DETERMINATION OF CYPERMETHRIN IN CRUDE PALM OIL AND CRUDE PALM KERNEL OIL BY SOLID PHASE EXTRACTION AND GAS CHROMATOGRAPHY ELECTRON CAPTURE DETECTION

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erbicides and insecticides are the main pesticides used in oil palm plantations. Being relatively cheap, insecticide such as cypermethrin often used to protect the oil palm

against leaf-eating insects. However, insecticides may have adverse effects on the environment and human health, and the presence of insecticide residues in food and food ingredients is of great concern. As such, reliable analytical methods for the detection and quantification of insecticide residues in palm oil and palm kernel oil are needed to safeguard public health.

SCOPE

The test method described herein is for the determination of cypermethrin in crude palm oil (CPO) and crude palm kernel oil (CPKO).

DEFINITION

Cypermethrin is the common name for (RS)- α -cyano-3 phenoxybenzyl (1RS)-cis,trans-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate ($C_{22}H_{19}Cl_2NO_3$). Aimcocyper, Ambush, Ammo, Cynoff and Cypercopal are some of the trade names for cypermethrin. In its pure form, cypermethrin is an odourless crystal, while a sample of 93% purity is a viscous semi-solid. Its melting point is in the range of 60°C-80°C. Cypermethrin has low solubility in water (0.01 mg litre-1) and its molecular weight is 416.3 (Kidd and James, 1991). Figure 1 shows the chemical structure of cypermethrin.

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Principle

Cypermethrin is extracted from oil matrices using acetonitrile. The extract is then subjected to low

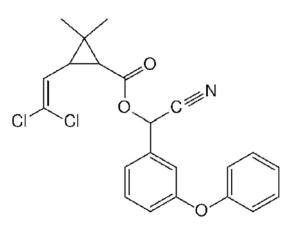


Figure 1. Chemical structure of cypermethrin.

temperature precipitation where the analyte partitions into the polar acetonitrile layer, while the bulk of the frozen oil phase can then be filtered off. The acetonitrile filtrate is then cleaned up by passing it through a solid phase extraction (SPE) cartridge (*Figure 2*). The detection and quantification of cypermethrin is by gas chromatography using an electron capture detector (GC-ECD) (*Figure 3*).

Recovery Studies

Recoveries of cypermethrin from CPO samples spiked with 0.05-1.0 mg kg⁻¹ cypermethrin standards range from 87%-98% with coefficients of variation between 4% and 8%. *Figure 4* shows the GC-ECD chromatograms of (A) standard cypermethrin at 0.1 μ g ml⁻¹, (B) Blank CPO, and (C) CPO spiked with 0.1 μ g ml⁻¹ cypermethrin standard.



Figure 2. Solid phase extraction manifold.





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Figure 3. GC-ECD for cypermethrin analysis.

For the extraction of cypermethrin from CPKO samples spiked with 0.05- $1.0~\mu g~g^{-1}$ of cypermethrin standard, the recoveries are between 83%-100% with coefficients of variation ranging from 3%-10%.

The limit of detection of cypermethrin in both CPO and CPKO is $0.01~\mu g$ ml⁻¹. *Figure 5* shows the GC-ECD chromatograms of (A) standard cypermethrin at $0.1~\mu g$ ml⁻¹, (B) blank CPKO, and (C) CPKO spiked with $0.1~\mu g$ ml⁻¹ cypermethrin.

SERVICES AVAILABLE

Services on offer include quantification of cypermethrin in CPO and CPKO.

Private laboratories are encouraged to adopt this method as part of their scope of analyses. The cost of method transfer including competency training for the analyst is negotiable.

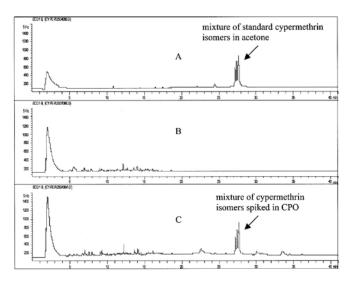


Figure 4. GC-ECD chromatograms of (A) standard cypermethrin at 0.1 μg ml⁻¹, (B) untreated CPO and (C) CPO spiked with 0.1 μg ml⁻¹ cypermethrin.

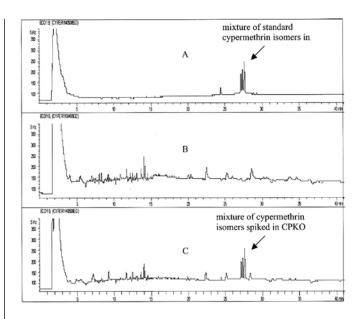


Figure 5. GC-ECD chromatograms of (A) standard cypermethrin at 0.1 μ g ml⁻¹, (B) blank CPKO and (C) CPKO spiked with 0.1 μ g ml⁻¹ cypermethrin.

CONCLUSION

Cypermethrin is used in oil palm plantations to control leaf-eating pests such as bagworms, nettle caterpillars and rhinoceros beetles. Therefore, it is important that the palm oil industry has a reference method for determining cypermethrin residues in CPO and CPKO.

REFERENCE

KIDD, H and JAMES, D R (1991). *The Agrochemicals Handbook*. Third edition. Unwin Brothers Limited, Old Woking, Surrey.

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