PATENT SPECIFICATION

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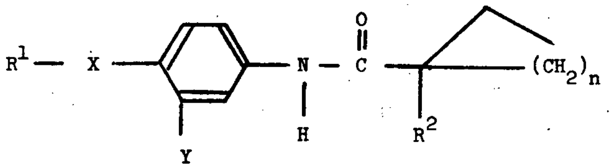
(19)

(54) CYCLOALKANE CARBOXANILIDES AND THEIR USE AS HERBICIDES

(71) We, SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V., a company organised under the laws of The Netherlands, of 30 Carel van Bylandtlaan, The Hague, The Netherlands, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

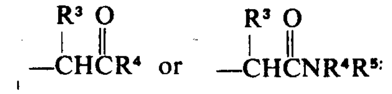
The present invention relates to novel cyclo-alkane-carboxanilide derivatives which possess useful herbicidal activity.

The invention provides a cycloalkanecarboxanilide derivative of the general formula:



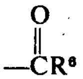
(I)

wherein X represents an O, S, SO, SO_2 or —NR^3 moiety; Y represents a fluorine, chlorine or bromine atom, a cyano, nitro, $C(R^3)$=NOR^3 or $C(O)R^3$ group, or a group —Zp-alkyl in which the alkyl portion contains from 1 to 6 carbon atoms and can be substituted by one or more fluorine, chlorine or bromine atoms and Z represents an O, S, SO or SO_2 group; R^1 represents an alkyl group of from 1 to 6 carbon atoms, optionally substituted by one or more fluorine, chlorine or bromine atoms or represents an alkoxyalkyl group in which each alkyl group contains from 1 to 6 carbon atoms, a cycloalkyl group (as hereinafter defined) having from 3 to 7 carbon atoms in the ring, an aralkyl group of from 7 to 9 carbon atoms optionally ring-substituted by one or two fluorine, chlorine or bromine atoms or by an alkyl group of from 1 to 4 carbon atoms, or a group of the formula:



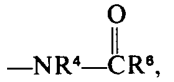
$$-\overset{R^3}{\underset{}{C}}H\overset{O}{\underset{}{\overset{\|}{C}}}R^4 \quad\text{or}\quad -\overset{R^3}{C}H\overset{O}{\overset{\|}{C}}NR^4R^5\text{:}$$

and, when X represents the group NR^3 then R^1 can also represent hydrogen or a group



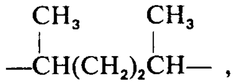
$$-\overset{O}{\overset{\|}{C}}R^6$$

or $—SO_2R^6$; and when X represents the group SO_2 then R^1 can also represent a group $—NH^4R^5$,



$$—NR^4—\overset{\overset{\displaystyle O}{\|}}{C}R^6,$$

or $—NR^4SO_2R^6$; R^2 represents an alkyl group of from 1 to 6 carbon atoms, a fluorine, chlorine or bromine atom, or an alkylthio group having from 1 to 6 carbon atoms; with the proviso that when Y represents a NO_2 group, and X represents an O-moiety, then R^1 does not represent a methyl group; R^3, R^4 and R^5 each independently represents a hydrogen atom or an alkyl or cycloalkyl (as hereinafter defined) group of up to 6 carbon atoms and R^5 can also represent an alkoxy group of up to 6 carbon atoms; when X represents the group $—NR^3—$ then R^1 and R^3 together can form a part of a heterocyclic ring, for example $—CH_2—(CH_2)_mCH_2—$ in which m is 2 or 3,



$$—\overset{\overset{\displaystyle CH_3}{|}}{C}H(CH_2)_2\overset{\overset{\displaystyle CH_3}{|}}{C}H— ,$$

or $—CH_2CH_2OCH_2CH_2—$; when R^1 represents the group $—NR^4R^5$ then R^4 and R^5 together can form a part of a heterocyclic ring, for example as is shown above for R^1 and R^3 when taken together; R^6 represents an alkyl group of from 1 to 6 carbon atoms or an aryl or aralkyl group of from 6 to 10 carbon atoms optionally substituted by one or more fluorine, chlorine or bromine atoms; n is 1 or 2; and p is 0 or 1.

Throughout this specification and claims, the term "cycloalkyl" applied to the groups R^1, R^3, R^4 and R^5 should be understood to include cycloalkyl groups which are alkylated on the ring or linked to the rest of the molecule by an alkylene moiety: i.e. "cycloalkyl" includes alkylcycloalkyl and cycloalkylalkyl.

Preferably n represents 1.

R^2 in the formula I may for example represent a methyl, ethyl, propyl, n-butyl or methylthio group. Preferably R^2 represents a chlorine or bromine atom or, especially, a methyl group.

The group Y may for example represent a chlorine, bromine or fluorine atom, or a cyano, nitro, formyl, hydroximinomethyl, trifluormethyl, trifluoromethoxy, methyl, ethyl, methylsulphonyl or trifluoromethylsulphonyl group. Preferably Y represents a chlorine atom or a methyl, nitro or, especially, trifluoromethyl, group.

If R^1 represents an alkyl group, it is preferably branched-chain. Suitable groups R^1 include methyl, ethyl, isopropyl isobutyl, secondary butyl, tertiary butyl, isoamyl, 2-chloroethyl, trifluoromethyl, cyclopropyl, cyclohexyl, methylcyclopropyl, cyclopropylmethyl, methoxyethyl, benzyl, phenethyl, p-chlorobenzyl and o-methylbenzyl. When X represents the group $—NR^3$, then R^1 can also represent, for example, an acetyl, propionyl, caproyl, benzoyl, methylsulphonyl or trifluoroacetyl group, and, when X represents the group SO_2 then R^1 can also for example represent an unsubstituted amino, monomethylamino, dimethylamino, acetamido or methylsulphonylamino group.

Preferably R^1 represents an alkyl group of 1 to 4 carbon atoms or a cycloalkyl group. Especially active are those compounds wherein R^1 represents a branched-chain alkyl, such as an isopropyl- or tert.-butyl group. Ethyl, methyl and cyclopropylmethyl derivatives and ring alkylated forms appear also to be highly active. Variations in activity, of course, depend on the individual combination of R^1, R^2, R^3, X and Y. Most preferably, R^1 represents a methyl, ethyl, isopropyl, tert. butyl, cyclopropylmethyl or methylcyclopropyl group.

R^3, R^4 and R^5 each represents independently a hydrogen atom, an alkyl group, such as methyl or ethyl group or a cycloalkyl group, such as a methylcyclopropyl group, and R^5 can also represent an alkoxy group, such as a methoxy or ethoxy group. When X represents the group $—NR^3$, then R^3 represents preferably an isopropyl, n-propyl or tert. butyl group.

R^6 may for example represent a methyl, ethyl, isopropyl, isobutyl, secondary-butyl, tertiary-butyl, isoamyl, hexyl, phenyl, *p*-chlorophenyl, 2-bromophenyl, naphthyl, phenethyl or benzyl group.

One group of compounds which are preferred because of their herbicidal properties are those compounds according to formula I, wherein X represents the O moiety; R^2 represents a methyl group; Y represents a trifluoromethyl group; and

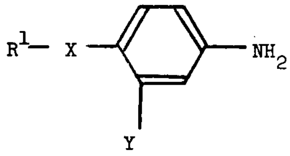
R^1 represents an alkyl group of from 1 to 4 carbon atoms, a cyclopropylmethyl or a 1-methylcyclopropyl group. The alkyl group is preferably a sec.-butyl or isopropyl group. Haloalkyl groups, such as 2-chloroethyl groups, are also preferred.

A further group of compounds which are preferred because of their herbicidal properties are those compounds wherein X represents the S, SO or SO_2 moiety, R^2 represents a methyl group; Y represents a trifluoromethyl group; and R^1 represents an alkyl group of 1 to 4 carbon atoms, such as a methyl, isopropyl or isobutyl group. Especially useful compounds are those wherein R^1 represents an isopropyl or an ethyl group.

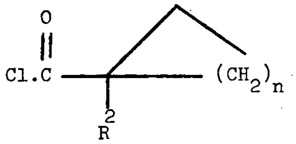
A further group of compounds preferred because of their herbicidal properties are those compounds of formula I, wherein X represents the group NR^3, wherein R^3 represents a hydrogen atom or an alkyl group of from 1 to 4 carbon atoms, such as a methyl, ethyl or propyl group; Y represents a trifluoromethyl group; and R^1 represents an alkyl group of from 1 to 4 carbon atoms, especially an isopropyl, t.-butyl or n-propyl group.

A further group of compounds preferred because of their herbicidal properties are those compounds wherein Y represents a nitro group and R^1 represents an alkyl group of 2 to 4 carbon atoms. Especially useful compounds appear to be those wherein R^1 represents an alkyl group of 2 to 4 carbon atoms, especially an isopropyl group, and X represents an O, S, SO or SO_2 moiety as certain of these compounds show useful crop selectivities. The corresponding compounds wherein X represents a NR^3 group are also highly active.

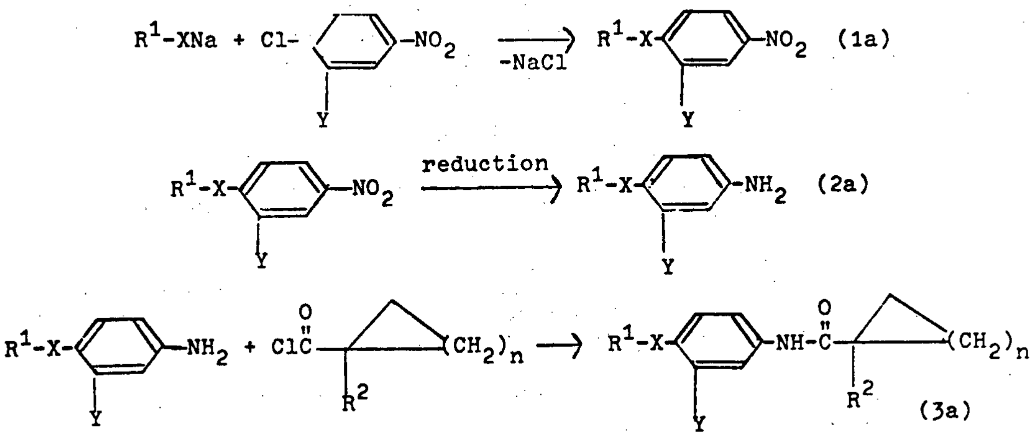
The invention also provides a process for the preparation of a compound according to the invention, which comprises reacting a 3,4-disubstituted aniline of formula:



with a cycloalkanecarboxylic acid chloride of formula:



Thus the compounds of the invention may for example be prepared by the following sequence of reactions:



The appropriate sodium alkoxide or mercaptide compound, or the appropriate amine, may be allowed to react with a 3-substituted-4-chloronitrobenzene in step (1a) to give a 3,4-disubstituted nitrobenzene, which is reduced in step (2a) to give the corresponding aniline. In step (3a) the aniline and a cycloalkane carboxylic chloride are allowed to react to give the desired cycloalkanecarboxanilide according to formula I.

Reaction (1a) is readily conducted by mixing the reactants in a solvent such as an alcohol, dimethyl sulphoxide or dimethylformamide at room temperature or at a moderately elevated temperature, for example up to 150°C.

The reduction of the 3,4-disubstituetd nitrobenzenes, step (2a) is readily carried out in boiling water containing iron filings and up to 5% of acetic or hydrochloric acid. However, any of numerous reduction techniques that reduce an aromatic nitro group to the corresponding amino group are applicable here (see, for instance, R. Schröter and F. Möller in "Methoden der Organischen Chemie", (Houbenn-Wyel), Vol. *11*, 1, part IV, pages 341—731, Georg Thieme Verlag, Stuttgart (1957)).

The corresponding 4'-hydrocarbylsulphinyl- or sulphonyl derivatives are prepared by treating the appropriate 4'-hydrocarbylthio derivative with 85% metachloroperoxybenzoic acid.

The acylation reaction (3a) is suitably conducted by treating the 3,4-disubstituted aniline with a cycloalkanecarboxylic acid chloride in a suitable solvent, such as ether, tetrahydrofuran, benzene, toluene or hexane in the presence of one molar equivalent of an organic or inorganic base that can serve as acceptor for the hydrogen chloride formed in the reaction. Organic bases, such as tertiary amines (pyridine, triethylamine, collidine, N,N-dimethylaniline, diethylisopropylamine) or inorganic bases (Na_2CO_3, $NaHCO_3$, K_2CO_3, $CaCO_3$) may be used to trap the hydrogen chloride formed during the acylation reaction.

The cycloalkanecarboxylic acid chlorides used in the reaction or simple esters from which they can be generated are generally known in the art (e.g. U.S. patents 3,277,171, 3,211,544 and South African patent application 64/1283).

The compounds according to the present invention, for example, 4'-(isopropylamino)-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide, 4'-(isopropoxy)-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide and 4'-(isopropylthio)-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide have been found to be useful for controlling undesirable plant growth. That is, certain members of the class have been found to be herbicidally effective against a wide range of plant species. Others of the class are effective only against a limited number of plant species and are considered to be selective herbicides. Some of the compounds exhibit a high degree of herbicidal activity in the control of a variety of economically important species of grasses and broad-leaved weeds. Some of the compounds are particularly useful as selective herbicides for use in certain important crops.

The invention therefore also provides a herbicidal composition comprising a carrier or a surface-active agent, or both a carrier and a surface-active agent, and, as active ingredient, at least one cycloalkane carboxanilide derivative according to formula I. The invention also provides a method of controlling undesired plant growth at a locus which comprises applying to the locus a herbicidally effective amount of a cycloalkane carboxanilide according to formula I.

A carrier is a solid or liquid material, which may be inorganic or organic and of synthetic or natural origin, with which the active compound is mixed or formulated to facilitate its application to the plant, seed, soil or other object to be treated, or its storage, transport or handling.

Suitable solid carriers are natural and synthetic clays and silicates, for example, natural silicas, such as diatomaceous earths; magnesium silicates, for example, talcs; magnesium aluminium silicates, for example, attapulgites and vermiculites; aluminium silicates, for example, kaolinites, montmorrilinites and micas; calcium carbonates; calcium sulphate; synthetic hydrated silicon oxides and synthetic calcium or aluminium silicates; elements, such as carbon and sulphur; natural and synthetic resins, for example, coumarone resins, polyvinyl chloride and styrene polymers and copolymers; solid polychlorophenols; bitumen, waxes, such as beeswax, paraffin wax, and chlorinated mineral waxes; and solid fertilizers, for example superphosphates.

Examples of suitable liquid carriers are water, alcohols, for example, isopropanol, gylcols; ketones, such as acetone, methyl ethyl ketone, methyl isobutyl ketone and cyclohexanone; ethers; aromatic hydrocarbons, such as benzene, toluene and xylene; petroleum fractions, for example, kerosene, light mineral oils; chlorinated hydrocarbons, such as carbon tetrachloride; perchloroethylene, trichloroethane, including liquefied normally vaporous gaseous compounds. Mixtures of different liquids are often suitable.

A surface-active agent may be an emulsifying agent or a dispersing agent or a wetting agent; it may be non-ionic or ionic. Any of the surface-active agents usually applied in formulating herbicides or insecticides may be used. Examples of suitable surface-active agents are the sodium or calcium salts of polyacrylic acids and lignin sulphonic acids; the condensation products of fatty acids or aliphatic amines or amides containing at least 12 carbon atoms in the molecule with ethylene oxide and/or propylene oxide; fatty acids esters of glycerol, sorbitan, sucrose or pentaerythritol; condensates of these with ethylene oxide and/or propylene oxide; condensation products of fatty alcohols or alkyl phenols, for example, p-octylphenol or p-octylcresol, with ethylene oxide and/or propylene oxide; sulphates or sulphonates of these condensation products, alkali or alkaline earth metal salts, preferably sodium salts or sulphuric or sulphonic acid esters containing at least 10 carbon atoms in the molecule, for example, sodium lauryl sulphate, sodium secondary alkyl sulphates, sodium salts of sulphonated castor oil, and sodium alkylaryl sulphonates, such as sodium dodecylbenzene sulphonate; and polymers of ethylene oxide and copolymers of ethylene oxide and propylene oxide.

The composition of the invention may be formulated as wettable powders, dusts, granules, solutions, emulsifiable concentrates, emulsions, suspension concentrates and aerosols. Wettable powders are usually compounded to contain 25, 50 or 75% by weight of toxicant and usually contain in addition to solid inert carrier, 3—10% by weight of a dispersing agent, 1—5% by weight of a surface-active agent and where necessary, 0—10% by weight of stabilizer(s) and/or other additives, such as penetrants or stickers. Dusts are usually formulated as a dust concentrate having a similar composition to that of a wettable powder but without a dispersant or surface-active agent, and are diluted in the field with further solid carrier to give a composition usually containing 0.5—10% by weight of toxicant. Granules are usually prepared to have a size between 10 and 100 BS mesh (1.676—0.152 mm), and may be manufactured by agglomeration or impregnation techniques. Generally, granules will contain 0.5—25% by weight of toxicant and 0—10% by weight of additives, such as stabilizers, slow-release modifiers and binding agents. Emulsifiable concentrates usually contain, in addition to the solvent and, when necessary, co-solvent, 10—50% by weight per volume of toxicant, 2—20% by weight per volume of emulsifiers and 0—20% by weight per volume of appropriate additives, such as stabilizers, penetrants and corrosion inhibitors. Suspension concentrates are compounded so as to obtain a stable, non-sedimenting, flowable product and usually contain 10—75% by weight of toxicant, 0.5—5% by weight of dispersing agents, 1—5% by weight of surface-active agent, 0.1—10% by weight of suspending agents, such as protective colloids and thixotropic agents, 0—10% by weight of appropriate additives, such as defoamers, corrosion inhibitors, stabilizers, penetrants and stickers, and water or an organic liquid in which the toxicant is substantially insoluble; certain organic solids or inorganic salts may be dissolved in the carrier to assist in preventing sedimentation or as anti-freeze agents for water.

Aqueous dispersions and emulsions, for example, compositions obtained by diluting a wettable powder or a concentrate according to the invention with water, also lie within the scope of the present invention. The said emulsions may be of the water-in-oil or of the oil-in-water type, and may have a thick mayonnaise-like consistency.

The compositions of the invention may also contain other ingredients, for example, other compounds possessing pesticidal, especially insecticidal, acaricidal, herbicidal or fungicidal properties.

The method of applying the compounds according to the present invention comprises applying a compound according to formula I, ordinarily in a composition of one of the aforementioned types, to a locus or area to be protected from undesirable plant growth. The active compound, of course, is applied in amounts sufficient to exert the desired action.

The amount of compound to be used in controlling undesirable vegetation will naturally depend on the condition of the vegetation, the degree of activity desired, the formulation used, the mode of application, the climate, the season of the year, and other variables. Recommendations as to precise amounts are, therefore, not possible. In general, however, application to the locus to be protected of from 0.1 to 10.0 pounds per acre of the compound used in this invention will be satisfactory.

The following Examples illustrate the invention. In the Examples, the identities of all compounds, intermediates and final, were confirmed by elemental analysis, and infrared and nuclear magnetic spectral analyses.

Example 1.

4'-Isopropoxy-3'-(trifluoromethyl)-1-methyl-cyclopropanecarboxanilide

a) *Preparation of 3-(trifluoromethyl)-4-isopropoxynitrobenzene*

To a chilled (5°C) solution of 118g (0.5 mole) of 3-(trifluoromethyl)-4-chloronitrobenzene in 100 ml of dimethyl sulfoxide (DMSO) was added dropwise a solution containing 41g (0.5 mole) of sodium isopropoxide in 300 ml of dimethyl sulphoxide. The dark reaction mixture was stirred at ambient temperature for one hour, poured into water and extracted with ether. The ether extracts were washed with water, dried and concentrated. Recrystallization of the residual liquid from hexane gave 115.5g (92.7%) of a white crystalline solid; m.p. 34—36°C (1a).

b) *Preparation of 3-(trifluoromethyl)-4-isopropoxyaniline*

To a refluxing mixture containing 113.3g (0.455 mole) of (1a) in 1200 ml of 5% aqueous acetic acid and 50 ml of methanol was added portionwise with stirring 137.5g of iron filings. The reaction mixture was refluxed for one hour, filtered while hot, cooled and extracted with ether. The ether extracts were dried and concentrated to give 72.3g (72.6%) of product as an amber oil (1b).

c) *Preparation of 4'-isopropoxy-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide*

To a stirred solution containing 10.9g (0.05 mole) of (1b) and 5.0g (0.05 mole) of triethylamine in 100 ml of tetrahydrofuran was added dropwise 5.9g (0.05 mole) of 1-methylcyclopropanecarboxylic acid chloride. This addition was exothermic to 55°C. The reaction mixture was stirred and refluxed for 30 minutes, and concentrated under reduced pressure. The resulting solid was washed with water, dried and recrystallized from ether to give 14.5g (97%) of a white crystalline solid; m.p. 106—109°C.

Example 2.

4'-(Benzyloxy-3'-trifluoromethyl-1-methycyclopropanecarboxanilide

a) *Preparation of 3-(trifluoromethyl)-4-(benzyloxy)nitrobenzene*

To a solution containing 45.1g (0.2 mole) of 3-(trifluoromethyl)-4-chloronitrobenzene in 100 ml of DMSO was added dropwise at ambient temperature a solution containing 26g (0.2 mole) of sodium benzyloxide in 75 ml of benzyl alcohol causing the temperature to rise to 40°C. After 48 hours at ambient temperature, the reaction mixture was poured into ice water and extracted with ether, and the extract was dried and concentrated to about 200 ml. To the concentrated solution was added 200ml of hexane and the resulting solution was cooled. Filtration gave 51.5g (87%) of colourless crystalline solid; m.p. 112—114°C (2a).

b) Preparation of 3-(trifluoromethyl)-4-(benzyloxy)aniline

To a refluxing mixture containing 64.5g (0.217 mole) of (2a) in 700 ml of 5% aqueous acetic acid and 30 ml of methanol was added portionwise within 15 minutes 65.5g of iron filings. The mixture was stirred and refluxed for an additional 30 minutes, filtered while hot, cooled and extracted with ether. The ether extract was washed with water, dried and concentrated to give 56.0g (97%) of product as a light-amber oil that crystallized on standing (2b).

c) *Preparation of 4'-(benzyloxy-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide*

To a solution of 2.7g (0.01 mole) of 2(b) and 2.7 g (0.01 mol) of triethylamine in 50 ml of tetrahydrofuran was added with stirring at ambient temperature 1.2g (0.01 mole) of 1-methylcyclopropanecarboxylic acid chloride. After one hour, the reaction mixture was poured into ice water and extracted with 200 ml of ether. The ether extract was dried and concentrated. Recrystallization of the residual solid from ether gave 3.6 g of product, m.p. 120—122°C.

Example 3.

4'-(Cyclopropylmethoxy-3'-(trifluoromethyl)-1-methylcylcopropanecarboxanilide

a) *Preparation of 3-(trifluoromethyl)-4-(cyclopropylmethoxy)nitrobenzene*

To a cooled (10°C) solution containing 22.5g (0.1 mole) of 3-(trifluoromethyl)4-chloronitrobenzene in 100 ml of DMSO was added with stirring at ambient temperature a solution containing 0.11 mole of sodium cyclopropylmethoxide (prepared by dissolving 4.9g of 57% sodium hydride in 7.9g of cyclopropylmethanol and 50ml of tetrahydrofuran) in 100 ml of DMSO. The reaction mixture was heated at 50°C for one hour, poured into ice water and filtered. Recrystallization of the filtrate from hexane gave 11.0g (42%) of product as a white crystalline solid, m.p. 41—44°C (3a).

b) *Preparation of 3-(trifluoromethyl)-4-(cyclopropylmethoxy)aniline*

To a stirring and refluxing mixture containing 11.0g (0.042 mole) of (3a) in 150 ml of 5% aqueous acetic acid was added 28g of iron filings and 10 ml of methanol. The mixture was refluxed and stirred vigorously for one hour, then extracted with ether after cooling. The ether extract was washed with aqueous sodium bicarbonate, dried, and concentrated to give 9.5g (98%) of product as an amber oil (3b).

c) *Preparation of 4'-(cyclopropylmethoxy)-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanalide*

To a solution of 4.6g (0.02 mole) of 3(b) and 2.0g (0.02 mole) of triethylamine in 30 ml of tetrahydrofuran was added 2.4g (0.02 mole) of 1-methylcyclopropanecarboxylic acid chloride. The mixture was refluxed for one hour, poured into ice water, and extracted with ether. The extract was dried and concentrated, and the residue was recrystallized from hexane to give 5.0g (79%) of white crystalline solid; m.p. 84—85°C.

Example 4.

4'-(Methylthio)-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide

a) *Preparation of 3-(trifluoromethyl)-4-(methylthio)-nitrobenzene*

To a stirred solution containing 45.1g (0.20 mole) of 3-(trifluoromethyl)-4-chloronitrobenzene and 15 g (0.30 mole) of methyl mercaptan in 150 ml of DMSO was added dropwise at ambient a solution containing 8.0g (0.20 mole) of sodium hydroxide in 20 ml of water. This addition was exothermic to 60°C. After one hour, the reaction mixture was poured into ice water. The product was filtered and dried to give 45.0g (95%) of yellow solid; m.p. 50°C (4a).

b) *Preparation of 3-(trifluoromethyl)-4-(methylthio)aniline*

To a mixture containing 45.0g (0.19 mole) of (4a) in refluxing 5% aqueous acetic acid was added 61g of iron powder. The reaction mixture was refluxed for 3 hours and filtered through Celite (Trade Mark — filter aid) while hot. The cooled filtrate was extracted with ether. The ether extract was washed with 10% aqueous sodium bicarbonate, and then with water, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure to give 31.8 (81%) of product as a light yellow oil (4b).

c) *Preparation of 4'-(methylthio)-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide*

To a stirred solution containing 31.8g (0.154 mole) of (4b) and 15.6g (0.154 mole) of triethylamine in 200 ml of tetrahydrofuran was added dropwise over 10 minutes 18.2g (0.154 mole) of 1-methylcyclopropanecarboxylic acid chloride. This addition was exothermic to 65°C. The mixture was stirred and refluxed for one hour, poured into ice water and filtered. The filter cake was washed with water and dried to give 43.5g (98%) of product as a yellow solid; m.p. 97—98°C.

Example 5.

4'-(Methylsulphinyl)-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide

To a chilled (5°C) solution containing 14.5g (0.05 mole) of 4'-(methylthio)-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide (prepared in Example 4 above) in 150 ml of chloroform was added dropwise over 20 minutes with stirring a solution of 10.2g (0.05 mole) of 85% meta-chloroperoxybenzoic acid in 150 ml of chloroform. The reaction solution was allowed to equilibrate gradually to 25°C, and after 16 hours, washed well with 10% sodium carbonate and water, dried, and concentrated. The residual solid was crystallized from methanol to give 13.0g (86%) of product as a light-cream colored solid; m.p. 181—184 C.

Example 6.

4'-(Methylsulphonyl)-3°-trifluoromethyl-1-methyl-cyclopropanecarboxanilide

To a stirred solution containing 7.0g (0.024 mole) of 4'-(methylthio)-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide (prepared in Example 4 above) in 200 ml of chloroform was added dropwise over a period of 10 minutes at ambient temperature 10.2g (0.05 mole) of 85% metachloroperoxybenzoic acid. The reaction was exothermic to 50°C. The reaction mixture was stirred for 16 hours, washed with 10% aqueous sodium carbonate and then with water. The chloroform layer was dried and concentrated to dryness. The residue was crystallized from methanol to give 7.5g (97%) of product as a light-cream solid; m.p. 135—138°C.

Example 7.

4'-(Isopropylthio)-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide

a) *Preparation of 4-(Isopropylthio)-3-(trifluoromethyl)nitrobenzene*

To a solution containing 112.8g (0.5 mole) of 2-chloro-5-nitrobenzotrifluoride and 46g (0.6 mole) of isopropyl mercaptan in 400 ml of DMSO was added dropwise over 0.5 hour 40g of 50% aqueous sodium hydroxide. This addition was exothermic to 50°C. After 24 hours, the reaction mixture was poured into ice water and extracted with 3 x 200 ml of methylene chloride. The combined extracts were washed with water, dried and concentrated to give 132g (99%) of product as an orange liquid (7a).

b) *Preparation of 4-(isopropylthio)-3-(trifluoromethyl)aniline*

A mixture containing 132.5g (0.5 mole) of the nitro compound (7a) in 800 ml of 5% aqueous acetic acid was heated to reflux with stirring. The heat mantle was removed and 150g of powdered iron was added at such a rate as to maintain reflux. After completion of the addition, the mixture was stirred and refluxed for one hour, and filtered while hot. The filtrate was extracted with ether (3 x 300 ml). The combined extracts were washed with 10% sodium carbonate, and then with water. The dried solution was concentrated to dryness under reduced pressure to give 70.5g (60%) of product as a yellow oil (7b).

c) *Preparation of 4'-(isopropylthio)-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide*

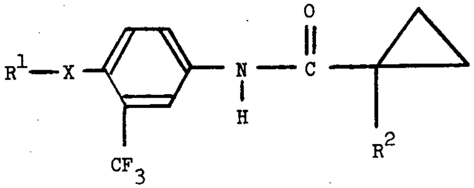
To a stirred solution containing 23.5g (0.1 mole) of the amine (7b) and 10.1g (0.1 mole) of triethylamine in 150 ml of tetrahydrofuran was added dropwise over 5 minutes 11.9g (0.1 mole) of 1-methylcyclopropylcarboxylic acid chloride. This addition was exothermic to 55°C. The mixture was refluxed for 1 hour, poured into ice water and filtered. The filter cake was recrystallized from hexane to give 25G (79%) of product as a colourless crystalline solid, m.p. 103—105°C.

Examples 8—38.

In the manner described above, and illustrated in foregoing Examples, additional cyclopropanecarboxanilides listed in Table 1 were prepared.

TABLE 1

Cyclopropanecarboxanilides



Example	X	R¹	R²	Yield (%)	M.P., °C
8	O	CH_3-	CH_3	96	106–108
9	O	C_2H_5-	CH_3	98	83–85
10	O	$CH_3OCH_2CH_2-$	CH_3	98	83–85
11	O	$C_2H_5CH(CH_3)-$	CH_3	89	93–95
12	O	$(CH_3)_2CHCH_2-$	CH_3	95	91–93
13	O	$CH_3(CH_2)_3-$	CH_3	72	68–70
14	$S\rightarrow O$	$(CH_3)_2CH-$	CH_3	91	143–145
15	$S\begin{matrix}\nearrow O\\ \searrow O\end{matrix}$	$(CH_3)_2CH-$	CH_3	75	140–143
16	S	$(CH_3)_2CHCH_2-$	CH_3	94	95–97
17	$S\rightarrow O$	$(CH_3)_2CHCH_2-$	CH_3	67	oil
18	$S\begin{matrix}\nearrow O\\ \searrow O\end{matrix}$	$(CH_3)_2CHCH_2-$	CH_3	91	oil
19	S	C_2H_5-	CH_3	86	68–69
21	$S\rightarrow O$	C_2H_5-	CH_3	81	136–139

TABLE 1 (Continued)

Example	X	R^1	R^2	Yield (%)	M.P., °C
21	$S\begin{smallmatrix}\nearrow O\\ \searrow O\end{smallmatrix}$	C_2H_5-	CH_3	83	140–143
22	S	$(CH_3)_2CHCH_2CH_2-$	CH_3	90	78–80
23	$S\rightarrow O$	$(CH_3)_2CHCH_2CH_2-$	CH_3	69	oil
24	$S\begin{smallmatrix}\nearrow O\\ \searrow O\end{smallmatrix}$	$(CH_3)_2CHCH_2CH_2-$	CH_3	100	oil
25	S	$n\text{-}C_5H_{11}-$	CH_3	88	60–62
26	$S\rightarrow O$	$n\text{-}C_5H_{11}-$	CH_3	69	oil
27	$S\begin{smallmatrix}\nearrow O\\ \searrow O\end{smallmatrix}$	$n\text{-}C_5H_{11}-$	CH_3	100	oil
28	S	$(CH_3)_3C-$	CH_3	92	144–146
29	S	$C_2H_5(CH_3)CH-$	CH_3	88	100–102
30	S	$n\text{-}C_6H_{13}-$	CH_3	89	67–69
31	S	$n\text{-}C_7H_{15}-$	CH_3	89	60–62
32	S	$(CH_3)_2CHCH_2$	$n\text{-}C_4H_9$	68	88–91
33	S	$C_6H_5CH_2-$	CH_3	85	100–102
34	$S\rightarrow O$	$C_6H_5CH_2-$	CH_3	98	oil
35	$S\begin{smallmatrix}\nearrow O\\ \searrow O\end{smallmatrix}$	$C_6H_5CH_2-$	CH_3	96	oil
36	S	C_2H_5-	C_2H_5	40	oil
37	O	$(CH_3)_2CH-$	C_2H_5	59	89–90
38	O	$ClCH_2CH-$	CH_3	97	83–86

Example 39.

4'-(Isopropylthio)-3'-nitro-1-methylcyclopropanecarboxanilide

a) *Preparation of 4-(isopropylthio)-3-nitroaniline*

To a stirred solution containing 34.5g (0.20 mole) of 4-chloro-3-nitroaniline and 24g (0.30 mole) of isopropyl mercaptan in 200 ml of DMSO was added dropwise at ambient temperature a solution of 8.0g (0.20 mole) of sodium hydroxide in 10 ml of water. The addition was exothermic to 60°C and the mixture became deep red in colour. After three days, the reaction mixture was poured into water and extracted with methylene chloride. The combined extracts were washed with methylene chloride. The combined extracts were washed with water, dried with anhydrous $MgSO_4$, filtered and concentrated to give 41.1g (97%) of product as a red-brown syrup (39a).

b) *Preparation of 4'-(isopropylthio)-3'-nitro-1-methylcyclopropanecarboxanilide*

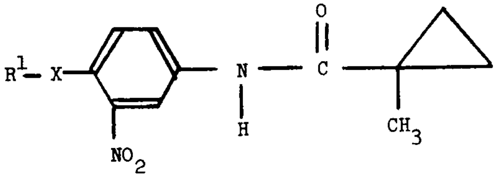
To a solution containing 16.0g (0.75 mole) of (39a) and 7.6g (0.75 mole) of triethylamine in 150 ml of tetrahydrofuran was added dropwise with stirring 8.9g (0.075 mole) of 1-methylcyclopropanecarboxylic acid chloride. This addition was exothermic to 60°C. The mixture was refluxed for one hour, poured over ice water and extracted with ether. The ether extract was dried and concentrated to give 20.7g of a dark red-brown oil. Purification by silica chromatography gave 105g (48%) of product as a light-yellow solid, melting point 92—94°C.

Examples 40—42.

In the manner described in Example 39, additional cyclopropanecarboxanilides listed in Table 2 were prepared.

TABLE 2

Cyclopropanecarboxanilides



Example	X	R¹	Yield (%)	Melting Point, °C
40	$S\rightarrow O$	$(CH_3)_2CH-$	73	143–145
41	$S\genfrac{}{}{0pt}{}{\nearrow O}{\searrow O}$	$(CH_3)_2CH-$	90	103–105
42	O	$(CH_3)_2CH$	47	100–101

Example 43.

4′-(Cyclopropylmethoxy)-3′-chloro-1-methyl-cyclopropanecarboxanilide

a) *Preparation of 3-chloro-4-(cyclopropylmethoxy)-nitrobenzene*

To a solution containing 38.4 g of 3,4-dichloronitrobenzene in 150 ml of DMSO was added 17.3 g of cyclopropylmethanol. This solution was stirred during the dropwise addition of 92 g of sodium hydroxide dissolved in 10 ml of water. This addition was exothermic to 45°C. The mixture was stirred and heated to 75—80°C for 18 hours, then poured over ice water and filtered. The filter cake was recrystallized from methanol to yield 27 g (59%) of light tan solid, melting point, 42—44°C (43a).

b) *Preparation of 3-chloro-4-(cyclopropylmethoxy)aniline*

To a heated mixture containing 26.7 g of (43a) in 300 ml of 5% aqueous acetic acid was added 56 g of iron filings and 15 ml of methanol. The mixture was refluxed and stirred vigorously for one hour. The mixture was filtered while hot and the cooled filtrate was extracted with ether. The ether extract was washed with aqueous sodium bicarbonate, dried, and concentrated to give 22.8 g (99%) of product, melting point 54—55°C (43b).

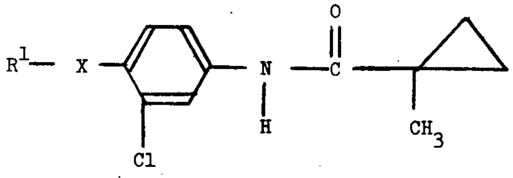
c) *Preparation of 4′-(cyclopropylmethoxy)-3′-chloro-1-methylcyclopropanecarboxanilide*

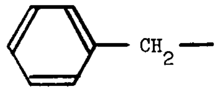
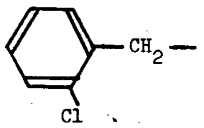
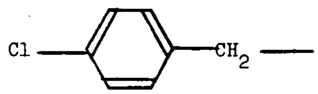
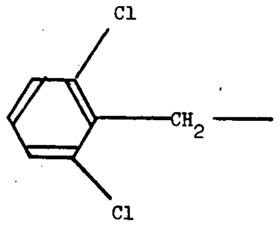
To a solution of 4.9 g of (43b) and 2.5 g of triethylamine in 50 ml of tetrahydrofuran was added dropwise 3.0 g of 1-methylcyclopropanecarboxylic acid chloride. The mixture was refluxed for 30 minutes, poured into ice water, and filtered, and the filtrate was recrystallized from methanol/water (4:1) to give 6.5 g (93%) of white crystalline solid; m.p. 111—113°C.

Examples 44—51.

In the manner described in Example 43 additional cyclopropanecarboxanilides listed in Table 3 were prepared.

TABLE 3
Cyclopropanecarboxanilides



Example	X	R¹	Yield (%)	Melting Point, °C
44	O		89	107–109
45	O		86	146–149
46	O		80	135–137
47	O		95	150–152
48	O	$CH_3OC_2H_2$–	75	74–75
49	O	$(CH_3)_2CH$–	67	107–110
50	O	CH_3	92	128–130
51	O		87	87–89

Example 52.

4′-Piperidinyl-3′-(trifluoromethyl)-1-methyl-cyclopropanecarboxanilide

a) *Preparation of 4-nitro-1-piperidinyl-2-(trifluoromethyl)benzene*

To a solution of 4-nitro-2-(trifluoromethyl)-chlorobenzene in 150 ml DMSO was added dropwise 17g of piperidine. The mixture was stirred at ambient temperature, while protected from air and moisture, for several days, then poured over ice water and extracted with methylene chloride. The extract was washed with cold water, dried and concentrated to give 27.4g (100%) of yellow-brown oil (52a).

b) *Preparation of 4-piperidinyl-3-(trifluoromethyl)aniline*

To about 500 ml of 5% acetic acid heated to 95°C was added 26g of (52a) 17g of iron powder was added portionwise at a rate to maintain reflux without an external source of heat. After the addition, the mixture was stirred while hot, and the cooled filtrate was extracted with ether. The ether extract was washed with 10% aqueous sodium bicarbonate and then with water, dried, and concentrated to 22g (96%) of yellow-brown oil (52b).

c) *Preparation of 4′-piperidinyl-3′-(trifluoromethyl)-1-methylcyclopropanecarboxanilide*

To 11g of (52b) in 75 ml of tetrahydrofuran and 4.6g of triethylamine was added dropwise 5.3g of 1-methyl-cyclopropanecarboxylic acid chloride, the reaction temperature going to 55°C. The mixture was then stirred and heated at 64°C for 1.5 hours, poured over ice water and extracted with ether. The ether extract was washed with water, dried and concentrated to give 13g (88%) of a yellow syrup, which was crystallized from hexane to give 12g (82%) of off-white solid; m.p. 109—11°C.

Example 53.

4′-(Isopropylamino)-3′-(trifluoromethyl)-1-methylcyclopropanecarboxanilide

a) *Preparation of N-isopropyl-4-nitro-2-(trifluoromethyl)aniline*

To a soution of 45g of 3-(trifluoromethyl)-4-chloronitrobenzene in 150 ml of DMSO was added 13g of isopropylamine. The mixture was heated at 80—90°C for two hours, poured over ice water, acidifed and filtered. The filtrate was dried to yield 49.6g of product; m.p. 35—36°C (53a).

b) *Preparation of 4-(isopropylamino)-3-(trifluoromethyl)aniline*

About 565 ml of 5% acetic acid was heated to 90°C and 49.6 g of (53a) was added. 123g of iron powder was added portionwise. The resulting mixture was refluxed for 1 hour, then filtered while hot. The filtrate was cooled and extracted with ether. The ether extract was washed with 10% aqueous sodium bicarbonate and then with water, dried and concentrated to give 38.7g (89%) of product as a light amber oil (53b).

c) *Preparation of 4′-(isopropylamino)-3′-(trifluoromethyl)-1-methylcyclopropanecarboxanilide*

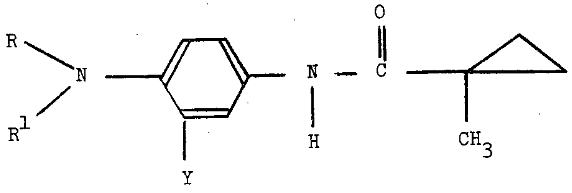
To a solution of about 19g of (53b) in 150 ml of tetrahydrofuran and 8.8g of triethylamine was added 118.5g of 1-methylcycloproppanecarboxylic acid chloride dropwise over 5 minutes at 25—64°C. The reaction mixture was stirred, refluxed for 30 minutes, poured over ice water, and extracted with ether. The ether extract was dried and concentrated, and the residue was crystallized from ether/petroleum ether to give 22g (84%) of grey solid; m.p. 100—102°C.

Example 54—67.

In the manner described in the Examples 52 and 53 additional cyclopropanecarboxanilides listed in Table 4 were prepared.

TABLE 4

Cyclopropanecarboxanilides



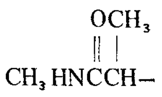
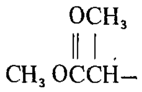
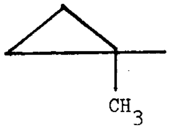
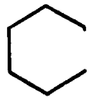
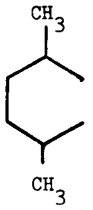
Example	R	R¹	Y	Yield (%)	Melting Point, °C
54	H	 $CH_3HN\overset{O}{\overset{\|}{C}}\overset{CH_3}{\overset{\vert}{C}}H-$	CF_3	58	223–226
55	H	 $CH_3O\overset{O}{\overset{\|}{C}}\overset{CH_3}{\overset{\vert}{C}}H-$	CF_3	90	86–88
56	H	H	CF_3	9	136–141
57	CH_3	CH_3	CF_3	85	89–91
58	CH_3	$(CH_3)_2CH-$	CF_3	84	98–100
59	H	$(CH_3)_3C-$	CF_3	93	105–108
60	H		CF_3	68	126–128
61	H		CF_3	79	120–122
62	C_3H_7	C_3H_7	CF_3	68	74–76

TABLE 4 (Continued)

Example	R	R¹	Y	Yield (%)	Melting Point, °C
63			CF_3	77	102–104
64			CF_3	98	oil
65	H	$(CH_3)_2CH$	NO_2	2	172–174
66	C_3H_7	$(CH_3)_3C-$	NO_2	4	211–214
67	H	C_3H_7	NO_2	31	77–79

Example 68.

4'-(isopropoxy)-3'-(trifluoromethyl)-1-chlorocyclopropanecarboxanilide

To a solution of 10.95g (1b) in 100 ml of tetrahydrofuran and 5.05 g of triethylamine was added dropwise at ambient temperature 6.95g of 1-chlorocyclopropanecarboxylic acid chloride. The reaction mixture was allowed to stand for two days, then concentrated to dryness. The resulting solid was washed with water and recrystallized from 80% aqueous methanol to give 10g (62%) of white solid; m.p. 106—108°C.

Example 69.

4'-isopropoxy-3'-(trifluoromethyl)-1-bromocyclobutanecarboxanilide

a) *Preparation of 1-bromocyclobutanecarboxylic acid chloride.*

A solution containing 82.8 g of ethyl 1-bromocyclobutanecarboxylate and 28 g of potassium hydroxide in 250 ml of 85% aqueous ethanol was refluxed for 15 minutes. The reaction mixture was concentrated to dryness under reduced pressure, treated with cold hydrochloric acid, and extracted with ether. The ether extract was dried and concentrated to give 53 g of a viscous mixture of acids. Treatment of this mixture witrh an excess of thionyl chloride under reflux conditions followed by distillation gave 18 g of a colourless liquid, boiling at 70—75°C (16 mm). Gas-liquid chromatography indicated the presence, in the approximate ratio of 4:1, of 1-bromocyclobutanecarboxylic acid chloride and 1-ethoxycyclobutanecarboxylic acid chloride.

b) *Preparation of 4'-isopropoxy-3'-(trifluoromethyl)-1-bromocyclobutanecarboxanilide*

To a solution containing 10.9 g of (1b) and 5.1 g of triethylamine in 175 ml of tetrahydrofuran was added dropwise at 10—20°C 12.1 g of (73a). After standing overnight, the reaction mixture was concentrated to dryness, washed with water, and extracted with ether. The ether extract was concentrated and the product was purified by silica chromatography. The first fraction comprised 3.0 g (16%) of the 1-ethoxycyclobutane ester as a tan solid, m.p. 48—50°C. The second fraction comprised 2.4 g (13%) of the 1-bromocyclobutane ester as a white solid, m.p. 82—84°C.

Example 70.

4'-(Isopropoxy)-3'-methyl-1-methylcyclopropanecarboxanilide

a) *Preparation of isopropyl ortho-tolyl ether*

To a solution of 54.1 g (0.5 mole) or ortho-cresol in 150 ml of DMSO was added portionwise and with stirring 12 g of 57% sodium hydroxide. This addition was exothermic to 45°C. After 2 hours at ambient temperature, 61.5 g (0.5 mole) of isopropyl bromide in 50 ml of DMSO was added dropwise with stirring. After 2 hours, the reaction mixture was poured into 1000 ml of ice water and extracted with ether. Distillation of the dried extract gave 53.9 g of product as a colorless liquid; b.p. 95—96°C (30 mm) (70a).

b) *Preparation of 2-isopropoxy-5-nitrotoluene*

To a chilled (6°C) solution containing 31.8 g (0.2 mole) of ether (70a) and 28.7 g of acetic anhydride in 200 ml of glacial acetic acid was added dropwise a solution of 13.9 g (0.22 mole) of 90% nitric acid in 100 ml of glacial acetic acid. The reaction mixture was allowed to stand at ambient temperature for 12 hours, poured into water and extracted with methylene chloride. The extract was washed with 5% sodium carbonate, water, dried, and concentrated to give 21.8 g of product as an amber oil (70b).

c) *Preparation of 4'-(isopropoxy)-3'-methyl-1-methylcyclopropanecarboxanilide*

A Parr shaker was charged with 21.8 g (0.11 mole) of (70b) and 2 g of 10% palladium-charcoal catalyst in 150 ml of tetrahydrofuran. The glas cylinder was pressurized with hydrogen (40 pounds) and shaken until hydrogen uptake ceased. The catalyst was removed by filtration. To the resulting solution was added 11.2 g of triethylamine and 12.6 g of 1-methylcyclopropanecarboxylic acid chloride. This addition was exothermic to 35°C. After 1 hour, the reaction mixture was concentrated to dryness and washed with water. Purification by silica chromatography gave 0.8 g of a white crystalline solid; m.p. 99—101°C.

Example 71.

4'-(Dimethylsulphomoyl)-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide

a) *Preparation of 4-nitro-2-(trifluoromethyl)-benzenesulphonyl chloride*

A mixture containing 57.5 g (0.28 mole) of 4-nitro-2-(trifluoromethyl)aniline in 200 ml of concentrated hydrochloric acid was heated to 90°C for 15 minutes and left stirring at ambient temperature for 18 hours. The resulting mixture was chilled to 4° and diazotized with 21.4 g (0.31 mole) of sodium nitrite in 50 ml of water. After 1 hour, the diazonium salt solution was added dropwise over 20 minutes and with stirring to a cold (5%10°C) solution containing 13 g of copper(II) chloride and 64 g of sulphur dioxide in 250 ml of glacial acetic acid. After 2 hours, the mixture was filtered. The filter cake was washed well with water and dried to give 68 g (84%) of product as a light tan solid; m.p. 77—79°C (71a).

b) *Preparation of N,N-dimethyl-4-nitro-2-(trifluoromethyl)benzenesulphonamide*

To a stirred solution containing 14 g (0.05 mole) of the sulphonyl chloride (71a) in 100 ml of tetrahydrofuran was added through a gas-inlet tube an excess of anhydrous dimethylamine causing the internal temperature to rise to 60°C. The mixture was stirred at ambient temperature for ½ hour, poured into 500 ml of ice water and filtered. The filter cake was washed with water and dried to give 14 g (94%) of product as a light tan solid; m.p. 95—97°C (71b).

c) *Preparation of 4-(dimethylsulphamoyl-3-(trifluoromethyl)aniline*

To a heated mixture containing 13.5 g (0.045 mole) of the amide (71b) in 250 ml of 5% acetic acid was added portionwise and with stirring 12 g of iron powder. The mixture was refluxed for ½ hour. The mixture was filtered while hot and the cooled filtrate was extracted with ether. The ether extract was washed with aqueous sodium bicarbonate, dried, concentrated and recrystallized from ether-hexane (1:2) to give 10.1 g (83%) of product as a colourless solid; m.p. 140—142°C (71c).

d) *Prepareation of 4'-(dimethylsulphamoyl-3'-(trifluoromethyl)-1-methylcyclopropanecarboxanilide*

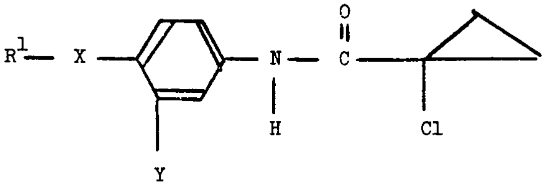
To a solution of 6.7 g (0.025 mole) of the aniline (71c) and 2.5 g of triethylamine in 50 ml of tetrahydrofuran was added dropwise 3.0 g (0.025 mole) of 1-methylcyclopropanecarboxylic acid chloride. The mixture was refluxed for one hour, poured into ice water, filtered and recrystallized from methanol to give 7.5 g (86%) of product as a white crystalline solid; m.p. 153—156°C.

Example 72—88.

In the manner described for the above examples, additional cyclopropanecarboxanilides listed in Tables 5 and 6 were prepared.

TABLE 5

Cyclopropanecarboxanilides



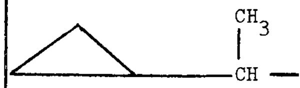
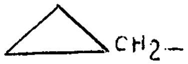
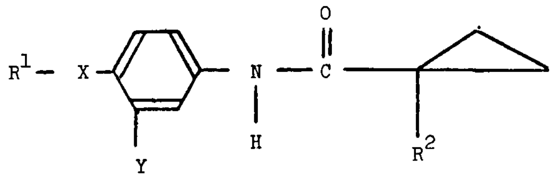
Example	X	Y	R¹	Yield (%)	Melting Point, °C
72	O	CF_3	C_2H_5	82	85–87
73	S	CF_3	C_2H_5	80	56–58
74	O	Cl		67	51–52
75	O	Cl		67	70–72
76	O	Cl	CH_3	76	95–98
77	NCH_3	SO_2CH_3	$(CH_3)_2CH-$	80	145–148
78	NCH_3	CF_3	$(CH_3)_2CH-$	65	55–57
79	O	CF_3	$Cl(CH_2)_2-$	100	oil

TABLE 6

Cyclopropanecarboxanilides



Example	Y	X	R¹	R²	Yield (%)	Melting Point, °C
80	CN	O	$(CH_3)_2$–CH–	CH_3	49	130–132
81	SO_2CH_3	NCH_3	$(CH_3)_2CH$–	CH_3	95	175–177
82	Br	NH	$(CH_3)_2CH$–	CH_3	26	89–90
83	SO_2CH_3	S	C_2H_5–	CH_3	56	130–134
84	CF_3	S	$(CH_3)_2CH$–	Cl	60	62–64
85	CF_3	NCH_3	$(CH_3)_2CH$–	C_2H_5	38	oil
86	Br	O	$(CH_3)_2CH$–	CH_3	32	99–101
87	CN	O	CH_3	CH_3	16	129–131
88	SCH_3	O	C_2H_5	CH_3	1	114–116

Example 89.

Demonstration of herbicidal activity

The pre-emergence herbicidal activity of compounds according to the invention was evaluated by planting seeds of garden cress, downey brome, wild mustard, velvet leaf, soybean, wheat, and cotton in test tubes, nominally measuring 25 x 200 millimeters, containing soil treated with the test compound at the rates of 0.1 and 1 mg per tube designated in Table I at Rates I and II, respectively. The planted soil was held under controlled conditions of temperature, moisture, and light for 13 to 14 days. The amount of germination and growth in each tube were evaluated on a 0 to 9 scale, 0 rating indicating no effect, 9 death of the seedlings or no germination.

The post-emergence activity of the compounds according to this invention was evaluated by spraying 7-day old crabgrass plants, 10-day old pigweed plants, 7-day old downey brome plants, 10-day old wild mustard, 10-day old velvet leaf, 14-day old soybean plants, 7-day old wheat and 14-day old cotton plants to run-off with a liquid formulation of the test compound at the rates of 0.62 milliliter of an 0.05% solution designated Rate I in Table I, and 0.56 milliliter of an 0.5% solution designated Rate II in Table I. The sprayed plants were held under controlled conditions for 10 to 11 days and the effect of the test compound was then evaluated visually, the results being rated on the 0 to 9 scale described above.

The results of the pre- and post-emergence tests are summarized in Table I.

The herbicidal activity of compounds according to the present invention was further determined with respect to several common species of weeds, by spraying a formulation of the test compound on the soil in which the weed seeds had been planted (pre-emergence test) or onto the foliage of the plants (post-emergence test). In each series of tests, the plants were grown in narrow trays and sprayed with the appropriate chemical. The solution of the test compound was sprayed over the tray, from one end to the other, the concentration of the test compound in the formulation varying logarithmically from a higher value at one end of the band to a lower value at the other end of the band. The effect of the test compound was evaluated visually and reported as the nominal rate of application, in pounds of test compound per acre (1.12 kg/ha) of the soil band, at which 90% inhibition of the growth of the weeds occurred, this being referred to as the 90% growth inhibition dosage (GID_{90}). Results of the pre-emergence and post-emergence tests, as well as the weed species involved, are set out in Tables II and III.

TABLE I

RESULTS OF THE HERBICIDE ACTIVITY SCREEN

	PRE-EMERGENCE (SOIL)											POST-EMERGENCE (FOLIAR)													
	Garden Cress		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton	Crab-grass		Pigweed		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton
Compound of Example No.	I	II	I	II	I	II	I	II	II	II	II	I	II	I	II	I	II	I	II	I	II	II	II	II
8	9	9	3	7	9	9	6	9	8	6	5	7	9	8	9	4	9	–		5	9	5	2	3
9	7	7	4	5	8	9	4	8	3	5	0	7	7	9	9	7	8	7	9	6	9	6	5	2
1	8	8	0	2	9	9	0	1	4	3	0	8	9	7	9	8	9	8	9	9	9	7	6	9
10	6	9	1	4	9	9	4	8	3	4	0	0	2	0	7	0	2	1	9	2	7	2	3	2
2	0	8	0	0	0	2	0	0	0	0	0	8	9	7	8	3	3	9	9	3	9	4	2	7
3	7	8	0	0	6	8	0	0	0	0	0	7	7	4	8	7	7	8	9	6	9	4	9	7

TABLE I

RESULTS OF THE HERBICIDE ACTIVITY SCREEN

	PRE-EMERGENCE (SOIL)											POST-EMERGENCE (FOLIAR)												
	Garden Cress		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton	Crab-grass		Pigweed		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton
Compound of Example No.	I	II	I	II	I	II	I	II	II	II	II	I	II	I	II	I	II	I	II	I	II	II	II	II
12	0	8	0	0	0	0	0	0	0	0	0	3	6	3	8	4	6	7	9	9	9	5	4	8
11	0	8	0	0	0	9	0	0	0	0	0	7	8	8	9	0	8	8	9	9	9	6	1	9
13	0	4	0	0	0	0	0	0	0	2	0	5	6	0	2	2	7	8	8	7	8	6	0	8
4	3	9	0	2	8	9	0	5	7	5	0	4	6	8	9	1	5	7	9	7	8	6	0	7
5	9	9	1	5	9	9	7	7	7	7	0	0	2	7	8	0	2	0	4	3	5	4	6	7
6	9	9	3	6	9	9	7	7	6	7	0	0	1	6	8	0	1	6	9	7	8	7	5	7
7	9	9	0	3	9	9	0	8	0	0	3	5	7	9	9	2	7	8	9	7	8	8	9	8

TABLE I

RESULTS OF THE HERBICIDE ACTIVITY SCREEN

	PRE-EMERGENCE (SOIL)											POST-EMERGENCE (FOLIAR)												
	Garden Grass		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton	Crab-grass		Pigweed		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton
Compound of Example No.	I	II	I	II	I	II	I	II	II	II	II	I	II	I	II	I	II	I	II	I	II	II	II	II
14	9	9	6	6	9	9	9	9	7	7	6	1	4	5	9	1	5	5	9	9	9	6	6	7
15	9	9	6	6	9	9	9	9	7	6	0	4	6	9	9	4	9	5	9	9	9	6	6	7
16	0	0	0	0	0	0	0	0	0	0	0	6	8	9	9	4	5	9	9	8	8	8	4	8
17	9	9	5	7	9	9	5	9	7	5	4	2	7	9	9	3	5	7	9	8	8	8	7	8
18	8	9	0	7	9	9	7	9	6	4	6	7	7	9	9	0	5	8	9	8	8	5	5	9
26	7	9	0	4	9	9	0	9	1	1	0	0	0	0	5	0	4	7	9	3	8	8	4	9
27	6	9	0	3	9	9	0	7	1	1	0	0	0	0	1	2	3	8	9	8	9	9	8	8

TABLE I

RESULTS OF THE HERBICIDE ACTIVITY SCREEN

	PRE-EMERGENCE (SOIL)											POST-EMERGENCE (FOLIAR)													
	Garden Cress		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton	Crab-grass		Pigweed		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton
Compound of Example No.	I	II	I	II	I	II	I	II	II	II	II	I	II	I	II	I	II	I	II	I	II	II	II	II
23	9	9	0	8	9	9	7	9	5	5	0	0	2	1	7	0	3	8	8	8	9	9	4	9
24	8	8	0	4	9	9	3	9	2	2	0	0	6	3	9	4	7	9	9	9	9	9	8	9
19	9	9	5	7	8	9	8	9	8	6	0	9	9	9	9	8	9	9	9	9	9	7	3	8
20	9	9	7	7	9	9	9	9	8	7	7	2	8	9	9	9	9	9	9	9	9	5	2	9
21	9	9	8	8	9	9	9	9	8	8	0	2	8	9	9	8	9	9	9	9	9	5	1	8
25	0	1	0	0	0	0	0	0	0	0	0	6	6	3	9	2	2	8	9	8	8	8	2	9

TABLE I

RESULTS OF THE HERBICIDE ACTIVITY SCREEN

	PRE-EMERGENCE (SOIL)										POST-EMERGENCE (Foliar)													
	Garden Cress		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton	Crab-grass		Pigweed		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton
Compound of Example No.	I	II	I	II	I	II	I	II	II	II	II	I	II	I	II	I	II	I	II	I	II	II	II	II
30	0	0	0	0	0	0	0	0	0	0	0	7	8	4	9	0	0	8	9	3	7	7	2	9
31	0	0	0	0	0	0	0	0	0	0	0	5	5	7	7	0	9	7	7	5	8	8	2	9
29	5	8	0	0	0	8	0	0	0	0	0	7	8	4	9	0	5	8	8	8	9	9	2	9
28	0	0	0	0	0	0	0	0	0	0	0	0	0	0	7	5	5	6	7	3	9	8	3	4
22	0	1	0	0	0	0	0	0	0	0	0	6	7	3	8	1	1	9	9	8	8	8	0	9
33	0	0	0	0	-	-	0	0	5	0	0	7	9	9	9	5	5	-	-	6	9	4	0	5
34	0	8	0	2	-	-	0	6	0	0	0	2	8	8	9	3	7	-	-	9	9	3	0	3

TABLE I

RESULTS OF THE HERBICIDE ACTIVITY SCREEN

	PRE-EMERGENCE (SOIL)											POST-EMERGENCE (FOLIAR)												
	Garden Cress		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton	Crab-grass		Pigweed		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton
Compound of Example No.	I	II	I	II	I	II	I	II	II	II	II	I	II	I	II	I	II	I	II	I	II	II	II	II
35	2	9	0	0	-	-	0	3	0	0	0	1	6	8	9	4	7	-	-	3	4	0	0	0
36	2	2	0	0	-	-	0	0	0	0	0	0	7	4	7	0	3	-	-	0	7	5	0	0
37	7	7	0	0	-	-	0	0	0	0	0	3	3	0	8	0	3	-	-	3	6	4	0	2
52	0	0	0	0	-	-	0	0	0	0	0	0	0	6	9	0	0	-	-	7	9	4	0	0
63	3	6	0	0	-	-	0	4	0	0	0	2	4	4	9	2	7	-	-	4	9	2	0	0
64	0	1	0	0	-	-	0	0	0	0	0	0	8	1	9	0	7	-	-	5	8	4	0	0
80	9	9	0	4	-	-	0	3	5	5	0	0	0	0	4	0	0	-	-	0	0	2	2	0

TABLE I

RESULTS OF THE HERBICIDE ACTIVITY SCREEN

	PRE-EMERGENCE (SOIL)											POST-EMERGENCE (FOLIAR)												
	Garden Cress		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton	Crab-grass		Pigweed		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton
Compound of Example No.	I	II	I	II	I	II	I	II	II	II	II	I	II	I	II	I	II	I	II	I	II	II	II	II
53	8	8	2	6	9	9	2	8	-	2	5	5	5	9	9	2	5	8	9	9	9	-	2	8
54	7	8	2	4	8	8	3	7	6	0	0	0	0	0	0	0	0	0	3	0	0	0	0	0
56	3	7	1	2	7	8	3	8	2	0	0	0	0	0	5	0	0	0	2	0	5	2	0	2
55	0	8	2	6	3	9	1	8	0	6	0	0	0	0	0	0	0	0	2	0	0	0	7	0
60	2	8	0	0	9	9	0	7	0	9	0	0	7	3	8	1	1	7	9	6	9	9	0	9
57	8	9	3	8	9	9	5	9	8	4	0	9	9	8	9	5	7	9	9	8	9	9	0	4
61	2	8	0	3	0	9	2	6	9	3	0	8	9	9	9	5	7	9	9	9	9	8	4	7

TABLE I

RESULTS OF THE HERBICIDE ACTIVITY SCREEN

	PRE-EMERGENCE (SOIL)										POST-EMERGENCE (FOLIAR)												
	Garden Cress		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton	Crab-grass		Pigweed		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton
Compound of Example No.	I	II	I	II	I	II	I	II	II	II	II	I	II	I	II	I	II	I	II	I	II	II	II	II
58	2	8	0	0	9	9	3	5	0	0	2	9	9	9	9	8	9	9	9	9	9	6	5	8
59	8	9	0	0	9	9	6	7	0	0	0	9	9	9	9	8	9	9	9	9	9	3	0	8
62	2	4	0	0	0	2	2	2	0	0	2	8	9	8	9	2	8	9	9	8	9	7	2	2
39	3	6	0	0	3	8	3	7	0	0	0	7	9	8	9	3	6	8	9	9	9	2	0	8
40	3	9	3	7	4	9	0	9	0	5	0	0	2	0	9	0	0	4	8	0	9	1	0	6
41	9	9	2	7	9	9	6	9	1	0	0	0	6	9	9	5	7	9	9	9	9	2	0	8

TABLE I

RESULTS OF THE HERBICIDE ACTIVITY SCREEN

	PRE-EMERGENCE (SOIL)											POST-EMERGENCE (FOLIAR)												
	Garden Cress		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton	Crab-grass		Pigweed		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton
Compound of Example No.	I	II	I	II	I	II	I	II	II	II	II	I	II	I	II	I	II	I	II	I	II	II	II	II
42	8	9	5	8	9	9	8	9	7	2	0	5	8	6	9	5	8	6	9	8	9	2	4	0
65	8	8	4	5	8	8	5	6	0	5	0	8	8	8	8	3	3	9	9	9	9	5	0	3
66	0	9	0	0	0	0	0	0	0	0	0	8	9	6	9	5	8	7	9	2	9	1	0	6
67	5	5	0	0	-	-	0	0	0	3	0	2	3	2	5	0	0	-	-	4	4	2	0	0
45	0	0	-	-	-	-	-	-	0	-	3	7	8	8	8	-	-	-	-	-	-	5	-	8
44	6	6	-	-	-	-	-	-	0	-	0	7	9	8	9	-	-	-	-	-	-	6	-	9
48	9	9	-	-	-	-	-	-	7	-	7	0	7	4	5	-	-	-	-	-	-	6	-	8

TABLE I

RESULTS OF THE HERBICIDE ACTIVITY SCREEN

	PRE-EMERGENCE (SOIL)											POST-EMERGENCE (FOLIAR)												
	Garden Cress		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton	Crab-grass		Pigweed		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton
Compound of Example No.	I	II	I	II	I	II	I	II	II	II	II	I	II	I	II	I	II	I	II	I	II	II	II	II
46	0	0	-	-	-	-	-	-	0	-	0	6	8	8	9	-	-	-	-	-	-	7	-	8
47	0	0	-	-	-	-	-	-	0	-	0	7	7	6	9	-	-	-	-	-	-	5	-	7
43	7	8	0	0	6	8	0	0	0	0	0	7	7	4	8	7	7	8	9	6	9	4	9	7
50	8	8	4	8	-	-	7	9	7	4	9	2	3	1	7	0	4	-	-	5	8	7	0	6
51	8	9	0	4	-	-	3	8	0	1	0	3	9	9	9	7	8	-	-	9	9	7	0	7
68	2	4	0	0	-	-	0	0	3	0	0	0	3	6	7	0	0	-	-	5	5	2	0	0
72	0	0	0	0	-	-	0	0	0	0	0	0	5	4	6	0	7	-	-	1	5	0	0	0

TABLE I

RESULTS OF THE HERBICIDE ACTIVITY SCREEN

	PRE-EMERGENCE (SOIL)											POST-EMERGENCE (FOLIAR)													
	Garden Cress		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton	Crab-grass		Pigweed		Downey Brome		Wild Mustard		Velvet Leaf		Soybean	Wheat	Cotton
Compound of Example No.	I	II	I	II	I	II	I	II	II	II	II	I	II	I	II	I	II	I	II	I	II	II	II	II
73	2	9	0	0	-	-	0	0	0	0	0	3	8	8	9	2	5	-	-	5	7	1	0	2
78	3	6	0	0	-	-	0	0	0	4	0	0	6	0	7	0	4	-	-	0	7	1	0	0
74	1	5	0	0	-	-	0	0	0	0	0	1	4	3	9	0	4	-	-	6	8	2	0	7
75	6	7	0	0	-	-	0	0	0	0	0	5	7	4	9	5	7	-	-	3	8	2	0	0
76	7	9	0	0	-	-	0	7	0	0	0	2	7	2	8	0	7	-	-	3	9	4	0	2
49	-	-	0	0	0	0	0	0	0	-	0	5	6	0	2	2	7	8	8	7	8	6	-	8

TABLE II

Results of Post-Emergence Foliar Application Herbicide Rate Evaluation Screen

GID 90

Compound of Example No.	Yellow Foxtail	Fall-Panicum	Crabgrass	Pigweed	Mustard	Velvet Leaf	Downey Brome	Barnyard grass
8	0.32	1.0	0.7	0.4	0.4	>2.0	–	–
9	<0.2	0.84	0.52	<0.2	<0.2	0.24	–	–
1	<0.2	0.52	<0.2	<0.2	<0.2	<0.2	–	–
2	2.0	>2.0	>2.0	<0.2	<0.2	>2.0	–	–
3	<0.2	1.0	0.3	0.3	<0.2	0.48	–	–
12	2.0	1.34	>2.0	2.0	0.22	0.44	>2.0	>2.0
11	0.84	1.34	1.0	0.92	0.22	0.3	2.0	1.6
13	>2.0	>2.0	>2.0	1.34	<0.22	1.0	>2.0	>2.0
30	1.0	>2.0	0.52	0.24	<0.22	2.0	>2.0	1.34
31	>2.0	>2.0	1.0	<0.22	0.22	>2.0	>2.0	1.5
29	1.36	1.0	1.36	0.26	<0.22	0.3	>2.0	>2.0
22	1.36	>2.0	1.34	0.22	0.22	1.0	>2.0	1.36
27	>2.0	>2.0	1.5	0.22	<0.22	0.7	>2.0	>2.0
23	0.7	0.7	1.34	0.4	0.22	<0.22	>2.0	0.7
24	0.84	1.36	0.70	0.64	<0.22	<0.22	1.34	0.26

TABLE II (Continued)

Component of Example No.	Yellow Foxtail	Fall-Panicum	Crabgrass	Pigweed	Mustard	Velvet Leaf	Downey Brome	Barnyard grass
44	>2.0	1.36	>2.0	<0.2	<0.2	>2.0	–	–
46	>2.0	>2.0	>2.0	<0.2	<0.2	>2.0	–	–
47	>2.0	>2.0	>2.0	>0.2	1.0	>2.0	–	–
48	>2.0	>2.0	>2.0	>2.0	2.0	1.0	–	–
43	1.36	1.64	0.84	0.7	0.26	1.0	>2.0	0.52
53	<0.22	0.58	<0.22	<0.22	<0.22	<0.22	1.8	<0.22
4	1.64	1.34	2.0	>2.0	1.0	1.0	>2.0	>2.0
6	>2.0	>2.0	>2.0	1.12	1.64	1.64	>2.0	>2.0
7	<0.22	0.52	0.26	<0.22	<0.22	0.3	1.12	0.22
15	0.7	0.92	0.64	<0.22	1.36	0.92	>2.0	0.84
16	<0.22	0.7	2.0	<0.22	<0.22	<0.22	2.0	0.22
17	0.76	0.76	2.0	0.58	0.76	0.48	>2.0	0.7
18	0.76	0.4	1.34	<0.22	0.36	<0.22	2.0	0.52

(The symbol < means "less than")
(The symbol > means "greater than")

TABLE III

Results of Pre-emergence Soil Application

GID_{90}

Compound of Example No.	Soil Type	Yellow-Foxtail	Fall-Panicum	Crabgrass	Pigweed	Wild Mustard	Velvet Leaf	Downey Brome	Barnyard Grass
4	Webster	>2.0	>2.0	>.20	1.34	>2.0	>2.0	>2.0	>2.0
	Hanford	>1.0	>1.0	>1.0	.58	0.7	>1.0	>1.0	>1.0
6	Webster	>2.0	>2.0	>2.0	1.5	>2.0	>2.0	>2.0	>2.0
	Hanford	>1.0	>1.0	>1.0	0.92	0.7	>1.0	>1.0	>1.0
7	Webster	>2.0	<1.0	>2.0	<1.0	<1.0	>2.0	>2.0	>2.0
	Hanford	>1.0	0.44	>1.0	<0.22	0.36	>1.0	>1.0	1.0
17	Webster	>2.0	1.12	>2.0	<1.0	<1.0	1.36	>2.0	>2.0
	Hanford	>1.0	1.0	>1.0	0.7	0.3	0.76	>1.0	>1.0
18	Webster	>2.0	<1.0	>2.0	<1.0	<1.0	1.64	>2.0	>2.0
	Hanford	>1.0	0.52	>1.0	0.26	0.7	0.76	>1.0	>1.0

(The symbol < means "less than")

(The symbol > means "greater than")

In many instances the compounds according to the invention possess a selective action against weeds in crop plant cultures. For example, control of grasses and broadleaf weeds in grain crops such as wheat can be achieved by post-emergence application of such compounds according to the invention as:

4′-(sec-butoxy)-3′-(trifluoromethyl)-1-methylcyclopropanecarboxanilide.

4′-(isobutoxy)-3′-(trifluoromethyl)-1-methylcyclopropanecarboxanilide,

4′-(isobutylsulphonyl)-3′-(trifluoromethyl)-1-methylcyclopropanecarboxanilide, or

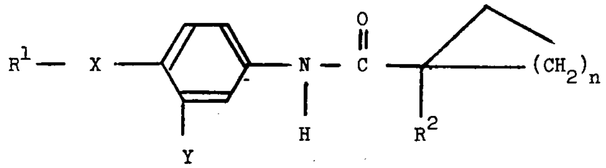
4′-(ethylthio)3′-(trifluoromethyl)-1-methylcyclopropanecarboxanilide

Control of weeds in soybean crops is an example of the selective herbicidal activity of 4′-(isopropylamino)-3′-(trifluoromethyl)-1-methylcyclopropanecarboxanilide or 4′-(isopropylthio)-3′-(trifluoromethyl)-1-methylcyclopropanecarboxanilide.

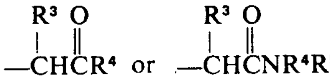
The above species and/or other species of the invention have likewise shown post-emergence, and in some cases, pre-emergence selective activity for peanuts, grain sorghum, cotton, rice, corn or alfalfa.

WHAT WE CLAIM IS:—

1. A compound of the general formula:

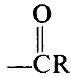


wherein X represents an O, S, SO, SO_2 or —NR^3 moiety; Y represents a flurorine, chlorine or bromine atom, a cyano, nitro, $C(R^3)=NOR^3$ or $C(O)R^3$ group, or a group —Zp-alkyl, in which the alkyl portion contains from 1 to 6 carbon atoms and can be substituted by one or more fluorine, clorine or bromine atoms and Z represents an O, S, SO or SO_2 group; R^1 represents an alkyl group of from 1 to 6 carbon atoms, optionally substituted by one or more fluorine, chlorine or bromine atoms or represents an alkoxyalkyl group in which each alkyl group contains from 1 to 6 carbon atoms, a cycloalkyl group (as hereinafter defined) having from 3 to 7 carbon atoms in the ring, an aralkyl group of from 7 to 9 carbon atoms optionally ring-substituted by one or two fluorine, chlorine or bromine atoms, or by an alkyl group of from 1 to 4 carbon atoms, or a group of the formula:



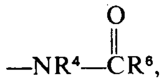
$$-\overset{\overset{R^3}{|}}{C}H\overset{\overset{O}{\|}}{C}R^4 \text{ or } -\overset{\overset{R^3}{|}}{C}H\overset{\overset{O}{\|}}{C}NR^4R^5;$$

and, when X represents the group NR^3 then R^1 can also represent a hydrogen atom or a group



$$-\overset{\overset{O}{\|}}{C}R^6$$

or —SO_2R^6; and when X represents the group SO_2 then R^1 can also represent a group —NH^4R^5,



$$-NR^4-\overset{\overset{O}{\|}}{C}R^6,$$

or —$NR^4SO_2R^6$; R^2 represents an alkyl group of from 1 to 6 carbon atoms, a fluorine, chlorine or bromine atom, or an alkylthio group having from 1 to 6 carbon atoms; with the proviso that when Y represents a NO_2 group, and X represents an O-moiety then R^1 does not represent a methyl group; R^3, R^4 and R^5 each independently represents a hydrogen atom or an alkyl or cycloalkyl (as hereinafter defined) group of up to 6 carbon atoms and R^5 can also represent an alkoxy group of up to 6 carbon atoms; when X represents the group NR^3 then R^1 and R^3 together can form a part of a heterocyclic ring: when R^1 represents the group NR^4R^5 then R^4 and R^5 together can form a part of a heterocyclic ring; R^6 represents an alkyl group of from 1 to 6 carbon atoms or an aryl or aralkyl group of from 6 to 10 carbon atoms optionally substituted by one or more fluorine, chlorine or bromine atoms; n is 1 or 2; and p is 0 or 1.

2. A compound according to claim 1, in which n is 1.

3. A compound acording to either claim 1 or claim 2, wherein R^2 represents a chlorine or bromine atom or a methyl group.

4. A compound according to claim 3, wherein R^2 represents a methyl group.

5. A compound according to any one of claims 1 to 4, wherein Y represents a chlorine atom or a methyl, trifluoromethyl or nitro group.

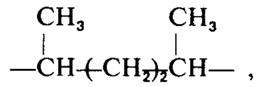
6. A compound according to claim 5, wherein Y represents a trifluoromethyl group.

7. A compound according to any one of claims 1 to 6, wherein R^1 represents an alkyl group of 1—4 carbon atoms or a cycloalkyl group.

8. A compound according to claim 7, wherein R^1 represents a methyl, ethyl, isopropyl, tert.-butyl, cyclopropylmethyl or methylcyclopropyl group.

9. A compound according to claim 8, wherein R^1 represents an isopropyl or tert.-butyl group.

10. A compound as claimed in any one of claims 1 to 6 in which either X represents NR^3 and R^1 and R^3 together represent $-CH_2-(CH_2)_m-CH_2-$ where m is 2 or 3,



$$-\underset{}{\overset{CH_3}{\overset{|}{C}}}H-(CH_2)_2-\overset{CH_3}{\overset{|}{C}}H- ,$$

or $-CH_2.CH_2.O.CH_2.CH_2-$,or R^1 represents $-NR^4R^5$ in which R^4 and R^5 together represents one of these groups.

11. A compound according to any one of claims 1 to 6, wherein R^3, R^4 and R^5 each independently represents a hydrogen atom or a methyl, ethyl or methylcyclopropyl group, or R^3 and R^4 have one of these meanings and R^5 represents a methoxy or ethoxy group.

12. A compound according to any one of claims 1 to 6, wherein X represents a group NR^3 in which R^3 represents an isopropyl,n-propyl or tert.-butyl group.

13. A compound according to either claim 1 or claim 2 wherein X represents a trifluoromethyl group; and R^1 represents an alkyl group of 1—4 carbon atoms, a cyclopropylmethyl or a 1-methylcyclopropyl group.

14. A compound according to either claim 1 or claim 2, wherein X represents an S, SO, or SO_2-moiety; Y represents a trifluoromethyl group; R^2 represents a methyl group; and R^1 represents an alkyl group of up to 4 carbon atoms.

15. A compound according to either claim 1 or claim 2, wherein X represents a group NR^3 wherein R^3 represents a hydrogen atom or an alkyl group of from 1 to 4 carbon atoms; Y represents a trifluoromethyl group; and R^1 represents an alkyl group of from 1 to 4 carbon atoms.

16. A compound according to either claim 1 or claim 2, wherein Y represents a nitro group, R^1 represents an alkyl group of 2—4 carbon atoms and X represents an O, S, SO or SO_2 moiety.

17. A compound according to claim 16, wherein R^1 represents an isopropyl group.

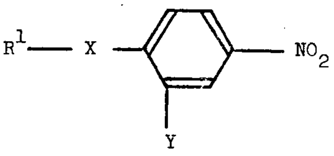
18. 4′ - (Isopropylamino) - 3′ - trifluoromethyl - 1 - methyl - cyclopropanecarboxanilide.

19. 4′-(Isopropoxy)-3′-trifluoromethyl-1-methylcyclopropanecarboxanilide.

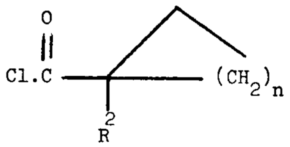
20. 4′-(Isopropylthio)-3′-trifluoromethyl-1-methylcyclopropanecarboxanilide.

21. A compound according to claim 1 and named in any one of Examples 2 to 6, 8 to 52 and 54 to 88 herein.

22. A process for the preparation of a compound as claimed in claim 1, which comprises reacting a 3,4-disubstituted aniline of formula:



with a cycloalkanecarboxylic acid chloride of the formula:



where X, Y, R^1, R^2 and n have the meanings given in claim 1, in the presence of a base.

23. A compound as claimed in claim 1, whenever prepared by a process as claimed in claim 22.

24. A herbicidal composition comprising as active ingredient a compound as claimed in any one of claims 1 to 21 and 23, and a carrier or a surface-active agent or both a carrier and a surface-active agent.

25. A method of controlling undesired plant growth at a locus which comprises applying to the locus a herbicidally effective amount of a compound as claimed in any one of claims 1 to 21 and 23, or a composition as claimed in claim 24.

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