3,746,727

BENZENESULFHYDROXAMIC ACIDS AND THEIR DERIVATIVES

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2 Claims

ABSTRACT OF THE DISCLOSURE

Certain novel ring-substituted benzenesulfhydroxamic acids and esters, useful as herbicides.

BACKGROUND OF THE INVENTION

Field of the invention

This invention relates to a novel class of benzenesulfhydroxamic acids and esters, substituted on the ring with a secondary amino and two nitro moieties, which exhibit high herbicidal activity.

Description of the prior art

The herbicidal ring-substituted benzenesulfhydroxamic acids and esters of this invention have not previously been disclosed nor is there any reference known which would anticipate them. U.S. Pat. 3,398,180 and 3,492,323 disclose a series of N-(2-propynoxy)sulfanilamides substituted in two unspecified positions on the ring by a series of different functional groups including dialkylamino and nitro, which are useful as flavoring agents and chemical intermediates. Other related art is U.S. Pat. 3,367,949 which discloses a series of substituted 3,5-dinitrosulfanilamides as herbicides.

SUMMARY OF THE INVENTION

A new series of benzenesulfhydroxamic acids and esters substituted in the para position, relative to the sulfhydroxamic acid or ester moiety, with a secondary amino moiety and in the meta positions with nitro moieties have been discovered. These novel compounds have shown high herbicidal activity in the control of several economically important species of grasses and broad leaved weeds.

The invention, accordingly, is a new class of benzenesulfhydroxamic acids and esters, their use as herbicides and herbicidal formulations containing them.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The novel compounds of this invention can be described by the general Formula I

wherein R¹ and R² are the same or different and each is alkyl of up to 4 carbon atoms, alkenyl of up to 4 carbon atoms, or cycloalkyl of up to 6 carbon atoms; R³ is hydrogen, alkyl of up to 4 carbon atoms, alkenyl of up to 4 carbon atoms, a lower alkali metal ion (Na, K or Li), a quaternary ammonium group wherein the cationic nitrogen is substituted with up to 4 alkyl groups each containing up to 3 carbon atoms or

wherein R⁵ is hydrogen, alkyl of up to 4 carbon atoms, alkenyl of up to 4 carbon atoms, cycloalkyl of up to 6 carbon atoms, —OR⁶ wherein R⁶ is alkyl of up to 4 carbon atoms; —NR²R³ wherein R² is hydrogen and R³ is alkyl of up to 4 carbon atoms, Q is oxygen or sulfur, with the proviso that when Q is oxygen, R¹ is hydrogen, alkyl of up to 4 carbon atoms, cycloalkyl of up to 6 carbon atoms or

wherein R⁸ is hydrogen, alkyl of up to 4 carbon atoms, alkenyl of up to 4 carbon atoms or —OR⁶ wherein R⁶ is as described above and when Q is sulfur R³ is

wherein R⁸ is as described above and R⁴ is alkyl of up to 2 carbon atoms wherein all the hydrogens have been replaced by chlorine or fluorine.

Exemplary species of the class of the invention include:

tetramethylammonium salt of 4-(dipropylamino)-N-hydroxy-3,5-dinitrobenzenesulfonylamide
4-(dipropylamino)-N-butoxy-N-butyl-3,5-dinitrobenzenesulfonylamide
4-(dipropylamino)-N-allyloxy-N-ethyl-3,5-dinitrobenzenesulfonylamide
N-(4-dipropylamino)-3,5-dinitrobenzenesulfonyl-N-methoxyformamide
4-(dipropylamino)-N-formoxy-N-methyl-3,5-dinitrobenzenesulfonylamide
N-(4-dipropylamino)-3,5-dinitrobenzenesulfonyl)-N-methoxyacrylamide
N-(4-dipropylamino)-3,5-dinitrobenzenesulfonyl)-N-allyloxyacrylamide
4-(dipropylamino)-N-acyrloxyloxy-N-methyl-3,5-dinitrobenzenesulfonylamide
N-(4-dipropylamino)-3,5-dinitrobenzenesulfonyl)-N-methoxyacyclopropane-carboxamide
N-(4-dipropylamino)-3,5-dinitrobenzenesulfonyl)-N-acetoxyacyclopropane-carboxamide
1-(4-dipropylamino)-3,5-dinitrobenzenesulfonyl)-1-allyloxy-3-methyleurea
4-(allylthioamino)-N-methoxy-N-methyl-3,5-dinitrobenzenesulfonylamide
4-(allyloxycyclopropylamino)-N-hydroxy-N-allyl-3,5-dinitrobenzenesulfonylamide
N-(4-allylthioamino)-3,5-dinitrobenzenesulfonyl)-N-hydroxyacyrlyamide
Diallylamine)-N-acyrloxyloxy-N-methyl-3,5-dinitrobenzenesulfonylamide
N-(4-cyclopropylethylamino)-3,5-dinitrobenzenesulfonyl-N-methoxyacyclopropane-carboxamide
N-(4-ethylpropylamino)-3,5-dinitrobenzenesulfonyl)-N-acetoxyformamide
N-(4-(butylmethyamino)-3,5-dinitrobenzenesulfonyl)-N-hydroxyformamide

Preferred because of their especially effective herbicidal properties are those compounds in the subclass wherein R¹ is alkyl of up to 4 carbon atoms or alkenyl of up to 3 carbon atoms; R² is alkyl of up to 4 carbon atoms or cyclopropyl; Q is oxygen or sulfur with the proviso that when Q is oxygen R² is hydrogen, alkyl of up to 3 carbon atoms or

wherein R⁵ represents alkyl of up to 3 carbon atoms, alkoxy of up to 3 carbon atoms or monoalkylamino of up to 3 carbon atoms and R⁴ is hydrogen, alkyl of up
to 4 carbon atoms, acyl of up to 3 carbon atoms, a lower alkali metal ion (Na, K or Li), or

wherein \( R^2 \) is alkyl of up to 3 carbon atoms; when \( Q \) is sulfur \( R^3 \) is

wherein \( R^7 \) represents alkyl of up to 3 carbon atoms and \( R^8 \) is trichloromethyl. Examples of such preferred herbicides are:

4-(dimethylamino)-N-hydroxy-3,5-dinitrobenzenesulfonamide

4-(methylpropylamino)-N-ethoxy-3,5-dinitrobenzenesulfonamide

N-acetoxy-N-(4-(dimethylamino)-3,5-dinitrobenzenesulfonyl)propionamide

N-propoxy-N-(4-(diisopropylamino)-3,5-dinitrobenzenesulfonfyl)acetamide

N-propionyloxy-N-(4-(butylethylamino)-3,5-dinitrobenzenesulfonfyl)propionamide

Sodium salt of 4-(diethylamino)-N-hydroxy-3,5-dinitrobenzenesulfonamide

Potassium salt of 4-(butylethylamino)-N-hydroxy-3,5-dinitrobenzenesulfonamide

4-(dimethylamino)-N-hydroxy-N-propyl-3,5-dinitrobenzenesulfonamide

4-(diethylamino)-N-acetoxy-N-methyl-3,5-dinitrobenzenesulfonamide

1-(4-(diethylamino)-3,5-dinitrobenzenesulfonyl)-1-methoxy-3-methylurea

1-(4-(butylethylamino)-3,5-dinitrobenzenesulfonfyl)-1-methoxy-3-propylurea

O-ethyl-N-(4-(diethylamino)-3,5-dinitrobenzenesulfonyl)-N-(ethoxy carbonyl)carbamate

N-(4-(diethylamino)-3,5-dinitrobenzenesulfonyl)-N-trichloromethylthioacetamide

N-(4-(diisopropylamino)-3,5-dinitrobenzenesulfonyl)-N-(trichloromethylthio)propionamide

4-(allylcyclopropylamino)-N-hydroxy-3,5-dinitrobenzenesulfonamide

N-propionyloxy-Np-(4-allylcyclopropylamino)-3,5-dinitrobenzenesulfonfyl)acetamide

N-propoxy-N-(4-(allylcyclopropylamino)-3,5-dinitrobenzenesulfonyl)acetamide

O-methyl-N-(4-(allylcyclopropylamino)-3,5-dinitrobenzenesulfonyl)-N-(ethoxy carbonyl)carbamate

N-(4-(allylcyclopropylamino)-3,5-dinitrobenzenesulfonyl)-N-(trichloromethylthio)acetamide

The compounds of the invention are solid at ambient temperature. They may be suitably formulated for use as herbicides, as wettable powders, dusts, granules, solutions, emulsifiable concentrates, emulsions and pastes. Wettable powders are usually compounded to contain 25, 50, 75 or up to 85% of toxicant and usually contain, in addition to solid carrier, 3–10% of a dispersing agent and, where necessary, 0–10% of stabilizer(s) and/or other additives such as penetrants or stickers. Dusts are usually formulated as a dust concentrate having a similar composition to that of a wettable powder but without a dispersant, and are diluted in the field with further solid carrier to give a composition usually containing 0.5–10% of toxicant. Granules are usually prepared to have a size between 10 and 100 BS mesh, and may be manufactured by agglomeration or impregnation techniques. Generally, granules will contain 0.5 to 25% of toxicant plus additives such as stabilizers, slow-release modifiers, binding agents, etc. Emulsifiable concentrates usually contain, in addition to the solvent and, when necessary, co-solvent, 10–50% s/v. toxicant, 2–20% w/v. emulsifiers and 0–20% of appropriate additives such as stabilizers, penetrants and corrosion inhibitors. Pastes are compounded so as to obtain a stable flowable product and usually contain 10–60% toxicant, 2–20% of appropriate additives and, as carrier, water or an organic liquid in which the toxicant is substantially insoluble. Except where indicated otherwise, all percentages given in this paragraph are percentages by weight.

For field application, the rate of application of the active agent may be varied from about 0.1 to 30 or more pounds per acre. It will be appreciated that the rate of application is subject to variation according to the particular active agent used, the particular species of plants involved and the local conditions; for example, temperature, humidity, soil moisture, chemical make-up soil and the like. Effective resolution of these factors is well within the skill of those versed in the herbicide art.

The herbicidal sulfonhydroxy acids described by Formula I above may be prepared by reaction of a benzene-sulfonyl chloride of Formula II

\[
R^4O—NRH^3
\]

wherein \( R^3 \) and \( R^4 \) are as defined above, with hydroxylamine. The reaction is suitably carried out in an aqueous tetrahydrofuran medium at a reaction temperature ranging from 5 to 25° C. with a reaction time of 0.1 to 2.5 hours. It has been found convenient to charge an excess of the hydroxylamine reactant to serve as an acceptor for the by-product hydrogen chloride which is generated by the reaction, however tertiary amines such as triethylamine or pyridine should also be suitable for this purpose. The alkylated and/or alkenylated sulfonhydroxy acid derivatives described by Formula I where \( Q \) is oxygen may be prepared by a reaction analogous to that described above for the sulfonhydroxy acids, using as the hydroxylamine reactant, a substituted hydroxylamine of the Formula III
The reaction is properly carried out in the presence of a base such as a tertiary amine, e.g., triethylamine, which serves as an acceptor of the by-product hydrogen chloride generated by the reaction.

The substituted benzenesulfonyl chlorides described by Formula II are known compounds and can be prepared from p-chlorobenzenesulfonic acid using a four-step synthesis scheme. In the initial reaction step the p-chlorobenzenesulfonic acid is nitrated in the 3 and 5 positions on the ring with a mixture of 90% nitric acid in 65% oleum. The dinitro intermediate is then converted to an alkali metal salt, e.g., sodium or potassium, by neutralization with the corresponding hydroxide and the salt compound is reacted with a secondary amine of the formula RNR'NHR wherein R and R' are as defined above to yield the alkali metal salt of a 4-(N,N-disubstituted amino)-3,5-dinitrobenzenesulfonic acid. This alkali metal salt is then converted to the corresponding sulfonyl chloride by treatment with phosphorus pentachloride in a solution of phosphor trichloride.

The thiolydroxamic acid derivatives of this invention, i.e., the compounds of Formula I wherein Q is sulfur, may be prepared by the reaction of an acylated benzenesulfonylamide of Formula IV.

![Structure](Formula IV)

with a polyhaloalkylsulfinyl chloride of the formula

\[ \text{CIS}_n\text{X} (2n-1) \]

wherein X is chlorine or fluorine and N is 1 or 2. This reaction is suitably carried out in a dioxane reaction medium at reaction temperatures ranging from ambient to 50°C with a reaction time of from 5 to 20 hours. The reaction is properly carried out in the presence of a base such as a tertiary amine e.g., triethylamine, which serves as an acceptor for the by-product hydrogen chloride generated by the reaction.

The acylated benzenesulfonylamides of Formula IV may be prepared by reacting the unacylated analogs with an acid anhydride of the formula

\[ (R-C=O)_x \]

wherein R is as defined above. The reaction is suitably carried out neat, or in an aromatic solvent such as xylene, at reflux temperatures with reaction times of from 1 to 2 hours. These unacylated benzenesulfonylamide analogs are known compounds, e.g., see U.S. Pat. 3,367,949.

The compounds of the invention, processes for their preparation and their herbicidal activity, are illustrated by the following examples, which in parts by weight (w.) and parts by volume (v.) bear the same relation as the kilogram to the liter and all temperatures are in degrees centigrade. The structures of the compounds, whose preparation is described in the following examples, were confirmed by infrared spectrum, nuclear magnetic resonance and elemental analysis.

**EXAMPLE I**

4-(dipropylamino)-N-hydroxy-3,5-dinitrobenzenesulfonamide (16.6 w.) melting at 185°C with decomposition.

**EXAMPLE II**

4-(dipropylamino)-N-hydroxy-3,5-dinitrobenzenesulfonamide (5.6 w.) prepared as in Example I was added to a solution of sodium methoxide (0.4 w.) in methanol (75 v.) for 60 minutes to give an amorphous, water soluble, red solid, sodium salt of 4-(dipropylamino)-N-hydroxy-3,5-dinitrobenzenesulfonamide (2.5 w.) which decomposed on heating to 110°C.

**EXAMPLE III**

N-acetoxy-N-(dipropylamino)-3,5-dinitrobenzenesulfonyl)-acetamide

A mixture of 4-(dipropylamino)-N-hydroxy-3,5-dinitrobenzenesulfonamide (6.0 w.) prepared as in Example I, and 50 v. of acetic anhydride was heated to reflux for 30 minutes. The reaction mixture was concentrated under vacuum. The residue was dissolved in 250 v. of ether, washed with aqueous sodium hydrogen carbonate, and dried over anhydrous magnesium sulfate. Concentration of the ether solution gave an orange viscous oil which was crystallized from a solvent mixture of ether (3 parts) and pentane (1 part) to afford an orange-yellow crystalline solid, N-acetoxy-N-(dipropylamino)-3,5-dinitrobenzenesulfon酰胺 (7.0 w.) melting at 89-91°C.
EXAMPLE IV

1-(4-(dipropylamino)-3,5-dinitrobenzenesulfonyl)-1-methoxy-3-methylurea

\[
\begin{align*}
\text{CH}_3 & \quad \text{N} \quad \text{O} \\
\text{SO}_2 & \quad \text{N} \quad \text{CH}_3 \\
\text{O} & \quad \text{CH}_3
\end{align*}
\]

4-(dipropylamino)-N-methoxy-3,5-dinitrobenzenesulfonamide (14.0 w.) prepared according to the technique described in Example I using methoxymine as the amine reactant was charged into a glass cylinder containing a mixture of methyl isocyanate (5 v.) and ether (200 v.). The cylinder was sealed and placed on a steam bath for 18 hours. The reaction mixture was chilled in an ice bath and filtered to yield a yellow crystalline solid, 1-(4-(dipropylamino)-3,5-dinitrobenzenesulfonyl)-1-methoxy-3-methylurea melting at 160-163° and 171-174°.

EXAMPLE V

Ethyl N-(4-dipropylamino)-3,5-dinitrobenzenesulfonyl-N-(ethoxycarbonyloxy)carbamate

\[
\begin{align*}
\text{CH}_2\text{CH}_2 & \quad \text{N} \\
\text{CH}_3 & \quad \text{O} \\
\text{SO}_2 & \quad \text{N} \\
\text{CH}_3 & \quad \text{O} \\
\text{CH}_2\text{CH}_2
\end{align*}
\]

Triethylamine (2.2 w.) was added to a solution of 4-(dipropylamino)-N-hydroxy-3,5-dinitrobenzenesulfonamide (3.6 w.), prepared as in Example I, in tetrahydrofuran (100 v.). The resulting dark solution was stirred during the dropwise addition (3 minutes) of 2.4 w. of ethyl chloroformate. This addition was exothermic to 45° and caused the color to change to a light yellow with solid suspended. The mixture was heated to 60° for 1.5 hours and then poured into ice water. The product was extracted with 3 x 100 v. of methylene chloride and dried over anhydrous magnesium sulfate. Evaporation of the solvent gave 2.5 w. of red oil which was crystallized from ether to yield a yellow crystalline solid, ethyl N-(4-(dipropylamino)-3,5-dinitrobenzenesulfonyl)-N-(ethoxycarbonyloxy)carbamate melting at 104-107°.

EXAMPLE VI

N-(4-dipropylamino)-3,5-dinitrobenzenesulfonyl)-N-(trichloromethylthio)acetamide

\[
\begin{align*}
\text{CH}_2\text{CH}_2 & \quad \text{N} \\
\text{CH}_3 & \quad \text{O} \\
\text{SO}_2 & \quad \text{N} \\
\text{CH}_3 & \quad \text{O} \\
\text{CH}_2\text{CH}_2
\end{align*}
\]

4-(dipropylamino)-3,5-dinitrobenzenesulfonamide (10 w.) was added to acetic anhydride (25 v.), and the mixture was heated at reflux for two hours. The volatile impurities were removed by distillation in a vacuum, and ice water was added to the residue. After stirring and scratch-crystallization using a yellow solid crystallized out of solution. The solid product was filtered, washed with water and dried. The product was then recrystallized from an ether-hexane mixture and dried in a vacuum, to yield N-(4-(dipropylamino)-3,5-dinitrobenzenesulfonyl)-N-(trichloromethylthio)acetamide melting at 148-149°. The structure was confirmed by elemental and IR analyses.

To a solution of 7.8 w. of N-(4-(dipropylamino)-3,5-dinitrobenzenesulfonyl)acetamide and 2.0 w. of triethylamine in 40 v. of dioxane was added dropwise at 10° with stirring a solution of 2.7 v. of trichloromethanesulfonyl chloride in 20 v. of dioxane. The mixture was stirred at ambient temperature for 3 hours and left standing for 16 hours. The reaction mixture was poured over ice water, extracted with 2 x 150 v. of methylene chloride. Drying (MgSO₄) and evaporating the solvent gave a brown yellow viscous oil which upon titration with ethanol yielded a yellow crystalline solid, N-(4-(dipropylamino)-3,5-dinitrobenzenesulfonyl)-N-(trichloromethylthio)acetamide (6.0 w.) melting at 111-114°. The structure was confirmed by NMR, IR and elemental analyses.

Analysis—Calculated (percent): C, 33.5; H, 3.5; N, 10.4. Found (percent): C, 33.8; H, 3.9; N, 10.8.

EXAMPLE VII

Following procedures similar to those given in previous examples, the following other species of the compounds of the invention were prepared (Symbols refer to formula V). In all cases the identity of the compounds was established by elemental, IR and/or NMR analyses.

<table>
<thead>
<tr>
<th>R₁</th>
<th>R₂</th>
<th>R₃</th>
<th>R₄</th>
</tr>
</thead>
<tbody>
<tr>
<td>(CH₂)₂N</td>
<td>SO₂</td>
<td>N</td>
<td>CH₃</td>
</tr>
<tr>
<td>CH₃</td>
<td>O</td>
<td>CH₃</td>
<td>CH₃</td>
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<tr>
<td>CH₃</td>
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<td>CH₃</td>
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<tr>
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<td>O</td>
<td>CH₃</td>
<td>CH₃</td>
</tr>
<tr>
<td>CH₃</td>
<td>O</td>
<td>CH₃</td>
<td>CH₃</td>
</tr>
</tbody>
</table>

EXAMPLE VIII

Pre-emergence herbicidal activity of the typical compounds of the invention was evaluated by planting weed
seeds in soil held in large test tubes, the soil having been treated with the test compound at the rate of 1 and 10 milligrams of test compound per tube, respectively. Seeds of water grass (*Echinochloa crus-galli*) and cress (*Lepidium sativum*) were germinated in treated soil under controlled conditions of temperature and light for 12 to 13 days prior to evaluation of the effectiveness of the treatments. At that time, the germination was noted and the treatments were rated on a 0 (no effect) to 9 (all dead) scale. The results are summarized in Table II.

We claim as our invention:

1. A compound defined by the formula:

   
   ![Chemical Structure](image)

   wherein R1, R3 and R4 are the same or different and each is alkyl of 1 to 4 carbon atoms and R4 is perchloro- or perfluoro-alkyl of 1 to 2 carbon atoms.

2. The compound according to claim 1 wherein R1 and R3 each is propyl, R2 is methyl and R4 is trichloro-methyl.

### Table II

<table>
<thead>
<tr>
<th>Q</th>
<th>R1</th>
<th>R2</th>
<th>R3</th>
<th>R4</th>
<th>Watergrass</th>
<th>Crass</th>
<th>Crabgrass</th>
<th>Pigmweed</th>
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<tr>
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<td>CH3CH3</td>
<td>H</td>
<td>H</td>
<td>4</td>
<td>7</td>
<td>1</td>
<td>5</td>
</tr>
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<td>CH3CH3</td>
<td>H</td>
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<td>0</td>
<td>8 9</td>
<td>7 8 1</td>
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<tr>
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<td>CH3CH3</td>
<td>CH3</td>
<td>O</td>
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<td>5 6</td>
<td>2 9 1 2 3 4</td>
<td>4 2 7</td>
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<tr>
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<td>CH3CH3</td>
<td>CH3</td>
<td>O</td>
<td>7 9 7 8 1 8</td>
<td>5 6</td>
<td>2 9 1 2 3 4</td>
<td>4 2 7</td>
</tr>
<tr>
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<td>CH3CH3</td>
<td>CH3</td>
<td>CH3</td>
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<td>0 0</td>
<td>0 0 1 1 7 5</td>
<td>4 8 7</td>
</tr>
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<td>CH3CH3</td>
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<td>5 6</td>
<td>3 8 4 5</td>
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<td>CH3CH3</td>
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<td>5 6</td>
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<tr>
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<td>CH3CH3</td>
<td>CH3</td>
<td>O</td>
<td>7 8 7 8 3 8</td>
<td>5 6</td>
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<td>CH3CH3</td>
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<td>O</td>
<td>7 8 7 8 3 8</td>
<td>5 6</td>
<td>3 8 4 5</td>
<td></td>
</tr>
</tbody>
</table>

Post-emergence herbicidal activity was evaluated by spraying dilute suspensions of the test compound in a 1:1 mixture of acetone and water with 0.5% wetting agent on crabgrass (*Digitaria sanguinalis*) and pigweed plants (*Amaranthus sp*) grown under controlled conditions at the rate of 1 and 10 pounds test compound per acre. After the plants were held for 10 to 11 days, they were rated for treatment effect on a 0 (no effect) to 9 (total plant kill) scale. The results of these test are shown in Table II.