Analysis of Surfactant by LC-MS (Application for Cleaning Validation)

Surfactants are added to various products including detergent, drugs, cosmetics, and pesticides, typically to reduce surface tension. Due to their prevalence, many incidents of food contamination with detergents, etc. and poisoning by accidental ingestion have been reported. For quality assurance at a drug manufacturing plant[1)], the operating procedures, which describe the cleaning method, must be designed to ensure that residual amounts of drugs, detergents, etc. are below allowable limits. Typical verification methods include the swab method, final rinsing method, and visual inspection method. TOC analyzers, HPLC, or absorption spectrophotometers are used as the analytical instruments in these tests. In the report, a highly reliable LC-MS method was used to analyze a detergent, the target material, and the results are introduced here.

1) "Handling of Ministerial Ordinance on Standards for Manufacturing Control and Quality Control for Drugs and Quasi-drugs" (PFSB/CND Notification No.0830-1, August, 2013)

5610 MS Detector

LC-MS Analysis

■ Analytical Conditions

Table 1 MS Detector Settings

Ionization method	ESI
Ionization mode	Negative
Ionization voltage	2100 V
Measurement mode	Scan: (*m/z* 200-800) SIM:(*m/z* 353)

Table 2 Conditions for HPLC Analysis

Column	LaChrom II C18 (5 µm)4.6 mm I.D. x 150 mm
Column temp.	40°C
Mobile phase	0.1 % HCOOH in H_2O (v/v) / CH_3OH = 15 / 85
Flow rate	1.0 mL/min (Split ratio = 1:250)
Injection vol.	10 µL

■ Sample Preparation

Model sample 1 : Commercially available detergent*1(Prepared by diluting the detergent with water)
Model sample 2 : Rinse solution obtained after washing the container with an appropriate amount of detergent
*1Content of surfactant in the detergent used:
Sodium alkyl ether sulfate ester (AES) + fatty acid alkanol amide (DA)=16%

* As AES does not contain any UV-absorbing functional groups, the analysis by a UV detector is difficult.
* The ion derived from AES (R-O-CH_2-$CH_2O)_n$-SO_3Na (m/z 353) is monitored.
* A calibration curve was prepared by using the Model sample 1 as the standard sample and the amount of detergent detected in Model sample 2 was calculated.

■ Confirmation of Ion Detected by Scan Analysis

Model Sample 1: Commercially Available Detergent (1000 mg/L)

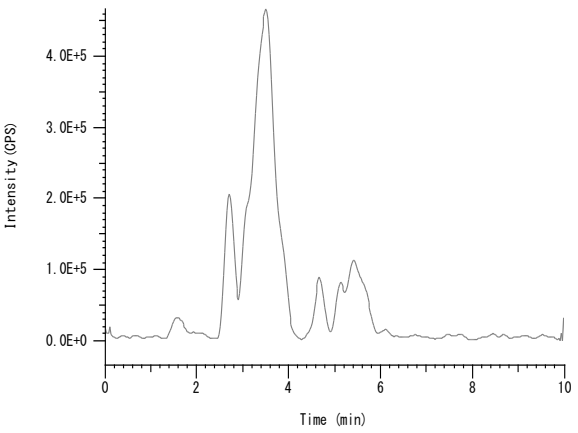

Figure 1 TIC by Scan Mode (*m/z* 200-800)

***m/z* 353** ← Monitor Target Ion

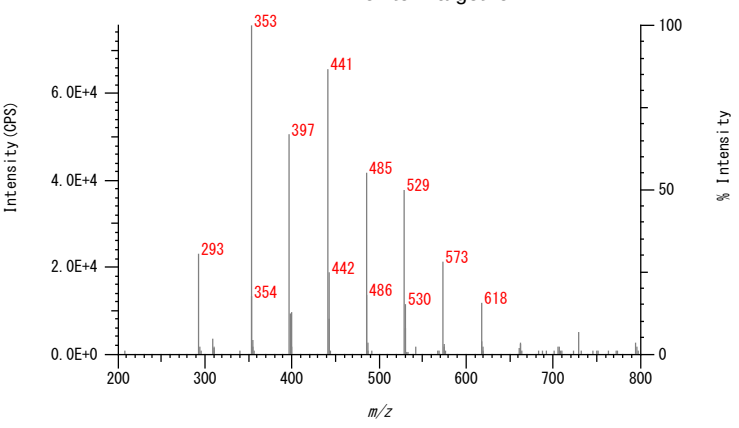

Figure 2 Mass Spectrum (3.5min)

Monitoring target ion (m/z 353) is detected from the commercially available detergent.

Confirmation of Linearity

■ **Confirmation of Linearity: SIM Mode (*m/z* 353)**

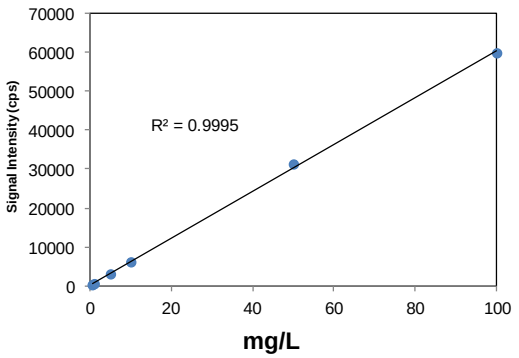

Figure 3 Calibration Curve (Amount of Detergent 0.5-100 mg/L)

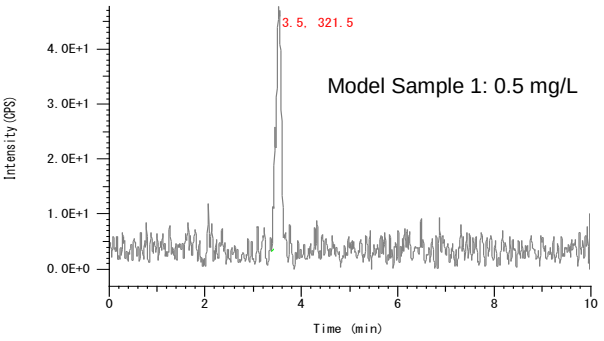

Figure 4 Chromatogram of *m/z* 353

For the Model sample 1, a good linearity with R^2=0.9995 was obtained over the concentration range of 0.5 – 100 mg/L and the lower limit of quantitation was found to be 0.5mg/L (SN=10).

Analysis Example of Rinse Solution

Model sample 2: The solution obtained from rinsing a container which had been previously washed with an appropriate amount of detergent.

1. Clean the beaker with Model sample 1
2. Rinse with tap water
3. Clean with 10 mL of water and analyze the collected rinse solution (1st rinse) ⇒ Rinse solution 1
4. Clean with 10 mL of water and analyze the collected rinse solution (2nd rinse) ⇒ Rinse solution 2

Confirmation by SIM Mode Chromatogram (*m/z* 353)

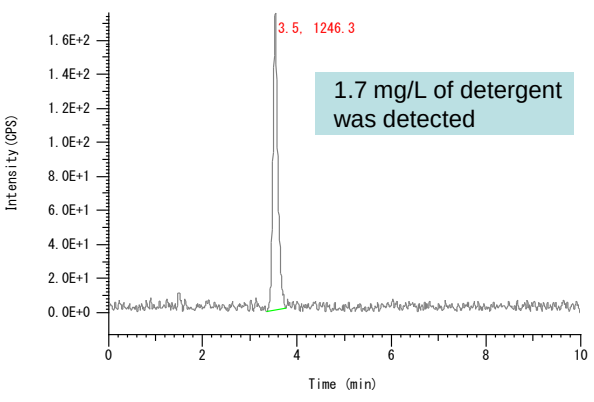

Figure 5 Chromatogram of Rinse Solution 1

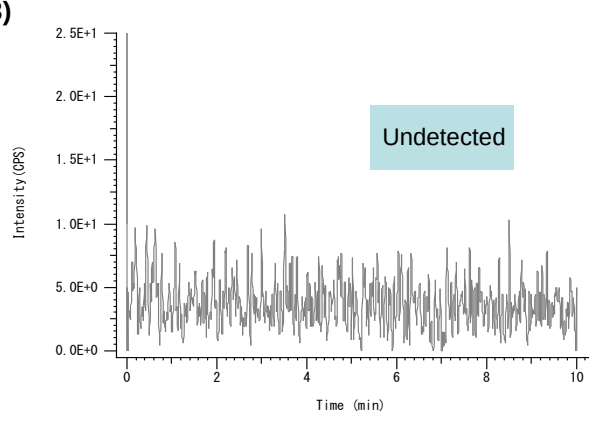

Figure 6 Chromatogram of Rinse Solution 2

Analysis of the rinse solution collected after cleaning (with Model sample 2), 1.7 mg/L of the detergent was detected from the Rinse solution 1 obtained after the 1st cleaning. In the Rinse solution 2, obtained after the 2nd cleaning, no peak was detected. As described above, by confirming the specific monitored ion, highly reliable analysis is possible.

<Main system configuration> Chromaster 5110 Pump, 5210 Autosampler, 5310 Column Oven, 5610 MS Detector

NOTE: These data are an example of measurement; the individual values cannot be guaranteed.

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