

PATENT SPECIFICATION

(11) 1 505 249

1 505 249

- (21) Application No. 13028/74 (22) Filed 23 March 1974
 (23) Complete Specification filed 19 March 1975
 (44) Complete Specification published 30 March 1978
 (51) INT CL² C07C 87/62; A01N 9/14, 9/16, 9/20; C07C 143/80, 147/12
 (52) Index at acceptance
 C2C 200 220 227 22Y 30Y 311 31Y 323 32Y 332 611 620
 631 660 661 694 791 79Y LP
 (72) Inventor HERSCHEL DONOVAN PORTER



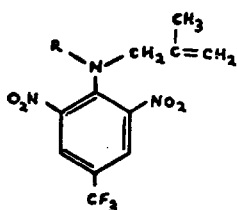
(54) IMPROVEMENTS IN OR RELATING TO HERBICIDES

(71) We, ELI LILLY AND COMPANY, a corporation of the State of Indiana, United States of America, having a principal place of business at 307 East McCarty Street, City of Indianapolis, State of Indiana, United States of America, 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:—

This invention relates to herbicides, more particularly to a class of novel 4 - substituted - 2,6 - dinitroanilines, to methods of preparing this novel class of compounds, to herbicidal compositions containing the novel dinitroanilines and to methods of weed control using the compounds of the invention.

The compounds of the invention have been found to have selective herbicidal activity; i.e. whilst they are phytotoxic to many classes of weeds, they are not harmful to important crops such as cotton, soybeans or edible beans. Accordingly, the compounds of the invention can be used for selective weed control in such crops.

According to one aspect of the present invention, there is provided a 2,6-dinitroaniline of general formula (I)



(I)

wherein R represents an ethyl, propyl or allyl group.

The new compounds of the invention are 2,6-dinitroanilines characterized by a 2-methylamino substituent. The other amino substituent is chosen from ethyl, propyl and allyl.

The compound in which R is ethyl is preferred.

In the interest of clarity, all of the compounds named herein are named as 2,6-dinitroanilines, even though some compounds might

be named otherwise according to alternative rules of organic chemical nomenclature.

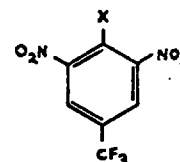
The present invention also provides

(a) A herbicidal composition comprising a compound of formula (I) in association with at least one inert carrier or diluent therefor.

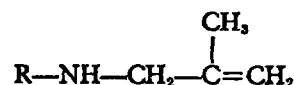
(b) A method of preparing a herbicidal composition as defined in (a) wherein the compound of formula (I) is admixed with at least one inert carrier therefor.

(c) A method of inhibiting the growth of weeds and/or preventing the germination of weed seeds in cropland, which comprises applying a herbicidally-effective amount of a compound of formula (I), to the cropland before the emergence of the weeds.

(d) A method of preparing a compound of formula (I) which comprises reacting a dinitrobenzene of formula



wherein X is a halogen atom preferably a chlorine atom with an amine of the formula



wherein R is as defined above.

The synthesis is preferably performed in an organic solvent, or in an aqueous alkaline medium, at a temperature from 0°C. to 100°C. or higher. Reaction times typically range from one hour to 24 hours, depending on the temperature.

All of the compounds used as starting materials in the synthesis of the compounds of this invention are commercially available, or can be prepared by procedures known in the chemical literature.

Useful organic solvents include aromatic hydrocarbons, such as benzene and toluene, alcohols, such as ethanol and isopropanol, and

117 FEB 1981

such other inert reaction solvents as tetrahydrofuran and dimethylsulfoxide. An acid acceptor such as triethylamine should be added to the organic solvent to absorb the hydrogen halide (e.g. HCl) liberated by the reaction. The reaction also goes well in aqueous alkaline media; the base used is preferably sodium carbonate, although other bases such as sodium hydroxide or potassium carbonate are also useful.

The reaction temperature may be varied from 0°C. to 100°C., or even higher when high-pressure equipment is used. It is most convenient to run the reaction at the reflux temperature of the reaction mixture. No side reactions or thermal degradation result from conducting the reactions at elevated temperatures.

In general, the reactions approach completion at rates dependent on the temperature. Thus, the rate of reaction can be increased by using a high-boiling reaction solvent and operating at reflux temperature. One hour is sufficient to produce a significant yield of any of the compounds. Even at low temperatures, 24 hours is sufficient to approach a maximum yield of any of the compounds.

The following Examples illustrate the preparation of compounds of the invention.

30

Example 1.

N - Allyl - *N* - methallyl - 4 - trifluoromethyl - 2,6 - dinitroaniline

35

To 67.5 g. (0.25 mole) of 4 - chloro - 3,5 - dinitro - α,α,α - benzotrifluoride in 500 ml. of commercial absolute ethanol was added 33.3 g. (0.30 mole) of *N* - allylmethallylamine and 30 g. (0.30 mole) of triethylamine. The mixture was heated under reflux overnight and cooled to room temperature.

40

The reaction product mixture was poured into 800 ml. of water and extracted with 700 ml. of diethyl ether. The ether layer was washed with 3 $\times$ 250 ml. portions of dilute aqueous hydrochloric acid and once with water. The

45

combined aqueous layers were extracted with 500 ml. of diethyl ether. This ether extract was washed once with dilute aqueous hydrochloric acid and once with water. The combined ether layers were dried over anhydrous

50

magnesium sulfate and the drying agent filtered off. The filtrate was concentrated to dryness *in vacuo*. The crystalline residue was dissolved in 400 ml. of hot petroleum ether (b.p. 60—71°C.), filtered, and the filtrate chilled at -5°C. Yellow platelets formed which were filtered off and air dried. The platelets formed which were filtered off and air dried. The platelets weight 58.5 g., had a melting point of 53—54°C., and were confirmed by elemental microanalysis as *N* - allyl - *N* - methallyl - 4 - trifluoro - methyl - 2,6 - dinitroaniline.

60

Calculated for $C_{14}H_{14}F_3N_3O_4$:

	Theoretical	Found
C	48.70	48.81
H	4.09	4.05
N	12.17	12.05

65

Example 2.

N - Ethyl - *N* - methallyl - 4 - trifluoromethyl - 2,6 - dinitroaniline

70

To 540 g. (2.0 mole) of 4 - chloro - 3,5 - dinitrobenzotrifluoride in 3.5 l. of commercial absolute ethanol was added 240 g. (2.4 mole) of *N*-ethylmethallylamine and 240 g. (2.4 mole) of triethylamine. The mixture was heated under reflux for two hours and allowed to stand at room temperature for about 20 hours. The reaction product mixture was diluted with an equal volume of water and extracted in several portions with 5 l. of diethyl ether. The combined ether extracts were washed consecutively with water, twice with dilute aqueous hydrochloric acid, and again with water. The aqueous layers were extracted with diethyl ether and the ether layer was washed with water. The combined ether layers were dried over anhydrous magnesium sulfate. The drying agent was filtered off and the filtrate was concentrated *in vacuo* to dryness. The residue was recrystallized two times from 1500 ml. of petroleum ether (b.p. 60—71°C.). There was obtained yellow crystals having a melting point of about 54—57°C. and weighing 209 g. NMR analysis identified the product as *N* - ethyl - *N* - methallyl - 4 - trifluoromethyl - 2,6 - dinitroaniline. Elemental microanalysis produced the following results:

75

80

85

90

95

Calculated for $C_{13}H_{14}F_3N_3O_4$:

	Theoretical	Found
C	46.85	46.79
H	4.23	4.01
N	12.61	12.58

100

Additional material was obtained by concentrating the filtrates and recrystallizing the crude material thereby obtained to yield product having a melting point of 54—57°C. and weighing 79 g. The total yield then was 627 g.

105

Example 3.

N - Methallyl - *N* - *n* - propyl - 4 - trifluoromethyl - 2,6 - dinitroaniline

110

To 270 g. (1.0 mole) of 4 - chloro - 3,5 - dinitrobenzotrifluoride in 1.5 l. of commercial absolute alcohol was added 135.6 g. (1.2 mole) of *N* - *n* - propylmethallylamine and 120 g. (1.2 mole) of triethylamine. The mixture was heated under reflux for about two hours. The reaction product mixture was poured into an equal volume of water and extracted with 2 l. of diethyl ether. The aqueous layer was extracted twice more with 500 ml. portions of diethyl ether. The combined ether layers were washed twice with dilute aqueous hydrochloric

115

120

acid and once with water. The ether solution was dried over anhydrous magnesium sulfate. The drying agent was filtered off and the filtrate was concentrated *in vacuo* to dryness.

- 5 The residual oil was crystallized from 600 ml. of petroleum ether (b.p. 60—71°C.). The crude product was collected and dissolved in 600 ml. of petroleum ether (b.p. 60—71°C.), heated with decolorizing charcoal and a little Florisil (Trade Mark) and filtered. The filtrate was chilled. The yellow crystals which separated were filtered off and air dried. They weighed 2.5—5 g. and had a melting point of about 41—43°C. The product was identified by NMR spectrum and elemental analysis as N - methallyl - N - n - propyl - 4 - trifluoro - methyl - 2,6 - dinitroaniline.

- 10 The filtrates were concentrated *in vacuo* and the oil chromatographed over a Florisil column. The first 800 ml. of yellow eluate gave 74.3 g. of crude product which, upon recrystallization from hexane, gave yellow crystals having a melting point of 41—43°C. and weighing 58.5 g.

- 15 The structure was confirmed by NMR spectrum. Additional material was obtained from the filtrates. Total yield was 295.5 g. Elemental microanalysis produced the following results:

- 20 Calculated for $C_{14}H_{16}F_3N_3O_4$:

	Theoretical	Found
C	48.42	48.23
H	4.64	4.37
N	12.10	11.98

- 25 The following herbicidal test method has been used to evaluate the compounds of this invention.

The soil in which the test plants were grown was a mixture of equal parts by volume

of sand and a medium-heavy topsoil. The plants were grown in galvanized steel flat pans, 35 cm. long by 25 cm. wide and 8 cm. deep. The bottoms of the pans were grooved and perforated for drainage.

The test compound was formulated by dissolving 120 mg. thereof in 2.5 ml. of a solution containing 10 g. of emulsifiers in a mixture of 1 liter of acetone and 1 liter of ethanol. The 2.5 ml. of solution was then dispersed in 22.5 ml. of water containing 0.045% of emulsifiers. The aqueous dispersion was appropriately diluted to prepare formulations which would supply the proper amounts of test compound when 12.5 ml. of dispersion was applied to each flat.

One gallon of the soil mixture was placed in each flat and levelled. Seeds of the test plants were planted in the soil in rows perpendicular to the long axis of the flat, and were covered with soil. One pint of liquid fertilizer containing 2.5 g. of soluble 18—25—18 fertilizer was added to each flat after planting.

The test compound was applied to the flat the same day the seeds were planted by spraying the compound dispersion evenly over the soil with an atomizing spray gun.

After treatment, the flats were moved to a greenhouse maintained at a temperature and humidity appropriate for the growth of plants. The plants were observed 12 days after treatment, and each species was rated on a 1—5 scale. A rating of 1 indicates that the plants emerged and grew normally, and a rating of 5 indicates that the plants did not emerge, or died soon after emergence. Ratings of 2, 3, or 4 indicate slight, moderate or severe injury, respectively. The results observed from testing compounds of the invention at 1 and at 4 lbs./acre rates were as follows. The compounds are identified by their Example numbers above.

WEEDS

CROPS

Compound Rate* No. lbs./A.	Corn	Cotton	Soybean	Wheat	Alfalfa	Sugarbeet	Rice	Cucumber	Tomato	Barnyard Grass	Lambsquarter	Large Crabgrass	Mustard	Pigweed	Foxtail	Wild Oat	Velvetleaf	Morning glory	Zinnia
1	1	1	1	3	2	2	3	1	2	4	3	5	2	3	4	3	2	1	2
4	4	1	3	3	3	2	4	1	2	4	4	5	2	4	4	4	3	2	1
2	3	-	1	2	1	3	4	1	1	5	4	5	1	4	5	4	1	1	1
4	4	-	3	5	4	4	5	3	3	5	5	5	3	5	5	5	3	2	2
3	4	3	2	2	2	3	4	3	2	4	4	5	2	4	4	4	3	2	2
4	4	1	3	4	3	3	5	4	3	4	4	5	3	4	4	4	3	2	2

* of active ingredient.

The new compounds of this invention are best used in the new herbicidal methods here disclosed. The herbicidal methods of this invention are methods of inhibiting the growth of weeds and preventing the germination of weed seeds in cropland which comprise the application of an herbicidally-effective amount of a compound of this invention to the cropland before the emergence of the weeds. The preferred embodiments of the methods are those using the preferred compounds named above.

As the data above makes clear, the herbicidal methods are safe for use in the treatment of many economically important crops, of which cotton, soybean and sugarbeet are particularly preferred. Other crops in which the compounds may beneficially be used include potatoes, tomatoes, tobacco, onions, melons, and cucumbers.

The compounds control many weeds which infest and injure the above and other crops, as the data above illustrate. Certain of the compounds control other noxious weeds, in-

15

20

25

13

as the data above illustrate. Certain of the compounds named above, control other noxious weeds, in-

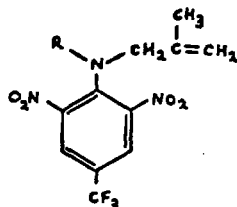
cluding *Solanum nigrum*, *Chenopodium album*, *Polygonum convolvulus*, *Datura stramonium*, *Portulaca oleracea*, *Convolvulus arvensis*, *Fumaria officinalis*, *Echinochloa crus-galli*, *Setaria* sp., *Poa annua*, and *Phalaris minor*.
In general, it is best to carry out the herbicidal methods by applying the compounds before the crop seeds are planted, but after the cropland has been prepared for planting. It is usually most effective to incorporate the compound in the top layer of soil by mixing the soil with an implement such as a rotary hoe, rolling cultivator or disc harrow after the compound has been applied to the cropland. The incorporation device should mix the compound into a layer of soil from about two to six inches deep.
At least an effective herbicidal amount of a compound must be applied to the cropland to carry out the herbicidal methods. In general, effective herbicidal amounts are in the range between 0.25 and 10 lbs./acre. The preferred range of rate of application is from 0.5 to 4 lbs./acre. It will be appreciated by those skilled in the art that at times amounts either below or above the named range will be necessary to obtain the best results from the practice of the herbicidal methods.
The compounds, herbicidal methods, and herbicidal compositions of this invention inhibit the growth of seedling weeds and the emergence of weeds to an extent which depends upon the species of the weed and the identity and the amounts of the compounds applied. In some instances, as the herbicidal test data above show, the whole population of the weed is killed. In other instances, the emerging weeds are only injured, or part of the weeds are killed and part are injured. The injury of a weed seedling growing in cropland is highly beneficial to the crop, because the normally growing crop plants shade out the slow growing weed. The shading effect of the crop is especially significant in a bushy crop such as cotton or any of the bean family and the compounds of the invention are particularly effective in controlling *Solanum nigrum* weeds which cause significant reduction of crop yield in areas of cotton growth.
While the compounds of this invention can be applied to cropland in the pure form, it is much more convenient to apply one of the herbicidal compositions here disclosed. The herbicidal compositions of this invention comprise one or more of the compounds of this invention combined with at least one inert carrier. Inert carriers may be either liquid or solid.
The inert carriers employed in the preparation of the compositions and the methods of manufacturing the compositions are those which are conventional in the agricultural chemicals art. Some explanation of the formulation of our herbicidal compositions will be nevertheless given, in order to ensure that the best methods are clear.
Very often, the compounds are formulated as concentrated compositions to be applied to the soil in the form of aqueous dispersions or emulsions which contain from 0.1% to 1% of the compound. Water-dispersible or emulsifiable compositions are either finely divided solids usually known as wettable powders, or liquids usually known as emulsifiable concentrates.
Herbicidal wettable powders comprise an intimate mixture of the active compound, an inert carrier, and surfactants. The concentration of the active compound is usually from 10 percent to 90 percent. The inert carrier is usually chosen from among the attapulgite clays, the montmorillonite clays, the diatomaceous earths, or the purified silicates. Effective surfactants, comprising from 0.5 percent to 10 percent of the wettable powder, are found among the sulfonated lignins, the condensed naphthalene-sulfonates, the naphthalenesulfonates, the alkylbenzenesulfonates, the alkyl sulfates and nonionic surfactants such as ethylene oxide adducts of phenol.
Typical herbicidal emulsifiable concentrates of the compounds comprise a convenient concentration of the active compound, such as from 1/2 to 6 lbs./gal. of liquid, dissolved in an inert carrier which is a mixture of water-immiscible organic solvent and emulsifiers. The organic solvent is chosen with regard to its solvent power and its cost. Useful organic solvents include the aromatic hydrocarbons, especially the xylenes, and the petroleum fractions, especially the high-boiling naphthalenic and olefinic portions of petroleum such as heavy aromatic naphtha. Other organic solvents may also be used, such as the terpenic solvents including rosin derivatives. Emulsifiers, comprising 0.5 to 10 percent of the emulsifiable concentrates, are chosen from the same types of surfactants used for wettable powders.
Granular compositions are also convenient forms in which to apply the compounds, by simply distributing the composition evenly over the cropland. Herbicidal granules typically comprise a compound dispersed on a granular inert carrier of which coarsely ground clay is typical. The particle size of such granules usually ranges from 0.1 to 3 mm. The usual formulation process comprises dissolving the compound in a solvent such as kerosene or heavy aromatic naphtha, and applying the solution to the carrier in an appropriate mixer. Less economically, the compound may be dispersed in a dough composed of damp clay or other carrier, which dough is then dried and coarsely ground to produce the desired granular product.
The herbicidal compositions may be applied to cropland by the use of conventional equip-

ment. For example, water-dispersed compositions are applied by means of sprayers which may be hand-carried, tractor-mounted, self-propelled or towed. Granular compositions are applied by any of the many applicators which are now in common use. The operator of the application equipment need only take care to apply an amount of the composition per acre which supplies the desired application rate of the compound, and to apply it evenly throughout the cropland area.

The herbicidal compositions of the invention may comprise as sole active ingredient a compound of formula (I). Alternatively, they may also comprise other herbicides such as urea herbicides, for instance: 3 - (3,4 - dichlorophenyl) - 1 - methoxy - 1 - methylurea; 3 - (4 - chlorophenyl) - 1 - methoxy - 1 - methylurea; 3 - (4 - bromophenyl) - 1 - methoxy - 1 - methylurea; 3 - (3,4 - dichlorophenyl) - 1,1 - dimethylurea; N' - 4 - (4 - chlorophenoxy)phenyl - N,N - dimethylurea; and N' - (3 - chloro - 4 - methoxyphenyl) - N,N - dimethylurea; triazine herbicides such as 2 - chloro - 4 - ethylamino - 6 - isopropylamino - 1,3,5 - triazine; 4 - amino - 6 - *t* - butyl - 3 - (methylthio) - 1,2,4 - triazin - 5 - one; 2 - chloro - 4,6 - bis - (ethylamino) - 1,3,5 - triazine; 4 - ethylamino - 2 - methylthio - 6 - *t* - butylamino - 1,3,5 - triazine; phenoxy herbicides such as 4 - (2,4 - dichlorophenoxy) - butyric acid; 4 - (4 - chloro - 2 - methylphenoxy) - butyric acid; and 2 - [(4 - chloro - *o* - tolyl)oxy] - propionic acid; carbamate herbicides such as isopropyl - N - (3 - chlorophenyl)carbamate; amide herbicides such as N - (4 - chlorophenyl) - 2,2 - dimethyl - valeramide and 2 - (α - naphthoxy) - N,N - diethyl - propionamide; or herbicides such as 2 - (1 - methyl - *n* - propyl) - 4,6 - dinitrophenol; 3,5 - dinitro - N,N - dipropylsulphanilamide; 5 - amino - 4 - chloro - 2 - phenyl - 3 - pyridazone and 1 - (5 - *t* - butyl - 1,3,4 - thiadiazol - 2 - yl) - 1,3 - dimethylurea, and thiolcarbamate herbicides such as S - ethyl - N,N - dipropyl thiolcarbamate.

WHAT WE CLAIM IS:—

1. A herbicidal composition comprising a 2,6-dinitroaniline of general formula (I):



(I)

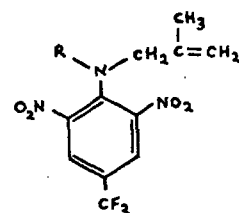
where R represents an ethyl, propyl or allyl group, in association with at least one inert carrier or diluent therefor.

2. A herbicidal composition as claimed in claim 1, wherein the compound of formula (I) is N - ethyl - N - methallyl - 4 - trifluoromethyl - 2,6 - dinitroaniline.

3. A herbicidal composition as claimed in claim 1 or 2, which additionally comprises a further herbicide.

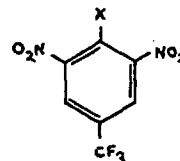
4. A herbicidal composition as claimed in claim 3, wherein said further herbicide is 2 - chloro - 4 - ethylamino - 6 - isopropylamino - 1,3,5 - triazine.

5. A method of preparing a 2,6 - dinitroaniline of general formula (I):

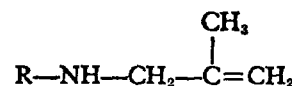


(I)

wherein R represents an ethyl, propyl or allyl group, which comprises reacting a dinitrobenzene of formula:



wherein X is a halogen atom preferably a chlorine atom, with an amine of the formula:



wherein R is as defined above.

6. A method according to claim 5 wherein said amine has the formula:



7. A 2,6-dinitroaniline of formula I whenever prepared by a process according to claim 5 or 6.

8. A method of inhibiting the growth of weeds and/or preventing the germination of weed seeds in cropland, which comprises



* C L M S *

14

6

1,505,249

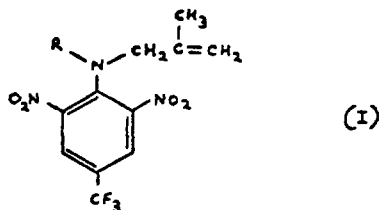
6

ment. For example, water-dispersed compositions are applied by means of sprayers which may be hand-carried, tractor-mounted, self-propelled or towed. Granular compositions are applied by any of the many applicators which are now in common use. The operator of the application equipment need only take care to apply an amount of the composition per acre which supplies the desired application rate of the compound, and to apply it evenly throughout the cropland area.

The herbicidal compositions of the invention may comprise as sole active ingredient a compound of formula (I). Alternatively, they may also comprise other herbicides such as urea herbicides, for instance: 3 - (3,4 - dichlorophenyl) - 1 - methoxy - 1 - methylurea; 3 - (4 - chlorophenyl) - 1 - methoxy - 1 - methylurea; 3 - (4 - bromophenyl) - 1 - methoxy - 1 - methylurea; 3 - (3,4 - dichlorophenyl) - 1,1 - dimethylurea; N' - 4 - (4 - chlorophenoxy)phenyl - N,N - dimethylurea; and N' - (3 - chloro - 4 - methoxyphenyl) - N,N - dimethylurea; triazine herbicides such as 2 - chloro - 4 - ethylamino - 6 - isopropylamino - 1,3,5 - triazine; 4 - amino - 6 - *t* - butyl - 3 - (methylthio) - 1,2,4 - triazin - 5 - one; 2 - chloro - 4,6 - bis - (ethylamino) - 1,3,5 - triazine; 4 - ethylamino - 2 - methylthio - 6 - *t* - butylamino - 1,3,5 - triazine; phenoxy herbicides such as 4 - (2,4 - dichlorophenoxy) - butyric acid; 4 - (4 - chloro - 2 - methylphenoxy) - butyric acid; and 2 - [(4 - chloro - *o* - tolyl)oxy] - propionic acid; carbamate herbicides such as isopropyl - N - (3 - chlorophenyl)carbamate; amide herbicides such as N - (4 - chlorophenyl) - 2,2 - dimethyl - valeramide and 2 - (α - naphthoxy) - N,N - diethyl - propionamide; or herbicides such as 2 - (1 - methyl - *n* - propyl) - 4,6 - dinitrophenol; 3,5 - dinitro - N,N - dipropylsulphanilamide; 5 - amino - 4 - chloro - 2 - phenyl - 3 - pyridazone and 1 - (5 - *t* - butyl - 1,3,4 - thiadiazol - 2 - yl) - 1,3 - dimethylurea, and thiocarbamate herbicides such as S - ethyl - N,N - dipropyl thiocarbamate.

WHAT WE CLAIM IS:—

1. A herbicidal composition comprising a 2,6-dinitroaniline of general formula (I):



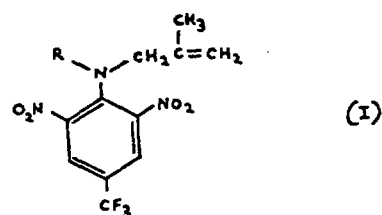
where R represents an ethyl, propyl or allyl group, in association with at least one inert carrier or diluent therefor.

2. A herbicidal composition as claimed in claim 1, wherein the compound of formula (I) is N - ethyl - N - methylallyl - 4 - trifluoromethyl - 2,6 - dinitroaniline.

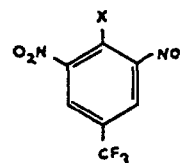
3. A herbicidal composition as claimed in claim 1 or 2, which additionally comprises a further herbicide.

4. A herbicidal composition as claimed in claim 3, wherein said further herbicide is 2 - chloro - 4 - ethylamino - 6 - isopropylamino - 1,3,5 - triazine.

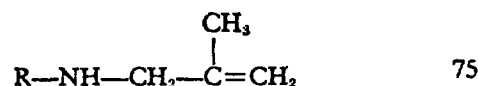
5. A method of preparing a 2,6 - dinitroaniline of general formula (I):



wherein R represents an ethyl, propyl or allyl group, which comprises reacting a dinitrobenzene of formula:



wherein X is a halogen atom preferably a chlorine atom, with an amine of the formula:



wherein R is as defined above.

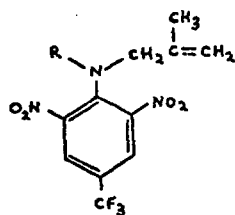
6. A method according to claim 5 wherein said amine has the formula:



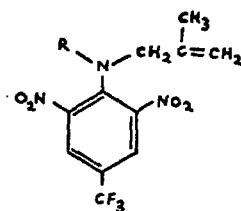
7. A 2,6-dinitroaniline of formula I whenever prepared by a process according to claim 5 or 6.

8. A method of inhibiting the growth of weeds and/or preventing the germination of weed seeds in cropland, which comprises

applying a herbicidally-effective amount of a 2,6-dinitroaniline of general formula (I):



(I)



(I)

wherein R represents an ethyl, propyl or allyl group.

5 wherein R represents an ethyl, propyl or allyl group, to the cropland before emergence of the weeds.

9. A method according to claim 8 wherein the 2,6-dinitroaniline is applied in the form of a composition as claimed in any one of claims 1 to 4.

10. A method of inhibiting the growth of weeds and/or preventing the growth of weed seeds in cropland, which comprises applying a herbicidally-effective amount of N - ethyl - N - methallyl - 4 - trifluoromethyl - 2,6 - dinitroaniline to the cropland before emergence of the weeds.

11. A method according to claim 10 wherein a combination of N - ethyl - N - methallyl - 4 - trifluoromethyl - 2,6 - dinitroaniline and 2 - chloro - 4 - ethylamino - 6 - isopropylamino - 1,3,5 - triazine is applied to the cropland.

12. A method according to any one of claims 8 to 11 wherein the crop is cotton.

13. A 2,6-dinitroaniline of general formula (I):

14. N - Ethyl - N - methallyl - 4 - trifluoromethyl - 2,6 - dinitroaniline.

15. A herbicidal composition comprising a compound of formula (I) substantially as hereinbefore described.

16. A method of preparing a compound of formula (I) substantially as hereinbefore described with particular reference to any one of the Examples.

17. A compound of formula (I) whenever prepared by the method of claim 16.

18. A method of inhibiting the growth of weeds and/or preventing the germination of weed seeds in cropland, according to claim 8, substantially as hereinbefore described.

19. A compound of formula (I) substantially as hereinbefore described with reference to any one of the foregoing Examples.

K. W. H. McVEY,
Chartered Patent Agent,
Erl Wood Manor,
Windlesham, Surrey, England.
Agent for the Applicants.

Printed for Her Majesty's Stationery Office by the Courier Press, Leamington Spa, 1978.
Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

