

# **Methodology**



## **Bromide by Segmented Flow Analysis (SFA)**

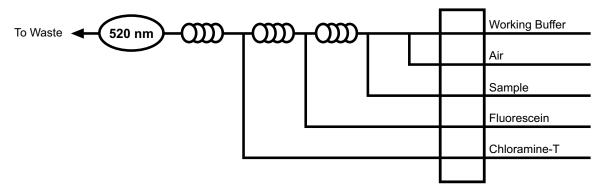
(Cartridge Part #A002706)

#### 1.0 Scope and Application

- 1.1 This method is used for the determination of bromide in drinking water, surface water, saline water, and domestic and industrial wastes.
- 1.2 The Method Detection Limit (MDL) of this method is 0.10 mg/L bromide. The applicable range of the method is 0.20–10 mg/L bromide. The range may be extended to analyze higher concentrations by sample dilution.

#### 2.0 Summary of Method

- 2.1 The sample, buffered to pH 5.6, reacts with chloramine-T trihydrate to oxidize bromide to hypobromous acid. Hypobromous acid reacts with fluorescein to form pink eosin (tetrabromofluorescein). The absorbance is measured at 520 nm (References 15.1–15.4, 15.7–15.9).
- 2.2 The quality of the analysis is assured through reproducible calibration and testing of the Segmented Flow Analysis (SFA) system.
- 2.3 A general flow diagram of the SFA 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 Iodine interferes quantitatively. In most water samples, iodine concentration is negligible. For best results, the iodine concentration can be determined separately and subtracted from the apparent bromide concentration determined by this method.
- 4.2 The presence of less than 0.50 mg/L cyanide or less than 500 mg/L chloride does not interfere. Reduce chloride interference by adding sodium thiosulfate.
- 4.3 Thiocyanate interferes linearly.

### 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 Acetic Acid, glacial, CH<sub>2</sub>COOH (FW 60.05)
  - 5.3.2 Ammonium Chloride, NH<sub>4</sub>Cl (FW 53.49)
  - 5.3.3 Chloramine-T Trihydrate, CH<sub>2</sub>C<sub>5</sub>H<sub>4</sub>SO<sub>5</sub>NNaCl•3H<sub>2</sub>O (FW 281.69)
  - 5.3.4 Fluorescein,  $C_{20}H_{10}O_5Na_2$  (FW 376.28)
  - 5.3.5 Nitric Acid, concentrated, HNO<sub>3</sub> (FW 63.01)
  - 5.3.6 Potassium Bromide, KBr (FW 119.01)
  - 5.3.7 Potassium Hydroxide, KOH (FW 56.11)
  - 5.3.8 Sodium Hydroxide, NaOH (FW 40.00)
- 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 Segmented Flow Analysis (SFA) 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 520-nm optical filter
  - 6.1.4 Data Acquisition System (PC or Notebook PC) with WinFLOW™ software
  - 6.1.5 Bromide Cartridge (Part #A002706)
- 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 Acetic Acid, glacial, CH<sub>2</sub>COOH (FW 60.05)
  - 7.1.2 Ammonium Chloride, NH<sub>4</sub>Cl (FW 53.49)
  - 7.1.3 Brij®-35, 30% w/v (Part #A21-0110-33)
  - 7.1.4 Chloramine-T Trihydrate, CH<sub>3</sub>C<sub>6</sub>H<sub>4</sub>SO<sub>2</sub>NNaCl•3H<sub>2</sub>O (FW 281.69)
  - 7.1.5 Deionized Water (ASTM Type I or II)
  - 7.1.6 Fluorescein,  $C_{20}H_{10}O_5Na_2$  (FW 376.28)
  - 7.1.7 Nitric Acid, concentrated, HNO<sub>3</sub> (FW 63.01)

- 7.1.8 Potassium Bromide, KBr (FW 119.01)
- 7.1.9 Potassium Hydroxide, KOH (FW 56.11)
- 7.1.10 Sodium Hydroxide, NaOH (FW 40.00)