

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

HALIMAH MUHAMAD, MAZNAH BINTI ZAINOL, ISMAIL SAHID and IDRIS ABU SEMAN

536

MPOB INFORMATION SERIES • ISSN 1511-7871 • JUNE 2010

MPOB TS No. 78

A pesticide is any biological, physical 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 the oil palm plantations, the fungicide hexaconazole is used to control *Ganoderma* infection that threatens to destroy or compromise the oil palm tree. The method of hexaconazole 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 hexaconazole in field samples such as water, soil and leaf in order to monitor the use and fate of the fungicide in oil palm plantations.

SCOPE

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

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 chemical 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 from the family of azole fungicides has 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 control of a range of fungi especially ascomycetes and basidiocetes.

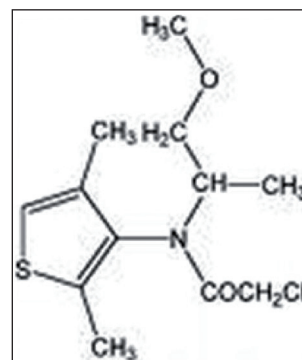


Figure 1. Chemical structure of hexaconazole.

DETERMINATION OF HEXACONAZOLE IN CPO AND CPKO

Principle

Hexaconazole is extracted from oil matrices using acetonitrile. The extract is then subjected to low-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 hexaconazole is by gas chromatography using an electron capture detector (GC-ECD) and is shown in Figure 3.



Figure 2. Solid phase extraction manifold.

ISSN 1511-7871



9 771511 787001

Malaysian Palm Oil Board, Ministry of Plantation Industries and Commodities, Malaysia

P. O. Box 10620, 50720 Kuala Lumpur, Malaysia. Tel: 03-87694400 Website: www.mpob.gov.my

Telefax: 03-89259446



MPOB

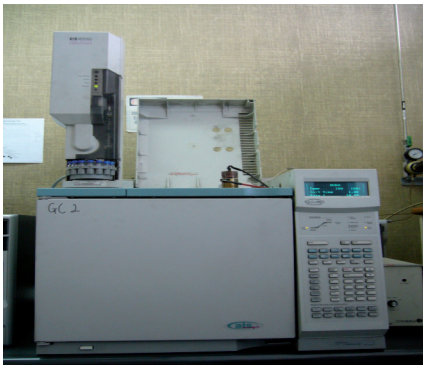


Figure 3. GC-ECD for hexaconazole analysis.

Recovery Studies

The recoveries from CPO samples spiked with $0.05 - 0.01 \mu\text{g ml}^{-1}$ hexaconazole standard ranged from 82.40% to 91.28% with standard deviations of less than 10%. Figure 4 shows the GC-ECD chromatograms obtained for a standard hexaconazole solution of $0.05 \mu\text{g ml}^{-1}$ an untreated sample of CPO (blank CPO) and a spiked sample of CPO containing $0.05 \mu\text{g ml}^{-1}$ hexaconazole.

Recoveries of hexaconazole in CPKO containing $0.05 - 0.01 \mu\text{g ml}^{-1}$ of hexaconazole ranged from 93.38% to 103.81% with relative standard deviation values below 6%. Figure 5 shows the GC-ECD chromatograms obtained for a standard hexaconazole solution of $0.05 \mu\text{g ml}^{-1}$, an untreated sample of CPKO and a spiked sample of CPKO containing $0.05 \mu\text{g ml}^{-1}$ hexaconazole. The detection limit (LOD) of hexaconazole was $2.0 \mu\text{g litre}^{-1}$.

SERVICES AVAILABLE

Services on offer include:

- Quantification of hexaconazole 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.

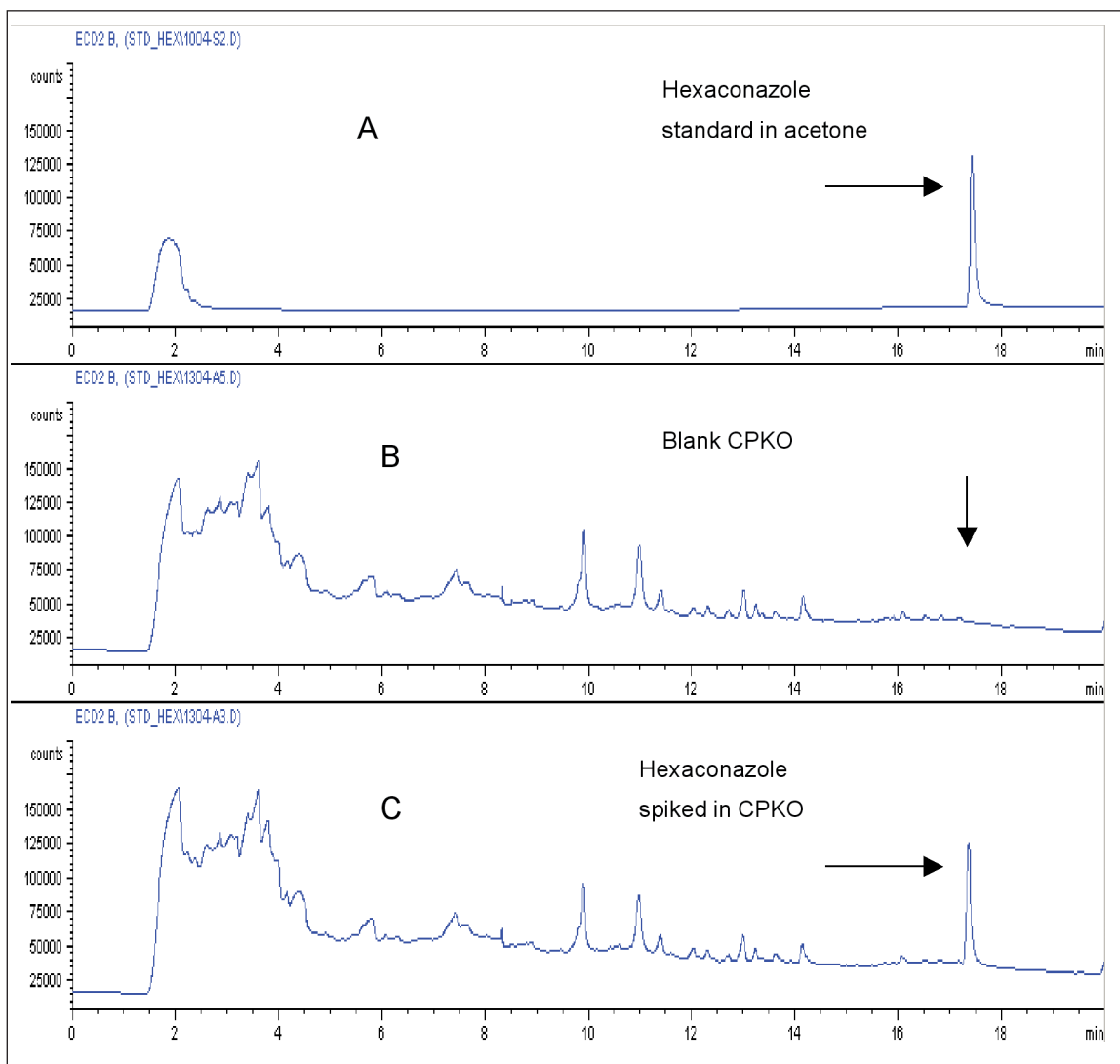


Figure 4. Chromatograms obtained for (A) hexaconazole standard solution ($0.05 \mu\text{g ml}^{-1}$), (B) blank CPO sample and (C) spiked CPO sample ($0.05 \mu\text{g ml}^{-1}$).

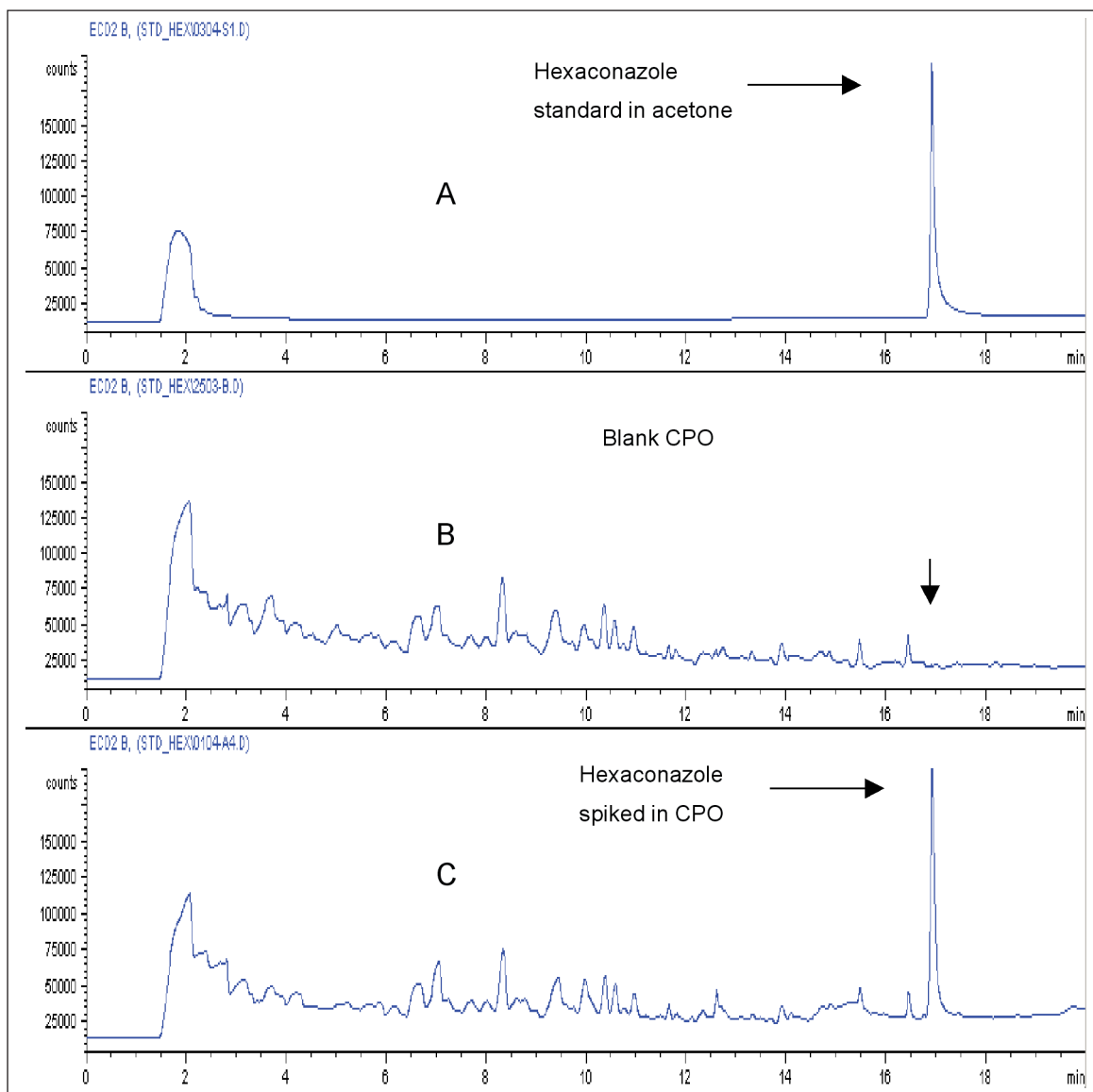


Figure 5. Chromatograms obtained for (A) hexaconazole standard solution ($0.05 \mu\text{g ml}^{-1}$), (B) blank CPKO sample and (C) spiked CPKO sample ($0.05 \mu\text{g ml}^{-1}$).

CONCLUSION

Hexaconazole is commonly used in oil palm plantations to control basal stem root (BSR) disease resulting from *Ganoderma* infections. Therefore, it is important that the palm oil industry has a reference method for determining hexaconazole residues in CPO and CPKO.

REFERENCE

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

For more information, kindly contact:

Director-General
MPOB
P. O. Box 10620
50720 Kuala Lumpur, Malaysia.
Tel: 03-8769 4400
Fax: 03-8925 9446
www.mpob.gov.my