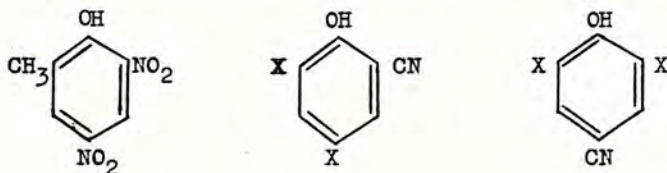


## IOXYNIL - A NEW SELECTIVE HERBICIDE

R. L. Wain  
Wye College, University of London

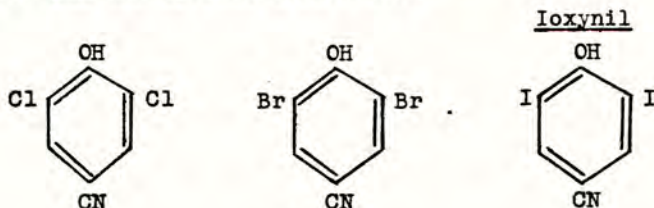
Since the herbicidal properties of 3:5-diiodo-4-hydroxybenzotrile were first discovered at Wye in 1959 it was considered appropriate that I should introduce this interesting new herbicide to this Conference. In a paper published in Nature (Wain, 1963) I outlined the thoughts which led to its preparation and examination as a herbicide and proposed for it the shorter name ioxynil. This name has now been generally adopted.

In a study on molecular compound formation carried out with Professor G.M. Bennett, F.R.S. some 20 years ago, we were able to establish that the nitro group ( $\text{NO}_2$ ) and the cyano group (CN) substituted into the benzene ring, exerted similar effects (Bennett and Wain, 1936). These groups indeed are both electron attracting groups of similar magnitude and they have similar dipole moments. Now, dinitro-ortho-cresol (DNC), well known in the field of crop protection, is highly toxic to a wide range of living organisms, including plants and this compound possesses two nitro groups. Now if CN can replace  $\text{NO}_2$  for the formation of molecular compounds what will be the effect on biological activity of replacing the nitro groups in DNC by -CN? To test out these ideas, derivatives of salicylnitrile and 4-hydroxybenzoic acid, as follows, were prepared:



D. N. C.

The first really interesting compound prepared in this investigation was 3:5-dibromo-4-hydroxybenzotrile which was found to be highly active as a contact herbicide. The corresponding 3:5-dichloro derivative was then made and found to be much less active. The diiodo compound (ioxynil), however, was more active than the dibromo derivative.



Since 1959 when these experiments were carried out, a large number of similar compounds have been prepared and examined at Wye. These include the mono halogen derivatives and compounds with other substituents replacing the -CN group. None of these, however, has been found to show the high herbicidal properties of ioxynil. We have investigated the behaviour of ioxynil within the plant and in the soil. I shall be giving a separate account at this Conference on some of our other studies which might be important in relation to its mode of herbicidal action. It is of interest here to note that one such line of investigation led logically to the examination of ioxynil as a molluscicide and it was found to be markedly active in this respect (Wain, 1963). Over the last five years we have carried out many pot experiments and field trials on the herbicidal activity of ioxynil and have investigated its possible use for the destruction of potato haulm. Similar investigations have been made by Carpenter and Heywood (1963) of May & Baker Ltd. and workers at Amchem Products Inc. (1963) who independently discovered the herbicidal properties of ioxynil in their screening programmes.

The compound has also been examined as a herbicide by Holly and Holroyd (1963) of the Weed Research Organisation. Other speakers will deal in some detail with the field performance of ioxynil.

It kills a wide range of weed species including mayweed (*Matricara matricarioides*) which is not easily controlled with phenoxy acids and, from all studies made to date, there is general agreement that ioxynil is an excellent post-emergence herbicide especially for use in cereal crops. The compound is likely to achieve considerable agricultural importance.

#### References

- AMCHEM PRODUCTS INC. (1963). Technical Service Data Sheet.
- BENNETT, G.M. and WAIN, R.L. (1936). Organic molecular compounds, Part II. J. Chem. Soc., p.1108.
- CARPENTER, K. and HEYWOOD, B.J. (1963). Herbicidal action of 3:5-dihalogeno-4-hydroxybenzotrile, Nature, 200, 28.
- HOLLY, K. and HOLROYD, J. (1963). Weed Research Org. Oxford, Tech. Rept. No.2.
- WAIN, R.L. (1963). 3:5-Dihalogeno-4-hydroxybenzotriles: New Herbicides with Molluscicidal Activity. Nature, 200, 28.

## DISCOVERY OF IOXYNIL AND ITS DEVELOPMENT IN THE UNITED STATES

R. D. Hart, J. R. Bishop, and A. R. Cooke  
Amchem Products, Inc., Ambler, Pennsylvania, U.S.A.

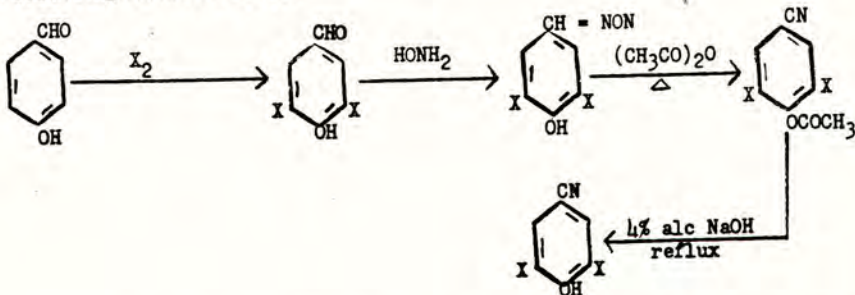
Summary In 1960, personnel of Amchem Products, Inc., discovered the herbicidal activity of 3,5-diiodo-4-hydroxybenzoxynitrile (ioxynil). This material exhibited selective post-emergence broadleaf weed control activity in cereals. A number of analogs of ioxynil were synthesized and tested, but only ioxynil and the bromo analog appeared to have practical value.

Field tests throughout the United States showed that a number of crops, including wheat, corn, rye, barley, oats and sorghum, have outstanding tolerance of ioxynil at rates which would control annual broadleaf weeds. Stage of growth appears to be important in regard to crop susceptibility and weed control. Other factors affecting performance of ioxynil are spray volume, temperature, rainfall and formulation.

Studies on the mode of action of ioxynil indicate that it is both a phosphorylation uncoupler and a photosynthetic inhibitor. Ioxynil does not move to any extent from treated areas on the foliage but may be taken up through the roots. Labeled ioxynil appears to be metabolized and re-incorporated into the starch, gluten and glucose fractions of the mature grain.

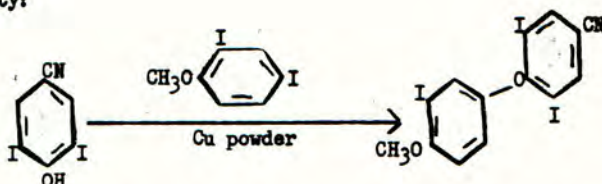
### HISTORY

Halogenated-4-hydroxybenzoxynitriles were first synthesized in Germany by Auwers and Reis (1896). They prepared the iodo, bromo, and chloro analogs by the following chemical route:



Schering-Kahlbaum Ag. (1929) referred to the preparation of ioxynil in conjunction with the synthesis of compounds related to halogen-substituted thyroxines. These compounds were prepared as follows and were tested for

thyroxine activity:



In December of 1960, Amchem evaluated as a herbicide 3,5-diiodo-4-hydroxybenzotrifluoride. It exhibited selective herbicidal activity both in a primary pre-emergence screening test at 16 lb/ac and in a primary post-emergence screening test at 8 lb/ac (Table 1). In subsequent greenhouse tests and a field test during early 1961, 3,5-diiodo-4-hydroxybenzotrifluoride continued to appear of interest as a herbicide particularly for post-emergence broadleaf weed control in cereals. Pre-emergence performance has not appeared significantly promising in early tests.

Table 1

Herbicidal activity of ioxynil in primary screening test

Test plant	Pre-emergence 16 lb/ac	Post-emergence 8 lb/ac
Alfalfa	88% control	100% control
Corn	0	63
<i>Avena fatua</i> (wild oats)	0	63
<i>Bromus tectorum</i> (downy brome)	25	75
<i>Setaria faberii</i> (foxtail)	25	25
<i>Echinochloa crusgalli</i> (barnyard grass)	25	100
<i>Digitaria sanguinalis</i> (crabgrass)	75	100
<i>Sorghum halepense</i> (Johnson grass)	25	50
Snapbeans	0	75
<i>Barbarea vulgaris</i> (yellow rocket)	100	100
<i>Stellaria media</i> (chickweed)	100	100
Soybeans	0	88
<i>Amaranthus retroflexus</i> (pigweed)	100	100
<i>Abutilon theophrasti</i> (velvet leaf)	100	100
<i>Chenopodium album</i> (lambsquarters)	100	100
<i>Rumex crispus</i> (curled dock)	100	100

An extensive program of synthesizing and testing analogs, derivatives, and related compounds was carried out during the summer and fall of 1961. This work revealed several compounds with appreciable herbicidal activity (Table 2). Besides selective weed control in crops, preliminary evaluations of the more active compounds were made on aquatic, perennial, and woody species. Investigations on the latter, especially, are being continued. Based on biological

and economic considerations, 3,5-dibromo-4-hydroxybenzotrile and the original 3,5-diiodo-4-hydroxybenzotrile appeared to have the most practical value.

Table 2

Summary of greenhouse comparison of the more active halogenated-4-hydroxybenzotriles

Test plant	Percent control - post-emergence treatment at 2 lb/ac				
	3-chloro	3,5-dichloro	3-bromo	3,5-dibromo	3,5-diiodo
<u>Alfalfa</u>	10	10	30	20	90
<u>Corn</u>	0	10	20	10	50
<u>Avena fatua</u>	0	10	50	20	50
<u>Bromus tectorum</u>	0	20	50	40	60
<u>Setaria faberii</u>	10	0	10	0	20
<u>Echinochloa crusgalli</u>	0	20	80	80	80
<u>Digitaria sanguinalis</u>	90	90	80	70	90
<u>Sorghum halepense</u>	0	20	30	10	30
<u>Snapbeans</u>	20	80	90	90	80
<u>Barbarea vulgaris</u>	100	100	100	100	100
<u>Stellaria media</u>	50	70	100	100	100
<u>Soybeans</u>	10	90	90	90	80
<u>Abutilon theophrasti</u>	100	100	100	100	100
<u>Chenopodium album</u>	90	90	100	100	100
<u>Rumex crispus</u>	50	90	100	100	100

Several pounds of 3,5-diiodo-4-hydroxybenzotrile and lesser amounts of other related compounds were synthesized during the winter of 1961. Preliminary formulation work was also undertaken and during March of 1962 the first samples were sent to Amchem licensees and various government cooperators throughout the world for herbicidal evaluation. Many tests, consisting of several hundred plots, were set up in the United States, Canada, England, Netherland, Denmark, Sweden, France, Japan and New Zealand between March and June of 1962. Ioxynil was tested for weed control in crops including wheat, barley, oats, rice, sorghum, corn, onions, flax, peanuts, alfalfa, and others. Most of this work was carried out with Amchem's formulation 62-70 (an emulsifiable oil soluble amine of ioxynil) but some tests also included Amchem's 62-177 (a 50% wettable powder of the free phenol), Amchem's 62-247 (a water soluble amine), and Amchem's 62-248 (an emulsifiable free phenol). More recently, formulations of sodium and lithium salts and of esters, alone and combined with wetting agents, have been developed.

Based on the encouraging results from these preliminary tests throughout the world, from 1962 until the present Amchem has carried out an extensive world-wide testing and development program on 3,5-diiodo-4-hydroxybenzotrile.

FIELD PERFORMANCE

Weeds controlled

Observation of field plots established throughout the United States in 1962, 1963, and 1964 has provided data on the susceptibility of many weed species (Anchem (1963)). Susceptibility of some of the more important weeds to ioxynil is listed in Table 3.

Table 3

Rate at which various weeds and crops are susceptible to ioxynil  
("Susceptible" = at least 80% of plants in plot killed)

Weeds	Common name	Pounds a.i. per acre			
		1-3	1-1	1-2	over 2
<u>Amaranthus retroflexus</u>	Pigweed	x			
<u>Ambrosia artemisiifolia</u>	Ragweed		x		
<u>Amsinckia spp.</u>	Kiddleneck	x			
<u>Anthemis arvensis</u>	Corn chamomile	x			
<u>A. cotula</u>	Dog fennel		x		
<u>Barbarea vulgaris</u>	Yellow rocket		x		
<u>Brassica spp.</u>	Mustard		x		
<u>Capsella bursa-pastoris</u>	Shepherds purse	x			
<u>Chenopodium album</u>	Lambsquarters		x		
<u>Cirsium arvense</u>	Canada thistle				x topkill
<u>Equisetum arvense</u>	Horsetail				xx topkill
<u>Fagopyrum tataricum</u>	Tartary buckwheat			x	
<u>Kochia scoparia</u>	Kochia	x			
<u>Lithospermum arvense</u>	Corn groomwell	x			
<u>L. officinale</u>	Groomwell		x		
<u>Matricaria chamomilla</u>	Wild chamomile	x			
<u>M. matricarioides</u>	Pineapple weed	x			
<u>Polygonum aviculare</u>	Prostrate knotweed		x		
<u>P. convolvulus</u>	Wild buckwheat		x		
<u>P. pennsylvanicum</u>	Smartweed		x		
<u>P. persicaria</u>	Ladythumb		x		
<u>P. scabrum</u>	Green smartweed			x	
<u>Portulaca oleracea</u>	Purslane		x		
<u>Ranunculus spp.</u>	Buttercup spp.			x	
<u>Salsola kali</u>	Russian thistle		x		
<u>Saponaria vaccaria</u>	Cow cockle		x		
<u>Scleranthus annuus</u>	Knawel	x			
<u>Senecio vulgaris</u>	Groundsel	x			
<u>Stellaria media</u>	Common chickweed	x			
<u>Thlaspi arvense</u>	Field pennycress	x			
<u>Urtica urens</u>	Nettle, annual		x		
<u>Veronica arvensis</u>	Corn speedwell	x			
<u>Xanthium spinosum</u>	Spiny clot burr		x		

## Response

Field plots throughout the United States have indicated that a number of crops including wheat, corn, rye, barley, oats, and sorghum have outstanding tolerance of ioxynil at rates which will control most annual broadleaf weeds. The tolerance of other species varies with stage of growth. For example, Fertig (1964) found that alfalfa is very susceptible to rates as low as  $\frac{1}{4}$  pound per acre in the 2- to 4-leaf stage of growth, but will survive  $\frac{1}{2}$  pound per acre rates at the 6-leaf stage and larger with only slight to moderate burning.

Volume of spray has affected crop response significantly, as indicated in Table 4. Also, the use of surfactants has increased the response of some weeds and may possibly affect crop response.

Table 4

Kill with 1/2 lb/ac ioxynil applied post-emergence in different volumes of water

Test plant	Volume of water per acre					
	100 gal	50 gal	25 gal	12.5 gal	6.25 gal	3.12 gal
Onions	95%	95%	60%	50%	40%	10%
<u>Polygonum pensylvanicum</u>	95	90	90	80	90	70
<u>Echinochloa crusgalli</u>	50	40	20	10	0	0
<u>Kochia scoparia</u>	90	90	80	70	70	60
<u>Amaranthus retroflexus</u>	100	100	100	100	100	80
<u>Bromus tectorum</u>	20	10	0	0	0	0
<u>Brassica sp.</u>	100	100	95	100	90	80
<u>Ipomea sp. (morning glory)</u>	95	100	100	100	90	80

Stage of growth is probably the most important factor affecting the control of weeds and the tolerance of crops. Table 5 summarized from work by Foy (1964) in California shows this effect on Amsinckia control in barley.

Table 5

Effects of ioxynil on Amsinckia sp. and barley treated at several stages of development

Stage of growth		Control*	Barley yield as % of check
Barley	<u>Amsinckia</u> spp.		
3-4 leaf	cotyledon	79%	104
5-8 leaf	1-4 inches	86	117
14 inches (tillered)	15 inches	28	83

\* average of  $\frac{1}{4}$ ,  $\frac{1}{2}$ , 1 pound per acre rates

Tests conducted on the Amchem Research Farm indicate that ioxynil performance is slightly better at higher temperatures. Stage of growth has appeared to be far more important, but factors such as temperature, light, and others affecting rate of growth are closely interrelated and more investigations are planned to clarify their effects.

Rain soon after application has appeared to reduce the herbicidal activity of ioxynil formulations in varying degrees. Tests at Amchem Research Farm showed that the oil soluble amine was less likely to be washed off and therefore more effective when rain followed treatment. In one test, two greenhouse flats of representative plants were treated post-emergence at  $\frac{1}{4}$  lb/ac ioxynil as the sodium salt and then one-half hour after treatment one flat was set outside in the rain (Table 6). During  $2\frac{1}{2}$  hours' exposure approximately  $\frac{1}{2}$ " of rain fell. In a subsequent test, flats treated with an oil soluble amine formulation were compared to the sodium salt by application of  $\frac{1}{2}$ " of water with a sprinkling can one hour after treatments (Table 7). Addition of a "sticker spreader" (Plyac) did not appear to reduce the loss of herbicidally active sodium salt exposed to rain in still another test.

Table 6

Effect of rain 1/2 hour after 1/4 lb/ac ioxynil treatment

Test plant	No rain	$\frac{1}{2}$ " rain
<u>Rumex crispus</u>	70% control	0% control
<u>Barbarea vulgaris</u>	70	0
<u>Stellaria media</u>	65	0
<u>Amaranthus retroflexus</u>	100	0
<u>Abutilon theophrasti</u>	35	0
<u>Chenopodium album</u>	100	0

Table 7

Formulation response to 1/2 inch simulated rain one hour after 1/4 lb/ac ioxynil treatment

Test plant	Oil soluble amine	Sodium salt
<u>Rumex crispus</u>	100% control	100% control
<u>Barbarea vulgaris</u>	80	30
<u>Stellaria media</u>	50	0
<u>Amaranthus retroflexus</u>	90	60
<u>Abutilon theophrasti</u>	70	30
<u>Chenopodium album</u>	100	90



#### MODE OF ACTION

Ioxynil is a very potent respiration inhibitor. Foy (1964) found that it decreases O<sub>2</sub> uptake in mitochondrial preparations when either alpha-ketoglutarate or succinate is used as the substrate. Preliminary studies by vanOorschot (1964) on intact plants show that both CO<sub>2</sub> uptake and transpiration are inhibited. It thus appears that the compound acts both as a phosphorylation uncoupler and as a photosynthetic inhibitor.

Using C<sup>14</sup> labelled ioxynil, Foy (1964) showed little movement from the treated area in barley and Amsinckia. This correlates with field studies which indicate that volume of spray, and hence coverage, affects crop response significantly. Using this same labelled material, Foy found little volatility of ioxynil at 25° C. In Amchem tests there was no biological volatility of ioxynil as measured by tomato response in a closed system with the free phenol at approximately 50° C.

#### RESIDUE

To determine the fate of ioxynil in wheat, Amchem conducted greenhouse tracer studies with C<sup>14</sup> ring labelled material. By harvest time, all radioactivity in the mature wheat grain appeared to be in starch, gluten, and glucose fractions. To determine the true residue picture of ioxynil under normal environmental conditions, an extensive residue sampling program has been conducted during 1964. Both wheat and barley treated with various rates of ioxynil have been collected from 24 locations for analysis.

#### REFERENCES

- AMCHEM PRODUCTS, INC. (1963) 3,5-diiodo-4-hydroxybenzoxonitrile. Tech. Service Sheet H-90. 24 p.
- AUWERS, K., & J. REIS (1896) Uber einige neue derivitate des p-oxybenzaldehydes, des p-cyanphenols und der p-oxybenzoesaure. Ber. Deut. Chem. Ges. 29:2355-2360.
- FERTIG, S. N. (1964) Unpublished data. (Cornell Univ.).
- FOY, C. L. (1964) Ioxynil - a new weed killer for use in cereal grains. Proc. Calif. Weed Conf. 16:90-98.
- OORSCHOT, J. L. P. VAN (1964) Unpublished data (Inst. Biol. en Scheikundig, Wageningen).
- SCHERING-KAHLBAUM AG. (1929) German patent 467,639.

A SUMMARY OF THE CHEMICAL AND BIOLOGICAL PROPERTIES OF  
IOXNYL AND BROMOXNYL

B.J. Heywood, K. Carpenter and Helen J. Cottrell  
Research Laboratories, May & Baker Ltd., Dagenham, Essex  
and  
Agricultural Research Station, May & Baker Ltd., Ongar, Essex

Synopsis. The chemical, physical and biological properties of ioxnyl and bromoxnyl as their alkali metal salts and n-octanoyl esters are compared and some of the factors influencing the herbicidal efficiency are briefly discussed.

The overall level of herbicidal activity of the two phenol salts is similar but there are important differences in species susceptibility which will influence the use of one or other in particular circumstances. In general, bromoxnyl octanoate is more active than the potassium salt on almost all species on which they have been compared, whereas ioxnyl octanoate is rarely more active than the salt and frequently less active. The reasons for this may well be linked with the physical and chemical properties.

The environmental factors which appear to have most influence on the activity of ioxnyl are light intensity, temperature, and the state of development of the plant. So far the relative importance of the contact and systemic effects cannot be disentangled.

INTRODUCTION

Since the original publications by Carpenter & Heywood (1963) more detailed information has been reported by May & Baker workers about the chemical and biological properties of ioxnyl and bromoxnyl (Carpenter, et al 1964a), the field activity (Terry & Wilson, 1964), and the herbicidal activity of related halohydroxybenzonnitriles (Carpenter et al 1964b). Technical bulletins on ioxnyl have also been published in the United Kingdom by the Weed Research Organisation (Holly & Holroyd 1963) and in the United States by Amchem workers (Amchem Technical Data 1963), and progress reports of herbicidal trials have also been made at European and American Weed Control conferences.

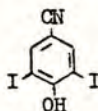
The present paper outlines some basic facts about the chemical and herbicidal properties, toxicology, residues and effects on wild life, of formulations of bromoxnyl and ioxnyl, which have been collected during the preliminary field assessment in 1963 and the more extensive evaluations during 1964 in a variety of different situations. This information includes some new material about the properties and activity of the n-octanoyl esters which have been widely tested during 1964, both in the U.K., North America and Australia. Detailed results of trials with the octanoyl esters are

being reported to the East (Cook, 1964) and the West (Clarke and Cook, 1964) Canadian Weed Control Conferences since much of the work on bromoxynil ester has been done in Canada.

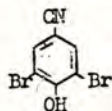
The practical use of these new herbicides in Britain (both alone and in mixtures with phenoxy alkanolic acids) in cereals will be discussed in a later session of the Conference by Terry (Session III) and by Soper (Session III) whilst the possibility of use in forage legumes will be discussed by Ball *et al* (Session IV).

#### PHYSICAL AND CHEMICAL PROPERTIES

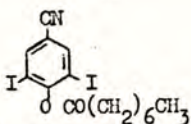
The chemical structures and names of the new herbicides are:-



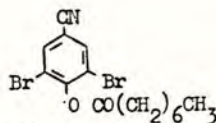
ioxynil I&EB 8873



bromoxynil I&EB 10064



ioxynil octanoate I&EB 11641



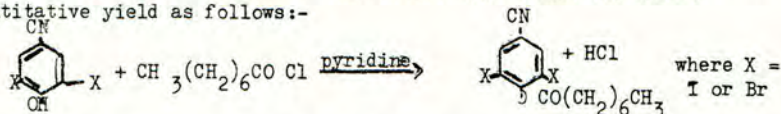
bromoxynil octanoate I&EB 10731

Both ioxynil and bromoxynil are high melting crystalline solids, with low solubility in water and only sparing solubility in most organic solvents. Unlike the nitrophenols, dinoseb and D&OC, ioxynil and bromoxynil and the alkali metal salts are colourless. They are comparatively stable compounds, but can be hydrolysed by treatment with strong aqueous alkalis and mineral acids, to the amide and acid. Also the halogen atoms are fairly firmly attached to the ring, except that under oxidising conditions iodine is liberated from ioxynil in the same way as with most *o*-iodophenols.

The sparing solubility of these compounds precludes the possibility of making concentrated solution formulations. However, phenols are acidic in character and these *p*-cyanophenols are no exception. On the contrary the presence of the electronegative cyano group para to the hydroxy group associated with the two halogen atoms in the ortho positions make ioxynil and bromoxynil fairly strong organic acids with *pK* of about 4.0 compared with phenol of 9.9. It is thus no surprise that both these herbicides form well defined salts with alkali metals which give neutral solutions in water. The ammonium and simple amine salts are also crystalline solids but with low solubility in water. The solubility of the alkali metal salts in water (4-16% w/v as phenol) is insufficient to provide commercially acceptable liquid concentrate. This difficulty can be overcome by the incorporation of suitable organic solvents. These solutions are stable to both high and low temperature storage. The

incorporation of salts of certain other weedkillers such as MCPA or mecoprop can be accomplished and some results with these formulations are described in later papers of this Conference. As the solubility of the calcium and magnesium salts of ioxynil and bromoxynil in water is appreciable, these formulations do not form precipitates even with very hard waters (up to 2000 ppm as CaCO<sub>3</sub>).

An alternative way of providing suitable formulations is by converting the phenols into esters. Since the esters are now different compounds it is not surprising that the range of weeds controlled is a little different (see herbicide section below). The esters from ioxynil and bromoxynil and octanoic acid have been selected for special study. Unlike the parent phenols, the esters derived from the higher fatty acids are low melting solids that are very soluble in mineral and aromatic oils, and so offer little difficulty in devising satisfactory formulations. Esters from acetic acid are not very different from the phenol in that they are still high melting solids with only sparing solubility in organic solvents. The esters can be made in almost quantitative yield as follows:-



Acid anhydrides can be used instead of acid chlorides. It should be noted that these esters have some of the properties of anhydrides since they are derived from two acidic components. While the salts and esters each have their appropriate place as weedkillers, it is worth noting in passing that the esters have a lower toxicity to mice in the acute oral test. Table 1 summarises some of the properties of these compounds.

#### RESIDUE STUDIES

There are many features concerning the ioxynil and bromoxynil molecules which facilitate detection even in the presence of plant material. Thus the moderately intense absorption band at 4.5 μ in the infra-red due to the C ≡ N stretching mode is particularly useful. Since acidic aromatic nitriles are not known in plants it has been possible to devise an extraction procedure that removes interfering plant material so that 0.1 ppm can be detected in a 200 g. sample. Most of residue data has been obtained using this method. To establish a correction factor for recoveries under a variety of conditions, radio-actively labelled material has been added to all samples examined by this method so that there is available data not only on the amount of residue present at a particular time but also in the recovery. Future residue analytical work will not require the use of labelled material.

Other methods for ioxynil residues that have been developed depend on the conversion of the phenol into the methyl ether using diazomethane. These ethers give a very good separation from plant debris by gas chromatography and since iodine containing compounds give a very intense

Table 1

Properties of ioxynil and bromoxynil and the esters

	Ioxynil		Bromoxynil	
	Phenol	Octanoyl ester	Phenol	Octanoyl ester
Appearance	White, crisp solid	Cream, waxy solid	White, crisp solid	Cream, waxy solid
Melting point °C.	212-213.5	49-54	194-5	45-6
MW	370.9	496.9	276.9	402.9
% phenol equivalent in ester	-	74.5	-	69.5
Solubility in water	50 ppm	insol.	130 ppm	insol.
Xylene (approx.)	< 2%	> 50%	< 2%	70%
LD <sub>50</sub> rats acute oral as mg/kg	110	390	190	260
Solubility salts in water as phenol				
sodium	14.0	-	4.2	-
potassium	10.7	-	6.1	-
pK (phenol 9.9)	3.96		4.08	

signal with an electron capture detector, small samples can be used with a sensitivity of about 0.01 ppm. Alternatively the methyl ether can be separated by thin layer chromatography and the ioxynil determined by the iodine present at the relevant spot. These two latter methods, like the I.R. method, are specific for ioxynil because of the chromatographic separation and detection procedures. It should be noted that they depend on a different feature of the molecule, namely, the halogen atoms and so are complementary to, rather than duplicate, the I.R. method.

Confirmation of residues and metabolites can be made by estimating the total iodine in the treated plants. This method is effective as the natural iodine level in plants is normally low, especially in grain grown on farms away from coastal areas.

By examination of the results in some 50 experiments in the U.K. it has been found that in wheat, oats, barley and rye, whether winter or

spring sown, the residue levels fall fairly rapidly so that the limit of detection (0.1 ppm) is reached in about five weeks, even when the dose rate is approximately double that recommended. Since the application of the herbicide is early in the growing season, this limit of detection is still at least five weeks before harvest. Also there is no residue in the straw at harvest. Table 2 gives median residue levels of ioxynil at various times after spraying in cereals following an application of 12-18 oz. per acre. These figures are derived from the inspection of the 50 experiments of the rate of disappearance of ioxynil. Data from other countries shows a similar or faster rate of disappearance of ioxynil.

Table 2

Residue levels of ioxynil in cereals following application of 12-18 oz/acre

Weeks after spraying:	1	2	3	5
Residue in ppm	7	2	0.6	<0.1

#### EFFECTS OF IOXYNIL ON WILD LIFE

##### A. Wild and game birds

Three experiments were carried out in Essex under normal farming conditions in the early summer of 1964. At each site, records were made of the territories and nests of a total of 23 species of wild birds including pheasants, partridges, mallard and wood pigeons; in an area of 2-3 acres of woodland. The surrounding area of cereals, about 10 acres in extent at each site, was then sprayed with a formulation containing 6 oz. ioxynil-sodium. The whole area was then searched for dead birds, and the development of the eggs and the chicks in the nests was also observed for about 10 days after spraying but no obvious effects on the adult population or development of eggs and chicks were observed. In an additional pilot experiment, 3 week old pheasant chicks were grazed for 21 days on a young grass and clover ley, immediately after spraying with 12 oz/acre ioxynil-sodium. No harmful effects to the pheasant chicks were noted.

##### B. Beneficial insects

The toxicity of bromoxynil and ioxynil towards beneficial insects has been evaluated by two different methods. In the first, a test for direct contact activity, salts and esters were considerably less toxic to honey bees and to mustard beetles\* than dinoseb and similar to MCPA when tested at two and three times (i.e. 16 and 24 oz/acre) the recommended practical rate. In the second method, honey bees were allowed to feed freely on sugar syrup containing known concentrations of ioxynil and bromoxynil. The LD50's for both herbicides were about one third of those for MCPA, indicating a similar hazard under field conditions when allowance is made for the lower dose required for herbicidal effect.

\* indicator species for beneficial coccinellids

## HERBICIDAL ACTIVITY

### Weed Control

The greater part of our work on herbicidal activity has been concerned with levels of weed control under practical conditions. The work carried out in 1963 has already been published (Terry & Wilson 1964) and has been followed up, in 1964, with a much larger scale programme comprising upwards of 250 replicated field experiments comparing up to 7 or 8 different formulations or mixtures containing ioxynil and bromoxynil in the U.K., northern Europe, Canada, the United States and Australia. This has been reinforced in the U.K. by about 120 farm trials and in other countries by Government and University research workers. From these programmes has come a considerable body of weed control information, the detail of which is being or will be published at this Conference and elsewhere.

In this paper we shall limit ourselves to the comparison of bromoxynil and ioxynil and their alkali metal salts and octanoyl esters. Table 3 summarises the relative activities of these compounds in terms of LD<sub>50</sub> for the more important weeds on which they have been compared side by side in the field by techniques capable of producing dose response data. It is based on field experimental results from various countries, but principally the U.K. and Canada, the details of which are or will be published elsewhere. In the experiments in the U.K., Canada and Australia, exactly the same application and evaluation techniques were used and all the values given are based on low volume application 6-15 gallons per acre. The same formulations were also used throughout most of the programme and the same solvents and surface active agents in the same proportions relative to the active ingredient, were incorporated in the two ester and salt formulations respectively.

Consistent results were obtained from different countries for a given weed species in spite of climatic variations occurring between the trials. Examples were Polygonum convolvulus (U.K., Europe, Canada and Australia), Amsinkia intermedia (U.K., U.S.A. and Australia) and Galeopsis tetrahit (U.K., Europe and Canada). Probably the most interesting feature of this table is that, whereas bromoxynil octanoate is almost invariably more active than bromoxynil salt; the same is not true for ioxynil. For some species ioxynil octanoate is less active than the salt.

As comparisons between all four compounds were limited to about a quarter of the total field programme Table 3 gives only a partial indication of their relative usefulness. In general, ioxynil as the sodium salt has so far proved to be the most generally useful under European conditions, especially in combination with the phenoxyalkanoic acids, whereas bromoxynil appears to have greater promise in some, but not all, North American conditions.

### Crop tolerance

Winter and spring sown cereals have tolerated two to four times the herbicidal rate of each compound, formulated either as a salt or

Table 3

Summary of comparative herbicidal activity in field experiments in cereals

Species	Growth Stage	Dose (phenol equivalent) producing 90% kill (oz/acre)			
		Bromoxynil K	Bromoxynil ester	Ioxynil Na	Ioxynil ester
<u>Amsinkia intermedia</u>	YP	4	4	8	>12
	MP	>12	8	>12	>12
<u>Chenopodium album</u>	Sd	3	3	3-6	4
	YP	3-4	3-5	2-8	4-8
	MP	8-12	-	>12	-
<u>Fagopyrum tartaricum</u>	Sd	2	2	4	6
	YP	2	2	6	6
	MP	4	3	6	6
<u>Galeopsis tetrahit</u>	Sd	8	6	4	8
	YP	12	8	6	8-12
<u>Polygonum aviculare</u>	Sd	6-8	3	8-12	6-12
	YP	12	8-12	12	12
<u>P. convolvulus</u>	Sd	2	2	2-4	2-6
	YP	2-4	2	4-6	4-8
	MP	4-8	4	6-8	8
<u>Saponaria vaccaria</u>	YP	>12	3	>12	8
	MP	*	10	*	8
<u>Sinapis arvensis</u>	Sd	2	2	2	2
	YP	2	2	2	2
	MP	8	2-8	6-8	3
<u>Stellaria media</u>	Sd	12	-	6-8	6-8
	YP	*	*	8	8
	MP	*	*	>12	>12
<u>Thlaspi arvense</u>	Sd	5	3	3	3
	YP	8	3	8	5

- Sd Seedling cotyledon (2-3 leaves)  
 YP Young plant (3-4 leaves to early bud)  
 MP Mature plant  
 \* No kill at 12 oz.



the octanoyl ester, at all stages of growth from two leaves to the beginning of jointing (Terry and Wilson 1964), (Carpenter, et al) 1964c).

Application after jointing has tended to produce lower yields in spring cereals than earlier applications, even in weed-free crops. Oats is perhaps more sensitive than wheat and barley but is more tolerant of bromoxynil than ioxynil. Formulations of the esters have been less extensively tested, but spring wheat and barley have tolerated high rates at stages between 4 leaves and tillering.

Results of small scale field experiments done in 1964 have confirmed that bromoxynil and bromoxynil ester are tolerated by seedling lucerne, both direct seeded and undersown, and will be reported by Ball et al in a later session. Canadian trials in direct seeded lucerne at later stages of growth have also been promising. The experimental evidence on the effects on red and white clovers is less promising.

#### Action of Ioxynil

In the earlier paper (Carpenter et al 1964b) we reported that ioxynil was primarily a contact herbicide with some translocated action. Under normal field conditions, the herbicide causes necrotic and blistered areas on the leaves of susceptible species within 24 hours, and finally death. There is some variation in the speed of action, but usually the weeds are killed between 2 and 7 days after spraying. Gramineous plants are not killed but may suffer local leaf scorch when treated with high rates. Broad leaved plants with waxy cuticles show intermediate susceptibility and areas of the leaf may be scorched, but the plants soon recover.

The translocated effects which follow topical application are in general sub-lethal, and are observed as chlorosis extending beyond areas which have retained the herbicide and also on untreated leaves. Nevertheless the production of discrete necrotic areas, which might be thought to be due to 'contact' action, also occurs in detached leaves if the ends of the petiole only are immersed in solutions of the herbicide. Ioxynil has no useful effect when applied pre-emergence, although it inhibits the germination of seeds when tested in vitro.

In practice the herbicidal action of ioxynil appears to be influenced by a number of different factors which have been previously enumerated (Carpenter et al, ibid). 1. Environmental factors such as temperature and light intensity. 2. Retention and distribution of spray deposits by foliage. 3. The growth stage of the plant.

#### Environmental factors

Practical observations have indicated that climatic conditions could influence both speed of action and level of activity. Preliminary experiments have shown that ioxynil acts more slowly when the temperature is lower; and herbicidal activity is related to light intensity, so that higher rates of ioxynil were required to kill (or to reduce growth of) Stellaria plants which received 75% and 40% of full daylight. These preliminary results, together with much circumstantial evidence, suggest

that greenhouse data on both comparative and absolute activity of these herbicides must be regarded with some reserve.

#### Distribution and retention of spray deposits

We have reported that differential spray retention does not wholly account for the selectivity of ioxynil (Carpenter *et al*, *ibid*). Terry and Wilson (*ibid*) showed that the addition of a wetting agent to the spray solution resulted in better weed control, especially of Chenopodium spp. and some Polygonum spp. On the other hand, the volume of application between the limits 10 gall/acre and 65 gall/acre generally had little influence on activity.

Experiments in the greenhouse have supported the practical observations, and one extreme example is worth quoting. It was found that about four times the amount of ioxynil was required to produce a given reduction in growth of a Ragharus leaf, when it was all applied in a single droplet than when it was applied in sixteen droplets. Intermediate effects were obtained with fewer droplets, indicating a response curve.

#### Effect of growth stages of plants

The early experiments in the greenhouse and the field showed that certain weed species became more resistant to ioxynil salts with advancing age. For example, it was calculated that the dose required to kill plants of Palapathifolium or Stellaria media increased by a factor of  $1\frac{1}{2}$  for every leaf expanded, and similarly the dose for Chenopodium increased by a factor of 2. Later experiments have proved that plants of P. lapathifolium became more resistant, because the upper leaves are themselves less susceptible to damage than the lower leaves. Although each leaf tends to become less resistant as it expands and ages, the number of new leaves produced as the plant grows seems to have an over-riding effect in reducing susceptibility. Other studies on leaves have shown that some parts are more susceptible than others to ioxynil, Na. Areas from which herbicidal effects are translocated are, however, small, e.g. the midribs of Stellaria leaves, and the axils of Polygonum, but 'contact' damage was greatest in the interveinal tissue where the cells are not lignified.

#### CONCLUSIONS

Ioxynil and bromoxynil formulations control a very wide spectrum of annual weeds and are well tolerated by cereals at early growth stages and by some leguminous crops, notably lucerne. Both herbicides also present little hazard to users, wild birds and mammals, and beneficial insects; and since residues disappear very rapidly to zero level, to consumers.

Ioxynil and bromoxynil can be readily formulated as salts or esters and the differences in weed spectra selectivity possessed by the two herbicides and their derivatives provide a choice of treatment according to the local agricultural situation. Ioxynil salt formulations are of most interest in European agriculture, probably in mixtures with phenoxy alkanolic herbicides. Bromoxynil ester formulations are of greater

promise in N. America and Australia.

The activity of both is influenced by light intensity, temperature and plant development but the part played by these and the relative importance of the contact and systemic effects are not yet understood.

#### REFERENCES

- AMCHEM Technical Service Data Sheet H-90 (1963).
- BALL, R.W.E., COTTRELL, H.J. and TERRY, H.J. (1964) Preliminary experiments with ioxynil and bromoxynil in forage legumes. In press. 7th Brit. Weed Cont. Conf., Session IV.
- CARPENTER, K., COTTRELL, H.J., HEYWOOD, B.J. and LEEDS, W.G. (1964b) Herbicidal activity of hydroxyhalogenobenzonitriles. In press. XVth International Symposium on Crop Protection, Ghent.
- CARPENTER, K., COTTRELL, H.J., de SILVA, W.H., HEYWOOD, B.J., LEEDS, W.G., RIVETT, K.F. and SOUNDY, M.L. (1964a) Chemical and biological properties of two new herbicides - ioxynil and bromoxynil. Weed Res. 4, 175-195.
- CARPENTER, K. and HEYWOOD, B.J. (1963) Herbicidal action of 3:5-dihalogeno-4-hydroxybenzonitriles. Nature, Lond., 200, 28-29.
- CARPENTER, K., TERRY, H.J., COMBELLACK, J.H. and SOPER, D. (1964c). The use of ioxynil as a selective herbicide in cereals in the United Kingdom. In press, 7th Brit. Weed Cont. Conf. Session III.
- CLARKE, F.C. and COOK, P.D. (1964) In press, West Canadian Weed Control Conference.
- COOK, P.D. (1964) In press, East Canadian Weed Control Conference.
- HOLLY, K. and HOLROYD, J. (1963) 3:5-Diiodo-4-hydroxybenzonitrile; a progress report on experimental work by the A.R.C. Weed Research Organisation May-Oct., 1963. W.R.O. Tech. Report No.2.
- TERRY, H.J. and WILSON, C.W. (1964) A field study of the factors affecting the herbicidal activity of ioxynil and bromoxynil and their tolerance by cereals. Weed Res. 4, 196-215.
- WAIN, R.L. (1963) 3:5-Dihalogeno-4-hydroxybenzonitriles: new herbicides with molluscicidal activity. Nature, Lond. 200, 28.

#### ACKNOWLEDGEMENTS

The authors would like to thank the following for providing much of the data given in this paper:-

Dr. R.F. Collins, Dr. W.H. de Silva, Mr. W.G. Leeds, Miss G. Mansfield, Dr. D.F. Muggleton, Mr. K.F. Rivett and Miss M.L. Soundy.

## Research Summary

### THE GROWTH OF BACTERIA, FUNGI AND ALGAE IN THE PRESENCE OF THE 3:5 DIHALOGENO-4-HYDROXYBENZONITRILES WITH COMPARATIVE DATA FOR SUBSTITUTED ARYLOXYALKANECARBOXYLIC ACIDS.

Wm. W. Fletcher and J. E. Smith,

Department of Applied Microbiology and Biology, The University of Strathclyde, Glasgow.

It is important that any chemical being used agriculturally and which may reach the ground should not adversely affect the micro-organisms on which fertility depends. We have therefore examined the effect of ioxynil (3:5 diiodo - 4 - hydroxybenzotrile) and bromoxynil (3:5 dibromo - 4 - hydroxybenzotrile) against a range of micro-organisms (bacteria, and fungi) with this in view. It is of considerable interest from a fundamental point of view, to determine whether there are differences in reaction between the various plant groups and we have therefore included algae in our experiments; and finally we have compared the toxicity for micro-organisms of these 3:5-dihalogeno - 4 - hydroxybenzotriles with the substituted aryloxyalkane-carboxylic acids (MCPA and MCPB).

We have in all investigated the growth of some 50 fungi, 18 bacteria, and 7 micro algae in the presence of the 3:5 - dihalogeno - 4 - hydroxybenzotriles. These were grown in suitable media either with (fungi) or without (bacteria, algae) agar. Results are shown in Table 1, for a representative series of bacteria and fungi and comparative data is supplied for the substituted aryloxyalkane-carboxylic acids. (The latter data are from Shennan, Jean and Fletcher, W. W. [in press].) The values given are the minimum inhibitory concentrations of herbicide required to prevent growth of micro-organisms grown in vitro.

From results in Table I and from results with other bacteria and fungi the following may be deduced:-

- (1) Ioxynil is more toxic to micro-organisms than is bromoxynil e.g. there is no growth of Bacillus subtilis in the presence of 100 p.p.m. ioxynil whereas 1000 p.p.m. bromoxynil does not prevent the growth of this bacterium. Similarly 100 p.p.m. ioxynil will inhibit the growth of Mucor hiemalis. Inhibition of the same order requires 1000 p.p.m. of bromoxynil.
- (2) MCPB is more toxic to micro-organisms than is MCPA. e.g. 10,000 p.p.m. MCPA is required to inhibit the growth of Aerobacter aerogenes but 5000 p.p.m. MCPB will have the same effect. Aspergillus niger is completely inhibited by 500 p.p.m. of MCPB but not by a concentration of 10,000 p.p.m. MCPA. This is a phenomenon that will be discussed in detail in the paper previously mentioned but it is of interest here to note that it implies an inherent toxicity of MCPB in itself apart from its conversion to MCPA.
- (3) MCPB is in general more toxic to fungi than it is to bacteria. All five fungi shown on this table are inhibited by 500 p.p.m. MCPB whereas only one bacterium (actinomycete) is inhibited.

- (4) Although it is not immediately obvious from these results we have found that in general ioxynil and bromoxynil are more toxic to fungi than they are to bacteria. With one exception the growth of all of the bacteria that we examined were unaffected by 10 p.p.m. ioxynil whereas the growth of all of the fungi (with one exception) were adversely affected.
- (5) Ioxynil (which will probably be the most widely used of the 3:5 dihalogeno - 4 - hydroxybenzonitriles) is more toxic to micro-organisms than is MCPA (which is much more widely used than is MCPB).
- (6) Although both ioxynil and bromoxynil are fairly iphibitory to bacteria and fungi at low concentrations, these concentrations are ~~as~~ far in excess of what are likely to be found in soil since the two herbicides will normally be used at only a few ounces per acre. Furthermore, they appear to be rapidly inactivated in the soil. Neither MCPA or MCPB are likely to be toxic at the rates used in agricultural practice to micro-organisms in the soil.

In order to compare the toxicity of the 3:5 dihalogeno - 4 - hydroxybenzonitriles for the heterotrophic fungi and bacteria on one hand and autotrophic organisms on the other experiments were set up using micro-algae. The results are presented in Table 2.

Perhaps the most interesting feature to emerge from the results in Table 2 is the great variability among micro-algae to these chemicals. Chlorella ellipsoidea, for example, is completely inhibited by 10 p.p.m. ioxynil whereas Chlamydomonas globosa is unaffected by 50 p.p.m. Even closely related species react differently. C.ellipsoidea as mentioned being inhibited by 10 p.p.m. ioxynil whereas C.pyrenoidosa still shows some growth at 50 p.p.m. It is also noticeable that in general ioxynil is more toxic than bromoxynil. This agrees with our findings for bacteria and fungi and with other workers findings for higher plants. The great susceptibility of Anabaena cylindrica. to the 3:5 dihalogeno - 4 - hydroxybenzonitriles is noteworthy. Whether this is generally true for blue-green algae is not known but is worthy of investigation.

TABLE 1.

Minimum Inhibitory Concentrations of herbicides (parts per million) to prevent growth of micro-organisms.

<u>Organism</u>	<u>Bromoxynil</u>	<u>Ioxynil</u>	<u>MCPA</u>	<u>MCPB</u>
Aerobacter aerogenes	>1000	1000	10,000	5,000
Bacillus subtilis	>1000	100	5,000	2,500
Nocardia $\alpha$ type	1000	100	2,500	500
Pseudomonas fluorescens	>1000	>1000	>10,000	>10,000
Sarcina lutea	>1000	1000	10,000	5,000
Aspergillus niger	100	100	>10,000	500
Alternaria solani	>1000	1000	>10,000	500
Botrytis cinerea	100	100	>10,000	500
Mucor hiemalis	1000	100	>10,000	500
Penicillium notatum	1000	1000	>10,000	500

TABLE 2.

The effect of Ioxynil and Bromoxynil on the growth of micro-algae.

Results are expressed as percentages of the control.

The concentration of chemicals is in parts per million (p.p.m.)

	<u>Bromoxynil (ppm)</u>					<u>Ioxynil (ppm)</u>				
	10	20	30	40	50	10	20	30	40	50
Anabaena cylindrica	100	0	0	0	0	0	0	0	0	0
Ankistrodesmus convolutus	62	51	31	20	15	0	0	0	0	0
Chlamydomonas globosa	100	100	100	100	100	100	100	100	100	100
Chlorella ellipsoidea	100	100	100	100	100	0	0	0	0	0
Chlorella pyrenoidosa	55	36	35	25	25	40	31	23	19	14
Pandorina morum	100	100	100	100	100	55	44	25	19	18
Stichococcus bacillaris	91	49	11	0	0	9	0	0	0	0

Each reading is the mean of five replicates.

4-AMINO-3,5,6-TRICHLOROPICOLINIC ACID  
A NEW SYSTEMIC HERBICIDE USEFUL FOR THE CONTROL  
OF MANY WORLD WIDE VEGETATION PROBLEMS

P. M. Ritty

The Dow Chemical Company, Midland, Michigan, U.S.A.

Summary. 4-amino-3,5,6-trichloropicolinic acid is proving to be one of the most valuable herbicide tools known for the control of unwanted vegetation, for conversion of marginal lands to productive ones, and for increasing world food production.

INTRODUCTION

Through intensive research, a new highly active systemic herbicide has been discovered by The Dow Chemical Company. This chemical is 4-amino-3,5,6-trichloropicolinic acid. (Hamaker, J. W. *et al*, 1963) Formulations such as TORDON\* 22K Herbicide which contains this new chemical are proving to be valuable tools in the control of weed and brush species around the world. Many widely distributed problem plants, resistant to the phenoxy compounds are being readily controlled.

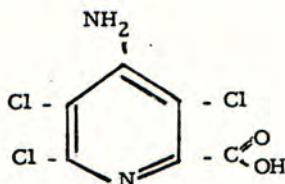
4-amino-3,5,6-trichloropicolinic acid is absorbed by both foliage and roots and readily translocated in plants. Various formulations both liquid and granular are being evaluated by private and government research organizations and by Dow scientists in the United Kingdom, Europe, U.S.A., and in many other countries.

Results, to date, show this herbicide to be exceptionally effective when applied at relatively low rates. Present and potential uses for this new herbicide are now apparent for controlling unwanted brush species on industrial sites, such as power, communication, pipeline, railroads, and on rangeland. Promising results are being obtained in selective weed control in small grain crops, sugar cane, and brassicas. (Watson, A. J. & Wiltse, M. G., 1963), (Gantz, R.L., & Laning, E.R. 1964), Laning, E.R., 1963)

\*Registered Trademark of The Dow Chemical Company

## CHEMICAL PROPERTIES

### 4-amino-3,5,6-trichloropicolinic acid



Its molecular formula is  $C_6H_3Cl_3N_2O_2$  with a molecular weight of 241.5 and it decomposes at 215 - 230°C.

In its purified physical state, 4-amino-3,5,6-trichloropicolinic acid is a white powder with a slight chlorine like odour. 4-amino-3,5,6-trichloropicolinic acid is soluble in various solvents: At 25°C, solubility is 430 ppm in water; 5,500 ppm in isopropyl alcohol; and 20,000 ppm in acetone. Oil solubility is low; for example: only 10 ppm in kerosene.

### TOXICITY

Acute and chronic toxicity tests, to date, have proven 4-amino-3,5,6-trichloropicolinic acid to be low in toxicity to humans, livestock and to wildlife. Acute oral toxicity tests have given LD<sub>50</sub> values for rabbits, mice, guinea pigs, chicks, and rats ranging from 2.0 g./kg. for rabbits to 8.2 g./kg. of body weight for rats. By comparison phenoxy compounds such as 2,4-D and 2,4,5-T have LD<sub>50</sub> ranges of 0.375 g./kg. for mice to 1.0 g./kg. of body weight for guinea pigs.

4-amino-3,5,6-trichloropicolinic acid is safe to handle and presents no serious hazard for eye and skin contact or through skin absorption.

Single oral doses and extended daily feedings of 4-amino-3,5,6-trichloropicolinic acid to sheep, yearling calves, swine, and chickens gave no evidence of ill effects, either in weight gains or feed conversion in these experiments. Results indicate no hazards exist for large animals resulting from accidental ingestion, per se, or consuming vegetation treated with 4-amino-3,5,6-trichloropicolinic acid.

Accidental contamination of stream or pond water, would not be hazardous to the fish population. Low fish toxicity has been shown by tests on numerous types of fish with median tolerance limits of 20 ppm for brown trout (Salmo trutta fario L.) and 70 ppm for brook trout -



(Salvelinus fontinalis Mitch.) (Watson, A. J. & Wiltse, M. G., 1963)

Aquatic biota such as ramshorn snails (Planorbis sp.) and daphnia (Daphnia sp.) were not affected at a concentration of 30 ppm of a. e. of 4-amino-3,5,6-trichloropicolinic acid. Some injury did occur at 40 ppm however.

Since the effects of chemicals on wildlife is a matter of serious concern, it is comforting that a chemical, which has the high biological activity of 4-amino-3,5,6-trichloropicolinic acid, presents little or no hazard to fish, game animals, and pets.

#### Soil persistence.

4-amino-3,5,6-trichloropicolinic acid retains its herbicidal activity in soils longer than 2,4-D or 2,4,5-T. It appears that, like 2,4-D and 2,4,5-T, it is easily leached under high rainfall conditions. Under non-leaching conditions in greenhouse tests, high rates of 4-amino-3,5,6-trichloropicolinic acid are very persistent.

Under field conditions, rates used for the control of noxious perennial weeds, two to three pounds per acre, have persisted into the year following treatment and highly sensitive plants have been injured when planted; however, grasses, grass crops and moderately sensitive plants can generally be grown the year following treatment without injury.

Where low rates have been used for selective weed control in crops (1/4 to 2 oz. a. e. /ac.) no residual amounts could be detected the following growing season by using highly sensitive plants in field and in laboratory bio-assay determination. The rate of loss of this herbicide from the soil occurs most rapidly at low concentrations. (Leasure, J. K., 1964)

Extensive field and laboratory experiments are being conducted throughout the world to more fully determine the fate of 4-amino-3,5,6-trichloropicolinic acid in soils.

### HERBICIDAL ACTION

#### General.

The initial herbicidal action on broadleaved plants after contact with sprays of this new herbicide, is one of leaf curling and epinasty of the more tender growth. Leaf kill is not as rapid as that observed following applications of 2,4-D and/or 2,4,5-T on woody species. 4-amino-3,5,6-trichloropicolinic acid is highly systemic and readily translocated into the roots. Reaction on the meristematic tissue is very pronounced. To obtain maximum response, however, the plants should be actively growing at the time of foliage treatment.

## Brush Control.

Used alone or in combination with phenoxy compounds, it is highly active, systemic, and effective on a wide variety of brush species. It is particularly efficient in preventing regrowth of root-collar and lateral root sprouting species.

Early research on this new herbicide was initially conducted by Dow scientists, first in the greenhouse and in a woody plant nursery, then extensively throughout North America. Various methods of application on brush were used such as leaf-stem (foliage) sprays during the growing season and cut surface treatments and injection during the dormant season. Granular applications were also made and found to be most effective when applied in spring or early summer. Soil type and rainfall patterns can affect results.

Because these exploratory experiments with 4-amino-3,5,6-trichloropicolinic acid on woody plants revealed this compound to be highly useful for the control of many brush species, extensive additional tests were set up in numerous countries to study dosage rates, time of application, and species response under widely varying environmental conditions.

Brush species, primarily coniferous, were studied in Canada. 4-amino-3,5,6-trichloropicolinic acid at less than 1 lb. a.e./100 gal., used as a wetting foliage spray controlled such species as balsam (Abies balsamea L.), Norway, white, and black spruce (Picea abies, P. glauca and P. niger) and hemlock (Tsuga canadensis).

In Australia various brush species (Baccharis, Cassia, Eucalyptus, Lantana, Mimosa, and Salix spp.) were controlled at low rates of a.e./ac. Gorse (Ulex europaeus), and other leguminous brush species were found to be very sensitive to this herbicide in New Zealand. In Jamaica, woody plant control in tropical vegetation using 4-amino-3,5,6-trichloropicolinic acid at 1/2 lb./100 gal. water applied as a wetting spray gave very effective control even on brush species generally considered resistant to 2,4-D and 2,4,5-T herbicides.

In other experiments in Europe, Africa and South America, Dow Research and Development personnel in co-operation with public and private research organizations have conducted experiments on many species of woody plants. To date, more than 185 species of brush or woody plants have been found to be moderately or highly susceptible to rates of less than 1 lb. per 100 gal. of this active herbicide when applied as a wetting spray. Some genera of highly susceptible plants throughout the world are Acacia, Acer, Alnus, Arctosphylos, Betula, Carya, Cassia, Cephalanthus, Cornus, Corylus, Cretaeagus, Cytisus, Diospyros, Gledetsia, Larix, Liriodendron, Mimosa, Morus, Nyssa, Oxydendron,

Populus, Prosopis, Prunus, Quercus, Rhus, Robina, Rosa, Rubus, Salix,  
Sambucus, Sida, Taxiodium, Turnefortia, Vitis, and Zanthoxylum.

#### Perennial Weed Control.

Deep rooted perennial weed control has been investigated in a number of countries. Complete eradication with one spray at the rate of 2 to 3 lb. a.e. /ac. has been obtained with wild morning glory (Convolvulus arvensis), Canada thistle (Cirsium arvense), Russian knapweed (Centaurea repens) and leafy spurge (Euphorbia esula).

#### Selective Herbicide Uses.

Selective use of 4-amino-3,5,6-trichloropicolinic acid in certain crops appears promising. Experiments are being evaluated on a global scale at this time. Another paper is being presented at this conference on the use of 4-amino-3,5,6-trichloropicolinic acid in cereal crops (H. M. Lawson). Other graminaceae appear to allow good crop tolerance. In sugar cane growing areas of the world, many weeds, resistant to presently used herbicides, have been shown to be moderately to highly susceptible. Good broadleaved weed control has been obtained in transplanted rice.

Beans of all types, tomatoes, peas, cotton, tobacco, grapes, cucurbits, sugar beets, and peanuts are all highly sensitive to small amounts of 4-amino-3,5,6-trichloropicolinic acid. Here in the British Isles, and in Europe, however, the brassica species show tolerance, and selective use in these crops appears very promising.

#### Pastures and Rangelands.

Excellent grass growth has followed applications for control of brush. Extended annual broadleaved weed control is also achieved. Thus, with little competition grass regeneration is rapid. The improvement of pasture lands through the conversion of mixed weed, brush, and grass stands to pure grass forage land has been repeatedly proven by experiments in South America, Australia and Africa.

### ANALYTICAL METHODS

Bioassay methods have been developed for the detection of 4-amino-3,5,6-trichloropicolinic acid in plants at sublethal doses. Residual effects in soils have also been studied by Hamaker (et al), using serial dilution techniques and live plants as indicators. These methods are useful in many types of investigations and are within the capabilities of most laboratories which can grow uniform bean seedlings.

Chemical methods for detection of possible residues in plant and animal tissue are being developed.

References.

- GANTZ, R. L. & LANING, E. R. (1964)  
Chemical Control of Woody Rangeland Species.  
*Biokemia* 3, 20-23
- HAMAKER, J. W. et al (1963)  
A Picolinic Acid Derivative; A Plant Growth Regulator.  
*Science* 141, 363
- LANING, E. R. (1963)  
Tordon - For the Control of Deep-Rooted Perennial Weeds.  
*Biokemia* 2, 2-5
- LEASURE, J. K. (1964)  
Bio-assay Methods for 4-amino-3,5,6-trichloropicolinic acid  
Weeds 12, 3, 232-233
- WATSON, A. J. & WILTSE, M. G. (1963)  
Tordon - For Brush Control on Utility Rights-of-Way.  
*Biokemia* 2, 11-14

NEW HERBICIDES

SUBSTITUTED CARBAMOYLMETHYL BIPYRIDILS  
PP 407 AND PP 745

H.M. Fox

Plant Protection Limited, Jealott's Hill Research Station,  
Bracknell, Berkshire

Summary Chemical, toxicological and biological information is given concerning two new substituted 1,1'-carbamoylmethyl-4,4'-bipyridylium compounds, coded PP 407 and PP 745.

INTRODUCTION

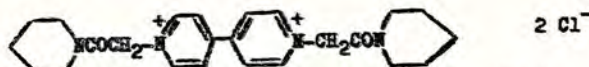
PP 407 and PP 745 are new herbicides, related to paraquat and diquat. They are similar to them in many of their properties but are very much more selective in their action. They are non-injurious to grass species and very effective against a number of broad leaved weeds many of which are resistant to MCPA and 2,4-D. PP 407 and PP 745 were evaluated for post-emergence weed control in cereals in 1963 and 1964 and in yield trials in 1964 and the results are presented in a separate paper (Fox and Beech 1964).

Chemical and Physical Properties

PP 407

Chemical name: 1,1'-di(pentamethylenecarbamoylmethyl)-4,4'-bipyridylium dichloride.

Structural formula:



Molecular formula:  $C_{24}H_{32}O_2N_4Cl_2$  Molecular weight: 479.2

Hydration: The monohydrate is obtained on drying recrystallised material at 100°C.

Solubility: Extremely soluble in water. Moderately soluble in methanol and ethanol, sparingly soluble or insoluble in most other organic solvents.

Melting point: 303°C (decomp)

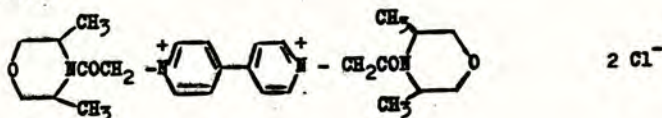
Stability: Unstable in alkaline solutions

Formulation: Aqueous solution 2 lb ion per Imp. gal.

PP 745

Chemical name: 1,1'-bis(3,5-dimethylmorpholinocarbonylmethyl)-4,4'-bipyridylium dichloride.

Structural formula:



Molecular formula:  $C_{26}H_{36}O_4N_4Cl_2$

Molecular weight: 539.2

Hydration: The monohydrate is obtained on drying recrystallised material at 100°C.

Solubility: Extremely soluble in water. Moderately soluble in methanol, almost insoluble in higher alcohols and other organic solvents.

Melting point: Decomposes above 300°C.

Stability & Formulation : As PP 407

#### Toxicological data

Pending completion of more accurate estimations of acute and sub-acute toxicity, the following data, obtained from experiments on female rats, are given to indicate the level of PP 407 and PP 745 toxicity to mammals.

	PP 407	PP 745
Acute oral toxicity (single dose) LD 50	800-1600 mg/kg	400-800 mg/kg
Interperitoneal LD 50	12.5-25 mg/kg	25-50 mg/kg

Observations to date indicate that the pharmacological properties of PP 407 and PP 745 are broadly similar to those of other bipyridyls studied.

Residue determinations indicate that no PP 407 or PP 745 residues were detectable in straw, husk or grains of wheat and barley crops treated in 1963 experiments.

#### Biological data

PP 407 and PP 745 are selective bipyridyls which show considerable promise for post-emergence weed control of broad leaved weeds in cereals. They are also likely to be of interest for selective weed control in sports turf and pastures. To ensure full effect, the chemicals should be applied with a non-ionic wetting agent such as 'Agral' 90 at a concentration of 0.1% in the diluted spray.

Good control of the following weeds, which are moderately resistant to MCPA and 2,4-D, has been obtained at the rates indicated.

<u>Anthemis cotula</u> (Stinking mayweed)	1 lb ion/ac
<u>Chrysanthemum segetum</u> (Corn marigold)	1 lb ion/ac
<u>Polygonum persicaria</u> (Redshank)	1 lb ion/ac
<u>Polygonum aviculare</u> (Knotgrass)	1.5 lb ion/ac
<u>Polygonum convolvulus</u> (Black bindweed)	1.5 lb ion/ac
<u>Spergula arvensis</u> (Corn spurrey)	1 lb ion/ac
<u>Tripleurospermum maritimum</u> ssp. <u>inodorum</u> (Scentless mayweed)	1 lb ion/ac
<u>Veronica</u> spp (Speedwell)	1 lb ion/ac

The following MCPA susceptible weeds have also been controlled satisfactorily -

<u>Chenopodium album</u> (Fat hen)	1 lb ion/ac
<u>Raphanus raphanistrum</u> (Ranch)	1 lb ion/ac
<u>Sinapis arvensis</u> (Charlock)	1 lb ion/ac

However Galium aparine (cleavers) and Stellaria media (chickweed) were moderately resistant to 1.5 lb ion/ac.

A minor degree of phytotoxicity to sprayed cereals has been observed but the crop rapidly recovers and shows no permanent effects in growth or ear development. Yield data show that the chemicals, at rates used for weed control, have not depressed grain yield. However PP 745 at two to three times the recommended dose did cause slight but nevertheless, significant yield depression in one trial on spring wheat.

PP 407 and PP 745 have a mode of action and properties similar to diquat and paraquat. They are quick acting, rainfast and non-toxic, contact herbicides with limited systemic properties. They are inactivated on contact with soil and their herbicidal efficiency is not affected to any extent by relatively low temperature conditions.

#### Reference

- FOX, H.M. and BEECH, C.R. (1964) Bipirydylium herbicides: Field trials with PP 407 and PP 745 as selective herbicides for weed control in cereals. Proc. 7th Brit. Weed Control Conf. (in press).

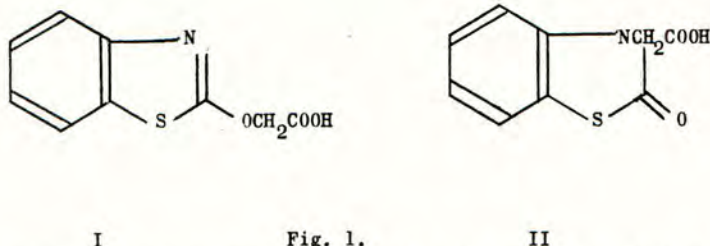
4-CHLORO-2-OXOBENZOTHIAZOLIN-3-YLACETIC ACID -  
A NEW PLANT GROWTH SUBSTANCE HERBICIDE

E. L. Leafe  
Beets Pure Drug Co. Ltd., Lenton Experimental Station

INTRODUCTION

4-Chlore-2-oxobenzethiazolin-3-ylacetic acid (R.D.7693) is a new plant growth substance which has been shown to possess highly specific herbicidal properties at very low application rates.

Growth substances based on the benzothiazole nucleus were first described by Jayne *et al* (1949), but the structures ascribed to them were later shown by Brookes and Leafe (1963) to be incorrect. The structure ascribed to the parent compound by Jayne *et al* (1) and the correct structure (II) are depicted in Fig. 1.



Substituted derivatives of II, and analogous compounds based on the benzothiazole nucleus have been evaluated as post-emergence herbicides. The 4-chloro derivative was selected for field evaluation and has been shown to be extremely active against chickweed (*Stellaria media*) and cleavers (*Galium aparine*).

CHEMICAL AND PHYSICAL PROPERTIES

R.D.7693 was synthesised from *o*-chloro-aniline by conversion to the corresponding thiourea followed by ring closure. The resulting 2-amino-4-chlorobenzothiazole was converted to the 4-chloro-2-hydroxy compound and this to R.D.7693 by condensation with ethyl chloroacetate followed by hydrolysis of the ester group.

R.D.7693 is a white crystalline material, M.P. 193°C, of low water solubility and is stable except under conditions of vigorous alkaline hydrolysis. The potassium salt is soluble in water (48%<sup>v</sup>/<sub>v</sub> at 20°C) and is a convenient form for formulation. Formulations may also be prepared from esters of R.D.7693.



## TOXICOLOGY

### Oral toxicity-Rat

R.D.7693 in the form of its potassium salt was administered by mouth to batches of 10 rats. The highest rate, 3,000 mg/Kg., caused no deaths.

### Dog

250 mg. and 500 mg/Kg. administered orally had no effect. 1000 mg/Kg. caused vomiting but no other effect.

## HERBICIDAL ACTIVITY

### Glasshouse screening tests

Table.1. lists the activity against three important weed species of 2-oxobenzothiazolin-3-ylacetic acid, its chloro derivatives, and some closely related compounds.

Dichloro-, trichloro- and methyl derivatives were less active. Replacement of the oxo- grouping by thiono - reduced activity: its replacement by imino- destroyed activity. Extension of the side chain reduced activity while replacement of the benzothiazole nucleus with the benzoxaline nucleus led to inactivity.

Table.1. indicates the extreme susceptibility of chickweed and cleavers to R.D.7693, a rate of 0.25 lb/acre giving complete or almost complete control. In comparison, under these conditions, a rate of 1-1.5 lb/acre of mecoprop was needed for an equivalent effect.

A more complete picture of the herbicidal properties and selectivity of R.D.7693 is given in Table.2.

Table 1

Comparative response of three weed species to post-emergence application  
of 2-oxobenzthiazolin-3-ylacetic acid, its derivatives and some closely  
related compounds

Compounds were formulated as their water soluble diethanolamine salts and applied to seedling plants at 100 gal/ac.

Injury rating 0 = no visible response 6 = 100% Kill.

	Cleavers			Chickweed			Redshank		
	<u>Galium aparine</u>			<u>Stellaria media</u>			<u>Polygonum persicaria</u>		
	<u>0.25</u>	<u>0.5</u>	<u>1.0</u>	<u>0.25</u>	<u>0.5</u>	<u>1.0</u>	<u>0.25</u>	<u>0.5</u>	<u>1.0</u> lb/ac.
2-oxobenzothiazolin-3-ylacetic acid	2	3	4	0	0	0	0	2	5
4-Chloro-2-oxobenzothiazolin-3-ylacetic acid	4	5	6	5	6	6	4	5	6
5-Chloro-2-oxobenzothiazolin-3-ylacetic acid	1	3	3	1	2	2	0	3	4
6-Chloro-2-oxobenzothiazolin-3-ylacetic acid	0	3	4	0	0	0	0	2	3
7-Chloro-2-oxobenzothiazolin-3-ylacetic acid	0	0	1	0	0	1	0	0	2
2-Iminobenzothiazolin-3-ylacetic acid	0	0	0	0	0	0	0	0	0
2-Thionobenzothiazolin-3-ylacetic acid	0	1	2	0	0	1	0	0	0
2-Oxobenzoxazolin-3-ylacetic acid	0	0	0	0	0	0	0	0	0
2-(4-Chloro-2-oxobenzothiazolin-3-yl)propionic acid	1	2	3	2	3	3	0	0	1

Table 2

The response of several crop and weed species to post-emergence application of R.D.7693 (4-Chloro-2-oxobenzothiazolin-5-ylacetic acid) as its diethanolamine salt

Injury rating 0 = no visible response 6 = 100% kill. Rate of application:-  
4 lbs. a.e./acre applied to seedling plants in a volume of 100 gal/ac.

<u>Cleavers</u> <u>Galium aparine</u>	<u>Fat Hen</u> <u>Chenopodium album</u>	<u>Redshank</u> <u>Polygonum persicaria</u>	<u>Marigold</u> <u>Chrysanthemum segetum</u>	<u>Chickweed</u> <u>Stellaria media</u>	<u>Wild Oat</u> <u>Avena fatua</u>	<u>Wheat</u>	<u>Sugar Beet</u>	<u>Pea</u>	<u>Clover</u>	<u>Kale</u>	
R.D.7693	6	5	6	4	6	0	0	5	6	3	4

The lower activity of R.D.7693 on kale and clover is in marked contrast to phenoxyacetic and phenoxypropionic acids, but R.D.7693 shares with the phenoxy-acids low activity towards the Graminae.

Field Trials

From the results of screening and extension tests, R.D.7693 was selected for field evaluation as a post-emergence herbicide.

Table.3. shows the results of a selection of field trials in which R.D.7693 was evaluated alone or in mixture with M.C.P.A. for the control of common weeds of cereal crops. R.D.7693 and M.C.P.A. were formulated as their potassium salts.

These results showed the high specificity of R.D.7693. For example, chickweed and cleavers, typically M.C.P.A. resistant, were adequately controlled at rates between 0.25 and 0.51lb/a.e.per acre. In contrast charlock and fat hen, typically M.C.P.A. susceptible, required rates in excess of 1.0 lb/acre. Redshank needed at least 1.0 lb/acre for adequate control and mayweeds proved resistant. No damage to cereals occurred in any of the field trials.

Table 3

The response in field trials of some common weed species to R.D.7693 alone and in mixture with M.C.P.A.

Injury rating 0 = No response, 5 = 100% control.

Hucknall, Notts.

Oats 4-5 leaf stage  
Weed - advanced seedling stage

	Rate lb/acre	Chickweed	Stellaria media	Charlock	Fat Hen	Chenopodium album
RD.7693	0.25	4		3		2
"	0.5	4		4		3
"	1.0	5		4		4
RD.7693/	0.25	} 5		5		5
MCPA	1.125					
RD.7693/	0.5	} 5		5		5
MCPA	1.125					
Mesoprop	2.4	5		5		5

Stanton, Notts.

Spring barley 5-6 leaf stage.  
Weed - advanced seedling stage.

	Rate lb/acre	Redshank	Polygonum persicaria	Fat Hen	Chenopodium album	Charlock	Sinapis arvensis
RD.7693	0.25	1		0			1
"	0.5	3		1			2
"	1.0	4		2			4
RD.7693/	0.25	} 2		5			5
MCPA	1.125						
RD.7693/	0.5	} 4		5			5
MCPA	1.125						
RD.7693/	1.0	} 4		5			5
MCPA	1.125						
Mesoprop	2.4	2		5			5
MCPA	1.125	2		5			5

Thurgarton, Notts.

Spring barley 5-6 leaf stage.  
Weed - advanced seedling stage.

	Rate lb/acre	Cleavers	Galium aparine	Chickweed	Stellaria media	Charlock	Sinapis arvensis
RD.7693	0.125	3		3			1
"	0.25	4		4			4
Mesoprop	2.4	4		5			5

Nailstone, Leics.

Spring barley.  
Weed - advanced seedling stage.

	Rate lb/acre	Redshank	Polygonum persicaria	Hempnettle	Galeopsis tetrahit	Chickweed	Stellaria media
RD.7693	0.125	1		0			3
"	0.25	2		0			5
Dichler- prep	2.4	4		3			5

For general weed control in cereals, R.D.7693, when used in conjunction with, for example, M.C.P.A. appeared largely to duplicate the action of other herbicides which have been developed in recent years. Recent work has therefore been directed towards its utilisation in situations in which neither the phenoxyacetic acids nor the phenoxypropionic acids can be used, for example, in direct and undersown legume crops where chickweed and cleavers have become a serious problem. Two other uses are under active investigation. 1) The control of chickweed and cleavers in crops which may tolerate the very low doses of R.D.7693 required and 2) combination with other herbicides whose spectrum of activity is deficient in respect of these two weeds.

#### ACKNOWLEDGEMENTS

I would like to thank Mr. R. F. Brookes and Dr. D. H. Godson who synthesised the compounds discussed in this paper and Mr. A. J. Mayes who carried out the field work.

#### REFERENCES

JAYNE, D.W., DAY, M.H., and NOLAN, K.G. U.S. Patent 2,468,075  
(April 26, 1949)

BROOKES, R.F., and LEAFE E.L., (1963)  
Structure and Plant Growth - regulating activity of some  
2-Benzothiazolyloxyacetic Acids and 2-Oxobenzothiazolin-3-ylacetic Acids.  
Nature 198 589-590

AN EXPERIMENTAL HERBICIDE FOR THE SELECTIVE CONTROL  
OF ANNUAL SEEDLING WEED GRASSES IN TURF AND CROPS

R. W. Varner, M. B. Weed and H. L. Ploeg  
E. I. du Pont de Nemours & Company, Inc.  
Wilmington, Delaware, U.S.A.

INTRODUCTION

1-(2-Methylcyclohexyl)-3-phenylurea is being offered to investigators for evaluation as a new chemical tool to be used in the selective control of certain annual seedling weed grasses. This herbicide has unique characteristics which make it an obvious candidate for the control of some undesirable grasses in turf and suggest it for many crop applications where annual grasses are an important part of the weed problem. Specific recommendations for turf use are given. For crop uses, however, resourceful evaluation under a range of conditions will be required to develop fully the prospective utilities that are outlined.

An outstanding feature of 1-(2-methylcyclohexyl)-3-phenylurea is its truly selective preemergence elimination of several annual seedling grasses from other grasses. It has been shown, for example, that treatment at the time of seeding a new turf will control crabgrass (Digitaria ischaemum and D. sanguinalis<sup>1/</sup>) and some other annual grasses without causing injury to germinating seeds of common temperate-region turf species.<sup>2/</sup> The extent of the tolerance of desirable turf grasses has been most strikingly demonstrated by the successful dry application of mixtures of turf seed and chemical to a prepared seedbed. Such applications have controlled annual weed grasses yet permitted the normal establishment of the new turf.

The selective control of some grasses with safety to seeds and seedlings of other grasses also extends to field crops. Applications preemergence to the crop have controlled such weeds as crabgrass, foxtail (Setaria viridis and S. lutescens) and downy brome (Bromus tectorum) without significant injury to some cereals. Expanded evaluation may disclose other instances of the beneficial removal of one grass from a planting of another. It

---

<sup>1/</sup> These two species are serious turf pests in a large part of the United States.

<sup>2/</sup> Annual bluegrass (Poa annua) has not been controlled in any stage of growth.

is to be hoped, for instance, that work in the United Kingdom and Europe will demonstrate that selective control of black-grass (Alopecurus sp.) is feasible.

A second distinctive characteristic of 1-(2-methylcyclohexyl)-3-phenylurea is its wide margin of safety on a great number of broadleaf crops. Thus, this material is worthy of consideration for the control of annual seedling grasses in broadleaf crops. The evaluation of the new candidate is particularly desirable in situations where susceptible annual grasses represent the critical problem or where broadleaf weeds can be removed by some other means.

A final unusual effect is the differential retarding or stunting of annual grasses when the chemical is applied post-emergence or preemergence at sub-lethal rates. This response appears to be due to inhibited root growth which leaves the affected plants poorly equipped to stand the effects of drouth or competition.

#### TOXICITY INFORMATION

Acute oral toxicity of 1-(2-methylcyclohexyl)-3-phenylurea for the white male rat is low, its Approximate Lethal Dose (ALD) being greater than 5000 mg/kg of body weight.

A 50% wettable powder exhibited low cumulative oral toxicity when administered to male rats at the extremely high rate of 3400 mg/kg/day of active ingredient for a total of ten doses.

The active ingredient was fed to male and female albino rats for 95 to 97 days at levels as high as 5000 - 7500 ppm (dietary level raised to the higher concentration after 6 weeks) in an adequate diet without nutritional or clinical evidence of toxicity. There was evidence from blood examinations of a slight anemia, but this was observed only in male rats fed the highest dietary level. Organ weight measurements and histological examination of tissues from animals that received the highest level disclosed no significant change attributable to the chemical.

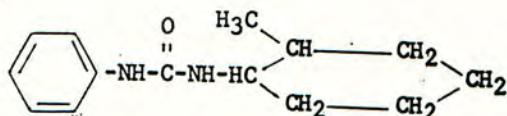
Two-year feeding tests with rats and dogs are in progress.

When either 10 mg of a 50% wettable powder formulation or 0.1 ml of a 10% suspension in propylene glycol was introduced into rabbits' eyes, the reactions were no greater than those that would result from the introduction of an inert material into the eyes.

The material was not irritating to guinea pig skin during prolonged contact as a 10% suspension in dimethyl phthalate. As a 25% suspension it was mildly irritating, as a 40% suspension it was moderately to strongly irritating. It did not, however, produce an allergic contact dermatitis.

PHYSICAL AND CHEMICAL PROPERTIES OF ACTIVE INGREDIENT<sup>1/</sup>

Structural Formula:



Physical Form:	white, crystalline solid
Molecular Formula:	C <sub>14</sub> H <sub>20</sub> N <sub>2</sub> O
Molecular Weight:	232.32
Melting Point:	133 - 138°C
Odor:	None
Solubility in water at 25°C:	18 ppm
Solubility in organic solvents:	soluble to the extent of 10% or more in ethyl alcohol, "Cellosolve", dimethyl formamide, dimethyl acetamide, methylene chloride and isophorone
K value in Keyport silt loam:	2.5 <sup>2/</sup>

<sup>1/</sup> Provided for test purposes as a free-flowing wettable powder containing 50% by weight of 1-(2-methylcyclohexyl)-3-phenylurea.

<sup>2/</sup> K value = ppm of herbicide adsorbed on soil (air-dry basis) which is in equilibrium with an aqueous solution containing 1 ppm of herbicide at 25°C.



## PROSPECTIVE HERBICIDAL USES

### Annual Grass Control in Turf

The unusual safety margin of this compound on a number of important sod grasses makes it an outstanding candidate for use on lawns, golf courses and other turf areas. The degree of safety is illustrated by the fact that seed of common types of Kentucky bluegrass (Poa pratensis), red fescue (Festuca rubra) and bentgrass (Agrostis sp.) have germinated and grown in soil containing eight times as much of the new compound as was required to control unwanted annual grasses such as crabgrass and foxtail. This unprecedented tolerance permits preemergence use in the planting of new lawns as well as complete freedom in reseeding parts of existing lawns either before or after treatment.

In planting a new turf, it is suggested that soil preparation and seeding be done in the usual manner and that the herbicide be applied immediately at rates in the range of two to six pounds of the active ingredient per acre. (The lower rate is proposed for sandy soils and the higher rate for soils containing high levels of clay or organic matter.) Such areas should be retreated about one month later with an additional three or four pounds of active ingredient per acre to provide control until the end of the season. Any watering program that will promote uniform germination and establishment of the turf stand should be adequate for optimum performance of the chemical.

Applications to established turf are most effective if made prior to the germination of the annual weed grass species to be controlled. Treatment just after a mowing and during the month preceding the earliest expected germination of the weed grasses has been satisfactory. For average use situations, ten pounds active per acre is adequate for full-season control. Where soils are very high in organic matter, a use rate of twelve pounds per acre is more desirable. These rates may be employed even where the turf contains areas recently planted or where there might be plans for prompt subsequent reseeding.

Results may be disappointing in an established turf if at least one-half inch of water is not received between the time of treatment and the germination of the weed grasses. Where applications are made late (i.e. near time of germination), it may be desirable to irrigate promptly to assure that the chemical moves into the soil sufficiently to control early-germinating weed grasses.

A necessary feature of a turf herbicide is that it be safe to trees and other ornamental plants. Thus, our evaluations have included applications at rates as high as 120 pounds active per acre to plots with trees and 40 pounds per acre to plots with representative ornamental shrubs and vines for two consecutive years without evidence of injury.

### Seedling Grass Control in Crops

In evaluating 1-(2-methylcyclohexyl)-3-phenylurea, it should be remembered that this material is a useful tool for the control of certain grasses growing from seed but that it has very limited activity on broadleaves.<sup>1/</sup> In general, the evaluation of the new material is suggested for two weed situations: (1) where seedling grasses are the limiting factor or (2) where broadleaf species present in a mixed weed population can be removed by other means. Methods of removing broadleaf weeds which may be employed in conjunction with the new compound include other chemicals or physical roguing.

Safety to most broadleaf crops is such that applications could be made both preemergence and then again after any mechanical cultivation necessary for the removal of broadleaf weeds. For those crops in which present production practice involves hand weeding, the cost of this operation could be greatly reduced by preventing the development of many of the grasses with the new chemical.

Applications are most effective if made preemergence to the seedling grass weeds to be controlled. Necessary use rates increase with the colloidal and organic matter content of the soil. Suggested levels for normal preemergence applications are:

For sandy and sandy loam soils	2-3 pounds active per acre
For heavier soils low or medium in organic matter content	4-6 pounds active per acre
For soils with sufficient organic matter to impart a dark color	6-10 pounds active per acre

---

<sup>1/</sup> Two to four pounds per acre will provide preemergence control of some broadleaf species including purslane (Portulaca oleracea) and lambsquarters (Chenopodium album).

Grass weeds that have been controlled by such applications include crabgrass, downy brome, foxtail (embracing *Setaria faberii*), barnyardgrass (*Echinochloa crusgalli*), witchgrass (*Panicum capillare*), Johnsongrass (*Sorghum halepense*; from seed only) and nimblewill (*Muhlenbergia schreberi*; from seed only).

Normal preemergence applications are effective in controlling weed grasses only if the active ingredient is carried to the depth of the germinating weed seeds. Treatments have generally been successful if one-half inch or more of rainfall or irrigation water reaches the soil within ten to fourteen days. Mechanical incorporation of the chemical into the soil may be useful where water cannot be relied upon to provide the necessary downward movement. The incorporation procedure should generally be such as to retain the chemical in the top one-half inch. If deeper incorporation is employed, suggested use rates should be increased to compensate for the dilution effect.

Postemergence applications, although less effective in eliminating weed grasses, do have a marked stunting effect on young seedlings of many grasses and may be useful under some circumstances. Stunted seedlings often are not an important factor in competition subsequently and, due to greatly restricted root growth, may fail to survive even brief periods of water shortage. Also, they are much easier to remove by mechanical means as a consequence of the poorer anchoring.

The pattern of soil disappearance has been studied over a three-year period on field plots situated in a temperate climate (lat. 40° north) under an annual rainfall of about 45 inches. There has been no evidence of residual chemical in the soil after one year even with applications much in excess of those required for weed control. Work on the mechanisms of loss has not advanced sufficiently to justify discussion at this time.

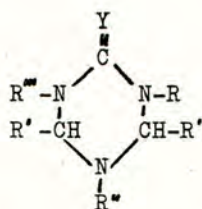
SUBSTITUTED PERHYDROTRIAZINES -  
A NEW GROUP OF HERBICIDES

L. Eue, H. Hack, W. Schäfer, R. Wegler  
Farbenfabriken Bayer AG, Leverkusen

Summary: Substituted perhydrotriazines are described as a new group with herbicidal properties. By means of several examples it is shown how the herbicidal properties change when the substituents at a symmetrical perhydrotriazine are varied. It is shown to be essential that at least one substituent must be of an aromatic nature. The compounds can be used for both pre-emergence as well as post-emergence applications, which, however, is largely dependent upon the substituents. The selectivity for the different crops and the general herbicidal potency are also dependent upon these substituents.

INTRODUCTION

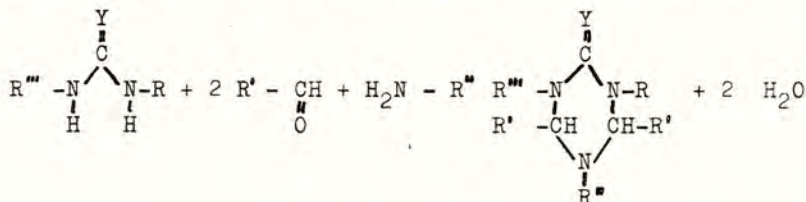
Tri- and tetra-substituted ureas are known as herbicides from the literature on this subject. The prime feature of these compounds is that they chiefly act as pre-emergence herbicides. They can be used as total herbicides or for selective weed control in different crops, the dosage being considerably reduced for selective weed control. Gysin et al. found that substituted triazines are suitable both for non-selective as well as for selective weed control. Recently uraciles and substituted pyrimidines came in commercial use as herbicides. In recent years we have carried out studies with compounds which can be termed as perhydrotriazines. We have applied the following general formula to this group of compounds:



where Y = oxygen, sulphur or NH. We shall not enter at this stage into the significance of the organic substituents R - R''', which can be seen from patents of Farbenfabriken Bayer.

#### METHOD AND MATERIALS

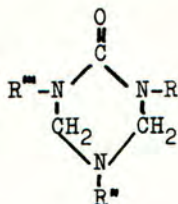
The compounds described in this paper can be prepared, for example, from monosubstituted or unsymmetrical disubstituted ureas, thioureas or guanidines by reaction with 2 moles of an aliphatic aldehyde and one mole of a primary amine in the sense of the following reaction scheme.



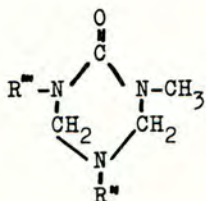
The compounds are tested in the greenhouse on suitable plant material. In the post-emergence experiment young plants of different genera are sprayed with solution and their behaviour is observed for 3 weeks. The testing of the compounds as pre-emergence herbicides is carried out in seed dishes in which seeds of different plant species are sown. The seed dishes are treated about 24 hours after sowing. Germination and growth are observed.

## RESULTS

To simplify the presentation of our results it should be pointed out that the more powerful herbicides come from the group of compounds for which Y = oxygen in the general formula I. We have also found that it is expedient to choose hydrogen for the substituents R. We, therefore, simplify our formula I in the sense of the following formula II



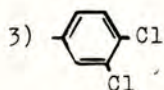
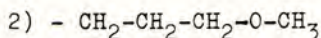
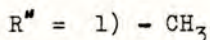
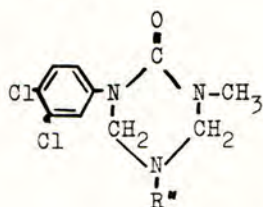
Of the numerous compounds we tested, it was ultimately found that some of the aliphatic substituted compounds are quite good herbicides but that they are excelled in their action by partly aromatic or cycloaliphatic substituted compounds. To simplify our survey R will be replaced by methyl in the following analysis. We thus obtain the general formula III



Compounds where R = OH or O-CH<sub>3</sub> although show good herbicidal activity, these compounds are not referred in this paper.

Let us continue to study the herbicidal action of our compounds by using the example where R''' means 3,4-dichlorophenyl and vary the substituents R''. This compounds can be prepared using nitrogen compounds able to react twice with formaldehyde.

Formula IV



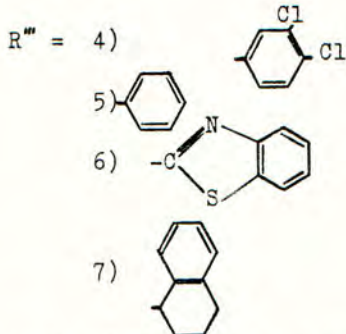
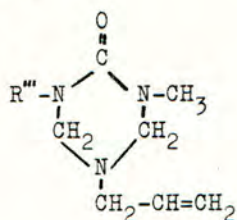
The compounds of this series usually have a good post-emergence action which, however, depends upon R'' . Below only three examples are given showing the varying selectivity in post-emergence application - at a spray concentration of 0,05 % tested on 20 different plant species, we observed the following herbicidal properties:

- 1) If R'' = methyl, there is no selectivity. The compound acts as a total herbicide.
- 2) If methoxypropyl is chosen as the substituents for R'' , the compound can be used for selective weed control in onions.
- 3) If R'' means 3,4-dichlorophenyl, this compound displays good selectivity in cereals and rice.

The pre-emergence action of the compounds is usually poor, outstanding being solely the compound 1) in which R'' = methyl. In contrast to post-emergence application it is a suitable pre-emergence herbicide (2 - 4 kg/ha) for selective weed control in crops of cereals, maize, cotton, potatoes, groundnuts and beans.

The three examples given above have shown how the herbicidal properties change when R'' is differently substituted; the following examples are intended to demonstrate how the herbicidal properties change when the substituents R''' are varied. In this case R'' will be allyl.

Formula V



- 4) With R''' as 3,4-dichlorophenyl. The post-emergence action of the compound is very good with selectivity in cereals and rice and the pre-emergence action is weak.
- 5) If R''' is phenyl, the compound has a good post-emergence and pre-emergence action. It can be used in potato crops for pre- and post-emergence treatments (3 - 4 kg/ha). In maize crops it can only be used as a pre-emergence treatment.
- 6) If 2-benzothiazolyl is chosen as R''' , the compound has practically no pre-emergence action but when used as a post-emergence treatment (0,1%) it gives a good effect with selectivity in rice, potatoes and beans.
- 7) If naphthyl is chosen as the substituent the herbicidal action disappears almost completely.

We have shown by means of the 7 examples how the herbicidal action changes by varying the substituents. We have meanwhile continued our studies, using substituted aromatics and heterocyclic aromatics as substitutes for R'' and R''' . The provision made above that the oxygen compounds are superior to the sulphur or nitrogen compounds does not hold generally. In these studies we have found some interesting compounds with a selective action, e.g. for beet-growing. These studies have not yet been concluded so that we cannot yet report on them.



## THE USE OF CCC TO PREVENT LODGING IN WHEAT

J. J. B. Caldicott and C. D. Lindley  
Cyanamid of Great Britain Limited, Bush House, Aldwych, London.

Summary Experimental work is described with 2-chloroethyl trimethylammonium chloride (CCC), a plant growth regulant used to prevent lodging in wheat. A field trial was carried out in 1963 using a seed treatment of CCC. Due to the hygroscopic nature of CCC this resulted in a lower seeding rate of the treated seed, which reduced plant stand and grain yield. Treatment resulted in a reduction of plant height on all varieties. Six field experiments were laid down in 1964 to observe the effect of a foliar spray of CCC on the winter variety Capelle-Desprez at the rate of 2.5 lb. a.i. per acre, applied at the 4th - 6th leaf stage. Two levels of nitrogen top dressing were applied, the higher level being 50% greater than the lower. Treatment resulted in a reduction of plant height in all trials. Detailed examination of stems indicated that the most pronounced shortening had taken place in the second and third internodes from the base of the plant. Lodging occurred in varying degrees in five trials and was reduced or eliminated in all of these by CCC treatment. Yield increases were recorded on CCC plots in three trials. The increases were greatest where lodging on the untreated plots was most pronounced. CCC treatment allowed the use of 50% extra nitrogen top dressing with less subsequent lodging than on untreated plots at the normal nitrogen level. Examination of the weights of 1000 grains, the numbers of fertile spikelets per ear, the numbers of grain per ear and the numbers of grains per fertile spikelet showed that these were not affected by CCC treatment.

### INTRODUCTION

The growth regulant 2-chloroethyl trimethylammonium chloride (CCC) was first described by Tolbert (1960<sup>a</sup>). The growth of wheat plants was affected by applications of CCC to the soil, by foliar sprays or by seed soaking prior to sowing. These changes consisted of shorter and thicker stems, broader leaves and more uniform growth. They were the opposite to those resulting from the use of gibberellic acid (Tolbert 1960<sup>b</sup>).

Experimental work was subsequently undertaken in Germany and Austria by Linser, Mayr and Bodo (1961), Linser and Kuhn (1961), Mayr, Primost and Rittmeyer (1962) and by Jung and Sturm (1964) to determine the practicality of using CCC to prevent lodging in wheat. Mayr *et al.* (1962) found that 3.6 lb. CCC per acre mixed with the fertiliser and applied at the 4th leaf stage, prevented the lodging of the weak-stawed variety "Tassilo" even when 160 units of nitrogen per acre were applied. Jung and Sturm (1964) induced a 20% shortening of stem length, the lower internodes being affected to the greatest extent, with 2.2 lb. CCC per acre, applied as a foliar spray at the 5th leaf stage. Treatment resulted in slightly increased yields even though little lodging occurred in the untreated control plots.

It was therefore decided to investigate the use of CCC on wheat in the United Kingdom where lodging can, in some years, be very pronounced.

#### METHOD AND MATERIALS

In 1963 one replicated trial was carried out with two levels of CCC seed dressing (1 lb. a.i. and 0.5 lb. a.i. per 100 lb. seed) on three varieties of spring wheat (Jufy I, Koga II and Opal). Observations were made on plant stand, plant height and yield.

Because the 1963 trials showed that the use of CCC as a seed dressing on wheat was not practicable, the 1964 trials were designed to use CCC as a foliar spray. Six replicated trials were laid down with threefold replication. Details of these are given in Table I. The winter variety Capelle-Desprez was used in each trial, chosen because it is the most widely grown variety. Plot size was approximately 1/24 acre. Two levels of nitrogenous fertiliser were employed. One level of CCC was applied at the rate of 2.5 lb. a.i. per acre to half the plots, giving a total of four treatments. Since the amount of nitrogen that can be applied as a top dressing is determined by the soil type, the previous cropping, and the levels of nitrogen applied in the seed bed, it was decided to use as the normal level that which the farmer used on the remainder of the field. In one case the farmer applied no nitrogen. The type of fertiliser used was ammonium nitrate combined with calcium carbonate (23% N<sub>2</sub>). CCC was applied as a 50% aqueous solution at the rate of 4 pints in 50 gallons of water per acre, using a small boom sprayer. The date of application was timed to coincide with the 4th - 6th leaf stage; in effect, either in the last week of April or the first week of May. In most cases the nitrogenous top dressing had been made about three weeks previously.

Table I - 1964 Trial details

Trial no.	Soil type	Previous crop	Units of nitrogen		Date of top dressing	Date of CCC application
			N	1½ N		
1	peat fen	sugar beet	nil	55	8 April	5 May
2	loam	potatoes	80	120	9 April	24 April
3	peat skirt	beans	60	90	9 April <sup>(1)</sup>	24 April
4	silt loam	potatoes	70	105	8 April	23 April
5	light loam	potatoes	80	120	8 April	24 April
6	loam	barley	80	120	22 April <sup>(2)</sup>	22 April

N = Normal level of nitrogen top dressing

1½ N = 1½ times normal level of nitrogen top dressing

- Notes: (1) In trial 3, 60 units of nitrogen were applied to the whole field in March. An extra 30 units were applied to the 1½ N plots on April 9th.
- (2) In trial 6, 40 units of nitrogen were applied to the whole field in March. An extra 40 units were applied to the N plots and an extra 80 units to the 1½ N plots on April 22nd.

Assessments of plant height were made at the beginning of June and at the end of June. Three readings were made per plot. The heights recorded included the ears. An assessment of lodging was made in mid-July following a period of high winds and storms. A second assessment was made in early August. Grade figures from one to ten were used for the degree of lodging in each plot. These were then translated into per cent lodging per treatment.

The trials were harvested by combine in August. One swath was cut through each plot so that 1/40 acre was harvested leaving guard strips on each side. Yields of grain were recorded and moisture contents taken. Weights were adjusted to 15% moisture content and expressed as cwt. per acre. From the bulk yield of each plot sub-samples of grain were taken and oven dried. Estimations of 1000-grain weights were made from these samples.

Straw and ear samples were taken at harvest by pulling handfuls of plants, including roots, at twenty random points in each plot. From these, sub-samples were taken and the following observations made. From each bulk sample ten complete plants (so far as could be determined) were taken at random. From each of these the second longest stem was selected. For each of these stems the individual internode lengths were recorded together with the total length of the stem. The ear was detached and a count was made of the number of fertile spikelets and the number of grains per fertile spikelet.

Grain and straw samples were taken for residue analyses and for milling and baking tests.

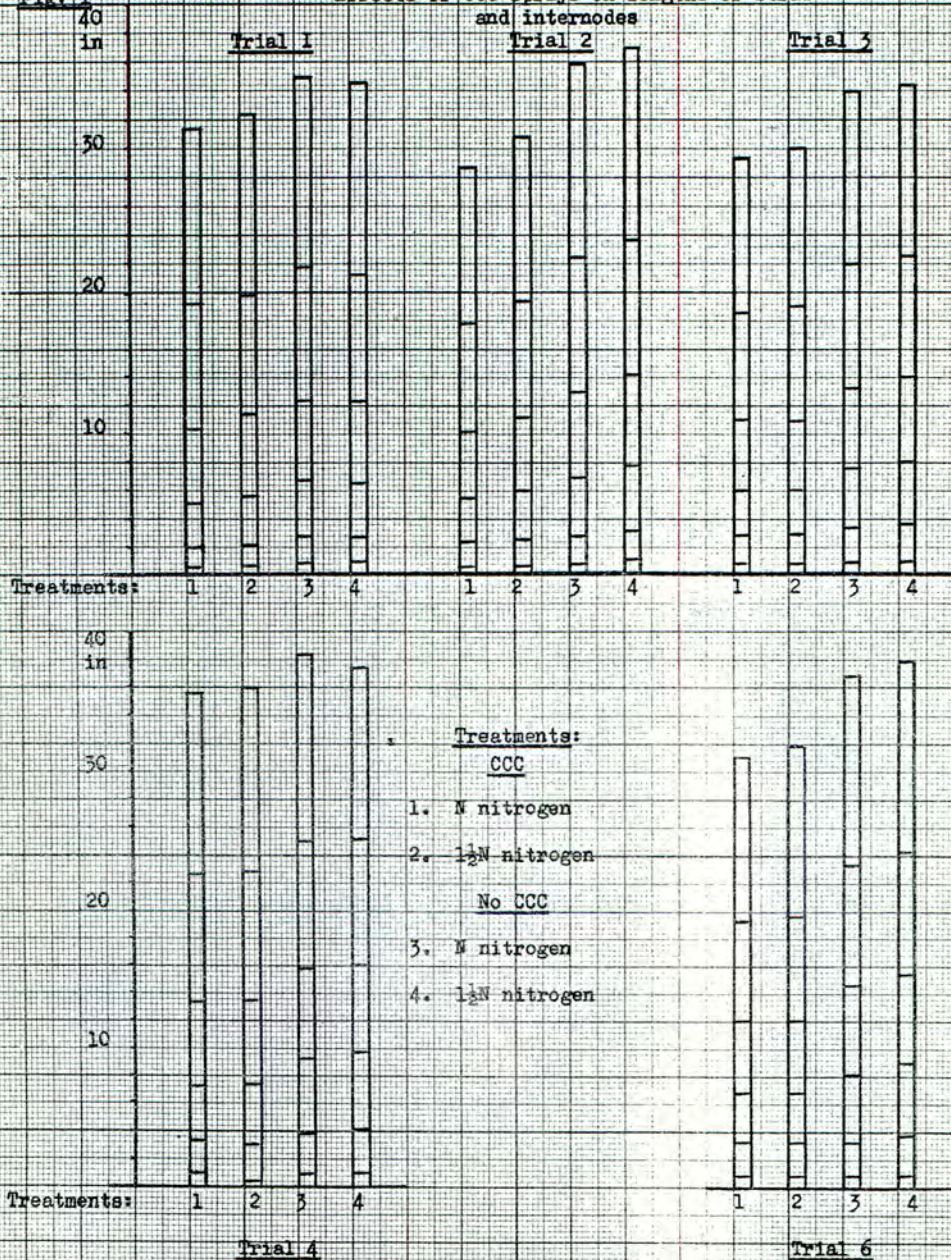
#### RESULTS

In the 1963 trials the CCC seed dressing affected the seeding rate because of its hygroscopic nature, this caused seeds to adhere so that a reduced plant stand was obtained on the treated plots. CCC treatment resulted in a yield depression on the varieties Jufy I and Koga II. Plant height was reduced by CCC, significantly on the varieties Jufy I and Opal, but not significantly on Koga II. There was no lodging on the control plots.

In 1964, significant reductions in crop height followed CCC treatment at both nitrogen levels in all cases except under the higher nitrogen level in trial 4 at the second observation. Shortening due to CCC was very marked, varying between two and seven inches. The higher level of nitrogen caused a slight but insignificant increase in height compared with the lower level in both treated and untreated plots. Figure 1 shows stem lengths at harvest compared for each trial and also the relative lengths of the internodes. No information is available for trial 5. All the reductions in height due to CCC are significant for the same nitrogen level except for the higher nitrogen level in trial 4. While the effects of CCC on total length are very evident it can also be seen how this length difference is associated with the comparative shortening of individual internodes. Maximum shortening occurred in the second or third internodes, sometimes in both. The degree of shortening in the other internodes was irregular, tending to become less marked towards the top of the stem.

Fig. 1

Effects of CCC sprays on lengths of straw and internodes



The trials were assessed for lodging in mid-July and again in August. Table II shows the mean per cent lodging for all trials for both dates of assessment.

In mid-July some lodging had occurred in four trials. In two of these, lodging was apparent only in the untreated plots with the  $1\frac{1}{2}$  N fertiliser treatment. In trial 3 lodging was considerable in one untreated plot of each level of fertiliser, but the differences were not significant. In trial 4 lodging occurred heavily in the field and, to varying degrees, in all plots. There was significantly less lodging (at  $P = 0.01$ ) in the CCC treated plots.

Table II: Effects of CCC sprays on lodging 1964

Treatment:	Per cent of area lodged				Least significant difference	
	CCC		Untreated		P=0.05	P=0.01
Nitrogen level:	N	$1\frac{1}{2}$ N	N	$1\frac{1}{2}$ N		
Trial 1 14 July 29 July	0	0	0	0	3.6	5.4
	0	0	6.6	9.1		
Trial 2 13 July 6 August	0	0	0	1.3	N.S. N.S.	
	0	0	0	6.6		
Trial 3 13 July 6 August	0	0	20.0	13.0	N.S. N.S.	
	0	3.3	25.0	23.0		
Trial 4 13 July 31 July	26.6	30.0	66.6	71.4	18.4 13.6	27.9 20.4
	13.3	25.0	83.3	85.0		
Trial 5 13 July 2 August	0	0	0	0		
	0	0	0	0		
Trial 6 13 July 4 August	0	0	0	6.6	N.S. 3.8	5.8
	0	0	2.5	6.6		

In the early August assessment, slight lodging was evident in trials 1 and 6 in the untreated plots, with a greater incidence in the  $1\frac{1}{2}$  N fertiliser plots. In trial 3 lodging had increased and was very severe in some untreated plots, but again because of irregular distribution, significance was not registered. In trial 4 the untreated plots were more heavily lodged, whereas the CCC plots had partially recovered. Differences between fertiliser levels, though evident, were not significant; but, between treatments they were highly so.

In Table III yields are given in cwt. per acre adjusted to 15% moisture content. Significant yield increases, varying from 1.5 cwt. to 5.9 cwt. per acre were recorded on the CCC treatments in trials 2, 3 and 4. The highest increase came in trial 4 in which the greatest degree of lodging occurred. In comparing levels of fertiliser there was a general increase in yield on the  $1\frac{1}{2}$  N plots as compared with the N level but this was

significant only in the CCC-treated plots in trial 4. Conversely, in trial 3 there was a significantly lower yield on the  $1\frac{1}{2}$  N-untreated plots than at the N level.

Table III: Effect of CCC sprays on grain yield 1964

Treatment:	Grain yield in cwt./acre (15% moisture)				Least significant difference	
	CCC		Untreated			
Nitrogen level:	N	$1\frac{1}{2}$ N	N	$1\frac{1}{2}$ N	P=0.05	P=0.01
Trial 1	43.0	47.0	45.16	47.3	N.S.	
Trial 2	44.8	47.3	43.0	44.6	2.6	4.0
Trial 3	38.3	39.2	36.8	35.2	1.1	1.6
Trial 4	46.7	48.7	41.5	42.8	1.9	2.8
Trial 5	42.4	43.8	43.6	43.0	N.S.	
Trial 6	32.6	32.6	34.8	34.5	N.S.	

Moisture contents of grain were recorded at harvesting at all the sites. No significant differences were found except in trial 4, where there were significantly lower moisture contents on the CCC treatments. This was probably due to the lower degree of lodging on those plots, permitting more favourable ripening conditions.

The measurements of the weights of 1000 grains showed that there was no effect of treatment in any of the trials. Significance was registered in trial 3 in which the figure for the  $1\frac{1}{2}$  N-untreated sample was significantly less than that of the N fertiliser + CCC treatment. This result reflects the low yield recorded in the  $1\frac{1}{2}$  N-untreated plots in the same trial.

Measurements of numbers of fertile spikelets per ear, numbers of grain per ear and numbers of grains per fertile spikelet showed no significant differences except in the numbers of grains per ear in trial 6, where the numbers recorded for the  $1\frac{1}{2}$  N-untreated plots was less than on all the other treatments. This result does not, however, relate to any yield differences.

Results of grain and straw residue analyses and of milling and baking tests are not yet available and will be published at a later date.

#### DISCUSSION

The prime object of these trials was to observe the effect of applications of CCC on lodging of wheat. Seed dressings proved to be impractical because of their physical nature, but sprays, applied at the 4th to 6th leaf

stage and which could probably be included with routine weed killer applications, were successful.

In 1964, some lodging occurred in five of the trials, and, when the general degree of lodging was not too severe, it was virtually eliminated from the CCC-treated plots. In the one trial (No. 4) in which severe lodging occurred, it was not completely prevented by application of CCC but was kept to such a low level that there was a very significant increase in yield. In these trials the plots were comparatively narrow and, therefore, when the untreated plots lodged, the adjacent treated plots were exposed to the wind. There was also some carry-over of lodging from one plot to the next.

The initial and most striking result of applying CCC is a marked reduction in the length of the stem. Mayr and Presoly (1963) have shown that the shortening of the stem is accompanied by an increase in the thickness of the stem wall and in the strength of the parenchyma ring, thus strengthening the stem. In these applications the greatest shortening occurred in the 2nd and 3rd internodes from the base, the region where the stem bends when lodging occurs. It is therefore probably important to time the application to the period before shooting and jointing takes place so that these lower internodes may fully benefit from the application. In Figure I, a small height increase is apparent as a result of increasing the level of nitrogen top dressing but this is more than offset by the effect of CCC so that a high level of nitrogen in combination with CCC is far less liable to lodge than a low level of nitrogen without CCC.

Three trials have shown a significant yield increase on the CCC treatments. Two of these were the trials in which the most lodging occurred. The greatest increase occurred in trial 4, where over 80% of the untreated plots had lodged by the end of July. Here yield was increased by 5.2 cwt./acre (12.5%) on the N-fertiliser plots and by 5.9 cwt./acre (13.8%) on the  $1\frac{1}{2}$  N plots. By comparing the use of extra nitrogen in combination with CCC with the normal level of nitrogen alone there was an increase of 7.2 cwt./acre (17.3%).

Mayr *et al.* (1962) found that CCC delayed ripening of grain. Our own observations of moisture contents at harvesting do not indicate that any delay in ripening occurred in these trials.

It is surprising that only in trial 4 was there a significant yield increase purely by virtue of applying extra fertiliser. There was an indication of such an increase in most trials, and this was more pronounced where CCC was applied.

It is not evident from the observations made exactly how the yield increases recorded have come about, since neither the 1000 grain weights, nor the numbers of grain per ear were significantly affected by treatment. Mayr *et al.* (1962) showed that the increase in yield was due to an increase of grain numbers per ear. It is certainly true in these trials that in all cases where there was a significant yield increase there was a higher figure for grains per fertile spikelet, but these differences were not themselves significant. In trial 4 the loss of yield on the untreated plots

was probably largely due to the physical difficulty of harvesting the badly lodged crop, and this factor was probably relevant in trial 3 as well. The yield increases in trial 2, however, are less easy to account for. It is possible that the use of CCC promotes more head-bearing tillers, but unfortunately the scope of this work did not allow such observations to be made.

The conclusions drawn from this work are that in conditions likely to cause lodging in normal wheat crops the use of CCC will keep lodging to a minimum and the losses in yield due to lodging will be reduced. It appears that the degree of protection given by CCC to the crop is such that the level of nitrogen top dressing may be increased by 50% and a resulting increase in yield obtained. These results confirm the work carried out with CCC elsewhere in Europe.

Further data is required to establish the optimum level and time of application of CCC in conjunction with varying levels of nitrogen on different wheat varieties grown on different soil types.

#### Acknowledgements

The authors are indebted to Mr. W. D. Fraser of the Field Testing Station, Wisbech, for assistance with the 1963 trial; also to Dr. E. C. Humphries of Rothamsted Experimental Station, Harpenden, for advice on methods of assessment. Thanks are due to the farmers who allowed these trials to be carried out.

#### References

- JUNG, J. and STURM, H. (1964) Wachstumregulierende Wirkung von Chlorcholinchlorid (CCC). *Landwirtschaftliche Forschung* 17, 1-9.
- LINSER, H., MAYR, H.H. and BODO, G. (1961) Ueber die Wirkung von Chlorcholinchlorid auf Sommerweizen. (Vorläufige Mitteilung). *Bodenkultur* 12, 279-280.
- LINSER, H. and KUHN, H. (1962) Lagerungshemmende bzw. standfestigkeitsstärkende Düngemittel auf Basis von gibberellinaseantagonistischen Stoffen der Gruppe CCC (Chlorcholinchlorid). *Z. Pflanzenernähr., Düng., Bodenkd* 96, 231-247.
- MAYR, H.H., PRIMOST, E. and RITTMAYER, G. (1962) Untersuchungen ueber die Erhoehung der Standfestigkeit von Getreide. I. Feldversuche mit Chlorcholinchlorid zu Winterweizen. *Bodenkultur* 13, 27-45.
- MAYR, H.H. and PRESOLY, E. (1963) Untersuchungen an mit Chlorcholinchlorid (CCC) behandelten Weizenpflanzen. Anatomisch-morphologische Ergebnisse. I. Mitteilung. *Z. Acker-und Pflanzenbau* 118, 109-124.
- TOLBERT, N.E. (1960<sup>a</sup>) (2-Chloroethyl) trimethylammonium chloride and related compounds as plant growth substances. I. Chemical structure and bioassay. *Jour. Biol. Chem.* 235, 475-479.
- TOLBERT, N.E. (1960<sup>b</sup>) (2-Chloroethyl) trimethylammonium chloride and related compounds as plant growth substances. II. Effect on growth of wheat. *Plant Physiol.* 35, 380-385.



## AN INTERIM REPORT ON IOXYNIL FOR WEED CONTROL IN CEREALS

R. Joice and J. Norris  
A. H. Marks and Co. Ltd.

Summary Following primary and secondary screening in the U.S.A., ioxynil has been tested in field trials for three seasons in the U.K. for post emergence weed control in cereals. During the first season it was found that this chemical was highly toxic to Mayweed species. As the control of perennial broadleaved weeds and certain other difficult weeds e.g. *Stellaria media* was not entirely satisfactory, mixtures of ioxynil and phenoxy acids were included in the second seasons trials. The second seasons work confirmed that ioxynil alone was not an adequate cereal herbicide and in consequence the study of mixtures was intensified in the third season. This work indicates that where the problem is Mayweed species with *Polygonum* species, Cleaver or Chickweed an ioxynil/phenoxypropionic mixture is desirable. Where Mayweed species and Hempnettle is predominant an ioxynil/MCPA mixture is probably more suitable.

Throughout the experiments all cereal crops have been found to be tolerant of ioxynil both alone or in mixture with the phenoxy acids.

### INTRODUCTION

In 1962 Amchem Products, Inc., supplied A. H. Marks & Co. Ltd., with a coded formulation of ioxynil (4-hydroxy-3,5-di-iodobenzonitrile). This chemical had shown considerable activity as a post-emergence herbicide in greenhouse screening trials. Susceptible weed species included *Stellaria media*, *Chenopodium album*, *Rumex* species, *Sinapis arvensis*, *Polygonum* species and *Galium aparine*. Cereals were found to be tolerant of the chemical. World wide field testing was initiated in 1962, the U.K. work being confined to small plot exploratory trials in cereals. The control of Mayweed was particularly interesting but the weed spectrum was limited in respect of broadleaved annual and perennial weeds. Extensive trials in 1963 were concentrated on the evaluation of Mayweed control together with a study of broadleaved weed control with ioxynil/phenoxyacid mixtures. In 1964 emphasis was laid on trials with ioxynil mixtures to confirm that these provide a broad spectrum weed control including Mayweed species.

The main aspects of the three year experiments which will be considered in this paper are :

1. Control of Mayweed with ioxynil and ioxynil/phenoxy acid mixtures.
2. The spectrum of weeds controlled with ioxynil and ioxynil/phenoxy acid mixtures.
3. Phytotoxic effects of ioxynil and ioxynil/phenoxyacid mixtures on cereals.

#### METHOD AND MATERIALS

Several formulations have been used over the three years trials the details of which are :

Code	Formulation
1962 Ioxynil A - ACP 62.70	Emulsifiable amine solution containing 2.4 lbs ioxynil/imperial gallon.
1963 Ioxynil B - ACP 62-177	A wettable powder containing 50% w/w ioxynil.
1964 Ioxynil C - ACP 63-303	Lithium salt solution containing 2.4 lbs ioxynil/imperial gallon.
Ioxynil D - AHM 64-2	Sodium ioxynil wettable powder containing 88% ioxynil.
Ioxynil E - AHM 64-4	Sodium salt solution containing 1 lb ioxynil per imperial gallon.

#### Evaluation of Weed Control

Small plots 1/400th or 1/200th acre were laid down in randomised blocks with 2 or 3 replications. Spraying was done with an Oxford Precision Sprayer at a total volume of 50 gal. per acre.

Details of the 1963 and 1964 Trials are summarised in Table I and II.

The code used for the stage of growth of crop and weeds in the following tables is :

<b>Weeds</b>	<b>Crop</b>
C = Cotyledon	L = Leaf
Sd = Seedling	T = Tiller

L = Leaf  
 YP = Young plant  
 MP = Mature plant  
 F = Flowering

ST = Secondary tiller  
 Sh = Shooting  
 J = Jointing  
 FT = Fully tillered

TABLE I  
 Data for Weed Control Evaluation Trials 1963

Site No.	63/1	63/2	63/3	63/4	63/5	63/6
Crop Variety	Wheat Capelle	Barley Proctor	Barley Vada	Wheat Jufy I	Barley Pallas	Oats Blenda
Stage of Growth	FT	4 L	3-4 L	Sh	Sh	Sh
<u>Main Weeds</u>						
Matricaria spp.	Sd-YP	Sd	2 L	NP *	NP	NP
Polygonum aviculare	"	4 L	4 L	NP	NP	NP
Galium aparine	"	NP	NP	NP	NP	NP
Stellaria media	"	6 L	YP	YP	YP	YP
Chenopodium album	"	2 L	4 L	YP	YP	YP
Lamium purpureum	NP	NP	2 L	NP	NP	NP
Veronica chamaedrys	NP	NP	2 L	NP	NP	NP
Cirsium arvense	NP	NP	NP	MP	MP	MP
Senecio vulgaris	NP	NP	NP	YP	YP	YP
Polygonum persicaria	NP	NP	NP	YP	YP	YP

\* NP = Not present

TABLE II  
Crop Data for Weed Control Evaluation Trials - 1964

Site No.	64/1		64/2	64/3	64/4		64/5	64/6	
	1st app	2nd app			1st app	2nd app		1st app	2nd app
Crop	Winter wheat		Winter wheat	Oats	Barley		Winter wheat	Winter wheat	
Variety	Capelle		Capelle	Condor	Pallas		Capelle	Capelle Hybrid 46 Champ-lein	
Stage of growth	6-7L FT 4 T 3 ST		FT	5½L 1 T	4½L FT 2 T		FT	5L-3T J 5L-3T J 6L-4T J	
Matricaria spp.	8 L	YP	YP	Sd	YP- Sd MP		7-9 L	NP*	NP
Polygonum aviculare	1 L	3 L	3 L	3-6 L	2- 3L	6- 7L	3-4 L	NP	NP
Polygonum persicaria	NP	NP	NP	3-4 L	2L	NP	NP	3L	7L
Polygonum convolvulus	NP	NP	NP	4 L	NP	NP	NP	2- 5L	4L
Stellaria media	YP	YP	YP	YP	Sd	F	F	Sd- YP	MP
Galium aparine	NP	NP	YP	NP	NP	NP	Sd	NP	NP
Chenopodium album	C	NP	4 L	2-4 L	Sd	Sd	NP	C- 8L	11L
Galeopsis tetrahit	NP	NP	NP	4-8 L	2L	2L	5L	NP	NP
Cirsium arvense	NP	NP	NP	NP	NP	NP	6-7in.	11L	10L

\* NP = Not present

Weed control was normally assessed by harvesting the weeds from random quadrats and recording the numbers and fresh weight of individual species. The total sample was approximately 10% of the sprayed area. Where this type of evaluation was not possible a visual assessment was carried out on the basis 0 = no control : 10 = 100% control.

### Crop Phytotoxicity

Observations were made throughout all trials each year for adverse effects on the cereals such as height reduction, deformed ears etc. In 1964 two trials were laid down to assess more accurately the effect of excess doses on three varieties of winter wheat, spring wheat and spring barley.

### Phytotoxicity Trial 1

The first trial consisted of four rows of each variety sown in strips with a hand seeder. Ioxynil was sprayed along the strips with a Wagenengin logarithmic sprayer at two stages of growth. Details of growth stages and maximum doses are given in Table III.

TABLE III  
Crop Data and Maximum Tolerated Dosages  
for Crop Phytotoxicity Trial 1

Crop	Stage of Growth		Max. Tolerated Dosages lb ioxynil/ac.	
	1st. app	2nd app	1st app	2nd app
Winter Wheat Hybrid 46	6 L	head	3.1	4.9
	5 T	emerged		
Champlein	5 L	"	"	"
	1 T			
Capelle	6 L	"	"	"
	1 T			
Spring Wheat Opal	4 L	J	3.1	3.1
	1 T			
Jufy I	4 L	"	"	"
	1 T			
Koga II	3½ L	"	"	"
	1 T			
Barley Pallas	5 L	J	3.1	3.1
	5 T			
Maris-Badger	5 L	"	"	"
	5 T			
Cambrinus	5 L	"	"	"
	5 T			

## Phytotoxicity Trial II

Assuming the following dosage levels of the ioxynil formulations to be standard, rates of twice and four times the standard were applied.

### STANDARD TREATMENTS

Ioxynil C	0.5	1b/ac
Ioxynil D	0.5	"
Ioxynil C + MCPA	0.25+0.75	1b/ac
" + 2,4-D	0.25+0.5	1b/ac
" + CMPP	0.25+1.0	1b/ac
" + 2,4-DP	0.25+1.25	1b/ac

The treatments were sprayed across three varieties of winter wheat, spring wheat and spring barley at two stages of growth. Details of the treatments and the growth stages are summarised in Table IV.

TABLE IV  
Crop Data for Phytotoxicity Trial II

Crop	Variety	1st application	2nd application
Spring wheat	Jufy I	4 L - 3 T	7 L-2 T - 1 J
"	Koga II	4 L - 1 T	8 L-2½ T - 1 J
"	Opal	4 L - 2½ T	6 L-2½ T - 1 J
Winter wheat	Hybrid 46	6 L - 3 T	6 L-2½ T - 1 J
"	Champlain	6 L - 3 T	7 L-3 T - 2 J
"	Capelle	5½L - 3 T	7 L-3 T - 1 J
Barley	Cambrinus	4 L - 2½T	8 L-3 T - 1 J
"	Maris Badger	3½L - 2½T	8 L-3 T - 1 J
"	Pallas	4 L - 2½T	8 L-3 T - 1 J

### Effect on Yield

Five trials were programmed in 1964 to assess the effect of the treatments on yield. Plots 3/40th acre were laid down in randomised blocks replicated three or four times. Spraying was done with a special experimental sprayer mounted on a tractor at a total volume of 50 gallons per acre. Harvesting was carried out with a 6 ft. Massey Ferguson Combine taking a single cut equivalent to 1/40th acre. Crop data for the trials is shown in Table V.

TABLE V  
Crop Data for Experiments to Study Effect on Yield 1964

Site	CY/1	CY/2		CY/3	CY/4	CY/5
		1st app	2nd app			
Crop	Wheat	Barley		Wheat	Barley	Barley
Variety	Capelle	Pallas		Capelle	Pallas	Freja
Stage of growth	6 L 2 T 2 J	5 L 3 T	FT 3 J	FT	6 L 2 T	FT 3 J

RESULTS

Evaluation of Weed Control  
1962 Trials

In 1962 the weed control with ioxynil was outstandingly good on Mayweed and other weeds such as Redshank and Fat Hen. The effect was not as good on Knotgrass and Chickweed and the results on perennial weeds e.g. Thistle were poor. Space does not allow for detailed results.

1963/64 Trials  
Control of Mayweed spp

The results for the control of Mayweed are shown in Table VI and VII for 1963 and 1964 respectively. The figures relate to the fresh weight expressed as a % of the untreated control or as visual assessments.

TABLE VI  
Fresh Wt. of Mayweed Species Expressed as % Untreated Control

Treatment	Rate lb/ac	Fresh Wt. of Mayweed species :								
		63/1			63/2			63/3		
		A	B	C	A	B	C	A	B	C
Ioxynil B	0.25	4.7	39.5	0.0	88.1	30.9	-	-	-	-
"	0.5	4.7	1.3	0.0	12.5	1.8	-	10.1	-	-
"	0.75	0.8	0.0	0.0	2.8	0.9	-	5.5	-	-
"	1.0	3.1	0.0	0.0	0.0	0.3	-	1.1	-	-
"	1.5	1.9	0.0	0.0	0.0	0.3	-	-	-	-

A = *M. matricarioides*  
B = *M. chamomilla*  
C = *T. maritimum* spp *indorum*

TABLE VII  
Fresh Wt. of Mayweed Expressed as % of Untreated Control

Treatment	Rate lb/ac	Fresh Wt. of Mayweed spp.					
		64/1*		64/2*	64/4		64/5
		1st app	2nd app		1st app	2nd app	
Ioxynil C	0.25	35.0	0.0	12.5	131.3	85.9	7.2
"	0.5	0.0	0.0	27.5	39.3	10.4	3.6
"	0.75	20.0	0.0	5.0	12.4	17.7	3.2
MCPA	1.25	15.0	0.0	50.0	78.1	39.2	44.1
MCPA+Ioxynil C	0.5+0.5	0.0	0.0	15.0	24.7	3.0	11.7
"	0.75+0.25	0.0	0.0	7.5	62.5	0.0	8.6
"	1.0+0.25	0.0	0.0	5.0	15.1	1.8	2.3
2,4-D	1.0	20.0	0.0	55.0	32.3	26.3	52.2
2,4-D+Ioxynil C	0.25+0.5	0.0	0.0	0.0	12.3	0.0	1.8
"	0.5+0.25	0.0	0.0	22.5	21.3	3.0	12.3
"	0.75+0.25	0.0	0.0	15.0	5.1	2.7	6.8
CMPP	2.25	35.0	0.0	2.5	42.8	35.9	47.0
CMPP+Ioxynil C	0.75+0.5	0.0	0.0	0.0	14.2	2.4	3.8
"	1.0+0.25	0.0	0.0	2.5	38.0	27.5	10.7
"	1.5+0.25	0.0	0.0	0.0	22.8	5.1	3.6
2,4-DP	2.5	0.0	0.0	0.0	19.5	9.9	30.6
2,4-DP+Ioxynil C	1.0+0.5	0.0	0.0	0.0	14.8	1.3	1.4
"	1.25+0.25	0.0	0.0	0.0	31.2	21.7	3.4
"	1.75+0.25	0.0	0.0	0.0	13.4	5.9	1.9
2,4-DP+2,4-D	2.0+0.5	0.0	0.0	0.0	15.9	6.6	18.7
2,4-DP+CMPP	1.25+1.25	0.0	0.0	0.0	25.5	12.6	18.7

Mayweed - Scentsless Mayweed-Tripleurospermum maritimum ssp. inodorum  
 \* visual assessment



Weed Control with Ioxynil and Ioxynil Mixtures

The overall weed control assessments for 1963 and 1964 are shown in Tables VIII and IX respectively. The figures represent the total fresh weight of all species harvested expressed as a % of the untreated control or a visual assessment.

TABLE VIII  
Fresh Wt. of Weeds Harvested Expressed as % of Untreated Control

Treatment	Rate lb/ac	Fresh Wt. of total weeds harvested:					
		63/1	63/2	63/3	63/4	63/5	63/6
Ioxynil B	0.25	41.2	41.7	14.4	-	-	-
"	0.5	43.5	4.2	-	5.0	5.0	-
"	0.75	24.2	2.1	6.5	5.0	5.0	10.0
"	1.0	14.3	1.3	1.2	-	-	-
"	1.5	9.1	0.8	-	-	-	-
"	2.0	-	-	-	-	-	-
MCPA+	0.75+	-	-	-	-	-	-
Ioxynil B	0.25	-	-	-	10.0	5.0	70.0
2,4-D+	0.5+	-	-	-	-	-	-
Ioxynil	0.25	-	-	-	10.0	5.0	-
CMPP	2.25	-	-	37.0	30.0	2.0	55.0
CMPP+	1.25+	-	-	-	-	-	-
Ioxynil B	0.25	-	-	-	15.0	5.0	35.0
2,4-DP	2.5	-	-	-	-	-	-
2,4-DP+	1.25+	-	-	-	-	-	-
Ioxynil	0.25	-	-	-	5.0	5.0	15.0
"	1.5+	-	-	-	-	-	-
"	0.25	-	-	-	5.0	20.0	10.0
2,4-DP+	2.0+	-	-	-	-	-	-
2,4-D	0.5	-	-	-	10.0	2.5	35.0

TABLE IX

Fresh Wt. of Weeds Harvested Expressed as % of Untreated Control

Treatment	Rate lb/ac	Fresh Wt. of total weeds harvested :									
		64/1*		64/2*	64/3	64/4		64/5	64/6		
		1st	2nd		1st	2nd		1st	2nd		
Ioxynil C	0.25	55.0	32.5	12.5	41.6	115.1	30.3	20.0	-	-	
"	0.5	17.5	17.5	27.5	35.9	39.5	25.5	8.8	25.4	38.6	
"	0.75	15.0	15.0	5.0	38.6	21.2	38.1	26.7	-	-	
MCPA	1.25	30.0	20.0	50.0	17.4	72.1	33.9	35.5	12.8	30.4	
MCPA+Ioxynil C	0.5+0.5	10.0	12.5	15.0	6.0	17.8	19.7	28.9	-	-	
"	0.75+0.75	17.5	12.5	7.5	6.1	93.0	7.8	8.6	2.8	8.5	
"	1.0+0.25	20.0	22.5	5.0	5.8	11.9	7.9	9.0	-	-	
2,4-D	1.0	52.5	70.0	55.0	1.8	45.4	53.4	43.1	4.7	0.0	
2,4-D+Ioxynil C	0.25+0.5	22.5	30.0	0.0	45.9	37.2	20.2	20.8	-	-	
"	0.5+0.25	22.5	42.5	22.5	38.3	22.4	33.8	15.7	71.7	16.7	
"	0.75+0.25	22.5	30.0	15.0	43.2	20.3	21.9	29.4	-	-	
CMPP	2.25	17.5	5.0	2.5	30.8	38.3	40.6	41.0	22.6	24.3	
CMPP+Ioxynil C	0.75+0.5	7.5	5.0	0.0	13.7	11.8	0.9	6.9	-	-	
"	1.0+0.25	5.0	10.0	2.5	23.4	30.6	8.6	13.7	12.2	7.6	
"	1.5+0.25	7.5	7.5	0.0	18.4	20.9	4.9	6.2	-	-	
2,4-DP	2.5	5.0	5.0	0.0	41.4	16.0	7.5	21.6	9.6	16.4	
2,4-DP+Ioxynil C	1.0+0.5	5.0	5.0	0.0	19.6	17.9	4.4	1.8	-	-	
2,4-DP+Ioxynil C	1.25+0.25	5.0	5.0	0.0	12.0	28.6	11.7	7.1	16.5	26.7	
"	1.75+0.25	5.0	5.0	0.0	18.0	9.7	3.8	2.3	-	-	
2,4-DP+2,4-D	2.0+0.5	5.0	5.0	0.0	30.7	12.8	5.0	14.9	-	-	

\* visual assessment

1st = 1st application

2nd = 2nd application

Tables X, XI and XII summarise the results obtained on several resistant weeds.

**TABLE X**

Fresh Wt. of *Polygonum persicaria*, *P. convolvulus* *P. aviculare* and *Galeopsis tetrahit* expressed as % Untreated Control

Treatments	Rate lb/ac	1. 64/6		2. 64/3	3. 63/1 64/4		4. 64/3 64/5		
		E	L		E	L			
Ioxynil C	0.5	2.5	7.5	0.7	79.9*	51.6	45.7	30.1	57.4
MCPA+	0.75+								
Ioxynil C	0.25	0.4	1.1	3.3	-	56.6	41.1	26.4	0.0
2,4-D+	0.5+								
Ioxynil C	0.25	0.0	8.0	0.0	-	16.8	14.2	59.7	32.0
CMPP+	1.0+								
Ioxynil C	0.25	0.7	3.3	0.0	-	38.6	52.6	40.5	102.2
2,4-DP+	1.25+								
Ioxynil C	0.25	0.0	1.6	0.0	-	63.7	-	17.9	47.8

N.B. E=1st application 1 = *P. persicaria* 3 = *P. aviculare*  
 L=2nd application 2 = *P. convolvulus* 4 = *G. tetrahit*  
 \* result for ioxynil B

**TABLE XI**

Fresh Wt. of *Stellaria media* and *Galium aparine* expressed as % of Untreated Control

Treatments	Rate lb/ac	S. media				G. aparine	
		64/3	64/4		64/5	64/2	64/5
			E	L			
Ioxynil C	0.5	40.5	23.1	14.1	8.8	30.0	6.3
MCPA+Ioxynil C	0.75+0.25	48.0	99.6	21.2	21.6	15.0	247.2
2,4-D+Ioxynil C	0.5+0.25	15.1	31.3	249.8	96.3	27.5	40.0
CMPP+Ioxynil C	1.0+0.25	0.0	9.0	3.8	11.8	2.5	0.0
2,4-DP+Ioxynil C	1.25+0.25	0.0	11.8	10.6	7.5	0.0	16.7

N.B. E = 1st application  
 L = 2nd application

**TABLE XII**

Fresh Wt. of *Cirsium arvense* Expressed as % of Untreated Control

Treatments	Rate lb/ac	64/5	64/6	
			E	L
Ioxynil C	0.5	37.6	25.4	41.3
MCPA+ioxynil C	0.75+0.25	1.2	1.7	8.3
2,4-D+Ioxynil C	0.5+0.25	2.3	247.5	15.3
CMPP+Ioxynil	1.0+0.25	13.7	44.6	6.5
2,4-DP+Ioxynil C	1.25+0.25	2.9	4.5	26.1

N.B. E = 1st application  
 L = 2nd application

### Polygonum spp

#### Polygonum persicaria

The 1963 results indicated that mixtures with 2,4-DP gave a better control than the ioxynil alone or with phenoxyacetic acids. In 1964 all the mixtures gave satisfactory control. Control appeared to be independent of the stage of growth of the weed.

#### Polygonum convolvulus

In 1963 results suggested that ioxynil controlled *P. convolvulus*. This was confirmed in 1964 when it was found that alone and in mixtures the control was entirely satisfactory.

#### Polygonum aviculare

Ioxynil alone did not give a satisfactory level of *P. aviculare* control over the three year trial period. Good control was obtained with ioxynil/2,4-DP except at site 64/4 where there was a particularly dense infestation. However in this trial a further treatment with an increased amount of 2,4-DP was used (results not recorded here) and the control of Knotgrass was good at both the stages of growth.

The ioxynil/MCPA mixture gave a better result than expected.

#### Stellaria media

The control of *S. media* was variable with ioxynil alone and also with the phenoxyacetic acid mixtures. Both the 2,4-DP and CMPP mixtures gave satisfactory control when applied to *S. media* at various growth stages.

#### Galium aparine

The control attained with ioxynil alone was rather variable. The mixtures with CMPP gave the best control with 2,4-DP mixtures next. Control with the phenoxyacetic mixtures was rather erratic.

#### Galeopsis tetrahit

Control with both ioxynil and ioxynil mixtures was rather variable. MCPA/ioxynil gave in general the best control. However 2,4-DP/ioxynil gave a good result at site 64/3 where the infestation was particularly dense.

## Cirsium arvense

Ioxynil alone gave variable results but in mixture with phenoxyacetic and propionics a satisfactory control was obtained. The MCPA mixture was the most active with the 2,4-DP mixture next.

## Crop Phytotoxicity

### Ioxynil

In 1962 ioxynil A at 1.0 lb/ac caused scorching and stunting of oats, wheat and barley, the relative phytotoxicity being in that order. With ioxynil B in 1963 no deleterious effects could be seen with rates up to 1.5 lb/ac in the various weed control plots. No damage was observed to winter wheat, spring wheat or spring barley in 1964 with ioxynil C and ioxynil D at rates up to 2.0 lb/ac.

### Ioxynil/phenoxyacid mixtures

At the standard dosage level the various phenoxyacid/ioxynil mixtures no effect was seen on winter wheat, spring wheat or spring barley. At twice the standard dosage very occasional ear deformities were noted whereas at four times the rate a considerable height reduction and ear deformities were noticeable.

### Effect on Yield of Cereals

All weights of cereals were corrected to 15% moisture content. These are shown in Table XIII and are expressed in cwt/acre.

TABLE XIII  
Yield of Cereals - 1964

Treatment	Rate lb/ac	CY/1	CY/2		CY/3	CY/4	CY/5
			1st app	2nd app			
Untreated control		47.1	45.0	49.0	-	41.2	33.7
Ioxynil C	0.5	44.3	49.8	47.9	40.0	41.7	-
"	1.0	44.2	47.0	46.2	36.4	41.8	-
"	2.0	44.3	43.9	46.6	40.4	40.8	31.6
Ioxynil E	2.0	-	-	-	-	-	30.7
2,4-DP	2.5	-	-	-	43.4	41.4	-
2,4-DP+C*	1.25+0.25	-	-	-	41.8	42.5	-
"	2.5+0.5	-	41.9	50.1	41.1	41.1	-
CMPP	2.25	-	-	-	42.3	-	-
CMPP+C	1.0+0.25	-	-	-	45.3	-	-
"	2.0+0.5	-	-	-	36.3	-	-
MCPA	1.25	-	-	-	-	40.2	-
MCPA+C	0.75+0.25	41.8	-	-	-	40.4	-
"	1.5+0.5	41.9	-	-	-	41.2	-

\* Ioxynil C

## DISCUSSION

### Control of Mayweed spp

From the three years trials it is now evident that ioxynil effectively controls Mayweed (*T. maritimum* and *M. chamomilla* and *M. matricariodes*) at the seedling and young plant stage. The mixtures of ioxynil with the phenoxy acids were equally as effective over the ranges tested. All the mixtures tested were good although the 2,4-DP mixture tended to give a more consistent result at the different growth stages.

### General Weed Control

The general level of weed control was superior with the ioxynil/phenoxyacid mixtures over the ranges tested than with either component alone. On average the ioxynil/phenoxy propionic mixtures gave a more consistent level of control particularly in the presence of *Stellaria media* and *Galium aparine*. In the presence of *Galeopsis tetrahit* ioxynil/MCPA mixtures were slightly better than the ioxynil/phenoxy-propionic mixtures.

### Crop Phytotoxicity

Ioxynil alone was not phytotoxic to cereals (except with the earlier formulations which have now been discarded), even when used at excessive rates.

When mixed with the phenoxyacids and applied at excessive doses the yields were not significantly affected.

# THE USE OF IOXYNIL AS A SELECTIVE HERBICIDE IN CEREALS IN THE UNITED KINGDOM

by

K. Carpenter and H. J. Terry  
Agricultural Research Station  
May & Baker Ltd., Ongar, Essex

and

J.H. Combellaack and D. Soper,  
May & Baker Ltd. Dagenham, Essex

Summary Field experiments and intensive field trials have been carried out to determine the herbicidal activity and the tolerance of spring and winter cereals to mixtures of ioxynil (4-hydroxy 3,5 di-iodobenzonitrile) with MCPA and mecoprop. Both mixtures controlled the important perennial weeds of cereals, Cirsium arvense and Sonchus arvensis at doses of 6oz ioxynil plus 9oz MCPA per acre, or 6oz ioxynil plus 24oz mecoprop per acre. The ioxynil/mecoprop mixture also controlled a number of other species normally resistant to phenoxyalkanoic acids and/or ioxynil applied on their own. These included Stellaria media, Galium aparine, Tripleurospermum maritimum and the seedling stages of Chrysanthemum segetum. Cereal yield and tolerance experiments were carried out on four varieties of spring wheat, seven varieties of spring and winter barley and five varieties of spring and winter oats. The addition of MCPA increased the risk of cereal ear distortion at early growth stages and the addition of mecoprop caused cereal yield reduction by applications made after the jointing stage. Neither mixture used at rates up to twice that necessary for effective weed control had any significant effect on cereal yield when applied between the two-leaf and the commencement of jointing stages on wheat and oats, and between the 3-4 leaf stage and the commencement of jointing on barley. Extensive field trials carried out on 91 farms throughout the United Kingdom have demonstrated the potentialities of both mixtures as broad spectrum herbicides in winter and spring cereals.

## INTRODUCTION

Laboratory and field experiments carried out in 1963 showed that ioxynil (4-hydroxy 3,5-di-iodobenzonitrile) is active against a wide range of common weeds of cereals (Carpenter et al 1964 a & b). Most annual species are controlled at doses between 3 and 8 oz/ac, but considerably higher rates are necessary for the control of perennial weeds, e.g. creeping thistle, perennial sowthistle, and docks (Terry & Wilson 1964). Control of these species is a requisite for any herbicide to be used on cereals in the UK, and it was therefore considered that if doses of ioxynil were to be kept within an economic range the addition of one of the phenoxyalkanoic acids was necessary for the control of perennial weeds. MCPA is the obvious addition if control of perennials is all that is desired, but mecoprop has the additional advantage of being highly effective against

chickweed and cleavers, both of which are less reliably controlled by lower doses of ioxynil.

The calculation of the proportions of MCPA or mecoprop in such mixtures depends on a knowledge of the minimum effective doses of the individual components for the main weed species under consideration. This information was based on our previous experiments with ioxynil and our own and published work with the phenoxyalkanoic acids. With this information it was possible to assess the activity of one herbicide in relation to another and to estimate the total effect of a mixture. These calculations assumed that the activities of ioxynil and a phenoxyalkanoic acid would be neither antagonistic nor synergistic. Greenhouse and field tests with varying proportions of ioxynil, MCPA and mecoprop confirmed that there was in fact no significant antagonism and satisfied us that the proportions in the mixtures could be based at least on the additive effects of the components. It was therefore decided that two mixtures would be suitable candidates for extensive study in the field. These were a 1:1½ mixture of ioxynil and MCPA, and a 1:4 mixture of ioxynil and mecoprop. The effective dose range of the ioxynil/MCPA mixture was calculated to be between 10 and 15oz total a.e./ac (4oz ioxynil + 6oz MCPA - 6oz ioxynil + 9oz MCPA) and of the ioxynil/mecoprop mixture between 20 and 30oz a.e./ac (4oz ioxynil + 16oz mecoprop - 6oz ioxynil + 24oz mecoprop).

The main object of our work on weed control in 1964 was to test our calculations in practice by showing -

1. That both mixtures controlled perennial weeds.
2. That ioxynil/mecoprop was more effective against chickweed and cleavers than ioxynil alone.
3. That both mixtures were acceptable as broad spectrum herbicides for use in cereals.

The 1963 work had shown that ioxynil used on its own was extremely safe from the point of view of cereal tolerance and yield. The introduction of the phenoxyalkanoic acids (particularly MCPA) was likely to reduce the margin of crop safety at early growth stages. Although as used in the mixtures the rates of MCPA and mecoprop were comparatively low, it was still necessary to establish the crop safety margin of the mixtures and to show that at field rates of use they were as safe as the individual components used alone.

The field work was divided into two scales of activity. The intensive field experiments summarised in Pt. I of this report were designed to provide specific information on the activity of the ioxynil/MCPA and ioxynil/mecoprop mixtures. This included quantitative comparisons of activity against perennial weeds and other species more resistant to ioxynil and the effects of application at different weed growth stages. Separate experiments were set up to study the effects of dose and timing of application on cereal tolerance and yield and to provide evidence for purposes of official approval. Only the main results of this year's work are summarised. Many of the detailed results have yet to be analysed but it is intended to report them in full elsewhere at a later date.

Pt. II of this report describes the extensive field trials carried out throughout the United Kingdom. These trials were designed to determine the farmer acceptability of the two mixtures used as broad spectrum herbicides under commercial conditions of application.



## Part I. Intensive Field Experiments

### METHODS

#### Materials and formulations

The following compounds and formulations were used throughout this work:

<u>Common name</u>	<u>Code No.</u>	<u>Formulation</u>
ioxynil	NPH 1250	Aqueous solution of sodium salt equivalent to 40% w/v of free phenol + wetter and organic solvent.
ioxynil/MCPA	NPH 1300	Aqueous solution of sodium salts equivalent to 20% ioxynil and 30% MCPA + wetter and organic solvent.
ioxynil/mecoprop	NPH 1310	Aqueous solution of sodium salts equivalent to 10% ioxynil and 40% mecoprop + wetter and organic solvent.
MCPA	-	Aqueous solution of potassium salt equivalent to 25% MCPA a.e.
GMPP (mecoprop)	-	Aqueous solution of potassium salt equivalent to 30% mecoprop a.e.

Doses quoted throughout this report are in terms of 'phenol equivalent' for ioxynil and 'acid equivalent' for phenoxyalkanoic acids.

#### Weed control experiments

A logarithmic dosage sprayer was used for all weed control experiments. Plots were 7'6" wide and approximately 100ft. long, enough to give a range of at least three half-dosage distances at a volume rate of 15.7 gal/ac. The starting doses were chosen so that the calculated minimum effective dose of the mixtures and individual compounds would fall approximately in the centre of each plot.

<u>Common name</u>	<u>Starting dose (oz/ac)</u>	<u>to (oz/ac)</u>
ioxynil	12	1.5
ioxynil/MCPA	12 + 18	1.5 + 2.25
ioxynil/mecoprop	12 + 48	1.5 + 6.0
MCPA	24	3.0
mecoprop	48	6.0

All sites were simple randomised layouts with two replicate plots per treatment.

#### Cereal yields

Eight experiments in commercial crops of winter and spring cereals were designed to show how variations of dose and timing of application might affect cereal yields. The three times of application covered the growth stages during which spraying would be likely to take place in commercial practice. The doses chosen for ioxynil formulations were based on the probable minimum and maximum doses of the mixtures likely to be used in the field. The lowest dose for the yield experiments was the probable maximum field dose and the upper dose was calculated as three times the probable minimum field dose, thus -

<u>Mixture</u>	<u>Probable field dose (oz/ac)</u>		<u>Cereal yield dose (oz/ac)</u>	
	<u>Low</u>	<u>High</u>	<u>Low (=high field dose)</u>	<u>High (=3xlow field dose)</u>
ioxynil/MCPA	4 + 6	6 + 9	6 + 9	12 + 18
ioxynil/mecoprop	4 + 16	6 + 24	6 + 24	12 + 48
ioxynil	6	9	9	18

The doses of MCPA and mecoprop were based on the highest dose that occurred in the mixtures and double this. This brought the highest test dose of mecoprop up to 96 oz/ac. This was considered excessive and likely to give unrealistic yields and was reduced to 72 oz/ac. The test doses of MCPA and mecoprop were finalised as:

	<u>Low rate</u>	<u>High rate</u>
MCPA	18 oz/ac	36 oz/ac
mecoprop	36 oz/ac	72 oz/ac

Unsprayed control plots were included and the plots of 0.03 ac. were large enough to be cut with a combine harvester. All sites were randomised block layouts with two replicates. The main plots were split between times of application with all treatments fully randomised within each sub-plot.

#### Cereal variety tolerance

Three sites were sown with recommended varieties of wheat, oats and barley and the treatments applied at four growth stages to cover the extremes of timing likely to be met in commercial practice. The compounds and doses were the same as used in the yield experiments. Varieties studied were -

Wheat: Koga II, Jufy, Opal and Atle.

Barley: Maris Badger, Maris Baldric, Cambrinus, Rika, Pallas, Proctor.

Oats: Blenda, Forward, Astor, Condor.

All sites were randomised block layouts with two replicates. Main plots were split between times of application. Sub-plots were further sub-divided into varieties and these again into treatment sub-sub plots.

#### Assessment methods

Weed control Preliminary assessments were made at some sites noting the point in each plot where complete control was achieved. At all sites weed counts were made using a  $\frac{1}{2}$  sq.yd. quadrat at fixed points down the logarithmically sprayed plots. The percentage control was calculated and the resultant figures used to plot probit regression lines. Activities of the treatments were then compared by their LD90's derived from the regression lines.

Cereal yields Observations of leaf scorch were made 3 and 10 days after each application time, using the standard scoring system (Terry & Wilson 1964). Each plot was harvested with a combine harvester and the yields recorded and corrected to a moisture content of 15%.

Cereal tolerance Observations of crop scorch were made at 3 and 10 days after spraying, and later each plot was examined for the presence of abnormal ears. All abnormalities, however, slight, were recorded. Some abnormal ears were recorded on unsprayed plots but these were usually missing grains, probably attributable to bird damage. The larger amounts of true distortion found on some treated plots were of the type usually associated with the effects of MCPA or 2,4-D. A measure of the vigour of the crop was obtained by measuring the mean height of the crop in each plot and the number of ears per sq.yd.

## RESULTS

### Weed control

The results shown in Table I illustrate the effects of the addition of the phenoxyalkanoic acids to ioxynil for the control of the more important species for which we have sufficient information. The weeds are arranged in alphabetical order of species and in ascending order of growth stage. This allows some demonstration of the effect of growth stage on weed control. A comparison of the effects of volume of application are given in Table 2. Consideration of the full spectrum of weeds controlled by the mixtures under farm conditions is best left for the discussion of the extensive field trials in Part II of this report.

Table 1. Control of weeds in cereals with mixtures of ioxynil with MCPA or mecoprop.

Species	No. of sites	Growth stage at spraying	Dose (oz/ac) to achieve 90% control				
			ioxynil	ioxynil + MCPA(1:1½)	ioxynil + CMPP(1:4)	MCPA	CMPP
Atriplex patula	1	Seedl 2-4 lvs	3.0	<u>1.5 + 2.2</u>	<u>1.5 + 6.0</u>	9	12
Brassica nigra	2	Seedling	6.0	<u>3.5 + 4.3</u>	<u>3.0 + 12.0</u>	9	18
Chenopodium album	2	Seedl 2-4 lvs	3.0	<u>1.5 + 2.3</u>	<u>1.5 + 6.0</u>	6	12
Chrysanthemum segetum	2	Cotyledon	7.0	<u>5.5 + 8.3</u>	<u>4.0 + 16.0</u>	>24	>48
	2	Seedl 2-4 lvs	11.0	<u>12.0 + 18.0</u>	<u>9.0 + 36.0</u>	>24	>48
	1	YP 4-8 lvs	-	-	<u>14.0 + 56.0</u>	-	>48
Cirsium * arvense	2	6 - 12"	7.0	2.5 + 3.7	2.0 + 8.0	8	-
Galeopsis tetrahit	2	Seedl 2-6 lvs	6.5	6.5 + 9.0	6.5 + 24.0	36	60
Galium aparine	2	Overwintered young plant	23.0	23.0 + 34.5	<u>11.0 + 44.0</u>	>24	62
Lamium spp.	1	Seedl 2-4 lvs	2.0	3.0 + 4.5	3.0 + 12.0	>24	-
Papaver rhoeas	1	Seedl 2-4 lvs	8.0	3.0 + 4.5	1.5 + 6.0	12	36
Polygonum aviculare	1	Seedl 2-6 lvs	10.0	<u>4.0 + 6.0</u>	<u>3.0 + 12.0</u>	18	24
	1	YP 4-8 lvs	12.0	<u>4.0 + 6.0</u>	<u>12.0 + 48.0</u>	>24	>48
	1	MP Flowering	12.0	<u>12.0 + 18.0</u>	<u>12.0 + 48.0</u>	>24	>48
Polygonum convolvulus	1	Cotyledon	6.0	<u>1.5 + 2.3</u>	<u>4.5 + 18.0</u>	>24	24
	3	Seedl 2-4 lvs	3.3	2.4 + 3.6	1.5 + 6.0	(18)	24
	1	YP 6-8 lvs	4.0	<u>3.0 + 4.5</u>	3.0 + 12.0	>24	36
Polygonum lapathifolium	5	Seedl to 4 lvs	3-6	<u>1.8 + 2.7</u>	<u>2.5 + 10.0</u>	>24	40- >48

\* Dose for minimum acceptable control. Plant height reduced by 75%, growing points checked and leaves chlorotic.

Doses underlined indicate where mixture has given better control than ioxynil alone.

Table 1 (contd.)

Species	No. of sites	Growth stage at spraying	Dose (oz/ac) to achieve 90% control				
			ioxynil	ioxynil + MCPA(1:1½)	ioxynil + CMFP(1:4)	MCPA	CMFP
Raphanus raphanistrum	1	Seedl 2-4 lvs	6.0	<u>3.0 + 4.5</u>	<u>1.5 + 6.0</u>	6	18
Sonchus arvensis*	1	6"	17.0	<u>8.0 + 12.0</u>	<u>5.0 + 20.0</u>	10	-
	1	3"	5.1	<u>1.4 + 2.1</u>	<u>1.3 + 5.2</u>	11	-
Spergula arvensis	2	Seedl 2-4 lvs	>12.0	<u>9.0 + 13.5</u>	<u>11.0 + 44.0</u>	>24	>48
Stellaria media	1	Cot. - Seedl	16.0	<u>10.0 + 15.0</u>	<u>5.6 + 22.4</u>	>24	12
	2	Seedl 2-6 lvs	8-12	<u>4.2 + 6.3</u>	<u>4.2 + 16.8</u>	>24	22
	1	YP 6-8 lvs	8.0	<u>3.5 + 5.2</u>	<u>2.2 + 8.8</u>	>24	12
Tripleurospermum maritimum	1	Seedl 4-6 lvs	12.0	<u>10.0 + 15.0</u>	<u>6.0 + 24.0</u>	36	70
	2	YP 8 lvs	14.0	<u>9.0 + 13.5</u>	<u>10.0 + 40.0</u>	40	70
	1	MP	14.5	<u>15.0 + 22.5</u>	<u>12.0 + 48.0</u>	-	-
Urtica urens	1	Seedl 3 lvs	>12.0	<u>3.0 + 4.5</u>	<u>6.0 + 24.0</u>	18	48
Viola spp.	3	Seedl 4 lvs	>12.0	9.0 + 13.5	<u>12.0 + 48.0</u>	>24	48

\* Dose for minimum acceptable control. Plant height reduced by 75%, growing points checked and leaves chlorotic.

Doses underlined indicate where mixture has given better control with less ioxynil than has ioxynil alone.

Table 2. Effect of volume of application.

Species	Site No.	Growth stage	Minimum dose (total a.e) required for 90% control			
			Ioxynil		Ioxynil/MCPA(1:1½)	
			5 gal.	10 gal.	5 gal.	15 gal.
Stellaria media	86/1	1" overwintered	11.0	9.0	>30.0	14.0
		4" overwintered	>12.0	>12.0	>30.0	25.0
Raphanus raphanistrum	86/2	Cotyledon	2.6	2.0	9.7	2.7
Tripleurospermum maritimum	86/2	Cotyledon	1.9	1.2	<3.7	2.7
		1" overwintered	>12.0	>12.0	>30.0	29.0

#### Crop tolerance and yield

Counts of abnormal ears showed that both wheat and barley were susceptible to damage at the early growth stages by some treatments. Full analyses of the results have been carried out, but the most applicable are those of the second and third order interactions. Tables 3 and 4 are abridged versions of the second order interactions of compound x rate x time

showing the relation between applications of the highest rates of the mixtures at four growth stages and the incidence of abnormal ears. These results are expanded in Table 5 to show the effects by all treatments on individual varieties at the two most susceptible growth stages. Although some abnormalities occurred on oats these were only slight and were not connected with any particular compound or dose rate.

Yields were obtained from 8 sites of commercial cereal crops. The full results for each crop are set out in Table 6.

Table 3. Spring wheat - Effect of applications of ioxynil, MCPA and mecoprop on the incidence of abnormal ears.

Growth stage at application	% abnormal ears at given dose (oz/ac)					
	ioxynil		ioxynil/MCPA	ioxynil/CMPP	MCPA	CMPP
	9	18	12 + 18	12 + 48	18	36
1-2 leaves	5.5	4.7	10.0	9.1	10.3	4.9
4-5 leaves	5.1	6.6	6.3	4.5	6.4	5.2
5-7 leaves	6.4	6.0	4.9	5.1	7.1	6.7
Jointing	6.1	7.0	6.0	6.7	5.6	5.0

Each figure is the mean of 8 plots derived from 2 replicates of each of 4 varieties.

L.S.D. between any treatment mean and control when  $P = 0.01 = 5.7$   
 Mean for control (unsprayed) plots = 5.5

Table 4. Spring barley - Effect of applications of ioxynil, MCPA and mecoprop on the incidence of abnormal ears.

Growth stage at application	% abnormal ears at given dose (oz/ac)					
	ioxynil		ioxynil/MCPA	ioxynil/CMPP	MCPA	CMPP
	9	18	12 + 18	12 + 48	18	36
2 leaves	5.6	4.8	16.7	2.9	9.9	8.3
3-4 leaves	3.8	3.4	7.5	6.1	5.1	3.6
5-7 leaves	2.8	2.5	3.1	2.6	3.3	2.5
Jointing	2.8	2.8	2.6	2.8	2.1	1.9

Each figure is the mean of 12 plots derived from 2 replicates of each of 6 varieties.

L.S.D. between treatment means and control when  $P = 0.01 = 4.2$   
 Mean for control (unsprayed) plots = 2.2

Table 5. Spring wheat and spring barley. Effect of dose on the incidence of abnormal ears in different varieties at the first two growth stages.

Growth stage at application	Variety	% distorted ears at given dose (oz/ac) *										Control
		ioxynil		ioxynil/MCPA		ioxynil/GMPP		MCPA		GMPP		
		9	18	6+9	12+18	6+34	12+48	18	36	36	72	
<b>Spring wheat:</b>												
1-2 leaf	Koga II	6	4	8	9	4	7	8	9	3	2	4
	Jufy	6	7	10	13	9	15	17	18	7	4	7
	Opal	7	5	12	12	6	9	6	9	6	6	5
	Atle	4	4	4	6	7	6	10	7	4	4	6
4-5 leaf	Koga II	4	6	3	5	5	3	4	5	5	5	4
	Jufy	9	11	11	9	9	6	8	8	7	11	7
	Opal	5	5	5	4	4	6	6	5	5	5	5
	Atle	2	6	4	7	3	3	7	8	4	2	6
<b>Spring barley:</b>												
2 leaf	Maris Badger	3	3	7	13	2	2	4	18	5	5	2
	Maris Baldric	9	5	8	15	5	4	11	48	7	6	2
	Proctor	7	8	12	18	4	2	9	23	9	10	3
	Cambrinus	5	4	9	16	5	3	13	46	12	15	3
	Pallas	5	6	19	45	5	6	11	69	11	6	3
	Rika	5	2	5	20	5	2	12	46	6	6	3
3-4 leaf	Maris Badger	4	4	9	19	2	19	14	11	5	12	2
	Maris Baldric	4	4	2	6	4	4	4	5	5	4	2
	Proctor	4	3	4	7	2	3	3	6	4	4	3
	Cambrinus	2	4	1	4	1	3	4	3	3	4	3
	Pallas	5	4	3	6	3	4	3	5	3	4	3
	Rika	3	3	5	5	2	4	3	3	3	3	3

Least significant difference between treatment means and relevant control when  $P = 0.05$ : Wheat - 9.0  
Barley - 8.0

\* Figures to nearest whole number

**Table 6.** Effect on cereal yields of applications of mixtures of ioxynil with MCPA or CMPP at three growth stages. (Yield ~~wt./acre~~)

Crop & Variety	Growth stage	Compound and dose (oz/ac)									
		Ioxynil		Ioxynil + MCPA		Ioxynil + CMPP		MCPA		CMPP	
		9	18	6+9	12+18	6+24	12+48	18	36	36	72
W. wheat	7-10 lvs	37	37	35	36	38	35	36	37	34	34
Capelle	10-13 lvs	35	30	31	29	32	31	31	31	31	28
	Jointing	33	32	33	26	28	21	32	33	33	30
LSD (P = 0.05) between treatments 6.0, between treatments & controls 4.0 Control mean = 33.4 C of V = 8%. Control weed no/sq.yd. = 31.											
S. wheat	2-3 lvs	34	35	33	33	34	34	31	32	34	33
Jufy	6-7 lvs	35	34	35	35	34	34	34	36	35	36
	Jointing	33	30	33	31	31	27	32	32	33	31
LSD (P = 0.05) between treatments 4.0, between treatments & controls 3.0 Control mean = 32.8 C of V = 8%. Control Weed no/sq.yd. = 102.											
W. oats	8 lvs	31	31	35	31	34	32	35	33	33	37
Penrhyn	7-10 lvs	32	32	36	32	32	31	39	34	33	33
	Jointing	28	25	34	29	34	26	39	36	34	34
LSD (P = 0.05) between treatments 7.0, between treatments & controls 5.0 Control mean = 35.3 C of V = 10%. Control weed no/sq.yd. = Weed free.											
W. barley	8-9 lvs	31	30	30	33	28	31	31	32	31	32
Dea	10-12 lvs	29	26	25	30	30	29	27	27	27	26
	Jointing	26	27	29	29	29	26	28	30	31	26
LSD (P = 0.05) between treatments 5.0, between treatments & controls 4.0 Control mean = 28.9 C of V = 10%. Control weed no/sq.yd. = Weed free.											
S. barley	3-4 lvs	30	27	30	30	28	31	28	26	24	32
Proctor	6-7 lvs	26	25	26	28	28	30	30	32	29	27
	Jointing	27	18	22	25	19	21	23	23	22	25
LSD (P = 0.05) between treatments 10.0, between treatments & controls 7.0 Control mean = 28.2 C of V = 18%. Control weed no/sq.yd. = 31.											
S. barley	3-4 lvs	31	32	27	30	30	29	28	30	29	27
Pallas	7-8 lvs	25	29	29	30	30	27	29	30	29	29
	Jointing	26	26	28	27	25	21	30	27	27	25
LSD when P = 0.05 between treatments 4.0, between treatments & controls 3.0 Control mean = 31.6 C of V = 5%. Control weeds no/sq.yd. = 83.											
S. barley	3-4 lvs	35	37	36	37	43	37	37	36	36	36
Rika	6-8 lvs	37	36	38	36	38	35	38	37	38	37
	Jointing	34	24	34	33	34	29	35	34	35	34
LSD (P = 0.05) between treatments 4.0, between treatments & controls 3.0 Control mean = 36.3 C of V = 6%. Control weeds no/sq.yd. = Weed free.											

## DISCUSSION

The results in Table 1 show that in general our choice of compound to supplement the activity of ioxynil has been verified in practice. The addition of relatively small doses of MCPA or mecoprop have reduced the dose of ioxynil required to control creeping thistle and sowthistle from 16oz to about 8oz/ac. Similarly we have demonstrated that adequate control of chickweed and cleavers can be achieved with rates of the ioxynil/mecoprop mixture containing less than 8oz/ac. ioxynil. Apart from the control of those particular species for which the mixtures were designed, there has been an improved and more uniform control of many of the other common weeds of cereals. In particular, mayweeds and corn marigold, both resistant to MCPA, were controlled in the seedling stage by doses of both mixtures containing less than 6oz/ac ioxynil and 9oz MCPA or 24oz mecoprop. Even at more advanced stages, practical control was achieved with comparatively low doses of the mixtures. The optimum stage for the control of most species is when the plants have developed from two to eight true leaves. Control of most weeds fell off rapidly after the 8 true leaf stage, and it was also found that the control of large overwintered weeds was erratic. The resistance of weeds at advanced growth stages has already been reported (Carpenter *et al* 1964). Last year's work showed that the activity of ioxynil was not affected by changes in volume of application over the range 18 to 65 gal/ac (Terry & Wilson 1964). At both sites at which applications of 5 and 15 gal/ac were compared, the activity of ioxynil was less at the 5 gal/ac rate (Table 2).

Measured in terms of the dose of ioxynil required, the 1:4 mixture of ioxynil and mecoprop was generally more active than the 1:1½ ioxynil/MCPA mixture. Both mixtures are, however, effective broad spectrum herbicides and the choice between them would depend both on the weeds present and economic considerations. The ioxynil/mecoprop mixture at doses up to 30 oz/ac (6oz ioxynil + 24oz mecoprop) would have the advantage over the MCPA mixture in that it would give better control of chickweed and cleavers and corn marigold.

### Crop tolerance and yield

Cereals are very tolerant to ioxynil alone and between the 2 leaf and jointing stages applications of up to 32 oz/ac have no effect on cereal quality and yield (Terry & Wilson 1964). The addition of MCPA caused a marked increase in the risk of ear distortion at the early growth stages of wheat (Table 3) and especially barley (Table 4). The weed control experiments have shown that the rate of use of the ioxynil/MCPA mixture would be 15oz/ac (6oz ioxynil + 9oz MCPA). On wheat a dose of twice this amount containing 18 oz/ac MCPA caused no more distortion than the 18 oz/ac MCPA treatment alone, and it is considered that the safety margin is sufficiently high to allow applications of 15 oz ioxynil/MCPA from the 2 full leaf stage (Table 5). On barley the combination of ioxynil and MCPA caused considerably more distortion than either ioxynil or MCPA alone. A dose of 15 oz/ac caused distortion on all varieties at the 2-leaf stage, but at the 3-4 leaf stage there is sufficient safety margin.

The addition of mecoprop to ioxynil did not cause any significant increase in the incidence of abnormal ears and the ioxynil/mecoprop



mixture was safely applied to spring wheat and spring barley at doses up to 60 oz/ac (12 oz ioxynil + 48oz mecoprop) or double the effective rate for weed control. Four varieties of oats were examined and although some abnormal ears were found, these could not be attributed to any particular treatment, variety or growth stage. On the sites used to measure the effect on cereal yield, applications made at the jointing stage or later caused some crop reduction on all cereal types (Table 6). This was most noticeable on treatments containing mecoprop, the addition of ioxynil causing greater yield losses than with mecoprop on its own. Neither ioxynil/MCPA nor ioxynil/mecoprop when applied at the fully tillered stage at 30 oz/ac and 60 oz/ac respectively (double their effective field doses) produced yields significantly different from MCPA or mecoprop applied at their normal rates of use. The small differences in yield between treatments and unsprayed control plots are due in part to small variations in weed control between different treatments.

## Part II. Extensive field trials.

### APPLICATION AND METHOD OF ASSESSMENT

A total of 91 sites were sprayed; 28 in Southern England, 24 in the Midlands, 23 in East Anglia, 14 in Scotland and 2 in Northern Ireland. At each site the following rates of application were used:

- a. 4oz ioxynil + 6oz MCPA per acre
  - b. 6oz ioxynil + 9oz MCPA per acre
  - c. 4oz ioxynil + 16oz mecoprop per acre
  - d. 6oz ioxynil + 24oz mecoprop per acre
  - e. Standard product (this varied with the particular weed problems and included standard commercial brands of: MCPA, 2,4-D, mecoprop, dichlorprop, dinoseb and MCPA/TBA, MCPA/mecoprop and MCPA/dichlorprop mixtures.)
- } Formulation code NPH/1300  
} Formulation code NPH/1310

Each treatment, unreplicated, was applied to approximately one acre of the crop in adjacent strips by the farmer's low-volume spray machine. In general, weeds were treated at the seedling stage, spring-sown cereals between the two-leaf and five-leaf stages, and winter-sown cereals at full tillering. In order to cope with the large number of trials it was necessary to extend the spraying season as much as possible, hence it was not feasible to treat every crop at the best time - a situation not unrelated to commercial practice.

Weed assessments were made before and after spraying by a fixed quadrat method, thus eliminating the need for a control, since it was impracticable to arrange for farmers to leave a large area of cereals unsprayed. The first assessment was carried out on the day of spraying or 2-3 days earlier, and the second assessment was made 14-21 days after spraying. The total number of each weed species present, and their growth stages, in four half-square-yard quadrats per treatment, was recorded for both assessments.

## RESULTS

### Effect on cereal varieties

Six varieties of winter cereals, mostly Capelle wheat, and fourteen varieties of spring cereals, dominated by Proctor barley, were sprayed. Leaf scorch was recorded at three sites on winter crops and ten sites in spring crops where 6oz ioxynil plus 24oz mecoprop was applied, and also at two spring sites where 4oz ioxynil plus 16oz mecoprop was used (see Table 7). It was not possible to relate this scorch to the volume of water in which the two treatments were applied (see Table 10).

In every case, crop scorch was soon outgrown, and no ear distortion or other phytotoxic symptom was observed.

### WEED CONTROL

Weed assessments were completed satisfactorily at 75 sites, and 73 different weed species were recorded; those occurring at five or more sites are listed in Table 8. The most common weed encountered was chickweed, which occurred in all winter cereals and also in most spring cereals. Corn spurrey, scarlet pimpernel, bugloss and corn marigold were found only in spring corn; parsley piert and mouse-ear chickweed were more prevalent in winter crops. The percentage weed control was calculated for each weed at 75 sites from the assessments made before and after spraying. The degree of control, in three categories, achieved at each site for each prevalent species, is summarised in Table 9.

Table 7. Effect on cereal varieties.

Crop	Variety	No. of sites	Sites with ioxynil/CMPP scorch	
			4 + 16 oz/ac	6 + 24 oz/ac
W. wheat	Capelle	20	0	2
	Hybrid 46	1	0	1
	Prof. Marechal	1	0	0
W. barley	Pioneer	2	0	0
	Dea	1	0	0
W. oats	Powys	1	0	0
S. wheat	Opal	4	1	1
	Jufy 1	2	0	0
S. barley	Proctor	26	1	3
	Pallas	7	0	2
	Ymer	6	0	0
	Cambrinus	5	0	0
	Rika	5	0	1
	Vada	2	0	1
	Union	2	0	1
	Bonus	1	0	0
	N59	1	0	0
	Freja	1	0	0
	Europa	1	0	0
S. oats	Condor	2	0	1
TOTAL		91	2	13

Table 8. Weed occurrence.

Weed	Latin name	No. of sites where present			
		Spring cereal (poss. max. = 53)	Winter cereal (poss. max. = 22).	Total (poss. max. = 75)	%
Chickweed	<i>Stellaria media</i>	47	22	69	92
Knotgrass	<i>Polygonum aviculare</i>	47	14	61	81
Speedwells	<i>Veronica</i> spp.	39	18	57	76
Mayweed (Scentless)	<i>Tripleurospermum maritimum</i> spp. <i>inodora</i>	34	18	52	69
Black Bindweed	<i>Polygonum convolvulus</i>	40	7	47	63
Fat Hen	<i>Chenopodium album</i>	38	4	42	56
Charlock	<i>Sinapis arvensis</i>	33	7	40	53
Cleavers	<i>Galium aparine</i>	28	11	39	52
Pansy	<i>Viola</i> spp.	24	10	34	45
Poppy	<i>Papaver rhoeas</i>	21	11	32	43
Groundsel	<i>Senecio vulgaris</i>	19	8	27	36
Redshank	<i>Polygonum persicaria</i>	21	5	26	35
Annual Sowthistle	<i>Sonchus oleraceus</i>	20	2	22	29
Creeping Thistle	<i>Cirsium arvense</i>	17	5	22	29
Shepherd's Purse	<i>Capsella bursa-pastoris</i>	13	8	21	28
Deadnettle (Red)	<i>Lamium purpureum</i>	12	7	19	25
Dock	<i>Rumex</i> spp.	12	6	18	24
Buttercup (Corn)	<i>Ranunculus arvensis</i>	9	8	17	23
Field Bindweed	<i>Convolvulus arvensis</i>	14	2	16	21
Fumitory	<i>Fumaria officinalis</i>	12	4	16	21
Orache	<i>Atriplex patula</i>	14	2	16	21
Forget-me-not	<i>Myosotis arvensis</i>	9	5	14	19
Runch	<i>Raphanus raphanistrum</i>	12	1	13	17
Corn spurrey	<i>Spergula arvensis</i>	12	0	12	16
Parsley Piert	<i>Aphanes arvensis</i>	3	8	11	15
Scarlet Pimpernel	<i>Anagallis arvensis</i>	10	0	10	13
Coltsfoot	<i>Tussilago farfara</i>	8	1	9	12
Mouse-ear Chickweed	<i>Cerastium arvense</i>	2	7	9	12
Nipplewort	<i>Lapsana communis</i>	7	1	8	11
Campion (White)	<i>Silene alba</i>	5	2	7	9
Hempnettle	<i>Galeopsis tetrahit</i>	4	3	7	9
Bugloss	<i>Lycopsis arvensis</i>	6	0	6	8
Wild Parsnip	<i>Pastinaca sativa</i>	4	2	6	8
Corn Marigold	<i>Chrysanthemum segetum</i>	5	0	5	7
Pale persicaria	<i>Polygonum lapathifolium</i>	4	1	5	7

Table 9. Response of prevalent weeds to ioxynil/MCPA and ioxynil/CMPP

These figures represent the number of sites at which there were at least 16 weeds/2 sq.yds. in at least one plot. A weed species is included only if it satisfied this criterion at three or more sites.

	% Control:	IOXYNIL + MCPA						IOXYNIL + CMPP						STANDARDS		
		4oz + 6oz			6oz + 9oz			4oz + 16oz			6oz + 24oz			91-100	71-90	<70
		91-100	71-90	<70	91-100	71-90	<70	91-100	71-90	<70	91-100	71-90	<70	91-100	71-90	<70
Annual Sowthistle	S	4	.0	1*	6	0	0	3	2	1	5	0	1	3	0	2
Deadnettle	S&W	2	0	2	1	1	2	2	0	2	2	0	2	2	1	0*
Black Bindweed	S	15	2	2*	17	2	2	15	4	2	18	1	2	13	4	2*
Buttercup (Corn)	S	2	1	1	3	0	1	2	1	1	1	2	1	1	1	2
Charlock	S	12	0	0	12	0	0	12	0	0	12	0	0	10	1	0*
Chickweed	S	12	6	11*	11	13	7	14	9	8	21	3	7	10	10	9*
	W	0	1	14*	0	2	14	1	5	10	3	8	5	4	5	7
Cleavers	S	0	2	2	2	1	1	1	2	1	3	1	0	0	1	2*
	W	2	0	2	2	0	2	3	0	1	2	1	1	1	0	3
Corn Marigold	S	0	0	3*	0	0	4	0	0	4	0	1	3	0	0	3*
Corn Spurrey	S	1	1	4	3	0	3	1	1	4	2	0	4	0	0	5*
Creeping Thistle	S	0	1	2	1	0	2	1	1	1	1	0	2	1	1	1
Fat Hen	S	21	0	0*	23	0	1	24	0	0	24	0	0	18	0	1*
Field Bindweed	S	4	0	0	3	1	0	3	1	0	2	2	0	2	0	2
Forget-me-not	S&W	3	1	0	3	1	0	3	0	1	4	0	0	2	2	0
Fumitory	S	2	0	1	2	0	1	2	1	0	3	0	0	3	0	0
Hempnettle	S&W	2	0	3	1	2	2	1	1	3	1	1	3	2	1	2
Ground Ivy	S	2	0	1	2	1	0	3	0	0	3	0	0	2	0	1

S = Spring  
W = Winter

\* = Variation in treatment total site nos. due to absence of the weed species before spraying on certain treatments.

Table 9 (contd.) Response of prevalent weeds to ioxynil/MCPA and ioxynil/CMPP.

	% Control	IOXYNIL + MCPA						IOXYNIL + CMPP						STANDARDS		
		4oz + 6 oz			6oz + 9oz			4oz + 16oz			6oz + 24oz			91-	71-	<70
		91- 100	71- 90	<70	91- 100	71- 90	<70	91- 100	71- 90	<70	91- 100	71- 90	<70	91- 100	71- 90	<70
Groundsel	S	2	0	1	3	0	0	3	0	0	3	0	0	3	0	0
Knotgrass	S	13	7	9	17	9	6	11	13	6*	21	6	5	10	6	11*
	W	5	0	0	4	1	0	3	1	1	4	1	0	3	1	1
Mayweed (Scentless)	S	9	3	5	10	4	3	8	5	4	10	4	3	3	4	9*
	W	3	2	5	2	3	5	5	2	3	5	4	1	3	3	4
Nipplewort	S	1	0	2	1	2	0	3	0	0	3	0	0	1	0	1*
Pansy	S	0	2	4	0	5	1	0	2	4	1	5	0	1	2	2*
Parsley Piert	W	0	3	2	1	1	4	1	3	2	5	0	1	0	0	5*
	S	6	1	0	6	0	0*	6	0	1	7	0	0	6	0	1
Poppy	W	2	0	3	3	2	1	5	0	1	6	0	0	4	1	1
	S	5	1	0	5	1	0	4	1	1	4	1	1	1	3	2
Redshank	S	2	1	0	3	0	0	2	0	0	3	0	0	0	1	1*
Scarlet Pimpernel	S	4	1	1	5	0	0	6	0	0	6	0	0	5	1	0
Shepherd's Purse	S	4	1	1	5	0	0	6	0	0	6	0	0	5	1	0
	W	1	1	1	2	0	1	3	0	0	1	3	0	1	0	1*
Small Spurge	S	1	1	1*	2	1	1	2	0	2	4	0	0	0	0	3*
Speedwells	S	13	2	6	15	5	1	11	7	3	15	2	4	4	8	6*
	W	1	2	6*	1	5	5	3	4	3*	6	1	3*	0	4	6*

S = Spring  
W = Winter

\* = Variation in treatment total site nos. due to absence of the weed species before spraying on certain treatments.

Table 10 Volume of water applied.

Gallons of water/acre	Total No. of sites	No. of sites		No. of sites where 6oz ioxynil + 24oz CMPP caused scorch	
		Spring cereals	Winter cereals	Spring cereals	Winter cereals
10	2	2	0	1	0
14	1	1	0	1	0
15	8	6	2	2	0
16	2	1	1	0	0
18	4	4	0	1	0
20	58	41	17	4	2
24	1	0	1	0	0
25	8	6	2	1	1
30	7	4	3	0	0
TOTAL	91	65	26	10	3

### DISCUSSION

#### Weed control in spring cereals

The following weeds were controlled from the seedling to young plant stages by all five treatments: charlock, fat hen, fumitory, groundsel, poppy, shepherd's purse. Both rates of the ioxynil/MCPA and ioxynil/mecoprop mixtures controlled the following weeds between the same growth stages, but variable results were obtained with the standards: annual sowthistle, forget-me-not, redshank, scarlet pimpernel. At the same weed growth stage, 6oz ioxynil + 9oz MCPA and 6oz ioxynil + 24oz mecoprop gave better control than 4oz ioxynil + 6oz MCPA and 4oz ioxynil + 16oz mecoprop and the standards, of the following weeds: black bindweed, corn buttercup, cleavers, field bindweed, scentless mayweed and speedwells, and spurrey at the seedling stage only. The ioxynil/mecoprop mixture gave better control than either the ioxynil/MCPA or standards of the following weeds: chickweed, knotgrass, nipplewort, pansy and small spurge, and affected corn marigold at the seedling stage only.

#### Weed control in winter cereals

Only 6oz ioxynil + 24oz mecoprop of the ioxynil formulations gave satisfactory control of weeds occurring in autumn corn cereals. Weeds controlled included parsley piert, poppy and shepherd's purse; of these only poppy was satisfactorily controlled by the standards. Some of the more important weeds such as chickweed and speedwells were partially resistant to all treatments.

### CONCLUSIONS

The spectrum of weeds controlled by ioxynil can be broadened by the addition of MCPA or mecoprop. The addition of 9oz MCPA or 24oz mecoprop to 6oz ioxynil ensures reliable shoot suppression of perennial weeds of cereals. The mixture with mecoprop also gives a more uniform control of certain annual species than is possible with ioxynil alone. The addition of the phenoxyalkanoic acids reduces the crop safety margin as compared with ioxynil alone, but both mixtures can be safely applied to wheat, oats and barley between the 3-leaf and the jointing stages at doses up to twice that necessary for weed control.

Under farm conditions of use, mixtures of 6oz ioxynil + 9oz MCPA per acre or 6oz ioxynil + 24oz mecoprop per acre have given good control of the principal annual and perennial weeds in spring sown cereal crops. The control of large overwintered weeds in autumn sown crops has been less reliable.

### ACKNOWLEDGMENTS

We should like to acknowledge the part played by Mr. C. W. Wilson in supervising the conduct of the intensive experiments in the field, and the co-operation of many farmers throughout Britain who provided trial sites. Thanks are also due to Mr. D. Mackenzie for his work on the statistical analyses and to several other colleagues for their practical assistance during the course of the work.

### REFERENCES

- Carpenter K, & Heywood B.J. 1963a. Herbicidal action of 3,5-dihalogeno-4-hydroxybenzonnitriles, *Nature*, Lond. 200, 28-29.
- Carpenter K, Cottrell H.J., de Silva, W.H., Heywood B.J., Leeds W.G., Rivett K.F., & Soundy, M.L., 1964b. Chemical and biological properties of two new herbicides - ioxynil and bromoxynil. *Weed Res.* 4., 175-195.
- Terry, H.J., & Wilson, C.W. 1964. Field experiments on the herbicidal activity and tolerance of cereals to ioxynil and bromoxynil, *Weed Res.* 4, 196-215.

## IOXYNIL IN SWEDEN

R. Elamzon

Gullviks Fabriks AB, Malmö 16, Sweden.

Summary. Ioxynil alone and in combination with phenoxy acids has been tested in cereals during the last three years in Sweden. Under good growing conditions, ioxynil alone has given good control of many weeds, especially Tripleurospermum maritimum ssp. inodorum, but under average conditions and especially against perennial weeds, mixtures with the phenoxy acids have been found more generally reliable. Field trials conducted in 1964 have therefore been concentrated on the evaluation of these mixtures.

### INTRODUCTION

In winter cereals Tripleurospermum maritimum ssp. inodorum, Stellaria media and Galium aparine are the most troublesome weeds. In spring sown cereals Chenopodium album, S. media, Polygonum spp., Galeopsis spp., Sinapis arvensis, T. maritimum ssp. inodorum, Viola arvensis and Cirsium arvense dominate. Some of these weeds are rather difficult to control with commonly used weedkillers.

The first field trials in Sweden with ioxynil (3,5-diiodo-4-hydroxy-benzonitrile) were laid out in 1962. As the results were promising, the work continued 1963-1964 with this new chemical in comparison with herbicides in current use.

### METHODS AND MATERIALS

In 1962 an oil soluble amine salt of ioxynil (ACP 62-70, 2 lbs/US gal) was used and in 1963 a 50 % wetttable powder formulation (ACP 62-177). During 1964 most of the work has been done with water solutions of lithium or sodium salts.

Formulations used in 1964:

ioxynil-Li 1 lb + MCPA-Li 3 lbs/US gal (ACP 63-339)  
ioxynil-Li 1,04 lb + 2,4-D-Li 2,08 lbs/US gal (ACP 63-334)  
ioxynil-Li 0,5 lb + dichlorprop 3 lbs/US gal (ACP 63-340)  
ioxynil-Li 0,5 lb + mecoprop 3,0 lbs/US gal  
ioxynil-Li 2 lbs/US gal. (ACP 63-303)  
ioxynil-Na 2 lbs/US gal (ACP 64-53)

Plot size 30 m<sup>2</sup>. Simple randomised blocks with 4 replications.

Spray volume 400 litres/ha. Knapsack sprayer with 5 Teejet 6505 nozzles on a 2 m sprayboom.

All weeds in 2 x 0,25 m<sup>2</sup> per plot were counted and weighed 4 - 5 weeks after treatment.

Yields were taken with a combine harvester.



Crops, winter cereals, mainly winter wheat, variety Starke.

Stage of growth from start of tillering up to the end of shooting.

Spring cereals, barley, oats and wheat of different varieties.  
Stage of growth from 2 leaves up to the end of shooting.

## RESULTS

### 1962 Experiments

The spring was warmer and wetter than normal.

Against S. media ioxynil at 0,5 - 1,5 kg/ha was not as good as 2,0 kg/ha mecoprop and MCPA 1,5 kg + dinoseb 2,0 kg/ha, but much better than 1,5 kg/ha 2,4-D. 1,0 - 1,5 kg/ha ioxynil was equal to mecoprop and MCPA + dinoseb against G. aparine and much better than 1,5 kg/ha 2,4-D. Against T. maritimum ssp. inodorum 1,0 - 1,5 kg/ha ioxynil was superior to all tested chemicals.

Spring barley was temporarily scorched by 1,5 kg/ha ioxynil to the same extent as by the mixture MCPA 1,5 kg + dinoseb 2,0 kg/ha. Ioxynil raised the yield a little less than the other weedkillers.

Table 1 1962 RESULTS FROM 2 TRIALS IN WINTER WHEAT AND 1 TRIAL IN SPRING BARLEY.

Chemicals	kg/ha	% Weeds <sup>x</sup>		% Yield <sup>x</sup>
		number	weight	
Control	-	100 (127/m <sup>2</sup> )	100 (397 g/m <sup>2</sup> )	100,0 (4561 kg/ha)
ioxynil (ACP 62-70)	0,5	45	27	106,2
"	1,0	33	22	101,1
"	1,5	27	16	100,6
mecoprop-K	2,0	33	16	106,6
2,4-D-amin	1,5	46	34	105,6
MCPA-Na + dinoseb-amin	1,5 + 2,0	33	20	106,5
				LSD <sup>xxx</sup> 8,3

<sup>x</sup> Weeds and yields are given as per cent of controls.

<sup>xxx</sup> Least significant difference at P = 0,05.

## 1963 Experiments

May and June were warm and very dry and weeds as well as cereals grew very slowly. Under these conditions none of the tested herbicides gave good weed control.

Winter cereals. 0,75 - 1,0 kg/ha ioxynil was necessary to give acceptable and uniform weedcontrol. At these rates ioxynil was equal to MCPA 1,0 kg + TBA 0,33 kg/ha and better than all standard treatments against T. maritimum ssp. inodorum and Matricaria chamomilla. 0,50 - 0,75 kg/ha ioxynil was not quite as good as 2,0 kg/ha mecoprop against S. media, but better than all other chemicals. Best effect was obtained when the weeds were sprayed at an early stage.

In one trial a very slight stunting of winter wheat was observed at 1 kg/ha ioxynil.

Ioxynil gave a little higher yield increase than other treatments.

Table 2 1963 RESULTS FROM 5 TRIALS IN WINTER CEREALS.

<u>Chemicals</u>	<u>kg/ha</u>	<u>% Weeds</u>	<u>% Yield</u>
Control	-	100 (102/m <sup>2</sup> )	100,0 (3669 kg/ha)
ioxynil (ACP 62-177)	0,25	54	105,8
"	0,50	39	107,2
"	0,75	31	106,4
"	1,00	28	100,8
Standard Treatments	X	38	104,2
MCPA + TBA	1,0 + 0,33	37	102,8
			LSD 7,2

X = Standard treatment  
mecoprop 2 kg + 2,4-D 0,5 kg/ha or  
MCPA 1,5 kg + dinoseb 2 kg/ha.

Spring cereals. 0,5 - 1,0 kg/ha ioxynil was equal to 2,0 kg/ha mecoprop and MCPA 1,5 kg + dinoseb 1,5 kg/ha against S. media. Against Viola arvensis, Myosotis arvensis and Polygonum persicaria 0,5 - 1,0 kg/ha ioxynil was superior. Ch. album and Brassica napus (weed rape) were rather resistant this year to ioxynil. Perennial weeds like Cirsium arvense, Stachys palustris and Mentha arvensis were nearly unaffected by ioxynil.

No damage on crop was observed. The crop suffered during the very dry spring and yield figures were very low and irregular.

Table 3 1963 RESULTS FROM 4 TRIALS IN SPRING CEREALS.

<u>Chemicals</u>	<u>kg/ha</u>	<u>% Weeds</u>	<u>% Yield</u>
Control	-	100 (188/m <sup>2</sup> )	100,0 (2753 kg/ha)
ioxynil (ACP 62-177)	0,25	42	97,7
"	0,50	31	96,6
"	0,75	21	100,5
"	1,00	20	101,2
mecoprop-K	2,0	55	94,4
MCPA-Na + dinoseb-amin	1,0 + 1,5	34	94,4
			LSD 5,4

1964 Experiments

The growth conditions this year were favourable. In order to get a broader weedcontrol mixtures of phenoxy acids and ioxynil were tested this year.

Winter wheat (Tables 4-6) The addition of ioxynil considerably increased the activity of all tested phenoxy acids and also responded with better results than ioxynil alone, even when used at higher rates. In all combinations the control of T. maritimum ssp. inodorum was improved by ioxynil. Mixtures of ioxynil with MCPA, mecoprop och dichlorprop were superior to ioxynil + 2,4-D especially where S. media and G. aparine were dominating. Matricaria chamomilla was more resistant against all herbicides than T. maritimum ssp. inodorum. MCPA + ioxynil worked faster than MCPA + TBA.

Yields were increased more by mixtures of ioxynil with MCPA and phenoxypropionic acids than by ioxynil + 2,4-D and MCPA + TBA. Ioxynil alone gave also a good yield increase.

Table 4 1964 RESULTS FROM 9 TRIALS IN WINTER WHEAT WITH IOXYNIL + MCPA.

<u>Chemicals</u>	<u>kg/ha</u>	<u>% Weeds</u>			<u>% Yield</u>	
		<u>total</u>	<u>number</u>			<u>weight</u>
			<u>1</u>	<u>2</u>		
Control	-	(111/m <sup>2</sup> )	100	100	(846 g/m <sup>2</sup> ) (6351 kg/ha)	
		100	100	100	100,0	
ioxynil + MCPA-Li	0,25 + 0,75	33	34	22	22	105,1
"	" 0,50 + 1,50	24	26	20	15	106,4
"	" 0,75 + 2,22	16	16	10	14	106,5
ioxynil-Li	0,75	32	34	24	19	109,2
MCPA-Na	1,5	42	36	73	33	105,4
TBA + MCPA-Na	0,25 + 0,75	40	37	53	32	103,8
						LSD 4,1

1 = S. media

2 = T. maritimum ssp. inodorum

Table 5 1964 RESULTS FROM 7 TRIALS IN WINTER WHEAT WITH IOXNYL + 2,4-D.

<u>Chemicals</u>	<u>kg/ha</u>	<u>% Weeds</u>				<u>% Yield</u>
		<u>total</u>	<u>number</u>		<u>weight</u>	
			<u>1</u>	<u>2</u>		
Control	-	(109/m <sup>2</sup> )			(631 g/m <sup>2</sup> )	(6790 kg/ha)
		100	100	100	100	100,0
ioxynil + 2,4-D-Li	0,25 + 0,50	44	51	32	39	99,9
" "	0,50 + 1,0	41	51	14	36	100,6
" "	0,75 + 1,5	29	40	5	26	99,6
ioxynil-Li	0,75	36	41	21	31	102,6
2,4-D-amin	1,0	51	57	50	60	96,2
" +dichlorprop-K	0,5 + 2,0	23	22	39	18	100,8
						LSD 4,5

1 = *S. media*

2 = *T. maritimum* ssp. *inodorum*

Table 6 1964 RESULTS FROM 5 TRIALS IN WINTER WHEAT WITH IOXNYL + PHENOXYPROPIONIC ACIDS.

<u>Chemicals</u>	<u>kg/ha</u>	<u>% Weeds</u>				<u>% Yield</u>
		<u>total</u>	<u>number</u>		<u>weight</u>	
			<u>1</u>	<u>2</u>		
Control	-	(109/m <sup>2</sup> )			(606 g/m <sup>2</sup> )	(6225 kg/ha)
		100	100	100	100	100,0
ioxynil + mecoprop-Li	0,25 + 1,5	19	18	10	10	110,4
ioxynil + dichlorprop-Li	0,25 + 1,5	29	34	16	21	110,6
mecoprop-K	2,5	22	13	39	12	112,2
dichlorprop-K	2,5	22	13	38	14	108,8
ioxynil-Li	0,75	41	64	12	39	111,0
dichlorprop-K + 2,4-D-amin	2,0 + 0,5	26	22	41	21	107,5
						LSD 4,8

1 = *S. media*

2 = *T. maritimum* ssp. *inodorum*

Spring cereals (Tables 7-8) Also in spring sown cereals the addition of ioxynil improved the effect of phenoxy acids. With ioxynil + MCPA good control was obtained of S. media, T. maritimum ssp. inodorum, P. persicaria, V. arvensis, M. arvensis, Galeopsis spp. and Veronica agrestis, whereas the effect against G. aparine was somewhat uncertain. The addition of ioxynil increased the effect of mecoprop and dichlorprop against T. maritimum ssp. inodorum, V. arvensis, M. arvensis and V. agrestis. Good control of P. persicaria was obtained with ioxynil + mecoprop.

Ioxynil mixtures had good effect also against perennial weeds like Cirsium arvense, Sonchus arvensis and Stachys palustris. Against these weeds ioxynil alone had only little effect.

No damage to crops was observed from the use of ioxynil, whereas in some cases dicamba + MCPA caused a slight bending of the straw, although without any influence on yield.

Formulations (Table 9) Comparisons between various formulations of pure ioxynil indicated that lithium and sodium salts had about the same effect as the wettable powder formulation.

Table 7 1964 RESULTS FROM 6 TRIALS IN SPRING CEREALS WITH IOXYNIL + MCPA.

Chemicals	kg/ha	number				Yield
		Weeds		weight	%	
		total	1			
Control	-	(335/m <sup>2</sup> )			(207 g/m <sup>2</sup> )	(4430 kg/ha)
		100	100	100	100	100,0
ioxynil + MCPA-Li	0,13 + 0,38	34	35	46	16	103,8
" "	0,25 + 0,75	19	18	40	10	104,1
" "	0,50 + 1,5	6	5	14	2	106,4
ioxynil-Li	0,50	16	20	23	10	102,3
MCPA-Na	1,0	45	35	75	29	104,7
dicamba + MCPA	0,07 + 1,0	27	8	83	20	107,2
						LSD 6,3

1 = S. media

2 = T. maritimum ssp. inodorum

Table 8 1964 RESULTS FROM 4 TRIALS IN SPRING CEREALS WITH IOXYNIL + PHENOXYPROPIC ACIDS.

Chemicals	kg/ha	% Weeds				% Yield
		total	number		weight	
			1	2		
Control	-	(375/m <sup>2</sup> ) 100			(221 g/m <sup>2</sup> ) 100	(4085 kg/ha) 100,0
ioxynil + mecoprop-Li	0,25 + 1,5	8	1	10	13	105,1
ioxynil + dichlorprop-Li	0,25 + 1,5	9	4	17	7	108,6
mecoprop-K	2,0	24	0	23	25	105,2
dichlorprop-K	2,0	16	1	29	20	101,5
ioxynil-Li	0,5	20	17	10	14	110,7
dicamba + MCPA	0,07 + 1,0	20	6	30	15	108,1
						LSD 10,3

1 = S. media

2 = T. maritimum ssp. inodorum

Table 9 1964 RESULTS FROM 3 TRIALS IN SPRING CEREALS WITH DIFFERENT IOXYNIL FORMULATIONS.

Chemicals	kg/ha	% Weeds		% Yield
		number	weight	
Control	-	100 (173/m <sup>2</sup> )	100 (359 g/m <sup>2</sup> )	100,0 (4767 kg/ha)
ioxynil-Li (ACP 63-303)	0,50	38	25	110,0
"	0,75	29	22	108,0
ioxynil-Na (ACP 64-53)	0,50	29	24	108,2
"	0,75	31	24	107,7
ioxynil wp (ACP 62-177)	0,50	44	32	110,6
"	0,75	30	19	110,5
				LSD 7,0

Spray volume. Several tests with different spray volumes have been conducted (200, 400, 600, 800 litres/ha). 400 litres per ha seemed to be necessary to get uniform weedcontrol.

The addition of three different types of surfactants did not improve the activity of pure ioxynil-Na at a spray volume of 400 litres/ha.

Environment. The temperature and the growth activity of the weeds appeared to be more important than the growth stage of the weeds at the time of application. The higher the temperature and the stronger the growth activity the better the weedcontrol obtained. A temperature above 10°C seemed necessary for good weedcontrol. Under comparable conditions younger and smaller weeds were, of course, easier to control than older and bigger weeds.

Soil type seemed to have very little influence on the activity of ioxynil.

Ioxynil acts mainly as a contact herbicide, but it has also a slow systemic activity. Applied to the soil ioxynil has even some pre-emergence effect on weeds.

Crop tolerances. Cereals seemed to tolerate fairly high amounts of ioxynil. In logarithmic plots oats and winter wheat tolerated as much as 10 kg/ha ioxynil-Na without yield reduction. A temporary burning could be observed at rates, exceeding 6 kg/ha ioxynil.

Undersown lucerne in spring wheat was not injured by 0,5 kg/ha ioxynil-Na.

Onion tolerated 1 kg/ha ioxynil-Na.

Other derivatives. In field trials ioxynil gave much better control of S. media, M. arvensis and V. arvensis than bromoxynil (3,5-dibromo-4-hydroxybenzotrile) and chloroxynil (3,5-dichloro-4-hydroxybenzotrile). Against T. maritimum ssp. inodorum chloroxynil and bromoxynil were somewhat superior to ioxynil.

In greenhouse tests ioxynil was considerably more effective against S. media and G. aparine than the two others. None of these derivatives has damaged barley or winter wheat.

#### DISCUSSION

Ioxynil has proved to be a promising herbicide that combines a good weedkilling action with a very good safety margin in cereals.

Best and cheapest weedcontrol is obtained in mixtures with phenoxy compounds. Ioxynil + MCPA is probably the best combination. An appropriate rate is in winter cereals ioxynil 0,5 kg + MCPA 1,5 kg/ha and in spring cereals ioxynil 0,25 kg + MCPA 0,75 kg/ha.

In fields, where S. media and G. aparine dominate, ioxynil 0,25 kg + mecoprop or dichlorprop 1,5 kg/ha give very good weedcontrol in winter cereals as well as in spring cereals.

## EXPERIMENTS ON THE USE OF IOXYNIL IN CEREALS

J. Hauteur and R. Canette

Compagnie Française de Produits Industriels, Asnières, Seine, France.

Summary : 17 trials were made to compare effectiveness of Ioxynil alone or in mixture with phenoxyacids for selective weedkilling of several cereals. No definite conclusions can be given on the two formulations of Ioxynil alone but it appears that mixtures are interesting and must be studied further on.

### INTRODUCTION

Further to last year experiment (J. Pécheur 2nd Celuma Conference) which studied IOXYNIL WETTABLE POWDER 50 % a. i. and mixtures with other materials, we compared this year effectiveness of IOXYNIL WETTABLE POWDER and IOXYNIL LITHIUM SALT (240 g/l a. i.) either alone or in mixture with 2,4-D or MCPA.

### METHOD AND MATERIALS

- Trials were made as block with two replications and plots were sprayed with Van der Weij logarithmic sprayer at 1/25 ratio. Plots were 15 m long x 2 m width and a 1,000 l. solution was applied per hectare.

- Cereals treated were :

a) Northern France :

- 1 trial on spring sown wheat,
- 2 trials on winter wheat,
- 1 trial on spring sown barley.

b) Southern France :

- 2 trials on winter wheat,
- 1 trial on hard wheat.

- Cereals were treated :

Ioxynil alone :

- Stage 1) when 2 to 4 leaves
- Stage 2) at tillering (beginning of)

Mixtures :

- Stage 3) end of tillering/beginning of jointing.

- Materials :

- I a) ACP 62-177 WP 50 % a.i. .... log 2.- to 0.080 kg/ha
  - b) ACP 63-303 240 g/l a.i. as lithium salt log 2.- to 0.080 kg/ha
  - c) DNOC 50 % a.i. .... log 10.- to 0.400 kg/ha
  
  - II a) 2,4-D amine salt ..... log 3.5 to 0.140 kg/ha
  - b) 2,4-D amine salt  
+ ACP 63-303 (1 to 1 ratio) } ..... log 2.- to 0.080 kg/ha
  - 2,4-D amine salt  
+ ACP 63-303 (2 to 1 ratio) } ..... log 2.- to 0.080 kg/ha
  - c) MCPA (K/Na salt) ..... log 4.- to 0.160 kg/ha
  - MCPA lithium salt  
+ ACP 63-303 (3 to 1 ratio) } ..... log 3.- to 0.120 kg/ha
  - MCPA lithium salt  
+ ACP 63-303 (2 to 1 ratio) } ..... log 3.- to 0.120 kg/ha
- .../...



- Key to main weeds

- |                          |                           |
|--------------------------|---------------------------|
| 1) Adonis sp.            | 13) Polygonum aviculare   |
| 2) Alchemilla sp.        | 14) Polygonum convolvulus |
| 3) Anagallis arvensis    | 15) Ranunculus arvensis   |
| 4) Cerastium sp.         | 16) Raphanus Raphanistrum |
| 5) Chenopodium album     | 17) Rumex lapathifolium   |
| 6) Fumaria officinalis   | 18) Sinapis arvensis      |
| 7) Galium aparine        | 19) Solanum nigrum        |
| 8) Lathyrus sp.          | 20) Sonchus arvensis      |
| 9) Matricaria Chamomilla | 21) Stellaria media       |
| 10) Mercurialis annua    | 22) Valerianella sp.      |
| 11) Myosotis sp.         | 23) Veronica sp.          |
| 12) Papaver Rhoeas       |                           |

RESULTS I (materials alone)

Results are tabulated hereunder giving the lowest rate necessary to obtain good control. Details concerning yield records were not available when writing this summary, so this is only an indication of the effectiveness as observed under French conditions.

Winter Wheat :

Stage	Date	Trial Nr.	Main weeds	Stage of weeds	Lowest rates giving good control 1 month aft. appl.
					62-177 : 63-303 : DNOG
1	3.11.64	North 1	13,14,18,19,21	em.	0,500 kg; 0,600 kg; 0,800 kg
	12.13.63	South 1	3,4,10,13,23	em.	0,240 kg; 0,700 kg; 1,250 kg
	12.20.63	South 2	5,7,9,11,16	em.	0,600 kg; 0,300 kg; 0,500 kg
2	4.22.64	North 1	13,14,18,19,21	2-4 l.	0,700 kg; 0,700 kg; 1,800 kg
	4.13.64	North 2	9,17,18,	em.	Excellent results all rates
	3.2.64	South 1	2,15,22,23	4-5 l.	0,600 kg; 1,400 kg; 5,000 kg
	2.18.64	South 2	4,5,7,8,9,11 16,23	2-4 l.	Even at 2 kg no results due to cold weather

Hard Wheat (Triticum durum)

1	3.18.64	South.	1,6,8,9,12 16,18,23	em. to 2-8 l.	0,700 kg; 0,500 kg; 3,500 kg
---	---------	--------	------------------------	------------------	------------------------------

Spring Wheat

1	5.12.64	North	5,6,14,18,20 21,23	1-2 l.	0,800 kg; 0,800 kg; 3,000 kg 6 and 20 not contr.
2	5.19.64	North	5,14,18,21,23	4-6 l.	0,500 kg; 1,000 kg; 2,500 kg

Barley :

1	4.28.64	North	9,13,18 (5,19,21 appeared aft. appl.)	1-2 l.	0,200 kg; 0,200 kg; 1,600 kg (5,19,21 control.)
2	5.12.64	North	9,13,14 18,20,21.	4-5 l.	0,330 kg; 0,130 kg; 3,000 kg (20 controlled by barley)

.../...

RESULTS II (Mixtures)

Winter Wheat

Stage	Date.	Trial Nr	Main weeds	Stage of weeds	Minimum rate giving good control 1 month aft. appl.		
					2,4-D	Mixtures	
3	4.7.64	South	2,15,22,23	before bloom	1,200 to	1/1	2/1
					3,500 kg	0,300 kg	0,540 kg
					MCPA.	Mixtures	
					3/1	2/1	
					1,200 kg	0,700 kg	0,500 kg
3	5.7.64	North	13,14,18,19,21	As this field was clean no conclusions can be given prior having yield results.			

Hard Wheat

Stage	Date.	Trial Nr	Main weeds	Stage of weeds	2,4-D	Mixtures	
						1/1	2/1
3	4.15.64	South	1,6,8,9,12,16,18,23	before bloom	2,100 to	1,700 to	1,500 to
					3,500 kg	2,000 kg	2,000 kg
					MCPA.	3/1	2/1
					4,000 kg	3,000 kg	3,000 kg
					(poor control only)		

Spring Sown Wheat

Stage	Date.	Trial Nr	Main weeds	Stage of weeds	2,4-D	Mixtures	
						1/1	2/1
3	5.28.64	North	5,14,18,20,21,23	before bloom	0,680 kg	0,300 kg	0,300 kg
					MCPA.	3/1	2/1
						0,900 kg	0,450 kg

Barley

Stage	Date.	Trial Nr	Main weeds	Stage of weeds	2,4-D	Mixtures	
						1/1	2/1
3	5.28.64	North	9,13,14,18,21.	before bloom	0,240 kg	0,400 kg	0,160 kg
					MCPA.	3/1	2/1
						1,000 kg	0,730 kg

- Materials alone

Tabulating these results does not allow to show all details but we can say that under our conditions it is difficult to determine which is the most effective formulation as results vary according to experimental conditions.

.../...

Phytotoxicity : On Spring cereals, and when applied at a late stage (end of tillering) Ioxynil gave some phytotoxicity at 1 and 2 kg a.i. This will be more precisely defined when yield results are known.

- Mixtures.

It can be seen from this data that if in Northern France, mixtures give interesting results, as compared to 2,4-D or MCPA alone there is still a certain weakness in Southern France.

Phytotoxicity : It seems that, in general, mixtures are less aggressive than 2,4-D or MCPA, this was already seen last year, and will be more clearly defined when yield results are known.

### CONCLUSIONS

- Ioxynil alone

Although under certain circumstances we had good results at rates lower than 500 g/ha (a. i.), it appears necessary, in general, for both formulations, to use higher rates.

- Mixtures

Give a better effectiveness than 2,4-D or MCPA alone, however under certain circumstances, effectiveness though superior to 2,4-D or MCPA alone, is still insufficient and so other mixtures should be studied.

-:-:-:-:-

## THE USE OF IOXNYL AS A SELECTIVE WEEDKILLER IN CEREALS

R. A. Fosse, W. R. Dudlik, and J. H. Kirch  
Amchem Products, Inc., Ambler, Pennsylvania, U.S.A.

Summary For controlling weeds in cereals, the chemicals now commonly used have major weaknesses. A new herbicide -- ioxnyl (3,5-diiodo-4-hydroxybenzotrile) -- appears to overcome these. Unlike 2,4-D and MCP acetic or propionic sprays, ioxnyl can be used safely on cereals in the fall of the year, and on cereals to be under-sown with legumes, being tolerated by cereals in early stages of growth and also exhibiting some tolerance by legumes. Ioxnyl therefore makes possible control of the fall-germinating weeds which reduce crop yields most substantially and make legume establishment difficult. Ioxnyl kills many annual weeds, including species resistant to earlier herbicides as presently recommended.

Weeds are most sensitive to ioxnyl when they are very young. Temperature, light, moisture, soil fertility, and other factors in the rate of weed growth may be important in ioxnyl's performance. Certain surfactants increase ioxnyl's effectiveness. Low volumes of carrier affect the kill of some weeds. Mixtures of ioxnyl and other herbicides may be useful for spring treatment of cereals. In the fall ioxnyl alone is preferable.

### INTRODUCTION

In 1962, 49,084,000 acres of wheat, 14,701,000 acres of barley and 30,202,000 acres of oats were seeded in the United States according to the U.S. Department of Agriculture (1963). Since the introduction of 2,4-D and MCPA in the early 1940's, these chemicals and their propionic counterparts have been sprayed annually on millions of acres of grain for broadleaf weed control. Most of this grain is planted in the fall and treated the next spring.

In field use, four weaknesses in the 2,4-D - MCP acetic or propionic grain spray program have been evident:

(1) Fall application of 2,4-D or MCP injures fall-seeded grains severely; these chemicals cannot be applied safely until spring [Ilnicki (1960)].

(2) By spring, when 2,4-D or MCP can be applied without injuring the fall-seeded grains, annual broadleaf weeds germinating with the crop have already competed so effectively that grain yields will be reduced significantly over the hand-weeded checks [Friesen (1958); Swan and Furtick (1962)].

(3) Certain weeds such as Amsinckia spp. (fiddleneck), Polygonum convolvulus (wild buckwheat) and Anthemis cotula (dog fennel) are resistant to 2,4-D and MCP sprays as presently recommended [Foy (1964)].

(4) Where grains are underseeded with legumes, 2,4-D and MCP sprays cannot be used until the crop forms a protective canopy over the legumes.

#### FIELD PERFORMANCE

Research conducted with ioxynil (3,5-diiodo-4-hydroxybenzotrile) on cereal grains indicates that they tolerate ioxynil well in early stages of growth and that optimum control of such weeds as Polygonum convolvulus, Amsinckia spp., Thlaspi arvense (pennycress), and Anthemis cotula can be obtained at this stage of growth. This means that, unlike 2,4-D and MCP acetics and propionics, ioxynil can be applied to fall-seeded grains in the fall and will control the weeds well without injuring the crop.

#### Stage of growth

Stage of growth is probably the most important factor affecting the control of weeds and the tolerance of crops. Table 1 summarized from work by Foy (1964) in California shows this effect on Amsinckia control in barley.

Table 1

Effect of ioxynil on Amsinckia sp. and barley treated at several stages of development.

Stage of growth		Control*	Barley yield as %
Barley	<u>Amsinckia</u> sp.		of check
3-4 leaf	cotyledon	79%	104
5-8 leaf	1-4 inches	86	117
14 inches (tillered)	15 inches	28	83

\*average of  $\frac{1}{8}$ ,  $\frac{1}{2}$ , 1 pound per acre rates

This information is further supported by Appleby and Furtick's (1964) data from Oregon in Tables 2 and 3. Although 2,4-D gave good control of Amsinckia in the November treatment yields were reduced significantly. Wheat treated with ioxynil at this time produced yields greater than the check.

Table 2

Control of <u>Amsinckia intermedia</u>			
Treatment	Rate	Nov. treatment	March treatment
Ioxynil	.25 lb/ac	87% control	52% control
Ioxynil	.50	93	62
2,4-D ester	.25	93	57
2,4-D ester	.50	100	75
2,4-D ester	.75	100	88

Table 3

## Average yield of Gaines wheat from Amsinckia experiment

Treatment	Rate	November treatment	March treatment
2,4-D ester	.25 lb/ac	45.7 bu/ac	55.7 bu/ac
2,4-D ester	.50	43.8	52.5
2,4-D ester	.75	44.7	52.5
Ioxynil	.25	58.2	53.3
Ioxynil	.50	56.5	48.3
Weedy check	0.00		53.7

Temperature and rainfall

The effects of temperature and rainfall on ioxynil performance have been reported by Hart (1964) earlier in these sessions. Ioxynil appears to perform better at higher temperatures, and its activity is reduced by rain soon after application.

Fall treatment

Under many conditions and with certain weed species it is advantageous to treat fall-seeded cereals in the fall for weed control. Some weeds such as *Amsinckia*, *Lithospermum arvense* (corn growwell) and *Scleranthus annuus* (knawel) make good growth in the fall and can have a severe competitive effect on the crop or become tolerant to 2,4-D before spraying time in the spring. Fall treatment with ioxynil permits higher yields. This is confirmed by Seely (1964) in Tables 4 and 5.

Table 4

## Scleranthus control in Cheyenne winter wheat

Treatment	Rate	Yield		Control*	
		when sprayed		when sprayed	
		9/27/63	5/11/63	9/27/63	5/11/63
Ioxynil	$\frac{1}{2}$ lb/ac	39.4 bu/ac	30.3 bu/ac	4.3	6.0
Ioxynil	1	37.7	21.2	4.0	6.3
Ioxynil	2	36.9	27.0	3.3	4.0
2,4-D	1	24.8	23.0	4.0	9.0
2,4-D	$1\frac{1}{2}$	27.4	23.0	4.0	7.3
2,4-D	2	26.1	20.1	3.7	8.7
Check	0	19.0	21.7	9.7	9.7

\* 0 = complete control; 10 = no control

Table 5

Lithospermum control in Omar winter wheat					
Treatment	Rate	Yield		Control*	
		when sprayed		when sprayed	
		10/17/63	4/28/64	10/17/63	4/28/64
Ioxynil	1 lb/ac	58.0 bu/ac	49.6 bu/ac	3.8	7.2
Ioxynil	1	66.8	45.4	3.3	6.3
Ioxynil	1	58.3	52.0	3.0	5.0
2,4-D	1	55.5	48.7	4.0	6.0
2,4-D	1	48.0	48.2	3.2	6.5
2,4-D	1	43.2	51.2	3.0	5.0
Check	0	55.0	51.6	7.0	8.3

\*0 = complete control; 10 = no control

Additional information in Table 6 from Furrer and Fertig (1964) in New York shows similar results on Vicia villosa (common vetch) and Barbarea vulgaris (yellow rocket) in winter wheat.

Table 6

Fall treatments and visual control ratings of Barbarea vulgaris and Vicia villosa.

Chemical	Rate	Control rating*		Wheat yield, lb% moisture
		<u>Barbarea vulgaris</u>	<u>Vicia villosa</u>	
2,4-D amine	0.25 lb/ac	3+	1	47.3 bu/ac
2,4-D amine	0.5	7	1	37.3
2,4-D amine	1.0	9	2	31.3
ACP 62-70B	0.25	4	4	46.8
ACP 62-70B	0.5	9	9	50.5
ACP 62-70B	1.0	9	9+	62.2
ACP 62-70B	1.7	9	9+	46.0
ACP 62-177	0.25	8	0	47.6
ACP 62-177	0.5	9	9	44.4
ACP 62-177	1.0	9	9+	47.6
ACP 62-177	2.0	9+	9+	49.0
Check	0.0	0	0	42.0

\*Average of 2 replications; observations of June 26, 1963.

10 = complete control; 0 = no control

Another advantage of fall spraying with ioxynil occurs in areas where legumes are to be under-seeded in the spring. Removal of the weeds prior to legume seeding enables the legumes to become established more rapidly, increases the yield of grains, and avoids setback by later sprays in the spring.

### Volume of spray

In the western part of the United States, about 40% of the grain acreage is sprayed with low volumes (2 to 5 gal/ac) from airplanes, and thus the volume of carrier used becomes important. Early tests at Anchem Research Farm suggested that volume of carrier can influence the effectiveness of ioxynil; in a greenhouse test using a lithium salt formulation, effectiveness declined as the volume was reduced (Table 7).

Table 7

Control with  $\frac{1}{2}$  lb/ac ioxynil applied post-emergence in different volumes of water.

Test plant	Volume of water per acre					
	100 gal	50 gal	25 gal	12.5 gal	6.25 gal	3.12 gal
Onion	95%	95%	60%	50%	40%	10%
<u>Polygonum pennsylvanicum</u>	95	90	90	80	90	70
<u>Echinochloa crusgalli</u>	50	40	20	10	0	0
<u>Kochia scoparia</u>	90	90	80	70	70	60
<u>Amaranthus retroflexus</u>	100	100	100	100	100	80
<u>Bromus tectorum</u>	20	10	0	0	0	0
<u>Brassica sp.</u>	100	100	95	100	90	80
<u>Ipomea sp. (morning glory)</u>	95	100	100	100	90	80

To further check the effect of volume of carrier on the performance of ioxynil, trials were established during 1963 and 1964 in California, Oregon, Washington, Montana, and New York. Data from these trials confirms that on certain weeds volume of carrier can affect results, although not under all conditions. This may be due to incomplete coverage, as ioxynil does not translocate readily. This in turn may be influenced by adequacy of penetration as well as generic response.

Furtick and Appleby (1963) found no noticeable differences on Chenopodium in Oregon. In 1964, as Table 8 shows, there was a definite effect on Anthemis cotula and a lesser effect on Brassica; there was no noticeable effect on other weeds in this trial.

Table 8.

Influence of volume of carrier on ioxynil sprayed at  $\frac{1}{2}$  lb/ac.

Volume	<u>Anthemis cotula</u>	<u>Brassica</u>
5 gpa	44% control	25% control
10	30	32
15	30	32
20	41	42
40	48	48



Volumes of carrier from 14 gal/ac to 39.2 gal/ac were tested in Montana by Guenther (1963, 1964). Increased control from higher volumes of carrier was noted on Polygonum convolvulus.

All available evidence indicates that for cereals there is an adequate safety margin between the amount of ioxynil required to control weeds and the amount that might injure the crop. The type of formulation has in some instances materially affected the initial response of the cereals and the weeds. Generally this effect has been overcome within a short period of time. There are, however, real differences in weed control on certain weeds and under certain conditions with different formulations, as shown by Appleby (1964) in Table 9.

Table 9

Formulation studies on ioxynil

Formulation	Rate	Brassica	Chenopodium
ACP 62-177 (wetable acid)	1/2 lb/ac	55% control	40% control
ACP 63-166 (oil soluble amine)	1/2	80	100
ACP 63-239 (lithium salt)	1/2	70	45

Trials conducted by Stewart (1963) in Montana show increases in weed control by addition of a surfactant (2% of final spray volume) as well as differences in formulation, as indicated in Table 10.

Table 10

Lithospermum control in winter wheat with ioxynil applied October 31, 1963

Treatment	Rate	Average control
ACP 62-177 (wetable acid)	.25 lb/ac	30%
ACP 62-177	.50	57
ACP 62-177	1.0	70
ACP 62-70 (oil soluble amine)	.25	17
ACP 62-70	.50	30
ACP 62-70	1.0	70
ACP 62-70 + 2% surfactant	.25	57
ACP 62-70 + 2% surfactant	.50	77
ACP 62-70 + 2% surfactant	1.0	70
Check	0.0	0

A trial Appleby (1964) conducted in Oregon indicated not only increased weed control with the addition of a surfactant but the difference between surfactants and species response. Treatments were applied June 20, 1964, when most weeds were approximately 3 to 5 inches tall. Plots were evaluated July 8.

Table 11

## Ioxynil formulation trial

Treatment	Rate	Average control			
		Anthemis	Chenopodium	Brassica	Scenecio
ACP 63-303 (lithium salt)	$\frac{1}{2}$ lb/ac	20%	18%	58%	65%
ACP 63-303	1	38	60	68	90
ACP 63-303+64-168A (non-ionic)	$\frac{1}{2}$ + 0.5%	33	83	85	95
ACP 63-303+64-168B (non-ionic)	$\frac{1}{2}$ + 0.5%	30	65	80	93
ACP 63-303+64-168C (anionic)	$\frac{1}{2}$ + 0.5%	30	93	95	93

## MIXTURES

Combinations of ioxynil with other selective herbicides such as 2,4-D, MCPA, 2,4-DP, MCPP, benzoic compounds, and others offer certain advantages over ioxynil alone. Such mixtures, which are limited to use in spring, have the advantage of being economical and of killing a wider range of weed species at later growth stages. The best herbicide to combine with ioxynil will depend on the weeds to be controlled and the time of application.

More effective weed control with lower rates of ioxynil has been evident in several tests, such as Guenther's (1964) reported in Table 12.

Table 12

## Wild buckwheat control in barley

Treatment	Rate	Stage applied barley	Degree of control	Average yield
Ioxynil	4 oz/ac	3-leaf	8	44.6 bu/ac
Ioxynil + 2,4-D ester	4 oz + 4 oz	3-leaf	9	47.8
Ioxynil + 2,4-D ester	4 oz + 8 oz	3-leaf	10	38.7
Ioxynil + MCPA	4 oz + 8 oz	3-leaf	9	42.9
2,4-D ester	8 oz	3-leaf	4	36.9
2,4-D amine	8 oz	3-leaf	4	41.5
Ioxynil	4 oz	Tillering	5	44.6
Ioxynil	8 oz	Tillering	6	41.6
Ioxynil	12 oz	Tillering	8	39.3
Ioxynil + 2,4-D ester	4 oz + 4 oz	Tillering	7	40.8
Ioxynil + 2,4-D ester	4 oz + 8 oz	Tillering	8	42.3
Ioxynil + MCPP	4 oz + 8 oz	Tillering	8	41.6
Ioxynil + 2,4-D	4 oz + 8 oz	Tillering	8	40.6
2,4-D amine	16 oz	Tillering	3	36.7
2,4-D ester	12 oz	Tillering	4	33.9
Weedy check	--	--	--	35.1

\*0 = no control; 10 = complete control

Hartman (1964), working with Salsola kali (Russian thistle) and Polygonum convolvulus, reported, "when applied alone, ioxynil provided rather erratic weed control as far as thistles and wild buckwheat were concerned. However, when ioxynil was combined with either 2,4-D low volatile ester or MCPA, excellent control of both wild buckwheat and Russian thistles was obtained. Also, these combinations showed no visible effects on the wheat plants".

#### CONCLUSION

Ioxynil overcomes the major weaknesses of herbicides presently used for controlling weeds in cereal crops. Ioxynil can be used in the fall on fall-seeded cereals. Weeds not controlled by spring application of 2,4-D or MCP can be controlled safely by fall treatment with ioxynil. When ioxynil is used in the fall, by eliminating weed competition in the early growth stages crop yields are greater than when conventional herbicides are used in the spring.

#### REFERENCES

- APPLEBY, A. P. (1964) Unpublished data (Oregon State Univ.).
- APPLEBY, A. P. and W. R. FURTICK (1964) Selective control of annual broadleaf weeds in grains with 3,5-diiodo-4-hydroxybenzotrile. Paper presented before the Weed Society Am.
- FOY, C. L. (1964) Ioxynil - a new weed killer for use in cereal grains. Proc. Calif. Weed Conf. 16:90-98.
- FRIESEN, G. (1958) Effects of weed competition in cereal grains and flax. Proc. Western Weed Control Conf. 16:14-18.
- FURRER, A. H. and S. N. FERTIG (1964) Weed control in winter wheat. Proc. Northeastern Weed Control Conf. 18:296-299.
- FURTICK, W. R. and A. P. APPLEBY (1963) Unpublished data (Oregon State Univ.).
- GUENTHNER, H. R. (1963) Evaluation of herbicides for control of wild buckwheat in cereals. Western Weed Control Conf. Res. Rept. 21:58-59.
- GUENTHNER, H. R. (1963, 1964) Unpublished data (Central Montana Branch Station).
- HART, R. D., W. R. DUDLIK, and A. R. COOKE (1964) Discovery of ioxynil and its development in the U.S.A. Paper presented before the British Weed Control Conf.
- HARTMAN, J. P. (1964) Unpublished data (Eastern Montana Branch Station).
- ILNICKI, R. D. (1960) The effects of 2,4-D, 4-(2,4-DB), 2-(2,4-DP), and silvex on wheat and spring oats. Proc. Northeastern Weed Control Conf. 14:266-267.
- SEELY, C. I. (1964) Unpublished data (Univ. Idaho).
- STEWART, V. R. (1963) Unpublished data (Montana Agr. Expt. Sta., N.W. Branch).
- SWAN, D., and W. R. FURTICK (1962) Competition of fiddleneck with wheat. Weeds 10:121-123.
- U. S. DEPARTMENT OF AGRICULTURE, Agricultural Statistics, 1963. U. S. Govt. Printing Office, Washington. 635 p.

## BIPYRIDILIUM HERBICIDES

### FIELD TRIALS WITH PP 407 AND PP 745 AS SELECTIVE HERBICIDES FOR WEED CONTROL IN CEREALS

H.M. Fox and C.R. Beech  
Plant Protection Limited, Jealott's Hill Research Station  
Bracknell, Berkshire

**Summary** Two new substituted 1,1'-carbamoylmethyl-4,4'-bipyridylium compounds coded PP 407 and PP 745 were evaluated for post-emergence weed control in cereals in 1963 and 64 and in yield trials in 1964. Both compounds controlled a number of weed species resistant to MCPA or 2,4-D and, in general, PP 745 showed slightly better activity than PP 407. Chrysanthemum segetum, Tripleurospermum maritimum ssp inodorum, Anthemis cotula and Polygonum persicaria were controlled by 1 lb ion/ac. Polygonum convolvulus and Polygonum aviculare were controlled by 1.5 lb ion/ac but Stellaria media and Galium aparine proved moderately resistant at this rate. At the rates used for weed control the chemicals caused slight crop scorch but the cereals recovered their "greenness" within three weeks of spraying and no depression in yield occurred. In one of eight yield trials, PP 745 at 3 lb ion/ac significantly reduced the yield of grain by 6% compared with untreated.

#### INTRODUCTION

The unique herbicidal properties of certain bipyridylium compounds, in particular 1,1'-ethylene-2,2'-bipyridylium cation (diquat) and 1,1'-dimethyl-4,4'-bipyridylium cation (paraquat), are now well established but both compounds are toxic to grass and broad leaved weed species (Cronshey 1961, Darter and Wright 1963). In connection with the development of these two herbicides many other closely related bipyridylium compounds were synthesised and examined for herbicidal activity in glasshouse tests at Jealott's Hill. During 1962 two compounds, namely PP 407 (1,1'-di(pentamethylenecarbamoylmethyl)-4,4'-bipyridylium dichloride) and PP 745 (1,1'-bis(3,5-dimethylmorpholinocarbonylmethyl)-4,4'-bipyridylium dichloride) were found to be very much more selective in their action in that they showed a high order of toxicity to broad leaved plants combined with relative safety to grass species. PP 407 and PP 745 were therefore compared in replicated small plot field trials during 1963 and again this year for post-emergence weed control in cereals. During 1964 replicated large plot trials were also laid down to determine whether the compounds affected the yield of wheat, barley and oats.

#### METHOD AND MATERIALS

##### Small plot weed control trials - 1963 and 1964

##### (i) Layout and details of application

PP 407 and PP 745 were formulated as aqueous solutions containing 2 lb cation per Imperial gallon and in the 1963 trials they were applied

at 0.75 and 1.5 lb ion/ac (3 and 6 pts/ac respectively). In 1964 they were also applied at these two rates and in addition at 1 lb ion/ac, i.e. 4 pts/ac. A 'hormone' weedkiller containing CMPP and 2,4-D ('Methoxone' Extra) was applied in each trial as a standard for comparison. In each trial all the treatments were applied in 20 gal of water per ac at 30 lb p.s.i. pressure with an Oxford precision sprayer. The bipyridyl treatments were applied with the addition of a non-ionic wetter ('Agral' 90 at 0.1% vol/vol).

The plot size was 6 yds x 4 yds (approx. 1/200th ac) and there were three replicates in 1963 and four in 1964. Two unsprayed (control) plots were included per replicate. The treatments were fully randomised within each block.

Nine trials were carried out each year; details of location, crop and variety, date of spraying and crop stage when sprayed are given in Table I.

Table I  
Small Plot Trial Details

Trial No.	County	Crop	Variety	Crop stage when sprayed		Date of spraying
				Leaves	Tillers	
1963						
B22	Berks.	W. Wheat	Prof. Marchal	5.3	4.1	April 23rd
B23	Hants.	W. Wheat	Cappelle	5.7	4.1	May 9th
B24	Bucks.	W. Wheat	Prof. Marchal	5.2	1.9	May 6th
B26	Berks.	S. Barley	Pallas	5.0	2.2	May 30th
B27	Oxon.	S. Barley	Proctor	4.6	1.4	May 16th
B28	Oxon.	S. Wheat	Opal	5.1	1.5	May 23rd
B29	Berks.	S. Wheat	Opal	5.1	0.7	May 22nd
B30	Hants.	S. Barley	Pallas	4.7	2.7	June 7th
B31	Hants.	S. Barley	Vada	6.0	3.0	June 6th
1964						
B38	Wilts.	W. Wheat	Cappelle	5.8	2.2	April 30th
B39	Hants.	W. Wheat	Prof. Marchal	6.1	2.3	May 6th
B40	Bucks.	W. Oats	Barnwell	6.1	3.4	May 12th
B41	Oxon.	S. Barley	Proctor	6.5	2.8	May 13th
B42	Hants.	S. Barley	Proctor	6.1	2.1	May 14th
B43	Berks.	S. Barley	Pallas	5.2	1.6	May 15th
B44	Hants.	S. Barley	Vada	5.9	1.4	May 20th
B45	Oxon.	S. Barley	Cambrinus	6.3	3.7	May 20th
B46	Hants.	S. Barley	Pallas	5.7	2.2	June 8th

W = Winter

S = Spring

(ii) Assessments

Sites with two or three weed species present in reasonable numbers and preferably species resistant to MCPA and 2,4-D were selected for the trials. Before the sprays were applied the amount of weed present in the plots was estimated by each of three observers. The percentage area of the ground covered by green material was estimated for each of the dominant species.

Within 48 hours after spraying, the bipyridyl treatments had noticeably scorched the weeds. The effect continued to increase with time over the next 14-21 days when the first post-spraying assessment was made. The percentage ground cover of each species was again estimated and in addition the degree to which the weed had been scorched was recorded using an arbitrary scale of 0-10 where 0 = no effect and 10 = weed completely scorched and killed. Two further ground cover assessments of weed control were made when the scorched weed had died and only unaffected green material remained. The first of these was made approximately 4-8 weeks after spraying and the second near to harvest, approximately 10-13 weeks after treatment.

Towards harvest when the final assessment of weed control was made on the individual species, the plots were graded for 'cleanliness' for all weeds, using an arbitrary scale where 0 = a weed free plot and 10 = a densely weedy plot.

(iii) Presentation of weed control data

The percentage area ground cover of weed for each treatment differed initially and the proportion of weed in the control plots varied with time, as a result of natural growth or senescence, therefore the percentage ground cover for any treatment at any post-spraying assessment time was adjusted using the following formula to give an index of weed control (I)

$$I = \frac{c \times T}{t \times C} \times 100$$

where c = cover on untreated plots at pre-spraying assessment

C = " " " " " post- " "  
 t = " " treated " " pre- " "  
 T = " " " " " post- " "

The weed control indices obtained express the amount of weed remaining in the treated plots as a percentage of that which, in theory, would have been present at any given time in the absence of treatment.

In order to simplify the presentation of the results the weed control indices have been grouped, scored numerically and classified as indicated in Table II. The scores for weed control, for individual species at the time of the second post-spraying assessment, are given in Tables IV-XI.

Table II  
Relationship between weed index and  
standard of weed control

Weed index range	Numerical score	Standard of weed control
Nil - 5	5	Excellent
5 - 10	4	Good
10 - 20	3	Satisfactory
20 - 40	2	Unsatisfactory
40 - 60	1	Poor
60+	0	Very Poor

### Large plot yield trials - 1964

For these trials both "clean" and weedy sites were chosen and PP 407 and PP 745 were evaluated at 1, 2 and 3 lb ion/ac (4, 8 and 12 pts/ac respectively). A weedkiller containing CMPP and 2,4-D ('Methoxone' Extra) was also applied in each trial at either 6 or 7 pts/ac according to whether the crop was spring or winter sown. At "clean" or very slightly weedy sites an unsprayed control plot was included in each replicate. The treatments were applied in 20 gals of water/ac at 25 p.s.i. pressure with a low volume spray unit mounted on the rear of a landrover. The bipyridyl treatments were applied with the addition of wetter ('Agral' 90 at 0.1% vol/vol). After spraying untreated plots were "wheeled" with the landrover so that the effect on yield would not be biased.

The plot size was 60 yds x 5 yds (one spray boom width) and the treatments, which were fully randomised, were replicated four times.

Eight trials were laid down and details of crop and variety etc. are given in Table III.

Table III  
Large plot yield trial details

Trial No.	Location	Crop	Variety	Crop stage when sprayed		Date of spraying 1964	Weed status of site
				Leaves	Tillers		
B51	Hants.	W. Oats	Powys	6.0	6.5	24th April	Clean
B52	Wilts.	W. Wheat	Cappelle	5.0	3.5	2nd May	Weedy
B53	Hants.	W. Barley	Dea	6.3	5.9	30th April	Clean
B54	Wilts.	S. Wheat	Opal	5.0	1.0	14th May	Weedy
B55	Hants.	S. Wheat	Opal	6.5	2.3	14th May	Slightly Weedy
B56	Hants.	W. Wheat (Spring Sown)	Rothwell-Perdix	6.7	2.3	20th May	Moderately Weedy
B57	Hants.	S. Barley	Union	5.9	2.3	15th May	Weedy
B58	Hants.	S. Barley	Proctor	6.1	2.5	21st May	Weedy

W = Winter

S = Spring

At harvest the plots were cut separately by a pusher type combine harvester. A single width cut (8 ft 6 in or 10 ft x 50 yds) down the centre of each plot was taken. The moisture content of the grain harvested from each plot was determined with an N.P.L. moisture meter and all yields were adjusted to 85% dry matter. Samples of grain were collected for thousand grain weight determinations. Two samples of 200 grains were counted and weighed for each plot and the weights multiplied by five and averaged.

### RESULTS

#### Small plot weed control trials - 1963 and 1964

##### Weed Control

(i) Chrysanthemum segetum (corn marigold) - Table IV

PP 407 and PP 745 gave good control at 1 lb/ac and excellent control of this species at 1.5 lb/ac in both years. The chemicals were equally effective and vastly superior to the CMPP/2,4-D mixture which, although it

reduced and suppressed the weed population, gave poor to unsatisfactory control.

- (ii) Tripleurospermum maritimum ssp inodorum (scentless mayweed)  
Anthemis cotula (stinking mayweed) - Table V

In 1963 both bipyrindyls gave satisfactory to good control of the mayweeds at 0.75 lb and excellent control at 1.5 lb and there was an indication that PP 407 was slightly superior to PP 745. However the results from the 1964 trials suggest that there is probably no difference in activity. Due to adverse weather at the beginning of April 1964, one of the trials (B38) was sprayed after the optimum stage and poor control of scentless mayweed was obtained. The overwintered mayweed was strong and the crop was beginning to meet in the rows. In the other 1964 trial excellent control was obtained at 1 lb/ac with either chemical. Over the two years PP 407 and PP 745 were as effective as or slightly superior to CMPP/2,4-D in controlling mayweed.

Table IV  
Chrysanthemum segetum (corn marigold)

Season		1963	1964		
Trial No.		B27	B41	B43	B44
Growth stage of weed		4-6 L.	Sd.-Y.P.	4 L.	6 L.
Height of weed		2"-3"	0"-5"	2"	2"-3"
2nd assessment - days after spraying		29	48	48	44
PP 407	0.75 lb ion/ac	5	3	4	4
	1.00 " " "	-	3	5	5
	1.50 " " "	5	4	5	5
PP 745	0.75 lb ion/ac	4	3	4	4
	1.00 " " "	-	4	5	5
	1.50 " " "	5	5	5	5
CMPP/2,4-D ( <i>'Methoxone' Extra</i> )	6 pts/ac	2	1	2	1
% ground cover of weed in control -					
	1. Before spraying	20.8	3.0	26.7	9.2
2. At 2nd assessment		48.4	19.2	53.8	24.3

Abbreviations - applicable to all tables

Br. = Branching

Fl. = Flowering

Fl.B. = Flower Bud

L. = Leaf/Leaves

M.P. = Mature Plant

Rs. = Rosette

Sd. = Seedling

Y.P. = Young Plant



Table V  
Tripleurospermum maritimum ssp inodorum (scentless mayweed)  
Anthemis cotula (stinking mayweed)

Season	1963				1964	
	B22 Sd.-Rs.	B23 Rs. 2"-4"	B26 Fl.B. 2"-4"	*B28 Rs.-Y.P. 1"-4"	B38 Rs.-Y.P. 4"	B45 Rs. 1½"-3"
Trial No.						
Growth stage of weed						
Height of weed	-	-	-	-	-	-
2nd assessment - days from spraying	48	54	36	42	55	38
PP 407						
0.75 lb ion/ac	4	4	4	3	1	5
1.00 " " "	-	-	-	-	2	5
1.50 " " "	5	5	5	5	2	5
PP 745						
0.75 lb ion/ac	3	3	5	3	2	4
1.00 " " "	-	-	-	-	1	5
1.50 " " "	5	4	5	4	2	5
CMPP/2,4-D						
6 pts/ac	-	-	1	4	-	3
('Methoxone' Extra)						
7 pts/ac	3	5	-	-	3	-
% ground cover of weed in control -						
1. Before spraying	9.9	1.5	5.0	17.9	7.7	3.5
2. At 2nd assessment	15.7	12.0	6.0	37.5	6.1	2.9

\* Mostly stinking mayweed with some scentless  
 All remaining trials were with scentless mayweed

(iii) Polygonum aviculare (knotgrass) - Table VI

PP 745 was more active than PP 407 against knotgrass which proved somewhat resistant to the bipyridyls. In three trials, where the infestation was moderately heavy, PP 745 at 1.5 lb/ac was as effective as the CMPP/2,4-D mixture, but in the fourth (B31, 1963), where the weed was dense, the chemical gave unsatisfactory control. PP 407 failed to give consistently good control even at the 1.5 lb/ac rate.

(iv) Polygonum convolvulus (Black bindweed) - Table VII

PP 745 was again more effective than PP 407 against this polygonum. Excellent control was obtained with PP 745 at 1.5 lb/ac; at this rate it was as effective as the CMPP/2,4-D mixture. From the 1964 results the 1 lb rate appears to be almost as effective and certainly more active than the 1.5 lb rate of PP 407.

(v) Polygonum persicaria (Redshank) - Table VIII

Of the three polygonum species encountered in these trials, redshank proved the most susceptible to the bipyridyls. PP 745 was again slightly more active than PP 407 giving good control in both years at 0.75 lb/ac and excellent control at 1 lb/ac in 1964. The bipyridyls in both seasons proved more effective than the CMPP/2,4-D mixture.

(vi) Stellaria media (Chickweed) - Table IX

Chickweed, particularly overwintered weed, proved resistant to the bipyridyls, neither compound gave satisfactory control at the 1.5 lb/ac rate.

Initially the chemicals severely scorched this species, later there was recovery, although the plants remained smaller and less vigorous. PP 745 showed somewhat greater activity than PP 407. It will be observed that the CMPP/2,4-D mixture gave poor control in one trial (B38, 1964) but this was the trial sprayed late due to adverse weather conditions in April.

(vii) Galium aparine (Cleavers) - Table IX

Cleavers were encountered in two of the 1964 trials and in both were sprayed when the weed was well established (6"-8" high and branching). Under these conditions this species showed resistance to the bipyridyls. Although initially scorched by the sprays, cleavers subsequently recovered their "greenness" remaining stunted compared with the plants in the untreated plots. PP 745 showed greater activity than PP 407 and, in fact, gave satisfactory control in one trial at 1.5 lb/ac.

(viii) Sinapis arvensis (Charlock)

Raphanus raphanistrum (Runch) - Table X

Chenopodium album (Fat hen)

These weeds normally controlled with MCPA, proved very susceptible to the bipyridyls; 1 lb/ac gave good to excellent control. PP 745 was even highly active at 0.75 lb/ac but PP 407 was not so positive at this rate.

(ix) Spargula arvensis (Spurrey) - Table XI

This species was susceptible to the bipyridyls but PP 745 was more active than PP 407 giving excellent control at 0.75 lb/ac.

Table VI

Polygonum aviculare (Knotgrass)

Season		1963		1964	
Trial No.		B23	B31	B42	B45
Growth stage of weed		5 L.	4-8 L.Br.	Br.	Br.
Height of weed		3-4"	2-3"	4-5"	2-3"
2nd assessment - days after spraying		54	32	48	47
PP 407	0.75 lb ion/ac	2	0	3	3
	1.00 " " "	-	-	2	3
	1.50 " " "	4	1	3	4
PP 745	0.75 lb ion/ac	3	2	3	5
	1.00 " " "	-	-	4	5
	1.50 " " "	5	2	5	5
CMPP/2,4-D	6 pts/ac	-	4	5	5
('Methoxone' Extra)	7 pts/ac	5	-	-	-
% ground cover of weed in control					
1. Before spraying		0.7	11.5	8.4	5.7
2. At 2nd assessment		9.5	15.7	10.8	6.4

Table VII  
Polygonum convolvulus (Black Bindweed)

Season	1963			1964		
	B24	B27	B41	B42	B45	
Trial No.	1-2 L.	2 L.	3 L.	Sd.-Y.P.	3 L.	
Growth stage of weed	2"	2"	-	0-3"	-	
Height of weed						
2nd assessment - days after spraying	42	28	48	48	47	
PP 407	0.75 lb ion/ac	2	0	2	3	
	1.00 " " "	-	-	1	4	
	1.50 " " "	2	3	2	4	
PP 745	0.75 lb ion/ac	3	2	3	4	
	1.00 " " "	-	-	3	5	
	1.50 " " "	4	5	5	5	
CMPP/2,4-D	6 pts/ac	-	4	5	5	
	('Methoxone Extra) 7 pts/ac	5	-	-	-	
% ground cover of weed in control						
1. Before spraying	2.0	2.3	1.9	8.5	1.7	
2. At 2nd assessment	9.9	5.0	4.6	12.3	4.9	

- (x) Veronica spp. (Speedwell) - Table XI  
Galeopsis tetrahit (Hemp nettle)

Speedwell occurred in one trial and proved very susceptible to both PP 407 and PP 745, good control was obtained at 0.75 lb/ac. In comparison the CMPP/2,4-D mixture gave poor control.

Against hemp nettle PP 745 was more effective than PP 407 giving good control at 1 lb/ac. The CMPP/2,4-D mixture again gave poor control of this species.

- (xi) All weed species - Table XII

The weed density gradings for 1963 show that, over all trials, there was no marked difference between PP 407 and PP 745 and that at 1.5 lb/ac they gave weed control comparable to the CMPP/2,4-D mixture. However, at 0.75 lb/ac they were less effective than the 'hormone' weedkiller. In 1964 PP 745, generally, was superior to PP 407 and both compounds at 1 lb/ac gave cleaner plots than the CMPP/2,4-D mixture. However, in three of the trials (B41, 43 and 44) the dominant weed was corn marigold which was not controlled by the 'hormone' weedkiller and the higher mean grading for this herbicide reflects this.

Table VIII  
Polygonum persicaria (Redshank)

Season	1963		1964
	B29	B30	B46
Trial No.	3-4 L.	4-6 L.	5 L.
Growth stage of weed	2-4"	2-3"	2-5"
Height of weed	35	34	38
2nd assessment - days after spraying	4	3	3
PP 407	0.75 lb ion/ac	-	4
	1.00 " " "	5	4
	1.50 " " "	5	5
PP 745	0.75 lb ion/ac	5	4
	1.00 " " "	-	5
	1.50 " " "	5	5
CMPP/2,4-D ( 'Methoxone' Extra) 6 pts/ac	4	3	2
% ground cover of weed in control -			
1. Before spraying	36.7	10.2	10.8
2. At 2nd assessment	23.4	52.5	34.0

Table IX  
Stellaria media (Chickweed)

Galium aparine  
(Cleavers)

Season	1963		1964		1964	
	B28	B31	B38	B40	B38	B40
Trial No.	Y.P. Br.	Y.P. Br.	M.P. Fl.	M.P. Fl.	Br.	Br.
Growth stage of weed	1-3"	-	-	-	6-8"	6-8"
Height of weed	42	32	55	42	55	42
2nd assessment - days after spraying	0	0	0	0	0	1
PP 407	0.75 lb ion/ac	-	0	0	0	-
	1.00 " " "	1	2	1	0	1
	1.50 " " "	2	1	0	3	2
PP 745	0.75 lb ion/ac	-	0	-	0	-
	1.00 " " "	3	2	0	2	0
	1.50 " " "					
CMPP/2,4-D ( 'Methoxone Extra)						
6 pts/ac	5	3	-	-	-	-
7 " "	-	-	1	5	4	1
% ground cover of weed in control						
1. Before spraying	4.0	5.5	12.9	6.5	4.7	6.3
2. At 2nd assessment	10.5	39.2	28.8	5.5	7.8	16.0

Table X

Sinapis arvensis  
 (Charlock)

Raphanus raphanistrum  
 (Runch)

Chenopodium album  
 (Fat Hen)

Season	1963		1964	1963		1964	
	B26	B29	B41	B30	B44	B46	
Trial No.	B26	B29	B41	B30	B44	B46	
Growth stage of weeds	Fl.B.	3-6 L.	-	6-8 L.	Sd.-Y.P.	6-8 L.	
Height of weed	4-8"	3-6"	3-4"	2-3"	-	4-5"	
2nd assessment - days after spraying	36	35	48	34	44	38	
0.75 lb ion/ac	4	3	3	2	4	2	
PP 407 1.00 " " "	-	-	4	-	5	3	
1.50 " " "	5	5	5	4	5	4	
0.75 lb ion/ac	5	5	5	5	3	5	
PP 745 1.00 " " "	-	-	5	-	5	5	
1.50 " " "	5	5	5	5	5	5	
CMPP/2,4-D 6 pts/ac ('Methoxone' Extra)	5	5	5	5	5	5	
% ground cover of weed in control							
1. Before spraying	7.3	2.7	1.1	2.2	3.9	13.4	
2. At 2nd assessment	7.5	4.5	2.1	8.4	4.7	6.0	

Table XI

Spergula arvensis  
 (Spurrey)

Veronica  
spp.

Galeopsis tetranit  
 (Hemp-Nettle)

Season	1963	1964	1964	1964
	B30	B44	B39	B38
Trial No.	B30	B44	B39	B38
Growth stage of weed	Br.	Br.	M.P. Fl.	2-3 L.
Height of weed	2"	3-4"	-	-
2nd assessment - days after spraying	34	44	42	55
0.75 lb ion/ac	2	4	4	2
PP 407 1.00 " " "	-	5	4	1
1.50 " " "	3	5	5	2
0.75 lb ion/ac	5	5	5	3
PP 745 1.00 " " "	-	5	5	4
1.50 " " "	5	5	5	5
CMPP/2,4-D 6 pts/ac ('Methoxone' Extra)	1	4	-	-
7 pts/ac	-	-	1	1
% ground cover of weed in control				
1. Before spraying	2.2	1.4	22.7	0.6
2. At 2nd assessment	1.2	1.0	4.0	4.4

### Phytotoxicity

PP 407 and PP 745 caused slight crop scorch; there was a browning of the tips of the leaves which tended to extend down the leaf margins. The effect was transient, the crops recovered their "greenness" generally within three weeks after spraying. No other effects of treatment were observed.

Table XII  
Weed Density Gradings  
(0 = Weed Free Plot 10 = Densely Weedy Plot)

Treatment	1963										1964									
	B22	B23	B24	B26	B27	B28	B29	B30	B31	Mean	B38	B40	B41	B42	B43	B44	B45	B46	Mean	
PP 407	0.75 lb ion/ac	0.5	1.2	2.0	0	1.7	2.3	0	2.0	4.7	1.6	6.5	3.0	2.8	4.5	2.0	0.8	1.0	4.8	3.2
	1.00 " " "	-	-	-	-	-	-	-	-	-	-	5.3	-	2.5	5.0	1.0	0.6	0.5	3.8	2.7
	1.50 " " "	0	0.7	2.0	0	0.7	1.0	0	1.0	3.8	1.0	4.5	2.5	2.0	3.8	1.0	0.4	0.3	2.3	2.1
PP 745	0.75 lb ion/ac	0.5	1.7	2.0	0	2.5	2.0	0	1.2	5.0	1.7	5.0	1.5	2.0	4.3	1.8	1.0	0.1	2.3	2.3
	1.00 " " "	-	-	-	-	-	-	-	-	-	-	6.3	-	1.8	2.8	1.5	0.3	0.3	2.0	2.1
	1.50 " " "	0	0.5	1.3	0	0.7	1.7	0	0.8	2.0	0.8	4.3	1.3	1.3	1.8	1.0	0.6	0	2.8	1.6
CMPP/2,4-D ( <i>'Methoxone' Extra</i> )	6 pts/ac	-	-	-	0	4.3	0.7	0	0.8	1.5	)0.8	-	-	3.5	0.8	5.3	5.0	0.1	4.8	)3.3
	7 pts/ac	0	0	0.2	-	-	-	-	-	-		4.3	2.3	-	-	-	-	-	-	
Untreated controls		0.4	5.4	5.7	0.8	10.0	9.5	3.6	10.0	8.5	6.0	9.1	5.3	6.8	8.2	10.0	5.5	3.6	8.5	7.1

N.B. No gradings for trial B39 as Veronica had died naturally by time of final assessment

Table XIII  
Grain Yields (cwts/ac) Adjusted to 85% Dry Matter

Trial Reference No.		B51	B52	B53	B54	B55	B56	B57	B58
Treatment		Winter oats Powys	Winter wheat Cappelle	Winter barley Dea	Spring wheat Opal	Spring wheat Opal	Winter wheat *Spring sown Rothwell- Perdix	Spring barley Union	Spring barley Proctor
PP 407	1.0 lb ion/ac	33.5	31.7	26.6	33.4	40.5	53.0	21.6	31.6
	2.0 lb " "	32.9	33.8	28.7	32.5	38.5	52.7	21.2	31.7
	3.0 lb " "	33.4	33.2	29.3	32.3	40.4	52.9	21.4	32.3
PP 745	1.0 lb ion/ac	34.5	32.2	27.1	34.9	41.3	52.3	22.2	31.8
	2.0 lb " "	33.1	32.8	27.3	33.0	40.8	54.0	21.0	32.3
	3.0 lb " "	33.0	34.9	28.4	31.4	39.2	51.5	21.8	28.9
CMPP/2,4-D ( 'Methoxone' Extra)	6 pts/ac	-	-	-	36.2	43.9	53.4	23.1	30.6
	7 pts/ac	33.7	33.3	27.8	-	-	-	-	-
Control untreated		33.5	-	25.7	-	41.8	-	-	-
Standard error (single plot)		±2.3	±1.8	±1.2	±1.3	±1.5	±1.5	±1.8	±2.3
Coefficient of variation		6.9%	5.4%	4.5%	3.8%	3.7%	2.9%	8.6%	7.2%
Significant difference between individual treatment means		Not Significant	Not Significant	1.8	1.9	2.2	Not Significant	Not Significant	Not Significant

- Notes: (1) PP 407 and PP 745 averaged over rates : No significant difference between chemicals in any of the trials.  
 (2) Rate means averaged over PP 407 & PP 745 : In trial B53 the 3.0 lb ion/ac rate gave a significantly higher yield than the 1.0 lb ion/ac rate. In trial B54 the 2.0 lb ion/ac and the 3.0 lb ion/ac gave a significantly lower yield than the 1.0 lb ion/ac rate. In the other 6 trials there were no significant differences.

## Large plot yield trials - 1964

### (a) Effect of treatment on yield

The yield data from the eight large plot trials are presented in Table XIII. Significant differences between treatments were obtained in three of the trials as follows:-

#### (i) B54 - spring wheat var. Opal

PP 407, at all rates, gave a significantly lower yield than the CMPP/2,4-D herbicide. This may be due to the relatively poor control obtained of a moderate infestation of knotgrass. There were no differences in yield between the rates of PP 407. The yield obtained from PP 745 at 1 lb/ac was not significantly different from the CMPP/2,4-D mixture but the 2 & 3 lb rates gave significantly lower yields. This is considered due to phytotoxicity as PP 745 at the higher rates gave satisfactory weed control.

#### (ii) B55 - spring wheat var. Opal

PP 407 and PP 745, at all rates, gave a significantly lower yield than the CMPP/2,4-D mixture but the last mentioned did not give a significantly higher yield than the untreated. PP 745 at the 1 and 2 lb rates and PP 407 at the 1 and 3 lb rates were not significantly different from the untreated but the 3 lb rate of PP 745 and the 2 lb rate of PP 407 did give a significantly lower yield than the untreated. As this was a slightly weedy site these differences are probably due to slight phytotoxicity at the higher rate, in particular with PP 745.

#### (iii) B53 - winter barley var. Dea

In contrast to the results from the previous two trials, PP 407 at 2 and 3 lb/ac and PP 745 at 3 lb/ac gave significantly higher yields than the untreated. Furthermore, PP 407 at 2 and 3 lb/ac gave significantly higher yields than the 1 lb rate. The CMPP/2,4-D mixture gave a significantly higher yield than the untreated but there was no difference between this herbicide and the bipyridyls. As this was a weed free trial, at the time of spraying and remained so throughout the season, it is difficult to explain the reason for the yield increases due to treatment.

### (b) Effect of treatment on thousand grain weight

In the eight trials there were no significant differences between individual treatments but in trial B54 the thousand grain weight for the mean of the 3 lb/ac treatments of PP 407 and PP 745 was significantly lower than that for the CMPP/2,4-D herbicide and the 1 and 2 lb rate means of the bipyridyls. In trial B56 the 3 lb rate mean was again significantly lower than the 1 and 2 lb rate means.

## DISCUSSION

The results from this series of field trials show that the two new substituted 1,1'-carbamoylmethyl-4,4'-bipyridylium compounds coded PP 407 and PP 745 are extremely effective as contact herbicides for selective post-emergence weed control in cereals. At 1-1.5 lb ion/ac they have given good control of a number of weed species either moderately or very resistant to MCPA and 2,4-D. In particular Chrysanthemum segetum, which at present is only satisfactorily controlled by the toxic dinitro compounds, DNOC and dinoseb, has proved very susceptible to both bipyridyls. Furthermore PP 407 and PP 745 have proved superior to a herbicide containing CMPP/2,4-D for the



control of Polygonum persicaria and Veronica spp. and PP 745 has also given better control of Galeopsis tetrahit. The experimental compounds have given control of Tripleurospermum maritimum ssp. inodorum certainly equal to and occasionally better than that achieved with the CMPP/2,4-D weedkiller. In addition to controlling these resistant weeds both PP 407 and PP 745 have given good control of the MCPA susceptible weeds, Sinapis arvensis, Raphanus raphanistrum and Chenopodium album. However certain species, Stellaria media and Galium aparine, for which the present recommended control is CMPP have shown resistance to PP 407 and PP 745.

The results indicate that PP 745 is more active than PP 407; particularly for the control of Polygonum convolvulus and Polygonum aviculare and in general activity towards Stellaria media and Galium aparine. PP 745, generally, gave good control of most of the species encountered in the weed control trials. However the data from the yield trials indicate that the greater herbicidal activity is correlated with a slight increase in crop phytotoxicity. In one trial (B54) the 2 & 3 lb rates of PP 745 gave a slight but nevertheless significant decrease in yield compared with the 1 lb rate whereas PP 407 did not. In another trial (B55) PP 745 at 3 lb gave a significantly lower yield than the untreated but PP 407 caused no depression at this rate.

Although both experimental compounds caused slight but transient scorch to the crop, the yield data show that at 1-1.5 lb/ac, the rates likely to be recommended for weed control, the chemicals are unlikely to depress yield.

PP 407 and PP 745 have properties similar to diquat and paraquat in that they are quick-acting, rainfast and non-toxic contact herbicides. They are inactivated on contact with soil and field experience, so far, with these herbicides suggests that temperature is probably not a critical factor governing their efficiency. A herbicide with these properties is likely to be of interest in the following situations:

- (i) For the control of specific weed problems, e.g. corn marigold
- (ii) For use where ear malformities are detrimental, e.g. cereal seed crops
- (iii) For use in a cool spring
- (iv) For use where rapid kill of weed is required and
- (v) For application to cereals immediately prior to undersowing.

#### Acknowledgements

The authors wish to thank their colleagues, Mr. J.C. Casselton and Mr. D.J. England for their assistance in the conduct of these field trials and for their help in the preparation of this paper. Acknowledgement is also made to the farmers who kindly allowed these trials to be carried out.

#### References

- Cronshey, J.F.H. (1961) A review of experimental work with diquat and related compounds. Weed Research 1 (1), p. 68-77
- Darter, I.E. & Wright, N. (1963) Paraquat: A new herbicide and desiccant. Pesticides Abstracts and News' Summary, Section C 2 (3), p. 203-206.

SELECTIVE WEED CONTROL IN BRITISH CEREAL CROPS  
WITH 4-AMINO-3,5,6-TRICHLOROPICOLINIC ACID.

H. M. Lawson

Dow Chemical Company (UK) Limited, Kings Lynn, Norfolk.

Summary. 4-amino-3,5,6-trichloropicolinic acid in combination with various phenoxy herbicides shows considerable promise for the control of a wide range of important weeds of British cereals. Suitable dosage rates of 4-amino-3,5,6-trichloropicolinic acid range from 3/8 oz. a.e./ac. upwards. Barley and oats are highly tolerant and possible formulations for commercial use in these crops may contain 3/4 oz. a.e./ac. Wheat is somewhat less tolerant to high levels of 4-amino-3,5,6-trichloropicolinic acid, but there appears to be adequate safety margin for formulations containing approximately 1/2 oz. a.e./ac.

INTRODUCTION

Field trials to evaluate 4-amino-3,5,6-trichloropicolinic acid for weed control in cereals in the United Kingdom were commenced following American information (1) that at very low dosage rates excellent control of Polygonum species could be obtained. Logarithmic dosage trials confirmed that this was the case for all four common annual Polygonum species of U.K. cereal crops. These, and other trials in various other European countries, also showed that various mayweed species were very susceptible at as little as 1/2 oz. a.e./ac. Chickweed, and brassica weeds such as runch and charlock were considerably less susceptible. The early evidence indicated therefore, that 4-amino-3,5,6-trichloropicolinic acid had considerable promise on two of the most important MCPA-resistant weed families of the United Kingdom, and that combinations with various phenoxy herbicides should result in very broad spectrum weed control.

Later work in the United Kingdom was of two kinds. Further logarithmic trials were carried out to evaluate the potassium salt of 4-amino-3,5,6-trichloropicolinic acid as TORDON\* 22K herbicide in mixture with several phenoxy herbicides to find the most suitable combination and optimum ratios for British conditions. In addition, a series of 10 identical, replicated, small plot trials was laid down to extend the information on weed control efficiency. 4-amino-3,5,6-trichloropicolinic acid alone at several dosage rates was compared against the same rates with added MCPA. The sites were well distributed over the country and included

\* Registered Trademark of The Dow Chemical Company

various types of weed association.

Very weedy crops were chosen, and accurate yield evaluation was not intended, but frequent notes were taken on crop reaction to herbicide treatment, and yield samples hand harvested. The results of these regional trials showed that MCPA/4-amino-3,5,6-trichloropicolinic acid combinations have considerable promise on a wide range of annual weeds at dosage rates which offer no hazard to the cereal crop. Chickweed, however, proved less susceptible to this combination than to mecoprop. Logarithmic trials confirmed that mecoprop/4-amino-3,5,6-trichloropicolinic acid combinations were most effective where this weed was present.

Further trials conducted this year to establish crop tolerance, were sited in major arable areas of the United Kingdom in weed free or only lightly infested crops. These identical trials were designed to assess critically the margin of safety of combined 4-amino-3,5,6-trichloropicolinic acid/MCPA and 4-amino-3,5,6-trichloropicolinic acid/mecoprop formulations on the major cereals. Plot size was 1/40th ac. with four replications, and all trials were combine harvested.

## SYNOPSIS OF RESULTS

### 1. Weed Control.

Table I summarises weed control performance, as compiled from 1963 regional field trials. Since the various trials encompassed all stages of growth and conditions of very dense and varied mixed weed populations in crops of varying vigour, the performance of 4-amino-3,5,6-trichloropicolinic acid alone, and in mixture with MCPA, is shown in comparison with that of mecoprop and with MCPA as standards at each site. Several of the sites were sprayed quite late.

4-amino-3,5,6-trichloropicolinic acid alone compared very favourably with the mecoprop standard, except where chickweed, charlock or runch were major weeds. The addition of 12 oz. (a.e.) MCPA to 4-amino-3,5,6-trichloropicolinic acid brought the brassica weeds into the spectrum and greatly enhanced the control of hempnettle, as well as contributing to the overall weed control. The relative weed control efficiency of as little as 3/8 oz. (a.e.) 4-amino-3,5,6-trichloropicolinic acid, particularly in association with MCPA, was most impressive.

Work in the United States, and in other countries where 2,4-D is the predominant phenoxy herbicide for cereal weed control, has indicated that combinations of 2,4-D with about  $\frac{1}{2}$  oz. a.e./ac. 4-amino-3,5,6-trichloropicolinic acid are also most promising. It appears, therefore, that 4-amino-3,5,6-trichloropicolinic acid can be used at remarkably low dosage rates in combination with a number of other commonly used

TABLE I.  
Percentage overall weed control in 1963 trials

Treatment (oz. a. e./ ac.) Trial	Meco- prop 40	MCPA 12	T* 3/8	T $\frac{3}{4}$	T $1\frac{1}{2}$	TMCPA 3/8 + 12	TMCPA $\frac{3}{4}$ + 12	TMCPA $1\frac{1}{2}$ + 12	Main weed spp present
CB 5 - Nth. Muskham (Nottinghamshire)	40	20	20	35	40	65	63	77	Pale persicaria Chickweed Sowthistle Cleavers Redshank Fat hen
CB 6 - Theale (Berkshire)	55	35	42	83	87	65	82	85	Redshank Pale persicaria Fat hen
CB 7 - Coventry (Warwickshire)	32	23	10	40	63	45	63	90	Corn marigold White charlock Pansy
CB 8 - Salisbury (Wiltshire)	77	53	55	53	85	73	77	87	Chickweed Speedwell Knotgrass B. Bindweed Mayweed Pansy
CB 9 - Nth. Creake (Norfolk)	77	50	53	75	83	82	95	96	Polygonum spp Pansy Mayweed Fat hen Speedwell Chickweed
CB 10 - Brotherton (Yorkshire)	10	12	13	43	75	53	67	90	Knotgrass Pale persicaria Fat hen
CB 11 - Radcliffe (Northumberland)	85	70	20	57	70	73	83	92	Speedwell B. Bindweed Knotgrass Chickweed Charlock
CB 12 - Dalbeattie (Kirkcudbright)	88	72	35	65	73	73	90	90	Hempnettle Corn marigold Chickweed Redshank
CB 13 - Dechmont (W. Lothian)	70	63	55	72	73	85	83	95	Hempnettle Knotgrass Yellow charlock Chickweed Mayweed Fat hen
CB 14 - Udney (Aberdeenshire)	83	55	57	73	85	85	83	88	Hempnettle Mayweed Chickweed Knotgrass Spurrey

\* 4-amino-3,5,6-trichloropicolinic acid

TABLE II.  
Average yield relative to weedy control (1963 trials).

Treatment (oz. a. e./ ac.) Trials	Control	Mecoprop 40	T* 3/8	T $\frac{3}{4}$	T $1\frac{1}{2}$	T:MCPA 3/8 + 12	T:MCPA $\frac{3}{4}$ + 12	T: MCPA $1\frac{1}{2}$ + 12
Barley (5 sites)	100	102	100	105	106	109	106	107
Wheat (2 sites)	100	117	109	108	115	122	110	116
Oats (2 sites)	100	106	105	98	96	121	104	102

Plot size -  $6^x \times 4^x$  with 3 replications - hand harvested.

TABLE III.  
Average yield relative to non-weedy control (1964 trials)

Treatment (oz. a. e./ ac.) Trials	Control	Mecoprop 36	Mecoprop:T 24 + $\frac{3}{4}$	Mecoprop:T 24 + $1\frac{1}{2}$	Mecoprop:T 24 + $2\frac{1}{4}$
Spring Barley (6 sites)	100	98	98	98	95
Spring Wheat (3 sites)	100	104	97	86	80
Winter Wheat (3 sites)	100	99	99	89	79
Spring Oats (1 site)	100	101	100	100	106

Plot size 1/40th acre with 4 replications - combine harvested.

\* 4-amino-3, 5, 6-trichloropicolinic acid

herbicides to control many of the important weed associations of cereal crops around the world.

## II. Yield and Crop Tolerance

Yields from the 1963 trials are summarised in Table II.

1964 yield trials were assessed in detail throughout the growing season. The stage of growth of the crops at treatment varied from the 5 leaf to the early jointing stage. The yield data are summarised in Table III.

In assessing Table III it is necessary to bear in mind that the optimum level of 4-amino-3,5,6-trichloropicolinic acid in these formulations for the control of susceptible British weed species appears to be about  $\frac{3}{4}$  oz. a. e. /ac. As far as barley and oats are concerned, it is clear that the margin of tolerance is sufficiently adequate for this dosage rate to be used. Neither cereal showed any signs of growth check, delayed maturity, or ear malformation at any of the dosage rates used in these trials.

The situation as regards wheat was more complex. Although the lowest dosage rate used (24 oz. a. e. mecoprop plus  $\frac{3}{4}$  oz. a. e. 4-amino-3,5,6-trichloropicolinic acid/ac.) had no adverse effects on growth or yield, the much higher rates gave variable responses. Therefore, more work is planned to determine margins of safety under varying conditions. Since as little as  $\frac{3}{8}$  oz. a. e. 4-amino-3,5,6-trichloropicolinic acid/ac. rivalled 12 oz. a. e. MCPA/ac. (see Table I) for overall weed control efficiency, except in the presence of brassica weeds, the somewhat lower tolerance of wheat still leaves room for suitable mixed formulations with a wide safety margin to be developed, particularly where such problem weeds as mayweed and Polygonum species are important.

Development in the United States and continental Europe is proceeding along similar lines.

### Soil Residues

4-amino-3,5,6-trichloropicolinic acid is more persistent in the soil than the phenoxy herbicides. High rates above 2 lb. a. e. /ac. may persist for extended periods. Recent experiments in the United States (2), on many different soil types, indicate that the very low dosage rates envisaged for use in cereal crops decompose more rapidly.

In the United Kingdom, recent assessments of various susceptible crops, including potatoes, sugar beet and clover, planted in the year after spring treatment of cereals, suggest that at the very low dosage rates involved, the disappearance of soil residues is quite rapid under

British conditions. A detailed programme of soil sampling is under way to establish the picture on major soil types.

#### Crop Residues.

Decay curves for 4-amino-3,5,6-trichloropicolinic acid are being plotted on samples of treated cereal vegetation sampled at intervals until harvesting, and trials to check the effects of possible straw residues on susceptible horticultural crops are planned. Grain samples from harvested trials are also being analysed for possible residues.

This paper is only intended as a progress report, and detailed results with statistical data will be presented in a later publication.

- (1) TORDON\* Information Manual prepared by : -  
The Dow Chemical Company, Midland, Michigan, U.S.A.
- (2) Unpublished reports - Plant Science, Research & Development  
Department, The Dow Chemical Company.

#### Acknowledgements.

The assistance and co-operation of the farmers on whose land these trials were conducted, is gratefully acknowledged.

A MECOPROP + 2,4-D MIXTURE FOR THE CONTROL OF  
MCPA-RESISTANT WEEDS IN CEREALS

K. G. Drake and A. F. Raybons  
Plant Protection Ltd., Fernhurst

Summary

Results are presented from seven trials in winter and spring cereals in which the efficiency of control of weeds resistant to MCPA by a mecoprop + 2,4-D mixture\* is compared with a standard dichlorprop treatment.. 45 oz. a.i. in spring cereals and 52.5 oz. a.i. of the mecoprop + 2,4-D mixture per acre in winter cereals caused no adverse phytotoxic effects to the crop and provided good control of Polygonum aviculare, P. convolvulus, P. persicaria, Stellaria media, Galium aparine and Tripleurospermum maritimum ssp. inodorum. The mixture was more active against P. aviculare, T. maritimum and S. media than 40 oz. of dichlorprop per acre although in one trial dichlorprop was more effective against G. aparine and in another was quicker to control P. persicaria.

INTRODUCTION

It has been shown that mecoprop gives excellent control of cleavers (Galium aparine) and chickweed (Stellaria media) (Lush, 1956). In order to improve the activity of this chemical against other MCPA-resistant weeds and more especially the Polygonum species a mixture of mecoprop and 2,4-D was devised and tested in field trials. This particular series was completed in 1964. The mecoprop + 2,4-D mixture was compared with dichlorprop already reported by a number of workers as being particularly effective against the Polygonum species (Parker, C. 1962; Holroyd, J. 1962; and Davison, J.G. and Evans, S.A. 1962).

METHODS AND MATERIALS

Four rates of a formulated mixture of the ethanolamine salts of mecoprop and 2,4-D were compared with the potassium salt of dichlorprop as a standard. The mixture contained 48 oz. of mecoprop with 12 oz. of 2,4-D per Imperial gallon. 6 pints/acre of this mixture on spring cereals and 7 pints on winter cereals (a commercial recommendation) were compared with a lower rate and two higher rates. The treatments are shown in Table 1.

Table 1. Treatments

Herbicide	Rate/acre			
	Spring Cereals		Winter Cereals	
	a.i. (oz.)	pt.	a.i. (oz.)	pt.
1 Unsprayed control	-	-	-	-
2 Dichlorprop	40		40	
3 Mecoprop + 2,4-D	30 + 7.5	5	36 + 9.0	6
4 Mecoprop + 2,4-D**	36 + 9.0	6	42 + 10.5	7
5 Mecoprop + 2,4-D	54 + 13.5	9	63 + 15.75	10.5
6 Mecoprop + 2,4-D	72 + 18	12	84 + 21	14

\* The work described was carried out with a standard product as sold under the proprietary name 'Methoxone' Extra.

\*\* Current (1964) commercial recommendation.



All treatments were applied in 20 gallons of water per acre using a Land Rover mounted sprayer.

In all, seven trials comprising two on winter wheat, one on spring wheat and four on spring barley were laid down. Treatments, including the unsprayed control, were replicated four times using a randomised block design and a plot size of 50 x 5 yards. Details of the crops sprayed and dates of spraying are shown in Table 2.

Table 2 Trial Details

No.	County	Crop	Variety	Date of spraying	Crop stage
A1	Wiltshire	Winter wheat	Cappelle	30.4	Fully tillered 12 - 15 in. high
A2	"	"	Charplein	11.5	Fully tillered 10 - 12 in. high
A3	"	Spring barley	Pallas	12.5	5 - 6 leaves 3 tillers.
A4	Gloucestershire	"	Proctor	12.5	5 - 6 leaves 2 tillers.
A5	"	"	Rika	14.5	5 leaves
A6	"	Spring wheat	Opal	20.5	5 - 6 leaves 1 tiller.
A7	Wiltshire	Spring barley	Rika	27.5	5 - 6 leaves 2 tillers.

A single scoring method using a single figure to incorporate both vigour and number of weed species per unit area of ground was used to assess the effects of spray treatment on each main weed species present. Mean scores were calculated from two observations made by three observers for each plot. By direct comparison with the unsprayed control the percentage weed control for each weed species was estimated. Assessments were repeated on three dates i.e. three, six and nine weeks after spraying.

A single scoring method was also used to assess the amount of leaf scorch present approximately one week after spraying where this occurred. The grain yield, later adjusted for differences in moisture content was estimated by combining a strip 40 yards long through each plot.

## RESULTS

### A Weed Control

The effects of spray treatments on individual weed species expressed as the percentage weed control on each date of assessment is shown in Tables 3 and 4.

Table 3. Percentage weed control

TRIAL	<u>Folygonum aviculare</u> barnyardgrass					<u>Folygonum convolvulus</u> black bindweed			<u>Folygonum persicaria</u> redshank	<u>Stellaria media</u> chickweed					<u>Tripleurospermum noritimum</u> scentless mayweed			
	A2	A3	A4	A5	Sd	A4	A6	Sd	A6	A1	A2	A5	A6	Sd-Sh	A2	A3	Sd-Sh	
Stage of weed growth at spraying	Sh	Sh	Sd	Sd		Sd	Sd		Sd	Sh	Sh	Sd	Sd-Sh		Sh	Sd-Sh		
CROP	WW	SB	SB	SB	Mean	SB	SW	Mean	SW	WW	WW	SB	SW	Mean	WW	SB	Mean	
Treatments	Weeks after spraying																	
Dichlorprop 4.0 oz.	3	44	60	54	63	55	65	83	74	85	49	69	94	64	69	38	72	55
	6	44	44	57	97	61	68	97	82	78	98	90	a	52	85	54	89	72
	9	55	67	a	92	71	a	95	95	88	a	94	a	53	71	68	67	67
Mecoprop + 2,4-D oz./ac. 30 + 7.5 or 36 + 9.0* (5 or 6 pt.)	3	77	64	69	85	74	65	79	72	50	48	81	94	65	72	49	54	51
	6	86	64	96	100	87	82	92	87	58	81	91	a	60	73	64	67	65
	9	85	78	a	100	88	a	95	95	73	a	98	a	66	88	84	73	78
36 + 9.0 or 42 + 10.5* (6 or 7 pt.)	3	74	78	63	86	75	67	48	58	35	65	85	94	63	77	47	54	50
	6	93	80	90	100	88	85	89	87	64	95	96	a	58	77	62	87	75
	9	81	85	a	100	89	a	86	86	85	a	100	a	75	92	85	93	89
54 + 13.5 or 63 + 15.75* (9 or 10½ pt.)	3	81	85	72	86	81	79	93	86	42	56	88	94	68	76	53	78	65
	6	92	86	88	100	92	77	97	87	67	100	100	a	68	92	79	85	82
	9	84	92	a	100	92	a	100	100	73	a	98	a	77	92	90	91	90
72 + 18 or 84 + 21* (12 or 14 pt.)	3	78	86	84	96	85	82	93	88	81	58	91	97	75	80	61	71	66
	6	92	89	90	100	93	83	97	90	78	98	100	a	74	93	76	96	86
	9	83	94	a	100	92	a	98	98	85	a	98	a	85	94	92	96	94

Sd. = seedlings  
Sh. = shoots

\*Higher rate used on Winter wheat (WW)  
Lower rate used on Spring wheat (SW) and barley (SB)

a = absent from unsprayed control

Table 4. Percentage weed control

TRIAL	Galium <u>cleavers</u>			Lambium <u>sp. + Galium</u> red dead nettle			Veronica sp. speedwell	Chrysanthemum <u>sp.</u> corn marigold	Lapsana <u>cornifolia</u> nigellwort
	A1 A2 A5	EP Sh Sh	EP S2	EP S2	EP S2	EP S2	A1	A7	A5
Stage of weed growth at spraying	EP Sh Sh	EP S2	EP S2	EP S2	EP S2	EP S2	EP	Sh	Sh
CROP	WW SW SW	Mean	WW SB	Mean	WW	SB	Mean	SB	SB
Treatments	Weeks after spray- ing								
Dichlorprop 40 oz.	3	50 84 74	69	46 64	55		39	26	64
	6	90 83 73	82	37 48	43		60	23	32
	9	93 83 78	85	6 a	6		a	24	39
Mecoprop + 2,4-D oz./ac. 30 + 7.5 or 36 + 9.0* (5 or 6 pt.)	3	46 83 63	64	15 38	42		48	53	49
	6	66 83 60	70	32 27	29		36	54	30
	9	77 90 63	77	24 a	24		a	56	41
36 + 9.0 or 42 + 10.5* (6 or 7 pt.)	3	51 70 61	62	39 47	43		50	65	51
	6	87 70 58	72	47 24	36		50	66	32
	9	95 78 75	82	18 a	18		a	73	42
54 + 13.5 or 63 + 15.75* (9 or 10½ pt.)	3	51 86 73	70	53 56	55		55	64	56
	6	96 95 77	89	46 48	47		48	76	56
	9	97 98 81	92	0 a	0		a	83	56
72 + 18 or 84 + 21* (12 or 14 pt.)	3	61 92 75	76	54 69	66		57	79	71
	6	98 91 79	89	58 50	48		88	85	64
	9	100 94 89	94	24 a	24		a	88	65

Sd. = seedlings

Sh. = shoots

EP = established plants

\*Higher rate used on Winter wheat (WW)

Lower rate used on Spring wheat (SW) and barley (SB)

a = absent from unsprayed control

(i) Polygonum aviculare (knotgrass) was always considerably reduced in vigour and number by the mecoprop + 2,4-D mixture at all rates of application. A small additional response to the highest compared with the lowest rates in two trials where the P. aviculare was more advanced at the time of spraying (A2 and A3) largely disappeared by the third assessment. Although the amount of P. aviculare in these two trials was reduced by the dichlorprop treatment, especially by the final assessment, the standard of control was lower than with the mecoprop/2,4-D treatment. In the remaining two trials with the P. aviculare at the seedling stage, although control improved in the dichlorprop plots with time, this was probably largely a result of crop competition since the weed was almost completely absent from the unsprayed control also in nine weeks after spraying.

(ii) Polygonum convolvulus (black bindweed) occurred in two trials, and was at the seedling stage in both cases at the time of spraying. Control was initially good in all treatments so that the weed eventually almost completely disappeared.

(iii) Polygonum persicaria (redshank). In the one trial where this weed occurred in significant proportions it was particularly quickly reduced in vigour by dichlorprop. The standard of control by the mecoprop + 2,4-D mixture although slower to take effect considerably improved with time.

(iv) Stellaria media (chickweed) was normally well controlled by the mecoprop + 2,4-D mixture although where the weed infestation was greatest (A6) there was a better response to the higher rates. In this trial the effect of dichlorprop was markedly less than even the lowest mecoprop + 2,4-D rate.

(v) Tripleurospermum maritimum ssp. inodorum (scentless rayweed). In a relatively open crop with a thick infestation of established T. maritimum plants (A2) the mecoprop + 2,4-D mixture produced markedly good and also superior control compared with the dichlorprop treatment. In a second trial where T. maritimum was between the seedling and shoot stage when sprayed (A3) the mecoprop + 2,4-D mixture virtually eliminated the weed at all but the lowest rate of application whereas the weeds in the plots sprayed with dichlorprop showed evidence of regrowth by the final assessment.

(vi) Gallium aparine (cleavers). On average the degree of control of G. aparine produced by the mecoprop + 2,4-D mixture at the lower rates compared favourably with the dichlorprop treatment, but the latter showed slightly greater activity in one trial (A5).

(vii) Other weeds. Of the other weeds which occurred in assessable proportions (these included Lamium purpureum (red dead nettle), Veronica sp. (speedwell), Lapsana communis (nipplewort) and Chrysanthemum segetum (corn marigold)) only the last mentioned, which was reduced in vigour at the highest rates of mecoprop + 2,4-D was affected by either treatment.

## B Crop Effects

### Leaf Scorch.

The effect of spray treatment on the amount of leaf scorch

persisting until approximately one week after spraying is shown in Table 5.

Table 5. The effect of spray treatment on the amount of leaf scorch approximately one week after spraying (Mean of 5 trials)

Treatment	Leaf scorch
	0 = no scorch 10 = scorch of terminal inch on 20% of leaves.
Dichlorprop 40 oz.	3
Mecoprop + 2,4-D 5 - 6 pt.*	3
Mecoprop + 2,4-D 6 - 7 pt.*	4
Mecoprop + 2,4-D 9 - 10.5 pt.*	7
Mecoprop + 2,4-D 12 - 14 pt.*	10

\* Higher rate used on winter wheat

The amount of leaf scorch varied from slight scorch of the end few millimetres on a few leaves to complete scorch of approximately 1 inch on 20% of the leaves. In each trial the scorch completely disappeared in 3 weeks.

### C Grain Yields

The effect of spray treatment on the yield of grain is shown in Table 6.

Table 6. The effect of spray treatment on the grain yield (cwt./ac. at 16% moisture content)

Treatment	TRIAL								Mean
	A1 W.	A2 W.	A3 S.	A4 S.	A5 S.	A6 W.	A7 S.		
	Wheat	Wheat	Barley	Barley	Barley	Wheat	Barley		
Unsprayed control	43.7	31.0	44.0	35.8	29.9	34.7	35.9	36.4	
Dichlorprop, 40 oz.	44.1	41.0	46.5	35.3	29.5	33.5	37.4	38.2	
Mecoprop + 2,4-D 5-6 pt.	48.8	39.1	46.1	36.8	29.9	32.3	36.8	38.5	
Mecoprop + 2,4-D 6-7 pt.	45.2	39.5	46.2	35.8	29.6	35.3	38.5	38.6	
Mecoprop + 2,4-D 9-10.5 pt.	46.3	39.6	47.2	35.9	29.3	34.4	37.5	38.6	
Mecoprop + 2,4-D 12-14 pt.	40.6	39.2	42.7	35.9	29.9	32.9	37.6	37.0	
SE <sup>±</sup>	2.31	0.98	1.02	0.39	0.41	0.89	0.78		
L.S.D. (0.05)	6.54	2.87	2.99	1.15	1.19	2.59	2.29		

While there was an increase in the mean grain yield in all treatments where spraying was carried out in individual trials the increase was only consistently significant ( $P = 0.05$ ) in A2 where the weed infestation was particularly great.

In no trial was there a significant reduction in grain yield compared with the unsprayed control where the highest rates of chemicals were applied. In two trials, however, (A1 and A3) the highest rate (12 - 14 p.t./ac. of mecoprop + 2,4-D) caused a yield reduction compared with the lowest rate.

#### DISCUSSION

The difference in the level of the mecoprop/2,4-D mixture used for weed control against weeds in autumn sown crops compared with those sown in the spring was a result of previous work (Wheeler, 1960) which had shown that overwintered, established weeds were better controlled by 40 oz. per acre of mecoprop than 32 oz. In this series of experiments this was supported by the results obtained in one trial (A1) on overwintered Galium aparine and Stellaria media which were better controlled by the higher level of application of the mecoprop/2,4-D mixture containing 42 oz. of mecoprop (7 pints per acre) compared with 36 oz. per acre (6 pints/acre) even though there was no similar response to 42 oz. in the second trial (A2).

The level of 6 pints/acre on spring sown cereals and 7 pints/acre on winter cereals gave good control of Polygonum aviculare, Stellaria media, Galium aparine, P. convolvulus, P. persicaria and Tripleurospermum maritimum ssp. inodorum in all cases where these weeds occurred. Compared with the standard rate of dichlorprop employed the mecoprop + 2,4-D mixture was advantageous on P. aviculare, T. maritimum and S. media particularly where the infestation of these weeds was dense or relatively well advanced in its stage of growth. Compared with 6 and 7 pints of the mecoprop + 2,4-D mixture on spring and winter cereals respectively 40 oz./ac. of dichlorprop was quicker to control Polygonum persicaria in the single trial where this weed occurred and slightly more effective on Galium aparine in one trial.

The amount of leaf tip scorch caused by the lowest rates of mecoprop + 2,4-D were similar or only marginally greater than that caused by the standard dichlorprop treatment while there was a complete absence of ear malformation in all trials.

It is important to note that the reduction in grain yield occurring at the highest rate of mecoprop + 2,4-D in two of the seven trials compared with the lowest rate was not significant when compared with the unsprayed control. While it is possible to explain this phenomenon through the absence of both weed competition and crop phytotoxicity in the plots where the lowest rate of chemical was applied, in both these trials spraying was carried out particularly late. The jointing stage had almost been reached when it was eventually possible to apply the chemical. In the remaining five trials where either the winter wheat was sprayed when fully tillered or the spring wheat or barley was at the five leaf stage or soon after, there was a complete absence of a trend towards a depression in grain yield even at the highest mecoprop + 2,4-D rate.

In conclusion, therefore, the data presented here show quite clearly that at the rate of 45 oz. a.i. on spring cereals and 52.5 oz. a.i. of the mecoprop + 2,4-D mixture/acre in winter cereals a broad spectrum of weed control is possible with no evidence of adverse effects in the form of crop phytotoxicity.

#### Acknowledgements

Grateful thanks are due to the farmers who allowed the trials to be carried out on their farms. The authors also wish to acknowledge the helpful advice freely given by Messrs. I.B. Darter and A.F.J. Wheeler and also the assistance given by Mr. M.G. Jackson at every stage of the work.

#### References

1. Davison, J.G. and Evans, S.A. (1962). The control of MCEA-resistant weeds, particularly Polygonum species, in cereals. Proc. 6th Brit. Weed Control Conf., 1960, p. 179.
2. Holroyd, J. (1960) New post-emergence herbicides in cereals; Part II. Field experiments. Proc. 6th Brit. Weed Control Conf., 1960, p. 157.
3. Lush, G.B. 1956. A new development in selective weed control. Part I. Introduction and weed control data. Proc. 3rd Brit. Weed Control Conf., 1956, p. 625.
4. Parker, C. New post-emergence herbicides in cereals; Part I Pot experiments. Proc. 6th Brit. Weed Control Conf., 1960, p. 141.
5. Wheeler, A.F.J. (1960). Selective weed control in cereals with mecoprop and sodium monochloracetate. Proc. 4th Brit. Weed Control Conf., 1958, p. 39.

WEED CONTROL IN CEREALS IN FRANCE  
PRESENT POSITION - EXPERIMENTS CARRIED OUT IN 1964

R. Faivre-Dupaigre - J. Rognon

Institut Technique des Céréales et des Fourrages - Paris

Summary Investigations carried out in France in half the cereal-growing areas have shown that weed control is mostly practised on the larger farms ; the products containing 2,4-D or MCPA are the most commonly used. The experiments carried out in 1963-64 gave the following results :

The mixture of dicamba and MCPA is well tolerated by spring barley, not quite so well by winter wheat. The mixture of dinoseb and 2,4-D or MCPA - at the doses which were used - proved toxic to spring barley, less toxic to winter wheat, although more toxic than 2,4-D or MCPA. Ioxynil is an interesting weed-killer. Against wild oats, in barley triallate and barban can successfully be used, but in winter wheat no product can be recommended. Against blackgrass, in wheat simazine is effective but it sometimes affects the growth of the wheat ; prometryne and the triazines G 36393 and GS 12344 do not seem to be much more selective ; dichlobenil and buturon show some measure of selectivity ; in winter barley triallate can be used effectively.

Agrostis spica-venti reacts in more or less the same way as blackgrass to these different products.

STATISTICAL DATA AND PRODUCTS USED

An investigation into cereal production was carried out in 1963 in 19 departments of the North, Centre and South-West of France, an area which produces approximately half of all the crops grown in France. This investigation, directed by the Ministry of Agriculture and the National Institute of Statistics and Economical Studies, gave the results listed in tables 1, 2 and 3. It made clear that the proportion of cereal-growing areas sprayed with chemicals varies very much from region to region and according to the size of farms, weed-control methods being more commonly used on the larger farms.

The main factor influencing a decision to use chemical weed-control in these different regions does not seem to be density of weed population, but rather farmer's technical capabilities, coupled with the size of his farm ; as a rule the owner of a large farm is a better farmer than the man with less land. In regions devoted to the intensive production of cereals it has become as customary to use weed-control methods as, for instance, to spread

---

Note : All the doses in this report are indicated in kg/ha of active ingredient except for mixtures whose doses are indicated in l/ha of commercial product.

In the tables the underlined results are significantly different from those of the untreated control plot at the level of significance

P = 0.05

1 kg/ha = 0.89 lb/ac.

1 l/ha = 0.09 Imp. gal/ac.



nitrate fertilizers in spring. The average increase in yield obtained as a result of weed control is 4 qx/ha, which is about 13 % of the yield of the unsprayed plots. It is reasonable to think that in the regions not studied, which are technically less developed, the yield increase gained through weed-control is greater.

These facts underline the economic advantage of weed-control and show that French farmers in certain regions and especially on small farms must be encouraged to use weed-control techniques.

		Less than 20 ha	20 to 49.99 ha	50 to 99.99 ha	100 ha and more	Total
Weed-control	WW	48.8	52.1	58.3	74.9	56.5
	SB	25.4	45.0	62.5	81.8	53.0
No Weed-Control	WW	51.2	47.0	41.7	25.1	43.5
	SB	74.6	55.0	37.5	18.1	47.0
Total	WW	100.0	100.0	100.0	100.0	100.0
	SB	100.0	100.0	100.0	100.0	100.0

**Table I** - Repartition of the areas sown with winter wheat (WW) and spring barley (SB), with or without weed-control, according to the category of tilled areas in the farm.

	W H E A T				B A R L E Y			
	No Weed Control	Weed Control	Total	Average yield qx/ha	No Weed Control	Weed Control	Total	Average yield qx/ha
North-Bassin Paris.	10.2	89.8	100	37.5	19.2	80.8	100	36.0
Centre	43.7	56.3	100	25.8	40.8	59.2	100	30.4
South-West	76.5	23.5	100	29.4	81.0	19.0	100	24.8

**Table 2** - Repartition by regions of areas (in %) sown with wheat and barley, with or without weed-control, and average yield.

#### WEED-CONTROL PRODUCTS IN USE

Table 3 shows the cereal-growing areas treated with the principal groups of herbicides.

**Phenols**, mostly DNOC, are little used in spite of their toxicity to certain weeds which are resistant to auxin regulators. This is due to the difficulty of application, particularly on wheat, at the end of winter.

The products containing 2,4-D and MCPA are, without question, the most frequently used, MCPA being used more on spring barley than on wheat. Mixtures of 2,4-D and MCPA are used for practical reasons, though there seems insufficient technical justification for their extensive use. The column headed "Miscellaneous" includes more recent products containing MCPB, mecoprop, dichlorprop, triallate, barban... We must point out that a licence for sale has been granted this year in France for products containing, on the one hand, mixtures of dinoseb and 2,4-D or MCPA, on the other hand, a

mixture of dicamba and MCPA. Herbicide manufacturers are putting on to the market products containing mixtures of active ingredients. The main ones now sold in France are :

2,4-D + MCPA	2,4-D + dichlorprop
2,4-D + mecoprop	MCPA + dichlorprop
MCPA + mecoprop	MCPA + dicamba
2,4-D + MCPA + mecoprop	Dinoseb + MCPA

Active ingredient		2,4-D	MCPA	2,4-D + MCPA	Phenols	Miscellaneous	No treatment	Total
North-Bassin Parisien	WW	22.7	31.9	16.7	8.6	9.9	10.2	100.0
	SB	7.6	43.8	16.1	7.5	5.8	19.2	100.0
Centre	WW	11.5	11.6	12.2	4.9	16.1	43.7	100.0
	SB	2.8	34.8	10.7	2.3	8.6	40.8	100.0
South-West	WW	17.8	0.7	3.7	-	1.3	76.5	100.0
	SB	10.9	4.9	2.2	-	1.0	81.0	100.0

Table 3 - Repartition in % of areas sown with winter wheat (WW) and spring barley (SB), according to the active ingredient of weed-killer.

#### MOST IMPORTANT WEEDS FOUND IN CEREAL CROPS AT PRESENT

##### Broad-leaved weeds

At the moment the most important broad-leaved weeds for the farmer are:

- Polygonum (*Polygonum aviculare* L., *P. convolvulus* L., *P. persicaria* L.) and wild chamomile (*Matricaria* sp.) both destroyed by products containing phenols, dichlorprop or dicamba.

- Heart's ease (*Viola tricolor* L.) is now frequent in cereals. Because of its small size it does not seem to interfere with crops.

- Cleavers (*Galium aparine* L.), killed by mecoprop and dichlorprop, is found mostly in Western France.

- Chickweed (*Stellaria media* L.) is common in cereals, but does not seem very harmful. Certain weeds are located in particular regions, where their control is not easy:

- Bifora radiens M. Bieb. is frequently found in Limagne.

- Lathyrus tuberosus L., up to now rare, seems to be spreading.

##### Gramineous weeds

The control of gramineous weeds in cereals presents difficult problems to the farmer. The most important of them is blackgrass (*Alopecurus myosuroides* Huds.). About 20 % of fields growing winter wheat are overgrown with this weed. It is a little rarer in winter barley and seldom seen in spring barley. Its density can be greater than 500 plants per m<sup>2</sup> in which case its growth may compete with the cereal as far as choking it. Loose silky bent (*Agrostis spica-venti* L.) is rather frequently seen in winter wheat too. Wild oat (*Avena fatua* L.) is sometimes abundant in the crops of spring barley in the Northern half of France. It is not so widespread in winter wheat crops. However it causes a problem in the West of France and in some parts of the Bassin Parisien. In the South of France, wheat crops are sometimes heavily infested by *Avena ludoviciana* Dur.. Finally in the West, rye-grass (*Lolium perenne* L.) sometimes grows in profusion in winter cereals.

## EXPERIMENTAL RESULTS OBTAINED IN 1964

### Products for the control of broad-leaved weeds

The experiment carried out in 1964 mainly aimed at assessing the action of recent products on the yield of cereals. Therefore most of the experiments have been on crops containing no or very few weeds. The products studied are:

#### 1° Dicamba and MCPA mixture

Commercial product containing 33 g/l of dicamba and 360 g/l of MCPA. The required dose is 4 l/ha of the commercial product. The action of that mixture on the yield of cereals is compared to the action of MCPA at a dosage rate equal to the rate of MCPA contained in the mixture.

#### 2° Mixture of dinoseb and 2,4-D or MCPA

The two products under review are :

- the "D" product containing 100 g/l of dinoseb and 125 g/l of MCPA. The required dose is 7.5 l/ha of the commercial product.
- the "PS" product containing 150 g/l of dinoseb and 180 g/l of 2,4-D. The required dose of this product is 4 l/ha of the commercial product.

#### 3° Ioxynil (4 hydroxy-3,5 diiodobenzonitrile)

This product is examined in simple trials in order to check its activity on certain weeds and, for a first approximation, its action on cereals.

### Conditions in which the trials have been undertaken

These are indicated in table 4. All the trials are carried out in a design having 6 replications. Usually the lowest dose used corresponds to the normal dose recommended by the manufacturer. Ioxynil is applied at the following doses : 0.250 - 0.500 - 0.750 and 1 kg/ha of active ingredient.

### Results

These are given in table 5.

### Discussion

The MCPA and dicamba mixture proves to be relatively safe on cereals, particularly barley, since double the normal dose does not usually cause any decrease in the yield. However we must notice that in most cases the treatments have caused deformities in the ears (they were shortened and widened), but these deformities do not appear to influence the yield. The toxicity of this mixture seems to be somewhat greater than that of MCPA on its own.

The mixture of dinoseb with 2,4-D or MCPA is rarely so well tolerated as 2,4-D or MCPA used in equivalent doses, in particular on spring barley. These products must be used carefully on winter wheat. We consider their use on spring barley as dangerous.

Ioxynil is an effective weed-killer at such doses as 0.300 to 0.500 kg/ha. No effect on the cereal development was observed even at the dose of 1 kg/ha. Thus ioxynil seems to be very selective in regard to cereals.

### Products for the control of gramineous weeds

#### 1° Wild oats

At present triallate and barban are satisfactorily used in France to control wild oats in crops of spring barley. For winter wheat, no product is satisfactory at present. Barban has sometimes been very toxic to wheat under certain conditions. Triallate is not quite selective enough to be used in wheat.

N°	P L A C E S	Sp	Variety	Stage of treatment	Temperature (in ° C)	Doses (l/ha of formulated product)			Reference product (dose in kg/ha A.I.)	
						a	b	c		
<u>Mixture dicamba + MCPA</u>										
Q1	ST AUBIN (S & O)	WW	Moisson	End of tillering	12	4	8	12	(MCPA = 1.44-2.88-4.32)	
Q2	TOURY (E & L)	"	Poncheau	Beginning of the jointing stage	15	"	"	"	"	"
Q3	ST DENIS S/LOIRE (L & C)	"	Cappelle	End of tillering	14	"	"	"	"	"
Q4	ST LEONARD (L & C)	"	Cappelle	Beginning of the jointing stage	18	"	"	"	"	"
Q5	DROIZY (Aisne)	"	Champlein	End of tillering	19	"	"	"	"	"
Q6	VILLERMAIN (L & C)	SB	Rika	End of tillering	21	"	"	"	"	"
Q7	DADONVILLE (Loiret)	"	Aurore	Beginning of the jointing stage	14	"	"	"	"	"
Q8	BRIERES les SCHELLES (S & O)	"	Cérès	End of tillering	15	"	"	"	"	"
Q9	FRANCAY (L & C)	"	Piroline	End of tillering	24	"	"	"	"	"
				Beginning of the jointing stage	22	"	"	"	"	"
<u>Mixture DNBP + MCPA ("D") and DNBP + 2,4-D ("PS")</u>						<u>(l/ha of formulated product)</u>				
M1	VILLERAS (S & O)	WW	Capitole	End of tillering	15	D 9	18	27	2,4-D = 1-2-3	
M2	ETAMPES (S & O)	"	Cappelle	End of tillering	15	PS 6	12	18	"	
M3	NOYANT (Aisne)	"	Cappelle	End of tillering	18	D 9	18	27	"	
M4	VALENCE D'AGEN (T & C)	"	Et.Choisy	End of tillering	18	PS 6	12	18	"	
M5	VALENCE D'AGEN (T & C)	"	Et.Choisy	Full tillering	17	D 8	16	24	"	
M6	BORDEAUX en GATINAIS (Loiret)	SB	Aurore	End of tillering	24	PS 5.25	10.5	15.75	"	
M7	ALLONNES (E & L)	"	Rika	End of tillering	16	D 9	18	27	"	
M8	AUTAINVILLE (L & C)	"	Rika	End of tillering	20	PS 6	12	18	MCPA = 1 - 2 - 3	
				End of tillering	16	D 4	8	12	"	
				End of tillering	20	PS 7	14	21	"	
				End of tillering	20	D 8.6	17.2	25.8	"	
				End of tillering	20	PS 4.75	9.5	14.25	"	

Table 4 - Conditions in which the trials have been carried out.

Mixture dicamba + MCPA

Doses		MCPA (kg/ha A.I.)			Mixture (l/ha of C.P.)			C.V	Yield	l.s.d.	
N <sup>o</sup>	Sp	0	1.44	2.88	4.32	4	8	12	%	C qx/ha	P=0.05
Q1	WW	100	105.7	107.3	104.2	103.6	102.2	91.8	5.6	50.3	6.6
	"	-	-	-	-	100.4	97.7	88.2			
Q2	"	100	98.2	100.6	95.9	102.2	96.0	96.3	6.9	37.9	NS
Q3	"	100	98.5	93.0	89.8	97.2	89.8	90.5	6.7	32.0	7.5
Q4	"	100	103.2	109.2	104.4	96.7	104.7	107.9	7.9	42.8	NS
Q5	"	100	98.1	95.7	94.4	91.7	89.7	90.4	4.4	50.4	4.9
Q6	SB	100	100.0	101.0	105.5	103.4	105.0	98.0	4.5	47.5	NS
Q7	"	100	106.0	100.4	107.5	107.3	101.3	95.6	7.5	32.9	NS
Q8	"	100	100.3	97.0	93.8	95.2	89.8	80.0	11.1	40.8	12.3
Q9	"	100	97.4	98.4	99.7	95.8	93.0	90.5	8.1	24.2	NS

Mixture dinoseb + MCPA ("D") and DNBP + 2,4-D ("PS")

N <sup>o</sup>	Sp	2,4-D or MCPA (kg/ha A.I.)				" D "			" PS "			C.V %	Yield C qx/ha	l.s.d P = 0.05
		0	1	2	3	a	b	c	a	b	c			
M1	WW	100	98.2	97.2	93.5	97.5	92.8	89.6	95.3	95.6	92.7	4.2	70.8	4.7
M2	(1)	100	100.1	98.8	99.4	102.3	102.0	104.5	102.8	103.0	101.9	6.1	42.1	NS
M3	"	100	97.4	97.1	95.0	96.3	91.4	86.2	102.1	98.7	94.8	6.0	60.0	6.7
M4	"	100	93.5	92.2	78.8	98.6	84.8	79.4	93.5	87.2	80.7	8.0	33.6	8.3
M5	"	100	96.1	95.4	92.0	92.6	36.4	80.0	-	-	-	5.2	45.9	5.6
M6	SB	100	100.9	97.1	99.9	93.4	92.5	88.7	100.1	97.5	97.5	6.5	48.0	7.4
M7	"	100	99.9	101.0	95.7	97.7	90.1	89.0				5.2	49.4	5.9
M8	"	100	100.6	97.8	93.8	88.5	81.1	79.0	95.4	91.7	86.5	8.9	41.9	7.5

(1) Rain during the 48 hours following the spraying.

Table 5 - Action of different mixtures on the yield of winter wheat and spring barley (in % of the untreated plots).

2° Blackgrass

No product is allowed to be used in France. In winter wheat, during the last few years, we have shown that simazine destroys that weed before its full tillering, from the minimum dose of 0.3 kg/ha. Winter wheat tolerates that dose very well, except under certain conditions of soil and climate where it has much more susceptibility. Severe frosts in particular seem to increase the susceptibility of wheat to simazine. It seems to be much less tolerated by winter barley than by wheat. The dose of 0.450 kg/ha causes a decrease of 15 % in the yield, rarely seen in wheat.

- Other triazines were experimented, comparatively to simazine, they are:
- prometryne (4,6 bis-isopropylamino-2 methylthio-s-triazine)
  - triazine G 36 393 (2 methylthio-4 isopropylamino-6 (2 methoxypropylamino)-s-triazine)
  - triazine GS 12 344 (2 methylthio-4 ethylamino-6 (2 methoxypropylamino)-s-triazine)

PLACES	Variety	Stage of Spraying	Doses (kg/ha)				C.V %	Yield C qx/ha	l.s.d. % C
			0	0.3	0.45	0.6			
Auvernaux (S & O)	Manon	3.5 1	100	90.8	84.3	78.8	9.9	37.9	14.1
Valpuseaux (S & O)	Atlas	4.5 1	100	95.1	86.5	88.0	6.9	37.5	7.9
Prenouvellon (L & C)	Bordia	Tillering	100	93.2	86.3	79.7	4.9	45.4	5.5

Table 6 - Action of simazine on the yield of winter barley (in % of the untreated plots).

In trials performed with these products over the last three years no definite superiority has been found of any one of them over simazine. Moreover, results seem to vary irregularly from place to place and from year to year (Table 7). Prometryne is successful at a dose of 0.75 to 1 kg/ha, applied at any stage of growth, from emergence to the fully tillered state, these results being better than those of the past two years. Triazine GS 12 344, at doses of 1 to 1.5 kg/ha, which appear to be the best doses, has an irregular effect on blackgrass and wheat. Triazine G 36 393 seems to be more selective. The most regular result seems to be obtained when the treatments are applied during tillering, at the dose of 1 kg/ha. We do not consider any of these three triazines to be regularly more effective and more selective than simazine.

PLACES	BLARU (S & O)		TIGERY (S & O)		ST PELLERIN (E & L)				CHAT. EN DUNOIS (E & L)				VILLERAS (S & O)		ST MARTIN (E & L)	
	31/1		27/11		2/1		22/2		23/1		9/4		26/3		12/3	
Date of spraying	B	W	B	W	B	W	B	W	B	W	B	W	B	W	B	W
Stage of growth	1.5	2.5	2	2.5	1.5	2.5	3	2	2	2.5	1-6	2-3	til	1-2	1-4	3-4
	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1
Prometryne																
0.75 kg AI	90	0	98	0	85	0	85	0	85	0	80	0	70	5	60	0
1 - -	98	5	100	10	95	0	98	5	98	0	98	10	95	15	85	0
1.25 - -	98	10	100	15	98	0	98	10	100	5	100	20	95	20	95	0
G 36 393																
0.75 kg AI	85	0	80	0	60	0	95	0	65	0	85	0	45	0	30	0
1 - -	90	5	90	15	75	0	98	0	85	10	90	0	65	5	80	0
1.25 - -	-	-	95	20	-	-	-	-	-	-	99	5	85	15	90	5
GS 12 344																
0.75 kg AI	45	0	50	0	50	0	60	0	85	5	85	0	0	0	50	0
1 - -	85	0	70	0	60	0	70	0	90	5	98	0	35	0	50	0
1.25 - -	90	0	80	5	75	0	75	0	98	10	99	5	60	0	75	0
1.50 - -	-	-	95	25	-	-	-	-	-	-	100	15	70	0	85	0

0 = no effect

100 = complete destruction

Table 7 - Comparative action of triazines on blackgrass (B) and winter wheat (W).

. triallate used pre-emergence causes a satisfactory level of blackgrass kill at the minimum dose of 1.2 kg/ha. The yield depression produced on

wheat at a dose of 1.2 is only slight, but it gains importance at the dose of 1.6 kg/ha (table 8). Triallate is not very selective in regard to wheat. On the contrary, in winter barley, triallate can be effectively used to destroy blackgrass at the doses of 1.2 - 1.4 kg/ha. At the dose of 2.5 kg/ha, triallate has no action on winter barley (table 8). Triallate is therefore very selective.

P L A C E S	Varieties	Doses in kg/ha of A.I.					C.V %	Yield C qx/ha	l.s. d.
		0	1.2	1.4	1.6	1.8			
<u>Winter Wheat</u>									
Flexanville (S & O)	Cappelle	100	102.2	95.0	91.1	91.8	6.5	52.7	7.6
Ch. en Serval (Oise)(1)	Champlain	100	87.8	90.2	85.3	79.6	11.0	39.9	11.9
Tivernon (Loiret)	Cappelle	100	96.1	100.0	94.3	98.9	5.2	45.0	NS
Plessis l'Ech. (L & C)	Cappelle	100	92.5	85.5	82.5	80.6	7.8	46.8	8.4
<u>Winter Barley</u>									
Rahart (L & C)	Arès	100	98.9	111.4	106.6	106.1	9.6	42.3	NS
Sargé (L & C)	Bordia	100	105.3	103.8	108.3	103.5	5.5	26.3	NS
Corroy (Marne)	Atlas	100	103.3	98.8	101.9	104.4	5.6	32.0	NS
Vallée aux Bleds(Aisne)	Manon	100	100.0	92.2	100.9	100.9	6.1	32.1	NS

(I) Very sandy soil.

Table 8 - Action of triallate on the yield of winter wheat and winter barley (1964).

. dichlobenil used during wheat tillering, at doses of 2 to 3 kg/ha, generally causes a satisfactory level of blackgrass kill and has little effect on the cereal, unless the treatment be applied on a very moist soil, or immediately before rainfall. In this case, unfortunately quite frequent in winter, dichlobenil is highly toxic applied to wheat.

. barban has a very irregular action on blackgrass and wheat, even at the low dosage rates of 0.250 and 0.375 kg/ha. We cannot trace a connection between the action of barban and the outside conditions during the spraying, nor with the date of spraying. This product is often very badly tolerated by winter barley, whatever its stage of growth during the spraying may be. We noticed important differences in the resistance to barban of different varieties of winter barley. Table 9 shows an important diminution of yield from the dose of 0.480kg/ha.

P L A C E S	Variety	Stage of growth	Date	Doses (kg/ha)				C.V %	Yield C qx/ha	l.s. d.
				0	0.48	0.72	0.96			
Bonvilliers (S & O)	Grignon	3.5 1	22/11	100	65.7	58.2	52.4	14.8	41.2	12.6
Rahart (L & C)	Arès	4 1	21/11	100	86.4	76.0	68.1	10.5	34.4	10.7

Table 9 - Action of barban on the yield of winter barley (1964) (in % of the untreated plots).

. buturon (N'-4-chlorophenyl)-N-isobutynyl-N methylurea) applied when

blackgrass has 2 or 3 leaves gives a satisfactory destruction at the dose of 2 kg/ha, but is toxic to wheat and winter barley as well (table 10). Treatments applied at a later stage may be more selective.

PLACES	CHEVANNES (Yonne)		ST PELLERIN (E & L)		VILLEFARGEAU (Yonne)		CLEVILLIERS (E & L)		
Date of spraying	13/2/64		21/2/64		14/2/64		7/2/64		
	B	WB	B	W	B	W	B	W	
Stage of spraying	2-2.5	3 1	3 1	4 1	0.5 1	1 1	3 1	3.5 1	
Doses in kg/ha A.I.	1	80	0	65	0	70	0	85	0
	1.5	90	15	90	5	90	15	-	-
	2	98	30	100	75	100	25	95	10
	3	100	75	100	90	100	40	100	15

0 = no action

100 = complete destruction

Table 10 - Action of buturon on blackgrass (B), wheat (W) and winter barley (WB) 1964.

### 3° Agrostis spica-venti

The following products seem to be effective when it is treated at the 3 to 5 leaf stage.

- simazine : 0.300 kg/ha
- prometryne : 1 kg/ha
- G 36 393 : 1 kg/ha
- GS 12 344 : 1.5 kg/ha
- dichlobenil : 2 kg/ha

The susceptibility of *Agrostis spica-venti* to these products seems to be almost the same as that of blackgrass. However these results have still to be confirmed.

### CONCLUSION

For the control of broad-leaved weeds, mixtures of different active ingredients are usually effective on cereal crops. Ioxynil seems to be highly selective in regard to cereals.

The problem of the control of wild oats in spring barley seems to be solved by the use of triallate and barban ; triallate also controls blackgrass satisfactorily in winter barley.

On the other hand, a satisfactory solution has not yet been found to the problem of controlling wild oats and more particularly blackgrass in winter wheat.

### Acknowledgments

The cited trials have been performed in collaboration with M. BALLACEY, A. de BUTLER, O. BENARD, from the I.T.C.F., whom we thank very much.



### References

- BARRALIS, G. (1961) Distribution et état d'infestation des graminées adventices en France.  
C.R. 1re Conférence du COLUMA - Paris, 263-270
- DE GOURNAY, X. (1963) Nouveaux essais de lutte contre le vulpin des champs dans les cultures de blé d'hiver.  
C.R. 2e Conférence du COLUMA - Paris, 84-100
- DE GOURNAY, X. - FAIVRE-DUPAIGRE, R. - ROGNON, J. (1963) Essais de destruction de la folle-avoine dans les cultures de blé.  
C.R. 2e Conférence du COLUMA - Paris, 123-131
- FAIVRE-DUPAIGRE, R. - ROGNON, J. - BALLACEY, M. (1963) Essais de destruction du vulpin dans les céréales.  
C.R. 2e Conférence du COLUMA - Paris, 101-122
- MOTHE, A. (1963) Enquête sur les productions de blé et d'orge en 1963.  
Etudes statistiques 4, 309-327
- ROGNON, J. - FAIVRE-DUPAIGRE, R. (1963) Toxicité comparée vis-à-vis des céréales de divers herbicides contre dicotylédones.  
C.R. 2e Conférence du COLUMA - Paris, 199-215
- ROGNON, J. - FAIVRE-DUPAIGRE, R. - BALLACEY, M. (1963) Destruction de la folle-avoine dans les cultures d'orge de printemps.  
C.R. 2e Conférence du COLUMA - Paris, 132-144
- PFEIFFER, R.K. - HARPUR, R.L. - PHILIPS, J.D. (1962) The place of mixtures of MCPA with 2-methoxy-3,6 dichlorobenzoïc acid in selective weed control in cereals.  
6 th British Weed Control Conference - Vol. 1, 215-217

GRAMINEOUS WEEDS IN CEREALS IN WEST GERMANY  
AND THEIR CONTROL

B. Rademacher, W. Koch and B. Würzer

Institut für Pflanzenschutz; Stuttgart-Hohenheim

INTRODUCTION

The importance of three gramineous weeds (*Apera spica-venti*, *Alopecurus myosuroides* and *Avena fatua*) in cereals in West Germany has greatly increased during the last few years. The effort to find suitable herbicides to control these three species has accordingly been very much intensified.

The use of calcium cyanamide (2.5 - 3 kg/ha) for the control of *Apera spica-venti* and *Alopecurus myosuroides* in winter wheat has been accepted by German Agriculture for a number of years. Although the effectiveness of this treatment is much influenced by weather conditions, satisfactory results on *Apera spica-venti* have nearly always been obtained following correct application. Results on blackgrass however have been less satisfactory and much more subject to variation, though control of both species in 1964 was exceptionally good.

Since it proved possible to control *Apera spica-venti* fairly satisfactorily with calcium cyanamide, research effort in winter cereals has concentrated on the control of blackgrass. Simazine (0.4 kg/ha a.i.) has found conditional acceptance in agriculture, but although the blackgrass control is satisfactory, despite the dependence of this treatment on weather conditions, the same cannot be said of cereal tolerance. For this reason simazine can only be recommended in cases of very severe blackgrass infestation. In 1964 however simazine gave very good results as regards both cereal tolerance and blackgrass control, and this was found both in farming practice and experimentally. Triallate (1.2 litre/ha a.i.) did not always give satisfactory blackgrass control in winter cereals, partly due to the relative sensitivity of winter wheat to this chemical.

Recently, experiments have been carried out on a combination of simazine and ioxynil as well as on some urea derivatives for the control of dicotyledonous and monocotyledonous weeds in winter wheat. This research report deals with the results obtained with these herbicides, which, it must be emphasised, are still in the experimental stage.

By and large, wild oats in Germany only occur in spring cereals since they either do not germinate in the autumn, or if they do are normally killed in the winter. Triallate and barban are already used on a large scale in agricultural practice and on the whole with very satisfactory results. In contrast to the previous years, however, results with triallate in 1964 have been less satisfactory in some localities. This can be ascribed to unusual weather conditions during the past spring. The beginning of the growing period was much delayed in the spring, and,

in most cases, the application of triallate was followed by very dry and warm conditions, which proved very favourable for the development of wild oats and unfavourable for the action of triallate.

Exactly the opposite was the case for barban. Since nearly all the wild oats germinated in the one flush, most of the plants reached the correct stage for spraying at the same time, and this resulted in exceptionally good control. No new problems were encountered in the use of either of the two chemicals and little experimental work on the control of wild oats in spring cereals was necessary in 1964.

#### METHOD AND MATERIALS

##### Observations on the increase of *Avena fatua* on arable land

On two adjoining fields with identical past cropping history an experiment was carried out to study the degree of wild oat infestation occurring with a normal crop rotation as compared with the infestation resulting from continuous cropping with spring cereals. On the one field the rotation was:-

1959 and 1964 - spring barley

1960 and 1962 - potatoes

1961 and 1963 - winter wheat.

On the other field spring cereals have been grown year after year since 1959. On each field the germinated wild oat plants were counted on twenty sample areas of 0.5 sq. metres. This assessment was made in 1959 and 1964 on the field which had a normal rotation and every year except 1961 on the field with continuous cereal production.

##### Control of annual grass weeds

Three field experiments in winter wheat on the control of *Apera spica-venti* and more particularly for the control of blackgrass were carried out on the Filderebene near Stuttgart (south-west Germany, approximately 450 metres above sea-level). The design was that of randomised blocks with four to five replications. Plot size was 15 sq. metres. The chemicals were applied with a 2-litre hand-syringe. Spray volume was 600 litres per hectare. In all experiments the winter wheat was planted on October 18th, 1963. Crop emergence took place early in November.

Crop damage due to chemical was assessed by scoring and in two experiments by actual yield assessment. The degree of grass control was also assessed by using a scoring method, as well as by counting the number of grass plants on plots of 3 x 0.5 sq. metres. The scoring system proposed by the study group on experimental techniques of the European Weed Research Council was used to score the herbicidal effects. This system works on the following non-linear scheme between 1 and 9:

Weed ... 1 = 0%, 2 = 2.5%, 3 = 5%, 4 = 10%, 5 = 15%, 6 = 25%, 7 = 35%,  
8 = 67.5%, 9 = 100% groundcover.

Crop ... 1 = no damage, 9 = complete kill.

### Chemicals

The following chemicals were used:

simazine.....50% wettable powder  
ioxynil (3,5-diiodo-4-hydroxybenzoxitrile)  
H 95-1 (N-p-chlorophenyl-N'-methyl-N'-isobutylurea).....50% active ingredient  
H 149, a mixture of:  
cyclooctyldimethylurea (OMU)+ N-p-chlorophenyl-N'-methyl-N'isobutylurea  
at a ratio of 1:1.....total active ingredient 50%  
monolinuron (N'-(4-chlorophenyl)-N-methoxy-N-methylurea) )

## RESULTS

### Observations on the increase of *Avena fatua* on arable land

As shown in table 1, continuous spring cereal production and the degree of wild oat infestation increased steadily over the first three years and this was followed by a dramatic increase in 1963, a year which was particularly favourable for the propagation of wild oats. In 1964 the degree of wild oat infestation decreased again to a level of about 1,000 plants per 10 sq. metres. The wild oat infestation in the normal rotation, comparing 1959 with 1964, shows no significant difference.

Table 1  
Number of wild oat plants on 10 sq. metres following a normal rotation  
as compared with continuous spring cereal cropping

	1959	1960	1962	1963	1964
Normal rotation	70	-	-	-	79
Continuous spring cereal growing	62	106	246	2208	1030

## Control of annual grasses

### Experiment 1

Application of the urea derivatives H 95-1 and H 149 as well as simazine for the control of *Alopecurus myosuroides* and *Apera spica-venti* in winter wheat (variety Heines VII). Development stage of the wheat at the time of application on the 10th of April, 1964, was E and for blackgrass and *Apera spica-venti* D-E-F. (See footnote)

Table 2  
Effect of simazine and of two urea derivatives  
on *Alopecurus myosuroides*, *Apera spica-venti* and winter wheat

Chemical and dosage (kg/ha product)	Effect on				
	<i>Alopecurus myosuroides</i>		<i>Apera spica-venti</i>	winter wheat	
	Score value	Ears/sq.m.	Score value	Score value	
	7.5.64	3.6.64	7.5.64	7.5.64	
		Abs. rel.			
untreated	9	362	100	9	
simazine* 0.75	4	60	16	3	
H 95-1* 3	4	74	20	6	
H 149* 3	3	81	22	3	

\* 50% active ingredient

All materials at the higher dosage rates gave a satisfactory control of blackgrass (*Alopecurus myosuroides*). Plants in the middle of tillering, however, were not adequately controlled. The effect against *Apera spica-venti* was very good. All materials caused some crop damage, and this was relatively severe in the case of H 95-1. Damage by the urea derivatives appeared in the form of chlorosis of the leaf tips and general thinning of the crop. It was particularly noticeable that on the 9th July, when the controls were already turning colour, treated plants were still dark green.

Development stage of cereals and grasses on the lines proposed by the Methods Group of the European Weed Research Council. A = germination, B = cotyledon stage, C = two-leaf stage, D = three-leaf stage, E = beginning of tillering, F = middle of tillering.

### Experiment 2

Application of the substituted urea derivative H 95-1 for the control of *Alopecurus myosuroides* in winter wheat (variety Pfeuffers Schernauer)

Table 3  
Effect of H 95/1 on *Alopecurus myosuroides* and winter wheat

Chemical and rate of application (product kg/ha)	Date of application	Development stage		Effect on					
				<i>Alopecurus m.</i>			winter wheat		
				Score 7.5	Ears/sq.m.		Score 7.5	Yield 100kg/ha	
					Abs.	rel.		Abs.	rel.
untreated	-	-	-	9	336	100	1	54.9	100
H 95-1 2.5	30/11/63	B-C	B-C	2	40	12	1	59.3	108
3.0	"	"	"	2	20	6	1.5	57.2	104
2.5	20/2/64	C	C	4	54	16	2.5	54.3	99
3.0	"	"	"	3	52	16	3.5	55.3	101
2.5	8/4/64	D(4)	D-E-F	2.5	56	16	1	56.6	103
3.0	"	"	"	2.5	65	19	2	57.5	105
simazine 0.75	"	"	"	2	38	11	1	59.5	108

Autumn application of H 95-1 led to complete kill of blackgrass. Thus the results were equivalent to simazine applied in the spring. Winter or spring application of H 95-1 was somewhat inferior to autumn application.

Significant damage to wheat in the form of necrosis of leaf tips and of a general thinning of the crop occurred only when H 95-1 was applied under frost conditions. A very slight effect was also noticed when H 95-1 was used at the rate of 3 kg/ha in the autumn.

### Experiment 3

Application of the urea derivatives, Monolinuron and Hoe 2831 A as well as Sp 6403 (simazine + ioxynil) and simazine alone for the control of *Alopecurus myosuroides* in winter wheat (variety Pfeuffers Schernauer).

Development stage of the crop of winter wheat at the time of application on the 8th of April, 1964, was D(4)-E, of the blackgrass D-E-F.

Table 4  
The effect of monlinuron, Hoe 2831 A, simazine and Sp. 6403  
on Alopecurus myosuroides and winter wheat

chemical and dosage (product kg/ha)	Effect on					
	Alopecurus myosuroides			winter wheat		
	Score value 7.5.64	Ears/sq. m.		Score value 7.5.64	Yield 100 kg/ha	
		Abs.	rel.		Abs.	rel.
untreated	9	224	100	1	50.0	100
simazine 0.75	2	33	15	1	53.9	107
monolinuron* 1.0	3.5	61	27	3	52.0	104
1.4	3	57	25	3.5	49.2	98
Hoe 2831 A 1.0	4	79	35	2.5	47.9	96
1.4	3	51	23	3	48.3	96
Sp. 6403 2.0	2	22	10	1	55.2	110

\* 80% active ingredient

Simazine and Sp. 6403 gave better blackgrass control than Monolinuron and Hoe 2831 A. Monlinuron and Hoe 2831 A at all rates of application caused slight crop damage, consisting of temporary leaf chlorosis, increasing necrosis of older leaves and some thinning of the crop. It was particularly interesting that when untreated controls were beginning to ripen, plots with the urea derivatives showed a dark green colour, an observation which corresponds with that mentioned under Experiment 2.

#### DISCUSSION

##### Observations on the increase of Avena fatua on arable land

Our experiment, comparing the degree of wild oat infestation over a normal six-year rotation with that of a six-year monoculture of cereals shows that by an appropriate rotation the incident of wild oat can be kept down to an acceptable level, while the monoculture of spring cereals led to a dramatic increase of wild oat. The fact that the figures for the wild oat infestation in 1963 were particularly high agrees with the general observation that 1963 was an exceptionally bad wild oat year in south-west Germany.

##### Control of annual grasses

The effect of simazine in 1964 was very good as regards both the control of blackgrass and of Apera spica-venti, as well as cereal tolerance. A weed reduction of 80% to 90% was obtained in all experiments, while slight damage symptoms could only be observed in one case. Yield increases

occurred in both yield experiments. As expected, the effect on *Apera spica-venti* was also good.

The combination of simazine with ioxynil (Sp 6403) was particularly interesting in view of the wide spectrum of weeds controlled and very good crop tolerance.

While in the case of simazine the optimum time of application has been determined during the last few years, in the case of substituted urea derivatives information has been obtained only in the 1964 season, and has to be confirmed next year. In the case of the BASF herbicides, H 95-1 and H 149, it was found that application during the winter was too dangerous, because of the damaging effect of frost. Such a winter application was furthermore also less effective against blackgrass than autumn or spring application. Monolinuron and the combination of monolinuron and linuron produced damage even after spring application. This damage appeared in the form of slight chlorosis of the leaf tips and a thinning of the crop. A delay in maturity was particularly significant with these herbicides. The degree of blackgrass control with the urea derivatives was in general satisfactory to good, and in the case of H 95-1 applied in the autumn equal to the degree of control obtained with simazine. As a general rule, spring application gave inferior control compared with autumn application. This can be ascribed to the fact that in the past year approximately 80% of blackgrass plants had germinated in the autumn, and had reached the tillering stage in early spring.

The experiments so far carried out with the above mentioned urea derivatives for the control of annual grasses in cereals showed interesting possibilities. Much more detailed investigations are however necessary before one or the other compound could be handed over for practical application.



THE USE OF DICHLOBENIL IN THE CONTROL OF  
ALOPECURUS MYOSUROIDES IN WINTERWHEAT.

J.C. Leroy  
Soci t  "La Quinol ine", Paris

Synopsis. On the basis of work carried out with dichlobenil in rice, tests were done in France for the control of blackgrass (*Alopecurus myosuroides*) in winterwheat. From very extensive tests, under a broad range of conditions, conclusions were drawn with regard to the selective use of dichlobenil in winterwheat. On the basis of these conclusions a big number of large scale tests under practical conditions were carried out resulting generally in a good control of blackgrass and a higher yield of grain. It can be concluded that dichlobenil is a promising agent for control of blackgrass in winterwheat.

INTRODUCTION

The discovery of the herbicidal activity of 2,6-dichlorobenzonitril a few years ago (Barnsley 1960, Koopman and Daams 1960, Barnsley and Rosher 1961, Daams and Barnsley 1961, Stovell 1962) has led to an intensive investigation into its biological properties. Among the large number of weeds, which proved to be susceptible to relatively low rates of application, one of the most important was *Echinochloa crus galli*.

Further to the work which had been done in the laboratory by J. Daams, the tests carried out in the Camargue in 1960 and 1961 showed the good selectivity of dichlobenil in firmly rooted rice, and, on the other hand, the efficacy of this herbicide on *Echinochloa crus galli* in the two- or three-leaf stage. Dichlobenil is now used in practice for the control of this weed in replanted rice; the rice is treated about 15 days after replanting.

These first observations gave us sufficient reason for studying the herbicidal effect of dichlobenil on *Avena fatua* in the North of France during the 1961/1962 season. One knows that this species of *Avena* occurs in this area in the spring in winterwheat.

The experience which had been gained in the rice fields justified the expectation that the herbicide would have a good effect on *Avena fatua* in the two- or three-leaf stages in the spring-treatments, and a good selectivity for winterwheat which has by that time sufficiently taken root.

Contrary to our expectations, the efficacy on *Avena fatua* was too irregular during these tests to be of economic interest, but the total of observations gave us the idea that dichlobenil might be effective on blackgrass (*Alopecurus myosuroides*) in post-emergence treatments.

## METHODS AND MATERIALS

Further to the already obtained results, systematic tests were carried out in 1962/1963 and 1963/1964 to find out the susceptible stages of winterwheat to dichlobenil and the susceptible stages of blackgrass.

In 1962/1963 forty-eight testing sites were chosen throughout France. At every site there were 5 different dates of treatment and 4 different dosages and a blank. Every test was carried out in three replications. As a result, about 3000 plots were under observation. The treatments have been carried out at well-defined growthstages of the wheat, from the one-leaf stage to the end of the tillering, without regard to the other conditions (weather, humidity of the soil etc.) at that moment. Accurate observations of these conditions enabled us to establish the influence of these conditions on the effect of the weed-killer.

In these trials the blackgrass was treated at all its different growthstages as a consequence of the timing of treatments. Many data on the difference in the biology of the blackgrass and the wheat have been gathered whereas at the same time the effect of dichlobenil on the blackgrass was established by counting the ears in each plot where the efficacy was good, whereas the selectivity on the wheat was evaluated with special attention to the relationship between the development and the climatic conditions.

On the basis of the data obtained, tests on a semi-practical scale have been done in the 1963/1964 season. The number of trials was 75. Plots were 100 meters long and had the width of the spraying boom. There were always 3 replications. In each trial treatment was executed on the best possible time with regard to meteorological conditions. Two different treatments, about 3 and 4 kg dichlobenil per ha, and an untreated control were included in each trial. Spraying took place at the 3 to 4 tiller-stage.

The plots were harvested with a combine and the yield of the whole plot was weighed. In each band the blackgrass has been counted on 3 to 5 plots of one square meter.

After having completed a spraying run the quantity of spraying liquid eventually left over was measured and from this the actual dosage of dichlobenil was calculated (see table 2 and Results). This spraying method was thought to be as close as possible to actual farmer practice.

Dichlobenil was used in the formulation of a wettable powder 50 % ').

---

' ) CASORON 133 w.p., trade name of Philips Duphar, the Netherlands.

## RESULTS

From the 1963/1964 trials it appeared that, in spite of an important lack of rainfall, yield increases were obtained in practically all plots, sprayed according to the conditions derived from the 1962/1963 tests. Table 1 gives a survey of the influence of the treatments on the yield in the 75 trials of 1963/1964, whereas table 2 gives results from a selection of representative tests.

Table 1  
Influence on the yield of treatments by dichlobenil

Yield as % of control	Nr of experiments	
50- 60	1 ')	
60- 70	0	
70- 80	1 ')	9 plots
80- 90	1	could not
90-100	1	be
100-110	11	harvested
110-120	19	due to
120-130	17	complete
130-140	6	lodging
140-150	6	
150-160	3	

' ) heavy rain during treatment.

Table 2 is given on the next page.

At one site a comparison was made between spraying by helicopter and by the normal land machine. As the stand of the blackgrass was not very dense, it was possible to compare both methods with regard to their influence on the selectivity of dichlobenil on the wheat

The results were:		
normal spraying	(3 kg dichlobenil/ha 4 " " " / "	Yield 4513 kg/ha " 4671 " / "
spraying by helicopter	(3 " " " / " 4 " " " / "	" 4600 " / " " 4367 " / "
	Blank	4413 " / "

## DISCUSSION

### 1. Mechanism of action of dichlobenil on blackgrass and on winterwheat.

The effect of dichlobenil on blackgrass and on winterwheat manifests itself by a slight foliar effect and an effect on the roots.

A comparative study of the development of the wheat and of the

Table 2

Results of some fieldtrials with dichlobenil in winterwheat for the control of blackgrass in France.

Site	Variety <sup>1)</sup>	Date of treatment	Growthstage wheat <sup>2)</sup>	Growthstage blackgrass	Dosage dichlobenil kg/ha	Blackgrass ears in control per m <sup>2</sup>	Percent control blackgrass	Yield winterwheat kg/ha	Yield increase above control kg/ha
La Fère	C	27/3	3 T	3 T	2,9	956	92,7	4550	+ 980
					3,5		93,0	4400	+ 830
Dury	C	28/2	3 T	2 à 3 T	2,5	1287	91,5	4360	+ 880
					3,-		95,3	4180	+ 700
Cutry	C	27/3	3 à 4 T	1 à 3 T	2,5	1010		5580	
					3,-		88,1	5580	+1320
					4,4		94,6	5520	+1160
Sergy	C	4/3	3 T	2 T	2,4	1152	88,4	4540	+1290
					3,-		94,5	4640	+1410
Le Tartre	C	12/3	3 T	3 T	2,5	295	99,7	5250	+ 830
Gaudran					3,-		100,0	4930	+ 510
Palluau	C	14/2	3 à 5 T	2 T à 5 T	3,-	393	98,7	4680	+1640
					4,-		99,-	4540	+1500
Palluau	C	14/2	2 à 5 T	2 à 5 T	2,5	211	98,6	4620	+ 980
					3,5		98,6	4210	+ 570
Issigeac	V	12/2	3 T	3 T à 5 T	2,9	777	92,4	3270	+ 790
					3,9		96,7	3660	+1180
Marignac	P	4/2	3 à 4 T	3 à 5 T	2,9	224	93,3	3820	+ 510
					3,8		95,1	3940	+ 640
Arrouède	Ch	14/2	5 à 7 T	3 à 7 T	3,1	456	83,1	3840	+ 700
					4,1		95,6	3850	+ 720
Ougnes	C	14/3	3 à 4 T	2 à 3 T	2,2	1253	89,7	3490	+1030
					2,9		92,8	3530	+1070
Villebaron	C	12/3	4 à 5 T	2 à 3 T	2,2	812	87,4	4230	+ 410
					2,7		94,2	4490	+ 670

<sup>1)</sup> C = Cappelle Ch = Champlein P = Poncheau V = Vilmorin

<sup>2)</sup> T = tiller

blackgrass makes it possible to draw up a hypothesis which might explain, to a certain extent, why dichlobenil can control blackgrass and is selective for winterwheat (see the development diagrams of the two plants, figure 1 and 2).

The blackgrass seeds germinate in general closer to the surface than the grain seeds. The primary roots of the blackgrass are little developed and seem to play, therefore, only a minor role during the tillering. The great majority of the blackgrass roots appears directly on the tillering level. The wheat, on the other hand, appears to form a network of fine roots on the primary roots, whereas the number of tillering roots is much smaller.

Shortly after a treatment, one notices that the roots from the tillering level become necrotic and die, both in wheat, when it has been sown too close to the surface, and in blackgrass; this leads to the death of the blackgrass which cannot continue to live from the small number of primary roots. The wheat, on the other hand, develops new roots under the sowing level, thus compensating the loss of the tillering roots. As the wheat has been sown at a certain depth (2-3 cm), most of the roots are protected.

## 2. Factors influencing the weedkilling effect of dichlobenil.

Environmental conditions influencing the effect of dichlobenil are:

### a. Wind.

In view of the volatility of dichlobenil, a strong or rather strong wind (10 m/second) at the time of treatment will lead to a considerable loss of active substance and will reduce the foliar intoxication and, to a certain extent, also the intoxication of the roots because part of the dichlobenil will be blown away from the leaves and from the treated soil.

### b. Temperature.

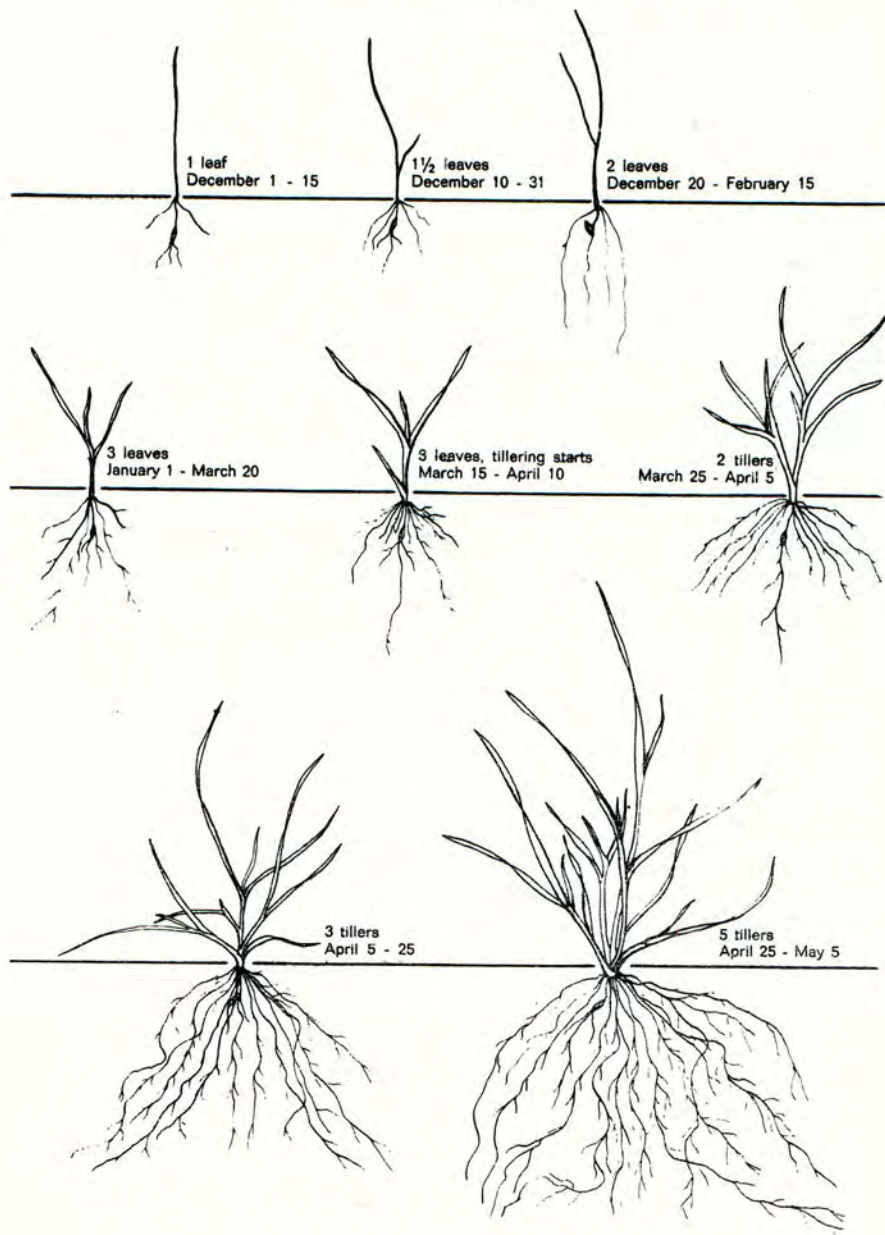
The temperature at the time of the treatment is of little importance, but the weeds are always easier to destroy when the temperature is mild, that is to say, favourable to their development.

Treatments in cold weather (temperature near zero) are possible only as long as the top layer of the soil has a certain, but not too high, humidity and the cold is of short duration. This, because the intoxication of the leaves and of the roots seems to take place only when the plants are growing. When there is a prolonged period of frost, no treatment should take place; when there is some night-frost, on the other hand, treatments can be carried out.

It is not advisable to treat the crop when the temperature is high (20-25°C), because of the volatility of dichlobenil. This weather condition is, on the other hand, not very likely in the periods in which the treatments should take place, i.e. from the end of January to the beginning of February in the South of France, and from the end of February to the beginning of March in the North.

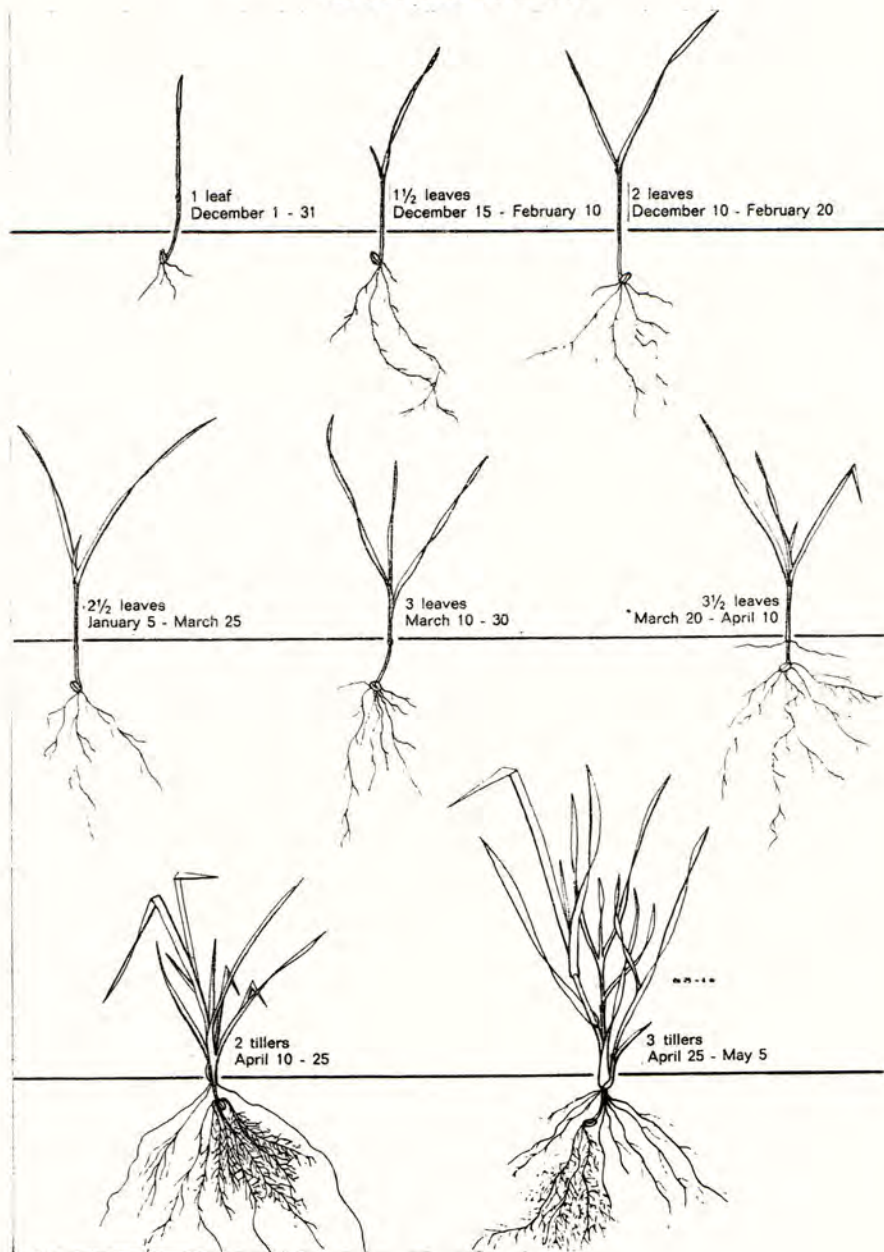
# GROWTH STAGES OF BLACKGRASS

Ile - de - France 1962-1963



# GROWTH STAGES OF WHEAT

Ile-de-France 1962-1963



#### c. Humidity of the soil at the time of treatment.

Although the dichlobenil which has been sprayed onto the leaves of a plant can lead to a foliar intoxication, this is of a transitory nature only and seems insufficient, in practice, to cause on its own the death of the plant.

The most important effect of dichlobenil is at the level of the roots (prolonged intoxication). When the roots are close to the surface (in the area where the dichlobenil is retained), this effect on the roots causes the death of the plant. But this intoxication is not fully effective when the roots are at a deeper level (over 3 cm).

The active substance, distributed over the soil, should be conducted quickly to the level of the roots of the young plants that are to be controlled. The surface-layer of the soil must, therefore, have a certain humidity at the time of treatment. Treatments carried out on dry or on a frozen soil will, as a rule, be very little effective.

The influence of the wind is particularly harmful in case the top layer of the soil is dry at the moment of treatment. The dichlobenil on the soil will then be blown off very soon, unless rain transports it rapidly to lower layers.

The best results have been reached when treatment was done on soils, that were neither too dry nor too wet to a depth of about 10 cm. If the soil is too wet, the effect remains excellent, but there will be rather considerable phytotoxicity on the wheat.

#### d. Condition of the wheat.

The health of the wheat seems to play a very important role in the final effect of dichlobenil. Treatments should be carried out only on healthy, vigorous and close-standing wheat. Under those conditions, the crop very rapidly establishes the ascendancy over the blackgrass and this completes the results of the treatment. Only rarely will dichlobenil have a good effect if the treatment is carried out on a poor stand of wheat.

The same holds true when the wheat is asphyxiated from an excess of water during the growth (either before or just after treatment). The fertilization too plays an important role in the final efficacy of the herbicide. If it is insufficient, the results are always mediocre, since the wheat must share a poor fertilization with very exacting weeds.

#### e. Nature of the soil.

With the exception of certain soils in the South of France, the wheat-fields have a composition with a high level of a colloidal loam-humus complex. Dichlobenil remains, under these conditions, a long time in the soil. However, there is as a rule little chance that the active substance will be phytotoxic by reaching the roots of the wheat. In the South of France, on the other hand, there are certain soils which are too calcary and too little fertilized (organic and mineral matter), as a result of which the effect of dichlobenil may be less pronounced. When these soils contain a certain amount of clay, spraying



cannot take place under humid conditions. Treatment can be carried out only during frosty weather or in dry spells. These conditions, above all when there has been a prolonged period of drought, combined with the weakness of the absorbing complex, explain certain failures. In these soils there can be phytotoxicity to a greater extent when heavy rainfall carries the dichlobenil to the level of the wheat-roots.

In some sites with very high humus content, dichlobenil has no effect. In practice this type of soil is used very seldom for wheat-growing in France. It occurred only at one test-site, where wheat was cultivated immediately after poplar trees had been cleared away from that site.

#### f. Cultivation measures after treatment.

All kinds of cultivation measures after treatment (such as rolling) can alter the distribution of the dichlobenil in the top layer of the soil, causing new emergence of weeds.

### 3. Factors influencing the selectivity of dichlobenil for winterwheat.

#### a. Vegetative stage of the wheat.

Throughout tillering, the wheat has a good resistance to dichlobenil, even when it is used at high dosages. It should nevertheless be pointed out that treatments given immediately before the shooting of the wheat (during the 3 weeks preceding it) can often lead to strong phytotoxicity. The treatments should not be executed later than about 3 weeks before shooting. Safest are the treatments carried out at the three-tiller stage.

#### b. Susceptibility of different varieties of winterwheat to dichlobenil.

Etoile de Choisy and Cappelle Desprez, varieties which are frequently grown in France, appear to be tolerant to dichlobenil.

#### c. Rainfall and humidity of the soil.

As pointed out earlier, treatment should be avoided when heavy rainfall is expected. Considerable phytotoxicity was regularly observed in wet soils, e.g. in low lying water-logged fields. The phytotoxicity seems to be definitely linked up with the humidity of the soil at the moment of treatment and afterwards.

#### d. Dosage

The dosage recommended for practical application is 6 kgs of the formulated product with 50 % active substance per hectare. To prevent overlapping during the spraying it is always best to use a spraying boom with a width equal to the sowing equipment.

One should not give the treatment less than 8 days after a cultivation measure (harrowing, rolling etc.), and one should not take such cultivation measures after the treatment. Apart from the damage they can do to the wheat, these operations alter, after the treatment, the distribution of dichlobenil in the soil and cause local emergence of weeds.

It is for the same reason that treatment should also be avoided when the wheat is attacked by slugs or flies or when it has suffered from frost.

e. Cultivation methods.

Wheat is very susceptible to all injuries brought about just before or after the treatment. The trampling of the testplots has often led to considerable phytotoxicity, no matter how careful the treatment itself had been done. In large tests, the traces made by the tractor wheels could sometimes be observed right up to the harvest.

f. Sowing depth.

All that has been said above applies only as long as the wheat has been sown at the normal depth (an average depth of 3 cm). Considerable phytotoxicity was noticed when the wheat had been sown near to the surface (at a depth of less than 1 cm).

4. Sensibility of different weeds to dichlobenil.

When using dichlobenil for the control of blackgrass in winter-wheat (treatment at the end of January/beginning of February in the South of France and at the end of February/beginning of March in the North) we observed the following sensibility of other weeds:

<u>Resistant plants</u>	
Papaver spp.	3 and 4 kg
Polygonum convolvulus	3 kg
Convolvulus arvensis	all dosages
Rumex sp.	"
Fumaria officinalis	"
Ranunculus arvensis	"
Potentilla repens	"
Agropyron repens	"
Agrostemma githago	"
<u>Moderately sensible plants</u>	
Cirsium arvense	3 and 4 kg
Vicia sp.	"
Avena fatua	"
Avena Ludoviciana	"
Anagallis arvensis	"
Raphanus raphanistrum	"
Sinapis arvensis	"
Lolium perenne	2 and 3 kg
<u>Sensible plants</u>	
Alopecurus myosuroides	2 kg
Matricaria spp.	"
Chenopodium spp.	"
Veronica hederifolia	"
Apera spica venti	"

### References

- BARNESLEY, G.E. (1960) The herbicidal properties of 2,6-dichlorobenzonitrile. Proc. 5th British Weed Control Conf. 2, 597-616.
- BARNESLEY, G.E. and ROSHER, P.H. (1961) The relationship between the herbicidal effect of 2,6-dichlorobenzonitrile and its persistence in the soil. Weed Research 1, 147-158.
- DAAMS, J. and BARNESLEY, G.E. (1961) Les propriétés herbicides du 2,6-dichlorobenzonitrile. Comptes Rendus des Journées d'Etudes sur les herbicides E.W.R.C.-Columa, Paris 1961, 60-67.
- KOOPMAN, H. and DAAMS, J. (1960) 2,6-Dichlorobenzonitrile: a new herbicide. Nature 186, 89.
- MASSINI, P. (1961) Movement of 2,6-dichlorobenzonitrile in soils and in plants in relation to its physical properties. Weed Research 1, 2-142.
- STOVELL, F.R. (1962) The use of dichlobenil (2,6-dichlorobenzonitrile) in cereals. Proc. 6th British Weed Control Conf. 1, 235-251.

THE EVALUATION OF TRIALLATE  
FOR THE CONTROL OF WILD OATS  
IN CEREALS IN THE U.K.

G.B. Lush and A.J. Mayes  
Boots Pure Drug Co. Ltd., Lenton Experimental Station

Summary An account is given of three years work on the field performance of 2,3,3-trichloroallyl diisopropylthiolcarbamate (triallate) in wheat and barley crops in the U.K. Factors influencing wild oat control and crop safety are described. The advantages of different techniques of usage are discussed.

INTRODUCTION

2,3-dichloroallyl diisopropylthiolcarbamate (diallate) was first described by Selleck<sup>1</sup> as having the ability to control *Avena fatua* (wild oat) in spring barley. Subsequent work by Selleck<sup>2</sup> and Hannah *et al*<sup>3</sup> showed that diallate could be used in a range of crops including spring wheat. The selectivity of the compound in wheat was markedly less than in barley and the discovery of the more selective properties of 2,3,3-trichloroallyl diisopropylthiolcarbamate (triallate) reported by Selleck<sup>4</sup> was a significant advance in pre-emergence wild oat control in cereal crops. The work referred to above showed that both compounds require to be incorporated into the soil to minimise volatilisation and that wild oat seedlings are killed as they pass through the chemically active layer whilst barley and wheat seedlings emerge safely through this layer provided the seed lies beneath it. Wheat and barley seedlings can be seriously injured if they come into contact with the chemically treated layer. Both compounds were described as having very effective wild oat control properties. Two methods of use are possible, "pre-drilling" where the herbicide is applied and incorporated into the soil before drilling, and "post-drilling" where application and incorporation are made after drilling.

At the 5th British Weed Control Conference, 1960, Holly<sup>5</sup>, Holroyd<sup>6</sup>, Evans<sup>7</sup> reported glasshouse and field experiments in the U.K. with diallate. Selleck and Hannah<sup>8</sup> described the field performance of triallate at the 6th British Weed Control Conference, 1962, where cereal work in the U.K. was reported by Evans<sup>9</sup>, Parker<sup>10</sup> and Holroyd<sup>11</sup>. In 1962 triallate was made available to the authors for field evaluation by courtesy of the Monsanto Chemicals Limited.

METHOD AND MATERIALS

In the work now reported two types of field trials were employed viz the detailed and the user trial. In the former each treatment was replicated into three blocks. Plot size varied between  $\frac{1}{100}$  and  $\frac{1}{40}$ th acre. The widespread user trials combined the function of confirming under varying conditions the aspects of usage established in the detailed trials with that of determining farmer acceptance. They were frequently supervised by fieldstaff in order to observe the actual details of the operation. These trials were unreplicated comparisons in one acre plots of two, or sometimes as many as four, treatments.

In all the trials one standard 40% miscible oil formulation of each compound was employed.

In 1962 the two compounds, diallate and triallate were compared in twelve detailed trials, six each in spring wheat and spring barley. In two of these trials several varieties were included and in one barley trial a wheat variety was included. Rates of use varied by 0.125 lb increments between 1-2 lb/ac a.i. Aspects of the timing of spraying in relation to drilling and incorporation in relation to spraying were investigated. Due to the importance of separating the crop seed from the chemically treated layer of soil, a comparison was planned in one trial, of applications made in crops drilled at two depths.

In 1963, triallate was again compared with diallate in a programme of ten detailed and eighty-four user trials. The detailed trials were designed to investigate:-

- a) Rate of use ( $\frac{3}{4}$  - 3 lb/ac a.i.)
- b) Timing of application (pre- and post-drilling with varying delays between drilling and spraying)
- c) Drilling depth 1 -  $1\frac{1}{2}$ ", 2<sup>+</sup> - 3".
- d) The relative efficacy of a range of implements for incorporation -  
Two types of chain harrow  
Three types of straight toothed harrow  
and Scandinavian type spring tined harrows were compared.
- e) Different depths of incorporation in relation to drilling depth.
- f) Relative susceptibility of spring barley and spring wheat.

In the user trials comparison was made between triallate and diallate at  $1\frac{1}{4}$  lb/ac a.i. in seventy-three spring barley and eleven spring wheat trials. Fifty-nine comparisons of the two chemicals were made by the post-drilling method and twenty-seven by the pre-drilling method.

In autumn, 1963, a limited number of detailed and user trials was laid down to investigate the possibility of application of tri-allate in winter cereals.

In 1964, several user trials were carried out to obtain further information on the performance of triallate in spring wheat.

## RESULTS

The spring of 1962 was characterised by extremely good but often loose (puffy) tilths, following the hard prolonged winter. There was a tendency for cereals to be sown in early February due to the ideal seed bed conditions at that time.

In the 1962 trials, control of wild oats by both compounds was good. In some trials where conditions were optimum 1 lb/ac a.i. of both compounds gave satisfactory wild oat control.  $1\frac{1}{4}$  lb/ac a.i. were always adequate. In all but one of the comparisons, the highest rates of triallate used ( $1\frac{1}{2}$  or 2 lb/ac a.i.) were superior to the lowest rate of diallate (1 lb/ac a.i.) in terms of crop safety. No varietal differences in susceptibility to diallate or triallate were found. In the comparison between wheat and barley the former was found to be more susceptible. This was much less marked with

triallate than diallate. The importance of carrying out incorporation on the day of spraying was demonstrated.

An attempt in one trial to drill at two different depths failed due to difficulty in controlling depth of the drill in the very fine tilth prevailing.

With both of the herbicides no difference was detected between pre- and post-drilling treatments in the one comparison made. From the standpoint of wild oat control, delays in drilling of less than three weeks did not reduce wild oat control. A feature of the detailed trials in 1962, was the occurrence of reduced crop stand in the wheel tracks of the tractor carrying the spraying and incorporation equipment. Selleck<sup>12</sup> has suggested that the reason for such wheeling effects in diallate and triallate treated crops is due to the greater capillarity of the soil in the wheel tracks making the chemical more rapidly and readily available resulting in more severe effects on the crop, also compressing the soil and placing treated soil closer to the seeds.

In 1963, results with triallate followed the same successful pattern as in 1962. In both detailed and user trials wild oat control at  $1\frac{1}{4}$  lb was generally very effective and the greater selectivity in barley and wheat was again amply demonstrated. The table shows typical detailed trial results.

Soil conditions in early spring, 1963, were initially very similar to those prevailing in 1962 but generally widespread rain later settled the loose tilths into ideal seed bed conditions. Under these conditions it was deemed more likely that depth of drilling comparisons could be made and in fact great attention was paid to the depth of drilling in all the trials carried out. In practice it was found to be difficult to make critical comparisons between exact drilling depths due to variation in the deposition of seed at given drill settings under different soil conditions. From a consideration of all trials carried out however, it was apparent that for the most favourable results, the seed should lie at  $1\frac{1}{2}$  - 2" below the soil surface. Drilling appreciably shallower than this reduced selectivity due to coincidence of seed and chemical layer while very deep drilling delays seed emergence and impairs vigour.

Considerable emphasis was placed on the post-drilling method of use of triallate, a technique found in other countries to be associated with a greater margin of crop safety than the pre-drilling method. In the trials being reported, whilst this was generally confirmed the pre-drilling method also gave an adequate margin of crop safety.

The main reason for this difference between the two methods would appear to be that in the pre-drilling method, the crop seed has to pass below the chemically treated layer of soil while in the post-drilling method the seed is already in place. A small amount of chemical may reach the seed via the tines of the incorporating implement but this would not appear to be appreciable. Another reason is that implements such as seed harrows used for post-drilling application are of shallower draught than those such as spring tined harrows used for pre-drilling incorporation. A third reason for somewhat greater safety was found in the farmers' reluctance to harrow too deeply over the top of his crop seed. This tends

to ensure that over deep incorporation does not take place. This tendency to incorporate somewhat too shallowly under these circumstances may explain the slightly less effective wild oat control by this method.

Wheeling effects were again noticeable but usually in 1963 only in the post-drilling treatments. These wheeling effects occurred also in untreated plots across which tractors had travelled after drilling. Recovery in control plots was usually greater than in treated plots. The power of recovery of wheat was markedly greater than that of barley. Whilst these wheeling effects are not directly attributable to the chemical they represent a factor to be considered when deciding which method of use to employ.

The detailed comparison in 1963 of different implements for post-drilling incorporation demonstrated that no particular implement could be singled out as ideal for all circumstances. As a type, however, spike and straight toothed harrows of medium weight gave the most generally successful results during that year. Under the loose soil conditions in 1962 rather lighter harrows were necessary whilst under firmer soil conditions than prevailed in 1963 heavier equipment might well be necessary. This indicates that choice of incorporating implements must depend on soil conditions and must therefore be made by the farmer at the time of use.

In 1964 the trial results in spring wheat confirmed the adequate selectivity of triallate in this crop.

In the winter cereal trials wild oat control results were good where seed bed conditions were favourable. In wet or coarser seed beds where good incorporation was not possible, wild oat control results were unsatisfactory. Where seed bed conditions were favourable the margin of crop safety with triallate was satisfactory. Further work on this method of use is in progress.

#### DISCUSSION

The efficacy of triallate for the control of wild oats in cereals and its safety margin in spring barley and wheat has been amply demonstrated at a rate of  $1\frac{1}{4}$  lb/ac a.i. Even so it is still important to ensure that the crop seed is separated from the chemically treated layer.

Good distribution of the chemical in the soil is the key to successful wild oat control and is an important factor in achieving crop safety. These conditions can only be achieved under conditions of good firm tilth. Wet or cloddy soil conditions do not enable good distribution to take place and results will not be satisfactory.

The chemical must remain above the crop seed and it is important therefore that incorporation is not too deep. In practice, a depth of penetration of the incorporating implement of not more than 3", deposits the bulk of the chemical in a very shallow surface layer. Drilling in such a way that the crop seed lies at  $1\frac{1}{2}$  - 2" below the soil surface ensures adequate separation of chemical and seed. Drilling the seed too deeply can of course be deleterious for crop husbandry reasons.

Successful results have been achieved by both pre- and post-drilling methods. The pre-drilling use is often associated with somewhat better wild oat control and the post-drilling method with somewhat greater crop safety. Apart from these considerations choice of method is largely a matter of the farmers convenience.

Application of triallate at the time of drilling winter cereals has shown promise provided soil conditions are favourable. Further work on this use is in progress.

#### Acknowledgements

The authors wish to acknowledge the collaboration of Dr. G.W. Selleck and Mr. D.M. Evans, of Monsanto Europe Limited and Monsanto Chemicals Limited respectively. They also wish to acknowledge the collaboration of Dr. E.L. Leafe for radio-active tracer soil studies and to express their appreciation for the assistance rendered by members of Boots fieldstaff, by the staff of Lenton Experimental Station and the many farmers who have been involved in trials.

#### References

- SELLECK, G.W. (1958) Various herbicides for wild oat control. Res. Rep. Nat. Weed Comm. (Western Section) 48-49.
- SELLECK, G.W. (1959) E.P.T.C. and Avadex for wild oat control in various crops, 1959. Res. Rep. Nat. Weed Comm. (Western Section) 52-53.
- HANNAH L.H., HAMEL, P.C. and SELLECK, G.W. (1960) The performance of 2,3-dichloroallyl diisopropylthiolcarbamate in the wild oat areas of North America. Proc. 5th Brit Weed Cont Conf 2, 481.
- SELLECK, G.W. (1961) 2,3-DCDT (Avadex) to date. Annual Conf of Manitoba Agronomists, Dec. 19 and 20 (1961), 38-39.
- HOLLY, K. (1960) Pot experiments with new herbicides for the control of wild oats. Proc. 5th Brit Weed Cont Conf 2, 533.
- HOLROYD, J. (1960) Some preliminary experiments with 2,3-dichloroallyl diisopropylthiolcarbamate. Proc. 5th Brit Weed Cont Conf 2, 495.
- EVANS, S.A. (1960) The control of wild oats (*Avena fatua*) with barban and 2,3-dichloroallyl diisopropylthiolcarbamate. A summary of N.A.A.S. experiments 1960. Proc. 5th Brit Weed Cont Conf 2, 503.
- SELLECK, G.W. and HANNAH, L.H. (1962) A review of 1962 field results with di-allate and triallate in the United States and Canada. Proc. 6th Brit Weed Cont Conf 1, 361.



- EVANS, S.A. (1962)                      The control of Avena fatua (wild oats) in spring cereals with di-allate, tri-allate and barban. Proc. 6th Brit Weed Cont Conf 1, 297.
- PARKER, C. (1962)                      Factors affecting the performance of di-allate and tri-allate in the control of Avena spp. in cereals. Part I Pot Experiments. Proc. 6th Brit Weed Cont Conf 1, 321.
- HOLROYD, J. (1962)                      Factors affecting the performance of di-allate and tri-allate in the control of Avena spp. in cereals. Part II. Field Experiments. Proc. 6th Brit Weed Cont Conf 1, 325.
- SELLECK, G.W. (1961)                      Private communication.

Notes relating to table on following page

Key

D-12	=	sprayed and incorporated 12 days before drilling.
D+	=	" " " immediately after drilling.
D+1	=	" " " 1 day " "
D+3	=	" " " 3 days " "

Note on yield figures

These trials were primarily designed for quantitative assessment of wild oat control and crop stand. It was considered of interest to present yield data from these same trials to indicate the correlation between the three factors. As these trials were not designed for assessing yields, a statistical analysis was not carried out.

Table of Typical Results of Detailed Trials in 1963

	Rate lb/ac a.i.	Date of spraying	Timing of application	% reduction of stand				% yield				Percentage control of wild oats	
				Barley		Wheat		Barley		Wheat		di-	tri-
				di-	tri-	di-	tri-	di-	tri-	di-	tri-	di-	tri-
				-allate	-allate	-allate	-allate	-allate	-allate	-allate	-allate	-allate	-allate
Farndon Notts	1 1½ 1¾ 2½	26 Apr.	D+	0	0	20	10	-	-	-	-	89.0	88.0
				20	0	60	15	125.6	125.7	110.6	109.7	92.1	91.2
				30	0	60	20	119.8	124.8	96.7	105.3	94.0	92.5
				40	0	70	30	126.4	128.1	70.8	113.3	98.7	99.1
Thoroton Notts	1½ 1¾	27 Mar.	D-12	25	0	30	0	126.0	126.0	107.5	115.0	91.8	94.3
				40	0	50	10	96.2	105.8	95.0	110.0	94.4	90.1
	1½ 1¾ 2½	8 Apr.	D+	0	0	15	10	104.0	104.0	92.5	107.5	92.5	98.6
				10	0	30	10	106.0	106.0	84.8	100.0	99.3	97.3
				20	10	50	20	96.0	100.0	82.6	100.0	100.0	99.3
Sedgebrooke Lincs	1 1½ 1¾ 2½	23 Apr.	D+1	0	0	10	0			95.2	103.6	41.1	42.2
				10	0	20	0	No data		100.5	105.2	62.4	68.8
				20	0	30	10	available		93.3	106.7	80.9	82.7
				30	10	50	15			85.5	111.8	92.7	91.3
Marston Lincs	¾ 1 1½ 1¾	25 Mar.	D+3 medium harrows 3-4" pene- tration	10	0			Crop				84.4	85.4
				20	10							89.9	94.6
				20	10			badly				95.6	89.1
				50	20			laid				97.5	94.1
	¾ 1 1½ 1¾	" "	D+3 light harrows 1½-2½" penetration	0	0							72.5	68.4
				10	0			at				81.3	81.3
				10	0							87.6	90.0
				20	10			harvest				84.9	96.6

Research Summary

THE GENETIC RESPONSE OF BARLEY VARIETIES TO DDT AND BARBAN AND  
ITS SIGNIFICANCE IN CROP PROTECTION

J. D. Hayes\* and R. K. Pfeiffer\*\*

\*Welsh Plant Breeding Station, Plas Gogerddan, Aberystwyth

\*\*Chesterford Park Research Station, Saffron Walden

The problem of developing chemical treatments of sufficient selectivity for controlling weeds in crops has so far invariably been approached through research for the appropriate molecular structure and formulation of herbicides. Our investigations show that the plant breeder can make a contribution to the solution of difficult selectivity problems by finding resistant crop varieties or even by directed breeding for herbicide resistance.

Differences have already been established in the reaction of barley to DDT and barban. The present investigations have been concerned with the genetical aspects of this problem. On susceptible varieties DDT causes leaf chlorosis, and barban produces two characteristic symptoms:

(i) barban chlorosis of the leaves and (ii) apical inhibition.

We have assessed the effect of DDT and barban on 11 varieties of barley in field and glasshouse trials and a summary of the results is given below.

The response of 11 barley varieties to DDT and barban

Variety	Treatment		
	DDT	Barban	
	Chlorosis of leaves	Chlorosis of leaves	Apical inhibition*
Plumage Archer	R	R	+++
Isaria	S	R	+
Spratt Archer	R	R	+
Kenia	R	S	+++
Rika	S	S	++
Proctor	R	R	++++
Maja	R	S	+++
Goldthorpe	R	R	+
Archer	R	R	+
Spratt	S	R	+
Carlsberg II	R	R	+

R = Resistant, S = Susceptible. \* + = highly resistant. +++ = extremely susceptible

Chlorosis following the application of DDT and barban is an all-or-nothing effect, whereas in apical inhibition there are several degrees of resistance. The criteria used to assess the effect of barban on apical inhibition were fresh weight of seedlings, fertile tiller production at maturity and visual estimates of damage at the seedling stage and at maturity.

Inheritance studies of the progeny of hybrids between the varieties listed in the table have shown that resistance to the chlorosis reaction of both DDT and barban is controlled by independently inherited single recessive genes. Resistance to apical inhibition on the other hand appears to be quantitatively inherited.

When  $F_2$  and  $F_3$  segregating populations in the field were subjected to spraying by barban, the proportion of resistant plants in successive generations was markedly increased. It is not yet certain whether the actual tolerance level of the extremely resistant genotypes from these crosses exceeds the range of the parents, but since Proctor, which was obtained from a population of a cross between Plumage Archer and Kenia, is more susceptible than either parent, clearly transgressive segregation for this character has occurred in one direction, and it is important to establish whether transgressive segregation can be obtained in the other direction i.e. greater resistance.

Three points of importance emerge from these investigations:

1. The differences in reaction of barley varieties to DDT and barban are under relatively simple genetic control and in these circumstances resistance can be fairly easily manipulated in a plant breeding programme.
2. The possible existence of similar control systems in other crops for other chemicals should be borne in mind when developing new varieties of crops and new chemical compounds.
3. Collaboration between plant breeder and chemist could lead to a far more efficient control of weed, pest and pathogen by the development of varieties of crop plants with a high degree of tolerance to the chemicals employed, especially under conditions where selectivity is narrow, such as in the control of other monocotyledonous weeds in cereals.