INTERNATIONAL STANDARD ISO 6353/2-1983/ADDENDUM 2

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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.

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, $c(\mathrm{HCl}) = 1$ 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, $c(\mathrm{HCl}) \approx 1$ 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 $\overline{x}$ and the standard deviation s, using the following equations:

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

and

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

where

x_i is an individual value;

N is the number of values measured.

W-71-25

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.

SO 6353/2-1983 (E)

Price based on 57 pages

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
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

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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
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 $\triangleq$ 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.

R 3 Ammonia solution (25 %)
NH_3

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 sample[1] 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, $c(1/2\ H_2SO_4) = 1$ 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 $c(NaOH) = 1$ mol/l.

1,00 ml of sulfuric acid solution, $c(1/2\ H_2SO_4) = 1{,}000$ mol/l, corresponds to 0,017 03 g of NH_3.

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 [$Ba(OH)_2.8H_2O$].

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 ≙ 0,002 % CO_2).

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 ≙ 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 ≙ 0,000 2 % PO_4).

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.

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[1] The same quality is also commercially available with an NH_3 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 ≙ 0,001 % SiO_2).

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 ≙ 0,000 2 % SO_4).

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 ≙ 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 ≙ 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 CH_3COONH_4

Relative molecular mass : 77,08

R 4.1 Specification

Assay (CH_3COONH_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, $c(NaOH) = 1$ mol/l, to a faint pink colour.

1,00 ml of sodium hydroxide solution, $c(NaOH) = 1,000$ mol/l, corresponds to 0,077 08 g of CH_3COONH_4.

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 ≙ 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 ≙ 0,001 % NO_3).

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 ≙ 0,002 % SO_4).

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 ≙ 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
NH_4Cl

Relative molecular mass : 53,49

R 5.1 Specification

Assay (NH_4Cl)	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, $c(NaOH) = 1$ mol/l, to a faint pink colour.

1,00 ml of sodium hydroxide solution, $c(NaOH) = 1,000$ mol/l, corresponds to 0,053 49 g of NH_4Cl.

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 $\triangleq$ 0,000 5 % PO_4).

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 $\triangleq$ 0,002 % SO_4).

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 $\triangleq$ 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 $BaCl_2.2H_2O$

Relative molecular mass : 244,28

R 6.1 Specification

Assay ($BaCl_2.2H_2O$)	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, $c(\mathrm{EDTA}) = 0{,}1$ mol/l, using the methylthymol blue mixture (4.3.7*).

1,00 ml of EDTA solution, $c(\mathrm{EDTA}) = 0{,}100$ mol/l, corresponds to 0,024 43 g of $BaCl_2.2H_2O$.

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 ≙ 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 % Use the extraction solvent in GM 35		248,3
Pb			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
$CHCl_3$

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

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 7.2.2 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, $c(\text{NaOH}) = 0{,}01$ 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 7.2.6 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, $c(Na_2S_2O_3) = 0{,}01$ 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 8 Citric acid monohydrate
$C_6H_8O_7.H_2O$

Relative molecular mass : 210,14

R 8.1 Specification

Assay ($C_6H_8O_7.H_2O$)	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 ≙ 0,001 % PO_4) and 2,5 ml of the sulfate SS I (2,5 ml ≙ 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 mol/l, to a pink colour that persists for 3 min.

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

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 ≙ 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 ≙ 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 ≙ 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 ≙ 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 ml ≙ 0,000 5 % 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 ± 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 $CuSO_4.5H_2O$

Relative molecular mass : 249,68

R 9.1 Specification

Assay ($CuSO_4.5H_2O$)	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, $c(Na_2S_2O_3) = 0,1$ mol/l, adding the starch (IS 4.3.11*) towards the end of the titration.

1,00 ml of sodium thiosulfate solution,
$c(Na_2S_2O_3) = 0,100$ mol/l, corresponds to 0,024 97 g of $CuSO_4.5H_2O$.

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 ≙ 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 ≙ 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 ≙ 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
C_6H_{12}

Relative molecular mass : 84,16

R 10.1 Specification

Assay (C_6H_{12})	99,5 % min.
Cyclohexene (C_6H_{10})	0,05 % max.
Benzene (C_6H_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
C_2H_5OH

Relative molecular mass : 46,07

R 11.1 Specification

Assay (C_2H_5OH)	99,8 % min.
Methanol (CH_3OH)	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/min1)
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, $c(\text{NaOH}) = 0{,}01$ 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, $c(1/2\ H_2SO_4) = 0{,}01$ 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 ± 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

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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 µl

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 $\triangleq$ 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 $C_{10}H_{14}N_2Na_2O_8.2H_2O$

Relative molecular mass : 372,24

R 12.1 Specification

pH (5 % solution) : 4 to 5

Assay ($C_{10}H_{14}N_2Na_2O_8.2H_2O$)	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, $c(1/2\ ZnCl_2) = 0,2$ mol/l, to a pink end-point, using the mordant black 11 mixture (4.3.8*).

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

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 ≙ 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 $E = -0,6$ 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 ≙ 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

R 13 Hydrochloric acid
HCl

Relative molecular mass : 36,46

R 13.1 Specification

Coloration in Hazen units	10 max.
Assay (HCl)	35 % min.[1]
Free chlorine (Cl)	0,000 2 % max.
Sulfate (SO_4)	0,000 2 % max.
Sulfite (SO_3)	0,000 2 % max.
Ammonium (NH_4)	0,000 3 % max.
Arsenic (As)	0,000005 % max.
Copper (Cu)	0,000 01 % max.
Lead (Pb)	0,000 02 % max.
Iron (Fe)	0,000 05 % max.
Sulfated ash	0,000 5 % max.

R 13.2 Preparation of test solution

Evaporate 200 g (168 ml) of the sample in a rotary evaporator to a volume of about 1 ml in a suitable air bath. Transfer quantitatively to a 50 ml one-mark volumetric flask, dilute to the mark and mix.

R 13.3 Tests

R 13.3.1 Coloration in Hazen units

Apply GM 36.

R 13.3.2 Assay

Weigh, to the nearest 0,000 1 g, a thin glass ampoule of suitable capacity provided with a capillary tube at one end.

Place about 2 ml of the sample in the ampoule, using for example the heating technique. Seal the ampoule without loss of glass, and weigh to the nearest 0,000 1 g.

Calculate by difference the mass of the test portion. Place the ampoule in a 500 ml conical flask containing 200 ml of water, and close the flask with a ground glass stopper. Shake the flask in order to break the ampoule and continue shaking until all vapour has been absorbed completely. Then open the flask, rinse the stopper and the wall of the flask carefully with water and grind the pieces of the broken ampoule thoroughly by means of a glass rod. Titrate the solution with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 1$ mol/l, using the methyl red (IS 4.3.6*).

1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 1{,}000$ mol/l, corresponds to 0,036 46 g of HCl.

R 13.3.3 Free chlorine

To 10 g (8,4 ml) of the sample add 25 ml of water and 1 ml of *o*-tolidine solution (0,1 % in hydrochloric acid 3 %).

Any yellow coloration in the test solution shall not be more intense than that of a similarly prepared standard matching solution, using 2 ml of the chlorine SS II (2 ml ≙ 0,000 2 % Cl) and 10 g of chlorine-free hydrochloric acid.[2]

R 13.3.4 Sulfate

Take 10 ml of the test solution (R 13.2) and apply GM 3.

Prepare a standard matching solution, using 8 ml of the sulfate SS II (8 ml ≙ 0,000 2 % SO_4).

R 13.3.5 Sulfite

In a 500 ml conical flask, mix 50 ml of water, 1 ml of potassium iodide solution (10 %) and 5 ml of the sample. Add 2 ml of the starch (IS 4.3.11*) and, if necessary, add, drop by drop, standard volumetric iodine solution, $c(1/2\ I_2) = 0{,}01$ mol/l, until a blue coloration just appears. Then add 100 g (84 ml) of the sample and titrate with the same iodine solution until a blue coloration reappears.

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

R 13.3.6 Ammonium

Neutralize 6,6 g (5,6 ml) of the sample, diluted with 50 ml of water, with sodium hydroxide solution (32 %), using indicator paper, and apply GM 10.

Prepare a standard matching solution, using 2 ml of the ammonium SS II (2 ml ≙ 0,000 3 % NH_4).

R 13.3.7 Arsenic

Dilute 20 g (16,8 ml) of the sample to 100 ml and apply GM 11.

Prepare a standard matching solution, using 1 ml of the arsenic SS III (1 ml ≙ 0,000 005 % As).

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[1] HCl is commercially available also in other concentrations conforming to the above specification.

[2] Chlorine-free hydrochloric acid remains colourless when tested according to this procedure.

R 13 Hydrochloric acid

R 13.3.8 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	50 ml of the test solution (R 13.2) diluted to 100 ml	Air-acetylene	324,7
Pb			217,0 or 283,3

R 13.3.9 Iron

Take 5 ml of the test solution (R 13.2), add 5 ml of water and apply GM 8.1.

Prepare a standard matching solution, using 1 ml of the iron SS II (1 ml ≙ 0,000 05 % Fe).

R 13.3.10 Sulfated ash

Take 200 g (168 ml) of the sample and apply GM 17.

The mass of the residue shall not exceed 1 mg.

R 14 Hydrogen peroxide H_2O_2

Relative molecular mass : 34,01

R 14.1 Specification

Assay (H_2O_2)	30 % min.[1]
Acidity (expressed in millimoles of H^+)	0,3/100 g max.
Chloride (Cl)	0,000 5 % max.
Phosphate (PO_4)	0,000 5 % max.
Sulfate (SO_4)	0,000 5 % max.
Total nitrogen (N)	0,002 % max.
Copper (Cu)	0,000 01 % max.
Iron (Fe)	0,000 02 % max.
Lead (Pb)	0,000 02 % max.
Residue after evaporation	0,005 % max.

R 14.2 Tests

R 14.2.1 Assay

Weigh, to the nearest 0,000 1 g, 0,20 g of the sample, mix with 50 ml of water and 20 ml of sulfuric acid solution (16 %) and 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 0,001 700 7 g of H_2O_2.

R 14.2.2 Acidity

Mix 10 g (9 ml) of the sample with 90 ml of carbon dioxide-free water and titrate with standard volumetric sodium hydroxide solution, $c(NaOH) = 0,01$ mol/l, using the methyl red (IS 4.3.6*).

The volume of titrant shall not exceed 3 ml.

R 14.2.3 Chloride

Take 2 g (1,8 ml) of the sample, add 30 ml of water and apply GM 2.

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

R 14.2.4 Phosphate

Evaporate 10 g (9 ml) of the sample to a low volume in a platinum crucible and apply GM 4.

Prepare a standard matching solution using 5 ml of the phosphate SS II (5 ml ≙ 0,000 5 % PO_4).

R 14.2.5 Sulfate

Take 10 g (9 ml) of the sample, add 15 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).

R 14.2.6 Total nitrogen

Take 2,5 g (2,25 ml) of the sample in a platinum dish, add 10 ml of water and 1 ml of the sulfuric acid (R 37), and evaporate to about 5 ml on a boiling water-bath. Add 120 ml of water and apply GM 6.

Prepare a standard matching solution, using 5 ml of the nitrogen SS II (5 ml ≙ 0,002 % N).

R 14.2.7 Copper and lead

Determine these elements by anodic stripping voltammetry according to GM 33, using the following conditions :

Preparation of the sample :

Take 100 g (90 ml) of the sample in a PTFE (or quartz) dish, add 0,5 ml of hydrochloric acid solution (30 %, high purity) and evaporate to dryness. Dissolve the residue in 1 ml of the same hydrochloric acid, and again evaporate to dryness. Dissolve the residue in 0,5 ml of hydrochloric acid solution (15 %, high purity), and add 25 ml of pyridine solution (1 %) (support electrolyte solution).

Working electrode	Hg
Reference electrode	Ag/AgCl, saturated KCl
Starting potential	− 0,75 V
Peak potentials	Cu : − 0,05 V Pb : − 0,31 V
Evaluation	By calibration graph

R 14.2.8 Iron

Determine this element by single-sweep polarography according to GM 32, using the following conditions :

Preparation of the sample :

Dissolve the residue (prepared in the same manner as described under R 14.2.7) from 100 g (90 ml) of the sample in 5 ml of hydrochloric acid solution, $c(HCl) = 1$ mol/l, and add one

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1) A suitable stabilizer may be used.

R 14 Hydrogen peroxide

pellet of the sodium hydroxide (R 34), 0,5 g of ammonium tartrate and 1 ml of ammonia solution (10 %) (support electrolyte solution).

Reference electrode	Hg
Peak potential	− 1,45 V
Evaluation	By calibration graph

R 14.2.9 Residue after evaporation

Decompose 100 g (90 ml) of the sample in a platinum dish, keeping the liquid cold. Evaporate to dryness and dry to constant mass at 105 ± 2 °C.

The mass of the residue shall not exceed 5 mg.

R 15 Hydroxylammonium chloride
$HONH_3Cl$

Relative molecular mass : 69,49

R 15.1 Specification

Assay ($HONH_3Cl$)	98 % min.
pH (5 % solution) : 2,5 to 4,0	
Sulfate (SO_4)	0,002 % max.
Ammonium (NH_4)	0,1 % max.
Copper (Cu)	0,001 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,001 % max.
Residue after ignition (at 600 °C)	0,01 % max.

R 15.2 Preparation of test solution

Dissolve 20 g of the sample, adjust with the ammonia solution (R 3) to pH 5, and dilute to 200 ml (the solution shall be clear and colourless).

R 15.3 Tests

R 15.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 1 g of the sample and dissolve in about 100 ml of water. Transfer the solution quantitatively to a 200 ml one-mark volumetric flask, dilute to the mark and mix.

Take 20,00 ml of this solution, add a mixture of 20 ml ammonium iron(III) sulfate solution [25 % $NH_4Fe(SO_4).12H_2O$] and 6 ml of sulfuric acid solution (25 %) and heat to boiling. After 5 min, dilute to 250 ml with freshly boiled and cooled water. Heat to 60 °C and, after addition of 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 0,003 475 g of $HONH_3Cl$.

R 15.3.2 pH

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

R 15.3.3 Sulfate

Take 20 ml of the test solution (R 15.2) and apply GM 3.

Prepare a standard matching solution, using 4 ml of the sulfate SS II (4 ml ≙ 0,002 % SO_4).

R 15.3.4 Ammonium

Dissolve 1 g of the sample in 25 ml of ethanol [95 % (*V*/*V*)] and add 1 ml of hexachloroplatinic(IV) acid (10 %).

No change in the solution shall occur within 15 min.

R 15.3.5 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	10 %	Air-acetylene	324,7
Pb			217,0 or 283,3

R 15.3.6 Iron

Take 20 ml of the test solution (R 15.2) and apply GM 8.1.

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

R 15.3.7 Residue after ignition

Moisten 10 g of the sample with the sulfuric acid (R 37), heat gently until no more fumes are evolved, and ignite at 600 °C to constant mass.

The mass of the residue shall not exceed 1 mg.

R 16 Magnesium chloride hexahydrate $MgCl_2.6H_2O$

Relative molecular mass : 203,31

R 16.1 Specification

Assay ($MgCl_2.6H_2O$)	98 % min.
pH (5 % solution) : 5,0 to 6,5	
Phosphate (PO_4)	0,000 5 % max.
Sulfate (SO_4)	0,002 % max.
Total nitrogen (N)	0,002 % max.
Barium (Ba)	0,005 % max.
Calcium (Ca)	0,01 % max.
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,000 5 % max.
Potassium (K)	0,005 % max.
Sodium (Na)	0,005 % max.

R 16.2 Preparation of test solution

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

R 16.3 Tests

R 16.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,80 g of the sample and dissolve in about 100 ml of water.

To this solution add 1 g of the ammonium chloride (R 5), 1 ml of the ammonia solution (R 3), 40 mg of the mordant black 11 mixture (4.3.8*) and titrate with standard volumetric EDTA solution, $c(1/2\ \text{EDTA}) = 0{,}2$ mol/l, to a blue colour.

1,00 ml of EDTA solution, $c(1/2\ \text{EDTA}) = 0{,}200$ mol/l, corresponds to 0,020 33 g of $MgCl_2.6H_2O$.

R 16.3.2 pH

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

R 16.3.3 Phosphate

Take 50 ml of the test solution (R 16.2), add 30 ml of water and apply GM 4.

Prepare a standard matching solution, using 5 ml of the phosphate SS II (5 ml ≙ 0,000 5 % PO_4).

R 16.3.4 Sulfate

Take 20 ml of the test solution (R 16.2) and apply GM 3.

Prepare a standard matching solution, using 8 ml of the sulfate SS II (8 ml ≙ 0,002 % SO_4).

R 16.3.5 Total nitrogen

Dilute 5 ml of the test solution (R 16.2) to 140 ml with water and apply GM 6.

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

R 16.3.6 Calcium, 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
Ca	5 %	Air-acetylene	422,7
Cu	10 % Use the extraction solvent in GM 35		324,7
Fe			248,3
Pb			217,0 or 283,3

R 16.3.7 Barium, potassium and sodium

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

Element	Concentration of solution	Flame	Wavelength nm
Ba	1 %	Oxygen-acetylene	553,6
K			766,5
Na	0,5 %		589,0

R 17 Magnesium oxide
MgO

Relative molecular mass : 40,30

R 17.1 Specification

Assay (MgO)	96,0 % min.
Substances insoluble in hydrochloric acid	0,01 % max.
Substances soluble in water	0,5 % max.
Carbonate (expressed as CO_2)	1,5 % max.
Chloride (Cl)	0,01 % max.
Sulfate (SO_4)	0,02 % max.
Total nitrogen (N)	0,002 % max.
Arsenic (As)	0,000 1 % max.
Barium and strontium (expressed as Ba)	0,005 % max.
Calcium (Ca)	0,05 % max.
Copper (Cu)	0,001 % max.
Heavy metals (expressed as Pb)	0,003 % max.
Iron (Fe)	0,01 % max.
Potassium (K)	0,005 % max.
Sodium (Na)	0,2 % max.
Zinc (Zn)	0,005 % max.
Loss after ignition (at 800 °C)	4,0 % max.

R 17.2 Preparation of test solution

Take the filtrate from the determination of substances insoluble in hydrochloric acid solution (see R 17.3.2) and adjust to pH 3 with the ammonia solution (R 3). Dilute with water to 200 ml.

R 17.3 Tests

R 17.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 1 g of the sample and dissolve in about 6 ml of hydrochloric acid solution (25 %) and 10 ml of water. Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

Take 25,00 ml of this solution, neutralize with the ammonia solution (R 3), and add 1 ml of the ammonia solution in excess. Add 50 mg of the mordant black 11 mixture (4.3.8*) and titrate with standard volumetric EDTA solution,
c(1/2 EDTA) = 0,2 mol/l, to a blue colour.

1,00 ml of EDTA solution, c(1/2 EDTA) = 0,200 mol/l, corresponds to 0,004 030 g of MgO.

R 17.3.2 Substances insoluble in hydrochloric acid

Dissolve 20 g of the sample in 40 ml of water and 150 ml of hydrochloric acid solution (25 %). Heat to boiling, cool, filter through a sintered-glass filter crucible (porosity No. 40 — pore size diameter 16 to 40 µm), retaining the filtrate for the preparation of the test solution (R 17.2), and dry the residue to constant mass at 105 °C.

The mass of the residue shall not exceed 2 mg.

R 17.3.3 Substances soluble in water

Take 3 g of the sample, add 90 ml of water, heat to boiling, and filter. Evaporate 60 ml of the filtrate to dryness, and dry the residue to constant mass at 600 °C.

The mass of the residue shall not exceed 10 mg.

R 17.3.4 Carbonate

To 1 g of the sample in a distillation apparatus, add about 80 ml of sulfuric acid solution (16 %), and absorb the carbon dioxide formed in an absorption flask containing a mixture (3:1:3 by volume) of 1,4-dioxane-benzylamine-ethanol [95 % (V/V)]. Titrate with standard volumetric tetrabutylammonium hydroxide solution, $c[(C_4H_9)_4NOH] = 0{,}1$ mol/l, using thymol blue solution (0,2 % in 1,4-dioxane) as indicator.

The volume of titrant shall not exceed 3,4 ml.

R 17.3.5 Chloride

Dissolve 0,5 g of the sample in 7 ml of nitric acid solution (25 %), add 20 ml of water, heat to boiling, and apply GM 2.

Prepare a standard matching solution, using 5 ml of the chloride SS II (5 ml ≙ 0,01 % Cl).

R 17.3.6 Sulfate

Dilute 5 ml of the test solution (R 17.2) with 20 ml of water, neutralize with the ammonia solution (R 3) and apply GM 3.

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

R 17.3.7 Total nitrogen

Dissolve 1 g of the sample in 8 ml of hydrochloric acid solution (25 %) and apply GM 6.

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

R 17 Magnesium oxide

R 17.3.8 Arsenic

Take 50 ml of the test solution (R 17.2) and apply GM 11.

Prepare a standard matching solution, using 5 ml of the arsenic SS III (5 ml ≙ 0,000 1 % As).

R 17.3.9 Barium and strontium

Mix 0,25 ml of barium chloride dihydrate solution [0,02 % $BaCl_2.2H_2O$ in 30 % (*V*/*V*) ethanol] and 1 ml of sodium sulfate decahydrate solution (40 % $Na_2SO_4.10H_2O$) (seeding solution). To this mixture add, after exactly 1 min, 10 ml of the test solution (R 17.2), which has been previously acidified with 0,5 ml of hydrochloric acid solution (25 %), and 10 ml of water.

After 10 min, any turbidity shall not be greater than that of a similarly prepared standard matching solution, using 5 ml of the barium SS II (5 ml ≙ 0,005 % Ba).

R 17.3.10 Calcium, copper and zinc

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

Element	Concentration of solution (in 73 g/l hydrochloric acid solution)	Flame	Resonance line nm
Ca	4 %	Air-acetylene	422,7
Cu			324,7
Zn			213,9

R 17.3.11 Heavy metals

Take 10 ml of the test solution (R 17.2), add 0,5 g of the ammonium chloride (R 5), neutralize with the ammonia solution (R 3) and apply GM 7.

Prepare a standard matching solution, using 3 ml of the lead SS II (3 ml ≙ 0,003 % Pb).

R 17.3.12 Iron

Take 5 ml of the test solution (R 17.2) and apply GM 8.1.

Prepare a standard matching solution, using 5 ml of the iron SS II (5 ml ≙ 0,01 % Fe).

R 17.3.13 Potassium and sodium

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

Element	Concentration of solution (in 73 g/l hydrochloric acid solution)	Flame	Wavelength nm
K	4 %	Oxygen-acetylene	766,5
Na	0,1 %		589,0

R 17.3.14 Loss after ignition

Ignite 0,5 g of the sample at 800 °C to constant mass.

The loss in mass shall not exceed 20 mg.

R 18 Methanol
CH_3OH

Relative molecular mass : 32,04

R 18.1 Specification

Assay (CH_3OH)	99,8 % min.
Density : 0,791 to 0,793 g/ml	
Residue after evaporation	0,001 % max.
Acidity (expressed in millimoles of H^+)	0,065/100 g max.
Alkalinity (expressed in millimoles of OH^-)	0,02/100 g max.
Permanganate-reducing substances (expressed as O)	0,000 5 % max.
Readily carbonizable substances	To pass test
Carbonyl compounds (expressed as CO)	0,005 % max.
Water	0,2 % max.

R 18.2 Tests

R 18.2.1 Assay

Apply GM 34, using the following conditions :

Stationary phase	20 % Carbowax 400
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	50 °C
Injection temperature	150 °C
Detection temperature	150 °C
Type of detector	Thermal conductivity
Carrier gas	Helium
Flow rate	30 ml/min
Test portion	5 µl

R 18.2.2 Density

Apply GM 24.1.

R 18.2.3 Residue after evaporation

Take 100 g (126 ml) of the sample and apply GM 14.

The mass of the residue shall not exceed 1 mg.

R 18.2.4 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 5,2 ml.

R 18.2.5 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 1,6 ml.

R 18.2.6 Permanganate-reducing substances

Take 16 g (20 ml) of the sample and apply GM 19.1, adding 0,1 ml of potassium permanganate solution (3,16 g/l) and allowing the test solution to stand at 15 °C for 10 min.

The pink colour shall not be completely discharged.

R 18.2.7 Readily carbonizable substances

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

The resulting solution shall be colourless or its colour intensity shall not exceed that of the sulfuric acid solution or of the sample.

R 18.2.8 Carbonyl compounds

Take 4 g (5 ml) of the sample and dilute to 20 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 18.2.9 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 19 Nitric acid
HNO_3

Relative molecular mass : 63,01

R 19.1 Specification

Assay (HNO_3)	65 % min.[1]
Chloride (Cl)	0,000 05 % max.
Sulfate (SO_4)	0,000 2 % max.
Arsenic (As)	0,000 001 % max.
Copper (Cu)	0,000 01 % max.
Iron (Fe)	0,000 02 % max.
Lead (Pb)	0,000 01 % max.
Sulfated ash	0,001 % max.

R 19.2 Preparation of test solution

Take 300 g (210 ml) of the sample in a 400 ml beaker, add 0,1 g of the anhydrous sodium carbonate (R 30) and evaporate to dryness. Dissolve the residue in 60 ml of water.

R 19.3 Tests

R 19.3.1 Assay

Weigh, to the nearest 0,000 1 g, a thin glass ampoule of suitable capacity, provided with a capillary tube at one end.

Place about 2 ml of the sample in the ampoule, using, for example, the heating technique. Seal the ampoule without loss of glass and weigh to the nearest 0,000 1 g.

Calculate by difference the mass of the test portion. Place the ampoule in a 250 ml conical flask containing 50 ml of water, and close the flask with a ground glass stopper. Shake the flask in order to break the ampoule and continue shaking until all vapour has been absorbed completely. Then open the flask, rinse the stopper and the wall of the flask carefully with water, and grind the broken ampoule thoroughly by means of a glass rod. Titrate the solution with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 1$ mol/l, using the methyl red (IS 4.3.6*).

1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 1{,}000$ mol/l, corresponds to 0,063 01 g of HNO_3.

R 19.3.2 Chloride

To 10 ml of water, add 10 ml of the test solution (R 19.2) and 1 ml of 17 g/l silver nitrate solution.

No turbidity shall be produced within 15 min.

R 19.3.3 Sulfate

Take 10 ml of the test solution (R 19.2) and apply GM 3.

Prepare a standard matching solution, using 10 ml of the sulfate SS II (10 ml ≙ 0,000 2 % SO_4).

R 19.3.4 Arsenic

Take 20 ml of the test solution (R 19.2) and apply GM 11.

Prepare a standard matching solution, using 1 ml of the arsenic SS III (1 ml ≙ 0,000 001 %).

R 19.3.5 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	20 ml of the test solution (R 19.2) diluted to 50 ml	Air-acetylene	324,7
Pb			217,0 or 283,3

R 19.3.6 Iron

Take 10 ml of the test solution (R 19.2) and apply GM 8.1.

Prepare a standard matching solution, using 1 ml of the iron SS II (1 ml ≙ 0,000 02 % Fe).

R 19.3.7 Sulfated ash

Take 100 g (70 ml) of the sample and apply GM 17.

The mass of the residue shall not exceed 1 mg.

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1) Nitric acid is commercially available also in other concentrations conforming to the above specification, which does not apply, however, to the nitric acid containing 95 to 100 % of HNO_3.

R 20 Oxalic acid dihydrate
$C_2H_2O_4.2H_2O$

Relative molecular mass : 126,07

R 20.1 Specification

Assay ($C_2H_2O_4.2H_2O$)	99,5 % min.
Chloride (Cl)	0,000 5 % max.
Sulfate (SO_4)	0,005 % max.
Total nitrogen (N)	0,001 % max.
Calcium (Ca)	0,001 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 2 % max.
Sulfated ash	0,01 % max.

R 20.2 Preparation of test solutions

R 20.2.1 Test solution I

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

R 20.2.2 Test solution II

Take 12 g of the sample and 1 g of anhydrous sodium carbonate. Add 1 drop of ammonium vanadate solution (1 %), and evaporate several times with 20 ml of the nitric acid (R 19) respectively until the substance is completely decomposed and no more oxides of nitrogen are evolved. Evaporate once more with 20 ml of hydrochloric acid solution (25 %) and dissolve the residue in 1 ml of the same hydrochloric acid solution and 24 ml of water.

R 20.2.3 Test solution III

Adopt the procedure specified in R 20.2.2, but using 2 g of the sample.

R 20.3 Tests

R 20.3.1 Assay

Weigh, to the nearest 0,000 1 g, 0,25 g of the sample and dissolve in about 50 ml of water.

Acidify the solution with the sulfuric acid (R 37), and titrate at between 60 and 80 °C 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 0,006 303 3 g of $C_2H_2O_4.2H_2O$.

R 20.3.2 Chloride

Take 20 ml of the test solution I (R 20.2.1) and apply GM 2.

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

R 20.3.3 Sulfate

Take 4 ml of the test solution II (R 20.2.2), add 14 ml of water and apply GM 3, without, however, acidifying the test solution II.

Prepare a standard matching solution, using 8 ml of the sulfate SS II (8 ml ≙ 0,005 % SO_4) and 4 ml of the test solution III (R 20.2.3).

R 20.3.4 Total nitrogen

Dilute 20 ml of the test solution I (R 20.2.1) to 100 ml with water and apply GM 6.

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

R 20.3.5 Calcium

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	Test solution I (R 20.2.1)	Air-acetylene	422,7

R 20.3.6 Heavy metals

Take 10 ml of the test solution II (R 20.2.2), neutralize with ammonia solution (10 %) and apply GM 7.

Prepare a standard matching solution, using 2 ml of the lead SS II (2 ml ≙ 0,000 5 % Pb) and 10 ml of the test solution III (R 20.2.3).

R 20.3.7 Iron

Take 20 ml of the test solution I, (R 20.2.1), 2 ml of 5-sulfosalicylic acid solution (10 %) and 5 ml of the ammonia solution (R 3).

The yellow coloration of the test solution shall not be more intense than that of a similarly prepared standard matching solution, using 0,4 ml of the iron SS II (0,4 ml ≙ 0,000 2 % Fe).

R 20.3.8 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 1 mg.

R 21 Perchloric acid (72 %)
$HClO_4$

Relative molecular mass : 100,46

R 21.1 Specification

Assay ($HClO_4$)	70 to 73 %[1]
Chlorate (ClO_3)	0,001 % max.
Chloride (Cl)	0,000 3 % max.
Free chlorine (Cl)	0,000 5 % max.
Phosphate and silicate (expressed as SiO_2)	0,005 % max.
Sulfate (SO_4)	0,001 % max.
Total nitrogen (N)	0,002 % max.
Arsenic (As)	0,000 005 % max.
Copper (Cu)	0,000 01 % max.
Iron (Fe)	0,000 2 % max.
Lead (Pb)	0,000 01 % max.
Manganese (Mn)	0,000 05 % max.
Silver (Ag)	0,000 5 % max.
Sulfated ash	0,003 % max.

R 21.2 Tests

R 21.2.1 Assay

Weigh, to the nearest 0,000 1 g, about 5 g of the sample and dilute with 50 ml of water. Titrate with standard volumetric sodium hydroxide solution, c(NaOH) = 1 mol/l, using the methyl red (IS 4.3.6*).

1,00 ml of sodium hydroxide solution, c(NaOH) = 1,000 mol/l, corresponds to 0,100 5 g of $HClO_4$.

R 21.2.2 Chlorate

Dilute 20 g (12 ml) of the sample with 33 ml of water and add 2 ml of the nitric acid (R 19), 0,1 g of sodium nitrite and 2,5 ml of 17 g/l silver nitrate solution.

After 5 min, any opalescence shall not be greater than that of a similarly prepared standard matching solution, using 8,5 ml of the chloride SS II (8,5 ml ≙ 0,001 % ClO_3).

R 21.2.3 Chloride

Dilute 10 g (6 ml) of the sample with 45 ml of water, add 1 ml of nitric acid solution (30 %) and apply GM 2.

Prepare a standard matching solution, using 3 ml of the chloride SS II (3 ml ≙ 0,000 3 % Cl).

R 21.2.4 Free chlorine

Dilute 1,8 g (1,1 ml) with 20 ml of freshly boiled and cooled water. Add 1 ml of cadmium iodide solution (10 %) and 1 ml of the starch (IS 4.3.11*) and set aside in the dark for 10 min.

Any blue colour produced shall be discharged by the addition of 0,05 ml of sodium thiosulfate solution, $c(Na_2S_2O_3)$ = 0,005 mol/l.

R 21.2.5 Phosphate and silicate

Neutralize 5 g (3 ml) of the sample with ammonia solution (10 %) and make up to 80 ml with water. Add 5 ml of hexaammonium heptamolybdate solution (10 %) and adjust to pH 1,8 with the hydrochloric acid (R 13). Add 10 ml of the hydrochloric acid (R 13), and extract with 20 ml of 4-methyl-2-pentanone. Wash the organic phase with hydrochloric acid solution (0,5 %) and reduce with 0,2 ml of tin(II) chloride solution [2 % $SnCl_2.2H_2O$ in the hydrochloric acid (R 13)].

The blue coloration of the organic phase shall not be more intense than that of a similarly prepared standard matching solution, using 2,5 ml of the silicate SS I (2,5 ml ≙ 0,005 % SiO_2).

R 21.2.6 Sulfate

Dilute 5 g (3 ml) of the sample with 10 ml of water, neutralize with the ammonia solution (R 3), dilute to 20 ml and apply GM 3.

Prepare a standard matching solution, using 5 ml of the sulfate SS II (5 ml ≙ 0,001 % SO_4).

R 21.2.7 Total nitrogen

Dilute 1 g (0,6 ml) of the sample with 140 ml of water and apply GM 6.

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[1] Perchloric acid is commercially available also in other concentrations conforming to the above specification.

R 21 Perchloric acid (72 %)

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

R 21.2.8 Arsenic

Dilute 40 g (24 ml) of the sample with 50 ml of water and apply GM 11.

Prepare a standard matching solution, using 2 ml of the arsenic SS III (2 ml ≙ 0,000 005 % As).

R 21.2.9 Copper, iron, lead, manganese, and silver

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

Element	Concentration of solution	Flame	Resonance line nm
Cu	Evaporate 500 g (290 ml) of the sample to dryness, dissolve the residue in 0,25 ml of the hydrochloric acid (R 13) and dilute to 50 ml with water	Air-acetylene	324,7
Fe			248,3
Pb			217,0 or 283,3
Mn			279,5
Ag			328,1

R 21.2.10 Sulfated ash

Take 33 g (20 ml) of the sample and apply GM 17.

The mass of the residue shall not exceed 1 mg.

R 22 Phosphoric acid
H_3PO_4

Relative molecular mass : 98,00

R 22.1 Specification

Assay (H_3PO_4)	85 % min.[1]
Chloride (Cl)	0,000 3 % max.
Sulfate (SO_4)	0,005 % max.
Arsenic (As)	0,000 1 % max.
Cadmium (Cd)	0,000 5 % max.
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,001 % max.
Lead (Pb)	0,001 % max.
Manganese (Mn)	0,000 5 % max.
Nickel (Ni)	0,000 5 % max.
Potassium (K)	0,005 % max.
Sodium (Na)	0,05 % max.
Zinc (Zn)	0,001 % max.
Volatile acids (expressed in millimoles of H^+)	0,02/100 g max.

R 22.2 Tests

R 22.2.1 Assay

Weigh, to the nearest 0,000 1 g, about 2,5 g (1,5 ml) of the sample and dilute with 100 ml of water. Add 0,5 ml of the thymolphthalein (IS 4.3.12*) and titrate with standard volumetric sodium hydroxide solution, *c*(NaOH) = 1 mol/l.

1,00 ml of sodium hydroxide solution, *c*(NaOH) = 1,000 mol/l, corresponds to 0,049 00 g of H_3PO_4.

R 22.2.2 Chloride

Dilute 5 g (3 ml) of the sample with 20 ml of water and apply GM 2.

Prepare a standard matching solution, using 1,5 ml of the chloride SS II (1,5 ml ≙ 0,000 3 % Cl).

R 22.2.3 Sulfate

Dilute 2 g (1,2 ml) of the sample with 20 ml of water and apply GM 3.

Prepare a standard matching solution, using 10 ml of the sulfate SS II (10 ml ≙ 0,005 % SO_4).

R 22.2.4 Arsenic

Dilute 5 g (3 ml) of the sample with 30 ml of water and apply GM 11.

Prepare a standard matching solution, using 5 ml of the arsenic SS III (5 ml ≙ 0,000 1 % As).

R 22.2.5 Cadmium, copper, iron, lead, manganese, nickel and zinc

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

Element	Concentration of solution	Flame	Resonance line nm
Cd	20 %	Air-acetylene	228,8
Cu			324,7
Fe			248,3
Pb	40 %		217,0 or 283,3
Mn	20 %		279,5
Ni			232,0 or 352,5
Zn			213,9

R 22.2.6 Potassium and sodium

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

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

R 22.2.7 Volatile acids

Dilute 60 g (35 ml) of the sample with 100 ml of freshly boiled and cooled water and distil off 50 ml. Add to the distillate 2 drops of the phenolphthalein (IS 4.3.9*) and titrate with standard volumetric sodium hydroxide solution,
c(NaOH) = 0,1 mol/l, just to a faint pink colour.

The volume of titrant shall not exceed 0,12 ml.

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[1] Phosphoric acid is commercially available also with a concentration of about 88 % of H_3PO_4, conforming to the above specification.

R 23 Potassium dichromate
$K_2Cr_2O_7$

Relative molecular mass : 294,19

R 23.1 Specification

Assay ($K_2Cr_2O_7$)	99,5 % min.
Chloride (Cl)	0,001 % max.
Sulfate (SO_4)	0,01 % max.
Calcium (Ca)	0,002 % max.
Copper (Cu)	0,001 % max.
Iron (Fe)	0,002 % max.
Lead (Pb)	0,005 % max.
Sodium (Na)	0,02 % max.
Loss after drying (at 100 °C)	0,05 % max.

R 23.2 Preparation of test solution

Dissolve 20 g of the sample in water and dilute to 200 ml.

R 23.3 Tests

R 23.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,2 g of the sample and dissolve in about 25 ml of water.

To this solution add 2 g of the potassium iodide (R 25) and stir until dissolved. Add a mixture of 100 ml of water and 10 ml of the hydrochloric acid (R 13), and allow to stand for 10 min in the dark.

Dilute with 100 ml of water and titrate the liberated iodine with standard volumetric sodium thiosulfate solution, $c(Na_2S_2O_3)$ = 0,1 mol/l, adding the starch (IS 4.3.11*) towards the end of the titration.

Carry out a blank test on the reagents used.

1,00 ml of sodium thiosulfate solution, $c(Na_2S_2O_3)$ = 0,100 mol/l, corresponds to 0,004 903 g of $K_2Cr_2O_7$.

R 23.3.2 Chloride

Take 10 ml of the test solution (R 23.2), add 20 ml of water and apply GM 2.

Prepare a standard matching solution, using 5 ml of the test solution (R 23.2) and 0,5 ml of the chloride SS II (0,5 ml ≙ 0,001 % Cl).

R 23.3.3 Sulfate

To 10 ml of the test solution (R 23.2), add 10 ml of hydrochloric acid solution (25 %) and 20 ml of tributyl phosphate, shake vigorously for 1 min, allow to separate and reject the organic phase. To the aqueous phase, add again 20 ml of tributyl phosphate and repeat the extraction. Wash the aqueous phase twice with 5 ml of diethyl ether and evaporate it to dryness. Dissolve the residue in 10 ml of water and apply GM 3.

Prepare a standard matching solution, using 1 ml of the sulfate SS I (1 ml ≙ 0,01 % SO_4).

R 23.3.4 Calcium, 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	10 %	Air-acetylene	324,7
Pb			217,0 or 283,3
Ca	4 %		422,7

R 23.3.5 Iron

To 10 ml of the test solution (R 23.2), add 10 ml of the hydrochloric acid (R 13) and 10 ml of the ethanol (R 11), allow to stand until the reduction is complete (green colour), add 1 ml of the hydrogen peroxide (R 14), and evaporate to a volume of 10 ml. Cool, transfer to a separating funnel, add 10 ml of 4-methyl-pentan-2-one, stir vigorously for 1 min, allow to separate and reject the green aqueous phase.

Wash the organic phase with 10 ml of a mixture of equal volumes of the hydrochloric acid (R 13) and of water, again allow to separate and reject the aqueous phase. To the organic phase, add 10 ml of water, stir vigorously for 1 min, allow to separate and apply GM 8.1 to the aqueous phase.

Prepare a standard matching solution in the same way, using 2 ml of the iron SS II (2 ml ≙ 0,002 % Fe).

R 23.3.6 Sodium

Determine this element by FES according to GM 30, using the following conditions :

Element	Concentration of solution	Flame	Wavelength nm
Na	2 %	Oxygen-acetylene	589,0

R 23.3.7 Loss after drying

Dry 2 g of the sample to constant mass at 100 to 105 °C.

The loss in mass shall not exceed 1 mg.

R 24 Potassium hydroxide
KOH

Relative molecular mass : 56,108

R 24.1 Specification

Assay (KOH)	85 % min.
Carbonate (expressed as K_2CO_3)	1,5 % max.
Chloride (Cl)	0,005 % max.
Phosphate (PO_4)	0,001 % max.
Silicate (expressed as SiO_2)	0,01 % max.
Sulfate (SO_4)	0,005 % max.
Total nitrogen (N)	0,001 % max.
Aluminium (Al)	0,002 % max.
Calcium (Ca)	0,002 % max.
Iron (Fe)	0,001 % max.
Lead (Pb)	0,001 % max.
Magnesium (Mg)	0,000 5 % max.
Nickel (Ni)	0,001 % max.
Sodium (Na)	0,5 % max.
Zinc (Zn)	0,001 % max.

R 24.2 Preparation of test solutions

R 24.2.1 Test solution I

Dissolve 50 g of the sample in about 200 ml of water in a polyethylene beaker and dilute to 250 ml. Store this solution in a polyethylene bottle.

R 24.2.2 Test solution II

Neutralize 100,0 ml of the test solution I (R 24.2.1) in a polyethylene beaker with the hydrochloric acid (R 13), keeping the mixture cold, and dilute to 200 ml.

R 24.3 Tests

R 24.3.1 Assay

Weigh to the nearest 0,000 1 g, about 2,5 g of the sample and dissolve in 50 ml of carbon dioxide-free water.

To this solution add 2,0 ml of barium chloride solution (10 % $BaCl_2.2H_2O$) and 2 drops of the phenolphthalein (IS 4.3.9*), and titrate with standard volumetric hydrochloric acid solution, $c(HCl) = 1$ mol/l, until the solution just becomes colourless.

1,00 ml of hydrochloric acid solution, $c(HCl) = 1,000$ mol/l, corresponds to 0,056 1 g of KOH.

Use the neutralized solution for test R 24.3.2.

R 24.3.2 Carbonate

Add 1 ml of the bromophenol blue (IS 4.3.2*) to the neutralized solution retained in test R 24.3.1, and titrate this solution with standard volumetric hydrochloric acid solution, $c(HCl) = 0,1$ mol/l, to a colour change from blue to yellow.

1,00 ml of hydrochloric acid solution, $c(HCl) = 0,100$ mol/l, corresponds to 0,006 91 g of K_2CO_3.

R 24.3.3 Chloride

Take 10 ml of the test solution I (R 24.2.1), neutralized with nitric acid solution (10 %) and apply GM 2.

Prepare a standard matching solution, using 10 ml of the chloride SS II (10 ml ≙ 0,005 % Cl).

R 24.3.4 Phosphate

Take 50 ml of the test solution II (R 24.2.2) and apply GM 4.

Prepare a standard matching solution, using 5 ml of the phosphate SS II (5 ml ≙ 0,001 % PO_4).

R 24.3.5 Silicate

Take 10 ml of the test solution II (R 24.2.2) and apply GM 5.

Prepare a standard matching solution, using 10 ml of the silicate SS II (10 ml ≙ 0,01 % SiO_2).

R 24.3.6 Sulfate

Take 25 ml of the test solution II (R 24.2.2) and apply GM 3.

Prepare a standard matching solution, using 12,5 ml of the sulfate SS II (12,5 ml ≙ 0,005 % SO_4).

R 24.3.7 Total nitrogen

Take 25 ml of the test solution I (R 24.2.1) and apply GM 6.

Prepare a standard matching solution, using 5 ml of the nitrogen SS II (5 ml ≙ 0,001 % N).

R 24 Potassium hydroxide

R 24.3.8 Aluminium

Take 10 ml of the test solution II (R 24.2.2) and apply GM 9.

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

R 24.3.9 Iron

Take 20 ml of the test solution II (R 24.2.2) and apply GM 8.1.

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

R 24.3.10 Calcium, lead, magnesium, nickel and zinc

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	10 %	Air-acetylene	422,7
Mg			285,2
Ni			232,0 or 352,5
Pb	4 % Use the extraction solvent in GM 35		217,0 or 283,3
Zn	10 %		213,9

R 24.3.11 Sodium

Determine this element by FES according to GM 30, using the following conditions :

Element	Concentration of solution	Flame	Wavelength nm
Na	1 %	Oxygen-acetylene	589,0

R 25 Potassium iodide
KI

Relative molecular mass : 166,01

R 25.1 Specification

Assay (KI)	99,5 % min.
pH (5 % solution) : 6 to 8	
Chloride and bromide (expressed as Cl)	0,01 % max.
Iodate and iodine (expressed as IO_3)	0,000 3 % max.
Phosphate (PO_4)	0,001 % max.
Sulfate (SO_4)	0,005 % max.
Total nitrogen (N)	0,001 % max.
Arsenic (As)	0,000 01 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Calcium (Ca)	0,001 % max.
Magnesium (Mg)	0,001 % max.
Sodium (Na)	0,05 % max.
Reducing substances	To pass the test

R 25.2 Preparation of test solution

Dissolve 40 g of the sample in about 150 ml of water and dilute to 200 ml.

Protect this solution from light and use only if colourless.

R 25.3 Tests

R 25.3.1 Assay

Weigh, to the nearest 0,000 1 g, 0,48 to 0,52 g of the sample and dissolve in 25 ml of water.

To this solution add 35,0 ml of standard volumetric silver nitrate solution, $c(AgNO_3) = 0,1$ mol/l, and then 5 ml of the nitric acid (R 19). Titrate with standard volumetric potassium thiocyanate solution, $c(KSCN) = 0,1$ mol/l, in presence of 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 0,016 6 g of KI.

R 25.3.2 pH

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

R 25.3.3 Chloride and bromide

To 2,5 ml of the test solution (R 25.2), add 10 ml of water, 2,5 ml of the ammonia solution (R 3) and then 35 ml of standard volumetric silver nitrate solution, $c(AgNO_3) = 0,1$ mol/l. Stir and filter. To 10 ml of the filtrate, add 2,5 ml of nitric acid solution (25 %).

At the same time, prepare a standard matching solution, using 1 ml of the chloride SS II (1 ml ≙ 0,01 % Cl). After 2 min, any opalescence in the test solution shall not be greater than that of the similarly prepared standard matching solution .

R 25.3.4 Iodate and iodine

Dissolve 2 g of the sample in 50 ml of water, and add 3 ml of standard volumetric sulfuric acid solution, $c(1/2\ H_2SO_4) = 0,1$ mol/l, and 5 ml of the starch (IS 4.3.11*).

No blue or violet coloration shall be observed within 10 s.

R 25.3.5 Phosphate

Take 10 ml of the test solution (R 25.2), and apply GM 4.

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

R 25.3.6 Sulfate

Take 10 ml of the test solution (R 25.2) and apply GM 3.

Prepare a standard matching solution, using 10 ml of the sulfate SS II (10 ml ≙ 0,005 % SO_4).

R 25.3.7 Total nitrogen

Take 25 ml of the test solution (R 25.2) and apply GM 6.

Prepare a standard matching solution, using 5 ml of the nitrogen SS II (5 ml ≙ 0,001 % N).

R 25.3.8 Arsenic

Take 50 ml of the test solution (R 25.2) and apply GM 11.

Prepare a standard matching solution, using 1 ml of the arsenic SS III (1 ml ≙ 0,000 01 % As).

R 25.3.9 Heavy metals

Take 20 ml of the test solution (R 25.2) and apply GM 7.

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

R 25 Potassium iodide

R 25.3.10 Iron

Take 10 ml of the test solution (R 25.2) and apply GM 8.1.

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

R 25.3.11 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	10 %	Air-acetylene	422,7
Mg	5 %		285,2

R 25.3.12 Sodium

Determine this element by FES according to GM 30, using the following conditions :

Element	Concentration of solution	Flame	Wavelength nm
Na	1 %	Oxygen-acetylene	589,0

R 25.3.13 Reducing substances

Dissolve 1,5 g of the sample in 10 ml of carbon dioxide-free water, and add successively 1 ml of sulfuric acid solution (20 %), 5 ml of the starch (IS 4.3.11*) and 0,05 ml of standard volumetric iodine solution, $c(1/2\ I_2) = 0{,}002$ mol/l.

After 30 s, the coloration shall not be completely discharged.

R 26 Potassium permanganate
$KMnO_4$

Relative molecular mass : 158,04

R 26.1 Specification

Assay ($KMnO_4$)	99 % min.
Water-insoluble matter	0,1 % max.
Chloride (Cl)	0,005 % max.
Sulfate (SO_4)	0,005 % max.
Total nitrogen (N)	0,005 % max.
Heavy metals (expressed as Pb)	0,003 % max.
Iron (Fe)	0,002 % max.

R 26.2 Preparation of test solution

Dissolve 5 g of the sample in 50 ml of water, add 30 ml of the hydrochloric acid (R 13) and evaporate to dryness on a boiling water-bath. Dissolve the residue in 5 ml of the hydrochloric acid (R 13) and again evaporate to dryness.

Dissolve the residue in about 30 ml of water and dilute to 50 ml.

R 26.3 Tests

R 26.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 1 g of the sample, place it in a 500 ml one-mark volumetric flask and dissolve, in about 200 ml of water. Dilute to the mark with water, and mix.

Take 50,0 ml of this solution, add 15 ml of potassium iodide solution (20 %) and 15 ml of sulfuric acid solution (20 %), and titrate the liberated iodine with 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 0,003 16 g of $KMnO_4$.

R 26.3.2 Water-insoluble matter

Take 2 g of the sample and apply GM 1.

The mass of the residue shall not exceed 2 mg.

R 26.3.3 Chloride

Dissolve 2 g of the sample in 35 ml of water, and add 5 ml of the nitric acid (R 19) and, drop by drop, 3 ml of the hydrogen peroxide (R 14). When the reduction is complete, dilute to exactly 100 ml. Take 10 ml of the reduced solution and apply GM 2.

Prepare a standard matching solution, using 1 ml of the chloride SS II (1 ml ≙ 0,005 % Cl).

R 26.3.4 Sulfate

Take 10 ml of the test solution (R 26.2) and apply GM 3.

Prepare a standard matching solution, using 5 ml of the sulfate SS II (5 ml ≙ 0,005 % SO_4).

R 26.3.5 Total nitrogen

Dissolve 1 g of the sample in 100 ml of water and apply GM 6.

Prepare a standard matching solution, using 5 ml of the nitrogen SS II (5 ml ≙ 0,005 % N).

R 26.3.6 Heavy metals

Take 5 ml of the test solution (R 26.2), add 5 ml of water and apply GM 7.

Prepare a standard matching solution, using 1,5 ml of the lead SS II (1,5 ml ≙ 0,003 % Pb).

R 26.3.7 Iron

Take 5 ml of the test solution (R 26.2), add 5 ml of water and apply GM 8.1.

Prepare a standard matching solution, using 1 ml of the iron SS II (1 ml ≙ 0,002 % Fe).

R 27 Potassium sodium tartrate tetrahydrate $C_4H_4O_6KNa.4H_2O$

Relative molecular mass : 282,23

R 27.1 Specification

Assay ($C_4H_4O_6KNa.4H_2O$)	99 % min.
pH (5 % solution) : 6,5 to 8,5	
Water-insoluble matter	0,005 % max.
Chloride (Cl)	0,001 % max.
Phosphate (PO_4)	0,002 % max.
Sulfate (SO_4)	0,005 % max.
Ammonium (NH_4)	0,002 % max.
Calcium (Ca)	0,005 % max.
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,000 5 % max.

R 27.2 Preparation of test solution

Dissolve 20 g of the sample in water and dilute to 200 ml.

R 27.3 Tests

R 27.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,5 g of the sample and dissolve, in 5 ml of formic acid (99 %).

To this solution add 50 ml of the acetic acid (R 1) and 15 ml of acetic anhydride. Titrate with standard volumetric perchloric acid solution, $c(HClO_4) = 0,1$ mol/l in anhydrous acetic acid, using the crystal violet (IS 4.3.3*).

1,00 ml of perchloric acid solution, $c(HClO_4) = 0,100$ mol/l, corresponds to 0,014 11 g of $C_4H_4O_6KNa.4H_2O$.

R 27.3.2 pH

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

R 27.3.3 Water-insoluble matter

Take 20 g of the sample and apply GM 1.

The mass of the residue shall not exceed 1 mg.

R 27.3.4 Chloride

Dilute 10 ml of the test solution (R 27.2) to 20 ml and apply GM 2.

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

R 27.3.5 Phosphate

Ignite 1 g of the sample in a platinum dish. Dissolve the residue in 5 ml of water and 5 ml of the nitric acid (R 19), and evaporate to dryness. Dissolve the residue in 20 ml of water and apply GM 4.

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

R 27.3.6 Sulfate

Take 10 ml of the test solution (R 27.2), acidify, drop by drop, with hydrochloric acid solution (25 %) and apply GM 3. Allow to stand for 1 h before observing the result.

Prepare a standard matching solution, using 5 ml of the sulfate SS II (5 ml ≙ 0,005 % SO_4).

R 27.3.7 Ammonium

Take 10 ml of the test solution (R 27.2) and apply GM 10.

Prepare a standard matching solution, using 2 ml of the ammonium SS II (2 ml ≙ 0,002 % NH_4).

R 27.3.8 Calcium, copper and lead

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	10 %	Air-acetylene	422,7
Cu	10 % Use the extraction solvent in GM 35		324,7
Pb			217,0 or 283,3

R 27.3.9 Iron

Take 20 ml of the test solution (R 27.2), acidify, drop by drop, with hydrochloric acid solution (20 %) to dissolve any precipitate and apply GM 8.1.

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

R 28 Silver nitrate
$AgNO_3$

Relative molecular mass : 169,87

R 28.1 Specification

Assay ($AgNO_3$)	99,8 % min.
Chloride (Cl)	0,001 % max.
Sulfate (SO_4)	0,002 % max.
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,001 % max.
Substances not precipitated by hydrochloric acid (expressed as sulfates)	0,01 % max.

R 28.2 Tests

R 28.2.1 Assay

Weigh, to the nearest 0,000 1 g, 0,855 g of the sample and dissolve in 50 ml of water.

To this solution add 50,00 ml of standard volumetric sodium chloride solution, $c(NaCl) = 0,1$ mol/l, 0,1 ml of sulfuric acid solution (50 %), 0,25 ml of saturated potassium sulfate solution and 20 ml of ethanol [95 % (V/V)]. Titrate potentiometrically with the standard volumetric sodium chloride solution, $c(NaCl) = 0,1$ mol/l, using Ag-Hg/Hg_2SO_4 electrodes, adding the titrant in increments of 0,1 ml from a microburette.

1,00 ml of sodium chloride solution, $c(NaCl) = 0,100$ mol/l, corresponds to 0,016 99 g of $AgNO_3$.

R 28.2.2 Chloride

Dissolve 2 g of the sample in 25 ml of water, add 1 ml of the nitric acid (R 19), allow to stand in the dark for 10 min and filter through a chloride-free filter (solution A).

Dissolve another 2 g of the sample in 25 ml of water, add 1 ml of the nitric acid (R 19) and allow to stand in the dark for 10 min. The opalescence of this solution shall not be greater than that of a standard matching solution prepared by adding 2 ml of the chloride SS II to the solution A
(2 ml ≙ 0,001 % Cl).

R 28.2.3 Sulfate

Mix 0,25 ml of potassium sulfate ethanolic solution [0,02 % in ethanol (30 % V/V)] with 1 ml of a saturated barium nitrate solution (seeding solution). To this mixture add, after 1 min, a solution of 1 g of the sample in 5 ml of water acidified with 0,5 ml of acetic acid (30 %).

After 5 min, any turbidity in the test solution shall not be greater than that of a similarly prepared standard matching solution, using 2 ml of the sulfate SS II
(2 ml ≙ 0,002 % SO_4).

R 28.2.4 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	Dissolve 10 g of the sample in 40 ml of water, add 10 g of ascorbic acid, stir well and allow to stand for 5 min. Filter, wash the filter with water, and dilute the combined filtrate and washings to 50 ml.	Air-acetylene	324,7
Fe			248,3
Pb			217,0 or 283,3

R 28.2.5 Substances not precipitated by hydrochloric acid

Dissolve 10 g of the sample in 300 ml of water, heat to boiling and add 12 ml of hydrochloric acid solution (20 %). Allow to stand in the dark for 12 h, filter and evaporate the filtrate to dryness. Dissolve the residue in 0,2 ml of hydrochloric acid solution (20 %) and 10 ml of water, boil and filter.

Add a few drops of the sulfuric acid (R 37) to the filtrate and evaporate in a tared dish or crucible. Continue heating at a low temperature until all fumes have disappeared, then heat to 650 ± 50 °C for 15 min, allow to cool in a desiccator, and weigh.

The mass of the residue shall not exceed 1 mg.

R 29 Sodium acetate trihydrate $CH_3COONa.3H_2O$

Relative molecular mass : 136,08

R 29.1 Specification

Assay ($CH_3COONa.3H_2O$)	99 % min.
pH (5 % solution) : 7,5 to 9	
Chloride (Cl)	0,000 5 % max.
Phosphate (PO_4)	0,000 5 % max.
Sulfate (SO_4)	0,002 % max.
Aluminium (Al)	0,000 5 % max.
Calcium (Ca)	0,002 % max
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,000 5 % max.
Potassium (K)	0,005 % max.
Permanganate-reducing substances (expressed as HCOOH)	0,005 % max.

R 29.2 Preparation of test solution

Dissolve 20 g of the sample in 100 ml of water and dilute to 200 ml.

R 29.3 Tests

R 29.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,4 g of the sample and dissolve in 25 ml of the acetic acid (R 1).

To this solution add 0,4 ml of 1-naphtholbenzene solution [0,1 % in acetic acid (R 1)] and titrate with standard volumetric perchloric acid solution, $c(HClO_4) = 0,1$ mol/l, in acetic acid (R 1), to a green colour.

1,00 ml of perchloric acid solution, $c(HClO_4) = 0,100$ mol/l, corresponds to 0,013 61 g of $CH_3COONa.3H_2O$.

R 29.3.2 pH

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

R 29.3.3 Chloride

Take 20 ml of the test solution (R 29.2) and apply GM 2, using 1 ml of the nitric acid (R 19) instead of nitric acid (25 %).

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

R 29.3.4 Phosphate

Take 30 ml of the test solution (R 29.2), add 50 ml of water, and apply GM 4.

Prepare a standard matching solution, using 1,5 ml of the phosphate SS II (1,5 ml ≙ 0,000 5 % PO_4).

R 29.3.5 Sulfate

Take 20 ml of the test solution (R 29.2) and apply GM 3.

Prepare a standard matching solution, using 4 ml of the sulfate SS II (4 ml ≙ 0,002 % SO_4).

R 29.3.6 Aluminium

Take 15 ml of the test solution (R 29.2) and apply GM 9.

Prepare a standard matching solution, using 5 ml of the test solution (R 29.2) and 0,5 ml of the aluminium SS II (0,5 ml ≙ 0,000 5 % Al).

R 29.3.7 Calcium

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

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

R 29.3.8 Copper and lead

Determine these elements by anodic stripping voltammetry according to GM 33, using a solution of 1 g of the sample in 25 ml of standard volumetric sulfuric acid solution, $c(1/2\ H_2SO_4) = 1$ mol/l.

Working electrode	Hg
Reference electrode	Ag/AgCl, saturated KCl
Starting potential	− 0,75 V
Peak potentials	Cu : + 0,185 V Pb : − 0,31 V
Evaluation	By standard solution additions

R 29 Sodium acetate trihydrate

R 29.3.9 Iron

Take 25 ml of the test solution (R 29.2), neutralized with hydrochloric acid solution (25 %), and apply GM 8.1.

Prepare a standard matching solution, using 5 ml of the test solution (R 29.2) and 1 ml of the iron SS II (1 ml ≙ 0,000 5 % Fe).

R 29.3.10 Potassium

Determine this element by FES according to GM 30, using the following conditions :

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

R 29.3.11 Permanganate-reducing substances

Dissolve 10 g of the sample in 100 ml of water, add 1 ml of standard volumetric potassium permanganate solution, $c(1/5\ KMnO_4) = 0{,}1$ mol/l, boil for 5 min, and cool.

The pink colour of the solution shall not disappear completely.

Add 2 g of the potassium iodide (R 25) and 20 ml of sulfuric acid solution (20 %), and titrate with standard volumetric sodium thiosulfate solution, $c(Na_2S_2O_3) = 0{,}1$ mol/l, until colourless.

At the same time carry out a blank test, using the same quantities of all the reagents (except the standard volumetric sodium thiosulfate solution), but omitting the test portion.

The volume of titrant required shall not exceed 0,25 ml, after allowing for the blank test.

R 30 Sodium carbonate, anhydrous Na_2CO_3

Relative molecular mass : 105,99

R 30.1 Specification

Assay (Na_2CO_3, after drying at 300 °C) ...	99,8 % min.
Chloride (Cl)	0,002 % max.
Phosphate and silicate (expressed as SiO_2)	0,005 % max.
Total nitrogen (N)	0,001 % max.
Total sulfur (expressed as SO_4)	0,005 % max.
Aluminium (Al)	0,001 % max.
Calcium (Ca)	0,005 % max
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Magnesium (Mg)	0,002 % max.
Potassium (K)	0,01 % max.
Loss after drying (at 300 °C)	1,0 % max.

R 30.2 Preparation of test solution

Dissolve 20 g of the sample in 80 ml of water, neutralize with hydrochloric acid solution (25 %), and add 5 ml of acid in excess. Boil the solution, cool, and dilute to 200 ml.

R 30.3 Tests

R 30.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 2 g of the sample, previously dried at 300 °C, and dissolve in 50 ml of water.

To this solution add 2 drops of the methyl orange (IS 4.3.5*) and titrate with standard volumetric hydrochloric acid solution, $c(HCl) = 1$ mol/l.

1,00 ml of hydrochloric acid solution, $c(HCl) = 1{,}000$ mol/l, corresponds to 0,052 995 g of Na_2CO_3.

R 30.3.2 Chloride

Dissolve 1 g of the sample in 15 ml of water and apply GM 2.

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

R 30.3.3 Phosphate and silicate

Dissolve 1 g of the sample in a mixture of 80 ml of water and 3 ml of hydrochloric acid solution (25 %). Heat to boiling and, after cooling, add 5 ml of hexaammonium heptamolybdate solution (10 %). Adjust to pH 1,8 with the hydrochloric acid (R 13) and heat the solution to boiling. After cooling, add 10 ml of the hydrochloric acid (R 13) and extract with 50 ml of 4-methyl-2-pentanone. Wash the organic phase with hydrochloric acid solution (0,5 %) and reduce with 0,2 ml of tin(II) chloride solution [2 % $SnCl_2.2H_2O$ in the hydrochloric acid (R 13)]. Dry the organic phase with anhydrous sodium sulfate.

The blue coloration of the organic phase shall not be more intense than that of a similarly prepared standard matching solution, using 5 ml of the silicate SS II (5 ml ≙ 0,005 % SiO_2).

R 30.3.4 Total nitrogen

Dissolve 2 g of the sample in 100 ml of water and apply GM 6.

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

R 30.3.5 Total sulfur

Dissolve 1 g of the sample in 10 ml of water, add 0,25 ml of saturated bromine water, boil, cool, neutralize with hydrochloric acid solution (25 %), and add 0,5 ml in excess. Boil to decolorize, cool, dilute to 20 ml, and apply GM 3.

Prepare a standard matching solution, using 5 ml of the sulfate SS II (5 ml ≙ 0,005 % SO_4).

R 30.3.6 Aluminium

Take 10 ml of the test solution (R 30.2), neutralized with ammonia solution (10 %), add 15 ml of the methanol (R 18) and apply GM 9.

Prepare a standard matching solution, using 1 ml of the aluminium SS II (1 ml ≙ 0,001 % Al).

R 30.3.7 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	Test solution (R 30.2)	Air-acetylene	422,7
Mg			285,2

R 30.3.8 Heavy metals

Take 20 ml of the test solution (R 30.2) and apply GM 7.

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

R 30 Sodium carbonate, anhydrous

R 30.3.9 Iron

Take 20 ml of the test solution (R 30.2) and apply GM 8.1.

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

R 30.3.10 Potassium

Determine this element by FES according to GM 30, using the following conditions :

Element	Concentration of solution	Flame	Wavelength nm
K	Test solution (R 30.2)	Oxygen-acetylene	766,5

R 30.3.11 *Loss after drying*

Dry 2 g of the sample to constant mass at 300 °C.

The loss in mass shall not exceed 20 mg.

R 31 Sodium carbonate decahydrate $Na_2CO_3.10H_2O$

Relative molecular mass : 286,14

R 31.1 Specification

Assay ($Na_2CO_3.10H_2O$)	99 to 102 %
Chloride (Cl)	0,001 % max.
Phosphate and silicate (expressed as SiO_2)	0,003 % max.
Total nitrogen (N)	0,000 5 % max.
Total sulfur (expressed as SO_4)	0,003 % max.
Aluminium (Al)	0,000 5 % max.
Calcium (Ca)	0,003 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 3 % max.
Magnesium (Mg)	0,000 3 % max.
Potassium (K)	0,005 % max.

R 31.2 Preparation of test solution

Dissolve 20 g of the sample in 60 ml of water, neutralize with hydrochloric acid solution (25 %) and add 2,5 ml of this acid in excess. Boil, cool and dilute to 100 ml.

R 31.3 Tests

R 31.3.1 Assay

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

To this solution add 2 drops of the methyl orange (IS 4.3.5*), and titrate with standard volumetric hydrochloric acid solution, $c(HCl) = 1$ mol/l.

1,00 ml of hydrochloric acid solution, $c(HCl) = 1{,}000$ mol/l, corresponds to 0,143 1 g of $Na_2CO_3.10H_2O$.

R 31.3.2 Chloride

Dissolve 2 g of the sample in 15 ml of water and apply GM 2.

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

R 31.3.3 Phosphate and silicate

Dissolve 2 g of the sample in a mixture of 80 ml of water and 3 ml of hydrochloric acid solution (25 %). Heat to boiling and after cooling add 5 ml of hexaammonium heptamolybdate solution (10 %). Adjust to pH 1,8 with the hydrochloric acid (R 13) and heat the solution to boiling. After cooling add 10 ml of the hydrochloric acid (R 13) and extract with 50 ml of 4-methyl-2-pentanone. Wash the organic phase with hydrochloric acid solution (0,5 %) and reduce with 0,2 ml of tin(II) chloride solution [2 % $SnCl_2.2H_2O$ in the hydrochloric acid (R 13)]. Dry the organic phase with anhydrous sodium sulfate.

The blue coloration of the organic phase shall not be more intense than that of a similarly prepared standard matching solution, using 6 ml of the silicate SS II (6 ml ≙ 0,003 % SiO_2).

R 31.3.4 Total nitrogen

Dissolve 4 g of the sample in 100 ml of water and apply GM 6.

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

R 31.3.5 Total sulfur

Dissolve 2 g of the sample in 10 ml of water, add 0,25 ml of saturated bromine water, boil, cool, neutralize with hydrochloric acid solution (25 %), and add 0,5 ml of this acid in excess. Boil to decolorize, cool, dilute to 20 ml, and apply GM 3.

Prepare a standard matching solution, using 6 ml of the sulfate SS II (6 ml ≙ 0,003 % SO_4).

R 31.3.6 Aluminium

Take 10 ml of the test solution (R 31.2), neutralized with ammonia solution (10 %), add 15 ml of the methanol (R 18), and apply GM 9.

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

R 31.3.7 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	10 %	Air-acetylene	422,7
Mg			285,2

R 31.3.8 Heavy metals

Take 20 ml of the test solution (R 31.2) and apply GM 7.

R 31 Sodium carbonate decahydrate

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

R 31.3.9 Iron

Take 15 ml of the test solution (R 31.2) and apply GM 8.1.

Prepare a standard matching solution, using 0,9 ml of the iron SS II (0,9 ml ≙ 0,000 3 % Fe).

R 31.3.10 Potassium

Determine this element by FES according to GM 30, using the following conditions :

Element	Concentration of solution	Flame	Wavelength nm
K	2 %	Oxygen-acetylene	766,5

R 32 Sodium chloride
NaCl

Relative molecular mass : 58,44

R 32.1 Specification

Assay (NaCl)	99,5 % min.
pH (5 % solution) : 5 to 8	
Bromide (Br)	0,01 % max.
Iodide (I)	0,002 % max.
Hexacyanoferrate(II) [$Fe(CN)_6$]	0,000 1 % max.
Phosphate (PO_4)	0,000 5 % max.
Sulfate (SO_4)	0,002 % max.
Total nitrogen (N)	0,001 % max.
Barium (Ba)	0,001 % max.
Calcium (Ca)	0,002 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 2 % max.
Magnesium (Mg)	0,002 % max.
Potassium (K)	0,01 % max.

R 32.2 Preparation of test solution

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

R 32.3 Tests

R 32.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,2 g of the sample and dissolve in 50 ml of water.

To this solution add 1 ml of the nitric acid (R 19) and 30,0 ml of the standard volumetric silver nitrate solution, $c(AgNO_3) = 0{,}1$ mol/l. Titrate potentiometrically with the standard volumetric silver nitrate solution, $c(AgNO_3) = 0{,}1$ mol/l, using Ag-Ag/Cl electrodes.

1,00 ml of silver nitrate solution, $c(AgNO_3) = 0{,}100$ mol/l, corresponds to 0,005 844 g of NaCl.

R 32.3.2 pH

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

R 32.3.3 Iodide

Dissolve 11 g of the sample in 50 ml of water, in a separating funnel, add 2 ml of the hydrochloric acid (R 13) and 10 ml of iron(III) chloride solution (4,5 %), mix thoroughly, and set aside for 5 min.

Prepare a standard matching solution, using 1 g of the sample, 10 ml of the bromide SS I (10 ml ≙ 0,01 % Br), and 2 ml of the iodide SS I (2 ml ≙ 0,002 % I) in another separating funnel.

To both sample solution and standard matching solution, add 10 ml of carbon tetrachloride and shake for 1 min.

Any pink coloration in the sample lower phase shall not be more intense than that in the standard matching lower phase.

Reserve both solutions for the test in R 32.3.4.

R 32.3.4 Bromide

Reject the lower phases of the solutions obtained in R 32.3.3, and wash the aqueous solutions twice with 10 ml of carbon tetrachloride. Add 10 ml of water, 65 ml of sulfuric acid solution (60 %) and 15 ml of the chromic acid (RS 4.2.4*), mix thoroughly and set aside for 5 min. Add 10 ml of carbon tetrachloride and shake for 1 min.

Any yellowish-blue coloration in the sample lower phase shall not be more intense than that in the standard matching lower phase.

R 32.3.5 Hexacyanoferrate(II)

Dissolve 3,5 g of the sample in 12 ml of water, add 0,2 ml of sulfuric acid solution (20 %) and 0,2 ml of the iron(II)/iron(III) mixture (RS 4.2.9*), mix thoroughly and set aside for 2 min. Add 1 ml of the sodium dihydrogen phosphate (RS 4.2.16*) and set aside for 30 min.

Any blue coloration shall not be more intense than that of a standard matching solution containing 1 g of the sample and 2,5 ml of the hexacyanoferrate(II) SS III [2,5 ml ≙ 0,000 1 % $Fe(CN)_6$].

R 32.3.6 Phosphate

Take 50 ml of the test solution (R 32.2), 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 ≙ 0,000 5 % PO_4).

R 32.3.7 Sulfate

Take 20 ml of the test solution (R 32.2), and apply GM 3.

Prepare a standard matching solution, using 4 ml of the sulfate SS II (4 ml ≙ 0,002 % SO_4).

R 32 Sodium chloride

R 32.3.8 Total nitrogen

Take 20 ml of the test solution (R 32.2), and apply GM 6.

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

R 32.3.9 Heavy metals

Take 40 ml of the test solution (R 32.2) and apply GM 7.

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

R 32.3.10 Iron

Take 20 ml of the test solution (R 32.2) and apply GM 8.1.

Prepare a standard matching solution, using 4 ml of the iron SS III (4 ml ≙ 0,000 2 % Fe).

R 32.3.11 Barium

Mix 0,25 ml of barium chloride dihydrate solution [0,02 % $BaCl_2.2H_2O$ in 30 % (*V*/*V*) ethanol] and 1 ml of sodium sulfate decahydrate solution (40 % $Na_2SO_4.10H_2O$) (seeding solution). To this mixture add, after exactly 1 min, 20 ml of the test solution (R 32.2), which has been previously acidified with 0,5 ml of hydrochloric acid solution (25 %) and set aside for 15 min.

Any turbidity shall not be greater than that of a similarly prepared standard matching solution, using 2 ml of the barium SS II (2 ml ≙ 0,001 % Ba).

R 32.3.12 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 32.3.13 Potassium

Determine this element by FES according to GM 30, using the following conditions :

Element	Concentration of solution	Flame	Wavelength nm
K	10 %	Oxygen-acetylene	766,5

R 33 Disodium hydrogenphosphate dodecahydrate $Na_2HPO_4.12H_2O$

Relative molecular mass : 358,14

R 33.1 Specification

Assay ($Na_2HPO_4.12H_2O$)	99 to 102 %
pH (5 % solution) : 9 to 9,4	
Chloride (Cl)	0,001 % max.
Sulfate (SO_4)	0,005 % max.
Total nitrogen (N)	0,002 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Potassium (K)	0,01 % max.

R 33.2 Preparation of test solution

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

R 33.3 Tests

R 33.3.1 Assay

Weigh, to the nearest 0,001 g, about 10 g of the sample and dissolve in 200 ml of carbon dioxide-free water. Titrate potentiometrically with standard volumetric sulfuric acid solution, $c(1/2\ H_2SO_4) = 1$ mol/l, to pH 4,4, using a glass indicator electrode.

1,00 ml of sulfuric acid solution,
$c(1/2\ H_2SO_4) = 1{,}000$ mol/l, corresponds to 0,358 14 g of $Na_2HPO_4.12H_2O$.

R 33.3.2 pH

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

R 33.3.3 Chloride

Take 10 ml of the test solution (R 33.2) and apply GM 2.

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

R 33.3.4 Sulfate

Take 20 ml of the test solution (R 33.2), neutralized with hydrochloric acid solution (25 %), and apply GM 3.

Prepare a standard matching solution, using 10 ml of the sulfate SS II (10 ml ≙ 0,005 % SO_4).

R 33.3.5 Total nitrogen

Take 10 ml of the test solution (R 33.2) and apply GM 6.

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

R 33.3.6 Heavy metals

Dissolve 6 g of the sample in 15 ml of hydrochloric acid solution (10 %) and dilute to 30 ml.

Take 20 ml of the solution, adjust to pH 4 and apply GM 7.

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

R 33.3.7 Iron

Dilute 40 ml of the test solution (R 33.2) with 20 ml of water, add 3 ml of hydrochloric acid solution (25 %), boil for 5 min, then mix with 2 ml of 5-sulfosalicylic acid solution (20 %) and 5 ml of the ammonia solution (R 3).

The yellow coloration shall not be more intense than that of a similarly prepared standard matching solution, using 2 ml of the iron SS II (2 ml ≙ 0,000 5 % Fe).

R 33.3.8 Potassium

Determine this element by FES according to GM 30, using the following conditions :

Element	Concentration of solution	Flame	Wavelength nm
K	1 %	Oxygen-acetylene	766,5

R 34 Sodium hydroxide
NaOH

Relative molecular mass : 40,00

R 34.1 Specification

Assay (NaOH)	98 % min.
Carbonate (expressed as Na_2CO_3)	1 % max.
Chloride (Cl)	0,005 % max.
Phosphate (PO_4)	0,001 % max.
Silicate (expressed as SiO_2)	0,01 % max.
Sulfate (SO_4)	0,005 % max.
Total nitrogen (N)	0,001 % max.
Aluminium (Al)	0,002 % max.
Arsenic (As)	0,000 1 % max.
Calcium (Ca)	0,002 % max.
Iron (Fe)	0,001 % max.
Lead (Pb)	0,001 % max.
Magnesium (Mg)	0,000 5 % max.
Nickel (Ni)	0,001 % max.
Potassium (K)	0,05 % max.
Zinc (Zn)	0,001 % max.

R 34.2 Preparation of test solutions

R 34.2.1 Test solution I

Dissolve 50 g of the sample in about 200 ml of water in a polyethylene beaker and dilute to 250 ml. Store this solution in a polyethylene bottle.

R 34.2.2 Test solution II

Neutralize 100,0 ml of the test solution I (R 34.2.1) in a polyethylene beaker with the hydrochloric acid (R 13), keeping the mixture cold, and dilute to 200 ml.

R 34.3 Test

R 34.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 1,7 g of the sample and dissolve in 50 ml of carbon dioxide-free water.

To this solution add 2,0 ml of barium chloride dihydrate solution (10 %) and 2 drops of the phenolphthalein (IS 4.3.9*), and titrate with standard volumetric hydrochloric acid solution, $c(HCl) = 1$ mol/l, until the solution just becomes colourless.

1,00 ml of hydrochloric acid solution, $c(HCl) = 1{,}000$ mol/l, corresponds to 0,040 0 g of NaOH.

Use the neutralized solution for test R 34.3.2.

R 34.3.2 Carbonate

Add 1 ml of the bromophenol blue (IS 4.3.2*) to the neutralized solution retained in test R 34.3.1, and titrate this solution with standard volumetric hydrochloric acid solution, $c(HCl) = 0{,}1$ mol/l, to a colour change from blue to yellow.

1,00 ml of hydrochloric acid solution, $c(HCl) = 0{,}100$ mol/l, corresponds to 0,005 3 g of Na_2CO_3.

R 34.3.3 Chloride

Take 10 ml of the test solution I (R 34.2.1), neutralized with nitric acid solution (10 %) and apply GM 2.

Prepare a standard matching solution, using 10 ml of the chloride SS II (10 ml ≙ 0,005 % Cl).

R 34.3.4 Phosphate

Take 50 ml of the test solution II (R 34.2.2) and apply GM 4.

Prepare a standard matching solution, using 5 ml of the phosphate SS II (5 ml ≙ 0,001 % PO_4).

R 34.3.5 Silicate

Take 10 ml of the test solution II (R 34.2.2) and apply GM 5.

Prepare a standard matching solution, using 10 ml of the silicate SS II (10 ml ≙ 0,01 % SiO_2).

R 34.3.6 Sulfate

Take 25 ml of the test solution II (R 34.2.2) and apply GM 3.

Prepare a standard matching solution, using 12,5 ml of the sulfate SS II (12,5 ml ≙ 0,005 % SO_4).

R 34.3.7 Total nitrogen

Take 25 ml of the test solution I (R 34.2.1) and apply GM 6.

Prepare a standard matching solution, using 5 ml of the nitrogen SS II (5 ml ≙ 0,001 % N).

R 34.3.8 Aluminium

Take 10 ml of the test solution II (R 34.2.2), and apply GM 9.

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

R 34 Sodium hydroxide

R 34.3.9 Arsenic

Take 50 ml of the test solution II (R 34.2.2), and apply GM 11.

Prepare a standard matching solution, using 5 ml of the arsenic SS III (5 ml ≙ 0,000 1 % As).

R 34.3.10 Iron

Take 20 ml of the test solution II (R 34.2.2) and apply GM 8.1.

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

R 34.3.11 Calcium, lead, magnesium, nickel and zinc

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	10 %	Air-acetylene	422,7
Mg			285,2
Ni			232,0 or 352,5
Pb	4 % Use the extraction solvent in GM 35		217,0 or 283,3
Zn	10 %		213,9

R 34.3.12 Potassium

Determine this element by FES according to GM 30, using the following conditions :

Element	Concentration of solution	Flame	Wavelength nm
K	1 %	Oxygen-acetylene	766,5

R 35 Sodium sulfate, anhydrous
Na_2SO_4

Relative molecular mass : 142,04

R 35.1 Specification

Assay (Na_2SO_4)	99 % min.
pH (5 % solution) : 5 to 8	
Chloride(Cl)	0,001 % max.
Phosphate (PO_4)	0,002 % max.
Total nitrogen (N)	0,000 5 % max.
Calcium (Ca).........................	0,01 % max.
Heavy metals (expressed as Pb)..........	0,001 % max.
Iron (Fe)	0,000 5 % max.
Potassium (K).......................	0,01 % max.
Loss after ignition (at 600 °C)	0,5 % max.

R 35.2 Preparation of test solution

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

R 35.3 Tests

R 35.3.1 Assay

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

Pass the aqueous solution through an anion-exchange column (strongly alkaline), wash the resin with water until the eluate reacts neutral to litmus, and titrate the combined eluates with standard volumetric hydrochloric acid solution, $c(HCl) = 0,5$ mol/l, using the methyl orange (IS 4.3.5*).

1,00 ml hydrochloric acid solution, $c(HCl) = 0,500$ mol/l, corresponds to 0,035 51 g of Na_2SO_4.

R 35.3.2 pH

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

R 35.3.3 Chloride

Take 20 ml of the test solution (R 35.2) and apply GM 2.

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

R 35.3.4 Phosphate

Take 20 ml of the test solution (R 35.2) and apply GM 4.

Prepare a standard matching solution, using 4 ml of the phosphate SS II (4 ml ≙ 0,002 % PO_4).

R 35.3.5 Total nitrogen

Take 40 ml of the test solution (R 35.2) and apply GM 6.

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

R 35.3.6 Calcium

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

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

R 35.3.7 Heavy metals

Take 20 ml of the test solution (R 35.2), and apply GM 7.

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

R 35.3.8 Iron

Take 20 ml of the test solution (R 35.2) and apply GM 8.1.

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

R 35.3.9 Potassium

Determine this element by FES according to GM 30, using the following conditions :

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

R 35.3.10 Loss after ignition

Ignite 2 g of the sample at 600 °C to constant mass.

The loss in mass shall not exceed 10 mg.

R 36 Sodium thiosulfate pentahydrate
$Na_2S_2O_3.5H_2O$

Relative molecular mass : 248,18

R 36.1 Specification

Assay ($Na_2S_2O_3.5H_2O$)	99,0 % min.
pH (5 % solution) : 5,5 to 7,5	
Chloride (Cl)	0,02 % max.
Sulfate and sulfite (expressed as SO_4)	0,1 % max.
Sulfide (S)	0,000 25 % max.
Total nitrogen (N)	0,005 % max.
Calcium (Ca)	0,003 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,000 5 % max.
Magnesium (Mg)	0,001 % max.

R 36.2 Tests

R 36.2.1 Assay

Weigh, to the nearest 0,000 1 g, about 1 g of the sample and dissolve in 30 ml of water. Titrate with standard volumetric iodine solution, $c(1/2\ I_2) = 0,1$ mol/l, using the starch (IS 4.3.11*).

1,00 ml of iodine solution, $c(1/2\ I_2) = 0,100$ mol/l, corresponds to 0,024 82 g of $Na_2S_2O_3.5H_2O$.

R 36.2.2 pH

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

R 36.2.3 Chloride

Dissolve 0,2 g of the sample in 5 ml of water, and add 1 ml of ammonia solution (10 %) and, drop by drop, 2 ml of the hydrogen peroxide (R 14). Evaporate to dryness at 100 °C, cool, dissolve the residue in 20 ml of water and apply GM 2.

Prepare a standard matching solution, using 4 ml of the chloride SS II (4 ml ≙ 0,02 % Cl).

R 36.2.4 Sulfate and sulfite

Dissolve 0,1 g of the sample in 10 ml of water, add 0,1 ml of standard volumetric iodine solution, $c(1/2\ I_2) = 0,1$ mol/l, until the solution is just faintly yellow, dilute to 20 ml, and apply GM 3.

Prepare a standard matching solution, using 10 ml of the sulfate SS II (10 ml ≙ 0,1 % SO_4).

R 36.2.5 Sulfide

Dissolve 2 g of the sample in 20 ml of water and add 0,3 ml of the basic lead acetate (RS 4.2.10*).

The dark coloration shall not be more intense than that of a similarly prepared standard matching solution, using 0,5 ml of the sulfide SS II (0,5 ml ≙ 0,000 25 % S).

R 36.2.6 Total nitrogen

Dissolve 1,5 g of the sample in 10 ml of water, add 5 ml of sulfuric acid solution (50 %) and boil for 5 min. Cool, filter and wash the filter with water until the volume of the combined filtrate and washings is 30 ml. Take 20 ml of the solution and apply GM 6.

Prepare a standard matching solution, using 5 ml of the nitrogen SS II (5 ml ≙ 0,005 % N).

R 36.2.7 Calcium, iron, lead and magnesium

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	10 %	Air-acetylene	422,7
Fe	4 % Use the extraction solvent in GM 35		248,3
Pb			217,0 or 283,3
Mg	10 %		285,2

R 37 Sulfuric acid
H_2SO_4

Relative molecular mass : 98,08

R 37.1 Specification

Coloration in Hazen units	10 max.
Assay (H_2SO_4)	95 % min.1)
Chloride (Cl)	0,000 1 % max.
Nitrate and nitrite (expressed as NO_3)	0,000 02 % max.
Ammonium (NH_4)	0,000 2 % max.
Arsenic (As)	0,000 005 % max.
Copper (Cu)	0,000 01 % max.
Iron (Fe)	0,000 05 % max.
Lead (Pb)	0,000 01 % max.
Permanganate reducing substances (expressed as SO_2)	0,000 5 % max.
Residue after ignition (at 650 °C)	0,001 % max.

R 37.2 Preparation of test solution

Place 200 g (109 ml) of the sample in a platinum dish, add 0,1 g of the anhydrous sodium carbonate (R 30) and heat the mixture at 650 ± 50 °C in a fume cupboard until all fuming ceases. To the residue, add 1 or 2 drops of the hydrochloric acid (R 13) and dilute with water to 100 ml.

R 37.3 Tests

R 37.3.1 Coloration in Hazen units

Apply GM 36.

R 37.3.2 Assay

Weigh to the nearest 0,000 1 g, a thin glass ampoule of suitable capacity, provided at one end with a capillary tube.

Place in the ampoule about 1 ml of the sample, using for example the heating technique. Seal the ampoule and weigh to the nearest 0,000 1 g.

Calculate by difference the mass of the test portion. Place the ampoule in a 250 ml conical flask containing 100 ml of water, and close the flask with a ground glass stopper. Shake the flask in order to break the ampoule, and leave for 2 to 3 min. Then open the flask, rinse the stopper, and grind thoroughly the broken ampoule by means of a glass rod. Titrate the solution with standard volumetric sodium hydroxide solution, $c(NaOH) = 1$ mol/l, using the methyl red (IS 4.3.6*).

1,00 ml of sodium hydroxide solution, $c(NaOH) = 1,000$ mol/l, corresponds to 0,049 04 g of H_2SO_4.

R 37.3.3 Chloride

To 40 ml of water in a 200 ml beaker, cooled in an ice bath, slowly add 25 g (13,6 ml) of the sample, stirring vigorously, and apply GM 2.

Prepare a standard matching solution, using 2,5 ml of the *chloride* SS II (2,5 ml ≙ 0,000 1 % Cl).

R 37.3.4 Nitrate and nitrite

To 15 ml of water in a 50 ml conical flask, cooled in an ice bath, continuously add 50 g (27 ml) of the sample stirring thoroughly. Then add 0,2 ml of brucine solution [5 % in the *acetic acid* (R 1)] and mix.

Any yellow coloration in 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 ≙ 0,000 02 % NO_3) and 50 ml of nitrate-free sulfuric acid.2)

R 37.3.5 Ammonium

Cautiously add 10 g (5,4 ml) of the sample to 30 ml of water while stirring, cool, neutralize with sodium hydroxide solution (32 %) to indicator paper, and apply GM 10.

Prepare a standard matching solution, using 2 ml of the ammonium SS II (2 ml ≙ 0,000 2 % NH_4).

R 37.3.6 Arsenic

Cautiously add 20 g (10,9 ml) of the sample to 50 ml of water while stirring, dilute to 100 ml, cool, and apply GM 11.

Prepare a standard matching solution, using 1 ml of the arsenic SS III (1 ml ≙ 0,000 005 % As).

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1) Sulfuric acid is commercially available also in other concentrations conforming to the above specification.

2) Nitrate-free sulfuric acid remains colourless when tested according to this procedure.

R 37 Sulfuric acid

R 37.3.7 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 37.2)	Air-acetylene	324,7
Pb			217,0 or 283,3

R 37.3.8 Iron

Take 20 ml of the test solution (R 37.2) and apply GM 8.1.

Prepare a standard matching solution, using 2 ml of the iron SS II (2 ml ≙ 0,000 05 % Fe).

R 37.3.9 Permanganate-reducing substances

To 90 ml of water in a conical flask, cooled in an ice bath, add slowly, in portions of about 5 ml, 64 g (35 ml) of the sample while stirring, keeping the temperature below 35 °C. When the solution has cooled down to 15 to 20 °C, add 0,1 ml of standard volumetric potassium permanganate solution, $c(1/5\ KMnO_4) = 0{,}1$ mol/l and mix.

After 2 min, the pink coloration shall not be discharged completely.

R 37.3.10 Residue after ignition

In a fume cupboard, heat 100 g (54 ml) of the sample in a tared platinum dish until all fuming ceases, ignite at 650 ± 50 °C for 15 min, and weigh.

The mass of the residue shall not exceed 1 mg.

R 38 Tin(II) chloride dihydrate $SnCl_2.2H_2O$

Relative molecular mass : 225,63

R 38.1 Specification

Assay ($SnCl_2.2H_2O$)	97 % min.
Sulfate (SO_4)	0,005 % max.
Arsenic (As)	0,000 2 % max.
Copper (Cu)	0,002 % max.
Iron (Fe)	0,003 % max.
Lead (Pb)	0,005 % max.
Substances not precipitated by hydrogen sulfide (expressed as sulfates)	0,05 % max.

R 38.2 Preparation of test solution

Dissolve 10 g of the sample in water, add 5 ml of the hydrochloric acid (R 13) to clarify the solution, and dilute to 50 ml.

R 38.3 Tests

R 38.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,4 g of the sample and place it in a dry 250 ml conical flask. Immediately add 50 ml of standard volumetric hydrochloric acid solution, $c(\text{HCl}) = 0,2$ mol/l, shake for 4 to 5 s, and then add 5 g of the potassium sodium tartrate tetrahydrate (R 27), followed by 3 g of sodium hydrogen carbonate. Titrate immediately with standard volumetric iodine solution, $c(1/2\ I_2) = 0,1$ mol/l, using the starch (IS 4.3.11*).

1,00 ml of iodine solution, $c(1/2\ I_2) = 0,100$, corresponds to 0,011 28 g of $SnCl_2.2H_2O$.

R 38.3.2 Sulfate

Take 10 ml of the test solution (R 38.2) and apply GM 3.

Prepare a standard matching solution, using 10 ml of the sulfate SS II (10 ml ≙ 0,005 % SO_4).

R 38.3.3 Arsenic

Dissolve 1 g of the sample in 5 ml of the hydrochloric acid (R 13) and 30 ml of water and apply GM 11.

Prepare a standard matching solution, using 2 ml of the arsenic SS III (2 ml ≙ 0,000 2 % As).

R 38.3.4 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	2 % solution of the sample in 36,5 g/l hydrochloric acid solution	Air-acetylene	324,7
Fe			248,3
Pb			217,0 or 283,3

R 38.3.5 Substances not precipitated by hydrogen sulfide

To 20 ml of the test solution (R 38.2), add 80 ml of water and pass hydrogen sulfide through the solution to precipitate the tin. Filter off the precipitate without washing the residue, and evaporate 50 ml of the filtrate in a weighed porcelain dish to about 2 to 3 ml. Add 0,1 ml of the sulfuric acid (R 37), evaporate to dryness, ignite at 600 ± 50 °C for 15 min, cool and reweigh.

The mass of the residue shall not exceed 1 mg.

R 39 Toluene
$C_6H_5CH_3$

Relative molecular mass : 92,14

R 39.1 Specification

Density : 0,865 to 0,869 g/ml

Assay ($C_6H_5CH_3$)	99,5 % min.
Residue after evaporation	0,002 % max.
Acidity (expressed in millimoles of H^+)	0,03/100 g max.
Alkalinity (expressed in millimoles of OH^-)	0,06/100 g max.
Readily carbonizable substances	To pass test
Sulfur compounds (expressed as S)	0,003 % max.
Thiophene homologues (expressed as C_4H_4S)	To pass test
Water	0,05 % max.

R 39.2 Tests

R 39.2.1 Density

Apply GM 24.1.

R 39.2.2 Assay

Apply GM 34, using the following conditions :

Stationary phase	20 % Carbowax 400
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	80 °C
Injection temperature	160 °C
Detection temperature	160 °C
Type of detector	Thermal conductivity
Carrier gas	Helium
Flow rate	30 ml/min
Sample size	5 µl

R 39.2.3 Residue after evaporation

Take 50 g (58 ml) of the sample and apply GM 14.

The mass of the residue shall not exceed 1 mg.

R 39.2.4 Acidity or alkalinity

Take 43 g (50 ml) of the sample and apply GM 13.2, titrating as appropriate with either standard volumetric sodium hydroxide solution, *c*(NaOH) = 0,01 mol/l, or with standard volumetric sulfuric acid solution, *c*($1/2\ H_2SO_4$) = 0,01 mol/l, using the phenol red (IS 4.3.10*).

In the case of acidity, the volume of titrant shall not exceed 0,7 ml and, in the case of alkalinity, not more than 1,3 ml.

R 39.2.5 Readily carbonizable substances

Shake 12,9 g (15 ml) of the sample with 5 ml of sulphuric acid (95,4 ± 0,5 %) for 15 to 20 s, and allow to stand for 15 min.

The upper toluene layer shall be colourless and the colour of the lower acid layer shall not exceed that of a colour standard described in GM 18 containing 5 g of cobalt(II) chloride hexahydrate, 40 g of iron(III) chloride hexahydrate and 20 ml of the hydrochloric acid (R 13) in 1 litre.

R 39.2.6 Sulfur compounds

Take 2 g (2,2 ml) of the sample and apply GM 21.

Prepare a standard matching solution, using 6 ml of the sulfur SS II (6 ml ≙ 0,003 % S).

R 39.2.7 Thiophene homologues

Carefully add 5 ml of the sample to form a separate layer over 5 ml of the isatin (IS 4.3.4*) in a clean, dry test tube, and allow to stand for 1 h.

No green or blue colour shall appear in the contact surface between the two liquids.

R 39.2.8 Water

Take 8,6 g (10 ml) of the sample and apply GM 12, using 20 ml of the methanol (R 18) as the solvent.

R 40 Zinc
Zn

Relative atomic mass : 65,38

R 40.1 Specification

Arsenic (As)	0,000 01 % max.
Iron (Fe)	0,01 % max.
Lead (Pb)	0,01 % max.

Description : Metal in the form of sticks, granulated lumps or pellets, but not as dust

R 40.2 Tests

R 40.2.1 Arsenic

Carry out the test according to GM 11, but using the apparatus shown in the figure, and proceed as follows :

Place 15 g of the sample in the flask. Place 120 ml of tin(II) chloride solution [0,4 % $SnCl_2.2H_2O$ in the hydrochloric acid (R 13)] in the dropping funnel. Run this solution into the flask, initially drop by drop, until the vigorous evolution of hydrogen subsides. Then continue the addition at such a rate as to ensure a steady evolution of hydrogen. This operation may take as much as 1 h.

Prepare a standard matching solution in the same manner, but placing 1 ml of the arsenic SS III (containing 1 µg of As) and 5 g of the sample in the flask and adding from the dropping funnel, 120 ml of the tin(II) chloride solution (1 ml ≙ 0,000 01 % As).

R 40.2.2 Iron and lead

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

Element	Concentration of solution (in 315 g/l nitric acid solution)	Flame	Resonance line nm
Fe	1 %	Air-acetylene	248,3
Pb	10 %		217,0 or 283,3

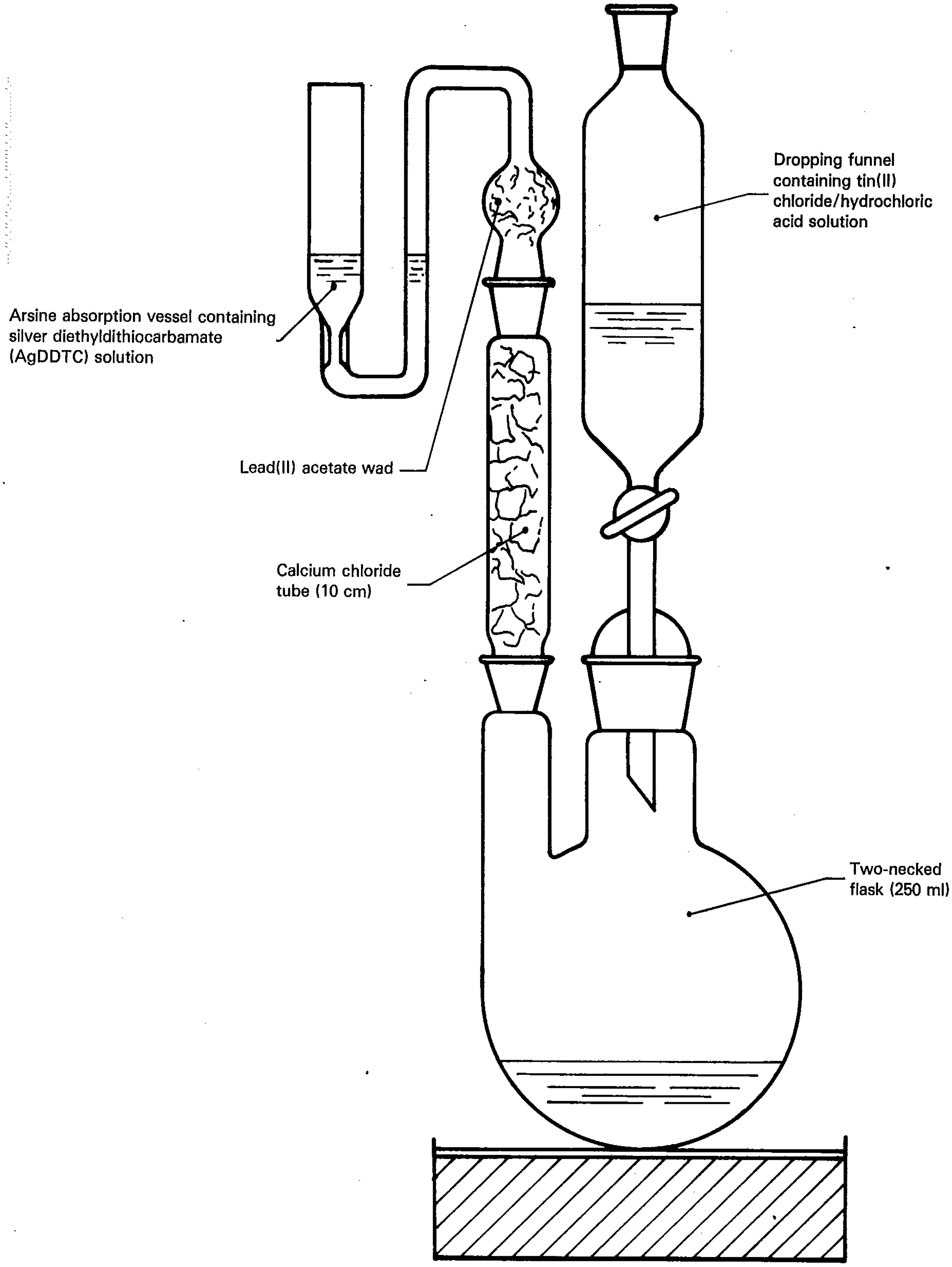

Figure — Apparatus for the determination of the arsenic content of zinc