

# Alkalinity, electrometric titration, automated

Parameter and Code:  
Alkalinity, I-2030-85 (mg/L as CaCO<sub>3</sub>): 00410

## 1. Application

This method is suitable for analyzing water with any amount of alkalinity. Sample aliquots for analysis and maximum titration volume of standard acid will depend on specifications in the manufacturer's instruction manual.

## 2. Summary of method

2.1 Alkalinity is determined by titrating the water sample with a standard solution of a strong acid. The end point of the titration is selected as pH 4.5.

2.2 For additional information on the principles of the method see alkalinity (I—1030).

## 3. Interferences

See alkalinity (I-1030).

## 4. Apparatus

4.1 *Automatic titrator*, with potentiometric assembly.

4.2 *Combination electrode* (glass and reference). Separate glass and reference electrodes are also satisfactory.

## 5. Reagents

5.1 *Sodium carbonate standard solution*, 1.00 mL = 1.00 mg HCO<sub>3</sub><sup>-1</sup>: Dry 1 g primary standard Na<sub>2</sub>CO<sub>3</sub> at 150 to 160°C for 2 h. Cool in a desiccator and dissolve 0.8685 g in carbon dioxide-free water; dilute to 1,000 mL

5.2 *Sulfuric acid standard solution*, 0.01639N, 1.00 mL = 1.00 mg HCO<sub>3</sub><sup>-1</sup>: *Cautiously* add 0.5 mL concentrated H<sub>2</sub>SO<sub>4</sub> (sp gr 1.84) to 950 mL water. (The titrant is stable for several months if protected from ammonia fumes, and it is usually prepared in larger

quantities.) After the solution has been thoroughly mixed, standardize by titrating 25.00 mL Na<sub>2</sub>CO<sub>3</sub> standard solution (1.00 mL = 1.00 mg HCO<sub>3</sub><sup>-1</sup>) to pH 4.5. Adjust the concentration of the sulfuric acid standard solution to exactly 0.01639N by dilution with water or by addition of dilute acid as indicated by the first titration. Confirm the exact normality by restandardization. Although the sulfuric acid standard solution is reasonably stable, its normality should be verified at least monthly (NOTE 1).

NOTE 1. Preparing standard sulfuric acid that is not exactly 0.01639N may be more convenient. Standard sulfuric acid that is approximately 0.01639N (but the exact normality of which is known) can be used if the appropriate factor is applied in the calculations.

## 6. Procedure

6.1 Water samples for the determination of alkalinity should not be filtered, diluted, concentrated, or altered in any way. The determination should be performed without delay after the sample bottle has been opened.

6.2 Set up the automatic titrator according to the directions given in the instruction manual.

6.3 From a settled, unfiltered sample, transfer or pipet a volume of sample to the vessel specified by the manufacturer.

6.4 Set the end point to pH 4.5 following the instructions provided with the instrument.

6.5 Place the samples in position in the titration assembly and activate the automatic potentiometric titration, using 0.01639N H<sub>2</sub>SO<sub>4</sub> as titrant. Continue titrations until all samples have been titrated.

## 7. Calculations

Total alkalinity as CaCO<sub>3</sub> (mg/L)=

$$\frac{1,000}{mL_s} \times 0.8202 \times (mL_a \text{ to pH } 4.5)$$

where

mL<sub>a</sub> and mL<sub>s</sub>= volumes of standard acid and sample, respectively.

## 8. Report

Report alkalinity, total (00410), concentrations as follows: less than 1,000 mg/L, whole numbers; 1,000 mg/L and above, three significant figures.

## 9. Precision

9.1 The standard deviation for alkalinity within the range of 18.4 to 303 mg/L for 17 samples was found to be independent of concentration. The 95-percent confidence interval for the average standard deviation of 6.7 mg/L ranged from 5.8 to 7.9 mg/L.

9.2 Precision for alkalinity for five of the 17 samples expressed in terms of percent relative standard deviation is as follows:

Number of laboratories	Mean (mg/L as CaCO <sub>3</sub> )	Relative std. deviation (percent)
7	18.4	13
3	101	17
3	101	2
5	151	12
10	303	2