DEPARTMENT OF AGRICULTURE, SOUTH AUSTRALIA

Agronomy Branch Report

THE USE OF DIURON FOR THE CONTROL OF ANNUAL WEEDS IN CEREAL CROPS (WHEAT AND BARLEY)

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Report No. 39

SUMMARY

Since 1966, Messrs. G.B. Baldwin, formerly Senior Research Officer (Cereal Herbicides) and M.J. Catt, Research Officer (Weed Control), have tested the herbicide, diuron, for the control of various weeds in cereal crops.

The reported trials indicate that diuron can be safely used to provide commercially acceptable weed control in wheat and barley crops resulting in yield increases. A spectrum of cereal weeds controlled by specified rates of diuron has been determined.

Zero residue levels have been found in grain of wheat crops treated with diuron.

The work reported here was used in part to secure a clearance from the Technical Committee on Agricultural Chemicals for the use of diuron in barley and wheat crops.

THE USE OF DIURON FOR THE CONTROL OF ANNUAL WEEDS IN CEREAL CROPS (WHEAT AND BARLEY)

1. INTRODUCTION:

Diuron[1] has been used for weed control in cereal crops in the United States for over ten years. In Australia, however, its development was curtailed by Dupont (Australia) Ltd., in favour of a similar herbicide, linuron[2]. Diuron has been used extensively in Australia for selective weed control in orchards, vineyards, lucerne and grass seed crops and also for total vegetation control. Screening trials in South Australia have indicated that this herbicide may have a place in cereal weed control, particularly since the patent on its production expired in 1970 and several companies are now marketing the material. All the work reported here involved the use of an 80% wettable powder formulation of diuron supplied by Dupont (Australia) Ltd. This is the commonly available form of the herbicide.

2. TRIALS:

2.1 Trial 1, 1968: Post-emergent annual ryegrass, *Lolium rigidum*, control in wheat

2.1.1 Details - Minilog trial in Insignia 49 wheat.

Sprayed 1 month post-sowing (1-2 leaf stage) of the crop.

Visual assessments indicated:-

* Complete control of annual ryegrass by diuron at rates in excess of 1.44 lbs. a.i. per acre (1.61 kg/ha).
* Heavy suppression (estimated 80% reduction in dry weight) at rates in excess of 0.81 lbs. a.i. per acre (0.91 kg/ha).
* Although transient yellowing of the crop took place, no apparent damage remained 4-5 weeks after treatment.

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[1] Diuron 3-(3,4 dichlorophenyl)-1, 1-dimethylurea

[2] Linuron 3-(3,4 dichlorophenyl)-1-methoxy-1-methyl urea

Three other sites containing minor quantities of annual ryegrass were examined and it was evident diuron was giving complete control of seedling ryegrass at a rate of 1 lb. a.i. per acre (1.12 kg/ha). Once again, only transient yellowing of the crop took place at this level.

2.2 Trial 2, 1969: Post-emergent annual ryegrass, Lolium rigidum, control in wheat

2.2.1 Details

As a result of Trial 1 above, field trails in 1969 examined the effect on yield and the yield components (heads/acre, grains/head and weight/grain) of post-emergent applications of diuron in annual ryegrass infested wheat.

At four of the five sites there was a marked increase in the number of heads of wheat per acre after post-emergent treatment of ryegrass with rates of diuron between 0.8 - 1.2 lbs. active per acre (0.9 - 1.35 kg/ha). This was significant at three of them. An increase in grain yield for these treatments was subsequently recorded at four sites. This was significant at two of them.

Tables 1 and 2 summarise these results.

Table 1: Mean Number of Heads per Sample (sample size varies between sites)

Treatment	Site				
	Bute 1	Blyth	Maitland	Gladstone	Bute 2
Untreated	34.3	22.3	184.4	64.7	60.8
Diuron 0.6 lbs. active/ acre (0.67 kg/ha)	40.7	21.6	191.5	74.5	62.4
Diuron 0.8 lbs. active/ acre (0.9 kg/ha)	*** 48.7	** 30.9	* 211.0	81.5	56.0
Diuron 1.0 lbs. active/ acre (1.1 kg/ha)	-	-	*** 238.4	70.3	-
Diuron 1.2 lbs. active/ acre (1.35 kg/ha)	** 43.1	** 31.3	** 226.0	71.7	61.4
Diuron 2.0 lbs. active/ acre (2.2 kg/ha)	-	-	221.7	79.1	-
Diuron 2.4 lbs. active/ acre (2.7 kg/ha)	-	-	193.1	77.6	-

* Treatment significant at the 5% level
** Treatment significant at the 1% level
*** Treatment significant at the 0.1% level

Table 2: Mean Yields in Bushels per Acre (kg/ha)

Treatment	Site				
	Bute 1	Blyth	Maitland	Gladstone	Bute 2
Untreated	16.5 (1098)	8.3 (554)	35.9 (2393)	13.5 (902)	22.2 (1481)
Diuron 0.6 lbs. active/ **acre** (0.67 kg/ha)	19.9 (1325*)	7.6 (508)	37.5 (2495)	18.5 (1230)	22.1 (1469)
Diuron 0.8 lbs. active/ **acre** (0.9 kg/ha)	20.3 (1354*)	9.1 (636)	37.8 (2521)	18.2 (1212)	21.2 (1411)
Diuron 1.0 lbs. active/ **acre** (1.1 kg/ha)	-	-	47.4 (3165***)	16.5 (1103)	-
Diuron 1.2 lbs. active/ **acre** (1.35 kg/ha)	18.9 (1258)	9.5 (712)	45.2 (3011***)	17.8 (1183)	15.3 (1017)
Diuron 1.6 lbs. active/ **acre** (1.8 kg/ha)	20.9 (1391**)	8.7 (581)	-	-	21.8 (1452)
Diuron 2.0 lbs. active/ **acre** (2.2 kg/ha)	-	-	42.8 (2857**)	20.8 (1383)	-
Diuron 2.4 lbs. active/ **acre** (2.7 kg/ha)	-	-	38.3 (2555)	20.5 (1366)	-
L.S.D's. between treatment means					
α = 0.05	2.8 (188)	N.S.	4.5 (299)	N.S.	N.S.
α = 0.01	3.8 (251)		6.1 (403)		
α = 0.001	4.9 (327)		8.1 (538)		

At the two sites where grain number per head and 100 **corn weight** were recorded, no significant difference between **treatments was** evident. There **was** a tendency at one site, **however,** for an increase to occur in both these yield components. On this site treatment took place fifteen days after **sowing.**

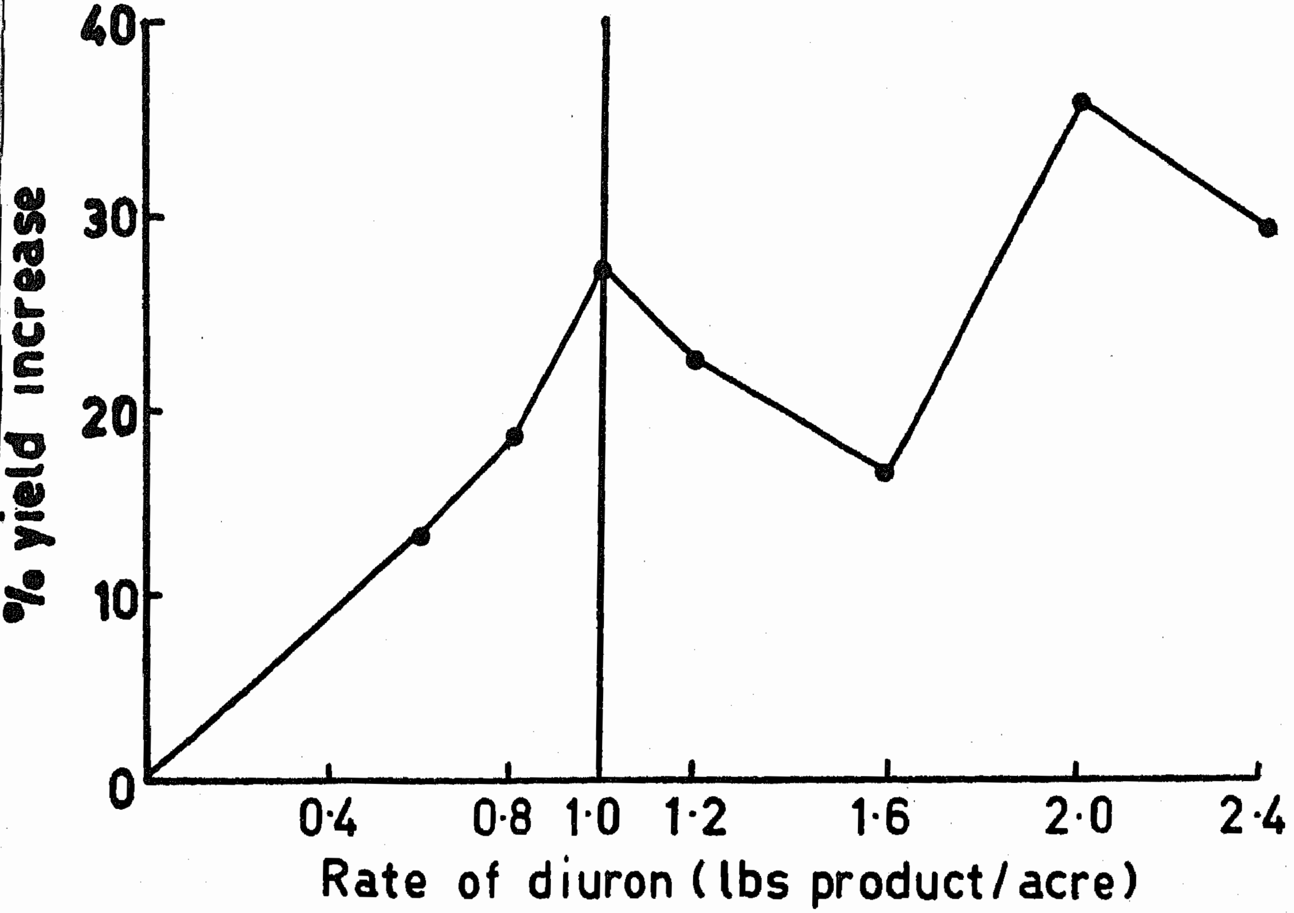
1969 results indicate that the early control of **annual** Wimmera ryegrass with diuron some 2-5 weeks after sowing **allows** the potential yield of **wheat** to be increased by increasing the number of heads per sample. Other yield components **are** little affected.

The site (coded Bute 2) where there was no increase in head number per sample, and no corresponding yield increase was sprayed some 7½ weeks after sowing. The crop had developed 1-2 tillers per plant at the time of treatment. This suggests that the potential yield of this crop had largely been determined at the time of treatment. There is a delay of some three weeks after treatment (depending on soil type and rainfall) before ryegrass control becomes evident and the removal of competition at this site, probably came too late to affect head number per acre. This site contained the lowest density of ryegrass (approximately 50 plants per square foot (535/m^2)).

At the remaining sites where the oven dry weight of the grass was measured some 6-8 weeks after treatment, all treatments gave a significant reduction. A fairly uniform pattern of reduction was noted regardless of initial density and subsequent growth. 0.6 lbs. active per acre (0.67 kg/ha) reduced oven dry weight by 40% when compared to untreated, while 0.8 lbs. active per acre (0.9 kg/ha) reduced it by 50-60% and 1.2 lbs. active per acre (1.35 kg/ha) by 70% of untreated. A rate of 1.6 lbs. active per acre (1.8 kg/ha) reduced the level of dry weight to below 25% of unsprayed.

A graph of yield (expressed as a percentage of untreated yield) against rate of diuron, showed that on these soils and with a ryegrass density in excess of 100 plants per square foot (1070/m^2) a 20-25% yield increase could be expected at a treatment level of 1.0 lbs. active per acre (1.12 kg/ha).

Mean % yield increase with control of annual rye-grass with diuron.



2.3 <u>Trial 3, 1968</u>: Early Post-emergent control of sheepweed, *Buglossoides arvense*, in cereals

2.3.1 <u>Details</u> - Minilog trial - Kulpara (Prior barley) and Kadina (Raven wheat)

The absolute minimum dosage for diuron could not be determined as it gave complete control for the entire length of the logarithmic plots at Kulpara. Damage occurred to the barley at rates of diuron greater than 30.0 ozs. product per acre (2.1 kg/ha).

In the trial at Kadina, 52 points of rain fell five to seven hours after herbicide application. Diuron activity was markedly reduced by the rain as shown in the following table. Crop damage was less for the wheat than that recorded on the barley.

Table 3: Effect of Rain on Post-emergent Control of Sheepweed by Diuron

Diuron 80% W.P.	No Rain	52 Points Rain	
P.D.	Complete Control	Complete Control Sheepweed Above	Loss in Efficiency
51.2	3.0*	12.3	310%

P.D. = Peak dosage in ozs. a.i./acre

* This value is the minimum value tested

Soil type, sheepweed size and growing conditions were the same at both sites.

2.4 <u>Trial 4, 1969</u>: Control of soursob, *Oxalis pes-caprae*, in cereals

2.4.1 <u>Details</u>

Minilog trials on heavy red brown earths and sandy loam soils for soursob control in wheat and barley at early tillering. Commercially acceptable control of soursob was achieved in barley on sandy soil at rates down to 1.2 lbs. a.i. per acre (1.34 kg/ha) whilst crop damage occurred at rates down to 2 lbs. a.i. per acre (2.24 kg/ha). On heavy soil soursob was suppressed at rates of diuron down to 1.5 lbs. a.i. per acre (1.68 kg/ha) whilst the wheat crop was affected at rates down to 2 lbs. a.i. per acre (2.24 kg/ha).

2.5 Trial 5, 1970: Control of Soursob, *Oxalis pes-caprae*, in wheat

2.5.1 Details

Two trials using fixed dosage rates selected from the previous minilog trials were carried out in 1970. Two soil types were used with Heron wheat.

Results of this trial have been summarised in Table 4.

Table 4: The Effect of Diuron on Yield of Wheat and Soursob Density

Soil Type	Herbicide	Rate ozs. Product/acre (kg/ha)	Yield Bushels/acre (kg/ha)	Soursob Plants (% of control)
Sandy Mallee	Diuron Diuron Diuron Control	13.5 (.95) 18 (1.26) 27 (1.89) -	21.0 (1400) 21.6 (1440) 16.8 (1120) 18.4 (1225)	50.6* 57.6* 35.3* 100
	L.S.D's. α=0.05		No significant differences	44.8
Red Brown Earth	Diuron Diuron Diuron Control	27 (1.89) 36 (2.52) 54 (3.78) -	33.9 (2260)** 34.0 (2265)** 33.1 (2205)* 26.5 (1765)	40 ** 18.4** 11.8** 100
	L.S.D's. α=0.05 α=0.01		5.0 (330) 6.75 (450)	30.4 41.1

Observations of the trial areas in 1971 confirmed that the weed control obtained has a permanent effect on bulb production.

As a result of this trial further testing was carried out in 1971.

2.6 Trial 6, 1971: Soursob, *Oxalis pes-caprae*, control in cereals

2.6.1 Details

Five trials on wheat and one on barley.

Table 5: The Effect of Diuron on the Yields of Wheat and Barley Following Soursob Control with Diuron

Crop	Wheat					Barley
Location	Alford	Boors Plains A	Boors Plains B	Two Wells	Korunye	White River
Variety	Halberd	Halberd	Halberd	Halberd	Gamenya	Clipper
Treatment						
Diuron 1 lb. product/acre (1.12 kg/ha)	31.9* (2130)	42.5 (2830)	47.7 (3240)	51.6* (3440)	13.6 (910)	30.8 (2050)
Diuron 1½ lbs. product/acre (1.68 kg/ha)	32.2* (2150)	48.0* (3200)	51.6* (3440)	53.0* (3530)	14.1 (940)	30.5 (2040)
Control	23.8 (1590)	41.7 (2780)	43.7 (2910)	43.1 (2870)	15.3 (1020)	31.3 (2090)
L.S.D. $\alpha = 0.05$	6.7 (445)	5.4 (370)	4.6 (310)	5.4 (360)	F. N.S.	F. N.S.

* Diuron applied at 3-5 leaf stage of the crop.

* Soursob dominant weed in all trials.

* Boors Plains A trial received 30 points of rain within 3 hours of application of diuron. Trial repeated alongside as Boors Plains B, 5 days later. It is suggested that diuron may be washed off leaves of the weed thus reducing its effectiveness, at least in foliar uptake. This could be reflected as the yield increase in Boors Plains A when compared to Boors Plains B at the 1 lb. product per acre (1.12 kg/ha).

* Korunye trial was badly infected with haydie and was patchy. F not significant, hence no L.S.D. figure was given.

* White River trial similarly was patchy and considerable grain had been lost due to wind before harvesting could be carried out. F not significant, hence no L.S.D. figure is given.

* All trials were laid out as randomised blocks with four replicates.

Weed Control Rating:-

1 = Complete control

9 = No control

No treatment 9

Diuron 1 lb./acre 2

Diuron 1½ lbs./acre 1 on sandy soils. Two trials were on heavier soil types.

These results were general through the trials.

Counts taken in June, 1972 at three of the sites and another trial at Roseworthy College from which yield figures were not obtained in 1971, are shown in Table 6

Table 6: Number of Soursob Plants per Square Link (Counts taken June, 1972)

Treatment	Roseworthy College	Alford	Two Wells	White River
Diuron 1 lb. product/acre	3.8	9.3	7.75	25.2
Diuron 1½ lbs. product/acre	0.45	9.95	3.8	13.7
Control	8.65	25.2	27.90	35.4
L.S.D. α = 0.05	5.40	5.95	5.46	8.33

2.7 Trial 7, 1969: Early post-emergent control of long-fruited wild turnip, *Brassica tournefortii*, in cereals

2.7.1 Details

A mini-log trial was conducted in Heron wheat on a sandy soil infested with long-fruited turnip.

A peak dosage of 1 lb. product diuron per acre (2.24 kg/ha) was used at two times of spraying. The early application was carried out when the turnip was 2" in diameter (5 cms.), 56 days post-sowing of the crop. The late spraying occurred when the turnip was 6-8" in diameter (15-20 cms.), 84 days post-sowing.

Complete control of the turnip was achieved at rates of 1 oz. product per acre (.07 kg/ha) and above at the early spraying and 2.5 ozs. product per acre (.18 kg/ha) and above at the later time.

2.8 Trial 8, 1969: Early post-emergent control of dead-nettle, *Lamium amplexicaule*, in wheat

2.8.1 Details

A mini-log trial was conducted in wheat infested with deadnettle.

A peak dosage of 2 lbs. product diuron per acre (2.24 kg/ha) was used. Complete control of seedlinn deadnettle occurred at rates of less than 2 ozs. product per acre (.14 kg/ha). Some visible crop suppression occurred at rates in excess of 12 ozs. product per acre (.84 kg/ha).

2.9 Trial 9, 1969: Early post-emergent control of wire-weed, *Polygonum aviculare*, in wheat at Gawler

2.9.1 Details

A mini-log trial was conducted in Dirk wheat on a sandy loam soil. The crop was at the 4-leaf stage at application, 50 days post-sowing. A peak dosage of 24 ozs. product per acre (1.68 kg/ha) was used. The wireweed was in the seedling stage. No visual damage to the crop occurred. Complete control of the weed was not achieved at the peak dosage. Commercially acceptable control was achieved at 18 ozs. product per acre (1.26 kg/ha). It is not considered economical to use diuron for wireweed control.

2.10 Trial 10, 1970: Clean crop tolerance trial conducted at Two Wells

2.10.1 Details

Heron wheat on a sandy loam soil was treated at the 4 leaf stage with 12, 24 and 48 ozs. product diuron per acre (.84, 1.68, 3.36 kg/ha) respectively. The site supposedly clean of weeds had light soursob, mustard and emex infestations. The latter two were removed at all rates. Soursob was removed at the two higher rates.

Yield figures - bushels/acre (kg/ha)

No treatment	18.8 (1250)
Diuron 12 ozs. product/acre (.84 kg/ha)	19.9 (1330)
Diuron 24 ozs. product/acre (1.68 kg/ha)	24.5 (1630)**
Diuron 48 ozs. product/acre (3.36 kg/ha)	19.0 (1270)
L.S.D. $\alpha = 0.05$	4.24 (270)
$\alpha = 0.01$	5.63 (375)

These results suggest wheat is tolerant to rates of diuron up to at least 24 ozs. product per acre (1.68 kg/ha).

2.11 Trial 11: Spiny emex, *Emex australis*, control

2.11.1 Details

Although this trial was carried out in an annual medic pasture, it is considered that the rate of diuron required for spiny emex control can be transposed to the cereal crop situation. The weed was at the 5-6 true leaf stage and 5-6" in diameter (12.5-15 cms.), when sprayed. Complete kill was obtained with 10.7 ozs. production diuron per acre (.75 kg/ha) and heavy suppression occurred at rates of 6.7 ozs. product per acre (.47 kg/ha) and above.

3. SUMMARY:

The results of the reported trials and farmer experience suggest that diuron can be used with safety in cereal crops (barley and wheat) to control the following weeds at the rates shown:-

		lb. 80% product/acre	kg/ha
Deadnettle	*Lamium amplexicaule*	3/8	(.42)
Spiny emex or three-corner jack	*Emex australis*	1/2	(.56)
King Island melilot	*Melilotus indica*	1/2	(.56)
Mustards	*Sisymbrium* spp.	1/2	(.56)
Poppy (rough and horned)	*Papaver hybridum* and *Glaucium flavum*	1/2	(.56)

		lb. 80% product/acre	kg/ha
Annual ryegrass (seedling)	Lolium rigidum	1 light soils 1¼ heavy soils	(1.12) (1.40)
Sheepweed	Buglossoides arvense	⅜	(.42)
Soursob	Oxalis pes-caprae	1 light soils 1½ heavy soils	(1.12) (1.68)
Saffron thistle	Carthamus lanatus	½	(.56)
Wild turnip (long fruited)	Brassica tournefortii	¼	(.28)
Yellow burr-weed	Amsinckia hispida	½	(.56)

The herbicide will not control wireweed, vetch, fumitory or perennial crop weeds.

The following remarks on use should be observed. Do not spray if rain is threatening. Use 50 mesh filters and have good tank agitation.

No information has been gathered from local sources on the use of diuron in oat crops. However, work conducted in Oregon indicates that oats are as tolerant to diuron as wheat. Barley is less tolerant than wheat or oats.

3.1 Analysis of Grain for Diuron Residues

The South Australian Department of Chemistry analysed fifteen samples of wheat and barley from Trial 6 in this report. The method involved extraction with acetone, cleaning up with hexane, partitioning with methylene chloride and carbon tetrachloride and analysis using a gas chromatograph giving a limit of detection of 0.3 ppm.

Samples of grain from control plots and plots treated with 1 and 1½ lbs. diuron product per acre (1.12 and 1.68 kg/ha) from each of the six sites were analysed. The two Boors Plains trials were pooled in this case.

No detectable diuron residues were found in any samples. This result satisfies the U.S.A. maximum residue level of 2 ppm. set for diuron residues in cereal grains.

The method of analysis used is included in Appendix I.

APPENDIX I

The Determination of Diuron in Wheat

The following method is taken from a paper to be submitted for publication in one of the journals on analytical chemistry.

1. Reagents

Acetone, B.P., Redistilled; use 250 mm fractionating column.

Petroleum Ether. BDH Analar. B.P. 40-60°C Redistilled (as for acetone).

Dichloromethane. BDH Analar. Redistilled (as for acetone).

Carbon Tetrachloride. BDH Analar. Redistilled (as for acetone)

Sodium sulphate. BDH Analar 2% m/v aqueous solution.

Sodium sulphate. BDH Analar anhydrous. Heat in an air oven or muffle furnace at 400°C for 16 hours. Store in desiccator.

Diuron "Karmex" - 80% m/m diuron.

Diuron standard solution - 10 μg/ml in acetone.

2. Apparatus

Small hammer mill "Glen Creston" (or similar, 1 mm screen

Soxhlet extractor

Rotary evaporator, "Buchi" (or similar)

Water bath, "Electrothermal" (or similar)

Separating funnels, 250 ml and 500 ml.

Tapered graduated tubes, 10 ml capacity

Drying column. Filter tube 200 mm x 20 mm i.d. with coarse fritted filter

Gas Chromatograph Column. 1.5% S.E. 30/Q.F.1 (1+1) on Anakrom ABS in glass column 180 cm. long x 4 mm i.d. (Other suitable column packings are 10% D.C. 200, 3% O.V.17 or 11% O.V.17/Q.F.1 (1.3+1) on Anakrom ABS or similar support).

Gas Chromatograph. "Packard" Model 7731 (or similar) with tritium electron-capture detector. Operating conditions:- Injection 225°C, Column 150°C, Outlet 240°C, Detectors 200°C. Carrier gas, N at 55 ml/min. Sensitivity 1X 10^{-9}A. Detector voltage 25V.

Recorder. "Rikendenshi" (or similar) Range 10 mv. Chart speed 10 mm/min.

U.V. Spectrophotometer. "Shimadzu" UV-200 (or similar). 10 mm quartz cells. Wave-length 247 mm. Reference, distilled water. (6 μg/ml diuron in aqueous solution produced ½ F.S.D.)

3. Sample Preparation & Extraction:

Grind the sample in the hammer mill. Weigh 10g into a Soxhlet thimble and extract with acetone for at least 4 hours (4-5 ml/minute). Reduce the volume of acetone to less than 25 ml in the rotary evaporator under vacuum and $\lessdot$45°C.

4. Clean-up:

Transfer the acetone extract to a 250 ml separating funnel with a few ml of acetone. Add 25 ml petroleum ether and 125 ml of 2% Na_2SO4 solution. Mix gently and thoroughly. Transfer the aqueous layer to a second separating funnel and extract as before with 25 ml. petroleum ether. Repeat once more and combine the petroleum ether extracts. Wash twice with 50 ml 2% Na_2SO4 solution. Combine the aqueous phases in a 500 ml separating funnel and discard the petroleum ether extracts.

Extract the combined aqueous solution twice with 50 ml dichloromethane and once with 50 ml carbon tetrachloride shaking vigorously each time for 2 minutes. Combine the $CHC1_2CC1_4$ phases and dry by passing through a 50 mm colum of anhydrous Na_2SO_4. Remove the solvents in the rotary evaporator under vacuum and $\lessdot$45°C until dry. Immediately transfer with acetone into a 10 ml tapered graduated tube. Evaporate to dryness under nitrogen and immediately re-dissolve in acetone. Adjust volume to 5 ml. Inject 1 1 into the gas chromatograph and using peak height calculate diuron content from a graph prepared by injecting suitable volumes of standard diuron solution.

Spike a diuron-free sample of wheat for recovery test and run a blank determination on all solvents, etc.

5. <u>U.V. Spectrophotometry</u> (alternative method):

Evaporate to dryness under nitrogen and immediately dissolve in 5 ml distilled water. Place solution in the cell of the spectrophotometer and measure the absorbance. Calculate the diuron content from a graph prepared from diuron standards. Repeat with the spiked and blank solutions.