Examination of a quicker method to analyze anionic surfactants through high performance liquid chromatography

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1. Introduction

Linear Alkylbenzene Sulfonate (LAS) is an anionic surfactant that is used in synthetic laundry detergents, and is a water quality criterion for water supply. Our bureau conducts analysis of LAS (five substances) every month for tap water and four times a year for raw and treated water at our purification plants.

According to evaluation methods for water quality standards stipulated by the central government (“prescribed evaluation methods”), the method to be taken for LAS analysis is solid-phase extraction and high-performance liquid chromatography. It takes time to preprocess and enrich samples through solid-phase extraction, with over half a day needed to complete measurement of a sample. When considering response and optimization of operations in the event of an accident, an important challenge to address would be to expedite analysis methods. To that end, we conducted a study to establish a method of analysis in which the water sample is directly injected into the analyzer without preprocessing (“direct injection”).

2. Experiment Details

(1) Study to achieve reproducibility
(2) Confirmation of linearity of calibration curve
(3) Calculation of limit of quantification
(4) Assessment based on guidelines for proper assessment of water quality evaluation methods

3. Experiment Results

(1) We used an eluent and separation column as stipulated by the prescribed evaluation method and examined a method for analysis using direct injection. At first we were unable to achieve test reproducibility for area value in the measurement of a number of same concentration samples, but after exercising caution over detergent contamination and glass adsorption by washing implements to be used with methanol and pre-rinsing vials with the sample to be tested, we were able to obtain stable area values.

(2) Linearity of the calibration curve was within the range of 0.004 \cdot 0.01 \text{ mg/L}, showing good linearity with a coefficient of determination of 0.99 or higher for all five substances.
(3) The limit of quantification was 0.004 mg/L for all five substances (limit of quantification of 0.02 mg/L for the total of the five substances; one-tenth of the water quality standard).

(4) At Kanamachi Purification Plant, a standard solution was added to raw and treated water samples to achieve 0.004 mg/L, and a branching experiment was conducted once a day (two samples each of raw and treated water) for five days. For both raw and treated water we were able to satisfy our objectives for trueness (81-103%; our goal 70-120%), repeatability (4-17%; our goal 20% or less) and intermediate precision (11 - 24%; our goal under 30%).

From the above results we were able to establish an analysis method using direct injection, which makes quick measurement possible by greatly reducing the analysis time from sample separation to conclusion of measurement from over half a day to within 10 minutes. By omitting the need for preprocessing, we were also able to reduce the amount of organic solvents and solid-phase extraction columns used, and control costs related to analysis.