
International Standard



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

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.

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	Hungary	Poland
Belgium	Ireland	Portugal
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Czechoslovakia	Korea, Dem. P. Rep. of	Switzerland
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The member bodies of the following countries expressed disapproval of the document on technical grounds :

India
USSR

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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 CH₃COOH

Relative molecular mass : 60,05

R 1.1 Specification

Assay (CH ₃ COOH)	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, $c(\text{NaOH}) = 1 \text{ mol/l}$, using the phenolphthalein (IS 4.3.9*).

1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 1,000 \text{ mol/l}$, corresponds to 0,060 05 g of CH₃COOH.

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 $\cong$ 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 $\cong$ 0,000 2 % SO₄).

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 $\cong$ 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 $50 \pm 2 \text{ }^\circ\text{C}$. 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, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,05 \text{ mol/l}$, 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, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,050 \text{ mol/l}$.

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

CH₃COCH₃

Relative molecular mass : 58,08

R 2.1 Specification

Assay (CH ₃ COCH ₃)	99,5 % min.
Methanol (CH ₃ OH)	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, $c(\text{NaOH}) = 0,01 \text{ mol/l}$, 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, $c(1/2 \text{ H}_2\text{SO}_4) = 0,01 \text{ mol/l}$, 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 $20,0 \pm 0,5 \text{ °C}$ 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 ($4 \text{ ml} \cong 0,002 \text{ % HCHO}$).

R 2.2.8 Water

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

R 3 Ammonia solution (25 %)

NH₃

Relative molecular mass : 17,03

R 3.1 Specification

Assay (NH ₃)	25 % min. ¹⁾
Carbonate (expressed as CO ₂)	0,002 % max.
Chloride (Cl)	0,000 05 % max.
Phosphate (PO ₄)	0,000 2 % max.
Silicate (expressed as SiO ₂)	0,001 % max.
Sulfate (SO ₄)	0,000 2 % max.
Sulfide (S)	0,000 02 % max.
Calcium (Ca)	0,000 1 % max.
Copper (Cu)	0,000 01 % max.
Iron (Fe)	0,000 02 % max.
Magnesium (Mg)	0,000 1 % max.
Lead (Pb)	0,000 05 % max.
Potassium (K)	0,000 1 % max.
Sodium (Na)	0,000 5 % max.
Permanganate-reducing substances (expressed as O)	0,000 8 % max.
Residue after evaporation	0,002 % max.

R 3.2 Preparation of test solutions and of mixed indicator solution

R 3.2.1 Test solution I

Evaporate 250 g (275 ml) of the sample¹⁾ to a volume of about 10 ml. Acidify with the nitric acid (R 19) and dilute to 100 ml with water.

R 3.2.2 Test solution II

Dissolve the residue after evaporation obtained in R 3.3.12 in 0,5 ml of the hydrochloric acid (R 13) and dilute to 100 ml with water.

R 3.2.3 Mixed indicator solution

Mix 1 volume of methyl red solution [0,2 % in 95 % (V/V) ethanol] with 1 volume of methylene blue solution [0,1 % in 95 % (V/V) ethanol].

R 3.3 Tests

R 3.3.1 Assay

Place 25,00 ml of standard volumetric sulfuric acid solution, $c(1/2 \text{ H}_2\text{SO}_4) = 1 \text{ mol/l}$, in a 100 ml conical flask fitted with a ground glass stopper and weigh to the nearest 0,000 1 g.

Add about 1,2 g of the sample and re-weigh to the nearest 0,000 1 g. Add 2 drops of the mixed indicator solution (R 3.2.3) and titrate the excess of acid with standard volumetric sodium hydroxide solution $c(\text{NaOH}) = 1 \text{ mol/l}$.

1,00 ml of sulfuric acid solution, $c(1/2 \text{ H}_2\text{SO}_4) = 1,000 \text{ mol/l}$, corresponds to 0,017 03 g of NH₃.

R 3.3.2 Carbonate

Dilute 5 g (5,5 ml) of the sample with 10 ml of carbon dioxide-free water and add 5 ml of a 4 % solution of barium hydroxide octahydrate [Ba(OH)₂·8H₂O].

After 5 min, any turbidity in the test solution shall not be greater than that of a similarly prepared standard matching solution using 10 ml of the carbonate SS II (10 ml $\cong$ 0,002 % CO₂).

R 3.3.3 Chloride

Take 20 ml of the test solution I (R 3.2.1) and apply GM 2.

Prepare a standard matching solution, using 2,5 ml of the chloride SS II (2,5 ml $\cong$ 0,000 05 % Cl).

R 3.3.4 Phosphate

Take 20 ml of the test solution I (R 3.2.1) and apply GM 4.

Prepare a standard matching solution, using 10 ml of the phosphate SS II (10 ml $\cong$ 0,000 2 % PO₄).

R 3.3.5 Silicate

Neutralize 20 g (22 ml) of the sample with the hydrochloric acid (R 13), dilute to approximately 50 ml and apply GM 5.

¹⁾ The same quality is also commercially available with an NH₃ content of about 30 % or 35 % and allowance should be made for this when preparing test solution I.

R 3 Ammonia solution (25 %)

Prepare a standard matching solution, using 20 ml of the silicate SS II (20 ml $\pm$ 0,001 % SiO₂).

R 3.3.6 Sulfate

Take 10 ml of the test solution I (R 3.2.1) and apply GM 3.

Prepare a standard matching solution, using 5 ml of the sulfate SS II (5 ml $\pm$ 0,000 2 % SO₄).

R 3.3.7 Sulfide

To 50 g (55 ml) of the sample, add 0,5 ml of the lead acetate (basic) (RS 4.2.10*).

The brown coloration of the test solution shall not be more intense than that of a similarly prepared standard matching solution using 1 ml of the freshly prepared sulfide SS II (1 ml $\pm$ 0,000 02 % S).

R 3.3.8 Calcium, copper, magnesium and lead

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	Test solution II (R 3.2.2)	Air-acetylene	422,7
Cu			324,7
Mg			285,2
Pb			217,0 or 283,3

R 3.3.9 Iron

Evaporate 50 g (55 ml) of the sample to dryness on a boiling water bath. Take up the residue in 0,5 ml of the hydrochloric acid (R 13) and apply GM 8.2.

Prepare a standard matching solution, using 10 ml of the iron SS III (10 ml $\pm$ 0,000 02 % Fe).

R 3.3.10 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 II (R 3.2.2)	Oxygen-acetylene	766,5
Na			589,0

R 3.3.11 Permanganate-reducing substances

To 20 g (22 ml) of the sample, add 20 ml water, mix thoroughly and acidify cautiously with 20 ml of sulfuric acid solution (50 %). Add 0,2 ml of 3,16 g/l potassium permanganate solution and boil for 5 min.

The pink colour shall not be completely discharged.

R 3.3.12 Residue after evaporation

Take 100 g (110 ml) of the sample and apply GM 14.

The mass of the residue shall not exceed 2 mg.

Retain this residue for the preparation of the test solution II (R 3.2.2).

R 4 Ammonium acetate

CH₃COONH₄

Relative molecular mass : 77,08

R 4.1 Specification

Assay (CH ₃ COONH ₄)	99 % min.
pH (5 % solution) : 6,5 to 7,5	
Chloride (Cl)	0,000 5 % max.
Nitrate (NO ₃)	0,001 % max.
Sulfate (SO ₄)	0,002 % max.
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,000 5 % max.
Sulfated ash	0,01 % max.

R 4.2 Preparation of test solution

Dissolve 40 g of the sample in 100 ml of water (the solution shall be clear and colourless) and dilute to 200 ml.

R 4.3 Tests

R 4.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 1,5 g of the sample and dissolve in about 20 ml of water.

To this solution add a mixture, previously neutralized against the phenolphthalein (IS 4.3.9*), of 25 ml of formaldehyde solution (35 %) and 25 ml of water. After 30 min, add 0,2 ml of the phenolphthalein IS and titrate with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 1 \text{ mol/l}$, to a faint pink colour.

1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 1,000 \text{ mol/l}$, corresponds to 0,077 08 g of CH₃COONH₄.

R 4.3.2 pH

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

R 4.3.3 Chloride

Take 20 ml of the test solution (R 4.2) and 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 $\cong$ 0,000 5 % Cl).

R 4.3.4 Nitrate

Take 5 ml of the test solution (R 4.2) and add 0,2 ml of brucine solution (0,5 %) in the acetic acid (R 1) and, cautiously add while cooling and mixing, 10 ml of the sulfuric acid (R 37).

The yellow coloration of the test solution shall not be more intense than that of a similarly prepared standard matching solution using 1 ml of the nitrate SS II (1 ml $\cong$ 0,001 % NO₃).

R 4.3.5 Sulfate

Take 10 ml of the test solution (R 4.2) and apply GM 3.

Prepare a standard matching solution, using 4 ml of the sulfate SS II (4 ml $\cong$ 0,002 % SO₄).

R 4.3.6 Copper and lead

Determine these elements by AAS according to GM 29, using 30 g of the sample and the following conditions :

Element	Concentration of solution	Flame	Resonance line nm
Cu	Test solution (R 4.2) Use the extraction solvent in GM 35	Air-acetylene	324,7
Pb			217,0 or 283,3

R 4.3.7 Iron

Take 20 ml of the test solution (R 4.2) and apply GM 8.1.

Prepare a standard matching solution, using 2 ml of the iron SS II (2 ml $\cong$ 0,000 5 % Fe).

R 4.3.8 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 1 mg.

R 5 Ammonium chloride NH₄Cl

Relative molecular mass : 53,49

R 5.1 Specification

Assay (NH ₄ Cl)	99,5 % min.
pH (5 % solution) : 4,5 to 5,5	
Phosphate (PO ₄)	0,000 5 % max.
Sulfate (SO ₄)	0,002 % max.
Calcium (Ca)	0,001 % max.
Iron (Fe)	0,000 2 % max.
Magnesium (Mg)	0,001 % max.
Potassium (K)	0,005 % max.
Sodium (Na)	0,005 % max.
Residue after ignition (at 650 °C)	0,05 % max.

R 5.2 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 5.3 Tests

R 5.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 1 g of the sample and dissolve in about 20 ml of water.

To this solution add a mixture, previously neutralized against the phenolphthalein (IS 4.3.9*), of 25 ml of formaldehyde solution (35 %) and 25 ml of water. After 30 min, add 0,2 ml of the phenolphthalein IS and titrate with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 1 \text{ mol/l}$, to a faint pink colour.

1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 1,000 \text{ mol/l}$, corresponds to 0,053 49 g of NH₄Cl.

R 5.3.2 pH

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

R 5.3.3 Phosphate

Take 50 ml of the test solution (R 5.2), add 30 ml of water and apply GM 4.

Prepare a standard matching solution, using 2,5 ml of the phosphate SS II (2,5 ml $\cong$ 0,000 5 % PO₄).

R 5.3.4 Sulfate

Take 20 ml of the test solution (R 5.2) and apply GM 3.

Prepare a standard matching solution, using 4 ml of the sulfate SS II (4 ml $\cong$ 0,002 % SO₄).

R 5.3.5 Calcium and magnesium

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	4 %	Air-acetylene	422,7
Mg			285,2

R 5.3.6 Iron

Take 20 ml of the test solution (R 5.2) and apply GM 8.1.

Prepare a standard matching solution, using 0,4 ml of the iron SS II (0,4 ml $\cong$ 0,000 2 % Fe).

R 5.3.7 Potassium and sodium

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

Element	Concentration of solution	Flame	Wavelength nm
K	4 %	Oxygen-acetylene	766,5
Na			589,0

R 5.3.8 Residue after ignition

Take 2 g of the sample and apply GM 15.

The mass of the residue shall not exceed 1 mg.

R 6 Barium chloride dihydrate

BaCl₂·2H₂O

Relative molecular mass : 244,28

R 6.1 Specification

Assay (BaCl ₂ ·2H ₂ O)	99,0 % min.
pH (5 % solution) : 5 to 8	
Total nitrogen (N)	0,002 % max.
Calcium (Ca)	0,05 % max.
Iron (Fe)	0,000 2 % max.
Lead (Pb)	0,001 % max.
Potassium (K)	0,005 % max.
Sodium (Na)	0,01 % max.
Strontium (Sr)	0,05 % max.

R 6.2 Tests

R 6.2.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,2 g of the sample and dissolve in about 100 ml of hot water.

To this solution, add 100 ml of the methanol (R 18) and 10 ml of the ammonia solution (R 3) and titrate with standard volumetric EDTA solution, $c(\text{EDTA}) = 0,1 \text{ mol/l}$, using the methylthymol blue mixture (4.3.7*).

1,00 ml of EDTA solution, $c(\text{EDTA}) = 0,100 \text{ mol/l}$, corresponds to 0,024 43 g of BaCl₂·2H₂O.

R 6.2.2 pH

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

R 6.2.3 Total nitrogen

Dissolve 1 g of the sample in 140 ml of water and apply GM 6.

Prepare a standard matching solution, using 2 ml of the nitrogen SS II (2 ml $\cong$ 0,002 % N).

R 6.2.4 Calcium, iron, lead and strontium

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	2 %	Air-acetylene	422,7
Fe	10 %		248,3
Pb	Use the extraction solvent in GM 35		217,0 or 283,3
Sr	2 %		460,7

R 6.2.5 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 %	Oxygen-acetylene	766,5
Na			589,0

R 7 Chloroform CHCl₃

Relative molecular mass : 119,38

R 7.1 Specification

Assay (CHCl ₃)	99 % min.
Ethanol (stabilizer)	0,3 to 1,0 %
Density : 1,471 to 1,484 g/ml	
Residue after evaporation	0,001 % max.
Acidity (expressed in millimoles of H ⁺)	0,03/100 g max.
Readily carbonizable substances	To pass test
Carbonyl compounds (expressed as CO)	0,005 % max.
Free chlorine	0,000 5 % max.
Chloride (Cl)	0,000 1 % max.
Metallic impurities	To pass test

$c(\text{NaOH}) = 0,01 \text{ mol/l}$, and using the phenolphthalein (IS 4.3.9*).

The volume of titrant shall not exceed 2,2 ml.

R 7.2.5 Readily carbonizable substances

Vigorously shake 20 ml of the sample with 15 ml of sulfuric acid (95,0 ± 0,5 %) and 0,2 ml of formaldehyde solution [40 % (V/V)] in a glass cylinder fitted with a ground glass stopper for 3 min. Allow the mixture to stand at room temperature for 30 min.

The volume of titrant shall not exceed 2,2 ml.

R 7.2 Test

R 7.2.1 Assay and ethanol

Apply GM 34, using the following conditions :

Stationary phase	20 % bis(2-ethylhexyl) sebacate
Support	Chromosorb W [0,18 to 0,25 mm 60 to 80 mesh ASTM]
Column length	2 m
Column internal diameter	5 mm
Column material	Stainless steel
Column temperature	60 °C
Injection temperature	150 °C
Detection temperature	200 °C
Type of detector	Thermal conductivity
Carrier gas	Helium
Flow rate	50 ml/min
Test portion	5 µl

R 7.2.6 Carbonyl compounds

Dilute 3 g (2 ml) of the sample to 15 ml with the carbonyl-free methanol (RS 4.2.11*). Take 2 ml of this solution and apply GM 23.

Prepare a standard matching solution, using 2 ml of the carbonyl SS II (2 ml ≅ 0,005 % CO).

R 7.2.7 Free chlorine

Shake 35 g (24 ml) of the sample with 10 ml of water to which have been added 1 ml of freshly prepared potassium iodide solution (10 %) and 2 drops of the starch (IS 4.3.11*) in a conical flask fitted with a ground glass stopper for 2 min. If either a blue or a violet colour is obtained, titrate with standard volumetric sodium thiosulfate solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,01 \text{ mol/l}$, shaking after each addition.

The volume of titrant shall not exceed 0,5 ml.

R 7.2.8 Chloride

To 10 g (6,8 ml) of the sample in a separating funnel, add 20 ml of water and 1 ml of nitric acid solution (25 %). Shake for 1 min, allow the liquid phases to separate and reject the lower (chloroform) phase. Filter the aqueous phase through a chloride-free filter paper, add 1 ml of approximately 17 g/l silver nitrate solution and allow to stand for 2 min.

Any opalescence shall not be greater than that of a similarly prepared standard matching solution, using 1 ml of the chloride SS II (1 ml ≅ 0,000 1 % Cl).

R 7.2.9 Metallic impurities

To 50 ml of the sample, add 0,1 ml of a 0,1 % solution of dithizone in chloroform.

The colour of the test solution shall be green or bluish-green.

R 7.2.2 Density

Apply GM 24.1.

R 7.2.3 Residue after evaporation

Take 100 g (68 ml) of the sample and apply GM 14, drying the residue for 30 min.

The mass of the residue shall not exceed 1 mg.

R 7.2.4 Acidity

Take 74 g (50 ml) of the sample and apply GM 13.2, titrating with standard volumetric sodium hydroxide solution,

R 8 Citric acid monohydrate

$C_6H_8O_7 \cdot H_2O$

Relative molecular mass : 210,14

R 8.1 Specification

Assay ($C_6H_8O_7 \cdot H_2O$)	99,5 % min.
Chloride (Cl)	0,000 5 % max.
Oxalate (C_2O_4)	0,05 % max.
Phosphate (PO_4)	0,001 % max.
Sulfate (SO_4)	0,005 % max.
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,000 5 % max.
Readily carbonizable substances	To pass test
Sulfated ash	0,02 % max.

R 8.2 Preparation of test solutions and of reference solution

R 8.2.1 Test solution I

Dissolve 5 g of the sample in a small amount of water and dilute to 50 ml.

R 8.2.2 Test solution II

To 7 g of the sample, add 0,2 ml of ammonium vanadate solution (1 %), and 15 ml of the nitric acid (R 19). Digest in a covered beaker on a boiling water bath until the reaction ceases; remove the cover and evaporate to dryness.

Add 10 ml of the nitric acid (R 19) and repeat the digestion and evaporation. Add 5 ml of hydrochloric acid solution (20 %) and evaporate to dryness. Dissolve the residue in a small amount of water containing 1 ml of hydrochloric acid solution (20 %), filter if necessary and dilute to 50 ml.

R 8.2.3 Test solution III

Dissolve the residue obtained in R 8.3.9 (sulfated ash), contained in the crucible or dish used, in 10 ml of hydrochloric acid solution (10 %). Cover the container, digest on a boiling water bath for 10 min, and dilute to 50 ml.

R 8.2.4 Reference solution

To 2 g of the sample, add 5 ml of the phosphate SS I (5 ml $\cong$ 0,001 % PO_4) and 2,5 ml of the sulfate SS I (2,5 ml $\cong$ 0,005 % SO_4) and proceed as for the preparation of the test solution II, using the same quantities of all the reagents.

R 8.3 Tests

R 8.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 2,5 g of the sample and dissolve in 100 ml of water.

To this solution, add 2 drops of the phenolphthalein (IS 4.3.9*) and titrate with standard volumetric sodium hydroxide solution, $c(NaOH) = 1 \text{ mol/l}$, to a pink colour that persists for 3 min.

1,00 ml of sodium hydroxide solution, $c(NaOH) = 1,000 \text{ mol/l}$, corresponds to 0,070 05 g of $C_6H_8O_7 \cdot H_2O$.

R 8.3.2 Chloride

Take 20 ml of the test solution I (R 8.2.1) and apply GM 2.

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

R 8.3.3 Oxalate

Dissolve 0,2 g of the sample in 10 ml of water and apply GM 22.

Prepare a standard matching solution, using 10 ml of the oxalate SS II (10 ml $\cong$ 0,05 % C_2O_4).

R 8.3.4 Phosphate

Take 20 ml of the test solution II (R 8.2.2) and apply GM 4.

Prepare a standard matching solution, using 20 ml of the reference solution (R 8.2.4) (20 ml $\cong$ 0,001 % PO_4).

R 8.3.5 Sulfate

Take 10 ml of the test solution II (R 8.2.2) and apply GM 3.

Prepare a standard matching solution, using 10 ml of the reference solution (R 8.2.4) (10 ml $\cong$ 0,005 % SO_4).

R 8.3.6 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 III (R 8.2.3)	Air-acetylene	324,7
Pb			217,0 or 283,3

R 8 Citric acid monohydrate

R 8.3.7 Iron

Take 20 ml of the test solution I (R 8.2.1) and apply GM 8.1.

Prepare a standard matching solution, using 1 ml of the iron SS II ($1 \text{ ml} \triangleq 0,0005 \% \text{ Fe}$).

R 8.3.8 Readily carbonizable substances

Take 0,3 g of powdered sample and apply GM 18, using 10 ml of sulfuric acid solution ($95 \pm 0,5 \%$) and heating the mixture at 110°C for 30 min.

The colour of the resulting solution shall not be more intense than that of the standard solution G (see GM 18).

R 8.3.9 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 2 mg.

Retain this residue for the preparation of the test solution III (R 8.2.3).

R 9 Copper(II) sulfate pentahydrate CuSO₄·5H₂O

Relative molecular mass : 249,68

R 9.1 Specification

Assay (CuSO ₄ ·5H ₂ O)	99,0 % min.
Chloride (Cl)	0,001 % max.
Total nitrogen (N)	0,003 % max.
Iron (Fe)	0,005 % max.
Nickel (Ni)	0,005 % max.
Potassium (K)	0,001 % max.
Sodium (Na)	0,005 % max.
Zinc (Zn)	0,05 % max.

R 9.2 Preparation of test solution

Dissolve 10 g of the sample in 50 ml of water and dilute to 100 ml.

R 9.3 Tests

R 9.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 1 g of the sample, and dissolve in about 60 ml of water.

To this solution add 5 ml of sulfuric acid solution (20 %) and 3 g of the potassium iodide (R 25). Titrate the liberated iodine with standard volumetric sodium thiosulfate solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}$, adding the starch (IS 4.3.11*) towards the end of the titration.

1,00 ml of sodium thiosulfate solution, $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,100 \text{ mol/l}$, corresponds to 0,024 97 g of CuSO₄·5H₂O.

R 9.3.2 Chloride

Take 10 ml of the test solution (R 9.2), dilute to 20 ml with water, and apply GM 2.

Prepare a standard matching solution, using 1 ml of the chloride SS II (1 ml $\cong$ 0,001 % Cl).

R 9.3.3 Total nitrogen

Take 10 ml of the test solution (R 9.2) and apply GM 6.

Prepare a standard matching solution, using 3 ml of the nitrogen SS II (3 ml $\cong$ 0,003 % N).

R 9.3.4 Iron

To 10 ml of the test solution (R 9.2), add 5 ml of the hydrochloric acid (R 13) and 1 drop of the nitric acid (R 19). Extract with 20 ml of 4-methyl-2-pentanone and wash the organic phase with 10 ml of hydrochloric acid solution (25 %).

Extract the iron from the organic phase with 20 ml of water. Separate the aqueous phase and apply GM 8.1.

Prepare a standard matching solution, using 5 ml of the iron SS II (5 ml $\cong$ 0,005 % Fe).

R 9.3.5 Nickel and zinc

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

Element	Concentration of solution	Flame	Resonance line nm
Ni	1 %	Air-acetylene	232,0 or 325,5
Zn			213,9

R 9.3.6 Potassium and sodium

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

Element	Concentration of solution	Flame	Wavelength nm
K	Test solution (R 9.2)	Oxygen-acetylene	766,5
Na			589,0

R 10 Cyclohexane C₆H₁₂

Relative molecular mass : 84,16

R 10.1 Specification

Assay (C ₆ H ₁₂)	99,5 % min.
Cyclohexene (C ₆ H ₁₀)	0,05 % max.
Benzene (C ₆ H ₆)	0,05 % max.
Density : 0,778 to 0,779 g/ml	
Freezing point	6,0 °C min.
Residue after evaporation	0,002 % max.
Readily carbonizable substances	To pass test
Water	0,01 % max.

R 10.2 Tests

R 10.2.1 Assay, cyclohexene and benzene

Apply GM 34, using the following conditions :

Stationary phase	5 % bentone + 5 % isodecyl phthalate
Support	Chromosorb W [0,18 to 0,25 mm 60 to 80 mesh ASTM]
Column length	4 m
Column internal diameter	4 mm
Column material	Stainless steel
Column temperature	80 °C
Injection temperature	150 °C
Detection temperature	200 °C
Type of detector	Thermal conductivity
Carrier gas	Helium
Flow rate	50 ml/min
Test portion	5 µl

R 10.2.2 Density

Apply GM 24.1.

R 10.2.3 Freezing point

Apply GM 25.3.

R 10.2.4 Residue after evaporation

Take 100 g (130 ml) of the sample and apply GM 14, drying the residue for 30 min.

The mass of the residue shall not exceed 2 mg.

R 10.2.5 Readily carbonizable substances

Take 10 ml of the sample, and apply GM 18, using 10 ml of sulfuric acid solution (95 ± 0,5 %), while cooling to 10 °C, and allow to stand for 5 min.

No darkening of the lower acid phase shall occur.

R 10.2.6 Water

Take 15,5 g (20 ml) of the sample and apply GM 12, using a mixture of 20 ml of the methanol (R 18), 10 ml of the chloroform (R 7) and 2 ml of the acetic acid (R 1) as the solvent.

R 11 Ethanol C₂H₅OH

Relative molecular mass : 46,07

R 11.1 Specification

Assay (C ₂ H ₅ OH)	99,8 % min.
Methanol (CH ₃ OH)	0,05 % max.
2-Propanol	0,01 % max.
Density : 0,789 to 0,791 g/ml	
Residue after evaporation	0,001 % max.
Acidity (expressed in millimoles of H ⁺) ...	0,035/100 g max.
Alkalinity (expressed in millimoles of OH ⁻)	0,02/100 g max.
Permanganate-reducing substances (expressed as O)	0,000 4 % max.
Readily carbonizable substances	To pass test
Higher alcohols	0,01 % max.
Carbonyl compounds (expressed as CO) ..	0,003 % max.
Water	0,2 % max.

R 11.2 Tests

R 11.2.1 Assay, methanol and 2-propanol

Apply GM 34, using the following conditions :

Stationary phase	Porapak QS [0,15 to 0,18 mm (80 to 100 mesh ASTM)]
Column length	3 m
Column internal diameter	2,5 mm
Column material	Stainless steel or, preferably, glass
Column temperature	130 °C
Injection temperature	150 °C
Detection temperature	150 °C
Type of detector	Flame ionization
Carrier gas	Nitrogen (or helium)
Flow rate	25 ml/min ¹⁾
Test portion	0,5 µl

R 11.2.2 Density

Apply GM 24.1.

R 11.2.3 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 11.2.4 Acidity

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

The volume of titrant shall not exceed 2,8 ml.

R 11.2.5 Alkalinity

Take 79 g (100 ml) of the sample, and apply GM 13.1, titrating with standard volumetric sulfuric acid solution, $c(1/2 \text{ H}_2\text{SO}_4) = 0,01 \text{ mol/l}$, and using the methyl red (IS 4.3.6*).

The volume of titrant shall not exceed 1,6 ml.

R 11.2.6 Permanganate-reducing substances

Take 20 g (25 ml) of the sample and apply GM 19.1, using 0,1 ml of potassium permanganate solution (3,16 g/l) and allowing the test solution to stand at 25 °C for 5 min.

The pink colour shall not be completely discharged.

R 11.2.7 Readily carbonizable substances

Cool 10 ml of sulfuric acid ($95,4 \pm 0,5 \%$) to 10 °C and add, drop by drop, with constant stirring, 10 ml of the sample, keeping the temperature of the mixture below 20 °C throughout.

The resulting solution shall be colourless.

R 11.2.8 Higher alcohols

Apply GM 34, using the following conditions :

Stationary phase	8 % Reoplex 400 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
Column temperature	80 to 200 °C
Temperature program	2 °C/min
Injection temperature	250 °C
Detection temperature	250 °C
Type of detector	Flame ionization

¹⁾ A higher flow rate is more suitable for some apparatus.

R 11 Ethanol

Carrier gas Nitrogen
Flow rate 25 ml/min
Test portion 1 μ l

R 11.2.9 Carbonyl compounds

Take 2 g (2,5 ml) of the sample and apply GM 23.

Prepare a standard matching solution using 6 ml of the carbonyl SS II (6 ml $\cong$ 0,003 % CO).

R 11.2.10 Water

Take 8 g (10 ml) of the sample and apply GM 12, using 20 ml of the methanol (R 18) as the solvent.

R 12 (Ethylenedinitrilo)tetraacetic acid, disodium salt dihydrate $C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$

Relative molecular mass : 372,24

R 12.1 Specification

pH (5 % solution) : 4 to 5

Assay ($C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$)	99 % min.
Chloride (Cl)	0,004 % max.
Nitrilotriacetic acid ($C_6H_6NO_6$)	0,05 % max.
Sulfate (SO_4)	0,02 % max.
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,001 % max.
Lead (Pb)	0,001 % max.

R 12.2 Tests

R 12.2.1 pH

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

R 12.2.2 Assay

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

To this solution add 10 ml of the buffer solution indicated below and titrate with standard volumetric zinc chloride solution, $c(1/2 ZnCl_2) = 0,2 \text{ mol/l}$, to a pink end-point, using the mordant black 11 mixture (4.3.8*).

1,00 ml of zinc chloride solution, $c(1/2 ZnCl_2) = 0,200 \text{ mol/l}$, corresponds to 0,037 22 g of $C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$.

Buffer solution : Dissolve 67,5 g of the ammonium chloride (R 5) in 150 ml of water, add 800 ml of the ammonia solution (R 3), dilute to 1 000 ml with water and mix.

R 12.2.3 Chloride

Dissolve 2,5 g of the sample in 40 ml of warm water, add 3 ml of nitric acid solution (25 %), cool, filter and apply GM 2.

Prepare a standard matching solution, using 10 ml of the chloride SS II (10 ml $\cong$ 0,004 % Cl).

R 12.2.4 Nitrilotriacetic acid

Dissolve 2 g of the sample in 35 ml of water and adjust to pH 2 with hydrochloric acid solution (20 %). Boil for 30 min and cool. Add sodium hydroxide solution (20 %) until the solution has a pH between 10 and 11.

Add cadmium chloride solution (5 %), drop by drop, keeping the pH between 10 and 11, until a slight white precipitate is formed. Filter, wash the precipitate, and dilute the combined filtrate and washings to 100 ml. To 50 ml, add 0,5 mg of nitrilotriacetic acid and polarograph according to GM 32 against the other 50 ml, starting at $E = -0,6 \text{ V}$.

The peak height of the sample solution without the added nitrilotriacetic acid shall not be greater than half the peak height of the solution containing the added nitrilotriacetic acid.

R 12.2.5 Sulfate

Take 0,5 g of the sample, mix with 0,2 g of the anhydrous sodium carbonate (R 30), and ignite in a platinum crucible. Dissolve the residue in 1 ml of hydrochloric acid solution (20 %) and 19 ml of water, and apply GM 3.

Prepare a standard matching solution, using 10 ml of the sulfate SS II (10 ml $\cong$ 0,02 % SO_4).

R 12.2.6 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	10 %	Air-acetylene	324,7
Fe			248,3
Pb			217,0 or 283,3