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NOTICE OF ENTITLEMENT

I, John David O'Connor, of 31 Market Street, Sydney, New South Wales, 2000, Australia, patent attorney for the Applicant/Nominated Person in respect of Application No. 73856/91 state the following:-

The Applicant/Nominated Person has entitlement from the actual inventors as follows:-

The Applicant/Nominated Person is the assignee of the actual inventors.

The Applicant/Nominated Person is the applicant of the basic application listed on the Patent Request.

The basic application listed on the Patent Request is the application first made in a Convention Country in respect of the invention.

Dated 28 September 1992

John David O'Connor

IRN: 156720

INSTR CODE: 52760

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Patent Request: Standard Patent

We, the Applicant/Nominated Person specified below, request we be granted a patent for the invention disclosed in the accompanying standard complete specification.

[70,71] **Applicant/Nominated Person:**
Ciba-Geigy AG, of Klybeckstrasse 141, 4002 Basel, Switzerland

[54] **Invention Title:**
Liquid Pesticide Concentrates

[72] **Inventors:**
Guy Dez, Jacques Lerivrey, Renè Schneider, Alexander Zurkinden and Carolyn Moore

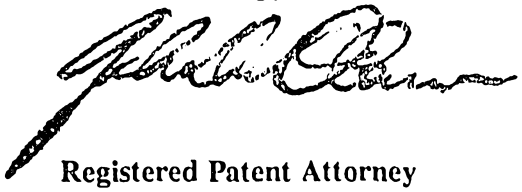
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Basic Convention Application Details

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Basic Applicant: Ciba-Geigy AG

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LIQUID PESTICIDE CONCENTRATES

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(56) Prior Art Documents
AU 643423 38403/89 B65D 65/38
AU 642855 38396/89 B65D 65/38
AU 642853 38375/89 B65D 65/38

(57) Claim

1. An agrochemical composition in packaged form, which comprises a water-soluble package; a liquid, non-aqueous concentrate having a viscosity of 1000 to 20 000cp and comprising an agrochemical and a viscosity increasing substance comprising a water-soluble polymer, a hydrophilic silic acid, a surfactant, or a mixture thereof.

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FORM 10

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COMPLETE SPECIFICATION

(ORIGINAL)

FOR OFFICE USE:

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Complete Specification Lodged:
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Related Art:

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Complete Specification for the invention entitled:

Liquid Pesticide Concentrates

The following statement is a full description of this invention, including the best method of performing it known to me/us

5845/6

Liquid pesticide concentrates

Abstract of the Disclosure

There are disclosed active ingredient concentrates in packaged form, which comprise a water-soluble bag and a liquid, non-aqueous pesticide concentrate.

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Liquid pesticide concentrates

The present invention relates to liquid pesticide concentrates on a non-aqueous basis, which are sealed in a bag made of water-soluble plastic film.

Pesticides are normally marketed in the form of concentrates which, before use, are diluted with water to spray mixtures. When handling the concentrates, the toxicity of the active ingredients makes it necessary to take steps to prevent the user coming in contact with the concentrates. In particular, the user must be protected from inhaling the dust arising from handling solid formulations such as powders and granules, and from skin contact with liquid formulations such as emulsifiable concentrates. The manufacturers of pesticide concentrates therefore recommend taking a number of precautions, such as wearing protective masks, suits and gloves.

To secure further improved protection for the user, the proposal has already been made to avoid the dust formation associated with handling solid pesticide concentrates and skin contact with liquid concentrates by packaging these concentrates in a bag made from water-soluble plastic film. The spray mixture is prepared from such concentrates packed in water-soluble material by putting the concentrate and its packaging into the amount of water necessary for preparing the spray mixture. The spray mixture forms immediately after dissolution of the packaging and dispersion of the contents. Direct contact of the user with the concentrate is thereby avoided. Moreover, packaging concentrates in water-soluble packaging materials has the advantage that the concentrates can be removed from the containers in which they are transported and stored to protect them from moisture without leaving any residues which would make it necessary to clean the containers. In addition, it is much easier to control the dosage of the active ingredient, as each bag contains a defined amount of active ingredient.

The water-soluble bags of liquid active ingredient concentrates of the prior art have, however, a relatively high susceptibility to shock and impact, so that, when subjected to sudden mechanical stress, for example on falling to the ground, the bags burst. Not only do these unfavourable properties lead in the transportation and handling of such bags to

constant loss and to contamination of the environment and/or of the user, but also to the loss of the advantages referred to above of the use of water-soluble packaging materials.

Hence it is the object of this invention to provide pesticide concentrates in packaged form which comprise a water-soluble packaging material and a liquid, non-aqueous pesticide concentrate, and which have increased stability to impact and shock.

According to a first embodiment of the present invention there is provided an agrochemical composition in packaged form, which comprises a water-soluble package; a liquid, non-aqueous concentrate having a viscosity of 1000 to 20 000cp and comprising an agrochemical and a viscosity increasing substance comprising a water-soluble polymer, a hydrophilic silic acid, a surfactant, or a mixture thereof.

In the simplest case, the pesticide concentrates of this invention are solutions of one or more active ingredients in a non-aqueous solvent, the viscosity of which concentrates has been adjusted 100-20 000 cp by addition of a viscosity increasing substance. The pesticide concentrates may also, however, be solutions of one or more active ingredients in a non-aqueous solvent, in which additionally one or more active ingredients which are insoluble in said solvent are suspended, the viscosity of which suspensions have also been adjusted to 1000-20 000 cp by addition of a viscosity increasing substance. Finally, the concentrates of the invention may also be suspensions of one or more active ingredients in a non-aqueous solvent, the viscosity of which suspensions have been adjusted to 1000-20 000 cp by addition of a viscosity increasing substance.

In addition to comprising one or more active ingredients, the solvent and the viscosity increasing substance, the concentrates of this invention may advantageously contain one or more dispersants. The addition of one or more dispersants is especially expedient if the active ingredient and/or the solvent and/or the viscosity increasing substance are insoluble in water and it is necessary to form a dispersion after diluting the concentrate with water.

Suitable non-aqueous solvents are the solvents which are customarily used in pesticide formulation and which are inert to the water-soluble packaging material, viz. those solvent which neither dissolve the water-soluble packaging material nor in which it swells or otherwise adversely affect it. With respect to this limitation, it must be borne in mind that solvents generally tend all the less to dissolve or cause the packaging material to swell the greater the concentration of the substances dissolved therein is. Suitable solvents are alcohols, alkanediols (glycols), diethylene glycol monoethers, ketones, esters, aliphatic and aromatic hydrocarbons, as well as mineral and vegetables oils. Particularly suitable

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alcohols are C_1-C_8alkanols, preferably C_3-C_6alkanols and C_5-C_6cycloalkanols. Particularly suitable alkanediols are ethylene glycol and propylene glycol. Particularly suitable diethylene glycol monoethers are diethylene glycol monomethyl ether und dipropylene glycol monomethyl ether. Particularly suitable ketones are C_4-C_8alkanones and C_5-C_6cycloalkanones. Suitable esters are preferably C_3-C_6alkylacetates and lactones of C_4-C_5hydroxyalkanecarboxylic acids. Particularly suitable aliphatic and aromatic hydrocarbons are hexane, cyclohexane, toluene, xylene, as well as mixtures of the alkylbenzenes which are commercially available under the registered trademarks ®Solvesso and ®Shellsol. Particularly suitable mineral oils are higher boiling petroleum fractions. Suitable vegetable oils are castor oil, rape oil, rape oil esters, cotton seed oil and soybean oil. Particularly suitable solvents are cyclohexanol, diacetone alcohol, cyclohexanone, butyrolactone, amyl acetate, toluene, xylene, as well as mixtures of alkylbenzenes having a boiling range of 130-170°C.

Suitable viscosity increasing substances for the purposes of the invention are preferably water-soluble polymers and hydrophilic silicic acid. Suitable water-soluble polymers are typically cellulose derivatives, proteins (gelatin) and polyvinylpyrrolidone having a molecular weight of 600 000-1000 000. In individual cases it is also possible to use surfactants, such as ethylene oxide-propylene oxide block polymers or dodecylbenzenesulfonates, as viscosity increasing substances. Preferred viscosity increasing substances are cellulose derivatives, such as methyl cellulose, ethyl cellulose, hydroxyethyl cellulose and hydroxypropyl cellulose.

The dispersants which may be suitably used are basically all nonionic, anionic and cationic dispersants customarily used in the art of pesticide formulation. It is preferred to use nonionic or anionic dispersants or mixtures of nonionic and anionic dispersants. Particularly suitable nonionic dispersants are ethylene oxide-propylene oxide block polymers, alkylphenol polyglycol ethers, as well as fatty acid and fatty alcohol polyglycol ethers. Particularly suitable anionic dispersants are salts of alkylbenzenesulfonic acids, for example calcium dodecylbenzenesulfonate, and also salts of mono- and diphenol polyglycol ether phosphates, as well as salts of monophenol polyglycol ether sulfates. Particularly suitable mixtures of anionic and nonionic dispersants are mixtures of alkylphenol polyglycol ethers and salts of mono- and diphenol polyglycol ether phosphates.

By water-soluble packaging materials are meant in the context of this invention preferably

films made of water-soluble polymers, for example of polyvinyl alcohol, polyvinyl pyrrolidone, copolymers of vinyl alcohol and vinyl pyrrolidone, copolymers of vinyl alcohol and vinyl acetate, as well as cellulosic films, for example carboxymethyl cellulose films. Polyvinyl alcohol films are the preferred packaging materials.

The active ingredient concentrates of this invention may in principle contain any agrochemical control agents, for example insecticides, acaricides, fungicides, herbicides and plant growth regulating substances.

The pesticide concentrates of this invention can be prepared by dissolving or suspending one or more active ingredients in a solution of the dispersant in a non-aqueous solvent, and then incorporating the viscosity increasing substance in the resultant mixture. The preparation of the concentrates can be carried out in a conventional mixing apparatus. If the individual components of the mixture are water-soluble, the addition of dispersant can be dispensed with, for in this case an aqueous solution is obtained direct from the concentrate upon dilution with water. If, however, one or more components of the concentrate are water-insoluble, then it is advantageous to add one or more dispersants in order to ensure the rapid and homogeneous dispersion of the components in water. The concentrate can then be filled immediately in equal portions into bags which can be made in known manner from films consisting of the polymers cited above. After they have been filled with the concentrate, the bags are sealed. The bags so obtained contain a defined amount of active ingredient per unit dose package. The individual bags can then be packed for transportation and storage individually or collectively in containers, for example cardboard, polyethylene, polypropylene or polyvinyl chloride containers, from which they can be readily removed immediately before use.

The bags of this invention are conveniently prepared by continuously feeding a web of water-soluble material 100-300 mm wide and 30-40 µm thick to a filling machine in which the web is first welded with a straight bead to a tubular film which is sealed at its end by a transverse weld. After being filled with the desired amount of active ingredient concentrate, the bag is then sealed with a severing weld, so as to obtain, on the one hand, a ready-for-use bag containing the active ingredient concentrate and, on the other, a new bag preformed from the tubular film into which the next portion of active ingredient concentrate can be filled. The welds can be made by heat welding at a temperature of ca. 300°C as well as by pulse welding or high-frequency welding.

The handling and application of the pesticide concentrates of this invention is exceedingly simple. One or more bags are taken from the container and put into a spray tank filled with water in which the bag made of water-soluble packaging material dissolves in a few minutes, and the concentrate disperses in the water to form a spray mixture. Not only are the previously known advantages of water-soluble bags thereby attained, namely avoidance of direct contact of the user with the concentrate, ready metering of the active ingredients without using graduated jars, and avoidance of contamination of the packing containers, but, in addition, the mechanical stability of the bags, especially the stability to sudden mechanical stress, is substantially increased in comparison with prior art bags of this kind containing liquid pesticide concentrates. Only by increasing the stability to sudden mechanical stresses is it possible to utilise fully the advantages accruing from the use of water-soluble packaging material.

The following Examples illustrate the invention in more detail.

A number of typical formulations of pesticide concentrates of this invention are illustrated below, without thereby implying any restriction of the present invention. Percentages are by weight. The preparation of the packs is also described.

Example 1

Propiconazole techn.	62.5	%
calcium dodecylbenzenesulfonate	4.5	%
castor oil polyglycol ether (EO 36)	4.0	%
isooctylphenol polyglycol ether (EO 8)	2.0	%
alkylbenzene C10-C13, to make up	100	%

viscosity: 11000 cp

Example 2

Propiconazole techn.	65.0	%
PO-EO block polymer (80 % EO; $\overline{MG}$: 8550)	4.5	%
castor oil polyglycol ether (EO 36)	8.8	%
sodium diamylsulfosuccinate	4.6	%

n-butanol, to make up	100	%
viscosity: 5000 cp		

Example 3

Propiconazole techn.	62.5	%
calcium dodecylbenzenesulfonate	3.0	%
isotridecyl alcohol polyglycol ether (EO 6)	3.0	%
EO-PO block polymer (20 % EO; $\overline{MG}$: 5000)	4.0	%
silicone oil	0.2	%
hydroxypropyl cellulose	1.0	%
cyclohexanone, to make up	100	%
viscosity: 2000 cp		

Example 4

Propiconazole techn.	62.5	%
calcium dodecylbenzenesulfonate	3.0	%
isotridecyl alcohol polyglycol ether (EO 6)	3.0	%
EO-PO block polymer (20 % EO; $\overline{MG}$: 5000)	4.0	%
silicone oil	0.2	%
hydroxypropyl cellulose	1.0	%
dipropylene glycol monomethyl ether, to make up	100	%
viscosity: 2000 cp		

Example 5

Propiconazole techn.	62.5	%
tristyrylphenol polyglycol ether (EO 20)	8.0	%
nonylphenol polyglycol ether (EO 8)	8.0	%
nonylphenol polyglycol ether (EO 8-10) phosphate	2.0	%

silicone oil	0.2	%
hydroxypropylcellulose	0.72	%
cyclohexanone, to make up	100	%
viscosity: 2400 cp		

Example 6

Penconazole techn.	25.0	%
propylene glycol	7.0	%
isotridecyl alcohol polyglycol ether (EO 6)	3.0	%
calcium dodecylbenzenesulfonate	4.0	%
EO-PO block polymer (20 % EO; $\overline{MG}$: 5000)	10.0	%
silicone oil	0.05	%
hydrophilic silicic acid	3.5	%
castor oil, to make up	100	%
viscosity: 5000 cp		

Example 7

Phosphamidon techn.	65.6	%
calcium dodecylbenzenesulfonate	1.6	%
polyglycerol phthalate	1.6	%
oleyl alcohol polyglycol ether (10 EO)	3.0	%
silicone oil	0.08	%
hydroxypropyl cellulose	0.82	%
dipropylene glycol monomethyl ester, to make up	100	%
viscosity: 3000 cp		

Beispiel 8

Monocrotophos techn.	60.6	%
hydroxypropylcellulose	4.0	%
dipropylene glycol methyl ether, to make up	100	%
viscosity: 3000 cp		

Example 9

propargyl 2-[4-(5-chloro-5-fluoropyrid-2-yloxy)phenoxy]-propioniate	24.0	%
5-chloro-8-(1-methylhexyloxycarbonylmethoxy)-quinoline	6.0	%
N-methylpyrrolidone	5.0	%
calcium dodecylbenzoate	7.0	%
nonylphenol polypropylene(13)polyethylene(20)-glycol	13.0	%
hydrophilic silicic acid	2.1	%
ethyl cellulose	2.1	%
anisole, to make up	100	%

viscosity: 2000 cp

Example 10: A web of polyvinyl alcohol (manufacturer: Syntana Handelsgesellschaft GmbH, Mülheim-Ruhr, FRG), 200 mm wide and 35 µm thick, is fed continuously to a filling machine (manufacturer: NEDI S.A., Saint-Mammes, France) in which it is welded by pulse welding with a straight bead to a tube which is sealed at its end with a transverse weld. After being filled with 100 g of active ingredient according to Example 3, the preformed bags so obtained are then separated so as to give sealed bags 135 mm in length which contain the active ingredient concentrate.

The bags so obtained dissolve in 25-200 l of water of 15-20°C in ca. 1 minute to give a ready-for-use spray mixture.

The claims defining the invention are as follows:

1. An agrochemical composition in packaged form, which comprises a water-soluble package; a liquid, non-aqueous concentrate having a viscosity of 1000 to 20 000cp and comprising an agrochemical and a viscosity increasing substance comprising a water-soluble polymer, a hydrophilic silic acid, a surfactant, or a mixture thereof.

2. A composition according to claim 1 wherein said package comprises a film made from a water-soluble polymer.

3. A composition according to claim 1 or claim 2 wherein said package comprises methylcellulose, ethylcellulose, hydroxyethylcellulose, hydroxypropylcellulose, or a mixture of any two or more thereof.

4. A composition according to claim 1 or claim 2, wherein said package comprises a film made from polyvinyl alcohol, polyvinylpyrrolidone, a copolymer of vinyl alcohol and vinylpyrrolidone, a copolymer of vinyl alcohol and vinyl acetate, or is a cellulosic film.

5. A composition according to claim 4, wherein said package comprises a polyvinyl alcohol film.

6. A composition according to any one of claims 1 to 5, wherein said agrochemical is an insecticide, acaricide, fungicide, herbicide, plant growth regulating substance, pesticide, or a mixture of any two or more thereof.

7. A composition according to any one of claims 1 to 6, wherein said agrochemical is propiconazole.

8. A concentrate according to any one of claims 1 to 7, wherein said viscosity increasing substance is a cellulose derivative, protein (gelatin) or polyvinylpyrrolidone having a molecular weight of 600 000 to 1 000 000.

9. A concentrate according to any one of claims 1 to 8, wherein said surfactant is an ethylene-propylene oxide block copolymer, a dodecylbenzenesulfonate or a mixture thereof.

10. A concentrate according to claim 9, wherein said dodecylbenzenesulfonate is calcium dodecylbenzenesulfonate.

11. A composition according to any one of claims 1 to 10, wherein said concentrate further comprises a solvent which is inert to said water-soluble package.

12. A composition according to claim 11, wherein said solvent is an alcohol, alkanediol (glycol), diethyleneglycol monoether, ketone, ester, aliphatic and aromatic hydrocarbon, mineral or vegetable oil, or a mixture of any two or more thereof.

13. A composition according to claim 11, wherein said solvent is cyclohexanol, diacetone alcohol, cyclohexanone, butyrolactone, amyl acetate, toluene, xylene, a mixture of alkylbenzenes having a boiling range of 130-170°C, or a mixture of any two or more thereof.

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14. A composition according to any one of claims 1 to 13, which further comprises one or more dispersants.

15. An agrochemical composition in packaged form, substantially as hereinbefore described with reference to any one of the Examples.

Dated 22 October, 1993
Ciba-Geigy AG

Patent Attorneys for the Applicant/Nominated Person
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