BRITISH STANDARD

BS EN ISO 3696:1995

Incorporating Amendment No. 1

Water for analytical laboratory use — Specification and test methods

The European Standard EN ISO 3696:1995 has the status of a British Standard

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Chemicals Standards Committee (CIC/-) to Technical Committee CIC/26, upon which the following bodies were represented:

British Pharmacopoeia Commission
Chemical Industries Association
Institute of Petroleum
Milk Marketing Board
Royal Society of Chemistry
Society of Glass Technology
Textile Institute

The following bodies were also represented in the drafting of the standard, through subcommittees and panels:

British Ceramic Research Association
British Effluent and Water Association
Standardization of Tar Products Tests Committee

This British Standard, having been prepared under the direction of the Chemicals Standards Committee, was published under the authority of the Board of BSI and comes into effect on
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The following BSI references relate to the work on this standard:
Committee reference CIC/26
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Amendments issued since publication

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8807	September 1995	Indicated by a sideline in the margin

Contents

National foreword

This British Standard has been prepared under the direction of the Chemicals Standards Committee. It is identical with ISO 3696:1987 "*Water for analytical laboratory use — Specification and test methods*", published by the International Organization for Standardization (ISO).

In 1995 the European Committee for Standardization (CEN) accepted ISO 3696:1987 as European Standard EN ISO 3696. As a consequence of implementing the European Standard this British Standard is renumbered as BS EN ISO 3696 and any reference to BS 3978 should be read as a reference to BS EN ISO 3696.

This standard is a revision of BS 3978:1966, which is withdrawn. The principal changes in this edition are as follows.

a) The single grade of water has been replaced by three grades, of which grade 3 is roughly equivalent to the water specified in the previous edition.

b) Residue on ignition is no longer specified and the requirement (for grade 3 water) for residue on evaporation has been tightened.

c) Electrical conductivity is now measured at 25 °C instead of 20 °C, and the requirement for grade 3 water has been changed from 1 mS/m to 0.5 mS/m.

d) The limit for oxidizable matter for grade 3 water has been tightened.

e) Requirements for ammonia content, chloride content and sulphate content are no longer included.

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

The symbol "l" has been used to denote litre (and in its submultiples). In British Standards it is current practice to use the symbol "L".

The spelling "sulfur", etc. has been used. In British Standards it is current practice to use the spelling "sulphur", etc.

Wherever the words "International Standard" appear, referring to this standard, they should be read as "British Standard".

Additional information. If water complying with BS 3978 is specified in another British Standard without stating which grade should be used, it is recommended that grade 3 be used (see clause **3**).

Textual errors. When adopting the text of the International Standard, the textual errors listed below were discovered. They have been marked in the text and have been reported to ISO in a proposal to amend the text of the International Standard.

a) In the note to clause **5** the cross-reference should read "see note 2 to clause **4**".

b) In paragraph 1, line 3 of **7.6.1.1**, the reference should be to **7.6.2.1** and not **7.5.2.3**.

In paragraph 1, line 4 of **7.6.1.1**, "mix the sample with a dry" should read "mix with the sand using a dry".

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, the EN ISO title page, pages 2 to 6 and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 3696

April 1995

ICS 71.040.30

Descriptors: Inorganic chemistry, chemical analysis, chemical reagents, water, classification, specification, tests, storage

English version

Water for analytical laboratory use — Specification and test methods

(ISO 3696:1987)

Eau pour laboratoire à usage analytique — Spécification et méthodes d'essai
(ISO 3696:1987)

Wasser für analytische Laborzwecke-Spezifikation und Prüfverfahren
(ISO 3696:1987)

This European Standard was approved by CEN on 1995-02-13. 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.

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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

Foreword

The text of the International Standard has been taken as a European Standard by the Technical Committee CEN/TC 19, Petroleum products, lubricants, and related products, from ISO/TC 47, Chemistry, of the International Organization for Standardization (ISO).

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 October 1995, and conflicting national standards shall be withdrawn at the latest by October 1995.

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

1 Scope and field of application

This International Standard specifies the requirements and corresponding test methods for three grades of water for laboratory use for the analysis of inorganic chemicals.

It is not applicable to water for organic trace analysis, to water for the analysis of surface active agents, or to water for biological or medical analysis.

NOTE For some purposes (for example for certain analytical methods or for tests in which the water is required to be sterile or pyrogen-free or of specified surface tension), additional specific tests and further purification or other treatment may be necessary.

2 Description

The material shall be a clear, colourless liquid as assessed by visual inspection.

3 Classification

This International Standard covers three grades of water as follows:

Grade 1

Essentially free from dissolved or colloidal ionic and organic contaminants and suitable for the most stringent analytical requirements including those of high-performance liquid chromatography; should be produced by further treatment of grade 2 water (for example reverse osmosis or deionization followed by filtration through a membrane filter of pore size 0,2 µm to remove particulate matter or redistillation from a fused silica apparatus).

Grade 2

Very low in inorganic, organic or colloidal contaminants and suitable for sensitive analytical purposes, including atomic absorption spectrometry (AAS) and the determination of constituents in trace quantities; should be produced, for example, by multiple distillation, or by deionization or reverse osmosis followed by distillation.

Grade 3

Suitable for most laboratory wet chemistry work and preparation of reagents solutions; should be produced, for example, by single distillation, by deionization, or by reverse osmosis. Unless otherwise specified, it should be used for ordinary analytical work.

NOTE It is assumed that the initial feed stock water is potable and reasonably pure. If it is heavily contaminated in any respect, some pretreatment may be necessary.

4 Requirements

The material shall comply with the appropriate requirements of the Table. Testing for compliance shall be carried out by means of the methods specified in clause **7**.

5 Sampling

A representative sample of the material of not less than 2 litres shall be taken from the bulk for the purpose of examination in accordance with this specification.

NOTE This sample is not used for measurements of conductivity on grade 1 and grade 2 water (see **7.2.2.1**)[1)].

The sample shall be placed in a suitable, clean, airtight container, reserved solely for water samples, which shall be of such a size that it is completely filled by the sample. Care shall be taken to avoid the risk of contaminating the contents in any way.

Aged containers (i.e. containers boiled for at least 2 h in a solution of hydrochloric acid, c(HCl) ≈ 1 mol/l, then twice for 1 h in distilled water) made of borosilicate glass can be used as well as suitable inert plastics containers (for example polyethylene or polypropylene), but it is essential to establish that the sample is unaffected by storage therein, particularly in respect of oxidizable matter and absorbance.

6 Storage

Contamination of water during storage may arise principally from dissolution of soluble constituents from glass or plastics containers or absorption of atmospheric carbon dioxide and of any other impurities present in the laboratory atmosphere.

For this reason, the storage of grade 1 and grade 2 water is not recommended, the water being prepared, as required, for immediate use.

Nevertheless, grade 2 water may be prepared in reasonable quantity and stored in suitable, inert, clean, airtight, full containers which have been thoroughly pre-rinsed with water of similar grade.

The storage of grade 3 water presents little problem, but the containers and storage conditions should be the same as those used for the storage of grade 2 water.

It is recommended that any storage container be reserved exclusively for the storage of a particular grade of water.

[1)] See national foreword for details of textual error.

Table — Requirements

Parameter	Grade 1	Grade 2	Grade 3	Test method
pH value at 25 °C inclusive range	Not applicable (see note 1)	Not applicable (see note 1)	5,0 to 7,5	Sub-clause **7.1**
Electrical conductivity mS/m at 25 °C, max.	0,01 (see note 2)	0,1 (see note 2)	0,5	Sub-clause **7.2**
Oxidizable matter Oxygen (O) content mg/l, max	Not applicable (see note 3)	0,08	0,4	Sub-clause **7.3**
Absorbance at 254 nm and 1 cm optical path length, absorbance units, max.	0,001	0,01	Not specified	Sub-clause **7.4**
Residue after evaporation on heating at 110 °C mg/kg, max.	Not applicable (see note 3)	1	2	Sub-clause **7.5**
Silica (SiO_2) content mg/l, max.	0,01	0,02	Not specified	Sub-clause **7.6**

NOTE 1 Because of the difficulties associated with measurement of the pH value of high-purity water, and the doubtful significance of the value obtained, limits for the pH of grade 1 and grade 2 water have not been specified.

NOTE 2 The values of conductivity for grade 1 and grade 2 water apply to freshly prepared water; during storage, it is possible for contaminants such as atmospheric carbon dioxide and alkalis from glass containers to be dissolved, leading to changes in conductivity.

NOTE 3 A limit for oxidizable matter and residue after evaporation for grade 1 water is not specified because of the difficulty of testing for compliance at this level of purity. The quality of grade 1 water is, however, assured by compliance with the other requirements and by its method of preparation.

7 Methods of test

It is essential that the determinations specified in this clause are carried out in a dust-free, pure atmosphere and that suitable precautions are taken to prevent any contamination of the sample and test portions.

7.1 Measurement of pH

7.1.1 *Apparatus*

Ordinary laboratory apparatus and

pH-meter, fitted with a glass measuring electrode and an Ag/AgCl reference electrode.

7.1.2 *Procedure*

Calibrate the pH-meter (**7.1.1**) in accordance with the manufacturer's instructions, using buffer solutions having pH values covering the range 4,0 to 8,0.

Transfer some of the laboratory sample to a suitable vessel and adjust the temperature of the water to 25 ± 1 °C. Introduce the electrodes and determine the pH.

7.2 Measurement of conductivity

7.2.1 *Apparatus*

Ordinary laboratory apparatus and

7.2.1.1 *Conical flask,* fitted with a guard tube containing self-indicating granular soda lime.

7.2.1.2 *Conductivity meter with cell for circulation,* having an "in-line" conductivity cell, with automatic temperature compensation, for measurements on grade 1 and grade 2 water.

NOTE If the instrument is not temperature-compensated an in-line heat-exchanger, capable of adjusting the temperature of the water under test to 25 ± 1 °C, should be fitted.

7.2.1.3 *Conductivity meter,* for measurements on grade 3 water.

7.2.2 *Procedure*

7.2.2.1 *Grade 1 and grade 2 water*

Using the conductivity meter (**7.2.1.2**), temperature-compensated to 25 ± 1 °C, measure the conductivity.

7.2.2.2 *Grade 3 water*

Transfer 400 ml of the sample into the flask (**7.2.1.1**), insert the guard tube and adjust the temperature of the water to 25 ± 1 °C. Measure the conductivity by means of the conductivity meter (**7.2.1.3**) in accordance with the manufacturer's operating instructions.

7.3 Limit test for oxidizable matter

NOTE The limits are equivalent to oxidizable matter, expressed as milligrams of oxygen (O) per litre, of 0,08 and 0,4 for grade 2 and grade 3 water respectively.

7.3.1 *Reagents*

Use grade 2 water to prepare the following reagent solutions.

7.3.1.1 *Sulfuric acid*, approximately 1 mol/l solution.

7.3.1.2 *Potassium permanganate*, standard volumetric solution, $c(1/5\ KMnO_4) = 0{,}01$ mol/l.

7.3.2 *Procedure*

7.3.2.1 *Test portion*

1 000 ml of grade 2 water or 200 ml of grade 3 water.

7.3.2.2 *Test*

Add 10 ml of the sulfuric acid solution (**7.3.1.1**) and 1,0 ml of the standard volumetric potassium permanganate solution (**7.3.1.2**) to the test portion (**7.3.2.1**), bring to the boil and boil for 5 min. Check that the colour of the test mixture is not completely discharged.

7.4 Measurement of absorbance

7.4.1 *Apparatus*

Ordinary laboratory apparatus and

7.4.1.1 *Spectrometer, with selectors for continuous variation*, or

7.4.1.2 *Spectrometer, with selectors for discontinuous variation*, fitted with filters providing maximum transmission in the neighbourhood of 254 nm.

7.4.1.3 *Optical cells*, of the same silica material, 1 cm and 2 cm path length.

NOTE If the available spectrometer is not sufficiently sensitive, the sensitivity may be increased by using optical cells of greater path length.

7.4.2 *Procedure*

Fill the 2 cm optical cell (**7.4.1.3**) with some of the sample. Measure the absorbance of this optical cell with the spectrometer (**7.4.1.1**), at a wavelength of about 254 nm, or with the spectrometer (**7.4.1.2**) fitted with suitable filters, after having adjusted the instrument to zero absorbance against the same water in the 1 cm optical cell.

7.5 Determination of residue after evaporation on heating at 110 °C

7.5.1 *Apparatus*

Ordinary laboratory apparatus and

7.5.1.1 *Rotary evaporator*, with a flask of capacity about 250 ml.

7.5.1.2 *Steam bath*

7.5.1.3 *Dish*, of platinum, silica or borosilicate glass, of capacity about 100 ml.

7.5.1.4 *Oven*, capable of being controlled at 110 ± 2 °C.

7.5.2 *Procedure*

7.5.2.1 *Test portion*

Transfer 1 000 ml of the laboratory sample into a stoppered measuring cylinder.

7.5.2.2 *Determination*

Introduce 100 ml of the test portion into the clean and dry rotary evaporator (**7.5.1.1**) and distill on the steam bath (**7.5.1.2**) under reduced pressure. As the water evaporates, add successive quantities of the test portion until the entire test portion has been evaporated to about 50 ml.

Transfer the residue quantitatively to the dish (**7.5.1.3**), previously heated for 2 h in the oven (**7.5.1.4**) controlled at 110 ± 2 °C, cooled in a desiccator and weighed to the nearest 0,000 1 g. Use two approximately 5 ml portions of the sample to effect the transfer.

Using the steam bath, evaporate the residue to dryness. Transfer the dish and residue from the steam bath to the oven, controlled at 110 ± 2 °C, and leave for about 2 h. Remove the dish from the oven, allow to cool to ambient temperature in a desiccator and weigh to the nearest 0,000 1 g. Repeat the operation of heating, cooling and weighing until the difference between two consecutive weighings does not exceed 0,000 2 g.

7.5.3 *Expression of results*

The residue after evaporation and heating at 110 °C, expressed in milligrams per kilogram, is numerically equal to the mass, in milligrams, of the residue, dried to constant mass.

7.6 Limit test for reactive silica

NOTE The limits are equivalent to silica contents, expressed as milligrams per litre of SiO_2, of 0,01 and 0,02 for grade 1 and grade 2 water respectively.

7.6.1 *Reagents*

7.6.1.1 *Silica*, standard solution I (concentrated).

Weigh, to the nearest 0,000 1 g, 1 g of finely ground pure silica sand (> 99,9 % of SiO_2), previously dried at 110 °C, into the platinum dish (**7.5.2.3**)[2)].
Add 4,5 g of sodium carbonate (anhydrous Na_2CO_3) and intimately mix the sample with a dry, smooth-ended glass rod. Ease the mixture into the centre of the dish and flatten it so that it covers an area about 30 mm in diameter. Cover the mixture with a further 0,5 g of the sodium carbonate, then gently brush any particles adhering to the glass rod into the dish.

Cover the dish with a platinum lid and place in the muffle furnace (**7.6.2.3**), controlled at 300 to 400 °C. Heat the mixture, gradually raising the temperature for about 10 min or until fusion is complete. Remove the dish from the furnace and gently swirl it to incorporate any particles. Allow to cool, wash into the dish any particles adhering to the underside of the lid with hot water, then dissolve the fused mass in hot water. Cool, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Transfer the solution to the plastics bottle for storage.

1 ml of this standard solution contains 1 mg of SiO_2.

7.6.1.2 *Silica*, standard solution II (diluted).

Transfer a 5,0 ml aliquot portion of the silica standard solution (**7.6.1.1**) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,005 mg of SiO_2.

Prepare this solution at the time of use.

7.6.1.3 *Ammonium molybdate*, 50 g/l solution.

Dissolve 5 g of powdered ammonium molybdate in a mixture of 80 ml of water and 20 ml of the sulfuric acid solution (**7.6.1.5**), without heating. Store in a plastics bottle.

7.6.1.4 *4-Methylaminophenol sulfate* (metol), indicator solution.

Dissolve 0,2 g of metol and 20 g of *di*potassium disulfite (potassium metabisulfite) in 100 ml of water, without heating. Store in a plastics bottle.

Discard the solution after 4 weeks, or if it shows prior signs of decomposition.

7.6.1.5 *Sulfuric acid*, $c(H_2SO_4)$ approximately 2,5 mol/l.

Carefully add, with stirring, 135 ml of sulfuric acid solution, $\varrho \approx 1{,}84$ g/ml, to sufficient water to produce 1 000 ml of solution. Store in a plastics bottle.

7.6.1.6 *Oxalic acid*, 50 g/l solution.

7.6.2 ***Apparatus***

Ordinary laboratory apparatus and

7.6.2.1 *Dish*, of platinum, of capacity about 250 ml.

7.6.2.2 *Matched Nessler cylinders*, of capacity 50 ml.

7.6.2.3 *Muffle furnace*, capable of being controlled at 300 to 400 °C.

7.6.2.4 *Water-bath*, capable of being controlled at approximately 60 °C.

7.6.3 ***Procedure***

7.6.3.1 *Test portion*

Take 520 ml of grade 1 water or 270 ml of grade 2 water.

7.6.3.2 *Test*

Evaporate the test portion (**7.6.3.1**) in the dish (**7.6.2.1**) in successive portions, to produce a final volume of 20 ml. Add 1 ml of the ammonium molybdate solution (**7.6.1.3**). After exactly 5 min, add 1 ml of the oxalic acid solution (**7.6.1.6**) and mix well. After 1 min, add 1 ml of the metol solution (**7.6.1.4**), and heat for 10 min in the water bath (**7.6.2.4**), maintained at approximately 60 °C. Transfer the solution to one of the matched Nessler cylinders (**7.6.2.2**).

Prepare a standard colour solution following the same procedure, but using a mixture of 19,0 ml of the sample and 1,0 ml of the standard silica solution (**7.6.1.2**) instead of the 20 ml resulting from the evaporation of the test portion (**7.6.3.1**). Transfer the solution to the other matched Nessler cylinder (**7.6.2.2**).

Viewing vertically downwards, check that the intensity of any blue colour produced in the test solution does not exceed that produced in the standard colour solution.

8 Test reports

Each test report shall include the following particulars:

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard, or regarded as optional.

[2] See national foreword for details of textual errors.

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