



PATENT SPECIFICATION

NO DRAWINGS

1028,818

1028,818



Date of Application and filing Complete Specification: April 11, 1963.

No. 14629/63.

Application made in United States of America (No. 188,981) on April 20, 1962.

Complete Specification Published: May 11, 1966.

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Index at acceptance:—A5 E(1C3B1, 1C3B3, 1C4A2, 1C4A3, 1C4A4, 1C4B2, 1C4B3, 1C4B4, 1C5A, 1C5B, 1C5D, 1C7B); C2 C(1D, 1Q2, 1Q6B1, 1Q6B2, 1Q8A, 1Q8C, 1Q9A, 1Q11J, 2B42C, 2B42G)

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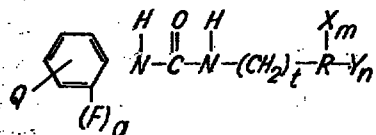
COMPLETE SPECIFICATION

Herbicidal Compositions comprising Urea Derivatives and methods for their use

We, E. I. DU PONT DE NEMOURS AND COMPANY, a Corporation organised and existing under the laws of the State of Delaware, United States of America, of Wilmington 98, State of Delaware, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to compositions of cycloalkyl phenyl ureas useful as plant growth regulants. More specifically, this invention relates to compositions of 1 - (cycloalkyl) - 3 - phenylurea derivatives and a method of retarding the growth of seedling weed grasses by applying thereto a growth retardant amount of the aforesaid composition.

The present invention includes a composition useful as a plant growth regulant which comprises as active agent one or more cycloalkyl phenylureas represented by the formula:



wherein R is a saturated cycloalkyl group containing from 5 to 8 carbon atoms or saturated bi-cycloalkyl group of 7 to 8 carbon atoms; X and Y are the same or different and are each a halogen atom or a methyl or ethyl group; t is 0 or 1; m is 0 or 1; n is 0 or 1; a is 0 or 1; Q is a hydrogen atom or a methyl group and with the limitation that when R is cyclopentyl, cycloheptyl, cyclooctyl or bi-cycloalkyl, Q if methyl must be in the meta position, and X and Y are

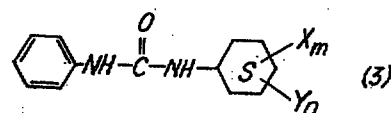
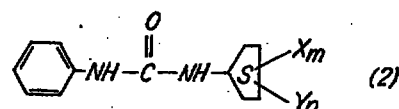
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the same or different and are each a halogen atom or a methyl group, or comprises as active agent an acid addition compound of a cycloalkyl phenylurea of the above formula, the composition containing a surface-active agent, or an inert solid diluent, or both.

We have found that the compositions described above remove effectively annual weed grasses from stands of broadleaf plants and perennial grasses.

The invention includes also a method of retarding the growth of seedling weed grasses which comprises applying to the locus of said grasses a growth retardant amount of a composition having as active ingredient either of the active agents specified above.

The substituted and unsubstituted cyclopentyl and cyclohexyl phenylureas of general formulae (2) and (3):



wherein X and Y and m and n have the same meaning as above, cause marked growth retardation of barnyard grass, foxtails, millet, Johnsongrass and Bermudagrass growing from seeds and can be used safely on cultivated annual grasses such as rye grass and rice.

The substituted cyclohexyl phenylureas of formula (3) above exhibit outstanding selec-

become a new invention (1962/221331) on Sept 4, 1962.

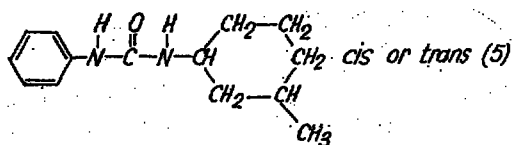
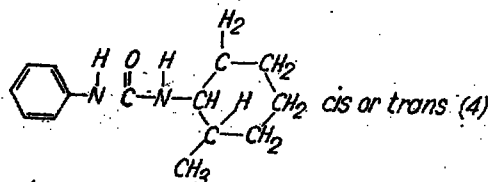
dicotyledonous



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tivity in retarding annual weed grasses growing among dicotyledonous plants and perennial grasses.

Particularly preferred compounds because of their outstanding growth retardation of annual weed grasses at low rates are represented by formulae (4) and (5):



In addition to the annual weed grasses some perennial weed plants such as Johnsongrass, Bermudagrass and the like are retarded when the perennial is growing from a seed. However, when the perennial has developed rhizomes a compound used in this invention has little effect on the plant. A home lawn owner desiring a Kentucky bluegrass, creeping fescue or bentgrass lawn when his neighbor has an established Bermudagrass lawn can successfully apply a compound of this invention to retard the growth of invading Bermudagrass from seeds while not worrying about affecting his neighbor's established lawn.

The 1 - (methylcycloalkyl) - 3 - (m - tolyl)ureas are safe to use at high rates on Kentucky bluegrass, bentgrass and creeping red fescue and will effectively retard the growth of millet.

The 1 - (methylcycloalkyl) - 3 - phenylureas cause marked retardation of annual weed grasses in agronomic and horticultural crops such as cucumbers, potatoes, squash, eggplant, tomatoes, peppers, red beets, cotton, soybeans, alfalfa, peanuts and the like.

An annual grass is defined in the Journal of Weed Society of America, Vol. 10, #3, July 1962, as any grass plant that completes its life cycle from seed in one year.

For the purposes of this specification, annual weed grasses, and perennial weed grasses growing from seed such as Bermudagrass and Johnsongrass are used generically as "seedling weed grasses".

Weeds are defined as any unwanted plant. For the purposes of this specification it is assumed that annual grasses that are com-

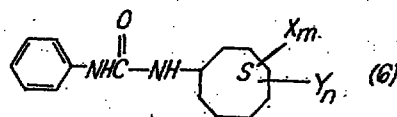
monly cultivated such as rye grass and rice are not weeds.

We have found, surprisingly, that the cycloalkylphenylurea compounds described above when applied in a pre-emergence or post-emergence treatment of an area in which crabgrass is growing, cause a marked growth retardation of the germinating or seedling crabgrass plant. It is believed that the retardation of growth of the crabgrass is caused by the effect on the crabgrass root system of the compounds described above; the crabgrass root system fails to grow and develop in a normal manner. Established turf grasses such as Kentucky bluegrass, bentgrass, creeping fescues and Bermudagrass show only slight retardation of growth and no injury. Therefore, crabgrass growing in a lawn treated with a compound above described is soon crowded out by desirable plants or destroyed by lack of an adequate supply of water and minerals necessary to sustain life.

The substituted and unsubstituted cyclopentyl and cyclohexyl phenylureas cause marked growth retardation of crabgrass growing in established lawns containing perennial grasses.

The cyclohexyl phenylureas of formula (3) above exhibit outstanding selectivity in retarding the growth of crabgrass around dicotyledonous plants.

The 1 - cyclooctyl - 3 - phenylureas of the following formula are particularly effective in retarding the growth of crabgrass without having an effect on other common lawn grasses:



wherein X and Y are the same or different and are each a halogen atom or a methyl group and m and n are each 0 or 1.

Particularly preferred compounds because of their outstanding action in retarding the growth of crabgrass at extremely low rates are the compounds represented by formulae (4) and (5) above.

The 1 - (methylcycloalkyl) - 3 - phenylureas are safe to use at even high rates on Kentucky bluegrass, bentgrass and creeping red fescue.

The 1 - cycloalkyl - 3 - (m - tolyl)ureas will retard the growth of crabgrass in established Bermudagrass lawns without deleterious effect on the Bermudagrass.

The 1 - (methylcycloalkyl) - 3 - (m - tolyl)ureas retard crabgrass growth in agronomic and horticultural crops.

We have further discovered that by coating seeds of perennial grasses such as bluegrass, bentgrass or fescue with a cycloalkyl phenylurea used in this invention and planting these seeds, there results a marked growth retardation of annual grass plants growing in the vicinity of the planted seed. Exceptional growth retardation is noted with such noxious annual grasses as crabgrass, foxtail and barnyard grass. Bermudagrass and Johnsongrass growing from seed are also retarded. Moreover, there is little retardation or injury to the seedling perennial grass plants emerging from seeds treated with the heptyl and octyl derivatives of the cycloalkyl phenylureas described herein.

The stickers which can be employed to attach finely ground or pulverulent cycloalkyl phenylurea to the seed surface can be selected from the adhesives conventionally used in formulating agricultural compositions, e.g. gums and resins and film-forming latexes. Aqueous solutions of natural and synthetic gums and resins can be employed such as gum Arabic, gum tragacanth, Guar gum, ammonium or alkali metal alginates, ammonium or alkali metal acrylates and polyvinyl alcohol, and cellulose derivatives, such as hydroxyethyl cellulose and methyl cellulose can be employed. Suspensions and solutions of starch and starch derivatives can also be used. Among the film-forming latexes that are useful as stickers are actual or synthetic rubber latex, polystyrene latex, polyacrylate latex, and polyethylene latex. Among these latexes a modified polyacrylic latex sold under the trade name of "Rhoplex" and a polyethylene latex sold under the trade name of "Plyac" are preferred. Water-soluble adhesives such as those which are conventionally used in paper manufacture can be employed advantageously. Clays that have binding action such as montmorillonite can also be employed.

The amount of sticker required in the compositions of this invention can range from 0.25% to 50% by weight based on the weight of the cycloalkyl phenyl ureas. Generally a level of 1 to 10% by weight of sticker based on the weight of the cycloalkyl phenyl ureas is preferred.

Preparation

The 1 - cyclohexyl - 3 - phenylureas can be made in the manner described in *Beaver et al.*, *J. Chem. Soc.*, 79, 1236 (1957). Substitutions on the cyclohexyl ring can be made by methods well known to the art. 1-Phenyl-3 - (2 - [cis], 4[trans] - dimethylcyclohexyl)urea and 1 - phenyl - 3 - (2[trans], 4[cis] - dimethylcyclohexyl)urea can be made in the manner described in A. Skita, *Ann* 427, 255 (1922) and in *Chem. Abstracts*, 16, 2321 (1922). 1 - (Methylcyclohexyl) - 3 - phenylurea can be made in the manner described by P. Mailhe, *Compt. rend.*, 174, 465 (1922) and in *Chem. Abstracts*, 16, 1942 (1922).

Additionally, the following compounds can be made in the manner described by A. Skita in *Ber.* 56B 1014 (1923) or *Chem. Abstracts*, 17, 3174 (1933).

1 - phenyl - 3 - cis(2 - methylcyclohexyl)urea 70
1 - phenyl - 3 - trans(2 - methylcyclohexyl)urea
1 - phenyl - 3 - cis(3 - methylcyclohexyl)urea
1 - phenyl - 3 - trans(3 - methylcyclohexyl)urea 75
1 - phenyl - 3 - cis(4 - methylcyclohexyl)urea
1 - phenyl - 3 - trans(4 - methylcyclohexyl)urea 80

The method for making the amino intermediates for the manufacture of other compounds used in this invention can be made by consulting *Beilstein* 12 (I) page 113 to page 118 (1933). This reference describes the manufacture of the following intermediates: cyclopentylamine, cyclohexylamine, 3-amino-1-methylcyclopentane and cyclooctylamine. On page 7 to 10 of *Beilstein* 12 (1929), the preparation of the following intermediates are described: 1 - amino - 1 - methylcyclopentane, 2 - amino - 1 - methylcyclopentane, cycloheptylamine and 1 - amino - 1 - methylcyclohexane. The following intermediates can be made by consulting *Beilstein* 12 (II) pages 14 to 22 (1950):

2 - chloro - 1 - aminocyclohexane
2 - bromo - 1 - aminocyclohexane
3 - bromo - 1 - aminocyclohexane
2 - amino - 1 - methylcyclohexane (cis and trans) 100
3 - amino - 1 - methylcyclohexane (cis and trans)
4 - amino - 1 - methylcyclohexane (cis and trans) 105
2 - amino - 1,3 - dimethylcyclohexane (cis, cis, cis)
2 - amino - 1,3 - dimethylcyclohexane (cis, cis, trans)
4 - amino - 1,3 - dimethylcyclohexane (trans, cis, trans) 110
5 - amino - 1,3 - dimethylcyclohexane (cis, cis, cis)
2 - amino - 1,4 - dimethylcyclohexane (cis, cis, cis) 115
2 - amino - 1,4 - dimethylcyclohexane (cis, cis, trans)
2 - amino - 1,4 - dimethylcyclohexane (cis, cis, trans)

Bicyclic amines are readily accessible from diene adducts. Some examples of bicyclic amine intermediates are as follows:

Alder et al, *Ber.*, 71B, 2451 (1938)
endo norbornylamine
exo norbornylamine 125
3-methylnorbornylamine
Semmler et al, *Ber.* 41, 866; and *Ber.* 41, 127.
4-amino-2-methylbicyclo[1,2,3]octane
2 - amino - 1,7 - dimethylbicyclo[1,2,2]-heptane 130



Am

Komppa, *Ann.*, 366, 75

3 - amino - 2,2 - dimethylbicyclo[1,2,2]-heptane

Beilstein, 12, 1st supplement, 125 (1933)

5 5 - amino - 2,2 - dimethylbicyclo[1,2,2]-heptane

Seka, et al, *Ber.*, 75, 1379

2-amino-bicyclo[2,2,2]octane

Komppa, et al. *Ann.*, 512, 172

10 2-aminobicyclo[1,2,2]heptane

The ring halogenated amines can be prepared by the procedure described in British Patent 885,878, published December 28, 1961. This consists of halogenation of the hydrogen halide or sulfate salts in carbon tetrachloride. The exact position of the halogen is not known. Mono and dihalogenated products are obtained by controlling the amount of halogen added.

20 The alkyl substituted cyclohexyl amines are easily prepared by catalytic hydrogenation of the alkyl anilines.

Typical of the method of preparation of the compounds of this invention is the reaction of a phenyl- or a substituted phenyl-isocyanate with the appropriate amine in an inert solvent such as toluene. The exothermic reaction is usually controlled by the rate of addition of the isocyanate to the amine or by external cooling. The products can be isolated by evaporation of the solvent from the reaction mixture, or by diluting the reaction mixture with an aliphatic hydrocarbon such as n-pentane. The products are crystalline solids. For example, phenylisocyanate reacts with cyclooctylamine to yield a novel crystalline solid, 1 - cyclooctyl - 3 - phenylurea, which melts at 154—155°C.

Mixtures of isomers can also be used in the procedures of this invention. The economical means of preparing the cycloalkylamines, such as 2- or 3-methylcyclohexylamine, results in a mixture of cis and trans isomers. No separation of the isomers is needed.

45 It is interesting to note that the compounds used in this invention have low solubility in water. Therefore, they will not ordinarily disappear rapidly from the soil due to rain or high soil moisture content.

50 The compounds used in this invention may be the 1/1 addition compounds with acids of the cycloalkyl phenylurea derivatives, for example, such acids as described in co-pending Patent Application No. 47731/62 (Serial 1,028,817). Particularly of interest are trichloroacetic acid and dodecylbenzenesulphonic acid.

60 The addition compounds can be prepared by mixing equal molar proportions of the acid and urea in an inert anhydrous solvent such as xylene at room temperature.

Additionally, phenolcycloalkylphenylurea complexes are formed from the phenols which are described in U.S. Patent No. 2,705,195.

Compositions

Compositions of this invention may be formulated by mixing a compound of this invention together with one or more surface-active agents.

The surface-active agent used in this invention can be a wetting, dispersing or an emulsifying agent which will assist dispersion of the compound. The surface-active agent or surfactant can include such anionic, cationic, and non-ionic agents as have heretofore been generally employed in plant control compositions of similar type. Suitable surface-active agents are set out, for example, in U.S. Patents Nos. 2,426,417, 2,655,447, 2,412,510, and 2,139,276. A detailed list of such agents is set forth in "Detergents and Emulsifiers—Up to Date" (1962) by John W. McCutcheon, Inc., and Bulletin E-607 of the Bureau of Entomology and Plant Quarantine of the U.S. Department of Agriculture.

Suitable surface-active agents for use in compositions of the present invention are: polyethylene glycol fatty esters and fatty alkylol amide condensates, alkyl aryl sulfonates, fatty alcohol sulfates, dialkyl esters of sodium sulfosuccinate, fatty acid esters of sodium isethionate, polyoxyethylene thioethers and long chain quaternary ammonium chloride compounds.

Surface-active dispersing agents such as sodium lignin sulfonates, low viscosity methyl cellulose, polymerized sodium salts of alkyl naphthalene sulfonic acids are also suitable in the growth retardant compositions of this invention.

Among the more preferred surfactants are the anionic and non-ionic types. Among the anionic surface-active agents, preferred ones are alkali metal or amine salts of alkyl benzene sulfonic acids such as dodecylbenzene sulfonic acid, sodium lauryl sulfate, alkyl naphthalene sulfonates, sodium N - methyl - N - oleoyl-taurate, oleic acid ester of sodium isethionate, dioctyl sodium sulfosuccinate, sodium dodecyl-diphenyloxide disulfonate. Among the non-ionic compounds, preferred members are alkyl phenoxy poly(ethyleneoxy) ethanols such as nonyl phenol adducts with ethylene oxide; trimethyl nonyl polyethylene glycol ethers, polyethylene oxide adducts of fatty and rosin acids, long chain alkyl mercaptan adducts with ethylene oxide and polyethylene oxide adducts with sorbitan fatty acid esters.

In general, less than 10% by weight of the surface-active agent will be used in compositions of this invention and ordinarily the amount of surface-active agents will range from 1—5% but may even be less than 1% by weight.

Additional surface-active agents can be added to the above formulation to increase the ratio of surface-active agent: active agent up to as high as 5:1 by weight. Normally

Anionic



the purpose of adding higher amounts of surfactant is to increase the growth retardant effect of the active compounds. When used at higher rates it is preferred that the surfactant be present in the range of one fifth to five parts surfactant for each one part of active agent.

Plant growth retardant compositions of this invention can contain, in addition to a surfactant, finely divided inert diluents such as talcs, natural clays including attapulgite clay and kaolinite clay, pyrophyllite, diatomaceous earths, synthetic fine silicas, calcium silicate, carbonates, calcium phosphates, sulfur, lime and such flours as walnut shell, wheat, redwood, soybeans and cottonseed. The amount of the finely divided inert solid diluent can vary widely but will generally range from 10 to 98% by weight of the growth retardant composition. The particle size can vary considerably but will ordinarily be somewhat under 50 microns in the finished formulation.

Plant growth regulant compositions of this invention may comprise an active agent and an inert solid diluent formulated into granules or pellets. In such compositions, the diluent will generally range from 65 to 99% and the active ingredient can range from 1 to 35%. It should be understood that it will not be necessary to include a surfactant in the granular and pelletized composition. To prepare granules the growth retardant can be dissolved in a solvent, and this solution can be sprayed over pre-formed clay granules, expanded vermiculite or the like to distribute the active ingredient over and throughout the granular mass. Such granules can range in particle size of from +60 mesh to +4 mesh, (U.S. Standard sieve sizes), and an active ingredient content of 1 to 6% is preferred. It is also possible to make such granules by mixing the finely divided diluent and finely divided growth retardant, for instance by gringing together, and then forming granules by adding water, tumbling, and drying the resulting spheres. Pellets can be prepared by extruding a mixture that comprises the cycloalkylphenylureas, pelleting clay diluent and water into strands, cutting these, and drying the product. Pellet size can range from 10 mesh to larger shapes such as $\frac{3}{8}$ inch cubes. Pellets preferably contain from 5 to 35% of the cycloalkylphenylureas. In addition to the diluents, pelletized and granular compositions can contain conventional additives such as binders, surfactants and the like.

In addition to the formulation described above, sand ground formulations can be prepared using the method described in Patent Specification No. 686,234. Using the methods described in this specification, fine particles of the active compounds within the scope of this invention will be dispersed evenly in a diluent.

Emulsifiable oil solutions can also be em-

ployed with one or more of the cycloalkylphenylureas of this invention. In these plant growth regulant compositions, the surface-active agent and an oil form a liquid which can be conveniently poured and measured. Such solutions can be mixed with water at the point of application to form an emulsion containing the growth retardant and the surface-active agent.

Application

The compounds of this invention can be applied directly to the soil as pre-emergence or as post-emergence treatments to plant foliage, for example, crabgrass, or they can be mixed intimately with the soil. Rates of application will usually be in the range of $\frac{1}{2}$ to 20 pounds per acre and will preferably be used at the rate of 1 to 15 pounds per acre. In applications to crabgrass, for example, rates as high as 24 pounds have been used in tests on established turf grasses without undesirable injury.

More specifically, as a pre-emergence application on crop lands, 2 to 8 pounds per acre of a compound used in this invention will retard the growth of crabgrass, barnyard grass, foxtail, millet and Johnsongrass and Bermudagrass growing from seed. There is a good safety margin on crop plants such as cucumbers, cotton, potatoes, flax, squash, lima beans, egg-plant, red clover, rye grass, rice, tomatoes, lespedza, peppers, soybeans, red beets, alfalfa, sugar beets, green beans, carrots, peanuts, and okra. The seedling weeds usually make an appearance above ground and then fail to develop showing symptoms of severe growth retardation. It is particularly surprising to note that the compounds used in this invention will retard annual grass weeds, such as, for example, crabgrass, barnyard grass and foxtail without harming cultivated annual grasses such as ryegrass and rice.

After an application of 4 to 16 pounds of active ingredient per acre on areas where perennial grasses are established and at a time before the weeds appear, barnyard, millet, and foxtail weed growth is severely retarded. As an application on established growing lawns before crabgrass appears, growth of the crabgrass is retarded with 2 to 16 pounds of active ingredient per acre. There is no injury to established Kentucky bluegrass, bentgrass, creeping red fescue or Bermudagrass growing from rhizomes. The weeds make an appearance above the ground and then fail to develop.

After a pre-emergence application of 2 to 8 pounds per acre of active ingredient on areas, e.g., lawns, seeded with perennial grasses at a time before the perennial grasses and weeds have appeared, development of crabgrass is prevented and the development of barnyard grass and foxtail grass is severely retarded. There is no undesirable effect on the development of such perennials as Kentucky

bluegrass, bentgrass or creeping red fescue.

As a pre-emergence application on crop lands 3 to 8 pounds per acre of active ingredient will retard markedly the development of millet, barnyard grass and Johnsongrass growing from seed. Seedlings that germinate prior to treatment and have reached the 1 to 2-leaf stage are retarded. As a directed post-emergence application on crop lands at any time from the moment the plants emerge to the three-leaf stage, 2 to 8 pounds per acre of active ingredient will prevent the further development of crabgrass. There is no perceptible injury to crop plants such as cucumbers, cotton, potatoes, flax, squash, lima beans, eggplant, red clover, tomatoes, lespedza, peppers, soybeans, red beets, alfalfa, sugar beets, green beans, carrots, peanuts, and okra.

As an application on areas containing newly seeded perennial grasses, e.g., lawns, at a time after the perennial grasses and weeds, e.g., crabgrass, have appeared, 2 to 8 pounds per acre of active ingredient will retard markedly the development of crabgrass, barnyard and seedling Johnsongrass. There is no perceptible injury to Kentucky bluegrass, bentgrass, creeping red fescue or Bermudagrass growing from established rhizomes.

As an application on established lawns after the crabgrass appears at emergence to 3-leaf stage, 3 to 16 pounds per acre of active ingredient will prevent the further development of crabgrass without injury to the established lawn grasses.

The compounds of this invention can also be applied admixed or in common solution with other chemicals which are used in agronomic and horticultural management and are compatible with the compounds of this invention. Such chemicals may be, but are not restricted to, the classes of chemicals com-

monly known as plant nutrients, fertilizers, insecticides, fungicides, herbicides and nematocides. Typical of the insecticides that can be used are:

1,2,3,4,10,10 - hexachloro - 6,7 - epoxy - 1,4,4a,5,6,7,8,8a - octahydro - 1,4,5,8 - dimethanonaphthalene (Dieldrin),
1,2,4,5,6,7,8,8 - octachloro - 4,7 - methano-3a,4,7,7a - tetrahydroindane (Chlordane),
1,1,1 - trichloro - 2,2 - bis(p - methoxy - phenyl)ethane (Methoxychlor),
1,1,1 - trichloro - 2,2 - bis(p - chlorophenyl)ethane (DDT), and
1 - naphthyl - N - methylcarbamate.

Fungicides that can be used in conjunction with the compounds of this invention include: metal salts of ethylene bisdithiocarbamic acid, e.g., sodium manganese, zinc and iron salts; N - trichloromethylmercapto - 4 - cyclohexene - 1,2 - dicarboximide; N - trichloromethylthiophthalimide; 2,3 - dichloro - 1,4 - naphthoquinone; 2,3,5,6 - tetrachloro - 1,4 - benzoquinone; 2,4 - dichloro - 6 - (p - chloroanilino) - s - triazine; copper A; metal salts of alkyl and dialkyl dithiocarbamic acids, e.g., the Zn, Na, K, Fe, Mn, Ni salts; zinc pyridinethione; S - (1 - oxido - 2 - pyridyl) - isothiuronium chloride; tetramethylthiuran disulfide; hydroxymercurichlorophenol and mixtures of these last two.

The control of annual weed grasses, e.g., crabgrass, with cycloalkylphenylureas often can be advantageously accomplished together with the use of conventional herbicides in situations where crabgrass is growing with weeds controlled by the second herbicide.

Illustrative of herbicides that can be used in conjunction with the compounds of this invention in sprays and granular formulations to take weeds out of desirable plants are the following:



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65

70

75

80

tetramethylthiuran



	<i>Herbicide</i>	<i>Desirable Plants</i>
	2,4 - dichlorophenoxyacetic acid	Lawn turf grasses, Sugar cane and Roadside Turf grasses
5	2,4,5 - trichlorophenoxyacetic acid	Lawn turf grasses and Roadside turf grasses
	2,4,5 - trichlorophenoxypropionic acid	Lawn turf grasses and Roadside turf grasses
	3 - amino - 2,5 - dichlorobenzoic acid	Soybeans
10	2,4 - bis(isopropylamino) - 6 - methylmercapto - s - triazine	Soybeans, cotton and sugar cane
	1 - n - butyl - 3 - (3,4 - dichlorophenyl) - 1 - methylurea	Ornamentals and Lawn turf grasses
	2 - chloro - 4,6 - bis(ethylamino) - s - triazine	Ornamentals
15	2 - chloro - 4 - ethylamino - 6 - isopropylamino - s - triazine	Sugar cane and pineapples
	3 - (p - chlorophenyl) - 1,1 - dimethylurea	Pineapple and sugar cane
	4 - (2,4 - dichlorophenoxy) - butyric acid	Alfalfa and other legumes
20	2 - methyl - 4 - chlorophenoxybutyric acid	Alfalfa and other legumes
	3 - (3,4 - dichlorophenyl) - 1,1 - dimethylurea	Sugar cane, pineapple, grass seed crops and alfalfa
	N - (3,4 - dichlorophenyl)methacrylamide	Cotton
	3 - (3,4 - dichlorophenyl) - 1 - methoxy - 1 - methylurea	Soybeans
25	1,2 - dihydropyridazine - 3,6 - dione	Roadside turf grasses
	2 - ethylamino - 4 - isopropylamino - 6 - methylmercapto - s - triazine	Sugar cane
	Sodium 2,4 - dichlorophenoxyethylsulfate	Soybeans



itself

The above may be in the form of salts or non-volatile ester formulations. Also contact herbicides may be included, such as cacodylic acid, potassium cyanate, dinitro-sec.-butyl phenol and 1,1'-ethylene-2,2'-bipyridylum dibromide (diquat). It is understood that the compounds and compositions of this invention are applied to the "locus" of the undesirable weeds. By "locus" is meant the plant itself when visible above the ground and when the plant is not visible, the immediate area of soil where the undesirable plant is developing.

The following additional examples, in which all percentages unless otherwise indicated are by weight, are provided so as to more clearly explain this invention.

Amine

chlorocyclooctylamine
dibromocyclooctylamine
methylcyclooctylamine
dimethylcyclooctylamine
fluorocyclooctylamine
dichlorocyclooctylamine

EXAMPLE 2

A solution of 17 parts by weight of cycloheptylamine in 150 parts of toluene is treated with 18.9 parts by weight of phenylisocyanate in 25 parts of toluene over a 22-minute period. The temperature increases from 22—55° C. The mixture is stirred for 1 hour and the solid

EXAMPLE 1

A solution of 25.4 parts by weight of cyclooctylamine in 200 parts of toluene is treated over an 18-minute period with 238 parts by weight of phenylisocyanate dissolved in 50 ml. of toluene. The temperature increases from 22—47° C. The clear yellow solution is stirred for 1½ hours. The solution is poured into an equal volume of n-pentane. The solid is filtered and washed with n-pentane. There is obtained 40.5 parts by weight of essentially pure 1 - cyclooctyl - 3 - phenylurea, m.p. 154—155° C.

TABLE I

By the above procedure using the following amines the corresponding phenylureas are obtained.

Urea

1-chlorocyclooctyl-3-phenylurea
1-dibromocyclooctyl-3-phenylurea
1-methylcyclooctyl-3-phenylurea
1-dimethylcyclooctyl-3-phenylurea
1-fluorocyclooctyl-3-phenylurea
1-dichlorocyclooctyl-3-phenylurea

collected by filtration. After washing with n-pentane there is obtained 32.5 parts by weight of 1 - cycloheptyl - 3 - phenylurea, m.p. 187.5—188.5° C.

Analysis calc'd. for $C_{14}H_{20}N_2O$: % N 12.1
Found: % N 12.3

TABLE II

By the above procedure using the following amines the corresponding phenylureas are obtained:

	Amine	Urea
5	chlorocycloheptylamine dichlorocycloheptylamine bromocycloheptylamine methylcycloheptylamine	1-chloro-cycloheptyl-3-phenylurea 1-dichlorocycloheptyl-3-phenylurea 1-bromocycloheptyl-3-phenylurea 1-methylcycloheptyl-3-phenylurea

EXAMPLE 3

- 10 A solution of 2-methylcyclohexylamine (cis-trans-mixture) (22.6 parts by weight) in 200 parts of toluene is treated with 23.8 parts by weight of phenylisocyanate over 44 minutes. The temperature increases from 23—41° C.
- 15 After stirring for 2 hours the solution is evaporated *in vacuo* on a steam bath. The residue crystallizes on cooling. After washing with n-pentane, the product melts at 120—122° C. It is a mixture of cis and trans 1-(2-methylcyclohexyl)-3-phenylurea.
- Analysis calc'd. for $C_{14}H_{20}N_2$: % N 12.1
Found: % N 12.0

TABLE III

By the above procedure using the following amines the corresponding phenylureas are obtained:

	Amine	Urea
25	2-chlorocyclohexylamine 2-bromocyclohexylamine 3-bromocyclohexylamine	1-(2-chlorocyclohexyl)-3-phenylurea 1-(2-bromocyclohexyl)-3-phenylurea 1-(3-bromocyclohexyl)-3-phenylurea
30	dichlorocyclohexylamine 2-iodocyclohexylamine	1-(dichlorocyclohexyl)-3-phenylurea 1-(2-iodocyclohexyl)-3-phenylurea

EXAMPLE 4

- 35 A solution of 17 parts by weight of cyclopentylamine in 150 parts of toluene is treated with 23.8 parts by weight of phenylisocyanate over 20 minutes. The temperature increases from 24—70° C. Stir for 1 hour after the addition and filter off the solids. After washing with n-pentane and drying, there is obtained 38.7 parts by weight of 1-cyclopentyl-3-phenylurea, m.p. 207.5—208.5° C.
- Analysis Calc'd. for $C_{12}H_{16}N_2$: % N 13.7
Found: % N 13.7

TABLE IV

By the above procedure using the following amines the phenylureas can be prepared:

	Amine	Urea
45	2-methylcyclopentylamine chlorocyclopentylamine dichlorocyclopentylamine bromocyclopentylamine	1-(2-methylcyclopentyl)-3-phenylurea 1-(chlorocyclopentyl)-3-phenylurea 1-(dichlorocyclopentyl)-3-phenylurea 1-(bromocyclopentyl)-3-phenylurea
50		

EXAMPLE 5

- 55 A suspension of 13.2 parts by weight of 2-norbornylamine-hydrochloride in 150 ml. of toluene is treated with 10 parts by weight of triethylamine. The triethylamine hydrochloride is filtered from the mixture and the filtrate is treated with 10.7 parts by weight of phenylisocyanate. After stirring for 2 hours the solid is filtered from the reaction mixture. There is obtained 4.3 parts by weight of 1-(2-norbornyl)-3-phenylurea m.p. 195—215° C. (Mixture of endo and exo isomers).
- Analysis calc'd. for $C_{14}H_{18}N_2$: % N 12.1
Found: % N 12.1

TABLE V

By the above procedure using the following amines the corresponding phenylureas are obtained:

	Amine	Urea
5	endonorbonylamine	endo-1-(2-norbonyl)-3-phenylurea
	exonorbonylamine	exo-1-(2-norbonyl)-3-phenylurea
	3-methylnorbonylamine	1-(3-methyl-2-norbonyl)-3-phenylurea
	2-amino-1,7-dimethylbicyclo [1,2,2] heptane	1-(1,7-dimethyl-2-norbonyl)-3-phenylurea
	3-amino 2,2-dimethylbicyclo [1,2,2] heptane	1-(3,3-dimethyl-2-norbonyl)-3-phenylurea
10	5-amino-2,2-dimethylbicyclo [1,2,2] heptane	1-(5,5-dimethyl-2-norbonyl)-3-phenylurea
	chloronorbonylamine	1-(chloronorbonyl)-3-phenylurea
	dichloronorbonylamine	1-(dichloronorbonyl)-3-phenylurea

EXAMPLE 6

A solution of 19.8 parts by weight of cyclohexylamine in 150 parts of toluene is treated with 26.6 parts by weight of m-tolylisocyanate in 25 parts of toluene. The addition requires 7 minutes and the temperature increases from 22—52° C. After stirring for ½ hour the product is filtered and washed with n-pentane. Essentially pure 1-cyclohexyl-3-m-tolylurea is obtained which melts at 150—152° C. (41.4 parts by weight).

Analysis calc'd. for $C_{14}H_{20}N_2$: % N 12.1
Found: % N 12.1

EXAMPLE 7

A solution of 12.4 parts by weight of cyclooctylamine in 100 parts of toluene is reacted with 13 parts of m-tolyl-isocyanate in 25 parts of toluene. Addition time is 8 minutes. The reaction is very exothermic and ice bath cooling is required to keep the temperature below 40° C. After stirring for ½ hour the reaction mixture is filtered and the product washed with n-pentane and dried. There is obtained 21.5 parts of 1-cyclooctyl-3-m-tolylurea, m.p. 87—89° C.

Analysis calc'd. for $C_{16}H_{24}N_2$: % N 10.7
Found: % N 10.5

TABLE VI

Using the procedure described in Examples 6 and 7 the following m-tolylureas can be prepared:

	Amine	Product
45	cyclopentylamine	1-cyclopentyl-3-m-tolylurea
	2-methylcyclohexylamine	1 - (2 - methylcyclohexyl) - 3 - m - tollylurea m.p. 147.5—150° C.
	3-methylcyclohexylamine	1 - (3 - methylcyclohexyl) - 3 - m - tollylurea m.p. 165—167.5° C.
50	cycloheptylamine	1 - cycloheptyl - 3 - m - tollylurea m.p. 145—147° C.
	1-(2-chlorocyclohexyl)-amine	1 - (2 - chlorocyclohexyl) - 3 - m - tollylurea

TABLE VII

Using the procedure described in Example 6 with the following isocyanates and amines the following ureas can be prepared:

	Amine	Isocyanate	Product
55	2-methylcyclohexyl-	o-tolyl	1-(2-methylcyclohexyl)-3-o-tolylurea
	2-methylcyclohexyl-	p-tolyl	1-(2-methylcyclohexyl)-3-p-tolylurea
	2-ethylcyclohexyl-	phenyl	1-(2-ethylcyclohexyl)-3-phenylurea
	2-i-propylcyclohexyl-	phenyl	1-(2-i-propylcyclohexyl)-3-phenylurea
60	2-s-butylcyclohexyl-	phenyl	1-(2-sec.-butylcyclohexyl)-3-phenylurea

EXAMPLE 8

The following granular formulation can be used for most solid cycloalkyl phenylureas used in this invention melting above 90° C. A wettable powder is first prepared by micro-pulverizing the following ingredients:

65	1-(3-methylcyclohexyl)-3-m-tolylurea	50%
	attapulgit clay	48%
70	dioctyl sodium sulfosuccinate	1%
	sodium lignin sulfonate	1%

The wettable powder is distributed over the surface of #4 vermiculite by tumbling together. To make the combination more adherent the mix is sprayed with a mixture of ethylene glycol and water. The final ratio of ingredients is as follows:

50% active wettable powder	4%
#4 vermiculite	86%
ethylene glycol	5%
water	5%



1-1

75

80

In a modification of the above formulation the ethylene glycol may be replaced by a mixture of the sodium sulfates of mixed long chain alcohol fatty acid esters and diethylene glycol acetate in a ratio between 1:10 and 10:1 by weight.

The granular formulation described above has utility as a pre-emergence treatment for retarding the growth of barnyard grass in an area containing established perennial grasses such as Kentucky bluegrass or creeping red fescue. The material is applied with a granule spreader at the rate of 5 pounds of active ingredient per acre prior to the germination of barnyard grass. Such excellent retardation of barnyard grass is obtained that it presents no competition to the established perennials. Some retardation of the established perennial species without injury is noted.

This formulation is used also for the retardation of barnyard grass in crops belonging to the bean family. A pre-emergence application of 2 to 3 pounds of active ingredient per acre essentially stops the growth of barnyard grass shortly after it emerges from the soil. A planting of beans shows no injurious effects from the plant growth regulant.

EXAMPLE 9

The following compound is substituted for the 1-(3-methylcyclohexyl)-3-phenylurea in Example 8 in like amount by weight and is formulated and applied in like manner. Like results are obtained.

Ex.

9 1 - (1 - methylcyclohexyl) - 3 - phenylurea

EXAMPLES 10—13

The following compounds are substituted one at a time for the 1-(3-methylcyclohexyl)-3-phenylurea in Example 8 in like amount by weight and applied in like manner as a pre-emergence treatment for retarding the growth of crabgrass in an established Kentucky bluegrass or creeping red fescue lawn:

Ex.

10 1 - cyclooctyl - 3 - phenylurea
11 1 - cyclooctyl - 3 - m - tolylurea
12 1 - (3 - methylcyclohexyl) - 3 - m - tolylurea
13 1 - (4 - methylcyclohexyl) - 3 - phenylurea

Such excellent retardation of crabgrass is obtained at application rates of four pounds of active ingredient per acre that the crabgrass presents no competition to the established lawn. Some retardation of the established lawn species without injury is noted.

This formulation is used also for the retardation of crabgrass in crops belonging to the Cruciferae family. A pre-emergence application of 2 to 3 pounds of active ingredient per acre essentially stops the growth of crab-

grass shortly after it emerges from the soil. A planting of mustard shows no injurious effects from the plant growth regulant.

EXAMPLE 14

The following wettable powder is suitable for any of the compounds described above which melt above 90° C.:

1-cyclohexyl-3-phenylurea	50%	70
diatomaceous silica	48%	
coconut acid ester of sodium isethionate	1%	
sodium lauryl sulfate	1%	

The ingredients are blended and micro-pulverized to pass a 60 mesh screen (U.S. Standard sieve size).

This wettable powder formulation in the amount of 8 pounds of active ingredient per acre is added to 80 gallons of water in a spray tank. A surfactant such as trimethyl nonyl ether of polyethylene glycol with ethylene oxide is added to this mixture at the rate of 6 pounds. This tank spray mixture is applied to an area which is infested with Johnson-grass and millet in the 1 to 2-leaf stage. Excellent retardation of these species is obtained. It turns dark green, shows very slow growth, and the root system fails to develop normally.

EXAMPLE 15

The following compound is substituted for the 1-cyclohexyl-3-phenylurea of Example 14 in like amount by weight and is formulated and applied in like manner. Like results are obtained.

Ex.

15 1 - (1 - methylcyclopentyl) - 3 - phenylurea

EXAMPLE 16

The procedure of Example 14 is repeated except that 4 pounds of active ingredient is added to 80 gallons of water in a spray tank. The tank spray mixture is applied to one acre of Bermudagrass lawn which is infested with crabgrass in the 1 to 2-leaf stage. Excellent retardation of crabgrass is obtained. It turns dark green, shows very slow growth, and the root system fails to develop normally. The crabgrass offers poor competition and is crowded out by the Bermudagrass. The Bermudagrass shows a reduced rate of growth without injury.

EXAMPLES 17—18

The following compounds are substituted one at a time for the 1-cyclohexyl-3-phenylurea used in Example 16 in like amount by weight and are formulated and applied in like manner. Like results are obtained.

Ex.

17 1 - m - tolyl - 3 - cycloheptylurea
18 1 - (1 - methylcyclopentyl) - 3 - phenylurea

EXAMPLE 19

A formulation example for 1-(2-methylcyclohexyl)-3-phenylurea follows:

5	1-(2-methylcyclohexyl)-3-phenylurea	25.00%
	hydrated attapulgite	1.75%
	Na ₂ HPO ₄	0.80%
	sodium pentachlorophenate	0.60%
10	sodium lignin sulfonate	15.00%
	water	56.85%

The above composition is mixed with an equal volume of Ottawa sand of 20—30 mesh and sandmilled for 45 minutes to yield a stable aqueous suspension which may be diluted with water for spray application.

This liquid dispersion is applied at the rate of 2 to 4 pounds of active ingredient per acre in 40 gallons of water for the regulation of growth of Johnsongrass in a planting of arbor vitae and privet. Johnsongrass treated in the 1 to 3-leaf stage is retarded markedly. Johnsongrass seeds in the soil germinate but the plants fail to grow.

This formulation has utility for the regulation of growth of Johnsongrass from seed in an established rye grass lawn. A rate of 3 to 4 pounds of active ingredient per acre in 40 gallons of water gives excellent retardation of Johnsongrass after treatment in the 2-leaf stage. Late germinating seedlings are retarded also.

A pre-emergence application of 4 pounds of active ingredient per acre in 40 gallons of water gives excellent growth retardation of barnyard grass in rice. The barnyard grass emerges and shows marked retardation.

The liquid dispersion is also applied at the rate of 2 to 4 pounds of active ingredient per acre in 40 gallons of water for the regulation of crabgrass growth in a planting of arbor-vitae and privet. Crabgrass treated in the 1—3 leaf stage is retarded markedly. Crabgrass seeds in the soil germinate but fail to grow.

This formulation has utility for the regulation of crabgrass growth in an established Kentucky 31 fescue lawn. A rate of 3 to 4 pounds of active ingredient per acre gives excellent retardation of crabgrass when treated in the two-leaf stage.

EXAMPLES 20—42

The following compounds are substituted one at a time for the 1-(2-methylcyclohexyl)-3-phenylurea in Example 19 in like amount by weight and are formulated and applied in like manner. Like results are obtained.

Example

20	1 - (2 - methylcyclohexyl) - 3 - methylurea
21	1 - (2 - chlorocyclohexyl) - 3 - methylurea
22	1 - (2 - chlorocyclohexyl) - 3 - phenylurea

23	1 - (2 - bromocyclohexyl) - 3 - phenylurea	65
24	1 - (2 - chlorocyclooctyl) - 3 - phenylurea	
24	1 - (dibromocyclooctyl) - 3 - phenylurea	
26	1 - (methylcyclooctyl) - 3 - phenylurea	70
27	1 - (2,5 - dimethylcyclooctyl) - 3 - phenylurea	
28	1 - (2 - fluorocyclooctyl) - 3 - phenylurea	75
29	1 - (dichlorocyclooctyl) - 3 - phenylurea	
30	1 - (2 - chlorocycloheptyl) - 3 - phenylurea	
31	1 - (dichlorocycloheptyl) - 3 - phenylurea	80
32	1 - (2 - bromocycloheptyl) - 3 - phenylurea	
33	1 - (2 - methylcycloheptyl) - 3 - phenylurea	85
34	1 - cyclohexyl - 3 - phenylurea	
35	1 - [2(cis),4(trans) - dimethylcyclohexyl] - 3 - phenylurea	
36	1 - [2(trans),4(cis) - dimethylcyclohexyl] - 3 - phenylurea	90
37	1 - cis(2 - methylcyclohexyl) - 3 - phenylurea	
38	1 - trans(2 - methylcyclohexyl) - 3 - phenylurea	
39	1 - cis(3 - methylcyclohexyl) - 3 - phenylurea	95
40	1 - trans(3 - methylcyclohexyl) - 3 - phenylurea	
41	1 - cis(4 - methylcyclohexyl) - 3 - phenylurea	100
42	1 - trans(4 - methylcyclohexyl) - 3 - phenylurea	

EXAMPLE 43

The following wettable powder can be diluted with water and applied as a post-emergence spray or it may be made up as a tank mix in conjunction with 0.2—5 parts of trimethyl nonyl polyethylene glycol ether per part of active ingredient:

1-(3-methylcyclohexyl)-3-phenylurea	84%	110
Montmorillinite clay	13%	
dioctyl sodium sulfosuccinate	2%	
partially desulfonated sodium lignin sulfonate	1%	115

This wettable powder formulation is applied at the rate of 4 pounds of active ingredient in conjunction with 4 pounds of surfactant in 40 gallons of water as a directed post-emergence spray to barnyard grass and crabgrass, independently, in the 1 to 3-leaf stage in a field of cotton when the cotton is 6 inches high. Excellent retardation of growth of the barnyard grass and the crabgrass is obtained. Late germinating seedlings are retarded severely. Upon examination of the weeds, a severe retardation of the root system is noted. The cotton shows normal growth.

EXAMPLE 44

Example 43 is repeated except that 1-cycloheptyl-3-phenylurea is used as the active ingredient and the wettable powder formulation is applied as a directed post-emergence spray to crabgrass in the 1 to 3-leaf stage in a field of cotton when the cotton is 6 inches high. Excellent retardation of growth of the crabgrass is obtained. Late germinating seedlings are retarded severely. Upon examination of the weeds a severe retardation of the root system is noted. The cotton shows normal growth.

EXAMPLE 45

The following emulsifiable oil can be extended with either oil or water:

1 - (2,4 - dimethylcyclohexyl)-3 - phenylurea	20%
a blend of polyoxyethylene ethers and oil soluble sulfonates	5%
alkylated naphthalene (principally alpha-methyl naphthalene)	75%

The above components are blended and agitated until a homogeneous mixture is obtained.

This emulsifiable formulation is extended with 80 gallons of diesel oil and applied at the rate of 12 pounds active ingredient per acre to a brick walk infested with foxtails and barnyard grass. Excellent growth regulation of these weeds is obtained. Weed seeds germinating after treatment show severe retardation.

A pre-emergence application of the above formulation at the rate of 2 to 4 pounds of active ingredient per acre in 40 gallons of water gives excellent retardation of barnyard grass in a newly planted field of soybeans.

EXAMPLE 46

Example 45 is repeated except that 1-cyclooctyl-3-phenylurea is used as the active ingredient and the emulsifiable formulation is applied to a brick walk infested with crabgrass. Excellent growth regulation of crabgrass is obtained. Weed seeds germinating after treatment show severe retardation.

A pre-emergence application of the above formulation at the rate of 2 to 4 pounds of active ingredient per acre in 40 gallons of water gives excellent retardation of crabgrass in a newly planted field of soybeans.

EXAMPLES 47-56

The following compounds are substituted one at a time for the 1 - cyclooctyl-3-phenylurea in Example 46 in like amount by weight and are formulated and applied in like manner. Like results are obtained.

Example

1 - norbornyl - 3 - phenylurea
1 - norbornyl - 3 - m - tolylurea
endo - 1 - (2 - norbornyl) - 3 - phenylurea

exo - 1 - (2 - norbornyl) - 3 - phenylurea	65
1 - (3 - methyl - 2 - norbornyl) - 3 - phenylurea	
1 - (1,7 - dimethyl - 2 - norbornyl) - 3 - phenylurea	
1 - (3,3 - dimethyl - 2 - norbornyl) - 3 - phenylurea	70
1 - (5,5 - dimethyl - 2 - norbornyl) - 3 - phenylurea	
1 - (chloronorbornyl) - 3 - phenylurea	75
1 - (dichloronorbornyl) - 3 - phenylurea	

EXAMPLE 57

The following wettable powder is applicable to the low melting solids described in the Examples as well as all the high melting solid compounds used in this invention.

1-cyclopentyl-3-phenylurea	25.0 %	
alkyl naphthalene sulfonic acid, Na salt	1.0 %	85
low viscosity methyl cellulose	0.25%	
attapulgitic clay	73.75%	

The above components are blended and micropulverized until homogeneous and then reblended.

The formulation described above is applied pre-emergence at the rate of 8 pounds of active ingredient per acre in 100 gallons of water for the control of growth of barnyard grass in areas containing established Kentucky 31 fescue. Excellent retardation of barnyard grass is obtained without noticeable damage to the desirable turf grass.

The formulation described above is applied pre-emergence at the rate of 4 pounds active ingredient per acre in 100 gallons of water for the control of growth of crabgrass in an established bluegrass-fescue lawn. Excellent retardation of crabgrass is obtained without noticeable damage to the desired turf grasses.

EXAMPLE 58

1 - (3 - methylcyclohexyl) - 3 - phenylurea	80%	
calcium lignin sulfonate	2%	
alkyl naphthalene sulfonic acid, Na salt	2%	110
CaSO ₄ · 2H ₂ O	1%	
tricalcium phosphate	7%	
attapulgitic clay	8%	

The ingredients are mixed and micropulverized to a particle size essentially below 50 microns and then reblended.

The above formulation is applied at a rate of 4-8 pounds of active ingredient in 60 gallons of water for the control of growth of seedling Johnsongrass which is 1 to 3 inches tall along a roadside. This application gives outstanding retardation of Johnsongrass. Germinating seedlings are retarded markedly.

The above formulation is applied at a rate of 4 pounds of active ingredient in 60 gallons of water for the control of growth of crabgrass

which are $\frac{1}{2}$ to 1 inch tall along a roadside. This application gives outstanding retardation of crabgrass. Germinating seedlings are retarded markedly.

5 EXAMPLES 59—62

The following compounds are substituted one at a time for the 1-(3-methylcyclohexyl)-3-phenylurea in Example 58 in like amount by weight and are formulated and applied in like manner. Like results are obtained.

10 Example

- 59 1 - (2 - methylcyclopentyl) - 3 - phenylurea
60 1 - (chlorocyclopentyl) - 3 - phenylurea
15 61 1-(dichlorocyclopentyl) - 3 - phenylurea
62 1 - (bromocyclopentyl) - 3 - phenylurea

20 EXAMPLE 63

Granular formulation

The following granular composition is formulated:

- 25 1 - (2 - methylcyclohexyl) - 3 - phenylurea 2%
15—30 mesh attaclay granules 98%

The active material can be applied to the attaclay granules in several ways. (1) The active material may be dissolved in a volatile solvent such as acetone and sprayed on the tumbling granules, or (2) wettable powder containing the active material may be distributed on the surface of the granules. By moistening with water or a mixture of water and ethylene glycol dusting off is prevented. In other granular formulation, if the melting point of the active ingredient is below about 99° C., the active ingredient can be melted and sprayed over a tumbling mass of preferred clay-, vermiculite-, or similar granules.

- 40 This granular formulation is applied pre-emergence with a granule spreader at the rate of 2—5 pounds per acre for the control of growth of foxtail in an area of established bluegrass-meadow fescue. The foxtail emerges from the soil but fails to grow.

- 45 This granular formulation is also applied pre-emergence with a granule spreader at the rate of 2—6 pounds of active ingredient per acre for the control of growth of crabgrass in an established bluegrass-meadow fescue lawn. The crabgrass emerges from the soil but fails to grow. The established lawn grasses show slight retardation with no objectionable injury.

50 EXAMPLES 64—129

The following compounds are substituted one at a time for the 1-(2-methylcyclohexyl)-3-phenylurea in Example 63 in like amount by weight and are formulated and applied in like manner. Like results are obtained.

60 Example

- 64 1 - cyclopentyl - 3 - m - tolylurea

- 65 1 - (3 - methylcyclohexyl) - 3 - m - tolylurea 65
66 1 - (2 - methylcyclohexyl) - 3 - m - tolylurea
67 1 - cycloheptyl - 3 - m - tolylurea
68 1 - (2 - chlorocyclohexyl) - 3 - m - tolylurea 70
69 1 - (2 - bromocyclooctyl) - 3 - phenylurea
70 1 - (2 - iodocyclooctyl) - 3 - phenylurea
71 1 - (2 - bromocyclohexyl) - 3 - phenylurea 75
72 1 - (dichlorocyclohexyl) - 3 - phenylurea
73 1 - (dibromocyclohexyl) - 3 - phenylurea 80
74 1 - (2 - iodocyclohexyl) - 3 - phenylurea
75 1 - (2 - fluorocyclohexyl) - 3 - phenylurea
76 1 - (1 - methylcyclohexyl) - 3 - phenylurea 85
77 1 - (2 - fluorocyclopentyl) - 3 - phenylurea
78 1 - (3 - iodocyclopentyl) - 3 - phenylurea 90
79 1 - (5 - chloro - 2 - norbornyl) - 3 - phenylurea
80 1 - (5,6 - dichloro - 2 - norbornyl) - 3 - phenylurea
81 1 - (bromo - 2 - norbornyl) - 3 - phenylurea 95
82 1 - (iodo - 2 - norbornyl) - 3 - phenylurea
83 1 - (chloro - 2 - methylcyclohexyl) - 3 - phenylurea 100
84 1 - (chloro - 3 - methyl - 2 - norbornyl) - 3 - phenylurea
85 1 - cyclohexyl - 3 - m - tolylurea
86 1 - cycloheptyl - 3 - m - tolylurea
87 1 - cyclopentyl - 3 - m - tolylurea 105
88 1 - (4 - methylcyclohexyl) - 3 - m - tolylurea
89 1 - cyclohexyl - 3 - o - tolylurea
90 1 - (2 - methylcyclohexyl) - 3 - m - tolylurea 110
91 1 - (2 - methylcyclohexylmethyl) - 3 - phenylurea
92 1 - (cyclooctylmethyl) - 3 - phenylurea
93 1 - (chlorocyclopentylmethyl) - 3 - m - tolylurea 115
94 1 - (4 - ethylcyclohexylmethyl) - 3 - p - tolylurea
95 1 - (4 - isopropylcyclohexylmethyl) - 3 - o - tolylurea 120
96 1 - (4 - butylcyclohexylmethyl) - 3 - m - tolylurea
97 1 - (chlorocyclohexylmethyl) - 3 - phenylurea
98 1 - (3 - butylcyclohexyl) - 3 - m - tolylurea 125

- 99 1 - (chlorocyclohexyl) - 3 - p - tolyl-urea
- 100 1 - (3 - methylcyclohexyl) - 3 - o - tolylurea
- 5 101 1 - (2 - methylcyclohexylmethyl) - 3 - o - tolylurea
- 102 1 - (2 - methylcyclohexylmethyl) - 3 - p - tolylurea
- 10 103 1 - (2,4 - dimethylcyclohexylmethyl) - 3 - m - tolylurea
- 104 1 - (2,5 - dimethylcyclohexylmethyl) - 3 - phenylurea
- 105 1 - (3,4 - dimethylcyclohexylmethyl) - 3 - phenylurea
- 15 106 1 - (2 - methyl - 4 - chlorocyclohexyl) - 3 - o - tolylurea
- 107 1 - (3,4 - dichlorocyclohexyl) - 3 - phenylurea
- 20 108 1 - (2 - methylcyclohexyl) - 3 - p - tolylurea
- 109 1 - (2 - methylcyclohexyl) - 3 - o - tolylurea
- 110 1 - (1 - methylcyclohexyl) - 3 - phenylurea
- 25 111 1 - (4 - methylcyclohexylmethyl) - 3 - phenylurea
- 112 1 - (2 - chlorocyclohexylmethyl) - 3 - phenylurea
- 113 1 - (5,6 - dichloronorbornylmethyl) - 3 - phenylurea
- 30 114 1 - (chloronorbornylmethyl) - 3 - phenylurea
- 115 1 - (cyclopentylmethyl) - 3 - phenylurea
- 35 116 1 - (cycloheptylmethyl) - 3 - phenylurea
- 117 1 - (2 - ethylcyclohexyl) - 3 - phenylurea
- 40 118 1 - (4 - isopropylcyclohexyl) - 3 - phenylurea
- 119 1 - (2 - sec.butylcyclohexyl) - 3 - phenylurea
- 120 1 - (2 - chloro - 4 - methylcyclohexyl) - 3 - phenylurea
- 45 121 1 - (3,5 - dimethylcyclohexyl) - 3 - phenylurea
- 122 1 - (2,4 - dimethylcyclohexyl) - 3 - p - tolylurea
- 123 1 - (1 - methylcyclohexyl) - 3 - m - tolylurea
- 50 124 1 - (2 - bromocyclooctylmethyl) - 3 - m - tolylurea
- 125 1 - (2 - fluorocyclopentylmethyl) - 3 - m - tolylurea
- 55 126 1 - (2 - iodocycloheptylmethyl) - 3 - m - tolylurea
- 127 1 - (2 - isopropylcyclohexylmethyl) - 3 - o - tolylurea
- 128 1 - (2,4 - dimethylcyclohexyl) - 3 - o - tolylurea
- 60 129 1 - (2 - bromocyclohexylmethyl) - 3 - p - tolylurea

EXAMPLE 130

Pellet formulation

65 The following pellet formulation is applic-

able to any cycloalkyl phenyl urea used in this invention.

1 - (4 - chlorocyclohexyl) - 3 - phenylurea	11%
anhydrous sodium sulfate	10%
Mississippi sub-bentonite	33%
kaolin	33%
lignin sodium sulfonate	13%

These ingredients are mixed and blended by micropulverizing. Approximately 18% water as calculated on a wet basis is added to the formulation and thoroughly mixed. The resulting product is extruded into pellets through a screw type extruder fitted with a die containing orifices of 3/32 inch. The extruded strands are conveniently dropped into small pellets as they come out of the extruder die. The pellets are air dried. After drying the pellets can be easily handled and applied as such. However, they readily soften and disintegrate when mildly agitated in water and can also be applied in water rather than as the pellets.

EXAMPLES 131-135

The following compounds are substituted one at a time for the 1-(4-chlorocyclohexyl)-3-phenylurea in Example 130 in like amount by weight and are formulated and applied in like manner. Like results are obtained.

Example

131	1 - (2 - chlorocyclohexyl) - 3 - phenylurea	95
132	1 - (2 - bromocyclohexyl) - 3 - phenylurea	
133	1 - (3 - bromocyclohexyl) - 3 - phenylurea	100
134	1 - (dichlorocyclohexyl) - 3 - phenylurea	
135	1 - (2 - iodocyclohexyl) - 3 - phenylurea	105

EXAMPLE 136

A dust formulation is prepared from any cycloalkyl phenylurea of this invention melting above 90° C. as follows:

1 - cyclohexyl - 3 - phenylurea	5%	110
talc	95%	

The ingredients are mixed and micropulverized to a state of subdivision in which most of the individual particles are below 50 microns in size.

EXAMPLE 137

The following wettable powder formulation is prepared:

1 - (2 - methylcyclohexyl) - 3 - phenylurea	52.6%	120
Pikes Peak Clay	44.4%	
Aerosol OT-B Na, dioctyl sulfosuccinate	1.5%	
Na, lignin sulfonate	1.5%	

The above components are blended and micropulverized until homogeneous and then reblended.

The formulation described above is applied pre-emergence at the rate of 2 to 4 pounds of active ingredient per acre in 40 gallons of water for the control of foxtail, millet and barnyard grass in a wide variety of economic crops. Excellent retardation of these weeds is obtained without significant injury to cucumbers, potatoes, tomatoes, squash, eggplant, peppers, red beets, sugar beets, carrots, okra, cotton, flax, lima beans, red clover, lespedza, soybeans, alfalfa, green beans and peanuts.

The formulation described above is applied pre-emergence at the rate of 4 pounds of active ingredient per acre in 100 gallons of water for the control of crabgrass growth in an established bluegrass-fescue lawn. Excellent retardation of crabgrass is obtained, without noticeable damage to the desirable turf grasses.

This formulation is also used pre-emergence at 8 pounds of active ingredient in 80 gallons of water for growth retardation of crabgrass without injury to a new seeding of creeping red fescue, Kentucky bluegrass and bentgrass.

EXAMPLE 138

To 90 ml. of n-hexane, 16.4 grams of trichloroacetic acid is added with stirring. To this mixture is added 21.8 grams of 1-(cyclohexyl)-3-phenylurea. A grayish oil is separated from the hexane. The remaining traces of hexane are removed by placing the oil under high vacuum for two hours. There is obtained 25.3 grams of partially crystallized 1 - (cyclohexyl) - 3 - phenylurea, trichloroacetic acid 1/1 addition compound.

The product of the above process is mixed with a quantity of 15—30 mesh attaclay granules so that the resulting formulation contains 2% active and 98% attaclay.

The active material can be applied to the attaclay granules in several ways. (1) The active material may be dissolved in a volatile solvent such as acetone and sprayed on the tumbling granules, or (2) wettable powder containing the active material may be distributed on the surface of the granules. By moistening with water or a mixture of water and ethylene glycol dusting off is prevented. In other granular formulations, if the melting point of the active ingredient is below about 99°C., the active ingredient can be melted and sprayed over a tumbling mass of preferred clay, vermiculite, or similar granules.

This granular formulation is applied separately as pre-emergence with a granule spreader at the rate of 2—3 pounds of active per acre for the control of growth of barnyard grass and crabgrass. The barnyard grass and the crabgrass emerges from the soil but fails to grow.

manner of Example 138 by substituting the appropriate starting materials, as will be known by an expert in the art, for the 1-(cyclohexyl)-3-phenylurea and trichloroacetic acid.

These compounds are substituted one at a time for the 1-(cyclohexyl)-3-phenylurea, trichloroacetic acid addition compound in Example 138 in like amount by weight and are formulated and applied in like manner. Like results are obtained.

Example

- | | | |
|-----|--|-----|
| 139 | 1 - (2 - methylcyclohexyl) - 3 - phenylurea 1/1 addition compound with trichloroacetic acid | 65 |
| 140 | 1 - (2 - methylcyclohexyl) - 3 - phenylurea 1/1 addition compound with dodecylbenzene sulfonic acid | 70 |
| 141 | 1 - (2 - methylcyclohexyl) - 3 - phenylurea 1/1 addition compound with phosphoric acid | 75 |
| 142 | 1 - (2 - methylcyclohexyl) - 3 - phenylurea 1/1 addition compound with 2,3,6-trichlorobenzoic acid | 80 |
| 143 | 1 - (2 - methylcyclohexyl) - 3 - phenylurea 1/1 addition compound with phenol | 85 |
| 144 | 1 - (2 - methylcyclohexyl) - 3 - phenylurea 1/1 addition compound with 2,4 - dinitro - o - sec. butyl phenol | 90 |
| 145 | 1 - (3 - methylcyclohexyl) - 3 - phenylurea 1/1 addition compound with 2,2-dichloropropionic acid | 95 |
| 146 | 1 - (3 - methylcyclohexyl) - 3 - phenylurea 1/1 addition compound with p-toluene sulfonic acid | 100 |
| 147 | 1 - cyclooctyl - 3 - phenylurea 1/1 addition compound with trichloroacetic acid | 105 |
| 148 | 1 - cyclopentyl - 3 - phenylurea 1/1 addition compound with 2,2,3-trichloropropionic acid | 110 |
| 149 | 1 - norbornyl - 3 - phenylurea 1/1 addition compound with 2,4-dichlorophenoxy acetic acid | 115 |
| 150 | 1 - (4 - methylcyclohexyl) - 3 - m - tolylurea 1/1 addition compound with 2,4,5-trichlorophenoxy acetic acid | 120 |
| 151 | 1 - cycloheptyl - 3 - phenylurea 1/1 addition compound with 2,4,5-trichlorophenoxy propionic acid | 125 |
| 152 | 1 - cyclopentyl - 3 - m - tolylurea 1/1 addition compound with phenol | |
| 153 | 1 - (2 - methylcyclohexyl) - 3 - o - tolylurea 1/1 addition compound with pentachlorophenol | |
| 154 | 1 - (2 - ethylcyclohexyl) - 3 - phenylurea 1/1 addition compound with trichloroacetic acid | |

EXAMPLES 139—158

The following compounds are made in the

- 155 1 - (2 - chlorocyclohexyl) - 3 - phenylurea 1/1 addition compound with trichloroacetic acid
- 5 156 1 - (2 - propylcyclohexyl) - 3 - phenylurea 1/1 addition compound with phenol
- 157 1 - (2,4 - dimethylcyclohexyl) - 3 - phenylurea 1/1 addition compound with dodecylbenzene sulfonic acid
- 10 158 1 - (3,5 - dimethylcyclohexyl) - 3 - phenylurea 1/1 addition compound with dodecylbenzene sulfonic acid

EXAMPLE 159

- 15 1 - (2 - methylcyclohexyl) - 3 - phenylurea 12 pounds
- Kentucky bluegrass seed 174 pounds
- low viscosity polyvinyl alcohol ("Elvanol" S1-05) 0.24 pound
- 20 polyacrylic resin emulsion "Rhoplex" 50% suspension 2.40 pounds

The above ingredients are blended by conventional methods. The active cyclohexyl phenylurea is admixed with the dispersing agent and water and then applied to the seeds. The sticker is then added. The seeds are tumbled while adding the other ingredients to effect good distribution. Warm air is forced through the mixture to remove moisture added to the slurry. There results a dry coated seed.

This composition is evenly distributed over one acre of prepared seed bed utilizing a fertilizer spreader. A lush growth of bluegrass develops having little or no competition from annual grasses.

EXAMPLE 160

- 40 1 - (o - fluorophenyl) - 3 - (2 - methylcyclohexyl)urea 52.6%
- Pikes Peak clay 44.4%
- Sodium dioctyl sulfosuccinate 1.5%
- Sodium lignin sulfonate 1.5%

The above components are blended and micropulverized until homogeneous and then rebleded.

The formula described above is applied pre-emergence at the rate of 4 pounds of active ingredient per acre in 100 gallons of water for the control of crabgrass growth in an established bluegrass-fescue lawn. Excellent retardation of crabgrass is obtained, without noticeable damage to the desirable turf grasses.

This formulation is also used pre-emergence at 8 pounds of active ingredient in 80 gallons of water for growth retardation of crabgrass without injury to a new seeding of creeping red fescue, Kentucky bluegrass and bentgrass.

EXAMPLES 161-170

- 60 The following compounds are substituted one at a time for the 1 - (o - fluorophenyl) - 3 - (2 - methylcyclohexyl)urea in Example 160

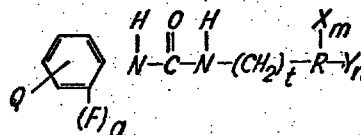
in like amount by weight and are formulated and applied in like manner. Like results are obtained.

Example

- 161 1 - (o - fluorophenyl) - 3 - cyclopentylurea
- 162 1 - (o - fluorophenyl) - 3 - (2 - methylcyclopentyl)urea 70
- 163 1 - (o - fluorophenyl) - 3 - (2 - methylcyclopentylmethyl)urea
- 164 1 - (o - fluorophenyl) - 3 - (2,3 - dimethylcyclopentylmethyl)urea
- 165 1 - (o - fluorophenyl) - 3 - cyclohexylurea 75
- 166 1 - (o - fluorophenyl) - 3 - (2 - methylcyclohexylmethyl)urea
- 167 1 - (o - fluorophenyl) - 3 - (3 - methylcyclohexyl)urea 80
- 168 1 - (o - fluorophenyl) - 3 - (3 - methylcyclohexylmethyl)urea
- 169 1 - (o - fluorophenyl) - 3 - (4 - methylcyclohexyl)urea
- 170 1 - (o - fluorophenyl) - 3 - (2 - chlorocyclohexyl)urea 85

WHAT WE CLAIM IS:—

1. A plant growth regulant composition which comprises as active agent one or more cycloalkyl phenylureas represented by the formula:



wherein R is a saturated cycloalkyl group containing from 5 to 8 carbon atoms or saturated bi-cycloalkyl group of 7 to 8 carbon atoms; X and Y are the same or different and are each a halogen atom or a methyl or ethyl group; t is 0 or 1; m is 0 or 1; n is 0 or 1; a is 0 or 1; Q is a hydrogen atom or a methyl group and with the limitation that when R is cyclopentyl, cycloheptyl, cyclooctyl or bicycloalkyl, Q if methyl must be in the meta position, and X and Y are the same or different and are each a halogen atom or a methyl group, or comprises as active agent an acid addition compound of a cycloalkyl phenylurea of the above formula, the composition containing a surface-active agent, or an inert solid diluent, or both.

2. A plant growth regulant composition as claimed in claim 1, containing by weight up to 5 parts of surface-active agent per part of active agent.

3. A plant growth regulant composition as claimed in claim 1 or claim 2, wherein the inert solid diluent is present in amount from 10% to 99% by weight, based on the weight of the composition.

4. A composition as claimed in claim 3,

- 155 1 - (2 - chlorocyclohexyl) - 3 - phenylurea 1/1 addition compound with trichloroacetic acid
- 5 156 1 - (2 - propylcyclohexyl) - 3 - phenylurea 1/1 addition compound with phenol
- 157 1 - (2,4 - dimethylcyclohexyl) - 3 - phenylurea 1/1 addition compound with dodecylbenzene sulfonic acid
- 10 158 1 - (3,5 - dimethylcyclohexyl) - 3 - phenylurea 1/1 addition compound with dodecylbenzene sulfonic acid

EXAMPLE 159

- 15 1 - (2 - methylcyclohexyl) - 3 - phenylurea 12 pounds
- Kentucky bluegrass seed 174 pounds
- low viscosity polyvinyl alcohol ("Elvanol" S1-05) 0.24 pound
- 20 polyacrylic resin emulsion "Rhoplex" 50% suspension 2.40 pounds

The above ingredients are blended by conventional methods. The active cyclohexyl phenylurea is admixed with the dispersing agent and water and then applied to the seeds. The sticker is then added. The seeds are tumbled while adding the other ingredients to effect good distribution. Warm air is forced through the mixture to remove moisture added to the slurry. There results a dry coated seed.

This composition is evenly distributed over one acre of prepared seed bed utilizing a fertilizer spreader. A lush growth of bluegrass develops having little or no competition from annual grasses.

EXAMPLE 160

- 1 - (o - fluorophenyl) - 3 - (2 - methylcyclohexyl)urea 52.6%
- 40 Pikes Peak clay 44.4%
- Sodium dioctyl sulfosuccinate 1.5%
- Sodium lignin sulfonate 1.5%

The above components are blended and micropulverized until homogeneous and then rebled.

The formula described above is applied pre-emergence at the rate of 4 pounds of active ingredient per acre in 100 gallons of water for the control of crabgrass growth in an established bluegrass-fescue lawn. Excellent retardation of crabgrass is obtained, without noticeable damage to the desirable turf grasses.

This formulation is also used pre-emergence at 8 pounds of active ingredient in 80 gallons of water for growth retardation of crabgrass without injury to a new seeding of creeping red fescue, Kentucky bluegrass and bentgrass.

EXAMPLES 161-170

- 60 The following compounds are substituted one at a time for the 1 - (o - fluorophenyl) - 3 - (2 - methylcyclohexyl)urea in Example 160

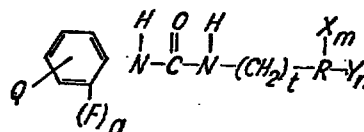
in like amount by weight and are formulated and applied in like manner. Like results are obtained.

Example

- 161 1 - (o - fluorophenyl) - 3 - cyclopentylurea
- 162 1 - (o - fluorophenyl) - 3 - (2 - methylcyclopentyl)urea 70
- 163 1 - (o - fluorophenyl) - 3 - (2 - methylcyclopentylmethyl)urea
- 164 1 - (o - fluorophenyl) - 3 - (2,3 - dimethylcyclopentylmethyl)urea
- 165 1 - (o - fluorophenyl) - 3 - cyclohexylurea 75
- 166 1 - (o - fluorophenyl) - 3 - (2 - methylcyclohexylmethyl)urea
- 167 1 - (o - fluorophenyl) - 3 - (3 - methylcyclohexyl)urea 80
- 168 1 - (o - fluorophenyl) - 3 - (3 - methylcyclohexylmethyl)urea
- 169 1 - (o - fluorophenyl) - 3 - (4 - methylcyclohexyl)urea
- 170 1 - (o - fluorophenyl) - 3 - (2 - chlorocyclohexyl)urea 85

WHAT WE CLAIM IS:—

1. A plant growth regulant composition which comprises as active agent one or more cycloalkyl phenylureas represented by the formula: 90



wherein R is a saturated cycloalkyl group containing from 5 to 8 carbon atoms or saturated bi-cycloalkyl group of 7 to 8 carbon atoms; 95

X and Y are the same or different and are each a halogen atom or a methyl or ethyl group; t is 0 or 1; m is 0 or 1; n is 0 or 1; a is 0 or 1; Q is a hydrogen atom or a methyl group and with the limitation that when R is cyclopentyl, cycloheptyl, cyclooctyl or bi-cycloalkyl, Q if methyl must be in the meta position, and X and Y are the same or different and are each a halogen atom or a methyl group, or comprises as active agent an acid addition compound of a cycloalkyl phenylurea of the above formula, the composition containing a surface-active agent, or an inert solid diluent, or both. 100

2. A plant growth regulant composition as claimed in claim 1, containing by weight up to 5 parts of surface-active agent per part of active agent. 105

3. A plant growth regulant composition as claimed in claim 1 or claim 2, wherein the inert solid diluent is present in amount from 10% to 99% by weight, based on the weight of the composition. 110

4. A composition as claimed in claim 3, 115



in which the solid diluent is a finely divided powder, the composition containing a surface-active agent so as to be a wettable powder.

5 5. A plant growth regulant composition as claimed in claim 3, in the form of granules or pellets.

10 6. A plant growth regulant composition as claimed in claim 5, wherein the inert solid diluent is present in amount from 65% to 99% by weight, based on the weight of the composition.

15 7. A plant growth regulant composition as claimed in claim 1 or claim 2, containing a liquid diluent.

15 8. A plant growth regulant composition as claimed in claim 7 in which the liquid diluent is a solvent for the active agent and containing a surface-active agent so that the composition is an emulsifiable solution.

20 9. A plant growth regulant composition as claimed in claim 8, wherein the active agent is present in amount from 0.5% to 35% by weight of the weight of the composition.

25 10. A plant growth regulant composition as claimed in any preceding claim, wherein the active agent is 1-(methylcyclohexyl)-3-phenylurea.

30 11. A plant growth regulant composition as claimed in any of claims 1-9, wherein the active agent is 1-(methylcyclohexyl)-3-tolylurea.

35 12. A plant growth regulant composition as claimed in any of claims 1-9, wherein the active agent is 1-(2-methylcyclohexyl)-3-phenylurea.

40 13. A plant growth regulant composition as claimed in any of claims 1-9, wherein the active agent is 1-(3-methylcyclohexyl)-3-phenylurea.

40 14. Perennial grass seeds coated with at least one of the active agents specified in claim 1 and an adhesive therefor.

45 15. Perennial grass seeds as claimed in claim 14, wherein the adhesive is present in amount of 0.25% to 50% by weight of the active agent.

50 16. A method of retarding the growth of seedling weed grasses which comprises applying to the locus of said grasses a growth retarding amount of a composition having as active agent at least one of the active agents specified in claim 1.

55 17. A method as claimed in claim 16, wherein the composition contains 1-(methylcyclohexyl)-3-phenylurea.

18. A method as claimed in claim 16, wherein the composition contains 1-(methylcyclohexyl)-3-tolylurea.

60 19. A method as claimed in claim 16, wherein the composition contains 1-(2-methyl-

cyclohexyl)-3-phenylurea.

20. A method according to claim 16, wherein the composition contains 1-(3-methylcyclohexyl)-3-phenylurea.

21. A method as claimed in any of claims 16 to 20, in which a solution of the active agent is applied to the locus of the grasses. 65

22. A method according to any of claims 16 to 21 wherein the composition is applied at a rate of $\frac{1}{2}$ to 20 pounds per acre. 70

23. A method as claimed in claim 16 in which the composition is as claimed in any one of claims 1 to 13.

24. A method of retarding the growth of seedling grasses as claimed in claim 16 and substantially as hereinbefore described. 75

25. A method of retarding the growth of seedling grasses substantially as described in the foregoing Examples.

26. A method of retarding the growth of crabgrass which comprises applying to the locus of said crabgrass a growth retarding amount of a composition having as active agent at least one of the active agents specified in claim 1. 80

27. A method as claimed in claim 26, in which the composition is as claimed in any one of claims 1-13. 85

28. A method for the selective control of crabgrass growing in the presence of perennial grasses which comprises applying to the locus of said crabgrass a growth retarding amount of a composition having as active agent at least one of the active agents specified in claim 1. 90

29. A method as claimed in claim 28 wherein the composition contains 1-(methylcyclohexyl)-3-phenylurea. 95

30. A method as claimed in claim 28 wherein the composition contains 1-(cycloheptyl)-3-phenylurea. 100

31. A method as claimed in claim 28 wherein the composition contains 1-(cyclooctyl)-3-phenylurea.

32. A method as claimed in claim 28 wherein the composition contains 1-(2-methylcyclohexyl)-3-phenylurea. 105

33. A method as claimed in claim 28 wherein the composition is as claimed in any one of claims 1 to 13. 110

34. A method as claimed in any one of claims 28 to 32 wherein the composition is applied at a rate of $\frac{1}{2}$ to 20 pounds per acre.

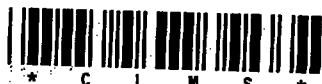
35. A method for the selective control of crabgrass as claimed in claim 28 and substantially as hereinbefore described. 115

36. A method for the selective control of crabgrass substantially as described in any of the foregoing Examples.



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Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press
(Leamington) Ltd.—1966. Published by The Patent Office, 25 Southampton Buildings,
London, W.C.2, from which copies may be obtained.



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