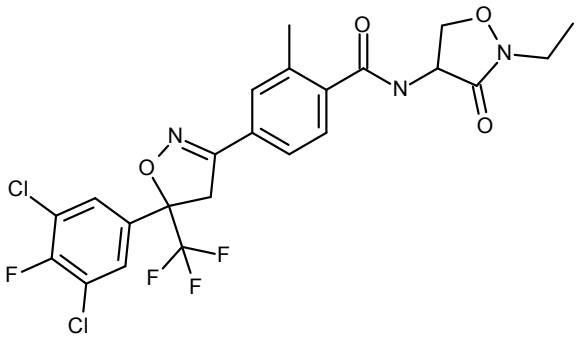
ISOCYCLOSERAM

XXX



ISO Common Name	Isocycloseram
Chemical Name	4-(5-(3,5-dichloro-4-fluorophenyl)-5-(trifluoromethyl)-4,5-dihydro-1,2-oxazol-3-yl)-N-(2-ethyl-3-oxo-1,2-oxazolidin-4-yl)-2-methylbenzamide
Empirical formula	$C_{23}H_{19}Cl_2F_4N_3O_4$
RMM	548.3
m.p.	138.9°C (412.0 K) AT 100.1 to 103.0 kPa
CAS Number	2061933-85-3

ISOCYCLOSERAM TECHNICAL
*XXX/TC/M/-

1 Sampling. Take at least 5 g.

2 Identity tests

2.1 Infrared. Prepare potassium bromide pellets of the sample and of pure isocycloseram and scan from 4000 to 600 cm^{-1}. The spectrum obtained from the sample should not differ significantly from that of the reference grade material. (Fig 1)

2.2 HPLC. Use the HPLC method below. The relative retention time of isocycloseram in the sample solution should not deviate by more than 2% from that of the calibration solution. The UV spectrum measured from this peak should match that obtained from the calibration substance.
(Fig 2)

3 Isocycloseram

OUTLINE OF METHOD

Isocycloseram is determined by reversed phase high performance liquid chromatography using UV detection at 265 nm and external standardization.

REAGENTS

Isocycloseram reference standard with known content.

Acetonitrile HPLC grade.

Deionized water.

Formic acid.

* Provisional CIPAC method 2023. Based on a method supplied by Syngenta Crop Protection AG, Switzerland

Calibration solution. Weigh in duplicate (to the nearest 0.1 mg) 45 – 55 mg (*s* mg) of isocycloseram reference standard into separate 100 ml volumetric flasks. Dissolve with 60 ml acetonitrile and treat for approx. 5 minutes in an ultrasonic bath or until completely dissolved. Cool down to ambient temperature and make up to volume with acetonitrile. Dilute 10.0 ml of this solution into a 50 ml volumetric flask and make up to volume with acetonitrile at ambient temperature. If necessary, clarify a portion of the solution using a PTFE 0.45 µm filter (solutions C_A and C_B).

APPARATUS

High performance liquid chromatograph equipped with an ultraviolet spectrophotometric detector and an injecting system capable of injecting 5 µl.

Column stainless steel, 100 x 4.6 mm (i.d.), packed with Kinetex C18, 2.6 µm, or equivalent material with the same selectivity.

Ultrasonic bath

Dispensable PTFE filters, solvent compatible, 25 mm, porosity 0.45 µm.

Electronic integrator or *data system*

PROCEDURE

(a) Liquid chromatographic conditions (typical):

Column	stainless steel, 100 x 4.6 mm (i.d.), packed with Kinetex C18, 2.6 µm, or equivalent material with the same selectivity
Column temperature	40 °C
Flow rate	1.0 ml/min
Detector wavelength	265 nm
Injection volume	5 µl
Run time	10 min
Retention time	approximately 5.3 min

Gradient

time (min)	% acetonitrile	% 0.1% v/v formic acid in water
0	60	40
4	60	40
4.1	95	5
6.9	95	5

7	60	40
10	60	40

(b) System equilibration. Prepare two calibration solutions. Inject 5 µl portions of solution C_A until the response factors obtained for two consecutive injections differ by less than 1 %. Then inject a 5 µl portion of the solution C_B. The response factor for this solution should not deviate by more than 5 % from that of solution C_A, otherwise prepare new calibration solutions.

(c) Sample preparation. Prepare solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) 45 – 55 mg (w mg) of the sample into a 100 ml volumetric flask. Dissolve with 60 ml acetonitrile and treat for approx. 5 minutes in an ultrasonic bath or until completely dissolved. Cool down to ambient temperature and make up to volume with acetonitrile. Dilute 10.0 ml of this solution into a 50 ml volumetric flask and make up to volume with acetonitrile at ambient temperature. If necessary, clarify a portion of the solution using a PTFE 0.45 µm filter (solutions S_A and S_B).

(d) Determination. If not otherwise requested inject in duplicate 5 µl portions of each sample solution, bracketing them with duplicate injections of the calibration solution as follows: calibration solution C_A, calibration solution C_B, calibration solution C_A, sample solution $S1_A$, sample solution $S1_B$, calibration solution C_A, sample solution $S2_A$, sample solution $S2_B$, calibration solution C_A, and so on for further samples. Measure the relevant peak areas.

(e) Calculation. Calculate the mean value of each pair of calibration response factors, bracketing the two injections of a sample, and use this value for calculating the isocycloseram contents of the bracketed sample injections.

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

$$\text{Content of isocycloseram} = \frac{f \times H_w}{w} \text{ g/kg}$$

where:

f = mean response factor

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

Repeatability r =
Reproducibility R =

ISOCYCLOSERAM WETTABLE POWDERS
*XXX/WP/M/-

1 Sampling. Take at least 20 g.

2 Identity tests

2.1 Infrared. As for technical XXX/TC/M/2.1.

2.2 HPLC. As for technical XXX/TC/M/2.2 and Fig 3.

3 Isocycloseram. As for isocycloseram technical XXX/TC/M/3, except:

Change (c) Sample preparation to:

(c) Sample preparation. Prepare solutions in duplicate for each sample. Homogenize the test sample thoroughly. Weigh (to the nearest 0.1 mg) sufficient sample to contain 45 – 55 mg (w mg) of isocycloseram (equal to 150 – 180 mg of isocycloseram formulation WP (15)) into a 50 ml volumetric flask. Dissolve with 35 ml acetonitrile and treat for approx. 5 minutes in an ultrasonic bath or until completely dissolved. Cool down to ambient temperature and make up to volume with acetonitrile. Dilute 10.0 ml of this solution into a 50 ml volumetric flask and make up to volume with acetonitrile at ambient temperature. Clarify a portion of the solution using a PTFE 0.45 µm filter, discarding the first 1 ml (sample solutions S_A and S_B).

Change (e) Calculation to

(e) Calculation. Calculate the mean value of each pair of calibration response factors, bracketing the two injections of a sample, and use this value for calculating the isocycloseram contents of the bracketed sample injections.

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

$$\text{Content of isocycloseram} = \frac{f \times H_w}{w*2} \text{ g/kg}$$

* Provisional CIPAC method 2023. Based on a method supplied by Syngenta Crop Protection AG, Switzerland

where:

f = mean response factor

H_s = peak area of isocycloseram in the calibration solution

H_w = peak area of isocycloseram in the sample solution

s = mass of isocycloseram reference standard in the calibration solution (mg)

w = mass of sample taken (mg)

P = purity of isocycloseram reference standard (g/kg)

Repeatability r =

Reproducibility R =

4 Suspensibility

REAGENTS AND APPARATUS As for XXX/TC/M/x and MT 184.

PROCEDURE

(a) Preparation of suspension and determination of sedimentation. MT 184

(b) Determination of xxxxx. Prepare solutions in duplicate for each sample. XXXXX

Repeatability r =

Reproducibility R =

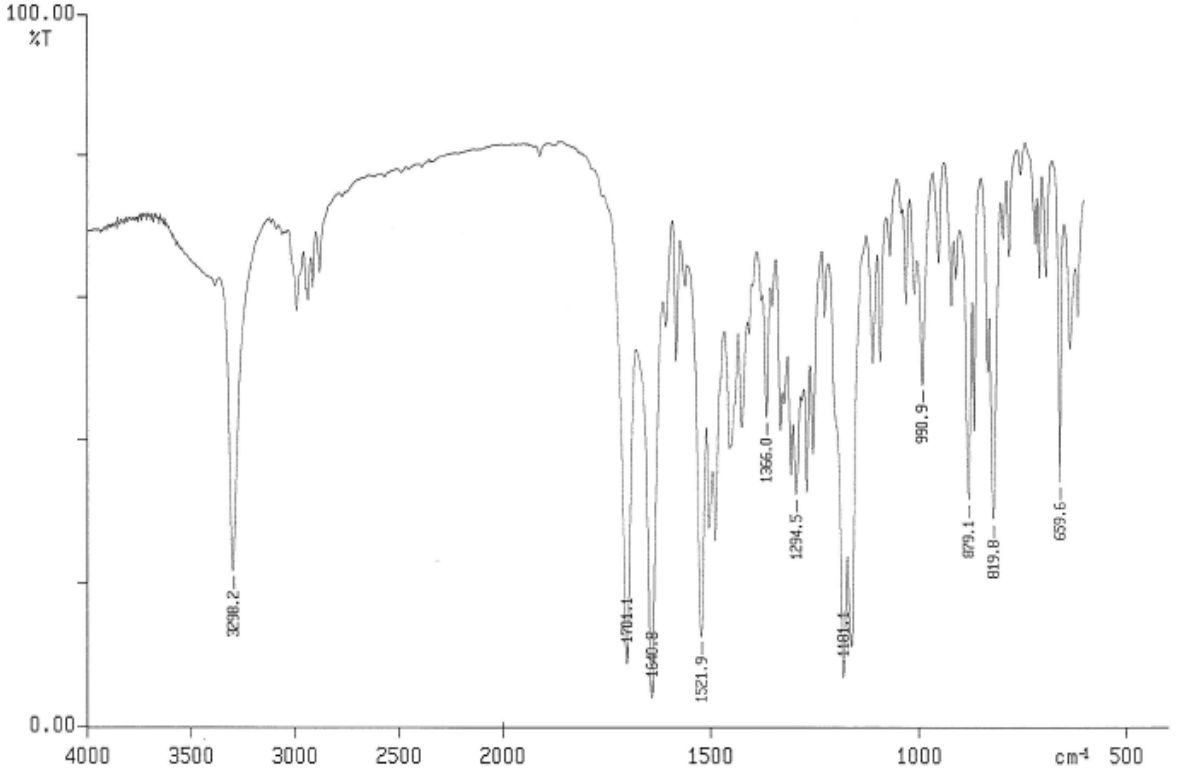


Fig 1 Typical IR spectrum according to xxx/TC/M/2.1

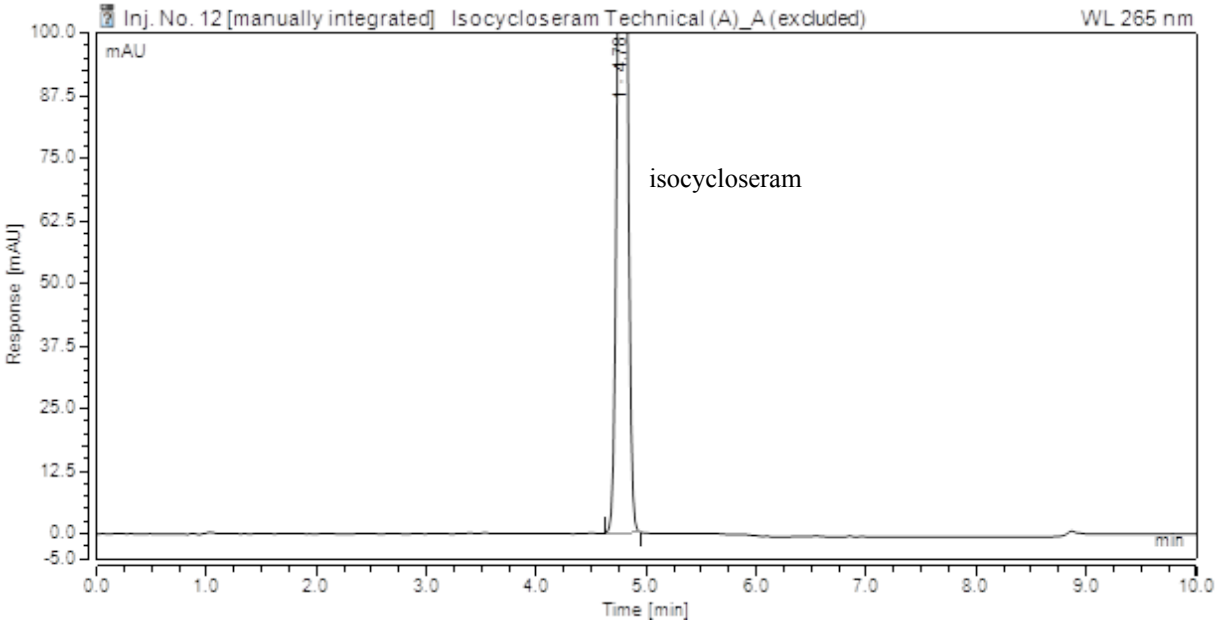


Fig 2 Typical chromatogram of Isocycloseram TC according to xxx/TC/M/2.2

Fig 4 Typical IR spectrum according to xxx/WP/M/2.1

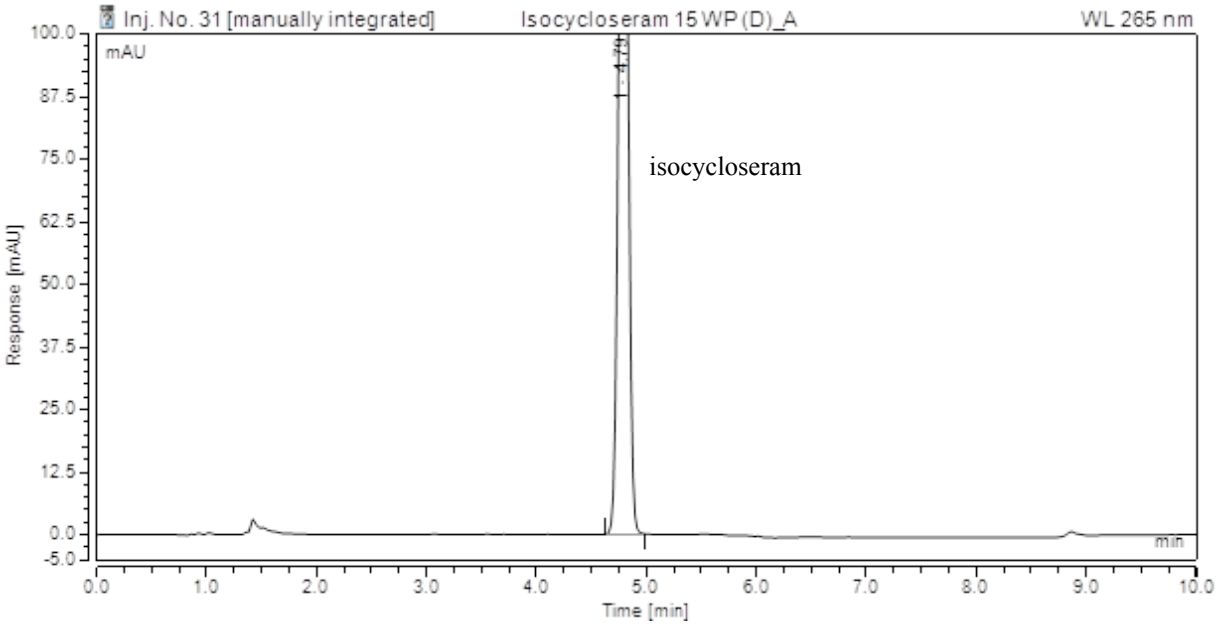


Fig 3 Typical chromatogram of Isocycloseram WP according to xxx/WP/M/2.2