968,662

PATENT SPECIFICATION

NO DRAWINGS

968,662

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Date of Application and filing Complete Specification: Sept. 21, 1960.
No. 32469/60.
(Patent of Addition to No. 968,661 dated Aug. 15, 1960).
Complete Specification Published: Sept. 2, 1964.
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Index at acceptance:—A5 E(1C2, 1C4A2, 1C4A3, 1C4B2, 1C4B3, 1C4B4, 1C12, 1C14B, 1C14C, 1C15)

International Classification:—A 01 n

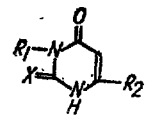
COMPLETE SPECIFICATION

Herbicidal Compositions containing 3-Substituted Uracils

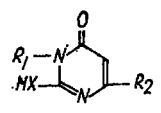
We, E.I. DU PONT DE NEMOURS AND COMPANY, a corporation organised and existing under the laws of the State of Delaware, United States of America, of Wilmington 98, State of Delaware, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the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

This invention relates to the use of 3-substituted uracils and is an improvement in or modification of the invention described in our Application No. 28,141/60 (Serial No. 968,661).

Our Application No. 28,141/60 discloses that certain 3-substituted uracils of defined formula have herbicidal activity. The present invention is concerned with a particular group of 3-substituted uracils within the broad disclosure of Application No. 28,141/60 (Serial No. 968,661), namely compounds of the general formula:

(I) 

or the corresponding isomeric formula:

(II) 

wherein R_1 represents an alkyl group containing from 3 to 6 carbon atoms; R_2 represents an alkyl group containing from 1 to 5 carbon atoms; and X represents oxygen or sulphur and alkali metal and alkaline earth metal salts thereof.

It will be noted that the definition above refers to the isomeric enol form of the uracil, as well as the usual keto form. It is thought that there is a tautomeric equilibrium between the two forms. However, for the sake of convenience the keto form is generally referred to herein.

Of the 3-substituted uracils defined above, 3-butyl-6-methyl uracil and its sodium salt are preferred.

The 3-substituted uracils can be formulated as herbicidal compositions comprising at least one 3-substituted uracil which is a compound of general formula I or II as defined above or an alkali metal or alkaline earth metal salt thereof, associated with a compatible diluent or surface active agent, or both. The term "herbicidal composition" is used herein to include both compositions which are ready for use as herbicides and concentrates which require dilution before use as herbicides.

The diluent may be a liquid other than an oil, especially water, or an oil, or a solid carrier. Two or more different types of diluent may be present, as for example in compositions obtained by dispersing a wettable powder in a liquid diluent. Liquid compositions may be in the form of solutions or dispersions. It is often advantageous to use the alkali metal and alkaline earth metal salts because they are usually soluble in water and can be formulated as aqueous solution.

Examples of oil carriers are aliphatic and aromatic hydrocarbons, for example, hydrocarbons of petroleum origin. Dispersions in oils may be prepared by milling the active material, usually with dispersing agents and suspending agents, with the oil in mills such as pebble mills. [The term "oil" is used to denote a liquid which is immiscible with water

[*Price 4s. 6d.*]

and soluble in ether and has a boiling point of at least 125°C.]. The amount of the herbicide in the dispersion may be from 10% or less to 48 and 50% of the oil dispersion. Preferably, the particles in such dispersions range from 1 to 50 microns, although the majority are preferably in the range of from 5 to 20 microns in average diameters. In general, the oils used include aliphatic and aromatic hydrocarbons and mixtures thereof, particularly those derived from petroleum and having a boiling point in the range from 125 to 400°C. A hydrocarbon oil having a boiling point lower than 125°C. should generally not be used because when sprayed from a nozzle, such a hydrocarbon tends to volatilize undesirably. Furthermore, low-boiling hydrocarbons present a serious fire hazard.

Compositions containing the uracils in association with a solid diluent may be in the form of powdered solids, granules or pellets. Powdered or dust compositions may be prepared by mixing the active material with finely divided inert solid carriers, preferably natural clays, diatomaceous earth, synthetic fine silica and calcium silicate, either in powder or granular form.

Granule or pellet compositions may be prepared by mixing the active material in finely divided form with a clay (with or without a water-soluble binder), moistening the mixture with 15—20% water, extruding the mass under pressure through an orifice, and either cutting the extruded material to size before drying to yield pellets, or first drying and then granulating to yield granules. Alternatively granules, but usually not pellets, may be made by spraying active material in solution or suspension upon the surface of a preformed granule of clay, vermiculite, or other suitable granular material. When the active material is soluble in the spray medium so that it can penetrate into the pores of the granular carrier, no binding agent is needed. When it is insoluble and in suspension form, a binder is needed to cause the active material to adhere to the surface. The binder may be soluble such as goulac or dextrin, or colloidally soluble such as swollen starch, glue or polyvinyl alcohol. The final step in either case is to remove the liquid medium, whether solvent or carrier.

The composition of the invention will usually contain a surface-active agent, in the case of concentrates the amount suitably being sufficient to render the concentrate, e.g. wettable powder, readily dispersible in water or in oil.

The term "surface-active agent" is used herein to include wetting agents, dispersing agents, suspending agents and emulsifying agents. Suitable surface-active agents are listed in, for example, United States Patent Specifications, 2,426,417; 2,655,447, and 2,412,510 and British Specification No. 492,742, "Soap and Chemical Specialties", *31*, No. 7, pages 50—61, No. 8, pages 48—61, No. 9, pages 52—67, and No. 10, pages 38—67 (1955), McCutcheon in "Soap and Chemical Specialties", December, 1957; January, February, March, 1958 and Bulletin E—607 of the Bureau of Entomology and Plant Quarantine of the United States Department of Agriculture. In general, less than 10% by weight of surface-active agent need be present in herbicidal compositions according to the invention, and the amount of surface-active agent may be 1% by weight or even less.

The herbicidal compositions of this invention may contain two or more of the substituted uracils. They may also be formulated to contain other herbicides in addition to the substituted uracils, e.g. other 3-substituted uracils as described in our Applications Nos. 28,141/60, 17,417/64, 17,418/64, 17,419/64 and 17,420/64 (Serial Nos. 968,661, 968,663, 968,664, 968,665 and 968,666), and known herbicides, for example 2,4-dimethyl-phenoxy-acetic acid, 2-methyl-4-chlorophenoxy acetic acid or another herbicide of the phenoxy-acetic acid type.

Optionally the compositions may contain conventional additives e.g. adhesives such as gelatin, and blood albumin; resins, for example rosin and alkyd resins, may also be used in certain compositions to increase retention or tenacity of deposits following application.

In a further aspect the invention provides a method of controlling the growth of weeds at a locus which comprises applying to the locus a herbicidally effective quantity of at least one 3-substituted uracil which is a compound of general formula I or II as hereinbefore defined or an alkali metal or alkaline earth metal salt thereof. The amount of active compound used will depend on the weeds to be controlled, the formulation used, the mode of application, the climate, season of the year, rainfall and other variables, and because of this it is not possible to state generally a particular rate of application which will be suitable for all situations.

The 3-substituted uracils have herbicidal activity. Some of them are especially useful in controlling the growth of weeds on uncultivated areas. Thus in suitable circumstances some of the uracils can be used in soil-foliage applications, a suitable application rate in most circumstances being from 20 to 80 lbs. per acre. In suitable cases the 3-substituted uracils can be used to obtain selective control of weeds in the presence of crops. For such use pre-emergence application is usually preferred, in which case rates as low at 0.5 pounds per acre can be effective though up to 4 or 5 pounds per acre may be needed in some instances. The uracils can also be used in some cases for post-emergence application.

A particularly valuable method for the pre-

paration of the sodium and potassium salts is the novel process which is described in detail and claimed in our Application No. 28,141/60 (Serial No. 968,661), which comprises (A) heating a urea of the formula:

$$R_1.NH.C.NH_2 \quad (\text{with } C=O)$$

with a β-keto ester of the formula:

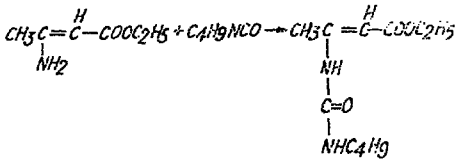
$$R^1O.C.CH_2.C.R_2 \quad (\text{with } C=O \text{ and } C=O)$$

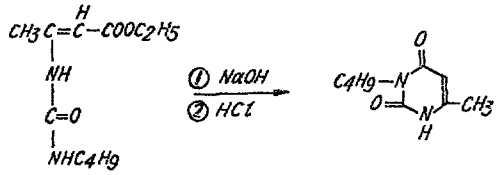
(wherein R^1 represents an alkyl group of 1 to 6 carbon atoms and R_1 and R_2 are as hereinbefore defined) in the presence of an acidic compound and under substantially anhydrous reaction conditions, to produce a 3-(3-substituted ureido) - 2,3 - unsaturated ester of the formula:

$$R_1.NH.C.NH.C=CH-C.OR^1 \quad (\text{with } C=O,\ R_2,\ C=O)$$

and (B) heating the said ester under substantially anhydrous conditions in the presence of a strong base of the appropriate metal. The corresponding uracil can be produced from the metal salt by treatment with an acid.

The 3-substituted uracils can also be prepared by the adaptation of methods known for the preparation of related compounds. For example a convenient method for the preparation of these compounds is illustrated by the following equations. (For further reference, note the publication by Behrend and Meyer in Ann. *314*, 219 (1901) and Ber. *33*, 622 (1900)):—





Generally, esters of β-amino-α,β-unsaturated acids (the starting reagents in equation 2) can be prepared by reacting the corresponding β-keto-esters with aqueous ammonia [Conrad and Epstein, Ber. *20*, 3054 (1887)]. The properly substituted β-amino-α,β-unsaturated esters are reacted with an alkyl isocyanate or ~~isithiocyanate~~ in an inert solvent such as toluene or xylene heated for a short interval of time at reflux temperature. The reaction mixture is chilled, filtered, and the filtrate distilled to remove the xylene. Generally, a viscous liquid residue remains [crude β-(3-substituted ureido) - α,β - unsaturated ester] which can be reacted without further purification with an aqueous alcoholic alkaline solution at reflux temperature to bring about the desired uracil ring closure. At this point the reaction mixture is made acidic with a strong acid such as hydrochloric acid and distilled to remove the alcohol. Upon chilling the remaining aqueous residue, the corresponding uracil separates out of solution as an essentially pure solid.

It is possible that the β-(3-substituted ureido)-α,β-unsaturated esters referred to above as intermediates may exist in either or both of the two tautomeric forms illustrated by formula (I)

$$(I)\quad C_4H_9NHCNHC=CH-COOC_2H_5 \rightleftarrows C_4H_9NHCN=C-CH_2COOC_2H_5 \quad (\text{with } O,\ CH_3,\ O,\ CH_3)$$

The alkali metal and alkaline earth metal salts can be prepared by conventional methods such as dissolving or suspending the uracil in an aqueous or nonaqueous solution of at least an equimolar amount of a base or basic salt containing the desired cation. Heat and agitation may be required for the conversion. For example, a sodium salt can be prepared by dissolving the uracil in water containing an equimolar amount of sodium hydroxide. The salt can then be isolated from the solution by removal of the water.

The invention is illustrated by the following Examples, which show typical compounds, their method of preparation, formulation and of application. The mesh sizes given are United States Standard Sieve sizes.

EXAMPLE 1

3-butyl-6-methyluracil

A solution composed of 100 pts. by weight of xylene and 19.8 pts. by weight of butyl isocyanate was added to a solution of 23 pts. by weight of methyl 3-aminocrotonate contained in 100 pts. by weight of xylene. The resulting mixture was stirred at reflux for one hour, chilled, filtered, and the filtrate distilled to remove the xylene. The remaining viscous liquid residue was diluted with 260 pts. by weight of 95% ethyl alcohol and 320 pts.

by weight of 8% aqueous sodium hydroxide solution. This mixture was stirred and refluxed for a period of 10 minutes, then cooled to about 50°C. and charged with sufficient concentrated hydrochloric acid to make the reaction mixture slightly acidic. After removing the alcohol from the reaction mixture by distillation, chilling the remaining aqueous filtrate caused solid 3-butyl-6-methyluracil to separate out of solution. This product, after air drying, may be used for formulation without further purification. A portion recrystallized from ethanol melted at 182—183°C.

The following compounds of this invention can be prepared similarly by substituting equivalent amounts of the amines and equivalent amounts of the isocyanates set forth in Table I for methyl 3-aminocrotonate and butyl isocyanate in Example 1 above.

TABLE I

Ex. No.	Amine	Parts by Wt.	Isocyanate	Parts by Wt.	Uracil Product
2	methyl 3-amino-crotonate	23	hexyl isocyanate	25.8	3-hexyl-6-methyluracil
3	methyl 3-aminocrotonate	23	propyl isocyanate	17	3-propyl-6-methyluracil
4	methyl 3-amino-3-ethyl-acrylate	25.8	butyl isocyanate	19.8	3-butyl-6-ethyluracil
5	methyl 3-amino-3-butyl-acrylate	31.4	amyl isocyanate	22.6	3-amyl-6-butyluracil

EXAMPLE 6

Preparation of 3-isopropyl-6-methyluracil

A mixture of 51 parts of isopropylurea, 65 parts of ethyl acetoacetate, and 2.5 parts of polyphosphoric acid is heated at reflux for 10 hours during which time 8 parts of water is collected in a trap. The solution is decanted and evaporated to an oil which is taken up in 250 parts of ethanol containing 26.5 parts of sodium methoxide. This mixture is heated at reflux for 10 minutes, then evaporated to an oil again and taken up in 500 parts of water. The water solution is washed with ether, then acidified with concentrated hydrochloric acid, whereupon 3-isopropyl-6-methyluracil separates as a white crystalline solid. The melting point of this solid is 186—187.5°C.

The following compounds of this invention are prepared similarly by substituting equivalent amounts of the β-keto esters and equivalent amounts of the ureas set forth in Table II for ethyl acetoacetate and butyl isocyanate in Example 6 above.

TABLE II

Ex. No.	β-Keto Ester	Parts by Wt.	Urea	Parts by Wt.	Uracil Product
7	ethyl acetoacetate	65	butylthiourea	66	3-butyl-6-methyl-2-thiouracil
8	ethyl acetoacetate	65	isopropylthiourea	59	3-isopropyl-6-methyl-2-thiouracil
9	ethyl 3-ketovalerate	72	amylurea	65	3-amyl-6-ethyluracil
10	ethyl 3-ketohexanoate	79	isoamylthiourea	73	3-isoamyl-6-propyl-2-thiouracil
11	ethyl acetoacetate	65	isobutylurea	58	3-isobutyl-6-methyluracil
12	ethyl acetoacetate	65	allylurea	50	3-allyl-6-methyluracil
13	ethyl 3-ketoheptanoate	86	allylthiourea	58	3-allyl-6-butyl-2-thiouracil
14	ethyl acetoacetate	65	propynylurea	49	3-propynyl-6-methyluracil
15	ethyl acetoacetate	65	hexylurea	72	3-hexyl-6-methyluracil

EXAMPLE 16

Preparation of 3-butyl-6-methyluracil, sodium salt

A solution of 4 parts of sodium hydroxide in 100 parts of water is treated with 18.2 parts of 3-butyl-6-methyluracil. Stirring and warming is employed to effect solution. The water is removed from the solution under reduced pressure, leaving 3-butyl-6-methyluracil, sodium salt as a white solid.

The alkali metal and alkaline earth metal salts of the compounds exemplified by Examples 1 to 15 can be prepared similarly by substituting equivalent amounts of substituted uracils for the 3-butyl-6-methyluracil of Example 16 above. The following list contains examples of typical salts of the invention:

17 3-isopropyl-6-methyluracil, sodium salt
18 3-butyl-6-methyl-2-thiouracil, potassium salt
19 3-amyl-6-propyluracil, barium salt
20 3-butyl-6-ethyluracil, sodium salt
21 3-isopropyl-6-propyl-2-thiouracil, sodium salt
22 3-propyl-6-methyluracil, calcium salt
23 3-isobutyl-6-methyluracil, magnesium salt

EXAMPLE 24

Wettable Powder Formulation

A wettable powder is prepared by blending the components in a ribbon blender, micropulverizing in a hammer mill until the particles are substantially all below 50 microns in size, and then reblending until the powder is homogeneous. A formulation to be prepared in this manner is as follows:

3-butyl-6-methyluracil	80.00%
attapulgite clay	18.25%
alkyl naphthalene sulphonic acid, Na salt	1.50%
low viscosity methylcellulose	0.25%

EXAMPLE 25

Herbicidal Pellets

3-hexyl-6-methyluracil	25%
alkyl naphthalene sulphonic acid, Na salt	1%
anhydrous sodium sulphate	10%
non-swelling clay	64%

The above composition is blended and micropulverized, then mixed with 15—20% water and extruded under pressure through an orifice to produce rods which are cut into pellets and dried.

EXAMPLE 26

Oil Suspension

An oil suspension is prepared by grinding the following ingredients together in a ball or roller mill until the solids are finely dispersed in the oil and the average particle size is less than 5 microns.

3-butyl-6-methyl-2-thiouracil	25%
Diesel oil	67%
alkyl aryl polyether alcohol	8%

EXAMPLE 27
Water Suspension

A water suspension is prepared by blending the dry ingredients in a ribbon blender and then grinding with water in a ball or roller mill until the solids are finely dispersed in the water and the average particle size is less than 5 microns.

3-isopropyl-6-methyl-2-thiouracil	25%
hydrated attapulgite	2%
lignin sulphonic acids	5%
water	68%

EXAMPLE 28
Wettable Powder Formulation

A wettable powder is prepared by blending the following components in a ribbon blender, followed by micropulverizing in a hammer mill until the particles are substantially all below 50 microns in size, and then reblending the powder until it is homogeneous.

3-amyl-6-ethyluracil	25%
Kaolin clay	73%
Dioctyl ester of sodium sulphosuccinic acid	1%
sodium lignin sulphonate	1%

EXAMPLE 29
Wettable Powder Formulation

A wettable powder is prepared by blending the following components in a ribbon blender, followed by micropulverizing in a hammer mill. The ground product is air reductionized until the particles are substantially all below 10 microns in size, and then it is reblended until homogeneous.

3-isobutyl-6-methyluracil	50.00%
synthetic precipitated hydrated silicon dioxide	48.10%
low viscosity methylcellulose	0.40%
alkyl naphthalene sulphonic acid, Na salt	1.50%

EXAMPLE 30
Herbicidal Granules

Herbicidal granules illustrating preparation and application of compositions of this invention are prepared as follows:

3,6-dibutyluracil	20%
attapulgite clay	78%
alkyl naphthalene sulphonic acid, Na salt	1%
lignin sulphonic acid, Na salt	1%

The ingredients are mixed in a ribbon blender until homogeneous and then charged to a pug mill where sufficient water is blended in to form a thick paste. The paste is discharged from the pug mill in the form of extrusions which are dried and broken by means of a rotary crusher into irregular granules roughly 1/4" to 1/8" in diameter.

EXAMPLE 31
Herbicidal Granules

The following procedure is employed to prepare a herbicidal granule:

3-allyl-6-methyluracil	12%
sand (20—30 mesh)	81%
sodium silicate (28% SiO_2; ratio $SiO_2/Na_2O = 3.25$)	7%

The granules are prepared by mixing the sand and sodium silicate solution (water glass) in a ribbon blender until the surface of the sand is completely wet and then dusting into the agitated mixture in the blender the powdered active ingredient. When the 3-allyl-6-methyluracil has adhered completely to the sand, the granules are discharged from the blender and allowed to dry.

EXAMPLE 32
Herbicidal Granules

3-butyl-6-methyluracil, Na salt	2%
10—30 mesh vermiculite	98%

To prepare these granules, the active material is dissolved in water and sprayed upon the vermiculite while it is being tumbled. The product is then dried.

EXAMPLE 33
Oil Suspension

An oil suspension is prepared by grinding the ingredients together in a ball or roller mill until the solids are finely dispersed in the oil and the average particle size of the active ingredient is less than 5 microns.

3-hexyl-6-methyluracil	25%
Diesel oil	67%
polyoxy ethylene sorbitan ester of mixed rosin and fatty acids	8%

This oil suspension is diluted with water to form a water emulsion.

EXAMPLE 34
Herbicidal Pellets

Herbicidal pellets are prepared as follows:

3-allyl-6-butyl-2-thiouracil	190 parts
water	280 parts
Hide glue (20% aqueous)	65 parts
glass frits (20—30 mesh)	750 parts

The active ingredient is first made into an aqueous suspension by ball milling with the water and hide glue. The hide glue is a typical adhesion material derived from animal by-products. The aqueous suspension is then sprayed onto the glass frits with continuous agitation in a ribbon blender and when complete coverage is obtained, removed and dried in a tunnel drier.

EXAMPLE 35
Fast Release Pellets

3-propyl-6-ethyluracil	25%
sodium sulphate anhydrous	10%
Sodium lignin sulphonate	10%
Ca, Mg bentonite	55%

The components are first blended together, micropulverized, moistened with 16—18% water, and extruded as 1/8" diameter cylinders. The cylinders are cut as they extrude to give 1/8 × 1/8" pellets which are then dried.

EXAMPLE 36
Herbicidal Pellets

Herbicidal pellets with a moderately rapid rate of release may be prepared as follows:

3-isoamyl-6-methyluracil	12%
polyvinyl alcohol (low viscosity)	2%
water	16%
pilled sodium nitrate (2—4 mesh)	70%

The active ingredient is first ball milled with the polyvinyl alcohol and water to form a thin paste, which is then added slowly to a ribbon blender agitating the pilled sodium nitrate. Steam heat can be applied to a jacket on the ribbon blender to hasten the drying. Blending and drying are continued until the coating is firmly adherent.

EXAMPLE 37
Wettable Powder

A wettable powder is made by blending the following ingredients in a ribbon blender:

3-propyl-6-amyluracil	50%
Diatomite	46%
polymerized sodium salts of alkyl naphthalene sulphonic acid	2%
glyceryl monostearate	2%

After blending, the composition is micropulverized in a hammer mill until the particles are substantially all less than 50 microns in size. The product is finally reblended in a ribbon blender until homogeneous.

EXAMPLE 38
Wettable Powder

The following composition is employed to prepare a wettable powder of compounds of the invention:

3-isoamyl-6-butyluracil	60%
synthetic calcium silicate	38%
polyvinyl alcohol (low viscosity)	1%
alkyl naphthalene sulfonic acid, Na salt	1%

The components are blended in a ribbon blender, micropulverized in a hammer mill until the particles are substantially all below 50 microns in diameter and then reblended until homogeneous in a ribbon blender.

EXAMPLE 39
Wettable Powder

A wettable powder is prepared by blending the following ingredients in a ribbon blender:

3-propyl-6-butyluracil	70.00%
attapulgite clay	28.25%
alkyl naphthalene sulphonic acid, Na salt	1.50%
low viscosity methylcellulose	0.25%

The blended mixture is micropulverized in a hammer mill until the particles are substantially all less than 50 microns in size, and then remixed in a ribbon blender until homogeneous.

EXAMPLE 40
Herbicidal Wettable Powder

The following ingredients are processed to prepare a formulation illustrating this invention:

3-hexyl-6-butyluracil	50%
Kaolin clay	48%
Dioctyl ester of sodium sulphosuccinic acid	1%
sodium lignin sulphonate	1%

The ingredients are mixed together in a ribbon blender and micropulverized in a hammer mill until the particles are substantially all less than 50 microns in size. The ground product is then remixed in a ribbon blender until homogeneous.

EXAMPLE 41
Wettable Powder Formulation

A wettable powder is prepared by blending the following ingredients in a ribbon blender:

3-isopropyl-6-methyluracil	60.00%
attapulgite clay	38.25%
alkyl naphthalene sulphonic acid, Na salt	1.50%
low viscosity methylcellulose	0.25%

The blended mixture is ground in a hammer mill until substantially all the particles have a diameter of less than 50 microns. The ground product is then reblended until homogeneous.

EXAMPLE 42
Wettable Powder Formulation

A wettable powder formulation is prepared by blending the following components in a ribbon blender:

3-butyl-6-ethyluracil	50%
Kaolin clay	48%

dioctyl ester of sodium sulpho-succinic acid	1%
sodium lignin sulphonate	1%

The blended mixture is ground in a hammer mill until the particles are substantially all less than 50 microns in size. The mixture is then reblended until homogeneous.

EXAMPLE 43
Water Suspension

A water suspension is prepared by blending the following dry ingredients in a ribbon blender and then grinding with water in a ball or roller mill:

3-butyi-6-propyl-2-thiouracil	25%
hydrated attapulgite	2%
lignin sulphonic acid	5%
water	68%

Grinding is continued until substantially all the particles in the suspension have been reduced to diameters of less than 5 microns.

EXAMPLE 44
Wettable Powder

This wettable powder is prepared in the manner described for Example 24, using the following ingredients:

3-butyl-6-ethyluracil, Ca salt	80.0%
sodium diamyl sulphosuccinate	0.4%
ditertiary acetylenic glycol	0.4%
oleyl ester of sodium isothionate	1.0%
Kaolin clay	18.2%

EXAMPLE 45
Herbicidal Granules

3-isoamyl-6-propyl-2-thiouracil, Na salt	10%
granular 15—30 mesh attapulgite	90%

To prepare these granules, the active material is dissolved in water and sprayed on the attapulgite while it is being tumbled. The product is then dried.

EXAMPLE 46
Herbicidal Granules

3-amyl-6-methyluracil	25%
6—15 mesh expanded vermiculite	75%

The above granular formulation is prepared by dissolving the active ingredient in water and spraying it on the vermiculite. The granular product is then dried.

EXAMPLE 47
Herbicidal Pellets

3-amyl-6-butyluracil	40%
alkyl naphthalene sulphonate, Na salt	1%
anhydrous sodium sulphate	5%
calcium, Mg bentonite	54%

The above composition is dry blended, micropulverized, moistened, and extruded. The extrusions are cut into pellets and dried.

EXAMPLE 48
Wettable Powder

The following ingredients are prepared as a wettable powder in the manner described for Example 24:

3-propyl-6-methyluracil, Mg salt	80.00%
alkyl naphthalene sulphonate, Na salt	1.75%
sodium lignin sulphonate	1.00%
Kaolin	17.25%

EXAMPLE 49
Herbicidal Solution

The following solution is prepared by simple mixing of the components:

3-butyl-6-methyluracil, Na salt	25%
sodium dodecyl benzene sulfonate	2%
water	73%

EXAMPLE 50

Two pounds of 3-butyl-6-methyluracil per acre are applied as a pre-emergence spray to corn and weed seeds planted in loam soil. Mustard, amaranthus, millet, ryegrass, and seedling Bermuda grass are killed. Corn planted 1½ inches deep is not injured and grows normally.

EXAMPLE 51

A pre-emergence spray treatment of soybeans planted 1 inch deep by 1.8 pounds of 3-butyl-6-methyluracil per acre gives 96% control of mixed annual grasses and broadleaves with no injury to the crop.

EXAMPLE 52

An over-all spray on a stand of established quackgrace using 20 pounds of 3-butyl-6-methyluracil per acre gives excellent control of quackgrass.

EXAMPLE 53

The wettable powder compositions of Examples 24, 28, 29, 37, 38, 39, 41, 42, 44 and 48 are used for pre-emergence application in agricultural crops such as soybeans. These formulations are dispersed in 80 gallons of water and sprayed from a tractor-mounted pressure sprayer. When applied at a concentration of 1—4 pounds of active ingredient per acre (as formulated), excellent control of germinating broadleaf and grass weeds is obtained, with no observable injury to the crops. Such compositions control pigweed, Lamb's quarter,

purslane, mustard, crabgrass, Foxtail, and miscellaneous other weeds.

EXAMPLE 54

The granular compositions of Examples 30, 31 and 45 are applied by hand or by a commercial spreader to control weeds around industrial sites, lumber yards, boundary fences, railroad rights-of-way, billboards, parking areas, and roadsides. They are also used for spot treatments of soil for control of woody plants, such as turkey oak, winged elm, and mesquite. When applied at a concentration of 20—80 pounds of active ingredient per acre, control of germinating annual weeds, established perennial weeds such as quackgrass and Johnson grass, and woody plants is obtained.

EXAMPLE 55

The emulsifiable oils of Examples 26 and 33 are useful for weed control in railroad yards and sidings. When these compositions are diluted with water and sprayed from a railroad spray car at a concentration of about 35 pounds of active ingredient per acre, vegetation such as quackgrass, crabgrass, Johnson grass, Bermuda grass, brome grass, ragweed, cockleburr, Lamb's quarter, and mare's-tail is controlled for an extended period.

EXAMPLE 56

The pellet compositions of Examples 25, 35, 36, and 47 are useful for weed control along highway guard rails, bridge abutments, safety fences, and highway signs. They are conveniently applied by hand at rates of about 30 to 55 pounds of active ingredient per acre.

EXAMPLE 57

The granular composition of Example 32 is useful for pre-emergence weed control in crops. This composition is applied with a tractor-mounted granule distributor at a concentration of 2.5 pounds of active ingredient per acre. Excellent control of crabgrass, giant Foxtail, pigweed, Lamb's quarter, and ragweed is obtained without noticeable injury to the crop.

EXAMPLE 58

The aqueous suspension compositions of Examples 27 and 43 are useful as pre-emergence weed controls in soybeans and sorghum. When applied at a concentration of 2.5 pounds of active ingredient per acre, excellent control of crabgrass, Foxtail, flower-of-an-hour, and velvet leaf is obtained, with no observable injury to the crops.

EXAMPLE 59

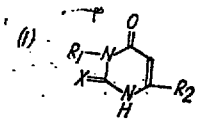
The solution of Example 49 is useful for pre-emergence weed control in crops and for weed control on industrial sites.

When applied to a field of cotton at a concentration of 2.0 pounds of active ingredient per acre, good control of annual broadleaf and grass weeds is obtained, without injury to the crop.

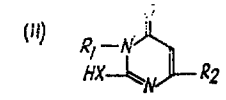
At rates of 20 to 35 pounds of active ingredient per acre, excellent control of goldenrod, ox-eye daisy, quackgrass, broomsedge, and orchardgrass is obtained along fence rows and around oil tanks at refineries.

WHAT WE CLAIM IS:—

1. A herbicidal composition comprising at least one 3-substituted uracil which is a compound of general formula:—



or the corresponding isomeric formula:—



wherein R_1 represents an alkyl radical containing 3 to 6 carbon atoms; R_2 represents an alkyl radical containing 1 to 5 carbon atoms; and X represents oxygen or sulphur; or an alkali metal or alkaline earth metal salt thereof, associated with a diluent which is at least in part an oil.

2. A herbicidal composition comprising a 3-substituted uracil which is a compound of general formula I or II as defined in Claim 1 or an alkali metal or alkaline earth metal salt thereof associated with a diluent which is at least in part a solid.

3. A herbicidal composition according to Claim 1 which contains a surface active agent.

4. A herbicidal composition according to Claim 2 which contains a surface active agent.

5. A herbicidal composition comprising a 3-substituted uracil which is a compound of general formula I or II as defined in Claim 1 or an alkali metal or alkaline earth metal salt thereof associated with a diluent which is a liquid other than an oil and a surface active agent.

6. A herbicidal composition according to Claim 5 wherein the liquid is water.

7. A herbicidal composition comprising a 3-substituted uracil which is a compound of general formula I or II as defined in Claim 1 or an alkali metal or alkaline earth metal salt thereof associated with a surface active agent.

8. A herbicidal composition according to

purslane, mustard, [illegible]ass, Foxtail, and miscellaneous other weeds.

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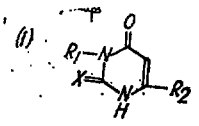
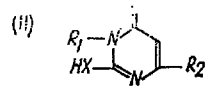


or the corresponding isomeric formula:—



wherein R_1 represents an alkyl radical containing 3 to 6 carbon atoms; R_2 represents an alkyl radical containing 1 to 5 carbon atoms; and X represents oxygen or sulphur; or an alkali metal or alkaline earth metal salt thereof, associated with a diluent which is at least in part an oil.

2. A herbicidal composition comprising a 3-substituted uracil which is a compound of general formula I or II as defined in Claim 1 or an alkali metal or alkaline earth metal salt thereof associated with a diluent which is at least in part a solid.

3. A herbicidal composition according to Claim 1 which contains a surface active agent.

4. A herbicidal composition according to Claim 2 which contains a surface active agent.

5. A herbicidal composition comprising a 3-substituted uracil which is a compound of general formula I or II as defined in Claim 1 or an alkali metal or alkaline earth metal salt thereof associated with a diluent which is a liquid other than an oil and a surface active agent.

6. A herbicidal composition according to Claim 5 wherein the liquid is water.

7. A herbicidal composition comprising a 3-substituted uracil which is a compound of general formula I or II as defined in Claim 1 or an alkali metal or alkaline earth metal salt thereof associated with a surface active agent.

8. A herbicidal composition according to

any one of Claims 3 to 7 which contains 1 to 10% by weight of the surface active agent.

9. A herbicidal composition according to any one of the preceding claims which contains, in addition to the 3-substituted uracil of formula I or II another compound having herbicidal properties.

10. A herbicidal composition according to Claim 9 wherein the other compound is one of the known herbicides specifically hereinbefore named.

11. A herbicidal composition according to any one of the preceding claims in the form of a concentrate which requires dilution before use as a herbicide.

12. A herbicidal composition according to any one of the preceding claims wherein X represents an oxygen atom.

13. A herbicidal composition according to any one of Claims 1 to 11 wherein the 3-substituted uracil is 3-butyl-6-methyl uracil or the sodium salt thereof.

14. A herbicidal composition according to any one of Claims 1 to 11 wherein the 3-substituted uracil is one of the compounds specifically hereinbefore named.

15. A herbicidal composition according to claim 1, 2 or 5 substantially as hereinbefore described.

16. A herbicidal composition according to Claim 1, 2 or 5 substantially as described in any one of the preceding Examples 24 to 49.

17. A method of controlling the growth of weeds at a locus which comprises applying to the locus a herbicidally effective quantity of at least one 3-substituted uracil which is a compound of general formula I or II as defined in Claim 1 or an alkali metal or alkaline earth metal salt thereof.

18. A method according to Claim 17 wherein the 3-substituted uracil is 3-butyl-6-methyl uracil or the sodium salt thereof.

19. A method according to claim 17 wherein the 3-substituted uracil is applied in the form of a herbicidal composition as claimed in any of Claims 1 to 16, where appropriate after dilution.

20. A method of controlling the growth of weeds according to Claim 17 wherein the growth of the weeds specified in any one of Examples 50 to 54 is controlled by the application of the 3-substituted uracil specified in that Example.

21. A method according to Claim 20 wherein the 3-substituted uracil is applied at the rate specified in the Example.

22. A method of controlling the growth of weeds according to Claim 17 substantially as hereinbefore described.

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Leamington Spa: Printed for Her Majesty's Stationery Office, by the Courier Press (Leamington) Ltd.—1964. Published by The Patent Office, 25 Southampton Buildings, London, W.C.2, from which copies may be obtained.