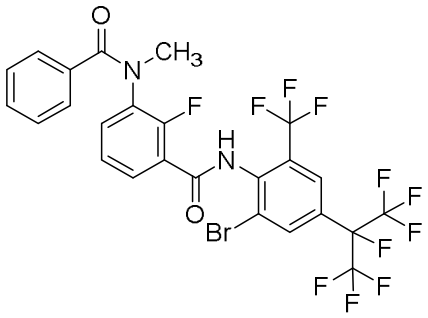
BROFLANILIDE
No.994



ISO common name	Broflanilide
Chemical name	*N*-[2-bromo-4-(perfluoropropan-2-yl)-6-(trifluoromethyl) phenyl]-2-fluoro-3-(*N*-methylbenzamido)benzamide (IUPAC) 3-(benzoylmethylamino)-*N*-[2-bromo-4-[1,2,2,2-tetrafluoro-1-(trifluoromethyl)ethyl]-6-(trifluoromethyl)phenyl]-2-fluorobenzamide (CAS)
CAS No.	1207727-04-5
Empirical formula	$C_{25}H_{14}BrF_{11}N_2O_2$
RMM	663.29
m.p.	154.0 - 155.5ºC
v.p.	9×10^{-9} Pa (25 ºC)
Solubility	In water: 0.71 mg/l, *n*-heptane: 0.096 g/l, xylene: 6.0 g/l, 1,2-dichloroethane: 110 g/l, acetone, ethyl acetate and methanol: >250 g/l, *n*-octanol: 7.4 g/l, acetonitrile: 77 g/l ; all at 20 ºC
Description	White to beige powder
Stability	Stable at room temperature
Formulation	Wettable powders

BROFLANILIDE TECHNICAL
994/TC/m/-

1. **Sampling.** Take at least 100 g

2. **Identity tests**

2.1 **HPLC.** Use the HPLC method below. The retention time of broflanilide for the sample solution should not deviate by more than 2% from that for the calibration solution.

2.2 **Infrared.** Apply sample to IR spectrophotometer and scan the sample directly from 4000 to 400 cm^{-1} to determine by ATR method. The spectrum produced from the sample should not differ significantly from that of the standard.

3. **Broflanilide**

OUTLINE OF METHOD Broflanilide is determined by reversed phase high performance liquid chromatography using UV detection at 254 nm and external standardization.

REAGENTS

Acetonitrile HPLC grade

Water HPLC grade

Broflanilide standard of known purity

Dilution solution Mobile phase

Mobile phase Acetonitrile – Water (65 + 35) (v/v)

Calibration solution. Weigh in duplicate (to the nearest 0.01 mg) 50 mg (*s* mg) of broflanilide standard into separate 100 ml volumetric flasks. Fill it up to the mark with dilution solution.

APPARATUS

High performance liquid chromatograph equipped with a detector suitable for operation at 254 nm, constant-temperature column compartment and an injector capable of delivering 20 µl

Column 250 x 4.6 mm (i.d.) Waters XSelect CSH C_{18} 5µm, or equivalent

Electronic integrator or data system

Filtration unit equipped with a PFTE membrane, 0.45 µm

Ultrasonic bath

PROCEDURE

(a) Liquid chromatographic conditions (typical):

Column	250 x 4.6 mm (i.d.) Waters XSelect CSH C_{18} 5µm, or equivalent
Mobile phase	Acetonitrile – Water, 65 + 35 (v/v)
Column temperature	40 °C
Flow rate	1.0 ml/min
Detection wavelength	254 nm
Injection volume	20 µl
Retention time	Broflanilide： approximately 11.5 min.

(b) Linearity check. Check the linearity of the detector response by injecting 20 µl of broflanilide reference standard solutions at concentrations of 0.5, 1 and 2 times that of the calibration solution.

(c) System equilibration. Inject 20 µl portions of the calibration solution C1 and repeat the injections until peak areas deviate by less than ±1.0% of two consecutive injections. Then inject consecutively two 20 µl portions of the second calibration solution (C2). The mean response factor for this solution should not deviate by more than 1.0% from that of the first calibration solution (C1), otherwise prepare new calibration solutions.

(d) Sample preparation. Prepare sample solutions in duplicate. Weigh (to the nearest 0.01 mg) enough sample to contain 50 mg of broflanilide into separate 100 ml volumetric flasks. Fill it up to the mark with dilution solution (sample solutions TC-1-A, TC-1-B, TC-2-A and TC-2-B). TC sample is prone to charge with static electricity.

(e) Determination. Inject in duplicate 20 µl portions of each sample solution bracketing them by injections of the calibration solutions as follows;
C1, TC-1-A, TC-1-A, C2, TC-1-B, TC-1-B, C1, TC-2-A, TC-2-A, C2 ...
Determine 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 broflanilide contents of the bracketed sample injections.

$$fi = \frac{s \times P}{Hs}$$

Where,

fi = individual response factor

Hs = peak area of broflanilide in the calibration solution

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

P = purity of broflanilide standard (g/kg)

$$\text{Broflanilide content} = \frac{f \times Hw}{w} \quad \text{(g/kg)}$$

Where,

f = mean response factor

Hw = peak area of broflanilide in the sample solution

w = mass of sample taken (mg)

Repeatability r = 35 to 47 g/kg at 997 g/kg active ingredient content

Reproducibility R = 47 to 52 g/kg at 997 g/kg active ingredient content

BROFLANILIDE WETTABLE POWDER
994/WP/m/-

1. Sampling. Take at least 500 g

2. Identity tests

2.1 HPLC. As for broflanilide technical 994/TC/m/2.1

2.2 Infrared. Suspend the sample in ethyl acetate and treat in an ultrasonic bath for 5 min. Filter the supernatant through a microfilter. Evaporate the filtrate and dry under reduced pressure at 40°C. Proceed as for broflanilide technical 994/TC/m/2.2.

3. Broflanilide. As for broflanilide technical 994/TC/m/3 except:

(d) Sample preparation. Prepare sample solutions in duplicate. Weigh (to the nearest 0.01 mg) enough sample to contain 50 mg of broflanilide (100 mg of broflanilide WP) into separate 100 ml volumetric flasks. Add about 70 ml of dilution solution, and place the flasks in an ultrasonic bath for about 5 min and fill up to the mark with dilution solution. And filter the supernatant through 0.45µm filter (sample solutions WP-1-A, WP-1-B, WP-2-A, WP-2-B, WP-3-A and WP-3-B).

Repeatability r = 11 to 12 g/kg at 500 to 502 g/kg active ingredient content
Reproducibility R = 21 to 24 g/kg at 500 to 502 g/kg active ingredient content

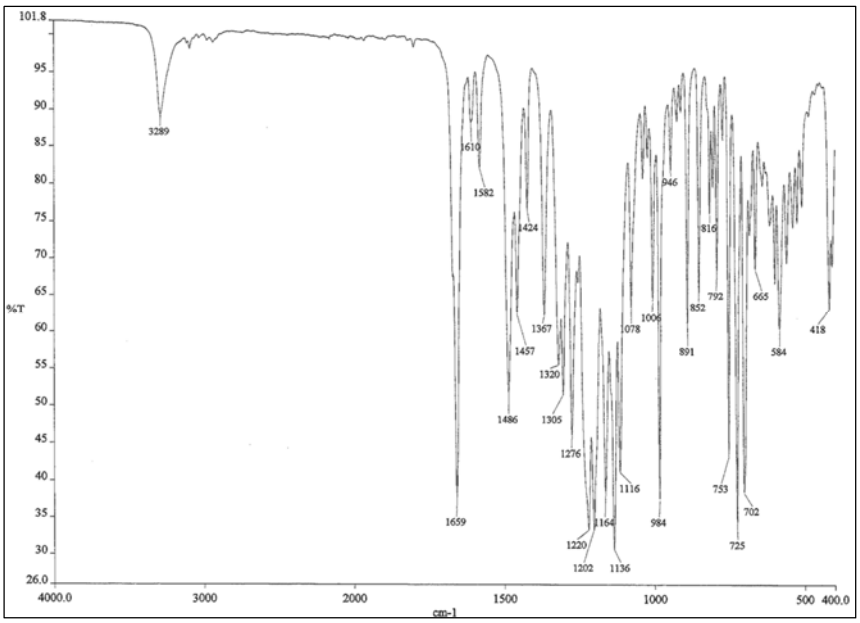


Figure 1 Infrared spectrum of broflanilide

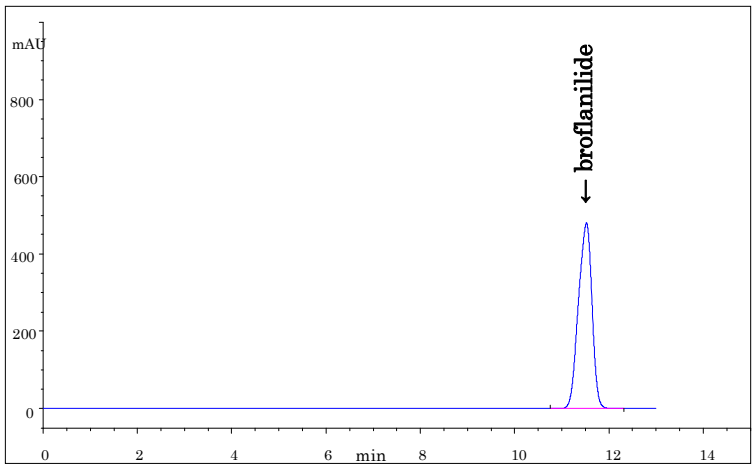


Figure 2 Chromatogram of broflanilide TC

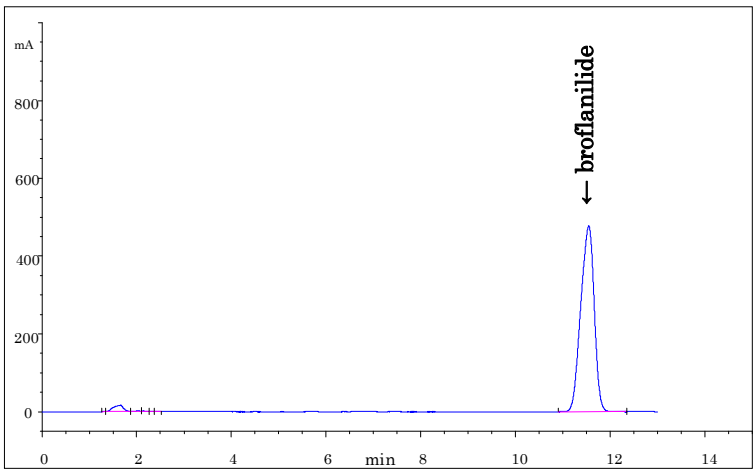


Figure 3 Chromatogram of broflanilide WP