

INSECTICIDAL AEROSOL SPRAY COMPOSITIONS**Technical Field**

The present invention relates to insecticidal aerosol spray compositions.

5 Background Art

A number of methods are known to deal with insects like cockroaches. One is the use of baits and traps. Another method is the use of insecticidal aerosol sprays.

10 The present invention relates to insecticidal aerosol spray compositions that have been developed to kill cockroaches, ants, fleas, silverfish, spiders, clothes moth, carpet beetle, Portuguese millipede and bed bugs.

One of the advantages of the aerosol spray is that it can be sprayed directly onto the insect for a fast kill.

15 A further advantage is that it can be sprayed onto a surface to produce long lasting barrier protection. For example, to keep cockroaches at bay, the insecticidal aerosol composition can be sprayed on to skirting boards, under cupboards, stoves and drawers.

20 The term "insecticide" is well known in the art and includes orthodox chemical insecticides and appropriate insect virus, bacterial or hormone compositions able to affect the specific insect species under attack. It has also been found that certain insecticides exhibit a repellence to insects. In particular, the present inventors have found that the pyrethrin, pyrethroid, cyanopyrethroid and others (e.g. the carbamates) class of
25 insecticide tends to have a repellent effect on insects, such as cockroaches.

This is clearly undesirable for surface applications and means of overcoming this defect has needed to be found to provide a more effective insecticidal aerosol spray.

30 The present inventors tested a number of insect attractants. On the whole, these attractants did not significantly mask the negative effect of the repellency of the insecticide. It was surprisingly found, however, that three attractants were significantly better than the others to enable a commercially acceptable insecticidal aerosol spray to be produced.

35 Given the volatile nature of some of the attractants used, it would be expected that once compositions of the invention had been sprayed onto a surface, attractancy would diminish rapidly with time. In fact it was

expected that attractancy would last no longer than about two weeks. Contrary to expectations, attractancy is maintained for a substantial period of time, in particular for at least 13 weeks.

Maintenance of this attractancy property is particularly beneficial in an insecticidal surface spray as it will produce prolonged effective insecticidal activity.

Disclosure of the Invention

According to the present invention there is provided an insect-attracting insecticidal aerosol spray composition containing an insect-attracting effective amount of 1 to 10% w/w of lauric acid, d-limonene, orange oil or mixtures thereof, wherein the concentration of lauric acid when present is 1% w/w or more.

In a preferred form, the insecticidal aerosol composition of the present invention which when sprayed onto a surface maintains an insecticidal and insect-attractancy for a period of 13 weeks or more.

Further according to the present invention there is provided the use of lauric acid, d-limonene, orange oil or mixtures thereof as an attractant in an insect-attracting insecticidal aerosol spray composition, wherein the concentration of lauric acid when present is 1% w/w or more.

Still further according to the present invention there is provided an insect-attracting insecticidal aerosol spray composition comprising:

- (a) 0.01 to 5% w/w of one or more insecticides;
- (b) 10 to 90% w/w of a solvent;
- (c) 10 to 90% w/w of a propellant; and
- (d) 1 to 10% w/w of an attractant being lauric acid, d-limonene, orange oil or mixtures thereof, wherein the concentration of lauric acid when present is 1% w/w or more.

Still further according to the present invention there is provided an insect-attracting insecticidal aerosol spray composition comprising:

- (a) 0.01 to 5% w/w of one or more insecticides;
- (b) 10 to 90% w/w of a solvent;
- (c) 2 to 10% w/w of a compressed gas propellant; and
- (d) 1 to 10% w/w of an attractant being lauric acid, d-limonene, orange oil or mixtures thereof, wherein the concentration of lauric acid when present is 1% w/w or more.

(a) Insecticide

Preferred insecticides are killer active insecticides, preferably of the pyrethrin, pyrethroid, cyanopyrethroid and others (e.g. the carbamates) class of insecticide, more preferably cyanopyrethroids, and most preferably cypermethrin (which is (RS)-alpha-cyano-3-phenoxybenzyl (1RS)-cis,trans-3-(2,2-dichlorovinyl)-2,2-dimethyl-cyclopropane carboxylate, commercially available from ICI) or tetramethrin (which is 3,4,5,6-tetrahydro-phthalidimomethyl (1RS) cis,transchrysanthemate, commercially available from Sumitomo) or mixtures thereof.

The one or more insecticides are preferably present in an amount of 0.05 to 3.0%, more preferably 0.2 to 0.6% by weight of the composition.

Advantageously the insecticides are ones that are insect repelling.

(b) Solvent

Preferably the solvent is selected from isoparaffins and normal paraffins (e.g. C₁₋₁₂ alkanes) or kerosene. An example of a commercially available solvent is a C₁₂ alkane (which is commercially available).

Preferably the solvent is present in an amount from 10 to 90%, more preferably 60 to 70% by weight of the composition.

(c) Propellant

Preferably the propellant is a hydrocarbon propellant, a dimethylether propellant or a compressed gas propellant.

The preferred hydrocarbon propellants are selected from methane, ethane, propane, n-butane, isobutane, pentane and isopentane.

Preferably the hydrocarbon or dimethylether propellant is present in an amount of 10 to 90%, more preferably about 10 to 50%, even more preferably 25 to 45%, and especially about 40% by weight of the composition.

When compressed gases are used as a propellant generally these will be carbon dioxide, nitrogen or air. It will be appreciated by one skilled in the art that lower concentrations of compressed gas propellants are required compared to the concentrations required for hydrocarbon or dimethylether propellants. Usually, compressed gas will be used at a concentration of 2 to 10%, and preferably about 5%.

The person skilled in the art will appreciate that the pressure in an aerosol package will be determined by propellant or mixture of propellant. This pressure will have a determining effect on spray rates. Hence for any

particular valve system, varying the propellant or propellant mixture will allow for the selection of a desired spray rate. Likewise for a particular propellant or propellant mixture, it is possible to select a valve and actuator system to achieve a desired rate.

5 (d) Attractants

The attractants suitable for the present invention are lauric acid, d-limonene, orange oil or mixtures thereof. Preferably, the attractant is orange oil.

10 Preferably, the amount of attractant used is from 1 to 10%, more preferably about 1 to 5%, most preferably 2 to 4%, and especially 3% by weight of the composition.

Optional Other Ingredients

15 The aerosol spray compositions according to the invention may further include one or more preservatives such as butylated hydroxytoluene (BHT). When a preservative is present, it is preferably present in an amount of 0.001 to 0.1% by weight based on the composition.

The aerosol spray compositions according to the invention may further include a booster for increasing the residual effect of the aerosol spray composition. A preferred booster is fumed silica powder.

20 When a booster is present, it is preferably present in an amount of 0.05 to 0.2% by weight of the composition.

Examples of preferred silica powder are highly dispersed amorphous hydrophilic silicon dioxide and highly dispersed amorphous hydrophobic silicon dioxide (commercially available).

25 The aerosol spray compositions according to the invention may also include a perfume. If a perfume is present, it is preferably present in an amount of 0.05 to 2% by weight of the composition.

30 The aerosol spray composition can be produced by dissolving the insecticide in the solvent preferably at an elevated temperature (i.e. 40 to 50°C) followed by cooling the mixture. Then this mixture, together with the attractant and one or more of the optional additives selected from preservatives, perfumes and boosters are placed into an empty aerosol can, followed by adding the propellant by normal methods, e.g. by normal hydrocarbon aerosol gassing.

35 In this specification all percentages are by weight, unless indicated to the contrary.

Modes for Carrying Out the Invention

The invention will now be illustrated by the following Examples.

EXAMPLE 1

0.286% of cypermethrin (70%) and 0.381% of tetramethrin (92%) are added to 66.118% of a commercially available C₁₂ alkane. This mixture is heated to 50°C to ensure complete dissolution of the two insecticides in the solvent.

This is then cooled to room temperature. The resultant mixture is then added to an empty aerosol can together with 0.1% of a perfume, 0.005 of BHT, 0.1% of a commercially available highly dispersed amorphous hydrophilic silicon dioxide, 0.01% of a commercially available highly dispersed amorphous hydrophobic silicon dioxide and 3% of orange oil. The aerosol can is then gassed with 30% of a Propellant H-55, a blend of 74% propane and 26% butane (commercially available from Esso) by well-known hydrocarbon gassing techniques.

Example 1 can be repeated using 3.0% d-limonene in place of 3.0% of orange oil.

EXAMPLE 2

Example 1 is repeated using 3.0% lauric acid in place of 3.0% of orange oil.

EXAMPLE 3

Example 1 is repeated as follows:

0.286% w/w of cypermethrin (70%) and 0.381% w/w of tetramethrin (92%) are added to 66.218% of a commercially available C₁₂ alkane. This mixture is heated to 50°C to ensure complete dissolution of the two insecticides.

This is then cooled to room temperature. The resultant mixture is then added to an empty aerosol can together with 0.005% w/w of butylated hydroxy toluene, 0.1% w/w of a commercially available highly dispersed amorphous hydrophobic silicon dioxide, 0.010% w/w of a commercially available dispersed amorphous hydrophobic silicon dioxide and 3% w/w of natural orange oil.

The aerosol can is then gassed with 7.792% of propane and 22.20% of butane by well known hydrocarbon gassing techniques.

COMPARATIVE EXAMPLE A.

0.286% of cypermethrin and 0.381% of tetramethrin are added to 69.118% of a solvent. This mixture is heated to 50°C to ensure complete dissolution of the two insecticides in the solvent.

5 This is then cooled to room temperature. The resultant mixture is then added to an empty aerosol can together with 0.1% of a perfume, 0.005 of BHT, 0.1% of a commercially available highly dispersed amorphous hydrophilic silicon dioxide and 0.01% of a commercially available highly dispersed amorphous hydrophobic silicon dioxide.

10 The aerosol can is then gassed with 30% of a propellant H-55 by well-known hydrocarbon gassing techniques.

COMPARATIVE EXAMPLES B - F

Example 1 is repeated using instead of 3% orange oil, one of the following attractants:

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Example	Attractant
B	3% of pineapple oil
C	3% of banana oil
D	3% of palmitic acid
E	3% of soy bean oil
F	3% of oleyl alcohol

APPLICATION EXAMPLE

Forty mixed age American cockroaches (*Periplaneta americana*) - mixed sex adults and mixed stage nymphs were placed in holding jars. The cockroaches were provided with harbourages and water but no food and left for 60 hours.

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The cockroaches were then placed in the test area for a further 24 hours, with no food but with water and harbourages.

Two containers, with ramps to facilitate the entry of cockroaches into the container, and which are 9 cm in diameter and 9.5 cm high were placed at opposite ends of the test area. Special care was taken not to disturb the cockroaches.

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A 5.5 cm filter disc was sprayed to deposit 1 g of the composition of Example 1 and allowed to dry for two hours after which the filter paper was placed in one of the containers.

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At the same time an untreated 5.5 cm filter disc was placed in the other container.

After 24 hours, the number of cockroaches in each container was counted.

5 Ten such tests were performed and from the results of each of the ten tests, an attractancy index was determined, which is calculated as follows:

$$\text{Attractancy Index} = \frac{\text{Number of cockroaches in treated container}}{\text{Number of cockroaches in untreated container}}$$

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An index of less than 1 is poor as it indicates that the tested product is worse than the control.

15 The Application Example was repeated using 1 g of each of the compositions of Example 2 and Comparative Examples A to F (in the place of 1 g of the composition of Example 1). For the composition of each of Example 2 and Comparative Examples A to F, ten such tests were performed, from which the attractancy index can be calculated. The results can be seen in Table 1 below.

20 Table 1

Test	Attractancy Index
Example 1	1.44
Example 2	1.27
Comparative Example A	0.27
Comparative Example B	0.32
Comparative Example C	0.63
Comparative Example D	0.43
Comparative Example E	0.60
Comparative Example F	0.58

25 As can be seen from the above results, the compositions of Examples 1 and 2 are significantly better at insect attractancy than the control (Comparative Example A) or compositions using other common attractants.

INSECTICIDAL PERFORMANCE

In order to demonstrate the insecticidal performance of the invention, Examples 1 and 2 (as previously described) were evaluated using the following method. For the purposes of comparison, a number of examples of prior art compositions were included.

Method

Explanation of Methodology:

In this study, a treated plate, 100 cm² in area was placed in a base arena 2,828 cm² in area. As the area of the plate was only approximately 3.5% of the total base area, cockroaches were not forced to walk across the plate and it could be easily avoided when obtaining food or water. Any cockroaches which were knocked down in the arena came into contact with the treated plate by choice. The addition of a successful attractant should either reduce the repellency of the surface spray or possibly attract the cockroaches onto the treated surface resulting in a higher percentage knockdown for that formulation.

A. Treatment Method

1. A 10 cm x 10 cm (100 cm² area) masonite plate was placed in the centre of a 0.25m² grid. A can of each example was held 20 cm from the grid and the entire area was sprayed for a period of 8 seconds (8 horizontal passes).
2. Control plates consisted of untreated masonite plates as above.
3. All plates were stored vertically in racks in an air conditioned laboratory at 22±2°C. The laboratory was fitted with windows so the plates were subject to a normal day/night cycle.

B. Bioassay Method

1. Twenty American cockroaches (5 adults and 15 mid to late stage nymphs) were placed in a large plastic arena (base area 2,828 cm² and height 46 cm). The sides of the arena were coated in fluon to prevent the cockroaches escaping.
2. The cockroaches were provided with a harbourage in the form of a roll of corrugated cardboard, food and a water source.
3. The cockroaches were left in each arena for 24 hours to acclimatise.
4. At 13 weeks post treatment, a treated masonite plate was carefully placed in each arena. The plate was placed on the opposite side of

the arena to the harbourage. The cockroaches were not disturbed when the plate was placed in the arena.

5. Knockdown of cockroaches was noted at 24, 48 and 72 hours after placement of the plates. The temperature during the test was $22\pm 2^{\circ}\text{C}$.

6. There were 5 replicates of each of each example and control. The placement of corrugated cardboard, food, water and plates were identical for each of the treatment and control replicates.

10 Table 2

Test	Percentage Knockdown of Cockroaches at Various Times After Plate Introduction		
	24 hours	48 hours	72 hours
Example 1	36	54	54
Example 2	24	28	38
Prior Art Comparison A	14	21	19
Prior Art Comparison B	4	7	9
Prior Art Comparison C	15	16	23
Control	2	2	2

15 The results shown in Table 2 clearly indicate that both Examples 1 and 2 showed superior knockdown to all other examples tested. Given that the assessment was performed 13 weeks after initial treatment, it is evident that these examples of the invention demonstrate that insecticidal performance is maintained over a prolonged period as a result of their attractancy.

20 It will be appreciated by persons skilled in the art that numerous variations and/or modifications may be made to the invention as shown in the specific embodiments without departing from the spirit or scope of the invention as broadly described. The present embodiments are, therefore, to be considered in all respects as illustrative and not restrictive.



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Claims

1. An insect-attracting insecticidal aerosol spray composition containing an insect-attracting effective amount of 1 to 10% w/w of lauric acid, d-limonene, orange oil or mixtures thereof, wherein the concentration of lauric acid when present is 1% w/w or more.
- 5 2. An insecticidal composition according to claim 1 containing a mixture of d-limonene and orange oil wherein the final concentration of the mixture is 1 to 10% w/w.
3. An insecticidal composition according to claim 1 or 2 which when
10 sprayed onto a surface maintains an insecticidal and insect-attractancy for a period of 13 weeks or more.
4. The use of lauric acid, d-limonene, orange oil or mixtures thereof as an attractant in an insect-attracting insecticidal aerosol spray composition, wherein the concentration of lauric acid when present is 1% w/w or more.
- 15 5. An insect-attracting insecticidal aerosol spray composition comprising:
 - (a) 0.01 to 5% w/w of one or more insecticides;
 - (b) 10 to 90% w/w of a solvent;
 - (c) 10 to 90% w/w of a propellant; and
 - 20 (d) 1 to 10% w/w of an attractant being lauric acid, d-limonene, orange oil or mixtures thereof, wherein the concentration of lauric acid when present is 1% w/w or more.
6. An insecticidal composition according to claim 5 wherein the propellant is selected from the group hydrocarbon and dimethylether.
- 25 7. An insecticidal composition according to claim 6 wherein the hydrocarbon propellant is selected from the group consisting of from methane, ethane, propane, n-butane, isobutane, pentane, isopentane, and mixtures thereof.
8. An insecticidal composition according to any one of claims 5 to 7
30 wherein the concentration of the propellant is 10 to 50% w/w.
9. An insecticidal composition according to claim 8 wherein the concentration of the propellant is 25 to 45% w/w.
10. An insecticidal composition according to claim 9 wherein the concentration of the propellant is 40% w/w.

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11. An insect-attracting insecticidal aerosol spray composition comprising:
- (a) 0.01 to 5% w/w of one or more insecticides;
- (b) 10 to 90% w/w of a solvent;
- 5 (c) 2 to 10% w/w of a compressed gas propellant; and
- (d) 1 to 10% w/w of an attractant being lauric acid, d-limonene, orange oil or mixtures thereof, wherein the concentration of lauric acid when present is 1% w/w or more.
12. An insecticidal composition according to claim 11 wherein the
- 10 compressed gas propellant is selected from the group consisting of carbon dioxide, nitrogen, and air.
13. An insecticidal composition according to claim 11 or 12 wherein the concentration of the propellant is 5% w/w.
14. An insecticidal composition according to any one of claims 5 to 13
- 15 wherein the one or more insecticides are selected from the group consisting of pyrethrin, pyrethroid, cyanopyrethroid and carbamate class of insecticide.
15. An insecticidal composition according to claim 14 wherein the one or more insecticides are cyanopyrethroids.
16. An insecticidal composition according to claim 15 wherein the
- 20 cyanopyrethroids are selected from the group consisting of cypermethrin ((RS)-alpha-cyano-3-phenoxybenzyl (1RS)-cis,trans-3-(2,2-dichlorovinyl)-2,2-dimethyl-cyclopropane carboxylate), tetramethrin (3,4,5,6-tetrahydro-phthalidimomethyl (1RS) cis ,transchrysanthemate), and mixtures thereof.
17. An insecticidal composition according to any one of claims 5 to 16
- 25 wherein the one or more insecticides are insect repelling.
18. An insecticidal composition according to any one of claims 5 to 17 wherein the concentration of the one or more insecticides is 0.05 to 3.0% w/w.
19. An insecticidal composition according to claim 18 wherein the
- 30 concentration of the one or more insecticides is 0.2 to 0.6% w/w.
20. An insecticidal composition according to any one of claims 5 to 19 wherein the solvent is selected from the group consisting of isoparaffins, normal paraffins, C₁₋₁₂ alkanes and kerosene.
21. An insecticidal composition according to any one of claims 5 to 20
- 35 wherein the concentration of the solvent is 60 to 70% w/w.

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22. An insecticidal composition according to any one of claims 5 to 21 wherein the attractant is orange oil.
23. An insecticidal composition according to any one of claims 5 to 22 wherein the concentration of the attractant is 1 to 5% w/w.
- 5 24. An insecticidal composition according to claim 23 wherein the concentration of the attractant is 2 to 4% w/w.
25. An insecticidal composition according to claim 24 wherein the concentration of the attractant is 3% w/w.
26. An insecticidal composition according to any one of claims 5 to 25
- 10 further including one or more preservatives, boosters or perfumes.
27. An insecticidal composition according to claim 26 wherein the preservative is butylated hydroxytoluene.
28. An insecticidal composition according to claim 26 or 27 wherein the concentration of the preservative is 0.001 to 0.1% w/w.
- 15 29. An insecticidal composition according to claim 26 wherein the booster is a fumed silica powder.
30. An insecticidal composition according to claim 29 wherein the fumed silica powder is selected from highly dispersed amorphous hydrophilic silicon dioxide and highly dispersed amorphous hydrophobic
- 20 silicon dioxide.
31. An insecticidal composition according to claim 29 or 30 wherein the concentration of the booster is 0.05 to 0.2% w/w.
32. An insecticidal composition according to claim 26 wherein the concentration of the perfume is 0.05 to 2% w/w.
- 25 33. A method of controlling insects on a surface for a period of 13 weeks or more comprising spraying an effective amount of an insecticidal composition according to any one of claims 5 to 32 onto the surface.
34. An insecticidal composition substantially as herein described with reference to any one of Examples 1 to 3.

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