Australian Standard®

Waters

Part 3: Determination of alkalinity—Acidimetric titration method

This Australian Standard was prepared by Committee CH/22, Methods for Examination of Waters. It was approved on behalf of the Council of Standards Australia on 28 January 1992 and published on 16 April 1992.

The following interests are represented on Committee CH/22:

Australian Government Analytical Laboratories
Australian Institute of Marine Science
Australian Mining Industry Council
Confederation of Australian Industry
Department of Administrative Services
Department of Conservation and Environment, Vic.
Department of Health, N.S.W.
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PREFACE

This Standard was prepared by the Standards Australia Committee on Methods of Examination of Waters, as a revision of AS 2449—1981 *Waters—Determination of alkalinity—Acidimetric titration method*. The method is technically equivalent to AS 2449, with the inclusion of editorial changes, additional information on the reaction mechanism, and separation of the phenolphthalein indicator from the mixed (methyl-red and bromocresol green) indicators, to allow easier endpoint determination. The method is based on Method 2320 in Standard Methods for the Examination of Water and Wastewater, 17th ed, APHA, AWWA, WPCF, 1989.

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FOREWORD

The alkalinity of water is primarily a function of the carbonate, bicarbonate and hydroxide contents. The measured values may also include contribution from borates, phosphates or silicates, if these are present. The result obtained is dependent upon the end-point pH used and, for a particular water, this should be established by a potentiometric titration using a pH meter. The most accurate end-point is obtained from the inflection point of a titration curve. The phenolphthalein alkalinity end-point is at pH 8.3 and, for most waters, the total alkalinity end-point may be taken as occurring at pH 4.5. Colour indicators may be used for routine and control titrations in the absence of chlorine, interfering colour and turbidity and, for preliminary titrations, to select the sample size and strength of titrant.

STANDARDS AUSTRALIA

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Part 3: Determination of alkalinity—Acidimetric titration method

1 SCOPE This Standard sets out a titrimetric method for the determination of alkalinity in waters. This method is applicable to waters where the alkalinity is in the range 1 mg/L to 2500 mg/L (expressed as $CaCO_3$) under the conditions of test.

NOTE: The method is subject to interference from soaps, oily matter, suspended solids or precipitates which may coat the glass electrode of the pH meter and cause a sluggish response. These interfering substances may also cause difficulty in detecting the indicator end-point.

2 REFERENCED DOCUMENTS The following documents are referred to in this Standard:

AS
2031 Selection of containers and preservation of water samples for chemical and microbiological analysis
2031.1 Part 1: Chemical

2162 Code of practice for the use of volumetric glassware

2164 One-mark volumetric flasks

2165 Burettes and bulb burettes

2166 One-mark pipettes

3 PRINCIPLE The alkalinity in a water sample is determined by titration with a standard acid solution to a specified pH. The end-point pH is detected by using either an electrometric titrator, single indicator or mixed indicator.

4 REACTIONS

$$OH^- + H^+ \rightarrow H_2O$$
$$CO_3^{2-} + H^+ \rightarrow HCO_3^-$$
$$HCO_3^- + H^+ \rightarrow H_2O + CO_2$$

5 REAGENTS AND MATERIALS

5.1 General requirements Use only reagents of analytical reagent grade, and only distilled or deionized water which has been freed of carbon dioxide. The distilled water should be freed of carbon dioxide by boiling for 15 min and cooling to room temperature in a container fitted with a soda-lime trap, immediately prior to use.

5.2 Solutions

5.2.1 *Nitric acid (ϱ_{20} 1.42 g/mL)*

5.2.2 *Sodium thiosulfate (approximately 0.1 mol/L)* Dissolve 25 g of sodium thiosulfate pentahydrate ($Na_2S_2O_3.5H_2O$) in water and dilute to 1 L.

5.2.3 *Standard sodium carbonate solution (approximately 0.025 mol/L)* Dry 3 g to 5 g of primary standard sodium carbonate (Na_2CO_3) at 250°C for 4 h and cool in a desiccator. Weigh 2.5 ± 0.2 g to the nearest milligram, transfer to a 1 L volumetric flask, dissolve in water, and dilute to volume with water. This solution shall be maintained free of carbon dioxide, for example by use of a soda lime trap or a nitrogen atmosphere.

5.2.4 *Standard hydrochloric acid (0.1 mol/L)* Dilute hydrochloric acid (8.3 mL for acid ϱ_{20} 1.180 g/mL or 10.0 mL for acid ϱ_{20} 1.160 g/mL) to 1 L. Standardize against 50.00 mL of standard sodium carbonate solution (5.2.3) plus 50 mL of water, by titrating potentiometrically in a beaker to about pH 5.

Lift out the electrodes, rinse into the same beaker with water, cover with a cover glass and boil gently for 3 min to 5 min. Cool to room temperature. Rinse the cover glass into the beaker, and finish the titration to the pH inflection point. Calculate the concentration using the following equation:

$$\text{HCl concentration (mol/L)} = \frac{c_1 \times V_1}{53.00 \times V_2} \quad \ldots . 5(1)$$

where

c_1 = concentration of standard sodium carbonate solution (5.2.3), in grams per litre
V_1 = volume of sodium carbonate solution taken for titration, in millilitres
V_2 = volume of standard hydrochloric acid (5.2.4) used to titrate to the end-point, in millilitres.

Use the determined hydrochloric acid concentration in subsequent calculations, or adjust to exactly 0.1000 mol/L.