

1031083



PATENT SPECIFICATION

NO DRAWINGS

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Inventors: HOWARD JOHNSTON, MARY SADAKO TOMITA and ROBERT THOMAS MARTIN

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COMPLETE SPECIFICATION

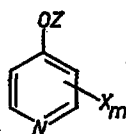
Compositions and method for Inhibiting Plant Growth

We, THE DOW CHEMICAL COMPANY, a Corporation organised and existing under the Laws of the State of Delaware, United States of America, of Midland, County of Midland, State of Michigan, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to compositions and methods for inhibiting plant growth. The invention further contemplates certain novel compounds.

Control of weeds is a continuing problem. Although the use of chemicals has greatly increased in recent years, they have not alleviated all the problems associated with weed control. Many materials accomplish top killing of foliage without killing the roots, from which subsequent regrowth often results in a more severe infestation than existed prior to treatment. Some materials are totally ineffective against seeds, while others may be effective immediately on application to soil but are readily de-activated under soil conditions. Still others have problems associated with handling which are of increasing significance in large scale operations.

It has now been found, according to the present invention, that germination and growth of undesirable vegetation can be suppressed, selectively if desired, by applying to plants, plant parts or to plant growth medium a polyhalopyridine compound having the formula



wherein m is 3 or 4, each X is a chlorine or

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bromine atom Z is $-H$, $-CH_3$, $-C_2H_4OC_2H_{2y+1}$, $-C_nH_{2n}OH$, $-M$, $-B$, $-E$ or $-C_nH_{2n}OE$; where n is an integer of from 2 to 4, y is an integer of from 1 to 4, M is sodium, potassium, zinc, manganese, magnesium, calcium, barium, aluminium, iron, chromium, nickel or cobalt, B is an amine salt group wherein the salt-forming base is ammonia, an alkylamine, an alkanolamine, an alkyleneamine, a heterocyclic amine or a quaternary ammonium base; E is an acyl, aryloxy-acyl, aroyl, aryl-acyl, hydroxycarbonyl, lower alkoxycarbonyl, lower alkylsulfonyl or arylsulfonyl radical, or a carbonic acid moiety or a nitrate, phosphate, phosphite, sulfite or sulfate group; and which organic radicals, i.e. those forming the ester can be further substituted with one or more chlorine or, bromine atoms or, hydroxyl, amino, or nitro groups.

Representative bases which form the nitrogen base salt group are mono- or polybasic amines; alkyl-, alkynyl-, alkenyl-, hydroxy-alkyl- or mixed alkyl-hydroxyalkylamines, aralkylamines, acyclic or homo- or heterocyclic amines; or ammonium or quaternary ammonium hydroxide. Typical suitable nitrogen bases include methylamine, ethylamine, isopropylamine, piperazine, 2,5-dimethylpiperazine, ammonium hydroxide, tetramethylammonium hydroxide, tetraethylammonium hydroxide, n -propylamine, n -butylamine, pentylamine, n -hexylamine, n -heptylamine, isooctylamine, trimethylamine, 1,3-dimethylbutylamine, diisopropylamine, N -methylisopropylamine, N -methyl-sec-butylamine, 2-aminopropyl alcohol, 3-amino- n -pentanol, isopropanolamine, 2-amino- n -pentanol, methylaminoethanol, ethylaminoethanol, isopropylaminoethanol, n -butylaminoethanol, bis(2-hydroxypropyl)amine, triethylamine, N,N -dimethyl- n -octylamine, tris(n -butyl)amine, ethanolamine, triisopropanolamine, triethanolamine, dimethylaminoethanol, diethylaminoethanol, di-butylaminoethanol, ethyldiethanolamine, di-

methyllamine, diethylamine, triisopropylamine, ethylenediamine, diethylenetriamine, triethylenetetramine, 1,2-propylenediamine, pyridine, α -, β -, and γ -picolines, 5 - ethyl - 2-methylpyridine, morpholine, di - n - propylamine, ethylethanolamine, aminoethylethanolamine, 2 - amino - 2 - methyl - 1 - propanol, diisopropanolamine, diethanolamine, tetraethylenepentamine, pentaethylenehexamine, cyclohexylamine, dicyclohexylamine, n-butyldiethanolamine, and dimethylaminoethanol.

Representative organic radicals forming ester groups, i.e., acyl, aryloxy-acyl, aroyl, aryl-acyl, lower alkoxy-carbonyl, lower alkylsulfonyl and arylsulfonyl which may be substituted with chlorine, bromine, hydroxy, amino or nitro and the carbonic acid moieties include acetyl, propionyl, butyryl, dimethylacetyl, chloroacetyl, 2,2 - dichloropropionyl, octanoyl, heptanoyl, trichloroacetyl, hexanoyl, stearoyl, 2,2,3 - trichloropropionyl, α - bromon - caproyl, α - bromoisobutyryl, α - bromoisovaleryl, (2,4 - dichlorophenoxy)acetyl, phenoxyacetyl, (2 - methyl - 4 - chlorophenoxy)acetyl, 2 - (2,4,5 - trichlorophenoxy)propionyl, (2,4,5 - trichlorophenoxy)acetyl, 2 - (2,4 - dichlorophenoxy)propionyl, 4 - (2,4 - dichlorophenoxy)butyryl, 4 - (2 - methyl - 4 - chlorophenoxy)butyryl, 2,3,6 - trichlorobenzoyl, 3 - amino - 2,5 - dichlorobenzoyl, 2,5 - dichloro - 3 - nitrobenzoyl, benzoyl, o-toluy, m-toluy, 2,6 - dichlorobenzoyl, o - chlorobenzoyl, m - chlorobenzoyl, p - toluy, 2,3 - dichlorobenzoyl, 2,4 - dinitrobenzoyl, p-nitrobenzoyl, p - chlorobenzoyl, 3,5 - dinitrobenzoyl, 2 - chloro - 2,5 - dinitrobenzoyl, phenylacetyl, (2,3,6 - trichlorophenyl)acetyl, (m - nitrophenyl)acetyl, (o - nitrophenyl)acetyl, (p - chlorophenyl)acetyl, (2,4 - dichlorophenyl)acetyl, 3 - phenylpropionyl, 4 - (2,4 - dichlorophenyl)butyryl, 3 - (p - chlorophenyl)propionyl, 4 - (o - chlorophenyl)butyryl, methylsulfonyl, ethylsulfonyl, n-octylsulfonyl, isopropylsulfonyl, n - amylsulfonyl, n - hexylsulfonyl, sec. - butylsulfonyl, phenylsulfonyl, p - tolylsulfonyl, 3,4 - xylylsulfonyl, p - chlorophenylsulfonyl, 2,4 - dichlorophenylsulfonyl, 2 - methyl - 4 - chlorophenylsulfonyl, salicyloyl, carbonate, bicarbonate, gluconate, citrate, and tartrate. Where the expression "lower" is employed in the present specification, it is meant to include up to 8 carbon atoms on the particular groups so qualified.

The preferred embodiments of the present invention are (1) methods for inhibiting plant growth which comprises applying to plants, plant parts e.g. foliage or to plant growth media a polyhalopyridine compound as above defined; and (2) compositions for inhibiting plant growth which contain as an active ingredient at least one polyhalopyridine compound as defined hereinbefore in admixture with an inert carrier comprising a finely di-

vided inert solid, an inert granular solid, a surface active dispersing agent, a polyglycol, beeswax, a paraffin wax, a nitrogen fertilizer, a potassium fertilizer, a phosphate fertilizer, ureaform, urea, or a metaphosphate. Particularly contemplated as desirable compositions are those which comprise at least one of the polyhalopyridine compounds as active ingredient in admixture with a finely divided inert solid, an inert granular solid, or a surface active dispersing agent.

The expression "surface active dispersing agent" as herein employed includes all agents which are capable of facilitating the dispersion of the toxicant in water or an organic solvent. The term thus includes solid emulsifying agents such as finely divided bentonite, pyrophyllite, Fuller's earth, attapulgite, and silica, as well as liquid and solid ionic and non-ionic wetting and dispersing agents, alkaline earth metal caseinates, alkyl aryl sulfonates sulfonated oils, complex organic ester derivatives, complex ether alcohols, condensation products of alkylene oxides with phenols or organic acids, polyoxyethylene derivatives of sorbitan esters, and mahogany soaps.

The term "finely divided inert solids" as herein employed refers to materials whose primary function is not as dispersant of the compounds in water or organic solvent but as carrier for dust composition. Such materials include chalk, talc, gypsum, and powdered walnut shells.

The term "inert granular solids" refers to mineral or other inert carriers which are suitable for dry application, e.g. corn cobs or sand, and differ from the "finely divided inert solids" principally in particle size.

According to the present invention, the polyhalopyridine compound can be compounded with any of the finely divided inert solids or solid surface active dispersing agents to form dust compositions by grinding, mixing or wetting the finely divided carrier with a solution of the toxicant in a volatile organic solvent. Dust mixtures can be employed either as treating compositions or as concentrates to be subsequently diluted with additional carrier. Dust concentrates compounded from liquid polyhalopyridine compounds generally contain from 1 to 30 percent by weight of the polyhalopyridine compound, while those compounded from solid polyhalopyridine compounds generally contain from 1 to 95 percent by weight of the toxicant.

The polyhalopyridine compounds can also be compounded with suitable water-miscible or water-immiscible organic liquids and surface active dispersing agents to produce liquid concentrates for further formulation with water and/or oil to prepare spray mixture in the form of aqueous dispersions or oil-in-water emulsion compositions. Preferred water-immiscible organic liquids include petroleum oil and distillates, toluene, xylene, cumene and

5 other aromatic hydrocarbon solvents, chlorinated aliphatic hydrocarbons, isoparaffin oil and other aliphatic hydrocarbon solvents. Water-miscible organic solvents include acetone, methyl ethyl ketone, cyclohexanone, alcohols, dimethylformamide, ether alcohols of ethylene glycol, and esters such as ethyl acetate, sec-butyl acetate or isopropyl acetate. When the hydroxypolyhalopyridine compounds are alkali metal, ammonium or amine salts, aqueous-concentrate compositions can be readily prepared. The salts can be first compounded in water-miscible organic solvent and added to a minimal amount of water or can be directly compounded in water or in a water-organic solvent mixture. Usually, the use of a procedure which includes addition of a small amount of a water-miscible organic solvent is preferred. Moreover, in the case of salt compositions, the salt need not be preformed but can be formed during the compounding process.

25 In the emulsifiable concentrates and other concentrate compositions described above, the polyhalopyridine compounds usually constitute from 1 to 95 percent by weight of the composition.

30 In a further embodiment of the present invention, the polyhalopyridine compositions can contain other plant growth modifying agents either as adjuvants or supplementary materials for both terrestrial and aquatic applications.

35 In carrying out the method of the present invention, the exact amount of the polyhalopyridine compound or composition thereof desirably administered varies with the type of growth inhibition to be achieved and whether the application is to be made to foliage or to soil or other growth medium. For example, in the treatment of grass weeds, soil application is preferred to foliage application and the amounts are governed thereby. Another factor is the plant species to be treated, as well as the presence or absence of desirable plants together with the undesirable species. Thus, selective grass control can be achieved by administration of sufficient polyhalopyridine compound to eradicate grasses without affecting broadleaves.

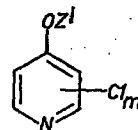
50 Effective control of terrestrial plants in soil can be achieved by the administration of as little as 0.001 p.p.m. (part by weight per million parts by weight of soil or growth medium). Except where selective control is desired, the upper limit is primarily a matter of economic practicability. Usually it is of no significant advantage to apply more than 80 pounds per acre (89.7 kg./hectare). Such administration provides a concentration of about 60 200 p.p.m. of halopyridine compound when distributed 30 mm. deep in the soil. In general, it has been found that where dangers from weathering and reseeding are secondary, good results can be obtained by the application of from 0.5 to 10 pounds per acre (0.56

to 11.2 kg./hectare). Under field conditions, the preferred range is from 1 to 30 pounds per acre (1.12 to 33.4 kg./hectare). Certain of the polyhalopyridine compounds can be employed for the selective control of grasses. For such control of grasses, it is preferred to provide the compounds in a proportion of from 0.2 to 50 p.p.m., while the preferred range for broadleaf control is from 25 p.p.m. to 200 p.p.m. distributed 30 mm. deep in the soil. In foliar applications, it is preferred that the foliage of plants be contacted with compositions containing from 1 ounce to 16 pounds of polyhalopyridine compound per 100 gallons (0.08 to 24 g./liter) of spray composition.

80 The application of the polyhalopyridine compounds or compositions thereof to plants or plant growth media can be carried out in any manner provided that an effective dosage is supplied. When the application is to be made by employing spray compositions, the concentration of the polyhalopyridine compound in the spray composition generally runs from 0.1 to 50 percent by weight or higher. In dust compositions, the concentration of the active ingredient can be from 1 to 85 per cent by weight.

95 The compositions of the invention can be applied to foliage or soil by conventional methods, e.g. with power dusters, boom and hand sprayers and spray dusters or from planes. For soil application, the modified or unmodified compositions can be mechanically mixed with the soil, applied to the surface of soil and thereafter mixed into the soil to a desired depth, or transported into the soil with a liquid carrier such as by injection, spraying or irrigation.

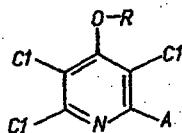
105 Especially preferred are the hydroxy polyhalopyridine compounds having the formula



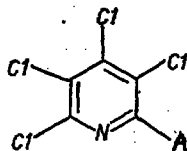
wherein Z' is $-H$, $-C_nH_{2n}OH$, $-M'$, $-B'$, $-E'$ and $C_nH_{2n}OE'$. In this formula m is 3 or 4; n is 2 to 4 inclusive, M' is sodium, potassium, zinc, manganese, magnesium, calcium, barium, aluminium, iron, chromium, nickel or cobalt; B' is ammonia or an alkanol- or an alkylamine containing from 1 to 18 carbon atoms, inclusive, E' is an acyl radical from 1 to 18 carbon atoms, an aroyl radical from 1 to 8 carbon atoms, an alkylsulfonyl radical from 1 to 3 carbon atoms, an arylsulfonyl radical from 1 to 8 carbon atoms, hydroxycarbonyl, sulfate, phosphate or nitrate.

120 The present invention further contemplates as novel compounds those agents of the invention which are 4-methoxy- or 4-(2-alkoxyethoxy)-polyhalopyridines wherein each X

in the foregoing formula represents a chlorine atom. These compounds, accordingly, have the formula



- 5 wherein A is hydrogen or a chlorine atom, and R is $-\text{CH}_3$ or $-\text{C}_2\text{H}_4\text{OC}_y\text{H}_{2y+1}$ where y is an integer from 1 to 4. Examples of the new compounds include 4 - methoxy - 2,3,5 - trichloropyridine, 4 - methoxy - 2,3,5,6 - tetrachloropyridine, 4 - (2,n - butoxyethoxy) - 2,3,5 - trichloropyridine, 4 - (2 - methoxyethoxy) - 2,3,5 - trichloropyridine, 4 - (2 - methoxyethoxy) - 2,3,5,6 - tetrachloropyridine, and 4 - (2,n - butoxyethoxy) - 2,3,5,6 - tetrachloropyridine. The 4-methoxy- compounds are white crystalline solids, and the alkoxyethoxy compounds are generally colorless or light colored solids or liquids. The compounds are generally soluble in organic solvents and substantially insoluble in water. The compounds of the present invention can be prepared by mixing together and reacting sodium methoxide or sodium 2-alkoxyethoxide and an appropriate polychloropyridine compound having the formula



- 30 wherein A is as defined above. Preferably the reactants are employed in substantially equimolecular proportions and are brought together in a solvent such as methanol. The reaction can be conveniently carried out at temperatures from ambient temperature to reflux temperature.

The following Examples illustrate the invention.

EXAMPLE 1

A concentrate composition was prepared by dissolving 58 milligrams of 2,3,5,6 - tetrachloro - 4 - pyridinol in 2-milliliters of acetone containinug 4 percent by volume of a polypropylene glycol oleate emulsifier (M.W. 700-800) and the resulting solution diluted to 24 milliliters with water containing 0.2 percent by volume of isoctylphenyl polyethoxyethanol wetting agent. The concentrate composition was diluted to prepare spray compositions containing 32, 16, 8 and 4 ounces (2.3, 1.2, 0.6 and 0.3 g./l.) of 2,3,5,6-tetrachloro - 4 - pyridinol per 100 gallons. In separate operations, the spray compositions were applied to the foliage of oat, cucumber and crab grass plants ranging from 4 to 6 inches (100-150 mm.) in height in three replications. The treated plants as well as untreated checks were maintained in a green house for 24 days and thereafter observed to determine the extent of kill of the plant species. The results indicated that at each spray concentration in all three replications of each plant species, the plants were dead whereas the check plants showed good continued growth.

EXAMPLE 2

In operations carried out in a manner similar to that described in Example 1, spray compositions containing 2,3,5-trichloro-4-pyridinol were prepared in concentrations of 32, 16 and 8 ounces per 100 gallons (2.3, 1.2, and 0.6 g./l.). The compositions were treated with aqueous sodium hydroxide to pH 7 to obtain sodium salt compositions of 2,3,5-trichloro-4-pyridinol; the resulting compositions were applied in three replications to the foliage of Johnson grass, crabgrass and water grass. Seventeen days after application, the plants were examined and compared to untreated ~~creek~~ plants to determine the extent of kill. The results expressed as average of three replications are given in the following table.

TABLE I

Rate		Percent Kill		
g./l.	Oz./100 gals.	Johnson grass	Crab-grass	Water Grass
2.3	32	98	100	98
1.2	16	97	100	98
0.6	8	97	100	95

EXAMPLE 3

40 Milligrams of 2,3,5 - trichloro - 4 - pyridinol was dissolved in 2 milliliters of acetone containing 4 percent by volume of a polypropylene glycol oleate non-ionic emulsifier, m.w. 700—800, and extended to 4 milliliters by the addition of water to produce a concentrate composition containing 10 milligrams of 2,3,5 - trichloro - 4 - pyridinol per milliliter of composition. The concentrate composition was diluted with water to obtain treating compositions of varying concentrations which were applied to the soil surface of pots having a surface area of 6.9 square inches (44.5 sq.cm.) per pot and containing from about 190 to 200 grams of air-dried soil. Thus, for example, a 1-milliliter portion of the concen-

trate composition was added to 29 milliliters of water and the resulting treating composition employed to drench the soil contained in a single pot, providing a treating rate of 10 milligrams per pot or about 20 pounds per acre (22.2 kg./hectare). Appropriate dilutions were then made and the diluted compositions applied to soil to provide treatment rates of 20, 10, 5 and 2.5 pounds per acre (22.2, 11.2, 5.6 and 2.8 kg./hectare). The process was carried out in duplicate and the pots so treated as well as check pots were then seeded with various plant species, capped with about 35—90 grams of sand, watered as necessary to maintain soil moisture and observed twenty-one days after planting. The results are set forth in Table II.

TABLE II

Percent Kill or Growth Reduction

Plant Species	kg./hectare			
	3.6	1.8	0.9	0.45
Bush beans	100	100	99	95
Carrot	100	100	100	100
Tomato	100	100	100	100
Cotton	100	99	90	90
Cucumber	90	90	90	90
Radish	95	95	90	90
Corn	100	100	100	100
Water Grass	100	100	100	99
Johnson Grass	99	99	99	99
Wheat	100	100	100	100

EXAMPLE 4

In an operation similar to that described in Example 3, 2,3,5 - trichloro - 4 - pyridinol was employed in treating soil seeded with

different species of plants and at additional concentrations. The results are set forth in Table III.

TABLE III

Rate		Percent Kill or Growth Reduction	
Lbs./acre	kg./hectare	Crabgrass	Oats
20	22.2	100	100
10	11.2	100	99
5	5.6	100	100
2.5	2.8	100	99
1.25	1.4	100	99
0.62	0.695	100	99
0.31	0.347	90	99
0.16	0.179	90	90
0.078	0.0875	90	90
0.039	0.0436	90	90
0.019	0.0213	90	90
0.0098	0.01	90	90

EXAMPLE 5

In separate operations carried out in a manner similar to that described in Examples 3 and 4, herbicidal effectiveness in soil was de-

termined for other hydroxy polyhalopyridine compounds. The results are set forth in Table IV.

TABLE IV

Compound	kg./hectare	Soil seeded to	Results
2,3,5,6-Tetrachloro-4-pyridinol	22.2, 11.2, 5.6, 2.8 and 0.695	Carrot, tomato, oats	Complete control at all rates
2,3,5,6-Tetrachloro-4-pyridyl acetate	44.3, 22.2, 11.2, 5.6, 2.8, 1.4	Oats	Complete control at all rates
2-(2,3,5-Tetrachloro-4-pyridyloxy)-ethanol	44.3, 22.2, 11.2, 5.6, and 2.8	Crabgrass, cucumber, oats	Complete control at all rates
Tris-2-(2,3,5-trichloro-4-pyridyloxy)-ethyl phosphite	11.2	Cucumber, oats	Complete control
2-(2,3,5-Trichloro-4-pyridyloxy)ethyl propionate	11.2	Cucumber, oats	Complete control
Bis-(2,3,5-trichloro-4-pyridyl phthalate	11.2	Oats	Complete control
3,5-Dibromo-2,6-dichloro-4-pyridinol	11.2	Oats	Complete control
2-Bromo-3,5-dichloro-4-pyridinol	11.2	Oats	Complete control
2,3,5-trichloro-4-pyridyl acetate	44.3, 11.2, 5.6, 2.8	Crabgrass, oats, radishes	Complete control at all rates

EXAMPLE 6

In separate operations carried out in a manner similar to that described in Example 1,

herbicidal effectiveness on foliage was determined for other hydroxy polyhalopyridine compounds as indicated below in Table V.

5

TABLE V

Compound	g./l.	Plants	Results
2,3,5,6-Tetrachloro-4-pyridinol	2.3, 1.2, and 0.6	Radishes, beans, cucumbers	Complete kill at all concentrations
2,3,5-Trichloro-4-pyridinol	2.3, 1.2, and 0.6	Radishes	"
2,3,5,6-Tetrachloro-4-pyridinol, sodium salt	2.3, 0.6, 0.3	Cucumbers	Complete kill
ammonium salt	"	"	"
triethylamine salt	"	"	"

EXAMPLE 7

10 4-METHOXY-2,3,5,6-TETRACHLORO PYRIDINE

0.92 Gram (0.04 gram atom) of metallic sodium was added in small pieces to 100 milliliters of dry methanol, with stirring, forming

sodium methoxide which remained dissolved in the excess methanol. To this was added, while stirring, 10 grams (0.04 mole) of pentachloropyridine and the mixture then heated to reflux temperature and heated under reflux for about 8 hours. The sodium chloride 15 20

by-product which formed was removed by filtration; the filtrate was allowed to cool and stand overnight whereupon the desired product precipitated. The product was recovered by filtration and recrystallized from hexane to obtain the purified product as a white crystal-

line solid having a melting point of 107°—109°C. The yield of the product was 7.3 grams or 74.5 percent of theoretical. The product had elemental analyses (in percent) as follows:

	Carbon	Hydrogen	Nitrogen	Chlorine
Theory	29.1	1.2	5.7	57.5
Found	29.26	1.3	5.6	57.4

EXAMPLE 8 4-METHOXY-2,3,5-TRICHLORO PYRIDINE

In a manner similar to that described in Example 7, 0.85 gram (0.037 gram atom) of metallic sodium was added in small pieces to 60 milliliters of dry methanol to obtain sodium methoxide which remained in solution. To this was added, while stirring, 8.0 grams (0.037 mole) of 2,3,4,5 - tetrachloropyridine

and the mixture heated at reflux temperature for about 4 hours. The sodium chloride by-product was removed by filtration and the filtrate subjected to reduced pressure to vaporize off the solvent. The product was recrystallized from hexane to obtain a purified product having a melting point of 60°—64°C. The product had elemental analyses (in percent) as follows:

	Carbon	Hydrogen	Nitrogen	Chlorine
Theory	34	1.88	6.57	50.1
Found	33.9	2.07	6.39	49.88

EXAMPLE 9 4-(2-n-BUTOXYETHOXY)-2,3,5- TRICHLOROPYRIDINE

1.3 Grams (0.057 gram atom) of sodium was added slowly in small portions to 60 grams (0.51 mole) of 2-n-butoxyethanol while the temperature was maintained from about 25° to 35°C. To the resulting mixture of sodium 2 - n - butoxyethoxide in 2 - n - butoxyethanol was added 10.9 grams (0.05 mole) of 2,3,4,5 - tetrachloropyridine and the resulting mixture heated at about 70°C. for about four hours. The reaction mixture was then

poured into ice-water and the resulting mixture extracted successively with methylene dichloride and ether to effect a separation between the organic materials and the sodium chloride by-product which remained in the aqueous phase. The combined extracts were dried and then subjected to distillation operations which were completed at 168°C. and 3 millimeters of mercury pressure to recover 4 - (2 - n - butoxyethoxy) - 2,3,5 - trichloropyridine as a light orange oil. The product had elemental analyses (in percent) as follows:

	Carbon	Hydrogen	Chlorine
Theory	44.2	4.68	35.8
Found	44.24	4.53	35.72

4 - (2 - Methoxyethoxy) - 2,3,5 - trichloropyridine prepared in a manner similar to that described above was a colorless liquid having a boiling point of 133—135°C./1.0 mm. Hg.

EXAMPLE 10 4 - (2 - METHOXYETHOXY) - 2,3,5,6- TETRACHLOROPYRIDINE

Sodium 2-methoxyethoxide was prepared by reacting 2.3 grams (0.10 gram atom) of sodium with excess (86 grams) 2 - methoxy-

ethanol. To the resulting mixture was added 23 grams (0.092 mole) of pentachloropyridine and the resulting mixture heated in the temperature range of from 90°C. to 100°C. for about four hours. The reaction mixture was then filtered to remove sodium chloride by-product and the filtrate was heated to a temperature of 150°C. and 2 millimeters of mercury pressure to distill off 2-methoxyethanol. The residue was thereafter washed with water and extracted into hexane. The hexane solu-

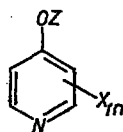
tion was dried and the solvent vaporized off to recover an oil from which, on standing, the desired 4 - (2 - methoxyethoxy) - 2,3,5,6-tetrachloropyridine crystallized out. The cry-

stals were filtered off and recrystallized from hexane to obtain a purified product having a melting point of 51°-52°C. The product had elemental analyses in percent as follows:

	Carbon	Hydrogen	Chlorine
Theory	33.1	2.41	46.6
Found	33.32	2.62	48.6

10 WHAT WE CLAIM IS:—

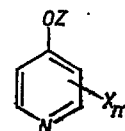
1. Herbicidal compositions comprising at least one polyhalopyridine compound having the formula



15 wherein m is 3 or 4; each X is a bromine or chlorine atom; Z is —H, —C_nH_{2n}OH, —CH₃, —C₂H₄OC_yH_{2y+1}, —M, —B, —E or —C_nH_{2n}OE; where n is an integer of from 2 to 4, y is an integer from 1 to 4, M is sodium, 20 potassium, zinc, manganese, magnesium, calcium, barium, aluminium, iron, chromium, nickel or cobalt, B is an amine salt group wherein the salt-forming base is ammonia, an alkylamine, an alkanolamine, an alkylene- 25 amine, a heterocyclic amine, or a quaternary ammonium base, E is an acyl, aryloxy-acyl, aroyl, aryl-acyl, lower alkoxy-carbonyl, hydroxycarbonyl, lower alkylsulfonyl or arylsulfonyl radical or a carbonic acid moiety, or 30 a nitrate, phosphate, phosphite, sulfite or sulfate group; and which organic radicals represented by E can be substituted with one or more chlorine or bromine atoms or hydroxyl, amino, or nitro groups, in admixture with an 35 inert carrier comprising a finely divided solid, as hereinbefore defined, a surface active dispersing agent as hereinbefore defined, an inert granular solid, as hereinbefore defined, a polyglycol, beeswax, a paraffin wax, a nitrogen fertilizer, a potassium fertilizer a phosphate 40 fertilizer, ureaform, urea or a metaphosphate.

2. Compositions in accordance with Claim 1, characterized in that the polyhalopyridine compound is 2,3,5,6, - tetrachloro - 4 - pyridinol, 2,3,5 - trichloro - 4 - pyridinol, 2- 45 (2,3,5,6 - tetrachloro - 4 - pyridoxy)ethanol, 2 - (2,3,5 - trichloro - 4 - pyridoxy)ethanol, 2,3,6 - trichloro - 4 - pyridinol, 2,3,5,6-tetrachloro - 4 - pyridyl acetate, or 2,3,5- 50 trichloro - 4 - pyridyl acetate.

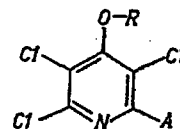
3. Method for inhibiting plant growth which comprises applying to plants, plant parts or to plant growth medium a polyhalopyridine compound having the formula



wherein m is 3 or 4; each X is a bromine or chlorine atom; Z is —H, —C_nH_{2n}OH, —CH₃, —C₂H₄OC_yH_{2y+1}, —M, —B, —E or —C_nH_{2n}OE; where n is an integer of from 2 to 4, y is an integer of from 1 to 4, M is sodium, potassium, zinc, manganese, magnesium, calcium, barium, aluminium, iron, chromium, nickel or cobalt, B is an amine salt group wherein the salt forming base is ammonia, an alkylamine, an alkanolamine, an alkylene- 60 amine, a heterocyclic amine, or a quaternary ammonium base; E is an acyl, aryloxy-acyl, aroyl, aryl-acyl, lower alkoxy-carbonyl, hydroxycarbonyl, lower alkylsulfonyl or arylsulfonyl radical, a carbonic acid moiety, or a nitrate, phosphate, phosphite, sulfite or sulfate group; and which organic radicals represented by E can be substituted with one or more chlorine or bromine atoms or hydroxyl, amino, or nitro groups. 75

4. Method in accordance with Claim 3 characterized in that the polyhalopyridine compound is 2,3,5,6 - tetrachloro - 4 - pyridinol, 2,3,5 - trichloro - 4 - pyridinol, 2- 80 (2,3,5,6 - tetrachloro - 4 - pyridoxy)ethanol, 2 - (2,3,5 - trichloro - 4 - pyridoxy)ethanol, 2,3,6 - trichloro - 4 - pyridinol, 2,3,5,6-tetrachloro - 4 - pyridyl acetate, or 2,3,5-trichloro - 4 - pyridyl acetate. 85

5. A compound of the general formula



wherein A is a hydrogen or chlorine atom, and R is —CH₃ or —C₂H₄OC_yH_{2y+1} where y is an integer from 1 to 4. 90

6. 4-Methoxy-2,3,5-trichloropyridine.

7. 4-Methoxy-2,3,5,6-tetrachloropyridine.

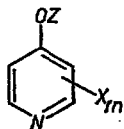
tion was dried and the solvent vaporized off to recover an oil from which, on standing, the desired 4 - (2 - methoxyethoxy) - 2,3,5,6-tetrachloropyridine crystallized out. The cry-

stals were filtered off and recrystallized from hexane to obtain a purified product having a melting point of 51°—52°C. The product had elemental analyses in percent as follows:

	Carbon	Hydrogen	Chlorine
Theory	33.1	2.41	46.6
Found	33.32	2.62	48.6

10 WHAT WE CLAIM IS:—

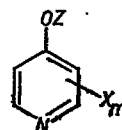
1. Herbicidal compositions comprising at least one polyhalopyridine compound having the formula



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2. Compositions in accordance with Claim 1, characterized in that the polyhalopyridine compound is 2,3,5,6, - tetrachloro - 4 - pyridinol, 2,3,5 - trichloro - 4 - pyridinol, 2- 45 (2,3,5,6 - tetrachloro - 4 - pyridoxy)ethanol, 2 - (2,3,5 - trichloro - 4 - pyridoxy)ethanol, 2,3,6 - trichloro - 4 - pyridinol, 2,3,5,6-tetrachloro - 4 - pyridyl acetate, or 2,3,5-trichloro - 4 - pyridyl acetate.

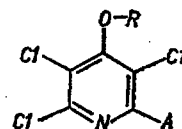
3. Method for inhibiting plant growth which comprises applying to plants, plant parts or to plant growth medium a polyhalopyridine compound having the formula



wherein m is 3 or 4; each X is a bromine or chlorine atom; Z is —H, —C_nH_{2n}OH, —CH₃, —C₂H₄OC_yH_{2y+1}, —M, —B, —E or —C_nH_{2n}OE; where n is an integer of from 2 to 4, y is an integer of from 1 to 4, M is sodium, potassium, zinc, manganese, magnesium, calcium, barium, aluminium, iron, chromium, nickel or cobalt, B is an amine salt group wherein the salt forming base is ammonia, an alkylamine, an alkanolamine, an alkylene-amine, a heterocyclic amine, or a quaternary ammonium base; E is an acyl, aryloxy-acyl, aroyl, aryl-acyl, lower alkoxycarbonyl, hydroxycarbonyl, lower alkylsulfonyl or aryl-sulfonyl radical, a carbonic acid moiety, or a nitrate, phosphate, phosphite, sulfite or sulfate group; and which organic radicals represented by E can be substituted with one or more chlorine or bromine atoms or hydroxyl, amino, or nitro groups.

4. Method in accordance with Claim 3 characterized in that the polyhalopyridine compound is 2,3,5,6 - tetrachloro - 4 - pyridinol, 2,3,5 - trichloro - 4 - pyridinol, 2- (2,3,5,6 - tetrachloro - 4 - pyridoxy)ethanol, 2 - (2,3,5 - trichloro - 4 - pyridoxy) - ethanol, 2,3,6 - trichloro - 4 - pyridinol, 2,3,5,6-tetrachloro - 4 - pyridyl acetate, or 2,3,5-trichloro - 4 - pyridyl acetate.

5. A compound of the general formula



wherein A is a hydrogen or chlorine atom, and R is —CH₃ or —C₂H₄OC_yH_{2y+1} where y is an integer from 1 to 4.

6. 4-Methoxy-2,3,5-trichloropyridine.

7. 4-Methoxy-2,3,5,6-tetrachloropyridine.



8. 4 - (2 - n - Butoxyethoxy) - 2,3,5 - tri-chloropyridine.
9. 4 - (2 - Methoxyethoxy) - 2,3,5 - tri-chloropyridine.
5 10. 4 - (2 - Methoxyethoxy) - 2,3,5,6-tetrachloropyridine.
11. 4 - (2 - n - Butoxyethoxy) - 2,3,5,6-tetrachloro - pyridine.
12. Compositions for the inhibition of
10 plant growth substantially as described with reference to any one of Examples 1 to 6.
13. Method for inhibiting plant growth substantially as described with reference to any one of the Examples 1 to 6.

BOULT, WADE & TENNANT, ^{RA}
111 & 112, Hatton Garden, London E.C.1.
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Agents for the Applicants.

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