

PATENT SPECIFICATION

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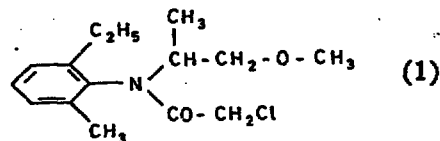
(54) AN N-SUBSTITUTED CHLOROACETANILIDE FOR REGULATING PLANT GROWTH

(71) We, CIBA-GEIGY A.G., a Swiss body Corporate of Basle, Switzerland, 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:—

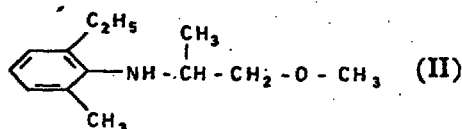
The present invention relates to an N-substituted chloroacetanilide, its manufacture, herbicidal and plant growth regulating compositions which contain this new compound as active ingredient, and the use of the new compound as herbicide and plant growth regulant.

Herbicidally active haloacetanilides are already known from the literature. Reference is made to the following patent specifications as representing the prior art: French Patent Specifications Nos. 1,337,529, 1,419,116 and 2,028,991; Belgian Patent Specification No. 746,288; and U.S. Patent Specifications Nos. 2,863,752, 3,442,945 and 3,547,620.

According to the present invention there is provided the compound, 2-ethyl-6-methyl-N-(1'-methoxyprop-2'-yl)-N-chloroacetanilide, of the formula I

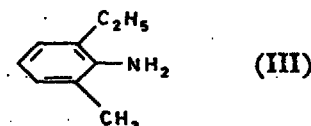


The chloroacetanilide of the formula I may be prepared by reacting the N-substituted aniline of the formula II



with a chloroacetylating agent, preferably an anhydride or halide of chloroacetic acid.

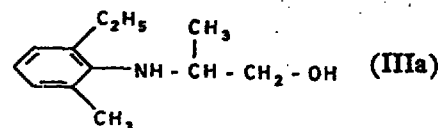
It is also possible to prepare the compound of the formula I by reacting the aniline of the formula III



with 2-halopropanol to introduce the hydroxy-alkyl chain



and then chloroacetylating the resulting compound of the formula IIa



preferably with an anhydride or halide of chloroacetic acid, and finally the still free OH group is etherified in acid medium (e.g. HCl, H₂SO₄) under mild conditions and in conventional manner with methyl alcohol.

The reactions can be carried out in the presence or absence of solvents or diluents which are inert towards the reactants. Examples of suitable solvents or diluents are: aliphatic, aromatic or halogenated hydrocarbons, such as methylene chloride, ethylene chloride, chloroform; ethers and ethereal compounds such as dialkyl ethers, dioxan, tetrahydrofuran; nitriles, such as acetonitrile; N,N-dialkylated amides, such as dimethyl formamide; also dimethyl sulphoxide, and also mixtures of these solvents.

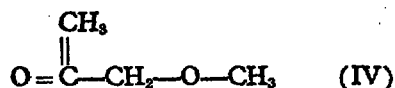
As suitable chloroacetylating agents there

are preferably used chloroacetic anhydride, and chloroacetic halides; such as chloroacetyl chloride. However, it is also possible to carry out the reaction with chloroacetic acid or its esters or amides. The reaction temperatures are generally known between 0° and 200°C, preferably between 20° and 100°C. Often, especially if chloroacetyl halides are used the chloroacetylation is carried out in the presence of an acid binding agent. Suitable acid binding agents are: tertiary amines, such as trialkylamines, e.g. triethylamine, pyridine and pyridine bases, or inorganic bases, such as the oxides and hydroxides, hydrogen carbonates and carbonates of alkali and alkaline earth metals. Furthermore it is also possible to use the aniline of the formula II as acid binding agent, in which case it must be used in excess.

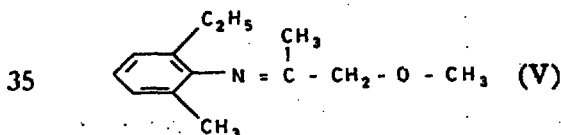
Compounds homologous to formula II and hydroxyalkyl derivatives homologous to formula IIa are known, e.g. from U.S. Patent Specifications Nos. 2,381,071, 2,759,943 as well as from J. Am. Soc. 84, 743 and Bull. Soc. chim. France 1962, 303 and 1965, 2037.

The starting material, which falls under the general formula II, can be manufactured by one of the following known methods, for example:

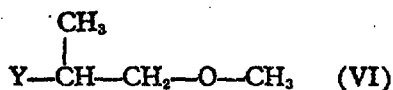
a) by condensation of the alkylated aniline III with the carbonyl compound IV



and simultaneous or subsequent catalytic hydrogenation of the resulting azomethine of the formula V



b) by reaction of the alkylaniline III with a compound of the formula VI



wherein Y represents a halogen atom or another acid radical, in particular an arylsulphonic acid radical. The compound of the formula VI with a benzenesulphonic acid radical Y is described e.g. in Can. J. Chem. 33, 1207, and that with a tosyloxy radical (CH₃-C₆H₄-SO₃) in British Patent Specification No. 869,083.

There are, of course, a number of other processes for the manufacture of the starting material of the formula II from ortho-alkylated anilines.

The compound of formula I is a stable compound and possesses good selective herbicidal properties. Accordingly, the invention provides a method of selectively combating undesirable plant growth at a locus which method comprises applying to the locus the compound of formula I. The locus is suitably an area in which a useful crop is to be grown. The compound of formula I may thus be used against annual grasses such as *Echinochloa* and related plants of the genera *Setaria*, and *Digitaria*, against grasses such as *Lolium* species and against many dicotyledonous weeds such as *Amaranthus*, *Sesbania*, *Chrysanthemum*, *Ipomoea*, *Sinapis*, *Galium*, *Pastinaca*, without causing damage to the cultivated plants in respect of which the use of the active substance is intended, for example soya, alfalfa, peas, beans, lentils, various types of ground nuts, such as peanuts, cotton, maize, coffee, tea, bananas, pineapples, sugar beet, sugar cane, potatoes, paprika, tomatoes, spinach, onions, aubergines, sunflowers, tobacco, *Brassica* species such as rape and cabbage, but also cereals, such as barley, oats, rye, wheat, or dry rice or water rice.

The compound of formula I may be applied either before or after the germination of the cultivated plants and of the weeds and grasses (pre- and postemergence); pre-emergence application is preferred. The rates of application are between 0.1 and 10 kg of active substance per hectare. But in preemergence application the weeds are virtually destroyed with a rate of application as low as 0.25 kg of active substance per hectare. Normally up to 10 kg of active substance per hectare are used to prevent railway embankments, factory grounds, roads etc. from becoming overgrown with weeds.

Furthermore the compound of the formula I also displays plant growth regulating properties. Accordingly the invention also provides a method of retarding the growth in height of grass in grasslands which method comprises applying to the grasslands the compound of formula I. Consequently the growth rate of a grassland (consisting of the genera *Poa pratensis*, *Agrostis tenuis* and *Festuca rubra*) treated in the spring shortly after the first cutting may be reduced by half when the active substance of the formula I is used in amounts of 5 kg of active substance per hectare, an observation period of 60 days being used as basis.

The following Examples illustrate the invention, including the manufacture of starting material.

Example 1.

a) A mixture of 540 g (4.0 moles) of 2-ethyl - 6 - methylaniline and 306 g (2.0 moles) of 2 - bromo - 1 methoxypropane is heated for 40 hours at reduced pressure (10

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mm Hg) to 120°C with stirring. After it has cooled, the light red, viscous solution is diluted with 200 ml of water and made alkaline with 210 ml of conc. sodium hydroxide solution.

- 5 The precipitated product is taken up in ether, and the ethereal solution is washed neutral with water, dried, and evaporated.

10 Distillation of the residue yields pure 2-ethyl - 6 - methyl - N - [1' - methoxyprop - 2' - yl] - aniline which boils at 64°—66°C/0.07 Torr.

- 15 b) A solution of 9.7 g (0.047 mole) of the intermediate described under a) and 5.05 g (0.05 mole) of triethylamine in 30 ml of benzene is treated dropwise with a solution of 5.65 g (0.05 mole) of chloroacetyl chloride in 10 ml of absolute benzene, and the mixture is further stirred for 2 hours at room temperature. The reaction mixture is diluted with ether, the solution repeatedly washed with water, and dried. The solvent mixture is evaporated off in vacuo to give pure 2-ethyl-6-methyl-N-[1'-methoxy-prop-2'-yl]-N-chloroacetanilide, n_D^{20} : 1.5301.

25 Analysis:

Calculated:

C 63.5 H 7.8 N 4.9%

found:

C 63.7 H 8.1 N 5.0%

Example 2.

Combating of *Echinochloa* species and other monocotyledonous weeds in various cultures of useful plants (preemergence method)

One day after the test plants have been sown in seed dishes, dilute aqueous suspensions of the active substances are sprayed in such concentrations on the surface of the soil as to correspond to rates of application of 2 kg, 1 kg and 0.5 kg per hectare. The seed dishes are kept at 22° and 25°C and about 70% relative humidity. The test is evaluated after 28 days according to the following rating:

9 = plants undamaged (as control test)
1 = plants destroyed
8—2 = intermediate stages of damage
— = not tested

Haloacetanilides known from US Patent Specification No. 3,547,620 were used as comparative compounds:

Compound A: 2-methyl-6-ethyl-N-(ethoxymethyl)-chloroacetanilide

Compound B: 2,6-diethyl-N-(methoxymethyl)-chloroacetanilide (commercial product).

The results are shown in the following Table I:

TABLE I

Compound	Rate of application	Echinochloa	Setaria	Digitaria	Cyperus	Alopecurus	Lolium	alfalfa	sugar beet	cotton	soya	wheat	maize
Compound of Formula I	2	1	1	1	1	2	1	8	8	9	9	7	9
	1	1	1	1	1	2	1	9	8	9	9	9	9
	0.5	1	1	1	1	4	1	9	9	9	9	9	9
A	2	1	1	1	1	1	1	6	4	6	8	3	6
	1	1	1	1	1	3	1	7	5	9	8	3	8
	0.5	1	1	1	—	4	1	8	6	9	9	7	9
B	2	1	1	1	—	2	2	8	5	9	8	3	9
	1	1	1	1	2	3	2	9	6	9	9	4	9
	0.5	1	2	1	—	4	4	9	7	9	9	9	9

It is evident that the compound according to the present invention possesses a more distinctly pronounced herbicidal selectivity than the comparative compounds A and B. Primarily, the compound of the formula I is better tolerated by cultures of useful plants.

Example 3.

Activity against undesirable grasses in low rates of application (premergence method)

Plastic tubs 30 cm deep and measuring 30 cm x 50 cm along the edges are filled with medium heavy arable soil. The respective seed is sown on the surface of the soil and then covered with about 2 cm of soil and moistened. The dilute active substance suspension

is applied within 24 hours. Concentrations are used which correspond to a rate of application of 1 kg, 0.5 kg, 0.25 kg and 0.125 kg of active substance per hectare. The tubs are kept at 22—25°C and about 70% relative humidity. As comparative compounds there were used the compound B of Example 2 and compound D = 2,6 - diethyl - N - (butoxyethyl)-chloroacetanilide, which cited as comparative compound in U.S. Patent Specification No. 3,547,620. Evaluation is carried out after 20 days. The result is given in Table 2 as the damage to the test plants expressed in percentages, with the range of more severe damage being more differentiated than that of medium or slight damage. On the other hand the range of slighter damage is more strongly differentiated in useful plants.

TABLE 2

Compound	rate of application	Digitaria sang.	Lolium perenne	Bromus tect.	Sorghum alnum	Echinochloa c.g.	Setaria italica	soya	cotton	maize	sugar beet
Compound of Formula I	1	100	100	100	100	100	100	0	0	2	5
	0.5	100	100	100	100	100	100	0	0	0	0
	0.25	98	100	98	85	100	98	0	0	0	0
	0.125	90	95	60	60	100	98	0	0	0	0
B	1	98	100	95	90	100	100	0	0	5	40
	0.5	85	98	90	85	95	100	0	0	0	25
	0.25	60	98	60	75	90	98	0	0	0	2
	0.125	0-35	75	0-35	0-35	85	95	0	0	0	0
D	1	85	75	0-35	75	98	98	0	0	0	0
	0.5	35	60	0-35	60	98	85	0	0	0	0
	0.25	0-35	0-35	0-35	0-35	75	35	0	0	0	0
	0.125	0-35	0-35	0-35	0-35	35	0-35	0	0	0	0

Comparative compound B is able to combat effectively the monocotyledonous weeds only at a rate of application of 1 kg/AS/ha or more, whereas comparative compound D has a

thoroughly inadequate activity and cannot be considered for this use.

In contrast, the compound of the formula I acts with complete effectiveness against

monocotyledonous weeds in rates of application down to 0.25 kg per hectare and partly to 0.125 kg per hectare. This test shows that the chloroacetanilide of the present invention largely meets the present day requirement for the quantitative reduction of herbicides in cultures of useful plants.

Example 4.

Herbicidal activity against naturally occurring weeds

For field tests in western France (Maine-et-Loire) maize was sown at intervals of 60 cm in ploughed and harrowed fields. The field was then divided up into plots of 4 m² each,

with an expectation of about 60—75 plants per plot. One day after the sowing the plots were treated with the active substance concentration intended for them and obtained by dilution from an emulsion concentrate. Active substances A and B from Example 2 were used as comparative compounds. The evaluation after 35 days referred to the state of the cultivated plants and to the grasses which had emerged naturally. Damage to the plants is expressed in Table 3 in the differentiated percentages of Example 3. It is evident that the compound according to the invention of the formula I is superior in its activity against weeds to the known compounds A and B.

TABLE 3

Compound	Rate of application	Echinochloa crus galli	Digitaria	Maize
Compound of Formula I	4	100	100	0
	2	100	100	0
	1	100	100	0
	0.5	100	100	0
A	4	100	100	2
	2	100	100	0
	1	95	95	0
	0.5	60	60	0
B	4	100	100	0
	2	95	100	0
	1	85	95	0
	0.5	60	<60	0

Example 5.

Growth inhibition in grasses (postemergence method)

Seeds of the grasses *Lolium perenne*, *Poa pratensis*, *Festuca ovina*, and *Dactylis glomerata* were sown in plastic dishes filled with an earth/turf/sand mixture. After 3 weeks the germinated grasses were cut back to a height of 4 cm above the soil and 2 days later sprayed with aqueous spray broths of active substances of the formula I. The amount of active sub-

stance corresponded to a rate of application of 5 kg of active substance per hectare. Fourteen days after application the growth of the grasses was evaluated according to the following linear rating:

1 = strong inhibition (no growth from the time of application)

9 = no inhibition (growth as untreated control)

The following results were obtained:

Compound	Lolium perenne	Poa pratensis	Festuca ovina	Dactylis glomerata
Compound of Formula I	2	3	2	3
Control	9	9	9	9

5 The invention further provides a herbicidal or plant growth regulant composition which comprises, as active ingredient, the compound of formula I together with one or more solid extenders or liquid diluents. The composition may for example be in a solid form containing a solid extender and optionally a surface active agent or in liquid form comprising a liquid diluent and a surface active agent.

10 The compositions according to the invention may be manufactured in known manner e.g. by intimately mixing and/or grinding the active substance of the formula I with the suitable extenders, optionally with the addition of dispersants or solvents which are not towards the active substance. The active substance may take and be used in the following forms:

20 **Solid forms:**

dusts, tracking agents, granules, coated granules, impregnated granules and homogeneous granules.

Liquid forms:

- 25 a) active substance in a form dispersible in water;
wetable powders, pastes, emulsions;
b) solutions.

30 To manufacture solid forms (dusts, tracking agents) the active substance is mixed with solid extenders. Suitable extenders are, for example, kaolin, talcum, bolus, loess, chalk, limestone, ground limestone, attaclay, dolomite, diatomaceous earth, precipitated silica, alkaline earth silicates, sodium and potassium aluminium silicates (feldspar and mica), calcium and magnesium sulphates, magnesium oxide, ground synthetic materials, fertilisers, for example ammonium sulphate, ammonium phosphate, ammonium nitrate, urea, ground vegetable products, such as corn meal, bark dust, sawdust, nutsheil meal, cellulose powder, residues of plant extractions, activated charcoal etc. These substances can either be used singly or in admixture with one another.

45 The particle size of the extenders for dusts is advantageously up to 0.1 mm, for tracking agents from about 0.075 to 0.2 mm, and for granules 0.2 mm or larger.

50 The solid forms contain the active substance in concentrations from 0.1% to 80%.

To these mixtures can also be added additives which stabilise the active substance and/or non-ionic, anionic and cationic surface active substances, which for example improve the adhesion of the active ingredient on plants or parts of plants (adhesives and agglutinants) and/or ensure a better wettability (wetting agents) and dispersibility (dispersing agents). Examples of suitable adhesives are the following:

olein/chalk mixture, cellulose derivatives (methyl cellulose, carboxymethyl cellulose), hydroxyethyl glycol ethers of monoalkyl and dialkyl phenols having 5 to 15 ethylene oxide radicals per molecule and 8 to 9 carbon atoms in the alkyl radical, lignin sulphonic acids, their alkali metal and alkaline earth metal salts, polyethylene glycol ethers (Carbowaxes—'Carbowax' is a registered Trade Mark), fatty alcohol polyethylene glycol ether having 5 to 20 ethylene oxide radicals per molecule and 8 to 18 carbon atoms in the fatty alcohol moiety, condensation products of urea and formaldehyde, and also latex products.

Preferred dispersions (suspensions and emulsions) are manufactured by mixing or grinding the active substance with extenders accompanied by the addition of dispersing agents and solvents, in the process of which there result firstly dispersible active substance concentrations, such as wettable powders and emulsifiable.

The water-dispersible concentrates of the active substance i.e. wettable powders, pastes and emulsifiable concentrates, are agents which can be diluted with water to any concentration desired. They consist of active substance, extender, optionally additives which stabilise the active substance, surface-active substances and anti-foam agents and, optionally, solvents. The active substance concentrations in these agents are from 5—80%.

Wettable powders and pastes are obtained by mixing and grinding the active substance with dispersing agents and pulverulent extenders in suitable apparatus until homogeneity is attained. Extenders are, for example, those mentioned for the solid forms of application. In some cases it is advantageous to use mixtures of different extenders. As dispersing agents there can be used, for example, condensation products of sulphonated naphthalene

- and sulphonated naphthalene derivatives with formaldehyde, condensation products of naphthalene or naphthalene sulphonic acids with phenol and formaldehyde, as well as alkali metal, ammonium and alkaline earth metal salts of lignin sulphonic acid, in addition, alkylaryl sulphonates, alkali and alkaline earth metal salts of dibutyl naphthalene sulphonic acid, fatty alcohol sulphates such as salts of sulphated hexadecanols, heptadecanols, octadecanols and salts of sulphated fatty alcohol glycol ethers, the sodium salt of oleoyl chitronate, the sodium salt of oleoyl methyl tauride, ditertiary acetylene glycols, dialkyl dilauryl ammonium chloride and fatty acid alkali and alkaline earth metal salts.

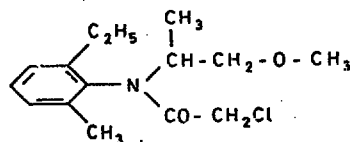
- Suitable anti-foam agents are silicones. The active substance is so mixed, ground, sieved and strained with the additives mentioned above that, in wettable powders, the solid particle size of from 0.02 to 0.04 mm and in pastes, of 0.03 mm is not exceeded. To produce emulsifiable concentrates and pastes, dispersing agents such as those given in the previous paragraphs, organic solvents and water are used. Examples of suitable solvents are the following: alcohols, benzene, xylene, toluene, dimethyl sulphoxide, N,N-dialkylated amides, N-oxides or amines (whereas N-oxides of trialkylamines are preferred), and mineral oil fractions boiling between 120 and 350°C. The solvents must be practically odourless, not phytotoxic inert to the active substances and not readily inflammable.

- Furthermore, the active substance according to the invention can be applied in the form of a solution. For this purpose the active substance of general formula I may be dissolved in suitable organic solvents, mixtures of solvents or in water. Aliphatic and aromatic hydrocarbons, chlorinated derivatives thereof, alkyl naphthalenes and mineral oils singly or in admixture, can be used as organic solvents. The solutions contain the active substance in a concentration range from 1% to 20%.

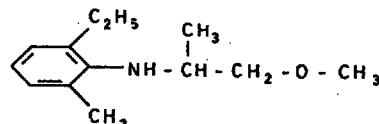
- In addition to the compound of formula I, the compositions can contain other biologically active substances or agents. Thus in order to broaden the activity spectrum the compositions may contain, for example pesticides e.g. insecticides, fungicides, bactericides, fungistatics, bacteriostatics, nematocides or further herbicides, in addition to the cited active substance of the formula I. The composition according to the invention may also contain plant fertilisers and trace elements.

WHAT WE CLAIM IS:—

1. 2 - Ethyl - 6 - methyl - N - (1'-methoxyprop - 2' - yl) - N - chloroacet-



2. A process for the preparation of the compound claimed in claim 1 which process comprises reacting a compound of the formula:



with a chloroacetylating agent.

3. A process according to claim 2 wherein the chloroacetylating agent is a halide or anhydride of chloroacetic acid.

4. A process for the preparation of the compound claimed in claim 1 substantially as hereinbefore described.

5. The compound according to claim 1 prepared by a process claimed in any of claims 2 to 4.

6. A herbicidal or plant growth regulant composition which comprises, as active ingredient, the compound claimed in claim 1 together with one or more solid extenders or liquid diluents.

7. A composition according to claim 6 which is in solid form and comprises the active ingredient together with a solid extender and optionally a surface active agents.

8. A composition according to claim 6 which is in liquid form and comprises the active ingredient together with a liquid diluent and a surface active agent.

9. A composition according to any one of claims 6 to 8 which also comprises one or more pesticidally active substances known *per se*.

10. A method of selectively combating undesirable plant growth at a locus which method comprises applying to the locus the compound claimed in claim 1.

11. A method according to claim 10 wherein the locus in an area in which a useful crop is to be grown.

12. A method according to claim 11, wherein the crop is maize.

13. A method according to claim 11, wherein the crop is soya.

14. A method according to claim 11 wherein the crop is cotton, sunflowers, sugar beet, potatoes, peas or beans.

15. A method of retarding the growth in height of grass in grasslands which method comprises applying to the grasslands the compound claimed in claim 1.

- and sulphonated naphthalene derivatives with formaldehyde, condensation products of naphthalene or naphthalene sulphonic acids with phenol and formaldehyde, as well as alkali metal, ammonium and alkaline earth metal salts of lignin sulphonic acid. in addition, alkylaryl sulphonates, alkali and alkaline earth metal salts of dibutyl naphthalene sulphonic acid, fatty alcohol sulphates such as salts of sulphated hexadecanols, heptadecanols, octadecanols and salts of sulphated fatty alcohol glycol ethers, the sodium salt of oleoyl chitinate, the sodium salt of oleoyl methyl tauride, ditertiary acetylene glycols, dialkyl dilauryl ammonium chloride and fatty acid alkali and alkaline earth metal salts.

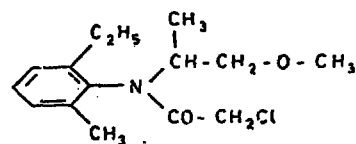
- Suitable anti-foam agents are silicones. The active substance is so mixed, ground, sieved and strained with the additives mentioned above that, in wettable powders, the solid particle size of from 0.02 to 0.04 mm and in pastes, of 0.03 mm is not exceeded. To produce emulsifiable concentrates and pastes, dispersing agents such as those given in the previous paragraphs, organic solvents and water are used. Examples of suitable solvents are the following: alcohols, benzene, xylene, toluene, dimethyl sulphoxide, N,N-dialkylated amides, N-oxides or amines (whereas N-oxides of trialkylamines are preferred), and mineral oil fractions boiling between 120 and 350°C. The solvents must be practically odourless, not phytotoxic inert to the active substances and not readily inflammable.

- Furthermore, the active substance according to the invention can be applied in the form of a solution. For this purpose the active substance of general formula I may be dissolved in suitable organic solvents, mixtures of solvents or in water. Aliphatic and aromatic hydrocarbons, chlorinated derivatives thereof, alkyl naphthalenes and mineral oils singly or in admixture, can be used as organic solvents. The solutions contain the active substance in a concentration range from 1% to 20%.

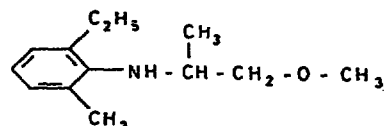
- In addition to the compound of formula I, the compositions can contain other biologically active substances or agents. Thus in order to broaden the activity spectrum the compositions may contain, for example pesticides e.g. insecticides, fungicides, bactericides, fungistatics, bacteriostatics, nematocides or further herbicides, in addition to the cited active substance of the formula I. The composition according to the invention may also contain plant fertilisers and trace elements.

WHAT WE CLAIM IS:—

1. 2 - Ethyl - 6 - methyl - N - (1'-methoxyprop - 2' - yl) - N - chloroacetanilide of the formula



2. A process for the preparation of the compound claimed in claim 1 which process comprises reacting a compound of the formula:



with a chloroacetylating agent.

3. A process according to claim 2 wherein the chloroacetylating agent is a halide or anhydride of chloroacetic acid.

4. A process for the preparation of the compound claimed in claim 1 substantially as hereinbefore described.

5. The compound according to claim 1 prepared by a process claimed in any of claims 2 to 4.

6. A herbicidal or plant growth regulant composition which comprises, as active ingredient, the compound claimed in claim 1 together with one or more solid extenders or liquid diluents.

7. A composition according to claim 6 which is in solid form and comprises the active ingredient together with a solid extender and optionally a surface active agents.

8. A composition according to claim 6 which is in liquid form and comprises the active ingredient together with a liquid diluent and a surface active agent.

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12. A method according to claim 11, wherein the crop is maize.

13. A method according to claim 11, wherein the crop is soya.

14. A method according to claim 11 wherein the crop is cotton, sunflowers, sugar beet, potatoes, peas or beans.

15. A method of retarding the growth in height of grass in grasslands which method comprises applying to the grasslands the compound claimed in claim 1.



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