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Water quality — Determination of sodium and potassium —

Part 1:
Determination of sodium by atomic absorption spectrometry

Qualité de l'eau — Dosage du sodium et du potassium —

Partie 1: Dosage du sodium par spectrométrie d'absorption atomique

Reference number
ISO 9964-1:1993(E)

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 9964-1 was prepared by Technical Committee ISO/TC 147, *Water quality*, Sub-Committee SC 2, *Physical, chemical, biochemical methods*.

ISO 9964 consists of the following parts, under the general title *Water quality — Determination of sodium and potassium*:

- *Part 1: Determination of sodium by atomic absorption spectrometry*
- *Part 2: Determination of potassium by atomic absorption spectrometry*
- *Part 3: Determination of sodium and potassium by flame emission spectrometry*

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Water quality — Determination of sodium and potassium —

Part 1:
Determination of sodium by atomic absorption spectrometry

1 Scope

1.1 Field of application

This International Standard specifies a method for the determination of dissolved sodium by flame atomic absorption spectrometry (AAS). It is intended for the analysis of raw and drinking water.

The method is applicable to water samples with a mass concentration of sodium in the range from 5 mg/l to 50 mg/l. This range can be extended to lower or higher limits if dilution factors are chosen which are different from the factor specified in clause 8.

1.2 Interferences

The ions normally present in raw and drinking waters do not interfere with the determination of sodium by atomic absorption spectrometry.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 9964. 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 9964 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 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.*

3 Principle

Addition of caesium chloride solution to the sample as an ionization suppressant. Aspiration of the sample directly into the air/acetylene flame of an atomic absorption spectrometer. Measurement of the absorbance at a wavelength of 589,0 nm.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only deionized water or water of equivalent purity.

4.1 Hydrochloric acid, $c(\mathrm{HCl}) \approx 11$ mol/l, $\rho = 1{,}18$ g/ml.

4.2 Nitric acid, $c(\mathrm{HNO_3}) \approx 16$ mol/l, $\rho = 1{,}41$ g/ml.

4.3 Caesium chloride, (CsCl), solution.

Dissolve 25 g of caesium chloride in a solution of 50 ml of hydrochloric acid (4.1) and 450 ml of water, and dilute to 1 litre with water in a one-mark volumetric flask.

One litre of this solution contains approximately 20 g of Cs.

NOTE 1 Nitric acid (4.2) may be used instead of hydrochloric acid (4.1).

4.4 Sodium, stock solution.

In a 1 000 ml one-mark volumetric flask, dissolve 2,542 g ± 0,005 g of sodium chloride (previously dried for at least 1 h at 140 °C ± 10 °C) in water and make up to the mark.

Store the solution, which is stable for at least 6 months, in a polyethylene bottle.

One litre of this solution contains 1 000 mg of Na.

Alternatively, use a commercially available, ready-made solution.

4.5 Sodium, standard solution.

Pipette 10 ml of the sodium stock solution (4.4) into a 1 000 ml one-mark volumetric flask. Make up to the mark with water.

The solution shall be prepared freshly when required.

1 ml of this standard solution contains 10 µg of Na.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Atomic absorption spectrometer, set up and operated according to the manufacturer's instructions, it is equipped with a burner for an air/acetylene flame, a hollow cathode lamp for sodium determination, and a compatible red-sensitive photomultiplier.

A spectral bandwith of < 0,3 nm is recommended.

5.2 Glassware of borosilicate glass, and polyethylene ware.

Clean both glass and polyethylene ware by soaking in an aqueous solution of 10 % (*V/V*) of nitric acid (4.2) followed by thorough rinsing with water. The apparatus should only be used for this method.

6 Sampling

Collect samples in clean polyethylene bottles (see ISO 5667-1 and ISO 5667-2). Sample preservation by acidification is not generally necessary.

NOTE 2 If other metal analyses are to be carried out on the samples, the samples can be preserved by addition of hydrochloric acid (4.1) or nitric acid (4.2) to give a pH of approximately 1. All samples, standards and blanks should contain the same concentration and the same type of acid.

7 Procedure

7.1 Preparation of test samples for presentation to the spectrometer

7.1.1 Filter samples containing particulate matter through an acid-washed filter of pore size 0,45 µm to prevent clogging of the nebulizer and burner systems. (The acid used for washing the filter should have the same concentration and be of the same type as the acid used in the sample preparation.)

NOTE 3 Instead of filtration, the particulate matter may be removed from suspension by using a centrifuge.

7.1.2 Take a number of 100 ml one-mark volumetric flasks according to the number of samples being analysed. To each of these flasks, add 10 ml of the caesium chloride solution (4.3).

7.1.3 Pipette, 2 ml of the sample into each of these caesium chloride solutions, and make up to the mark with water. If the concentration of the test solution is not in the optimum range of 0,1 mg/l to 1,0 mg/l of Na, adjust the volume of the sample appropriately.

7.2 Preparation of the set of calibration solutions

Add 10 ml of caesium chloride solution to each of a series of 100 ml one-mark volumetric flasks. Pipette 0 ml; 1,0 ml; 2,0 ml; 4,0 ml; 6,0 ml and 10,0 ml of the sodium standard solution (4.5) into the series flasks and make up each flask to the mark with water.

The calibration solutions will have the concentrations 0 mg/l; 0,1 mg/l; 0,2 mg/l; 0,4 mg/l; 0,6 mg/l and 1,0 mg/l of Na, respectively.

7.3 Calibration and determination

7.3.1 Set up the spectrometer according to the manufacturer's instructions by aspirating a calibration solution (7.2). Optimize the aspiration and flame conditions (aspiration rate, nature of the flame, position of the optical beam in the flame). Adjust the response of the instrument to zero absorbance with water.

7.3.2 Aspirate the calibration solutions (7.2) with an aspiration of water between each. Measure the absorbance at 589,0 nm. Prepare a calibration graph by plotting the respective absorbance readings on the ordinate against the mass concentrations of sodium on the abscissa.

Calculate from the graph the slope, b, in litres per milligram.

NOTE 4 The calibration graph is normally linear for the concentration range 0,1 mg/l to 1,0 mg/l.

7.3.3 Aspirate the test samples (7.1), with an aspiration of water between each, and determine the absorbance.

7.3.4 Carry out a blank determination with each batch of samples by applying the same procedure and using water instead of the sample.

NOTE 5 It is good practice to check the slope of the calibration graph at regular intervals (e.g. every 10 samples).

8 Calculation and expression of results

8.1 Use of calibration graph

Read the concentrations of sodium in the test solutions from the calibration graph (see 7.3.2). From these values, calculate the sodium concentration of the test sample, taking into account the volume of test sample taken (normally 2 ml) and the total volume of the volumetric flask (100 ml).

8.2 Method of calculation

If the calibration graph is linear, calculate the mass concentration of sodium, ρ_{Na}, in the sample, in milligrams per litre, using the equation

$$\rho_{\mathrm{Na}} = \frac{(A - A_0)\cdot V_{\mathrm{m}}}{V_{\mathrm{p}}\cdot b} \qquad \ldots (1)$$

where

A is the absorbance of the sample;

A_0 is the absorbance of the blank;

V_{m} is the volume, in millilitres, of the test portion (normally 2 ml);

V_{p} is the volume, in millilitres, of the volumetric flask (100 ml);

b is the slope of the calibration curve, in litres per milligram.

If required, calculate the amount-of-substance concentration, c_{Na}, in millimoles per litre, using the equation

$$c_{\mathrm{Na}} = \frac{\rho_{\mathrm{Na}}}{23{,}0} \qquad \ldots (2)$$

If the calibration curve is not linear, proceed as described in 8.1.

8.3 Precision

An interlaboratory trial, carried out in autumn 1991, using this method, produced the results given in table 1.

Table 1 — Precision data

Sample¹⁾	l	n	n_a %	$\bar{x}$ mg/l	σ_r mg/l	VC$_r$ %	σ_R mg/l	VC$_R$ %
A	8	21	13	6,11	0,211 4	3,5	0,112 4	1,8
B	8	24	0	65,2	2,503 4	3,8	0,588 1	0,9
C	8	21	13	294	11,071	3,8	2,320	0,8

l is the number of laboratories

n is the number of values

n_a is the percentage of outliers

$\bar{x}$ is the mean value

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

1) A: drinking water

B: surface water

C: waste water

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) precise identification of the water sample;

c) the results and the method of expression used, in accordance with clause 8;

d) any deviation from this method or any other circumstances which may have influenced the results.