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GLASSHOUSE INVESTIGATIONS WITH NEWER SOIL-APPLIED HERBICIDES FOR WEED CONTROL ON ORGANIC SOILS

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Summary Ten herbicides with pre-emergence weed control activity on mineral soil were tested on *Stellaria media* L., *Polygonum lapathifolium* L., *Poa annua* L. and turnip (four herbicides were also tested on barley) for pre-emergence weed control activity on an organic fine sandy loam and a peat soil. The experiments were carried out in pots placed in a temperate glasshouse. Treatments were both incorporated and also surface applied. The most promising herbicides were hexazinone, ethalfluralin and oxyfluorfen for general broad-leaved weed control and control of *P. annua*; incorporated treatments of trifop-methyl and NP48 (2-(1-allyloxyaminobutylidene)-5,5-dimethyl-4-methoxycarbonyl-cyclohexane-1,3-dione as the sodium salt) for the control of volunteer cereals in broad-leaved crops; K1441 (1-(α,α-dimethylbenzyl)-3-methyl-3-phenylurea) for the control of *P. annua*, and butam for the control of grass and certain broad-leaved weeds. Fluothiuron, triclopyr ester and 3,6-dichloropicolinic acid gave poor control of most species.

Résumé: Dix herbicides dont l'activité en pré-levée dans terres minérales est connue ont été expérimentés sur *Stellaria media* L., *Polygonum lapathifolium* L., *Poa annua* L. et navet (4 herbicides ont été essayés sur orge aussi) pour déterminer leur activité désherbante en pré-levée dans sols franco-sablonneux humifères et dans sols tourbeux. Les essais ont eu lieu sur des plantes cultivées en pots en serre tempéré, avec ou sans incorporation des produits. Les herbicides offrant les meilleures possibilités dans la lutte générale contre les dicotylédones et le *Poa annua* étaient le hexazinone, l'éthalfluraline et l'oxyfluorfène; contre les repousses de céréales en culture dicotylédonaire, le trifop-methyl et le NP48 (2-(1-allyloxyaminobutylidene)-5,5-dimethyl-4-methoxycarbonyl-cyclohexane-1,3-dione - sel de sodium); contre le *P. annua*, le K1441 (1-(α,α-dimethylbenzyl)-3-methyl-3-phenylurea) et contre les graminées et certains dicotylédones, le butam. Le fluothiuron, le triclopyr ester et l'acide 3,6-dichloropicolinique n'ont pas donné une destruction satisfaisante de la plupart des espèces adventices.

INTRODUCTION

Weeds are a major problem on organic soils such as are found in the fens of East Anglia. On these soils the activity of most soil-applied herbicides is reduced and there is always a need for more active herbicides.

The three glasshouse experiments described here investigate the weed control activity of some newer herbicides on organic soils. All of these, oxyfluorfen, K1441 (1-(α,α-dimethylbenzyl)-3-methyl-3-phenylurea), triclopyr ester, 3,6-dichloropicolinic acid, hexazinone, butam, fluothiuron, trifop-methyl, NP48 (2-(1-allyloxyaminobutylidene-5,5-dimethyl-4-methoxycarbonyl-cyclohexane-1,3-dione as the sodium salt) and ethalfluralin had already shown good pre-emergence weed control activity in pot experiments on mineral soils at WRO (Richardson et al., 1976; Richardson and Parker, 1976, 1977, 1978; Richardson, personal communication).

METHOD AND MATERIALS

The herbicides, their formulations, doses and time of application are given in Table 1 and the relevant dates for each of the three experiments in Table 2. All doses quoted are in kg a.i./ha. Stellaria media L., Polygonum lapathifolium L., Poa annua L. and turnip were used in all three experiments with barley in experiment three only. The two crop species were included as easily grown examples of dicotyledonous and a monocotyledonous species. The three weeds are common in most organic soils. Herbicide treatments were applied as incorporated or surface applications to two soils, one an organic fine sandy loam (19% organic matter content, pH 5.6) and the other a peat (50% organic matter content, pH 5.4).

Table 1

Herbicide	Formulation	Doses kg a.i./ha	Experiment number
hexazinone	(90% w/w w.s.p.)	0.2, 0.6, 1.8	1
ethalfluralin	(33% w/v e.c.)	0.4, 1.2, 3.6	1
triclopyr ester	(48% w/v e.c.)	0.4, 1.2, 3.6	2
oxyfluorfen	(24% w/v e.c.)	0.8, 2.4, 7.2	2
fluothiuron	(25% w/v e.c.)	0.8, 2.4, 7.2	2
K 1441	(50% w/w w.p.)	0.8, 2.4, 7.2	3
3,6-dichloropicolinic acid	(10% w/w w.p)	0.1, 0.3, 0.9	3
trifop-methyl	(16% w/v e.c.)	0.1, 0.3, 0.9	3
butam	(72% w/v e.c.)	0.8, 2.4, 7.2	3
NP 48	(75% w/w w.p.)	0.25, 0.75, 2.25	3

Table 2

Dates of planting and harvesting

	Experiment number		
	1	2	3
Pots sown and treatments applied	7 Dec 76	19 Apr 77	27 Jul 77
Fresh weights taken:			
Stellaria media	12 Jan 77	21 June 77	6 Sept 77
Polygonum lapathifolium	21 Jan 77	18 July 77	9 Sept 77
Poa annua	21 Jan 77	18 July 77	6 Sept 77
turnip	6 Jan 77	10 June 77	23 Aug 77
barley	-	-	22 Aug 77

Ten P. annua, eight P. lapathifolium, eight turnip (var. Green Globe), fifteen S. media or six barley (var. Maris Mink) seeds were sown per 6.4 cm pot. In each experiment all pots were placed in randomised blocks in a temperate glasshouse. Four replications of each treatment were used. The incorporated treatments were sprayed in open top metal trays 19 cm by 14 cm by 6.4 cm deep and were thoroughly mixed by inversion of the soil three times through a large plastic funnel before the soil was placed in pots and the species planted. Treatments were applied by the WRO pot sprayer fitted with an 8002E 'Spraying Systems Teejet' delivering 366 l/ha at 2 bars pressure.

Naturally occurring weeds were removed by hand at regular intervals during the experiment. Growth was assessed when untreated plants were approximately 6 to 8 cm high. The foliage was cut at ground level and fresh weights taken. Plant numbers were also recorded.

RESULTS

Table 3 lists those herbicides that caused a significant (at $P = 0.05$) depression in the fresh weights of the five species. The full results of species on which statistical differences were observed for hexazinone and ethalfluralin, oxyfluorfen and K1441 and butam are given in tables 4, 5 and 6 respectively.

The fresh weights of foliage per pot were transformed to $\log_{10}(10x + 1)$ for statistical reasons. The detransformed data is not included because only comparisons between treatments and the control were required. Herbicide effects on plant numbers were similar to those for plant fresh weights and are omitted.

Hexazinone. This herbicide significantly reduced the weights of all four species. At the lower doses it was more active on the organic fine sandy loam than the peat and was more effective incorporated than surface applied.

Ethalfluralin. _P. annua_ was controlled by all doses of this herbicide. Turnip was least affected with only the highest dose (3.6 kg/ha) significantly reducing fresh weights. There were few differences between the two soils or the two methods of application.

Triclopyr. High doses of triclopyr controlled turnip and, when incorporated, _S. media_ growing on the high organic matter soil. _P. lapathifolium_ and _P. annua_ were not affected.

Oxyfluorfen. _S. media_ was not controlled by this herbicide and so this species was not included in table 5. The two highest doses significantly reduced the weights of the other species, especially in the lower organic matter soil. It tended to be most active when surface applied. The highest dose, surface applied to _P. lapathifolium_ growing in the organic sandy loam, completely controlled the weed.

Fluothiuron. This herbicide did not give good weed control. It only significantly affected _P. annua_ as a surface application of 7.2 kg/ha on the high organic matter soil.

_K1441_. The 0.8 and 2.4 kg/ha doses had little effect on any of the species apart from _P. annua_. _P. lapathifolium_ was not controlled by any dose (and is omitted from table 6), but _S. media_, turnip and barley were affected by 7.2 kg/ha. There were some indications that it was more active on the lower organic matter soil but it behaved similarly when applied as a surface or incorporated treatment.

3,6-dichloropicolinic acid. The incorporated treatment on the organic fine sandy loam had some effect on _S. media_ but otherwise this herbicide had little weed control activity.

Trifop-methyl. Incorporated treatments on both soils reduced the fresh weight of barley but it had no effect on any of the other test species.

Table 3

Herbicides having a significant depression (at P = 0.05) on the fresh weights of the five species

Herbicides	S. media				P. lapathifolium				P. annua				Turnip				Barley			
	+O.L.		P.		O.L.		P.		O.L.		P.		O.L.		P.		O.L.		P.	
	S	I	S	I	S	I	S	I	S	I	S	I	S	I	S	I	S	I	S	I
hexazinone	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	NOT TESTED			
ethalfluralin	*	*	*	*		*	*	*	*	*	*	*	*	*		*	NOT TESTED			
triclopyr ester				*									*		*	*	NOT TESTED			
oxyfluorfen						*	*		*		*	*	*				NOT TESTED			
fluothiuron											*									
K1441		*								*	*	*	*	*				*		*
3,6-dichloropicolinic acid		*																		
trifop-methyl																		*		*
butam		*	*	*						*	*	*					*	*		*
NP48																		*	*	*

* significant control at P> 0.05 by at least one dose level of the herbicide

+ O.L. = organic fine sandy loam; P. = peat;

S, surface-applied; I, incorporated.

Table 4

Fresh weights of *S. media*, *P. lapathifolium*, *P. annua* and turnip expressed as $\log_{10}$ (10x+1) fresh weight/pot (g) for hexazinone and ethalfluralin

Herbicide	Application method	Dose (kg a.i./ha)	*S. media*		*P. lapathifolium*		*P. annua*		Turnip	
			+O.L.	P.	O.L.	P.	O.L.	P.	O.L.	P.
hexazinone	surface	0.2	0.004	0.654	0.009	0.137	0.042	0.194	0.025	0.677
"	"	0.6	0.004	0.316	0.044	0.029	0.006	0.006	0.013	0.025
"	"	1.8	0.004	0.004	0.009	0.009	0.004	0.006	0.009	0.013
"	incorporated	0.2	0.004	0.004	0.013	0.009	0.006	0.065	0.106	0.063
"	"	0.6	0.004	0.002	0.009	0.025	0.006	0.006	0.021	0.009
"	"	1.8	0.004	0.004	0.009	0.064	0.006	0.004	0.044	0.045
ethalfluralin	surface	0.4	0.250	0.506	0.660	0.473	0	0	1.185	1.543
"	"	1.2	0.085	0.271	0.249	0.337	0	0	1.172	1.399
"	"	3.6	0.040	0.112	0.341	0.129	0	0.002	0.883	1.167
"	incorporated	0.4	0.341	0.637	0.579	0.400	0	0	1.168	1.445
"	"	1.2	0.036	0.152	0.385	0.164	0	0	1.047	1.441
"	"	3.6	0	0.066	0.093	0.148	0	0	0.771	0.841
control			0.605	0.702	0.548	0.463	0.454	0.350	1.393	1.501
S.E. of treated means			± 0.094		± 0.077		± 0.064		± 0.124	
S.E. of control means			± 0.067		± 0.056		± 0.045		± 0.088	

+O.L. = organic fine sandy loam; P. = peat.

Table 5

Fresh weights of P. lapathifolium, P. annua and turnip expressed as $\log_{10}(10x+1)$ for oxyfluorfen

Application method	Dose (kg a.i./ha)	P. lapathifolium		P. annua		Turnip	
		+O.L.	P.	O.L.	P.	O.L.	P.
surface	0.8	0.322	1.179	0.224	0.253	0.805	1.358
"	2.4	0	0.861	0	0	0.995	1.387
"	7.2	0	0	0	0.016	0	1.466
incorporated	0.8	1.330	1.100	0.750	0.731	1.028	1.298
"	2.4	0.019	0.813	0.406	0.627	0.981	1.604
"	7.2	0	0.693	0	0	0.442	1.548
control		0.834	1.085	0.770	1.030	1.148	1.522
S.E. of treated means		± 0.231		± 0.173		± 0.141	
S.E. of control means		± 0.163		± 0.122		± 0.100	

+O.L. = organic fine sandy loam; P. = peat

Butam. Turnip and P. lapathifolium were not controlled by butam. It had some activity on S. media and barley whilst P. annua was eliminated by the highest dose (7.2 kg/ha). It appeared to be more active incorporated than surface applied and these incorporated treatments when applied to the lower organic matter soil, particularly those on barley, were more active than on the peat.

NP48. This herbicide behaved similarly to trifop-methyl, but the surface application at 2.25 kg/ha on the high organic matter soil also affected the barley.

DISCUSSION

The results of these experiments, like all obtained in the glasshouse, cannot be directly related to field conditions. However they do give indications of pre-emergence weed control activity on organic soils.

Although hexazinone was generally more active incorporated than surface applied, the difference may not be large enough to justify incorporation. Successful incorporation under field conditions usually involves an extra cultivation and if rotary cultivation is necessary it usually leaves a fine seedbed that is susceptible to wind erosion, even after rolling. This herbicide is being developed by the manufacturers as a non-selective herbicide but Richardson and Parker (1977) suggested that on mineral soils some selective control of P. lapathifolium and S. media plus some other weeds might be possible in such crops as carrot, pea, wheat and dwarf bean. Such selectivity could be useful on organic soils.

Ethalfluralin has a similar weed spectrum to trifluralin (Richardson, personal communication) but this experiment has shown it to have good activity on organic soil. Trifluralin at normal rates has poor activity on organic soils. If the rates of ethalfluralin necessary for weed control on organic soil are not prohibitively expensive this compound could prove very useful.

Both trifop-methyl and NP48 have been suggested as herbicides for grass weed

Table 6

Fresh weights of *S. media*, *P. annua*, turnip and barley expressed as $\log_{10}(10x+1)$ for K1441 and butam

Herbicide	Application method	Dose (kg a.i./ha)	*S. media*		*P. annua*		Turnip		Barley	
			[+]O.L.	P.	O.L.	P.	O.L.	P.	O.L.	P.
K1441	surface	0.8	1.413	1.623	0.440	0.964	1.284	1.529	1.196	1.528
"	"	2.4	1.422	1.777	0.195	0.878	1.168	1.523	1.098	1.473
"	"	7.2	1.527	1.704	0	0.128	1.060	1.358	1.148	1.503
"	incorporated	0.8	1.583	1.718	0.439	0.996	1.588	1.697	1.344	1.345
"	"	2.4	1.472	1.730	0.130	0.377	1.370	1.674	1.107	1.175
"	"	7.2	0.633	1.574	0	0.224	0.970	1.611	1.025	1.180
butam	surface	0.8	1.492	1.568	0.290	0.453	1.447	1.508	1.093	1.479
"	"	2.4	1.335	1.611	0.440	0.478	1.465	1.435	0.906	1.450
"	"	7.2	1.050	0.668	0	0	1.438	1.482	0.858	1.363
"	incorporated	0.8	0.540	0.900	0.078	0.953	1.483	1.539	1.077	1.277
"	"	2.4	0.526	0.663	0.057	0.312	1.593	1.642	0.893	0.900
"	"	7.2	0.494	1.191	0	0	1.495	1.569	0.663	0.652
control			1.353	1.620	0.366	0.880	1.382	1.383	1.212	1.390
S.E. of treated means			± 0.173		± 0.136		± 0.074		± 0.038	
S.E. of control means			± 0.122		± 0.097		± 0.053		± 0.027	

[+]O.L. = organic fine sandy loam; P. = peat control in broad-leaved crops (Richardson and Parker 1977, 1978). These workers found that both herbicides (NP48 was only tested as a post-emergence treatment) did not control P. annua. The pre-emergence control of barley by these herbicides when incorporated into an organic soil suggests that they can be used pre-emergence but due to the problems of incorporation mentioned above they are probably best used as post-emergence applications. Pre-emergence treatments of both herbicides appear to be safe to broad-leaved species.

Butam is an interesting possibility for pre-emergence weed control on organic soils. It controlled P. annua which can be an important weed on these soils and also S. media which suggests some broad-leaved weed control. Glasshouse work on mineral soils reported by Richardson and Parker (1977) suggested that butam might selectively control S. media and other weeds in such crops as radish, pea and dwarf bean. It may be advantageous to incorporate this herbicide to improve its residual weed control activity.

Richardson et al. (1976) suggest that oxyfluorfen could selectively control P. lapathifolium and P. annua in field bean growing on mineral soils. It is doubtful whether it would be selective in any of the vegetable or arable crops grown on organic soils. None of these were tolerant to the rates required to control P. lapathifolium and P. annua. However some other weeds not tested in this organic soil experiment, such as Tripleurospermum maritimum L. and Chenopodium album L., were reported to be selectively controlled on mineral soils.

Whilst K1441 might give useful pre-emergence control of P. annua in such crops as carrots, lettuce, sugar beet and peas (Richardson and Parker, 1976) it has only limited control of other weeds found in these crops on organic soils.

Although Richardson and Parker (1976) reported that triclopyr salt gave some selective control of S. media in barley, oat, field bean and pea the limited activity of this herbicide as the ester formulation on organic soils suggests that it will not be of use as a pre-emergence application. Fluothiuron and 3,6-dichloropicolinic acid did not show enough activity to warrant further testing as pre-emergence herbicides on organic soils.

Of the herbicides tested, hexazinone, ethalfluralin, butam and oxyfluorfen all warrant further testing for pre-emergence weed control on organic soils.

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OLEORESIN STIMULATION WITH PARAQUAT

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Summary Precisely controlled applications of paraquat to pine trees greatly increase production of oleoresin by the tree. To achieve maximum oleoresin yield, the herbicide dose must be adjusted to tree species, tree size and cultural conditions. Paraquat-stimulated oleoresin can be recovered by the normal processes of sulphate pulping or solvent extraction. EPA registration has been obtained and commercial production of paraquat-stimulated oleoresin is beginning in the USA. In addition to being industrially profitable, this use of paraquat provides a potentially renewable resource of valuable organic compounds.

INTRODUCTION

Herbicides are normally used to completely kill unwanted plants. However, they can be used to usefully modify the growth or reaction of crop plants by selectively killing specific tissues or groups of cells. An example of this selective cytotoxicity is the use of sulphuric acid, 2,4-D and other formulations in tapping of pine trees for oleoresin production.

Pine oleoresin occurs in a system of intercommunicating resin ducts which are distributed throughout the tissues of the tree. It is synthesised by epithelial parenchyma cells which line the ducts. The turgor pressure of these cells maintains the oleoresin at a pressure of up to 10 atmospheres. When the ducts are severed by tapping, this pressure causes oleoresin to exude from the tapping cut.

Application of cytotoxic agents to the tapping cut kills the epithelial cells close to the cut. This widens the lumina of the severed ducts, thereby increasing initial flow rate and retarding plugging of the ducts (Ostrom *et al*., 1958). The most widely used flow stimulants possess limited translocation activity. This causes a front of chemical to advance beyond the tapping cut, opening additional resin ducts without need for further mechanical treatment. This significantly reduces labour costs in comparison with traditional methods (Snow 1949).

Screening for more effective oleoresin flow stimulants led to the discovery that the bipyridylium herbicides, paraquat and diquat, increased deposition of large quantities of oleoresin in the wood close to the site of treatment (Kommissarov *et al*., 1968, Roberts 1973). Extraction of oleoresin from pine trees is highly profitable to the sulphate pulping, distillation and solvent extraction industries. These industries are particularly active in the southern states of the USA. It is in that country that much of the industrial development of paraquat for oleoresin stimulation has been carried out.

This paper describes some results of ICI research into the use of paraquat for oleoresin stimulation in species which are commercially important outside the USA.

METHODS AND MATERIALS

All experiments described here were carried out on uniform-aged plantation trees of a size normally felled for pulpwood (i.e., diameter at breast-height (d.b.h.) of 15-30 cm). Treatment solutions were prepared by diluting 'Gramoxone' (20% w/v paraquat ion plus adjuvants) with locally available tap water.

Where precise control of paraquat dose was necessary, this dose, made up in 10 ml of water, was injected into a downward sloping, 11 mm diameter, hole bored along a chord at the base of the trunk, such that the hole intercepted approximately one-third of the medullary rays. An injection axe as supplied by TSI Company of New Jersey, was used in some treatments. This instrument is a light axe, the head of which incorporates a spring loaded piston. In use the axe cuts a 'pocket' through the bark into the wood of the tree. The inertia of the piston automatically injects 1 ml of treatment solution through a hole in the blade. Treatment solution is supplied to the head *via* a plastic tube from a container on the operator's belt. Six pockets were spaced at equal distances around the trunk at each height.

The most frequently used method of application in this trials programme was the 'streak' which is derived from USA tapping practice and terminology. The streak consisted of an area of trunk 2-3 cm high and extending around one-third of the circumference of the trunk, from which the bark and phloem were removed by axe, billhook or tapping tool.

Two streaks were made on the opposite sides of the trunk, leaving two untreated areas each one-sixth of the circumference between their ends. Paraquat solution was applied to run off on the freshly prepared surface. As a basis for comparison it has been assumed that 1 ml of treatment solution was retained per streak. However, tests show that this underestimates the retention of trees greater than 18 cm diameter.

All treatments were applied in spring and early summer. Unless otherwise stated, the trees were harvested in the winter following treatment.

After felling, the treated trees were cut into 50 cm lengths, commencing 50 cm above the treatment site. All bark and phloem were removed from the upper end of each section. A small chain saw lubricated with glycerol was used to make a second cut across the debarked section, collecting the sawdust so produced. This sawdust provided a representative sample of the wood at that height. Care was taken to avoid contamination of this sample with mineral oil, bark or knots, all of which interfere with oleoresin assay.

The sawdust samples were deep frozen in sealed polythene bags, until required for assay. They were then thawed, well mixed and a 5 g sub-sample taken. This sub-sample was soxhlet-extracted with three cycles of spectrally pure methanol, air-dried for several hours and finally dried at 95°C for 48 hours before weighing.

The methanol extract was diluted with further methanol if necessary and its absorbance measured at 242 nm. Its absorbance was compared with that of a standard curve produced by known dilutions of oleoresin extracted from the experimental population of trees. These data were amalgamated to produce a value of percent oleoresin in extractive-free, dry wood. This was converted to kg oleoresin per section of trunk by a computer programme based upon measurements made of tree geometry and wood density of each experimental population.

RESULTS

a) Method of Application

Early trials indicated that paraquat did not readily move through mature pine bark, but that when applied directly to the xylem its effects extended radially and upwards from the point of application. The efficacy of several methods of application was compared over a range of dose rates and on several species. The dose-response curve varied with method of application (Table 1). At the optimum dose rate for each method, the relative efficacy of the alternative methods of application was constant over the range of species tested.

Table 1

Oloeresin yield (kg) of *P. nigra* treated with paraquat

Dose (mg paraquat ion per tree)	2 x ⅓ circumference streak	2 boreholes on opposite chords	1 radial borehole	injection axe
40	2.82	2.08	1.98	4.18
80	5.85	3.91	2.32	6.34
120	7.41	3.93	2.65	5.27

SE ± 2.18

There appeared to be little difference in overall efficacy between injection axe and streak treatment. These treatments were significantly better than borehole treatments.

Comparison of labour output by two application methods was made on 25 cm diameter *P. sylvestris* trees growing 5 metres apart on flat terrain free from undergrowth. Six injection axe blows or 2 x ⅓ circumference streaks treated with billhook and sheep drench gun were applied to each tree. Injection axe treatment permitted 112 trees to be treated per hour. Application by streaks achieved 73 trees treated per hour. The injection axe was considered by the operators to be initially more tiring but less clumsy than streak application. However, long-term use of an injection axe indicates that it is prone to blockage which may not be immediately detected by the operator. It also requires more careful maintenance than equipment required for streaks.

b) Dose-response Relationship

With all methods of application, oleoresin yield varied directly with paraquat dose up to an optimum dose, and above this, yield declined with increasing dose. This overall response appears to reflect the existence of an optimum level of paraquat in the tissues for oleoresin stimulation, above which excessive phytotoxicity occurs. As the applied dose increases, translocation results in rapid attainment of supra-optimal concentration in the tissues near the site of application and displacement of the optimum concentration to regions higher up the trunk. The dose which produces maximum yield per tree is determined by interplay of maintaining optimum concentration in the large-diameter base of the trunk and translocation of effect to higher regions (Table 2).

Table 2

Weight (kg) of oleoresin in trunk sections of P. halepensis

Paraquat ion concentration (% w/v)	Height above treatment point 0-50cm	50-100cm	100-150cm	150-250cm	Total
0.0	0.14	0.11	0.10	0.12	0.47
0.5	0.16	0.09	0.07	0.11	0.43
1.0	0.72	0.51	0.11	0.13	1.47
2.0	1.06	0.40	0.26	0.11	1.83
3.0	1.28	0.52	0.24	0.17	2.21
4.0	1.87	0.85	0.40	0.11	3.23
6.0	1.11	0.96	0.74	0.52	3.33
SE(±)	0.34	0.29	0.32	0.21	0.44

c) Reaction of different pine species

It was not possible to treat a range of pine species growing on the same site. Thus a direct comparison of dose-response relationships between species cannot be made due to confounding factors such as site and climatic differences. Nevertheless, several trials on individual species support the conclusion that the optimum paraquat dose and potential oleoresin yield of one species growing in conditions normal to that species are different to the optimum dose and yield of other species in their normal environment. Optimum dose rates tend to be lower for species which have high growth rates and porous wood, such as P. elliottii and P. laricio. Yields tend to be high in naturally resinous species such as P. pinaster and P. elliottii (Table 3).

Table 3

Oleoresin yield (kg) of different pine species

Paraquat ion concentration (% w/v)	P.sylvestris	P.laricio	P.pinaster	P.elliottii	P.patula
0	0.65	1.02	2.29	2.00	1.21
1	1.77	2.03	3.94	11.10	2.50
3	1.95	3.60	5.79	11.40	6.19
4	7.20	2.38	5.91	16.00	5.45
6	7.41	2.31	14.46	10.40	4.22
SE(±)	2.18	0.91	3.46	6.2	1.72
Tree age (years)	26	18	40	14	15
Tree diameter (cm)	22	25	25	26	28

d) Tree size

Trees greater than 25 cm d.b.h. and trees less than 16 cm d.b.h. growing in the same plantation were treated with equal doses of paraquat injected into a borehole.

The oleoresin yields (Table 4) showed that the large trees had a higher optimum paraquat dose, and high potential yield than small trees.

Table 4

Oleoresin yield (kg) of *P. sylvestris*

Dose of paraquat ion (mg/tree)	Large trees		Small trees	
	Total	Increase over control	Total	Increase over control
0.0	0.89	0.00	0.21	0.00
20	2.08	1.19	0.72	0.51
40	2.70	1.81	0.75	0.54
80	3.05	2.16	0.61	0.40
120	3.93	3.04	0.60	0.39
SE(±)	2.18	2.11	0.59	0.68

e) Further factors which affect yield

Oleoresin yield increases with the length of time between paraquat treatment and harvest. Thus in a trial on *P. sylvestris* treated in early summer, mean yield per tree was 2.6, 2.8, 3.4 and 3.5 kg oleoresin, in autumn, spring and autumn of the following year and the following spring respectively. Several trials support the conclusion that oleoresin yield continues to increase for at least two years following treatment. There is also evidence that the rate of oleoresin increase is greater during the growing season than in the dormant season.

Comparison of yields from trees treated with the same dose but growing in different environments is not a strictly valid test of environmental effects since the trees do not belong to the same population. Nevertheless, the yields obtained from trials on *P. sylvestris* growing in contrasting environments indicate that, in practice, oleoresin yields are lower where growth is limited by exposure, low temperature or drought (Table 5).

Table 5

Oleoresin yield (kg) of *P. sylvestris* at different sites

Paraquat ion concentration (% w/v)	Southern England	Central Spain	Lowland Scotland	Highland Scotland
0	0.65	0.59	0.63	0.71
1	1.77	0.93	1.78	1.02
3	2.95	1.37	2.58	1.20
6	7.41	2.82	4.78	1.47
SE(±)	2.18	0.76	1.70	0.88
				0
Tree age (years):	26	73	42	45
Mean tree diameter(cm):	23	28	20	11

f) Effect on oleoresin composition

Pine oleoresin is composed mainly of a mixture of volatile monoterpenes and terpenoids, less volatile higher terpenoids, fatty acids and sterols. These components are important to different commercial users. The relative concentrations of these constituents in paraquat-treated and untreated wood of the same species was investigated by standard extraction and gas-liquid chromatography procedures.

The rate of increase in volatile components of oleoresin of P. radiata was four times that of the non-volatile components in the first two weeks after treating seedling trees. Similarly, paraquat-treated P. pinaster harvested one year after treatment produced 196% more volatile and 77% more non-volatile oleoresin constituents than the control trees. This increased to 322% volatiles and 256% non-volatiles in the second year after treatment. The main increase in non-volatile constituents was due to increased resin acids, with a small increase in unsaponifiable constituents and no increase in fatty acids.

The volatile constituents of oleoresin from the paraquat-treated and untreated portions of the same tree were compared. In all species investigated, the paraquat-stimulated oleoresin contained a higher concentration of monoterpenes and lower concentration of oxygenated terpenoids than the untreated oleoresin (Table 6).

Table 6

Composition (%) of oleoresin volatiles

	P. sylvestris		P.pinaster	
	untreated	treated	untreated	treated
α -pinene	65	78	41	52
β -pinene	2	9	24	28
Other monoterpenes	4	5	7	12
Oxygenated monoterpenes	24	3	26	4
Sesquiterpenes	5	5	2	4

g) Side effects of treatment

Paraquat treatment kills the phloem and cambial tissues above the site of application. The extent of this is influenced by dose rate and seldom exceeds 3 metres above the treatment site. This results in an absence of wood growth in the paraquat-affected zone, although in some instances a compensatory increase of cambial activity in adjacent regions has been observed.

The dead phloem is commonly invaded by insects normally associated with stressed trees, such as Tomicus piniperda L.. Longicorn beetles infested treated wood of trees which were maintained into the second growing season. In no case did the non-paraquat affected tissues of a treated tree become infested. In fact, in some trials with a high intensity of infestation by Tomicus piniperda, the larvae consumed all dead phloem before completing their life cycle and died without attacking adjacent healthy phloem. Not a single tree has been killed by insect attack in >100 development trials carried out by ICI in six countries.

In some trials, the paraquat-affected wood was infected by unidentified blue-stain fungi, particularly at high dose rates on small trees. This fungus did not invade adjacent untreated wood, and seldom extended more than 1 metre above the treatment site.

The effects of doses of paraquat optimally stimulating oleoresin production seldom exceeded 3 metres above the site of application. Dose rates as high as 400 mg per tree of paraquat ion have been applied to P. sylvestris without killing the tree. However, in a trial with injection axe and streak treatments applied during periods of rapid transpiration, 5% of the trees died. Death was associated with high dose rate, and injection axe treatment. This appeared to cause 'ring barking', or death of phloem around a substantial part of the trunk at treatment height.

DISCUSSION

The mode of action of paraquat in stimulating oleoresin production has not been completely elucidated. The mechanism may be a direct stimulation of oleoresin synthesis by xylem parenchyma cells (Clason, 1976) or an indirect effect mediated by paraquat damage to the resin ducts. Both hypotheses involve selective killing of cells at the base of the tree without damage to the photosynthetic tissue in the crown. Consequently, the extent of paraquat phytotoxicity must be carefully regulated to minimise undesirable side effects.

The severity of effect is influenced by tree species, size, vigour, environmental conditions, dose rate and method of application. Only the last two factors can be precisely controlled, but it is possible to select trees for treatment bearing in mind the effects of the former factors. Thus choice of large trees belonging to a rapidly growing, resinous species established on sites which permit a high growth rate will tend to maximise oleoresin production from a given treatment.

Since a single treatment continues to stimulate oleoresin production for more than one year after application, there is a strong case for harvesting treated trees at least one growing season after paraquat application.

The advantage of delayed harvest will be modified by administrative and possibly further technical considerations. In the USA, Dendroctonus frontalis Zim, a bark beetle, rapidly kills paraquat-treated trees. This problem has encouraged a trend towards a short interval between treatment and harvest in the USA. Insects which rapidly kill pine trees are not common outside the Americas. Insect-induced mortality has not been observed in the extensive trials programme carried out by ICI in Australasia, Asia, Africa and Europe. Consequently, it is considered probable that oleoresin stimulation in these continents will have an economic advantage over its practice in the USA.

In all trials carried out with 2 x ⅓ diameter streaks, the optimum dose of paraquat (or highest dose if this was sub-optimal) at least doubled oleoresin yield compared with control trees. In some species treated one year before harvest, yields have been up to 4 or 6 times those of untreated controls. This increase in yield is mainly due to an increase in resin acids and monoterpenes. These are the most commercially valuable fractions of the total oleoresin.

Paraquat-stimulated oleoresin can be recovered by the normal processes of kraft-pulping, solvent extraction or distillation. The most economic method is by pulping. Large scale trials in the USA have shown that the increased oleoresin content of paraquat-treated pines can be profitably recovered without modificaiton to normal pulping procedures (Rothrock and Rhyne, 1976).

Infestation by insects and blue-stain mould do not affect the pulping characteristics of the wood. There is no evidence that the absence of wood increment in paraquat affected zones of the trunk is in fact a loss of harvestable wood increment. However, even if this were so, the value of the 1 kg or so per tree which would otherwise have been produced in the affected areas is much less than the value of oleoresin produced.

The value of oleoresin is not less than $150 per tonne, its recovery costs during kraft pulping are only a few dollars per tonne. Pulpwood costs less than $50 per tonne. It is, therefore, easy to understand why the possibilities of using paraquat to stimulate oleoresin production has been greeted with such enthusiasm by the pulp industry of the USA. Intensive development has demonstrated the efficacy, safety and favourable residue levels of the treatment. EPA approval has been granted and commercial use is beginning.

In addition to augmenting the profit of forest and pulping industries, paraquat-stimulation of oleoresin has wider implications. The terpenoid components of pine oleoresin are valuable chemical raw materials which cannot be profitably extracted from other plants, and which can be made only at high energy cost from fossil hydrocarbons. Nevertheless, until recently there has been a trend towards using synthetic substitutes because of the falling world production and fluctuating prices of natural oleoresin. Paraquat-stimulation potentially can increase oleoresin production and stabilise prices, thus rendering this renewable resource of hydrocarbons commercially and socially competitive with crude oil for production of selected organic compounds.

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THE EFFECT OF CHLOMETHOXYNIL ON TRANSPLANTED PADDY RICE UNDER UNFAVOURABLE GROWTH CONDITIONS

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Summary The rooting and early growth of transplanted paddy rice were markedly reduced when cut straws were incorporated in the soil shortly before transplanting. This growth reduction was correlated with root growth inhibition first observed about one week after transplanting. The chloroplast protein in the leaves and protein and protein-SH content in the roots were also decreased. In addition, the oxidation-reduction potential in the soil was rapidly reduced after the incorporation of cut straws. Chlomethoxynil did not increase the damage caused by straw incorporation, nor did it influence reduction of growth at low temperature.

INTRODUCTION

Chlomethoxynil*(2,4-dichlorophenyl-3-methoxy-4-nitrophenyl ether) discovered and developed by Nihon Nohyaku Co. Ltd. and Ishihara Sangyo Kaisha Ltd. was evaluated as a pre- and early post-emergence herbicide in paddy rice in Japan. Chlomethoxynil at 2.1 to 2.8 kg a.i./ha is effective against Echinochloa oryzicola, and many other weeds including Monochoria vaginalis, Cyperus difformis, Eleocharis acicularis, Sagittaria pygmaea and Scirpus juncoides. This herbicide also has good selectivity between rice and weeds with long residual activity in paddy field (Fujikawa et al. 1973).

It has been commonly practiced in paddy field in Japan that straws cut by combine-harvester have been incorporated in the soil after harvest in autumn or shortly before transplanting in spring to save the lavour for their removal and to maintain soil fertility. However, the incomplete decomposition of straws in the soil when incorporated shortly before transplanting is known to be unfavourable some times for the rooting and early growth of transplanted paddy rice. This damage increases when young seedlings at about 2.5 leaf age are transplanted under low temperature conditions. Unfortunately, very little information is available on the effect of herbicide on rice growth under unfavourable growth condition such as the incorporation of cut straws in the soil.

The purpose of the present study is to examine the effect of chlomethoxynil on transplanted paddy rice in relation to the incorporation of cut straws in the soil and to low temperature.

METHODS AND MATERIALS

Effect of incorporation of cut straw. The experiment to examine the interaction between different dosages of chlomethoxynil and the incorpora-

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* Common name approved by JMAF

tion of cut straws in the soil was conducted in the open in 1978. The temperature averaged 23°C by day and 16°C by night during the experiment. Dried straws at 10 t/ha were cut in small pieces and mixed with sandy clay loam in 15 cm diameter pots. One day after incorporation, young seedlings at about 2.5 leaf age were transplanted and a 7 % granular formulation of chlomethoxynil at 2.1 and 6.3 kg a.i./ha was applied two days after transplanting under flooding conditions. Plant height, leaf age, fresh weight of shoots and roots were determined 28 days after treatment.

Another experiment was conducted to examine the effect of double treatment of chlomethoxynil at excess dosage on transplanted paddy rice grown in the soil with different amounts of straw incorporated, under greenhouse conditions. Dried straws at 5, 10 and 20 t/ha were mixed with soil two days before transplanting. Two and fifteen days later, chlomethoxynil at 6.3 a.i./ha was applied twice under flooding conditions, a water depth of 3 cm being maintained during the experiment. Plant height (longest vegetative leaf), number of shoots, dry weight of shoots and roots per plant were examined 39 days after transplanting.

Changes in oxidation-reduction potential in the soil at 30°C with and without straw incorporation were determined using the glass electrode method at several intervals.

Effect of temperature. Transplanted paddy rice was treated with chlomethoxynil at 2.1 kg a.i./ha and grown in growth cabinets at day/night temperature of 30/25°C, 25/20°C, 20/15°C. Natural daylight was supplemented with low-intensity fluorescent lamps during the experiment. Leaf age, plant height, number of shoots, dry weight of shoots and roots per plant were determined 26 days after transplanting.

Effect on chloroplast protein. The second and third leaves were sampled 22 days after transplanting and analysed for chloroplast protein. The procedure for chloroplast isolation and determination of chloroplast protein were almost similar to those outlined by Jagendorf et al. (1957) and Akobundu et al. (1975), respectively.

Effect on protein and protein-SH. Protein-SH was analysed by the method of Sakai (1968).

RESULTS AND DISCUSSION

Effect of incorporation of cut straw. Table 1 shows the effect of different dosages of chlomethoxynil in relation to the incorporation of cut straw in the soil. Incorporation caused a marked reduction in plant height and fresh weight of shoots and roots per plant (73%, 58% and 43% of control). Shoot growth reduction was closely related to root growth inhibition first observed about one week after transplanting. Chlomethoxynil at 2.1 and 6.3 kg a.i./ha caused some growth reduction in the absence of straw incorporation, but did not substantially increase damage to the crop caused by straw incorporation.

In the second experiment, plant height, number of shoots and dry weight of shoots per plant were progressively reduced as the amount of straw increased as shown in Table 2. The double treatment of chlomethoxynil at the excess dosage of 6.3 kg a.i./ha caused no growth reduction alone, nor did it increase damage attributable to straw incorporation.

Changes in oxidation-reduction potential. The incorporation of cut straw in the soil markedly affected the oxidation-reduction potential. As indicated in Fig. 1, the potential at pH 6 (Eh 6) rapidly decreased to about -0.15 V five days after flooding when cut straw was incorporated, whereas without incorporated straw, the potential gradually decreased, maintaining oxidizing conditions during the experiment. Reducing conditions in the soil seem to be an important factor in causing plant growth inhibition, as many workers have pointed out.

Table 1

Effect of dosages of chlomethoxynil and incorporation of cut straw in the soil

Amount of straw (t/ha)	Dosages (kg a.i./ha)	leaf age	Height (cm)	Fresh weight (mg/plant)	
				Shoots	Roots
0	0	3.5 (100)	19.2 (100)	210.5 (100)	122.8 (100)
	2.1	3.5	18.3	190.6	97.2
	6.3	3.5	16.7	169.8	89.1
10	0	3.2 (100)	14.1 (73)	123.0 (58)	53.2 (43)
	2.1	3.2	14.4	117.1	54.1
	6.3	3.1	13.8	110.1	44.6

Table 2

Effect of double treatment of chlomethoxynil at excess dosage and amount of cut straw in the soil

Amount of straw (t/ha)	Dosages (kg a.i./ha)	Height (cm)	Number of shoots/plant	Dry weight (g/plant)	
				Shoots	Roots
0	0	67.4 (100)	4.9 (100)	1.00 (100)	0.22 (100)
	6.3	66.0	4.9	0.99	0.22
5	0	65.9 (98)	4.4 (90)	0.97	0.31 (141)
	6.3	63.2	4.0	0.72	0.24
10	0	63.6 (94)	3.7 (76)	0.70 (70)	0.28 (127)
	6.3	60.7	4.1	0.76	0.30
20	0	54.6 (81)	2.3 (47)	0.40 (40)	0.18 (82)
	6.3	55.3	2.7	0.38	0.20

Effect of temperature. Fig. 2 indicates the effect of chlomethoxynil on transplanted paddy rice grown under three temperature regimes. Low temperature at day/night of 20/15°C considerably reduced rice growth, particularly dry weight accumulation in the shoots. No difference in growth between chlomethoxynil-treated and untreated rice within the three temperature regimes were observed.

Effect on chloroplast protein in the leaves. As chloroplasts are known to have the capacity for protein synthesis, the content of chloroplast protein was determined to examine the physiological activity of treated rice. Table 3 shows the effect of different dosages of chlomethoxynil

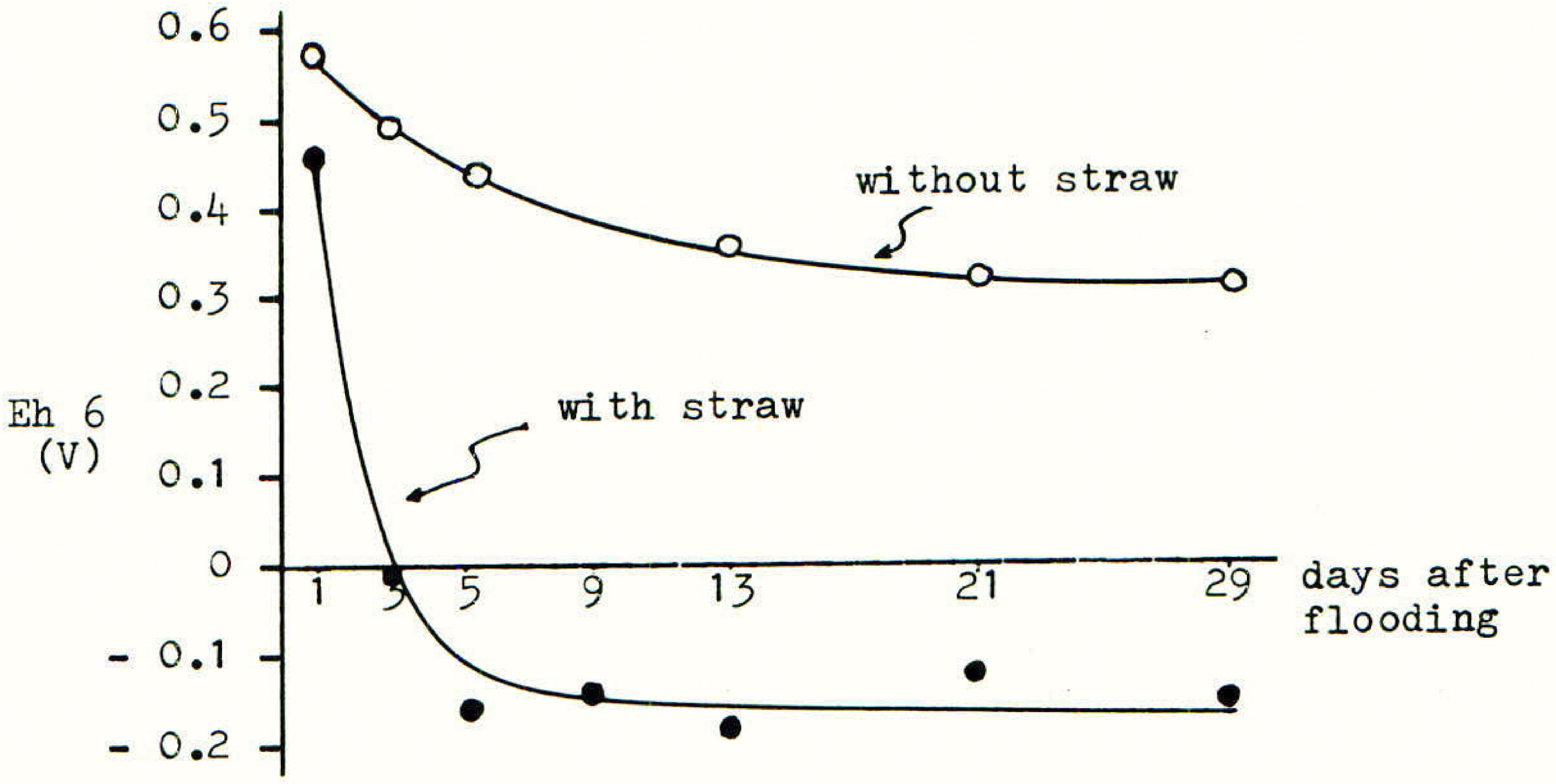

Fig. 1

Changes in oxidation-reduction potential

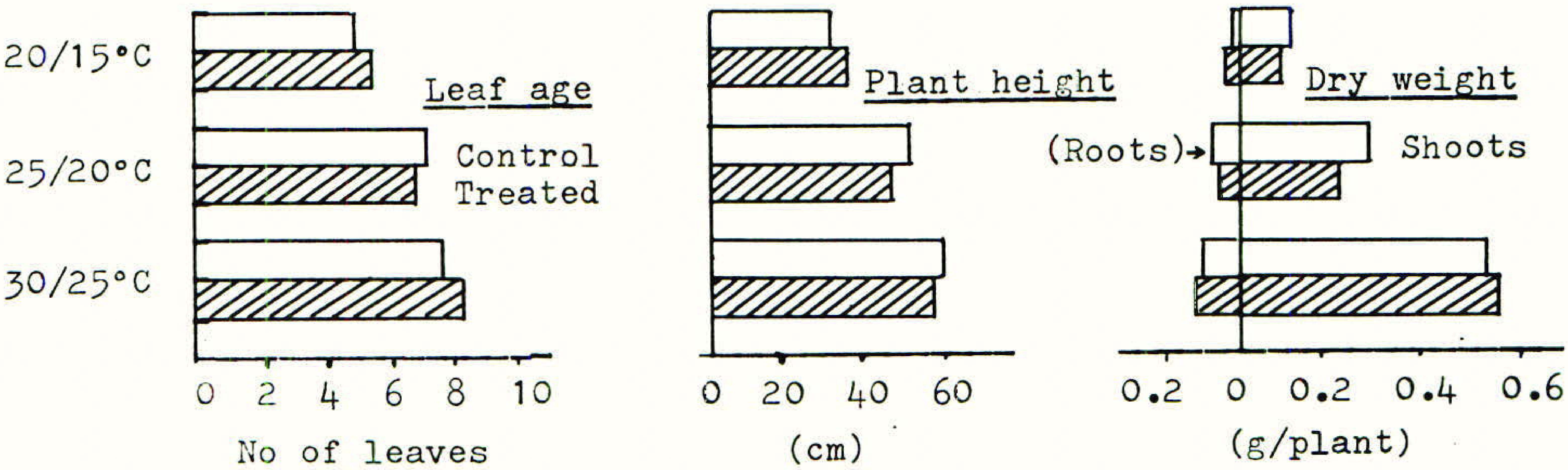

Fig. 2

Effect of temperature

and the incorporation of cut straw on chloroplast protein in the leaves. At recommended dosage of 2.1 kg a.i./ha, chlomethoxynil showed no effect, but at the higher dosage of 6.3 kg a.i./ha, chloroplast protein was reduced. Incorporation of cut straw markedly reduced the content, to 40% of that of control plants.

Akobundu et al. (1975) reported in their study on the synergism of atrazine and alachlor combinations on Japanese millet that atrazine alone and in combination with alachlor caused a marked reduction in the chloroplast protein content. Singh et al. (1967) also found a reduction in the chloroplast protein in oats treated with atrazine. The present result, in conjunction with the reports mentioned above, indicates that the physiological activity of green plant is reflected by chloroplast protein content.

Table 3

Effect on chloroplast protein content

Amount of straw (t/ha)	Dosages (kg a.i./ha)	Chloroplast protein content mg/g fw	Chloroplast protein content µg/plant
0	0	1.23 (100)	0.62 (100)
	2.1	1.24	0.62
	6.3	0.84	0.42
10	0	0.49 (40)	0.25 (40)
	2.1	0.66	0.33
	6.3	0.30	0.15

Effect on protein and protein-SH in the roots. The protein and protein-SH content were determined to examine the relation between rooting ability and protein-SH. As shown in Table 4, the incorporation of cut straw in the soil increased the protein-SH content to 171% on protein basis , compared with control. This result is unexpected because soluble-SH such as glutathione is known to increase when active cell division occurs. The increase in protein-SH may be related to the conformational change through SH/S-S ratio. Further studies will be needed to clarify the present result. Chlomethoxynil at 2.1 and 6.3 kg a.i./ha had little effect on the content.

Table 4

Effect on protein and protein-SH content of roots

Amount of straw (t/ha)	Dosages (kg a.i./ha)	Protein content		Protein-SH content		
		µg/mg fw	µg/plant	µg/mg fw	µg/mg protein	µg/plant
0	0	9.70 (100)	1.80 (100)	0.10 (100)	10.4 (100)	11.4
	2.1	8.34	1.06	0.08	10.0	10.6
	6.3	8.20	0.91	0.10	12.2	11.1
10	0	14.16 (146)	0.75 (42)	0.25 (250)	17.8 (171)	13.3
	2.1	15.11	0.78	0.25	16.2	12.6
	6.3	13.41	0.62	0.31	23.4	14.4

CONCLUSION

The conclusion derived from the present results is that chlomethoxynil can be safely used for transplanted paddy rice under unfavourable growth conditions such as cut straw incorporation in soil and low temperature. Tanigawa *et al*. (1978) supported the present result in their study on the effect of several herbicides on the paddy rice grown with straw-incorporated soil by showing that chlomethoxynil was the safest of those tested.

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