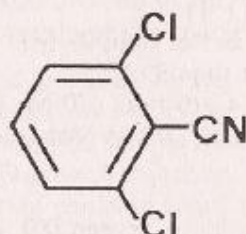


DICHLOBENIL 73

**DICHLOBENIL**

73



<i>ISO common name</i>	Dichlobenil
<i>Chemical name</i>	2,6-Dichlorobenzonitrile (IUPAC and CA; 1194-65-6)
<i>Empirical formula</i>	C <sub>7</sub> H <sub>3</sub> Cl <sub>2</sub> N
<i>RMM</i>	172.0
<i>m.p.</i>	145–146°C
<i>v.p.</i>	74 mPa (at 20°C) 148.8 Pa (at 100°C)
<i>Solubility</i>	Water 18 mg/l (at 20°C) Slightly soluble in organic solvents
<i>Description</i>	The pure material is a white crystalline solid with an aromatic odour
<i>Formulations</i>	Formulated as wettable powders and granules

**DICHLOBENIL TECHNICAL**

\*73/TC/M/-

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

2 **Identity tests**

**Infrared.** Prepare 13 mm diameter KBr discs using 700 µg of sample and about 150 mg of KBr and also using a standard dichlobenil. Scan the discs from 4000–250 cm<sup>-1</sup>. The spectrum produced from the sample disc should not differ significantly from that from the standard.

3 **Dichlobenil**

**OUTLINE OF METHOD** Dichlobenil is dissolved in dichloroethane and chromatographed on Chromosorb P coated with Carbowax 20 M. The content of dichlobenil is determined using methyl myristate as internal standard.

\* CIPAC method 1976. Prepared by the Dichlobenil Panel, NL; Chairman: A van Rossum (Duphar).

## REAGENTS

*1,2-dichloroethane*, pure

*Diethylether*, pure

*Dichloroethane—diethylether* (1:1)

*Dichlobenil*, of known purity, better than 99.5%

*Methyl myristate*, purity better than 99.5%

*Column* packed with 10% Carbowax 20 M (Applied Science Lab.) on Chromosorb PAW, 100-200 mesh (Johns-Manville) (*Note* 1)

*Nitrogen*, pure (carrier gas)

*Hydrogen*, pure (burner gas)

*Air* (burner gas): pure nitrogen/pure oxygen (80 + 20)

*Internal standard solution*: Dissolve 0.80 g methyl myristate (*q* g) in 100 ml dichloroethane-diethylether.

*Calibration solution*: Weigh accurately into a conical flask a mass of about 0.10 g (= *r* g) of pure dichlobenil. Pipette in 5.00 ml of the internal standard solution and add 45 ml of dichloroethane-diethylether.

## APPARATUS

*Gas chromatograph*, suitable for on-column injection, glass columns, equipped with flame ionization detector, injection port heating and preferably detector heating.

*Recorder*

*Microsyringe*, 10  $\mu$ l

*Magnetic stirrer*

## PROCEDURE

(a) *Column preparation*. Dissolve 2 g of Carbowax 20 M in chloroform. Transfer this solution to a suction flask, add slowly 18 g of Chromosorb PAW, 100-120 mesh and add sufficient chloroform to cover the solid support completely.

Reduce the pressure in the flask with a water aspirator for 2 minutes. Transfer the contents to a crystallizing dish.

Heat the slurry under careful stirring until the liquid has evaporated but the solid support is still moist.

Then heat without stirring until the solid support is virtually dry.

Finally dry under vacuum at 60°C for 16 hours. Remove fine particles by sieving over a 120-mesh sieve.

Fill a 1.8 m glass column (3 mm int. diam.; 6 mm ext diam.) with the column packing. Condition the column at 210°C for at least 16 hours, using carrier gas at about 25 ml/min. Do not connect the column to the detector during this process.

(b) *Operating conditions*.

*Column temperature*: 200°C

*Injection port temperature*: 210°C

*Detector temperature*: 210°C

*Flow rate carrier gas*: 25 ml/min

(c) *Sample preparation.* Weigh, to the nearest mg, into a 100 ml volumetric flask a mass of approximately 2.0 g ( $w$  g) of dichlobenil. Dissolve and make up to volume with dichloroethane-diethylether. Pipette 5.00 ml ( $x$  ml) of this solution into a 100 ml conical flask. Add 5.00 ml of the internal standard solution and complete to 50 ml with dichloroethane-diethylether.

(d) *Determination.* Inject 2  $\mu$ l portions of the calibration solution until the response factor ( $f$ ) varies by less than 1% for successive injections (*Note 2*). Inject in duplicate 2  $\mu$ l portions of the sample solution, followed by 2  $\mu$ l portions of the calibration solution. For each injection measure the response of the dichlobenil and methyl myristate peaks in terms of:

- (a) the peak height multiplied by retention time or,
- (b) a digital integrator count.

(e) *Calculations.* Calculate the response factor ( $f$ ) of pure dichlobenil using the following formula:

$$f = \frac{I_q \times r \times v_1 \times p}{I_c \times q \times 100}$$

$I_q$  = peak area of the internal standard in calibration solution

$I_c$  = peak area of standard dichlobenil in calibration solution

$p$  = purity of dichlobenil % m/m

$q$  = mass of internal standard in g

$r$  = mass of standard dichlobenil in g

$v_1$  = dilution factor of the internal standard =  $\frac{100}{5} = 20$

The response factor of dichlobenil with respect to methyl myristate is approx. 1.5.

The content of dichlobenil:

$$= \frac{I_d \times q \times f \times v_2 \times 100}{I_m \times w \times v_1} \text{ m/m \%}$$

$I_d$  = peak area of dichlobenil in sample solution

$I_m$  = peak area of the internal standard in sample solution

$f$  = mean response factor found

$q$  = mass of internal standard in g

$v_1$  = dilution factor of the internal standard =  $\frac{100}{5} = 20$

$v_2$  = dilution factor of the sample concerned =  $\frac{100}{x}$

$x$  = ml pipetted for in the conical flask

$w$  = mass of sample in g

*Note 1* As alternative support, Gas Chrom. R can be used.

*Note 2* The relative retention time of dichlobenil with respect to methyl myristate (1.00; approx. 7 min) is 1.60.

## DICHLOBENIL WETTABLE POWDERS

\*73/WP/M/

- 1 **Sampling.** Take at least 500 g.
- 2 **Identity test.** Extract the active ingredient with 1,2-dichloroethane, evaporate the solvent and confirm the identity by 73/TC/M/2.

3 **Dichlobenil**

**OUTLINE OF METHOD** Dichlobenil is extracted with dichloroethane-diethylether and determined as for the technical material.

**REAGENTS AND APPARATUS** As for 73/TC/M/3.

**PROCEDURE** As for 73/TC/M/3 except

(c) *Sample preparation*

Weigh, to the nearest mg, sufficient sample ( $w$  g) to contain approximately 1.0 g of dichlobenil into a 100 ml volumetric flask. Add dichloroethane-diethylether, swirl, make up to volume and mix. Allow the insoluble material to settle and pipette 10.00 ml ( $x$  ml) of the clear supernatant solution into a 100 ml conical flask. Continue according to 73/TC/M/3.c from 'Add 5.00 ml . . . '.

4 **Suspensibility**

- (a) *Preparation of suspension.* MT 15.1 (i)
- (b) *Determination of sedimentation.* MT 15.1 (ii)
- (c) *Determination of dichlobenil in the bottom 25 ml of suspension*

**APPARATUS**

As for 73/TC/M/3 together with:  
*Separating funnel, 250 ml.*

**DETERMINATION**

After removal of the top 225 ml of suspension (MT 15.1), transfer the residual 25 ml quantitatively with water to a separating funnel. Extract two times with 10.00 ml of dichloroethane. Collect the dichloroethane layer in a 100 ml conical flask. Add 5.00 ml of the internal standard solution and 25 ml of dichloroethane-diethylether.

Proceed according to 73/TC/M/3.d.

The mass of dichlobenil in the bottom 25 ml is:

$$\text{mass of dichlobenil} = \frac{I_d \times q \times f}{I_m \times v_1} = Q \text{ g}$$

(d) *Calculation*

$$\text{Suspensibility} = \frac{111(c - Q)}{c} \%$$

where:

$c$  g = mass of dichlobenil in the sample taken for preparing the suspension.

\* CIPAC Method 1976.

DICHLOBENIL GRANULES

\*73/GR/M/-

- 1 **Sampling.** Take at least 1 kg.
- 2 **Identity test.** Extract the active ingredient with 1,2-dichloroethane, evaporate the solvent and confirm the identity by 73/TC/M/2.

3 **Dichlobenil**

**OUTLINE OF METHOD** Dichlobenil is extracted with dichloroethane and determined as for the technical material.

**REAGENTS AND APPARATUS** As for 73/TC/M/3 together with *borosilicate glass filter paper* (Whatman GF 82)

**PROCEDURE** As for 73/TC/M/3 except

(c) *Sample preparation*

Weigh to the nearest 0.01 g about 6.0 g (w g) sample into a 100 ml conical flask. Add 20 ml of dichloroethane and stir for 10 minutes, using a magnetic stirrer.

Transfer to a glass filter provided with a glass fibre paper and filter with suction.

Wash the granules 5 × with 5 ml of dichloroethane.

Collect the filtrates in a 100 ml volumetric flask and make up to volume with diethylether.

Pipette into a 100 ml conical flask a quantity (x ml) of this solution containing about 0.1 g dichlobenil (*Note 3*).

Continue according to 73/TC/M/3.c from 'Add 5.00 ml . . . '.

*Note 3* For 20% granules pipette 10.00 ml.

For 7% granules pipette 25.00 ml.

<sup>1</sup>CIPAC Method 1976.