BRITISH STANDARD

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1989

Reagents for chemical analysis —

Part 3: Specifications (second series)

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Committees responsible for this British Standard

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British Pharmacopoeia Commission
Chemical Industries Association
Institute of Petroleum
Milk Marketing Board
Royal Society of Chemistry
Society of Glass Technology

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National foreword

This Part of BS 6376 has been prepared under the direction of the Chemicals Standards Policy Committee.

Part 1 of this British Standard is identical with ISO 6353-1:1982 *“Reagents for chemical analysis — Part 1: General test methods”* published by the International Organization for Standardization (ISO), and it specifies general test methods to which references are made in the specifications in Part 2 and Part 3. Part 2 of this British Standard is identical with ISO 6353-2:1983 *“Reagents for chemical analysis — Part 2: Specifications — First series”* and comprises a series of specifications for 41 reagents.

This Part of BS 6376 is based on ISO 6353-3:1987 but the final text of the International Standard has been amended to meet some of the objections raised by the United Kingdom when commenting on the draft International Standard. As a consequence, this Part of BS 6376 is technically equivalent to ISO 6353-3 except that the specification for 1,4-dioxane (R 60) differs in the following respects.

a) The requirement and test for residue after evaporation have been omitted.

b) The test for determination of iron content involves the use of a matching solution, instead of atomic absorption spectroscopy.

c) The test for determination of peroxide content has been omitted.

The other United Kingdom objections to the draft International Standard which were not taken into account when ISO 6353-3 was finally published were more general and related to the style of presentation rather than technical content. In order to retain a high degree of equivalence between the British Standard and the International Standard, these points have not been incorporated at this stage but will be raised again during subsequent reviews and revisions.

For ease of production, it has been found convenient to reproduce the text of ISO 6353-3, which has been amended to incorporate the differences outlined above.

For claims of compliance and purchasing orders relating to a specification in this British Standard, it is strongly recommended that the following form of words (using iodine as an example) be adopted:

> “Iodine complying with BS 6376-3: R 68 (1989)”.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 102, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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1 Scope

This Part of BS 6376 gives specifications and indicates the test methods to be used for checking conformity with these specifications for a second series of reagents used in analytical chemistry the numbering system of which continues the first series specified in BS 6376-2.

This document should be read in conjunction with BS 6376-1, 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 BS 6376-1, clause **4**, which describes the preparation of

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

— reagent solutions (RS);

— indicator solutions (IS).

In this part of BS 6376, asterisked clause reference numbers refer to the 1983 edition of BS 6376-1.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

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

General remarks

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

2 For iron determinations according to BS 6337-3 (GM 8.1), no photometric measurement will be executed; the determinations will be carried out using a matching solution as indicated in the relevant monograph.

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

R 41 Acetic anhydride $(CH_3CO)_2O$

Relative molecular mass: 102,09

R 41.1 Specification

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

R 41.2 Preparation of test solution

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

R 41.3 Tests

R 41.3.1 Assay

Pipette 50 ml of morpholine methanolic solution (RS)1) into each of two 250 ml glass-stoppered flasks. For the test portion, weigh accurately 1,8 to 2,0 g of the acetic anhydride from a Lunge pipette into the first flask and swirl to effect dissolution. Reserve the second flask for the determination of the quantity of morpholine mixed with the sample. Allow the flasks to stand at room temperature for 5 min. Add 0,20 to 0,25 ml of methyl yellow — methylene blue mixed indicator (IS)2)3)4).

Titrate each solution with a standard volumetric hydrochloric acid methanolic solution, *c*(HCl) = 0,5 mol/l,1) to the end-point at which the green colour changes to amber.

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

R 41.3.2 Residue after evaporation

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

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

R 41.3.3 Chloride

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

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

R 41.3.4 Sulfate

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

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

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1) **Reagent solutions (RS)**

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

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

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

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

2) **Indicator solution (IS)**

Methyl yellow – methylene blue, mixed solution.

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

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

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

R 41.3.5 Copper, iron and lead

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

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

R 41.3.6 Permanganate-reducing substances

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

The pink colour shall not disappear completely.

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

Relative molecular mass: 392,14

R 42.1 Specification

Assay $[(NH_4)_2Fe(SO_4)_2.6H_2O]$	99 % min.
pH (5 % solution)	3 to 5
Chloride (Cl)	0,001 % max.
Phosphate (PO4)	0,002 % max.
Calcium (Ca)	0,01 % max.
Copper (Cu)	0,002 % max.
Iron(III) (Fe)	0,02 % max.
Lead (Pb)	0,002 % max.
Magnesium (Mg)	0,01 % max.
Manganese (Mn)	0,05 % max.
Potassium (K)	0,01 % max.
Sodium (Na)	0,01 % max.
Zinc (Zn)	0,003 % max.

R 42.2 Preparation of test solution

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

R 42.3 Tests

R 42.3.1 Assay

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

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

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

R 42.3.2 pH

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

R 42.3.3 Chloride

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

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

R 42.3.4 Phosphate

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

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

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

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

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

R 42.3.6 Iron(III)

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

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

R 42.3.7 Potassium and sodium

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

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

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

Relative molecular mass: 482,18

R 43.1 Specification

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

R 43.2 Tests

R 43.2.1 Assay

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

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

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

R 43.2.2 Water-insoluble matter

Take 20 g of the sample and apply GM 1.

The mass of the residue shall not exceed 1 mg.

R 43.2.3 Chloride

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

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

R 43.2.4 Copper, lead, magnesium, manganese and zinc

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

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

R 43.2.5 Iron(II)

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

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

R 43.2.6 Potassium and sodium

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

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

R 44 Ammonium nitrate NH_4NO_3

Relative molecular mass: 80,04

R 44.1 Specification

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

R 44.2 Preparation of test solution

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

R 44.3 Tests

R 44.3.1 Assay

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

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

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

R 44.3.2 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 1 mg.

Reserve the residue for the determination of iron.

R 44.3.3 pH

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

R 44.3.4 Calcium

Determine this element by AAS according to GM 29.

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

R 44.3.5 Chloride

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

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

R 44.3.6 Nitrite

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

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

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

R 44.3.7 Phosphate

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

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

R 44.3.8 Sulfate

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

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

R 44.3.9 Iron

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

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

R 44.3.10 Heavy metals

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

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

R 45 Ammonium sulfate $(NH_4)_2SO_4$

Relative molecular mass: 132,14

R 45.1 Specification

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

R 45.2 Preparation of test solution

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

R 45.3 Tests

R 45.3.1 Assay

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

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

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

R 45.3.2 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 1 mg.

Reserve the residue for the determination of iron.

R 45.3.3 pH

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

R 45.3.4 Chloride

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

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

R 45.3.5 Phosphate

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

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

R 45.3.6 Arsenic

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

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

R 45.3.7 Iron

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

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

R 45.3.8 Heavy metals

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

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

R 46 Ammonium thiocyanate NH_4SCN

Relative molecular mass: 76,12

R 46.1 Specification

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

R 46.2 Tests

R 46.2.1 Assay

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

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

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

R 46.2.2 pH

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

R 46.2.3 Chloride

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

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

R 46.2.4 Sulfate

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

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

R 46.2.5 Sulfide

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

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

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

R 46.2.6 Copper, iron and lead

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

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

R 46.2.7 Iodine-consuming substances

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

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

R 46.2.8 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 3 mg.

R 47 Barium hydroxide octahydrate $Ba(OH)_2.8H_2O$

Relative molecular mass: 315,48

R 47.1 Specification

Assay [$Ba(OH)_2.8H_2O$]	98,0 % min.
Substances insoluble in hydrochloric acid	0,005 % max.
Carbonate (expressed as $BaCO_3$)	2,0 % max.
Chloride (Cl)	0,001 % max.
Sulfide (S)	0,001 % max.
Calcium (Ca)	0,05 % max.
Iron (Fe)	0,001 % max.
Lead (Pb)	0,001 % max.
Strontium (Sr)	1,5 % max.

R 47.2 Tests

R 47.2.1 Assay

Weigh, to the nearest 0,000 1 g, about 6 g of the sample, dissolve in 100 ml of carbon dioxide-free water and titrate with standard volumetric hydrochloric acid solution, *c*(HCl) = 1 mol/l, using the phenolphthalein (IS 4.3.9*).

Retain the solution for the carbonate test.

1,00 ml of hydrochloric acid solution, *c*(HCl) = 1,000 mol/l, corresponds to 0,157 7 g of $Ba(OH)_2.8H_2O$.

R 47.2.2 Substances insoluble in hydrochloric acid

Dissolve 20 g of the sample in a mixture of 15 ml of the hydrochloric acid (R 13) and 400 ml of water. Dilute to 500 ml with water and filter through a sintered glass filter crucible with porosity P 40 (pore size index 16 to 40 µm)], wash the residue with water and dry to constant mass at 105 °C.

The mass of the residue shall not exceed 1 mg.

R 47.2.3 Carbonate

To the solution retained from the assay (R 47.2.1), add 2,0 ml of standard volumetric hydrochloric acid solution, *c*(HCl) = 1 mol/l, boil, cool and titrate with standard volumetric sodium hydroxide solution, *c*(NaOH) = 1 mol/l.

The volume of titrant shall not be less than 0,75 ml (0,75 ml ≙ 2,0 % $BaCO_3$).

R 47.2.4 Chloride

Dissolve 1 g of the sample in a mixture of 2 ml of nitric acid solution (25 %) and 48 ml of water. Boil for 1 min, cool and apply GM 2.

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

R 47.2.5 Sulfide

Dissolve 1 g of the sample in a mixture of 4 ml of water and 5 ml of the acetic acid (R 1). Add 0,5 ml of the lead acetate (basic) (RS 4.2.10*).

Compare any darkening with that produced by the similar treatment of 1 ml of sulfide SS II prepared by diluting freshly prepared sulfide SS I (1 ml ≙ 0,001 % S).

R 47.2.6 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	0,5 %	Air-ethine	422,7
Sr			460,7
Fe	Dissolve 3 g of the sample in 150 ml of water and apply solvent extraction procedure according to GM 35		248,3
Pb			217,0 or 283,3

R 48 Benzene C_6H_6

Relative molecular mass: 78,11

R 48.1 Specification

Colour in Hazen units	10 max.
Density at 20 °C	0,877 to 0,879 g/ml
Assay (C_6H_6)	99,5 % min.
Crystallizing point	5,2 °C min.
Residue after evaporation	0,001 % max.
Acidity (expressed in millimoles of H +)	0,01/100 g max.
Alkalinity (expressed in millimoles of OH –)	0,01/100 g max.
Readily carbonizable substances	To pass test
Sulfur compounds (expressed as S)	0,003 % max.
Thiophene (C_4H_4S)	0,000 2 % max.
Water (H_2O)	0,05 % max.

R 48.2 Tests

R 48.2.1 Colour in Hazen units

Apply GM 36.

R 48.2.2 Density

Apply GM 24.1.

R 48.2.3 Assay

Apply GM 34, using the following conditions:

Stationary phase	15 % Apiezon L or silicone oil
Support	Chromosorb P [0,15 to 0,18 mm (80 to 100 mesh ASTM)]
Column length	2 to 4 m
Column internal diameter	3 mm
Column material	Stainless steel or glass
Column temperature	70 °C
Injection temperature	100 °C
Detection temperature	About 200 °C
Type of detector	Flame ionization
Carrier gas	Nitrogen
Flow rate	About 30 ml/min
Test portion	0,1 µl

R 48.2.4 Freezing point (solidification point)

Apply GM 25.3.

R 48.2.5 Residue after evaporation

Take 100 g (114 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 48.2.6 Acidity

Take 88 g (100 ml) of the sample and apply GM 13.2, 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 0,45 ml.

R 48.2.7 Alkalinity

Take 88 g (100 ml) of the sample and apply GM 13.2, 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*)[5].

The volume of titrant shall not exceed 0,45 ml.

R 48.2.8 Readily carbonizable substances

Shake 8,8 g (10 ml) of the sample with 5 ml of sulfuric acid solution (95,4 ± 0,5 %) for 15 to 20 s and allow to stand for 15 min.

The benzene layer shall be colourless and the colour of the acid layer shall not be more intense than that of a colour standard containing, per litre, 5 g of cobalt(II) chloride hexahydrate, 40 g of iron(III) chloride hexahydrate and 20 ml of the hydrochloric acid (R 13).

R 48.2.9 Sulfur compounds

Take 2 g (2,3 ml) of the sample and apply GM 21.

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

R 48.2.10 Thiophene

Carefully add 5 ml of the sample to form a separate layer over 5 ml of the isatin (IS 4.3.4*)[6] 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 48.2.11 Water

Take 17,6 or 18 g (20 ml) of the sample and apply GM 12, using 20 ml of the methanol (R 18) as the solvent.

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[5] IUPAC name: 2-[4-(dimethylamino) phenylazo] benzoic acid.

[6] IUPAC name: indoline-2,3-dione.

R 49 2,2′-Bipyridyl $(C_5H_4N)_2$

Relative molecular mass: 156,19

R 49.1 Specification

Assay ($C_{10}H_8N_2$)	99,5 % min.
Acid insoluble matter and coloured impurities	To pass test
Melting interval	69,0 to 72,0 °C
Sulfated ash	0,2 % max.

R 49.2 Tests

R 49.2.1 Assay

Weigh, to the nearest 0,000 1 g, 0,6 g of the sample and dissolve in 50 ml of the acetic acid (R 1).

To this solution add 0,4 ml of 1-naphtholbenzein[7] solution [0,1 % in the acetic acid (R 1)] and titrate with standard volumetric perchloric acid solution, $c(HClO_4) = 0,1$ mol/l, in the acetic acid (R 1) to a green colour.

1,00 ml of perchloric acid solution, $c(HClO_4) = 0,100$ mol/l, corresponds to 0,015 62 g of $C_{10}H_8N_2$.

R 49.2.2 Acid-insoluble matter and coloured impurities

Dissolve 1 g of the sample in 10 ml of hydrochloric acid solution, (3,65 %) and dilute to 100 ml with water.

The solution shall remain clear and colourless.

R 49.2.3 Melting range

Apply GM 25.2.

R 49.2.4 Sulfated ash

Take 1 g of the sample and apply GM 16.

The mass of the residue shall not exceed 2 mg.

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[7] IUPAC name: 4-[α-(4-hydroxy-1-naphthyl)benzylidene]naphthalen-1(4*H*)-one.

R 50 Boric acid H_3BO_3

Relative molecular mass: 61,83

R 50.1 Specification

Assay (H_3BO_3)	99,5 % min.
Substances insoluble in methanol	0,005 % max.
Substances non-volatile with methanol	0,05 % max.
Chloride (Cl)	0,001 % max.
Phosphate (PO_4)	0,001 % max.
Sulfate (SO_4)	0,002 % max.
Calcium (Ca)	0,005 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,001 % max.

R 50.2 Preparation of test and reference solutions

R 50.2.1 Test solution

To 10 g of the sample add 1 ml of sodium carbonate solution (1 %), 100 ml of the methanol (R 18), and 5 ml of the hydrochloric acid (R 13). Digest in a covered beaker on a steam bath to effect dissolution, then uncover and evaporate to dryness. Add 75 ml of the methanol (R 18) and 5 ml of the hydrochloric acid (R 13) and repeat the digestion and evaporation. Dissolve the residue in 5 ml of acetic acid solution, (6 %), and digest in a covered beaker on the steam bath for 5 min. Filter and dilute to 100 ml with water.

R 50.2.2 Reference solution

To 1 ml of sodium carbonate solution (1 %), add

1 ml of the phosphate SS I (1 ml $\triangleq$ 0,001 % PO_4);

2 ml of the sulfate SS I (2 ml $\triangleq$ 0,002 % SO_4);

and proceed as described for the preparation of the test solution (R 50.2.1), using the same quantities of reagents.

R 50.3 Tests

R 50.3.1 Assay

Mix 100 ml of glycerol with 100 ml of water and adjust the pH to 9,0, using a calibrated pH meter, by adding standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 0{,}01$ mol/l. Dissolve 2,5 g of the sample, weighed to nearest 0,000 1 g in the mixture and titrate again to pH 9,0 with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 1$ mol/l.

1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 1{,}000$ mol/l, corresponds to 0,061 83 g of H_3BO_3.

R 50.3.2 Substances insoluble in methanol

Heat 20 g of the sample with 200 ml of the methanol (R 18) under complete reflux until the acid is dissolved, then reflux for 30 min. Filter through a tared filtering crucible, wash thoroughly with the hot methanol (R 18) and dry at 105 °C.

The mass of the insoluble residue shall not exceed 1 mg.

R 50.3.3 Substances non-volatile with methanol

To 2,0 g of the powdered acid in a platinum dish, add 25 ml of the methanol (R 18) and 0,5 ml of the hydrochloric acid (R 13) and evaporate to dryness. Add 15 ml of the methanol (R 18) and 0,3 ml of the hydrochloric acid (R 13) and repeat the evaporation. To the residue add 0,10 or 0,15 ml of sulfuric acid (R 37) and ignite at 800 ± 25 °C for 15 min.

The mass of the residue shall not exceed 1 mg.

R 50.3.4 Chloride

Dissolve 2 g of the sample in 40 ml of warm water and filter if necessary. To 20 ml of this solution apply GM 2.

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

R 50.3.5 Phosphate

Take 20 ml of the test solution (R 50.2.1) and apply GM 4.

Prepare a standard matching solution, using 20 ml of the reference solution (R 50.2.2).

R 50.3.6 Sulfate

Take 10 ml of the test solution (R 50.2.1) and apply GM 3.

Prepare a standard matching solution, using 10 ml of the reference solution (R 50.2.2).

R 50.3.7 Calcium, 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	Test solution (R 50.2.1)	Air-ethine	422,7
Fe			248,3
Pb			217,0

R 51 Bromine Br

Relative atomic mass: 79,90

R 51.1 Specification

Assay (Br)	99,0 % min.
Chlorine (Cl)	0,05 % max.
Iodine (I)	0,001 % max.
Sulfate (SO_4)	0,005 % max.
Heavy metals (expressed as Pb)	0,000 2 % max.
Iron (Fe)	0,000 2 % max.
Organic bromine compounds	To pass test
Residue after evaporation	0,005 % max.

R 51.2 Preparation of test solution

To 40 ml of water in a porcelain dish add 20 g (6,4 ml) of the sample and 1 ml of sodium carbonate solution (1 %). Evaporate to dryness on a water-bath, add 1 ml of hydrochloric acid solution (25 %) and again evaporate to dryness. Dissolve the residue in 1 ml of water and 0,1 ml of hydrochloric acid solution (25 %) with gentle heating and dilute to 40 ml with water.

R 51.3 Tests

R 51.3.1 Assay

Seal a thin-walled glass ampoule containing about 0,3 g of the sample, weighed to the nearest 0,000 1 g, and transfer it into a 500 ml conical flask containing 200 ml of water and 5 ml of the potassium iodide (R 25).

Close the flask by means of a ground glass stopper and break the ampoule by shaking vigorously. Lift and rinse the stopper with water and titrate 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 0,007 99 g of Br.

R 51.3.2 Chlorine

Transfer 10 g (3,2 ml) of the sample to a round-bottomed flask containing some small glass beads and 70 ml of anhydrous sodium carbonate (R 30) solution (10 %). Slowly add enough ammonia solution (R 3) just to decolorize the solution. When the evolution of nitrogen ceases boil for 3 to 4 min, add 30 ml of the nitric acid (R 19) and bring again to boiling while passing a stream of nitrogen through the solution until completely decolorized. The volume of the solution shall not be reduced to less than 80 ml.

After cooling add 5,0 ml of standard volumetric silver nitrate solution, $c(AgNO_3)$ = 0,1 mol/l, and 0,5 ml of dibutyl phthalate. Shake vigorously for 1 min and then titrate the excess of silver nitrate with standard volumetric ammonium thiocyanate solution, $c(NH_4SCN)$ = 0,1 mol/l, using the ammonium iron(III) sulfate (IS 4.3.1*).

1,00 ml of silver nitrate solution, $c(AgNO_3)$ = 0,100 mol/l, corresponds to 0,003 545 g of Cl.

R 51.3.3 Iodine

To 10,5 g (3,4 ml) of the sample add 50 ml of water and 1 ml of potassium chloride solution (10 %). Boil the solution until decolorized while keeping its volume constant by the addition of water. Heat for a further 2 min, then cool, add 5 ml of potassium iodide solution (10 %) and 5 ml of sulfuric acid solution (20 %).

The yellow coloration obtained shall not be more intense than that of a similarly prepared standard matching solution, using 0,5 g (0,15 ml) of the sample and 1 ml of the iodide SS I (1 ml $\triangleq$ 0,001 % I).

R 51.3.4 Sulfate

Take 4 ml of the test solution (R 51.2), dilute to 10 ml with water and apply GM 3.

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

R 51.3.5 Heavy metals

Take 10 ml of the test solution (R 51.2) and apply GM 7.

Prepare a standard matching solution, using 1 ml of the lead SS II (1 ml ≙ 0,000 2 % Pb).

R 51.3.6 Iron

Take 20 ml of the test solution (R 51.2) and apply GM 8.1.

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

R 51.3.7 Organic bromine compounds

Mix 3 g (1 ml) of the sample with 30 ml of sodium hydroxide solution (8 %).

After 1 h, the solution shall be free from any oily droplets.

R 51.3.8 Residue after evaporation

Transfer 20 g (6,4 ml) of the sample to a weighed porcelain dish and apply GM 14.

The mass of the residue shall not exceed 1 mg.

R 52 1-Butanol, Butyl alcohol $CH_3(CH_2)_2CH_2OH$

Relative molecular mass: 74,12

R 52.1 Specification

Colour in Hazen units	10 max.
Density at 20 °C	0,808 to 0,811 g/ml
Assay ($C_4H_{10}O$)	99 % min.
Residue after evaporation	0,002 % max.
Acidity (expressed in millimoles of H^+)	0,1/100 g max.
Carbonyl compounds (expressed as CO)	0,02 % max.
Water (H_2O)	0,2 % max.

R 52.2 Tests

R 52.2.1 Colour in Hazen units

Apply GM 36.

R 52.2.2 Density

Apply GM 24.1.

R 52.2.3 Assay

Apply GM 34, using the following conditions:

Stationary phase	20 % Carbowax 400 (or equivalent)
Support	Chromosorb W (or equivalent) [0,15 to 0,18 mm (80 to 100 mesh ASTM)]
Column length	2 m
Column internal diameter	5 mm
Column material	Stainless steel
Column temperature	100 °C
Injection temperature	180 °C
Detection temperature	180 °C
Type of detector	Thermal conductivity
Carrier gas	Helium
Flow rate	About 60 ml/min
Test portion	10 µl

R 52.2.4 Residue after evaporation

Take 50 g (62 ml) of the sample and apply GM 14, drying the residue for 30 min.

The mass of residue shall not exceed 1 mg.

R 52.2.5 Acidity

Take 20 g (25 ml) of the sample and apply GM 13.1, titrating with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 0{,}1$ mol/l and using bromothymol blue[8].

The volume of titrant shall not exceed 0,2 ml.

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[8] IUPAC name: 2,2′-dibromo-6,6′-diisopropyl-3,3′ dimethyl-4,4′-(1,1-dioxo-2,1λ^6-benzoxathiole-3,3-diyl)diphenol.

R 52.2.6 Carbonyl compounds

Dilute 1 g (1,3 ml) of the sample to 20 ml with the carbonyl-free methanol (RS 4.2.11*). Take 1 ml of this solution and apply GM 23.

Prepare a standard matching solution, using 1 ml of the carbonyl SS II (1 ml ≙ 0,01 % CO).

R 52.2.7 Water

Take 10 g (12,5 ml) of the sample and apply GM 12, using 20 ml of the methanol (R 18) as the solvent.

R 53 Calcium carbonate $CaCO_3$

Relative molecular mass: 100,09

R 53.1 Specification

Assay ($CaCO_3$)	99,5 % min.
Substances insoluble in hydrochloric acid	0,005 % max.
Chloride (Cl)	0,01 % max.
Sulfate (SO_4)	0,005 % max.
Barium (Ba)	0,02 % max.
Iron (Fe)	0,001 % max.
Lead (Pb)	0,001 % max.
Magnesium (Mg)	0,03 % max.
Potassium (K)	0,005 % max.
Sodium (Na)	0,1 % max.
Strontium (Sr)	0,05 % max.

R 53.2 Tests

R 53.2.1 Assay

Suspend 2 g, weighed to the nearest 0,000 1 g, in 50 ml of water, carefully add 50,0 ml of standard volumetric hydrochloric acid solution, *c*(HCl) = 1 mol/l, boil, cool and titrate the excess acid with standard volumetric sodium hydroxide solution, *c*(NaOH) = 1 mol/l, using the bromophenol blue (IS 4.3.2*).

1,00 ml of hydrochloric acid solution, *c*(HCl) = 1,000 mol/l, corresponds to 0,050 05 g of $CaCO_3$.

R 53.2.2 Substances insoluble in hydrochloric acid

Dissolve 50 g of the sample in a mixture of 225 ml of the hydrochloric acid (R 13) and 200 ml of water, filter through a sintered glass filter crucible (porosity 16 to 40 µm), wash the residue with diluted hydrochloric acid and dry the residue to constant mass at 105 °C.

The mass of the residue shall not exceed 2,5 mg.

R 53.2.3 Chloride

Dissolve 2 g of the sample in a mixture of 5 ml of nitric acid solution (25 %) and 45 ml of water, boil, cool and apply GM 2.

Prepare a standard matching solution, using 2 ml of the chloride SS I (2 ml ≙ 0,01 % Cl).

R 53.2.4 Sulfate

Dissolve 2 g of the sample in a mixture of 5 ml of the hydrochloric acid (R 13) and 15 ml of water and apply GM 3.

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

R 53.2.5 Barium, potassium and sodium

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

Element	Concentration of solution	Flame	Wavelength nm
Ba	1 % solution of the sample in hydrochloric acid solution, *c*(HCl) = 0,5 mol/l	Air-ethine	455,4
K			766,5
Na			589,0

R 53.2.6 Iron, lead, magnesium and strontium

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

Element	Concentration of solution	Flame	Resonance line nm
Fe	2 % solution of the sample in hydrochloric acid solution, *c*(HCl) = 0,5 mol/l Apply the solvent extraction procedure according to GM 35	Air-ethine	248,3
Pb			217,0 or 283,3
Mg	0,5 % solution of the sample in hydrochloric acid solution, *c*(HCl) = 0,1 mol/l		285,2
Sr	0,2 % solution of the sample in hydrochloric acid solution, *c*(HCl) = 0,1 mol/l		460,7

R 54 Carbon tetrachloride CCl_4

Relative molecular mass: 153,82

R 54.1 Specification

Colour in Hazen units	10 max.
Density at 20 °C	1,592 to 1,598 g/ml
Assay (CCl_4)	99,5 % min.
Residue after evaporation	0,001 % max.
Acidity (expressed as millimoles of H^+)	0,02/100 g max.
Free chlorine	0,000 1 % max.
Carbon disulfide (CS_2)	0,002 % max.
Metallic impurities	To pass test
Water (H_2O)	0,02 % max.

R 54.2 Tests

R 54.2.1 Colour in Hazen units

Apply GM 36.

R 54.2.2 Density

Apply GM 24.1.

R 54.2.3 Assay

Apply GM 34, using the following conditions:

Stationary phase	15 % Apiezon L
Support	Chromosorb P-AW-DMCS [0,15 to 0,18 mm (80 to 100 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	150 °C
Type of detector	Flame ionization
Carrier gas	Nitrogen
Flow rate	About 60 ml/min
Test portion	0,1 µl

R 54.2.4 Residue after evaporation

Take 100 g (63 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 54.2.5 Acidity

Take 20 g (13 ml) of the sample and apply GM 13.2, 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 0,2 ml.

R 54.2.6 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 glass stoppered flask for 2 min. If either a blue or 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 0,1 ml.

R 54.2.7 Carbon disulfide

To 10 g (6,3 ml) of the sample add 1 ml of ethanol absolute and 3 ml of an alkaline lead(II) acetate solution (RS)[9], and boil under reflux for 15 min. Allow to cool.

After 5 min any yellow coloration shall not be more intense than that of a similarly treated mixture of carbon disulfide-free carbon tetrachloride and 2 ml of a carbon disulfide SS I (prepared by mixing carbon disulfide with carbon disulfide-free carbon tetrachloride) (2 ml ≙ 0,002 % CS_2).

R 54.2.8 Metallic impurities

To 50 ml of the sample add 0,1 ml of dithizone solution [0,1 % in the chloroform (R 7)].

The colour of the resulting solution shall be green with no trace of yellow.

R 54.2.9 Water

Take 20 g (13 ml) of the sample and apply GM 12, using 20 ml of the methanol (R 18) as the solvent.

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[9] **Reagent solution (RS)**
Lead(II) acetate, alkaline solution.
Dissolve 1,7 g lead(II) acetate trihydrate ($C_4H_6O_4Pb.3H_2O$) in 50 ml of water and add 3,1 g of trisodium citrate dihydrate ($C_6H_5O_7Na_3.2H_2O$) and 50 g of potassium hydroxide (KOH). Allow to cool and dilute to 100 ml.

R 55 Cobalt(II) chloride hexahydrate $CoCl_2.6H_2O$

Relative molecular mass: 237,93

R 55.1 Specification

Assay ($CoCl_2.6H_2O$)	99 % min.
Water-insoluble matter	0,01 % max.
Sulfate (SO_4)	0,01 % max.
Substances not precipitated by ammonium sulfide	0,25 % max.
Copper (Cu)	0,001 % max.
Nickel (Ni)	0,05 % max.
Zinc (Zn)	0,002 % max.

R 55.2 Tests

R 55.2.1 Assay

Weigh, to the nearest 0,000 1 g, about 1 g of the sample and dissolve in 300 ml of water.

To this solution add 4 g of hydroxylammonium chloride and 25 ml of ammonia solution (R 3). Titrate at 80 °C with standard volumetric EDTA[10] solution, c(EDTA) = 0,1 mol/l, using the methylthymol blue mixture (4.3.7*)[11], until the colour changes from blue to purple.

1,00 ml of EDTA solution, c(EDTA) = 0,100 mol/l, corresponds to 0,023 79 g of $CoCl_2.6H_2O$.

R 55.2.2 Water-insoluble matter

Take 10 g of the sample and apply GM 1.

The mass of the residue shall not exceed 1 mg.

R 55.2.3 Sulfate

Dissolve 10 g of the sample in 100 ml of water, filter, add 1 ml of the hydrochloric acid (R 13) and heat to boiling on a steam bath. Add 1,5 g of the barium chloride dihydrate (R 6), digest in a covered beaker placed on the steam bath for 2 h and allow to stand for 12 h. If a precipitate is formed, filter, wash thoroughly, and ignite at 800 ± 25 °C.

The mass of the precipitate shall not be more than 2,4 mg greater than the mass obtained in a complete blank test.

R 55.2.4 Substances not precipitated by ammonium sulfide

Dissolve 2 g of the sample in 20 ml of water. Add 1 g of the ammonium chloride (R 5) and 5 ml of ammonia solution (R 3), dilute with water to 90 ml, and pass in hydrogen sulfide until all the cobalt is precipitated. Dilute with water to 100 ml, mix well, and filter. Evaporate 50 ml of the clear filtrate nearly to dryness in a tared dish. Cool, add 0,05 ml of the sulfuric acid (R 37), and ignite carefully to volatilize the excess salts and acids. Finally, ignite at 800 ± 25 °C for 15 min.

The mass of the residue shall not exceed 2,5 mg.

R 55.2.5 Copper, nickel and zinc

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

Element	Concentration of solution	Flame	Resonance line nm
Cu	5 %	Air-ethine	324,7
Ni	0,5 %		232,0
Zn	5 %		213,9

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[10] IUPAC name: disodium dihydrogen ethylenedinitrilotetraacetate.

[11] IUPAC name: tetrasodium 2,2′-dimethyl-6,6′-dihydroxy-5,5′-diisopropyl-3,3′-(1,1-dioxo-$2,1\lambda^6$-benzoxathiole-3,3-diyl)dibenzyldinitrilotetraacetate.

R 56 Diammonium oxalate monohydrate $(NH_4)_2C_2O_4.H_2O$

Relative molecular mass: 142,11

R 56.1 Specification

Assay $[(NH_4)_2C_2O_4.H_2O]$	99,5 % min.
Chloride (Cl)	0,000 5 % max.
Nitrate (NO_3)	0,002 % max.
Sulfate (SO_4)	0,005 % max.
Calcium (Ca)	0,001 % max.
Heavy metals (expressed as Pb)	0,001 % max.
Iron (Fe)	0,000 2 % max.
Magnesium (Mg)	0,001 % max.
Potassium (K)	0,001 % max.
Sodium (Na)	0,001 % max.

R 56.2 Preparation of test solutions

R 56.2.1 Test solution I

Dissolve 10 g of the sample in water and dilute to 200 ml (the solution shall be clear and colourless).

R 56.2.2 Test solution II

To 5 g of the sample, add 1 drop of ammonium trioxovanadate solution (1 %), evaporate to dryness several times with the nitric acid (R 19) until decomposition is complete and then with hydrochloric acid solution (25 %). Dissolve the residue in 1 ml of hydrochloric acid solution (25 %) and dilute to 50 ml with water.

R 56.2.3 Test solution III

Adopt the procedure specified in 56.2.2, but using 2,5 g of the sample.

R 56.3 Tests

R 56.3.1 Assay

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

To this solution, add 6 ml of the sulfuric acid (R 37). Heat to 60 °C and titrate with a standard volumetric potassium permanganate solution, $c(1/5\ KMnO_4) = 0{,}02$ 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 0,007 105 5 g of $(NH_4)_2C_2O_4.H_2O$.

R 56.3.2 Chloride

To 20 ml of the test solution I (R 56.2.1), add 10 ml of nitric acid solution (25 %) and apply GM 2.

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

R 56.3.3 Nitrate

Dissolve 1 g of the sample in 10 ml of sulfuric acid solution (16 %) and add 0,2 ml of brucine solution (0,5 % in glacial acetic acid) and 20 ml of the sulfuric acid (R 37).

After 10 min, the yellow coloration of the resulting solution shall not be more intense than that of a similarly prepared standard matching solution, using 2 ml of the nitrate SS II (2 ml $\triangleq$ 0,002 % NO_3).

R 56.3.4 Sulfate

Take 20 ml of the test solution II (R 56.2.2) and apply GM 3, without addition of 0,5 ml of hydrochloric acid solution (20 %).

Prepare a standard matching solution, using 20 ml of the test solution III (R 56.2.3) and 5 ml of the sulfate SS II (5 ml $\triangleq$ 0,005 % SO_4).

R 56.3.5 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-ethine	422,7
Mg			285,2

R 56.3.6 Heavy metals

Neutralize 20 ml of the test solution II (R 56.2.2) and apply GM 7.

Prepare a standard matching solution, using 20 ml of the test solution III (R 56.2.3) and 1 ml of the lead SS II (1 ml ≙ 0,001 % Pb).

R 56.3.7 Iron

To 20 ml of the test solution I (R 56.2.1), add 2 ml of 5-sulfosalicylic acid solution (10 %) and 5 ml of the ammonia solution (R 3).

After 10 min, the yellow coloration of the resulting solution shall not be more intense than that of a similarly prepared standard matching solution, using 0,2 ml of the iron SS II (0,2 ml ≙ 0,000 2 % Fe).

R 46.3.8 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 %	Air-ethine	766,5
Na			589,0

R 57 Dichloromethane CH_2Cl_2

Relative molecular mass: 84,93

R 57.1 Specification

Colour in Hazen units	10 max.
Density at 20 °C	1,320 to 1,330 g/ml
Assay (CH_2Cl_2)	99,5 % min.
Residue after evaporation	0,002 % max.
Acidity (expressed in millimoles of H^+)	0,03/100 g max.
Free chlorine (Cl)	0,000 1 % max.
Iron (Fe)	0,000 1 % max.
Water (H_2O)	0,05 % max.

R 57.2 Tests

R 57.2.1 Colour in Hazen units

Apply GM 36.

R 57.2.2 Density

Apply GM 24.1.

R 57.2.3 Assay

Apply GM 34, using the following conditions:

Stationary phase	15 % Apiezon L
Support	Chromosorb P [0,15 to 0,18 mm (80 to 100 mesh ASTM)]
Column length	2 to 4 m
Column internal diameter	About 3 mm
Column material	Stainless steel or glass
Column temperature	40 to 50 °C
Injection temperature	About 100 °C
Detection temperature	150 to 300 °C
Type of detector	Flame ionization
Carrier gas	Nitrogen
Flow rate	40 to 50 ml/min
Test portion	0,1 µl

R 57.2.4 Residue after evaporation

Take 100 g (75,5 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 57.2.5 Acidity

Take 30 g (22,5 ml) of the sample and apply GM 13.2, 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 0,45 ml.

R 57.2.6 Free chlorine

Shake 35 g (25,5 ml) of the sample with 20 ml of freshly boiled and cooled water containing 1 ml of cadmium iodide solution (0,15 %) and 1 ml of starch (IS 4.3.11*). Set aside in the dark for 10 min and titrate with standard volumetric sodium thiosulfate solution, $c(Na_2S_2O_3) = 0,01$ mol/l.

The volume of titrant shall not exceed 0,1 ml.

R 57.2.7 Iron

Evaporate 20 g (15 ml) of the sample to dryness with 5 mg of the sodium carbonate (R 30) and dissolve the residue in 10 ml of water containing 0,5 ml of the nitric acid (R 19).

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

Element	Concentration of solution	Flame	Resonance line nm
Fe	Solution prepared as described above	Air-ethine	248,3

R 57.2.8 Water

Take 20 g (15 ml) of the sample and apply GM 12, using 20 ml of the methanol (R 18) as the solvent.

R 58 Diethyl ether $(C_2H_5)_2O$

Relative molecular mass: 74,12

R 58.1 Specification

Colour in Hazen units	10 max.
Density at 20 °C	0,713 ± 0,001 g/ml
Assay [$(C_2H_5)_2O$]	99,5 % min.[a]
Ethanol (C_2H_5OH)	0,05 % max.
Methanol (CH_3OH)	0,02 % max.
Residue after evaporation	0,001 % max.
Acidity (expressed in millimoles of H^+)	0,02/100 g max.
Carbonyl compounds (expressed as CO)	0,001 % max.
Peroxide (expressed as H_2O_2)	0,000 03 % max.
Substances darkened by sulfuric acid	To pass test
Water (H_2O)	0,2 % max.

[a] Diethyl ether usually contains a suitable amount of a stabilizer.

R 58.2 Tests

R 58.2.1 Colour in Hazen units

Apply GM 36.

R 58.2.2 Density

Apply GM 24.1.

R 58.2.3 Assay, ethanol and methanol

Apply GM 34, using the following conditions:

Stationary phase	15 % bis(2-ethylhexyl) sebacate
Support	Chromosorb G-AW-DMCS [0,15 to 0,18 mm (80 to 100 mesh ASTM)] or Volaspher A2
Column length	4 m
Column internal diameter	2,5 mm
Column material	Steel or glass
Column temperature	50 °C
Injection temperature	150 °C
Detection temperature	150 °C
Type of detector	Flame ionization
Carrier gas	Nitrogen
Flow rate	25 ml/min
Test portion	0,2 µl

R 58.2.4 Residue after evaporation

Take 100 g (141 ml) of the sample and apply GM 14, drying the residue for 30 min. The mass of the residue shall not exceed 1 mg.

NOTE This test should not be carried out unless the requirement for peroxide has been met.

R 58.2.5 Acidity

Take 20 g (28 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 bromothymol blue as indicator.

The volume of titrant shall not exceed 0,2 ml.

R 58.2.6 Carbonyl compounds

To 1 g (1,4 ml) of the sample, in a colourless glass cylinder with glass stopper, add 2 ml of the carbonyl-free methanol (RS 4.2.11*) and 2 ml of the 2,4-dinitrophenylhydrazine (RS 4.2.7*), stopper the cylinder, shake and allow to stand for 30 min. Remove the stopper, place the cylinder in a hot water bath at 50 to 55 °C and boil off the ether with the aid of a stream of nitrogen. Remove the cylinder from the water bath, cool to room temperature, add 8 ml of pyridine, 2 ml of water and 2 ml of the methanolic potassium hydroxide (RS 4.2.15*), shake, allow to stand for 10 min and dilute to 25 ml with the carbonyl-free methanol (RS 4.2.11*).

The dark red colour of the resulting solution shall not be more intense than that of a similarly prepared standard matching solution, using 1 ml of the carbonyl SS II (1 ml $\triangleq$ 0,001 % CO).

R 58.2.7 Peroxide

Fill a 120 ml ground glass stoppered flask completely with 10 ml of potassium iodide solution (10 %) and 110 ml (78 g) of the sample. Close the bottle, shake vigorously, and allow to stand in the dark.

After 1 h, the aqueous phase shall remain colourless.

Detection limit: 0,000 03 % H_2O_2.

R 58.2.8 Substances darkened by sulfuric acid

Cool 10 ml of the sulfuric acid (R 37) to 10 °C and add to it dropwise, with stirring, 10 ml of the sample.

After 5 min, the coloration of the clear solution shall not be more than 20 Hazen units.

R 58.2.9 Water

Take 14 g (20 ml) of the sample and apply GM 12, using 20 ml of the methanol (R 18) as the solvent.

R 59 Dimethylformamide $HCON(CH_3)_2$

Relative molecular mass: 73,09

R 59.1 Specification

Colour in Hazen units	10 max.
Density at 20 °C	0,945 to 0,950 g/ml
Assay [$HCON(CH_3)_2O$]	99,5 % min.
Residue after evaporation	0,005 % max.
Acidity (expressed in millimoles of H^+)	0,1/100 g max.
Alkalinity (expressed in millimoles of OH^-)	0,1/100 g max.
Iron (Fe)	0,000 5 % max.
Water (H_2O)	0,1 % max.

R 59.2 Tests

R 59.2.1 Colour in Hazen units

Apply GM 36.

R 59.2.2 Density

Apply GM 24.1.

R 59.2.3 Assay

Apply GM 34, using the following conditions:

Stationary phase	15 % Carbowax 20 M or PEG 400
Support	Chromosorb W [0,15to 0,18 mm (80 to 100 mesh ASTM)] or Celite
Column length	2 to 4 m
Column internal diameter	About 3 mm
Column material	Stainless steel or glass
Column temperature	100 to 150 °C
Injection temperature	About 165 °C
Detection temperature	200 to 300 °C
Type of detector	Flame ionization
Carrier gas	Nitrogen
Flow rate	About 50 ml/min
Test portion	0,1 µl

R 59.2.4 Residue after evaporation

Take 100 g (105 ml) of the sample and apply GM 14, drying the residue for 30 min.

The mass of the residue shall not exceed 5 mg.

R 59.2.5 Acidity

Take 10 g (10,5 ml) of the sample and apply GM 13.1, titrating with standard volumetric sodium hydroxide solution, (NaOH) = 0,01 mol/l, using the phenolphthalein (IS 4.3.9*).

The volume of titrant shall not exceed 1 ml.

R 59.2.6 Alkalinity

Take 10 g (10,5 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, using the methyl red (IS 4.3.6*).

The volume of titrant shall not exceed 1 ml.

R 59.2.7 Iron

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

Element	**Concentration of solution**	**Flame**	**Resonance line** nm
Fe	20 %	Air-ethine	248,3

R 59.2.8 Water

Take 20 g (21 ml) of the sample and apply GM 12, using 20 ml of the methanol (R 18) as the solvent.

R 60 1,4-Dioxane $CH_2\text{-}CH_2\text{-}O\text{-}CH_2\text{-}CH_2\text{-}O$

Relative molecular mass: 88,11

R 60.1 Specification

Colour in Hazen units	10 max.
Density at 20 °C	1,030 to 1,035 g/ml
Assay ($C_4H_8O_2$)	99,5 % min.
Freezing point	11,0 °C min.
Acidity (expressed in millimoles of H^+)	0,3/100 g max.
Iron (Fe)	0,000 1 % max.
Water (H_2O)	0,10 % max.

R 60.2 Tests

R 60.2.1 Colour in Hazen units

Apply GM 36.

R 60.2.2 Density

Apply GM 24.1.

R 60.2.3 Assay

Apply GM 34, using the following conditions:

Stationary phase	Silicone oil OV 17 – 15 %
Support	Chromosorb G-AW-DMCS [0,15 to 0,18 mm (80 to 100 mesh ASTM)]
Column length	4 m
Column internal diameter	4 mm
Column material	Stainless steel
Column temperature	130 °C
Injection temperature	250 °C
Detection temperature	250 °C
Type of detector	Thermal conductivity or flame ionization
Carrier gas	Helium
Flow rate	50 ml/min
Test portion	5 µl or 0,2 µl

R 60.2.4 Freezing point

Apply GM 25.3.

R 60.2.5 Acidity

Take 33 g (32 ml) of the sample and apply GM 13.1, titrating with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 0{,}1$ mol/l, and using the phenolphthalein (IS 4.3.9*).

The volume of titrant shall not exceed 1 ml.

R 60.2.6 Iron

Dilute 10 g (9,7 ml) of the sample to 60 ml with water and apply GM 8.1.

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

R 60.2.7 Water

Take 20 g (19,5 ml) of the sample and apply GM 12, using 20 ml of the methanol (R 18) as the solvent.

R 61 Disodium tetraborate decahydrate $Na_2B_4O_7.10H_2O$

Relative molecular mass: 381,36

R 61.1 Specification

Assay ($Na_2B_4O_7.10H_2O$)	99,5 % min.
Chloride (Cl)	0,001 % max.
Phosphate (PO_4)	0,002 % max.
Sulfate (SO_4)	0,005 % max.
Calcium (Ca)	0,005 % max.
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,001 % max.

R 61.2 Tests

R 61.2.1 Assay

Weigh, to the nearest 0,000 1 g, about 4 g of the sample and dissolve in 100 ml of water and 25,0 ml of standard volumetric hydrochloric acid solution, *c*(HCl) = 1 mol/l. Neutralize with standard volumetric sodium hydroxide solution, *c*(NaOH) = 1 mol/l, using the methyl red (IS 4.3.6*). Add 15 g of mannitol and titrate with sodium hydroxide solution, *c*(NaOH) = 1 mol/l, to pH 9,0.

1,00 ml of sodium hydroxide solution, *c*(NaOH) = 1,000 mol/l, corresponds to 0,095 34 g of $Na_2B_4O_7.10H_2O$.

R 61.2.2 Chloride

Dissolve 1 g of the sample in a mixture of 48 ml of water and 2 ml of nitric acid solution (25 %) and apply GM 2.

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

R 61.2.3 Phosphate

Dissolve 1 g of the sample in 20 ml of warm water and apply GM 4.

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

R 61.2.4 Sulfate

Dissolve 1 g of the sample in 20 ml of warm water and apply GM 3.

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

R 61.2.5 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-ethine	422,7
Cu	Dissolve 6 g of the sample in 150 ml of water and apply solvent extraction procedure according to GM 35.		324,7
Fe			248,3
Pb			217,0 or 283,3

R 62 Ethyl acetate $CH_3COOC_2H_5$

Relative molecular mass: 88,11

R 62.1 Specification

Colour in Hazen units	10 max.
Density at 20 °C	0,898 to 0,902 g/ml
Assay ($C_4H_8O_2$)	99,5 % min.
Ethanol (C_2H_5OH)	0,1 % max.
Methanol (CH_3OH)	0,1 % max.
Methyl acetate ($CH_3CO_2CH_3$)	0,1 % max.
Residue after evaporation	0,002 % max.
Acidity (expressed in millimoles of H^+)	0,08/100 g max.
Readily carbonizable substances	To pass test
Water (H_2O)	0,1 % max.

R 62.2 Tests

R 62.2.1 Colour in Hazen units

Apply GM 36.

R 62.2.2 Density

Apply GM 24.1.

R 62.2.3 Assay, ethanol, methanol and methyl acetate

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 mm
Column material	Stainless steel
Column temperature	Raising from 90 to 240 °C (6 °C/min)
Injection temperature	250 °C
Detection temperature	250 °C
Type of detector	Thermal conductivity
Carrier gas	Helium
Flow rate	20 ml/min
Test portion	3 µl

R 62.2.4 Residue after evaporation

Take 50 g (55,5 ml) of the sample and apply GM 14

The mass of the residue shall not exceed 1 mg.

R 62.2.5 Acidity

To 20 ml of the methanol (R 18) add 0,1 ml of the phenolphthalein (IS 4.3.9*) and sufficient standard volumetric sodium hydroxide solution, $c(NaOH) = 0,01$ mol/l, to produce a pink coloration. Cool in an ice bath and add 20 g (22,2 ml) of the sample. If necessary, titrate with standard volumetric sodium hydroxide solution, $c(NaOH) = 0,01$ mol/l, until the initial pink coloration is just restored.

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

R 62.2.6 Readily carbonizable substances

Cool 5 ml of sulfuric acid solution (95 ± 0,5 %) to 10 °C and slowly, with continuous shaking, add 5 ml of the sample.

After 5 min, the coloration of the resulting solution shall not be more intense than that of iodine solution, $c(1/2\ I_2) = 5 \times 10^{-5}$ mol/l.

R 62.2.7 Water

Take 10 g (11,1 ml) of the sample and apply GM 12, using 20 ml of the methanol (R 18) as the solvent.

R 63 Formaldehyde solution H-CHO

Relative molecular mass: 30,03

R 63.1 Specification

Colour in Hazen units	10 max.
Density at 20 °C	1,075 to 1,090 g/ml
Assay (HCHO)	37,0 to 40,0 % (*m*/*m*)
Methanol (CH_3OH)	8,0 to 14,0 %
Residue on heating	0,005 % max.
Acidity (expressed in millimoles of H^+)	1,0/100 g max.
Chloride (Cl)	0,000 2 % max.
Sulfate (SO_4)	0,002 % max.
Iron (Fe)	0,000 2 % max.
Lead (Pb)	0,000 2 % max.

R 63.2 Tests

R 63.2.1 Colour in Hazen units

Apply GM 36.

R 63.2.2 Density

Apply GM 24.1.

R 63.2.3 Assay

Weigh, to the nearest 0,000 1 g, about 3 g of the sample, add 50,0 ml of standard volumetric sodium hydroxide solution, *c*(NaOH) = 1 mol/l and 25 ml of hydrogen peroxide solution (6 %). Warm on a steam bath for 30 min, cool and titrate with standard volumetric hydrochloric acid solution, *c*(HCl) = 1 mol/l, using the phenolphthalein (IS 4.3.9*). Carry out a similar determination, omitting the sample.

1,00 ml of hydrochloric acid solution, *c*(HCl) = 1,000 mol/l, corresponds to 0,030 03 g of HCHO.

R 63.2.4 Methanol

Apply GM 34, using the following conditions:

Stationary phase	—
Support	Porapak T [0,15 to 0,18 mm (80 to 100 mesh ASTM)]
Column length	About 1,5 m
Column internal diameter	3 to 4 mm
Column material	Glass
Column temperature	100 to 150 °C
Injection temperature	100 to 200 °C
Detection temperature	200 to 300 °C
Type of detector	Flame ionization
Carrier gas	Nitrogen
Flow rate	About 50 ml/min
Test portion	1 µl

R 63.2.5 Residue on heating

Take 50 g (46 ml) of the sample and apply GM 15, drying the residue for 30 min.

The mass of the residue shall not exceed 2,5 mg.

R 63.2.6 Acidity

Take 20 g (18,5 ml) of the sample and apply GM 13.1, titrating with standard volumetric sodium hydroxide solution, $c(\mathrm{NaOH}) = 0{,}1$ mol/l, using the phenolphthalein (IS 4.3.9*).

The volume of titrant shall not exceed 2 ml.

R 63.2.7 Chloride

Dilute 5 g (4,6 ml) of the sample with 45 ml of water and apply GM 2.

Prepare a standard matching solution, using 1 ml of the chloride SS II (1 ml $\triangleq$ 0,000 2 % (I).

R 63.2.8 Sulfate

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

Prepare a standard matching solution, using 0,5 ml of the sulfate SS I (0,5 ml $\triangleq$ 0,002 % SO_4).

R 63.2.9 Iron and lead

Dilute 5 g (4,5 ml) of the sample to 10 ml with water and determine these elements by AAS according to GM 29, using the following conditions:

Element	Concentration of solution	Flame	Resonance line nm
Fe	50 %	Air-ethine	248,3
Pb			217,0 or 283,3

R 64 Glycerol $HOCH_2CHOHCH_2OH$

Relative molecular mass: 92,10

R 64.1 Specification

Colour in Hazen units	10 max.
Assay ($C_3H_8O_3$, calculated for the anhydrous compound)	99 % min.
Acidity (expressed in millimoles of H^+)	0,05/100 g max.
Alkalinity (expressed in millimoles of OH^-)	0,03/100 g max.
Sulfated ash	0,005 % max.
Ammonium (NH_4)	0,000 5 % max.
Arsenic (As)	0,000 1 % max.
Heavy metals (expressed as Pb)	0,000 1 % max.
Iron (Fe)	0,000 1 % max.
Chloride (Cl)	0,000 1 % max.
Sulfate (SO_4)	0,000 5 % max.
Esters of fatty acids (expressed as glycerol tributyrate)	0,05 % max.
Glyceraldehyde	To pass test (about 0,003 %)
Readily carbonizable substances	To pass test
Water (H_2O)	1 % max.[a]

[a] Glycerol conforming otherwise to the above specification is also commercially available with a water content of about 13 %.

R 64.2 Preparation of test solution

Dilute 20 g (16 ml) of the sample to 100 ml with water.

R 64.3 Tests

R 64.3.1 Colour in Hazen units

Apply GM 36.

R 64.3.2 Assay

Transfer 0,1 ml of the sample to a small distillation flask, add 0,5 ml of acetonitrile and 1 ml of trimethylsilyl *N*-(trimethylsilyl) acetimidate. Boil under reflux for 45 min and allow to cool (solution A).

Apply GM 34, using the calculation for the internal normalization method (approximate estimations, where the calibration factors are considered to be equal to 1) and the following conditions:

Stationary phase	10 % silicone SE 52
Support	Embacel [0,15 to 0,25 mm (60 to 100 mesh ASTM)]
Column length	3 m
Column internal diameter	2 mm
Column material	Stainless steel
Column temperature	Raising from 130 to 300 °C (4 °C/min)
Injection temperature	250 °C
Detection temperature	300 °C
Type of detector	Thermal conductivity
Carrier gas	Helium

Flow rate	20 ml/min
Test portion (solution A)	20 µl

R 64.3.3 Acidity or alkalinity

Take 30 g (24 ml) of the sample and apply GM 13.1, titrating with either standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 0{,}01$ mol/l, or hydrochloric acid solution, $c(\text{HCl}) = 0{,}01$ mol/l, and using bromothymol blue as indicator.

In the case of acidity, the volume of titrant shall not exceed 1,65 ml and, in the case of alkalinity, not more than 1 ml.

R 64.3.4 Sulfated ash

Take 20 g (16 ml) of the sample and apply GM 17.

The mass of the residue shall not exceed 1 mg.

Reserve the residue for the determination of iron.

R 64.3.5 Ammonium

Take 20 ml of the test solution (R 64.2) and apply GM 6 omitting the addition of Devarda's alloy and distilling immediately.

Prepare a standard matching solution, using 2 ml of the ammonium SS II (2 ml ≙ 0,000 5 % NH_4).

R 64.3.6 Arsenic

Take 10 ml of the test solution (R 64.2) and apply GM 11.

Prepare a standard matching solution, using 2 ml of the arsenic SS III (2 ml ≙ 0,000 1 % As).

R 64.3.7 Heavy metals

Dilute 10 g (8 ml) of the sample to 20 ml with water and apply GM 7.

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

R 64.3.8 Iron

To the residue of test (R 64.3.4) add 1 ml of the hydrochloric acid (R 13) and evaporate to dryness on a water bath. Dissolve the residue in 0,2 ml of hydrochloric acid solution (25 %) and dilute to 20 ml with water.

Take 10 ml of this solution 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 64.3.9 Chloride

Take 25 ml of the test solution (R 64.2) and apply GM 2.

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

R 64.3.10 Sulfate

Take 25 ml of the test solution (R 64.2) add 5 ml of the methanol (R 18) and apply GM 3.

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

R 64.3.11 Esters of fatty acids

To 40 g (32 ml) of the sample add 50 ml of carbon dioxide-free water and 10 ml of standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 0{,}1$ mol/l. Heat on a boiling water bath under reflux for 30 min. Cool and titrate the excess of sodium hydroxide with standard volumetric hydrochloric acid solution, $c(\text{HCl}) = 0{,}1$ mol/l, using thymol blue as indicator.

Carry out a blank determination replacing the sample by an equal volume of water.

1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 0{,}100$ mol/l, corresponds to 0,010 08 g of glycerol tributyrate.

The volume of the standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 0{,}1$ mol/l, required shall not exceed 2 ml.

R 64.3.12 Glyceraldehyde

To 5 ml of the sample add 5 ml of diluted ammonia solution (5 %), 0,5 ml of silver nitrate solution (1,7 %) and heat to 60 °C for 5 min, taking care to protect the solution from light.

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

The solution shall remain clear and colourless.

R 64.3.13 Readily carbonizable substances

To 5 ml of the sample add slowly 5 ml of sulfuric acid solution (95 ± 0,5 %), shake vigorously and allow to stand for 1 h.

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

R 64.3.14 Water

Weigh, to the nearest 0,000 1 g, 2 g of the sample, dilute with 20 ml of the methanol (R 18) and apply GM 12.

R 65 Hexaammonium heptamolybdate tetrahydrate $(NH_4)_6Mo_7O_{24}.4H_2O$

Relative molecular mass: 1 235,86

R 65.1 Specification

Assay $[(NH_4)_6Mo_7O_{24}.4H_2O]$	99 % min.
Water-insoluble matter	0,01 % max.
Chloride (Cl)	0,002 % max.
Arsenate, phosphate and silicate (expressed as SiO_2)	0,001 % max.
Sulfate (SO_4)	0,02 % max.
Lead (Pb)	0,001 % max.

R 65.2 Tests

R 65.2.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,3 g of the sample and dissolve it in 50 ml of water, add sufficient hexamethylene-tetramine to adjust the pH to between 5 and 6 (approximately 4 g). Heat to 60 °C and titrate with a standard volumetric lead nitrate solution, $c[Pb(NO_3)_2] = 0,05$ mol/l, using a 0,1 % aqueous solution of 4-(2-pyridylazo) resorcinol, disodium salt (PAR)[12] as indicator, adding further amounts of hexamethylene-tetramine to keep the solution orange, until the colour changes sharply from orange to red.

1,00 ml of lead(II) nitrate solution, $c[Pb(NO_3)_2] = 0,050$ mol/l, corresponds to 0,008 828 g of $(NH_4)_6Mo_7O_{24}.4H_2O$.

R 65.2.2 Water-insoluble matter

Take 10 g of the sample and apply GM 1.

The mass of the residue shall not exceed 1 mg.

R 65.2.3 Chloride

Dissolve 1 g of the sample in 50 ml of water, and filter if necessary, through a chloride-free filter. To 25 ml of the solution add 2 ml of the nitric acid (R 19) and apply GM 2.

Prepare a standard matching solution, using 1 ml of the chloride SS II (1 ml $\triangleq$ 0,002 % Cl).

R 65.2.4 Arsenate, phosphate and silicate

In a platinum or plastics vessel dissolve 2,5 g of the sample in 70 ml of water; at the same time prepare a reference solution containing 0,5 g of the sample and 2 ml of the silicate SS II (2 ml $\triangleq$ 0,001 % SiO_2). Adjust the pH to 3 to 4 with hydrochloric acid solution (ϱ approximately 1,19 g/ml) diluted (1 + 9) and transfer the solution to two glass beakers; add 1 to 2 ml of bromine water, adjust the pH to 1,7 to 1,9 with hydrochloric acid solution (R 13) diluted (1 + 9) and allow to stand for 30 min, then add 10 ml of the hydrochloric acid (R 13), dilute to 100 ml and transfer both solutions to two separating funnels. Extract with a mixture of 1 ml of 1-butanol and 30 ml of 4-methyl-2-pentanone, discharge the aqueous phases and wash the ketonic phases three times with 10 ml of hydrochloric acid solution (ϱ approximately 1,19 g/ml) diluted (1 + 99), discharging each time the aqueous phases; add then 10 ml of hydrochloric acid solution (ϱ approximately 1,19 g/ml) diluted (1 + 99) and reduce with 0,2 ml of tin(II) chloride solution [2 % $SnCl_2.2H_2O$ (R 38) in the hydrochloric acid (R 13)].

The blue colour of the sample solution shall not be more intense than that of the reference solution.

R 65.2.5 Sulfate

Dissolve 1 g of the sample in 5 ml of hot water, add 5 ml of the nitric acid (R 19) and evaporate to dryness. Digest the residue with 1 ml of 10 % hydrochloric acid solution and 10 ml of water, dilute to 40 ml and filter.

Take 20 ml of the filtrate and apply GM 3.

Prepare a standard matching solution, using 1 ml of the sulfate SS I(1 ml $\triangleq$ 0,02 % SO_4).

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[12] IUPAC name: disodium 4-(2-pyridinylazo)benzene-1,3-diolate.

R 65.2.6 Lead

Dissolve 1 g of the sample in 50 ml of water, add 10 ml of the ammonia solution (R 3), 2 ml of ammonium citrate solution (40 %) and 2 ml of potassium cyanide solution (10 %), adjust the pH to 9 to 9,5 and extract with two 5 ml portions of dithizone[13] solution (0,002 % in carbon tetrachloride).

Compare the resulting red colour with that of a standard matching solution obtained using 1 ml of the lead SS II, equally treated (1 ml $\triangleq$ 0,001 % Pb).

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[13] IUPAC name: 1,5-diphenylthiocarbazone.

R 66 Hydrobromic acid HBr

Relative molecular mass: 80,92

R 66.1 Specification

Assay (HBr)	47 to 49 %[a]
Chloride (Cl)	0,03 % max.
Iodide (I)	To pass test (about 0,002 %)
Phosphate (PO_4)	0,000 5 % max.
Sulfate and sulfite (expressed as SO_4)	0,003 % max.
Arsenic (As)	0,000 2 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 2 % max.
Sulfated ash	0,005 % max.

[a] 40 % and 50 % hydrobromic acid conforming otherwise to the above specification are also commercially available.

R 66.2 Preparation of test solution

To 50 g (34 ml) of the sample add dropwise just enough saturated bromine water to obtain a persistent yellow coloration and then 1 ml of sodium carbonate (R 30) solution (1 %). Evaporate to dryness on a water bath, dissolve the residue in 1 ml of water and 0,1 ml of hydrochloric acid solution (25 %) by gently heating and then dilute to 50 ml with water.

R 66.3 Tests

R 66.3.1 Assay

Seal a thin-walled glass ampoule containing about 0,3 g of the sample, weighed to the nearest 0,000 1 g, and transfer it to a 500 ml conical flask containing 200 ml of water. Close the flask by means of a ground glass stopper. Break the ampoule by shaking vigorously and after 5 min lift and rinse the stopper with water. Titrate with standard volumetric sodium hydroxide solution, $c(NaOH) = 1$ mol/l, using the methyl orange (IS 4.3.5*)[14].

1,00 ml of sodium hydroxide solution, $c(NaOH) = 1{,}000$ mol/l, corresponds to 0,080 92 g of HBr.

R 66.3.2 Chloride

Transfer 1 g (0,68 ml) of the sample to a round-bottomed flask containing small glass beads and a mixture of 35 ml of water and 15 ml of the nitric acid (R 19). Heat slowly just to boiling and pass a stream of nitrogen through the solution until it is completely decolorized. The volume of the solution should not be reduced to less than 40 ml. After cooling dilute to 100 ml.

To 20 ml of this solution add 1 ml of standard volumetric silver nitrate solution, $c(AgNO_3) = 0{,}1$ mol/l.

Compare the turbidity obtained with that of a similarly prepared standard matching solution, using 3 ml of the chloride SS I (3 ml $\triangleq$ 0,03 % Cl).

R 66.3.3 Iodide

Dilute 4 g (2,7 ml) of the sample with 20 ml of water, add 0,2 ml of a 10 % solution of iron(III) chloride and 5 ml of chloroform (R 7). Allow to stand for 5 min and then shake for 1 min.

The chloroform layer shall not show any violet colour.

R 66.3.4 Phosphate

Take 5 ml of the test solution (R 66.2), dilute to 20 ml with 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).

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[14] IUPAC name: sodium 4-[4(dimethylamino)phenylazo]benzenesulfonate.

R 66.3.5 Sulfate and sulfite

Take 5 ml of the test solution (R 66.2), dilute to 20 ml with water and apply GM 3.

Prepare a standard matching solution, using 1,5 ml of the sulfate SS I (1,5 ml $\triangleq$ 0,003 % SO_4).

R 66.3.6 Arsenic

Dilute 1 g (0,68 ml) of the sample to 40 ml with water and apply GM 11.

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

R 66.3.7 Heavy metals

Take 4 ml of the test solution (R 66.2), dilute to 20 ml with water and apply GM 7.

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

R 66.3.8 Iron

Take 10 ml of the test solution (R 66.2) and apply GM 8.1.

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

R 66.3.9 Sulfated ash

Take 40 g (27 ml) of the sample and apply GM 17.

The mass of the residue shall not exceed 2 mg.

R 67 Hydrofluoric acid HF

Relative molecular mass: 20,0

R 67.1 Specification

Assay (HF)	40 to 42 %[a]
Chloride (Cl)	0,001 % max.
Hexafluorosilicate (SiF_6)	0,05 % max.
Phosphate (PO_4)	0,000 2 % max.
Sulfate and sulfite (expressed as SO_4)	0,002 % max.
Heavy metals (expressed as Pb)	0,000 1 % max.
Iron (Fe)	0,000 1 % max.
Sulfated ash	0,002 % max.

[a] 48 % hydrofluoric acid conforming otherwise to the above specification is also commercially available.

R 67.2 Preparation of test solution

Transfer 50 g (44,2 ml) of the sample to a platinum dish and add 2 ml of sodium carbonate (R 30) solution (1 %) and 1 ml of hydrogen peroxide solution (30 %). Evaporate to dryness on a water-bath and dissolve the residue in 1 ml of water and 0,2 ml of hydrochloric acid solution (25 %) by gently heating and dilute to 50 ml with water.

R 67.3 Tests

R 67.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 2 g of the sample into a platinum crucible containing 5 ml of water and cover with a lid. Rinse with about 50 ml of water into a platinum dish or into a beaker made of plastic material. 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 0,020 01 g of HF.

R 67.3.2 Chloride

Dilute 2 g (1,8 ml) of the sample to 20 ml with water and apply GM 2.

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

R 67.3.3 Hexafluorosilicate

To 0,5 g (0,45 ml) of the sample in a platinum dish add 0,5 g of boric acid, 5 ml of water and 1 g of the sodium carbonate (R 30). When reaction ceases, dissolve completely heating gently. Dilute to 20 ml with water and apply GM 5.

Prepare a standard matching solution under the same conditions, using 2,5 ml of fluorosilicate SS I (2,5 ml ≙ 0,05 % SiF_6).

R 67.3.4 Phosphate

Take 20 ml of the test solution (R 67.2) and apply GM 4.

Prepare a standard matching solution, using 4 ml of the phosphate SS II (4 ml ≙ 0,000 2 % PO_4).

67.3.5 Sulfate and sulfite

Take 5 ml of the test solution (R 67.2), dilute to 10 ml with water and apply GM 3.

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

R 67.3.6 Heavy metals

Take 10 ml of the test solution (R 67.2) and apply GM 7.

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

R 67.3.7 Iron

Take 10 ml of the test solution (R 67.2) 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 67.3.8 Sulfated ash

Transfer 50 g (44,2 mml) of the sample to a weighed platinum dish and apply GM 17.

The mass of the residue shall not exceed 1 mg.

R 68 Iodine I_2

Relative atomic mass: 253,8

R 68.1 Specification

Assay (I_2)	99,8 % min.
Chlorine and bromine (expressed as Cl)	0,005 % max.
Residue after evaporation	0,005 % max.

R 68.2 Preparation of test solution

Dissolve 1 g of the sample in 5 ml of standard volumetric sodium hydroxide solution, $c(NaOH) = 2$ mol/l (the solution shall be clear and colourless).

R 68.3 Tests

R 68.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,5 g of the sample and dissolve in a solution of 2 g of potassium iodide in 5 ml of water. Dilute to 50 ml with water, add 5 ml of sulfuric acid solution (16 %) and titrate 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 0,012 69 g of I.

R 68.3.2 Chlorine and bromine

Dissolve 0,2 g of the sample in 20 ml of hot hydrazinium sulfate solution (0,6 %) and neutralize with standard volumetric sodium hydroxide solution, $c(NaOH) = 1$ mol/l. After adding 0,5 ml of the hydrogen peroxide solution (R 14) and 0,2 ml of the phosphoric acid (R 22) remove the iodine by heating. Cool, 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 $\triangleq$ 0,005 % Cl).

R 68.3.3 Residue after evaporation

Take 20 g of the sample and apply GM 14.

The mass of the residue shall not exceed 1 mg.

R 69 Iron(II) sulfate heptahydrate $FeSO_4.7H_2O$

Relative molecular mass: 278,02

R 69.1 Specification

Assay ($FeSO_4.7H_2O$)	99 % min.
Total nitrogen (N)	0,001 % max.
Chloride (Cl)	0,001 % max.
Phosphate (PO_4)	0,001 % max.
Arsenic (As)	0,000 2 % max.
Copper (Cu)	0,002 5 % max.
Iron(III) (Fe)	0,025 % max.
Lead (Pb)	0,002 5 % max.
Manganese (Mn)	0,05 % max.
Zinc (Zn)	0,005 % max.
Substances not precipitated by ammonia	0,1 % max.

R 69.2 Preparation of test solutions

R 69.2.1 Test solution I

Dissolve 15 g of the sample in about 100 ml of water, add 1 ml of sulfuric acid solution (20 %) and dilute to 150 ml with water.

R 69.2.2 Test solution II

Dissolve 2 g of the sample in 10 ml of water, add 3 ml of the nitric acid (R 19), heat for 1 to 2 min, allow to cool and add 15 ml of the hydrochloric acid (R 13). Transfer this solution to a 100 ml separating funnel, add 40 ml of 4-methyl-2-pentanone and shake vigorously for 1 min. Separate the aqueous phase, concentrate to about 3 ml and dilute to 20 ml with water.

R 69.3 Tests

R 69.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 1 g of the sample, dissolve in a cooled solution containing 100 ml of water, 10 ml of the sulfuric acid (R 37) and 5 ml of the phosphoric acid (R 22) and titrate immediately with standard volumetric potassium permanganate solution, $c(1/5\ KMnO_4) = 0,1$ mol/l.

1,00 ml of potassium permanganate solution, $c(1/5\ KMnO_4 = 0,100$ mol/l, corresponds to 0,027 8 g of $FeSO_4.7H_2O$.

R 69.3.2 Total nitrogen

Take 20 ml of the test solution I (R 69.2.1) and apply GM 6.

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

R 69.3.3 Chloride

Take 10 ml of the test solution I (R 69.2.1) and apply GM 2.

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

R 69.3.4 Phosphate

Take 10 ml of the test solution I (R 69.2.1), add 1 ml of the nitric acid (R 19) and apply GM 4.

Prepare a standard matching solution, using 1 ml of the phosphate SS II (1 ml $\triangleq$ 0,001 % PO_4).

R 69.3.5 Arsenic

Take 10 ml of the test solution I (R 69.2.1) and apply GM 11.

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

R 69.3.6 Copper, lead and zinc

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

Element	Concentration of solution	Flame	Resonance line nm
Cu	Test solution II (R 69.2.2)	Air-ethine	324,7
Pb			217,0 or 283,3
Zn			213,9

R 69.3.7 Iron(III)

Quickly dissolve 0,5 g of the sample in 20 ml of cooled water, add 1 ml of the hydrochloric acid (R 13) and 5 ml of potassium thiocyanate solution (10 %).

Simultaneously prepare a standard matching solution under the same conditions, using 12,5 ml of the iron SS II (12,5 ml $\triangleq$ 0,025 % Fe).

R 69.3.8 Manganese

Take 10 ml of the test solution I (R 69.2.1), add 40 ml of water, 5 ml of the nitric acid (R 19), 5 ml of the phosphoric acid (R 22) and 1 g of potassium periodate. Heat until the evolution of vapours of iodine ceases, keep boiling for about 2 min and leave on a hot water-bath for 30 min. Cool and dilute to 100 ml with water.

Prepare a standard matching solution under the same conditions, using 5 ml of the manganese SS I (5 ml $\triangleq$ 0,05 % Mn).

R 69.3.9 Substances not precipitated by ammonia

To 50 ml of the test solution I (R 69.2.1) add 5 ml of the nitric acid (R 19), boil for 10 min and precipitate the iron by slowly adding 50 ml of ammonia solution (6,8 %) in a fume-cupboard. Keep on a boiling water-bath for 5 min and dilute to 150 ml with water.

Filter and evaporate 60 ml of the filtrate in a weighed dish. Ignite the residue at 600 ± 50 °C to constant mass.

The mass of the residue shall not exceed 2 mg.

R 70 L-Ascorbic acid $C_6H_8O_6$

Relative molecular mass: 176,13

R 70.1 Specification

Assay ($C_6H_8O_6$)	99,7 % min.
Specific optical rotation	$[\alpha]_{D}^{20}$: + 20,5° to + 21,5°
Chloride (Cl)	0,005 % max.
Sulfate (SO_4)	0,002 % max.
Heavy metals (expressed as Pb)	0,001 % max.
Iron (Fe)	0,000 2 % max.
Loss after drying (at 105 °C)	0,1 % max.
Sulfated ash	0,05 % max.

R 70.2 Preparation of test solution

Dissolve 10 g of the sample in water and dilute to 100 ml (the solution shall be clear and colourless).

R 70.3 Tests

R 70.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,4 g of the sample, dissolve in 80 ml of water, add 2 ml of sulfuric acid solution (25 %) and 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 0,008 806 g of $C_6H_8O_6$.

R 70.3.2 Specific optical rotation

Apply GM 26, using the following conditions:

Solvent	Water
Concentration	c = 10 g/100 ml of solution
Tube length	l = 10 cm

R 70.3.3 Chloride

Heat 10 ml of the test solution (R 70.2) with 5 ml of the nitric acid (R 19) for 1 min, cool and apply GM 2, without addition of 1 ml of nitric acid solution (25 %).

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

R 70.3.4 Sulfate

Take 10 ml of the test solution (R 70.2) and apply GM 3.

Prepare a standard matching solution, using 2 ml of the sulfate SS II (2 ml ≙ 0,002 % SO_4).

R 70.3.5 Heavy metals

To 10 ml of the test solution (R 70.2), add 1 g of sodium acetate anhydrous and apply GM 7.

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

R 70.3.6 Iron

Take 10 ml of the test solution (R 70.2) and apply GM 8.1.

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

R 70.3.7 Loss after drying

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

The loss in mass shall not exceed 2 mg.

R 70.3.8 Sulfated ash

Take 2 g of the sample and apply GM 16.

The mass of the residue shall not exceed 1 mg.

R 71 Lead(II) acetate trihydrate $Pb(CH_3COO)_2.3H_2O$

Relative molecular mass: 379,33

R 71.1 Specification

Assay [$Pb(CH_3COO)_2.3H_2O$]	99,5 % min.
Chloride (Cl)	0,000 5 % max.
Nitrate (NO_3)	0,002 % max.
Calcium (Ca)	0,005 % max.
Copper (Cu)	0,001 % max.
Iron (Fe)	0,001 % max.
Potassium (K)	0,005 % max.
Sodium (Na)	0,005 % max.

R 71.2 Preparation of test solution

Dissolve 20 g of the sample in carbon dioxide-free water containing 0,2 ml of glacial acetic acid and dilute to 200 ml with the same water (the solution shall be clear and colourless).

R 71.3 Tests

R 71.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,6 g of the sample and dissolve in 100 ml of water.

To this solution add 20 ml of potassium sodium tartrate solution (28,2 %) and 50 mg of the mordant black 11 mixture (4.3.8*)[15]. After addition of 2 ml of the ammonia solution (R 3), titrate at 40 °C with standard volumetric EDTA solution, *c*(EDTA) = 0,1 mol/l, to a blue colour.

1,00 ml of EDTA solution, *c*(EDTA) = 0,100 mol/l, corresponds to 0,037 933 g of $(CH_3COO)_2Pb.3H_2O$.

R 71.3.2 Chloride

Take 10 ml of the test solution (R 71.2) and apply GM 2.

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

R 71.3.3 Nitrate

In a Kjeldahl apparatus dilute 20 ml of the test solution (R 71.2) to 140 ml with water and add 10 ml of sodium hydroxide solution (27 %). Remove ammonia by heating, add 1,0 g of Devarda's alloy and then apply GM 6 beginning with "Allow to stand for 1 h".

Prepare a standard matching solution, using 4 ml of the nitrate SS II (4 ml $\triangleq$ 0,002 % NO_3).

R 71.3.4 Calcium and copper

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	4 %	Air-ethine	422,7
Cu			324,7

R 71.3.5 Iron

Mix 10 ml of the test solution (R 71.2) with 2,5 ml of the sulfuric acid (R 37), filter and wash the precipitate with 10 ml of water. Add to the filtrate 2 ml of 5-sulfosalicylic acid solution (20 %) and 10 ml of the ammonia solution (R 3).

After 10 min, the yellow coloration of the resulting solution shall not be more intense than that of a similarly prepared standard matching solution, using 1 ml of the iron SS II (1 ml $\triangleq$ 0,001 % Fe).

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[15] IUPAC name: sodium 3-hydroxy-4-(1-hydroxy-2-naphthylazo)-7-nitronaphthalene-1-sulfonate.

R 71.3.6 Potassium and sodium

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

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

R 72 Mercury(II) chloride $HgCl_2$

Relative molecular mass: 271,50

R 72.1 Specification

Assay ($HgCl_2$)	99,5 % min.
Water-insoluble matter	0,01 % max.
Mercury(I) chloride (Hg_2Cl_2)	0,05 % max.
Total nitrogen (N)	0,002 % max.
Iron (Fe)	0,002 % max.
Lead (Pb)	0,001 % max.
Residue after reduction	0,02 % max.

R 72.2 Tests

R 72.2.1 Assay

Weigh, to the nearest 0,000 1 g, about 1 g of the sample and dissolve in 100 ml of water, containing 10 g of the sodium chloride (R 32).

To this solution add 45,0 ml of standard volumetric EDTA solution, $c(\text{EDTA}) = 0{,}1$ mol/l, and 10 ml of buffer solution (RS)[16] and back titrate the excess EDTA with standard volumetric magnesium chloride solution, $c(1/2\ MgCl_2) = 0{,}1$ mol/l, until the colour just changes to violet, using the mordant black 11 mixture (4.3.8*).

1,00 ml of EDTA solution, $c(\text{EDTA}) = 0{,}100$ mol/l, corresponds to 0,027 15 g of $HgCl_2$.

R 72.2.2 Water-insoluble matter

Take 10 g of the sample and apply GM 1.

The mass of the residue shall not exceed 1 mg.

R 72.2.3 Mercury(I) chloride

Dissolve 2 g of the sample in 50 ml of the ethanol (R 11). Filter through a filter crucible and carefully wash the residue on the filter with two 10 ml portions of the same ethanol. Dry at 50 °C and weigh.

The mass of the residue shall not exceed 1 mg.

R 72.2.4 Total nitrogen

Dissolve 2,5 g of the sample in 50 ml of water, add 1 ml of the hydrochloric acid (R 13) and 5 g of coarse zinc powder. Heat and shake the mixture for about 10 min until the solution becomes clear. Dilute to 200 ml and filter. Take 100 ml of the filtrate and apply GM 6.

Prepare a standard matching solution, using 2,5 ml of the nitrogen SS II (2,5 ml $\triangleq$ 0,002 % N).

R 72.2.5 Iron

Dissolve 2 g of the sample by gently warming with 20 ml of water. Transfer 5 ml of this solution to a suitable separating funnel, add 10 ml of acidified ammonium nitrate solution (RS)[16] and 5 ml of thenoyltrifluoroacetone[17] solution (15 % in xylene). Shake for 30 min, allow to separate and decant the organic layer.

The colour of the organic phase shall not be more intense than that of a similarly prepared standard matching solution, using 1 ml of the iron SS II (1 ml $\triangleq$ 0,002 % Fe).

R 72.2.6 Lead

Evaporate the solution after reduction (as described in R 2.7) to dryness and dissolve the residue in 1 ml of the nitric acid (R 19), add 4 ml of water and then dilute to 10,0 ml.

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[16] **Reagents solutions (RS)**

a) **Ammonia standard buffer.**

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

b) **Acidified ammonium nitrate solution.**

Dissolve 720 g of the ammonium nitrate (R 45) in 400 ml of water, add 128 ml of nitric acid solution (70 %) and dilute to 1 000 ml with water.

[17] IUPAC name: 4,4,4-trifluoro-1-(2-thienyl)butane-1,3-dione.

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

Element	Concentration of solution	Flame	Resonance line nm
Pb	Solution prepared as described above	Air-ethine	217,0 or 283,3

R 72.2.7 Residue after reduction

Dissolve 10,0 g in 25 ml of water plus 25 ml of the ammonia solution (R 3). Add 75 ml of formic acid (98 %) and reflux until all the mercury is reduced to metal. Cool, filter through a well-washed filter paper, and wash with a small quantity of water. Dilute the combined filtrate and washings to 150 ml and store 75 ml of this solution for determination of lead (R 72.2.6).

To the other part (75 ml) add 1 ml of the sulfuric acid (R 37) and evaporate in a tared dish under a well-ventilated hood. Continue heating until the excess sulfuric acid has been volatilized. Finally, ignite at 800 ± 25 °C for 15 min. Correct for the mass obtained in complete blank test.

The mass of the residue shall not exceed 1 mg.

R 73 Petroleum spirit 40/60

R 73.1 Specification

Colour in Hazen units	10 max.
Density at 20 °C	0,640 to 0,655 g/ml
Distillation yield	Not less than 90 % distils between 40 and 60 °C
Residue after evaporation	0,001 % max.
Benzene (C_6H_6)	0,025 % max.
Sulfur compounds (expressed as S)	0,005 % max.
Iron (Fe)	0,000 1 % max.
Lead (Pb)	0,000 1 % max.
Water (H_2O)	0,015 % max.

R 73.2 Tests

R 73.2.1 Colour in Hazen units

Apply GM 36.

R 73.2.2 Density

Apply GM 24.1.

R 73.2.3 Distillation interval

Apply GM 25.1.

R 73.2.4 Residue after evaporation

Take 50 g (77,5 ml) of the sample and apply GM 14.

The mass of the residue shall not exceed 0,5 mg.

R 73.2.5 Benzene

Measure the absorbance of the sample in a cell of optical path length 1 cm at 255 nm against water as the reference.

The benzene content, expressed as percentage by mass of C_6H_6, is given by the formula

$$\frac{A}{17{,}6}$$

where A is the absorbance of the sample.

R 73.2.6 Sulfur compounds

Take 1 g (1,5 ml) of the sample and apply GM 21.

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

R 73.2.7 Iron and lead

Evaporate 13 g (20 ml) of the sample to dryness with 5 mg of the sodium carbonate (R 30) and dissolve the residue in 10 ml of water containing 0,5 ml of the nitric acid (R 19).

Determine the iron and lead by AAS according to GM 29, using the following conditions:

Element	Concentration of solution	Flame	Resonance line nm
Fe	Solution prepared as described above	Air-ethine	248,3
Pb			217,0 or 283,3

R 73.2.8 Water

Take 19,5 g (30 ml) of the sample, add 5 ml of *N*-ethyl-piperidine as a catalyst and apply GM 12, using a mixture of 20 ml of the methanol (R 18) and 10 ml of the chloroform (R 7) as the solvent.

R 74 Phosphorus(V) oxide P_2O_5

Relative molecular mass: 141,94

R 74.1 Specification

Assay (P_2O_5)	98 % min.
Water-insoluble matter	0,02 % max.
Total nitrogen(N)	0,002 % max.
Heavy metals (expressed as Pb)	0,005 % max.
Permanganate-reducing substances (expressed as P_2O_3)	0,02 % max.

R 74.2 Preparation of test solution

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

R 74.3 Tests

R 74.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 1 g of the sample, dissolve cautiously in a beaker containing 100 ml of water and evaporate to about 25 ml. Cool, dilute to 120 ml and titrate with standard volumetric sodium hydroxide solution, *c*(NaOH) = 1 mol/l, using the thymolphthalein (IS 4.3.12*)[18].

1,00 ml of sodium hydroxide solution, *c*(NaOH) = 1,000 mol/l, corresponds to 0,035 49 g of P_2O_5.

R 74.3.2 Water-insoluble matter

Take 10 g of the sample and apply GM 1.

The mass of the residue shall not exceed 2 mg.

NOTE Add the sample with care to the water maintained at about boiling temperature.

R 74.3.3 Total nitrogen

Take 10 ml of the test solution (R 74.2) and apply GM 6.

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

R 74.3.4 Heavy metals

Dilute 10 ml of the test solution (R 74.2) to about 20 ml with water and neutralize with the ammonia solution (R 3) to a pink colour, using 2 drops of the phenolphthalein (IS 4.3.9*). Adjust to a pH-value between 3 and 4 by adding the acetic acid (R 1) and dilute to 50 ml with water (solution A).

Take 30 ml of the solution A and apply GM 7.

Prepare a standard matching solution by adding 10 ml of the solution A to 2 ml of the lead SS II (2 ml ≙ 0,005 % Pb).

R 74.3.5 Permanganate-reducing substances

Dissolve cautiously 10 ± 0,5 g of the sample in 100 ml of water, add 10 ml of sodium hydroxide (R 34) solution (30 %) followed by sodium hydrogen carbonate until effervescence ceases and then add 5,0 ml of standard volumetric potassium permanganate solution, *c*1/5 $KMnO_4$) = 0,1 mol/l. Boil for 5 min, cool and add 10 ml of potassium iodide solution (20 %) and 20 ml of sulfuric acid solution (20 %). Titrate with standard volumetric sodium thiosulfate solution, *c*($Na_2S_2O_3$) = 0,1 mol/l, using the starch (IS 4.3.11*).

Carry out a blank determination replacing the test portion by 7 ml of the sulfuric acid (R 37).

1,00 ml of sodium thiosulfate solution, *c*($Na_2S_2O_3$) = 0,100 mol/l, corresponds to 0,002 75 g of P_2O_3.

The difference between the volumes of sodium thiosulfate solution consumed shall not exceed 0,75 ml.

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[18] IUPAC name: 3,3-bis(4-hydroxy-5-isopropyl-2-methylphenyl)phthalide.

R 75 Potassium bromate $KBrO_3$

Relative molecular mass: 167,01

R 75.1 Specification

Assay ($KBrO_3$)	99,7 % min.
pH (5 % solution)	5 to 9
Bromide (Br)	0,02 % max.
Sulfate (SO_4)	0,005 % max.
Total nitrogen (N)	0,001 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Sodium (Na)	0,01 % max.

R 75.2 Preparation of test solutions

R 75.2.1 Test solution I

Dissolve 10 g of the sample in water and dilute to 200 ml (the solution shall be clear and colourless).

R 75.2.2 Test solution II

Evaporate 2 g of the sample twice with 10 ml of the hydrochloric acid (R 13). Dissolve the residue in 1 ml of the hydrochloric acid (R 13) and 20 ml of water, neutralize with ammonia solution and dilute to 30 ml with water.

R 75.3 Tests

R 75.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,1 g of the sample and dissolve in 50 ml of water. To this solution add 2 g of potassium iodide and 5 ml of sulfuric acid solution (16 %). After 10 min, 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 0,002 783 5 g of $KBrO_3$.

R 75.3.2 pH

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

R 75.3.3 Bromide

Dissolve 5 g of the sample in 60 ml of water and add 0,5 ml of sulfuric acid solution (4,9 %).

After 1 h, the yellow coloration shall not be more intense than that of a similarly prepared standard matching solution, using 2 g of the sample and 6 ml of the bromide SS I (6 ml $\triangleq$ 0,02 % Br).

R 75.3.4 Sulfate

Take 15 ml of the test solution II (R 75.2.2) and apply GM 3.

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

R 75.3.5 Total nitrogen

Take 20 ml of the test solution I (R 75.2.1), dilute to 140 ml with water and apply GM 6.

Prepare a standard matching solution, using 1 ml of the nitrogen SS II (1 ml $\triangleq$ 0,001 % N).

R 75.3.6 Heavy metals

Take 15 ml of the test solution II (R 75.2.2) and apply GM 7.

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

R 75.3.7 Iron

Mix 20 ml of the test solution I (R 75.2.1) with 2 ml of 5-sulfosalicylic acid solution (10 %) and 3 ml of the ammonia solution (R 3).

After 10 min, the yellow coloration shall not be more intense than that of a similarly prepared standard matching solution, using 0,5 ml of the iron SS II (0,5 ml $\triangleq$ 0,000 5 % Fe).

R 75.3.8 Sodium

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

Element	Concentration of solution	Flame	Wavelength nm
Na	4 %	Air-ethine	589,0

R 76 Potassium carbonate K_2CO_3

Relative molecular mass: 138,21

R 76.1 Specification

Assay (K_2CO_3, after drying at 300 °C)	99,0 % min.
Water-insoluble matter	0,005 % max.
Chloride (Cl)	0,003 % max.
Phosphate and silicate (expressed as SiO_2)	0,005 % max.
Total sulfur (expressed as SO_4)	0,003 % max.
Total nitrogen (N)	0,001 % max.
Calcium (Ca)	0,002 % max.
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,000 5 % max.
Magnesium (Mg)	0,002 % max.
Sodium (Na)	0,05 % max.
Loss after drying (at 300 °C)	1,0 % max.

R 76.2 Tests

R 76.2.1 Assay

Weigh, to the nearest 0,000 1 g, about 2,5 g of the sample, previously dried at 300 °C, dissolve in 50 ml of water and titrate with standard volumetric hydrochloric acid solution, $c(\mathrm{HCl}) = 1$ mol/l, using the bromophenol blue (IS 4.3.2*)[19].

1,00 ml of hydrochloric acid solution, $c(\mathrm{HCl}) = 1{,}00$ mol/l, corresponds to 0,069 11 g of K_2CO_3.

R 76.2.2 Water-insoluble matter

Take 50 g of the sample and apply GM 1.

The mass of the residue shall not exceed 2,5 mg.

R 76.2.3 Chloride

Dissolve 1 g of the sample in a mixture of 45 ml of water and 5 ml of nitric acid solution (25 %) and apply GM 2.

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

R 76.2.4 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, cool, add 5 ml of hexaammonium heptamolybdate solution (10 %), adjust the pH to 1,8 and heat to boiling again. Cool, 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 % $SnCl_2.2H_2O$) (R 38) in the hydrochloric acid (R 13)]. Dry the organic phase with anhydrous sodium sulfate (R 35).

The blue colour 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 76.2.5 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 with water and apply GM 3.

Prepare a standard matching solution, using 3 ml of the sulfate SS II (3 ml ≙ 0,003 % SO_4).

R 76.2.6 Total nitrogen

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

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[19] IUPAC name: 2,2′,6,6′-tetrabromo-4,4′-(1,1-dioxo-2,1λ⁶-benzoxathiole-3,3-diyl)diphenol.

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

R 76.2.7 Calcium, copper, 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	5 % solution of the sample in hydrochloric acid solution, c(HCl) = 0,5 mol/l	Air-ethine	422,7
Cu	Dissolve 12 g of the sample in 100 ml of water, add 30 ml of hydrochloric acid solution (25 %), boil, cool and dilute to 150 ml with water. Apply solvent extraction procedure according to GM 35		324,7
Fe			248,3
Pb			217,0 or 283,3
Mg	1 % solution of the sample in hydrochloric acid solution, c(HCl) = 0,5 mol/l		285,2

R 76.2.8 Sodium

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

Element	Concentration of solution	Flame	Wavelength nm
Na	1 % solution of the sample in hydrochloric acid solution, c(HCl) = 0,5 mol/l	Air-ethine	589,0

R 76.2.9 Loss after drying

Dry 3 g of the sample to constant mass at 300 °C.

The loss in mass shall not exceed 30 mg.

R 77 Potassium chloride KCl

Relative molecular mass: 74,55

R 77.1 Specification

Assay (KCl)	99,5 % min.
pH (5 % solution)	5 to 8
Bromide (Br)	0,01 % max.
Iodide (I)	0,002 % max.
Phosphate (PO_4)	0,000 5 % max.
Sulfate (SO_4)	0,002 % max.
Total nitrogen (N)	0,001 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 2 % max.
Barium (Ba)	0,001 % max.
Calcium (Ca)	0,002 % max.
Magnesium (Mg)	0,002 % max.
Sodium (Na)	0,02 % max.

R 77.2 Preparation of test solution

Dissolve 20 g of the sample in 150 ml of water (the solution shall be clear and colourless) and dilute to 200 ml.

R 77.3 Tests

R 77.3.1 Assay

Weigh, to the nearest 0,000 1 g, 0,3 g of the sample and dissolve in a mixture of 50 ml of water and 10 ml of the nitric acid (R 19), in a ground glass stoppered vessel.

To this solution add 50 ml of standard volumetric silver nitrate solution, $c(AgNO_3)$ = 0,1 mol/l, and 3 ml of nitrobenzene, and shake vigorously for 1 min. Titrate the excess silver nitrate with standard volumetric ammonium thiocyanate solution, $c(NH_4SCN)$ = 0,1 mol/l, using the ammonium iron(III) sulfate (IS 4.3.1*).

1,00 ml of silver nitrate solution, $c(AgNO_3)$ = 0,100 mol/l, corresponds to 0,007 455 g of KCl.

R 77.3.2 pH

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

R 77.3.3 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 $\triangleq$ 0,01 % Br), and 2 ml of the iodide SS I (2 ml $\triangleq$ 0,002 % I), in another separating funnel.

To both sample solution and standard matching solution, add 10 ml of the carbon tetrachloride (R 54) 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 77.3.4.

R 77.3.4 Bromide

Reject the lower phases of the solutions obtained in R 77.3.3 and wash the aqueous solutions twice with 10 ml of the carbon tetrachloride (R 54). 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-brown coloration in the sample lower phase shall not be more intense than that in the standard matching lower phase.

R 77.3.5 Phosphate

Take 50 ml of the test solution (R 77.2), 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 77.3.6 Sulfate

Take 20 ml of the test solution (R 77.2) and apply GM 3.

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

R 77.3.7 Total nitrogen

Take 20 ml of the test solution (R 77.2) and apply GM 6.

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

R 77.3.8 Heavy metals

Take 40 ml of the test solution (R 77.2) and apply GM 7.

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

R 77.3.9 Iron

Take 20 ml of the test solution (R 77.2) and apply GM 8.1.

Prepare a standard matching solution, using 4 ml of the iron SS III (4 ml ≙ 0,00 2 % Fe).

R 77.3.10 Calcium, magnesium and barium

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	4 %	Air-ethine	422,7
Mg			285,2
Ba	1 %	Dinitrogen oxide-ethine	553,5

R 77.3.11 Sodium

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

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

R 78 Potassium cyanide KCN

Relative molecular mass: 65,12

R 78.1 Specification

Assay (KCN)	97,0 % min.
Chloride (Cl)	0,1 % max.
Phosphate (PO_4)	0,01 % max.
Sulfate (SO_4)	0,01 % max.
Sulfide (S)	0,001 % max.
Thiocyanate (SCN)	0,01 % max.
Iron (Fe)	0,01 % max.
Lead (Pb)	0,000 5 % max.
Sodium (Na)	0,5 % max.

R 78.2 Tests

R 78.2.1 Assay

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

To this solution add 5 ml of ammonia solution (10 %), 1 drop of potassium iodide solution (15 %) and titrate with standard volumetric silver nitrate solution, $c(AgNO_3) = 0{,}1$ mol/l, until a trace of turbidity persists.

1,00 ml of silver nitrate solution, $c(AgNO_3) = 0{,}100$ mol/l, corresponds to 0,013 02 g of KCN.

R 78.2.2 Chloride

Dissolve 1 g of the sample in 50 ml of water, add 25 ml of the formaldehyde solution (R 63), 5 ml of the nitric acid (R 19) and 5,0 ml of standard volumetric silver nitrate solution, $c(AgNO_3) = 0{,}1$ mol/l. Filter, wash with water and titrate the combined filtrate and washings with standard volumetric ammonium thiocyanate solution, $c(NH_4SCN) = 0{,}1$ mol/l, using the ammonium iron(III) sulfate (IS 4.3.1*).

The volume of the standard volumetric ammonium thiocyanate solution, $c(NH_4SCN) = 0{,}1$ mol/l, shall not be less than 4,70 ml.

R 78.2.3 Phosphate

Dissolve 0,1 g of the sample in 5 ml of water, add 1 ml of the hydrochloric acid (R 13) and evaporate to dryness in a fume cupboard. Add 2,5 ml of the hydrochloric acid (R 13) and again evaporate to dryness. Dissolve the residue in 20 ml of water and apply GM 4.

Prepare a standard matching solution, using 1 ml of the phosphate SS II (1 ml $\triangleq$ 0,01 % PO_4).

R 78.2.4 Sulfate

Dissolve 1 g of the sample in 15 ml of water, add 2 ml of the hydrochloric acid (R 13) and boil gently in a fume cupboard for 5 min. Cool, neutralize with sodium hydroxide solution (20 %), dilute to 20 ml with water and apply GM 3.

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

R 78.2.5 Sulfide

Dissolve 1 g of the sample in 20 ml of water, add 5 ml of ammonia solution (10 %) and 0,5 ml of the lead acetate, basic (RS 4.2.10*).

Compare any darkening with that produced by the similar treatment of 1 ml of sulfide SS II prepared by diluting freshly prepared sulfide SS I (1 ml $\triangleq$ 0,001 % S).

R 78.2.6 Thiocyanate

Dissolve 2 g of the sample in 20 ml of water, add 4 ml of the hydrochloric acid (R 13) and 1 drop of the iron(III) chloride (RS 4.2.8*).

No red colour shall be apparent when compared with a mixture of 20 ml of water, 4 ml of the hydrochloric acid (R 13) and 1 drop of the iron(III) chloride RS.

R 78.2.7 Iron and lead

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

Element	Concentration of solution	Flame	Resonance line nm
Fe	1 %		248,3
Pb	Dissolve 15 g of the sample in 150 ml of water and apply solvent extraction procedure according to GM 35	Air-ethine	217,0 or 283,3

WARNING — As a safety precaution prepare the test solution for GM 29 and GM 35 in a fume cupboard with a sufficient draught and pass the effluent from the spray chamber of the spectrometer as well as any unused remainders of the test solution into an alkaline hypochlorite solution in order to destroy any hydrogen cyanide.

R 78.2.8 Sodium

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

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

R 79 Potassium dihydrogen phosphate KH_2PO_4

Relative molecular mass: 136,09

R 79.1 Specification

Assay (KH_2PO_4, after drying at 110 °C)	99,5 % min.
pH (5 % solution)	4,2 to 4,5
Chloride (Cl)	0,000 5 % max.
Sulfate (SO_4)	0,005 % max.
Total nitrogen (N)	0,001 % max.
Heavy metals (expressed as Pb)	0,001 % max.
Iron (Fe)	0,002 % max.
Sodium (Na)	0,02 % max.
Loss after drying (at 110 °C)	0,2 % max.

R 79.2 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 79.3 Tests

R 79.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 5 g of the sample dried at 110 °C, dissolve in 95 ml of freshly boiled and cooled water and titrate potentiometrically with standard volumetric sodium hydroxide solution, c(NaOH) = 1 mol/l, to the inflection at about pH 9,2, using a glass indicator electrode.

1,00 ml of sodium hydroxide solution, c(NaOH) = 1,000 mol/l, corresponds to 0,136 09 g of KH_2PO_4.

R 79.3.2 pH

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

R 79.3.3 Chloride

Take 20 ml of the test solution (R 79.2) and apply GM 2.

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

R 79.3.4 Sulfate

Take 10 ml of the test solution (R 79.2) and apply GM 3.

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

R 79.3.5 Total nitrogen

Take 20 ml of the test solution (R 79.2), dilute to 140 ml with water and apply GM 6.

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

R 79.3.6 Heavy metals

Take 30 ml of the test solution (R 79.2) and apply GM 7.

Prepare a standard matching solution, using 10 ml of the test solution (R 79.2) and 2 ml of the lead SS II (2 ml ≙ 0,001 % Pb).

R 79.3.7 Iron

To 10 ml of the test solution (R 79.2), add 2 ml of 5-sulfosalicylic acid solution (20 %) and 5 ml of the ammonia solution (R 3).

After 1 min, the yellow coloration of the resulting solution shall not be more intense than that of similarly prepared standard matching solution, using 2 ml of the iron SS II (2 ml ≙ 0,002 % Fe).

R 79.3.8 Sodium

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

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

R 79.3.9 Loss after drying

Dry 5 g of the sample at 110 °C for 1 h.

The loss in mass shall not exceed 10 mg.

R 80 Potassium hexacyanoferrate(II) trihydrate $K_4[Fe(CN)_6].3H_2O$

Relative molecular mass: 422,41

R 80.1 Specification

Assay $\{K_4[Fe(CN)_6].3H_2O\}$	99 % min.
Water-insoluble matter	0,005 % max.
Chloride (Cl)	0,01 % max.
Sulfate (SO_4)	0,005 % max.
Sodium (Na)	0,02 % max.

R 80.2 Preparation of test solution

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

R 80.3 Tests

R 80.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 1 g of the sample and dissolve in 200 ml of water.

To this solution, add 10 ml of the sulfuric acid (R 37), 2 ml of the phosphoric acid (R 22) and titrate with standard volumetric potassium permanganate solution, $c(1/5\ KMnO_4) = 0{,}1$ mol/l, until the colour permanganate just persists.

1,00 ml of potassium permanganate solution, $c(1/5\ KMnO_4) = 0{,}100$ mol/l, corresponds to 0,042 24 g of $K_4[Fe(CN)_6].3H_2O$.

R 80.3.2 Water-insoluble matter

Take 20 g of the sample and apply GM 1.

The mass of the residue shall not exceed 1 mg.

R 80.3.3 Chloride

To 20 ml of the test solution (R 80.2), add 30 ml of water and 50 ml of copper(II) sulfate (R 9) solution (5 %). After 15 min, filter or centrifugate the reaction mixture.

Take 10 ml of the clear filtrate and apply GM 2.

Prepare a standard matching solution, using 2 ml of the chloride SS II (2 ml $\triangleq$ 0,01 % Cl).

R 80.3.4 Sulfate

Take 10 ml of the test solution (R 80.2) and apply GM 3.

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

R 80.3.5 Sodium

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

Element	Concentration of solution	Flame	Resonance line nm
Na	0,5 %	Air-ethine	589,0

R 81 Potassium hexacyanoferrate(III) $K_3[Fe(CN)_6]$

Relative molecular mass: 329,26

R 81.1 Specification

Assay $\{K_3[Fe(CN)_6]\}$	99 % min.
Water-insoluble matter	0,005 % max.
Chloride (Cl)	0,01 % max.
Hexacyanoferrate(II) $\{[Fe(CN)_6]^{4-}\}$	0,02 % max.
Sulfate (SO_4)	0,005 % max.
Sodium (Na)	0,02 % max.

R 81.2 Preparation of test solution

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

R 81.3 Tests

R 81.3.1 Assay

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

To this solution add 2 g of the potassium iodide (R 25), 3 g of zinc sulfate heptahydrate ($ZnSO_4.7H_2O$) and 1 ml of the hydrochloric acid (R 13). 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*).

Carry out a blank determination.

1,00 ml of sodium thiosulfate solution, $c(Na_2S_2O_3) = 0,100$ mol/l, corresponds to 0,032 93 g of $K_3[Fe(CN)_6]$.

R 81.3.2 Water-insoluble matter

Take 20 g of the sample and apply GM 1.

The mass of the residue shall not exceed 1 mg.

R 81.3.3 Chloride

To 20 ml of the test solution (R 81.2), add 30 ml of water and 50 ml of copper(II) sulfate (R 9) solution (5 %). After 15 min, filter or centrifugate the reaction mixture.

Take 10 ml of the clear filtrate and apply GM 2.

Prepare a standard matching solution, using 2 ml of the chloride SS II (2 ml $\triangleq$ 0,01 % Cl).

R 81.3.4 Hexacyanoferrate(II)

To 10 ml of the test solution (R 80.2) add 20 ml of water and 0,25 ml of the iron SS I.

After 15 min, any blue-green coloration shall not be more intense than that of a similarly prepared standard matching solution, using 5 ml of the test solution, 15 ml of water and 10 ml of the hexacyanoferrate(II) SS II {10 ml $\triangleq$ 0,002 % $[Fe(CN)_6]^{4-}$}.

R 81.3.5 Sulfate

Take 10 ml of the test solution (R 81.2) and apply GM 3.

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

R 81.3.6 Sodium

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

Element	Concentration of solution	Flame	Resonance line nm
Na	0,5 %	Air-ethine	589,0

R 82 Potassium hydrogen phthalate $KOOC{\cdot}C_6H_4COOH$

Relative molecular mass: 204,23

R 82.1 Specification

Assay ($C_8H_5KO_4$)	99,8 % min.
Chloride (Cl)	0,002 % max.
Total sulfur (expressed as SO_4)	0,006 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Sodium (Na)	0,01 % max.
Loss after drying (at 105 °C)	0,05 % max.

R 82.2 Preparation of test solution

Dissolve 10 g of the sample by heating in water and dilute to 100 ml (the solution shall be clear and colourless).

R 82.3 Tests

R 82.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 4 g of the sample, dissolve in 100 ml of carbon dioxide-free water and titrate with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 1$ mol/l, using the phenolphthalein (IS 4.3.9*).

1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 1{,}000$ mol/l, corresponds to 0,204 23 g of $C_8H_5KO_4$.

R 82.3.2 Chloride

Dissolve 5 g of the sample in 30 ml of hot water, cool and add 10 ml of the nitric acid (R 19). Filter the precipitated phthalic acid on a filter washed free of chloride and dilute the filtrate to 50 ml with water.

Take 10 ml of this solution and apply GM 2.

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

R 82.3.3 Total sulfur

Mix 2 g of the sample in a porcelain dish with 1 g of sodium carbonate, add 15 ml of water, evaporate to dryness and ignite at 800 °C for 2 h. Allow to cool, add 20 ml of water and 2 ml of the hydrogen peroxide (R 14) and heat on a boiling water-bath for 15 min. Add 5 ml of hydrochloric acid solution (25 %) and again evaporate to dryness. Dissolve the residue in 10 ml of water, filter and dilute to 20 ml with water.

Take this solution and apply GM 3.

Prepare a standard matching solution, using 12 ml of the sulfate SS II (12 ml $\triangleq$ 0,006 % SO_4).

R 82.3.4 Heavy metals

Take 20 ml of the test solution (R 82.2) and apply GM 7.

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

R 82.3.5 Iron

Take 20 ml of the test solution (R 82.2) and apply GM 8.1.

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

R 82.3.6 Sodium

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

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

R 82.3.7 Loss after drying

Dry 4 g of the sample at 105 °C to constant mass.

The loss in mass shall not exceed 2 mg.

R 83 Potassium iodate KIO_3

Relative molecular mass: 214,00

R 83.1 Specification

Assay (KIO_3)	99,7 % min.
pH (5 % solution)	5 to 8
Chloride, chlorate, bromide and bromate (expressed as Cl)	0,02 % max.
Iodide (I)	0,002 % max.
Sulfate (SO_4)	0,005 % max.
Total nitrogen (N)	0,002 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,001 % max.
Sodium (Na)	0,01 % max.
Loss after drying (at 130 °C)	0,05 % max.

R 83.2 Preparation of test solutions

R 83.2.1 Test solution I

Dissolve 10 g of the sample in water and dilute to 200 ml (the solution shall be clear and colourless).

R 83.2.2 Test solution II

Evaporate 6 g of the sample twice with 25 ml of the hydrochloric acid (R 13) and heat until the iodine is removed. Dissolve the residue in 2,5 ml of hydrochloric acid solution (25 %) and dilute to 50 ml with water.

R 83.2.3 Test solution III

Adopt the procedure specified in R 83.2.2, but using 1 g of the sample.

R 83.3 Tests

R 83.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 1,5 g of the sample, dissolve in water in a 200 ml one-mark volumetric flask and dilute to the mark.

Take 20,0 ml of this solution, add 3 g of the potassium iodide (R 25) and 5 ml of hydrochloric acid solution (25 %) and dilute with 100 ml of water. Titrate the liberated iodine with standard volumetric sodium thiosulfate solution, $c(Na_2S_2O_3) = 0,1$ mol/l, adding 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 0,003 566 7 g of KIO_3.

R 83.3.2 pH

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

R 83.3.3 Chloride, chlorate, bromide and bromate

Dilute 5 ml of the test solution I (R 83.2.1) to 15 ml with water, add 20 ml of sulfurous acid solution (5 % SO_2) and heat on a steam bath for 30 min. Heat to boiling, cool, add 10 ml of the ammonia solution (R 3), 20 ml of silver nitrate solution (1,7 %) and dilute to 70 ml with water. Filter, take 35 ml of the filtrate and acidify with 6 ml of the nitric acid (R 19).

After 5 min, any opalescence shall not be greater than that of a similarly prepared standard matching solution, using 5 ml of the chloride SS II (5 ml $\triangleq$ 0,02 % Cl).

R 83.3.4 Iodide

Take 25 ml of the test solution I (R 83.2.1), add 1 ml of sulfuric acid solution (9,8 %) and then shake with 1 ml of the chloroform (R 7).

The violet coloration of the chloroform layer shall not be more intense than that of a similarly prepared standard matching solution, using 5 ml of the test solution I (R 83.2.1) and 2 ml of the iodide SS II (2 ml $\triangleq$ 0,002 % I).

R 83.3.5 Sulfate

Take 10 ml of the test solution II (R 83.2.2) and apply GM 3, without addition of 0,5 ml of hydrochloric acid solution (20 %).

Prepare a standard matching solution, using 10 ml of the test solution III (R 83.2.3) and 5 ml of the sulfate SS II (5 ml ≙ 0,005 % SO_4).

R 83.3.6 Total nitrogen

Dilute 20 ml of the test solution I (R 83.2.1) 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 83.3.7 Heavy metals

Neutralise 20 ml of the test solution II (R 83.2.2) and apply GM 7.

Prepare a standard matching solution, using 20 ml of the test solution III (R 83.2.3) and 1 ml of the lead SS II (1 ml ≙ 0,000 5 % Pb).

R 83.3.8 Iron

Take 10 ml of the test solution II (R 83.2.2) and apply GM 8.1.

Prepare a standard matching solution, using 10 ml of the test solution III (R 83.2.3) and 1 ml of the iron SS II (1 ml ≙ 0,001 % Fe).

R 83.3.9 Sodium

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

Element	Concentration of solution	Flame	Wavelength nm
Na	4 %	Air-ethine	589,0

R 83.3.10 Loss after drying

Dry 2 g of the sample at 130 °C to constant mass.

The loss in mass shall not exceed 1 mg.

R 84 Potassium nitrate KNO_3

Relative molecular mass: 101,10

R 84.1 Specification

Assay (KNO_3)	99 % min.
pH (5 % solution)	5 to 8
Chloride (Cl)	0,001 % max.
Iodate	0,000 5 % max.
Nitrite	0,001 % max.
Phosphate (PO_4)	0,000 5 % max.
Sulfate (SO_4)	0,003 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Ammonium (NH_4)	0,001 % max.
Calcium (Ca)	0,005 % max.
Sodium (Na)	0,02 % max.

R 84.2 Preparation of test solution

Dissolve 20 g of the sample in 150 ml of water (the solution shall be clear and colourless) and dilute to 200 ml.

R 84.3 Tests

R 84.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,4 g of the sample, dissolve in 50 ml of carbon dioxide-free water and pass the solution through a cation-exchange column previously washed with water until washings are neutral (diameter of the column: about 15 mm; height of the resin bed: about 30 cm). Titrate the eluate with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 0,1$ mol/l, using the methyl red (IS 4.3.6*).

1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 0,100$ mol/l, corresponds to 0,010 11 g of KNO_3.

R 84.3.2 pH

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

R 84.3.3 Chloride

Take 10 ml of the test solution (R 84.2), add 10 ml of water and apply GM 2.

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

R 84.3.4 Iodate and nitrite

Dissolve 1 g of the sample in 10 ml of water, and add 0,10 ml of potassium iodide solution (10 %), 1 ml of the chloroform (R 7) and 1 ml of the acetic acid (R 1). Shake gently for 5 min.

The chloroform shall not acquire a pink or violet colour.

R 84.3.5 Phosphate

Take 40 ml of the test solution (R 84.2) and apply GM 4.

Prepare a standard matching solution, using 2 ml of the phosphate SS II (2 ml $\triangleq$ 0,000 5 % PO_4).

R 84.3.6 Sulfate

To 30 ml of the test solution (R 84.2), add 20 ml of the hydrochloric acid (R 13), and evaporate to dryness. Dissolve the residue in 20 ml of hydrochloric acid solution (20 %), and again evaporate to dryness. Dissolve the residue in 20 ml of water, filter if necessary and apply GM 3.

Prepare a standard matching solution, using 9 ml of the sulfate SS II (9 ml $\triangleq$ 0,003 % SO_4).

R 84.3.7 Heavy metals

Take 20 ml of the test solution (R 84.2) and apply GM 7.

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

R 84.3.8 Iron

To 40 ml of the test solution (R 84.2) add 15 ml of the hydrochloric acid (R 13), and evaporate to dryness. Dissolve the residue in 15 ml of hydrochloric acid solution (20 %), and again evaporate to dryness. Dissolve the residue in water and dilute to 40 ml.

Take 20 ml of this solution 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 84.3.9 Ammonium

Take 40 ml of the test solution (R 84.2) and apply GM 10.

Prepare a standard matching solution, using 4 ml of the ammonium SS II (4 ml ≙ 0,001 % NH_4).

R 84.3.10 Calcium

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	2 %	Air-ethine	422,7

R 84.3.11 Sodium

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

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

R 85 Potassium sulfate K_2SO_4

Relative molecular mass: 174,27

R 85.1 Specification

Assay (K_2SO_4)	99,0 % min.
pH (5 % solution)	5 to 8
Total nitrogen (N)	0,000 5 % max.
Chloride (Cl)	0,000 5 % max.
Arsenic (As)	0,000 2 % max.
Calcium (Ca)	0,01 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Sodium (Na)	0,03 % max.

R 85.2 Preparation of test solution

Dissolve 20 g of the sample in 150 ml of water and dilute to 200 ml. (The solution shall be clear and colourless.)

R 85.3 Tests

R 85.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 2 g of the sample and dissolve in 50 ml of water. Pass this solution through a cation exchange column (strongly acid) previously wash with water until washings are neutral (diameter of the column: about 15 mm; height of the resin bed: about 30 cm). Wash with water and titrate the combined eluate and washings with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 1$ mol/l, using the phenolphthalein (IS 4.3.9*).

1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 1{,}000$ mol/l, corresponds to 0,087 135 g of K_2SO_4.

R 85.3.2 pH

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

R 85.3.3 Total nitrogen

Take 50 ml of the test solution (R 85.2) and apply GM 6.

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

R 85.3.4 Chloride

Take 20 ml of the test solution (R 85.2) and apply GM 2.

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

R 85.3.5 Arsenic

Take 10 ml of the test solution (R 85.2) and apply GM 11.

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

R 85.3.6 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-ethine	422,7

R 85.3.7 Heavy metals

Take 20 ml of the test solution (R 85.2) and apply GM 7.

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

R 85.3.8 Iron

Take 30 ml of the test solution (R 85.2) and apply GM 8.1.

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

R 85.3.9 Sodium

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

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

R 86 Potassium thiocyanate KSCN

Relative molecular mass: 97,18

R 86.1 Specification

Assay (KSCN)	98,0 % min.
pH (5 % solution)	5,3 to 8,5
Chloride (Cl)	0,005 % max.
Sulfate (SO_4)	0,005 % max.
Sulfide (S)	0,001 % max.
Ammonium (NH_4)	0,002 % max.
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,000 1 % max.
Lead (Pb)	0,000 5 % max.
Sodium (Na)	0,015 % max.
Iodine-consuming substances (expressed as I)	0,025 % max.

R 86.2 Tests

R 86.2.1 Assay

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

To this solution, add 5 ml of nitric acid solution (25 %) and 50,0 ml standard volumetric silver nitrate solution, $c(AgNO_3) = 0,1$ mol/l, and titrate the excess silver nitrate with standard volumetric ammonium thiocyanate solution, $c(NH_4SCN) = 0,1$ mol/l, using the ammonium iron(III) sulfate (IS 4.3.1*).

1,00 ml of silver nitrate solution, $c(AgNO_3) = 0,100$ mol/l, corresponds to 0,009 718 g of KSCN.

R 86.2.2 pH

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

R 86.2.3 Chloride

Dissolve 1 g of the sample with 1 g of ammonium nitrate in 20 ml of water, add 10 ml of the hydrogen peroxide (R 14) and 2 ml of sodium hydroxide solution (10 %). Warm and shake carefully. After the reaction is finished, add another 10 ml of the hydrogen peroxide (R 14), boil for 5 min, cool and add 10 ml of nitric acid solution (25 %) Apply GM 2.

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

R 86.2.4 Sulfate

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

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

R 86.2.5 Sulfide

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

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

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

R 86.2.6 Ammonium

Dissolve 1 g of the sample in 75 ml of water and apply GM 10.

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

R 86.2.7 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 15 g of the sample in 150 ml of water and apply solvent extraction procedure according to GM 35	Air-ethine	324,7
Fe			248,3
Pb			217,0 or 283,3

R 85.2.8 Sodium

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

Element	Concentration of solution	Flame	Wavelength nm
Na	4 %	Air-ethine	589,0

R 86.2.9 Iodine-consuming substances

Dissolve 5 g of the sample in 50 ml of water, add 1 ml of sulfuric acid solution (10 %), 1 g of the potassium iodide (R 25) and titrate with standard volumetric iodine solution, $c(1/2\ I_2) = 0{,}01$ mol/l, using the starch (IS 4.3.11*).

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

R 87 2-Propanol C_3H_7OH

Relative molecular mass: 60,10

R 87.1 Specification

Assay (C_3H_8O)	99,7 % min.
Methanol (CH_3OH)	0,1 % max.
Density at 20 °C	0,784 to 0,786 g/ml
Residue after evaporation	0,001 % max.
Acidity (expressed in millimoles of H^+)	0,03/100 g max.
Iron (Fe)	0,000 01 % max.
Carbonyl compounds (expressed as CO)	0,005 % max.
Water (H_2O)	0,1 % max.

R 87.2 Tests

R 87.2.1 Assay and methanol

Apply GM 34, using the following conditions:

Stationary phase	Polyethylene glycol 400 or 1 500
Support	Wall-coated open tubular column (WCOT-column)
Column length	25 to 50 m
Column internal diameter	0,2 to 0,3 mm
Column material	Glass
Column temperature	70 °C
Injection temperature	150 °C
Detection temperature	150 °C
Type of detector	Flame ionization
Carrier gas	Nitrogen
Flow rate	0,3 ml/min
Split	1 : 200
Test portion	0,5 µl

R 87.2.2 Density

Apply GM 24.1.

R 87.2.3 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.

Reserve the residue for the determination of iron.

R 87.2.4 Acidity

Take 100 g (127 ml) of the sample and apply GM 13.1, titrating with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 0{,}02$ mol/l, and using bromothymol blue as indicator.

The volume of titrant shall not exceed 1,5 ml.

R 87.2.5 Iron

Dissolve the residue of test (R 87.2.3) in 1 ml of hydrochloric acid solution (25 %) and 9 ml of water and apply GM 8.1.

Prepare a standard matching solution, using 1 ml of the iron SS II (1 ml $\triangleq$ 0,000 01 % Fe).

R 87.2.6 Carbonyl compounds

Take 1 g (1,3 ml) of the sample and apply GM 23.

Prepare a standard matching solution, using 5 ml of the carbonyl SS II (5 ml $\triangleq$ 0,005 % CO).

R 87.2.7 Water

Take 7,8 g (10 ml) of the sample and apply GM 12, using 20 ml of the methanol (R 18) as the solvent.

R 88 Sodium fluoride NaF

Relative molecular mass: 41,99

R 88.1 Specification

Assay (NaF)	99 % min.
Water-insoluble matter	0,02 % max.
Acidity (expressed in millimoles of H^+)	2,5/100 g max.
Alkalinity (expressed in millimoles of OH^-)	2/100 g max.
Chloride (Cl)	0,003 % max.
Hexafluorosilicate (SiF_6)	0,1 % max.
Sulfate (SO_4)	0,01 % max.
Heavy metals (expressed as Pb)	0,001 % max.
Iron (Fe)	0,002 % max.
Loss after drying (at 150 °C)	0,3 % max.

R 88.2 Preparation of test and reference solutions

R 88.2.1 Test solution

To 7 g of the sample in a platinum dish add 10 ml of the hydrochloric acid (R 13) and evaporate to dryness on a water bath. Repeat this procedure three times, then dissolve the residue in water, dilute to 50 ml and filter, if necessary.

R 88.2.2 Reference solution

To 2 g of the sample in a platinum dish add 5 ml of the sulfate SS I (5 ml $\triangleq$ 0,01 % SO_4) and proceed as described for the preparation of the test solution (R 88.2.1), using the same quantities of reagents.

R 88.3 Tests

R 88.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,15 g of the sample and dissolve in 25 ml of water. Pass this solution through an ion exchange column (diameter of the column: about 15 mm) charged to a height of about 30 cm with Amberlite IR 120 (H^+) cation exchange-resin previously washed with water until the washings are neutral. Wash with water and collect the eluate and washings in a polyethylene beaker. Titrate with standard volumetric sodium hydroxide solution, $c(NaOH) = 0,1$ mol/l, using the phenolphthalein (IS 4.3.9*).

1,00 ml of sodium hydroxide solution, $c(NaOH) = 0,100$ mol/l, corresponds to 0,004 2 g of NaF.

R 88.3.2 Water-insoluble matter

Take 10 g of the sample and apply GM 1.

The mass of the residue shall not exceed 2 mg.

R 88.3.3 Acidity or alkalinity

Dissolve 2 g of the sample in 60 ml of water in a platinum dish, add 10 ml of potassium nitrate solution (30 %) and 0,3 ml of the phenolphthalein (IS 4.3.9*) and cool to 0 °C.

If the solution is colourless, titrate with standard volumetric sodium hydroxide solution, $c(NaOH) = 0,1$ mol/l, until a pink colour remains stable for 15 s. The volume of titrant shall not exceed 0,5 ml.

If the solution shows a pink colour, titrate with standard volumetric hydrochloric acid solution, $c(HCl) = 0,1$ mol/l, until the solution is just colourless. The volume of titrant shall not exceed 0,4 ml.

Reserve the solution for the test R 88.3.5.

R 88.3.4 Chloride

Dissolve 0,5 g of the sample in 20 ml of water, add 0,2 g of the boric acid (R 50) and apply GM 2.

Prepare a standard matching solution, using 1,5 ml of the chloride SS II (1,5 ml $\triangleq$ 0,003 % Cl).

R 88.3.5 Fluorosilicate

Take the solution obtained in test (R 88.3.3) and bring to boiling. If the solution turns colourless, titrate with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 0{,}1$ mol/l, until a permanent pink colour is produced.

1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 0{,}100$ mol/l, corresponds to 0,003 55 g of SiF_6.

The volume of titrant shall not exceed 0,55 ml.

R 88.3.6 Sulfate

Take 20 ml of the test solution (R 88.2.1) and apply GM 3.

Prepare a standard matching solution, using 20 ml of the reference solution (R 88.2.2).

R 88.3.7 Heavy metals

Dissolve 2 g of the sample in 55 ml of water by heating in a platinum dish. Transfer this solution to a polyethylene beaker, adjust to a pH-value between 3 and 4 using the acetic acid (R 1) and dilute to 60 ml (solution A).

Take 40 ml of the solution A and apply GM 7.

Prepare a standard matching solution, using 10 ml of the solution A with 1 ml of the lead SS II (1 ml $\triangleq$ 0,001 % Pb).

R 88.3.8 Iron

Take 10 ml of the test solution (R 88.2.1) and apply GM 8.1.

Prepare a standard matching solution, using 1 ml of the Iron SS I (1 ml $\triangleq$ 0,002 % Fe).

R 88.3.9 Loss after drying

Ignite 2 g of the sample at 150 °C to constant mass.

The loss in mass shall not exceed 6 mg.

R 89 Sodium hydrogen carbonate $NaHCO_3$

Relative molecular mass: 84,01

R 89.1 Specification

Assay ($NaHCO_3$)	99,5 % min.
Chloride (Cl)	0,001 % max.
Phosphate and silicate (expressed as SiO_2)	0,005 % max.
Sulfate (SO_4)	0,003 % max.
Total nitrogen (N)	0,000 5 % max.
Calcium (Ca)	0,01 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Magnesium (Mg)	0,01 % max.
Potassium (K)	0,005 % max.
Substances reducing iodine (expressed as I)	0,006 5 % max.

R 89.2 Preparation of test solution

Dissolve 20 g of the sample in 150 ml of boiling water and neutralize with about 20 ml of the hydrochloric acid (R 13). Heat just to boiling, cool, and dilute to 200 ml with water.

R 89.3 Tests

R 89.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 3 g of the sample, dissolve in 50 ml of water, and titrate with standard volumetric hydrochloric acid solution, $c(HCl) = 1$ mol/l, using the methyl orange (IS 4.3.5*).

1,00 ml of hydrochloric acid solution, $c(HCl) = 1{,}000$ mol/l, corresponds to 0,084 007 g of $NaHCO_3$.

R 89.3.2 Chloride

Dissolve 1 g of the sample in 14 ml of water and 5 ml of nitric acid solution (25 %) and apply GM 2.

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

R 89.3.3 Phosphate and silicate

To 10 ml of the test solution (R 89.2), add 5 ml of hexaammonium heptamolybdate solution (10 %), dilute to 80 ml with water and adjust to pH 1,8. Heat to boiling, cool, add 10 ml of the hydrochloric acid (R 13) and extract with 50 ml of 4-methyl-2-pentanone. Wash the organic phase three times with 20 ml of hydrochloric acid solution (0,5 %) and reduce with 0,2 ml of tin(II) chloride solution [2 % $SnCl_2\,2H_2O$ in 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 5 ml of the silicate SS II (5 ml ≙ 0,005 % SiO_2).

R 89.3.4 Sulfate

Take 20 ml of the test solution (R 89.2) and apply GM 3.

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

R 89.3.5 Total nitrogen

Take 20 ml of the test solution (R 89.2), dilute to 140 ml with water and apply GM 6.

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

R 89.3.6 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-ethine	422,7

R 89.3.7 Heavy metals

Take 20 ml of the test solution (R 89.2) and apply GM 7.

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

R 89.3.8 Iron

Take 20 ml of the test solution (R 89.2) 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 89.3.9 Magnesium

To 5 ml of the test solution (R 89.2), add 5 ml of sodium hydroxide solution (27 %) and 0,2 ml of titan yellow[20] solution (0,1 %) Cl 19540.

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

R 89.3.10 Potassium

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

Element	Concentration of solution	Flame	Wavelength nm
K	4 %	Air-ethine	766,5

R 89.3.11 Substances reducing iodine

Dissolve 2 g of potassium iodide (R 25) in 200 ml of water, add 2 ml of the starch (IS 4.3.11*) and add standard volumetric iodine solution, $c(1/2\ I_2) = 0{,}1$ mol/l, until a blue colour is produced. Then add 10 g of the sample.

If the blue colour disappears, it shall reappear after addition of 0,05 ml of standard volumetric iodine solution, $c(1/2\ I_2) = 0{,}1$ mol/l.

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[20] IUPAC name: disodium 6,6′-dimethyl-2,2′-(triazene-1,3-diyldi-*p*-phenylene)bisbenzothiazole-7-sulfonate.

R 90 Sodium nitrate $NaNO_3$

Relative molecular mass: 84,99

R 90.1 Specification

Assay ($NaNO_3$)	99 % min.
pH (5 % solution)	5 to 8
Chloride (Cl)	0,001 % max.
Iodate (IO_3)	0,000 5 % max.
Nitrite (NO_2)	0,001 % max.
Phosphate (PO_4)	0,000 5 % max.
Sulfate (SO_4)	0,003 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Ammonium (NH_4)	0,002 % max.
Calcium (Ca)	0,01 % max.
Potassium (K)	0,01 % max.

R 90.2 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 90.3 Tests

R 90.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,3 g of the sample, dissolve in 50 ml of carbon dioxide-free water and pass the solution through a cation-exchange column previously washed with water until washings are neutral (diameter of the column: about 15 mm; height of the resin bed: about 30 cm). Titrate the eluate with standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 0,1$ mol/l, using the methyl red (IS 4.3.6*).

1,00 ml of sodium hydroxide solution, $c(\text{NaOH}) = 0,100$ mol/l, corresponds to 0,008 499 g of $NaNO_3$.

R 90.3.2 pH

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

R 90.3.3 Chloride

Take 10 ml of the test solution (R 90.2) add 10 ml of water and apply GM 2.

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

R 90.3.4 Iodate and nitrite

Dissolve 1 g of the sample in 10 ml of water, and add 0,10 ml of potassium iodide solution (10 %), 1 ml of the chloroform (R 7) and 1 ml of the acetic acid (R 1). Shake gently for 5 min.

The chloroform shall not acquire a pink or violet colour.

R 90.3.5 Phosphate

Take 40 ml of the test solution (R 90.2) and apply GM 4.

Prepare a standard matching solution, using 2 ml of the phosphate SS II (2 ml $\triangleq$ 0,000 5 % PO_4).

R 90.3.6 Sulfate

To 30 ml of the test solution (R 90.2) add 20 ml of the hydrochloric acid (R 13), and evaporate to dryness. Dissolve the residue in 20 ml of hydrochloric acid solution (20 %), and again evaporate to dryness. Dissolve the residue in 20 ml of water, filter if necessary, and apply GM 3.

Prepare a standard matching solution, using 9 ml of the sulfate SS II (9 ml $\triangleq$ 0,003 % SO_4).

R 90.3.7 Heavy metals

Take 20 ml of the test solution (R 90.2) and apply GM 7

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

R 90.3.8 Iron

To 40 ml of the test solution (R 90.2) add 15 ml of the hydrochloric acid (R 13) and evaporate to dryness. Dissolve the residue in 15 ml of hydrochloric acid solution (20 %), and again evaporate to dryness. Dissolve the residue in water and dilute to 40 ml.

Take 20 ml of this solution 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 90.3.9 Ammonium

Take 20 ml of the test solution (R 90.2) and apply GM 10.

Prepare a standard matching solution, using 4 ml of the ammonium SS II (4 ml ≙ 0,002 % NH_4).

R 90.3.10 Calcium

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	2 %	Air-ethine	422,7

R 90.3.11 Potassium

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

Element	Concentration of solution	Flame	Wavelength nm
K	4 %	Air-ethine	766,5

R 91 Sodium peroxide Na_2O_2

Relative molecular mass: 77,98

R 91.1 Specification

Assay (Na_2O_2)	95 % min.
Chloride (Cl)	0,002 % max.
Phosphate (PO_4)	0,000 5 % max.
Sulfate (SO_4)	0,001 % max.
Total nitrogen (N)	0,003 % max.
Aluminium (Al)	0,005 % max.
Heavy metals (expressed as Pb)	0,002 % max.
Iron (Fe)	0,005 % max.

R 91.2 Preparation of test solution

In a polyethylene beaker add 20 g of the sample in small portions to 100 ml of water and heat on a steam bath until the reaction ceases. Cool, neutralize with about 63 ml of hydrochloric acid solution (25 %), add 3 ml in excess and heat. Cool and dilute to 200 ml with water.

R 91.3 Tests

R 91.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,5 g of the sample, dissolve in small portions in about 400 ml of sulfuric acid (1 %) in a 500 ml one-mark volumetric flask and dilute to the mark with the same solvent.

Take 100,00 ml of this solution 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 0,003 898 9 g of Na_2O_2.

R 91.3.2 Chloride

Dissolve 1 g of the sample in small portions in 30 ml of water and 5 ml of nitric acid solution (25 %). Heat for 2 min, cool and apply GM 2.

Prepare a standard matching solution, using 5 ml of nitric acid solution (25 %) and 2 ml of the chloride SS II (2 ml ≙ 0,002 % Cl).

R 91.3.3 Phosphate

Take 50 ml of the test solution (R 91.2) 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 91.3.4 Sulfate

Take 40 ml of the test solution (R 91.2) and apply GM 3, without addition of 0,5 ml of hydrochloric acid solution (20 %).

Prepare a standard matching solution, using 0,4 ml of the hydrochloric acid (R 13) and 4 ml of the sulfate SS II (4 ml ≙ 0,001 % SO_4).

R 91.3.5 Total nitrogen

Place 25 ml of water and 1 ml of the sulfuric acid (R 37) in a platinum dish and with cooling add 1 g of the sample. Allow to stand over night until decomposition is complete, dilute to 140 ml with water and apply GM 6.

Prepare a standard matching solution, using 1 ml of the sulfuric acid (R 37) and 3 ml of the nitrogen SS II (3 ml ≙ 0,003 % N).

R 91.3.6 Aluminium

To 5 ml of the test solution (R 91.2), add 0,1 ml of acetic acid solution (30 %), 5 ml of sodium acetate trihydrate solution (20 %) and 10 ml of the methanol (R 18), and mix with 0,1 ml of morin Cl 75660[21] solution [0,2 % in the methanol (R 18)].

After 10 min, the green-yellow fluorescence under a UV lamp shall not be more intense than that of a similarly prepared standard matching solution, using 2,5 ml of the aluminium SS II (2,5 ml ≙ 0,005 % Al).

R 91.3.7 Heavy metals

Take 20 ml of the test solution (R 91.2) and neutralize with ammonia solution (10 %). Add 0,5 ml of acetic acid solution (30 %) and 1 ml of ascorbic acid solution (1 %) and then saturate with hydrogen sulfide.

The brown coloration shall not be more intense than that of a similarly prepared standard matching solution, using 4 ml of lead SS II (4 ml ≙ 0,002 % Pb).

R 91.3.8 Iron

Take 5 ml of the test solution (R 91.2) and apply GM 8.1.

Prepare a standard matching solution using 2,5 ml of the iron SS II (2,5 ml 0,005 % Fe).

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[21] IUPAC name: 2′,3,4′,5,7-Pentahydroxyflavone.

R 92 Starch, soluble $(C_6H_{10}O_5)_n$

R 92.1 Specification

pH (2 % solution)	6,0 to 7,5
Sensitivity to iodine	To pass test
Substances reducing Fehling's reagent (expressed as maltose)	0,7 % max.
Loss after drying (at 105 °C)	13 % max.
Sulfated ash	0,5 % max.

R 92.2 Preparation of test solution

Prepare a paste of 2 g of the sample and 10 ml of water, add 90 ml of hot water and heat to boiling for 2 min (the solution shall be almost clear and after cooling it may become opalescent or turbid but shall not gel).

R 92.3 Tests

R 92.3.1 pH

Determine the pH of the test solution (R 92.2) according to GM 31.1, using a calibrated pH meter.

R 92.3.2 Sensitivity to iodine

Mix 2,5 ml of the test solution (R 92.2) with 97,5 ml of water and 0,5 ml of standard volumetric iodine solution, $c(1/2\ I_2)$ = 0,01 mol/l.

The blue colour shall disappear after addition of 0,5 ml of standard volumetric sodium thiosulfate solution, $c(Na_2S_2O_3)$ = 0,01 mol/l.

R 92.3.3 Substances reducing Fehling's reagent

Take 10 g of the sample and shake with 100 ml of water for 15 min. After 12 h, filter through a sintered-glass filter crucible. To 50 ml of the filtrate add 50 ml of Fehling's reagent and heat to boiling for 2 min. Filter off the copper(I) oxide, wash with water, ethanol and diethyl ether and dry at 105 °C to constant mass.

The mass of the residue shall not exceed 50 mg (50 mg $Cu_2O \triangleq$ 0,7 % maltose).

R 92.3.4 Loss after drying

Dry 1 g of the sample at 105 °C for 2 h.

The loss in mass shall not exceed 130 mg.

R 92.3.5 Sulfated ash

Take 1 g of the sample and apply GM 17.

The mass of the residue shall not exceed 5 mg.

R 93 (+)-Tartaric acid $HOOC(CHOH)_2COOH$

Relative molecular mass: 150,09

R 93.1 Specification

Assay ($C_4H_6O_6$)	99,5 % min.
Chloride (Cl)	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.
Sulfated ash	0,01 % max.

R 93.2 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 93.3 Tests

R 93.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 3 g of the sample, dissolve in 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 0,075 045 g of $C_4H_6O_6$.

R 93.3.2 Chloride

Take 20 ml of the test solution (R 93.2), acidify with 1 ml of the nitric acid (R 19) and apply GM 2.

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

R 93.3.3 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	4 %	Air-ethine	422,7
Cu	5 %		324,7
Pb			217,0 or 283,3

R 93.3.4 Iron

Dissolve 0,05 g of potassium peroxodisulfate in a mixture of 20 ml of the test solution (R 93.2), 30 ml of water and 5 ml of the hydrochloric acid (R 13) and add 2 ml of ammonium thiocyanate solution (5 %).

After 1 min, the red coloration shall not be more intense than that of a similarly prepared standard matching solution, using 1 ml of the iron SS II (1 ml ≙ 0,000 5 % Fe).

R 93.3.5 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 1 mg.

R 94 Trisodium citrate dihydrate $C_6H_5Na_3O_7.2H_2O$

Relative molecular mass: 294,10

R 94.1 Specification

Assay ($C_6H_5Na_3O_7.2H_2O$)	99,0 % min.
pH (5 % solution)	7,5 to 9,0
Total nitrogen (N)	0,001 % max.
Chloride (Cl)	0,001 % max.
Phosphate (PO_4)	0,002 % max.
Sulfate (SO_4)	0,005 % max.
Heavy metals (expressed as Pb)	0,000 5 % max.
Iron (Fe)	0,000 5 % max.
Readily carbonizable substances	To pass test

R 94.2 Preparation of test and reference solutions

R 94.2.1 Test solution I

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

R 94.2.2 Test solution II

To 7 g of the sample add 0,2 ml of trioxovanadate of ammonium solution (1 %) and 20 ml of the nitric acid (R 19) and keep this mixture in a beaker on a boiling water bath until the reaction has ceased. Evaporate to dryness. Add 10 ml of the nitric acid (R 19) and repeat the decomposition and evaporation on the water bath. Add 10 ml of hydrochloric acid solution (20 %), evaporate to dryness and repeat this procedure once again. Then dissolve the residue in a few millilitres of water, containing 1 ml of hydrochloric acid solution (20 %), filter if necessary and dilute to 50 ml with water.

R 94.2.3 Reference solution

To 2 g of the sample add 1 ml of the phosphate SS I (1 ml $\triangleq$ 0,002 % PO_4) and 2,5 ml of the sulfate SS I (2,5 ml $\triangleq$ 0,005 % SO_4) and proceed as described for the preparation of test solution II (R 94.2.2), using the same quantities of reagents.

R 94.3 Tests

R 94.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,2 g of the sample and dissolve in 30 ml of the acetic acid (R 1).

To this solution add 0,4 ml of 1-naphtholbenzein[22] solution [0,1 % in the acetic acid (R 1)] and titrate with standard volumetric perchloric acid solution, $c(HClO_4)$ = 0,1 mol/l, in the acetic acid (R 1), to a green colour.

1,00 ml of perchloric acid solution, $c(HClO_4)$ = 0,100 mol/l, corresponds to 0,009 803 g of $C_6H_5Na_3O_7.2H_2O$.

R 94.3.2 pH

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

R 94.3.3 Total nitrogen

Dissolve 2 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 $\triangleq$ 0,001 % N).

R 94.3.4 Chloride

Take 10 ml of the test solution I (R 94.2.1), dilute to 20 ml with water and apply GM 2.

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

R 94.3.5 Phosphate

Take 20 ml of the test solution II (R 94.2.2) and apply GM 4.

Prepare a standard matching solution, using 20 ml of the reference solution (R 94.2.3).

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[22] IUPAC name: 4-[α(4-hydroxy-1-naphthyl)benzylidene]naphthalen-1(4*H*)-one.

R 94.3.6 Sulfate

Take 10 ml of the test solution II (R 94.2.2) and apply GM 3.

Prepare a standard matching solution, using 10 ml of the reference solution (R 94.2.3).

R 94.3.7 Heavy metals

Take 10 ml of the test solution II (R 94.2.2), add 0,5 ml of water and apply GM 7.

Prepare a standard matching solution, using 10 ml of the reference solution (R 94.2.3) and 0,5 ml of the lead SS II (0,5 ml $\triangleq$ 0,000 5 % Pb).

R 94.3.8 Iron

Take 12,5 ml of the test solution I (R 94.2.1), neutralize with hydrochloric acid solution (25 %) and apply GM 8.1.

Prepare a standard matching solution, using 2,5 ml of the test solution I (R 94.2.1) and 1 ml of the iron SS II (1 ml $\triangleq$ 0,000 5 % Fe).

R 94.3.9 Readily carbonizable substances

Take 0,5 g of powdered sample and apply GM 18, using 10 ml of sulfuric acid (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 95 Xylene[23] $C_6H_4(CH_3)_2$

Relative molecular mass: 106,17

R 95.1 Specification

Colour in Hazen units	10 max.
Density at 20 °C	0,860 to 0,870 g/ml
Distillation interval	137 to 142 °C (min. 95 % shall distil within 3 °C)
Acidity (expressed in millimoles of H^+)	0,025/100 g max.
Alkalinity (expressed in millimoles of OH^-)	0,025/100 g max.
Residue after evaporation	0,002 % max.
Readily carbonizable substances	To pass test
Sulfur compounds (expressed as S)	0,003 % max.
Thiophene and homologues (expressed as C_4H_4S)	To pass test (about 0,000 1 %)
Benzene (C_6H_6)	0,1 % max.
Toluene (C_7H_8)	1 % max.
Water (H_2O)	0,03 % max.

R 95.2 Tests

R 95.2.1 Colour in Hazen units

Apply GM 36.

R 95.2.2 Density

Apply GM 24.1.

R 95.2.3 Distillation interval

Apply GM 25.1.

R 95.2.4 Acidity or alkalinity

To 60 ml of the methanol (R 18) add 0,2 ml of bromothymol blue solution and neutralize if necessary with either standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 0{,}01$ mol/l, or hydrochloric acid solution, $c(\text{HCl}) = 0{,}01$ mol/l. Add 50 g (58 ml) of the sample and titrate with either standard volumetric sodium hydroxide solution, $c(\text{NaOH}) = 0{,}01$ mol/l, or the standard volumetric hydrochloric acid solution, $c(\text{HCl}) = 0{,}01$ mol/l, to neutrality.

The volume of titrant shall not exceed 1,25 ml.

R 95.2.5 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 95.2.6 Readily carbonizable substances

Shake vigorously 12,9 g (15 ml) of the sample with 5 ml of sulfuric acid solution (95,4 ± 0,5 %) for 2 min and allow to stand for 5 min.

The colour of the acid layer shall not be more intense than that of the standard solution K (see GM 18).

R 95.2.7 Sulfur compounds

Take 2 g (2,3 ml) of the sample and apply GM 21.

Prepare a standard matching solution, using 6 ml of the sulfur SS II ($6\ \text{ml} \triangleq 0{,}003\ \%\ \text{S}$).

R 95.2.8 Thiophene and homologues

Carefully add 5 ml of the sample to form a separate layer over 5 ml of the isatin (IS 4.3.4*)[24] in a clean, dry test tube, and allow to stand for 1 h.

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[23] This reagent is generally a mixture of the isomeric xylenes and ethylbenzene.

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

R 95.2.9 Benzene and toluene

Apply GM 34, using the following conditions:

Stationary phase	10 % of a mixture (50/50) of didodecyl-phthalate with bentone 34
Support	Embacel [0,15 to 0,25 mm (60 to 100 mesh ASTM)]
Column length	6 m
Column internal diameter	2 mm
Column material	Stainless steel
Column temperature	100 °C
Injection temperature	200 °C
Detection temperature	250 °C
Type of detector	Thermal conductivity
Carrier gas	Helium
Flow rate	20 ml/min
Test portion	5 µl

R 95.2.10 Water

Take 10 g (11,6 ml) of the sample and apply GM 12, using 20 ml of the methanol (R 18) as the solvent.

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[24] IUPAC name: indoline-2,3-dione.

R 96 Zinc chloride $ZnCl_2$

Relative molecular mass: 136,30

R 96.1 Specification

Assay ($ZnCl_2$)	98 % min.
Oxide chloride (expressed as ZnO)	1,2 % max.
Sulfate (SO_4)	0,002 % max.
Total nitrogen (N)	0,002 % max.
Cadmium (Cd)	0,000 5 % max.
Calcium (Ca)	0,001 % max.
Copper (Cu)	0,001 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,001 % max.
Sodium (Na)	0,005 % max.

R 96.2 Preparation of test solution

Dissolve 20 g of the sample in water, add a few drops of hydrochloric acid solution (25 %) to clarify the solution and dilute to 100 ml (the solution shall be clear and colourless).

R 96.3 Tests

R 96.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,3 g of the sample, dissolve in 50 ml of water, and add a few drops of hydrochloric acid solution (25 %) to clarify the solution. Add 3 g of potassium sodium tartrate tetrahydrate, neutralize with the ammonia solution (R 3) and add 1 ml in excess. Add 50 mg of mordant black 11 (4.3.8*) and titrate with standard volumetric EDTA solution, $c(\text{EDTA}) = 0{,}1$ mol/l, to a blue colour.

1,00 ml of EDTA solution, $c(\text{EDTA}) = 0{,}100$ mol/l, corresponds to 0,013 628 g of $ZnCl_2$.

R 96.3.2 Oxide chloride

Weigh, to the nearest 0,000 1 g, about 10 g of the sample, dissolve in 100 ml of water and titrate with standard volumetric hydrochloric acid solution, $c(\text{HCl}) = 1$ mol/l, using the methyl orange (IS 4.3.5*).

The volume of titrant shall not exceed 3 ml.

1,00 ml of hydrochloric acid solution, $c(\text{HCl}) = 1{,}000$ mol/l, corresponds to 0,040 69 g of ZnO.

R 96.3.3 Sulfate

Take 10 ml of the test solution (R 96.2) 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 96.3.4 Total nitrogen

Take 5 ml of the test solution (R 96.2), dilute to 140 ml with water and apply GM 6.

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

R 96.3.5 Cadmium, 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 hydrochloric acid solution (0,36 %).

Working electrode	Hg
Reference electrode	Ag/AgCl, saturated KCl
Starting potential	– 0,75 V
Peak potentials	Cd: – 0,55 V Cu: – 0,07 V Pb: – 0,36 V
Evaluation	By standard solution additions

R 96.3.6 Calcium

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	4 % solution of the sample in hydrochloric acid solution (2 %)	Air-ethine	422,7

R 96.3.7 Iron

Take 10 ml of the test solution (R 96.2) and apply GM 8.1. Before diluting to 50 ml add 10 ml of the ammonia solution (R 3).

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

R 96.3.8 Sodium

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

Element	Concentration of solution	Flame	Wavelength nm
Na	4 % solution of the sample in hydrochloric acid solution (2 %)	Air-ethine	589,0

R 97 Zinc sulfate heptahydrate $ZnSO_4.7H_2O$

Relative molecular mass: 287,56

R 97.1 Specification

Assay ($ZnSO_4.7H_2O$)	99,5 % min.
pH (5 % solution)	4,4 to 6,0
Chloride (Cl)	0,000 5 % max.
Total nitrogen (N)	0,001 % max.
Arsenic (As)	0,000 05 % max.
Cadmium (Cd)	0,000 5 % max.
Calcium (Ca)	0,001 % max.
Copper (Cu)	0,001 % max.
Iron (Fe)	0,000 5 % max.
Lead (Pb)	0,001 % max.
Manganese (Mn)	0,000 3 % max.
Sodium (Na)	0,005 % max.

R 97.2 Preparation of test solution

Dissolve 50 g of the sample in water and dilute to 250 ml (the solution shall be clear and colourless).

R 97.3 Tests

R 97.3.1 Assay

Weigh, to the nearest 0,000 1 g, about 0,7 g of the sample, dissolve in 50 ml of water, and add 3 g of potassium sodium tartrate tetrahydrate. Neutralize with the ammonia solution (R 3) and add 1 ml in excess. Add 50 mg of mordant black 11 mixture (4.3.8*) and titrate with standard volumetric EDTA solution, $c(\text{EDTA}) = 0{,}1$ mol/l, to a blue colour.

1,00 ml of EDTA solution, $c(\text{EDTA}) = 0{,}100$ mol/l, corresponds to 0,028 754 g of $ZnSO_4.7H_2O$.

R 97.3.2 pH

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

R 97.3.3 Chloride

Take 25 ml of the test solution (R 97.2) and apply GM 2.

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

R 97.3.4 Total nitrogen

Take 10 ml of the test solution (R 97.2), dilute to 140 ml with water and apply GM 6.

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

R 97.3.5 Arsenic

Take 50 ml of the test solution (R 97.2) and apply GM 11.

Prepare a standard matching solution, using 5 ml of the arsenic SS III (5 ml $\triangleq$ 0,000 05 % As).

R 97.3.6 Cadmium, 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 hydrochloric acid solution (0,36 %).

Working electrode	Hg
Reference electrode	Ag/AgCl, saturated KCl
Starting potential	– 0,75 V
Peak potentials	Cd: – 0,55 V Cu: – 0,07 V Pb: – 0,36 V
Evaluation	By standard solution additions

R 97.3.7 Calcium and manganese

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	4 %	Air-ethine	422,7
Mn			279,5

R 97.3.8 Iron

Take 20 ml of the test solution (R 97.2) and apply GM 8.1. Before diluting to 50 ml add 10 ml of the ammonia solution (R 3).

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

R 97.3.9 Sodium

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

Element	Concentration of solution	Flame	Wavelength nm
Na	4 %	Air-ethine	589,0

Publications referred to

BS 6337, *General methods of chemical analysis.*
BS 6337-3, *Method for determination of iron content (1,10-phenanthroline spectrophotometric method).*
BS 6376, *Reagents for chemical analysis.*
BS 6376-1, *Methods of test.*
BS 6376-2, *Specifications (first series).*

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