

MULTIRESIDUE ANALYSIS OF ORGANOCHLORINE PESTICIDES IN OIL MATRIX USING SOLID PHASE EXTRACTION

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411

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Organochlorine pesticides (OCPs) are now largely banned in Malaysia. However, with their long persistence, their residues may still be in the environment and food chain. OCPs are lipid soluble and their determination in oil requires pre-extraction from the oil matrix followed by a clean-up procedure. The latter is to prevent deterioration of the column and contamination of the detector in the gas chromatography (GC) system used for quantifying the OCPs.

SCOPE

The test method outlined below prescribes the requirements for determination of OCPs in crude palm oil.

DEFINITION

This method describes the requirements for determining OCPs in crude palm oil. The OCP compounds are α -BHC, β -BHC, γ -BHC (lindane), heptachlor, heptachlor epoxide, *cis*-chlordane, *trans*-chlordane, DDE, dieldrin, DDD and methoxychlor.

DETERMINATION OF OCPs IN CPO

Principle

The method involves extraction of the OCPs with hexane saturated with acetonitrile. The OCPs are preferentially partitioned into the polar acetonitrile layer while the bulk of the lipids remains solubilized in the non-polar hexane. The acetonitrile extract is then subject to a clean-up procedure using solid phase extraction (Figure 1). The OCPs in the cleaned extract are then analysed by GC equipped with a micro electron capture detector (GC- μ ECD).



Figure 1. Solid phase extraction manifold.

Recovery

Recoveries of OCPs from crude palm oil samples spiked with OCP standards in the range 0.02-0.33 $\mu\text{g g}^{-1}$ were 81.2% - 108.6%, with coefficients of variation of 0.99%-12.98%. The limits of detection and quantification for the various OCPs using GC- μ ECD (Figure 2) were 13-84 ng g^{-1} and



Figure 2. The GC- μ ECD for OCPs analysis.

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43-280 ng g⁻¹ respectively. Figure 3 shows the GC chromatogram of standard OCPs (0.3 µg ml⁻¹)

SERVICES AVAILABLE

- Quantification of OCPs in CPO;
- Cost of analysis per sample – RM 350; and
- Private laboratories are encouraged to adopt this method as one of the analyses covered in their

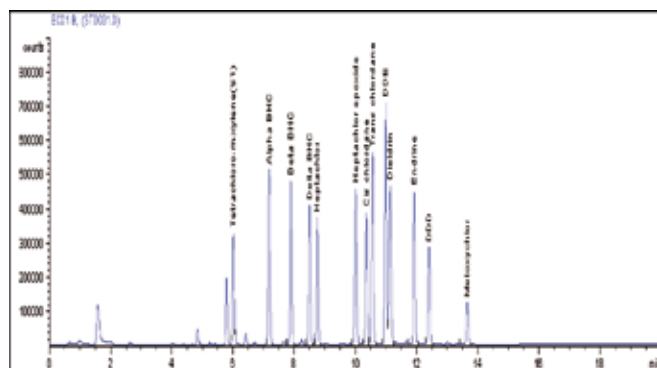


Figure 3. Chromatogram of standard OCPs.

scope of laboratory accreditation. The cost for method transfer, including competency training for an analyst, is negotiable.

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