SLOVENSKI STANDARD
SIST ISO 7890-1:1996

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Kakovost vode - Določanje nitrata - 1. del: Spektrofotometrijska metoda z 2,6 dimetilfenolom

Water quality -- Determination of nitrate -- Part 1: 2,6-Dimethylphenol spectrometric method

Qualité de l'eau -- Dosage des nitrates -- Partie 1: Méthode spectrométrique au diméthyl-2,6 phénol

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International Standard 7890/1

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Water quality — Determination of nitrate — Part 1 : 2,6-Dimethylphenol spectrometric method

Qualité de l'eau — Dosage des nitrates — Partie 1: Méthode spectrométrique au diméthyl-2,6 phénol

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7890/1 was prepared by Technical Committee ISO/TC 147, *Water quality*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Water quality — Determination of nitrate — Part 1: 2,6-Dimethylphenol spectrometric method

1 Scope and field of application

1.1 Substance determined

This part of ISO 7890 specifies a method for the determination of nitrate ion in water.

1.2 Type of sample

This method is applicable to the direct analysis of potable and raw water.

NOTE — Polluted waters and saline waters should be analysed by the procedures given in ISO 7890/2, *Water quality — Determination of nitrate — Part 2: 4-Fluorophenol spectrometric method after distillation.*

1.3 Range

A nitrate nitrogen concentration, ϱ_N, of up to 25 mg/l in the test portion can be determined.

1.4 Limit of detection

A nitrate nitrogen concentration of ϱ_N = 0,06 mg/l.

1.5 Sensitivity

A nitrate nitrogen content of ϱ_N = 25 mg/l gives an absorbance of about 1,5 units in a cell of path length 10 mm.

1.6 Interferences

Potential interference from nitrite nitrogen at concentrations of up to at least ϱ_N = 5 mg/l is controlled by the use of amidosulfonic acid.

Chloride may seriously interfere, but can be removed by addition of silver sulfate to the test sample and filtration prior to taking the test portions (see clause 8). The effect of chloride on the determination and the effectiveness of the chloride removal procedure are shown in the annex.

2 Principle

Reaction of nitrate with 2,6-dimethylphenol in the presence of sulfuric and phosphoric acids to produce 4-nitro-2,6-dimethylphenol. The reaction time is about 5 min. Spectrometric measurement of the absorbance of the reaction product at 324 nm and reading of the nitrate concentration in the test portion from a calibration graph.

3 Reagents

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

3.1 Glacial acetic acid (CH_3COOH), $\varrho \approx$ 1,05 g/ml.

3.2 2,6-Dimethylphenol solution, 1,2 g/l.

Dissolve 1,2 ± 0,1 g of 2,6-dimethylphenol [$(CH_3)_2C_6H_3OH$] in 1 000 ± 10 ml of glacial acetic acid (3.1).

Store in a glass bottle.

This solution is stable for 1 week.

3.3 Acid mixture.

WARNING — When using this acid mixture, eye protection and protective clothing are essential. The mixture should never be pipetted by mouth.

Cautiously mix 500 ± 5 ml of sulfuric acid (H_2SO_4) (ϱ = 1,84 g/ml) with 500 ± 5 ml of orthophosphoric acid (H_3PO_4) (ϱ = 1,69 g/ml), in a 2 litre glass beaker. Add 0,040 ± 0,005 g of amidosulfonic acid (NH_2SO_3H) to the mixture and dissolve.

Store in a glass-stoppered bottle.

This solution is stable indefinitely.

3.4 **Nitrate,** stock solution, ϱ_N = 1 000 mg/l.

Dissolve 7,218 ± 0,001 g of potassium nitrate (KNO_3) (previously dried at 105 °C for at least 2 h) in about 750 ml of water in a 1 000 ml one-mark volumetric flask. Make up to the mark with water.

Store in a glass bottle for not more than 2 months.

3.5 **Nitrate,** standard solution, ϱ_N = 100 mg/l.

Pipette 50 ml of the nitrate stock solution (3.4) into a 500 ml one-mark volumetric flask. Make up to the mark with water.

Store in a glass bottle for not more than 1 month.

1 ml of this standard solution corresponds to 0,1 mg of nitrate nitrogen.

4 Apparatus

Usual laboratory equipment, and

Spectrometer, capable of operating at a wavelength of 324 nm and equipped with cells of path length 10 mm.

5 Sampling and samples

Laboratory samples should be collected in glass or polyethylene bottles and should be analysed as soon as possible after collection. Storage of samples at between 2 and 5 °C may preserve many types of sample, but checks should be made to confirm this with each sample type.

Laboratory samples containing suspended matter should be allowed to settle, or be filtered through a glass fibre paper, before taking the test portion.

6 Procedure

6.1 Test portion

Pipette 5 ml of the laboratory sample (clause 5).

6.2 Blank test

Carry out a blank test in parallel with the determination, using 5 ml of water instead of the test portion.

6.3 Calibration

6.3.1 Preparation of the set of calibration solutions

Into a series of six 100 ml one-mark volumetric flasks add, by pipette, 1; 5; 10; 15; 20; and 25 ml of nitrate standard solution (3.5). Make up to the mark with water. These solutions contain 1; 5; 10; 15; 20; and 25 mg of nitrate nitrogen per litre respectively.

Store these solutions in glass bottles for not more than 1 week.

6.3.2 Colour development

Pipette 35 ml of the acid mixture (3.3) into each of a series of six dry 100 ml conical flasks, using a safety pipette filler. Pipette 5 ml of the calibration solutions (6.3.1) into each flask. Pipette into each flask 5 ml of 2,6-dimethylphenol solution (3.2). Mix the flask contents thoroughly by swirling, and allow to stand for between 10 and 60 min.

6.3.3 Spectrometric measurements

Measure the absorbance of each calibration solution at 324 nm in a cell of path length 10 mm, against water in the reference cell.

6.3.4 Plotting the calibration graph

Subtract the absorbance of the blank test (6.2) from the absorbances of the calibration solutions and plot a graph of absorbance against concentration, expressed in milligrams per litre, of nitrate nitrogen. This graph should be linear and should pass through the origin.

6.4 Determination

Proceed as in 6.3.2 and 6.3.3, using one 100 ml conical flask and the test portion (6.1) instead of the calibration solutions.

7 Expression of results

7.1 Calculation

The absorbance, A_0, due to nitrate nitrogen in the test portion is given by the equation

$$A_0 = A_1 - A_2$$

where

A_1 is the absorbance of the test portion (6.4);

A_2 is the absorbance of the blank (6.2).

Read off from the calibration graph (6.3.4) the nitrate nitrogen concentration, ϱ_N, expressed in milligrams per litre, corresponding to the absorbance, A_0.

The result may be expressed in various ways (see table 1).

Table 1

	$c(NO_3^-)$	$\varrho_{NO_3^-}$	ϱ_N
	mmol/l	mg/l	mg/l
$c(NO_3^-)$ = 1 mmol/l	1	62	14,01
$\varrho_{NO_3^-}$ = 1 mg/l	0,016 1	1	0,226
ϱ_N = 1 mg/l	0,071 4	4,427	1

Example:

A nitrate concentration, $\varrho_{NO_3^-}$, of 1 mg/l corresponds to a nitrate nitrogen concentration, ϱ_N, of 0,226 mg/l.

7.2 Precision

7.2.1 Repeatability

Repeatability standard deviations have been determined[1] on standard solutions as shown in table 2.

Table 2

Nitrate content, ϱ_N	Standard deviation	Degrees of freedom
mg/l	mg/l	
3,00	0,20	4
6,00	0,35	4
9,00	0,25	4
12,00	0,14	4
24,65	0,07	19

7.2.2 Reproducibility

Reproducibility standard deviations have been determined[1] in an interlaboratory exercise as shown in table 3.

Table 3

Sample	Nitrate content, ϱ_N	Total standard deviation ϱ_N	Degrees of freedom
	mg/l	mg/l	
Potable water	2,6	0,17	21
Ground water	5,8	0,71	20
Surface water	10,2	0,29	18
Sewage effluent	15,9	0,82	20

8 Special cases

8.1 General

The effect of chloride on this method is shown in the annex. If the chloride concentration in a laboratory sample is likely to interfere, chloride shall be removed from a test sample by precipitation with silver sulfate.

8.2 Principle and reactions

Silver sulfate reacts with chloride ion according to the equation

$$Ag_2SO_4 + 2Cl^- \rightarrow 2AgCl + SO_4^{2-}$$

Therefore, 312 mg of silver sulfate will precipitate 71 mg of chloride ion. Addition of approximately twice this stoichiometric amount of silver sulfate to a test sample to ensure complete precipitation of chloride. Filtration of the test sample before withdrawing a test portion for analysis.

8.3 Reagents

8.3.1 Silver sulfate solution, 4,4 g/l.

Dissolve 4,40 ± 0,02 g of silver sulfate (Ag_2SO_4) in about 950 ml of water and dilute to 1 litre with water in a measuring cylinder.

This solution is stable indefinitely if stored immediately in an amber glass bottle.

8.4 Apparatus and materials

Usual laboratory apparatus, and

Fine porosity ashless filter papers. No nitrate should be leached from them during filtration.

8.5 Procedure

First determine the chloride concentration, ϱ_{Cl}, expressed in milligrams per litre, of the laboratory sample.

Pipette 25 ml of the laboratory sample into a dry 50 ml beaker. Add, from a burette, 0,05 ϱ_{Cl} ml of silver sulfate solution (8.3), where ϱ_{Cl} is the chloride concentration already determined. Note the volume of silver sulfate solution added.

Swirl the beaker contents to mix, and then filter the mixture through a filter paper (8.4). Collect the filtrate in a dry 50 ml beaker.

Carry out the procedure (clause 6) using 5 ml of this filtrate as the test portion.

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1) Data from the Federal Republic of Germany.

8.6 Expression of results

Proceed as in 7.1. The nitrate concentration, ϱ'_N, expressed in milligrams per litre, of the laboratory sample is given by the equation

$$\varrho'_N = \frac{\varrho_N (25 + V)}{25}$$

where

ϱ_N is the nitrate concentration, expressed in milligrams per litre, read from the calibration graph;

V is the volume, in millilitres, of silver sulfate solution added (8.5).

9 Test report

The test report shall contain the following information:

a) a reference to this part of ISO 7890;

b) precise identification of the sample;

c) details of the storage of the laboratory sample before analysis;

d) a statement of the repeatability achieved by the laboratory when using this method;

e) the result in terms of ϱ_N, expressed in milligrams per litre, or $\varrho_{NO_3^-}$, expressed in milligrams per litre, or $c(NO_3^-)$, expressed in millimoles per litre;

f) any deviation from the standard procedure or any other circumstance that may have affected the result.