



## Sulfite by Segmented Flow Analysis (SFA)

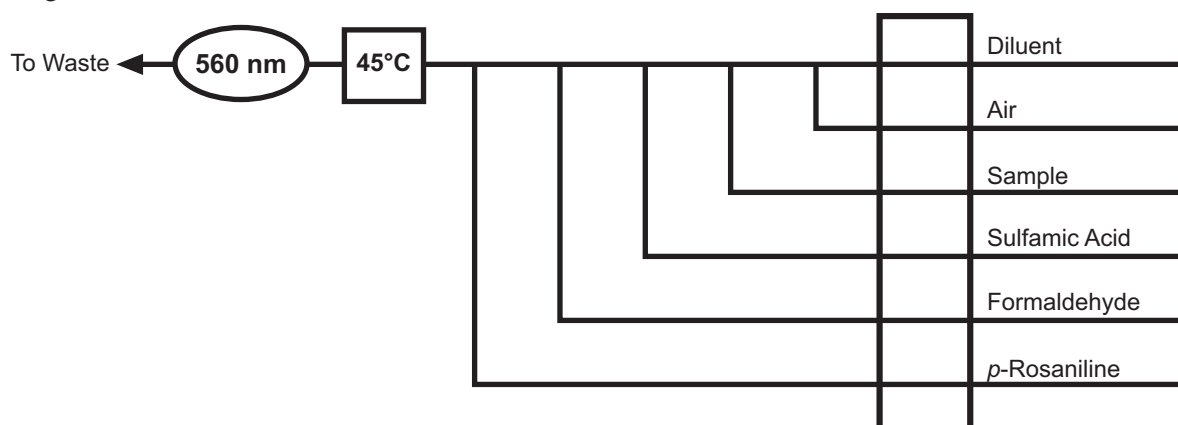
(Cartridge Part #A002725)

### 1.0 Scope and Application

- 1.1 This method is used for the determination of sulfite in drinking water, surface water, saline water, and domestic and industrial wastes.
- 1.2 The Method Detection Limit (MDL) of this method is 0.052 mg/L sulfite ( $\text{SO}_3^{2-}$ ). The applicable range of the method is 0.20–30 mg/L sulfite. The range may be extended to analyze higher concentrations by sample dilution.

### 2.0 Summary of Method

- 2.1 Sulfite reacts with acidified *p*-rosaniline hydrochloride to form a *p*-rosaniline methylsulfonic acid complex. The absorbance of this violet colored product is measured at 560 nm (References 15.1 and 15.4).
- 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 Nitrite concentrations of 2 mg/L or greater will interfere. Add sulfamic acid to remove this interference.

### 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 Ethylenediaminetetraacetic Acid, Disodium Salt Dihydrate (EDTA),  $C_{10}H_{16}N_2Na_2O_8 \cdot 2H_2O$  (FW 372.24)
- 5.3.2 Formaldehyde, 37% w/v,  $HCHO$  (FW 30.03)
- 5.3.3 Methanol,  $CH_3OH$  (FW 32.04)
- 5.3.4 Phosphoric Acid, concentrated,  $H_3PO_4$  (FW 98.00)
- 5.3.5 *p*-Rosaniline Hydrochloride,  $C_{19}H_{17}N_3 \cdot HCl$  (FW 323.83)
- 5.3.6 Sodium Metabisulfite,  $Na_2S_2O_5$  (FW 190.10)
- 5.3.7 Sulfamic Acid,  $H_2NSO_3$  (FW 97.09)
- 5.3.8 Triton® X-100,  $4-(C_8H_{17})C_6H_4(OCH_2CH_2)_nOH$ ,  $n \sim 10$
- 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 560-nm optical filter
  - 6.1.4 Data Acquisition System (PC or Notebook PC) with WinFLOW™ software
  - 6.1.5 Sulfite Cartridge (Part #A002725)
- 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 Deionized Water (ASTM Type I or II)
  - 7.1.2 Ethylenediaminetetraacetic Acid, Disodium Salt Dihydrate (EDTA),  $C_{10}H_{16}N_2Na_2O_8 \cdot 2H_2O$  (FW 372.24)
  - 7.1.3 Formaldehyde, 37% w/v, HCHO (FW 30.03)
  - 7.1.4 Methanol,  $CH_3OH$  (FW 32.04)
  - 7.1.5 Phosphoric Acid, concentrated,  $H_3PO_4$  (FW 98.00)
  - 7.1.6 *p*-Rosaniline Hydrochloride,  $C_{19}H_{17}N_3 \cdot HCl$  (FW 323.83)
  - 7.1.7 Sodium Metabisulfite,  $Na_2S_2O_5$  (FW 190.10)
  - 7.1.8 Sulfamic Acid,  $H_2NSO_3$  (FW 97.09)

7.1.9 Triton X-100,  $4-(\text{C}_8\text{H}_{17})\text{C}_6\text{H}_4(\text{OCH}_2\text{CH}_2)_n\text{OH}$ ,  $n \sim 10$