O·I·Analytical **Methodology**

ISO 9001

Alkalinity, USEPA by Flow Injection Analysis (FIA)

(Cartridge Part #320182)

1.0 Scope and Application

1.1 This method is used for the determination of alkalinity in drinking water, surface water, saline water, and domestic and industrial wastes according to USEPA Method 310.2 (Reference 15.4).

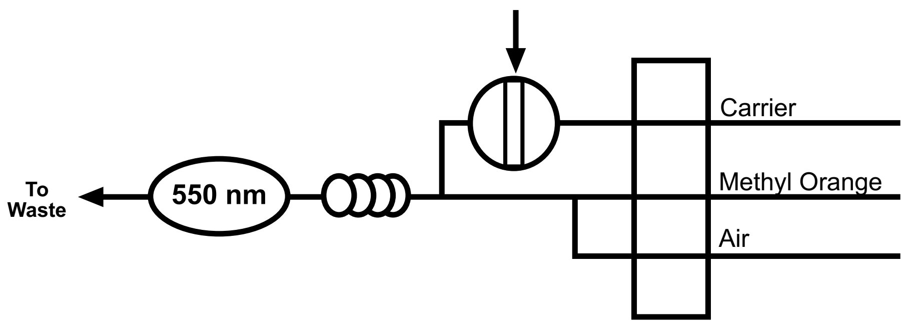
1.2 The applicable range of the method is 10–200 mg/L calcium carbonate ($CaCO_3$) using a 100-µL sample loop. Using a 50-µL sample loop, the range is 25–500 mg/L calcium carbonate. The Method Detection Limit (MDL) of this method is 2.1 mg/L calcium carbonate using the 100-µL sample loop and 3.0 mg/L calcium carbonate with the 50-µL sample loop. The range may be extended to analyze higher concentrations by sample dilution.

2.0 Summary of Method

2.1 Samples are mixed with a methyl orange indicator solution that is weakly buffered at pH 3.1. Alkalinity from carbonates, bicarbonates, and hydroxides causes the color of the indicator solution to change from red to yellow. The absorbance is measured at 550 nm, which is the wavelength of the maximum absorbance of the indicator. Since this method uses an inverse chemistry, a decrease in absorbance at this wavelength is used to measure alkalinity. This decrease is directly proportional to the alkalinity of the sample (Reference 15.4).

2.2 The quality of the analysis is assured through reproducible calibration and testing of the Flow Injection Analysis (FIA) system.

2.3 A general flow diagram of the FIA 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 Alkalinity of samples with pH values less than 3.1 cannot be determined by this method.

4.2 Residual chlorine bleaches the indicator solution and interferes with the assay. Treat samples containing chlorine with sodium thiosulfate (Section 8.5).

4.3 Color or background absorbance at 550 nm may interfere with the assay.

4.4 Filter samples prior to analysis to remove turbidity. If samples are filtered, this method is not approved for NPDES (National Pollutant Discharge Elimination System) monitoring.

4.5 Temperature variances in the laboratory will cause baseline noise and drift.

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 Hydrochloric Acid, concentrated, HCl (FW 36.46)

5.3.2 Methyl Orange, $C_{14}H_{14}N_3O_3SNa$ (FW 327.33)

5.3.3 Potassium Acid Phthalate, $C_8H_5O_4K$ (FW 204.23)

5.3.4 Sodium Carbonate, anhydrous, Na_2CO_3 (FW 105.99)

5.3.5 Sodium Hydroxide, NaOH (FW 40.00)

5.3.6 Sodium Thiosulfate, $Na_2S_2O_3$ (FW 158.11)

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 Flow Injection Analysis (FIA) 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 550-nm optical filter

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

6.1.5 Alkalinity, USEPA Cartridge (Part #320182)

6.1.6 For FIA, Flow Solution IV must be equipped with the FIA option.

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 DOWFAX® 2A1 (Part #A000080)

7.1.3 Hydrochloric Acid, concentrated, HCl (FW 36.46)

7.1.4 Methyl Orange, $C_{14}H_{14}N_3O_3SNa$ (FW 327.33)

7.1.5 Potassium Acid Phthalate, $C_8H_5O_4K$ (FW 204.23)

7.1.6 Sodium Carbonate, anhydrous, Na_2CO_3 (FW 105.99)

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