595. Flazasulfuron

HPLC method

CIPAC Full Scale Collaborative Trial

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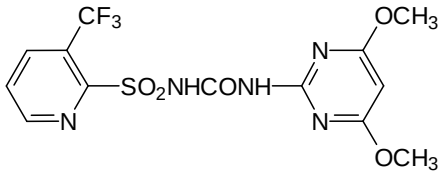
FLAZASULFURON
595



ISO common name:	Flazasulfuron
Chemical name:	1-(4,6-dimethoxypyrimidin-2-yl)-3-(3-trifluoromethyl-2-pyridylsulfonyl)urea (IUPAC)
CAS-Number:	104040-78-0
RMM:	407.36
Empirical formula:	$C_{13}H_{12}F_3N_5O_5S$
m.p.	180 °C
V.p.	< 1.33 x 10^{-5} Pa at 25°C, 35°C, 45 °C
Solubility	In water at 25 °C: 2.1 g/l at pH 7, 0.027 g/l at pH 5 and not stable at pH 9 In n-hexane : 0.5 mg/l at 25°C In toluene: 0.56 g/l at 25°C In dichloromethane: 22.1 g/l at 25°C In methanol: 4.2 g/l at 25°C In acetone: 22.7 g/l at 25°C In ethyl acetate: 6.9 g/l at 25°C In n-octanol: 0.20 g/l at 25°C In acetonitrile: 8.7 g/l at 25°C
Stability	Keep frozen (< -18 °C) when not in use and avoid exposure to light.
Hydrolysis	DT_{50} at 22 °C: 16.6 days (pH 7), 13.1 days (pH 9) and 17.4 hours (pH 4)
Description	Form : White powder
Formulation	Water dispersible granules

FLAZASULFURON TECHNICAL
595/TC/M/-

1 Sampling. Take at least 20 g.

2 Identity test

2.1 HPLC. Use the reversed phase HPLC method 3 described below. The relative retention time of the flazasulfuron peak 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.

3 Flazasulfuron

OUTLINE OF METHOD. Flazasulfuron is determined by reversed phase high performance liquid chromatography using UV detection at 260 nm and external standard calibration.

REAGENTS

Flazasulfuron reference standard of known content
Water HPLC grade
Acetonitrile HPLC grade
Acetic acid, 99.5%, analytical reagent grade
Mobile phase: water (0.05% acetic acid):acetonitrile (45:55, v/v)
Solvent: acetonitrile
Calibration solutions. Weigh in duplicate (to the nearest 0.1 mg) 40 mg of flazasulfuron reference standard (*s* mg) into separate volumetric flasks (100 ml). Add acetonitrile (about 75 ml) and sonicate for 5 minutes until complete dissolution. Allow the solutions to cool to ambient temperature and fill to the mark with acetonitrile (calibration solutions C_A and C_B). Mix well.

APPARATUS

High performance liquid chromatograph equipped with a detector suitable for operation at 260 nm (UV-detection) and an injection system capable of injecting 10 µl
Liquid chromatographic column stainless steel, 250 x 4.6 mm (i.d), packed with C_{18}, 5 µm, Agilent Zorbax Eclipse XDB or equivalent with the same selectivity
Electronic integrator or data system
Ultrasonic bath

PROCEDURE

(a) Chromatographic conditions (typical)

Column temperature	40°C
Flow rate	1.0 ml/min
Detector wavelength	260 nm
Injection volume	10 µl
Retention time	approximately 6 min
Mobile phase	water (0.05% acetic acid):acetonitrile (45:55, v/v)
Elution	isocratic

(b) Equilibration of the system. Pump sufficient mobile phase through the column to equilibrate the system. Inject 10 µl portions of the calibration solution C_A and repeat the injections until retention times and peak areas deviate by less than ± 0.5 % from the mean for three successive injections.

(c) Sample preparation. Prepare sample solutions in duplicate for each sample. Weigh (to the nearest 0.1 mg) sufficient sample (*w* mg) to contain about 40 mg flazasulfuron into a volumetric flask (100 ml). Add acetonitrile (about 75 ml) and sonicate for 5 minutes until complete dissolution. Allow the solutions to cool to ambient temperature and fill to the mark with acetonitrile (sample solutions S_1 and S_2). Mix thoroughly. Filter an aliquot of each prepared solution through a 0.45 µm PTFE filter prior to analysis.
Note: Analyse all samples within 8 hours of preparation.

(d) Determination. Inject 10 µl portions of the second calibration solution (C_B) for two successive injections. The mean response factor for this solution should deviate by no more than 1% from that for the first calibration solution (C_A) (see paragraph (b) Equilibration of the system), otherwise the calibration solutions should be prepared again.

Inject in duplicate 10 µl portions of each sample solution (S_1, S_2, …,etc.) bracketing them by single injections of calibration solutions (C_A and C_B) using the following sequence:

$C_A, S_1, S_1, S_2, S_2, C_B, S_3, S_3, S_4, S_4, C_A$…

(e) Calculation. Determine the peak area of flazasulfuron and calculate the mean value of response factors from the calibration solutions bracketing the injections of the sample solutions and use this value for calculating the flazasulfuron content of the bracketed sample solutions. The flazasulfuron content is the mean value of two sample solutions.

$$f_i = \frac{s \times \mathrm{P}}{H_s}$$

$$\text{flazasulfuron content} = \frac{f \times H_w}{w} \quad \text{[g/kg] } (M)$$

where:

f_i = individual response factor
f = mean response factor
H_s = peak area of flazasulfuron in the calibration solution
H_w = peak area of flazasulfuron in the sample solution
s = mass of flazasulfuron reference standard in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of flazasulfuron reference standard (g/kg)

FLAZASULFURON WATER DISPERSIBLE GRANULES

595/WG/M/-

1 Sampling. Take at least 100 g.

2 Identity test

2.1 HPLC. As for flazasulfuron technical 595/TC/M/2.1

3 Flazasulfuron

As for flazasulfuron technical 595/TC/M/3 except

(c) Sample preparation. Prepare sample solutions in duplicate for each sample. Grind the sample in a mortar in order to obtain a fine powder. Weigh (to the nearest 0.1 mg) sufficient sample (*w* mg) to contain about 40 mg flazasulfuron into a volumetric flask (100 ml). Add acetonitrile (about 75 ml) and sonicate for at least 15 minutes until complete dissolution. Allow the solutions to cool to ambient temperature and fill to the mark with acetonitrile (sample solutions S_1 and S_2). Mix thoroughly. Filter an aliquot of each prepared solution portion of each prepared solution through a 0.45 µm PTFE filter prior to analysis.
Note: Analyse all samples within 8 hours of preparation.

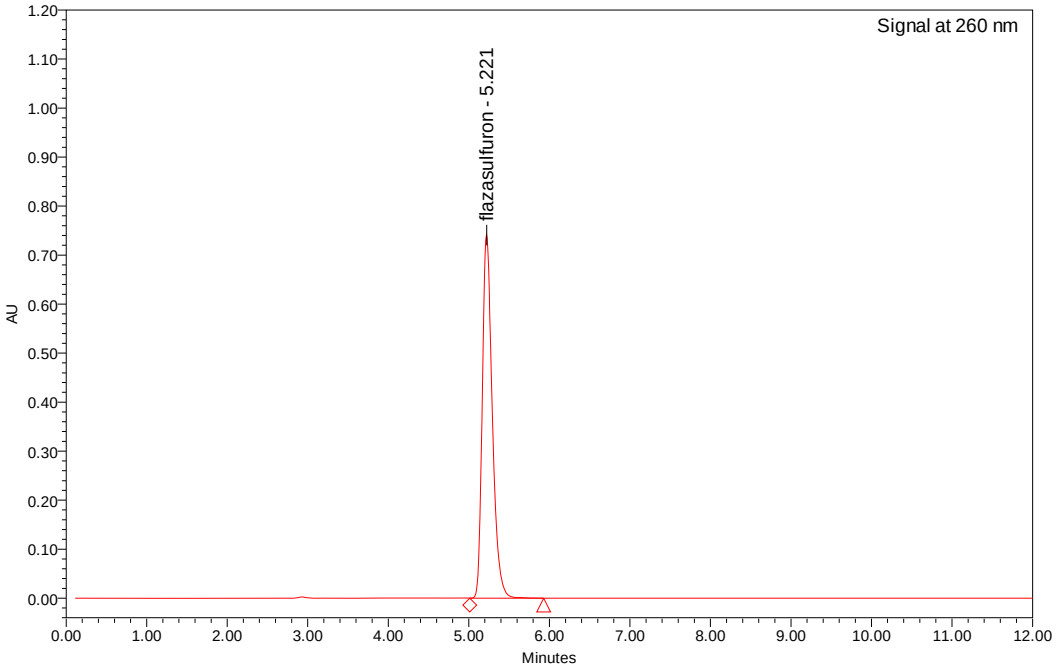


Fig 1 Typical HPLC-chromatogram of calibration solution

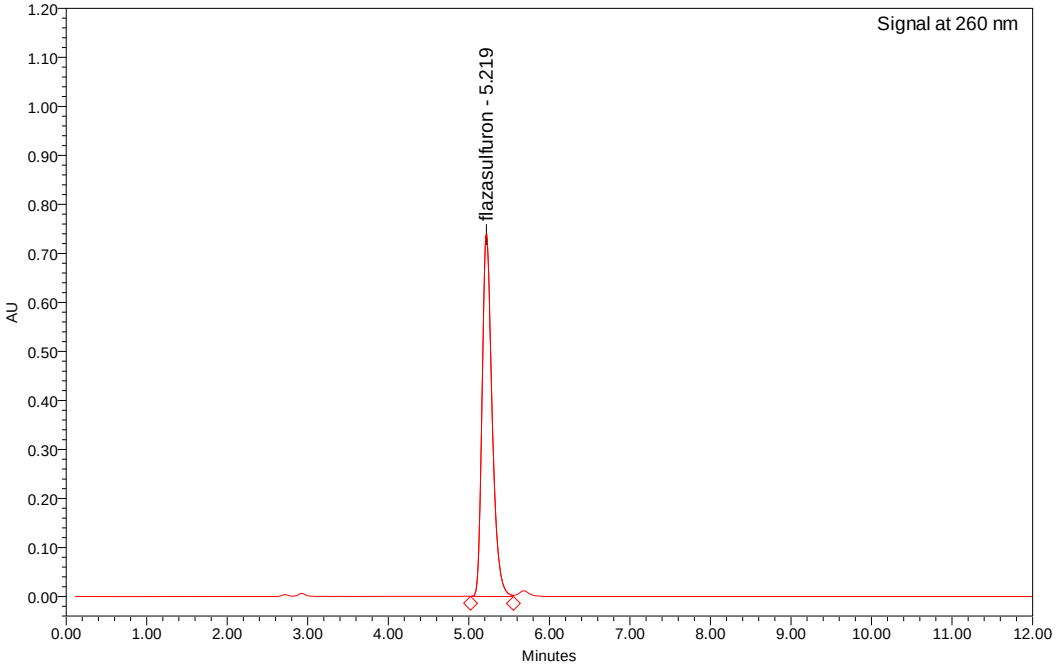


Fig 2 Typical HPLC-chromatogram of Flazasulfuron technical material

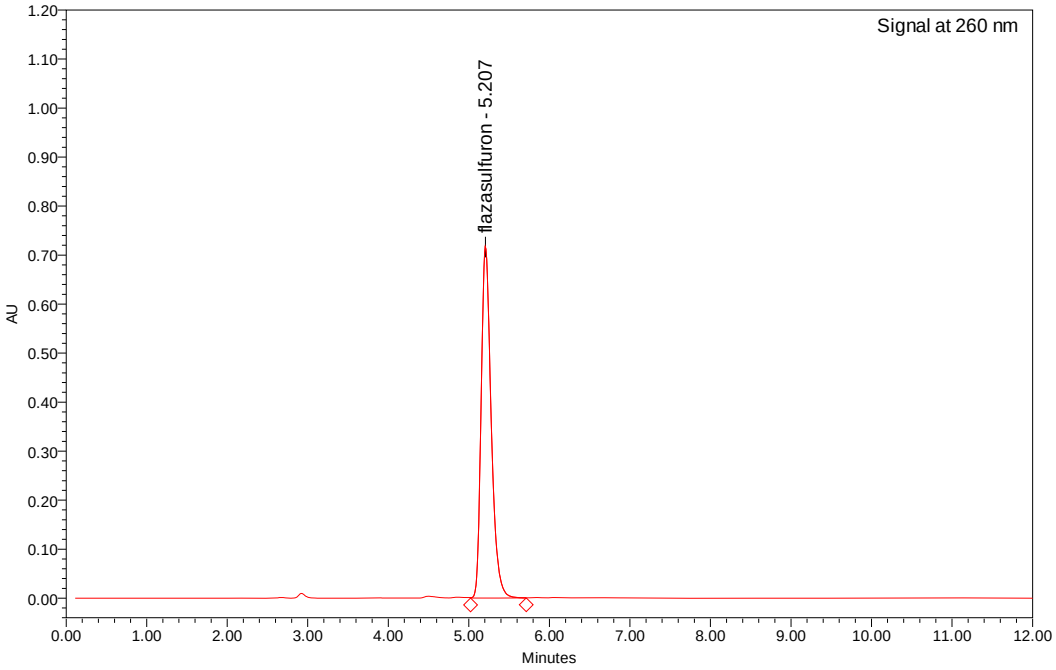


Fig 3 Typical HPLC-chromatogram of Flazasulfuron 25 WG