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Water quality — Determination of free chlorine and total chlorine —

Part 3:

**Iodometric titration method for the determination of
total chlorine**

Qualité de l'eau — Dosage du chlore libre et du chlore total —

Partie 3: Méthode par titrage iodométrique pour le dosage du chlore total



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Foreword

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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 7393-3 was prepared by Technical Committee ISO/TC 147, *Water quality*.

This second edition cancels and replaces the first edition (ISO 7393-3 : 1986), of which it constitutes a minor revision.

ISO 7393 consists of the following parts, under the general title *Water quality — Determination of free chlorine and total chlorine*:

- *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*

Annex A forms an integral part of this part of ISO 7393. Annexes B and C are for information only.

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Water quality — Determination of free chlorine and total chlorine —

Part 3:

Iodometric titration method for the determination of total chlorine

1 Scope

This part of ISO 7393 specifies an iodometric titration method for the determination of total chlorine in water.

The method is applicable for the measurement of concentrations in terms of chlorine (Cl_2), from 0,01 mmol/l to 0,21 mmol/l (0,71 mg/l to 15 mg/l).

Several substances interfere in the determination (see clause 10).

In annex B a method for direct titration is specified. This is usually applied to the determination of chlorine concentrations above 7 $\mu\text{mol/l}$ (0,5 mg/l) in treated drinking water.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 7393. 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 7393 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 : 1982, *Water quality — Sampling — Part 2: Guidance on sampling techniques.*

3 Definitions (see table 1)

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

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

3.2 combined chlorine: The 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" or "combined chlorine" or both.

3.4 chloramines: Derivatives of ammonia by substitution of one, two or three hydrogen atoms with chlorine atoms (monochloramine NH_2Cl , dichloramine NHCl_2 , and nitrogen trichloride NCl_3) and all chlorinated derivatives of organic nitrogen compounds.

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

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

4 Principle

Reaction in acid solution of total chlorine and potassium iodide with liberation of free iodine. Instantaneous reduction of the iodine by a known excess of thiosulfate standard solution previously added to the solution. Titration of the unreacted thiosulfate with potassium iodate standard reference solution.

5 Reagents

During the analysis, use only reagents of recognized analytical grade, and water as specified in 5.1.

5.1 Water, free from chlorine and reducing substances.

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

Into two 250 ml chlorine-demand free conical flasks (see clause 6) place, in order,

a) in the first: 100 ml of the water to be checked, and about 1 g of potassium iodide (5.2), 2 ml of phosphoric acid (5.3) and 1 ml of starch solution (5.6);

b) in the second: 100 ml of the water to be checked, about 1 g of potassium iodide (5.2), 2 ml of phosphoric acid (5.3) and 1 ml of starch solution (5.6). Add 10 drops of 0,1 g/l sodium hypochlorite solution (obtained by dilution of commercial concentrated sodium hypochlorite solution, of which the concentration is determined iodometrically).

No coloration shall appear in the first flask when the water is free from chlorine. In the second flask a light blue colour shall appear.

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 final quality has to be rechecked.

A procedure for chlorination followed by dechlorination is given in annex A.

5.2 Potassium iodide (KI) crystals.

5.3 Phosphoric acid, solution, $c(\text{H}_3\text{PO}_4) \approx 0,87 \text{ mol/l}$.

Dissolve 64 ml of phosphoric acid ($\rho = 1,69 \text{ g/ml}$) in water, cool and dilute to 1 000 ml.

5.4 Potassium iodate, standard reference solution, $c(1/6 \text{ KIO}_3) = 10 \text{ mmol/l}$.

Weigh, to the nearest milligram, 0,360 g of dry potassium iodate. Dissolve in water in a 1 000 ml one-mark volumetric flask, make up to the mark with water and mix.

5.5 Sodium thiosulfate, standard volumetric solution, $c(\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}) \approx 10 \text{ mmol/l}$.

5.5.1 Preparation

Dissolve about 2,48 g of sodium thiosulfate in about 250 ml of water in a 1 000 ml one-mark volumetric flask. Make up to the mark with water and mix.

5.5.2 Standardization

Standardize the solution daily, or immediately prior to use, as follows.

Place 200 ml of water (5.1) in a 500 ml conical flask. Add about 1 g of potassium iodide (5.2) and then introduce, by means of a pipette, 10,0 ml of sodium thiosulfate solution (5.5.1) for stan-

dardization, 2 ml of phosphoric acid (5.3) and 1 ml of starch solution (5.6). Titrate immediately with the potassium iodate standard reference solution (5.4) until the appearance of a blue coloration persisting for at least 30 s occurs. Note the volume of iodate consumed.

The actual concentration, c_1 , expressed in millimoles per litre, of the sodium thiosulfate solution is given by the equation

$$c_1 = \frac{V_2 c_2}{V_1}$$

where

c_2 is the concentration, expressed in millimoles per litre, of the potassium iodate standard reference solution [$c(1/6 \text{ KIO}_3) = 10 \text{ mmol/l}$];

V_1 is the volume, in millilitres, of sodium thiosulfate solution (5.5.1) used for the standardization ($V_1 = 10 \text{ ml}$);

V_2 is the volume, in millilitres, of potassium iodate standard reference solution used in the titration.

5.6 Starch, 5 g/l solution, or similar commercial indicator.

6 Apparatus

Usual laboratory equipment and

Burette, with fine tip, permitting the delivery of about 30 drops/ml, measuring up to 25 ml, graduated in divisions of 0,05 ml.

NOTE ON PREPARATION OF GLASSWARE

Chlorine-demand free glassware is obtained by filling with 0,1 g/l sodium hypochlorite then, after 1 h, rinsing thoroughly with distilled water and with water free from chlorine-demand.

7 Sampling and samples

See ISO 5667-1 and ISO 5667-2.

8 Procedure

8.1 Test portion

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

Take a test portion of volume not greater than 200 ml and containing not more than 0,21 mmol/l (15 mg/l) of total chlorine. If the amount of total chlorine is expected to exceed this concentration, dilute the sample with water (5.1) and take a test portion of a volume not exceeding 200 ml.

8.2 Determination

Place the test portion (8.1) in a 500 ml conical flask. Add, in order, about 1 g of potassium iodide (5.2), 2 ml of phosphoric



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