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3,235,357

METHOD FOR THE CONTROL OF UNDESIRABLE VEGETATION

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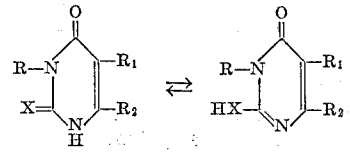
19 Claims. (Cl. 71—2.5)

This application is a continuation-in-part of copending application Serial Nos. 84,980 and 159,746 filed January 26, 1961, and December 15, 1961, respectively, which in turn are continuations-in-part of application Serial Nos. 48,837, filed August 11, 1960; 12,959, filed March 7, 1960; and 833,704, filed August 14, 1959, all now abandoned.

This invention relates to the use of 3,5,6-substituted uracils as herbicides, and to certain of these uracils and their derivatives as new compounds.

More particularly, this invention is directed to compositions and methods employing, as an active herbicidal ingredient, at least one compound of the formula

(1)



where:

R is alkyl of 1 through 10 carbon atoms, substituted alkyl of 1 through 8 carbon atoms, aryl of 5 through 10 carbon atoms, substituted phenyl, aralkyl of 5 through 13 carbon atoms, substituted aralkyl of 5 through 13 carbon atoms, alkenyl of 3 through 8 carbon atoms, alkynyl of 3 through 8 carbon atoms, cycloalkyl of 3 through 12 carbon atoms, cycloalkenyl of 4 through 12 carbon atoms, cycloalkyl alkyl of 4 through 13 carbon atoms, cycloalkenyl alkyl of 5 through 13 carbon atoms, (substituted cycloalkyl)alkyl of 5 through 14 carbon atoms, (substituted cycloalkenyl)alkyl of 5 through 14 carbon atoms, and cyano;

R_1 is chlorine, fluorine, bromine, iodine, methyl, ethyl, propyl, butyl, methoxy, ethoxy, propoxy, butoxy, nitro, alkoxymethyl of 2 through 6 carbon atoms, hydroxy alkyl of 1 through 6 carbon atoms, alkenyl of 3 through 6 carbon atoms, thiocyanato, cyano, thiolmethyl, alkylthio containing 1 through 4 carbon atoms, bromomethyl, methylthiomethyl, fluoromethyl, chloromethyl, phenylthiomethyl or carboxymethylthiomethyl;

R_2 is chloro, bromo, alkyl of 1 through 5 carbon atoms, chloroalkyl of 1 through 4 carbon atoms, bromoalkyl of 1 through 4 carbon atoms, or alkoxy of 1 through 5 carbon atoms; and

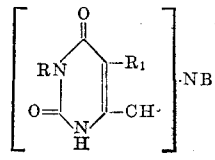
X is oxygen or sulfur.

The salts of these compounds can also be used according to this invention. By "salts" is meant those compounds formed with such cations as sodium, potassium, lithium, calcium, magnesium, barium, strontium, iron, manganese and quaternary ammonium.

Some of the uracils of Formula 1 also form novel 1:1 addition compounds with nitrogenous bases. The exact structure of these compounds is not known. Although the compounds are, generally speaking, poorly soluble

in water, they are, according to the best available information, believed to be essentially salt-like in structure. They will be symbolized by the following formula, with the understanding that it is representative only, and is not intended to illustrate actual structure:

(2)



where:

R and R_1 are defined as in Formula 1, and

NB is a nitrogenous base having an ionization constant K_b of $\geq 10^{-9}$ in water.

Suitable nitrogenous bases are substituted, unsubstituted, cyclic and acyclic

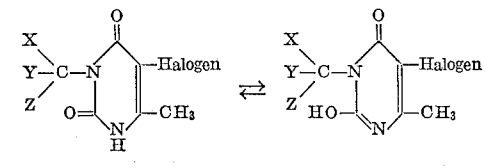
Amines
Amidines, and
Guanidines.

The amines can be primary, secondary or tertiary amines, polyamines, arylamines, or heterocyclicamines. Illustrative of such amines are:

Sec-butylamine
2-amino-2-methyl-1,3-propanediol
Trimethylenediamine
Ethanolamine
Dodecylamine
Ethylenediamine
Hexamethylenediamine
Cocoadiamine
Tallowdiamine
Hexamethyleneimine
Cyclohexylamine
Methoxypropylamine
Methylamine
Dimethylamine
Trimethylamine
Ammonia
Ethylamine
Propylamine
Butylamine
Octylamine
Pyridine
Piperidine
Tetramethylguanidine
Acetamidine
Benzylamine
Diethylenediamine
2-aminobutanol-1
2-aminooctanol-1

Within the scope of Formula 1 is a group of novel compounds. These compounds are those of the formula

(3)

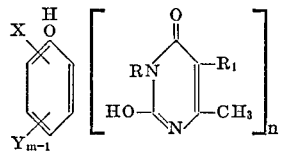


where:

X is methyl or ethyl,
Y is hydrogen or methyl, and
Z is an alkyl group of 1 through 6 carbon atoms, and the salts of these compounds, as defined for Formula 1.

Certain 3,5,6-substituted uracils of Formula 1 also form water stable, novel complexes with phenol and substituted phenols. These complexes have the formula

(4)



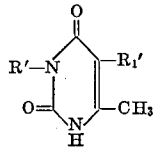
where:

R and R_1 are as defined in Formula 1,
X is hydrogen, chlorine, nitro, alkyl of 1 through 3 carbon atoms, bromine or $—OR_3$,
R_3 is alkyl of 1 through 3 carbon atoms,
Y is chlorine or alkyl of 1 through 3 carbon atoms,
m is a number 1 through 5, and
n is 1 or 2.

These complexes are also herbicidal, and in this respect, have some advantages over the uracils per se, viz., higher solubility in oils and solvents. They are formulated into herbicidal compositions in the same way as are the uracils themselves.

Preferred for use according to this invention because they are effective as herbicides at lower rates of application are compounds of the formula

(5)



where:

R′ is alkyl of 2 through 8 carbon atoms, substituted alkyl of 2 through 8 carbon atoms, phenyl, substituted phenyl, aralkyl of 5 through 10 carbon atoms, substituted aralkyl of 5 through 10 carbon atoms, cycloalkyl of 3 through 12 carbon atoms, cycloalkenyl of 5 through 12 carbon atoms, cycloalkyl alkyl of 4 through 13 carbon atoms, and (substituted cycloalkyl) alkyl of 5 through 14 carbon atoms; and
R_1' is chlorine, bromine, iodine, methyl, hydroxymethyl, methoxymethyl and nitro.

Especially preferred for use because of their herbicidal effectiveness are:

3-(1-lower alkylethyl)-5-halogeno-6-methyluracils,
3-(2-lower alkylethyl)-5-halogeno-6-methyluracils,
3-(1-lower alkylisopropyl)-5-halogeno-6-methyluracils,
3-(cycloalkyl)-5-halogeno-6-methyluracils,
3-(cycloalkenyl)-5-halogeno-6-methyluracils,
3-(phenyl)-5-halogeno-6-methyluracils,
3-(bicycloalkyl)-5-halogeno-6-methyluracils,
3-(bicycloalkenyl)-5-halogeno-6-methyluracils,
3-(tricycloalkyl)-5-halogeno-6-methyluracils, and
3-(tricycloalkenyl)-5-halogeno-6-methyluracils.

In the foregoing list, "lower alkyl" means an alkyl radical containing 1 through 4 carbon atoms.

In Formulae 1, 2, 4 and 5 the term "substituted alkyl" is intended to include such radicals as

Bromoalkyl of 1 through 8 carbon atoms,
Chloroalkyl of 1 through 8 carbon atoms,
Hydroxyalkyl of 1 through 8 carbon atoms,
Alkoxyalkyl of 2 through 8 carbon atoms,
Alkoxy carbonyl alkyl of 3 through 8 carbon atoms, and
Cyanoalkyl of 2 through 8 carbon atoms.

Similarly, the terms "aryl" and "substituted phenyl" embrace radicals such as

Phenyl,
Naphthyl,
O-biphenyl,
Pyridyl,
Chlorophenyl,
Bromophenyl,
Alkoxyphenyl,
Dibromophenyl,
Fluorophenyl,
Trichlorophenyl,
Alkylphenyl of 7 through 11 carbon atoms,
Dialkylphenyl of 8 through 12 carbon atoms,
Chloroalkylphenyl of 7 through 10 carbon atoms,
Nitrochlorophenyl,
Nitrophenyl,
Dichloronitrophenyl,
Chloroalkoxyphenyl of 7 through 11 carbon atoms,
Trifluoromethylphenyl,
Tetrahydronaphthyl, and
Indenyl.

The terms "aralkyl" and "substituted aralkyl" are intended to include such radicals as

Furfuryl,
Benzyl,
Phenylalkyl of 8 through 11 carbon atoms (total),
Chlorobenzyl,
Dichlorobenzyl,
Alkylbenzyl of 8 through 11 carbon atoms (total),
Dialkylbenzyl of 9 through 13 carbon atoms (total),
Nitrobenzyl,
Alkoxybenzyl of 8 through 11 carbon atoms (total), and
Naphthylmethyl.

The terms "cycloalkyl," "cycloalkenyl," "cycloalkyl alkyl," and "cycloalkenyl alkyl" will include

Cyclohexyl,
Cyclohexenyl,
Cyclohexylalkyl,
Cyclohexenylalkyl,
Cyclopentyl,
Cyclopentenyl,
Cyclopentylalkyl,
Cyclopentenylalkyl,
Norbornyl,
Norbornenyl,
Norbornylalkyl,
Norbornenylalkyl,
Bicyclo(2,2,2)octyl,
Bicyclo(2,2,2)octenyl,
Bicyclo(2,2,2)octylalkyl,
Bicyclo(2,2,2)octenylalkyl,
Cyclopropyl,
Cyclobutyl,
Cyclobutylalkyl,
Cyclobutenyl,
Cyclobutenylalkyl,
Hexahydroindanyl,
Tetrahydroindanyl,
Hexahydroindenyl,
Hexahydroindenyl alkyl,
Tetrahydroindanyl alkyl,
Hexahydroindanyl alkyl,
Hexahydro-4,7-methanoindenyl,
Tetrahydro-4,7-methanoindanyl,
Hexahydro-4,7-methanoindanyl,
Hexahydro-4,7-methanoindenyl alkyl,
Tetrahydro-4,7-methanoindanyl alkyl,
Hexohydro-4,7-methanoindanyl alkyl,
Decahydronaphthyl,
Decahydronaphthyl alkyl,
Tetrahydronaphthyl,

Tetrahydronaphthyl alkyl,
Decahydro-1,4-methanonaphthyl,
Decahydro-1,4-methanonaphthyl alkyl,
Octahydro-1,4-methanonaphthyl,
Octahydro-1,4-methanonaphthyl alkyl,
Decahydro-1,4-5,8-dimethanonaphthyl,
Decahydro-1,4-5,8-dimethanonaphthyl alkyl,
Octahydro-1,4-5,8-dimethanonaphthyl, and
Octahydro-1,4-5,8-dimethanonaphthyl alkyl.

These cyclic substituents can be further substituted with alkyl groups 1 through 4 carbon atoms, methoxy, chlorine and bromine.

In the process of combining the various 3,5,6-substituents described above for R, R_1, and R_2, it is surprising to find that strikingly similar herbicidal activity can be obtained with seemingly unrelated substituents. On the other hand, it is equally surprising to find useful selective phytotoxicity (applicable in certain crop areas) when a relatively minor structural modification for R (for example a normal alkyl chain altered to a branched chain) is made. Each 5-substituent (described for R_1 above) for example, has a maximum and minimum range of herbicidal effectiveness and/or specie selectivity dependent on the nature of the substituent in the 3- or 6-position. There is little continuity of herbicidal effect which can be ascribed to a particular substituent independently of the other two substituents in the 3,5,6-positions.

UTILITY

These uracils represent a new class of herbicides offering farmers and property owners a new and effective method for the control of undesirable vegetation. These compounds are unique in that they exert their action against both broadleaf and grass weeds, are effective against hard-to-kill nutsedge and perennial grasses such as quack grass, Johnson grass, and Bermuda grass, and are effective on highly adsorptive substrates such as railroad ballast, heavy clay soil, and soils high in organic matter.

This combination of properties makes these compounds useful wherever general weed control is required, such as industrial areas, railroad rights-of-way, and areas adjacent to croplands in agricultural areas.

Certain of the uracils also exhibit selective herbicidal action in crops. By properly selecting a uracil of the invention and a rate and time of application, annual grass and broadleaf seedlings in such crops as asparagus, corn, flax, sugar cane, pineapple, safflower, peanuts, citrus, alfalfa, strawberries, gladiolus, stone fruits and cucurbits can be controlled.

By proper selection of rate and time of application, certain of the uracils can also be used to control weeds growing in dormant crops.

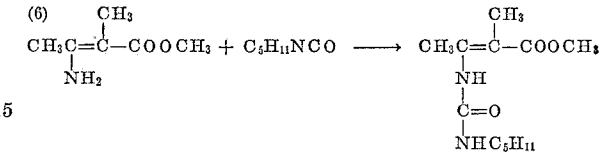
This selective activity and activity on weeds growing in dormant crops is described in more detail in the examples.

The precise amounts of uracils to be used in any given situation will, of course, vary according to the particular end result desired, the use involved, the plant and soil involved, the formulation used, the mode of application, prevailing weather conditions, foliage density and like factors. Since so many variables play a role, it is not possible to indicate a rate of application suitable for all situations. Broadly speaking, the compounds are used at levels of about ¼ pound per acre to about 25 pounds per acre. For selective weed control in crops, rates of ¼ to 8 pounds per acre will generally be used. More of the active material can be used to control difficult-to-kill species growing under adverse conditions. Economic factors, such as inaccessibility of the area to be treated, e.g., fire breaks in forests, may also favor higher rates, with less frequent treatments.

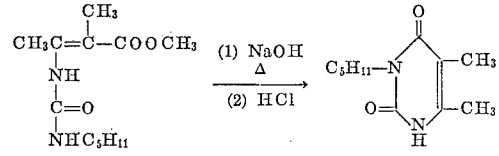
PREPARATION OF THE COMPOUNDS

Those uracils of Formula 1 where R_1 is alkyl, or alkenyl, as well as the uracil starting reactants for the reactions of Equations 11 to 15, can be prepared by methods heretofore described in the literature. For example, one method for the preparation of these compounds is illustrated by the following equations:

(6)



(7)



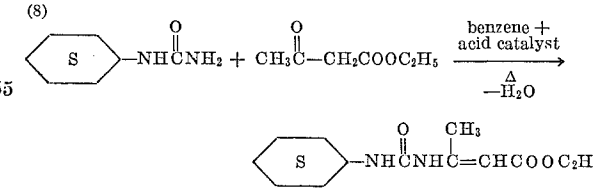
For more general details, note the publication by Behrend and Myer in Ann., 314, 219 (1901), and also Ber., 33, 622 (1900).

In the method of Equations 6 and 7, the esters of β-amino-α,β-unsaturated acids are first prepared by reacting the corresponding β-keto esters with aqueous ammonia [Conrad and Epstein, Ber., 20, 3054 (1887)]. These properly substituted β-amino-α,β-unsaturated esters are then reacted with an isocyanate or isothiocyanate in an inert solvent such as toluene or xylene, and heated for a short interval of time at reflux temperature.

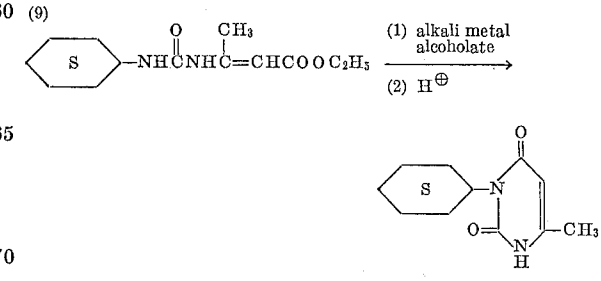
The reaction mixture is chilled, filtered, and the filtrate distilled to remove the solvent. Generally, a viscous liquid residue remains which is crude 3-(3-substituted-ureido)-α,β-unsaturated ester. This can be reacted without further purification with an aqueous alcoholic alkaline solution at reflux temperature to bring about the desired uracil ring closure. At this point, the reaction is made slightly acidic with a strong acid such as hydrochloric acid and distilled to remove the alcohol. After the remaining aqueous solution has been chilled, the corresponding substituted uracil separates as an essentially pure solid.

Another method for preparing the uracil starting reactants is illustrated by the following equations:

(8)



(9)

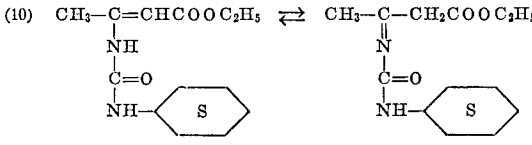


According to Equations 8 and 9, an appropriately substituted urea or thiourea is reacted with a β-keto ester or

an α-substituted β-keto ester substituted with such radicals as alkoxy, fluorine, alkyl, or alkenyl, and an acid catalyst, at reflux in a solvent from which water is removed continuously. After the water has all been removed, the solution is stripped and taken up in ethanol containing a base such as sodium methoxide. After a few minutes reflux, the solvent is removed, and the residual oil taken up in water and acidified, whereupon the desired product separates in crystalline form.

The product formed at the end of the first step, i.e., after the water has been removed, is a ureido compound of the type referred to in Equation 8. It can be isolated and purified if desired; however, this is not necessary or advantageous.

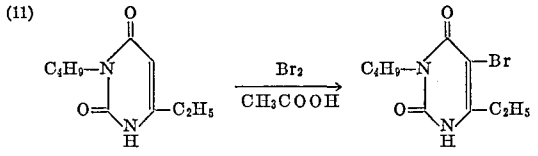
These ureido compounds referred to above are believed to exist in either or both of two tautomeric forms, as illustrated in the following equation:

(10) 

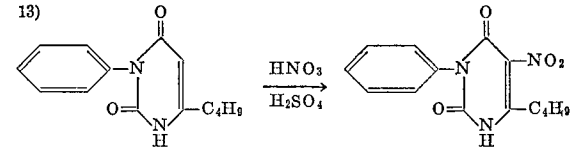
3-substituted-6-methyluracil starting reactants can also be formed by the interaction of alkylurethans and N-substituted acetoacetamides in the presence of alkoxide.

The uracils which are substituted in the 5-position with halogen, nitro, thiocyanato, chloromethyl and hydroxymethyl groups can be prepared by methods heretofore described in the literature for related compounds.

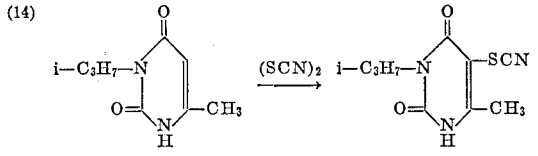
For example, the preparation of those compounds having a halogen substituent in the 5-position is illustrated by Equation 11. For more general details, see J. Am. Chem. Soc. 61, 1015 (1939); Ann., 305, 314; Ann., 352, 242; and Ann., 441, 192.

(11) 

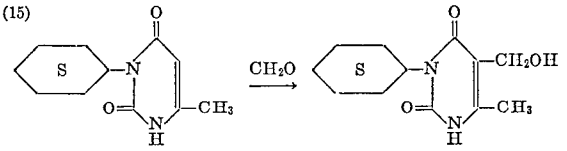
The 5-nitro uracils are prepared by direct nitration of uracils having no substituent in the 5-position, as illustrated in Equation 13. For a description of this method, see J. Am. Chem. Soc., 30, 1156 (1908).

13) 

The 5-thiocyano uracils are prepared by direct thiocyanogenation as illustrated by Equation 14. For more details concerning this procedure, see J. Am. Chem. Soc., 63, 2323 (1941); Org. Syntheses, Coll. Vol. II, 574 (1943); and Helv. Chim. Acta, 19, 1411 (1936).

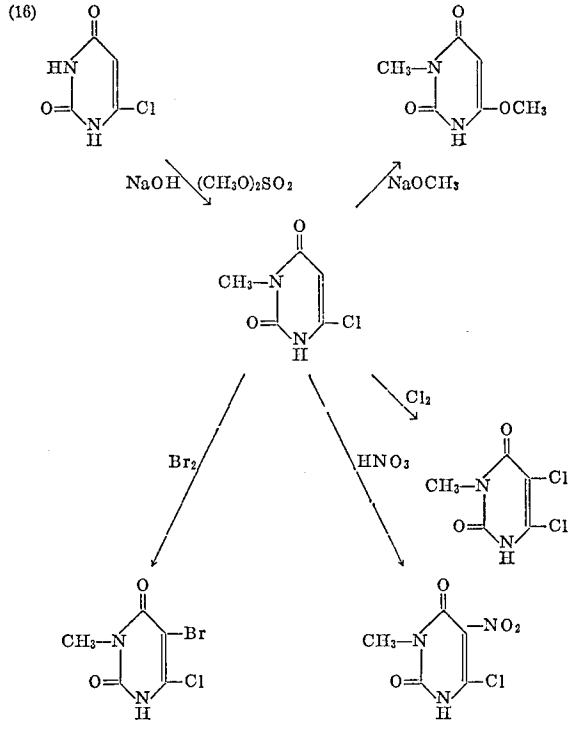
(14) 

The reaction of formaldehyde with uracils not substituted in the 5-position gives uracils substituted in the 5-position with a hydroxymethyl group, as illustrated by Equation 15. For greater detail see Gazz. Chim. Ital., 79, 447 (1949).

(15) 

These 5-hydroxymethyl uracils can be easily reacted with alcohols, methylmercaptan, thiophenols, thionylchloride, or mercaptoacetic acid to give corresponding 5-alkoxymethyl, -methylthiomethyl, -phenylthiomethyl, -chloromethyl, and carboxymethylthiomethyl uracils.

6-chlorouracil, prepared by acidic hydrolysis of 2,4-dimethoxy-6-chloropyrimidine (see British Patent 677,-342), can serve as an intermediate for the preparation of many uracils. The following diagram illustrates this:

(16) 

Methods for carrying out the reactions illustrated in the foregoing diagram are well described in pyrimidine literature. These reactions, as well as many related ones, will be easily carried out by those skilled in this field of chemistry.

The salts of the compounds of Formula 1 are prepared by conventional methods such as dissolving the free uracil in an aqueous or nonaqueous solution of at least an equimolar amount of a base or basic salt containing the desired cation. 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 uracil salts which are not soluble in water can be prepared by treating an aqueous solution of an alkali metal salt of the uracil with an aqueous solution of a water-soluble salt of the metal.

The quaternary ammonium salts of the compounds of Formula 1 are prepared by reacting the substituted uracil with an appropriate quaternary ammonium hydroxide. Since these hydroxides are generally available in solution,

the reaction is most conveniently carried out in the same solvent. If the solvent-free salt is desired, it can be easily prepared by removing the solvent.

Alternatively, the quaternary ammonium salts of the uracils can be prepared in a dry inert solvent such as toluene or xylene. The appropriate quaternary ammonium halide is then added with stirring and, if necessary, mild heating. The sodium halide which forms is removed by filtration, leaving the quaternary ammonium salt of the uracil in solution. If desired, the solvent-free salt can be prepared by removing the solvent, preferably in vacuo.

The nitrogenous base-addition compounds of Formula 2 are prepared by mixing together equimolar quantities of an appropriate uracil and a nitrogenous base. The mixture is gradually heated, with stirring, until a clear melt is formed. On cooling, the addition compound crystallizes. This product can then be recrystallized from a solvent such as benzene, cyclohexane, nitromethane or acetonitrile.

It is sometimes advantageous to use an inert solvent medium to carry out the reaction. Such a solvent moderates the reaction by acting as a heat sink, and allows better control of the reaction, especially if it is being carried out on a large scale. Suitable inert solvents are benzene, cyclohexane, nitromethane, acetonitrile and dioxane.

When an inert solvent is used, the addition compounds are prepared by dissolving the amine in the solvent and then adding the uracil gradually, with stirring. Stirring is continued for from ten minutes to two hours. Mild heating may be necessary. Some addition compounds precipitate and can be removed by filtration. Other addition compounds are isolated by evaporating the solvent. The addition compounds prepared in this way are suitable for use without further purification, but can be purified by recrystallization if desired.

In some instances, the uracil and amine are highly soluble in the inert solvent, but the addition compound is not, and so it can be filtered off pure when the reaction is complete.

The complexes of Formula 4 are formed by co-melting the uracil and phenol in a 1:1 to 2:1 (uracil:phenol) ratio. They can also be formed by codissolving the reactants, in the same ratio, in a nonpolar solvent such as nitromethane or a mixture of nitromethane and cyclohexane. Process conditions and isolation procedures are the same as those described above for the addition compounds.

HERBICIDAL COMPOSITIONS

The uracil compounds in Formulae 1 through 5 can be prepared for use by incorporating them with adjuvants.

The amount of herbicide in such preparations can vary over a wide range according to need. Generally speaking, they will contain from about 0.5 to 95%, by weight of a uracil.

Powder and dust preparations can be made by mixing uracils of the invention with finely-divided solids such as talcs, natural clays, pyrophyllite, diatomeous earth; flours such as walnut shell, wheat, redwood, soya bean and cotton seed; or inorganic substances such as magnesium carbonate, calcium carbonate, calcium phosphate, sulfur and lime. These preparations are made by thoroughly blending the active ingredient and the solid. The particles in such preparations are preferably less than 50 microns in average diameter.

Water-soluble preparations can be prepared by mixing a uracil with an alkaline solubilizing agent. Solid bases having a pH of at least 9.5 in a 1% aqueous solution, such as sodium or potassium phosphates, silicates, carbonates, borates, oxides or hydroxides, are suitable. The preparations can contain from 0.5 to 80% active ingredient and from 5 to 99.5% of the solubilizing agent.

Granules and pellets can be made by mixing a finely-divided uracil with a suitable clay, moistening this mixture with from 15 to 20% by weight of water, and then extruding the mass through a suitable die under pressure. The extrusions are cut into pre-determined lengths and then dried. These pellets can be granulated if desired.

Granules or pellets can also be prepared by spraying a suspension or solution of a uracil onto the surface of a preformed granule of clay, vermiculite or other suitable granular material. If the uracil is in solution, it will penetrate into the pores of the granule and so will adhere without the aid of a binding agent. When the active material is insoluble in the liquid and is carried as a suspension, it is preferable that a binding agent such as goulac, dextrin, swollen starch, glue or polyvinyl alcohol be added. In either case, the granule is then dried and ready for use.

The uracils can also be prepared in non-aqueous liquids. Aliphatic and aromatic hydrocarbons, especially those derived from petroleum and having boiling points of from 125° C. to 400° C. are preferred. Hydrocarbons having lower boiling points should not be used because of their undesirable volatilization characteristics and inflammability. These liquid preparations are made by milling the components in a mill such as a pebble mill until the particles have average diameters of from 1 to 50 microns, preferably 5 to 20 microns.

The herbicidal preparations, whatever physical form they take, can also contain a surface-active agent. The surfactant renders the preparations readily dispersible in liquids and improves their action on waxy leaves and the like. For general application, surface-active agents are used in the preparations at concentrations of from about 1 to 10%, by weight. Levels of from 0.5 to 6 parts of surfactant for each part of uracil, however, give unusual and unexpected results. Preparations having these higher levels of surfactants show greater herbicidal effectiveness than can be expected from a consideration of activity of the components used separately.

The term "surface-active agent" is intended to include wetting agents, dispersing agents, suspending agents and emulsifying agents. Surface-active agents suitable for use are set forth in "Detergents and Emulsifiers . . . up-to-date," 1962, John W. McCutcheon, Inc., Morristown, New Jersey. Other surface-active agents which can be used in these preparations are listed in U.S. Patents 2,139,276; 2,412,510; 2,426,417; 2,655,447; and Bulletin E–607 of the Bureau of Entomology and Plant Quarantine of the U.S. Department of Agriculture.

The preparations can also optionally contain adhesives such as gelatin, blood albumin and such resins as rosin alkyd resins. These increase retention and tenacity of deposits following application.

The salts of the compounds of Formula 1 are especially advantageous for use as herbicides because they are soluble in water and can be applied as aqueous solutions.

With respect to the nitrogenous base-addition compounds of Formula 2, it has been found that preparation with polar low-molecular weight amines, such as ethanolamines, propanolamines and butanolamines gives addition compounds soluble in water, especially when the amine is present in excess. Other amines, such as piperidine and octanolamines give addition compounds which are soluble in both water (with an excess of amine present) and hydrocarbon solvents. At the other end of the scale, amines such as dodecylamines, cocoaamines and tallowamines give the addition compounds high hydrocarbon solubility.

Thus, it is apparent that by properly selecting an amine and forming an addition compound with it, uracils of Formula 2 can be formulated as aqueous solutions, wettable powders, or as an oil-emulsifiable or oil-extendable formulation. In this way, the nitrogenous base-addition compounds give formulation and application advantages, while still maintaining the desirable herbicidal characteristics of the parent uracils.

FORMULATION WITH OTHER HERBICIDES

The herbicidal compositions of this invention can be formulated to contain two or more of the uracils. They can also be formulated to contain other known herbicides in addition to the uracils to give compositions which have advantages over the individual components.

Among the known herbicides which can be combined with the uracils of Formula 1 are:

SUBSTITUTED UREAS

3-(3,4-dichlorophenyl)-1,1-dimethylurea
3-(4-chlorophenyl)-1,1-dimethylurea
3-phenyl-1,1-dimethylurea
3-(3,4-dichlorophenyl)-3-methoxy-1,1-dimethylurea
3-(4-chlorophenyl)-3-methoxy-1,1-dimethylurea
3-(3,4-dichlorophenyl)-1-n-butyl-1-methylurea
3-(3,4-dichlorophenyl)-1-methoxy-1-methylurea
3-(4-chlorophenyl)-1-methoxy-1-methylurea
3-(3,4-dichlorophenyl)-1,1,3-trimethylurea
3-(3,4-dichlorophenyl)-1,1-diethylurea
3-(p-chlorophenoxyphenyl)-1,1-dimethylurea

These ureas can be mixed with the uracils of this invention in proportions of form 1:4 to 4:1, respectively, the preferred ratio being 1:2 to 2:1.

SUBSTITUTED TRIAZINES

2-chloro-4,6-bis(ethylamino)-s-triazine
2-chloro-4-ethylamino-6-isopropylamino-s-triazine
2-chloro-4,6-bis(methoxypropylamino)-s-triazine
2-methoxy-4,6-bis(isopropylamino)-s-triazine
2-diethylamino-4-isopropylacetamido-6-methoxy-s-triazine
2-isopropylamino-4-methoxyethylamino-6-methylmercapto-s-triazine
2-methylmercapto-4,6-bis(isopropylamino)-s-triazine
2-methylmercapto-4,6-bis(ethylamino)-s-triazine
2-methylmercapto-4-ethylamino-6-isopropylamino-s-triazine
2-methoxy-4,6-bis(ethylamino)-s-triazine
2-methoxy-4-ethylamino-6-isopropylamino-s-triazine
2-chloro-4,6-bis(isopropylamino)-s-triazine

These triazines can be mixed with the uracils of this invention in proportions of from 1:4 to 4:1, respectively, the preferred ratio being 1:2 to 2:1.

PHENOLS

Dinitro-o-sec.-butylphenol and is salts
Pentachlorophenol and its salts

These phenols can be mixed with the uracils of this invention in the proportions of 1:10 to 20:1, respectively, the preferred ratio being 1:5 to 5:1.

CARBOXYLIC ACIDS AND DERIVATIVES

The following carboxylic acids and derivatives can be mixed with the uracils of this invention in the listed proportions:

A. 2,3,6-trichlorobenzoic acid and its salts
2,3,5,6-tetrachlorobenzoic acid and its salts
2-methoxy-3,5,6-trichlorobenzoic acid and its salts
2-methoxy-3,6-dichlorobenzoic acid and its salts
3-amino-2,5-dichlorobenzoic acid and its salts
3-nitro-2,5-dichlorobenzoic acid and its salts
2-methyl-3,6-dichlorobenzoic acid and its salts
2,4-dichlorophenoxyacetic acid and its salts and esters
2,4,5-trichlorophenoxyacetic acid and its salts and esters
(2-methyl-4-chlorophenoxy)acetic acid and its salts and esters
2-(2,4,5-trichlorophenoxy)propionic acid and its salts and esters
2-(2,4,5-trichlorophenoxy)ethyl-2,2-dichloro-propionate
4-(2,4-dichlorophenoxy)butyric acid and its salts and esters
4-(2-methyl-4-chlorophenoxy)butyric acid and its salts and esters
2,3,6-trichlorobenzyloxypropanol

Mixed in a 1:16 to 8:1 ratio, preferably a 1:4 to 4:1 ratio.

B. 2,6-dichlorobenzonitrile

Mixed in a 1:4 to 4:1 ratio, preferably a 1:3 to 3:1 ratio.

C. Trichloroacetic acid and its salts

Mixed in a 1:2 to 25:1 ratio, preferably a 1:1 to 8:1 ratio.

D. 2,2-dichloropropionic acid and its salts

Mixed in a 1:4 to 8:1 ratio, preferably a 1:2 to 4:1 ratio.

E. N,N-di(n-propyl)thiolcarbamic acid, ethyl ester
N,N-di(n-propyl)thiolcarbamic acid, n-propyl ester
N-ethyl-N-(n-butyl)thiolcarbamic acid, ethyl ester
N-ethyl-N-(n-butyl)thiolcarbamic acid, n-propyl ester

Mixed in a 1:2 to 24:1 ratio, preferably a 1:1 to 12:1 ratio.

F. N-phenylcarbamic acid, isopropyl ester
N-(m-chlorophenyl)carbamic acid, isopropyl ester
N-(m-chlorophenyl)carbamic acid, 4-chloro-2-butynyl ester

Mixed in a 1:2 to 24:1 ratio, preferably a 1:1 to 12:1 ratio.

G. 2,3,6-trichlorophenylacetic acid and its salts

Mixed in a 1:12 to 8:1 ratio, preferably a 1:4 to 4:1 ratio.

H. 2-chloro-N-N-diallylacetamide
Maleic hydrazide

Mixed in a 1:2 to 10:1 ratio, preferably a 1:1 to 5:1 ratio.

INORGANIC AND MIXED INORGANIC-ORGANIC SALTS

The following salts can be mixed with the uracils in the listed proportions:

A. Calcium propylarsonate
Disodium monomethylarsonate
Octyl-dodecylammoniummethylarsonate
Dimethylarsinic acid

Mixed in a 1:4 to 4:1 ratio, preferably a 1:2 to 2:1 ratio.

B. Sodium arsenite

Mixed in a 1:5 to 40:1 ratio, preferably a 1:4 to 25:1 ratio.

C. Lead arsenate
Calcium arsenate

Mixed in a 150:1 to 600:1 ratio, preferably a 100:1 to 400:1 ratio.

D. Sodium tetraborate hydrated, granulated
Sodium metaborate
Sodium pentaborate
Polyborchlorate
Unrefined borate ore such as borascu

Mixed in a 3:1 to 1500:1 ratio, preferably in a 6:1 to 1000:1 ratio.

stituted uracils and halogens set forth in the table for the uracils and halogens in Examples 1, 2, and 3:

Uracil starting reactant	Parts by weight	Halogen	Parts by weight	Uracil product
3-cyclohexyl-6-methyluracil	20.8	Cl	8.0	3-cyclohexyl-5-chloro-6-methyluracil.
3-cyclopentyl-6-propyluracil	22.2	Br	17.0	3-cyclopentyl-5-bromo-6-propyluracil.
3-cyclopentenyl-6-methyluracil	19.0	Cl	8.0	3-cyclopentenyl-5-chloro-6-methyluracil.
3-cyclohexyl-6-methyl-2-thiouracil	22.4	Cl	8.0	3-cyclohexyl-5-chloro-6-methyl-2-thiouracil.
3-cycloheptyl-6-butyluracil	26.4	Br	17.0	3-cycloheptyl-5-bromo-6-butyluracil.
3-cycloheptyl-6-methyluracil	22.2	Cl	8.0	3-cycloheptyl-5-chloro-6-methyluracil.
3-norbornyl-6-methyluracil	22.0	Br	17.0	3-norbornyl-5-bromo-6-methyluracil.
3-cyclopentenyl-6-isobutyluracil	23.4	Cl	8.0	3-cyclopentenyl-5-chloro-6-isobutyluracil.
3-cyclooctyl-6-ethyluracil	24.9	Cl	8.0	3-cyclooctyl-5-chloro-6-ethyluracil.
3-cyclopentyl-6-methyluracil	19.4	Br	17.0	3-cyclopentyl-5-bromo-6-methyluracil.
3-norbornylmethyl-6-methyluracil	23.4	Cl	8.0	3-norbornylmethyl-5-chloro-6-methyluracil.
3,6-dimethyluracil	14.0	Cl	8.0	3,6-dimethyl-5-chlorouracil.
3-ethyl-6-methyluracil	15.4	Cl	8.0	3-ethyl-5-chloro-6-methyluracil.
3-tert.-butyl-6-methyluracil	18.2	Cl	8.0	3-tert.-butyl-5-chloro-6-methyluracil.
3-sec.-octyl-6-methyluracil	23.8	Cl	8.0	3-sec.-octyl-5-chloro-6-methyluracil.
3-(1,1-dimethylhexyl)-6-methyluracil	23.8	Br	17.0	3-(1,1-dimethylhexyl)-5-bromo-6-methyluracil.
3-heptyl-6-methyluracil	22.4	Cl	8.0	3-heptyl-5-chloro-6-methyluracil.
3-(4-bromobutyl)-6-methyluracil	26.6	Br	17.0	3-(4-bromobutyl)-5-bromo-6-methyluracil.
3-methyl-6-ethyluracil	15.4	Cl	8.0	3-methyl-5-chloro-6-ethyluracil.
3-methyl-6-butyluracil	18.2	Cl	8.0	3-methyl-5-chloro-6-butyluracil.
3,6-diethyluracil	16.8	Br	17.0	3,6-diethyl-5-bromouracil.
3,6-dibutyluracil	22.4	Cl	8.0	3,6-dibutyl-5-chlorouracil.
3-hexyl-6-ethyluracil	22.4	Cl	8.0	3-hexyl-5-chloro-6-ethyluracil.
3-isopropyl-6-methyluracil	16.8	Br	17.0	3-isopropyl-5-bromo-6-methyluracil.
3-phenyl-6-methyluracil	20.2	Cl	8.0	3-phenyl-5-chloro-6-methyluracil.
Do	20.2	Br	17.0	3-phenyl-5-bromo-6-methyluracil.
3-phenyl-6-ethyluracil	21.6	Cl	8.0	3-phenyl-5-chloro-6-ethyluracil.
3-phenyl-6-propyluracil	23.0	Cl	8.0	3-phenyl-5-chloro-6-propyluracil.
3-(3-chloro-4-butylphenyl)-6-methyluracil	30.4	Cl	8.0	3-(3-chloro-4-butylphenyl)-5-chloro-6-methyluracil.
3-(3,5-dichloro-4-methylphenyl)-6-ethyluracil	29.9	Cl	8.0	3-(3,5-dichloro-4-methylphenyl)-5-chloro-6-ethyluracil.
3-(m-methoxyphenyl)-6-methyluracil	23.2	Br	17.0	3-(m-methoxyphenyl)-5-bromo-6-methyluracil.
3-(m-fluorophenyl)-6-methyluracil	22.0	Cl	8.0	3-(m-fluorophenyl)-5-chloro-6-methyluracil.
3-phenyl-6-butyluracil	24.4	Cl	8.0	3-phenyl-5-chloro-6-butyluracil.
3-(p-bromophenyl)-6-methyluracil	28.1	Cl	8.0	3-(p-bromophneyl)-5-chloro-6-methyluracil.
3-(3,5-dichloro-4-ethoxyphenyl)-6-methyluracil	31.5	Br	17.0	3-(3,5-dichloro-4-ethoxyphenyl)-5-bromo-6-methyluracil.
3-(m-trifluoromethylphenyl)-6-methyluracil	26.5	Cl	8.0	3-(m-trifluoromethylphenyl)-5-chloro-6-methyluracil.
Do	26.5	Br	17.0	3-(m-trifluoromethylphenyl)-5-bromo-6-methyluracil.
3-(3-nitro-4-chlorophenyl)-6-methyluracil	28.0	Cl	8.0	3-(3-nitro-4-chlorophenyl)-5-chloro-6-methyluracil.
3-isopropyl-6-methyluracil	16.8	Cl	8.0	3-isopropyl-5-chloro-6-methyluracil.
3-p-tolyl-6-amyluracil	27.2	Cl	8.0	3-p-tolyl-5-chloro-6-amyluracil.
3-(3,4-dimethylphenyl)-6-methyluracil	23.0	Br	17.0	3-(3,4-dimethylphenyl)-5-bromo-6-methyluracil.
3-norbornylmethyl-6-methyluracil	23.4	Br	17.0	3-norbornylmethyl-5-bromo-6-methyluracil.
3-(x-chloronorbornyl)-6-methyluracil	25.4	Cl	8.0	5-chloro-3-(x-chloronorbornyl)-6-methyluracil.
3-(3-chloro-4-methylphenyl)-6-methyluracil	25.1	Cl	8.0	3-(3-chloro-4-methylphenyl)-5-chloro-6-methyluracil.
3-(3-chloro-4-methoxyphenyl)-6-methyluracil	26.7	Br	17.0	3-(3-chloro-4-methoxyphenyl)-5-bromo-6-methyluracil.
3-(o-chlorophenyl)-6-methyluracil	23.7	Cl	8.0	3-(o-chlorophenyl)-5-chloro-6-methyluracil.
3-(p-chlorophenyl)-6-propyluracil	26.5	Cl	8.0	3-(p-chlorophenyl)-5-chloro-6-propyluracil.
3-(m-chlorophenyl)-6-methyluracil	23.7	Cl	8.0	3-(m-chlorophenyl)-5-chloro-6-methyluracil.
3-(p-butylbenzyl)-6-methyluracil	27.2	Cl	8.0	3-(p-butylbenzyl)-5-chloro-6-methyluracil.
3-(3,4-dichlorophenyl)-6-ethyluracil	28.5	Cl	8.0	3-(3,4-dichlorophenyl)-5-chloro-6-ethyluracil.
3-benzyl-6-methyluracil	21.6	Cl	8.0	3-benzyl-5-chloro-6-methyluracil.
3-benzyl-6-ethyluracil	23.0	Br	17.0	3-benzyl-5-bromo-6-ethyluracil.
3-(β-phenethyl)-6-methyluracil	23.0	Cl	8.0	3-(β-phenethyl)-5-chloro-6-methyluracil.
3-(p-chlorobenzyl)-6-methyluracil	26.3	Br	17.0	3-(p-chlorobenzyl)-5-bromo-6-methyluracil.
3-benzyl-6-propyluracil	25.8	Cl	8.0	3-benzyl-5-chloro-6-propyluracil.
3-(p-tolyl)-6-ethyl-2-thiouracil	24.6	Br	17.0	3-(p-tolyl)-5-bromo-6-ethyl-2-thiouracil.
3-cyclooctyl-6-methyluracil	23.8	Br	17.0	3-cyclooctyl-5-bromo-6-methyluracil.
3-(3-chloro-4-nitrophenyl)-6-propyl-2-thiouracil	32.5	Cl	8.0	3-(3-chloro-4-nitrophenyl)-5-chloro-6-propyl-2-thiouracil.
3-allyl-6-methyluracil	16.6	Br	17.0	3-allyl-5-bromo-6-methyluracil.
3-propynyl-6-propyl-2-thiouracil	20.8	Br	17.0	3-propynyl-5-bromo-6-propyl-2-thiouracil.
3-isopropyl-6-chlorouracil	18.8	Cl	8.0	3-isopropyl-5,6-dichlorouracil.
3-octenyl-6-methyluracil	23.6	Br	17.0	3-octenyl-5-bromo-6-methyluracil.
3-(2-methoxyethyl)-6-methyluracil	20.4	Br	17.0	3-(2-methoxyethyl)-5-bromo-6-methyluracil.
3-butyl-6-methyl-2-thiouracil	19.8	Cl	8.0	3-butyl-5-chloro-6-methyl-2-thiouracil.
3-cyclopropylmethyl-6-methyluracil	18.0	Br	17.0	3-cyclopropylmethyl-5-bromo-6-methyluracil.
3-isobutyl-6-methyl-2-thiouracil	21.4	Br	17.0	3-isobutyl-5-bromo-6-methyl-2-thiouracil.
3-cyclopentyl-6-chlorouracil	21.4	Br	17.0	3-cyclopentyl-5-bromo-6-chlorouracil.
3-(norbornenyl)-6-methyluracil	21.8	Cl	8.0	3-(norbornenyl)-5-chloro-6-methyluracil.
3-(bornyl)-6-methyluracil	26.1	Cl	8.0	3-bornyl-5-chloro-6-methyluracil.
3-ethyl-6-butyl-2-thiouracil	22.8	Br	17.0	3-ethyl-5-bromo-6-butyl-2-thiouracil.
3-(α-naphthyl)-6-methyluracil	25.5	Br	17.0	3-(α-naphthyl)-5-bromo-6-methyluracil.
3-m-pyridyl-6-methyluracil	19.9	Br	17.0	3-(m-pyridyl)-5-bromo-6-methyluracil.
3-sec.-amylphenyl-6-methyluracil	26.7	Cl	8.0	3-(sec.-amylphenyl)-5-choloro-6-methyluracil.
3-(carvacryl)-6-methyluracil	25.8	Cl	8.0	3-(carvacryl)-5-chloro-6-methyluracil.
3-(norbornenyl)-6-methyluracil	21.8	Cl	24.0	3-(dichloronorbornenyl)-5-chloro-6-methyluracil.

Example 4.—Preparation of 3-sec.-butyl-5-chloro-6-methyluracil

A mixture of 182 parts of 3-sec.-butyl-6-methyluracil, 500 parts of glacial acetic acid, and 82 parts of sodium acetate was stirred at 20–25° C. while 150 parts of sulfuryl chloride were added over a one-hour period. Stirring was continued for one hour longer, after which the reaction mixture was evaporated and the resulting solid recrystallized from an ethanol-water mixture to give 3-sec.-butyl-5-chloro-6-methyluracil as a crystalline solid melting at 153–155° C.

The following 5-chloro uracils are prepared in a similar fashion by substituting the designated amounts of the listed uracil starting reactants for the 3-sec.-butyl-6-methyluracil:

Uracil starting reactant	Parts by weight	Uracil product
3-(fenchyl)-6-methyluracil	276	3-(fenchyl)-5-chloro-6-methyluracil.
3-(3-cyclohexen-1-yl methyl)-6-methyluracil	218	3-(3-cyclohexen-1-yl methyl)-5-chloro-6-methyluracil.
3-(2-methylcyclohexyl)-6-methyluracil	222	3-(2-methylcyclohexyl)-5-chloro-6-methyluracil.
e-(3-methylcyclohexyl)-6-methyluracil	222	3-(3-methylcyclohexyl)-5-chloro-6-methyluracil.
3-(cyclohexylmethyl)-6-methyluracil	222	3-(3-cyclohexylmethyl)-5-chloro-6-methyluracil.
3-(4-methoxy-3-cyclohexene-1-methyl)-6-methyluracil	251	3-(4-methoxy-3-cyclohexene-1-methyl)-5-chloro-6-methyluracil.
3-(2,3-dichloro-3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indanyl)-6-methyluracil.	343	3-(2,3-dichloro-3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indanyl)-5-chloro-6-methyluracil.

E. Ammonium thiocyanate

Mixed in a 1:10 to 20:1 ratio, preferably a 1:5 to 5:1 ratio.

F. Sodium chlorate

Mixed in a 1:1 to 40:1 ratio, preferably a 2:1 to 20:1 ratio.

G. Ammonium sulfamate

Mixed in a 1:1 to 100:1 ratio, preferably a 1:1 to 50:1 ratio.

OTHER ORGANIC HERBICIDES

These organic herbicides can be mixed with the uracils in the listed proportions:

A. 5,6-dihydro-(4A,6A)-dipyrido-(1,2-A,2',1'-C)pyrazinium dibromide

Mixed in a 1:20 to 16:1 ratio, preferably a 1:5 to 5:1 ratio.

B. 3-amino-1,2,4-triazole

Mixed in a 1:20 to 20:1 ratio, preferably a 1:5 to 5:1 ratio.

C. 3,6-endoxohexahydrophthalic acid

Mixed in a 1:3 to 20:1 ratio, preferably a 1:2 to 10:1 ratio.

D. Hexachloroacetone

Mixed in a 1:2 to 16:1 ratio, preferably a 1:1 to 8:1 ratio.

E. Diphenylacetonitrile
N,N-dimethyl-α,α-diphenylacetamide
N,N-di-n-propyl-2,6-dinitro-4-trifluoromethylaniline
N,N-di-n-propyl-2,6-dinitro-4-methylaniline

Mixed in a 1:10 to 30:1 ratio, preferably a 1:5 to 20:1 ratio.

F. O-(2,4-dichlorophenyl)-O-methyl-isopropylphosphoramidothiate
2,3,5,6-tetrachloroterephthalic acid, dimethyl ester

Mixed in a 1:4 to 20:1 ratio, preferably a 1:3 to 15:1 ratio.

G. 2,4-dichloro-4'-nitrodiphenyl ether

Mixed in a 1:10 to 30:1 ratio, preferably a 1:5 to 20:1 ratio.

OTHER SUBSTITUTED URACILS

These uracils can be mixed with other substituted uracils, in the proportions listed below. Methods for the preparation of the listed uracils can be found in copending applications Serial Nos. 167,434, filed February 1, 1962; 89,672, filed February 16, 1961; 89,673, filed February 16, 1961; and 89,674, filed February 16, 1961.

A. 3-cyclohexyl-6-methyluracil
3-cyclohexyl-6-ethyluracil
3-cyclohexyl-6-sec.-butyluracil
3-norbornyl-6-methyluracil
3-cyclopentyl-6-methyluracil
3-cyclohexyl-6-isopropyluracil

Mixed in a 1:4 to 4:1 ratio, preferably a 1:2 to 2:1 ratio.

B. 3-cyclohexyl-5,6-trimethyleneuracil
3-sec.-butyl-5,6-trimethyleneuracil
3-isopropyl-5,6-trimethyleneuracil
3-isopropyl-5,6-tetramethyleneuracil
3-isopropyl-5,6-pentamethyleneuracil

Mixed in a 1:6 to 6:1 ratio, preferably a 1:4 to 4:1 ratio.

C. 3-cyclohexyl-5-bromouracil
3-cyclohexyl-5-chlorouracil
3-isopropyl-5-bromouracil
3-sec.-butyl-5-bromouracil
3-sec.-butyl-5-chlorouracil

Mixed in a 1:6 to 6:1 ratio, preferably a 1:2 to 2:1 ratio.

D. 3-isopropyl-1-trichloromethylthio-5-bromo-6-methyluracil
3-cyclohexyl-1-trichloromethylthio-5-bromo-6-methyluracil
3-sec.-butyl-1-acetyl-5-bromo-6-methyluracil
3-isopropyl-1-acetyl-5-bromo-6-methyluracil
3-isopropyl-1-trichloromethylthio-5-chloro-6-methyluracil

Mixed in a 1:4 to 4:1 ratio, preferably a 1:2 to 2:1 ratio.

All of the foregoing ratios are weight ratios.

EXAMPLES

In order that the invention may be better understood, the following examples are given:

PREPARATION OF ACTIVE INGREDIENTS

Example 1.—Preparation of 3-butyl-5-chloro-6-methyluracil

Eight parts of chlorine are added to a stirred solution of 18.2 parts of 3-butyl-6-methyluracil in 100 parts of glacial acetic acid. The temperature is maintained below 30° C. during this addition. The solution is stirred ½ hour longer at room temperature, then poured into ice water, whereupon a solid separates. This solid is collected by filtration and washed well with water to give 21 parts of crude 3-butyl-5-chloro-6-methyluracil, melting at 120–150° C. It is then recrystallized from a mixture of cyclohexane and ethyl acetate to give 7.5 parts of pure 3-butyl-5-chloro-6-methyluracil, melting at 163–164° C.

Example 2.—Preparation of 5-bromo-3-butyl-6-methyluracil

Seventeen parts of bromine are added over a 20-minute period to a stirred solution of 18.2 parts of 3-butyl-6-methyluracil in 100 ml. of glacial acetic acid. The temperature is maintained below 30° C. during this addition. The solution is stirred at room temperature for an additional 1½ hours, then poured into a mixture of ice and water, with stirring. The white solid which separates is collected by filtration, washed well with water, and dried in a vacuum oven. Thirty-five parts of essentially pure 5-bromo-3-butyl-6-methyluracil are obtained. The compound can be purified by recrystallization from a mixture of hexane and ethyl acetate to give 17.5 parts of pure material, appearing as white needles and melting at 158–160° C.

Example 3.—Preparation of 3-cyclohexyl-5-bromo-6-methyluracil

Seventeen parts of bromine are added over a 20-minute period to a stirred solution of 20.8 parts of 3-cyclohexyl-6-methyluracil in 100 parts of glacial acetic acid. The temperature is maintained below 30° C. during the addition. The solution is stirred at 20–30° C. for an additional 1½ hours, and is then poured, with stirring, into 2 volumes of ice water, whereupon a white solid separates. This solid is collected by filtration, washed well with water, and dried in a vacuum to give essentially pure 3-cyclohexyl-5-bromo-6-methyluracil. It can be recrystallized from ethanol if desired. The melting point of the material is 243–244° C.

The following compounds are prepared as in Examples 1, 2 and 3 by substituting the listed amounts of the sub-

Uracil starting reactant	Parts by weight	Uracil product
3-(1,2,3,4-tetrahydronaphth-5-yl)-6-methyluracil	270	3-(1,2,3,4-tetrahydronaphth-5-yl)-5-chloro-6-methyluracil.
3-(2-decahydronaphthyl)-6-methyluracil	276	3-(2-decahydronaphthyl)-5-chloro-6-methyluracil.
3-tert.-butyl-6-methyluracil	182	3-tert.-butyl-5-chloro-6-methyluracil.
3-(1-ethylpropyl)-6-methyluracil	196	3-(1-ethylpropyl)-5-chloro-6-methyluracil.
3-(1-ethylbutyl)-6-methyluracil	210	3-(1-ethylbutyl)-5-chloro-6-methyluracil.
3-(1-methylpentyl)-6-methyluracil	210	3-(1-methylpentyl)-5-chloro-6-methyluracil.
3-(1-ethyl-1-methylpropyl)-6-methyluracil	210	3-(1-ethyl-1-methylpropyl)-5-chloro-6-methyluracil.
3-(1-ethylpentyl)-6-methyluracil	224	3-(1-ethylpentyl)-5-chloro-6-methyluracil.
3-(1,1-dimethylpentyl)-6-methyluracil	224	3-(1,1-dimethylpentyl)-5-chloro-6-methyluracil.
3-(1-ethyl-1-methylbutyl)-6-methyluracil	224	3-(1-ethyl-1-methylbutyl)-5-chloro-6-methyluracil.
3-(1-ethyl-1-methylpentyl)-6-methyluracil	238	3-(1-ethyl-1-methylpentyl)-5-chloro-6-methyluracil.
3-(2,4,5-trichlorophenyl)-6-methyluracil	305	3-(2,4,5-trichlorophenyl)-5-chloro-6-methyluracil.
3-(o-biphenyl)-6-methyluracil	278	3-(o-biphenyl)-5-chloro-6-methyluracil.
3-(3-chloro-4-sec.-amyloxyphenyl)-6-methyluracil	299	3-(3-chloro-4-sec.-amyloxyphenyl)-5-chloro-6-methyluracil.
3-(sec.-amyloxyphenyl)-6-methyluracil	288	3-(sec.-amyloxyphenyl)-5-chloro-6-methyluracil.
3-(2,5-dichlorophenyl)-6-methyluracil	271	3-(2,5-dichlorophenyl)-5-chloro-6-methyluracil.
3-(2,5-dibromophenyl)-6-methyluracil	360	3-(2,5-dibromophenyl)-5-chloro-6-methyluracil.
3-(octen-7-yl)-6-methyluracil	88	3-(7,8 dichlorooctyl)-5-chloro-6-methyluracil.

Example 5.—Preparation of 3-sec.-butyl-5-bromo-6-methyluracil

A solution of 182 parts of 3-sec.-butyl-6-methyluracil in 700 parts of acetic acid containing 82 parts of sodium acetate was treated with 160 parts of bromine. After standing overnight, the mixture, which contained some solid, was evaporated to a solid under reduced pressure. The solid was recrystallized from an ethanol-water mixture to give, as a white crystalline solid, 3-sec.-butyl-5-bromo-6-methyluracil melting at 157.5–160° C.

The following 5-bromo uracils are similarly prepared by substituting the designated amounts of the listed uracil starting reactants for the 3-sec.-butyl-6-methyluracil:

Example 6.—Preparation of 3-phenyl-5-bromo-6-methyl-2-thiouracil

A mixture of 34 parts by weight of bromine and 200 parts by weight of glacial acetic acid is added gradually at 20° C. to a stirring mixture of 21.8 parts by weight of 3-phenyl-6-methyl-2-thiouracil, 200 parts by weight of glacial acetic acid and 200 parts by weight of anhydrous sodium acetate. The entire mixture is stirred until the bromine absorption is complete. At this point, 13.0 parts by weight of zinc dust are added gradually over a period of ½ hour.

The reaction is stirred for an additional ½ hour, then diluted with 4 volumes of water and the solid removed

Uracil starting reactant	Parts by weight	Uracil product
3-tert.-butyl-6-methyluracil	182	3-tert.-butyl-5-bromo-6-methyluracil.
3-(1-methylbutyl)-6-methyluracil	196	3-(1-methylbutyl)-5-bromo-6-methyluracil.
3-(1,1-dimethylpropyl)-6-methyluracil	195	3-(1,1-dimethylpropyl)-5-bromo-6-methyluracil.
3-(1,1-dimethylbutyl)-6-methyluracil	210	3-(1,1-dimethylbutyl)-5-bromo-6-methyluracil.
3-(1-methylhexyl)-6-methyluracil	224	3-(1-methylhexyl)-5-bromo-6-methyluracil.
3-(1-ethylhexyl)-6-methyluracil	238	3-(1-ethylhexyl)-5-bromo-6-methyluracil.
3-(1-methylheptyl)-6-methyluracil	238	3-(1-methylheptyl)-5-bromo-6-methyluracil.
3-(1,1-dimethylhexyl)-6-methyluracil	238	3-(1,1-dimethylhexyl)-5-bromo-6-methyluracil.
3-isobutyl-6-methyluracil	182	3-isobutyl-5-bromo-6-methyluracil.
3-(m-nitrophenyl)-6-methyluracil	227	3-(m-nitrophenyl)-5-bromo-6-methyluracil.
3-(2,5-dichloro-4-nitrophenyl)-6-methyluracil	298	3-(2,5-dichloro-4-nitrophenyl)-5-bromo-6-methyluracil.
3-[p-(tert.-butyl)benzyl]-6-methyluracil	272	3-[p-(tert.-butyl)benzyl]-5-bromo-6-methyluracil.
3-(p-methylbenzyl)-6-methyluracil	230	3-p-methylbenzyl)-5-bromo-6-methyluracil.
3-(3,4-dichlorobenzyl)-6-methyluracil	275	3-(3,4-dichlorobenzyl)-5-bromo-6-methyluracil.
3-(5-phenylamyl)-6-methyluracil	272	3-(5-phenylamyl)-5-bromo-6-methyluracil.
3-(p-nitrobenzyl)-6-methyluracil	261	3-(p-nitrobenzyl)-5-bromo-6-methyluracil.
3-(3,4-dimethylbenzyl)-6-methyluracil	244	3-(3,4-dimethylbenzyl)-5-bromo-6-methyluracil.
3-(2-methyl-5-isopropylbenzyl)-6-methyluracil	272	3-(2-methyl-5-isopropylbenzyl)-5-bromo-6-methyluracil.
3-(p-methoxybenzyl)-6-methyluracil	246	3-(p-methoxybenzyl)-5-bromo-6-methyluracil.
3-[p-(sec.-butoxy)benzyl]-6-methyluracil	288	3-[p-(sec.-butoxy)benzyl]-5-bromo-6-methyluracil.
3-(2-chloroethyl)-6-methyluracil	188	3-(2-chlcroethyl)-5-bromo-6-methyluracil.
3-(octen-7-yl)-6-methyluracil	88	3-(7,8-dibromooctyl)-5-bromo-6-methyluracil.
3-(2-bromoethyl)-6-methyluracil	233	3-(2-bromoethyl)-5-bromo-6-methyluracil.
3-(isopropyl)-6-bromouracil	233	3-(isopropyl)-5,6-dibromouracil.
3-(2-hydroxypropyl)-6-methyluracil	186	3-(2-hydroxypropyl)-5-bromo-6-methyluracil.
3-(3-hydroxypropyl)-6-methyluracil	186	3-(3-hydroxypropyl)-5-bromo-6-methyluracil.
3-(1,1,5-trimethyl-5-hydroxypentyl)-6-methyluracil	255	3-(1,1,5-trimethyl-5-hydroxypentyl)-5-bromo-6-methyluracil.
3-(2-hydroxyethyl)-6-methyluracil	170	3-(2-hydroxyethyl)-5-bromo-6-methyluracil.
3-(3-methoxypropyl)-6-methyluracil	198	3-(3-methoxypropyl)-5-bromo-6-methyluracil.
3-(3-ethoxyethyl)-6-methyluracil	196	3-(2-ethoxyethyl)-5-bromo-6-methyluracil.
3-(5-methoxycarbonylpentyl)-6-methyluracil	242	3-(5-methoxycarbonylpentyl)-5-bromo-6-methyluracil.
3-(ethoxycarbonylmethyl)-6-methyluracil	212	3-(ethoxycarbonylmethyl)-5-bromo-6-methyluracil.
3-(5-cyanopentyl)-6-methyluracil	221	3-(5-cyanopentyl)-5-bromo-6-methyluracil.
3-(2-cyanoethyl)-6-methyluracil	179	3-(2-cyanoethyl)-5-bromo-6-methyluracil.
3-(2-buten-3-yl)-6-methyluracil	182	3-(2-buten-3-yl)-5-bromo-6-methyluracil.
3-(2-buten-2-yl)-6-methyluracil	182	3-(2-buten-2-yl)-5-bromo-6-methyluracil.
3-(norbornenyl)-6-methyluracil	218	3-(norbornenyl)-5-bromo-6-methyluracil.
Do	72	3-(dibromonorbornyl)-5-bromo-6-methyluracil.
3-(bornyl)-6-methyluracil	261	3-(bornyl)-5-bromo-6-methyluracil.
3-(1,2-dimethylcyclopentyl)-6-methyluracil	219	3-(1,2-dimethylcyclopentyl)-5-bromo-6-methyluracil.
3-(α-methyl-1-cyclopentyl-1-ethyl)-6-methyluracil	234	3-(α-methyl-1-cyclopentyl-1-ethyl)-5-bromo-6-methyluracil.
3-(fenchyl)-6-methyluracil	276	3-(fenchyl)-5-bromo-6-methyluracil.
3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indenyl)-6-methyluracil	272	3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indenyl)-5-bromo-6-methyluracil.
3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indenyl)-6-methyluracil	91	3-(2,3-dibromo-3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indanyl)-5-bromo-6-methyluracil.
3-(p-methoxycyclohexyl)-6-methyluracil	207	3-(p-methoxycyclohexyl)-5-bromo-6-methyluracil.
3-(bicyclo[3,2,1]oct-3-yl)-6-methyluracil	248	3-(bicyclo[3,2,1]oct-3-yl)-5-bromo-6-methyluracil.
3-(bicyclo[2,2,2]oct-2-yl methyl)-6-methyluracil	262	3-(bicyclo[2,2,2]oct-2-yl methyl)-5-bromo-6-methyluracil.
3-(bicyclo[2,2,2]oct-2-yl)-6-methyluracil	248	3-(bicyclo[2,2,2]oct-2-yl)-5-bromo-6-methyluracil.
3-(norbornenylmethyl)-6-methyluracil	232	3-(norbornenylmethyl)-5-bromo-6-methyluracil.
3-(6-methoxy-3,3-dimethylindanyl)-6-methyluracil	289	3-(6-methoxy-3,3-dimethylindanyl)-6-methyl-5-bromouracil.
3-(1-decahydronaphthyl)-6-methyluracil	276	3-(1-decahydronaphthyl)-5-bromo-6-methyluracil.
3-(1-1,2,3,4-tetrahydronaphthyl)-6-methyluracil	270	3-(1-1,2,3,4-tetrahydronaphthyl)-5-bromo-6-methyluracil.
3-(3a,4,5.6,7,7a-hexahydro-4,7-methano-5-indanyl)-6-methyluracil	274	3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indanyl)-5-bromo-6-methyluracil.

by filtration. The solid is extracted with 200 parts by weight of a 1 N sodium hydroxide solution. Acidification of the basic filtrate gives essentially pure solid 3-phenyl-5-bromo-6-methyl-2-thiouracil, melting at 230–232° C.

Example 7.—Preparation of 5-iodo-3-isopropyl-6-methyluracil

A mixture of 168 parts by weight of 3-isopropyl-6-methyluracil, 1000 parts by weight of acetic acid, and 253 parts by weight of iodine is stirred at 100° C. as 75 parts by weight of fuming nitric acid are gradually added. When the addition is complete, the dark colored solution is refluxed for about one-half hour and then cooled to ice-bath temperature.

Excess iodine which precipitates is filtered off and the filtrate is diluted with 4000 parts by weight of cold water. The iodine remaining in solution is reduced to iodide ion by adding a saturated solution of sodium bisulfite until the solution becomes colorless.

The aqueous solution is extracted with 6000 parts by weight of methylene chloride. The organic layer is separated, washed with saturated sodium bicarbonate solution, and then dried with 200 parts by weight of magnesium sulfate.

The 5-iodo-3-isopropyl-6-methyluracil is recrystallized from acetonitrile. It melts at 181° C.

Example 8.—Preparation of 3-butyl-6-methyl-5-nitrouracil

A solution of 40 parts by volume of fuming nitric acid and 40 parts by volume of fuming sulfuric acid (20% SO_3) is stirred at 25–30° C. while 20 parts by weight of 3-butyl-6-methyluracil are added portion-wise over a 30-minute period. The solution is stirred an additional 30 minutes, then poured slowly into 4 volumes of a stirred ice-water mixture. The uracil separates out as a pale yellow solid. It is collected by filtration, washed well with water, and air-dried. One recrystallization from 1,1,1-trichloroethane gives ananlytically pure 3-butyl-6-methyl-5-nitrouracil, melting at 170–171° C.

Example 9

The following 5-nitro substituted uracils can be prepared in a manner similar to Example 8 above by substituting equivalent amounts of the substituted uracil starting reactants set forth in the following table for the 3-butyl-6-methyluracil shown in Example 8:

Uracil starting reactant	5-nitrouracil product
3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indenyl)-6-methyluracil.	3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indenyl)-5-nitro-6-methyluracil.
3-cyclohexyl-6-chlorouracil	3-cyclohexyl-5-nitro-6-chlorouracil.
3-(3-methoxypropyl)-6-methyluracil.	3-(3-methoxypropyl)-6-methyl-5-nitrouracil.
3-tert.-butyl-6-methyluracil	3-tert.-butyl-6-methyl-5-nitrouracil.
3-(p-chlorophenyl)-6-propyluracil	3-(p-chlorophenyl)-5-nitro-6-propyluracil.
3-(o-methoxycyclohexyl)-6-methyluracil.	3-(o-methoxycyclohexyl)-6-methyl-5-nitrouracil.
3-benzyl-6-methyl-2-thiouracil	3-benzyl-6-methyl-5-nitro-2-thiouracil.
3-decyl-6-butyluracil	3-decyl-6-butyl-5-nitrouracil.
3-cyclohexyl-6-methyluracil	3-cyclohexyl-6-methyl-5-nitrouracil.
3-cyclopentenyl-6-butyl-2-thiouracil.	3-cyclopentenyl-6-butyl-5-nitro-2-thiouracil.
3-isopropyl-6-methyluracil	3-isopropyl-6-methyl-5-nitrouracil.
3-sec.-butyl-6-methyluracil	3-sec.-butyl-6-methyl-5-nitrouracil.
3-cyclohexyl-6-ethyluracil	3-cyclohexyl-6-ethyl-5-nitrouracil.
3-phenyl-6-methyluracil	3-phenyl-6-methyl-5-nitrouracil.

Example 10.—Preparation of 3-butyl-5,6-dimethyluracil

A mixture of 23.2 parts by weight of butylurea 28.8 parts by weight of ethyl 2-methyl-3-ketobutyrate, 0.5 part by weight of 85% orthophosphoric acid, and 200 parts by weight of benzene was heated at reflux. Water was collected in a Dean-Stark trap. After 24 hours, 2.5 parts by weight of water had been collected and there appeared to be no more separating. The solution was decanted and evaporated to a mixture of viscous oil and solid, which was taken up in 200 parts by weight of ethanol containing 10.6 parts by weight of sodium methoxide and heated at reflux for 10 minutes. Most of the ethanol was removed under reduced pressure and the residue was taken up in just sufficient water to dissolve it. Concentrated hydrochloric acid was added to lower the pH to 4 and the resulting white solid was collected by filtration. This was recrystallized from an ethanol-water mixture to give pure 3-butyl-5,6-dimethyluracil, melting at 116.5–117° C.

The following uracils are similarly prepared by substituting equivalent amounts of the ureas and thioureas and equivalent amounts of the β-keto esters set forth in the table for the butylurea and ethyl 2-methyl-3-ketobutyrate:

β-Keto ester	Parts by weight	Urea	Parts by weight	Uracil product
Ethyl 2-methyl-3-ketobutyrate	28.8	Sec.-amylurea	26.0	3-sec.-amyl-5,6-dimethyluracil.
Do	28.8	Isopropylurea	21.0	3-isopropyl-5,6-dimethyluracil.
Ethyl 2-butyl-3-ketovalerate	39.8	Amylurea	26.0	3-amyl-5-butyl-6-ethyluracil.
Ethyl 2-ethyl-3-ketovalerate	34.2	Propylthiourea	23.6	3-propyl-5,6-diethyl-2-thiouracil.
Ethyl 2-methyl-3-ketobutyrate	28.8	Sec.-butylurea	23.5	3-sec.-butyl-5,6-dimethyluracil.
Do	28.8	Allylurea	20.0	3-allyl-5,6-dimethyluracil.
Ethyl 2-methyl-3-ketoheptanoate	37.0	Propynylurea	19.6	3-propynyl-5-methyl-6-butyluracil.
Ethyl 2-methyl-3-ketobutyrate	28.8	Benzylurea	30.0	3-benzyl-5,6-dimethyluracil.
Ethyl 2-ethyl-3-ketooctanoate	45.4	Benzylthiourea	33.2	3-benzyl-5-ethyl-6-amyl-2-thiouracil.
Ethyl 2-methyl-3-ketohexanoate	34.2	β-phenethylurea	32.8	3-(β-phenethyl)-5-methyl-6-propyluracil.
Ethyl 2-methyl-3-ketobutyrate	28.8	Furfurylurea	28.0	3-furfuryl-5,6-dimethyluracil.
Do	28.8	Tert.-amylurea	26.0	3-tert.-amyl-5,6-dimethyluracil.
Do	28.8	1-(hexahydro-4,7-methano-5-indanyl) urea.	39.0	3-(3a-4-5-6-7-7a-hexahydro-4,7-methano-5-indanyl)-5,6-dimethyluracil.
Do	28.8	Phenylurea	27.2	3-phenyl-5,6-dimethyluracil.
Ethyl 2-butyl-3-ketovalerate	39.8	p-chlorophenyl-thiourea.	37.2	3-(p-chlorophenyl)-5-butyl-6-ethyl-2-thiouracil.
Do	34.2	m-Tolylurea	30.0	3-(m-tolyl)-5,6-diethyluracil.
Ethyl 2-methyl-3-ketooctanoate	39.8	p-Anisylurea	33.2	3-(p-anisyl)-5-methyl-6-amyluracil.
Ethyl 2-ethyl-3-ketobutyrate	31.4	o-Nitrophenylthiourea	39.4	3-(o-nitrophenyl)-5-ethyl-6-methyl-2-thiouracil.
Ethyl 2-methyl-3-ketobutyrate	28.8	Octyn-7-ylurea	33.6	3-(octyn-7-yl)-5,6-dimethyluracil.
Ethyl 2-propyl-3-ketohexanoate	39.8	Allylurea	20.0	3-allyl-5,6-dipropyluracil.
Ethyl 2-methyl-3-ketobutyrate	28.8	β-phenethylthiourea	36.0	3-(β-phenethyl)-5,6-dimethyl-2-thiouracil.
Ethyl 2-fluoro-3-ketobutyrate	29.4	Sec.-butylurea	23.5	3-sec.-butyl-5-fluoro-6-methyluracil.
Do	29.4	2-(5-indanyl) ethylurea	38.0	3-[2-(5-indanyl)ethyl]-5-fluoro-6-methyluracil.
Ethyl 2-methyl-3-ketobutyrate	28.8	Cyclopentenylurea	25.8	3-cyclopentenyl-5,6-dimethyluracil.

Example 11.—Preparation of 3-cyclohexyl-5-hydroxymethyl-6-methyluracil

A mixture of 208 parts by weight of 3-cyclohexyl-6-methyluracil, 1400 parts by weight of water, 315 parts by weight of ethyl alcohol, 66 parts by weight of paraformaldehyde, and 20 parts by weight of barium hydroxide is heated until the components are completely dissolved. The solution is then stripped and the oil which remains is extracted with ether. This ether extract is dried with magnesium sulfate, filtered, and concentrated at reduced pressure to a slightly gummy solid. This solid is recrystallized from acetonitrile to give 3-cyclohexyl-5-hydroxymethyl-6-methyluracil, melting at 175–176° C.

The following compounds are similarly prepared by substituting equivalent amounts of the designated uracil starting reactants for 3-cyclohexyl-6-methyluracil:

Uracil starting reactant	5-hydroxyalkyluracil products
3-isopropyl-6-methyluracil	3-isopropyl-5-hydroxymethyl-6-methyluracil.
3-cyclohexyl-6-ethyl-2-thiouracil	3-cyclohexyl-6-ethyl-5-hydroxymethyl-2-thiouracil.
3-(bicyclo[2,2,2]oct-5-en-2-yl)-6-methyluracil	3-(bicyclo[2,2,2]oct-5-en-2-yl)-5-hydroxymethyl-6-methyluracil.
3-benzyl-6-methyluracil	3-benzyl-5-hydroxymethyl-6-methyluracil.
3-cylcopentenyl-6-propyluracil	3-cyclopentenyl-5-hydroxymethyl-6-propyluracil.
3-(α-ethylbicyclo[2,2,2]oct-5-en-2-yl-methyl)-6-methyluracil	3-(α-ethylbicyclo[2,2,2]oct-5-en-2-yl-methyl)-5-hydroxymethyl-6-methyluracil.
3-(1,1-dimethylpropyl)-6-ethyl-2-thiouracil	3-(1,1-dimethylpropyl)-6-ethyl-5-hydroxymethyluracil.
3-(*p*-chlorophenyl)-6-butyluracil	3-(*p*-chlorophenyl)-6-butyl-5-hydroxymethyluracil.
3-cyclooctyl-6-methyluracil	3-cyclooctyl-5-hydroxymethyl-6-methyluracil.
3-allyl-6-methyluracil	3-allyl-5-hydroxymethyl-6-methyluracil.
3-(hexahydro-5-indenylmethyl)-6-methyluracil	3-(hexahydro-5-indenylmethyl)-5-hydroxymethyl-6-methyluracil.
3-(7-bromo-4-indenyl)-6-methyluracil	3-(7-bromo-4-indenyl)-5-hydroxymethyl-6-methyluracil.
3-(hexahydro-1-indenyl)-6-methyluracil	3-(hexahydro-1-indenyl)-5-hydroxymethyl-6-methyluracil.
3-(1,4,5,6-tetrachloro-7,7-dimethoxy bicyclo[2,2,1]hept-5-en-2-yl-methyl)-6-methyluracil.	3-(1,4,5,6-tetrachloro-7,7-dimethoxy bicyclo[2,2,1]hept-5-en-2-yl-methyl)-5-hydroxymethyl-6-methyluracil.
3-(5,6,7,8,10,10-hexachloro-1,2,3,4,4a,5,8,8a-octahydro-1,4-5,8-dimethano-2-naphthyl)-6-methyluracil.	3-(5,6,7,8,10,10-hexachloro-1,2,3,4,4a,5,8,8a-octahydro-1,4-5,8-dimethano-2-naphthyl)-5-hydroxymethyl-6-methyluracil.
3-(5,6,7,8,9,9-hexachloro-1,2,3,4,4a,5,8,8a-octahydro-5,8-methano-2-naphthyl)-6-ethyluracil.	3-(5,6,7,8,9,9-hexachloro-1,2,3,4,4a,5,8,8a-octahydro-5,8-methano-2-naphthyl)-5-hydroxymethyl-6-ethyluracil.
3-(5,6,7,8,9,9-hexachloro-1,2,3,4,4a,5,8,8a-octahydro-5,8-methano-2-naphthyl)-6-methyluracil.	3-(5,6,7,8,9,9-hexachloro-1,2,3,4,4a,5,8,8a-octahydro-5,8-methano-2-naphthyl)-5-hydroxymethyl-6-methyluracil.
3-(5,6,7,8-tetrachloro-decahydro-10,10-dimethoxy-1,4-5,8-dimethano-2-naphthyl)-6-methyluracil.	3-(5,6,7,8-tetrachloro-decahydro-10,10-dimethoxy-1,4-5,8-dimethano-2-naphthyl)-5-hydroxymethyl-6-methyluracil.
3-(5,6,7,8,10,10-hexachloro-decahydro-1,4-5,8-dimethano-2-naphthyl)-6-methyluracil.	3-(5,6,7,8,10,10-hexachloro-decahydro-1,4-5,8-dimethano-2-naphthyl)-5-hydroxymethyl-6-methyluracil.
3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indanyl)-6-methyluracil	3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indanyl)-5-hydroxymethyl-6-methyluracil.

Example 12.—Preparation of 3-isopropopyl-5-methoxymethyl-6-methyluracil

To a warm solution of 21.7 parts by weight of 5-chloromethyl-3-isopropyl-6-methyluracil in 150–200 parts by weight of methanol is added 5.4 parts by weight of sodium methoxide. The solution is heated to reflux for 15 minutes. The methanol is then evaporated, and the product extracted with ether. Evaporation of the ether yields 3-isopropyl-5-methoxymethyl-6-methyluracil in a crystalline state.

Example 13.—Preparation of 3-isopropyl-5-methylthiomethyl-6-methyluracil

A suspension of 21.7 parts by weight of 5-chloromethyl-3-isopropyl-6-methyluracil and 7.0 parts by weight of sodium methylmercaptide in 100 parts by weight of tetrahydrofuran is heated to reflux for 45 minutes while gaseous methylmercaptan is added at a slow rate. The solvent is then evaporated and the product extracted from the residue with ether. Evaporation of the ether yields the desired 3-isopropyl-5-methylthiomethyl-6-methyluracil in a crystalline state.

Example 14.—Preparation of 3-isopropyl-5-methoxymethyl-6-methyluracil

A mixture of 168 parts of 3-isopropyl-6-methyluracil, 1400 parts of water, 375 parts of ethanol, 66 parts of p-formaldehyde and 20 parts of barium hydroxide is heated and stirred at reflux for ½ hour or until a fine solid begins to precipitate. The solution is chilled, neutralized with dilute hydrochloric acid, and concentrated to a viscous oil at reduced pressure at 50–80° C.

The oil (194 parts) is stirred with 500 parts of methanol, and the solution is filtered free of the inorganic salts. Ten parts of chloroacetic acid are added to the solution and it is charged into a bomb and heated at 125° C. for 5 hours. The reaction mixture is concentrated to an oil, which is taken up in 400 parts of acetonitrile, refluxed for a short time with 5 parts of decolorizing carbon, filtered hot and concentrated to an oil.

The oil is dissolved in 250 parts of methanol and chilled to about —50° C. Pure, white 3-isopropyl-5-methoxymethyl-6-methyluracil precipitates. It is filtered off and dried. It melts at 116.5° C. to 118.5° C.

Example 15.—Preparation of 3-isopropyl-5 methylthiomethyl-6-methyluracil

An autoclave is charged with 198 parts of 5-hydroxymethyl-3-isopropyl-6-methyluracil, 1500 parts of methanol and 100 parts of methyl mercaptan. The mixture is shaken and heated at 125° C. for 5 hours. It is then cooled and poured into 7000 parts of ice and water.

The white solid which precipitates is filtered off and recrystallized from acetonitrile or nitromethane giving pure 3-isopropyl-5-methylthiomethyl-6-methyluracil.

The following compounds are prepared as in Examples 12, 13, 14 and 15 by substituting equivalent amounts of the appropriate substituted uracils and equivalent amounts of appropriate alkoxides, alkylmercaptides or arylmercaptides for the substituted uracils and sodium methoxide or alkylmercaptide in Examples 12 and 13, or by employing the alternate routes described in Examples 14 and 15. Equivalent amounts of hydrogen sulfide can be substituted for methyl mercaptan in Example 15 to give the corresponding 5-mercaptomethyl substituted uracil.

3-cyclohexyl-5-methoxymethyl-6-methyluracil
3-cyclooctyl-5-methylthiomethyl-6-ethyl-2-thiouracil
3-cyclohexyl-5-sec.-amyloxymethyl-6-chlorouracil
3-octyl-5-propoxymethyl-6-methyluracil
3-phenyl-5-methoxymethyl-6-methyluracil
3-(p-chlorophenyl)-5-ethoxymethyl-6-propyluracil
3-allyl-5-ethoxymethyl-6-ethyl-2-thiouracil
3-benzyl-5-ethylthiomethyl-6-methyluracil
3-cyclopentenyl-5-methylthiomethyl-6-methyluracil
3-butyl-5-methoxymethyl-6-ethyluracil
3-tert.-butyl-5-methoxymethyl-6-methyluracil
3-norbornenyl-5-methoxymethyl-6-methyluracil
3-cyclohexyl-5-mercaptomethyl-6-methyluracil
3-cyclohexyl-5-carboxymethylthiomethyl-6-methyluracil
3-octyn-2-yl-5-methoxymethyl-6-methyluracil
3-(2,3-dichloro-2,3,4,5,6,7,7a-hexahydro-4,7-methano-indanyl)-5-methoxymethyl-6-methyluracil

3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indenyl)-5-indanyl-methoxymethyl-6-methyluracil
3-decahydronaphthyl-5-methoxymethyl-6-methyluracil
3-boryl-5-methoxymethyl-6-methyluracil
3-(5,6,7,8-tetrahydronaphth-1-yl)-5-methoxymethyl-6-methyluracil
3-(1,2-dimethylcyclopentyl)-5-ethoxymethyl-6-methyluracil
3-isopropyl-5-phenylthiomethyl-6-methyluracil
3-cyclohexyl-5-phenylthiomethyl-6-methyluracil
3-(5,6,7,8,9,9-hexachloro-1,2,3,4,4a,5,8,8a-octahydro-5,8-methano-2-naphthyl)-5-methoxymethyl-6-methyluracil
3-(5,6,7,8,10,10-hexachloro-1,2,3,4,4a,5,8,8a-octahydro-1,4-5,8-dimethano-2-naphthyl)-5-methoxymethyl-6-methyluracil
3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indanyl)-5-methoxymethyl-6-methyluracil
3-norbornyl-5-methylthiomethyl-6-methyluracil
3-fenchyl-5-methoxymethyl-6-methyluracil
3-o-methylcyclohexyl-5-methoxymethyl-6-methyluracil

Example 16.—Preparation of 3-isopropyl-5-chloromethyl-6-methyluracil

A mixture of 17 parts by weight of 3-isopropyl-6-methyluracil and 38 parts by weight of chloromethyl-methyl ether is heated in an autoclave under endogenous pressure at 100° C. for 25 hours. The material is cooled and the excess reagent evaporated under vacuum. The product is extracted from the residue with dry dioxane. The dioxane is then evaporated to dryness, leaving essentially pure 5-chloromethyl-3-isopropyl-6-methyluracil as a residue.

Example 17.—Preparation of 5-chloromethyl-3-isopropyl-6-methyluracil

To 452 parts of rapidly stirred thionyl chloride, maintained below 25° C. with an ice bath, are gradually added 198 parts of 5-hydroxymethyl-3-isopropyl-6-methyluracil. Caution should be used in carrying out this reaction because of the large amounts of acidic gases produced and the vigor of the reaction.

An efficient condenser is used to retain the thionyl chloride reactant, and a scrubber is used to wash away the gases produced.

When a complete solution has been obtained, it is distilled to dryness at reduced pressure at 50° C. The solid is triturated with 200 parts of a 1:1 mixture of 1,1,2-trichloroethane and heptane, and filtered.

The solid is then recrystallized from 600 parts of the same solvent, giving pure 5-chloromethyl-3-isopropyl-6-methyluracil, M.P. 151.5° C. to 153° C.

The following 5-halomethyluracils can be prepared by the method of either Example 16 or 17 by substituting an appropriate uracil for the 3-isopropyl-6-methyluracil of Example 16, or by substituting an appropriate thionyl halide and substituted uracil for the thionyl chloride and 5-hydroxymethyl-4-isopropyl-6-methyluracil of Example 17:

3-cyclohexyl-5-chloromethyl-6-methyluracil
3-(p-anisyl)-5-chloromethyl-6-propyl-2-thiouracil
3-propynyl-5-chloromethyl-6-ethyluracil
3-octyl-5-chloromethyl-6-methyluracil
3-sec.-butyl-6-bromo-5-chloromethyluracil
3-(β-phenethyl)-5-chloromethyl-6-methyl-2-thiouracil
3-isoamyl-5-chloromethyl-6-butyluracil
3-(m-nitrophenyl)-5-chloromethyl-6-methyluracil
3-cyclohexyl-5-bromomethyl-6-methyluracil
3-cyclohexyl-5-fluoromethyl-6-methyluracil
3-sec.-butyl-5-fluoromethyl-6-methyluracil
3-sec.-butyl-5-bromomethyl-6-methyluracil

Example 18.—Preparation of 3-sec.-butyl-6-methyl-5-thiocyanatouracil

A mixture of 182 parts of 3-sec.-butyl-6-methyluracil, 700 parts of acetic acid and 152 parts of ammonium thiocyanate is stirred at 10° C. to 15° C. as 160 parts of bromine dissolved in 180 parts of acetic acid are gradually added over a period of 2 to 4 hours. When the addition is complete, the reaction is stirred for a short time at 15° C. to insure completion, and then diluted with 9000 parts of ice and water.

The mixture is extracted with 4000 parts of dichloromethane. The organic layer is separated, filtered free of polymeric material, washed with sodium bicarbonate solution, dried over magnesium sulfate, filtered and concentrated to dryness at reduced pressure.

The solid residue is triturated with cyclohexane and filtered. It is recrystallized from 1,1,2-trichloroethane to give pure, white 3-sec.-butyl-6-methyl-5-thiocyanato uracil, M.P. 157° C. to 158° C.

Example 19.—Preparation of 3-pentyl-6-methyl-5-thiocyanato-2-thiouracil

A suspension of 39 parts by weight of ammonium thiocyanate in 200 parts by volume of carbon tetrachloride is treated slowly and with vigorous agitation with 17.6 parts by weight of bromine. Occasional external cooling is required to hold the temperature of the reaction mixture at 15–20° C. At the end of the addition, stirring is continued until the bromine color is discharged, after which the solution is filtered rapidly, preferably in the absence of moisture. To this solution is added 21.2 parts by weight of 3-pentyl-6-methyl-2-thiouracil, and the resulting mixture is stirred at room temperature for 5 hours. At the end of this time, the solvent is removed, leaving behind essentially pure 3-pentyl-6-methyl-5-thiocyano-2-thiouracil.

The following compounds are similarly prepared by substituting equivalent amounts of the appropriate uracil starting reactants for 3-pentyl-6-methyl-2-thiouracil:

3-cyclopentenyl-6-methyl-5-thiocyanatouracil
3-heptyl-6-ethyl-5-thiocyanato-2-thiouracil
3-norbornyl-6-methyl-5-thiocyanatouracil
3-(m-nitrophenyl)-6-butyl-5-thiocyanatouracil
3-benzyl-6-methyl-5-thiocyanatouracil
3-(β-phenethyl)-6-methyl-5-thiocyanatouracil
3-cyclohexyl-6-methyl-5-thiocyanatouracil
3-cyclopentyl-6-butyl-5-thiocyanatouracil
3-cycloheptenyl-6-ethyl-5-thiocyanato-2-thiouracil
3-isopropyl-6-methyl-5-thiocyanatouracil
6-methyl-3-phenyl-5-thiocyanatouracil

Example 20.—Preparation of 5-chloro-6-chloromethyl-3-isopropyluracil

A mixture of 168 parts of 3-isopropyl-6-methyluracil, 100 parts of acetic acid and 650 parts of water is vigorously stirred as 147 parts of chlorine are gradually added as a gas. The temperature is maintained at 30° C. to 35° C. with a cooling bath.

The white slurry is stirred for an additional hour and 400 parts of concentrated hydrochloric acid are then added gradually with stirring. The slurry is heated at reflux for 5 hours, chilled to about 20° C. and filtered. The filter cake is resuspended in 500 parts of cold water and refiltered.

The dry filter cake is recrystallized from acetonitrile to give 5-chloro-6-chloromethyl-3-isopropyluracil, M.P. 186° C. to 188° C.

The following compounds are similarly prepared by substituting equivalent amounts of the appropriate uracil starting reactants, halogen, and halogen acid for the 3-isopropyl-6-methyluracil, chlorine and hydrochloric acid:

5-bromo-6-bromomethyl-3-isopropyluracil
5-chloro-6-chloromethyl-3-cyclohexyluracil
5-chloro-6-chloromethyl-3-phenyluracil
5-bromo-6-bromomethyl-3-(3-chlorophenyl)uracil
3-sec.-butyl-5-chloro-6-chloromethyluracil
3-sec.-butyl-5-chloro-6-(α-chlorobutyl)uracil

3-cyclopentyl-5-chloro-6-(α-chloroethyl)uracil
3-cycloheptyl-5-chloro-6-chloromethyluracil

Example 21.—Preparation of 3-phenyl-5-methoxy-6-methyl-2-thiouracil

A mixture of 34 parts of bromine and 200 parts of glacial acetic acid is added gradually at 20° C. to a stirred mixture of 21.8 parts of 3-phenyl-6-methyl-2-thiouracil, 250 parts of glacial acetic acid and 200 parts of anhydrous sodium acetate. Stirring is continued until the bromine absorption is complete.

At this point, the mixture is distilled under reduced pressure to remove the acetic acid. The residue is combined with 350 parts of methanol and 130 parts of sodium methoxide and heated at reflux under anhydrous conditions for 5 hours. Methanol is removed from the reaction by distillation and the remaining solid is added to 250 parts of glacial acetic acid.

The latter mixture is stirred while 13.0 parts of zinc dust are added over a ½ hour period. Stirring is continued for an additional half hour, then the reaction is diluted with 4 volumes of water and the solid filtered.

The solid is extracted with 200 parts of 1 Normal sodium hydroxide solution and acidification of the basic filtrate gives essentially pure 3-phenyl-5-methoxy-6-methyl-2-thiouracil.

The following compounds can be prepared in a similar fashion by substituting equivalent amounts of the appropriate thiouracil starting reactant and sodium alkoxide or sodium mercaptide for the 3-phenyl-6-methyl-2-thiouracil and sodium methoxide:

3-isopropyl-5-methoxy-6-methyl-2-thiouracil
3-sec.-butyl-5-methoxy-6-methyl-2-thiouracil
3-cyclohexyl-5-methoxy-6-methyl-2-thiouracil
3-decahydronaphth-1-yl-5-methoxy-6-methyl-2-thiouracil
3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-idenyl)-5-methoxy-6-methyl-2-thiouracil
3-tert.-butyl-5-butoxy-6-methyl-2-thiouracil
3-isopropyl-5-isopropoxy-6-methyl-2-thiouracil
3-norbornyl-5-methoxy-6-methyl-2-thiouracil
3-isopropyl-5-methylthio-6-methyl-2-thiouracil
3-sec.-butyl-5-butylthio-6-methyl-2-thiouracil
3-cyclohexyl-5-isopropylthio-6-methyl-2-thiouracil

Example 22.—Preparation of 3-cyclohexyl-5-methoxy-6-methyluracil

A total of 25.4 parts of 3-cyclohexyl-5-methoxy-6-methyl-2-thiouracil and 250 parts of 2 Normal sodium hydroxide is stirred rapidly as 14.0 parts of dimethylsulfate are added gradually over a period of ½ hour. A product forms which is collected and added to 200 parts of a 10% solution of chloroacetic acid.

The resulting mixture is heated with rapid stirring at reflux temperature for a 2 hour period. The entire reaction is then cooled and filtered, yielding essentially pure 3-cyclohexyl-5-methoxy-6-methyluracil.

The following compounds can be prepared in a like fashion by substituting equivalent amounts of the appropriate thiouracil starting reactant for 3-cyclohexyl-5-methoxy-6-methyl-2-thiouracil:

3-norbornyl-5-methoxy-6-methyluracil
3-tert.-butyl-5-butoxy-6-methyluracil
3-decahydronaphth-1-yl-5-methoxy-6-methyluracil
3-isopropyl-5-methoxy-6-methyluracil
3-isopropyl-5-isopropoxy-6-methyluracil
3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indenyl)-5-methoxy-6-methyluracil
3-isopropyl-5-ethylthio-6-methyluracil
3-cyclohexyl-5-methylthio-6-methyluracil
3-tert.-butyl-5-propylthio-6-methyluracil

Example 23.—Preparation of 5-cyano-3-cyclohexyl-6-methyluracil

A mixture of 142 parts of cyclohexylurea, 178 parts of triethyl orthoacetate and 72 parts of malononitrile is stirred at reflux for two hours. The volatile substances are distilled off at reduced pressure giving a solid residue of [1-(3-cyclohexylureido)ethylidene]malononitrile, which is recrystallized from aqueous ethanol.

A mixture of 218 parts of thio[1-(3-cyclohexylureido)ethylidene]malononitrile, 700 parts of methanol and 54 parts of sodium methoxide is protected from atmospheric moisture and allowed to stand at room temperature for five days. The alcohol is removed by distillation at reduced pressure at room temperature, and the resulting solid is dissolved in 2000 parts of cold water. Acetic acid is gradually added until the mixture is slightly acidic.

The 3-cyclohexyl-6-methyl-5-cyanocytosine which precipitates is filtered off, washed with water, dried, and recrystallized from ethanol.

Two hundred and thirty-two parts of this 3-cyclohexyl-6-methyl-5-cyanocytosine are dissolved in 1430 parts of 3 Normal hydrochloric acid, and the solution is refluxed for 3 hours. After cooling, the resulting white solid 5-cyano-3-cyclohexyl-6-methyluracil is filtered off and recrystallized from ethanol.

The following 3,6-disubstituted-5-cyanouracils can be prepared in the same fashion by substituting an appropriate urea for cyclohexyl urea and an appropriate triethyl orthoester for the triethyl orthoacetate:

5-cyano-3-isopropyl-6-methyluracil
3-sec.-butyl-5-cyano-6-methyluracil
5-cyano-6-methyl-3-norbornyluracil
5cyano-6-methyl-3-phenyluracil
5-cyano-6-methyl-3-phenyluracil
3-cyclohexyl-5-cyano-6-methyluracil

Example 24.—Preparation of 3-cyclohexyl-5-(2-hydroxyethyl)-6-methyluracil

A mixture of 426 parts by weight of cyclohexylurea, 423 parts by weight of 2-acetylbutyrolactone, 879 parts by weight of benzene, 1030 parts by weight of dioxane, and 40 parts by weight of 85% phosphoric acid is stirred at reflux temperature.

The water given off is removed by azeotropic distillation. When no more water is given off, the solution is cooled, decanted, and concentrated to dryness under reduced pressure. Three recrystallizations of the resulting solid from acetonitrile give 2-(2-hydroxyethyl)-3-cyclohexylureido)crotonic acid, γ-lactone.

A mixture of 302 parts by weight of the 2-(2-hydroxyethyl)-3-(3-cyclohexylureido)crotonic acid, γ-lactone, 1580 parts by weight of absolute ethanol, and 130 parts by weight of sodium methoxide is refluxed for 15 minutes. It is then concentrated to dryness at reduced pressure, and the residue is dissolved in 1500 parts by weight of water.

This solution is cooled, acidified with hydrochloric acid to pH 5, and the resulting white precipitate, 3-cyclohexyl-5-(2-hydroxyethyl)-6-methyluracil, is filtered off, dried, and recrystallized from a mixture of ethanol and water.

The following uracils are similarly prepared by replacing the cyclohexylurea with equivalent amounts of appropriate ureas and by replacing 2-acetylbutyrolactone, where indicated, with equivalent amounts of the corresponding homologues:

5-(2-hydroxyethyl)-3-isopropyl-6-methyluracil
5-(2-hydroxyethyl)-6-methyl-3-phenyluracil
5-(2-hydroxypropyl)-6-methyl-3-norbornyluracil
3-sec.-butyl-5-(2-hydroxyhexyl)-6-methyluracil

Example 25.—Preparation of 3-butyl-5-chloro-6-methyluracil, sodium salt

A solution of 4 parts of sodium hydroxide in 100 parts of water is treated with 21.7 parts of 3-butyl-5-chloro-6-methyluracil. Stirring and warming is employed to effect solution. The water is removed from the solution under reduced pressure, leaving 3-butyl-5-chloro-6-methyluracil, sodium salt, as a white solid.

The metal salts of the compounds prepared according

to the foregoing examples can be prepared as in Example 25 by substituting equivalent amounts of other substituted uracils and other metallic hydroxides for the 3-butyl-5-chloro-6-methyluracil and sodium hydroxide. The following list contains examples of salts prepared in this fashion:

3-sec.-butyl-5-chloro-6-methyluracil, sodium salt
3-butyl-5-bromo-6-methyluracil, potassium salt
3-hexyl-5-chloro-6-ethyluracil, 1/2 calcium salt
3-phenyl-5-chloro-6-methyluracil, sodium salt
3-(m-fluorophenyl)-5-bromo-6-methyluracil, 1/2 magnesium salt
3-(p-cumyl)-5-chloro-6-propyluracil, potassium salt
3-(β-phenethyl)-5-bromo-6-butyluracil, 1/2 calcium salt
3-allyl-5-chloro-6-methyl-2-thiouracil, strontium salt
3-isoamyl-5-bromo-6-methyluracil, potassium salt
3 - (3a,4,5,6,7,7a - hexahydro-4,7-methano-5-indanyl)-5-bromo-6-methyluracil, sodium salt
3-(p-chlorophenyl)-5-nitro-6-propyluracil, 1/2 barium salt
3-allyl-5-bromo-6-methyluracil, lithium salt
3-isopropyl-5-bromo-6-methyluracil, sodium salt
3-isopropyl-5,6-dimethyl-2-thiouracil, sodium salt
3-propynyl-5-methyl-6-butyluracil, potassium salt
3-benzyl-5,6-dimethyluracil, iron salt
3-sec.-butyl-5-chloro-6-methyluracil, 1/2 calcium salt
3-sec.-butyl-5-bromo-6-methyluracil, sodium salt
3-phenyl-5,6-dimethyluracil, sodium salt
3-(p-anisyl)-5-methyl-6-amyluracil, sodium salt
3-allyl-5,6-dipropyluracil, 1/2 magnesium salt
3-sec.-butyl-5-nitro-6-methyluracil, sodium salt
3-benzyl-5-methoxy-6-propyl-2-thiouracil, 1/2 barium salt
3-phenyl-5-methoxy-6-methyluracil, sodium salt
3-butyl-6-methyl-5-thiocyanouracil, manganous salt
3-phenyl-6-butyl-5-thiocyanouracil, 1/2 calcium salt
3-phenyl-5-bromo-6-methyluracil, sodium salt
3-sec.-butyl-5-bromo-6-methyluracil, potassium salt
3 - (1,1 - dimethylbutyl)-5-chloro-6-methyluracil, sodium salt
3-phenyl-5-bromo-6-methyluracil, potassium salt
3-phenyl-5-bromo-6-methyluracil, lithium salt
3-norbornyl-5-bromo-6-methyluracil, potassium salt
3 - norbornenyl-5-hydroxymethyl-6-methyluracil, 1/2 calcium salt
3-cyclopentenyl-5,6-dimethyluracil, 1/2 calcium salt
3-cyclopropylmethyl-5-bromo-6-methyluracil, sodium salt
3-cyclohexyl-5-methoxy-6-methyluracil, sodium salt
3-isopropyl-5-nitro-6-methyluracil, sodium salt
3-(m-tolyl)-5-bromo-6-methyluracil, sodium salt
3-(m-chlorophenyl)-5-chloro-6-methyluracil, sodium salt
3-cyclohexyl-5-bromo-6-methyluracil, sodium salt
3-cyclohexyl-5-chloro-6-methyluracil, sodium salt
3-cyclopentenyl-5-chloro-6-isobutyluracil, 1/2 calcium salt
3-cyclohexyl-5-fluoro-6-methyluracil, potassium salt
3-cyclohexyl-6-methyl-5-nitrouracil, sodium salt
3 - cyclopentenyl-5-methylthiomethyl-6-methyluracil, lithium salt
3-cyclohexyl-6-methyl-5-thiocyanouracil, sodium salt
3-tert.-butyl-5-chloro-6-methyluracil, sodium salt
3 - (1-ethyl propyl)-5-bromo-6-methyluracil, potassium salt
3-tert.-butyl-5-chloro-6-methyluracil, sodium salt
3-allyl-5-allyl-6-propyluracil, sodium salt

Example 26.—Preparation of 1:1 complex of 5-bromo-3-isopropyl-6-methyluracil and pentachlorophenol

A mixture of 247 parts of 5-bromo-3-isopropyl-6-methyluracil, 266 parts of pentachlorophenol and 1250 parts of cyclohexane is stirred at reflux as 50 parts of nitromethane are gradually added. The physical appearance of the solid changes rapidly. When no further change is noticed, the mixture is chilled and the solid product is filtered off and recrystallized from nitromethane. The complex melts at 142–143° C.

The following complexes can be prepared in a similar fashion by substituting equivalent amounts of the proper phenol and uracil for the 5-bromo-3-isopropyl-6-methyluracil and pentachlorophenol:

1:1 complex pentachlorophenol and 5-bromo-3-sec.-butyl-6-methyluracil
1:1 complex pentachlorophenol and 5-bromo-3-tert.-butyl-6-methyluracil
1:1 complex pentachlorophenol and 5-bromo-3-(2-methylbutyl)-6-methyluracil
1:1 complex m-methylphenol and 5-bromo-3-sec.-butyl-6-methyluracil
1:1 complex p-nitrophenol and 5-bromo-3-sec.-butyl-6-methyluracil
1:1 complex phenol and 3-isopropyl-5-nitro-6-methyluracil
1:1 complex p-methoxyphenol and 5-bromo-3-sec.-butyl-6-methyluracil

Example 27.—Preparation of 2:1 complex of 5-bromo-3-isopropyl-6-methyluracil and phenol

A dry mixture of 247 parts of 5-bromo-3-isopropyl-6-methyluracil and 47 parts of phenol is gradually heated until a clear melt is formed. This is stirred for a short time to insure complex formation, and then cooled. The resulting solid cake, M.P. 140.5–142° C., is sufficiently pure for incorporation into herbicidal formulations.

The following complexes can be formed by substituting molecularly equivalent quantities of the appropriate phenols for phenol, and the appropriate uracils for 5-bromo-3-isopropyl-6-methyluracil:

1:2 complex p-chlorophenol and 5-bromo-3-isopropyl-6-methyluracil
1:2 complex p-nitrophenol and 5-bromo-3-isopropyl-6-methyluracil
1:2 complex p-methoxyphenol and 5-bromo-3-isopropyl-6-methyluracil
1:2 complex p-methylphenol and 5-bromo-3-isopropyl-6-methyluracil
1:2 complex pentachlorophenol and 5-chloro-3-sec.-butyl-6-methyluracil
1:2 complex pentachlorophenol and 5-chloro-3-isopropyl-6-methyluracil
1:2 complex phenol and 3-phenyl-5-bromo-6-methyluracil

Example 28.—Preparation of 2:1 complex of 5-bromo-3-isopropyl-6-methyluracil and phenol

A mixture of 247 parts of 5-bromo-3-isopropyl-6-methyluracil, 1200 parts of cyclohexane and 47 parts of phenol is refluxed with stirring for from 5 to 30 minutes. This slurry is filtered while hot, and the solid filter cake is recrystallized from 300 parts of nitromethane. The pure 2:1 solid complex thus formed is filtered off, and dried at room temperature in air.

Example 29.—Preparation of the ethanolamine addition compound of 5-bromo-3-isopropyl-6-methyluracil

A solution is prepared by mixing together 247 parts of 5-bromo-3-isopropyl-6-methyluracil, 392 parts of acetonitrile and 61 parts of ethanolamine. The solvent is distilled off at reduced pressure and the oil which remains gradually solidifies. The molar addition compound is recrystallized from nitromethane; it melts at 80–83° C.

Example 30.—Preparation of the octylamine addition compound of 5-bromo-3-isopropyl-6-methyluracil

A mixture containing one part of octylamine and one part of 5-bromo-3-isopropyl-6-methyluracil is stirred and maintained at about 40° C. with gentle heating until the ingredients are in complete solution. The solution is cooled and the hard granular crystals which form are filtered off and washed three times with small portions of cold benzene. The resulting molar addition compound melts at 74–78° C.

Example 31.—Preparation of the ethylenediamine addition compound of 5-bromo-3-isopropyl-6-methyluracil

Two hundred and forty-seven parts of 5-bromo-3-iso-

propyl-6-methyluracil are dissolved in 300 parts of acetonitrile at ambient temperature. Sixty parts of ethylenediamine are added and the mixture is stirred vigorously. The solid white addition compound which gradually precipitates is filtered off and dried at room temperature under reduced pressure. The addition compound melts at 102–106° C. It can be further purified by recrystallization from acetonitrile if desired.

The following addition compounds are prepared according to any of the procedures of Examples 29, 30 and 31 by substituting molar equivalents of a properly substituted uracil and an apppropriate amine for 5-bromo-3-isopropyl - 6 - methyluracil, ethanolamine, octylamine or ethylenediamine:

5-bromo-3-sec.-butyl-6-methyluracil·ethanolamine addition compound
3-sec.-butyl-5-chloro-6-methyluracil·ethanolamine addition compound
5-bromo-3-sec.-butyl-6-methyluracil·ethylenediamine addition compound
5-bromo-3-sec.-butyl-6-methyluracil·dodecylamine addition compound
5-bromo-3-sec.-butyl-6-methyluracil·cocoadiamine addition compound
5-bromo-3-isopropyl-6-methyluracil·dodecylamine addition compound
5-chloro-3-isopropyl-6-methyluracil·ethanolamine addition compound
5-bromo-3-tert.-butyl-6-methyluracil·ethanolamine addition compound
5-bromo-3-tert.-butyl-6-methyluracil·dodecylamine addition compound
5,6-dimethyl-3-isopropyluracil·ethanolamine addition compound
5,6-dimethyl-3-isopropyluracil·dodecylamine addition compound
5-bromo-6-methyl-3-phenyluracil·ethylenediamine addition compound
5-bromo-6-methyl-3-phenyluracil·dodecylamine addition compound
5-chloro-6-methyl-3-phenyluracil·ethanolamine addition compound
3-isopropyl-6-methyl-5-nitrouracil·ethanolamine addition compound
5-bromo-6-methyl-3-sec.-pentyluracil·ethanolamine addition compound
5-bromo-3-(3-chlorophenyl)-6-methyluracil·piperidine addition compound
5-bromo-6-methyl-3-norbornyluracil·piperidine addition compound
3-isopropyl-6-methyl-5-nitrouracil·ammonia addition compound
3-isopropyl-6-methyl-5-nitrouracil·methylamine addition compound
5-bromo-6-ethyl-3-isopropyluracil·ethanolamine addition compound

Example 32.—Preparation of the tetrabutylammonium salt of 5,6-dimethyl-3-isopropyluracil

A total of 182 parts of 5,6-dimethyl-3-isopropyluracil is gradually added, with stirring, to 1000 parts of a one-molar solution of tetrabutylammonium hydroxide in methanol. When solution is complete, the solvent is distilled off at reduced pressure. The while solid tetrabutylammonium salt of 5,6-dimethyl-3-isopropyluracil which remains is sufficiently pure for incorporation into herbicidal formulations.

The following tertaalkylammonium salts can be similarly prepared by substituting molar equivalent amounts of a properly substituted uracil and an appropriate quaternary ammonium hydroxide for 5,6-dimethyl-3-isopropyluracil and tetrabutylammonium hydroxide:

Tetramethylammonium salt of 3-tert.-butyl-5-chloro-6-methyluracil
Tetramethylammonium salt of 3-sec.-butyl-5-chloro-6-methyluracil
Tetramethylammonium salt of 3-sec.-butyl-5,6-dimethyluracil
Tetramethylammonium salt of 5-bromo-3-sec.-butyl-6-methyluracil
Tetramethylammonium salt of 5-bromo-3-(2-methylpropyl)-6-methyluracil
Tetrabutylammonium salt of 3-isopropyl-6-methyl-5-nitrouracil
Tetrabutylammonium salt of 3-sec.-butyl-6-methyl-5-nitrouracil
Tetrabutylammonium salt of 5-bromo-6-methyl-3-phenyluracil
Tetrabutylammonium salt of 5-chloro-6-methyl-3-phenyluracil
Tetrabutylammonium salt of 5,6-dimethyl-3-phenyluracil
Tetrabutylammonium salt of 5-bromo-3-(3-chlorophenyl)-6-methyluracil
Tetrabutylammonium salt of 5-bromo-6-methyl-3-norbornyluracil
Trimethyldodecylammonium salt of 5-chloro-3-cyclohexyl-6-methyluracil
1,1′-ethylene-2,2′-dipyridinium salt of -di-(5-bromo-3-isopropyl-6-methyluracil)
Trimethyldodecylammonium salt of 5-bromo-3-isopropyl-6-methyluracil

FORMULATIONS

Liquid Compositions

Aqueous Solutions

Example 33

	Percent
3-cyclopentyl-5-chloro-6-methyluracil, Na salt	20
Sodium lauryl sulfate	2
Water	78

The solution is prepared by dissolving the two soluble salts in the water, with agitation. This solution is suitable for quick dilution to desired spray levels.

Other soluble salts suitable for preparation of water concentrates are

3-sec.-butyl-5-bromo-6-methyluracil, sodium salt
3-isopropyl-5-methoxy-6-propyluracil, tetrabutylammonium salt
3-isopropyl-5-bromo-6-methyluracil, sodium salt
3-isopropyl-5-bromo-6-methyluracil, trimethyldodecylammonium salt

This aqueous solution is used for post-emergence weed control. A concentration of 1.0 pound of active ingredient per acre in 30 gallons of water gives excellent control of crabgrass, pigweed, velvetweed, and flower-of-an-hour.

At concentrations of 10 to 20 pounds per acre in 80 gallons of water, this composition gives excellent control of a wide variety of annual and broadleaf weeds growing in railroad yards on railroad ballast.

Example 34

	Percent
5-bromo-3-isopropyl-6-methyluracil	50
Ethanolamine	50

The components are mixed together at room temperature until a clear solution is formed. This solution is infinitely extendable with water and can be diluted to any concentration.

Ten pounds of this formulation are mixed with 60 gallons of water in a spray tank. Ten pounds of trimethylnonyl ether of polyethylene glycol, are added. Only slight agitation is required for complete mixing.

Sixty gallons of this solution are applied to an acre of roadside. Excellent initial kill of foliage and residual weed control is obtained. Such species as wild oats, cheatgrass, crabgrass, foxtails, ryegrass, volunteer small grain and wild mustard are controlled.

The following uracils can be substituted, in equivalent amounts, for 5-bromo-3-isopropyl-6-methyluracil, with excellent results:

3-sec.-butyl-5-bromo-6-methyluracil
3-tert.-butyl-5-bromo-6-methyluracil
3-cyclohexyl-5,6-dimethyluracil

AQUEOUS SUSPENSIONS AND DISPERSIONS

Example 35

	Percent
3-cyclohexyl-5,6-dimethyluracil	28.0
Sodium lignin sulfonate	15.0
Hydrated attapulgite	2.0
Disodium phosphate	0.8
Sodium pentachlorophenate	0.5
Water	53.7

The above ingredients are mixed and pebble-milled or sand-milled until the average particle size of the active material is substantially less than 5 microns. The resulting stable thixotropic suspension does not cake and can be readily diluted with water to form a dilute, very slow settling suspension which requires no agitation during application.

This aqueous suspension, applied as a directed spray at 1 to 3 pounds of active ingredient per acre in 30 gallons of water, gives good pre-emergence control of barnyard grass, mustard species, and jungle rice in sugarcane.

Example 36

	Percent
3-isopropyl-5,6-dimethyluracil	30.00
Sodium lignin sulfonate	15.00
Hydrated attapulgite	1.75
Anhydrous disodium phosphate	0.80
Sodium pentachlorophenate	0.50
Water	51.95

The above components are mixed together and pebble-milled or sand-milled until substantially all the active material is below 5 microns in particle size. The resultant stable aqueous suspension can be readily diluted with water to give a highly dispersd, very slowly settling composition which can be sprayed from equipment having no means for agitating the suspension.

This composition is applied at 20 pounds of active ingredient per acre in 100 gallons of water for control of annual and perennial broadleaf and grass weeds growing along pipeline rights-of-way. Excellent control of broomsedge, crabgrass, nutsedge, Johnson grass, pigweed, goldenrod, and evening primrose is obtained.

Example 37

A water suspension is prepared by grinding the following ingredients 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.

	Percent
3-(3a,4,5,6,7,7a-hexahydro - 4,7 - methano-5-indenyl)-5-hydroxymethyl-6-methyluracil	25
Hydrated attapulgite	2
Lignin sulfonic acids, sodium salt	5
Water	68

Example 38

A water suspension is prepared by grinding the following ingredients with water in a ball or roller mill:

	Percent
3-isopropyl-5-bromo-6-methyluracil	25
Hydrated attapulgite	2
Lignin sulfonic acid, sodium salt	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 39

The water suspensions of Examples 37 and 38 are useful for the control of seedling brush and annual weeds growing along fence rows. Application concentrations of 10 to 20 pounds of active ingredient per acre in 120 gallons of water give control of oak and willow seedlings, crabgrass, and chickweed.

Example 40

	Percent
3-cyclohexyl-5-bromo-6-methyluracil	15.0
3-(p-chlorophenyl)-1,1-dimethylurea	15.0
Modified polyacrylic acid, Na salt	0.4
Low viscosity polyvinyl alcohol	1.0
Water	68.6

This composition is wet-milled until the particles are substantially all below 10 microns in size.

The formulation is applied in 40 gallons of water at the rate of 2 pounds (active) per acre as a directed post-emergence spray for the control of young seedling annual grasses and broadleaf weeds growing in asparagus. Excellent control of crabgrass, pigweed, chickweed, bachelor's button, and seedling Johnson grass is obtained.

Example 41

	Percent
3-cyclohexyl-5-chloro-6-methyluracil	15
2-chloro-4,6-bis(ethylamino)-s-triazine	15
Sodium lignin sulfonate	10
Wyoming bentonite	2
Water	58

This composition is wet-milled until the particles are substantially all below 10 microns in size, to give a stable dispersion concentrate.

This formulation is useful for the control of annual and perennial boadleaf and grass weeds growing in firebreaks. An application of 20 pounds (active) per acre in 100 gallons of water gives excellent control of pigweed, seedling bindweed, buckhorn plantain, asters, crabgrass, bromegrass, and Johnson grass.

Example 42

	Percent
3-cyclohexyl-5-bromo-6-methyluracil	13.00
Pentachlorophenol	16.00
Sodium lignin sulfonate	15.00
Hydrated attapulgite	1.75
Water	54.25

These components are wet-milled until the particles are substantially all below 10 microns in size, to give a stable dispersion.

The composition is useful for the control of existing vegetation. It gives extended residual control of weeds. An application of 22 pounds (active) per acre in 100 gallons of water gives rapid initial kill of daisy, goldenrod, bouncing Bet, ragweed, bromegrasses, and annual ryegrass, with control for an extended period.

AQUEOUS CONCENTRATES

Example 43

	Percent
3-cyclohexyl-5,6-dimethyluracil, sodium salt	25
Sodium lauryl sulfate	1
Water	74

An aqueous concentrate is prepared by dissolving the two solid components in water. The concentrate can be easily diluted to use levels and sprayed.

3-cyclohexyl-5-bromo-6-methyluracil, sodium salt and 3-cyclohexyl-5-chloro-6-methyluracil, sodium salt are also formulated easily as aqueous concentrates by this method.

These aqueous concentrates are conveniently applied with a pressure-type hand sprayer. One and one-half pounds of active ingredient per acre in 40 gallons of water give excellent pre-emergence control of foxtail, watergrass, and Johnson grass seedlings in sugarcane. These

formulations, at 20 pounds of active ingredient per acre, are also useful for general control of annual and perennial weeds on industrial sites and railroad ballast.

Example 44

	Percent
2-tert.-butyl-5-chloro-6-butyluracil, potassium salt	25
Sodium lauryl sulfate	1
Water	74

The concentrate is prepared as a homogeneous solution by agitating the solid components in the water. It can then be readily diluted to use levels with water.

This formulation is used at 1 to 2 pounds of active ingredients per acre in 30 gallons of water for the post-emergence control of annual weeds in sugar cane. A directed spray to seedling crabgrass, water grass, pigweed, and lamb's-quarters gives excellent control of these weeds.

OIL FORMULATIONS

Example 45

	Percent
5-bromo-3-sec.-butyl-6-methyluracil	25
Dodecylamine	25
Aromatic heavy naphtha	50

These components are mixed until a clear solution results.

Sixty pounds of this formulation are mixed with 80 gallons of Lion Herbicidal Oil No. 6 and applied at the rate of 15 pounds of active ingredient per acre to a weed infestation of annual and perennial broadleaves and grasses growing along a railroad right-of-way. Excellent initial contact kill of weeds, followed by good residual weed control, is obtained. Such weed species as pigweed, ragweed, Johnson grass, broomsedge, foxtail, crabgrass, dock and wild carrot are controlled.

5 - bromo - 3 - tert. - butyl - 6 - methyluracil, 5 - bromo-3-phenyl-6-methyluracil and the 1:1 addition compound of 5-bromo-3-sec.-butyl-6-methyluracil with dodecylamine can be formulated in a similar fashion, with excellent herbicidal results.

Example 46

	Percent
3-sec.-butyl-5-bromo-6-methyluracil	40
Soya lecithin	3
Substantially aliphatic, low viscosity mineral oil, e.g., kerosene or diesel oil	57

The oil suspension is prepared by pregrinding the active material and mixing it with the other components with agitation, or by blending all the components together, then pebble-milling or sand-milling them to reduce the particle size of the active component. The product is suitable for dilution with weed oils to form an oil spray.

This formulation is diluted with 80 gallons of an herbicidal oil such as Lion Herbicidal Oil No. 6 and applied at 12 pounds of active ingredients per acre for general over-all weed control along cyclone fences and railroad ballast.

Good control is obtained for several months. Quack grass, cheat, witch grass, buttonweed, and Jimson weed are controlled.

Example 47

	Percent
3-cyclohexyl-5-bromo-6-methyluracil	25
Blend of polyalcohol carboxylic esters and oil-soluble petroleum sulfonates	6
Diesel oil	69

These components are mixed together and milled in a roller mill, pebble mill, or sand mill until the particles of the active component are substantially all below 10 microns in size. The resulting suspension can be emulsified in water or diluted further with weed oils for spray application.

This formulation is diluted with 80 gallons of Lion Herbicidal Oil No. 6 and applied at 10 to 20 pounds of active ingredient per acre for the control of weeds such as morning glory, chickweed, pigweed, lamb's-quarter, yarrow, ragweed, wild carrot, quack grass, witchgrass, crabgrass, and oak and maple seedlings growing along railroad rights-of-way. Excellent control is obtained.

Example 48

	Percent
3-isopropyl-5-bromo-6-methyluracil	20.0
Alkyl aryl polyether alcohol	2.5
Oil soluble petroleum sulfonate	2.5
Methyl isobutyl ketone	75.0

The emulsifiable oil is prepared by mixing the above components until a homogeneous solution results. It can then be emulsified in water for application.

This emulsifiable oil is useful for weed control on railroad rights-of-way, in railroad yards, and on sidings. When this composition is diluted with 100 gallons of water per acre and sprayed from a railroad spray car at 10 pounds of active ingredient per acre, mixed vegetation such as quack grass, crabgrass, Bermuda grass, bromegrass, ragweed, cockleburr, lamb's-quarters, and pigweed is controlled for an extended period.

Example 49

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.

	Percent
3-benzyl-5-chloro-6-methyluracil	25
Soya lecithin	5
Aliphatic hydrocarbon oil	70

This oil suspension is applied in 50 gallons of water, at a concentration of 16–20 pounds of active ingredient per acre, as an over-all spray on a stand of established nutsedge grass. Excellent control of the nutsedge is obtained. A check of the tubers shows severe injury.

Example 50

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 of the active ingredient is less than 5 microns.

	Percent
3-(p-cumyl)-5-chloro-6-methyluracil	25
Diesel oil	67
Polyoxyethylene sorbitan ester of mixed rosin and fatty acids	8

This oil suspension is diluted with water to form a water emulsion and is applied at a concentration of 2 pounds of active ingredient per acre as a directed spray to grasses growing in sugar cane. This treatment gives excellent control of mixed annular graseses and broadleaves.

Example 51

	Percent
3-(p-tolyl)-5-bromo-6-methyl-2-thiouracil	15
Diesel oil	80
Polyoxyethylene sorbitan esters of mixed rosin and fatty acids	5

These ingredients are ground 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. This oil suspension is diluted with water to form a water emulsion for application to plants.

3-sec.-butyl-5-bromo-6-methyluracil and 3-isopropyl-5-bromo-6-methyluracil are also prepared as oil suspensions in this manner.

Example 52

The oil suspension compositions of Examples 49, 50,

and 51, when applied to a drainage ditch infested with mixed annual and perennial broadleaf and grass weeds at 25 pounds of active ingredient per acre in 150 gallons of water, give excellent control of vegetation. The ditch remains bare for an extended period.

These oil compositions are also useful for weed control in railroad yards and cattle yards. When diluted with 160 gallons of water per acre and sprayed from a railroad spray car at about 25 pounds of active ingredient per acre, weeds such as quack grass, crabgrass, Johnson grass, Bermuda grass, nutsedge, brome grass, ragweed, cocklebur, lamb's-quarter, and mare's-tail are controlled for an extended period.

Example 53

Composition I:	Percent
3-sec.-butyl-5-bromo-6-methyluracil	25
3,4-dichlorophenyl-1,1,3-trimethylurea	25
Ethylenediamine dodecyl benzene sulfonate	2
Synthetic fine silica	48

These ingredients are blended and micropulverized.

Composition II:	Percent
Diesel oil	97
Mixed polyoxyethylated sorbitan tall oil ester and oil-soluble petroleum sulfonates	3

Just before use, Composition I is mixed with Composition II to form an emulsifiable dispersion containing 30% insoluble solids.

This oil dispersion controls weeds along railroad rights-of-way. An application of 12 pounds (active) in 80 to 120 gallons of water gives rapid initial kill of existing vegetation such as quack grass, goldenrod, orchard grass, bluegrass, evening primrose, and yarrow. Extended weed control is obtained with this application.

Example 54

	Percent
3-isopropyl-5-bromo-6-methyluracil	25
2-methoxy-4,6-bis(isopropylamino)-s-triazine	50
Polyoxyethylated tert.-octyl phenol	3
Calcined montmorillonite clay	22

These components are blended and micropulverized.

The formulation is outstanding for the control of hard-to-kill perennial grasses. A spray rate of 20 pounds (active) in 80 gallons of Lion Herbicidal Oil No. 6 gives excellent control of panic grass, broomsedge, Johnson grass, and vasey grass growing along oil pipeline rights-of-way.

Example 55

	Percent
3-isopropyl-5-chloro-6-methyluracil	40
2,4,5-trichlorophenoxyacetic acid propylene glycol butyl ether ester	10
Mixed polyoxyethylated sorbitan monooleate and ethylenediamine dodecyl benzene sulfonate	5
Synthetic fine silica	45

These ingredients are blended, micropulverized, and reblended.

This oil dispersible powder is used to maintain weed-free areas around electric power poles by dispersing it in Lion Herbicidal Oil No. 6 and spraying it. A weed infestation of blackberry, honeysuckle, goldenrod, speedwell, poison ivy, pokeweed, corn cockle, crabgrass and panic grass is controlled by use of 15 pounds (active) per acre of this formulation in 80 gallons of oil.

Example 56

	Percent
3-isopropyl-5-bromo-6-methyluracil	25
3-isopropyl-1-trichloromethylthio-5-bromo-6-methyluracil	50
Mixed petroleum sulfonates and polyoxyethylene tall oil esters	5
Attapulgite clay	17
Synthetic fine silica	3

These ingredients are blended and micropulverized to give an oil-dispersible powder.

This composition is extended with 100 gallons of diesel oil and sprayed at the rate of 16 pounds of active ingredient per acre. It gives excellent control of horsetail, broomsedge, Johnson grass, and quack grass along guard rails and along railroad rights-of-way.

TANK MIXES

Example 57

Ten pounds of 3-sec.-butyl-5-bromo-6-methyluracil as an 80% wettable powder and 2 pounds in 4,6-dinitro-orthosecondary butylphenol in 4 gallons of oil are blended as a tank mix and applied at 12 pounds of active herbicide per acre in 100 gallons of water to weeds growing along fence rows. Quick kill of annual and perennial broadleaf and grass weeds is obtained, with excellent residual weed control.

Example 58

Twelve pounds of an 80% water-dispersible powder formulation of 3-isopropyl-5-bromo-6-methyluracil and 24 pounds of 85% 2,2-dichloropropionic acid, sodium salt are dispersed and mixed in 100 gallons of water.

This composition is good for the control of perennial grasses and broadleaf weeds on railroad rights-of-way. An application of 100 gallons of this formulation per acre give good control of Johnson grass, Bermuda grass, nutsedge, crabgrass, plantain, ragweed, and beggar-tick.

Example 59

Nineteen pounds of an 80% water-dispersible powder formulation of 3-isopropyl-5-bromo-6-methyluracil or 3-sec.-butyl-5-bromo-6-methyluracil and 25 pounds of 2,2-dichloropropionic acid, sodium salt (85%) are dispersed and mixed in 50 to 100 gallons of water. This composition, sprayed on one acre, gives good control of both annual and perennial grasses and broadleaf weeds growing along railroad rights-of-way and around loading dock installations.

Such difficult-to-kill weeds as crabgrass, Bermuda grass, Johnson grass, Vasey grass, nutsedge, dock, ragweed, lamb's-quarters, pigweed, goatweed, carpetweed, beggar-tick, Spanish needle, nightshade, black medic, knotweed, plantain, spotted spurge and velvetleaf are controlled by this application.

Example 60

Ten pounds of 3-isopropyl-5-chloro-6-methyluracil as an 80% wettable powder and 35 pounds of ammonium sulfamate are blended as a tank mix.

An application of 45 pounds per acre (active) in 100 gallons of water controls annual and perennial broadleaf and grass weeds growing around oil tanks and along roadsides, giving rapid contact action and extended residual weed control. Such weeds as crabgrass, broomsedge, cocklebur, flower-of-an-hour, and oak, maple, and sweet gum seedlings are controlled by this treatment.

SOLID COMPOSITIONS

DUSTS

Example 61

	Percent
5-bromo-6-methyl-3-(3,4-xylyl)uracil	90.0
Alkyl naphthalene sulfonate, Na salt	2.0
Low viscosity methyl cellulose	0.3
Attapulgite clay	7.7

These components are blended and micropulzerized until the particles of uracil have been reduced to about 10 microns in diameters, then reblended.

A similar high strength composition can also be made with 5-bromo-6-methyl-3-m-tolyluracil, 5-bromo-6-methyl-3-p-tolyluracil, or 5-bromo-6-methyl-3-(α,α,α-trifluoro-m-tolyl)uracil.

These formulations give excellent weed control when applied pre-emergence or post-emergence at rates of 4

pounds per acre to crabgrass, wild oats, wild mustard, volunteer alfalfa, foxtail and lamb's quarters.

GRANULES

Example 62

	Percent
3-sec.-butyl-5-bromo-6-methyluracil, Na salt	25
Granular 8–15 mesh attapulgite clay	75

A granular composition is prepared by dissolving the active ingredient in water and spraying this solution on the attapulgite granules while they are tumbled. The resulting granules are then dried.

The granules are applied by hand for "spot treatment" of undesirable bunch grasses growing in agricultural areas. An application of 20 to 30 pounds of active ingredient per acre gives good control of Dallis and Vasey grass.

Example 63

	Percent
3-cyclohexyl-5-bromo-6-methyluracil	40
Anhydrous sodium sulfate	10
Non-swelling sub-bentonite clay	50

These components are blended and micropulverized then moistened with water and granulated. The granules are then dried and screened.

These granules are applied by hand or by special spreaders at 10 to 25 pounds of active ingredient per acre. They control peppergrass, chickweed, crabgrass, pigweed, smartweed, barnyard grass, goose grass, and quack grass on industrial sites, along boundary fences and railroad rights-of-way, in parking areas, along roadsides, and under billboards.

Example 64

	Percent
3-isopropyl-5-chloro-6-methyluracil, Na salt	25
Granular 8–15 mesh vermiculite	75

The granules are prepared by dissolving the active ingredient in water, spraying the solution on tumbling vermiculite granules, and then drying.

This formulation is used as a "spot treatment" for areas infested with Russian knapweed, bindweed, and deep-rooted perennial weeds. It is applied at 3 pounds per 1000 square feet with a specially adapted spreader.

Example 65

	Percent
3-isopropyl-5-bromo-6-methyluracil	40
Anhydrous sodium sulfate	10
Non-swelling Ca, Mg bentonite	49
Alkyl naphthalene sulfonate, Na salt	1

These components are formulated as 4 to 8 mesh granules by blending and grinding the components, then moist-granulating them, followed by drying and screening.

The granules are broadcast at a level of 10 pounds of active ingredients per acre for the excellent control of oak brush growing on light sandy soil.

Example 66

	Percent
3-allyl-5-chloro-6-methyluracil	20
Attapulgite clay	78
Alkyl naphthalene sulfonic acid, Na salt	1
Lignin sulfonic acid, Na salt	1

These 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 a rotary crusher into irregular granules.

3-isopropyl-5-bromo-6-methyluracil is also prepared as a granule in this manner.

Example 67

The procedure of Example 66 is used with the following ingredients to give granules of high density.

	Percent
3-isopropyl-5-chloro-6-butyluracil	12
Sand (20–30 mesh)	81
Sodium silicate (28% SiO_2; ratio SiO_2/Na_2O=3.25)	7

Example 68 (fast release)

	Percent
3-amyl-5-chloro-6-methyluracil, sodium salt	20
10–20 mesh granular, expanded vermiculite	80

The active ingredient is dissolved in water, and the solution is then sprayed on the vermiculite while it is tumbled in a blender. The product is then dried.

Example 69

This product is prepared in the same manner as in Example 68, using the following ingredients:

	Percent
3-sec.-butyl-5-chloro-6-ethyluracil, Na salt	2
Granular 15–30 mesh Attaclay	98

Example 70

	Percent
3-phenyl-5-chloro-6-methyluracil, sodium salt	25
8–20 mesh expanded vermiculite	75

These granules are prepared by dissolving the active ingredient in water and spraying it on the vermiculite while keeping the vermiculite in motion to assure good distribution, and then drying the product.

Other active materials suitable for the preparation of this type of granule are:

3-benzyl-5-chloro-6-methyluracil, potassium salt
3-tert.-butyl-5-bromo-6-methyluracil, sodium salt
3-(1-ethylpropyl)-5-chloro-6-methyluracil, potassium salt
3-norbornyl-5-methoxy-6-methyluracil, sodium salt
3-butyl-5-bromo-6-methyluracil, 1/2 calcium salt

Example 71

The granular compositions of Examples 66, 67, 68, 69 and 70 are applied by hand or by specially built spreaders. At concentrations of 25 pounds of active ingredient per acre, they control broadleaf and grass weeds in lumber yards, along railroad rights-of-way, in fire lanes and around billboards, and in parking areas and roadsides.

They can be applied as soil treatments, at 20 pounds of active ingredient per acre, for the control of such woody plants as privet, elm, ash, oak, maple, and willow. This concentration also gives control of germinating annual weeds and established perennial weeds such as quack grass and plantain.

Example 72

	Percent
3-tert.-butyl-5-chloro-6-methyluracil	3.3
3-phenyl-1,1-dimethylurea	6.7
California Ca, Mg sub-bentonite	75.0
Anhydrous sodium sulfate	15.0

This composition is blended, micropulverized, pug-milled with about 20% water, moist-granulated and then dried. It is then screened to give 15–30 mesh granules.

Applied at 15 pounds of active ingredient per acre, these granules give excellent control of such weeds as bitterweed, partridge pea, beggar's-lice, ragweed, crabgrass, cheat, and common lespedeza, growing along guard rails and around sign boards.

Example 73

	Percent
3-cyclohexyl-5-bromo-6-methyluracil	15
2-chloro-4,6-bis(ethylamino)-s-triazine	15
Sodium lignin sulfonate	10
Swollen corn starch	4
Water	56

The active components, the sodium lignin sulfonate, and sufficient water to make a 50% slurry are first ball-milled

until the particles are less than 10 microns in size. The starch is then swelled with heat in the balance of the water and mixed with the dispersion.

Four parts of this dispersion are then sprayed on 26 parts of tumbling granular attaclay and dried to give granules containing 4% active ingredient.

A spring application of 2 pounds (active) per acre of these 4% granules gives excellent pre-emergence control of such annual weeds as wild carrot, crabgrass, goldenrod, lamb's-quarters, ragweed, and dog-fennel in asparagus.

Example 74

	Percent
3-sec.-butyl-5-bromo-6-methyluracil	3.75
2,3,6-trichlorobenzoic acid, Na salt	11.25
Attaclay, 15–30 mesh	85.00

The trichlorobenzoic acid salt is dissolved in water. Finely ground uracil is then suspended in this solution, with vigorous agitation. The mix is sprayed on tumbling attaclay and dried.

These granules are applied at the rate of 20 pounds (active) per acre for the control of field bindweed, crabgrass, ragweed, bluegrass, and pigweed growing in lumber yards.

As little as 10 pounds (active) per acre gives control of such annual grasses as crabgrass and foxtail and controls a heavy infestation of Virginia creeper.

Example 75

	Percent
3-isopropyl-5-chloro-6-methyluracil	2.0
Sodium chlorate	38.4
Sodium borate	59.6

A mixture of the crystalline chlorate and borate is placed in a blender. Finely ground uracil is slurried in water and sprayed on the chlorate-borate mixture while it is blended.

This formulation is effective against grass and broadleaf weed infestations, and can be easily applied on railroad rights-of-way. An application of 400 pounds per acre of these granules gives outstanding control of little bluestem, broomsedge, foxtail, crabgrass, ragweed, pigweed, henbit, and knotweed.

Example 76

	Percent
3-sec.-butyl-5-chloro-6-methyluracil	5.0
3-sec.-butyl-5,6-trimethyleneuracil	10.0
Na_2SO_4, anhydrous	10.0
Sodium lignin sulfonate	10.0
Mississippi sub-bentonite	32.5
Kaolin clay	32.5

These ingredients are blended and micropulverized until substantially all particles are less than 50 microns in size. The blend is then pug-milled with 15–20% water, moist-granulated, dried, and screened to 8–30 mesh.

When this formulation is applied at the rate of 6 pounds (active) per acre, excellent control of a wide variety of annual weeds is obtained.

Example 77

	Percent
3-isopropyl-5-nitro-6-methyluracil	1
Granular 15–30 mesh attapulgite	99

The uracil is dissolved in acetone and sprayed as a fine fog on tumbling granular attaclay. The solvent is then evaporated.

This granular formulation is applied at the rate of 120 pounds per acre. Excellent pre-emergence control of germinating weeds is obtained in a newly-planted field of peanuts. Carpetweed, foxtail, crabgrass, and lamb's-quarters are controlled with no visible injury to the crop.

Example 78

	Percent
3,5-di-(sec.-butyl)-6-methyluracil	25
Alkyl naphthalene sulfonic acid, sodium salt	1
Lignin sulfonic acid, sodium salt	1
Attapulgite clay	73

These ingredients are blended and then charged to a grinding mill where the are ground and moist granulated. The resulting granules are then dried and screened.

The following uracils can be formulated in the same fashion:

3-sec.-butyl-5-thicyano-6-methyluracil
3-cyclohexyl-5-carboxymethylthiomethyl-6-methyluracil
3-(p-bromophenyl)-5-bromo-6-methyluracil
3-(o-nitrophenyl)-5-bromo-6-methyluracil
3-(2,5-dichlorophenyl)-5-bromo-6-methyluracil
3-isopropyl-5-butoxymethyl-5-methyluracil
3-propynyl-5-methyl-6-butyluracil
3-(tert.-butylphenyl)-5-bromo-6-butyluracil
3-(3,4-dimethylbenzyl)-5-(2-hydroxyproyl)-6-methyluracil
3-(4-isopropylcyclohexyl)-5-chloro-6-methyluracil
3-(4-chlorocyclohexyl)-5,6-dimethyluracil
3-(4-bromocyclohexyl)-5,6-dibromouracil
3-(decahydro-1,4-5,8-dimethanonaphthyl)-5-hydroxymethyl-6-methyluracil
3-(3-methoxycyclohexyl)-5-hydroxymethyl-6-methyluracil
3-(4-chlorocyclohexylmethyl)-5,6-dichlorouracil
3-(decahydro-1,4-5,8-dimethanonaphthylmethyl)-5-hydroxymethyl-6-methyluracil

These compositions control cheat and wild oats growing along roadsides when applied pre-emergence at 12–15 pounds (active) per acre.

PELLETS

Example 79

	Percent
3-isopropyl-5-chloro-6-methyluracil	25
Anhydrous sodium sulfate	10
Sodium lignin sulfonate	10
Ca, Mg. bentonite	55

These components are blended and micropulverized, then moistened with 18–20% water and extruded through die holes. The extusions are cut into pellets and then dried.

These pellets are useful for weed control along highway guard rails, around bridges, cyclone fences, and highway signs. They are applied by hand, at 10 to 25 pounds of active ingredient per acre. Excellent control of such woody plants as oak, maple, sweet gum, and willow is obtained.

Example 80

	Percent
3-phenyl-5-bromo-6-methyl-2-thiouracil	25
Allkyl naphthalene sulfonic acid, Na salt	1
Anhydrous sodium sulfate	10
Non-swelling montmorillonoid type clay (Pikes Peak clay)	64

These components are blended and micropulverized, then wetted with 18–25% water and extruded through a die. The extrusions are cut into ⅛ inch pellets and then dried.

This formulation is used for dry application to the soil for control of undesirable woody plants in fence rows and utility rights-of-way. A basal application of one tablespoonful on the ground at the base of each brush cluster gives excellent control of birch, box elder, wild cherry, privet, willow, dogwood, oak, sweetgum, and poplar.

Example 81

	Percent
3-butyl-5-bromo-6-methyluracil	25
Alkyl naphthalene sulfonic acid, Na salt	1
Anhydrous sodium sulfate	10
Non-swelling clay	64

These ingredients are 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 82

	Parts
3-ethyl-5-bromo-6-methyluracil	190
Water	280
Hide glue (20% aqueous)	65
Glass frits (20–30 mesh)	750

The active ingredient is first made into an aqueous suspension by ball milling it with water and hide glue. This aqueous suspension is then sprayed onto the glass frits with continuous agitation in a ribbon blender. When complete coverage is obtained, the frits are removed and dried in a tunnel drier.

Example 83 (fast release)

	Percent
3-allyl-5-fluoro-6-methyluracil	25
Non-swelling Ca, Mg. bentonite	75

The above mixture is blended and micropulverized. It is then moistened with 20–30% water and extruded through an orifice. The extrusions are cut into short lengths and dried.

Example 84

	Percent
3-butyl-5-chloro-6-methyluracil	12
Polyvinyl alcohol (low viscosity)	2
Water	16
Prilled 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 prilled 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 adheres firmly. These pellets have a moderately rapid rate of release.

3 - isopropyl - 5-bromo-6-methyluracil and 3-butyl-5-bromo-6-methyluracial can also be formulated as pellets in this manner.

Example 85

	Percent
3-sec.-amyl-5-chloro-6-methyluracil	25.0
Anhydrous sodium sulfate	10.0
Alkyl naphthalene sulfonic acid, Na salt	1.0
Non-swelling Ca, Mg bentonite	64.0

This mixture is prepared as an extruded pellet by first blending and grinding the ingredients in a micropulverizer, moistening the mixture with 18–22% water and extruding it through a die. The extrusions are cut into short lengths at the die face and then dried.

Other active compounds suitable for formulation in a like manner are:

3-cyclopentenyl-5,6-dimethyluracil
3-isopropyl-5-chloro-6-methyluracil
3-(1-methylbutyl)-5-bromo-6-methyluracil
3-(sec.-butyl)-5-bromo-6-methyluracil
3-norbornenyl-5,6-dimethyluracil
3-decahydronaphthyl-5-hydroxymethyl-6-methyluracil

Example 86

The pellet compositions of Examples 81, 82, 83, 84 and 85 are applied by hand methods at 25 pounds of active ingredient per acre for control of annual and perennial weeds along guard rails, safety fences, and highway markers and dividers.

Example 87

The pellet compositions of Examples 82, 83, 84 and 85, when applied by airplane at concentrations of 12 pounds of active material per acre, give effective control of post-oak, blackjack oak, and winged elm brush.

Example 88

	Percent
3-isopropyl-5-chloro-6-methyluracil	6.67
3-phenyl-1,1-dimethylurea	13.33
Alkyl naphthalene sulfonic acid, Na salt	1.00
Sodium sulfate, anhydrous	10.00
California sub-bentonite	69.00

These ingredients are blended and micropulverized until the particles are substantially all below 50 microns in size. The blend is then pug-milled with 15–20% water and extruded through ⅛"–$\frac{9}{64}$" holes. The extrusions are cut into ⅛ inch lengths and dried.

These pellets are applied to the soil so that one tablespoonful covers an area of ½ to 1 square foot. Such woody plant species as hickory, persimmon, sumac, white and red oak, grape, winged and slippery elm, Osage orange, and wild cherry are controlled for a 6 to 12 month period after treatment.

Example 89

	Percent
3-isopropyl-5-bromo-6-methyluracil	14.25
2,3,5,6-tetrachlorobenzoic acid, Na salt	5.75
Anhydrous sodium sulfate	10.00
California sub-bentonite	70.00

These ingredients are processed as in Example 88.

This formulation controls plant colonies containing perennial grasses and woody vines. A spot treatment by hand at 15 pounds (active) per acre to young growing foliage gives excellent control of trumpet vine, Virginia creeper, panic grass, broomsedge, oak seedlings, and sweet gum seedlings.

Example 90

	Percent
3-isopropyl-5-bromo-6-methyluracil	2.5
3-isopropyl-5-bromouracil	2.5
Sodium sulfate, anhydrous	10.0
Sodium lignin sulfonate	10.0
Mississippi sub-bentonite	37.5
Kaolin clay	37.5

These ingredients are blended and micropulverized until substantially all particles are below 50 microns in size. The blend is then pug-milled with 15–18% water and extruded through ⅛" diameter holes. A high-speed wire blade cuts the extrusions into slices $\frac{1}{32}$"–$\frac{1}{16}$" thick.

These pellets are applied at 12 pounds (active) per acre to young foliage, to give excellent control of annual weeds growing in fire control lanes. Crab grass, foxtail, ragweed, pigweed, and lamb's-quarters are controlled with no regrowth for several months.

Example 91

	Percent
5-bromo-6-methyl-3-(2-norbornylmethyl)uracil (exo-endo mixture)	25
Low viscosity methyl cellulose	3
Kaolin clay	72

These ingredients are mixed and micropulverized, then pug-milled with 16–20% water and extruded through a $\frac{9}{64}$ die hole to form rods which are cut as formed by a revolving knife into ⅛ inch lengths. The resulting pellets are then dried.

Sixty pounds per acre of this formulation gives excellent control of ragweed, volunteer ryegrass, wild mustard, plantain, carpet weed, crab grass, foxtail, cocklebur, and purslane growing around signposts and telephone poles.

3-(3a,4,5,6,7,7a-hexahydro-4,7-methano-5-indanyl) - 5-

bromo-6-methyluracil can be substituted in this formulation and used as described with equivalent results.

Example 92

	Percent
3-sec.-butyl-5-bromo-6-methoxyuracil	15
Low viscosity methyl cellulose	3
Kaolin clay	82

The components are blended, micropulverized, mixed with approximately 20% water, extruded, cut into pellets and dried.

The following substituted uracils can also be formulated in this way:

3-cyclohexyl-5-methylthiomethyl-6-methyluracil
3-(p-methoxyphenyl)-5-bromo-6-methyluracil
3-(p-fluorophenyl)-5-bromo-6-methyluracil
3-cyclohexyl-5-methylthio-6-methyluracil
3-isopropyl-5-phenylthiomethyl-6-methyluracil
3-cyclohexyl-5-isopropoxymethyl-6-methyluracil
3-(sec.-butyl)-5-bromo-6-ethoxyuracil
3-isopropyl-5-isopropyl-6-methyluracil
3-isopropyl-5-isopropoxy-6-methyluracil
3-(5-nitro-2-furfuryl)-5-butylthio-6-isopropyluracil
3-cyclopentyl-5-bromoethyl-6-methyluracil
3-cyclooctyl-5-fluoromethyl-6-methyluracil
3-isopropyl-5-butenyl-6-methyluracil
3-cyclohexyl-5-chloro-6-(1-bromopropyl)uracil

These formulations control weeds around highway guardrails. A hand application of 12–16 pounds (active) per acre gives good control of wild mustard, lamb's-quarters, velvetleaf, and chickweed.

SOLUBLE POWDERS

Example 93

	Percent
3-isopropyl-5-chloro-6-methyluracil, sodium salt	90
Synthetic fine silica	10

These ingredients are blended and micropulverized to give a free-flowing, noncaking powder. When added to a spray tank, the active ingredient dissolves rapidly, leaving the silica in suspension.

This composition is used for the control of mixtures of annual and perennial weeds growing around farm buildings. An application of 15 pounds of active material per acre in 60 gallons of water gives excellent control of pepper grass, wild mustard, burdock, quack grass, and crabgrass.

Example 94

	Percent
3-sec.-butyl-5-bromo-6-methyluracil, sodium salt	90
Diatomaceous silica	10

A noncaking, soluble powder is prepared by blending and micropulverizing the ingredients. When the powder is placed in water in a spray tank, the soluble active material dissolves, leaving the silica in suspension.

Other soluble uracil salts which can be formulated in the same manner are:

3-sec.-butyl-5-nitro-6-methyluracil, sodium salt
3-phenyl-5-bromo-6-ethyluracil, potassium salt
3-ethyl-5-thiocyano-6-propyl-2-thiouracil, lithium salt
3-isopropyl-5-methoxymethyl-6-chlorouracil, sodium salt
3-propyl-5-fluoro-6-methyluracil, potassium salt

These soluble salt compositions are used for control of mixed annual and perennial vegetation growing around oil tank installations. Concentrations of 20 pounds of active ingredient per acre in 80 gallons of water give excellent weed control for an extended period.

Example 95

This formulation is prepared by blending, micropulverizing and then reblending the following ingredients:

	Percent
3-isopropyl-5-bromo-6-methyluracil	20
Sodium metaborate	40
Dioctylsodium sulfosuccinate	30
Sodium lignin sulfonate	2
Finely-divided synthetic silica	8

The active ingredient in this formulation is soluble in water when the formulation is added to water to make a 4% concentration.

One hundred gallons of a 4% concentration of this formulation are applied to an area of one acre in a sawmill yard infested with annual weeds. Outstanding control of crabgrass, foxtail, ragweed, barnyard grass, bromegrass and pigweed is obtained.

Example 96

	Percent
3-isopropyl-5-bromo-6-methyluracil	50
Na_3PO_4, anhydrous	45
Alkyl aryl sulfonate, sodium salt	1
Sodium lignin sulfonate	1
Finely-divided synthetic silica	3

These ingredients are blended, micropulverized and reblended. Na_3PO_4 can be replaced entirely or in part by K_3PO_4, Na_2SiO_3, K_2SiO_3, Na_2CO_3, K_2CO_3, $NaBO_2$, Na_2O, K_2O, NaOH or KOH, or a combination of these.

An application of a solution of 8 pounds (active) in 80 gallons of water to a one-acre railroad yard controls cocklebur, velvetleaf, lamb's-quarters, purslane, pigweed, bromegrass, ryegrass, crabgrass, barnyard grass and foxtail.

Example 97

	Percent
3-sec.-butyl-5-bromo-6-methyluracil	50
Na_2SiO_3, anhydrous	21
K_2CO_3, anhydrous	21
Dioctyl sodium sulfosuccinate	2
Sodium lignin sulfonate	5
Finely-divided synthetic silica	1

These ingredients are blended, micropulverized and reblended. An application of 10 pounds (active) in 80 gallons of water per acre controls crabgrass, foxtail, water grass, Indian grass, goldenrod, asters and ragweed on an industrial site.

A directed pre-emergence spray of one pound (active) in 40 gallons of water controls crabgrass, water grass, foxtail, mustard, pigweed and lamb's-quarters in ratoon sugarcane 12 inches high.

WETTABLE POWDERS

Example 98

	Percent
3-sec.-butyl-5-chloro-6-methyluracil, Ca salt	80.0
Sodium lauryl sulfate	0.6
Sodium lignin sulfonate	2.0
Kaolin clay	17.4

These components are blended and micropulverized until the solids are substantially all below 50 microns in particle size. The mixture is then reblended until it is homogeneous.

This wettable powder is used as a general purpose weed killer on industrial sites and railroad ballast. Ten to twenty pounds of active ingredient per acre in 100 gallons of water gives excellent control of goldenrod, evening primrose, pokeweed, oxeye daisy, cocklebur, goose grass, crabgrass, and love grass.

Twenty pounds of active ingredient per acre in 50 gallons water gives excellent control of nutsedge.

Example 99

	Percent
3-cyclohexyl-5-chloro-6-methyluracil	80.0
Dioctyl sodium sulfosuccinate concreted with sodium benzoate (Aerosol OTB)	2.0
Partially desulfonated sodium lignin sulfonate	1.0
Calcined, non-swelling montmorillonite type clay (Pikes Peak clay)	17.0

This powder is prepared as in Example 98, but is also passed through an air attrition mill until substantially all the particles are below 10 microns in size.

This composition is used for pre-emergence application in such agricultural crops as sugar cane, asparagus, and safflower. The powder is dispersed in 40 gallons of water and sprayed with a pressure sprayer. One-half to two pounds of active ingredient per acre gives excellent control of pigweed, lamb's-quarters, purslane, mustard, crabgrass, foxtail, and water grass.

Example 100

	Percent
3-butyl-5,6-dimethyluracil	80.0
Sodium lauryl sulfate	0.6
Partially desulfonated sodium lignin sulfonate	1.0
Calcined, non-swelling montmorillonite type clay (Pikes Peak clay)	18.4

A wettable powder is prepared by blending the components and then micropulverizing them until substantially all the particles are below 50 microns in size.

This formulation is used at 20 pounds of active ingredient per acre in 100 gallons of water for weed control around oil tank installations and on railroad ballast. Excellent control of quack grass, crabgrass, goose grass, Johnson grass, bitterweed, oxeye daisy, mare's-tail, maple, oak, and willow brush is obtained.

At 30 pounds of active ingredient per acre as a spot treatment, excellent control of such deep-rooted weeds as bindweed and Canada thistle is obtained.

3-tert.-butyl-5-bromo-6-methyluracil and 3-(1-ethyl-1-methylpropyl)-5-chloro-6-methyluracil can also be used in this composition with excellent results.

Example 101

	Percent
3-sec.-butyl-5-chloro-6-methyluracil	80.00
Alkyl naphthalene sulfonic acid, Na salt	1.75
Low viscosity methylcellulose	0.25
Kaolin	18.00

This wettable powder is prepared in the same manner as in Example 100. It is used for the control of annual broadleaf and grass weeds growing around telephone poles and highway markers. An application of 5 pounds of active ingredient per acre in 40 gallons of water gives excellent control of crabgrass, water grass, black-eyed Susan, carpetweed, and chickweed.

Example 102

	Percent
3-(m-chlorophenyl)-5-chloro-6-methyluracil	50
Kaolin clay	48
Dioctyl ester of sodium sulfosuccinic acid	1
Sodium lignin sulfonate	1

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

Example 103

	Percent
3-butyl-5-chloro-6-methyluracil	80.00
Attapulgite clay	17.25
Alkyl naphthalene sulfonic acid, Na salt	2.00
Low viscosity methylcellulose	0.25
Dioctyl sodium sulfosuccinate	0.50

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

3-isopropyl-5-bromo-6-methyluracil and 3-tert.-butyl-5-bromo-6-methyluracil can also be formulated as wettable powders in this manner.

This formulation, at a rate of one pound (active) in 30 gallons of water, is applied as a directed spray to newly emerged grasses and broadleaf weeds growing in safflower. Wild mustard, millet, barnyard grass and crabgrass are killed.

Four to seven pounds (active) per acre of these formulations in 30 gallons of water give effective control of quack grass along fence rows.

At concentrations of 12 pounds of active ingredient per acre, these compositions give excellent control of annual and perennial weeds growing on railroad rights-of-way and industrial sites.

Example 104

	Percent
3-sec.-butyl-6-methyl-5-thiocyanouracil	80.0
Dioctyl sodium sulfosuccinate concreted with sodium benzoate	0.4
Ditertiary acetylenic glycol	0.5
Oleylester of sodium isethionate	1.0
Attapulgite clay	18.1

A wettable powder is prepared in the manner described for Example 103.

Example 105

	Percent
3-butyl-5-chloro-6-methyluracil, 1/2 Ca salt	80
Dodecylbenzene sulfonic acid, Na salt	2
Sodium lignin sulfonate	1
Attapulgite clay	17

The powder is also prepared in the manner of Example 103.

Other insoluble or sparingly soluble salts suitable for formulation in this fashion are:

3-phenyl-5-chloro-6-methyl-2-thiouracil, 1/2 Ca salt
3-butyl-5-bromo-6-methyl-2-thiouracil, 1/2 Mg salt
3-phenyl-5-bromo-6-ethyluracil, 1/2 Ca salt
3-sec.-butyl-5-cyano-6-methyluracil, 1/2 Ca salt
3-(4-methoxycyclohex-3-enemethyl)-5,6-dimethyluracil
3-[1-methyl-2-(cyclopenten-1-yl)ethyl]-5-methoxymethyl-6-methyluracil

Example 106

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

	Percent
3-phenyl-5-chloro-6-methyluracil	50.00
Synthetic precipitated hydrated silicon dioxide	45.75
Low viscosity methylcellulose	0.25
Alkyl naphthalene sulfonic acid, Na salt	2.00
Polyoxyethylene esters of mixed fatty and rosin acids	2.00

Example 107

	Percent
3-cyclohexyl-5-methoxymethyl-6-methyluracil	50.0
Alkyl naphthalene sulfonic acid, sodium salt	1.0
Sodium salt of polymerized alkyl naphthalene sulfonic acid	0.5
Calcined, non-swelling Montmorillonite type clay (Pikes Peak clay)	48.5

A wettable powder is prepared in the manner described for Example 106.

Other active materials which can be formulated into wettable powders in the same fashion are:

3,5-diallyl-6-methyluracil
3-cyclohexyl-5-bromo-6-chlorouracil
3-(1,1-dimethylpropyl)-5-bromo-6-methyluracil
3-bornyl-5-chloro-6-methyluracil
3-cyclohexyl-5-iodo-6-methyluracil
3-(o-methylcyclohexyl)-5-bromo-6-methyluracil
3-cyclopropyl-5-bromo-6-methyluracil
3-cyclobutyl-5-bromo-6-methyluracil
3-cyclobutenyl-5-bromo-6-methyluracil
3-cyclopropylmethyl-5-bromo-6-methyluracil
3-cyclobutenylmethyl-5-bromo-6-methyluracil

Example 108

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

	Percent
3-norbornyl-5-chloro-6-methyluracil	25
Kaolin clay	70
Dioctyl ester of sodium sulfosuccinic acid	1
Sodium lignin sulfonate	4

Example 109

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

	Percent
3-butyl-5-bromo-6-methyl-2-thiouracil	50
Diatomite	46
Polymerized sodium salts of alkyl naphthalene sulfonic 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 110

	Percent
3-isopropyl-5-fluoro-6-methyluracil	60
Synthetic calcium silicate	38
Polyvinyl alcohol (low viscosity)	1
Alkyl naphthalene sulfonic acid, Na salt	1

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

Example 111

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

	Percent
3-sec.-butyl-6-methyl-5-nitrouracil	60.00
Attapulgite clay	38.90
Dioctyl ester of sodium sulfosuccinic acid	0.40
Low viscosity methylcellulose	0.30
Aliphatic substituted butyndiols and octyndiols	0.40

The blended mixture is then 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 112

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

	Percent
3-cyclohexylmethyl-5-chloro-6-methyluracil	50
Kaolin clay	46
Dioctyl ester of sodium sulfosuccinic acid	1
Sodium lignin sulfonate	3

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

Example 113

Any one of the wettable powder compositions of Examples 103–112 is dispersed in 150 gallons of water and sprayed from a pressure sprayer. When applied at 16 pounds of active ingredient per acre as a foliar spray, excellent control of quack grass, Bermuda grass, Johnson grass, water grass, foxtail, and crabgrass is obtained.

Example 114

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

	Percent
3-tert.-butyl-5-ethyl-6-methyluracil	70.00
Attapulgite clay	28.25
Alkyl naphthalene sulfonic acid, Na salt	1.50
Low viscosity methylcellulose	0.25

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

Example 115

	Percent
3-isopropyl-5-bromo-6-methyluracil	80.0
Sodium lauryl sulfate	0.6
Sodium lignin sulfate	2.0
Kaolin clay	17.4

These components are blended and micropulverized until the solids are substantially all below 50 microns in particle size. This mixture is then reblended until it is homogeneous.

3-sec.-butyl-5-chloro-6-methyluracil and 3-sec.-butyl-5-bromo-6-methyluracil can be similarly formulated.

These wettable powders are used for preemergence application in sugar cane. The powders are dispersed in 40 gallons of water and applied with a tractor sprayer. One and one-half to two and one-half pounds of active ingredient per acre give excellent control of crab grass, water grass, seedling Johnson grass, giant foxtail, mustard, cocklebur, and ragweed, with a favorable safety factor.

The following uracils can also be formulated in this way. When used at the rate of ten pounds (active) in 40 gallons of water per acre, they give excellent control of the same weeds growing in an automobile junk yard:

3-cyclohexyl-5-hydroxymethyl-6-methyluracil
3-cyano-5,6-dimethyluracil
3-cyano-5-bromo-6-methyluracil
3-phenyl-5-bromo-6-isopropoxyuracil
3-sec.-butyl-5-bromo-6-sec.-amyloxyuracil

Example 116

	Percent
3-isopropyl-5-bromo-6-methyluracil	40
3-(3,4-dichlorophenyl)-1,1-dimethylurea	40
Alkyl aryl polyether alcohol 40%, extended on a silica powder	4
Substituted acetylenic glycol	1
Partially desulfonated sodium lignin sulfonate	2
Kaolin clay	10
Synthetic fine silica	3

This formulation is applied in 100 gallons of water, at 20 pounds of active ingredient per acre, for the control of finger grass, foxtail, love grass, nutgrass, Vasey grass, broomsedge, sand spur, goldenrod, ragweed, beggartick, and spurge growing around oil tanks. The formulation gives a good rapid kill of foliage and has long-lasting residual activity in the soil.

Two pounds (active) of this formulation is dispersed in 40 gallons of water. When applied pre-emergence or early post-emergence to the tender weeds, excellent control of annual grasses and broadleaf weeds growing in

sugar cane is obtained. A post-emergence spray, directed under the cane plants, gives good control of ricegrass, crabgrass, goose grass, seedling Johnson grass, Ageratum, pigweed, lamb's-quarters and velvetleaf.

Example 117

	Percent
3-isopropyl-5-chloro-6-methyluracil	32.00
2 - chloro - 4-ethylamino-6-isopropylamino-s-triazine	48.00
Alkyl naphthalene sulfonic acid	1.75
Methyl cellulose	0.25
Attapulgite clay	18.00

This formulation is applied in 100 gallons of water, at 15 pounds of active ingredient per acre, for the control of a wide variety of broadleaf and grass species growing along a railroad right-of-way. Excellent control of annual ryegrass, bromegrasses, common ragweed, henbit, speedwells, goldenrod, daisy, asters, and buckhorn plantain is obtained following a spring application to youth and growing weeds.

Example 118

	Percent
3-sec.-butyl-5-bromo-6-methyluracil	3
N-(m-chlorophenyl)carbamic acid, isopropyl ester	24
Sodium dodecyl benzene sulfonate	1
Sodium N-methyl-N-palmitoyl taurate	1
Attapulgite clay	71

This formulation is used for the pre-emergence control of germinating annual grass and broadleaf weeds in sugar cane. An application of 4½ pounds (active) per acre gives good control of crabgrass, foxtail, ricegrass, seedling Johnson grass, and pigwed, without injury to the cane.

Example 119

	Percent
3-sec.-butyl-5-chloro-6-methyluracil	9.0
Sodium dodecyl benzene sulfonate	0.1
Chlorate-borate mixture: 60% sodium chlorate–40% sodium metaborate	90.9

The chlorate-borate mix is charged into a drum. A slurry of 9 parts of uracil in 20 parts of water containing the sodium dodecyl benzene sulfonate is sprayed on the mix while it is tumbled. The mixture is then dried.

This mixture is useful for the control of broadleaf and hard-to-kill grass species. One hundred and ten pounds of this formulation, mixed in 300 gallons of water per acre, gives outstanding control of crabgrass, bromegrass, broomsedge, big and little bluestems, ragweed, primrose, goldenrod, and fall aster.

Example 120

	Percent
3-isopropyl-5-bromo-6-methyluracil	25
3-isopropyl-6-methyluracil	50
Oleyl ester of sodium isethionate	1
Alkylnaphthalene sulfonic acid, Na salt	1
Kaolin clay	20
Synthetic fine silica	3

This formulation is useful for the control of weeds along power lines. At rates of 15 to 25 pounds (active) per acre in 80 to 125 gallons of water, commercial control of crabgrass, foxtail, briars, ragweed, carpetweed, daisy, pigeon pea, Johnson grass, and Bermuda grass is obtained.

Example 121

	Percent
3-sec.-butyl-5-bromo-6-methyluracil	60
3-amino-1,2,4-triazole	20
Dioctyl sodium sulfosuccinate, 85–15 condensate with sodium benzoate	1
Substituted acetylenic glycol	1
Partially desulfonated sodium lignin sulfonate	2
Attapulgite clay	16

About 6 ounces of this formulation are mixed in 2 gallons of water and are sprayed on 1000 square feet of earth around incinerators and outdoor fire places in a park to kill poison ivy, quack grass, and a mixture of annual weeds.

Example 122

	Percent
3-isopropyl-5-bromo-6-methyluracil	40
Sodium dodecylbenzenesulfonate	20
Partially desulfonated sodium lignin sulfonate	1
Synthetic fine silica	39

These ingredients are blended and micropulverized until the uracil particles are below 50 microns in diameter, and are then reblended to obtain homogeneity.

This formulation is applied at the rate of 8 pounds per acre in 300 gallons of water to a population of actively-growing grasses. After one week, contact burn of foxtail, crabgrass, cheat, wild oats, ryegrass and seedling bromegrass is obtained. Extended residual control of these species is noted.

Example 123

	Percent
Technical 95% 3-isopropyl-5-bromo-6-methyluracil	85.5
Alkyl naphthalene sulfonic acid, Na salt	2.0
Calcium salt of partially desulfonated lignin sulfonic acid	0.5
Powdered gypsum ($CaSO_4 \cdot 2H_2O$)	2.0
Precipitated tricalcium phosphate	2.5
Attapulgite clay	7.5

These components are blended and micropulverized until the product shows less than one percent retention on a 325 mesh screen when wet screened.

This formulation, applied at the rate of 15 pounds (active) in 100 gallons of water, gives excellent control of broadleaved weeds such as plantain, danelion, dock, smooth brome, Indian grass, and broomsedge grow along railroad rights-of-way. Excellent residual weed control is obtained.

For a tank mix, five pounds of this powder are dispersed in 60 gallons of water to which are added 10 pounds of a polyoxyethylated alkyl mercaptan. One week after application, excellent contact burn is noted on annual blueglass, Kentucky bluegrass, seedling Johnson grass, crabgrass, ragweed, wild mustard and cocklebur growing along a fence row. Extended residual control of these species is obtained.

Example 124

	Percent
Technical 95% 3-sec.-butyl-5-bromo-6-methyluracil	85.5
Alkyl naphthalene sulfonic acid, Na salt	2.0
Calcium salt of partially desulfonated lignin sulfonic acid	0.5
Powdered gypsum ($CaSO_4 \cdot 2H_2O$)	2.0
Precipitated tricalcium phosphate	2.5
Attapulgite clay	7.5

These components are blended and micropulverized until the product shows less than one percent retention on a 325 mesh screen when wet screened.

Ten pounds of this formulation are dispersed in 100 gallons of herbicidal oil (e.g. Lion Herbicidal Oil No. 6). When this mixture is applied post-emergence to one acre around lumber yards, quick kill of the weed population is obtained, followed by extended residual weed control. Crabgrass, bluegrass, cinquefoil, goldenrod, Spanish needle, Bermuda grass, seedling Johnson grass and foxtail are controlled.

Example 125

	Percent
3-phenyl-5-bromo-6-methyluracil	80.0
Attapulgite clay	12.5
Tricalcium phosphate	3.0
Gypsum ($CaSO_4 \cdot 2H_2O$)	2.0
Calcium salt of partially desulfonated lignin sulfonic acid	0.5
Alkyl naphthalene sulfonic acid, Na salt	2.0

This wettable powder is prepared by blending and micropulverizing the mixture until the uracil particles are under 50 microns in diameter, then reblending to obtain homogeneity.

The formulation is applied pre-emergence at the rate of two pounds (active) per acre in 30 gallons of water to a field planted to flax. Excellent control of foxtail, crabgrass, barnyard grass, wild oats, wild mustard, ragweed, and nut grass germinating from tubers is obtained. The flax shows excellent growth and seed production.

Example 126

	Percent
3-phenyl-5-bromo-6-methyluracil	29.00
Sodium lignin sulfonate	15.00
Na_2HPO_4	.96
Tetramethylthiuram disulfide	.50
Hydrated attapulgite	1.75
Water	52.79

These ingredients are mixed and wet milled until the uracil particles are below ten microns in diameter.

This formulation is used as in Example 125, with equivalent results.

The formulations of Example 125 and 126 can be used for industrial weed control at rates of 15–20 pounds (active) per acre. A spring application gives excellent control of crabgrass, goose grass, foxtails, seedling broomsedge, goldenrod, fireweed and annual ragweed.

Example 127

	Percent
3-isopropyl-5-nitro-6-methyluracil	28.0
Sodium lignin sulfonate	15.0
Sodium pentachlorophenate	.7
Hydrated attapulgite	1.5
Water	54.8

These components are mixed and wet milled with sand until the particles of active material are below 10 microns in diameter.

When this formulation is applied pre-emergence at the rate of one pound (active) per acre in 30 gallons of water, excellent control of germinating weeds is obtained in a field planted to peanuts. Crabgrass, foxtail, barnyard grass, lamb's-quarters, cocklebur and chickweed are controlled. The peanuts grow vigorously without undue injury.

Example 128

	Percent
5-bromo-6-methyl-3-(2-norbornyl)uracil (exo-endo mixture)	80.0
Dioctyl sodium sulfosuccinate, concreted 85/15 with sodium benzoate	1.0
Sodium N-methyl-N-palmitoyltaurate	1.0
Synthetic fine calcium silicate	18.0

These components are blended, micropulverized until the particles of uracil have been reduced to about 10 microns in diameter, then reblended.

This formulation is applied at the rate of 15 pounds (active) per acre in 80 gallons water to a weed population growing under television broadcasting towers. Cheat, foxtail, crabgrass, quack grass, barnyard grass, dandelion, buckhorn, lamb's-quarters, lespedeza, and pigweed are controlled. Extended residual weed control is obtained.

Example 129

	Percent
5-bromo-3-isopropyl-6-methyluracil, 2:1 complex with phenol	75.0
Sodium lauryl sulfate	0.6
Sodium lignin sulfonate	2.0
Calcined montmorillonite clay (Pikes Peak clay)	22.4

These ingredients are blended and micropulverized until the complex particles are below 50 microns in diameter. The mixture is then reblended.

This wettable powder is useful for weed control on railroad rights-of-way, sidings, and yards. When diluted with 100 gallons of water and sprayed at the rate of 25 pounds of active ingredient per acre, quack grass, crabgrass, Bermuda grass, bromegrass, ragweed, cocklebur, and lamb's-quarters are controlled.

The following uracil complexes can be substituted, in equivalent amounts, for 5-bromo-3-isopropyl-6-methyluracil:

2:1 complex with phenol, with similar results:
1:2 complex phenol and 5-bromo-3-sec.-butyl-6-methyluracil
1:2 complex phenol and 5-bromo-3-tert.-butyl-6-methyluracil
1:1 complex pentachlorophenol and 5-bromo-3-sec.-butyl-6-methyluracil
1:1 complex pentachlorophenol and 5-bromo-3-tert.-butyl-6-methyluracil
1:1 complex p-chlorophenol and 5-bromo-3-isopropyl-6-methyluracil
1:1 complex p-methoxyphenol and 5-bromo-3-isopropyl-6-methyluracil

Example 130

A wettable powder is prepared by blending the following ingredients, followed by micropulverizing:

	Percent
5-bromo-3-isopropyl-6-methyluracil	84.0
Attapulgite clay	8.5
Tricalcium phosphate	3.0
Calcium sulfate, dihydrate	2.0
Calcium lignin sulfonate	0.5
Alkyl aryl sulfonate	2.0

The formulation is applied, at a rate of 0.2 to 0.4 pound (active) in 40 gallons of water, as a foliar spray to cheat grass growing in a field of dormant alfalfa. Effective weed control is obtained.

The formulation is also useful for controlling perennial grasses in citrus. An application of 7 to 10 pounds (active) per acre to young and actively growing Johnson grass and Burmuda grass in a citrus orchard gives effective control of these weeds during the growing season.

This wettable powder can also be used to control winter annual weeds in established plantings of strawberries and rasphberries. An application of ½ to 1 pound (active) per acre, directed to the actively growing weeds while the berry plantings are dormant, gives effective control of mustard, chickweed, groundsel, annual bluegrass and annual ryegrass.

The formulation also gives effective control of annual broadleaf and grass weeds in pineapple. An application of 1 to 2 pounds (active) per acre in 40 gallons of water, applied pre-emergence to weeds as an interspace application, gives effective control of crabgrass, goose grass, annual bluegrass, pigweed and lamb's-quarters.

Example 131

A wettable powder is prepared by blending the following components in a ribbon blender and micropulverizing them until the particles are below 50 microns in diameter. The mixture is then reblended until homogeneous.

	Percent
1:1 addition compound of 3-isopropyl-5-bromo-6-methyluracil with octylamine	25
Attapulgite clay	70
Dioctyl ester of sodium sulfosuccinic acid	1
Sodium lignin sulfonate	4

This wettable powder is dispersed in 60 gallons of water and applied at the rate of fifteen pounds of active ingredient per acre on an unsurfaced parking lot. Volunteer small grains, crabgrass, ryegrass, wild mustard, chickweed, and dandelion are controlled.

Example 132

	Percent
3-cyclohexyl-5-ethyl-6-methyluracil	45.00
Alkyl naphthalent sulfonic acid, sodium salt	1.50
Low viscosity methyl cellulose	0.25
Synthetic calcium silicate	53.25

These ingredients are blended and micropulverized until the particles are 50 microns or less in diameter. The mixture is then reblended.

The following uracils can be substituted, in equivalent amounts, for the 3-cyclohexyl-5-ethyl-6-methyluracil:

3-isopropyl-5-chloromethyl-6-methyluracil
3-(p-methylphenyl)-5-bromo-6-methyluracil
3-(3,4-dimethylphenyl)-5-bromo-6-methyluracil
3-(p-isopropylphenyl)-5-bromo-6-methyluracil
3-(2-methyl-3-chlorophenyl)-5-bromo-6-methyluracil
3-(2-methyl-3-isopropylphenyl)-5-bromo-6-methyluracil
3-(3-chloro-4-nitrophenyl)-5-bromo-6-methyluracil
3-(3-chloro-4-methoxyphenyl)-5-bromo-6-methyluracil
3-(p-chlorobenzyl)-5-bromo-6-methyluracil
3-(p-methylbenzyl)-5-bromo-6-methyluracil
3-(p-methoxycyclohexyl)-5-bromo-6-methyluracil
3-(4-methylcyclohexyl methyl)-5-bromo-6-methyluracil
3-(4-propylcyclohexyl methyl)-5-bromo-6-methyluracil
3-isopropyl-5-bromo-6-bromomethyluracil
3-isopropyl-5-chloro-6-chloromethyluracil

These formulations control annual broadleaf and grass weeds growing along boardwalks. An application of 10 pounds (active) per acre in 40 gallons of water, made on young vegetation, gives excellent control of crabgrass, foxtail, barnyard grass, chickweed, wild mustard and lamb's-quarters.

Example 133

	Percent
3-(inden-2-yl)-5-bromo-6-methyluracil	40
Dioctyl ester of sulfosuccinic acid, sodium salt	1
Lignin sulfonic acid, sodium salt	3
Kaolin clay	56

These ingredients are blended and micropulverized until the particles have average diameters of less than 50 microns. The ground product is then reblended.

The following uracils can be formulated in a similar fashion:

3-(1,2,3,4-tetrahydronaphth-5-yl)-5-bromo-6-methyluracil
3-(2-phenylethyl)-5-bromo-6-methyluracil
3-(p-nitrobenzyl)-5-bromo-6-methyluracil
3-(p-sec.-butoxybenzyl)-5-bromo-6-methyluracil
3-(1-naphthylmethyl)-5-bromo-6-methyluracil
3-(7-octenyl)-5-bromo-6-methyluracil
3-(cyano)-5-bromo-6-methyluracil
3-(sec.-butyl)-5-bromo-6-methoxyuracil
3-(sec.-butyl)-5,6-dibromouracil
3-cyclohexyl-5,6-dichlorouracil
3-methoxyethyl-5-bromo-6-methyluracil
3-hydroxyethyl-5,6-dimethyluracil
3-ethoxycarbonylmethyl-5-bromo-6-methyluracil
3-(3-chloro-1-methylpropyl)-5-chloro-6-methyluracil
3-(4-bromobutyl)-5-bromo-6-methyluracil
3-(2-cyanoethyl)-5-bromo-6-methyluracil
3-naphthyl-5-fluoro-6-methyluracil
3-pyridyl-5-chloro-6-methyluracil
3-(3,4-dibromophenyl)-5-hydroxymethyl-6-methyluracil
3-(3,4,5-trichlorophenyl)-5-hydroxymethyl-6-methyluracil
3-tetrahydronaphthyl-5-hydroxyethyl-6-methyluracil

These compositions control annual weeds growing on the edge of airport runway strips. A pre-emergence treatment of 16 pounds (active) per acre in 50 gallons of water gives excellent control of crabgrass, cheat, foxtail, wild mustard, and annual bluegrass.

Example 134

3-(1-ethylpropyl)-5-bromo-6-methyluracil is formulated as an 80% wettable powder according to the procedure set forth in Example 98.

An application of 1–2 pounds (active) of this formulation per acre in 40 gallons of water gives excellent control of ryegrass, crabgrass, barnyard grass, foxtail, goose grass, Johnson grass, pigweed, lamb's-quarters and wild mustard growing in a field of sugarcane. Seedling weeds in the 2-leaf stage are controlled.

Example 135

3-isopropyl-5-bromo-6-methyluracil is formulated as an 80% wettable powder according to the procedure described in Example 115.

An application of 2 pounds (active) of this formulation per acre in 40 gallons of water is made around and under peach trees in the fruit-producing stage growing in Keyport silt loam. The application is made in the spring when weeds are beginning vigorous growth. Good practical weed control is gained the first year.

One year later a second application is made to the same area with similar results.

These treatments control such weeds as bluegrass, crabgrass, foxtail, ryegrass, bromegrass, barnyard grass, pigweed, lamb's-quarters and chickweed.

Example 136

5-bromo-6-methyl-3-(2-norbornylmethyl)uracil is formulated as an 80%% wettable powder according to the procedure set forth in Example 128.

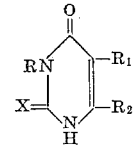
This formulation is applied pre-emergence at a rate of from 2–4 pounds (active) per acre in 40 gallons of water to newly-planted fields of cucumbers, watermelons and cantaloupe.

These treatments give excellent control of crabgrass, foxtail, barnyard grass, ryegrass, pigweed, lamb's-quarters, flower-of-an-hour and wild mustard.

The embodiments of the invention in which an exclusive property or privilege is claimed are:

1. A method for the control of undesirable vegetation comprising applying to a locus to be protected in an amount sufficient to exert herbicidal action a compound selected from the group consisting of

(a) compounds of the formula



where

R is selected from the group consisting of alkyl of 1 through 10 carbon atoms, substituted alkyl of 1 through 8 carbon atoms, wherein said substituent is selected from the group consisting of bromine, chlorine, hydroxy, alkoxy, alkoxycarbonyl, and cyano, aryl of 5 through 10 carbon atoms, substituted phenyl, wherein said substituent is selected from the group consisting of chlorine, bromine, fluorine, alkoxy of 1–5 carbon atoms, alkyl of 1–6 carbon atoms, nitro, trifluoromethyl, 1,2-tetramethylene, and 1,2-trimethylenylene, aralkyl of 5

through 13 carbon atoms, substituted aralkyl of 5 through 13 carbon atoms, wherein said substituent is selected from the group consisting of chlorine, nitro, alkyl, and alkoxy, tetrahydronaphthylalkyl, alkenyl of 3 through 8 carbon atoms, alkynyl of 3 through 8 carbon atoms, cycloalkyl of 3 through 12 carbon atoms, substituted cycloalkyl of 3 through 12 carbon atoms, wherein said substituent is selected from the group consisting of bromine, chlorine, methoxy, and alkyl, cycloalkenyl of 4 through 12 carbon atoms, substituted cycloalkenyl of 4 through 12 carbon atoms, wherein said substituent is selected from the group consisting of bromine, chlorine, methoxy, and alkyl, cycloalkyl alkyl of 4 through 13 carbon atoms, cycloalkenyl alkyl of 5 through 13 carbon atoms, (substituted cycloalkyl)alkyl of 5 through 14 carbon atoms, wherein said substituent is selected from the group consisting of bromine, chlorine, methoxy, and alkyl, (substituted cycloalkenyl)alkyl of 5 through 14 carbon atoms, wherein said substituent is selected from the group consisting of bromine, chlorine, methoxy, and alkyl, and cyano;

R_1 is selected from the group consisting of chlorine, fluorine, bromine, iodine, methyl, ethyl, propyl, butyl, methoxy, ethoxy, propoxy, butoxy, nitro alkoxymethyl of 2 through 6 carbon atoms, hydroxy alkyl of 1 through 6 carbon atoms, alkenyl of 3 through 6 carbon atoms, thiocyanato, cyano, thiolmethyl, alkylthio of 1 through 4 carbon atoms, bromomethyl, fluoromethyl, chloromethyl, methylthiomethyl, phenylthiomethyl, and carboxymethylthiomethyl;

R_2 is selected from the group consisting of chlorine, bromine, alkyl of 1 through 5 carbon atoms, chloroalkyl of 1 through 4 carbon atoms, bromoalkyl of 1 through 4 carbon atoms, and alkoxy of 1 through 5 carbon atoms; and

X is selected from the group consisting of oxygen and sulfur; and

(b) the sodium, potassium, lithium, calcium, magnesium, barium, strontium, iron, manganese, and quaternary ammonium salts of the compounds in (a).

2. The method of claim **1** wherein the herbicidal active compound is 5-bromo-3-sec-butyl-6-methyluracil.

3. The method of claim **1** wherein the herbicidal active compound is 5-bromo-3-sec-butyl-6-methyluracil, sodium salt.

4. The method of claim **1** wherein the herbicidal active compound is 5-chloro-3-sec-butyl-6-methyluracil.

5. The method of claim **1** wherein the herbicidal active compound is 5-bromo-3-tert-butyl-6-methyluracil.

6. The method of claim **1** wherein the herbicidal active compound is 5chloro-3-tert-butyl-6-methyluracil.

7. The method of claim **1** wherein the herbicidal active compound is 5-bromo-3-(1-ethylpropyl)-6-methyluracil.

8. The method of claim **1** wherein the herbicidal active compound is 5-chloro-3-(1-ethylpropyl)-6-methyluracil.

9. The method of claim **1** wherein the herbicidal active compound is 5-bromo-3-isopropyl-6-methyluracil.

10. The method of claim **1** wherein the herbicidal active compound is 5-chloro-3-isopropyl-6-methyluracil.

11. The method of claim **1** wherein the herbicidal active compound is 5-chloro-3-cyclohexylmethyl-6-methyluracil.

12. The method of claim **1** wherein the herbicidal active compound is 3-cyclohexyl-5-methoxy-6-methyluracil.

13. The method of claim **1** wherein the herbicidal active compound is 3-sec-butyl-5,6-dimethyluracil.

14. The method of claim **1** wherein the herbicidal active compound is 5-bromo-6-methyl-3-norbornylmethyluracil.

15. The method of claim **1** wherein the herbicidal active compound is 3-sec-butyl-6-methyl-5-nitrouracil.

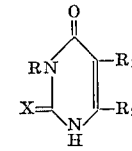
16. The method of claim **1** wherein the herbicidal active compound is 3-cyclohexyl-5,6-dimethyluracil.

17. The method of claim **1** wherein the herbicidal active compound is 5-bromo-3-cyclohexyl-6-methyluracil.

18. The method of claim **1** wherein the herbicidal active compound is 5-chloro-3-phenyl-6-methyluracil.

19. A method for the control of undesirable vegetation comprising applying to a locus to be protected in an amount sufficient to exert herbicidal action a compound selected from the group consisting of:

(a) compounds of the formula



where

R is selected from the group consisting of alkyl of 1 through 10 carbon atoms, substituted alkyl of 1 through 8 carbon atoms, wherein said substituent is selected from the group consisting of bromine, chlorine, hydroxy, alkoxy, alkoxycarbonyl, and cyano, aryl of 5 through 10 carbon atoms, substituted phenyl, wherein said substituent is selected from the group consisting of chlorine, bromine, fluorine, alkoxy of 1–5 carbon atoms, alkyl of 1–6 carbon atoms, nitro, trifluoromethyl, 1,2-tetramethylene, and 1,2-trimethylenylene, aralkyl of 5 through 13 carbon atoms, substituted aralkyl of 5 through 13 carbon atoms, wherein said substituent is selected from the group consisting of chlorine, nitro, alkyl, and alkoxy, tetrahydronaphthylalkyl, alkenyl of 3 through 8 carbon atoms, alkynyl of 3 through 8 carbon atoms, cycloalkyl of 3 through 12 carbon atoms, substituted cycloalkyl of 3 through 12 carbon atoms, wherein said substituent is selected from the group consisting of bromine, chlorine, methoxy, and alkyl, cycloalkenyl of 4 through 12 carbon atoms, substituted cycloalkenyl of 4 through 12 carbon atoms, wherein said substituent is selected from the group consisting of bromine, chlorine, methoxy, and alkyl, cycloalkyl alkyl of 4 through 13 carbon atoms, cycloalkenyl alkyl of 5 through 13 carbon atoms, (substituted cycloalkyl)alkyl of 5 through 14 carbon atoms, wherein said substituent is selected from the group consisting of bromine, chlorine, methoxy, and alkyl, (substituted cycloalkenyl)alkyl of 5 through 14 carbon atoms, wherein said substituent is selected from the group consisting of bromine, chlorine, methoxy, and alkyl, and cyano;

R_1 is selected from the group consisting of chlorine, fluorine, bromine, iodine, methyl, ethyl, propyl, butyl, methoxy, ethoxy, propoxy, butoxy, nitro, alkoxymethyl of 2 through 6 carbon atoms, hydroxy alkyl of 1 through 6 carbon atoms, alkenyl of 3 through 6 carbon atoms, thiocyanato, cyano, thiolmethyl, alkylthio of 1 through 4 carbon atoms, bromomethyl, fluoromethyl, chloromethyl, methylthiomethyl, phenylthiomethyl, and carboxymethylthiomethyl;

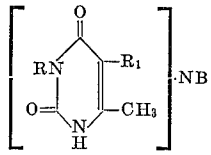
R_2 is selected from the group consisting of chlorine, bromine, alkyl of 1 through 5 carbon atoms, chloroalkyl of 1 through 4 carbon atoms, bromoalkyl of 1 through 4 carbon atoms, and alkoxy of 1 through 5 carbon atoms; and

X is selected from the group consisting of oxygen and sulfur; and

(b) the sodium, potassium, lithium, calcium, magnesium, barium, strontium, iron, manganese, and quat-

ernary ammonium salts of the compounds in (a);

(c) compounds of the formula

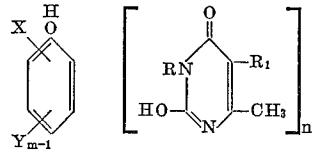


where

R and R_1 are as defined above; and

NB is a nitrogenous base having an ionization constant K_b of $\geqq 10^{-9}$ in water; and

(d) compounds of the formula



where

R and R_1 are as defined above;

X is selected from the group consisting of hydrogen,

chlorine, bromine, nitro, alkyl of 1 through 3 carbon atoms, and $—OR_3$ where R_3 is alkyl of 1 through 3 carbon atoms;

Y is selected from the group consisting of chlorine and alkyl of 1 through 3 carbon atoms;

m is a whole number 1 through 5; and

n is selected from the group consisting of 1 and 2.

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