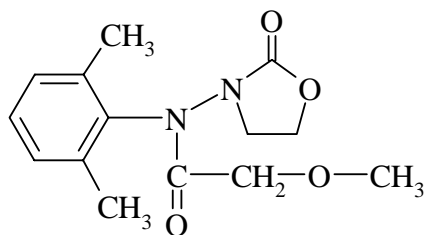


OXADIXYL
397



<i>ISO common name</i>	Oxadixyl
<i>Chemical name</i>	2-Methoxy- <i>N</i> -(2-oxo-1,3-oxazolidin-3-yl)acet- 2',6'-xylidide; <i>N</i> -(2,6-dimethylphenyl)-2- methoxy- <i>N</i> -(2-oxo-3-oxazolidinyl)acetamide (CA; 77732-09-3)
<i>Empirical formula</i>	C ₁₄ H ₁₈ N ₂ O ₄
<i>RMM</i>	278.3
<i>m.p.</i>	104 - 105 °C
<i>v.p.</i>	3.3 × 10 ⁻⁶ Pa at 20 °C
<i>Solubility</i>	In water: 3.4 g/l at 25 °C; soluble in common organic solvents; slightly soluble in diethyl ether and petroleum ether
<i>Description</i>	Colourless crystals
<i>Stability</i>	Stable in aqueous solutions at pH 5 - 9
<i>Formulations</i>	Wettable powders

OXADIXYL TECHNICAL

*397/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC Use the HPLC method below. The relative retention time of oxadixyl with respect to the internal standard for the sample solution should not deviate by more than 1.5 % from that of the calibration solution.

2.2 Infrared. Prepare potassium bromide discs from the sample and from an oxadixyl standard. Scan the discs from 400 to 4000 cm^{-1} . The spectrum from the sample should not differ significantly from that of the standard.

3 Oxadixyl

OUTLINE OF METHOD Oxadixyl is extracted with or dissolved in acetonitrile and determined by reverse phase high performance liquid chromatography with a UV-detector, using benzophenone as an internal standard.

REAGENTS

Oxadixyl of known purity

Benzophenone internal standard

Acetonitrile HPLC quality

Water HPLC quality

Internal standard solution. Dissolve benzophenone (140 ± 10 mg) in acetonitrile in a dispenser or volumetric flask (1 l), make up to volume and mix well.

Calibration solution. Weigh (to the nearest 0.1 mg) oxadixyl reference standard (130 ± 10 mg, s mg) into a volumetric flask (50 ml). Add by dispenser or pipette internal standard solution (50.0 ml), close the flask and mix well.

* CIPAC method 1995. Prepared by the Swiss Committee (PAC-CH). Chairman: H-P Bosshardt. Based on a method supplied by Sandoz AG, Switzerland.

APPARATUS

High performance liquid chromatograph equipped with a UV-detector, an automatic injector (10 μ l), an electronic integrator or data system and a column oven (35 °C)

Chromatographic column 250 \times 4 (i.d.) mm, Hibar LiChrosorb RP-Select B column (Merck), 5 μ m

Ultrasonic bath

PROCEDURE

(a) *Operating conditions* (typical):

<i>Mobile phase</i>	water - acetonitrile, 60 + 40 (v/v)
<i>Flow rate</i>	1.5 ml/min
<i>Temperature of column</i>	35 °C
<i>Detector wavelength</i>	267 nm
<i>Retention times</i>	oxadixyl: about 4.8 min internal standard: about 20.6 min
<i>Run time</i>	about 24 min

(b) *Preparation of sample*. Weigh (to the nearest 0.1 mg) oxadixyl technical (130 ± 10 mg, w mg) into a volumetric flask (50 ml). Add by dispenser or pipette internal standard solution (50.0 ml), close the flask and mix well.

(c) *Determination*. Inject 10 μ l aliquots of the calibration solution until the oxadixyl and to internal standard peak area ratios vary by less than 1%. Inject in duplicate 10 μ l aliquots of the calibration solution. Then inject each sample solution in duplicate. After each series of 3 samples make duplicate injections of the calibration solution. Repeat the sequence until all samples are analysed. Calculate the oxadixyl to internal standard peak ratio for each injection.

(d) Calculation

$$\text{Oxadixyl content} = \frac{R \times s \times P}{R' \times w} \text{ g/kg}$$

where:

R = peak area ratio of oxadixyl to the internal standard for the sample solution

R' = peak area ratio of oxadixyl to the internal standard for the calibration solution

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

w = mass of sample taken (mg)

P = purity of the standard oxadixyl (g/kg)

Repeatability r = 3.2 g/kg at 986 g/kg active ingredient content

Reproducibility R = 3.7 g/kg at 986 g/kg active ingredient content

OXADIXYL WETTABLE POWDERS

*397/WP/M/-

1 Sampling. Take at least 500 g.

2 Identity tests

2.1 HPLC. As for oxadixyl technical 397/TC/M/2.1.

2.2 Infrared. Prepare a potassium bromide disc from the sample and from an oxadixyl standard. Scan the discs from 400 to 4000 cm^{-1} . The spectrum from the sample should show the characteristic bands for oxadixyl in the 1800 to 1690 cm^{-1} region.

3 Oxadixyl

SCOPE The method is also applicable to oxadixyl in mixtures with copper oxychloride and mancozeb.

Proceed as for oxadixyl technical 397/TC/M/3 except:

(b) Sample preparation. Weigh (to the nearest 0.1 mg) enough sample to contain 130 ± 10 mg oxadixyl (w mg) into an stoppered Erlenmeyer flask (100 ml). Add by dispenser or pipette internal standard solution

* CIPAC method 1995. Prepared by the Swiss Committee (PAC-CH). Chairman: H-P Bosshardt. Based on a method supplied by Sandoz AG, Switzerland.

(50.0 ml), shake and place the flask in an ultrasonic bath for 10 minutes. Centrifuge for 10 minutes to obtain a clear solution.

For oxadixyl/mancozeb combinations add at:

(a) Operating conditions

Initially set the ratio of water/acetonitrile in the mobile phase at 60 + 40 (v/v). Then about 23 minutes after injection change the water/acetonitrile ratio of the mobile phase to 5 + 95 (v/v), within one minute, and hold for ten minutes. Return to the initial conditions, wait for twelve minutes (for equilibration) and then start the next analysis.

Repeatability r	= 1.2 g/kg at 266 g/kg active ingredient content
	= 0.7 g/kg at 104 g/kg active ingredient content (+ copper)
	= 0.8 g/kg at 100 g/kg active ingredient content (+ mancozeb)
	= 0.9 g/kg at 83 g/kg active ingredient content (+ mancozeb)
	Reproducibility R
= 2.9 g/kg at 266 g/kg active ingredient content	
= 0.9 g/kg at 104 g/kg active ingredient content (+ copper)	
= 2.5 g/kg at 100 g/kg active ingredient content (+ mancozeb)	
= 1.8 g/kg at 83 g/kg active ingredient content (+ mancozeb)	

4 Suspending (Draft method)

(a) Preparation of suspension. MT 15.1 (i).

(b) Determination of sedimentation. MT 15.1 (ii).

(c) Determination of active ingredient(s) in the bottom 25 ml of suspension. After removal of the top 225 ml of suspension, transfer the bottom 25.0 ml of suspension quantitatively to a tared evaporating dish. Evaporate nearly to dryness on a boiling water bath. Dry in an oven at 100 °C for 5 min, allow to cool in a desiccator and reweigh.

(d) *Calculation*

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

where:

c = mass of active ingredient(s) in the sample taken for the preparation of the suspension (g)

Q = mass of active ingredient(s) in the bottom 25 ml of suspension (g)