

TECHNICAL IODOFENPHOS

Specification WHO/SIT/22.R2
Approved 25 September 1989

1. Specification

1.1 Material

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

1.2 Chemical and physical requirements

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

1.2.1 *Iodofenphos content (g/kg basis)*

The iodofenphos content shall be declared (not less than 930 g/kg) and, when determined by the method described in section 2.1, the content obtained shall not differ from that declared by more than ± 20 g.

1.2.2 *Acidity*

The acidity of the material, determined by the method described in WHO/M/3, shall not be higher than 3 g/kg, calculated as H₂SO₄.

1.2.3 *Material insoluble in acetone*

The material insoluble in acetone as determined by the method described in WHO/M/21.R1, shall not be higher than 5 g/kg.

1.2.4 *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 to specification WHO/SIT/22.R2
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 *Special 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 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 (WHO/M/8.R1). The latter may not always be practicable owing to its unreliability at very low water contents. In the event of a dispute, the Karl Fischer method shall be the referee.

IODOFENPHOS WATER-DISPERSIBLE POWDER

Specification WHO/SIF/33.R2
Approved 25 September 1989

1. Specification

1.1 Description and ingredients

The material shall consist of a homogeneous mixture of technical iodofenphos together with filler(s) and any other necessary formulants and shall be in the form of a fine, free-flowing powder that wets out readily on stirring into water. The technical iodofenphos used in the manufacture of the water-dispersible powder shall comply with the requirements of specification WHO/SIT/22.R2.

1.2 Chemical and physical requirements

The material sampled from any part of the consignment (see method WHO/M/1), 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 nominal content by more than the following amounts:

<i>Nominal content</i>	<i>Tolerance permitted</i>
Up to 500 g/kg	$\pm 4\%$ of the nominal content
Above 500 g/kg	± 20 g/kg

The average content of all samples taken shall not be lower than the nominal 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 μ m sieve when tested by the method described in WHO/M/4.R1.

1.2.3 *Suspensibility*

In standard hard water after heat stability treatment. 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 standard hard water from the powder subjected to the heat stability treatment described in section 2.3.

1.2.4 *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 water-dispersible 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 water-dispersible powder to specification WHO/SIF/33.R2
Iodofenphos ... g/kg
Batch or reference number, and date of test
Net weight of contents
Date of formulation

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 *Special 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 filler¹ 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*

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

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.

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

2.2 Suspensibility after heat stability treatment

2.2.1 *Outline of method*

A suspension of known concentrate of iodofenphos in 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 *Special 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 *Special reagents*

Standard hard water. Dissolve 0.304 g of anhydrous calcium chloride and 0.139 g of magnesium chloride hexahydrate in distilled water and make up to 1 litre. This provides water with a hardness of 342 mg/l, calculated as calcium carbonate. Check the hardness by method WHO/M/26 and correct if appropriate.

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 water² at $30^{\circ}\text{C} \pm 1^{\circ}\text{C}$ 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 $30 \pm 1^{\circ}\text{C}$ for rinsing, and again avoiding mechanical disintegration of any lumps. Immediately add sufficient water at $30 \pm 1^{\circ}\text{C}$ to bring the 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. During agitation the cylinder must be thermally insulated from the hands to maintain the prescribed temperature of the suspension. Allow the graduated cylinder to stand for 30 minutes in a water bath at $30 \pm 1^{\circ}\text{C}$, taking care that the bath is free from vibrations.

² Whenever water is mentioned in this section, use standard hard water.

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 100°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 (p g/kg).

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

$$m_1 = \frac{p \times m}{1\ 000}$$

Where 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.

$$\text{Suspensibility (\%)} = \frac{(m_2 - m_1) \times 111.1}{m_2}$$

2.3 Heat stability treatment

Fill a 100 ml wide-mouthed glass bottle to within 1 cm of the top with the sample. Seal the bottle with a phenolic plastic cap having a soft liner. Turn the cap firmly to ensure a tight seal and place the bottle in a forced-draught oven maintained at $54 \pm 2^{\circ}\text{C}$ for 3 days. At the end of the heating period, remove the bottle from the oven and allow it to come to room temperature before removing the cap.

After heat stability treatment, the sample should not be exposed to heat, bright sunshine, or high atmospheric humidity.