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BRITISH STANDARD

BS EN ISO 9963-1:1996
BS 6068-2.51: 1996

Water quality Determination of alkalinity —

Part 1: Determination of total and composite alkalinity

The European Standard EN ISO 9963-1:1995 has the status of a British Standard

ICS 13.060.40

Confirmed July 2008

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted by Technical Committee EH/3, Water quality, to Subcommittee EH/3/2, Physical, chemical and biochemical methods, upon which the following bodies were represented:

The Association of the Laboratory Supply Industry
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British Ceramic Research
British Gas plc
British Soft Drinks Association Ltd.
Chemical Industries' Association
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Swimming Pool and Allied Trades Association Ltd.
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National foreword

This British Standard has been prepared by Subcommittee EH/3/2 and is the English language version of EN ISO 9963-1:1995 *Water quality — Determination of alkalinity — Part 1: Determination of total and composite alkalinity* published by the European Committe for Standardization (CEN). It is identical with ISO 9963-1:1994, published by the International Organization for Standardization (ISO).

Cross-references

Publication referred to	Corresponding British Standard
ISO 3696:1987	BS 3978:1987 *Specification for water for laboratory use* BS EN 25667:1993 *Water quality. Sampling*
EN 25667-1:1993 (ISO 5667-1:1980)	BS EN 25667-1:1994 *Guidance on the design of sampling programmes*
EN 25667-2:1993 (ISO 5667-2:1991)	BS EN 25667-2:1993 *Guidance on sampling techniques*
	BS 6068 *Water quality* Part 1 *Glossary*
ISO 6107-2:1989	Section 1.2:1990 *Additional terms relating to types of water, and treatment and storage of water and waste water, and terms used in sampling and analysis of water*
IEC 746-2:1982	BS 6438 *Electrochemical analyzers* Part 2:1984 *Method for specifying performance of pH value analyzers*

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Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 8, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 9963-1

December 1995

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Descriptors: Water, quality, water tests, chemical analysis, determination, alkalinity, volumetric analysis

English version

Water quality — Determination of alkalinity — Part 1: Determination of total and composite alkalinity

(ISO 9963-1:1994)

Qualité de l'eau — Détermination de l'alcalinité — Partie 1: Détermination de l'alcalinité totale et composite
(ISO 9963-1:1994)

Wasserbeschaffenheit — Bestimmung der Alkalinität — Teil 1: Bestimmung de gesamten und der zusammengesetzten Alkalinität
(ISO 9963-1:1994)

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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Foreword

The text of the International Standard from ISO/TC 147, Water quality, of the International Organization for Standardization (ISO) has been taken over as a European Standard by the Technical Committee CEN/TC 230, Water analysis.

This European Standard consists of the following parts:

EN ISO 9963-1, *Water quality — Determination of alkalinity — Part 1: Determination of total and composite alkalinity.*

EN ISO 9963-2, *Water quality — Determination of alkalinity — Part 2: Determination of carbonate alkalinity.*

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 1996, and conflicting national standards shall be withdrawn at the latest by June 1996.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard: Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland, United Kingdom.

NOTE Normative references to International Standard are listed in Annex ZA (normative).

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1 Scope

This part of ISO 9963 specifies a method for the titrimetric determination of alkalinity. It is intended for the analysis of natural and treated water, and waste water, and can be used directly for waters having an alkalinity concentration of up to 20 mmol/l. For samples containing higher concentrations of alkalinity, a smaller test portion can be used for analysis. The recommended lower limit is 0,4 mmol/l. Suspended matter in the form of carbonate may interfere with the analysis. This interference can be reduced by filtration prior to the titration.

The endpoint detection, using a pH-meter, is less prone to interferences than the use of the indicator.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 9963. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 9963 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 5667-1:1980, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes.*

ISO 5667-2:1991, *Water quality — Sampling — Part 2: Guidance on sampling techniques.*

ISO 6107-2:1989, *Water quality — Vocabulary — Part 2.*

IEC 746-2:1982, *Expression of performance of electrochemical analyzers — Part 2: pH Value.*

3 Definitions

For the purposes of this part of ISO 9963, the following definitions apply.

3.1
alkalinity (A)

the quantitative capacity of aqueous media to react with hydrogen ions [ISO 6107-2]

3.2
methyl red (methyl orange) endpoint alkalinity

an arbitrary measurement of the total alkalinity (A_T) of water obtained by titration to the methyl red (methyl orange) indicator endpoint (pH 4,5); to assess the equivalent hydrogen carbonate, carbonate and hydroxide concentration of water

3.3
phenolphthalein endpoint alkalinity; composite alkalinity (A_p)

the measurement by titration to the phenolphthalein endpoint (pH 8,3) of that portion of alkalinity arbitrarily attributed to all the hydroxyl and half the carbonate content of a water [ISO 6107-2]

NOTE 1 The alkalinity of water is primarily a function of the hydrogen carbonate, carbonate and hydroxide concentrations. Other buffering substances (X) such as ammonia, borate, phosphate, silicate and organic anions may be included in the determination.

$$A_P \approx c\left(CO_3^{2-}\right) - c(CO_2aq) + c(OH^-) - c(H^+) + c(X)$$

$$A_T \approx 2c\left(CO_3^{2-}\right) + c(HCO_3^-) + c(OH^-) - c(H^+) + c(X)$$

by definition, composite alkalinity is zero for waters which have a pH value of 8,3 or less

4 Principle

The sample is titrated with standard acid solution to fixed pH endpoint values of 8,3 and 4,5. These endpoints, which are determined visually or potentiometrically, are the selected equivalence points for the determinations of the three principal components: hydrogen carbonate, carbonate and hydroxide. The pH 8,3 endpoint approximates to the equivalent concentrations of carbonate and carbon dioxide and represents the titration of approximately all the hydroxide and half of the carbonate present. The pH 4,5 endpoint approximates the equivalence point for hydrogen ion and hydrogen carbonate and allows for the determination of the total alkalinity of the sample.

NOTE 2 The equivalent point values depend on the ionic strength as well as the concentration of total inorganic carbon and may not always be optimum at the chosen endpoints.

Whilst methyl orange and methyl red have been commonly used in the past as indicators for the determination of total alkalinity, in practice the use of different indicator systems produce slightly different results in alkalinity titrations. For alkalinity determinations in accordance with this part of ISO 9963, the correct bromocresol green-methyl red indicator solution as defined in **5.6** should be used.

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5 Reagents

Use only reagents of recognized analytical grade.

Commercially available, ready-made solutions may be used.

5.1 *Water,* grade 2 in accordance with ISO 3696, free of interfering concentrations of acid or alkali and with a conductivity of less than 0,1 mS/m.

5.2 *Sodium carbonate,* standard solution, $c(Na_2CO_3) \approx 0{,}025$ mol/l.

Dry 3 g to 5 g of sodium carbonate (Na_2CO_3) at 250 °C ± 10 °C for 4 h. Allow to cool in a desiccator. Dissolve 2,65 g ± 0,20 g (m, weighed to the nearest 0,001 g) in water and dilute in a volumetric flask to 1 000 ml.

This solution is stable for at least one month if stored in a refrigerator at 4 °C and 8 °C.

5.3 *Hydrochloric acid,* c(HCl) ≈ 0,10 mol/l. Dilute 8,6 ml ± 0,1 ml of hydrochloric acid (1,16 g/ml) to 1 000 ml with water. Standardize this solution as follows, using either potentiometric (**5.3.1**) or visual endpoint detection (**5.3.2**).

5.3.1 *Potentiometric detection*

Pipette 25,0 ml ± 01 ml (V_1) of the sodium carbonate solution (**5.2**) into a titration vessel and add 75 ml ± 5 ml of water (**5.1**). Place the vessel on a magnetic stirrer and dip into the solution a plastics-coated magnetic stirrer bar and electrodes previously connected to a calibrated pH-meter. Start the stirrer and stir at a rate at which a vortex is just not perceptible. Titrate with 0,10 mol/l hydrochloric acid solution (**5.3**) until the meter reads pH 4,5 ± 0,05. Note the volume V_2, in millilitres, of acid consumed.

5.3.2 *Visual endpoint detection*

Pipette 25,0 ml ± 0,1 ml (V_1) of approximately 0,025 mol/l sodium carbonate solution (**5.2**) into a 250 ml Erlenmeyer flask, and add 75 ml ± 5 ml of water and 0,1 ml ± 0,02 ml of bromocresol green-methyl red indicator solution (**5.6**). Titrate with 0,10 mol/l hydrochloric acid solution (**5.3**) until the greenish-blue colour disappears. Note the volume V_2, in millilitres, of acid consumed.

5.3.3 *Blank determination*

Using 100 ml ± 5 ml of water, carry out a blank determination according to the appropriate procedure (**5.3.1** or **5.3.2**) and note the volume V_3, in millilitres, of acid consumed.

5.3.4 *Calculation of the amount-of-substance concentration of the hydrochloric acid*

$$c(\mathrm{HCl},1) = \frac{mV_1}{53{,}00(V_2 - V_3)}$$

where

c(HCl,1) is the actual concentration, expressed in moles per litre, of the nominally 0,10 mol/l hydrochloric acid solution (**5.3**);

m is the mass, in grams, of sodium carbonate taken for the preparation of standard solution (**5.2**);

V_1 is the volume, in millilitres, of sodium carbonate standard solution (**5.2**) taken for titration (normally 25 ml);

V_2 is the volume, in millilitres, of hydrochloric acid solution (**5.3**) consumed in the titration of the sodium carbonate standard solution (**5.2**);

V_3 is the volume, in millilitres, of hydrochloric acid solution (**5.3**) consumed in the blank titration.

Standardize this solution at least weekly.

5.4 *Hydrochloric acid*, c(HCl) ≈ 0,02 mol/l.

Pipette 100 ml ± 1 ml of hydrochloric acid solution (**5.3**) into a 500 ml volumetric flask. Dilute to volume with water and mix well. Prepare freshly before use.

Calculate the amount-of-substance concentration as follows:

$$c(\mathrm{HCl},2) = 0{,}2 \times c(\mathrm{HCl},1)$$

where

c(HCl,1) is the actual concentration, expressed in moles per litre, of the nominally 0,10 mol/l hydrochloric acid solution (**5.3**);

c(HCl,2) is the actual concentration, expressed in moles per litre, of the nominally 0,02 mol/l hydrochloric acid solution (**5.4**).

5.5 *Phenolphthalein indicator solution*

Dissolve 1,0 g ± 0,1 g of phenolphthalein in 100 ml ± 2 ml of ethanol [> 90 % (*V*/*V*) ethanol] and dilute with water to 200 ml ± 4 ml. Mix well.

5.6 *Bromocresol green-methyl red indicator solution*

Dissolve 0,200 g ± 0,005 g of bromocresol green and 0,015 g ± 0,002 g of methyl red in 100 ml ± 4 ml of ethanol [> 90 % (*V*/*V*) ethanol]. Store in an amber glass bottle.

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5.7 *Sodium thiosulfate solution,* $c(Na_2S_2O_3.5H_2O) \approx 0{,}1$ mol/l.

Dissolve 2,5 g ± 0,2 g of sodium thiosulfate pentahydrate ($Na_2S_2O_3.5H_2O$) in 100 ml ± 5 ml of water. Store in an amber glass bottle in the refrigerator for a maximum of six months.

6 Apparatus

Usual laboratory equipment and, in particular, the following should be used.

6.1 *Magnetic stirrer,* and plastics-coated stirring bar.

6.2 *pH-meter,* with a compatible electrode system suitable for the measurement of pH to within ± 0,05 pH units over the range 4 to 10, and a suitable titration vessel designed so that contact with air is minimized. The equipment shall be set up, calibrated (preferably using buffers with pH values 4, 7 and 9), and used according to IEC 746-2.

6.3 *Precision burette,* of capacity 10 ml, graduated in divisions of 0,02 ml, and conforming to the requirements of ISO 385-1.

7 Sampling and sample treatment

Collect samples in clean polyethylene or borosilicate glass bottles with a volume of at least 100 ml. Fill the bottle completely with the sample and insert the stopper so that no air remains inside the bottle. Ideally analyse the samples immediately after collection. If this is not possible, store in a cool place. In order to avoid nitrification or scaling. Many types of samples are little affected during storage. Test for any effects of storage on the type of samples analysed. (See ISO 5667-1 and ISO 5667-2.)

8 Procedure

Strongly coloured or turbid samples should be analysed by the potentiometric method.

8.1 Potentiometric method

Calibrate the pH-meter according to **6.2**.

NOTE 3 The potentiometric titration is free from interference caused by oxidizing agents, although difficulties in endpoint detection may be experienced in the presence of organic substances. Soaps, oily substances, etc. may coat the glass electrode and cause a sluggish response. Additional time should be allowed between titrant additions to let the electrode come to equilibrium, and the electrodes should be cleaned frequently.

8.1.1 *Determination of composite alkalinity titratable to pH 8,3 (phenolphthalein alkalinity)*

NOTE 4 Absorption of atmospheric carbon dioxide during the titration of composite alkalinity can lower the results.

Pipette 100 ml ± 1 ml of sample (volume V_4) into the titration vessel. Place the vessel on a magnetic stirrer and dip a plastics-coated stirring bar and pH-electrodes into the solution. Start the stirrer motor and stir at a rate at which a vortex is just not perceptible. Measure the pH value of the sample and, if it is found to be 8,3 or less, record the composite alkalinity titratable to pH 8,3 as zero. If the alkalinity is in the range 4 mmol/l to 20 mmol/l, use 0,1 mol/l hydrochloric acid (**5.3**). If the alkalinity is in the range 0,4 mmol/l to 4 mmol/l, use 0,02 mol/l hydrochloric acid (**5.4**). Titrate the sample with the appropriate acid and note the volume V_5, in millilitres, of acid consumed.

Retain the solution for use in the determination of the total alkalinity.

8.1.2 *Determination of total alkalinity*

Continue to titrate the solution reserved from the determination of composite alkalinity titratable to pH 8,3 (see **8.1.1**) with the appropriate hydrochloric acid solution until the meter reads pH 4,5 ± 0,05 (in the vicinity of pH 4,5, add the titrant drop by drop and allow at least 30 s for the electrodes to attain equilibrium with the solution). Note the total volume V_6, in millilitres, of acid required.

8.2 Visual method

NOTE 5 Remove any free chlorine present by adding 0,1 ml of sodium thiosulfate solution (**5.7**) per 200 ml of sample. This procedure removes up to 1,8 mg/l of chlorine.

8.2.1 *Determination of composite alkalinity titratable to pH 8,3 (phenolphthalein alkalinity)*

NOTE 6 Absorption of atmospheric carbon dioxide during the titration of composite alkalinity can lower the results.

Pipette 100 ml ± 1 ml of sample (volume V_4) into a 250 ml Erlenmeyer flask and add 0,1 ml ± 0,02 ml of phenolphthalein indicator solution (**5.5**). If a pink colour is not obtained, regard the composite alkalinity titratable to pH 8,3 as zero. Titrate pink-coloured samples with acid until the pink colour disappears. If the alkalinity is in the range 4 mmol/l to 20 mmol/l, use 0,1 mol/l hydrochloric acid (**5.3**). If the alkalinity is in the range 0,4 mmol/l to 4 mmol/l, use 0,02 mol/l hydrochloric acid (**5.4**). Note the volume V_5, in millilitres, of acid consumed.

Retain the solution for use in the determination of the total alkalinity.

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8.2.2 *Determination of total alkalinity*

Add 0,1 ml ± 0,02 ml of bromocresol green-methyl red indicator solution (**5.6**) to the solution reserved from the determination of composite alkalinity titratable to pH 8,3 (see **8.2.1**). Continue to titrate with the appropriate hydrochloric acid solution until the colour changes from greenish-blue to grey. Note the total volume V_6, in millilitres, of acid consumed.

9 Expression of results

9.1 Calculation

9.1.1 *Composite alkalinity titratable to pH 8,3 (phenolphthalein alkalinity)*

$$A_P = \frac{c(\mathrm{HCl}) \times V_5 \times 1\,000}{V_4}$$

where

A_p is the capacity to react with hydrogen ions, expressed in millimoles per litre, of composite alkalinity titratable to pH 8,3;

c(HCl) is the actual concentration, expressed in moles per litre, of the hydrochloric acid solution (**5.3** or **5.4**) used;

V_4 is the volume, in millilitres, of the test portion (normally 100 ml);

V_5 is the volume, in millilitres, of hydrochloric acid solution (**5.3** or **5.4**) consumed to reach pH 8,3.

9.1.2 *Total alkalinity*

$$A_T = \frac{c(\mathrm{HCl}) \times V_6 \times 1\,000}{V_4}$$

where

A_T is the capacity to react with hydrogen ions, expressed in millimoles per litre, of total alkalinity titratable to pH 4,5;

c(HCl) is the actual concentration, expressed in moles per litre, of the hydrochloric acid solution (**5.3** or **5.4**) used;

V_4 is the volume, in millilitres, of the test portion (normally 100 ml);

V_6 is the volume, in millilitres, of hydrochloric acid solution (**5.3** or **5.4**) consumed to reach pH 4,5.

10 Test report

The test report shall include the following information:

a) a reference to this part of ISO 9963;

b) precise identification of the sample;

c) the results, expressed in millinoles (H^+) per litre;

d) any departure from the procedure specified or any other circumstance that may have affected the results.

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Annex A (informative)
Information on factors for the conversion of alkalinity values to alternative units

Alkalinity values may be expressed in alternative units. Factors for the conversion from millimoles per litre are given in Table A.1.

Table A.1

Alternative unit for the expression of results	Conversion factor
mmol/l $CaCO_3$	0,50
mg/l $CaCO_3$	50
mg/l HCO_3^-	61
Parts/100 000	5,0
English degree (= 1 Clark degree)	3,50
German degree	2,80
French degree	5,0
U.S. degree	2,90

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Annex ZA (normative)
Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

IEC publication	Year	Title	EN/HD	Year
ISO 5667-1	1980	*Water quality — Sampling — Part 1: Guidance on the design of sampling programmes*	EN 25667-1	1993
ISO 5667-2	1991	*Water quality — Sampling — Part 2: Guidance on sampling techniques*	EN 25667-2	1993

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List of references

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