

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952-197

APPLICATION FOR A PATENT

601151

APPLICATION ACCEPTED AND AMENDMENTS

ALLOWED 19.6.90

XXX We, ISTITUTO GUIDO DONEGANI S.p.A.

of via Caduti del Lavoro, 28100 NOVARA, ITALY

hereby apply for a grant of a Patent for an invention entitled:

BENZOYL-UREAS HAVING INSECTICIDE ACTIVITY

which is described in the accompanying complete specification.

This Application is a Convention Application and is based on the Application(s) numbered : 20973 A/85

for a Patent or similar protection made in Italy

on 30 May 1985

LODGED AT SUB-OFFICE

27 MAY 1986

Sydney

XXX Our address for service is care of GRIFFITH HASSEL & FRAZER, Patent Attorneys of 71 York Street, Sydney 2000, in the State of New South Wales, Commonwealth of Australia.

Dated this 27th day of May 1986

ISTITUTO GUIDO DONEGANI S.p.A.By ~~xxx~~ their Patent AttorneyGRIFFITH HASSEL & FRAZER

To : THE COMMISSIONER OF PATENTS
COMMONWEALTH OF AUSTRALIA

ASSIGNEE-APPLICANT

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952 (AS AMENDED)

DECLARATION IN SUPPORT OF AN APPLICATION FOR A PATENT

(Name of applicant) In support of an Application made by: ISTITUTO GUIDO DONEGANI S.p.A.

(Title) for a patent for an invention entitled: BENZOYL-UREAS HAVING INSECTICIDE ACTIVITY

(Full name of I, signatory) A. Collina
(Address of signatory) of Caduti del Lavoro, 28100 NOVARA, ITALY

do solemnly and sincerely declare as follows:

1. I am authorised by the above mentioned applicant for the patent to make this Declaration on its behalf.

2. The name and address of each actual inventor of the invention is as follows:

(Insert details of inventor/s) Pietro MASSARDO ; Paolo PICCARDI ; Franco RAMA and Vincenzo CAPRIOLI

of 5, via Fra' Bartolomeo, 20123 MILAN, ITALY ; 8, via E. De Marchi, 20125 MILAN, ITALY ; 1, via Rodi, 21052 Busto Arsizio, VARESE, ITALY and 8, via Loriga, 28028 S. Martino Siccomario, PAVIA, ITALY, respectively

(Insert details of assignment, etc.) and the fact(s) upon which the applicant is entitled to make this application are as follows:

The applicant is the assignee of the said invention from the said inventors

LODGED AT SUB-OFFICE

27 MAY 1986
Sydney

(Delete paragraphs 3 and 4 for Non-Convention application) 3. The basic application(s) as defined by Section 141 of the Act was(were) made as follows:

Country Italy on 30 May 1985
in the name(s) Istituto Guido Donegani S.p.A.
and in on
in the name(s)
and in on
in the name(s)

4. The basic application(s) referred to in the preceding paragraph of this Declaration was(were) the first application(s) made in a Convention country in respect of the invention the subject of this application.

(Place and date of signing) Declared at Novara, ITALY this 28th day of April 1986

Signed: ISTITUTO GUIDO DONEGANI S.p.A. (Managing Director)
Position: AMMINISTRATORE DELEGATO (Prof. A. Collina)

(12) PATENT ABRIDGMENT (11) Document No. AU-B-57963/86
(19) AUSTRALIAN PATENT OFFICE (11) Acceptance No. 601151

(54) Title
BENZOYL-UREAS HAVING INSECTICIDE ACTIVITY

International Patent Classification(s)
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(21) Application No. : **57963/86** (22) Application Date : **27.05.86**

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20973/85 30.05.85 IT ITALY

(43) Publication Date : **04.12.86**

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(71) Applicant(s)
ISTITUTO GUIDO DONEGANI S.P.A.

(72) Inventor(s)
PIETRO MASSARDO; PAOLO PICCARDI; FRANCO RAMA; VINCENZO CAPRIOLI

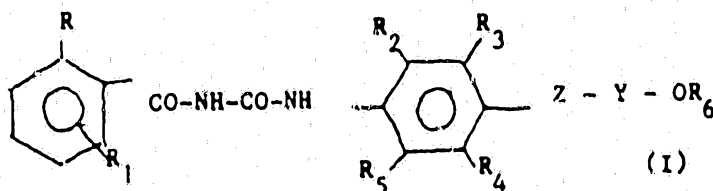
(74) Attorney or Agent
GRIFFITH HACK & CO. S. DNEY

(56) Prior Art Documents
DE 3235419
AU 82529/87
AU 16347/83

(57) As above
 mentioned, the compounds having formula (I) are endowed with a high insecticide activity which shows chiefly against insect larvae and eggs. Amongst these, the ones belonging to the Lepidoptera, Dipthera and Coleoptera orders, may be particularly fought by means of the compounds having formula (I).

CLAIM

1. Compounds having formula:



wherein:

.../2

R represents a chlorine or fluorine atom;

R₁ represents a hydrogen, chlorine, or fluorine atom;

R₂ and R₅, equal to or differing from each other, represent a hydrogen atom, a halogen atom, an alkyl group containing from 1-4 carbon atoms;

R₃ and R₄, equal to or differing from each other, represent a hydrogen atom, halogen atom, an alkyl, haloalkyl, alkoxy, haloalkoxy, alkenyl, haloalkenyl, alkenyloxy, haloalkenyloxy, or alkynyl group;

Z represents an oxygen atom, or a sulphur atom;

Y represents a halo-ethylene or haloethenyl group;

R₆ represents haloalkyl C₁-C₄, haloalkenyl C₃-C₄, cycloalkyl C₃-C₄, halocycloalkyl C₃-C₄ or cycloalkenyl C₃-C₄.



601151

Form 10

PATENTS ACT 1952

COMPLETE SPECIFICATION

(ORIGINAL)

FOR OFFICE USE

Short Title:

Int. Cl:

Application Number: 57963/86.
Lodged:

Complete Specification—Lodged:
Accepted:
Lapsed:
Published:

Priority:

Related Art:

This document contains the
amendments made under
Section 49 and is correct for
printing

LODGED AT SUB-OFFICE
27 MAY 1986
Sydney

Name of Applicant: TO BE COMPLETED BY APPLICANT
ISTITUTO GUIDO DONEGANI S.p.A.

Address of Applicant: via Caduti del Lavoro, 28100, NOVARA, ITALY

Actual Inventor: Pietro MASSARDO ; Paolo PICCARDI ; Franco RAMA
and Vincenzo CAPRIOLI

Address for Service: GRIFFITH HASSEL & FRAZER
71 YORK STREET
SYDNEY, N.S.W. 2000, AUSTRALIA

Complete Specification for the invention entitled: BENZOYL-UREAS HAVING INSECTICIDE ACTIVITY

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

* Note: The description is to be typed in double spacing, pica type face, in an area not exceeding 250 mm in depth and 180 mm in width, on tough white paper of good quality and it is to be inserted inside this form.

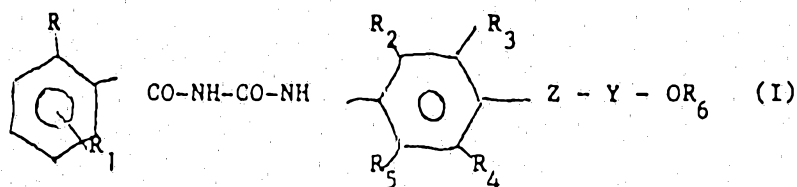
The present invention relates to benzoylurea derivatives having insecticide activity and, more precisely, relates to 1-benzoyl-3-aryl-urea derivatives which are particularly active against insect larvae and eggs, noxious in agrarian and civil field and in the use of such derivatives.

5 Furthermore, the invention relates to the process of synthesis of these urea. Several 1-benzoyl-3-aryl-urea derivatives, endowed with insecticide activity, are known.

Among them, Diflubenzuron, usual appellation of compound 1-(2,6-difluorobenzoyl)-3-(4-chlorophenyl)-urea, specified in U.S.A. patent n° 3.933.908 (U.S. Philips Corporation).

Diflubenzuron, however, is suspected of being a cancerogen [European Chem. News 6 (16), 29 (1978)], as it contains the unit of 4-chloro-aniline in the molecule.

We have now found 1-benzoyl-3-aryl-urea derivatives, which form the object of the present invention, and having general formula: -



wherein:

R represents a chlorine or fluorine atom;

R₁ represents a hydrogen, chlorine or fluorine atom;

R₂ and R₅, equal to or differing from each other, represent hydrogen, halogen atom, an alkyl group containing from 1 - 4 carbon atoms;

R₃ and R₄, equal to or differing from each other, represent a hydrogen, halogen atom, an alkyl, haloalkyl, alkoxy, haloalkoxy, alkenyl, haloalkenyl, alkenyloxy, haloalkenyloxy, or alkinyl group;

Z represents an oxygen atom, a sulphur atom, or a group NR_7 , where R₇ is an alkyl C₁-C₃ or H;

Y represents an alkylene group containing from 2-4 carbon atoms, a haloethylene or haloethenyl group;

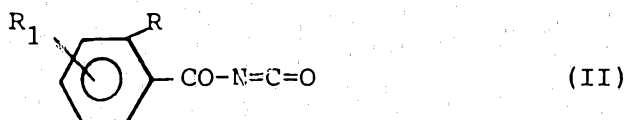
R₆ represents an alkyl C₁-C₄, haloalkyl C₁-C₄, alkenyl C₃-C₄, haloalkenyl C₃-C₄, cycloalkyl C₃-C₄, halocycloalkyl C₃-C₄ or cycloalkenyl C₃-C₄ group.

In the aforesaid definitions, the term halogen is meant preferably as a fluorine, chlorine or bromine atom.

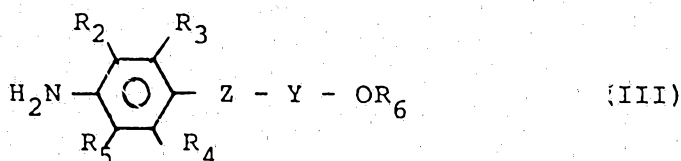
The compounds having formula (I) are endowed with insecticide activity and are fit for use in agrarian, forestal, civil and veterinary field, in the fight against insect infestations.



In the specification of the preparation of the compounds having formula (1) reported hereinafter, symbols R , R_1 , R_2 , R_3 , R_4 , R_5 , R_6 , Z and Y have the meaning reported in formula (1) unless otherwise specified. The compounds having formula (1) are obtained by reaction between a benzoyl-isocyanate having formula:



with an aromatic amine having formula:



The reaction does not require the presence of catalysts, and it is carried out in an inert solvent, and at a temperature ranging between 0° and the boiling temperature of the mixture.

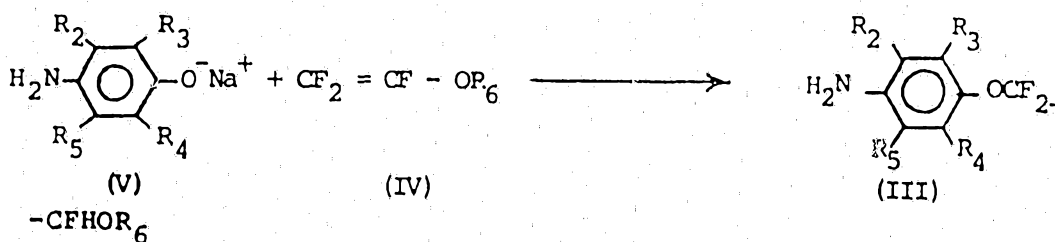
Aromatic hydrocarbons, chlorine hydrocarbons, ethers, ketones, and acetonitrile are suitable solvents.

The benzoyl-isocyanates having formula (II) are known compounds, in some cases they may be found on the market.

Amines having formula (III) may require a specific preparation.

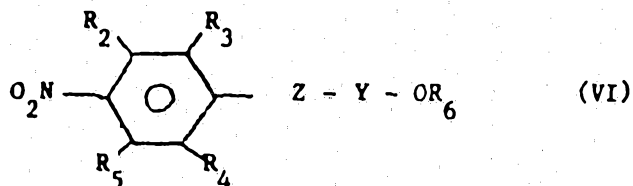
In particular, amines having formula (III) may be prepared according to known methods, for instance:

a) a reaction of a sodium or potassium salt of a suitable amino-phenol (V), in dipolar aprotic solvents, with compound $\text{CF}_2 = \text{CF} - \text{OR}_6$ (IV) wherein R_6 has the meaning given in formula (1), a temperature ranging between 0° and the room temperature, according to equation:

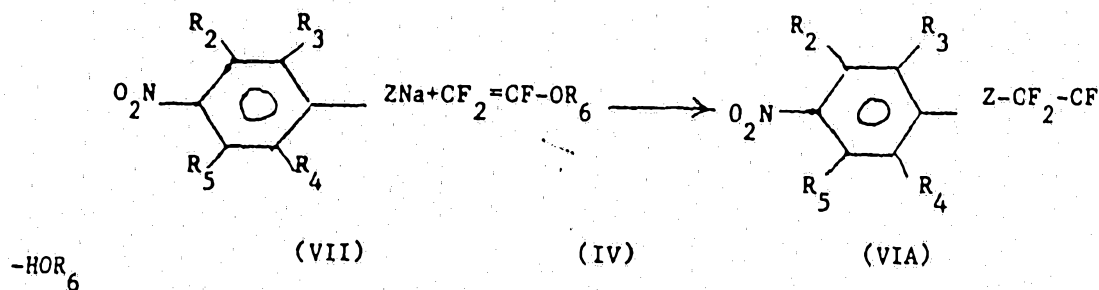


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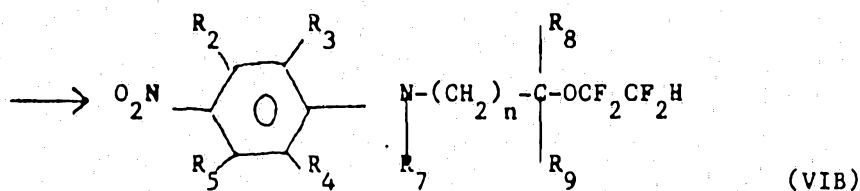
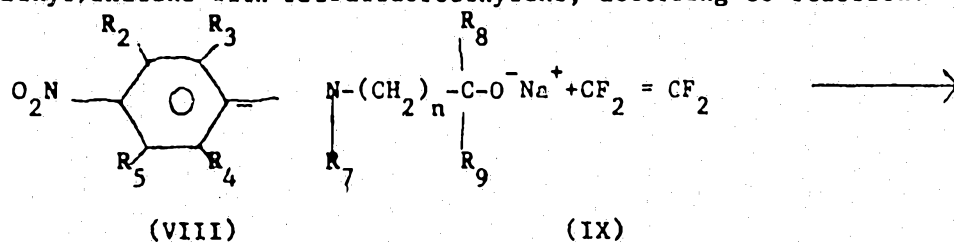
b) reduction, according to known techniques, of nitroderivatives having formula:



In turn, the nitroderivatives having formula (VI) may be prepared, according to traditional techniques, for instance those wherein Y is haloethylene, by reacting a suitable 4-nitrophenyl or 4-nitrothiophenol sodium or potassium salt having formula (VII) with the compound having formula (VI), in suitable dipolar aprotic solvents, according to the equation:



else those wherein Z is =NR₇ and Y is alkenyl, for instance, may be prepared by reacting the sodium salt of a suitable 4-nitro-N-(~~ω~~hydroxy-alkyl)aniline with tetrafluoroethylene, according to reaction:

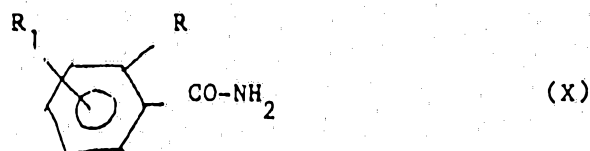


wherein R₈ and R₉ represent a hydrogen atom or an alkyl C₁-C₃ and n = 0, 1, 2, 3.

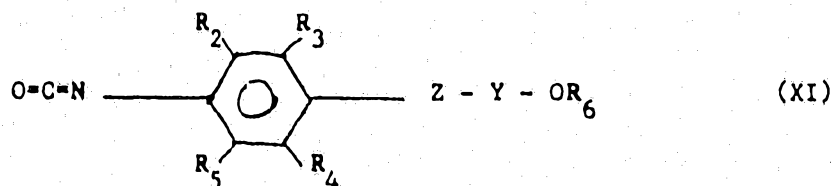
The ethers having formula (IV) may be prepared according to the known method specified, for instance, in J. Am. Chem. Soc., 82, 5116 (1960) when R₆ is alkyl or aryl and in J. Org. Chem., 48, 242 (1983) when R₆ is polyhaloalkyl.

As appears obvious to the skilled technician, different alternative procedures may be used for the synthesis of the intermediates, and of the products having formula (I).

An alternative procedure for the synthesis of the compounds having formula (I) consists, for instance, in reacting a benzamide having formula:



with an isocyanate having formula:



Such reaction is carried out under conditions similar to those specified hereinbefore for the reaction between the benzoyl-isocyanate having formula (II) and the amine having formula (III).

5 The preparation of the isocyanates having formula (XI) foresees however, the preparation of amines having formula (III) and their reaction with phosgene.

10 This aspect, together with the consideration that the benzoyl-isocyanates having formula (II) are available as much as the amides having formula (X) are available, leads to the general preference of the synthesis method mentioned hereinbefore, namely, the reaction between the compounds having formula (II) and (III). As above mentioned, the compounds having formula (I) are endowed with a high insecticide activity which shows chiefly against insect larvae and eggs. Amongst these, the ones belonging to the Lepidoptera, Diptera and Coleoptera orders, may be particularly fought by means of the compounds having formula (I).

20 These orders comprise a great many species, important for their noxiousness in agrarian, forestal, civil and veterinary field. Therefore, the compounds having formula (I) are fit for various uses, namely, for instance, the defense of agricultural cultivations against phytophagous insect infestations, the protection of environments against mosquitoes and flies, the protection of breeding-cattle against some cattle parasites, etc. Furthermore, the compounds having formula (I) show a collateral acaricide activity.

30 For practical uses, the compounds having general formula (I) may be utilized as such or, more conveniently, in the form of compositions containing, other than one or more of the compounds having formula (I) as active constituent, inert solid or liquid carriers and, optionally, other additives. According to the usual formulative practice, the compositions may be in the form of wettable powders, emulsifiable concentrates, etc.

The amount of active constitute in the composition varies within wide ranges (1 - 95% in weight) depending on the type of composition and the use to which such composition is allotted.

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Whensoever particular situations so require, or, in order to enlarge the action spectrum, other active substances such as, for instance, other insecticides or acaricides, may be added to the compositions.

The amount of active substances (compound having formula I) to be distributed for the insecticide treatment, depends on various factors such as, for instance, the type and degree of infestation, the environment in which infestation is had (agrarian cultivation, basins or water-courses, organic substrata of varied nature), the type of employed composition, climatic and environmental factors, available application means, etc. Generally, amounts of active substances ranging between 0,01 and 1 Kg/ha are sufficient for a good disinfection.

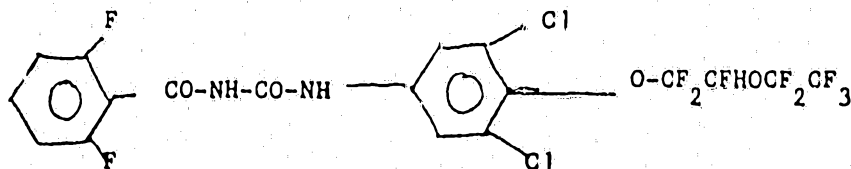
The following examples are given in order to better illustrate the invention:

The following abbreviations are used in the spectra of nuclear magnetic resonance of the proton (^1H -NMR) and of fluorine (^{19}F -NMR), reported in the examples:

s = singlet;
m = multiplet or unresolved complex signal;
d = doublet;
t = triplet;
b = (broad) = broad signal;
ABq = quartet of AB type.

EXAMPLE 1.

Preparation of N-2,6-difluorobenzoyl-N'-3,5-dichloro-4-[1,1,2-trifluoro-2-(perfluoroethoxy)ethoxy]phenylurea (compound n. 1)



1,0 g. of 3,5 dichloro 4-[1,1-2trifluoro-2-(perfluoroethoxy)ethoxy]aniline
(as per subsequent example 3), dissolved in 10 ml. of anhydrous ether, are
5 introduced into a flask having a 50 ml. capacity and equipped with cooler,
dropping funnel and magnetic stirrer.

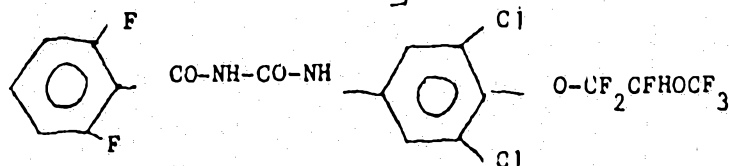
Then, 0,7 g. of 2,6-difluorobenzoylisocyanate, suspended in 10 ml. of
anhydrous tetrahydrofurane, are dripped at room temperature. The mix-
10 ture is kept under stirring for 60 minutes, then cooled and filtered. The
precipitate is concentrated under reduced pressure and the product is sep-
erated for filtering. 1,28 g. of benzoylurea is obtained having a melt-
ing point of 128° - 130°C.



EXAMPLE 2

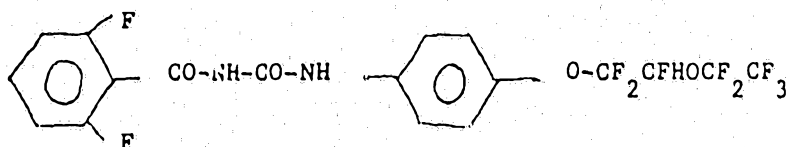
Starting from the anilines specified in subsequent example 3 and operating under conditions similar to those specified in example 1, the following compounds have been prepared, using 2,6 difluorobenzoylisocyanate:

5 - compound n. 2: N-2,6 difluorobenzoyl-N'-3,5-dichloro-4-[1,1,2-trifluoro-2-(trifluoromethoxy)ethoxy]phenylurea



m.p. = 108°-110°C

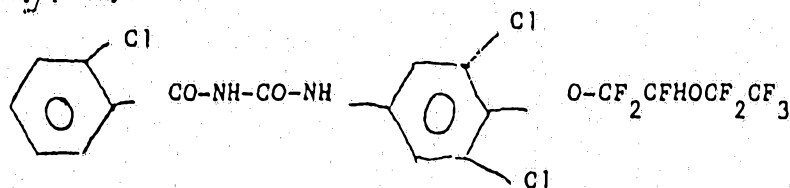
10 - compound n. 3: N-2,6 difluorobenzoyl-N'-4-[1,1,2-trifluoro-2-(perfluoroethoxy)ethoxy]phenylurea



m.p. = 153°-155°C

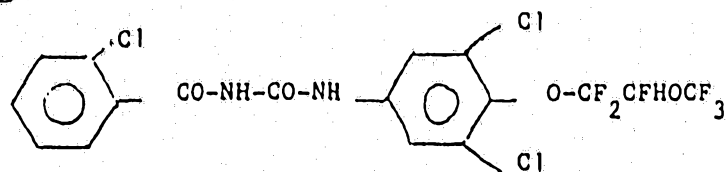
whilst when using the 2-chlorobenzoylisocyanate, the following compounds have been prepared:

20 - compound n. 4: N-2-chlorobenzoyl-N'-3,5 dichloro-4-[1,1,2-trifluoro-2-(perfluoroethoxy)ethoxy]phenylurea



m.p. = 109° - 110°C

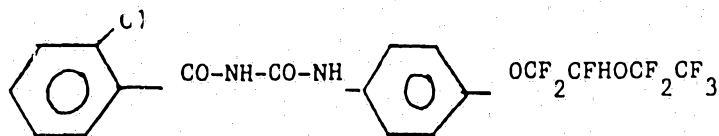
25 - compound n. 5: N-2-chlorobenzoyl-N'-3,5-dichloro-4-[1,1,2-trifluoro-2(trifluoromethoxy)ethoxy]phenylurea



m.p. = 138° - 140°C



- compound n. 6 N-2-chlorobenzoyl-N'-4[1,1,2-trifluoro-2-(perfluoroethoxy)ethoxy] phenylurea



m.p. = 120° - 122°

EXAMPLE 3

Preparation of the intermediate anilines.

32,5 millimoles of 2,6 dichloro4aminophenol, dissolved in 20 ml. of anhydrous dioxane, are introduced, under nitrogen, into a 2-necked flask having a 50 ml. capacity and equipped with cooler, magnetic stirrer and thermometer.

At room temperature, 8,12 millimoles of metallic sodium (or hydrosodium) are added, and stirring is effected at room temperature until the formation of sodium salt is had.

Under nitrogen atmosphere, the mass is transferred into a second flask being 3-necked and having a 100 ml. capacity, and equipped with ethanol dried-ice cooler and magnetic stirrer, and dilution is effected with 20 ml. of anhydrous DMF. Cooling is effected at -30°C, and 32,5 millimoles of the suitable vinyalkylether (IV), in a sole portion and without solvents, are added. The mass is left overnight to return to room temperature whilst stirring.

Finally, the mass is poured into a solution of 10% soda, extracted with ethylether and purified for column chromatography, recuperating the phenol not converted.

Using $\text{CF}_2=\text{CF}-\text{OCF}_2\text{CF}_3$ such as vinyl ether, 4 g. of 3,5-dichloro-4/[1,1,2-trifluoro-2(perfluoroethoxy)ethoxy]aniline, have been obtained with a conversion of 30%

$^1\text{H-NMR}$ 6,6(s, 2H, arom.); 6,4-5,8 (dt, 1H, -CHF-); 3,8 (ab, 2H, NH_2);

$^{19}\text{F-NMR}$ -89,5 - -90,9(ABq, 2F, $-\text{OCF}_2-\text{CHF}_2$); -91,05 (s, 3F, CF_3); -96,12 (ABq, 2F, $-\text{OCF}_2-\text{CF}_2$); -148,75 - -149(dt, 1F, $-\text{CF}_2-\text{CHF}_2$)



Using $\text{CF}_2=\text{CF}-\text{OCF}_3$ such as vinyl ether, 5g. of 3,5-dichloro-4-[1,1,2-(trifluoromethoxy)ethoxy]aniline, has been obtained, with a conversion of 46%.

$^1\text{H-NMR}$: 6,65 (s, 2H, arom); 6,15 - 5,75 (dt, 1H, -CHF-); 4,2 (sb, 2H, NH_2) $^{19}\text{F-NMR}$: -64,7 (s, 3F, $-\text{CF}_3$); -90,45 (m, 2F, $-\text{CF}_2-$); -149,5 (dm, 1F, -CHF)



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By operating under conditions similar to those described hereinbefore,
but using the 4-aminophenol and $\text{CF}_2=\text{CFOCF}_2\text{CF}_3$ such as vinyl ether, 8 g.
of 4-[1,1,2-trifluoro-2(perfluoroethoxy)ethoxy]aniline, have been obtained
with a conversion of 71%

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 $^1\text{H-NMR}$: 6,95-6,55 (dd, 4H, arom.); 6,15-5,9 (dt. 1H, -CFH-);
3,7 (s, 2H, -NH₂); $^{19}\text{F-NMR}$: -91,8 (s, 3F, CF₃); -149,6 (dm, 1F, -CHF-);
-94,4- -96,3 (ABq, 2F, -OCF₂-CFH-); -91,5- -91,9 (ABq, 2F, -OCF₂CF₃).

EXAMPLE 4

Testing of the insecticide activity.

10 Test 1

Residual immediate activity on larvae of Spodoptera littoralis (Lepidoptera).

Tobacco leaves were treated by mechanical spraying with a water-acetone
solution of the product under examination, having an acetone content of
10% by volume and containing a surfactant.

After complete evaporation of the solvents, the leaves were infested with
second-age larvae of Lepidoptera.

The infested leaves were kept in suitable conditioned environment over
the test.

Likewise, tobacco leaves were infested and kept, after having been treat-
ed only with a water-acetone solution containing 10% of acetone and the
surfactant, to be used as reference (comparison treatment).

At a distance of ten days after the infestation, and after having re-
newed at least once the treated substratum, a computation was made of
the dead larvae with respect to the comparison treatment.

25 Test 2

Activity on larvae of Aedes aegypti (Diptera).

spring water (297 ml.) was mixed with an acetic solution (3 ml.) of
the product under examination in a suitable concentration.

25 Dipter larvae, being 4 days of age, suitable fed, were introduced
into the obtained solution. Other larvae were introduced for comparison

purposes into a water-acetone solution (3 ml. acetone, 197 spring water without any active product.

Every 2-3 days, note was taken of the dead larvae and pupae and of the adults normally emerged from the cocoon, till the emergent from the cocoon of the insects in the comparison solution was over.

The activity of the product under examination was expressed as a percent ratio of dead individuals, compared with the total number of treated individuals.

The insecticide activity in the above-mentioned tests was expressed according to the following scale of values.

- 5 = complete activity (98-100% death)
- 4 = high activity (80-97% death)
- 3 = fair activity (60-79% death)
- 2 = sufficient activity (40-59% death)
- 1 = poor activity (20-39% death)
- 0 = negligible or of no value activity (0-19% death)

In the following table 1, the insecticide activity data in the intended doses are reported, expressed through the above scale of values.

Table 1

Insecticide activity

compound n.

Test 1

Test 2

dose: 0,001%

dose: 0,0005%

dose: 0,01 ppm

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REFERENCE. *

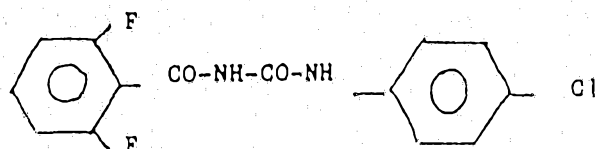
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(*) the following compound was taken as reference

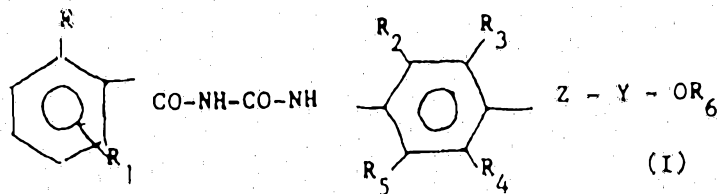
1-(2,6-difluorobenzoyl)3-(4-chlorophenyl)urea) having formula:



specified in patent USA 3,933,908.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. Compounds having formula:



wherein:

R represents a chlorine or fluorine atom;

15 R₁ represents a hydrogen, chlorine, or fluorine atom;

R₂ and R₅, equal to or differing from each other, represent a hydrogen atom, a halogen atom, an alkyl group containing from 1-4 carbon atoms;

20 R₃ and R₄, equal to or differing from each other, represent a hydrogen atom, halogen atom, an alkyl, haloalkyl, alkoxy, haloalkoxy, alkenyl, haloalkenyl, alkenyloxy, haloalkenyloxy, or alkynyl group;

Z represents an oxygen atom, or a sulphur atom;

Y represents a halo-ethylene or haloethenyl group;

25 R₆ represents haloalkyl C₁-C₄, haloalkenyl C₃-C₄, cycloalkyl C₃-C₄, halocycloalkyl C₃-C₄ or cycloalkenyl C₃-C₄.

2. Compound according to claim 1, which is the

30 N-2,6-difluorobenzoyl-N'-3,5-dichloro-4-[1,1,2-trifluoro-2-(perfluoroethoxy)ethoxy]phenylurea,

3. Compound according to claim 1, which is the

35 N-2,6-difluorobenzoyl-N'-3,5-dichloro-4-[1,1,2-trifluoro-2-(trifluoromethoxy)ethoxy]phenylurea.



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4. Compound according to Claim 1, which is the N-2,6-difluorobenzoyl-N'-4-[1,1,2-trifluoro-2-(perfluoroethoxy)ethoxy]phenylurea.

5. Compound according to Claim 1, which is the N-2-chlorobenzoyl-N'-3,5-dichloro-4-[1,1,2-trifluoro-2-(perfluoroethoxy)ethoxy]phenylurea.

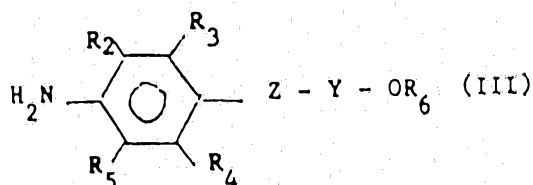
6. Compound according to claim 1, which is the N-2-chlorobenzoyl-N'-3,5-dichloro-4-[1,1,2-trifluoro-2-(trifluoromethoxy)ethoxy]phenylurea.

7. Compound according to claim 1, which is the N-2-chlorobenzoyl-N'-4-[1,1,2-trifluoro-2-(perfluoroethoxy)ethoxy]phenylurea.

8. Process for preparation of compounds according to claim 1, consisting in reacting, in an inert solvent and at a temperature ranging between 0°C and the boiling temperature of the reaction mixture, a benzoyliso-cyanate having formula:



(wherein R and R_1 have the same meaning reported in claim 1), with an aromatic amine having formula:



(wherein R_2 , R_3 , R_4 , R_5 , R_6 , Z and Y have the same meanings reported in claim 1).



9. A method for fighting infestations of noxious insects consisting in the distribution in the infestation zone, of an effective amount of one or more compounds according to claim 1, either as such, or in the form of a composition containing an inert solid or liquid carrier.

10. Insecticide compositions containing, as active ingredient, one or more compounds according to Claim 1, together with inert solid or liquid carriers.

11. A compound having formula (I) substantially as herein described with reference to the Examples and excluding any Comparative Examples.

DATED this 11th day of April, 1990

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By their Patent Attorneys

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