International Standard ISO 6353/2

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

Reagents for chemical analysis — Part 2 : Specifications — First series

Réactifs pour analyse chimique — Partie 2 : Spécifications — Première série

First edition — 1983-12-15

UDC 543.4 **Ref. No. ISO 6353/2-1983 (E)**

Descriptors : chemical analysis, chemical reagents, specifications, tests.

Price based on 57 pages

ISO 6353/2-1983 (E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been authorized 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.

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.

International Standard ISO 6353/2 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in August 1981.

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

Austria
Belgium
Brazil
China
Czechoslovakia
Egypt, Arab Rep. of
France
Germany, F. R.
Hungary
Ireland
Italy
Japan
Korea, Dem. P. Rep. of
Korea, Rep. of
Netherlands
Philippines
Poland
Portugal
Romania
South Africa, Rep. of
Switzerland
United Kingdom

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

India
USSR

Printed in Switzerland

INTERNATIONAL STANDARD ISO 6353/2-1983/ADDENDUM 2

Publié 1986-11-15

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

Reagents for chemical analysis — Part 2: Specifications — First series

Réactifs pour analyses chimiques — Partie 2: Spécifications — Première série

ADDENDUM 2

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.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

Addendum 2 to International Standard ISO 6353/2-1983 was prepared by Technical Committee ISO/TC 47, *Chemistry*.

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.

UDC 543.4 **Ref. No. ISO 6353/2-1983/Add.2-1986 (E)**

Descriptors : chemical analysis, chemical reagents, specifications, tests.

Printed in Switzerland

Price based on 1 page

PS 30 Sodium carbonate, primary standard Na_2CO_3

Relative molecular mass: 105,988

PS 30.1 Specification

Assay (Na_2CO_3, after drying at 285 °C) ... 99,95 to 100,05 %

The mean value of the assay shall be in the range 99,95 to 100,05 % and the standard deviation shall not exceed ±0,05 %.

All other properties shall comply with the requirements for reagent R 30.

PS 30.2 Test

PS 30.2.1 Standardization of hydrochloric acid solution, $c(HCl) = 1$ mol/l

Weigh, to the nearest 0,000 1 g, about 1,9 g of electrolytic silver (99,995 %), and dissolve it in 10 ml of nitric acid (R 19) by gently heating. Cool and dilute to about 80 ml with water. Titrate with hydrochloric acid solution, $c(HCl) \approx 1$ mol/l, according to GM 31.2. Use a potentiometer accurate to ± 1 mV and a 25 ml burette graduated in 0,05 ml divisions and complying with class A of ISO 385/1. Operate at the calibration temperature of the burette (for example 20 ± 1 °C).

The concentration c, in moles of HCl per litre, of the hydrochloric acid solution is given by the equation

$$c = \frac{m_1}{0{,}107\,87\ V_1}$$

where

m_1 is the mass, in grams, of electrolytic silver weighed;

V_1 is the volume, in millilitres, of hydrochloric acid solution used for the titration;

Carry out ten titrations and calculate the mean value.

PS 30.2.2 Determination of sodium carbonate

Dry the sodium carbonate at 285 °C for at least 2 h, then place it in a desiccator for 30 min.

Weigh, to the nearest 0,000 1 g, about 0,93 g of the sodium carbonate, and dissolve it in 150 ml of water. Add, using a 25 ml burette graduated in 0,05 ml divisions and complying with class A of ISO 385/1, 16,00 ml of the hydrochloric acid solution, cautiously and while stirring, and then heat to boiling. Cool and titrate, using the same burette, with the same hydrochloric acid solution according to GM 31.2. Use the same potentiometer, accurate as in PS 30.2.1. Operate at the calibration temperature of the burette (for example 20 ± 1 °C).

The assay, expressed as a percentage by mass of Na_2CO_3, is given by the formula

$$\frac{5{,}299\,4\ V_2\ c}{m_2}$$

where

V_2 is the total volume, in millilitres, of hydrochloric acid solution used for the determination;

c is the concentration, in moles of HCl per litre, of hydrochloric acid solution used;

m_2 is the mass, in grams, of sodium carbonate weighed.

Carry out at least ten titrations and calculate the mean value $\bar{x}$ and the standard deviation s, using the following equations:

$$\bar{x} = \frac{\sum_{i=1}^{N} x_i}{N}$$

and

$$s = \sqrt{\frac{\sum_{i=1}^{N} (x_i - \bar{x})^2}{N-1}}$$

where

x_i is an individual value;

N is the number of values measured.

Contents

Reagents for chemical analysis — Part 2 : Specifications — First series

1 Scope and field of application

This part of ISO 6353 gives specifications and indicates the test methods to be used for checking conformity with these specifications for a first series of reagents used in analytical chemistry.

This document should be read in conjunction with ISO 6353/1 which describes the general test methods (GM) applicable to the requirements of the reagent specifications and gives such general information as is required for the correct use of the standard.

Particular attention is drawn to ISO 6353/1, clause 4, which describes the preparation of

— standard solutions (SS) at dilutions I, II and III;

— reagents solutions (RS);

— indicator solutions (IS).

In this part of ISO 6353, asterisked clause reference numbers refer to ISO 6353/1.

2 Reagents (abbreviation : R), Specifications — First series

General remark — In all tests involving comparison with a standard matching solution, the result (for example colour intensity) obtained on the test solution shall not be greater than that obtained on the specified standard matching solution.

R 1 Acetic acid CH_3COOH

Relative molecular mass : 60,05

R 1.1 Specification

Assay (CH_3COOH)	99,7 % min.
Density : 1,05 g/ml	
Chloride (Cl)	0,000 1 % max.
Sulfate (SO_4)	0,000 2 % max.
Copper (Cu)	0,000 05 % max.
Iron (Fe)	0,000 1 % max.
Lead (Pb)	0,000 05 % max.
Dichromate-reducing substances (expressed as O)	0,008 % max.
Residue after evaporation	0,003 % max.

R 1.2 Preparation of test solution

To 100 g (95 ml) of the sample, add 1 ml of sodium carbonate solution (1 %) and evaporate to dryness on a boiling water bath. Dissolve the residue in a little water, add 1 ml of the hydrochloric acid (R 13), and dilute to 50 ml with water.

R 1.3 Tests

R 1.3.1 Assay

Weigh, to the nearest 0,000 1 g, 2 to 3 g of the sample, dilute with 50 ml of water and titrate with standard volumetric sodium hydroxide solution, c(NaOH) = 1 mol/l, using the phenolphthalein (IS 4.3.9*).

1,00 ml of sodium hydroxide solution, c(NaOH) = 1,000 mol/l, corresponds to 0,060 05 g of CH_3COOH.

R 1.3.2 Chloride

Dilute 10 g (9,5 ml) of the sample with water to 30 ml, and apply GM 2.

Prepare a standard matching solution, using 10 ml of the chloride SS III (10 ml ≙ 0,000 1 % Cl).

R 1.3.3 Sulfate

Take 12,5 ml of the test solution (R 1.2) and apply GM 3.

Prepare a standard matching solution, using 5 ml of the sulfate SS II (5 ml ≙ 0,000 2 % SO_4).

R 1.3.4 Copper and lead

Determine these elements by AAS according to GM 29, using the following conditions :

Element	Concentration of solution	Flame	Resonance line nm
Cu	Test solution (R 1.2)	Air-acetylene	324,7
Pb	Test solution (R 1.2)		217,0 or 283,3

R 1.3.5 Iron

Take 5 ml of the test solution (R 1.2) and apply GM 8.1.

Prepare a standard matching solution, using 1 ml of the iron SS II (1 ml ≙ 0,000 1 % Fe).

R 1.3.6 Dichromate-reducing substances

To 10,00 ml of 4,90 g/l potassium dichromate solution in a conical flask fitted with a ground glass stopper, add cautiously, while cooling and mixing, 10 ml of the sulfuric acid (R 37) and cool to ambient temperature.

Add 10 g (9,5 ml) of the sample and allow to stand for 1 h at 50 ± 2 °C. Dilute to 50 ml with water, allow to cool to ambient temperature, add 5 ml of potassium iodide solution (10 %) and titrate with standard volumetric sodium thiosulfate solution, $c(Na_2S_2O_3)$ = 0,05 mol/l, using the starch (IS 4.3.11*).

Carry out in parallel a blank test.

The dichromate-reducing substances, expressed as a percentage by mass as oxygen, are given by the formula

$$0{,}000\,4\,(V_2 - V_1) \times 10$$

where

V_1 is the volume, in millilitres, of standard volumetric sodium thiosulfate solution used for the determination;

V_2 is the volume, in millilitres, of standard volumetric sodium thiosulfate solution used for the blank test;

0,000 4 is the mass, in grams, of oxygen corresponding to 1,00 ml of sodium thiosulfate solution, $c(Na_2S_2O_3)$ = 0,050 mol/l.

R 1.3.7 Residue after evaporation

Take 50 g (48 ml) of the sample and apply GM 14.

The mass of the residue shall not exceed 1,5 mg.

R 2 Acetone
CH_3COCH_3

Relative molecular mass : 58,08

R 2.1 Specification

Assay (CH_3COCH_3)	99,5 % min.
Methanol (CH_3OH)	0,05 % max.
Density : 0,787 to 0,793 g/ml	
Residue after evaporation	0,001 % max.
Acidity (expressed in millimoles of H^+)	0,05/100 g max.
Alkalinity (expressed in millimoles of OH^-)	0,05/100 g max.
Permanganate-reducing substances (expressed as O)	0,000 3 % max.
Aldehydes (expressed as HCHO)	0,002 % max.
Water	0,3 % max.

R 2.2 Tests

R 2.2.1 Assay and methanol

Apply GM 34, using the following conditions :

Stationary phase	10 % Carbowax 400
Support	Chromosorb G-AW-DMCS [0,125 to 0,150 mm (100 to 120 mesh ASTM)]
Column length	3 m
Column internal diameter	2,5 mm
Column material	Stainless steel or, preferably, glass
Column temperature	60 °C
Injection temperature	150 °C
Detection temperature	150 °C
Type of detector	Flame ionization
Carrier gas	Nitrogen
Flow rate	25 ml/min
Test portion	0,5 µl

R 2.2.2 Density

Apply GM 24.1.

R 2.2.3 Residue after evaporation

Take 100 g (127 ml) of the sample and apply GM 14.

The mass of the residue shall not exceed 1 mg.

R 2.2.4 Acidity

Take 79 g (100 ml) of the sample and apply GM 13.1, titrating with standard volumetric sodium hydroxide solution, c(NaOH) = 0,01 mol/l, and using the phenolphthalein (IS 4.3.9*).

The volume of titrant shall not exceed 4 ml.

R 2.2.5 Alkalinity

Take 79 g (100 ml) of the sample and apply GM 13.1, titrating with standard volumetric sulfuric acid solution, $c(1/2\ H_2SO_4)$ = 0,01 mol/l, and using the methyl red (IS 4.3.6*).

The volume of titrant shall not exceed 4 ml.

R 2.2.6 Permanganate-reducing substances

Take 40 g (50 ml) of the sample and apply GM 19.1, adding 0,15 ml of 3,16 g/l potassium permanganate solution. Allow the test solution to stand at 20,0 ± 0,5 °C for 15 min.

The pink colour shall not be completely discharged.

R 2.2.7 Aldehydes

Take 2 g (2,5 ml) of the sample and apply GM 20.

Prepare a standard matching solution, using 4 ml of the formaldehyde SS II (4 ml ≙ 0,002 % HCHO).

R 2.2.8 Water

Take 7,9 g (10 ml) of the sample, dilute to 30 ml with pyridine and apply GM 12.