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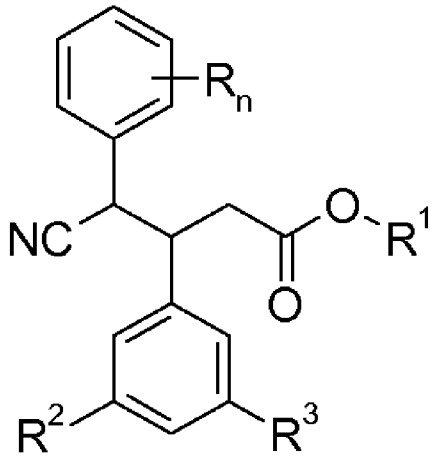
(54) Title: SUBSTITUTED CYANOBUTYRATES HAVING HERBICIDAL ACTION



(I)

(57) Abstract: Substituted cyanobutyrates of the formula I in which the variables are defined according to the description, processes and intermediates for preparing the compounds of the formula I and their N-oxides, their agriculturally suitable salts, compositions comprising them and their use as herbicides, and also a method for controlling unwanted vegetation.

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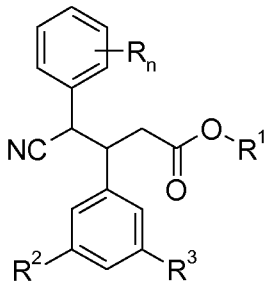
Substituted cyanobutyrates having herbicidal action

Description

The present invention relates to a substituted cyanobutyrate compound of the formula I



I

in which the variables have the following meaning:

R is halogen, cyano, nitro, C_1-C_4-alkyl, C_1-C_4-haloalkyl, C_2-C_6-alkenyl, C_2-C_6-alkynyl, C_1-C_4-alkoxy, C_1-C_4-alkylthio or Z-(tri-C_1-C_4-alkyl)silyl;

R^1 is hydrogen, Z-CN, C_1-C_8-alkyl, Z-C_3-C_6-cycloalkyl, C_1-C_8-haloalkyl, C_1-C_4-alkoxy-C_1-C_4-alkyl, C_1-C_4-alkylthio-C_1-C_4-alkyl, C_3-C_8-alkenyl, Z-C_3-C_6-cycloalkenyl, C_3-C_8-alkynyl, NR^iR^{ii}, Z-(tri-C_1-C_4-alkyl)silyl, Z-N=C(R^a)$_2$, Z-C(R^a)=NR^a, Z-C(=T)-R^a, Z-P(=T)(R^a)$_2$, Z-phenyl, a 3- to 7-membered monocyclic or 9- or 10-membered bicyclic saturated, unsaturated or aromatic heterocycle which contains 1, 2, 3 or 4 heteroatoms selected from the group consisting of O, N and S, which may carry carbonyl or thiocarbonyl groups and which is attached via carbon or nitrogen,

T is O or S;

R^a is hydrogen, TH, C_1-C_8-alkyl, C_1-C_4-haloalkyl, Z-C_3-C_6-cycloalkyl, C_2-C_8-alkenyl, Z-C_5-C_6-cycloalkenyl, C_2-C_8-alkynyl, Z-C_1-C_6-alkoxy, Z-C_1-C_4-haloalkoxy, Z-C_3-C_8-alkenyloxy, Z-C_3-C_8-alkynyloxy, Z-C_1-C_4-alkylthio, Z-C_1-C_4-haloalkylthio, Z-C_3-C_8-alkenylthio, Z-C_3-C_8-alkynylthio, NR^iR^{ii}, C_1-C_6-alkylsulfonyl, oxy-C_1-C_3-alkyleneoxy, Z-C(=T)-R^A, Z-T-C(=T)-R^A, Z-(tri-C_1-C_4-alkyl)silyl, Z-phenyl, Z-phenoxy, Z-phenylamino or a 3- to 7-membered monocyclic or 9- or 10-membered bicyclic heterocycle which contains 1, 2, 3 or 4 heteroatoms selected from the group consisting of O, N and S, where the cyclic groups are unsubstituted or may carry 1, 2, 3 or 4 groups R^b and/or carbonyl or thiocarbonyl groups;

R^i, R^{ii} independently of one another are hydrogen, C_1-C_8-alkyl, C_1-C_4-haloalkyl, C_3-C_8-alkenyl, C_3-C_8-alkynyl, Z-C_3-C_6-cycloalkyl, Z-C_1-C_8-alkoxy, Z-C_1-C_8-alkylthio, Z-C_1-C_8-haloalkoxy, Z-C_1-C_8-haloalkylthio, Z-C(=T)-R^A, where R^A is TH, C_1-C_4-alkyl, C_1-C_4-alkoxy or C_1-C_4-alkylthio;

R^i and R^{ii} together with the nitrogen atom to which they are attached may also form a 3- to 7-membered monocyclic or 9- or 10-

membered bicyclic heterocycle which contains 1, 2, 3 or 4 heteroatoms selected from the group consisting of O, N and S and which may carry carbonyl or thiocarbonyl groups;

R^b independently of one another are Z-CN, Z-TH, Z-NO_2, Z-halogen, C_1-C_8-alkyl, C_1-C_4-haloalkyl, C_2-C_8-alkenyl, C_2-C_8-alkynyl, Z-C_1-C_8-alkoxy, Z-C_1-C_8-alkylthio, Z-C_1-C_8-haloalkylthio, Z-C_1-C_8-haloalkoxy, Z-C_3-C_{10}-cycloalkyl, T-Z-C_3-C_{10}-cycloalkyl, Z-C(=T)-R^a, NR^iR^{ii}, Z-(tri-C_1-C_4-alkyl)silyl, Z-phenyl and $S(O)_mR^{bb}$, where

R^{bb} is C_1-C_8-alkyl or C_1-C_6-haloalkyl;

m is 0, 1 or 2;

R^b together with the group R^b attached to the adjacent carbon atom may also form a five- or six-membered saturated or partially or fully unsaturated ring which, in addition to carbon atoms, may contain 1, 2 or 3 heteroatoms selected from the group consisting of O, N and S;

Z is a covalent bond or C_1-C_8-alkylene;

n is 0, 1, 2, 3, 4 or 5;

R^2,R^3 independently of one another are halogen, cyano, nitro, C_1-C_4-alkyl, C_1-C_4-alkoxy, $S(O)_mR^{bb}$, NR^iR^{ii}, C_1-C_4-haloalkyl or C_1-C_4-haloalkoxy;

where in the groups R and R^1 and their subsubstituents, the carbon chains and/or the cyclic groups may be partially or fully substituted by groups R^a and/or R^b,

or an N-oxide or an agriculturally suitable salt thereof;

with the proviso that R^2 and R^3 are not both chlorine if the index n is 0 or 1 and R is halogen or R_n is 3,5-Cl_2 and R^1 is C_1-C_4-alkyl or H; and

R^2 and R^3 are not both fluorine if the index n is 1 and R is halogen and R^1 is C_1-C_4-alkyl.

Moreover, the invention relates to processes and intermediates for preparing the compounds of the formula I and the N-oxides thereof, the agriculturally usable salts thereof, to compositions comprising them and to their use as herbicides, i.e. for controlling harmful plants, and also to a method for controlling unwanted vegetation which comprises allowing a herbicidally effective amount of at least one compound of the formula I or of an agriculturally suitable salt thereof to act on plants, their seed and/or their habitat.

Further embodiments of the present invention can be found in the claims, the description and the examples. It is to be understood that the features mentioned above and those still to be illustrated below of the subject matter of the invention can be applied not only in the respective given combination but also in other combinations without leaving the scope of the invention.

EP-A 5 341, EP-A 266 725, EP-A 270 830, JP 04/297 454, JP 04/297 455 and JP

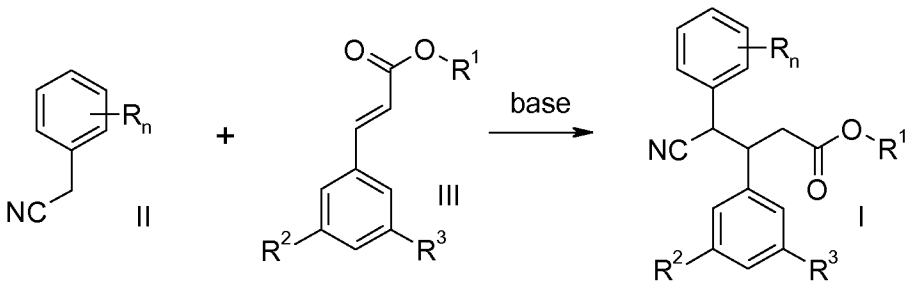
05/058 979 disclose herbicidal cyanobutyrates; however, their herbicidal action especially at low application rates and/or their compatibility with crop plants leave scope for improvement.

It is an object of the present invention to provide compounds having herbicidal action. To be provided are in particular active compounds having strong herbicidal action, in particular even at low application rates, whose compatibility with crop plants is sufficient for commercial application.

These and further objects are achieved by the compounds of the formula I defined at the outset and by their N-oxides and also their agriculturally suitable salts.

The compounds according to the invention can be prepared analogously to the synthesis routes described in the documents cited according to standard processes of organic chemistry, for example according to the following synthesis route:

Phenylacetonitrile derivatives of the formula II can be reacted with cinnamate derivatives of the formula III according to a Michael addition to give compounds of the formula I. In the formulae II and III, the variables have the meaning given for formula I.



This reaction is usually carried out at temperatures of from -100°C to 150°C, preferably from -78°C to 50°C, in a solvent in the presence of a base and/or a catalyst [cf. J. Chem. Soc. (1945), p. 438].

Suitable solvents are aliphatic hydrocarbons, such as pentane, hexane, cyclohexane and petroleum ether, aromatic hydrocarbons, such as toluene, o-, m- and p-xylene, halogenated hydrocarbons, such as methylene chloride, chloroform and chlorobenzene, ethers, such as diethyl ether, diisopropyl ether, tert-butyl methyl ether, dioxane, anisole and tetrahydrofuran (THF), nitriles, such as acetonitrile and propionitrile, ketones, such as acetone, methyl ethyl ketone, diethyl ketone and tert-butyl methyl ketone, alcohols, such as methanol, ethanol, n-propanol, isopropanol, n-butanol and tert-butanol, and also dimethyl sulfoxide, dimethylformamide and dimethylacetamide and water, particularly preferably THF and methanol. It is also possible to use mixtures of the solvents mentioned.

Suitable bases are, in general, inorganic compounds, such as alkali metal and alkaline earth metal hydroxides, such as lithium hydroxide, sodium hydroxide,

potassium hydroxide and calcium hydroxide, alkali metal and alkaline earth metal oxides, such as lithium oxide, sodium oxide, calcium oxide and magnesium oxide, alkali metal and alkaline earth metal hydrides, such as lithium hydride, sodium hydride, potassium hydride and calcium hydride, alkali metal amides, such as lithium amide, sodium amide and potassium amide, alkali metal and alkaline earth metal carbonates, such as lithium carbonate, potassium carbonate and calcium carbonate, and also alkali metal bicarbonates, such as sodium bicarbonate, organometallic compounds, in particular alkali metal alkyls, such as methyllithium, butyllithium and phenyllithium, alkylmagnesium halides, such as methylmagnesium chloride, and also alkali metal and alkaline earth metal alkoxides, such as sodium methoxide, sodium ethoxide, potassium ethoxide, potassium tert-butoxide and dimethoxymagnesium, moreover organic bases, for example tertiary amines, such as trimethylamine, triethylamine, tributylamine, diisopropylethylamine and N-methylpiperidine, pyridine, substituted pyridines, such as collidine, lutidine and 4-dimethylaminopyridine, and also bicyclic amines. Particular preference is given to potassium tert-butoxide, lithium bis(trimethylsilyl)amide and 7-methyl-1,5,7-triazabicyclo[4.4.0]dec-5-ene.

The bases are generally employed in catalytic amounts; however, they can also be used in equimolar amounts, in excess or, if appropriate, as solvents.

Suitable for use as acidic catalysts are inorganic acids, such as hydrofluoric acid, hydrochloric acid, hydrobromic acid, sulfuric acid and perchloric acid, Lewis acids, such as boron trifluoride, aluminum trichloride, iron(III) chloride, tin(IV) chloride, titanium(IV) chloride, scandium(III) triflate and zinc(II) chloride, and also organic acids, such as formic acid, acetic acid, propionic acid, oxalic acid, toluenesulfonic acid, benzenesulfonic acid, camphorsulfonic acid, citric acid and trifluoroacetic acid.

The acids are generally employed in catalytic amounts; however, they can also be used in equimolar amounts, in excess or, if appropriate, as solvent.

Alternatively, the compounds of the formula I can also be obtained by transesterification of other cyanobutyrates. This may take place by various methods, for example those described below:

The transesterification can be carried out in the presence of molecular sieves in an alcohol R^1-OH, if appropriate in an aprotic solvent. This reaction is usually carried out at temperatures of from 0°C to 180°C, preferably from 20°C to 80°C, in the presence of a Lewis or Brønsted acid or an enzyme [cf. J. Org. Chem. 2002, 67, 431].

Suitable solvents are aliphatic hydrocarbons, such as pentane, hexane, cyclohexane and petroleum ether, aromatic hydrocarbons, such as toluene, o-, m- and p-xylene, halogenated hydrocarbons, such as methylene chloride, chloroform and chlorobenzene, ethers, such as diethyl ether, diisopropyl ether, tert-butyl methyl ether, dioxane, anisole and THF, nitriles, such as acetonitrile and propionitrile, ketones, such as dimethyl sulfoxide, dimethylformamide and dimethylacetamide, particularly

preferably the alcohol R^1-OH. It is also possible to use mixtures of the solvents mentioned.

Alternatively, the transesterification may be carried out by acidic or basic hydrolysis of other cyanobutyrates (a), followed by reaction with an alcohol R^1-OH. These reactions are usually carried out at temperatures of from 0°C to 120°C, preferably from 20°C to 50°C, in the presence of a base or an acid and/or a catalyst [cf. J. Am. Chem. Soc. 2007, 129 (43), 13321; J. Org. Chem. 1984, 49 (22), 4287.].

Suitable solvents are aliphatic hydrocarbons, such as pentane, hexane, cyclohexane and petroleum ether, aromatic hydrocarbons, such as toluene, o-, m- and p-xylene, halogenated hydrocarbons, such as methylene chloride, chloroform and chlorobenzene, ethers, such as diethyl ether, diisopropyl ether, tert-butyl methyl ether, dioxane, anisole and THF, nitriles, such as acetonitrile and propionitrile, ketones, such as acetone, methyl ethyl ketone, diethyl ketone and tert-butyl methyl ketone, alcohols, such as methanol, ethanol, n-propanol, isopropanol, n-butanol and tert-butanol, and also dimethyl sulfoxide, dimethylformamide and dimethylacetamide and water, particularly preferably (a) water, THF; (b) R^1-OH. It is also possible to use mixtures of the solvents mentioned.

Suitable bases are, in general, inorganic compounds, such as alkali metal and alkaline earth metal hydroxides, such as lithium hydroxide, sodium hydroxide, potassium hydroxide and calcium hydroxide, alkali metal and alkaline earth metal oxides, such as lithium oxide, sodium oxide, calcium oxide and magnesium oxide, alkali metal and alkaline earth metal hydrides, such as lithium hydride, sodium hydride, potassium hydride and calcium hydride, alkali metal amides, such as lithium amide, sodium amide and potassium amide, alkali metal and alkaline earth metal carbonates, such as lithium carbonate, potassium carbonate and calcium carbonate, and also alkali metal bicarbonates, such as sodium bicarbonate, organometallic compounds, in particular alkali metal alkyls, such as methyllithium, butyllithium and phenyllithium, alkylmagnesium halides, such as methylmagnesium chloride, and also alkali metal and alkaline earth metal alkoxides, such as sodium methoxide, sodium ethoxide, potassium ethoxide, potassium tert-butoxide and dimethoxymagnesium, moreover organic bases, for example tertiary amines, such as trimethylamine, triethylamine, tributylamine, diisopropylethylamine and N-methylpiperidine, pyridine, substituted pyridines, such as collidine, lutidine and 4-dimethylaminopyridine, and also bicyclic amines. Particular preference is given to lithium hydroxide.

The bases are generally employed in catalytic amounts; however, they can also be used in equimolar amounts, in excess or, if appropriate, as solvents.

Suitable for use as acids and acidic catalysts are inorganic acids, such as hydrofluoric acid, hydrochloric acid, hydrobromic acid, sulfuric acid and perchloric acid, Lewis acids, such as boron trifluoride, aluminum trichloride, iron(III) chloride, tin(IV) chloride, titanium(IV) chloride and zinc(II) chloride, and also organic acids, such as

formic acid, acetic acid, propionic acid, oxalic acid, toluenesulfonic acid, benzenesulfonic acid, camphorsulfonic acid, citric acid and trifluoroacetic acid.

The acids are generally employed in catalytic amounts; however, they can also be used in equimolar amounts, in excess or, if appropriate, as solvent.

The starting materials required for preparing the compounds I are known from the literature or can be prepared in accordance with the literature cited.

The reaction mixtures are worked up in a customary manner, for example by mixing with water, separating the phases and, if appropriate, chromatographic purification of the crude products. Some of the intermediates and end products are obtained in the form of colorless or slightly brownish viscous oils which are purified or freed from volatile components under reduced pressure and at moderately elevated temperature. If the intermediates and end products are obtained as solids, the purification can also be carried out by recrystallization or digestion.

If individual compounds I cannot be obtained by the routes described above, they can be prepared by derivatization of other compounds I.

If the synthesis yields mixtures of isomers, a separation is generally however not necessarily required since in some cases the individual isomers can be interconverted during work-up for use or during application (for example under the action of light, acids or bases). Such conversions may also take place after application, for example in the case of the treatment of plants in the treated plant or in the harmful plant to be controlled.

The organic moieties mentioned for the substituents of the compounds according to the invention are collective terms for individual enumerations of the individual group members. All hydrocarbon chains, such as alkyl, haloalkyl, alkenyl, alkynyl, and the alkyl moieties and alkenyl moieties in alkoxy, haloalkoxy, alkylamino, dialkylamino, N-alkylsulfonylamino, alkenyloxy, alkynyloxy, alkoxyamino, alkylaminosulfonylamino, dialkylaminosulfonylamino, alkenylamino, alkynylamino, N-(alkenyl)-N-(alkyl)amino, N-(alkynyl)-N-(alkyl)amino, N-(alkoxy)-N-(alkyl)amino, N-(alkenyl)-N-(alkoxy)amino or N-(alkynyl)-N-(alkoxy)amino can be straight-chain or branched.

The prefix C_n-C_m- indicates the respective number of carbons of the hydrocarbon unit. Unless indicated otherwise, halogenated substituents preferably carry one to five identical or different halogen atoms, in particular fluorine atoms or chlorine atoms.

The meaning halogen denotes in each case fluorine, chlorine, bromine or iodine.

Examples of other meanings are:

alkyl and the alkyl moieties for example in alkoxy, alkylamino, dialkylamino, N-alkylsulfonylamino, alkylaminosulfonylamino, dialkylaminosulfonylamino, N-(alkenyl)-N-(alkyl)amino, N-(alkynyl)-N-(alkyl)amino, N-(alkoxy)-N-(alkyl)amino: saturated straight-chain or branched hydrocarbon radicals having one or more carbon atoms, for example 1 or 2, 1 to 4 or 1 to 6 carbon atoms, for example C_1-C_6-alkyl, such as methyl, ethyl,

propyl, 1-methylethyl, butyl, 1-methylpropyl, 2-methylpropyl, 1,1-dimethylethyl, pentyl, 1-methylbutyl, 2-methylbutyl, 3-methylbutyl, 2,2-dimethylpropyl, 1-ethylpropyl, hexyl, 1,1-dimethylpropyl, 1,2-dimethylpropyl, 1-methylpentyl, 2-methylpentyl, 3-methylpentyl, 4-methylpentyl, 1,1-dimethylbutyl, 1,2-dimethylbutyl, 1,3-dimethylbutyl, 2,2-dimethylbutyl, 2,3-dimethylbutyl, 3,3-dimethylbutyl, 1-ethylbutyl, 2-ethylbutyl, 1,1,2-trimethylpropyl, 1,2,2-trimethylpropyl, 1-ethyl-1-methylpropyl, 1-ethyl-2-methylpropyl. In one embodiment according to the invention, alkyl denotes small alkyl groups, such as C_1-C_4-alkyl. In another embodiment according to the invention, alkyl denotes relatively large alkyl groups, such as C_5-C_6-alkyl.

Haloalkyl: an alkyl radical as mentioned above, some or all of whose hydrogen atoms are substituted by halogen atoms, such as fluorine, chlorine, bromine and/or iodine, for example chloromethyl, dichloromethyl, trichloromethyl, fluoromethyl, difluoromethyl, trifluoromethyl, chlorofluoromethyl, dichlorofluoromethyl, chlorodifluoromethyl, 2-fluoroethyl, 2-chloroethyl, 2-bromoethyl, 2-iodoethyl, 2,2-difluoroethyl, 2,2,2-trifluoroethyl, 2-chloro-2-fluoroethyl, 2-chloro-2,2-difluoroethyl, 2,2-dichloro-2-fluoroethyl, 2,2,2-trichloroethyl, pentafluoroethyl, 2-fluoropropyl, 3-fluoropropyl, 2,2-difluoropropyl, 2,3-difluoropropyl, 2-chloropropyl, 3-chloropropyl, 2,3-dichloropropyl, 2-bromopropyl, 3-bromopropyl, 3,3,3-trifluoropropyl, 3,3,3-trichloropropyl, 2,2,3,3,3-pentafluoropropyl, heptafluoropropyl, 1-(fluoromethyl)-2-fluoroethyl, 1-(chloromethyl)-2-chloroethyl, 1-(bromomethyl)-2-bromoethyl, 4-fluorobutyl, 4-chlorobutyl, 4-bromobutyl and nonafluorobutyl.

Cycloalkyl and the cycloalkyl moieties for example in cycloalkoxy or cycloalkylcarbonyl: monocyclic saturated hydrocarbon groups having three or more carbon atoms, for example 3 to 6 carbon ring members, such as cyclopropyl, cyclobutyl, cyclopentyl and cyclohexyl.

Alkenyl and the alkenyl moieties for example in alkenylamino, alkenyloxy, N-(alkenyl)-N-(alkyl)amino, N-(alkenyl)-N-(alkoxy)amino: monounsaturated straight-chain or branched hydrocarbon radicals having two or more carbon atoms, for example 2 to 4, 2 to 6 or 3 to 6 carbon atoms, and a double bond in any position, for example C_2-C_6-alkenyl, such as ethenyl, 1-propenyl, 2-propenyl, 1-methylethenyl, 1-butenyl, 2-butenyl, 3-butenyl, 1-methyl-1-propenyl, 2-methyl-1-propenyl, 1-methyl-2-propenyl, 2-methyl-2-propenyl, 1-pentenyl, 2-pentenyl, 3-pentenyl, 4-pentenyl, 1-methyl-1-butenyl, 2-methyl-1-butenyl, 3-methyl-1-butenyl, 1-methyl-2-butenyl, 2-methyl-2-butenyl, 3-methyl-2-butenyl, 1-methyl-3-butenyl, 2-methyl-3-butenyl, 3-methyl-3-butenyl, 1,1-dimethyl-2-propenyl, 1,2-dimethyl-1-propenyl, 1,2-dimethyl-2-propenyl, 1-ethyl-1-propenyl, 1-ethyl-2-propenyl, 1-hexenyl, 2-hexenyl, 3-hexenyl, 4-hexenyl, 5-hexenyl, 1-methyl-1-pentenyl, 2-methyl-1-pentenyl, 3-methyl-1-pentenyl, 4-methyl-1-pentenyl, 1-methyl-2-pentenyl, 2-methyl-2-pentenyl, 3-methyl-2-pentenyl, 4-methyl-2-pentenyl, 1-methyl-3-pentenyl, 2-methyl-3-pentenyl, 3-methyl-3-pentenyl, 4-methyl-3-pentenyl, 1-methyl-4-pentenyl, 2-methyl-4-pentenyl, 3-methyl-4-pentenyl, 4-methyl-4-pentenyl, 1,1-dimethyl-2-butenyl, 1,1-dimethyl-3-butenyl, 1,2-dimethyl-1-butenyl, 1,2-

dimethyl-2-butenyl, 1,2-dimethyl-3-butenyl, 1,3-dimethyl-1-butenyl, 1,3-dimethyl-2-butenyl, 1,3-dimethyl-3-butenyl, 2,2-dimethyl-3-butenyl, 2,3-dimethyl-1-butenyl, 2,3-dimethyl-2-butenyl, 2,3-dimethyl-3-butenyl, 3,3-dimethyl-1-butenyl, 3,3-dimethyl-2-butenyl, 1-ethyl-1-butenyl, 1-ethyl-2-butenyl, 1-ethyl-3-butenyl, 2-ethyl-1-butenyl, 2-ethyl-2-butenyl, 2-ethyl-3-butenyl, 1,1,2-trimethyl-2-propenyl, 1-ethyl-1-methyl-2-propenyl, 1-ethyl-2-methyl-1-propenyl, 1-ethyl-2-methyl-2-propenyl.

Cycloalkenyl: monocyclic monounsaturated hydrocarbon groups having 3 to 6, preferably 5 or 6, carbon ring members, such as cyclopenten-1-yl, cyclopenten-3-yl, cyclohexen-1-yl, cyclohexen-3-yl, cyclohexen-4-yl.

Alkynyl and the alkynyl moieties for example in alkynyloxy, alkynylamino, N-(alkynyl)-N-(alkyl)amino or N-(alkynyl)-N-(alkoxy)amino: straight-chain or branched hydrocarbon groups having two or more carbon atoms, for example 2 to 4, 2 to 6 or 3 to 6 carbon atoms, and a triple bond in any position, for example C_2-C_6-alkynyl, such as ethynyl, 1-propynyl, 2-propynyl, 1-butynyl, 2-butynyl, 3-butynyl, 1-methyl-2-propynyl, 1-pentynyl, 2-pentynyl, 3-pentynyl, 4-pentynyl, 1-methyl-2-butynyl, 1-methyl-3-butynyl, 2-methyl-3-butynyl, 3-methyl-1-butynyl, 1,1-dimethyl-2-propynyl, 1-ethyl-2-propynyl, 1-hexynyl, 2-hexynyl, 3-hexynyl, 4-hexynyl, 5-hexynyl, 1-methyl-2-pentynyl, 1-methyl-3-pentynyl, 1-methyl-4-pentynyl, 2-methyl-3-pentynyl, 2-methyl-4-pentynyl, 3-methyl-1-pentynyl, 3-methyl-4-pentynyl, 4-methyl-1-pentynyl, 4-methyl-2-pentynyl, 1,1-dimethyl-2-butynyl, 1,1-dimethyl-3-butynyl, 1,2-dimethyl-3-butynyl, 2,2-dimethyl-3-butynyl, 3,3-dimethyl-1-butynyl, 1-ethyl-2-butynyl, 1-ethyl-3-butynyl, 2-ethyl-3-butynyl, 1-ethyl-1-methyl-2-propynyl.

Alkoxy: alkyl as defined above which is attached via an oxygen atom, for example methoxy, ethoxy, n-propoxy, 1-methylethoxy, butoxy, 1-methylpropoxy, 2-methylpropoxy or 1,1-dimethylethoxy, pentoxy, 1-methylbutoxy, 2-methylbutoxy, 3-methylbutoxy, 1,1-dimethylpropoxy, 1,2-dimethylpropoxy, 2,2-dimethylpropoxy, 1-ethylpropoxy, hexoxy, 1-methylpentoxy, 2-methylpentoxy, 3-methylpentoxy, 4-methylpentoxy, 1,1-dimethylbutoxy, 1,2-dimethylbutoxy, 1,3-dimethylbutoxy, 2,2-dimethylbutoxy, 2,3-dimethylbutoxy, 3,3-dimethylbutoxy, 1-ethylbutoxy, 2-ethylbutoxy, 1,1,2-trimethylpropoxy, 1,2,2-trimethylpropoxy, 1-ethyl-1-methylpropoxy or 1-ethyl-2-methylpropoxy.

Alkylene: divalent unbranched chains of 3 to 5 CH_2 groups, for example CH_2, CH_2CH_2, $CH_2CH_2CH_2$, $CH_2CH_2CH_2CH_2$ and $CH_2CH_2CH_2CH_2CH_2$.

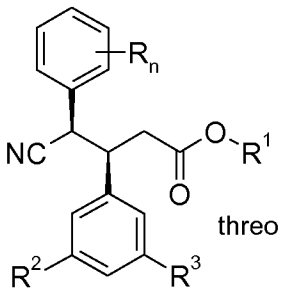
Oxyalkylene: divalent unbranched chains of 2 to 4 CH_2 groups, where one valency is attached to the skeleton via an oxygen atom, for example OCH_2CH_2, $OCH_2CH_2CH_2$ and $OCH_2CH_2CH_2CH_2$.

Oxyalkyleneoxy: divalent unbranched chains of 1 to 3 CH_2 groups, where both valencies are attached to the skeleton via an oxygen atom, for example OCH_2O, OCH_2CH_2O and $OCH_2CH_2CH_2O$;

A 5- or 6-membered heterocycle: a cyclic group which has 5 or 6 ring atoms, 1, 2, 3 or 4 ring atoms being heteroatoms selected from the group consisting of O, S and N,

where the cyclic group is saturated, partially unsaturated or aromatic.

The compounds of the formula I contain two centers of chirality and may, depending on the substitution pattern, contain one or more further. Accordingly, the compounds according to the invention can be present as pure enantiomers or diastereomers or as enantiomer or diastereomer mixtures. The invention provides both the pure enantiomers or diastereomers and their mixtures.



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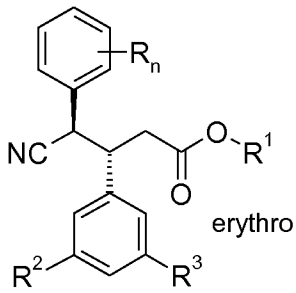

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The compounds of the formula I may also be present in the form of the N-oxides and/or of their agriculturally useful salts, the type of salt generally not being important. Suitable salts are generally the salts of those cations or the acid addition salts of those acids whose cations and anions, respectively, have no adverse effect on the herbicidal activity of the compounds I.

Suitable cations are in particular ions of the alkali metals, preferably lithium, sodium or potassium, of the alkaline earth metals, preferably calcium or magnesium, and of the transition metals, preferably manganese, copper, zinc or iron. Another cation that may be used is ammonium, where, if desired, one to four hydrogen atoms may be replaced by C_1-C_4-alkyl, hydroxy-C_1-C_4-alkyl, C_1-C_4-alkoxy-C_1-C_4-alkyl, hydroxy-C_1-C_4-alkoxy-C_1-C_4-alkyl, phenyl or benzyl, preferably ammonium, dimethylammonium, diisopropylammonium, tetramethylammonium, tetrabutylammonium, 2-(2-hydroxyeth-1-oxy)eth-1-ylammonium, di(2-hydroxyeth-1-yl)ammonium, trimethylbenzylammonium. Another suitable ammonium cation is the pyridine nitrogen atom of the formula I quaternized by alkylation or arylation. Also suitable are phosphonium ions, sulfonium ions, preferably tri(C_1-C_4-alkyl)sulfonium, or sulfoxonium ions, preferably tri(C_1-C_4-alkyl)sulfoxonium.

Anions of suitable acid addition salts are primarily chloride, bromide, fluoride, hydrogensulfate, sulfate, dihydrogenphosphate, hydrogenphosphate, nitrate, bicarbonate, carbonate, hexafluorosilicate, hexafluorophosphate, benzoate and also the anions of C_1-C_4-alkanoic acids, preferably formate, acetate, propionate, butyrate or trifluoroacetate.

With respect to the variables, the particularly preferred embodiments of the intermediates correspond to those of the groups of the formula I.

In a particular embodiment, the variables of the compounds of the formula I have the

following meanings, these meanings, both on their own and in combination with one another, being particular embodiments of the compounds of the formula I:

In one embodiment of the compounds of the formula I, the index n has a value from 0 to 3, preferably 0 or 1, in particular 1. In a further embodiment, n is 0. If at least one group R is present, it is preferably located in positions 3, 4 and/or 5, in particular 3 or 4. If the index n has the value 2, the groups R are preferably located in positions 3,5 or 3,4.

Embodiments of the group(s) R relate to halogen, in particular chlorine or fluorine. Further embodiments of the group(s) R relate to cyano or nitro, in particular cyano.

Particularly preferred embodiments of group R_n include the following: 3-F; 3,4-F_2; 3,5-F_2; 3,4,5-F_3; 3-F,4-Cl; 3-Cl,4-F; 4-Cl; 4-F; 3-CN; 3-Cl.

Preferred embodiments of group R^1 relate to hydrogen, C_1-C_8-alkyl, cyano-C_1-C_4-alkyl, C_3-C_6-alkenyl, C_3-C_6-alkynyl, C_1-C_8-haloalkyl, optionally substituted Z-phenyl or NR^iR^{ii}-C_1-C_4-alkyl, where R^i is hydrogen or C_1-C_4-alkyl and R^{ii} is hydrogen, C_1-C_4-alkyl or C(=O)-R^A. Particularly preferred embodiments of group R^1 relate to hydrogen, C_1-C_4-alkyl, cyanomethyl, allyl, propargyl, C_1-C_4-haloalkyl comprising preferably 1 to 3 halogen atoms, phenyl or benzyl, which aromatic groups may be partially or fully substituted. Particularly preferred embodiments of R^1 include the following: H, CH_3, C_2H_5, CH_2CN, $CH_2C\equiv CH$, CH_2CH_2F, CH_2CHF_2, CH_2CF_3, CH_2OCH_3. In another preferred embodiment, R^1 is selected from the group consisting of H, CH_3 and $CH_2C\equiv CH$. R^1 is in particular selected from the group consisting of H and CH_3.

Preferred embodiments of group R^2 relate to halogen, cyano, nitro, C_1-C_4-alkyl, C_1-C_4-alkoxy, $S(O)_mR^{bb}$, NR^iR^{ii}, C_1-C_4-haloalkyl, C_1-C_4-haloalkoxy. Particularly preferred embodiments of group R^2 include halogen, CN, NO_2, CH_3, CHF_2, CF_3, $OCHF_2$ and OCF_3. Especially preferred embodiments of group R^2 are selected from the group consisting of fluorine, chlorine, bromine and iodine.

More particularly preferably, R^2 is chlorine.

More particularly preferably, R^2 is fluorine.

More particularly preferably, R^2 is bromine.

Preferred embodiments of group R^3 relate to halogen, cyano, nitro, C_1-C_4-alkyl, C_1-C_4-alkoxy, $S(O)_mR^{bb}$, NR^iR^{ii}, C_1-C_4-haloalkyl, C_1-C_4-haloalkoxy. Particularly preferred embodiments of group R^3 relate to halogen, CN, NO_2, CH_3, CHF_2, CF_3, $OCHF_2$, OCF_3. Especially preferred embodiments of group R^3 are selected from the group consisting of fluorine, CN, NO_2, CHF_2, CF_3, $OCHF_2$ and OCF_3.

More particularly preferably, R^3 is fluorine.

More particularly preferably, R^3 is chlorine.

More particularly preferably, R^3 is bromine.

More particularly preferably, R^3 is CF_3.
More particularly preferably, R^3 is OCF_3.
More particularly preferably, R^3 is $OCHF_2$.

In a preferred embodiment of the compounds according to the invention, R^2 and R^3 are different, in particular if at least one group R^2 and/or R^3, preferably both, are halogen, such as Cl and/or F.

Especially preferably, the combination of R^2 and R^3 is selected from the group consisting of 3-F,5-CH_3, 3-F,5-$OCHF_2$, 3-F,5-Cl, 3-F,5-Br, 3,5-F_2 and 3,5-Cl_2. In another preferred embodiment, the combination of R^2 and R^3 is selected from the group consisting of 3-F,5-CH_3, 3-F,5-$OCHF_2$, 3-F,5-Cl and 3-F,5-Br.

In particular with a view to their use, preference is given to the compounds of the formula I compiled in the tables below. The groups mentioned for a substituent in the tables are furthermore per se, independently of the combination in which they are mentioned, a particularly preferred aspect of the substituent in question.

Table 1
Compounds of the formula I in which R_n is 3-CN and the combination of R^1, R^2 and R^3 for a compound corresponds in each case to one row of table A

Table 2
Compounds of the formula I in which R_n is 4-Cl and the combination of R^1, R^2 and R^3 for a compound corresponds in each case to one row of table A, excepting A-10 and A-19

Table 3
Compounds of the formula I in which R_n is 3-F and the combination of R^1, R^2 and R^3 for a compound corresponds in each case to one row of table A, excepting A-10 and A-19

Table 4
Compounds of the formula I in which R_n is 4-F and the combination of R^1, R^2 and R^3 for a compound corresponds in each case to one row of table A, excepting A-10 and A-19

Table 5
Compounds of the formula I in which R_n is 3,4-F_2 and the combination of R^1, R^2 and R^3 for a compound corresponds in each case to one row of table A

Table 6
Compounds of the formula I in which R_n is 3,5-F_2 and the combination of R^1, R^2 and R^3 for a compound corresponds in each case to one row of table A

Table 7

Compounds of the formula I in which R_n is 3-F,4-Cl and the combination of R^1, R^2 and R^3 for a compound corresponds in each case to one row of table A

Table 8

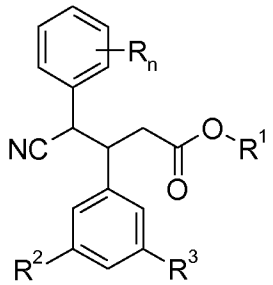
Compounds of the formula I in which R_n is 3,4,5-F_3 and the combination of R^1, R^2 and R^3 for a compound corresponds in each case to one row of table A

Table 9

Compounds of the formula I in which R_n is 3-Cl,4-F and the combination of R^1, R^2 and R^3 for a compound corresponds in each case to one row of table A

Table A

Compounds of the formula I



I

No.	R^1	R^2	R^3
A-1	H	F	F
A-2	H	F	Cl
A-3	H	F	Br
A-4	H	F	CN
A-5	H	F	NO_2
A-6	H	F	CF_3
A-7	H	F	OCH_3
A-8	H	F	OCF_3
A-9	H	F	$OCHF_2$
A-10	CH_3	F	F
A-11	CH_3	F	Cl
A-12	CH_3	F	Br
A-13	CH_3	F	CN
A-14	CH_3	F	NO_2
A-15	CH_3	F	CF_3

No.	R^1	R^2	R^3
A-16	CH_3	F	OCH_3
A-17	CH_3	F	OCF_3
A-18	CH_3	F	$OCHF_2$
A-19	C_2H_5	F	F
A-20	C_2H_5	F	Cl
A-21	C_2H_5	F	Br
A-22	C_2H_5	F	CN
A-23	C_2H_5	F	NO_2
A-24	C_2H_5	F	CF_3
A-25	C_2H_5	F	OCH_3
A-26	C_2H_5	F	OCF_3
A-27	C_2H_5	F	$OCHF_2$
A-28	CH_2CN	F	F
A-29	CH_2CN	F	Cl
A-30	CH_2CN	F	Br
A-31	CH_2CN	F	CN
A-32	CH_2CN	F	NO_2
A-33	CH_2CN	F	CF_3
A-34	CH_2CN	F	OCH_3
A-35	CH_2CN	F	OCF_3
A-36	CH_2CN	F	$OCHF_2$
A-37	$CH_2C\equiv CH$	F	F
A-38	$CH_2C\equiv CH$	F	Cl
A-39	$CH_2C\equiv CH$	F	Br
A-40	$CH_2C\equiv CH$	F	CN
A-41	$CH_2C\equiv CH$	F	NO_2
A-42	$CH_2C\equiv CH$	F	CF_3
A-43	$CH_2C\equiv CH$	F	OCH_3
A-44	$CH_2C\equiv CH$	F	OCF_3
A-45	$CH_2C\equiv CH$	F	$OCHF_2$
A-46	CH_2CH_2F	F	F
A-47	CH_2CH_2F	F	Cl
A-48	CH_2CH_2F	F	Br
A-49	CH_2CH_2F	F	CN
A-50	CH_2CH_2F	F	NO_2
A-51	CH_2CH_2F	F	CF_3
A-52	CH_2CH_2F	F	OCH_3
A-53	CH_2CH_2F	F	OCF_3

No.	R^1	R^2	R^3
A-54	CH_2CH_2F	F	$OCHF_2$
A-55	CH_2CHF_2	F	F
A-56	CH_2CHF_2	F	Cl
A-57	CH_2CHF_2	F	Br
A-58	CH_2CHF_2	F	CN
A-59	CH_2CHF_2	F	NO_2
A-60	CH_2CHF_2	F	CF_3
A-61	CH_2CHF_2	F	OCH_3
A-62	CH_2CHF_2	F	OCF_3
A-63	CH_2CHF_2	F	$OCHF_2$
A-64	CH_2CF_3	F	F
A-65	CH_2CF_3	F	Cl
A-66	CH_2CF_3	F	Br
A-67	CH_2CF_3	F	CN
A-68	CH_2CF_3	F	NO_2
A-69	CH_2CF_3	F	CF_3
A-70	CH_2CF_3	F	OCH_3
A-71	CH_2CF_3	F	OCF_3
A-72	CH_2CF_3	F	$OCHF_2$
A-73	CH_2OCH_3	F	F
A-74	CH_2OCH_3	F	Cl
A-75	CH_2OCH_3	F	Br
A-76	CH_2OCH_3	F	CN
A-77	CH_2OCH_3	F	NO_2
A-78	CH_2OCH_3	F	CF_3
A-79	CH_2OCH_3	F	OCH_3
A-80	CH_2OCH_3	F	OCF_3
A-81	CH_2OCH_3	F	$OCHF_2$

The compounds I and their agriculturally useful salts are suitable, both as isomer mixtures and in the form of the pure isomers, as herbicides. They are suitable as such or as an appropriately formulated composition. The herbicidal compositions comprising the compound I, in particular the preferred aspects thereof, control vegetation on non-crop areas very efficiently, especially at high rates of application. They act against broad-leaved weeds and weed grasses in crops such as wheat, rice, corn, soybeans and cotton without causing any significant damage to the crop plants. This effect is mainly observed at low rates of application.

Depending on the application method in question, the compounds I, in particular the

preferred aspects thereof, or compositions comprising them can additionally be employed in a further number of crop plants for eliminating unwanted plants. Examples of suitable crops are the following:

Allium cepa, Ananas comosus, Arachis hypogaea, Asparagus officinalis, Avena sativa, Beta vulgaris spec. altissima, Beta vulgaris spec. rapa, Brassica napus var. napus, Brassica napus var. napobrassica, Brassica rapa var. silvestris, Brassica oleracea, Brassica nigra, Camellia sinensis, Carthamus tinctorius, Carya illinoinensis, Citrus limon, Citrus sinensis, Coffea arabica (Coffea canephora, Coffea liberica), Cucumis sativus, Cynodon dactylon, Daucus carota, Elaeis guineensis, Fragaria vesca, Glycine max, Gossypium hirsutum, (Gossypium arboreum, Gossypium herbaceum, Gossypium vitifolium), Helianthus annuus, Hevea brasiliensis, Hordeum vulgare, Humulus lupulus, Ipomoea batatas, Juglans regia, Lens culinaris, Linum usitatissimum, Lycopersicon lycopersicum, Malus spec., Manihot esculenta, Medicago sativa, Musa spec., Nicotiana tabacum (N.rustica), Olea europaea, Oryza sativa, Phaseolus lunatus, Phaseolus vulgaris, Picea abies, Pinus spec., Pistacia vera, Pisum sativum, Prunus avium, Prunus persica, Pyrus communis, Prunus armeniaca, Prunus cerasus, Prunus dulcis and Prunus domestica, Ribes sylvestre, Ricinus communis, Saccharum officinarum, Secale cereale, Sinapis alba, Solanum tuberosum, Sorghum bicolor (s. vulgare), Theobroma cacao, Trifolium pratense, Triticum aestivum, Triticale, Triticum durum, Vicia faba, Vitis vinifera, Zea mays.

The term "crop plants" also includes plants which have been modified by breeding, mutagenesis or genetic engineering. Genetically modified plants are plants whose genetic material has been modified in a manner which does not occur under natural conditions by crossing, mutations or natural recombination (i.e. reassembly of the genetic information). Here, in general, one or more genes are integrated into the genetic material of the plant to improve the properties of the plant.

Accordingly, the term "crop plants" also includes plants which, by breeding and genetic engineering, have acquired tolerance to certain classes of herbicides, such as hydroxyphenylpyruvate dioxygenase (HPPD) inhibitors, acetolactate synthase (ALS) inhibitors, such as, for example, sulfonylureas (EP-A 257 993, US 5,013,659) or imidazolinones (see, for example, US 6,222,100, WO 01/82685, WO 00/26390, WO 97/41218, WO 98/02526, WO 98/02527, WO 04/106529, WO 05/20673, WO 03/14357, WO 03/13225, WO 03/14356, WO 04/16073), enolpyruvylshikimate 3-phosphate synthase (EPSPS) inhibitors, such as, for example, glyphosate (see, for example, WO 92/00377), glutamine synthetase (GS) inhibitors, such as, for example, glufosinate (see, for example, EP-A 242 236, EP-A 242 246), or oxynil herbicides (see, for example, US 5,559,024).

Numerous crop plants, for example Clearfield® oilseed rape, tolerant to

imidazolinones, for example imazamox, have been generated with the aid of classic breeding methods (mutagenesis). Crop plants such as soybeans, cotton, corn, beet and oilseed rape, resistant to glyphosate or glufosinate, which are available under the tradenames RoundupReady® (glyphosate) and Liberty Link® (glufosinate) have been generated with the aid of genetic engineering methods.

Accordingly, the term "crop plants" also includes plants which, with the aid of genetic engineering, produce one or more toxins, for example those of the bacterial strain *Bacillus* ssp. Toxins which are produced by such genetically modified plants include, for example, insecticidal proteins of *Bacillus* spp., in particular *B. thuringiensis*, such as the endotoxins Cry1Ab, Cry1Ac, Cry1F, Cry1Fa2, Cry2Ab, Cry3A, Cry3Bb1, Cry9c, Cry34Ab1 or Cry35Ab1; or vegetative insecticidal proteins (VIPs), for example VIP1, VIP2, VIP3, or VIP3A; insecticidal proteins of nematode-colonizing bacteria, for example *Photorhabdus* spp. or *Xenorhabdus* spp.; toxins of animal organisms, for example wasp, spider or scorpion toxins; fungal toxins, for example from Streptomycetes; plant lectins, for example from peas or barley; agglutinins; proteinase inhibitors, for example trypsin inhibitors, serine protease inhibitors, patatin, cystatin or papain inhibitors, ribosome-inactivating proteins (RIPs), for example ricin, corn-RIP, abrin, luffin, saporin or bryodin; steroid-metabolizing enzymes, for example 3-hydroxysteroid oxidase, ecdysteroid-IDP glycosyl transferase, cholesterol oxidase, ecdysone inhibitors, or HMG-CoA reductase; ion channel blockers, for example inhibitors of sodium channels or calcium channels; juvenile hormone esterase; receptors of the diuretic hormone (helicokinin receptors); stilbene synthase, bibenzyl synthase, chitinases and glucanases. In the plants, these toxins may also be produced as pretoxins, hybrid proteins or truncated or otherwise modified proteins. Hybrid proteins are characterized by a novel combination of different protein domains (see, for example, WO 2002/015701). Further examples of such toxins or genetically modified plants which produce these toxins are disclosed in EP-A 374 753, WO 93/007278, WO 95/34656, EP-A 427 529, EP-A 451 878, WO 03/018810 and WO 03/052073. The methods for producing these genetically modified plants are known to the person skilled in the art and disclosed, for example, in the publications mentioned above. Numerous of the toxins mentioned above bestow, upon the plants by which they are produced, tolerance to pests from all taxonomic classes of arthropods, in particular to beetles (Coeleropta), dipterans (Diptera) and butterflies (Lepidoptera) and to nematodes (Nematoda).

Genetically modified plants which produce one or more genes coding for insecticidal toxins are described, for example, in the publications mentioned above, and some of them are commercially available, such as, for example, YieldGard® (corn varieties producing the toxin Cry1Ab), YieldGard® Plus (corn varieties which produce the toxins Cry1Ab and Cry3Bb1), Starlink® (corn varieties which produce the toxin Cry9c),

Herculex® RW (corn varieties which produce the toxins Cry34Ab1, Cry35Ab1 and the enzyme phosphinothricin-N-acetyltransferase [PAT]); NuCOTN® 33B (cotton varieties which produce the toxin Cry1Ac), Bollgard® I (cotton varieties which produce the toxin Cry1Ac), Bollgard® II (cotton varieties which produce the toxins Cry1Ac and Cry2Ab2); VIPCOT® (cotton varieties which produce a VIP toxin); NewLeaf® (potato varieties which produce the toxin Cry3A); Bt-Xtra®, NatureGard®, KnockOut®, BiteGard®, Protecta®, Bt11 (for example Agrisure® CB) and Bt176 from Syngenta Seeds SAS, France (corn varieties which produce the toxin Cry1Ab and the PAT enyzme), MIR604 from Syngenta Seeds SAS, France (corn varieties which produce a modified version of the toxin Cry3A, see WO 03/018810), MON 863 from Monsanto Europe S.A., Belgium (corn varieties which produce the toxin Cry3Bb1), IPC 531 from Monsanto Europe S.A., Belgium (cotton varieties which produce a modified version of the toxin Cry1Ac) and 1507 from Pioneer Overseas Corporation, Belgium (corn varieties which produce the toxin Cry1F and the PAT enzyme).

Accordingly, the term "crop plants" also includes plants which, with the aid of genetic engineering, produce one or more proteins which are more robust or have increased resistance to bacterial, viral or fungal pathogens, such as, for example, pathogenesis-related proteins (PR proteins, see EP 392 225), resistance proteins (for example potato varieties producing two resistance genes against *Phytophthora infestans* from the wild Mexican potato *Solanum bulbocastanum*) or T4 lysozyme (for example potato cultivars which, by producing this protein, are resistant to bacteria such as *Erwinia amylvora*).

Accordingly, the term "crop plants" also includes plants whose productivity has been improved with the aid of genetic engineering methods, for example by enhancing the potential yield (for example biomass, grain yield, starch, oil or protein content), tolerance to drought, salt or other limiting environmental factors or resistance to pests and fungal, bacterial and viral pathogens.

The term "crop plants" also includes plants whose ingredients have been modified with the aid of genetic engineering methods in particular for improving human or animal diet, for example by oil plants producing health-promoting long-chain omega 3 fatty acids or monounsaturated omega 9 fatty acids (for example Nexera® oilseed rape).

The term "crop plants" also includes plants which have been modified with the aid of genetic engineering methods for improving the production of raw materials, for example by increasing the amylopectin content of potatoes (Amflora® potato).

Furthermore, it has been found that the compounds of the formula I are also suitable for the defoliation and/or desiccation of plant parts, for which crop plants such as cotton, potato, oilseed rape, sunflower, soybean or field beans, in particular cotton, are suitable. In this regard, there have been found compositions for the desiccation and/or defoliation of plants, processes for preparing these compositions and methods for desiccating and/or defoliating plants using the compounds of the formula I.

As desiccants, the compounds of the formula I are particularly suitable for

desiccating the above-ground parts of crop plants such as potato, oilseed rape, sunflower and soybean, but also cereals. This makes possible the fully mechanical harvesting of these important crop plants.

Also of economic interest is to facilitate harvesting, which is made possible by concentrating within a certain period of time the dehiscence, or reduction of adhesion to the tree, in citrus fruit, olives and other species and varieties of pomaceous fruit, stone fruit and nuts. The same mechanism, i.e. the promotion of the development of abscission tissue between fruit part or leaf part and shoot part of the plants is also essential for the readily controllable defoliation of useful plants, in particular cotton.

Moreover, a shortening of the time interval in which the individual cotton plants mature leads to an increased fiber quality after harvesting.

The compounds I, or the herbicidal compositions comprising the compounds I, can be used, for example, in the form of ready-to-spray aqueous solutions, powders, suspensions, also highly concentrated aqueous, oily or other suspensions or dispersions, emulsions, oil dispersions, pastes, dusts, materials for broadcasting, or granules, by means of spraying, atomizing, dusting, spreading, watering or treatment of the seed or mixing with the seed. The use forms depend on the intended purpose; in each case, they should ensure the finest possible distribution of the active ingredients according to the invention.

The herbicidal compositions comprise a herbicidally effective amount of at least one compound of the formula I or an agriculturally useful salt of I, and auxiliaries which are customary for the formulation of crop protection agents.

Examples of auxiliaries customary for the formulation of crop protection agents are inert auxiliaries, solid carriers, surfactants (such as dispersants, protective colloids, emulsifiers, wetting agents and tackifiers), organic and inorganic thickeners, bactericides, antifreeze agents, antifoams, optionally colorants and, for seed formulations, adhesives.

Examples of thickeners (i.e. compounds which impart to the formulation modified flow properties, i.e. high viscosity in the state of rest and low viscosity in motion) are polysaccharides, such as xanthan gum (Kelzan® from Kelco), Rhodopol® 23 (Rhone Poulenc) or Veegum® (from R.T. Vanderbilt), and also organic and inorganic sheet minerals, such as Attaclay® (from Engelhardt).

Examples of antifoams are silicone emulsions (such as, for example, Silikon® SRE, Wacker or Rhodorsil® from Rhodia), long-chain alcohols, fatty acids, salts of fatty acids, organofluorine compounds and mixtures thereof.

Bactericides can be added for stabilizing the aqueous herbicidal formulation. Examples of bactericides are bactericides based on diclorophen and benzyl alcohol hemiformal (Proxel® from ICI or Acticide® RS from Thor Chemie and Kathon® MK

from Rohm & Haas), and also isothiazolinone derivates, such as alkylisothiazolinones and benzisothiazolinones (Acticide MBS from Thor Chemie).

Examples of antifreeze agents are ethylene glycol, propylene glycol, urea or glycerol.

Examples of colorants are both sparingly water-soluble pigments and water-soluble dyes. Examples which may be mentioned are the dyes known under the names Rhodamin B, C.I. Pigment Red 112 and C.I. Solvent Red 1, and also pigment blue 15:4, pigment blue 15:3, pigment blue 15:2, pigment blue 15:1, pigment blue 80, pigment yellow 1, pigment yellow 13, pigment red 112, pigment red 48:2, pigment red 48:1, pigment red 57:1, pigment red 53:1, pigment orange 43, pigment orange 34, pigment orange 5, pigment green 36, pigment green 7, pigment white 6, pigment brown 25, basic violet 10, basic violet 49, acid red 51, acid red 52, acid red 14, acid blue 9, acid yellow 23, basic red 10, basic red 108.

Examples of adhesives are polyvinylpyrrolidone, polyvinyl acetate, polyvinyl alcohol and tylose.

Suitable inert auxiliaries are, for example, the following:

mineral oil fractions of medium to high boiling point, such as kerosene and diesel oil, furthermore coal tar oils and oils of vegetable or animal origin, aliphatic, cyclic and aromatic hydrocarbons, for example paraffin, tetrahydronaphthalene, alkylated naphthalenes and their derivatives, alkylated benzenes and their derivatives, alcohols such as methanol, ethanol, propanol, butanol and cyclohexanol, ketones such as cyclohexanone or strongly polar solvents, for example amines such as N-methylpyrrolidone, and water.

Solid carriers are mineral earths such as silicas, silica gels, silicates, talc, kaolin, limestone, lime, chalk, bole, loess, clay, dolomite, diatomaceous earth, calcium sulfate, magnesium sulfate and magnesium oxide, ground synthetic materials, fertilizers such as ammonium sulfate, ammonium phosphate, ammonium nitrate and ureas, and products of vegetable origin, such as cereal meal, tree bark meal, wood meal and nutshell meal, cellulose powders, or other solid carriers.

Suitable surfactants (adjuvants, wetting agents, tackifiers, dispersants and also emulsifiers) are the alkali metal salts, alkaline earth metal salts and ammonium salts of aromatic sulfonic acids, for example lignosulfonic acids (e.g. Borresperse®-types, Borregaard), phenolsulfonic acids, naphthalenesulfonic acids (Morwet types, Akzo Nobel) and dibutylnaphthalenesulfonic acid (Nekal® types, BASF SE), and of fatty acids, alkyl- and alkylarylsulfonates, alkyl sulfates, lauryl ether sulfates and fatty alcohol sulfates, and salts of sulfated hexa-, hepta- and octadecanols, and also of fatty alcohol glycol ethers, condensates of sulfonated naphthalene and its derivatives with formaldehyde, condensates of naphthalene or of the naphthalenesulfonic acids with phenol and formaldehyde, polyoxyethylene octylphenol ether, ethoxylated isooctyl-, octyl- or nonylphenol, alkylphenyl or tributylphenyl polyglycol ether, alkylaryl polyether alcohols, isotridecyl alcohol, fatty alcohol/ethylene oxide condensates, ethoxylated

castor oil, polyoxyethylene alkyl ethers or polyoxypropylene alkyl ethers, lauryl alcohol polyglycol ether acetate, sorbitol esters, lignosulfite waste liquors and proteins, denatured proteins, polysaccharides (e.g. methylcellulose), hydrophobically modified starches, polyvinyl alcohol (Mowiol types Clariant), polycarboxylates (BASF SE, Sokalan® types), polyalkoxylates, polyvinylamine (BASF SE, Lupamine® types), polyethyleneimine (BASF SE, Lupasol® types), polyvinylpyrrolidone and copolymers thereof.

Powders, materials for broadcasting and dusts can be prepared by mixing or grinding the active ingredients together with a solid carrier.

Granules, for example coated granules, impregnated granules and homogeneous granules, can be prepared by binding the active ingredients to solid carriers.

Aqueous use forms can be prepared from emulsion concentrates, suspensions, pastes, wettable powders or water-dispersible granules by adding water. To prepare emulsions, pastes or oil dispersions, the compounds of the formula I or Ia, either as such or dissolved in an oil or solvent, can be homogenized in water by means of a wetting agent, tackifier, dispersant or emulsifier. Alternatively, it is also possible to prepare concentrates comprising active substance, wetting agent, tackifier, dispersant or emulsifier and, if desired, solvent or oil, which are suitable for dilution with water.

The concentrations of the compounds of the formula I in the ready-to-use preparations can be varied within wide ranges. In general, the formulations comprise from 0.001 to 98% by weight, preferably 0.01 to 95% by weight of at least one active compound. The active compounds are employed in a purity of from 90% to 100%, preferably 95% to 100% (according to NMR spectrum).

The compounds I of the invention can for example be formulated as follows:

1. Products for dilution with water

A Water-soluble concentrates

10 parts by weight of active compound are dissolved in 90 parts by weight of water or a water-soluble solvent. As an alternative, wetters or other adjuvants are added. The active compound dissolves upon dilution with water. This gives a formulation with an active compound content of 10% by weight.

B Dispersible concentrates

20 parts by weight of active compound are dissolved in 70 parts by weight of cyclohexanone with addition of 10 parts by weight of a dispersant, for example polyvinylpyrrolidone. Dilution with water gives a dispersion. The active compound content is 20% by weight.

C Emulsifiable concentrates

15 parts by weight of active compound are dissolved in 75 parts by weight of an organic solvent (e.g. alkylaromatics) with addition of calcium dodecylbenzenesulfonate and castor oil ethoxylate (in each case 5 parts by weight). Dilution with water gives an emulsion. The formulation has an active compound content of 15% by weight.

D Emulsions

25 parts by weight of active compound are dissolved in 35 parts by weight of an organic solvent (e.g. alkylaromatics) with addition of calcium dodecylbenzenesulfonate and castor oil ethoxylate (in each case 5 parts by weight). This mixture is introduced into 30 parts by weight of water by means of an emulsifier (e.g. Ultraturrax) and made into a homogeneous emulsion. Dilution with water gives an emulsion. The formulation has an active compound content of 25% by weight.

E Suspensions

In an agitated ball mill, 20 parts by weight of active compound are comminuted with addition of 10 parts by weight of dispersants and wetters and 70 parts by weight of water or an organic solvent to give a fine active compound suspension. Dilution with water gives a stable suspension of the active compound. The active compound content in the formulation is 20% by weight.

F Water-dispersible granules and water-soluble granules

50 parts by weight of active compound are ground finely with addition of 50 parts by weight 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. The formulation has an active compound content of 50% by weight.

G Water-dispersible powders and water-soluble powders

75 parts by weight of active compound are ground in a rotor-stator mill with addition of 25 parts by weight of dispersants, wetters and silica gel. Dilution with water gives a stable dispersion or solution of the active compound. The active compound content of the formulation is 75% by weight.

H Gel formulations

In a ball mill, 20 parts by weight of active compound, 10 parts by weight of dispersant, 1 part by weight of gelling agent and 70 parts by weight of water or of an organic solvent are ground to give a fine suspension. Dilution with water gives a stable suspension with active compound content of 20% by weight.

2. Products to be applied undiluted

I Dusts

5 parts by weight of active compound are ground finely and mixed intimately with 95 parts by weight of finely divided kaolin. This gives a dusting powder with an active compound content of 5% by weight.

J Granules (GR, FG, GG, MG)

0.5 parts by weight of active compound are ground finely and associated with 99.5 parts by weight of carriers. Current methods here are extrusion, spray-drying or the fluidized bed. This gives granules to be applied undiluted with an active compound content of 0.5% by weight.

K ULV solutions (UL)

10 parts by weight of active compound are dissolved in 90 parts by weight of an organic solvent, for example xylene. This gives a product to be applied undiluted with an active compound content of 10% by weight.

The compounds I or the herbicidal compositions comprising them can be applied pre- or post-emergence, or together with the seed of a crop plant. It is also possible to apply the herbicidal compositions or active compounds by applying seed, pretreated with the herbicidal compositions or active compounds, of a crop plant. If the active compounds are less well tolerated by certain crop plants, application techniques may be used in which the herbicidal compositions are sprayed, with the aid of the spraying equipment, in such a way that as far as possible they do not come into contact with the leaves of the sensitive crop plants, while the active compounds reach the leaves of undesirable plants growing underneath, or the bare soil surface (post-directed, lay-by).

In a further embodiment, the compounds of the formula I or the herbicidal compositions can be applied by treating seed.

The treatment of seed comprises essentially all procedures familiar to the person skilled in the art (seed dressing, seed coating, seed dusting, seed soaking, seed film coating, seed multilayer coating, seed encrusting, seed dripping and seed pelleting) based on the compounds of the formula I according to the invention or the compositions prepared therefrom. Here, the herbicidal compositions can be applied diluted or undiluted.

The term seed comprises seed of all types, such as, for example, corns, seeds, fruits, tubers, cuttings and similar forms. Here, preferably, the term seed describes corns and seeds.

The seed used can be seed of the useful plants mentioned above, but also the seed of transgenic plants or plants obtained by customary breeding methods.

The rates of application of active compound are from 0.001 to 3.0, preferably 0.01 to 1.0, kg/ha of active substance (a.s.), depending on the control target, the season, the target plants and the growth stage. To treat the seed, the compounds I are generally employed in amounts of from 0.001 to 10 kg per 100 kg of seed.

It may also be advantageous to use the compounds of the formula I in combination with safeners. Safeners are chemical compounds which prevent or reduce damage to useful plants without substantially affecting the herbicidal action of the compounds of the formula I on unwanted plants. They can be used both before sowing (for example in the treatment of seed, or on cuttings or seedlings) and before or after the emergence of the useful plant. The safeners and the compounds of the formula I can be used simultaneously or in succession. Suitable safeners are, for example, (quinolin-8-oxy)acetic acids, 1-phenyl-5-haloalkyl-1*H*-1,2,4-triazole-3-carboxylic acids, 1-phenyl-4,5-dihydro-5-alkyl-1*H*-pyrazole-3,5-dicarboxylic acids, 4,5-dihydro-5,5-diaryl-3-

isoxazolecarboxylic acids, dichloroacetamides, alpha-oximinophenylacetonitriles, acetophenone oximes, 4,6-dihalo-2-phenylpyrimidines, N-[[4-(aminocarbonyl)phenyl]-sulfonyl]-2-benzamides, 1,8-naphthalic anhydride, 2-halo-4-(haloalkyl)-5-thiazole-carboxylic acids, phosphorothiolates and O-phenyl N-alkylcarbamates and their agriculturally useful salts and, provided that they have an acid function, their agriculturally useful derivatives, such as amides, esters and thioesters.

To broaden the activity spectrum and to obtain synergistic effects, the compounds of the formula I can be mixed and jointly applied with numerous representatives of other herbicidal or growth-regulating groups of active compounds or with safeners. Suitable mixing partners are, for example, 1,2,4-thiadiazoles, 1,3,4-thiadiazoles, amides, aminophosphoric acid and its derivatives, aminotriazoles, anilides, aryloxy/heteroaryl-oxyalkanoic acids and their derivatives, benzoic acid and its derivatives, benzothiadiazinones, 2-(hetaroyl/aroyl)-1,3-cyclohexanediones, heteroaryl aryl ketones, benzylisoxazolidinones, meta-CF_3-phenyl derivatives, carbamates, quinoline carboxylic acid and its derivatives, chloroacetanilides, cyclohexenone oxime ether derivates, diazines, dichloropropionic acid and its derivatives, dihydrobenzofurans, dihydrofuran-3-ones, dinitroanilines, dinitrophenols, diphenyl ethers, dipyridyls, halocarboxylic acids and their derivatives, ureas, 3-phenyluracils, imidazoles, imidazolinones, N-phenyl-3,4,5,6-tetrahydrophthalimides, oxadiazoles, oxiranes, phenols, aryloxy- and heteroaryloxyphenoxypropionic esters, phenylacetic acid and its derivatives, 2-phenylpropionic acid and its derivatives, pyrazoles, phenylpyrazoles, pyridazines, pyridinecarboxylic acid and its derivatives, pyrimidyl ethers, sulfonamides, sulfonylureas, triazines, triazinones, triazolinones, triazolecarboxamides, uracils and also phenylpyrazolines and isoxazolines and their derivatives.

Moreover, it may be useful to apply the compounds I alone or in combination with other herbicides or else also mixed with further crop protection agents, jointly, for example with compositions for controlling pests or phytopathogenic fungi or bacteria. Also of interest is the miscibility with mineral salt solutions which are employed for alleviating nutritional and trace element deficiencies. Other additives such as nonphytotoxic oils and oil concentrates may also be added.

Examples of herbicides which can be used in combination with the compounds of the formula I according to the present invention are:

b1) from the group of the lipid biosynthesis inhibitors:

alloxydim, alloxydim-sodium, butroxydim, clethodim, clodinafop, clodinafop-propargyl, cycloxydim, cyhalofop, cyhalofop-butyl, diclofop, diclofop-methyl, fenoxaprop, fenoxaprop-ethyl, fenoxaprop-P, fenoxaprop-P-ethyl, fluazifop, fluazifop-butyl, fluazifop-P, fluazifop-P-butyl, haloxyfop, haloxyfop-methyl, haloxyfop-P, haloxyfop-P-methyl, metamifop, pinoxaden, profoxydim, propaquizafop, quizalofop, quizalofop-ethyl, quizalofop-tefuryl, quizalofop-P, quizalofop-P-ethyl, quizalofop-P-tefuryl, sethoxydim, tepraloxydim, tralkoxydim, benfuresate, butylate, cycloate, dalapon, dimepiperate, EPTC, esprocarb, ethofumesate, flupropanate, molinate,

orbencarb, pebulate, prosulfocarb, TCA, thiobencarb, tiocarbazil, triallate and vernolate;

b2) from the group of the ALS inhibitors:

amidosulfuron, azimsulfuron, bensulfuron, bensulfuron-methyl, bispyribac, bispyribac-sodium, chlorimuron, chlorimuron-ethyl, chlorsulfuron, cinosulfuron, cloransulam, cloransulam-methyl, cyclosulfamuron, diclosulam, ethametsulfuron, ethametsulfuron-methyl, ethoxysulfuron, flazasulfuron, florasulam, flucarbazone, flucarbazone-sodium, flucetosulfuron, flumetsulam, flupyrsulfuron, flupyrsulfuron-methyl-sodium, foramsulfuron, halosulfuron, halosulfuron-methyl, imazamethabenz, imazamethabenz-methyl, imazamox, imazapic, imazapyr, imazaquin, imazethapyr, imazosulfuron, iodosulfuron, iodosulfuron-methyl-sodium, mesosulfuron, metosulam, metsulfuron, metsulfuron-methyl, nicosulfuron, orthosulfamuron, oxasulfuron, penoxsulam, primisulfuron, primisulfuron-methyl, propoxycarbazone, propoxycarbazone-sodium, prosulfuron, pyrazosulfuron, pyrazosulfuron-ethyl, pyribenzoxim, pyrimisulfan, pyriftalid, pyriminobac, pyriminobac-methyl, pyrithiobac, pyrithiobac-sodium, pyroxsulam, rimsulfuron, sulfometuron, sulfometuron-methyl, sulfosulfuron, thiencarbazone, thiencarbazone-methyl, thifensulfuron, thifensulfuron-methyl, triasulfuron, tribenuron, tribenuron-methyl, trifloxysulfuron, triflusulfuron, triflusulfuron-methyl and tritosulfuron;

b3) from the group of the photosynthesis inhibitors:

ametryn, amicarbazone, atrazine, bentazone, bentazone-sodium, bromacil, bromofenoxim, bromoxynil and its salts and esters, chlorobromuron, chloridazone, chlorotoluron, chloroxuron, cyanazine, desmedipham, desmetryn, dimefuron, dimethametryn, diquat, diquat-dibromide, diuron, fluometuron, hexazinone, ioxynil and its salts and esters, isoproturon, isouron, karbutilate, lenacil, linuron, metamitron, methabenzthiazuron, metobenzuron, metoxuron, metribuzin, monolinuron, neburon, paraquat, paraquat-dichloride, paraquat-dimetilsulfate, pentanochlor, phenmedipham, phenmedipham-ethyl, prometon, prometryn, propanil, propazine, pyridafol, pyridate, siduron, simazine, simetryn, tebuthiuron, terbacil, terbumeton, terbuthylazine, terbutryn, thidiazuron and trietazine;

b4) from the group of the protoporphyrinogen-IX oxidase inhibitors:

acifluorfen, acifluorfen-sodium, azafenidin, bencarbazone, benzfendizone, bifenox, butafenacil, carfentrazone, carfentrazone-ethyl, chlomethoxyfen, cinidon-ethyl, fluazolate, flufenpyr, flufenpyr-ethyl, flumiclorac, flumiclorac-pentyl, flumioxazin, fluoroglycofen, fluoroglycofen-ethyl, fluthiacet, fluthiacet-methyl, fomesafen, halosafen, lactofen, oxadiargyl, oxadiazon, oxyfluorfen, pentoxazone, profluazol, pyraclonil, pyraflufen, pyraflufen-ethyl, saflufenacil, sulfentrazone, thidiazimin, 2-chloro-5-[3,6-dihydro-3-methyl-2,6-dioxo-4-(trifluoromethyl)-1(2*H*)-pyrimidinyl]-4-fluoro-N-[(isopropyl)-methylsulfamoyl]benzamide (B-1; CAS 372137-35-4), ethyl [3-[2-chloro-4-fluoro-5-(1-methyl-6-trifluoromethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-3-yl)phenoxy]-2-pyridyloxy]acetate (B-2; CAS 353292-31-6), N-ethyl-3-(2,6-dichloro-4-trifluoro-

methylphenoxy)-5-methyl-1*H*-pyrazole-1-carboxamide (B-3; CAS 452098-92-9), N-tetrahydrofurfuryl-3-(2,6-dichloro-4-trifluoromethylphenoxy)-5-methyl-1*H*-pyrazole-1-carboxamide (B-4; CAS 915396-43-9), N-ethyl-3-(2-chloro-6-fluoro-4-trifluoromethyl-phenoxy)-5-methyl-1*H*-pyrazole-1-carboxamide (B-5; CAS 452099-05-7) and N-tetrahydrofurfuryl-3-(2-chloro-6-fluoro-4-trifluoromethylphenoxy)-5-methyl-1*H*-pyrazole-1-carboxamide (B-6; CAS 452100-03-7);

b5) from the group of the bleacher herbicides:

aclonifen, amitrol, beflubutamid, benzobicyclon, benzofenap, clomazone, diflufenican, fluridone, flurochloridone, flurtamone, isoxaflutole, mesotrione, norflurazon, picolinafen, pyrasulfutole, pyrazolynate, pyrazoxyfen, sulcotrione, tefuryltrione, tembotrione, topramezone, 4-hydroxy-3-[[2-[(2-methoxyethoxy)methyl]-6-(trifluoromethyl)-3-pyridyl]carbonyl]bicyclo[3.2.1]oct-3-en-2-one (B-7; CAS 352010-68-5) and 4-(3-trifluoromethylphenoxy)-2-(4-trifluoromethylphenyl)pyrimidine (B-8; CAS 180608-33-7);

b6) from the group of the EPSP synthase inhibitors:

glyphosate, glyphosate-isopropylammonium and glyphosate-trimesium (sulfosate);

b7) from the group of the glutamine synthase inhibitors:

bilanaphos (bialaphos), bilanaphos-sodium, glufosinate and glufosinate-ammonium;

b8) from the group of the DHP synthase inhibitors:

asulam;

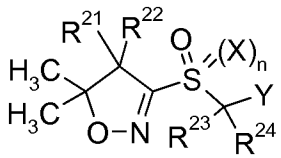
b9) from the group of the mitose inhibitors:

amiprophos, amiprophos-methyl, benfluralin, butamiphos, butralin, carbetamide, chlorpropham, chlorthal, chlorthal-dimethyl, dinitramine, dithiopyr, ethalfluralin, fluchloralin, oryzalin, pendimethalin, prodiamine, propham, propyzamide, tebutam, thiazopyr and trifluralin;

b10) from the group of the VLCFA inhibitors:

acetochlor, alachlor, anilofos, butachlor, cafenstrole, dimethachlor, dimethanamid, dimethenamid-P, diphenamid, fentrazamide, flufenacet, mefenacet, metazachlor, metolachlor, metolachlor-S, naproanilide, napropamide, pethoxamid, piperophos, pretilachlor, propachlor, propisochlor, pyroxasulfone (KIH-485) and thenylchlor;

Compounds of the formula 2:

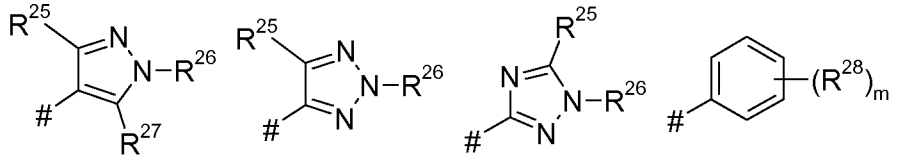


2

in which the variables have the following meanings:

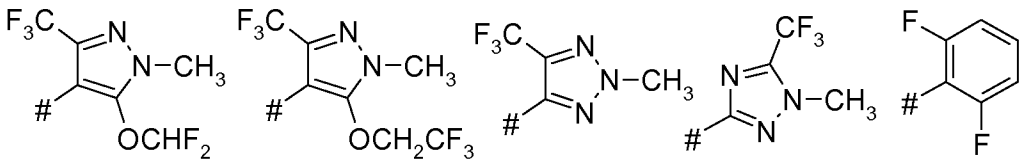
Y is phenyl or 5- or 6-membered heteroaryl as defined at the outset, which radicals may be substituted by one to three groups R^{aa}; R^{21},R^{22},R^{23},R^{24} are H, halogen or C_1-C_4-alkyl; X is O or NH; N is 0 or 1.

Compounds of the formula 2 have in particular the following meanings:

Y is  ,

where # denotes the bond to the skeleton of the molecule; and R^{21},R^{22},R^{23},R^{24} are H, Cl, F or CH_3; R^{25} is halogen, C_1-C_4-alkyl or C_1-C_4-haloalkyl; R^{26} is C_1-C_4-alkyl; R^{27} is halogen, C_1-C_4-alkoxy or C_1-C_4-haloalkoxy; R^{28} is H, halogen, C_1-C_4-alkyl, C_1-C_4-haloalkyl or C_1-C_4-haloalkoxy; M is 0, 1, 2 or 3; X is oxygen; N is 0 or 1.

Preferred compounds of the formula 2 have the following meanings:

Y is 

R^{21} is H; R^{22},R^{23} are F; R^{24} is H or F; X is oxygen; N is 0 or 1.

Particularly preferred compounds of the formula 2 are:
3-[5-(2,2-difluoroethoxy)-1-methyl-3-trifluoromethyl-1H-pyrazol-4-ylmethane-sulfonyl]-4-fluoro-5,5-dimethyl-4,5-dihydroisoxazole (2-1); 3-{[5-(2,2-difluoro-ethoxy)-1-methyl-3-trifluoromethyl-1H-pyrazol-4-yl]fluoromethanesulfonyl}-5,5-dimethyl-4,5-dihydroisoxazole (2-2); 4-(4-fluoro-5,5-dimethyl-4,5-dihydroisoxazole-3-sulfonyl-methyl)-2-methyl-5-trifluoromethyl-2H-[1,2,3]triazole (2-3); 4-[(5,5-dimethyl-4,5-dihydroisoxazole-3-sulfonyl)fluoromethyl]-2-methyl-5-trifluoromethyl-2H-[1,2,3]triazole (2-4); 4-(5,5-dimethyl-4,5-dihydroisoxazole-3-sulfonylmethyl)-2-methyl-5-trifluoro-methyl-2H-[1,2,3]triazole (2-5); 3-{[5-(2,2-difluoroethoxy)-1-methyl-3-trifluoromethyl-1H-pyrazol-4-yl]difluoromethanesulfonyl}-5,5-dimethyl-4,5-dihydroisoxazole (2-6); 4-[(5,5-dimethyl-4,5-dihydroisoxazole-3-sulfonyl)difluoromethyl]-2-methyl-5-trifluoro-methyl-2H-[1,2,3]triazole (2-7); 3-{[5-(2,2-difluoroethoxy)-1-methyl-3-trifluoromethyl-1H-pyrazol-4-yl]difluoromethanesulfonyl}-4-fluoro-5,5-dimethyl-4,5-dihydroisoxazole (2-8); 4-[difluoro-(4-fluoro-5,5-dimethyl-4,5-dihydroisoxazole-3-sulfonyl)methyl]-2-methyl-5-trifluoromethyl-2H-[1,2,3]triazole (2-9);

b11) from the group of the cellulose biosynthesis inhibitors:
chlorthiamid, dichlobenil, flupoxam and isoxaben;

b12) from the group of the decoupler herbicides:
dinoseb, dinoterb and DNOC and its salts;

b13) from the group of the auxin herbicides:
2,4-D and its salts and esters, 2,4-DB and its salts and esters, aminopyralid and its salts such as aminopyralid-tris(2-hydroxypropyl)ammonium and its esters, benazolin, benazolin-ethyl, chloramben and its salts and esters, clomeprop, clopyralid and its salts and esters, dicamba and its salts and esters, dichlorprop and its salts and esters,

dichlorprop-P and its salts and esters, fluroxypyr, fluroxypyr-butometyl, fluroxypyr-meptyl, MCPA and its salts and esters, MCPA-thioethyl, MCPB and its salts and esters, mecoprop and its salts and esters, mecoprop-P and its salts and esters, picloram and its salts and esters, quinclorac, quinmerac, TBA (2,3,6) and its salts and esters, triclopyr and its salts and esters, and 5,6-dichloro-2-cyclopropyl-4-pyrimidinecarboxylic acid (B-9; CAS 858956-08-8) and its salts and esters;

b14) from the group of the auxin transport inhibitors: diflufenzopyr, diflufenzopyr-sodium, naptalam and naptalam-sodium;

b15) from the group of the other herbicides: bromobutide, chlorflurenol, chlorflurenol-methyl, cinmethylin, cumyluron, dalapon, dazomet, difenzoquat, difenzoquat-metilsulfate, dimethipin, DSMA, dymron, endothal and its salts, etobenzanid, flamprop, flamprop-isopropyl, flamprop-methyl, flamprop-M-isopropyl, flamprop-M-methyl, flurenol, flurenol-butyl, flurprimidol, fosamine, fosamine-ammonium, indanofan, maleic hydrazide, mefluidide, metam, methyl azide, methyl bromide, methyl-dymron, methyl iodide, MSMA, oleic acid, oxaziclomefone, pelargonic acid, pyributicarb, quinoclamine, triaziflam, tridiphane and 6-chloro-3-(2-cyclopropyl-6-methylphenoxy)-4-pyridazinol (B-10; CAS 499223-49-3) and its salts and esters.

Examples of preferred safeners C are benoxacor, cloquintocet, cyometrinil, cyprosulfamide, dichlormid, dicyclonone, dietholate, fenchlorazole, fenclorim, flurazole, fluxofenim, furilazole, isoxadifen, mefenpyr, mephenate, naphthalic anhydride, oxabetrinil, 4-(dichloroacetyl)-1-oxa-4-azaspiro[4.5]decane (B-11; MON4660, CAS 71526-07-3) and 2,2,5-trimethyl-3-(dichloroacetyl)-1,3-oxazolidine (B-12; R-29148, CAS 52836-31-4).

The active compounds of groups b1) to b15) and the safeners C are known herbicides and safeners, see, for example, The Compendium of Pesticide Common Names (http://www.alanwood.net/pesticides/); B. Hock, C. Fedtke, R. R. Schmidt, Herbizide [Herbicides], Georg Thieme Verlag, Stuttgart, 1995. Further herbicidally active compounds are known from WO 96/26202, WO 97/41116, WO 97/41117, WO 97/41118, WO 01/83459 and WO 2008/074991 and from W. Krämer et al. (ed.) "Modern Crop Protection Compounds", Vol. 1, Wiley VCH, 2007 and the literature quoted therein.

The compounds I and the compositions according to the invention may also have a plant-strengthening action. Accordingly, they are suitable for mobilizing the defense system of the plants against attack by unwanted microorganisms, such as harmful fungi, but also viruses and bacteria. Plant-strengthening (resistance-inducing) substances are to be understood as meaning, in the present context, those substances which are capable of stimulating the defense system of treated plants in such a way that, when subsequently inoculated by unwanted microorganisms, the treated plants display a substantial degree of resistance to these microorganisms.

The compounds I can be employed for protecting plants against attack by unwanted

microorganisms within a certain period of time after the treatment. The period of time within which their protection is effected generally extends from 1 to 28 days, preferably from 1 to 14 days, after the treatment of the plants with the compounds I, or, after treatment of the seed, for up to 9 months after sowing.

The compounds I and the compositions according to the invention are also suitable for increasing the harvest yield.

Moreover, they have reduced toxicity and are tolerated well by the plants.

Hereinbelow, the preparation of compounds of the formula I is illustrated by way of examples, without limiting the subject matter of the present invention to the examples shown.

Synthesis examples

With appropriate modification of the starting materials, the procedures given in the synthesis examples below were used to obtain further compounds I. The compounds obtained in this manner are listed in the table that follows, together with physical data.

The products shown below were characterized by melting point determination, by NMR spectroscopy or by the mass ([m/z]) or retention time (RT; [min.]) determined by HPLC-MS spectrometry.

HPLC-MS = high performance liquid chromatography-coupled mass spectrometry;
HPLC column:

RP-18 column (Chromolith Speed ROD from Merck KgaA, Germany), 50*4.6 mm; mobile phase: acetonitrile + 0.1% trifluoroacetic acid (TFA)/water + 0.1% TFA using a gradient from 5:95 to 100:0 over 5 minutes at 40°C, flow rate 1.8 ml/min.
MS: quadrupole electrospray ionization, 80 V (positive mode).

I. Preparation examples

Example 1: Preparation of methyl 3-(3-bromo-5-fluorophenyl)-4-cyano-4-(3-fluorophenyl)butyrate [I-316]

At -78°C and under argon, 248 mg of (3-fluorophenyl)acetonitrile were added dropwise to a solution of 238 mg of potassium tert-butoxide in 2 ml of THF, and a solution of 500 mg of methyl 3-(3-bromo-5-fluorophenyl)acrylate in 1 ml of THF was then added. After 1 h of stirring at -78°C, 5 ml of CH_2Cl_2 were added. After washing with 2N HCl, sat. $NaHCO_3$ solution and sat. NaCl solution, drying of the organic phase and removal of the solvent under reduced pressure, the residue was purified by preparative HPLC.

This gave 215 mg of the title compound (erythro:threo ratio 1.9:1).

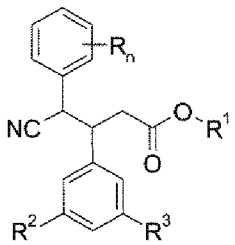
Example 2: Preparation of 3-(3-bromo-5-fluorophenyl)-4-cyano-4-(3-fluorophenyl)-butyric acid [I-309]

80 mg of LiOH were added to a solution of 800 mg of methyl 3-(3-bromo-5-fluorophenyl)-4-cyano-4-(3-fluorophenyl)butyrate in 5 ml of THF. After 3 h of stirring at 45°C, 10 ml of CH_2Cl_2 were added at 20-25°C. After addition of 2N HCl and extraction with CH_2Cl_2, the organic phase was washed with sat. NaCl solution and dried and the solvent was removed under reduced pressure. 450 mg of the residue were separated off for further reactions. The remainder was purified by chromatography on silica gel (cyclohexane:ethyl acetate 9:1->4:1->ethyl acetate). This gave 47 mg of the acid (erythro:threo ratio 1:1).

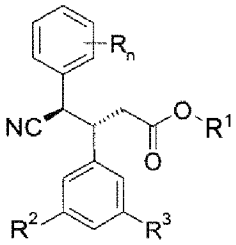
Example 3: Preparation of prop-2-ynyl 3-(3-bromo-5-fluorophenyl)-4-cyano-4-(3-fluorophenyl)butyrate [I-339]

A solution of 148 mg of 3-(3-bromo-5-fluorophenyl)-4-cyano-4-(3-fluorophenyl)butyric acid, 113 mg of 3-(ethyliminomethylideneamino)-N,N-dimethylpropane-1-amine and 4.8 mg of N,N-dimethylpyridine-4-amine in 3 ml of CH_2Cl_2 was stirred at 20-25°C. After 10 min, 44 mg of propargyl alcohol were added. After about 15 hours of stirring, the reaction was terminated by addition of 2N HCl and the solution was washed with sat. $NaHCO_3$ solution and sat. NaCl solution. After drying of the organic phase and removal of the solvent under reduced pressure, the residue was purified by preparative HPLC. This gave 10 mg of the title compound (erythro:threo ratio 3:2).

Table I: Compounds of the formula I:



I racemate

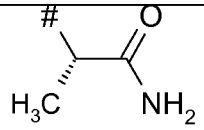
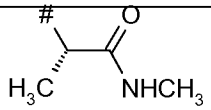
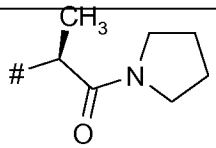
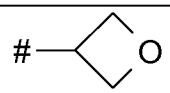


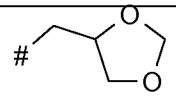
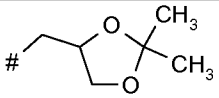
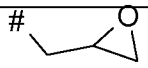
isomer 1

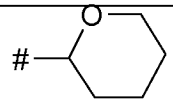
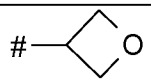
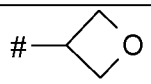
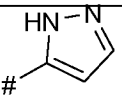
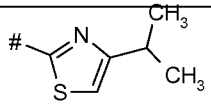
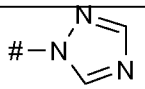
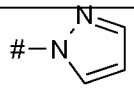
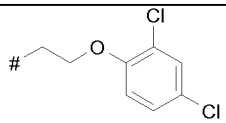
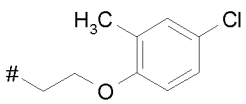
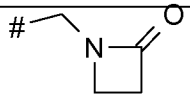
No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-1	2-Cl,5-F	CH_3	Cl	Cl	
I-2	2,4-Cl_2	CH_3	Cl	Cl	
I-3	2,3-Cl_2	CH_3	Cl	Cl	
I-4	3-CF_3	CH_3	Cl	Cl	
I-5	3-CN	CH_3	Cl	Cl	
I-6	4-Br	CH_3	Cl	Cl	
I-7	2,6-F_2	CH_3	Cl	Cl	

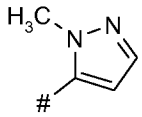
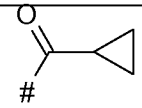
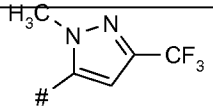
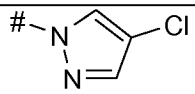
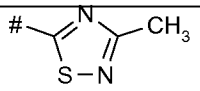
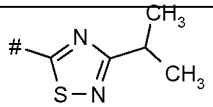
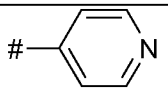
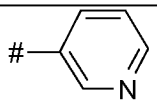
No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-8	3,4-F_2	CH_3	Cl	Cl	
I-9	3-F,4-CF_3	CH_3	Cl	Cl	
I-10	3-CF_3	CH_3	Cl	Cl	
I-11	3-NO_2	CH_3	Cl	Cl	
I-12	2-SCF_3	CH_3	Cl	Cl	
I-13	-	CH_2CH_3	CH_3	CH_3	3.813/322 (M+1)
I-14	-	CH_3	CH_3	CH_3	3.639/308 (M+1)
I-15	-	CH_3	$C(CH_3)_3$	$C(CH_3)_3$	4.476/499 (M+107)
I-16	4-OCH_2CH_3	CH_3	Cl	Cl	
I-17	4-SCH_3	CH_3	Cl	Cl	
I-18	4-F	CH_3	F	F	3.540/334 (M+1)
I-19	-	CH_3	Cl	Cl	3.823/347 (M-1)
I-20	2-CH_3	CH_3	Cl	Cl	
I-21	4-Cl	CH_3	Cl	Cl	
I-22	3-F	CH_3	Cl	Cl	
I-23	3-I	CH_3	Cl	Cl	
I-24	4-I	CH_3	Cl	Cl	
I-25	4-$CH=CH_2$	CH_3	Cl	Cl	
I-26	4-$C(CH_3)_3$	CH_3	Cl	Cl	
I-27	3-$OCHF_2$	CH_3	Cl	Cl	
I-28	3-SCF_3	CH_3	Cl	Cl	
I-29	2-$OCHF_2$	CH_3	Cl	Cl	
I-30	4-SCF_3	CH_3	Cl	Cl	
I-31	3-OCH_3	CH_3	Cl	Cl	
I-32	4-OCH_3	CH_3	Cl	Cl	
I-33	3-CH_3	CH_3	Cl	Cl	
I-34	2,4-F_2	CH_3	Cl	Cl	
I-35	4-$NHC(=O)CF_3$	CH_3	Cl	Cl	
I-36	3,4,5-F_3	CH_3	Cl	Cl	
I-37	-	$CH(CH_3)$-$C(=O)OCH_3$	Cl	Cl	
I-38	4-F	H	F	F	2.914/342 (M+23)
I-39	-	H	Cl	Cl	3.165/367 (M+23)
I-40	-	CH_3	Br	Br	3.831/459 (M+22)
I-41	-	CH_3	OCH_3	OCH_3	3.295/340 (M+1)
I-42	-	CH_3	F	CF_3	3.714 / 365

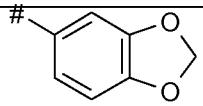
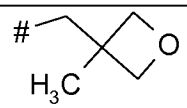
No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-43	-	CH_3	F	F	3.433/316 (M+1)
I-44	-	CH_3	F	Cl	3.423/354 (M+23)
I-45	2,5-F_2	$CH_2C\equiv CH$	Cl	Cl	3.638/430 (M+22)
I-46	2,5-F_2	$CH[C(CH_3)_3]_2$	Cl	Cl	4.097 / 520
I-47	2,5-F_2	$CH_2C_3H_5$	Cl	Cl	3.920/446 (M+22)
I-48	2,5-F_2	$CH_2CH=CH_2$	Cl	Cl	3.812 / 410
I-49	2,5-F_2	$CH_2CH_2CH_3$	Cl	Cl	3.922/434 (M+22)
I-50	2,5-F_2	H	Cl	Cl	3.319/392 (M+22)
I-51	3-F,4-Cl	CH_3	Cl	Cl	3.962/421 (M+21)
I-52	3-Cl,5-F	CH_3	Cl	Cl	3.970/421 (M+21)
I-53	4-NO_2	CH_3	Cl	Cl	3.663/415 (M+23)
I-54	3-OCF_3,4-Cl	CH_3	Cl	Cl	3.233/415 (M-51)
I-55	2,3,5-F_3	CH_3	Cl	Cl	3.825/423 (M+21)
I-56	4-C≡ CH	CH_3	Cl	Cl	3.801/394 (M+22)
I-57	2,5-F_2	$CH(CH_3)_2$	Cl	Cl	4.189/434 (M+22)
I-58	2,5-F_2	$CH_2CH(CH_3)_2$	Cl	Cl	4.342/448 (M+22)
I-59	3-Br	CH_3	Cl	Cl	3.860/449 (M+26)
I-60	2-Cl,5-F	CH_3	Cl	Cl	3.876 / 402 (M+2)
I-61	3,5-F_2	CH_3	Cl	Cl	3.500/339 (M-45)
I-62	4-$OCHF_2$	CH_3	Cl	Cl	3.884/436 (M+22)
I-63	2,4,5-F_3	CH_3	Cl	Cl	3.995/424 (M+22)
I-64	2,5-F_2	N=C(CH_3)-CH(CH_3)SCH_3	Cl	Cl	3.924 / 420 (M-65)
I-65	4-F	$CH_2C\equiv CH$	Cl	Cl	4.012/412 (M+23)
I-66	3-F,4-OCH_3	CH_3	Cl	Cl	3.780/418 (M+34)
I-67	3,5-F_2, 4-OCH_3	CH_3	Cl	Cl	3.908 / 436 (M+22)
I-68	3-F	$CH_2C\equiv CH$	Cl	Cl	3.950/412 (M+23)
I-69	3-F,5-CH_3	CH_3	Cl	Cl	4.095/402 (M+22)
I-70	2,5-F_2	CH_3	F	Cl	3.716/390 (M+23)
I-71	4-F	CH_3	F	Cl	3.687/350 (M+1)
I-72	-	$CH_2C\equiv CH$	Cl	Cl	3.902/394 (M+22)
I-73	-	CH_2CN	Cl	Cl	3.628/390 (M+17)
I-74	-	$CH(CH_3)C_3H_5$	Cl	Cl	
I-75	-	$CH(CH_3)$-$CH_2C\equiv CH$	Cl	Cl	
I-76	2,5-F_2	CH_2CN	Cl	Cl	3.643 / 409
I-77	4-F	$CH_2C\equiv CH$	F	F	3.527/380 (M+23)

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-78	-	$CH_2C\equiv CH$	F	F	3.631/361 (M+22)
I-79	-	$C(CH_3)_2CH=CH-C(CH_3)_2NH_2$	Cl	Cl	3.539 / 440 (M-19)
I-80	-		Cl	Cl	3.417 / 418 (M-1)
I-81	-		Cl	Cl	3.562 / 432 (M-1)
I-82	-		Cl	Cl	3.686 / 458 (M-1)
I-83	-	$CH(CH=CH_2)-CH_2CN$	Cl	Cl	3.828/435 (M+22)
I-84	-	$CH(C_2H_5)-C\equiv CCH_3$	Cl	Cl	4.431/436 (M+22)
I-85	-	$CH(C_2H_5)C\equiv CH$	Cl	Cl	4.178/466 (M+66)
I-86	-	$CH(C_2H_5)-CH=CH_2$	Cl	Cl	4.489/424 (M+22)
I-87	-	$CH(C_2H_5)-CH_2C\equiv CH$	Cl	Cl	4.343/436 (M+22)
I-88	-	$CH_2C\equiv CH$	F	Cl	3.773/378 (M+23)
I-89	-	$CH_2CH=CH_2$	Cl	Cl	4.107 / 374
I-90	-	$CH_2CH_2CH_3$	Cl	Cl	4.229 / 376
I-91	-	$C(CH_3)_2CH=CH_2$	Cl	Cl	4.311/424 (M+22)
I-92	-	$C(CH_3)_2CH_2CH_3$	Cl	Cl	4.462/426 (M+22)
I-93	-		Cl	Cl	3.674 / 390
I-94	-	$C(CH_3)_2CN$	Cl	Cl	3.928/423 (M+22)
I-95	-	$(1\text{-}C_2H_5)C_3H_5$	Cl	Cl	4.281/424 (M+22)
I-96	-	$CH(CH_3)-C(=O)CH_3$	Cl	Cl	3.807/426 (M+22)
I-97	4-F	CH_3	Cl	Cl	3.763/388 (M-1)
I-98	-	$CH(CH_3)CN$	Cl	Cl	3.835/409 (M+22)
I-99	-	$CH(CH_3)C\equiv CH$	Cl	Cl	4.014/408 (M+22)
I-100	$2,5\text{-}F_2$	CH_3	Cl	Cl	
I-101	$3,4\text{-}F_2$	$CH_2C\equiv CH$	Cl	Cl	3.945/430 (M+22)
I-102	-	$(1\text{-}CN)C_6H_{11}$	Cl	Cl	4.198/463 (M+22)
I-103	-	$(1\text{-}C\equiv CH)C_6H_{11}$	Cl	Cl	4.408/462 (M+22)
I-104	-	$CH_2C\equiv CCH_3$	Cl	Cl	4.034/386 (M-64)
I-105	-	$C(CH_3)_2C\equiv CH$	Cl	Cl	4.059/422 (M+22)
I-106	-	$(1\text{-}CN)C_5H_9$	Cl	Cl	4.246/448 (M+22)

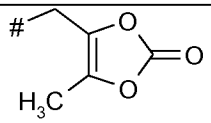
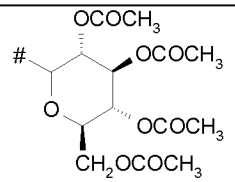
No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-107	-	CH_2CH_2-C(=O)OCH_3	Cl	Cl	3.796/442 (M+22)
I-108	-	$CH_2C(=O)OCH_3$	Cl	Cl	3.789/428 (M+22)
I-109	-	$CH_2C(=O)CH_3$	Cl	Cl	3.680/412 (M+22)
I-110	-		Cl	Cl	3.716 / 442 (M+22)
I-111	-		Cl	Cl	3.967 / 390 (M-58)
I-112	-	$CH_2OCH_2CH_3$	Cl	Cl	3.951/414 (M+22)
I-113	-	$CH_2OCH_2CH_2$-OCH_3	Cl	Cl	3.693/444 (M+22)
I-114	-	$CH_2CH_2C\equiv CH$	Cl	Cl	
I-115	-	CH_3	F	CH_3	3.560 / 312 (M+1)
I-116		$CH_2CH=CClCH_3$	Cl	Cl	
I-117	-	$CH_2CH=NO$-$CH_2CH=CH_2$	Cl	Cl	
I-118	-	$CH_2C\equiv CC_2C_5$	Cl	Cl	
I-119	-	$CH_2CH=NOCH_3$	Cl	Cl	3.871 + 4.233 / 426 (M+21) + 497(M+92)
I-120	-	$CH_2C\equiv CC_3C_7$	Cl	Cl	4.294 / 414
I-121	2,5-F_2	$CH_2C\equiv CH$	F	Cl	3.765/413 (M+22)
I-122	-	CH_3	Br	Cl	3.784/416 (M+24)
I-123	-	CH_2SCH_3	Cl	Cl	3.872/416 (M+22)
I-124	-	$CH_2CH=C=CH_2$	Cl	Cl	3.989/408 (M+22)
I-125	-		Cl	Cl	
I-126	-	$N=C[CH(CH_3)_2]_2$	Cl	Cl	4.268/467 (M+22)
I-127	-	$CH_2C\equiv CH$	F	CH_3	3.524 / 336 (M+1)
I-128	-	$CH_2C\equiv CH$	F	Br	3.586/424 (M+24)
I-129	-	CH_2OCH_3	Cl	Cl	3.681/399 (M+23)
I-130	3-Cl	CH_3	Cl	Cl	3.928/384 (M+H)
I-131	3-F,4-CH_3	CH_3	Cl	Cl	3.957/380 (M+H)
I-132	3-Cl,4-F	CH_3	Cl	Cl	3.956/402 (M+H)
I-133	2-CH_3,5-F	CH_3	Cl	Cl	3.892/380 (M+H)
I-134	4-OH	CH_3	Cl	Cl	3.238 / 386 (M+Na)

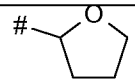
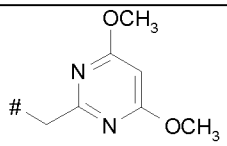
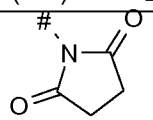
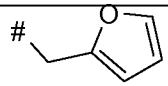
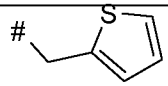
No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-135	-		Cl	Cl	3.245 / 355 (M-62)
I-136	-	$CH_2C\equiv CH$	CF_3	CF_3	3.801/461 (M+22)
I-137	-	$CH_2C(CH_3)=CH_2$	Cl	Cl	4.018 / 388
I-138	4-F	$CH_2C\equiv CH$	F	Cl	3.604/396 (M+23)
I-139	-	$N=C(CH_3)_2$	Cl	Cl	3.583/388 (M-2)
I-140	-	$CH_2CCl=CH_2$	Cl	Cl	4.017/429 (M+21)
I-141	-	$CH_2C\equiv CH$	Cl	Br	3.953/439 (M+23)
I-142	-	$CH_2C\equiv CH$	Cl	OCF_3	3.795/443 (M+22)
I-143	-	$N=CHCH_3$	Cl	Cl	
I-144	-	$CH_2SO_2CH_3$	Cl	Cl	3.532 / 425
I-145	-	2,6-Br_2,4-CN-C_6H_2	Cl	Cl	4.125/615 (M+22)
I-146	-	2,4-Cl_2-C_6H_3	Cl	Cl	4.296/501 (M+22)
I-147	4-F		F	F	3.260 / 376 (M+1)
I-148	4-F		F	Cl	3.405 / 392 (M+1)
I-149	2,3,5,6-F_4	CH_3	Cl	Cl	isomer 1:
I-150	2,3,5,6-F_4	$CH_2C\equiv CH$	Cl	Cl	
I-151	2,3,5,6-F_4	CH_3	Cl	Cl	
I-152	-		Cl	Cl	3.454 / 422 (M+23)
I-153	-		Cl	Cl	4.345 / 144 (M-314)
I-154	-		Cl	Cl	3.077 / 291 (M-110)
I-155	-		Cl	Cl	3.206 / 355 (M-45)
I-156	-		Cl	Cl	4.576 / 545 (M+22)
I-157	-		Cl	Cl	4.621 / 523 (M+21)
I-158	-		Cl	Cl	3.353 / 439 (M+22)

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-159	-		Cl	Cl	3.500 / 207 (M-207)
I-160	-		Cl	Cl	
I-161	-	H	F	F	2.881 / 302 (M+1)
I-162	-	$CH_2C\equiv CH$	Br	Br	3.925/483 (M+23)
I-163	3-Cl	$CH_2C\equiv CH$	Cl	Cl	4.123/427 (M+22)
I-164	-		Cl	Cl	4.019 / 374 (M-108)
I-165	-		Cl	Cl	3.792 / 326 (M-108)
I-166	4-F	CH_2CN	F	F	3.324/381 (M+23)
I-167	4-F	CH_2CN	F	Cl	3.647/397 (M+23)
I-168	$2,3\text{-}F_2$	CH_3	Cl	Cl	
I-169	F_5	CH_3	Cl	Cl	3.885/438 (M+H)
I-170	$2,3,4\text{-}F_3$	CH_3	Cl	Cl	
I-171	2-F	CH_3	Cl	Cl	
I-172	$2,4,6\text{-}F_3$	CH_3	Cl	Cl	
I-173	-		Cl	Cl	3.242 / 396 (M-35)
I-174	-		Cl	Cl	
I-175	-		Cl	Cl	3.438 / 334 (M-77)
I-176	-	CH_2CH_2F	Cl	Cl	3.841/402 (M+22)
I-177	-	CH_2CF_3	Cl	Cl	4.106/438 (M+22)
I-178	-		Cl	Cl	3.340 / 301 (M-110)
I-179	-	$CH_2C\equiv CH$	CN	CN	3.841/352 (M-1)
I-180	-	$CH_2C\equiv CH$	I	I	4.075/577 (M+22)
I-181	4-OC(=O) $N(CH_3)_2$	CH_3	Cl	Cl	
I-182	4- $OC(=O)CH_3$	CH_3	Cl	Cl	3.761 / 428 (M+Na)

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-183	-	$N=C(CH_3)-C(=O)OCH_3$	Cl	Cl	3.693 / 455 (M+22)
I-184	4-$OC(=O)NHCH_3$	CH_3	Cl	Cl	
I-185	3-OH	CH_3	Cl	Cl	
I-186	3,5-Cl_2	CH_3	F	Cl	4.122/424(M+Na)
I-187	3-Cl	CH_3	F	Cl	3.885/366 (M+H)
I-188	3-Br	CH_3	F	Cl	3.926/434(M+Na)
I-189	3-$OC(=O)NHCH_3$	CH_3	Cl	Cl	
I-190	-	$NHC(=O)CH_3$	Cl	Cl	3.344/413 (M+23)
I-191	-	NH_2	Cl	Cl	3.341/437 (M+88)
I-192	-	4-OCH_3-C_6H_4	Cl	Cl	4.179/462 (M+22)
I-193	-	4-SCH_3-C_6H_4	Cl	Cl	4.361/477 (M+21)
I-194	-	CH_3	Cl	I	3.837/461 (M+22)
I-195	-	CH_3	F	I	3.657/445 (M+22)
I-196	3,4-$[C(O)CH_3]_2$	CH_3	Cl	Cl	3.661 / 486 (M+Na)
I-197	3-F,4-Cl	CH_3	F	Cl	3.904/406(M+Na)
I-198	3,4-OH_2	CH_3	Cl	Cl	3.082/402(M+Na)
I-199	3-I	CH_3	F	Cl	3.924/480(M+Na)
I-200	4-I	CH_3	F	Cl	3.979/480(M+Na)
I-201	3-CN	CH_3	Cl	Cl	3.730/395(M+Na)
I-202	4-CN	CH_3	Cl	Cl	3.713/373 (M+H)
I-203	3-CN	CH_3	F	Cl	3.553/357 (M+H)
I-204	4-CN	CH_3	F	Cl	3.567/357 (M+H)
I-205	3-CN	CH_3	Cl	Cl	isomer 1: 3.730 / 395 (M+Na)
I-206	4-CN	CH_3	Cl	Cl	isomer 1: 3.567 / 357 (M+H)
I-207	-		Cl	Cl	4.133 / 475 (M+22)
I-208	-	4-OCF_3-C_6H_4	Cl	Cl	4.454/516 (M+23)
I-209	4-F		F	Cl	3.612 / 420 (M+1)
I-210	-	3-$N(CH_3)_2$-C_6H_4	Cl	Cl	3.772 / 453

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-211	-	$CH_2N(CH_3)$-$COOCH_3$	Cl	Cl	3.737/357(M+Na)
I-212	-	$CH_2CON(CH_3)_2$	Cl	Cl	3.496/419 (M+H)
I-213	4-C≡ CH	CH_3	F	Cl	3.696/378(M+Na)
I-214	3-C≡ CH	CH_3	Cl	Cl	3.874/395(M+Na)
I-215	-	$NHCOOCH_3$	Cl	Cl	4.190/316 (M-91)
I-216	-	$CH_2CH_2OCH_3$	Cl	Cl	3.737/392 (M+H)
I-217	-	$(CH_2)_3OCH_3$	Cl	Cl	3.861/406 (M+H)
I-218	3-C≡ CH	CH_3	F	Cl	3.778/356 (M+H)
I-219	-	$^+N(CH_3)_3$-$(CH_2)_{11}CH_3$	Cl	Cl	
I-220	-	$^+N(CH_3)_2Bz$-$(CH_2)_{15}CH_3$	Cl	Cl	
I-221	-	$^+N(CH_3)_2Bz$-$(CH_2)_{13}CH_3$	Cl	Cl	
I-222	-	$^+N(CH_3)_2Bz$-$(CH_2)_7CH_3$	Cl	Cl	
I-223	-	$^+N(C_4H_9)_3Bz$	Cl	Cl	
I-224	3-Cl,4-F	CH_3	F	Cl	3.926/406(M+Na)
I-225	3,4-Br_2	CH_3	Cl	Cl	
I-226	-	C_6H_5	Cl	Cl	
I-227	-	CH_2C≡ CBr	Cl	Cl	4.040/474(M+Na)
I-228	3,4,5-$(OCH_3)_3$	CH_3	Cl	Cl	3.564/440 (M+H)
I-229	3,4-CN_2	CH_3	Cl	Cl	3.769/409 (M+11)
I-230	3-CN	CH_2C≡ CH	Cl	Cl	3.684/420(M+Na)
I-231	3-CN	CH_2C≡ CH	F	Cl	3.540/381 (M+H)
I-232	4-CN	CH_2C≡ CH	Cl	Cl	3.692/420(M+Na)
I-233	4-CN	CH_2C≡ CH	F	Cl	3.547/381 (M+H)
I-234	3-CN	CH_2OCH_3	Cl	Cl	3.531/425(M+Na)
I-235	3-CN	CH_2OCH_3	F	Cl	3.372/409(M+Na)
I-236	3-CN	$CH_2N(CH_3)$-$COOCH_3$	Cl	Cl	
I-237	3-F	$CH_2N(CH_3)$-$COOCH_3$	Cl	Cl	3.695/475(M+Na)
I-238	-	$CH_2C(=O)$-$OCH_2CH_2OCH_3$	Cl	Cl	3.712 / 472 (M+Na)
I-239	-	$CH_2C(=O)$-OCH_2COOCH_3	Cl	Cl	3.705 / 486 (M+Na)
I-240	-	CH_2CH_2Br	Cl	Cl	3.964/463 (M+22)
I-241	-	$CH=CH_2$	Cl	Cl	

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-242	-		Cl	Cl	3.722 / 468 (M+22)
I-243	-	$CH_2N(CH_3)$-$COOCH_3$	F	Cl	3.596/441(M+Na)
I-244	-	$CH_2N(CH_3)$-CO-$O(CH_2)_2OCH_3$	Cl	Cl	3.730 / 501 (M+Na)
I-245	-	CH_2OCH_3	F	Cl	3.631/384 (M+23)
I-246	-	$^+N[CH(CH_3)_2]_2$	Cl	Cl	
I-247	-	$^+NH_3O(CH_2)_2O$-$(CH_2)_2OH$	Cl	Cl	
I-248	4-CN	CH_2OCH_3	F	Cl	
I-249	4-CN	CH_2OCH_3	Cl	Cl	
I-250	3-CH_2CN	CH_3	Cl	Cl	3.479/409(M+Na)
I-251	-	CH_2CH_2CH-$[CH(CN)C_6H_5]$-$(3,5\text{-}Cl_2\text{-}C_6H_3)$	Cl	Cl	
I-252	3,4-F_2	H	Cl	Cl	3.318/392 (M+22)
I-253	3-$COOCH_3$	CH_3	Cl	Cl	3.794/374 (M-30)
I-254	3,4,5-F_3	H	Cl	Cl	3.618 / 387
I-255	3-CN	H	Cl	Cl	3.112/381(M+Na)
I-256	4-CN	H	Cl	Cl	3.217/381(M+Na)
I-257	4-F	H	F	Cl	3.264/336 (M+1)
I-258	-	$CF_2C\equiv C$-$Si(CH(CH_3)_2)_3$	Cl	Cl	
I-259	3-F	H	F	Cl	3.259 / 335
I-260	3-CHF_2	H	Cl	Cl	
I-261	3-CN	H	F	Cl	3.123/343 (M+H)
I-262	3-CHF_2	CH_3	Cl	Cl	
I-263	-		Cl	Cl	3.824 / 686 (M+Na)
I-264	-	$C(OC_2H_5)=CH_2$	Cl	Cl	
I-265	-	CH_2CH_2COOH	Cl	Cl	
I-266	-	CH_3	Cl	CN	3.390/362 (M+23)
I-267	-	Li^+	Cl	Cl	
I-268	3-CN,4-F	$CH_2C\equiv CH$	F	Cl	2.934/409 (M+11)

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-269	-	$CH_2C\equiv CH$	CN	Cl	3.773/385 (M+23)
I-270	4-OH	H	Cl	Cl	3.000/372(M+Na)
I-271	4-CF_3	CH_3	Cl	Cl	4.045/417 (M+H)
I-272	-		Cl	Cl	3.420 / 405 (M+H)
I-273	-	CH_2OCOCH_3	Cl	Cl	3.677/428(M+Na)
I-274	-	CH_2Cl	Cl	Cl	3.842/404(M+Na)
I-275	-		Cl	Cl	4.207 / 486 (M+H)
I-276	3-CN	CH_3	Cl	CF_3	
I-277	3-F	CH_3	Cl	CF_3	3.961/400 (M+H)
I-278	3-F	$CH_2C\equiv CH$	Cl	CF_3	
I-279	4-F	CH_3	CF_3	Cl	3.972/400 (M+H)
I-280	4-F	$CH_2C\equiv CH$	CF_3	Cl	4.155/424 (M+H)
I-281	-	$CH_2N[CH(CH_3)_2]CO$-OCH_3	Cl	Cl	4.160 / 406 (M+Na)
I-282	-	CH_2CH_2CN	Cl	Cl	
I-283	-	$CH_2C(=O)$-$OCH_2C_6H_5$	Cl	Cl	
I-284	-		Cl	Cl	3.422 / 447 (M+18)
I-285	-		Cl	Cl	3.893 / 435 (M+23)
I-286	-	$CH_2CH_2SCH_3$	Cl	Cl	3.889/429 (M+23)
I-287	-	CH_2-$P(=O)(OC_2H_5)_2$	Cl	Cl	3.664/484 (M+1)
I-288	-		Cl	Cl	4.272 / 451 (M+23)
I-289	-	CH_2-$P(=O)(OCH_3)_2$	Cl	Cl	3.951/369 (M-87)
I-290	-	CH_3	CF_3	Cl	
I-291	-	$CH_2C\equiv CH$	CF_3	Cl	3.808/407 (M+H)
I-292	2-Br	CH_3	Cl	Cl	4.093/450 (M+23)
I-293	3-CN	H	CF_3	Cl	3.347/375 (M-18)
I-294	4-F	CH_3	CF_3	Br	
I-295	3-F	H	Cl	CF_3	3.439/367 (M-18)
I-296	4-F	H	Cl	CF_3	3.446/367 (M-18)
I-297	3-F,4-Cl	CH_3	F	F	3.660/368 (M+H)

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-298	-	H	Cl	CF_3	3.531/350 (M-18)
I-299	3-F,4-Cl	CH_3	CF_3	Cl	
I-300	4-F	CH_3	F	CN	3.248/382 (M+42)
I-301	3-CN	CH_2CHF_2	Cl	Cl	3.646/424 (M+H)
I-302	4-Cl	CH_2CHF_2	F	F	3.864/400 (M+H)
I-303	3-F,4-Cl	CH_2CHF_2	F	F	3.880/418 (M+H)
I-304	4-F	CH_3	I	CF_3	
I-305	4-F	CH_2CHF_2	Cl	Cl	
I-306	3,4,5-F_3	H	F	Br	
I-307	3,5-F_2	H	F	Br	
I-308	-	H	Br	F	
I-309	3-F	H	Br	F	
I-310	4-F	CH_3	F	Br	
I-311	4-Cl	CH_3	F	Br	
I-312	3,4-F_2	CH_3	Br	F	
I-313	3,4,5-F_3	CH_3	Br	F	
I-314	3,5-F_2	CH_3	F	Br	
I-315	-	CH_3	F	Br	
I-316	3-F	CH_3	Br	F	
I-317	3-F	CH_3	Cl	F	3.704 / 349
I-318	3,4-F_2	CH_3	Cl	F	3.766 / 367
I-319	3,5-F_2	CH_3	F	Cl	3.774 / 367
I-320	3,4,5-F_3	CH_3	F	Cl	3.862 / 385
I-321	4-Cl	CH_3	Cl	F	3.712 / 366
I-322	3-CN	CH_3	Br	F	
I-323	4-F	CH_2CHF_2	F	Br	
I-324	4-F	CH_2OCH_3	F	Br	
I-325	3-CN	H	Br	F	
I-326	4-Cl	H	Br	F	
I-327	3,4-F_2	H	Br	F	
I-328	4-F	H	Br	F	
I-329	4-Cl	CH_2CHF_2	Br	F	
I-330	3,4-F_2	CH_2CHF_2	Br	F	
I-331	3,4,5-F_3	CH_2CHF_2	Br	F	
I-332	3,5-F_2	CH_2CHF_2	Br	F	
I-333	-	CH_2CHF_2	Br	F	
I-334	3-F	CH_2CHF_2	Br	F	

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-335	4-Cl	$CH_2C\equiv CH$	Br	F	
I-336	3,4-F_2	$CH_2C\equiv CH$	Br	F	
I-337	3,4,5-F_3	$CH_2C\equiv CH$	Br	F	
I-338	3,5-F_2	$CH_2C\equiv CH$	Br	F	
I-339	3-F	$CH_2C\equiv CH$	Br	F	
I-340	4-Cl	CH_2OCH_3	F	Br	
I-341	3,4-F_2	CH_2OCH_3	F	Br	
I-342	3,4,5-F_3	CH_2OCH_3	F	Br	
I-343	3,5-F_2	CH_2OCH_3	F	Br	
I-344	-	CH_2OCH_3	F	Br	
I-345	3-F	CH_2OCH_3	F	Br	
I-346	3,4,5-F_3	H	F	Cl	3.457/372 (M+1)
I-347	3,4-F_2	H	F	Cl	3.353 / 353
I-348	3,5-F_2	H	F	Cl	3.238/354 (M+1)
I-349	4-Cl	H	F	Cl	3.472/351 (M-1)
I-350	3-I	CH_3	F	F	
I-351	3-F	CH_3	F	F	
I-352	3,4,5-F_3	CH_3	F	F	
I-353	3,5-F_2	CH_3	F	F	
I-354	3,4-F_2	CH_3	F	F	
I-355	3-CN	CH_3	F	F	
I-356	4-I	CH_3	F	F	
I-357	4-Cl	H	F	F	
I-358	3-I	H	F	F	
I-359	3-F	H	F	F	
I-360	4-F	CH_2CHF_2	F	F	
I-361	3,4,5-F_3	H	F	F	
I-362	3,4-F_2	H	F	F	
I-363	4-I	H	F	F	
I-364	3-CN	CH_2CHF_2	Br	F	
I-365	3-CN	CH_2CN	Br	F	
I-366	3,5-F_2	H	F	F	
I-367	-	CH_2CHF_2	F	F	
I-368	-	CH_2OCH_3	F	F	
I-369	4-F	CH_3	F	I	
I-370	4-F	H	F	I	
I-371	-	H	F	Cl	2.982 / 392 (M-1)

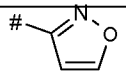
No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-372	3,4-F_2	CH_2OCH_3	F	Cl	3.519/419 (M+22)
I-373	-	CH_2CHF_2	F	Cl	3.929 / 382 (M+1)
I-374	-	CH_2CF_3	Cl	F	4.098 / 400 (M+1)
I-375	4-F	CH_2CHF_2	F	Cl	3.951/296(M-103)
I-376	4-F	CH_2CF_3	F	Cl	4.114 / 418 (M+1)
I-377	3,4-F_2	$CH_2C\equiv CH$	Cl	F	3.956 / 392 (M+1)
I-378	3,4-F_2	CH_2CHF_2	Cl	F	3.748/408 (M-9)
I-379	3,4-F_2	CH_2CF_3	Cl	F	4.168/330 (M-5)
I-380	3-F	$CH_2C\equiv CH$	Cl	F	3.895/374 (M+1)
I-381	3-F	CH_2OCH_3	F	Cl	3.828/380 (M+1)
I-382	3-F	CH_2CHF_2	F	Cl	3.946/296(M-103)
I-383	3-F	CH_2CF_3	F	Cl	4.090 / 418 (M+1)
I-384	3,5-F_2	CH_2OCH_3	F	Cl	3.901/415 (M+18)
I-385	3,5-F_2	CH_2CHF_2	F	Cl	4.001/296(M-126)
I-386	3,5-F_2	CH_2CF_3	F	Cl	4.151/334(M-101)
I-387	3,4,5-F_3	$CH_2C\equiv CH$	Cl	F	4.042 / 410 (M+1)
I-388	3,4,5-F_3	CH_2OCH_3	F	Cl	3.989 / 416 (M+1)
I-389	3,4,5-F_3	CH_2CHF_2	F	Cl	4.077/296(M-139)
I-390	3,4,5-F_3	CH_2CF_3	F	Cl	4.238/480 (M+17)
I-391	4-Cl	$CH_2C\equiv CH$	Cl	F	4.080 / 390
I-392	4-Cl	CH_2OCH_3	F	Cl	4.018 / 396
I-393	4-Cl	CH_2CHF_2	F	Cl	4.118/334 (M-82)
I-394	4-Cl	CH_2CF_3	F	Cl	4.259 / 434
I-395	3-CN	CH_2CF_3	F	Cl	3.902/296(M-128)
I-396	3,5-F_2	$CH_2C\equiv CH$	Cl	F	3.961/296 (M-95)
I-397	3-CN	CH_2OCH_3	F	Br	
I-398	3-CN	H	F	F	
I-399	3-CN	CH_2CHF_2	F	Cl	3.712 / 407 (M+1)
I-400	2,4-F_2	CH_3	F	Br	3.870/411 (M-1)
I-401	2,5-F_2	CH_3	F	Br	3.823/413 (M+1)
I-402	3-F,4-Cl	CH_3	F	Br	4.045/429 (M+1)
I-403	3-F,5-Cl	CH_3	F	Br	4.065/429 (M+1)
I-404	3-F,5-Br	H	F	Br	3.624/437 (M+23)
I-405	2-F,4-F	H	F	Br	3.388/399 (M+1)
I-406	3-F,5-Br	CH_3	F	Br	4.050/473 (M)
I-407	3-F,-Br	H	F	Br	3.604/481 (M+22)
I-408	3-F,-Cl	H	F	Br	3.557/437 (M+23)

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-409	2,4-F_2	H	Cl	Cl	3.441/372 (M+1)
I-410	-	H	Cl	Br	3.876/465 (M+21)
I-411	3-Cl	CH_3	F	F	3.684/350 (M+1)
I-412	3,5-Cl_2	CH_3	F	F	3.918/3874 (M)
I-413	3-F	CH_3	Br	Br	3.924/455 (M)
I-414	4-F	CH_3	Br	Br	3.932/455 (M)
I-415	3-Cl	CH_3	F	Br	3.866/411 (M+1)
I-416	3-F	$CH_2CHC(CH_3)_2$	F	Br	4.258/137 (M-311)
I-417	3-F	$CH_2CH_2CHC(CH_3)_2$	F	Br	4.366/486 (M-24)
I-418	3-F	$CH_2CH_2CH_2CHC(CH_3)_2$	F	Br	4.323/472 (M+24)
I-419	3-CN	CH_3	Cl	Br	3.691/418 (M+1)
I-420	4-F	CH_3	Cl	Br	3.918/411 (M+1)
I-421	4-F	H	Cl	Br	3.612/397 (M+1)
I-422	3-CN	H	Cl	Br	3.384/405 (M+2)
I-423	4-F	CH_3	CF_3	F	3.951/406 (M+23)
I-424	3,4-F_2	CH_3	CF_3	F	3.999/401 (M+1)
I-425	3,4,5-F_3	CH_3	CF_3	F	4.084/442 (M+23)
I-426	3,5-F_2	CH_3	CF_3	F	4.010/402 (M+1)
I-427	4-F	H	CF_3	F	4.498/369 (M+1)
I-428	3,4-F_2	H	CF_3	F	3.566/388 (M+1)
I-429	3,4,5-F_3	H	CF_3	F	3.672/406 (M+1)
I-430	3,5-F_2	H	CF_3	F	3.477/370 (M-17)
I-431	3-F	CH_3	CF_3	F	3.469/384 (M+1)
I-432	3-CN	CH_3	CF_3	F	3.695/391 (M+1)
I-433	-	H	CF_3	F	3.341 / 333 (M-18)
I-434	3-F	H	CF_3	F	3.641/352 (M-17)
I-435	3-CN	H	CF_3	F	3.462/415 (M+39)
I-436	3-F	$CH_2CH_2CHCH_2$	F	Br	4.266/457 (M+23)
I-437	3-F	CH_2CHCH_2	F	Br	4.101/420 (M)
I-438	4-F	CH_3	$OCHF_2$	Cl	3.889/420 (M+23)
I-439	3-F	CH_3	$OCHF_2$	Cl	3.877/399 (M+1)
I-440	-	CH_3	$OCHF_2$	Cl	3.791/402 (M+23)
I-441	4-F	H	$OCHF_2$	Cl	3.409/415 (M+23)
I-442	4-F	H	$OCHF_2$	Cl	3.458 /.385 (M+1)

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-443	-	H	$OCHF_2$	Cl	3.379/366 (M+1)
I-444	4-F	CH_2Cl	F	Br	4.004/362 (M-66)
I-445	4-F	CH_2CF_3	F	Br	4.132/364 (M-98)
I-446	4-F	CH_2-P(=O)$(OCH_3)_2$	F	Br	3.483/504 (M+2)
I-447	4-F	CH_2CN	F	Br	3.757/419 (M)
I-448	4-F	Bz	F	Br	4.316/492 (M+22)
I-449	4-F	CH_2CHCH_2	F	Br	4.104/420 (M)
I-450	4-F	CH_2CH_3	F	Br	4.043/410 (M+2)
I-451	4-F	$CH_2CH_2CH_3$	F	Br	4.218/422 (M)
I-452	4-F	CH_2CHCl_2	F	Br	4.215/500 (M+23)
I-453	4-F	4-Cl-C_6H_5	F	Br	4.435/512 (M+22)
I-454	4-F	2-Cl-C_6H_5	F	Br	4.348/514 (M+24)
I-455	4-F	3-Cl-C_6H_5	F	Br	4.430/514 (M+24)
I-456	4-F	2,4-Cl_2-C_6H_5	F	Br	4.607/547 (M+22)
I-457	4-F	2,6-Cl_2-C_6H_5	F	Br	4.481/547 (M+22)
I-458	4-F	2,5-Cl_2-C_6H_5	F	Br	4.554/548 (M+23)
I-459	4-F	3,5-Cl_2-C_6H_5	F	Br	4.697/547 (M+22)
I-460	4-F	2,4,6-Cl_3-C_6H_5	F	Br	4.776/581 (M+22)
I-461	4-F	4-F-C_6H_5	F	Br	4.258/496 (M+22)
I-462	4-F	2-F-C_6H_5	F	Br	4.240/498 (M+24)
I-463	4-F	3-F- C_6H_5	F	Br	4.274/498 (M+24)
I-464	4-F	2,4-F_2-C_6H_5	F	Br	4.367/514 (M+22)
I-465	4-F	2-F-Bz	F	Br	4.39/488 (M)
I-466	4-F	2,5-F_2-Bz	F	Br	4.309/508 (M+2)
I-467	4-F	2,4-F_2-Bz	F	Br	4.333/506 (M)
I-468	4-F	2,6-F_2-Bz	F	Br	4.283/506 (M)
I-469	4-F	2,3-F_2-Bz	F	Br	4.320/506 (M)
I-470	4-F	CH_2CCl_3	F	Br	4.421/511 (M+23)
I-471	3,4-F_2	CH_3	F	Br	4.117/430 (M+2)
I-472	-	CH_3	F	CN	3.464/323 (M+1)
I-473	3,4-F_2	CH_3	F	CN	3.567/359 (M+1)
I-474	3-F	CH_3	F	CN	3.497/341 (M+1)
I-475	3,5-F_2	CH_3	F	CN	3.575/359 (M+1)
I-476	3,4,5-F_3	CH_3	F	CN	3.687/378 (M+2)
I-477	3-CN	CH_3	F	CN	3.270/348 (M+1)
I-478	3-F	CH_3	Cl	Br	4.064/412 (M+2)
I-479	3,4-F_2	H	Cl	Br	3.655/415 (M+1)

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-480	4-F	CH_3	$OCHF_2$	F	3.735/382 (M+1)
I-481	3-F	CH_3	$OCHF_2$	F	3.638/382 (M+1)
I-482	-	CH_3	$OCHF_2$	F	3.612/364 (M+1)
I-483	4-F	H	$OCHF_2$	F	3.313/396 (M+2)
I-484	3-F	H	$OCHF_2$	F	3.340/368 (M+1)
I-485	-	H	$OCHF_2$	F	3.208/350 (M+1)
I-486	4-F	CH_3	OCF_3	Br	3.461/460 (M)
I-487	3,5-F_2	CH_3	Cl	Br	4.000/430 (M+2)
I-488	3,4,5-F_3	CH_3	Cl	Br	4.086/447 (M+1)
I-489	3,5-F_2	H	Cl	Br	3.645/102 (M-312)
I-490	3,4,5-F_3	H	Cl	Br	3.748/102 (M-330)
I-491	3-F	H	Cl	Br	3.567/397 (M+1)
I-492	4-F	CH_3	CN	Br	3.566/368 (M-33)
I-493	2-$OCHF_2$	CH_3	F	Br	3.901/443 (M+1)
I-494	2-$OCHF_2$	H	F	Br	3.480/430 (M+1)
I-495	3-$OCHF_2$	CH_3	F	Br	3.899/464 (M+23)
I-496	3-$OCHF_2$	H	F	Br	3.405/451 (M+23)
I-497	4-$OCHF_2$	CH_3	F	Br	3.834/465 (M+23)
I-498	4-F	CH_3	CF_3	CF_3	4.126/434 (M+1)
I-499	-	CH_3	CF_3	CF_3	4.120/438 (M+23)
I-500	4-$OCHF_2$	CH_3	F	Br	3.410/451 (M+23)
I-501	-	C_6H_5	F	Cl	4.161/411 (M+18)
I-502	-	2-F-C_6H_5	F	Cl	4.173/429 (M+18)
I-503	-	3-Cl-C_6H_5	F	Cl	4.30/445 (M+17)
I-504	-	4-F-C_6H_5	F	Cl	4.190/429 (M+18)
I-505	3-F	C_6H_5	F	Cl	4.169/429 (M+18)
I-506	3-F	2-F-C_6H_5	F	Cl	4.179/447 (M+18)
I-507	3-F	3-Cl-C_6H_5	F	Cl	4.371/463 (M+17)
I-508	3-F	4-F-C_6H_5	F	Cl	4.197/447 (M+18)
I-509	4-F	C_6H_5	F	Cl	4.177/429 (M+18)
I-510	4-F	2-F-C_6H_5	F	Cl	4.186/447 (M+18)
I-511	4-F	3-Cl-C_6H_5	F	Cl	4.375/463 (M+17)
I-512	4-F	4-F-C_6H_5	F	Cl	4.203/447 (M+18)
I-513	3,5-F_2	C_6H_5	F	Cl	4.222/447 (M+18)
I-514	3,5-F_2	2-F-C_6H_5	F	Cl	4.229/465 (M+18)

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-515	3,5-F_2	3-Cl-C_6H_5	F	Cl	4.411/481 (M+17)
I-516	3,5-F_2	4-F-C_6H_5	F	Cl	4.244/465 (M+18)
I-517	4-CH_3	CH_3	F	Cl	3.976/346 (M+1)
I-518	4-CF_3	CH_3	F	Cl	3.758/536 (M+137)
I-519	4-CF_3	CH_3	F	Br	4.105/444 (M)
I-520	4-CF_3	CH_3	F	F	3.924/384 (M+1)
I-521	4-F	H	OCF_3	Br	3.752/446 (M)
I-522	-	CF_3	Cl	Cl	4.552/412 (M+23)
I-523	-	H	CN	F	3.028/331 (M+23)
I-524	4-CH_3	CH_3	F	F	4.147/320 (M-150)
I-525	4-CH_3	CH_3	F	Br	3.940/437 (M+21)
I-526	-	CH_3	CN	Br	3.643/383 (M)
I-527	4-F	CH_3	F	CH_3	3.731/330 (M+1)
I-528	4-F	CH_3	OCH_3	F	3.626/346 (M+1)
I-529	-	CH_3	F	OCH_3	3.560/328 (M+1)
I-530	-	CH_3	CN	OCF_3	3.769/389 (M+1)
I-531	4-F	CH_3	CN	OCF_3	3.789/407 (M+23)
I-532	-	CH_3	F	NO_2	3.571/360 (M+18)
I-533	4-F	CH_3	CN	Cl	3.606/324 (M-32)
I-534	3,5-F_2	CH_3	CN	Br	3.714/386 (M-86)
I-535	3,4,5-F_3	CH_3	CN	Br	3.801/438 (M+1)
I-536	-	CH_3	$OCHF_2$	Br	3.861/424 (M)
I-537	4-F	CH_3	$OCHF_2$	Br	3.894/465 (M+23)
I-538	3-F	CH_3	$OCHF_2$	Br	3.908/465 (M+23)
I-539	-	CH_3	$OCHF_2$	CN	3.540/393 (M+23)
I-540	4-F	CH_3	$OCHF_2$	CN	3.573/411 (M+23)
I-541	-	2-CO_2H-C_6H_5	Cl	Cl	3.896/436 (M-18)
I-542	-	2-CO_2CH_3-C_6H_5	Cl	Cl	4.307/436 (M-32)
I-543	-	2-F-C_6H_5	Cl	Cl	4.315/316 (M-112)
I-544	-	2-CF_3-C_6H_5	Cl	Cl	4.470/316 (M-162)
I-545	-	2-OCF_3-C_6H_5	Cl	Cl	4.520/316 (M-178)
I-546	-	2-OCH_3-C_6H_5	Cl	Cl	4.290/440 (M)

No.	R_n	R^1	R^2	R^3	phys. data (HPLC: RT[min]/m/z)
I-547	-	2-OCH_2CH_3-C_6H_5	Cl	Cl	4.431/454 (M)
I-548	-	2-CH_3-C_6H_5	Cl	Cl	4.435/424 (M)
I-549	-	2-CN-C_6H_5	Cl	Cl	4.164/435 (M)
I-550	-	2-Cl-C_6H_5	Cl	Cl	4.430/316 (M-128)
I-551	-	2-$COCH_3$-C_6H_5	Cl	Cl	4.185/454 (M+2)
I-552	-	2-NO_2-C_6H_5	Cl	Cl	4.225/316 (M-139)
I-553	-	3-F-C_6H_5	Cl	Cl	4.355/316 (M-112)
I-554	-	3,5-F_2-C_6H_5	Cl	Cl	4.442/316 (M-130)
I-555	-		Cl	Cl	3.963/401 (M)
I-556	-	H	F	Cl	3.321/340 (M+23)
I-557	-	H	F	$OCHF_2$	3.302/373 (M+23)

Unless indicated otherwise, the compounds are present as a racemate

Bz denotes a benzyl group ($CH_2C_6H_5$)

# denotes the bond through which the group R^1 is attached

Use examples

The herbicidal activity of the compounds of the formula I was demonstrated by the following greenhouse experiments:

The culture containers used were plastic flowerpots containing loamy sand with approximately 3.0% of humus as the substrate. The seeds of the test plants were sown separately for each species.

For the pre-emergence treatment, the active compounds, which had been suspended or emulsified in water, were applied directly after sowing by means of finely distributing nozzles. The containers were irrigated gently to promote germination and growth and subsequently covered with transparent plastic hoods until the plants had rooted. This cover caused uniform germination of the test plants, unless this has been impaired by the active compounds.

For the post-emergence treatment, the test plants were first grown to a height of 3 to 15 cm, depending on the plant habit, and then treated with the active compounds which

had been suspended or emulsified in water. For this purpose, the test plants were either sown directly and grown in the same containers, or they were first grown separately as seedlings and transplanted into the test containers a few days prior to treatment.

Depending on the species, the plants were kept at 10 - 25°C or 20 - 35°C. The test period extended over 2 to 4 weeks. During this time, the plants were tended, and their response to the individual treatments was evaluated.

Evaluation was carried out using a scale from 0 to 100. 100 means no emergence of the plants, or complete destruction of at least the aerial moieties, and 0 means no damage, or normal course of growth. A good herbicidal activity is given at values of at least 70 and a very good herbicidal activity is given at values of at least 85.

The plants used in the greenhouse experiments belonged to the following species:

Bayer code	Scientific name	Common name
ALOMY	Alopecurus myosuroides	Blackgrass
AMARE	Amaranthus retroflexus	Carelessweed
AVEFA	Avena fatua	spring wild-oat
GALAP	Galium aparine	Goosegrass
POLCO	Fallopia convolvulus	Bearbine
SETVI	Setaria viridis	Green foxtail

Applied by the post-emergence method at an application rate of 0.125 kg/ha, the compounds I-431, I-434, I-444, I-445, I-446, I-502, I-504, I-505, I-506, I-507, I-508, I-509, I-510, I-513, I-514, I-523, I-526 and I-555 showed very good herbicidal activity and the compounds I-430, I-432, I-433, I-447, I-484, I-492 and I-501 showed good herbicidal activity against ALOMY.

Applied by the post-emergence method at an application rate of 0.250 kg/ha, the compounds I-411, I-472, I-473, I-474, I-475, I-476, I-480 and I-512 showed very good herbicidal activity and the compounds I-422, I-424 and I-426 showed good herbicidal activity against ALOMY.

Applied by the post-emergence method at an application rate of 0.125 kg/ha, the compounds I-434, I-444, I-445, I-448, I-449, I-451, I-453, I-454, I-455, I-462, I-463, I-464, I-465, I-466, I-467, I-468, I-547 and I-555 showed very good herbicidal activity and the compounds I-429, I-433, I-469 showed good herbicidal activity against AMARE.

Applied by the post-emergence method at an application rate of 0.250 kg/ha, the

compounds I-411, I-422 and I-424 showed very good herbicidal activity against AMARE.

Applied by the post-emergence method at an application rate of 0.125 kg/ha, the compounds I-430, I-431, I-432, I-446, I-447, I-448, I-449, I-451, I-453, I-454, I-455, I-459, I-462, I-463, I-464, I-465, I-466, I-467, I-468, I-469, I-523, I-526 and I-547 showed very good herbicidal activity against AVEFA.

Applied by the post-emergence method at an application rate of 0.250 kg/ha, the compounds I-472, I-473, I-474, I-475, I-476, I-481 and I-482 showed very good herbicidal activity against AVEFA.

Applied by the post-emergence method at an application rate of 0.125 kg/ha, the compounds I-427, I-428, I-429, I-492, I-502, I-505, I-506, I-507, I-508, I-509, I-510, I-513 and I-514 showed very good herbicidal activity and the compounds I-501 and I-504 showed good herbicidal activity against GALAP.

Applied by the post-emergence method at an application rate of 0.250 kg/ha, the compounds I-426 and I-512 showed very good herbicidal activity against GALAP.

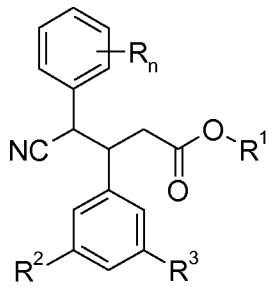
Applied by the post-emergence method at an application rate of 0.125 kg/ha, the compounds I-427 and I-533 showed very good herbicidal activity and the compound I-428 showed good herbicidal activity against POLCO.

Applied by the post-emergence method at an application rate of 0.125 kg/ha, the compounds I-459 and I-484 showed very good herbicidal activity and the compound I-533 showed good herbicidal activity against SETVI.

Applied by the post-emergence method at an application rate of 0.250 kg/ha, the compound I-480 showed very good herbicidal activity against SETVI.

Claims

1. A cyanobutyrate compound of the formula I



I

in which the variables have the following meaning:

R is halogen, cyano, nitro, C_1-C_4-alkyl, C_1-C_4-haloalkyl, C_2-C_6-alkenyl, C_2-C_6-alkynyl, C_1-C_4-alkoxy, C_1-C_4-alkylthio or Z-(tri-C_1-C_4-alkyl)silyl;

R^1 is hydrogen, Z-CN, C_1-C_8-alkyl, Z-C_3-C_6-cycloalkyl, C_1-C_8-haloalkyl, C_1-C_4-alkoxy-C_1-C_4-alkyl, C_1-C_4-alkylthio-C_1-C_4-alkyl, C_3-C_8-alkenyl, Z-C_3-C_6-cycloalkenyl, C_3-C_8-alkynyl, NR^iR^{ii}, Z-(tri-C_1-C_4-alkyl)silyl, Z-N=$C(R^a)_2$, Z-$C(R^a)$=NR^a, Z-C(=T)-R^a, Z-P(=T)$(R^a)_2$, Z-phenyl or a 3- to 7-membered monocyclic or 9- or 10-membered bicyclic saturated, unsaturated or aromatic heterocycle which contains 1, 2, 3 or 4 heteroatoms selected from the group consisting of O, N and S, which may carry carbonyl or thiocarbonyl groups and which is attached via carbon or nitrogen,

T is O or S;

R^a is hydrogen, TH, C_1-C_8-alkyl, C_1-C_4-haloalkyl, Z-C_3-C_6-cycloalkyl, C_2-C_8-alkenyl, Z-C_5-C_6-cycloalkenyl, C_2-C_8-alkynyl, Z-C_1-C_6-alkoxy, Z-C_1-C_4-haloalkoxy, Z-C_3-C_8-alkenyloxy, Z-C_3-C_8-alkynyloxy, Z-C_1-C_4-alkylthio, Z-C_1-C_4-haloalkylthio, Z-C_3-C_8-alkenylthio, Z-C_3-C_8-alkynylthio, NR^iR^{ii}, C_1-C_6-alkylsulfonyl, oxy-C_1-C_3-alkyleneoxy, Z-C(=T)-R^A, Z-T-C(=T)-R^A, Z-(tri-C_1-C_4-alkyl)silyl, Z-phenyl, Z-phenoxy, Z-phenylamino or a 3- to 7-membered monocyclic or 9- or 10-membered bicyclic heterocycle which contains 1, 2, 3 or 4 heteroatoms selected from the group consisting of O, N and S, where the cyclic groups are unsubstituted or may carry 1, 2, 3 or 4 groups R^b and/or carbonyl or thiocarbonyl groups;

R^i, R^{ii} independently of one another are hydrogen, C_1-C_8-alkyl, C_1-C_4-haloalkyl, C_3-C_8-alkenyl, C_3-C_8-alkynyl, Z-C_3-C_6-cycloalkyl, Z-C_1-C_8-alkoxy, Z-C_1-C_8-alkylthio, Z-C_1-C_8-haloalkoxy, Z-C_1-C_8-haloalkylthio, Z-C(=T)-R^A, where R^A is TH, C_1-C_4-alkyl, C_1-C_4-alkoxy or C_1-C_4-alkylthio;

R^i and R^{ii} together with the nitrogen atom to which they are attached may also form a 3- to 7-membered monocyclic or 9- or 10-membered bicyclic heterocycle which contains 1, 2, 3 or 4 heteroatoms selected

from the group consisting of O, N and S and which may carry carbonyl or thiocarbonyl groups;

R^b independently of one another are Z-CN, Z-TH, Z-NO_2, Z-halogen, C_1-C_8-alkyl, C_1-C_4-haloalkyl, C_2-C_8-alkenyl, C_2-C_8-alkynyl, Z-C_1-C_8-alkoxy, Z-C_1-C_8-haloalkoxy, Z-C_1-C_8-alkylthio, Z-C_1-C_8-haloalkylthio, Z-C_3-C_{10}-cycloalkyl, T-Z-C_3-C_{10}-cycloalkyl, Z-C(=T)-R^a, NR^iR^{ii}, Z-(tri-C_1-C_4-alkyl)silyl, Z-phenyl and $S(O)_mR^{bb}$, where R^{bb} is C_1-C_8-alkyl or C_1-C_6-haloalkyl; m is 0, 1 or 2;

R^b together with the group R^b attached to the adjacent carbon atom may also form a five- or six-membered saturated or partially or fully unsaturated ring which, in addition to carbon atoms, may contain 1, 2 or 3 heteroatoms selected from the group consisting of O, N and S;

Z is a covalent bond or C_1-C_8-alkylene;

n is 0, 1, 2, 3, 4 or 5;

R^2,R^3 independently of one another are halogen, cyano, nitro, C_1-C_4-alkyl, C_1-C_4-alkoxy, $S(O)_mR^{bb}$, NR^iR^{ii}, C_1-C_4-haloalkyl or C_1-C_4-haloalkoxy;

where in the groups R and R^1 and their subsubstituents, the carbon chains and/or the cyclic groups may be partially or fully substituted by groups R^a and/or R^b,

or an N-oxide or an agriculturally suitable salt thereof;

with the proviso that R^2 and R^3 are not both chlorine if the index n is 0 or 1 and R is halogen or R_n is 3,5-Cl_2 and R^1 is C_1-C_4-alkyl or H; and

R^2 and R^3 are not both fluorine if the index n is 1 and R is halogen and R^1 is C_1-C_4-alkyl.

2. The compound of the formula I according to claim 1 in which R^2 and R^3 are different and R is Cl and/or F.

3. The compound of the formula I according to claim 1 in which R^2 and R^3 are different.

4. The compound of the formula I according to any of claims 1 to 3 in which R^2 is fluorine.

5. The compound of the formula I according to any of claims 1 to 4 in which the combination of R^2 and R^3 is selected from the group consisting of 3-F,5-CH_3, 3-F,5-$OCHF_2$, 3-F,5-Cl, 3-F,5-Br, 3,5-F_2 and 3,5-Cl_2.

6. The compound of the formula I according to any of claims 1 to 5 in which R is chlorine, fluorine, cyano or nitro and the index n has the value 1 or 2.

7. The compound of the formula I according to any of claims 1 to 6 in which R_n is 3-F; 3,4-F_2; 3,5-F_2; 3,4,5-F_3; 3-F,4-Cl; 3-Cl,4-F; 4-Cl; 4-F; 3-Cl or 3-CN.

8. The compound of the formula I according to any of claims 1 to 6 in which R is fluorine or cyano and the index n has the value 1.

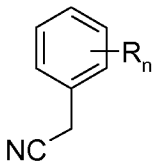
9. The compound of the formula I according to any of claims 1 to 8 in which R^1 is H, CH_3, C_2H_5, CH_2CN, C_1-C_2-haloalkyl, CH_2OCH_3 or $CH_2C\equiv CH$.

10. The compound of the formula I according to any of claims 1 to 9 in which R^1 is H.

11. The compound of the formula I according to any of claims 1 to 9 in which R^1 is CH_3.

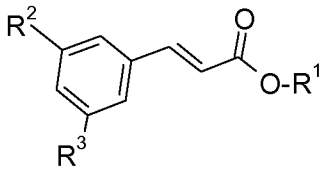
12. The compound of the formula I according to any of claims 1 to 9 in which R^1 is $CH_2C\equiv CH$.

13. A process for preparing the compound of the formula I as defined in any of claims 1 to 12 comprising reacting a phenylacetonitrile derivative of the formula II



II

with a cinnamate derivative of the formula III



III

under basic conditions.

14. A composition comprising a herbicidally effective amount of at least one cyanobutyrate compound of the formula I or an agriculturally suitable salt thereof as defined in any of claims 1 to 12 and auxiliaries customary for formulating crop protection agents.

15. A method for controlling unwanted vegetation which comprises allowing a herbicidally effective amount of at least one cyanobutyrate compound of the formula I or of an agriculturally suitable salt thereof as defined in any of claims 1 to 12 to act on plants, their seed and/or their habitat.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2011/051730

A. CLASSIFICATION OF SUBJECT MATTER
INV. C07C255/41 C07C255/44 C07C255/55 C07C255/57 C07C271/44
C07H15/04 A01N37/34 A01N43/00 C07C313/12 C07C323/20
C07C323/47 C07C323/62 C07D205/08 C07D213/68 C07D231/12

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)
C07C C07H C07D C07F A01N A01P

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)

EPO-Internal, WPI Data, CHEM ABS Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 270 830 A1 (AMERICAN CYANAMID CO [US]) 15 June 1988 (1988-06-15) cited in the application table IV: the first entry on page 47 table Ia: entries 8-11 on page 15 table III: both entries on page 43 table IV: the second entry on page 46 claim 1 abstract	1-15

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family 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" 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

"T" 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.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
17 February 2011	23/02/2011

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Fitz, Wolfgang

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2011/051730

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 266 725 A1 (AMERICAN CYANAMID CO [US]) 11 May 1988 (1988-05-11) cited in the application page 10; table I; compounds 16,20 page 12; table II; compound 38 page 15; table III; compound 62 page 51; table IV; compound 88 claim 1 abstract page 8, line 41 - line 44 -----	1-15
X	EP 0 005 341 A2 (AMERICAN CYANAMID CO [US]) 14 November 1979 (1979-11-14) cited in the application page 7, line 9 - line 10 page 8, line 26 - line 27 table II: the second entry on page 61 claim 1 abstract -----	1-15

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
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