INTERNATIONAL STANDARD

ISO 10530

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Water quality — Determination of dissolved sulfide — Photometric method using methylene blue

Qualité de l'eau — Dosage des sulfures dissous — Méthode photométrique au bleu de méthylène

Reference number
ISO 10530:1992(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10530 was prepared by Technical Committee ISO/TC 147, *Water quality*, Sub-Committee SC 2, *Physical, chemical, biochemical methods*.

Annex A forms an integral part of this International Standard.

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Water quality — Determination of dissolved sulfide — Photometric method using methylene blue

1 Scope

1.1 Application range

This International Standard specifies a photometric method for the determination of dissolved sulfide in water. The method is applicable to the determination of dissolved sulfide in a mass concentration range from 0,04 mg/l to 1,5 mg/l.

Higher concentrations may be determined by reducing and subsequently diluting the volume of the water sample used.

The method is applicable to waste water and natural waters requiring filtration.

1.2 Interferences

The following ions do not interfere with the method as long as the mass concentrations specified below are not reached or exceeded:

Cyanide	2 mg/l
Iodide	20 mg/l
Thiosulfate	900 mg/l
Thiocyanate	900 mg/l
Sulfite	700 mg/l

When applying this procedure, the determination of the sulfide portion from polysulfides will be incomplete.

Mass concentrations of carbon disulfide < 10 mg/l and/or ethyl mercaptan < 1 mg/l do not interfere with the method.

Waters which are not filterable according to clause 6 cannot be analysed by this method. In those cases sulfide which is easily liberated at pH 4 is determined. (An International Standard covering this is being prepared.)

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 5667-3:1985, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples.*

3 Principle

Filtration of the water sample in order to separate the suspended matter and sulfides of low solubility. Conservation of the sulfide in the filtrate by addition of ascorbate solution. Stripping of the sulfides from the filtrate using nitrogen and transfer into a receiving flask containing an aqueous zinc acetate solution.

Formation of leukomethylene blue in the receiving flask on the addition of acid dimethyl-*p*-phenylendiamine solution and oxidation to methylene blue by addition of iron(III) ions. Measurement of the absorbance of this complex at a wavelength of 665 nm.

4 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity which shall be freed from oxygen by appropriate measures, such as boiling out or gassing with nitrogen.

4.1 Sulfuric acid, $\rho(H_2SO_4) = 1{,}84$ g/ml.

4.2 Sodium hydroxide (NaOH), 32 % (m/m) solution, c(NaOH) ≈ 10 mol/l.

4.3 Zinc acetate solution.

Dissolve 20 g of zinc acetate-dihydrate [$Zn(CH_3COO)_2.2H_2O$] in water and make up to 1 l.

Some turbidity may occur, but this will not interfere with the determination.

4.4 Phthalate buffer solution, pH 4,0 ± 0,1.

Dissolve 80 g of potassium hydrogenphthalate ($C_8H_5KO_4$) in 920 ml of water. Check the pH of this solution and, if necessary, adjust to pH 4,0 by adding diluted sodium hydroxide solution [e.g. c(NaOH) = 1 mol/l] or hydrochloric acid solution [c(HCl) = 1 mol/l].

4.5 Ascorbate solution, pH 10 ± 0,1.

Dissolve 10 g of ascorbic acid ($C_6H_8O_6$) in 90 ml of water and adjust to pH 10 by adding sodium hydroxide solution (4.2). Stopper the bottle immediately.

Prepare this solution freshly before use.

4.6 Colour-forming reagent solution.

Suspend, in a 1 000 ml measuring flask, 2 g of N,N-dimethyl-1,4-phenyl diammonium chloride ($C_8H_{14}Cl_2N_2$) in 200 ml of water.

Cautiously add 200 ml of sulfuric acid (4.1), cool, and dilute to volume with water.

4.7 Ammonium iron(III) sulfate solution.

Place 50 g of ammonium iron(III) sulfate dodecahydrate [$NH_4Fe(SO_4)_2.12H_2O$] in a 500 ml measuring flask. Add 10 ml of sulfuric acid (4.1) and then cautiously dilute to volume with water.

4.8 Sodium sulfide stock solution.

Place an adequate quantity of sodium sulfide hydrate [$Na_2S.xH_2O$, (x = 7-9)] corresponding to approximately 0,5 g of sulfur as sulfide, with a thiosulfate content < 0,5 %, in a 1 000 ml measuring flask. Dissolve in water (clause 4) and dilute to volume.

This solution is stable for 2 d to 3 d.

Prior to use, the exact concentration is determined iodometrically (see annex A).

4.9 Sodium sulfide standard solution.

Pipette 10 ml of sodium sulfide stock solution (4.8) into a 1 000 ml measuring flask. Dilute to volume with water.

1 ml of this Standard solution contains approximately 5 µg of sulfide. The exact concentration is determined iodometrically (see annex A).

Prepare this solution freshly before use.

5 Apparatus

5.1 Filtration device, e.g. three-ring piston syringe, of capacity 50 ml, with one-way filtering attachment (pore size 0,45 µ/m) (see figure 1).

Alternatively, for waters which are difficult to filter, a pressure filtration device with membrane filter (pore size 0,45 µm) can be used. (See figure 2.)

5.2 Stripping apparatus for the separation of sulfide, for example as shown in figure 3. It consists of a reaction flask, of capacity 250 ml, with a lateral ground-glass joint attachment for the drop funnel, of capacity 100 ml, provided with a gas inlet tube ending at the bottom of the flask, vertically mounted condenser or riser tube, and an absorption vessel.

Dimensions: see figure 3.

5.3 Measuring cylinder, of capacity 25 ml.

5.4 Measuring flasks, of capacity 50 ml, 100 ml, 500 ml and 1 000 ml.

5.5 Measuring pipettes, of capacity 1 ml and 10 ml.

5.6 One-mark pipette, of capacity 1 ml, 2 ml, 5 ml, 10 ml, 20 ml, 50 ml and 100 ml.

5.7 Dispensers.

5.8 Microlitre syringes.

5.9 Gas supply with nitrogen, of high purity [99.996 % (m/m) pure].

5.10 Gas flow measuring device, suitable for a volume flow of 40 l/h.

5.11 pH-meter, equipped with an appropriate electrode.

5.12 Spectrometer or **filter photometer**, suitable for absorbance measurements at 665 nm.

5.13 Cuvettes, of path length 1 cm.

Dimensions in millimetres

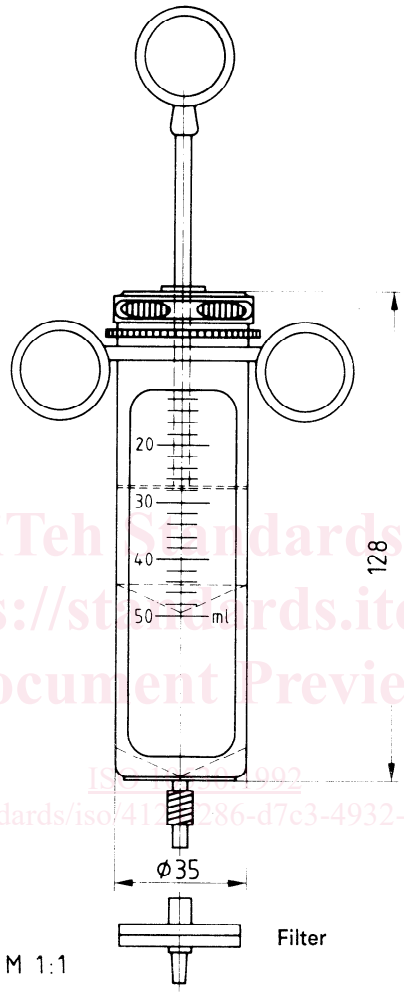

Figure 1 — Three-ring piston syringe

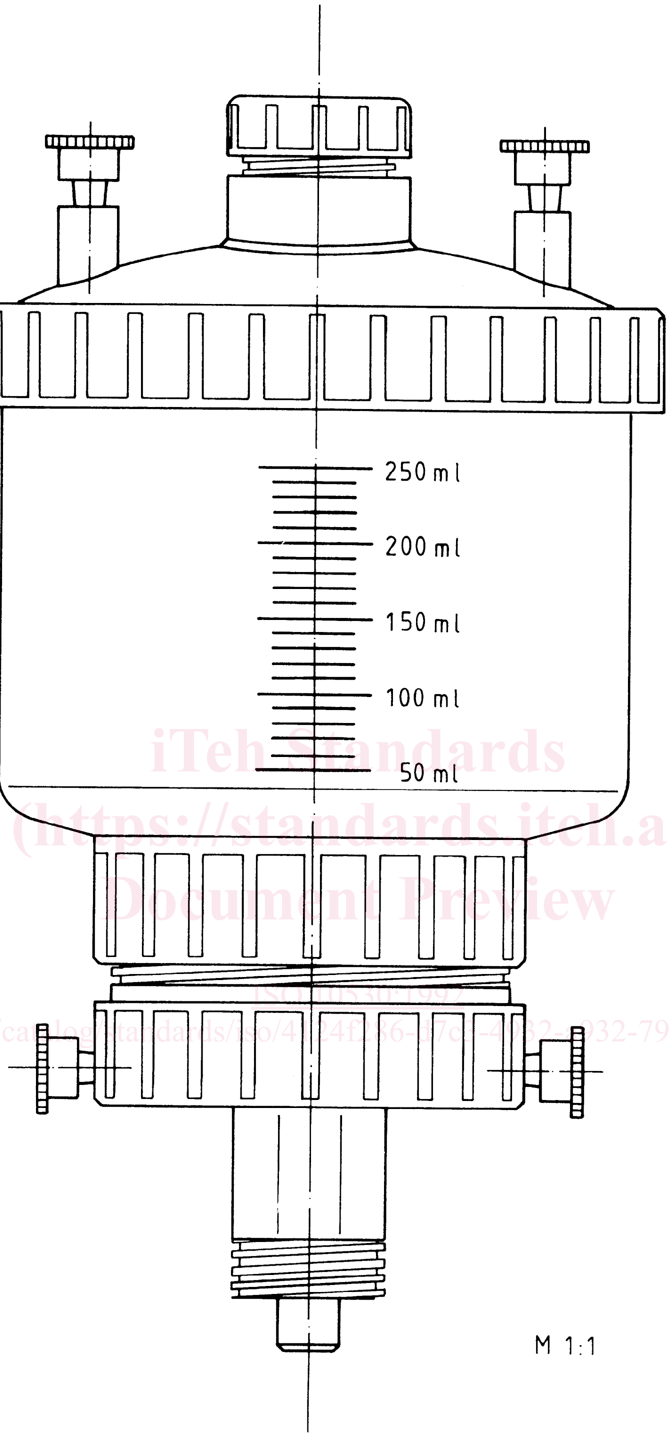

Figure 2 — Membrane filtration device