GLYPHOSATE
284

See Glyphosate, CIPAC 1C, *p.2132*.

GLYPHOSATE WATER SOLUBLE GRANULES
[*]**284**/SG/(M)/-

1 Sampling. Take et least 500 g.

2 Identity test. Use the HPLC method below. The retention time of glyphosate for the sample solution should not deviate by more than 1.5 % from that for the calibration solution.

3 Glyphosate

OUTLINE OF METHOD The sample is dissolved in a phosphate buffered mobile phase and the glyphosate is determined by high performance liquid chromatography using a ion exchange column, UV detection at 195 nm, and external standardisation.

REAGENTS

Water HPLC grade. Use this water for all solutions.
Methanol HPLC grade
Potassium dihydrogen phosphate primary reference grade
Phosphoric acid 85 %
Glyphosate standard of known purity. Dry at 105 °C before use.
Mobile phase. Dissolve potassium dihydrogen phosphate (0.8437 g) in water (960 ml). Add methanol (40 ml) and mix well. Using a pH meter adjust the pH to 2.1 with phosphoric acid. Filter and degas before use.
Calibration solutions. Weigh (to the nearest 0.1 mg) into separate volumetric flasks (100 ml) 0.2, 0.4 and 0.6 g of glyphosate. Dissolve in, and fill to the mark with mobile phase. Mix well (solutions C_A, C_B, and C_C respectively).

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[*] Provisional AOAC-CIPAC method 1996.

APPARATUS

Liquid chromatograph capable of generating a pressure of at least 21 MPa (about 300 psi), and equipped with a UV detector capable of operating at 195 nm and a 50 µl fixed loop injector

Chromtographic column 250 × 4.6 (i.d.) mm, stainless steel, packed with a strong ion exchange resin, Whatman Anion Exchange PN 422-002, or equivalent

Electronic integrator or data system

pH meter calibrated at pH 2.0

PROCEDURE

(a) Operating conditions (typical):

Eluent flow rate	set to produce a retention time given below
Temperature	ambient
Injection volume	50 µl
Detector wavelength	195 nm
Retention time	glyphosate: 3.9 to 5.9 min. Adjust the pH of the mobile phase and the flow rate if necessary.
Run time	16 min. Most samples contain a late eluting peak which approximately doubles the run time for full elution.

(b) System suitability check. Equilibrate the column by pumping mobile phase through the column for about one hour. Then inject a practice sample and check if baseline separation of the glyphosate peak is obtained from both surfactants peaks (retention time 1 to 3 min) as well as the small impurity peak immediately following the glyphosate peak. Then make duplicate injections (50 µl) of the three calibration solutions and record the peak areas. A plot of the mean peak area versus the mass (in mg) should give a linear curve. The regression coefficient should be not less than 0.999. The individual response factors (area to mass ratio) should not differ by more than 0.01.

(c) Preparation of sample. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) enough sample to contain about 0.4 g glyphosate (*w* mg). Dissolve in, and fill to the mark with mobile phase. Mix well.

(d) Determination. Inject in duplicate 50 µl aliquots of the sample solution bracketed by duplicate injections of calibration solutions C_A and C_B. Measure the peak areas and calculate the mean for each solution. Taking the results of the two calibration solutions establish the regression line of the response versus the mass (mg). The slope and the intercept should be within the limits of the regression curve established under *(b).*

(e) Calculation

$$\text{Content of glyphosate} = \frac{(H - b) \times P}{a \times w} \text{ 1g/kg}$$

where:

H = peak area of glyphosate in the sample solution

a = slope of the regression line

b = intercept of the regression line

P = purity of the glyphosate standard g/kg

w = mass of sample taken (mg)

Repeatability r = 11, 9 and 11 g/kg at 634, 689 and 731 g/kg active ingredient content respectively

= 5 to 8 g/kg at 874 g/kg active ingredient content

Reproducibility R = 11, 14 and 13 g/kg at 634, 689 and 731 g/kg active ingredient content respectively

= 6 to 8 g/kg at 874 g/kg active ingredient content