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Worldwide Review Of The New Cereal Herbicide - DPX 4189

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Summary DPX 4189 is a very active new herbicide with excellent crop selectivity in wheat, barley, oats and rye. The compound is non-volatile with activity at 10-25 g a.i./ha on most broadleaved weeds and giving some suppression of a few grass weeds. Several species tolerant to phenoxy and benzoic herbicides are sensitive. DPX 4189 is active due to uptake through both foliage and root system.

Résumé DPX 4189 est un nouvel herbicide particulièrement actif doué d'une excellente selectivité sur blé, orge, avoine et seigle. Le produit n'est pas volatile. Son efficacité se situe entre les doses de 10 et 25 gm m.a. à l'hectare pour le controle de la plupart des mauvaises herbes à feuilles larges et il permet d'obtenir l'élimination d'un certain nombre de graminées. Plusieures éspèces résistantes aux herbicides de la famille des phenoxys et des dérivées benzoïques sont sensibles. DPX 4189 est actif grâce à sa pénétration à la fois par voie foliaire et radiculaire.

INTRODUCTION

DPX 4189 (2-chloro-N- [(4-methoxy - 6-methyl-1,3,5-triazin-2-yl)-benzenesulfonamide) has been developed as a selective herbicide for cereals such as wheat, barley, oats and rye. Introductory papers by Finnerty and Schehl (1979) and Baber and McKinley (1980) gave regionalized reviews of field research for the North Central Plains and the Pacific Northwest of the United States respectively. Jensen (1980) summarized his field research in Scandinavia showing efficacy at remarkably low rates with enhanced activity on certain weed species with the addition of a nonionic surfactant. Levitt, et al (1980) summarized initial worldwide developmental efforts of DPX 4189.

Miller (1979) reported good control of broadleaved and some grass weeds under North Dakota conditions. Spring wheat and barley tolerance was excellent. There was no differential yield response to postemergence DPX 4189 treatments among seven spring wheat cultivars, four barley, three oat and three durum wheat cultivars in Minnesota (Hageman and Behrens, 1979). Excellent efficacy has been reported in the Intermountain Area of the United States (Morishita and Lee, 1980). O'Sullivan (1980) reported on broad spectrum weed control in Western Canada and possibly some antagonism by DPX 4189 towards certain wild oat herbicides. Nalewaja (1980) pointed out the need for longer residual herbicides which have selective tolerance to the following crop. DPX 4189 for fallow weed control before wheat, increasing the energy efficiency of crop production is an example of this.

This new herbicide has favorable toxicology with a low acute oral toxicity; 5,545 mg/kg for male rats. DPX 4189 showed no adverse effects in reproductivity tests, no skin irritation, mild temporary eye effects and low order of dermal and inhalation toxicity. It is not mutagenic, embryotoxic or teratogenic. The residue picture appears favorable in cereal crops for both grain and straw.

The purpose of this paper is to give a summary of the most promising ways DPX 4189 can provide weed control in cereal crops.

METHODS AND MATERIALS

Initial field testing was done using an 80% wettable powder while trials in 1980 used 20 and 75% dry flowable formulations. Tests in 1978 evaluated rates of 70-280 g a.i./ha. Rates were reduced in 1979 to 20 g a.i./ha and in some cases 10 g a.i./ha. In 1980, most research was conducted at 10-40 g a.i./ha and some as low as 2.5 g a.i./ha.

Most trials used small replicated plots, CO_2 pressurized hand carried sprayers and spray volumes of 80-400 l/ha. Aerial tests used 15-30 l/ha.

Recorded effects varied among countries but included weed counts, weights or estimates by species. In each test, the crop was rated for vigor; many tests were harvested to determine yield.

RESULTS

International results are summarized by crop tolerance, weed control and rates as influenced by crop rotation.

Crop Tolerance

Winter and spring wheat and barley have shown greatest tolerance to postemergence treatments. Rates as high as 70-100 g a.i./ha have commonly not reduced yield. Wheat has adequate tolerance to preemergence treatments while barley does not.

Under European conditions, winter wheat has shown no injury at 40 g a.i./ha preemergence or early postemergence in the autumn which offers at least a 2X margin of safety. Autumn postemergence treatments up to 30 g a.i./ha, are safe on winter barley.

Weed Control

This active new compound has a remarkably flat curve in response to rates. Rates in field research have been reduced in the last two years from 70-280 g a.i./ha to 5-60 g a.i./ha with very little loss of activity on many broadleaved weeds. The low rates still contribute to the control of grass weeds.

In the spring cereal belt of Canada and North Central United States, 20-30 g a.i./ha by itself as a postemergence treatment is adequate to control the typical broadleaf weed spectrum. This commonly includes *Cirsium arvense*, *Galium*, *Galeopsis*, *Chenopodium*, *Polygonum spp*., *Stellaria*, *Kochia*, *Amaranthus*, *Spergula*, *Salsola*, Rumex, Matricaria and all of the Cruciferae family where the compound has been tested. In the winter cereal belt in North America and Australia, similar rates also control Helianthus, Erodium, Lithospermum, Amsinckia, Lamium, Oxalis and Emex. At rates of 35-70 g a.i./ha, early postemergence treatments provide significant control of Lolium, Setaria, and Poa.

Climatic conditions vary considerably within the cereal belt of North America. DPX 4189 performs as well under cold as warm air temperatures. Herbicidal responses develop fastest under good growing conditions - warm wet weather. Under dry conditions, the susceptible weeds cease growth and remain suppressed for several weeks before death. In some tests in low humidity situations, linseed oil and nonionic surfactants e.g. Surfactant WK and Citowett have enhanced activity on certain broadleaf and grassy weeds without loss of crop selectivity. Chenopodium album, Salsola kali and Chrysanthemum are the most responsive to surfactant addition while Lolium multiflorum, Helianthus annuus, Polygonum aviculare, P. convolvulus and Galium aparine have shown significantly increased sensitivity.

DPX 4189 activity is less sensitive to soil texture, pH and organic matter than most other soil residual herbicides. Treatments on gravelly-sandy soils with less than 1% organic matter have shown the most activity.

In NW Europe DPX 4189 has demonstrated additive activity on Alopecurus myosuroides as well as broadening the spectrum of some commonly used herbicides (Table 1).

Table 1

Percent Weed Control In Winter Wheat By Preemergence Treatments in France (Mean of Nine Trials)

		Weed Species									
Treatment	Rate g a.i./ha	Alopecurus	Poa	Lolium	Veronica	Matricaria	Stellaria	Ranunculus	Papaver	Poly. avic.	Viola
Neburon	3600	35	87	80	22	90	95	60	100	81	95
Neburon + DPX 4189	3600 + 15	83	94	80	52	90	99	100	100	95	100
Neburon + DPX 4189	3600 + 30	81	97	90	63	97	100	100	100	99	100
Chlortoluron	2500	68	80	100	10	96	87	40	52	73	40
Chlortoluron + DPX 4189	2500 + 15	92	94	100	61	93	94	100	62	89	90
Chlortoluron + DPX 4189	2500 + 30	94	97	100	67	100	98	100	60	93	100
Isoproturon	2500	66	67	100	25	55	57	35	45	33	30
Isoproturon + DPX 4189	2500 + 15	81	88	70	52	63	95	90	85	63	90
Isoproturon + DPX 4189	2500 + 30	84	90	100	57	87	97	95	58	70	95
Methabenzthiazuron	2800	71	93	100	63	100	91	50	100	73	90
Methabenzthiazuron + DPX 4189	2800 + 15	96	98	100	94	100	99	100	100	83	90
Methabenzthiazuron + DPX 4189	2800 + 30	95	95	100	94	99	100	100	100	95	90

Grass weeds are generally more susceptible to treatments before emergence or early post versus late postemergence. Rainfall has been more consistent in activating DPX 4189 than shallow incorporation. Early postemergence treatment is optimal for most broadleaved weeds. The soil residual effects of a postemergence treatment, if activated by rainfall, eliminates or markedly retards the growth of most weeds emerging after treatment.

DPX 4189 was tested in combination with other herbicides to give broader spectrum spring weed control. Five and 10 g a.i./ha of DPX 4189 as a postemergence application in the spring has considerable activity on Stellaria and Matricaria and contributes to control of Galium and Lamium (Tables 2 & 3). The combination of DPX 4189 + Ioxynil or MCPP gave acceptable control of problem spectrums of broadleaved weeds.

Table 2

Percent Broadleaf Weed Control Obtained By Postemergence DPX 4189 And Other Sprays In A Winter Barley Trial In W. Germany

Treatment	Rate g a.i./ha	Weed Species Stellaria	Viola	Galium	Lamium
DPX 4189	5	90	15	55	50
DPX 4189	10	95	20	75	60
DPX 4189	20	95	30	95	80
Ioxynil	300	65	45	60	30
DPX 4189 + Ioxynil	10 + 200	98	95	98	98
MCPP	820	90	*	98	20
DPX 4189 + MCPP	10 + 490	99	*	98	85

*Weed Not Present

Table 3

Percent Broadleaf Weed Control With Postemergence DPX 4189 And Other Sprays In Winter Cereals in the UK (Mean of Three Trials)

Treatment	Rate g a.i./ha	Weed Species Veronica	Viola	Stellaria	Matricaria	Galium	Lamium
DPX 4189	10	39	42	90	98	66	57
DPX 4189	20	60	38	100	84	89	50
DPX 4189 + Ioxynil	10 + 300	56	32	92	100	71	82
DPX 4189 + Ioxynil + Bromoxynil	10 + 150 + 150	70	59	97	93	100	100
Ioxynil	300	65	33	45	75	78	18
Ioxynil + Bromoxynil + MCPP	125 125 690	70	63	95	67	89	86

Variable soils and climatic conditions in Europe have not significantly changed efficacy. Some trials in France and Turkey were installed in temperatures of 2-5°C with consistently better results than with hormone type herbicides which may be temperature sensitive.

Rates/Crop Rotations

DPX 4189 has a half-life in the soil of 1-2 months. Degradation is by hydrolysis to inactive compounds. The rate of breakdown is influenced by soil temperature - nil below 5°C. Hydrolysis is faster with low soil pH.

Although the half-life is typical of soil residual herbicides, some crops are sensitive to the usual small residues. Corn, sorghum, flax, potatoes, soybeans and, of course, cereal crops adequately tolerate the residues present in the year following treatment. Rates of 60-140 g a.i./ha have shown no injury to these crops twelve months after treatment. Sugar beet, mustard and rape are more sensitive; the rate of application in a preceeding cereal crop has to be lower if a sensitive crop is to follow. Field research in the UK, France and W. Germany shows a more rapid disappearance with autumn compared to spring applications. Rates of 80 g a.i./ha and below on winter cereal in the autumn have not injured sugar beet planted 18 months later. In contrast, spring treatments have resulted in detectable injury to sugar beet from 30 g a.i./ha in these three countries.

DISCUSSION

DPX 4189 promises to be a useful herbicide for cereals. Its broad spectrum of activity and excellent crop tolerance are desirable especially when combined with the unusually good flexibility in time of application and its relatively low sensitivity to soil type, rainfall and temperature variabilities.

Environmentally this new compound is desirable due to the small amount of material that is applied per hectare. In many areas non-volatility is also helpful.

The primary mode of action is inhibition of plant cell division (Ray 1980). This plant response is very evident in field results as growth on sensitive species stops or is generally very suppressed. Weeds that are large at time of treatment may survive for several weeks but not signigicantly compete with the crop or give problems in harvesting.

DPX 4189 has demonstrated control of several hormone tolerant species as well as some generally resistant species e.g. Cirsium arvense, Galeopsis tetrahit, Chrysanthemum segetum, Chorispora tenella, Lepidium campestre, Matricaria spp., Emex australis, Galium aparine, Lithospermum spp., Amsinckia lycopsoides, Oxalis pes-caprae, Apera spica-venti and Ranunculus spp.

Where sensitive crops like sugar beet and autumn seeded rape are grown in rotation, the rates to evaluate are 15-30 g a.i./ha as a preemergence treatment on winter wheat or as an early post treatment on winter wheat or barley in the autumn. Combinations of such rates with reduced rates of methabenzthiazuron, isoproturon, neburon or nitrofen have been impressive in Europe while combinations with diuron and metribuzin have been good in North America since they help control Lolium spp. and Bromus spp. respectively. Addition of a surfactant to DPX 4189 assures more foliar activity under dry conditions. Where Avena fatua is a problem, combinations or sequential treatments with a wild oat herbicide are necessary.

Selective residual activity of DPX 4189 can be utilized where cereals, corn, sorghum, potatoes or flax follow the treated crop. DPX 4189 at higher rates (20-60 g a.i./ha) can generally provide full season, broad spectrum control of most weeds with the exception of Avena fatua, Solanum and Bromus. It has much to offer in providing weed control between the short, idle periods between continuous cereal crops and in the long, fallow periods where no crop is grown for one season. The following cereal crop is tolerant to the soil residues. These fallow practices are common to the semi-arid areas of the Great Plains of North America and Australia.

DPX 4189 is environmentally a safe compound which will provide the cereal grower a unique tool to achieve full season broad spectrum weed control. It appears to be compatible with most other cereal herbicides and is flexible in time of application.

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STUDIES ON THE MODE OF ACTION OF DPX-4189

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Summary DPX-4189 is a new selective herbicide from Du Pont for broad spectrum weed control in cereals. Mode of action studies have revealed that it is a very potent and rapid inhibitor of cell division and plant growth. Cell division was inhibited within 6 hrs of application at micromolar or nanomolar concentrations. Cell elongation was not initially affected and photosynthesis, respiration, protein synthesis and RNA synthesis all remained normal after growth had been arrested.

INTRODUCTION

DPX-4189 (2-chloro-N-[(4-methoxy-6-methyl-1,3,5-triazin-2-yl)-aminocarbonyl]benzenesulfonamide) is a new herbicide from Du Pont for weed control in cereals. An important feature of this compound is its very high herbicidal activity. Recommended rates for weed control in wheat, oats and barley are between 10-60 g a.i./ha (Levitt, et al., 1980). Its mammalian toxicity is low, LD_{50} for male rats, 5,545 mg/kg (Du Pont, 1980).

Although seed germination is not normally affected, subsequent seedling growth is severely inhibited in susceptible plant species. Plant death is often slow and is accompanied by chlorosis, necrosis, terminal bud death and vein discoloration. DPX-4189 is readily taken up by the foliage and roots of plants and moves systemically. Tolerance in crops like wheat and barley is primarily related to the degradation of DPX-4189 to inactive products.

In order to better understand the unique herbicidal activity of DPX-4189, investigations were carried out to determine its mode of action. Numerous metabolic processes were studied including photosynthesis, respiration, nucleic acid and protein synthesis, as well as plant growth.

METHODS AND MATERIALS

Growth of Plant Material. Soybean, corn, and wheat used for intact plant studies were grown in controlled environmental rooms with the conditions optimum for each species. Corn and pea seedlings used for nucleic acid and protein synthesis studies were grown in the dark at 30°C and used 48 hours after sowing. Explants of dormant tubers of *Helianthus tuberosus* were grown in solution culture under sterile conditions in Whites medium containing 2×10^{-6} M 2,4D which induces cell division and rapid growth.

Plant Growth Measurements. Continuous growth measurements of corn and wheat seedlings were made using a linear variable differential transformer (Model #243-000, Trans Tek Inc., Ellington, Conn.) essentially as described by Hsiao, et.al. (1970).

Photosynthesis and Respiration. Photosynthesis was measured by following either ferricyanide catalyzed O_2 evolution or the methyl viologen catalyzed Mehler reaction using a Clark type oxygen electrode. Both pea and spinach chloroplast preparations were used (Lilly, *et al.*, 1975). Respiration was measured by determining the rate of O_2 uptake by excised pea roots using an oxygen electrode.

Nucleic Acid and Protein Synthesis. Following various herbicide treatments, plant tissue (usually intact seedlings) were placed in solutions containing either 1-^{14}C-leucine (Spec. Act. 54.7 mCi/mmole, 0.1 μCi/ml) for protein synthesis or methyl-^{3}H-thymidine (Spec. Act. 20 Ci/mmole, 1 μCi/ml) for DNA synthesis, for periods of time up to 1 hour. The primary roots of the seedlings were then excised and the nucleic acids and protein extracted according to Smillie and Krotkov (1960). Radioactivity in the final fractions was determined by liquid scintillation counting.

Chromosome Staining. For determinations of mitotic indices, roots of peas or *Vicia faba* (broad beans) were treated with DPX-4189 and fixed in ethanol:acetic acid (3:1) overnight at 4°C. The tips were excised, stained with Feulgens reagent and then squashed and mounted according to Conger and Fairchild (1953).

RESULTS

Effects on Plant Growth. One of the most pronounced effects of DPX-4189 on susceptible plants is growth inhibition (Figure 1). Net increases in the fresh weight of roots and shoots of corn seedlings grown in solution culture with various concentrations of DPX-4189 for 48 hours were significantly reduced. Growth inhibition was apparent at the lowest concentration tested, 2.8×10^{-9} M (1 ppb). These results also indicate that DPX-4189 is translocated since shoot injury was observed even though only the roots came in contact with the herbicide. Other experiments have shown that application of as little as 20 ng of DPX-4189 to the shoot tip of a soybean seedling will completely stop shoot growth.

In order to obtain a more detailed analysis of DPX-4189 inhibition of plant growth, continuous recordings of corn growth were made using a linear variable differential transformer (Hsiao, et.al., 1970). Growth rates of untreated corn generally ranged between 30-40 μm/min (Figure 2). A foliar application of 2.8×10^{-5} M DPX-4189 (10 ppm) rapidly reduced this rate. Within six hours of treatment, growth was only 50% of the control. Wheat, a species resistant to

DPX-4189, was unaffected by this concentration of DPX-4189. These results indicate that any physiological process proposed as involved in the primary mode of action of DPX-4189 must be affected within this period.

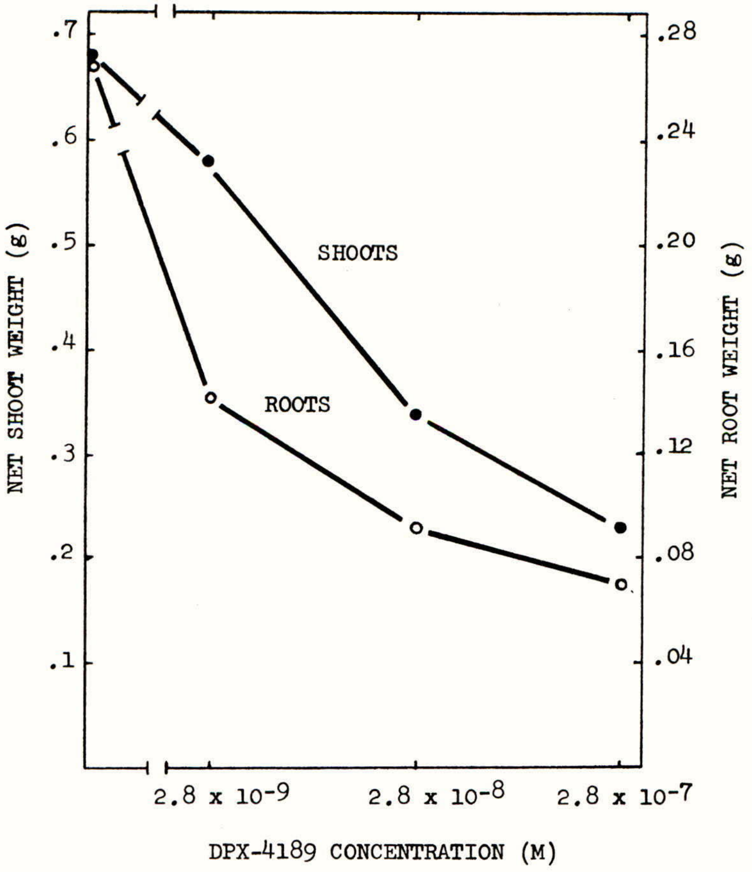

Figure 1 - Effect of DPX-4189 on growth of corn seedlings in solution culture. Plants were grown for 48 hrs in solutions containing various concentrations of DPX-4189. Values are net increases over initial weight.

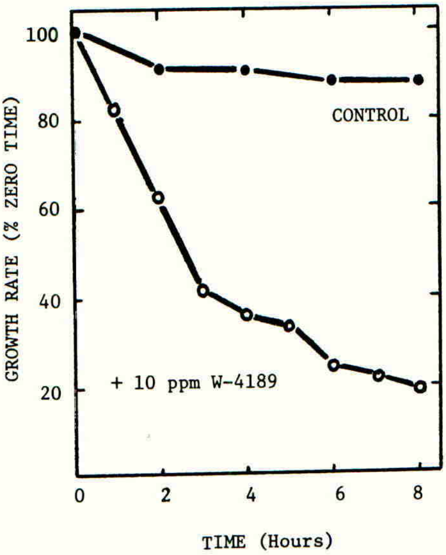

Figure 2 - Continuous growth measurements of corn seedlings as made with a linear variable differential transformer. Zero time rates are 32.4 µm/min for the control and 37.7 µm/min for the treated plants.

Effect on Photosynthesis and Respiration. No direct effects of DPX-4189 were observed on photosynthesis at herbicidal rates that completely inhibited growth.

Photosynthetic electron transport was determined in isolated chloroplasts by measuring either ferricyanide dependent O_2 evolution or the methyl viologen catalyzed Mehler reaction using an oxygen electrode. The rates of O_2 evolution or uptake ranged between 30-40 µmoles O_2/mg Chl-hr in coupled chloroplasts and between 130-180 µmoles O_2/mg Chl-hr in uncoupled chloroplasts. The presence of DPX-4189 concentrations up to 2.8×10^{-4} M had no effect even when chloroplasts were preincubated for 2 hours in the presence of these high levels of the herbicide. Other measurements of photosynthesis including $^{14}CO_2$ incorporation into leaves of treated plants as well as analysis of leaf fluorescence induction curves consistently showed that DPX-4189 does not affect photosynthesis.

Respiration also was not initially affected by DPX-4189. Rates of O_2 uptake by pea roots treated with 2.8×10^{-5} M DPX-4189 (10 ppm) for as long as 48 hours were identical to control rates.

Cell Elongation and Cell Division. Whether the growth inhibition of DPX-4189 was due to an inhibition of cell elongation, cell division, or both was assessed.

Several bioassays involving plant hormones were used to evaluate the effects of DPX-4189 on cell elongation. Neither indoleacetic acid induced elongation of sub-apical sections of etiolated pea stems, cytokinin induced cell expansion in cucumber cotyledons, nor gibberellic acid induced elongation of lettuce hypocotyls were affected by treatments of up to 2.8×10^{-5} M DPX-4189.

Although the elongation component of plant cell growth was found to be unaffected by DPX-4189, the cell division component of growth was extremely sensitive. The mitotic index of *Vicia faba* roots and pea roots was found to be greatly reduced after treatment with DPX-4189 (Table 1), from 6.4 division figures per 100 cells to 0.9. Analysis of the frequency of the various mitotic stages indicate there is no change in the frequency of any one particular mitotic phase (Table 1). This suggests that DPX-4189 acts not by blocking active division but by interfering with some necessary step prior to prophase.

Table 1

The Effect of DPX-4189 on the Mitotic Index and Frequency of Mitotic Stages in Vicia faba Root Tips

Treatment	Avg. No. of Mitotic Figs per 100 Cells	% Distribution Prophase	 Metaphase	 Anaphase	 Telophase
Control	6.4	46	21	19	15
+2.8×10^{-6} M DPX-4189	0.9	51	20	15	14

Another method for determining cell division activity involves measuring the amount of radioactive thymidine incorporated into the DNA of a cell. This widely used technique provides information on the amount of DNA being synthesized in cells, which in turn is a function of the amount of cell division occurring. DPX-4189 was found to be a very potent inhibitor of cell division as measured by ^{3}H-thymidine incorporation. Inhibition of cell division in corn root tips could be detected within 1-2 hours after treatment with 2.8×10^{-6} M DPX-4189 (Figure 3). After 6 hours, the amount of thymidine incorporated into DNA of treated roots is usually inhibited by 80-90%. At the same time, total thymidine uptake is largely unaf-

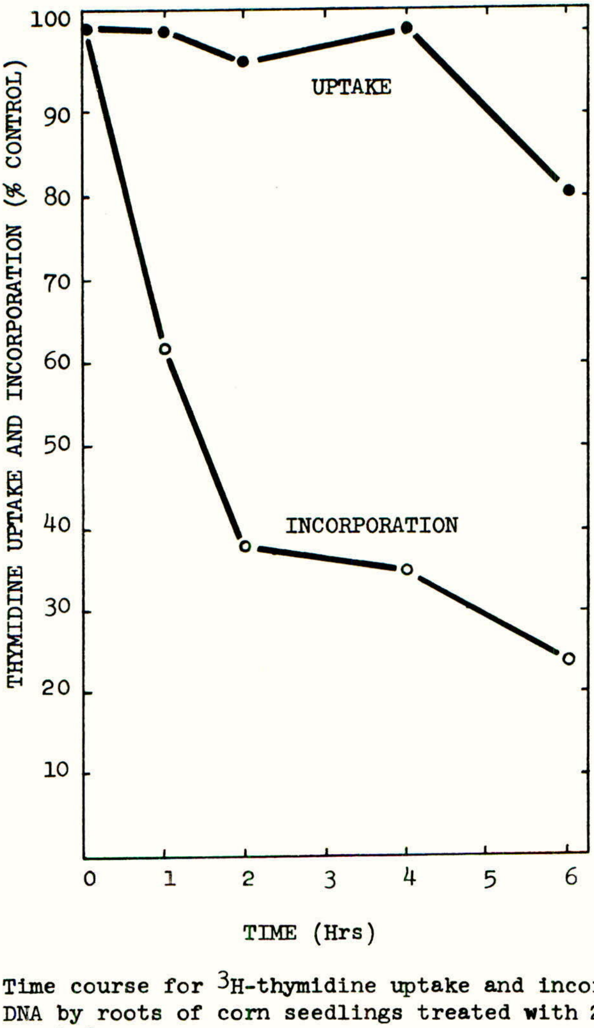

Figure 3 - Time course for ^{3}H-thymidine uptake and incorporation into DNA by roots of corn seedlings treated with 2.8×10^{-6} M DPX-4189

fected (Figure 3). Thus, this inhibition is not simply the result of reduced thymidine uptake. Similar responses have been found for pea roots and sterile cultures of tuber explants of Helianthus tuberosus (Jerusalem artichoke).

The cell division process appears to be quite sensitive to the inhibitory effects of DPX-4189. Treatment of corn roots for 4 hours with as little as 2.8×10^{-8} M DPX-4189 results in a 50% inhibition. Tuber explants grown in sterile culture are even more sensitive with inhibition being observed at 2.8×10^{-9} M DPX-4189.

The inhibition of cell division by DPX-4189 is a very specific effect. Under conditions where cell division in corn roots is inhibited 80-90% (2.8×10^{-6} M, 6 hr), protein and RNA synthesis are not significantly affected (Table 2). Specificity also extends to comparisons with other herbicides. At concentrations equivalent to a 1 ppm solution of DPX-4189 (2.8×10^{-6} M), neither diuron, metribuzin, hexazinone, trifluralin, propham, nor diallate were effective inhibitors of cell division as measured by the ^{3}H-thymidine incorporation technique.

Table 2

The Effect of DPX-4189 on RNA and Protein Synthesis in Corn Root Tips

Seedlings were Treated for 6 Hours with 2.8×10^{-6} M DPX-4189

	Precursor Incorporation Dpm/g fr wt		
	Control	+ DPX-4189	% Control
RNA (^{3}H-Uridine)	7.39×10^5	6.01×10^5	81
Protein (^{14}C-Leucine)	1.02×10^7	1.14×10^7	112

DISCUSSION

The principal mode of action of DPX-4189 appears to be growth inhibition. Consistent with field studies showing extremely potent herbicidal activity (Levitt, et.al., 1980), laboratory experiments have shown that DPX-4189 is a very active inhibitor of plant growth at concentrations as low as 2.8×10^{-9} M. At levels where DPX-4189 inhibits plant growth, major physiological processes such as photosynthesis, respiration and protein synthesis are not initially affected. Likewise, cell elongation, mediated by plant hormones, is not especially sensitive towards inhibition by this herbicide. Rather, DPX-4189 appears to act by inhibiting plant cell division. Inhibition is rapid, occurring within 1-2 hours of treatment, and can be caused by as little as 2.8×10^{-9} M DPX-4189 in sensitive species.

The possible effects of DPX-4189 on the metabolic and physiological processes described in this report were all studied at relatively short treatment times. It can be assumed that with longer treatment times, secondary effects would also become apparent as plant vigor declines. Concentrations of DPX-4189 used in this study were quite low, between 2.8 x 10^{-5} and 2.8 x 10^{-9} M, not only because adequate responses were detectable at these levels, but also since use of higher concentrations could induce secondary effects unrelated to the primary action of this important new herbicide.

Collectively, these results indicate that the primary target site of DPX-4189 is on plant cell division. The exact biochemical lesion responsible for this inhibition, however, is not yet known.

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POTENTIAL FOR EXTENDING THE SELECTIVITY OF DPX 4189 BY USE OF HERBICIDE SAFENERS

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Summary Pot experiments have shown that the tolerance of maize to DPX 4189 (2-chloro-N-[(4-methoxy-6-methyl)(1,3,5-triazine-2yl)amino carbonyl]benzene sulphonamide) can be greatly increased by seed dressing with the herbicide safener 1,8-naphthalic anhydride (NA) and to a lesser degree by R 25788 (N,N-diallyl-2,2-dichloroacetamide). *Rottboellia exaltata*, which is difficult to control in maize, could perhaps be selectively controlled at 10-20 g DPX 4189/ha. The safening effect of NA was also demonstrated on sorghum and rice and it could contribute to the control of wild or red rice in rice at about 5 g DPX 4189/ha. Wheat and barley were also well protected by NA but only to a small degree by R 25788. Although these crops are tolerant of relatively high doses of DPX 4189, safener could allow use of doses in excess of 100 g/ha for control of difficult weeds such as *Bromus* spp. Seed dressings with NA failed to protect oil seed rape, sugar beet, perennial ryegrass, dwarf bean or onion against DPX 4189.

INTRODUCTION

Herbicide safeners ("protectants" or "antidotes") have been the subject of previous research at WRO and routine herbicide evaluation experiments now include maize with and without a seed dressing of 1,8-naphthalic anhydride (NA) at 0.5 or 1% and sorghum with and without seed treatment with cyometrinil. One of these tests indicated that both crops could be protected against DPX 4189 by safeners (Richardson *et al*, 1980). Results of further experiments are reported below.

METHODS AND MATERIALS

All experiments were conducted in the glasshouses at WRO, using a sandy loam soil supplemented with NPK and trace elements. Plastic pots of 10 cm diameter were used in all experiments except 1 and 2 where "long tom" pots (11 cm diameter and 12 cm high) were used. All treatments were replicated three times.

Seed dressings were applied by shaking the weighed amounts of safener and seed in glass tubes or polyethylene bags. The amount of safener retained by the seeds was not measured after shaking but was inevitably somewhat less than the amount weighed out.

Herbicides were applied by a laboratory sprayer comprising a single Teejet band-spray nozzle travelling at 0.71 kph and delivering 437 l/ha (370 l/ha in experiments 4 to 6) at an operating pressure of 207 kPa. Subsequent watering was applied from overhead to the soil surface as required.

Details for individual experiments are given in Table 1.

In experiment 1 the herbicide for half the pots was sprayed onto tins of soil 6 cm deep and mixed thoroughly before being used to make the upper 6 cm in the pots ("incorporated") and for the other half, was sprayed onto the soil surface following sowing.

Assessment was mainly by measurement of fresh and dry weights of shoots and dry weight of roots 4-6 weeks after sowing. Results were subjected to analysis of variance.

Table 1

Experiment details

Exp. No.	Species	Varieties	No. of seeds/ pot	Sowing depth (cm)	Planting/ spray date	Harvest date	Temp. (°C)
1	Maize	Julia	5	2.0	26.3.80	28.4.80 to 1.5.80	16-30
	Sorghum	G 623	8	2.0			
	Rice	IR 298	10	2.0			
	Rottboellia exaltata		15	2.0			
2	Maize	Julia	4	2.0	15.5.80	9-11.6.80	18-30
	Rottboellia exaltata		15	2.0			
3	Maize	see text	4	2.0	19.6.80	11-18.7.80	17-30
	Rice	" "	10	2.0			
	Rottboellia exaltata		15	2.0			
4	Wheat	Maris Huntsman	8	1.0	11.4.80	13.5.80	4-28
	Barley	Maris Mink	8	1.0			
5	Wheat	Maris Huntsman	8	1.0	4.7.80	29.7.80	10-32
	Maize	Julia	4	2.0			
6	Wheat	see text	8	1.0	10.7.80	30.7.80	10-32
	Barley	" "	8	1.0			

RESULTS

Experiment 1 Figure 1 confirms the four to eight-fold protection of maize by NA at 0.5%. Toxicity due to DPX 4189 alone was greater than in the earlier work (Richardson et al, 1980) and 20 g/ha even with NA was damaging, especially to root growth. Rottboellia exaltata, a weed of maize resistant to the standard triazine and amide herbicides, was well suppressed by 20 g/ha but not by 10 g/ha. Some plants were severely damaged at the lower dose but many recovered well. Pre-planting incorporated treatments caused slightly greater damage to both maize and R. exaltata, than corresponding pre-emergence sprays. The safener was equally effective with a protection factor exceeding eight-fold. Sorghum and rice also received four to eight-fold protection by NA at 0.5% but were more sensitive than maize, being damaged by 5 g/ha (Fig. 2).

FIG. 1

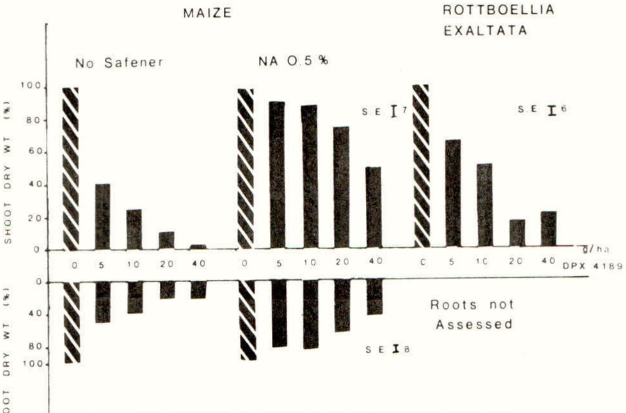

FIG. 1 DRY WEIGHTS OF MAIZE AND ROTTBOELLIA EXALTATA FOLLOWING PRE-EMERGENCE TREATMENT WITH DPX 4189 (EXPERIMENT 1)

FIG. 2

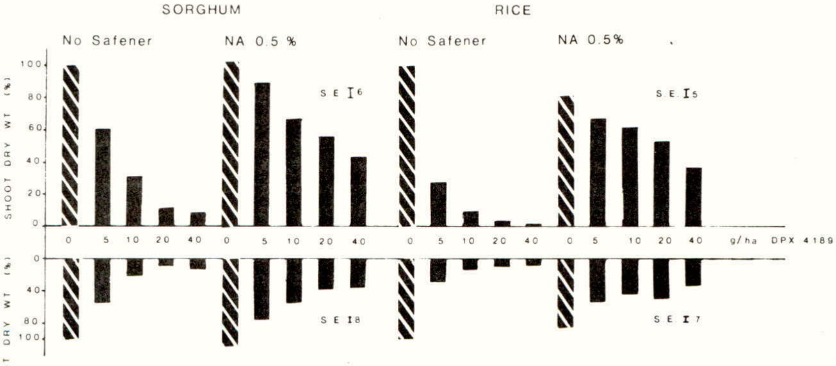

FIG 2 DRY WEIGHTS OF SORGHUM AND RICE FOLLOWING PRE-EMERGENCE TREATMENT WITH DPX 4189 (EXPERIMENT 1)

Experiment 2 Still higher activity of DPX 4189 was demonstrated in this experiment, perhaps due to higher temperatures but protection of maize by NA at 0.5% was at least four-fold. Maize with protection showed little growth reduction with 5 or 10 g/ha DPX 4189. R. exaltata (Fig. 3) was relatively susceptible at these concentrations. R 25788 as either a seed dressing at 0.5% or a pre-emergence spray at 1 kg/ha gave approximately four-fold protection.

Post-emergence treatments applied one week after sowing, when plants were 8-12 cm high, were also "safened" by NA 0.5% as a seed dressing although at this stage R. exaltata was more tolerant so selective control was not achieved by DPX 4189 up to 20 g/ha (results not presented). No surfactant was added and spray retention was very low.

FIG. 3

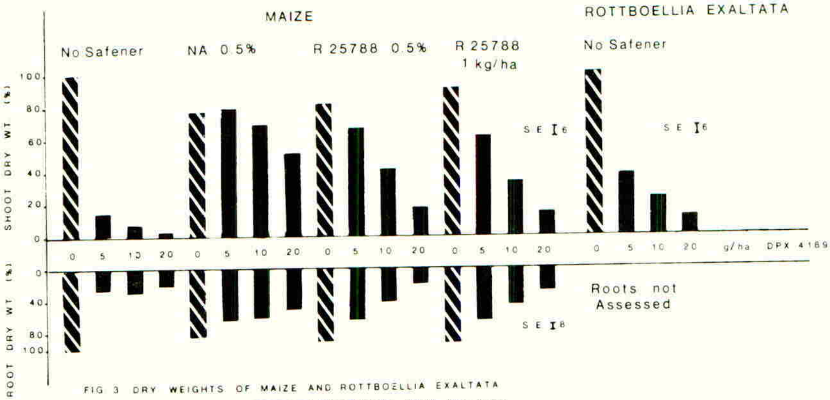

FIG 3 DRY WEIGHTS OF MAIZE AND ROTTBOELLIA EXALTATA FOLLOWING PRE-EMERGENCE TREATMENT WITH DPX 4189 (EXPERIMENT 2)

Experiment 3 Maize var Julia was no better protected against higher doses of DPX 4189 by 1% than by 0.5% NA. Protection by NA as a seed dressing against post-emergence treatments was good but even with surfactant, control of R. exaltata was inadequate at 20 g/ha (Fig. 4). Maize var. Caldera was also well protected by NA 0.5% seed dressing but the South African hybrid SA 52 and the var. Western Yellow from Nigeria were less well protected and showed some acute chlorotic symptoms from the NA dressing alone as well as the usual mild retardation.

Rice var IR 298 was well protected by NA. There were no symptoms with DPX 4189 other than a retardation in growth (Fig. 5). Unprotected, the rice varieties IR 298 and Blue Bonnett and the red rice were all severely suppressed by 5 and 10 g/ha although the West African wild rice O. barthii was more resistant. DPX 4189 affected two of the rice cultivars differently. IR 298 showed no chlorosis even when severely stunted while Blue Bonnett showed considerable chlorosis not completely prevented by NA. Protection of this latter variety was erratic, perhaps because of uneven retention of NA on the seeds.

FIG. 4

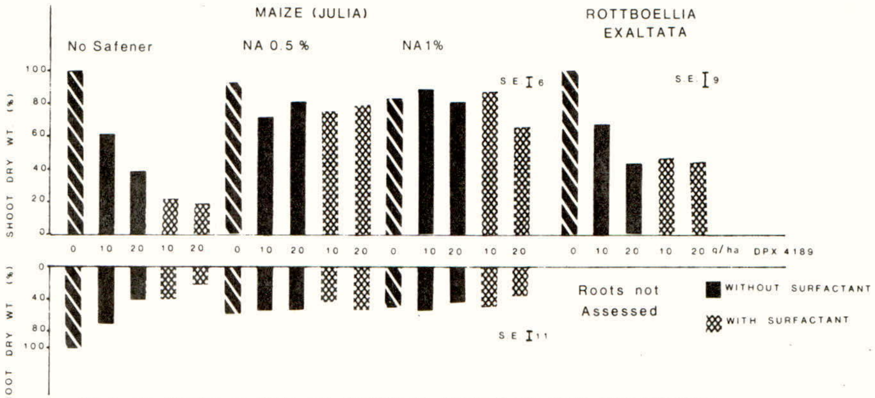

FIG 4 DRY WEIGHTS OF MAIZE AND ROTTBOELLIA EXALTATA FOLLOWING POST-EMERGENCE SPRAYS OF DPX 4189 WITH OR WITHOUT SURFACTANT, ONE WEEK AFTER SOWING. (EXPERIMENT 3)

FIG. 5

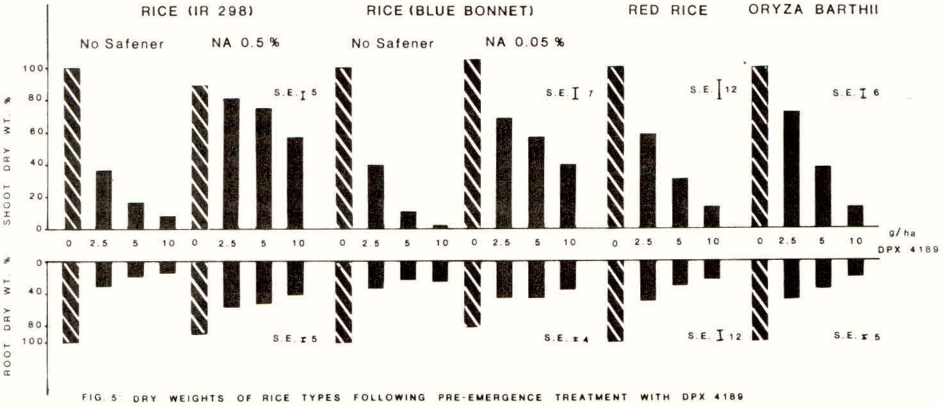

FIG. 5 DRY WEIGHTS OF RICE TYPES FOLLOWING PRE-EMERGENCE TREATMENT WITH DPX 4189 (EXPERIMENT 3)

Experiment 4 Figure 6 shows that wheat and barley were well protected from DPX 4189 by NA but R 25788 was much less effective as a safener particularly on wheat. In this same experiment, rape, sugar beet, onion, dwarf bean and perennial ryegrass were sensitive to DPX 4189 at doses ranging from 5 to 20 g/ha and neither NA nor R 25788 afforded any protection.

FIG. 6

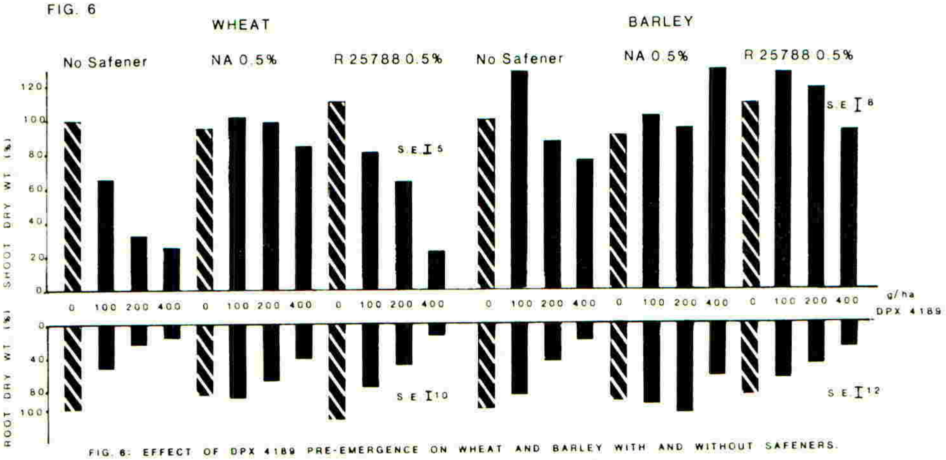

FIG. 6: EFFECT OF DPX 4189 PRE-EMERGENCE ON WHEAT AND BARLEY WITH AND WITHOUT SAFENERS. (EXPERIMENT 4)

Experiment 5 Here the degree of injury to maize and wheat caused by DPX 4189 was reduced at dressings of NA below those used in the earlier experiments; 0.125% was as effective as 0.5% on both maize and wheat although the degree of protection was better for maize (herbicide doses 10 and 20 g/ha) than for wheat (150 and 300 g/ha). Some inconsistencies were apparent with the protection being better at the higher rather than the lower dose of DPX 4189.

Experiment 6 In addition to 'Maris Huntsman' wheat and 'Maris Mink' barley (Exp. 4) some protection of other varieties of both species (Sicco, Maris Dove and Mardler wheats, Triumph, Athene and Sonja barleys) was gained by the use of NA 0.5% seed dressings in conjunction with DPX 4189 at 100 and 200 g/ha. Some effect of NA alone was apparent on some varieties, usually as an overall yellowing or chlorosis of leaves, although two varieties, 'Mardler' wheat and 'Athene' barley also suffered a reduction of dry weight in both shoots and roots. Both were protected to some extent from DPX 4189 injury by NA, but less than the other varieties tested. The other three wheat varieties when protected by NA were unaffected by DPX 4189 at 100 g/ha. 'Triumph' when protected was the only barley variety to withstand fully this dose of the herbicide. Here 'Maris Mink' showed much less protection than in experiment 5 and much more sensitivity to DPX 4189 alone.

DISCUSSION

The use of herbicide safeners has been under development for approximately 10 years since the introduction of 1,8-naphthalic anhydride by the Gulf Co in USA for protection of maize against EPTC (Hoffman, 1969). Only two further safeners have been introduced commercially; R 25788 by Stauffer Chemical Co and cyometrinil by Ciba-Geigy Agrochemicals and both the range of crops protected and the range of herbicides against which protection is possible are still very limited. The only commercial uses of safeners are NA or R 25788 to protect maize from EPTC and other thiolcarbamate herbicides and cyometrinil to protect sorghum from metolachlor and alachlor. Experimentally, sorghum and rice have been well protected by NA against alachlor. NA makes possible selective control of a wild rice (Oryza punctata) by alachlor in rice, (Parker and Dean, 1976) a treatment given limited commercial use in Swaziland. Blair (1979) was less successful in protecting wheat from tri-allate and field bean (Vicia faba) from EPTC with NA, although Blair and Dean (1976) showed that NA could be used effectively to protect maize against perfluidone. This permitted selective control of R. exaltata which has increased seriously in North and South America, West and South Africa and the Philippines in both maize and sugar cane. Blair and Dean's perfluidone work was not followed up because an alternative selective herbicide treatment, pendimethalin, was discovered (Rhodesia, 1975). However, although pendimethalin has given adequate control under good soil moisture conditions it tends to fail under drier conditions and an alternative is, therefore, still needed.

The results reported in this paper do not demonstrate a clear cut selectivity against R. exaltata but further work would appear justified. Although somewhat retarded at 20 g DPX 4189/ha, maize showed no distinct herbicide symptoms and full recovery appeared quite probable in the varieties Julia and Caldera 535. The approximately eight-fold protection approached that provided in the commercially used safener combinations (Blair et al, 1976), while the seed treatment with NA protects the maize against post-emergence treatment for at least one week after sowing.

The protection of sorghum by NA from DPX 4189 was more striking than that by cyometrinil but the practical value of the four-fold protection achieved is doubtful in view of the availability of cyometrinil for use in conjunction with metolachlor.

In rice (IR 298) only lower doses of about 5 g/ha seem likely to be tolerated even with the use of NA. These doses did not provide control of the wild rice species O. barthii, but were more active on the North American red rice type which is more closely related to the crop, and further testing would appear worthwhile. As with maize it seemed quite probable that longer term experiments could show recovery from the early retardation. It will, however, be necessary to look further at varietal differences in susceptibility.

Although the protection from DPX 4189 damage afforded by NA to wheat and barley is somewhat academic in view of the relatively high tolerance of these species, nevertheless the fact that such protection is possible is of interest. DPX 4189 is the forerunner of a new chemical group or family of very active herbicides, the sulphonyl ureas. If protection from crop damage can be attained by safeners, provided that such a combination of treatments is economical, the benefits to weed control could be considerable, for example for affording a means to combat the more serious weed problems such as Bromus spp and volunteer cereal species. The results of experiment 5 suggest that further work at lower seed dressing concentrations of NA may be worthwhile. More work is required regarding the actual amounts retained by the seeds and the relative importance of seed size. Also the time for which the protective effect of NA operates needs investigation, a preliminary experiment suggested this had virtually disappeared by the time the plants had reached the 3-leaf stage.

Acknowledgements

We wish to acknowledge the technical assistance provided by Miss Della Stringer and Mr Graham White and the Statistics Section for analysis of the data. We also thank Du Pont, Stauffer Chemicals and Gulf Co for herbicide and safener samples and Ciba-Geigy for the provision of sorghum seed treated with cyometrinil. The work on tropical species was financed by HM Overseas Development Administration.

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IMPROVED HERBICIDAL PERFORMANCE OF DPX 4189 ON OIL-SEED RAPE BY THE ADDITION OF SURFACTANTS

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Summary The addition of certain non-ionic surfactants to solutions of 2-chloro-N-[(4-methoxy-6-methyl-1,3,5-triazine-2-yl)amino-carbonyl]-benzenesulphonamide (DPX 4189 as 80% wettable powder) increased its herbicidal effects on oil-seed rape seedlings (*Brassica napus*). DPX 4189 solutions with and without surfactant (0.5% of total volume) were applied at 345 l/ha (5 g/ha a.i.). All the surfactants enhanced DPX 4189 activity, but in the order, Agral (most effective), Citowett plus, Renex-36 and Atplus 300F (least effective). In a separate experiment a series of Ethylan surfactants with differing hydrophile-lipophile balance values also increased activity when added to DPX 4189 spray solutions.

A correlation between increased herbicidal activity and greater spray retention was demonstrated, and this finding is discussed as a possible explanation of the improved effectiveness of the herbicide in the presence of additional surfactants.

INTRODUCTION

Extensive tests in Europe and N. America since 1979 have shown DPX 4189 to be a most promising herbicide for the control of broad-leaved weeds in cereals (Bingeman, 1979; Jensen, 1980). The high biological activity of this compound and the possibility of persistence as a soil residue particularly in areas of low rainfall and low winter temperatures suggest its use at very low doses. For this and for economic reasons the incorporation in spray solutions of DPX 4189 (80% wettable powder formulation) of additional surfactant as a means of increasing herbicidal effectiveness at low rates (see Jensen, 1980) has been investigated.

Using oil-seed rape seedlings as a test plant, this work evaluates the effectiveness of different non-ionic surfactants in increasing herbicide activity and spray retention on leaf surfaces. Species differences were investigated in an experiment with four common weeds.

METHOD AND MATERIALS

Pot experiments to assess herbicidal activity

Seeds of oil-seed rape (*Brassica napus*, var. Nene Jet) were sown in pots containing 550 g of sandy loam (10% clay, 10% silt, 60% coarse sand, 20% fine sand, 2-3% organic matter; pH 7.2) with added fertilizer (ammonium sulphate, 1 g;

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magnesium sulphate, 1 g; superphosphate of lime, 3 g; potassium sulphate, 1 g; to each kg of loam). The seedlings were thinned to four plants per pot and grown to the two leaf stage in a glasshouse before treatment.

DPX 4189 spray 'solutions' were made from 80% wettable powder and surfactants were added as indicated (see Table 1). As the treatments with DPX 4189 used low rates (5-20 g/ha), the concentration of the active ingredient (approximately 15-60 mg/l) was well within the water solubility of the compound (125 mg/l at 25°C). It is therefore probable that the herbicide, although prepared from a wettable powder formulation, was applied as a 'true solution'. The solutions were applied at 345 l/ha through a Spraying System 'Tee-jet' 8002 at a pressure of 2.04 bar. 4 to 6 replicates were used for each treatment in the experiments described. Pots were arranged in a randomized block design in a glasshouse. Watering was carried out at soil level to avoid wetting the foliage. Two weeks after treatment, the four plants from each pot were cut at soil level and fresh weights recorded.

All experiments were repeated at least once and typical results are reported.

Assessment of spray retention

Oil-seed rape seedlings were grown to the 2 leaf stage as described above. Fluorescein (0.1% w/v as sodium salt) was added to DPX 4189 solutions (5 g a.i./ha) in the presence or absence of surfactants. Eight replicate pots (32 seedlings) received each treatment. After 30 minutes, the plants from each pot were cut at soil level and washed for 1 minute in 50 ml of sodium hydroxide solution (0.005 M). The fluorescein concentration in the 'washings' was estimated by measuring the fluorescence and comparing with a standard curve. Results are expressed as the total volume of spray retained on the leaves of the four seedlings in each pot (in ul) and this value was corrected to take into account variations in plant size (µl/g dry wt.).

Table 1

Non-ionic surfactants added to DPX 4189 formulated spray solution

Trade name	Active ingredient	† Hydrophile-lipophile balance
Agral	90% alkyl phenol ethylene oxide condensate	12.0
Atplus 300F	80% polyoxylethylene sorbitan fatty acid ester	-
Citowett plus	50% alkylaryl polyglycol ether	11.0
Renex-36	Polyoxyethylene(6)tridecyl ether	11.4
Ethylans	Nonyl phenol ethylene oxide condensates:-	
	N 5.5*	10.5
	N 8*	12.2
	N 12*	14.0
	N 20*	16.0
	N 35*	17.4

* Average number of moles of ethylene oxide per mole of nonyl phenol.
† Manufacturers' data.

RESULTS AND DISCUSSION

The effect of added surfactants on herbicidal activity

DPX 4189 was effective at a rate of 5 g/ha, reducing the final fresh weight of the oil-seed rape plants to 14.2% of unsprayed controls (Table 2). The addition of each of four non-ionic surfactants to sprays of DPX 4189 enhanced performance. The greatest improvement occurred with Agral, treated plants were only 3.9% of unsprayed controls. In a separate experiment the effect of the four surfactants with higher levels of DPX 4189 (10, 15 and 30 g/ha) gave similar results. However, the toxicity of the herbicide alone at the highest dose rate (30 g/ha) was so severe that little increase on the addition of surfactant was possible. When 30 and 50% of corn-oil was included with Agral, and this mixture compared with Agral alone as an additive to DPX 4189, no further improvement was indicated.

Table 2.

Effect of added surfactants on DPX 4189 herbicidal activity when sprayed at 5 g/ha on oil-seed rape

Treatment	Fresh wt. g/pot	% of control
Control (unsprayed)	25.40	-
DPX 4189, alone	3.61	14.2
DPX 4189 + Agral 0.5%	0.99	3.9
DPX 4189 + Atplus 300F, 0.5%	1.68	6.6
DPX 4189 + Citowett plus, 0.5%	1.16	4.6
DPX 4189 + Renex-36, 0.5%	1.30	5.1
LSD (0.05)	0.61	

The effect of concentration of Agral on the herbicidal activity of DPX 4189

Six concentrations of Agral from 0.025 to 1% of the total spray volume were added to DPX 4189 'solution' and sprayed at a rate of 5 g/ha on oil-seed rape plants. The fresh weights of the plants two weeks later are shown in Table 3. The addition of Agral improved the performance of the formulated DPX 4189 and with relatively small amounts of surfactant. A twenty-fold increase in rate, from 0.025% to 0.5%, produced relatively little further improvement.

Table 3.

Effect of differing concentrations of Agral on the activity of DPX 4189 (5 g/ha) on oil-seed rape seedlings

Treatment	Fresh wt. g/pot	% of control
Control (demineralized water)	27.86	-
DPX 4189	10.67	38.3
DPX 4189 + Agral 0.025%	8.16	29.3
DPX 4189 + Agral 0.05%	8.10	29.1
DPX 4189 + Agral 0.1%	8.62	30.9
DPX 4189 + Agral 0.25%	7.59	27.2
DPX 4189 + Agral 0.5%	7.54	27.1
DPX 4189 + Agral 1.0%	6.42	23.0
LSD (0.05)	2.02	

Effect of Ethylan surfactants with differing hydrophile-lipophile balances on the herbicidal activity of DPX 4189

By varying the ratio of ethylene oxide to nonylphenol molecules in the Ethylan surfactants, a series of products is obtained with a range of hydrophile-lipophile balance values (HLB, see Table 1). Five of these surfactants were added, at 0.25%, to a DPX 4189 spray applied at 5 g/ha to oil-seed rape seedlings. The fresh weights of the seedlings when harvested two weeks later are shown in Table 4.

Table 4.

The effect of a series of Ethylan surfactants at 0.5% on the herbicidal activity of DPX 4189 (5 g/ha) using oil-seed rape as test plant

Treatment	Fresh wt. g/pot	% of control
Control (unsprayed)	21.4	-
DPX 4189, alone	9.2	43.0
DPX 4189 + Ethylan N 5.5	5.1	23.8
DPX 4189 + Ethylan N 8	4.5	21.0
DPX 4189 + Ethylan N 12	4.8	22.4
DPX 4189 + Ethylan N 20	4.4	20.6
DPX 4189 + Ethylan N 35	7.1	33.2
LSD (0.05)	1.5	

As with the other surfactants tested, the Ethylans improved the activity of DPX 4189. They were not equally effective, those with intermediate HLB values being marginally better than Ethylan N 35 which had the highest HLB value (Table 4). Diminished effectiveness by compounds at the extremities of a range has previously been commented upon (Foy and Smith, 1969). Ethylan N 8, a surfactant widely used in agriculture, was amongst the most effective.

The effect of added surfactants on the retention of DPX 4189 sprays

To establish the part played by changed spray retention on the increased herbicidal activity of sprays incorporating additional surfactants, the data in Table 5 were obtained. DPX 4189 spray solution (5 g/ha) was used with Agral, Atplus 300F, Citowett plus or Renex 36 (0.25%) and fluorescein (0.1%) for application at 345 l/ha to oil-seed rape seedlings. Spray retention, assessed by measurement of fluorescein remaining on the leaf surfaces, generally agreed with herbicidal activity, indicated by reduction in fresh weight. Agral, the most effective additive for increased DPX toxicity (Table 2), gave the highest spray retention (52% more than the DPX 4189 alone). Similarly, with Citowett plus and Renex-36, higher retention was associated with greater damage. Atplus 300F sprayed with the herbicide produced greater damage than the DPX 4189 alone(Table 2) although there was no increase in spray retention (Table 5). Although these results relate to herbicidal activity with a higher concentration of surfactants than that used in the retention studies, spray retention is clearly a major effect of surfactants, but probably not the only one. Other processes such as increased uptake through cuticular damage could also be involved.

Table 5

Effect of surfactants on the retention of fluorescein in the solution of DPX 4189 (5 g/ha) when sprayed on to leaves of oil-seed rape seedlings

Treatment	Spray deposition (ul/g dry wt.)	% of "no surfactant"
DPX 4189 + fluorescein, 0.1%	31.8	-
DPX 4189 + Agral, 0.25% + fluorescein, 0.1%	48.4	152.2
DPX 4189 + Atplus-300F, 0.25% + fluorescein, 0.1%	29.3	92.1
DPX 4189 + Citowett plus, 0.25% + fluorescein, 0.1%	40.0	125.8
DPX 4189 + Renex-36, 0.25% + fluorescein, 0.1%	35.4	111.3
LSD (0.05)	2.79	

Response of seedlings of other species to DPX 4189 and the effect of added Agral

Using similar cultural methods to those described for oil-seed rape, seedlings of chickweed (Stellaria media), cleavers (Galium aparine), mayweed (Tripleurospermum maritimum) and spurry (Spergula arvensis) were grown and sprayed with DPX 4189 at 10 g, 15 g and 20 g/ha. Agral (0.5%) was added to the herbicide solution, and 2,4-D, as its amine salt (0.5 kg/ha) was included for comparison. Results (Table 6) suggest that spurry is well controlled by DPX 4189 with or without the addition of Agral and by 2,4-D. The other species were less well controlled by 2,4-D than by DPX 4189. With spurry higher doses were unable to increase the already severe herbicidal effects, and similarly with chickweed and mayweed the addition of surfactant did not increase toxicity. Although the control of cleavers by DPX 4189 was good (20% of control at 10 g/ha), the inclusion of Agral improved its performance (9.5% of control with Agral, 0.5% v/v: Table 6). In field experiments Jensen (1980) also showed differences between weed species in their response to DPX 4189 with added Citowett plus. For example, chickweed control was only slightly improved by the addition of Citowett plus. Here, in the greenhouse tests, the inclusion of Agral was even less effective with this weed species.

Table 6

Response of other weed species to DPX 4189 with Agral and also to 2,4-D (amine salt)

Treatment	Fresh weight (g/pot) Chickweed	Cleavers	Mayweed	Spurry
Control (unsprayed)	20.3	9.4	7.4	8.8
DPX 4189 10 g/ha	5.8	1.9	0.8	0.5
15 g/ha	5.2	1.4	0.9	0.3
20 g/ha	5.8	1.9	0.9	0.4
DPX 4189 10 g/ha + Agral	4.8	0.9	0.8	0.2
15 g/ha + Agral	5.1	1.0	0.9	0.3
20 g/ha + Agral	5.0	0.8	1.0	0.2
2,4-D (Amine salt) 0.5 kg/ha	9.6	9.3	2.2	0.5
LSD (0.05)	1.3	1.0	0.6	1.1

Acknowledgements

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Proceedings 1980 British Crop Protection Conference - Weeds

FLUAZIFOP BUTYL - A NEW SELECTIVE HERBICIDE FOR THE CONTROL OF ANNUAL AND PERENNIAL GRASS WEEDS

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Summary

Fluazifop-butyl*, butyl 2-[4-(6-trifluoromethyl-2-pyridyloxy) phenoxy] propionate, formerly PP009, is a new post-emergence herbicide for the selective control of annual and perennial grass weeds primarily for use in broad leaf crops. Extensive field trials have shown it can be safely used in over sixty different crops to control the major grass weeds. Excellent and consistent post-emergence control of annual grass weeds has been achieved at rates of 0.125-0.5 kg ai/ha and perennial grass weeds at rates of 0.5-2.0 kg ai/ha.

Fluazifop-butyl has a low order of toxicity.

Resumé

Le fluazifop-butyl (Butyl 2-[4(5 trifluromethyl-2-pyridyloxy phenoxy] propionate), également dénommé PP009 est un nouvel herbicide de post-émergence pour le control des graminées annuelles et vivaces dans les cultrues de dicotylées. De nombreaux essais ont montré que le fluazifop-butyl peut être utilisé sur plus de soixante culture différentes, donnant un excellent contrôle des graminées en traitement de post-émergence. Les doses varient de 125 a 500 g m.a./ha sur graminées annuelles et de 500 g à 2000 g m.a./ha sur graminées vivaces;

Le fluazifop-butyl est un produit peu toxique.

INTRODUCTION

Fluazifop-butyl (code number PP009) is a new herbicide currently being developed by Plant Protection Division of ICI. Field tests in over seventy different countries throughout the world have confirmed fluazifop-butyl as a systemic herbicide, highly active against both annual and perennial grass weeds and selective to non-graminaceous crops. This paper gives general information on fluazifop-butyl and reports results from six field trials representative of the global programme carried out between 1978 and 1980. The trials covered a wide range of agricultural and climatic conditions and illustrate the principal herbicidal properties of fluazifop-butyl.

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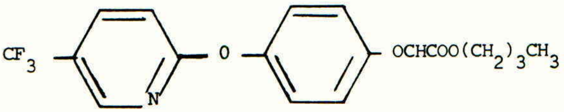
* Proposed common name

CHEMICAL AND PHYSICAL PROPERTIES

Structure



Chemical name : Butyl 2-[4-(5-trifluoromethyl-2-pyridyloxy) phenoxy] propionate

Proposed common name : fluazifop-butyl

Code numbers : PP009, TF1169 in Canada

Appearance : Light straw coloured odourless liquid

Melting point : Approximately 5°C

Boiling point : Approximately 170°C at 0.5mm hg.

Solubility : Water - 2ppm, propylene glycol 2%. Completely miscible in methanol, acetone cyclohexanone, hexane, xylene and methylene dichloride.

Vapour pressure : 5.5 x 10.5 pa at 20°C

Density/specific gravity : 1.21 cm - 3 at 20°C

Formulation : 25% e.c.

TOXICOLOGY

Results from acute and sub-acute toxicological studies indicate that fluazifop-butyl and its major metabolite, fluazifop are of a low order of toxicity. The acute oral LD_{50} values to the rat and mouse are approximately 3000 and 1500 mg/kg respectively and the 24 hour dermal LD_{50} to the rat and rabbit are in excess of 5000 mg/kg.

Fluazifop-butyl is slightly irritant to rat and rabbit skin and is a weak sensitiser of guinea pig skin.

Fluazifop-butyl is also of low toxicity to birds and invertebrates. The acute oral LD_{50} for mallard ducks is >17,000 mg/kg, the highest dose that could be administered. No effect was seen on bees when administered both

TABLE 1 - EXPERIMENTAL DETAILS

Country	SPAIN	BRAZIL	MALAYSIA	UK	USA	
Site	Valencia	Holambra Sao Paulo	Malacca	East Anglia	Yucatan Plantation Vichsburg	
Trial References	A	B	C	D	E	F
Crop	no crop	no crop	no crop	Sugar Beet	Cotton DPL61	no crop
Plot Size	5 x 1m	2 x 4m	2 x 3m	2 x 10m	3 x 15m	2m x 11m
Replicate Blocks	2	2	3	4	4	4
Sprayer	C02 pressurised	CO2 pressurised	Oxford Precision	CO2 pressurised	Tractor Compressed Air	CO2 pressurised
Nozzle	Allmans '00'	Tee Jet 8002 (0.5m spacing)	Allman '00'	Tee Jet 8002	Tee Jet 8003 (1m spacing)	Tee Jet 8003 (0.5m spacing)
Volume/litre/ha	250	250	400	200	260	230
Pressure (bar)	1.6	2.1	2.1	2.1	1.6	3.2
Assessment Method	Visual Assessment % kill Over total plant	Visual Assessment % kill Over total plant	Visual Assessment % kill Over total plant	Weed counts per $8m^2$ or $5x25cm^2$ quadrats	Visual Assessment % kill Over total plant	Visual Assessment % kill Over total plant

Agral 90 wetter was added to all PP009 treatments at a rate of 0.1% spray volume.

orally and by contact at 240 mg ai per bee and 120 mg ai per bee respectively. Earthworms were unaffected 1 and 6 months after application to field plots at 5 kg ai/ha. The EC_{50} to *Daphnia magna* was >10 mg ai per litre after 24 and 48 hours. Fluazifop-butyl is moderately toxic to fish; the LC_{50} at 96 hours for rainbow trout (*Salmo gairdneri*) is 1.6 ppm.

MODE OF ACTION

Fluazifop-butyl moves in both the xylem and the phloem, and appears to adversely interfere with ATP production. The first visible symptom, usually within 48 hours of application, is a cessation of growth. Meristematic tissue in the nodes and buds become necrotic, young leaves show chlorosis thrn necrosis. The oldest leaves show senescent pigment changes.

The selectivity of fluazifop-butyl is believed to be due to rapid degradation followed by conjugate formation in broad leaf plants.

METHODS AND MATERIALS

Experimental details of six trials selected from the extensive field programme carried out, to illustrate the principal herbicidal properties of fluazifop-butyl are listed in Table 1. In all trials a 25% ec formulation of fluazifop-butyl was used. A leaf wetting surfactant, 'Agral 90', is recommended for use with fluazifop-butyl and this was included in all PP009 treatments at a rate of 0.1% of the spray volume. Appropriate commercially available products were used as standard treatments in most trials; eg Alloxydim Na as a 75% w.p., Diclofop-methyl as a 36% e.c., Glyphosate as a 36% a.c.

RESULTS

Crop tolerance

A wide range of non-graminaceous crops showed tolerance to fluazifop-butyl at rates at least twice those required for effective grass weed control, Table 2.

TABLE 2

Crops shown to be tolerant to fluazifop-butyl by post-emergence sprays

Apples
Apricot
Bananas
Blackcurrant
Blackgram
Broad bean
Brussel sprouts
Bulbs
Cabbage
Carrot
Cassava
Cauliflower
Cherries
Clover
Cocoa
Coffee

Cotton
Flax
Field Beans
Groundnuts
Jute
Kale
Kenaf
Lettuce
Linseed
Lucerne
Lupins
Melon
Mungbean
Oil palm
Oil seed rape
Onions

Parsnip
Peas
Peaches
Potatoes
Radish
Raspberries
Redcurrants
Redgram
Rubber
Soyabean
Spinach
Strawberries
Sugar beet
Sunflower
Swede

Tea
Tomato
Turnips
Vines
Whitebeans

Legume cover crops
(*Pueraria*,
Centrosemma
Calopogonium)

Temperate annual grass control

Results from an autumn trial at Valencia, Spain are given in Table 3

TABLE 3

Effect of different application times of fluazifop-butyl on control of annual grasses (Spain 1979 trials ref A

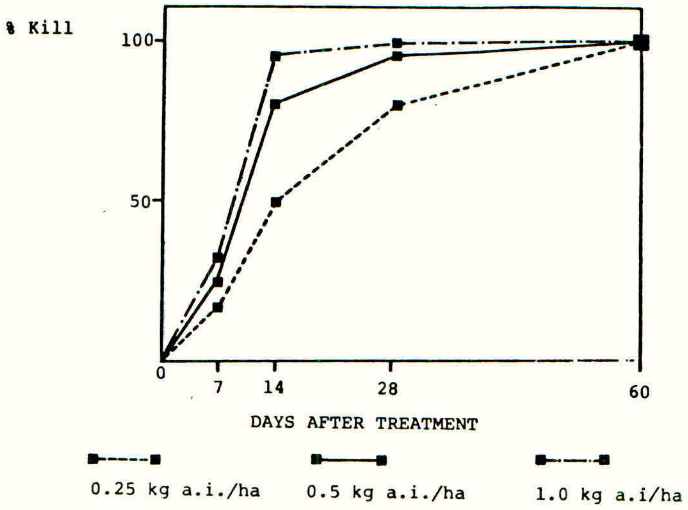
				percent kill				
Application	Chemical Treatment	Rate(kg ai/ha)	Assessment (Days After Treatment)	Lolium perenne	Alopercurus myosuroides	Avena fatua	Barley	Wheat
Pre-emergence	Fluazifop-butyl	0.25	44	30	0	15	0	0
	"	0.5	44	45	30	35	20	10
	"	1.0	44	70	60	55	35	30
Early post-emergence (2-3 leaves)	Fluazifop-butyl	0.25	60	75	80	100	90	90
	"	0.5	60	85	100	100	95	100
	Alloxydim Na	1.0	60	65	50	100	45	15
Late Post-emergence (4 leaves to tillering)	Fluazifop-butyl	0.25	60	75	95	100	100	100
	"	0.5	60	90	100	100	100	100
	Alloxydim Na	1.0	60	30	50	90	25	15
	Untreated control	-	-	0	0	0	0	0

The pre-emergence results indicate a useful soil component of activity. However, weed control is not comparable to post emergence applications. Post-emergence grass weed control was excellent at 0.25 - 0.5 kg/ha and the rate required was independent of the growth stage. These results also show the good activity of fluazifop-butyl against wheat and barley.

The results displayed in Figure 1, taken from the same trial as table 3, show the relatively slow action on grass weeds. Growth is halted rapidly, but existing leaves are slow to die. The speed of action is accelerated in some species by higher rates of application.

FIGURE 1

Speed of action of fluazifop-butyl on Avena fatua (Spain 1979)



Temperate perennial grass control

Fluazifop-butyl shows good control of Agropyron repens in a cultivated situation where fragmentation of rhizomes has occurred (Table 4).

TABLE 4

Effect of fluazifop-butyl on Agropyron repens in sugar beet (UK 1980 - trial reference D

Chemical Treatment	Rate (kg ai/ha)	Percent kill Growth stage weed 4-5 leaves
Fluazifop-butyl	0.25	97
	0.5	99
	1.0	97
Alloxydim Na	1.88	97
Untreated Control	-	(28 tillers per sq metre)

Warm temperate annual grass control

Warm temperate annual grasses were effectively controlled at low rates (0.14 to 0.28 kg/ha) (Table 5).

TABLE 5

Effect of fluazifop-butyl on annual grasses 28 days after treatment (USA 1979 trials ref F

Application	Chemical Treatment	Rate (kg ai/ha)	percent kill Echinochloa crus-galli	Brachiaria platyphylla	Digitaria spp	Eleusine indica
Early post-emergence (0-3 leaves)	Fluazifop-butyl	0.14	59 BC	99 A	79 A	92 A
	"	0.28	96 AB	100 A	99 A	100 A
	"	0.56	99 A	100 A	100 A	100 A
	Diclofop-methyl	1.12	38 D	89 A	95 A	98 A
Late post-emergence (early tillering)	Fluazifop-butyl	0.14	45 B	80 B	91 A	97 A
	"	0.28	83 A	99 A	98 A	99 A
	"	0.56	92 A	99 A	97 A	99 A
	Dichlofop-methyl	1.12	8 C	86 B	71 B	99 A

(treatments with no letter in common are significantly different at the 5% level).

The results from Table 5 show that similar rates of fluazifop-butyl are required to give good control of widely differing growth stages.

Warm temperate perennial grass control

Excellent control of Sorghum halepense has been obtained in a cropping situation with fluazifop-butyl and results are presented in Table 6.

TABLE 6

Effect of fluazifop-butyl on Sorghum halepense (fragmented rhizomes) in Cotton (USA 1979 trial ref E

APPLICATION/ GROWTH STAGE	CHEMICAL TREATMENT	RATE (KG AI/HA)	PERCENT KILL DAYS AFTER TREATMENT 28	97	105
Early Post-emergence 2 leaves crop stage (S halepense 20cm height)	Fluazifop-butyl	0.56	88 A	-	48 B
	"	0.84	71 B	-	35 B
	"	1.12	92 A	-	78 A
	"	0.28+0.28	95 A	-	73 A
	(split application)	(8 day interval)			
Mid-post-emergence 4-6 leaves crop stage (S halepense 40cm height)	Fluazifop-butyl	0.56	98 A	73 A	-
	"	0.84	99 A	78 A	-
	"	1.12	99 A	89 A	-

(treatments with no letter in common are significantly different at the 5% level)

A 0.56 kg/ha applied at the mid post-emergence stage gave season-long control. At the earlier growth stage twice this rate was needed for similar control. A split application of 0.28 kg/ha followed by 0.28 kg/ha eight days later also gave season-long control. These results suggest that the herbicide may be less well translocated into the rhizome system at this early stage, allowing some weed recovery.

This trial was sprayed under conditions of high air humidity and active weed growth. Other trials have shown that under conditions of low air humidity or when the weed is drought-stressed, higher rates are needed for good control. Under such adverse regimes a split application helps to reduce the overall dose needed.

Higher rates are required to control this weed when rhizomes remain unfragmented.

Tropical grass weed control

Paspalum conjugatum, an important perennial weed of tropical plantation agriculture, is very effectively controlled by fluazifop-butyl (Table 8).

TABLE 8

Effect of fluazifop-butyl on Paspalum conjugatum in unshaded conditions (Malaysia 1978 trial ref C

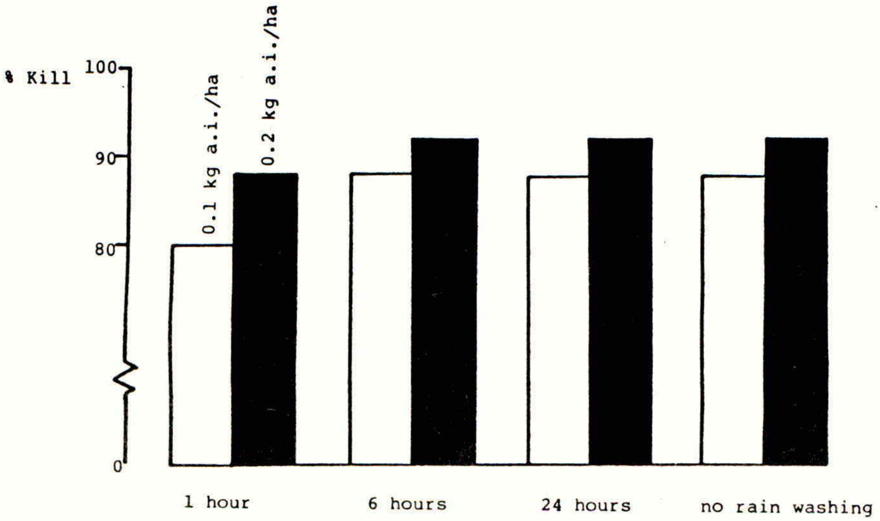
CHEMICAL TREATMENT	Rate (kg ai/ha)	PERCENT KILL (DAYS AFTER TREATMENT) 7	14	28	56	84	112
Fluazifop-butyl	0.5	12	50	88	92	88	77
Fluazifop-butyl	1.0	15	55	88	96	92	85
Glyphosate	0.75	10	38	67	73	45	21
Untreated Control	-	<5	<5	<5	<5	<5	<5

Rainfastness

Figure 2 shows the results of a rainwashing trial carried out in Brazil. Simulated rain (28mm) was applied with a sprinkler irrigation system 1, 6 and 24 hours after spraying. Even after 1 hour, rainwashing had little effect on fluazifop-butyl activity. Results are presented as mean values for three grass species, Echinochloa crus-galli, seedling Sorghum halepense and Cenchrus echinatus.

FIGURE 2

Effect of simulated rainwashing on activity of fluazifop-butyl against three grass species (Brazil 1979 trial ref B



DISCUSSION

Fluazifop-butyl is a highly active systemic herbicide which provides effective control of most problem annual and perennial grass weeds. The compound compares very favourably with commercially available post emergence grass weed killers, and is selective to broad leaved crops.

Its good activity against cereals will be useful in controlling volunteer weed problems that often occur in broad leaf crops following a cereal.

Post-emergence control of annual grass weed control by fluazifop-butyl appears to be independent of the growth stage allowing a flexibility of application, which linked with the bonus of some soil residual activity suggest considerable potential for use from fluazifop-butyl in agriculture, horticulture and forestry.

Fluazifop-butyl offers really effective post-emergence control of many of the most serious perennial weeds in crop situations. (Finney Sutton 1980)

Acknowledgement :

Fluazifop-butyl is a patented compound of Ishihara Sangyo Kaisha Ltd. ICI and Ishihara SK are jointly developing the compound for worldwide use in agriculture, horticulture and forestry.

References

FINNEY J R SUTTON P B (1980) Planned Grass Weed Control with Fluazifop-butyl in broad leaved crops. Proceedings 1980 British Crop Protection Conference - Weeds.

NOTES

NP55 - A NEW HERBICIDE FOR GRASS WEED CONTROL IN VEGETABLE, FRUIT, FODDER AND PROCESSING CROPS

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Summary: NP55 has been evaluated over, three seasons, in wide range of broad-leaved crops for annual and perennial grass weed control.

Doses between 0.32 - 3.3 kg a.i./ha have shown a high level of selectivity in the crops tested which included various beets, crucifers, legumes and strawberries.

The control of annual and perennial grass weeds including *Avena* spp., *Alopecurus myosuroides*, volunteer barley, *Agropyron repens* and *Agrostis gigantea*, was achieved at doses of 0.37 kg a.i./ha and 0.83 kg a.i./ha respectively. Mixtures with appropriate broad-leaved weed herbicides for beet and cruciferous crops were tested and no effect on crop tolerance or efficacy was noted with any mixture.

Resumé L'efficacité de NP55 contre les graminées annuelles et vivaces (était etudée pendant trois saisons) dans une grande gamme de cultures dicotyledones.

Aux doses entre 0.32 et 3.3 kg m.a./ha la sélecticité est bonne pour toutes les cultures expérimentées; les betterave, les cruciferes les légumes et les fraises y compris.

L'efficacité contre les graminées annuelles et vivaces (*Avena* spp. *Alopecurus myosuroides*, repousses de l'orge, *Agropyron repens* et *Agrostis gigantea*) est obtenu au dose de 0.37 et 0.83 kg m.a./ha respectivement.

L'efficacité et la selectivité de NP55 pour les betteroyes et les cruciferes n'est pas réduite en application de melange extemperane d'une herbicide anti dicotyledone.

INTRODUCTION

NP55 is a new selective herbicide belonging to the same group of chemical compounds as alloxydim-sodium (NP48 Na) and has similar properties. Thus the product possesses both pre and post-emergence activity specifically against *Gramineae*. Residual pre-emergence activity is of short persistence and the product is preferentially used post-emergence.

A series of small plot trials were commenced in the UK in Spring 1979. Tank mixes with commercially available broad-leaved weed materials were tested where appropriate.

METHODS AND MATERIALS

Methods

(i) Application: All treatments were applied with a self-propelled precision small plot sprayer at volume rates of 350 l/ha and 206.4 l/ha in 1979 and 1980 respectively using flat fan 'Tee-Jets' at a pressure of 2.07 bars.

(ii) Layout: Trials were of randomised block design with 3 or 4 replicates. Plot sizes were usually 2.5m x 7m.

(iii) Assessments: Visual assessments of crop condition were made using a 0 - 100% system in 2.5% increments where 100% = complete kill 0 = complete tolerance.

All scores were compared with the unsprayed control, a positive sign denoting an improvement in the crop over the untreated control.

Weed growth stages are quoted using the standard reference Zadoks scale (Zadoks *et al* 1974).

(iv) Yields: Onions - fixed row lengths were hand lifted and weighed.

Brassicae - fixed row lengths were hand cut and weighed recording number of cabbages.

Weed control

Weed counts in 2 x 0.5m^2 quadrats per plot were carried out, recording numbers and heights of the individual weed species present.

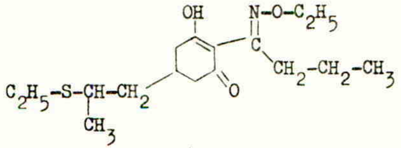
Crops

The crops and varieties tested are shown in table 1.

Chemical and physical properties of NP55

Chemical name: 2-(N-ethoxybutyrimidoyl)-5-(2-ethylthiopropyl)-3-hydroxy-2-cyclohexen-1-one.

Structure:



Odour/Appearance: Odourless, oily liquid.

Solubility: Soluble in organic solvent.
Solubility in water at 25°C. ...... 24.5 ppm.

Vapour pressure: 10^{-6} mmHg (estimated).

Toxicity: Acute oral LD_{50} mg/kg
Rats ca 1500 - 2500
Mice ca 6000 - 6500

Table 1

Crop				
Beets				
Sugar beet	Vytomo	S. Klein Mono	Nomo	Bush Mono G.
Fodder beet	Monorosa			
Red beet	Detroit Globe			
Mangels	Wintergold			
Onions				
Jap. overwinter	Imai	Senshyu		
Main crop bulb	Augusta			
Brassicae				
Summer cabbage	Primo	Greyhound	Celtic	
Winter cabbage	Stonehead	Jan King		
Sprouts	Citadel			
Broccoli	Italian sprouting			
Cauliflower	All Year Round			
S. greens	Pixie	Hardy Offenham		
Oilseed rape	Jet Neuf			
Strawberries	C. favorite	Red Gauntlet		
Broadbeans	Beryl			
Dwarf beans	Cascade	Provider		

Materials and formulations used

material	formulation	trade name
NP55	18.4% w/v EC	ARD 34/02
Alloxydim sodium	75% w/w SP	'Clout'
Phenmedipham	11.4% w/v EC	"Betanal E"
3,6-Dichloropicolinic acid	35% w/w WP	"Benazalox"
Diclofop-methyl	36% w/v EC	"Hoegrass"
Barban	12.5% w/v EC	"Carbyne"
	Adjuvant oil	"Siffren"
	" "	"Actipron"
Nitrofen	25% w/v EC	"Tok E"

Table 2

Mean control of annual grass weeds with NP55 over the period spring - autumn

% control of weed bulk (Nos times height)

		Spring treatment			Autumn treatment			
Compound	rate kg a.i./ha	Avena fatua	Avena fatua	Alopecurus myosuroides	Volunteer barley	Alopecurus myosuroides	Volunteer barley	Volunteer barley
NP55	0.32	93*	-	98	-	-	-	-
	0.37	97*	97	99	93	100	85	97
"	0.64	-	-	-	96	100	96	97
NP55 + 3,6-dichloropicolinic acid + benazolin	0.37+ 0.35	-	-	-	-	-	-	89
NP55 + oil	0.32 + 5.0 l/ha	99	99	-	-	-	-	-
Alloxydim-Na	0.94	94	-	-	78	99	-	89
Alloxydim-Na + phenmedipham	0.94 + 1.14	96	-	99	-	-	-	-
Diclofop-methyl	1.26	92	-	88	-	-	-	-
Barban	0.625	68	-	69	-	-	-	-
Crop		Sugar beet			Autumn onions		Spring Greens	Oilseed rape
Assessment post spray		3-5 weeks			6-8 weeks		4-5 weeks	3-4 weeks
Weed growth stage at spray		12-23		12-24	12-23	12-23	12-22	12-21
No of sites		5	1	3	3	1	2	3

*Treatment applied as a tank mix with phenmedipham 1.4 kg ai/ha.

RESULTS AND DISCUSSION

Weed control

The control of graminaceous weeds is shown in table 2. Good control of volunteer barley and blackgrass up to early tillering was obtained with NP55 at 0.37 kg a.i./ha. However, in the dry autumn of 1979, with spring greens, 0.64 kg a.i./ha of NP55 was needed for good control of volunteer barley. With 0.37 kg a.i./ha control was only adequate. Active growth and adequate soil moisture seem necessary for good control of volunteer cereal by NP55.

In sugar beet NP55 with or without phenmedipham (table 2) applied when the weeds were at growth stages 12-23 (Zadoks) gave good control of wild oats and blackgrass in both 1979 and 1980. The NP55 treatments were more effective than the standard treatment diclofop-methyl and barban.

Table 3

The reduction in couch shoot growth with NP55 in annual and perennial crops, 1979 and 1980

% reduction in weed foliage bulk (Nos. times height)

		weed species							
Compound	rate kg a.i./ha	Agropyron repens -p	Agropyron repens +p	A.repens/Agrostis gigantea -p	+p	A.repens -p	A.repens -p	A.repens -p	Ag.gigantea -p
NP55 ± phenmedipham (p)	0.46 ± 1.14	10	98	-	-	87	81	63	100
" "	0.64 ± 1.14	-	-	94	96	-	-	-	-
" "	0.69 ± 1.14	83	99	-	-	75	91	87	100
" "	0.83 ± 1.14	-	-	96	89	-	-	-	-
" "	0.92 ± 1.14	88	99	-	-	96	84	82	100
NP55 + oil	0.46 + 5.0 l/ha	78	-	-	-	95	90	-	-
" "	0.64 + 2.5 l/ha	-	-	96	-	-	-	-	-
Alloxydim-Na	1.88	97	-	-	-	95	86	95	95
Crop		Sugar beet				Onions	Summer cabbage	Strawberries	
Weeds assessed post spray		3-4 weeks				2-3 wks	4 wks	4-6 weeks	
Weed growth stage		2-4 leaves/shoot throughout							
Occurrence		2	2	3	3	2	1	5	1

Data on perennial grass weed control in arable crops is shown in table 3. Reliable suppression of *Agropyron* repens and *Agrostis gigantea* required doses of NP55 with or without phenmedipham in excess of 0.69 kg a.i./ha for an equivalent effect to alloxydim sodium at recommended rates. In strawberries control of *A. gigantea* was better than *A. repens*.

The addition of adjuvant oil to NP55 gave improved control of both annual and perennial grass weed species (tables 2 and 3).

Table 4

Field crop tolerance of NP55. Values are the highest scores recorded at any one time throughout evaluation period. 1979-1980

Scored on 0-100% system (100 = dead)

Compound	rate kg a.i./ha	Sugar beet*	Sugar beet	Onions main crop	Onions overwintered	Summer cabbage	Spring greens	Oilseed rape	Strawberries
NP55	0.32	5	-	-	-	-	-	-	-
"	0.37	5	5	-	0	-	0	3	-
	0.46	0	-	2.2	-	0	-	-	+6.1
	0.64	-	-	-	0	-	4.2	3	-
	0.69	1.1	-	1.6	-	0	-	-	+10.2
	0.83	-	0.8	-	-	-	-	-	-
	0.92	1.1	-	1.6	0	0	0	9	+11.0
	1.7	-	0	-	-	-	-	-	-
	3.3	-	0	-	-	-	-	-	-
NP55 + oil	0.64 + 2.5 l/ha	-	0	-	-	-	-	-	-
NP55 + 3,6-dichloropicolinic acid + benazolin	0.37 + 0.35	-	-	-	-	-	-	0.3	-
Alloxydim-Na	0.94	-	0	-	0	-	-	-	-
	1.88	-	0	1.1	-	-	-	-	+ 4.3
No. of sites		7	-	3	2	1	2	3	5
Growth stage at spraying		2-6L		2-3L	2-3L	10-15L	3-5L	2-4L	In bud

All scores negative except where indicated.
* Tank mix with phenmedipham 1.14 kg ai/ha.

Table 5

Tolerance of various varieties of crops to NP55
Highest scores recorded up to 1 month post spray

0-100% score

Compound	rate kg a.i./ha	Days post spray	Crop species								
			Mangels (Wintergold)	Fodder Beet (Monorosa)	Red Beet (Detroit Globe)	Swedes (Western perfection)	Sprouts (Citadel)	Broccoli (Italian sprouting)	Cabbage (Greyhound)	Cabbage (January King)	Cabbage (Primo)
NP55	0.83	7	0	0	0	0	0	0	0	0	0
		14	0	0	6.3	0	0	0	0	0	0
		28	0	0	0	0	0	0	0	0	0
NP55	1.7	7	0	0	0	3.3	0	2.5	1.3	0	0
		14	0	0	7.5	0	0	0	0	0	3.3
		28	0	0	0	0	0	0	0	0	0
NP55	3.3	7	2.5	2.5	5.0	20	0	10	3.3	5	7.5
		14	3.1	3.5	8.8	10	0	9.2	3.3	5	3.3
		28	1.0	0	0	0	0	0	0	0	0
NP55 + phenmedipham	0.83 + 1.14	7	0	0	0.6	-	-	-	-	-	-
		14	0	1.3	10	-	-	-	-	-	-
		28	2.5	0	1.3	-	-	-	-	-	-
NP55 + nitrofen	0.83 + 0.28	7	-	-	-	2.5	0	0	0	0	0
		14	-	-	-	0	0	0	0	0	0
		28	-	-	-	0	0	0	0	0	0
Growth stage at spraying			4-7L	4-9L	4-7L	5-8L	6-8L transplant	5-6L	4-7L	3-5L	3-6L

Crop tolerance

Table 6

Tolerance of broad beans and dwarf green beans

Logarithmic screening results

Crop	Variety	Growth stage at spraying	Max tol dose kg a.i./ha
Broad beans	Beryl	5 L	2.86
Dwarf green beans	Cascade	$2\frac{1}{2}$L	2.6
" " "	Dark Seeded Provider	$2\frac{1}{2}$L	2.6

NP55 was well tolerated by all the crops tested at rates from 0.92 to 3.3 kg a.i./ha depending on trial type (tables 4, 5, 6, 7). A rate of 3.3 kg a.i./ha is about 4 times that anticipated as needed for couch control.

With strawberries the crop was increased, probably due to the removal of intense couch competition. With several crops a slight transient phytotoxic effect occurred with the 4 x rate. Crops fully recovered in under 4 weeks. Good tolerance was also shown to mixtures of NP55 with phenmedipham, 3,6 - dichloropicolinic acid/benazolin nitrofen or oil adjuvants in sugar beet, rape and cruciferous vegetable crops.

Harvest yields (table 6) reflected the excellent crop tolerance (tables 4, 5 and 6). The variability of yields in the overwintered onions (site 2) can be attributed solely to the natural fluctuations in this crop. In spring greens all treated plots showed increases over the unsprayed controls including one site where initial weed control was affected by adverse weather conditions.

Table 7

Harvest yields of overwintered vegetables. 1979/80

Total yields as % of unsprayed control and average plant weight in grammes

			Overwintered onions**				Spring greens			
			Total weight		Av.weight		Total weight		Av.weight	
Material	Kg a.i./ha	Site No	1	2	1	2	1	2	1	2
NP55	0.37		93	118	83	76	290*	232*	92	93
	0.67		97	90	84	69	284*	277*	93	108
	0.92		117	82	86	71	181*	-	80	-
Alloxydim-Na	0.938		97	86	80	80	271*	321*	79	138
" "	1.88		95	91	75	87	284*	312*	91	134
U/S Control Yield			37t/ha	51t/ha	72	74	7.75t/ha	5.60t/ha	40	46
Level of significance			N/S	N/S						

Treated September/October 1979. Harvested June 1980. * Significant at 5% level.
** Weed free trials.

Broad-leaved weed control

At rates from 0.32 to 3.3 kg a.i./ha NP55 showed no measurable activity on broad-leaved weed species. Mixing with phenmedipham had no detrimental effect on either grass or broad-leaved weed control.

Acknowledgements

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References: ZADOKS, J.C., CHANG, T.T. and ZONZAK, C.F. (1974). A decimal code for the growth stages of cereals. Weed Research, 14, 415-421.

Proceedings 1980 British Crop Protection Conference-Weeds

NEW THIOPYRIMIDINES AS HERBICIDES

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Summary A new serie of 5-thiopyrimidines has shown good herbicidal properties. They are selective, pre and post-emergence, in gramineous crops as cereals, maize, rice and sorghum. They control grasses and broad-leaved weeds and absorbtion occurs through both leaves and roots. Mammalian toxicity appears to be low.

Résumé Une nouvelle famille de thio-5-pyrimidines a montré de bonnes propriétés herbicides. Ces produits sont sélectifs, entre autres des céréales, du maïs, du riz et du sorgho. Ils détruisent un grand nombre de mono et dicotylédones adventices, et ils sont absorbés par voie racinaire et foliaire. Leur toxicité sur mammifères est faible.

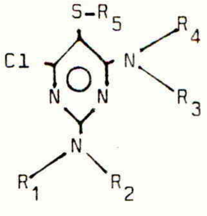
INTRODUCTION

5-thiopyrimidines are new herbicides being developed by P.C.U.K. This paper describes the chemical structure of the group, the pre and post-emergence herbicidal activity, the effect of structure on activity and some results of preliminary field testing.

CHEMICAL AND PHYSICAL STRUCTURE

These new compounds have the general structure shown in fig. 1

Fig 1. general structure



R_1 R_2; R_3 R_4 and R_5 are either a hydrogen atom or an alkyl group. They are obtained from 2-4-6-trichloro-5-thiopyrimidines, by reaction with ammonia and/or various amines.

Some of the synthesized compounds are shown in table 1.

Table 1

The structures of some 5-thiopyrimidines and their melting point

Code Index	R_1	R_2	R_3	R_4	R_5	Melting point (°C)
	Side group (fig.1)					
A	H	C_2H_5	H	H	CH_3	124
B	H	H	H	CH_3	CH_3	100
C	H	H	H	H	C_2H_5	182
D	H	H	H	H	C_3H_7	129
E	H	H	H	H	CH_3	154
F	C_2H_5	C_2H_5	H	H	CH_3	68
G	H	CH_3	H	H	CH_3	200
H	H	H	H	CH_3	CH_3	-
I	H	CH_3	H	CH_3	CH_3	-

Primary screening herbicidal activity

Method and materials - Two concentrations of active ingredient, in water , were formulated with a surfactant "Tween 20". These dilutions were sprayed at 1.000 l/ha on to, either seeds (pre-emergence treatments), or fifteen days old plants (post-emergence treatments).

The plants used were : wheat, French bean, sugar beet, mustard, dandelion and maize.

Observations were made, either after twenty days (pre-emergence tests), or fifteen days (post-emergence tests).

The plants were evaluated against a : 0 to 100 scale , where 0 represents no difference between the treated and untreated seeds or plants, 100 represents complete death.

Results - Sample results are shown in tables 2 and 3.

Table 2 shows that compounds A,B,C,D have good post-emergence activity against a range of species : sugar beet, mustard, dandelion. A and C are active against gramineous plants : wheat and maize. B and D have a good selectivity in maize. C has a good selectivity in French bean.

Table 2

Post-emergence herbicidal activity of some 5-thiopyrimidines compounds
Values : 0-100 relative scale (0 no effect - 100 dead)

Compounds (Table 1)	Rate (kg a.i/ha)	Wheat	French bean	Sugar beet	Mustard	Dandelion	Maize
A	10	75	100	100	100	100	100
	2.5	70	100	100	100	100	70
B	10	65	100	100	100	100	0
	2.5	0	0	100	100	100	0
C	10	50	70	100	100	100	70
	2.5	20	50	100	100	100	15
D	10	60	0	100	100	100	0
	2.5	15	0	100	100	100	0

Table 3

Pre-emergence herbicidal activity of some 5-thiopyrimidines
Values : 0-100 relative scale (0 no effect - 100 dead)

Compounds (Table 1)	Rate (kg a.i/ha)	Wheat	French bean	Sugar beet	Mustard	Dandelion	Maize
A	10	15	50	100	100	100	0
	2.5	0	0	0	0	0	0
B	10	0	0	0	0	0	0
	2.5	0	0	0	0	0	0
E	10	45	80	100	100	100	0
	2.5	15	0	100	50	100	0
F	10	75	100	100	100	100	0
	2.5	50	80	100	100	100	0

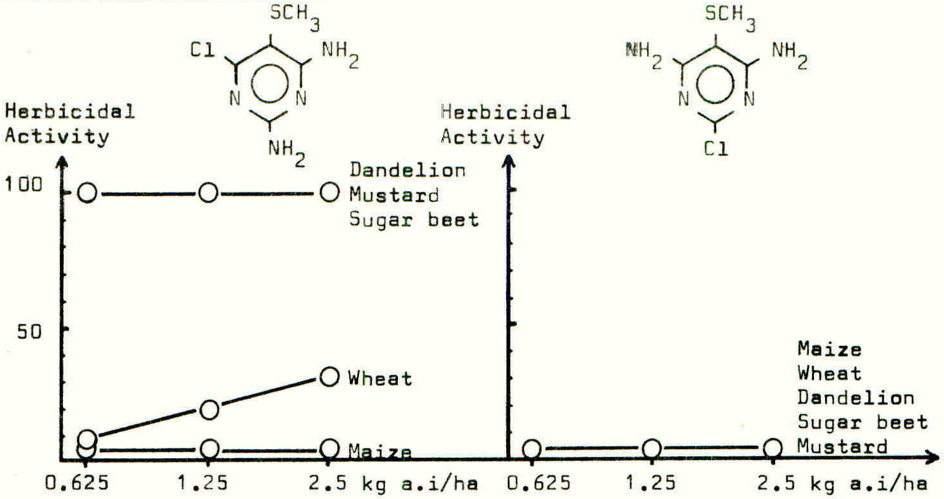
In pre-emergence test (table 3) the compound B is inactive, the compound A is slightly active, but the compounds E and F show a good herbicidal activity and they are selective in maize.

Structure - Activity relations

Thio group : A thio group at the 5-position is necessary to give good herbicidal properties to these compounds. Substitution of this group or a change in its position reduces the herbicidal activity.

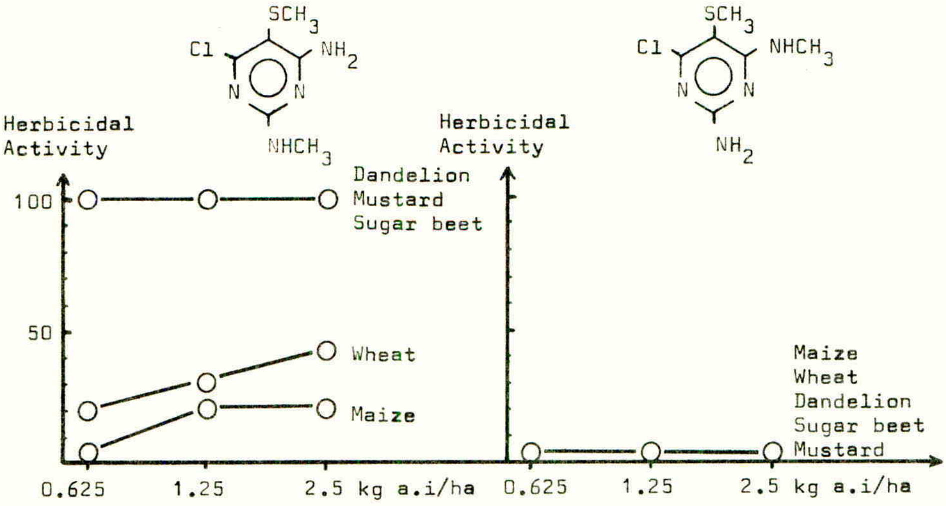
Chlorine atom on the ring : The chlorine must be in 6-position (fig. 2). The 2-chloro isomers have no herbicidal properties. Substitution of the chlorine by other groups or functions reduces the herbicidal activity.

Fig. 2 : The effect of the position of the chlorine atom on herbicidal activity against a range of crops (0 no effect - 100 dead).



Amino groups : Herbicidal activity is greatest when the 4-amino group is unsubstituted (fig. 3). The structure of the 2-group is less important.

Fig. 3 : The role of the 4-amino group on postemergence herbicidal activity (0 no effect - 100 dead).



Plot tests and field tests

Glass house experiments have shown that some of those products were selective in cereal crops, maize, rice and sorghum and since 1978, 250 field tests have been carried out.

Plot tests : The products were used as a wettable powder (80 %) in water and sprayed on crops and weeds both pre and post-emergence. Effects were evaluated on a 0 to 10 scale :

0 : no difference between treated and untreated plants ;
10 : total control.

Effects of compounds E and F (table 1) on wheat barley and maize and some of common weeds are shown in table 4.

Table 4

Post-emergence herbicidal activity - plot tests - values are scores on a 0 to 10 scale - 0 no effect - 10 dead

	Products-rates (kg a.i/ha)					
		E			F	
Species	0.625	1.25	2.5	0.625	1.25	2.5
Wheat	0	1	3	0	2	4
Barley	0	0	2	0	2	3
Maize	0	0	0	0	0	0
Amarantus retroflexus	10	10	10	8	10	10
Chenopodium album	5	10	10	10	10	10
Solanum nigrum	7	10	10	10	10	10
Mercurialis annua	6	10	10	8	10	10
Sonchus oleraceus	2	8	10	5	10	10
Convolvulus sp.	0	2	3	0	2	4
Panicum miliaceum	0	8	10	2	7	10
Digitaria sanguinalis	2	5	10	3	4	10

No damage occured to the crop plants from compounds E and F at a rate of 0.625 kg a.i/ha, high rate of 2.5 kg a.i/ha caused some damage particularly on wheat and with product F. E controled *Amarantus retroflexus* at 0.625 kg a.i/ha and 3 others species at 1.25 kg/ha. Product F controled *Chenopodium album* and *Solanum nigrum* at 0.625 kg a.i/ha and 3 others species at 1.25 kg a.i/ha.

Field tests : The product were again used as a wettable powder formulation (80 % a.i) in water and sprayed at 1.000 l/ha.

The effect of a range of concentration of product E, (table 1) applied on wheat, is shown in table 5.

Table 5

The effect of a pre-emergent treatment with a 5-thiopyrimidine : product E, applied on wheat to control *Alopecurus myosuroïdes*

Treatment (Table 1)	Rate (kg a.i/ha)	*A.myosuroïdes* plant n°/m2	Effect on wheat (0-10 scale) (1)
E	0.6	122	0
	1.2	10	0.5
	1.8	5	2
Untreated		120	0

(1) 0 : no phytotoxicity
10 : dead

In that essay, there is a good control of *A. myosuroïdes* with 1.2 kg a.i/ha of product E. In most of the experiments,"5-thiopyrimidines" appear potentially usefull herbicides in cereals to control : *Alopecurus myosuroïdes* - *Poa annua* - *Sinapis arvensis* - *Ranunculus repens* - *Viola tricolor* - *Stellaria media* - *Veronica sp*. - *Matricaria inodora* ; and in maize to control : *Panicum miliaceum* - *Digitaria sanguinalis* - *Amarantus retroflexus* - *Ambrosia artemisiaefolia* - *Chenopodium album* - *Polygonum persicaria* - *P. aviculare* - *Solanum nigrum*.

Toxicology

The acute oral toxicity of the 5-thiopyrimidine on rats were 300 mg/kg $<$ LD 50 $<$ 900 mg/kg.

Compounds appeared to have no mutagenic effect (Ames and Micronucleus tests) and no teratogenic effect on rats and rabbits.

Conclusion

The 5-thiopyrimidines are very promising herbicides active against a range of broad-leaved and grass weeds. They show good selectivity in cereal crops,rice, maize and sorghum.

Proceedings 1980 British Crop Protection Conference - Weeds

THE CONTROL OF AGROPYRON REPENS AND BROAD-LEAVED WEEDS PRE-HARVEST OF WHEAT AND BARLEY WITH THE ISOPROPYLAMINE SALT OF GLYPHOSATE

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Summary Twenty five small plot experiments each of four replicates show that glyphosate applied pre-harvest of wheat and barley is effective in controlling annual and perennial grass and broad-leaved weeds. Efficacy data were supplemented by sixty four field-size observations applied by farmers. Applications made when the moisture content of the cereal grain is below a bulk mean of 30% are safe to both the crop and the treated cereal grains when assessed for germination energy and capacity, nitrogen content and the resultant bread-making qualities of wheat and malting qualities of barley.

Résumé 25 essais a 4 repetitions effectués sur petites parcelles ont demontre que le glyphosate applique en pre-recolte du froment et de l'orge, est efficace pour la destruction des graminees et dicotyledones annuelles et vivaces. Des preuves d'efficacite ont ete apportees par les observations provenant de 64 essais en champs conduits par les fermiers. Les traitements effectues lorsque le taux d'humidite des graines de cereales est inferieur a une moyenne de 30% sont tout-a-fait sors pour la culture et les graines, ainsi que cela a ete verifie par des tests de pouvoir germinatif, de teneur azotee, de la valeur panifiable du froment et de celle de l'orge utilise en brasserie.

INTRODUCTION

The use of the isopropylamine salt of N-phosphonomethyl glycine glyphosate 360 grammes/litre for the control of Agropyron repens and other perennial grass weeds in cereal stubbles was introduced to the United Kingdom in 1974 (Evans 1972). Since then, this treatment has become the standard for the control of perennial grass weeds throughout the country irrespective of harvest date and subsequent cultivation system.

Cereal husbandry has changed significantly since 1974. The area of winter cereals has increased from 1.3 to 2.2 million ha between 1974 and 1979 (MAFF 1975 and 1980); the date of drilling the cereal crops has become earlier as winter barley has become an increasing proportion of the winter cereal crop. However, the farm labour force has declined from 260,000 in 1974 to 155,000 in 1979 with the extra winter cereals being drilled only with more powerful equipment and a general change in cultivation method towards tine cultivation only instead of mould-board ploughing with subsequent disc or tine cultivations.

Reduced soil disturbance has lead to less physical damage to the rhizomes of perennial grasses and a tendency for them to be located shllower than where fields are ploughed. Perennial grass weeds are now, therefore, quicker to emerge in winter cereal crops; can make more growth before soil temperatures fall below 6°C; better utilize nutrients applied to the crop and so are much more competitive with early sown winter cereals under reduced cultivation than with October/November drilled crops where soil was ploughed (Nuyken 1975).

Glyphosate is most effective when applied at the time new rhizome is being produced and the plant tillers. With the above change in culture and the increasing tendency to burn straw or stubble, glyphosate has sometimes been applied before the optimum stage of weed growth for control. Where harvest is delayed until late September/early October, frequent in Northern England and Scotland, the date when perennial grasses reach the growth stage susceptible to glyphosate often coincides with a period of low temperatures or frost. This induces dormancy in the weeds and seriously limits glyphosate translocation (Gottrup et al 1976, Caseley J 1972). Experiments at the A.R.C. Weed Research Organisation (Davison, 1972) showed broad-leaved weeds could be controlled at this stage of growth, while trials in both the U.S.A. and Europe (Kemmer, 1978 and Friesen, 1977) indicated that Agropyron repens should be sensitive if active growth was occurring and senescence had not commenced.

As a result, the possibility of applying glyphosate pre-harvest of cereal and other small seeded crops was evaluated. Commercial factors indicated that priority should be given to wheat and barley.

Wheat and barley grains reach their maximum dry matter content at c. 37% moisture content (Mitchell et al 1980). After this, the grain matures and exists independently of the rest of the plant. When the bulk moisture content of the grain is 30%, 95% of individual grains (by weight) have under 34% moisture. The risk at this stage of reducing yield is negligible.

A label recommendation for the application of up to 1.44 kg/ha glyphosate has been granted Full Commercial Clearance and Approval under U.K. registration schemes. This paper describes the treatments of glyphosate pre-harvest of wheat and barley and gives results of crop and grain assessments and perennial weed control at up to one year after treatment.

METHOD AND MATERIALS

Twenty five field experiments, all using randomised block design with four replicates were carried out in 1978 and 1979. Plot sizes varied from 4 x 15m to 6 x 25m. Details of experimental sites; dates of treatment and harvest; crop growth stage and moisture content at treatment and target weeds are given in Table 1. Treatments were all applied with modified Oxford Precision Sprayers using a 2m boom with Tee-jet nozzle tips (No.11002). Sprays were applied at volumes from 200 to 250 l/ha at a pressure of 1.4 - 2.0 bars. The average period between spraying and harvest was 13 days. The mean bulk moisture content of cereal grains at spraying was under 30% in all experiments.

Rates of glyphosate ranged from 0.36 to 4.32 kg/ha, applied as a single dose. Data on efficacy were obtained in 1979 in 64 farmer tests where rates of 1.44 and 2.16 kg/ha glyphosate were applied to a wide range of weed species including Agropyron repens, Agrostis gigantea, Arrhenatherum elatius, Lolium spp., Poa spp., Cirsium arvense, Phragmites communis, Convolvulus arvensis, Tussilago farfara and Volunteer potato.

Crop Effects

Grain moisture content at harvest and treatment was assessed using a Wet-Tone moisture metre (Wet-Tone Instruments Ltd., Kirkaldy, Fifeshire, Scotland.) with crop

TABLE 1

Experimental Details

Site	Species	Cultivar	Herbicide Application Date	Crop Growth Zadok et al	Harvest Date	% Grain Moisture Content at Application.	Target Weed
1 Peterborough, Cambs	W.W.	Hobbit	21.8.78	90	31.8.78	19	*Polygonum bistorta* Volunteer potato
2 Sleaford, Lincs	W.W.	Atou	21.8.78	88	5.9.78	27	*Agropyron repens*
3 Wisbech, Cambs	W.W.	Hobbit	23.8.78	90	13.9.78	19	*Calystegia sepium* *Mentha arvensis* *Sonchus arvensis* *Cirsium arvense*
4 Lechlade, Glos	S.B.	Aramir	8.8.78	75	2.9.78	70	*A.repens*
5 Lechlade, Glos	S.B.	Aramir	25.8.78	90	2.9.78	25	*A.repens*
6 Lechlade, Glos	W.B.	Sonja	8.8.78	91	11.8.78	18	*A.repens* *Convolvulus arvensis*
7 Mollington, Oxon	W.W.	Flanders	24.8.78	90	31.8.78	25	*A.repens*
8 Stamford Bridge, Yorks	S.B.	Lofa abed	11.8.78	90	24.8.78	24	*A.repens* *Stellaria media*
9 Wawne, Humberside	W.W.	Score	24.8.78	87	6.9.78	23	*A.repens*
10 Thirsk, Yorks	W.W.	Mega	25.8.78	88	5.9.78	25	*A.repens*
11 Thirsk, Yorks	W.W.	Mega	1.9.78	91	5.9.78	22	*A.repens*
12 Peterborough, Cambs	W.W.	Kador	20.8.79	87	7.9.79	23	*P.bistorta*
13 Boston, Lincs	W.W.	Huntsman	21.8.79	88	14.9.79	24	*A.repens*
14 Yelden, Beds	S.W.	Highbury	6.9.79	88	21.9.79	25	*A.repens*
15 Nymphsfield, Glos	S.B.	Aramir	29.8.79	90	24.9.79	22	*A.repens*
16 Lechlade, Glos	S.B.	Lofa abed	30.8.79	91	6.9.79	19	*A.repens*
17 Urchfont, Wilts	W.W.	Hustler	22.8.79	91	3.9.79	19	*A.repens*
18 Dorsington, Warwick	S.W.		29.8.79	87	15.9.79	26	*A.repens*
19 Angram, Yorks	W.B.	Sonja	21.7.79	87	27.7.79	23	*Matricaria* spp.
20 Bossall, York	W.W.	Kinsman	6.9.79	91	13.9.79	19	*A.repens*
21 Stamford Bridge, Yorks	W.B.	Igri	2.8.79	87	9.8.79	26	*A.repens*
22 Nymphsfield, Glos	S.B.	Aramir	20.8.79	80	3.9.79	35	*A.repens*
23 Lechlade, Glos	S.B.	Lofa abed	16.8.79	80	30.8.79	35	*A.repens*
24 Vekington, Glos	S.W.	Timmo	17.8.79	83	31.8.79	30	*A.repens*
25 Vekington, Glos	S.W.	Timmo	17.8.79	76	31.8.79	40	*A.repens*

W.W. - Winter Wheat S.W. - Spring Wheat W.B. - Winter Barley S.B. - Spring Barley growth stage measured using the scale devised by Zadok et al (Zadok 1974). Crop yield was taken on samples using either the bread-knife technique or a Wintersteiger plot combine with a 1.75m cut. The former was used for samples required exactly seven days after treatment for residue analysis and where the crop was too immature for combine harvesting.

Crop Grain Effects

Odour, discolouration and shedding were assessed at harvest. After harvest, thousand grain weights were measured as this seemed the only component of crop yield (grain weight, number grains per head, number heads per unit area) which glyphosate might have influenced. The numbers of fertile tillers and grains per ear were uniform between treatments and replicates before treatment. Grain germination and nitrogen percentages were assessed.

The grain from winter cultivars of wheat and barley was drilled in October 1979 and from the spring cultivars in spring 1980, using a 20 row combine drill with rows 7 and 14 blocked. Thus, 6 rows of each treatment from each replicated trial and farmer observation were drilled in 2 replicates with assessments made on field growth characteristics up to seeding. Plots received standard husbandry with a proprietary herbicide to control annual broad-leaved weeds but with no other pesticide to avoid confusion marks. Barley samples were subjected to the standard malting test of the Institute of Brewers and the wheat samples were subjected to tests to assess bread-making characteristics.

Weed Effects

Visual die-back of weeds were assessed 7, 13 and 35 days after treatment in both 1978 and 1979. Regrowth was assessed 3,6 and 12 months after treatment as number of weed shoots in a randomly placed quadrat. The relative size of treated and untreated plots meant that treated areas tended to become reinfested. Weed numbers were assessed regardless of the risk of physical reinfestation due to cultivation. It was anticipated that levels of control in the small plot replicated trials would be lower than in the larger plot farmer applied observation sites.

RESULTS

Crop Effects

Table 2 summarises crop yields from sites 12, 14 and 16 for 1979, the year of treatment.

TABLE 2

The Effect of Rate of glyphosate on Cereal Grain Yield (t/ha)at 15% M.C.

Glyphosate Kg/ha	Site Number			Mean %
	12	14	16	
0	5.09	5.37	2.07	100
0.36	5.83	5.54	1.72	104
0.72	5.34	5.33	1.90	100
1.44	5.29	5.46	1.78	100
2.16	5.53	5.33	1.92	102
2.88	-	5.29	1.86	
4.32	5.09	-	-	
Harvest, days after spraying	17	15	7	13

There were no significant differences in the yield of cereals between untreated and plots treated with glyphosate up to 4.32 kg/ha.

The 1979 crops growing on the areas treated pre-harvest of wheat and barley in 1978 were taken to yield to assess the competitive nature of the A.repens infestation. Results are at Table 3.

TABLE 3

1979 Winter Wheat Yields, t/ha at 15% M.C., on Areas Treated with glyphosate pre-harvest in 1978

Glyphosate Kg/ha	Site Number 2	5	10	Mean %
0	2.1	1.7	7.2	100
1.44	4.0	1.8	12.1	162
2.16	4.6	1.75	11.4	161

The results show that the areas treated with glyphosate pre-harvest of cereals in 1978 were over 60% higher in yield in the 1979 harvest. This is a measure of the competitive ability of A.repens.

Crop Grain Effects

a) Moisture Content of Cereal Grain.

Increasing the rate of glyphosate from 0.36 to 2.88 Kg/ha (Table 4) did not effect the moisture content of treated grain. The effect of rainfall on the moisture content of treated and untreated was similar. Treated grain is not at more risk from the weather.

TABLE 4

Effect of glyphosate on Grain Moisture Content - 1979

Site	Grain Moisture At Spraying %	Grain Moisture at Harvest % Glyphosate Rate kg/ha 0	0.36	0.72	1.44	2.16	2.88
12	23	15.7	15.7	15.6	15.5	15.2	15.2
13	24	15.5	14.7	15.0	15.0	15.5	15.5
14	25	23.0	22.5	23.0	23.0	23.0	22.5
15	22	21.2	19.4	19.6	19.2	18.9	18.5
16	19	16.1	15.7	15.4	15.2	15.1	15.6
17	19	19.7	19.7	19.0	19.0	18.4	19.3
18	26	27.9	25.6	22.1	21.8	26.1	23.6
19	23	18.0	19.0	18.0	19.5	14.7	19.7
20	19	15.5	15.5	15.2	15.5	16.0	15.7
21	26	16.2	16.7	17.0	16.0	15.5	15.2
Mean	22.6	19.4	18.9	18.5	18.5	18.4	19.0

The difference in the moisture content of grain treated with upto 2.88 Kg/ha glyphosate between treatment and harvest, which occurred at a mean of 13 days after treatment, were not biologically different to untreated grain.

b) Malting Quality of Barley.

The malting potential of the barley grains was assessed on the standard protocol of The Brewers Society. Results for grain assessments from site 16, Lechlade, are shown in Table 5.

TABLE 5

Malting Quality of Barley

Glyphosate Kg/ha	Energy	Germination % Capacity	Tetrazolium	Crude Protein - %
0	99.5	99.5	99.3	11.8
1.44	98.5	98.9	99.3	13.1
2.88	99.0	99.3	99.1	13.3

Glyphosate at 1.44 and 2.88 kg/ha did not effect the three germination characteristics measured with no biological or statistical differences showing between treated and untreated. The difference in the Crude Protein % between treated and untreated was not repeated in other assessments.

c) Milling Quality of Wheat.

Wheat grains from sites 12 (Kador), 13 (Maris Huntsman), 20 (Kinsman) and 14 (Highbury) were assessed for their milling qualities in terms of crude protein % (Table 6) and Hagberg Falling Number (Table 7).

TABLE 6

The Effect of glyphosate on the Crude Protein % of Wheat

Glyphosate Kg/ha	Crude Protein % Cultivar Kador	M. Huntsman	Kinsman	Highbury
0	12.7	8.5	11.3	10.9
0.36	13.0	8.5	12.5	10.6
0.72	11.3	8.5	11.5	10.7
1.44	12.9	8.1	12.0	11.3
2.16	12.9	8.5	11.7	10.9
4.32	12.9	8.1	12.4	11.2
Mean:	12.6	8.3	12.0	10.9

The differences in crude protein % between untreated and grains treated with up to 4.32 kg/ha glyphosate were not biologically or statistically different.

TABLE 7

The Effect of glyphosate on the Hagberg Falling Number of Wheat

Glyphosate Kg/ha	Hagberg Falling Number Kador	M.Huntsman	Kinsman	Highbury
0	276	65	137	321
0.36	314	65	228	319
0.72	219	67	137	330
1.44	299	78	218	290
2.16	340	72	187	330
4.32	316	70	229	368
Mean:	297	70	200	327

The differences in the Hagberg Falling Number are considered to be within normal limits for each of the cultivars tested and were not biologically different between treatments and the untreated grain.

d) Thousand Grain Weights.

Glyphosate at upto 2.88 Kg/ha had no effect on the thousand grain weight of cereal grains (Table 8).

TABLE 8

The Effect of glyphosate on the Weight of Treated Grains

Glyphosate Kg/ha	Thousand Grain Weight - (g) Site Number						
	14	15	16	17	18	20	Mean
0	37.7	43.0	41.3	40.2	41.6	43.1	41.1
0.36	37.4	43.4	38.3	40.2	41.7	42.7	40.6
0.72	36.6	44.7	39.5	40.9	39.6	44.3	40.9
1.44	37.2	44.1	38.9	42.7	41.0	43.6	41.2
2.16	37.2	42.0	40.7	42.4	39.8	42.3	40.7
2.88	38.4	43.2	40.0	38.7	39.8	43.8	40.7
Mean:	37.4	43.4	39.8	40.9	40.6	43.3	

Glyphosate rates upto 2.88 produced no biological differences between treatments and the untreated on the 6 sites assessed. All thousand grain weights were normal for the cultivar and the husbandry system adopted.

Weed Control Effects

Sites treated with glyphosate pre-harvest of cereals in 1978 were assessed for the control of weeds at upto one year after treatment (Table 9). The assessments 13 days after treatment coincided with harvest date and were an assessment of die-back of treated weeds. Other assessments refer to the actual control of plants.

TABLE 9

The Effect of Pre-Harvest glyphosate on Weed Control

Weed Species.	Control of Weed Species - % Glyphosate rate-Kg/ha					
	1.44			2.16		
	Days After Treatment					
	12	35	360	12	35	360
Agropyron repens	95	99	94	98	99	98
Calystegia sepium	--	99	97	--	99	99
Cirsium arvense	--	99	97	--	99	99
Convolvulus arvensis	--	--	95	--	--	95
Mentha arvensis	--	99	97	--	99	99
Polygonum bistorta	55	85	98	70	95	98
Volunteer potato	90	99	93	90	99	93
Sonchus arvensis	--	99	97	--	99	97

The speed of die-back of the perennial dicotyledon weeds is slower than the monocotyledons. The control one year after treatment was 93% or over for all species which occurred at sites 1 to 11 in Table 1. All the assessments were made in commercial crops which were subjected to the standard cultivation techniques adopted by each farmer.

DISCUSSION

Glyphosate applied pre-harvest at 0.36 - 4.32 Kg/ha of wheat and barley reduced the content of all green crop and weed growth in the treated crops and consequently improved harvesting and subsequent burning of straw and reduced the cost of cleaning and conditioning grain. Crop damage due to the passage of the herbicide sprayer was non-existent in all the autumn sown crops, all of which had either tramlines or well-defined wheelways. In the spring-sown crops damage caused by the spray machine was kept to a minimum by fitting crop dividers to the tractor wheels and harvesting at right angles to the direction of spraying. No yield loss resulted from pre-harvest spraying in autumn or spring-sown crops.

Crop grain effects were biologically and statistically non-significant for all parameters tested. The results quoted have been confirmed by results of similar assessments in 1980. Straw effects likely to influence use of treated straw, including mulching fruit crops and growth media for crops under glass, have not been fully tested, although preliminary results from composting treated straw for mushroom production indicate that biological differences between treated and untreated straw are non-significant.

Weed control effects have been outstanding, when assessed upto one year after treating A.repens and a range of commonly occurring perennial dicotyledon weeds.Late developing crop tillers and actively-growing annual weeds have been so well controlled between application date and harvesting that faster harvesting and cheaper grain cleaning and drying has resulted.

The new use for glyphosate described was introduced commercially in 1980.

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THE HERBICIDAL ACTIVITY OF A NOVEL COMBINATION OF BIFENOX AND MECOPROP APPLIED POST-EMERGENCE TO CEREAL CROPS

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Summary The combination of bifenox plus mecoprop Mg^{2+} salt, formulated as a wettable powder (EL-5591, 150 + 370 g/kg) has demonstrated selective control of a wide range of broadleaf weeds when applied post-emergence in cereal crops. In particular, high levels of activity were observed against Viola spp., Veronica spp., and Galium aparine. Control of the latter two species was maintained irrespective of size at application. Despite variability in activity against Compositae and Polygonaceae application of EL-5591 in the autumn or to small seedlings in the spring provided adequate control.
The optimum dose rates of EL-5591 w.p. were shown to be 4.0 kg product/ha for control of small seedlings and for autumn applications and 5.0 kg/ha for larger seedlings and more tolerant species.

INTRODUCTION

The potential of bifenox (methyl 5-(2,4-dichlorophenoxy)-2-nitrobenzoate) as a herbicide for the control of annual broadleaf weeds in a number of crops was described in detail by Kruger et al., (1974). Crops demonstrating tolerance to pre-emergence application included soybeans, maize, sorghum, sunflower, safflower, small grain cereals and rice. In addition, selectivity to post-emergence applications was apparent in small grain cereals and rice.

Bifenox was subsequently introduced in the U.S.A. in 1975 for use in soybeans and results of studies to elucidate the basis of the selectivity in that crop were reported by Leather and Foy. (1978).

Extensive research has been carried out on rice in Japan (Tamura & Hara, 1975; Kuwatsuka et al., 1975; Ohyama & Kuwatsuka, 1975) and the U.S.A. (Baker, 1975 & 1976; Dean et al., 1976 & 1977) culminating in a U.S.A. federal registration for both pre and post-emergence applications (Huvar & Dreger, 1978). Recent U.S.A. registrations were also reported for use of bifenox pre-emergence in grain sorghum and post-emergence in wheat.

The selectivity of bifenox in spring wheat was confirmed in the U.S.A. where application at 0.25, 0.5 and 1.0 kg a.i./ha was made at a number of crop growth stages and to a number of cultivars (Behm & Arnold, 1975).

Data from field trials in 1978 and 1979, which were carried out in a number of European countries with tank-mix combinations of bifenox and mecoprop, determined that the optimum application rates for these herbicides were 0.75 and 1.85 kg a.i./ha, respectively. As well as excellent efficacy against a range of broadleaf weeds including Matricaria spp., Galium aparine, Veronica spp and Sinapis arvensis the mixture showed selectivity at doses as high as 2.0 kg a.i./ha bifenox and 4.0 kg a.i./ha mecoprop. The good selectivity was particularly apparent from the seed yields obtained from those trials where the high dose rates were applied.

The results from an extensive 1980 field trial programme with a wettable powder formulation of bifenox and mecoprop are presented below.

METHOD AND MATERIALS

Field trials details

Two hundred and thirty-seven field trials were carried out in Europe in 1980. The trials were distributed throughout Belgium, France, Holland, Italy, Spain, United Kingdom and West Germany and included winter wheat, winter barley, winter oats, spring barley and spring wheat. Design of field trials varied between locations with most countries, except France, utilizing a randomized blocks design with 4 replicates. Trials in France were also of a randomized blocks design but with 2 replicates and paired controls. Treatments were applied with knapsack sprayers in volumes between 200 and 500 l/ha to plots of 12 to 45 m^2. Crop stage at application varied between 3 leaves and the appearance of the second node.

The following treatments were applied to the trials at the rates noted: EL-5591, bifenox + mecoprop Mg^{2+} salt, 150 + 370 g/kg w.p. 3.5,4.0 & 5.0 kg formulated product/ha.

Reference A. Ioxynil + bromoxynil + mecoprop iso-octyl ester, 75 + 75 + 376 g/l 2.5 & 3.5 l formulated product/ha.

Reference B. Dinoterb + mecoprop salt, 150 + 250 g/l 8.0 l formulated product/ha.

Reference C. Ioxynil + mecoprop ester, 110 + 440 g/l 3.0 l formulated product/ha.

Reference D. Mecoprop amine + MCPA + 2,4-D + picloram, 440 + 60 + 60 + 4 g/l 4 l formulated product/ha.

Reference E. Bromofenoxim + terbuthylazine, 310 + 190 g/l 1.7 l formulated product/ha.

Assessments

All trials were visually assessed utilizing the Barratt and Horsfall (1945) rating system and the values converted to percentages. Crop injury observations were made 7-15 and 20-30 days after application. Crop vigour and weed control were assessed 20-30 and 40-50 days after application. The trials were also monitored for ear injury following emergence thereof. Standard deviations were calculated from the mean percentage values from the French trials for both injury and herbicidal efficacy.

RESULTS

Selectivity

Similar results were obtained from trials in all countries. The data derived from the trials in France illustrate the excellent selectivity of EL-5591.

Table 1 shows the mean crop injury, manifested as discrete necrotic areas on the leaves, observed in France 7-15 and 20-30 days after application of EL-5591 and 4 reference products.

Table 1

Mean percentage leaf area damaged in trials in France, 1980

Treatment	Rate/ha	Winter Wheat		Winter Barley		Spring Barley	
		Spraying/assessment interval (d)					
		7-15	20-30	7-15	20-30	7-15	20-30
EL-5591	4 kg	2.2(1.9)	0.5(0.9)	1.2(1.5)	0.2(0.7)	2.4(2.5)	0.5(1.0)
	5 kg	2.6(2.2)	0.7(1.3)	2.1(3.0)	0.3(0.8)	3.8(3.4)	0.7(1.5)
Ref. A	2.5 l	9.1(7.7)	3.1(4.6)	6.7(8.3)	1.7(2.9)	3.3(2.6)	1.3(3.0)
Ref. B	8.0 l	5.7(8.1)	0.7(1.4)	5.3(8.9)	0.3(0.8)	7.9(14.7)	0.2(0.5)
Ref. C	3.0 l	1.4(2.0)	0.4(0.9)	0.8(1.4)	0.2(0.5)	0.3(0.6)	0(-)
Ref. D	4.0 l	0.7(1.1)	0.7(1.8)	0.8(1.4)	0.2(0.7)	0(-)	0.2(0.5)
Number of trials from which data were derived		64	41	43	35	11	5

Numbers in parentheses are standard deviations

It is apparent that all applications can cause initial crop scorch to the 3 crops tested.

However, there was considerable variability, evidenced by the high standard deviations, in the degree of injury between trials as well as between treatments. In particular, the % leaf area damaged in winter wheat by dinoterb plus mecoprop (Ref. B) varied, between trials, from 0 to 37.5% when assessment was made 7-15 days after application. It was also apparent that during the subsequent 1 to 2 weeks the injury was outgrown in all treatments. No differences in response were detected between the cultivars employed in the trials.

No treatment related effects were noted on crop-vigour or on the developing ear following application of EL-5591 or reference products.

Herbicidal efficacy

Table 2 lists the species controlled by EL-5591. For each species, the number of trials from which a mean control level of between 80 and 100% was derived is also presented. The trials are arranged according to the minimum application rate required to provide this mean control level.

It is apparent that most of the species listed were susceptible to a dose rate of 4.0 kg product/ha. However, in the same trials higher levels of control were obtained with 5.0 kg product/ha.

Table 2

Number of trials in which 4.0 or 5.0 kg product/ha were required to achieve 80-100% control

	Dose rate (kg/ha)			Dose rate (kg/ha)	
Species	4.0	5.0	Species	4.0	5.0
Adonis aestivalis	1	1	Polygonum aviculare	5	8
Anagallis arvensis	3	0	Polygonum convolvulus	7	0
Arabidopsis thaliana	1	8	Polygonum persicaria	2	0
Atriplex patula	0	4	Ranunculus arvensis	6	0
Capsella bursa-pastoris	9	0	Ranunculus ficaria	1	0
Cardamine hirsuta	1	0	Raphanus raphanistrum	12	0
Carduus nutans	4	0	Rumex acetosella	2	0
Cerastium glomeratum	9	2	Scandix pecten-veneris	0	6
Chenopodium album	6	0	Sinapis arvensis	4	4
Fumaria officinalis	5	3	Spergula arvensis	4	0
Galeopsis tetrahit	3	0	Stellaria media	25	26
Galium aparine	31	0	Valerianella locusta	2	0
Juncus bufonius	2	0	Valerianella rimosa	4	0
Lamium amplexicaule	10	0	Veronica agrestis	1	0
Lamium purpureum	5	0	Veronica hederifolia	43	0
Lapsana communis	3	0	Veronica persica	8	0
Legousia speculum-veneris	2	0	Veronica spp.	17	0
Lithospermum arvense	0	4	Vicia spp.	6	0
Papaver rhoeas	13	25	Viola spp.	12	23

With certain species, namely: Fumaria officinalis, Papaver rhoeas, Polygonum aviculare, Sinapis arvensis, Stellaria media and Viola spp., the rate required to achieve 80-100% control varied between 4.0 and 5.0 kg product/ha. For these species the size of plants and climatic conditions at application were critical and a rate of 5.0 kg product/ha ensured acceptable control.

The mean percentage weed control of the most common species in French trials following application of EL-5591 at 4.0 and 5.0 kg product/ha and 4 reference products is presented in Table 3. Excellent control of Veronica hederifolia, the most frequent species in French trials was consistently obtained at both 4.0 and 5.0 kg/ha and the level of control was superior to that obtained from the reference products. Viola spp were well controlled at the higher dosage, with again, poor results apparent from the reference products. Comparable control of Papaver rhoeas and Stellaria media was observed with 5.0 kg/ha EL-5591 and the references. Response of Polygonum aviculare to EL-5591 was variable, with EL-5591 providing similar control to ioxynil plus bromoxynil plus mecoprop (reference A) and dinoterb plus mecoprop (reference B) but lower control than the other references.

Aphanes arvensis was not well controlled with EL-5591 and control of Matricaria spp in contrast to the results from trials in 1978, was generally lower than that of the reference products although severe stunting of the plants and scorching of the foliage did occur.

Table 3

Mean percentage weed control of the most common species in winter and spring cereal trials, France 1980

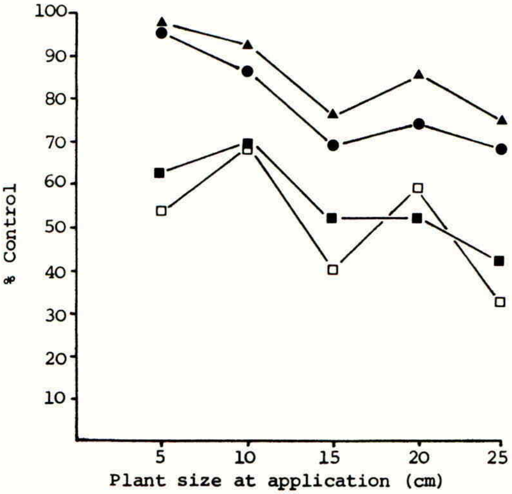
	Rate/ ha	Aphanes arvensis	Galium aparine	Matricaria spp	Papaver rhoeas	Polygonum aviculare	Stellaria media	Veronica hederifolia	Viola spp
EL-5591	4.0 kg	43(40.0)	82(24.9)	53(27.5)	79(24.1)	68(34.0)	77(30.5)	81(27.2)	73(32.3)
	5.0 kg	44(37.1)	93(7.1)	60(28.3)	92(6.6)	74(36.8)	82(22.7)	84(24.8)	82(28.0)
Ref. A	2.5 l	71(34.8)	80(24.0)	82(24.9)	91(14.1)	79(27.2)	72(30.4)	62(37.1)	49(38.2)
Ref. B	8.0 l	76(35.8)	77(24.3)	71(34.8)	93(19.1)	63(36.4)	88(15.4)	61(39.0)*	31(31.1)
Ref. C	3.0 l	71(33.4)	79(16.5)	69(31.0)	81(27.5)	85(13.2)	83(20.4)	58(38.9)*	43(35.1)
Ref. D	4.0 l	55(29.9)	83(23.1)	87(15.5)	94(5.7)	94(4.9)	81(23.8)	40(36.5)	42(35.0)
Number of trials		9	15	27	14	9	16	32	22

* values derived from 26 trials
Numbers in parentheses are standard deviations

In general, early applications of EL-5591 showed higher levels of control i.e. when the weeds were at the seedling stage. However, control of Galium aparine was excellent irrespective of size. (Fig. 1)

Figure 1

Effect of size at time of application on control of Galium aparine by EL-5591 at 4.0 kg/ha (●), 5.0 kg/ha (▲), reference A (■) and reference D (□) in 15 trials in France 1980



At the smallest size (5 cm) a similar effect was observed at 4.0 and 5.0 kg product/ha, although applications of 5.0 kg product/ha provided better control of the larger plants. A considerable decrease in control of Galium aparine was apparent with both reference products when the size at application increased from 10 to 25 cm. The recommended application rate of ioxynil plus bromoxynil plus mecoprop increases with increase in seedling size which would probably offset the decrease in control observed.

The high activity of early applications was demonstrated in a U.K. trial where autumn applications of EL-5591 were made to winter wheat (Table 4).

Table 4

Mean percentage weed control following autumn applications in winter wheat, U.K. 1979/80

	Rate/ha	Capsella bursa-pastoris	Lamium purpureum	Matricaria spp	Papaver rhoeas	Stellaria media	Veronica spp	Viola arvensis
EL-5591	3.5 kg	98.2b	98.8a	77.7b	95.3a	93.4b	98.8ab	88.8a
	4.0 kg	100.0a	99.4a	97.6a	100.0a	98.2ab	100.0a	99.4a
	5.0 kg	100.0a	100.0a	97.6a	99.4a	100.0a	100.0a	98.8a
Ref. A	3.5 l	99.4ab	98.2a	98.2a	98.8a	99.4a	94.4b	97.6a
Ref. E	1.7 l	100.0a	100.0a	97.6a	99.4a	100.0a	100.0a	96.7a

Numbers, in columns, followed by same letter not significantly different at P = 0.05

Excellent control of all species present, including Matricaria spp, was apparent with rates of 4.0 and 5.0 kg product/ha.

DISCUSSION

The data reported here from trials carried out throughout Europe show that the combination of bifenox and mecoprop (15 + 37)% wettable powder at 4.0 or 5.0 kg/ha provides selective control of a wide range of broadleaf weeds in cereal crops. In common with other phenoxy-based, post-emergence herbicides there was some foliar scorch which was rapidly outgrown. Bifenox alone was shown to cause severe foliar scorch when applied to cereals as an emulsifiable concentrate. (Richardson & Dean, 1973). Formulation as a wettable powder has improved the selectivity of the combination considerably and development of alternative formulations is continuing. No effects on crop vigour or ear development were noted for a wide range of application times. In order to confirm the results obtained in 1979, yield data will be obtained from a number of 1980 trials.

Excellent control of overwintered *Veronica* spp, *Viola* spp and *Galium aparine* was demonstrated throughout the trial programme with greater efficacy apparent against all species following early applications. In particular, autumn application of EL-5591 provided excellent weed control and for this use 4.0 kg product/ha appeared to be the optimum dose rate.

It was also noted that early spring applications of EL-5591 in Italian and French trials controlled *Polygonum* spp germinating subsequently. This was also confirmed in glasshouse tests where pre-emergence activity from surface applications of EL-5591 was demonstrated against a number of broadleaf weeds. (Glasgow *et al*., unpublished).

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