

# ANALYTICAL METHOD FOR ANALYSES OF DIMETHYL SULFATE IN PALM-BASED METHYL ESTER SULFONATE

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The technology involved a method to detect low levels of dimethyl sulfate in methyl ester sulfonate using a gas chromatographic-mass spectrometer method.

## BACKGROUND

Methyl ester sulfonate (MES) is an active ingredient used in the formulation of cleaning and washing products. This active ingredient is produced through sulphonation of methyl esters. Dimethyl sulfate (DMS) (Figure 1) is a by-product which may be produced under certain processing conditions during the production of MES. To-date, there is no quantitative method for detection of DMS in palm-based MES. DMS is suspected to be a potential human carcinogen. It is a confirmed animal carcinogen.

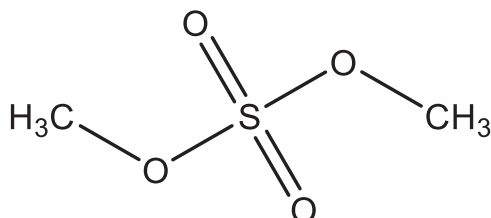


Figure 1. Chemical structure of dimethyl sulphate.

## PROBLEM STATEMENT

Currently, there is no quantitative method available to the local industry to detect and monitor the formation of DMS in commercial MES.

The service involved detection and quantifying the presence of residual DMS in palm-based MES of different carbon homologs.

## METHODOLOGY

The method involves solvent extraction of DMS from MES. The detection and quantification is carried out using a GC-Mass Spectrometer

detector (MSD). Quantification was based on an external standard calibration (Figure 2) using an authentic standard. The total ion chromatogram for DMS is shown in Figure 3. The GC-MSD instrument used for the analysis is as shown in Figure 4.

## NOVELTY OF THE SERVICE

This is the first reported method to quantify DMS at low levels in palm-based MES. Currently there is no alternative method locally and abroad providing this type of analysis.

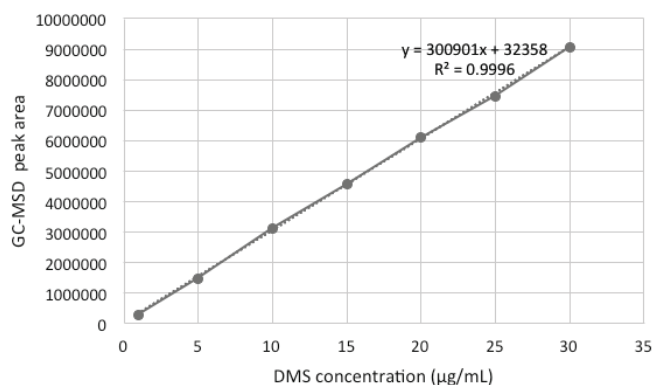


Figure 2. Example of calibration curve for DMS in hexane.

## QUALITY OF THE ANALYTICAL SERVICE

The quality of this analytical method was evaluated by method validation. It was found that the method showed good linearity as indicated by the coefficient correlation ( $R^2$ ) of 0.9996 (Figure 3) for the calibration curve. The accuracy of the method was in the range of 80%-100% which was well within the acceptable range recommended by the International Conference on Harmonisation (US FDA). The repeatability of the method was good as indicated by relative standard deviation of < 11% for both intra-day and inter-day evaluation. The limit of detection is 1 ppm.



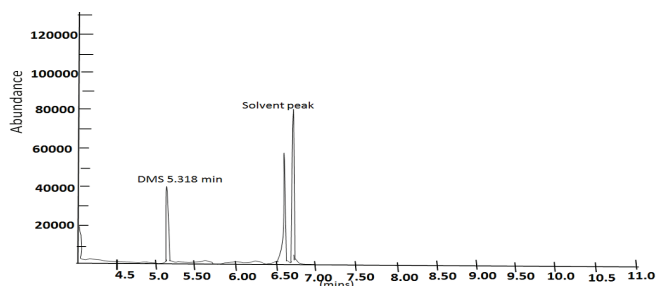


Figure 3. Total ion chromatogram of dimethyl sulphate (DMS) in hexane.



Figure 4. Gas-chromatography-mass spectrometer detector for determination of dimethyl sulfate in palm-based methyl ester sulfonate.

## BENEFITS

A simple, accurate, precise, sensitive and selective method for the detection and quantification of DMS in palm-based MES.

## SERVICES OFFERED

MPOB offers analytical services for the quantification of DMS in palm-based MES. The certificate of analysis (COA) will be issued for the analysis.

## INNOVATION

- Please contact Ms Bonnie Tay Yen Ping at email: [bonnie@mpob.gov.my](mailto:bonnie@mpob.gov.my) for request of analyses.
- The request will be registered through an online Laboratory Information Management System (LIMS) for data traceability.
- Analyses will be carried out within 10 working days.
- Certificate of analysis (COA) will be ready within additional five working days and delivered via postal mail or fax.

## COST OF ANALYSIS

### Price Recommendation:

Total cost with quantification of DMS (calibration curve and one MES sample) = RM 1650

Test without calibration curve or subsequent additional MES sample = RM 500

## REFERENCES

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