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INSECTICIDAL AEROSOL

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(71) Applicant(s)
SUMITOMO CHEMICAL COMPANY, LIMITED

(72) Inventor(s)
KAZUNOBU DOHARA; TADAHIRO MATSUNAGA; MOTOMITSU SHIRAISHI; GORO SHINJO

(74) Attorney or Agent
GRIFFITH HACK & CO, GPO Box 1285K, MELBOURNE VIC 3001

(57) Claim

1. A mono-layer liquid phase type water-based insecticidal aerosol which comprises

(A) a base liquid for aerosol including as an insecticidally active ingredient at least one pyrethroidal compound of:

3-phenoxybenzyl 2,2-dimethyl-3-(2,2-dichloro-vinyl)-cyclopropanecarboxylate (permethrin),

3-phenoxybenzyl chrsanthemate (phenothrin),

5-benzyl-3-furymethyl chrysanthemate (resmethrin),

1-ethynyl-2-methyl-2-pentenyl chrysanthemate,

2-(4-ethoxyphenyl)-5-(4-fluoro-3-phenoxy)-phenyl-2-methylpentane or

2-[1-methyl-2-(4-phenoxyphenoxy)ethoxypyridine (pyriproxyfen),

an organic solvent of:

isopropyl alcohol,

n-propyl alcohol,

ethyl alcohol,

propylene glycol,

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propylene glycol methyl ether,
dipropylene glycol methyl ether,
tripropylene glycol methyl ether or
acetone,
and a buffer solution, which base liquid has a pH of
from 7.0 to 11.0, and

(B) dimethyl ether as a propellant.

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COMPLETE SPECIFICATION

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Name of Applicant:

SUMITOMO CHEMICAL COMPANY,
LIMITED

Address of Applicant: 15, KITAHAMA-5-CHOME
HIGASHI-KU
OSAKA
JAPAN

Actual Inventor:

Address for Service: GRIFFITH HACK & CO.,
601 St. Kilda Road,
Melbourne, Victoria 3004,
Australia.

Complete Specification for the invention entitled:
INSECTICIDAL AEROSOL

The following statement is a full description of this invention
including the best method of performing it known to me:-

1 The present invention relates to a mono-layer
liquid phase type water-based insecticidal aerosol.

 Recently, since water-based insecticidal
aerosols can be averted from inflammability and toxicity
5 to mammals, and since their manufacturing cost is
relatively low, various developments have been forwarded
on them.

 However, most of the conventionally known water-
based aerosols are the so-called two-layer liquid phase type
10 water-based ones, that is, the liquid phase separates in two
layers. Consequently, before the use of such aerosols,
homogenizing the liquid phase by shaking is not
avoidable.

 To avoid the inconvenience, the so-called mono-
15 layer liquid phase type water-based insecticidal aerosols
have been developped. This type of aerosols can be prepared
by dispersing an insecticidally active ingredient such as
a pyrethroidal insecticidal compound in water with the
aid of ethanol, etc. followed by blending thereto a
20 propellant such as dimethyl ether, etc.

 However, this type of insecticidal aerosols,
when put to practical use, have serious problems that
corrosion occurs on the inner wall of the aerosol
container made of tinplate during the storage, which
25 in turn causes the leak of the propellant gas, and

1 that the effect of the aerosols is lowered by the
decomposition of the insecticidal compound contained in
the aerosols.

The present inventors have extensively studied
5 to develop a mono-layer liquid phase type water-based
insecticidal aerosol not causing such the problems, and as
a result, have found that a mono-layer liquid phase type
water-based insecticidal aerosol which comprises

(A) a base liquid for aerosol containing as an
10 insecticidally active ingredient at least one pyrethroidal
compound selected from the group consisting of:

3-phenoxybenzyl 2,2-dimethyl-3-(2,2-dichloro-
vinyl)cyclopropanecarboxylate (permethrin),

3-phenoxybenzyl chrysanthemate (phenothrin),
15 5-benzyl-3-furylmethyl chrysanthemate
(resmethrin),

1-ethynyl-2-methyl-2-pentenyl chrysanthemate,

2-(4-ethoxyphenyl)-5-(4-fluoro-3-phenoxy)-
phenyl-2-methylpentane and

20 2-[1-methyl-2-(4-phenoxyphenoxy)ethoxy]pyridine
(pyriproxyfen),

an organic solvent selected from the group consisting of:

isopropyl alcohol,

n-propyl alcohol,

25 ethyl alcohol,

propylene glycol,

propylene glycol methyl ether,

dipropylene glycol methyl ether,

1 tripropylene glycol methyl ether and
 acetone,
and a buffer solution, which base liquid has a pH of
from 7.0 to 11.0, and

5 (B) dimethyl ether as a propellant, can be suited
to this object. The present inventors thus attained
to the present invention.

 The insecticidal aerosol of the present
invention can be kept in a homogeneous liquid phase
10 without causing separation in two layers even when it is
stored for a long period of time at a relatively high
temperature. In addition, there is no generation of
rust on the wall of the aerosol container, and the
insecticidally active ingredient remains stable.

15 Accordingly, the insecticidal aerosol of the
present invention can be used as they are and requires
no previous shaking at the time of application, and also
they can exhibit an excellent effect as an insecticide.

 Each of the pyrethroidal compounds belonging
20 to the foregoing group used as the insecticidally active
ingredient has steric and optical isomers. And these
isomers and their mixtures may be used in the present
invention.

 The insecticidally active ingredient is usually
25 blended in the aerosol in an amount of 0.01 to 2% by
weight, preferably 0.03 to 1% by weight based on the
total weight of the aerosol.

 Specific examples of the buffer solutions are:

- 1 ammonium benzoate-NaOH buffer solution,
sodium benzoate-benzoic acid buffer solution,
ammonium benzoate-ammonia buffer solution,
ammonium benzoate-benzoic acid buffer solution,
5 KH_2PO_4 -NaOH buffer solution,
NaOH-sodium bimalate buffer solution,
tris-maleate^{*}-NaOH buffer solution and

* mixture of tris(hydroxymethyl)aminomethane
and maleic acid

- 10 Na_2CO_3 - NaHCO_3 buffer solution.

The buffer solution is incorporated in the aerosol
in an amount of 10 to 55% by weight, preferably from 20 to
50% by weight based on the total weight of the aerosol.

- 15 The amount of dimethyl ether, a propellant, used
is usually from 10 to 80% by weight, preferably from 30 to
60% by weight based on the total weight of the aerosol.

The amount of the organic solvent used is
usually from 10 to 70% by weight, preferably from 18 to
40% by weight.

- 20 In the insecticidal aerosols of the present
invention, surface active agents, synergists, perfumes,
fungicides, etc. may be used together if necessary.

- 25 As the synergists, conventional ones such as
piperonyl butoxide, S-421, MGK-264, Synepirin, etc. may
be used.

The insecticidal aerosols of the present
invention may be prepared, for example, by the following
method: Prescribed amounts of the insecticidally active

1 ingredient, organic solvents and if necessary, surface
active agents, synergists, perfumes, fungicides, etc. are
mixed at room temperature or under heating and put in an
aerosol container; a prescribed amount of the buffer
5 solution having a prescribed pH value is added, and the
base liquid for aerosol thus obtained is conditioned so
as to have a pH in a range of from 7.0 to 11.0; and after
mounting a valve portion on the aerosol container,
dimethyl ether is charged into the container under
10 pressure through the valve portion.

The present invention will be illustrated in
more detail with reference to the following examples and
comparative examples, but it is not limited to these
examples.

15 In the following examples, parts mean a part
by weight.

EXAMPLE 1

0.3 Part of phenothrin and 24.7 parts of
isopropyl alcohol were well mixed under heating and
20 introduced into an aerosol container made of tinplate.
To the mixture were added 30.0 parts of an ammonium
benzoate-ammonia buffer solution, which had been prepared
by adding a 29% aqueous ammonia to a 1.0% w/w aqueous
ammonium benzoate solution and then conditioning the pH
25 of the solution to 11.3. Thus, a base liquid for aerosol
having a pH of 11.0 was prepared. Thereafter, a valve
portion was mounted on the aerosol container, and 45.0

1 parts of dimethyl ether were charged into the container
under pressure through the valve portion.

The insecticidal aerosol of the present invention
thus prepared was stored at 45°C for 3 months. Then the
5 aerosol in the container was observed. It was found that
the aerosol was not recognized to be separated, remaining
homogeneous and transparent. The content of phenothrin
in the aerosol was determined by gas chromatography
under the following conditions and compared with the
10 content which had been determined immediately after
preparation of the aerosol. As a result, the percentage
of the remainder of phenothrin was found to be 95.5%.

The generation of rust on the wall of the
aerosol container was not observed.

15 Apparatus: FID

Column: 2% DEGS [Chromosorb W (AW, DMCS, 100-
120 mesh)].

Glass column of 1.1 m x 3 mmø in size.

Column temperature: 210°C

20 N₂ flow rate: 50 ml/min

Internal standard: Tetramethrin

EXAMPLE 2

0.3 Part of resmethrin and 24.7 parts of
isopropyl alcohol were well mixed under heating and
25 introduced into an aerosol container made of tinplate.
To the resulting mixture were added 30.0 parts of an
ammonium benzoate-NaOH buffer solution, which had been

1 prepared by adding a 10% w/w aqueous NaOH solution to a
1.0% w/w aqueous ammonium benzoate solution and then
conditioning the pH of the solution to 11.3. Thus, a
base liquid for aerosol having a pH of 11.0 was prepared.

5 Thereafter, a valve portion was mounted on the aerosol
container, and 45.0 parts of dimethyl ether were charged
into the container under pressure through the valve
portion.

The insecticidal aerosol of the present
10 invention thus prepared was stored at 45°C for 3 months.
Then the aerosol in the container was observed. It was
found that the aerosol was not recognized to be separated,
remaining homogeneous and transparent. The content of
resmethrin in the aerosol was determined by gas
15 chromatography under the following conditions and compared
with the content which had been determined immediately
after preparation of the aerosol. As a result, the
percentage of the remainder of resmethrin was found to
be 93.8%.

20 The generation of rust on the wall of the aerosol
container was not observed.

Apparatus, column, column temperature and N₂
flow rate: Same as in Example 1.

Internal standard: Phenothrin

25 EXAMPLE 3

0.3 Part of permethrin, 0.5 part of sorbitan
monolaurate and 24.2 parts of isopropyl alcohol were well

1 mixed under heating and introduced into an aerosol
container made of tinplate. To the mixture were added
20.0 parts of an ammonium benzoate-ammonia buffer solution,
which had been prepared by adding a 29% aqueous ammonia
5 to a 1.0% w/w aqueous ammonium benzoate solution and then
conditioning the pH of the solution to 11.3. Thus, a
base liquid for aerosol having a pH of 11.0 was prepared.
Thereafter, a valve portion was mounted on the aerosol
container, and 55.0 parts of dimethyl ether were charged
10 into the container under pressure through the valve
portion.

The insecticidal aerosol of the present
invention thus prepared was stored at 45°C for 3 months.
Then the aerosol in the container was observed. It was
15 found that the aerosol was not recognized to be separated,
remaining homogeneous and transparent. The content of
permethrin in the aerosol was determined by gas chromato-
graphy in the same manner as in Example 1 and compared
with the content which had been determined immediately
20 after preparation of the aerosol. As a result, the
percentage of the remainder of permethrin was found to
be 91.9%.

The generation of rust on the wall of the
aerosol container was not observed.

25 EXAMPLE 4

0.3 Part of (RS)-1-ethynyl-2-methyl-2-pentenyl
(1R)-cis, trans-chrysanthemate (empenthrin) and 24.7

1 parts of propylene glycol were well mixed under heating
and introduced into an aerosol container made of tinplate.
To the resulting mixture were added 30.0 parts of an
ammonium benzoate-ammonia buffer solution, which had been
5 prepared by adding a 29% aqueous ammonia to a 1.0% w/w
aqueous ammonium benzoate solution and then conditioning
the pH of the mixed solution to 10.5. Thus, a base
liquid for aerosol having a pH of 10.0 was prepared.
Thereafter, a valve portion was mounted on the aerosol
10 container, and 45.0 parts of dimethyl ether were charged
into the container under pressure through the valve
portion.

The insecticidal aerosol of the present
invention thus prepared was stored at 45°C for 3 months.
15 Then the aerosol in the container was observed. It was
found that the aerosol was not recognized to be separated,
remaining homogeneous and transparent. The content of
empenethrin in the aerosol was determined by gas chromato-
graphy under the following conditions and compared with
20 the content which had been determined immediately after
preparation of the aerosol. As a result, the percentage
of the remainder of empenethrin was found to be 98.7%.

The generation of rust on the wall of the
aerosol container was not observed.

25 Apparatus: FID

Column: 5% SE-30 [Uniport HP (100-120 mesh)].

Glass column of 1.1 m x 3 mmø in size.

Column temperature: 150°C

1 N₂ flow rate: 50 ml/min
 Internal standard: Dimethyl phthalate

EXAMPLE 5

 0.3 Part of pyriproxyfen and 24.7 parts of ethyl
5 alcohol were well mixed under heating and introduced into
 an aerosol container made of tinplate. To the mixture
 were added 30.0 parts of an ammonium benzoate-ammonia
 buffer solution, which had been prepared by adding a 29%
 aqueous ammonia to a 1.0% w/w aqueous ammonium benzoate
10 solution and then conditioning the pH of the mixed
 solution to 10.5. Thus, a base liquid for aerosol having
 a pH of 10.0 was prepared. Thereafter, a valve portion
 was mounted on the aerosol container, and 45.0 parts
 of dimethyl ether were charged into the container under
15 pressure through the valve portion.

 The insecticidal aerosol of the present
 invention thus prepared was stored at 45°C for 3 months.
 Then the aerosol in the container was observed. It was
 found that the aerosol was not recognized to be separated,
20 remaining homogeneous and transparent. The content of
 pyriproxyfen in the aerosol was determined by gas
 chromatography under the following conditions and
 compared with the content which had been determined
 immediately after preparation of the aerosol. As a
25 result, the percentage of the remainder of pyriproxyfen
 was found to be 97.0%.

 The generation of rust on the wall of the

1 aerosol container was not observed.

Apparatus: FID

Column: 5% OV-101 [Uniport HP (100-120 mesh)].

Glass column of 1.1 m x 3 mmØ in size.

5 Column temperature: 260°C

N₂ flow rate: 50 ml/min

Internal standard: Triphenyl phosphate

EXAMPLE 6

0.3 Part of 2-(4-ethoxyphenyl)-5-(4-fluoro-3-
10 phenoxy)phenyl-2-methylpentane and 24.7 parts of
tripropylene glycol methyl ether were well mixed under
heating and introduced into an aerosol container made of
tinplate. To the mixture were added 30.0 parts of an
ammonium benzoate-ammonia buffer solution, which had been
15 prepared by adding a 29% aqueous ammonia to a 1.0% w/w
aqueous ammonium benzoate solution and then conditioning
the pH of the mixed solution to 10.5. Thus, a base
liquid for aerosol having a pH of 10.0 was prepared.
Thereafter, a valve portion was mounted on the aerosol
20 container, and 45.0 parts of dimethyl ether were charged
into the container under pressure through the valve
portion.

The insecticidal aerosol of the present
invention thus prepared was stored at 45°C for 3 months.
25 Then the aerosol in the container was observed. It was
found that the aerosol was not recognized to be
separated, remaining homogeneous and transparent. The

content of 2-(4-ethoxyphenyl)-5-(4-fluoro-3-phenoxy)-phenyl-2-methylpentane in the aerosol was determined by gas chromatography in the same manner as in Example 5 and compared with the content which had been determined immediately after preparation of the aerosol. As a result, the percentage of the remainder of 2-(4-ethoxyphenyl)-5-(4-fluoro-3-phenoxy) phenyl-2-methylpentane was found to be 92.1%.

The generation of rust on the wall of the aerosol container was not observed.

EXAMPLE 7

An aerosol was prepared by repeating the same procedure as in Example 1 except that an ammonium benzoate-ammonia buffer solution having a pH of 10.5 was used instead of that having a pH of 11.3 and the base liquid for aerosol was conditioned to have a pH of 10.0.

The insecticidal aerosol of the present invention thus prepared was stored at 45°C for 3 months. Then, the aerosol in the container was observed. As a result, the aerosol was not recognized to be separated and was remained homogeneous and transparent. The content of phenothrin the insecticidal active ingredient, in the aerosol was determined by gas chromatography in the same manner as in Example 1 and compared with the content which had been determined immediately after preparation of the aerosol. As a result, the percentage of the remainder of phenothrin was found to be 96.0%.

The generation of rust on the wall of the aerosol container was not observed.

EXAMPLE 8

An aerosol was prepared by repeating the same procedure as in Example 1 except that an ammonium benzoate-ammonia buffer solution having a pH of 9.5 was used instead of that having a pH of 11.3 and the base liquid for aerosol was conditioned to have a pH of 9.0.



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The insecticidal aerosol of the present invention thus prepared was stored at 45°C for 3 months.

Then, the aerosol in the container was observed. As a result, the aerosol was not recognized to be separated and was remained homogeneous and transparent. The content of phenothrin, the insecticidal active ingredient, in the aerosol was determined by gas chromatography in the same manner as in Example 1 and compared with the content which had been determined immediately after the preparation of the aerosol. As a result, the percentage of the remainder of phenothrin was found to be 97.1%.

The generation of rust on the wall of the aerosol container was not observed.

EXAMPLE 9

An aerosol was prepared by repeating the same procedure as in Example 3 except that an ammonium benzoate-ammonia buffer solution having a pH of 8.5 was used instead of that having a pH of 11.3 and the base liquid for aerosol was conditioned to have a pH of 8.0.

The insecticidal aerosol of the present invention thus prepared was stored at 45°C for 3 months. Then, the aerosol in the container was observed. As a result, the aerosol was not recognized to be separated and was remained homogeneous and transparent. The content of permethrin, the insecticidal active ingredient, in the aerosol was determined by gas chromatography in the same manner as in Example 1 and compared with the content which had been determined immediately after preparation of the aerosol. As a result, the percentage of the remainder of permethrin was found to be 96.2%.

The generation of rust on the wall of the aerosol container was not observed.



EXAMPLE 10

An aerosol was prepared by repeating the same procedure as in Example 1 except that an ammonium benzoate-ammonia buffer solution having a pH of 6.5 was used instead of that having a pH of 11.3 and the base liquid for aerosol was conditioned to have a pH of 7.0.

The insecticidal aerosol of the present invention thus prepared was stored at 45°C for 3 months. Then, the aerosol in the container was observed. As a result, the aerosol was not recognized to be separated and was remained homogeneous and transparent. The content of phenothrin, the insecticidal active ingredient, in the aerosol was determined by gas chromatography in the same manner as in Example 1 and compared with the content which had been determined immediately after preparation of the aerosol. As a result, the percentage of the remainder of phenothrin was found to be 96.7%.

The generation of rust on the wall of the aerosol container was not observed.

EXAMPLE 11

0.3 part of phenothrin and 24.7 parts of isopropyl alcohol were well mixed under heating and introduced into an aerosol container made of tinfoil. To the mixture were added 45.0 parts of an ammonium benzoate-ammonia buffer solution, which had been prepared by adding a 29% aqueous ammonia to a 1.0% w/w aqueous ammonium benzoate solution and then conditioning the pH of the solution to 10.5. Thus, a base liquid for aerosol having a pH of 10.0 was prepared. Thereafter, a valve portion was mounted on the aerosol container, and 30.0 parts of dimethyl ether were charged into the container under pressure through the valve portion.

The insecticidal aerosol of the present invention thus prepared was stored at 45°C for 3 months. Then, the aerosol in the container was observed. As a result, the aerosol was not recognized to be separated



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and was remained homogeneous and transparent. The content of phenothrin, the insecticidal active ingredient, in the aerosol was determined by gas chromatography in the same manner as in Example 1 and compared with the content which had been determined immediately after preparation of the aerosol. As a result, the percentage of the remainder of phenothrin was found to be 98.5%.

The generation of rust on the wall of the aerosol container was not observed.

COMPARATIVE EXAMPLE 1

An aerosol was prepared in the same manner as in Example 3 except that an ammonium benzoate-ammonia buffer solution having a pH of 13.3 was used, and that the pH of the base liquid for aerosol was conditioned to 13.0.

The insecticidal aerosol thus obtained was stored at 45°C for 3 months. Then the permethrin in the aerosol was determined by gas chromatography in the same manner as in Example 3 and compared with the content which had been determined immediately after preparation of the aerosol. As a result, the percentage of the remainder of permethrin was found to be 54.3%.

In addition, the generation of rust on the wall of the aerosol container was observed.

COMPARATIVE EXAMPLE 2

An aerosol was prepared by repeating the same procedure as in Example 7 except that phenothrin was replaced by prallethrin.

The insecticidal aerosol thus prepared was stored at 45°C for 3 months. Then, the content of prallethrin, the insecticidal active ingredient, in the aerosol was determined by gas chromatography under the conditions shown below and compared with the content which had been determined immediately after preparation of the aerosol.

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Apparatus, Column and N₂ flow rate: same as in
Example 1

Column temperature: 190°C

Internal standard: Fluoranthene

As a result, the percentage of the remainder
of prallethrin was found to be 35.0%.

COMPARATIVE EXAMPLE 3

An aerosol was prepared by repeating the same
procedure as in Example 7 except that phenothrin was
replaced by cyphenothrin.

The insecticidal aerosol thus prepared was
stored at 45°C for 3 months. Then, the content of
cyphenothrin, the insecticidal active ingredient, in the
aerosol was determined by gas chromatography in the same
manner as in Example 1 and compared with the content
which had been determined immediately after preparation
of the aerosol. As a result, the percentage of the
remainder of cyphenothrin was found to be 45.2%.

COMPARATIVE EXAMPLE 4

An aerosol was prepared by repeating the same
procedure as in Example 7 except that phenothrin was
replaced by tetramethrin.

The insecticidal aerosol thus prepared was
stored at 45°C for one month. Then, the content of
tetramethrin, the insecticidal active ingredient, in the
aerosol was determined by gas chromatography under the
conditions shown below and compared with the content
which had been determined immediately after preparation
of the aerosol.

Apparatus, Column temperature and N₂ flow rate:
same as in Example 1

Internal standard: Phenothrin

As a result, the percentage of the remainder
of tetramethrin was found to be less than 1.0%.



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COMPARATIVE EXAMPLE 5

An aerosol was prepared by repeating the same procedure as in Example 7 except that phenothrin was replaced by fenitrothion.

The insecticidal aerosol thus prepared was stored at 45°C for 3 months. Then, the content of fenitrothion, the insecticidal active ingredient, in the aerosol was determined by gas chromatography under the conditions shown below and compared with the content which had been determined immediately after preparation of the aerosol.

Apparatus, Column and N₂ flow rate: same as in
Example 1

Column temperature: 195°C

Internal standard: Cyanophos

As a result, the percentage of the remainder of fenitrothion was found to be less than 1.0%.

COMPARATIVE EXAMPLE 6

An aerosol was prepared by repeating the same procedure as in Example 7 except that phenothrin was replaced by deltamethrin.

The insecticidal aerosol thus prepared was stored at 45°C for one month. Then, the content of deltamethrin, the insecticidal active ingredient, in the aerosol was determined by gas chromatography under the conditions shown below and compared with the content which had been determined immediately after preparation of the aerosol.

Apparatus and N₂ flow rate: same as in Example 1

Column: glass column having a length of 1.1 m
and a diameter of 3 mm, charged with 3%
OV-101 [Sumikasorb HP (100-120 mesh)]

Column temperature: 230°C

Internal standard: Phenylbiphenyl-1,3,4-
oxadiazole

As a result, the percentage of the remainder



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of deltamethrin was found to be less than 1.0%.

COMPARATIVE EXAMPLE 7

An aerosol was prepared by repeating the same procedure as in Example 7 except that phenothrin was replaced by propoxur.

The insecticidal aerosol thus prepared was stored at 45°C for 3 months. Then, the content of propoxur, the insecticidal active ingredient, in the aerosol was determined by gas chromatography under the conditions shown below and compared with the content which had been determined immediately after preparation of the aerosol.

Apparatus and N₂ flow rate: same as in Example 1

Column: glass column having a length of 1.1 m
and a diameter of 3 mm, charged with 2%
XE-60 [Chromosorb W (HP, 100-120 mesh)]

Column temperature: 170°C

Internal standard: n-Butyl sebacate

As a result, the percentage of the remainder of propoxur was found to be less than 1.0%.

COMPARATIVE EXAMPLE 8

An aerosol was prepared by repeating the same procedure as in Example 7 except that phenothrin was replaced by cypermethrin.

The insecticidal aerosol thus prepared was stored at 45°C for one month. Then, the content of cypermethrin, the insecticidal active ingredient, in the aerosol was determined by gas chromatography under the conditions shown below and compared with the content which had been determined immediately after preparation of the aerosol.

Apparatus and N₂ flow rate: same as in Example 1

Column: glass column having a length of 1.1 m
and a diameter of 3 mm, charged with 3%
OV-101 [Sumikasorb HP (100-120 mesh)]

Column temperature: 250°C



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Internal standard: Diisooctyl phthalate

As a result, the percentage of the remainder of cypermethrin was found to be less than 1.8%.

COMPARATIVE EXAMPLE 9

An aerosol was prepared by repeating the same procedure as in Example 7 except that phenothrin was replaced by fenvalerate.

the insecticidal aerosol thus prepared was stored at 45°C for 3 months. Then, the content of fenvalerate, the insecticidal active ingredient, in the aerosol was determined by gas chromatography under the conditions shown below and compared with the content which had been determined immediately after preparation of the aerosol.

Apparatus: FID

Column: glass column having a length of 2.1 m
and a diameter of 3 mm, charged with 2%
Dexsil 300 GC [Sumikasorb HP (80-100
mesh)]

Column temperature: 270°C

Internal standard: Dioctyl sebacate

As a result, the percentage of the remainder of fenvalerate was found to be less than 1.0%.

COMPARATIVE EXAMPLE 10

As aerosol was prepared by repeating the same procedure as in Example 7 except that phenothrin was replaced by cyfluthrin.

The insecticidal aerosol thus prepared was stored at 45°C for one month. Then, the content of cyfluthrin, the insecticidal active ingredient, in the aerosol was determined by gas chromatography under the conditions shown below and compared with the content which had been determined immediately after preparation of the aerosol.

Apparatus and N₂ flow rate: same as in Example 1

Column: glass column having a length of 1.1 m



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and a diameter of 3 mm, charged with 3%

OV-101 [Sumikasorb HP (100-120 mesh)]

Column temperature: 230°C

Internal standard: Phenylbiphenyl-1,3,4-
oxadiazole

As a result, the percentage of the remainder
of cyfluthrin was found to be less than 1.0%.

COMPARATIVE EXAMPLE 11

An aerosol was prepared by repeating the same
procedure as in Example 7 except that phenothrin was
replaced by natural pyrethrins containing pyrethrins I
and II.

The insecticidal aerosol thus prepared was
stored at 45°C for 3 months. Then, the contents of
pyrethrins I and II, the main insecticidal active
ingredients, in the aerosol were determined by gas
chromatography under the conditions shown below and
compared with the contents which had been determined
immediately after preparation of the aerosol.

Apparatus and N₂ flow rate: same as in Example 1

Column: glass column having a length of 1 m and
a diameter of 3 mm, charged with 5% XE-
60 [Sumikasorb HP (80-100 mesh)]

Column temperature: 190 -230°C (temperature
increasing rate: 3°C/min)

Internal standard: Dicyclohexyl phthalate

As a result, the percentages of the remainders
of pyrethrins I and II were found to be 32.9% and less
than 1.0%, respectively.

COMPARATIVE EXAMPLE 12

An aerosol was prepared by repeating the same
procedure as in Example 7 except that phenothrin was
replaced by metoxadiazone.

The insecticidal aerosol thus prepared was
stored at 45°C for one month. Then, the content of
metoxadiazone, the insecticidal active ingredient, in



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the aerosol was determined by gas chromatography under the conditions shown below and compared with the content which had been determined immediately after preparation of the aerosol.

Apparatus and N₂ flow rate: same as in Example 1

Column: glass column having a length of 1.1 m
and a diameter of 3 mm, charged with 2%
XE-60 [Chromosorb W (HP, 100-120 mesh)]

Column temperature: 170°C

Internal standard: n-Butyl sebacate

As a result, the percentage of the remainder of metoxadiazone was found to be less than 1.0%.

COMPARATIVE EXAMPLE 13

An aerosol was prepared by repeating the same procedure as in Example 11 except that phenothrin was replaced by allethrin.

The insecticidal aerosol thus prepared was stored at 45°C for 3 months. Then, the content of allethrin, the insecticidal active ingredient, in the aerosol was determined by gas chromatography under the conditions shown below and compared with the content which had been determined immediately after preparations of the aerosol.

Apparatus, column and N₂ flow rate: same as in
Example 1

Column temperature: 190°C

Internal standard: Fluoranthene

As a result, the percentage of the remainder of allethrin was found to be 55.3%.

COMPARATIVE EXAMPLE 14

An aerosol was prepared by repeating the same procedure as in Example 11 except that phenothrin was replaced by furamethrin.

The insecticidal aerosol thus prepared was stored at 45°C for one month. Then, the content of furamethrin, the insecticidal active ingredient, in the



- 12(j) -

aerosol was determined by gas chromatography under the conditions shown below and compared with the content which had been determined immediately after preparation of the aerosol.

Apparatus and N₂ flow rate: same as in Example 1

Column: glass column having a length of 1.1 m
and a diameter of 3 mm, charged with 5%
XE-60 [Uniport HP (60-80 mesh)]

Column temperature: 160°C

Internal standard: Dibutyl phthalate

As a result, the percentage of the remainder
of allethrin was found to be 58.1%.



THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A mono-layer liquid phase type water-based insecticidal aerosol which comprises

(A) a base liquid for aerosol including as an insecticidally active ingredient at least one pyrethroidal compound of:

3-phenoxybenzyl 2,2-dimethyl-3-(2,2-dichloro-vinyl)-cyclopropanecarboxylate (permethrin),

3-phenoxybenzyl chrsanthemate (phenothrin),

5-benzyl-3-furymethyl chrysanthemate (resmethrin),

1-ethynyl-2-methyl-2-pentenyl chrysanthemate,

2-(4-ethoxyphenyl)-5-(4-fluoro-3-phenoxy)-phenyl-2-methylpentane or

2-[1-methyl-2-(4-phenoxyphenoxy)ethoxylpyridine (pyriproxyfen),

an organic solvent of:

isopropyl alcohol,

n-propyl alcohol,

ethyl alcohol,

propylene glycol,

propylene glycol methyl ether,

dipropylene glycol methyl ether,

tripropylene glycol methyl ether or

acetone,

and a buffer solution, which base liquid has a pH of from 7.0 to 11.0, and

(B) dimethyl ether as a propellant.

2. An aerosol according to Claim 1, wherein dimethyl ether as a propellant is blended in the aerosol in an amount of 10 to 80% by weight based on the total weight of the aerosol.

3. An aerosol according to Claim 1 or 2, which comprises an organic solvent of isopropyl alcohol, n-propyl alcohol, ethyl alcohol, propylene glycol, propylene glycol methyl ether, dipropylene glycol methyl ether, tripropylene glycol methyl ether or acetone in an amount of from 10 to 70% by weight based



on the total weight of the aerosol.

4. An aerosol according to anyone of Claims 1 to 3, which comprises a buffer solution of from 10 to 55% by weight based on the total weight of the aerosol.

5. An aerosol according to anyone of Claims 1 to 4, which comprises said pyrethroidal compound in an amount of from 0.01 to 2% by weight based on the total weight of the aerosol.

6. A mono-layer liquid phase type water-based insecticidal aerosol substantially as hereinbefore described with reference to anyone of the forgoing examples apart from the comparative examples.

DATED THIS 4th DAY OF October 1990

SUMITOMO CHEMICAL COMPANY LIMITED
By Its Patent Attorneys:

GRIFFITH HACK & CO.
Fellows Institute of Patent
Attorneys of Australia.

