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HERBICIDAL FORMULATIONS

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8 Claims. (Cl. 71—2.7)

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This invention relates to herbicidal formulations; more specifically, this invention relates to improved herbicidal formulations comprising a derivative of trichloroacetic acid.

Of the various types of formulations employed in the prevention and growth or the destruction of weeds, aqueous emulsions having one or more active herbicides dissolved or dispersed therein are most frequently utilized. Such emulsions offer many advantages. For example, from the standpoints of shipping and application, the active herbicides or a concentrate containing the same may be shipped directly to the area of application and diluted and/or emulsified with water immediately prior to application.

Oils are also generally employed in herbicidal formulations and the oils per se may be active herbicides. The inclusion of oils also permits a better contact between the weed or plant to be eradicated and the active herbicide present in the emulsion.

The salts of trichloroacetic acid have a recognized utility as selective herbicides. It is impossible, however, to prepare stable oil-in-water herbicidal emulsions containing such salts of trichlororoacetic acid. The salts are not soluble in oil and, hence, can not be dissolved in the oil phase of such an emulsion. It has further been found that the salts of trichloroacetic acid are excellent aqueous emulsion breakers. It is, therefore, impossible to prepare stable and permanent oil-in-water type herbicidal emulsions having salts of trichloroacetic acid dissolved or dispersed in the aqueous phase of such an emulsion. Consequently, heretofore it has not been possible to prepare a stable oil-in-water type emulsion having incorporated therein as one of the active herbicides, such a derivative of trichloroacetic acid as a salt thereof.

It is an object of this invention to provide an improved herbicidal formulation employing a derivative of trichloroacetic acid as one of the active ingredients.

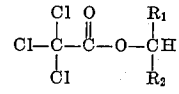
It is a further object of this invention to provide an improved herbicidal formulation comprising an oil and a derivative of trichloroacetic acid.

Another object of this invention is to provide an improved herbicidal formulation comprising an oil-in-water type emulsion having dissolved or dispersed therein, a derivative of trichloroacetic acid.

Further objects of this invention will become apparent from the description of the novel compositions which follow.

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It has now been discovered that an alkyl ester of trichloroacetic acid having the formula



wherein R_1 and R_2 represent alkyl radicals which together contain not more than 7 carbon atoms, or a solution thereof in oil may be emulsified into a substantially permanent oil-in-water type herbicidal emulsion. As a result of this invention, it is, therefore, now possible to prepare herbicidal formulations comprising an aqueous emulsion of a derivative of trichloroacetic acid, either as the sole active herbicide or together with other oil soluble selective herbicides, and/or herbicidal oils. The following examples are typical of the outstanding herbicidal formulations that may be prepared in accordance with the teachings of this invention:

Example I

75 pounds of isopropyl trichloroacetate were combined by mechanical mixing with 75 pounds of oil and 7.5 pounds of a suitable oil soluble emulsifying agent to form a stable emulsifiable concentrate. The resulting concentrate is emulsified with 200 gallons of water to form an exceptionally stable oil-in-water emulsion formulation. After spraying this emulsion substantially uniformly over a one acre tract of grassy, weed-covered land, the leaves and stalks of the plants are found to be completely killed within a few days.

Example II

3 pounds of pentachlorophenol are dissolved in 5 gallons of an aromatic oil and emulsified with 2.5 gallons of water with the aid of a suitable emulsifying agent to form a concentrated oil-in-water emulsion. Thereafter, 12.5 pounds of isopropyl trichloroacetate are added and then about 100 gallons of water are incorporated with stirring. The resulting emulsion is found to have a stability comparable to the product of Example I and a high degree of herbicidal activity.

Example III

2 pounds of pentachlorophenol are dissolved in 5 gallons of oil and emulsified with 2.5 gallons of water with the aid of a suitable emulsifying agent to form a concentrated oil-in-water emulsion. Therefore, 2 pounds of the isopropyl ester of 2,4-dichlorophenoxyacetic acid and 10 pounds of isopropyl trichloroacetate are added and then about 200 gallons of water are incorporated with

stirring. The resulting emulsion is comparable with the product of Example II in stability and herbicidal activity.

Example IV

Example I is repeated except that a stoichiometrically equivalent amount of secondary butyl trichloroacetate is substituted for isopropyl trichloroacetate. The product is generally similar in its characteristics to the product of Example I.

Example V

Example III is repeated except that a stoichiometrically equivalent amount of 1-methylheptyl trichloroacetate is substituted for isopropyl trichloroacetate. A generally similar product is obtained.

Example VI

5 pounds of an oil soluble emulsifying agent is dissolved in 100 pounds of isopropyl trichloroacetate to form an emulsifiable concentrate which is then dispersed with agitation in 250 gallons of water. A highly active herbicidal emulsion of remarkable stability results.

The esters of trichloroacetic acid utilized in the novel compositions of this invention may be conveniently prepared by esterifying trichloroacetic acid with a secondary alcohol containing at least 3 and not more than 8 carbon atoms. While the use of isopropyl trichloroacetate represents a preferred embodiment of this invention, the following alkyl trichloroacetates are illustrative of the compounds which may be utilized in the novel compositions of this invention:

Secondary butyl trichloroacetate
1-methylbutyl trichloroacetate
1,2-dimethylpropyl trichloroacetate
1-ethylpropyl trichloroacetate
1-methylpentyl trichloroacetate
1-ethylbutyl trichloroacetate
1-ethyl-2-methylpropyl trichloroacetate
1,3-dimethylbutyl trichloroacetate
1,2-dimethylbutyl trichloroacetate
1-methyl-2-ethylbutyl trichloroacetate
1-isopropylbutyl trichloroacetate
1-ethyl-3-methylbutyl trichloroacetate
1-methyl-4-methylpentyl trichloroacetate
1-ethyl-2-methylbutyl trichloroacetate
1-methylhexyl trichloroacetate
1-propylbutyl trichloroacetate
1-methylheptyl trichloroacetate

In contrast to the outstandingly stable oil-in-water type emulsions which may be prepared in accordance with this invention are the relatively unstable oil-in-water type emulsions obtained with other esters of trichloroacetic acid. This uniqueness of the novel compositions of this invention is very clearly demonstrated by the following emulsion stability test wherein the result obtained on the formulation containing isopropyl trichloroacetate is indicative of the behavior of herbicidal oil-in-water type emulsions prepared in accordance with the teachings of this invention. Three concentrated herbicidal emulsions were prepared, each containing the following common ingredients:

1 gallon of an aromatic oil having a boiling point within the range of 360° F. to 620° F.
½ gallon water
0.6 lb. of pentachlorophenol
0.11 lb. of an emulsifier containing 77% by weight of sodiumdodecylbenzenesulfonate and 23% by weight of a sodium petroleum ("mahogany") sulfonate.

In addition, the first emulsion contained 2.58 lbs. of ethyl trichloroacetate. The second emulsion contained 2.79 lbs. of isopropyl trichloroacetate, and the third emulsion contained 2.96 lbs. of butyl trichloroacetate. The above prepared concentrated herbicidal emulsions were allowed to stand and the lapse of time before the emulsion broke noted. It was found that the emulsion containing the ethyl trichloroacetate broke in approximately 8 days and could not be re-emulsified. The emulsion containing the butyl trichloroacetate broke in approximately 10 days and could not be re-emulsified. However, the emulsion containing isopropyl trichloroacetate did not begin to break until the elapse of approximately 21 days, and could be re-emulsified by merely shaking the formulation. It was also found that an oil-in-water type emulsion identical to those described above except that it contained 2.5 lbs. of sodium trichloroacetate (which is equivalent to 2.79 lbs. of isopropyl trichloroacetate) in place of an ester of trichloroacetic acid, broke almost immediately after formation. It is clearly evident from these data that isopropyl trichloroacetate possesses an outstanding hydrolytic stability which permits the preparation of stable herbicidal emulsions.

When the novel herbicidal formulations of this invention contain oils, there may be used any of the common oil bases, such as Diesel oils, mineral oils, fuel oils, pine oil and aromatic oils, such as those aromatic oils boiling within the range of 176° F. to 760° F., or those oils which are aromatic in nature and contain at least 15% of aromatic hydrocarbons boiling within the range of 176° F. and 760° F. Typical examples of these aromatic hydrocarbon oils are benzene, toluene, ethylbenzene, xylene, alkyl naphthalenes such as the methyl substituted naphthalenes, ethylnaphthalenes, etc., and mixtures of oils containing these and/or other aromatic hydrocarbons. These herbicidal oil formulations may also have dissolved therein any of the commonly used oil soluble herbicidal agents well known to those skilled in the art, such as the halogenated phenols, the aryloxy and substituted aryloxy aliphatic carboxylic acids and their esters, etc.

Examples of emulsifying agents which may be used in the herbicidal formulations of the invention include various wetting, dispersing and emulsifying agents, such as the alkyl aryl sulfonates, of which sodium dodecylbenzenesulfonate is a particular example, sulfated fatty alcohols, alkyl sulfonates, sulfated esters and acids, amide sulfates and sulfonates, sulfated and sulfonated oils, fats, and waxes, fatty esters of glycerol and glycol and other polyhydric alcohols, such as sorbitol, the condensation products of ethylene oxide with fatty acids, abietic acid, alkyl phenols, such as nonyl phenol, and mercaptans, such as tert.-dodecyl mercaptan, etc., as well as mixtures of these and/or other surface-active agents.

Emulsifiable concentrates of the trichloroacetic acid esters of the invention may be prepared in various forms as indicated by the examples, e. g. solutions of the esters in oil bases, solutions of emulsifying agents in the esters and concentrated oil-in-water emulsions containing the esters, etc.

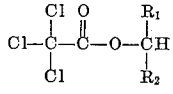
The concentration of the alkyl trichloroacetate in the oil-base formulation may be varied over a wide range. The concentration utilized is

governed by the herbicidal activity desired, the concentration desired in the finished emulsion formulation, and the solubility of the alkyl trichloroacetate in the particular oil base utilized. For most purposes, a concentration within the range of from about 10% to about 75% by weight of the alkyl trichloroacetate based on the oil-alkyl trichloroacetate solution has been found most satisfactory.

Dilute herbicidal emulsions may be prepared from the emulsifiable concentrates or concentrated emulsions of this invention by simple dilution accompanied by stirring or other agitation of the mixture of concentrate and water. Such herbicidal emulsions may be prepared at the site of application or may be prepared in advance and transported to the site of application in the form of finished emulsions. Preferably, however, for convenience and economy, the emulsions are prepared immediately prior to use. The amount of the alkyl trichloroacetate contained in the dilute emulsions may be substantially varied depending at least in part on the use to which the emulsion is to be placed. Usually from 0.05 to 10% of the ester is used based on the weight of the entire emulsion.

What is claimed is:

1. An improved herbicidal composition comprising an oil, an emulsifying agent, and an alkyl trichloroacetate having the formula



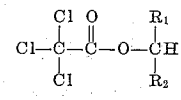
wherein R_1 and R_2 represent alkyl radicals which together contain not more than 7 carbon atoms, said composition being emulsifiable with water to form a stable emulsion.

2. The composition as described in claim 1 wherein the alkyl trichloroacetate is isopropyl trichloroacetate.

3. The composition as described in claim 1 wherein the alkyl trichloroacetate is sec.-butyl trichloroacetate.

4. The composition as described in claim 1 wherein the alkyl trichloroacetate is 1-methylheptyl trichloroacetate.

5. An improved herbicidal composition comprising a stable oil-in-water emulsion containing an alkyl trichloroacetate having the formula



wherein R_1 and R_2 represent alkyl radicals which together contain not more than 7 carbon atoms.

6. The composition as described in claim 5 wherein the alkyl trichloroacetate is isopropyl trichloroacetate.

7. The composition as described in claim 5 wherein the alkyl trichloroacetate is sec.-butyl trichloroacetate.

8. The composition as described in claim 5 wherein the alkyl trichloroacetate is 1-methylheptyl trichloroacetate.

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