

141912

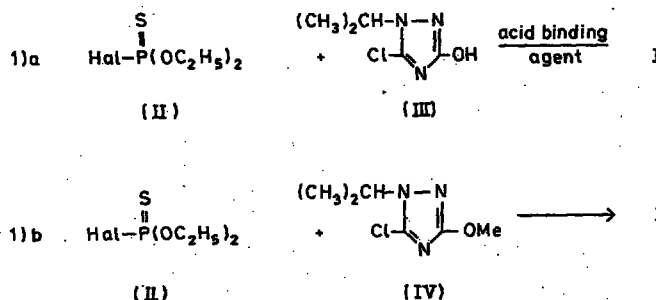
- (19)
- 

(54) TRIAZOLYLORGANOPHOSPHORUS DERIVATIVE AND ITS USE IN PEST CONTROL.

5 The present invention relates to a triazolyorganophosphorus derivative, its preparation and use in pest control.

$$\text{(CH}_3\text{)}_2\text{CH}-\text{N}=\text{N}-\text{C}(\text{Cl})=\text{N}-\text{O}-\text{P}(\text{OC}_2\text{H}_5)_2 \quad \text{(I)}$$

(3)]-phosphorothionate.
The compound of formula I can be manufactured analogously to known methods, e.g. as shown schematically below



The following bases, for example, are suitable as acid binding agents, tertiary amines, such as triethylamine, dimethylaniline, pyridine, inorganic bases, such as hydroxides and carbonates of alkali and alkaline earth metals, preferably sodium and potassium hydroxide.

The starting materials of the formulae III and IV are new and can be manufactured, for example, by chlorination of 1-isopropyl-3-hydroxy-triazole which is unsub-

stituted in the 5-position, or of an ester thereof followed in the latter case by hydrolysis.

The compound of formula I has insecticidal, nematocidal and acaricidal activity. Accordingly the invention provides a method of combating insects, nematodes or acarina which method comprises applying to a locus infested or liable to be infested with the insects, nematodes or acarina the compound of formula I. The compounds may be used against all development stages of the insects, nematodes and acarina. The compounds are particularly effective against insects, nematodes and acarina which are harmful to seeds, tubers, plants or plant parts.

The compound of formula I may be used as pure active substance or, in composition form, together with a solid extender, a solid extender and a surface active agent or a liquid diluent and a surface active agent. The compositions can be solid or liquid and can contain substances conventionally used in formulation technique such, for example, as solvents, dispersants, wetting agents, adhesives, thickeners, binders and/or fertilisers.

For application, the compound of formula I may be processed to a dust, emulsifiable concentrate, granulate, dispersion, spray, solution, or suspension in the conventional formulation which is commonly employed in application technology. Mention may also be made of "cattle dips" and "spray races", in which aqueous preparations are used.

The compositions according to the invention may be manufactured in known manner, e.g. by intimately mixing and/or grinding the compound of formula I with a suitable solid extender, optionally with the addition of dispersants or solvents which are inert towards the active substances. The active substance may take and be used in the following forms:

Solid forms:

Dusts, tracking agents, granules, coated granules, impregnated granules and homogeneous granules.

Liquid forms:

- a) formulations dispersible in water: wettable powders, pastes, emulsions;
- b) solutions.

To manufacture solid forms (dusts, tracking agents), the active substance is mixed with a solid extender. Suitable extenders are, for example: kaolin, talcum, bolus, loess, chalk, limestone, ground limestone, attaclay, dolomite, diatomaceous earth, precipitated silica, alkaline earth metal silicates, sodium and potassium aluminium silicates (feldspar and mica), calcium and magnesium sulphates, magnesium oxide, ground synthetic materials, fertilisers, for example ammonium sulphate, ammonium phosphate, ammonium nitrate, urea, ground vegetable products, such as corn meal, bark dust, sawdust, nutshell meal, cellulose powder, residues of plant extractions, and activated charcoal. These substances can either be used alone or in admixture with one another.

Granules can be very easily manufactured by dissolving the compound of formula I in an organic solvent and applying the resulting solution to a granulated material, for example attapulgit, SiO_2 , granicalcium, or bentonite and then evaporating the solvent.

Polymer granules can also be manufactured by mixing the compound of formula I with polymerisable compounds (urea/formaldehyde; dicycandiamide/formaldehyde; melamine/formaldehyde or others), whereupon a mild polymerisation is carried out that does not affect the active substance and in the process of which the granulation is carried out during the gel formation. It is more advantageous to impregnate finished, porous polymer granules (urea/formaldehyde, polyacrylonitrile, polyester or others) which have a specific surface area and a favourable predeterminable adsorption/desorption ratio, with the active substance, for example in the form of its solutions (in a low boiling solvent) and to remove the solvent. Polymer granules of this kind in the form of microgranules having a bulk density of 300 g/litre to 6000 g/litre can also be manufactured with the aid of atomisers. The dusting can be carried out from aircraft over extensive areas of cultures of useful plants.

It is also possible to obtain granules by compacting the extender with the active substance and subsequently comminuting the product.

To these mixtures can also be added additives which stabilize the active substance and/or non-ionic, anionic and cationic surface active substances, which, for example, improve the adhesion of the active ingredient on plants or parts of plants (adhesives and agglutinants) and/or ensure a better wettability (wetting agents) and

dispersibility (dispersing agents). Examples of suitable adhesives are the following: olein/chalk mixture, cellulose derivatives (methyl cellulose, carboxymethyl cellulose), hydroxyethyl glycol ethers of monoalkyl and dialkyl phenols having 5 to 15 ethylene oxide radicals per molecule and 8 to 9 carbon atoms in the alkyl radical, lignin sulfonic acids, their alkali metal and alkaline earth metal salts, polyethylene glycol ethers (Carbowaxes—"Carbowax" is a registered Trade Mark), fatty alcohol polyethylene glycol ethers having 5 to 20 ethylene oxide radicals per molecule and 8 to 18 carbon atoms in the fatty alcohol moiety, condensation products of ethylene oxide/propylene oxide, polyvinyl pyrrolidones, polyvinyl alcohols, condensation products of urea and formaldehyde, and also latex products.

The water-dispersible concentrates of the active substance, i.e. wettable powders, pastes and emulsifiable concentrates, are agents which can be diluted with water to any concentration desired. They consist of active substance, extender, optionally additives which stabilize the active substance, surface-active substance and anti-foam agents and, optionally, solvents.

Wettable powders and pastes may be obtained by mixing and grinding the active substance with dispersing agents and pulverulent extenders in suitable apparatus until homogeneity is attained. Suitable extenders are, for example, those mentioned for the solid forms of application. In some cases it is advantageous to use mixtures of different extenders. As dispersing agents there can be used, for example, condensation products of sulfonated naphthalene and sulfonated naphthalene derivatives with formaldehyde, condensation products of naphthalene or naphthalene sulfonic acids with phenol and formaldehyde, as well as alkali, ammonium and alkaline earth metal salts of lignin sulfonic acid, in addition, alkylaryl sulfonates, alkali and alkaline earth metal salts of dibutyl naphthalene sulfonic acid, fatty alcohol sulfates such as salts of sulfated hexadecanols, heptadecanols, octadecanols, and salts of sulfated fatty alcohol glycol ethers, the sodium salt of oleoyl ethionate, the sodium salt of oleoyl methyl tauride, ditertiary acetylene glycols, dialkyl dilauryl ammonium chloride and fatty acid alkali and alkaline earth metal salts.

Suitable anti-foam agents are silicones.

The active substance may be mixed, ground, sieved and strained with the additives cited hereinabove in such a manner that, in wettable powders, the solid particle size of from 0.02 to 0.04 mm and in pastes of 0.03 mm is not exceeded. To produce emulsifiable concentrates and pastes, dispersing agents such as those cited above, organic solvents and water are used. Examples of suitable solvents are: alcohols, benzene, xylene, toluene, dimethyl sulfoxide, and mineral oil fractions boiling between 120 and 350°C. The solvents must be practically odourless, not phytotoxic, and inert to the active substance.

Furthermore, the compound of formula I can be applied in the form of a solution. For this purpose the compound of formula I is dissolved in a suitable organic solvent, mixtures of solvents or in water. Aliphatic and aromatic hydrocarbons, chlorinated derivatives thereof, alkyl naphthalenes, and mineral oils singly or in admixture with each other, can be used as organic solvents.

The content of active substance in the above described compositions is generally 0.1% to 95%, in which connection it should be mentioned that, in the case of application from aircraft or some other suitable means of application, it is possible to use concentrations of up to 99.5% or even pure active substance. (Throughout this specification parts and percentages are by weight unless otherwise specified.)

The active substances of the formula I can, for example, be formulated as follows:

Dusts

The following substances are used to manufacture a) a 5% and b) a 2% dust:

- a) 5 parts of active substance of formula I
95 parts of talcum
- b) 2 parts of active substance of formula I
1 part of highly disperse silica
97 parts of talcum.

The active substance is mixed with the extender(s) and ground.

Granulate

The following substances are used to produce 5% granulate:

- 5 parts of active substance of formula I,
0.25 parts of epichlorohydrin,
0.25 parts of cetyl polyglycol ether,
3.50 parts of polyethylene glycol,
91 parts of kaolin (particle size 0.3—0.8 mm).

The active substance is mixed with epichlorohydrin and dissolved with 6 parts of acetone; the polyethylene glycol and cetyl polyglycol ether are then added. The thus obtained solution is sprayed on to kaolin, and the acetone subsequently evaporated in vacuo.

Wettable powder:

The following constituents are used for the preparation of a) a 40%, b) and c) a 25%, and d) a 10% wettable powder:

- a) 40 parts of active substance of formula I
5 parts of sodium lignin sulphonate,
1 part of sodium dibutyl-naphthalene sulphonate,
54 parts of silica acid.
- b) 25 parts of active substance of formula I,
4.5 parts of calcium lignin sulphonate,
1.9 parts of Champagne chalk/hydroxyethyl cellulose mixture (1:1),
1.5 parts of sodium dibutyl naphthalene sulphonate,
19.5 parts of silica acid,
19.5 parts of Champagne chalk,
28.1 parts of kaolin.
- c) 25 parts of active substance of formula I,
2.5 parts of isooctylphenoxy-polyoxyethylene-ethanol,
1.7 parts of Champagne chalk/hydroxyethyl cellulose mixture (1:1),
8.3 parts of sodium aluminium silicate,
16.5 parts of kieselguhr,
46 parts of kaolin.
- d) 10 parts of active substance of formula I,
3 parts of a mixture of the sodium salts of saturated fatty alcohol sulphates,
5 parts of naphthalenesulphonic acid/formaldehyde condensate,
82 parts of kaolin.

The active substance is intimately mixed, in suitable mixers, with the additives, the mixture being then ground in the appropriate mills and rollers. The wettable powder obtained can be diluted with water to give a suspension of the desired concentration.

Emulsifiable concentrate:

The following substances are used to produce a 25% emulsifiable concentrate:

- 25 parts of active substance of formula I,
2.5 parts of epoxidised vegetable oil,
10 parts of an alkylarylsulphonate/fatty alcoholpolyglycol ether mixture,
5 parts of dimethylformamide,
57.5 parts of xylene.

From this concentrate it is possible to produce, by dilution with water, emulsions of desired concentration.

Spray:

The following constituents are used to prepare a 5% spray:

- 5 parts of active substance of formula I,
1 part of epichlorohydrin,
94 parts of benzine (boiling limits 160°—190°C).

In addition the compositions of the present invention may contain other biologic-

5

2

14

1

15

1

20

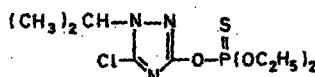
24

2

34

123

3



4

1

4

•

1

2

At 80—100% kill after the first control, a fresh infestation with 5 larvae each

5

5

Action against ticks

10

15

15

20

20

EXAMPLE 4

Acaracidal action

25

25

30

30

35

35

40

40

45

- 15



(wherein Hal represents a halogen atom), in the presence of an acid binding agent, with the compound of formula III:



5

5

Action against ticks

10

10

15

20

20

Acaracidal action

25

30

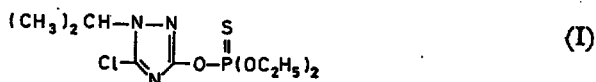
35

35

40

45

45

$$\text{(CH}_3\text{)}_2\text{CH-N-N} \begin{array}{c} \text{S} \\ \parallel \\ \text{O-P(OC}_2\text{H}_5\text{)}_2 \end{array} \begin{array}{c} \text{Cl} \\ \diagup \quad \diagdown \\ \text{N} \end{array} \quad \text{(I)}$$


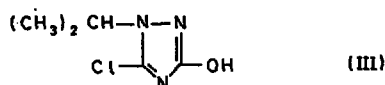
(wherein Hal represents a halogen atom), in the presence of an acid binding agent, with the compound of formula III:



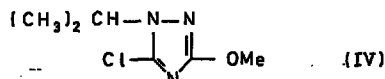
1,419,132

7

7



3. Process for preparing the compound claimed in claim 1 which process comprises reacting a compound of formula II as defined in claim 2 with a compound of formula IV:



(wherein Me represents a monovalent metal atom).

4. Process according to claim 3 wherein, in the compound of formula IV, Me represents an alkali metal atom.

5. Process according to claim 4 wherein Me represents a sodium or potassium atom.

6. Process according to any one of claims 2 to 5 wherein, in the compound of formula II, Hal represents a chlorine or bromine atom.

7. Process for preparing the compound claimed in claim 1 substantially as described in Example 1.

8. Compound according to claim 1 prepared by the process claimed in any one of claims 2 to 7.

9. A pesticidal composition which comprises, as active ingredient, the compound claimed in claim 1 together with a solid extender, a solid extender and a surface active agent or a liquid diluent and a surface active agent.

10. A solid composition according to claim 9.

11. A composition according to claim 10 in the form of a granulate.

12. A composition according to claim 11 wherein the granules are coated or impregnated with the active ingredient.

13. A liquid composition according to claim 9.

14. A pesticidal composition containing the compound claimed in claim 1 and in the form of a dust, granulate, wettable powder, emulsifiable concentrate or spray specifically described herein.

15. Method of combating insects, nematodes or acarina which method comprises applying to a locus infested with or liable to be infested with the insects nematodes or acarina the compound claimed in claim 1.

16. Method according to claim 15 wherein the insects, nematodes or acarina combated are harmful to seeds, tubers, plants or plant parts.

17. The compound of formula I defined in claim 1 in a container which bears instructions to use the compound in combating insects, nematodes or acarina.

Agents for the Applicants,
GALLAFENT & CO.,
Chartered Patent Agents,
8, Staple Inn,
London, WC1V 7QH.

Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1975.
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from
which copies may be obtained.