

The compound 1,4-dioxane can be formed as a by-product during the polymerisation of ethylene oxide (EO) to produce a polyethoxylate surfactant such as fatty alcohol ethoxylates (FAEO) and polysorbates, as a result of the combination and rearrangement of EO. Polyethoxylated raw materials are widely used in cosmetic and personal care (CPC) products as surfactants, emulsifiers, foaming agents and dispersants. These CPC products may contain 1,4-dioxane if the ethoxylated product containing 1,4-dioxane is used in their formulations. The 1,4-dioxane was demonstrated by studies to be toxic when it was found that laboratory animals developed cancer when exposed to 1,4-dioxane at the lowest parts per billion level over the animal's lifetime. Therefore, a reliable analytical method for detection and quantification of low levels of 1,4-dioxane in the raw materials such as FAEO is needed to ensure that there is no 1,4-dioxane present in the formulated CPC products. The six-member ring compound  $C_4H_8O_2$  (Figure 1), also named as dioxane, 1,4-diethylene dioxide, p-dioxane or diethylene ether, is a heterocyclic organic compound appearing as a clear, colourless liquid at room temperature and pressure. It is classified as an ether and readily dissolves in water and is therefore highly hygroscopic. It has a molecular weight of  $88.11 \text{ g mol}^{-1}$ , a density of  $1.033 \text{ g ml}^{-1}$ , a boiling point of  $101.1^\circ\text{C}$ , and a melting point of  $11.8^\circ\text{C}$ .

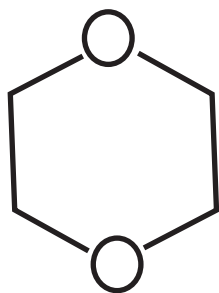


Figure 1. Structure of 1,4-dioxane.

## OBJECTIVE

To detect and quantify the presence of 1,4-dioxane in palm-based FAEO.

## METHODOLOGY

In a typical experiment, palm-based FAEO sample was accurately weighed into a 5-ml volumetric flask and HPLC grade acetonitrile is filled to the mark. This method involves injecting the sample into the GC-FID system (Figure 2) without any clean-up procedure. Quantification of 1,4-dioxane was carried out by external calibration method using a six-point curve (from  $0.5 \mu\text{g ml}^{-1}$  to  $70 \mu\text{g ml}^{-1}$ ).



Figure 2. GC-FID system for analysis of 1,4-dioxane.

## RECOVERY STUDIES

This method involved spiking/recovery work at five levels of 1,4-dioxane concentration (ranging from  $30 \mu\text{g g}^{-1}$  to  $500 \mu\text{g g}^{-1}$ ) for FAEO LS 3 (three moles EO) and FAEO LS7 (seven moles EO). Recoveries for both matrices were good, ranging between 98% to 105%, and coefficient of variation (CV) less than 5% for all levels of spiking. Figure 3 shows the representative GC-FID chromatograms

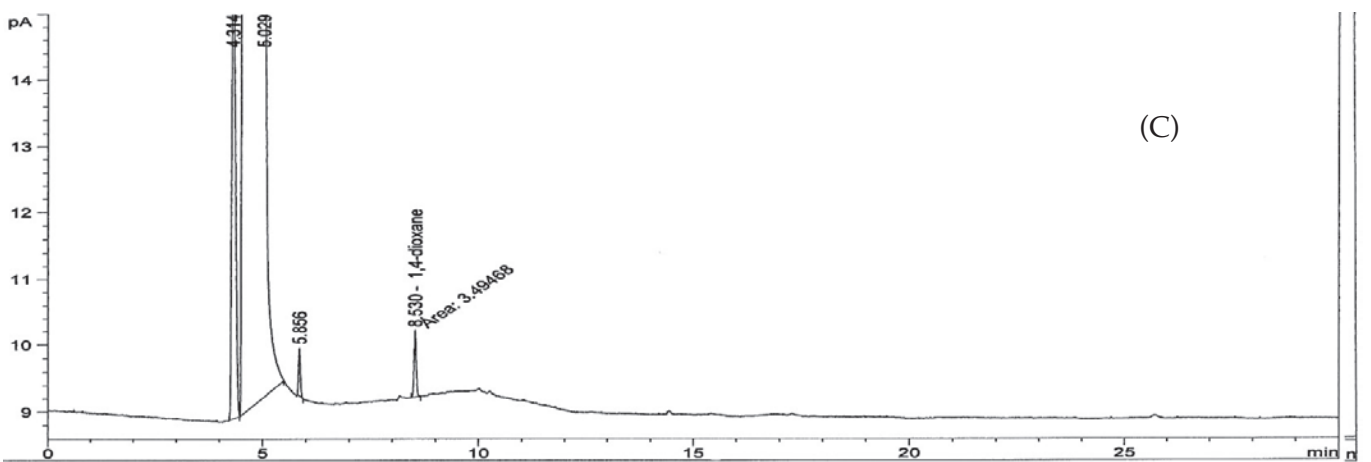
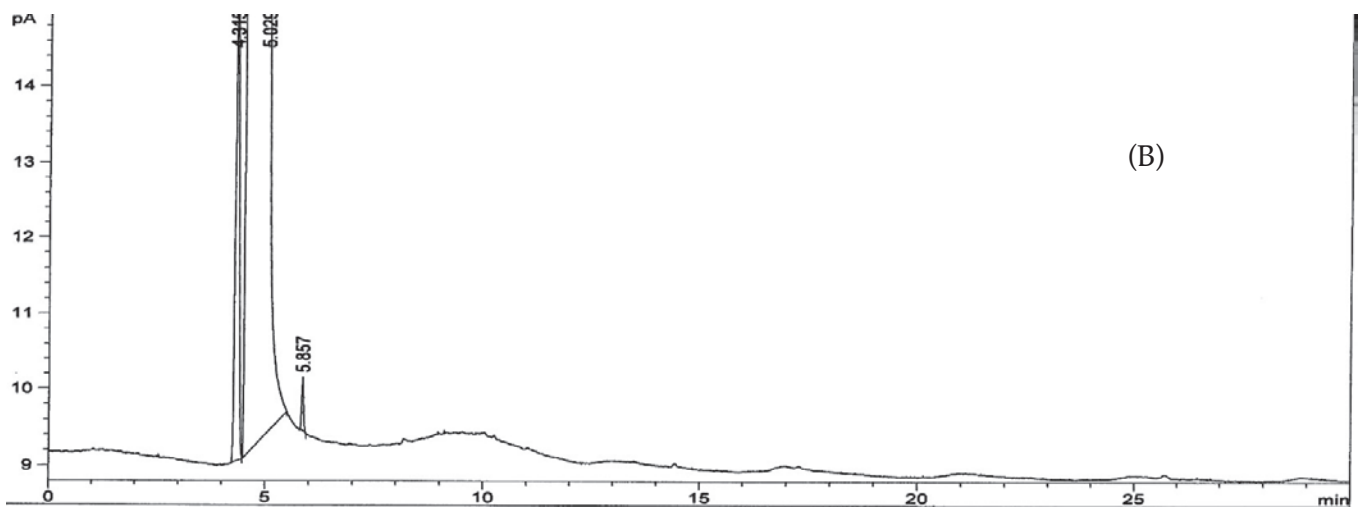
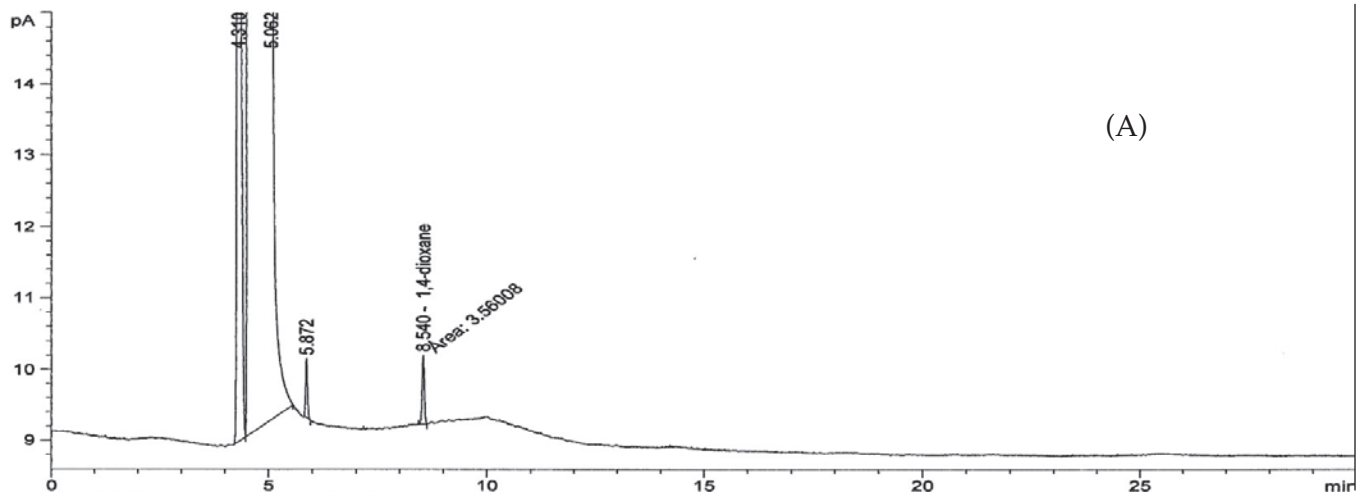


Figure 3. GC-FID chromatograms of (A) 1,4-dioxane standard ( $6 \mu\text{g ml}^{-1}$ ), (B) fatty alcohol ethoxylates (FAEO) without 1,4-dioxane (blank) and FAEO spiked with  $6 \mu\text{g ml}^{-1}$  of 1,4-dioxane.

**TABLE 1. METHOD VALIDATION PARAMETERS**

Calibration curve (0.5 – 70 µg ml <sup>-1</sup> )	Linear, coefficient of correlation of 0.999
Limit of detection	10 µg g <sup>-1</sup>
Limit of quantification	30 µg g <sup>-1</sup>
Intra-day precision	CV less than 5.3%
Inter-day precision	CV less than 5.3%
Specificity	Applicable only for fatty alcohol ethoxylates (FAEO) from 1 to 7 moles ethylene oxide (EO) or higher which has similar GC-FID profiles

of 6 µg ml<sup>-1</sup> 1,4-dioxane, blank FAEO LS3 and FAEO LS3 spiked with 6 µg ml<sup>-1</sup> of 1,4-dioxane. Table 1 shows the method validation parameters.

**BENEFIT**

An accurate, reliable and simple method for monitoring the presence of 1,4-dioxane in palm-based FAEO.

**TYPE OF SERVICE**

Quantification of 1,4-dioxane in palm-based FAEO.  
 Minimum cost per batch with one FAEO sample = RM 110.  
 For each additional FAEO sample @RM 20 (but could be subjected to revision from time to time).

**CLIENT**

Palm-based FAEO manufacturers.

**REFERENCES**

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GOSELIN, R E; HODGE, H C; SMITH, R P and GLEASON, M N (1976). *Clinical Toxicology of Commercial Products*. Williams & Wilkins, Baltimore. p. 243.

MATHESON, L; RUSSELL, G; MACARTHUR B and SHEATS, W B (2009). 1,4-Dioxane - a current topic for household detergent and personal care formulators. 100<sup>th</sup> AOCS Annual Meeting.

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](http://www.mpob.gov.my)