

# TECHNICAL IODOFENPHOS

Specification WHO/SIT/22.R3

Revised 10 December 1999

## 1. Specification

### 1.1 Description

The material shall consist of iodofenphos together with related manufacturing impurities and shall be in the form of a white crystalline solid free from visible extraneous matter and added modifying agents.

### 1.2 Chemical and physical requirements

The material, sampled from any part of the consignment (see method WHO/M/1.R1), shall comply with the requirements of section 1.1 and with the following requirements.

#### 1.2.1 *Identity test*

Where the identity of the active ingredient is in doubt, then it shall comply with at least one additional test.

#### 1.2.2 *Iodofenphos content (g/kg basis)*

The iodofenphos content shall be declared (not less than 910 g/kg) and, when determined by the method described in section 2.1, the mean measured content shall not be lower than the declared content.

#### 1.2.3 *Acidity*

The acidity of the material, determined by the CIPAC method MT 31 (CIPAC Handbook F, p.96), shall not be higher than 3 g/kg, calculated as H<sub>2</sub>SO<sub>4</sub>.

#### 1.2.4 *Material insoluble in acetone*

The material insoluble in acetone as determined by the CIPAC method MT 27 (CIPAC Handbook F, p.88), shall not be higher than 5 g/kg.

#### 1.2.5 *Water content*

The water content, determined by the method described in section 2.2, shall not be higher than 5 g/kg.

### 1.3 Packing and marking of packages

The technical iodofenphos shall be packed in suitable clean containers, as specified in the order. All packages shall bear, durably and legibly marked on the container the following:

Manufacturer's name  
Technical iodofenphos  
Batch or reference number, and date of test  
Net weight of contents  
Date of manufacture

and the following minimum cautionary notice:

Iodofenphos is an organophosphorous compound that inhibits cholinesterase. It may be hazardous if swallowed. Do not inhale spray mist. Wash hands and exposed skin thoroughly after using.

Keep the material out of the reach of children and well away from foodstuffs and animal feed and their containers.

If poisoning occurs, call a physician. Atropine and pralidoxime are specific antidotes, and artificial respiration may be needed.

## 2. Methods of determining chemical and physical properties

### 2.1 Iodofenphos content

#### 2.1.1 *Outline of method*

The sample is dissolved in glacial acetic acid and oxidized with an excess of acidified standard bromide-bromate solution. The excess bromate is determined by adding potassium iodide and titrating the liberated iodine with standard sodium thiosulfate solution.

#### 2.1.2 *Reagents*

*Standard bromide-bromate solution* containing 0.1 mol/L potassium bromide and 1/60 mol/L potassium bromate.

*Standard sodium thiosulfate solution* 1/10 mol/L

*Potassium iodide solution* 200 g/L

*Aqueous starch solution* 10 g/L

#### 2.1.3 *Procedure*

Weigh (to the nearest 0.1 mg) about 175 mg of the sample into a 300 mL iodine flask. Add 10 mL of glacial acetic acid followed by 5 mL of concentrated hydrochloric acid (relative density 1.19). Mix the contents well and add exactly 50 mL of the standard bromide-bromate solution. The yellow colour that forms should persist; if it does not, add an additional 25 mL of the standard bromide-bromate solution. Allow to stand to 2 minutes.

Add 50 mL of distilled water and 10 mL of the potassium iodide solution. Stopper the flask, mix the contents well, and allow the mixture to stand for about 3 minutes. Titrate the liberated iodine with the standard 0.1 mol/L sodium thiosulfate solution, using starch solution as indicator near the end-point. Carry out a blank determination with 25 mL of standard bromide-bromate solution.

#### 2.1.4 Calculation

$$\text{Iodofenphos content (g/kg)} = \frac{(2v_2 - v_1) \times c \times 51.63}{m}$$

Where  $v_1$  = volume (mL) of 0.1 mol/L sodium thiosulfate required for the sample

$v_2$  = volume (mL) of 0.1 mol/L sodium thiosulfate required for the blank

$m$  = mass (g) of the sample

$c$  = substance concentration (mol/L) of standard sodium thiosulfate.

## 2.2 Water content

Determine the water content by the Karl Fischer electrometric titration method (WHO/M/7.R1) or by the Dean and Stark distillation method (CIPAC method MT 30.2, CIPAC Handbook F, p.94). The latter may not always be practicable owing to its unreliability at very low water contents. In case of dispute, the Karl Fischer method shall be the referee.

# IODOFENPHOS WETTABLE POWDER

Specification WHO/SIF/33.R3  
Revised 10 December 1999

## 1. Specification

### 1.1 Description

The material shall consist of a homogeneous mixture of technical iodofenphos, complying with the requirements of the WHO specification WHO/SIT/22.R3, in the form of a fine, free-flowing powder that wets out readily on stirring into water, together with filler(s) and any other necessary formulants. It shall be free from visible extraneous matter and hard lump.

### 1.2 Chemical and physical requirements

The material sampled from any part of the consignment (see method WHO/M/1.R1), shall comply with the requirements of section 1.1 and with the following requirements.

#### 1.2.1 *Iodofenphos content (g/kg basis)*

The content of iodofenphos determined by the method described in section 2.1, shall not differ from the declared content by more than the following amounts:

<i>Declared content</i>	<i>Tolerance permitted</i>
Above 250 up to 500 g/kg	$\pm 5\%$ of the declared content
Above 500 g/kg	$\pm 25$ g/kg

The average content of all samples taken shall not be lower than the declared content.

#### 1.2.2 *Sieving after heat stability treatment*

Not less than 98% of the powder after heat stability treatment (section 2.3) shall pass through a 75  $\mu\text{m}$  sieve when tested by the CIPAC method MT 59.3 (CIPAC Handbook F, p.179).

#### 1.2.3 *Suspensibility after heat stability treatment*

*In WHO standard hard water* . When tested by the method described in section 2.2, a minimum of 50% of the iodofenphos (12.5 g/L) shall be in suspension 30 minutes after agitating a suspension containing 25 g/L of iodofenphos, prepared in WHO standard hard water from the powder subjected to the heat stability treatment described in section 2.3.

#### 1.2.4 *Persistent foam*

*In WHO standard soft water (WHO/M/29)*. When tested by the method CIPAC MT 47.2 (CIPAC Handbook F, p.152) a maximum of 60 mL of foam shall be observed after 1 minute.

#### 1.2.5 *Wettability after heat stability treatment*

*In WHO standard hard water (WHO/M/29)*. When tested by the method CIPAC MT 53.3 (CIPAC Handbook F, p.164) the formulation shall be completely wetted in 2 min. without swirling

#### 1.2.6 *Heat stability*

The powder after treatment as described in section 2.3, shall comply with the requirements of section 1.2.1 of this specification.

### **1.3 Packing and marking of packages**

The iodofenphos wettable powder shall be packed in suitable, clean drums, as specified in the order. The drums shall contain a lining or bag of polyethylene or equivalent, with a nominal thickness of 0.1 mm. The lining or bag shall be hermetically sealed after filling.

All packages shall bear, durably and legibly marked on the container, the following:

Manufacturer's name  
Iodofenphos wettable powder  
Iodofenphos ... g/kg  
Batch or reference number, and date of test  
Net weight of contents  
Date of formulation  
Instruction for use

and the following minimum cautionary notice:

Iodofenphos is an organophosphorous compound that inhibits cholinesterase. It may be hazardous if swallowed. Do not inhale spray mist. Wash hands and exposed skin thoroughly after using.

Keep the material out of the reach of children and well away from foodstuffs and animal feed and their containers.

If poisoning occurs, call a physician. Atropine and pralidoxime are specific antidotes, and artificial respiration may be needed.

## **2. Methods of determining chemical and physical properties**

### **2.1 Iodofenphos content**

#### *2.1.1 Outline of method*

The sample is mixed with glacial acetic acid and an aliquot is oxidized with an excess of acidified standard bromide-bromate solution. The excess of bromate is determined by adding potassium iodide and titrating the liberated iodine with standard sodium thiosulfate solution.

#### *2.1.2 Reagents*

*Standard bromide-bromate solution* 1/60 mol/L potassium bromate in 0.1 mol/L potassium bromide

*Standard sodium thiosulfate solution* 1/10 mol/L

*Potassium iodide solution* 200 g/L

*Aqueous starch solution* 10 g/L

#### *2.1.3 Procedure*

Weigh (to the nearest 0.1 mg) an amount of the sample containing about 0.8 g of iodofenphos and disperse in glacial acetic acid in a 100 mL volumetric flask. Stopper the flask, shake well, make up to the mark with glacial acetic acid, and mix again. Allow the filler<sup>1</sup> to settle and pipette 20 mL of the clear solution into a 300 mL iodine flask. Add 5 mL of concentrated hydrochloric acid, mix well, and add exactly 50 mL of the standard bromide-bromate solution. The yellow colour that forms should persist; if it does not, add a further 25 mL of the standard bromide-bromate solution. Allow to stand for 2 minutes.

Add 50 mL of distilled water and then 10 mL of the solution of potassium iodide in water. Stopper the flask, mix the contents well, and allow the mixture to stand for 3 minutes. Titrate the liberated iodine with the standard 0.1 mol/L sodium thiosulfate solution, using starch solution as indicator near the end-point. Carry out a blank determination with 25 mL of standard bromide-bromate solution.

#### *2.1.4 Calculation*

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<sup>1</sup> A correction for the volume occupied by the filler should be made whenever this volume is appreciable.

$$\text{Iodofenphos content (g/kg)} = \frac{(2v_2 - v_1) \times c \times 252.15}{m}$$

Where  $v_1$  = volume (mL) of standard sodium thiosulfate required for the sample  
 $v_2$  = volume (mL) of standard sodium thiosulfate used for the blank  
 $m$  = mass (g) of the sample  
 $c$  = substance concentration (mol/L) of standard sodium thiosulfate.

## 2.2 Suspensibility

### 2.2.1 Outline of method

A suspension of known concentrate of iodofenphos in WHO standard hard water is prepared, poured into a 250 mL graduated cylinder maintained at a constant temperature, and allowed to remain undisturbed for 30 minutes. The top 9/10 ths are drawn off and the content of iodofenphos in the bottom 1/10 th is determined, so allowing to evaluate the active ingredient mass still in suspension after 30 minutes.

### 2.2.2 Apparatus

1. A 250 mL graduated cylinder with a ground-glass stopper and a distance of 20-21.5 cm between the bottom and the 250 mL calibration mark.
2. A glass tube, about 40 cm long and about 5 mm in internal diameter, pointed at one end of an opening of 2-3 mm, the other end being connected to a suitable source of suction.

### 2.2.3 Reagents

WHO standard hard water. See WHO method WHO/M/29.

### 2.2.4 Procedure

Weigh (to the nearest 10 mg) into a 100 mL beaker an amount of the sample to form 250 mL of a suspension containing 25 g/L of iodofenphos. Add a volume of water<sup>2</sup> at  $30^{\circ}\text{C} \pm 2^{\circ}\text{C}$  equal to at least twice the mass of the sample taken. Allow to stand to 30 seconds and then stir by hand for 30 seconds with a glass rod, 4-6 mm diameter, at not more than 4 revolutions per second, making no deliberate attempt to break up any lumps. Then immediately transfer the mixture quantitatively to the 250 mL graduated cylinder, using water at  $30 \pm 2^{\circ}\text{C}$  for rinsing, and again avoiding mechanical disintegration of any lumps. Immediately add sufficient water at  $30 \pm 2^{\circ}\text{C}$  to bring the

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<sup>2</sup> Whenever water is mentioned in this section, use WHO standard hard water.

volume to the 250 mL mark. Insert the stopper and invert the cylinder end over end 30 times at the rate of one complete cycle every 2 seconds. This operation should be carried out as smoothly as possible, keeping the axis of rotation fixed. Allow the graduated cylinder to stand for 30 minutes in a water bath at  $30 \pm 2^{\circ}\text{C}$ , taking care that the bath is free from vibrations.

Should excessive flocculation occur during the test, the material is unsatisfactory.

At the end of the 30 minutes settling period, insert the glass tube into the cylinder and, during 10-15 seconds with a minimum of disturbance, withdraw nine tenths of the suspension, i.e., 225 mL by means of the suction tube. This is achieved by maintaining the tip of the glass tube just below the sinking surface of the suspension. Discard the withdrawn suspension.

Transfer the contents of the cylinder containing the retained one-tenth of the suspension quantitatively into a tared large evaporating dish ( $\underline{w}$ 'g). Evaporate the water by heating on a boiling water-bath. Remove the dish as soon as the last traces of water have evaporated. Dry in an oven at about  $100^{\circ}\text{C}$  for 15 minutes. Cool and reweigh ( $\underline{w}$  g).

Mass of residue (m) =  $\underline{w} - \underline{w}'$ . (in g)

Where  $\underline{w}$  = mass of the evaporating dish containing the residue (in g)  
 $\underline{w}'$  = mass of the evaporating dish (in g).

Homogenize carefully the residue. Transfer a quantity of sample containing about 0.8 g of iodofenphos (weighed to the nearest 0.1 mg) in a 100 mL volumetric flask. Disperse in glacial acetic acid. Continue as in section 2.1.3 and determine the iodofenphos content ( $\underline{p}$  g/kg).

The total mass of iodofenphos ( $m_1$ ) in the retained bottom one-tenth of the suspension is:

$$\underline{m}_1 = \frac{\underline{p} \times \underline{m}}{1\ 000}$$

Where  $\underline{m}$  = mass of residue (g) determined here above.

From the value obtained in section 2.1 for the content of the active ingredient, calculate the mass ( $m_2$ ) of iodofenphos present in the initial sample taken for the suspensibility test.

$$\text{Suspensibility (\%)} = \frac{(\underline{m}_2 - \underline{m}_1) \times 111.1}{\underline{m}_2}$$

### 2.3 Heat stability treatment

54°C ± 2°C for 14 days (CIPAC method MT 46.1, CIPAC Handbook F, p.149), unless other temperatures and times are requested (FAO Manual on the development and use of FAO specifications for plant protection products, n° 149, p.33).

After completion of the heat stability treatment, the samples should not be exposed to heat, bright sunshine, or atmospheric humidity.

If required the test should be conducted in commercial type pack.