SLOVENSKI STANDARD
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Reagenti za kemijsko analizo - 3. del: Specifikacije - Druga serija

Reagents for chemical analysis -- Part 3: Specifications -- Second series

Réactifs pour analyse chimique -- Partie 3: Spécifications -- Deuxième série

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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Reagents for chemical analysis —

Part 3:
Specifications — Second series

Réactifs pour analyses chimiques —

Partie 3: Spécifications — Deuxième série

Reference number
ISO 6353-3 : 1987 (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.

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.

International Standard ISO 6353-3 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.

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Reagents for chemical analysis —

Part 3:
Specifications — Second 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 second series of reagents used in analytical chemistry the numbering system of which continues the first series specified in ISO 6353-2.

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;
- reagent solutions (RS);
- indicator solutions (IS).

In this part of ISO 6353, asterisked clause reference numbers refer to the 1982 edition of ISO 6353-1.

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

General remarks

1 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.

2 For all iron determinations according to ISO 6685, no photometric measurement will be executed; the determinations will be carried out using a matching solution as indicated in the relevant monograph.

3 Trivial names of indicators are used in the clauses and the IUPAC names are given in footnotes.

R 41 Acetic anhydride $(CH_3CO)_2O$

Relative molecular mass: 102,09

R 41.1 Specification

Assay [$(CH_3CO)_2O$]	97,0 % min.
Residue after evaporation	0,003 % max.
Chloride (Cl)	0,000 5 % max.
Sulfate (SO_4)	0,000 5 % max.
Copper (Cu)	0,000 1 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,000 1 % max.
Permanganate-reducing substances (expressed as O)	0,02 % max.

R 41.2 Preparation of test solution

Dilute 37 ml (40 g) of the sample to 200 ml with water (1 ml ≙ 0,2 g).

R 41.3 Tests

R 41.3.1 Assay

Pipette 50 ml of morpholine methanolic solution (RS)[1] into each of two 250 ml glass-stoppered flasks. For the test portion, weigh accurately 1,8 to 2,0 g of the acetic anhydride from a Lunge pipette into the first flask and swirl to effect dissolution. Reserve the second flask for the determination of the quantity of morpholine mixed with the sample. Allow the flasks to stand at room temperature for 5 min. Add 0,20 to 0,25 ml of methyl yellow - methylene blue mixed indicator (IS)[2][3][4]. Titrate each solution with a standard volumetric hydrochloric acid methanolic solution, c(HCl) = 0,5 mol/l,[1] to the end-point at which the green colour changes to amber.

1,00 ml of hydrochloric acid methanolic solution, c(HCl) = 0,500 mol/l, corresponds to 0,051 05 g of $(CH_3CO)_2O$.

R 41.3.2 Residue after evaporation

Take 50 g (46 ml) of the sample and apply GM 14 drying the residue for 30 min.

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

R 41.3.3 Chloride

Dilute 10 ml of the test solution (R 41.2) to 20 ml with water and apply GM 2.

Prepare a standard matching solution, using 1 ml of the chloride SS I (1 ml ≙ 0,000 5 % Cl).

R 41.3.4 Sulfate

To 50 ml of the test solution (R 41.2) add 1 ml of sodium carbonate solution (1 %) and evaporate to dryness on a boiling water bath. Dissolve the residue in 10 ml of water and apply GM 3.

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

1) **Reagent solutions (RS)**

a) **Hydrochloric acid,** methanolic standard volumetric solution, c(HCl) = 0,5 mol/l.

Transfer 84 ml of hydrochloric acid, c(HCl) = 6 mol/l, to a 1 000 ml one-mark volumetric flask and dilute to the mark with the methanol (R 18). Standardize daily against a standard volumetric sodium hydroxide solution, c(NaOH) = 0,5 mol/l, using 0,2 ml of the phenolphthalein (IS 4.3.9*) IUPAC name: [3,3-bis(4-hydroxyphenyl)phthalide]. The reagent is best handled in an automatic burette assembly.

b) **Morpholine,** 0,5 mol/l methanolic solution.

Dilute 44 ml of redistilled morpholene to 1 litre with the methanol (R 18). To facilitate removal of aliquots fit the bottle with a two-hole rubber stopper and insert a 50 ml pipette through one hole so that the tip dips below the surface of the liquid. Through the other hole insert a short piece of glass tubing to which is attached a rubber atomizer bulb.

2) **Indicator solution (IS)**

Methyl yellow - methylene blue, mixed solution.

Dissolve 1,0 g of methyl yellow CI 11020 and 0,1 g of methylene blue CI 52015 in 125 ml of the methanol (R 18).

3) IUPAC name: *N,N*-dimethyl-4-(phenylazo)aniline.

4) IUPAC name: 3,7-bis(dimethylamino-5λ^4-phenothiazin-5-ylium chloride.

R 41 Acetic anhydride

R 41.3.5 Copper, iron and lead

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

Element	Concentration of solution	Flame	Resonance line nm
Cu	Evaporate 37 ml (40 g) of the sample, dissolve the residue with 3 ml of warm hydrochloric acid (R 13) and dilute to 20 ml with water.	Air-ethine	324,7
Fe			248,3
Pb			217,0

R 41.3.6 Permanganate-reducing substances

To 10 ml of the test solution (R 41.2) add 0,5 ml of potassium permanganate solution (0,32 %) and allow to stand for 5 min.

The pink colour shall not disappear completely.

R 42 Ammonium iron(II) sulfate hexahydrate $(NH_4)_2Fe(SO_4)_2.6H_2O$

Relative molecular mass: 392,14

R 42.1 Specification

Assay [$(NH_4)_2Fe(SO_4)_2.6H_2O$]	99 % min.
pH (5 % solution	3 to 5
Chloride (Cl)	0,001 % max.
Phosphate (PO_4)	0,002 % max.
Calcium (Ca)	0,01 % max.
Copper (Cu)	0,002 % max.
Iron(III) (Fe)	0,02 % max.
Lead (Pb)	0,002 % max.
Magnesium (Mg)	0,01 % max.
Manganese (Mn)	0,05 % max.
Potassium (K)	0,01 % max.
Sodium (Na)	0,01 % max.
Zinc (Zn)	0,003 % max.

R 42.2 Preparation of test solution

Dissolve 10 g of the sample in water with the addition of 2 ml of sulfuric acid solution (96 %) and dilute to 200 ml with water (the solution shall be clear).

R 42.3 Tests

R 42.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 1,5 g of the sample, dissolve in water and add 20 ml of sulfuric acid solution (16 %) and 2 ml of the phosphoric acid (R 22).

Titrate with standard volumetric potassium permanganate solution, $c(1/5\ KMnO_4) = 0{,}1$ mol/l, to a faint pink colour.

1,00 ml of potassium permanganate solution, $c(1/5\ KMnO_4) = 0{,}100$ mol/l, corresponds to 0,039 214 g of $(NH_4)_2Fe(SO_4)_2.6H_2O$.

R 42.3.2 pH

Determine the pH of a 5 % solution of the sample according to GM 31.1, using a calibrated pH meter.

R 42.3.3 Chloride

Take 30 ml of the test solution (R 42.2), add 5 ml of the nitric acid (R 19), heat to boiling and after cooling apply GM 2.

Prepare a standard matching solution, using 10 ml of the test solution and 1 ml of the chloride SS II (1 ml ≙ 0,001 % Cl).

R 42.3.4 Phosphate

Dissolve 1 g of the sample in 20 ml of water, oxidize with 4 ml of the nitric acid (R 19) and remove the nitrogen oxides by boiling. Cool, dilute to 80 ml with water and apply GM 4.

Prepare a standard matching solution, using 2 ml of the phosphate SS II (2 ml ≙ 0,002 % PO_4).

R 42.3.5 Calcium, copper, lead, magnesium, manganese and zinc

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	Test solution (R 42.2)	Air-ethine	422,7
Cu			324,7
Pb			217,0 or 283,3
Mg	10 ml of the test solution (R 42.2) diluted to 100 ml		285,2
Mn			279,5
Zn	Test solution (R 42.2)		213,9

R 42.3.6 Iron(III)

Dissolve 0,5 g of the sample in 10 ml of carbon dioxide-free water containing 0,5 ml of hydrochloric acid solution (25 %). Add 2 ml of 5-sulfosalicylic acid solution (10 %) and close the test tube.

After 15 min, the red coloration of the resulting solution shall be not more intense than that of a similarly prepared standard matching solution, using 5 ml of the iron SS II (5 ml ≙ 0,01 % Fe).

R 42.3.7 Potassium and sodium

Determine these elements by FES according to GM 30, using the following conditions:

Element	Concentration of solution	Flame	Wavelength nm
K	Test solution (R 42.2)	Air-ethine	766,5
Na			589,0

R 43 Ammonium iron(III) sulfate dodecahydrate $NH_4Fe(SO_4)_2.12H_2O$

Relative molecular mass: 482,18

R 43.1 Specification

Assay [$NH_4Fe(SO_4)_2.12H_2O$]	99,0 % min.
Water-insoluble matter	0,005 % max.
Chloride (Cl)	0,000 5 % max.
Copper (Cu)	0,002 % max.
Iron(II) (Fe)	0,001 % max.
Lead (Pb)	0,001 % max.
Magnesium (Mg)	0,001 % max.
Manganese (Mn)	0,005 % max.
Potassium (K)	0,01 % max.
Sodium (Na)	0,01 % max.
Zinc (Zn)	0,003 % max.

R 43.2 Tests

R 43.2.1 Assay

Weigh, to the nearest 0,000 1 g, about 2 g of the sample, dissolve in 20 ml of water, add 5 ml of hydrochloric acid solution (15 %), 3 g of the potassium iodide (R 25) and set aside in the dark for 5 min.

Titrate with a standard volumetric sodium thiosulfate solution, $c(Na_2S_2O_3)$ = 0,1 mol/l, using the starch (IS 4.3.11*).

1,00 ml of sodium thiosulfate solution, $c(Na_2S_2O_3)$ = 0,100 mol/l, corresponds to 0,048 22 g of $NH_4Fe(SO_4)_2.12H_2O$.

R 43.2.2 Water-insoluble matter

Take 20 g of the sample and apply GM 1.

The mass of the residue shall not exceed 1 mg.

R 43.2.3 Chloride

Dissolve 2 g of the sample in 50 ml of water and apply GM 2.

Prepare a standard matching solution, using 1 ml of the chloride SS II (1 ml ≙ 0,0005 % Cl).

R 43.2.4 Copper, lead, magnesium, manganese and zinc

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

Element	Concentration of solution	Flame	Resonance line nm
Cu	10 %	Air-ethine	324,7
Pb			217,0 or 283,3
Mn			279,5
Mg	1 %		285,2
Zn			213,9

R 43.2.5 Iron(II)

Dissolve 1,5 g of the sample in 30 ml of water, add 1 ml of sulfuric acid (15 %) and 0,1 ml of a freshly prepared solution of potassium hexacyanoferrate(III) (5 %).

Compare any resulting blue colour with that of a solution prepared from 0,5 g of the sample, 30 ml of water, 1 ml of sulfuric acid (15 %), 0,1 ml of a freshly prepared solution of potassium hexacyanoferrate(III) (5 %) and 0,1 ml of a solution containing 0,5 g of $FeSO_4.7H_2O$ + 10 ml of sulfuric acid (15 %) in 1 000 ml, [0,1 ml ≙ 0,001 % Fe(II)].

R 43.2.6 Potassium and sodium

Determine these elements by FES according to GM 30, using the following conditions:

Element	Concentration of solution	Flame	Wavelength nm
K	2 %	Air-ethine	766,5
Na	1 %		589,0

R 44 Ammonium nitrate NH_4NO_3

Relative molecular mass: 80,04

R 44.1 Specification

Assay (NH_4NO_3)	99 % min.
Sulfated ash	0,01 % max.
pH (5 % solution)	4,5 to 6,0
Calcium (Ca)	0,003 % max.
Chloride (Cl)	0,000 5 % max.
Nitrite (NO_2)	0,000 5 % max.
Phosphate (PO_4)	0,001 % max.
Sulfate (SO_4)	0,005 % max.
Iron (Fe)	0,000 5 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.

R 44.2 Preparation of test solution

Dissolve 20 g of the sample in 100 ml of water (the solution shall be clear and colourless) and dilute to 200 ml.

R 44.3 Tests

R 44.3.1 Assay

Weigh, to the nearest 0,000 1 g, 3 g of the sample and dissolve in 50 ml of water.

To this solution add 20 ml of formaldehyde solution (R 63) previously mixed with 20 ml of water and neutralized with standard volumetric sodium hydroxide solution, c(NaOH) = 1 mol/l, using 0,2 ml of the phenolphthalein (IS 4.3.9*). Mix, allow to stand for 30 min, then titrate with standard volumetric sodium hydroxide solution, c(NaOH) = 1 mol/l, to a pink colour which persists for 5 min.

1,00 ml of sodium hydroxide solution, c(NaOH) = 1,000 mol/l, corresponds to 0,080 04 g of NH_4NO_3.

R 44.3.2 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 1 mg.

Reserve the residue for the determination of iron.

R 44.3.3 pH

Determine the pH of a 5 % solution of the sample according to GM 31.1, using a glass indicator electrode.

R 44.3.4 Calcium

Determine this element by AAS according to GM 29.

Element	Concentration of solution	Flame	Resonance line nm
Cu	10 %	Air-ethine	422,7

R 44.3.5 Chloride

Take 20 ml of the test solution (R 44.2) and apply GM 2.

Prepare a standard matching solution, using 1 ml of the chloride SS II (1 ml ≙ 0,000 5 % Cl).

R 44.3.6 Nitrite

Dissolve 2 g in 10 ml of water and 1 ml of 10 % sulfuric acid solution and 1 ml of *m*-phenylenediamine dihydrochloride solution (0,5 %).

The brown-yellowish colour shall not be more intense than that of a similarly prepared standard matching solution, using 1 ml of the nitrite SS II (1 ml ≙ 0,000 5 % NO_2).

NOTE — The phenylenediamine solution shall be colourless; otherwise shall be decolorized with active carbon.

R 44.3.7 Phosphate

Take 20 ml of the test solution (R 44.2) and apply GM 4.

Prepare a standard matching solution, using 2 ml of the phosphate SS II (2 ml ≙ 0,001 % PO_4).

R 44.3.8 Sulfate

Dissolve 2 g of the sample in 10 ml of warm water, add 1 ml of sodium carbonate solution (1 %), evaporate and ignite gently until the ammonium nitrate is volatilized. To the residue add 10 ml of water, 1 ml of hydrochloric acid solution (3,65 %) and a few drops of bromine water, and boil for 1 min. Filter if necessary, wash with water, dilute the solution to 20 ml and apply GM 3.

Prepare a standard matching solution, using 1 ml of the sulfate SS I (1 ml ≙ 0,002 % SO_4).

R 44 Ammonium nitrate

R 44.3.9 Iron

Determine this element by AAS according to GM 29, using the following conditions:

Element	Concentration of solution	Flame	Resonance line nm
Fe	Dissolve the residue "sulfated ash" with 3 ml of warm hydrochloric acid (R 13) and dilute with water to 50 ml.	Air-ethine	248,3

R 44.3.10 Heavy metals

Take 20 ml of the test solution (R 44.2) and apply GM 7.

Prepare a standard matching solution, using 1 ml of the lead SS II (1 ml ≙ 0,000 5 % Pb).

R 45 Ammonium sulfate $(NH_4)_2SO_4$

Relative molecular mass: 132,14

R 45.1 Specification

Assay [$(NH_4)_2SO_4$]	99,0 % min.
Sulfated ash	0,01 % max.
pH (5 % solution)	4,8 to 6,0
Chloride (Cl)	0,000 5 % max.
Phosphate (PO_4)	0,001 % max.
Arsenic (As)	0,000 02 % max.
Iron (Fe)	0,000 5 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.

R 45.2 Preparation of test solution

Dissolve 20 g of the sample in 100 ml of water (the solution shall be clear and colourless) and dilute to 200 ml.

R 45.3 Tests

R 45.3.1 Assay

Weigh, to the nearest 0,000 1 g, 2 g of the sample, and dissolve in 40 ml of water.

To this solution add 20 ml of formaldehyde solution (R 63) previously mixed with 20 ml of water and neutralized with standard volumetric sodium hydroxide solution, c(NaOH) = 1 mol/l, using 0,2 ml of the phenolphthalein (IS 4.3.9*). Mix, allow to stand for 30 min, then titrate with standard volumetric sodium hydroxide solution, c(NaOH) = 1 mol/l, to a pink colour which persists for 5 min.

1,00 ml of sodium hydroxide solution, c(NaOH) = 1,000 mol/l, corresponds to 0,066 07 g of $(NH_4)_2SO_4$.

R 45.3.2 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 1 mg.

Reserve the residue for the determination of iron.

R 45.3.3 pH

Determine the pH of a 5 % solution of the sample according to GM 31.1, using a glass indicator electrode.

R 45.3.4 Chloride

Take 20 ml of the test solution (R 45.2) and apply GM 2.

Prepare a standard matching solution, using 1 ml of the chloride SS II (1 ml ≙ 0,000 5 % Cl).

R 45.3.5 Phosphate

Take 20 ml of the test solution (R 45.2) and apply GM 4.

Prepare a standard matching solution, using 2 ml of the phosphate SS II (2 ml ≙ 0,001 % PO_4).

R 45.3.6 Arsenic

Take 50 ml of the test solution (R 45.2) and apply GM 11.

Prepare a standard matching solution, using 1 ml of the arsenic SS III (1 ml ≙ 0,000 02 % As).

R 45.3.7 Iron

Determine this element by AAS according to GM 29, using the following conditions:

Element	Concentration of solution	Flame	Resonance line nm
Fe	Dissolve the residue "sulfated ash" with 3 ml of warm hydrochloric acid (R 13) and dilute to 50 ml with water.	Air-ethine	248,3

R 45.3.8 Heavy metals

Take 20 ml of the test solution (R 45.2) and apply GM 7.

Prepare a standard matching solution, using 1 ml of the lead SS II (1 ml ≙ 0,000 5 % Pb).

R 46 Ammonium thiocyanate NH_4SCN

Relative molecular mass: 76,12

R 46.1 Specification

Assay (NH_4SCN)	98,0 % min.
pH (5 % solution)	4,5 to 6,0
Chloride (Cl)	0,005 % max.
Sulfate (SO_4)	0,005 % max.
Sulfide (S)	0,001 % max.
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,000 1 % max.
Lead (Pb)	0,000 5 % max.
Iodine-consuming substances (expressed as I)	0,025 % max.
Sulfated ash	0,03 % max.

R 46.2 Tests

R 46.2.1 Assay

Weigh, to the nearest 0,000 1 g, 0,28 to 0,32 g of the sample and dissolve in 50 ml of water.

To this solution add 5 ml of nitric acid solution (25 %) and 50,0 ml of standard volumetric silver nitrate solution, $c(AgNO_3)$ = 0,1 mol/l. Titrate with standard volumetric potassium thiocyanate solution, $c(KSCN)$ = 0,1 mol/l, using the ammonium iron(III) sulfate (IS 4.3.1*) to the first pink coloration.

1,00 ml of silver nitrate solution, $c(AgNO_3)$ = 0,100 mol/l, corresponds to 0,007 612 g of NH_4SCN.

R 46.2.2 pH

Determine the pH of a 5 % solution of the sample according to GM 31.1, using a glass indicator electrode.

R 46.2.3 Chloride

Dissolve 1 g of the sample in 15 ml of water, add 10 ml of the hydrogen peroxide (R 14) and 2 ml of sodium hydroxide solution (10 %). Warm and shake carefully. After the reaction is finished, add another 10 ml of the hydrogen peroxide (R 14), boil for 5 min, cool, add 10 ml of nitric acid solution (25 %) and dilute to 50 ml with water. Apply GM 2.

Prepare a standard matching solution, using 5 ml of the chloride SS II and the quantities of reagents used in the test (5 ml ≙ 0,005 % Cl).

R 46.2.4 Sulfate

Dissolve 2 g of the sample in 20 ml of water and apply GM 3.

Prepare a standard matching solution, using 1 ml of the sulfate SS I (1 ml ≙ 0,005 % SO_4).

R 46.2.5 Sulfide

Dissolve 2 g of the sample in 20 ml of water, add 20 ml of the ammonia (R 3) and 3 ml of silver nitrate solution (1,7 %).

WARNING — Ammoniacal silver solution can be explosive; the solution should therefore be discarded immediately.

Compare any darkening with that produced by the similar treatment of 2 ml of sulfide SS II prepared by diluting freshly prepared sulfide SS I (2 ml ≙ 0,001 % S).

R 46.2.6 Copper, iron and lead

Determine these elements by AAS according to the solvent extraction procedure of GM 35, using the following conditions:

Element	Concentration of solution	Flame	Resonance line nm
Cu	5 %	Air-ethine	324,7
Pb			217,0 or 283,3
Fe	20 %		248,3

R 46.2.7 Iodine-consuming substances

Dissolve 5 g of the sample in 45 ml of water, add 1 ml of sulfuric acid solution, (20 %), 1 g of the potassium iodide (R 25) and titrate with standard volumetric iodine solution, $c(1/2\ I_2)$ = 0,01 mol/l, using the starch (IS 4.3.11*).

The volume of titrant shall not exceed 1 ml (1 ml ≙ 0,025 % I).

R 46.2.8 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 3 mg.