



SLOVENSKI STANDARD SIST EN ISO 7393-2:2000

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Nadomešča:
SIST ISO 7393-2:1996

Kakovost vode - Določevanje prostega in celotnega klora - 2. del: Kolorimetrijska metoda z uporabo N,N-diethyl-1,4-fenildiamina za potrebe redne kontrole (ISO 7393-2:1985)

Water quality - Determination of free chlorine and total chlorine - Part 2: Colorimetric method using N, N-diethyl-1, 4-phenylenediamine, for routine control purposes (ISO 7393-2:1985)

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Wasserbeschaffenheit - Bestimmung von freiem Chlor und Gesamtchlor - Teil 2: Kolorimetrisches Verfahren mit N, N-Diethyl-1, 4-Phenylendiam für Routinekontrollen (ISO 7393-2:1985)

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Qualité de l'eau - Dosage du chlore libre et du chlore total - Partie 2: Méthode colorimétrique a la N, N-diéthylphénylène-1, 4-diamine destinée aux contrôles de routine (ISO 7393-2:1985)

Ta slovenski standard je istoveten z: EN ISO 7393-2:2000

ICS:

13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances
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EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN ISO 7393-2

January 2000

ICS 13.060

English version

Water quality - Determination of free chlorine and total chlorine -
Part 2: Colorimetric method using *N,N*-diethyl-1,4-
phenylenediamine for routine control purposes (ISO 7393-
2:1985)

Qualité de l'eau - Dosage du chlore libre et du chlore total -
Partie 2: Méthode colorimétrique à la *N,N*-
diéthylphénylène-1,4 diamine destinée aux contrôles de
routine (ISO 7393-2:1985)

Wasserbeschaffenheit - Bestimmung von freiem Chlor und
Gesamtchlor - Teil 2: Kolorimetrisches Verfahren mit *N,N*-
Diethyl-1,4-Phenyldiam für Routinekontrollen (ISO 7393-
2:1985)

This European Standard was approved by CEN on 20 January 2000.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

[SIST EN ISO 7393-2:2000](http://standards.iteh.ai/SIST/EN/ISO/7393-2/2000)

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

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Foreword

The text of the International Standard from Technical Committee ISO/TC 147 "Water quality" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 230 "Water analysis", the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by July 2000, and conflicting national standards shall be withdrawn at the latest by July 2000.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 7393-2:1985 has been approved by CEN as a European Standard without any modification.

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International Standard



7393/2

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Water quality — Determination of free chlorine and total chlorine — Part 2 : Colorimetric method using *N,N*-diethyl-1,4-phenylenediamine, for routine control purposes

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Qualité de l'eau — Dosage du chlore libre et du chlore total — Partie 2 : Méthode colorimétrique à la N,N-diéthylphénylène-1,4 diamine destinée aux contrôles de routine

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Descriptors : water, quality, chemical analysis, determination of content, chlorine, colorimetric analysis.

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 7393/2 was prepared by Technical Committee ISO/TC 147, *Water quality*.

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Water quality — Determination of free chlorine and total chlorine —

Part 2: Colorimetric method using *N,N*-diethyl-1,4-phenylenediamine, for routine control purposes

0 Introduction

ISO 7393 consists of the following parts:

Part 1: Titrimetric method using *N,N*-diethyl-1,4-phenylenediamine.

Part 2: Colorimetric method using *N,N*-diethyl-1,4-phenylenediamine, for routine control purposes.

Part 3: Iodometric titration method for the determination of total chlorine.¹⁾

1 Scope and field of application

This part of ISO 7393 specifies a method for the determination of free chlorine and total chlorine in water, readily applicable to field testing; it is based on measurement of the colour intensity by visual comparison of the colour with a scale of standards which is regularly calibrated.

Sea water and waters containing bromides and iodides comprise a group for which special procedures are required.^[2]

This method is applicable to concentrations, in terms of chlorine (Cl_2), from 0,000 4 to 0,07 mmol/l (0,03 to 5 mg/l) total chlorine. For higher concentrations the test portion must be diluted. If the speed of operation and the compactness of the equipment are not overriding requirements, spectrometric measurement is described as an alternative procedure.

In annex A a procedure is presented for the differentiation of combined chlorine of the monochloramine type, combined chlorine of the dichloramine type and combined chlorine in the form of nitrogen trichloride.

Interferences are noted in clauses 7 and 9.

2 Definitions (see table 1)

For the purpose of this part of ISO 7393, the following definitions apply.

2.1 free chlorine: Chlorine present in the form of hypochlorous acid, hypochlorite ion or dissolved elemental chlorine.

2.2 combined chlorine: The fraction of total chlorine present in the form of chloramines and organic chloramines.

2.3 total chlorine: Chlorine present in the form of "free chlorine" or "combined chlorine" or both.

2.4 chloramines: Derivatives of ammonia by substitution of one, two or three hydrogen atoms with chlorine atoms (monochloramine NH_2Cl , dichloramine NHCl_2 , nitrogen trichloride NCl_3) and all chlorinated derivatives of organic nitrogen compounds as determined by the method specified in this part of ISO 7393.

3 Principle

3.1 Determination of free chlorine

Direct reaction with the *N,N*-diethyl-1,4-phenylenediamine (DPD) and formation of a red compound at pH 6,2 to 6,5. Measurement of the colour intensity by visual comparison of the colour with a scale of permanent glass standards, or by spectrometry.

3.2 Determination of total chlorine

Reaction with DPD in the presence of an excess of potassium iodide then measurement as in 3.1.

Table 1 — Terms and synonyms in relation to actual compounds in the solution

Term	Synonym		Compounds
Free chlorine	Free chlorine	Active free chlorine	Elemental chlorine, hypochlorous acid
		Potential free chlorine	Hypochlorite
Total chlorine	Total residual chlorine		Elemental chlorine, hypochlorous acid, hypochlorite, and chloramines

1) At present at the stage of draft.

ISO 7393/2-1985 (E)

4 Reagents

During the analysis, use only reagents of recognized analytical grade, and only water as specified in 4.1.

4.1 Water, free from oxidizing and reducing substances.

Demineralized or distilled water of which the quality is checked as follows.

Into two 250 ml chlorine-demand-free conical flasks (clause 5) place, in order,

a) in the first: 100 ml of the water to be checked and about 1 g of potassium iodide (4.4); mix and after 1 min add 5 ml of buffer solution (4.2) and 5,0 ml of DPD reagent (4.3);

b) in the second: 100 ml of the water to be checked and two drops of sodium hypochlorite solution (4.7); then, after 2 min, 5,0 ml of buffer solution (4.2) and 5 ml of DPD reagent (4.3).

No coloration should appear in the first flask whereas it is essential that a light pink coloration appears in the second flask.

In the case of demineralized or distilled water not having the desired quality it must be chlorinated. After a period of contact followed by dechlorination the quality finally has to be rechecked.

A procedure for the preparation of water free from oxidizing and reducing substances is given in annex B.

4.2 Buffer solution, pH 6,5.

Dissolve in water (4.1) in this order: 24 g of anhydrous disodium hydrogen phosphate (Na_2HPO_4) or 60,5 g of the dodecahydrate form ($\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$) and 46 g of potassium dihydrogen phosphate (KH_2PO_4). Add 100 ml of 8 g/l disodium dihydrogenethylenedinitrilotetraacetate dihydrate (disodium EDTA dihydrate, $\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_8\text{Na}_2 \cdot 2\text{H}_2\text{O}$) solution (or 0,8 g of the solid form).

If necessary, add 0,020 g of mercury(II) chloride (HgCl_2), to prevent mould growth and interference in the free chlorine test caused by any trace amounts of iodide in the reagents.

Dilute to 1 000 ml and mix.

NOTE — Solutions containing mercury should be disposed of safely (for example a method is specified in ISO 5790, *Inorganic chemical products for industrial use — General method for determination of chloride content — Mercurimetric method*).

4.3 *N,N*-diethyl-1,4-phenylenediamine sulfate (DPD)

$[\text{NH}_2\text{-C}_6\text{H}_4\text{-N}(\text{C}_2\text{H}_5)_2\text{-H}_2\text{SO}_4]$, solution, 1,1 g/l.

Mix 250 ml water (4.1), 2 ml sulfuric acid ($\rho = 1,84$ g/ml) and 25 ml of 8 g/l disodium EDTA dihydrate solution (or 0,2 g of the solid form). Dissolve in this mixture 1,1 g of anhydrous DPD or 1,5 g of the pentahydrate form, dilute to 1 000 ml and mix.

Store the reagent in a dark bottle protected from heat.

Renew the solution after 1 month or when it becomes discoloured.

4.4 Potassium iodide, crystals.

NOTE — Reagents 4.2, 4.3 and 4.4 may be conveniently replaced by combined reagents commercially available in the form of stable powder or tablets.

4.5 Sulfuric acid, $c(\text{H}_2\text{SO}_4) \approx 1$ mol/l.

Take 800 ml water (4.1) and add cautiously with continuous stirring 54 ml of sulfuric acid ($\rho = 1,84$ g/ml). Cool to room temperature and transfer the solution to a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix well.

4.6 Sodium hydroxide, $c(\text{NaOH}) \approx 2$ mol/l.

Weigh 80 g of sodium hydroxide pellets and add to 800 ml water (4.1) in a conical flask. Stir continuously until all pellets are dissolved. Wait until the solution has cooled to room temperature and transfer this solution to a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix well.

4.7 Sodium hypochlorite, solution, $\rho(\text{Cl}_2)$ about 0,1 g/l.

Prepare by dilution of concentrated commercial sodium hypochlorite solution.

4.8 Potassium iodate, stock solution, $\rho(\text{KIO}_3) = 1,006$ g/l.

Dissolve 1,006 g of potassium iodate (KIO_3) in about 250 ml water (4.1) in a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix.

4.9 Potassium iodate, standard solution, $\rho(\text{KIO}_3) = 10,06$ mg/l.

Take 10 ml of stock solution (4.8), place in a 1 000 ml one-mark volumetric flask, add about 1 g potassium iodide (4.4) and make up to the mark with water (4.1).

Prepare this solution on the day of use.

1 ml of this standard solution contains 10,06 μg of KIO_3 .

10,06 μg of KIO_3 is equivalent to 0,141 μmol Cl_2 .

4.10 Sodium arsenite (NaAsO_2), solution, 2 g/l; or thioacetamide (CH_3CSNH_2), solution, 2,5 g/l.

5 Apparatus

Ordinary laboratory apparatus, and

Colorimetric equipment, comprising one of the following:

5.1 Comparator, equipped with a scale of permanent glass colour standards specially set up for the DPD technique and suitable for concentrations from 0,000 4 to 0,07 mmol/l (0,03 to 5 mg/l) of chlorine.

5.2 Spectrometer, with a selector for continuous wavelength variation, suitable for use at 510 nm and equipped with rectangular cells with an optical path length of 10 mm or greater.

5.3 Spectrometer, with a selector for discontinuous wavelength variation, having its maximum transmission as near as possible to 510 nm and rectangular cells with an optical path length of 10 mm or greater.

NOTE ON THE PREPARATION OF GLASSWARE

Chlorine-demand-free glassware is obtained by filling with sodium hypochlorite solution (4.7) then, after 1 h, rinsing copiously with water (4.1). During the course of the analysis one set of glassware should be kept for the determination of free chlorine and another for the determination of total chlorine in order to avoid contamination of the free chlorine set.

6 Procedure

6.1 Test sample

Start determination immediately after taking samples. At all times avoid bright light, agitation and heat.

6.2 Test portions

Take two test portions, each of 100,0 ml (V_0). If the concentration of total chlorine exceeds 70 $\mu\text{mol/l}$ (5 mg/l) it is necessary to take a smaller volume, V_1 , of test sample and to dilute with water (4.1) to 100,0 ml.

6.3 Calibration

Place in a series of 100 ml one-mark volumetric flasks, increasing quantities of the potassium iodate standard solution (4.9) in such a way as to set up a scale extending from $c(\text{Cl}_2) = 0,423$ up to 70,5 $\mu\text{mol/l}$ [$\rho(\text{Cl}_2) = 0,03$ up to 5 mg/l; 0,3 up to 50 ml of standard solution (4.9)]. Add 1,0 ml sulfuric acid (4.5) and after 1 min, 1,0 ml of sodium hydroxide solution (4.6). Dilute to 100 ml with water (4.1). Transfer the contents of each flask, without rinsing, into a 250 ml conical flask containing 5 ml buffer solution (4.2) and 5 ml DPD reagent (4.3), added less than 1 min prior to the transfer, and mix (see the note). Then fill the measuring cell successively with each of the prepared standard matching solutions and measure within 2 min one of the following:

- the colour intensity with the comparator (5.1),
- the absorbance, against water in the reference cell, with a spectrometer (5.2 or 5.3).

As required, check and make any necessary corrections to the comparator scale of standards or prepare a calibration graph for the spectrometer. Carry out a calibration for each fresh preparation of DPD reagent and check daily one point on the scale or on the graph.

NOTE — Prepare each standard matching solution separately to avoid the mixture of buffer and reagent standing too long in advance and the appearance of a false red colour.

6.4 Determination of free chlorine

Transfer the first test portion, without rinsing, to a 250 ml conical flask containing 5 ml of buffer solution (4.2) and 5 ml of DPD reagent (4.3) and mix (see the note to 6.3). Fill the measuring cell with this treated solution and immediately measure the colour under the same conditions as adopted for the calibration (6.3). Record c_1 , the concentration reading from the comparator scale or calibration graph (6.3).

In the case of an unknown water, possibly being very acid, or very alkaline or with a high concentration of salts, it is advisable to verify that the volume of buffer solution (4.2) added is sufficient to bring the water to pH 6,2 to 6,5. If not, use a greater volume of the buffer solution (4.2).

6.5 Determination of total chlorine

Transfer the second test portion, without rinsing, to a 250 ml conical flask containing 5 ml of buffer solution (4.2) and 5 ml of DPD reagent (4.3), add about 1 g of potassium iodide (4.4) and mix (see the note to 6.3). Fill the measuring cell with this treated solution and after 2 min measure the colour under the same conditions as adopted for the calibration (6.3). Record c_2 , the concentration reading from the comparator scale or calibration graph (6.3).

In the case of an unknown water, possibly being very acid, or very alkaline or with a high concentration of salts, it is advisable to verify that the volume of buffer solution (4.2) added is sufficient to bring the water to pH 6,2 to 6,5. If not, use a greater volume of the buffer solution (4.2).

7 Correction of interference due to the presence of oxidized manganese

Determine the effect of oxidized manganese by carrying out a supplementary determination on a further test portion (6.2) previously treated with the arsenite or thioacetamide solution (4.10) in order to neutralize all oxidizing compounds other than oxidized manganese.

Place this test portion in a 250 ml conical flask, add 1 ml of sodium arsenite solution (4.10) or thioacetamide solution (4.10) and mix. Again add 5,0 ml of buffer solution (4.2) and 5,0 ml of DPD reagent (4.3) and mix.

Fill the measuring cell with this treated solution and immediately measure the colour under the same conditions as adopted for the calibration (6.3). Record c_3 , the concentration reading from the comparator scale or calibration graph (6.3), corresponding to the oxidized manganese present.

In using comparators with permanent glass colour standards the arsenite or thioacetamide treated sample may be used as a blank to compensate for any interference colour so long as the time of addition of reagents is the same for both blank and sample.