

# TECHNICAL TRICHLORFON

Specification WHO/SIT/13.R4  
Approved 25 September 1989

## 1. Specification

### 1.1 Material

The material shall consist of trichlorfon together with related manufacturing compounds and shall be in the form of a white or near-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 *Setting-point*

The setting-point of the material, determined by the method described in section 2.1, shall not be lower than 77°C.

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

The trichlorfon content shall be declared (not less than 980 g/kg) and, when determined by the method described in section 2.2, the content obtained shall not differ from that declared by more than  $\pm 10$  g.

#### 1.2.3 *Acidity*

The acidity of the material, determined by the method described in section 2.3, shall not be higher than 3 g/kg calculated at  $\text{H}_2\text{SO}_4$ .

#### 1.2.4 *Material insoluble in acetone*

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

#### 1.2.5 *Water content*

The water content, determined by the method described in WHO/M/7.R.1, shall not be higher than 3 g/kg.

### 1.3 Packing and marking of packages

The technical trichlorfon 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 trichlorfon to specification WHO/SIT/13.R4  
Batch or reference number, and date of test  
Net weight of contents  
Date of manufacture

and the following minimum cautionary notice:

Trichlorfon is an organophosphorus compound that inhibits cholinesterase. It is poisonous if swallowed or absorbed through the skin. Avoid skin contact; wear protective gloves and clean protective clothing while using the material. Wash thoroughly with soap and water after using.

Keep the material out of 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 Setting-point

#### 2.1.1 *Outline of the method*

The sample is melted and then allowed to cool. The setting-point is the temperature at which solidification occurs.

#### 2.1.2 *Procedure*

Place in a 20 cm boiling-tube of 2.5 cm internal diameter with a wall thickness of  $2 \pm 0.1$  mm a sufficient amount of the sample to give, when melted, a depth of liquid of approximately 7.5 cm. Melt carefully by immersing the tube to a depth of 10 cm in an oil-bath, not allowing the temperature of the melt to exceed 80°C. Add 6 g of calcium sulfate previously dried at 180°C.

Fit the boiling-tube with a cork collar and insert it to within 1.3 cm of the bottom of a 15 cm boiling-tube of approximately 4 cm diameter; then immerse the two tubes to a depth of 10 cm in a water-bath maintained at 55°C. Place in the inner boiling-tube a stirrer consisting of a glass rod bent at one end in the form of a ring. A thermometer graduated in one-tenths of a degree is then clamped in a central position with its bulb 2.5 cm from

the bottom of the tube. The temperature of the melt should be approximately 5°C higher than the anticipated setting-point at this stage. Stir at a rate of about two strokes per second, by moving the stirrer up and down, until the material begins to thicken, at which point stir vigorously to work into the melt any material that has solidified on the walls of the tube. Stop stirring when the temperature stops falling and remains constant for some time<sup>1</sup>. Record this temperature as the setting-point after making any corrections necessary for the thermometer calibration and the emergent stem.

*Emergent-stem correction.* The correction for the emergent stem in mercury-filled thermometers, to be added to the temperature reading, is calculated from the following formula:

$$T_c = N \times 0.00015 \times (T - t)$$

Where	$T_c$ =	correction to be applied to the observed temperature of the setting-point.
	$N$ =	number of degrees on the scale of the thermometer between the top of the inner boiling-tube and the level of the mercury.
	$T$ =	temperature reading on the thermometer in the melt.
	$t$ =	temperature of the stem at the midpoint of the exposed mercury thread.

## 2.2 Trichlorfon content

### 2.2.1 *Outline of method*

The sample is hydrolysed quantitatively in a mixture of methanol and ethanolamine to form chlorine ions. The trichlorfon content is then measured by the difference between the total chlorine thus produced and the amount of inorganic (ionic) chlorine present in the sample before hydrolysis.

### 2.2.2 *Determination of hydrolysable plus inorganic chlorine*

Weigh (to the nearest 0.1 mg) about 1 g of the sample into a 250 ml conical flask and dissolve in 90 ml of anhydrous methanol. Add 10 ml of 2-aminoethanol<sup>2</sup> and keep for exactly 1 hour at 20°C ± 0.5°C. Cool in ice water and add 50 ml of chlorine-free 200 g/l nitric acid. Keep at 20°C and titrate electrometrically with 0.1 mol/l silver nitrate. Make a blank determination (without sample) following the exact procedure given above in order to obtain a chloride correction value for all reagents used.

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<sup>1</sup> In some instances, there may be a slight rise in the temperature of the material; if this occurs, record the highest steady temperature as the setting-point.

<sup>2</sup> 2-Aminoethanol of over 990 g/kg purity.

### 2.2.3 *Determination of inorganic chlorine*

Weigh (to the nearest 0.1 mg) about 1 g of the sample into a 250 ml conical flask and dissolve in 100 ml of distilled water. Keep for 5 minutes at room temperature, acidify with 5 ml of chlorine-free 200 g/l nitric acid, and titrate electrometrically with 0.1 mol/l silver nitrate.

### 2.2.4 *Calculation*

$$\text{Trichlorfon content (g/kg)} = \frac{(v_1 - v_2)}{(m_1 - m_2)} \times 25.75$$

$v_1$  = volume (ml) of 0.1 mol/l silver nitrate equivalent to the total chlorine (hydrolysable plus inorganic)

$v_2$  = volume (ml) of 0.1 mol/l silver nitrate equivalent to the inorganic chlorine

$m_1$  = mass (g) of sample used for the total chlorine determination (hydrolysable plus inorganic)

$m_2$  = mass (g) of sample used for the inorganic chlorine determination

## 2.3 **Acidity**

### 2.3.1 *Procedure*

Weigh (to the nearest 10 mg) 10 g of the sample and dissolve it in 100 ml of distilled water, with gentle warming if necessary. Titrate immediately at 10-15°C with 0.02 mol/l sodium hydroxide, using methyl red as indicator. Carry out a blank determination on 100 ml of distilled water with 0.02 mol/l sodium hydroxide.

### 2.3.2 *Calculation*

Acidity, g/kg, calculated as:

$$\text{H}_2\text{SO}_4 = 0.098 \times (v_1 - v_2)$$

Where  $v_1$  = volume (ml) of 0.02 mol/l sodium hydroxide used for the sample

$v_2$  = volume (ml) of 0.02 mol/l sodium hydroxide used for the blank

The blank may take the form of a small titre with 0.02 mol/l hydrochloric acid, in which case, Acidity, g/kg, calculated as:

$$\text{H}_2\text{SO}_4 = 0.098 \times (v_1 + v_3)$$

Where  $v_1$  = volume (ml) of 0.02 mol/l sodium hydroxide used for the sample

$v_3$  = volume (ml) of 0.02 mol/l hydrochloric acid used for the blank

Alternatively, the end-point may be determined electrometrically.

# TRICHLORFON

## WATER-SOLUBLE POWDER

Specification WHO/SIF/45.R1  
Approved 25 September 1989

### 1. Specification

#### 1.1 Description and ingredients

The material shall consist of a homogeneous mixture of technical trichlorfon with a water-soluble filler and shall be in the form of a free-flowing powder, white to light brown in colour, which is soluble in water. The technical trichlorfon used in the manufacture of the powder shall comply with the requirements of specification WHO/SIT/13.R4.

#### 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 *Trichlorfon content (g/kg basis)*

The content of trichlorfon, 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	-5 to +10% of the nominal content
Above 500 g/kg	-25 to +50 g/kg

##### 1.2.2 *Wet sieving after heat stability treatment*

Not less than 97% of the powder after the heat stability treatment as described in section 2.5 shall pass through a 45 mm sieve when tested by the method WHO/M/4.R1.<sup>1</sup>

##### 1.2.3 *Water content*

The water content, determined by the method described in section 2.2, shall not be higher than 15 g/kg.

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<sup>1</sup> Owing to active ingredient solubility the method WHO/M/4.R1 should be modified as follows: a 500 ml beaker shall be used and the powder shall be dissolved in 250 ml of water for 3 minutes.

1.2.4 *Acidity*<sup>2</sup>

The acidity of the powder, determined by the method described in section 2.3, shall not be higher than 5 g/kg calculated as H<sub>2</sub>SO<sub>4</sub>.

1.2.5 *Water-insoluble material*

The content of solid material insoluble in water shall be declared in g/kg and, when determined by the method described in section 2.4, the content obtained shall not differ from that declared by more than ±1.5%.

1.2.6 *Heat stability*

The sample after heat stability treatment as described in section 2.5 shall comply with the requirement of section 1.2.1 and 1.2.4 (except that the maximum permitted acidity shall be 8 g/kg calculated as H<sub>2</sub>SO<sub>4</sub>).

**1.3 Packing and marking of packages**

The trichlorfon water-soluble powder shall be packed in suitable, clean drums, as specified in the order. The drums shall contain an inner liner or bag of polyethylene or equivalent, with a nominal thickness of 0.1 mm. The inner liner or bag shall be hermetically sealed after filling.

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

Manufacturer's name  
Trichlorfon water-soluble powder to specification WHO/SIF/45.R1  
Trichlorfon .... g/kg  
Batch or reference number and date of test  
Net weight of contents  
Date of formulation

and the following minimum cautionary notice:

Trichlorfon is an organophosphorus compound that inhibits cholinesterase. It is poisonous if swallowed or absorbed through the skin. Avoid skin contact; wear protective gloves and clean protective clothing while using the material. Wash thoroughly with soap and water after using.

Keep the material out of reach of children and well away from foodstuffs, animal feed and their containers. If poisoning occurs, call a physician.

Atropine and pralidoxime are specific antidotes, and artificial respiration may be needed.

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<sup>2</sup> This maximum limit is given for products which do not contain acidic stabilizers. If these are present, the maximum acidity shall be declared.

## 2. Methods of determining chemical and physical properties

### 2.1 Trichlorfon content

#### 2.1.1 *Outline of method*

The sample is hydrolysed quantitatively in a mixture of methanol and ethanolamine to form chlorine ions. The trichlorfon content is then measured by the difference between the total chlorine thus produced and the amount of inorganic (ionic) chlorine present in the sample before hydrolysis.

#### 2.1.2 *Determination of hydrolysable plus inorganic chlorine*

Weigh (to the nearest 0.1 mg) an amount of the sample containing about 1 g of trichlorfon into a 250 ml conical flask and dissolve it in 90 ml of anhydrous methanol. Add 10 ml of 2-aminoethanol<sup>3</sup> and keep for exactly 1 hour at  $20 \pm 0.5^\circ\text{C}$ . Cool in ice water and add 50 ml of chlorine-free 200 ml/l nitric acid. Keep at  $20^\circ\text{C}$  and titrate electrometrically with 0.1 mol/l silver nitrate.

Make a blank determination (without sample) following the exact procedure given above in order to obtain a chloride correction value for all reagents used.

#### 2.1.3 *Determination of inorganic chlorine*

Weigh (to the nearest 0.1 mg) an amount of sample containing about 1 g of trichlorfon into a 250 ml conical flask and dissolve it in 100 ml of distilled water. Keep for 5 minutes at room temperature, acidify with 5 ml of chlorine-free 200 g/l nitric acid, and titrate electrometrically with 0.1 mol/l silver nitrate.

#### 2.1.4 *Calculation*

$$\text{Trichlorfon content (g/kg)} = \frac{(v_1 - v_2)}{(m_1 - m_2)} \times 25.75$$

$v_1$  = volume (ml) of 0.1 mol/l silver nitrate equivalent to the total chlorine (hydrolysable plus inorganic).

$v_2$  = volume (ml) of 0.1 mol/l silver nitrate equivalent to the inorganic chlorine

$m_1$  = mass (g) of sample used for the total chlorine determination. (hydrolysable plus inorganic chlorine).

$m_2$  = mass (g) of sample used for the inorganic chlorine determination.

<sup>3</sup> 2-Aminoethanol of over 990 g/kg purity.

## 2.2 Water content

Determine the water content by the Karl Fischer electrometric titration method (see WHO/M/7.R1) or by the Dean and Stark distillation method (see WHO/M/8.R1). In the event of a dispute, the Karl Fischer method shall be the referee method.

## 2.3 Acidity

### 2.3.1 Procedure

Dissolve 10 g (weighed to the nearest 10 mg) of the sample in 250 ml of distilled water, filter, and titrate immediately at 10-15°C with 0.02 mol/l sodium hydroxide, using methyl red as indicator. Carry out a blank determination on 250 ml of distilled water with 0.02 mol/l sodium hydroxide.

### 2.3.2 Calculation

Acidity (g/kg) calculated as  $\text{H}_2\text{SO}_4 = 0.098 \times (v_1 - v_2)$

Where  $v_1$  = volume (ml) of 0.02 mol/l sodium hydroxide used for the sample  
 $v_2$  = volume (ml) of 0.02 mol/l sodium hydroxide used for the blank

The blank may take the form of a small titre with 0.02 mol/l hydrochloric acid, in which case, acidity (g/kg) calculated as  $\text{H}_2\text{SO}_4 = 0.098 \times (v_1 + v_3)$

Where  $v_1$  = volume (ml) of 0.02 mol/l sodium hydroxide used for the sample  
 $v_3$  = volume (ml) of 0.02 mol/l hydrochloric acid used for the blank

## 2.4 Water-insoluble material

### 2.4.1 Procedure

Dry a sintered-glass crucible (i.e. Gooch crucible), porosity No. 3, at 105°C and weigh ( $x$  g). Weigh (to the nearest 1 mg) about 5 g of the sample into a 500 ml beaker, add 250 ml of distilled water, and heat to 30°C, while stirring. Filter the solution through the dry, tared crucible and wash twice with 100 ml portions of distilled water at approximately 30°C. Dry to constant weight in an oven at 110°C and weigh ( $y$  g).

### 2.4.2 Calculation

$$\text{Water-insoluble material (g/kg)} = \frac{a \times 1000}{m}$$

Where  $a = (y - x) = \text{mass (g) of the residue}$   
 $m = \text{mass (g) of the sample}$

## 2.5 Heat stability treatment

Fill a 50 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  $54 \pm 1^\circ\text{C}$  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.