

The pesticides used in oil palm plantations are mainly herbicides and insecticides. As insecticides are relatively cheap, they are freely used, like deltamethrin against leaf-eating insects. The insecticides may have adverse effects on the environment and human health, while their residues are a concern in food quality and safety. Reliable analytical methods for insecticide determination in palm oil are needed to safeguard public health.

SCOPE

The test methods herein described are for the determination of deltamethrin in CPO and CPKO.

DEFINITION

Deltamethrin is the common name for (*S*)- α -cyano-3-phenoxybenzyl (1*R*, 3*R*)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate, the formula for which is $C_{22}H_{19}Br_2NO_3$. It is the active ingredient in several insecticides, e.g. Butoflin, Butox and Crackdown. Deltamethrin is a synthetic pyrethroid, a colourless crystal with a melting point between 98°C-101°C. It has low solubility in water at 0.002 mg litre⁻¹ at 20°C. Its molecular weight is 505.21 and its chemical structure is shown in Figure 1.

DETERMINATION OF DELTAMETHRIN IN CPO

Principle

Deltamethrin is extracted from CPO using a mixture of petroleum ether and acetonitrile. It is

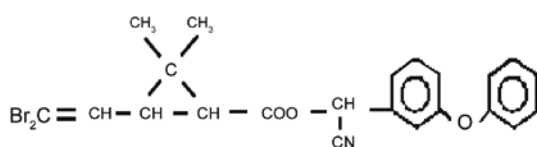


Figure 1. Chemical structure of deltamethrin.

preferentially partitioned in the polar acetonitrile layer while the bulk of the lipids dissolves in the non-polar petroleum ether. The acetonitrile extract is then cleaned up by passing it through a solid phase extraction cartridge (Figure 2). The detection and quantification of deltamethrin is by gas chromatography with an electron capture detector (GC-ECD) (Figure 3).

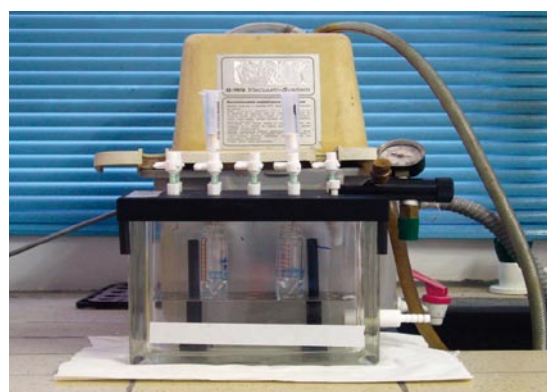


Figure 2. Solid phase extraction manifold.



Figure 3. GC-ECD for deltamethrin analysis.

Recovery

The recoveries of deltamethrin from CPO samples spiked with a deltamethrin standard at 0.02 - 0.5 $\mu\text{g ml}^{-1}$ were 78.1% - 100.9%, with coefficients of

variation of 1.2% to 4.3%. The limit of detection for deltamethrin using GC-ECD is $0.01 \mu\text{g ml}^{-1}$. Figure 4 is the GC-ECD chromatograms of (A)

untreated CPO, (B) standard deltamethrin, $0.1 \mu\text{g ml}^{-1}$, (C) CPO spiked to $0.1 \mu\text{g g}^{-1}$ deltamethrin, and (D) blank n-hexane.

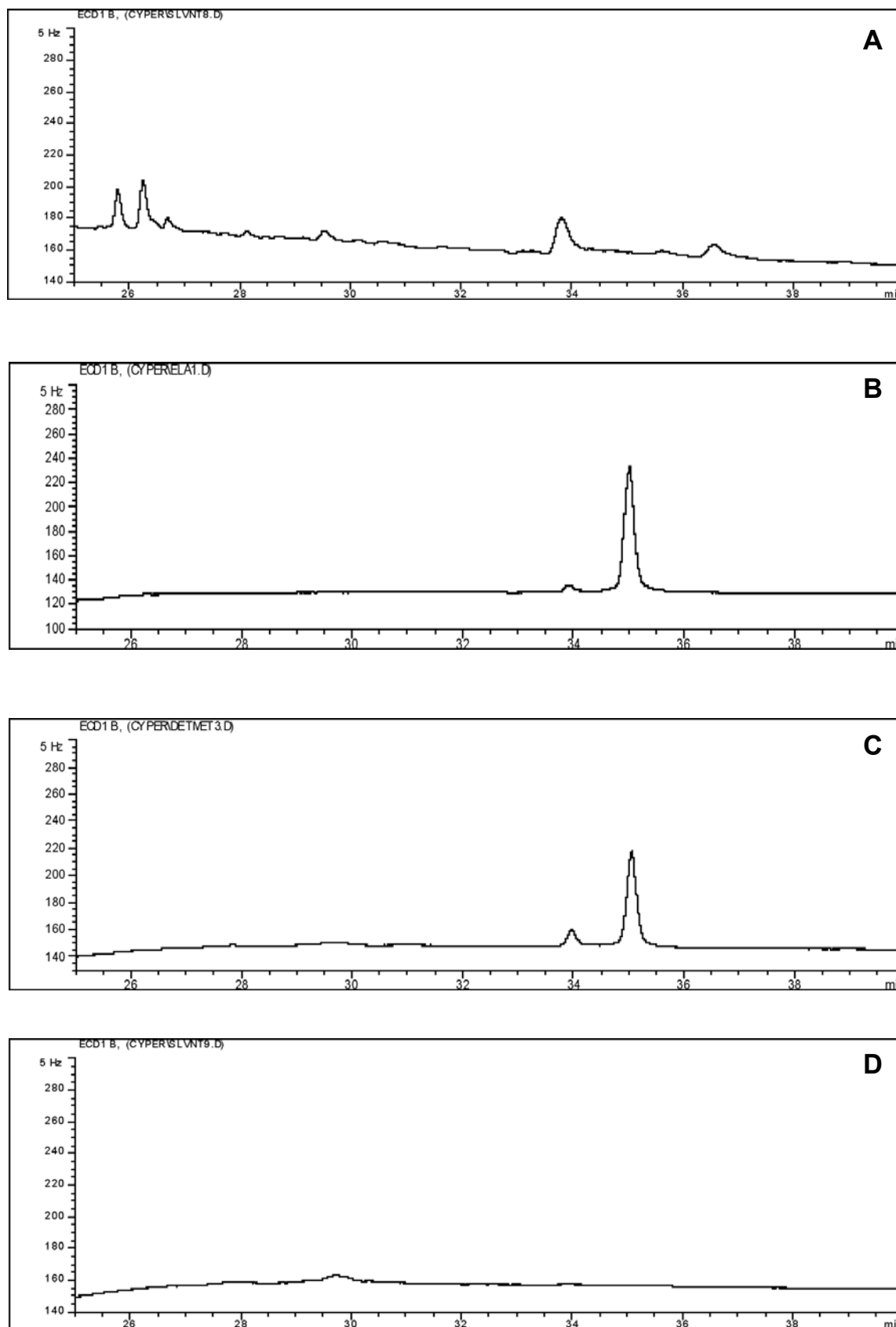


Figure 4. The GC-ECD chromatograms of (A) untreated CPO, (B) standard deltamethrin, $0.1 \mu\text{g ml}^{-1}$, (C) CPO spiked to $0.1 \mu\text{g g}^{-1}$ deltamethrin, and (D) blank n-hexane.

DETERMINATION OF DELTAMETHRIN IN CPKO

Principle

Deltamethrin is extracted from CPKO using acetonitrile, followed by low temperature treatment to separate the oil from the acetonitrile containing the deltamethrin. The detection and quantification of deltamethrin is by GC-ECD.

Recovery

The recoveries of deltamethrin from CPKO spiked to 0.03 - 0.30 $\mu\text{g g}^{-1}$ deltamethrin were 82.4% - 96.9%, with coefficients of variation of 1.0% to 3.3%. Figure 5 shows the GC-ECD chromatograms of A) untreated CPKO, B) standard deltamethrin solution, 0.5 $\mu\text{g ml}^{-1}$, C) CPKO spiked to 0.5 $\mu\text{g g}^{-1}$ deltamethrine, and D) blank n-hexane.

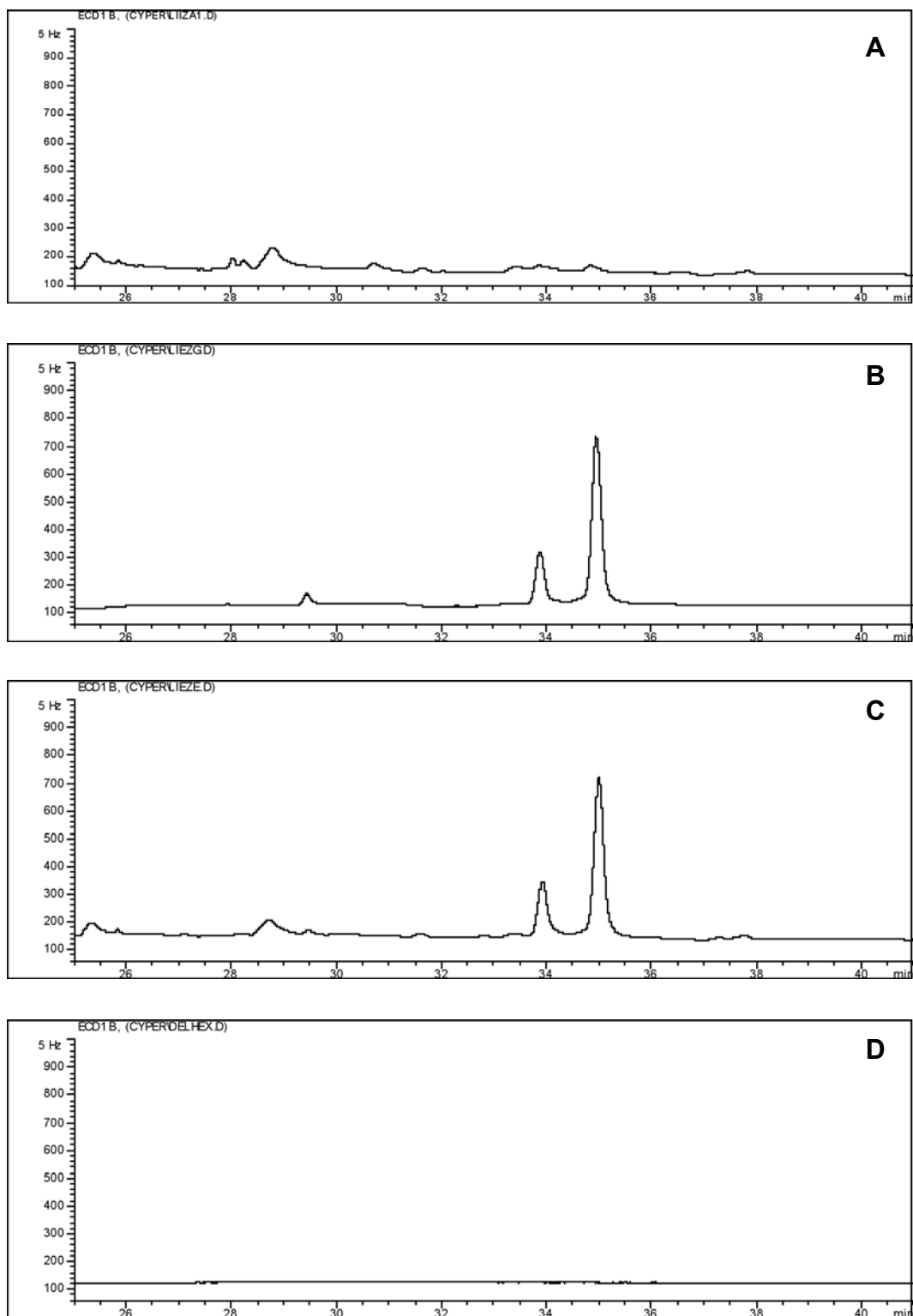


Figure 5. The GC-ECD chromatograms of A) untreated CPKO, B) standard deltamethrin solution, 0.5 $\mu\text{g ml}^{-1}$, C) CPKO spiked to 5 $\mu\text{g g}^{-1}$ deltamethrin, and D) blank n-hexane.

SERVICES AVAILABLE

- Quantification of deltamethrin in CPO and CPKO;
- Cost of analysis per sample per matrix – RM 350; and
- Private laboratories are encouraged to adopt this method as part of their scope of analyses. The cost of method transfer including competency training for an analyst is negotiable.

CONCLUSION

Deltamethrin is used in oil palm to control leaf-eating insects. It is important that the oil palm industry has a reference method for determining its residues in CPO and PKO.

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