

**DICHLOBENIL**  
**73**

See CIPAC 1B, p 1769.

**DICHLOBENIL TECHNICAL**  
**\*73/TC/M2/-**

**GAS CHROMATOGRAPHIC CAPILLARY METHOD**

**1 Sampling.** Take at least 100 g.

**2 Identity tests**

**2.1 GLC.** Use the GLC method below. The difference between the retention times of dichlobenil and the internal standard for the sample solution should not differ by more than 10 s from that for the calibration solution.

**2.2 Infrared.** As for dichlobenil technical **73/TC/M/2** CIPAC 1B, p 1770.

**3 Dichlobenil**

**OUTLINE OF METHOD** Dichlobenil is dissolved in or extracted with acetone. The obtained solution is chromatographed on a capillary column with OV-17 as stationary phase. The dichlobenil content is calculated from the peak area with 4-chlorobenzonitrile as internal standard.

**REAGENTS**

*Acetone*

*Dichlobenil* of known purity

*4-Chlorobenzonitrile* pure (internal standard)

*Internal standard solution.* Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) about 1000 mg (*r* mg) 4-chlorobenzonitrile. Dissolve in acetone and make up to volume with acetone.

*Calibration solution.* Weigh (to the nearest 0.1 mg) into a glass-stoppered conical flask (100 ml) about 100 mg (*s* mg) pure dichlobenil. Add by pipette internal standard solution (10.0 ml), acetone (40 ml) and mix well.

\* CIPAC method 1992. Prepared by the Dutch Committee (DUPAC). Chairman: A. Martijn. Based on a method supplied by Duphar B.V., Netherlands.

## APPARATUS

*Gas chromatograph* with flame ionization detector, suited for capillary gaschromatography with split injection

*Column* fused silica, 30 m × 0.32 (i.d.) mm, coated with 0.25 μm OV-17

*Filter unit* with 0.5 μm filter (Millex SR 0.5 μm or equivalent)

*Ultrasonic bath*

## μPROCEDURE

(a) *Operating conditions (typical):*

<i>Column temperature</i>	130 °C
<i>Injection port temperature</i>	300 °C
<i>Detector temperature</i>	250 °C
<i>Injection mode</i>	split injection
<i>Pressure at split</i>	100 kPa
<i>Carrier gas</i>	nitrogen
<i>Flow rate</i>	75 ml/min (through liner)
<i>Injection volume</i>	1 μl
<i>Retention time</i>	dichlobenil: about 2.3 min 4-chlorobenzonitrile: about 1.3 min

(b) *Preparation of sample.* Weigh (to the nearest 0.1 mg) into a glass-stoppered conical flask (100 ml) enough sample (*w* mg) to contain about 100 mg dichlobenil. Add by pipette internal standard solution (10.0 ml), acetone (40 ml) and mix well.

(c) *Determination.* Inject 1 μl portions of the calibration solution until the response factor agrees within 2%. Otherwise repeat the calibration. Then inject in duplicate 1 μl portions of the sample solution, followed by another calibration solution injection. Measure the relevant peak areas. Calculate the mean response factor from the calibration solution injections preceding and following the two sample solution injections.

$$f = \frac{I_r \times s \times P}{H_s \times r}$$

$$\text{Content of dichlobenil} = \frac{f \times H_w \times r}{I_q \times w} \text{ g/kg}$$

## DICHLOBENIL 73

where:

- $H_s$  = peak area of dichlobenil in the calibration solution  
 $I_r$  = peak area of the internal standard in the calibration solution  
 $H_w$  = peak area of dichlobenil in the sample solution  
 $I_q$  = peak area of the internal standard in the sample solution  
 $s$  = mass of dichlobenil in the calibration solution (mg)  
 $r$  = mass of internal standard in the calibration and sample solution (mg)  
 $w$  = mass of sample taken  
 $P$  = purity of the standard dichlobenil (g/kg)  
 $f$  = response factor

**Repeatability r** = 24.6 g/kg at 950 g/kg active ingredient content

**Reproducibility R** = 24.6 g/kg at 950 g/kg active ingredient content

### DICHLOBENIL WETTABLE POWDERS

\*73/WP/M2/-

**1 Sampling.** Take at least 500 g.

**2 Identity tests.** As for dichlobenil wettable powders 73/WP/M/2, CIPAC 1B, p 1772 and technical 73/TC/M2/2.

**3 Dichlobenil** As for dichlobenil technical 73/TC/M2/3 except:

(b) *Preparation of sample.* Weigh (to the nearest 0.1 mg) into a glass-stoppered conical flask (100 ml) enough sample ( $w$  mg) to contain about 100 mg dichlobenil. Add by pipette calibration solution (10.0 ml) and acetone (25 ml). Place in an ultrasonic bath for 10 min. Add acetone (15 ml), mix well and allow to settle any solid material. If necessary filter the solution through a 0.5  $\mu$ m filter. Otherwise use the clear supernatant.

**4 Suspensibility.** As for dichlobenil wettable powders 73/WP/M/4, CIPAC 1B, p 1772.

\* CIPAC method 1992. Prepared by the Dutch Committee (DUPAC). Chairman: A. Martijn. Based on a method supplied by Duphar B.V., Netherlands.

**DICHLOBENIL GRANULES**  
**\*73/GR/M2/-**

1 Sampling. Take at least 1 kg.

2 Identity tests. As for dichlobenil wettable powders, 73/WP/M2/2.

3 Dichlobenil. As for dichlobenil wettable powders **73**/WP/M2/3.

**Repeatability r** = 1.0 g/kg at 40 g/kg active ingredient content  
2.3 g/kg at 70 g/kg active ingredient content  
3.7 g/kg at 200 g/kg active ingredient content

**Reproducibility R** = 1.9 g/kg at 40 g/kg active ingredient content  
3.1 g/kg at 70 g/kg active ingredient content  
5.3 g/kg at 200 g/kg active ingredient content

\* CIPAC method 1992. Prepared by the Dutch Committee (DUPAC). Chairman: A. Martijn. Based on a method supplied by Duphar B.V., Netherlands.