

INTERNATIONAL
STANDARD

ISO
9964-3

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**Water quality — Determination of sodium
and potassium —**

Part 3:

Determination of sodium and potassium by
flame emission spectrometry

*Qualité de l'eau — Dosage du sodium et du potassium —
Partie 3: Dosage par spectrométrie d'émission de flamme*



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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 9964-3 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*

Annex A of this part of ISO 9964 is for information only.

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Water quality — Determination of sodium and potassium —

Part 3:

Determination of sodium and potassium by flame emission spectrometry

1 Scope

1.1 Field of application

This International Standard specifies a method for the determination of dissolved sodium and potassium by flame emission spectrometry (FES). It is intended for the analysis of raw and drinking waters.

The method is applicable to water samples with a mass concentration of sodium and potassium of up to 10 mg/l. For samples containing higher concentrations of sodium and potassium, a smaller test portion is taken for analysis. The lower limits of determination are less than 0,1 mg/l for both sodium and potassium.

1.2 Interferences

The ions normally present in raw and drinking waters do not interfere with the flame emission spectrometric method for sodium and potassium if an ionization suppressant is present.

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

Aspiration of the sample into a gas flame of sufficient thermal energy to cause any sodium and potassium present to emit its characteristic radiation. Measurement of the intensity at a wavelength of 589,0 nm for sodium and 766,5 nm for potassium.

When using the air/acetylene flame, the addition of caesium chloride solution as an ionization buffer is necessary.

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

4.2 Nitric acid, $c(\text{HNO}_3) \approx 16 \text{ mol/l}$, $\rho = 1,41 \text{ g/ml}$.

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 $5,084 \text{ g} \pm 0,005 \text{ g}$ of sodium chloride (previously dried for at least 1 h at $140 \text{ }^\circ\text{C} \pm 10 \text{ }^\circ\text{C}$) 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 2 000 mg of Na.

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

4.5 Potassium, stock solution.

In a 1 000 ml one-mark volumetric flask, dissolve $3,814 \text{ g} \pm 0,005 \text{ g}$ of potassium chloride (previously dried for at least 1 h at $140 \text{ }^\circ\text{C} \pm 10 \text{ }^\circ\text{C}$) 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 2 000 mg of K.

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

4.6 Sodium/potassium, standard solution.

Pipette 10 ml of the sodium stock solution (4.4) and the potassium stock solution (4.5) 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 20 μg of Na and 20 μg of K.

5 Apparatus

Ordinary laboratory apparatus and

5.1 Flame photometer, equipped with an appropriate interference filter for the determination of sodium and potassium, or

Atomic absorption spectrometer, in the emission mode, set up and operated according to the manufacturer's instructions.

5.2 Glassware 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 deionized 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 necessary.

NOTE 2 If other metal analyses are to be carried out on the samples, the sample can be preserved using 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 instrument

7.1.1 Filter samples containing particulate matter through an acid-washed filter of pore size 0,45 μm . (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 50 ml one-mark volumetric flasks according to the number of samples being analysed. To each of these flasks, add up to 40 ml of the test sample and 5 ml of the caesium chloride solution (4.3). Make up to the mark with water.

7.2 Preparation of the set of calibration solutions

Pipette 0 ml; 1,0 ml; 2,5 ml; 5 ml; 10 ml; 15 ml; 20 ml and 25 ml of the sodium/potassium standard solution (4.6) and 5 ml of the caesium chloride solution (4.3) into a series of 50 ml one-mark volumetric flasks and make up each flask to the mark with water.

The calibration solutions will have the concentration 0 mg/l; 0,4 mg/l; 1,0 mg/l; 2,0 mg/l; 4,0 mg/l; 6,0 mg/l; 8,0 mg/l and 10,0 mg/l of Na and K, respectively.

NOTE 4 Addition of the caesium chloride solution (4.3) can be omitted for the test samples (7.1) and the calibration solutions (7.2) if flames other than an air/acetylene flame are used.

7.3 Calibration and determination

7.3.1 Set up the spectrometer/photometer according to the manufacturer's instructions. Fit the appropriate filter for the metal being measured. Aspirate the calibration solution (7.2). Optimize the aspiration and flame conditions (aspiration rate, nature of the flame).

7.3.1.1 For flame photometer. Aspirate water and adjust the scale reading to zero deflection (0 % reading). Aspirate the 10 mg/l calibration solution (see 7.2) and adjust the scale reading to full deflection (100 %) reading). Repeat the aspiration of water and the 10 mg/l calibration solution until the 0 % and 100 % readings, respectively, are obtained without the need of further adjustment.

7.3.1.2 For atomic absorption spectrometer in the emission mode. Aspirate water and adjust the response for the instrument to zero absorbance.

7.3.2 Aspirate the calibration solutions (7.2) with an aspiration of water between each. Measure the instrument response of each solution (at a wavelength of 589,0 nm for sodium and at 766,5 nm for potassium) using the spectrometer. Prepare calibration graphs for sodium and potassium by plotting the appropriate instrument response on the ordinate against the mass concentrations of sodium and potassium on the abscissa.

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

NOTE 5 The calibration graph is normally linear up to 10 mg/l but can show a slight curvature, especially when a flame emission spectrometer is used.

7.3.3 Aspirate the test samples (7.1) with an aspiration of water between each, and measure the instrument response.

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 6 It is good practice to check the slope of calibration graphs 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 and potassium in the test solutions from the calibration graph (see

7.3.2). From these values, calculate the sodium and potassium concentration of the test sample, taking into account the volume of the test sample taken (normally 40 ml) and the total volume of the volumetric flask (50 ml).

8.2 Method of calculation

If the calibration graph is linear, calculate the mass concentrations of sodium, ρ_{Na} , and potassium, ρ_{K} , in the sample, in milligrams per litre, using the equations

$$\rho_{\text{Na}} = \frac{(R_{\text{Na}} - R_0) \cdot V_m}{V_p \cdot b} \quad \dots (1)$$

$$\rho_{\text{K}} = \frac{(R_{\text{K}} - R_0) \cdot V_m}{V_p \cdot b} \quad \dots (2)$$

where

R is the response of the sample;

R_0 is the response of the blank;

V_m is the volume, in millilitres, of the test portion (normally 40 ml);

V_p is the volume, in millilitres, of the volumetric flask (50 ml);

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

If required, calculate the amount of substance concentrations, c_{Na} and c_{K} , in millimoles per litre, using the equations

$$c_{\text{Na}} = \frac{\rho_{\text{Na}}}{23,0} \quad \dots (3)$$

$$c_{\text{K}} = \frac{\rho_{\text{K}}}{39,1} \quad \dots (4)$$

8.3 Precision

An interlaboratory trial, carried out in spring 1992, produced the results given in table 1.

Table 1 — Precision data

Sample ¹⁾	l	n	n _b %	\bar{x} mg/l	σ_r mg/l	VC _r %	σ_R mg/l	VC _R %
Sodium								
A	7	21	0	4,38	0,396	9,0	0,080	1,8
B	7	21	0	63,1	1,539	2,4	0,568	0,9
C	7	21	0	87,0	2,843	3,3	0,363	0,4
Potassium								
A	7	18	0	0,37	0,092	24,9	0,052	14,0
B	7	21	0	5,28	0,213	4,0	0,040	0,8
C	7	18	14	15,1	0,296	2,0	0,149	1,0
l	is the number of laboratories			σ_r	is the repeatability standard deviation			
n	is the number of values			VC _r	is the repeatability variation coefficient			
n _b	is the percentage of outliers			σ_R	is the reproducibility standard deviation			
\bar{x}	is the mean value			VC _R	is the reproducibility variation coefficient			
1) A: drinking water B: river water C: municipal 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.