### Applicant

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**Agent:**


### Title

**Title:** EFFERVESCENT PESTICIDE TABLET WITH METAL PERBORATE

### Abstract

An effervescent tablet formulation comprising about 0.1 % to 75 % of a water-soluble pesticide and about 25 % to 99 % anhydrous metal perborate salt characterized by rapid breakup in cold water.
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EFFERVESCENT PESTICIDE TABLET WITH METAL PEROBorate

WO 90/00007 discloses pesticidal tablets comprising an acid and a base which react upon contact with water to produce the effervescent reaction that causes the pesticide to rapidly disperse. The present tablets differ from those of WO 90/00007 in several important aspects including the fact that it is the reaction of anhydrous perborate salts with water that produces the effervescent reaction.

SUMMARY OF THE INVENTION

This invention concerns a tablet formulation comprising, by total weight of the formulation, about 0.1% to 75% of a water-soluble pesticide which is solid at room temperature, and 25% to 99.9% of an anhydrous metal perborate salt.

By "tablet formulation" is meant the tablet made from the composition described herein, as well as the composition formulated in accordance with this disclosure but not in tablet form. By "anhydrous metal perborate salt" is meant a sodium, lithium, or potassium salt having a water content less than about 2% by weight as determined by coulometric measurement.

Preferred ranges of the composition are 10% to 70%, and more preferred 30% to 60%, of the pesticide, and 30% to 90%, and more preferred 40% to 70%, of the anhydrous metal perborate.

Contemplated water-soluble, solid pesticides include those selected from the following classes, including mixtures thereof: herbicides, fungicides, bactericides, and insecticides. Preferred pesticides are those having a melting point of at least about 75°C and solubility in pH 7 water at 20°C of at least about 2% by weight.

Examples of suitable water-soluble pesticides include: insecticides such as methomyl and oxamyl; fungicides such as dodine salts, phosethyl-Al, kasugamycin, and validamycin; bactericides such as streptomycin and tribasic copper sulfate; and herbicides such as sulfonlurea salts, acifluorfen salts, ammonium sulfamate, amitrole, bromoxynil salts, cacodylic acid salts, clopyralid salts, calcium salt of methylarsonic acid, dalapon salts, dazomet, dicamba salts, difenzoquat methyl sulfate, diquat, 2-methyl-4,6-dinitrophenol salts, disodium salt of methylarsonic acid, endotheall, fenac, salt of fenuron and trichloroacetic acid, fluoroxypr salts, fomesafen, fosamine ammonium, glyphosate salts, haloxyfop salts, hexaflurare, imazaquin salts, imazethapyr salts, ioxynil sodium salt, monoammonium salt of methylarsonic acid, (4-chloro-2-methylphenoxy)acetic acid salts, MCPP salts, mecoprop salts, mefluidide salts, metam sodium, monuron,
salt, monosodium salt of methylarsonic acid, naptalam, paraquat salts, picloram salts, quinclorac salts, sodium 2-chloro-6-[(4,6-dimethoxy-2-pyrimidinyl)thio]benzoate, trichloroacetic acid salts, triclopyr salts, (2,4-dichlorophenoxy)acetic acid salts, and 4-(2,4-dichlorophenoxy)butanoic acid. Contemplated sulfonylurea salts have the formula:

\[
\begin{array}{c}
\text{J-SO}_2\text{NCNR} \\
\text{W} \\
\text{X} \\
\text{Y} \\
\text{Z} \\
\text{M}^+ \\
\end{array}
\]

wherein J is selected from the group

J-1, J-2, J-3, J-4, J-5, J-6, J-7, J-8, J-9.
R is selected from the group H and CH₃;
R¹ is selected from the group F, Cl, Br, NO₂, C₁-C₄ alkyl, C₁-C₄ haloalkyl,
C₃-C₄ cycloalkyl, C₂-C₄ haloalkenyl, C₁-C₄ alkoxy, C₁-C₄
haloalkoxy, C₂-C₄ alkoxyalkoxy, CO₂R¹², C(O)NR¹³R¹⁴,
SO₂NR¹⁵R¹⁶, S(O)ₙR¹⁷, C(O)R¹⁸, CH₂CN and L;
R² is selected from the group H, F, Cl, Br, CN, CH₃, OCH₃, SCH₃, CF₃
and OCF₂H;
R³ is selected from the group Cl, NO₂, CO₂CH₃, CO₂CH₂CH₃,
SO₂N(CH₃)₂, SO₂CH₃, SO₂CH₂CH₃, OCH₃ and OCH₂CH₃
R⁴ is selected from the group C₁-C₃ alkyl, C₁-C₂ haloalkyl, C₁-C₂ alkoxy,
C₂-C₄ haloalkenyl, F, Cl, Br, NO₂, CO₂R¹², C(O)NR¹³R¹⁴,
SO₂NR¹⁵R¹⁶, S(O)ₙR¹⁷, C(O)R¹⁸ and L;
R⁵ is selected from the group H, F, Cl, Br and CH₃;
R⁶ is selected from the group C₁-C₃ alkyl, C₁-C₂ alkoxy, C₂-C₄
haloalkenyl, F, Cl, Br, CO₂R¹², C(O)NR¹³R¹⁴, SO₂NR¹⁵R¹⁶,
S(O)ₙR¹⁷, C(O)R¹⁸ and L;
R⁷ is selected from the group H, F, Cl, CH₃ and CF₃;
R⁸ is selected from the group H, C₁-C₃ alkyl and pyridyl;
R⁹ is selected from the group C₁-C₃ alkyl, C₁-C₂ alkoxy, F, Cl, Br, NO₂,
CO₂R¹², SO₂NR¹⁵R¹⁶, S(O)ₙR¹⁷, OCF₂H, C(O)R¹⁸, C₂-C₄
haloalkenyl and L;
R₁₀ is selected from the group H, Cl, F, Br, C₁-C₃ alkyl and C₁-C₂ alkoxy;
R₁¹ is selected from the group H, C₁-C₃ alkyl, C₁-C₂ alkoxy, C₂-C₄
haloalkenyl, F, Cl, Br, CO₂R₁², C(O)NR¹³R¹⁴, SO₂NR¹⁵R¹⁶,
S(O)ₙR¹⁷, C(O)R¹⁸ and L;
R₁² is selected from the group allyl and propargyl and C₁-C₃ optionally
substituted by at least one member independently selected from
halogen, C₁-C₂ alkoxy and CN;
R₁³ is selected from the group H, C₁-C₃ alkyl and C₁-C₂ alkoxy;
R₁⁴ is C₁-C₂ alkyl;
R₁⁵ is selected from the group H, C₁-C₃ alkyl, C₁-C₂ alkoxy, allyl and
cyclopropyl;
R₁⁶ is selected from the group H and C₁-C₃ alkyl;
R₁⁷ is selected from the group C₁-C₃ alkyl, C₁-C₃ haloalkyl, allyl and
propargyl;
R₁⁸ is selected from the group C₁-C₄ alkyl, C₁-C₄ haloalkyl and C₃-C₅
cycloalkyl optionally substituted by halogen;
n is 0, 1 or 2;
M is a cation;
L is

\[
\text{R}_j
\]

R₂₀ is selected from the group H and C₁-C₃ alkyl;
W is selected from the group O and S;
X is selected from the group H, C₁-C₄ alkyl, C₁-C₄ alkoxy, C₁-C₄
haloalkoxy, C₁-C₄ haloalkyl, C₁-C₄ haloalkythio, C₁-C₄ alkythio,
halogen, C₂-C₅ alkoxyalkyl, C₂-C₅ alkoxyalkoxy, amino, C₁-C₃
alkylamino and di(C₁-C₃ alkyl)amino;
Y is selected from the group H, C₁-C₄ alkyl, C₁-C₄ alkoxy, C₁-C₄
haloalkoxy, C₁-C₄ alkythio, C₁-C₄ haloalkythio, C₂-C₅ alkoxyalkyl,
C₂-C₅ alkoxyalkoxy, amino, C₁-C₃ alkylamino, di(C₁-C₃
alkyl)amino, C₃-C₄ alkenyloxy, C₃-C₄ alkynyloxy, C₂-C₅
alkylthioalkyl, C₂-C₅ alkylsulfinylalkyl, C₂-C₅ alkylsulfonylalkyl,
C₁-C₄ haloalkyl, C₂-C₄ alkylnyl, C₃-C₅ cycloalkyl, azido and cyano; and

Z is selected from the group CH and N;

provided that i) when one or both of X and Y is C₁ haloalkoxy, then Z is CH; and ii) when X is halogen, then Z is CH and Y is OCH₃, OCH₂CH₃, N(OCH₂)₂CH₃, NHCH₃, N(CH₃)₂ or OCF₃H.

Preferred active ingredients are salts of the following sulfonylureas: chlorsulfuron; sulfometuron; chlorimuron ethyl; metsulfuron methyl; methyl 2-[[[(4,6-dimethoxy-2-pyrimidinyl)-amino]carbonyl]-amino]sulfonyl]-6-(trifluoromethyl)-3-pyridinecarboxylate; ethamsulfuron methyl; triasulfuron; ethyl 5-[[[(4,6-dimethoxy-2-pyrimidinyl)amino]carbonyl]-amino]sulfonyl]-1-methyl-1H-pyrazole-4-carboxylate; N-[[4,6-dimethoxy-2-pyrimidinylamino]carbonyl]-3-(ethylsulfonyl)-2-pyridinesulfonamide; thifensulfuron; tribenuron methyl; bensulfuron methyl; nicosulfuron; methyl 2-[[[[4,6-bis(difluoromethoxy)-2-pyrimidinyl]amino]carbonyl]amino]sulfonyl]-benzoate; methyl 2-[[[[4-dimethylamino]-6-(2,2,2-trifluoroethoxy)-1,3,5-triazin-2-yl]amino]-carbonyl]amino]sulfonyl]-3-methylbenzoate; and N-[[4,6-dimethoxy-2-pyrimidinyl]amino]carbonyl]-1-methyl-4-(2-methyl-2H-tetrazol-5-yl)-1H-pyrazole-5-sulfonamide.

Preferred salts are the sodium, potassium, calcium, magnesium, ammonium and alkylammonium salts of a sulfonylurea. Most preferred sulfonylurea salts are the sodium and calcium salts of tribenuron methyl, the potassium salt of thifensulfuron methyl, the ammonium salt of chlorsulfuron and the potassium salt of metsulfuron methyl.

DETAILED DESCRIPTION OF THE INVENTION

The most common method for applying pesticides is as aqueous solutions or dispersions which are sprayed onto the field or crop using ground or aerial spray rigs. A tablet containing the pesticidal component is an effective form for introducing the pesticide into the water in the spray tank. It is substantially impossible to obtain rapid break-up of a tablet without the use of effervescence. Rapid break-up in water is desirable for the convenience of the growers who require quick turnaround times for the preparation of the spray solutions and dispersions.

Known effervescent pesticide tablets comprise a water-insoluble pesticide, an organic acid and a carbonate or bicarbonate base. The acid and base react in
an aqueous environment to produce carbon dioxide gas which aids in the break-up of the tablet and dispersion of the pesticide. However, the rate of the acid-base effervescent reaction slows significantly when the tablet comprises a water-soluble pesticide. A soft "hydrogel" is believed to form around the tablet to inhibit water from contacting the tablet and facilitating the reaction. Disintegration rates are therefore too slow for practical applications. In cases where the pesticide is a water-soluble metal salt, the acid in the tablet may react with the pesticide to give the water-insoluble acid form.

No hydrogel formation or precipitate is observed in the reaction of the tablets of the present invention with water. Effervescence begins instantaneously and complete disintegration occurs in less than 10 minutes, most often in less than 5 minutes using even the cold water drawn from wells in the early spring. A clear aqueous solution forms with the pesticide homogeneously dissolved therein.

Inert ingredients up to about 74.9% of the total weight of the composition can be employed. Such inert ingredients are components, complementary to the pesticide, which are known in the tablet art to improve tablet disintegration rate, dispersibility, stability during storage, and the like. Examples of optional components include a manganese, copper or iron salt catalyst; a dispersant; a disintegrant; an anionic or nonionic wetting agent; a flow aid, and a desiccant. The amounts and types of such ingredients will be readily determinable by one skilled in the tabletting art given the disclosure herein.

The effervescent reaction is due to the anhydrous metal perborate reacting with the water to liberate oxygen. Any such compound which is compatible with the pesticide and liberates oxygen upon hydration is suitable for the tablets of the present invention. The preferred compound is anhydrous sodium perborate (also known as sodium peroxymonoborate, NaBO₃).

Metal perborate is commonly available commercially as the monohydrate. The monohydrate must be converted to its anhydrous state in order for the effervescent reaction to occur. The conversion of the metal perborate monohydrate to its anhydrous form can be accomplished by oven-drying the granules at 135°C for 24 hours in a vacuum oven with a nitrogen bleed to obtain a pressure of 1.33 x 10³ Pascal. The sodium perborate is spread in a layer less than 2 cm in thickness to facilitate drying. As a result of the drying, sodium perborate has been found to change color from white to yellow.
The success of the drying procedure can be tested by blending the dried sample to a homogenous mixture, and then dropping a small amount of the mixture into a beaker of water. If all the material reacts (i.e., effervesces) on the surface of the water, and no residue (i.e., monohydrate) falls to the bottom of the beaker, then the anhydrous state was achieved.

If small amounts of the monohydrate perborate are not converted to the anhydrous form during drying, a metal salt can be added in catalytic amounts to decompose the monohydrate to produce oxygen and other products. Monohydrate which is not destroyed by a catalyst will not effervesce when contacted with water, but it does dissolve within the time necessary for disintegration of the tablet (i.e., less than 10 minutes). Preferred catalyst salts include manganese, copper or iron metal oxides or carbonates. Most preferred is iron (II) oxide.

Dispersants can be added to aid the initial dispersion of the particles of the active ingredient which are liberated during disintegration of the tablet. Suitable dispersants include sodium, potassium and calcium salts of naphthalene sulfonic acid formaldehyde condensates; lithium, sodium, potassium, calcium, and ammonium salts of lignosulfonates such as Polyfon H® and Lignosol TSF®; sodium, potassium and ammonium salts of polyacrylates and carboxylates, e.g., Tamol 731 SD®, sodium salts of maleic anhydride-isobutylene copolymers; and water soluble nonionic polymers such as polyvinylpyrrolidone, polyethylene oxides and cellulose derivatives. Preferred dispersants include the sodium, potassium, ammonium and calcium salts of naphthalene sulfonic acid formaldehyde condensates, with the ammonium salts, specifically Lomar PWA®, more preferred.

Disintegrants facilitate penetration of the water into the interior of the tablet through a wicking or swelling action. Water-insoluble cross-linked polyvinylpolypyrrolidone is a preferred disintegrant.

A wetting agent can be used to control the size of the oxygen bubbles formed during the acid-base reaction. The anionic wetting agents include alkylbenzene sulfonates, alkyl and dialkynaphthalene sulfonates, alkyl and alcohol sulfates, sulfoalkylamides, carboxylates, alpha-olefin sulfonates and dialkyl sulfosuccinates. The nonionic wetting agents include acetylenic diols, ethylene oxide-propylene oxide copolymers, alkylphenol ethoxylates, tristyrlphenol ethoxylates, fatty acid ethoxylates, alcohol ethoxylates, sorbitan
fatty acid ester ethoxylates and castor oil ethoxylates. The preferred wetting agents are sodium dialkyl sulfosuccinates of which sodium diisobutyl sulfosuccinate (Monawet® MB-100), sodium diamy1 sulfosuccinate and sodium dicyclohexyl sulfosuccinate are more preferred.

Flow aids can be added to facilitate transfer of the dry ingredients from the feed hopper to the tablet die. Suitable flow aids include silica and diatomaceous earth.

A dessicant is another optional component of the formulation of the invention. As indicated in Example 2 below, a tablet in a sealed container without a dessicant remains effervescent after storage. However, if a dessicant is desirable, it can be external to the tablet, or incorporated into the tablet matrix. Internal desiccants can be those which "chemically bind" water in that they undergo chemical reactions with water to form a new compound. An example of this type of material is CaO which reacts with water to form Ca(OH)₂. Other materials representative of those which react in this manner are magnesium oxide and boric anhydride.

The internal desiccant can also be of the type which "physically adsorb" water and are selected from the group consisting of highly-dispersed silicic acids such as silica gel; aluminum oxide; clays such as montmorillonite; and amorphous and crystalline aluminosilicates such as molecular sieves and zeolites.

Combinations of these desiccants with those that form hydroxides and hydrates can be used. Kirk-Othmer's Encyclopedia of Chemical Technology (3rd ed., Vol. 8, p 115) describes desiccants suitable for use in the tablet formulation of this invention as Type 1 and Type 4 desiccants. Either type can be employed, singly or in combination, as long as the desiccant does not expand when it picks-up water. Such expansion causes the tablet to crack or crumble on long term storage.

Internal desiccants suitable for the tablet formulation of this invention also include materials that chemically bind water, not in the sense of a chemical reaction that forms a hydroxide, but in the sense of a chemical reaction that produces a hydrate. Representative of useful desiccants that form hydrates are CaSO₄, NaOAc, MgSO₄, Na₂SO₄, CaCl₂, and ZnSO₄. Representative of the hydrate-forming reaction is that undergone by CaCl₂ to form CaCl₂·6H₂O. One or more desiccants from each group, the hydroxide-forming and the hydrate-forming, can be employed, alone or in combination, depending on the particular properties sought by the formulator.
In addition, inert fillers such as sugar or clay can be added as long as they do not affect the chemical stability of the active ingredient(s). Materials such as glidants, anti-adherents, and lubricants can also be employed to facilitate production in the tablet press. For example, lubricants such as magnesium stearate or boric acid can be used. Such lubricants and anti-adherents can be brushed onto the die surface or incorporated into the formulation.

The tablets are typically prepared in the following manner. The solid watersoluble pesticide is passed through a 30 mesh screen to remove oversized particles. Granular anhydrous sodium perborate prepared as described above is blended with the pesticide and, if desired, the inert ingredients. The blend can be milled, e.g., in an air or hammermill, or compacted into tablet form without milling. Blends which are not milled and thereby comprise larger particle sizes are desirable for rapid tablet break-up.

The tablets can be prepared using conventional tablet-making equipment. Their diameter can vary from about 1 cm or less, to 7.5 cm, depending on the tablet weight desired. Flat-faced, beveled-edge punches, with or without a breakline, produce attractive tablets.

Tablets are formed in a hydraulic press with a capacity of 18,000 kg of force. Pressures between about 3.43x10 \(^7\) to 6.86x10 \(^7\) Pascals produce tablets which remain intact during storage and handling and break-up rapidly. Break-up times are determined by dropping a tablet, typically 7.5 g into 1000 mL of water. The "end point" of final dissolution is determined by the cessation of the effervescent reaction.

The tablets described in the following Examples were 3.5 cm in diameter, and were made with a hand-operated hydraulic press at a pressure of 525 kg/cm\(^2\).

**EXAMPLES 1-4**

The following ingredients were milled for 1 minute in a CRC\(^\circ\) analytical laboratory mill.

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<th>Ingredient</th>
<th>Example 1</th>
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<td>tribenuron methyl, sodium salt</td>
<td>4.00</td>
<td>4.00</td>
<td>4.00</td>
<td>4.00</td>
</tr>
<tr>
<td>anhydrous sodium perborate</td>
<td>4.00</td>
<td>4.00</td>
<td>3.52</td>
<td>4.00</td>
</tr>
<tr>
<td>dessicant molecular sieves</td>
<td>external</td>
<td>-</td>
<td>0.48</td>
<td>-</td>
</tr>
<tr>
<td>CaSO(_4)</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>external</td>
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The milled ingredients, referred to hereinafter as the premixes, were blended well and aged for 3 weeks at 45°C in sealed glass jars. In Examples 1 and 4, the molecular sieves and CaSO₄ were packaged separately and added to the glass jars as external dessicants. The jar containing the premix of Example 2 contained no dessicant, and the premix of Example 3 had the molecular sieves incorporated into the formulation. The premixes were then cooled, and 7.5 g of each premix was tabletted. The tablets were added to water and dissolution times were measured. The resulting aqueous mixtures were passed through a stack of 50, 100, and 200 mesh screens. Particulate residue was determined by visual inspection of the screens.

<table>
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<th>Example</th>
<th>Dissolution time (min.)</th>
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<td>1</td>
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<td>2</td>
<td>4.32</td>
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</tr>
<tr>
<td>3</td>
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<td>Light¹</td>
</tr>
<tr>
<td>4</td>
<td>4.88</td>
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¹ The color suggested the residue was the molecular sieves.

**EXAMPLE 5**

The following ingredients were blended without a milling step.

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<th>Ingredient</th>
<th>Weight (g)</th>
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<td>tribenuron methyl, sodium salt</td>
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<tr>
<td>anhydrous sodium perborate</td>
<td>4.00</td>
</tr>
</tbody>
</table>

A tablet was made from 7.5 grams of the premix. The tablet was added to water and the dissolution time was measured. The resulting aqueous mixture was passed through a stack of 50, 100, and 200 mesh screens. No residue was found upon visual inspection of the screens.

<table>
<thead>
<tr>
<th>Example</th>
<th>Dissolution time (min.)</th>
<th>Residue Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>3.80</td>
<td>None</td>
</tr>
</tbody>
</table>
EXAMPLES 6 AND 7

The following ingredients were blended, milled and made into a tablet as described in Examples 1-4.

<table>
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<tr>
<th>Ingredient</th>
<th>Example 6</th>
<th>Example 7</th>
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<tr>
<td>tribenuron methyl, sodium salt</td>
<td>4.00</td>
<td>4.00</td>
</tr>
<tr>
<td>anhydrous sodium perborate</td>
<td>3.98</td>
<td>3.98</td>
</tr>
<tr>
<td>catalyst</td>
<td></td>
<td></td>
</tr>
<tr>
<td>cupric carbonate</td>
<td>0.02</td>
<td>-</td>
</tr>
<tr>
<td>cupric oxide</td>
<td>-</td>
<td>0.02</td>
</tr>
</tbody>
</table>

Tablets were made from 7.5 grams of the premixes. The tablets were added to water and the dissolution times were measured. The resulting aqueous mixtures were passed through a stack of 50, 100, and 200 mesh screens. Particulate residue was determined by visual inspection of the screens.

<table>
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<tr>
<th>Example</th>
<th>Dissolution time (min.)</th>
<th>Residue Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>4.25</td>
<td>None(^1)</td>
</tr>
<tr>
<td>7</td>
<td>4.92</td>
<td>None</td>
</tr>
</tbody>
</table>

\(^1\)One thin undissolved flake was observed, and it passed through the screens.
What is claimed is:

1. A tablet formulation comprising, by total weight, about (1) 0.1% to 75% of a water-soluble pesticide that has a melting point of at least 75°C and a solubility in pH 7 water at 20°C of at least about 2% by weight, and (2) 25% to 99.9% of an anhydrous metal perborate salt selected from the group sodium, lithium, and potassium perborate having a water content less than about 2% by weight.

2. A tablet according to Claim 1 comprising 10 to 70% of a pesticide and 30 to 90% of a perborate salt.

3. A tablet according to Claim 2 comprising 30 to 60% of a pesticide and 40 to 70% of a perborate salt.

4. A tablet formulation according to Claim 1 wherein the pesticide is selected from at least one member of the group herbicides, fungicides, bactericides, and insecticides.

5. A tablet formulation according to Claim 4 wherein the pesticide is a sulfonylurea salt having the formula

\[
\begin{array}{c}
W \\
\begin{array}{c}
\text{J-SO}_{2}\text{NCHR}
\end{array} \\
\bigcirc \\
\text{N} \\
\begin{array}{c}
\text{Z}
\end{array} \\
\begin{array}{c}
\text{X}
\end{array} \\
M^+
\end{array}
\]

\[
\begin{array}{c}
\text{J-1} \\
\text{J-2} \\
\text{J-3} \\
\text{J-4} \\
\text{J-5} \\
\text{J-6}
\end{array}
\]

wherein J is selected from the group
R is selected from the group H and CH₃;
R¹ is selected from the group F, Cl, Br, NO₂, C₁₋C₄ alkyl, C₁₋C₄ haloalkyl,
C₃₋C₄ cycloalkyl, C₂₋C₄ haloalkenyl, C₁₋C₄ alkoxy, C₁₋C₄
haloalkoxy, C₂₋C₄ alkoxyalkoxy, CO₂R¹₂, C(O)NR¹³R¹⁴,
SO₂NR¹⁵R¹⁶, S(O)ₙR¹⁷, C(O)R¹⁸, CH₂CN and L;
R² is selected from the group H, F, Cl, Br, CN, CH₃, OCH₃, SCH₃, CF₃
and OCF₂H;
R³ is selected from the group Cl, NO₂, CO₂CH₃, CO₂CH₂CH₃,
SO₂N(CH₃)₂, SO₂CH₃, SO₂CH₂CH₃, OCH₃, and OCH₂CH₃
R⁴ is selected from the group C₁₋C₃ alkyl, C₁₋C₂ haloalkyl, C₁₋C₂ alkoxy,
C₂₋C₄ haloalkenyl, F, Cl, Br, NO₂, CO₂R¹₂, C(O)NR¹³R¹⁴,
SO₂NR¹⁵R¹⁶, S(O)ₙR¹⁷, C(O)R¹⁸ and L;
R⁵ is selected from the group H, F, Cl, Br and CH₃;
R^6 is selected from the group C_1-C_3 alkyl, C_1-C_2 alkoxy, C_2-C_4 haloalkenyl, F, Cl, Br, CO_2R^{12}, C(O)NR^{13}R^{14}, SO_2NR^{15}R^{16}, S(O)R^{17}, C(O)R^{18} and L;
R^7 is selected from the group H, F, Cl, CH_3 and CF_3;
R^8 is selected from the group H, C_1-C_3 alkyl and pyridyl;
R^9 is selected from the group C_1-C_3 alkyl, C_1-C_2 alkoxy, F, Cl, Br, NO_2, CO_2R^{12}, SO_2NR^{15}R^{16}, S(O)NR^{17}, OCF_2H, C(O)R^{18}, C_2-C_4 haloalkenyl and L;
R^{10} is selected from the group H, Cl, F, Br, C_1-C_3 alkyl and C_1-C_2 alkoxy;
R^{11} is selected from the group H, C_1-C_3 alkyl, C_1-C_2 alkoxy, C_2-C_4 haloalkenyl, F, Cl, Br, CO_2R^{12}, C(O)NR^{13}R^{14}, SO_2NR^{15}R^{16}, S(O)R^{17}, C(O)R^{18} and L;
R^{12} is selected from the group allyl and propargyl and C_1-C_3 optionally substituted by at least one member independently selected from halogen, C_1-C_2 alkoxy and CN;
R^{13} is selected from the group H, C_1-C_3 alkyl and C_1-C_2 alkoxy;
R^{14} is C_1-C_2 alkyl;
R^{15} is selected from the group H, C_1-C_3 alkyl, C_1-C_2 alkoxy, allyl and cyclopropyl;
R^{16} is selected from the group H and C_1-C_3 alkyl;
R^{17} is selected from the group C_1-C_3 alkyl, C_1-C_3 haloalkyl, allyl and propargyl;
R^{18} is selected from the group C_1-C_4 alkyl, C_1-C_4 haloalkyl and C_3-C_5 cycloalkyl optionally substituted by halogen;
n is 0, 1 or 2;
M is a cation;
L is

```
N--N
|   |
N--N
```

R_j is selected from the group H and C_1-C_3 alkyl;
W is selected from the group O and S;
X is selected from the group H, C_1-C_4 alkyl, C_1-C_4 alkoxy, C_1-C_4 haloalkoxy, C_1-C_4 haloalkyl, C_1-C_4 haloalkylthio, C_1-C_4 alkylthio,
halogen, C₂⁻C₅ alkoxyalkyl, C₂⁻C₅ alkoxyalkoxy, amino, C₁⁻C₃ alkylamino and di(C₁⁻C₃ alkyl)amino;

Y is selected from the group H, C₁⁻C₄ alkyl, C₁⁻C₄ alkoxy, C₁⁻C₄ haloalkoxy, C₁⁻C₄ alkylthio, C₁⁻C₄ haloalkylthio, C₂⁻C₅ alkoxyalkyl, C₂⁻C₅ alkoxyalkoxy, amino, C₁⁻C₃ alkylamino, di(C₁⁻C₃ alkyl)amino, C₃⁻C₄ alkenyloxy, C₃⁻C₄ alkynyloxy, C₂⁻C₅ alkylthioalkyl, C₂⁻C₅ alkylsulfinylalkyl, C₂⁻C₅ alkylsulfonylalkyl, C₁⁻C₄ haloalkyl, C₂⁻C₄ alkenyl, C₃⁻C₅ cycloalkyl, azido and cyano; and

Z is selected from the group CH and N;

provided that i) when one or both of X and Y is C₁ haloalkoxy, then Z is CH; and ii) when X is halogen, then Z is CH and Y is OCH₃, OCH₂CH₃, N(OCH₃)CH₃, NHCH₃, N(CH₃)₂ or OCF₂H.

6. A tablet formulation according to Claim 5 wherein the metal cation of the sulfonylurea salt is selected from the group sodium, potassium, calcium, magnesium, ammonium and alkylammonium.

7. A tablet formulation according to Claim 6 wherein the sulfonylurea is selected from the group: chlorsulfuron; sulfometuron; chlorimuron ethyl; metsulfuron methyl; methyl 2-[(4,6-dimethoxy-2-pyrimidinyl)-amino][carbonyl]-amino][sulfonyl]-6-(trifluoromethyl)-3-pyridinecarboxylate; ethametsulfuron methyl; triasulfuron; ethyl 5-[(4,6-dimethoxy-2-pyrimidinyl)amino][carbonyl]-amino][sulfonyl]-1-methyl-1H-pyrazole-4-carboxylate; N-[(4,6-dimethoxy-2-pyrimidinylamino)[carbonyl]-3-(ethylsulfonyl)-2-pyridinesulfonamide; thifensulfuron; tribenuron methyl; bensulfuron methyl; nicosulfuron; methyl 2-[[4,6-bis(difluoromethoxy)-2-pyrimidinyl]amino][carbonyl][amino][sulfonyl]-benzoate; methyl 2-[[4-dimethylamino]-6-(2,2,2-trifluoroethoxy)-1,3,5-triazin-2-yl]amino][carbonyl][amino][sulfonyl]-3-methylbenzoate; and N-[(4,6-dimethoxy-2-pyrimidinyl)amino][carbonyl]-1-methyl-4-(2-methyl-2H-tetrazol-5-yl)-1H-pyrazole-5-sulfonamide.

8. A tablet formulation according to Claim 1 in the form of a tablet.
A. CLASSIFICATION OF SUBJECT MATTER

IPC 5 A01N25/34 A01N47/36 //A01N25/34,47:36), (A01N47/36, 25:34)

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 A01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
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<tbody>
<tr>
<td>X</td>
<td>GB,A,1 424 084 (E.R.HOLLOWAY) 4 February 1976 see the whole document</td>
<td>1-4, 8</td>
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<tr>
<td>Y</td>
<td>WO,A,90 00007 (E.I.DU PONT DE NEMOURS) 11 January 1990 cited in the application</td>
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<td>A</td>
<td>EP,A,0 081 962 (WARNER-LAMBERT) 22 June 1983 see the whole document</td>
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<td>GB,A,2 095 694 (E.R.HOLLOWAY) 6 October 1982 see the whole document</td>
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Further documents are listed in the continuation of box C. Patent family members are listed in annex.

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"E" earlier document but published on or after the international filing date

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"F" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search: 30 March 1994

Date of mailing of the international search report: 19. 04. 94

Name and mailing address of the ISA:
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NL - 2280 HV Rijswijk
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Fax (+31-70) 340-3016

Authorized officer: Lamers, W
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<td>US,A,3 421 842 (L.R.DARBEE ET AL.) 14 January 1969 see column 4, line 22 - line 30 -----</td>
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