	336	12	1	-10	8	232	226	20	1	-5	-6	287	233	14
0	2	0	105	77	9	0	6	-6	152	129	17	0	10	0	446	458	12	1	-9	-5	199	181	27	1	-5	-4	181	183	17
0	2	1	1042	998	16	0	6	-5	334	380	11	0	10	1	248	287	15	1	-9	-3	372	326	15	1	-5	-3	517	492	13
0	2	2	475	452	11	0	6	-4	127	130	15	0	10	3	291	304	13	1	-9	-1	230	216	18	1	-5	-2	374	364	10
0	2	3	854	865	15	0	6	-2	369	382	10	0	11	-10	284	221	27	1	-9	0	569	497	16	1	-5	0	510	533	12
0	2	5	271	278	8	0	6	-1	179	188	11	0	11	-6	246	213	22	1	-9	2	389	403	12	1	-5	1	623	517	14
0	2	6	212	223	9	0	6	0	806	844	15	0	11	-4	348	367	16	1	-9	3	150	329	11	1	-5	2	586	544	14
0	2	7	267	300	10	0	6	1	180	190	10	0	11	-3	289	306	18	1	-9	4	168	162	21	1	-5	3	241	248	11
0	2	8	206	256	12	0	6	2	147	166	13	0	11	-2	326	281	15	1	-9	5	466	435	13	1	-5	4	119	12	17
0	2	9	177	192	12	0	6	3	597	612	16	0	11	-1	370	364	13	1	-9	6	152	94	25	1	-5	5	535	526	13
0	3	-8	569	609	15	0	6	4	133	119	16	0	11	0	289	300	15	1	-8	-8	220	149	26	1	-5	6	315	329	12
0	3	-7	187	204	14	0	6	5	254	282	12	0	11	1	254	247	16	1	-8	-6	483	402	16	1	-5	7	139	182	22
0	3	-6	318	328	10	0	6	6	334	347	12	0	12	-7	225	113	29	1	-8	-5	336	285	17	1	-5	8	716	698	17
0	3	-5	1062	1038	19	0	6	7	229	188	14	0	12	-6	308	262	23	1	-8	-3	436	412	13	1	-5	10	194	193	19
0	3	-4	539	520	14	0	6	8	287	308	14	0	12	-3	325	276	18	1	-8	-1	327	292	13	1	-5	11	229	287	18
0	3	-3	861	857	15	0	6	9	167	178	18	0	12	-2	300	276	19	1	-8	0	471	409	13	1	-4	-9	156	158	26
0	3	-1	570	604	11	0	7	-8	337	366	14	0	12	3	180	168	23	1	-8	1	255	242	13	1	-4	-8	326	311	15
0	3	0	244	213	7	0	7	-6	274	262	14	0	13	-2	226	223	26	1	-8	2	677	670	17	1	-4	-7	394	364	13
0	3	1	996	961	16	0	7	-5	640	689	18	0	13	0	215	203	22	1	-8	3	373	377	10	1	-4	-6	504	463	14
0	3	2	876	848	15	0	7	-3	485	516	11	0	13	3	283	287	20	1	-8	4	303	282	12	1	-4	-5	520	490	14
0	3	3	1146	1100	19	0	7	-2	918	920	18	0	14	0	234	211	26	1	-8	5	751	710	20	1	-4	-3	178	182	13
0	3	5	429	454	12	0	7	-1	302	303	9	0	15	0	291	237	28	1	-8	6	482	490	13	1	-4	-2	321	323	9
0	3	6	658	660	16	0	7	0	434	484	11	1	-15	0	213	162	35	1	-8	7	304	267	14	1	-4	-1	445	462	11
0	3	7	362	398	10	0	7	1	177	182	12	1	-14	-3	257	232	28	1	-8	8	217	240	20	1	-4	0	1142	1257	19
1	-4	1	543	535	12	1	0	-4	1088	1075	18	1	3	3	1301	1229	21	1	7	-2	474	518	13	1	12	0	196	155	21
1	-4	3	150	147	13	1	0	-3	1223	1237	19	1	3	5	360	371	10	1	7	-1	620	621	16	1	13	-4	197	100	31
1	-4	4	311	333	10	1	0	-2	201	180	7	1	3	6	484	486	13	1	7	0	442	475	12	1	13	-3	249	214	28
1	-4	5	817	806	18	1	0	2	617	567	11	1	3	7	139	171	18	1	7	1	158	167	14	1	13	2	304	278	19
1	-4	6	409	404	11	1	0	3	626	611	12	1	3	8	121	99	19	1	7	2	590	581	16	1	14	-3	220	155	36
1	-4	7	131	157	21	1	0	4	177	165	8	1	3	10	120	187	18	1	7	3	183	164	14	1	14	-1	219	215	32
1	-4	8	696	695	16	1	0	6	224	239	10	1	4	-12	280	318	21	1	7	4	298	298	12	1	14	0	307	233	23
1	-4	11	168	220	21	1	0	7	234	260	11	1	4	-8	503	519	14	1	7	5	331	348	13	1	14	2	257	202	26
1	-3	-7	196	214	17	1	0	8	381	400	11	1	4	-5	269	272	10	1	7	6	288	286	13	1	15	-3	296	199	34
1	-3	-6	681	682	19	1	0	11	195	247	16	1	4	-4	360	370	10	1	7	8	159	150	21	1	15	-1	277	250	32
1	-3	-5	610	594	17	1	1	-10	169	173	23	1	4	-3	321	312	9	1	8	-9	309	319	19	2	-15	5	213	170	30
1	-3	-4	321	303	9	1	1	-8	234	258	16	1	4	-2	343	366	9	1	8	-8	322	327	19	2	-15	8	317	263	31
1	-3	-3	543	518	14	1	1	-7	531	527	14	1	4	-1	439	424	12	1	8	-6	392	379	14	2	-14	5	399	303	17
1	-3	-2	425	382	10	1	1	-6	448	465	11	1	4	0	442	433	12	1	8	-5	534	534	13	2	-14	8	273	222	29
1	-3	-1	765	801	14	1	1	-5	329	336	9	1	4	1	143	103	10	1	8	-4	235	238	14	2	-13	0	212	180	30
1	-3	0	1432	1763	22	1	1	-4	404	396	10	1	4	2	555	534	13	1	8	-3	260	260	12	2	-13	2	291	269	20
1	-3	1	143	120	10	1	1	-3	1119	1088	18	1	4	3	1149	1106	19	1	8	-2	326	325	11	2	-13	4	190	116	25
1	-3	2	474	490	12	1	1	-2	215	212	7	1	4	5	377	361	11	1	8	-1	306	298	11	2	-13	5	399	359	17
1	-3	3	533	515	13	1	1	-1	1210	1195	18	1	4	6	275	279	11	1	8	1	386	400	10	2	-12	-6	204	97	32
1	-3	4	543	511	14	1	1	1	264	255	6	1	4	7	355	353	11	1	8	2	560	549	14	2	-12	-3	197	209	30
1	-3	5	816	793	17	1	1	2	116	110	10	1	4	10	133	160	21	1	8	3	133	148	22	2	-12	-1	194	199	30
1	-3	6	387	385	10	1	1	3	118	115	10	1	5	-12	234	225	23	1	8	4	232	236	14	2	-12	0	523	474	15
1	-3	7	370	374	11	1	1	4	292	301	8	1	5	-8	209	208	19	1	8	5	277	317	15	2	-12	2	204	171	24
1	-3	8	249	259	14	1	1	5	157	164	9	1	5	-5	140	142	17	1	8	6	193	215	17	2	-12	5	235	161	21
1	-2	-8	157	139	17	1	1	6	292	318	9	1	5	-4	317	343	9	1	8	7	136	72	21	2	-12	9	213	166	24
1	-2	-7	137	130	19	1	1	7	126	114	17	1	5	-3	384	412	10	1	9	-9	345	352	23	2	-11	-4	258	287	24
1	-2	-6	369	378	10	1	1	8	259	305	11	1	5	-1	1032	1025	18	1	9	-8	253	281	25	2	-11	-3	384	372	17
1	-2	-5	495	511	12	1	1	11	219	233	14	1	5	0	739	744	15	1	9	-6	286	309	19	2	-11	-1	418	413	16
1	-2	-3	663	657	14	1	2	-9	205	201	18	1	5	1	172	146	10	1	9	-5	392	379	13	2	-11	0	681	620	18
1	-2	-2	173	181	8	1	2	-8	306	302	14	1	5	2	270	286	9	1	9	-4	430	418	12	2	-11	2	454	415	14
1	-2	-1	218	221	7	1	2	-7	433	428	11	1	5	3	535	533	15	1	9	-3	174	136	18	2	-11	3	397	377	13
1	-2	1	452	450	10	1	2	-6	429	477	11	1	5	4	129	112	17	1	9	-2	190	194	17	2	-11	8	219	185	23
1	-2	2	746	734	13	1	2	-5	477	478	12	1	5	5	483	448	13	1	9	-1	272	260	13	2	-10	-4	310	281	19
1	-2	3	886	863	15	1	2	-4	141	130	13	1	5	6	210	196	14	1	9	0	194	191	15	2	-10	-3	493	473	15
1	-2	4	522	442	14	1	2	-3	102	93	15	1	5	7	319	323	12	1	9	3	183	189	19	2	-10	-1	459	442	14
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1	-2	7	442	430	11	1	2	-1	1153	1166	18	1	6	-6	140	126	22	1	10	-4	367	357	15	2	-10	1	195	179	20
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11	-7	0	203	238	28
11	-7	2	600	568	16
11	-7	5	166	198	23
11	-7	6	148	124	24
11	-6	-3	286	252	24
11	-6	-1	250	264	22
11	-6	2	402	345	14
11	-6	3	294	266	14
11	-6	5	378	368	14
11	-6	8	180	191	22
11	-5	-3	292	290	23
11	-5	-1	204	201	21
11	-5	2	427	366	13
11	-5	3	230	210	16
11	-5	5	618	564	16
11	-5	8	238	262	17
11	-4	-3	248	268	22
11	-4	-1	289	296	18
11	-4	2	532	494	15
11	-4	3	160	119	21
11	-4	5	606	550	16
11	-4	8	207	218	18
11	-3	-6	201	260	32
11	-3	-4	311	302	21
11	-3	-3	284	301	20
11	-3	-1	253	265	17
11	-3	0	321	343	15
11	-3	1	367	344	14
11	-3	2	480	488	14
11	-3	3	176	182	20
11	-3	4	158	181	22
11	-3	5	482	466	13
11	-2	-6	206	227	28
11	-2	-3	344	397	17
11	-2	0	475	495	13
11	-2	1	294	327	15
11	-2	2	356	369	12
11	-2	4	236	257	14
11	-2	5	296	298	12
11	-2	7	134	163	21
11	-2	8	162	153	18
11	-1	-6	333	383	23
11	-1	-5	188	123	30
11	-1	-4	262	309	23
11	-1	-3	448	513	16
11	-1	0	433	449	13
11	-1	1	149	142	21
11	-1	2	274	274	14
11	-1	4	196	195	14
11	-1	5	186	231	15
11	-1	8	169	191	18
11	0	-6	356	441	28
11	0	-4	394	410	17
11	0	-3	453	500	17
11	0	0	251	226	17
11	0	4	184	190	15
11	0	5	200	245	14
11	1	-7	245	231	35
11	1	-6	349	374	26
11	1	-4	225	242	29
11	1	-3	410	450	18
11	1	5	133	156	21
11	2	-7	230	252	38
11	2	-3	310	322	22
11	2	-1	181	188	26
11	2	0	248	247	19
11	2	3	198	239	22
11	2	5	155	191	23
11	3	-7	218	195	36
11	3	-4	215	111	27
11	3	0	321	342	19
11	3	2	162	167	25
11	3	3	313	281	15
11	4	-3	235	255	29
11	4	0	405	424	16
11	4	2	178	174	26
11	4	3	178	208	25
11	5	-3	250	287	26
11	5	0	367	387	18
11	5	2	176	173	26
11	5	3	171	140	28
11	6	-7	227	69	34
11	6	0	253	273	23
12	-17	5	297	106	28
12	-15	-4	252	119	40
12	-15	5	204	191	32
12	-15	7	222	162	29
12	-14	5	235	252	27
12	-13	-3	219	78	34
12	-13	4	199	185	27
12	-13	5	246	245	24
12	-12	-3	239	166	32
12	-12	5	217	201	24
12	-12	8	170	93	27
12	-11	-6	262	231	37
12	-11	-3	267	314	34
12	-11	-1	326	284	24
12	-11	2	202	223	27
12	-11	4	227	242	22
12	-11	7	265	221	17
12	-10	-3	352	412	26
12	-10	2	235	189	23
12	-9	-3	355	337	24
12	-9	-1	309	298	21
12	-9	2	223	209	22
12	-8	-3	218	189	32
12	-8	-2	263	228	26
12	-8	-1	398	401	20
12	-8	1	172	162	26
12	-8	2	332	360	18
12	-8	5	263	270	16
12	-7	-2	238	225	25
12	-7	-1	350	387	20
12	-7	2	372	378	16
12	-7	5	316	328	15
12	-7	8	207	128	17
12	-6	-1	379	359	17
12	-6	0	342	335	18
12	-6	2	389	378	15
12	-6	3	217	190	20
12	-6	5	358	374	14
12	-6	8	150	155	24
12	-5	-3	351	364	19
12	-5	-2	181	181	30
12	-5	0	448	411	15
12	-5	2	504	468	14
12	-5	4	278	260	14
12	-5	5	301	360	17
12	-4	-3	230	280	27
12	-4	-2	245	281	24
12	-4	0	426	438	15
12	-4	1	434	413	14
12	-4	2	490	479	14
12	-4	4	289	272	15
12	-4	5	296	316	14
12	-3	-1	275	276	18
12	-3	0	223	202	19
12	-3	1	522	549	14
12	-3	2	254	287	16
12	-3	3	145	105	21
12	-3	4	242	275	17
12	-3	5	284	258	13
12	-2	-6	263	244	26
12	-2	-3	306	309	18
12	-2	-2	170	190	28
12	-2	-1	262	288	18
12	-2	1	265	295	17
12	-2	2	160	200	21
12	-2	5	200	205	16
12	-1	-4	214	245	28
12	-1	3	296	342	19
12	-1	-2	181	175	25
12	-1	-1	166	161	23
12	-1	4	202	236	16
12	-1	5	163	202	19
12	0	0	163	129	24
12	0	4	205	260	14
12	0	5	163	170	15
12	1	-7	276	330	35
12	1	0	207	185	23
12	1	3	157	171	22
12	1	4	157	189	20
12	2	-3	198	190	32
12	2	0	243	249	22
12	2	2	208	189	20
12	2	3	206	171	18
12	2	4	161	170	25
12	3	0	335	361	19
12	4	-4	212	284	33
12	4	-3	205	239	32
12	4	0	262	291	23
12	5	-1	227	247	26
12	6	-1	234	239	24
13	-15	4	216	185	35
13	-14	1	274	224	26
13	-14	2	274	217	24
13	-13	-5	235	32	38
13	-13	2	235	216	27
13	-13	5	183	123	27
13	-13	7	205	155	22
13	-12	-3	223	198	37
13	-12	2	193	169	29
13	-11	1	204	184	27
13	-11	3	184	190	28
13	-11	4	188	243	29
13	-10	4	181	163	25
13	-9	6	190	134	22
13	-9	7	183	173	23
13	-7	-3	261	186	26
13	-7	0	333	369	20
13	-7	1	262	249	21
13	-7	4	251	169	17
13	-6	-4	255	240	31
13	-6	-1	313	278	23
13	-6	0	327	354	21
13	-6	1	302	336	21
13	-6	4	275	280	17
13	-5	-3	249	234	26
13	-5	-2	251	235	25
13	-5	-1	285	287	22
13	-5	1	386	382	17
13	-5	4	320	335	15
13	-5	5	148	116	24
13	-4	-4	244	223	29
13	-4	-2	334	356	20
13	-4	-1	294	319	22
13	-4	1	397	395	16
13	-4	2	309	336	17
13	-4	4	265	273	16
13	-3	-3	231	75	25
13	-3	-2	333	353	20
13	-3	-1	262	257	20
13	-3	2	221	252	20
13	-2	-2	294	308	20
13	-2	4	189	230	20
13	-1	-7	292	292	28
13	-1	1	177	226	23
13	-1	4	268	322	14
13	-1	5	167	199	18
13	0	-7	302	260	30
13	0	1	175	226	24
13	0	4	256	300	14
13	1	1	319	281	16
13	1	2	156	142	24
13	2	-1	220	211	25
13	2	1	243	253	22
13	2	4	239	228	19
13	3	-1	273	282	23
13	4	-4	304	360	28
13	4	-1	254	245	25
13	5	-4	252	325	31
13	5	-1	204	212	28
14	-12	0	219	121	29
14	-11	4	233	246	24
14	-11	5	187	125	25
14	-10	1	215	160	21
14	-10	4	215	222	24
14	-10	7	196	205	23
14	-9	1	233	260	27
14	-9	4	204	225	25
14	-9	6	200	207	22
14	-9	7	173	192	26
14	-8	1	318	319	21
14	-8	4	278	288	19
14	-8	6	245	221	18
14	-8	7	156	144	24
14	-7	-1	213	245	31
14	-7	1	277	251	21
14	-7	4	268	285	19
14	-6	-4	264	369	34
14	-6	-1	324	333	21
14	-6	0	192	86	28
14	-6	1	201	226	27
14	-6	4	225	220	21
14	-6	6	187	148	20
14	-5	-6	253	69	30
14	-5	-5	210	131	34
14	-5	-4	217	278	35
14	-5	-2	301	352	24
14	-5	-1	334	317	20
14	-5	1	264	285	23
14	-5	4	162	186	25
14	-4	-2	340	350	21
14	-4	-1	304	279	22
14	-4	1	335	370	18
14	-3	-3	195	204	32
14	-3	1	263	333	20
14	-3	2	169	147	24
14	-3	4	155	176	24
14	-2	1	224	265	19
14	-2	4	265	289	16
14	-1	1	262	274	17
14	-1	2	156	182	23
14	-1	4	260	336	17
14	0	1	304	298	17
14	0	4	216	294	16
14	1	-5	229	206	35
14	1	1	196	224	27
14	2	-1	230	236	27
14	3	-4	289	296	28
15	-12	1	255	211	25
15	-10	4	258	311	23
15	-9	3	180	136	26
15	-9	4	203	241	28
15	-8	-4	236	225	35
15	-8	4	235	193	21
15	-5	-4	217	222	35
15	-5	4	223	175	19
15	-4	-4	219	184	33
15	-3	-1	215	212	26
15	-3	1	192	221	24
15	-2	1	238	243	20
15	-2	4	189	243	20
15	-1	1	227	237	21
15	0	0	233	252	24
15	1	-1	218	165	26
15	1	0	220	243	26
16	-11	3	199	194	31
16	-8	4	198	144	25
16	-5	0	210	77	27
16	-2	-1	210	247	32
16	-2	0	196	218	28
17	-12	0	258	216	28

APPENDIX 6.III

$[Cd(S_2CNiBu)_2]$

Table 6.III.1: Crystallographic Parameters for $[Cd(S_2CN(CH_2CH(CH_2)_2)_2)_2]$

Formula	$C_{18}H_{36}CdN_2S_4$
Formula weight	521.15
Crystal morphology / colour	block / colourless
Crystal dimensions (mm)	0.13 x 0.19 x 0.32
Crystal system	monoclinic
Space group	*C*2/*c* (#15)
a (Å)	49.97(5)
b (Å)	9.597(3)
c (Å)	23.675(8)
α (°)	90
β (°)	116.41(3)
γ (°)	90
V (Å^3)	10169(9)
Z	16
$\rho_{calc.}$ (g cm^{-3})	1.361
F(000)	4320
Temperature (°K)	293
μ (cm^{-1})	11.93
θ limits, cell (°)	19.5 – 21.0
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 35, 0 – 12, -28 – 28
Range of transmission factors	0.968 – 1
Scan technique	ω:2θ
No. of data measured	9002
No. of unique data	8569
R_{amal}	0.089
No. of parameters	451
R (I≥0σ(I))	0.116
R_w (I≥0σ(I))	0.150
R (I≥3σ(I))	0.060
R_w (I≥3σ(I))	0.070
Maximum and minimum residual electron density (e Å^{-3})	2.73, -1.89

Table 6.III.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Cd(S_2CN(CH_2CH(CH_3)2)2)2]$

Atom	*x*	*y*	*z*
Cd(1)	-0.01339(1)	-0.08684(6)	-0.45207(3)
Cd(2)	-0.27705(2)	-0.34796(7)	-0.57333(3)
S(1a)	-0.06620(5)	-0.0080(2)	-0.47434(9)
S(1b)	-0.28251(6)	-0.2970(3)	-0.6833(1)
S(2a)	-0.02620(5)	-0.1758(2)	-0.36360(9)
S(2b)	-0.33055(6)	-0.4251(3)	-0.66009(10)
S(3b)	-0.26737(6)	-0.5495(3)	-0.4966(1)
S(3a)	0.02352(5)	-0.2686(2)	-0.45852(8)
S(4a)	-0.02103(5)	-0.1298(2)	-0.57452(8)
S(4b)	-0.21729(6)	-0.3709(2)	-0.48646(10)
N(1a)	-0.0822(2)	-0.1020(6)	-0.3876(3)
N(1b)	-0.3344(2)	-0.3745(6)	-0.7741(3)
N(2a)	0.0116(1)	-0.3534(5)	-0.5742(2)
N(2b)	-0.2126(2)	-0.5706(6)	-0.4024(3)
C(1b)	-0.3177(2)	-0.3675(7)	-0.7124(3)
C(1a)	-0.0601(2)	-0.0976(7)	-0.4056(3)
C(2a)	-0.0770(2)	-0.1679(8)	-0.3276(3)
C(2b)	-0.3630(2)	-0.448(1)	-0.8000(4)
C(3a)	-0.0698(3)	-0.070(1)	-0.2730(4)
C(3b)	-0.3618(4)	-0.592(2)	-0.8181(10)
C(4a)	-0.0639(2)	-0.1510(10)	-0.2140(4)
C(4b)	-0.3930(4)	-0.657(2)	-0.8415(8)
C(5a)	-0.0563(5)	0.051(1)	-0.2686(5)
C(5b)	-0.3436(4)	-0.656(1)	-0.8312(8)

Table 6.III.2: Cont.

Atom	*x*	*y*	*z*
C(6b)	-0.3263(2)	-0.3102(9)	-0.8198(3)
C(6a)	-0.1111(2)	-0.0394(9)	-0.4241(4)
C(7a)	-0.1317(2)	-0.127(1)	-0.4781(4)
C(7b)	-0.3374(4)	-0.166(2)	-0.8385(9)
C(8a)	-0.1613(3)	-0.045(1)	-0.5131(5)
C(8b)	-0.3571(6)	-0.100(1)	-0.8273(7)
C(9a)	-0.1378(2)	-0.268(1)	-0.4597(5)
C(9b)	-0.3291(3)	-0.115(2)	-0.8888(7)
C(10b)	-0.2310(2)	-0.5034(7)	-0.4559(3)
C(10a)	0.0051(2)	-0.2604(7)	-0.5394(3)
C(11b)	-0.2234(2)	-0.6880(9)	-0.3778(4)
C(11a)	0.0328(2)	-0.4673(7)	-0.5451(3)
C(12b)	-0.2292(3)	-0.661(1)	-0.3222(5)
C(12a)	0.0650(2)	-0.4301(8)	-0.5213(4)
C(13b)	-0.2372(4)	-0.794(1)	-0.2991(7)
C(13a)	0.0834(2)	-0.556(1)	-0.4864(4)
C(14b)	-0.2532(4)	-0.555(1)	-0.3346(8)
C(14a)	0.0731(2)	-0.386(1)	-0.5735(5)
C(15b)	-0.1815(2)	-0.5310(10)	-0.3647(4)
C(15a)	-0.0037(2)	-0.3522(8)	-0.6435(3)
C(16b)	-0.1600(3)	-0.631(1)	-0.3708(6)
C(16a)	-0.0282(2)	-0.4627(8)	-0.6724(3)
C(17b)	-0.1282(3)	-0.578(2)	-0.3290(6)
C(17a)	-0.0539(2)	-0.4378(8)	-0.6558(4)
C(18b)	-0.1643(3)	-0.680(2)	-0.4282(8)
C(18a)	-0.0394(2)	-0.464(1)	-0.7437(4)

Table 6.III.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Cd(S_2CN(CH_2CH(CH_3)2)2)2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cd(1)	0.0507(5)	0.0445(3)	0.0574(3)	-0.0021(3)	0.0356(3)	-0.0044(3)
Cd(2)	0.0747(6)	0.0613(4)	0.0511(3)	-0.0027(4)	0.0067(3)	-0.0046(3)
S(1a)	0.056(2)	0.071(1)	0.060(1)	0.011(1)	0.035(1)	0.017(1)
S(1b)	0.058(2)	0.086(2)	0.065(1)	-0.016(1)	0.015(1)	-0.005(1)
S(2a)	0.048(2)	0.065(1)	0.058(1)	0.008(1)	0.031(1)	0.0091(10)
S(2b)	0.074(2)	0.099(2)	0.051(1)	-0.024(1)	0.027(1)	-0.007(1)
S(3b)	0.053(2)	0.081(2)	0.092(2)	-0.012(1)	0.009(1)	0.011(1)
S(3a)	0.060(2)	0.047(1)	0.0425(10)	0.0088(10)	0.025(1)	0.0006(8)
S(4a)	0.036(1)	0.040(1)	0.0510(10)	0.0060(9)	0.0170(9)	-0.0016(8)
S(4b)	0.060(2)	0.067(2)	0.064(1)	-0.003(1)	0.026(1)	-0.002(1)
N(1a)	0.042(5)	0.052(4)	0.046(3)	0.001(3)	0.028(4)	-0.004(3)
N(1b)	0.051(6)	0.064(4)	0.045(3)	-0.010(4)	0.016(3)	0.000(3)
N(2a)	0.043(5)	0.036(3)	0.041(3)	0.003(3)	0.020(3)	-0.003(3)
N(2b)	0.041(5)	0.048(4)	0.049(3)	-0.002(3)	0.016(3)	0.002(3)
C(1b)	0.045(6)	0.050(5)	0.046(4)	-0.001(4)	0.011(4)	-0.002(3)
C(1a)	0.062(7)	0.037(4)	0.049(4)	-0.001(4)	0.024(4)	-0.012(3)
C(2a)	0.048(7)	0.057(5)	0.056(4)	-0.006(4)	0.038(4)	0.000(4)
C(2b)	0.054(8)	0.105(8)	0.045(4)	-0.011(6)	0.017(4)	-0.001(5)
C(3a)	0.14(1)	0.068(7)	0.051(5)	0.012(6)	0.039(6)	-0.003(5)
C(3b)	0.12(2)	0.18(2)	0.39(3)	-0.10(1)	0.17(2)	-0.22(2)
C(4a)	0.109(9)	0.089(7)	0.049(5)	0.012(6)	0.036(5)	0.005(5)
C(4b)	0.17(2)	0.22(2)	0.29(2)	-0.13(1)	0.13(2)	-0.18(2)
C(5a)	0.41(3)	0.090(10)	0.084(8)	-0.10(1)	0.09(1)	-0.033(7)
C(5b)	0.14(2)	0.077(9)	0.24(2)	0.001(9)	0.08(1)	-0.030(10)

Table 6.III.3: Cont.

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(6b)	0.043(7)	0.086(7)	0.054(5)	-0.007(5)	0.014(4)	0.005(4)
C(6a)	0.047(7)	0.071(6)	0.059(5)	0.007(5)	0.030(5)	0.006(4)
C(7a)	0.034(7)	0.115(9)	0.067(5)	0.000(6)	0.022(5)	-0.012(6)
C(7b)	0.18(2)	0.17(2)	0.28(2)	0.11(1)	0.18(2)	0.15(2)
C(8a)	0.08(1)	0.19(1)	0.075(7)	0.043(9)	0.019(6)	0.002(8)
C(8b)	0.40(3)	0.10(1)	0.16(1)	0.12(1)	0.17(2)	0.062(9)
C(9a)	0.081(10)	0.116(9)	0.103(8)	-0.038(7)	0.048(7)	-0.046(7)
C(9b)	0.15(2)	0.18(1)	0.18(1)	0.03(1)	0.08(1)	0.11(1)
C(10b)	0.044(6)	0.043(4)	0.056(4)	-0.008(4)	0.021(4)	-0.015(4)
C(10a)	0.041(6)	0.034(4)	0.057(4)	-0.008(3)	0.029(4)	0.000(3)
C(11b)	0.081(9)	0.072(6)	0.069(5)	0.007(5)	0.033(5)	0.005(5)
C(11a)	0.035(6)	0.047(4)	0.058(4)	0.008(4)	0.021(4)	-0.007(4)
C(12b)	0.11(1)	0.106(10)	0.120(9)	0.008(8)	0.066(8)	0.006(7)
C(12a)	0.039(7)	0.050(5)	0.067(5)	0.002(4)	0.025(4)	-0.017(4)
C(13b)	0.23(2)	0.12(1)	0.20(1)	0.03(1)	0.16(1)	0.08(1)
C(13a)	0.072(8)	0.086(7)	0.079(6)	0.011(6)	0.024(6)	-0.002(5)
C(14b)	0.29(2)	0.10(1)	0.24(2)	-0.05(1)	0.21(2)	-0.05(1)
C(14a)	0.075(9)	0.098(8)	0.108(8)	-0.009(6)	0.057(7)	0.000(6)
C(15b)	0.040(8)	0.084(7)	0.063(5)	-0.011(5)	0.007(5)	0.006(5)
C(15a)	0.046(6)	0.053(5)	0.047(4)	-0.003(4)	0.029(4)	-0.008(4)
C(16b)	0.045(10)	0.17(1)	0.114(9)	0.017(8)	0.015(7)	-0.036(9)
C(16a)	0.059(7)	0.048(5)	0.047(4)	0.003(4)	0.018(4)	-0.007(4)
C(17b)	0.09(1)	0.26(2)	0.13(1)	0.02(1)	0.018(10)	0.04(1)
C(17a)	0.067(8)	0.057(5)	0.058(5)	-0.008(5)	0.022(5)	-0.007(4)
C(18b)	0.13(1)	0.18(2)	0.23(2)	-0.01(1)	0.09(1)	-0.10(1)
C(18a)	0.063(8)	0.112(8)	0.056(5)	-0.013(6)	0.018(5)	-0.018(5)

Table 6.III.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Cd(S_2CN(CH_2CH(CH_3)2)2)2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	-0.0605	-0.2323	-0.3161	4.5830
H(2b)	-0.0949	-0.2190	-0.3342	4.5830
H(2c)	-0.3768	-0.3982	-0.8370	6.6304
H(2d)	-0.3704	-0.4482	-0.7684	6.6304
H(3a)	-0.0898	-0.0366	-0.2835	8.4251
H(3b)	-0.3542	-0.6286	-0.7756	19.7974
H(4a)	-0.0811	-0.2091	-0.2216	7.7725
H(4b)	-0.0605	-0.0869	-0.1799	7.7725
H(4c)	-0.0465	-0.2096	-0.2027	7.7725
H(4d)	-0.4024	-0.6217	-0.8163	20.6965
H(4e)	-0.4049	-0.6333	-0.8852	20.6965
H(4f)	-0.3910	-0.7576	-0.8369	20.6965
H(5a)	-0.0349	0.0359	-0.2539	18.9745
H(5b)	-0.0593	0.1109	-0.2388	18.9745
H(5c)	-0.0644	0.0961	-0.3095	18.9745
H(5d)	-0.3451	-0.7556	-0.8251	14.5306
H(5e)	-0.3479	-0.6389	-0.8748	14.5306
H(5f)	-0.3234	-0.6253	-0.8038	14.5306
H(6a)	-0.1204	-0.0228	-0.3965	5.3948
H(6b)	-0.1081	0.0487	-0.4407	5.3948
H(6c)	-0.3334	-0.3705	-0.8566	6.0398
H(6d)	-0.3047	-0.3048	-0.8011	6.0398
H(7a)	-0.1228	-0.1394	-0.5069	6.8203
H(7b)	-0.3566	-0.2172	-0.8771	16.2054

Table 6.III.4: Cont.

Atom	*x*	*y*	*z*	B_{eq}
H(8a)	-0.1572	0.0416	-0.5295	11.4928
H(8b)	-0.1695	-0.0233	-0.4840	11.4928
H(8c)	-0.1754	-0.1001	-0.5477	11.4928
H(8d)	-0.3779	-0.1094	-0.8628	19.1737
H(8e)	-0.3582	-0.1388	-0.7900	19.1737
H(8f)	-0.3527	-0.0026	-0.8211	19.1737
H(9a)	-0.1508	-0.3194	-0.4972	9.2128
H(9b)	-0.1475	-0.2580	-0.4324	9.2128
H(9c)	-0.1191	-0.3180	-0.4376	9.2128
H(9d)	-0.3076	-0.1268	-0.8735	15.9469
H(9e)	-0.3392	-0.1692	-0.9282	15.9469
H(9f)	-0.3343	-0.0183	-0.8981	15.9469
H(11a)	0.0298	-0.5021	-0.5099	4.3883
H(11b)	0.0282	-0.5405	-0.5763	4.3883
H(11c)	-0.2421	-0.7201	-0.4118	7.0704
H(11d)	-0.2086	-0.7616	-0.3662	7.0704
H(12a)	0.0695	-0.3537	-0.4916	4.8521
H(12b)	-0.2108	-0.6260	-0.2886	10.1292
H(13a)	0.0789	-0.6334	-0.5154	7.8340
H(13b)	0.0786	-0.5806	-0.4523	7.8340
H(13c)	0.1045	-0.5330	-0.4694	7.8340
H(13d)	-0.2206	-0.8588	-0.2856	15.5113
H(13e)	-0.2547	-0.8354	-0.3328	15.5113
H(13f)	-0.2414	-0.7729	-0.2637	15.5113
H(14a)	0.0702	-0.4636	-0.6017	8.2775
H(14b)	0.0939	-0.3567	-0.5550	8.2775
H(14c)	0.0605	-0.3088	-0.5967	8.2775

Table 6.III.4: Cont.

Atom	*x*	*y*	*z*	*B*eq
H(14d)	-0.2563	-0.5440	-0.2972	16.4400
H(14e)	-0.2717	-0.5861	-0.3694	16.4400
H(14f)	-0.2473	-0.4666	-0.3453	16.4400
H(15a)	0.0110	-0.3679	-0.6589	4.3452
H(15b)	-0.0128	-0.2612	-0.6571	4.3452
H(15c)	-0.1775	-0.5267	-0.3207	6.4446
H(15d)	-0.1783	-0.4396	-0.3783	6.4446
H(16a)	-0.1617	-0.7129	-0.3489	11.1178
H(16b)	-0.0196	-0.5531	-0.6560	5.0488
H(17a)	-0.0633	-0.3490	-0.6731	5.9326
H(17b)	-0.0464	-0.4366	-0.6103	5.9326
H(17c)	-0.0686	-0.5119	-0.6735	5.9326
H(17d)	-0.1265	-0.5520	-0.2878	16.2395
H(17e)	-0.1139	-0.6507	-0.3241	16.2395
H(17f)	-0.1241	-0.4968	-0.3484	16.2395
H(18a)	-0.0479	-0.3739	-0.7609	7.5442
H(18b)	-0.0546	-0.5355	-0.7621	7.5442
H(18c)	-0.0229	-0.4835	-0.7535	7.5442
H(18d)	-0.1620	-0.6041	-0.4529	16.3877
H(18e)	-0.1498	-0.7518	-0.4225	16.3877
H(18f)	-0.1843	-0.7181	-0.4502	16.3877

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 6.III.5: Bond distances (Å) for $[Cd(S_2CN(CH_2CH(CH_3)2)2)2]$

Cd(1) — Cd(1)*	3.524(1)	N(2a) — C(11a)	1.464(9)
Cd(1) — S(1a)	2.565(3)	N(2a) — C(15a)	1.469(8)
Cd(1) — S(2a)	2.592(2)	N(2b) — C(10b)	1.352(9)
Cd(1) — S(3a)	2.592(2)	N(2b) — C(11b)	1.48(1)
Cd(1) — S(4a)	2.781(2)	N(2b) — C(15b)	1.46(1)
Cd(1) — S(4a)*	2.591(2)	C(2a) — C(3a)	1.50(1)
Cd(2) — S(1b)	2.541(2)	C(2b) — C(3b)	1.45(2)
Cd(2) — S(2b)	2.652(3)	C(3a) — C(4a)	1.51(1)
Cd(2) — S(3b)	2.549(3)	C(3a) — C(5a)	1.33(1)
Cd(2) — S(4b)	2.781(3)	C(3b) — C(4b)	1.54(2)
Cd(2) — S(4b)**	2.618(2)	C(3b) — C(5b)	1.25(2)
S(1a) — C(1a)	1.743(8)	C(6b) — C(7b)	1.48(2)
S(1b) — C(1b)	1.715(8)	C(6a) — C(7a)	1.50(1)
S(2a) — C(1a)	1.709(9)	C(7a) — C(8a)	1.55(1)
S(2b) — C(1b)	1.721(8)	C(7a) — C(9a)	1.49(1)
S(3b) — C(10b)	1.696(8)	C(7b) — C(8b)	1.29(2)
S(3a) — C(10a)	1.718(7)	C(7b) — C(9b)	1.51(2)
S(4a) — C(10a)	1.733(7)	C(11b) — C(12b)	1.49(1)
S(4b) — C(10b)	1.746(8)	C(11a) — C(12a)	1.49(1)
N(1a) — C(1a)	1.35(1)	C(12b) — C(13b)	1.51(2)
N(1a) — C(2a)	1.469(8)	C(12b) — C(14b)	1.50(2)
N(1a) — C(6a)	1.44(1)	C(12a) — C(13a)	1.52(1)
N(1b) — C(1b)	1.324(8)	C(12a) — C(14a)	1.52(1)
N(1b) — C(2b)	1.46(1)	C(15b) — C(16b)	1.49(1)
N(1b) — C(6b)	1.45(1)	C(15a) — C(16a)	1.53(1)
N(2a) — C(10a)	1.349(8)	C(16b) — C(17b)	1.54(2)

Table 6.III.5: Cont.

C(16b) — C(18b)	1.36(2)	C(16a) — C(18a)	1.52(1)
C(16a) — C(17a)	1.52(1)		

* symmetry operation: -*x*, -*y*, -1-*z*; ** symmetry operation: -0.5-*x*, -0.5-*y*, -1-*z*

Table 6.III.6: Bond angles (deg.) for $[Cd(S_2CN(CH_2CH(CH_3)_2)_2)_2]$

Cd(1)* — Cd(1) — S(1a)	112.26(6)	S(4b) — C(10b) — N(2b)	120.6(6)
Cd(1)* — Cd(1) — S(2a)	167.72(6)	S(3a) — C(10a) — S(4a)	118.9(4)
Cd(1)* — Cd(1) — S(3a)	78.26(6)	S(3a) — C(10a) — N(2a)	119.8(5)
Cd(1)* — Cd(1) — S(4a)	46.71(4)	S(4a) — C(10a) — N(2a)	121.3(5)
Cd(1)* — Cd(1) — S(4a)*	51.38(5)	N(2b) — C(11b) — C(12b)	117.7(8)
S(1a) — Cd(1) — S(2a)	70.23(7)	Cd(2) — S(1b) — C(1b)	87.7(3)
S(1a) — Cd(1) — S(3a)	151.70(8)	Cd(1) — S(2a) — C(1a)	85.5(3)
S(1a) — Cd(1) — S(4a)	100.23(6)	Cd(2) — S(2b) — C(1b)	84.0(3)
S(1a) — Cd(1) — S(4a)*	108.94(8)	Cd(2) — S(3b) — C(10b)	91.3(3)
S(2a) — Cd(1) — S(3a)	105.31(7)	Cd(1) — S(3a) — C(10a)	89.6(3)
S(2a) — Cd(1) — S(4a)	145.56(7)	Cd(1) — S(4a) — Cd(1)*	81.91(5)
S(2a) — Cd(1) — S(4a)*	116.35(7)	Cd(1) — S(4a) — C(10a)	83.2(2)
S(3a) — Cd(1) — S(4a)	67.07(5)	Cd(1) — S(4a)* — C(10a)*	100.8(3)
S(3a) — Cd(1) — S(4a)*	98.13(9)	Cd(2) — S(4b) — Cd(2)**	89.87(8)
S(4a) — Cd(1) — S(4a)*	98.09(5)	Cd(2) — S(4b) — C(10b)	82.8(3)
S(1b) — Cd(2) — S(2b)	69.40(8)	Cd(2) — S(4b)** — C(10b)**	101.8(2)
S(1b) — Cd(2) — S(3b)	140.92(9)	C(1a) — N(1a) — C(2a)	120.1(6)
S(1b) — Cd(2) — S(4b)	111.33(9)	C(1a) — N(1a) — C(6a)	122.6(6)
S(1b) — Cd(2) — S(4b)**	114.29(8)	C(2a) — N(1a) — C(6a)	117.3(6)
S(2b) — Cd(2) — S(3b)	98.43(8)	C(1b) — N(1b) — C(2b)	120.6(6)
S(2b) — Cd(2) — S(4b)	158.76(8)	C(1b) — N(1b) — C(6b)	123.5(7)
S(2b) — Cd(2) — S(4b)**	109.18(9)	C(2b) — N(1b) — C(6b)	115.9(6)
S(3b) — Cd(2) — S(4b)	67.16(8)	C(10a) — N(2a) — C(11a)	121.7(5)
S(3b) — Cd(2) — S(4b)**	104.79(8)	C(10a) — N(2a) — C(15a)	122.0(6)
S(4b) — Cd(2) — S(4b)**	90.13(8)	C(11a) — N(2a) — C(15a)	116.1(5)
Cd(1) — S(1a) — C(1a)	85.7(3)	C(10b) — N(2b) — C(11b)	121.3(7)

Table 6.III.6: Cont.

S(1a) — C(1a) — S(2a)	118.5(5)	C(10b) — N(2b) — C(15b)	122.9(6)
S(1a) — C(1a) — N(1a)	118.8(6)	C(11b) — N(2b) — C(15b)	115.8(6)
S(2a) — C(1a) — N(1a)	122.7(6)	S(1b) — C(1b) — S(2b)	118.8(4)
N(1a) — C(2a) — C(3a)	115.7(6)	S(1b) — C(1b) — N(1b)	119.7(6)
N(1b) — C(2b) — C(3b)	115.0(9)	S(2b) — C(1b) — N(1b)	121.4(6)
C(2a) — C(3a) — C(4a)	110.4(8)	N(2a) — C(11a) — C(12a)	115.6(6)
C(2a) — C(3a) — C(5a)	122.4(9)	C(11b) — C(12b) — C(13b)	111(1)
C(4a) — C(3a) — C(5a)	119.7(9)	C(11b) — C(12b) — C(14b)	113(1)
C(2b) — C(3b) — C(4b)	109(1)	C(13b) — C(12b) — C(14b)	109(1)
C(2b) — C(3b) — C(5b)	133(1)	C(11a) — C(12a) — C(13a)	107.9(7)
C(4b) — C(3b) — C(5b)	116(1)	C(11a) — C(12a) — C(14a)	113.0(7)
N(1b) — C(6b) — C(7b)	115.3(9)	C(13a) — C(12a) — C(14a)	110.2(7)
N(1a) — C(6a) — C(7a)	113.8(7)	N(2b) — C(15b) — C(16b)	113.1(8)
C(6a) — C(7a) — C(8a)	107.4(9)	N(2a) — C(15a) — C(16a)	114.2(6)
C(6a) — C(7a) — C(9a)	114.4(8)	C(15b) — C(16b) — C(17b)	108(1)
C(8a) — C(7a) — C(9a)	110(1)	C(15b) — C(16b) — C(18b)	121(1)
C(6b) — C(7b) — C(8b)	128(1)	C(17b) — C(16b) — C(18b)	114(1)
C(6b) — C(7b) — C(9b)	110(1)	C(15a) — C(16a) — C(17a)	111.9(6)
C(8b) — C(7b) — C(9b)	120(1)	C(15a) — C(16a) — C(18a)	109.2(7)
S(3b) — C(10b) — S(4b)	118.3(5)	C(17a) — C(16a) — C(18a)	110.5(7)
S(3b) — C(10b) — N(2b)	121.1(6)		

* symmetry operation: -*x*, -*y*, -1-*z*; ** symmetry operation: -0.5-*x*, -0.5-*y*, -1-*z*

Table 6.III.7: Observed and calculated structure factors for $[Cd(S_2CN(CH_2CH(CH_3)_2)_2)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	-8	4779	4810	99	0	4	15	769	784	99	0	8	18	-178	209	99	1	1	3	497	492	99	1	3	4	413	434	99
0	0	4	623	631	99	0	4	16	638	655	99	0	8	19	-245	266	99	1	1	4	2824	2747	99	1	3	5	2309	2310	99
0	0	6	1461	1545	99	0	4	17	1852	1837	99	0	8	20	333	279	99	1	1	5	1583	1581	99	1	3	6	406	438	99
0	0	10	2940	2882	99	0	4	18	176	297	99	0	8	21	205	188	99	1	1	6	1077	1044	99	1	3	7	1218	1294	99
0	0	12	578	627	99	0	4	19	560	593	99	0	10	0	453	364	99	1	1	7	3387	3370	99	1	3	8	1698	1553	99
0	0	14	918	854	99	0	4	20	439	458	99	0	10	1	545	444	99	1	1	8	2287	2318	99	1	3	9	1115	1053	99
0	0	16	290	380	99	0	4	21	606	579	99	0	10	2	274	59	99	1	1	9	2983	2994	99	1	3	10	2249	2255	99
0	0	18	294	47	99	0	4	22	423	390	99	0	10	3	998	913	99	1	1	10	2935	3042	99	1	3	11	502	499	99
0	0	20	570	628	99	0	4	23	-236	241	99	0	10	4	-273	112	99	1	1	11	458	433	99	1	3	12	529	544	99
0	0	22	189	203	99	0	4	24	55	80	99	0	10	5	666	587	99	1	1	12	2442	2447	99	1	3	13	1297	1450	99
0	0	24	407	414	99	0	4	25	191	428	99	0	10	6	170	239	99	1	1	13	2078	2151	99	1	3	14	228	273	99
0	0	26	407	436	99	0	4	26	184	79	99	0	10	7	481	452	99	1	1	14	218	277	99	1	3	15	754	774	99
0	2	0	2763	2886	99	0	6	0	175	228	99	0	10	8	147	45	99	1	1	15	1831	1765	99	1	3	16	1188	1224	99
0	2	1	6914	7230	99	0	6	1	480	489	99	0	10	9	270	221	99	1	1	16	-353	42	99	1	3	17	764	828	99
0	2	2	2039	1994	99	0	6	2	873	851	99	0	10	10	167	170	99	1	1	17	827	893	99	1	3	18	540	610	99
0	2	3	5634	5697	99	0	6	3	364	348	99	0	10	11	133	183	99	1	1	18	523	492	99	1	3	19	182	214	99
0	2	4	2869	2798	99	0	6	4	1258	1224	99	0	10	12	855	660	99	1	1	19	241	235	99	1	3	20	264	251	99
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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34	6	5	169	109	99
34	6	6	112	29	99
34	6	7	-391	48	99
34	6	8	-147	29	99
34	6	9	-218	5	99
34	6	10	396	354	99
34	6	11	198	525	99
34	6	12	378	335	99
34	8	-22	409	444	99
34	8	-21	-135	25	99
34	8	-20	281	301	99
34	8	-19	214	268	99
34	8	-18	-322	42	99
34	8	-17	796	698	99
34	8	-16	501	439	99
34	8	-15	774	883	99
34	8	-14	747	708	99
34	8	-13	540	606	99
34	8	-12	430	385	99
34	8	-11	118	165	99
34	8	-10	439	356	99
34	8	-9	911	961	99
34	8	-8	502	498	99
34	8	-7	906	1090	99
34	8	-6	302	281	99
34	8	-5	324	444	99
34	8	-4	146	108	99
34	8	-3	-263	208	99
34	8	-2	230	265	99
34	8	-1	-90	60	99
34	8	0	53	51	99
34	8	1	322	286	99
34	8	2	243	169	99
34	8	3	86	52	99
34	8	4	168	211	99
34	8	5	264	129	99
34	8	6	302	62	99
34	8	7	259	139	99
34	8	8	-160	150	99
34	10	-15	184	276	99
34	10	-14	-326	228	99
34	10	-13	-163	89	99
34	10	-12	273	402	99
34	10	-11	-162	59	99
34	10	-10	-166	418	99
34	10	-9	80	188	99
34	10	-8	219	48	99
34	10	-7	589	582	99
34	10	-6	-142	284	99
34	10	-5	415	515	99
34	10	-4	222	196	99
34	10	-3	186	12	99
34	10	-2	-101	182	99
34	10	-1	174	113	99
34	10	0	-191	153	99
35	1	-28	149	278	99
35	1	-27	487	470	99
35	1	-26	-150	19	99
35	1	-25	409	333	99
35	1	-24	465	381	99
35	1	-23	-277	244	99
35	1	-22	1010	1020	99
35	1	-21	573	442	99
35	1	-20	613	676	99
35	1	-19	793	812	99
35	1	-18	352	592	99
35	1	-17	470	588	99
35	1	-16	-21	68	99
35	1	-15	-309	32	99
35	1	-14	1257	1202	99
35	1	-13	1149	1038	99
35	1	-12	1554	1581	99
35	1	-11	1470	1550	99
35	1	-10	1028	960	99
35	1	-9	299	31	99
35	1	-8	1410	1335	99
35	1	-7	827	904	99
35	1	-6	417	479	99
35	1	-5	512	454	99
35	1	-4	975	972	99
35	1	-3	516	345	99
35	1	-2	-440	361	99
35	1	-1	1216	1073	99
35	1	0	-360	239	99
35	1	1	1315	1295	99
35	1	2	1095	932	99
35	1	3	-197	254	99
35	1	4	491	624	99
35	1	5	485	621	99
35	1	6	202	266	99
35	1	7	924	936	99
35	1	8	445	543	99
35	1	9	454	418	99
35	1	10	901	823	99
35	1	11	251	300	99
35	1	12	781	666	99
35	1	13	460	449	99
35	1	14	393	333	99
35	1	15	389	511	99
35	3	-28	-459	135	99
35	3	-27	754	740	99
35	3	-26	362	289	99
35	3	-25	929	1036	99
35	3	-24	151	255	99
35	3	-23	430	536	99
35	3	-22	454	409	99
35	3	-21	85	482	99
35	3	-20	412	541	99
35	3	-19	632	767	99
35	3	-18	176	140	99
35	3	-17	987	1062	99
35	3	-16	126	95	99
35	3	-15	928	1027	99
35	3	-14	442	591	99
35	3	-13	568	477	99
35	3	-12	-70	138	99
35	3	-11	701	769	99
35	3	-10	778	623	99
35	3	-9	1308	1273	99
35	3	-8	540	453	99
35	3	-7	1965	1918	99
35	3	-6	400	268	99
35	3	-5	1494	1533	99
35	3	-4	347	45	99
35	3	-3	1169	1145	99
35	3	-2	1068	1071	99
35	3	-1	808	807	99
35	3	0	485	507	99
35	3	1	890	960	99
35	3	2	140	89	99
35	3	3	328	45	99
35	3	4	-183	327	99
35	3	5	907	978	99
35	3	6	294	420	99
35	3	7	619	581	99
35	3	8	191	7	99
35	3	9	654	527	99
35	3	10	255	246	99
35	3	11	-199	44	99
35	3	12	195	38	99
35	3	13	264	534	99
35	3	14	-74	88	99
35	5	-27	-264	68	99
35	5	-26	-282	83	99
35	5	-25	-231	66	99
35	5	-24	603	647	99
35	5	-23	308	142	99
35	5	-22	633	661	99
35	5	-21	-229	81	99
35	5	-20	346	319	99
35	5	-19	382	352	99
35	5	-18	233	326	99
35	5	-17	-96	44	99
35	5	-16	134	143	99
35	5	-15	893	867	99
35	5	-14	424	337	99
35	5	-13	963	970	99
35	5	-12	1419	1457	99
35	5	-11	463	543	99
35	5	-10	1575	1635	99
35	5	-9	704	640	99
35	5	-8	525	566	99
35	5	-7	1322	1247	99
35	5	-6	165	101	99
35	5	-5	949	995	99
35	5	-4	406	410	99
35	5	-3	276	397	99
35	5	-2	905	945	99
35	5	-1	672	546	99
35	5	0	154	361	99
35	5	1	787	804	99
35	5	2	-310	19	99
35	5	3	210	465	99
35	5	4	257	112	99
35	5	5	26	213	99
35	5	6	423	464	99
35	5	7	712	569	99
35	5	8	269	87	99
35	5	9	575	508	99
35	5	10	157	181	99
35	5	11	256	36	99
35	5	12	334	150	99
35	5	13	281	95	99
35	7	-24	255	280	99
35	7	-23	90	13	99
35	7	-22	459	404	99
35	7	-21	-121	240	99
35	7	-20	342	354	99
35	7	-19	113	258	99
35	7	-18	356	294	99
35	7	-17	-345	109	99
35	7	-16	-428	247	99
35	7	-15	340	307	99
35	7	-14	354	394	99
35	7	-13	451	476	99
35	7	-12	776	848	99
35	7	-11	-141	75	99
35	7	-10	985	1046	99
35	7	-9	641	576	99
35	7	-8	915	977	99
35	7	-7	225	414	99
35	7	-6	117	412	99
35	7	-5	143	269	99
35	7	-4	508	347	99
35	7	-3	281	300	99
35	7	-2	518	507	99
35	7	-1	-313	174	99
35	7	0	468	543	99
35	7	1	132	405	99
35	7	2	316	414	99
35	7	3	-185	181	99
35	7	4	-201	62	99
35	7	5	237	196	99
35	7	6	-226	54	99
35	7	7	-228	30	99
35	7	8	220	95	99
35	7	9	-30	9	99
35	7	10	236	72	99
35	9	-19	-352	159	99
35	9	-18	112	225	99
35	9	-17	-304	420	99
35	9	-16	399	401	99
35	9	-15	826	813	99
35	9	-14	-43	276	99
35	9	-13	683	686	99
35	9	-12	224	210	99
35	9	-11	319	264	99
35	9	-10	519	411	99
35	9	-9	136	293	99
35	9	-8	239	287	99
35	9	-7	713	829	99
35	9	-6	85	7	99
35	9	-5	778	943	99
35	9	-4	310	204	99
35	9	-3	179	102	99
35	9	-2	326	458	99
35	9	-1	319	266	99
35	9	0	244	309	99
35	9	1	431	408	99
35	9	2	146	105	99
35	9	3	492	496	99
35	9	4	238	193	99

APPENDIX 6.IV

$[Cd(S_2CN(Et)Cy)_2]$

Table 6.IV.1: Crystallographic Parameters for $[Cd(S_2CN(CH_2CH_3)(CH(CH_2)_5))_2]$

Formula	$C_{18}H_{22}CdN_2S_4$
Formula weight	517.11
Crystal morphology / colour	block / colourless
Crystal dimensions (mm)	0.10 x 0.10 x 0.40
Crystal system	monoclinic
Space group	$P2_1/c$ (#14)
a (Å)	8.541(4)
b (Å)	19.688(8)
c (Å)	13.847(3)
α (°)	90
β (°)	94.29(3)
γ (°)	90
V (Å^3)	2322(1)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.479
$F(000)$	1064
Temperature (°K)	200
μ (cm^{-1})	13.06
θ limits, cell (°)	16.9 – 19.8
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 11, 0 – 25, -18 – 18
Range of transmission factors	0.932 – 1
Scan technique	ω:2θ
No. of data measured	5904
No. of unique data	5547
R_{amal}	0.102
No. of parameters	226
R (I≥0σ(I))	0.124
R_w (I≥0σ(I))	0.121
R (I≥3σ(I))	0.055
R_w (I≥3σ(I))	0.053
Maximum and minimum residual electron density (e Å^{-3})	1.15, -1.41

Table 6.IV.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Cd(S_2CN(CH_2CH_3)(CH(CH_2)5))2]$

Atom	*x*	*y*	*z*
Cd	0.59259(6)	0.05051(3)	-0.09557(4)
S(1)	0.5342(2)	0.17586(9)	-0.0695(1)
S(2)	0.6144(2)	0.11815(9)	-0.2574(1)
S(3)	0.8036(2)	-0.04051(9)	-0.0675(1)
S(4)	0.6625(2)	0.02220(8)	0.1019(1)
N(1)	0.5501(6)	0.2488(3)	-0.2307(4)
N(2)	0.8661(6)	-0.0801(3)	0.1129(4)
C(1)	0.5667(7)	0.1878(3)	-0.1909(4)
C(2)	0.5884(9)	0.2599(4)	-0.3319(5)
C(3)	0.760(1)	0.2728(5)	-0.3412(6)
C(4)	0.4962(8)	0.3080(3)	-0.1742(5)
C(5)	0.368(1)	0.3480(5)	-0.2281(7)
C(6)	0.309(2)	0.4024(6)	-0.1623(7)
C(7)	0.438(2)	0.4492(5)	-0.1232(9)
C(8)	0.568(2)	0.4084(6)	-0.0735(10)
C(9)	0.630(1)	0.3534(5)	-0.1388(7)
C(10)	0.7846(7)	-0.0388(3)	0.0561(4)
C(11)	0.9847(7)	-0.1278(4)	0.0760(5)
C(12)	0.9170(8)	-0.1934(3)	0.0360(6)
C(13)	0.8530(7)	-0.0815(3)	0.2202(5)
C(14)	0.9801(8)	-0.0370(3)	0.2692(5)
C(15)	0.9680(9)	-0.0347(4)	0.3796(5)
C(16)	0.9659(9)	-0.1064(4)	0.4207(5)
C(17)	0.8348(9)	-0.1482(4)	0.3709(6)
C(18)	0.8498(8)	-0.1508(4)	0.2631(5)

Table 6.IV.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Cd(S_2CN(CH_2CH_3)(CH(CH_2)_5))_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cd	0.0377(3)	0.0390(3)	0.0410(3)	0.0006(3)	0.0019(2)	0.0069(3)
S(1)	0.066(1)	0.041(1)	0.0299(9)	0.0018(9)	0.0048(9)	0.0045(8)
S(2)	0.053(1)	0.047(1)	0.0359(10)	0.0101(9)	0.0082(9)	0.0022(8)
S(3)	0.043(1)	0.049(1)	0.0329(9)	0.0065(8)	0.0040(8)	0.0012(9)
S(4)	0.0374(10)	0.0324(8)	0.0354(9)	0.0013(7)	0.0004(7)	0.0001(7)
N(1)	0.045(4)	0.040(3)	0.036(3)	0.008(3)	0.006(3)	0.010(3)
N(2)	0.028(3)	0.034(3)	0.035(3)	0.000(2)	0.002(2)	-0.010(2)
C(1)	0.030(4)	0.047(4)	0.032(4)	0.002(3)	0.004(3)	0.001(3)
C(2)	0.070(6)	0.071(6)	0.036(4)	0.024(4)	0.021(4)	0.026(4)
C(3)	0.076(6)	0.087(7)	0.077(6)	0.018(5)	0.044(5)	0.032(5)
C(4)	0.058(5)	0.036(4)	0.042(4)	0.002(3)	0.011(4)	0.017(3)
C(5)	0.122(9)	0.093(7)	0.075(7)	0.066(7)	-0.017(6)	-0.020(6)
C(6)	0.20(1)	0.115(10)	0.076(7)	0.124(10)	-0.028(8)	-0.040(7)
C(7)	0.30(2)	0.050(6)	0.091(9)	0.031(10)	0.08(1)	-0.010(7)
C(8)	0.14(1)	0.099(9)	0.15(1)	-0.051(8)	0.047(9)	-0.064(9)
C(9)	0.094(8)	0.090(7)	0.093(7)	-0.022(6)	0.022(6)	-0.046(6)
C(10)	0.022(3)	0.031(4)	0.040(4)	0.002(3)	-0.005(3)	-0.008(3)
C(11)	0.030(4)	0.061(5)	0.046(4)	0.006(3)	0.004(3)	-0.004(4)
C(12)	0.048(5)	0.041(4)	0.069(5)	0.006(3)	0.006(4)	-0.009(4)
C(13)	0.027(4)	0.040(4)	0.038(4)	0.004(3)	-0.001(3)	0.007(3)
C(14)	0.046(4)	0.049(5)	0.041(4)	0.005(3)	0.004(3)	-0.007(3)
C(15)	0.060(5)	0.063(6)	0.041(4)	-0.002(4)	0.003(4)	-0.009(4)
C(16)	0.063(6)	0.080(6)	0.036(4)	0.011(5)	0.003(4)	-0.001(4)
C(17)	0.060(6)	0.075(6)	0.059(5)	0.007(5)	0.006(4)	0.037(5)
C(18)	0.054(5)	0.045(4)	0.048(5)	-0.001(4)	-0.003(4)	0.016(4)

Table 6.IV.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Cd(S_2CN(CH_2CH_3)(CH(CH_2)5))2]^*$

Atom	*x*	*y*	*z*	*B*eq
H(2a)	0.5577	0.2198	-0.3695	5.5204
H(2b)	0.5295	0.2988	-0.3576	5.5204
H(3a)	0.7920	0.3134	-0.3051	7.3958
H(3b)	0.8207	0.2343	-0.3156	7.3958
H(3c)	0.7785	0.2791	-0.4089	7.3958
H(4a)	0.4523	0.2896	-0.1171	4.2883
H(5a)	0.2820	0.3177	-0.2487	9.2929
H(5b)	0.4082	0.3692	-0.2843	9.2929
H(6a)	0.2628	0.3806	-0.1083	12.4746
H(6b)	0.2291	0.4291	-0.1987	12.4746
H(7a)	0.3975	0.4806	-0.0774	13.4578
H(7b)	0.4785	0.4744	-0.1763	13.4578
H(8a)	0.6540	0.4387	-0.0532	11.9940
H(8b)	0.5294	0.3867	-0.0171	11.9940
H(9a)	0.6754	0.3745	-0.1937	8.6754
H(9b)	0.7101	0.3268	-0.1025	8.6754
H(11a)	1.0614	-0.1384	0.1291	4.3105
H(11b)	1.0362	-0.1050	0.0250	4.3105
H(12a)	0.8675	-0.2176	0.0866	4.9884
H(12b)	0.8395	-0.1838	-0.0170	4.9884
H(12c)	1.0003	-0.2211	0.0128	4.9884
H(13a)	0.7535	-0.0604	0.2316	3.3202
H(14a)	1.0820	-0.0551	0.2560	4.2576
H(14b)	0.9692	0.0087	0.2434	4.2576

Table 6.IV.4: Cont.

Atom	x	y	z	B_{eq}
H(15a)	0.8721	-0.0115	0.3933	5.2019
H(15b)	1.0576	-0.0103	0.4095	5.2019
H(16a)	1.0656	-0.1282	0.4116	5.6696
H(16b)	0.9508	-0.1039	0.4894	5.6696
H(17a)	0.8398	-0.1941	0.3967	6.1418
H(17b)	0.7347	-0.1280	0.3831	6.1418
H(18a)	0.7610	-0.1756	0.2328	4.6793
H(18b)	0.9463	-0.1742	0.2509	4.6793

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 6.IV.5: Bond distances (Å) for [Cd($S_2CN(CH_2CH_3)(CH(CH_2)5)2$))2]

Cd — S(1)	2.549(2)	C(2) — C(3)	1.50(1)
Cd — S(2)	2.625(2)	C(4) — C(5)	1.50(1)
Cd — S(3)	2.551(2)	C(4) — C(9)	1.51(1)
Cd — S(4)	2.811(2)	C(5) — C(6)	1.52(1)
Cd — S(4)*	2.603(2)	C(6) — C(7)	1.51(2)
S(1) — C(1)	1.741(6)	C(7) — C(8)	1.50(2)
S(2) — C(1)	1.718(6)	C(8) — C(9)	1.53(1)
S(3) — C(10)	1.732(6)	C(11) — C(12)	1.504(9)
S(4) — C(10)	1.741(6)	C(13) — C(14)	1.516(9)
N(1) — C(1)	1.325(7)	C(13) — C(18)	1.490(9)
N(1) — C(2)	1.479(8)	C(14) — C(15)	1.540(9)
N(1) — C(4)	1.495(8)	C(15) — C(16)	1.52(1)
N(2) — C(10)	1.296(7)	C(16) — C(17)	1.51(1)
N(2) — C(11)	1.498(8)	C(17) — C(18)	1.51(1)
N(2) — C(13)	1.499(7)		

* symmetry operation: 1-*x*, -*y*, -*z*

Table 6.IV.6: Bond angles (deg.) for [Cd($S_2CN(CH_2CH_3)(CH(CH_2)_5)$)2]

S(1) — Cd — S(2)	69.94(6)	S(1) — C(1) — N(1)	120.3(5)
S(1) — Cd — S(3)	143.35(6)	S(2) — C(1) — N(1)	121.5(5)
S(1) — Cd — S(4)	94.80(5)	N(1) — C(2) — C(3)	113.1(7)
S(1) — Cd — S(4)*	111.41(6)	N(1) — C(4) — C(5)	113.0(6)
S(2) — Cd — S(3)	113.08(6)	N(1) — C(4) — C(9)	112.1(6)
S(2) — Cd — S(4)	155.17(6)	C(5) — C(4) — C(9)	111.1(7)
S(2) — Cd — S(4)*	111.27(6)	C(4) — C(5) — C(6)	109.6(7)
S(3) — Cd — S(4)	67.43(5)	C(5) — C(6) — C(7)	112(1)
S(3) — Cd — S(4)*	101.46(6)	C(6) — C(7) — C(8)	109.6(9)
S(4) — Cd — S(4)*	92.29(5)	C(7) — C(8) — C(9)	113(1)
Cd — S(1) — C(1)	86.9(2)	C(4) — C(9) — C(8)	108.8(8)
Cd — S(2) — C(1)	85.0(2)	S(3) — C(10) — S(4)	118.4(3)
Cd — S(3) — C(10)	91.1(2)	S(3) — C(10) — N(2)	120.1(4)
Cd — S(4) — Cd*	87.71(5)	S(4) — C(10) — N(2)	121.4(5)
Cd — S(4) — C(10)	82.6(2)	N(2) — C(11) — C(12)	114.4(5)
Cd — S(4) * — C(10)*	97.5(2)	N(2) — C(13) — C(14)	109.3(5)
C(1) — N(1) — C(2)	120.2(6)	N(2) — C(13) — C(18)	114.7(6)
C(1) — N(1) — C(4)	121.1(5)	C(14) — C(13) — C(18)	112.6(5)
C(2) — N(1) — C(4)	118.6(5)	C(13) — C(14) — C(15)	111.3(6)
C(10) — N(2) — C(11)	122.2(5)	C(14) — C(15) — C(16)	110.3(6)
C(10) — N(2) — C(13)	122.4(5)	C(15) — C(16) — C(17)	111.3(6)
C(11) — N(2) — C(13)	115.3(5)	C(16) — C(17) — C(18)	110.8(6)
S(1) — C(1) — S(2)	118.1(4)	C(13) — C(18) — C(17)	111.7(6)

* symmetry operation: 1-*x*, -*y*, -*z*

Table 6.IV.7: Observed and calculated structure factors for $[Cd(S_2CN(CH_2CH_3)(CH(CH_2)_5))_2]$

h	k	l	Fo	Fc	sigF2
0	0	4	1670	1635	99
0	0	6	2825	2829	99
0	0	8	-131	66	99
0	0	10	440	459	99
0	0	12	655	722	99
0	0	14	-67	186	99
0	0	16	113	75	99
0	0	18	83	29	99
0	1	2	1180	1149	99
0	1	3	393	359	99
0	1	4	671	682	99
0	1	5	1898	1932	99
0	1	6	329	302	99
0	1	7	1132	1157	99
0	1	8	432	414	99
0	1	9	304	292	99
0	1	10	195	210	99
0	1	11	797	858	99
0	1	12	108	83	99
0	1	13	-52	12	99
0	1	14	67	10	99
0	1	15	102	157	99
0	1	16	-82	76	99
0	1	17	105	158	99
0	1	18	44	16	99
0	2	2	1254	1245	99
0	2	3	749	731	99
0	2	4	1442	1448	99
0	2	5	1231	1200	99
0	2	6	1737	1739	99
0	2	7	1138	1121	99
0	2	8	289	269	99
0	2	9	234	255	99
0	2	10	841	816	99
0	2	11	171	91	99
0	2	12	201	190	99
0	2	13	273	303	99
0	2	14	235	232	99
0	2	15	102	23	99
0	2	16	237	236	99
0	2	17	56	62	99
0	3	1	1064	1073	99
0	3	2	1504	1549	99
0	3	3	596	602	99
0	3	4	297	322	99
0	3	5	1094	1097	99
0	3	6	781	774	99
0	3	7	293	289	99
0	3	8	382	356	99
0	3	9	614	605	99
0	3	10	194	189	99
0	3	11	317	316	99
0	3	12	497	494	99
0	3	13	79	9	99
0	3	14	209	222	99
0	3	15	392	386	99
0	3	16	47	13	99
0	3	17	-65	50	99
0	4	0	131	153	99
0	4	1	826	849	99
0	4	2	665	641	99
0	4	3	173	199	99
0	4	4	874	910	99
0	4	5	344	327	99
0	4	6	-14	39	99
0	4	7	1028	1006	99
0	4	8	185	188	99
0	4	9	227	228	99
0	4	10	484	492	99
0	4	11	472	454	99
0	4	12	48	49	99
0	4	13	599	556	99
0	4	14	97	128	99
0	4	15	117	92	99
0	4	16	198	185	99
0	4	17	244	315	99
0	5	1	1153	1156	99
0	5	2	506	490	99
0	5	3	125	117	99
0	5	4	370	378	99
0	5	5	201	225	99
0	5	6	548	561	99
0	5	7	422	424	99
0	5	8	864	858	99
0	5	9	204	176	99
0	5	10	343	314	99
0	5	11	12	85	99
0	5	12	495	490	99
0	5	13	188	121	99
0	5	14	353	349	99
0	5	15	92	10	99
0	5	16	155	165	99
0	5	17	75	69	99
0	6	0	106	82	99
0	6	1	99	109	99
0	6	2	217	232	99
0	6	3	871	907	99
0	6	4	72	78	99
0	6	5	6	84	99
0	6	6	-117	30	99
0	6	7	357	359	99
0	6	8	223	181	99
0	6	9	453	441	99
0	6	10	115	139	99
0	6	11	452	428	99
0	6	12	-64	110	99
0	6	13	250	203	99
0	6	14	-26	96	99
0	6	15	-73	159	99
0	6	16	162	181	99
0	6	17	115	217	99
0	7	1	893	898	99
0	7	2	892	915	99
0	7	3	411	404	99
0	7	4	456	461	99
0	7	5	358	373	99
0	7	6	91	82	99
0	7	7	319	301	99
0	7	8	360	360	99
0	7	9	345	328	99
0	7	10	335	314	99
0	7	11	37	75	99
0	7	12	211	224	99
0	7	13	-27	107	99
0	7	14	272	220	99
0	7	15	189	156	99
0	7	16	71	9	99
0	7	17	-114	33	99
0	8	0	1118	1157	99
0	8	1	314	301	99
0	8	2	1004	1006	99
0	8	3	371	371	99
0	8	4	905	908	99
0	8	5	162	155	99
0	8	6	552	591	99
0	8	7	-77	90	99
0	8	8	543	554	99
0	8	9	175	173	99
0	8	10	588	575	99
0	8	11	-171	8	99
0	8	12	-92	107	99
0	8	13	186	157	99
0	8	14	-74	64	99
0	8	15	118	39	99
0	8	16	88	101	99
0	8	17	-167	51	99
0	9	1	1317	1354	99
0	9	2	835	793	99
0	9	3	147	123	99
0	9	4	7	86	99
0	9	5	1027	1075	99
0	9	6	196	166	99
0	9	7	977	940	99
0	9	8	65	86	99
0	9	9	60	118	99
0	9	10	98	88	99
0	9	11	449	451	99
0	9	12	104	75	99
0	9	13	330	258	99
0	9	14	-159	47	99
0	9	15	-69	83	99
0	9	16	-152	50	99
0	10	0	1391	1416	99
0	10	1	225	210	99
0	10	2	277	315	99
0	10	3	800	812	99
0	10	4	782	804	99
0	10	5	70	3	99
0	10	6	999	998	99
0	10	7	154	86	99
0	10	8	141	117	99
0	10	9	511	469	99
0	10	10	412	384	99
0	10	11	78	5	99
0	10	12	252	221	99
0	10	13	-157	69	99
0	10	14	178	8	99
0	10	15	-202	114	99
0	10	16	161	188	99
0	11	1	770	789	99
0	11	2	830	846	99
0	11	3	-61	60	99
0	11	4	522	531	99
0	11	5	837	855	99
0	11	6	181	173	99
0	11	7	373	332	99
0	11	8	845	816	99
0	11	9	81	26	99
0	11	10	87	31	99
0	11	11	577	557	99
0	11	12	-117	49	99
0	11	13	-156	60	99
0	11	14	291	233	99
0	11	15	149	223	99
0	11	16	136	61	99
0	12	0	1026	1053	99
0	12	1	228	243	99
0	12	2	366	335	99
0	12	3	690	696	99
0	12	4	415	412	99
0	12	5	132	118	99
0	12	6	715	707	99
0	12	7	538	558	99
0	12	8	-190	83	99
0	12	9	380	361	99
0	12	10	233	235	99
0	12	11	282	238	99
0	12	12	237	213	99
0	12	13	340	325	99
0	12	14	195	209	99
0	12	15	172	94	99
0	13	1	710	688	99
0	13	2	710	719	99
0	13	3	135	163	99
0	13	4	451	454	99
0	13	5	596	588	99
0	13	6	592	579	99
0	13	7	178	362	99
0	13	8	525	504	99
0	13	9	-238	31	99
0	13	10	-97	45	99
0	13	11	239	215	99
0	13	12	506	461	99
0	13	13	88	40	99
0	13	14	246	252	99
0	13	15	97	60	99
0	14	0	965	974	99
0	14	1	672	654	99
0	14	2	206	241	99
0	14	3	704	708	99
0	14	4	56	99	99
0	14	5	224	226	99
0	14	6	552	539	99
0	14	7	663	651	99
0	14	8	193	202	99
0	14	9	354	345	99
0	14	10	-156	55	99
0	14	11	97	140	99
0	14	12	-84	178	99
0	14	13	531	454	99
0	14	14	74	28	99
0	14	15	-70	38	99
0	15	1	119	234	99
0	15	2	905	894	99
0	15	3	-178	14	99
0	15	4	558	570	99
0	15	5	194	160	99
0	15	6	308	308	99
0	15	7	64	115	99
0	15	8	526	505	99
0	15	9	-159	120	99
0	15	10	221	188	99
0	15	11	131	47	99
0	15	12	381	345	99
0	15	13	-24	6	99
0	15	14	56	173	99
0	16	0	203	193	99
0	16	1	491	497	99
0	16	2	35	81	99
0	16	3	609	592	99
0	16	4	84	115	99
0	16	5	263	238	99
0	16	6	62	128	99
0	16	7	594	576	99
0	16	8	60	53	99
0	16	9	351	338	99
0	16	10	-58	11	99
0	16	11	-181	33	99
0	16	12	121	188	99
0	16	13	306	336	99
0	16	14	47	34	99
0	17	1	302	312	99
0	17	2	318	335	99
0	17	3	-127	57	99
0	17	4	313	305	99
0	17	5	239	246	99
0	17	6	417	409	99
0	17	7	194	189	99
0	17	8	319	313	99
0	17	9	-190	54	99
0	17	10	-108	17	99
0	17	11	83	191	99
0	17	12	280	291	99
0	17	13	126	118	99
0	18	0	240	211	99
0	18	1	-5	53	99
0	18	2	186	159	99
0	18	3	74	187	99
0	18	4	-32	142	99
0	18	5	282	274	99
0	18	6	351	354	99
0	18	7	194	235	99
0	18	8	-18	31	99
0	18	9	-278	43	99
0	18	10	-36	99	99
0	18	11	310	283	99
0	18	12	203	236	99
0	19	1	130	94	99
0	19	2	26	98	99
0	19	3	-71	5	99
0	19	4	64	152	99
0	19	5	231	245	99
0	19	6	96	107	99
0	19	7	66	70	99
0	19	8	-58	56	99
0	19	9	123	84	99
0	19	10	194	161	99
0	19	11	-70	212	99
0	19	12	203	181	99
0	20	0	224	231	99
0	20	1	-39	27	99
0	20	2	132	85	99
0	20	3	98	33	99
0	20	4	182	197	99
0	20	5	-63	89	99
0	20	6	66	105	99
0	20	7	-64	27	99
0	20	8	-126	16	99
0	20	9	-139	32	99
0	20	10	91	163	99
0	20	11	-111	220	99
0	21	1	222	190	99
0	21	2	-143	31	99
0	21	3	61	9	99
0	21	4	112	22	99
0	21	5	129	146	99
0	21	6	36	17	99
0	21	7	80	132	99
0	21	8	40	66	99
0	21	9	-47	60	99
0	21	10	94	23	99
0	22	0	262	245	99
0	22	1	62	28	99
0	22	2	192	223	99
0	22	3	84	187	99
0	22	4	73	217	99
0	22	5	-86	22	99
0	22	6	-79	114	99
0	22	7	71	65	99
0	22	8	85	167	99
0	22	9	-136	136	99
0	23	1	200	203	99
0	23	2	253	280	99
0	23	3	58	11	99
0	23	4	155	181	99
0	23	5	106	126	99
0	23	6	-105	61	99
0	23	7	24	163	99
0	23	8	-71	156	99
0	24	0	105	105	99
0	24	1	-161	98	99
0	24	2	44	4	99
0	24	3	313	373	99
0	24	4	83	40	99
0	24	5	81	15	99
0	24	6	58	125	99
0	25	1	61	37	99
0	25	2	260	288	99
0	25	3	-132	27	99
0	25	4	184	192	99
1	0	-18	-39	76	99
1	0	-16	376	357	99
1	0	-14	274	299	99
1	0	-12	586	573	99
1	0	-10	915	894	99
1	0	-8	403	396	99
1	0	-6	811	880	99
1	0	-4	2090	2091	99
1	0	-2	671	711	99
1	0	2	122	136	99
1	0	4	-52	67	99
1	0	6	1070	1071	99
1	0	8	481	445	99
1	0	10	140	109	99
1	0	12	483	517	99
1	0	14	212	256	99
1	0	16	52	26	99
1	1	-18	123	21	99
1	1	-17	256	274	99
1	1	-16	-128	49	99
1	1	-15	366	335	99
1	1	-14	-105	76	99
1	1	-13	222	166	99
1	1	-12	149	149	99
1	1	-11	995	978	99
1	1	-10	224	214	99
1	1	-9	930	894	99
1	1	-8	485	466	99
1	1	-7	313	296	99
1	1	-6	65	32	99
1	1	-5	1749	1794	99
1	1	-4	72	71	99
1	1	-3	636	640	99
1	1	-2	1051	1044	99
1	1	1	1306	1331	99
1	1	2	1293	1322	99
1	1	3	69	23	99
1	1	4	574	577	99
1	1	5	888	891	99
1	1	6	154	173	99
1	1	7	694	659	99
1	1	8	434	443	99
1	1	9	204	220	99
1	1	10	316	352	99
1	1	11	324	294	99
1	1	12	177	234	99
1	1	13	145	106	99
1	1	14	83	142	99
1	1	15	144	158	99
1	1	16	83	109	99
1	1	17	110	60	99
1	2	-18	71	61	99
1	2	-17	-140	49	99
1	2	-16	437	397	99
1	2	-15	92	109	99
1	2	-14	420	424	99
1	2	-13	167	164	99
1	2	-12	169	169	99
1	2	-11	144	116	99
1	2	-10	1009	1005	99
1	2	-9	143	169	99
1	2	-8	454	471	99
1	2	-7	326	323	99
1	2	-6	585	594	99
1	2	-5	65	50	99
1	2	-4	1327	1337	99
1	2	-3	371	364	99
1	2	-2	481	478	99
1	2	-1	45	54	99
1	2	0	79	76	99
1	2	1	560	607	99
1	2	2	1354	1437	99
1	2	3	708	701	99
1	2	4	433	431	99
1	2	5	855	887	99
1	2	6	532	512	99
1	2	7	431	429	99
1	2	8	218	191	99
1	2	9	551	566	99
1	2	10	311	315	99
1	2	11	514	514	99
1	2	12	102	127	99
1	2	13	269	261	99
1	2	14	182	200	99
1	2	15	119	140	99
1	2	16	209	223	99
1	2	17	73	171	99
1	3	-17	92	116	99
1	3	-16	-44	128	99
1	3	-15	574	545	99
1	3	-14	19	85	99
1	3	-13	116	74	99
1	3	-12	506	496	99
1	3	-11	341	306	99
1	3	-10	83	115	99
1	3	-9	864	836	99
1	3	-8	-8	54	99
1	3	-7	128	79	99
1	3	-6	1064	1089	99
1	3	-5	404	425	99
1	3	-4	792	786	99
1	3	-3	1139	1115	99
1	3	-2	1628	1629	99
1	3	-1	475	491	99
1	3	0	312	336	99
1	3	1	761	817	99
1	3	2	1001	1018	99
1	3	3	233	243	99
1	3	4	982	1009	99
1	3	5	567	600	99
1	3	6	60	56	99
1	3	7	64	87	99
1	3	8	731	745	99
1	3	9	468	485	99
1	3	10	665	651	99
1	3	11	188	172	99
1	3	12	69	94	99
1	3	13	123	162	99
1	3	14	480	458	99
1	3	15	299	323	99
1	3	16	-91	209	99
1	3	17	91	61	99
1	4	-17	372	365	99
1	4	-16	240	269	99
1	4	-15	223	227	99
1	4	-14	133	131	99
1	4	-13	337	310	99
1	4	-12	177	163	99
1	4	-11	567	572	99
1	4	-10	494	478	99
1	4	-9	230	258	99
1	4	-8	170	178	99
1	4	-7	268	279	99
1	4	-6	375	386	99
1	4	-5	706	721	99
1	4	-4	854	824	99
1	4	-3	302	345	99
1	4	-2	607	666	99
1	4	-1	363	424	99
1	4	0	415	450	99
1	4	1	39	2	99
1	4	2	90	61	99
1	4	3	623	660	99
1	4	4	715	712	99
1	4	5	669	649	99
1	4	6	-49	129	99
1	4	7	655	663	99
1	4	8	233	210	99
1	4	9	706	714	99
1	4	10	455	456	99
1	4	11	274	295	99
1	4	12	39	129	99
1	4	13	584	583	99
1	4	14	144	33	99
1	4	15	443	405	99
1	4	16	146	179	99
1	4	17	-104	18	99
1	5	-17	150	45	99
1	5	-16	434	416	99
1	5	-15	115	57	99
1	5	-14	91	20	99
1	5	-13	317	296	99
1	5	-12	434	426	99
1	5	-11	-205	71	99
1	5	-10	656	641	99
1	5	-9	330	329	99
1	5	-8	459	445	99
1	5	-7	508	508	99
1	5	-6	618	630	99
1	5	-5	323	301	99
1	5	-4	-68	40	99
1	5	-3	366	383	99
1	5	-2	692	808	99
1	5	-1	789	892	99
1	5	0	560	614	99
1	5	1	106	104	99
1	5	2	967	1027	99
1	5	3	255	260	99
1	5	4	1296	1341	99
1	5	5	224	218	99
1	5	6	-110	74	99
1	5	7	111	89	99
1	5	8	1160	1171	99
1	5	9	75	5	99
1	5	10	549	545	99
1	5	11	47	53	99
1	5	12	153	127	99
1	5	13	109	167	99
1	5	14	586	556	99
1	5	15	-124	53	99

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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11	3	-1	161	127	99
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APPENDIX 7.I

$[Hg(S_2COnBu)_2]$

Table 7.I.1: Crystallographic Parameters for $[Hg(S_2COCH_2CH_2CH_2CH_3)_2]$

Formula	$C_{10}H_{18}HgS_4O_2$
Formula weight	499.08
Crystal morphology / colour	block / pale yellow
Crystal dimensions (mm)	0.05 x 0.21 x 0.45
Crystal system	orthorhombic
Space group	*Pbcn* (# 60)
a (Å)	7.418(5)
b (Å)	25.66(5)
c (Å)	8.61(3)
α (°)	90
β (°)	90
γ (°)	90
V (Å^3)	1639(4)
Z	4
$\rho_{calc.}$ (g cm^{-3})	2.022
$F(000)$	952
Temperature (°K)	293
μ (cm^{-1})	99.10
θ limits, cell (°)	19.8 – 23.0
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 9, 0 – 28, -11 – 0
Range of transmission factors	0.925 – 1
Scan technique	ω:2θ
No. of data measured	2227
No. of unique data	1910
No. of parameters	75
R (I≥0σ(I))	0.144
R_w (I≥0σ(I))	0.163
R (I≥3σ(I))	0.066
R_w (I≥3σ(I))	0.074
Maximum and minimum residual electron density (e Å^{-3})	5.00, -3.63 (located near Hg atom)

Table 7.I.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Hg(S_2COCH_2CH_2CH_2CH_3)2]$

Atom	*x*	*y*	*z*
Hg	0	0.23473(4)	0.25
S(1)	0.2476(5)	0.2587(2)	0.0821(5)
S(2)	0.2955(5)	0.3491(2)	-0.1248(5)
O(1)	0.025(1)	0.3340(4)	0.066(1)
C(1)	0.177(2)	0.3168(6)	0.011(2)
C(2)	-0.055(2)	0.3823(7)	0.013(2)
C(3)	-0.238(2)	0.3869(7)	0.088(2)
C(4)	-0.329(2)	0.4381(9)	0.047(2)
C(5)	-0.254(3)	0.4841(9)	0.129(3)

Table 7.I.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Hg(S_2COCH_2CH_2CH_2CH_3)2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	0.047(2)	0.042(3)	0.066(3)	0.008(2)	0.017(2)	0.002(2)
S(2)	0.058(2)	0.038(2)	0.065(3)	0.003(2)	0.017(2)	0.002(2)
O(1)	0.037(5)	0.043(6)	0.075(7)	0.014(5)	-0.004(5)	-0.006(6)
C(1)	0.039(7)	0.033(9)	0.07(1)	0.004(8)	-0.002(7)	-0.014(9)
C(2)	0.06(1)	0.035(10)	0.08(1)	0.007(8)	0.001(8)	-0.022(10)
C(3)	0.059(9)	0.04(1)	0.09(1)	0.005(9)	0.00(1)	-0.01(1)
C(4)	0.08(1)	0.08(2)	0.09(2)	0.03(1)	0.00(1)	-0.02(1)
C(5)	0.13(2)	0.07(2)	0.15(2)	0.04(2)	-0.02(2)	-0.02(2)

Table 7.I.4: Fractional atomic coordinates and B_{eq} values (Å^2) for the hydrogen atoms in [Hg($S_2COCH_2CH_2CH_2CH_3$)$_2$]*

Atom	x	y	z	B_{eq}
H(2a)	0.0206	0.4115	0.0440	5.4817
H(2b)	-0.0671	0.3819	-0.0987	5.4817
H(3a)	-0.2240	0.3851	0.2003	5.9597
H(3b)	-0.3132	0.3583	0.0534	5.9597
H(4a)	-0.3146	0.4437	-0.0642	7.6597
H(4b)	-0.4558	0.4354	0.0715	7.6597
H(5a)	-0.1256	0.4869	0.1082	10.9046
H(5b)	-0.3143	0.5154	0.0941	10.9046
H(5c)	-0.2722	0.4800	0.2403	10.9046

* H atoms were included in their calculated positions, C-H 0.97Å.

Table 7.I.5: Bond distances (Å) for [Hg($S_2COCH_2CH_2CH_2CH_3$)$_2$]

Hg — S(1)	2.417(4)	O(1) — C(2)	1.45(2)
Hg — S(2)*	2.844(4)	C(2) — C(3)	1.51(2)
S(1) — C(1)	1.69(2)	C(3) — C(4)	1.52(2)
S(2) — C(1)	1.68(2)	C(4) — C(5)	1.49(3)
O(1) — C(1)	1.30(2)		

* symmetry operation: 0.5 – x, 0.5 – y, 0.5 + z.

Table 7.I.6: Bond angles (deg.) for [Hg($S_2COCH_2CH_2CH_2CH_3$)2]

S(1) — Hg — S(1)**	150.5(2)	S(1) — C(1) — S(2)	121.8(9)
S(1) — Hg — S(2)*	90.8(1)	S(1) — C(1) — O(1)	116(1)
S(1)** — Hg — S(2)*	111.8(1)	S(2) — C(1) — O(1)	123(1)
S(2)* — Hg — S(2)***	81.8(2)	O(1) — C(2) — C(3)	108(1)
Hg — S(1) — C(1)	102.0(5)	C(2) — C(3) — C(4)	111(2)
Hg — S(2) — C(1)	99.7(5)	C(3) — C(4) — C(5)	114(2)
C(1) — O(1) — C(2)	122(1)		

* symmetry operation: 0.5 – *x*, 0.5 – *y*, 0.5 + *z*. ** symmetry operation: - *x*, *y*, 0.5 – *z*, *** symmetry operation: -0.5 + *x*, 0.5 – *y*, - *z*.

Table 7.I.7: Observed and calculated structure factors for $[Hg(S_2COCH_2CH_2CH_2CH_3)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	-10	240	422	99	0	10	-5	1408	1221	99	0	20	-2	370	353	99	1	1	-5	1936	1809	99	1	6	-6	270	346	99
0	0	-8	616	924	99	0	10	-4	311	299	99	0	20	-1	1019	1045	99	1	1	-4	54	27	99	1	6	-5	-43	60	99
0	0	-6	1421	1825	99	0	10	-3	1819	1721	99	0	20	0	112	67	99	1	1	-3	1954	1876	99	1	6	-4	337	291	99
0	0	-4	1166	1298	99	0	10	-2	975	899	99	0	22	-8	124	190	99	1	1	-2	603	590	99	1	6	-3	372	386	99
0	0	-2	735	700	99	0	10	-1	1422	1529	99	0	22	-7	350	415	99	1	1	-1	3265	3254	99	1	6	-2	1352	1340	99
0	2	-11	178	24	99	0	10	0	2561	2624	99	0	22	-6	141	179	99	1	2	-11	-408	31	99	1	6	-1	35	139	99
0	2	-10	-76	413	99	0	12	-10	221	142	99	0	22	-5	356	377	99	1	2	-10	-146	96	99	1	7	-10	331	279	99
0	2	-9	257	225	99	0	12	-9	691	586	99	0	22	-4	482	590	99	1	2	-9	76	98	99	1	7	-9	466	465	99
0	2	-8	636	788	99	0	12	-8	206	213	99	0	22	-3	775	768	99	1	2	-8	-50	132	99	1	7	-8	357	401	99
0	2	-7	383	455	99	0	12	-7	954	872	99	0	22	-2	616	604	99	1	2	-7	139	215	99	1	7	-7	587	723	99
0	2	-6	1490	1683	99	0	12	-6	836	711	99	0	22	-1	914	916	99	1	2	-6	87	153	99	1	7	-6	673	791	99
0	2	-5	409	398	99	0	12	-5	891	763	99	0	22	0	-214	94	99	1	2	-5	217	206	99	1	7	-5	1027	1139	99
0	2	-4	1711	1724	99	0	12	-4	272	229	99	0	24	-7	196	291	99	1	2	-4	780	670	99	1	7	-4	1196	1280	99
0	2	-3	848	830	99	0	12	-3	1536	1467	99	0	24	-6	-230	241	99	1	2	-3	444	410	99	1	7	-3	1879	1893	99
0	2	-2	2004	2071	99	0	12	-2	476	458	99	0	24	-5	125	212	99	1	2	-2	1021	1058	99	1	7	-2	1180	1166	99
0	4	-11	-217	93	99	0	12	-1	2086	2276	99	0	24	-4	464	316	99	1	2	-1	323	280	99	1	7	-1	1724	1779	99
0	4	-10	349	407	99	0	12	0	1794	1792	99	0	24	-3	665	683	99	1	3	-11	258	330	99	1	7	0	2064	1882	99
0	4	-9	317	324	99	0	14	-10	-79	30	99	0	24	-2	535	556	99	1	3	-10	241	118	99	1	8	-10	130	95	99
0	4	-8	550	536	99	0	14	-9	623	589	99	0	24	-1	588	605	99	1	3	-9	421	581	99	1	8	-9	-140	86	99
0	4	-7	484	497	99	0	14	-8	100	25	99	0	24	0	752	725	99	1	3	-8	-126	310	99	1	8	-8	-191	112	99
0	4	-6	1597	1533	99	0	14	-7	776	715	99	0	26	-7	138	128	99	1	3	-7	902	983	99	1	8	-7	-152	218	99
0	4	-5	288	294	99	0	14	-6	376	342	99	0	26	-6	200	280	99	1	3	-6	357	363	99	1	8	-6	112	126	99
0	4	-4	1999	1871	99	0	14	-5	956	884	99	0	26	-5	222	232	99	1	3	-5	1697	1630	99	1	8	-5	78	131	99
0	4	-3	842	799	99	0	14	-4	638	583	99	0	26	-4	339	305	99	1	3	-4	329	267	99	1	8	-4	367	348	99
0	4	-2	2770	2684	99	0	14	-3	1725	1666	99	0	26	-3	676	672	99	1	3	-3	2245	2204	99	1	8	-3	291	303	99
0	4	-1	1583	1540	99	0	14	-2	164	94	99	0	26	-2	521	493	99	1	3	-2	1398	1390	99	1	8	-2	48	61	99
0	4	0	2036	2128	99	0	14	-1	1642	1705	99	0	26	-1	239	198	99	1	3	-1	2636	2689	99	1	8	-1	365	361	99
0	6	-11	222	190	99	0	14	0	813	784	99	0	26	0	780	785	99	1	3	0	124	104	99	1	9	-10	379	317	99
0	6	-10	384	343	99	0	16	-9	502	460	99	0	28	-6	88	297	99	1	4	-11	-173	9	99	1	9	-9	217	364	99
0	6	-9	-80	295	99	0	16	-8	-270	100	99	0	28	-5	-325	195	99	1	4	-10	124	14	99	1	9	-8	434	469	99
0	6	-8	400	419	99	0	16	-7	565	550	99	0	28	-4	198	395	99	1	4	-9	188	60	99	1	9	-7	564	619	99
0	6	-7	439	448	99	0	16	-6	185	107	99	0	28	-3	433	503	99	1	4	-8	132	171	99	1	9	-6	789	967	99
0	6	-6	1498	1391	99	0	16	-5	907	888	99	0	28	-2	500	501	99	1	4	-7	38	79	99	1	9	-5	765	912	99
0	6	-5	1069	997	99	0	16	-4	431	386	99	0	28	-1	-145	47	99	1	4	-6	211	206	99	1	9	-4	1151	1256	99
0	6	-4	2003	1856	99	0	16	-3	1842	1971	99	0	28	0	535	488	99	1	4	-5	129	199	99	1	9	-3	1135	1101	99
0	6	-3	1813	1665	99	0	16	-2	348	348	99	0	30	-4	486	421	99	1	4	-4	681	600	99	1	9	-2	1672	1667	99
0	6	-2	1970	1857	99	0	16	-1	1083	1147	99	0	30	-3	198	248	99	1	4	-3	-138	21	99	1	9	-1	1563	1626	99
0	6	-1	1016	899	99	0	16	0	246	226	99	0	30	-2	442	481	99	1	4	-2	1384	1411	99	1	9	0	1778	1656	99
0	6	0	1740	1824	99	0	18	-9	137	141	99	0	30	-1	-258	27	99	1	4	-1	1520	1546	99	1	10	-10	-210	151	99
0	8	-10	232	257	99	0	18	-8	183	64	99	0	30	0	132	196	99	1	5	-11	-112	301	99	1	10	-9	201	41	99
0	8	-9	420	365	99	0	18	-7	410	446	99	0	32	-3	82	43	99	1	5	-10	-357	193	99	1	10	-8	255	163	99
0	8	-8	549	534	99	0	18	-6	120	61	99	0	32	-2	167	356	99	1	5	-9	332	513	99	1	10	-7	196	163	99
0	8	-7	486	439	99	0	18	-5	882	909	99	0	32	-1	151	53	99	1	5	-8	345	407	99	1	10	-6	-62	231	99
0	8	-6	1335	1185	99	0	18	-4	326	321	99	0	32	0	104	332	99	1	5	-7	753	887	99	1	10	-5	151	183	99
0	8	-5	1469	1299	99	0	18	-3	2058	1845	99	1	0	-10	187	158	99	1	5	-6	470	524	99	1	10	-4	401	359	99
0	8	-4	857	725	99	0	18	-2	133	74	99	1	0	-8	184	137	99	1	5	-5	1277	1296	99	1	10	-3	119	1	99
0	8	-3	2120	1972	99	0	18	-1	767	738	99	1	0	-6	327	372	99	1	5	-4	907	936	99	1	10	-2	-29	19	99
0	8	-2	1637	1587	99	0	18	0	247	264	99	1	0	-4	467	373	99	1	5	-3	2488	2474	99	1	10	-1	168	148	99
0	8	-1	988	1166	99	0	20	-9	14	326	99	1	0	-2	901	860	99	1	5	-2	1199	1192	99	1	11	-10	413	314	99
0	8	0	2829	2954	99	0	20	-8	-152	78	99	1	1	-11	173	327	99	1	5	-1	1908	1965	99	1	11	-9	180	231	99
0	10	-10	221	231	99	0	20	-7	427	437	99	1	1	-10	140	48	99	1	5	0	1227	1204	99	1	11	-8	551	601	99
0	10	-9	578	480	99	0	20	-6	149	120	99	1	1	-9	537	636	99	1	6	-10	82	2	99	1	11	-7	509	476	99
0	10	-8	430	429	99	0	20	-5	698	713	99	1	1	-8	161	102	99	1	6	-9	-154	18	99	1	11	-6	851	953	99
0	10	-7	693	636	99	0	20	-4	628	672	99	1	1	-7	996	1062	99	1	6	-8	111	147	99	1	11	-5	490	638	99
0	10	-6	1113	968	99	0	20	-3	1153	1042	99	1	1	-6	168	155	99	1	6	-7	96	156	99	1	11	-4	1131	1281	99
1	11	-3	668	651	99	1	17	-8	472	442	99	1	23	-6	498	437	99	1	31	0	74	72	99	2	5	-9	-190	30	99
1	11	-2	1538	1510	99	1	17	-7	-46	64	99	1	23	-5	-215	281	99	1	32	-3	235	55	99	2	5	-8	197	24	99
1	11	-1	1198	1208	99	1	17	-6	876	785	99	1	23	-4	454	463	99	1	32	-2	-347	54	99	2	5	-7	122	43	99
1	11	0	2314	2266	99	1	17	-5	-274	82	99	1	23	-3	521	529	99	1	32	-1	252	190	99	2	5	-6	108	40	99
1	12	-10	-112	76	99	1	17	-4	1021	1097	99	1	23	-2	599	648	99	1	33	-1	360	319	99	2	5	-5	275	162	99
1	12	-9	-287	67	99	1	17	-3	192	63	99	1	23	-1	482	496	99	1	33	0	-185	25	99	2	5	-4	124	29	99
1	12	-8	114	102	99	1	17	-2	1525	1510	99	1	23	0	853	863	99	2	0	-10	477	478	99	2	5	-3	107	154	99
1	12	-7	181	2	99	1	17	-1	264	182	99	1	24	-7	33	40	99	2	0	-8	735	683	99	2	5	-2	48	25	99
1	12	-6	186	144	99	1	17	0	1097	1096	99	1	24	-6	-215	102	99	2	0	-6	1088	985	99	2	5	-1	448	462	99
1	12	-5	425	534	99	1	18	-9	-193	37	99	1	24	-5	175	230	99	2	0	-4	2785	2687	99	2	6	-10	228	361	99
1	12	-4	413	372	99	1	18	-8	237	41	99	1	24	-4	249	60	99	2	0	-2	2630	2657	99	2	6	-9	296	347	99
1	12	-3	671	590	99	1	18	-7	-112	183	99	1	24	-3	146	163	99	2	0	0	634	596	99	2	6	-8	882	902	99
1	12	-2	299	252	99	1	18	-6	188	81	99	1	24	-2	347	292	99	2	1	-10	260	27	99	2	6	-7	574	544	99
1	12	-1	715	685	99	1	18	-5	-86	77	99	1	24	-1	249	288	99	2	1	-9	193	79	99	2	6	-6	543	524	99
1	13	-10	388	299	99	1	18	-4	256	232	99	1	25	-7	402	280	99	2	1	-8	171	3	99	2	6	-5	831	734	99
1	13	-9	199	128	99	1	18	-3	124	348	99	1	25	-6	370	279	99	2	1	-7	-273	85	99	2	6	-4	1451	1383	99
1	13	-8	757	667	99	1	18	-2	67	188	99	1	25	-5	199	303	99	2	1	-6	-121	44	99	2	6	-3	712	741	99
1	13	-7	220	215	99	1	18	-1	158	39	99	1	25	-4	232	355	99	2	1	-5	177	200	99	2	6	-2	2332	2337	99
1	13	-6	811	851	99	1	19	-9	114	75	99	1	25	-3	554	494	99	2	1	-4	111	149	99	2	6	-1	1655	1650	99
1	13	-5	424	505	99	1	19	-8	326	386	99	1	25	-2	583	551	99	2	1	-3	444	394	99	2	6	0	1618	1704	99
1	13	-4	1173	1308	99	1	19	-7	157	99	99	1	25	-1	545	552	99	2	1	-2	402	402	99	2	7	-10	108	31	99
1	13	-3	314	316	99	1	19	-6	845	680	99	1	25	0	358	405	99	2	1	-1	58	50	99	2	7	-9	118	20	99
1	13	-2	1633	1520	99	1	19	-5	-259	274	99	1	26	-6	-316	117	99	2	2	-10	490	472	99	2	7	-8	201	57	99
1	13	-1	741	680	99	1	19	-4	934	922	99	1	26	-5	213	114	99	2	2	-9	63	25	99	2	7	-7	36	37	99
1	13	0	1982	2097	99	1	19	-3	170	248	99	1	26	-4	137	10	99	2	2	-8	799	765	99	2	7	-6	283	215	99
1	14	-10	266	18	99	1	19	-2	1050	1122	99	1	26	-3	222	26	99	2	2	-7	-140	122	99	2	7	-5	230	154	99
1	14	-9	-125	32	99	1	19	-1	361	291	99	1	26	-2	196	232	99	2	2	-6	1099	1009	99	2	7	-4	106	33	99
1	14	-8	-95	51	99	1	19	0	1161	1187	99	1	26	-1	166	81	99	2	2	-5	537	473	99	2	7	-3	264	298	99
1	14	-7	-33	62	99	1	20	-8	231	34	99	1	27	-6	-345	123	99	2	2	-4	2098	2040	99	2	7	-2	302	301	99
1	14	-6	-42	136	99	1	20	-7	241	103	99	1	27	-5	221	374	99	2	2	-3	727	750	99	2	7	-1	398	408	99
1	14	-5	280	339	99	1	20	-6	250	181	99	1	27	-4	-278	334	99	2	2	-2	2569	2707	99	2	8	-10	151	302	99
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5	20	-1	198	144	99	6	2	-5	150	124	99	6	8	-1	794	741	99	6	15	-1	-149	140	99	7	0	-6	104	55	99
5	21	-6	405	352	99	6	2	-4	926	940	99	6	8	0	236	201	99	6	16	-6	-107	115	99	7	0	-4	-127	142	99
5	21	-5	183	240	99	6	2	-3	150	129	99	6	9	-8	-200	8	99	6	16	-5	505	501	99	7	0	-2	288	195	99
5	21	-4	170	323	99	6	2	-2	1155	1105	99	6	9	-7	174	104	99	6	16	-4	-283	127	99	7	1	-7	353	377	99
5	21	-3	348	355	99	6	2	-1	264	235	99	6	9	-6	191	70	99	6	16	-3	507	485	99	7	1	-6	-75	46	99
5	21	-2	517	523	99	6	2	0	854	856	99	6	9	-5	-118	74	99	6	16	-2	30	69	99	7	1	-5	542	571	99
5	21	-1	-119	132	99	6	3	-8	107	6	99	6	9	-4	101	86	99	6	16	-1	720	726	99	7	1	-4	128	162	99
5	21	0	619	672	99	6	3	-7	-157	1	99	6	9	-3	117	138	99	6	16	0	106	107	99	7	1	-3	817	791	99
5	22	-6	239	38	99	6	3	-6	89	108	99	6	9	-2	199	273	99	6	17	-6	-74	120	99	7	1	-2	79	45	99
5	22	-5	-342	62	99	6	3	-5	126	2	99	6	9	-1	108	113	99	6	17	-5	-275	21	99	7	1	-1	726	703	99
5	22	-4	105	80	99	6	3	-4	-108	58	99	6	10	-8	66	222	99	6	17	-4	112	18	99	7	1	0	121	15	99

h	k	l	Fo	Fc	sigF2
7	2	-7	-74	9	99
7	2	-6	-173	47	99
7	2	-5	76	78	99
7	2	-4	-209	129	99
7	2	-3	-151	5	99
7	2	-2	284	200	99
7	2	-1	191	148	99
7	3	-7	359	399	99
7	3	-6	71	124	99
7	3	-5	407	450	99
7	3	-4	320	332	99
7	3	-3	674	631	99
7	3	-2	197	207	99
7	3	-1	860	817	99
7	3	0	89	51	99
7	4	-7	89	20	99
7	4	-6	127	43	99
7	4	-5	-156	21	99
7	4	-4	62	170	99
7	4	-3	99	84	99
7	4	-2	247	180	99
7	4	-1	88	151	99
7	5	-7	419	395	99
7	5	-6	203	193	99
7	5	-5	291	302	99
7	5	-4	280	287	99
7	5	-3	548	543	99
7	5	-2	408	391	99
7	5	-1	769	808	99
7	5	0	254	253	99
7	6	-7	-64	61	99
7	6	-6	224	70	99
7	6	-5	200	132	99
7	6	-4	-215	183	99
7	6	-3	-190	54	99
7	6	-2	211	193	99
7	6	-1	52	97	99
7	7	-7	338	320	99
7	7	-6	162	250	99
7	7	-5	297	279	99
7	7	-4	273	249	99
7	7	-3	600	552	99
7	7	-2	463	444	99
7	7	-1	595	584	99
7	7	0	629	618	99
7	8	-7	21	44	99
7	8	-6	74	8	99
7	8	-5	-71	111	99
7	8	-4	125	154	99
7	8	-3	149	14	99
7	8	-2	229	180	99
7	8	-1	206	224	99
7	9	-7	231	214	99
7	9	-6	279	301	99
7	9	-5	288	317	99
7	9	-4	385	357	99
7	9	-3	500	497	99
7	9	-2	483	447	99
7	9	-1	370	358	99
7	9	0	759	727	99
7	10	-6	-115	40	99
7	10	-5	-84	17	99
7	10	-4	106	158	99
7	10	-3	106	87	99
7	10	-2	219	233	99
7	10	-1	145	183	99
7	11	-6	308	329	99
7	11	-5	191	258	99
7	11	-4	533	535	99
7	11	-3	341	349	99
7	11	-2	504	495	99
7	11	-1	213	232	99
7	11	0	555	512	99
7	12	-6	132	41	99
7	12	-5	-84	6	99
7	12	-4	-45	71	99
7	12	-3	123	56	99
7	12	-2	186	110	99
7	12	-1	109	134	99
7	13	-6	305	334	99
7	13	-5	200	94	99
7	13	-4	559	567	99
7	13	-3	231	146	99
7	13	-2	552	534	99
7	13	-1	203	221	99
7	13	0	427	393	99
7	14	-6	31	11	99
7	14	-5	-98	11	99
7	14	-4	187	18	99
7	14	-3	-135	11	99
7	14	-2	55	12	99
7	14	-1	146	17	99
7	15	-5	93	54	99
7	15	-4	445	458	99
7	15	-3	164	18	99
7	15	-2	487	515	99
7	15	-1	71	154	99
7	15	0	528	472	99
7	16	-5	-346	76	99
7	16	-4	-146	74	99
7	16	-3	95	25	99
7	16	-2	127	67	99
7	16	-1	187	95	99
7	17	-5	224	92	99
7	17	-4	-147	334	99
7	17	-3	-183	11	99
7	17	-2	419	433	99
7	17	-1	71	8	99
7	17	0	640	611	99
7	18	-4	-194	86	99
7	18	-3	144	26	99
7	18	-2	137	127	99
7	18	-1	-121	127	99
7	19	-4	287	295	99
7	19	-3	-247	24	99
7	19	-2	315	380	99
7	19	-1	-208	175	99
7	19	0	463	471	99
7	20	-3	149	40	99
7	20	-2	138	106	99
7	20	-1	157	101	99
7	21	-3	-162	52	99
7	21	-2	216	318	99
7	21	-1	271	243	99
7	21	0	321	280	99
7	22	-2	247	14	99
7	22	-1	-59	81	99
7	23	0	109	206	99
8	0	-6	407	455	99
8	0	-4	377	383	99
8	0	-2	587	544	99
8	0	0	701	642	99
8	1	-6	83	47	99
8	1	-5	146	52	99
8	1	-4	116	56	99
8	1	-3	70	73	99
8	1	-2	-125	14	99
8	1	-1	-91	116	99
8	2	-6	441	432	99
8	2	-5	-65	96	99
8	2	-4	398	383	99
8	2	-3	183	203	99
8	2	-2	481	471	99
8	2	-1	-35	33	99
8	2	0	701	714	99
8	3	-6	-43	68	99
8	3	-5	-87	13	99
8	3	-4	96	72	99
8	3	-3	108	11	99
8	3	-2	-157	36	99
8	3	-1	104	58	99
8	4	-6	381	390	99
8	4	-5	39	137	99
8	4	-4	361	320	99
8	4	-3	282	273	99
8	4	-2	378	388	99
8	4	-1	140	154	99
8	4	0	758	787	99
8	5	-6	101	3	99
8	5	-5	-106	35	99
8	5	-4	132	13	99
8	5	-3	-114	27	99
8	5	-2	75	10	99
8	5	-1	173	149	99
8	6	-5	197	176	99
8	6	-4	289	266	99
8	6	-3	242	253	99
8	6	-2	330	331	99
8	6	-1	274	311	99
8	6	0	676	704	99
8	7	-5	-217	12	99
8	7	-4	53	37	99
8	7	-3	-150	55	99
8	7	-2	45	80	99
8	7	-1	-195	120	99
8	8	-5	-70	200	99
8	8	-4	150	261	99
8	8	-3	316	315	99
8	8	-2	277	271	99
8	8	-1	388	374	99
8	8	0	527	534	99
8	9	-5	73	39	99
8	9	-4	109	61	99
8	9	-3	95	102	99
8	9	-2	125	38	99
8	9	-1	69	9	99
8	10	-5	245	253	99
8	10	-4	242	251	99
8	10	-3	444	421	99
8	10	-2	20	211	99
8	10	-1	370	351	99
8	10	0	338	330	99
8	11	-5	40	29	99
8	11	-4	-92	20	99
8	11	-3	148	59	99
8	11	-2	-44	54	99
8	11	-1	-124	78	99
8	12	-4	139	125	99
8	12	-3	507	466	99
8	12	-2	43	111	99
8	12	-1	384	340	99
8	12	0	278	300	99
8	13	-4	-88	30	99
8	13	-3	-121	4	99
8	13	-2	-55	46	99
8	13	-1	-136	4	99
8	14	-4	102	58	99
8	14	-3	450	483	99
8	14	-2	122	58	99
8	14	-1	338	319	99
8	14	0	154	239	99
8	15	-3	171	40	99
8	15	-2	-88	28	99
8	15	-1	173	65	99
8	16	-3	426	403	99
8	16	-2	87	12	99
8	16	-1	378	333	99
8	16	0	96	100	99
8	17	-2	183	93	99
8	17	-1	147	68	99
8	18	-1	277	303	99
8	18	0	-247	25	99
9	0	-4	118	126	99
9	0	-2	-35	197	99
9	1	-4	120	28	99
9	1	-3	304	318	99
9	1	-2	-12	34	99
9	1	-1	460	437	99
9	1	0	100	73	99
9	2	-3	-150	38	99
9	2	-2	126	146	99
9	2	-1	128	31	99
9	3	-3	324	339	99
9	3	-2	159	86	99
9	3	-1	360	361	99
9	3	0	227	196	99
9	4	-3	10	15	99
9	4	-2	114	37	99
9	4	-1	-140	10	99
9	5	-3	298	300	99
9	5	-2	147	162	99
9	5	-1	322	303	99
9	5	0	226	206	99
9	6	-3	142	29	99
9	6	-2	-164	3	99
9	6	-1	101	69	99
9	7	-3	236	245	99
9	7	-2	260	224	99
9	7	-1	250	279	99
9	7	0	217	184	99
9	8	-3	91	43	99
9	8	-2	-126	79	99
9	8	-1	126	51	99
9	9	-2	251	245	99
9	9	-1	97	249	99
9	9	0	261	252	99
9	10	-2	168	172	99
9	10	-1	-172	27	99
9	11	-1	224	194	99
9	11	0	357	305	99

APPENDIX 7.II

$[Hg(S_2CO\text{-}3\text{-}MeBu)_2]$

Table 7.II.1: Crystallographic Parameters for $[Hg(S_2COCH_2CH_2CH(CH_3)2)2]$

Formula	$C_{12}H_{22}HgO_2S_4$
Formula weight	527.13
Crystal morphology / colour	needle / pale yellow
Crystal dimensions (mm)	0.07 x 0.08 x 0.42
Crystal system	monoclinic
Space group	*Cc* (# 9)
a (Å)	28.72(2)
b (Å)	5.828(9)
c (Å)	11.41(2)
α (°)	90
β (°)	99.7(1)
γ (°)	90
V (Å^3)	1882(4)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.859
F(000)	1016
Temperature (°K)	200
μ (cm^{-1})	86.34
θ limits, cell (°)	15.3 – 18.9
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 37, 0 – 7, -14 – 14
Range of transmission factors	0.895 – 1
Scan technique	ω:2θ
No. of data measured	2459
No. of unique data	2412
R_{amal}	0.087
No. of parameters	170
R (I≥0σ(I))	0.116
R_w (I≥0σ(I))	0.135
R (I≥3σ(I))	0.054
R_w (I≥3σ(I))	0.052
Maximum and minimum residual electron density (e Å^{-3})	2.81, -3.11

Table 7.II.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Hg(S_2COCH_2CH_2CH(CH_3)_2)_2]$

Atom	*x*	*y*	*z*
Hg	0	0.0416(1)	0.5
S(1)	0.0118(4)	-0.3805(9)	0.5277(8)
S(2)	0.0632(3)	-0.7438(9)	0.4190(6)
S(3)	-0.0719(3)	0.161(1)	0.3470(6)
S(4)	-0.0202(3)	-0.1379(9)	0.2031(5)
O(1)	0.0740(6)	-0.297(3)	0.392(1)
O(2)	-0.0818(6)	0.156(3)	0.115(1)
C(1)	0.0514(7)	-0.465(4)	0.442(2)
C(2)	0.109(1)	-0.369(5)	0.318(3)
C(3)	0.1244(10)	-0.143(4)	0.267(2)
C(4)	0.162(1)	-0.176(5)	0.191(3)
C(5)	0.207(1)	-0.276(6)	0.257(3)
C(6)	0.172(1)	0.061(5)	0.137(3)
C(7)	-0.0608(7)	0.072(4)	0.215(2)
C(8)	-0.113(1)	0.354(5)	0.109(3)
C(9)	-0.137(1)	0.393(5)	-0.009(2)
C(10)	-0.171(1)	0.208(6)	-0.065(3)
C(11)	-0.214(1)	0.22(1)	-0.003(3)
C(12)	-0.185(1)	0.251(8)	-0.191(3)

Table 7.II.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Hg(S_2COCH_2CH_2CH(CH_3)2)2]$

Atom	$U11$	$U22$	$U33$	$U12$	$U13$	$U23$
Hg	0.0460(5)	0.0424(3)	0.0410(4)	-0.007(1)	0.0104(3)	-0.001(1)
S(1)	0.069(9)	0.044(2)	0.087(8)	0.005(4)	0.058(7)	0.005(3)
S(2)	0.052(5)	0.038(3)	0.080(5)	0.002(3)	0.034(4)	0.000(3)
S(3)	0.055(5)	0.091(5)	0.045(4)	0.014(4)	0.012(4)	-0.008(3)
S(4)	0.060(5)	0.048(3)	0.030(3)	-0.004(3)	0.012(3)	-0.003(2)
O(1)	0.04(1)	0.048(9)	0.038(9)	0.002(9)	0.018(8)	-0.012(8)
O(2)	0.06(1)	0.050(9)	0.034(10)	0.01(1)	-0.003(9)	-0.003(8)
C(1)	0.02(1)	0.043(10)	0.06(1)	-0.03(1)	0.02(1)	-0.03(1)
C(2)	0.07(2)	0.05(1)	0.10(2)	-0.01(2)	0.07(2)	-0.01(2)
C(3)	0.06(2)	0.07(2)	0.06(2)	-0.01(2)	0.02(2)	0.01(1)
C(4)	0.07(2)	0.07(2)	0.12(3)	0.01(2)	0.07(2)	-0.01(2)
C(5)	0.08(3)	0.12(3)	0.13(3)	0.02(2)	0.05(2)	0.02(2)
C(6)	0.14(3)	0.09(2)	0.11(3)	0.00(2)	0.06(3)	0.06(2)
C(7)	0.01(1)	0.08(2)	0.04(1)	0.00(1)	0.00(1)	0.00(1)
C(8)	0.07(3)	0.07(2)	0.10(3)	0.01(2)	0.04(2)	-0.01(2)
C(9)	0.05(2)	0.10(2)	0.05(2)	0.01(2)	-0.01(1)	0.02(1)
C(10)	0.03(2)	0.11(3)	0.11(3)	-0.01(2)	-0.02(2)	0.01(2)
C(11)	0.04(2)	0.45(9)	0.11(3)	0.01(4)	0.04(2)	0.09(4)
C(12)	0.12(4)	0.15(4)	0.10(3)	0.03(3)	0.02(3)	0.05(3)

Table 7.II.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Hg(S_2COCH_2CH_2CH(CH_3)_2)_2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.0950	-0.4715	0.2553	6.4436
H(2b)	0.1359	-0.4440	0.3663	6.4436
H(3a)	0.0970	-0.0746	0.2178	5.8088
H(3b)	0.1363	-0.0409	0.3317	5.8088
H(4a)	0.1503	-0.2793	0.1263	7.6171
H(5a)	0.1997	-0.4202	0.2928	9.9169
H(5b)	0.2288	-0.3013	0.2034	9.9169
H(5c)	0.2203	-0.1701	0.3195	9.9169
H(6a)	0.1426	0.1241	0.0950	10.0367
H(6b)	0.1941	0.0424	0.0822	10.0367
H(6c)	0.1853	0.1651	0.2004	10.0367
H(8a)	-0.1363	0.3275	0.1594	6.9361
H(8b)	-0.0944	0.4884	0.1360	6.9361
H(9a)	-0.1540	0.5355	-0.0093	6.3223
H(9b)	-0.1126	0.4095	-0.0594	6.3223
H(10a)	-0.1565	0.0586	-0.0531	7.9544
H(11a)	-0.2358	0.0949	-0.0324	18.5267
H(11b)	-0.2040	0.1988	0.0822	18.5267
H(11c)	-0.2299	0.3642	-0.0183	18.5267
H(12a)	-0.2016	0.3977	-0.2030	11.7559
H(12b)	-0.1574	0.2556	-0.2291	11.7559
H(12c)	-0.2062	0.1302	-0.2267	11.7559

* H atoms were included in their calculated positions, C-H 0.97Å.

Table 7.II.5: Bond distances (Å) for [Hg($S_2COCH_2CH_2CH(CH_3)_2)_2$]

Hg — S(1)	2.496(6)	O(2) — C(7)	1.29(2)
Hg — S(2)*	2.507(6)	O(2) — C(8)	1.45(3)
Hg — S(3)	2.566(8)	C(2) — C(3)	1.53(3)
Hg — S(4)**	2.546(5)	C(3) — C(4)	1.51(3)
S(1) — C(1)	1.69(2)	C(4) — C(5)	1.48(4)
S(2) — C(1)	1.69(2)	C(4) — C(6)	1.56(4)
S(3) — C(7)	1.67(2)	C(8) — C(9)	1.42(4)
S(4) — C(7)	1.71(2)	C(9) — C(10)	1.53(4)
O(1) — C(1)	1.35(2)	C(10) — C(11)	1.52(4)
O(1) — C(2)	1.48(2)	C(10) — C(12)	1.45(4)

* symmetry operation: x, $1 + y$, z. ** symmetry operation: x, $- y$, $0.5 + z$.

Table 7.II.6: Bond angles (deg.) for $[Hg(S_2COCH_2CH_2CH(CH_3)_2)_2]$

S(1) — Hg — S(2)*	116.6(3)	S(2) — C(1) — O(1)	121(1)
S(1) — Hg — S(3)	115.4(3)	O(1) — C(2) — C(3)	104(2)
S(1) — Hg — S(4)**	98.6(2)	C(2) — C(3) — C(4)	113(2)
S(2)* — Hg — S(3)	99.4(2)	C(3) — C(4) — C(5)	113(3)
S(2)* — Hg — S(4)**	121.1(2)	C(3) — C(4) — C(6)	108(2)
S(3) — Hg — S(4)**	106.2(2)	C(5) — C(4) — C(6)	111(3)
Hg — S(1) — C(1)	107.8(8)	S(3) — C(7) — S(4)	122(1)
Hg — S(2)* — C(1)*	104.2(7)	S(3) — C(7) — O(2)	123(2)
Hg — S(3) — C(7)	106.0(8)	S(4) — C(7) — O(2)	115(1)
Hg — S(4)** — C(7)**	100.3(7)	O(2) — C(8) — C(9)	112(2)
C(1) — O(1) — C(2)	117(2)	C(8) — C(9) — C(10)	117(3)
C(7) — O(2) — C(8)	123(2)	C(9) — C(10) — C(11)	108(3)
S(1) — C(1) — S(2)	123(1)	C(9) — C(10) — C(12)	110(3)
S(1) — C(1) — O(1)	117(1)	C(11) — C(10) — C(12)	110(3)

* symmetry operation: x, $1 + y$, z. ** symmetry operation: x, $- y$, $0.5 + z$.

Table 7.II.7: Observed and calculated structure factors for $[Hg(S_2COCH_2CH_2CH(CH_3)_2)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	2	2795	2732	99	1	1	-4	2160	2125	99	1	5	-2	444	462	99	2	2	2	1821	1718	99
0	0	4	2337	2386	99	1	1	-3	1350	1347	99	1	5	-1	1051	1123	99	2	2	3	1104	1031	99
0	0	6	2085	2083	99	1	1	-2	1959	1972	99	1	5	0	327	308	99	2	2	4	1852	1799	99
0	0	8	1347	1381	99	1	1	-1	1169	1046	99	1	5	1	1293	1287	99	2	2	5	1307	1381	99
0	0	10	1339	1298	99	1	1	0	2935	2956	99	1	5	2	392	355	99	2	2	6	1742	1667	99
0	0	12	731	673	99	1	1	1	733	774	99	1	5	3	1264	1268	99	2	2	7	625	642	99
0	0	14	609	537	99	1	1	2	1319	1313	99	1	5	4	434	397	99	2	2	8	1286	1205	99
0	2	0	1139	1122	99	1	1	3	686	680	99	1	5	5	527	585	99	2	2	9	222	340	99
0	2	1	2059	2048	99	1	1	4	1949	1976	99	1	5	6	305	270	99	2	2	10	775	687	99
0	2	2	2387	2308	99	1	1	5	870	907	99	1	5	7	602	646	99	2	2	11	232	472	99
0	2	3	901	908	99	1	1	6	1956	1947	99	1	5	8	293	219	99	2	2	12	464	448	99
0	2	4	2107	2108	99	1	1	7	391	356	99	1	5	9	669	567	99	2	2	13	275	213	99
0	2	5	1540	1604	99	1	1	8	1237	1179	99	1	5	10	372	109	99	2	2	14	-107	356	99
0	2	6	1644	1615	99	1	1	9	214	122	99	1	5	11	286	385	99	2	4	-12	441	361	99
0	2	7	794	839	99	1	1	10	1155	1070	99	1	7	-5	435	498	99	2	4	-11	553	532	99
0	2	8	1237	1118	99	1	1	11	132	365	99	1	7	-4	-117	172	99	2	4	-10	-267	322	99
0	2	9	413	478	99	1	1	12	652	614	99	1	7	-3	440	515	99	2	4	-9	617	540	99
0	2	10	786	714	99	1	1	13	-445	207	99	1	7	-2	182	209	99	2	4	-8	253	354	99
0	2	11	506	491	99	1	1	14	542	475	99	1	7	-1	376	473	99	2	4	-7	891	821	99
0	2	12	602	491	99	1	3	-13	-82	241	99	1	7	0	86	61	99	2	4	-6	402	428	99
0	2	13	166	227	99	1	3	-12	395	492	99	1	7	1	356	415	99	2	4	-5	1095	1099	99
0	2	14	355	381	99	1	3	-11	528	438	99	1	7	2	-188	177	99	2	4	-4	244	246	99
0	4	0	347	358	99	1	3	-10	592	613	99	1	7	3	490	475	99	2	4	-3	1194	1271	99
0	4	1	1279	1250	99	1	3	-9	721	739	99	1	7	4	-114	167	99	2	4	-2	849	913	99
0	4	2	869	933	99	1	3	-8	764	813	99	1	7	5	234	502	99	2	4	-1	1178	1202	99
0	4	3	1476	1498	99	1	3	-7	910	918	99	2	0	-14	582	616	99	2	4	0	586	603	99
0	4	4	471	446	99	1	3	-6	856	915	99	2	0	-12	417	497	99	2	4	1	1376	1356	99
0	4	5	923	914	99	1	3	-5	1210	1212	99	2	0	-10	1177	1134	99	2	4	2	928	902	99
0	4	6	345	269	99	1	3	-4	1225	1354	99	2	0	-8	1766	1849	99	2	4	3	1423	1364	99
0	4	7	817	784	99	1	3	-3	1074	1098	99	2	0	-6	2128	2181	99	2	4	4	659	628	99
0	4	8	490	448	99	1	3	-2	1625	1684	99	2	0	-4	2029	2032	99	2	4	5	831	827	99
0	4	9	653	633	99	1	3	-1	926	910	99	2	0	-2	2816	2743	99	2	4	6	373	368	99
0	4	10	-119	241	99	1	3	0	1155	1160	99	2	0	2	3343	3473	99	2	4	7	874	854	99
0	4	11	529	467	99	1	3	1	771	762	99	2	0	4	2392	2448	99	2	4	8	495	533	99
0	4	12	474	294	99	1	3	2	1932	1922	99	2	0	6	1882	1869	99	2	4	9	620	623	99
0	6	0	89	2	99	1	3	3	898	933	99	2	0	8	700	748	99	2	4	10	290	188	99
0	6	1	532	585	99	1	3	4	1282	1190	99	2	0	10	1273	1239	99	2	4	11	448	377	99
0	6	2	72	173	99	1	3	5	1085	1116	99	2	0	12	799	691	99	2	4	12	259	258	99
0	6	3	656	682	99	1	3	6	869	823	99	2	0	14	-425	436	99	2	6	-9	441	112	99
0	6	4	-168	81	99	1	3	7	880	896	99	2	2	-14	206	351	99	2	6	-8	-86	72	99
0	6	5	403	456	99	1	3	8	1104	975	99	2	2	-13	151	226	99	2	6	-7	543	589	99
0	6	6	-336	67	99	1	3	9	681	719	99	2	2	-12	555	582	99	2	6	-6	122	74	99
0	6	7	456	518	99	1	3	10	458	482	99	2	2	-11	-153	320	99	2	6	-5	436	489	99
0	6	8	-190	98	99	1	3	11	456	460	99	2	2	-10	747	776	99	2	6	-4	169	82	99
0	6	9	147	375	99	1	3	12	307	408	99	2	2	-9	786	749	99	2	6	-3	562	530	99
1	1	-14	475	548	99	1	3	13	458	243	99	2	2	-8	901	927	99	2	6	-2	196	110	99
1	1	-13	389	240	99	1	5	-11	520	453	99	2	2	-7	803	762	99	2	6	-1	635	673	99
1	1	-12	686	558	99	1	5	-10	157	100	99	2	2	-6	1361	1478	99	2	6	0	120	111	99
1	1	-11	216	253	99	1	5	-9	610	528	99	2	2	-5	1348	1312	99	2	6	1	499	612	99
1	1	-10	1064	1010	99	1	5	-8	262	182	99	2	2	-4	2537	2548	99	2	6	2	-154	156	99
1	1	-9	524	410	99	1	5	-7	837	723	99	2	2	-3	1060	1022	99	2	6	3	735	720	99
1	1	-8	1498	1504	99	1	5	-6	224	329	99	2	2	-2	2123	2091	99	2	6	4	190	165	99
1	1	-7	153	90	99	1	5	-5	732	724	99	2	2	-1	2104	2086	99	2	6	5	488	461	99
1	1	-6	1671	1730	99	1	5	-4	350	378	99	2	2	0	2046	1970	99	2	6	6	-168	78	99
1	1	-5	419	408	99	1	5	-3	966	1065	99	2	2	1	1854	1794	99	2	6	7	467	481	99
3	3	11	499	503	99	4	2	-9	910	943	99	4	6	-2	-199	59	99	5	3	2	1541	1432	99
3	3	12	302	368	99	4	2	-8	836	865	99	4	6	-1	681	690	99	5	3	3	724	743	99
3	3	13	357	277	99	4	2	-7	618	585	99	4	6	0	167	163	99	5	3	4	1180	1107	99
3	5	-11	482	465	99	4	2	-6	1460	1485	99	4	6	1	576	587	99	5	3	5	984	953	99
3	5	-10	187	88	99	4	2	-5	1155	1095	99	4	6	2	158	122	99	5	3	6	801	749	99
3	5	-9	563	489	99	4	2	-4	2014	2003	99	4	6	3	762	705	99	5	3	7	626	676	99
3	5	-8	-132	222	99	4	2	-3	1591	1506	99	4	6	4	156	78	99	5	3	8	983	923	99
3	5	-7	904	857	99	4	2	-2	2528	2453	99	4	6	5	481	473	99	5	3	9	720	627	99
3	5	-6	313	298	99	4	2	-1	1774	1706	99	4	6	6	-428	112	99	5	3	10	442	356	99
3	5	-5	782	796	99	4	2	0	2238	2136	99	4	6	7	473	482	99	5	3	11	545	503	99
3	5	-4	245	323	99	4	2	1	1415	1328	99	4	6	8	102	37	99	5	3	12	-279	359	99
3	5	-3	909	916	99	4	2	2	1668	1611	99	5	1	-14	524	546	99	5	3	13	350	267	99
3	5	-2	493	504	99	4	2	3	1060	1033	99	5	1	-13	343	171	99	5	5	-11	-128	456	99
3	5	-1	1060	1037	99	4	2	4	1341	1327	99	5	1	-12	662	631	99	5	5	-10	169	131	99
3	5	0	279	284	99	4	2	5	1260	1267	99	5	1	-11	259	203	99	5	5	-9	426	457	99
3	5	1	1288	1235	99	4	2	6	1438	1368	99	5	1	-10	1121	1109	99	5	5	-8	-131	239	99
3	5	2	145	265	99	4	2	7	522	564	99	5	1	-9	951	878	99	5	5	-7	908	912	99
3	5	3	1307	1268	99	4	2	8	1267	1226	99	5	1	-8	1395	1408	99	5	5	-6	222	223	99
3	5	4	389	385	99	4	2	9	259	407	99	5	1	-7	-37	170	99	5	5	-5	871	871	99
3	5	5	653	669	99	4	2	10	723	715	99	5	1	-6	1622	1591	99	5	5	-4	377	330	99
3	5	6	241	228	99	4	2	11	167	456	99	5	1	-5	166	136	99	5	5	-3	882	910	99
3	5	7	602	642	99	4	2	12	419	432	99	5	1	-4	2401	2414	99	5	5	-2	562	554	99
3	5	8	309	225	99	4	2	13	-438	155	99	5	1	-3	589	562	99	5	5	-1	1153	1072	99
3	5	9	-47	499	99	4	4	-12	83	357	99	5	1	-2	2297	2112	99	5	5	0	425	371	99
3	5	10	193	90	99	4	4	-11	593	566	99	5	1	-1	1106	1087	99	5	5	1	978	918	99
3	7	-5	429	464	99	4	4	-10	315	310	99	5	1	0	2641	2915	99	5	5	2	373	302	99
3	7	-4	149	167	99	4	4	-9	640	559	99	5	1	1	630	696	99	5	5	3	1358	1223	99
3	7	-3	544	598	99	4	4	-8	428	458	99	5	1	2	2294	2384	99	5	5	4	402	333	99
3	7	-2	293	230	99	4	4	-7	894	861	99	5	1	3	323	338	99	5	5	5	702	772	99
3	7	-1	474	547	99	4	4	-6	524	594	99	5	1	4	1763	1777	99	5	5	6	-171	185	99
3	7	0	135	87	99	4	4	-5	1050	1035	99	5	1	5	833	870	99	5	5	7	608	685	99
3	7	1	402	404	99	4	4	-4	255	233	99	5	1	6	1720	1698	99	5	5	8	142	213	99
3	7	2	66	153	99	4	4	-3	1104	1057	99	5	1	7	213	260	99	5	5	9	402	434	99
3	7	3	400	411	99	4	4	-2	462	472	99	5	1	8	989	921	99	5	5	10	36	125	99
3	7	4	-235	144	99	4	4	-1	1387	1331	99	5	1	9	229	321	99	5	7	-5	351	446	99
3	7	5	375	402	99	4	4	0	872	831	99	5	1	10	1145	1041	99	5	7	-4	139	97	99
4	0	-14	653	634	99	4	4	1	1381	1301	99	5	1	11	296	266	99	5	7	-3	502	611	99
4	0	-12	257	474	99	4	4	2	867	815	99	5	1	12	692	598	99	5	7	-2	283	254	99
4	0	-10	1182	1195	99	4	4	3	1263	1227	99	5	1	13	-489	41	99	5	7	-1	544	603	99
4	0	-8	1869	1882	99	4	4	4	779	698	99	5	1	14	-318	337	99	5	7	0	186	140	99
4	0	-6	1999	2048	99	4	4	5	924	939	99	5	3	-13	392	330	99	5	7	1	430	463	99
4	0	-4	3008	3006	99	4	4	6	569	457	99	5	3	-12	571	513	99	5	7	2	-211	154	99
4	0	-2	2485	2437	99	4	4	7	829	833	99	5	3	-11	551	430	99	5	7	3	351	381	99
4	0	2	4464	4801	99	4	4	8	529	525	99	5	3	-10	232	388	99	5	7	4	130	104	99
4	0	4	3222	3295	99	4	4	9	610	569	99	5	3	-9	664	663	99	5	7	5	337	335	99
4	0	6	2172	2161	99	4	4	10	242	166	99	5	3	-8	830	858	99	6	0	-14	577	586	99
4	0	8	572	566	99	4	4	11	418	330	99	5	3	-7	710	644	99	6	0	-12	501	581	99
4	0	10	1120	1133	99	4	4	12	-189	215	99	5	3	-6	1304	1343	99	6	0	-10	1281	1266	99
4	0	12	748	675	99	4	6	-9	270	325	99	5	3	-5	1280	1238	99	6	0	-8	1618	1676	99
4	0	14	517	355	99	4	6	-8	-240	62	99	5	3	-4	1218	1170	99	6	0	-6	2079	2070	99
4	2	-14	215	342	99	4	6	-7	551	579	99	5	3	-3	1263	1252	99	6	0	-4	3246	3201	99
4	2	-13	279	187	99	4	6	-6	-117	28	99	5	3	-2	1587	1545	99	6	0	-2	2769	2720	99
4	2	-12	676	659	99	4	6	-5	485	512	99	5	3	-1	1455	1368	99	6	0	0	2616	2682	99
4	2	-11	355	248	99	4	6	-4	-109	179	99	5	3	0	1684	1592	99	6	0	2	3172	3052	99
4	2	-10	743	723	99	4	6	-3	510	589	99	5	3	1	1339	1252	99	6	0	4	3205	3227	99
6	0	6	2182	2157	99																		
6	0	8	831	854	99																		
6	0	10	1080	1008	99																		
6	0	12	615	587	99																		
6	0	14	-796	343	99																		
6	2	-14	371	382	99																		
6	2	-13	144	224	99																		
6	2	-12	756	670	99																		
6	2	-11	121	338	99																		
6	2	-10	768	787	99																		
6	2	-9	941	915	99																		
6	2	-8	1018	1080	99																		
6	2	-7	550	550	99																		
6	2	-6	1278	1331	99																		
6	2	-5	1212	1161	99																		
6	2	-4	1492	1520	99																		
6	2	-3	1878	1788	99																		
6	2	-2	2667	2475	99																		
6	2	-1	1356	1340	99																		
6	2	0	2429	2361	99																		
6	2	1	1814	1837	99																		
6	2	2	1971	1983	99																		
6	2	3	1166	1171	99																		
6	2	4	1136	1122	99																		
6	2	5	1312	1324	99																		
6	2	6	1392	1313	99																		
6	2	7	510	494	99																		
6	2	8	1205	1149	99																		
6	2	9	523	541	99																		
6	2	10	814	691	99																		
6	2	11	391	445	99																		
6	2	12	348	479	99																		
6	2	13	209	84	99																		
6	4	-12	271	299	99																		
6	4	-11	508	547	99																		
6	4	-10	401	295	99																		
6	4	-9	425	609	99																		
6	4	-8	437	504	99																		
6	4	-7	875	845	99																		
6	4	-6	653	657	99																		
6	4	-5	1097	1060	99																		
6	4	-4	178	224	99																		
6	4	-3	1042	1035	99																		
6	4	-2	245	291	99																		
6	4	-1	1426	1368	99																		
6	4	0	541	575	99																		
6	4	1	1315	1201	99																		
6	4	2	726	651	99																		
6	4	3	1194	1217	99																		
6	4	4	744	699	99																		
6	4	5	956	917	99																		
6	4	6	581	471	99																		
6	4	7	758	695	99																		
6	4	8	622	603	99																		
6	4	9	571	633	99																		

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
6	4	10	379	188	99	7	3	-6	1335	1366	99	8	0	-6	2092	2052	99	8	4	5	872	959	99	9	3	-10	420	493	99
6	4	11	314	333	99	7	3	-5	1062	1025	99	8	0	-4	3749	3757	99	8	4	6	529	480	99	9	3	-9	644	681	99
6	6	-9	241	313	99	7	3	-4	937	928	99	8	0	-2	3149	3201	99	8	4	7	716	765	99	9	3	-8	662	726	99
6	6	-8	-44	102	99	7	3	-3	1322	1270	99	8	0	0	1792	1822	99	8	4	8	659	493	99	9	3	-7	535	464	99
6	6	-7	506	511	99	7	3	-2	1815	1722	99	8	0	2	2738	2874	99	8	4	9	524	518	99	9	3	-6	1308	1363	99
6	6	-6	231	122	99	7	3	-1	1256	1131	99	8	0	4	2246	2299	99	8	4	10	436	224	99	9	3	-5	811	755	99
6	6	-5	551	542	99	7	3	0	1645	1521	99	8	0	6	2145	2055	99	8	4	11	468	319	99	9	3	-4	1012	969	99
6	6	-4	-45	194	99	7	3	1	1396	1341	99	8	0	8	1318	1236	99	8	5	-8	-75	78	99	9	3	-3	1094	1066	99
6	6	-3	524	540	99	7	3	2	1425	1304	99	8	0	10	1001	882	99	8	5	-7	427	464	99	9	3	-2	1718	1637	99
6	6	-2	116	90	99	7	3	3	818	859	99	8	0	12	641	470	99	8	5	-6	225	162	99	9	3	-1	1105	1052	99
6	6	-1	637	630	99	7	3	4	1137	1161	99	8	2	-14	269	396	99	8	5	-5	508	510	99	9	3	0	1496	1462	99
6	6	0	175	85	99	7	3	5	693	747	99	8	2	-13	306	324	99	8	6	-4	168	130	99	9	3	1	1484	1392	99
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23	5	2	-626	158	99
23	5	3	489	334	99
23	5	4	-483	84	99
23	5	5	-356	408	99
24	0	-12	719	723	99
24	0	-10	660	684	99
24	0	-8	1147	1081	99
24	0	-6	1269	1206	99
24	0	-4	1362	1324	99
24	0	-2	1197	1176	99
24	0	0	768	852	99
24	0	2	1072	1051	99
24	0	4	-276	612	99
24	0	6	-346	563	99
24	0	8	631	587	99
24	2	-12	322	408	99
24	2	-11	422	429	99
24	2	-10	303	496	99
24	2	-9	231	357	99
24	2	-8	662	589	99
24	2	-7	576	505	99
24	2	-6	657	637	99
24	2	-5	517	541	99
24	2	-4	659	594	99
24	2	-3	617	636	99
24	2	-2	864	939	99
24	2	-1	310	410	99
24	2	0	812	892	99
24	2	1	294	414	99
24	2	2	740	805	99
24	2	3	401	449	99
24	2	4	483	636	99
24	2	5	247	196	99
24	2	6	240	474	99
24	2	7	538	266	99
24	2	8	-657	300	99
24	2	9	398	155	99
24	4	-9	454	441	99
24	4	-8	200	196	99
24	4	-7	553	519	99
24	4	-6	320	264	99
24	4	-5	537	536	99
24	4	-4	-472	319	99
24	4	-3	498	498	99
24	4	-2	139	210	99
24	4	-1	538	631	99
24	4	0	432	273	99
24	4	1	166	546	99
24	4	2	555	263	99
24	4	3	-574	446	99
24	4	4	-439	358	99
24	4	5	505	425	99
24	4	6	-497	296	99
25	1	-12	399	419	99
25	1	-11	248	116	99
25	1	-10	467	414	99
25	1	-9	-491	78	99
25	1	-8	784	628	99
25	1	-7	329	335	99
25	1	-6	965	898	99
25	1	-5	536	576	99
25	1	-4	969	891	99
25	1	-3	-175	397	99
25	1	-2	958	976	99
25	1	-1	-118	74	99
25	1	0	800	880	99
25	1	1	304	231	99
25	1	2	966	968	99
25	1	3	-526	353	99
25	1	4	-129	607	99
25	1	5	-486	181	99
25	1	6	510	499	99
25	1	7	-712	225	99
25	1	8	-857	478	99
25	1	9	-694	87	99
25	3	-10	461	459	99
25	3	-9	224	324	99
25	3	-8	496	515	99
25	3	-7	410	336	99
25	3	-6	473	453	99
25	3	-5	427	321	99
25	3	-4	724	655	99
25	3	-3	570	513	99
25	3	-2	634	696	99
25	3	-1	113	610	99
25	3	0	504	498	99
25	3	1	479	606	99
25	3	2	-226	343	99
25	3	3	509	438	99
25	3	4	-579	417	99
25	3	5	421	324	99
25	3	6	-379	317	99
25	3	7	447	279	99
25	5	-6	143	193	99
25	5	-5	500	547	99
25	5	-4	-424	236	99
25	5	-3	438	488	99
25	5	-2	228	157	99
25	5	-1	396	449	99
25	5	0	-458	202	99
25	5	1	50	342	99
25	5	2	151	154	99
25	5	3	374	276	99
26	0	-12	728	686	99
26	0	-10	611	514	99
26	0	-8	926	843	99
26	0	-6	1271	1230	99
26	0	-4	722	750	99
26	0	-2	995	980	99
26	0	0	730	743	99
26	0	2	-470	733	99
26	0	4	690	602	99
26	0	6	-878	502	99
26	0	8	499	523	99
26	2	-11	-306	404	99
26	2	-10	623	543	99
26	2	-9	-177	300	99
26	2	-8	615	584	99
26	2	-7	611	597	99
26	2	-6	326	526	99
26	2	-5	209	374	99
26	2	-4	687	591	99
26	2	-3	325	359	99
26	2	-2	630	750	99
26	2	-1	485	556	99
26	2	0	645	718	99
26	2	1	-461	382	99
26	2	2	684	701	99
26	2	3	433	293	99
26	2	4	394	614	99
26	2	5	397	181	99
26	2	6	266	368	99
26	2	7	436	245	99
26	2	8	-439	255	99
26	4	-8	246	121	99
26	4	-7	365	407	99
26	4	-6	-149	194	99
26	4	-5	487	497	99
26	4	-4	-67	409	99
26	4	-3	457	500	99
26	4	-2	-130	246	99
26	4	-1	-418	440	99
26	4	0	334	217	99
26	4	1	607	443	99
26	4	2	348	224	99
26	4	3	436	389	99
26	4	4	-656	246	99
26	4	5	-637	386	99
27	1	-11	229	84	99
27	1	-10	163	309	99
27	1	-9	186	156	99
27	1	-8	516	541	99
27	1	-7	-327	222	99
27	1	-6	808	772	99
27	1	-5	195	289	99
27	1	-4	731	692	99
27	1	-3	638	543	99
27	1	-2	874	927	99
27	1	-1	-363	282	99
27	1	0	752	748	99
27	1	1	279	289	99
27	1	2	758	785	99
27	1	3	240	121	99
27	1	4	548	593	99
27	1	5	-1145	92	99
27	1	6	-559	417	99
27	1	7	341	174	99
27	1	8	-1152	374	99
27	3	-10	307	469	99
27	3	-9	418	378	99
27	3	-8	467	382	99
27	3	-7	368	398	99
27	3	-6	-404	410	99
27	3	-5	201	314	99
27	3	-4	574	634	99
27	3	-3	431	380	99
36	2	-2	195	345	99
37	1	-3	-276	119	99
37	1	-2	222	309	99

APPENDIX 7.III

$[Hg(S_2CO\text{-}3,3\text{-}Me_2Bu)_2]$

Table 7.III.1: Crystallographic Parameters for $[Hg(S_2COCH_2CH_2C(CH_3)_3)_2]$

Formula	$C_{14}H_{26}HgS_4O_2$
Formula weight	531.17
Crystal morphology / colour	block / pale yellow
Crystal dimensions (mm)	0.13 x 0.26 x 0.27
Crystal system	monoclinic
Space group	$P2_1/c$ (# 14)
a (Å)	30.045(4)
b (Å)	5.955(8)
c (Å)	11.712(2)
α (°)	90
β (°)	97.07(2)
γ (°)	90
V (Å^3)	2079(1)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.773
$F(000)$	1032
Temperature (°K)	293
μ (cm^{-1})	78.19
θ limits, cell (°)	18.2 – 21.3
θ limits, data (°)	3.0 – 27.5
hkl range	-36 – 36, -7 – 0, 0 – 15
Range of transmission factors	0.910 – 1
Scan technique	ω:2θ
No. of data measured	5536
No. of unique data	5290
R_{amal}	0.095
No. of parameters	185
R (I≥0σ(I))	0.082
R_w (I≥0σ(I))	0.091
R (I≥3σ(I))	0.041
R_w (I≥3σ(I))	0.036
Maximum and minimum residual electron density (e Å^{-3})	2.81, -3.11

Table 7.III.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Hg(S_2COCH_2CH_2C(CH_3)_3)_2]$

Atom	*x*	*y*	*z*
Hg	0.24984(1)	0.23726(6)	0.15132(3)
S(1)	0.2414(1)	-0.1800(4)	0.1315(2)
S(2)	0.19162(9)	-0.5357(4)	0.2392(2)
S(3)	0.26567(9)	0.3652(4)	-0.0457(2)
S(4)	0.32066(9)	0.1567(5)	-0.2109(2)
O(1)	0.1803(2)	-0.103(1)	0.2598(5)
O(2)	0.3231(2)	0.056(1)	0.0078(5)
C(1)	0.2033(3)	-0.264(1)	0.2148(7)
C(2)	0.1467(4)	-0.160(2)	0.336(1)
C(3)	0.1224(5)	0.043(2)	0.358(1)
C(4)	0.0806(5)	0.031(2)	0.404(1)
C(5)	0.0679(7)	0.261(2)	0.440(2)
C(6)	0.0831(5)	-0.116(3)	0.508(1)
C(7)	0.0436(5)	-0.071(4)	0.329(2)
C(8)	0.3039(3)	0.181(1)	-0.0797(7)
C(9)	0.3572(3)	-0.111(2)	-0.0139(8)
C(10)	0.3787(3)	-0.186(2)	0.1048(8)
C(11)	0.4218(3)	-0.322(2)	0.1028(9)
C(12)	0.4594(4)	-0.180(2)	0.0642(9)
C(13)	0.4130(4)	-0.520(2)	0.022(1)
C(14)	0.4371(4)	-0.407(3)	0.222(1)

Table 7.III.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Hg(S_2COCH_2CH_2C(CH_3)3)2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Hg	0.0500(2)	0.0490(2)	0.0485(2)	0.0006(3)	0.0063(1)	0.0019(2)
S(1)	0.090(2)	0.043(1)	0.088(2)	-0.006(1)	0.051(2)	-0.007(1)
S(2)	0.057(2)	0.046(1)	0.101(2)	0.003(1)	0.035(2)	0.007(1)
S(3)	0.066(2)	0.061(1)	0.042(1)	0.025(1)	0.006(1)	0.003(1)
S(4)	0.052(2)	0.127(2)	0.041(1)	0.029(2)	0.004(1)	-0.004(1)
O(1)	0.056(5)	0.050(4)	0.081(4)	0.011(3)	0.027(4)	0.002(3)
O(2)	0.046(4)	0.067(4)	0.049(3)	0.012(3)	0.011(3)	0.004(3)
C(1)	0.050(5)	0.051(5)	0.059(5)	0.012(6)	0.008(4)	-0.007(5)
C(2)	0.084(9)	0.076(7)	0.133(10)	0.013(7)	0.078(8)	-0.003(7)
C(3)	0.14(1)	0.11(1)	0.26(2)	0.01(1)	0.15(1)	-0.01(1)
C(4)	0.069(9)	0.10(1)	0.14(1)	0.014(8)	0.043(9)	0.014(9)
C(5)	0.26(2)	0.07(1)	0.48(4)	0.03(1)	0.24(2)	0.00(2)
C(6)	0.15(2)	0.15(1)	0.18(2)	-0.01(1)	0.12(1)	0.00(1)
C(7)	0.08(1)	0.29(3)	0.23(2)	0.00(2)	0.01(1)	-0.06(2)
C(8)	0.035(5)	0.064(6)	0.044(5)	-0.012(4)	-0.001(4)	0.002(4)
C(9)	0.069(8)	0.081(7)	0.066(6)	0.031(6)	0.011(5)	0.003(6)
C(10)	0.060(7)	0.078(7)	0.072(6)	0.016(6)	0.016(5)	0.023(5)
C(11)	0.052(7)	0.071(7)	0.080(7)	-0.001(6)	0.007(5)	0.018(5)
C(12)	0.060(7)	0.087(8)	0.097(8)	-0.008(6)	-0.002(6)	0.022(6)
C(13)	0.061(9)	0.076(8)	0.21(1)	0.014(7)	0.004(9)	-0.015(9)
C(14)	0.071(9)	0.20(2)	0.11(1)	0.05(1)	0.017(8)	0.07(1)

Table 7.III.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Hg(S_2COCH_2CH_2C(CH_3)_3)_2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.1261	-0.2711	0.2992	8.7
H(2b)	0.1614	-0.2196	0.4079	8.7
H(3a)	0.1166	0.1203	0.2846	15.3
H(3b)	0.1424	0.1319	0.4107	15.3
H(5a)	0.0913	0.3208	0.4929	23.9
H(5b)	0.0632	0.3550	0.3739	23.9
H(5c)	0.0411	0.2522	0.4748	23.9
H(6a)	0.0553	-0.1117	0.5389	14.7
H(6b)	0.0892	-0.2663	0.4872	14.7
H(6c)	0.1063	-0.0650	0.5645	14.7
H(7a)	0.0175	-0.0743	0.3673	18.7
H(7b)	0.0378	0.0134	0.2601	18.7
H(7c)	0.0515	-0.2208	0.3109	18.7
H(9a)	0.3433	-0.2378	-0.0566	6.8
H(9b)	0.3795	-0.0443	-0.0567	6.8
H(10a)	0.3856	-0.0538	0.1517	6.6
H(10b)	0.3573	-0.2785	0.1391	6.6
H(12a)	0.4858	-0.2730	0.0618	7.8
H(12b)	0.4500	-0.1197	-0.0118	7.8
H(12c)	0.4664	-0.0577	0.1180	7.8
H(13a)	0.3907	-0.6175	0.0489	11.0
H(13b)	0.4021	-0.4661	-0.0548	11.0
H(13c)	0.4406	-0.6031	0.0192	11.0
H(14a)	0.4136	-0.4983	0.2488	12.1

Table 7.III.4 Cont.

Atom	x	y	z	B_{eq}
H(14b)	0.4639	-0.4978	0.2212	12.1
H(14c)	0.4437	-0.2813	0.2742	12.1

* H atoms were included in their calculated positions, C-H 0.97Å.

Table 7.III.5: Bond distances (Å) for $[Hg(S_2COCH_2CH_2C(CH_3)_3)_2]$

Hg — S(1)	2.506(2)	O(2) — C(9)	1.47(1)
Hg — S(2)*	2.528(3)	C(2) — C(3)	1.45(2)
Hg — S(3)	2.530(2)	C(3) — C(4)	1.43(2)
Hg — S(4)**	2.586(3)	C(4) — C(5)	1.49(2)
S(1) — C(1)	1.670(9)	C(4) — C(6)	1.50(2)
S(2) — C(1)	1.685(9)	C(4) — C(7)	1.47(2)
S(3) — C(8)	1.673(9)	C(9) — C(10)	1.53(1)
S(4) — C(8)	1.680(8)	C(10) — C(11)	1.53(1)
O(1) — C(1)	1.328(9)	C(11) — C(12)	1.52(1)
O(1) — C(2)	1.47(1)	C(11) — C(13)	1.52(1)
O(2) — C(8)	1.338(9)	C(11) — C(14)	1.51(1)

* symmetry operation: x, $1 + y$, z. ** symmetry operation: x, $0.5 - y$, $0.5 + z$.

Table 7.III.6: Bond angles (deg.) for [Hg($S_2COCH_2CH_2C(CH_3)_3$)2]

S(1) — Hg — S(2)*	120.03(8)	C(3) — C(4) — C(5)	109(1)
S(1) — Hg — S(3)	104.02(8)	C(3) — C(4) — C(6)	112(1)
S(1) — Hg — S(4)**	111.5(1)	C(3) — C(4) — C(7)	115(1)
S(2)* — Hg — S(3)	115.31(8)	C(5) — C(4) — C(6)	107(1)
S(2)* — Hg — S(4)**	99.94(9)	C(5) — C(4) — C(7)	110(2)
S(3) — Hg — S(4)**	105.27(8)	C(6) — C(4) — C(7)	102(1)
Hg — S(1) — C(1)	108.3(3)	S(3) — C(8) — S(4)	124.4(5)
Hg — S(2)* — C(1)*	106.0(3)	S(3) — C(8) — O(2)	115.2(6)
Hg — S(3) — C(8)	103.4(3)	S(4) — C(8) — O(2)	120.3(6)
Hg — S(4)** — C(8)**	103.6(3)	O(2) — C(9) — C(10)	105.4(7)
C(1) — O(1) — C(2)	120.3(7)	C(9) — C(10) — C(11)	113.9(8)
C(8) — O(2) — C(9)	119.1(7)	C(10) — C(11) — C(12)	112.0(8)
S(1) — C(1) — S(2)	124.0(5)	C(10) — C(11) — C(13)	109.8(9)
S(1) — C(1) — O(1)	116.2(7)	C(10) — C(11) — C(14)	109.1(9)
S(2) — C(1) — O(1)	119.8(6)	C(12) — C(11) — C(13)	108.8(9)
O(1) — C(2) — C(3)	108.3(9)	C(12) — C(11) — C(14)	108.0(9)
C(2) — C(3) — C(4)	121(1)	C(13) — C(11) — C(14)	109(1)

* symmetry operation: x, $1 + y$, z. ** symmetry operation: x, $0.5 - y$, $0.5 + z$.

Table 7.III.7: Observed and calculated structure factors for $[Hg(S_2COCH_2CH_2C(CH_3)_3)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	-1	1	295	289	99	0	-4	7	124	76	99	1	-5	3	223	213	99	-1	0	8	1449	1426	99	2	-6	9	27	80	99
0	-1	2	1166	1179	99	0	-4	6	755	771	99	1	-5	4	712	702	99	-1	0	6	1710	1744	99	2	-5	0	150	165	99
0	-1	3	67	106	99	0	-4	5	115	116	99	1	-5	5	530	557	99	-1	0	4	1967	1913	99	2	-5	1	319	323	99
0	-1	4	1768	1736	99	0	-4	4	929	928	99	1	-5	6	562	539	99	-1	0	2	2017	1996	99	2	-5	2	1197	1167	99
0	-1	5	49	8	99	0	-4	3	220	225	99	1	-5	7	180	43	99	-1	-1	15	114	51	99	2	-5	3	328	307	99
0	-1	6	1193	1192	99	0	-4	2	597	604	99	1	-5	8	325	309	99	-1	-1	14	344	317	99	2	-5	4	425	458	99
0	-1	7	302	334	99	0	-4	1	299	277	99	1	-5	9	116	132	99	-1	-1	13	133	34	99	2	-5	5	148	108	99
0	-1	8	1455	1480	99	0	-4	0	1249	1202	99	1	-5	10	356	352	99	-1	-1	12	220	181	99	2	-5	6	300	233	99
0	-1	9	94	120	99	0	-5	11	120	115	99	1	-5	11	-47	102	99	-1	-1	11	144	85	99	2	-5	7	283	311	99
0	-1	10	225	219	99	0	-5	10	147	31	99	1	-4	0	165	129	99	-1	-1	10	1008	960	99	2	-5	8	562	532	99
0	-1	11	91	97	99	0	-5	9	102	148	99	1	-4	1	167	198	99	-1	-1	9	-109	126	99	2	-5	9	48	146	99
0	-1	12	486	501	99	0	-5	8	478	498	99	1	-4	2	1398	1399	99	-1	-1	8	72	144	99	2	-5	10	-155	49	99
0	-1	13	145	83	99	0	-5	7	210	231	99	1	-4	3	509	528	99	-1	-1	7	237	227	99	2	-5	11	-163	91	99
0	-1	14	344	342	99	0	-5	6	261	283	99	1	-4	4	715	711	99	-1	-1	6	1370	1348	99	2	-4	0	1164	1136	99
0	-1	15	109	63	99	0	-5	5	96	58	99	1	-4	5	252	235	99	-1	-1	5	514	513	99	2	-4	1	253	270	99
0	-2	14	287	292	99	0	-5	4	385	406	99	1	-4	6	183	191	99	-1	-1	4	1169	1176	99	2	-4	2	673	697	99
0	-2	13	123	32	99	0	-5	3	425	421	99	1	-4	7	120	64	99	-1	-1	3	1032	1055	99	2	-4	3	-91	126	99
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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-27	-1	11	73	141	99	-28	-1	8	555	539	99	-29	-1	3	125	154	99	-30	-2	3	169	121	99	-31	-4	2	219	366	99
-27	-1	10	205	236	99	-28	-1	7	133	114	99	-29	-1	2	303	332	99	-30	-2	2	267	303	99	-31	-4	1	172	124	99
-27	-1	9	-135	150	99	-28	-1	6	379	322	99	-29	-1	1	-110	88	99	-30	-2	1	-169	52	99	-32	0	10	406	407	99
-27	-1	8	134	91	99	-28	-1	5	-237	95	99	-29	-2	10	210	90	99	-30	-3	9	157	50	99	-32	0	8	184	155	99
-27	-1	7	154	138	99	-28	-1	4	275	291	99	-29	-2	9	-104	16	99	-30	-3	8	366	341	99	-32	0	6	315	366	99
-27	-1	6	588	568	99	-28	-1	3	221	98	99	-29	-2	8	547	545	99	-30	-3	7	-204	27	99	-32	0	4	320	337	99
-27	-1	5	-217	116	99	-28	-1	2	704	748	99	-29	-2	7	-56	33	99	-30	-3	6	273	254	99	-32	0	2	382	379	99
-27	-1	4	752	766	99	-28	-1	1	-78	126	99	-29	-2	6	402	358	99	-30	-3	5	259	190	99	-32	-1	10	-173	20	99
-27	-1	3	-51	197	99	-28	-2	11	123	50	99	-29	-2	4	358	327	99	-30	-3	4	212	277	99	-32	-1	9	-288	127	99
-27	-1	2	-145	229	99	-28	-2	10	514	490	99	-29	-2	3	171	20	99	-30	-3	3	92	138	99	-32	-1	8	331	317	99
-27	-1	1	-55	198	99	-28	-2	9	171	32	99	-29	-2	4	248	279	99	-30	-3	2	314	418	99	-32	-1	7	-40	68	99
-32	-1	6	78	213	99	-33	-3	1	-102	145	99	-36	0	4	248	279	99												
-32	-1	5	249	112	99	-33	-4	3	-170	33	99	-36	0	2	143	151	99												
-32	-1	4	233	353	99	-33	-4	2	199	257	99	-36	-1	7	82	31	99												
-32	-1	3	276	63	99	-33	-4	1	-102	83	99	-36	-1	6	-155	136	99												
-32	-1	2	612	674	99	-34	0	8	141	123	99	-36	-1	5	165	7	99												
-32	-1	1	-111	86	99	-34	0	6	370	301	99	-36	-1	4	140	216	99												
-32	-2	9	-232	30	99	-34	0	4	354	340	99	-36	-1	3	-118	74	99												
-32	-2	8	-99	59	99	-34	0	2	275	263	99	-36	-1	2	142	383	99												
-32	-2	7	130	9	99	-34	-1	8	-133	235	99	-36	-1	1	59	25	99												
-32	-2	6	328	384	99	-34	-1	7	84	43	99	-36	-2	6	296	368	99												
-32	-2	5	90	199	99	-34	-1	6	112	155	99	-36	-2	5	193	65	99												
-32	-2	4	332	424	99	-34	-1	5	267	56	99	-36	-2	4	256	336	99												
-32	-2	3	88	35	99	-34	-1	4	253	310	99	-36	-2	3	64	13	99												
-32	-2	2	107	131	99	-34	-1	3	-271	63	99	-36	-2	2	182	89	99												
-32	-2	1	-56	13	99	-34	-1	2	487	525	99	-36	-2	1	-153	57	99												
-32	-3	8	305	307	99	-34	-1	1	151	32	99	-36	-3	2	230	226	99												
-32	-3	7	248	26	99	-34	-2	8	-120	15	99	-36	-3	1	-166	52	99												
-32	-3	6	157	210	99	-34	-2	7	-178	25	99	-37	0	6	178	123	99												
-32	-3	5	-241	94	99	-34	-2	6	422	403	99	-37	0	4	-132	209	99												
-32	-3	4	135	231	99	-34	-2	5	178	199	99	-37	0	2	250	350	99												
-32	-3	3	-200	68	99	-34	-2	4	93	362	99	-37	-1	6	187	226	99												
-32	-3	2	274	342	99	-34	-2	3	150	9	99	-37	-1	5	245	81	99												
-32	-3	1	173	61	99	-34	-2	2	-115	45	99	-37	-1	4	290	313	99												
-32	-4	5	-12	79	99	-34	-2	1	-107	58	99	-37	-1	3	-67	2	99												
-32	-4	4	177	231	99	-34	-3	6	164	186	99	-37	-1	2	-146	85	99												
-32	-4	3	-81	47	99	-34	-3	5	176	42	99	-37	-1	1	125	9	99												
-32	-4	2	163	63	99	-34	-3	4	-165	203	99	-37	-2	4	255	251	99												
-32	-4	1	-209	155	99	-34	-3	3	-294	28	99	-37	-2	3	-37	44	99												
-33	0	8	427	387	99	-34	-3	2	145	290	99	-37	-2	2	290	298	99												
-33	0	6	113	107	99	-34	-3	1	72	55	99	-37	-2	1	140	85	99												
-33	0	4	431	471	99	-35	0	8	375	370	99	-38	0	4	282	259	99												
-33	0	2	633	644	99	-35	0	6	203	103	99	-38	0	2	232	126	99												
-33	-1	9	90	36	99	-35	0	4	324	336	99	-38	-1	4	-297	132	99												
-33	-1	8	79	175	99	-35	0	2	431	474	99	-38	-1	3	-205	87	99												
-33	-1	7	-144	67	99	-35	-1	8	202	135	99	-38	-1	2	244	266	99												
-33	-1	6	342	291	99	-35	-1	7	54	107	99	-38	-1	1	-162	97	99												
-33	-1	5	171	180	99	-35	-1	6	-113	238	99	-39	0	2	167	231	99												
-33	-1	4	318	338	99	-35	-1	5	-178	139	99																		
-33	-1	3	145	53	99	-35	-1	4	291	350	99																		
-33	-1	2	193	204	99	-35	-1	3	174	25	99																		
-33	-1	1	121	51	99	-35	-1	2	-158	149	99																		
-33	-2	8	387	410	99	-35	-1	1	-116	30	99																		
-33	-2	7	111	34	99	-35	-2	7	-282	25	99																		
-33	-2	6	-7	213	99	-35	-2	6	165	160	99																		
-33	-2	5	-168	74	99	-35	-2	5	215	115	99																		
-33	-2	4	249	286	99	-35	-2	4	-194	282	99																		
-33	-2	3	-152	20	99	-35	-2	3	-261	12	99																		
-33	-2	2	315	436	99	-35	-2	2	303	360	99																		
-33	-2	1	-163	59	99	-35	-2	1	85	88	99																		
-33	-3	7	-115	29	99	-35	-3	5	141	50	99																		
-33	-3	6	165	308	99	-35	-3	4	163	282	99																		
-33	-3	5	148	99	99	-35	-3	3	-111	128	99																		
-33	-3	4	283	343	99	-35	-3	2	180	38	99																		
-33	-3	3	22	43	99	-35	-3	1	102	126	99																		
-33	-3	2	-295	69	99	-36	0	6	184	298	99																		

APPENDIX 7.IV

$[Hg(S_2COnPent)_2]$

Table 7.IV.1: Crystallographic Parameters for [Hg($S_2CO(CH_2CH_2CH_2CH_2CH_3)_2$]

Formula	$C_{12}H_{22}HgO_2S_4$
Formula weight	441.00
Crystal morphology / colour	needle / pale yellow
Crystal dimensions (mm)	0.08 x 0.15 x 0.42
Crystal system	monoclinic
Space group	*Cc* (# 9)
a (Å)	27.32(1)
b (Å)	5.854(7)
c (Å)	11.384(7)
α (°)	90
β (°)	95.35(5)
γ (°)	90
V (Å^3)	1813(2)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.931
F(000)	1016
Temperature (°K)	113
μ (cm^{-1})	89.25
θ limits, cell (°)	26.5 – 27.5
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 35, 0 – 7, -14 – 14
Range of transmission factors	0.933 – 1
Scan technique	ω:2θ
No. of data measured	2351
No. of unique data	2303
R_{amal}	0.185
No. of parameters	172
R (I≥0σ(I))	0.053
R_w (I≥0σ(I))	0.069
R (I≥3σ(I))	0.029
R_w (I≥3σ(I))	0.034
Maximum and minimum residual electron density (e Å^{-3})	1.48, -1.38

Table 7.IV.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Hg(S_2COCH_2CH_2CH_2CH_2CH_3)_2]$

Atom	*x*	*y*	*z*
Hg	0	0.05048(6)	0
S(1)	-0.0671(1)	0.2666(4)	0.0903(2)
S(2)	-0.0131(1)	0.6254(4)	-0.0295(2)
S(3)	0.02100(10)	0.1430(5)	-0.2089(2)
S(4)	0.0740(1)	-0.1780(5)	-0.3591(2)
O(1)	-0.0774(3)	0.709(1)	0.1164(6)
O(2)	0.0850(3)	-0.149(1)	-0.1287(6)
C(1)	-0.0540(4)	0.542(2)	0.0632(8)
C(2)	-0.1116(4)	0.651(2)	0.2049(9)
C(3)	-0.1255(4)	0.874(2)	0.2573(9)
C(4)	-0.1658(4)	0.852(2)	0.3403(9)
C(5)	-0.1752(4)	1.074(2)	0.402(1)
C(6)	-0.2180(5)	1.058(3)	0.481(1)
C(7)	0.0616(3)	-0.073(2)	-0.2272(8)
C(8)	0.1174(4)	-0.347(2)	-0.131(1)
C(9)	0.1393(4)	-0.387(2)	-0.005(1)
C(10)	0.1734(4)	-0.194(2)	0.043(1)
C(11)	0.1912(4)	-0.221(2)	0.172(1)
C(12)	0.2265(5)	-0.038(3)	0.219(1)

Table 7.IV.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Hg(S_2COCH_2CH_2CH_2CH_2CH_3)_2]$

Atom	$U11$	$U22$	$U33$	$U12$	$U13$	$U23$
Hg	0.0213(2)	0.0211(1)	0.0211(1)	0.0018(3)	0.0020(1)	0.0000(3)
S(1)	0.024(1)	0.016(1)	0.035(1)	-0.001(1)	0.009(1)	-0.002(1)
S(2)	0.029(2)	0.017(1)	0.027(1)	-0.0018(10)	0.0131(9)	-0.0012(10)
S(3)	0.024(1)	0.024(1)	0.0166(9)	0.004(1)	0.0000(10)	0.0015(10)
S(4)	0.021(1)	0.045(2)	0.020(1)	0.008(1)	0.0011(10)	-0.008(1)
O(1)	0.017(4)	0.021(4)	0.027(3)	0.003(3)	0.004(3)	0.000(3)
O(2)	0.012(4)	0.030(4)	0.024(3)	0.008(3)	-0.005(3)	0.001(3)
C(1)	0.014(4)	0.013(4)	0.027(4)	0.001(4)	-0.003(3)	-0.003(4)
C(2)	0.009(5)	0.033(5)	0.023(5)	0.000(4)	-0.001(4)	0.004(5)
C(3)	0.013(5)	0.029(5)	0.027(5)	-0.006(4)	0.003(4)	-0.002(4)
C(4)	0.008(5)	0.038(6)	0.032(5)	-0.007(4)	-0.002(4)	-0.005(5)
C(5)	0.020(6)	0.047(8)	0.041(6)	0.000(5)	0.004(5)	-0.013(6)
C(6)	0.030(7)	0.066(10)	0.041(6)	0.000(7)	0.017(5)	-0.022(7)
C(7)	0.006(4)	0.026(5)	0.024(4)	-0.001(4)	-0.003(3)	-0.004(4)
C(8)	0.017(6)	0.027(5)	0.041(6)	0.002(5)	-0.005(5)	-0.006(5)
C(9)	0.021(6)	0.033(6)	0.037(6)	0.001(5)	-0.003(5)	0.003(5)
C(10)	0.014(5)	0.032(6)	0.044(6)	0.004(5)	-0.004(5)	0.012(5)
C(11)	0.018(6)	0.042(7)	0.042(6)	0.005(5)	-0.007(5)	0.008(6)
C(12)	0.029(7)	0.063(9)	0.043(7)	0.002(7)	-0.014(6)	-0.011(7)

Table 7.IV.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Hg(S_2COCH_2CH_2CH_2CH_2CH_3)_2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	-0.1400	0.5771	0.1683	2.0465
H(2b)	-0.0960	0.5547	0.2640	2.0465
H(3a)	-0.1365	0.9746	0.1951	2.1412
H(3b)	-0.0970	0.9361	0.3004	2.1412
H(4a)	-0.1953	0.8046	0.2959	2.4562
H(4b)	-0.1563	0.7389	0.3982	2.4562
H(5a)	-0.1829	1.1885	0.3439	3.3392
H(5b)	-0.1462	1.1165	0.4496	3.3392
H(6a)	-0.2475	1.0197	0.4344	4.2526
H(6b)	-0.2109	0.9429	0.5394	4.2526
H(6c)	-0.2220	1.2005	0.5190	4.2526
H(8a)	0.0991	-0.4776	-0.1591	2.7106
H(8b)	0.1428	-0.3185	-0.1805	2.7106
H(9a)	0.1131	-0.3999	0.0447	2.8042
H(9b)	0.1575	-0.5256	-0.0017	2.8042
H(10a)	0.1562	-0.0537	0.0329	2.8559
H(10b)	0.2012	-0.1915	-0.0014	2.8559
H(11a)	0.1634	-0.2183	0.2160	3.3125
H(11b)	0.2072	-0.3647	0.1820	3.3125
H(12a)	0.2110	0.1071	0.2096	4.4082
H(12b)	0.2549	-0.0408	0.1770	4.4082
H(12c)	0.2356	-0.0648	0.3004	4.4082

* H atoms were included in their calculated positions, C-H 0.97Å.

Table 7.IV.5: Bond distances (Å) for $[Hg(S_2COCH_2CH_2CH_2CH_2CH_3)_2]$

Hg — S(1)	2.524(3)	O(2) — C(7)	1.31(1)
Hg — S(2)*	2.532(3)	O(2) — C(8)	1.46(1)
Hg — S(3)	2.555(2)	C(2) — C(3)	1.50(2)
Hg — S(4)**	2.572(3)	C(3) — C(4)	1.52(1)
S(1) — C(1)	1.69(1)	C(4) — C(5)	1.51(2)
S(2) — C(1)	1.68(1)	C(5) — C(6)	1.55(2)
S(3) — C(7)	1.71(1)	C(8) — C(9)	1.52(2)
S(4) — C(7)	1.69(1)	C(9) — C(10)	1.53(2)
O(1) — C(1)	1.34(1)	C(10) — C(11)	1.51(2)
O(1) — C(2)	1.48(1)	C(11) — C(12)	1.51(2)

* symmetry operation: x, -1 + y, z. ** symmetry operation: x, -y, 0.5 + z.

Table 7.IV.6: Bond angles (deg.) for [Hg($S_2COCH_2CH_2CH_2CH_2CH_3$)2]

S(1) — Hg — S(2)*	116.60(8)	S(1) — C(1) — O(1)	119.7(7)
S(1) — Hg — S(3)	120.50(9)	S(2) — C(1) — O(1)	116.4(7)
S(1) — Hg — S(4)**	99.15(9)	O(1) — C(2) — C(3)	105.9(8)
S(2) — Hg — S(3)	97.25(8)	C(2) — C(3) — C(4)	113.6(9)
S(2)* — Hg — S(4)**	117.45(9)	C(3) — C(4) — C(5)	112(1)
S(3) — Hg — S(4)**	106.69(8)	C(4) — C(5) — C(6)	113(1)
Hg — S(1) — C(1)	103.3(4)	S(3) — C(7) — S(4)	124.4(6)
Hg — S(2)* — C(1)*	107.2(3)	S(3) — C(7) — O(2)	114.6(7)
Hg — S(3) — C(7)	99.3(3)	S(4) — C(7) — O(2)	121.0(7)
Hg — S(4)** — C(7)**	103.8(3)	O(2) — C(8) — C(9)	106.9(9)
C(1) — O(1) — C(2)	120.0(8)	C(8) — C(9) — C(10)	113(1)
C(7) — O(2) — C(8)	120.0(8)	C(9) — C(10) — C(11)	113(1)
S(1) — C(1) — S(2)	124.0(6)	C(10) — C(11) — C(12)	114(1)

* symmetry operation: x, -1 + y, z. ** symmetry operation: x, -y, 0.5 + z.

Table 7.IV.7: Observed and calculated structure factors for $[Hg(S_2COCH_2CH_2CH_2CH_2CH_3)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	2	3154	2965	99	1	1	-4	2114	2200	99	1	5	-2	346	356	99	2	2	2	1825	1782	99	2	6	8	303	286	99
0	0	4	2621	2539	99	1	1	-3	974	952	99	1	5	-1	1420	1445	99	2	2	3	1602	1515	99	3	1	-14	896	936	99
0	0	6	2404	2306	99	1	1	-2	2329	2273	99	1	5	0	294	266	99	2	2	4	1815	1801	99	3	1	-13	-72	222	99
0	0	8	2051	2033	99	1	1	-1	1014	944	99	1	5	1	1903	1928	99	2	2	5	1882	1926	99	3	1	-12	824	875	99
0	0	10	1537	1527	99	1	1	0	2916	3061	99	1	5	2	141	3	99	2	2	6	1750	1788	99	3	1	-11	435	407	99
0	0	12	838	794	99	1	1	1	393	371	99	1	5	3	1764	1768	99	2	2	7	992	1044	99	3	1	-10	1300	1378	99
0	0	14	1047	1039	99	1	1	2	1108	1089	99	1	5	4	390	394	99	2	2	8	1463	1543	99	3	1	-9	1180	1204	99
0	2	0	1281	1278	99	1	1	3	1233	1267	99	1	5	5	1053	1001	99	2	2	9	731	741	99	3	1	-8	1679	1713	99
0	2	1	2323	2249	99	1	1	4	2192	2261	99	1	5	6	309	294	99	2	2	10	980	961	99	3	1	-7	313	302	99
0	2	2	2174	2085	99	1	1	5	943	939	99	1	5	7	1208	1164	99	2	2	11	661	669	99	3	1	-6	1719	1769	99
0	2	3	1331	1240	99	1	1	6	1744	1847	99	1	5	8	88	27	99	2	2	12	722	715	99	3	1	-5	374	370	99
0	2	4	2703	2592	99	1	1	7	436	446	99	1	5	9	977	965	99	2	2	13	373	392	99	3	1	-4	2351	2398	99
0	2	5	1895	1913	99	1	1	8	1784	1820	99	1	5	10	171	171	99	2	2	14	541	541	99	3	1	-3	784	739	99
0	2	6	1616	1652	99	1	1	9	579	583	99	1	5	11	945	914	99	2	4	-12	494	494	99	3	1	-2	2241	2212	99
0	2	7	1119	1158	99	1	1	10	1338	1340	99	1	7	-5	801	798	99	2	4	-11	1163	1137	99	3	1	-1	2169	2190	99
0	2	8	1176	1224	99	1	1	11	489	485	99	1	7	-4	822	792	99	2	4	-10	425	449	99	3	1	0	2494	2486	99
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24	4	-1	1060	1057	99
24	4	0	379	350	99
24	4	1	862	904	99
24	4	2	222	249	99
24	4	3	844	859	99
24	4	4	572	516	99
24	4	5	1002	1039	99
24	4	6	552	492	99
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25	1	-10	471	518	99
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25	1	-8	874	937	99
25	1	-7	314	383	99
25	1	-6	1097	1178	99
25	1	-5	481	512	99
25	1	-4	1173	1168	99
25	1	-3	771	764	99
25	1	-2	1419	1373	99
25	1	-1	444	457	99
25	1	0	1206	1194	99
25	1	1	519	463	99
25	1	2	1619	1589	99
25	1	3	566	614	99
25	1	4	898	959	99
25	1	5	260	381	99
25	1	6	997	1057	99
25	1	7	570	573	99
25	1	8	852	922	99
25	1	9	125	300	99
25	3	-9	880	845	99
25	3	-8	464	464	99
25	3	-7	872	843	99
25	3	-6	390	419	99
25	3	-5	780	756	99
25	3	-4	828	806	99
25	3	-3	856	845	99
25	3	-2	806	787	99
25	3	-1	1092	1092	99
25	3	0	650	635	99
25	3	1	1135	1153	99
25	3	2	355	310	99
25	3	3	1078	1107	99
25	3	4	600	607	99
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25	3	6	478	522	99
25	3	7	703	675	99
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25	5	-4	-224	19	99
25	5	-3	908	880	99
25	5	-2	-199	237	99
25	5	-1	945	1001	99
25	5	0	216	229	99
25	5	1	600	707	99
25	5	2	-182	102	99
25	5	3	513	522	99
26	0	-10	759	723	99
26	0	-8	1077	1067	99
26	0	-6	1502	1503	99
26	0	-4	898	879	99
26	0	-2	1268	1244	99
26	0	0	1172	1151	99
26	0	2	1306	1244	99
26	0	4	684	718	99
26	0	6	849	881	99
26	0	8	1067	1137	99
26	2	-10	754	768	99
26	2	-9	632	649	99
26	2	-8	865	859	99
26	2	-7	930	917	99
26	2	-6	826	800	99
26	2	-5	529	510	99
26	2	-4	940	925	99
26	2	-3	551	534	99
26	2	-2	878	865	99
26	2	-1	1038	960	99
26	2	0	692	705	99
26	2	1	571	538	99
26	2	2	1003	1036	99
26	2	3	543	567	99
26	2	4	1057	1158	99
26	2	5	112	258	99
26	2	6	856	874	99
26	2	7	581	580	99
26	2	8	499	425	99
26	4	-7	609	595	99
26	4	-6	-61	167	99
26	4	-5	906	894	99
26	4	-4	575	523	99
26	4	-3	1039	1035	99
26	4	-2	391	397	99
26	4	-1	870	917	99
26	4	0	565	543	99
26	4	1	755	760	99
26	4	2	302	178	99
26	4	3	742	738	99
26	4	4	372	330	99
26	4	5	848	871	99
27	1	-10	607	621	99
27	1	-9	182	91	99
27	1	-8	837	842	99
27	1	-7	156	232	99
27	1	-6	1137	1117	99
27	1	-5	137	149	99
27	1	-4	908	936	99
27	1	-3	790	762	99
27	1	-2	1440	1160	99

h	k	l	Fo	Fc	sigF2
27	1	-1	777	712	99
27	1	0	1180	1139	99
27	1	1	603	574	99
27	1	2	1501	1462	99
27	1	3	493	436	99
27	1	4	1079	1112	99
27	1	5	205	150	99
27	1	6	804	900	99
27	1	7	646	604	99
27	1	8	848	880	99
27	3	-8	406	449	99
27	3	-7	828	816	99
27	3	-6	508	484	99
27	3	-5	725	729	99
27	3	-4	915	866	99
27	3	-3	838	848	99
27	3	-2	485	476	99
27	3	-1	783	773	99
27	3	0	739	765	99
27	3	1	876	867	99
27	3	2	395	412	99
27	3	3	894	951	99
27	3	4	544	574	99
27	3	5	846	838	99
27	3	6	444	365	99
28	0	-10	940	919	99
28	0	-8	1005	1036	99
28	0	-6	1249	1234	99
28	0	-4	866	813	99
28	0	-2	1509	1464	99
28	0	0	1200	1148	99
28	0	2	1395	1338	99
28	0	4	873	822	99
28	0	6	645	710	99
28	0	8	937	966	99
28	2	-9	556	568	99
28	2	-8	761	743	99
28	2	-7	880	875	99
28	2	-6	928	923	99
28	2	-5	520	500	99
28	2	-4	1062	997	99
28	2	-3	576	593	99
28	2	-2	841	860	99
28	2	-1	601	607	99
28	2	0	684	650	99
28	2	1	651	635	99
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28	2	4	850	900	99
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28	2	6	687	726	99
28	2	7	647	647	99
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28	4	-4	223	277	99
28	4	-3	981	973	99
28	4	-2	401	343	99
28	4	-1	761	771	99
28	4	0	518	551	99
28	4	1	741	776	99
28	4	2	251	143	99
28	4	3	744	731	99
29	1	-9	169	300	99
29	1	-8	726	717	99
29	1	-7	462	468	99
29	1	-6	922	916	99
29	1	-5	153	135	99
29	1	-4	764	795	99
29	1	-3	280	290	99
29	1	-2	1327	1291	99
29	1	-1	818	797	99
29	1	0	1103	1043	99
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29	1	2	1112	1076	99
29	1	3	-188	133	99
29	1	4	873	911	99
29	1	5	267	225	99
29	1	6	795	830	99
29	1	7	333	473	99
29	3	-7	766	763	99
29	3	-6	468	404	99
29	3	-5	747	740	99
29	3	-4	675	682	99
29	3	-3	867	892	99
29	3	-2	321	293	99
29	3	-1	580	609	99
29	3	0	591	584	99
29	3	1	766	795	99
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29	3	3	857	898	99
29	3	4	514	493	99
29	3	5	752	803	99
30	0	-8	968	960	99
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30	0	-4	901	868	99
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30	0	2	999	966	99
30	0	4	848	790	99
30	0	6	640	732	99
30	2	-8	614	569	99
30	2	-7	832	824	99
30	2	-6	765	789	99
30	2	-5	320	386	99
30	2	-4	923	925	99
30	2	-3	599	591	99
30	2	-2	701	725	99
30	2	-1	530	529	99
30	2	0	659	648	99
30	2	1	424	411	99
30	2	2	585	594	99
30	2	3	474	468	99
30	2	4	710	707	99
30	2	5	477	436	99
30	4	-2	203	156	99
30	4	-1	691	700	99
30	4	0	514	520	99
31	1	-8	769	766	99
31	1	-7	521	537	99
31	1	-6	856	808	99
31	1	-5	297	146	99
31	1	-4	918	897	99
31	1	-3	-145	255	99
31	1	-2	1049	1069	99
31	1	-1	694	701	99
31	1	0	1063	1038	99
31	1	1	386	452	99
31	1	2	799	792	99
31	1	3	406	432	99
31	1	4	1018	1088	99
31	1	5	258	293	99
31	3	-5	725	758	99
31	3	-4	610	512	99
31	3	-3	850	874	99
31	3	-2	-310	223	99
31	3	-1	658	662	99
31	3	0	483	477	99
31	3	1	638	696	99
31	3	2	652	658	99
31	3	3	638	705	99
32	0	-6	751	741	99
32	0	-4	678	666	99
32	0	-2	1105	1083	99
32	0	0	654	654	99
32	0	2	573	537	99
32	0	4	1185	1151	99
32	2	-6	762	759	99
32	2	-5	424	404	99
32	2	-4	882	903	99
32	2	-3	584	608	99
32	2	-2	675	679	99
32	2	-1	520	524	99
32	2	0	828	806	99
32	2	1	-133	100	99
32	2	2	602	677	99
32	2	3	522	516	99
33	1	-6	731	740	99
33	1	-5	-185	295	99
33	1	-4	832	847	99
33	1	-3	208	361	99
33	1	-2	986	971	99
33	1	-1	295	284	99
33	1	0	883	839	99
33	1	1	256	315	99
33	1	2	952	888	99
33	1	3	728	639	99
34	0	-4	709	689	99
34	0	-2	855	854	99
34	0	0	668	632	99
34	0	2	792	748	99
34	2	-3	363	408	99
34	2	-2	634	644	99
34	2	-1	463	453	99
34	2	0	802	805	99
35	1	-3	363	354	99
35	1	-2	790	823	99
35	1	-1	-186	71	99
35	1	0	693	742	99

APPENDIX 7.V

$[Hg(S_2CNMe_2)_2]$

Table 7.V.1: Crystallographic Parameters for $[Hg(S_2CN(CH_3)_2)_2]$

Formula	$C_6H_{12}HgN_2S_4$
Formula weight	441.00
Crystal morphology / colour	plate / pale green
Crystal dimensions (mm)	0.08 x 0.10 x 0.40
Crystal system	monoclinic
Space group	*C*2/*c* (# 15)
a (Å)	17.308(1)
b (Å)	7.558(3)
c (Å)	9.978(2)
α (°)	90
β (°)	113.24(1)
γ (°)	90
V (Å^3)	1199.3(5)
Z	4
$\rho_{calc.}$ (g cm^{-3})	2.442
F(000)	824
Temperature (°K)	293
μ (cm^{-1})	134.61
θ limits, cell (°)	21.0 – 24.2
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 21, 0 – 8, -12 – 12
Range of transmission factors	0.817 – 1
Scan technique	ω:2θ
No. of data measured	1535
No. of unique data	1489
R_{amal}	0.035
No. of parameters	61
R (I≥0σ(I))	0.089
R_w (I≥0σ(I))	0.098
R (I≥3σ(I))	0.041
R_w (I≥3σ(I))	0.045
Maximum and minimum residual electron density (e Å^{-3})	1.76, -3.79

Table 7.V.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Hg(S_2CN(CH_3)_2)_2]$

Atom	*x*	*y*	*z*
Hg	0	0	0
S(1)	0.0458(2)	0.2878(3)	-0.0303(3)
S(2)	0.1251(1)	-0.0041(4)	-0.1378(3)
N(1)	0.1515(4)	0.340(1)	-0.1565(7)
C(1)	0.1115(5)	0.215(1)	-0.1126(9)
C(2)	0.2086(7)	0.292(1)	-0.231(1)
C(3)	0.1395(7)	0.533(1)	-0.142(1)

Table 7.V.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Hg(S_2CN(CH_3)_2)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Hg	0.0539(3)	0.0585(3)	0.0858(5)	-0.0026(4)	0.0376(3)	0.0130(4)
S(1)	0.048(1)	0.047(1)	0.041(1)	0.001(1)	0.023(1)	-0.004(1)
S(2)	0.044(1)	0.045(1)	0.060(1)	0.002(2)	0.024(1)	-0.003(2)
N(1)	0.036(4)	0.056(6)	0.033(4)	-0.002(4)	0.006(3)	-0.004(4)
C(1)	0.031(4)	0.047(5)	0.039(5)	0.000(4)	0.011(4)	0.003(4)
C(2)	0.071(6)	0.076(8)	0.063(7)	-0.011(6)	0.051(6)	-0.010(6)
C(3)	0.070(7)	0.027(7)	0.063(7)	-0.004(4)	0.021(5)	0.002(4)

Table 7.V.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Hg(S_2CN(CH_3)2)2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(3a)	0.1721	0.5970	-0.1834	5.2135
H(3b)	0.1571	0.5624	-0.0422	5.2135
H(3c)	0.0818	0.5617	-0.1926	5.2135
H(4a)	0.1768	0.2300	-0.3214	5.9028
H(4b)	0.2536	0.2171	-0.1682	5.9028
H(4c)	0.2323	0.3995	-0.2533	5.9028

* H atoms were included in their calculated positions, C-H 0.97Å.

Table 7.V.5: Bond distances (Å) for $[Hg(S_2CN(CH_3)2)2]$

Hg — S(1)	2.375(2)	N(1) — C(1)	1.34(1)
Hg — S(2)	2.988(2)	N(1) — C(2)	1.50(1)
S(1) — C(1)	1.735(8)	N(1) — C(3)	1.49(1)
S(2) — C(1)	1.702(9)		

Table 7.V.6: Bond angles (deg.) for [Hg($S_2CN(CH_3)_2$)2]

S(1) — Hg — S(1)*	180(-)	C(1) — N(1) — C(2)	121.4(8)
S(1) — Hg — S(2)	67.16(8)	C(1) — N(1) — C(3)	123.4(8)
S(1) — Hg — S(2)*	112.84(8)	C(2) — N(1) — C(3)	115.1(8)
S(2) — Hg — S(2)*	180(-)	S(1) — C(1) — S(2)	122.3(5)
Hg — S(1) — C(1)	94.7(3)	S(1) — C(1) — N(1)	116.7(7)
Hg — S(2) — C(1)	75.8(3)	S(2) — C(1) — N(1)	121.1(7)

* atom related by centre of symmetry

Table 7.V.7: Observed and calculated structure factors for $[Hg(S_2CN(CH_3)_2)_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	2	2806	2979	99	1	1	-2	2274	2201	99	1	5	9	-259	87	99	2	2	3	290	282	99	2	6	-1	-272	29	99
0	0	4	1424	1336	99	1	1	-1	1296	1320	99	1	5	10	162	158	99	2	2	4	895	888	99	2	6	0	467	440	99
0	0	6	1631	1583	99	1	1	1	-24	59	99	1	7	-8	182	184	99	2	2	5	28	86	99	2	6	1	-110	71	99
0	0	8	923	892	99	1	1	2	2487	2588	99	1	7	-7	-128	86	99	2	2	6	249	258	99	2	6	2	535	505	99
0	0	10	-274	46	99	1	1	3	741	646	99	1	7	-6	452	458	99	2	2	7	140	129	99	2	6	3	156	84	99
0	2	0	2378	2312	99	1	1	4	1357	1306	99	1	7	-5	-37	29	99	2	2	8	461	428	99	2	6	4	235	232	99
0	2	1	436	427	99	1	1	5	183	173	99	1	7	-4	613	600	99	2	2	9	-73	168	99	2	6	5	-304	69	99
0	2	2	1700	1671	99	1	1	6	804	773	99	1	7	-3	144	73	99	2	2	10	150	168	99	2	6	6	121	100	99
0	2	3	96	128	99	1	1	7	274	211	99	1	7	-2	735	725	99	2	2	11	269	92	99	3	1	-12	216	235	99
0	2	4	664	668	99	1	1	8	612	605	99	1	7	-1	-165	114	99	2	4	-11	139	11	99	3	1	-11	158	72	99
0	2	5	202	245	99	1	1	9	-174	212	99	1	7	0	1118	1080	99	2	4	-10	160	43	99	3	1	-10	287	291	99
0	2	6	1040	1090	99	1	1	10	270	248	99	1	7	1	-47	28	99	2	4	-9	73	64	99	3	1	-9	163	85	99
0	2	7	340	306	99	1	1	11	223	145	99	1	7	2	966	950	99	2	4	-8	290	307	99	3	1	-8	542	562	99
0	2	8	627	608	99	1	3	-11	-44	113	99	1	7	3	104	146	99	2	4	-7	185	186	99	3	1	-7	427	423	99
0	2	9	85	190	99	1	3	-10	-102	43	99	1	7	4	582	548	99	2	4	-6	1202	1162	99	3	1	-6	1600	1559	99
0	2	10	-82	159	99	1	3	-9	301	319	99	1	7	5	-103	69	99	2	4	-5	311	300	99	3	1	-5	476	448	99
0	2	11	84	18	99	1	3	-8	357	383	99	1	7	6	419	401	99	2	4	-4	1417	1394	99	3	1	-4	2468	2400	99
0	4	0	2767	2898	99	1	3	-7	305	307	99	1	7	7	-206	26	99	2	4	-3	-132	166	99	3	1	-3	881	738	99
0	4	1	340	279	99	1	3	-6	1222	1256	99	1	7	8	236	248	99	2	4	-2	1207	1225	99	3	1	-2	1935	1938	99
0	4	2	1771	1787	99	1	3	-5	342	363	99	1	9	-5	-287	66	99	2	4	-1	133	122	99	3	1	-1	494	490	99
0	4	3	100	37	99	1	3	-4	1607	1645	99	1	9	-4	133	116	99	2	4	0	1994	1981	99	3	1	0	2488	2431	99
0	4	4	906	930	99	1	3	-3	446	443	99	1	9	-3	-136	97	99	2	4	1	439	412	99	3	1	1	1423	1345	99
0	4	5	-32	91	99	1	3	-2	1685	1716	99	1	9	-2	-174	81	99	2	4	2	2265	2409	99	3	1	2	2400	2421	99
0	4	6	902	880	99	1	3	-1	868	727	99	1	9	-1	153	155	99	2	4	3	56	65	99	3	1	3	149	137	99
0	4	7	201	194	99	1	3	0	3041	3191	99	1	9	0	322	356	99	2	4	4	1214	1146	99	3	1	4	1493	1463	99
0	4	8	601	591	99	1	3	1	116	5	99	1	9	1	-92	18	99	2	4	5	171	91	99	3	1	5	312	218	99
0	4	9	188	133	99	1	3	2	2549	2663	99	1	9	2	280	300	99	2	4	6	520	509	99	3	1	6	492	537	99
0	4	10	143	110	99	1	3	3	517	497	99	1	9	3	148	150	99	2	4	7	-210	41	99	3	1	7	73	69	99
0	6	0	1357	1422	99	1	3	4	1187	1171	99	1	9	4	-168	48	99	2	4	8	438	427	99	3	1	8	322	292	99
0	6	1	101	54	99	1	3	5	349	307	99	2	0	-12	177	49	99	2	4	9	129	120	99	3	1	9	134	81	99
0	6	2	793	762	99	1	3	6	1018	962	99	2	0	-10	-135	55	99	2	4	10	141	245	99	3	1	10	174	197	99
0	6	3	-69	161	99	1	3	7	163	204	99	2	0	-8	155	209	99	2	6	-9	218	177	99	3	1	11	-322	111	99
0	6	4	332	347	99	1	3	8	624	631	99	2	0	-6	1819	1825	99	2	6	-8	125	133	99	3	3	-11	-270	57	99
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15	1	-12	-118	78	99	16	0	-8	1017	999	99	17	1	-1	265	288	99	18	4	-3	-181	114	99	21	1	-6	216	278	99
15	1	-11	158	78	99	16	0	-6	1159	1098	99	17	1	0	686	708	99	18	4	-2	249	304	99	21	1	-5	-291	23	99

21	1	-4	512	476	99
21	1	-3	106	34	99
21	1	-2	475	436	99
21	1	-1	-257	21	99
21	3	-7	-233	169	99
21	3	-6	245	290	99
21	3	-5	174	100	99
21	3	-4	531	484	99
21	3	-3	147	46	99
22	0	-6	341	380	99
22	0	-4	361	445	99
22	2	-5	197	101	99

APPENDIX 7.VI

$[Hg(S_2CNiBu_2)_2]$

Table 7.VI.1: Crystallographic Parameters for [Hg($S_2CN(CH_2CH(CH_3)2)2)2$]

Formula	$C_{18}H_{36}HgN_2S_4$
Formula weight	609.33
Crystal morphology / colour	block / pale yellow
Crystal dimensions (mm)	0.16 x 0.24 x 0.29
Crystal system	monoclinic
Space group	*C*2/*c* (# 15)
a (Å)	26.27(2)
b (Å)	13.654(8)
c (Å)	13.863(5)
α (°)	90
β (°)	96.64(4)
γ (°)	90
V (Å^3)	4938(4)
Z	8
$\rho_{calc.}$ (g cm^{-3})	1.639
F(000)	2416
Temperature (°K)	110
μ (cm^{-1})	65.92
θ limits, cell (°)	25.9 – 27.5
θ limits, data (°)	3.0 – 25.0
hkl range	0 – 31, 0 – 15, -16 – 16
Range of transmission factors	0.923 – 1
Scan technique	ω:2θ
No. of data measured	6072
No. of unique data	5946
R_{amal}	0.198
No. of parameters	210
R (I≥0σ(I))	0.078
R_w (I≥0σ(I))	0.086
R (I≥3σ(I))	0.037
R_w (I≥3σ(I))	0.037
Maximum and minimum residual electron density (e Å^{-3})	2.57, -2.22

To refine the structure $[Hg(S_2CNiBu_2)_2]$ the general procedure outlined in chapter 2 was followed but disorder was noted in the C(7)-C(9) isobutyl group. The C(8) atom was refined isotropically over two positions, each of equal weight. The C(15) atom was refined isotropically as attempts to refine this atom with anisotropic displacement parameters resulted in a non-positive definite values.

Difficulties with this refinement may be traced to the relatively poor quality of the crystal, crystal instability (30% decay during the data collection; correction applied) and/or the inadequacy of the empirical absorption correction.

Table 7.VI.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Hg(S_2CN(CH_2CH(CH_3)2)2)2]$

Atom	*x*	*y*	*z*
Hg	0.17488(1)	0.22378(3)	0.08766(2)
S(1)	0.20701(8)	0.1915(1)	0.2551(1)
S(2)	0.12661(8)	0.0645(2)	0.1510(1)
S(3)	0.18380(7)	0.2699(2)	-0.0794(1)
S(4)	0.09972(8)	0.3516(2)	0.0229(1)
N(1)	0.1567(2)	0.0543(4)	0.3413(4)
N(2)	0.1129(2)	0.3928(5)	-0.1619(4)
C(1)	0.1621(3)	0.0975(5)	0.2576(5)
C(2)	0.1881(3)	0.0789(6)	0.4339(5)
C(3)	0.2305(3)	0.0060(6)	0.4655(5)
C(4)	0.2650(3)	-0.0159(6)	0.3863(6)
C(5)	0.2597(3)	0.0406(7)	0.5595(6)
C(6)	0.1197(3)	-0.0255(6)	0.3492(5)
C(7)	0.0668(5)	0.005(1)	0.3665(9)
C(8a)	0.045(1)	0.097(2)	0.348(2)
C(8b)	0.0603(8)	0.090(2)	0.429(2)
C(9)	0.0333(4)	-0.0808(7)	0.3830(7)
C(10)	0.1290(3)	0.3447(5)	-0.0820(5)
C(11)	0.1361(3)	0.3783(6)	-0.2521(5)
C(12)	0.1180(3)	0.2878(6)	-0.3082(5)
C(13)	0.1476(3)	0.2737(7)	-0.3953(6)
C(14)	0.0612(3)	0.2870(7)	-0.3389(6)
C(15)	0.0681(3)	0.4584(5)	-0.1639(5)
C(16)	0.0809(3)	0.5588(6)	-0.1200(6)

Table 7.VI.2: Cont.

Atom	*x*	*y*	*z*
C(17)	0.1118(3)	0.6203(6)	-0.1836(6)
C(18)	0.0311(3)	0.6098(6)	-0.1031(6)

Table 7.VI.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Hg(S_2CN(CH_2CH(CH_3)2)2)2]$

Atom	$U11$	$U22$	$U33$	$U12$	$U13$	$U23$
Hg	0.0310(2)	0.0305(2)	0.0303(2)	0.0045(2)	0.0039(1)	0.0063(2)
S(1)	0.012(1)	0.030(1)	0.033(1)	-0.0037(9)	0.0007(9)	0.0059(9)
S(2)	0.022(1)	0.032(1)	0.030(1)	-0.005(1)	0.002(1)	-0.001(1)
S(3)	0.011(1)	0.030(1)	0.033(1)	0.008(1)	0.0046(8)	0.004(1)
S(4)	0.013(1)	0.039(1)	0.032(1)	0.007(1)	0.0094(9)	0.004(1)
N(1)	0.007(4)	0.022(4)	0.031(4)	-0.001(3)	0.003(3)	0.000(3)
N(2)	0.006(4)	0.032(4)	0.020(3)	-0.001(3)	-0.003(3)	0.001(3)
C(1)	0.013(5)	0.020(5)	0.030(4)	0.003(4)	0.005(4)	0.002(4)
C(2)	0.012(5)	0.030(5)	0.030(4)	0.000(4)	0.003(4)	0.000(4)
C(3)	0.012(5)	0.026(5)	0.033(4)	-0.001(4)	0.000(4)	0.009(4)
C(4)	0.014(5)	0.039(6)	0.049(5)	0.007(4)	0.006(4)	0.010(5)
C(5)	0.024(6)	0.043(6)	0.047(6)	0.008(5)	-0.008(5)	0.007(5)
C(6)	0.014(5)	0.032(5)	0.029(4)	-0.007(4)	-0.002(4)	0.001(4)
C(10)	0.007(4)	0.025(5)	0.033(4)	-0.002(4)	0.008(4)	-0.006(4)
C(11)	0.011(5)	0.030(5)	0.021(4)	0.000(4)	-0.001(3)	-0.002(4)
C(12)	0.025(5)	0.031(6)	0.037(5)	-0.004(5)	0.008(4)	-0.001(4)
C(13)	0.026(5)	0.049(6)	0.039(5)	-0.003(5)	0.009(4)	-0.018(5)
C(14)	0.030(6)	0.051(7)	0.042(5)	-0.016(5)	0.007(4)	-0.013(5)
C(16)	0.009(5)	0.025(5)	0.043(5)	0.009(4)	-0.005(4)	0.002(4)
C(17)	0.027(6)	0.030(5)	0.037(5)	-0.002(4)	-0.002(4)	-0.003(4)
C(18)	0.025(6)	0.032(6)	0.047(5)	0.010(5)	0.013(5)	0.003(4)

Table 7.VI.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in [Hg($S_2CN(CH_2CH(CH_3)_2)_2)_2$]*

Atom	x	y	z	B_{eq}
H(2a)	0.2042	0.1424	0.4264	0.0287
H(2b)	0.1658	0.0831	0.4843	0.0287
H(3a)	0.2138	-0.0558	0.4794	0.0283
H(4a)	0.2814	0.0439	0.3690	0.0403
H(4b)	0.2905	-0.0641	0.4091	0.0403
H(4c)	0.2439	-0.0416	0.3289	0.0403
H(5a)	0.2362	0.0468	0.6089	0.0469
H(5b)	0.2862	-0.0065	0.5819	0.0469
H(5c)	0.2752	0.1035	0.5497	0.0469
H(6a)	0.1324	-0.0681	0.4031	0.0305
H(6b)	0.1151	-0.0624	0.2899	0.0305
H(9a)	0.0483	-0.1144	0.4425	0.0622
H(9b)	-0.0011	-0.0621	0.3897	0.0622
H(9c)	0.0350	-0.1237	0.3285	0.0622
H(11a)	0.1283	0.4349	-0.2937	0.0249
H(11b)	0.1732	0.3740	-0.2357	0.0249
H(12a)	0.1259	0.2320	-0.2653	0.0367
H(13a)	0.1843	0.2689	-0.3740	0.0453
H(13b)	0.1364	0.2144	-0.4300	0.0453
H(13c)	0.1419	0.3295	-0.4391	0.0453
H(14a)	0.0509	0.2266	-0.3715	0.0492
H(14b)	0.0427	0.2948	-0.2822	0.0492
H(14c)	0.0520	0.3419	-0.3830	0.0492
H(15a)	0.0431	0.4273	-0.1277	0.0222

Table 7.VI.4: Cont.

Atom	x	y	z	B_{eq}
H(15b)	0.0537	0.4664	-0.2311	0.0222
H(16a)	0.1014	0.5496	-0.0583	0.0317
H(17a)	0.1196	0.6827	-0.1523	0.0386
H(17b)	0.1430	0.5865	-0.1937	0.0386
H(17c)	0.0914	0.6313	-0.2461	0.0386
H(18a)	0.0129	0.5704	-0.0603	0.0406
H(18b)	0.0389	0.6734	-0.0738	0.0406
H(18c)	0.0099	0.6186	-0.1648	0.0406

* H atoms were included in their calculated positions, C-H 0.97 Å.

Table 7.VI.5: Bond distances (Å) for $[Hg(S_2CN(CH_2CH(CH_3)2)2)2]$

Hg — S(1)	2.417(2)	C(2) — C(3)	1.52(1)
Hg — S(2)	2.714(2)	C(3) — C(4)	1.53(1)
Hg — S(3)	2.437(2)	C(3) — C(5)	1.51(1)
Hg — S(4)	2.709(2)	C(6) — C(7)	1.50(1)
S(1) — C(1)	1.746(8)	C(7) — C(8a)	1.39(3)
S(2) — C(1)	1.714(8)	C(7) — C(8b)	1.47(2)
S(3) — C(10)	1.763(7)	C(7) — C(9)	1.50(1)
S(4) — C(10)	1.724(7)	C(8a) — C(8b)	1.16(3)
N(1) — C(1)	1.325(8)	C(11) — C(12)	1.51(1)
N(1) — C(2)	1.481(9)	C(12) — C(13)	1.52(1)
N(1) — C(6)	1.473(9)	C(12) — C(14)	1.50(1)
N(2) — C(10)	1.315(9)	C(15) — C(16)	1.52(1)
N(2) — C(11)	1.465(8)	C(16) — C(17)	1.52(1)
N(2) — C(15)	1.477(9)	C(16) — C(18)	1.52(1)

Table 7.VI.6: Bond angles (deg.) for [Hg($S_2CN(CH_2CH(CH_3)2)2)2$]

S(1) — Hg — S(2)	70.58(7)	C(4) — C(3) — C(5)	113.4(7)
S(1) — Hg — S(3)	153.95(7)	N(1) — C(6) — C(7)	115.9(8)
S(1) — Hg — S(4)	126.57(6)	C(6) — C(7) — C(8a)	127(2)
S(2) — Hg — S(3)	127.90(7)	C(6) — C(7) — C(8b)	119(1)
S(2) — Hg — S(4)	105.88(7)	C(6) — C(7) — C(9)	112(1)
S(3) — Hg — S(4)	70.44(6)	C(8a) — C(7) — C(8b)	48(1)
Hg — S(1) — C(1)	89.5(2)	C(8a) — C(7) — C(9)	119(2)
Hg — S(2) — C(1)	80.8(2)	C(8b) — C(7) — C(9)	114(1)
Hg — S(3) — C(10)	90.1(2)	C(7) — C(8a) — C(8b)	69(2)
Hg — S(4) — C(10)	82.3(3)	C(7) — C(8b) — C(8a)	63(2)
C(1) — N(1) — C(2)	123.5(6)	S(3) — C(10) — S(4)	117.2(4)
C(1) — N(1) — C(6)	122.3(6)	S(3) — C(10) — N(2)	119.3(5)
C(2) — N(1) — C(6)	114.2(6)	S(4) — C(10) — N(2)	123.5(6)
C(10) — N(2) — C(11)	121.8(6)	N(2) — C(11) — C(12)	114.4(6)
C(10) — N(2) — C(15)	119.7(6)	C(11) — C(12) — C(13)	110.7(6)
C(11) — N(2) — C(15)	118.3(6)	C(11) — C(12) — C(14)	113.3(7)
S(1) — C(1) — S(2)	118.5(4)	C(13) — C(12) — C(14)	110.9(7)
S(1) — C(1) — N(1)	119.1(6)	N(2) — C(15) — C(16)	113.5(6)
S(2) — C(1) — N(1)	122.3(6)	C(15) — C(16) — C(17)	112.0(6)
N(1) — C(2) — C(3)	114.6(6)	C(15) — C(16) — C(18)	108.6(7)
C(2) — C(3) — C(4)	113.3(6)	C(17) — C(16) — C(18)	111.2(7)
C(2) — C(3) — C(5)	109.0(7)		

Table 7.VI.7: Observed and calculated structure factors for $[Hg(S_2CN(CH_2CH(CH_3)_2)_2)_2]$

h	k	l	Fo	Fc	sigF2
0	0	4	5101	5105	99
0	0	6	5082	5186	99
0	0	8	533	451	99
0	0	10	1061	1108	99
0	0	12	3744	3575	99
0	0	14	118	58	99
0	0	16	1453	1398	99
0	2	-6	5928	5890	99
0	2	1	3564	3659	99
0	2	2	2249	2223	99
0	2	3	564	577	99
0	2	4	1059	1013	99
0	2	5	583	572	99
0	2	7	1562	1617	99
0	2	8	1255	1321	99
0	2	9	878	830	99
0	2	10	2753	2729	99
0	2	11	2034	1993	99
0	2	12	2653	2764	99
0	2	13	514	587	99
0	2	14	-278	106	99
0	2	15	593	574	99
0	2	16	815	925	99
0	4	0	3992	4113	99
0	4	1	2372	2388	99
0	4	2	1982	2006	99
0	4	3	3112	3163	99
0	4	4	3517	3559	99
0	4	5	2533	2481	99
0	4	6	3461	3574	99
0	4	7	2943	2977	99
0	4	8	1157	1158	99
0	4	9	2589	2589	99
0	4	10	1731	1757	99
0	4	11	356	390	99
0	4	12	1754	1679	99
0	4	13	995	999	99
0	4	14	-383	464	99
0	4	15	-108	387	99
0	6	0	3642	3584	99
0	6	1	3513	3506	99
0	6	2	609	658	99
0	6	3	3746	3669	99
0	6	4	3038	3061	99
0	6	5	1295	1255	99
0	6	6	2009	2110	99
0	6	7	1322	1302	99
0	6	8	849	814	99
0	6	9	2253	2201	99
0	6	10	1059	1131	99
0	6	11	797	834	99
0	6	12	1277	1336	99
0	6	13	1517	1647	99
0	6	14	472	369	99
0	6	15	1404	1472	99
1	1	0	551	474	99
1	1	1	2911	2865	99
1	1	2	1266	1285	99
1	1	3	4037	4000	99
1	1	4	379	423	99
1	1	5	1879	1891	99
1	1	6	1018	973	99
1	1	7	1708	1643	99
1	1	8	101	396	99
1	1	9	2143	2138	99
1	1	10	1346	1333	99
1	1	11	657	658	99
1	1	12	-560	158	99
1	1	13	1291	1418	99
1	1	14	-424	366	99
1	1	0	69	35	99
1	1	1	1389	1405	99
1	1	2	40	758	99
1	1	3	3303	3275	99
1	1	4	503	523	99
1	1	5	1308	1261	99
1	1	6	967	930	99
1	1	7	1849	1799	99
1	1	8	286	54	99
1	1	9	2589	2577	99
1	1	10	-315	200	99
1	1	11	-512	175	99
1	1	12	354	237	99
1	1	0	1080	1236	99
1	1	1	1316	1374	99
1	1	2	-315	140	99
1	1	3	3077	3130	99
1	1	4	298	398	99
1	1	5	-463	178	99
1	1	6	729	649	99
1	1	7	1191	1158	99
1	1	8	650	513	99
1	1	9	2190	2227	99
1	1	10	-446	118	99
1	1	11	467	512	99
1	1	0	1590	1676	99
1	1	1	-401	480	99
1	1	2	1031	1096	99
1	1	3	1967	1981	99
1	1	4	792	761	99
1	1	5	764	637	99
1	1	6	833	867	99
1	1	7	906	909	99
1	1	8	546	549	99
1	1	0	1093	1165	99
1	1	1	433	498	99
1	1	2	1007	1018	99
1	1	-16	236	96	99
1	1	-15	1197	1221	99
1	1	-14	617	624	99
1	1	-13	645	646	99
1	1	-12	252	78	99
1	1	-11	2716	2690	99
1	1	-10	1274	1267	99
1	1	-9	1209	1236	99
1	1	-8	428	288	99
1	1	-7	1739	1690	99
1	1	-6	739	710	99
1	1	-5	5858	5805	99
1	1	-4	988	1013	99
1	1	-3	1889	1878	99
1	1	-2	498	377	99
1	1	2	789	708	99
1	1	3	2662	2735	99
1	1	4	1148	1243	99
1	1	5	3200	3216	99
1	1	6	203	148	99
1	1	7	3530	3655	99
1	1	8	533	670	99
1	1	9	131	327	99
1	1	10	514	607	99
1	1	11	2800	2766	99
1	1	12	-387	360	99
1	1	13	2499	2461	99
1	1	14	-392	154	99
1	1	15	-434	206	99
1	1	16	399	333	99
1	3	-16	464	485	99
1	3	-15	674	677	99
1	3	-14	745	692	99
1	3	-13	480	458	99
1	3	-12	582	638	99
1	3	-11	2569	2631	99
1	3	-10	965	961	99
1	3	-9	1804	1797	99
1	3	-8	1852	1903	99
1	3	-7	2315	2343	99
1	3	-6	2307	2294	99
1	3	-5	5008	5026	99
1	3	-4	1655	1631	99
1	3	-3	356	313	99
1	3	-2	2646	2682	99
1	3	-1	4640	4571	99
1	3	0	1576	1525	99
1	3	1	3856	3954	99
1	3	2	2676	2660	99
1	3	3	1467	1444	99
1	3	4	2127	2119	99
1	3	5	3234	3282	99
1	3	6	1015	978	99
1	3	7	4589	4741	99
1	3	8	1420	1346	99
1	3	9	646	623	99
1	3	10	1796	1781	99
1	3	11	2214	2169	99
1	3	12	242	148	99
1	3	13	1766	1715	99
1	3	14	625	582	99
1	3	15	213	115	99
1	3	16	453	481	99
1	5	-15	787	769	99
1	5	-14	863	953	99
1	5	-13	949	990	99
1	5	-12	1016	962	99
1	5	-11	2127	2157	99
1	5	-10	1130	1218	99
1	5	-9	1194	1215	99
1	5	-8	2644	2651	99
1	5	-7	524	533	99
1	5	-6	1379	1354	99
1	5	-5	2799	2721	99
1	5	-4	3050	2979	99
1	5	-3	2188	2420	99
1	5	-2	2526	2497	99
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1	5	0	1335	1348	99
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26	0	-4	315	393	99	26	8	-4	-536	340	99	27	7	-2	1271	1271	99	29	1	2	137	210	99						
26	0	-2	704	752	99	26	8	-3	1264	1279	99	27	7	-1	750	656	99	29	1	3	1031	1121	99						
26	0	0	1949	1978	99	26	8	-2	659	568	99	27	7	0	1052	1196	99	29	1	4	-797	165	99						
26	0	2	1013	831	99	26	8	-1	-532	330	99	27	7	1	-407	320	99	29	3	-6	-393	813	99						
26	0	4	627	621	99	26	8	0	-182	111	99	27	7	2	1013	945	99	29	3	-5	1290	1280	99						
26	0	6	1870	1970	99	26	8	1	949	838	99	28	0	-8	443	266	99	29	3	-4	212	420	99						
26	2	-10	97	688	99	26	8	2	-716	125	99	28	0	-6	1638	1692	99	29	3	-3	1492	1324	99						
26	2	-9	747	877	99	27	1	-9	259	488	99	28	0	-4	1735	1713	99	29	3	-2	-503	170	99						
26	2	-8	612	246	99	27	1	-8	435	217	99	28	0	-2	882	828	99	29	3	-1	397	254	99						
26	2	-7	656	576	99	27	1	-7	658	728	99	28	0	0	1563	1323	99	29	3	0	1129	1137	99						
26	2	-6	1961	1896	99	27	1	-6	427	84	99	28	0	2	2218	2259	99	29	3	1	2402	2148	99						
26	2	-5	450	570	99	27	1	-5	1792	1941	99	28	0	4	-130	469	99	29	3	2	-605	502	99						
26	2	-4	887	866	99	27	1	-4	210	102	99	28	2	-8	352	612	99	29	3	3	840	915	99						
26	2	-3	225	282	99	27	1	-3	-386	260	99	28	2	-7	-432	769	99	29	5	-5	937	795	99						
26	2	-2	1428	1286	99	27	1	-2	296	147	99	28	2	-6	1370	1393	99	29	5	-4	-277	450	99						
26	2	-1	907	876	99	27	1	-1	1089	1091	99	28	2	-5	384	412	99	29	5	-3	-140	421	99						
26	2	0	2528	2412	99	27	1	0	-645	409	99	28	2	-4	2050	2174	99	29	5	-2	991	654	99						
26	2	1	-206	290	99	27	1	1	2650	2692	99	28	2	-3	-426	214	99	29	5	-1	778	557	99						
26	2	2	-488	204	99	27	1	2	672	791	99	28	2	-2	264	177	99	29	5	0	1778	1637	99						
26	2	3	-395	208	99	27	1	3	654	555	99	28	2	-1	-578	151	99	29	5	1	1003	976	99						
26	2	4	715	724	99	27	1	4	-513	368	99	28	2	0	1954	2047	99	30	0	-6	-869	168	99						
26	2	5	264	45	99	27	1	5	951	785	99	28	2	1	-577	141	99	30	0	-4	2347	2316	99						
26	2	6	954	1103	99	27	1	6	560	236	99	28	2	2	1493	1426	99	30	0	-2	1250	1335	99						
26	2	7	1127	1198	99	27	3	-9	942	1053	99	28	2	3	529	547	99	30	0	0	-462	307	99						
26	4	-9	120	604	99	27	3	-8	983	1178	99	28	2	4	444	251	99	30	0	2	981	1045	99						
26	4	-8	984	779	99	27	3	-7	712	558	99	28	2	5	619	858	99	30	2	-5	513	393	99						
26	4	-7	889	941	99	27	3	-6	1115	1110	99	28	4	-7	1136	1073	99	30	2	-4	1567	1511	99						
26	4	-6	1491	1562	99	27	3	-5	2376	2386	99	28	4	-6	1025	946	99	30	2	-3	-292	660	99						
26	4	-5	567	841	99	27	3	-4	695	558	99	28	4	-5	327	674	99	30	2	-2	1173	1159	99						

APPENDIX 7.VII

$[Hg(S_2CN(iPr)Cy)_2]$

Table 7.VII.1: Crystallographic Parameters for $[Hg(S_2CN(CH(CH_3)_2)(CH(CH_2)_5))_2]$

Formula	$C_{20}H_{26}HgN_2S_4$
Formula weight	633.35
Crystal morphology / colour	block / pale yellow
Crystal dimensions (mm)	0.07 x 0.21 x 0.26
Crystal system	triclinic
Space group	$P\bar{1}$ (#2)
a (Å)	11.534(6)
b (Å)	12.291(9)
c (Å)	9.559(4)
α (°)	91.06(5)
β (°)	110.18(4)
γ (°)	72.00(5)
V (Å^3)	1203(1)
Z	2
$\rho_{calc.}$ (g cm^{-3})	1.747
$F(000)$	628
Temperature (°K)	200
μ (cm^{-1})	67.33
θ limits, cell (°)	20.3 – 23.1
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 15, -15 – 15, -12 – 12
Range of transmission factors	0.972 – 1
Scan technique	ω:2θ
No. of data measured	5834
No. of unique data	5557
R_{amal}	0.076
No. of parameters	239
R ($I \geq 0\sigma(I)$)	0.087
R_w ($I \geq 0\sigma(I)$)	0.103
R ($I \geq 3\sigma(I)$)	0.043
R_w ($I \geq 3\sigma(I)$)	0.046
Maximum and minimum residual electron density (e Å^{-3})	3.19, -4.43

Table 7.VII.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Hg(S_2CN(CH(CH_3)_2)(CH(CH_2)_5))_2]$

Atom	*x*	*y*	*z*
Hg	0.15027(3)	0.01335(3)	0.14234(3)
S(1)	0.2102(2)	0.1382(2)	0.0081(2)
S(2)	0.3826(2)	0.0370(2)	0.3161(2)
S(3)	0.0413(2)	-0.0488(1)	0.2916(2)
S(4)	0.1722(2)	-0.2083(1)	0.1180(2)
N(1)	0.4410(5)	0.1681(4)	0.1477(5)
N(2)	0.0962(5)	-0.2756(4)	0.3283(5)
C(1)	0.3562(6)	0.1193(5)	0.1594(7)
C(2)	0.5686(6)	0.1577(6)	0.2694(7)
C(3)	0.5512(7)	0.2127(6)	0.4091(8)
C(4)	0.6643(7)	0.0376(7)	0.3059(8)
C(5)	0.4134(6)	0.2380(5)	0.0089(6)
C(6)	0.5135(7)	0.1914(6)	-0.0643(7)
C(7)	0.4699(7)	0.2631(7)	-0.2152(8)
C(8)	0.4506(8)	0.3879(7)	-0.1931(9)
C(9)	0.3535(8)	0.4352(7)	-0.1153(9)
C(10)	0.3932(7)	0.3641(6)	0.0337(8)
C(11)	0.1037(6)	-0.1900(5)	0.2528(6)
C(12)	0.1467(7)	-0.3987(6)	0.3047(7)
C(13)	0.2948(8)	-0.4420(6)	0.3502(9)
C(14)	0.0760(8)	-0.4268(6)	0.1476(8)
C(15)	0.0411(6)	-0.2537(5)	0.4506(7)
C(16)	0.1451(7)	-0.3077(7)	0.6022(7)
C(17)	0.0875(8)	-0.2769(6)	0.7249(8)

Table 7.VII.2: Cont.

Atom	*x*	*y*	*z*
C(18)	-0.0320(9)	-0.3138(7)	0.6943(9)
C(19)	-0.1331(8)	-0.2618(9)	0.5437(10)
C(20)	-0.0782(7)	-0.2911(8)	0.4178(8)

Table 7.VII.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms i [$Hg(S_2CN(CH(CH_3)_2)(CH(CH_2)_5))_2$]

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	0.0199(8)	0.035(1)	0.0238(8)	-0.0039(8)	0.0002(7)	0.0033(7)
S(2)	0.0320(9)	0.034(1)	0.0205(8)	-0.0067(8)	0.0042(7)	-0.0008(7)
S(3)	0.0290(9)	0.0174(9)	0.0335(9)	0.0023(7)	0.0178(7)	-0.0070(7)
S(4)	0.0378(10)	0.0272(9)	0.0278(9)	-0.0046(8)	0.0218(8)	-0.0134(7)
N(1)	0.017(3)	0.020(3)	0.018(3)	0.004(2)	0.005(2)	-0.004(2)
N(2)	0.025(3)	0.015(3)	0.020(3)	0.004(2)	0.011(2)	-0.007(2)
C(1)	0.023(3)	0.014(3)	0.024(3)	0.002(3)	0.009(3)	-0.013(2)
C(2)	0.018(3)	0.034(4)	0.028(3)	-0.003(3)	0.007(3)	-0.010(3)
C(3)	0.041(4)	0.034(4)	0.025(4)	-0.010(4)	0.006(3)	-0.017(3)
C(4)	0.019(4)	0.046(5)	0.034(4)	0.008(3)	0.002(3)	-0.009(3)
C(5)	0.020(3)	0.025(4)	0.017(3)	-0.002(3)	0.004(2)	-0.005(2)
C(6)	0.031(4)	0.024(4)	0.029(4)	0.004(3)	0.016(3)	-0.008(3)
C(7)	0.037(4)	0.056(5)	0.030(4)	-0.011(4)	0.018(3)	-0.007(3)
C(8)	0.050(5)	0.046(5)	0.041(4)	-0.008(4)	0.020(4)	0.007(4)
C(9)	0.049(5)	0.030(4)	0.047(5)	-0.002(4)	0.017(4)	0.004(3)
C(10)	0.040(4)	0.023(4)	0.036(4)	0.004(3)	0.019(3)	-0.006(3)
C(11)	0.018(3)	0.021(3)	0.016(3)	0.003(3)	0.007(2)	-0.009(2)
C(12)	0.036(4)	0.018(4)	0.030(4)	-0.002(3)	0.015(3)	-0.010(3)
C(13)	0.043(5)	0.025(4)	0.045(5)	0.013(4)	0.019(4)	-0.005(3)
C(14)	0.051(5)	0.028(4)	0.031(4)	-0.009(4)	0.016(3)	-0.016(3)
C(15)	0.030(4)	0.016(3)	0.024(3)	0.010(3)	0.015(3)	-0.005(3)
C(16)	0.031(4)	0.043(5)	0.020(3)	-0.003(3)	0.009(3)	-0.011(3)
C(17)	0.058(5)	0.024(4)	0.028(4)	0.004(4)	0.020(4)	-0.013(3)
C(18)	0.074(6)	0.040(5)	0.039(4)	-0.003(4)	0.043(4)	-0.003(4)

Table 7.VII.3: Cont.

Atom	$U11$	$U22$	$U33$	$U12$	$U13$	$U23$
C(19)	0.042(5)	0.088(8)	0.046(5)	-0.006(5)	0.026(4)	0.006(5)
C(20)	0.029(4)	0.065(6)	0.029(4)	-0.004(4)	0.008(3)	-0.002(4)

Table 7.VII.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Hg(S_2CN(CH(CH_3)_2)(CH(CH_2)_5))_2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.6080	0.2030	0.2300	2.6846
H(3a)	0.6359	0.2083	0.4819	3.3232
H(3b)	0.4971	0.2926	0.3816	3.3232
H(3c)	0.5097	0.1720	0.4522	3.3232
H(4a)	0.6302	-0.0120	0.3466	3.7424
H(4b)	0.6776	0.0090	0.2155	3.7424
H(4c)	0.7468	0.0385	0.3790	3.7424
H(5a)	0.3311	0.2342	-0.0614	2.1223
H(6a)	0.5214	0.1119	-0.0816	2.8000
H(6b)	0.5972	0.1961	0.0008	2.8000
H(7a)	0.5363	0.2351	-0.2597	3.8134
H(7b)	0.3884	0.2550	-0.2818	3.8134
H(8a)	0.4184	0.4313	-0.2901	4.3819
H(8b)	0.5337	0.3963	-0.1326	4.3819
H(9a)	0.2683	0.4337	-0.1801	4.1490
H(9b)	0.3493	0.5138	-0.0961	4.1490
H(10a)	0.4738	0.3721	0.1029	3.2987
H(10b)	0.3249	0.3918	0.0754	3.2987
H(12a)	0.1272	-0.4414	0.3731	2.7074
H(13a)	0.3238	-0.5238	0.3406	3.9924
H(13b)	0.3203	-0.4017	0.2856	3.9924
H(13c)	0.3344	-0.4282	0.4534	3.9924
H(14a)	-0.0170	-0.4033	0.1281	3.5332
H(14b)	0.0919	-0.3860	0.0739	3.5332

Table 7.VII.4: Cont.

Atom	*x*	*y*	*z*	B_{eq}
H(14c)	0.1083	-0.5089	0.1414	3.5332
H(15a)	0.0133	-0.1712	0.4556	2.4379
H(16a)	0.1753	-0.3906	0.6017	3.1668
H(16b)	0.2180	-0.2788	0.6213	3.1668
H(17a)	0.1527	-0.3152	0.8202	3.7730
H(17b)	0.0630	-0.1943	0.7294	3.7730
H(18a)	-0.0064	-0.3969	0.6967	4.5796
H(18b)	-0.0690	-0.2892	0.7711	4.5796
H(19a)	-0.1635	-0.1789	0.5442	5.5549
H(19b)	-0.2057	-0.2910	0.5251	5.5549
H(20a)	-0.1440	-0.2514	0.3236	4.1544
H(20b)	-0.0542	-0.3734	0.4111	4.1544

* H atoms were included in their calculated positions, C-H 0.97Å.

Table 7.VII.5: Bond distances (Å) for $[Hg(S_2CN(CH(CH_3)_2)(CH(CH_2)_5))_2]$

Hg — S(1)	2.412(2)	C(2) — C(4)	1.50(1)
Hg — S(2)	2.723(2)	C(5) — C(6)	1.512(9)
Hg — S(3)	2.464(2)	C(5) — C(10)	1.521(9)
Hg — S(4)	2.670(2)	C(6) — C(7)	1.544(9)
S(1) — C(1)	1.753(6)	C(7) — C(8)	1.50(1)
S(2) — C(1)	1.705(6)	C(8) — C(9)	1.52(1)
S(3) — C(11)	1.748(6)	C(9) — C(10)	1.53(1)
S(4) — C(11)	1.707(6)	C(12) — C(13)	1.52(1)
N(1) — C(1)	1.330(8)	C(12) — C(14)	1.53(1)
N(1) — C(2)	1.498(8)	C(15) — C(16)	1.525(9)
N(1) — C(5)	1.477(7)	C(15) — C(20)	1.51(1)
N(2) — C(11)	1.329(7)	C(16) — C(17)	1.53(1)
N(2) — C(12)	1.488(8)	C(17) — C(18)	1.51(1)
N(2) — C(15)	1.495(8)	C(18) — C(19)	1.50(1)
C(2) — C(3)	1.53(1)	C(19) — C(20)	1.53(1)

Table 7.VII.6: Bond angles (deg.) for [Hg($S_2CN(CH(CH_3)_2)(CH(CH_2)_5)$)2]

S(1) — Hg — S(2)	69.97(5)	N(1) — C(5) — C(6)	113.2(5)
S(1) — Hg — S(3)	160.02(6)	N(1) — C(5) — C(10)	111.7(5)
S(1) — Hg — S(4)	129.07(6)	C(6) — C(5) — C(10)	111.9(5)
S(2) — Hg — S(3)	112.32(6)	C(5) — C(6) — C(7)	109.5(5)
S(2) — Hg — S(4)	110.77(6)	C(6) — C(7) — C(8)	110.8(6)
S(3) — Hg — S(4)	69.80(5)	C(7) — C(8) — C(9)	111.7(6)
Hg — S(1) — C(1)	90.7(2)	C(8) — C(9) — C(10)	111.5(6)
Hg — S(2) — C(1)	81.8(2)	C(5) — C(10) — C(9)	110.1(6)
Hg — S(3) — C(11)	88.9(2)	S(3) — C(11) — S(4)	116.7(3)
Hg — S(4) — C(11)	83.2(2)	S(3) — C(11) — N(2)	119.2(5)
C(1) — N(1) — C(2)	124.1(5)	S(4) — C(11) — N(2)	124.0(5)
C(1) — N(1) — C(5)	120.8(5)	N(2) — C(12) — C(13)	112.4(6)
C(2) — N(1) — C(5)	115.1(5)	N(2) — C(12) — C(14)	113.3(5)
C(11) — N(2) — C(12)	123.6(5)	C(13) — C(12) — C(14)	113.1(6)
C(11) — N(2) — C(15)	121.1(5)	N(2) — C(15) — C(16)	111.2(5)
C(12) — N(2) — C(15)	115.2(5)	N(2) — C(15) — C(20)	112.2(6)
S(1) — C(1) — S(2)	117.4(3)	C(16) — C(15) — C(20)	111.9(6)
S(1) — C(1) — N(1)	119.5(5)	C(15) — C(16) — C(17)	109.6(6)
S(2) — C(1) — N(1)	123.1(5)	C(16) — C(17) — C(18)	111.2(6)
N(1) — C(2) — C(3)	112.5(5)	C(17) — C(18) — C(19)	111.1(6)
N(1) — C(2) — C(4)	114.0(6)	C(18) — C(19) — C(20)	111.8(7)
C(3) — C(2) — C(4)	112.5(6)	C(15) — C(20) — C(19)	109.7(7)

Table 7.VII.7: Observed and calculated structure factors for $[Hg(S_2CN(CH(CH_3)_2)(CH(CH_2)_5))_2]$

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
0	0	2	810	760	99	0	3	-10	64	56	99	0	5	1	1026	934	99	0	8	-7	278	275	99	0	11	-7	80	64	99
0	0	3	580	541	99	0	3	-9	-66	22	99	0	5	2	551	553	99	0	8	-6	25	26	99	0	11	-6	246	198	99
0	0	4	1044	982	99	0	3	-8	101	79	99	0	5	3	1034	1071	99	0	8	-5	246	221	99	0	11	-5	314	288	99
0	0	5	562	531	99	0	3	-7	545	574	99	0	5	4	750	759	99	0	8	-4	325	313	99	0	11	-4	405	407	99
0	0	6	771	733	99	0	3	-6	323	336	99	0	5	5	105	114	99	0	8	-3	263	240	99	0	11	-3	296	287	99
0	0	7	1236	1175	99	0	3	-5	623	629	99	0	5	6	465	476	99	0	8	-2	623	664	99	0	11	-2	245	213	99
0	0	8	380	347	99	0	3	-4	851	884	99	0	5	7	571	578	99	0	8	-1	981	949	99	0	11	-1	508	497	99
0	0	9	94	54	99	0	3	-3	381	392	99	0	5	8	292	307	99	0	8	0	473	460	99	0	11	0	73	111	99
0	0	10	82	155	99	0	3	-2	267	272	99	0	5	9	41	77	99	0	8	1	-49	51	99	0	11	1	185	177	99
0	0	11	346	312	99	0	3	-1	290	308	99	0	5	10	273	339	99	0	8	2	462	454	99	0	11	2	-112	255	99
0	1	-11	229	259	99	0	3	0	1258	1267	99	0	5	11	154	201	99	0	8	3	763	786	99	0	11	3	551	579	99
0	1	-10	183	235	99	0	3	1	99	117	99	0	6	-10	44	60	99	0	8	4	157	193	99	0	11	4	-140	121	99
0	1	-9	90	31	99	0	3	2	1174	1225	99	0	6	-9	-99	23	99	0	8	5	642	621	99	0	11	5	397	359	99
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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12	15	-5	76	32	99
12	15	-4	337	313	99
12	11	-7	84	113	99
12	11	-6	335	333	99
12	11	-5	342	328	99
12	11	-4	112	127	99
12	11	-3	182	165	99
12	11	-2	370	358	99
12	11	-1	243	232	99
12	11	0	94	138	99
12	11	1	348	340	99
12	11	2	119	188	99
12	12	-7	-57	78	99
12	12	-6	308	305	99
12	12	-5	258	255	99
12	12	-4	-84	14	99
12	12	-3	103	115	99
12	12	-2	230	227	99
12	12	-1	191	193	99
12	12	0	140	134	99
12	12	1	360	355	99
12	12	2	121	146	99
12	13	-5	202	199	99
12	13	-4	-110	49	99
12	13	-3	163	161	99
12	13	-2	129	131	99
12	13	-1	9	89	99
12	13	0	169	128	99
13	2	-3	332	321	99
13	2	-2	-52	55	99
13	2	-1	231	250	99
13	2	0	411	414	99
13	2	1	303	317	99
13	3	-9	119	95	99
13	3	-8	240	233	99
13	3	-7	416	408	99
13	3	-6	294	296	99
13	3	-5	-74	62	99
13	3	-4	232	214	99
13	3	-3	323	299	99
13	3	-2	-56	71	99
13	3	-1	240	235	99
13	3	0	335	370	99
13	3	1	-56	214	99
13	4	-9	86	56	99
13	4	-8	233	211	99
13	4	-7	327	291	99
13	4	-6	185	136	99
13	4	-5	153	154	99
13	4	-4	217	221	99
13	4	-3	268	276	99
13	4	-2	142	129	99
13	4	-1	233	218	99
13	4	0	284	253	99
13	4	1	-93	35	99
13	4	2	183	104	99
13	5	-9	51	32	99
13	5	-8	143	163	99
13	5	-7	283	268	99
13	5	-6	-88	39	99
13	5	-5	201	230	99
13	5	-4	350	340	99
13	5	-3	359	340	99
13	5	-2	202	95	99
13	5	-1	304	283	99
13	5	0	232	248	99
13	5	1	-117	48	99
13	5	2	226	164	99
13	6	-9	105	30	99
13	6	-8	299	259	99
13	6	-7	332	302	99
13	6	-6	-125	29	99
13	6	-5	222	174	99
13	6	-4	363	365	99
13	6	-3	423	406	99
13	6	-2	129	112	99
13	6	-1	335	323	99
13	6	0	287	274	99
13	6	1	-134	31	99
13	6	2	49	78	99
13	7	-8	244	245	99
13	7	-7	362	353	99
13	7	-6	-69	65	99
13	7	-5	239	222	99
13	7	-4	264	259	99
13	7	-3	371	352	99
13	7	-2	127	107	99
13	7	-1	339	322	99
13	7	0	297	318	99
13	7	1	42	55	99
13	8	-8	185	196	99
13	8	-7	380	337	99
13	8	-6	135	91	99
13	8	-5	238	220	99
13	8	-4	192	170	99
13	8	-3	206	188	99
13	8	-2	76	27	99
13	8	-1	297	283	99
13	8	0	298	302	99
13	8	1	133	84	99
13	9	-7	338	319	99
13	9	-6	33	25	99
13	9	-5	215	191	99
13	9	-4	203	198	99
13	9	-3	149	90	99
13	9	-2	35	83	99
13	9	-1	323	301	99
13	9	0	250	262	99
13	9	1	-78	12	99
13	10	-7	292	284	99
13	10	-6	60	14	99
13	10	-5	234	213	99
13	10	-4	207	220	99
13	10	-3	59	105	99
13	10	-2	139	101	99
13	10	-1	352	358	99
13	10	0	304	279	99
13	11	-5	217	205	99
13	11	-4	194	170	99
13	11	-3	139	151	99
13	11	-2	-104	59	99
13	11	-1	305	315	99
13	11	0	138	1	99
13	11	1	-118	161	99
13	12	-5	115	201	99
13	12	-4	190	125	99
13	12	-3	95	153	99
13	12	-2	274	264	99
13	12	-1	215	170	99
13	12	0	110	140	99
13	12	1	73	56	99
13	13	-4	139	228	99
13	13	-3	124	100	99
13	13	-2	269	197	99

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF
14	2	-1	386	385	99	14	10	-3	164	111	99																		
14	3	-8	358	366	99	15	5	-4	153	32	99																		
14	3	-7	-94	132	99																								
14	3	-6	157	135	99																								
14	3	-5	124	180	99																								
14	3	-4	251	293	99																								
14	3	-3	132	237	99																								
14	3	-2	72	172	99																								
14	3	-1	77	201	99																								
14	4	-8	216	213	99																								
14	4	-7	21	52	99																								
14	4	-6	212	139	99																								
14	4	-5	277	235	99																								
14	4	-4	260	250	99																								
14	4	-3	218	174	99																								
14	4	-2	267	207	99																								
14	4	-1	232	247	99																								
14	4	0	155	66	99																								
14	5	-8	122	213	99																								
14	5	-7	114	38	99																								
14	5	-6	204	201	99																								
14	5	-5	243	242	99																								
14	5	-4	233	249	99																								
14	5	-3	86	76	99																								
14	5	-2	179	221	99																								
14	5	-1	298	280	99																								
14	5	0	60	32	99																								
14	6	-7	47	46	99																								
14	6	-6	320	301	99																								
14	6	-5	222	225	99																								
14	6	-4	233	245	99																								
14	6	-3	-157	50	99																								
14	6	-2	307	310	99																								
14	6	-1	316	300	99																								
14	6	0	-111	75	99																								
14	7	-7	137	108	99																								
14	7	-6	177	193	99																								
14	7	-5	241	232	99																								
14	7	-4	141	165	99																								
14	7	-3	-52	35	99																								
14	7	-2	279	264	99																								
14	7	-1	341	326	99																								
14	8	-7	-124	102	99																								
14	8	-6	156	95	99																								
14	8	-5	209	199	99																								
14	8	-4	-55	81	99																								
14	8	-3	-133	60	99																								
14	8	-2	216	219	99																								
14	8	-1	223	263	99																								
14	9	-6	175	145	99																								
14	9	-5	180	158	99																								
14	9	-4	78	103	99																								
14	9	-3	-53	103	99																								
14	9	-2	293	269	99																								
14	10	-4	153	131	99																								

APPENDIX 7.VIII

$[Hg(S_2CN(Et)Cy)_2]$

Table 7.VIII.1: Crystallographic Parameters for $[Hg(S_2CN(CH_2CH_3)(CH(CH_2)_5))_2]$

Formula	$C_{18}H_{16}HgN_2S_4$
Formula weight	589.17
Crystal morphology / colour	needle / pale yellow
Crystal dimensions (mm)	0.07 x 0.13 x 0.31
Crystal system	monoclinic
Space group	$P2_1/c$ (# 14)
a (Å)	8.459(6)
b (Å)	19.506(9)
c (Å)	13.955(6)
α (°)	90
β (°)	92.89(4)
γ (°)	90
V (Å^3)	2299(1)
Z	4
$\rho_{calc.}$ (g cm^{-3})	1.748
$F(000)$	1128
Temperature (°K)	200
μ (cm^{-1})	70.79
θ limits, cell (°)	16.9 – 19.5
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 10, 0 – 23, -17 – 15
Range of transmission factors	0.945 – 1
Scan technique	ω:2θ
No. of data measured	5797
No. of unique data	5441
R_{amal}	0.100
No. of parameters	226
R (I≥0σ(I))	0.101
R_w (I≥0σ(I))	0.098
R (I≥3σ(I))	0.041
R_w (I≥3σ(I))	0.041
Maximum and minimum residual electron density (e Å^{-3})	3.60, -5.87 (located near Hg atom)

Table 7.VIII.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Hg(S_2CN(CH_2CH_3)(CH(CH_2)5))2]$

Atom	*x*	*y*	*z*
Hg	0.10527(4)	0.04730(2)	0.39405(2)
S(1)	0.3021(2)	-0.0408(1)	0.4389(1)
S(2)	0.1599(2)	0.0241(1)	0.6091(1)
S(3)	0.0424(3)	0.1717(1)	0.4169(1)
S(4)	0.1198(3)	0.1170(1)	0.2270(1)
N(1)	0.3611(6)	-0.0816(3)	0.6170(4)
N(2)	0.0498(7)	0.2482(4)	0.2596(5)
C(1)	0.2799(7)	-0.0377(4)	0.5625(5)
C(2)	0.4797(8)	-0.1283(4)	0.5787(6)
C(3)	0.411(1)	-0.1950(4)	0.5404(6)
C(4)	0.3462(8)	-0.0829(4)	0.7238(5)
C(5)	0.348(1)	-0.1534(5)	0.7670(6)
C(6)	0.330(1)	-0.1487(6)	0.8738(7)
C(7)	0.460(1)	-0.1056(6)	0.9226(6)
C(8)	0.461(1)	-0.0339(5)	0.8800(6)
C(9)	0.4748(9)	-0.0356(5)	0.7712(6)
C(10)	0.0697(8)	0.1854(4)	0.2962(5)
C(11)	0.087(1)	0.2619(5)	0.1587(5)
C(12)	0.259(1)	0.2767(6)	0.1481(8)
C(13)	-0.0047(9)	0.3055(4)	0.3182(6)
C(14)	0.135(1)	0.3477(6)	0.3624(7)
C(15)	0.070(2)	0.3996(6)	0.4317(9)
C(16)	-0.049(2)	0.4451(6)	0.3834(9)
C(17)	-0.186(2)	0.4050(8)	0.3373(9)
C(18)	-0.126(1)	0.3510(6)	0.2654(7)

Table 7.VIII.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Hg(S_2CN(CH_2CH_3)(CH(CH_2)_5))_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Hg	0.0362(2)	0.0374(2)	0.0378(2)	0.0018(2)	0.0018(1)	0.0098(2)
S(1)	0.039(1)	0.048(1)	0.022(1)	0.009(1)	0.0007(8)	-0.001(1)
S(2)	0.032(1)	0.028(1)	0.027(1)	0.0019(8)	0.0034(8)	-0.0019(8)
S(3)	0.052(1)	0.033(1)	0.024(1)	0.003(1)	0.0080(9)	0.0021(9)
S(4)	0.053(1)	0.041(1)	0.024(1)	0.010(1)	0.007(1)	0.001(1)
N(1)	0.027(3)	0.028(3)	0.024(4)	0.000(3)	0.002(3)	0.001(3)
N(2)	0.037(4)	0.042(4)	0.026(4)	0.002(3)	0.013(3)	0.007(3)
C(1)	0.017(3)	0.020(4)	0.030(4)	-0.008(3)	-0.002(3)	0.004(3)
C(2)	0.024(4)	0.036(5)	0.041(5)	0.008(3)	0.008(3)	-0.003(4)
C(3)	0.048(5)	0.039(5)	0.045(6)	0.000(4)	0.003(4)	-0.017(4)
C(4)	0.022(4)	0.048(5)	0.017(4)	0.009(3)	0.006(3)	0.006(4)
C(5)	0.049(5)	0.044(6)	0.038(5)	-0.001(4)	-0.007(4)	0.015(4)
C(6)	0.046(5)	0.074(8)	0.049(6)	-0.001(5)	0.000(5)	0.024(6)
C(7)	0.057(6)	0.087(8)	0.025(5)	0.013(5)	-0.006(4)	0.014(5)
C(8)	0.059(6)	0.065(8)	0.029(5)	0.003(5)	-0.001(4)	-0.002(5)
C(9)	0.044(5)	0.049(6)	0.034(5)	-0.009(4)	0.004(4)	-0.002(4)
C(10)	0.031(4)	0.033(5)	0.021(4)	0.005(3)	0.005(3)	0.010(3)
C(11)	0.072(6)	0.055(6)	0.016(5)	0.020(5)	0.021(4)	0.012(4)
C(12)	0.072(7)	0.074(8)	0.062(7)	0.012(6)	0.033(6)	0.023(6)
C(13)	0.039(5)	0.039(5)	0.028(5)	-0.001(4)	0.009(4)	0.004(4)
C(14)	0.088(8)	0.056(7)	0.047(6)	-0.018(6)	0.006(5)	-0.010(5)
C(15)	0.11(1)	0.060(8)	0.086(10)	-0.019(7)	0.027(8)	-0.007(7)
C(16)	0.19(2)	0.041(7)	0.066(8)	0.014(9)	0.032(9)	-0.010(7)
C(17)	0.12(1)	0.12(1)	0.062(8)	0.092(10)	0.002(8)	0.003(8)
C(18)	0.088(8)	0.071(8)	0.045(6)	0.035(6)	-0.003(5)	-0.001(6)

Table 7.VIII.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in $[Hg(S_2CN(CH_2CH_3)(CH(CH_2)_5))_2]$*

Atom	*x*	*y*	*z*	B_{eq}
H(2a)	0.5305	-0.1052	0.5270	3.1835
H(2b)	0.5580	-0.1388	0.6298	3.1835
H(3a)	0.3627	-0.2196	0.5918	4.1686
H(3b)	0.3312	-0.1854	0.4897	4.1686
H(3c)	0.4943	-0.2227	0.5151	4.1686
H(4a)	0.2446	-0.0626	0.7363	2.7540
H(5a)	0.2604	-0.1798	0.7381	4.1705
H(5b)	0.4468	-0.1758	0.7548	4.1705
H(6a)	0.3339	-0.1945	0.9007	5.3507
H(6b)	0.2281	-0.1280	0.8854	5.3507
H(7a)	0.5614	-0.1273	0.9139	5.3431
H(7b)	0.4421	-0.1024	0.9906	5.3431
H(8a)	0.5510	-0.0089	0.9085	4.8550
H(8b)	0.3641	-0.0108	0.8945	4.8550
H(9a)	0.4613	0.0105	0.7459	4.0086
H(9b)	0.5786	-0.0527	0.7567	4.0086
H(11a)	0.0576	0.2220	0.1202	4.4484
H(11b)	0.0255	0.3011	0.1358	4.4484
H(12a)	0.3220	0.2381	0.1719	6.4441
H(12b)	0.2783	0.2842	0.0809	6.4441
H(12c)	0.2891	0.3174	0.1846	6.4441
H(13a)	-0.0574	0.2852	0.3714	3.3336
H(14a)	0.2090	0.3175	0.3966	6.0493
H(14b)	0.1877	0.3714	0.3121	6.0493

Table 7.VIII.4: Cont.

Atom	*x*	*y*	*z*	B_{eq}
H(15a)	0.0204	0.3752	0.4829	8.1128
H(15b)	0.1562	0.4273	0.4584	8.1128
H(16a)	0.0010	0.4712	0.3342	9.2047
H(16b)	-0.0899	0.4763	0.4304	9.2047
H(17a)	-0.2577	0.4364	0.3033	9.6004
H(17b)	-0.2414	0.3816	0.3869	9.6004
H(18a)	-0.0776	0.3741	0.2128	6.4726
H(18b)	-0.2142	0.3234	0.2405	6.4726

* H atoms were included in their calculated positions, C-H 0.97Å.

Table 7.VIII.5: Bond distances (Å) for [Hg($S_2CN(CH_2CH_3)(CH(CH_2)_5)$)2]

Hg — S(1)	2.452(2)	C(2) — C(3)	1.51(1)
Hg — S(2)	3.047(2)	C(4) — C(5)	1.50(1)
Hg — S(2)*	2.639(2)	C(4) — C(9)	1.55(1)
Hg — S(3)	2.508(2)	C(5) — C(6)	1.51(1)
Hg — S(4)	2.707(2)	C(6) — C(7)	1.52(1)
S(1) — C(1)	1.746(7)	C(7) — C(8)	1.52(1)
S(2) — C(1)	1.725(7)	C(8) — C(9)	1.53(1)
S(3) — C(10)	1.733(7)	C(11) — C(12)	1.50(1)
S(4) — C(10)	1.713(8)	C(13) — C(14)	1.54(1)
N(1) — C(1)	1.315(8)	C(13) — C(18)	1.52(1)
N(1) — C(2)	1.476(9)	C(14) — C(15)	1.52(1)
N(1) — C(4)	1.503(9)	C(15) — C(16)	1.48(2)
N(2) — C(10)	1.334(9)	C(16) — C(17)	1.51(2)
N(2) — C(11)	1.481(9)	C(17) — C(18)	1.56(1)
N(2) — C(13)	1.47(1)		

* symmetry operation: *-x*, *-y*, 1 – *z*.

Table 7.VIII.6: Bond angles (deg.) for [Hg($S_2CN(CH_2CH_3)(CH(CH_2)_5)$)2]

S(1) — Hg — S(2)	65.00(6)	S(1) — C(1) — N(1)	118.0(5)
S(1) — Hg — S(2)*	101.53(8)	S(2) — C(1) — N(1)	122.5(5)
S(1) — Hg — S(3)	142.30(7)	N(1) — C(2) — C(3)	113.7(6)
S(1) — Hg — S(4)	120.80(7)	N(1) — C(4) — C(5)	114.5(7)
S(2) — Hg — S(2)*	91.40(6)	N(1) — C(4) — C(9)	108.8(6)
S(2) — Hg — S(3)	92.33(6)	C(5) — C(4) — C(9)	112.6(6)
S(2) — Hg — S(4)	155.71(6)	C(4) — C(5) — C(6)	110.0(8)
S(2)* — Hg — S(3)	109.11(7)	C(5) — C(6) — C(7)	111.7(7)
S(2)* — Hg — S(4)	109.06(6)	C(6) — C(7) — C(8)	110.7(7)
S(3) — Hg — S(4)	69.07(6)	C(7) — C(8) — C(9)	111.8(8)
Hg — S(1) — C(1)	97.0(2)	C(4) — C(9) — C(8)	110.6(7)
Hg — S(2) — Hg*	88.60(6)	S(3) — C(10) — S(4)	118.5(4)
Hg — S(2) — C(1)	78.2(2)	S(3) — C(10) — N(2)	119.6(6)
Hg — S(2)* — C(1)*	98.1(2)	S(4) — C(10) — N(2)	121.9(6)
Hg — S(3) — C(10)	89.2(3)	N(2) — C(11) — C(12)	112.4(7)
Hg — S(4) — C(10)	83.3(2)	N(2) — C(13) — C(14)	111.9(7)
C(1) — N(1) — C(2)	122.5(6)	N(2) — C(13) — C(18)	113.4(7)
C(1) — N(1) — C(4)	121.2(6)	C(14) — C(13) — C(18)	111.5(8)
C(2) — N(1) — C(4)	116.1(6)	C(13) — C(14) — C(15)	108.4(9)
C(10) — N(2) — C(11)	120.1(7)	C(14) — C(15) — C(16)	112(1)
C(10) — N(2) — C(13)	121.4(6)	C(15) — C(16) — C(17)	112(1)
C(11) — N(2) — C(13)	118.5(6)	C(16) — C(17) — C(18)	111(1)
S(1) — C(1) — S(2)	119.4(4)	C(13) — C(18) — C(17)	108.5(8)

* symmetry operation: $-x$, $-y$, $1 - z$.

Table 7.VIII.7: Observed and calculated structure factors for $[Hg(S_2CN(CH_2CH_3)(CH(CH_2)_5))_2]$

h	k	l	Fo	Fc	sigF2
0	0	4	2964	3053	99
0	0	6	3192	3218	99
0	0	8	1235	1237	99
0	0	10	1314	1282	99
0	0	12	62	80	99
0	0	14	707	699	99
0	0	16	-172	191	99
0	0	18	603	607	99
0	1	2	1455	1367	99
0	1	3	461	442	99
0	1	4	864	837	99
0	1	5	3057	3089	99
0	1	6	194	270	99
0	1	7	464	433	99
0	1	8	643	630	99
0	1	9	1223	1232	99
0	1	10	151	142	99
0	1	11	902	877	99
0	1	12	597	547	99
0	1	13	917	888	99
0	1	14	-309	39	99
0	1	15	454	428	99
0	1	16	182	33	99
0	1	17	-43	217	99
0	1	18	-188	92	99
0	2	2	1348	1317	99
0	2	3	1310	1246	99
0	2	4	2384	2365	99
0	2	5	705	693	99
0	2	6	1845	1833	99
0	2	7	1768	1790	99
0	2	8	940	948	99
0	2	9	455	471	99
0	2	10	1170	1222	99
0	2	11	633	679	99
0	2	12	451	472	99
0	2	13	375	398	99
0	2	14	794	763	99
0	2	15	-273	136	99
0	2	16	-112	51	99
0	2	17	287	314	99
0	2	18	578	645	99
0	3	1	1683	1721	99
0	3	2	2312	2394	99
0	3	3	1070	1068	99
0	3	4	666	670	99
0	3	5	1576	1479	99
0	3	6	1775	1817	99
0	3	7	178	62	99
0	3	8	690	676	99
0	3	9	1160	1147	99
0	3	10	20	222	99
0	3	11	-115	63	99
0	3	12	968	1015	99
0	3	13	589	552	99
0	3	14	-133	104	99
0	3	15	444	425	99
0	3	16	170	326	99
0	3	17	497	513	99
0	3	18	320	337	99
0	4	0	655	618	99
0	4	1	1507	1556	99
0	4	2	700	669	99
0	4	3	1040	1054	99
0	4	4	1331	1359	99
0	4	5	885	881	99
0	4	6	285	190	99
0	4	7	1762	1786	99
0	4	8	447	437	99
0	4	9	-140	87	99
0	4	10	382	387	99
0	4	11	1155	1092	99
0	4	12	287	221	99
0	4	13	628	639	99
0	4	14	576	573	99
0	4	15	306	347	99
0	4	16	220	168	99
0	4	17	435	525	99
0	5	1	1327	1347	99
0	5	2	1608	1576	99
0	5	3	158	186	99
0	5	4	511	480	99
0	5	5	392	359	99
0	5	6	1384	1416	99
0	5	7	423	434	99
0	5	8	1429	1386	99
0	5	9	258	194	99
0	5	10	918	915	99
0	5	11	-157	55	99
0	5	12	1028	1042	99
0	5	13	-84	104	99
0	5	14	165	156	99
0	5	15	-51	88	99
0	5	16	551	668	99
0	5	17	85	52	99
0	6	0	150	131	99
0	6	1	850	857	99
0	6	2	108	121	99
0	6	3	1656	1669	99
0	6	4	-169	90	99
0	6	5	489	499	99
0	6	6	107	50	99
0	6	7	1584	1540	99
0	6	8	290	191	99
0	6	9	348	293	99
0	6	10	-192	50	99
0	6	11	1054	1062	99
0	6	12	174	21	99
0	6	13	591	669	99
0	6	14	-172	123	99
0	6	15	423	322	99
0	6	16	-194	129	99
0	6	17	297	348	99
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0	7	2	1943	1979	99
0	7	3	679	653	99
0	7	4	550	511	99
0	7	5	652	634	99
0	7	6	978	972	99
0	7	7	54	177	99
0	7	8	1263	1206	99
0	7	9	753	753	99
0	7	10	314	316	99
0	7	11	147	101	99
0	7	12	776	782	99
0	7	13	206	101	99
0	7	14	-49	335	99
0	7	15	217	352	99
0	7	16	165	248	99
0	7	17	-292	100	99
0	8	0	1695	1712	99
0	8	1	950	905	99
0	8	2	895	931	99
0	8	3	1142	1201	99
0	8	4	1447	1499	99
0	8	5	540	552	99
0	8	6	854	848	99
0	8	7	967	956	99
0	8	8	130	138	99
0	8	9	224	270	99
0	8	10	959	960	99
0	8	11	559	501	99
0	8	12	22	40	99
0	8	13	499	489	99
0	8	14	478	496	99
0	8	15	239	105	99
0	8	16	187	83	99
0	8	17	186	181	99
0	9	1	1905	1969	99
0	9	2	261	197	99
0	9	3	472	474	99
0	9	4	194	242	99
0	9	5	1658	1671	99
0	9	6	690	655	99
0	9	7	931	912	99
0	9	8	-152	145	99
0	9	9	1052	1020	99
0	9	10	124	110	99
0	9	11	502	560	99
0	9	12	377	342	99
0	9	13	-144	192	99
0	9	14	62	16	99
0	9	15	380	446	99
0	9	16	82	80	99
0	10	0	2374	2334	99
0	10	1	-343	41	99
0	10	2	508	486	99
0	10	3	428	464	99
0	10	4	1626	1689	99
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0	10	8	855	858	99
0	10	9	279	304	99
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0	10	11	143	169	99
0	10	12	168	37	99
0	10	13	206	87	99
0	10	14	537	594	99
0	10	15	185	134	99
0	10	16	217	66	99
0	11	1	1553	1550	99
0	11	2	820	855	99
0	11	3	477	488	99
0	11	4	319	350	99
0	11	5	1659	1639	99
0	11	6	239	201	99
0	11	7	-295	44	99
0	11	8	861	868	99
0	11	9	522	556	99
0	11	10	135	219	99
0	11	11	473	499	99
0	11	12	149	62	99
0	11	13	580	527	99
0	11	14	127	322	99
0	11	15	265	338	99
0	11	16	231	172	99
0	12	0	1919	1881	99
0	12	1	-53	289	99
0	12	2	524	484	99
0	12	3	840	863	99
0	12	4	1161	1176	99
0	12	5	190	203	99
0	12	6	863	883	99
0	12	7	633	668	99
0	12	8	458	490	99
0	12	9	291	298	99
0	12	10	656	679	99
0	12	11	345	392	99
0	12	12	373	346	99
0	12	13	383	424	99
0	12	14	455	532	99
0	12	15	145	234	99
0	13	1	1202	1179	99
0	13	2	1037	1021	99
0	13	3	-158	222	99
0	13	4	435	469	99
0	13	5	1242	1240	99
0	13	6	874	871	99
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0	13	8	545	594	99
0	13	9	451	462	99
0	13	10	320	272	99
0	13	11	-133	242	99
0	13	12	640	677	99
0	13	13	550	562	99
0	13	14	-316	39	99
0	13	15	248	81	99
0	14	0	1672	1572	99
0	14	1	998	928	99
0	14	2	160	265	99
0	14	3	1062	1047	99
0	14	4	640	627	99
0	14	5	-19	168	99
0	14	6	777	806	99
0	14	7	1002	1043	99
0	14	8	235	204	99
0	14	9	208	207	99
0	14	10	-156	368	99
0	14	11	712	652	99
0	14	12	-131	91	99
0	14	13	361	441	99
0	14	14	200	194	99
0	14	15	319	154	99
0	15	1	538	599	99
0	15	2	1422	1402	99
0	15	3	199	261	99
0	15	4	707	713	99
0	15	5	601	552	99
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0	15	13	-271	168	99
0	15	14	242	48	99
0	16	0	-158	152	99
0	16	1	1005	979	99
0	16	2	-247	157	99
0	16	3	1065	1041	99
0	16	4	350	314	99
0	16	5	261	108	99
0	16	6	-279	31	99
0	16	7	1245	1210	99
0	16	8	150	160	99
0	16	9	358	378	99
0	16	10	191	103	99
0	16	11	618	625	99
0	16	12	153	204	99
0	16	13	464	519	99
0	16	14	-154	9	99
0	17	1	201	251	99
0	17	2	1036	1003	99
0	17	3	67	63	99
0	17	4	470	495	99
0	17	5	365	315	99
0	17	6	954	956	99
0	17	7	-112	104	99
0	17	8	634	650	99
0	17	9	197	38	99
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0	18	9	177	43	99
0	18	10	334	348	99
0	18	11	270	559	99
0	18	12	242	228	99
0	19	1	359	316	99
0	19	2	365	488	99
0	19	3	-64	85	99
0	19	4	166	122	99
0	19	5	490	495	99
0	19	6	316	365	99
0	19	7	204	66	99
0	19	8	223	234	99
0	19	9	150	264	99
0	19	10	414	450	99
0	19	11	202	308	99
0	19	12	-232	265	99
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0	20	1	-144	139	99
0	20	2	-16	161	99
0	20	3	308	238	99
0	20	4	456	455	99
0	20	5	303	248	99
0	20	6	276	275	99
0	20	7	207	255	99
0	20	8	-194	221	99
0	20	9	-182	117	99
0	20	10	151	283	99
0	20	11	393	357	99
0	21	1	457	450	99
0	21	2	-161	88	99
0	21	3	226	213	99
0	21	4	-232	36	99
0	21	5	368	382	99
0	21	6	239	90	99
0	21	7	-114	89	99
0	17	3	67	63	99
0	17	4	470	495	99
0	17	5	365	315	99
0	17	6	954	956	99
0	17	7	-112	104	99
0	17	8	634	650	99
0	17	9	197	38	99
0	17	10	246	284	99
0	17	11	235	373	99
0	17	12	608	648	99
0	17	13	21	90	99
0	18	0	569	464	99
0	18	1	396	383	99
0	18	2	154	183	99
0	18	3	641	643	99
0	18	4	353	348	99
0	18	5	436	433	99
0	18	6	439	467	99
0	18	7	640	674	99
0	18	8	-118	59	99
0	18	9	177	43	99
0	18	10	334	348	99
0	18	11	270	559	99
0	18	12	242	228	99
0	19	1	359	316	99
0	19	2	365	488	99
0	19	3	-64	85	99
0	19	4	166	122	99
0	19	5	490	495	99
0	19	6	316	365	99
0	19	7	204	66	99
0	19	8	223	234	99
0	19	9	150	264	99
0	19	10	414	450	99
0	19	11	202	308	99
0	19	12	-232	265	99
0	20	0	551	581	99
0	20	1	-144	139	99
0	20	2	-16	161	99
0	20	3	308	238	99
0	20	4	456	455	99
0	20	5	303	248	99
0	20	6	276	275	99
0	20	7	207	255	99
0	20	8	-194	221	99
0	20	9	-182	117	99
0	20	10	151	283	99
0	20	11	393	357	99
0	21	1	457	450	99
0	21	2	-161	88	99
0	21	3	226	213	99
0	21	4	-232	36	99
0	21	5	368	382	99
0	21	6	239	90	99
0	21	7	-114	89	99
1	1	-9	1761	1748	99
1	1	-8	457	462	99
1	1	-7	685	714	99
1	1	-6	415	458	99
1	1	-5	2358	2389	99
1	1	-4	210	190	99
1	1	-3	1827	1904	99
1	1	-2	1199	1221	99
1	1	1	2441	2424	99
1	1	2	1419	1432	99
1	1	3	-101	86	99
1	1	4	982	1007	99
1	1	5	1975	1984	99
1	1	6	115	190	99
1	1	7	733	706	99
1	1	8	817	820	99
1	1	9	1046	980	99
1	1	10	488	485	99
1	1	11	991	1005	99
1	1	12	114	243	99
1	1	13	447	403	99
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1	1	15	601	610	99
1	1	16	237	189	99
1	1	17	-283	200	99
1	2	-18	151	416	99
1	2	-17	386	329	99
1	2	-16	218	61	99
1	2	-15	-398	209	99
1	2	-14	671	670	99
1	2	-13	59	46	99
1	2	-12	596	579	99
1	2	-11	926	934	99
1	2	-10	1047	1017	99
1	2	-9	-82	80	99
1	2	-8	1153	1163	99
1	2	-7	540	533	99
1	2	-6	203	198	99
1	2	-5	938	955	99
1	2	-4	2339	2378	99
1	2	-3	253	250	99
1	2	-2	160	166	99
1	2	-1	714	706	99
1	2	0	876	872	99
1	2	1	607	597	99
1	2	2	2122	2189	99
1	2	3	1255	1259	99
1	2	4	940	917	99
1	2	5	1061	1050	99
1	2	6	1121	1127	99
1	2	7	1100	1115	99
1	2	8	473	519	99
1	2	9	1064	1014	99
1	2	10	995	977	99
1	2	11	197	150	99
1	2	12	119	79	99
1	2	13	673	626	99
1	2	14	737	739	99
1	2	15	-295	285	99
1	2	16	369	385	99
1	2	17	219	167	99
1	3	-18	-157	85	99
1	3	-17	290	381	99
1	3	-16	447	514	99
1	3	-15	295	276	99
1	3	-14	617	543	99
1	3	-13	652	595	99
1	3	-12	591	576	99
1	3	-11	-123	151	99
1	3	-10	907	954	99
1	3	-9	1107	1085	99
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1	3	-7	586	600	99
1	3	-6	1844	1837	99
1	3	-5	637	628	99
1	3	-4	421	400	99
1	3	-3	1930	2011	99
1	3	-2	2165	2249	99
1	3	-1	637	647	99
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1	3	2	1630	1626	99
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1	3	13	456	486	99
1	3	14	417	500	99
1	3	15	666	657	99
1	3	16	-162	47	99
1	3	17	-151	210	99
1	4	-17	343	414	99
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1	4	-15	900	908	99
1	4	-14	452	467	99
1	4	-13	307	186	99
1	4	-12	154	61	99
1	4	-11	1285	1218	99
1	4	-10	258	217	99
1	4	-9	1017	1033	99
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1	4	-7	417	476	99
1	4	-6	446	450	99
1	4	-5	1702	1684	99
1	4	-4	1153	1130	99
1	4	-3	467	461	99
1	4	-2	315	291	99
1	4	-1	1463	1498	99
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1	4	5	984	958	99
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1	4	7	1285	1344	99
1	4	8	143	37	99
1	4	9	1106	1086	99
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1	4	12	115	108	99
1	4	13	999	1013	99
1	4	14	334	348	99
1	4	15	246	102	99
1	4	16	203	157	99
1	4	17	227	366	99
1	5	-17	200	63	99
1	5	-16	783	807	99
1	5	-15	256	96	99
1	5	-14	857	772	99
1	5	-13	248	182	99
1	5	-12	584	625	99
1	5	-11	-451	97	99
1	5	-10	1651	1547	99
1	5	-9	753	728	99
1	5	-8	-207	95	99
1	5	-7	312	347	99
1	5	-6	1518	1534	99
1	5	-5	524	505	99
1	5	-4	649	640	99
1	5	-3	722	741	99
1	5	-2	1646	1603	99
1	5	-1	1053	1019	99
1	5	0	1391	1335	99
1	5	1	305	292	99
1	5	2	1710	1696	99
1	5	3	443	428	99
1	5	4	2158	2164	99
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1	5	6	365	362	99
1	5	7	275	219	99
1	5	8	2050	2087	99
1	5	9	434	433	99
1	5	10	363	411	99
1	5	11	-241	96	99
1	5	12	720	700	99
1	5	13	-162	31	99
1	5	14	697	722	99
1	5	15	-115	149	99

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
2	18	2	833	814	99	2	21	-9	312	348	99	2	24	1	288	217	99	3	1	17	494	386	99	3	3	2	1080	1096	99
2	18	3	230	341	99	2	21	-8	-386	34	99	2	24	2	227	425	99	3	2	-17	-277	94	99	3	3	3	1643	1669	99
2	18	4	276	227	99	2	21	-7	743	698	99	2	24	3	238	265	99	3	2	-16	619	629	99	3	3	4	776	768	99
2	18	5	425	481	99	2	21	-6	-78	28	99	2	24	4	-129	4	99	3	2	-15	-253	77	99	3	3	5	328	339	99
2	18	6	762	743	99	2	21	-5	223	115	99	3	0	-16	475	499	99	3	2	-14	422	429	99	3	3	6	1192	1190	99
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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8	4	9	200	126	99	8	7	-8	386	394	99	8	9	3	643	651	99	8	12	-2	573	564	99	8	16	-4	85	48	99
8	4	10	225	5	99	8	7	-7	335	250	99	8	9	4	192	40	99	8	12	-1	154	197	99	8	16	-3	420	413	99
8	4	11	484	427	99	8	7	-6	-268	122	99	8	9	5	192	121	99	8	12	0	419	379	99	8	16	-2	196	62	99
8	5	-12	145	154	99	8	7	-5	206	220	99	8	9	6	176	204	99	8	12	1	69	331	99	8	16	-1	-233	215	99
8	5	-11	220	36	99	8	7	-4	516	552	99	8	9	7	407	402	99	8	12	2	646	671	99	8	16	0	185	103	99
8	5	-10	221	285	99	8	7	-3	166	161	99	8	9	8	-189	53	99	8	12	3	79	20	99	8	16	1	660	619	99
8	5	-9	290	132	99	8	7	-2	-166	275	99	8	9	9	473	460	99	8	12	4	404	435	99	8	16	2	-84	139	99
8	5	-8	575	507	99	8	7	-1	406	376	99	8	9	10	77	79	99	8	12	5	307	297	99	8	16	3	186	23	99
8	5	-7	-226	55	99	8	7	0	563	565	99	8	10	-10	486	545	99	8	12	6	-271	187	99	8	16	4	89	13	99
8	5	-6	293	262	99	8	7	1	12	53	99	8	10	-9	215	87	99	8	12	7	235	148	99	8	17	-3	-228	48	99
8	5	-5	207	18	99	8	7	2	639	651	99	8	10	-8	127	248	99	8	12	8	536	548	99	8	17	-2	308	103	99
8	5	-4	992	1003	99	8	7	3	405	417	99	8	10	-7	203	22	99	8	13	-8	334	375	99	8	17	-1	-378	129	99
8	5	-3	227	183	99	8	7	4	207	213	99	8	10	-6	613	600	99	8	13	-7	369	275	99	8	17	0	479	475	99
8	5	-2	201	287	99	8	7	5	178	234	99	8	10	-5	297	178	99	8	13	-6	323	260	99	8	17	1	-123	33	99
8	5	-1	-180	14	99	8	7	6	419	460	99	8	10	-4	-133	161	99	8	13	-5	402	355	99	9	0	-10	477	501	99
8	5	0	849	841	99	8	7	7	174	193	99	8	10	-3	139	120	99	8	13	-4	511	535	99	9	0	-8	180	138	99
8	5	1	284	244	99	8	7	8	167	118	99	8	10	-2	668	653	99	8	13	-3	327	313	99	9	0	-6	412	405	99
8	5	2	905	922	99	8	7	9	385	331	99	8	10	-1	-252	68	99	8	13	-2	185	121	99	9	0	-4	413	307	99
8	5	3	229	191	99	8	7	10	365	346	99	8	10	0	146	293	99	8	13	-1	371	454	99	9	0	-2	410	361	99
8	5	4	268	290	99	8	8	-11	-91	183	99	8	10	1	42	270	99	8	13	0	463	531	99	9	0	0	578	609	99
8	5	5	-177	112	99	8	8	-10	230	299	99	8	10	2	454	474	99	8	13	1	291	290	99	9	0	2	363	384	99
8	5	6	684	680	99	8	8	-9	371	360	99	8	10	3	141	16	99	8	13	2	306	414	99	9	0	4	601	592	99
8	5	7	136	169	99	8	8	-8	-54	57	99	8	10	4	529	511	99	8	13	3	449	405	99	9	0	6	38	173	99
8	5	8	154	240	99	8	8	-7	59	128	99	8	10	5	-147	70	99	8	13	4	25	235	99	9	0	8	584	545	99
8	5	9	135	72	99	8	8	-6	297	330	99	8	10	6	170	228	99	8	13	5	162	163	99	9	1	-11	371	328	99
8	5	10	324	389	99	8	8	-5	268	253	99	8	10	7	-261	80	99	8	13	6	200	430	99	9	1	-10	-243	60	99
8	5	11	324	214	99	8	8	-4	-104	208	99	8	10	8	590	597	99	8	13	7	469	467	99	9	1	-9	471	335	99
8	6	-12	-232	61	99	8	8	-3	531	542	99	8	10	9	-296	40	99	8	14	-8	-272	18	99	9	1	-8	-119	71	99
8	6	-11	171	88	99	8	8	-2	503	491	99	8	11	-10	-118	35	99	8	14	-7	-329	182	99	9	1	-7	-377	183	99
8	6	-10	184	54	99	8	8	-1	146	184	99	8	11	-9	-182	128	99	8	14	-6	269	247	99	9	1	-6	197	205	99
8	6	-9	404	440	99	8	8	0	30	219	99	8	11	-8	-54	125	99	8	14	-5	559	534	99	9	1	-5	465	472	99
8	6	-8	105	41	99	8	8	1	482	503	99	8	11	-7	520	528	99	8	14	-4	-191	29	99	9	1	-4	-251	109	99
8	6	-7	209	206	99	8	8	2	343	317	99	8	11	-6	49	24	99	8	14	-3	507	418	99	9	1	-3	-63	152	99
8	6	-6	76	214	99	8	8	3	178	125	99	8	11	-5	497	456	99	8	14	-2	263	273	99	9	1	-2	117	184	99
8	6	-5	529	515	99	8	8	4	476	467	99	8	11	-4	-52	113	99	8	14	-1	319	264	99	9	1	-1	631	631	99
8	6	-4	173	29	99	8	8	5	230	260	99	8	11	-3	342	371	99	8	14	0	-41	36	99	9	1	0	201	42	99
8	6	-3	803	815	99	8	8	6	192	116	99	8	11	-2	-346	40	99	8	14	1	756	671	99	9	1	1	201	87	99
8	6	-2	164	118	99	8	8	7	99	198	99	8	11	-1	633	648	99	8	14	2	347	332	99	9	1	2	145	93	99
8	6	-1	423	413	99	8	8	8	214	411	99	8	11	0	121	57	99	8	14	3	-166	57	99	9	1	3	556	559	99
8	6	0	168	222	99	8	8	9	-259	205	99	8	11	1	155	108	99	8	14	4	-290	200	99	9	1	4	95	8	99
8	6	1	813	835	99	8	8	10	249	118	99	8	11	2	248	98	99	8	14	5	358	403	99	9	1	5	462	487	99
8	6	2	157	42	99	8	9	-11	174	364	99	8	11	3	746	737	99	8	14	6	82	237	99	9	1	6	172	184	99
8	6	3	400	401	99	8	9	-10	-197	95	99	8	11	4	300	244	99	8	15	-6	125	106	99	9	1	7	241	292	99
8	6	4	342	322	99	8	9	-9	225	284	99	8	11	5	247	197	99	8	15	-5	92	67	99	9	1	8	114	21	99
8	6	5	622	581	99	8	9	-8	143	128	99	8	11	6	-163	63	99	8	15	-4	621	616	99	9	1	9	446	428	99
8	6	6	76	81	99	8	9	-7	213	387	99	8	11	7	502	508	99	8	15	-3	116	58	99	9	2	-11	-306	259	99
8	6	7	523	486	99	8	9	-6	-293	14	99	8	11	8	-187	31	99	8	15	-2	-77	135	99	9	2	-10	294	332	99
8	6	8	140	84	99	8	9	-5	310	405	99	8	11	9	412	415	99	8	15	-1	146	66	99	9	2	-9	112	191	99
8	6	9	245	232	99	8	9	-4	411	406	99	8	12	-9	411	384	99	8	15	0	626	628	99	9	2	-8	219	115	99
8	6	10	64	309	99	8	9	-3	308	283	99	8	12	-8	131	215	99	8	15	1	123	123	99	9	2	-7	437	465	99
8	6	11	361	267	99	8	9	-2	192	178	99	8	12	-7	-203	27	99	8	15	2	-156	352	99	9	2	-6	504	462	99
8	7	-12	222	192	99	8	9	-1	430	445	99	8	12	-6	566	594	99	8	15	3	138	211	99	9	2	-5	-292	18	99

h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
9	2	-4	-276	145	99	9	5	-9	-269	58	99	9	7	8	-215	178	99	9	11	-1	542	514	99	10	1	4	117	95	99
9	2	-3	236	416	99	9	5	-8	815	779	99	9	8	-9	132	99	99	9	11	0	159	74	99	10	1	5	320	397	99
9	2	-2	396	403	99	9	5	-7	-253	5	99	9	8	-8	182	195	99	9	11	1	308	269	99	10	1	6	170	23	99
9	2	-1	336	299	99	9	5	-6	434	407	99	9	8	-7	528	541	99	9	11	2	-179	36	99	10	2	-8	213	245	99
9	2	0	517	526	99	9	5	-5	212	31	99	9	8	-6	274	313	99	9	11	3	482	502	99	10	2	-7	237	247	99
9	2	1	386	380	99	9	5	-4	535	557	99	9	8	-5	-244	145	99	9	11	4	181	62	99	10	2	-6	208	229	99
9	2	2	181	273	99	9	5	-3	-52	4	99	9	8	-4	412	418	99	9	11	5	464	489	99	10	2	-5	209	314	99
9	2	3	231	159	99	9	5	-2	837	853	99	9	8	-3	464	497	99	9	11	6	193	69	99	10	2	-4	468	433	99
9	2	4	576	601	99	9	5	-1	163	43	99	9	8	-2	287	236	99	9	12	-6	410	319	99	10	2	-3	-90	238	99
9	2	5	149	104	99	9	5	0	212	192	99	9	8	-1	227	299	99	9	12	-5	145	142	99	10	2	-2	121	79	99
9	2	6	340	185	99	9	5	1	116	110	99	9	8	0	303	387	99	9	12	-4	-202	205	99	10	2	-1	307	247	99
9	2	7	290	351	99	9	5	2	756	751	99	9	8	1	345	257	99	9	12	-3	202	209	99	10	2	0	499	488	99
9	2	8	349	379	99	9	5	3	-172	50	99	9	8	2	-179	11	99	9	12	-2	200	281	99	10	2	1	-190	28	99
9	2	9	234	147	99	9	5	4	202	123	99	9	8	3	456	395	99	9	12	-1	183	35	99	10	2	2	290	361	99
9	3	-10	135	6	99	9	5	5	-293	42	99	9	8	4	363	417	99	9	12	0	475	482	99	10	2	3	77	250	99
9	3	-9	307	243	99	9	5	6	544	528	99	9	8	5	316	265	99	9	12	1	-183	203	99	10	2	4	-125	182	99
9	3	-8	574	558	99	9	5	7	-179	59	99	9	8	6	32	145	99	9	12	2	227	203	99	10	2	5	191	90	99
9	3	-7	201	281	99	9	5	8	313	313	99	9	8	7	281	299	99	9	12	3	208	177	99	10	2	6	197	196	99
9	3	-6	55	272	99	9	5	9	-283	8	99	9	9	-8	-216	226	99	9	12	4	521	521	99	10	3	-8	267	246	99
9	3	-5	279	216	99	9	6	-10	161	241	99	9	9	-7	163	134	99	9	12	5	-131	101	99	10	3	-7	238	20	99
9	3	-4	511	444	99	9	6	-9	220	304	99	9	9	-6	356	315	99	9	13	-5	343	323	99	10	3	-6	500	487	99
9	3	-3	139	49	99	9	6	-8	-238	55	99	9	9	-5	-99	411	99	9	13	-4	158	309	99	10	3	-5	188	246	99
9	3	-2	522	550	99	9	6	-7	758	734	99	9	9	-4	240	226	99	9	13	-3	188	85	99	10	3	-4	-152	99	99
9	3	-1	555	545	99	9	6	-6	-341	226	99	9	9	-3	-59	166	99	9	13	-2	184	229	99	10	3	-3	133	278	99
9	3	0	143	194	99	9	6	-5	204	90	99	9	9	-2	366	289	99	9	13	-1	330	271	99	10	3	-2	379	411	99
9	3	1	150	137	99	9	6	-4	-210	116	99	9	9	-1	514	520	99	9	13	0	-45	120	99	10	3	-1	168	212	99
9	3	2	555	547	99	9	6	-3	779	822	99	9	9	0	140	28	99	9	13	1	287	84	99	10	3	0	-200	262	99
9	3	3	475	475	99	9	6	-2	190	150	99	9	9	1	221	243	99	9	13	2	371	350	99	10	3	1	395	425	99
9	3	4	-125	87	99	9	6	-1	314	362	99	9	9	2	355	334	99	9	13	3	185	302	99	10	3	2	310	383	99
9	3	5	210	335	99	9	6	0	-87	259	99	9	9	3	343	319	99	9	14	-3	257	383	99	10	3	3	-244	49	99
9	3	6	499	491	99	9	6	1	407	358	99	9	9	4	143	69	99	9	14	-2	188	82	99	10	3	4	331	284	99
9	3	7	160	85	99	9	6	2	-207	9	99	9	9	5	466	444	99	9	14	-1	149	81	99	10	3	5	326	315	99
9	3	8	174	192	99	9	6	3	497	577	99	9	9	6	303	277	99	9	14	0	157	111	99	10	3	6	203	208	99
9	3	9	-202	308	99	9	6	4	-212	207	99	9	9	7	-237	89	99	9	14	1	334	279	99	10	4	-7	473	477	99
9	4	-10	260	123	99	9	6	5	308	320	99	9	10	-8	132	207	99	9	14	2	54	83	99	10	4	-6	-314	92	99
9	4	-9	389	452	99	9	6	6	186	155	99	9	10	-7	-49	184	99	10	0	-8	208	187	99	10	4	-5	-111	251	99
9	4	-8	86	29	99	9	6	7	496	469	99	9	10	-6	401	349	99	10	0	-6	-171	81	99	10	4	-4	198	159	99
9	4	-7	693	644	99	9	6	8	52	81	99	9	10	-5	152	38	99	10	0	-4	627	648	99	10	4	-3	112	418	99
9	4	-6	190	115	99	9	7	-9	-75	312	99	9	10	-4	357	359	99	10	0	-2	-85	113	99	10	4	-2	285	5	99
9	4	-5	146	122	99	9	7	-8	691	627	99	9	10	-3	177	138	99	10	0	0	438	461	99	10	4	-1	449	471	99
9	4	-4	-189	122	99	9	7	-7	254	183	99	9	10	-2	302	352	99	10	0	2	507	524	99	10	4	0	188	207	99
9	4	-3	753	775	99	9	7	-6	468	431	99	9	10	-1	131	173	99	10	0	4	-244	147	99	10	4	1	157	214	99
9	4	-2	327	287	99	9	7	-5	209	346	99	9	10	0	447	482	99	10	0	6	461	460	99	10	4	2	224	175	99
9	4	-1	420	420	99	9	7	-4	138	247	99	9	10	1	-156	167	99	10	1	-8	-331	19	99	10	4	3	432	478	99
9	4	0	40	54	99	9	7	-3	-107	127	99	9	10	2	149	34	99	10	1	-7	121	202	99	10	4	4	187	125	99
9	4	1	681	674	99	9	7	-2	748	772	99	9	10	3	-171	147	99	10	1	-6	287	269	99	10	4	5	143	20	99
9	4	2	-260	13	99	9	7	-1	509	425	99	9	10	4	719	686	99	10	1	-5	560	514	99	10	4	6	173	107	99
9	4	3	541	519	99	9	7	0	-268	63	99	9	10	5	156	86	99	10	1	-4	-165	5	99	10	5	-7	143	32	99
9	4	4	365	353	99	9	7	1	196	216	99	9	10	6	297	182	99	10	1	-3	352	384	99	10	5	-6	476	530	99
9	4	5	-205	260	99	9	7	2	405	435	99	9	11	-7	171	134	99	10	1	-2	190	163	99	10	5	-5	181	89	99
9	4	6	189	72	99	9	7	3	250	227	99	9	11	-6	182	131	99	10	1	-1	127	191	99	10	5	-4	-253	123	99
9	4	7	464	508	99	9	7	4	166	137	99	9	11	-5	392	420	99	10	1	0	-142	92	99	10	5	-3	-122	76	99
9	4	8	-193	71	99	9	7	5	170	330	99	9	11	-4	-67	29	99	10	1	1	677	675	99	10	5	-2	546	576	99
9	4	9	152	10	99	9	7	6	386	407	99	9	11	-3	196	68	99	10	1	2	208	99	99	10	5	-1	57	10	99
9	5	-10	-153	7	99	9	7	7	-251	28	99	9	11	-2	146	13	99	10	1	3	109	154	99	10	5	0	198	156	99

h	k	l	Fo	Fc	sigF2
10	5	1	-272	19	99
10	5	2	481	490	99
10	5	3	-236	64	99
10	5	4	351	341	99
10	5	5	60	11	99
10	6	-6	196	111	99
10	6	-5	-27	255	99
10	6	-4	-67	118	99
10	6	-3	422	421	99
10	6	-2	296	85	99
10	6	-1	398	392	99
10	6	0	-283	159	99
10	6	1	241	214	99
10	6	2	-102	72	99
10	6	3	501	483	99
10	6	4	146	172	99
10	6	5	197	60	99
10	7	-6	252	434	99
10	7	-5	325	213	99
10	7	-4	-265	45	99
10	7	-3	-151	130	99
10	7	-2	478	519	99
10	7	-1	196	218	99
10	7	0	-177	72	99
10	7	1	78	247	99
10	7	2	410	359	99
10	7	3	171	34	99
10	7	4	213	192	99
10	8	-5	174	230	99
10	8	-4	135	336	99
10	8	-3	25	269	99
10	8	-2	197	17	99
10	8	-1	356	318	99
10	8	0	219	384	99
10	8	1	314	147	99
10	8	2	-94	218	99
10	8	3	330	302	99
10	8	4	271	223	99
10	9	-4	-308	123	99
10	9	-3	443	344	99
10	9	-2	181	270	99
10	9	-1	299	306	99
10	9	0	166	72	99
10	9	1	306	403	99
10	9	2	331	303	99
10	9	3	-398	15	99
10	10	-3	52	173	99
10	10	-2	-77	21	99
10	10	-1	-90	116	99
10	10	0	446	455	99
10	10	1	291	188	99

APPENDIX 7.IX

$[Hg(S_2CNnBu_2)_2]$

Table 7.IX.1: Crystallographic Parameters for $[Hg(S_2CN(CH_2CH_2CH_2CH_3)2)2]$

Formula	$C_{18}H_{36}HgN_2S_4$
Formula weight	609.33
Crystal morphology / colour	block / pale yellow
Crystal dimensions (mm)	0.32 x 0.36 x 0.42
Crystal system	monoclinic
Space group	*C*2/*c* (# 15)
a (Å)	23.210(6)
b (Å)	16.48(1)
c (Å)	16.285(6)
α (°)	90
β (°)	126.81(2)
γ (°)	90
V (Å^3)	4987(3)
Z	8
$\rho_{calc.}$ (g cm^{-3})	1.623
F(000)	2416
Temperature (°K)	200
μ (cm^{-1})	65.28
θ limits, cell (°)	18.4 – 20.7
θ limits, data (°)	3.0 – 27.5
hkl range	0 – 30, 0 – 21, -21 – 14
Range of transmission factors	0.966 – 1
Scan technique	ω:2θ
No. of data measured	6135
No. of unique data	5990
R_{amal}	0.103
No. of parameters	206
R (I≥0σ(I))	0.157
R_w (I≥0σ(I))	0.141
R (I≥3σ(I))	0.065
R_w (I≥3σ(I))	0.067
Maximum and minimum residual electron density (e Å^{-3})	2.99, -4.12

To refine the structure of $[Hg(S_2CNnBu_2)_2]$ the general procedure outlined in chapter 2 was followed but significant thermal motion was found, associated with some of the n-butyl groups. Despite the large thermal motion, multiple sites for these atoms were not located suggesting that the disorder is dynamic in nature. The carbon atoms of the C(11)-C(14) butyl group was refined isotropically with restrained C-C bond distances of 1.50(2) Å.

Table 7.IX.2: Fractional atomic coordinates for the non-hydrogen atoms in $[Hg(S_2CN(CH_2CH_2CH_2CH_3)_2)_2]$

Atom	*x*	*y*	*z*
Hg	0.10216(2)	0.06498(2)	0.30871(3)
S(1)	0.0551(1)	-0.0585(1)	0.2032(2)
S(2)	-0.0480(1)	0.0797(1)	0.0911(2)
S(3)	0.1419(1)	0.1957(1)	0.2731(2)
S(4)	0.2412(1)	0.0871(2)	0.4481(2)
N(1)	-0.0642(3)	-0.0700(4)	0.0160(5)
N(2)	0.2828(4)	0.2226(5)	0.4106(6)
C(1)	-0.0228(4)	-0.0210(5)	0.0966(6)
C(2)	-0.0424(5)	-0.1559(5)	0.0196(7)
C(3)	-0.0648(5)	-0.2130(6)	0.0697(8)
C(4)	-0.0348(8)	-0.2950(7)	0.079(1)
C(5)	-0.0669(8)	-0.3590(8)	0.103(1)
C(6)	-0.1337(5)	-0.0431(5)	-0.0809(6)
C(7)	-0.1988(5)	-0.0638(5)	-0.0869(6)
C(8)	-0.2667(5)	-0.0372(6)	-0.1860(8)
C(9)	-0.3333(5)	-0.0622(7)	-0.1996(9)
C(10)	0.2268(5)	0.1730(5)	0.3785(6)
C(11)	0.3533(6)	0.2132(7)	0.5073(9)
C(12)	0.3985(8)	0.1567(8)	0.497(1)
C(13)	0.4661(10)	0.163(1)	0.608(1)
C(14)	0.511(1)	0.094(1)	0.617(2)
C(15)	0.2723(5)	0.2965(5)	0.3509(7)

Table 7.IX.2: Cont.

Atom	*x*	*y*	*z*
C(16)	0.2812(6)	0.2770(6)	0.2639(8)
C(17)	0.2653(5)	0.3472(6)	0.1977(8)
C(18)	0.2770(6)	0.3290(7)	0.1173(8)

Table 7.IX.3: Anisotropic thermal parameters ($Å^2$) for the non-hydrogen atoms in $[Hg(S_2CN(CH_2CH_2CH_2CH_3)_2)_2]$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Hg	0.0724(2)	0.0644(2)	0.0645(2)	-0.0163(2)	0.0494(2)	-0.0046(2)
S(1)	0.058(1)	0.053(1)	0.066(1)	0.005(1)	0.041(1)	-0.001(1)
S(2)	0.074(2)	0.049(1)	0.064(1)	0.008(1)	0.052(1)	0.008(1)
S(3)	0.076(2)	0.050(1)	0.069(2)	-0.007(1)	0.051(1)	0.001(1)
S(4)	0.069(2)	0.082(2)	0.076(2)	-0.013(1)	0.040(1)	0.019(1)
N(1)	0.068(4)	0.055(3)	0.055(3)	-0.012(3)	0.049(3)	-0.004(3)
N(2)	0.087(5)	0.074(5)	0.071(5)	-0.026(4)	0.056(4)	-0.009(4)
C(1)	0.058(4)	0.050(3)	0.058(4)	-0.004(3)	0.048(2)	-0.004(3)
C(2)	0.089(7)	0.056(4)	0.067(6)	-0.002(5)	0.053(5)	-0.006(5)
C(3)	0.079(7)	0.065(5)	0.089(7)	-0.016(5)	0.056(6)	0.005(6)
C(4)	0.21(1)	0.051(6)	0.22(2)	0.019(7)	0.16(1)	0.012(9)
C(5)	0.24(2)	0.080(10)	0.27(2)	-0.014(10)	0.18(2)	0.04(1)
C(6)	0.076(4)	0.071(7)	0.048(4)	-0.013(5)	0.038(4)	-0.001(4)
C(7)	0.074(5)	0.050(5)	0.062(5)	-0.005(5)	0.045(4)	-0.003(5)
C(8)	0.071(5)	0.079(7)	0.082(7)	0.004(5)	0.031(5)	0.010(6)
C(9)	0.070(7)	0.118(10)	0.14(1)	-0.015(7)	0.057(7)	-0.020(9)
C(10)	0.078(3)	0.067(5)	0.057(5)	-0.009(3)	0.053(3)	-0.012(3)
C(15)	0.085(7)	0.058(5)	0.076(7)	-0.025(5)	0.049(6)	-0.011(4)
C(16)	0.124(9)	0.071(6)	0.097(8)	-0.013(6)	0.095(8)	-0.018(5)
C(17)	0.112(8)	0.079(7)	0.091(8)	-0.032(6)	0.079(7)	-0.018(5)
C(18)	0.156(10)	0.129(10)	0.093(8)	-0.044(9)	0.110(8)	-0.038(7)

Table 7.IX.4: Fractional atomic coordinates and B_{eq} values ($Å^2$) for the hydrogen atoms in [Hg($S_2CN(CH_2CH_2CH_2CH_3)_2)_2$]*

Atom	x	y	z	B_{eq}
H(2a)	0.0094	-0.1580	0.0581	6.2566
H(2b)	-0.0649	-0.1743	-0.0502	6.2566
H(3a)	-0.1169	-0.2162	0.0277	7.1198
H(3b)	-0.0463	-0.1930	0.1372	7.1198
H(4a)	0.0170	-0.2934	0.1320	13.3150
H(4b)	-0.0435	-0.3083	0.0135	13.3150
H(5a)	-0.1182	-0.3609	0.0502	16.2200
H(5b)	-0.0455	-0.4108	0.1081	16.2200
H(5c)	-0.0574	-0.3462	0.1687	16.2200
H(6a)	-0.1383	-0.0688	-0.1384	6.1330
H(6b)	-0.1320	0.0154	-0.0866	6.1330
H(7a)	-0.1955	-0.0371	-0.0310	5.6765
H(7b)	-0.2006	-0.1221	-0.0804	5.6765
H(8a)	-0.2674	-0.0603	-0.2416	8.2625
H(8b)	-0.2661	0.0215	-0.1895	8.2625
H(9a)	-0.3342	-0.0382	-0.1459	10.5090
H(9b)	-0.3751	-0.0449	-0.2659	10.5090
H(9c)	-0.3341	-0.1212	-0.1948	10.5090
H(11a)	0.3481	0.1924	0.5580	9.6208
H(11b)	0.3765	0.2662	0.5290	9.6208
H(12a)	0.3789	0.1034	0.4771	13.7582
H(12b)	0.4073	0.1758	0.4498	13.7582

Table 7.IX.4: Cont.

Atom	x	y	z	B_{eq}
H(13a)	0.4872	0.2131	0.6210	25.4524
H(13b)	0.4514	0.1540	0.6523	25.4524
H(14a)	0.4887	0.0447	0.6051	28.7888
H(14b)	0.5246	0.1039	0.5742	28.7888
H(14c)	0.5553	0.0950	0.6885	28.7888
H(15a)	0.2242	0.3176	0.3194	6.3635
H(15b)	0.3075	0.3370	0.3968	6.3635
H(16a)	0.3306	0.2604	0.2961	7.7060
H(16b)	0.2489	0.2332	0.2219	7.7060
H(17a)	0.2153	0.3625	0.1628	7.8232
H(17b)	0.2961	0.3919	0.2396	7.8232
H(18a)	0.3270	0.3153	0.1506	9.8564
H(18b)	0.2647	0.3768	0.0743	9.8564
H(18c)	0.2466	0.2841	0.0747	9.8564

[k] H atoms were included in their calculated positions, C-H 0.97 Å.

Table 7.IX.5: Bond distances (Å) for [Hg($S_2CN(CH_2CH_2CH_2CH_3)_2$)2]

Hg — S(1)	2.457(3)	N(2) — C(15)	1.49(1)
Hg — S(2)	3.157(3)	C(2) — C(3)	1.53(2)
Hg — S(2)*	2.601(3)	C(3) — C(4)	1.49(2)
Hg — S(3)	2.544(3)	C(4) — C(5)	1.48(2)
Hg — S(4)	2.631(3)	C(6) — C(7)	1.49(2)
S(1) — C(1)	1.70(1)	C(7) — C(8)	1.49(2)
S(2) — C(1)	1.74(1)	C(8) — C(9)	1.48(2)
S(3) — C(10)	1.71(1)	C(11) — C(12)	1.49(1)
S(4) — C(10)	1.72(1)	C(12) — C(13)	1.54(1)
N(1) — C(1)	1.34(1)	C(13) — C(14)	1.48(1)
N(1) — C(2)	1.49(1)	C(15) — C(16)	1.58(2)
N(1) — C(6)	1.50(1)	C(16) — C(17)	1.47(2)
N(2) — C(10)	1.35(1)	C(17) — C(18)	1.52(2)
N(2) — C(11)	1.45(2)		

* symmetry operation: $-x$, y, $0.5 - z$

Table 7.IX.6: Bond angles (deg.) for $[Hg(S_2CN(CH_2CH_2CH_2CH_3)2)2]$

S(1) — Hg — S(2)	63.21(9)	C(10) — N(2) — C(15)	120(1)
S(1) — Hg — S(2)*	109.65(9)	C(11) — N(2) — C(15)	116(1)
S(1) — Hg — S(3)	127.7(1)	S(1) — C(1) — S(2)	121.0(7)
S(1) — Hg — S(4)	121.3(1)	S(1) — C(1) — N(1)	118.9(8)
S(2) — Hg — S(2)*	94.23(9)	S(2) — C(1) — N(1)	120.1(9)
S(2) — Hg — S(3)	89.21(9)	N(1) — C(2) — C(3)	113(1)
S(2) — Hg — S(4)	156.41(9)	C(2) — C(3) — C(4)	109(1)
S(2)* — Hg — S(3)	116.2(1)	C(3) — C(4) — C(5)	113(2)
S(2)* — Hg — S(4)	104.5(1)	N(1) — C(6) — C(7)	114.2(9)
S(3) — Hg — S(4)	69.8(1)	C(6) — C(7) — C(8)	112(1)
Hg — S(1) — C(1)	99.7(4)	C(7) — C(8) — C(9)	114(1)
Hg — S(2) — Hg*	84.94(8)	S(3) — C(10) — S(4)	119.7(7)
Hg — S(2) — C(1)	76.0(4)	S(3) — C(10) — N(2)	121.4(9)
Hg — S(2)* — C(1)*	98.8(3)	S(4) — C(10) — N(2)	118.9(9)
Hg — S(3) — C(10)	86.7(4)	N(2) — C(11) — C(12)	111(1)
Hg — S(4) — C(10)	83.8(4)	C(11) — C(12) — C(13)	96(2)
C(1) — N(1) — C(2)	120(1)	C(12) — C(13) — C(14)	103(2)
C(1) — N(1) — C(6)	123(1)	N(2) — C(15) — C(16)	111(1)
C(2) — N(1) — C(6)	116.6(9)	C(15) — C(16) — C(17)	113(1)
C(10) — N(2) — C(11)	124(1)	C(16) — C(17) — C(18)	112(1)

* symmetry operation: - x, y, 0.5 - z

Table 7.IX.7: Observed and calculated structure factors for $[Hg(S_2CN(CH_2CH_2CH_2CH_3)_2)_2]$

h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF	h	k	l	Fo	Fc	sigF
0	0	2	2993	3291	99	0	6	15	344	211	99	0	14	6	381	371	99	1	1	6	2720	2757	99	1	5	-6	195	130	99
0	0	4	387	394	99	0	6	16	-369	236	99	0	14	7	209	333	99	1	1	7	1071	1002	99	1	5	-5	1344	1327	99
0	0	6	840	876	99	0	8	0	6454	6383	99	0	14	8	819	873	99	1	1	8	2237	2214	99	1	5	-4	493	459	99
0	0	8	3247	3178	99	0	8	1	1025	1015	99	0	14	9	120	259	99	1	1	9	866	828	99	1	5	-3	2505	2577	99
0	0	10	2532	2500	99	0	8	2	2960	3066	99	0	14	10	628	614	99	1	1	10	1358	1357	99	1	5	-2	1370	1358	99
0	0	12	377	361	99	0	8	3	518	447	99	0	14	11	-280	101	99	1	1	11	-109	215	99	1	5	-1	3626	3699	99
0	0	14	235	262	99	0	8	4	569	606	99	0	14	12	-196	46	99	1	1	12	391	368	99	1	5	0	2053	1997	99
0	0	16	360	338	99	0	8	5	634	582	99	0	16	0	1216	1150	99	1	1	13	228	180	99	1	5	1	1445	1396	99
0	2	2	2633	2457	99	0	8	6	1119	1089	99	0	16	1	592	568	99	1	1	14	595	597	99	1	5	2	496	467	99
0	2	3	328	287	99	0	8	7	216	66	99	0	16	2	1186	1176	99	1	1	15	-296	139	99	1	5	3	1974	1871	99
0	2	4	232	199	99	0	8	8	2008	2096	99	0	16	3	-100	137	99	1	1	16	67	193	99	1	5	4	508	473	99
0	2	5	372	294	99	0	8	9	490	493	99	0	16	4	316	228	99	1	1	-17	236	304	99	1	5	5	2441	2404	99
0	2	6	1551	1545	99	0	8	10	1401	1460	99	0	16	5	348	230	99	1	1	-16	333	76	99	1	5	6	1683	1657	99
0	2	7	1358	1469	99	0	8	11	274	252	99	0	16	6	415	513	99	1	1	-15	203	32	99	1	5	7	2281	2289	99
0	2	8	2826	2794	99	0	8	12	313	291	99	0	16	7	47	27	99	1	1	-14	-334	29	99	1	5	8	581	488	99
0	2	9	1962	1942	99	0	8	13	116	17	99	0	16	8	684	620	99	1	1	-13	600	550	99	1	5	9	1091	1043	99
0	2	10	1595	1607	99	0	8	14	308	114	99	0	16	9	-409	281	99	1	1	-12	305	252	99	1	5	10	453	356	99
0	2	11	321	431	99	0	8	15	-232	13	99	0	16	10	369	448	99	1	1	-11	993	976	99	1	5	11	-185	161	99
0	2	12	334	196	99	0	10	0	1509	1527	99	0	16	11	237	81	99	1	1	-10	1344	1233	99	1	5	12	-228	207	99
0	2	13	-306	116	99	0	10	1	2576	2555	99	0	18	0	-284	169	99	1	1	-9	2899	2896	99	1	5	13	533	727	99
0	2	14	317	270	99	0	10	2	1188	1399	99	0	18	1	594	605	99	1	1	-8	1352	1442	99	1	5	14	337	344	99
0	2	15	227	340	99	0	10	3	410	396	99	0	18	2	369	335	99	1	1	-7	355	276	99	1	5	15	360	426	99
0	2	16	181	286	99	0	10	4	-299	62	99	0	18	3	209	179	99	1	1	-6	458	498	99	1	5	16	328	156	99
0	4	0	1008	901	99	0	10	5	-207	96	99	0	18	4	-79	68	99	1	1	-5	1076	1188	99	1	7	-16	-437	213	99
0	4	1	5107	4968	99	0	10	6	1227	1244	99	0	18	5	-308	146	99	1	1	-4	160	153	99	1	7	-15	-177	51	99
0	4	2	870	795	99	0	10	7	838	823	99	0	18	6	351	392	99	1	1	-3	1094	1166	99	1	7	-14	-402	200	99
0	4	3	385	308	99	0	10	8	1108	1082	99	0	18	7	-174	316	99	1	1	-2	630	473	99	1	7	-13	-310	41	99
0	4	4	271	77	99	0	10	9	1164	1178	99	0	18	8	234	240	99	1	1	-1	4486	4611	99	1	7	-12	642	660	99
0	4	5	985	953	99	0	10	10	935	934	99	0	18	9	332	361	99	1	1	0	2803	2706	99	1	7	-11	192	64	99
0	4	6	391	342	99	0	10	11	232	299	99	0	20	0	155	108	99	1	1	1	2836	2642	99	1	7	-10	1884	1902	99
0	4	7	2692	2714	99	0	10	12	60	87	99	0	20	1	-258	222	99	1	1	2	980	916	99	1	7	-9	-128	81	99
0	4	8	-344	125	99	0	10	13	-183	52	99	0	20	2	-133	59	99	1	1	3	2128	2066	99	1	7	-8	1576	1546	99
0	4	9	2448	2391	99	0	10	14	323	125	99	0	20	3	327	211	99	1	1	4	1251	1204	99	1	7	-7	388	408	99
0	4	10	-360	72	99	0	10	15	-273	220	99	0	20	4	421	125	99	1	1	5	2680	2623	99	1	7	-6	697	608	99
0	4	11	576	495	99	0	12	0	485	516	99	0	20	5	-146	224	99	1	1	6	706	670	99	1	7	-5	740	725	99
0	4	12	-440	196	99	0	12	1	1638	1446	99	0	20	6	-250	60	99	1	1	7	2269	2224	99	1	7	-4	1454	1509	99
0	4	13	279	265	99	0	12	2	293	362	99	1	1	-17	-479	105	99	1	1	8	538	372	99	1	7	-3	1710	1701	99
0	4	14	-371	69	99	0	12	3	289	158	99	1	1	-16	233	263	99	1	1	9	1201	1145	99	1	7	-2	4268	4250	99
0	4	15	-147	452	99	0	12	4	208	179	99	1	1	-15	261	108	99	1	1	10	139	263	99	1	7	-1	189	164	99
0	4	16	-319	42	99	0	12	5	770	824	99	1	1	-14	383	257	99	1	1	11	-243	130	99	1	7	0	4732	4864	99
0	6	0	4450	4660	99	0	12	6	258	32	99	1	1	-13	-39	64	99	1	1	12	147	180	99	1	7	1	773	844	99
0	6	1	3090	3105	99	0	12	7	885	890	99	1	1	-12	850	863	99	1	1	13	897	926	99	1	7	2	998	1011	99
0	6	2	1918	1765	99	0	12	8	271	176	99	1	1	-11	96	251	99	1	1	14	321	214	99	1	7	3	302	149	99
0	6	3	1257	1112	99	0	12	9	1162	1114	99	1	1	-10	2318	2402	99	1	1	15	361	548	99	1	7	4	744	818	99
0	6	4	400	348	99	0	12	10	263	228	99	1	1	-9	652	682	99	1	1	16	273	130	99	1	7	5	844	885	99
0	6	5	-103	395	99	0	12	11	-170	223	99	1	1	-8	2409	2395	99	1	5	-16	251	81	99	1	7	6	2223	2178	99
0	6	6	787	759	99	0	12	12	-426	85	99	1	1	-7	429	393	99	1	5	-15	-536	52	99	1	7	7	367	404	99
0	6	7	1000	1007	99	0	12	13	332	164	99	1	1	-6	907	830	99	1	5	-14	109	128	99	1	7	8	1991	1927	99
0	6	8	2334	2412	99	0	12	14	132	78	99	1	1	-5	848	847	99	1	5	-13	150	321	99	1	7	9	233	273	99
0	6	9	1371	1438	99	0	14	0	2036	2046	99	1	1	-4	2119	2145	99	1	5	-12	418	412	99	1	7	10	801	764	99
0	6	10	1217	1216	99	0	14	1	846	750	99	1	1	-3	2328	2332	99	1	5	-11	1013	1019	99	1	7	11	-40	93	99
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h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2	h	k	l	Fo	Fc	sigF2
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29	5	-14	201	235	99
29	5	-13	291	33	99
29	5	-12	-273	22	99
29	5	-11	338	284	99
29	5	-10	194	42	99
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30	2	-13	-278	88	99
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25	7	-13	-357	149	99
25	7	-12	417	387	99
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25	7	-10	601	573	99
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25	7	-8	464	544	99
25	7	-7	68	19	99
25	7	-6	169	162	99
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25	7	-3	186	126	99
25	9	-16	-374	203	99
25	9	-15	-174	215	99
25	9	-14	154	79	99
25	9	-13	-487	70	99
25	9	-12	575	542	99
25	9	-11	-201	47	99
25	9	-10	479	511	99
25	9	-9	435	280	99
25	9	-8	338	366	99
25	9	-7	161	270	99
25	9	-6	-204	14	99
25	9	-5	217	96	99
25	11	-14	-250	158	99
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25	11	-9	406	451	99
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26	0	-14	478	393	99
26	0	-12	710	692	99
26	0	-10	603	615	99
26	0	-8	259	269	99
26	0	-6	385	273	99
26	0	-4	595	563	99
26	2	-19	221	363	99
26	2	-18	-333	283	99
26	2	-17	302	301	99
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26	2	-15	193	29	99
26	2	-14	128	303	99
26	2	-13	272	280	99
26	2	-12	655	643	99
26	2	-11	424	313	99
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26	2	-9	180	277	99
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h	k	l	Fo	Fc	sigF2
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26	2	-4	-393	296	99
26	2	-3	343	272	99
26	4	-19	284	350	99
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26	4	-17	291	336	99
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26	4	-14	213	43	99
26	4	-13	532	542	99
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26	4	-11	531	585	99
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26	4	-9	501	461	99
26	4	-8	-304	144	99
26	4	-7	-261	135	99
26	4	-6	228	192	99
26	4	-5	272	243	99
26	4	-4	-117	68	99
26	4	-3	-219	260	99
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26	6	-16	158	78	99
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26	6	-14	317	258	99
26	6	-13	249	275	99
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26	6	-10	243	480	99
26	6	-9	-239	81	99
26	6	-8	442	404	99
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26	6	-5	145	286	99
26	6	-4	-301	344	99
26	8	-16	218	52	99
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26	8	-14	451	301	99
26	8	-13	204	69	99
26	8	-12	546	562	99
26	8	-11	-348	45	99
26	8	-10	458	423	99
26	8	-9	-48	160	99
26	8	-8	257	202	99
26	8	-7	-173	204	99
26	8	-6	370	237	99
26	10	-14	-376	348	99
26	10	-13	156	194	99
26	10	-12	372	395	99
26	10	-11	270	304	99
26	10	-10	288	249	99
26	10	-9	271	322	99
26	10	-8	-342	77	99
27	1	-18	375	234	99

h	k	l	Fo	Fc	sigF2
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27	1	-16	58	267	99
27	1	-15	-207	88	99
27	1	-14	563	550	99
27	1	-13	160	141	99
27	1	-12	546	568	99
27	1	-11	-63	45	99
27	1	-10	209	314	99
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27	1	-8	-164	37	99
27	1	-7	-378	136	99
27	1	-6	496	476	99
27	1	-5	-344	251	99
27	1	-4	371	371	99
27	3	-18	-256	33	99
27	3	-17	161	20	99
27	3	-16	179	197	99
27	3	-15	377	462	99
27	3	-14	173	294	99
27	3	-13	533	589	99
27	3	-12	399	322	99
27	3	-11	259	265	99
27	3	-10	-190	91	99
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27	3	-7	239	211	99
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27	3	-5	296	457	99
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27	5	-15	378	401	99
27	5	-14	-157	335	99
27	5	-13	559	469	99
27	5	-12	526	383	99
27	5	-11	-366	130	99
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27	5	-9	-144	8	99
27	5	-8	76	228	99
27	5	-7	134	352	99
27	5	-6	132	144	99
27	5	-5	350	384	99
27	7	-16	211	175	99
27	7	-15	290	176	99
27	7	-14	444	477	99
27	7	-13	229	72	99
27	7	-12	285	448	99
27	7	-11	-377	57	99
27	7	-10	104	263	99
27	7	-9	-167	197	99
27	7	-8	317	56	99
27	7	-7	-137	246	99
27	9	-14	489	435	99
27	9	-13	-293	184	99
27	9	-12	222	344	99
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h	k	l	Fo	Fc	sigF2
27	9	-10	121	147	99
27	9	-9	258	186	99
28	0	-18	91	37	99
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28	0	-12	275	289	99
28	0	-10	-335	53	99
28	0	-8	265	169	99
28	0	-6	-214	432	99
28	2	-17	178	94	99
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28	2	-15	392	397	99
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28	2	-13	282	297	99
28	2	-12	182	211	99
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28	2	-6	274	323	99
28	4	-17	302	254	99
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