



Anionic Surfactants by Continuous Flow Analysis (CFA)

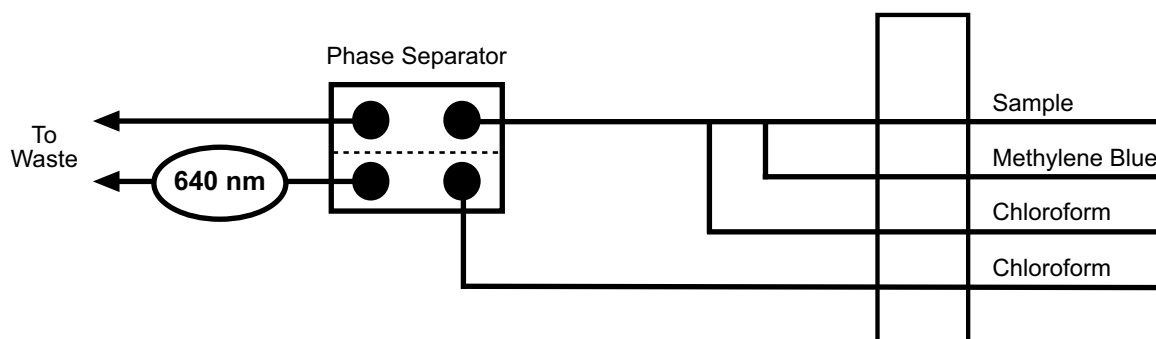
(Cartridge Part #A002705)

1.0 Scope and Application

- 1.1 This method is used for the determination of anionic surfactants in fresh water and wastewaters.
- 1.2 The Method Detection Limit (MDL) of this method is 0.005 mg/L for sodium dodecyl sulfate (SDS). The applicable range for these surfactant types is 0.03–5.0 mg/L as methylene blue active substances (MBAS), based on sodium dodecyl sulfate and/or 1-dodecanesulfonic acid. The range may be extended to analyze higher concentrations by sample dilution.

2.0 Summary of Method

- 2.1 The anionic surfactants most often found in wastewater are soluble sodium salts of the alkyl sulfates and the alkyl benzene sulfonates. When such anionic surfactants are mixed with the water soluble cationic dye, methylene blue, an ion pair is formed. In mixtures of chloroform and water, the ion pairs will extract into the chloroform layer, transferring the blue color into the organic phase. The amount of anionic surfactants in the sample can be found by comparing a standard absorbance versus concentration curve at 640 nm, the absorbance maximum of methylene blue in chloroform.
- 2.2 The quality of the analysis is assured through reproducible calibration and testing of the Continuous Flow Analysis (CFA) system.
- 2.3 A general flow diagram of the CFA system is shown below (see Section 17.0 for a detailed flow diagram).



3.0 Definitions

Definitions for terms used in this method are provided in Section 16.0, "Glossary of Definitions and Purposes."

4.0 Interferences

- 4.1 Methylene blue solutions must be protected from oxidizing substances.
- 4.2 The reaction of methylene blue dye with anionic surfactants is not stoichiometric, and the color sensitivity of the dye is different for each class of surfactant. For the most accurate results, a calibration curve of absorbance versus concentration covering the complete analytical range should be made for each class of anionic surfactant to be measured.
- 4.3 The presence of heavy metals or cationic surfactants can remove some of the anionic surfactants from solution.
- 4.4 High chloride concentrations can interfere with the solubility of methylene blue.
- 4.5 Anions interfere by forming ion pairs with methylene blue. Wastewater samples typically contain chlorine (Cl^-) and nitrate (NO_3^-), and their effect can be measured according to Burkhardt (Reference 15.1).

5.0 Safety

- 5.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been fully established. Each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level.
- 5.2 For reference purposes, a file of Material Safety Data Sheets (MSDS) for each chemical used in this method should be available to all personnel involved in this chemical analysis. The preparation of a formal safety plan is also advisable.
- 5.3 The following chemicals used in this method may be highly toxic or hazardous and should be handled with extreme caution at all times. Consult the appropriate MSDS before handling.
 - 5.3.1 Chloroform, spectral grade or the equivalent, CHCl_3 (FW 119.38)
 - 5.3.2 1-Dodecanesulfonic Acid, Sodium Salt, $\text{C}_{12}\text{H}_{25}\text{SO}_3\text{Na}$ (FW 272.38)
 - 5.3.3 Methylene Blue Chloride Trihydrate, $\text{C}_{16}\text{ClH}_{18}\text{N}_3\text{S}\cdot 3\text{H}_2\text{O}$ (FW 373.90)
 - 5.3.4 Sodium Dodecyl Sulfate, purity 99.0%, $\text{C}_{12}\text{H}_{25}\text{OSO}_3\text{Na}$ (FW 288.38)
 - 5.3.5 Sodium Phosphate Monobasic Monohydrate, $\text{NaH}_2\text{PO}_4\cdot\text{H}_2\text{O}$ (FW 137.99)
 - 5.3.6 Sulfuric Acid, concentrated, H_2SO_4 (FW 98.08)

- 5.4 Unknown samples may be potentially hazardous and should be handled with extreme caution at all times.
- 5.5 Proper personal protective equipment (PPE) should be used when handling or working in the presence of chemicals.
- 5.6 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

6.0 Apparatus, Equipment, and Supplies

- 6.1 Continuous Flow Analysis (CFA) System (OI Analytical Flow Solution® IV) consisting of the following:
 - 6.1.1 Model 502 Multichannel Peristaltic Pump
 - 6.1.2 Random Access (RA) Autosampler
 - 6.1.3 Expanded Range (ER) Photometric Detector with 5-mm path length flowcell and 640-nm optical filter
 - 6.1.4 Data Acquisition System (PC or Notebook PC) with WinFLOW™ software
 - 6.1.5 Anionic Surfactants Cartridge (OI Analytical Part #A002705)
- 6.2 Sampling equipment—Sample bottle, amber glass, with polytetrafluoroethylene (PTFE)-lined cap. Clean by washing with detergent and water, rinsing with two aliquots of reagent water, and drying by baking at 110°–150°C for a minimum of one hour.
- 6.3 Standard laboratory equipment including volumetric flasks, pipettes, syringes, etc. should all be cleaned, rinsed, and dried per bottle cleaning procedure in Section 6.2.

7.0 Reagents and Calibrants

- 7.1 Raw Materials
 - 7.1.1 Chloroform, spectral grade or the equivalent, CHCl_3 (FW 119.38)
 - 7.1.2 Deionized Water (ASTM Type I or II)
 - 7.1.3 1-Dodecanesulfonic Acid, Sodium Salt, $\text{C}_{12}\text{H}_{25}\text{SO}_3\text{Na}$ (FW 272.38)
 - 7.1.4 Methylene Blue Chloride Trihydrate, $\text{C}_{16}\text{ClH}_{18}\text{N}_3\text{S}\cdot 3\text{H}_2\text{O}$ (FW 373.90)
 - 7.1.5 Sodium Dodecyl Sulfate, purity 99.0%, $\text{C}_{12}\text{H}_{25}\text{OSO}_3\text{Na}$ (FW 288.38)

7.1.6 Sodium Phosphate Monobasic Monohydrate, $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$ (FW 137.99)

7.1.7 Sulfuric Acid, concentrated, H_2SO_4 (FW 98.08)

reagent water under a slight vacuum when not in use.