Sulfide by Segmented Flow Analysis (SFA)

(Cartridge Part #A002724)

1.0 Scope and Application

1.1 This method is used for the determination of sulfide in drinking water, surface water, saline water, and domestic and industrial wastes.

1.2 The Method Detection Limit (MDL) of this method is 0.04 mg/L sulfide. The applicable range of the method is 0.20–20 mg/L sulfide. The range may be extended to analyze higher concentrations by sample dilution.

2.0 Summary of Method

2.1 Sulfide reacts with p-aminodimethylaniline (p-AMA) and ferric chloride to form methylene blue. The absorbance is measured at 660 nm (Reference 15.2). This method does not detect acid insoluble sulfides.

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 Strong reducing agents, such as thiosulfate at concentrations above 10 mg/L, inhibit color formation.

4.2 Samples with background absorbance at the analytical wavelength may interfere.

4.3 Filter or centrifuge turbid samples prior to determination.

4.4 Consult Reference 15.4 for treatment procedures for the removal of significant interferences.

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 \( p \)-Aminodimethylaniline (\( N,N \)-Dimethyl-1,3-Phenylenediamine Dihydrochloride), \( C_8H_{12}N_2 \cdot 2HCl \) (FW 209.12)

5.3.2 Cadmium Sulfate Tri(octahydrate), \( 3CdSO_4 \cdot 8H_2O \) (FW 769.51)

5.3.3 Ferric Chloride Hexahydrate, \( FeCl_3 \cdot 6H_2O \) (FW 270.30)

5.3.4 Hydrochloric Acid, concentrated, HCl (FW 36.46)

5.3.5 Potassium Biiodate, \( KH(IO_3)_2 \) (FW 389.92)

5.3.6 Potassium Iodide, KI (FW 166.01)

5.3.7 Sodium Carboxymethylcellulose, \( R_n-OCH_2COONa \)

5.3.8 Sodium Hydroxide, NaOH (FW 40.00)

5.3.9 Sodium Sulfide Nonahydrate, \( Na_2S \cdot 9H_2O \) (FW 240.18)
5.3.10 Sodium Thiosulfate Pentahydrate, Na₂S₂O₃•5H₂O (FW 158.11)

5.3.11 Starch Indicator, 0.5% solution

5.3.12 Sulfuric Acid, concentrated, H₂SO₄ (FW 98.08)

5.3.13 Zinc Acetate Dihydrate, Zn(CH₃CO₂)₂•2H₂O (FW 219.50)

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 660-nm optical filter

6.1.4 Data Acquisition System (PC or Notebook PC) with WinFLOW™ software

6.1.5 Sulfide Cartridge (Part #A002724)

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 p-Aminodimethylaniline (N,N-Dimethyl-1,3-Phenylenediamine Dihydrochloride), C₈H₁₂N₂•2HCl (FW 209.12)
7.1.2  Brij®-35, 30% w/v (Part #A21-0110-33)
7.1.3  Cadmium Sulfate Tri(octahydrate), 3CdSO₄•8H₂O (FW 769.51)
7.1.4  Deionized Water (ASTM Type I or II)
7.1.5  Ferric Chloride Hexahydrate, FeCl₃•6H₂O (FW 270.30)
7.1.6  Hydrochloric Acid, concentrated, HCl (FW 36.46)
7.1.7  Potassium Biiodate, KH(IO₃)₂ (FW 389.92)
7.1.8  Potassium Iodide, KI (FW 166.01)
7.1.9  Sodium Carboxymethylcellulose, Rₙ-OCH₂COONa
7.1.10 Sodium Hydroxide, NaOH (FW 40.00)
7.1.11 Sodium Sulfide Nonahydrate, Na₂S•9H₂O (FW 240.18)
7.1.12 Sodium Thiosulfate Pentahydrate, Na₂S₂O₃•5H₂O (FW 158.11)
7.1.13 Starch Indicator, 0.5% solution
7.1.14 Sulfuric Acid, concentrated, H₂SO₄ (FW 98.08)
7.1.15 Zinc Acetate Dihydrate, Zn(CH₃CO₂)₂•2H₂O (FW 219.50)