

TECHNICAL DICHLORVOS

Specification WHO/SIT/16.R3
Approved 25 September 1989

1. Specification

1.1 Material

The material shall consist of dichlorvos together with related manufacturing compounds and shall be a pale amber-coloured liquid 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 *Dichlorvos content (g/kg basis)*

The dichlorvos content shall be declared (not less than 970 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/kg.

1.2.2 *Acidity*

The acidity of the material, determined by the method described in WHO/M/3 shall not be higher than 2 g/kg, calculated as H₂SO₄.

1.2.4 *Water content¹*

The water content determined by the method described in WHO/M/7.R1, shall not be higher than 1 g/kg.

1.3 Packing and marking of packages

The technical dichlorvos shall be packed in suitable, clean containers, as specified in the order.

¹ Dichlorvos is hygroscopic and should be sampled in such a way as to ensure minimum entry of water vapour.

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

Manufacturer's name
Technical dichlorvos to specification WHO/SIT/16.R3
Batch or reference number, and date of test
Net weight of contents
Date of manufacture

and the following minimum cautionary notice:

POISON

(Skull-and-cross-bones emblem)

Dichlorvos is an organophosphorus compound that inhibits cholinesterase. It is poisonous if swallowed, inhaled, or absorbed through the skin. Wear protective gloves, clean protective clothing, goggles, and a respirator of the organic-vapour type when handling this material. Avoid prolonged exposure to fumes. Wash hands and exposed skin after handling and before eating, and bathe immediately after work.

Keep the material out of the reach of children and well away from foodstuffs, animal feed and their containers. Ensure that containers are tightly sealed, and stored and disposed of in such a way as to prevent accidental contact. In case of contact, immediately remove contaminated clothing and wash the skin thoroughly with soap and water; for eyes, flush with water for 15 minutes.

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 Dichlorvos content

2.1.1 *Outline of method*

The sample is dissolved in acetone with diethyl pimelate added as internal standard. The dichlorvos content is determined by gas-liquid chromatography using a 3% OV-225 on Gas-Chrom Q or Chromosorb W-HP column.

2.1.2 *Special apparatus*

1. *Gas-liquid chromatograph.* The instrument should be one that is designed for use with glass columns and that is equipped with an on-column injection system, a high-sensitivity flame-ionization detector and a suitable recorder or electronic integrator.
2. *Chromatographic column.* The column should be a borosilicate glass tube 150 cm long, 4 mm in internal diameter, and 6 mm in external diameter, bent to fit the chromatograph.

3. *Column-packing material.* Gas-Chrom Q (100-120 mesh) or Chromosorb W-HP (100-120 mesh) treated with 3% OV-225.

2.1.3 *Special reagents*

Dichlorvos standard. Analytical grade, of known purity.

Internal standard. Diethyl pimelate of known purity (about 990 g/kg) and free from components that elute at the same time as dichlorvos under the chromatographic conditions given in section 2.1.6.

2.1.4 *Preparation of standard solutions*

Internal standard solution. Prepare an acetone solution containing diethyl pimelate at a concentration of 4 mg/ml. Sufficient solution should be prepared for all calibration and sample solutions.

Dichlorvos calibration solutions. Into two separate 50 ml volumetric flasks weigh (to the nearest 0.1 mg) approximately 0.200 and 0.250 g of analytical standard grade dichlorvos. To each flask add by pipette 25 ml of the internal standard solution. Dilute to volume with acetone and mix thoroughly.

2.1.5 *Preparation and conditioning of column*

See method WHO/M/20.

2.1.6 *Operating conditions for gas-liquid chromatography*

The conditions given below are typical values and may have to be adjusted to obtain optimum results from a given apparatus.

Temperatures

Oven 140⁰C

Injection port 160⁰C

Flame-ionization detector 250⁰C

Gas flow rates

Hydrogen and air As recommended for the detector by the manufacturer.

Carrier gas (nitrogen) 40 ml/min.

Retention times

Dichlorvos peak 5.0 min.

Internal standard peak 9.5 min.

2.1.7 *Linearity check*

Inject 2-6 μl of the calibration solution containing about 5 mg of dichlorvos/ml (section 2.1.4) and adjust the instrument controls so that the maximum of the larger peak (either from dichlorvos or diethyl pimelate) gives a recorder deflection of 80-90%. Note the volume injected ($y \mu\text{l}$).

Inject duplicate volumes of $y \mu\text{l}$ of each of the calibration solutions. Measure the dichlorvos and diethyl pimelate peak areas. For each injection calculate the calibration factor F by means of the following equation:

$$F = \frac{m_1 \times P \times \text{area of diethyl pimelate peak}}{1\,000 \times \text{area of dichlorvos peak}}$$

where m_1 = mass (g) of dichlorvos in the calibration solution

P = purity (g/kg) of the dichlorvos standard

Repeat the injections until the values of F obtained for each calibration solution do not differ from each other by more than $\pm 0.5\%$.

2.1.8 *Sample preparation and analysis*

Weigh in duplicate (to the nearest 0.1 mg) 0.220-0.240 g of technical material into two 50 ml volumetric flasks. To each flask add, using the same pipette as used to prepare the dichlorvos calibration solution, 25 ml of the internal standard solution. Dilute to volume with acetone and mix thoroughly.

Inject duplicate volumes of $y \mu\text{l}$ (section of 2.1.7), of each sample solution. Make each group of four sample injections between two injections of the same calibration solution, e.g., calibration solution A injection, followed by sample injections 1, 2, 3 and 4 and again calibration solution A injection. Determine the calibration factors F_a and F_b for each of the calibration solution injections. Calculate the mean calibration factor F_c from the equation:

$$F_c = \frac{F_a + F_b}{2}$$

If the calibration factors F_a and F_b differ by more than $\pm 0.5\%$ of the mean F_c , repeat both calibration solution and sample injections. If the mean calibration F_c differs from F obtained in the linearity check above by more than $\pm 2\%$, stabilize the operating conditions and recalibrate before proceeding with the analysis.

2.1.9 Calculation

$$\text{Dichlorvos content (g / kg)} = \frac{F_c \times A_s}{m_2 \times A_b} \times 1000$$

- F_c = mean calibration factor for dichlorvos (section 2.1.8)
 m_2 = mass (g) of sample taken
 A_b = area of diethyl pimelate peak in the sample solution
 A_s = area of dichlorvos peak in the sample solution

DICHLORVOS EMULSIFIABLE CONCENTRATE

Specification WHO/SIF/39.R1
Approved 25 September 1989

1. Specification

1.1 Description and ingredients

The material shall consist of technical dichlorvos dissolved in suitable solvents, with other necessary formulants added. It shall be in the form of a stable liquid, free from suspended matter and sediment. The technical dichlorvos used in the manufacture of the concentrate shall comply with the requirements of specification WHO/SIT/16.R3.

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 *Dichlorvos content (g/kg basis)*

The content of dichlorvos, 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% of the nominal content
Above 500 g/kg	± 25 g/kg

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

1.2.2 *Water content*

The water content determined by the method described in WHO/M/7.R1 shall not be higher than 1 g/kg.

1.2.3 *Acidity or alkalinity*

The acidity or alkalinity of the concentrate, determined by the method described in WHO/M/3, shall not be higher than 5 g/kg, calculated as H₂SO₄, or 0.1 g/kg calculated as NaOH.

1.2.4 *Cold test*

No separation of solid or oily material shall occur when the concentrate is tested as described in method WHO/M/23.

1.2.5 *Flash point*

The flash point of the product, determined by the method WHO/M/10.R1, shall comply with all national and/or international transport regulations.

1.2.6 *Stability of emulsion*

In standard soft water. Any separation, including creaming/oiling at the top and oiling/sedimentation at the bottom, of 100 ml of emulsion prepared in standard soft water with 5 ml of concentrate shall not exceed 2 ml when tested as described in WHO/M/13.R3.

In standard hard water. Any separation, including creaming/oiling at the top and oiling/sedimentation at the bottom, of 100 ml of emulsion prepared in standard hard water with 5 ml of concentrate shall not exceed 2 ml when tested as described in WHO/M/13.R3.

1.2.7 *Heat stability*

The concentrate, after treatment as described in section 2.2 shall comply with the requirements of sections 1.2.1, 1.2.3 and 1.2.6 of this specification.

1.3 Packing and marking of packages

The dichlorvos emulsifiable concentrate 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
Dichlorvos emulsifiable concentrate to specification WHO/SIF/39.R1
Dichlorvos g/kg
Batch or reference number, and date of test
Net weight of contents
Instruction for dilution
Date of manufacture

and the following minimum cautionary notice:

POISON

(Skull-and-cross-bones emblem)

Dichlorvos is an organophosphorus compound that inhibits cholinesterase. It is poisonous if swallowed, inhaled, or absorbed through the skin. Wear protective gloves, clean protective clothing, goggles, and a respirator of the organic-vapour type when handling this material. Avoid prolonged exposure to fumes. Wash hands and exposed skin after handling and before eating, and bathe immediately after work.

Keep the material out of the reach of children and well away from foodstuffs, animal feed and their containers. Ensure that containers are tightly sealed, and stored and disposed of in such a way as to prevent accidental contact. In case of contact, immediately remove contaminated clothing and wash the skin thoroughly with soap and water; for eyes, flush with water for 15 minutes.

If poisoning occurs, call a physician. Atropine and pralidoxime are specific antidotes and artificial respiration may be needed. The label should include a warning that the product should be stored in a cool, dry place.

2. Methods of determining chemical and physical properties

2.1 Dichlorvos content

2.1.1 *Outline of method*

The sample is dissolved in acetone with diethyl pimelate added as internal standard. The dichlorvos content is determined by gas-liquid chromatography using a 3% OV-225 on Gas-Chrom Q or Chromosorb W-HP column.

2.1.2 *Special apparatus*

1. *Gas-liquid chromatograph.* The instrument should be one that is designed for use with glass columns and that is equipped with an on-column injection system, a high-sensitivity flame-ionization detector and a suitable recorder or electronic integrator.
2. *Chromatographic column.* The column should be a borosilicate glass tube 150 cm long, 4 mm in internal diameter, and 6 mm in external diameter, bent to fit the chromatograph.
3. *Column-packing material.* Gas-Chrom Q (100-120 mesh) or Chromosorb W-HP (100-120 mesh) treated with 3% OV-225.

2.1.3 *Special reagents*

Dichlorvos standard. Analytical grade, of known purity.

Internal standard. Diethyl pimelate of known purity (about 990 g/kg) and free from components that elute at the same time as dichlorvos under the chromatographic conditions given in section 2.1.6.

2.1.4 *Preparation of standard solutions*

Internal standard solutions. Prepare an acetone solution containing diethyl pimelate at a concentration of 4 mg/ml. Sufficient solution should be prepared for all calibration and sample solutions.

Dichlorvos calibration solution. Into two separate 100 ml volumetric flasks weigh (to the nearest 0.1 mg) approximately 0.400 g and 0.500 g of analytical standard grade dichlorvos. To each flask add by pipette 50 ml of the internal standard solution. Dilute to volume with acetone and mix thoroughly.

2.1.5 *Preparation and conditioning of column*

See method WHO/M/20.

2.1.6 *Operating conditions for gas-liquid chromatography*

The conditions given below are typical values and may be adjusted to obtain optimum results from a given apparatus.

Temperatures

Oven	140 ⁰ C
Injection port	160 ⁰ C
Flame-ionization detector	250 ⁰ C

Gas flow rates

Hydrogen and air	As recommended for the detector by the manufacturer.
Carrier gas (nitrogen)	40 ml/min.

Retention times

Dichlorvos peak	5.0 min.
Internal standard peak	9.5 min.

2.1.7 *Linearity check*

Inject 2-6 µl of the calibration solution containing about 5 mg of dichlorvos/ml (section 2.1.4) and adjust the instrument controls so that the maximum of the larger peak (either from dichlorvos or diethyl pimelate) gives a recorder deflection of 80-90%. Note the volume injected (y µl).

Inject duplicate volumes of $y \mu\text{l}$ of each of the calibration solutions. Measure the dichlorvos and diethyl pimelate peak areas. For each injection calculate the calibration factor F by means of the following equation:

$$F = \frac{m_1 \times P \times \text{area of diethyl pimelate peak}}{1\,000 \times \text{area of dichlorvos peak}}$$

m_1 = mass (g) of dichlorvos in the calibration solution

P = purity (g/kg) of the dichlorvos standard

Repeat the injections until the values of F obtained for each calibration solution do not differ from each other by more than $\pm 0.5\%$.

2.1.8 Sample preparation and analysis

Weigh in duplicate (to the nearest 0.1 mg) into two 100 ml volumetric flasks an amount of sample sufficient to contain about 0.450g dichlorvos. To each 100 ml flask, add, using the same pipette as used to prepare the dichlorvos calibration solution, 50 ml of the internal standard solution. Dilute to volume with acetone and mix thoroughly. Inject duplicate volumes of $y \mu\text{l}$ (section 2.1.7) of each sample solution. Make each group of four sample injections between two injections of the same calibration solution, e.g., calibration solution A injection, followed by sample injections 1, 2, 3 and 4 and again calibration solution A injection. Determine the calibration factors F_a and F_b for each of the calibration solution injections. Calculate the mean calibration factor F_c from the equation.

If the calibration factors F_a and F_b differ by more than $\pm 0.5\%$ of the mean F_c , repeat both calibration solution and sample injections.

$$F_c = \frac{F_a + F_b}{2}$$

If the mean calibration F_c differs from F obtained in the linearity check above by more than $\pm 2\%$, stabilize the operating conditions and recalibrate before proceeding with the analysis.

2.1.9 Calculation

$$\text{Dichlorvos content (g / kg)} = \frac{F_c \times A_s}{m_2 \times A_b} \times 1000$$

F_c = mean calibration factor for dichlorvos (section 2.1.8)

m_2 = mass (g) of sample taken

A_b = area of diethyl pimelate peak in the sample solution

A_s = area of dichlorvos peak in the sample solution