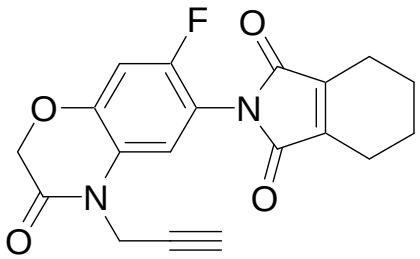
FLUMIOXAZIN

578



ISO common name	Flumioxazin
Chemical name	*N*-(7-fluoro-3,4-dihydro-3-oxo-4-prop-2-ynyl-2*H*-1,4-benzoxazin-6-yl)cyclohex-1-ene-1,2-dicarboximide (IUPAC); 2-[7-fluoro-3,4-dihydro-3-oxo-4-(2-propynyl)-2*H*-1,4-benzoxazin-6-yl]-4,5,6,7-tetrahydro-1*H*-isoindole-1,3(2*H*)-dione (CA)
CAS No.	103361-09-7
Empirical formula	$C_{19}H_{15}FN_2O_4$
RMM	354.3
v.p.	3.2 x 10^{-3} Pa (22°C)
Solubility	In water, 1.79 mg/l; acetone, 17 g/l; acetonitrile, 32.3 g/l; methanol, 1.6 g/l; ethyl acetate, 17.8 g/l; dichloromethane, 191 g/l; *n*-hexane, 1.6 g/l
Description	White to yellowish brown crystalline powder

FLUMIOXAZIN

578/TC/m/-

1 Sampling. Take at least 100 g.

2 Identity tests

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

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

3 Flumioxazin

OUTLINE OF METHOD Flumioxazin is determined by reversed phase high-performance liquid chromatography using UV detection at 288 nm and external standardisation.

REAGENTS

Acetonitrile HPLC grade

Water HPLC grade

Flumioxazin standard of known purity. Store refrigerated.

Mobile phase acetonitrile - water, 50 + 50 (v/v)

Calibration solution. Weigh in duplicate (to the nearest 0.1 mg) approximately 50 mg (*s* mg) of flumioxazin standard into separate volumetric flasks (100 ml). Add acetonitrile (about 80 ml) and place the flasks in an ultrasonic bath for 10 min. Allow to cool to room temperature, dilute to volume with acetonitrile. Mix thoroughly (Solutions C_A and C_B).

APPARATUS

High performance liquid chromatograph equipped with a detector suitable for operation at 288 nm, constant-temperature column compartment and an injector capable of delivering 10 µl.

Column 250 × 4.6 mm (i.d.), stainless steel, packed with Phenomenex Gemini 5µ C18 (5 µm), or equivalent.

Electric integrator or data system

Ultrasonic bath

PROCEDURE

(a) Liquid chromatographic conditions (typical):

Column	stainless steel, 250 x 4.6 (i.d.) mm, packed with Phenomenex Gemini 5µ C18 (5 µm), or equivalent.
Mobile phase	acetonitrile - water, 50 + 50 (v/v)
Temperature	40°C
Flow rate	1.0 ml/min
Detector wavelength	288 nm
Injection volume	10 µl
Retention time	flumioxazin: about 11 min

(b) Linearity check. Check the linearity of the detector response by injecting 10 µl of solutions with flumioxazin 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. Weigh in duplicate (to the nearest 0.1 mg) sufficient sample to contain about 50 mg (*w* mg) of flumioxazin into separate volumetric flasks (100 ml). Add acetonitrile (about 80 ml) and place the flasks in an ultrasonic bath for 10 min. Allow to cool to room temperature, dilute to volume with acetonitrile. Mix thoroughly (Solutions S_A and S_B).

(e) Determination. Inject in duplicate 10 µl portions of each sample solution bracketing them by injections of the calibration solutions as follows; calibration solution C_A, sample solution S_A, sample solution S_A, calibration solution C_B, sample solution S_B, sample solution S_B, calibration solution C_A, 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 flumioxazin contents of the bracketed sample injections.

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

$$\text{Flumioxazin content} = \frac{f \times H_w}{w} \text{ (g/kg)}$$

where:

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

Repeatability r = 5.5 g/kg at 994 g/kg active ingredient content
Reproducibility R = 7.3 g/kg at 994 g/kg active ingredient content

FLUMIOXAZIN WETTABLE POWDER
578/WP/m/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. As for **578**/TC/m/2.1

2.2 Infrared. Extract the sample with a suitable solvent, filter and evaporate the solvent with a stream of clean dry air. Proceed as for **578**/TC/m/2.2

3 FLUMIOXAZIN. As for **578**/TC/m/3 except:

change 'PROCEDURE *(d) Preparation of sample solution.*' as follows:
(d) Preparation of sample solution. Weigh in duplicate (to the nearest 0.1 mg) sufficient sample to contain about 50 mg (*w* mg) of flumioxazin into separate volumetric flasks (100 ml). Add acetonitrile (about 80 ml) and place the flasks in an ultrasonic bath for 10 min. Allow to cool to room temperature, dilute to volume with acetonitrile. Mix thoroughly. Filter a

portion of each sample solution through a 0.45 μm filter prior to analysis (Solutions S_A and S_B).

Repeatability r = 7.2 g/kg at 516 g/kg active ingredient content
Reproducibility R = 26 g/kg at 516 g/kg active ingredient content

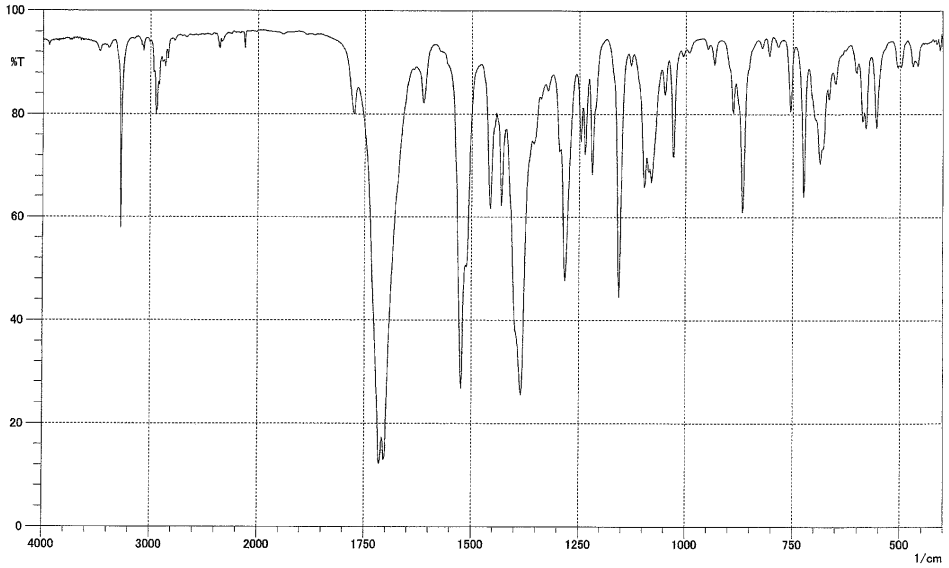


Figure 1 Infrared Spectrum of Flumioxazin

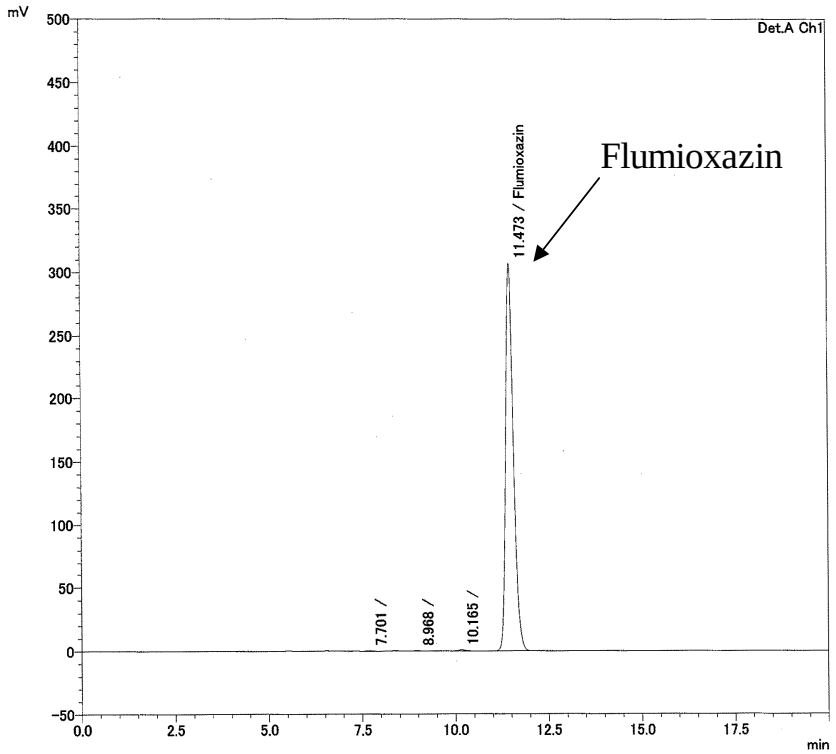


Figure 1 Example of Liquid Chromatogram of Flumioxazin TC

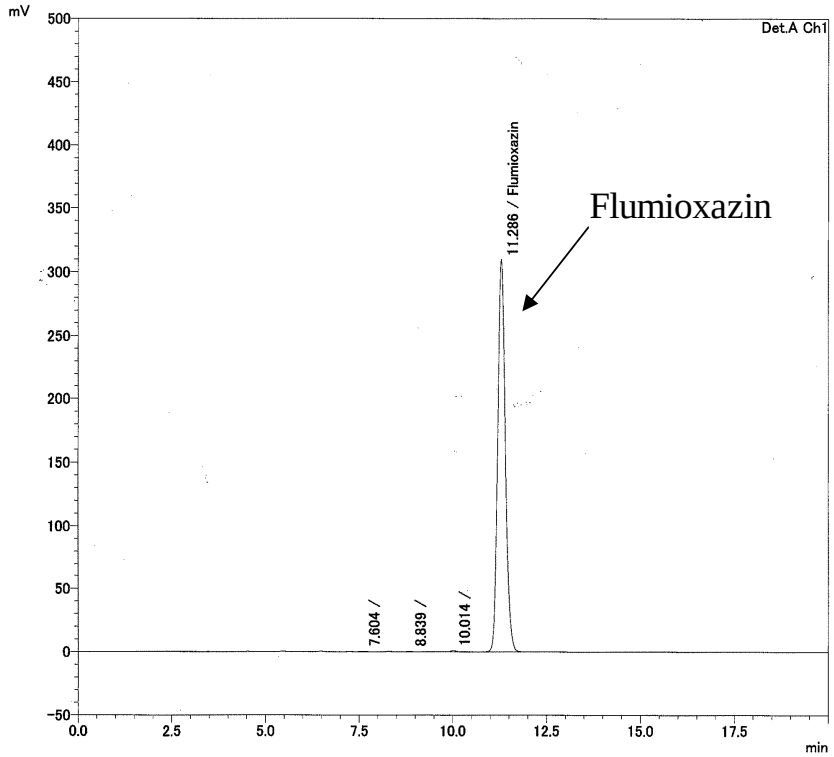


Figure 2 Example of Liquid Chromatogram of Flumioxazin WP