

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

*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*

First edition — 1985-10-15

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UDC 543.3 : 543.43 : 546.13

Ref. No. ISO 7393/2-1985 (E)

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

Foreword

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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.

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,

- 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);
- 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.

8 Expression of results

8.1 Method of calculation

8.1.1 Calculation of the free chlorine concentration

The concentration of free chlorine, $c(\text{Cl}_2)$, expressed in millimoles per litre, is given by the equation

$$c(\text{Cl}_2) = \frac{(c_1 - c_3) V_0}{V_1}$$

where

c_1 is the concentration, expressed in millimoles of Cl_2 per litre, of chlorine as determined in 6.4;

c_3 is the concentration, expressed in millimoles of Cl_2 per litre, corresponding to the oxidized manganese present (see clause 7);

NOTE — If oxidized manganese is absent, $c_3 = 0$.

V_0 is the maximum volume, in millilitres, of the test portion (6.2) ($V_0 = 100,0$ ml);

V_1 is the volume, in millilitres, of test sample in the test portion (6.2).

8.1.2 Calculation of the total chlorine concentration

The concentration of total chlorine, $c(\text{Cl}_2)$, expressed in millimoles per litre, is given by the equation

$$c(\text{Cl}_2) = \frac{(c_2 - c_3) V_0}{V_1}$$

where

c_2 is the concentration, expressed in millimoles of Cl_2 per litre, of chlorine as determined in 6.5;

c_3 , V_0 and V_1 are as defined in 8.1.1.

8.2 Conversion of amount of substance concentration to mass concentration

The chlorine concentration expressed in moles per litre may be expressed in grams per litre by multiplying by a conversion factor of 70,91.

8.3 Repeatability and reproducibility

To obtain an indication of repeatability and reproducibility, figures are taken from measurements obtained by methods which are the same in principle as that specified in this part of ISO 7393.

The USA-EPA Environmental Monitoring and Support Laboratory^[1] evaluated the colorimetric method with the following results.

For distilled water samples at concentrations of $c(\text{Cl}_2) = 5,5$ and $50,9 \mu\text{mol/l}$ [$\rho(\text{Cl}_2) = 0,39$ and $3,61 \text{ mg/l}$] total chlorine, the relative standard deviations were 3,1 %, and 3,2 %, respectively. Using drinking water containing $c(\text{Cl}_2) = 13,3 \mu\text{mol/l}$ [$\rho(\text{Cl}_2) = 0,94 \text{ mg/l}$] total chlorine, the relative standard deviation was 0,8 %. With river water at a concentration of $c(\text{Cl}_2) = 12,1 \mu\text{mol/l}$ [$\rho(\text{Cl}_2) = 0,86 \text{ mg/l}$] total chlorine and domestic sewage at a concentration of $c(\text{Cl}_2) = 15,1 \mu\text{mol/l}$ [$\rho(\text{Cl}_2) = 1,07 \text{ mg/l}$] total chlorine, the relative standard deviations were 1,9 % and 2,4 %, respectively.

Results published by the British Department of the Environment^[2] based on data obtained at the Water Research Centre^[3] showed for free chlorine concentrations of $c(\text{Cl}_2) = 1,4$ and $7,1 \mu\text{mol/l}$ [$\rho(\text{Cl}_2) = 0,1$ and $0,5 \text{ mg/l}$], relative standard deviations of 4,0 % and 2,0 %, respectively.

The results presented in the preceding paragraphs relate to replicate determinations in the same laboratory and thus provide a measure of the repeatability of the method. Attempts in the past to quantify the reproducibility of the method by distribution of samples to different laboratories have produced unreliable results because of a general instability of solutions containing free and combined chlorine. More recently it has been found by the Quality Assurance Branch of EMSL-Cincinnati^[6] that a sealed vial of sodium hypochlorite in very

Table 2 — Analytical parameters from interlaboratory analysis for free residual chlorine

True value		Method code ¹⁾	Number of laboratories (observations)	Mean		Standard deviation	
$c(\text{Cl}_2)$	$\rho(\text{Cl}_2)$			$\mu\text{mol/l}$	(mg/l)	$\mu\text{mol/l}$	(mg/l)
7	(0,5)	A	6	6,2	(0,44)	1,3	(0,09)
		B	7	6,8	(0,48)	1,8	(0,13)
11,3	(0,80)	A	10	10,9	(0,77)	1,1	(0,08)
		B	14	11,1	(0,79)	4,1	(0,29)
		C	6	11,6	(0,82)	1,3	(0,09)
15,5	(1,10)	A	10	15,5	(1,10)	2,0	(0,14)
		B	14	16,2	(1,15)	5,5	(0,39)
		C	6	16,5	(1,17)	1,1	(0,08)
18,2	(1,29)	A	6	18,6	(1,32)	1,3	(0,09)
		B	7	19,9	(1,41)	5,4	(0,38)

1) A: Iodometric titration amperometric

B: DPD colorimetric

C: DPD titrimetric

pure water is remarkably stable when stored in the dark inside a mailing tube. Subsequent evaluation by various US federal and state laboratories has produced the analytical parameters listed in table 2 for methods in current use.

9 Interferences

Two types of interference may be noted.

9.1 Interference by other chlorine compounds

A fraction of any chlorine dioxide that might be present is measured as total chlorine. This interference may be corrected by determining the chlorine dioxide in the water.^[2, 4, 5]

9.2 Interference by compounds other than chlorine compounds

Oxidation of DPD is not specifically caused by chlorine compounds. Depending on the concentration and the chemical oxidation potential, the reaction is effected by other oxidizing agents. The following substances may be mentioned in particular: bromine, iodine, bromamines, iodoamines, ozone,

hydrogen peroxide, chromate, oxidized manganese, nitrite, iron(III) ions and copper ions. The interference is suppressed by the disodium EDTA in reagents 4.2 and 4.3 in the case of copper(II) ions ($< 8 \text{ mg/l}$) and iron(III) ions ($< 20 \text{ mg/l}$).

Interference by chromate may be eliminated by addition of barium chloride.^[7]

10 Test report

The test report shall include the following information:

- a) a reference to this part of ISO 7393;
- b) all information necessary for complete identification of the sample;
- c) the results and the method of expression used;
- d) details of any operations not included in this part of ISO 7393, or regarded as optional, together with any circumstance that may have affected the results.

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Annex A

Separate determinations of combined chlorine of the monochloramine type, combined chlorine of the dichloramine type and of combined chlorine in the form of nitrogen trichloride

A.1 Applicability

This annex specifies a method for the differentiation between combined chlorine of the monochloramine type, combined chlorine of the dichloramine type and combined chlorine in the form of nitrogen trichloride. The field of application of the method is the same as that for concentrations of free chlorine and total chlorine (see clause 1).

A.2 Principle

After determination of free chlorine and total chlorine, colorimetric measurement of two further test portions:

- on the third test portion: reaction with DPD limited to free chlorine and to combined chlorine of the monochloramine type by the addition of a small quantity of potassium iodide;
- on the fourth test portion, by addition of a small quantity of potassium iodide before the addition of buffer and DPD reagent: reaction with DPD by free chlorine, by combined chlorine of the monochloramine type and one half of the nitrogen trichloride.

Combined chlorine of the dichloramine type does not react in either of these two cases. Calculation of the concentration of combined chlorine of the monochloramine and dichloramine types and the concentration of nitrogen trichloride.

A.3 Reagents

The reagents given in clause 4 and

Potassium iodide, solution, 5 g/l.

Prepare this solution on the day of use and store in a brown bottle.

A.4 Apparatus

See clause 5.

A.5 Procedure

A.5.1 Test sample

See 6.1.

A.5.2 Test portions

Work on two test portions similar to those in 6.2.

A.5.3 Calibration

See 6.3.

A.5.4 Determination of free chlorine and combined chlorine of the monochloramine type

Place rapidly in a 250 ml conical flask in this order: 5,0 ml of buffer solution (4.2), 5,0 ml of DPD reagent (4.3), the third test portion and two drops (about 0,1 ml) of potassium iodide solution (clause A.3) or a very small crystal of potassium iodide (about 0,5 mg) and mix. Fill the measuring cell with this treated solution and immediately measure the colour under the same conditions as adopted for the calibration (A.5.3). Record c_4 , the concentration reading from the comparator scale or calibration graph (A.5.3).

A.5.5 Determination of free chlorine, combined chlorine of the monochloramine type and one half of the nitrogen trichloride

Place in a 250 ml beaker, the fourth test portion and two drops (about 0,1 ml) of potassium iodide solution (clause A.3) or a very small crystal of potassium iodide (about 0,5 mg) and mix. Transfer the contents of the beaker to a 250 ml conical flask containing 5,0 ml of buffer solution (4.2) and 5,0 ml of DPD reagent (4.3) added less than 1 min prior to the transfer. Fill the measuring cell with this treated solution and immediately measure the colour under the same conditions as adopted for the calibration (A.5.3). Record c_5 , the concentration reading from the comparator scale or calibration graph (A.5.3).

A.6 Expression of results

A.6.1 Method of calculation

A.6.1.1 Calculation of the concentration of combined chlorine of the monochloramine type

The concentration of combined chlorine of the monochloramine type, $c(\text{Cl}_2)$, expressed in millimoles per litre, is given by the equation

$$c(\text{Cl}_2) = \frac{(c_4 - c_1)V_0}{V_1}$$

where

c_4 is the concentration, expressed in millimoles of Cl_2 per litre, of chlorine as determined in A.5.4;

c_1 , V_0 and V_1 are as defined in clause 8.1.

A.6.1.2 Calculation of the concentration of combined chlorine of the dichloramine type

The concentration of combined chlorine of the dichloramine type, $c(\text{Cl}_2)$, expressed in millimoles per litre is given by the equation

$$c(\text{Cl}_2) = \frac{(c_2 - 2c_5 + c_4) V_0}{V_1}$$

where

c_2 , V_0 and V_1 are as defined in 8.1;

c_4 is as defined in A.6.1.1;

c_5 is the concentration, expressed in millimoles of Cl_2 per litre, of chlorine as determined in A.5.5.

A.6.1.3 Calculation of the concentration of combined chlorine in the form of nitrogen trichloride

The concentration of combined chlorine in the form of nitrogen trichloride, $c(\text{Cl}_2)$, expressed in millimoles per litre, is given by the equation

$$c(\text{Cl}_2) = \frac{2 (c_5 - c_4) V_0}{V_1}$$

where

c_5 is as defined in A.6.1.2;

c_4 is as defined in A.6.1.1;

V_0 and V_1 are as defined in 8.1.

A.6.2 Conversion of amount of substance concentration to mass concentration

The chlorine concentration expressed in moles per litre may be expressed in grams per litre by multiplying by a conversion factor of 70,91.