LINDANE[1]

Specification WHO/SIT/3.R5
Approved 25 September1989

1. Specification

1.1 Material

The material shall consist of the gamma-isomer of HCH together with related manufacturing compounds and shall be in the form of white or near-white granules, flakes, or powder, free from extraneous impurities or added modifying agents and with no more than a faint odour.

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 *Gamma-HCH isomer content (g/kg basis)*

The gamma-HCH isomer content shall be declared (not less than 990g/kg) and, when determined by the method described in section 2.1, the content obtained shall not differ from that declared by more than ± 2 g.

1.2.2 *Melting point*

The melting point of the material, determined by the method described in WHO/M/5, shall not be lower than 112°C and shall not be depressed when mixed with an equal quantity of pure gamma-HCH.

1.2.3 *Alpha-HCH isomer content*

The alpha-HCH isomer content determined by the method described in section 2.2, shall not be higher than 0.5% of the gamma-HCH isomer content found under 1.2.1.

1.2.4 *Acidity*

The acidity of the material determined by the method described in WHO/M/3 shall not be higher than 1.5g/kg calculated as H_2SO_4.

[1] Lindane is a grade of HCH (BHC) containig not less than 990g/kg gamma-HCH isomer.

1.2.5 *Material insoluble in acetone*

The material insoluble in acetone determined by the method described in WHO/M/21.R1 shall not be higher than 1g/kg.

1.2.6 *Water content*

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

1.3 Packing and marking of packages

The lindane 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
Lindane to specification WHO/SIT/3.R5
Batch or reference number, and date of test
Net weight of contents
Date of manufacture

and the following minimum cautionary notice:
Keep well away from foodstuffs and animal feed and their containers.

2. Methods of determining chemical and physical properties

2.1 Gamma-HCH isomer content

2.1.1 *Outline of method*

The sample is melted in an atmosphere of nitrogen, allowed to cool at a controlled rate, the freezing point determined and hence the gamma-HCH isomer content calculated.

2.1.2 *Special apparatus*

1. 20 cm boiling-tube of 2.5 cm internal diameter (sample tube).
2. 15 cm boiling-tube of approximately 4 cm internal diameter (air jacket).
3. Loop stirrer and gas inlet-figure 1. The stirrer is a glass capillary loop with a vertical glass stem and loop arranged to surround, closely, the thermometer bulb.

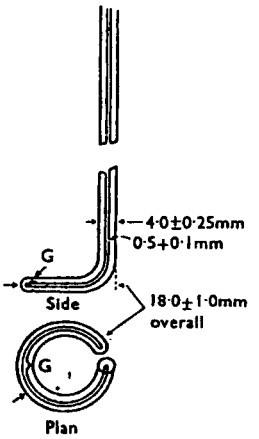

Figure 1, Side and plan view of loop stirrer

It is made of 0.5 mm bore, 5 mm outside diameter capillary tube with a pin-hole blown in the end of the capillary.

4. Standard short-range thermometer (99.5-130.5°C) such as that described in British Standard 593:1954. Thermometers should have an official certificate of examination and should be checked against a fixed reference point every 6 months.

5. Magnifying glass to fit the thermometer so that readings can be made to ±0.02°C. For accurate observations, such a device must be used to obviate parallax.

6. Drying-train for nitrogen, containing magnesium perchlorate or self-indicating silica gel and a pressure-regulating device containing mercury to a depth: 16 mm.

2.1.3 *Procedure*

Place in a 20 cm boiling-tube of 2.5 cm internal diameter (sample tube) a sufficient amount of the sample to give, when melted, a depth of liquid of approximately 7.5 cm (about 25 g). Melt carefully by immersing the tube to a depth of 10 cm in an oil-bath at approximately 120°C. By means of the loop stirrer connected to the nitrogen drying-train, pass nitrogen as a fine stream of bubbles through the melt for 1 hour. 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 (air jacket) then immerse the two tubes to a depth of 10 cm in an oil-bath at 106°C[1] , maintaining the flow of nitrogen through the stirrer.

[1] The electrically heated apparatus described by Toops, E.E.& Riddick, J.A. Analytical chemistry, 23:1106 (1951), is very suitable and may be used in place of the oil-bath.

A standard short-range thermometer graduated in one-tenths of a degree is clamped in a central position with its bulb 2.5 cm from the bottom of the tube. Stir at the rate of about two strokes per second, by moving the stirrer up and down. When the material begins to thicken, work into the melt any portions of the material that have solidified on the walls of the tube[2] .

Read the temperature at 1 minute intervals, using a magnifying device, and take the first three consecutive readings during which the temperature remains constant as the setting-point[3] . Apply corrections as 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:

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

Tc = 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.

1.2.3 *Calculation*

Gamma-HCH isomer content (g/kg) = 10 antilog [2- (0.00643 x T)]

T = 112.86 minus the setting-point of sample.
An impurity of 1% depresses the setting-point by 0.7°C.

2.2 Alpha-HCH isomer content

The analytical method is obtainable on request from WHO, Division of Control of Tropical Diseases, CH 1211 Geneva 27, Switzerland.

---

[2] Since the gamma-HCH isomer undergoes slight supercooling, mobile crystals should always be present to initiate freezing.

[3] In some instances, there may be a slight rise in the temperature of the material; in such a case, record the highest steady temperature as the setting-point. In the event of superheating exceeding 0.25°C, results should be rejected and the determination repeated.

LINDANE[1]
WATER-DISPERSIBLE POWDER

Specification WHO/SIF/2.R7
Approved 25 September 1989

1. Specification

1.1 Description and ingredients

The material shall consist of a homogeneous mixture of technical lindane together with filler(s) and any other necessary formulants and shall be in the form of a fine, free-flowing, white to cream colored powder that wets out readily on stirring into water. The lindane used in the manufacture of the water-dispersible powder shall comply with the requirements of specification WHO/SIT/3.R5.

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 *Gamma-HCH isomer content (g/kg basis)*

The content of gamma-HCH isomer, determined by the method described in section 2.1, shall not differ from the nominal content by more than the following amounts:

Nominal content	*Tolerance permitted*
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 *Alpha-HCH isomer content*

The alpha-HCH isomer content, determined by the method described in section 2.2, shall not be higher than 0.5% of the gamma-HCH isomer content found under 1.2.1

[1] Lindane is a grade of HCH (BHC) containing not less than 990 g/kg gamma-HCH isomer.

1.2.3 *Acidity or alkalinity*

The acidity or alkalinity of the powder, determined by the method described in WHO/M/3, shall not be higher than 2 g/kg calculated as H_2SO_4 or 2g/kg calculated as NaOH.

1.2.4 *Sieving after heat stability treatment*

Not less than 98% of the powder heat stability treatment (section 2.4) shall pass through a 75 μm sieve when tested by the method described in WHO/M/4.R1.

1.2.5 *Suspensibility*

In standard hard water after heat stability treatment. When tested by the method described in section 2.3, a minimum of 50% of the gamma-HCH isomer (2.5g/1) shall be in suspension 30 minutes after agitating a suspension containing 5g/1 of lindane (gamma-HCH isomer), prepared in standard hard water from the powder subjected to the heat stability treatment described in section 2.4.

1.2.6 *Heat stability*

The powder after treatment as described in section 2.4, shall comply with the requirements of section 1.2.1 and 1.2.3 of this specification.

1.3 Packing and marking of packages

The lindane water-dispersible powder shall be packed in suitable, clean drums, as specified in the order. The drums shall have a minimum capacity of 2 litres for every kilogram of powder and 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
Lindane water-dispersible powder to specification WHO/SIF/2.R7
Gamma-HCH isomer content...g/kg
Batch or reference number, and date of test
Net weight of contents
Date of manufacture

and the following minimum cautionary notice:

Keep well away from foodstuffs and animal feed and their containers.

2. Methods of determining chemical and physical properties

2.1 Gamma-HCH isomer content

2.1.1. *Outline of method*

HCH, gamma-isomer, is extracted from the sample with ethyl acetate and di-n- propyl phthalate is added as an internal standard. The gamma-isomer content is determined by gas-liquid chromatography using a flame-ionization detector.

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 and a high-sensitivity flame-ionization detector.

2. *Chromatographic column.* The column should be a borosilicate glass tube 183 cm long, 2 mm in internal diameter, and 6 mm in external diameter, acid washed and dry, bent to fit the chromatograph.

3. *Column packing material.* Chromosorb W-HP (100-120 mesh) treated with 7.5% OV-210.

2.1.3 *Special reagents*

Gamma-HCH isomer standard. Analytical grade, of known purity.
Internal standard. Di-n-propyl phthalate, purity better than 995 g/kg.

2.1.4 *Preparation of standard solutions.*

Internal standard solution. Weigh about 2.5 g of di-n-propyl phthalate into a 100 ml volumetric flask, dilute to volume with ethyl acetate and mix.
Gamma-HCH isomer calibration solution. Weigh (to the nearest 0.1 mg) about 250 mg of HCH, gamma-isomer standard into a 50 ml screw-capped bottle. Add by pipette 5.0 ml of internal standard solution and 20.0 ml of ethyl acetate and shake for 30 seconds.

2.1.5 *Preparation and conditioning of column*

See method WHO/M/20.

2.1.6 *Operating conditions for gas-liquid chromatography*

The temperatures, gas flow rates, and retention times given below are typical values and may have to be adjusted to obtain optimum results from a given apparatus.

Temperatures	
Oven	160°C
Injection port	220°C
Flame-ionization detector	250°C
Gas flow rates	
Hydrogen}	As recommended for the
Air}	detector by the manufacturer
Carrier gas (nitrogen)	10 ml/min.
Retention times	
HCH alpha-isomer peak[2]	11 min.
HCH gamma-isomer peak	14 min.
HCH beta-isomer peak	17 min.
HCH delta-plus epsilon-isomers peak	20 min.
Di-n-propyl phthalate peak	23 min.

2.1.7 *Sample preparation and analysis*

Weigh (to the nearest 0.1 mg) a quantity of sample containing about 250 mg of HCH, gamma-isomer into a 50 ml screw-capped bottle. Add by pipette 5.0 ml of internal standard solution and 20.0 ml of ethyl acetate and shake for 30 seconds. Filter or centrifuge to remove insoluble particles.

Insert 1 µl portions of calibration solution of gamma-HCH isomer until the response ratios (peak area or peak height) between gamma-HCH isomer and internal standard agree to within 2%. Make duplicate injections of calibration solution followed by duplicate injections of sample solution. Recalibrate after not more than 4 injections of sample solutions.

2.1.8 *Calculation*

For each injection, the response ratio (r) is given by the equation:

$$r = \frac{\textit{area (or height) of gamma-HCH isomer peak}}{\textit{area (or height) of internal standard peak}}$$

$$\textit{gamma-HCH isomer content (g/kg)} = \frac{r_2 \times m_1 \times p}{r_1 \times m_2}$$

[2] If this peak appears to be important, proceed to the determination of HCH alpha-isomer content (section 2.2)

Where r_1 = average response ratio for calibration solution
r_2 = average response ratio for sample solution
m_1= mass of gamma-HCH isomer standard in the calibration solution (mg)
m_2= mass of sample taken (mg)
P = purity of gamma-HCH isomer standard (g/kg)

2.2 Alpha-HCH isomer content

The analytical method is obtainable on request from WHO, Division of Control of Tropical Diseases, CH 1211 Geneva 27, Switzerland.

2.3 Suspensibility after heat stability treatment

2.3.1 *Outline of method*

A suspension of known concentration of lindane (gamma-HCH isomer) 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 gamma-HCH isomer in the bottom 1/10 th is determined, so allowing to evaluate the active ingredient mass still in suspension after 30 minutes.

2.3.2 *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 mark.
2. A glass tube, about 40 cm long and about 5 mm in internal diameter, drawn out at one end to an opening of 2-3 mm, the other end being connected to suitable source of suction.

2.3.3 *Special reagent*

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/1, calculated as calcium carbonate. Check the hardness by method WHO/M/26 and correct if appropriate.

2.3.4 *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 5 g/1 gamma-HCH isomer. Add a volume of water[3] at 30 ± 1°C equal to at least twice the mass of the sample taken. Allow to stand for 30 seconds and then stir by hand for 30 seconds with a glass rod, 4-6 mm diameter, at not

[3] Whenever water is mentioned in this section use standard hard water.

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 ± 1°C for rinsing, and again avoiding mechanical disintegration of any lumps. Immediately add sufficient water at 30 ± 1°C to bring the volume to the 250 ml mark. Stopper the cylinder and mix by inverting and righting it 30 times at a rate of one complete cycle every 2 seconds. This operation should be carried out as smoothly as possible, keeping the axis of rotation fixed. 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 ±1°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, with a minimum of disturbance, withdraw nine-tenths of the suspension (i.e. 225 ml) during 10-15 seconds by means of the suction tube. This is achieved by maintaining the tip of the glass tube just below the sinking top level of the suspension. Discard the suspension withdrawn. Determine the mass of gamma-HCH isomer in the retained bottom one-tenth of the suspension, including the sediment, by transferring it quantitatively with water into a tarred large evaporating dish ($\underline{w}'$ g). Evaporate the water by heating. Remove the dish as soon as the last traces of water have evaporated. Cool and reweigh ($\underline{w}$ g).

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

$\underline{w}$ = 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 250 mg of gamma-HCH isomer[4] weighed to the nearest 0.1 mg, to a 50 ml stoppered conical flask equipped with teflon-lined screw cap. Add by pipette 5.0 ml of internal standard solution and 20.0 ml of ethyl acetate, shake, filter or centrifuge and continue as described in section 2.1.7.

Calculate the gamma-HCH isomer content ($\underline{p}$ g/kg) according to section 2.1.8. The total mass of gamma-HCH isomer (m_1) in the retained bottom one-tenth of the suspension is:

$$m_1 = \frac{p \times m}{1000}$$

m = mass of residue (g) determined here above.

[4] If segregation between lindane and formulants occurs during the sedimentation, the sample weight has to be adapted accordingly.

2.3.5 *Calculation*

From the value obtained in section 2.1 for the content of gamma-HCH isomer (g/kg), calculate the mass of gamma-HCH isomer (m_2) in the initial sample taken for the suspensibility test.

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

m_1 = total mass of gamma-HCH isomer in the retained bottom one-tenth of the suspension (g).

m_2 = mass of gamma-HCH isomer in the initial sample (g).

2.4 Heat stability treatment

Fill a 50 ml[5] 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 ± 2°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 completion of the heat stability treatment, the sample should not be exposed to heat, bright sunshine, or atmospheric humidity.

[5] If a larger quantitiy of the sample is required for the tests, use a 100 ml bottle.

LINDANE[1] EMULSIFIABLE CONCENTRATE

Specification WHO/SIF/5.R7
Approved 25 September 1989

1. Specification

1.1 Description and ingredients

The material shall consist of lindane[1] 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 lindane used in the manufacture of the concentrate shall comply with the requirements of specification WHO/SIT/3.R5

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 *Gamma-HCH isomer content (g/kg basis)*

The content of gamma-HCH isomer determined by the method described in section 2.1, shall not differ from the nominal content by more than the following amounts:

Nominal content	*Tolerance permitted*
Up to 400 g/kg	± 5% of the nominal content
Above 400 g/kg	± 20 g/kg

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

1.2.2 *Alpha-HCH isomer content*

The alpha-HCH isomer content is determined by the method described in section 2.2, shall not be higher than 0.5% of the gamma-HCH isomer content found under 1.2.1.

[1] Lindane is a grade of HCH (BHC) containing not less than 990 g/kg gamma-HCH isomer.

1.2.3 *Water content*

The water content determined by the method described in WHO/M/7.R1, shall not be higher than 1.5 g/kg.

1.2.4 *Acidity or alkalinity*

The acidity or alkalinity of the concentrate, determined by the method described in WHO/M/3, shall not me higher than 0.5 g/kg calculated as H_2SO_4 or 0.5 g/kg calculated as NaOH.

1.2.5 *Cold test*

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

1.2.6 *Flash* point

The flash point of the product shall comply with all national and /or international transport regulations. (see method WHO/M/10.R1).

1.2.7 *Stability of the emulsion*

In standard soft water. Any separation, including creaming/oiling at the top an 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 the method 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 the method WHO/M/13.R3.

1.2.8 *Heat stability*

The concentrate, after treatment as described in section 2.3, shall comply with the requirements of section 1.2.1, 1.2.4 and 1.2.7 of this specification.

1.3 Packing and marking of packages

The lindane emulsifiable concentrate shall be packed in suitable, clean drums, as specified in the order.

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

Manufacturer's name
Lindane emulsifiable concentrate to specification WHO/SIF/5.R7
Gamma-HCH isomer content....g/kg
Batch or reference number, and date of test
Net weight of contents
Instructions for dilution
Date of manufacture

and the following minimum cautionary notice:
Keep well away from foodstuffs and animal feed and their containers.

2. Methods of determining chemical and physical properties

2.1 Gamma-HCH isomer content

2.1.1 *Outline of method*

The sample is dissolved in ethyl acetate with di-n-propyl phthalate added as an internal standard. The gamma-HCH isomer content is determined by gas-liquid chromatography using a flame-ionization detector.

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 and a high-sensitivity flame-ionization detector.

2. *Chromatographic column.* the column should be a borosilicate glass tube 183 cm long, 2 mm in internal diameter, and 6 mm in external diameter, acid washed and dry, bent to fit the chromatograph.

3. *Column packing materiaL.* Chromosorb W-HP (100-120 mesh) treated with 7.5% OV-210.

2.1.3 *Special reagents*

Gamma-HCH isomer standard. Analytical grade, of known purity.
Internal standard. Di-n-propyl phthalate, purity better than 995 g/kg.

2.1.4 *Preparation of standard solution.*

Internal standard solution. Weigh about 2.5 g of di-n-propyl phthalate into a 100 ml volumetric flask, dilute to volume with ethyl acetate and mix.

Gamma-HCH isomer calibration solution. Weigh (to the nearest 0.1 mg) about 250 mg of gamma-HCH isomer standard into a 50 ml screw-capped bottle. Add by pipette 5.0 ml of internal standard solution and 20.0 ml of ethyl acetate and shake for 30 seconds.

2.1.5 *Preparation and conditioning of column*
See method WHO/M/20

2.1.6 *Operating conditions for gas-liquid chromatography*

The temperatures, gas flow rates, and retention times given below are typical values and may have to be adjusted to obtain optimum results from a given apparatus.

Temperatures	
Oven	160°C
Injection port	220°C
Flame-ionization detector	250°C
Gas flow rates	
Hydrogen }	As recommended for the
Air }	detector by the manufacturer
Carrier gas (nitrogen)	10 ml/min.
Retention times	
HCH alpha-isomer peak[2]	11 min.
HCH gamma-isomer peak	14 min.
HCH beta-isomer peak	17 min.
HCH delta-plus epilson-isomers peak	20 min.
Di-n-propyl phthalate peak	23 min.

2.1.7 *Sample preparation and analysis*

Weigh (to the nearest 0.1 mg) a quantity of sample containing about 250 mg of gamma-HCH isomer into a 50 ml screw-capped bottle. Add by pipette 5.0 ml of internal standard solution and 20.0 ml of ethyl acetate and shake for 30 seconds.

Inject 1 µl portions of calibration solution of gamma-HCH isomer until the response ratios (peak area or peak height) between gamma-HCH isomer and internal standard agree to within 2%. Make dupplicate injections of calibration solution followed by duplicate injections of sample solution. Recalibrate after not more than 4 injections of sample solutions.

[2] If this peak appears to be important, proceed to the determination of HCH alpha-isomer content (section 2.2)

2.1.8 *Calculation*

For each injection, the response ratio (r) is given by the equation:

$$r = \frac{\textit{area (or height) of gamma-HCH isomer peak}}{\textit{area (or height) of internal standard peak}}$$

$$\textit{gamma-HCH isomer content (g/kg)} = \frac{r_2 \times m_1 \times p}{r_1 \times m_2}$$

Where r_1 = average response ratio for calibration solution
r_2 = average response ratio for sample solution
m_1= mass of gamma-HCH isomer standard in the calibration solution (mg)
m_2= mass of sample taken (mg)
P = purity of gamma-HCH isomer standard (g/kg)

2.2 Alpha-HCH isomer content

The analytical method is obtainable on request from WHO, Division of Control of Tropical Diseases, CH 1211 Geneva 27, Switzerland.

2.3 Heat stability

Keep 50 ml of the sample for 4\3 days at a temperature of 54± 2°C in a glass container sealed to avoid loss of volatile solvent, and then cool to room temperature.

LINDANE[1] DUSTABLE POWDER

Specification WHO/SIF/17.R6
Approved 25 September 1989

1. Specification

1.1 Description and ingredients

The material shall consist of a homogeneous mixture of lindane together with carriers and any other necessary formulants. It shall be a fine, free-flowing powder free from hard lumps. The lindane used in the manufacture of the powder shall comply with the requirements of specification WHO/SIT/3.R5

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 *Gamma-HCH isomer content (g/kg basis)*

The content of gamma-HCH isomer determined by the method described in section 2.1, shall not differ from the nominal content by more than ± 10 %.

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

1.2.2 *Alpha-HCH isomer content*

The alpha-HCH isomer content determined by the method described in section 2.2, shall not be higher than 0.5% of the gamma-HCH isomer content found under 1.2.1.

1.2.4 *Acidity or alkalinity*

The acidity or alkalinity of the powder, determined by the method described in WHO/M/3, shall not be higher than 1 g/kg calculated as H_2SO_4 or 2 g/kg calculated as NaOH.

[1] Lindane is a grade of HCH (BHC) continaing not less than 990 g/kg gamma-HCH isomer.

1.2.4 *Sieving after heat stability treatment*

Not less than 98% of the powder after heat stability treatment as described in section 2.3 shall pass through a 150 μm sieve when tested by the method described in WHO/M/4.R1. For powders intended for personal use, the residue remaining on the sieve shall be free from grittiness.

1.2.5 *Dustability after heat stability treatment*

After heat stability treatment, as described in section 2.3, the powder shall issue freely without clogging or bridging, when tested in hand dusting apparatus conforming to specification WHO/EQP/4.R2[2].

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 and 1.2.3 of this specification.

1.3 Packing and marking of packages

The lindane dustable powder shall be packed in suitable, clean, airtight drums, as specified in the order.

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

Manufacturer's name
Lindane dustable powder to specification WHO/SIF/17.R6
Gamma-HCH isomer content...g/kg
Batch or reference number, and date of test
Net weight of contents
Date of manufacture

and the following minimum cautionary notice:

Keep well away from foodstuffs and animal feed and their containers.

[2] Equipement for vector control. 3rd ed.Geneva, World Health Organization, 1990, p.128.

2. Methods of determining chemical and physical properties

2.1 Gamma-HCH isomer content

2.1.1. *Outline of method*

Gamma-HCH isomer, is extracted from the sample with ethyl acetate and di-n- propyl phthalate is added as an internal standard. The gamma-isomer content is determined by gas-liquid chromatography using a flame-ionization detector.

2.1.2 *Special apparatus*

1. *Gas-liquid chromatography.* The instrument should be one that is designed for use with glass columns and that is equipped with an on-column injection system and a high-sensitivity flame-ionization detector.

2. *Chromatographic column.* The column should be a borosilicate glass tube 183 cm long, 2 mm in internal diameter, and 6 mm in external diameter, acid washed and dry, bent to fit the chromatograph.

3. *Column packing material.* Chromosorb W-HP (100-120 mesh) treated with 7.5% OV-210.

2.1.3 *Special reagents*

Gamma-HCH isomer standard. Analytical grade, of known purity.
Internal standard. Di-n-propyl phthalate, purity better than 995 g/kg.

2.1.4 *Preparation of standard solutions.*

Internal standard solution. Weigh about 2.5 g of di-n-propyl phthalate into a 100 ml volumetric flask, dilute to volume with ethyl acetate and mix.

Gamma-HCH isomer calibration solution. Weigh (to the nearest 0.1 mg) about 250 mg of gamma-HCH isomer standard into a 50ml screw-capped bottle. Add by pipette 5.0 ml of internal standard solution and 20.0 ml of ethyl acetate and shake for 30 seconds.

2.1.5 *Preparation and conditioning of column*
See method WHO/M/20.

2.1.6 *Operating conditions for gas-liquid chromatography*

The temperatures, gas flow rates, and retention times given below are typical values and may have to be adjusted to obtain optimum results from a given apparatus.

Temperatures	
Oven	160°C
Injection port	220°C
Flame-ionization detector	250°C
Gas flow rates	
Hydrogen}	As recommended for the
Air}	detector by the manufacturer
Carrier gas (nitrogen)	10 ml/min.
Retention times	
HCH alpha-isomer peak[3]	11 min.
HCH gamma-isomer peak	14 min.
HCH beta-isomer peak	17 min.
HCH delta-plus epsilon-isomers peak	20 min.
Di-n-propyl phthalate peak	23 min.

2.1.7 *Sample preparation and analysis*

Weigh (to the nearest 10 mg) a quantity of sample containing about 250 mg of gamma-HCH isomer into a 50 ml screw-capped bottle. Add by pipette 5.0 ml of internal standard solution and 20.0 ml of ethyl acetate and shake for 30 seconds. Filter or centrifuge to remove insoluble particles.

Inject 1 µl portions of calibration solution of gamma-HCH isomer until the response ratios (peak area or peak height) between gamma-HCH isomer and internal standard agree to within 2%. Make duplicate injections of calibration solution followed by duplicate injections of sample solution. Recalibrate after not more than 4 injections of sample solutions.

[3] If this peak appears to be important, proceed to the determination of alpha-HCH isomer content (section 2.2).

2.1.8 *Calculation*

For each injection, the response ratio (r) is given by the equation:

$$r = \frac{\textit{area (or height) of HCH, gamma isomer peak}}{\textit{area (or height) of internal standard peak}}$$

$$\textit{gamma-HCH isomer content (g/kg)} = \frac{r_2 \times m_1 \times p}{r_1 \times m_2}$$

r_1 = average response ratio for calibration solution
r_2 = average response ratio for sample solution
m_1= mass of gamma-HCH isomer standard in the calibration solution (mg)
m_2= mass of sample taken (mg)
P = purity of gamma-HCH isomer standard (g/kg)

2.2 Alpha-HCH isomer content

The analytical method is obtainable on request from WHO, Division of Control of Tropical Diseases, CH 1211 Geneva 27, Switzerland.

2.3 Heat stability treatment

For the sieve test, section 1.2.4, place 20 g of the sample in a 100 ml wide-mouthed bottle fitted with a vinyl-plastic-lined screw-cap. For the dustability test, section 1.2.5, place 250 g of the sample in a 1 litre wide-mouthed bottle fitted with a vinyl-plastic-lined screw-cap. Place the bottles in an oven maintained at 54 ± 2°C for 3 days. Take the samples from the oven and allow them to cool to room temperature before removing the caps.

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