

**LINDANE TECHNICAL
488/TC/M3/-**

As for lindane technical 4/TC/M3/-, CIPAC 1C, p. 1977 together with:

4 Alpha-HCH*

OUTLINE OF METHOD The sample is dissolved in ethyl acetate with hexachlorobenzene added as internal standard. Separation is carried out using a wide bore WCOT fused silica capillary column with automatic injection and flame ionisation detection. The alpha-HCH content is determined by comparison with calibration solutions.

REAGENTS*Ethyl acetate*

Hexachlorobenzene internal standard. Select a batch which, when chromatographed under the conditions given below for the determination of alpha-HCH gives no peak with a similar retention time as for alpha-HCH.

Internal standard solution. Dissolve hexachlorobenzene (0.020 g) in ethyl acetate (100 ml; Solution I). Ensure that a sufficient quantity of this solution is prepared for all samples and calibration standards to be analysed.

Alpha-HCH working standard of known purity

Calibration solution. Weigh in duplicate (to the nearest 0.1 mg) about 0.020 g (*s g*) of alpha-HCH standard into volumetric flasks (100 ml). Dilute to volume with ethyl acetate and shake thoroughly for 5 to 10 min to dissolve the alpha-HCH standard. Transfer by pipette 10.0 ml of each solution into Erlenmeyer flasks (150 ml). Add to each by pipette internal standard solution (solution I, 10.0 ml) and 80 ml of ethyl acetate (Solutions C₁ and C₂).

APPARATUS

Gas chromatograph capable of operating in the range 80 to 300 °C, equipped with a flame ionisation detector, an injector suitable for use with wide bore fused silica capillary columns and an autosampler

Column 15 m × 0.53 (i.d.) mm and 0.83 µm film thickness coated with DB608 (or equivalent).

* Tentative CIPAC method 1994. Based on a method prepared by F Gomez, Rhône Poulenc Agro, France.

Automatic digital integrator or chromatography data system compatible with the gas chromatograph

PROCEDURE

(a) Gas chromatographic conditions (typical):

Injector system

Injector	Split/splitless in the splitless mode with deactivated glass liner. It is very important to minimize the contact of the vapourised sample with any metallic surface. On-column injection may be used.
Injector temperature	250 °C
Splitless time	60 s
Purge flow	60 ml/min
Injection volume	1 µl

Detector system

Type	FID
Range	High sensitivity
Temperature	280 °C

Oven temperatures

Initial temperature	80 °C
Time	1 min
Ramp	10 °C/min up to 250 °C
Final temperature	250 °C
Time	10 min

Gas flow rates

Helium (carrier)	10 ml/min
Nitrogen (make up)) flow rates as recommended for) the detector)
Hydrogen	
Air	

All gases should be purified through molecular sieves. The carrier gas should be further purified through an oxygen trap.

<i>Retention times</i>	hexachlorobenzene : 11.7 min
	alpha-HCH : 12.2 min
	gamma-HCH : 13.2 min

(b) Preparation of sample. Weigh (to the nearest 0.1 mg) enough sample to contain about 1 g (*w g*) of lindane into a screw-capped bottle (150 ml). Add by pipette internal standard solution (10.0 ml) and ethyl acetate (about 90 ml). Shake the flask thoroughly for 5 to 10 min (use an ultrasonic water bath if available) to dissolve the sample (Solution S).

Prepare a solution without internal standard by dissolving enough sample to contain about 1 g of lindane in 100 ml of ethyl acetate (Solution S₀).

(c) *Equilibration of the system.* Carry out 1 µl injections of Solutions I and S₀ and check whether there are any interfering peaks from impurities. If there are, try another batch of hexachlorobenzene or make any necessary corrections by MT 114. Inject calibration solutions C₁ and C₂ to equilibrate the system and use the data from these chromatograms to set the integration parameters. Calculate the relative response factors for these injections to check stability of the instrument. Response factors should not differ by more than ± 1% from the mean.

(d) *Determination.* Inject 1 µl aliquots of the calibration and sample solutions in the following sequence :

C₁, S₁, S₁, C₂, C₁, S₂, S₂, C₂.....

Determine the peak areas.

(d) *Calculation.* Calculate the relative response factors (f_1, f_2) for the pair of calibration injections which bracket the sample injections and obtain the mean response factor (f).

$$f_i = \frac{I_r - s - P}{H_s} 1$$

where :

- f_i = individual response factor
- I_r = area of the internal standard peak in the calibration solution
- H_s = area of the alpha-HCH peak in the calibration solution
- s = mass of alpha-HCH standard in the calibration solution (g)
- P = purity of alpha HCH standard (g/kg)

For each sample injection, calculate the alpha-HCH content.

$$\text{Alpha-HCH content} = \frac{f - H_w}{I_q - w - 10} 2 \text{ g/kg}$$

where :

- f = mean response factor
- H_w = area of the alpha-HCH peak in the sample solution
- I_q = area of the internal standard peak in the sample solution
- w = mass of sample taken (g)

Take the mean of the two values as the alpha-HCH content in the sample.

Repeatability r	= 0.05 to 0.11 g/kg at 0.3 g/kg alpha-HCH content
Reproducibility R	= 0.06 to 0.11 g/kg at 0.3 g/kg alpha-HCH content

LINDANE SUSPENSION CONCENTRATES 488/SC/M/-

As for lindane technical 488/TC/M3/-, except substitute the following for 4(b):

(b) *Preparation of sample.* Weigh (to the nearest 0.1 mg) enough sample to contain about 1 g of lindane (w g) into a screw capped bottle (150 ml). Add by pipette internal standard solution (10.0 ml) and ethyl acetate (about 90 ml). Shake the flask thoroughly for 5 to 10 minutes (use an ultrasonic water bath if available) to dissolve the sample (Solution S). Allow to settle any solid material and filter the supernatant liquid through a suitable filter. Prepare a solution without internal standard by dissolving enough sample to contain about 1 g of lindane in 100 ml of ethyl acetate (Solution S₀). Allow any solid material to settle.

Repeatability r	= 0.25 to 0.26 g/kg at 1.7 g/kg alpha-HCH content
Reproducibility R	= 0.35 to 0.45 g/kg at 1.7 g/kg alpha-HCH content

LINDANE WETTABLE POWDERS 488/WP/M2/-

As for lindane wettable powders 4/WP/M2/-, CIPAC 1C *p.* 1979, together with:

5 Alpha-HCH*. As for lindane suspension concentrates 488/SC/M/4.

Repeatability r	= 0.023 g/kg at 0.27 g/kg alpha-HCH content
Reproducibility R	= 0.044 g/kg at 0.27 g/kg alpha-HCH content

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