Title: FUNGICIDAL COMPOSITIONS BASED ON FLUDIOXONIL

Abstract: Novel fungicidal compositions for the treatment of phytopathogenic diseases of crop plants and against infestation on propagation stock of plants or on other vegetable material, especially phytopathogenic fungi, and to a method of combating phytopathogenic diseases on crop plants or in post-harvest storage and for seed dressing. The invention relates in particular to the control or prevention of diseases in the post-harvest storage period of harvested fruits. It has now been found that the use of a) 4-(2,2-difluoro-1,3-benzodioxol-7-yl)pyrrole-3-carbonitrile ("fludioxonil") (The Pesticide Manual, 11th. edition, 1997, 334), component I in association with b) either 2-(thiazol-4-yl)benzimidazol, component IIA ("thiabenmadazol") (The Pesticide Manual, 11th. edition, 1997, 701); or 1-(β-allyoxy-2,4-dichlorophenylethyl)imidazole, component IIB ("imizalil") (The Pesticide Manual, 11th. edition, 1997, 410) is particularly effective in combating or preventing fungal diseases of crop plants. These combinations exhibit synergistic fungicidal activity.
Fungicidal compositions based on fludioxonil

The present invention relates to novel fungicidal compositions for the treatment of phytopathogenic diseases of crop plants and against infestation on propagation stock of plants or on other vegetable material, especially phytopathogenic fungi, and to a method of combating phytopathogenic diseases on crop plants or in post-harvest storage and for seed dressing.

The invention relates in particular to the control or prevention of diseases in the post-harvest storage period of harvested fruits.

It has now been found that the use of
a) 4-(2,2-difluoro-1,3-benzodioxol-7-yl)pyrrole-3-carbonitrile ("fludioxonil") (The Pesticide Manual, 11th. edition, 1997, 334), component I
in association with
b) either 2-(thiazol-4-yl)benzimidazol, component IIA ("thiabendazole") (The Pesticide Manual, 11th. edition, 1997, 701); or
1-(β-allyloxy-2,4-dichlorophenylethyl)imidazole, component IIB ("imazalil") (The Pesticide Manual, 11th. edition, 1997, 410)
is particularly effective in combating or preventing fungal diseases of crop plants. These combinations exhibit synergistic fungicidal activity.

Favorable mixture ratios of the two active ingredients are I:II = 20:1 to 1:20, preferably I:II = 10:1 to 1:10 and 5:1 to 1:5.

The active ingredient combinations I+II according to the invention have very advantageous properties in the protection of plants and during post-harvest storage of fruits against the outbreak of disease.

The active ingredient combinations are effective against phytopathogenic fungi belonging to the following classes: Ascomycetes (e.g. Venturia, Podosphaera, Erysiphe, Monilinia, Sclerotinia, Mycosphaerella, Uncinula); Basidiomycetes (e.g. the genus Hemileia,
Rhizoctonia, Tilletia, Puccinia); Fungi imperfecti (e.g. Botrytis, Helminthosporium, Rhynchosporium, Fusarium, Septoria, Cercospora, Alternaria, Penicillium spp., Pyricularia and Pseudocercospora herpotrichoides).

With the present active ingredient compositions, the microorganisms appearing on plants or plant parts (fruits, flower, foliage, stems, tubers, roots) of different useful plants can be stopped or destroyed, whereby plant parts growing later also remain free from such microorganisms. They may also be used as post-harvest application or as dressing, or the treatment of plant propagation material, specially seeds.

Target crops for the areas of indication disclosed herein comprise within the scope of this invention e.g. the following species of plants: cereals (wheat, barley, rye, oats, rice, sorghum and related crops); beet (sugar beet and fodder beet); pomes, stone fruit and soft fruit (apples, pears, plums, peaches, almonds, cherries, strawberries, raspberries and blackberries); leguminous plants (beans, lentils, peas, soybeans); oil plants (rape, mustard, poppy, olives, sunflowers, coconut, castor oil plants, cocoa beans, groundnuts); cucumber plants (marrows, cucumbers, melons); fibre plants (cotton, flax, hemp, jute); citrus fruit (oranges, lemons, grapefruit, mandarins); vegetables (spinach, lettuce, asparagus, cabbages, carrots, onions, tomatoes, potatoes, paprika); lauraceae (avocados, cinnamon, camphor); or plants such as maize, tobacco, nuts, coffee, sugar cane, tea, vines, hops, turf, bananas and natural rubber plants, as well as ornamentals (flowers, shrubs, broad-leaved trees and evergreens, such as conifers) and their seeds. This list does not represent any limitation.

The combinations of the present invention may also be used in the area of protecting technical material against attack of fungi. Technical areas include wood, paper, leather, constructions, cooling and heating systems, ventilation and air conditioning systems, and the like. The combinations according the present invention can prevent the disadvantageous effects such as decay, discoloration or mold.

Throughout this document the expression combination stands for the various combinations of the components I and II, e.g. in a single "ready-mix" form, in a combined spray mixture composed from separate formulations of the single active ingredient components, e.g. a "tank-mix", and in a combined use of the single active ingredients when applied in a
sequential manner, i.e. one after the other with a reasonable short period, e.g. a few hours or
days. The order of applying the components I and II is not essential for working the present
invention.

The combinations according to the present invention are particularly effective against Botrytis
spp., Fusarium spp. and Penicillium spp., in particular against pathogens of fruits plants such
as citrus, pomes, stone fruits and soft fruits and bananas as well as potatoes and the
concerning fruits during post-harvest storage.

The amount of combination of the invention to be applied, will depend on various factors such
as the compound employed, the subject of the treatment (plant, soil, post-harvest, seed), the
type of treatment (e.g. spraying, dusting, seed dressing), the purpose of the treatment
(prophylactic or therapeutic), the type of fungi to be treated and the application time.

It has been found that the use of compounds IIA and IIB in combination with the compound of
formula I surprisingly and substantially enhance the effectiveness of the latter against fungi,
and vice versa. Additionally, the method of the invention is effective against a wider spectrum
of such fungi that can be combated with the active ingredients of this method, when used
solely.

The invention also relates to a method of controlling fungi, which comprises treating a site, for
example a plant, its locus of growth, or during post-harvest storage of fruits, that is infested or
liable to be infested by fungi with the active component I and with the active component II in
any desired order or simultaneously.

The weight ratio of I:II is so selected as to give a synergistic fungicidal action. The synergistic
action of the composition is apparent from the fact that the fungicidal action of the
composition of I + II is greater than the sum of the fungicidal actions of I and II.

The method of the invention comprises applying to the plants to be treated or the locus
thereof in admixture or separately, a fungicidally effective aggregate amount of a compound I
and a compound of component b).
The term locus as used herein is intended to embrace the fields on which the treated crop plants are growing, or where the seeds of cultivated plants are sown, or the place where the seed will be placed into the soil. The term seed is intended to embrace plant propagating material such as cuttings, seedlings, seeds, germinated or soaked seeds.

The combinations are applied by treating the fungi or the seeds, plants or materials threatened by fungus attack, or the soil with a fungicidally effective amount of the active ingredients.

The novel combinations are extremely effective against phytopathogenic fungi. Some of the have a systemic action and can be used as foliar and soil fungicides, for post-harvest fruits during storage and for seed dressing.

The agents may be applied before or after infection of the materials, plants, during post-harvest storage of fruits or seeds by the fungi.

When applied to the plants or the post-harvest fruits the compound of formula I is applied at a rate of 10 to 150 g/100 l, particularly 20 to 100 g/100 l, e.g. 20, 50, or 100g/100 l, in association with 10 to 1000 g/100 l, particularly 30 to 600 g/100 l, e.g. 30 g/100 l, 40 g/100 l, 75 g/100 l, 80 g/100 l, 100 g/100 l, 125 g/100 l, 150 g/100 l, 175 g/100 l, 200 g/100 l, 300 g/100 l, 500 g/100 l, depending on the class of chemical employed as component b).

Where the component b) is compound II A for example 50 to 200 g a.i./100 l is applied in association with the compound of formula I. Where the component b) is compound II B for example 25 to 200 g a.i./100 l is applied in association with the compound of formula I.

In agricultural practice the application rates of the combination depend on the type of effect desired, and range from 5 to 1000 g of active ingredient per 100 l.

When the active ingredients are used for treating seed, rates of 0.001 to 50 g a.i. per kg, and preferably from 0.01 to 10g per kg of seed are generally sufficient.

The invention also provides fungicidal compositions comprising a compound of formula I and a compound of component b).
The composition of the invention may be employed in any conventional form, for example in the form of a twin pack, an instant granulate, a flowable or a wettable powder in combination with agriculturally acceptable adjuvants. Such compositions may be produced in conventional manner, e.g. by mixing the active ingredients with appropriate adjuvants (diluents or solvents and optionally other formulating ingredients such as surfactants).

The term diluent as used herein means any liquid or solid agriculturally acceptable material including carriers which may be added to the active constituents to bring them in an easier or improved applicable form, respectively, to a usable or desirable strength of activity. Suitable solvents are: aromatic hydrocarbons, preferably the fractions containing 8 to 12 carbon atoms, e.g. xylene mixtures or substituted napthalenes, phthalates, such as dibutyl phthalate or dioctyl phthalate, aliphatic hydrocarbons, such as cyclohexane or paraffins, alcohols and glycols and their ethers and esters, such as ethanol, ethylene glycol, ethylene glycol monomethyl or monoethyl ether, ketones, such as cyclohexanone, strongly polar solvents, such as N-methyl-2-pyrrolidone, dimethyl sulfoxide or dimethylformamide, as well as vegetable oils or epoxidised vegetable oils, such as epoxidised coconut oil or soybean oil; or water. The solid carriers used, e.g. for dusts and dispersible powders, are normally natural mineral fillers, such as calcite, talcum, kaolin, montmorillonite or attapulgite. In order to improve the physical properties it is also possible to add highly dispersed silicic acid or highly dispersed absorbent polymers. Suitable granulated adsorptive carriers are porous types, for example pumice, broken brick, sepiolite or bentonite, and suitable non-absorbent carriers are, for example, calcite or sand. In addition, a great number of materials of inorganic or organic nature can be used, e.g. especially dolomite or pulverized plant residues. Depending upon the nature of the compounds of formula I and component b) to be formulated, suitable surface-active compounds are non-ionic, cationic and/or anionic surfactants having good emulsifying, dispersing and wetting properties. The term "surfactants" will also be understood as comprising mixtures of surfactants. Particularly advantageous application-promoting adjuvants are also natural or synthetic phospholipids of the cephalin and lecithin series, e.g. phosphatidylethanolamine, phosphatidylserine, phosphatidylglycerol and lyssolecithin.

Particularly formulations to be applied in spraying forms such as water dispersible concentrates or wettable powders may contain surfactants such as wetting and dispersing agents, e.g. the condensation product of formaldehyde with naphthalene sulphonate, an
alkylarylsulphonate, a lignin sulphonate, a fatty alkyl sulphate, and ethoxylated alkylphenol and an ethoxylated fatty alcohol.

A seed dressing formulation is applied in a manner known per se to the seeds employing the combination of the invention and a diluent in suitable seed dressing formulation form, e.g. as an aqueous suspension or in a dry powder form having good adherence to the seeds. Such seed dressing formulations are known in the art. Seed dressing formulations may contain the single active ingredients or the combination of active ingredients in encapsulated form, e.g. as slow release capsules or microcapsules.

In general, the formulations include from 0.01 to 90% by weight of active agent, from 0 to 20% agriculturally acceptable surfactant and 10 to 99.99% solid or liquid adjuvant(s), the active agent consisting of at least the compound of formula I together with a compound of component b), and optionally other active agents, particularly microbicides or conservatives or the like. Concentrate forms of compositions generally contain in between about 2 and 80%, preferably between about 5 and 70% by weight of active agent. Application forms of formulation may for example contain from 0.01 to 20% by weight, preferably from 0.01 to 5% by weight of active agent.

The Examples which follow serve to illustrate the invention, "active ingredient" denoting a mixture of compound I and a compound of component b) in a specific mixing ratio.

Formulations may be prepared analogously to those described in, for example, WO 97/33890.

**Slow Release Capsule Suspension**

28 parts of a combination of the component I and a compound of component b), or of each of these compounds separately, are mixed with 2 parts of an aromatic solvent and 7 parts of toluene diisocyanate/polyethylene-polyphenylisocyanate-mixture (8:1). This mixture is emulsified in a mixture of 1.2 parts of polyvinylalcohol, 0.05 parts of a defoamer and 51.6 parts of water until the desired particle size is achieved. To this emulsion a mixture of 2.8 parts
1,6-diaminohexane in 5.3 parts of water is added. The mixture is agitated until the polymerization reaction is completed.

The obtained capsule suspension is stabilized by adding 0.25 parts of a thickener and 3 parts of a dispersing agent. The capsule suspension formulation contains 28% of the active ingredients. The medium capsule diameter is 8-15 microns. The resulting formulation is applied to seeds as an aqueous suspension in an apparatus suitable for that purpose.

**Seed Dressing Formulation**

25 parts of a combination of components I and II, 15 parts of dialkylphenoxypoly(ethylenoxy)ethanol, 15 parts of fine silica, 44 parts of fine kaolin, 0.5 parts of Rhodamine B as a colorant and 0.5 parts of Xanthan Gum are mixed and ground in a contraplex mill at approx. 10000 rpm to an average particle size of below 20 microns. The resulting formulation is applied to the seeds as an aqueous suspension in an apparatus suitable for that purpose.

Whereas commercial products will preferably be formulated as concentrates, the end user will normally employ dilute formulations.

**Biological Examples**

A synergistic effect exists whenever the action of an active ingredient combination is greater than the sum of the actions of the individual components.

The action to be expected E for a given active ingredient combination obeys the so-called COLBY formula and can be calculated as follows (COLBY, S.R. "Calculating synergistic and antagonistic responses of herbicide combination". Weeds, Vol. 15, pages 20-22; 1967):

\[
\text{ppm} = \text{milligrams of active ingredient (a.i.) per litre of spray mixture}
\]

\[
X = \% \text{ action by active ingredient I using p ppm of active ingredient}
\]

\[
Y = \% \text{ action by active ingredient II using q ppm of active ingredient}
\]
According to Colby, the expected (additive) action of active ingredients I+II using p+q ppm of active ingredient is

\[ E = X + Y - \frac{X \cdot Y}{100} \]

If the action actually observed (O) is greater than the expected action (E), then the action of the combination is superadditive, i.e. there is a synergistic effect.

Alternatively the synergistic action may also be determined from the dose response curves according to the so-called WADLEY method. With this method the efficacy of the a.i. is determined by comparing the degree of fungal attack on treated plants with that on untreated, similarly inoculated and incubated check plants. Each a.i. is tested at 4 to 5 concentrations. The dose response curves are used to establish the EC90 (i.e. concentration of a.i. providing 90% disease control) of the single compounds as well as of the combinations (EC90\text{observed}). The thus experimentally found values of the mixtures at a given weight ratio are compared with the values that would have been found were only a complementary efficacy of the components was present (EC 90 (A+B)\text{expected}). The EC90 (A+B)\text{expected} is calculated according to Wadley (Levi et al., EPPO- Bulletin 16, 1986, 651-657):

\[ \text{EC 90 (A+B)}\text{expected} = \frac{a + b}{a \cdot \frac{\text{EC90} (A)\text{observed}}{a} + b \cdot \frac{\text{EC90} (B)\text{observed}}{b}} \]

wherein a and b are the weight ratios of the compounds A and B in the mixture and the indexes (A), (B), (A+B) refer to the observed EC 90 values of the compounds A, B or the given combination A+B thereof. The ratio EC90 (A+B)\text{expected} / EC90 (A+B)\text{observed} expresses the factor of interaction (F). In case of synergism, F is >1.

Example B-1: Efficacy against Botrytis cinerea on apple
In an apple fruit cv. Golden Delicious 3 holes were drilled and each filled with 50 µl droplets of the formulated test composition. Two hours after application 50 µl of a spore suspension of B. cinerea (4 x 10^5 conidia/ml) were pipetted on the application sites. After an incubation period of 5 days at 20°C in a growth chamber the % infected fruit area was determined. The
activity was calculated relative to the disease rate on untreated apple fruits. The fungicide interactions in the mixture were calculated according to Colby.

**Results:**

<table>
<thead>
<tr>
<th>Fludioxonil mg a.i./l</th>
<th>Thiabendazole mg a.i./l</th>
<th>Ratio</th>
<th>% activity observed</th>
<th>% activity expected</th>
<th>SF Colby</th>
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Table 2

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<tr>
<th>Fludioxonil mg a.i./l</th>
<th>Imazalil mg a.i./l</th>
<th>Ratio</th>
<th>% activity observed</th>
<th>% activity expected</th>
<th>SF Colby</th>
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Example B-2: Efficacy against *Fusarium* on wheat

A conidia suspension of *Fusarium* (7 x 10^6 conidia/ml) is mixed with the formulated test composition. The mixture is applied into a pouch which was previously equipped with a filter paper. After application wheat seeds (cv. Orestis) are sown into then upper fault of the filter paper. The prepared pouches are then incubated for 11 days at approx. 10-18°C and a relative humidity of 100% with a photo period of 14 hours. The evaluation is made by assessing the degree of disease occurrence in the form of brown lesions on the roots.

Example B-3: Efficacy against *Penicillium* on lemon fruits

Lemon fruits were inoculated by placing 50 μl spore suspension with 0.1% tween 20 and a density of 500000 spores/ml into each hole (two holes/fruit) which are prepared by boring the holes into the lemon peel approximately 1 cm diameter and a depth of 1-1.5 mm. The fruits were stored in covered plastic boxes under light at 20°C and checked 5-7 days after inoculation.

The mixtures according to the invention exhibit good activity in these examples.
WHAT IS CLAIMED IS:

1. A method of combating phytopathogenic diseases on crop plants which comprises applying to the crop plants or the locus thereof being infested with said phytopathogenic disease an effective amount of
   a) 4-(2,2-difluoro-1,3-benzodioxol-7-yl)pyrrole-3-carbonitrile, component I
   in association with
   b) either 2-(thiazol-4-yl)benzimidazol, component IIA; or
   1-[(β-allyloxy-2,4-dichlorophenylethyl)]imidazole, component IIB
   which synergistically enhances the activity against phytopathogenic diseases.

2. A method according to claim 1, wherein component II is the compound IIA.

3. A method according to claim 1, wherein component II is the compound IIB.

4. A method according to any one of claims 1 to 3, wherein the phytopathogenic fungi belongs to the class of fungi imperfecti.

5. A fungicidal composition comprising a fungicidally effective combination of component a) and component b) as defined in claim 1, wherein the components are present in amounts which synergistically enhances the activity against phytopathogenic diseases.

6. A composition according to claim 5, wherein the weight ratio of a) to b) is between 20 : 1 and 1 : 20.

7. A composition according to claim 5 or 6, wherein component II is the compound IIA.

8. A composition according to claim 5 or 6, wherein component II is the compound IIB.

9. A composition according to any one of claims 5 to 8 to combat the class of fungi imperfecti.
10. A method according to any one of claims 1 to 4 for treating fruits during post-harvest storage.

11. A method according to any one of claims 1 to 4 and 10, which comprises treating the plants, or its locus of growth, or the post-harvest fruits, which is infected or liable to be infected, with at least one active component I and one active component II in any order or simultaneously.
### INTERNATIONAL SEARCH REPORT

**A. CLASSIFICATION OF SUBJECT MATTER**

| IPC | A01N43/36 // (A01N43/36, 43:78, 43:50) |

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

- Minimum documentation searched (classification system followed by classification symbols)
  - IPC 7 A01N

- Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

- Electronic data base consulted during the International search (name of data base and, where practical, search terms used)
  - CHEM ABS Data, EPO-Internal, WPI Data, PAJ, BIOSIS

### C. DOCUMENTS CONSIDERED TO BE RELEVANT

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<th>Category</th>
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<td>WO 98 57543 A (NOVARTIS ERFIND VERWALT GMBH; NOVARTIS AG (CH); MUELLER KASPAR (CH)) 23 December 1998 (1998-12-23)</td>
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* Further documents are listed in the continuation of box C.

* Patent family members are listed in annex.

* Special categories of cited documents:
  - *A* document defining the general state of the art which is not considered to be of particular relevance
  - *E* earlier document but published on or after the international filing date
  - *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
  - *O* of document referring to an oral disclosure, use, exhibition or other means
  - *P* document published prior to the international filing date but later than the priority date claimed
  - **X** later document published after the International filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
  - **X** document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
  - **Y** document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
  - **A** document member of the same patent family

**Date of the actual completion of the International search**

6 November 2001

**Date of mailing of the International search report**

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL – 2280 HV Rijswijk
Tel: (+31-70) 340-3040, Tx: 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Bertrand, F
INTERNATIONAL SEARCH REPORT

Box I  Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.: because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.: because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:

3. ☐ Claims Nos.: because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II  Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☑ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest.

☒ No protest accompanied the payment of additional search fees.
This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. Claims: 1,4-6,9-11(all partly) and 2,7

   method of combating phytopathogenic diseases and composition therefor containing fludioxonil and thiabendazole.

2. Claims: 1,4-6,9-11(all partly) and 3,8

   method of combating phytopathogenic diseases and composition therefor containing fludioxonil and imazalil.
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