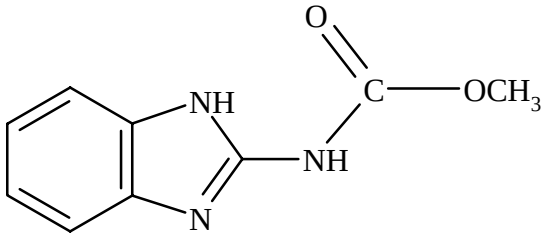
CARBENDAZIM
263



ISO common name	Carbendazim
Chemical name	Methyl benzimidazol-2-ylcarbamate] (IUPAC); 1*H*-benzimidazol-2-yl carbamic acid methyl ester (CA; *10605-21-7*)
Empirical formula	$C_9H_9N_3O_2$
RMM	191.2
m.p.	310 °C (decomp.)
v.p.	Less than 10^{-9} Pa at 20 °C
Solubility	In water: 8 mg/l at 20°C; nearly insoluble in common organic solvents
Description	White, odourless solid
Stability	Stable for at least 2 years at 50 °C. Stable under acidic and neutral conditions, but hydrolysed under alkaline conditions
Formulations	Wettable powders, water dispersible granules and suspension concentrates

CARBENDAZIM TECHNICAL
[*]**263**/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The retention time of carbendazim in the sample solution should not deviate by more than 1% from that of the calibration solution.

2.2 UV. Use the UV method below. The spectra obtained from the sample and the standard should not differ significantly.

2.3 Infrared. Prepare potassium bromide discs from the sample and pure carbendazim using 700 mg material and 150 mg potassium bromide. Scan the discs from 4000 to 600 cm^{-1}. The spectrum obtained from the sample disc should not differ significantly from that of the standard.

3 Carbendazim

3.1 Ultraviolet spectroscopic method

OUTLINE OF METHOD Carbendazim is dissolved in aqueous hydrochloric acid and the absorption of the solution is measured between 220 and 350 nm. The content is calculated from the absorption at 283 nm.

REAGENTS

Carbendazim standard of known purity (minimum purity 990 g/kg)
Hydrochloric acid aqueous solution, *c*(HCl) = 1 mol/l

APPARATUS

Spectrophotometer
Cuvettes quartz, 1 cm
Volumetric flasks 500 ml
Pipettes 25 ml
Ultrasonic bath

PROCEDURE

(a) Preparation of the calibration solution. Weigh (to the nearest 0.1 mg) into a volumetric flask (500 ml) about 80 mg (*s* mg) pure carbendazim, add hydrochloric acid solution (about 250 ml, *c* (HCl) = 1 mol/l) and place the flask in an ultrasonic bath for at least 20 min. If no ultrasonic bath is available

[*] CIPAC method 1982. Prepared by the the German PAC (DAPA). Chairman: W Weinmann. Based on a method supplied by BASF AG, Germany.

allow to stand the solution for at least 2 h with occasional shaking. Very pure material with a well developed crystal structure may need more time for dissolution. Allow to cool to room temperature and make up to volume with hydrochloric acid solution. Transfer by pipette 25 ml of this solution to a volumetric flask (500 ml) and dilute to volume with hydrochloric acid solution.

(b) Preparation of sample. Weigh (to the nearest 0.1 mg) into a volumetric flask (500 ml) sufficient sample to contain about 80 mg (*w* mg) carbendazim and proceed as for *(a) Preparation of the calibration solution* from: 'add hydrochloric acid...'

(c) Determination. Measure the UV absorption of the calibration and sample solutions between 220 and 350 nm. The peak heights should be about the same. If not, adjust the sample weight and repeat the measurement. Draw for each curve the baseline from valley to valley (at about 256 and 304 nm) and determine the distance between the maximum of the peak and the baseline for the sample and the calibration solutions (*A* and *A'* respectively) at 283 nm.

(d) Calculation

$$\text{Content of carbendazim} = \frac{A \times s \times P}{A' \times w} \text{ g/kg}$$

where:

A = the peak height of the sample solution
A' = the peak height of the calibration solution
s = mass of carbendazim in the calibration solution (mg)
w = mass of carbendazim in the sample solution (mg)
P = purity of carbendazim standard (g/kg)

Repeatability r = 10 g/kg at 993 and 986 g/kg active ingredient content
= 9 g/kg at 903 g/kg active ingredient content
Reproducibility R = 20 - 41 g/kg at 903 and 986 g/kg active ingredient content
= 39 g/kg at 903 g/kg active ingredient content

3.2 HPLC method

OUTLINE OF METHOD Carbendazim is dissolved in dioxane-sulphuric acid and determined by HPLC on a reverse phase column (C_{18}) using a methanol-water-sulphuric acid mixture as mobile phase and external standardisation.

REAGENTS

Methanol
Dioxane
Sulphuric acid solution $c(\frac{1}{2} H_2SO_4) = 1$ mol/l
Mobile phase. Prepare a 35 % (v/v) methanol aqueous solution and add sulphuric acid 0.5% v/v).
Diluting solvent. Mix sulphuric acid solution (10 ml) and dioxane (90 ml).
Carbendazim standard of known purity (minimum purity at least 990 g/kg)
Calibration solution. Weigh (to the nearest 0.1 mg) about 200 mg (*s* mg) carbendazim standard into a volumetric flask (100 ml). Add diluting solvent (about 80 ml) and place the flask in an ultrasonic bath for 20 min. If an ultrasonic bath is not available, shake for at least 2 hours. Allow to cool to room temperature, fill to the mark with diluting solvent, and mix well. Transfer by pipette 10 ml of this solution to a volumetric flask (100 ml), fill to the mark with mobile phase and mix well.

APPARATUS

High performance liquid chromatograph equipped with a variable wavelength detector and a 20 µl loop injector
Column 300 × 3 (i.d.) mm, stainless steel, packed with Partisil 10 µm ODS or equivalent
Integrator/data system
Ultrasonic bath

PROCEDURE

(a) Operating conditions (typical):

Flow rate	1.5 ml/minute
Detector wavelength	282 nm
Injection volume	20 µl
Retention time	carbendazim: approximately 4 min

(b) Preparation of sample solution. Weigh (to the nearest 0.1 mg) sufficient sample to contain about 200 mg (*w* mg) carbendazim standard into a volumetric flask (100 ml). Add diluting solvent (about 80 ml) and place the flask in an ultrasonic bath for 20 min. If an ultrasonic bath is not available, shake for at least 2 hours. (NOTE: *Because of the low solubility and the slow rate of dissolution, intense mixing is critical).* Allow to cool to room temperature, fill to the mark with diluting solvent, and mix well. Transfer by pipette 10 ml of this solution to a volumetric flask (100 ml), fill to the mark with mobile phase and mix well.

(c) Determination. Inject into the liquid chromatograph 20 µl portions of the calibration solution until the peak areas of two successive injections differ by less than ± 2%. Then inject duplicate 20 µl portions of the sample solution, followed by another injection of the calibration solution. Average the two peak areas of the sample solutions and of the calibration solutions following and preceding the sample injections.

(d) Calculation

$$\text{Content of carbendazim} = \frac{H_w \times s \times P}{H_s \times w} \text{ g/kg}$$

where:

H_s = area of carbendazim peak in the calibration solution
H_w = area of carbendazim peak in the sample solution
s = mass of carbendazim in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of carbendazim reference substance (g/kg)

Repeatability r	=	15 g/kg at 993 g/kg active ingredient content
	=	15 g/kg at 898 g/kg active ingredient content
Reproducibility R	=	29 g/kg at 993 g/kg active ingredient content
	=	32 g/kg at 898 g/kg active ingredient content

CARBENDAZIM WETTABLE POWDERS

[*]**263**/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests

2.1 HPLC. As for carbendazim technical **263**/TC/M/2.1.

2.2 UV. As for carbendazim technical **263**/TC/M/2.2.

3 Carbendazim.

3.1 Ultraviolet spectroscopic method. As for carbendazim technical **263**/TC/M/3.1 except:

(b) Preparation of sample solution. Weigh (to the nearest 0.1 mg) into a volumetric flask (500 ml) enough sample to contain about 80 mg (*w* mg) carbendazim, add hydrochloric acid solution (about 250 ml, *c*(HCl) = 1 mol/l)

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and place the flask in an ultrasonic bath for at least 20 min. If no ultrasonic bath is available allow to stand the solution for at least 2 h with occasional shaking. Allow to cool to room temperature and make up to volume with hydrochloric acid solution. Allow to stand for 30 min in order to allow to settle any insoluble material. Transfer by pipette 25 ml of this solution to a volumetric flask (500 ml) and dilute to volume with hydrochloric acid solution. Filter a portion of the solution through a suitable microfilter.

Repeatability r	=	6 g/kg at 599 g/kg active ingredient content
	=	7 g/kg at 501 g/kg active ingredient content
Reproducibility R	=	29 g/kg at 599 g/kg active ingredient content
	=	21 g/kg at 501 g/kg active ingredient content

3.2 HPLC method. As for carbendazim technical **263**/TC/M/3.2 except:
(b) Preparation of sample solution. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) enough sample to contain about 200 mg (*w* mg) carbendazim.
Add diluting solvent (about 80 ml) and place the flask in an ultrasonic bath for 20 min. If an ultrasonic bath is not available, shake for at least 2 hours. Allow to cool to room temperature, fill to the mark with diluting solvent, and mix well. Allow to stand for 30 min in order to allow any insoluble material to settle. Transfer by pipette 10 ml of this solution to a volumetric flask (100 ml), fill to the mark with mobile phase and mix well. Filter a portion through a suitable micro-filter.

Repeatability r	=	11 g/kg at 588 g/kg active ingredient content
	=	9 g/kg at 494 g/kg active ingredient content
Reproducibility R	=	29 g/kg at 588 g/kg active ingredient content
	=	15 g/kg at 494 g/kg active ingredient content

CARBENDAZIM WATER DISPERSIBLE GRANULES
[*]**263**/WG/M/-

1 Sampling. Take at least 500 g.

2 Identity tests
2.1 HPLC. As for carbendazim technical **263**/TC/M/2.1.
2.2 UV. As for carbendazim technical **263**/TC/M/2.2.

3 Carbendazim. As for carbendazim wettable powders **263**/WP/M/3.

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