DETERMINATION OF HEXACONAZOLE IN FIELD SAMPLES USING GAS CHROMATOGRAPHY- ELECTRON CAPTURE DETECTION

(GC-ECD)

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pesticide is any biological, physical

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or chemical substance used to control, destroy or repel a pest. A fungicide is a chemical pesticide compound that kills or inhibits the growth of fungi. As fungicides can be toxic to humans and present chronic problems when contaminated food is consumed by people, it is important to monitor the use of fungicides in agriculture. In addition, there are environmental issues associated with the use of fungicides in agriculture, including accumulation in the soil and runoff into rivers and lakes.

In oil palm plantations, the fungicide hexaconazole is used to control *Ganoderma* infection that threatens to destroy or compromise the palm. The method of hexaconzole application is usually through soil drenching, trunk injection or a combination of the two methods. It is therefore important to have a method to determine the residual amount of hexaconzole in the field such as in samples of water, soil and leaf to monitor the use and fate of the fungicide in oil palm plantations.

SCOPE

The test method prescribes the requirements for the determination of hexaconazole in field samples such as of soil, water and leaf of the oil palm.

DEFINITION

Hexaconazole is the common name for the fungicide (RS)-2-(2,4-dichlorophenyl)-1-(1H-1,2,4-triazol-1-yl)hexan-2-ol. This fungicide is available in a variety of formulations under trade names such as Anvil (Syngenta), AGR AGI 5 EC (AGR Smart) and Swat (Zeenex Agroscience). Hexaconazole is from the family of azole fungicides with a melting

point ranging from 110°C - 112°C and a molecular mass of 314.2 (Kidd and James, 1991). The structure of hexaconazole is shown in *Figure 1*. It is a systemic fungicide with protective and curative functions. The mode of action is through the inhibition of ergosterol biosynthesis, and, thus, it can be used for the control of a range of fungi especially ascomycetes and basidiocetes.

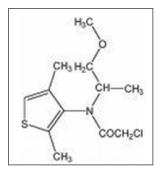


Figure 1. Chemical structure of hexaconazole.

DETERMINATION OF HEXACONAZOLE IN FIELD SAMPLES

PRINCIPLE

Soil

The method involves the extraction of hexaconazole from the soil matrix using dichloromethane. The extract is then subjected to solid phase extraction (SPE) for the clean-up step (*Figure* 2). The detection and quantification of hexaconazole is by gas chromatography attached to an electron capture detector (GC-ECD) as shown in *Figure* 3.

Water

Hexaconazole is extracted from water by loading the test sample onto a SPE C_{18} cartridge and eluting at a flow rate of 8 - 10 ml sec $^{-1}$ with methanol. The detection and quantification of hexaconazole is by GC-ECD.



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Figure 2. Solid phase extraction manifold.

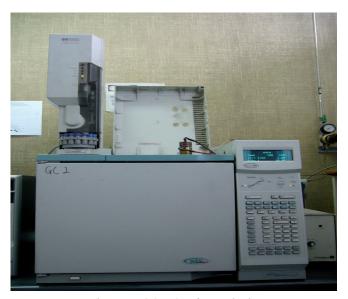


Figure 3. GC-ECD for analysis.

Leaf

Hexaconazole is extracted from leaf samples using a mixture of acetone, ethyl acetate and water with the aid of an ultrasonic bath. The extract is then dried and redissolved in ethyl acetate and cyclohexane before being subjected to gel permeation chromatography (GPC) as shown in *Figure 4*. Compounds with higher molecular weight such as chlorophyll will pass through first and subsequently the analyte is collected and concentrated by using a rotary evaporator. The detection and quantification of hexaconazole is by GC-ECD.



Figure 4. Gel permeation chromatography.

RECOVERY STUDIES

For all the field samples, the detection limit (LOD) of hexaconazole using GC-ECD was 2.0 μ g litre⁻¹. The retention time of hexaconazole was 16.9 min. *Figure 5* shows the GC-ECD chromatograms of (a) hexaconazole standard solution, (b) spiked soil sample, (c) spiked water sample, and (d) spiked leaf sample.

Soil

Recoveries of hexaconazole from soil samples spiked with 0.1 - $8.0~\mu g$ litre⁻¹ hexaconazole standard solutions ranged from 99% - 106% with coefficients of variation between 1.1% and 5.6%.

Water

Recoveries of hexaconazole from water samples spiked with 0.5 - $8.0~\mu g$ litre⁻¹ hexaconazole standards ranged from 95% - 105% with coefficients of variation between 2% and 6%.

Leaf

Recoveries of hexaconazole from leaf samples spiked with 0.005 - 0.01 μg ml⁻¹ hexaconazole standard solutions ranged from 90.56% - 103.11% with a standard deviation of less than 10%.

SERVICES AVAILABLE

Services on offer include:

- Quantification of hexaconazole in samples of soil, water and leaf of the oil palm.
- 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.

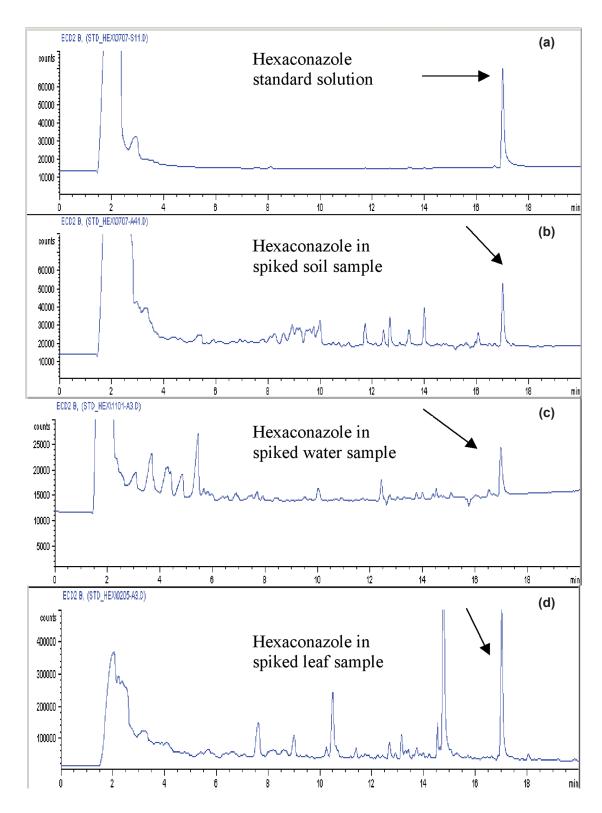


Figure 5. Chromatograms obtained for (a) hexaconazole standard solution, (b) spiked soil sample, (c) spiked water sample and (d) spiked leaf sample.

CONCLUSION

Hexaconazole is commonly used in oil palm plantations to control basal stem root (BSR) disease caused by *Ganoderma* infections. Therefore, it is important that the palm oil industry has a reference method for determining hexaconazole residues in soil, water and oil palm leaf samples collected from oil palm plantations.

REFERENCE

KIDD, H and JAMES, D R (1991). *The Agrochemicals Handbook*. 3rd Edition. Unwin Brothers Limited, Old Woking, Surrey.

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