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**Water quality — Determination of borate —
Spectrometric method using azomethine-H**

iTeh STANDARD PREVIEW
*Qualité de l'eau — Dosage du borate — Méthode spectrométrique à
l'azométhine-H*
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 9390 was prepared by Technical Committee ISO/TC 147, *Water quality*.

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Introduction

The natural borate content of groundwater 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.

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Water quality — Determination of borate — Spectrometric method using azomethine-H

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) ($\rho = 1,21$ g/ml), 5 ml of phosphoric acid (H_3PO_4) ($\rho = 1,71$ g/ml), 1,0 g of citric acid ($C_6H_8O_7 \cdot H_2O$) and 1,0 g of disodiummethylenediamine-tetraacetic acid-dihydrate ($C_{10}H_{14}N_2Na_2O_8 \cdot H_2O$) by stirring and gentle heating.

1) IUPAC name.

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 1000 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 1000 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 [$Ca(OH)_2$].

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 $20\text{ }^\circ\text{C} \pm 1\text{ }^\circ\text{C}$, 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 $30\text{ }^\circ\text{C}$. 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 polytetrafluoroethylene) 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 rins-

ing.) 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

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 reference to this International Standard;
- all information necessary for the complete identification of the sample;
- the results and the method of expression used;
- details of the removal of interferences, where applicable;
- 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.

Table 1 — Precision data

Sample	l	n	Outlier	x	\bar{x}	WFR	σ_r	VC_r	σ_R	VC_R
			%	mg/l	mg/l	%	mg/l	%	mg/l	%
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.

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