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Water for analytical laboratory use — Specification and test methods

Eau pour laboratoire à usage analytique — Spécification et méthodes d'essai

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Foreword

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Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Water for analytical laboratory use — Specification and test methods

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.

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

NOTES

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.

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.

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.

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).

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, $c(\mathrm{HCl}) \approx 1$ 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.

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). 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, $\rho \approx 1,84$ 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