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(54) Title
CYANOPHENYL PYRROLES

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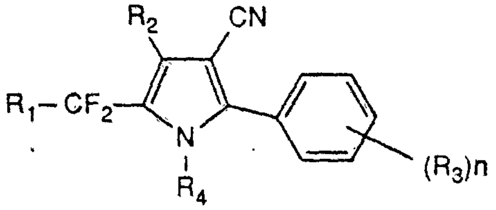
(57) Claim

1. A compound of the formula



(I),

in which n is the number 1, 2, 3, 4 or 5, where, if n is greater than 1, the radicals R_3 are identical or different; R_1 is halo-C_1-C_9alkyl; R_2 is halogen; R_3 is hydrogen, cyano, nitro, halogen, C_1-C_4alkyl, C_1-C_4alkoxy, C_1-C_4alkylthio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkyl, halo-C_1-C_4alkoxy, halo-C_1-C_4alkylthio, halo-C_1-C_4alkanesulfinyl or halo-C_1-C_4alkanesulfonyl, and/or two substituents R_3 which are bonded to adjacent C atoms of the phenyl ring together are a bridge selected from the group of bridges comprising -O-CH_2-O-, -O-CH_2-CH_2-O-, -O-CH_2-CH_2-, -O-CH_2-CH_2-CH_2- and -CH=CH-CH=CH-, unsubstituted or substituted by halogen, C_1-C_4alkyl, C_1-C_4alkoxy or halo-C_1-C_4alkyl; and R_4 is hydrogen, C_1-C_4alkyl, C_3-C_4alkenyl, halo-C_3-C_4alkenyl, C_3-C_4alkynyl, cyano, C_1-C_4alkylthio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkanesulfonyl, di-C_1-C_4alkylaminosulfonyl, di-C_1-C_4alkylaminocarbonyl or a C_1-C_4alkyl group which is substituted by one or more substituents selected from the group comprising halogen, C_1-C_4alkoxy, hydroxyl, C_1-C_4alkylthio, C_2-C_6alkoxyalkoxy, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, C_1-C_4alkylcarbonyl, C_1-C_4alkoxycarbonyl, C_1-C_4alkylcarbonyloxy, cyano, C_2-C_4alkenylcarbonyloxy, phenyl, phenoxy, phenylthio, benzyloxy, benzoyloxy, benzoyl, phenylsulfinyl and phenylsulfonyl, where the phenyl rings contained in these

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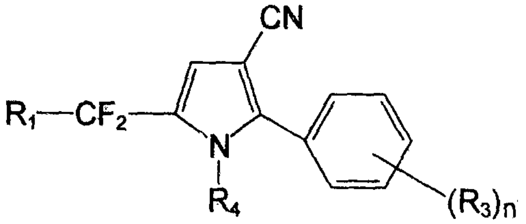
phenyl, phenoxy, phenylthio, benzyloxy, benzoyloxy, benzoyl, phenylsulfinyl and phenylsulfonyl substituents can in each case be substituted by halogen, C_1-C_4alkyl, C_1-C_4alkoxy, halo-C_1-C_4alkyl, halo-C_1-C_4alkoxy, C_1-C_4alkylthio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkylthio, halo-C_1-C_4-alkanesulfinyl, halo-C_1-C_4-alkanesulfonyl, cyano or nitro,
in free form or in salt form.

13. A process for the preparation of a compound of the formula I, defined in claim 1, in free form or in salt form, which comprises

a) introducing the halogen substituent R_2 into the 4-position of the pyrrole ring of a compound of the formula



(II),

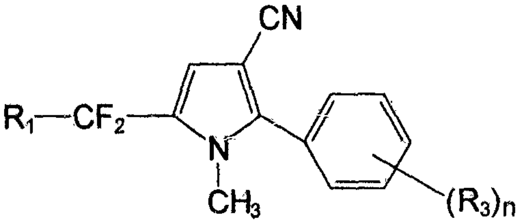
in which R_1, R_3, R_4, and n are as defined in claim 1, or into a salt thereof, by reaction with a halogenating agent, optionally in the presence of a base, or

b) to prepare a compound of the formula I in which R_4 is other than hydrogen, or a salt of such a compound, reacting a compound of the formula I in which R_4 is hydrogen, or a salt of such a compound, with a compound of the formula

X-R_4 (III),

in which R_4 is as defined in claim 1, with the exception of hydrogen, and X is a leaving group, or, if appropriate, with a salt thereof, optionally in the presence of a base, or

c) to prepare a compound of the formula I, in which R_4 is methyl or a group R_2CH_2-, or a salt of such a compound, reacting, optionally in the presence of a base, optionally in the presence of a radical initiator, a compound of the formula



(VII),

in which R_1, R_3 and n are as defined in claim 1, or a salt thereof, with a halogenating agent

and, in each, if desired, converting the compound of the formula I so prepared, in free form or in salt form, into a different compound of the formula I, resolving an isomer mixture, and isolating the isomer desired and/or converting a free compound of the formula I into a salt, or converting a salt of a compound of the formula I into the free compound of the formula I or into a different salt.

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PATENTS ACT 1990

COMPLETE SPECIFICATION

FOR A STANDARD PATENT

ORIGINAL

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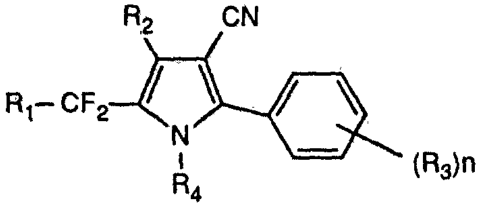
Invention Title:	Cyanophenylpyrroles

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

PI/5-18772/A

Cyanophenylpyrroles

The invention relates to compounds of the formula



(I),

in which n is the number 1, 2, 3, 4 or 5, where, if n is greater than 1, the radicals R_3 are identical or different; R_1 is halo-C_1-C_9alkyl; R_2 is halogen; R_3 is hydrogen, cyano, nitro, halogen, C_1-C_4alkyl, C_1-C_4alkoxy, C_1-C_4alkylthio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkyl, halo-C_1-C_4alkoxy, halo-C_1-C_4alkylthio, halo-C_1-C_4alkanesulfinyl or halo-C_1-C_4alkanesulfonyl, and/or two substituents R_3 which are bonded to adjacent C atoms of the phenyl ring together are a bridge selected from the group of bridges comprising $-O-CH_2-O-$, $-O-CH_2-CH_2-O-$, $-O-CH_2-CH_2-$, $-O-CH_2-CH_2-CH_2-$ and $-CH=CH-CH=CH-$, unsubstituted or substituted by halogen, C_1-C_4alkyl, C_1-C_4alkoxy or halo-C_1-C_4alkyl; and R_4 is hydrogen, C_1-C_4alkyl, C_3-C_4alkenyl, halo-C_3-C_4alkenyl, C_3-C_4alkynyl, cyano, C_1-C_4alkylthio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkanesulfonyl, di-C_1-C_4alkylaminosulfonyl, di-C_1-C_4alkylaminocarbonyl or a C_1-C_4alkyl group which is substituted by one or more substituents selected from the group comprising halogen, C_1-C_4alkoxy, hydroxyl, C_1-C_4alkylthio, C_2-C_6alkoxyalkoxy, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, C_1-C_4alkylcarbonyl, C_1-C_4alkoxycarbonyl, C_1-C_4alkylcarbonyloxy, cyano, C_2-C_4alkenylcarbonyloxy, phenyl, phenoxy, phenylthio, benzyloxy, benzoyloxy, benzoyl, phenylsulfinyl and phenylsulfonyl, where the phenyl rings contained in these phenyl, phenoxy, phenylthio, benzyloxy, benzoyloxy, benzoyl, phenylsulfinyl and phenylsulfonyl substituents can in each case be substituted by halogen, C_1-C_4alkyl, C_1-C_4alkoxy, halo-C_1-C_4alkyl, halo-C_1-C_4alkoxy, C_1-C_4alkylthio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkylthio, halo-C_1-C_4-alkanesulfinyl, halo-C_1-C_4-alkanesulfonyl, cyano or nitro, in free form or in salt form, to a process for the preparation and to the use of these compounds, to pesticides whose active ingredient is selected from amongst these compounds in free form or in agrochemically usable salt form, to a process for the preparation and to the use of these compositions, to plant propagation material treated with these compositions, to a method for controlling pests, to intermediates, in free form or in salt form, for the preparation of these compounds, and to a process for the preparation and the use of these intermediates.

EP-A-0 347 488, EP-A-0 372 263, EP-A-0 426 948, EP-A-0 434 940 and EP-A-0 492 171 propose cyanophenylpyrrole derivatives as acaricidal and insecticidal active ingredients in pesticides. However, the biological properties of the compounds described in these publications are not entirely satisfactory in the pest control sector, and this is why it has been found necessary to provide other compounds having pest-controlling properties, in particular for the control of insects and of representatives from the order of the Acarina, and this object is achieved according to the invention by providing the present compounds I which, surprisingly, are additionally also suitable for controlling phytopathogenic fungi.

Compounds I which have at least one basic centre can form for example acid addition salts. These acid addition salts are formed, for example, with strong inorganic acids such as mineral acids, for example perchloric acid, sulfuric acid, nitric acid, nitrous acid, a phosphoric acid or a hydrohalic acid, with strong organic carboxylic acids, for example unsubstituted or halogen-substituted C_1-C_4alkanecarboxylic acids, for example acetic acid, or unsaturated or saturated dicarboxylic acids, for example oxalic acid, malonic acid, succinic acid, maleic acid, fumaric acid or phthalic acid, or hydroxycarboxylic acids, for example ascorbic acid, lactic acid, malic acid, tartaric acid or citric acid, or benzoic acid, or with organic sulfonic acids for example C_1-C_4alkane- or arylsulfonic acids which are unsubstituted or substituted for example by halogen, for example methane- or p-toluenesulfonic acid. Furthermore, compounds I having at least one acidic group can form salts with bases. Suitable salts with bases are, for example, metal salts such as alkali metal salts or alkaline earth metal salts, for example sodium salts, potassium salts or magnesium salts, or salts with ammonia or an organic amine, such as morpholine, piperidine, pyrrolidine, a mono-, di- or tri-lower alkylamine, for example ethyl-, diethyl-, triethyl-, or dimethylpropylamine, or a mono-, di- or trihydroxy lower-alkylamine, for example mono-, di- or triethanolamine. Furthermore, corresponding internal salts may possibly be formed. Preferred within the scope of the invention are agrochemically advantageous salts; however, the invention also comprises salts which are disadvantageous for agrochemical purposes, for example salts which are toxic to bees or fish and which are employed, for example, for the isolation or purification of free compounds I or their agrochemically usable salts. Due to the close relation between the compounds I in free form and in the form of their salts, the free compounds I, or salts thereof, which are mentioned hereinbefore and hereinafter are also to be understood as meaning analogously and expediently, where appropriate, the corresponding salts, or the free compounds I, respectively.

Unless otherwise defined, the general terms used hereinbefore and hereinafter have the meanings listed hereinafter.

Halogen - as a group per se and as structural element of other groups and compounds such as haloalkyl, haloalkenyl, haloalkoxy, haloalkylthio, haloalkanesulfinyl and haloalkanesulfonyl - is fluorine, chlorine, bromine or iodine, in particular fluorine, chlorine or bromine, especially fluorine or chlorine, in particular fluorine. Halogen R_2 is especially chlorine or bromine, in particular bromine.

Carbon-containing groups and compounds contain, unless otherwise defined, in each case 1 up to and including 9, preferably 1 up to and including 6 but 1 up to and including 4 being preferred, in particular 1 or 2, carbon atoms.

Alkyl - as a group per se and as structural element of other groups and compounds such as haloalkyl, alkoxy, alkoxyalkoxy, haloalkoxy, alkylthio, haloalkylthio, alkanesulfinyl, haloalkanesulfinyl, alkanesulfonyl, haloalkanesulfonyl, dialkylaminosulfonyl, dialkylaminocarbonyl, alkylcarbonyl, alkoxycarbonyl and alkylcarbonyloxy - is either straight-chain, i.e. methyl, ethyl, propyl, butyl, pentyl, hexyl, heptyl, octyl or nonyl, or branched, for example isopropyl, isobutyl, sec-butyl, tert-butyl, isopentyl, neopentyl or isooctyl, in each case with due consideration of the number of carbon atoms contained in each individual case in the particular group or compound.

Alkenyl, haloalkenyl, alkenylcarbonyloxy and alkynyl are straight-chain or branched and contain in each case two or, preferably, one unsaturated carbon bond(s). The double bonds or triple bonds of these substituents are separated from the remainder of the compound I, preferably by at least one saturated carbon atom. The following may be mentioned by way of example: allyl, methallyl, but-2-enyl, but-3-enyl, allylcarbonyloxy, propargyl, but-2-ynyl and but-3-ynyl.

Halogen-substituted carbon-containing groups and compounds such as haloalkyl, haloalkenyl, haloalkoxy, haloalkylthio, haloalkanesulfinyl and haloalkanesulfonyl can be partially halogenated or perhalogenated, where, in the case of multiple halogenation, the halogen substituents can be identical or different. Examples of haloalkyl - as a group per se and as structural element of other groups and compounds such as haloalkyl, haloalkoxy, haloalkylthio, haloalkanesulfinyl and haloalkanesulfonyl - are methyl, mono- to trisubstituted by fluorine, chlorine and/or bromine, such as CHF_2 or CF_3; ethyl, mono- to pentasubstituted by fluorine, chlorine and/or bromine, such as CH_2CF_3, CF_2CF_3, CF_2CCl_3, CF_2CHCl_2, CF_2CHF_2, CF_2CFCl_2, CF_2CHBr_2, CF_2CHClF, CF_2CHBrF or $CClFCHClF$; propyl or isopropyl, mono- to heptasubstituted by fluorine, chlorine and/or bromine, such as $CH_2CHBrCH_2Br$, CF_2CHFCF_3, $CH_2CF_2CF_3$, $CF_2CF_2CF_3$ or $CH(CF_3)_2$; and butyl or one of the isomers thereof, mono- to nonasubstituted by fluorine, chlorine and/or bromine, such as $CF(CF_3)CHFCF_3$, $CF_2(CF_2)_2CF_3$ or $CH_2(CF_2)_2CF_3$. Haloalkyl R_1 is preferably exclusively fluorinated, in particular perfluorinated. Examples of haloalkenyl are 2-chloroprop-1-en-3-yl, 2,3-dichloroprop-1-en-3-yl and 2,3-dibromoprop-1-en-3-yl.

The two alkyl groups in dialkylaminosulfonyl and dialkylaminocarbonyl substituents can in each case be identical or different. The following may be mentioned by way of example: $-SO_2-N(CH_3)_2$, $-SO_2-N(C_2H_5)_2$, $-SO_2-N(CH_3)C_2H_5$, $-SO_2-N(C_3H_7)_2$, $-SO_2-N(C_4H_9)_2$, $-CO-N(CH_3)_2$, $-CO-N(C_2H_5)_2$, $-CO-N(CH_3)C_2H_5$, $-CO-N(C_3H_7)_2$, $-CO-N(CH_3)C_3H_7$-i, $-CO-N(CH_3)C_3H_7$-n, $-CO-N(CH_3)C_4H_9$-n, $-CO-N(C_4H_9)_2$ and $-CO-N(C_2H_5)C_3H_7$-n.

In alkoxyalkoxy, an alkoxy group bonded to the remainder of the compound I is substituted by a further alkoxy group, it being possible for both carbon chains independently of one another to be straight-chain or branched; examples are methoxymethoxy, methoxyethoxy, ethoxyethoxy, ethoxymethoxy, propoxymethoxy, methoxypropoxy, butoxymethoxy and propoxyethoxy.

If the abovementioned bridges which are formed by two substituents R_3 together are substituted, then either of the hydrogen atoms in the unsubstituted skeleton is replaced by an alkyl, alkoxy or haloalkyl substituent, or one or more than one, in particular all, of the hydrogen atoms in the unsubstituted skeleton are replaced by identical or different halogen. Examples of such substituted bridges are $-O-CF_2-O-$, for example, 2,3-$O-CF_2-O-$ or 3,4-$O-CF_2-O-$, $-O-CF_2-CH_2-O-$, $-O-CF_2-CF_2-O-$, $-O-CHF-CHCl-$, $-O-CH-CH(CH_3)-CH_2-$, 2,3-$CH=CH-CH=CH-$, 3,4-$CH=CH-CH=CH-$, $-CH=C(OCH_3)-CH=CH-$ and $-CH=CH-C(CF_3)=CH-$.

The alkyl skeleton in the substituted alkyl groups R_4 has preferably two or, in particular, one of the abovementioned substituents; however, if it is a halogen-substituted alkyl group R_4, then one or more than one, for example all, of the hydrogen atoms in the unsubstituted alkyl skeleton are replaced by identical or different halogen. If the substituted alkyl groups R_4 contain a substituted phenyl ring, then this phenyl ring has preferably two or, in particular, one of the abovementioned substituents; however, if it is a halogen-substituted phenyl ring, then 1, 2, 3, 4 or all of the phenyl hydrogen atoms can be replaced by identical or different halogen. The alkyl skeleton is preferably a methyl group.

The following are preferred embodiments within the scope of the invention:

(1) A compound of the formula I in which R_1 is perfluoro-C_1-C_6alkyl, in particular perfluoro-C_1-C_4alkyl, especially CF_3 or C_2F_5, in particular CF_3;

(2) A compound of the formula I in which R_2 is chlorine or bromine, in particular bromine;

(3) A compound of the formula I in which n is the number 1, 2, 3, 4 or 5, in particular 1, 2 or 3 or, if all radicals R_3 simultaneously are halogen, 1, 2, 3, 4, or 5, where, if n is greater than 1, the radicals R_3 are identical or different, and R_3 is halogen, halo-C_1-C_4alkyl, halo-C_1-C_4alkoxy, cyano, C_1-C_4alkyl, C_1-C_4alkoxy, C_1-C_4alkylthio or C_1-C_4alkanesulfonyl, and/or two substitutents R_3 bonded to adjacent C atoms of the phenyl ring together are -O-C(halogen)$_2$-O-, in particular -O-CF_2-O-, or -CH=CH-CH=CH-,
in particular the number 1 or 2, where, if n is 2, the radicals R_3 are identical, and R_3 is halogen, halo-C_1-C_4alkyl or halo-C_1-C_4alkoxy, or two substituents R_3 bonded to two adjacent C atoms of the phenyl ring together are -CH=CH-CH=CH-, especially the number 1 and R_3 is trifluoromethyl, trifluoromethoxy or chlorine;

(4) A compound of the formula I in which R_4 is hydrogen, C_1-C_4alkyl, C_3-C_4alkenyl, halo-C_3-C_4alkenyl, C_3-C_4alkynyl, cyano, C_1-C_4alkylthio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkanesulfonyl, di-C_1-C_4alkylaminosulfonyl, di-C_1-C_4alkylaminocarbonyl or a C_1-C_4alkyl group substituted by one or more substituents selected from the group comprising halogen, C_1-C_4alkoxy, hydroxyl, C_1-C_4alkylthio, C_2-C_6alkoxyalkoxy, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, C_1-C_4alkylcarbonyl, C_1-C_4alkoxycarbonyl, C_1-C_4alkylcarbonyloxy, cyano, C_2-C_4alkenylcarbonyloxy, phenyl, phenoxy, phenylthio, benzyloxy, benzoyloxy, benzoyl,

phenylsulfinyl and phenylsulfonyl, where the phenyl rings in these phenyl, phenoxy, phenylthio, benzyloxy, benzoyloxy, benzoyl, phenylsulfinyl and phenylsulfonyl substituents can in each case be substituted by halogen, C_1-C_4alkyl, C_1-C_4alkoxy, halo-C_1-C_4alkyl, halo-C_1-C_4alkoxy, C_1-C_4alkylthio, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkylthio, halo-C_1-C_4alkanesulfinyl, halo-C_1-C_4alkanesulfonyl, cyano or nitro, in particular hydrogen, C_1-C_4alkyl, C_3-C_4alkynyl, C_1-C_4alkanesulfonyl, di-C_1-C_4alkylaminosulfonyl, or a C_1-C_4alkyl group substituted by C_1-C_4alkoxy, C_1-C_4alkylthio, C_2-C_6alkoxyalkoxy, C_1-C_4alkanesulfonyl, C_1-C_4alkylcarbonyl, C_1-C_4alkoxycarbonyl, cyano, monohalophenyl or phenoxy,
especially hydrogen, C_1-C_4alkyl, or a C_1-C_4alkyl group substituted by C_1-C_4alkoxy or C_2-C_6alkoxyalkoxy, in particular hydrogen, C_1-C_4alkyl or C_2-C_6alkoxyalkyl,
preferably methyl, methoxymethyl or ethoxymethyl,
in particular hydrogen or ethoxymethyl;

(5) A compound of the formula I in which R_1 is perfluoro-C_1-C_6alkyl, R_2 is chlorine or bromine, R_3 is halogen, halo-C_1-C_4alkyl, Halo-C_1-C_4alkoxy, cyano, C_1-C_4alkyl, C_1-C_4alkoxy, C_1-C_4alkylthio or C_1-C_4alkanesulfonyl, R_4 is hydrogen, C_1-C_4alkyl or C_2-C_6alkoxyalkyl, and n is the number one, two or three, where n can be the number one, two, three, four or five if all radicals R_3 simultaneously are halogen;

(6) A compound of the formula I in which R_1 is trifluoromethyl or pentafluoroethyl, R_2 is bromine, R_3 is fluorine, chlorine, trifluoromethyl, fluoro-C_1-C_3alkoxy, methylthio, cyano or C_1-C_3alkoxy, R_4 is hydrogen, methyl, methoxymethyl or ethoxymethyl, and n is the number one, two or three;

(7) A compound of the formula I in which R_1 is trifluoromethyl, R_2 is chlorine or bromine, R_3 is fluorine, chlorine, trifluoromethyl, fluoro-C_1-C_3alkoxy, methylthio, cyano or C_1-C_3alkoxy, R_4 is hydrogen, methyl, methoxymethyl or ethoxymethyl, and n is the number one, two or three;

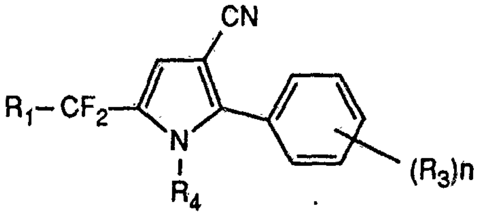
(8) A compound of the formula I in which n is the number 1, R_1 is trifluoromethyl or pentafluoroethyl, R_2 is bromine, R_3 is trifluoromethyl, trifluoromethoxy or chlorine, and R_4 is hydrogen or ethoxymethyl.

Particularly preferred within the scope of the invention are the compounds of the formula I mentioned in Examples H2, H3, H5 and H6.

Individually preferred compounds within the scope of the invention are
(a) 4-Bromo-2-(4-chlorophenyl)-3-cyano-5-heptafluoropropyl-pyrrole,
(b) 4-Bromo-2-(4-chlorophenyl)-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-pyrrole,
(c) 4-Bromo-2-(4-chlorophenyl)-3-cyano-1-ethoxymethyl-5-heptafluoropropyl-pyrrole,
(d) 4-Chloro-2-(4-chlorophenyl)-3-cyano-5-heptafluoropropyl-pyrrole,
(e) 4-Chloro-2-(4-chlorophenyl)-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-pyrrole,
(f) 4-Chloro-2-(4-chlorophenyl)-3-cyano-1-ethoxymethyl-5-heptafluoropropyl-pyrrole,
(g) 4-Bromo-3-cyano-5-pentafluoroethyl-2-(4-trifluoromethylphenyl)-pyrrole,
(h) 4-Chloro-3-cyano-5-pentafluoroethyl-2-(4-trifluoromethylphenyl)-pyrrole,
(i) 4-Bromo-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-2-(4-trifluoromethylphenyl)-pyrrole,
(j) 4-Chloro-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-2-(4-trifluoromethylphenyl)-pyrrole,
(k) 4-Bromo-3-cyano-5-pentafluoroethyl-2-(4-trifluoromethoxyphenyl)-pyrrole,
(l) 4-Chloro-3-cyano-5-pentafluoroethyl-2-(4-trifluoromethoxyphenyl)-pyrrole,
(m) 4-Bromo-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-2-(4-trifluoromethoxyphenyl)-pyrrole and
(n) 4-Chloro-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-2-(4-trifluoromethoxyphenyl)-pyrrole.

A further subject of the invention is the process for the preparation of the compounds of the formula I, in free form or in salt form, which comprises, for example

a) introducing the halogen substituent R_2 into the 4-position of the pyrrole ring of a compound of the formula



(II),

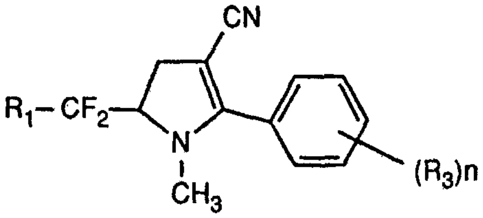
in which R_1, R_3, R_4 and n are as defined in formula I, or into a salt thereof, by reaction with a halogenating agent, preferably in the presence of a base, or

b) to prepare a compound of the formula I in which R_4 is other than hydrogen, or a salt thereof, reacting a compound of the formula I which can be obtained, for example, according to variant a), and in which R_4 is hydrogen, or a salt of such a compound, with a compound of the formula

$$X\text{-}R_4 \qquad\qquad \text{(III)}$$

which is known or which can be prepared analogously to corresponding known compounds and in which R_4 is as defined in formula I, with the exception of hydrogen, and X is a leaving group, or, if appropriate, with a salt thereof, preferably in the presence of a base, or

c) to prepare a compound of the formula I, in which R_4 is methyl or a group R_2CH_2-, or a salt thereof, reacting, optionally in the presence of a base, optionally in the presence of a radical initiator, a compound of the formula



(VII),

in which R_1, R_3 and n are as defined in formula I, or a salt thereof, with a halogenating agent

and, in each case, if desired, converting a compound of the formula I, in free form or in salt form, which can be obtained according to the process or by a different route, into a different compound of the formula I, resolving an isomer mixture which can be obtained according to the process, and isolating the isomer desired and/or converting a free compound of the formula I which can be obtained according to the process into a salt, or a salt of a compound of the formula I which can be obtained according to the process into the free compound of the formula I or into a different salt.

What has been said hereinbefore for salts of compounds I applies analogously to starting materials listed hereinbefore and hereinafter with regard to salts thereof.

The reactions described hereinbefore and hereinafter are carried out in a manner known per se, for example in the absence or, conventionally, in the presence of a suitable solvent or diluent or a mixture of these, the process being carried out, depending on the circumstances, with cooling, at room temperature or with heating, for example in a temperature range of approximately -80°C to the boiling point of the reaction medium, preferably from approximately -20°C to approximately +150°C, and, if required, in a sealed container, under pressure, under an inert gas atmosphere and/or under anhydrous conditions. Particularly advantageous reaction conditions can be found in the examples.

The starting materials listed hereinbefore and hereinafter which are used for the preparation of the compounds I, in free form or in salt form, are known or can be prepared by methods known per se, for example according to the instructions below.

Variant a):
Examples of suitable halogenating agents are elemental halogens, such as elemental chlorine, bromine or iodine, sulfuryl halides such as sulfuryl chloride or sulfuryl bromide, or N-halosuccinimides, such as N-chlorosuccinimide or N-bromosuccinimide.

Examples of suitable bases for facilitating the reaction with the halogenating agent are hydroxides, hydrides, amides, alkanolates, acetates, carbonates, dialkylamides or alkylsilylamides of alkali metals or alkaline earth metals, or alkylamines, alkylenediamines, free or N-alkylated, saturated or unsaturated cycloalkylamines, basic heterocycles, ammonium hydroxides and carbocyclic amines. Examples which may be mentioned are sodium hydroxide, sodium hydride, sodium amide, sodium methanolate, sodium acetate, sodium carbonate, potassium tert-butanolate, potassium hydroxide, potassium carbonate, potassium hydride, lithium diisopropylamide, potassium bis(trimethylsilyl)amide, calcium hydride, triethylamine, diisopropylethylamine, triethylenediamine, cyclohexylamine, N-cyclohexyl-N,N-dimethylamine, N,N-diethylaniline, pyridine, 4-(N,N-dimethylamino)pyridine, quinuclidine, N-methylmorpholine, benzyltrimethylammonium hydroxide and 1,5-diazabicyclo[5.4.0]undec-5-ene (DBU).

The reactants can be reacted with each other as such, i.e. without the addition of a solvent or diluent, for example in the melt. However, the addition of an inert solvent or diluent or a mixture of these is advantageous in most cases. Examples of such solvents or diluents which may be mentioned are: aromatic, aliphatic and alicyclic hydrocarbons and halohydrocarbons such as benzene, toluene, xylene, mesitylene, tetralin, chlorobenzene, dichlorobenzene, bromobenzene, petroleum ether, hexane, cyclohexane, dichloromethane, trichloromethane, tetrachloromethane, dichloroethane, trichloroethene or tetrachloroethene; esters such as ethyl acetate; ethers such as diethyl ether, diisopropyl ether, dibutyl ether, tert-butyl methyl ether, ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, ethylene glycol dimethyl ether, dimethoxydiethyl ether, tetrahydrofuran or dioxane; ketones such as acetone, methyl ethyl ketone or methyl isobutyl ketone; alcohols, such as methanol, ethanol, propanol, isopropanol, butanol, ethylene glycol or glycerol; amides such as N,N-dimethylformamide, N,N-diethylformamide, N,N-dimethylacetamide, N-methylpyrrolidone or hexamethylphosphoric triamide; nitriles such as acetonitrile; sulfoxides such as dimethyl sulfoxide; and acids, for example strong organic carboxylic acids such as unsubstituted or, for example, halogen-substituted, C_1-C_4-alkanecarboxylic acids, for example formic acid, acetic acid or propionic acid. If the reaction is carried out in the presence of a base, bases which are employed in excess such as triethylamine, pyridine, N-methylmorpholine or N,N-diethylaniline, can also act as solvents or diluents.

The reaction is advantageously carried out in a temperature range of from approximately 0°C to approximately +180°C, preferably from approximately +10°C to approximately +130°C, in many cases in the range between room temperature and the reflux temperature of the reaction mixture. In most cases, the temperature of the reaction mixture rises automatically by the heat of reaction which is liberated. To complete the halogenation reaction, it is advantageous to finish by briefly heating the reaction mixture to reflux point.

Variant b:

Examples of suitable leaving groups X in the compounds III are hydroxyl, C_1-C_8alkoxy, halo-C_1-C_8alkoxy, C_1-C_8alkanoyloxy, mercapto, C_1-C_8alkylthio, halo-C_1-C_8alkylthio, C_1-C_8alkanesulfonyloxy, halo-C_1-C_8alkanesulfonyloxy, benzenesulfonyloxy, toluenesulfonyloxy and halogen.

Examples of suitable bases for facilitating the elimination of HX are of the type described in variant a).

The reactants can be reacted with each other as such, i.e. without the addition of a solvent or diluent, for example in the melt. However, the addition of an inert solvent or diluent or a mixture of these is advantageous in most cases. Examples of such solvents or diluents which may be mentioned are: aromatic, aliphatic and alicyclic hydrocarbons and halohydrocarbons such as benzene, toluene, xylene, mesitylene, tetralin, chlorobenzene, dichlorobenzene, bromobenzene, petroleum ether, hexane, cyclohexane, dichloromethane, trichloromethane, tetrachloromethane, dichloroethane, trichloroethene or tetrachloroethene; esters such as ethyl acetate; ethers such as diethyl ether, diisopropyl ether, dibutyl ether, tert-butyl methyl ether, ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, ethylene glycol dimethyl ether, dimethoxydiethyl ether, tetrahydrofuran or dioxane; ketones such as acetone, methyl ethyl ketone or methyl isobutyl ketone; alcohols, such as methanol, ethanol, propanol, isopropanol, butanol, ethylene glycol or glycerol; amides such as N,N-dimethylformamide, N,N-diethylformamide, N,N-dimethylacetamide, N-methylpyrrolidone or hexamethylphosphoric triamide; nitriles such as acetonitrile; and sulfoxides such as dimethyl sulfoxides. If the reaction is carried out in the presence of a base, bases which are employed in excess such as triethylamine, pyridine, N-methylmorpholine or N,N-diethylaniline, can also act as solvents or diluents.

The reaction is advantageously carried out in a temperature range of from approximately 0°C to approximately +180°C, preferably from approximately +10°C to approximately +130°C, in many cases in the range between room temperature and the reflux temperature of the reaction mixture.

Variant c):

Examples of suitable halogenating agents are of the type described in variant a).

Examples of suitable bases for facilitating the reaction with the halogenating agent are of the type described in variant a).

Examples of suitable radical initiators are peroxo compounds, such as dibenzoyl peroxide or di-tert.-butyl peroxide, azo compounds, such as 2,2'-azobisisobutyronitrile, and light.

The reactants can be reacted with each other as such, i.e. without the addition of a solvent or diluent, for example in the melt. However, the addition of an inert solvent or diluent or a mixture of these is advantageous in most cases. Examples of such solvents or diluents which may be mentioned are of the type described in variant b).

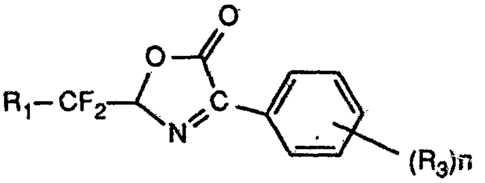
The reaction is advantageously carried out in a temperature range of from approximately 0°C to approximately +180°C, preferably from approximately +10°C to approximately +130°C, in many cases in the range between room temperature and the reflux temperature of the reaction mixture.

It is possible to prepare different products of the formula I when variant c) is carried out, depending on the number of equivalents of halogenating agent used. At least two equivalents of halogenating agent have to be used. By means of the first equivalent the dihydropyrrole derivative VII is oxidised and the corresponding pyrrole intermediate is thus obtained ("compound A"). By means of the second equivalent of halogenating agent the halogen substituent R_2 is introduced into the 4-position of the pyrrole ring of the compound A, thus yielding the corresponding compound I, wherein R_4 is methyl ("compound B"). In order to obtain the corresponding compound I, wherein R_4 is a group R_2CH_2-, i. e. in order to mono-halogenate the methyl group R_4 of the compound B, a third equivalent of halogenating agent is needed. The term "equivalent" is to be understood in this paragraph in such a way, that it comprises not only the exact amount corresponding to the exact equivalent, but also a certain range, for example approximately 10%, above and below this exact amount. It is possible to isolate the compound A and/or the compound B, but preferably the reaction is carried out in the form of a one-pot-reaction without isolation of the compounds A and B. It is also possible to add the whole amount of halogenating agent at the beginning of the reaction, but preferably the halogenating agent is added portionwise, for example in several portions of one equivalent each. The mono-halogenation of the methyl group R_4 of compound B is preferably carried out in the presence of a radical initiator.

The compounds II which are used as educts in process variant a), in free form or in salt form, are novel and also form a subject of the invention. Particularly preferred compounds of the formula II within the scope of the invention are those mentioned in Examples H1 and H6.

A further subject of the invention is the process for the preparation of the compounds of the formula II, in free form or in salt form, which comprises, for example,

d) to prepare a compound of the formula II in which R_4 is hydrogen, or a salt thereof, reacting a compound of the formula



(IV)

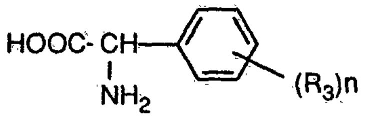
in which R_1, R_3 and n are as defined in formula I, or a salt of such a compound, with 2-chloroacrylonitrile, preferably in a high-boiling polar solvent such as nitromethane, and at the reflux temperature of the reaction mixture, or

e) to prepare a compound of the formula II in which R_4 is other than hydrogen, or a salt of such a compound, reacting a compound of the formula II which can be obtained, for example, in accordance with variant d), and in which R_4 is hydrogen, or a salt of such a compound, preferably in the presence of a base, for example of the type given in the case of variant a), with a compound of the formula

$$X\text{-}R_4 \qquad \text{(III)}$$

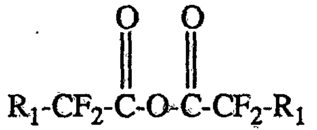
which is known or which can be prepared analogously to corresponding known compounds and in which R_4 is as defined in formula I with the exception of hydrogen, and X is a leaving group, for example of the type given in variant b), or, if appropriate, with a salt thereof, and, if desired, in each case converting a compound of the formula II which can be obtained according to the process or by a different route, in free form or in salt form, into a different compound of the formula II, resolving an isomer mixture which can be obtained according to the process, and isolating the isomer desired, and/or converting a free compound of the formula II which can be obtained according to the process into a salt, or converting a salt of a compound of the formula II which can be obtained according to the process into the free compound of the formula II or into a different salt.

The compounds IV, in free form or in salt form, are known or can be prepared analogously to known compounds, for example by reacting a compound of the formula



(V),

which is known or which can be prepared analogously to corresponding known compounds and in which R_3 and n are as defined in formula I, or a salt of such a compound, with a compound of the formula



$$R_1\text{-}CF_2\text{-}\overset{\overset{\displaystyle O}{\|}}{C}\text{-}O\text{-}\overset{\overset{\displaystyle O}{\|}}{C}\text{-}CF_2\text{-}R_1$$

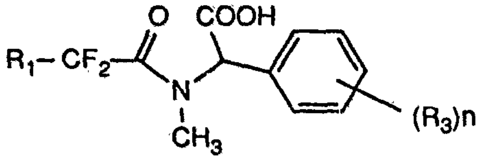
(VI),

which is known or which can be prepared analogously to corresponding known compounds and in which R_1 is as defined in formula I, the process preferably being carried out under atmospheric pressure, if appropriate in the presence of a solvent or diluent, for example of the type given in the case of variant a), in particular in the presence of a high-boiling aromatic hydrocarbon such as toluene, xylene, mesitylene or tetralin, and at temperatures between 70°C and 200°C, in particular between 120°C and 180°C, preferably at the boiling point of the reaction mixture.

The compounds VII which are used as educts in process variant c), in free form or in salt form, are novel and also form a subject of the invention.

A further subject of the invention is the process for the preparation of the compounds of the formula VII, in free form or in salt form, which comprises, for example,

f) reacting a compound of the formula



(VIII),

in which R_1, R_3 and n are as defined in formula I, or a salt of such a compound, with acrylonitrile, preferably in a high-boiling polar solvent, such as acetonitrile, in the presence of an acid anhydride, such as acetic anhydride, and at the reflux temperature of the reaction mixture,
and, if desired, converting a compound of the formula VII which can be obtained according to the process or by a different route, in free form or in salt form, into a different compound of the formula VII, resolving an isomer mixture which can be obtained according to the process, and isolating the isomer desired, and/or converting a free compound of the formula VII which can be obtained according to the process into a salt, or converting a salt of a compound of the formula VII which can be obtained according to the process into the free compound of the formula VII or into a different salt.

The compounds VIII are known or can be prepared analogously to corresponding known compounds.

A compound I, II or VII which can be obtained according to the process or by a different route can be converted into a different compound I, II or VII in a manner known per se by replacing one or more substituents of the starting compound I, II or VII in the customary manner by (an)other substituent(s) according to the invention.

For example,
- hydroxyl groups (as substituents of alkyl groups R_4) can be alkylated to give alkoxy groups (as substituents of alkyl groups R_4);
- halogen R_3 can be introduced into unsubstituted positions of the phenyl ring;
- mercapto groups R_3 and/or R_4 can be oxidised to give sulfinyl or sulfonyl groups R_3 and/or R_4 or sulfinyl groups R_3 and/or R_4 can be oxidised to give sulfonyl groups R_3

and/or R_4;
- methyl groups R_4 can be mono-halogenated to give mono-halomethyl groups R_4; or
- the halogen substituent present in mono-halomethyl groups R_4 can be replaced by an alkoxy group to give a mono-alkoxymethyl group R_4.

Depending on the choice of the reaction conditions and starting materials which are suitable in each case, it is possible to replace, in one reaction step, only one substituent by a different substituent according to the invention, or a plurality of substituents can be replaced by other substituents according to the invention in the same reaction step.

Salts of compounds I, II or VII can be prepared in a manner known per se. For example, acid addition salts of compounds I, II or VII are obtained by treatment with a suitable acid or a suitable ion-exchanger reagent, and salts with bases are obtained by treatment with a suitable base or a suitable ion-exchanger reagent.

Salts of compounds I, II or VII can be converted in the customary manner into the free compounds I, II or VII, respectively, for example acid addition salts by treatment with a suitable basic agent or a suitable ion-exchanger reagent and salts with bases for example by treatment with a suitable acid or a suitable ion-exchanger reagent.

Salts of compounds I, II or VII can be converted into other salts of compounds I, II or VII, respectively, in a manner known per se, for example acid addition salts into different acid addition salts, for example by treatment of a salt of an inorganic acid such as a hydrochloride with a suitable metal salt such as a sodium salt, barium salt or silver salt, of an acid, for example with silver acetate, in a suitable solvent in which an inorganic salt which forms, for example silver chloride, is insoluble and so precipitates from the reaction mixture.

Depending on the procedure or reaction conditions, the compounds I, II or VII which have salt-forming properties can be obtained in free form or in the form of salts.

The compounds I, II or VII, in free form or in salt form, can exist in the form of one of the isomers which are possible or as a mixture thereof, for example as pure isomers, such as antipodes and/or diastereomers, or as isomer mixtures such as enantiomer mixtures, for example racemates, diastereomer mixtures or racemate mixtures, depending, for example, on the number, absolute and relative configuration of asymmetric carbon atoms in the molecule and/or depending on the configuration of non-aromatic double bonds in the molecule; the invention relates to the pure isomers as well as to all isomer mixtures which are possible and is to be understood accordingly hereinbefore and hereinafter, even when stereochemical details are not mentioned specifically in each case.

Diastereomer mixtures and racemate mixtures of compounds I, II or VII, in free form or in salt form, which can be obtained according to the process - depending on the choice of starting materials and procedures - or by other routes can be resolved in the known manner based on the physicochemical differences of the components to give the pure diastereomers or racemates, for example by fractional crystallisation, distillation and/or chromatography.

Enantiomer mixtures such as racemates which can be obtained accordingly can be resolved by known methods to give the optical antipodes, for example by recrystallisation from an optically active solvent, by chromatography on chiral adsorbents, for example high-pressure liquid chromatography (HPLC) on acetylcellulose, with the aid of suitable microorganisms, by cleavage with specific immobilised enzymes, via the formation of inclusion compounds, for example using chiral crown ethers, where only one enantiomer is sequestered, or by conversion into diastereomeric salts, for example by reacting a basic end product racemate with an optically active acid such as carboxylic acid, for example camphoric acid, tartaric acid or malic acid, or sulfonic acid, for example camphorsulfonic acid, and resolution of the resulting diastereomer mixture, for example on the basis of their different solubilities by fractional crystallisation to give the diastereomers from which the enantiomer desired can be liberated by the action of suitable agents, for example basic agents.

In addition to the resolution of corresponding isomer mixtures, pure diastereomers, or enantiomers, can also be obtained according to the invention by generally known methods of diastereoselective, or enantioselective, synthesis, for example by carrying out the process according to the invention with educts having a suitable stereochemistry.

It is advantageous to isolate, or synthesise, in each case the biologically more active isomer, for example enantiomer or diastereomer, or isomer mixture, for example enantiomer mixture or diastereomer mixture, if the individual components have different biological activities.

The compounds I, II or VII, in free form or in salt form, can also be obtained in the form of their hydrates and/or can include other solvents, for example those which can be used for the crystallisation of compounds in solid form.

The invention relates to all those embodiments of the process in which a compound which can be obtained in any step of the process is used as starting material or intermediate and all, or some, of the missing steps are carried out, or in which a starting material in the form of a derivative or salt and/or racemates or antipodes thereof are used or, in particular, formed under the reaction conditions.

It is preferred to use those starting materials and intermediates, in each case in free form or in salt form, in the process of the present invention which lead to the compounds I or salts thereof which have been described at the outset as being particularly valuable.

In particular, the invention relates to the preparation processes described in Examples H1 to H6.

Another subject of the invention is novel starting materials and intermediates, in each case in free form or in salt form, which are used according to the invention for the preparation of the compounds I or salts thereof, a process for their preparation, and their use as starting materials and intermediates for the preparation of the compounds I; in particular, this applies to the compounds II and VII.

The compounds I according to the invention are valuable active ingredients in the field of pest control which have a very favourable biocidal spectrum when used preventively and/or curatively, even at low application concentrations, while being well tolerated by warm-blooded species, fish and plants. The active ingredients according to the invention are active against all or individual development stages of normally sensitive, but also resistant, animal pests such as insects and representatives from the order of the Acarina, and of phytopathogenic fungi. The insecticidal and/or acaricidal action of the active ingredients according to the invention can be demonstrated either directly, i.e. in destruction of the pests, which is immediately effective or only after some time has elapsed, for example during moulting, or indirectly, for example by reduced oviposition and/or hatching rate, good action meaning a destruction rate (mortality) of at least 50 to 60 %.

The abovementioned animal pests include, for example:

from the order of the Lepidoptera, for example

Acleris spp., Adoxophyes spp., Aegeria spp., Agrotis spp., Alabama argillaceae, Amylois spp., Anticarsia gemmatalis, Archips spp., Argyrotaenia spp., Autographa spp., Busseola fusca, Cadra cautella, Carposina nipponensis, Chilo spp., Choristoneura spp., Clysia ambiguella, Cnaphalocrocis spp., Cnephasia spp., Cochylis spp., Coleophora spp., Crocidolomia binotalis, Cryptophlebia leucotreta, Cydia spp., Diatraea spp., Diparopsis castanea, Earias spp., Ephestia spp., Eucosma spp., Eupoecilia ambiguella, Euproctis spp., Euxoa spp., Grapholita spp., Hedya nubiferana, Heliothis spp., Hellula undalis, Hyphantria cunea, Keiferia lycopersicella, Leucoptera scitella, Lithocollethis spp., Lobesia botrana, Lymantria spp., Lyonetia spp., Malacosoma spp., Mamestra brassicae, Manduca sexta, Operophtera spp., Ostrinia nubilalis, Pammene spp., Pandemis spp., Panolis flammea, Pectinophora gossypiella, Phthorimaea operculella, Pieris rapae, Pieris spp., Plutella xylostella, Prays spp., Scirpophaga spp., Sesamia spp., Sparganothis spp., Spodoptera spp., Synanthedon spp., Thaumetopoea spp., Tortrix spp., Trichoplusia ni and Yponomeuta spp.;

from the order of the Coleoptera, for example

Agriotes spp., Anthonomus spp., Atomaria linearis, Chaetocnema tibialis, Cosmopolites spp., Curculio spp., Dermestes spp., Diabrotica spp., Epilachna spp., Eremnus spp., Leptinotarsa decemlineata, Lissorhoptrus spp., Melolontha spp., Orycaephilus spp., Otiorhynchus spp., Phlyctinus spp., Popillia spp., Psylliodes spp., Rhizopertha spp., Scarabeidae, Sitophilus spp., Sitotroga spp., Tenebrio spp., Tribolium spp. and Trogoderma spp.;

from the order of the Orthoptera, for example

Blatta spp., Blattella spp., Gryllotalpa spp., Leucophaea maderae, Locusta spp., Periplaneta spp. and Schistocerca spp.;

from the order of the Isoptera, for example

Reticulitermes spp.;

from the order of the Psocoptera, for example

Liposcelis spp.;

from the order of the Anoplura, for example

Haematopinus spp., Linognathus spp., Pediculus spp., Pemphigus spp. and Phylloxera spp.;

from the order of the Mallophaga, for example

Damalinea spp. and Trichodectes spp.;

from the order of the Thysanoptera, for example

Frankliniella spp., Hercinothrips spp., Taeniothrips spp., Thrips palmi, Thrips tabaci and Scirtothrips aurantii;
from the order of the Heteroptera, for example
Cimex spp., Distantiella theobroma, Dysdercus spp., Euchistus spp. Eurygaster spp. Leptocorisa spp., Nezara spp., Piesma spp., Rhodnius spp., Sahlbergella singularis, Scotinophara spp. and Triatoma spp.;
from the order of the Homoptera, for example
Aleurothrixus floccosus, Aleyrodes brassicae, Aonidiella spp., Aphididae, Aphis spp., Aspidiotus spp., Bemisia tabaci, Ceroplaster spp., Chrysomphalus aonidium, Chrysomphalus dictyospermi, Coccus hesperidum, Empoasca spp., Eriosoma larigerum, Erythroneura spp., Gascardia spp., Laodelphax spp., Lecanium corni, Lepidosaphes spp., Macrosiphus spp., Myzus spp., Nephotettix spp., Nilaparvata spp., Paratoria spp., Pemphigus spp., Planococcus spp., Pseudaulacaspis spp., Pseudococcus spp., Psylla spp., Pulvinaria aethiopica, Quadraspidiotus spp., Rhopalosiphum spp., Saissetia spp., Scaphoideus spp., Schizaphis spp., Sitobion spp., Trialeurodes vaporariorum, Trioza erytreae and Unaspis citri;
from the order of the Hymenoptera, for example
Acromyrmex, Atta spp., Cephus spp., Diprion spp., Diprionidae, Gilpinia polytoma, Hoplocampa spp., Lasius spp., Monomorium pharaonis, Neodiprion spp., Solenopsis spp. and Vespa spp.;
from the order of the Diptera, for example
Aedes spp., Antherigona soccata, Bibio hortulanus, Calliphora erythrocephala, Ceratitis spp., Chrysomyia spp., Culex spp., Cuterebra spp., Dacus spp., Drosophila melanogaster, Fannia spp., Gastrophilus spp., Glossina spp., Hypoderma spp., Hyppobosca spp., Liriomyza spp., Lucilia spp., Melanagromyza spp., Musca spp., Oestrus spp., Orseolia spp., Oscinella frit, Pegomyia hyoscyami, Phorbia spp., Rhagoletis pomonella, Sciara spp., Stomoxys spp., Tabanus spp., Tannia spp. and Tipula spp.;
from the order of the Siphonaptera, for example
Ceratophyllus spp. and Xenopsylla cheopis;
from the order of the Thysanura, for example
Lepisma saccharina and
from the order of the Acarina, for example
Acarus siro, Aceria sheldoni, Aculus schlechtendali, Amblyomma spp., Argas spp., Boophilus spp., Brevipalpus spp., Bryobia praetiosa, Calipitrimerus spp., Chorioptes spp., Dermanyssus gallinae, Eotetranychus carpini, Eriophyes spp., Hyalomma spp., Ixodes spp., Olygonychus pratensis, Ornithodoros spp., Panonychus spp., Phyllocoptruta oleivora,

Polyphagotarsonemus latus, Psoroptes spp., Rhipicephalus spp., Rhizoglyphus spp., Sarcoptes spp., Tarsonemus spp. and Tetranychus spp..

The abovementioned phytopathogenic fungi include, for example:
from the class of the Fungi imperfecti, for example
Botrytis spp., Pyricularia spp., Helminthosporium spp., Fusarium spp., Septoria spp., Cercospora spp. and Alternaria spp.;
from the class of the Basidiomycetes, for example
Rhizoctonia spp., Hemileia spp. and Puccinia spp.;
from the class of the Ascomycetes, for example
Venturia spp., Erysiphe spp., Podosphaera spp., Monilinia spp. and Uncinula spp.; and
from the class of the Oomycetes, for example
Phytophthora spp., Pythium spp. and Plasmopara spp..

The active ingredients according to the invention allow the control, i.e. reduction or destruction, of pests of the abovementioned type which are found in particular on plants, especially on useful plants and ornamentals in agriculture, in horticulture and in forestry, or on parts of such plants, such as fruits, flowers, foliage, stalks, tubers or roots, and in some cases the protection against these pests extends to parts of plants which are formed at a later point in time.

Suitable target crops are, in particular, cereals, such as wheat, barley, rye, oats, rice, maize or sorghum; beet such as sugar beet or fodder beet; fruit, for example pomaceous fruit, stone fruit and soft fruit, such as apples, pears, plums, peaches, almonds, cherries or berries, for example strawberries, raspberries or blackberries; pulses such as beans, lentils, peas or soybeans; oil crops such as oil-seed rape, mustard, poppy, olives, sunflowers, coconut, castor-oil plant, cacao or groundnuts; cucurbits such as pumpkins, cucumbers or melons; fibre plants such as cotton, flax, hemp or jute; citrus fruit such as oranges, lemons, grapefruit or tangerines; vegetables such as spinach, lettuce, asparagus, cabbage species, carrots, onions, tomatoes, potatoes or bell peppers; Lauraceae such as avocado, cinnamonium or camphor; and also tobacco, nuts, coffee, egg plants, sugar cane, tea, pepper, vines, hops, Musaceae, latex plants and ornamentals.

The active ingredients according to the invention are particularly suitable for controlling insects and representatives from the order of the Acarina, in particular feeding insects which are plant-injurious, such as Anthonomus grandis, Diabrotica balteata, Heliothis virescens larvae, Plutella xylostella and Spodoptera littoralis larvae, and spider mites, such as Tetranychus spp., in cotton, fruit, maize, soybean, oil-seed rape and vegetable crops.

Other fields of application of the active ingredients according to the invention are the protection of stored products and stores and of material, and, in the hygiene sector, in particular the protection of domestic animals and productive livestock against pests of the abovementioned type.

The invention therefore also relates to pesticides such as emulsifiable concentrates, suspension concentrates, directly sprayable or dilutable solutions, spreadable pastes, dilute emulsions, wettable powders, soluble powders, dispersible powders, dusts, granules or encapsulations in polymeric substances, which comprise - at least - one of the active ingredients according to the invention and which are to be selected depending on the intended aims and prevailing circumstances.

The active ingredient in these compositions is employed as pure active ingredient, for example a solid active ingredient in a specific particle size, or, preferably, together with - at least - one of the auxiliaries conventionally used in the art of formulation, such as extenders, for example solvents or solid carriers, or surface-active compounds (surfactants).

The following are possible as solvents: aromatic hydrocarbons which may be partially hydrogenated, preferably the fractions C_8 to C_{12} of alkylbenzenes, such as xylene mixtures, alkylated naphthalenes or tetrahydronaphthalenes, aliphatic or cycloaliphatic hydrocarbons such as paraffins or cyclohexane, alcohols such as ethanol, propanol or butanol, glycols as well as their ethers and esters such as propylene glycol, dipropylene glycol ether, ethylene glycol or ethylene glycol monomethyl ether or ethylene glycol monoethyl ether, ketones such as cyclohexanone, isophorone or diacetone alcohol, strongly polar solvents such as N-methyl-pyrrolid-2-one, dimethyl sulfoxide or N,N-dimethylformamide, water, unepoxidised or epoxidised vegetable oils such as unepoxidised or epoxidised rapeseed oil, castor oil, coconut oil or soya oil, and silicone oils.

Solid carriers which are generally used, for example for dusts and dispersible powders, are ground natural minerals such as calcite, talc, kaolin, montmorillonite or attapulgite. To improve the physical properties, it is also possible to add highly-disperse silica or highly-disperse absorptive polymers. Possible particulate adsorptive carriers for granules are either porous types, for example pumice, brick grit, sepiolite or bentonite, or non-sorptive carrier materials, for example calcite or sand. Moreover, a large number of pregranulated materials of inorganic or organic nature can be used, in particular dolomite or comminuted plant residues.

Suitable surface-active compounds are non-ionic, cationic and/or anionic surfactants or surfactant mixtures having good emulsifying, dispersing and wetting properties, depending on the nature of the active ingredient to be formulated. The surfactants listed below are only exemplary; a large number of other surfactants which are customarily used in the art of the formulation and suitable according to the invention are described in the specialist literature.

Suitable non-ionic surfactants are mainly polyglycol ether derivatives of aliphatic or cycloaliphatic alcohols, saturated or unsaturated fatty acids and alkylphenols, which can contain 3 to 30 glycol ether groups and 8 to 20 carbon atoms in the (aliphatic) hydrocarbon radical and 6 to 18 carbon atoms in the alkyl radical of the alkylphenols. Other suitable non-ionic surfactants are water-soluble polyethylene oxide adducts with polypropylene glycol, ethylenediaminopolypropylene glycol and alkylpolypropylene glycol which have 1 to 10 carbon atoms in the alkyl chain and contain 20 to 250 ethylene glycol ether groups and 10 to 100 propylene glycol ether groups. The compounds mentioned generally contain 1 to 5 ethylene glycol units per propylene glycol unit. Examples which may be mentioned are nonylphenol polyethoxyethanols, castor oil polyglycol ethers, polypropylene/polyethylene oxide adducts, tributylphenoxypolyethoxyethanol, polyethylene glycol and octylphenoxypolyethoxyethanol. Other suitable substances are fatty acid esters of polyoxyethylene sorbitan, such as polyoxyethylene sorbitan trioleate.

The cationic surfactants are mainly quaternary ammonium salts which contain at least one alkyl radical having 8 to 22 C atoms as substituents and which have lower halogenated or free alkyl, benzyl or lower hydroxyalkyl radicals as further substituents. The salts are preferably in the form of halides, methylsulfates or ethylsulfates. Examples which may be mentioned are stearyltrimethylammonium chloride and benzyldi(2-chloroethyl)ethylammonium bromide.

Suitable anionic surfactants can be either water-soluble soaps or water-soluble synthetic surface-active compounds. Suitable soaps which may be mentioned are the alkali metal salts, alkaline earth metal salts and substituted or unsubstituted ammonium salts of higher fatty acids (C_{10}-C_{22}), such as the sodium salts or potassium salts of oleic or stearic acid or of natural mixtures of fatty acids which can be obtained, for example, from coconut oil or tallow oil; fatty acid methyltaurides may furthermore be mentioned. However, synthetic surfactants are used more frequently, in particular fatty sulfonates, fatty sulfates, sulfonated benzimidazole derivatives or alkylaryl sulfonates. The fatty sulfonates and fatty sulfates are generally in the form of alkali metal salts, alkaline earth metal salts or substituted or unsubstituted ammonium salts and generally have an alkyl radical having 8 to 22 C atoms, alkyl also embracing the alkyl moiety of acyl radicals; examples which may be mentioned are the sodium or the calcium salt of ligninsulfonic acid, of the dodecylsulfuric acid ester or of a fatty alcohol sulfate mixture prepared from natural fatty acids. This group also includes the salts of the sulfuric acid esters and sulfonic acids of fatty alcohol/ethylene oxide adducts. The sulfonated benzimidazole derivatives preferably contain 2 sulfonyl groups and one fatty acid radical having 8 to 22 C atoms. Examples of alkylaryl sulfonates are the sodium, calcium or triethanolammonium salts of dodecylbenzenesulfonic acid, of dibutylnaphthalenesulfonic acid or of a naphthalenesulfonic acid/formaldehyde condensation product. Other suitable compounds are corresponding phosphates, for example salts of the phosphoric acid ester of a p-nonylphenol/(4-14)ethylene oxide adduct, or phospholipids.

As a rule, the compositions comprise 0.1 to 99 %, in particular 0.1 to 95 %, of active ingredient and 1 to 99.9 %, in particular 5 to 99.9 %, of - at least - one solid or liquid auxiliary, where, as a rule, 0 to 25 %, in particular 0.1 to 20 %, of the composition can be surfactants (% is in each case to be understood as meaning per cent by weight). While concentrated compositions are more preferred as commercially available goods, the end consumer generally uses dilute compositions which have considerably lower concentrations of active ingredient. In particular, preferred compositions are composed as follows (% = per cent by weight):

Emulsifiable concentrates:

Active ingredient:	1 to 90 %,	preferably 5 to 20 %
Surfactant:	1 to 30 %,	preferably 10 to 20%
Solvent:	5 to 98 %,	preferably 70 to 85 %

Dusts:

Active ingredient:	0.1 to 10 %,	preferably 0.1 to 1 %
Solid carrier:	99.9 to 90 %,	preferably 99.9 to 99 %

Suspension concentrates:

Active ingredient:	5 to 75 %,	preferably 10 to 50 %
Water:	94 to 24 %,	preferably 88 to 30 %
Surfactant:	1 to 40 %,	preferably 2 to 30 %

Wettable powders:

Active ingredient:	0.5 to 90 %,	preferably 1 to 80 %
Surfactant:	0.5 to 20 %,	preferably 1 to 15 %
Solid carrier:	5 to 99 %,	preferably 15 to 98 %

Granules:

Active ingredient:	0.5 to 30 %,	preferably 3 to 15 %
Solid carrier:	99.5 to 70 %,	preferably 97 to 85 %

The activity of the compositions according to the invention can be broadened considerably and adapted to prevailing circumstances by adding other insecticidal, acaricidal and/or fungicidal active ingredients. Suitable examples of active ingredients which are added are representatives of the following classes of active ingredients: organophosphorus compounds, nitrophenols and derivatives, formamidines, ureas, carbamates, pyrethroids, chlorinated hydrocarbons and Bacillus thuringiensis preparations. The compositions according to the invention can also comprise further solid or liquid auxiliaries, such as stabilisers, for example epoxidised or unepoxidised vegetable oils (for example epoxidised coconut oil, rapeseed oil or soya oil), defoamers, for example silicone oil, preservatives, viscosity regulators, binders and/or adhesives, and also fertilisers or other active ingredients for achieving specific effects, for example bactericides, nematicides, molluscicides or selective herbicides.

The compositions according to the invention are prepared in a manner known per se, for example in the absence of auxiliaries by grinding and/or screening of a solid active ingredient, or mixture of active ingredients, for example to obtain a certain particle size, and in the presence of at least one auxiliary for example by intimately mixing and/or grinding the active ingredient, or mixture of active ingredients, with the auxiliary(ies).

These processes for the preparation of the compositions according to the invention and the use of the compounds I for the preparation of these compositions are also part of the invention.

The invention furthermore relates to the methods by which the compositions are applied, i.e. the methods for controlling pests of the abovementioned type, such as spraying, atomising, dusting, brushing on, dressing, scattering or pouring, to be selected depending on the intended aims and the prevailing circumstances, and to the use of the compositions for controlling pests of the abovementioned type. Typical application concentrations are between 0.1 and 1000 ppm, preferably between 0.1 and 500 ppm, of active ingredient. The application rates per hectare are generally 1 to 2000 g of active ingredient per hectare, in particular 10 to 1000 g/ha, preferably 20 to 600 g/ha.

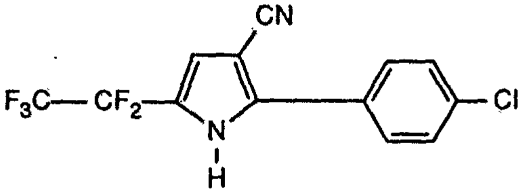
A preferred application method in the field of crop protection is application to the foliage of the plants (foliar application), where the frequency of application and the dosage rate can be adjusted as a function of the severity of infestation with the pest in question. Alternatively, the active ingredient can reach the plants via the root system (systemic action), by allowing the locus of the plants to absorb a liquid composition or by incorporating the active ingredient in solid form into the locus of the plants, for example the soil, such as in the form of granules (soil application). In the case of paddy rice, such granules can be metered into the flooded paddy field.

The compositions according to the invention are also suitable for protecting plant propagation material, for example seeds, such as fruits, tubers or grains, or plant cuttings, against fungal infections and animal pests. The propagation material can be treated with the composition before use, for example seed can be dressed before sowing. The active ingredients according to the invention can also be applied to seed kernels (coating) either by immersing the kernels in a liquid composition or by coating them with a solid composition. It is also possible to apply the composition to the site where the propagation material is used, for example during sowing into the seed furrow. The invention also relates to these treatment methods for plant propagation material and to the plant propagation material which has been treated in this manner.

The following examples are intended to illustrate the invention. They impose no limitation on the invention. Temperatures are given in degrees centigrade.

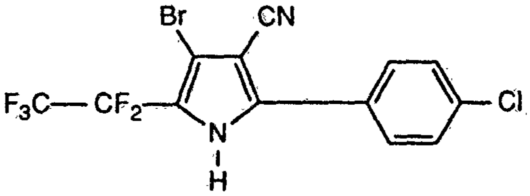
Preparation examples

Example H1: 2-(4-Chlorophenyl)-3-cyano-5-pentafluoroethyl-pyrrole (Table 2, Compound No. 2.1).



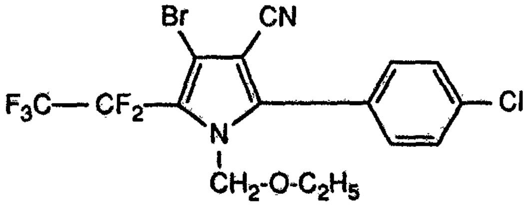
A mixture of 47 g of 4-(4-chlorophenyl)-2,5-dihydro-5-oxo-2-pentafluoroethyl-oxazole, 131.1 g of 2-chloroacrylonitrile and 750 ml of nitromethane is refluxed for 22 hours and then cooled to approx. +5°. The solid which has precipitated is filtered off and washed with a little cold dichloroethane. This gives, if desired with additional working up of the mother liquor, the title compound which melts at 223.5 to 224°.

Example H2: 4-Bromo-2-(4-chlorophenyl)-3-cyano-5-pentafluoroethyl-pyrrole (Table 1, Compound No. 1.1).



20 g of 2-(4-chlorophenyl)-3-cyano-5-pentafluoroethyl-pyrrole and 12.8 g of sodium acetate are introduced into 400 ml of acetic acid. The mixture is stirred for 15 minutes (a clear solution forms), treated dropwise in the course of 1 hour at room temperature with a solution of 49.8 g of bromine in 100 ml of acetic acid, stirred for 2 hours at room temperature, poured into 2 l of sodium hydrogen sulfite solution (10 %) and filtered. The colourless crystals obtained are washed with water and dried in vacuo at +50°. This gives the title compound which melts at 213 to 215° with decomposition.

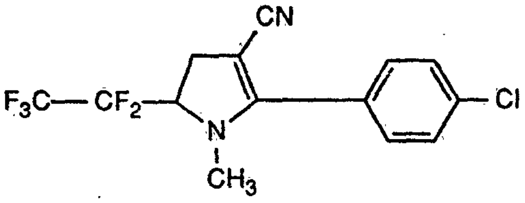
Example H3: 4-Bromo-2-(4-chlorophenyl)-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-pyrrole (Table 1, Compound No. 1.3).



5 g of 4-bromo-2-(4-chlorophenyl)-3-cyano-5-pentafluoroethyl-pyrrole are dissolved in

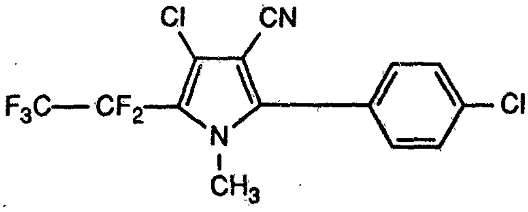
50 ml of dry tetrahydrofuran, and the solution is treated with 2.7 g of potassium tert-butanolate at room temperature. 2.7 g of chloromethyl ethyl ether are subsequently added dropwise. The reaction mixture is stirred for 22 hours at room temperature, poured into 400 ml of water and extracted three times with diethyl ether. The organic phases are combined, washed with NaCl solution, dried over Na_2SO_4 and evaporated in vacuo. The result is yellow crystals which are recrystallised from 10 ml of hexane. This gives the title compound which melts at 69 to 70.5°.

Example H4: 2-(4-Chlorophenyl)-3-cyano-4,5-dihydro-1-methyl-5-pentafluoroethyl-pyrrole.



A mixture of 15 g of N-carboxy-4-chlorophenyl-methyl-N-methyl-N-pentafluoroethyl-carbonyl-amine, 100 ml of acetonitrile, 4.2 ml of acrylonitrile, 11 g of acetic anhydride and 15 drops of triethylamine is heated under reflux for 5.5 hours and then evaporated to dryness. Purification of the residue by column chromatography [SiO_2; hexane/ethyl acetate (9:1)] yields the title compound, which melts at 113 to 116°.

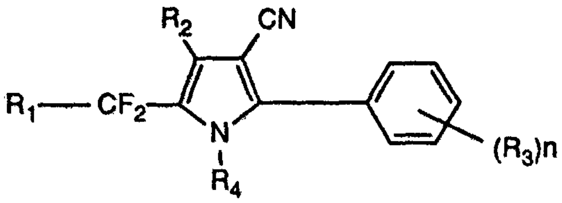
Example H5: 4-Chloro-2-(4-chlorophenyl)-3-cyano-1-methyl-5-pentafluoroethyl-pyrrole (Table 1, Compound No. 1.29).



A saturated solution of 12 g of N-chlorosuccinimide in N,N-dimethylformamide is added over a period of 30 minutes to a mixture of 10 g of 2-(4-chlorophenyl)-3-cyano-4,5-dihydro-1-methyl-5-pentafluoroethyl-pyrrole and 10 ml of N,N-dimethylformamide. The reaction mixture is heated to 100° for 30 minutes and is then poured, while stirring, into 1 l of water. The oily precipitate is taken up in ethyl acetate, and the ethyl acetate phase is washed several times with water, dried and evaporated to dryness. Recrystallisation of the residue using ethyl acetate/hexane yields the title compound, which melts at 120°.

Example H6: In a manner analogous to that described in Examples H1 to H5, it is possible to also prepare the other compounds listed in Tables 1 and 2. In the column "physical data" of these tables, temperatures given in each case denote the melting point of the compound in question, and "n_D^T" is the refractive index of the compound in question at a temperature of T°C.

Table 1



Comp No.	R_1	R_2	R_3	n	R_4	Physical data
1.1	CF_3	Br	4-Cl	1	H	213-215°
1.2	C_2F_5	Br	4-Cl	1	H	199-201°
1.3	CF_3	Br	4-Cl	1	$CH_2OC_2H_5$	69-70.5°
1.4	C_2F_5	Br	4-Cl	1	$CH_2OC_2H_5$	79-80°
1.5	C_2F_5	Br	4-F	1	H	219-220.5°
1.6	CF_3	Br	4-F	1	$CH_2OC_2H_5$	85.5-86°
1.7	C_2F_5	Br	4-F	1	$CH_2OC_2H_5$	81.5-83°
1.8	CF_3	Br	4-F	1	H	222.5-224°
1.9	CF_3	Br	4-Cl	1	CH_3	137-139°
1.10	CF_3	Br	4-CF_3	1	H	238-239.5°
1.11	C_2F_5	Br	4-CF_3	1	H	201-202°
1.12	C_2F_5	Br	4-Cl	1	$CH_2O(CH_2)_2OCH_3$	81.5-82°
1.13	C_2F_5	Br	4-Cl	1	CH_3	162.5-163.5°
1.14	CF_3	Br	4-CF_3	1	$CH_2OC_2H_5$	86-87.5°
1.15	C_2F_5	Br	4-CF_3	1	$CH_2OC_2H_5$	100.5-102.5°
1.16	C_6F_{13}-n	Br	4-Cl	1	H	189-190°
1.17	C_6F_{13}-n	Br	4-Cl	1	$CH_2OC_2H_5$	67-69°
1.18	CF_3	Br	4-Cl	1	$CH_2O(CH_2)_2OCH_3$	$n_D^{26} = 1.5228$
1.19	CF_3	Br	2,4$(Cl)_2$	2	H	172-174°
1.20	C_2F_5	Br	2,4$(Cl)_2$	2	H	155-156°
1.21	CF_3	Br	2,4$(Cl)_2$	2	$CH_2OC_2H_5$	$n_D^{26.5} = 1.5325$
1.22	C_2F_5	Br	2,4$(Cl)_2$	2	$CH_2OC_2H_5$	$n_D^{26.5} = 1.5134$
1.23	CF_3	Br	3,4$(Cl)_2$	2	CH_3	
1.24	CF_3	Br	3,4$(Cl)_2$	2	$CH_2OC_2H_5$	114-116°
1.25	CF_3	Br	3,4$(Cl)_2$	2	CH_2CN	
1.26	CF_3	Br	3,4$(Cl)_2$	2	$CH_2C{\equiv}CH$	

Comp No.	R_1	R_2	R_3	n	R_4	Physical data
1.27	C_2F_5	Br	3,4$(Cl)_2$	2	CH_3	
1.28	C_2F_5	Br	3,4$(Cl)_2$	2	$CH_2OC_2H_5$	107-108°
1.29	CF_3	Cl	4-Cl	1	CH_3	120°
1.30	CF_3	Cl	4-Cl	1	$CH_2OC_2H_5$	76.5-77°
1.31	C_2F_5	Cl	4-Cl	1	CH_3	
1.32	C_2F_5	Cl	4-Cl	1	$CH_2OC_2H_5$	78.5-80°
1.33	CF_3	Cl	4-CF_3	1	CH_3	
1.34	CF_3	Cl	4-CF_3	1	$CH_2OC_2H_5$	
1.35	C_2F_5	Cl	4-CF_3	1	CH_3	
1.36	C_2F_5	Cl	4-CF_3	1	$CH_2OC_2H_5$	
1.37	CF_3	Cl	2,4$(Cl)_2$	2	CH_3	
1.38	CF_3	Cl	2,4$(Cl)_2$	2	$CH_2OC_2H_5$	
1.39	C_2F_5	Cl	2,4$(Cl)_2$	2	CH_3	
1.40	C_2F_5	Cl	2,4$(Cl)_2$	2	$CH_2OC_2H_5$	
1.41	CF_3	Cl	3,4$(Cl)_2$	2	CH_3	
1.42	CF_3	Cl	3,4$(Cl)_2$	2	$CH_2OC_2H_5$	
1.43	C_2F_5	Cl	3,4$(Cl)_2$	2	CH_3	
1.44	C_2F_5	Cl	3,4$(Cl)_2$	2	$CH_2OC_2H_5$	
1.45	CF_3	Br	4-Cl	1	C_2H_5	
1.46	CF_3	Br	4-Cl	1	$CH_2C\equiv CH$	
1.47	CF_3	Br	4-Cl	1	CH_2CN	
1.48	CF_3	Br	4-Cl	1	CH_2O-C_6H_5	
1.49	CF_3	Br	4-Cl	1	CH_2OCH_3	
1.50	CF_3	Br	4-Cl	1	CH_2SCH_3	
1.51	CF_3	Br	4-Cl	1	CH_2COCH_3	
1.52	CF_3	Br	4-Cl	1	CH_2COOCH_3	
1.53	CF_3	Br	4-Cl	1	SO_2CH_3	
1.54	CF_3	Br	4-Cl	1	$SO_2N(CH_3)_2$	
1.55	CF_3	Br	4-Cl	1	$CH_2SC_2H_5$	
1.56	CF_3	Br	4-Cl	1	$CH_2SO_2C_2H_5$	
1.57	CF_3	Br	4-Cl	1	CH_2-C_6H_4-Cl(4)	
1.58	C_2F_5	Br	4-Cl	1	C_2H_5	

Comp No.	R_1	R_2	R_3	n	R_4	Physical data
1.59	C_2F_5	Br	4-Cl	1	$CH_2C{\equiv}CH$	
1.60	C_2F_5	Br	4-Cl	1	CH_2CN	
1.61	C_2F_5	Br	4-Cl	1	$CH_2O\text{-}C_6H_5$	
1.62	C_2F_5	Br	4-Cl	1	CH_2OCH_3	
1.63	C_2F_5	Br	4-Cl	1	CH_2SCH_3	
1.64	C_2F_5	Br	4-Cl	1	CH_2COCH_3	
1.65	C_2F_5	Br	4-Cl	1	CH_2COOCH_3	
1.66	C_2F_5	Br	4-Cl	1	SO_2CH_3	
1.67	C_2F_5	Br	4-Cl	1	$SO_2N(CH_3)_2$	
1.68	C_2F_5	Br	4-Cl	1	$CH_2SC_2H_5$	
1.69	C_2F_5	Br	4-Cl	1	$CH_2SO_2C_2H_5$	
1.70	C_2F_5	Br	4-Cl	1	$CH_2\text{-}C_6H_4\text{-}Cl(4)$	
1.71	CF_3	Br	$2,4,6(Cl)_3$	3	CH_3	
1.72	CF_3	Br	$2,4,6(Cl)_3$	3	$CH_2OC_2H_5$	
1.73	C_2F_5	Br	$2,4,6(Cl)_3$	3	CH_3	
1.74	C_2F_5	Br	$2,4,6(Cl)_3$	3	$CH_2OC_2H_5$	
1.75	CF_3	Br	4-Br	1	CH_3	
1.76	CF_3	Br	4-Br	1	$CH_2OC_2H_5$	
1.77	C_2F_5	Br	4-Br	1	CH_3	
1.78	C_2F_5	Br	4-Br	1	$CH_2OC_2H_5$	
1.79	CF_3	Br	$4\text{-}OCF_3$	1	CH_3	
1.80	CF_3	Br	$4\text{-}OCF_3$	1	$CH_2OC_2H_5$	$n_D^{22}=1.4872$
1.81	C_2F_5	Br	$4\text{-}OCF_3$	1	CH_3	
1.82	C_2F_5	Br	$4\text{-}OCF_3$	1	$CH_2OC_2H_5$	$n_D^{22.5}= 1.4732$
1.83	CF_3	Br	$4\text{-}OCF_2CHF_2$	1	CH_3	
1.84	CF_3	Br	$4\text{-}OCF_2CHF_2$	1	$CH_2OC_2H_5$	
1.85	C_2F_5	Br	$4\text{-}OCF_2CHF_2$	1	CH_3	
1.86	C_2F_5	Br	$4\text{-}OCF_2CHF_2$	1	$CH_2OC_2H_5$	
1.87	CF_3	Br	$4\text{-}OCF_2CHFCF_3$	1	CH_3	
1.88	CF_3	Br	$4\text{-}OCF_2CHFCF_3$	1	$CH_2OC_2H_5$	
1.89	C_2F_5	Br	$4\text{-}OCF_2CHFCF_3$	1	CH_3	

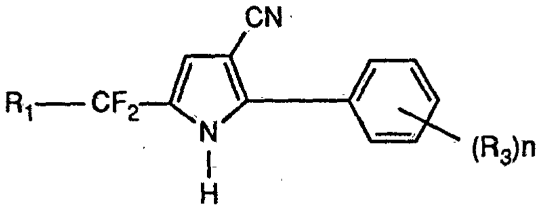
Comp No.	R_1	R_2	R_3	n	R_4	Physical data
1.90	C_2F_5	Br	4-OCF_2CHFCF_3	1	$CH_2OC_2H_5$	
1.91	CF_3	Br	2,3,4,5,6$(F)_5$	5	CH_3	
1.92	CF_3	Br	2,3,4,5,6$(F)_5$	5	$CH_2OC_2H_5$	
1.93	C_2F_5	Br	2,3,4,5,6$(F)_5$	5	CH_3	
1.94	C_2F_5	Br	2,3,4,5,6$(F)_5$	5	$CH_2OC_2H_5$	
1.95	CF_3	Br	4-OCH_2CF_3	1	CH_3	
1.96	CF_3	Br	4-OCH_2CF_3	1	$CH_2OC_2H_5$	
1.97	C_2F_5	Br	4-OCH_2CF_3	1	CH_3	
1.98	C_2F_5	Br	4-OCH_2CF_3	1	$CH_2OC_2H_5$	
1.99	CF_3	Br	2,6$(Cl)_2$,4CF_3	3	CH_3	
1.100	CF_3	Br	2,6$(Cl)_2$,4CF_3	3	$CH_2OC_2H_5$	
1.101	C_2F_5	Br	2,6$(Cl)_2$,4CF_3	3	CH_3	
1.102	C_2F_5	Br	2,6$(Cl)_2$,4CF_3	3	$CH_2OC_2H_5$	
1.103	CF_3	Br	3,4-$(CH=CH)_2$	2	CH_3	
1.104	CF_3	Br	3,4-$(CH=CH)_2$	2	$CH_2OC_2H_5$	110-112°
1.105	C_2F_5	Br	3,4-$(CH=CH)_2$	2	$CH_2OC_2H_5$	131.5-133.5°
1.106	C_2F_5	Br	3,4-$(CH=CH)_2$	2	CH_3	
1.107	CF_3	Br	2,3-$(CH=CH)_2$	2	CH_3	
1.108	CF_3	Br	2,3-$(CH=CH)_2$	2	$CH_2OC_2H_5$	
1.109	C_2F_5	Br	2,3-$(CH=CH)_2$	2	$CH_2OC_2H_5$	
1.110	C_2F_5	Br	2,3-$(CH=CH)_2$	2	CH_3	
1.111	CF_3	Br	2,3-$(CH=CH)_2$,4-Cl	3	CH_3	
1.112	CF_3	Br	2,3-$(CH=CH)_2$,4-Cl	3	$CH_2OC_2H_5$	
1.113	C_2F_5	Br	2,3-$(CH=CH)_2$,4-Cl	3	CH_3	
1.114	C_2F_5	Br	2,3-$(CH=CH)_2$,4-Cl	3	$CH_2OC_2H_5$	
1.115	CF_3	Br	3,4-OCF_2O	2	CH_3	
1.116	CF_3	Br	3,4-OCF_2O	2	$CH_2OC_2H_5$	
1.117	C_2F_5	Br	3,4-OCF_2O	2	$CH_2OC_2H_5$	
1.118	C_2F_5	Br	3,4-OCF_2O	2	CH_3	
1.119	CF_3	Br	4-CH_3SO_2	1	CH_3	
1.120	CF_3	Br	4-CH_3SO_2	1	$CH_2OC_2H_5$	
1.121	C_2F_5	Br	4-CH_3SO_2	1	$CH_2OC_2H_5$	

Comp No.	R_1	R_2	R_3	n	R_4	Physical data
1.122	C_2F_5	Br	4-CH_3SO_2	1	CH_3	
1.123	CF_3	Br	4-CH_3S	1	CH_3	
1.124	CF_3	Br	4-CH_3S	1	$CH_2OC_2H_5$	
1.125	C_2F_5	Br	4-CH_3S	1	$CH_2OC_2H_5$	
1.126	C_2F_5	Br	4-CH_3S	1	CH_3	
1.127	CF_3	Br	4-CH_3	1	CH_3	
1.128	CF_3	Br	4-CH_3	1	$CH_2OC_2H_5$	
1.129	C_2F_5	Br	4-CH_3	1	$CH_2OC_2H_5$	
1.130	C_2F_5	Br	4-CH_3	1	CH_3	
1.131	CF_3	Br	4-OCH_3	1	CH_3	
1.132	CF_3	Br	4-OCH_3	1	$CH_2OC_2H_5$	
1.133	C_2F_5	Br	4-OC_2H_5	1	$CH_2OC_2H_5$	
1.134	C_2F_5	Br	4-OC_2H_5	1	CH_3	
1.135	CF_3	Br	3-Cl	1	CH_3	
1.136	CF_3	Br	3-Cl	1	$CH_2OC_2H_5$	
1.137	C_2F_5	Br	3-Cl	1	$CH_2OC_2H_5$	
1.138	C_2F_5	Br	3-Cl	1	CH_3	
1.139	CF_3	Br	3-CF_3	1	CH_3	
1.140	CF_3	Br	3-CF_3	1	$CH_2OC_2H_5$	
1.141	C_2F_5	Br	3-CF_3	1	$CH_2OC_2H_5$	
1.142	C_2F_5	Br	3-CF_3	1	CH_3	
1.143	CF_3	Br	4-CN	1	CH_3	
1.144	CF_3	Br	4-CN	1	$CH_2OC_2H_5$	
1.145	C_2F_5	Br	4-CN	1	$CH_2OC_2H_5$	
1.146	C_2F_5	Br	4-CN	1	CH_3	
1.147	CF_3	Br	3,4$(Cl)_2$	2	H	207.5-209°
1.148	CF_3	Cl	4-Cl	1	H	211.5-213°
1.149	C_2F_5	Br	3,4$(Cl)_2$	2	H	195.5-197°
1.150	C_2F_5	Cl	4-Cl	1	H	195.5-197°
1.151	CF_3	Br	3,4-$(CH=CH)_2$	2	H	247-248°
1.152	C_2F_5	Br	3,4-$(CH=CH)_2$	2	H	197-198.5°
1.153	CF_3	Br	4-OCF_3	1	H	212.5-215°

Comp No.	R_1	R_2	R_3	n	R_4	Physical data
1.154	C_2F_5	Br	4-OCF_3	1	H	209.5-211°
1.155	CF_3	Cl	4-OCF_3	1	H	214-214.5°
1.156	C_2F_5	Cl	4-OCF_3	1	H	206-208°
1.157	CF_3	Cl	4-OCF_3	1	$CH_2OC_2H_5$	$n_D^{23}= 1.4761$
1.158	C_2F_5	Cl	4-OCF_3	1	$CH_2OC_2H_5$	$n_D^{22.5}= 1.4636$
1.159	CF_3	Cl	4-F	1	H	
1.160	CF_3	Cl	4-F	1	$CH_2OC_2H_5$	
1.161	CF_3	Cl	4-CF_3	1	H	
1.162	C_2F_5	Cl	4-F	1	H	
1.163	C_2F_5	Cl	4-F	1	$CH_2OC_2H_5$	
1.164	CF_3	Cl	2,4$(Cl)_2$	2	H	
1.165	C_2F_5	Cl	2,4$(Cl)_2$	2	H	
1.166	CF_3	Cl	3,4$(Cl)_2$	2	H	
1.167	C_2F_5	Cl	3,4$(Cl)_2$	2	H	
1.168	CF_3	Cl	4-Br	1	H	
1.169	C_2F_5	Cl	4-Br	1	H	
1.170	CF_3	Cl	4-Br	1	$CH_2OC_2H_5$	
1.171	C_2F_5	Cl	4-Br	1	$CH_2OC_2H_5$	
1.172	CF_3	Br	4-Br	1	H	
1.173	C_2F_5	Br	4-Br	1	H	
1.174	CF_3	Br	4-OCF_2CHF_2	1	H	
1.175	C_2F_5	Br	4-OCF_2CHF_2	1	H	
1.176	CF_3	Cl	4-OCF_2CHF_2	1	H	
1.177	CF_3	Cl	4-OCF_2CHF_2	1	$CH_2OC_2H_5$	
1.178	C_2F_5	Cl	4-OCF_2CHF_2	1	$CH_2OC_2H_5$	
1.179	CF_3	Br	3,4-OCF_2O	2	H	
1.180	C_2F_5	Br	3,4-OCF_2O	2	H	
1.181	CF_3	Cl	3,4-OCF_2O	2	H	
1.182	CF_3	Cl	3,4-OCF_2O	2	$CH_2OC_2H_5$	
1.183	C_2F_5	Cl	3,4-OCF_2O	2	H	
1.184	C_2F_5	Cl	3,4-OCF_2O	2	$CH_2OC_2H_5$	

Comp No.	R_1	R_2	R_3	n	R_4	Physical data
1.185	CF_3	Br	2,3-OCF_2O	2	H	
1.186	CF_3	Br	2,3-OCF_2O	2	$CH_2OC_2H_5$	
1.187	C_2F_5	Br	2,3-OCF_2O	2	H	
1.188	C_2F_5	Br	2,3-OCF_2O	2	$CH_2OC_2H_5$	
1.189	CF_3	Cl	2,3-OCF_2O	2	H	
1.190	CF_3	Cl	2,3-OCF_2O	2	$CH_2OC_2H_5$	
1.191	C_2F_5	Cl	2,3-OCF_2O	2	H	
1.192	C_2F_5	Cl	2,3-OCF_2O	2	$CH_2OC_2H_5$	
1.193	C_2F_5	Br	4-OCH_3	1	CH_3	
1.194	C_2F_5	Br	4-OCH_3	1	$CH_2OC_2H_5$	

Table 2



Comp No.	R_1	R_3	n	Physical data
2.1	CF_3	4-Cl	1	223.5-224°
2.2	C_2F_5	4-Cl	1	189.5-192°
2.3	CF_3	4-F	1	223-226°
2.4	C_2F_5	4-F	1	190.5-191°
2.5	CF_3	4-CF_3	1	230-231.5°
2.6	C_2F_5	4-CF_3	1	187.5-188.5°
2.7	C_6F_{13}-n	4-Cl	1	193-193.5°
2.8	CF_3	2,4$(Cl)_2$	2	164-166°
2.9	C_2F_5	2,4$(Cl)_2$	2	153-156°
2.10	CF_3	3,4$(Cl)_2$	2	196.5-198°
2.11	C_2F_5	3,4$(Cl)_2$	2	198-198.5°
2.12	CF_3	2,4,6$(Cl)_3$	3	
2.13	C_2F_5	2,4,6$(Cl)_3$	3	
2.14	CF_3	4-Br	1	

Comp No.	R_1	R_3	n	Physical data
2.15	C_2F_5	4-Br	1	
2.16	CF_3	4-OCF_3	1	224.5-226°
2.17	C_2F_5	4-OCF_3	1	195.5-197°
2.18	CF_3	4-OCF_2CHF_2	1	
2.19	C_2F_5	4-OCF_2CHF_2	1	
2.20	CF_3	4-OCF_2CHFCF_3	1	
2.21	C_2F_5	4-OCF_2CHFCF_3	1	
2.22	CF_3	2,3,4,5,6$(F)_5$	5	
2.23	C_2F_5	2,3,4,5,6$(F)_5$	5	
2.24	CF_3	4-OCH_2CF_3	1	
2.25	C_2F_5	4-OCH_2CF_3	1	
2.26	CF_3	2,6$(Cl)_2$,4CF_3	3	
2.27	C_2F_5	2,6$(Cl)_2$,4CF_3	3	
2.28	CF_3	3,4-$(CH{=}CH)_2$	2	141.5-143°
2.29	C_2F_5	3,4-$(CH{=}CH)_2$	2	148-149°
2.30	CF_3	2,3-$(CH{=}CH)_2$	2	
2.31	C_2F_5	2,3-$(CH{=}CH)_2$	2	
2.32	CF_3	2,3-$(CH{=}CH)_2$,4Cl	3	
2.33	C_2F_5	2,3-$(CH{=}CH)_2$,4Cl	3	
2.34	CF_3	3,4-OCF_2O	2	
2.35	C_2F_5	3,4-OCF_2O	2	
2.36	CF_3	4-CH_3SO_2	1	
2.37	C_2F_5	4-CH_3SO_2	1	
2.38	CF_3	4-CH_3S	1	
2.39	C_2F_5	4-CH_3S	1	
2.40	CF_3	4-CH_3	1	
2.41	C_2F_5	4-CH_3	1	
2.42	CF_3	4-OCH_3	1	
2.43	C_2F_5	4-OCH_3	1	
2.44	CF_3	3-Cl	1	
2.45	C_2F_5	3-Cl	1	
2.46	CF_3	3-CF_3	1	
2.47	C_2F_5	3-CF_3	1	

Comp No.	R_1	R_3	n	Physical data
2.48	CF_3	4-CN	1	
2.49	C_2F_5	4-CN	1	
2.50	C_2F_5	4-OC_2H_5	1	
2.51	CF_3	2,3-OCF_2O	2	
2.52	C_2F_5	2,3-OCF_2O	2	

Formulation examples (% = per cent by weight)

Example F1: Emulsion concentrates	a)	b)	c)
Active ingredient No. 1.23	25 %	40 %	50 %
Calcium dodecylbenzenesulfonate	5 %	8 %	6 %
Castor oil polyethylene glycol ether (36 mol of EO)	5 %	-	-
Tributylphenol polyethylene glycol ether (30 mol of EO)	-	12 %	4 %
Cyclohexanone	-	15 %	20 %
xylene mixture	65 %	25 %	20 %

Emulsions of any desired concentration can be prepared from such concentrates by dilution with water.

Example F2: Solutions	a)	b)	c)	d)
Active ingredient No. 1.23	80 %	10 %	5 %	95 %
Ethylene glycol monomethyl ether	20 %	-	-	-
Polyethylene glycol MW 400	-	70 %	-	-
N-methyl-2-pyrrolidone	-	20 %	-	-
Epoxidised coconut oil	-	-	1 %	5 %
Petroleum spirit (boiling range 160-190°C)	-	-	94 %	-

The solutions are suitable for use in the form of microdrops.

<u>Example F3: Granules</u>	a)	b)	c)	d)
Active ingredient No. 1.22	5 %	10 %	8 %	21 %
Kaolin	94 %	-	79 %	54 %
Highly-disperse silica	1 %	-	13 %	7 %
Attapulgite	-	90 %	-	18 %

The active ingredient is dissolved in dichloromethane, the solution is sprayed onto the carrier, and the solvent is subsequently evaporated in vacuo.

<u>Example F4: Dusts</u>	a)	b)
Active ingredient No. 1.22	2 %	5 %
Highly-disperse silica	1 %	5 %
Talc	97 %	-
Kaolin	-	90 %

Ready-for-use dusts are obtained by intimately mixing the carriers with the active ingredient.

<u>Example F5: Wettable powders</u>	a)	b)	c)
Active ingredient No. 1.2	25 %	50 %	75 %
Sodium ligninsulfonate	5 %	5 %	-
Sodium laurylsulfate	3 %	-	5 %
Sodium diisobutylnaphthalenesulfonate	-	6 %	10 %
Octylphenol polyethylene glycol ether (7-8 mol of EO)	-	2 %	-
Highly-disperse silica	5 %	10 %	10 %
Kaolin	62 %	27 %	-

The active ingredient is mixed with the additives, and the mixture is ground thoroughly in a suitable mill. This gives wettable powders which can be diluted with water to give suspensions of any desired concentration.

<u>Example F6: Emulsion concentrate</u>	
Active ingredient No. 1.3	10 %

Octylphenol polyethylene glycol ether (4-5 mol of EO)	3 %
Calcium dodecylbenzenesulfonate	3 %
Castor oil polyglycol ether (36 mol of EO)	4 %
Cyclohexanone	30 %
Xylene mixture	50 %

Emulsions of any desired concentration can be prepared from this concentrate by dilution with water.

Example F7: Dusts	a)	b)
Active ingredient No. 1.2	5 %	8 %
Talc	95 %	-
Kaolin	-	92 %

Ready-for-use dusts are obtained by mixing the active ingredient with the carrier and grinding the mixture on a suitable mill.

Example F8: Extruder granules

Active ingredient No. 1.4	10 %
Sodium ligninsulfonate	2 %
Carboxymethylcellulose	1 %
Kaolin	87 %

The active ingredient is mixed with the additives, and the mixture is ground and moistened with water. This mixture is extruded, granulated and subsequently dried in a stream of air.

Example F9: Coated granules

Active ingredient No. 1.2	3 %
Polyethylene glycol (MW 200)	3 %
Kaolin	94 %

In a mixer, the kaolin which has been moistened with polyethylene glycol is coated uniformly with the finely ground active ingredient. Dust-free coated granules are obtained in this manner.

Example F10: Suspension concentrate

Active ingredient No. 1.3	40 %
Ethylene glycol	10 %
Nonylphenol polyethylene glycol ether (15 mol of EO)	6 %
Sodium ligninsulfonate	10 %
Carboxymethylcellulose	1 %
37% aqueous formaldehyde solution	0.2 %
Silicone oil in the form of a 75 % aqueous emulsion	0.8 %
Water	32 %

The finely ground active ingredient is mixed intimately with the additives. This gives a suspension concentrate from which suspensions of any desired concentration can be prepared by dilution with water.

Biological examples (% = per cent by weight, unless otherwise indicated)

A. Insecticidal action

Example B1: Action against Nilaparvata lugens
Rice plants are treated with an aqueous emulsion spray mixture comprising 400 ppm of active ingredient. When the spray coating has dried on, the rice plants are populated with cicada larvae of the 2nd and 3rd stage. 21 days later, the test is evaluated. The percentage reduction in the population (% action) is determined by comparing the number of surviving cicadas on the treated and on the untreated plants.
In this test, a good action is shown by the compounds of Table 1. An action of more than 80 % is shown, in particular, by the compounds Nos. 1.2, 1.3 and 1.4.

Example B2: Action against Heliothis virescens (ovi-/larvicide)
Eggs of Heliothis virescens which have been deposited on cotton wool are sprayed with an aqueous emulsion spray mixture comprising 400 ppm of active ingredient. After 8 days, the percentage hatching rate of the eggs and the survival rates of the caterpillars are evaluated by comparing them with untreated control batches (% reduction of the population).

In this test, a good action is shown by the compounds of Table 1. An action of more than 80 % is shown, in particular, by the compounds Nos. 1.2, 1.3 and 1.4.

Example B3: Action against Heliothis virescens

Young soybean plants are sprayed with an aqueous emulsion spray mixture comprising 400 ppm of active ingredient. When the spray coating has dried on, the plants are populated with 10 caterpillars of the first stage of Heliothis virescens and placed in a plastic container. The test is evaluated 6 days later. The percentage reduction in the population and in the damage caused by feeding (% action) is determined by the comparisons of the number of dead caterpillars and damage caused by feeding between the treated and untreated plants.

In this test, a good action is shown by the compounds of Table 1. An action of more than 80 % is shown, in particular, by the compounds Nos. 1.2, 1.3 and 1.4.

Example B4: Action against Spodoptera littoralis

Young soybean plants are sprayed with an aqueous emulsion spray mixture comprising 400 ppm of active ingredient. When the spray coating has dried on, the plants are populated with 10 caterpillars of the third stage of Spodoptera littoralis and placed in a plastic container. The test is evaluated 3 days later. The percentage reduction in the population and the percentage reduction in the damage caused by feeding (% action) is determined by the comparisons of the number of dead caterpillars and damage caused by feeding between the treated and untreated plants.

In this test, a good action is shown by the compounds of Table 1. An action of more than 80 % is shown, in particular, by the compounds Nos. 1.2, 1.3 and 1.4.

Example B5: Action against Aphis craccivora

Pea seedlings are infected with Aphis craccivora, subsequently sprayed with a spray mixture comprising 400 ppm of active ingredient and then incubated at 20°C. The test is evaluated after 3 and 6 days. The percentage reduction in the population (% action) is determined by comparing the number of dead aphids on the treated and untreated plants.

In this test, a good action is shown by the compounds of Table 1. An action of more than 80 % is shown, in particular, by the compounds Nos. 1.2, 1.3 and 1.4.

Example B6: Action against Crocidolmia binotalis

Young cabbage plants are sprayed with an aqueous emulsion spray mixture comprising 400 ppm of active ingredient. When the spray coating has dried on, the cabbage plants are

populated with 10 caterpillars of the third stage of Crocidolmia binotalis and placed in a plastic container. The test is evaluated 3 days later. The percentage reduction in the population and the percentage reduction in the damage caused by feeding (% action) are determined by the comparisons of the number of dead caterpillars and damage caused by feeding between the treated and untreated plants.
In this test, a good action is shown by the compounds of Table 1. An action of more than 80 % is shown, in particular, by the compounds Nos. 1.2, 1.3 and 1.4.

Example B7: Action against Nilaparvata lugens (systemic)
Pots with rice plants are placed in an aqueous emulsion solution comprising 10 ppm of the active ingredient. The plants are subsequently populated with larvae of the 2nd and 3rd stage. The test is evaluated 6 days later. The percentage reduction in the population (% action) is determined by comparing the number of cicadas on the treated and untreated plants.
In this test, a good action is shown by the compounds of Table 1. An action of over 80 % is shown, in particular, by compound No. 1.4.

Example B8: Action against Diabrotica balteata
Maize seedlings are sprayed with an aqueous emulsion spray mixture comprising 400 ppm of active ingredient. When the spray coating has dried on, the seedlings are populated with 10 larvae of the second stage of Diabrotica balteata and placed in a plastic container. The test is evaluated 6 days later. The percentage reduction in the population (% action) is determined by comparing the number of dead larvae between the treated and untreated plants.
In this test, a good action is shown by the compounds of Table 1. An action of over 80 % is shown, in particular, by the compounds Nos. 1.2, 1.3, 1.4 and 1.14.

Example B9: Action against Anthonomus grandis
Young cotton plants are sprayed with an aqueous emulsion spray mixture comprising 400 ppm of active ingredient. After the spray coating has dried on, the cotton plants are populated with 10 adult Anthonomus grandis and placed in a plastic container. The test is evaluated 3 days later. The percentage reduction in the population and the percentage reduction in the damage caused by feeding (% action) are determined by the comparisons of the number of dead beetles and damage caused by feeding between the treated and untreated plants.
In this test, a good action is shown by the compounds of Table 1. An action of more than

80 % is shown, in particular, by the compounds Nos. 1.2, 1.3 and 1.4.

Example B10: Action against Plutella xylostella
Young cabbage plants are sprayed with an aqueous emulsion spray mixture comprising 400 ppm of active ingredient. When the spray coating has dried on, the plants are populated with 10 caterpillars of the 3rd stage of Plutella xylostella and placed in a plastic container. The test is evaluated 3 days later. The percentage reduction in the population and the percentage reduction in the damage caused by feeding (% action) are determined by the comparisons of the number of dead caterpillars and damage caused by feeding between the treated and untreated plants.
In this test, a good action is shown by the compounds of Table 1.

Example B11: Action against Aonidiella aurantii
Potato tubers are populated with crawlers of Aonidiella aurantii. After about 2 weeks, the potatoes are dipped into an aqueous emulsion spray mixture, or suspension spray mixture, comprising 400 ppm of active ingredient. After the tubers have dried, they are incubated in a plastic container. To evaluate the test after 10 to 12 weeks, the survival rate of the crawlers of the first subsequent generation of the treated population is compared with that of untreated control batches. In this test, a good action is shown by compounds of Table 1.

Example B12: Action against Bemisia tabaci
Dwarf bean plants are placed in gauze cages and populated with adult Bemisia tabaci. After oviposition, all adults are removed. 10 days later, the plants together with the nymphs thereon are sprayed with an aqueous emulsion spray mixture comprising 400 ppm of active ingredient. After a further 14 days, the percentage hatching rate of the eggs is evaluated by comparing them with untreated control batches.
In this test, a good action is shown by the compounds of Table 1.

Example B13: Action against Blattella germanica
A solution (0.1 %) of the active ingredient in acetone is placed into a Petri dish in such an amount that this corresponds to an application rate of 2 g/m^2. When the solvent has evaporated, 20 nymphs of Blattella germanica (last nymphal stage) are placed in the dish and exposed to the action of the test substance over 2 hours. The nymphs are then anaesthetised using CO_2, transferred to a fresh Petri dish and kept in the dark at 25° and 50 to 70 % atmospheric humidity. After 48 hours, the insecticidal action is determined by calculating the destruction rate.

In this test, a good action is shown by the compounds of Table 1. An action of over 80 % is shown, in particular, by compound No. 1.3.

Example B14: Action against Lucilia cuprina

Batches of 30 to 50 freshly deposited eggs of Lucilia cuprina are placed in test tubes in which 4 ml of nutrient medium have previously been mixed with 1 ml of test solution comprising 16 ppm of active ingredient. After inoculation of the culture medium, the test tubes are sealed with a cotton wool plug and incubated in the incubator for 4 days at 30°. Up to this point in time, larvae approximately 1 cm in length (stage 3) develop in the untreated medium. If the test substance is active, then the larvae are either dead or their development is clearly slowed down at this point in time. The test is evaluated after 96 hours.

In this test, a good action is shown by the compounds of Table 1.

Example B15: Action against Musca domestica

A sugar lump is treated with such an amount of test substance solution that the concentration of test substance in the sugar is 250 ppm after drying overnight. The lump which has been treated in this manner is placed on an aluminium dish together with a wet cotton wool ball and 10 adults of an OP-resistant strain of Musca domestica. The dish is covered with a glass beaker and incubated at 25°. The mortality rate is determined after 24 hours.

In this test, a good action is shown by the compounds of Table 1. An action of over 80 % is shown, in particular, by the compounds Nos. 1.3 and 1.4.

Example B16: Action against Ctenocephalides felis

20 to 25 flea eggs are placed into a horizontally positioned 50 ml cell culture flask containing 15 g of flea larvae nutrient medium comprising 100 ppm of active ingredient. The flasks are incubated in an incubator at 26-27° and 60-70 % atmospheric humidity. After 21 days, the flasks are checked for the presence of adult fleas, pupae which have not hatched and larvae.

In this test, a good action is shown by the compounds of Table 1.

B. Acaricidal action

Example B17: Action against Dermanyssus gallinae

Into a glass container which is open at the top there are introduced 2 to 3 ml of a solution

comprising 10 ppm of active ingredient and approx. 200 individuals of Dermanyssus gallinae in various development stages. The container is sealed with a cotton wool plug, shaken for 10 minutes (until the mites are wetted completely) and then briefly turned upside down so that the remainder of the test solution can be absorbed by the cotton wool. After 3 days, the mortality of the mites is determined (in per cent) by counting the dead individuals.
In this test, a good action is shown by the compounds of Table 1.

Example B18: Action against Tetranychus urticae
Young bean plants are populated with a mixed population of Tetranychus urticae and, 1 day later, sprayed with an aqueous emulsion spray mixture comprising 400 ppm of active ingredient. The plants are subsequently incubated for 6 days at 25°C and then evaluated. The percentage reduction in the population (% action) is determined by the comparisons of the number of dead eggs, larvae and adults on the treated and untreated plants.
In this test, a good action is shown by the compounds of Table 1. An action of more than 80 % is shown, in particular, by the compounds Nos. 1.2, 1.3 and 1.4.

Example B19: Action against Boophilus microplus
Adult female ticks which have sucked themselves full are glued onto a PVC plate and covered with a cotton wool ball. For the treatment, 10 ml of an aqueous test solution comprising 125 ppm of active ingredient is poured over the test animals. The cotton wool ball is then removed, and the ticks are incubated for 4 weeks for oviposition. The action against Boophilus microplus is demonstrated either in the form of mortality or sterility in the case of females or in the form of an ovicidal action in the case of the eggs.
In this test, a good action is shown by the compounds of Table 1.

C. Fungicidal action

Example B20: Action against Puccinia graminis on wheat
a) Residual-protective action
Test method: 6 days after sowing, wheat plants are sprayed to drip point with an aqueous spray mixture (0.02 % active substance) prepared with a wettable powder comprising one of the active ingredients according to the invention, and, 24 hours later, infected with a uredospore suspension of the fungus. After an incubation time of 48 hours (conditions: 95 to 100 per cent relative atmospheric humidity at 20°), the plants are placed in a greenhouse at 22°. The assessment of the action of the active substance is based on the

rust pustule development 12 days after infection.
Test result: Compounds of Table 1 show a good residual-protective action against Puccinia graminis on wheat, for example the compounds Nos. 1.2 and 1.3 reduce fungal infestation to 20 to 5 %. In contrast, infected control plants which have not been treated with the active substance show a fungal infestation of 100 %.

b) Systemic action
Test method: 5 days after sowing, an aqueous spray mixture (0.006 % active substance relative to the soil volume) prepared with a wettable powder comprising one of the active ingredients according to the invention is poured next to wheat plants. Care is taken that the spray mixture does not come into contact with aerial parts of the plants. 48 hours later, the plants are infected with a uredospore suspension of the fungus. After an incubation time of 48 hours (conditions: 95 to 100 per cent relative atmospheric humidity at 20°), the plants are placed in a greenhouse at 22°. The assessment of the action of the active substance is based on the rust pustule development 12 days after infection.
Test result: Compounds of Table 1 show a good systemic action against Puccinia graminis on wheat.

Example B21: Action against Phytophthora infestans on tomatoes
a) Residual-protective action
Test method: Tomato plants are grown for three weeks and then sprayed to drip point with an aqueous spray mixture (0.02 % of active substance) prepared with a wettable powder comprising one of the active ingredients according to the invention, and, 24 hours later, infected with a sporangia suspension of the fungus. The assessment of the action of the active substance is based on the fungal infestation 5 days after infection, during which a relative atmospheric humidity of 90 to 100 per cent and a temperature of 20° are maintained.
Test result: Compounds of Table 1 show a good residual-protective action against Phytophthora infestans on tomatoes, for example the compounds Nos. 1.2, 1.3 and 1.4 reduce the fungal infestation to 20 to 0 %. In contrast, infected control plants which have not been treated with the active substance show a fungal infestation of 100 %.

b) Systemic action
Test method: Tomato plants are grown for three weeks, and an aqueous spray mixture (0.006 % of active substance relative to the soil volume) which has been prepared with a wettable powder comprising one of the active ingredients according to the invention is

then poured next to the tomato plants. Care is taken that the spray mixture does not come into contact with aerial parts of the plants. After 48 hours, the plants are infected with a sporangia suspension of the fungus. The assessment of the action of the active substance is based on the fungal infestation 5 days after infection, during which a relative atmospheric humidity of 90 to 100 per cent and a temperature of 20° are maintained.
Test result: Compounds of Table 1 show a good systemic action against Phytophthora infestans on tomatoes.

Example B22: Residual-protective action against Cercospora arachidicola on groundnuts
Test method: Groundnut plants 10 to 15 cm in height are sprayed to drip point with an aqueous spray mixture (0.02 % of active substance) prepared with a wettable powder comprising one of the active ingredients according to the invention, and, 48 hours later, infected with a conidia suspension of the fungus. The plants are incubated for 72 hours at 21°C and high atmospheric humidity and subsequently placed in a greenhouse until the typical lesions on the leaves occur. The action of the active substance is assessed 12 days after infection based on number and size of the lesions on the leaves.
Test result: Compounds of Table 1 show a good residual-protective action against Cercospora arachidicola on groundnuts.

Example B23: Action against Plasmopara viticola on vines
a) Residual-protective action
Test method: Vine seedlings in the 4- to 5-leaf stage are sprayed to drip point with an aqueous spray mixture (0.02 % of active substance) prepared with a wettable powder comprising one of the active ingredients according to the invention and, 24 hours later, infected with a sporangia suspension of the fungus. The assessment of the action of the active substance is based on the fungal infestation 6 days after infection, during which a relative atmospheric humidity of 95 to 100 per cent and a temperature of 20° are maintained.
Test result: Compounds of Table 1 show a good preventive residual-protective action against Plasmopara viticola on vines, for example the compounds Nos. 1.2, 1.3 and 1.4 reduce the fungal infestation to 20 to 5 %. In contrast, infected control plants which have not been treated with the active substance show a fungal infestation of 100 %.

b) Residual-protective action
Test method: Vine seedlings in the 4- to 5-leaf stage are infected with a sporangia suspension of the fungus, incubated for 24 hours in a humid chamber (conditions: 95 to

100 per cent relative atmospheric humidity at 20°) and then sprayed to drip point with an aqueous spray mixture (0.02 % of active substance) prepared with a wettable powder comprising one of the active ingredients according to the invention. When the spray coating has dried on, the plants are returned to the humid chamber. The assessment of the action of the active substance is based on the fungal infestation 6 days after infection.
Test result: Compounds of Table 1 show a good curative residual-protective action against Plasmopara viticola on vines, for example the compounds Nos. 1.2, 1.3 and 1.4 reduce the fungal infestation to 20 to 5 %. In contrast, infected control plants which have not been treated with the active substance show a fungal infestation of 100 %.

Example B24: Action against Erysiphe graminis on barley

a) Residual-protective action

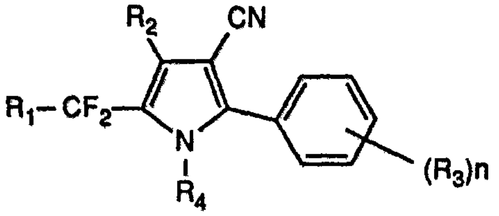
Test method: Barley plants approximately 8 cm in height are sprayed to drip point with an aqueous spray mixture (0.02 % of active substance) prepared with a wettable powder comprising one of the active ingredients according to the invention and, 3 to 4 hours later, dusted with conidia of the fungus. The infected plants are placed in a greenhouse at 22°. The assessment of the action of the active substance is based on the fungal infestation 10 days after infection.
Test result: Compounds of Table 1 show a good residual-protective action against Erysiphe graminis on barley, for example the compounds Nos. 1.2, 1.3 and 1.4 reduce the fungal infestation to 20 to 5 %. In contrast, infected control plants which have not been treated with the active substance show a fungal infestation of 100 %.

b) Systemic action

Test method: An aqueous spray mixture (0.002 % of active substance relative to the soil volume) prepared with a wettable powder comprising one of the active ingredients according to the invention is poured next to barley plants approximately 8 cm in height. Care is taken that the spray mixture does not come into contact with aerial parts of the plants. 48 hours later, the plants are dusted with conidia of the fungus. The infected plants are placed in a greenhouse at 22°. The assessment of the action of the active compound is based on the fungal infestation 10 days after infection.
Test result: Compounds of Table 1 show a good systemic action against Erysiphe graminis on barley.

~~WHAT IS CLAIMED IS:~~ The claims defining the invention are as follows:

1. A compound of the formula



(I),

in which n is the number 1, 2, 3, 4 or 5, where, if n is greater than 1, the radicals R_3 are identical or different; R_1 is halo-C_1-C_9alkyl; R_2 is halogen; R_3 is hydrogen, cyano, nitro, halogen, C_1-C_4alkyl, C_1-C_4alkoxy, C_1-C_4alkylthio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkyl, halo-C_1-C_4alkoxy, halo-C_1-C_4alkylthio, halo-C_1-C_4alkanesulfinyl or halo-C_1-C_4alkanesulfonyl, and/or two substituents R_3 which are bonded to adjacent C atoms of the phenyl ring together are a bridge selected from the group of bridges comprising -O-CH_2-O-, -O-CH_2-CH_2-O-, -O-CH_2-CH_2-, -O-CH_2-CH_2-CH_2- and -CH=CH-CH=CH-, unsubstituted or substituted by halogen, C_1-C_4alkyl, C_1-C_4alkoxy or halo-C_1-C_4alkyl; and R_4 is hydrogen, C_1-C_4alkyl, C_3-C_4alkenyl, halo-C_3-C_4alkenyl, C_3-C_4alkynyl, cyano, C_1-C_4alkylthio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkanesulfonyl, di-C_1-C_4alkylaminosulfonyl, di-C_1-C_4alkylaminocarbonyl or a C_1-C_4alkyl group which is substituted by one or more substituents selected from the group comprising halogen, C_1-C_4alkoxy, hydroxyl, C_1-C_4alkylthio, C_2-C_6alkoxyalkoxy, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, C_1-C_4alkylcarbonyl, C_1-C_4alkoxycarbonyl, C_1-C_4alkylcarbonyloxy, cyano, C_2-C_4alkenylcarbonyloxy, phenyl, phenoxy, phenylthio, benzyloxy, benzoyloxy, benzoyl, phenylsulfinyl and phenylsulfonyl, where the phenyl rings contained in these phenyl, phenoxy, phenylthio, benzyloxy, benzoyloxy, benzoyl, phenylsulfinyl and phenylsulfonyl substituents can in each case be substituted by halogen, C_1-C_4alkyl, C_1-C_4alkoxy, halo-C_1-C_4alkyl, halo-C_1-C_4alkoxy, C_1-C_4alkylthio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkylthio, halo-C_1-C_4-alkanesulfinyl, halo-C_1-C_4-alkanesulfonyl, cyano or nitro,
in free form or in salt form.

2. A compound of the formula I, according to claim 1, in free form.

3. A compound of the formula I, according to claim 2, in which R_1 is perfluoro-C_1-C_6alkyl.

4. A compound of the formula I, according to claim 2, in which R_2 is chlorine or bromine.

5. A compound of the formula I, according to claim 2, in which n is the number 1,2,3,4 or 5, where, if n is greater than 1, the radicals R_3 are identical or different, and R_3 is halogen, halo-C_1-C_4alkyl, halo-C_1-C_4alkoxy, cyano, C_1-C_4alkyl, C_1-C_4alkoxy, C_1-C_4alkythio or C_1-C_4alkanesulfonyl, and/or two substituents R_3 bonded to adjacent C atoms of the phenyl ring together are -O-C(halogen)$_2$-O-, or -CH=CH-CH=CH-.

6. A compound of the formula I, according to claim 2, in which R_4 is hydrogen, C_1-C_4alkyl, C_3-C_4alkenyl, halo-C_3-C_4alkenyl, C_3-C_4alkynyl, cyano, C_1-C_4alkythio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkanesulfonyl, di-C_1-C_4alkylaminosulfonyl, di-C_1-C_4alkylaminocarbonyl or a C_1-C_4alkyl group substituted by one or more substituents selected from the group comprising halogen, C_1-C_4alkoxy, hydroxyl, C_1-C_4alkylthio, C_2-C_6alkoxyalkoxy, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, C_1-C_4alkylcarbonyl, C_1-C_4alkoxycarbonyl, C_1-C_4alkylcarbonyloxy, cyano, C_2C_4alkenylcarbonyloxy, phenyl, phenoxy, phenylthio, benzyloxy, benzoyloxy, benzoyl, phenylsulfinyl and phenylsulfonyl, where the phenyl rings in these phenyl, phenoxy, phenylthio, benzyloxy, benzoyloxy, benzoyl, phenylsulfinyl and phenylsulfonyl substituents can in each case be substituted by halogen, C_1-C_4alkyl, C_1-C_4alkoxy, halo-C_1-C_4alkyl, halo-C_1-C_4alkoxy, C_1-C_4alkylthio, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkylthio, halo-C_1-C_4alkanesulfinyl, halo-C_1-C_4alkanesulfonyl, cyano or nitro.

7. A compound of the formula I, according to claim 2, in which R_4 is hydrogen, C_1-C_4alkyl, C_3-C_4alkynyl, C_1-C_4alkanesulfonyl, di-C_1-C_4alkylaminosulfonyl, or a C_1-C_4alkyl group substituted by C_1-C_4alkoxy, C_1-C_4alkylthio, C_2-C_6alkoxyalkoxy, C_1-C_4alkanesulfonyl, C_1-C_4alkylcarbonyl, C_1-C_4alkoxycarbonyl, cyano, monohalophenyl or phenoxy.

8. A compound of the formula I, according to claim 2, in which R_1 is perfluoro-C_1-C_6alkyl, R_2 is chlorine or bromine, R_3 is halogen, halo-C_1-C_4alkyl, halo-C_1-C_4alkoxy, cyano, C_1-C_4alkyl, C_1-C_4alkoxy, C_1-C_4alkylthio or C_1-C_4alkanesulfonyl, R_4 is hydrogen, C_1-C_4alkyl or C_2-C_6alkoxyalkyl, and n is the number one, two or three, where n can be the number one, two, three, four or five if all radicals R_3 simultaneously are halogen.

9. A compound of the formula I, according to claim 8, in which R_1 is trifluoromethyl or pentafluoroethyl, R_2 is bromine, R_3 is fluorine, chlorine, trifluoromethyl, fluoro-C_1-C_3alkoxy, methylthio, cyano or C_1-C_3alkoxy, R_4 is hydrogen, methyl, methoxymethyl or ethoxymethyl, and n is the number one, two or three.

10. A compound of formula I, according to claim 8, in which R_1 is trifluoromethyl, R_2 is chlorine or bromine, R_3 is fluorine, chlorine, trifluoromethyl, fluoro-C_1-C_3alkoxy, methylthio, cyano or C_1-C_3alkoxy, R_4 is hydrogen, methyl, methoxymethyl or ethoxymethyl, and n is the number one, two or three.

11. A compound of the formula I, according to claim 9, in which n is the number 1, R_1 is trifluoromethyl or pentafluoroethyl, R_2 is bromine, R_3 is trifluoromethyl, trifluoromethoxy or chlorine, and R_4 is hydrogen or ethoxymethyl.

12. A compound of the formula I, according to claim 2, selected from the group comprising the compounds:

(a) 4-Bromo-2-(4-chlorophenyl)-3-cyano-5-heptafluoropropyl-pyrrole,

(b) 4-Bromo-2-(4-chlorophenyl)-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-pyrrole,

(c) 4-Bromo-2-(4-chlorophenyl)-3-cyano-1-1-ethoxymethyl-5-heptafluoropropyl-pyrrole,

(d) 4-Chloro-2-(4-chlorophenyl)-3-cyano-5-heptafluoropropyl-pyrrole,

(e) 4-Chloro-2-(4-chlorophenyl)-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-pyrrole,

(f) 4-Chloro-2-(4-chlorophenyl)-3-cyano-1-ethoxymethyl-5-heptafluoropropyl-pyrrole,

(g) 4-Bromo-3-cyano-5-pentafluoroethyl-2-(4-trifluoromethylphenyl)-pyrrole,

(h) 4-Chloro-3-cyano-5-pentafluoroethyl-2-(4-trifluoromethylphenyl)-pyrrole,

(i) 4-Bromo-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-2-(4-trifluoromethylphenyl)-pyrrole,

(j) 4-Chloro-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-2-(4-trifluoromethylphenyl)-pyrrole,

(k) 4-Bromo-3-cyano-5-pentafluoroethyl-2-(4-trifluoromethoxyphenyl)-pyrrole,

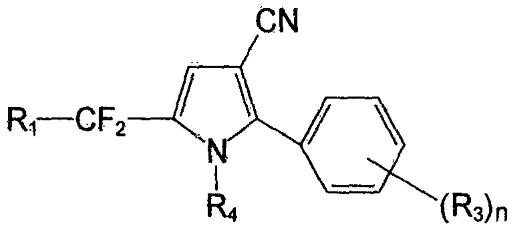
(l) 4-Chloro-3-cyano-5-pentafluoroethyl-2-(4-trifluoromethoxyphenyl)-pyrrole,

(m) 4-Bromo-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-2-(4-trifluoromethoxyphenyl)-pyrrole, and

(n) 4-Chloro-3-cyano-1-ethoxymethyl-5-pentafluoroethyl-2-(4-trifluoromethylphenyl)-pyrrole.

13. A process for the preparation of a compound of the formula I, defined in claim 1, in free form or in salt form, which comprises

a) introducing the halogen substituent R_2 into the 4-position of the pyrrole ring of a compound of the formula



(II),

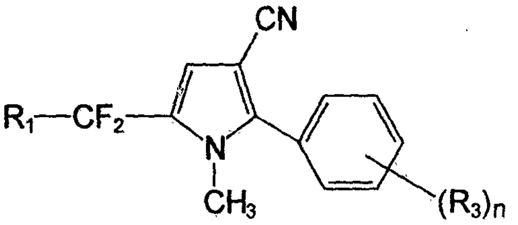
in which R_1, R_3, R_4, and n are as defined in claim 1, or into a salt thereof, by reaction with a halogenating agent, optionally in the presence of a base, or

b) to prepare a compound of the formula I in which R_4 is other than hydrogen, or a salt of such a compound, reacting a compound of the formula I in which R_4 is hydrogen, or a salt of such a compound, with a compound of the formula

$X\text{-}R_4$ (III),

in which R_4 is as defined in claim 1, with the exception of hydrogen, and X is a leaving group, or, if appropriate, with a salt thereof, optionally in the presence of a base, or

c) to prepare a compound of the formula I, in which R_4 is methyl or a group R_2CH_2-, or a salt of such a compound, reacting, optionally in the presence of a base, optionally in the presence of a radical initiator, a compound of the formula



(VII),

in which R_1, R_3 and n are as defined in claim 1, or a salt thereof, with a halogenating agent

and, in each, if desired, converting the compound of the formula I so prepared, in free form or in salt form, into a different compound of the formula I, resolving an isomer mixture, and isolating the isomer desired and/or converting a free compound of the formula I into a salt, or converting a salt of a compound of the formula I into the free compound of the formula I or into a different salt.

14. A pesticide which comprises, as active ingredient, at least one compound of the formula I, according to claim 1, in free form or in agrochemically useable salt form, and at least one auxiliary.

15. A process for the preparation of a pesticide according to claim 14, which comprises intimately mixing and/or grinding the active ingredient with the auxiliary(ies).

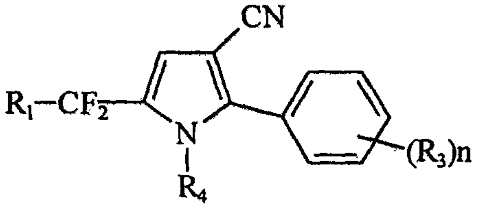
16. A method of controlling pests which comprises applying a pesticide according to claim 14 to the pests or to their environment.

17. A method according to claim 16 wherein the pests are insects, representatives of the order of the Acarina and/or phytopathogenic fungi.

18. A method according to claim 16 for the protection of plant propagation material which comprises treating the propagation material or the site where the propagation material is used.

19. Plant propagation material whenever treated according to the method of claim 18.

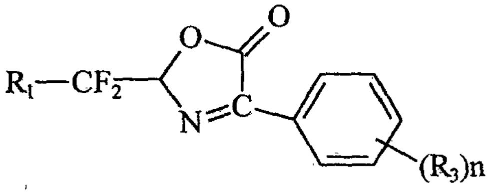
20. A compound of the formula


(II),

in which R_1, R_3, R_4 and n are as defined in claim 1, in free form or in salt form.

21. A process for the preparation of a compound of the formula II, defined in claim 20, in free form or in salt form, which comprises

a) to prepare a compound of the formula II in which R_4 is hydrogen, or a salt thereof, reacting a compound of the formula


(IV)

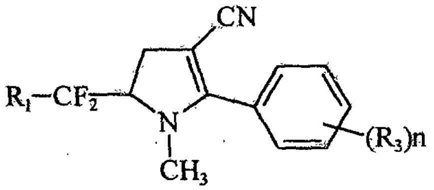
in which R_1, R_3 and n are as defined in claim 1, or a salt of such a compound, with 2-chloroacrylonitrile, optionally in a high-boiling polar solvent, and at the reflux temperature of the reaction mixture, or

b) to prepare a compound of the formula II in which R_4 is other than hydrogen, or a salt of such a compound, reacting a compound of the formula II in which R_4 is hydrogen, or a salt of such a compound, optionally in the presence of a base, with a compound of the formula

$X\text{-}R_4$ (III)

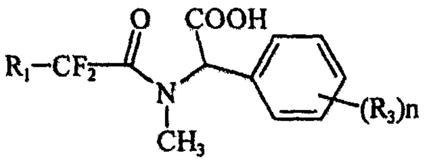
in which R_4 is as defined in claim 1 with the exception of hydrogen, and X is a leaving group, or, if appropriate, with a salt thereof, and, if desired, in each case converting the compound of the formula II so prepared, in free form or in salt form, into a different compound of the formula II, resolving an isomer mixture and isolating the isomer desired, and/or converting a free compound of the formula II into a salt, or converting a salt of a compound of the formula II into the free compound of the formula II or into a different salt.

22. A compound of the formula


(VII),

in which R_1, R_3 and n are as defined in claim 1, in free form or in salt form.

23. A process for the preparation of a compound of the formula VII, defined in claim 22, in free form or in salt form, which comprises reacting a compound of the formula



(VIII),

in which R_1, R_3 and n are as defined in claim 1, or a salt of such a compound, with acrylonitrile optionally in a high-boiling polar solvent, in the presence of an acid anhydride, and at the reflux temperature of the reaction mixture, and, if desired, converting the compound of the formula VII so prepared in free form or in salt form, into a different compound of the formula VII, resolving an isomer mixture and isolating the isomer desired, and/or converting a free compound of the formula VII into a salt, or converting a salt of a compound of the formula VII into the free compound of the formula VII or into a different salt.

24. A cyanophenylpyrrole derivative, substantially as hereinbefore described with reference to any one of the preparation examples.

25. A process for the preparation of a cyanophenylpyrrole derivative, substantially as hereinbefore described with reference to any one of the preparation examples.

26. A pesticide, substantially as hereinbefore described with reference to any one of the formulation examples.

Dated 5 October, 1995
Ciba-Geigy AG

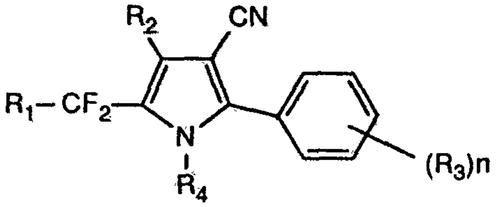
Patent Attorneys for the Applicant/Nominated Person
SPRUSON & FERGUSON

PI/5-18772/A

Cyanophenylpyrroles

Abstract

Compounds of the formula



(I),

in which n is the number 1, 2, 3, 4 or 5, where, if n is greater than 1, the radicals R_3 are identical or different; R_1 is halo-C_1-C_9alkyl; R_2 is halogen; R_3 is hydrogen, cyano, nitro, halogen, C_1-C_4alkyl, C_1-C_4alkoxy, C_1-C_4alkylthio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkyl, halo-C_1-C_4alkoxy, halo-C_1-C_4alkylthio, halo-C_1-C_4alkanesulfinyl or halo-C_1-C_4alkanesulfonyl, and/or two substituents R_3 which are bonded to adjacent C atoms of the phenyl ring together are a bridge selected from the group of bridges comprising -O-CH_2-O-, -O-CH_2-CH_2-O-, -O-CH_2-CH_2-, -O-CH_2-CH_2-CH_2- and -CH=CH-CH=CH-, unsubstituted or substituted by halogen, C_1-C_4alkyl, C_1-C_4alkoxy or halo-C_1-C_4alkyl; and R_4 is hydrogen, an unsubstituted or substituted C_1-C_4alkyl group, C_3-C_4alkenyl, halo-C_3-C_4alkenyl, C_3-C_4alkynyl, cyano, C_1-C_4alkylthio, C_1-C_4alkanesulfinyl, C_1-C_4alkanesulfonyl, halo-C_1-C_4alkanesulfonyl, di-C_1-C_4alkylaminosulfonyl or di-C_1-C_4alkylaminocarbonyl, in free form or in salt form, can be used as agrochemical active ingredients and can be prepared in a manner known per se.