

This test method describes a gas chromatographic mass selective detector (GC-MSD) procedure for the determination of 2-monochloropropane-1,2-diol (2-MCPD) esters, 3-monochloropropane-1,2-diol (3-MCPD) esters and Glycidol esters (GE) in oils and fats.

The 2-MCPD esters, 3-MCPD esters and GE are food-processing contaminants which occur during the processing of edible oil. *Figure 1* shows the chemical structures of 3-MCPD esters and GE. The formation of 2- and 3-MCPD esters is due to the interaction of triacylglycerols (TAG) or glycerol with chlorides at high temperatures (Ramli *et al.*, 2015). On the other hand, the formation of GE is suspected to be correlated to diglycerides (DAG) present in vegetable oils (Hamlet *et al.*, 2011; Matthäus *et al.*, 2011).

Since 2009, MPOB has verified and conducted analysis for determination of 3-MCPD esters using method by the Federal German Institute for Risk Assessment (BfR). This method was successfully transferred during the Transfer of Technology Seminar in 2011 (TOS No. 105). In 2014, MPOB has started verifying AOCS Official Method Cd 29a-13 for determination of 2- and 3-MCPD esters and GE in oils and fats.

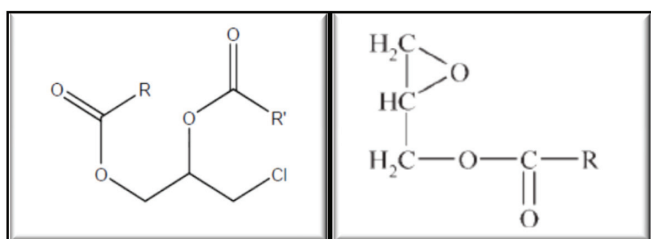


Figure 1. Chemical structures of 3-MCPD esters and GE.

PRINCIPLE

The AOCS Official Method Cd 29a-13 is based on the simultaneous determination of 2-MCPD esters, 3-MCPD esters and GE in edible oils and fats. The GE was first converted into 3-MBPD monoesters in acid solution containing bromide salt. The 3-MBPD esters, together with 2-MCPD esters and 3-MCPD esters were then converted into the free form in acid methanolic solution. The fatty acid methyl esters generated during the reaction were extracted out from the sample; and 2-MCPD, 3-MCPD and 3-MBPD were then derivatised with phenylboronic acid prior to gas chromatography-mass spectrometry (GC-MS) analysis. The GC-MS instrument used for the analysis is illustrated in *Figure 2*.



Figure 2. GC-MS instrument for the analyses of 2- and 3-MCPD esters and GE in oils and fats.

RESULTS

Method verification was carried out to obtain good linearity of calibration curve, as well as to determine the limit of detection (LOD) of these compounds. Results showed that calibration curves of these contaminants had good linearity,

with regression linear, $R^2 \geq 0.999$ (Figure 3). LOD for 2-MCPD esters and 3-MCPD esters is 0.01 mg kg^{-1} ; whilst LOD for GE is 0.05 mg kg^{-1} . Figure 4 shows GC chromatogram obtained for the three compounds.

RAMLI, MR; SIEW, WL; IBRAHIM, NA; KUNTOM, A and RAZAK, R A A (2015). Other factors to consider in the formation of chloropropanediol fatty esters in oil processes. *Food Addit. & Contam.: Part A*. DOI: 10.1080/19440049.2015.1032368.

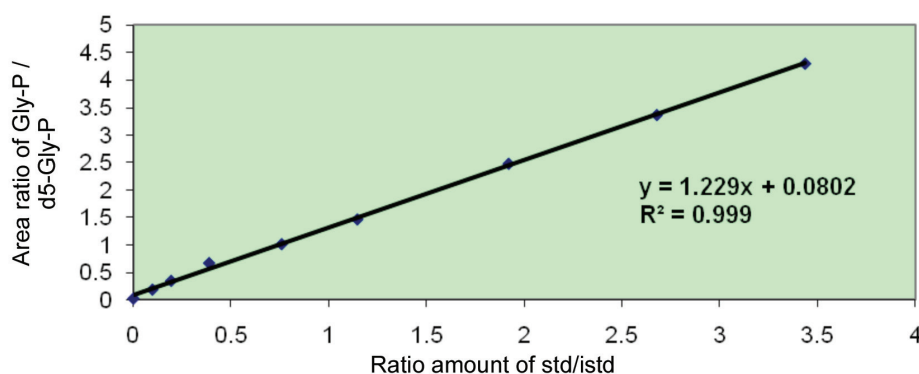


Figure 3. Example of calibration curve for GE.

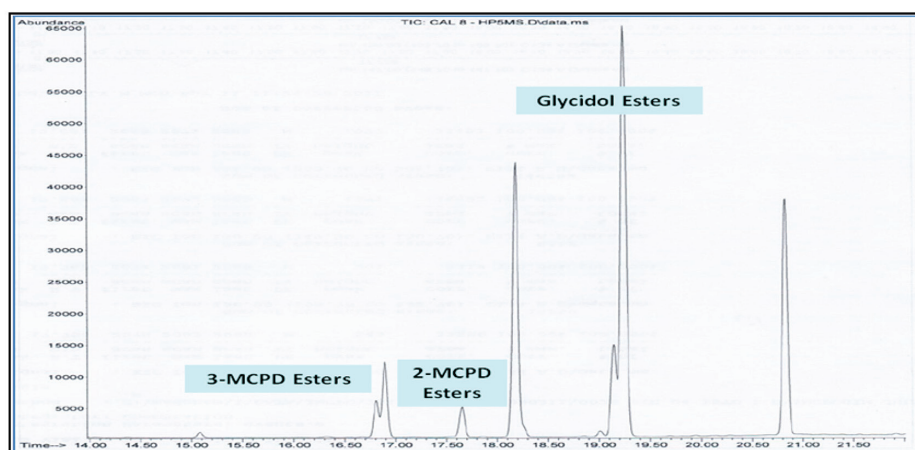


Figure 4. GC chromatogram of the three contaminants.

CONCLUSION

The GC-MS instrument can easily be maintained, hence, this method can be applied as a routine analysis.

REFERENCES

AOCS. Determination of 2- and 3-MCPD fatty acid esters and Glycidol fatty acid esters in edible oils and fats by acid transesterification. Official Method Cd 29a-13.

HAMLET, G C; ASUNCION, L; VELIŠEK, J; DOLEŽAL, M; ZELINKOVÁ, Z and CREWS, C (2011). Formation and occurrence of esters of 3-chloropropane-1, 2-diol (3-CPD) in foods: what we know and what we assume. *Eur. J. Lipid Sci. Technol.*, 113: 279-303.

MATTHÄUS, B; PUDEL, F; FEHLING, P; VOSMANN, K and FREUDENSTEIN, A (2011). Strategies for the reduction of 3-MCPD esters and related compounds in vegetable oils. *Eur. J. Lipid Sci. Technol.*, 113: 380-386.

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