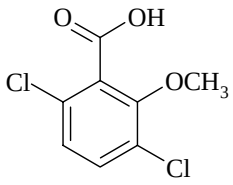
# DICAMBA
# 85




| | |
|---|---|
| *ISO common name* | Dicamba |
| *Chemical name* | 3,6-Dichloro-2-methoxybenzoic acid (IUPAC and CA; *1918-00-9*) |
| *Empirical formula* | $C_8H_6Cl_2O_3$ |
| *RMM* | 221.0 |
| *m.p.* | 114-116 °C |
| *v.p.* | $1.67 \times 10^{-3}$ Pa at 25 °C |
| *Solubility* | In water 6 g/l at 25 °C, soluble in most organic solvents like ethanol, acetone, dioxane and dimethyl sulfoxide |
| *Description* | Colourless solid |
| *Stability* | Very stable under normal conditions |
| *Formulations* | Soluble liquids, water dispersible granules |

# DICAMBA TECHNICAL
## *85/TC/M/-

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

**2 Identity tests**

**2.1 HPLC.** Use the HPLC method described below. The retention time of dicamba for the sample solution should not deviate from that of the calibration solution by more than 0.2 minutes.

**2.2 Infrared.** Prepare potassium bromide discs from the sample and from dicamba standard. Scan the discs from 4000-600 $cm^{-1}$ The spectrum obtained from the sample should not differ significantly from that of the standard (Fig. 11).

**3 Dicamba**

OUTLINE OF METHOD Dicamba is dissolved in methanol and determined by high performance liquid chromatography on a reversed phase column ($RP_{18}$) using UV detection and external standardisation.

REAGENTS

*Acetic acid* glacial

*Dicamba* of known purity better than 980 g/kg

*Methanol* eluent B

*Phosphoric acid*

*Phosphoric acid* 0.1% aqueous solution, eluent A

*Calibration solution.* Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) 90 to 110 mg of dicamba reference standard (*s* mg). Dissolve and make up to volume with methanol. Prepare in duplicate (solutions $C_1$ and $C_2$).

APPARATUS

*High performance liquid chromatograph* equipped with a UV detector, an automatic injector (10 µl) and an electronic integrator or data system

*Chromatographic column* 250 × 4.0 mm (i.d.) Nucleosil $C_{18}$ column, 5 µm or equivalent

* CIPAC method 2001, Prepared by the Swiss Committee. Chairman M D Müller. Based on a method supplied by Novartis, Schwitzerland.

PROCEDURE

*(a) Operating conditions* (typical):

| | | | | |
|---|---|---|---|---|
| *Column temperature*: | 20 - 40 °C | | | |
| *Gradient* | time (min) | A | B | v/v |
| | 0 | 65 | 35 | |
| | 20 | 15 | 85 | |
| | 21 | 65 | 35 | |
| | 30 | 65 | 35 | |
| *Flow rate* | 1.5 ml/min | | | |
| *Detector wavelength* | 280 nm | | | |
| *Injection volume* | 10 µl | | | |
| *Retention time* | dicamba: about 12.2 min | | | |

*(b) Preparation of sample solution*. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) sufficient sample to contain 90 to 110 mg of dicamba (*w* mg). Dissolve and make up to volume with methanol.

*(c) System equilibration*. Inject 10 µl portions of calibration solution $C_1$ until the response factor of dicamba obtained for two consecutive injections differs by less than 1%. Then inject 10 µl portions of calibration solution $C_2$. The response factor of dicamba for this solution should not deviate by more than 1% from that for the first calibration solution, otherwise prepare new calibration solutions.

*(d) Determination*. Inject in duplicate 1 µl portions of the calibration solutions ($C_1$ and $C_2$) and sample solutions ($S_1$, $S_2$, ...) in the following sequence:

$$C_1, C_1, S_1, S_1, S_2, S_2, C_2, C_2, S_3, S_3, S_4, S_4, C_1, C_1\ldots.$$

Calculate the mean of the four response factors (*f*) for dicamba from the calibration solutions bracketing the injections of the two sample solutions. Use these values for calculating the contents of these two samples.

*(e) Calculation.* Calculate the response factor (*f*) for dicamba using the following formula:

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

$$\text{Content of dicamba} = \frac{H_x \times f}{w} \text{ g/kg}$$

where:
$H_s$ = peak area of dicamba in the calibration solution
$H_w$ = peak area of dicamba in the sample solution
$f$ = response factor
$s$ = mass of dicamba in the calibration solution (mg)
$w$ = mass of the sample taken (mg)
$P$ = purity of dicamba reference standard (g/kg)

**Repeatability r** = 24 g/kg at 902 g/kg active ingredient content
**Reproducibility R** = 36 g/kg at 902 g/kg active ingredient content

Based on a collaborative study with 31 participants and 124 values.

## DICAMBA SOLUBLE LIQUIDS
## *85/SL/M/-

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

**2 Identity tests**
**2.1 HPLC.** As for **85**/TC/M/2.1.
**2.2 Infrared.** Acidify the test material with hydrochloric acid and extract the dicamba acid with diethyl ether. Extract the ether layer with potassium hydrogen carbonate. Discard the ether layer, acidify the aqueous layer with hydrochloric acid and evaporate the aqueous phase to dryness. Proceed as for with dicamba technical **85**/TC/M/2.2.

**3 Dicamba.** As for **85**/TC/M/3.

**Repeatability r** = 12 at 402 g/kg active ingredient content
**Reproducibility R** = 16 at 402 g/kg active ingredient content

Based on a collaborative study with 28 participants and 112 values.

* CIPAC method 2001. Prepared by the Swiss Committee. Chairman M D Müller. Based on a method supplied by Novartis, Schwitzerland.

## DICAMBA WATER DISPERSIBLE GRANULES
## *85/WG/M/-

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

**2 Identity tests**
**2.1 HPLC.** As for **85**/TC/M/2.1.
**2.2 Infrared.** As for dicamba soluble liquids **85**/SL/M/2.2.

**3 Dicamba.** As for **85**/TC/M/3 except:

REAGENTS
add:
*Extraction solvent*, methanol-glacial acetic acid, 98 + 2 (v/v)

and change *(b) Preparation of sample solution* to:
Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) sufficient sample to contain 90 to 110 mg of dicamba (*w* mg). Add extraction solvent (90 ml) and place the sample in an ultrasonic bath for approximately 10 min. Allow to cool to room temperature and make up to volume extraction solvent. Clarify the solution by filtration or centrifugation.

**Repeatability r** = 21 at 709 g/kg active ingredient content
**Reproducibility R** = 24 at 709 g/kg active ingredient content

Based on a collaborative study with 27 participants and 108 values.

**4 Suspensibility** (Draft)

*(a) Preparation of suspension and determination of sedimentation.* MT 168.

*(b) Determination of dicamba in the bottom 25 ml of suspension*. After removal of the top 225 ml of suspension, transfer the bottom 25 ml of suspension quantitatively with water to a cylindrical flask (100 ml). Evaporate the water by heating in a water bath at about 90°C. Add methanol (100.0 ml) and place the sample in an ultrasonic bath for approximately 5 min. Centrifuge or filter an aliquot of the sample solution and inject this clear solution. Determine the mass (*Q* mg) according to **85**/TC/M/3 using a calibration solution with the appropriate mass of dicamba.

* CIPAC method 2001. Prepared by the Swiss Committee. Chairman M D Müller. Based on a method supplied by Novartis, Schwitzerland.

*(c) Calculation*

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

where:

$c$ = mass of dicamba in the sample for preparing the suspension (mg)

$Q$ = mass of dicamba in the bottom 25 ml of suspension (mg)

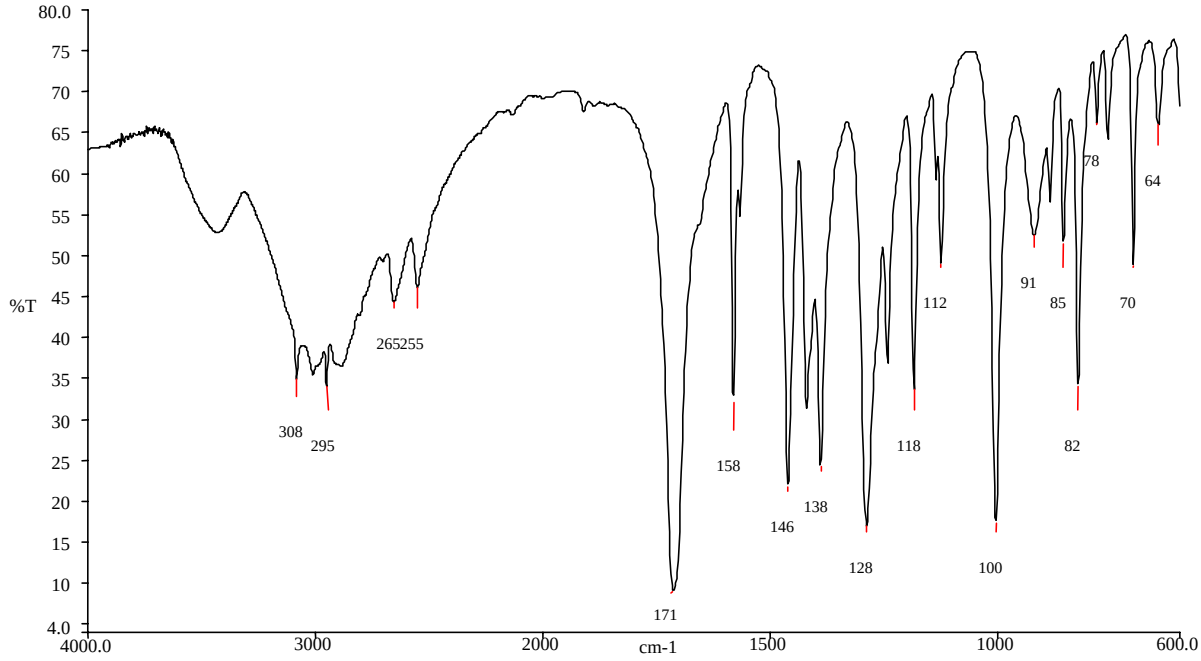



**Fig. 11** Infrared spectrum of dicamba