SHIMADZU APPLICATION NEWS

LA190-E009

● HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

No.L282

HPLC Analysis of Isocyanuric Acid and LAS Anionic Surfactant by the JWWA* Method (* Japan Water Works Association)

The mains water supply is essential to maintain safe and comfortable life. The JWWA method prescribes analysis methods for chemical components in tap water. The 2001 revision of the JWWA Test Method added isocyanuric acid as a controlled substance. It is normally used as a disinfectant in swimming pools, but is also used to sterilize drinking waters in overseas refugee camps.

Surface-active agents act to lower surface tension, and offer many other unique properties, including permeability, wettability, emulsifiability, foamability, dispersibility, and suspensibility. In addition to the domestic environment, surface-active agents are employed in a wide range of industrial fields, including textiles, paper, metals, cosmetics, pharmaceuticals, foods, and civil engineering. Anionic surfactants have been widely distributed throughout the water environment via household effluent discharged into rivers, either directly or via a sewage treatment plant. Anionic surfactants exist as various types, including linear alkylbenzene sulfonates (LAS), alkylether sulfates (AES), α-olefin sulfonates (AOS), and alkyl sulfates (AS). The commonest types used today are LAS. The JWWA Test Method previously prescribed LAS analysis by HPLC.

This Application News introduces examples of the analysis of isocyanuric acid and LAS under the analytical conditions prescribed in the 2001 revision of the JWWA Test Method.

■ Analysis of Isocyanuric Acid

Fig. 1 shows the analysis result for an isocyanuric acid standard. A 10mg/L sample was prepared from sodium dichloroisocyanate according to the JWWA Test Method, and 20μL of sample was injected. This sample must be prepared each time it is used, as isocyanuric acid is unstable in water and readily decomposes.

Fig. 2 shows the UV spectrum of isocyanuric acid detected by a photodiode array detector. As isocyanuric acid absorbs UV in the low-wavelength range only, careful column selection is required to ensure sufficient retention time to separate impurities in the sample.

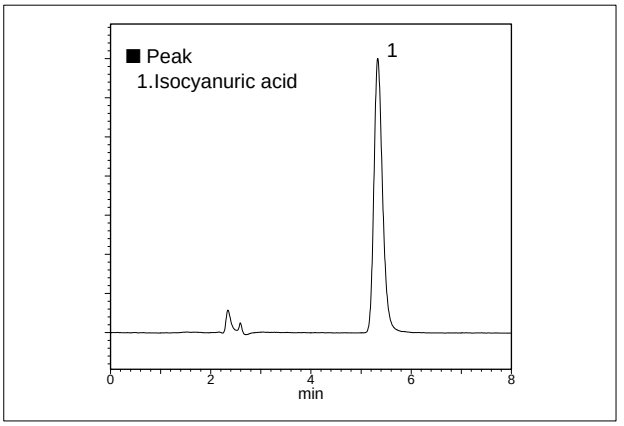

Fig. 1 Chromatogram of Isocyanuric Acid (10μg/mL, 20μL injected)

Table 1 Analytical Conditions

Column	:Shodex RSpak DE-613 (150mmL.×6.0 mm i.d.)
Mobile Phase	:10mM (Sodium) phosphate buffer (pH 7)
Flow Rate	:0.8mL/min
Temperature	:40˚C
Detection	:SPD-10AV*VP* at 220nm SPD-M10A*VP* 200~360nm

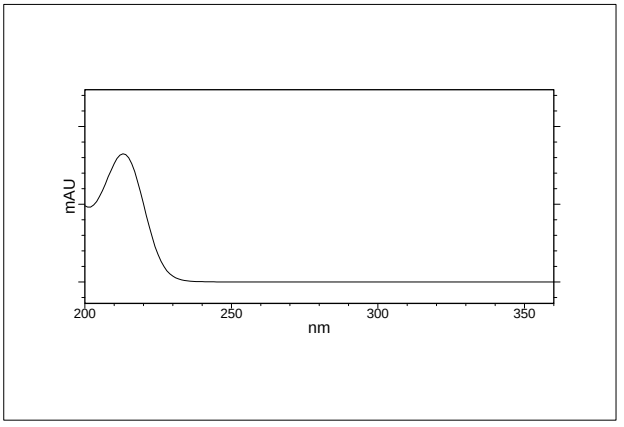

Fig. 2 UV spectrum of Isocyanuric Acid

■ Repeatability of the Isocyanuric Acid Analysis

Table 2 shows the results of five measurements on a 500µg/L sample of isocyanuric acid. This concentration is equivalent to a 1µg/L concentration in a raw water sample after 500x concentration during pretreatment.

Table 2 Repeatability

Isocyanuric acid	Retention time(min)	Peak area
1	5.330	14731
2	5.328	14677
3	5.330	15161
4	5.333	14969
5	5.329	14985
Average	5.330	14905
Standard deviation	0.0019	199.0
RSD(%)	0.036	1.34

(500µg/L, 20µL)

■ Analysis of LAS

The HPLC analysis of LAS is to be conducted with the UV or fluorescence detector. Fig. 3 shows examples of these analyses. The upper chromatogram shows the results using the fluorescence detector and the lower chromatogram the results with the UV detector. Table 3 shows the analytical conditions. The LAS standard contained components with different numbers of carbon atoms.

Fig. 4 shows the analysis results for a domestic synthetic detergent. A 500mg/L sample of the detergent was dissolved in water and 20µL of this sample was injected. The results indicate that the various isomers were detected for $C_{10\text{-}13}$.

Table 3 Analytical Conditions

Column	:Shim-pack VP-ODS (250mmL. ×4.6mm i.d.)
Mobile Phase	:0.1M Sodium Perchlorate/Acetonitrile = 35/65
Flow Rate	:1.0mL/min
Temperature	:40˚C
Detection	:RF-10AXL Ex at 221nm, Em at 284nm SPD-10AVP at 225nm

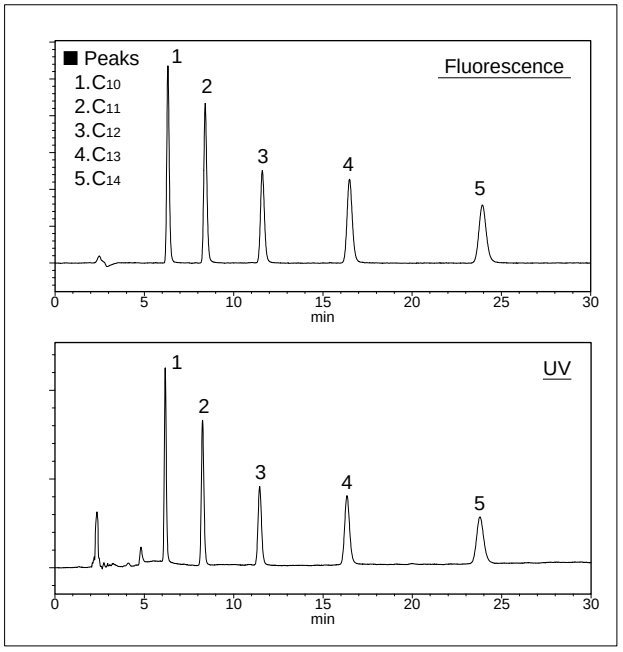

Fig. 3 Chromatograms of LAS (2mg/L, 20µL injected)
Upper: RF-10AXL; Ex at 221 nm, Em at 284 nm
Lower: SPD-10AVP at 225 nm

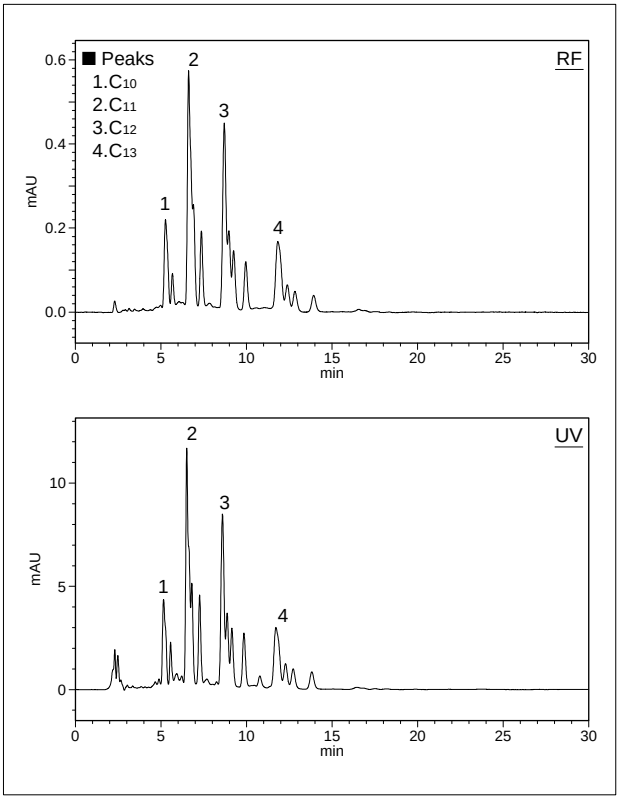

Fig. 4 Chromatograms of Synthetic Detergent (500mg/L, 20µL injected)
Upper: RF-10AXL; Ex at 221 nm, Em at 284 nm
Lower: SPD-10AVP at 225 nm

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SHIMADZU CORPORATION. International Marketing Division

3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax. 81(3)3219-5710
Cable Add.:SHIMADZU TOKYO

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