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International Standard



6353/2

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

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

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## Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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

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

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

India  
USSR

## Contents

	Page
1 Scope and field of application	1
2 Reagents (abbreviation : R) — Specifications — First series	1
R 1 Acetic acid	2
R 2 Acetone	3
R 3 Ammonia solution	4
R 4 Ammonium acetate	6
R 5 Ammonium chloride	7
R 6 Barium chloride dihydrate	8
R 7 Chloroform	9
R 8 Citric acid monohydrate	10
R 9 Copper(II) sulphate pentahydrate	12
R 10 Cyclohexane	13
R 11 Ethanol	14
R 12 (Ethylenedinitrilo)tetraacetic acid, disodium salt dihydrate	16
R 13 Hydrochloric acid	17
R 14 Hydrogen peroxide	19
R 15 Hydroxylammonium chloride	21
R 16 Magnesium chloride hexahydrate	22
R 17 Magnesium oxide	23
R 18 Methanol	25
R 19 Nitric acid	26
R 20 Oxalic acid dihydrate	27
R 21 Perchloric acid	28
R 22 Phosphoric acid	30
R 23 Potassium dichromate	31
R 24 Potassium hydroxide	32
R 25 Potassium iodide	34
R 26 Potassium permanganate	36
R 27 Potassium sodium tartrate tetrahydrate	37
R 28 Silver nitrate	38
R 29 Sodium acetate trihydrate	39
R 30 Sodium carbonate anhydrous	41
R 31 Sodium carbonate decahydrate	43
R 32 Sodium chloride	45
R 33 Disodium hydrogenphosphate dodecahydrate	47
R 34 Sodium hydroxide	48
R 35 Sodium sulfate, anhydrous	50
R 36 Sodium thiosulfate pentahydrate	51
R 37 Sulfuric acid	52
R 38 Tin(II) chloride dihydrate	54
R 39 Toluene	55
R 40 Zinc	56

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

## 1 Scope and field of application

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

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

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

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

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

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

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

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

## R 1 Acetic acid CH<sub>3</sub>COOH

Relative molecular mass : 60,05

### R 1.1 Specification

Assay (CH <sub>3</sub> COOH) .....	99,7 % min.
Density : 1,05 g/ml	
Chloride (Cl) .....	0,000 1 % max.
Sulfate (SO <sub>4</sub> ) .....	0,000 2 % max.
Copper (Cu) .....	0,000 05 % max.
Iron (Fe) .....	0,000 1 % max.
Lead (Pb) .....	0,000 05 % max.
Dichromate-reducing substances (expressed as O) .....	0,008 % max.
Residue after evaporation .....	0,003 % max.

### R 1.2 Preparation of test solution

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

### R 1.3 Tests

#### R 1.3.1 Assay

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

1,00 ml of sodium hydroxide solution,  $c(\text{NaOH}) = 1,000 \text{ mol/l}$ , corresponds to 0,060 05 g of CH<sub>3</sub>COOH.

#### R 1.3.2 Chloride

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

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

#### R 1.3.3 Sulfate

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

Prepare a standard matching solution, using 5 ml of the sulfate SS II (5 ml  $\cong$  0,000 2 % SO<sub>4</sub>).

### R 1.3.4 Copper and lead

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

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

### R 1.3.5 Iron

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

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

### R 1.3.6 Dichromate-reducing substances

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

Add 10 g (9,5 ml) of the sample and allow to stand for 1 h at  $50 \pm 2 \text{ }^\circ\text{C}$ . Dilute to 50 ml with water, allow to cool to ambient temperature, add 5 ml of potassium iodide solution (10 %) and titrate with standard volumetric sodium thiosulfate solution,  $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,05 \text{ mol/l}$ , using the starch (IS 4.3.11\*).

Carry out in parallel a blank test.

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

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

where

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

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

0,000 4 is the mass, in grams, of oxygen corresponding to 1,00 ml of sodium thiosulfate solution,  $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,050 \text{ mol/l}$ .

### R 1.3.7 Residue after evaporation

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

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

## R 2 Acetone

### CH<sub>3</sub>COCH<sub>3</sub>

Relative molecular mass : 58,08

#### R 2.1 Specification

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

#### R 2.2 Tests

##### R 2.2.1 Assay and methanol

Apply GM 34, using the following conditions :

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

##### R 2.2.2 Density

Apply GM 24.1.

##### R 2.2.3 Residue after evaporation

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

The mass of the residue shall not exceed 1 mg.

##### R 2.2.4 Acidity

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

The volume of titrant shall not exceed 4 ml.

##### R 2.2.5 Alkalinity

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

The volume of titrant shall not exceed 4 ml.

##### R 2.2.6 Permanganate-reducing substances

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

The pink colour shall not be completely discharged.

##### R 2.2.7 Aldehydes

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

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

##### R 2.2.8 Water

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

## R 3 Ammonia solution (25 %)

### NH<sub>3</sub>

Relative molecular mass : 17,03

#### R 3.1 Specification

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

#### R 3.2 Preparation of test solutions and of mixed indicator solution

##### R 3.2.1 Test solution I

Evaporate 250 g (275 ml) of the sample<sup>1)</sup> to a volume of about 10 ml. Acidify with the nitric acid (R 19) and dilute to 100 ml with water.

##### R 3.2.2 Test solution II

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

##### R 3.2.3 Mixed indicator solution

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

#### R 3.3 Tests

##### R 3.3.1 Assay

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

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

1,00 ml of sulfuric acid solution,  $c(1/2 \text{ H}_2\text{SO}_4) = 1,000 \text{ mol/l}$ , corresponds to 0,017 03 g of NH<sub>3</sub>.

##### R 3.3.2 Carbonate

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

After 5 min, any turbidity in the test solution shall not be greater than that of a similarly prepared standard matching solution using 10 ml of the carbonate SS II (10 ml  $\cong$  0,002 % CO<sub>2</sub>).

##### R 3.3.3 Chloride

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

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

##### R 3.3.4 Phosphate

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

Prepare a standard matching solution, using 10 ml of the phosphate SS II (10 ml  $\cong$  0,000 2 % PO<sub>4</sub>).

##### R 3.3.5 Silicate

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

<sup>1)</sup> The same quality is also commercially available with an NH<sub>3</sub> content of about 30 % or 35 % and allowance should be made for this when preparing test solution I.

### R 3 Ammonia solution (25 %)

Prepare a standard matching solution, using 20 ml of the silicate SS II (20 ml  $\cong$  0,001 % SiO<sub>2</sub>).

#### R 3.3.6 Sulfate

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

Prepare a standard matching solution, using 5 ml of the sulfate SS II (5 ml  $\cong$  0,000 2 % SO<sub>4</sub>).

#### R 3.3.7 Sulfide

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

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

#### R 3.3.8 Calcium, copper, magnesium and lead

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

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

#### R 3.3.9 Iron

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

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

#### R 3.3.10 Potassium and sodium

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

Element	Concentration of solution	Flame	Wavelength nm
K	Test solution II (R 3.2.2)	Oxygen-acetylene	766,5
Na			589,0

#### R 3.3.11 Permanganate-reducing substances

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

The pink colour shall not be completely discharged.

#### R 3.3.12 Residue after evaporation

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

The mass of the residue shall not exceed 2 mg.

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

## R 4 Ammonium acetate CH<sub>3</sub>COONH<sub>4</sub>

Relative molecular mass : 77,08

### R 4.1 Specification

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

### R 4.2 Preparation of test solution

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

### R 4.3 Tests

#### R 4.3.1 Assay

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

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

1,00 ml of sodium hydroxide solution,  $c(\text{NaOH}) = 1,000 \text{ mol/l}$ , corresponds to 0,077 08 g of CH<sub>3</sub>COONH<sub>4</sub>.

#### R 4.3.2 pH

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

#### R 4.3.3 Chloride

Take 20 ml of the test solution (R 4.2) and apply GM 2.

Prepare a standard matching solution, using 10 ml of the test solution and 1 ml of the chloride SS II (1 ml  $\cong$  0,000 5 % Cl).

#### R 4.3.4 Nitrate

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

The yellow coloration of the test solution shall not be more intense than that of a similarly prepared standard matching solution using 1 ml of the nitrate SS II (1 ml  $\cong$  0,001 % NO<sub>3</sub>).

#### R 4.3.5 Sulfate

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

Prepare a standard matching solution, using 4 ml of the sulfate SS II (4 ml  $\cong$  0,002 % SO<sub>4</sub>).

#### R 4.3.6 Copper and lead

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

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

#### R 4.3.7 Iron

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

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

#### R 4.3.8 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 1 mg.

## R 5 Ammonium chloride NH<sub>4</sub>Cl

Relative molecular mass : 53,49

### R 5.1 Specification

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

### R 5.2 Preparation of test solution

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

### R 5.3 Tests

#### R 5.3.1 Assay

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

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

1,00 ml of sodium hydroxide solution,  $c(\text{NaOH}) = 1,000 \text{ mol/l}$ , corresponds to 0,053 49 g of NH<sub>4</sub>Cl.

#### R 5.3.2 pH

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

#### R 5.3.3 Phosphate

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

Prepare a standard matching solution, using 2,5 ml of the phosphate SS II (2,5 ml  $\cong$  0,000 5 % PO<sub>4</sub>).

#### R 5.3.4 Sulfate

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

Prepare a standard matching solution, using 4 ml of the sulfate SS II (4 ml  $\cong$  0,002 % SO<sub>4</sub>).

#### R 5.3.5 Calcium and magnesium

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

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

#### R 5.3.6 Iron

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

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

#### R 5.3.7 Potassium and sodium

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

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

#### R 5.3.8 Residue after ignition

Take 2 g of the sample and apply GM 15.

The mass of the residue shall not exceed 1 mg.

## R 6 Barium chloride dihydrate BaCl<sub>2</sub>·2H<sub>2</sub>O

Relative molecular mass : 244,28

### R 6.1 Specification

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

### R 6.2 Tests

#### R 6.2.1 Assay

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

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

1,00 ml of EDTA solution,  $c(\text{EDTA}) = 0,100 \text{ mol/l}$ , corresponds to 0,024 43 g of BaCl<sub>2</sub>·2H<sub>2</sub>O.

#### R 6.2.2 pH

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

#### R 6.2.3 Total nitrogen

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

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

#### R 6.2.4 Calcium, iron, lead and strontium

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

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

#### R 6.2.5 Potassium and sodium

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

Element	Concentration of solution	Flame	Wavelength nm
K	2 %	Oxygen-acetylene	766,5
Na			589,0

## R 7 Chloroform CHCl<sub>3</sub>

Relative molecular mass : 119,38

### R 7.1 Specification

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

### R 7.2 Test

#### R 7.2.1 Assay and ethanol

Apply GM 34, using the following conditions :

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

#### R 7.2.2 Density

Apply GM 24.1.

#### R 7.2.3 Residue after evaporation

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

The mass of the residue shall not exceed 1 mg.

#### R 7.2.4 Acidity

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

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

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

#### R 7.2.5 Readily carbonizable substances

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

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

#### R 7.2.6 Carbonyl compounds

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

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

#### R 7.2.7 Free chlorine

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

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

#### R 7.2.8 Chloride

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

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

#### R 7.2.9 Metallic impurities

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

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

## R 8 Citric acid monohydrate

### $C_6H_8O_7 \cdot H_2O$

Relative molecular mass : 210,14

#### R 8.1 Specification

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

#### R 8.2 Preparation of test solutions and of reference solution

##### R 8.2.1 Test solution I

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

##### R 8.2.2 Test solution II

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

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

##### R 8.2.3 Test solution III

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

##### R 8.2.4 Reference solution

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

#### R 8.3 Tests

##### R 8.3.1 Assay

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

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

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

##### R 8.3.2 Chloride

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

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

##### R 8.3.3 Oxalate

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

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

##### R 8.3.4 Phosphate

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

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

##### R 8.3.5 Sulfate

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

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

##### R 8.3.6 Copper and lead

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

Element	Concentration of solution	Flame	Resonance line nm
Cu	Test solution III (R 8.2.3)	Air-acetylene	324,7
Pb			217,0 or 283,3

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## R 8 Citric acid monohydrate

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### R 8.3.7 Iron

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

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

### R 8.3.8 Readily carbonizable substances

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

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

### R 8.3.9 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 2 mg.

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

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## R 9 Copper(II) sulfate pentahydrate CuSO<sub>4</sub>·5H<sub>2</sub>O

Relative molecular mass : 249,68

### R 9.1 Specification

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

### R 9.2 Preparation of test solution

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

### R 9.3 Tests

#### R 9.3.1 Assay

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

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

1,00 ml of sodium thiosulfate solution,  $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,100 \text{ mol/l}$ , corresponds to 0,024 97 g of CuSO<sub>4</sub>·5H<sub>2</sub>O.

#### R 9.3.2 Chloride

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

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

#### R 9.3.3 Total nitrogen

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

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

#### R 9.3.4 Iron

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

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

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

#### R 9.3.5 Nickel and zinc

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

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

#### R 9.3.6 Potassium and sodium

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

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

## R 10 Cyclohexane C<sub>6</sub>H<sub>12</sub>

Relative molecular mass : 84,16

### R 10.1 Specification

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

### R 10.2 Tests

#### R 10.2.1 Assay, cyclohexene and benzene

Apply GM 34, using the following conditions :

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

### R 10.2.2 Density

Apply GM 24.1.

### R 10.2.3 Freezing point

Apply GM 25.3.

### R 10.2.4 Residue after evaporation

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

The mass of the residue shall not exceed 2 mg.

### R 10.2.5 Readily carbonizable substances

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

No darkening of the lower acid phase shall occur.

### R 10.2.6 Water

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

## R 11 Ethanol C<sub>2</sub>H<sub>5</sub>OH

Relative molecular mass : 46,07

### R 11.1 Specification

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

### R 11.2 Tests

#### R 11.2.1 Assay, methanol and 2-propanol

Apply GM 34, using the following conditions :

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

#### R 11.2.2 Density

Apply GM 24.1.

#### R 11.2.3 Residue after evaporation

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

The mass of the residue shall not exceed 1 mg.

#### R 11.2.4 Acidity

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

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

#### R 11.2.5 Alkalinity

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

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

#### R 11.2.6 Permanganate-reducing substances

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

The pink colour shall not be completely discharged.

#### R 11.2.7 Readily carbonizable substances

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

The resulting solution shall be colourless.

#### R 11.2.8 Higher alcohols

Apply GM 34, using the following conditions :

Stationary phase .....	8 % Reoplex 400 Chromosorb G-AW-DMCS [0,125 to 0,150 mm (100 to 120 mesh ASTM)]
Column length.....	3 m
Column internal diameter.....	2,5 mm
Column material .....	Stainless steel
Column temperature .....	80 to 200 °C
Temperature program .....	2 °C/min
Injection temperature .....	250 °C
Detection temperature .....	250 °C
Type of detector .....	Flame ionization

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

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**R 11 Ethanol**

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Carrier gas ..... Nitrogen  
Flow rate ..... 25 ml/min  
Test portion ..... 1  $\mu$ l

**R 11.2.9 Carbonyl compounds**

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

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

**R 11.2.10 Water**

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

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

Relative molecular mass : 372,24

### R 12.1 Specification

pH (5 % solution) : 4 to 5

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

### R 12.2 Tests

#### R 12.2.1 pH

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

#### R 12.2.2 Assay

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

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

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

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

#### R 12.2.3 Chloride

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

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

#### R 12.2.4 Nitrilotriacetic acid

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

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

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

#### R 12.2.5 Sulfate

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

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

#### R 12.2.6 Copper, iron and lead

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

Element	Concentration of solution	Flame	Resonance line nm
Cu	10 %	Air-acetylene	324,7
Fe			248,3
Pb			217,0 or 283,3

## R 13 Hydrochloric acid HCl

Relative molecular mass : 36,46

### R 13.1 Specification

Coloration in Hazen units . . . . .	10 max.
Assay (HCl) . . . . .	35 % min. <sup>1)</sup>
Free chlorine (Cl) . . . . .	0,000 2 % max.
Sulfate (SO <sub>4</sub> ) . . . . .	0,000 2 % max.
Sulfite (SO <sub>3</sub> ) . . . . .	0,000 2 % max.
Ammonium (NH <sub>4</sub> ) . . . . .	0,000 3 % max.
Arsenic (As) . . . . .	0,000 005 % max.
Copper (Cu) . . . . .	0,000 01 % max.
Lead (Pb) . . . . .	0,000 02 % max.
Iron (Fe) . . . . .	0,000 05 % max.
Sulfated ash . . . . .	0,000 5 % max.

### R 13.2 Preparation of test solution

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

### R 13.3 Tests

#### R 13.3.1 Coloration in Hazen units

Apply GM 36.

#### R 13.3.2 Assay

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

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

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

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

#### R 13.3.3 Free chlorine

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

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

#### R 13.3.4 Sulfate

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

Prepare a standard matching solution, using 8 ml of the sulfate SS II (8 ml  $\cong$  0,000 2 % SO<sub>4</sub>).

#### R 13.3.5 Sulfite

In a 500 ml conical flask, mix 50 ml of water, 1 ml of potassium iodide solution (10 %) and 5 ml of the sample. Add 2 ml of the starch (IS 4.3.11\*) and, if necessary, add, drop by drop, standard volumetric iodine solution,  $c(1/2 \text{ I}_2) = 0,01 \text{ mol/l}$ , until a blue coloration just appears. Then add 100 g (84 ml) of the sample and titrate with the same iodine solution until a blue coloration reappears.

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

#### R 13.3.6 Ammonium

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

Prepare a standard matching solution, using 2 ml of the ammonium SS II (2 ml  $\cong$  0,000 3 % NH<sub>4</sub>).

#### R 13.3.7 Arsenic

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

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

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

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

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**R 13 Hydrochloric acid**


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**R 13.3.8 Copper and lead**

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

Element	Concentration of solution	Flame	Resonance line nm
Cu	50 ml of the test solution (R 13.2) diluted to 100 ml	Air-acetylene	324,7
Pb			217,0 or 283,3

**R 13.3.9 Iron**

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

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

**R 13.3.10 Sulfated ash**

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

The mass of the residue shall not exceed 1 mg.

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## R 14 Hydrogen peroxide H<sub>2</sub>O<sub>2</sub>

Relative molecular mass : 34,01

### R 14.1 Specification

Assay (H <sub>2</sub> O <sub>2</sub> ) .....	30 % min. <sup>1)</sup>
Acidity (expressed in millimoles of H <sup>+</sup> ) ...	0,3/100 g max.
Chloride (Cl) .....	0,000 5 % max.
Phosphate (PO <sub>4</sub> ) .....	0,000 5 % max.
Sulfate (SO <sub>4</sub> ) .....	0,000 5 % max.
Total nitrogen (N) .....	0,002 % max.
Copper (Cu) .....	0,000 01 % max.
Iron (Fe) .....	0,000 02 % max.
Lead (Pb) .....	0,000 02 % max.
Residue after evaporation .....	0,005 % max.

### R 14.2 Tests

#### R 14.2.1 Assay

Weigh, to the nearest 0,000 1 g, 0,20 g of the sample, mix with 50 ml of water and 20 ml of sulfuric acid solution (16 %) and titrate with standard volumetric potassium permanganate solution,  $c(1/5 \text{ KMnO}_4) = 0,1 \text{ mol/l}$ , to a faint pink colour.

1,00 ml of potassium permanganate solution,  $c(1/5 \text{ KMnO}_4) = 0,100 \text{ mol/l}$ , corresponds to 0,001 700 7 g of H<sub>2</sub>O<sub>2</sub>.

#### R 14.2.2 Acidity

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

The volume of titrant shall not exceed 3 ml.

#### R 14.2.3 Chloride

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

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

#### R 14.2.4 Phosphate

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

Prepare a standard matching solution using 5 ml of the phosphate SS II (5 ml  $\cong$  0,000 5 % PO<sub>4</sub>).

#### R 14.2.5 Sulfate

Take 10 g (9 ml) of the sample, add 15 ml of water and apply GM 3.

Prepare a standard matching solution, using 5 ml of the sulfate SS II (5 ml  $\cong$  0,000 5 % SO<sub>4</sub>).

#### R 14.2.6 Total nitrogen

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

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

#### R 14.2.7 Copper and lead

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

Preparation of the sample :

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

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

#### R 14.2.8 Iron

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

Preparation of the sample :

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

1) A suitable stabilizer may be used.

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**R 14 Hydrogen peroxide**

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

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

**R 14.2.9 Residue after evaporation**

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

The mass of the residue shall not exceed 5 mg.

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## R 15 Hydroxylammonium chloride HONH<sub>3</sub>Cl

Relative molecular mass : 69,49

### R 15.1 Specification

Assay (HONH <sub>3</sub> Cl) .....	98 % min.
pH (5 % solution) : 2,5 to 4,0	
Sulfate (SO <sub>4</sub> ) .....	0,002 % max.
Ammonium (NH <sub>4</sub> ) .....	0,1 % max.
Copper (Cu) .....	0,001 % max.
Iron (Fe) .....	0,000 5 % max.
Lead (Pb) .....	0,001 % max.
Residue after ignition (at 600 °C) .....	0,01 % max.

### R 15.2 Preparation of test solution

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

### R 15.3 Tests

#### R 15.3.1 Assay

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

Take 20,00 ml of this solution, add a mixture of 20 ml ammonium iron(III) sulfate solution [25 % NH<sub>4</sub>Fe(SO<sub>4</sub>).12H<sub>2</sub>O] and 6 ml of sulfuric acid solution (25 %) and heat to boiling. After 5 min, dilute to 250 ml with freshly boiled and cooled water. Heat to 60 °C and, after addition of 2 ml of the phosphoric acid (R 22), titrate with standard volumetric potassium permanganate solution,  $c(1/5 \text{ KMnO}_4) = 0,1 \text{ mol/l}$ , to a faint pink colour.

1,00 ml of potassium permanganate solution,  $c(1/5 \text{ KMnO}_4) = 0,100 \text{ mol/l}$ , corresponds to 0,003 475 g of HONH<sub>3</sub>Cl.

#### R 15.3.2 pH

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

#### R 15.3.3 Sulfate

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

Prepare a standard matching solution, using 4 ml of the sulfate SS II (4 ml  $\cong$  0,002 % SO<sub>4</sub>).

#### R 15.3.4 Ammonium

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

No change in the solution shall occur within 15 min.

#### R 15.3.5 Copper and lead

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

Element	Concentration of solution	Flame	Resonance line nm
Cu	10 %	Air-acetylene	324,7
Pb			217,0 or 283,3

#### R 15.3.6 Iron

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

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

#### R 15.3.7 Residue after ignition

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

The mass of the residue shall not exceed 1 mg.

## R 16 Magnesium chloride hexahydrate MgCl<sub>2</sub>·6H<sub>2</sub>O

Relative molecular mass : 203,31

### R 16.1 Specification

Assay (MgCl <sub>2</sub> ·6H <sub>2</sub> O) .....	98 % min.
pH (5 % solution) : 5,0 to 6,5	
Phosphate (PO <sub>4</sub> ) .....	0,000 5 % max.
Sulfate (SO <sub>4</sub> ) .....	0,002 % max.
Total nitrogen (N) .....	0,002 % max.
Barium (Ba) .....	0,005 % max.
Calcium (Ca) .....	0,01 % max.
Copper (Cu) .....	0,000 5 % max.
Iron (Fe) .....	0,000 5 % max.
Lead (Pb) .....	0,000 5 % max.
Potassium (K) .....	0,005 % max.
Sodium (Na) .....	0,005 % max.

### R 16.2 Preparation of test solution

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

### R 16.3 Tests

#### R 16.3.1 Assay

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

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

1,00 ml of EDTA solution,  $c(1/2 \text{ EDTA}) = 0,200 \text{ mol/l}$ , corresponds to 0,020 33 g of MgCl<sub>2</sub>·6H<sub>2</sub>O.

#### R 16.3.2 pH

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

#### R 16.3.3 Phosphate

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

Prepare a standard matching solution, using 5 ml of the phosphate SS II (5 ml  $\cong$  0,000 5 % PO<sub>4</sub>).

#### R 16.3.4 Sulfate

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

Prepare a standard matching solution, using 8 ml of the sulfate SS II (8 ml  $\cong$  0,002 % SO<sub>4</sub>).

#### R 16.3.5 Total nitrogen

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

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

#### R 16.3.6 Calcium, copper, iron and lead

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	5 %	Air-acetylene	422,7
Cu	10 % Use the extraction solvent in GM 35		324,7
Fe			248,3
Pb			217,0 or 283,3

#### R 16.3.7 Barium, potassium and sodium

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

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

## R 17 Magnesium oxide MgO

Relative molecular mass : 40,30

### R 17.1 Specification

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

### R 17.2 Preparation of test solution

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

### R 17.3 Tests

#### R 17.3.1 Assay

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

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

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

#### R 17.3.2 Substances insoluble in hydrochloric acid

Dissolve 20 g of the sample in 40 ml of water and 150 ml of hydrochloric acid solution (25 %).

Heat to boiling, cool, filter through a sintered-glass filter crucible (porosity No. 40 — pore size diameter 16 to 40  $\mu\text{m}$ ), retaining the filtrate for the preparation of the test solution (R 17.2), and dry the residue to constant mass at 105 °C.

The mass of the residue shall not exceed 2 mg.

#### R 17.3.3 Substances soluble in water

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

The mass of the residue shall not exceed 10 mg.

#### R 17.3.4 Carbonate

To 1 g of the sample in a distillation apparatus, add about 80 ml of sulfuric acid solution (16 %), and absorb the carbon dioxide formed in an absorption flask containing a mixture (3:1:3 by volume) of 1,4-dioxane-benzylamine-ethanol [95 % (V/V)]. Titrate with standard volumetric tetrabutylammonium hydroxide solution,  $c[(\text{C}_4\text{H}_9)_4\text{NOH}] = 0,1 \text{ mol/l}$ , using thymol blue solution (0,2 % in 1,4-dioxane) as indicator.

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

#### R 17.3.5 Chloride

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

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

#### R 17.3.6 Sulfate

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

Prepare a standard matching solution, using 10 ml of the sulfate SS II (10 ml  $\cong$  0,02 % SO<sub>4</sub>).

#### R 17.3.7 Total nitrogen

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

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

**R 17 Magnesium oxide**

**R 17.3.8 Arsenic**

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

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

**R 17.3.9 Barium and strontium**

Mix 0,25 ml of barium chloride dihydrate solution [0,02 % BaCl<sub>2</sub>·2H<sub>2</sub>O in 30 % (V/V) ethanol] and 1 ml of sodium sulfate decahydrate solution (40 % Na<sub>2</sub>SO<sub>4</sub>·10H<sub>2</sub>O) (seeding solution). To this mixture add, after exactly 1 min, 10 ml of the test solution (R 17.2), which has been previously acidified with 0,5 ml of hydrochloric acid solution (25 %), and 10 ml of water.

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

**R 17.3.10 Calcium, copper and zinc**

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

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

**R 17.3.11 Heavy metals**

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

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

**R 17.3.12 Iron**

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

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

**R 17.3.13 Potassium and sodium**

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

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

**R 17.3.14 Loss after ignition**

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

The loss in mass shall not exceed 20 mg.

## R 18 Methanol CH<sub>3</sub>OH

Relative molecular mass : 32,04

### R 18.1 Specification

Assay (CH <sub>3</sub> OH) .....	99,8 % min.
Density : 0,791 to 0,793 g/ml	
Residue after evaporation .....	0,001 % max.
Acidity (expressed in millimoles of H <sup>+</sup> ) ...	0,065/100 g max.
Alkalinity (expressed in millimoles of OH <sup>-</sup> )	0,02/100 g max.
Permanganate-reducing substances (expressed as O) .....	0,000 5 % max.
Readily carbonizable substances .....	To pass test
Carbonyl compounds (expressed as CO) ..	0,005 % max.
Water .....	0,2 % max.

### R 18.2 Tests

#### R 18.2.1 Assay

Apply GM 34, using the following conditions :

Stationary phase .....	20 % Carbowax 400
Support .....	Chromosorb W [0,18 to 0,25 mm (60 to 80 mesh ASTM)]
Column length .....	2 m
Column internal diameter .....	5 mm
Column material .....	Stainless steel
Column temperature .....	50 °C
Injection temperature .....	150 °C
Detection temperature .....	150 °C
Type of detector .....	Thermal conductivity
Carrier gas .....	Helium
Flow rate .....	30 ml/min
Test portion .....	5 µl

#### R 18.2.2 Density

Apply GM 24.1.

#### R 18.2.3 Residue after evaporation

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

The mass of the residue shall not exceed 1 mg.

#### R 18.2.4 Acidity

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

The volume of titrant shall not exceed 5,2 ml.

#### R 18.2.5 Alkalinity

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

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

#### R 18.2.6 Permanganate-reducing substances

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

The pink colour shall not be completely discharged.

#### R 18.2.7 Readily carbonizable substances

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

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

#### R 18.2.8 Carbonyl compounds

Take 4 g (5 ml) of the sample and dilute to 20 ml with the carbonyl-free methanol (RS 4.2.11\*). Take 2 ml of this solution and apply GM 23.

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

#### R 18.2.9 Water

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

## R 19 Nitric acid HNO<sub>3</sub>

Relative molecular mass : 63,01

### R 19.1 Specification

Assay (HNO <sub>3</sub> ) .....	65 % min. <sup>1)</sup>
Chloride (Cl) .....	0,000 05 % max.
Sulfate (SO <sub>4</sub> ) .....	0,000 2 % max.
Arsenic (As) .....	0,000 001 % max.
Copper (Cu) .....	0,000 01 % max.
Iron (Fe) .....	0,000 02 % max.
Lead (Pb) .....	0,000 01 % max.
Sulfated ash .....	0,001 % max.

### R 19.2 Preparation of test solution

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

### R 19.3 Tests

#### R 19.3.1 Assay

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

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

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

1,00 ml of sodium hydroxide solution,  $c(\text{NaOH}) = 1,000 \text{ mol/l}$ , corresponds to 0,063 01 g of HNO<sub>3</sub>.

#### R 19.3.2 Chloride

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

No turbidity shall be produced within 15 min.

#### R 19.3.3 Sulfate

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

Prepare a standard matching solution, using 10 ml of the sulfate SS II (10 ml  $\cong$  0,000 2 % SO<sub>4</sub>).

#### R 19.3.4 Arsenic

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

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

#### R 19.3.5 Copper and lead

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

Element	Concentration of solution	Flame	Resonance line nm
Cu	20 ml of the test solution (R 19.2) diluted to 50 ml	Air-acetylene	324,7
Pb			217,0 or 283,3

#### R 19.3.6 Iron

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

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

#### R 19.3.7 Sulfated ash

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

The mass of the residue shall not exceed 1 mg.

<sup>1)</sup> Nitric acid is commercially available also in other concentrations conforming to the above specification, which does not apply, however, to the nitric acid containing 95 to 100 % of HNO<sub>3</sub>.

## R 20 Oxalic acid dihydrate C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>·2H<sub>2</sub>O

Relative molecular mass : 126,07

### R 20.1 Specification

Assay (C <sub>2</sub> H <sub>2</sub> O <sub>4</sub> ·2H <sub>2</sub> O) .....	99,5 % min.
Chloride (Cl) .....	0,000 5 % max.
Sulfate (SO <sub>4</sub> ) .....	0,005 % max.
Total nitrogen (N) .....	0,001 % max.
Calcium (Ca) .....	0,001 % max.
Heavy metals (expressed as Pb) .....	0,000 5 % max.
Iron (Fe) .....	0,000 2 % max.
Sulfated ash .....	0,01 % max.

### R 20.2 Preparation of test solutions

#### R 20.2.1 Test solution I

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

#### R 20.2.2 Test solution II

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

#### R 20.2.3 Test solution III

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

### R 20.3 Tests

#### R 20.3.1 Assay

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

Acidify the solution with the sulfuric acid (R 37), and titrate at between 60 and 80 °C with standard volumetric potassium permanganate solution,  $c(1/5 \text{ KMnO}_4) = 0,1 \text{ mol/l}$ , to a faint pink colour.

1,00 ml of potassium permanganate solution,  $c(1/5 \text{ KMnO}_4) = 0,100 \text{ mol/l}$ , corresponds to 0,006 303 3 g of C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>·2H<sub>2</sub>O.

#### R 20.3.2 Chloride

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

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

#### R 20.3.3 Sulfate

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

Prepare a standard matching solution, using 8 ml of the sulfate SS II (8 ml  $\cong$  0,005 % SO<sub>4</sub>) and 4 ml of the test solution III (R 20.2.3).

#### R 20.3.4 Total nitrogen

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

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

#### R 20.3.5 Calcium

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

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

#### R 20.3.6 Heavy metals

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

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

#### R 20.3.7 Iron

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

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

#### R 20.3.8 Sulfated ash

Take 10 g of the sample and apply GM 16.

The mass of the residue shall not exceed 1 mg.

## R 21 Perchloric acid (72 %)

### HClO<sub>4</sub>

Relative molecular mass : 100,46

#### R 21.1 Specification

Assay (HClO <sub>4</sub> ) .....	70 to 73 % <sup>1)</sup>
Chlorate (ClO <sub>3</sub> ) .....	0,001 % max.
Chloride (Cl) .....	0,000 3 % max.
Free chlorine (Cl) .....	0,000 5 % max.
Phosphate and silicate (expressed as SiO <sub>2</sub> )	0,005 % max.
Sulfate (SO <sub>4</sub> ) .....	0,001 % max.
Total nitrogen (N) .....	0,002 % max.
Arsenic (As) .....	0,000 005 % max.
Copper (Cu) .....	0,000 01 % max.
Iron (Fe) .....	0,000 2 % max.
Lead (Pb) .....	0,000 01 % max.
Manganese (Mn) .....	0,000 05 % max.
Silver (Ag) .....	0,000 5 % max.
Sulfated ash .....	0,003 % max.

#### R 21.2 Tests

##### R 21.2.1 Assay

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

1,00 ml of sodium hydroxide solution,  $c(\text{NaOH}) = 1,000 \text{ mol/l}$ , corresponds to 0,100 5 g of HClO<sub>4</sub>.

##### R 21.2.2 Chlorate

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

After 5 min, any opalescence shall not be greater than that of a similarly prepared standard matching solution, using 8,5 ml of the chloride SS II (8,5 ml  $\cong$  0,001 % ClO<sub>3</sub>).

##### R 21.2.3 Chloride

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

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

##### R 21.2.4 Free chlorine

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

Any blue colour produced shall be discharged by the addition of 0,05 ml of sodium thiosulfate solution,  $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,005 \text{ mol/l}$ .

##### R 21.2.5 Phosphate and silicate

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

The blue coloration of the organic phase shall not be more intense than that of a similarly prepared standard matching solution, using 2,5 ml of the silicate SS I (2,5 ml  $\cong$  0,005 % SiO<sub>2</sub>).

##### R 21.2.6 Sulfate

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

Prepare a standard matching solution, using 5 ml of the sulfate SS II (5 ml  $\cong$  0,001 % SO<sub>4</sub>).

##### R 21.2.7 Total nitrogen

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

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

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**R 21 Perchloric acid (72 %)**


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

**R 21.2.8 Arsenic**

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

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

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

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

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

**R 21.2.10 Sulfated ash**

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

The mass of the residue shall not exceed 1 mg.

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## R 22 Phosphoric acid H<sub>3</sub>PO<sub>4</sub>

Relative molecular mass : 98,00

### R 22.1 Specification

Assay (H <sub>3</sub> PO <sub>4</sub> )	85 % min. <sup>1)</sup>
Chloride (Cl)	0,000 3 % max.
Sulfate (SO <sub>4</sub> )	0,005 % max.
Arsenic (As)	0,000 1 % max.
Cadmium (Cd)	0,000 5 % max.
Copper (Cu)	0,000 5 % max.
Iron (Fe)	0,001 % max.
Lead (Pb)	0,001 % max.
Manganese (Mn)	0,000 5 % max.
Nickel (Ni)	0,000 5 % max.
Potassium (K)	0,005 % max.
Sodium (Na)	0,05 % max.
Zinc (Zn)	0,001 % max.
Volatile acids (expressed in millimoles of H <sup>+</sup> )	0,02/100 g max.

### R 22.2 Tests

#### R 22.2.1 Assay

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

1,00 ml of sodium hydroxide solution,  $c(\text{NaOH}) = 1,000 \text{ mol/l}$ , corresponds to 0,049 00 g of H<sub>3</sub>PO<sub>4</sub>.

#### R 22.2.2 Chloride

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

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

#### R 22.2.3 Sulfate

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

Prepare a standard matching solution, using 10 ml of the sulfate SS II (10 ml  $\cong$  0,005 % SO<sub>4</sub>).

#### R 22.2.4 Arsenic

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

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

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

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

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

#### R 22.2.6 Potassium and sodium

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

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

#### R 22.2.7 Volatile acids

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

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

<sup>1)</sup> Phosphoric acid is commercially available also with a concentration of about 88 % of H<sub>3</sub>PO<sub>4</sub>, conforming to the above specification.

## R 23 Potassium dichromate

### $K_2Cr_2O_7$

Relative molecular mass : 294,19

#### R 23.1 Specification

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

#### R 23.2 Preparation of test solution

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

#### R 23.3 Tests

##### R 23.3.1 Assay

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

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

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

Carry out a blank test on the reagents used.

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

##### R 23.3.2 Chloride

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

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

##### R 23.3.3 Sulfate

To 10 ml of the test solution (R 23.2), add 10 ml of hydrochloric acid solution (25 %) and 20 ml of tributyl phosphate, shake vigorously for 1 min, allow to separate and

reject the organic phase. To the aqueous phase, add again 20 ml of tributyl phosphate and repeat the extraction. Wash the aqueous phase twice with 5 ml of diethyl ether and evaporate it to dryness. Dissolve the residue in 10 ml of water and apply GM 3.

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

##### R 23.3.4 Calcium, copper and lead

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

Element	Concentration of solution	Flame	Resonance line nm
Cu	10 %	Air-acetylene	324,7
Pb			217,0 or 283,3
Ca	4 %		422,7

##### R 23.3.5 Iron

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

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

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

##### R 23.3.6 Sodium

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

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

##### R 23.3.7 Loss after drying

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

The loss in mass shall not exceed 1 mg.

## R 24 Potassium hydroxide KOH

Relative molecular mass : 56,108

### R 24.1 Specification

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

### R 24.2 Preparation of test solutions

#### R 24.2.1 Test solution I

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

#### R 24.2.2 Test solution II

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

### R 24.3 Tests

#### R 24.3.1 Assay

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

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

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

Use the neutralized solution for test R 24.3.2.

#### R 24.3.2 Carbonate

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

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

#### R 24.3.3 Chloride

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

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

#### R 24.3.4 Phosphate

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

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

#### R 24.3.5 Silicate

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

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

#### R 24.3.6 Sulfate

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

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

#### R 24.3.7 Total nitrogen

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

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

## R 24 Potassium hydroxide

### R 24.3.8 Aluminium

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

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

### R 24.3.9 Iron

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

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

### R 24.3.10 Calcium, lead, magnesium, nickel and zinc

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

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

### R 24.3.11 Sodium

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

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

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## R 25 Potassium iodide KI

Relative molecular mass : 166,01

### R 25.1 Specification

Assay (KI) .....	99,5 % min.
pH (5 % solution) : 6 to 8	
Chloride and bromide (expressed as Cl) ...	0,01 % max.
Iodate and iodine (expressed as IO <sub>3</sub> ) .....	0,000 3 % max.
Phosphate (PO <sub>4</sub> ) .....	0,001 % max.
Sulfate (SO <sub>4</sub> ) .....	0,005 % max.
Total nitrogen (N) .....	0,001 % max.
Arsenic (As) .....	0,000 01 % max.
Heavy metals (expressed as Pb) .....	0,000 5 % max.
Iron (Fe) .....	0,000 5 % max.
Calcium (Ca) .....	0,001 % max.
Magnesium (Mg) .....	0,001 % max.
Sodium (Na) .....	0,05 % max.
Reducing substances .....	To pass the test

### R 25.2 Preparation of test solution

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

Protect this solution from light and use only if colourless.

### R 25.3 Tests

#### R 25.3.1 Assay

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

To this solution add 35,0 ml of standard volumetric silver nitrate solution,  $c(\text{AgNO}_3) = 0,1 \text{ mol/l}$ , and then 5 ml of the nitric acid (R 19). Titrate with standard volumetric potassium thiocyanate solution,  $c(\text{KSCN}) = 0,1 \text{ mol/l}$ , in presence of the ammonium iron(III) sulfate (IS 4.3.1\*) to the first pink coloration.

1,00 ml of silver nitrate solution,  $c(\text{AgNO}_3) = 0,100 \text{ mol/l}$ , corresponds to 0,016 6 g of KI.

#### R 25.3.2 pH

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

#### R 25.3.3 Chloride and bromide

To 2,5 ml of the test solution (R 25.2), add 10 ml of water, 2,5 ml of the ammonia solution (R 3) and then 35 ml of stan-

dard volumetric silver nitrate solution,  $c(\text{AgNO}_3) = 0,1 \text{ mol/l}$ . Stir and filter. To 10 ml of the filtrate, add 2,5 ml of nitric acid solution (25 %).

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

#### R 25.3.4 Iodate and iodine

Dissolve 2 g of the sample in 50 ml of water, and add 3 ml of standard volumetric sulfuric acid solution,  $c(1/2 \text{ H}_2\text{SO}_4) = 0,1 \text{ mol/l}$ , and 5 ml of the starch (IS 4.3.11\*).

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

#### R 25.3.5 Phosphate

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

Prepare a standard matching solution, using 2 ml of the phosphate SS II (2 ml  $\cong$  0,001 % PO<sub>4</sub>).

#### R 25.3.6 Sulfate

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

Prepare a standard matching solution, using 10 ml of the sulfate SS II (10 ml  $\cong$  0,005 % SO<sub>4</sub>).

#### R 25.3.7 Total nitrogen

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

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

#### R 25.3.8 Arsenic

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

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

#### R 25.3.9 Heavy metals

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

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

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**R 25 Potassium iodide**


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**R 25.3.10 Iron**

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

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

**R 25.3.11 Calcium and magnesium**

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	10 %	Air-acetylene	422,7
Mg	5 %		285,2

**R 25.3.12 Sodium**

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

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

**R 25.3.13 Reducing substances**

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

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

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## R 26 Potassium permanganate KMnO<sub>4</sub>

Relative molecular mass : 158,04

### R 26.1 Specification

Assay (KMnO <sub>4</sub> )	99 % min.
Water-insoluble matter	0,1 % max.
Chloride (Cl)	0,005 % max.
Sulfate (SO <sub>4</sub> )	0,005 % max.
Total nitrogen (N)	0,005 % max.
Heavy metals (expressed as Pb)	0,003 % max.
Iron (Fe)	0,002 % max.

### R 26.2 Preparation of test solution

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

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

### R 26.3 Tests

#### R 26.3.1 Assay

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

Take 50,0 ml of this solution, add 15 ml of potassium iodide solution (20 %) and 15 ml of sulfuric acid solution (20 %), and titrate the liberated iodine with standard volumetric sodium thiosulfate solution,  $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}$ , using the starch (IS 4.3.11\*).

1,00 ml of sodium thiosulfate solution,  $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,100 \text{ mol/l}$ , corresponds to 0,003 16 g of KMnO<sub>4</sub>.

#### R 26.3.2 Water-insoluble matter

Take 2 g of the sample and apply GM 1.

The mass of the residue shall not exceed 2 mg.

#### R 26.3.3 Chloride

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

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

#### R 26.3.4 Sulfate

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

Prepare a standard matching solution, using 5 ml of the sulfate SS II (5 ml  $\cong$  0,005 % SO<sub>4</sub>).

#### R 26.3.5 Total nitrogen

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

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

#### R 26.3.6 Heavy metals

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

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

#### R 26.3.7 Iron

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

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

## R 27 Potassium sodium tartrate tetrahydrate

### $C_4H_4O_6KNa \cdot 4H_2O$

Relative molecular mass : 282,23

#### R 27.1 Specification

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

#### R 27.2 Preparation of test solution

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

#### R 27.3 Tests

##### R 27.3.1 Assay

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

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

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

##### R 27.3.2 pH

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

##### R 27.3.3 Water-insoluble matter

Take 20 g of the sample and apply GM 1.

The mass of the residue shall not exceed 1 mg.

##### R 27.3.4 Chloride

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

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

##### R 27.3.5 Phosphate

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

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

##### R 27.3.6 Sulfate

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

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

##### R 27.3.7 Ammonium

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

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

##### R 27.3.8 Calcium, copper and lead

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

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

##### R 27.3.9 Iron

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

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

## R 28 Silver nitrate AgNO<sub>3</sub>

Relative molecular mass : 169,87

### R 28.1 Specification

Assay (AgNO <sub>3</sub> ) .....	99,8 % min.
Chloride (Cl) .....	0,001 % max.
Sulfate (SO <sub>4</sub> ) .....	0,002 % max.
Copper (Cu) .....	0,000 5 % max.
Iron (Fe) .....	0,000 5 % max.
Lead (Pb) .....	0,001 % max.
Substances not precipitated by hydrochloric acid (expressed as sulfates) .....	0,01 % max.

### R 28.2 Tests

#### R 28.2.1 Assay

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

To this solution add 50,00 ml of standard volumetric sodium chloride solution,  $c(\text{NaCl}) = 0,1 \text{ mol/l}$ , 0,1 ml of sulfuric acid solution (50 %), 0,25 ml of saturated potassium sulfate solution and 20 ml of ethanol [95 % (V/V)]. Titrate potentiometrically with the standard volumetric sodium chloride solution,  $c(\text{NaCl}) = 0,1 \text{ mol/l}$ , using Ag-Hg/Hg<sub>2</sub>SO<sub>4</sub> electrodes, adding the titrant in increments of 0,1 ml from a microburette.

1,00 ml of sodium chloride solution,  $c(\text{NaCl}) = 0,100 \text{ mol/l}$ , corresponds to 0,016 99 g of AgNO<sub>3</sub>.

#### R 28.2.2 Chloride

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

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

#### R 28.2.3 Sulfate

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

After 5 min, any turbidity in the test solution shall not be greater than that of a similarly prepared standard matching solution, using 2 ml of the sulfate SS II (2 ml  $\cong$  0,002 % SO<sub>4</sub>).

#### R 28.2.4 Copper, iron and lead

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

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

#### R 28.2.5 Substances not precipitated by hydrochloric acid

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

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

The mass of the residue shall not exceed 1 mg.

## R 29 Sodium acetate trihydrate CH<sub>3</sub>COONa.3H<sub>2</sub>O

Relative molecular mass : 136,08

### R 29.1 Specification

Assay (CH <sub>3</sub> COONa.3H <sub>2</sub> O) .....	99 % min.
pH (5 % solution) : 7,5 to 9	
Chloride (Cl) .....	0,000 5 % max.
Phosphate (PO <sub>4</sub> ) .....	0,000 5 % max.
Sulfate (SO <sub>4</sub> ) .....	0,002 % max.
Aluminium (Al) .....	0,000 5 % max.
Calcium (Ca) .....	0,002 % max
Copper (Cu) .....	0,000 5 % max.
Iron (Fe) .....	0,000 5 % max.
Lead (Pb) .....	0,000 5 % max.
Potassium (K) .....	0,005 % max.
Permanganate-reducing substances (expressed as HCOOH) .....	0,005 % max.

### R 29.2 Preparation of test solution

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

### R 29.3 Tests

#### R 29.3.1 Assay

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

To this solution add 0,4 ml of 1-naphtholbenzene solution [0,1 % in acetic acid (R 1)] and titrate with standard volumetric perchloric acid solution,  $c(\text{HClO}_4) = 0,1 \text{ mol/l}$ , in acetic acid (R 1), to a green colour.

1,00 ml of perchloric acid solution,  $c(\text{HClO}_4) = 0,100 \text{ mol/l}$ , corresponds to 0,013 61 g of CH<sub>3</sub>COONa.3H<sub>2</sub>O.

#### R 29.3.2 pH

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

#### R 29.3.3 Chloride

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

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

#### R 29.3.4 Phosphate

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

Prepare a standard matching solution, using 1,5 ml of the phosphate SS II (1,5 ml  $\cong$  0,000 5 % PO<sub>4</sub>).

#### R 29.3.5 Sulfate

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

Prepare a standard matching solution, using 4 ml of the sulfate SS II (4 ml  $\cong$  0,002 % SO<sub>4</sub>).

#### R 29.3.6 Aluminium

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

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

#### R 29.3.7 Calcium

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

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

#### R 29.3.8 Copper and lead

Determine these elements by anodic stripping voltammetry according to GM 33, using a solution of 1 g of the sample in 25 ml of standard volumetric sulfuric acid solution,  $c(1/2 \text{ H}_2\text{SO}_4) = 1 \text{ mol/l}$ .

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

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**R 29 Sodium acetate trihydrate**


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**R 29.3.9 Iron**

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

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

**R 29.3.10 Potassium**

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

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

**R 29.3.11 Permanganate-reducing substances**

Dissolve 10 g of the sample in 100 ml of water, add 1 ml of standard volumetric potassium permanganate solution,  $c(1/5 \text{ KMnO}_4) = 0,1 \text{ mol/l}$ , boil for 5 min, and cool.

The pink colour of the solution shall not disappear completely.

Add 2 g of the potassium iodide (R 25) and 20 ml of sulfuric acid solution (20 %), and titrate with standard volumetric sodium thiosulfate solution,  $c(\text{Na}_2\text{S}_2\text{O}_3) = 0,1 \text{ mol/l}$ , until colourless.

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

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

## R 30 Sodium carbonate, anhydrous

### Na<sub>2</sub>CO<sub>3</sub>

Relative molecular mass : 105,99

#### R 30.1 Specification

Assay (Na <sub>2</sub> CO <sub>3</sub> , after drying at 300 °C) ...	99,8 % min.
Chloride (Cl) .....	0,002 % max.
Phosphate and silicate (expressed as SiO <sub>2</sub> )	0,005 % max.
Total nitrogen (N) .....	0,001 % max.
Total sulfur (expressed as SO <sub>4</sub> ) .....	0,005 % max.
Aluminium (Al) .....	0,001 % max.
Calcium (Ca) .....	0,005 % max.
Heavy metals (expressed as Pb) .....	0,000 5 % max.
Iron (Fe) .....	0,000 5 % max.
Magnesium (Mg) .....	0,002 % max.
Potassium (K) .....	0,01 % max.
Loss after drying (at 300 °C) .....	1,0 % max.

#### R 30.2 Preparation of test solution

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

#### R 30.3 Tests

##### R 30.3.1 Assay

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

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

1,00 ml of hydrochloric acid solution,  $c(\text{HCl}) = 1,000 \text{ mol/l}$ , corresponds to 0,052 995 g of Na<sub>2</sub>CO<sub>3</sub>.

##### R 30.3.2 Chloride

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

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

##### R 30.3.3 Phosphate and silicate

Dissolve 1 g of the sample in a mixture of 80 ml of water and 3 ml of hydrochloric acid solution (25 %). Heat to boiling and, after cooling, add 5 ml of hexaammonium heptamolybdate solution (10 %). Adjust to pH 1,8 with the hydrochloric acid (R 13) and heat the solution to boiling. After cooling, add 10 ml

of the hydrochloric acid (R 13) and extract with 50 ml of 4-methyl-2-pentanone. Wash the organic phase with hydrochloric acid solution (0,5 %) and reduce with 0,2 ml of tin(II) chloride solution [2 % SnCl<sub>2</sub>·2H<sub>2</sub>O in the hydrochloric acid (R 13)]. Dry the organic phase with anhydrous sodium sulfate.

The blue coloration of the organic phase shall not be more intense than that of a similarly prepared standard matching solution, using 5 ml of the silicate SS II (5 ml  $\cong$  0,005 % SiO<sub>2</sub>).

##### R 30.3.4 Total nitrogen

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

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

##### R 30.3.5 Total sulfur

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

Prepare a standard matching solution, using 5 ml of the sulfate SS II (5 ml  $\cong$  0,005 % SO<sub>4</sub>).

##### R 30.3.6 Aluminium

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

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

##### R 30.3.7 Calcium and magnesium

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	Test solution (R 30.2)	Air-acetylene	422,7
Mg			285,2

##### R 30.3.8 Heavy metals

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

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

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**R 30 Sodium carbonate, anhydrous**

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**R 30.3.9 Iron**

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

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

**R 30.3.10 Potassium**

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

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

**R 30.3.11 Loss after drying**

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

The loss in mass shall not exceed 20 mg

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## R 31 Sodium carbonate decahydrate

### Na<sub>2</sub>CO<sub>3</sub>·10H<sub>2</sub>O

Relative molecular mass : 286,14

#### R 31.1 Specification

Assay (Na <sub>2</sub> CO <sub>3</sub> ·10H <sub>2</sub> O) .....	99 to 102 %
Chloride (Cl) .....	0,001 % max.
Phosphate and silicate (expressed as SiO <sub>2</sub> ) .....	0,003 % max.
Total nitrogen (N) .....	0,000 5 % max.
Total sulfur (expressed as SO <sub>4</sub> ) .....	0,003 % max.
Aluminium (Al) .....	0,000 5 % max.
Calcium (Ca) .....	0,003 % max.
Heavy metals (expressed as Pb) .....	0,000 5 % max.
Iron (Fe) .....	0,000 3 % max.
Magnesium (Mg) .....	0,000 3 % max.
Potassium (K) .....	0,005 % max.

#### R 31.2 Preparation of test solution

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

#### R 31.3 Tests

##### R 31.3.1 Assay

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

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

1,00 ml of hydrochloric acid solution,  $c(\text{HCl}) = 1,000 \text{ mol/l}$ , corresponds to 0,143 1 g of Na<sub>2</sub>CO<sub>3</sub>·10H<sub>2</sub>O.

##### R 31.3.2 Chloride

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

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

##### R 31.3.3 Phosphate and silicate

Dissolve 2 g of the sample in a mixture of 80 ml of water and 3 ml of hydrochloric acid solution (25 %). Heat to boiling and after cooling add 5 ml of hexaammonium heptamolybdate solution (10 %). Adjust to pH 1,8 with the hydrochloric acid (R 13)

and heat the solution to boiling. After cooling add 10 ml of the hydrochloric acid (R 13) and extract with 50 ml of 4-methyl-2-pentanone. Wash the organic phase with hydrochloric acid solution (0,5 %) and reduce with 0,2 ml of tin(II) chloride solution [2 % SnCl<sub>2</sub>·2H<sub>2</sub>O in the hydrochloric acid (R 13)]. Dry the organic phase with anhydrous sodium sulfate.

The blue coloration of the organic phase shall not be more intense than that of a similarly prepared standard matching solution, using 6 ml of the silicate SS II (6 ml  $\cong$  0,003 % SiO<sub>2</sub>).

##### R 31.3.4 Total nitrogen

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

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

##### R 31.3.5 Total sulfur

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

Prepare a standard matching solution, using 6 ml of the sulfate SS II (6 ml  $\cong$  0,003 % SO<sub>4</sub>).

##### R 31.3.6 Aluminium

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

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

##### R 31.3.7 Calcium and magnesium

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

Element	Concentration of solution	Flame	Resonance line nm
Ca	10 %	Air-acetylene	422,7
Mg			285,2

##### R 31.3.8 Heavy metals

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

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**R 31 Sodium carbonate decahydrate**

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

**R 31.3.9 Iron**

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

Prepare a standard matching solution, using 0,9 ml of the iron SS II ( $0,9 \text{ ml} \cong 0,000 3 \% \text{ Fe}$ ).

**R 31.3.10 Potassium**

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

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

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## R 32 Sodium chloride

### NaCl

Relative molecular mass : 58,44

#### R 32.1 Specification

Assay (NaCl) .....	99,5 % min.
pH (5 % solution) : 5 to 8	
Bromide (Br) .....	0,01 % max.
Iodide (I) .....	0,002 % max.
Hexacyanoferrate(II) [Fe(CN) <sub>6</sub> ] .....	0,000 1 % max.
Phosphate (PO <sub>4</sub> ) .....	0,000 5 % max.
Sulfate (SO <sub>4</sub> ) .....	0,002 % max.
Total nitrogen (N) .....	0,001 % max.
Barium (Ba) .....	0,001 % max.
Calcium (Ca) .....	0,002 % max.
Heavy metals (expressed as Pb) .....	0,000 5 % max.
Iron (Fe) .....	0,000 2 % max.
Magnesium (Mg) .....	0,002 % max.
Potassium (K) .....	0,01 % max.

#### R 32.2 Preparation of test solution

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

#### R 32.3 Tests

##### R 32.3.1 Assay

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

To this solution add 1 ml of the nitric acid (R 19) and 30,0 ml of the standard volumetric silver nitrate solution,  $c(\text{AgNO}_3) = 0,1 \text{ mol/l}$ . Titrate potentiometrically with the standard volumetric silver nitrate solution,  $c(\text{AgNO}_3) = 0,1 \text{ mol/l}$ , using Ag-Ag/Cl electrodes.

1,00 ml of silver nitrate solution,  $c(\text{AgNO}_3) = 0,100 \text{ mol/l}$ , corresponds to 0,005 844 g of NaCl.

##### R 32.3.2 pH

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

##### R 32.3.3 Iodide

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

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

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

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

Reserve both solutions for the test in R 32.3.4.

##### R 32.3.4 Bromide

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

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

##### R 32.3.5 Hexacyanoferrate(II)

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

Any blue coloration shall not be more intense than that of a standard matching solution containing 1 g of the sample and 2,5 ml of the hexacyanoferrate(II) SS III [2,5 ml  $\cong$  0,000 1 % Fe(CN)<sub>6</sub>].

##### R 32.3.6 Phosphate

Take 50 ml of the test solution (R 32.2), add 30 ml of water and apply GM 4.

Prepare a standard matching solution, using 2,5 ml of the phosphate SS II (2,5 ml  $\cong$  0,000 5 % PO<sub>4</sub>).

##### R 32.3.7 Sulfate

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

Prepare a standard matching solution, using 4 ml of the sulfate SS II (4 ml  $\cong$  0,002 % SO<sub>4</sub>).

## R 32 Sodium chloride

### R 32.3.8 Total nitrogen

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

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

### R 32.3.9 Heavy metals

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

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

### R 32.3.10 Iron

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

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

### R 32.3.11 Barium

Mix 0,25 ml of barium chloride dihydrate solution [0,02 %  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  in 30 % (V/V) ethanol] and 1 ml of sodium sulfate decahydrate solution (40 %  $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ ) (seeding solution). To this mixture add, after exactly 1 min, 20 ml of the test solu-

tion (R 32.2), which has been previously acidified with 0,5 ml of hydrochloric acid solution (25 %) and set aside for 15 min.

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

### R 32.3.12 Calcium and magnesium

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

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

### R 32.3.13 Potassium

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

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

## R 33 Disodium hydrogenphosphate dodecahydrate Na<sub>2</sub>HPO<sub>4</sub>·12H<sub>2</sub>O

Relative molecular mass : 358,14

### R 33.1 Specification

Assay (Na <sub>2</sub> HPO <sub>4</sub> ·12H <sub>2</sub> O) .....	99 to 102 %
pH (5 % solution) : 9 to 9,4	
Chloride (Cl) .....	0,001 % max.
Sulfate (SO <sub>4</sub> ) .....	0,005 % max.
Total nitrogen (N) .....	0,002 % max.
Heavy metals (expressed as Pb) .....	0,000 5 % max.
Iron (Fe) .....	0,000 5 % max.
Potassium (K) .....	0,01 % max.

### R 33.2 Preparation of test solution

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

### R 33.3 Tests

#### R 33.3.1 Assay

Weigh, to the nearest 0,001 g, about 10 g of the sample and dissolve in 200 ml of carbon dioxide-free water. Titrate potentiometrically with standard volumetric sulfuric acid solution,  $c(1/2 \text{ H}_2\text{SO}_4) = 1 \text{ mol/l}$ , to pH 4,4, using a glass indicator electrode.

1,00 ml of sulfuric acid solution,  $c(1/2 \text{ H}_2\text{SO}_4) = 1,000 \text{ mol/l}$ , corresponds to 0,358 14 g of Na<sub>2</sub>HPO<sub>4</sub>·12H<sub>2</sub>O.

#### R 33.3.2 pH

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

#### R 33.3.3 Chloride

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

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

#### R 33.3.4 Sulfate

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

Prepare a standard matching solution, using 10 ml of the sulfate SS II (10 ml  $\cong$  0,005 % SO<sub>4</sub>).

#### R 33.3.5 Total nitrogen

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

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

#### R 33.3.6 Heavy metals

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

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

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

#### R 33.3.7 Iron

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

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

#### R 33.3.8 Potassium

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

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