

Reagents for chemical analysis —

Part 1: Methods of test

[ISO title: Reagents for chemical analysis — Part 1: General test methods]

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Committees responsible for this British Standard

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

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National foreword

This British Standard has been prepared under the direction of the Chemicals Standards Committee. Part 1 is identical with ISO 6353-1 *“Reagents for chemical analysis — Part 1: General test methods”*, published in 1982 by the International Organization for Standardization (ISO).

Further parts of this International Standard are to be published which will comprise specifications for a series of laboratory reagents and which will make appropriate reference to the methods specified in part 1. Subject to their approval by the UK, they will be published as further Parts of this British Standard.

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.

Wherever the words “International Standard” and “this part of ISO 6353” appear, referring to this standard, they should be read as “British Standard” and “this Part of BS 6376” respectively.

Cross-references

International Standard	Corresponding British Standard
ISO 31	BS 5775 <i>Specification for quantities, units and symbols</i> (Identical)
ISO 758:1976	BS 4522:1970 <i>Method for the determination of density of liquids at 20 °C</i> (Technically equivalent)
ISO 759:1981	BS 4524:1983 <i>Method for determination of residue on evaporation on a water bath</i> (Identical)
ISO 2211:1973	BS 5339:1976 <i>Method of measurement of colour in Hazen units (platinum-cobalt scale) of liquid chemical products</i> (Identical)
ISO 6685:1982	BS 6337 <i>General methods of chemical analysis</i> Part 3:1983 <i>Method for determination of iron content (1,10-phenanthroline spectrophotometric method)</i> (Identical)

ISO 918, the revision of ISO/R 918 (to which BS 4591 is technically equivalent), and to which reference is made in **5.2.5.1**, is in course of preparation. Subject to the acceptability of the revised text, it is expected that ISO 918 will be published, without alteration, as the revision of BS 4591.

The Technical Committee has reviewed the provisions of ISO 760, to which reference is made in **5.12**, ISO 1392, to which reference is made in **5.25.3** and ISO 2718 to which reference is made in **5.34**, and has decided that they are acceptable for use in conjunction with this standard.

Related British Standards for ISO 760, ISO 1392 and ISO 2718 are BS 2511 *“Methods for the determination of water (Karl Fischer method)”*, BS 4633 *“Method for the determination of crystallizing point”* and BS 5443 *“Recommendations for a standard layout for methods of chemical analysis by gas chromatography”*, respectively.

Additional information

Water. Unless otherwise stated, water complying with the requirements of these determinations is specified in BS 3978 *“Water for laboratory use”*.

Ethanol. The ethanol used in these determinations may be replaced for these purposes by industrial methylated spirits, 95 % (V/V), complying with the requirements of BS 3591. It should be noted that the use of industrial methylated spirits is governed by The Methylated Spirits Regulations, 1952 (S.I. 1952, No 2230) as amended by The Alcoholic Liquors (Amendment of Units and Methods of Measurement) Regulation 1979 (S.I. 1979, No 1149). It is not permissible to use duty-free ethanol, received under the provisions of the Alcoholic Liquor Duties Act 1979, Section 10, for purposes for which industrial methylated spirits is an acceptable alternative to ethanol.

Storage of stock standard solutions. Glass bottles may be used for storage of stock standard solutions in place of the plastics bottles recommended in 4.1.1, unless otherwise stated in the remainder of 4.1.

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.

3.10. In line 10, “manufactures” should be read as “manufacturers”.

4.2.3. In line 7, “soon any turbidity appears” should be read as “soon as any turbidity appears”.

4.2.6.2. In line 6, “starch solutions” should be read as “starch solution”.

5.9.2. In line 1, “solution using” should read “solution to”.

This standard describes a method of test only and should not be referred to as a specification defining limits of purity. Reference to the standard should indicate that the method of test used is in accordance with BS 6376-1.

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, pages 1 to 16, an inside back cover 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.

1 Scope and field of application

This part of ISO 6353 specifies general test methods for verifying the compliance of reagents for chemical analysis with the specifications given in other parts of this International Standard.

2 References

- ISO 31, *Quantities, units and symbols*.
 ISO 758, *Liquid chemical products for industrial use — Determination of density at 20 °C*.
 ISO 759, *Volatile organic liquids for industrial use — Determination of dry residue after evaporation on a water bath — General method*.
 ISO 760, *Determination of water — Karl Fischer method (General method)*.
 ISO 918, *Volatile organic liquids for industrial use — Determination of distillation characteristics — General method*¹⁾.
 ISO 1392, *Determination of crystallizing point — General method*.
 ISO 2211, *Liquid chemical products — Measurement of colour in Hazen units (platinum-cobalt scale)*.
 ISO 2718, *Standard layout for a method of chemical analysis by gas chromatography*.
 ISO 6685, *Chemical products for industrial use — General method for determination of iron content — 1,10-Phenanthroline spectrophotometric method*²⁾.

3 General information

- 3.1** The nomenclature for chemical compounds used in this International Standard in general conforms to the rules published by the International Union of Pure and Applied Chemistry (IUPAC).
3.2 In all reactions or operations described, use only distilled or deionized water. Carbon dioxide-free water, if required, may be prepared by boiling water of the above grade for about 10 min and protecting from the atmosphere during cooling and storing.
3.3 Unless otherwise stated, solutions are aqueous and, dilutions shall be made with water.
3.4 The symbol “%” indicates percentage by mass (*m/m*), unless otherwise stated.
3.5 The reference number of a general test method, abbreviated GM, as given in the individual tests, refers to the number of the method in clause 5.

3.6 The reagents used shall conform to the specifications in ISO 6353. In the absence of such a specification, reagents of suitable analytical grade shall be used.

NOTE Reagents specified in this International Standard are identified R .., e.g. sodium chloride is R 31.

3.7 Unless otherwise stated, values for density refer to the density at 20 °C.

3.8 Temperatures are expressed in degrees Celsius (°C).

3.9 The following additional abbreviations are used in this International Standard:

AgDDTC	silver diethyldithiocarbamate
APDC	ammonium pyrrolidine-1-carbodithioate
EDTA	ethylenediaminetetraacetic acid, disodium salt
AAS	atomic absorption spectroscopy
FES	flame emission spectroscopy
GC	gas chromatography
SS	standard solution
IS	indicator solution
RS	reagent solution
GM	general test method
R	reagent
MAS	molecular absorption spectrophotometry

3.10 Warning

The physical and chemical properties of the chemicals being handled, in particular those relating to physiological effects, combustibility and explosive tendencies, may be such as to present significant health and safety hazards. Although the degree of risk is extremely variable, it should be assumed, in the absence of specific information to the contrary, that the handling of any chemical will involve hazards of this kind.

The provision of exhaustive details in respect of hazards and associated safety procedures is not considered to fall within the scope of this International Standard as most manufacturers³⁾ of chemicals are very willing to advise prospective users on the handling of their products. In addition, national regulations on the packaging and labelling of hazardous chemicals should ensure that adequate information is given on the hazards associated with the use of chemicals.

¹⁾ At present at the stage of draft. (Revision of ISO/R 918.)

²⁾ At present at the stage of draft.

³⁾ See national foreword, textual errors.

4 Solutions for use in test methods

4.1 Standard solutions

Prepare stock standard solutions (4.1.1) and dilute standard solutions (4.1.2) as follows.

4.1.1 Stock standard solutions

Dissolve the constituents indicated in column 2 of Table 1, dilute to the mark in a 1 000 ml one-mark volumetric flask and mix.

It is recommended that all stock standard solutions of inorganic compounds be stored in bottles of suitable plastic material, unless otherwise stated.

4.1.2 Dilute standard solutions

Prepare dilute standard solutions I, II and III at the time of use by diluting the stock standard solutions (4.1.1) in one-mark volumetric flasks of appropriate capacity and in the precise volume ratios 1/10, 1/100, 1/1 000, respectively.

4.2 Reagent solutions

Prepare the reagent solutions as follows.

4.2.1 Ammonium metavanadate (RS)

Dissolve 2,5 g of ammonium metavanadate in 500 ml of boiling water, cool, add 20 ml of nitric acid solution (R 19), cool and dilute to 1 000 ml. Store in a polyethylene bottle.

4.2.2 Borate standard buffer (RS)

Using the carbon dioxide-free water (see 3.2), dissolve 3,81 g of sodium tetraborate decahydrate and dilute to 1 000 ml. Store protected from atmospheric carbon dioxide.

4.2.3 Calcium hydroxide standard buffer (RS)

Prepare a saturated solution at 25 °C. Determine the calcium hydroxide concentration by titration with standard volumetric hydrochloric acid solution, $c(\text{HCl}) = 0,1 \text{ mol/l}$, using phenol red (IS 4.3.10) as indicator. The concentration $c[1/2 \text{ Ca}(\text{OH})_2]$ shall be between 0,040 0 and 0,041 2 mol/l. Store protected from atmospheric carbon dioxide and reject the solution as soon any turbidity appears⁴⁾.

4.2.4 Chromic acid (RS)

Dissolve 100 g of chromium trioxide in sulphuric acid solution (35 %) and dilute to 1 000 ml with the same acid.

4.2.5 Cobalt(II) chloride (RS)

4.2.5.1 Preparation

Dissolve 60 g of cobalt(II) chloride hexahydrate in about 900 ml of a mixture of 25 ml of hydrochloric acid solution (R 13) and 975 ml of water and dilute to 1 000 ml with the same mixture. Determine the concentration by the method specified in 4.2.5.2 and adjust it to 59,5 mg of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ per millilitre using a calculated quantity of diluted hydrochloric acid solution.

4.2.5.2 Titration

Place 5,0 ml of the solution (4.2.5.1), 5 ml of hydrogen peroxide solution (3 %) and 10 ml of sodium hydroxide solution (27 %) in a 200 ml conical flask fitted with a ground glass stopper. With the stopper removed, boil gently for 10 min, allow to cool, add 60 ml of sulphuric acid (10 %) and 2 g of potassium iodide (R 25). Stopper the flask and dissolve the precipitate by shaking gently. Titrate the liberated iodine with standard volumetric sodium thiosulphate solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}$, adding 10 drops of the starch solution (IS 4.3.11) towards the end of the titration.

The end-point is reached when the blue colour has just been discharged.

1 ml of sodium thiosulphate solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}$, corresponds to 23,79 mg of $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$.

⁴⁾ See national foreword, textual errors.