O·I·Analytical

Methodology

Hardness by Segmented Flow Analysis (SFA)

(Cartridge Part #A002711)

1.0 Scope and Application

1.1 This method is used for the determination of hardness in drinking water, surface water, and process water.

1.2 In the standard cartridge configuration, the Method Detection Limit (MDL) is 2.0 mg/L calcium carbonate ($CaCO_3$) with an applicable range of 30–400 mg/L calcium carbonate. The MDL for the low range configuration of the cartridge is 0.5 mg/L calcium carbonate with an applicable range of 5–100 mg/L calcium carbonate. The range may be extended to analyze higher concentrations by sample dilution.

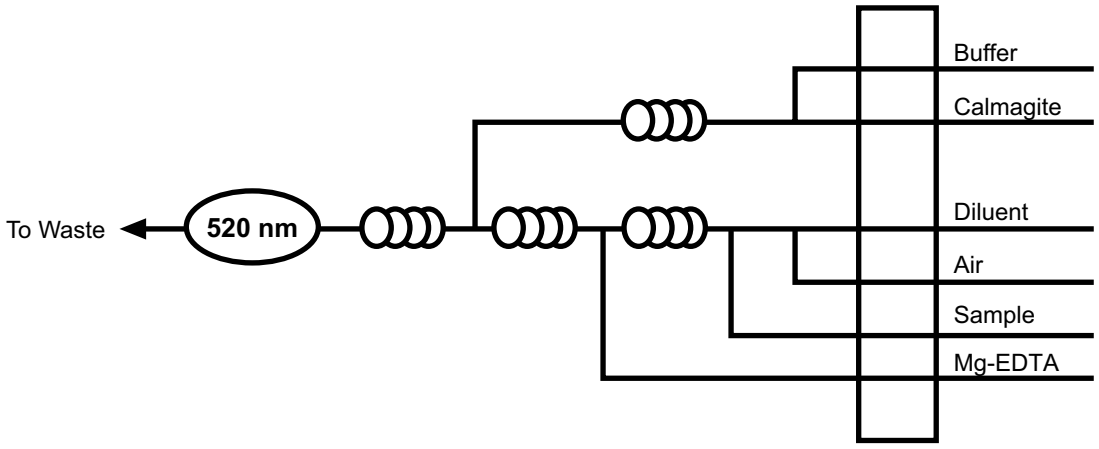
2.0 Summary of Method

2.1 The primary ions that contribute to water hardness are calcium(II) and magnesium(II). Ions of other elements such as strontium, iron, aluminum, zinc, and manganese also add to water hardness, but their concentrations are typically negligible relative to those of calcium and magnesium.

2.2 The magnesium disodium salt of ethylenedinitrilotetraacetic acid (Mg-EDTA) exchanges the magnesium for calcium on an equivalent basis. The free magnesium that is present in the sample plus the magnesium displaced from the Mg-EDTA reacts with calmagite, buffered at pH 10. This reaction produces a red-violet complex, and the absorbance is measured at 520 nm. The concentration of this complex is directly proportional to hardness. The results are expressed as weight of calcium carbonate per volume (Reference 15.2).

2.3 The quality of the analysis is assured through reproducible calibration and testing of the Segmented Flow Analysis (SFA) system.

2.4 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 There are no significant interferences. Filter turbid samples prior to determination.

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 Chloride, NH_4Cl (FW 53.50)

5.3.2 Ammonium Hydroxide, 28–30% as NH_3, NH_4OH (FW 35.05)

5.3.3 Calmagite, $C_{17}H_{14}N_2O_5S$ (FW 358.38)

5.3.4 Ethylenedinitrilotetraacetic Acid, Magnesium Disodium Salt, $(NaO_2CCH_2)_2NCH_2CH_2N(CH_2CO_2)_2Mg$ (FW 358.51)

5.3.5 Hydrochloric Acid, concentrated, HCl (FW 36.46)

5.3.6 Methyl Red Indicator Solution, 0.1% w/v

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 Hardness Cartridge (Part #A002711)

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 Chloride, NH_4Cl (FW 53.50)

7.1.2 Ammonium Hydroxide, 28–30% as NH_3, NH_4OH (FW 35.05)

7.1.3 Brij®-35, 30% w/v (Part #A21-0110-33)

7.1.4 Calmagite, $C_{17}H_{14}N_2O_5S$ (FW 358.38)

7.1.5 Deionized Water (ASTM Type I or II)

7.1.6 Ethylenedinitrilotetraacetic Acid, Magnesium Disodium Salt, $(NaO_2CCH_2)_2NCH_2CH_2N(CH_2CO_2)_2Mg$ (FW 358.51)

7.1.7 Hydrochloric Acid, concentrated, HCl (FW 36.46)

7.1.8 Methyl Red Indicator Solution, 0.1% w/v