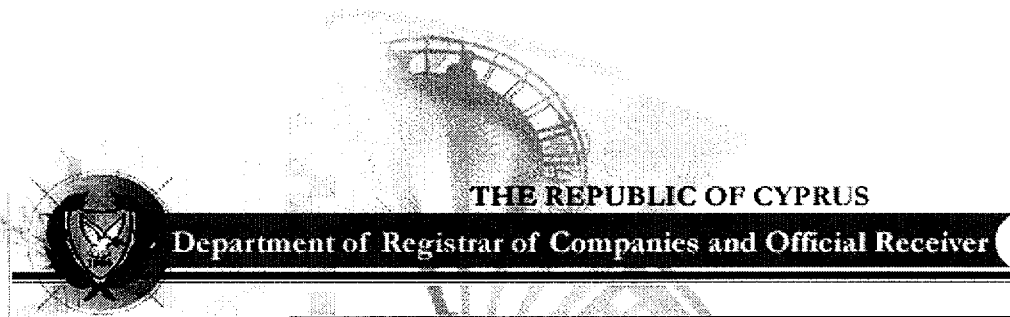




**ΚΥΠΡΙΑΚΟ ΓΡΑΦΕΙΟ ΔΙΠΛΩΜΑΤΩΝ
ΕΥΡΕΣΙΤΕΧΝΙΑΣ
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(71) Applicant

**Monsanto Company,
800 North Lindbergh
Boulevard, St. Louis,
Missouri 63166, United
States of America**

(72) Inventor

John Paul Chupp

(74) Agent

**M. F. Clarke,
Monsanto House, 10—18
Victoria Street, London,
SW1H 0NQ**

(54) **Herbicidal 2-Haloacetanilides**

(57) A group of N-
hydrocarbyloxymethyl-2-
haloacetanilide compounds, herbicidal
compositions containing said
compounds as the active ingredient
and herbicidal method of use in

various crops, particularly, corn and
soybeans and other crops including
cotton, peanuts, rape, bush beans,
wheat and sorghum. The herbicides
herein are particularly effective
against hard-to-kill perennial weeds
such as quackgrass and nutsedges
and are also effective against annual
weeds.

GB 2 072 181 A

Certain of the chemical formulae appearing in the printed specification were submitted after the date of filing, the formulae originally submitted being incapable of being satisfactorily reproduced.

SPECIFICATION

Herbicidal 2-Haloacetanilides

Background of the Invention

Field of the Invention

- 5 This invention pertains to the field of 2-haloacetanilides and their use in the agronomic arts. e.g., as herbicides. 5

Description of the Prior Art

- 10 The prior art relevant to this invention includes numerous disclosures of 2-haloacetanilides which may be unsubstituted or substituted with a wide variety of substituents on the anilide nitrogen atom and/or on the anilide ring including alkyl, alkoxy, alkoxyalkyl, haloalkyl, halogen, etc., radicals. 10

- As more relevant to the invention compounds, which are characterized by having a hydrocarbyloxymethyl radical on the anilide nitrogen, a trifluoromethyl radical in one ortho position and a methyl radical or, in specific cases, an ethyl radical or hydrogen in the other ortho position, the closest prior art known to the inventor appears to be Belgian Patent No. 810,763. The most relevant 15 disclosures in the '763 patent are the generic disclosures of N-alkoxyethyl- or alkyl-substituted alkoxyethyl-2-chloroacetanilides which may be substituted on the anilide ring at the ortho and meta positions with one or more radicals selected from a plurality of radicals including halogen, alkyl, alkoxy or trifluoromethyl ($-\text{CF}_3$). In more particular, the list of compounds in Table II includes species substituted with a $-\text{CF}_3$ radical in one ortho position with no substituent in the other ortho position 20 (Compound Numbers 37—48) and compounds having $-\text{CF}_3$ substitution in a meta position with one ortho position unsubstituted and the other ortho position substituted with a methoxy radical (Compound Numbers 22—25) or a chlorine atom (Compound Numbers 33—36). However, there is no specific disclosure or exemplary species in the '763 patent of 2-haloacetanilides substituted with a $-\text{CF}_3$ radical in one ortho position and a methyl or ethyl radical in the other ortho position as in the 25 compounds of this invention. Moreover, the compounds in the '763 patent are characterized by an alkoxyalkyl group substituted on the anilide nitrogen atom wherein the alkylene moiety must have no less than two carbon atoms between the nitrogen atom and the oxygen atom of the alkoxy moiety. In contrast, the compounds of this invention are further characterized, in part, by the substitution on the anilide nitrogen atom with an alkoxymethyl radical. The significance of the above distinctions between 30 the compounds of the '763 patent and this invention will be made manifest by reference to the comparative herbicidal data herein clearly establishing the superiority of the invention compounds in terms of unit activity, selectivity, weed-control spectrum and crop safety. 30

- Other pertinent prior art less relevant than the above-mentioned '763 patent includes U.S. Patent Numbers 3,966,811 and 4,152,137, German Application 2,402,983, British Application Number 35 2,013,188 and South African Patent Number 74/0767. Although these references contain generic disclosures of 2-haloacetanilides which may include among other substituents $-\text{CF}_3$ substitution on the anilide ring, the only disclosures of any species of such compounds in any of these references are found in said U.S. Patent 3,966,811 and South African Patent No. 74/0767. Even so, neither of the '811 nor '767 patents contain any species of ortho $-\text{CF}_3$ -substituted compounds generally or more 40 particularly, such compounds further substituted in the other ortho position with a methyl or ethyl radical; the only $-\text{CF}_3$ -substituted species disclosed in these patents are meta- $-\text{CF}_3$ -substituted compounds. Moreover, like the above '763 Belgian patent, both of the '811 U.S. patent and '767 South African patent require that there be no less than two carbon atoms between the anilide nitrogen and the oxygen of the alkoxy moiety, a requirement described in all the foregoing prior art references, 45 except said '137 U.S. patent which, as mentioned above, discloses no $-\text{CF}_3$ -substituted species whatever. 45

- Of the above-mentioned most relevant prior art, only said Belgian Patent No. 810,763 and South African Patent No. 74/0767 disclose any herbicidal data relative to N-alkoxyalkyl-2-haloacetanilides having $-\text{CF}_3$ substitution on the anilide ring. Even so, such disclosures are vague, indefinite and 50 incomplete. For example, said '763 patent discloses limited herbicidal data for only one $-\text{CF}_3$ -substituted compound, viz., Compound No. 37 (which is the same compound as in Example 2), i.e., 2-trifluoromethyl-N-(2'-methoxyethyl)-2-chloroacetanilide. In Table 3 of said '763 patent, Compound No. 37 is shown to destroy or seriously injure certain unidentified species of "Cyperus", Setaria, Digitaria and Echinochloa, with little injury to the weeds Avena fatua and an unidentified species of Lolium in 55 certain crops. The lack of specific identity of five of the six weeds tested precludes a meaningful evaluation of the herbicidal efficacy of said Compound 37. 55

- Similarly, said '767 South African patent discloses limited herbicidal data for only one $-\text{CF}_3$ -substituted compound, viz., Compound No. 78, i.e., 2,6-dimethyl-3-trifluoromethyl-N-(2'-methoxyethyl)-N-2-chloroacetanilide. The only herbicidal data disclosed for Compound No. 78 is in 60 Example 5 wherein Compound No. 78 is said to exhibit very strong growth inhibition of four species of grass weeds. However, there is no laboratory or field data disclosed in said '767 patent relative to the effect of Compound No. 78 on any crop or whether that compound exhibits selective control of any weed in any crop, thus rendering any meaningful evaluation of that compound based on such limited 60

data impossible. Still further, the above most relevant references, while disclosing herbicidal activity on a variety of weeds, do not disclose any data for any —CF₃—substituted-N-alkoxy-alkyl compounds which are shown to additionally and/or simultaneously control or suppress in one or more crops the hard-to-kill perennial weeds, quackgrass, yellow and purple nutsedges and a broad spectrum of annual weeds including such hard-to-kill annual grass weeds such as seedling johnsongrass, shattercane, alexandergrass (*Brachiaria plantaginea*), panicums (Texas and wild proso millet), red rice and itchgrass (Raoulgrass), while also controlling or suppressing other noxious perennial and annular weeds, e.g., fall panicum, smartweed, lambsquarters, pigweed, foxtails (e.g., giant and yellow), crabgrass, barnyardgrass, morningglory, velvetleaf, cocklebur, purslane, hemp sesbania, prickly sida, etc.

A highly useful and desirable property of herbicides is the ability to maintain weed control over an extended period of time, the longer the better, during each crop season. With many prior art herbicides, weed control is adequate only for 2 or 3 weeks, or, in some superior cases, perhaps up to 4—6 weeks, before the chemical loses its effective phytotoxic properties. Accordingly, one disadvantage of most prior art herbicides is their relatively short soil longevity.

Another disadvantage of some prior art herbicides, somewhat related to soil longevity under normal weather conditions, is the lack of weed control persistence under heavy rainfall which inactivates many herbicides.

A further disadvantage of many prior art herbicides is limitation of their use in specified types of soil, i.e., while some herbicides are effective in soils having small amounts of organic matter, they are ineffective in other soils high in organic matter or vice-versa. It is, therefore, advantageous that a herbicide be useful in all types of soil ranging from light organic to heavy clay and muck.

Yet another disadvantage of many prior art herbicides is the limitation to a particular effective mode of application, i.e., as a pre-emergence surface application or as a soil incorporation, pre- and/or post-plant mode of application. It is highly desirable to be able to apply a herbicide in any mode of application, whether by surface application or soil incorporation.

And, finally, a disadvantage in some herbicides is the necessity to adopt and maintain special handling procedures due to the toxic nature thereof. Hence, a further desideratum is that a herbicide be safe to handle.

It is, therefore, an object of this invention to provide a group of herbicidal compounds which overcome the above-mentioned disadvantages of the prior art and provide a multiplicity of advantages heretofore unachieved in a single group of herbicides.

It is an object of this invention to provide herbicides which control and/or suppress hard-to-kill perennial and annual weeds such as described above, while maintaining crop safety in a plurality of crops, particularly corn and soybeans, and others including cotton, peanuts, rape, bush beans, wheat and/or sorghum.

It is a further object of this invention to provide herbicidal effectiveness in the soil for periods ranging up to 12 weeks.

Yet another object of this invention is to provide herbicides which resist leaching and dilution due to high moisture conditions, e.g., as heavy rainfall.

Still another object of this invention is the provision of herbicides which are effective over a wide range of soils, e.g., ranging from light-medium organic to heavy clay and muck.

Another advantage of the herbicides of this invention is the flexibility available in the mode of application, i.e., by pre-emergence surface application and by soil incorporation.

Finally, it is an advantage of the herbicides of this invention that they are safe and require no special handling procedures.

The above and other objects of the invention will become apparent from the detailed description below.

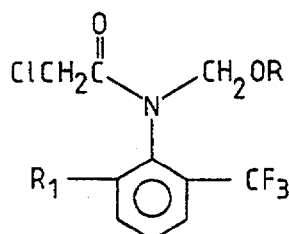
Summary of the Invention

The present invention relates to herbicidally active compounds, herbicidal compositions containing these compounds as active ingredients and herbicidal method of use of said compositions in particular crops.

It has now been found that a selective group of 2-haloacetanilides characterized by specific combinations of specific hydrocarbyloxymethyl radicals on the anilide nitrogen atom, a trifluoromethyl (—CF₃) radical in one ortho position and a methyl or ethyl radical or hydrogen atom in the other ortho position possess unexpectedly superior and outstanding herbicidal properties vis-a-vis prior art herbicides, including homologous compounds of the most relevant prior art.

A primary feature of the herbicidal compositions of this invention is their ability to control a wide spectrum of weeds, including weeds controllable by current herbicides and, additionally, a plurality of weeds which, individually and/or collectively, have heretofore escaped control by a single class of known herbicides, while maintaining crop safety with respect to one or more of a plurality of crops including, particularly, corn and soybeans, but others such as cotton, peanuts, rape, sorghum, wheat and snap beans. While prior art herbicides are useful for controlling a variety of weeds, including on occasion certain resistant weeds, the unique herbicides of this invention have been found to be capable of controlling or greatly suppressing a plurality of resistant perennial and annual weeds, such as the

- perennials quackgrass, yellow and purple nutsedges, annual broadleaves such as prickly sida, hemp sesbania, smartweed, lambsquarters, pigweed and annual grasses such as seeding johnsongrass, shattercane, alexandergrass, Texas panicum, wild proso millet, red rice, itchgrass and other noxious weeds such as fall panicum, foxtails, barnyardgrass, crabgrass, etc. Weed stand reduction has also been achieved in resistant weeds such as ragweed, velvetleaf, morningglory, cocklebur, purslane, etc.
- The compounds of this invention are characterized by the formula



wherein

- R is C₁₋₅ alkyl or alkoxyalkyl or alkenyl or alkynyl having up to 5 carbon atoms; and
 R₁ is hydrogen, methyl or ethyl; provided that:
 When R₁ is hydrogen, R is isopropyl; and
 When R₁ is ethyl, R is ethyl, n-propyl or isopropyl.
 Preferred species of this invention are compounds wherein R₁ in the above formula is methyl or ethyl and R is a C₂₋₄ alkyl radical. Individual preferred species of this invention are as follows:
 N-(ethoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide.
 N-(n-propoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
 N-(isopropoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
 N-(isobutoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
 N-(ethoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide.
 N-(n-propoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide.
 N-(isopropoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide.
 Other species of this invention will be described below.
 The utility of the compounds of this invention as the active ingredient in herbicidal compositions formulated therewith and the method of use thereof will be described below.

25 Detailed Description of the Invention

- The compounds of this invention may be made by the N-alkylation of the anion of the appropriate secondary 2-haloacetanilide with an alkylating agent under basic conditions as exemplified in Example 1 herein. Modification of said N-alkylation process involves the in-situ preparation of halomethyl alkyl ethers used as starting materials in said N-alkylation process and is described in Example 2 for preparing another species of the invention.

Example 1

This example describes the preparation of N-(ethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.

- 2'-Trifluoromethyl-6'-methyl-2-chloroacetanilide, 4.02 gm (0.016 mol), chloromethyl ethyl ether, 3.02 gm (0.032 mol) and benzyl triethylammonium bromide, 2.0 g, (phase transfer catalyst) were mixed in 75 ml of methylene chloride in a 500 ml round bottom flask equipped with mechanical stirrer and thermometer. Sodium hydroxide (50%), 15 ml, were added all at once with vigorous stirring giving rise to an exotherm to 26°C. After about 5 minutes gas chromatography indicated that the reaction was complete. After 15 minutes, ice and water were added, the layers separated and the organic layer washed with 2.5% sodium chloride, dried, filtered and stripped. The dark-colored residue was distilled on a Kugelrohr and 3.4 g of a yellow oil fraction boiling at 110—115°C at 0.1 mm collected. This fraction was taken up in cyclohexane and purified by HPLC using 20% ethyl acetate in cyclohexane. Further distillation of peak fraction by Kugelrohr yielded 3.2 gm (65% yield) of colorless oil, b.p. 100—110°C at 0.1 mm Hg; on standing a white solid, m.p. 41°C—43°C crystallized out.

- Anal.* for C₁₃H₁₅ClF₃NO₂ (%)
- | Element | Theory | Found |
|---------|--------|-------|
| C | 50.41 | 50.02 |
| H | 4.88 | 4.81 |
| N | 4.52 | 4.38 |

50 Example 2

This example illustrates the use of an improved alternative process by which the compounds of

this invention may be prepared. A feature of the process of this invention is that in-situ formation of the alkylating agent, thus effecting a more efficient, economical and simple operation.

5 A slurry of ethylene glycol monomethyl ether 7.3 gm (0.096 mol) and paraformaldehyde, 1.44 gm (0.048 mol) in 100 ml of methylene chloride solvent was cooled in an ice water bath and 5.9 gm (0.048 mol) of acetyl bromide added. After stirring for about 45 minutes, 4.03 gm (0.016 mol) of 2'-trifluoromethyl-6'-methyl-2-chloroacetanilide and 2.0 gm of benzyl triethylammonium chloride were added. Fifty milliliters of 50% NaOH were added all at once. Gas chromatography indicated complete reaction after about 5 minutes. Ice and water were added to the mixture to effect phase separation and the organic phase was then separated, dried, filtered and stripped. The residue was vacuum distilled on a short path still to yield 4.2 gm (77% yield) of clear, colorless oil, b.p. 150—160°C at 0.05 mm Hg. 10

Anal. for $C_{14}H_{17}ClF_3NO_3$ (%)

	<i>Element</i>	<i>Theory</i>	<i>Found</i>	
15	C	49.49	49.33	
	H	5.04	5.04	15
	N	4.12	4.08	

The product was identified as N-(2-methoxyethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.

20 If the N-alkylation process is allowed to develop too high or too low temperatures, various impurities may be generated, e.g., sec-anilide, corresponding imidate, α -alkoxyamide or diketopiperazines. Such impurities may be removed by washing the organic layer with a dilute aqueous salt of acid solution, e.g., 2—3% NaCl or 5% HCl. 20

Examples 3—13

25 Following substantially the same procedures, quantities of reactants and general reaction conditions as described in Examples 1 or 2, but substituting the appropriate starting sec-anilide and alkylating agent to obtain the end product, other compounds according to the above formula are prepared; these compounds are identified in Table I. 25

Table I

Example No.	Compound	Empirical Formula	B.P. °C (mm Hg)	Element	Analysis	
					Calculated	Found
3	N-(isopropoxymethyl)-2'-trifluoro-methyl-2-chloroacetanilide	C ₁₃ H ₁₅ F ₃ CINO ₂	100—101 (0.05)	C	50.41	50.52
				H	4.88	4.89
				N	4.52	4.46
4	N-(n-propoxymethyl)-2'-trifluoro-methyl-6'-methyl-2-chloroacetanilide	C ₁₄ H ₁₇ ClF ₃ NO ₂	110—120°C (0.1)	C	51.94	51.80
				H	5.29	5.17
				N	4.33	4.28
5	N-(isopropoxymethyl)-2'-trifluoro-methyl-6'-methyl-2-chloroacetanilide	C ₁₄ H ₁₇ ClF ₃ NO ₂	110—120°C (0.1)	C	51.94	51.69
				H	5.29	5.23
				N	4.33	4.22
6	N-(isobutoxymethyl)-2'-trifluoro-methyl-6'-methyl-2-chloroacetanilide	C ₁₅ H ₁₉ ClF ₃ NO ₂	130—140°C (0.1)	C	53.34	53.07
				H	5.67	5.61
				N	4.15	4.01
7	N-(methoxymethyl)-2'-trifluoro-methyl-6'-methyl-2-chloroacetanilide	C ₁₂ H ₁₃ ClF ₃ NO ₂	Oil	C	48.47	49.94
				H	4.43	4.44
				N	4.74	4.70
8	N-(n-butoxymethyl)-2'-trifluoro-methyl-6'-methyl-2-chloroacetanilide	C ₁₅ H ₁₉ ClF ₃ NO ₂	135—138°C (0.05)	C	53.34	53.32
				H	5.67	5.68
				N	4.15	4.14
9	N-(sec-butoxymethyl)-2'-trifluoro-methyl-6'-methyl-2-chloroacetanilide	C ₁₅ H ₁₉ ClF ₃ NO ₂	135—142°C (0.05)	C	53.34	53.23
				H	5.67	5.67
				N	4.15	4.13
10	N-(allyloxymethyl)-2'-trifluoro-methyl-6'-methyl-2-chloroacetanilide	C ₁₄ H ₁₅ ClF ₃ NO ₂	123—125°C (0.1)	C	52.27	52.11
				H	4.70	4.74
				N	4.35	4.34
11	N-(propargyloxymethyl)-2'-tri-fluoromethyl-6'-methyl-2-chloroacetanilide	C ₁₄ H ₁₃ ClF ₃ NO ₂	Oil	C	52.59	52.44
				H	4.10	4.14
				N	4.38	4.34
12	N-(isoamyloxymethyl)-2'-tri-fluoromethyl-6'-methyl-2-chloroacetanilide	C ₁₆ H ₂₁ ClF ₃ NO ₂	150°C (0.05)	C	54.63	54.66
				H	6.02	6.03
				N	3.98	4.00
13	N-(ethoxymethyl)-2'-trifluoro-methyl-6'-ethyl-2-chloroacetanilide	C ₁₄ H ₁₇ ClF ₃ NO ₂	133—135°C (0.02)	C	51.94	51.32
				H	5.29	5.26
				N	4.33	4.39
14	N-(n-propoxymethyl)-2'-tri-fluoromethyl-6'-ethyl-2-chloroacetanilide	C ₁₅ H ₁₉ ClF ₃ NO ₂	Colorless Oil	C	53.34	53.74
				H	5.67	5.77
				N	4.15	4.16
15	N-(isopropoxymethyl)-2'-tri-fluoromethyl-6'-ethyl-2-chloroacetanilide	C ₁₅ H ₁₉ ClF ₃ NO	100—105°C (0.01)	C	53.34	53.43
				H	5.67	5.74
				N	4.15	4.18

The secondary anilide starting materials used in the above examples to prepare the compounds of this invention are suitably prepared by conventional chloroacetylation of the appropriate primary amine as exemplified in Example 14 below.

Example 14

This example illustrates the preparation of the secondary anilide starting material used to prepare a species of the compound of this example, i.e., the compound of Example 13.

2-Trifluoromethyl-6-ethylaniline, 6.0 gm (0.03174 mol) was dissolved in 75 ml of toluene and 3.77 gm (0.033 mol) of chloroacetyl chloride added cautiously. The resulting slurry was raised to reflux temperature and held for 4 hours. Thereafter, the mixture was diluted with an equal volume of hexane and the mixture let stand. The product crystallized and the resulting solid was filtered and air dried to give 5.8 gm (69% yield); the filtrate was stripped to produce another 2.7 gm of white solid, m.p. 121—124°C (sealed tube).

Anal. for $C_{11}H_{11}ClF_3NO$ (%)

Element	Theory	Found
C	49.73	49.36
H	4.17	4.09
N	5.27	5.38

The product was identified as 2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide.

Primary amines of the type used to prepare secondary anilides by haloacetylation as described above are known in the literature; see, e.g., the above-mentioned U.S. Patent 3,966,811 and British Application No. 2,013,188.

As noted above, the compounds of this invention have been found to be effective as herbicides, particularly as pre-emergence herbicides, although post-emergence activity has also been shown. The pre-emergence tests referred to herein include both greenhouse and field tests. In the greenhouse tests, the herbicide is applied either as a surface application after planting the seeds or vegetative propagules or by incorporation into a quantity of soil to be applied as a cover layer over the test seeds in pre-seeded test containers. In the field tests, the herbicide may be applied pre-plant incorporation ("P.P.I.") into the soil, i.e., the herbicide is applied to the surface of the soil, then incorporated therein by mixing means followed by planting of the crop seeds, or the herbicide may be applied to the surface ("S.A", surface application) after the crop seed is planted.

The surface application ("S.A") test method used in the greenhouse is performed as follows: Containers, e.g., aluminum pans typically 9.5"×5.25"×2.75" (24.13 cm×13.34 cm×6.99 cm) or plastic pots 3.75"×3.75"×3" (9.53 cm×9.53 cm×7.62 cm) having drain holes in the bottom, are level-filled with Ray silt loam soil then compacted to a level 0.5 inch (1.27 cm) from the top of the pots. The pots are then seeded with plant species to be tested, covered with a 0.5 inch layer of the test soils. The herbicide is then applied to the surface of the soil, e.g., with a belt sprayer at 20 gal/A, 30 psi (187 l/ha, 2.11 kg/cm²). Each pot receives 0.25 inch (0.64 cm) water as overhead irrigation and the pots are then placed on greenhouse benches for subsequent sub-irrigation as needed. As an alternative procedure, the overhead irrigation may be omitted. Observations of herbicidal effects are made about three weeks after treatment.

The herbicide treatment by soil incorporation ("S.I.") used in the greenhouse tests are as follows:

A good grade of top soil is placed in aluminum pans and compacted to a depth of three-eighths to one-half inch from the top of the pan. On the top of the soil is placed a number of seeds or vegetative propagules of various plant species. The soil required to level fill the pans after seeding or adding vegetative propagules is weighed into a pan. The soil and a known amount of the active ingredient applied in a solvent or as a wettable powder suspension are thoroughly mixed, and used to cover the prepared pans. After treatment, the pans are given an initial overhead irrigation of water, equivalent to one-fourth inch (0.64 cm) rainfall, then watered by subirrigation as need to give adequate moisture for germination and growth. As an alternative procedure, the overhead irrigation may be omitted.

Observations are made about 2—3 weeks after seeding and treatment.

Tables II and III summarize results of tests conducted to determine the pre-emergence herbicidal activity of the compounds of this invention; in these tests, the herbicides were applied by soil incorporation and sub-irrigation watering only. The herbicidal rating was obtained by means of a fixed scale based on the percent injury of each plant species. The ratings are defined as follows:

% Control	Rating
0—24	0
25—49	1
50—74	2
75—100	3
Undetermined	5

The plant species utilized in one set of tests, the data for which are shown in Table II, are identified by letter in accordance with the following legend:

5	A Canada Thistle	E Lambsquarters	I Johnson grass	5
	B Cocklebur	F Smartweed	J Downy Brome	
	C Velvetleaf	G Yellow Nutsedge	K Barnyard grass	
	D Morningglory	H Quackgrass		

Table II
Pre-emergent

10	Compound of Example No.	kg/ha	Plant Species											10
			A	B	C	D	E	F	G	H	I	J	K	
	1	11.2	5	3	3	3	3	3	3	3	3	3	3	
		5.6	5	2	2	3	3	3	3	3	3	3	3	
15	2	11.2	2	1	1	3	2	2	3	3	3	3	3	15
		5.6	3	2	1	3	2	3	0	2	2	3	3	
	3	11.2	3	1	2	3	3	3	3	3	2	3	3	
		5.6	2	0	2	3	3	2	3	3	1	3	3	
	4	11.2	5	3	2	3	3	3	3	3	3	3	3	
		5.6	5	2	1	2	3	3	3	3	3	3	3	
20	5	11.2	5	3	2	3	3	3	3	3	3	3	3	20
		5.6	5	0	0	3	3	3	3	3	1	3	3	
	6	11.2	5	2	1	3	3	3	3	3	3	3	3	
		5.6	5	2	0	1	3	3	3	3	3	3	3	
25	7	11.2	3	1	2	2	3	0	2	3	1	3	3	25
		5.6	1	0	1	1	3	0	1	3	1	3	3	
	8	11.2	3	2	0	2	3	3	3	3	3	3	3	
		5.6	3	1	0	3	3	3	3	3	2	3	3	
	9	11.2	3	2	2	3	3	3	3	3	3	3	3	
		5.6	3	2	1	2	3	3	3	3	1	3	3	
30	10	11.2	3	2	1	3	3	3	3	3	3	3	3	30
		5.6	3	1	1	2	3	3	3	3	0	3	3	
	11	11.2	3	1	1	3	3	3	3	3	0	3	3	
		5.6	3	1	1	2	3	3	1	3	1	3	3	
35	12	11.2	3	2	0	2	3	3	3	3	0	3	3	35
		5.6	3	1	0	2	1	2	3	3	3	3	3	
	13	11.2	3	2	2	3	3	3	3	3	3	3	3	
		5.6	3	2	2	3	3	3	3	3	2	3	3	

The compounds were further tested by utilizing the above procedure on the following plant species:

40	L Soybean	R Hemp Sesbania	40
	M Sugarbeet	E Lambsquarters	
	N Wheat	F Smartweed	
	O Rice	C Velvetleaf	
45	P Sorghum	J Downy brome	45
	B Cocklebur	S Panicum Spp.	
	Q Wild Buckwheat	K Barnyardgrass	
	D Morningglory	T Crabgrass	

The results are summarized in Table III.

Table III
Pre-emergent

Compound of Example																		
5	No.	kg/ha	L	M	N	O	P	B	Q	D	R	E	F	C	J	S	K	T
10	1	5.6	1	2	3	3	3	0	3	3	3	3	3	2	3	3	3	3
		1.12	0	1	1	2	3	0	1	2	2	3	2	0	3	3	3	3
		0.28	0	1	0	2	1	0	1	0	3	3	3	0	2	3	3	3
		0.056	0	0	0	0	0	0	0	0	1	1	1	0	0	2	2	3
		0.0112	0	0	0	0	0	0	0	0	0	1	1	0	0	1	2	2
15	2	5.6	1	3	2	3	3	1	2	3	3	3	3	0	3	3	3	5
		1.12	0	3	2	3	2	0	1	3	1	2	2	0	3	3	3	5
		0.28	0	2	0	1	1	0	0	1	1	1	0	0	3	3	3	5
		0.056	0	1	0	0	0	0	0	0	0	0	0	0	1	1	3	5
		0.0112	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	5
20	3	5.6	0	3	3	3	3	1	3	3	3	3	3	1	2	3	3	3
		1.12	0	1	1	3	3	1	0	3	1	3	0	0	3	3	3	3
		0.28	0	0	0	2	2	0	0	0	0	3	0	0	1	3	3	3
		0.056	0	0	0	1	0	0	0	0	0	2	3	0	0	3	3	3
		0.0112	0	0	0	0	0	0	0	0	0	1	0	0	0	3	3	3
25	4	5.6	2	3	3	3	3	2	3	3	3	3	3	1	3	3	3	3
		1.12	1	3	2	3	3	1	3	2	3	3	3	1	3	3	3	3
		0.28	0	1	2	2	1	0	1	0	1	3	3	0	2	3	3	3
		0.056	0	1	1	1	2	2	1	0	2	1	1	0	0	2	2	3
		0.0112	0	1	0	0	0	0	0	0	0	1	0	0	0	3	3	3
30	5	5.6	1	2	3	3	3	1	3	3	3	3	3	1	3	3	3	3
		1.12	0	2	2	3	3	0	2	0	2	3	3	0	3	3	3	3
		0.28	0	2	1	3	3	2	2	3	2	3	3	0	3	3	3	3
		0.056	0	1	0	1	2	1	2	0	1	3	1	0	2	3	3	3
		0.0112	0	1	0	0	0	1	1	0	0	3	1	0	1	2	3	3
35	6	5.6	1	3	3	3	3	2	3	3	3	3	3	1	3	3	3	3
		1.12	0	1	0	3	3	1	3	3	1	3	3	0	3	3	3	3
		0.28	0	2	0	2	2	2	2	1	3	3	3	0	3	3	3	3
		0.056	0	2	0	1	2	1	2	2	2	3	1	0	2	3	3	3
		0.0112	0	2	0	1	0	2	2	2	2	3	1	0	1	1	3	3
40	7	5.6	0	3	2	2	3	0	2	2	2	3	0	0	3	3	3	3
		1.12	0	1	0	0	1	0	1	0	0	2	0	0	0	2	3	3
		0.28	0	0	0	0	0	0	1	2	0	0	0	0	0	2	2	3
		0.056	0	1	0	1	0	0	0	0	0	3	2	0	0	0	2	3
45	8	5.6	2	3	3	3	3	2	3	3	3	3	3	1	3	3	3	3
		1.12	1	2	3	3	3	0	2	0	1	3	3	0	3	3	3	3
		0.28	0	1	2	1	2	0	2	0	2	1	1	0	2	3	3	3
		0.056	0	0	1	1	1	0	0	0	0	1	1	0	1	3	3	3
		0.0112	1	0	0	0	0	1	0	0	1	0	0	0	1	0	2	3
50	9	5.6	2	2	3	3	3	2	3	3	3	2	3	1	3	3	3	3
		1.12	1	2	3	3	1	1	3	1	2	2	2	0	3	3	3	3
		0.28	1	2	2	3	2	0	1	1	2	1	1	0	3	3	3	3
		0.056	0	0	0	0	0	0	0	0	0	2	1	0	2	3	3	3
		0.0112	0	0	0	0	0	0	0	0	0	0	0	0	0	0	3	3
55	10	5.6	3	3	3	3	3	2	3	3	3	3	3	0	3	3	3	3
		1.12	1	2	3	3	3	0	3	3	2	2	3	0	3	3	3	3
		0.28	0	2	2	2	2	0	1	0	2	3	2	0	3	3	3	3
		0.056	0	1	0	1	0	0	0	0	1	0	0	0	0	3	3	3
		0.0112	0	0	0	0	0	0	0	0	0	0	0	0	0	1	2	3

Table III (Cont'd)
Pre-emergent

Compound
of Example

No.	kg/ha	L	M	N	O	P	B	Q	D	R	E	F	C	J	S	K	T
11	5.6	0	3	3	3	3	1	3	3	3	3	3	1	3	3	3	5
	1.12	0	3	1	3	1	1	1	2	1	2	2	0	3	3	3	5
	0.28	0	2	1	2	1	0	0	0	1	1	1	0	3	3	3	5
	0.056	0	1	0	0	0	0	0	0	5	0	0	0	1	2	3	5
	0.0112	0	1	0	0	0	0	0	0	0	0	0	0	0	1	3	5
12	5.6	1	2	2	3	3	1	2	2	0	2	1	0	3	3	3	3
	1.12	1	2	1	2	3	0	2	2	1	2	2	0	3	3	3	3
	0.28	0	1	0	1	0	0	1	0	0	1	0	0	1	2	3	2
	0.056	0	5	0	0	0	0	5	0	0	0	0	0	0	0	2	0
13	5.6	2	3	3	3	3	2	3	3	3	3	3	2	3	3	3	3
	1.12	1	2	3	3	3	0	2	2	3	2	3	0	3	3	3	3
	0.28	2	2	3	3	1	0	1	0	3	1	3	0	3	3	3	3
	0.056	0	1	1	1	0	0	1	0	0	0	0	0	2	3	3	3
	0.0112	0	0	0	0	0	0	0	0	1	5	5	0	0	0	2	2

- 20 The herbicides of this invention have been found to possess unexpectedly superior properties as pre-emergence herbicides, most particularly in the selective control of the hard-to-kill perennial and annual weeds, including such perennials as quackgrass, yellow and purple nutsedges; annual broadleaf weeds such as prickly sida, hemp sesbania, smartweed, lambsquarters, pigweed and annual grasses such as shattercane, alexandergrass, johnsongrass, Texas panicum, wild proso millet, red rice, itchgrass
- 25 (Raoulgrass; *Rottboellia exaltata*) foxtails, e.g., green, yellow and giant) barnyardgrass, and large crabgrass. Weed stand reduction has also been achieved on other resistant species such as ragweed, velvetleaf, morningglory, cocklebur, purslane, etc.
- 30 Selective control and increased suppression of the above-mentioned weeds with the invention herbicides has been found in a variety of crops including, of particular interest, corn and soybeans, but others such as cotton, peanuts, rape, snapbeans (bush beans), wheat and sorghum; however, the latter two crops are usually less tolerant to the invention herbicides than are the other named crops; such reduced tolerance may be improved by the use of safeners, i.e., herbicide antidotes.
- 35 In the discussion and tables of data below, reference is made to herbicide application rates symbolized as "GR₁₅" and "GR₈₅"; these rates are given in kilograms per hectare (kg/ha) which are convertible into pounds per acre (lbs/A) by dividing the kg/ha rate by 1.12. GR₁₅ defines the maximum rate of herbicide required to produce 15% or less crop injury, and GR₈₅ defines the minimum rate required to achieve 85% inhibition of weeds. The GR₁₅ and GR₈₅ rates are used as a measure of potential commercial performance, it being understood, of course, that suitable commercial herbicides may exhibit greater or lesser plant injuries within reasonable limits.
- 40 A further guide to the effectiveness of a chemical as a selective herbicide is the "selectivity factor" ("SF") for a herbicide in given crops and weeds. The selectivity factor is a measure of the relative degree of crop safety and weed injury and is expressed in terms of the GR₁₅/GR₈₅ ratio, i.e., the GR₁₅ rate for the crop divided by the GR₈₅ rate for the weed, both rates in kg/ha (lb/A). In the tables below, selectivity factors are shown in parenthesis following the GR₈₅ rate for each weed; the symbol
- 45 "NS" indicates "non-selective". Marginal or undetermined selectivity is indicated by a dash (—).
- 50 Since crop tolerance and weed control are inter-related, a brief discussion of this relationship in terms of selectivity factors is meaningful. In general, it is desirable that crop safety factors, i.e., herbicide tolerance values, be high, since higher concentrations of herbicide are frequently desired for one reason or another. Conversely, it is desirable that weed control rates be small, i.e., the herbicide possesses high unit activity, for economical and possibly ecological reasons. However, small rates of application of a herbicide may not be adequate to control certain weeds and a larger rate may be required. Hence the best herbicides are those which control the greatest number of weeds with the least amount of herbicide and provide the greatest degree of crop safety, i.e., crop tolerance.
- 55 Accordingly, use is made of selectivity factors (defined above) to quantify the relationship between crop safety and weed control. With reference to the selectivity factors listed in the tables, the higher the numerical value, the greater selectivity of the herbicide for weed control in a given crop.
- 60 In order to illustrate the unexpectedly superior properties of the compounds of this invention both on an absolute basis and on a relative basis, comparative tests were conducted in the greenhouse with compounds of the prior art most closely related in chemical structure to the invention compounds. Said prior art compounds are identified as follows (using the same nomenclature as for the invention compounds):

A. N-(methoxyethyl)-2'-trifluoromethyl-2-chloroacetanilide.

B. N-(ethoxyethyl)-2'-trifluoromethyl-2-chloroacetanilide.

In Tables IV and V, pre-emergence herbicidal activity data are presented comparing the relative efficacy of the invention compounds and said prior art compounds as selective herbicides against the resistant and troublesome perennial weeds quackgrass and yellow nutsedge in soybeans and corn, respectively. These weeds are commonly associated with such major crops as corn and soybeans. The test data in Table IV and V were obtained under identical conditions and represent the average of two replicate runs (except for the compound of Example 13 which was present in one comparative test). The test procedure was the same as described for Tables II and III, but modified by the application of an initial overhead irrigation equivalent to 1/4" (0.64 cm) rainfall; subsequent watering was accomplished by subirrigation. "NS" means non-selective within the test limits.

Table IV

Compound	GR ₁₅ Rate (Kg/Ha)	GR ₈₅ Rate (Kg/Ha)	
	Soybeans	Quackgrass	Yellow Nutsedge
A	>2.24	>2.39 (NS)	1.13 (1.98)
B	1.96	0.61 (3.21)	0.44 (4.45)
Ex. 1	2.67	0.17 (15.6)	0.17 (15.6)
Ex. 3	1.68	0.27 (6.22)	0.19 (8.84)
Ex. 4	1.12	0.13 (8.62)	0.17 (6.59)
Ex. 5	1.46	0.26 (5.62)	0.26 (5.62)
Ex. 13	2.13	0.20 (10.65)	0.17 (12.53)

Referring to the data in Table IV, it will be noted that every invention compound exhibited exceedingly higher selectivity factors (values in parentheses) against both quackgrass and yellow nutsedge in soybeans than the compounds of the prior art. In more particular, it is noted that the unit activities (relative phytotoxicity per unit of herbicide) of the invention compounds are markedly higher against quackgrass and yellow nutsedge than those of the prior art compounds, while maintaining crop safety. Of special note are the outstandingly high selectivity factors of the compounds of Examples 1 and 13.

Further comparative data showing the relative efficacy of the invention compounds vis-a-vis the above prior art compounds against quackgrass and yellow nutsedge in corn is presented in Table V.

Table V

Compound	GR ₁₅ Rate (Kg/Ha)	GR ₈₅ Rate (Kg/Ha)	
	Corn	Quackgrass	Yellow Nutsedge
A	>2.24	>2.39 (NS)	1.13 (1.98)
B	0.81	0.61 (1.33)	0.44 (1.84)
Ex. 1	0.76	0.17 (4.47)	0.17 (4.47)
Ex. 3	1.94	0.27 (7.19)	0.19 (10.2)
Ex. 4	0.76	0.13 (5.85)	0.17 (4.47)
Ex. 5	1.83	0.26 (7.04)	0.26 (7.04)
Ex. 13	2.24	0.20 (11.2)	0.17 (13.18)

Referring to the data in Table V, it will be noted that every invention compound exhibited exceedingly higher selectivity factors against both quackgrass and yellow nutsedge in corn than the compounds of the prior art. Again, it is noted that unit activities of the invention compounds were markedly higher against quackgrass and yellow nutsedge than those of the invention compounds while maintaining crop safety. Particular note is taken of the large selectivity factors for the compounds of Examples 3, 5 and 13, especially in comparison to those of the prior art compounds.

From the comparative data shown in Tables IV and V, it will be apparent that the invention compounds exhibited outstandingly higher and unexpectedly superior herbicidal efficacy against the herbicidally resistant perennial weeds quackgrass and yellow nutsedge in two major crops, i.e., soybeans and corn, than Compounds A and B, the most closely related compounds in the prior art.

Additionally, pre-emergence herbicidal data from other tests have established that compounds according to this invention also selectively control quackgrass, yellow nutsedge and/or other weeds in one or more of the crops cotton, peanuts, bush beans, wheat, sorghum and/or rape. For example, in Table VI are presented data showing the herbicidal selectivity of the compounds of Examples 1 and 3 against quackgrass in rape, snap beans, sorghum and wheat. Unless otherwise noted, greenhouse tests

in Table VI and in other tables below involved herbicide treatments by soil incorporation and an initial overhead irrigation followed by subirrigation as described above.

Table VI

5	Compound	<i>GR₁₅</i> Rate (Kg/Ha)				<i>GR₈₅</i> Rate (Kg/Ha)	5
		Rape	Snapbeans	Sorghum	Wheat	Quackgrass	
10	Ex. 1	0.86	—	—	—	0.12 (7.2)	10
		—	0.90	—	—	0.12 (7.5)	
		—	—	0.24	—	0.12 (2.0)	
	Ex. 3	—	—	—	0.21	0.12 (1.8)	
		1.9	—	—	—	0.28 (6.8)	
		—	2.24	—	—	0.28 (8.0)	
15		—	—	0.84	—	0.28 (3.0)	15
		—	—	—	0.28	0.28 (1.0)	

15 The compound of Example 1 was also tested in the field to determine its pre-emergence selectivity against foxtail (spp), barnyardgrass and white proso millet in a plurality of crops; the data (representing three replicate runs) are shown in Table VII for both surface application (S.A.) and soil incorporation (PPI, i.e., preplant incorporation) of the herbicide. The seeds were planted in a fine seedbed of silt loam of intermediate moisture. The seeds were planted at a depth of two inches (5.08 cm). First rainfall (0.2"; 0.51 cm) occurred the day following treatment, the second rain (0.25"; 64 cm) two days after treatment; cumulative rainfall 22 days after treatment was 1.8" (4.57 cm). 20
Observations were made 6 weeks after treatment.

Table VII
Percent Inhibition

Com- Pound	Appli- cation Mode	Rate (Kg/Ha)	Field Corn	Sweet Corn	Soy- beans	Pea- nuts	Cotton	Sorghum	Bush Bean	Rape	Fox- tail (Spp)	Barn- yard- grass	White Proso Millet
Ex. 1	S.A.	0.56	0	0	0	0	0	0	0	0	62	63	63
		1.12	0	0	5	0	13	3	8	0	92	93	83
		2.24	0	7	0	8	20	17	20	5	93	93	95
		4.48	15	27	13	22	25	77	32	33	98	98	97
	P.P.I.	0.56	0	0	5	17	13	32	23	0	75	75	23
		1.12	0	3	7	13	32	53	10	0	90	93	58
		2.24	5	27	20	15	37	92	37	15	98	98	80
		4.48	32	40	40	28	82	98	43	40	100	100	85

The data in Table VII show that, in general, the compound of Example 1 performed better as a selective herbicide in the surface application mode than when incorporated in the soil. In more particular, in the surface application tests the herbicide selectively controlled the three weeds in the test at application rates above 0.58 kg/ha (0.5 lb/A) while maintaining crop safety (i.e., up to about 15% maximum injury) in field corn and soybeans up to 4.48 kg/ha (4.0 lb/A) and safety in sweet corn, peanuts, and rape at greater than 2.24 kg/ha (2.0 lb/A) and in cotton, sorghum and bush beans at just under 2.24 kg/ha. In the PPI tests, the compound of Example 1 selectively controlled the foxtail species and barnyardgrass at less than 1.12 kg/ha (1.0 lb/A) while maintaining crop safety in field corn, peanuts and rape at rates up to 2.24 kg/ha and slightly below 2.24 kg/ha for soybeans.

Other field test data for the compound of Example 1 showed premergence selective control of other weeds in soybeans, corn, cotton and/or peanuts such as purple nutsedge, giant foxtail, yellow foxtail, lambsquarters, morningglory, cocklebur, velvetleaf, Pennsylvania smartweed, prickly sida, purslane, crabgrass, goosegrass, Texas panicum, Florida pusley and/or bristly starbur. It will be understood by those skilled in the art that not all of the named weeds are selectively controlled in all of the named crops under all conditions of climate, soil, moisture and/or herbicide application modes. Selective data for control of the foregoing weeds in the crops soybeans, corn, cotton and peanuts from a plurality of field tests in various locations under various conditions of soil, moisture, etc. is shown collectively in Tables VIII—XI, respectively. In the tables "WAT" means "Weeks after Treatment" of the plants with the herbicide, applied either by surface application ("S.A.") or by preplant

incorporation ("PPI") in the soil; application rate data for each crop/weed combination is shown in terms of GR₁₅ and GR₈₅ rates (defined above); the GR₁₅/GR₈₅ ratio resulting in the selectivity factor, "S.F."; "NS" indicates non-selective and a dash (—) indicates marginal or indeterminate selectivity, e.g., because actual GR₁₅ and GR₈₅ rates were higher or lower than maximum or minimum rates used in the indicated test). In Tables VIII—XI, a blank indicates that the plant species was not in a particular test plot or that the data was not obtained or was less significant than other data presented, e.g., some 3 WAT observations were omitted in favor of 6 WAT data or 6 WAT data omitted because the 3 WAT data was definitive.

Table VIII

Compound	Crop/Weed Combination	Appln. Mode	WAT	GR ₁₅ /GR ₈₅	S.F.
Ex. 1	Soybeans/Giant foxtail	S.A.	3		
			5	>4.48/<1.12	(>4.0)
		P.P.I.	3		
			5	>4.48/<1.12	(>4.0)
	Soybeans/Yellow foxtail		8	>4.48/<1.12	(>4.0)
		S.A.	6	2.52/2.8	(NS)
		P.P.I.	6	1.4/<1.12	(>1.3)
		S.A.	6	2.52/2.52	(1.0)
	Soybeans/Lambsquarters		6	1.4/2.8	(NS)
		S.A.	6	2.52/2.52	(1.0)
		P.P.I.	6	1.4/2.8	(NS)
		P.P.I.	8	4.48/2.32	(1.8)
	Soybeans/Pennsylvania Smartweed				

The data in Table VIII show that the compound of Example 1 selectively controlled giant and yellow foxtails, lambsquarters and Pennsylvania smartweed in soybeans from 6—8 WAT by either the S.A. or P.P.I. modes of application.

Table IX

Compound	Crop/Weed Combination	Appln. Mode	WAT	GR ₁₅ /GR ₈₅	S.F.
Ex. 1	Corn/Giant foxtail	S.A.	6	4.48/<1.12	(4.0)
		S.A.	6.5	>7.84/8.4	(NS)
		P.P.I.	6	4.76/1.96	(2.5)
		P.P.I.	6.5	>6.72/4.48	(1.5)
	Corn/Morning-glory	S.A.	3	>4.48/>4.48	(—)
		S.A.	3	>4.48/>4.48	(—)
		P.P.I.	3	4.76/>4.48	(—)
		P.P.I.	6	4.76/>4.48	(—)
	Corn/Cockle-	S.A.	3	>4.48/>4.48	(—)
		S.A.	6	>4.48/>4.48	(—)
		P.P.I.	3	4.76/>4.48	(—)
		P.P.I.	6	4.76/>4.48	(—)

The data in Table IX show that the compound of Example 1 selectively controlled giant foxtail in corn from 6—6.5 WAT by either the S.A. or P.P.I. mode of application; selectivity of morningglory and cocklebur was undetermined at test rates, but suppression of these weeds was exhibited.

Table X

5	Compound	Crop/Weed Combination	Appln. Mode	WAT	GR ₁₅ /GR ₈₅	S.F.	5
	Ex. 1	Cotton/Purple nutsedge	S.A.	6	3.64/1.68	(2.17)	
			S.A.	9	3.36/2.24	(1.5)	
			P.P.I.	6	3.36/4.2	(NS)	
10		Cotton/Prickly sida	S.A.	6	3.64/3.08	(1.18)	10
			S.A.	9	3.36/4.76	(NS)	
			P.P.I.	6	3.36/4.76	(NS)	
		Cotton/Purslane	S.A.	9	3.36/1.96	(1.7)	
15		Cotton/Crabgrass (smooth and hairy)	S.A.	7	1.4/<1.12	(>1.25)	15
			P.P.I.	7	0.84/1.12	(NS)	
		Cotton/Goosegrass	P.P.I.	7	0.84/<1.12	(—)	

20 The data in Table X show that the compound of Example 1 selectively controlled purple nutsedge and purslane up to 9 WAT, prickly sida up to 6 WAT and crabgrass up to 7 WAT; control of goosegrass was marginal or indeterminate.

In Table XI is presented pre-emergence activity data for the compound of Example 1 against three resistant annual weeds, i.e., Texas panicum, bristly starbur and Florida pusley in peanuts for

25 periods up to 12 WAT. The data in Table XI represent the average of three replicate runs in sandy loam soil having 1.3% organic matter, 79.2% sand and 10% clay; herbicide surface applied.

Table XI

Percent Inhibition

30	Compound	Rate (Kg/Ha)	Peanuts			Texas Panicum			Bristly Starbur			Florida Pusley			30
			WAT			WAT			WAT			WAT			
			4	8	12	4	8	12	4	8	12	4	8	12	
35	Ex. 1	2.24	5	0	0	50	67	40		78	40	0	90	35	
		3.36	10	0	0	68	63	55		85	60	0			
		4.48	17	7	0	85	88	78		95	100	0	95 95		

Reference to the data in Table XI shows that the compound of Example 1 selectively controlled Texas panicum in peanuts for up to 8 weeks and provided a large degree of control even at 12 WAT with about 4.48 kg/ha (4.0 lb/A); selective control of bristly starbur was achieved at 3.36 kg/ha (3.0 lb/A) for 8 weeks and complete control maintained for 12 WAT at 4.48 kg/ha (4.0 lb/A) and selective control of Florida pusley was achieved with less than 2.24 kg/ha at 8 WAT and with 4.48 kg/ha 95% control was attained at 12 WAT.

In other greenhouse tests, compounds according to this invention have shown selective control of a variety of annual and perennial weeds in various crops. As further illustration, the compound of Example 1 selectively controlled purple nutsedge in both corn and soybeans, the respective crop/weed GR₁₅/GR₈₅ ratios (expressed in kg/ha) being 0.67/0.25 (S.F.=2.7) in corn and 1.12/.25 (S.F.=4.5) in soybeans. The compound of Example 11 has shown selective control of yellow nutsedge and quackgrass in corn and soybeans. The respective crop/yellow nutsedge GR₁₅/GR₈₅ ratios being >2.24/0.05 (S.F.=2.4) in corn; 2.24/0.5 (S.F.=4.5) in soybeans and the respective GR₁₅/GR₈₅ ratios for both corn and soybeans in quackgrass being >2.24/0.5 (S.F.=>4.5). In a test against yellow nutsedge

in cotton, the GR_{15}/GR_{85} ratio (average of two replicates) was 1.96/0.95 (S.F.=2.1). Similarly, the compound of Example 13 exhibited selective control of yellow nutsedge in cotton and of quackgrass in wheat, the respective GR_{15}/GR_{85} ratios being 0.7/0.47 (S.F.=1.7) in cotton and 0.58/0.47 (S.F.=1.2) in wheat.

5 In one multi-crop test in the greenhouse, the compounds of Examples 1, 13, 14 and 15 were tested against yellow nutsedge in cotton, soybeans, corn and rice; each compound was non-selective with respect to yellow nutsedge in rice. However, markedly high selectivities for yellow nutsedge in cotton, soybeans and corn were shown for each of the compounds in the test; the respective GR_{15} and GR_{85} rates for these compounds are shown in Table XII; selectivity factors are shown in parenthesis after each crop. 10

Table XII

Compound	GR_{85} (Kg/Ha)		GR_{15} (Kg/Ha)		
	Yellow Nutsedge	Cotton	Soybeans	Corn	
Ex. 1	0.24	1.96 (8.2)	0.87 (3.6)	0.69 (2.9)	
Ex. 13	0.21	2.52 (12.0)	1.96 (9.3)	2.52 (12.0)	
Ex. 14	0.38	2.8 (7.4)	2.24 (5.9)	1.68 (4.4)	
Ex. 15	0.44	2.8 (6.4)	1.96 (4.5)	1.96 (4.5)	

20 The compounds of Examples 1 and 13—15 were further tested against quackgrass in wheat, soybeans and corn; each compound was found to be non-selective in wheat. The selectivity data for the above compounds against quackgrass in soybeans and corn is shown in Table XIII.

Table XIII

Compound	GR_{85} (Kg/Ha)		GR_{15} (Kg/Ha)	
	Quackgrass	Soybeans	Corn	
Ex. 1	0.07	0.81 (11.6)	0.69 (9.9)	
Ex. 13	0.36	1.68 (4.7)	1.68 (4.7)	
Ex. 14	0.45	1.46 (3.2)	2.52 (5.6)	
Ex. 15	0.75	1.96 (2.6)	2.24 (3.0)	

35 In yet another greenhouse tests of herbicidal efficacy, the compounds of Examples 1 and 3 were tested against a number of annual grasses including resistant weeds such as Texas panicum, seedling johnsongrass, shattercane, alexandergrass, wild proso millet (*panicum milicaeum*), red rice and itchgrass. The results of these tests are shown in Table XIV; selectivity factors are noted in parentheses; a dash indicating marginal or undetermined selectivity. 35

Table XIV

Com- pound	GR ₁₅ Rate (Kg/Ha)		GR ₉₅ Rate (Kg/Ha)						
	Soybeans	Texas Panicum	S. John- songrass	Shatter- cane	Alex- ander- grass	Wild Proso millet	Fall Panicum	Red Rice	Itch- grass
Ex. 1	>1.12	1.12 (>1.0)	0.14 (>8.0)	0.28 (>4.0)	0.56 (>2.0)	1.12 (>1.0)	<0.07 (>16.0)	0.2 (>5.6)	1.12 (>1.0)
Ex. 3	>1.12	>1.12 (—)	0.28 (>4.0)	0.93 (>1.2)	>1.12 (>1.0)	1.12 (—)	<0.07 (>16.0)	0.14 (>8.0)	>1.12 (—)

The data in Table XIV indicate that the compound of Example 1 selectively controlled every annual weed in the test in soybeans. The compound of Example 3 exhibited positive selective control against all weeds, except Texas panicum, wild proso millet and itchgrass at the maximum test rate of 1.12 kg/ha; a higher rate would have been required to determine selectivity of the compound against the three weeds not exhibiting selective control at the test rate.

A distinct advantage of a herbicide is its ability to function in a wide variety of soil types. Accordingly, data is presented in Table XV showing the herbicidal effect of the compound of Example 1 on quackgrass in soybeans in a wide variety of soil types of varying organic matter and clay content. The herbicide treatments were by soil incorporation with seeds planted 0.375 in. (0.95 cm) deep, with 0.25 in (0.64 cm) overhead irrigation. Observations were made about three weeks after treatment. Selectivity factors are shown in parentheses.

The data in Table XV show that the compound of Example 1 appears to be quite insensitive to soil types of varying organic matter content, exhibiting selective control of quackgrass in soybeans in soils ranging from 1.0% to 60% organic matter and clay content from at least 1.8% to about 37%. The data on soybeans in Wabash silty clay was indeterminate in this test. Also, the indicated selectivity factors in sarpy and Drummer silty clay and Florida muck are minimum values, since the maximum test rate was 2.24 kg/ha and the herbicide was safe on soybeans at the same rate above 2.24 kg/ha.

Laboratory tests were conducted to determine the resistance of herbicides according to this invention to leaching into the soil and resulting herbicidal efficacy. In these tests, the compounds of Examples 1 and 4—6 were formulated in acetone and then

Table XV

Compound	Soil Type	Organic Matter %	Clay %	GR ₁₅ Rate GR ₈₅ Rate	
				(Kg/Ha)	
				Soybeans	Quackgrass
Ex. 1	Ray silt loam	1.0	9.6	1.12	0.22 (5.1)
	Sarpy silty clay	2.3	30.35	>2.24	0.28 (>8.0)
	Wabash silty clay	4.3	33.0	—	0.28 (—)
	Drummer silty clay	6.0	37.0	2.24	0.11 (20.0)
	Florida sand	6.8	1.8	1.96	0.2 (9.8)
	Florida muck	60	—	>2.24	0.58 (>3.9)

sprayed at different concentrations onto a weighed amount of Ray silt loam and Drummer silty clay loam contained in pots having filter paper covering drainage holes in the pot bottoms. The pots containing the treated soil were subjected to leaching by placing on a turntable which rotated under two nozzle tips of a water container calibrated to deliver one inch (2.5 cm) of water per hour simulating rainfall. Leaching rates were adjusted by varying the amount of time on the turntable. Water was delivered to the soil in the pots and allowed to percolate through the filter paper and drainage holes. The pots were then allowed to sit for three days at ambient room temperature. The treated soil in the pots was then removed, crumbled and placed as a surface layer on top of other pots containing the above soils seeded with barnyardgrass seeds. The pots were then placed on greenhouse benches, sub-irrigated and allowed to grow for 2—3 weeks. Visual ratings of percent growth inhibition compared to control (untreated) pots and fresh weights for barnyardgrass were recorded; the data from the control represents six replications and that for the test compounds three replications; test data are shown in Table XVI. Fresh weights of the weed were not measured for the tests in Drummer silty clay loam soil.

Table XVI

Barnyardgrass

Com-pound	Rate (Kg/Ha)	Rain (cm)	Percent Inhibition		Fresh Weight (Grams)	
			Ray Silt Loam	Drummer Silt Clay	Ray Silt Loam	Drummer Silt Clay
Control	0	0			4.37	
	0	1.27			3.88	
	0	5.08			4.14	
	0	10.16			3.98	

Avg. 4.10

Table XVI (Cont'd)

Barnyardgrass

	Com- pound	Rate (Kg/Ha)	Rain (cm)	Percent Inhibition		Fresh Weight (Grams)		
				Ray Silt Loam	Drummer Silt Clay	Ray Silt Loam	Drummer Silt Clay	
5	Ex. 1	2.24	0	100	100	0	—	5
			1.27	100	100	0	—	
			5.08	95	100	0.20	—	
			10.16	35	100	2.65	—	
10		0.56	0	100	100	0	—	10
			1.27	100	100	0	—	
			5.08	70	100	1.21	—	
			10.16	33	100	2.73	—	
15		0.14	0	95	100	0.20	—	15
			1.27	96	97	0.17	—	
			5.08	58	95	1.74	—	
			10.16	20	20	3.26	—	
20	Ex. 4	2.24	0	100	100	0	—	20
			1.27	100	100	0	—	
			5.08	100	100	0	—	
			10.16	95	100	0.22	—	
25		0.56	0	100	100	0	—	25
			1.27	100	100	0	—	
			5.08	94	100	0.24	—	
			10.16	52	100	1.97	—	
30		0.14	0	99	81	0.05	—	30
			1.27	95	99	0.22	—	
			5.08	63	94	1.53	—	
			10.16	14	54	3.15	—	
35	Ex. 5	2.24	0	100	100	0	—	35
			1.27	100	100	0	—	
			5.08	92	100	0.31	—	
			10.16	71	100	1.18	—	
40		0.56	0	99	100	0.02	—	40
			1.27	100	100	0	—	
			5.08	89	100	0.44	—	
			10.16	17	96	3.41	—	
45		0.14	0	98	70	0	—	45
			1.27	90	96	0.41	—	
			5.08	38	85	2.77	—	
			10.61	12	47	3.62	—	
50	Ex. 6	0.24	0	100	100	0	—	50
			1.27	100	100	0	—	
			5.08	99	100	0.03	—	
			10.16	84	100	0.64	—	
55		0.56	0	99	50	0.04	—	55
			1.27	99	100	0.02	—	
			5.08	90	93	0.41	—	
			10.16	72	84	1.17	—	
60		0.14	0	84	12	0.66	—	60
			1.27	90	26	0.43	—	
			5.08	58	20	1.72	—	
			10.16	29	12	2.92	—	

Reference to the data in Table XVI indicate that the compounds of this invention were quite resistant to leaching into the soil under varying conditions of rainfall. In particular, at the 2.24 kg/ha (2.0 lb/A) rate of application, each of the invention compounds controlled barnyardgrass under the equivalent of 10.16 cm (4.0 inches) of rainfall in Ray silt loam and Drummer silt clay loam soil except the compounds of Examples 1 and 5 in Ray silt wherein control was maintained under the equivalent of 5.03 cm (2.0 inches) of rainfall. Even at the low application rate of 0.14 kg/ha (0.125 lb/A), the compounds of Examples 1, 4 and 5 controlled barnyardgrass in Drummer silty clay loam under the equivalent of 5.08 cm rainfall.

Toxicology studies on the compound of Example 1 have indicated the compound to be quite safe. It is slightly toxic (OLD_{50} 2300 mg/kg; MLD_{50} >5010 mg/kg) and has slight eye irritation and no skin irritation. No special handling procedures beyond normal precautions are deemed necessary.

Therefore, it will be appreciated from the foregoing detailed description that compounds according to this invention have demonstrated unexpected and outstandingly superior herbicidal properties both absolutely and relative to the most structurally-relevant compounds of the prior art.

More particularly, the compounds of this invention have proven to be outstanding selective herbicides, particularly in the control of hard-to-kill perennial and annual grasses in soybeans and corn, but also in peanuts, cotton and other crops. In more particular, compounds according to this invention exhibit outstanding control of perennial weeds such as quackgrass and nutsedges and resistant annual grasses such as Texas panicum, itchgrass, wild proso millet, alexandergrass, seedling johnsongrass, shattercane and/or red rice, while also controlling and/or suppressing other less-resistant annual grasses and perennials.

The herbicidal compositions of this invention including concentrates which require dilution prior to application contain at least one active ingredient and an adjuvant in liquid or solid form. The compositions are prepared by admixing the active ingredient with an adjuvant including diluents, extenders, carriers and conditioning agents to provide compositions in the form of finely-divided particulate solids, granules, pellets, solutions, dispersions or emulsions. Thus the active ingredient can be used with an adjuvant such as a finely-divided solid, a liquid of organic origin, water, a wetting agent, a dispersing agent, an emulsifying agent or any suitable combination of these.

The compositions of this invention, particularly liquids and wettable powders, preferably contain as a conditioning agent one or more surface-active agents in amounts sufficient to render a given composition readily dispersible in water or in oil. The incorporation of a surface-active agent into the compositions greatly enhances their efficacy. By the term "surface-active agent" it is understood that wetting agents, dispersing agents, suspending agents and emulsifying agents are included therein. Anionic, cationic and nonionic agents can be used with equal facility.

Preferred wetting agents are alkyl benzene and alkyl naphthalene sulfonates, fulfated fatty alcohols, amines or acid amides, long chain acid esters of sodium isothionate, esters of sodium sulfosuccinate, sulfated or sulfonated, fatty acid esters, petroleum sulfonates, sulfonated vegetable oils, ditertiary acetylenic glycols, polyoxyethylene derivatives of alkylphenols (particularly isooctylphenol and nonylphenol) and polyoxyethylene derivatives of the mono-higher fatty acid esters of hexitol anhydrides (e.g., sorbitan). Preferred dispersants are methyl cellulose, polyvinyl alcohol, sodium lignin sulfonates, polymeric alkyl, naphthalene sulfonates, sodium naphthalene sulfonate, and the polymethylene bisnaphthalene sulfonate.

Wettable powders are water-dispersible compositions containing one or more active ingredients, an inert solid extender and one or more wetting and dispersing agents. The inert solid extenders are usually of mineral origin such as the natural clays, diatomaceous earth and synthetic minerals derived from silica and the like. Examples of such extenders include kaolinites, attapulgite clay and synthetic magnesium silicate. The wettable powders compositions of this invention usually contain from about 0.5 to 60 parts (preferably from 5—20 parts) of active ingredient, from about 0.25 to 25 parts (preferably 1—15 parts) of wetting agent, from about 0.25 to 25 parts (preferably 1.0—15 parts) of dispersant and from 5 to about 95 parts (preferably 5—50 parts) of inert solid extender, all parts being by weight of the total composition. Where required, from about 0.1 to 2.0 parts of the solid inert extender can be replaced by a corrosion inhibitor or anti-foaming agent or both.

Other formulations include dust concentrates comprising from 0.1 to 60% by weight of the active ingredient on a suitable extender; these dusts may be diluted for application at concentrations within the range of from about 0.1—10% by weight.

Aqueous suspensions or emulsions may be prepared by stirring an aqueous mixture of a water-insoluble active ingredient and an emulsification agent until uniform and then homogenized to give stable emulsion of very finely-divided particles. The resulting concentrated aqueous suspension is characterized by its extremely small particle size, so that when diluted and sprayed, coverage is very uniform. Suitable concentrations of these formulations contain from about 0.1—60% preferably 5—50% by weight of active ingredient, the upper limit being determined by the solubility limit of active ingredient in the solvent.

In another form of aqueous suspensions, a water-immiscible herbicide is encapsulated to form microencapsulated phase dispersed in an aqueous phase. In one embodiment, minute capsules are formed by bringing together an aqueous phase containing a lignin sulfonate emulsifier and a water-

immiscible chemical and polymethylene polyphenylisocyanate, dispersing the water-immiscible phase in the aqueous phase followed by addition of a polyfunctional amine. The isocyanate and amine compounds react to form a solid urea shell wall around particles of the water-immiscible chemical, thus forming microcapsules thereof. Generally, the concentration of the microencapsulated material

5 will range from about 480 to 700 g/l of total composition preferably 480 to 600 g/l. 5

Concentrates are usually solutions of active ingredient in water-immiscible or partially water-immiscible solvents together with a surface active agent. Suitable solvents for the active ingredient of this invention include dimethylformide, dimethylsulfoxide, N-methylpyrrolidone, hydrocarbons and water-immiscible ethers, esters or ketones. However, other high strength liquid concentrates may be

10 formulated by dissolving the active ingredient in a solvent then diluting, e.g., with kerosene, to spray concentration. 10

The concentrate compositions herein generally contain from about 0.1 to 95 parts (preferably 5—60 parts) active ingredient, about 0.25 to 50 parts (preferably 1—25 parts) surface active agent and where required about 4 to 94 parts solvent, all parts being by weight based on the total weight of

15 emulsifiable oil. 15

Granules are physically stable particulate compositions comprising active ingredient adhering to or distributed through a basic matrix of an inert, finely-divided particulate extender. In order to aid leaching of the active ingredient from the particulate, a surface active agent such as those listed hereinbefore can be present in the composition. Natural clays, pyrophyllites, illite and vermiculite are

20 examples of operable classes of particulate mineral extenders. The preferred extenders are the porous, absorptive, preformed particles such as preformed and screened particulate attapulgite or heat expanded, particulate vermiculite and the finely-divided clays such as kaolin clays, hydrated attapulgite or bentonitic clays. These extenders are sprayed or blended with the active ingredient to form the

herbicidal granules.

25 The granular compositions of this invention may contain from about 0.1 to about 30 parts preferably from about 3 to 20 parts by weight of active ingredient per 100 parts by weight of clay and 0 to about 5 parts by weight of surface active agent per 100 parts by weight of particulate clay. 25

The compositions of this invention can also contain other additaments, for example, fertilizers, other herbicides, other pesticides, safeners and the like used as adjuvants or in combination with any of the above-described adjuvants. Chemicals useful in combination with the active ingredients of this invention include, for example, triazines, ureas, carbamates, acetamides, acetanilides, uracils, acetic acid or phenol derivatives, thiocarbamates, triazol benzoic acids, nitriles, biphenyl ethers and the like such as:

30 the above-described adjuvants. Chemicals useful in combination with the active ingredients of this invention include, for example, triazines, ureas, carbamates, acetamides, acetanilides, uracils, acetic acid or phenol derivatives, thiocarbamates, triazol benzoic acids, nitriles, biphenyl ethers and the like such as: 30

Heterocyclic Nitrogen/Sulfur Derivatives

35 2-Chloro-4-ethylamino-6-isopropylamino-s-triazine. 35
2-Chloro-4,6-bis(isopropylamino)-s-triazine.
2-Chloro-4,6-bis(ethylamino)-s-triazine.
3-Isopropyl-1H-2,1,3-benzothiadiazin-4-(3H)-one-2,2-dioxide.
3-Amino-1,2,4-triazole.
40 6,7-Dihydropyrido(1,2-a:2',1'-c)-pyrazidiinium salt. 40
5-Bromo-3-isopropyl-6-methyluracil.
1,1'-Dimethyl-4,4'-bipyridinium.

Ureas

45 N'-(4-chlorophenoxy)phenyl-N,N-dimethylurea. 45
N,N-dimethyl-N'-(3-chloro-4-methylphenyl) urea.
3-(3,4-dichlorophenyl)-1,1-dimethylurea.
1,3-Dimethyl-3-(2-benzothiazolyl) urea.
3-(p-Chlorophenyl)-1,1-dimethylurea.
1-Butyl-3-(3,4-dichlorophenyl)-1-methylurea.

50 Carbamates/Thiocarbamates

2-Chloroallyl diethyldithiocarbamate.
S-(4-chlorobenzyl)N,N-diethylthiocarbamate.
Isopropyl N-(3-chlorophenyl) carbamate.
S-2,3-dichloroallyl N,N-diisopropylthiocarbamate.
55 Ethyl N,N-dipropylthiocarbamate. 55
S-propyl dipropylthiocarbamate.

Acetamides/Acetanilides/Anilines/Amides

2-Chloro-N,N-diallylacetamide.
N,N-dimethyl-2,2-diphenylacetamide.
60 N-(2,4-dimethyl-5-[(trifluoromethyl)sulfonyl]amino)phenyl)acetamide. 60
N-Isopropyl-2-chloroacetanilide.

2',6'-Diethyl-N-methoxymethyl-2-chloroacetanilide.
 2'-Methyl-6'-ethyl-N-(2-methoxyprop-2-yl)-2-chloroacetanilide.
 α,α,α -Trifluoro-2,6-dinitro-N,N-dipropyl-p-toluidine.
 N-(1,1-dimethylpropynyl)-3,5-dichlorobenzamide.

5 Acids/Esters/Alcohols

- 2,2-Dichloropropionic acid.
 2-Methyl-4-chlorophenoxyacetic acid.
 2,4-Dichlorophenoxyacetic acid.
 Methyl-2-[4-(2,4-dichlorophenoxy)phenoxy]propionate.
 10 3-Amino-2,5-dichlorobenzoic acid. 10
 2-Methoxy-3,6-dichlorobenzoic acid.
 2,3,6-Trichlorophenylacetic acid.
 N-1-naphthylphthalamide acid.
 Sodium 5-[2-chloro-4-(trifluoromethyl)phenoxy]-2-nitrobenzoate.
 15 4,6-Dinitro-o-sec-butylphenol. 15
 N-(phosphonomethyl) glycine and its C₁₋₆ monoalkyl amine and alkaline metal salts and combinations thereof.

Ethers

- 2,4-Dichlorophenyl-4-nitrophenyl ether.
 20 2-Chloro- α,α,α -trifluoro-p-tolyl-3-ethoxy-4-nitrodiphenyl ether. 20

Miscellaneous

- 2,6-Dichlorobenzonitrile.
 Monosodium acid methanearsonate disodium methanearsonate.
 Fertilizers useful in combination with the active ingredients include, for example, ammonium
 25 nitrate, urea, potash and superphosphate. Other useful additives include materials in which plant 25
 organisms take root and grow such as compost, manure, humus, sand and the like.
 Herbicidal formulations of the types described above are exemplified in several illustrative
 embodiments below.

I. Emulsifiable Concentrates

- | | Weight Percent | |
|--|----------------|----|
| 30 A. Compound of Example No. 1 | 50.0 | 30 |
| Calcium dodecylbenzene sulfonate/polyoxyethylene ethers blend (e.g., Atlox® 3437F) | 4.85 | |
| Calcium dodecylbenzene sulfonate (FloMo 60H) | 0.14 | |
| 35 C ₉ aromatic solvent | 45.00 | 35 |
| | 100.00 | |
| | 85.0 | |
| B. Compound of Example No. 4 | 4.0 | |
| Calcium dodecyl sulfonate/alkylaryl polyether alcohol blend | 11.0 | |
| C ₉ aromatic hydrocarbons solvent | 100.00 | 40 |
| 40 | | |
| | 5.0 | |
| C. Compound of Example No. 6 | 1.0 | |
| Calcium dodecylbenzene sulfonate/polyoxyethylene ethers blend (e.g., Atlox 3437F) | 94.0 | |
| Xylene | 100.00 | 45 |
| 45 | | |

II. Liquid Concentrates

- | | Weight Percent | |
|------------------------------|----------------|----|
| A. Compound of Example No. 4 | 10.0 | |
| Xylene | 90.0 | |
| 50 | 100.00 | 50 |
| | 85.0 | |
| B. Compound of Example No. 5 | 15.0 | |
| Dimethyl sulfoxide | 100.00 | |

Weight Percent

C. Compound of Example No. 6		50.0	
N-methylpyrrolidone		50.0	
		100.00	
5	D. Compound of Example No. 7	5.0	5
	Ethoxylated castor oil	20.0	
	Rhodamine B	.5	
	Dimethyl formamide	74.5	
		100.00	
10	III. Emulsions		10
	A. Compound of Example No. 12	40.0	
	Polyoxyethylene/polyoxypropylene block with butanol (e.g., Tergitol® XH)	4.0	
	Water	56.0	
15		100.00	15
	B. Compound of Example No. 13	5.0	
	Polyoxyethylene/polyoxypropylene block copolymer with butanol	3.5	
	Water	91.5	
		100.00	
20	IV. Wettable Powders		20
		<i>Weight Percent</i>	
	A. Compound of Example No. 1	25.0	
	Sodium lignosulfonate	3.0	
	Sodium N-methyl-N-oleyl-taurate	1.0	
25	Amorphous silica (synthetic)	71.0	25
		100.00	
	B. Compound of Example No. 12	80.0	
	Sodium dioctyl sulfosuccinate	1.25	
	Calcium lignosulfonate	2.75	
30	Amorphous silica (synthetic)	16.00	30
		100.00	
	C. Compound of Example No. 13	10.0	
	Sodium lignosulfonate	3.0	
	Sodium N-methyl-N-oleyl-taurate	1.0	
35	Kaolinite clay	86.0	35
		100.00	
	V. Dusts		
		<i>Weight Percent</i>	
	A. Compound of Example No. 7	2.0	
	Attapulgite	98.0	
40		100.00	40
	B. Compound of Example No. 8	60.0	
	Montmorillonite	40.0	
		100.00	
	C. Compound of Example No. 9	30.0	
45	Bentonite	70.0	45
		100.00	

	D. Compound of Example No. 10 Diatomaceous earth	<i>Weight Percent</i> 1.0 99.0 100.00	
5	VI. Granules		5
	A. Compound of Example No. 1 Granular attapulgite (20/40 mesh)	<i>Weight Percent</i> 15.0 85.0 100.00	
10	B. Compound of Example No. 6 Diatomaceous earth (20/40)	30.0 70.0 100.00	10
	C. Compound of Example No. 12 Bentonite (20/40)	0.5 99.5 100.00	
15			15
	D. Compound of Example No. 13 Pyrophyllite (20/40)	5.0 95.0 100.00	
	VII. Microcapsules		
20	A. Compound of Example No. 1 encapsulated in polyurea shell wall Sodium lignosulfonate (e.g. Reax 88®B) Water	<i>Weight Percent</i> 49.2 0.9 49.9 100.00	20
25	B. Compound of Example No. 12 encapsulated in polyurea shell wall Potassium lignosulfonate (e.g., Reax® C-21) Water	10.0 .5 89.5 100.00	25
30	C. Compound of Example No. 13 encapsulated in polyurea shell wall Magnesium salt of lignosulfate (Treax, LTM®) Water	80.0 2.0 18.0 100.00	30

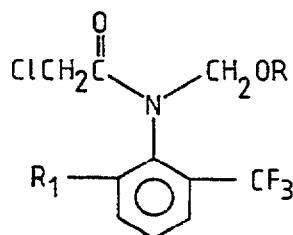
- When operating in accordance with the present invention, effective amounts of the acetanilides of this invention are applied to the soil containing the plants, or are incorporated into aquatic media in any convenient fashion. The application of liquid and particulate solid compositions to the soil can be carried out by conventional methods, e.g., power dusters, boom and hand sprayers and spray dusters. The compositions can also be applied from airplanes as a dust or a spray because of their effectiveness at low dosages. The application of herbicidal compositions to aquatic plants is usually carried out by adding the compositions to the aquatic media in the area where control of the aquatic plants is desired.
- The application of an effective amount of the compounds of this invention to the locus of undesired weeds is essential and critical for the practice of the present invention. The exact amount of active ingredient to be employed is dependent upon various factors, including the plant species and stage of development thereof, the type and condition of soil, the amount of rainfall and the specific acetanilide employed. In selective pre-emergence application to the plants or to the soil a dosage of from 0.02 to about 11.2 kg/ha, preferably from about 0.04 to about 5.60 kg/ha, or suitably from 1.12 to 5.6 kg/ha of acetanilide is usually employed. Lower or higher rates may be required in some instances. One skilled in the art can readily determine from this specification, including the above examples, the optimum rate to be applied in any particular case.

The term "soil" is employed in its broadest sense to be inclusive of all conventional "soils" as defined in Webster's New International Dictionary, Second Edition, Unabridged (1961). Thus the term refers to any substance or media in which vegetation may take root and grow, and includes not only earth but also compost, manure, muck, humus, sand and the like, adapted to support plant growth.

- 5 Although the invention is described with respect to specific modifications, the details thereof are not to be construed as limitations except to the extent indicated in the following claims. 5

Claims

1. Compound having the formula



- 10 wherein 10

R is C₁₋₅ alkyl or alkoxyalkyl or alkenyl or alkynyl having up to 5 carbon atoms and R₁ is hydrogen, methyl or ethyl; provided that:

when R₁ is hydrogen, R is isopropyl and

when R₁ is ethyl, R is ethyl, n-propyl or isopropyl.

- 15 2. Compounds according to Claim 1, wherein R is C₂₋₄ alkyl. 15

3. Compounds according to Claim 2, wherein R₁ is methyl.

4. Compound according to Claim 3 which is N-(ethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.

- 20 5. Compound according to Claim 3 which is N-(n-propoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide. 20

6. Compound according to Claim 3 which is N-(isopropoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.

7. Compound according to Claim 3 which is N-(isobutoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.

- 25 8. Compound according to Claim 2, wherein R₁ is ethyl. 25

9. Compound according to Claim 8 which is N-(ethoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide.

10. Compound according to Claim 8 which is N-(n-propoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide.

- 30 11. Compound according to Claim 8 which is N-(isopropoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide. 30

12. Compound according to Claim 2, wherein R₁ is hydrogen.

13. Compound according to Claim 12 which is N-(isopropoxymethyl)-2'-trifluoromethyl-2-chloroacetanilide.

- 35 14. Compound according to Claim 1, wherein R is a C₃₋₅ alkenyl radical. 35

15. Compound according to Claim 14 which is N-(allyloxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.

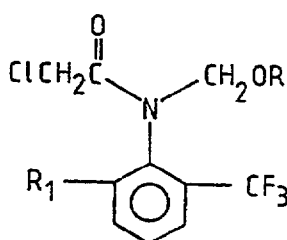
16. Compound according to Claim 1, wherein R is C₃₋₅ alkynyl radical.

- 40 17. Compound according to Claim 16 which is N-(propargyloxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide. 40

18. Compound according to Claim 1, wherein R is an alkoxyalkyl radical having up to 5 carbon atoms.

19. Compound according to Claim 18 which is N-(2-methoxyethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.

- 45 20. Herbicidal composition comprising an adjuvant and a herbicidally effective amount of a compound of the formula 45

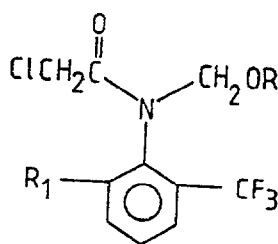


wherein

R is C₁₋₅ alkyl or alkoxyalkyl or alkenyl or alkynyl having up to 5 carbon atoms and

R_1 is hydrogen, methyl or ethyl; provided that
when R_1 is hydrogen, R is isopropyl and
when R_1 is ethyl, R is ethyl, n-propyl or isopropyl.

21. Composition according to Claim 20, wherein in said compound R is C_{2-4} alkyl.
- 5 22. Composition according to Claim 21, wherein in said compound R_1 is methyl. 5
23. Composition according to Claim 22, wherein said compound is N-(ethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
24. Composition according to Claim 22, wherein said compound is N-(n-propoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
- 10 25. Composition according to Claim 22, wherein said compound is N-(isopropoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide. 10
26. Composition according to Claim 22, wherein said compound is N-(isobutoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
27. Composition according to Claim 21, wherein R_1 is ethyl.
- 15 28. Composition according to Claim 27, wherein said compound is N-(ethoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide. 15
29. Composition according to Claim 27, wherein said compound is N-(n-propoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide.
30. Composition according to Claim 27, wherein said compound is N-(isopropoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide.
- 20 31. Composition according to Claim 21, wherein R_1 is hydrogen. 20
32. Composition according to Claim 31, wherein said compound is N-(isopropoxymethyl)-2'-trifluoromethyl-2-chloroacetanilide.
33. Composition according to Claim 20, wherein in said compound R is an alkenyl radical having up to 5 carbon atoms.
- 25 34. Composition according to Claim 33, wherein said compound is N-(allyloxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide. 25
35. Composition according to Claim 20, wherein in said compound R is an alkynyl radical having up to 5 carbon atoms.
- 30 36. Composition according to Claim 35, wherein said compound is N-(propargyloxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide. 30
37. Composition according to Claim 20, wherein in said compound R is an alkoxyalkyl radical having up to 5 carbon atoms.
38. Composition according to Claim 37, wherein said compound is N-(methoxyethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
- 35 39. Method for combatting undesirable plants in crops which comprises applying to the locus thereof a herbicidally effective amount of a compound having the formula 35



- wherein
- 40 R is C_{1-5} alkyl or alkoxyalkyl or alkenyl or alkynyl having up to 5 carbon atoms and 40
- R_1 is hydrogen, methyl or ethyl; provided that:
when R_1 is hydrogen, R is isopropyl and
when R_1 is ethyl, R is ethyl, n-propyl or isopropyl.
40. Method according to Claim 39, wherein in said compound R is C_{2-4} alkyl.
- 45 41. Method according to Claim 40, wherein in said compound R_1 is methyl. 45
42. Method according to Claim 41, wherein said compound is N-(ethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
43. Method according to Claim 41, wherein said compound is N-(n-propoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
- 50 44. Method according to Claim 41, wherein said compound is N-(isopropoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide. 50
45. Method according to Claim 41, wherein said compound is N-(isobutoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
46. Method according to Claim 40, wherein R_1 is ethyl.
- 55 47. Method according to Claim 46, wherein said compound is N-(ethoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide. 55

48. Method according to Claim 46, wherein said compound is N-(n-propoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide.
49. Method according to Claim 46, wherein said compound is N-(isopropoxymethyl)-2'-trifluoromethyl-6'-ethyl-2-chloroacetanilide.
- 5 50. Method according to Claim 40, wherein R₁ is hydrogen. 5
51. Method according to Claim 50, wherein said compound is N-(isopropoxymethyl)-2'-trifluoromethyl-2-chloroacetanilide.
52. Method according to Claim 39, wherein in said compound R is an alkenyl radical having up to 5 carbon atoms.
- 10 53. Method according to Claim 52, wherein said compound is N-(allyloxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide. 10
54. Method according to Claim 39, wherein in said compound R is an alkynyl radical having up to 5 carbon atoms.
55. Method according to Claim 54, wherein said compound is N-(propargyloxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide. 15
56. Method according to Claim 39, wherein in said compound R is an alkoxyalkyl radical having up to 5 carbon atoms.
57. Method according to Claim 56, wherein said compound is N-(methoxyethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
- 20 58. Method according to Claim 39, wherein said crops are corn, sorghum, soybeans, cotton, peanuts, bush beans, wheat and rape. 20
59. Method according to Claim 58, wherein said compound is N-(ethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
60. Method for combatting undesirable plants in corn which comprises applying to the locus thereof a herbicidally effective amount of N-(ethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide. 25
61. Method for combatting undesirable plants in soybeans which comprises applying to the locus thereof a herbicidally effective amount of N-(ethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.
- 30 62. Method for combatting undesirable plants in cotton which comprises applying to the locus thereof a herbicidally effective amount of N-(ethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide. 30
63. Method for combatting undesirable plants in rape which comprises applying to the locus thereof a herbicidally effective amount of N-(ethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide. 35
64. Method for combatting undesirable plants in snapbeans which comprises applying to the locus thereof a herbicidally effective amount of N-(ethoxymethyl)-2'-trifluoromethyl-6'-methyl-2-chloroacetanilide.

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