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# International Standard



# 6353/1

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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## Reagents for chemical analysis — Part 1 : General test methods

*Réactifs pour analyse chimique — Partie 1 : Méthodes générales d'essai*

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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.

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It has been approved by the member bodies of the following countries :

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The member bodies of the following countries expressed disapproval of the document on technical grounds :

Australia  
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# Reagents for chemical analysis — Part 1 : General test methods

## 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*.<sup>1)</sup>

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*.<sup>2)</sup>

## 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).

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

2) At present at the stage of draft.

**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 manufacturers 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.

## 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 appears.

#### 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  $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ .

Table 1 — Preparations of stock standard solutions

Reagent name	Mass of substance required to prepare 1 000 ml of solution	1 ml of solution corresponds to
Acetaldehyde	1,00 g of $\text{CH}_3\text{CHO}$	0,001 g of $\text{CH}_3\text{CHO}$
Aluminium	17,60 g of $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ + 10 ml $\text{H}_2\text{SO}_4$ (25 %)	0,001 g of Al
Ammonium	2,97 g of $\text{NH}_4\text{Cl}$ (R 5)	0,001 g of $\text{NH}_4$ or 0,000 776 6 g of N
Arsenic	1,32 g of $\text{As}_2\text{O}_3$ dissolved in 3 ml of NaOH solution (27 %) by warming.	0,001 g of As
Barium	1,78 g of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ (R 6)	0,001 g of Ba
Bismuth	1,00 g of Bi dissolved in 6 ml of $\text{HNO}_3$ (R 19), and nitrous gases removed by boiling.	0,001 g of Bi
Bromate	1,31 g of $\text{KBrO}_3$	0,001 g of $\text{BrO}_3$
Bromide	1,49 g of $\text{KBr}$	0,001 g of Br
Calcium	3,67 g of $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ . Alternatively, 2,50 g of $\text{CaCO}_3$ dissolved in 25 ml of $\text{HCl}$ (10 %) solution and $\text{CO}_2$ removed by boiling.	0,001 g of Ca
Carbonate	2,41 g of $\text{Na}_2\text{CO}_3$ (R 30)	0,001 g of $\text{CO}_2$ or 0,000 27 g of C
Carbonyl	10,43 g of acetone (R 2) corresponding to 5,0 g of CO, weighed into a 100 ml one-mark volumetric flask containing 50 ml of carbonyl-free methanol (RS 4.2.11), diluted to the mark with the same methanol and mixed thoroughly. Take 20,0 ml of this solution and dilute to 1 000 ml. Use the same methanol for all dilutions.	0,001 g of CO
Chlorate	1,47 g of $\text{KClO}_3$	0,001 g of $\text{ClO}_3$
Chloride	1,65 g of $\text{NaCl}$ (R 32)	0,001 g of Cl
Chlorine	3,97 g of Chloramine T (trihydrate)	0,001 g of active chlorine
Chromium	2,83 g of $\text{K}_2\text{Cr}_2\text{O}_7$ (R 23)	0,001 g of Cr
Cobalt	4,94 g of $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	0,001 g of Co
Copper	3,93g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (R 9)	0,001 g of Cu
Fluoride	2,21 g of $\text{NaF}$	0,001 g of F
Formaldehyde	2,86 g of formaldehyde solution (35 %)	0,001 g of $\text{HCHO}$
Hexacyanoferrate(II)	1,99 g of $\text{K}_4[\text{Fe}(\text{CN})_6] \cdot 3\text{H}_2\text{O}$	0,001 g of $\text{Fe}(\text{CN})_6$
Hexafluorosilicate	3,38 g of $\text{H}_2\text{SiF}_6$ (30 %) solution	0,001 g of $\text{SiF}_6$
Iron	8,63 g of $\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ + 10 ml of $\text{H}_2\text{SO}_4$ (25 %) solution	0,001 g of Fe
Iodate	1,22 g of $\text{KIO}_3$	0,001 g of $\text{IO}_3$
Iodide	1,31 g of $\text{KI}$ (R 25)	0,001 g of I
Lead	1,60 g of $\text{Pb}(\text{NO}_3)_2$ + 1 ml of $\text{HNO}_3$ (R 19)	0,001 g of Pb
Magnesium	10,14 g of $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	0,001 g of Mg
Manganese	3,08 g of $\text{MnSO}_4 \cdot \text{H}_2\text{O}$	0,001 g of Mn
Mercury	1,62 g of $\text{Hg}(\text{NO}_3)_2$ + 10 ml of $\text{HNO}_3$ (R 19)	0,001 g of Hg
Molybdenum	1,84 g of $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$	0,001 g of Mo
Nickel	4,48 g of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ or 4,78 g of $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$	0,001 g of Ni
Nitrate	1,37 g of $\text{NaNO}_3$	0,001 g of $\text{NO}_3$
Nitrite	1,50 g of $\text{NaNO}_2$	0,001 g of $\text{NO}_2$
Nitrogen	6,07 g of $\text{NaNO}_3$	0,001 g of N
Oxalate	1,43 g of $\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ (R 20)	0,001 g of $\text{C}_2\text{O}_4$
Phosphate	1,43 g of $\text{KH}_2\text{PO}_4$	0,001 g of $\text{PO}_4$
Phosphorus	4,39 g of $\text{KH}_2\text{PO}_4$	0,001 g of P
Potassium	2,59 g of $\text{KNO}_3$	0,001 g of K
Silicate	1,00 g of silicic acid heated at 900 °C and dissolved in 8 ml of NaOH solution (27 %)	0,001 g of $\text{SiO}_2$
Silver	1,58 g of $\text{AgNO}_3$ (R 28). Store the solution in a dark glass bottle.	0,001 g of Ag
Sodium	2,54 g of $\text{NaCl}$ (R 32)	0,001 g of Na
Sulphate	1,81 g of $\text{K}_2\text{SO}_4$	0,001 g of $\text{SO}_4$
Sulphide	7,49 g of $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$	0,001 g of S
Sulphur	5,44 g of $\text{K}_2\text{SO}_4$	0,001 g of S
Thiocyanate	1,31 g of $\text{NH}_4\text{SCN}$	0,001 g of SCN
Thiosulphate	2,21 g of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ (R 36)	0,001 g of $\text{S}_2\text{O}_3$
Titanium	21,47 g of $\text{TiCl}_3$ solution (15 %) + 20 ml of $\text{HCl}$ solution (25 %)	0,001 g of Ti
Zinc	4,40 g of $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$	0,001 g of Zn



**4.2.6 Copper(II) sulphate (RS)****4.2.6.1 Preparation**

Dissolve 63 g of copper(II) sulphate pentahydrate (R 9) 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 4.2.6.2 and adjust it to 62,4 mg of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  per millilitre using a calculated quantity of the diluted hydrochloric acid solution.

**4.2.6.2 Titration**

Place 10,0 ml of the solution (4.2.6.1), 50 ml of water, 12 ml of approximately 12 % acetic acid solution and 3 g of potassium iodide (R 25), in a 200 ml conical flask fitted with a ground glass stopper. Titrate the liberated iodine with standard volumetric sodium thiosulphate solution,  $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}$ , using 10 drops of the starch solutions (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 24,97 mg of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ .

**4.2.7 2,4-Dinitrophenylhydrazine (RS)**

Dissolve 50 mg of 2,4-dinitrophenylhydrazine in 25 ml of carbonyl-free methanol (RS 4.2.11) and 2 ml of hydrochloric acid solution (R 13), and dilute to 50 ml with water. Discard after 2 weeks.

**4.2.8 Iron(III) chloride (RS)****4.2.8.1 Preparation**

Dissolve 46 g of iron(III) 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 4.2.8.2 and adjust it to 45,0 mg of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  per millilitre using a calculated quantity of the diluted hydrochloric acid solution.

**4.2.8.2 Titration**

Place 10,0 ml of the solution (4.2.8.1), 15 ml of water, 5 ml of the hydrochloric acid solution (R 13) and 4 g of potassium iodide (R 25) in a 200 ml conical flask fitted with a ground glass stopper. Stopper the flask, allow to stand in the dark for 15 min and add 100 ml of water; titrate the liberated iodine with standard volumetric sodium thiosulphate solution,  $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}$ , using 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 27,03 mg of  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ .

**4.2.9 Iron(II)/iron(III) mixture (RS)**

Dissolve 10 g of ammonium iron(II) sulphate hexahydrate and 1 g of ammonium iron(III) sulphate dodecahydrate in water, add 5 ml of sulphuric acid solution (20 %), and dilute to 100 ml.

**4.2.10 Lead acetate (basic) (RS)**

Dissolve 5 g of lead(II) acetate trihydrate and 15 g of sodium hydroxide (R 34) in 80 ml of water and dilute to 100 ml.

**4.2.11 Methanol, carbonyl-free (RS)**

Add 10 g of 2,4-dinitrophenylhydrazine and 0,5 ml of hydrochloric acid solution (R 13) to 2 litres of methanol (R 18), reflux for 2 h and then distil, rejecting the first 50 ml of distillate. Stir the methanol during the distillation using a magnetic stirrer to avoid bumping. Stored in a tightly stoppered bottle, the methanol will remain carbonyl-free indefinitely.

**4.2.12 Oxalate standard buffer (RS)**

Dissolve 12,71 g of potassium tetraoxalate dihydrate in water and dilute to 1 000 ml.

**4.2.13 Phosphate standard buffer (RS)**

Dissolve 3,40 g of potassium dihydrogen phosphate and 3,55 g of sodium hydrogen phosphate, both previously dried for 2 h at  $120 \pm 10^\circ\text{C}$ , in the carbon dioxide-free water (see 3.2) and dilute to 1 000 ml with the same water.

**4.2.14 Phthalate standard buffer (RS)**

Dissolve 10,21 g of potassium hydrogen phthalate, previously dried for 1 h at  $110^\circ\text{C}$ , in water and dilute to 1 000 ml.

**4.2.15 Potassium hydroxide methanolic solution (RS)**

Mix 15,0 ml of potassium hydroxide solution (33 %) with 50 ml of carbonyl-free methanol (RS 4.2.11). Discard after 2 weeks.

**4.2.16 Sodium dihydrogen phosphate (RS)**

Dissolve 20 g of sodium dihydrogen phosphate monohydrate in water, add 1 ml of sulphuric acid solution (20 %), and dilute to 100 ml.

**4.2.17 Tartrate standard buffer (RS)**

Prepare a saturated solution of racemic potassium hydrogen tartrate by shaking an excess vigorously with water at  $25^\circ\text{C}$ . The solution has a limited shelf life which may be extended by adding a small crystal of thymol.

**4.3 Indicator solutions**

Prepare the indicator solutions as follows.



**4.3.1 Ammonium iron(III) sulphate (IS)**

Dissolve 33 g of ammonium iron(III) sulphate dodecahydrate in 67 ml of nitric acid solution (12 %).

**4.3.2 Bromophenol blue (IS)**

Warm 0,1 g of bromophenol blue with 3,0 ml of 0,2 % sodium hydroxide solution and 5 ml of 95 % (V/V) ethanol. When dissolution is complete, add 50 ml of 95 % (V/V) ethanol and dilute to 250 ml with water.

**4.3.3 Crystal violet (IS)**

Dissolve 1,25 g of crystal violet C.I. 42.555 in 250 ml of acetic acid (R 1).

**4.3.4 Isatin (IS)**

Dissolve 0,50 g of isatin in 50 ml of sulphuric acid solution (R 37) (solution A).

Dissolve 0,50 g of iron(III) chloride hexahydrate in 2 ml of water and dilute to 100 ml with sulphuric acid solution (R 37) stirring until the evolution of gas ceases (solution B).

Immediately before use, add 5,0 ml of solution B to 2,5 ml of solution A and dilute to 100 ml with sulphuric acid solution (R 37).

**4.3.5 Methyl orange (IS)**

Dissolve 0,1 g of methyl orange C.I. 13025 in 50 ml of 95 % (V/V) ethanol and dilute to 250 ml with water.

**4.3.6 Methyl red (IS)**

Warm 25 mg of finely powdered methyl red C.I. 13020 with 0,95 ml of 0,2 % sodium hydroxide solution and 5 ml of 95 % (V/V) ethanol. When dissolution is complete, add 125 ml of 95 % (V/V) ethanol and dilute to 250 ml with water.

**4.3.7 Methylthymol blue mixture**

Triturate 1 g of methylthymol blue with 100 g of potassium nitrate to a fine powder.

**4.3.8 Mordant black 11 mixture**

Triturate 1 g of Mordant black 11 C.I. 14645 and 100 g of sodium chloride to a fine powder.

**4.3.9 Phenolphthalein (IS)**

Dissolve 2,5 g of phenolphthalein in 250 ml of 95 % (V/V) ethanol.

**4.3.10 Phenol red (IS)**

Warm 50 mg of phenol red with 2,85 ml of 0,2 % sodium hydroxide solution and 5 ml of 95 % (V/V) ethanol. After solu-

tion is effected, add 50 ml of 95 % (V/V) ethanol and dilute to 250 ml with water.

**4.3.11 Starch (IS)**

Make a paste of 1,0 g of soluble starch with 5 ml of water and add the paste to 100 ml of boiling water, with stirring. Boil for a few minutes and cool. Discard the solution after 2 weeks.

NOTE — The shelf life of the solution may be extended to several months by adding a few drops of formaldehyde solution.

**4.3.12 Thymolphthalein (IS)**

Dissolve 0,50 g of thymolphthalein in 60 ml of 95 % (V/V) ethanol and dilute to 250 ml with water.

**5 General test methods (GM)****5.1 Water-insoluble matter (GM 1)**

Dissolve the specified test portion as completely as possible in a suitable volume of boiling water, cool and filter through a sintered glass filter porosity P 40 (pore size diameter 16 to 40 µm), previously dried for 1 h at  $105 \pm 2$  °C, cooled in a desiccator and weighed to the nearest 0,1 mg. Wash the residue with water, dry for 1 h at  $105 \pm 2$  °C, cool in a desiccator and then reweigh to the nearest 0,1 mg. Calculate the mass of residue.

**5.2 Chloride (GM 2)**

Acidify the specified volume of the test solution with 1 ml of nitric acid solution (25 %) and add 1 ml of approximately 1,7 % silver nitrate solution.

Allow to stand for 2 min. Compare the opalescence with that obtained with the specified standard matching solution.

**5.3 Sulphate (GM 3)**

Mix 0,25 ml of 0,02 % potassium sulphate solution in 30 % (V/V) ethanol with 1 ml of 25 % barium chloride dihydrate solution (seeding solution). To this mixture add, after exactly 1 min, the specified volume of the test solution which has previously been acidified with 0,5 ml of 20 % hydrochloric acid solution.

Allow to stand for 5 min and compare the turbidity with that obtained with the specified standard matching solution.

**5.4 Phosphate (GM 4)**

Add 5 ml of 10 % ammonium molybdate solution to the specified volume of the test solution. Adjust the pH to 1,8 and heat the solution to boiling. Cool, add 12,5 ml of 15 % hydrochloric acid solution and extract with 20 ml of diethyl ether. Wash the organic phase with 5 % hydrochloric acid solution and reduce with 0,2 ml of 2 % tin(II) chloride dihydrate solution in hydrochloric acid solution (R 13).

Compare the blue colour of the organic phase with that obtained with the specified standard matching solution.

## 5.5 Silicate (GM 5)

Mix the specified volume of the test solution with 4,5 ml of 5 % sulphuric acid solution and 1 ml of 10 % ammonium molybdate solution. After 5 min, add 5 ml of 5 % oxalic acid solution and after a further 5 min, add 0,2 ml of 2 % tin(II) chloride dihydrate solution in hydrochloric acid solution (R 13).

Compare the blue colour with that obtained with the specified standard matching solution.

## 5.6 Total nitrogen (GM 6)

To the specified volume of the test solution, diluted if necessary to 140 ml, in a Kjeldahl apparatus consisting of a Kjeldahl flask fitted with a distillation unit, add 5 ml of 32 % sodium hydroxide solution and 1,0 g of Devarda's alloy or aluminium wire. Allow to stand for 1 h. Distil 75 ml of the reaction mixture into a graduated cylinder containing 5,0 ml of 0,5 % sulphuric acid solution. Add 3 ml of 32 % sodium hydroxide solution and 2 ml of Nessler's reagent and dilute to 100 ml.

Compare the yellow colour with that obtained with the specified standard matching solution.

## 5.7 Heavy metals (as Pb) (GM 7)

Add 0,2 ml of 30 % acetic acid solution to the specified volume of the test solution and saturate with hydrogen sulphide or use a suitable quantity of aqueous solution of hydrogen sulphide.

Compare the brown colour with that obtained with the specified standard matching solution.

## 5.8 Iron (GM 8)

### 5.8.1 1,10-Phenanthroline method (GM 8.1)

See ISO 6685.

### 5.8.2 Bathophenanthroline method (GM 8.2)

#### 5.8.2.1 Buffer solution (pH 4,5)

Dissolve 164 g of sodium acetate trihydrate (R 29) and 115 ml of acetic acid (R 1) in water. Dilute to 1 000 ml.

#### 5.8.2.2 Procedure

Add 1 ml of 10 % ascorbic acid solution, 15 ml of buffer solution (5.8.2.1) and 1 ml of 0,1 % solution of bathophenanthroline disulphonic acid *d*/sodium salt to the specified volume of the test solution. Dilute to 50 ml and allow to stand for 15 min. Compare the red colour with that obtained with the specified standard matching solution.

## 5.9 Aluminium (GM 9)

### 5.9.1 Aluminon reagent

Add 0,25 g of aluminon (ammonium aurine tricarboxylate) and 5 g of gum acacia in 250 ml of water and warm to dissolve. Add 87 g of ammonium acetate (R 4) and dissolve. Add 145 ml of 15 % hydrochloric acid solution and dilute to 500 ml. Filter if necessary. Discard the solution after 1 month.

### 5.9.2 Procedure

Neutralize the specified volume of the test solution using litmus, add 1 ml of 30 % acetic acid solution and adjust the pH of the solution to 4,5 with 10 % ammonia solution. Add 0,1 ml of thioacetic acid and 3 ml of the aluminon reagent (5.9.1), heat at about 100 °C for 10 min and cool.

Compare the red colour with that obtained with the specified standard matching solution.

## 5.10 Ammonia (GM 10)

Dilute the specified volume of the test solution to 75 ml. Add 3 ml of 32 % sodium hydroxide solution and 2 ml of Nessler's reagent and dilute to 100 ml.

Compare the yellow colour with that obtained with the specified standard matching solution.

## 5.11 Arsenic (GM 11)

To the sample solution, add 10 ml of 0,4 % tin(II) chloride dihydrate solution in hydrochloric acid solution (R 13), 5 ml of 15 % potassium iodide solution and 1 ml of 2 % copper(II) sulphate pentahydrate solution. Allow this mixture to react with 8 g of zinc granules (R 40). Trap the arsine formed in an absorption vessel containing 5 ml of 0,5 % AgDDTC solution in pyridine (see the figure).

Measure the red colour with that obtained with the specified standard matching solution.

## 5.12 Water — Karl Fischer method (GM 12)

See ISO 760.

## 5.13 Acidity and alkalinity (GM 13)

### 5.13.1 Acidity or alkalinity in water-miscible liquid products (GM 13.1)

#### 5.13.1.1 Procedure

Place 100 ml of water in a 250 ml conical flask and boil for 5 min in order to eliminate carbon dioxide. After cooling slightly, add the specified volume of the test solution and boil gently for a further 5 min. Then close the flask with a stopper fitted with a soda-lime guard tube and allow the solution to cool to room temperature. Finally, add the specified indicator and titrate with the specified titrant solution, to reach the appropriate end-point stable for at least 15 s.

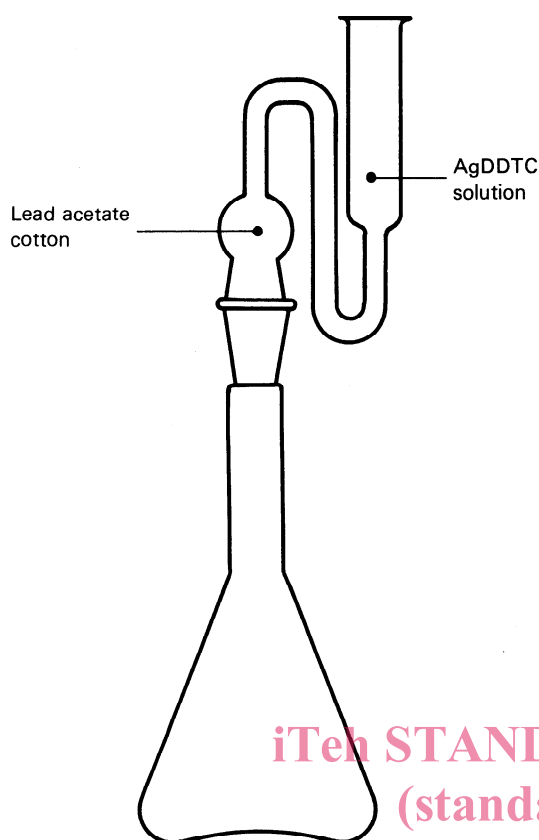


Figure — Absorption apparatus for use in arsenic test

#### 5.13.1.2 Expression of results

The acidity or alkalinity, in millimoles of  $H^+$  or  $OH^-$ , as appropriate, per 100 g of product, is given by the formula

$$\frac{V \times c}{m} \times 100$$

where

$V$  is the volume, in millilitres, of the standard volumetric solution used;

$c$  is the concentration, in moles of  $H^+$  or  $OH^-$  per litre, of the standard volumetric solution;

$m$  is the mass, in grams, of sample present in the specified volume of the test solution.

#### 5.13.2 Acidity or alkalinity in water-immiscible liquid products (GM 13.2)

##### 5.13.2.1 Procedure

Add 100 ml of water, previously neutralized against the specified indicator, to the specified volume of the test solution in a separating funnel, and shake for 3 min. Allow the two phases to separate, titrate 50 ml of the aqueous phase with the specified titrant solution, to reach the appropriate end-point stable for at least 15 s.

#### 5.13.2.2 Expression of results

The acidity or alkalinity, in millimoles of  $H^+$  or  $OH^-$ , as appropriate, per 100 g of product, is given by the formula

$$\frac{V \times c \times 100}{m \times 50} \times 100$$

$$= \frac{V \times c}{m} \times 200$$

where

$V$  is the volume, in millilitres, of the standard volumetric solution used;

$c$  is the concentration, in moles of  $H^+$  or  $OH^-$  per litre, of the standard volumetric solution;

$m$  is the mass, in grams, of sample present in the specified volume of test solution.

#### 5.14 Residue on evaporation (GM 14) (see also ISO 759)

Evaporate the specified test portion to dryness on a boiling water bath, using a suitable weighed evaporation dish of about 150 ml capacity (platinum, glass, silica). Dry to constant mass at  $105 \pm 2^\circ C$ , as specified in the specification for each product concerned.

#### 5.15 Residue on heating (GM 15)

Place the specified test portion in a suitable crucible or dish, previously heated in an oven, controlled at  $650 \pm 50^\circ C$ , for 15 min, cooled in a desiccator and weighed to the nearest 0,1 mg. Heat by increasing the temperature slowly, until the test portion is completely volatilized or charred. In the case of liquid products, heat without boiling until the test portion is completely evaporated, avoiding the ignition of organic products. Finally, heat the dish and residue in the oven, controlled at  $650 \pm 50^\circ C$ , for 15 min (unless otherwise specified), cool in a desiccator, and weigh to the nearest 0,1 mg.

NOTE — If may be more convenient to volatilize or char larger test portions in successive fractions.

#### 5.16 Sulphated ash in solid products (GM 16)

Place the specified test portion in a suitable crucible or dish, previously heated in an oven, controlled at  $650 \pm 50^\circ C$ , for 15 min, cooled in a desiccator and weighed to the nearest 0,1 mg. Heat by increasing the temperature slowly, until the test portion is completely volatilized or charred, avoiding the ignition of organic products. Allow to cool, add 0,25 ml of sulphuric acid solution (R 37) to the residue and continue heating gently until all fumes have disappeared. Then heat the dish and the residue in the oven, controlled at  $650 \pm 50^\circ C$ , for 15 min (unless otherwise specified), cool in a desiccator and weigh to the nearest 0,1 mg.

NOTE — It may be more convenient to volatilize or char larger test portions in successive fractions.