



Total Kjeldahl Nitrogen (TKN), USEPA by Segmented Flow Analysis (SFA)

(Cartridge Part #A002597)

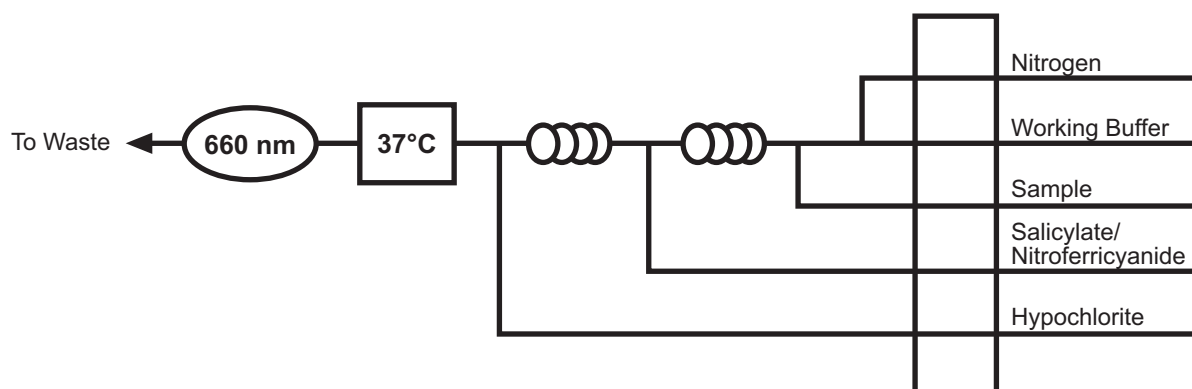
1.0 Scope and Application

- 1.1 This method is used for the determination of Total Kjeldahl Nitrogen (TKN) in drinking water, surface water, saline water, and domestic and industrial wastes according to USEPA Method 351.2 (Reference 15.4).
- 1.2 During digestion, amino acids, proteins, peptides, and other nitrogen compounds of biological origin are converted to ammonium sulfate. Nitrogenous compounds of some industrial wastes, such as amines, nitro compounds, hydrazones, oximes, semicarbazones, and some tertiary amines, may not be converted.
- 1.3 The Method Detection Limit (MDL) of this method is 0.02 mg/L nitrogen (N). The applicable range of the method is 0.10–20 mg/L nitrogen. The range may be extended to analyze higher concentrations by sample dilution.

2.0 Summary of Method

- 2.1 The sample is digested prior to analysis in the presence of sulfuric acid, potassium sulfate, and a mercury catalyst at a final temperature of 380°C. Free ammonia and organic nitrogen compounds are converted to ammonium sulfate under these conditions.
- 2.2 The ammonium reacts with salicylate and hypochlorite in a buffered alkaline solution in the presence of sodium nitroferricyanide (pH 12.8–13) to form the salicylic acid analog of indophenol blue. The blue-green color produced is measured at 660 nm (Reference 15.4).

- 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 Precipitation of calcium and magnesium hydroxides is eliminated by potassium sodium tartrate in the working buffer.
- 4.2 Filter or centrifuge turbid digestates prior to the analysis.
- 4.3 Digestates with background absorbances at the analytical wavelength may interfere with the analysis.

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 Ammonium Sulfate, $(\text{NH}_4)_2\text{SO}_4$ (FW 132.15)

5.3.2 Chloroform, CHCl_3 (FW 119.38)

- 5.3.3 Hydrochloric Acid, concentrated, HCl (FW 36.46)
- 5.3.4 Potassium Sodium Tartrate Tetrahydrate, $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$ (FW 282.23)
- 5.3.5 Potassium Sulfate, K_2SO_4 (FW 174.26)
- 5.3.6 Red Mercuric Oxide, HgO (FW 216.61)
- 5.3.7 Sodium Hydroxide, NaOH (FW 40.00)
- 5.3.8 Sodium Hypochlorite, 5.25% available chlorine (household bleach), NaOCl (FW 74.44)
- 5.3.9 Sodium Nitroferricyanide Dihydrate, $\text{Na}_2\text{Fe}(\text{CN})_5\text{NO} \cdot 2\text{H}_2\text{O}$ (FW 297.95)
- 5.3.10 Sodium Phosphate Dibasic Anhydrous, Na_2HPO_4 (FW 141.96)
- 5.3.11 Sodium Salicylate, $\text{NaC}_7\text{H}_5\text{O}_3$ (FW 160.11)
- 5.3.12 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 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 TKN, USEPA Cartridge (Part #A002597)
- 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 Ammonium Sulfate, $(\text{NH}_4)_2\text{SO}_4$ (FW 132.15)
- 7.1.2 Brij®-35, 30% w/v (Part #A21-0110-33)
- 7.1.3 Chloroform, CHCl_3 (FW 119.38)
- 7.1.4 Deionized Water (ASTM Type I or II)
- 7.1.5 Hydrochloric Acid, concentrated, HCl (FW 36.46)
- 7.1.6 Potassium Sodium Tartrate Tetrahydrate, $\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$ (FW 282.23)
- 7.1.7 Potassium Sulfate, K_2SO_4 (FW 174.26)
- 7.1.8 Red Mercuric Oxide, HgO (FW 216.61)
- 7.1.9 Sodium Hydroxide, NaOH (FW 40.00)
- 7.1.10 Sodium Hypochlorite, 5.25% available chlorine (household bleach), NaOCl (FW 74.44)
- 7.1.11 Sodium Nitroferrocyanide Dihydrate, $\text{Na}_2\text{Fe}(\text{CN})_5\text{NO} \cdot 2\text{H}_2\text{O}$ (FW 297.95)
- 7.1.12 Sodium Phosphate Dibasic Anhydrous, Na_2HPO_4 (FW 141.96)
- 7.1.13 Sodium Salicylate, $\text{NaC}_7\text{H}_5\text{O}_3$ (FW 160.11)
- 7.1.14 Sulfuric Acid, concentrated, H_2SO_4 (FW 98.08)
- 7.1.15 Teflon® or glass boiling stones