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A progress report on experimental work

by the A.R.C. Weed Research Organisation,

May - October, 1963.

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K. Holly and J. Holroyd

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3,5-DIIODO-4-HYDROXYBENZONITRILE

A Progress Report on Experimental Work by the A.R.C. Weed Research Organisation May-October, 1963.

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SUMMARY

3,5-diiodo-4-hydroxybenzonitrile was tested for post-emergence selectivity on 46 species of temperate and tropical weeds and crops in pots. Other pot experiments were concerned with (a)the susceptibility of seedlings germinating in treated soil and (b)the reduction of effectiveness of foliar applications by subsequent simulated rainfall. Two field experiments evaluated its potential for the control of a range of annual weeds, particularly Matricaria recutita and Chrysanthemum segetum, in spring wheat and spring barley crops. Results were promising and suggest that the herbicide has possibilities for selective weed control in cereals and other grass crops and perhaps also in certain leguminous and vegetable crops. There is a discussion of the further research that is desirable on this herbicide.

INTRODUCTION

In early May 1963 a quantity of 3,5-diiodo-4-hydroxybenzonitrile was supplied to the A.R.C. Weed Research Organisation as a promising new herbicide. Accompanying information from the manufacturer indicated that field trials in 1962 in Europe and the U.S.A. had shown good selectivity for the control of annual weeds (including Matricaria spp., Polygonum spp., Galium aparine, Stellaria media and Fumaria officinalis) in wheat, oats and barley when the crop had tillered. The herbicide was supplied as a 50% w/w wettable powder (ACP.62-177A, originating from Amchem Products Inc. via A. H. Marks & Co. Ltd.). All experiments described in this report were conducted with this formulation. This chemical was stated to have an acute oral LD_{50} to rats of 118 mg/kg and it was therefore recommended that it should be handled with care.

Recently two publications have appeared from other sources describing the herbicidal properties of this chemical (Wain, R.L. Nature 200,28 and Carpenter, K. & Haywood, B.J. Nature 200, 28-9). These papers likewise indicate the selective potentialities of this and related compounds for annual weed control in grass crops and also in some leguminous crops.

POT EXPERIMENTS

The wettable powder formulation of 3,5-diiodo-4-hydroxybenzonitrile was applied at three doses in a standard type post-emergence selectivity experiment on 46 species.

In this experiment (G.63.6), all species were grown in a standard potting compost, University of California No.IIC mix (except for rice and the soft fruits grown in soil). Unless specified to the contrary all species were seedlings, sprayed at the 2-4 leaf stage, the sowings being staggered to obtain all species at approximately the same stage of growth at the time of spraying. The exceptions were:

Agropyron repens, grown from rhizome sections, the shoots having 3-4 leaves when sprayed

Cyperus rotundus, grown from tubers, the shoots having 8-9 leaves when sprayed

Strawberry - newly established plants from runners, with the fourth leaf expanding

Raspberry - newly established plants from suckers with the sixth leaf developing

Blackcurrant - rooted cuttings with about 7 leaves

The herbicide was applied on 16:8:63 at doses of 0.125, 0.5 and 2.0 lb/ac (a.i.) as a suspension in distilled water, at a volume rate of 29 gal/ac. using a Teejet 8002E fan nozzle. Visual observation of spray retention at the lowest dose suggested that retention was good by almost all species, the poorest retention being by pea and Papaver rhoeas. After spraying the plants were kept in the greenhouse for a day at temperatures within the range 9-28°C, with a mean temperature of c.15°C. Humidity was relatively high at and after spraying. All plants were given a heavy overhead watering to wash residues off the foliage one day after spraying. Thereafter the tropical species remained in a heated greenhouse section whilst the remainder were moved into the open.

Many dicotolydenous species were showing scorch and necrotic spots within a day of spraying, the severity increasing with dose. All monocotyledonous species, together with groundnut, pea, dwarf bean and the soft fruits, appeared unaffected at this time. Observations almost a week after spraying showed that necrosis had become more severe in susceptible species. It was accompanied by retardation of growth and chlorosis of any young leaves that had continued to develop. In many instances plants were already dead. Some grasses, notably wheat, barley, sorghum, Poa annua and Agropyron repens, continued to be unaffected. Other grasses at the higher doses showed one or more of the following symptoms:-

a) a certain amount of scorch of the older leaves (Italian ryegrass, oats, cocksfoot)
b) necrosis of the leaf tips (cocksfoot, rice)
c) a little chlorosis of the young leaves (Alopecurus myosuroides, maize, Eleusine indica)

Other species continuing to show only a slight effect were onion, which tended to be flaccid at the highest dose, and pea which showed some chlorotic patches on the leaflets but was otherwise unaffected.

Final observations were made $2\frac{1}{2}$ to $3\frac{1}{2}$ weeks after spraying, by which time untreated control plants of all species had made a very substantial amount of growth and all grasses, for example, were tillering vigorously. Assessments consisted of a count of all plants still alive and a score of the state of the surviving plants on a scale 0-7 as follows:

0 = completely dead

1 = moribund but not all tissue dead

2 = alive, with some green tissue, but not making any growth

3 = very stunted but apparently still making a little growth

4 = considerable inhibition of growth

5 = readily distinguishable inhibition of growth

6 = some detectable effect as compared with control - colour difference, morphological abnormality, epinasty or very slight reduction in growth

7 = indistinguishable from control

The results obtained are presented in Figure 1. All species are listed on the left, divided into groups as follows:

A Temperate agricultural crops

B Vegetable crops

C Temperate weeds

D Tropical crops

E Tropical weeds

F Fruit crops

Cultivars of a few weed species, namely Chrysanthemum segetum, Sinapis alba, Raphanus raphanistrum and Papaver rhoeas, are used because of difficulties in obtaining even germination of seed of the wild species. For each dose on each species there are a pair of horizontal lines in the Figure; the upper one represents the percentage survival of plants, the lower one the score as described above. The vertical broken line on the right represents the control level; i.e. a survival of 100% and a score of 7. A circle indicates that all plants were dead.

The outstanding feature of these results is the high degree of resistance of all grasses to as much as 2 lb/ac. Rice was anomalous in that it showed an appreciable mortality at the highest dose. This result should be regarded with caution as there was considerable plant to plant variation in the response of rice to this and other herbicides in this particular experiment.

Other crop species showing a noteworthy degree of resistance were pea and onion, and to a lesser extent groundnut and dwarf bean. An appreciable degree of selectivity seems to be indicated in these crops. At the lowest dose a number of other crops showed some resistance and insofar as a few species were still susceptible at this dose there might be possibilities of a limited but useful selectivity. Interesting examples are lucerne, the clovers, carrot and the fruit crops.

The most susceptible species were the easily wetted Brassicae - Sinapis alba and turnip, together with Tripleurospermum maritimum and cotton. At 0.5 lb/ac an impressive control of dicotolydenous seedling weeds was obtained; as well as those already mentioned, Chrysanthemum segetum, Senecio jacobaea, Raphanus raphanistrum, Papaver rhoeas and Chenopodium album were all killed, while Polygonum lapathifolium, Rumex crispus and Stellaria media were very severely affected. An exception was Galium aparine which showed a very high degree of resistance. As with so many herbicides, Cyperus rotundus showed its usual resistance, not even exhibiting any scorch in spite of the ease with which its foliage is wetted by the spray.

A second experiment (G.63.11) investigated the possibility of action on germinating seedlings in the soil and any selectivity arising therefrom. Doses of 0.125, 0.5 and 2.0 lb/ac were applied to the soil at a volume rate of 29 gal/ac. Within 20 minutes of application, the soil in the container was thoroughly mixed to its full depth (2.5 inches) to secure even incorporation of the herbicide. Twenty two weed and crop species were then sown into the treated soil at appropriate depths. The containers were kept in the greenhouse from sowing (9.10.63) at a minimum temperature of 12°C, with a watering regime which precluded leaching of the herbicide out of the containers. Even the top dose did not cause a severe retardation of growth of any species. Thus it seems that foliar entry is the more important route for securing herbicidal effects and action through the soil is relatively unimportant.

One further pot experiment (G.63.9) followed up the relative susceptibility to this herbicide shown by seedlings of Rumex crispus in the post-emergence selectivity experiment. A supply of established plants of Rumex obtusifolius, sown 4½ months previously, was available. These had a crown of 5-6 leaves and a well developed rootstock. The object of the experiment was to ascertain whether certain new herbicides, of which 3,5-diiodo-4-hydroxybenzonitrile was one, had permanent effects on a perennial weed and whether a short or long period before the onset of rain is necessary for full effectiveness. Accordingly, doses of 0.0625, 0.25 and 1.0 lb/ac. in 30 gal/ac of water were applied at about 9.30 a.m. on 11.9.63, and various sets of plants were given a heavy overhead washing to remove spray residues from the leaves 1, 6 and 24 hours after spraying. All plants were kept in a greenhouse section for the duration of the experiment. Over the ensuing month various effects developed, primarily a retardation of growth, slight at the lowest dose and considerable at the highest dose, and a chlorosis of the leaves expanded at spraying, increasing in severity with increasing dose. In the next month, however, recovery occurred and at the end of two months after spraying plants treated with this herbicide were not readily

distinguishable from controls and there was obviously no permanent effect. Comparison of the symptoms produced by the plants washed at various times suggested that they were more severe where washing had been postponed for 6 hours after spraying than where they had been washed after 1 hour.

FIELD EXPERIMENTS

The wettable powder formulation of 3,5-diiodo-4-hydroxybenzonitrile (ACP.62-177A) was included in two screening experiments during 1963, together with a number of other candidate herbicides, to assess its potential as a post-emergence herbicide for the selective control of Chrysanthemum segetum and Matricaria recutita in spring barley and spring wheat.

The two experiments (E.9.63 at Begbroke and E.12.63 at Culworth) were identical in layout; a randomised block design replicated twice with a control plot between each pair of treated plots. Half of each plot was drilled with a spring cereal (Pallas barley in E.9.63 sown on 9.4.63; Jufy wheat in E.12.63 sown on 8.4.63). Plot size was 6 x 2 yd. with a 1 yd. discard between each plot.

The treatments (1.0, 2.0 and 4.0 lb/ac a.i.) were applied post-emergence on the crops and weeds by means of an Oxford Precision Sprayer at a volume rate of 21.5 gal/ac. Table I gives relevant data.

The pre-spraying densities of the various weeds at Begbroke and Culworth were assessed by counting the numbers of plants present in 40 random 6 in. quadrats (20 in crop, 20 on fallow) on each of 8 control plots on 13.5.63 and 6.5.63 respectively. The stages of growth of both the weeds and the crops were assessed at the time of treatment by examining 20 random individual plants of each species at 4 equally spaced positions on each experiment (see Table I).

The post-spraying assessments were made at Begbroke on 5.7.63 by counting the number of plants of Matricaria recutita and Chrysanthemum segetum present in 20 random 1 ft. quadrats (10 in crop, 10 on fallow) on each plot.

The post-spraying assessments of Chrysanthemum segetum at Culworth were made similarly on 18.6.63.

The Stellaria media at Culworth was so dense that counts were not possible and it was scored for density only. The crop density was assessed at Culworth (on 3.7.63) and Begbroke (on 5.7.63) when all the heads were fully emerged by counting the number of heads present in four 18 in. random quadrats on each plot.

Observations were also made at intervals.

All counts and scores have been expressed as percentages of the adjacent control plot and are summarised in Tables II and III. Details of observations are given in Table IV.

Crops - Both spring barley and spring wheat were very little damaged by any of the treatments except for a temporary scorching of the leaf tips. This occurred at all doses but was most extensive at 4 lb/ac. However, the figures for head counts and straw length (Begbroke only) give no indication of a significant long-term reduction in growth. There was no evidence of any deformity or sterility of the inflorescences of either the wheat or barley.

Weeds - As indicated in Tables III and IV, doses of 1 lb/ac gave a good control of a number of annual weeds. These included Matricaria recutita, Raphanus raphanistrum, Papaver rhoeas and Veronica spp. which were only present in any number at Begbroke. Stellaria media was well controlled at Begbroke, but not so successfully at Culworth. Here the plant density was very high (over 200 plants/sq ft and it was overshadowed by Chrysanthemum segetum; in addition, there was some secondary germination, particularly on the treated plots. The density of Chrysanthemum segetum was very little reduced by any of the treatments at either Begbroke or Culworth but growth was checked. In the absence of the crop the weed was only temporarily reduced and with the suppression of the other weeds, particularly at Begbroke, it became completely dominant. However, in the presence of a crop, although its numbers were little affected, its vigour was reduced. This was so even at Culworth where although the growth of the wheat was relatively poor and non-competitive, the check to the Chrysanthemum segetum and other weeds on the treated plots was sufficient to allow the development of a greater number of heads of wheat.

These two experiments were designed as screening experiments and the results are tentative, but it does seem that 3,5-diiodo-4-hydroxybenzonitrile can give a very useful selective control of many annual broad leaf weeds in spring cereals at doses of 1-2 lb/ac. The control of Matricaria recutita is particularly useful, but Chrysanthemum segetum is only temporarily checked and satisfactory control seems unlikely.

Table I

Density and Stage of Growth of the Crops and Weeds at the time of treatment

Experiment No.	E/9/63				E/12/63			
Site	Begbroke				Culworth			
Date of application	22/5/63				15/5/63			
	Crop (spring barley)		No crop		Crop (spring wheat)		No crop	
Crop or Weed	Stage of growth	Density* No/sq.ft.	Stage of growth	Density* No/sq.ft.	Stage of growth	Density* No/sq.ft.	Stage of growth	Density* No/sq.ft.
Crop	5-5½ leaves	-	-	-	3 leaves	-	-	-
C. segetum	2-3 "	51	2½-3 leaves	43	3-5 "	24	3-5 leaves	32
M. recutita	5-9 "	105	4½-8 "	95	-	-	-	-
V. hederifolia	Cot.-2½ "	28	Cot.-2½ "	23	-	-	-	-
P. rhoeas	2-8 "	8	2-8 "	5	-	-	-	-
R. raphanistrum	2-5 "	19	2-4½ "	16	-	-	-	-
S. media	-		-		4-6 leaves	210	1-2 leaves	86
Soil type	Sandy loam				mod. heavy clay			
Temperature. Dry bulb	12.2°C				9.2°C			
Wet bulb	10.0°C				8.3°C			
First rain after treatment	28/5/63				19/5/63			

*Weeds counted on 13/5/63 at Begbroke on 6/5/63 at Culworth

Table II

The effect of 3,5-diiodo-4-hydroxybenzonitrile on the head density and straw length of Spring Cereals

(Figures are the mean of two replicates and are expressed as percentages of the adjacent controls)

Site	Begbroke		Culworth	
Crop	Spring barley (Pallas)		Spring wheat (Jufy)	
Date of assessment	5/7/63		3/7/63	
Dose of herbicide	Head counts	Straw length	Head counts	Straw length
1 lb/ac.	79	93	109	-
2 lb/ac.	97	100	113	-
4 lb/ac.	91	96	110	-
Control	100	100	100	-
Mean no./ft^2.	28	28 in.	27	-

Table III

The effect of 3,5-diiodo-4-hydroxybenzonitrile on the density of C. segetum, M. recutita and S. media

(Figures are the mean of two replicates and are expressed as percentages of the adjacent controls)

Site	Begbroke				Culworth			
Weed	C. segetum		M. recutita		C. segetum		S. media*	
Date of assessment	5/7/63		5/7/63		18/6/63		18/6/63	
Dose of herbicide	Crop	No crop	Crop	No crop	Crop	No crop	Crop	No crop
1 lb/ac.	99	133	4	43	95	104	80	81
2 lb/ac.	125	106	0	15	95	125	42	43
4 lb/ac.	89	66	0	2	60	89	44	51
Control	100	100	100	100	100	100	100	100
Mean no. of plants/ft^2.	7.9	9.4	12.5	15.9	19.6	28.9	> 200	

* The S. media figures are based on a mean of three independent scores for density per plot. The figures for the other weeds are actual plant counts.

Table IV

Visible effects on crops and weeds one week and one month after treatment

(Begbroke only).

Crop or Weed	One week after application Treatment			One month after application Treatment		
	1 lb/ac.	2 lb/ac.	4 lb/ac.	1 lb/ac.	2 lb/ac.	4 lb/ac.
Spring barley (Pallas)	Slight leaf tip scorch	Slight leaf scorch	Slight scorch	Apparently normal	Apparently normal	Slight apparent reduction
Chrysanthemum segetum	some chlorosis only slight scorch	increased chlorosis and scorch	severe chlorosis and scorch	Apparently normal but checked in crop.	Apparently normal checked in crop	Slightly checked checked in crop.
Matricaria recutita	severe scorch centres green	severe scorch apparently dead	severe scorch apparently dead	very reduced	almost absent	absent
Raphanus raphanistrum	severe scorch	severe scorch apparently dead	" "	very reduced	absent	absent
Stellaria media	severe scorch	severe scorch apparently dead	"	-	-	-
Papaver rhoeas	severe scorch	severe scorch apparently dead	" "	-	-	-
Veronica spp. (V. hederifolia & V. persica)	severe scorch	severe scorch apparently dead	"	-	-	-
Senecio vulgaris	absent	severe scorch apparently dead	absent	-	-	-

DISCUSSION

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3,5-diiodo-4-hydroxybenzonitrile has shown some interesting selectivities when applied to plants post-emergence. In the pot experiments the Gramineae tested showed a high degree of tolerance, as did members of the Papilionaceae to a lesser extent. At the other extreme, members of the Cruciferae and Compositae appeared particularly susceptible except for species such as kale and cabbage which have a very waxy leaf surface. In general the species whose foliage is normally reckoned to be difficult to wet were resistant, even though differences in retention with the formulation used did not appear outstanding. This resistance may be associated more with barriers to penetration than with the minimising of retention.

The almost complete resistance of the grasses to this herbicide at the doses tested indicates a number of potential uses. The use investigated most fully so far was for the control of annual weeds in temperate cereals. No significant adverse effects were produced on wheat or barley in either pot or field experiments or on oat in pot experiments (this cereal was not tested in the field). This applies to a range of stages of growth because early stages were used in the pot experiments while field applications were made in the tillering phase. The apparent resistance at early growth stages is particularly important as it may enable control of young annual weeds to be obtained early in the life of the crop.

This herbicide appears to have useful toxicity to a range of annual dicotyledonous weeds, some of which are difficult to control in cereals by established herbicides. Results on the mayweeds (Tripleurospermum and Matricaria spp.) were outstanding in both field and pot experiments as were those on Raphanus and Papaver. Other important cereal weeds controlled easily in pots or field plots were Chenopodium album, Senecio vulgaris and Sinapis alba, while Stellaria media and Veronica spp. received a very severe check. Information on Polygonum spp. suggests that they may be intermediate in response but more data are needed for this important group of weeds. Competition from a vigorous cereal crop would be likely to improve the control of many of these weeds still further. Chrysanthemum segetum was the only species to differ greatly in susceptibility between the pot and field experiments. A much higher mortality was achieved in the pot experiment than in the field. A possible explanation is that because of germination difficulties a cultivated ornamental selection of this species was used in the pot experiments. This selection is not readily distinguishable morphologically from the wild species, but its foliage does retain aqueous sprays more readily. This suggests some difference in the waxy surface coatings, or the cuticles of the two types. Accordingly the response of this species in the field experiments should be taken as more representative of the likely behaviour of this weed.

The only other dicotyledonous weed showing considerable resistance was Galium aparine. This species can be controlled in cereals by other herbicides which might be used separately or in mixture with the present benzonitrile. Other limitations are that grass weeds are likely to prove completely resistant

and, if the results on Rumex spp. are representative, little useful effect will be obtained on established perennial weeds. Finally it should be pointed out that all the experiments have been conducted under favourable environmental conditions. It remains to be seen whether weed control is so good when temperatures are low or rain quickly follows spraying.

The partial resistance of seedlings of lucerne and the clovers, coupled with the resistance of grass seedlings, suggests that there may be possibilities for the use of carefully chosen doses for the control of young plants of very susceptible weeds, such as the mayweeds and some Cruciferae, in undersown cereals, direct-sown lucerne and direct-sown ley mixtures.

Equally important possibilities are indicated for control of annual dicotyledonous weeds in:-

1) Sub-tropical and tropical grain crops such as maize, sorghum and rice.
2) Herbage grasses grown for seed production.
3) Grass crops grown for purposes other than seed production, such as sugar cane.

A primary disadvantage, however, particularly with tropical crops, will again be the inability of the herbicide to control grass and perennial weeds.

Peas are another important crop in which this herbicide has distinct possibilities. These experiments suggest that it is selective enough to control many weeds, including mayweeds which are an important problem in peas grown for freezing because of difficulties in separating flower heads from the peas. Other leguminous crops for which this herbicide might be useful are the various Phaseolus beans and groundnut.

Further crops possessing a useful degree of resistance, as indicated by the pot experiments, are onion and carrot. With the former a broad spectrum of weed control should be feasible, but the resistance of carrot is less and only control of a more limited range of weeds would be possible. Insofar as there are a number of other new herbicides showing promise for weed control in carrots the prospects for the use of 3,5-diiodo-4-hydroxybenzonitrile would appear less in carrot than in onion.

The results on the fruit crops, particularly blackcurrant, suggest the feasibility of directed spray applications around the base of bushes. Such a use might be possible in any woody perennial crop, temperate or tropical, where annual dicotyledonous weeds are a problem. The limitations are that care would be needed in many instances to avoid spray deposition on crop foliage, and grasses and perennials would not be controlled; also paraquat, an effective broad spectrum herbicide, is already available for this type of use in many situations.

Finally, the low activity on plants germinating in soil into which

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3,5-diiodo-4-hydroxybenzonitrile has been mixed suggests that the effect of herbicide residues in the soil on subsequent susceptible crops will not be a problem. On the other hand, it also means that this herbicide cannot be expected to control weeds germinating subsequent to its application, unless it is much more effective when left as a thin layer on the soil surface than when mixed in to the soil.

Recommendations for further research

1) A larger field experiment programme on the use of this herbicide for weed control in cereals is obviously justified. Experiments should be spread over a wide range of weed species to obtain information on tolerances and susceptibilities. Factors requiring detailed investigation in both pot and field experiments are:

a. the influence of stage of growth of both crop and weeds on their susceptibility or resistance

b. the effect of formulation on selectivity, bearing in mind that a liquid formulation has distinct practical advantages over a wettable powder; an attempt should also be made to see whether selectivity can be improved by the addition of a surface-active agent, particularly with reference to the control of species such as Chrysanthemum segetum

c. the effect of volume rate of spray fluid on selectivity

d. physico-chemical and biological compatibility with other herbicides which might be added to control Galium aparine or perennial weeds

2) Possibilities for selective use in undersown cereals and direct-sown legume-grass mixtures require separate detailed investigation in pots and in the field, particularly with respect to the importance of stage of growth of the legume at time of treatment and the factors governing its recovery from any damage

3) Preliminary field experiments on the use of this herbicide in any grass crop, temperate or tropical, are justifiable on the present evidence. Steps should be taken to bring it to the attention of people concerned with weed control in those crops

4) Preliminary field experiments should be encouraged or instigated to verify the other suggestions for selective uses made in the discussion, notably in peas, onions, Phaseolus beans and groundnuts

5) Because of the appreciable mammalian toxicity of this herbicide, crop residue studies in respect of the edible portions of crop should be encouraged at an early stage of experimentation

6) The present field experiments have been conducted under favourable environmental conditions. With a herbicide of this type which is relying almost completely on foliar entry, controlled experiments on speed of entry and the effect of temperature, humidity and rainfall after spraying are clearly desirable.

Figure 1. Results of a post-emergence application to a range of species in pots

The upper horizontal line of each pair = percentage survival of plants; the lower horizontal line = score on scale 0 - 7. A circle indicates that all plants died. The broken vertical line indicates the control level.

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