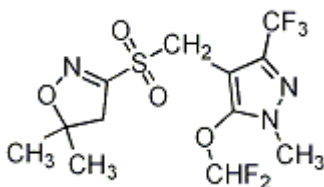


Pyroxasulfone

817



ISO common name Pyroxasulfone

Chemical name 3-[5-(difluoromethoxy)-1-methyl-3-(trifluoromethyl)pyrazol-4-ylmethylsulfonyl]-4,5-dihydro-5,5-dimethyl-1,2-oxazole (IUPAC);
3-[[[5-(difluoromethoxy)-1-methyl-3-(trifluoromethyl)-1H-pyrazol-4-yl]methyl]sulfonyl]-4,5-dihydro-5,5-dimethylisoxazole (CA: 447399-55-5)

Empirical formula C₁₂H₁₄F₅N₃O₄S

RMM 391.3

Description White Crystal

Formulations Suspension concentrates (SC), water dispersible granules (WG)

PYROXASULFONE TECHNICAL

*817/TC/(M)/-

1. Sampling. Take at least 100 g.

2. Identity tests

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

2.2 Infrared. Prepare potassium bromide discs from the sample and pyroxasulfone

standard. Scan the discs from 4000 to 400 cm^{-1} . The spectrum from the sample should not differ significantly from that of the standard.

3. Pyroxasulfone

OUTLINE OF METHOD Pyroxasulfone is determined by reversed phase high-performance liquid chromatography using UV detection at 225 nm and external standardization.

REAGENTS

Pyroxasulfone standard of known purity

Acetonitrile, HPLC grade

Water HPLC grade

Mobile phase Acetonitrile – water, 45+55 (v/v)

Calibration solutions. Weigh in duplicate (to the nearest 0.1 mg) into a volumetric flask (25 ml) about 25 mg of Pyroxasulfone standard (s mg). Add acetonitrile (about 20 ml) into the flask, place the flask in an ultrasonic bath for 2 min. Allow to cool to ambient temperature. Dilute to volume with acetonitrile (Solutions C_A and C_B). Mix thoroughly.

APPARATUS

High performance liquid chromatograph equipped with a detector suitable for operation at 225nm, constant-temperature column compartment and an injector capable of delivering 10 μ l.

Column XBridge® C18, 150 \times 4.6 (i.d.) mm, 5 μ m particle size, or equivalent.

Electronic integrator or data system

Ultrasonic bath

PROCEDURE

(a) *Liquid chromatograph conditions* (typical):

Column XBridge® C18, 150 \times 4.6 (i.d.) mm, 5 μ m particle size, or equivalent.

Mobile phase Acetonitrile – water, 45+55 (v/v)

Column temperature 35°C

Injection volume 10 μ l

Flow rate 1.0 ml/min

Detector wavelength 225 nm

Run time 20 min

Retention time Pyroxasulfone: about 9.3 min

(b) *Linearity check.* Check the linearity of the detector response by injecting 10 μ l of

solutions with pyroxasulfone concentrations 0.5, 1 and 2 times that of the calibration solution before conducting analysis.

(c) *System equilibration.* Prepare two calibration solutions. Inject 10 µl portions of the first one until the response factors obtained for two consecutive injections differ by less than 1.0%. Then inject a 10 µl portion of the second solution. The response factor for this solution should not deviate by more than 1.0% from that for the first calibration solution, otherwise prepare new calibration solutions.

(d) *Preparation of sample solution.* Prepare sample solutions in duplicate for each sample. Weigh in duplicate (to the nearest 0.1 mg) into a volumetric flask (25 ml) sufficient sample to contain approximately 25 mg of Pyroxasulfone (w mg). Add acetonitrile (about 20 ml) into the flask, place the flask in an ultrasonic bath for 2 min. Allow to cool to ambient temperature. Dilute to volume with acetonitrile (Solutions S_A and S_B). Mix thoroughly. Filter aliquot of each solution through a 0.45 µm PTPE filter prior to analysis.

(e) *Determination.* Inject in duplicate 10µl portions of each sample solution bracketing them by injections of the calibration solutions as follows: C_{A1} , S_{A1} , S_{A2} , C_{B1} , S_{B1} , S_{B2} , C_{A2} , and so on. Measure the relevant peak areas.

(f) *Calculation.* Calculate the mean value of each pair of response factors bracketing the two injections of a sample and use this value for calculating the pyroxasulfone contents of the bracketed sample injections. The pyroxasulfone content is the mean value of two sample solutions.

$$f_i = \frac{s \times P}{H_S}$$

$$\text{Pyroxasulfone content} = \frac{s \times H_w}{w} \quad \text{g/kg}$$

where:

f_i = individual response factor

f = mean response factor

H_S = peak area of pyroxasulfone in the calibration solution

H_w = peak area of pyroxasulfone in the sample solution

s = mass of pyroxasulfone standard taken (mg)

w = mass of sample taken (mg)

P = purity of pyroxasulfone standard (g/kg)

PYROXASULFONE SUSPENSION CONCENTRATES

*817/SC/(M)/-

1. Sampling. Take at least 100 ml.

2. Identity tests.

2.1 HPLC. As for Pyroxasulfone technical 817/TC/(M)/2.1

2.2 Infrared. As for Pyroxasulfone technical 817/TC/(M)/2.2

3. Pyroxasulfone. As for Pyroxasulfone technical 817/TC/M/3.

4. Suspensibility (draft method)

REAGENTS AND APPARATUS as for 817/TC/(M)/3 and MT 184.1.

PROCEDURE

(a) *Preparation of suspension and determination of sedimentation.* MT 184.1

(b) *Determination of pyroxasulfone in the bottom 25 ml of suspension.* After removal of the top 225 ml of suspension add 25 ml of acetonitrile to the remaining 25 ml. Place the cylinder in an ultrasonic bath for 5 min. Allow to cool to room temperature, mix well by inverting the cylinder several times. Take a suitable aliquot of the solution and determine the mass of pyroxasulfone (Q g) by 817/TC/(M)/3.

(c) *Calculation*

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

Where:

c = mass of pyroxasulfone in the sample taken for the preparation of the suspension (g)

Q = mass of pyroxasulfone in the bottom 25 ml of suspension (g)

PYROXASULFONE WATER DISPERSIBLE GRANULES

*817/WG/(M)/-

1. Sampling. Take at least 100 g.

2. Identity tests.

2.1 HPLC. As for Pyroxasulfone technical 817/TC/(M)/2.1

2.2 Infrared. As for Pyroxasulfone technical 817/TC/(M)/2.2

3. Pyroxasulfone As for Pyroxasulfone technical 817/TC/M/3.

4. Suspensibility (draft method)

REAGENTS AND APPARATUS as for 817/TC/(M)/3 and MT 184.1.

PROCEDURE

(a) Preparation of suspension and determination of sedimentation. MT 184.1

(b) Determination of pyroxasulfone in the bottom 25 ml of suspension. After removal of the top 225 ml of suspension add 25 ml of acetonitrile to the remaining 25 ml. Place the cylinder in an ultrasonic bath for 5 min. Allow to cool to room temperature, mix well by inverting the cylinder several times. Take a suitable aliquot of the solution and determine the mass of pyroxasulfone (Q g) by 817/TC/(M)/3.

(c) Calculation

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

Where:

c = mass of pyroxasulfone in the sample taken for the preparation of the suspension (g)

Q = mass of pyroxasulfone in the bottom 25 ml of suspension (g)

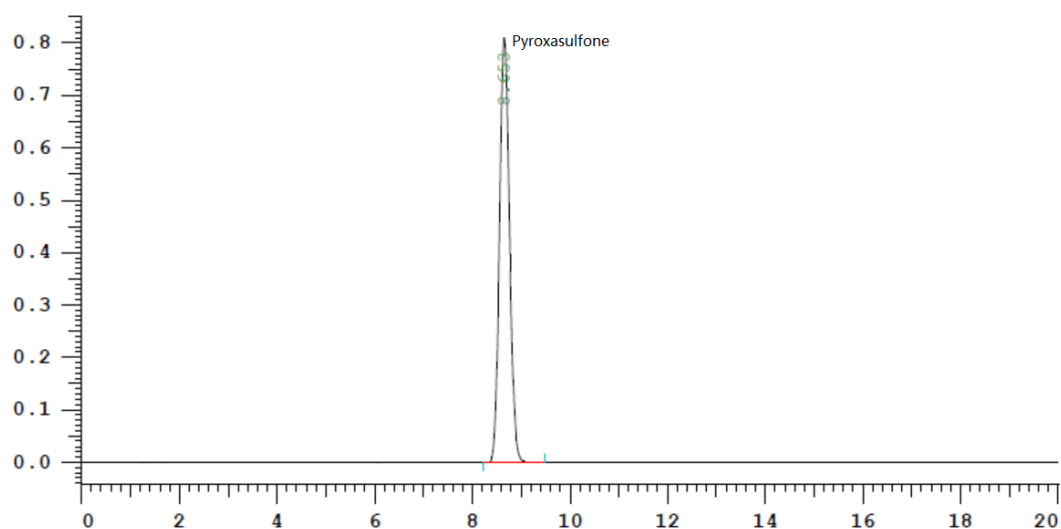


Fig.1 HPLC chromatogram of Pyroxasulfone standard

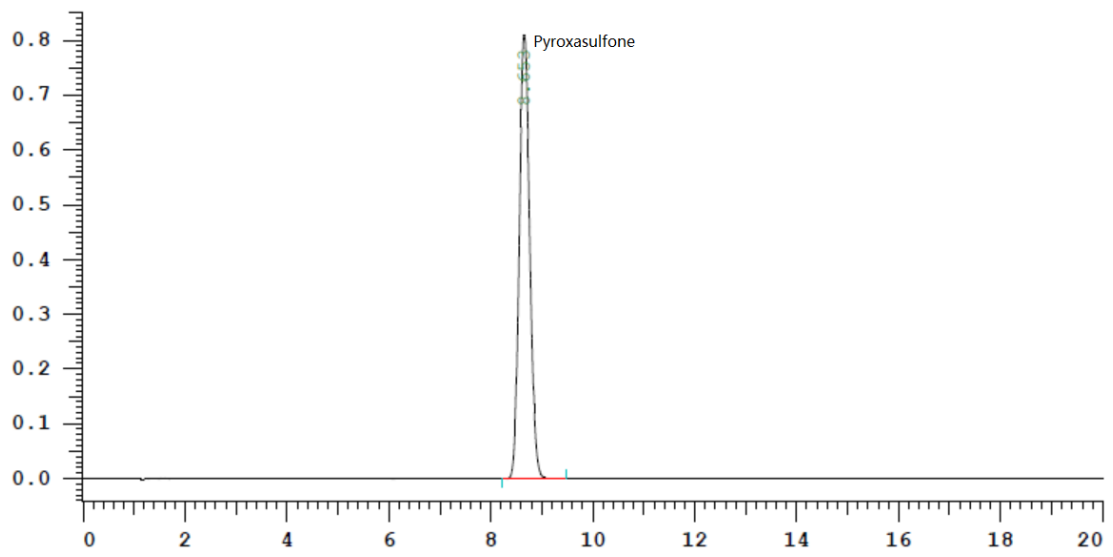


Fig.2 HPLC chromatogram of Pyroxasulfone TC

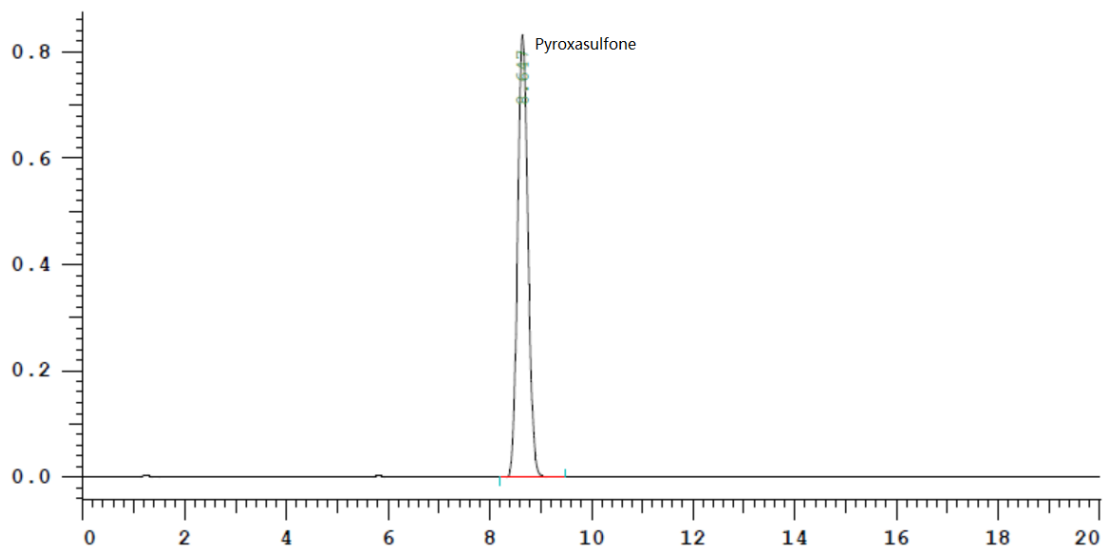


Fig.3 HPLC chromatogram of Pyroxasulfone SC

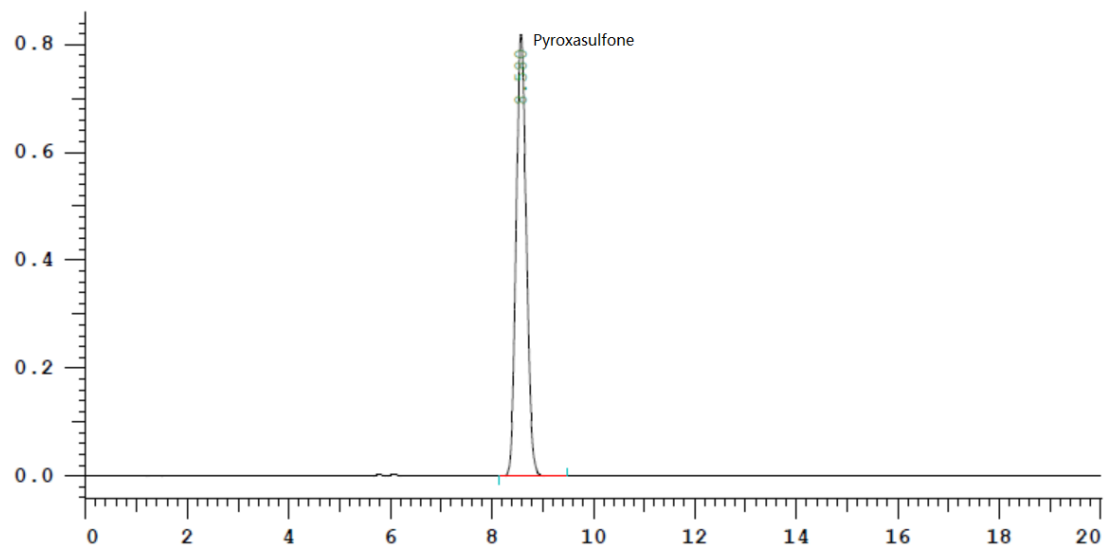


Fig.4 HPLC chromatogram of Pyroxasulfone WG