BRITISH STANDARD

BS 6068-2.40:
1991
ISO 9390:1990

Water quality —

Part 2: Physical, chemical and biochemical methods —

Section 2.40 Method for the determination of borate by spectrometry using azomethine-H

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Environment and Pollution Standards Policy Committee (EPC/-) to Technical Committee EPC/44, upon which the following bodies were represented:

Association of Consulting Scientists
British Association for Chemical Specialists
British Gas plc
Chemical Industries Association
Department of the Environment for Northern Ireland
Department of the Environment (Water Directorate)
Department of Trade and Industry (Laboratory of the Government Chemist)
Electricity Supply Industry in England and Wales
Industry Water Society
Institute of Petroleum
Institution of Gas Engineers
Institution of Water and Environmental Management
National Rivers Authority
Royal Institute of Public Health and Hygiene
Royal Society of Chemistry
Scottish Association of Directors of Water and Sewerage Services
Soap and Detergent Industry Association
Water Companies Association
Water Research Centre
Water Services Association of England and Wales

This British Standard, having been prepared under the direction of the Environment and Pollution Standards Policy Committee, was published under the authority of the Standards Board and comes into effect on 28 June 1991

The following BSI references relate to the work on this standard:
Committee reference EPC/44
Draft for comment 88/52170 DC

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Amendments issued since publication

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Contents

National foreword

This Section of BS 6068, which has been prepared under the direction of the Environment and Pollution Standards Policy Committee, is identical with ISO 9390:1990 "*Water quality — Determination of borate — Spectrometric method using azomethine-H*", published by the International Organization for Standardization (ISO).

The international standard was prepared by subcommittee 2, Physical, chemical and biochemical methods, of ISO Technical Committee 147, Water quality, with the active participation and approval of the UK.

NOTE The tests described in this Section of BS 6068 should only be carried out in laboratories with suitable facilities and by suitably qualified persons with an appropriate level of chemical expertise. Standard chemical procedures should be followed throughout.

BS 6068 is being published in a series of Parts subdivided into Sections that will generally correspond to particular international standards. Sections are being, or will be, published in Parts 1 to 7, which together with Part 0, are listed below.

— *Part 0: Introduction;*
— *Part 1: Glossary;*
— *Part 2: Physical, chemical and biochemical methods;*
— *Part 3: Radiological methods;*
— *Part 4: Microbiological methods;*
— *Part 5: Biological methods;*
— *Part 6: Sampling;*
— *Part 7: Precision and accuracy.*

Cross-reference

International standard	Corresponding British Standard
ISO 5667-3:1985	BS 6068 *Water quality* Section 6.3:1986 *Guidance on the preservation and handling of samples* (Identical)

Textual errors. When adopting the text of the international standard, the following textual errors were discovered. They have been marked in the text and have been reported to ISO in a proposal to amend the text of the international standard.

In **4.1**, line 4, "L + – ascorbic acid" should be read as "L (+) – ascorbic acid".

In **4.2**, at the end of line 3, insert "(produced by carefully mixing together 20 ml of sulfuric acid (ρ = 1.84 g/ml) and 80 ml of distilled water)" after "(ρ = 1.21 g/ml)". In line 6, ($C_{10}H_{14}N_2Na_2O_8 \cdot H_2O$) should be read as "($C_{10}H_{14}N_2Na_2O_8 \cdot H_2O$)".

In the title of **7.4.3**, "facteur" should be read as "factor".

In Table 1, the numerical values for "σ_r" and "σ_R" should be transposed, and in column 10 delete "mgl" and insert "mg/l".

Additional information. It is recommended that the pH value of the resulting solution from **4.2** is checked before the solution is used. In **7.2**, line 4, "per 10 mm" means per 10 mm of cell path length.

In **8.1**, definition of A_0, it is the absorbance of the blank using the same optical cell path length as for the sample. In Table 1, the standard deviations are based on the mean values.

The international standard has an introduction which is as follows.

"The natural borate content of ground water and surface water is small. The borate content of surface water can be significantly increased due to waste water discharges, because borate compounds are ingredients of domestic washing agents."

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 4, 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.

blank

1 Scope

1.1 Application range

This International Standard specifies a spectrometric method for the determination of borate in water. The method is applicable to the determination of borate in concentrations between 0,01 mg and 1 mg of boron per litre. The working range may be extended by dilution.

This method is applicable to potable water, and to ground, surface and saline waters that are not heavily polluted.

1.2 Interferences

Interferences are unlikely when analysing drinking water. Mg, Zn, Ca, Na, K, phosphate, sulfate, and nitrate are known not to interfere. Mn, Zr, Cr, Ti, Cu, V, Al, Be, and Fe may cause high results.

Interference by the presence of colouration, humic acid, and/or undissolved substances may be removed by suitable procedures (e.g. destruction of the colour, filtration through a column filled with activated carbon).

2 Normative reference

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

ISO 5667-3:1985, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*

3 Principle

Reaction of azomethine-H, which is the condensation product of H-acid (8-amino-naphth-1-ol-3,6-disulfonic acid) and salicylaldehyde, with dissolved forms of borate at a pH of about 6. Formation of a yellow complex that is measured spectrometrically at the absorption maximum in the range of 410 nm to 420 nm (see also **7.1**).

4 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity stored in polyethylene bottles.

4.1 *Azomethine-H,* solution

Dissolve 1,0 g of azomethine-H sodium salt [8-N-(2-hydroxybenzylidene)-amino-naphth-1-ol-3,6-disulfonic acid][1] ($C_{17}H_{12}NNaO_8S_2$) and 3,0 g of L + – ascorbic acid ($C_6H_8O_6$) in water and dilute to 100 ml in a one-mark volumetric flask.

The solution is stable for up to a week when stored in a polyethylene bottle at a temperature of between 4 °C and 6 °C.

4.2 *Buffer solution,* pH = 5,9

Mix 250 g of ammonium acetate (CH_3COONH_4), 250 ml of water, 80 ml of sulfuric acid (H_2SO_4) (ρ = 1,21 g/ml), 5 ml of phosphoric acid (H_2PO_4) (ρ = 1,71 g/ml), 1,0 g of citric acid ($C_6H_8O_7 \cdot H_2O$) and 1,0 g of disodiumethylenediamine-tetraacetic acid-dihydrate ($C_{10}H_{14}N_2Na_2O_8 \cdot H_2O$) by stirring and gentle heating.

4.3 *Reagent solution*

Mix equal volumes of reagents **4.1** and **4.2**. Prepare this solution on the day of use and store in a polyethylene bottle.

4.4 *Borate*, stock solution corresponding to 1,0 g of B per litre.

Dissolve 5,719 g of boric acid (H_3BO_3) in 1 000 ml of water.

Store in a polyethylene bottle.

1 ml of this stock solution contains 1,0 mg of borate, expressed as B.

4.5 *Boron*, standard solution 1 corresponding to 10,0 mg of B per litre.

Dilute 10 ml of borate stock solution (**4.4**) to 1 000 ml with water.

1 ml of this standard solution contains 10,0 µg of borate, expressed as B.

4.6 *Boron*, standard solution 2 corresponding to 1,0 mg of B per litre.

Dilute 10 ml of borate standard solution (**4.5**) to 100 ml with water.

1 ml of this standard solution contains 1,0 µg of borate, expressed as B.

4.7 *Calcium hydroxide* [$Ca(OH)_2$]

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[1] IUPAC name.

5 Apparatus

Ordinary laboratory apparatus made of polypropylene, polyethylene or polytetrafluoroethylene, where applicable, and

Spectrometer, for use in the wavelength range of 410 nm to 420 nm, with cells of an optical path length between 10 mm and 50 mm.

6 Sampling and samples

Samples for the determination of borate shall not be taken using borosilicate glass containers. The samples shall be preserved according to ISO 5667-3.

7 Procedure

7.1 Determination

Transfer 25,0 ml of the sample, or a smaller amount of the sample diluted to 25 ml with distilled water, into a 100 ml polyethylene flask. Add 10 ml of azomethine-H reagent (**4.1**). Mix and allow to stand in the dark for 2 h at 20 °C ± 1 °C, then measure the absorbance at the absorption maximum in the range of 410 nm to 420 nm against distilled water in a cell of optical pathlength 10 mm, using the spectrometer set up according to the manufacturer's instructions and after setting the zero with distilled water in the cell. Alternatively use a cell of 50 mm optical pathlength for low boron concentrations of up to about 0,2 mg of boron per litre. Check the wavelength of the absorption maximum whenever a new batch of this reagent is used.

NOTE 1 The reaction time may be shortened by keeping the treated sample at a temperature of 30 °C. In this case, the sample, the blank and the calibration samples should be treated accordingly, because the intensity of colour is temperature dependent.

7.2 Blank test

Carry out a blank test by treating 25 ml of water as described in **7.1**. Ensure that the blank value is in the range of 0,1 absorption units to 0,17 absorption units per 10 mm; if the absorption is higher then check the reagents and the distilled water for their borate content.

NOTE 2 The following procedure may be used to check the quality of reagents and the distilled water.

Measure into three separate borate-free beakers (preferably polytetrafluorethylene) 25 ml, 100 ml and 250 ml aliquots of the distilled water. Make each slightly alkaline by the addition of the same small (e.g. 200 mg) amount of calcium hydroxide (**4.7**) to each. Evaporate the 100 ml and 250 ml aliquots to a volume of just less than 25 ml and adjust their volumes to precisely 25 ml by the addition of a little extra distilled water, as necessary. Carry out the procedure given in **7.1** on these aliquots.

Carry out a blank determination with each of the aliquots. If borate is present in the distilled water, the borate found increases in proportion to the volume of the aliquot taken. Erratic results indicate external borate contamination. Relatively high but constant results indicate impure reagents.

7.3 Prevention of contamination

As borate is widespread in the environment, significant contamination may occur during trace determinations.

The following sources of contamination, and remedies, should be considered.

Laboratory glassware is usually made from borosilicate glass. Special borate-free thermally resistant glass is obtainable, but for routine purposes, old borosilicate glass, well rinsed in hydrochloric acid, may be used for acidic solutions, but should never be used for neutral or alkaline solutions, or for prolonged storage at any pH value. (Borosilicate glassware previously used with alkaline solutions shall not be used without very thorough acid rinsing.) Polyethylene flasks and plastics pipettes are preferable.

Detergents and soaps used for glassware and labcoats should be borate free, and the use of towels and tissues for drying shall be avoided.

Toiletries, talcum powder and cosmetics used by technicians often contain borate and should be avoided or removed, especially prior to undertaking accurate low-level determinations.

Water and reagents may contain borate and blanks should be carried out at least in duplicate and should agree.

7.4 Calibration

7.4.1 *0,00 mg/l to 0,20 mg/l of boron calibration graph*

To a series of six 25 ml one mark plastics flasks add respectively 0 ml, 1 ml, 2 ml, 3 ml, 4 ml and 5 ml of boron standard solution 2 (**4.6**), dilute to the mark with distilled water and mix. This gives concentrations of 0 mg; 0,04 mg; 0,08 mg; 0,12 mg; 0,16 mg and 0,20 mg of boron per litre respectively. Analyze each standard solution as described in **7.1**, measuring the absorbance values in a 50 mm optical path length cell compared against distilled water. Prepare a calibration graph by plotting the absorbance values against the known concentrations in milligrams of boron per litre for each standard.

7.4.2 *0,00 mg/l to 1,00 mg/l of boron calibration graph*

Repeat the above calibration,
using 0 ml, 5 ml, 10 ml, 15 ml, 20 ml and 25 ml of boron standard solution 2 (**4.6**) respectively to give concentrations
of 0 mg; 0,2 mg; 0,4 mg; 0,6 mg; 0,8 mg and 1,0 mg of boron per litre respectively. Analyze each standard solution as described in **7.1** but this time measuring the absorbance values using a 10 mm optical path length cell compared against distilled water. Prepare a separate calibration graph.

7.4.3 *Calculation of facteur* f[2]

It is essential that a linear calibration graph be achieved in both cases; if not then check the solutions and repeat the calibration. Calculate the reciprocal value for the slope, factor f, for each graph.

8 Expression of results

8.1 Calculation

Calculate the borate content, in milligrams of boron per litre, from the formula

$$\frac{(A_1 - A_0) \cdot f \cdot V_{1\,\max}}{V_1}$$

where

A_1	is the absorbance of the sample;
A_0	is the absorbance of the blank;
V_1	is the volume, in millilitres, of the sample;
$V_{1\,\max}$	is the maximum volume, in millilitres, of the sample;
f	is the calibration factor, determined from the appropriate calibration curve (reciprocal value of the slope, in milligrams of boron per litre).

8.2 Precision

An interlaboratory trial, carried out in Germany, F.R., delivered the values for boron given in Table 1.

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all information necessary for the complete identification of the sample;

c) the results and the method of expression used;

d) details of the removal of interferences, where applicable;

e) details of any operations not included in this International Standard, or regarded as optional, together with any other circumstances that may have affected the results.

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[2] See national foreword for details of textual errors.

Table 1 — Precision data

[a]	**Sample**	l	n	**Outlier**	x	$\bar{x}$	**WFR**	σ_r	VC_r	σ_R	VC_R
[a]				%	mg/l	mg/l	%	mg/l	%	mgl	%
	Standard solution	20	76	14,6	0,25	0,234	93,5	0,027	11,4	0,014	5,9
	Surface water	19	71	20	—	0,151		0,019	12,3	0,008	5,6
	Mineral water	20	75	15,7	—	0,050	—	0,036	7,2	0,015	3,0
	Effluent from a biological treatment plant	19	74	16,9	—	1,060	—	0,069	6,5	0,035	3,3

where

l	is the number of laboratories;
n	is the number of values;
x	is the true value;
$\bar{x}$	is the mean value;
WFR	is the recovery rate;
σ_r	is the repeatability standard deviation;
VC_r	is the repeatability variation coefficient;
σ_R	is the reproducibility standard deviation;
VC_R	is the reproducibility variation coefficient.

[a] See national foreword for details of textual errors.

Publication(s) referred to

See national foreword.

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BSI
389 Chiswick High Road
London
W4 4AL