SLOVENSKI STANDARD
oSIST prEN ISO 7393-2:2017

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

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/DIS 7393-2)

Wasserbeschaffenheit - Bestimmung von freiem Chlor und Gesamtchlor - Teil 2: Kolorimetrisches Verfahren mit N,N-Diethyl-1,4-Phenylendiamin für Routinekontrollen

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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13.060.50	Preiskava vode na kemične snovi	Examination of water for chemical substances

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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

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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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

This second edition cancels and replaces the first edition (ISO 7393-2:1985), which has been technically revised.

A new Annex C (informative) has been included with the title: Disposable planar reagent-filled cuvettes using a mesofluidic channgel pump/colorimeter.

A list of all parts in the ISO 7393 series can be found on the ISO website.

Water quality — Determination of free chlorine and total chlorine —

Part 2:

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

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

IMPORTANT — It is essential that tests conducted in accordance with this document be carried out by suitably qualified staff.

1 Scope

This document specifies a method for the determination of free chlorine and total chlorine in water, readily applicable to lab- and field-testing; it is based on measurement of the absorption the red DPD colour complex in a photometer or the colour intensity by visual comparison of the colour with a scale of standards that is regularly calibrated.

Seawater and waters containing bromides and iodides comprise a group for which special procedures are required.

This method is in practice applicable to concentrations, in terms of chlorine (Cl_2), from e.g. 0,000 4 mmol/l to 0,07 mmol/l (e.g. 0,03 mg/l to 5 mg/l) total chlorine. For higher concentrations, the test portion must be diluted.

Commonly, the method is applied as a field method with mobile photometers and commercially available ready-for-use reagents (liquid reagents, powders and pellets). It is essential that those reagents fully comply with minimum requirements and contain the essential reagents and a buffer system suitable to adjust the measurement solution to a pH range of 6,2 to 6,5. If there is doubt that water samples have uncommon pH values and/or buffer capacities the user has to check and if necessary to adjust the sample pH to the required range. The pH of the sample shall be within the range of pH 4 and 8. Adjust, if necessary, with sodium hydroxide solution or sulfuric acid before the test.

A procedure 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 is presented in Annex A. In Annex C, a procedure is presented for the determination of free and total chlorine in drinking and other low polluted waters, for disposable planar reagent-filled cuvettes using a mesofluidic channel pump/colorimeter.

2 Normative References

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 5667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes and sampling techniques*

ISO 5667-3, *Water quality — Sampling — Part 3: Preservation and handling of water samples*

ISO 8466-1, *Water quality — Calibration and evaluation of analytical methods and estimation of performance characteristics — Part 1: Statistical evaluation of the linear calibration function*

3 Terms and definitions

See also Table 1.

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org./obp

3.1
free chlorine
chlorine present in the form of hypochlorous acid, hypochlorite ion or dissolved elemental chlorine

3.2
combined chlorine
fraction of total chlorine present in the form of chloramines and organic chloramines

3.3
total chlorine
chlorine present in the form of “free chlorine” and “combined chlorine”

3.4
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 document

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

4 Principle

4.1 Determination of free chlorine

Direct reaction with the *N*,*N*-*dialkyl*-1,4-*phenylenediamine* (DPD) and formation of a red colour complex at pH 6,2 to 6,5. Measurement of the colour intensity by photometry, or alternatively by visual comparison of the colour with a scale of permanent glass, plastics standards or colour card comparators.

4.2 Determination of total chlorine

Reaction with DPD in the presence of an excess of potassium iodide then measurement as in 4.1.

5 Interferences

5.1 Interference by other chlorine compounds

Chlorine dioxide that might be present in the sample in addition to chlorine is measured as total chlorine. This interference may be corrected by specific determining of chlorine dioxide in the water (see References[2],[4] and[5]).

If chlorine dioxide is present in the sample as the only disinfectant it may be measured with the DPD method described in this standard with the appropriate conversion factor. Other chlorine compounds do not specifically cause oxidation of DPD.

5.2 Interference by compounds other than chlorine compounds

Depending on the concentration and the chemical oxidation potential, other oxidizing agents effect the reaction, for example: bromine, iodine, bromamines, iodoamines, ozone, hydrogen peroxide, chromate, oxidized manganese, nitrite, iron(III) ions, peracetic acid and copper ions. The interference from Cu(II) (< 8 mg/l) and iron (< 20 mg/l) is suppressed by the disodium EDTA in reagents 6.2 and 6.3.

NOTE Bromine and monobromamine contribute to the disinfection effect and regularly occur in chlorine based disinfection products.

lnterference by chromate may be eliminated by addition of excess barium chloride. The user has to validate how to cope with this interference.

5.3 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 (9.2) previously treated with the arsenite or thioacetamide solution (6.10) 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 (6.10) and mix. Again add 5,0 ml of buffer solution (6.2) and 5,0 ml of DPD reagent (6.3) and mix.

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

In using comparators with permanent glass colour standards or plastics standards or colour card comparators 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.

5.4 Interference due to turbid and coloured samples

If samples are turbid or in the case precipitation occurs due to the addition of the buffer solution they have to be filtered. The filtration equipment and the filter material must be chlorine demand free. This has to be checked accordingly. See 7.3 for a procedure to prepare the glassware.

In the case of low turbidity or if samples are coloured a blank value matching can help to overcome the interference.

6 Reagents

During the analysis, use only reagents of recognized analytical grade, and only water as specified in 6.1.

6.1 Water as specified in ISO 3696, grade 2, 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 7) place, in order,

a) in the first: 100 ml of the water to be checked and about 1 g of potassium iodide (6.4); mix and after 1 min add 5 ml of buffer solution (6.2) and 5,0 ml of DPD reagent (6.3);

b) in the second: 100 ml of the water to be checked and two drops of sodium hypochlorite solution (6.7); then, after 2 min, 5,0 ml of buffer solution (6.2) and 5 ml of DPD reagent (6.3).

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.

6.1.1 Procedure to produce water free from oxidizing and reducing substances

To obtain dilution water of the desired quality, demineralized or distilled water is first chlorinated to a level of about 0,14 mmol/l (10 mg/l) and stored in a well-stoppered carboy for at least 16 h. The water is then dechlorinated by exposure to UV irradiation, sunlight for several hours or by contact with active carbon. Finally check the quality using the procedures as given in Clause 9. The user has to make sure that also the glassware is chlorine demand free. The procedure is described in 7.3.

6.2 Buffer solution, pH 6,5.

Dissolve in water (6.1) in this order: 24 g of anhydrous disodium hydrogen phosphate (Na_2HPO_4) or 60,5 g of the dodecahydrate form ($Na_2HPO_4 \cdot 12H_2O$) and 46 g of potassium dihydrogen phosphate (KH_2PO_4). Add 100 ml of 8 g/l disodium dihydrogenethylenedinitrilotetraacetate dihydrate (disodium EDTA dihydrate, $C_{10}H_{14}N_2O_8Na_2 \cdot 2H_2O$) 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. The buffer solution is stable for up to three months if stored in a tightly sealed container in the dark. In reference to guaranteed stability of ready-to-use buffer solutions, see recommendations of the manufacturer.

The buffer solution is an essential part for a proper reaction of DPD with chlorine. Therefore, this buffer system also applies to the various reagents provided in ready-to-use test kits. Such ready-to-use test kits are commonly intended for a certain range of buffer capacities in the samples. Therefore, the buffer capacity of the test kit may be too low. Therefore, the user should make sure that the pH of the final reagent sample mix lies between 6.2 and 6.5. To prevent contamination of the sample, pH checks may be carried out using a pH-meter or non-bleeding pH-test strips. If necessary, samples should be adjusted to the correct pH range using hydrochloric acid or sodium hydroxide solution. If there is no information on the buffer used or the buffer capacity of the test kit the manufacturer of the test kit shall not refer to this document.

Solutions containing mercury should be disposed of safely.

6.3 *N,N-dialkyl*-1,4-*phenylenediamine* sulfate (DPD), [$NH_2\text{-}C_6H_4\text{-}N(C_2H_5)_{2/3} \cdot H_2SO_4$], solution, 1,1 g/l.

The DPD reagent is commercially available. It is available from numerous sources and the usage is handy especially when tests are carried out with test kits on site. It is essential that commercially available DPD reagents contain suitable amounts of acid and EDTA as well as a suitable DPD concentration. If ready-to-use DPD regents are used, evidence has to be given that the composition is as suitable as the formulation given below. If there is no such information the manufacturer of the ready-to-use test kit shall not refer to this document.