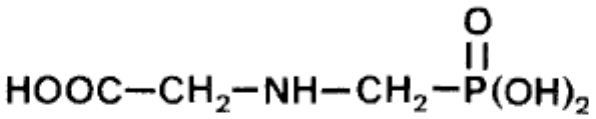
GLYPHOSATE

284



ISO common name	Glyphosate
Chemical name	N-(phosphonomethyl)glycine (IUPAC and CA; *1071-83-6*)
Empirical formula	$C_8H_8NPO_4$
RMM	169·1
m.p.	230°C (decomp.)
Solubility	12 g/l in water at 25°C; insoluble in common organic solvents
Description	The pure material is a white solid
Formulations	Water based solution of glyphosate mono-(isopropylamine) salt

GLYPHOSATE TECHNICAL

***284**/TC/(M)/–

1 Sampling. Take a sample of at least 50 g.

2 Identity tests. The identity is checked simultaneously with the determination of glyphosate and is confirmed if the difference between the retention time of glyphosate with respect to the external standard for the sample solution does not deviate by more than 1·5% from that for the calibration solution.

3 Glyphosate

OUTLINE OF METHOD Phosphate buffer mobile phase solutions containing glyphosate at a concentration of approximately 4·0 mg/ml are prepared by dissolution of appropriate subsamples. The glyphosate content is determined by ion-exchange chromatography through a fixed volume loop into a 250 × 4·6 mm i.d., stainless steel column packed with a strong anion exchange resin. Measurements are made using a UV detector at 195 nm and electronic area integration. External standardization is used.

REAGENTS

Methanol HPLC grade
Water HPLC grade. Use this water for all solutions
Potassium dihydrogenphosphate. Primary reagent grade
Phosphoric acid 85% reagent grade

* AOAC-CIPAC Provisional Method 1983.

Glyphosate. Reference grade standard of known purity. Dry for 2 hr at 105°C before weighing.
Mobile phase. Dissolve 0·8437 g potassium dihydrogen phosphate in 960 ml water. Add 40 ml of methanol and mix well. Adjust the pH of the solution to 1·9 with 85% phosphoric acid using a pH meter standardized at pH 2·0. The mobile phase must be filtered and degassed before use.
Calibration solution. Into a 100 ml volumetric flask, weigh (to the nearest 0·1 mg) a mass of 400 ± 10 mg (*s* mg) of reference grade glyphosate. Dilute to volume with mobile phase and stir or shake to dissolve. 30 min may be required to dissolve the standard. The solution is stable for at least 1 week.

Apparatus

Ion Exchange or Liquid Chromatograph fitted with an ultraviolet detector capable of operating at 195 nm and a pulse-free pump capable of generating a pressure of at least 8 MPa (about 1200 psi). The injection system should be a loop (50 µl) e.g. Rheodyne Model 7120 syringe loading available from Supelco Inc., U.S.A. or equivalent.
Microsyringe suitable for use with the injection system
Strip chart recorder 10 mV full scale
Electronic integrator capable of handling the detector output
HPLC column 250 × 4·6 mm i.d., stainless steel packed with a strong ion exchange resin e.g. Partisil 10 SAX as supplied by Whatman Inc. or equivalent
pH meter. Any pH meter with glass electrode, standardized at pH 2·0 with buffered solution.

Procedure

(*a*) *Operating conditions* (typical):
Column temperature: ambient
Flow rate: 2·3 ml/min
Pressure: about 8 MPa
Wavelength: 195 nm
Retention time: glyphosate 2·5 to 4·0 min
Attenuation: 0·2 AUFS (absorbance unit full scale)
Chart speed: 0·5 cm/min
Injection volume: 50 µl

(*b*) *Sample preparation.* Weigh in duplicate (to the nearest 0·1 mg) sufficient sample (*w* g) to contain 400 mg of glyphosate and transfer into 100 ml volumetric flasks containing 50 ml of mobile phase. Treat each sample solution as follows: Dilute to volume with mobile phase and mix well.

(*c*) *Determination.* Pump the mobile phase through the chromatograph until a steady base line is obtained. This may take up to 1 hr with a new column. When new columns are installed or when the instrument has not been used for 24 hrs make at least 6 rapid injections of 50 µl each of the calibration solution before beginning the analysis. Inject 50 µl aliquots of the calibration solution until the peak areas for successive injections agree to within +1%. Then inject 50 µl aliquots of the sample solutions until the peak areas agree to within ±1%. Let all

components from sample solutions elute (approx. 10–12 min.) before making the next injection.

(*d*) *Calculation.* Average peak areas from 2 successive injections of calibration solution that agreed to within ±1% (R') and from 2 successive injections of sample that agreed to within ±1% (R).

$$\text{The content of glyphosate} = \frac{R \times s \times P}{R' \times w} \text{ g/kg}$$

where: s = mass of standard glyphosate in the calibration solution (in mg)
w = mass of sample (in mg)
P = purity of standard glyphosate (g/kg)

To convert g/kg glyphosate to isopropylamine salt multiply the result found by 1·3496.

GLYPHOSATE SOLUBLE CONCENTRATE

284/SL/(M)/–

1 Sampling. Take a sample of at least 500 ml.

2 Identity tests. As for the technical **284**/TC/(M)/2.

3 Glyphosate. As for the technical **284**/TC/(M)/3, except:

PROCEDURE

(*d*) *Calculation.* If the concentration of glyphosate or glyphosate isopropylamine salt is required in terms of weight/volume, determine the density (d) of the formulation using MT 3, CIPAC 1, *p. 83*. Calculate the glyphosate content as follows:

$$\text{glyphosate content (g/l)} = \text{glyphosate g/kg} \times d$$

The glyphosate isopropylamine salt content can be calculated from glyphosate g/l multiplied by 1·3496.