Spruson & Ferguson

598085

COMMONWEALTH OF AUSTRALIA
THE PATENTS ACT 1952

AUSTRALIA
CONVENTION
STANDARD
PATENT
APPLICATION
FORM
SFP2

CONVENTION APPLICATION FOR STANDARD PATENT OR A STANDARD PATENT OF ADDITION

APPLICATION ACCEPTED AND AMENDMENTS ALLOWED 27-3-90

Full name(s) of Applicant(s)

I/We Stauffer Agricultural Chemicals Company, Inc.,

Address(es) of Applicant(s)

of c/o ICI Americas Inc, Concord Pike and New Murphy Road, Wilmington, Delaware, 19897, United States of America

hereby apply for the grant of a standard patent ~~patent of addition~~

for an invention entitled

Title of Invention

Fungicidal Carbanilates

LODGED AT SUB-OFFICE
- 4 JAN 1988
Sydney

PATENT OFFICE
LODGED AT SUB-OFFICE
ONE HUNDRED DOLLARS

which is described in the accompanying complete specification.

DETAILS OF BASIC APPLICATION(~~S~~)

Number(~~s~~) of Basic Application(~~s~~)

000607

Name(~~s~~) of Convention Country(~~ies~~) in which Basic Application(~~s~~) was/~~were~~ filed

United States of America

~~(respectively)~~

Date(~~s~~) of Basic Application(~~s~~)

5 January 1987 ~~(respectively)~~

FEE STAMPED VALUE £5
$ 155

PATENT OFFICE
SUB-OFFICE
FIVE DOLLARS

~~My~~/Our address for service is:

C/- **Spruson & Ferguson**
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Dated this FOURTH day of JANUARY 1988

Stauffer Agricultural Chemicals Company, Inc.

N J Andersen
Registered Patent Attorney

To: The Commissioner of Patents

SFP2

11/81

Spruson & Ferguson

COMMONWEALTH OF AUSTRALIA
THE PATENTS ACT 1952
DECLARATION IN SUPPORT OF A CONVENTION APPLICATION FOR A PATENT

AUSTRALIA CONVENTION STANDARD & PETTY PATENT DECLARATION SFP4

In support of the Convention Application made for a patent for an invention entitled:

~~PR-7593~~

Title of Invention: Fungicidal Carbanilates

I/~~We~~ John Marshall Hamilton, Secretary

Full name(s) and address(es) of Declarant(s)

of Stauffer Agricultural Chemicals Company, Inc. c/o ICI Americas Inc, Concord Pike and New Murphy Road, Wilmington, Delaware 19897, United States of America

do solemnly and sincerely declare as follows:—

Full name(s) of Applicant(s)

~~1. I am/We are the applicant(s) for the patent~~

(or, in the case of an application by a body corporate)

1. I am/~~We are~~ authorised by Stauffer Agricultural Chemicals Company the applicant(~~s~~) for the patent to make this declaration on its/~~their~~ behalf.

2. The basic application(~~s~~) as defined by Section 141 of the Act was/~~were~~ made

Basic Country(ies): in United States of America

Priority Date(s): on 5 January, 1987

Basic Applicant(s): by DON R. BAKER, KEITH H. BROWNELL and CHARLES KEZERIAN

~~3. I am/We are the actual inventor(s) of the invention referred to in the basic application(s)~~

Full name(s) and address(es) of inventor(s)

(or where a person other than the inventor is the applicant)

3. DON ROBERT BAKER, KEITH HARVEY BROWNELL and CHARLES KEZERIAN

of 15 Muth Drive, Orinda, California 94563; 4751 Elmhurst Drive, San Jose, California 95129 and 26 Descanso Drive, Orinda, California 94563, all in the United States of America (respectively)

~~is~~/are the actual inventor(s) of the invention and the facts upon which the applicant(~~s~~) is/~~are~~ entitled to make the application are as follows:

Set out how Applicant(s) derive title from actual inventor(s) e.g. The Applicant(s) is/are the assignee(s) of the invention from the inventor(s)

STAUFFER AGRICULTURAL CHEMICALS COMPANY, INC. is the assignee of STAUFFER CHEMICAL COMPANY which is the assignee of the actual inventors.

4. The basic application(~~s~~) referred to in paragraph 2 of this Declaration was/~~were~~ the first application(~~s~~) made in a Convention country in respect of the invention (~~s~~) the subject of the application.

Declared at Westport, Connecticut this 17th day of December 1987

STAUFFER AGRICULTURAL CHEMICALS COMPANY, INC.

John Marshall Hamilton

John Marshall Hamilton
Secretary

SFP4 To: The Commissioner of Patents 11/81

(12) PATENT ABRIDGMENT (11) Document No. AU-B-10026/88
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 598085

(54) Title
FUNGICIDAL CARBANILATES

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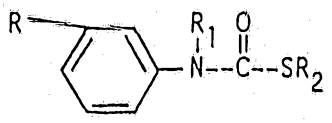
(74) Attorney or Agent
SPRUSON & FERGUSON

(56) Prior Art Documents
US 3781327

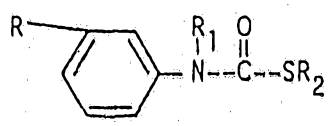
(57) Claim

1. A compound having the structural formula



wherein R is methoxy or allyloxy; R_1 is selected from the group consisting of C_2-C_4 alkoxyalkyl and formyl; and R_2 is C_1-C_6 alkyl.

7. A fungicidal composition comprising a fungicidally effective amount of a compound having the structural formula



where R is methoxy or allyloxy; R_1 is selected from the group consisting of C_2-C_4 alkoxyalkyl ~~from~~ and formyl; and R_2 is C_1-C_6 alkyl; and an inert diluent carrier.

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COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952

COMPLETE SPECIFICATION

(ORIGINAL)

FOR OFFICE USE:

Class Int. Class

Complete Specification Lodged:

Accepted:

Published:

Priority:

Related Art:

This document contains the amendments made under Section 49.

and is correct for printing.

Name of Applicant: Stauffer Agricultural Chemicals Company, Inc.

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31 Market Street, Sydney,
New South Wales, 2000, Australia

Complete Specification for the invention entitled:

Fungicidal Carbanilates

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

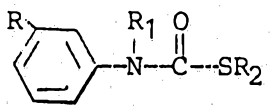
SBR:ALB:5W

PR-7593

FUNGICIDAL CARBANILATES

Abstract of the Disclosure

Novel fungicidal carbanilates having the general structural formula



wherein R is methoxy or allyloxy; R_1 is selected from the group consisting of hydrogen, C_2-C_4 alkoxyalkyl and formyl; and R_2 is C_1-C_6 alkyl, preferably C_2-C_3 alkyl which are highly effective fungicides are disclosed herein.

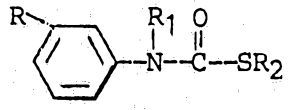
FUNGICIDAL CARBANILATES

Background of the Invention

Fungal growth on agriculturally important crops such as barley, rice, tomatoes, wheat, beans, roses, grapes and other agriculturally important crops can cause heavy loses in both quantity and quality of agricultural products. It is therefore extremely desirable to have means of preventing, controlling or eliminating fungal growth. Much preventative spraying with commercial fungicides is conducted to attempt to prevent the establishment and growth of fungi on agriculturally important crops.

Summary of the Invention

Novel fungicidal carbanilates having the formula

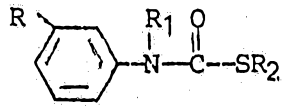


wherein R is methoxy or allyloxy; R_1 is selected from the group consisting of hydrogen, C_2-C_4 alkoxyalkyl and formyl; and R_2 is C_1-C_6 alkyl, preferably C_2-C_3 alkyl, are highly effective fungicides.

The term "fungicide" is used to mean a compound which prevents, destroys or inhibits fungal growth.

Detailed Description

The novel fungicidal compounds of this invention are carbanilates having the general formula



wherein R is methoxy or allyloxy; R_1 is selected from the group consisting of hydrogen, C_2-C_4 alkoxyalkyl and formyl; and R_2 is C_1-C_6 alkyl, preferably C_2-C_3 alkyl are highly effective fungicides.

By the term "alkyl" is meant both straight and branched chain alkyl.

General Methods of Preparation

The compounds of the type where R_1 is hydrogen are prepared by reaction of the appropriate chlorothiolformate with the appropriate aniline in an inert solvent with an acid binding agent such as pyridine, triethylamine, dimethylaniline, sodium or potassium hydroxide or carbonate.

The compounds of the type where R_1 is alkoxyalkyl are prepared by reaction of the compounds described above where R_1 is hdyrogen with an alkoxyalkyl halide in the presence of an appropriate acid binding agent such as sodium hydride in an inert solvent such as ether or tetrahydrofuran.

The compounds of the type where R_1 is formyl are prepared by the reaction of the appropriate chlorothiolformate with an N-silyl formanilide in an inert solvent such as methylene chloride, chloroform, benzene or toluene or the like at room temperature or up to approximately 60°C.

The intermediate N-silyl formanilide is prepared from the appropriate formanilide and a silyl chloride with an acid binding agent such as triethylamine in an inert solvent such as benzene.

EXAMPLE 1

Preparation of S-Ethyl 3-allyloxy thiolcarbanilate

Ethyl chlorothiolformate (4.4 ml, 0.042 mole) was added over a period of two minutes to a solution of 3-allyloxyaniline (6.2 g, 0.0416 mole), methylene chloride (100 ml) and piperidine (4.0 ml, 0.05 mole). The temperature was maintained at 20-25°C with cooling. The resulting solution was allowed to stand overnight and then washed with water (100 ml), 5% hydrochloric acid solution (50 ml) and water (100 ml). This washed solution was dried over anhydrous magnesium sulfate, filtered and evaporated in vacuo to give a solid that was triturated with hexane to yield 8.8 g of the desired product, m.p. 75-79°C.

EXAMPLE 2

Preparation of S-Ethyl 3-methoxythiolcarbanilate

A solution was prepared from 3-methoxyaniline (15.0 g, 0.17 mole), methylene chloride (100 ml) and pyridine (9.5 g, 0.17 mole). This solution was cooled to approximately 5°C under nitrogen and to it was added ethyl chlorothiolformate (21.2 g, 0.17 mole) dropwise. The temperature was maintained at approximately 13-15°C. The reaction was stirred overnight and washed with water, (2 times, 100 ml); dried over anhydrous magnesium sulfate, filtered and evaporated in vacuo to yield 33.5 g of the desired solid product (m.p. 39-41°C).

EXAMPLE 3

Preparation of N-Ethylthiocarbonyl-3-methoxyformanilide

Trimethylsilyl chloride (21.6 g, 0.20 mole) was added to a solution of 3-methoxyformanilide (22 g, 0.146 mole) in benzene (200 ml) at 10°C under a dry argon atmosphere. To this solution was added a solution of triethylamine (20 g, 0.20 mole) and benzene (50 ml) over a period of 30 minutes. The reaction mixture was stirred at room temperature for 30 minutes and filtered. The filler cake was washed with benzene (2 times with 100 ml portions). The combined filtrates were distilled and the fraction boiling at 102-104°C at 0.25 mm pressure collected to yield 29 g of the desired N-trimethylsilyl-3-methoxyformanilide.

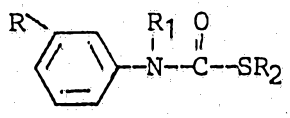
The N-trimethylsilyl-3-methoxyformanilide (4.46 g, 0.02 mole) was added to a solution of ethyl chlorothiolformate (3 g) dissolved in methylene chloride (25 ml). The reaction was allowed to stand overnight at room temperature and evaporated in vacuo at 45°C to give a 5.2 g residue. This was washed at 0°C with hexane (100 ml) by decantation to yield 3.5 g of the desired product as a clear syrup, that solidifed and had a melting point of 42-45°C.

EXAMPLE 4

Preparation of S-Ethyl-N-ethoxymethyl-3-allyloxythiolcarbaniliate

The product of Example 1 (3.6 g, 0.015 mole) was dissolved in dry tetrahydrofuran (50 ml) and to this solution was added sodium hydride (0.38 g, 0.016 mole) under a dry nitrogen atmosphere with stirring for one hour. The reaction was exothermic and hydrogen gas evolved. To this solution was added chloromethyl ethyl ether (1.38 ml, 0.015 mole) and the resulting reaction was exothermic to 36°C. This mixture was stirred for 2 hours until the apparent pH of the reaction dropped to pH 6. The reaction was diluted with ether (100 ml) and methanol (5 ml), washed with water (3 x 100 ml); dried over anhydrous magnesium sulfate and evaporated in vacuo to give 3.9 g of the desired product as an oil.

TABLE I



Cmpd. No.	R	R_1	R_2	n_D^{30} or melting point °C
1	$-OCH_2-CH=CH_2$	-H	$-C_2H_5$	75.0 - 79.0
2	$-OCH_2-CH=CH_2$	-H	$-CH(CH_3)_2$	34.0 - 36.0
3	$-OCH_2-CH=CH_2$	$-CH_2OC_2H_5$	$-C_2H_5$	brown oil
4	$-OCH_2-CH=CH_2$	$-CH_2OC_2H_5$	$-CH(CH_3)_2$	brown oil
5	$-OCH_3$	$-CH_2OC_2H_5$	$-C_2H_5$	1.5452
6	$-OCH_3$	$-\overset{O}{\overset{\|}{C}}H$	$-C_2H_5$	42.0 - 45.0
7	$-OCH_3$	$-\overset{O}{\overset{\|}{C}}H$	$-C_{H3}$	57.0 - 59.0
8	$-OCH_3$	$-\overset{O}{\overset{\|}{C}}H$	$-C_3H_7$	41.0 - 43.0
9	$-OCH_3$	$-\overset{O}{\overset{\|}{C}}H$	$-C_4H_9$	1.5680
10	$-OCH_3$	-H	$-C_2H_5$	39.0 - 41.0

EXAMPLE 5

Preventative Spray Evaluation Procedures

Barley Powdery Mildew (BPM)

Northrup King Sunbar 401 barley seed is planted (12 seeds/2" pot) in a sandy-loam soil seven days prior to testing. The test compound is diluted in a 50/50 acetone/water solution to produce concentratons decreasing from 750 ppm (parts per million). Twelve ml of test solution are sprayed onto the barley plants with atomizing sprayers.

Twenty-four hours later, test plants are placed in an inoculation box equipped with a circulating fan. Barley plants with heavily sporulating Erysiphe graminis lesions are placed in front of the fan to dislodge and distribute the spores. After two minutes the fan is shut off and the chamber is left closed five minutes for the spores to settle. Inoculated plants are then placed on an automatic sub-irrigation greenhouse bench.

Results are recorded seven days following inoculation as percent disease control based on the percent reduction in lesion area as compared to the untreated control plants. Compound concentrations which provide 90% disease control (EC 90) are determined from dosage/dilution curves.

Leaf Rust (LR)

Seven seeds of Anza wheat are planted in 2" pots in a sandy-loam soil 12 days prior to testing. The compound to be tested is diluted with a 50/50 acetone/water solution to produce concentrations decreasing from 750 ppm. Twelve ml of test solution are sprayed onto the wheat plants with an atomizing sprayer.

A suspension of Puccinia recondita urediniospores is prepared by vacuuming spores from wheat leaves with uredinia pustules and suspending 10^5 spores/ml in deionized water plus 0.5% Tween® 20. Plants are inoculated 24 hours after treatment by spraying with the spore suspension to runoff, allowing it to dry on the leaves, respraying to runoff, and then placing the plants into a dark mist chamber. Following 48 hours in the mist, plants are moved to a subirrigation greenhouse bench.

Results are recorded ten days following inoculation as percent disease control based on the percent reduction in lesion area as compared to the untreated control plants. Compound concentrations which provide 90% disease control (EC 90) are determined from dosage/dilution curves.

Rose Mold (RM)

Two white rose petals are placed in a petri dish lined with wet filter paper. The compound to be tested is diluted with a 50/50 acetone/water solution to produce concentrations decreasing from 750 ppm. One half ml of test solution is atomized onto the petals, and allowed to dry.

Inoculum is prepared by adding two 5 mm plugs from a two-week old Botrytis cineria culture grown on Elliot's V-8 agar, to 10 ml sterile distilled water plus 0.5% grape juice. A 20 ul drop of this inoculum suspension is placed on each petal. Petri dishes with inoculated petals are stored in sealed plastic boxes to maintain saturated humidity.

Results are read four days following inoculation as a percent reduction in necrotic area compared to the acetone/water controls. Compound concentrations which provide 90% disease control (EC 90) are determined from dosage/dilution curves.

The results are presented in Table II. The results are presented as approximate EC 90 in parts per million.

TABLE II

Cmpd. No.	BPM	LR	RM
1			30
2	250	750	50
3		500	250
4			750
5			250
6			25
7			70
8			70
9			750
10			30

The compounds of this invention are particularly effective against rose mold (Botrytis) and are particularly effective as preventative foliar sprays and curative foliar sprays when compared to standard commercial compounds used as Botrytis preventative and curative sprays. Fungi on which the compounds of the present invention are particularly effective are as follows: Botyris cinerea, Erysiphe graminis and Puccinia recondita.

The compounds of the present invention are useful as fungicides and can be applied in a variety of ways at various concentrations. In practice, the compounds herein defined are formulated into fungicidal compositions, by admixture, in fungicidally effective amounts, with the adjuvants and carriers normally employed for facilitating the dispersion of active ingredients for agricultural applications, recognizing the fact that the formulation and mode of application of a toxicant may affect the activity of the materials in a given application. Thus, these active fungicidal compounds may be formulated as granules of relatively large particle size, as wettable powders, as emulsifiable concentrates, as powdery dusts, as solutions or as any of several other known types of formulations, depending upon the desired mode of application. Preferred formulations for preventative or curative fungicidal applications are wettable powders, emulsifiable concentrates and granules. These formulations may contain as little as about 0.5% to as much as about 95% or more by weight of active ingredient. A fungicidally effective amount depends upon the nature of the disease to be controlled and the rate of application varies from about 0.05 to approximately 25 pounds per acre, preferably from about 0.1 to about 10 pounds per acre.

Wettable powders are in the form of finely divided particles which disperse readily in water or other dispersants. The wettable powder is ultimately applied to the plant or soil either as a dry dust or as a dispersion in water or other liquid. Typical carriers for wettable powders include fuller's earth, kaolin clays, silicas and other readily wet organic or inorganic diluents. Wettable powders normally are prepared to contain about 5% to about 95% of the active ingredient and usually also contain a small amount of wetting, dispersing, or emulsifying agent to facilitate wetting and dispersion.

Dry flowables or water dispersible granules are agglomerated wettable powders made by either pan granulation or by fluidized bed. The dry flowable is ultimately applied to the soil as a dispersion in water or other liquid. These granules are dust-free and free flowing when dry and yet upon dilution in water, form homogeneous dispersions. Typical carriers for dry flowables include fuller's earth, kaolin clays, silicas and other readily wet organic or inorganic diluents. The dry flowables normally are prepared to contain from about 5% to about 95% of the active ingredient and usually contain a small amount of wetting, dispersing or emulsifying agent to facilitate wetting and dispersion.

Emulsifier concentrates are homogeneous liquid compositions which are dispersible in water or other dispersant, and may consist entirely of the active compound with a liquid or solid emusifying agent, or may also contain a liquid carrier, such as xylene, heavy aromatic naphtha, isophorone and other non-volatile organic solvents. For fungicidal application, these concentrates are dispersed in water or other liquid carrier and normally applied as a spray to the area to be treated. The percentage by weight of the essential active ingredient may vary according to the manner in which the composition is to be applied, but in general comprises about 0.5% to 95% of active ingredient by weight of the fungicidal composition.

Granular formulations wherein the toxicant is carried on relatively coarse particles, are usually applied without dilution to the area in which disease control is desired. Typical carriers for granular formulations include sand, fuller's earth, bentonite clays, vermiculite, perlite and other organic or inorganic materials which absorb or which may be coated with the toxicant. Granular formulations normally are prepared to contain about 5% to about 25% of active ingredients which may include surface-active agents such as wetting agents, dispersing agents or emulsifiers; oil such as heavy aromatic naphthas, kerosene or other petroleum fractions, or vegetable oils; and/or stickers such as dextrins, glue or synthetic resins.

Typical wetting, dispersing or emulsifying agents used in agricultural formulations include, for example, the alkyl and alkylaryl

sulfonates and sulfates and their sodium salts; polyhydroxy alcohols; and other types of surface-active agents, many of which are available in commerce. The surface-active agent, when used, normally comprises from 0.1% to 15% by weight of the fungicidal composition.

Dusts, which are free-flowing admixtures of the active ingredient with finely divided solids such as talc, clays, flours and other organic and inorganic solids which act as dispersants and carriers for the toxicant, are useful formulations for soil-incorporating application.

Pastes, which are homogeneous suspensions of a finely divided solid toxicant in a liquid carrier such as water or oil, are employed for specific purposes. These formulations normally contain about 5% to about 95% of active ingredient by weight, and may also contain small amounts of a wetting, dispersing or emulsifying agent to facilitate dispersion. For application, the pastes are normally diluted and applied as a spray to the area to be treated.

EXAMPLES OF TYPICAL FORMULATIONS

Oil

Ingredient	Weight %
Compound 1	1
Oil solvent-heavy aromatic naphtha	99
Total	100

Emulsifiable Concentrate

Compound 2	50
Kerosene	45
Emulsifying agent (mixture of long chain ethoxylated polyethers with long chain sulfonate)	5
Total	100

Emulsifiable Concentrate

Compound 3	90
Kerosene	5
Emulsifying agent (mixture of long chain ethoxylated polyethers with long chain sulfonate)	5
Total	100

Dusts and/or Powders

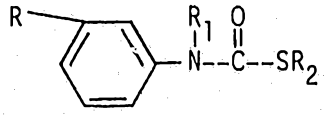
Ingredient	Wt. %	Wt. %	Wt. %
Compound 4	0.5	50.0	90.0
Attapulgite Clay Powder	93.5	44.0	4.0
Sodium lignin sulfonate	5.0	5.0	5.0
Sodium dioctyl sulfosuccinate	1.0	1.0	1.0
Total	100.0	100.0	100.0

Other useful formulations for fungicidal applications include simple solutions of the active ingredient in a dispersant in which it is completely soluble at the desired concentration, such as acetone, alkylated naphthalenes, xylene and other organic solvents. Pressurized sprays, typically aerosols, wherein the active ingredient is dispersed in finely divided form as a result of vaporization of a low boiling dispersant solvent carrier, such as the Freons, may also be used.

The fungicidal compositions of this invention are applied to the plants in the conventional manner. Thus, the dust and liquid compositions can be applied to the plant by the use of power-dusters, boom and hand sprayers and spray dusters. The compositions can also be applied from airplanes as a dust or a spray because they are effective in very low dosages.

The claims defining the invention are as follows:

1. A compound having the structural formula



wherein R is methoxy or allyloxy; R_1 is selected from the group consisting of C_2-C_4 alkoxyalkyl and formyl; and R_2 is C_1-C_6 alkyl.

2. The compound of Claim 1 wherein R is $-OCH_2CH=CH_2$ and R_2 is $-C_2H_5$.

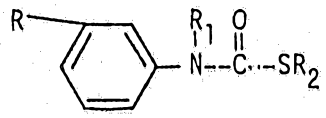
3. The compound of Claim 1 wherein R is $-OCH_2CH=CH_2$ and R_2 is $-CH(CH_3)_2$.

4. The compound of Claim 1 wherein R is $-OCH_3$, and R_1 is $-\overset{O}{\overset{\|}{C}}H$ and R_2 is $-C_2H_5$.

5. The compound of Claim 1 wherein R is $-OCH_3$, R_1 is $-\overset{O}{\overset{\|}{C}}H$ and R_2 is $-CH_3$.

6. The compound of Claim 1 wherein R is $-OCH_3$ and R_2 is $-C_2H_5$.

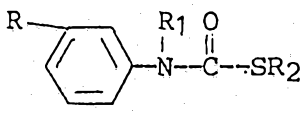
7. A fungicidal composition comprising a fungicidally effective amount of a compound having the structural formula



where R is methoxy or allyloxy; R_1 is selected from the group consisting of C_2-C_4 alkoxyalkyl ~~from~~ and formyl; and R_2 is C_1-C_6 alkyl; and an inert diluent carrier.

8. The method of controlling fungi comprising applying to the area where control is desired, a fungicidally effective amount of a compound having the formula

12



wherein R is methoxy or allyloxy; R_1 is selected from the group consisting of hydrogen, C_2-C_4 alkoxyalkyl and formyl; and R_2 is C_1-C_6 alkyl.

9. The method of Claim 8 wherein R is $-OCH_2CH=CH_2$, R_1 is -H and R_2 is $-C_2H_5$.

10. The method of Claim 8 wherein R is $-OCH_3$, R_1 is -H and R_2 is $-C_2H_5$.

11. A fungicidal carbanilate substantially as hereinbefore described with reference to any one of the Examples.

DATED this FIFTEENTH day of DECEMBER 1987

Stauffer Agricultural Chemicals Company, Inc.

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