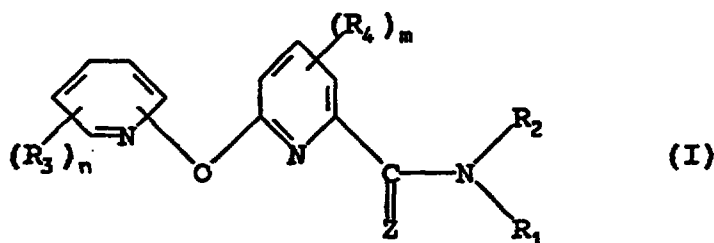


T 952 FF

HERBICIDAL PICOLINAMIDE DERIVATIVES

The present invention relates to certain new picolinamide derivatives, their preparation, herbicidal compositions containing them, and their use in combating undesired plant growth.

- 5 In accordance with the present invention there is provided a compound of the general formula:



- 10 wherein R_1 and R_2 each independently represents a hydrogen atom, or an alkyl, aryl, alkenyl, alkynyl, cycloalkyl, cycloalkylalkyl, aralkyl, alkaryl, hydroxy, alkoxy, alkenyloxy, alkynyloxy, alkylcarbonyl, alkoxy carbonyl, amino, mono- or di-alkylamino,
- 15 alkoxy carbonylamino group, in which groups any alkyl, alkenyl, alkynyl, cycloalkyl or aryl moiety is optionally substituted, an optionally substituted heterocyclic group, an arylamino group optionally substituted by a halogen atom, a dialkylcarbamoyl
- 20 group, a trialkylsilyl group or a fused ring structure, or together form an alkenylene chain or an optionally substituted alkylene chain which is optionally interrupted by an oxygen or sulphur atom or by a group -NR- in which R represents a hydrogen atom or an alkyl
- 25 group; R_3 represents a hydrogen or halogen atom, or an



alkyl, alkoxy, aryl or aryloxy group optionally substituted by one or more of the same or different substituents selected from halogen atoms and cyano, hydroxy and alkoxy groups, or represents a cyano, carboxy, alkoxycarbonyl, (alkylthio)carbonyl, alkylcarbonyl, amido, alkylamido, nitro, alkenyloxy, alkynyloxy, alkylthio, haloalkylthio, alkenylthio, alkynylthio, alkylsulphinyl, alkylsulphonyl, alkyloximinoalkyl or alkenyloximinoalkyl group; R_4 represents a halogen atom or an alkyl or haloalkyl group; Z represents an oxygen or sulphur atom; m is 0-3; and n is 1-4.

Where substituted, any of the alkyl, alkenyl, alkynyl, cycloalkyl or aryl moieties in R_1 and R_2 may be substituted by one or more of the same or different substituents selected from halogen atoms and hydroxy, cyano, alkyl, alkoxy, alkylthio, alkoxycarbonyl, aryl, aryloxy, and mono- or di-alkylamino groups.

When any of the substituents R_1 , R_2 , R_3 and R_4 represents or contains an alkyl, alkenyl or alkynyl substituent group, this may be linear or branched and suitably has up to 12, preferably up to 6, carbon atoms. Preferred alkyl groups are methyl, ethyl, n-propyl, i-propyl, n-butyl, i-butyl, s-butyl and t-butyl. Where a cycloalkyl substituent group is present, this suitably has from 3 to 6 carbon ring atoms. An aryl group is suitably a phenyl group. An alkenylene or alkylene chain suitably has 3 to 6 chain members. A halogen atom suitably denotes a fluorine, chlorine or bromine atom; preferred haloalkyl groups are fluoroethyl, chloroethyl, trifluoromethyl and trifluoroethyl groups.

The substituent(s) R_3 may be at any free position or combination of positions on the pyridyloxy ring.

Each substituent R_3 is preferably a hydrogen,

chlorine or bromine atom, or a methyl or trifluoromethyl group.

Preferably m is 0 and n is 1-2.

Preferably Z represents an oxygen atom.

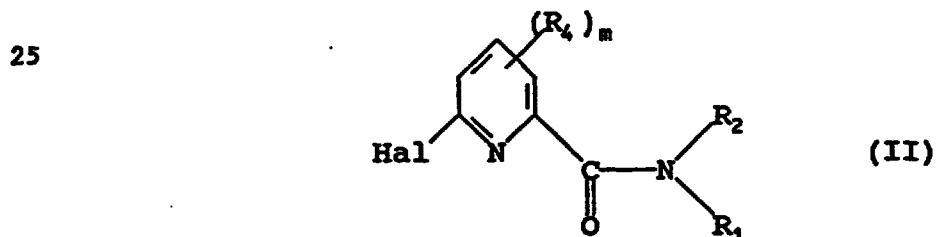
5 The substituents R_1 and R_2 when individually present may be the same or different. Preferably R_1 represents an unsubstituted C_{1-6} alkyl group, a C_{1-5} alkyl group substituted by an alkoxy, aryloxy or cyano group or by one or more halogen atoms, for example a
10 trifluoroethyl group, a C_{1-6} alkoxy group optionally aryl-substituted, a C_{2-7} alkynyl group, a C_{2-6} alkenyl group, a C_{2-6} alkenyloxy group, an unsubstituted aryl group, especially a phenyl group, a substituted aryl group, especially a phenyl group substituted by one or
15 more halogen atoms, an aralkyl group, especially a benzyl or phenethyl group, an alkaryl group, especially a tolyl group, a cycloalkyl group, especially a cyclopropyl or cyclopentyl group, a substituted cycloalkylalkyl group, a heterocyclic group, especially
20 a furfuryl or substituted isoxazolyl group, a dialkylaminoalkyl group, a naphthyl group or a trimethylsilyl group, while R_2 represents a hydrogen atom or an optionally substituted C_{1-6} alkyl group, especially a methyl, ethyl, n-propyl, i-propyl, i-butyl
25 or cyanomethyl group, and R_3 represents a hydrogen or halogen atom or a C_{1-6} alkyl or C_{1-6} haloalkyl group.

Particularly preferred are compounds in which R_1 represents a methyl, ethyl, n-propyl, i-propyl, dimethylpropyl, n-butyl, i-butyl, s-butyl,
30 t-butyl, methoxy, ethoxy, t-butoxy, methoxyethyl, cyanomethyl, fluoroethyl, chloroethyl, trifluoroethyl, allyl, allyloxy, dimethylaminoethyl, propargyl, dimethylpropargyl, diethylpropargyl, phenethyl, pivalyl, furfuryl, naphthyl, methylphenoxyethyl,
35 trimethylsilyl, t-butyl-isoxazolyl, cyclopropyl,

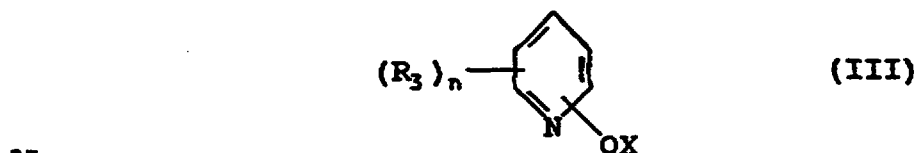
cyclopentyl, unsubstituted or monofluoro-, difluoro- or monochloro-substituted phenyl, benzyl, fluorobenzyl, benzyloxy, tolyl, or dichloro-substituted cyclopropylmethyl group, R_2 represents a hydrogen atom or a methyl, ethyl, n-propyl, i-propyl, i-butyl or cyanomethyl group, and R_3 represents a hydrogen, chlorine or bromine atom or a methyl or trifluoromethyl group.

When R_1 and R_2 together represent an alkylene chain, suitably the chain consists of 4 or 5 chain atoms, and is for example a group $-(CH_2)_4-$, $-(CH_2)_5-$, $-C(CH_3)-(CH_2)_4-$, $-CH_2-C(CH_3)-(CH_2)_3-$, $-(CH_2)_2-C(CH_3)-(CH_2)_2-$, $-(CH_2)_2O(CH_2)_2-$ or $(CH_2)_2NR(CH_2)_2-$ in which R is a C_{1-2} alkyl group, suitably methyl. When R_1 and R_2 together represent an alkenylene chain, suitably the chain consists of 4 or 5 chain atoms, and is for example a group $-(CH_2)_2-CH=CH-CH_2-$.

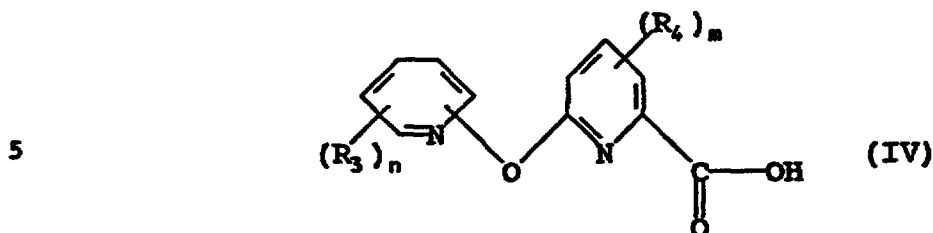
In accordance with the present invention there is also provided a process for the preparation of a compound of the general formula I, in which Z represents an oxygen atom, which comprises either (A) reacting a compound of the general formula:



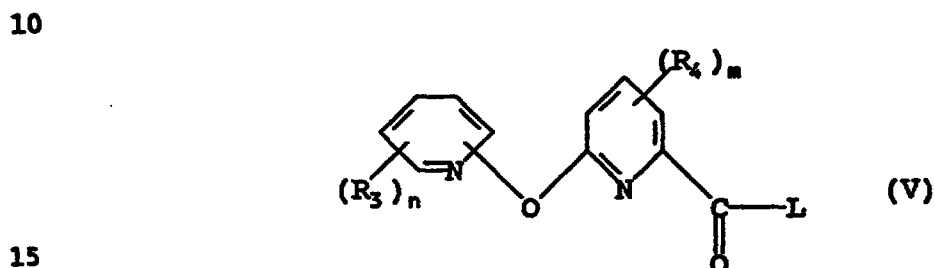
30 with a compound of the general formula:



or (B) by converting a compound of the general formula:



into a derivative of the general formula:



which is then reacted with an amine of the general
formula NHR_1R_2 , wherein R_1 , R_2 , R_3 , R_4 , m and n are as
20 defined above, Hal represents a halogen atom, X
represents a hydrogen atom or an alkali metal atom, and
L is a leaving group, with the optional further step of
converting the resultant compound of the general
formula I into a further compound of the general
25 formula I.

A leaving group is any group that will, under the
reaction conditions, cleave from the starting material
thus promoting reaction at a specific site.

30 The leaving group L may suitably be a halogen
atom, for example a bromine or, especially, a chlorine
atom, an alkoxy group, suitably C_{1-4} alkoxy, especially
methoxy, or an imidazole group.

The conversion of a resultant compound of the
general formula I into a further compound of the
35 general formula I may suitably be such as to replace a

group of R_1 and/or R_2 by a different group. For example, when R_1 and/or R_2 represents a hydrogen atom it may be replaced by an alkyl group, suitably by reaction with an alkyl halide.

5 Suitably, Hal represents a bromine atom or, especially, a chlorine atom. Suitably, X represents a hydrogen atom.

 When X represents an alkali metal atom, it is suitably a potassium or, especially, a sodium atom.

10 The process is suitably carried out in the presence of an inert organic solvent, for example dimethylformamide or dimethylsulphoxide, or an aromatic hydrocarbon, for example benzene or toluene, or a halogenated hydrocarbon, for example dichloromethane,
15 or an ether, for example diethyl ether, or an ester, for example ethyl acetate.

 The process is suitably carried out at a temperature in the range of 0 to 100°C, preferably at the reflux temperature of the reaction mixture, and
20 suitably in the presence of a base, for example potassium hydroxide, and a copper catalyst, such as cuprous chloride.

 Suitably, the reaction is carried out using substantially equimolar amounts of the reactants.
25 However, it can be expedient to use one reactant in excess.

 Compounds of the general formula I wherein Z represents a sulphur atom are suitably prepared by reaction of a compound of the general formula I wherein
30 Z represents an oxygen atom, with phosphorous pentasulphide under standard conditions, for example by heating, suitably under reflux, in the presence of an inert aromatic solvent, for example benzene, toluene, pyridine or quinoline.

35

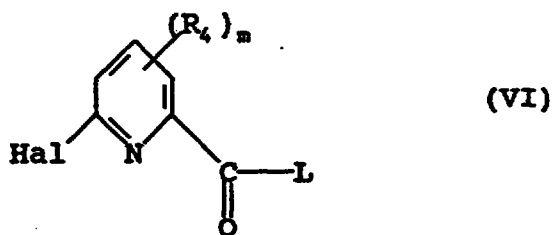
The compounds of the present invention may be isolated and purified by conventional techniques, for example by solvent extraction, evaporation followed by recrystallisation or by chromatography on silica.

5 The compounds of formula III are either known or can be prepared by conventional techniques.

When X represents an alkali metal atom, the process of method (A) may be carried out by preparation of the alkali metal pyridinolite from the corresponding
10 pyridinol using an alkali metal alkoxide, such as sodium methoxide, followed by treatment of the pyridinolite with a substantially equimolar amount of compound II, suitably at an elevated temperature, for example under reflux, with a copper catalyst, such as
15 cuprous chloride, in pyridine in the presence of an aromatic hydrocarbon, such as xylene, as is described in GB-A-2050168, or in dimethylformamide or dimethylsulphoxide.

Compounds of general formula II may suitably be
20 prepared by reacting an amine of general formula NHR_1R_2 , in which R_1 and R_2 are as defined above, with a 2-halo-6-pyridine carboxylic acid derivative of the general formula:

25



30

in which R_4 , m and Hal are as defined above and L is a leaving group. The leaving group L is as defined above.

35

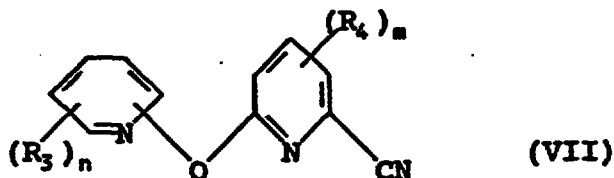
This reaction is suitably carried out in the presence of an inert organic solvent, for example dimethylformamide or acetonitrile, or an aromatic hydrocarbon, for example benzene or toluene, or a
5 halogenated hydrocarbon, for example dichloromethane or an ether, for example diethyl ether, or an ester, for example ethyl acetate; suitably at a temperature in the range of from 0 to 100°C. Suitably, this reaction is carried out using substantially equimolar amounts of
10 the reactants. However, it can be expedient to use one reactant in excess, conveniently the amine.

Compounds of formula VI may be prepared from corresponding substituted picolinic acids by standard methods for the preparation of, for example, esters,
15 using, for example, alcohols and acid catalysts or thionyl chloride, or of acid chlorides and acid bromides, using, for example, thionyl chloride or thionyl bromide, or of imidazole derivatives, using, for example, carbonyl diimidazole. The acid compounds
20 themselves can be prepared by standard methods from chloropicolinic acid or ester thereof. Chloropicolinic acid, or ester thereof, may be prepared by the methods described in J. Pharm. Belg. (1980), 35 1, 5-11.

The substituted amines NHR_1R_2 are either known or
25 can be prepared by conventional techniques.

The conversion of a compound of the general formula IV into a compound of the general formula V can be carried out by standard methods for the preparation of, for example, esters, using, for example, alcohols
30 and acid catalysts or thionyl chloride, or of acid chlorides and acid bromides, using, for example, thionyl chloride or thionyl bromide, or of imidazole derivatives, using, for example, carbonyl diimidazole.

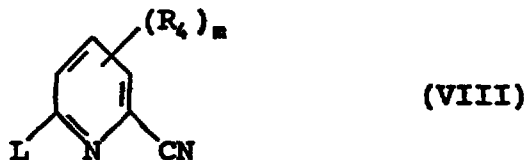
Compounds of the general formula IV may suitably be prepared by hydrolysis of compounds of the general formula:



in which R_3 , R_4 , m and n are as defined above.

10 This reaction is suitably carried out in the presence of an inert solvent such as water or ethylene glycol, using as reactants acids such as hydrochloric acid, sulphuric acid and phosphoric acid or bases such as potassium hydroxide and sodium hydroxide, at a

15 temperature in the range 0-150°C. Compounds of the general formula VII, which form a further aspect of the present invention, may suitably be prepared by reacting a compound of the general formula:



25 in which R_4 , m and L are as defined above, with a compound of the general formula III which is also defined above. The conditions of such reaction are as for the reaction between compounds of the general formulae III and II described above.

30 Compounds of the general formula VIII are either known or can be prepared by conventional techniques, such as, for example, described in T. Sakamoto et al., Chem. Pharm. Bull., 33 (1985), 565-571, or in
35 Björn Elman, Tetrahedron, 41 (1985), 4941-4948.

The compounds of general formula I have been found to have useful herbicidal activity. Also, compounds of general formula VII have been found to have some herbicidal activity. Accordingly, the present invention further provides a herbicidal composition comprising a compound of formula I or formula VII as defined above in association with at least one carrier, and a method of making such a composition is also provided which comprises bringing a compound of formula I or formula VII into association with at least one carrier.

A carrier in a composition according to the invention is any material with which the active ingredient is formulated to facilitate application to the locus to be treated, which may for example be a plant, seed or soil, or to facilitate storage, transport or handling. A carrier may be a solid or a liquid, including a material which is normally gaseous but which has been compressed to form a liquid, and any of the carriers normally used in formulating herbicidal compositions may be used. Preferably compositions according to the invention contain 0.5 to 95% by weight of active ingredient.

Suitable solid carriers include natural and synthetic clays and silicates, for example natural silicas such as diatomaceous earths; magnesium silicates, for example talcs; magnesium aluminium silicates, for example attapulgites and vermiculites; aluminium silicates, for example kaolinites, montmorillonites and micas; calcium carbonate; calcium sulphate; ammonium sulphate; synthetic hydrated silicon oxides and synthetic calcium or aluminium silicates; elements, for example carbon and sulphur; natural and synthetic resins, for example coumarone resins, polyvinyl chloride, and styrene polymers and

copolymers; solid polychlorophenols; bitumen; waxes; and solid fertilisers, for example superphosphates.

Suitable liquid carriers include water; alcohols, for example isopropanol and glycols; ketones, for
5 example acetone, methyl ethyl ketone, methyl isobutyl ketone and cyclohexanone; ethers; aromatic or aliphatic hydrocarbons, for example benzene, toluene and xylene; petroleum fractions, for example kerosine and light mineral oils; chlorinated hydrocarbons, for
10 example carbon tetrachloride, perchloroethylene and trichloroethane. Mixtures of different liquids are often suitable.

Agricultural compositions are often formulated and transported in a concentrated form which is
15 subsequently diluted by the user before application. The presence of small amounts of a carrier which is a surface-active agent facilitates this process of dilution. Thus preferably at least one carrier in a composition according to the invention is a
20 surface-active agent. For example the composition may contain at least two carriers, at least one of which is a surface-active agent.

A surface-active agent may be an emulsifying agent, a dispersing agent or a wetting agent; it may be
25 nonionic or ionic. Examples of suitable surface-active agents include the sodium or calcium salts of polyacrylic acids and lignin sulphonic acids; the condensation of fatty acids or aliphatic amines or amides containing at least 12 carbon atoms in the
30 molecule with ethylene oxide and/or propylene oxide; fatty acid esters of glycerol, sorbitan, sucrose or pentaerythritol; condensates of these with ethylene oxide and/or propylene oxide; condensation products of fatty alcohol or alkyl phenols, for example
35 p-octylphenol or p-octylcresol, with ethylene oxide

and/or propylene oxide; sulphates or sulphonates of these condensation products; alkali or alkaline earth metal salts, preferably sodium salts, of sulphuric or sulphononic acid esters containing at least 10 carbon
5 atoms in the molecule, for example sodium lauryl sulphate, sodium secondary alkyl sulphates, sodium salts of sulphonated castor oil, and sodium alkylaryl sulphonates such as dodecylbenzene sulphonate; and
10 polymers of ethylene oxide and copolymers of ethylene oxide and propylene oxide.

The compositions of the invention may for example be formulated as wettable powders, dusts, granules, solutions, emulsifiable concentrates, emulsions, suspension concentrates and aerosols. Wettable powders
15 usually contain 25, 50 or 75% w of active ingredient and usually contain in addition to solid inert carrier, 3-10% w of a dispersing agent and, where necessary, 0-10% w of stabiliser(s) and/or other additives such as penetrants or stickers. Dusts are usually formulated
20 as a dust concentrate having a similar composition to that of a wettable powder but without a dispersant, and are diluted in the field with further solid carrier to give a composition usually containing $\frac{1}{2}$ -10% w of active ingredient. Granules are usually prepared to have a
25 size between 10 and 100 BS mesh (1.676 - 0.152 mm), and may be manufactured by agglomeration or impregnation techniques. Generally, granules will contain $\frac{1}{2}$ -75% w active ingredient and 0-10% w of additives such as stabilisers, surfactants, slow release modifiers and
30 binding agents. The so-called "dry-flowable powders" consist of relatively small granules having a relatively high concentration of active ingredient. Emulsifiable concentrates usually contain, in addition to a solvent and, when necessary, co-solvent, 10-50%
35 w/v active ingredient, 2-20% w/v emulsifiers and 0-20%

w/v of other additives such as stabilisers, penetrants and corrosion inhibitors. Suspension concentrates are usually compounded so as to obtain a stable, non-sedimenting flowable product and usually contain
5 10-75% w active ingredient, 0.5-15% w of dispersing agents, 0.1-10% w of suspending agents such as protective colloids and thixotropic agents, 0-10% w of other additives such as defoamers, corrosion inhibitors, stabilisers, penetrants and stickers, and
10 water or an organic liquid in which the active ingredient is substantially insoluble; certain organic solids or inorganic salts may be present dissolved in the formulation to assist in preventing sedimentation or as anti-freeze agents for water.

15 Aqueous dispersions and emulsions, for example compositions obtained by diluting a wettable powder or a concentrate according to the invention with water, also lie within the scope of the invention. The said emulsions may be of the water-in-oil or of the
20 oil-in-water type, and may have a thick 'mayonnaise'-like consistency.

The composition of the invention may also contain other active ingredients, for example compounds possessing insecticidal or fungicidal properties or
25 other herbicides.

The present invention still further provides the use as a herbicide of a compound of the general formula I as defined above or a composition as defined above and a method of combating undesired plant growth at a
30 locus with such a compound or composition according to the present invention. The locus may, for example, be the soil or plants in a crop area. The dosage of active ingredient used, may, for example, be in the range of from 0.01 to 10 kg/ha, suitably 0.05 to 4
35 kg/ha.

Examples 1-119 below illustrate the process of method (A) of the invention; Examples 1 to 11 illustrate the preparation of intermediates of general formula II, while Examples 12 to 119 illustrate the preparation of compounds of general formula I.

EXAMPLE 1

Preparation of

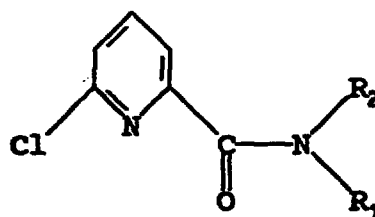
N-(4-fluorophenyl)-2-chloro-6-pyridinecarboxamide

6-chloropicolinic acid (51.2g, 0.325 mol) in thionyl chloride (90 ml) was stirred and heated to reflux for 2 hours. The excess thionyl chloride was evaporated in vacuo and diethylether (250 ml) added to the residual 6-chloropicolinoyl chloride. A solution of 4-fluoroaniline (37.5g, 0.338 mol) in diethylether (100 ml) was added with stirring, maintaining the temperature below 20°C. After the addition, the reaction mixture was stirred overnight at ambient temperature. Water was added to the reaction mixture and the organic layer separated. After a further washing with water and drying with anhydrous magnesium sulphate, the solvent was removed in vacuo to give the title compound as a pale brown solid (66g, 82%), mp 98°C.

EXAMPLES 2 TO 11

By methods analogous to that of Example 1, further compounds of the general formula II were prepared by reaction of compounds of the general formula NHR_1R_2 with compounds of general formula IV. Details are given in Table I.

Table I



Example No.	R ₁	R ₂	mp (°C)	Yield (%)
2	phenyl	H	90	87
3	2,4-F ₂ -phenyl	H	102	69
4	2-F-phenyl	H	88	91
5	3-F-phenyl	H	105	54
6	4-Cl-phenyl	H	111	84
7	benzyl	H	77	71
8	2,2,2-trifluoroethyl	H	82	95
9	n-propyl	H	65	60
10	cyclopropyl	H	77	71
11	n-propyl	n-propyl	oil	85

Elemental analysis data for the intermediates of general formula II described above is set out in Table II below.

Table II

		Analysis (%)					
		C		H		N	
Example No		Calc.	Found	Calc.	Found	Calc.	Found
1		57.5	57.6	3.2	3.4	11.2	11.2
2		62.0	62.1	3.9	4.1	12.0	12.1
3		53.6	53.6	2.6	2.9	10.4	10.4
4		57.5	57.3	3.2	3.5	11.2	11.1

	5	57.5	57.3	3.2	3.5	11.2	11.1
	6	54.0	53.7	3.0	3.0	10.5	10.8
	7	63.3	62.8	4.5	4.9	11.4	11.4
	8	40.3	40.6	2.5	2.8	11.7	11.6
5	9	54.4	54.6	5.6	5.5	14.1	13.9
	10	55.0	54.1	4.6	5.0	14.3	14.1
	11	59.9	60.2	7.1	7.3	11.6	11.5

EXAMPLE 12

10 Preparation of N-(4-fluorophenyl)-2-(2'-chloro-4'-pyridyloxy)-6-pyridinecarboxamide

2-chloro-4-pyridinol (1.3g, 10mmol) was added to a solution of potassium hydroxide (0.56g) in methanol (20 ml). The solvent was evaporated in vacuo
15 after toluene (50 ml) was added to give the anhydrous potassium pyridoxylate. The residue was resolved in anhydrous N,N-dimethylformamide (7 ml). After the addition of N-(4-fluorophenyl)-2-chloro-6-pyridinecarboxamide (2.5g, 10 mmol) and cuprous
20 chloride (0.1 g) the mixture was heated to reflux for 10 hours. After cooling, the mixture was poured into water (150 ml) and ethyl acetate (100 ml). The organic layer was separated and the aqueous phase extracted one more time with ethyl acetate (50 ml). The combined
25 extracts were dried with anhydrous magnesium sulphate and the solvent removed in vacuo. The residue was purified by flash silica gel column chromatography using hexane/ethyl acetate (7:3). The title compound was obtained as a pale yellow solid (1.8g, 52%), mp
30 102°C.

EXAMPLES 13 TO 52

By methods analogous to that of Example 12, further compounds of the general formula I were

prepared by reaction of compounds of general formula II with compounds of general formula III. Details are given in Table III.

5

Table III

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Ex. No.	R	R ₁	R ₂	mp (°C)	Yield (%)
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13	3-Cl-5-pyridyloxy	4-F-phenyl	H	130	36
14	2-Cl-6-pyridyloxy	4-F-phenyl	H	147	26
15	3-pyridyloxy	4-F-phenyl	H	111	10
16	3-Cl-6-pyridyloxy	4-F-phenyl	H	128	20
17	2-Cl-4-pyridyloxy	phenyl	H	106	29
18	2-Cl-4-pyridyloxy	2-F-phenyl	H	128	52
19	2-Cl-4-pyridyloxy	2,4-F ₂ -phenyl	H	129	40
20	6-CH ₃ -2-pyridyloxy	4-F-phenyl	H	115	29
21	3-Br-5-CH ₃ -2-pyridyloxy	4-F-phenyl	H	250	5
22	3-Cl-5-pyridyloxy	2,4-F ₂ -phenyl	H	126	62
23	3-Cl-5-pyridyloxy	phenyl	H	122	36
24	3-Cl-5-pyridyloxy	2-F-phenyl	H	130	56
25	3-Cl-5-pyridyloxy	n-propyl	H	100	39
26	2-Cl-3-pyridyloxy	4-F-phenyl	H	150	29
27	6-CH ₃ -3-pyridyloxy	4-F-phenyl	H	102	46
28	2-Cl-4-pyridyloxy	4-Cl-phenyl	H	140	24

	29	2-Cl-4-pyridyloxy	4-tolyl	H	124	21
	30	2-Cl-4-pyridyloxy	benzyl	H	110	10
	31	2-Cl-4-pyridyloxy	2,2,2-tri-fluoroethyl	H	114	76
5	32	2-Cl-4-pyridyloxy	3-F-phenyl	H	105	30
	33	3-Cl-5-pyridyloxy	3-F-phenyl	H	156	51
	34	3-Cl-5-pyridyloxy	cyclopropyl	H	102	14
	35	2-Cl-6-pyridyloxy	2,4-F ₂ -phenyl	H	150	22
10	36	3-Cl-6-pyridyloxy	phenyl	H	100	25
	37	3-Cl-6-pyridyloxy	2,4-F ₂ -phenyl	H	168	37
	38	2-CH ₃ -6-pyridyloxy	phenyl	H	112	53
	39	3-Cl-6-pyridyloxy	n-propyl	n-propyl	oil	22
15	40	2-CH ₃ -5-pyridyloxy	benzyl	H	oil	26
	41	2-CH ₃ -6-pyridyloxy	cyclopropyl	H	oil	10
	42	5-CH ₃ -6-pyridyloxy	4-F-phenyl	H	169	55
	43	3-Cl-5-pyridyloxy	2,2,2-tri-fluoroethyl	H	127	55
20	44	3-Cl-6-pyridyloxy	n-propyl	H	181	17
	45	2-CH ₃ -6-pyridyloxy	benzyl	H	oil	55
	46	2-CH ₃ -6-pyridyloxy	n-propyl	H	oil	30
	47	2-CH ₃ -6-pyridyloxy	2,2,2-tri-fluoroethyl	H	95	36
25	48	2-CH ₃ -4-CF ₃ -6-pyridyloxy	4-F-phenyl	H	109	55
	49	2-Cl-6-pyridyloxy	phenyl	H	112	41
	50	2-CH ₃ -6-pyridyloxy	2,4-F ₂ -phenyl	H	105	28
30	51	2-CH ₃ -6-pyridyloxy	n-propyl	H	oil	23
	52	3-Cl-5-pyridyloxy	benzyl	H	105	20

EXAMPLE 53

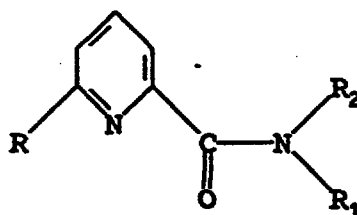
Preparation of N-(2,4-difluorophenyl)-N-ethyl-2-(3'-chloro-5'-pyridyloxy)-6-pyridinecarboxamide

Sodium hydride (0.04g, 1.1 mmol) was added to a solution of N-(2,4-difluorophenyl)-2-(3'-chloro-5'-pyridyloxy)-6-pyridinecarboxamide (0.4g, 1.1 mmol) in anhydrous tetrahydrofuran (10 ml). After stirring for 30 minutes, ethyl iodide (0.18g, 1.1 mmol) was added carefully and the mixture was heated to reflux for 10 hours. After cooling, the mixture was poured into water (75 ml) and ethyl acetate (100 ml). The organic layer was separated and the aqueous phase was extracted in addition with ethyl acetate (100 ml). The combined extracts were dried with magnesium sulphate and the solvent removed in vacuo. The residue was purified by flash silica gel column chromatography using hexane/ethyl acetate (7:3). The title compound was obtained as a white solid (0.16g, 37%), mp 84°C.

EXAMPLES 54 TO 119

By methods analogous to that of Example 53, further compounds of the general formula I were prepared by conversion of compounds of the general formula I. Details are given in Table IV.

Table IV



Ex. No.	R	R ₁	R ₂	mp (°C)	Yield (%)
54	2-Cl-4-pyridyloxy	phenyl	ethyl	93	12
55	2-Cl-4-pyridyloxy	4-F-phenyl	ethyl	108	17
56	2-Cl-6-pyridyloxy	2,4-F ₂ -phenyl	ethyl	oil	35
57	2-Cl-4-pyridyloxy	2,4-F ₂ -phenyl	ethyl	100	22
58	2-Cl-4-pyridyloxy	2,4-F ₂ -phenyl	methyl	oil	61
59	3-Cl-5-pyridyloxy	4-F-phenyl	methyl	oil	71
60	3-Cl-5-pyridyloxy	4-F-phenyl	ethyl	oil	63
61	2-CH ₃ -6-pyridyloxy	4-F-phenyl	methyl	oil	84
62	2-CH ₃ -6-pyridyloxy	4-F-phenyl	ethyl	120	88
63	3-Cl-6-pyridyloxy	2,4-F ₂ -phenyl	methyl	oil	77
64	3-Cl-6-pyridyloxy	2,4-F ₂ -phenyl	ethyl	110	75
65	2-CH ₃ -6-pyridyloxy	phenyl	methyl	87	56
66	2-CH ₃ -6-pyridyloxy	phenyl	ethyl	92	58
67	3-Cl-6-pyridyloxy	4-F-phenyl	methyl	oil	65
68	3-Cl-6-pyridyloxy	4-F-phenyl	ethyl	110	55
69	3-Cl-5-pyridyloxy	2,2,2-tri-fluoroethyl	methyl	oil	66
70	2-Cl-5-pyridyloxy	benzyl	methyl	oil	72
71	3-Cl-6-pyridyloxy	phenyl	methyl	121	57
72	2-Cl-4-pyridyloxy	2,2,2-tri-fluoroethyl	methyl	oil	83
73	5-CH ₃ -6-pyridyloxy	4-F-phenyl	methyl	155	77
74	2-CH ₃ -6-pyridyloxy	benzyl	methyl	oil	64
75	2-Cl-4-pyridyloxy	2,2,2-tri-fluoroethyl	ethyl	oil	60
76	2-Cl-6-pyridyloxy	2,2,2-tri-fluoroethyl	methyl	oil	53

77	2-Cl-4-pyridyloxy	2,2-Cl ₂ cyclopropyl- methyl	methyl	oil	65
78	2-Cl-6-pyridyloxy	2,2-Cl ₂ cyclopropyl- methyl	methyl	oil	82
79	3-pyridyloxy	2,2-Cl ₂ cyclopropyl- methyl	methyl	oil	64
80	2-Cl-4-pyridyloxy	n-propyl	n-propyl	oil	78
81	2-Cl-6-pyridyloxy	4-F-phenyl	methyl	oil	63
82	3-Cl-5-pyridyloxy	2,2-Cl ₂ cyclopropyl- methyl	methyl	oil	65
83	2-Cl-6-pyridyloxy	benzyl	methyl	oil	77
84	2-Cl-4-pyridyloxy	ethyl	methyl	45	82
85	2-Cl-4-pyridyloxy	2-fluoro- ethyl	methyl	oil	39
86	3-Cl-6-pyridyloxy	benzyl	methyl	oil	94
87	3-Cl-6-pyridyloxy	2,2,2-tri- fluoromethyl	methyl	114	76
88	2-Cl-4-pyridyloxy	cyclopentyl	methyl	oil	56
89	2-Cl-4-pyridyloxy	cyclopropyl	methyl	oil	60
90	2-Cl-4-pyridyloxy	cyclopropyl	ethyl	oil	68
91	2-Cl-4-pyridyloxy	3-F-phenyl	methyl	99	62
92	2-Cl-4-pyridyloxy	3-F-phenyl	ethyl	oil	53
93	2-Cl-4-pyridyloxy	cyanomethyl	methyl	124	61
94	2-Cl-4-pyridyloxy	phenyl	methyl	112	40
95	2-Cl-4-pyridyloxy	n-propyl	methyl	oil	44
96	2-Cl-4-pyridyloxy	2-methoxy- ethyl	methyl	oil	60
97	2-Cl-4-pyridyloxy	n-propyl	ethyl	oil	78
98	2-Cl-4-pyridyloxy	methoxy	methyl	oil	74
99	2-Cl-4-pyridyloxy	allyl	methyl	53	62
100	2-Cl-4-pyridyloxy	4-F-phenyl	methyl	101	48
101	2-Cl-4-pyridyloxy	s-butyl	methyl	oil	54
102	2-Cl-4-pyridyloxy	s-butyl	ethyl	oil	46

	103	2-Cl-4-pyridyloxy	cyclopropyl	n-propyl	oil	53
	104	2-Cl-4-pyridyloxy	i-propyl	methyl	oil	95
	105	2-Cl-4-pyridyloxy	i-propyl	ethyl	oil	40
	106	2-Cl-4-pyridyloxy	4-F-benzyl	methyl	oil	64
5	107	2-Cl-4-pyridyloxy	4-F-benzyl	ethyl	oil	37
	108	2-Cl-4-pyridyloxy	methyl	methyl	oil	82
	109	2-Cl-4-pyridyloxy	n-butyl	methyl	oil	65
	110	2-Cl-4-pyridyloxy	n-butyl	ethyl	oil	46
	111	2-Cl-4-pyridyloxy	i-butyl	methyl	oil	69
10	112	2-Cl-4-pyridyloxy	i-butyl	ethyl	oil	42
	113	2-Cl-4-pyridyloxy	t-butyl	methyl	oil	55
	114	2-Cl-4-pyridyloxy	t-butyl	ethyl	oil	30
	115	2-Cl-4-pyridyloxy	cyclopentyl	ethyl	oil	56
	116	2-Cl-4-pyridyloxy	phenyl	i-propyl	oil	43
15	117	2-Cl-4-pyridyloxy	1-phenethyl	methyl	oil	90
	118	2-Cl-4-pyridyloxy	1-phenethyl	ethyl	oil	25
	119	2-Cl-4-pyridyloxy	pivalyl	methyl	oil	48

Examples 120 to 193 below illustrate the process of method (B) of the present invention; Examples 120 to 126 illustrate the preparation of intermediate compounds of the general formula VII which are believed to be novel and form a further aspect of the present invention; Examples 127 to 132 illustrate the preparation of intermediate compounds of the general formula IV; and Examples 133 to 193 illustrate the preparation of compounds of the general formula I.

EXAMPLE 120

Preparation of 2-(2'-chloro-4'-pyridyloxy)-pyridine-6-carbonitrile

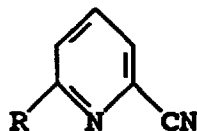
2-chloro-4-hydroxypyridine (6g, 46.3 mmol) was added to a solution of potassium hydroxide (2.9g, 50 mmol, 1.1 eq) in anhydrous methanol (20 ml) at ambient temperature. The solvent was evaporated in vacuo after

toluene (100 ml) was added to give the dry potassium salt. The residue was resolved in anhydrous N,N-dimethylformamide (15 ml) and 6-chloro-pyridine-2-carbonitrile (6.1g, 45 mmol) and cuprous chloride (0.3g) was added. The reaction mixture was heated to reflux under stirring for 3 hours. After cooling, the mixture was poured into water (100 ml) and the aqueous layer extracted 3 times with ethyl acetate (100 ml). The combined extracts were dried with anhydrous magnesium sulphate and the solvent was removed in vacuo. The residue was purified by flash silica gel column chromatography using hexane/ethyl acetate (1:1). The title compound was obtained as white crystals (7.3g, 70%), mp 121°C.

EXAMPLES 121 TO 126

By methods analogous to that of Example 120, further compounds of the general formula VII were prepared by reaction of compounds of the general formula III with compounds of the general formula VIII. Details are given in Table V.

Table V



Example No.	R	mp (°C)	Yield (%)
121	3-Cl-6-pyridyloxy	173	39
122	3-Cl-5-pyridyloxy	87	66
123	2-CH ₃ -5-pyridyloxy	103	60

124	3-CF ₃ -6-pyridyloxy	119	15
125	3-pyridyloxy	104	34
126	2-Cl-6-pyridyloxy	143	24

5 EXAMPLE 127

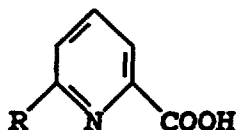
Preparation of 2-(2'-chloro-4'-pyridyloxy)-pyridine-6-carboxylic acid

2-(2'-chloro-4'-pyridyloxy)-pyridine-6-carbonitrile (7.3g, 31.5 mmol) (from Example 120) was suspended in concentrated hydrochloric acid (10 ml) and heated to reflux for 40 minutes. After cooling, water was added and the mixture was diluted with a 40% aqueous solution of potassium hydroxide until the mixture became clear. The solution was extracted with ethyl acetate. The aqueous layer was then acidified with dilute sulphuric acid to precipitate the title compound as a white solid (5.6g, 72%), mp 185°C.

EXAMPLES 128 TO 132

By methods analogous to that of Example 127, further compounds of the general formula IV were prepared by hydrolysis of compounds of the general formula VII. Details are given in Table VI.

Table VI



Example No.	R	mp (°C)	Yield (%)
128	3-Cl-6-pyridyloxy	201	86
129	3-Cl-5-pyridyloxy	250	98

130	3-pyridyloxy	210	91
131	3-CF ₃ -6-pyridyloxy	270	78
132	2-Cl-6-pyridyloxy	172	84

5 EXAMPLE 133

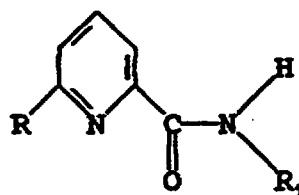
Preparation of N-(2',2'-dichlorocyclopropyl)-methyl-2-
(2'-chloro-4'-pyridyloxy)-6-pyridinecarboxamide

2-(2'-chloro-4'-pyridyloxy)-pyridine-6-carboxylic
acid (1g, 4 mmol) (from Example 127) in thionyl
10 chloride was heated to reflux for 30 minutes. The
excess thionyl chloride was evaporated in vacuo and
acetonitrile (5 ml) was added to the residue. A
solution of (2',2'-dichlorocyclopropyl)-methylamine
(0.6g, 4.4 mmol) and triethylamine (1.5 ml) in
15 acetonitrile (5 ml) was added with stirring at ambient
temperature and the mixture was left overnight. The
solvent was evaporated in vacuo and the residue
resolved in ethyl acetate (25 ml). After extracting
with dilute aqueous potassium hydroxide, the organic
20 layer was dried with anhydrous magnesium sulphate. The
solvent was removed in vacuo and the crude product
purified by flash silica gel column chromatography
using hexane/ethyl acetate (1:1). The title compound
was obtained as a pale yellow solid (0.63g, 50%),
25 mp 106°C.

EXAMPLES 134 TO 174

By methods analogous to that of Example 133,
further compounds of the general formula I were
30 prepared by conversion of compounds of the general
formula IV into compounds of the general formula V and
then reaction with compounds of the general formula
NHR₁R₂. Details are given in Table VII.

Table VII



Ex. No.	R	R ₁	mp (°C)	Yield (%)
134	3-CF ₃ -6-pyridyloxy	2,2,2-tri fluoroethyl	157	39
135	3-Cl-5-pyridyloxy	2-fluoroethyl	105	68
136	2-Cl-4-pyridyloxy	cyclopropyl	oil	72
137	2-Cl-4-pyridyloxy	2-chloroethyl	oil	75
138	2-Cl-6-pyridyloxy	2,2-Cl ₂ -cyclo- propylmethyl	oil	58
139	2-Cl-6-pyridyloxy	3-F-phenyl	88	58
140	2-Cl-6-pyridyloxy	benzyl	oil	73
141	2-Cl-4-pyridyloxy	trimethylsilyl	oil	26
142	2-Cl-4-pyridyloxy	2-fluoroethyl	oil	14
143	3-Cl-6-pyridyloxy	3-F-phenyl	202	56
144	3-Cl-6-pyridyloxy	benzyl	176	65
145	2-Cl-4-pyridyloxy	cyclopentyl	oil	51
146	2-Cl-4-pyridyloxy	cyanomethyl	137	56
147	2-Cl-4-pyridyloxy	2-methoxyethyl	oil	66
148	2-Cl-4-pyridyloxy	1-methyl-2- phenoxyethyl	oil	70
149	2-Cl-4-pyridyloxy	propargyl	103	59
150	2-Cl-4-pyridyloxy	allyloxy	100	44
151	2-Cl-4-pyridyloxy	allyl	76	70
152	2-Cl-4-pyridyloxy	ethoxy	112	82
153	2-Cl-4-pyridyloxy	2-naphthyl	125	50

	154	2-Cl-4-pyridyloxy	1-naphthyl	181	36
	155	2-Cl-4-pyridyloxy	methyl	154	68
	156	2-Cl-4-pyridyloxy	s-butyl	72	68
	157	2-Cl-4-pyridyloxy	i-propyl	oil	51
5	158	2-Cl-4-pyridyloxy	1,1-dimethyl propargyl	87	40
	159	2-Cl-4-pyridyloxy	4-F-benzyl	98	40
	160	2-Cl-4-pyridyloxy	ethyl	91	32
	161	2-Cl-4-pyridyloxy	n-butyl	oil	49
10	162	2-Cl-4-pyridyloxy	i-butyl	oil	39
	163	2-Cl-4-pyridyloxy	t-butyl	70	48
	164	2-Cl-4-pyridyloxy	1,1-diethyl propargyl	oil	70
	165	2-Cl-4-pyridyloxy	1,1-dimethyl propyl	oil	67
15	166	2-Cl-4-pyridyloxy	furfuryl	66	61
	167	2-Cl-4-pyridyloxy	1-phenethyl	97	51
	168	2-Cl-4-pyridyloxy	3-t-butyl- isoxazol-5-yl	108	13
20	169	2-Cl-4-pyridyloxy	benzyloxy	122	37
	170	2-Cl-4-pyridyloxy	t-butoxy	136	31
	171	2-Cl-4-pyridyloxy	n-propyl	75	77
	172	2-Cl-4-pyridyloxy	3,5-F ₂ -phenyl	153	13
	173	2-Cl-4-pyridyloxy	pivalyl	oil	36
25	174	2-Cl-4-pyridyloxy	2-dimethylamino ethyl	oil	17

EXAMPLE 175

Preparation of N-(2,2,2-trifluoroethyl)-2-(3'-pyridyl- oxy)-6-pyridinecarboxamide

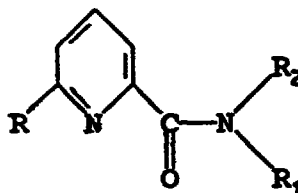
30 To a solution of 2-(3'-pyridyloxy)-pyridine-6-
carboxylic acid (0.65g, 3 mmol) in anhydrous
tetrahydrofuran (10 ml) was added carbonyldiimidazole
(0.54g, 3.3 mmol) and stirred for 30 minutes,
35 maintaining the temperature up to 40°C.

Trifluoroethylamine (0.4g, 4 mmol) was added and the reaction mixture heated to 50°C. After 2 hours, the solution was poured into water (50 ml) and extracted twice with ethyl acetate (100 ml). The combined
 5 extracts were dried with anhydrous magnesium sulphate and the solvent was removed in vacuo. The oily residue was heated with hexane to precipitate the title compound as a white solid (0.6g, 67%), mp 92°C.

10 EXAMPLES 176 TO 192

By methods analogous to that of Example 175, further compounds of the general formula I were prepared by conversion of compounds of the general
 15 formula IV into compounds of the general formula V and then reaction with compounds of the general formula NHR_1R_2 . Details are given in Table VIII.

Table VIII



25

Ex. No.	R	R ₁	R ₂	mp (°C)	Yield (%)
176	3-Cl-5-pyridyloxy	benzyl	methyl	oil	71
30 177	3-Cl-5-pyridyloxy	2,2-Cl ₂ cyclo- propyl- methyl	H	108	69
178	3-Cl-5-pyridyloxy	n-propyl	n-propyl	oil	65
179	3-pyridyloxy	n-propyl	n-propyl	oil	78
35 180	3-pyridyloxy	benzyl	H	110	55

	181	3-pyridyloxy	3-F-phenyl	H	107	70
	182	3-pyridyloxy	benzyl	methyl	oil	63
5	183	2-Cl-6-pyridyloxy	2,2,2-trifluoroethyl	H	oil	84
	184	3-Cl-5-pyridyloxy	morpholino		95	47
	185	2-Cl-4-pyridyloxy	pyrrolidino		oil	33
	186	2-Cl-4-pyridyloxy	piperidino		oil	39
10	187	2-Cl-4-pyridyloxy	2-methylpiperidino		oil	38
	188	2-Cl-4-pyridyloxy	3-methylpiperidino		oil	30
	189	2-Cl-4-pyridyloxy	4-methylpiperidino		oil	30
	190	2-Cl-4-pyridyloxy	3,4-dehydro-piperidino		66	43
15	191	2-Cl-4-pyridyloxy	cyano-methyl	cyano-methyl	155	15
	192	2-Cl-4-pyridyloxy	i-butyl	i-butyl	oil	63

EXAMPLE 193

20 Preparation of N-methyl-N-(2,2,2-trifluoroethyl)-2-(2'-chloro-6'-pyridyloxy)-6-pyridinecarboxamide

Sodium hydride (0.39g, 1.3 mmol, 80% in a dispersion of mineral oil) was added under stirring to a solution of N-trifluoroethyl-2-(2'-chloro-6'-pyridyloxy)-6-pyridinecarboxamide (0.4g, 1.3 mmol) in anhydrous tetrahydrofuran (5 ml) at ambient temperature. After 30 minutes, methyl iodide (0.2g, 1.4 mmol) was added and the mixture heated to reflux for 2 hours. The reaction mixture was poured into water (50 ml) and ethyl acetate (50 ml). The aqueous layer was extracted twice with ethyl acetate and the combined extracts were dried with anhydrous magnesium sulphate. The solvent was removed in vacuo and the residue purified by flash silica gel column

35

chromatography using hexane/ethyl acetate (1:1). The title compound was obtained as a yellow oil (53%).

Elemental analysis data for the compounds of general formula I described above is set out in
5 Table IX below.

Table IX

Analysis (%)

10	Example No.	C		H		N	
		Calc.	Found	Calc.	Found	Calc.	Found
15	12	59.4	59.7	3.2	3.4	12.2	12.2
	13	59.4	59.4	3.2	3.5	12.2	11.9
	14	59.4	59.6	3.2	3.4	12.2	12.1
	15	66.0	66.1	3.9	4.0	13.6	13.4
	16	59.4	59.6	3.2	3.3	12.2	12.1
	17	62.7	63.0	3.7	3.8	12.9	12.8
	18	62.9	62.2	3.2	3.4	12.2	12.1
	19	56.5	56.2	2.8	2.9	11.6	11.5
20	20	66.9	66.9	4.4	4.5	13.0	12.8
	21	53.8	53.6	3.3	3.3	10.5	10.3
	22	56.5	56.4	2.8	2.9	11.6	11.6
	23	62.7	63.0	3.7	3.8	12.6	12.8
	24	59.4	59.5	3.2	3.3	12.2	12.2
	25	57.6	58.0	4.8	4.9	14.4	14.4
25	26	59.4	59.4	3.2	3.3	12.2	12.1
	27	66.9	67.0	4.4	4.5	13.0	12.9
	28	56.5	56.6	3.1	3.3	11.6	11.5
	29	63.4	63.9	4.1	4.3	12.3	12.3
	30	63.4	63.7	4.1	4.4	12.3	12.5
	31	52.7	52.6	3.1	3.3	14.2	14.3
30	32	59.4	58.7	3.2	3.4	12.2	11.9
	33	59.4	59.3	3.2	3.4	12.2	12.1
	34	57.8	58.3	4.2	4.4	14.5	14.5
	35	57.8	58.3	4.2	4.4	14.5	14.5

35	56.5	56.1	3.0	3.6	11.7	12.4
36	62.7	62.8	3.7	4.0	12.9	13.1
37	56.4	56.6	2.8	3.0	11.6	11.6
38	70.8	70.6	5.0	5.2	13.8	13.8
39	61.0	60.1	6.0	6.1	12.5	12.1
40	70.5	70.5	5.4	5.6	13.2	13.2
41	66.9	67.1	5.6	5.9	15.6	15.6
42	66.9	66.7	4.4	4.4	13.0	13.1
43	47.1	47.2	2.7	2.9	12.7	12.6
44	57.8	57.7	4.8	4.9	14.4	14.4
45	71.5	71.5	5.4	5.6	13.2	13.6
46	66.4	65.9	6.3	6.2	15.5	15.2
47	54.0	54.1	3.9	4.1	13.5	13.6
48	58.9	58.8	3.4	3.6	10.7	10.7
49	62.7	62.1	3.7	4.1	12.9	12.7
50	63.3	63.3	3.8	4.2	12.3	12.3
51	69.0	69.0	7.4	7.3	13.4	13.4
52	63.6	63.8	4.2	4.2	12.4	12.5
53	59.6	59.9	3.6	4.2	10.8	11.1
54	64.3	64.7	4.8	5.0	11.8	12.0
55	61.2	61.6	4.3	4.4	11.3	11.3
56	58.6	59.8	3.6	3.9	10.7	10.2
57	58.6	58.7	3.6	3.3	10.8	10.6
58	57.5	57.9	3.2	3.7	11.2	11.0
59	60.4	60.1	3.7	3.5	11.8	11.5
60	61.4	61.5	4.1	4.2	11.3	10.6
61	67.7	66.7	4.8	5.3	12.5	12.1
62	68.4	68.2	5.2	5.4	12.0	11.8
63	57.5	58.3	3.2	3.8	11.2	11.4
64	58.6	58.8	3.6	3.3	10.8	10.7
65	71.5	71.0	5.4	5.6	13.2	13.7
66	72.1	71.5	5.7	6.5	12.6	12.9
67	60.4	60.1	3.7	3.4	11.8	11.9
68	61.4	61.4	4.1	4.1	11.3	12.0
69	48.6	48.6	3.2	3.6	12.2	11.9

70	64.5	64.7	4.6	4.9	11.9	11.4
71	63.6	63.9	4.2	4.6	12.4	12.3
72	48.6	49.0	3.2	3.4	12.2	12.1
73	67.6	68.1	4.8	5.1	12.5	12.3
74	72.1	72.7	5.7	6.1	12.6	12.6
75	50.1	49.6	3.6	3.6	11.7	12.1
76	48.6	49.0	3.2	3.2	12.2	12.4
77	49.7	49.7	3.6	3.7	10.9	11.1
78	49.7	49.4	3.6	3.7	10.9	10.8
79	54.6	54.6	4.3	4.3	11.9	12.0
80	61.2	61.0	6.0	6.1	12.6	12.8
81	60.4	60.4	3.7	3.8	11.8	11.8
82	49.7	49.7	3.7	3.7	10.9	10.8
83	64.5	64.2	4.6	4.6	11.9	11.7
84	57.6	57.7	4.8	5.0	14.4	14.3
85	54.3	54.1	4.2	4.2	13.6	13.5
86	64.5	64.6	4.6	4.4	11.9	11.7
87	48.7	48.4	3.2	3.1	12.1	12.0
88	61.6	61.8	5.4	5.2	12.7	12.7
89	59.3	59.1	4.6	4.2	13.8	13.4
90	60.5	60.2	5.0	5.2	13.2	13.0
91	60.4	60.4	3.6	3.6	11.7	11.8
92	61.4	61.3	4.0	3.7	11.3	11.0
93	55.5	55.1	3.7	3.7	18.5	18.2
94	63.6	63.6	4.1	4.2	12.4	12.1
95	59.0	58.7	5.2	4.9	13.7	13.6
96	56.0	56.3	5.0	5.0	13.1	13.0
97	60.1	60.0	5.6	5.3	13.3	13.2
98	53.2	53.2	4.1	4.0	14.3	14.1
99	59.3	59.4	4.6	4.4	13.8	13.6
100	60.4	60.3	3.6	3.3	11.7	11.4
101	60.1	60.0	5.6	5.6	13.1	13.1
102	61.2	61.6	6.0	6.3	12.6	12.4
103	61.6	61.5	5.4	5.1	12.7	12.3
104	58.9	58.4	5.2	5.1	13.7	13.3

105	60.1	60.0	5.6	5.5	13.1	13.0
106	61.4	61.0	4.0	3.8	11.3	11.1
107	62.3	62.2	4.4	4.6	10.9	11.0
108	56.2	56.2	4.3	4.2	15.1	15.1
109	60.1	59.9	5.6	5.5	13.1	13.0
110	61.2	61.0	6.0	6.1	12.6	12.6
111	60.1	60.0	5.6	5.2	13.1	13.2
112	61.2	61.2	6.0	6.2	12.6	12.4
113	60.1	59.8	5.6	5.4	13.1	13.1
114	61.2	61.2	6.0	6.1	12.6	12.3
115	62.5	62.3	5.8	5.6	12.2	12.1
116	65.3	65.0	4.9	4.4	11.4	11.3
117	65.3	65.2	4.9	4.7	11.4	11.4
118	66.1	66.5	5.2	5.0	11.0	10.8
119	61.2	61.0	6.0	5.6	12.6	12.3
120	57.0	57.0	2.6	2.7	18.1	17.4
121	57.0	57.1	2.6	2.7	18.1	17.5
122	57.0	56.7	2.6	2.7	18.1	17.6
123	68.2	68.2	4.3	4.1	19.9	20.1
124	54.4	54.4	2.3	2.4	15.8	15.4
125	67.0	66.8	3.6	3.6	21.3	21.5
126	57.0	57.1	2.6	2.4	18.1	17.9
127	52.7	52.3	2.8	2.7	11.2	11.2
128	52.7	53.1	2.8	3.1	11.2	11.0
129	52.7	52.3	2.8	2.9	11.2	10.9
130	61.2	61.6	3.7	3.7	13.0	13.1
131	50.7	50.3	2.5	2.7	9.9	9.6
132	52.7	52.9	2.8	3.0	11.2	10.9
133	48.4	48.0	3.3	3.4	11.3	11.1
134	46.0	46.2	2.5	2.8	11.5	11.3
135	52.8	52.8	3.7	3.7	14.2	14.1
136	58.1	58.4	4.1	4.4	14.5	14.3
137	50.0	49.7	3.5	3.5	13.5	13.7
138	48.4	48.6	3.2	3.3	11.3	11.3
139	59.4	59.5	3.2	3.4	12.2	12.1

140	63.6	63.6	4.1	4.2	12.4	12.2
141	52.3	52.0	5.0	4.7	13.1	13.0
142	52.8	52.8	3.7	3.8	14.2	14.0
143	59.4	59.3	3.2	3.4	12.2	12.0
144	63.6	63.3	4.1	4.3	12.4	12.3
145	60.5	60.5	5.1	5.1	13.2	13.1
146	54.1	53.9	3.1	3.2	19.4	19.5
147	54.7	54.7	4.6	4.7	13.7	13.8
148	65.3	65.4	4.9	5.2	11.4	11.5
149	58.5	58.7	3.5	3.4	14.6	14.5
150	55.0	54.7	3.9	3.6	13.7	13.4
151	58.1	58.1	4.1	4.2	14.5	14.5
152	53.2	53.0	4.1	4.3	14.3	14.4
153	67.1	67.0	3.7	3.7	11.2	11.4
154	67.1	66.9	3.7	3.3	11.2	11.1
155	54.7	54.5	3.8	4.2	16.0	16.0
156	58.9	58.4	5.3	5.6	13.7	13.7
157	57.7	57.5	4.8	4.7	14.4	14.3
158	60.9	60.8	4.4	4.5	13.3	13.2
159	60.5	60.5	3.6	3.5	11.7	11.7
160	56.3	56.2	4.3	4.1	15.1	15.2
161	58.9	58.7	5.2	5.2	13.7	13.9
162	58.9	58.9	5.2	5.4	13.7	13.4
163	58.9	58.9	5.3	5.6	13.7	13.6
164	62.9	62.8	5.3	5.3	12.2	12.2
165	60.1	59.9	5.7	5.9	13.1	13.0
166	58.3	58.3	3.7	3.8	12.7	12.7
167	64.5	64.2	4.6	5.0	11.9	11.5
168	58.0	57.7	4.6	4.5	15.0	15.0
169	60.8	60.8	4.0	4.1	11.8	11.8
170	56.0	55.9	5.0	5.1	13.1	13.0
171	57.6	57.6	4.8	5.0	14.4	14.3
172	55.0	55.0	2.8	2.9	11.6	11.2
173	60.1	60.1	5.7	5.8	13.1	13.0
174	56.2	56.0	5.3	5.1	17.5	17.4

	175	52.5	51.3	3.4	3.6	15.1	15.5
	176	64.5	64.5	4.6	4.7	11.9	11.6
	177	48.4	47.7	3.3	3.5	11.3	11.2
	178	61.2	60.5	6.0	6.2	12.6	12.3
5	179	68.2	67.3	7.1	7.3	14.0	14.2
	180	70.8	70.8	5.0	5.2	13.8	14.1
	181	66.0	66.1	3.9	4.1	13.6	13.6
	182	71.5	71.2	5.4	5.5	13.2	13.2
	183	47.1	46.9	2.7	2.9	12.7	12.5
10	184	56.3	56.4	4.4	4.4	13.1	13.1
	185	59.3	59.2	4.6	4.3	13.8	13.6
	186	60.5	60.2	5.0	5.3	13.2	13.1
	187	61.4	61.1	5.4	5.3	12.6	12.3
	188	61.4	61.3	5.4	5.0	12.6	12.2
15	189	61.4	61.2	5.4	5.4	12.6	12.7
	190	60.9	60.8	4.4	4.5	13.3	13.3
	191	55.0	55.1	3.1	3.4	21.4	21.3
	192	63.1	63.1	6.7	6.8	11.6	11.1
	193	48.6	49.1	3.2	3.2	11.2	12.4
20							

EXAMPLE 194

Herbicidal Activity

To evaluate their herbicidal activity, compounds according to the invention were tested using as
 25 representative range of plants: maize, Zea mays (Mz);
 rice, Oryza sativa (R); barnyard grass, Echinochloa
crusgalli (BG); oat, Avena sativa (O); linseed, Linum
usitatissimum (L); mustard, Sinapsis alba (M); sugar
 beet, Beta vulgaris (SB); and soya bean, Glycine max
 30 (S).

The tests fall into two categories, pre-emergence
 and post-emergence. The pre-emergence tests involved
 spraying a liquid formulation of the compound onto the
 soil in which the seeds of the plant species mentioned
 35 above had recently been sown. The post-emergence tests

involved two types of test, viz., soil drench and foliar spray tests. In the soil drench tests the soil in which the seedling plants of the above species were growing was drenched with a liquid formulation
5 containing a compound of the invention, and in the foliar spray tests the seedling plants were sprayed with such a formulation.

The soil used in the tests was a prepared horticultural loam.

10 The formulations used in the tests were prepared from solutions of the test compounds in acetone containing 0.4% by weight of an alkylphenol/ethylene oxide condensate available under the trade mark TRITON X-155. These acetone solutions were diluted with water
15 and the resulting formulations applied at dosage levels corresponding to 5 kg or 1 kg of active material per hectare in a volume equivalent to 600 litres per hectare in the soil spray and foliar spray test, and at a dosage of level equivalent to 10 kilograms of active
20 material per hectare in a volume equivalent to approximately 3,000 litres per hectare in the soil drench tests.

In the pre-emergence tests untreated sown soil and in the post-emergence tests untreated soil bearing
25 seedling plants were used as controls.

The herbicidal effects of the test compounds were assessed visually twelve days after spraying the foliage and the soil, and thirteen days after drenching the soil and were recorded on a 0-9 scale. A rating 0
30 indicates growth as untreated control, a rating 9 indicates death. An increase of 1 unit on the linear scale approximates to a 10% increase in the level of effect.

The results of the tests are set out in Table X below, in which the compounds are identified by reference to the preceding Examples. Absence of a numeral in the Table indicates a zero rating; an asterisk indicates that no result was obtained.

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TABLE X

Compound of Ex. No.		Soil drench 10 kg/ha					Dosage		Foliar spray					Pre-emergence												
		Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
12	7	7	8	8	5	7	8	3	5	7	5	9	5	7	8	9	7	5	4	8	6	8	9	9	6	6
									1	5	2	8	4	7	8	8	7	2	2	6	5	4	6	8	2	2
13	5	3	6	3	2	5	5	2	5	6	4	9	5	5	6	9	9	8	2	2	6	1	3	6	7	7
									1	4	2	9	4	6	9	9	6	1	1	4	1	1	4	5	5	5
14	3	4	1	4	3	4	3	5	5	2	1	8	3	6	8	9	6						3	3	3	3
								1	1	2		7	1	5	8	8	5						3	2	2	2
15	5			3	3			5	5	3	2	8	2	6	8	8	7			2		3	6	4	2	2
								1	1	1	1	7	1	4	8	7	6					2		2	2	2
16	4	2	4	2	1	6	2	2	5	5	1	8	4	6	9	9	7	1		1		2	1		1	1
								1	1	2		7	2	5	9	9	6					2			2	2

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TABLE X (continued)

Compound of Ex. No.	Soil drench 10 kg/ha						Dosage kg/ha						Foliar spray						Pre-emergence					
	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
17	7	7	8	7	6	7	7	2	5	6	5	8	5	8	9	9	8	5	5	6	6	4	8	3
									1	5	5	8	4	7	9	9	8	3		3	2	2	6	2
18	6	6	7	6	5	5	6	4	5	5	5	8	7	7	9	9	7	3	1	6	1	2	7	1
									1	4	5	8	4	7	9	9	7	2		2	1	1	4	2
19	7	6	7	6	4	5	7	2	5	5	5	8	6	7	9	9	7	4	2	4	5	3	7	2
									1	4	2	7	3	7	9	9	7	3		2	3	1	5	4
20			4		3	2			5	4	3	6	4	6	9	9	6						4	
									1	2	2	3	2	5	8	7	5						2	
21	*	*	*	*	*	*	*	*	5	3					5	2	3							
									1						2		1							

TABLE X (continued)

Compound of Ex. No.	Soil drench 10 kg/ha					Dosage kg/ha					Foliar spray					Pre-emergence				
	Mz	R	BG	O	L	M	S	Mz	R	BG	O	L	M	S	B	G	O	L	M	S
27								5	4	5	5	2	5	8	8	6			4	
								1	2	2	3	1	3	7	6	4			2	
28	5	5	6	6	2	3	4	5	8	4	8	5	7	9	9	8	4	5	2	5
								1	5	2	7	3	7	9	9	7		1	3	3
29	5	5	7	5		4	6	1	5	8	5	8	7	7	8	9	7	5	3	2
									1	6	4	7	3	6	8	9	7	2	1	4
30	6	5	6	5	3	6	8	4	5	6	4	8	4	7	9	9	8	5	2	8
									1	4	2	6	2	6	9	9	8	4		7
31	9	9	8	7	8	9	9	8	5	8	7	9	7	8	9	9	9	9	9	9
									1	5	4	8	5	8	9	9	8	9	9	9

TABLE X (continued)

Compound of Ex. No.		Soil drench 10 kg/ha					Dosage kg/ha	Foliar spray					Pre-emergence												
		Mz	R	BG	O	L		M	SB	S	Mz	R	BG	O	L	M	SB	S							
32	6	4	6	6	2	4	5	5	8	6	9	7	8	9	9	8	6	3	7	6	7	9	8	4	
								1	6	4	8	5	8	8	9	8	4	1	5	5	6	8	8		
33	2		4	5	2	3	*	5	6		8	4	5	9	9	7	3		4		3	7	4	3	
								1	3		6	3	5	8	8	6	1		1		1	7	1	1	
34	7	5	7	3	7	9	9	7	5	3	3	9	6	8	8	9	8	8	5	6	4	7	8	9	8
								1	1	1	1	4	4	5	8	7	6	1			3	7	5	3	
35								5	2		3		5	8	8	5									
								1	1		2		3	8	7	4									
36	2	1	5			6	6	5	4	3	7	3	6	9	9	6					4				
								1		1	5	1	5	8	9	6									2

TABLE X (continued)

Compound of Ex. No.		Soil drench 10 kg/ha						Dosage	Foliar spray						Pre-emergence											
		Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
43		7	6	7	6	6	8	9	7	5	3	3	5	1	8	9	9	8	7	6	9	4	6	9	9	7
										1	2		2		6	9	8	6	4	2	5		2	8	9	2
44						1	5	6	4	5					2	4	3	3					4	5	1	
										1								2							2	
45										5			2		4	7	8	6					3	3		
										1					1	5	4	4								
46										5	4	1	4	2	6	7	8	6			1		4	7		
										1					2	4	5	5					1	2		
47										5	4		5		6	8	8	6	3	2	4		4	7	7	5
										1	1		2		5	7	6	6					1	5	6	1

TABLE X (continued)

Compound																									
of		Soil drench 10 kg/ha						Dosage		Foliar spray						Pre-emergence									
Ex. No.	Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
48									5	4	1	1	2	4	6	9	4								
									1	2				2	3	6	2								
49	4		5		2	3			5	7		6	4	6	7	9	6			3			3		
									1	4		5	1	6	7	9	5			1			1		
50									5	5		6	4	6	7	9	6						2		3
									1	2		4	1	5	7	9	6						1		3
51									5	3	2	3		6	7	8	5					2		5	4
									1	1		1		3	5	4	2					1		5	3
52									5	3		3		5	9	9	6								
									1	2		2		2	7	6	4								

TABLE X (continued)

Compound of Ex. No.	Soil drench 10 kg/ha						Dosage kg/ha						Foliar spray						Pre-emergence						
	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	
58	7	6	7	6	7	8	8	7	5	8	4	8	6	7	9	9	8	7	6	7	8	8	9	9	8
									1	3	2	4	4	7	9	9	8	2		4	4	4	9	9	7
59	6	5	6	4	6	8	7	6	5	4	3	7	5	7	9	9	8	6	5	6	5	5	9	9	6
									1	2		3	3	6	9	9	7	3		2		3	8	8	3
60	5	*	4	2	3	7	6	4	5	6	2	7	4	7	9	9	6	5	3	7	3	5	9	9	6
									1	3		3	1	6	9	9	5	2		3		3	8	7	
61									5			2		5	8	7	4					7	3		
									1					2	7	4	2					4			
62									5			6		6	8	7	3					7	3		
									1					3	5	2						2			

TABLE X (continued)

Compound of		Soil drench 10 kg/ha					Dosage		Foliar spray					Pre-emergence											
Ex. No.	Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
63					7	7	1		5	2		2		1	9	9	4						4		
									1	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*
64	*	*	*	*	*	*	*	*	5	2		3	1	4	9	8	2						8	8	
									1			1		2	8	7						5	2		
65					2	6	2		5	3	1	3	1	5	9	7	5					6	2	5	
									1	1				2	6	4	3					3			
66									5	5	2	5	3	6	9	9	5					6	2		
									1	2		2		3	7	5	3					5			
67	*	*	*	*	*	*	*	*	5			2		6	9	9	4		2			9	9		
									1					3	8	8	4					8	8		

TABLE X (continued)

Compound of Ex. No.	Soil drench 10 kg/ha						Dosage kg/ha						Foliar spray						Pre-emergence					
	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
68					3	8	7	3	5	4	2	3	1	6	9	9	6		2		2	9	9	
									1	3		2		4	8	8	5				1	7	7	
69	4		3	2	4	7	8	5	5	6	2	5	3	7	9	9	7	6	8	2	2	7	9	6
									1	2				4	8	7	6	1			1	5	5	
70	7	6	7	6	6	7	7	*	5	7	4	8	5	8	9	9	9	7	7	7	6	6	9	8
									1	3		2	2	7	9	9	8	5	2	4	2	3	7	2
71			2		5	8	9	3	5	5		4		6	9	9	6		2		2	9	8	2
									1					3	8	9	5					7	5	
72	8	7	7	7	8	9	*	9	5	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*
									1	2	1	4	2	7	9	9	8	6	7	5	6	5	9	7

TABLE X (continued)

Compound of Ex. No.		Soil drench 10 kg/ha						Dosage						Foliar spray						Pre-emergence						
		Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
78		*	*	*	*	*	*	*	*	5	6	2	2	2	7	8	6									
										1					6	6	5									
79										5		7	4	2	8	7	7									
										1					3	3	6									
80		8	6	8	7	5	6	*	7	5	7	1	8	6	7	8	9	8	6	3	8	3	2	7	8	1
										1	6		8	4	6	8	9	8	3	2	7		2	6	7	
81		5		4		4	7	6	5	5	5	1	8	3	5	8	9	9	2		5		5	9	8	5
										1	2	1	4	1	3	7	9	8					3	8	7	
82										5	3		8		3	9	9	8								
										1	3		3		3	6	8	8								

TABLE X (continued)

Compound of		Soil drench 10 kg/ha						Dosage						Foliar spray						Pre-emergence					
Ex. No.	Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
83									5	4		7			7	7	7								
									1			2			5	5	4								
84	7		6	3	6	7	8	7	5	2	2	4	3	4	7	7	7	4		2		2	4	6	3
									1					3	6	5	6						2		
85	6		6	5	3	7	8	7	5	4				5	7	7	8		3		4	6	7		
									1	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*
86									5			4	2	4	7	7	4								
									1					2	5										
87									5	3		4	2	4	7	6	4								
									1						4	3									

TABLE X (continued)

Compound of		Soil drench 10 kg/ha						Dosage	Foliar spray						Pre-emergence											
Ex. No.		Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
88	6			6	5	5	8	9	7	5	5		9	5	5	8	8	9	7	3	5	4	4	8	6	6
										1	2		6	4	4	7	8	8	4		4			6	2	
89	9	6	5	6	6	9	9	9	9	5	4		9	4	6	8	9	8	8	6	9	4	5	9	9	9
										1	2		5		5	7	8	7	7	2	3	2	2	8	7	7
90	9	9	8	7	7	9	9			5	8	4	9	8	7	8	9	9	9	8	9	9	9	9	9	9
										1	3		8	6	6	8	9	8	7	6	8	6	6	9	9	8
91	7	7	8	7	6	7	9	5		5	7	6	9	7	7	8	9	9	7	5	8	6	7	9	9	7
										1	5	2	8	6	7	8	9	8	5	2	6	4	6	9	9	5
92	7	6	8	7	6	7	9	5		5	6	5	9	6	7	8	9	9	5	4	8	6	7	9	9	9
										1	6	5	8	5	7	7	9	8	2		7	5	6	9	9	5

TABLE X (continued)

Compound of Ex. No.		Soil drench 10 kg/ha						Dosage		Foliar spray						Pre-emergence											
		Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	
93	3					5	6	4	7	5	4			2	2	5	7	7	8	3			2		6	4	5
										1	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	
94	7	8	7	6	5	7	8	8		5	7	6	8	7	6	7	9	9	7	6	7	6	8	9	7		
						1	2	3	7	6	6	7	8	8	6	5	7	4	8	8	6						
95	7	7	6	6	6	7	8	7		5	4	4	9	3	6	7	7	9	7	4	8	7	3	8	7		
						1	2	3	2		5	6	7	8	5					2	2	2	7	6	2		
96					3	5	6	7		5	5		4		5	7	6	9	5		4		3	6	5		
										1					3	6	5	5						2	2		
97	7	6	7	7	5	8	9	8		5	7	3	8	7	6	7	8	9	7	5	8	6	5	8	9		
						1	4	1	7	2	5	6	7	8	6	2	2	2	2	4	7	8	3				

TABLE X (continued)

Compound of Ex. No.	Soil drench 10 kg/ha						Dosage kg/ha						Foliar spray						Pre-emergence					
	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
98	5		6		4	6	6		5	4		6	3	3	7	8	7	6		6		8	7	
									1	2			2	6	7	4			2			6	6	
99	4		3	2	3	6	4	6	5	5		8	4	5	8	8	7	3	7	6	4	7	7	6
									1	2		3	2	4	7	6	7	3	2			2	2	4
100	7	6	7	7	7	8	9	8	5	6	4	9	7	7	9	9	8	6	4	7	7	9	9	8
									1	2	1	7	6	7	9	7	8	5	2	6	6	9	9	7
101	4				4	5	6	6	5	3		8	3	4	7	8	8	6		4		9	8	5
									1	1				2	6	8	8					7	8	2
102	7	6	7	7	7	8	9	8	5	5	4	8	6	7	8	9	8	6	6	9	7	4	9	8
									1	2	4	7	5	6	7	8	8	6	5	7	6	2	8	9

TABLE X (continued)

Compound of Ex. No.		Soil drench 10 kg/ha					Dosage	Foliar spray					Pre-emergence													
		Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
103		8	8	7	7	6	7	7	8	5	4	4	9	9	7	8	9	8	7	5	9	9	8	9	9	9
										1	2	3	8	6	6	8	8	8	6	2	9	6	4	8	9	8
104		4				3	6	6	6	5	4		5	4	6	7	9	8	7	5	2	3	7	8	6	
										1			2		2	6	8	8	2				6	7	2	
105		*	*	*	*	*	*	*	*	5	4	4	4	9	6	6	7	9	8	7	5	9	4	5	9	9
										1	2	2	4	2	5	7	8	7	4	2	7		4	7	8	
106		4		6		3	2			5	5	3	8	4	6	7	9	6	6	5	4	3	7	6	3	
										1	2	2	6	2	5	6	9	5	2	2			6	5		
107		6	3	3	3	1	5	6		5	3		7	4	5	7	9	5	4		5	6	3	7	7	
										1	2		6	2	4	6	8	4			2		2	7	5	

TABLE X (continued)

Compound of Ex. No.	Soil drench 10 kg/ha						Dosage kg/ha						Foliar spray						Pre-emergence					
	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
108					4	6	7	5	5				4	6	7	6						5	6	
									1	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*
109	6		3	4	5	5	2	3	5	5	3	9	5	6	9	8	6	5	7	3	2	9	9	4
									1	2		2	2	5	7	8	6	4	2	2		7	8	2
110	*	*	*	*	*	*	*	*	5	6	3	8	6	6	8	9	8	7	6	9	4	5	9	8
									1	4		7	5	5	7	9	8	7	5	9	2	2	8	6
111	7		4		3	8	7	6	5	5		8	6	7	8	9	8	7	4	9	6	5	9	7
									1	2		5	2	6	7	9	7	5		6	2	2	8	2
112	7	3	8	6	5	7	7	6	5	7		9	6	7	8	9	8	8	5	9	6	5	9	9
									1	4		7	5	6	8	8	8	7	2	6	2	2	9	7

TABLE X (continued)

Compound of		Soil drench 10 kg/ha				Dosage		Foliar spray				Pre-emergence													
Ex. No.	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	
113			3	6		5	5	4		8	4	6	8	9	7	4	6		5	6	7	6			
							1			2	1	5	7	7	7	2	2		2	5	2				
114			5	4	3	4	5	3		7	6	3	7	9	5				6	4					
							1	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*		
115	7		3	2	4	6	4	6	5	5	4	8	6	7	8	9	8	7	1	6	2	2	8	9	8
							1	4		4	4	6	7	9	8	5	4		2	7	8	7			
116		6	5	6	4	5	5	4		7	6	8	9	9	7	4	7		6	9	9	7			
							1	2		2	4	7	8	9	7	2	6		5	7	9	7			
117			4	2			5	5	2	8	4	6	9	9	7		4		6	5					
							1	4		4	2	5	8	8	6				5	2					

TABLE X (continued)

Compound of		Soil drench 10 kg/ha					Dosage		Foliar spray					Pre-emergence											
Ex. No.	Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
126									5			2		3	6	6	5								
									1					1	5	4	4								
133	7	6	7	5	5	7	7	4	5	7	4	7	3	7	9	9	8	7	3	5	3	4	9	8	3
									1	5	2	5	1	7	8	9	7	5		2	1	2	8	7	
134									5	3		4		5	7	6	5								
									1	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*
135	6	4	1		6	5	2		5	3	2	9	6	7	8	8	7	6	2	7	4	5	8	9	4
									1	2		7	4	5	7	6	7	2		3			6	2	
136	8	8	8	7	7	9	8		5	9	7	9	9	8	9	9	8	9	8	9	9	7	9	9	3
									1	7	4	8	7	7	8	9	8	8	7	9	8	2	9	8	1

TABLE X (continued)

Compound of		Soil drench 10 kg/ha								Dosage	Foliar spray								Pre-emergence							
Ex. No.	Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	
142	7	6	8	6	4	7	8	8	5	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	*	
									1			4	3	6	6	8	7							6	2	
143	*	*	*	*	*	*	*	*	5			4		2	6	4										
									1																	
144									5			3	6	2												
									1					2												
145	7	7	8	7	4	7	9	7	5	8	4	9	4	7	8	9	9	7	5	9	6	6	8	8	6	
									1	6	2	8	3	6	7	9	8	6	2	8	3	5	7	8	2	
146	7	6	7	3	6	5	6	8	5	5		6	4	7	7	9	9	7	2	6	4	5	7	9	6	
									1			2	1	5	7	9	9	2	2		2	4	5			

TABLE X (continued)

Compound of		Soil drench 10 kg/ha					Dosage		Foliar spray					Pre-emergence												
Ex. No.		Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
152		7	7	7	7	5	7	8	6	5	7	7	5	6	7	8	7	7	7	6	7	6	7	9	9	6
										1	4	6	4	5	7	8	6	7	2	5	4	3	8	8	2	
153						2	4	3		5	4	6	4	5	8	8	7									
										1	2	5		4	8	7	6									
154										5	4	3	2	4	6	8	6									
										1	2		1	4	6	8	5									
155		6	5	6	2	6	4	3	2	5	5	7	4	6	7	9	8	8	6	9	4	5	9	9	8	
										1	2	6	2	5	7	9	8	7	2	5	2	5	9	8	2	
156		7	7	6	7	6	8	8	7	5	7	5	9	7	7	9	9	8	6	9	7	9	9	9	8	
										1	5	5	7	5	6	8	8	7	5	9	6	8	9	9	6	

TABLE X (continued)

Compound of Ex. No.	Soil drench 10 kg/ha						Dosage kg/ha						Foliar spray						Pre-emergence					
	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
157	8	7	7	8	6	8	8	8	5	7	4	9	8	7	8	8	7	8	9	8	7	9	9	8
									1	5		7	2	6	7	7	7	7	9	6	5	9	9	7
158	4		6	4	2	7	6	6	5	5	3	9	6	7	9	9	8	7	4	6	4	6	9	6
									1	4		7	4	6	8	9	6	2		5		2	7	2
159	5		4		2	4	2	1	5	5	2	8	5	6	8	9	6	5		6	3	2	7	4
									1	2	1	7	2	5	7	8	6	2		2		1	7	2
160	7	7	7	6	5	7	7	6	5	7	5	9	8	6	9	9	8	7	7	9	8	9	9	6
									1	5	2	8	6	5	9	9	8	7	2	9	7	5	9	2
161	7	5	7	7	4	4	9	6	5	6	4	9	7	6	9	9	8	6	6	9	7	5	9	8
									1	5	2	8	6	5	9	9	7	5	4	9	6	2	9	7

TABLE X (continued)

Compound of Ex. No.	Soil drench 10 kg/ha						Dosage	Foliar spray						Pre-emergence											
	Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
162	7	4	7	6	3	9	7	4	5	7	4	9	7	6	9	9	8	7	7	9	6	5	9	9	8
									1	5	2	8	7	5	9	9	8	6	5	8	2	2	9	9	8
163	4		3	2	3	5	2	4	5	4		9	6	6	9	9	7	5		9	2	2	9	8	4
									1	2		8	5	2	8	9	6			6			7	7	
164					4	2			5	6		6	4	4	8	9	5					3	6	4	
									1			4	2	3	7	8	2					2	2	2	
165					4	6			5	4		9	5	3	8	9	6			4		6	7		
									1	2		4	2	2	7	8	5			2		5	2		
166									5	4	2	6	4	6	8	9	6			7		4	7	7	4
									1	3		2	2	5	8	8	5			7		2	7	7	

TABLE X (continued)

Compound of Ex. No.		Soil drench 10 kg/ha						Dosage	Foliar spray						Pre-emergence										
		Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB
167		6	3	4	3	4	2	5	6		7	3	7	9	9	7			5	3	2	6	6	2	
							1	2			4	2	5	8	8	6			3			6	2		
168	4	2			3	2	4	5	5		6	2	3	9	5	6						6			
								1	2		5		1	8	2	5						6			
169								5	4		3	3	4	8	9	5						2	2		
								1	2				3	8	8	4						2			
170	8	6	7	7	7	9	9	8	5	8	4	9	8	7	9	9	8	9	8	9	9	9	9	8	
								1	7	2	8	6	7	9	9	8	8	7	9	8	9	9	9	8	
171	7	7	8	8	2	9	9	8	5	9	7	9	8	7	9	9	8	9	9	9	6	9	9	9	
								1	7	5	9	6	6	9	9	8	8	5	9	7	5	9	9	6	

TABLE X (continued)

Compound of Ex. No.	Soil drench 10 kg/ha						Dosage kg/ha						Foliar spray						Pre-emergence					
	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
180									5	4			5		3	7	8	6						
								1	1			2		2	6	7	3							
181									5	6			6	2	6	9	9	7						
								1	4			2		5	6	7	2							
182	5	3	6	2		5	4		5	6	3	8	2	7	9	9	7	4		5	3	3	8	8
								1	5			5		5	7	9	5	1		1		1	7	7
183	6	4	6	4	6	7	8	7	5	6			7	2	7	8	9	8	7	6	7	3	8	7
									1	4			2		6	7	9	8	5	2	6	1	7	6
184									5	3	1	5	1	2	6	3	7							
									1	2		2		2	1	4								

TABLE X (continued)

Compound of Ex. No.	Soil drench 10 kg/ha						Dosage	Foliar spray						Pre-emergence												
	Mz	R	BG	O	L	M	SB	S	kg/ha	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	
185	6	2	6	5	4	7	2	6	5	5	5	7	3	6	7	8	8	7	7	6	3	7	4	6	6	
									1	2	2	2		2	6	6	6	5	2			6		2		
186				4	3	5	4	5	5	3	3	4	3	2	7	8	6	2	4	2	6	7	4		4	6
									1		2				6	6	5									
187						3			5	4	3	5	3	5	3	5	6	5	3	2		5	3			
									1	2					2	2	2				2					
188	5	2	4			5	6	2	5	5	8	4	4	4	7	9	6	5	7			9	9			
									1	2	7	2	3	6	8	2	2	2	2			7	7			
189									5	5	7	3	5	7	9	7	5	4			7	3				
									1	2	2		2	7	8	6						2				

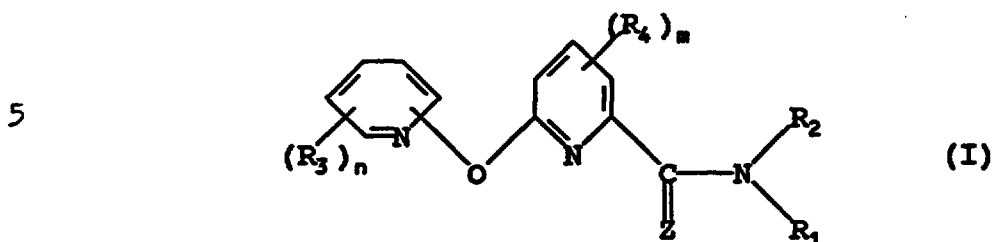
TABLE X (continued)

Compound of Ex. No.	Soil drench 10 kg/ha						Dosage kg/ha						Foliar spray						Pre-emergence					
	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S	Mz	R	BG	O	L	M	SB	S
190	1				3		2		5	4	2	7	4	6	8	9	6		5	3	4	7	5	5
								1	2					4	7	6	7					2		
191					6		4		5	4		3		5	7	9	9				5	3		
								1	2					4	7	8	7				2			



C L A I M S

1. A compound of the general formula:



wherein R_1 and R_2 each independently represents a
hydrogen atom, or an alkyl, aryl, alkenyl, alkynyl,
10 cycloalkyl, cycloalkylalkyl, aralkyl, alkaryl, hydroxy,
alkoxy, alkenyloxy, alkynyloxy, alkylcarbonyl,
alkoxycarbonyl, amino, mono- or di-alkylamino,
alkoxycarbonylamino group, in which groups any alkyl,
alkenyl, alkynyl, cycloalkyl or aryl moiety is
15 optionally substituted, an optionally substituted
heterocyclic group, an arylamino group optionally
substituted by a halogen atom, a dialkylcarbamoyl
group, a trialkylsilyl group or a fused ring structure,
or together form an alkenylene chain or an optionally
20 substituted alkylene chain which is optionally
interrupted by an oxygen or sulphur atom or by a group
-NR- in which R represents a hydrogen atom or an alkyl
group; R_3 represents a hydrogen or halogen atom, or an
alkyl, alkoxy, aryl or aryloxy group optionally
25 substituted by one or more of the same or different
substituents selected from halogen atoms and cyano,
hydroxy and alkoxy groups, or represents a cyano,
carboxy, alkoxycarbonyl, (alkylthio)carbonyl,



alkylcarbonyl, amido, alkylamido, nitro, alkenyloxy, alkynyloxy, alkylthio, haloalkylthio, alkenylthio, alkynylthio, alkylsulphanyl, alkylsulphonyl, alkyloximinoalkyl or alkenyloximinoalkyl group; R_4 represents a halogen atom or an alkyl or haloalkyl group; Z represents an oxygen or sulphur atom; m is 0-3; and n is 1-4.

2. A compound as claimed in claim 1 wherein when any one of the substituents R_1 , R_2 , R_3 and R_4 represents or contains an alkyl, alkenyl or alkynyl substituent group, this has up to 12, preferably up to 6, carbon atoms; any cycloalkyl group has from 3 to 6 carbon ring atoms; and any alkenylene or alkylene chain has from 3 to 6 chain members.

3. A compound as claimed in claim 1 or 2 wherein R_1 represents an unsubstituted C_{1-6} alkyl group, a C_{1-5} alkyl group substituted by an alkoxy, aryloxy or cyano group or by one or more halogen atoms, a C_{1-6} alkoxy group optionally aryl-substituted, a C_{2-7} alkynyl group, a C_{2-6} alkenyl group, a C_{2-6} alkenyloxy group, an unsubstituted aryl group, an aryl group substituted by one or more halogen atoms, or an aralkyl, alkaryl, C_{3-6} cycloalkyl, substituted (C_{3-6} cycloalkyl)alkyl group, a heterocyclic group, a dialkylaminoalkyl group, a naphthyl group or a trimethylsilyl group; R_2 represents a hydrogen atom or an optionally substituted C_{1-6} alkyl group; and R_3 represents a hydrogen or halogen atom, or a C_{1-6} alkyl or C_{1-6} haloalkyl group.

4. A compound as claimed in claim 1, 2 or 3 wherein m is 0.

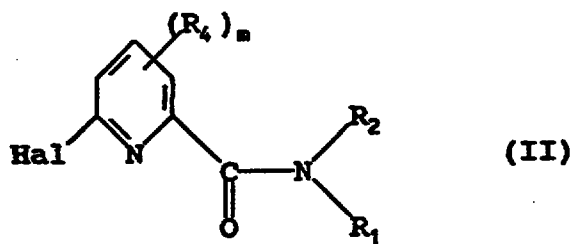
5. A compound as claimed in any one of claims 1 to 4 wherein Z represents an oxygen atom.

6. A process for the preparation of a compound as claimed in claim 1, in which Z represents an oxygen



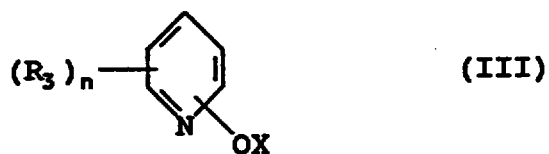
atom, which comprises either (A) reacting a compound of the general formula:

5



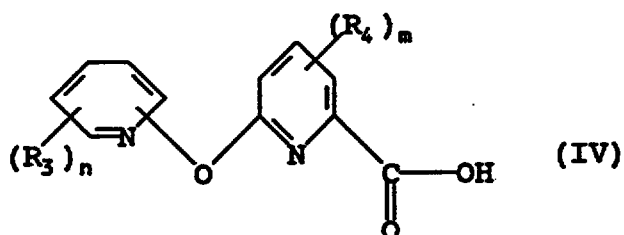
10 with a compound of the general formula:

15



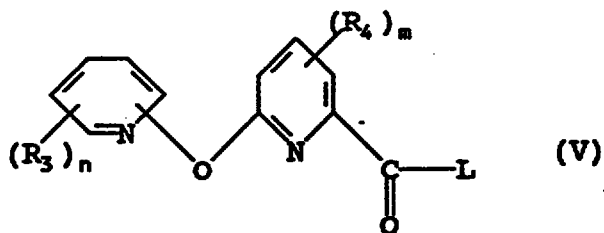
or (B) by converting a compound of the general formula:

20



25 into a derivative of the general formula:

30

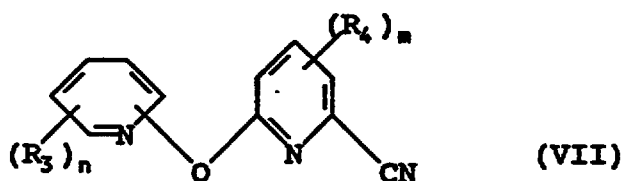


35 which is then reacted with an amine of the general formula NHR_1R_2 , wherein R_1 , R_2 , R_3 , R_4 , m and n are as



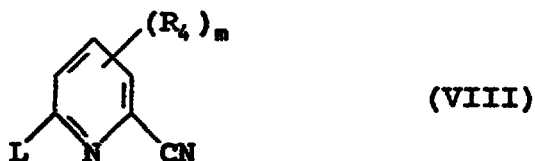
defined in claim 1, Hal represents a halogen atom, X represents a hydrogen atom or an alkali metal atom, and L is a leaving group, with the optional further step of converting the resultant compound of the general formula I into a further compound of the general formula I.

7. A compound of the general formula:



15 in which R_3 , R_4 , m and n are as defined in any one of claims 1 to 4.

8. A process for the preparation of a compound as claimed in claim 7, which comprises reacting a compound of the general formula



25 in which R_4 , m and L are as defined in claim 6, with a compound of the general formula III as defined in claim 6.

9. A herbicidal composition comprising a compound as claimed in any one of claims 1 to 5 or in claim 7, together with at least one carrier.

10. A composition as claimed in claim 9, which comprises at least two carriers, at least one of which is a surface-active agent.

11. A method of combating undesired plant growth at a locus, which comprises treating the locus with a



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compound of formula I as claimed in any one of claims 1 to 5, with a compound of formula VII as claimed in claim 7, or with a composition as claimed in claim 9 or 10.

- 5 12. The use as a herbicide of a compound as claimed in any one of claims 1 to 5 or in claim 7, or a composition as claimed in claim 9 or 10.

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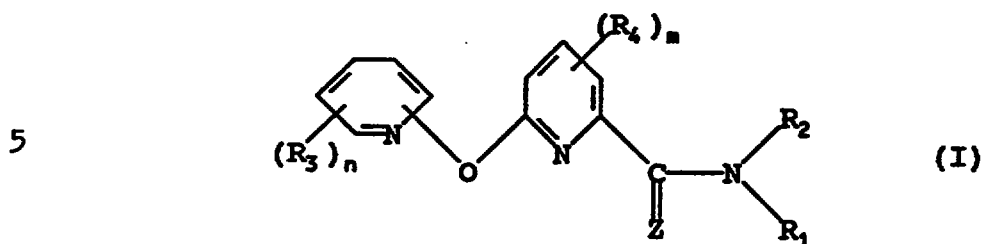
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A B S T R A C T

HERBICIDAL PICOLINAMIDE DERIVATIVES

Compounds of the formula:



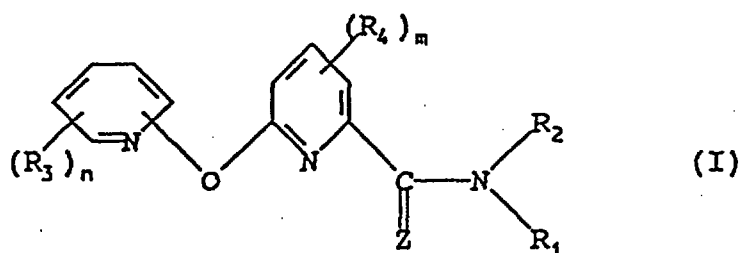
wherein R_1 and R_2 each independently represents hydrogen, optionally substituted alkyl, aryl, alkenyl, alkynyl, cycloalkyl, cycloalkylalkyl, aralkyl, alkaryl, hydroxy, alkoxy, alkenyloxy, alkynyloxy, alkylcarbonyl, alkoxy carbonyl, amino, mono- or di-alkylamino, alkoxy carbonylamino, optionally substituted heterocycle or arylamino, dialkylcarbamoyl, trialkylsilyl or fused ring structure, or together form an alkenylene chain or an alkylene chain which is optionally interrupted by oxygen or sulphur or $-NR-$, R being hydrogen or alkyl; R_3 represents hydrogen or halogen, or optionally substituted alkyl, alkoxy, aryl or aryloxy, or cyano, carboxy, alkoxy carbonyl, (alkylthio)carbonyl, alkylcarbonyl, amido, alkylamido, nitro, alkenyloxy, alkynyloxy, alkylthio, haloalkylthio, alkenylthio, alkynylthio, alkylsulphinyl, alkylsulphonyl, alkyloximinoalkyl or alkenyloximinoalkyl; R_4 represents halogen, alkyl or haloalkyl; Z represents oxygen or sulphur; m is 0-3; and n is 1-4, and certain intermediates in their production, exhibit herbicidal activity.

07 JUL 1998

HERBICIDAL PICOLINAMIDE DERIVATIVES

The present invention relates to certain new picolinamide derivatives, their preparation, herbicidal compositions containing them, and their use in combating undesired plant growth. US-A-4270946 teaches that certain N-aryl-2-phenoxy nicotinamides are useful as herbicides. From US-A-4493730 it is known that 2-cyano-3-nitro-6-(2-chloro-4-trifluoromethylphenoxy) pyridine can be used as a herbicide.

- 5 In accordance with the present invention there is provided a compound of the general formula:



- 10 wherein R_1 and R_2 each independently represents a hydrogen atom, or an alkyl, aryl, alkenyl, alkynyl, cycloalkyl, cycloalkylalkyl, aralkyl, alkaryl, hydroxy, alkoxy, alkenyloxy, alkynyloxy, alkylcarbonyl, alkoxy carbonyl, amino, mono- or di-alkylamino, alkoxy carbonylamino group, in which groups any alkyl, alkenyl, alkynyl, cycloalkyl or aryl moiety is optionally substituted, an optionally substituted heterocyclic group, an arylamino group optionally substituted by a halogen atom, a dialkylcarbamoyl group, a trialkylsilyl group or a fused ring structure, or together form an alkenylene chain or an optionally substituted alkylene chain which is optionally interrupted by an oxygen or sulphur atom or by a group -NR- in which R represents a hydrogen atom or an alkyl group; R_3 represents a hydrogen or halogen atom, or an
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