

DETERMINATION OF GLYPHOSATE RESIDUE IN OIL MATRIX PART I. PRE-COLUMN DERIVATIZATION METHOD

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289

MPOB INFORMATION SERIES • ISSN 1511-7871 • JUNE 2005

MPOB TT No. 292

SCOPE

This test method prescribes the requirements for the determination of glyphosate residue in oil matrix.

DEFINITION

Glyphosate is the common name for *N*-(phosphonomethyl) glycerine $C_3H_8NO_5P$ and is sold commercially as Roundup. Pure glyphosate has a zwitterions structure (Figure 1). The crystals are colourless with a melting point of $200^{\circ}C$, a bulk density of 0.5 g litre^{-1} , a vapour pressure of 0.04 mPa and a solubility in water of 12 g litre^{-1} . The acid form of glyphosate is less soluble in water than the salt.

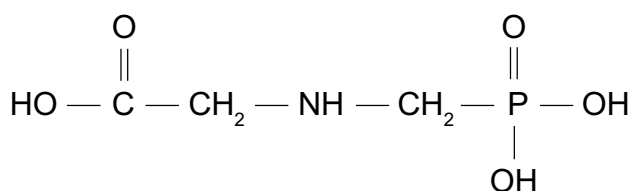


Figure 1. Structure of *N*-(phosphonomethyl) glycerine or glyphosate.

PRINCIPLE

The method involved extracting the residue from the oil matrix using chloroform, cation exchange

clean up, derivatization using 9-fluorenyl methylchloroformate followed by high performance chromatographic analysis using fluorescence detector (Figures 2 and 3).

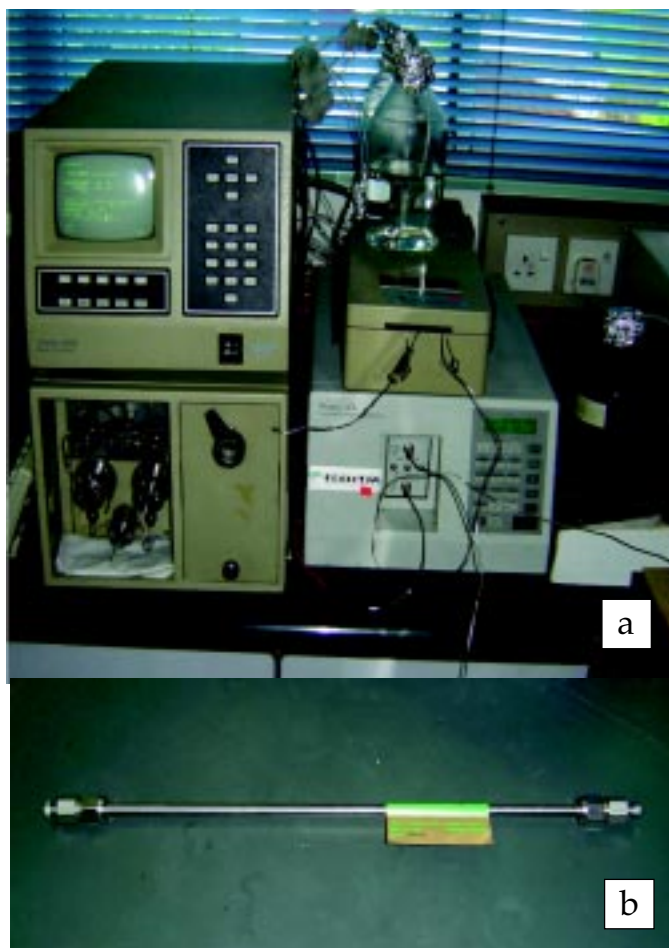


Figure 2. HPLC with (a) fluorescence detector and (b) cation exchange column for glyphosate analysis.

ISSN 1511-7871



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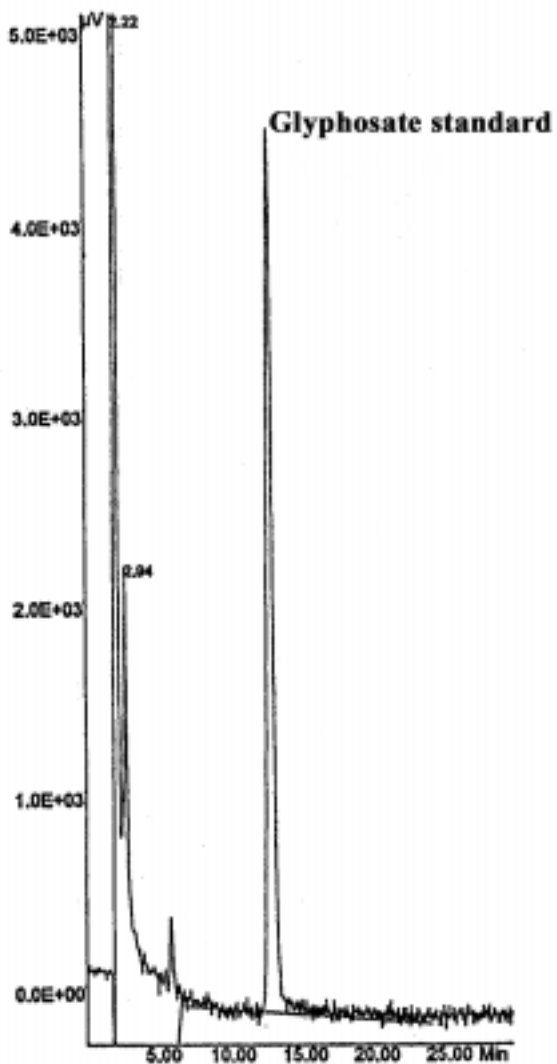


Figure 3. Chromatogram of standard glyphosate.

RECOVERY

Recoveries of glyphosate at the range of 0.05-1.00 $\mu\text{g g}^{-1}$ were 68.5%-101.1%.

Coefficient of variations were <10% for high concentrations and <15% for low concentrations.

Limit of detection was 0.02 $\mu\text{g g}^{-1}$.

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