

REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
PUBLICATION PARTICULARS AND ABSTRACT
[Section 32 (3) (a) - Regulations 22 (1) (g) and 31]

OFFICIAL APPLICATION NO		LODGING DATE	ACCEPTANCE DATE
21	01 2006/03847	22 15 May 2006	43 27-07-2007

INTERNATIONAL CLASSIFICATION	Not for publication
51 A01N	Classified by:

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TITLE OF INVENTION	
54	FUNGICIDAL MIXTURES FOR CONTROLLING RICE PATHOGENS

EARLIEST PRIORITY CLAIMED	COUNTRY	NUMBER	DATE
NB - Use International abbreviation for country (See Schedule 4)	33 DE	31 103 49 097.3	32 17 Oct 2003

57	Abstract (not more than 150 words) and figure of the drawings to which the abstract refers, are attached.	Number of sheets	26
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(12) NACH DEM VERTRAG ÜBER DIE INTERNATIONALE ZUSAMMENARBEIT AUF DEM GEBIET DES PATENTWESENS (PCT) VERÖFFENTLICHTE INTERNATIONALE ANMELDUNG

(19) Weltorganisation für geistiges Eigentum
Internationales Büro



(43) Internationales Veröffentlichungsdatum
28. April 2005 (28.04.2005)

PCT

(10) Internationale Veröffentlichungsnummer
WO 2005/036960 A3

(51) Internationale Patentklassifikation⁷: A01N 43/90 //
(A01N 43/90, 59:02)

(81) Bestimmungsstaaten (soweit nicht anders angegeben, für jede verfügbare nationale Schutzrechtsart): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LI, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(21) Internationales Aktenzeichen: PCT/EP2004/011257

(22) Internationales Anmeldedatum:
8. Oktober 2004 (08.10.2004)

(25) Einreichungssprache: Deutsch

(26) Veröffentlichungssprache: Deutsch

(84) Bestimmungsstaaten (soweit nicht anders angegeben, für jede verfügbare regionale Schutzrechtsart): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), eurasisches (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), europäisches (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

(30) Angaben zur Priorität:
103 49 097.3 17. Oktober 2003 (17.10.2003) DE

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Veröffentlicht:

— mit internationalem Recherchenbericht
— vor Ablauf der für Änderungen der Ansprüche geltenden Frist; Veröffentlichung wird wiederholt, falls Änderungen eintreffen

(88) Veröffentlichungsdatum des Internationalen Recherchenberichts: 7. Juli 2005

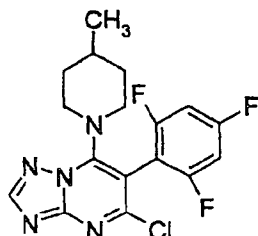
(74) Gemeinsamer Vertreter: BASF Aktiengesellschaft; 67056 Ludwigshafen (DE).

Zur Erklärung der Zweibuchstaben-Codes und der anderen Abkürzungen wird auf die Erklärungen ("Guidance Notes on Codes and Abbreviations") am Anfang jeder regulären Ausgabe der PCT-Gazette verwiesen.

(54) Title: FUNGICIDAL MIXTURES FOR CONTROLLING RICE PATHOGENS

WO 2005/036960 A3

(54) Bezeichnung: FUNGIZIDE MISCHUNGEN ZUR BEKÄMPFUNG VON REISPATHOGENEN



(I)

(57) Abstract: The invention relates to fungicidal mixtures for controlling rice pathogens. Said mixtures comprise as the active components 1) the triazolopyrimidine derivative of formula (I), and 2) sulfur (II), in a synergistically effective amount. The invention also relates to a method for controlling rice pathogens using mixtures of compound (I) with sulfur (II) and to the use of compound (I) together with sulfur (II) for producing said mixtures, and to agents containing said mixtures.

(57) Zusammenfassung: Fungizide Mischungen zur Bekämpfung von Reispathogenen, enthaltend als aktive Komponenten 1) das Triazolopyrimidinderivat der Formel (I), und 2) Schwefel (II), in einer synergistisch wirksamen Menge, Verfahren zur Bekämpfung von

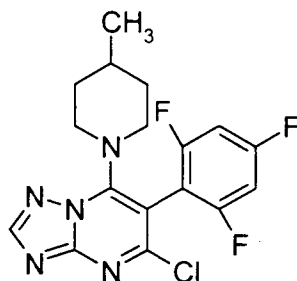
Reispathogenen mit Mischungen der Verbindung I mit Schwefel (II) und die Verwendung der Verbindung I mit Schwefel (II) zur Herstellung derartiger Mischungen sowie Mittel, die diese Mischungen enthalten.

Fungicidal mixtures for controlling rice pathogens

The present invention relates to fungicidal mixtures for controlling rice pathogens, which mixtures comprise, as active components,

5

1) the triazolopyrimidine derivative of the formula I,



and

10 2) sulfur (II),

in a synergistically effective amount.

Moreover, the invention relates to a method for controlling rice pathogens using
15 mixtures of the compound I with sulfur (II) and to the use of the compound I with sulfur (II) for preparing such mixtures and compositions comprising these mixtures.

The compound I, 5-chloro-7-(4-methylpiperidin-1-yl)-6-(2,4,6-trifluorophenyl)-[1,2,4]triazolo[1,5-a]pyrimidine, its preparation and its action against harmful fungi are known
20 from the literature (WO 98/46607).

The fungicidal action of sulfur (II) has been known for a long time. Finely divided sulfur is widely used as a fungicide against mildew diseases, mainly in fruit growing and viticulture.

25

Mixtures of triazolopyrimidine derivatives with sulfur (II) are known in a general manner from EP-A 988 790. The compound I is embraced by the general disclosure of this publication, but not explicitly mentioned. Accordingly, the combination of compound I with sulfur is novel.

30

The synergistic mixtures known from EP-A 988 790 are described as being fungicidally active against various diseases of cereals, fruit and vegetables, for example mildew on wheat and barley or gray mold on apples.

Owing to the special cultivation conditions of rice plants, the requirements that a rice fungicide has to meet are considerably different from those that fungicides used in cereal or fruit growing have to meet. There are differences in the application method :
5 in modern rice cultivation, in addition to foliar application, which is conventional in many places, the fungicide is applied directly onto the soil during or shortly after sowing. The fungicide is taken up into the plant via the roots and transported in the sap of the plant to the plant parts to be protected. In contrast, in cereal or fruit growing, the fungicide is usually applied onto the leaves or the fruits; accordingly, in these crops the systemic action of the active compounds is considerably less important.

10

Moreover, rice pathogens are typically different from those in cereals or fruit. *Pyricularia oryzae*, *Cochliobolus miyabeanus* and *Corticium sasakii* (syn. *Rhizoctonia solani*) are the pathogens of the diseases most prevalent in rice plants. *Rhizoctonia solani* is the only pathogen of agricultural significance from the sub-class
15 *Agaricomycetidae*. In contrast to most other fungi, this fungus attacks the plant not via spores but via a mycelium infection.

For this reason, findings concerning the fungicidal activity in the cultivation of cereals or fruit cannot be transferred to rice crops.

20

Practical agricultural experience has shown that the repeated and exclusive application of an individual active compound in the control of harmful fungi leads in many cases to a rapid selection of such fungus strains which have developed natural or adapted resistance against the active compound in question. Effective control of these fungi
25 with the active compound in question is then no longer possible.

To reduce the risk of selection of resistant fungus strains, mixtures of different active compounds are nowadays usually employed for controlling harmful fungi. By combining active compounds having different mechanisms of action, it is possible to ensure
30 successful control over a relatively long period of time.

A need exists, with a view to effective resistance management and an effective control of rice pathogens at application rates which are as low as possible, mixtures which, whilst having a reduced total amount of active compounds applied, have an improved
35 effect against the harmful fungi.

We have found that this need is fulfilled by the mixtures defined at the outset. Moreover, we have found that simultaneous, that is joint or separate, application of the compounds I and sulfur (II) or successive application of the compounds I and sulfur (II)

allows better control of rice pathogens than is possible with the individual active compounds.

5 The mixtures of the compound I and sulfur (II), or the compound I and sulfur (II) used simultaneously, that is jointly or separately, exhibit outstanding action against rice pathogens from the classes of the *Ascomycetes*, *Deuteromycetes* and *Basidiomycetes*. They can be used for the treatment of seed and as foliar- and soil-acting fungicides.

10 They are especially important for controlling harmful fungi on rice plants and their seeds, such as *Bipolaris* and *Drechslera* species, and also *Pyricularia oryzae*. They are particularly suitable for controlling brown spot of rice, caused by *Cochliobolus miyabeanus*.

15 In addition, the combination according to the invention of the compound I and sulfur (II) can also be used for controlling other pathogens, such as, for example, *Septoria* and *Puccinia* species in cereals and *Alternaria* and *Boytritis* species in vegetables, fruit and grapevines.

20 When preparing the mixtures, it is preferred to employ the pure active compounds I and II, to which further active compounds against harmful fungi or other pests, such as insects, arachnids or nematodes, or else herbicidal or growth-regulating active compounds or fertilizers can be added as required.

25 Further suitable active compounds in the above sense are, in particular, fungicides selected from the following group:

- acylalanines, such as benalaxyl, metalaxyl, ofurace or oxadixyl,
- amine derivatives, such as aldimorph, dodemorph, fenpropidin, guazatine, iminoctadine or tridemorph,
- 30 • antibiotics, such as cycloheximide, griseofulvin, kasugamycin, natamycin, polyoxin or streptomycin,
- azoles, such as bitertanol, bromoconazole, cyproconazole, difenoconazole, dinitroconazole, enilconazole, fenbuconazole, fluquinconazole, flusilazole, flutriafol, hexaconazole, imazalil, ipconazole, myclobutanil, penconazole, propiconazole,
- 35 • prochloraz, prothioconazole, simeconazole, tetraconazole, triadimefon, triadimenol, triflumizole or triticonazole,
- dicarboximides, such as myclozolin or procymidone,
- dithiocarbamates, such as ferbam, nabam, metam, propineb, polycarbamate, ziram or zineb,

- heterocyclic compounds, such as anilazine, boscalid, carbendazim, carboxin, oxycarboxin, cyazofamid, dazomet, famoxadone, fenamidone, fuberidazole, flutolanil, furametpyr, isoprothiolane, mepronil, nuarimol, probenazole, pyroquilon, silthiofam, thiabendazole, thifluzamide, tiadinil, tricyclazole or triforine,
- 5 • nitrophenyl derivatives, such as binapacryl, dinocap, dinobuton or nitrophthal-isopropyl,
- phenylpyrroles, such as fenpiclonil or fludioxonil,
- other fungicides, such as acibenzolar-S-methyl, carpropamid, chlorothalonil, cyflufenamid, cymoxanil, diclomezine, diclocymet, diethofencarb, edifenphos,
- 10 ethaboxam, fentin acetate, fenoxanil, ferimzone, fosetyl, hexachlorobenzene, metrafenone, pencycuron, propamocarb, phthalide, tolclofos-methyl, quintozene or zoxamide,
- strobilurins, such as fluoxastrobin, metominostrobin, oryastrobin, or pyraclostrobin,
- sulfenic acid derivatives, such as captafol,
- 15 • cinnamides and analogous compounds, such as flumetover.

In one embodiment of the mixtures according to the invention, the compounds I and II are admixed with a further fungicide III or two fungicides III and IV. Preference is given to mixtures of the compounds I and II with a component III. Particular preference is given to mixtures of the compounds I and II.

The compound I and the sulfur (II) can be applied simultaneously, that is jointly or separately, or in succession, the sequence, in the case of separate application, generally not having any effect on the result of the control measures.

In the control of harmful fungi pathogenic to rice plants, the separate or joint application of the compounds I and II or of the mixtures of the compounds I and II is carried out by spraying or dusting the seeds, the seedlings, the plants or the soils before or after sowing of the plants or before or after emergence of the plants. The compound I and sulfur (II) are preferably applied by spraying the leaves. The application of the compounds can also be carried out by applying granules or by dusting the soils.

The compound I and sulfur (II) are usually applied in a weight ratio of from 100:1 to 1:100, preferably from 20:1 to 1:50, in particular from 2:1 to 1:20.

The components III and, if appropriate, IV are, if desired, added in a ratio of from 20:1 to 1:20 to the compound I.

Depending on the type of compound and on the desired effect, the application rates of the mixtures according to the invention are from 1 kg/ha to 5 kg/ha.

Correspondingly, the application rates of the compound I are generally from 1 to 5 1000 g/ha, preferably from 10 to 750 g/ha, in particular from 20 to 500 g/ha.

Correspondingly, the application rates of sulfur (II) are generally from 1 to 5 kg/ha.

In the treatment of seed, the application rates of the mixture are generally from 0.001 to 10 5 kg/100 kg of seed, preferably from 0.01 to 3 kg/100 kg, in particular from 0.01 to 1 kg/100 kg.

The mixtures according to the invention or the compounds I and II can be converted into the customary formulations, for example solutions, emulsions, suspensions, dusts, 15 powders, pastes and granules. The application form depends on the particular purpose; in each case, it should ensure a fine and uniform distribution of the compound according to the invention.

The formulations are prepared in a known manner, for example by extending the active 20 compound with solvents and/or carriers, if desired using emulsifiers and dispersants. Solvents/auxiliaries which are suitable are essentially:

- water, aromatic solvents (for example Solvesso products, xylene), paraffins (for example mineral fractions), alcohols (for example methanol, butanol, pentanol, benzyl alcohol), ketones (for example cyclohexanone, gamma-butyrolactone), 25 pyrrolidones (NMP, NOP), acetates (glycol diacetate), glycols, fatty acid dimethylamides, fatty acids and fatty acid esters. In principle, solvent mixtures may also be used.
- carriers such as ground natural minerals (for example kaolins, clays, talc, chalk) and ground synthetic minerals (for example highly disperse silica, silicates); 30 emulsifiers such as nonionic and anionic emulsifiers (for example polyoxyethylene fatty alcohol ethers, alkylsulfonates and arylsulfonates) and dispersants such as lignin-sulfite waste liquors and methylcellulose.

Suitable surfactants are alkali metal, alkaline earth metal and ammonium salts of 35 lignosulfonic acid, naphthalenesulfonic acid, phenolsulfonic acid, dibutyl-naphthalenesulfonic acid, alkylarylsulfonates, alkyl sulfates, alkylsulfonates, fatty alcohol sulfates, fatty acids and sulfated fatty alcohol glycol ethers, furthermore condensates of sulfonated naphthalene and naphthalene derivatives with formaldehyde, condensates of naphthalene or of naphthalenesulfonic acid with phenol

and formaldehyde, polyoxyethylene octylphenyl ether, ethoxylated isooctylphenol, octylphenol, nonylphenol, alkylphenyl polyglycol ethers, tributylphenyl polyglycol ether, tristearylphenyl polyglycol ether, alkylaryl polyether alcohols, alcohol and fatty alcohol/ethylene oxide condensates, ethoxylated castor oil, polyoxyethylene alkyl
5 ethers, ethoxylated polyoxypropylene, lauryl alcohol polyglycol ether acetal, sorbitol esters, lignin-sulfite waste liquors and methylcellulose.

Substances which are suitable for the preparation of directly sprayable solutions, emulsions, pastes or oil dispersions are mineral oil fractions of medium to high boiling
10 point, such as kerosene or diesel oil, furthermore coal tar oils and oils of vegetable or animal origin, aliphatic, cyclic and aromatic hydrocarbons, for example toluene, xylene, paraffin, tetrahydronaphthalene, alkylated naphthalenes or their derivatives, methanol, ethanol, propanol, butanol, cyclohexanol, cyclohexanone, isophorone, strongly polar solvents, for example dimethyl sulfoxide, N-methylpyrrolidone and water.

15

Powders, materials for spreading and dustable products can be prepared by mixing or concomitantly grinding the active substances with a solid carrier.

Granules, for example coated granules, impregnated granules and homogeneous
20 granules, can be prepared by binding the active compounds to solid carriers. Examples of solid carriers are mineral earths such as silica gels, silicates, talc, kaolin, attaclay, limestone, lime, chalk, bole, loess, clay, dolomite, diatomaceous earth, calcium sulfate, magnesium sulfate, magnesium oxide, ground synthetic materials, fertilizers, such as, for example, ammonium sulfate, ammonium phosphate, ammonium nitrate, ureas, and
25 products of vegetable origin, such as cereal meal, tree bark meal, wood meal and nutshell meal, cellulose powders and other solid carriers.

In general, the formulations comprise from 0.01 to 95% by weight, preferably from 0.1
30 to 90% by weight, of the active compounds. The active compounds are employed in a purity of from 90% to 100%, preferably 95% to 100% (according to NMR spectrum).

The following are examples of formulations: 1. Products for dilution with water

A) Water-soluble concentrates (SL)

35 10 parts by weight of the active compounds are dissolved in water or in a water-soluble solvent. As an alternative, wetters or other auxiliaries are added. The active compound dissolves upon dilution with water.

B) Dispersible concentrates (DC)

20 parts by weight of the active compounds are dissolved in cyclohexanone with addition of a dispersant, for example polyvinylpyrrolidone. Dilution with water gives a dispersion.

5

C) Emulsifiable concentrates (EC)

15 parts by weight of the active compounds are dissolved in xylene with addition of calcium dodecylbenzenesulfonate and castor oil ethoxylate (in each case 5% strength). Dilution with water gives an emulsion.

10

D) Emulsions (EW, EO)

40 parts by weight of the active compounds are dissolved in xylene with addition of calcium dodecylbenzenesulfonate and castor oil ethoxylate (in each case 5% strength). This mixture is introduced into water by means of an emulsifier (Ultraturrax) and made into a homogeneous emulsion. Dilution with water gives an emulsion.

15

E) Suspensions (SC, OD)

In an agitated ball mill, 20 parts by weight of the active compounds are comminuted with addition of dispersants, wetters and water or an organic solvent to give a fine active compound suspension. Dilution with water gives a stable suspension of the active compound.

20

F) Water-dispersible granules and water-soluble granules (WG, SG)

50 parts by weight of the active compounds are ground finely with addition of dispersants and wetters and made into water-dispersible or water-soluble granules by means of technical appliances (for example extrusion, spray tower, fluidized bed). Dilution with water gives a stable dispersion or solution of the active compound.

25

G) Water-dispersible powders and water-soluble powders (WP, SP)

75 parts by weight of the active compounds are ground in a rotor-stator mill with addition of dispersants, wetters and silica gel. Dilution with water gives a stable dispersion or solution of the active compound.

30

2. Products to be applied undiluted

35

H) Dustable powders (DP)

5 parts by weight of the active compounds are ground finely and mixed intimately with 95% of finely divided kaolin. This gives a dustable product.

40

I) Granules (GR, FG, GG, MG)

0.5 part by weight of the active compounds is ground finely and associated with 95.5% carriers. Current methods are extrusion, spray-drying or the fluidized bed. This gives granules to be applied undiluted.

5 J) ULV solutions (UL)

10 parts by weight of the active compounds are dissolved in an organic solvent, for example xylene. This gives a product to be applied undiluted.

The active compounds can be used as such, in the form of their formulations or the use forms prepared therefrom, for example in the form of directly sprayable solutions, powders, suspensions or dispersions, emulsions, oil dispersions, pastes, dustable products, materials for spreading, or granules, by means of spraying, atomizing, dusting, spreading or pouring. The use forms depend entirely on the intended purposes; they are intended to ensure in each case the finest possible distribution of the active compounds according to the invention.

Aqueous use forms can be prepared from emulsion concentrates, pastes or wettable powders (sprayable powders, oil dispersions) by adding water. To prepare emulsions, pastes or oil dispersions, the substances, as such or dissolved in an oil or solvent, can be homogenized in water by means of a wetter, tackifier, dispersant or emulsifier. Alternatively, it is possible to prepare concentrates composed of active substance, wetter, tackifier, dispersant or emulsifier and, if appropriate, solvent or oil, and such concentrates are suitable for dilution with water.

25 The active compound concentrations in the ready-to-use preparations can be varied within relatively wide ranges. In general, they are from 0.0001 to 10%, preferably from 0.01 to 1%.

30 The active compounds may also be used successfully in the ultra-low-volume process (ULV), it being possible to apply formulations comprising over 95% by weight of active compound, or even to apply the active compound without additives.

35 Various types of oils, wetters, adjuvants, herbicides, fungicides, other pesticides, or bactericides may be added to the active compounds, if appropriate just immediately prior to use (tank mix). These agents are typically admixed with the agents according to the invention in a weight ratio of 1:10 to 10:1.

The compounds I and II or the mixtures or the corresponding formulations are applied by treating the harmful fungi or the plants, seeds, soils, areas, materials or spaces to be kept free from them with a fungicidally effective amount of the mixture or, in the case of separate application, of the compounds I and II. Application can be carried out before or after infection by the harmful fungi.

The fungicidal action of the compound and the mixtures can be demonstrated by the experiments below:

The active compounds, separately or jointly, were prepared as a stock solution with 0.25% by weight of active compound in acetone or DMSO. 1% by weight of the emulsifier Uniperol® EL (wetting agent having emulsifying and dispersing action based on ethoxylated alkylphenols) was added to this solution, and the solution was diluted with water to the desired concentration.

Use example – activity against brown spot of rice caused by *Cochliobolus miyabeanus*, protective application

Leaves of potted rice seedlings of the cultivar "Tai-Nong 67" were sprayed to runoff point with an aqueous suspension of the concentration of active compound stated below. The next day, the plants were inoculated with an aqueous spore suspension of *Cochliobolus miyabeanus*. The test plants were then placed in climatized chambers at 22 - 24°C and 95 - 99 % relative atmospheric humidity for six days. The extent of the development of the infection on the leaves was then determined visually.

Evaluation was carried out by determining the percentage of infected leaf area. These percentages were converted into efficacies.

The efficacy (E) is calculated as follows using Abbot's formula:

30

$$E = (1 - \alpha/\beta) \cdot 100$$

α corresponds to the fungicidal infection of the treated plants in % and

35 β corresponds to the fungicidal infection of the untreated (control) plants in %

An efficacy of 0 means that the infection level of the treated plants corresponds to that of the untreated control plants; an efficacy of 100 means that the treated plants were not infected.

The expected efficacies of mixtures of active compounds are determined using Colby's formula (R.S. Colby, Weeds, 15, 20-22, 1967) and compared with the observed efficacies.

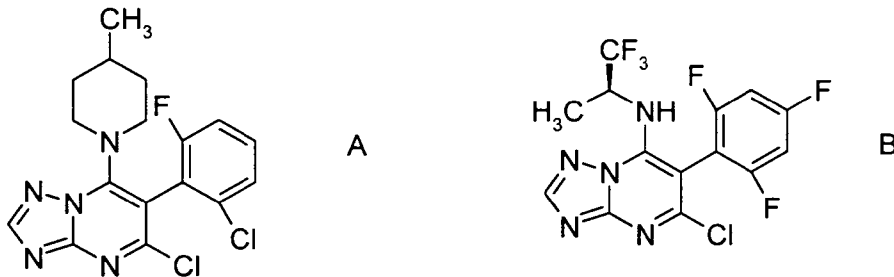
5 Colby's formula:

$$E = x + y - x \cdot y / 100$$

- 10 E expected efficacy, expressed in % of the untreated control, when using the mixture of the active compounds A and B at the concentrations a and b
- x efficacy, expressed in % of the untreated control, when using the active compound A at the concentration a
- y efficacy, expressed in % of the untreated control, when using the active compound B at the concentration b

15

The comparative compounds used were compounds A and B which are known from the sulfur mixtures described in EP-A 988 790:



20

Table A - individual active compounds

Ex-ample	Active compound	Concentration of active compound in the spray liquor [ppm]	Efficacy in % of the untreated control
1	control (untreated)	-	(84% infection)
2	I	4	28
		1	4
3	II (sulfur)	4	4
		1	4
4	comparative compound A	4	40
		1	28
5	comparative compound B	4	16
		1	0

Table B – mixtures according to the invention

Ex-ample	Mixture of active compounds Concentration Mixing ratio	Observed efficacy	Calculated efficacy*)
6	I + II 4 + 1 ppm 4:1	76	31
7	I + II 1 + 4 ppm 1:4	64	9

*) efficacy calculated using Colby's formula

Table C – comparative tests

Ex-ample	Mixture of active compounds Concentration Mixing ratio	Observed efficacy	Calculated efficacy*)
8	A + II 4 + 1 ppm 4:1	40	43
9	A + II 1 + 4 ppm 1:4	28	31
10	B + II 4 + 1 ppm 4:1	16	20
11	B + II 1 + 4 ppm 1:4	4	4

5 *) efficacy calculated using Colby's formula

The test results show that the mixtures according to the invention, by virtue of synergism, are considerably more effective than the sulfur mixtures known from EP-A 988 790, which are only moderately effective.

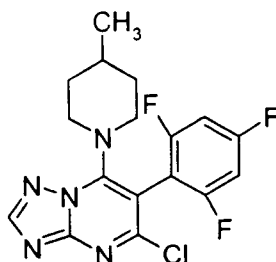
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"Comprises/comprising" when used in this specification is taken to specify the presence of stated features, integers, steps or components but does not preclude the presence or addition of one or more other features, integers, steps or components or groups thereof.

15

We claim:

1. A fungicidal mixture, which mixture comprises
- 5 1) the triazolopyrimidine derivative of the formula I



and

- 2) sulfur (II),
- 10 in a synergistically effective amount.
2. The fungicidal mixture as claimed in claim 1 comprising the compound of the formula I and sulfur (II) in a weight ratio of from 100:1 to 1:100.
- 15 3. The fungicidal composition comprising a liquid or solid carrier and a mixture as claimed in claim 1 or 2.
4. A method for controlling rice-pathogenic harmful fungi, which comprises treating the fungi, their habitat or the seed, the soil or the plants to be protected against fungal attack with an effective amount of the compound I and sulfur (II) as set forth in claim 1.
- 20 5. The method according to claim 4, wherein the compounds I and II as set forth in claim 1 are applied simultaneously, that is jointly or separately, or in succession.
- 25 6. The method according to claim 4, wherein the mixture as claimed in claim 1 or 2 is applied in an amount of from 1 kg/ha to 5 kg/ha.
- 30 7. The method according to claim 4 or 5, wherein the mixture as claimed in claim 1 or 2 is applied in an amount of from 0.001 to 5 kg/100 kg of seed.
- 35 8. The method according to any one of claims 4 to 7, wherein the harmful fungus *Cochliobolus miyabeanus* is controlled.

9. Seed comprising the mixture as claimed in claim 1 or 2 in an amount of from 0.001 to 5 kg/100.
- 5 10. The use of the compound I and sulfur (II) as set forth in claim 1 for preparing a composition suitable for controlling rice-pathogenic harmful fungi.
11. A fungicidal mixture as claimed in claim 1 or 2, substantially as hereinbefore described or exemplified.
- 10 12. A fungicidal mixture including any new and inventive integer or combination of integers, substantially as herein described.
13. A fungicidal composition as claimed in claim 3, substantially as hereinbefore described or exemplified.
- 15 14. A fungicidal composition including any new and inventive integer or combination of integers, substantially as herein described.
- 20 15. A method according to the invention for controlling harmful fungi, substantially as hereinbefore described or exemplified.
16. A method for controlling harmful fungi including any new and inventive integer or combination of integers, substantially as herein described.
- 25 17. Seed as claimed in claim 9, substantially as hereinbefore described or exemplified.
18. Seed according to the invention including any new and inventive integer or combination of integers, substantially as herein described.
- 30 19. The use of the compound I and compound II as claimed in claim 10, substantially as hereinbefore described or exemplified.
- 35 20. The use of the compound I and compound II including any new and inventive integer or combination of integers, substantially as herein described.