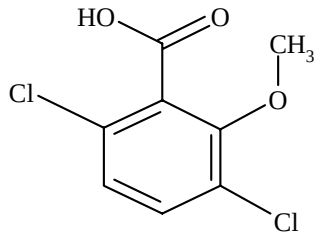
DICAMBA
85



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 to 116 °C
v.p.	0.5 Pa at 100 °C
Solubility	In water: 4.5 g/l at 25 °C; moderately soluble in xylene; readily soluble in ethanol and ketones
Description	White crystalline solid
Stability	Stable and resistant to strong oxidation and hydrolysis under normal conditions
Formulations	Amine salt aqueous solutions and in mixtures with 2,4-D, MCPA and mecoprop as potassium or amine salt solutions

DICAMBA TECHNICAL
*__85__/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

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3 Dicamba

OUTLINE OF METHOD The sample is dissolved in carbon disulphide and dicamba is determined by infrared spectroscopy.

REAGENTS

Dicamba standard of known purity
Carbon disulphide
Calibration solution. Weigh (to the nearest 0.1 mg) 200 ± 5 mg (*s* mg) dicamba into a volumetric flask (25 ml). Add carbon disulphide, shake and dilute to volume with carbon disulphide.

APPARATUS

Infrared spectrophotometer
Sodium chloride cells matched pair, 0.2 mm

PROCEDURE

(a) Preparation of sample. Weigh (to the nearest 0.1 mg) enough sample to contain 200 ± 5 mg (*w* mg) dicamba into a volumetric flask (25 ml), add carbon disulphide, shake to dissolve and dilute to volume with carbon disulphide.

(b) Determination. Record the spectra of the sample and calibration solutions from 1100 to 930 cm^{-1} (9.3 to 10.7 μm) using sodium chloride cells. Use carbon disulphide in the reference cell. Determine the absorbance ΔA of the sample and $\Delta A'$ of the standard at 1012 cm^{-1} (9.89 μm) from horizontal baseline tangent to the minimum between 1075 and 1035 cm^{-1} (9.3 to 9.66 μm).

*AOAC-CIPAC method 1973.

(c) Calculation

$$\text{Dicamba content} = \frac{\Delta A \times s \times P}{w \times \Delta A'} \text{ 1g/kg}$$

where:

ΔA = difference of the absorbance of the sample at 1012 cm^{-1} and the baseline absorbance tangent to minimum between 1075 and 1035 cm^{-1}

$\Delta A'$ = difference of the absorbance of the standard at 1012 cm^{-1} and the baseline absorbance tangent to minimum between 1075 and 1035 cm^{-1}

s = mass of dicamba in the calibration solution (mg)

w = mass of sample taken (mg)

P = purity of dicamba standard (g/kg)

DICAMBA DIMETHYLAMINE AQUEOUS SOLUTIONS

[*] **85.102**/SL/M/-

1 Sampling. Take at least 1 l.

2 Identity tests

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3 Dicamba

OUTLINE OF METHOD The sample is dissolved in acetone and dicamba is determined by infrared spectroscopy using barium fluoride cells.

REAGENTS

Dicamba standard of known purity

Acetone infrared quality

Dimethyl amine DMA, 60 % aqueous solution

Calibration solution. Weigh (to the nearest 0.1 mg) 11.98 ± 0.02 g (*s* mg) dicamba standard into a beaker (50 ml). Add water (5 ml) and DMA solution (4 ml). Adjust the pH to 7.0 using a magnetic stirrer (all solids should be dissolved by this time). Rinse each pH electrode twice with water (2 × 1 ml, i.e. a total of 4 ml) collecting the rinses in the beaker. Cool the solution to room temperature and transfer quantitatively to a volumetric flask (25 ml). Rinse the beaker twice with water collecting the rinses in the flask. Dilute to volume with water and mix thoroughly. Pipette 5.0 ml of this solution into a volumetric flask (25 ml) and dilute to

[*] AOAC-CIPAC method 1973.

volume with acetone.

APPARATUS

Infrared spectrophotometer
Barium fluoride cells pathlength 25 µm
pH meter
Magnetic stirrer

PROCEDURE

(a) Preparation of sample. Weigh (to the nearest 0.1 mg) enough sample to contain 2.4 g (*w* mg) dicamba into a volumetric flask (25 ml), add acetone, shake to dissolve and make up to volume with acetone.

(b) Determination. Record the spectra of the sample and calibration solutions from 1070 to 930 cm^{-1} (9.3 to 10.7 µm) using the barium fluoride cell. Use air in the reference beam. Determine the absorbance ΔA of the sample and $\Delta A'$ of the standard at 1012 cm^{-1} (9.89 µm) from horizontal baseline tangent to the minimum between 1020 and 1070 cm^{-1} (9.4 to 9.7 µm).

(d) Calculation

$$\text{Dicamba content} = \frac{\Delta A \times s \times P}{w \times \Delta A' \times 5} \text{ 2g/kg}$$

where:
ΔA = difference of the absorbance of the sample at 1012 cm^{-1} and the baseline absorbance tangent to minimum between 1020 and 1070 cm^{-1}
$\Delta A'$ = difference of the absorbance of the standard at 1012 cm^{-1} and the baseline absorbance tangent to minimum between 1020 and 1070 cm^{-1}
s = mass of dicamba in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of dicamba standard (g/kg)

DICAMBA + MCPA DIMETHYLAMINE AQUEOUS SOLUTIONS
*85.102 + 2.102/SL/M/-

1 Sampling. Take at least 1 l.

2 Identity tests

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3 Dicamba and MCPA

OUTLINE OF METHOD The active ingredients are precipitated with hydrochloric acid, extracted with chloroform and evaporated to dryness. The residue is dissolved in acetone and dicamba and MCPA are determined by infrared spectroscopy.

REAGENTS

Dicamba standard of known purity
MCPA standard of known purity
Acetone infrared quality
Chloroform
Hydrochloric acid concentrated solution
Sodium sulphate anhydrous, freshly dried at 105 °C for 2 h and coarsely powdered
Calibration solution. Weigh (to the nearest 0.1 mg) between 180 and 220 mg dicamba and between 580 and 620 mg of MCPA (*s* mg respectively) standard into a tared weighing bottle. Pipette acetone (25.0 ml) into the bottle and swirl until completely dissolved.

APPARATUS
Infrared spectrophotometer
Sodium chloride cells matched pair, pathlength 0.2 mm

PROCEDURE

(a) Preparation of sample. Weigh (to the nearest 0.1 mg) enough sample to contain between 180 and 220 mg dicamba and between 580 and 620 mg of MCPA (*w* mg respectively) into a tared weighing bottle. Add water (5 ml) and transfer quantitatively to a separating funnel (125 ml) with water (5 to 10

*Provisional AOAC-CIPAC method 1972.

ml).
Add hydrochloric acid dropwise with constant stirring to pH 1 (check with indicator paper), then add excess (5 drops). Pipette in chloroform (25 ml) and shake to dissolve the precipitate. Run the chloroform layer into a conical flask (125 ml) and re-extract the aqueous layer with chloroform (2 x 15 ml). Add boiling chips to the combined extracts and evaporate on a steam bath to complete dryness. Allow to dry in the hood overnight at room temperature (Do not dry in air or a vacuum oven). Pipette in acetone (25.0 ml) and swirl to dissolve completely the residue. Add a few g of anhydrous sodium sulphate if there is any water present.

(b) Determination. Record the infrared spectrum and measure the absorbance in the matched cells with acetone in the reference cell at the following wavelengths:

Range	1135 to 930 cm^{-1} (8.8 to 10.75 µm)
Dicamba peak	1012 cm^{-1} (9.89 µm)
MCPA peak	1070 cm^{-1} (9.35 µm). Other isomers of MCPA will contribute to the absorbance at this wavelength and, if present, a high MCPA content will be obtained.
Baseline	Horizontal tangent to minimum at 970 to 965 cm^{-1} (10.3 to 10.4 µm) for both constituents

$$\text{MCPA (or dicamba) content} = \frac{\Delta A \times s \times P}{w \times \Delta A' \times 5} 3\text{g/kg}$$

where:

ΔA = difference of the absorbance of MCPA (or dicamba) for the sample at 1070 (or 1012 cm^{-1} respectively) and the baseline absorbance tangent to minimum between 970 to 965 cm^{-1}

$\Delta A'$ = difference of the absorbance of MCPA (or dicamba) for the standard at 1070 (or 1012 cm^{-1} respectively) and the baseline absorbance tangent to minimum between 970 to 965 cm^{-1}

s = mass of MCPA (or dicamba) in the calibration solution (mg)

w = mass of sample taken (mg)

P = purity of MCPA (or dicamba) standard (g/kg)

DICAMBA + 2,4-D DIMETHYLAMINE AQUEOUS SOLUTIONS
[*]**85.102 + 1.102**/SL/M/-

1 Sampling. Take at least 1 l.

2 Identity tests

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3 Dicamba and 2,4-D

OUTLINE OF METHOD The active ingredients are precipitated with hydrochloric acid, extracted with chloroform and evaporated to dryness. The residue is dissolved in in acetone and dicamba and 2,4-D are determined by infrared spectroscopy.

REAGENTS

Dicamba standard of known purity
2,4-D standard of known purity
Acetone infrared quality
Chloroform
Hydrochloric acid concentrated solution
Sodium sulphate anhydrous, freshly dried at 105 °C for 2 h and coarsely powdered
Calibration solution. Weigh (to the nearest 0.1 mg) between 180 and 220 mg dicamba and between 380 and 420 mg of 2,4-D (*s* mg respectively) standard into a tared weighing bottle. Pipette acetone (25.0 ml) into the bottle and swirl until completely dissolved.

APPARATUS

Infrared spectrophotometer
Sodium chloride cells matched pair, pathlength 0.2 mm

PROCEDURE

(a) Preparation of sample. Weigh (to the nearest 0.1 mg) enough sample to contain between 180 and 220 mg dicamba and between 380 and 420 mg of 2,4-D (*w* mg respectively) into a tared weighing bottle. Add water (5 ml) and transfer quantitatively to a separating funnel (125 ml) with water (5 to 10 ml).

[*]Provisional AOAC-CIPAC method 1972.

Add hydrochloric acid dropwise with constant stirring to pH 1 (check with indicator paper), then add excess (5 drops). Pipette in chloroform (25 ml) and shake to dissolve the precipitate. Run the chloroform layer into a conical flask (125 ml) and re-extract the aqueous layer with chloroform (2 × 15 ml). Add boiling chips to the combined extracts and evaporate on a steam bath to complete dryness. Allow to dry in the hood overnight at room temperature (Do not dry in air or a vacuum oven). Pipette in acetone (25.0 ml) and swirl to dissolve completely the residue. Add a few g of anhydrous sodium sulphate if there is any water present.

(b) Determination. Record the infrared spectrum and measure the absorbance in the matched cells with acetone in the reference cell at the following wavelengths:

Range	1139 to 930 cm^{-1} (8.8 to 10.75 μm)
Dicamba peak	1012 cm^{-1} (9.89 μm)
2,4-D peak	1080 cm^{-1} (9.26 μm).
Baseline	Horizontal tangent to minimum at 970 to 960 cm^{-1} (10.3 to 10.4 μm) for both constituents

$$\text{2,4-D (or dicamba) content} = \frac{\Delta A \times s \times P}{w \times \Delta A' \times 5}\ 4\text{g/kg}$$

where:

ΔA = difference of the absorbance of 2,4-D (or dicamba) for the sample at 1080 (or 1012 cm^{-1} respectively) and the baseline absorbance tangent to minimum between 970 to 960 cm^{-1}

$\Delta A'$ = difference of the absorbance of 2,4-D (or dicamba) for the standard at (1080 or 1012 cm^{-1} respectively) and the baseline absorbance tangent to minimum between 970 to 960 cm^{-1}

s = mass of 2,4-D (or dicamba) in the calibration solution (mg)

w = mass of sample taken (mg)

P = purity of 2,4-D (or dicamba) standard (g/kg)