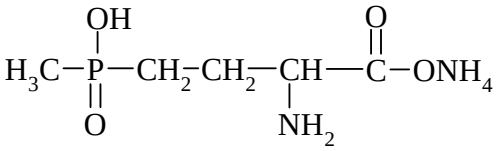
GLUFOSINATE-AMMONIUM
437



ISO common name	Glufosinate-ammonium
Chemical name	Ammonium-DL-homoalanin-4-yl(methyl)-phosphinate (IUPAC) (CA; *77182-82-2)*
Empirical formula	$C_5H_{15}N_2O_4P$
RMM	198.2
m.p.	Decomposes
v.p.	Not measurable
Solubility	In water: 1370 g/l; low solubility in most organic solvents (less than 1 g/l)
Description	Colourless crystals
Stability	Stable under abiotic conditions
Formulations	Soluble concentrates

GLUFOSINATE-AMMONIUM TECHNICAL
*__437__/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The retention time of glufosinate from the sample solution should not deviate by more than 5 % from that from the calibration solution.

3 Glufosinate-ammonium

OUTLINE OF METHOD Glufosinate-ammonium is dissolved in an aqueous solution of potassium dihydrogen phosphate and determined by HPLC with a strongly basic anion exchange column and UV detection at 195 nm.

REAGENTS

Potassium dihydrogen phosphate

Eluent. Prepare a solution of potassium dihydrogen phosphate in highly purified (HPLC grade or bi-distilled) water, $c(KH_2PO_4)$ = 0.1 mol/l.

Acetonitrile HPLC grade

Phosphoric acid solution $c(H_3PO_4)$ = 0.1 mol/l

Dichloromethane HPLC grade

Glufosinate-ammonium standard of known purity

Calibration solution. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) about 100 mg (*s* mg) of glufosinate-ammonium standard, dissolve in eluent solution (about 50 ml) by shaking until a homogeneous solution is obtained. Make up to volume with eluent solution.

* CIPAC method 1993. Prepared by the German Committee (DAPA). Chairman: Dr W Dobrat. Based on a method supplied by Hoechst AG, Germany.

APPARATUS

High performance liquid chromatograph consisting of a constant flow pump (e.g. DuPont model 8800), a 20 µl loop injector (e.g. Rheodyne 7410), a UV spectrophotometer operating at 195 nm, and a column oven heated at 30 °C

Column stainless steel, 125 × 4.6 (i.d.) mm packed with Nucleosil SB, 5µm

Electronic integrator or *data processing system*

PROCEDURE

(a) Preparation of the column. Prepare new columns (usually filled by the manufacturer with octane or a comparable solvent) as follows:

Connect the column to the HPLC instrument, but *not* to the detector. Flush the column consecutively with dichoromethane (50 ml), acetonitrile (50 ml), phosphoric acid (50 ml, $c(H_3PO_4)$ = 0.1 mol/l) and finally eluent solution (200 ml). Maintain a flow rate of 1 to 2 ml/min. Then connect the column to the detector and maintain the operating conditions below until a stable baseline is obtained. This may take about 8 h.

(b) Operating conditions (typical):

Eluent	potassium dihydrogen phosphate solution, 0.1 mol/l
Flow rate	1.3 ml/min
Column temperature	30 °C
Detector wavelength	195 nm
Injection volume	20 µl. It is essential that the loop is filled completely.
Detector sensitivity	The linear range of the detector must not be exceeded, otherwise the sample masses have to be adapted.
Retention time	glufosinate: about 2.5 min. Adjust the flow rate if the retention time is not between 2 and 3 min.

(c) Preparation of sample. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) enough sample to contain about 100 mg (*w* mg) of glufosinate-ammonium. Add eluent solution (about 50 ml), shake the flask until a homogeneous solution is obtained and make up to volume with eluent solution.

(d) Determination. Inject 20 µl portions of two calibration solutions of which the glufosinate-ammonium content differs at least 10 % and measure the glufosinate-ammonium peak areas. Inject each calibration solution at least twice and calculate the response factor (*f*). The individual factors should not deviate from the mean by more than 0.5 %, otherwise repeat the calibration sequence. Then inject in duplicate 20 µl portions of each sample solution. After a series of 4 sample injections repeat the injection of the calibration solution at the end of the series. Measure the relevant peak areas. Use the average calibration factors of the calibration solutions preceding and following the series of sample solution injections to calculate the glufosinate-ammonium content.

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

$$\text{Glufosinate-ammonium content} = \frac{H_w \times f}{w} \text{ g/kg}$$

where:

f	=	response factor
H_s	=	peak area of glufosinate-ammonium in the calibration solution
H_w	=	peak area of glufosinate-ammonium in the sample solution
s	=	mass of glufosinate-ammonium in the calibration solution (mg)
w	=	mass of sample taken (mg)
P	=	purity of glufosinate-ammonium standard (g/kg)

Repeatability r = 25 g/kg at 950 g/kg active ingredient content
Reproducibility R = 34 g/kg at 950 g/kg active ingredient content

GLUFOSINATE-AMMONIUM SOLUBLE CONCENTRATES
*437/SL/M/-

1 Sampling. Take at least 500 ml.

2 Identity tests. As for glufosinate-ammonium technical **437**/TC/M/2.

3 Glufosinate-ammonium. As for glufosinate-ammonium technical **437**/TC/M/3.

Repeatability r	=	3.2 g/kg at 140 g/kg active ingredient content
	=	3.4 g/kg at 180 g/kg active ingredient content
	=	16 g/kg at 500 g/kg active ingredient content
Reproducibility R	=	4.3 g/kg at 140 g/kg active ingredient content
	=	4.5 g/kg at 180 g/kg active ingredient content
	=	16 g/kg at 500 g/kg active ingredient content

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