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- (54) Benævnelse: **SYNERGISTISKE KULTURPLANTEFORLIGELIGE KOMBINATIONER INDEHOLDENDE HERBICIDER FRA GRUPPEN AF BENZOYL CYCLOHEXANDIONER TIL ANVENDELSE I RISKULTURER**
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## Description

The invention relates to the technical field of plant protection compositions which can be used against unwanted  
5 vegetation and which comprise, as active substances, a combination of at least two herbicides.

More especially, it relates to herbicidal combinations for use  
10 in rice which comprise, as active substances, a herbicide selected from the group consisting of benzoylcyclohexanediones in combination with at least one additional herbicide.

Herbicides from the abovementioned group of  
15 benzoylcyclohexanediones are known from numerous documents. Thus, for example, the herbicidal action of numerous such compounds is described in WO 00/21924. Some of the benzoylcyclohexanediones mentioned therein exhibit a satisfactory herbicidal action against harmful plants occurring in rice crops.

20 However, in practice, there are frequently disadvantages associated with use of the benzoylcyclohexanediones known from these documents. Thus, the herbicidal activity is not always satisfactory or, with a satisfactory herbicidal activity,  
25 undesirable damage to the rice plants is observed.

The effectiveness of herbicides depends, inter alia, on the  
30 type of herbicide used, the application rate thereof, the composition, the harmful plants to be controlled each time, the climatic and soil conditions, and the like. A further criterion is the duration of the action or the rate of degradation of the herbicide. Changes in the sensitivity of harmful plants to an active substance which may occur with  
35 relatively long use or in geographically restricted areas are also to be taken into account, if appropriate. Such changes are expressed as a more or less serious loss in activity and can only to a limited extent be compensated for by higher herbicide application rates.

Because of the multitude of possible influencing factors, there is virtually no individual active substance which combines in itself the properties desired for different requirements, in particular with regard to the harmful plant species and the climatic zones. In addition, there is the constant problem of achieving the effect with an ever lower herbicide application rate. A lower application rate reduces not only the amount of an active substance required for the application but generally also reduces the amounts of formulation auxiliaries necessary. Both reduce the economic cost and improve the ecological compatibility of the herbicide treatment.

One method frequently used for improving the application profile of a herbicide consists in combining the active substance of one or more other active substances which contribute the additional properties desired. However, the combined use of several active substances not infrequently results in phenomena of physical and biological incompatibility, e.g. lack of stability of a combined formulation, decomposition of an active substance or antagonism of the active substances. On the other hand, what is desired are combinations of active substances with a favorable activity profile, high stability and the greatest possible synergistically strengthened activity which makes possible a reduction in the application rate in comparison with individual application of the active substances to be combined.

WO 02/089582 and WO 02/085118 describe herbicidal mixtures of particular benzoyl-1,3-cyclohexanediones with various herbicides. WO 02/085120 describes herbicidal mixtures of particular benzoyl-1,3-cyclohexanediones with safeners. However, in practice, there are serious disadvantages to these mixtures. Thus, their compatibility with regard to useful plants, in particular rice, is not always satisfactory and their activity with regard to harmful plants is likewise not

always satisfactory.

It is an object of the present invention to provide herbicidal combinations, in particular for use in rice crops, with  
5 improved properties in comparison with the state of the art.

The invention relates to herbicidal combinations, characterized in that they comprise an effective amount of

10 A) the compound tembotrione and its agriculturally customary salts (component A),

B) the compound benzobicyclon (component B),

15 and

C) if appropriate, the safener isoxadifen-ethyl (component C).

20 The combinations according to the invention comprise the components A, B and C in a weight ratio of a:b:c, in which a and b can assume, in each case independently of one another, values of from 1 to 200, preferably 1 to 100, and c can assume a value of from 0 to 200, preferably 0 to 100.

25

The terms "component A", "herbicide A" and "active substance A" are to be understood as synonymous in the context of the present description. The same applies for the terms "component B", "herbicide B" and "active substance B" and also "component  
30 C", "safener C" and "active substance C".

In the combinations according to the invention, it is generally necessary to have application rates in the range from 10 to 1000 g, preferably 10 to 500 g, of active substance  
35 per hectare (ai/ha) of the component A and 1 to 1000 g, preferably 5 to 500 g, of the component B. Component C is normally used in an application rate in the range from 0 to 500 g, preferably 0 to 400 g, of active substance per hectare

(ai/ha).

Optimum weight ratios can depend on the respective field of application, on the weed spectrum and on the active substance combination used and can be determined in preliminary experiments.

The active substances mentioned above with their common names are well known, for example from "The Pesticide Manual", 14th edition, 2006, British Crop Protection Council, and the website "<http://www.alanwood.net/pesticides/>". If, in the context of this description, the shortened form of the common name of an active substance is used, this each time includes all common routers, such as the esters and salts, and isomers, in particular optical isomers, in particular the commercially available form or forms. If an ester or salt is described by the common name, this also each time includes all other common derivatives, such as other esters and salts, the free acids and neutral compounds, and isomers, in particular optical isomers, in particular the commercially available form or forms. The chemical compound names given describe at least one of the compounds included under the common name, frequently a preferred compound.

The combinations according to the invention are very well suited to the selective controlling of harmful plants in rice crops.

The combinations according to the invention can be used in all types of application normal for rice herbicides. They are particularly advantageously used in the spray application and in the submerged application. In the submerged application, the paddy water already covers the ground by up to 30 mm at the time of the application. The combinations according to the invention are then directly placed in the paddy water, e.g. in the form of granules. Worldwide, the spray application is used predominantly with seeded rice and the submerged application is used predominantly with transplanted rice.

The combinations according to the invention include a broad weed spectrum. They are suitable for example for the controlling of annual and perennial harmful plants, such as, for example, from the species *Abutilon*, *Alopecurus*, *Avena*, *Chenopodium*, *Cynodon*, *Cyperus*, *Digitaria*, *Echinochloa*, *Elymus*, *Galium*, *Ipomoea*, *Lamium*, *Matricaria*, *Scirpus*, *Setaria*, *Sorghum*, *Veronica*, *Viola* and *Xanthium*, in particular *Echinochloa* spp., *Leptochloa* spp., *Scirpus* spp., *Cyperus* spp., *Sagittaria* spp., *Monochoria* spp., *Lindernia* spp., *Eleocharis* spp. and *Sesbania* spp.

The herbicidal combinations according to the invention are also distinguished in that the effective dosages of the components A and B used in the combinations are reduced with regard to an individual dosage, so that a reduction in the necessary application rates of the active substances is rendered possible.

The herbicidal combinations according to the invention exhibit, in a preferred embodiment, synergistic effects with simultaneously high compatibility with regard to cultivated plants. The synergistic effects and the high compatibility with regard to cultivated plants can be observed, e.g., with combined application of the components A, B and C; however, it can also frequently be detected when the active substances are applied at different times (splitting). It is also possible to apply the individual herbicides and safeners or the herbicidal safener combinations in several portions (sequential application), e.g. pre-emergence applications, followed by post-emergence applications or early post-emergence applications, followed by medium or late post-emergence applications. Preference is given in this connection to the combined or virtually simultaneous application of the active substances of the herbicide combination according to the invention.

The synergistic effects allow a reduction in the application

rates of the individual active substances, a greater potency at the same application rate, the control of species hitherto not included (gaps), an extension of the period of application and/or a reduction in the number of individual applications  
5 necessary and, as a result for the user, weed controlling systems which are more advantageous economically and ecologically.

The invention also includes those herbicidal combinations  
10 which, in addition to the components A, B and C, if appropriate also comprise one or more additional agrochemical active substances with a different structure, such as herbicides, insecticides, fungicides or safeners. The preferred conditions explained above and below are likewise  
15 valid for such combinations. These additional agrochemical active substances can be applied both in the combinations according to the invention, as "ready mix", and as "tank mix", by jointly diluting the separately formulated or partially separately formulated components.

20 The invention likewise also in particular includes those combinations which, in addition to the components A, B and C, also comprise fertilizers, such as ammonium sulfate, ammonium nitrate, urea, potassium nitrate and mixtures thereof. The preferred conditions explained above and below are likewise  
25 valid for such combinations.

The invention furthermore also includes those combinations which, in addition to the components A, B and C, also comprise  
30 adjuvants, such as emulsifiers, dispersants, mineral and vegetable oils, and mixtures thereof. The preferred conditions explained above and below are likewise valid for such combinations.

35 The combinations according to the invention can exist both as mixed formulations of the herbicides A and B and also the safener C, if appropriate with additional conventional formulation auxiliaries, which are then used in the



conventional way diluted with water, or be prepared as "tank mixes" by jointly diluting the separately formulated or partially separately formulated components with water or with aqueous solutions of fertilizers, for example such as  
5 mentioned above.

The combinations according to the invention are very well suited to controlling harmful plants, in particular harmful plants in rice crops. Another subject matter of the invention  
10 is accordingly a method for controlling unwanted vegetation, characterized in that one or more of the combinations according to the invention are applied to the harmful plants, to plant parts thereof or to the area under cultivation.

15 The components A, B and, if appropriate, C can be formulated in different ways, depending on which biological and/or chemical/physical parameters are specified. The following are possible, for example, as general formulation possibilities:  
20 wettable powders (WP), emulsifiable concentrates (EC), aqueous solutions (SL), emulsions (EW), such as oil-in-water and water-in-oil emulsions, sprayable solutions or emulsions, oil- or water-based dispersions, suspoemulsions, dustable  
powders (DP), seed dressings, granules for soil application or broadcasting, water-dispersible granules (WG), ULV  
25 formulations, microcapsules or waxes.

The individual formulation types are known in principle and are described, for example, in: Winnacker-Küchler, "Chemische Technologie" [Chemical Technology], Volume 7, C. Hauser  
30 Verlag, Munich, 4th edition, 1986; van Valkenburg, "Pesticide Formulations", Marcel Dekker, N.Y., 1973; K. Martens, "Spray Drying Handbook", 3rd Ed., 1979, G. Goodwin Ltd., London. The formulation auxiliaries necessary, such as inert materials, surfactants, solvents and additional additives, are likewise  
35 known and are described, for example, in: Watkins, "Handbook of Insecticide Dust Diluents and Carriers", 2nd Ed., Darland Books, Caldwell, N.J.; H.v. Olphen, "Introduction to Clay Colloid Chemistry", 2nd Ed., J. Wiley & Sons, N.Y.; Marsden,

"Solvents Guide", 2nd Ed., Interscience, N.Y., 1950; McCutcheon's, "Detergents and Emulsifiers Annual", MC Publ. Corp., Ridgewood, N.J.; Sisley and Wood, "Encyclopedia of Surface Active Agents", Chem. Publ. Co. Inc., N.Y., 1964; 5 Schönfeldt, "Grenzflächenaktive Äthylenoxidaddukte" [Surface-active Ethylene Oxide Adducts], Wiss. Verlagsgesellschaft, Stuttgart, 1976; Winnacker-Küchler, "Chemische Technologie" [Chemical Technology], Volume 7, C. Hauser Verlag Munich, 4th Ed., 1986.

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Based on these formulations, combinations with additional pesticidally active substances, such as other herbicides, fungicides or insecticides, and also safeners, fertilizers and/or growth regulators, can also be prepared, e.g. in the 15 form of a ready mix or as tank mix.

20

Wettable powders are preparations which can be uniformly dispersed in water and which, in addition to the active substance, also comprise ionic or nonionic surfactants (wetting agents, dispersants), e.g. polyoxyethylated 20 alkylphenols, polyethoxylated fatty alcohols or fatty amines, alkanesulfonates or alkylbenzenesulfonates, sodium lignosulfonate, sodium 2,2'-dinaphthylmethane-6,6'-disulfonate, sodium dibutyl-naphthalenesulfonate or sodium 25 oleoymethyltaurinate, in addition to a diluent or inert substance.

30

Emulsifiable concentrates are prepared by dissolving the active substance in an organic solvent, e.g. butanol, cyclohexanone, dimethylformamide, xylene or also higher-boiling aromatic compounds or hydrocarbons, with addition of one or more ionic or nonionic surfactants (emulsifiers). Use may be made, as emulsifiers, for example, of: calcium 35 alkylarylsulfonates, such as calcium dodecylbenzenesulfonate, or nonionic emulsifiers, such as fatty acid polyglycol esters, alkylaryl polyglycol ethers, fatty alcohol polyglycol ethers, propylene oxide/ethylene oxide condensation products, alkyl polyethers, sorbitan fatty acid esters, polyoxyethylene

sorbitan fatty acid esters or polyoxyethylene sorbitol esters.

Dustable powders are obtained by milling the active substance with finely divided solid materials, e.g. talc, natural clays, such as kaolin, bentonite and pyrophyllite, or diatomaceous earth.

Granules can be prepared either by spraying the active substance onto adsorptive granulated inert material or by applying active substance concentrates to the surface of carriers, such as sand or kaolinite, or of granulated inert material using binders, e.g. polyvinyl alcohol, sodium polyacrylate or else mineral oils. Suitable active substances can also be granulated in the standard way for the preparation of fertilizer granules, if desired as a mixture with fertilizers. Water-dispersible granules are generally prepared according to methods such as spray drying, fluidized-bed granulation, disk granulation, mixing with high-speed mixers and extrusion without solid inert material.

20

The agrochemical compositions generally comprise from 0.1 to 99 percent by weight, in particular from 0.2 to 95 percent by weight, of active substances A, B and, if appropriate, C, and also from 99.8 to 5 percent by weight of formulation agents customary in plant protection, the following concentrations being normal according to the type of formulation: in wettable powders, the active substance concentration is, e.g., from approximately 10 to 95 percent by weight, the balance for 100 percent by weight consisting of standard formulation constituents. With emulsifiable concentrates, the active substance concentration can be, e.g., from 5 to 80 percent by weight. Formulations in the form of dust for the most part comprise from 5 to 20 percent by weight of active substance, sprayable solutions from approximately 0.2 to 25 percent by weight of active substance. With granules, such as dispersible granules, the active substance content partly depends on whether the active compound is present in liquid or solid form and on which granulation auxiliaries and fillers are being

35

used. With water-dispersible granules, the content is generally between 10 and 90 percent by weight. In addition, the active substance formulations mentioned comprise, if appropriate, the stickers, wetting agents, dispersing agents, emulsifying agents, preservatives, antifreeze agents, solvents, fillers, colorants, carriers, antifoaming agents, evaporation inhibitors, pH regulators or viscosity regulators which are standard in each case.

10 For use, the formulations existing in commercially available form are, if appropriate, diluted in the standard way, e.g. using water for wettable powders, emulsifiable concentrates, dispersions and water-dispersible granules. Compositions in the form of dusts, soil granules, granules for broadcasting and sprayable solutions are normally not diluted further with additional inert materials before use.

The active substances can be applied to the plants, plant parts, plant seeds or the area under cultivation (arable land), preferably to the green plants and plant parts and, if appropriate, additionally to the arable land.

One possibility of use is the joint application of the active substances in the form of tank mixes, where the concentrated formulations, which are optimally formulated, of the individual active substances are mixed together in a tank with water and the spray mixture obtained is applied.

A joint herbicidal formulation of the combination according to the invention of components A, B and, if appropriate, C has the advantage of being able to be applied more easily because the amounts of the components have already been adjusted to the correct ratio to one another. Moreover, the auxiliaries in the formulation can be optimally matched to one another, while a tank mix of different formulations can give undesirable combinations of auxiliaries.

#### **A. Formulation examples**

- 5 a) A dustable powder (WP) is obtained by mixing 10 parts by weight of an active substance/active substance mixture and 90 parts by weight of talc as inert material and comminuting in a hammer mill.
- 10 b) A wettable powder readily dispersible in water (WG) is obtained by mixing 25 parts by weight of active substance/active substance mixture, 64 parts by weight of kaolin-comprising quartz as inert material, 10 parts by weight of potassium lignosulfonate and 1 part by weight of sodium oleoylmethyltaurinate as wetting and dispersing agent and milling in a pin mill.
- 15 c) A dispersion concentrate readily dispersible in water is obtained by mixing 20 parts by weight of an active substance/active substance mixture with 6 parts by weight of alkylphenol polyglycol ether (Triton X 207), 3 parts by weight of isotridecanol polyglycol ether (8 EO) and 71 parts by weight of paraffinic mineral oil (boiling range, e.g., 20 approximately 255 to 277°C) and milling in a friction ball mill to a fineness of less than 5 microns.
- 25 d) An emulsifiable concentrate (EC) is obtained from 15 parts by weight of an active substance/active substance mixture, 75 parts by weight of cyclohexanone as solvent and 10 parts by weight of oxyethylated nonylphenol as emulsifier.
- 30 e) A water-dispersible granule is obtained by mixing 75 parts by weight of an active substance/active substance mixture,
- 10 parts by weight of calcium lignosulfonate,
- 35 5 parts by weight of sodium lauryl sulfate,
- 3 parts by weight of polyvinyl alcohol and

7 parts by weight of kaolin

milling on a pin mill and granulating the powder in a  
5 fluidized bed by spraying on water as granulation liquid.

f) A water-dispersible granule is also obtained by  
homogenizing and precomminuting

10 25 parts by weight of an active substance/active substance  
mixture,

5 parts by weight of sodium 2,2'-dinaphthylmethane-6,6'-  
disulfonate,

15

2 parts by weight of sodium oleoymethyltaurinate,

1 part by weight of polyvinyl alcohol,

20 17 parts by weight of calcium carbonate and

50 parts by weight of water

on a colloid mill, subsequently milling on a bead mill and  
25 atomizing and drying the suspension thus obtained in a spray  
tower using a single-substance nozzle.

## **B. Biological examples**

30 Post-emergence weed action

Seeds or root pieces of harmful mono- and dicotyledonous  
plants are placed in sandy clay soil in pots, covered with  
earth and secured in a greenhouse under good growing  
35 conditions (temperature, air humidity, water supply).  
Approximately three weeks after sowing, the plants are treated  
at the 2- to 3-leaf stage with the herbicidal active  
substances or the combinations according to the invention. The

combinations according to the invention formulated as wettable powders or as emulsion concentrates are sprayed, in spray application, on to the green plant parts in different dosages with a water application rate of 600 to 800 l/ha (corrected).

5 Immediately up to a few days after application, the test receptacles accumulate water over the ground surface by up to 30 mm. With the water application (submerged application), on the other hand, the ground in the closed test receptacle is already covered with the paddy water up to 30 mm at the time  
10 of the application. The formulated active substances are here added directly to the paddy water. After an exposure time of the test plants in the greenhouse of an additional 3 to 4 weeks under optimum growing conditions, the effect of the preparations is evaluated visually in comparison with  
15 untreated controls. The combinations according to the invention exhibit, even in post-emergence, a very good herbicidal activity against a broad spectrum of economically important harmful plants. In this connection, actions of the combinations according to the invention which exceed the  
20 formal sum of the actions of the herbicides when applied individually are frequently observed. The values observed for the tests show, with suitable low dosages, an action of the combinations which lie above the expected values according to Colby.

25  
When the combinations according to the invention are used, herbicidal actions on a harmful plant species which exceed the formal sum of the actions of the herbicides present when applied singly are frequently observed. Alternatively, it can  
30 in many cases be observed that a lower application rate for the herbicidal combination is needed in order to achieve, in comparison with the individual preparations, the same action with a harmful plant species. Such improvements in action or improvements in effectiveness or economies in application rate  
35 are a strong indication of a synergistic effect.

If the observed activity values already exceed the formal sum of the values for the tests with individual applications, then

they likewise exceed the expected value according to Colby, which is calculated according to the following formula and is likewise regarded as an indication of synergy (cf. S.R. Colby in Weeds, 15 (1967), pp. 20 to 22):

5

$$E = A + B - \frac{A \times B}{100}$$

In this connection:

10 A, B = action of the component A or B in percent at a dosage of a or b grams ai /ha,

E = expected value in % at a dosage of a+b grams ai/ha.

15 The values observed for the test examples mentioned below are **greater than** (harmful plants) or **lower than** (cultivated plants) the expected values according to Colby.

The abbreviations are:

20

SCPSS	Scirpus juncoides
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Table 1: Herbicidal active substances

<b>Compound</b>
tembotrione: A1
benzobicyclon: B5
isoxadifen- ethyl: C1

25 Table 2: Post-emergence action

<b>Compound</b>	<b>Dosage</b> [g a.i./ha]	<b>Action</b> <b>against</b> <b>SCPSS</b>
A1	12.5	40%



B5	37.5	60%
A1 + B5	12.5 + 37.5	90%
<b>Colby expected value:</b>		76%
<b>Difference:</b>		+18%

## Patentkrav

1. Herbicide kombinationer, kendetegnet ved et virksomt indhold af

- 5 A) forbindelsen tembotrione samt dennes i landbruget gængse salte (bestanddel A) og  
B) forbindelsen benzobicyclon (bestanddel B).

10 2. Herbicide kombinationer ifølge krav 1, kendetegnet ved et virksomt indhold af

- A) forbindelsen tembotrione samt dennes i landbruget gængse salte (bestanddel A),  
B) forbindelsen benzobicyclon (bestanddel B) og  
C) safeneren isoxadifen-ethyl (bestanddel C).

15

3. Fremgangsmåde til bekæmpelse af uønsket plantevækst i riskulturer, kendetegnet ved, at man applicerer en eller flere herbicide kombinationer ifølge krav 1 eller 2 på de skadelige planter, plantedele heraf eller på dyrkningsarealet.

20

4. Anvendelse af en herbicid kombination ifølge et af kravene 1 til 2 til bekæmpelse af uønsket plantevækst i riskulturer.