BRITISH STANDARD

BS EN 889:2004

Chemicals used for treatment of water intended for human consumption — Iron (II) sulfate

The European Standard EN 889:2004 has the status of a British Standard

ICS 71.100.80

British Standards

National foreword

This British Standard is the official English language version of EN 889:2004. It supersedes BS EN 889:1999 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/59, Chemicals for drinking water treatment, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English version

Chemicals used for treatment of water intended for human consumption - Iron (II) sulfate

Produits chimiques utilisés pour le traitement de l'eau destinée à la consommation humaine - Sulfate de fer (II)

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Eisen (II) sulfat

This European Standard was approved by CEN on 30 September 2004.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Foreword

This document (EN 889:2004) has been prepared by Technical Committee CEN/TC 164 “Water supply”, the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2005, and conflicting national standards shall be withdrawn at the latest by May 2005.

This document supersedes EN 889:1998.

Significant technical differences between this edition and EN 889:1998 are as follows:

a) replacement of the reference of to EU Directive 80/778 of 15 July 1980 with the latest Directive in force (see[1]);

b) expansion of annex A by addition of A.2 "quality of commercial product";

c) deletion of annex D.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

In respect of potential adverse effects on the quality of water intended for human consumption, caused by the product covered by this Standard:

a) this Standard provides no information as to whether the product may be used without restriction in any of the Member States of the EU or EFTA;

b) It should be noted that, while awaiting the adoption of verifiable European criteria, existing national regulations concerning the use and/or the characteristics of this product remain in force.

NOTE Conformity with the standard does not confer or imply acceptance or approval of the product in any of the Member States of the EU or EFTA. The use of the product covered by this document is subject to regulation or control by National Authorities.

1 Scope

This document is applicable to iron (II) sulfate heptahydrate used for treatment of water intended for human consumption. It describes the characteristics of iron (II) sulfate heptahydrate and specifies the requirements and the corresponding analytical methods for iron (II) sulfate heptahydrate (analytical methods are given in Annex B) and gives information on its use in water treatment.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods* (ISO 3696:1987).

ISO 3165, *Sampling of chemical products for industrial use - Safety in sampling*.

ISO 6206, *Chemical products for industrial use - Sampling - Vocabulary*.

ISO 8213, *Chemical products for industrial use - Sampling techniques - Solid chemical products in the form of particles varying from powders to coarse lumps*.

3 Description

3.1 Identification

3.1.1 Chemical name

Iron (II) sulfate heptahydrate.

3.1.2 Synonym or common names

Ferrous sulfate, iron vitriol, copperas, green salt.

3.1.3 Relative molecular mass

278,02.

3.1.4 Empirical formula

$FeSO_4 . 7H_2O$.

3.1.5 Chemical formula

$FeSO_4 . 7H_2O$.

3.1.6 CAS Registry Number[1]

7782-63-0 ($FeSO_4 . 7H_2O$).

7720-78-7 ($FeSO_4$).

3.1.7 EINECS reference[2]

231-753-5 ($FeSO_4$).

3.2 Commercial forms

The iron (II) sulfate heptahydrate is available as wet crystals (a) or free-flowing crystals (b).

3.3 Physical properties

3.3.1 Appearance

The iron (II) sulfate heptahydrate is a light green crystal.

3.3.2 Density

The density of iron (II) sulfate heptahydrate is equal to 1,9 g/cm^3 at 20 °C.

The bulk density of commercial form a) is equal approximately to 1 kg/dm^3 and of commercial form b) approximately to 0,8 kg/dm^3.

3.3.3 Solubility (in water)

The solubility of iron (II) sulfate heptahydrate is 487 g/l at 20 °C.

3.3.4 Vapour pressure

Not applicable.

3.3.5 Boiling point at 100 kPa[3]

Not applicable.

3.3.6 Melting point

For the iron (II) sulfate heptahydrate the melting starts at approximately 64 °C, leading to decomposition.

3.3.7 Specific heat

Not known.

3.3.8 Viscosity (dynamic)

Not applicable.

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[1] Chemical Abstracts Service Registry Number.

[2] European Inventory of Existing Commercial Chemical Substances.

[3] 100 kPa = 1 bar.

3.3.9 Critical temperature

Not applicable.

3.3.10 Critical pressure

Not applicable.

3.3.11 Physical hardness

Not applicable.

3.4 Chemical properties

Iron (II) sulfate heptahydrate and especially solutions of iron (II) sulfate heptahydrate have acid and reducing properties. They react by oxidation or hydrolysis (depending on the pH).

4 Purity criteria

4.1 General

This document specifies the minimum purity requirements for iron (II) sulfate used for the treatment of water intended for human consumption. Limits are given for impurities commonly present in the product. Depending on the raw material and the manufacturing process other impurities may be present and, if so, this shall be notified to the user and when necessary to relevant authorities.

NOTE Users of this product should check the national regulations in order to clarify whether it is of appropriate purity for treatment of water intended for human consumption, taking into account raw water quality, required dosage, contents of other impurities and additives used in the product not stated in this product standard.

Limits have been given for impurities and chemical parameters where these are likely to be present in significant quantities from the current production process and raw materials. If the production process or raw materials leads to significant quantities of impurities, by-products, or additives being present, this shall be notified to the user.

4.2 Composition of commercial product

The product shall contain not less than a mass fraction of 82,1 % of $FeSO_4 . 7H_2O$ (i.e. not less than a mass fraction of 16 % of Fe) and shall be within ± 3 % of the manufacturer's declared values.

4.3 Impurities and main by-products

The product shall conform to the requirements specified in Table 1.

The concentration limits refer to Fe (II).

Table 1 — Impurities

Impurity		Limit Mass fraction of Fe (II) content in %		
		Grade 1	**Grade 2**	**Grade 3**
Manganese	max.	0,5	1	2
Insoluble matters :				
- wet crystal form (a)	max.	0,6	0,6	0,6
- free flowing form (b)	max.	3	3	3

NOTE An excess of insoluble matters indicates the presence of foreign matter. Iron as a component of the product will usually be removed in the treatment process.

4.4 Chemical parameters

The product shall conform to the requirements specified in Table 2.

The concentration limits are specified in milligrams per kilogram of Fe (II).

Table 2 — Chemical parameters

Parameter		Limit in mg/kg of Fe (II)		
		type 1	**type 2**	**type 3**
Arsenic (As)	max.	1	20	50
Cadmium (Cd)	max.	1	25	50
Chromium (Cr)	max.	100	350	500
Mercury (Hg)	max.	0,1	5	10
Nickel (Ni)	max.	300	350	500
Lead (Pb)	max.	10	100	400
Antimony (Sb)	max.	10	20	60
Selenium (Se)	max.	1	20	60

NOTE Cyanide (CN^-),pesticides and polycyclic aromatic hydrocarbons are not relevant since the raw materials used in the manufacturing process are free of them. For maximum impact of iron (II) sulfate on trace metal content in drinking water see A.2.

5 Test methods

5.1 Sampling

Observe the general recommendations of ISO 3165 and take into account ISO 6206. Prepare the laboratory sample required by the relevant procedure described in ISO 8213.

5.2 Analyses

5.2.1 Main product

Iron (II) sulfate is determined as Fe (II) content in the test sample by titrimetry with potassium dichromate solution (see B.1).

5.2.2 Impurities

5.2.2.1 Manganese

The manganese content shall be determined by flame atomic absorption spectrometry (FAAS) (see B.2).

5.2.2.2 Insoluble matters

The percentage mass fraction of the insoluble matters shall be determined in accordance with the method described in B.3.

5.2.3 Chemical parameters

5.2.3.1 General

The contents of chemical parameters shall be determined by atomic absorption spectrometry (AAS).

5.2.3.2 Preparation of sample solution

5.2.3.2.1 General

Oxidation and wet digestion is used to bring the samples into a stable solution.

5.2.3.2.2 Principle

Oxidation with hydrogen peroxide (H_2O_2) followed by digestion with hydrochloric acid (HCl).

5.2.3.2.3 Reagents

All reagents shall be of a recognized analytical grade and the water used shall conform to the grade 3 specified in EN ISO 3696.

5.2.3.2.3.1 Hydrochloric acid (HCl), solution, mass fraction 30 %.

5.2.3.2.3.2 Hydrogen peroxide (H_2O_2), solution, mass fraction 30 %.

5.2.3.2.4 Apparatus

Ordinary laboratory apparatus and glassware together with the following

5.2.3.2.4.1 Analytical balance.

5.2.3.2.4.2 Graduated cylinder, capacity 50 ml.

5.2.3.2.4.3 Round flask with reflux condenser.

5.2.3.2.4.4 Hot plate.

5.2.3.2.4.5 Volumetric flask, capacity 200 ml.

5.2.3.2.5 Procedure

Dissolve with 20 ml of water 20,0 g of the iron salt. Add to iron (II)-samples 30 ml hydrogen peroxide solution (5.2.3.2.3.2). After adding 50 ml hydrochloric acid (5.2.3.2.3.1) boil the solution for 15 min by using a reflux condenser (5.2.3.2.4.3). Cool down the solution, transfer to a 200 ml volumetric flask (5.2.3.2.4.5) and fill up to the mark with water. This is the sample solution.

5.2.3.3 Arsenic

The arsenic content shall be determined by hydride generation atomic absorption spectrometry (see B.4).

5.2.3.4 Cadmium

The cadmium content shall be determined by graphite furnace atomic absorption spectrometry (see B.6).

5.2.3.5 Chromium

The chromium content shall be determined by graphite furnace atomic absorption spectrometry (see B.6).

5.2.3.6 Mercury

The mercury content shall be determined by cold vapour atomic absorption spectrometry (see B.5).

5.2.3.7 Nickel

The nickel content shall be determined by graphite furnace atomic absorption spectrometry (see B.6).

5.2.3.8 Lead

The lead content shall be determined by graphite furnace atomic absorption spectrometry (see B.6).

5.2.3.9 Antimony

The antimony content shall be determined by hydride generation atomic absorption spectrometry (see B.4).

5.2.3.10 Selenium

The selenium content shall be determined by hydride generation atomic absorption spectrometry (see B.4).

6 Labelling - Transportation - Storage

6.1 Means of delivery

In order that the purity of the product is not affected, the means of delivery shall not have been used previously for any different product or it shall have been specially cleaned and prepared before use.

6.2 Risk and safety labelling according to the EU Directives[4)]

Iron (II) sulfate is not subject to labelling regulations.

NOTE Annex I of the Directive 67/548/EEC on Classification, packaging and labelling of dangerous substances and its amendments and adaptations in the European Union contains a list of substances classified by the EU. Substances not in this Annex I should be classified on the basis of their intrinsic properties according to the criteria in the Directive by the person responsible for the marketing of the substance.

6.3 Transportation regulations and labelling

Iron (II) sulfate is not listed under a UN number [5)].

Iron (II) sulfate is not classified as a dangerous product for road, rail, sea and air transportation.

6.4 Marking

The marking shall include the following information:

- the name "iron (II) sulfate", trade name, commercial form, grade and type;
- the net mass;
- the name and the address of the supplier and/or manufacturer;
- the statement "this product conforms to EN 889".

6.5 Storage

6.5.1 Long term stability

- Commercial form a) : Depending upon conditions of storage discolouration can occur due to oxidation to the ferric form. Storage time shall therefore be as short as possible. Normally it is made up into solution and used promptly;
- commercial form b) : Unlimited stability when it is stored at temperature not over 40 °C in a dry place.

6.5.2 Storage incompatibilities

Iron (II) sulfate heptahydrate and its solutions have an acid character similar to diluted solutions of sulfuric acid and are therefore corrosive to most common metals, also to concrete. Contact with these materials has to be avoided. Keep away from alkaline or oxidation agents. Avoid contact with agents capable of liberating chlorine.

[4)] See [2].

[5)] United Nations Number.

Annex A
(informative)

General information on iron (II) sulfate

A.1 Origin

A.1.1 Raw materials

The product is manufactured from:

- iron-titanium ore and sulfuric acid;
- steel and sulfuric acid (pickle liquor).

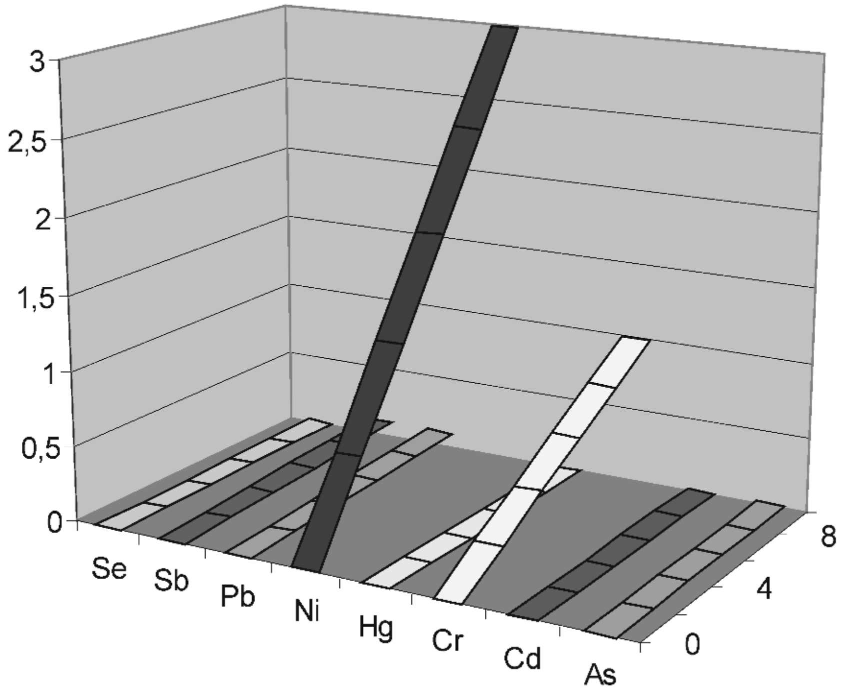
A.1.2 Manufacturing process

Iron (II) sulfate is produced by: Crystallization of oversaturated solutions of iron (II) sulfate heptahydrate in sulfuric acid by cooling and separation of the crystals by centrifuges or filters.

For the commercial form b) : Additional removal of the moisture by drying.

A.2 Quality of commercial product

The three types of iron (II) sulfate specified in Table 2 reflect the quality of commercially available products. Figures A.1 to A.3 show the maximum concentrations of trace metals that would be added to the raw water by the addition of products corresponding to the purity levels specified in Table 2. It can be seen that the concentrations of metal added are well below the Parametric Values given in the EU Directive 98/83/EC (see [1]) at typical product doses. Furthermore, the figures overstate the concentrations of metals that would be present in the treated water since a substantial proportion of the trace metals will be incorporated in the sludge. Users of this product should select an appropriate grade and type to enable them to achieve treated water quality targets taking into account raw water characteristics, required dosage, process plant conditions and other relevant factors.



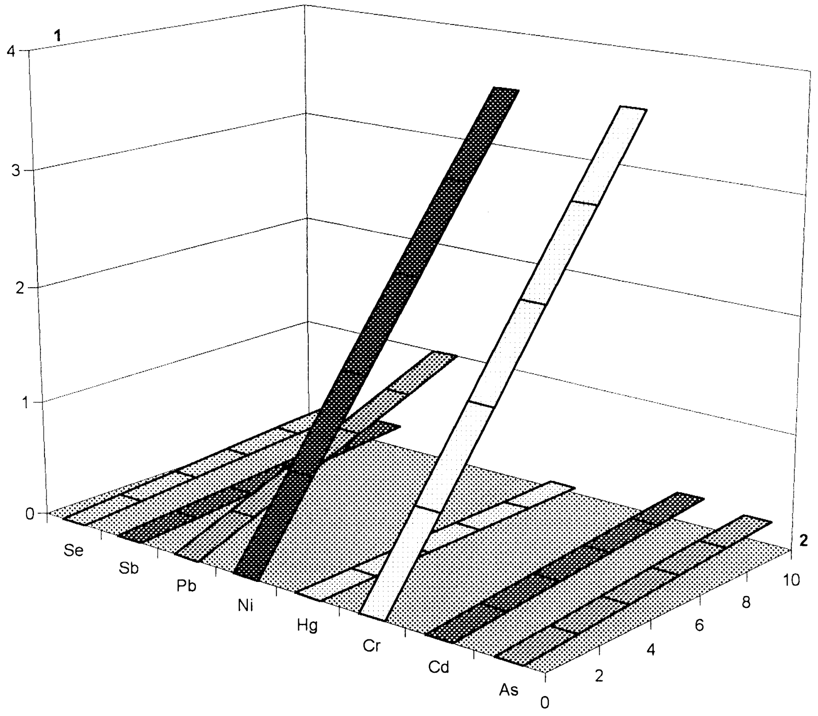
Element	Drinking water limit ug/l
As	10
Cd	5
Cr	50
Hg	1
Ni	20
Pb	10
Sb	5
Se	10

Key

1 Maximum addition to water µg/l metal

2 Product dosage mg/l Fe - Typical dose

Figure A.1 — Maximum impact of iron (II) sulfate, type 1, on trace metal content of water



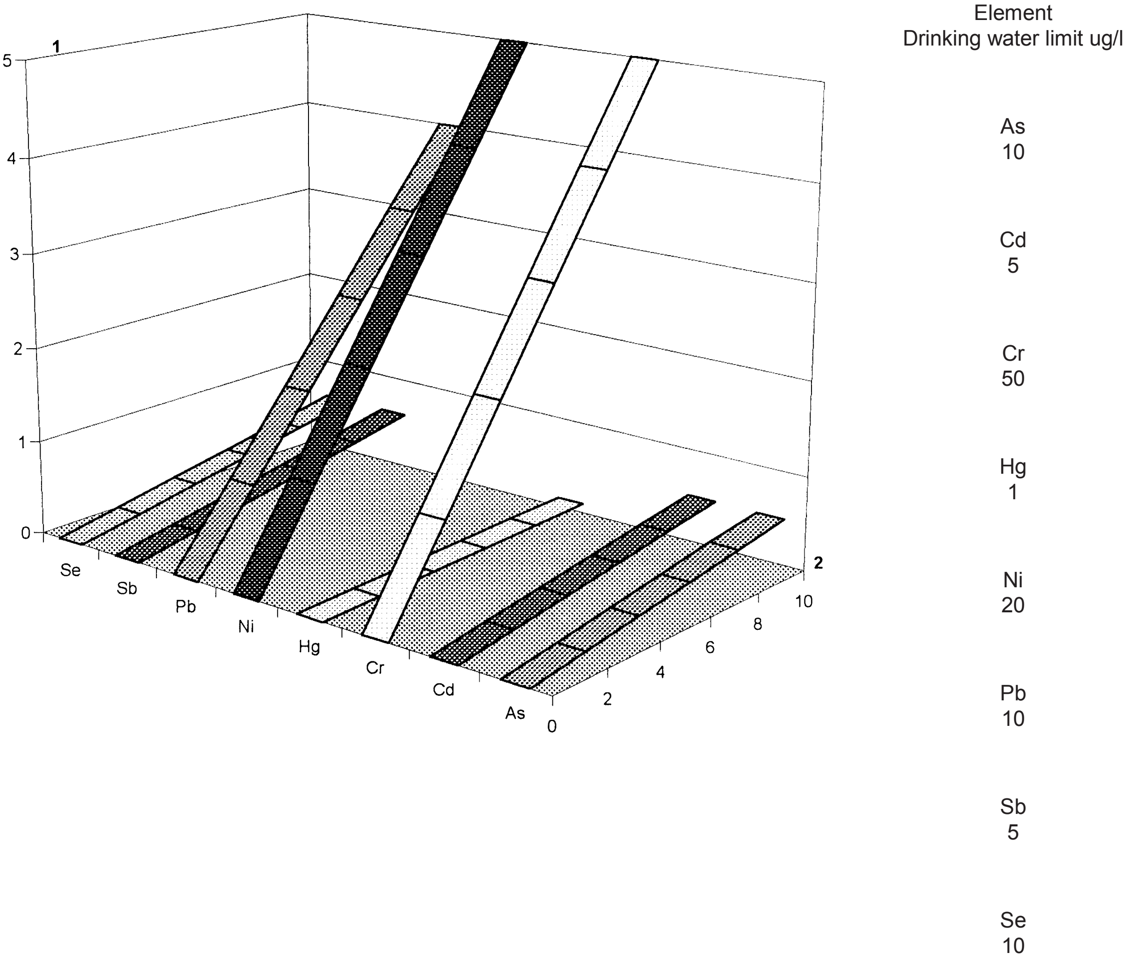
Element	Drinking water limit ug/l
As	10
Cd	5
Cr	50
Hg	1
Ni	20
Pb	10
Sb	5
Se	10

Key

1 Maximum addition to water µg/l metal

2 Product dosage mg/l Fe - Typical dose

Figure A.2 — Maximum impact of iron (II) sulfate, type 2, on trace metal content of water



Key

1 Maximum addition to water µg/l metal

2 Product dosage mg/l Fe - Typical dose

Figure A.3 — Maximum impact of iron (II) sulfate, type 3, on trace metal content of water

A.3 Use

A.3.1 Function

The product is used as primary coagulant.

A.3.2 Form in which it is used

The product is used saturated or highly concentrated, not lower than a mass fraction of 20 %. Over-diluted solutions can hydrolyse and form a precipitate.

A.3.3 Treatment dose

The treatment dose is variable depending on raw water quality and corresponds to approximately 20 g/m^3 to 50 g/m^3 (corresponding to a treatment dose between 4 g/m^3 and 10 g/m^3 expressed as Fe).

A.3.4 Means of application

In order to use iron (II) sulfate heptahydrate as a coagulant, a previous or simultaneous oxidation to iron (III) state should be necessary. The highly concentrated solution is normally dosed by acid-resistant metering pumps. To promote a rapid dispersion a high turbulence at the point of addition is desirable.

A.3.5 Secondary effects

Increase of the sulfate content and chloride concentration if chlorine is used for oxidation. Reduction of alkalinity and pH value.

A.3.6 Removal of excess product

The coagulation process includes the hydrolysis of the ferric ions to ferric hydroxide. This precipitate is removed by sedimentation, flotation and/or filtration.

A.4 General rules relating to safety

A.4.1 Rules for safe handling and use

The supplier will provide current safety instructions.

A.4.2 Emergency procedures

A.4.2.1 First aid

In case of contact with skin, the affected area should be washed with water.

In case of contact with eyes, they should be immediately rinsed with water for at least 10 min, medical advice should be sought.

If swallowed, provided patient is conscious, the mouth should be washed out with water and 5 % (*m*/*m*) sodium bicarbonate solution followed by a demulcent such as milk should be given. Medical advice should be sought.

A.4.2.2 Spillage

As much solid product as possible should be removed. Then it should be rinsed with water.

A.4.2.3 Fire

Product is not combustible.

Annex B
(normative)

Analytical methods

B.1 Determination of iron (II) : Fe (II)

B.1.1 General

This method applies to product with iron contents greater than a mass fraction of 0,2 %.

B.1.2 Principle

Iron is directly titrated with potassium dichromate solution.

B.1.3 Reagents

All reagents shall be of a recognized analytical grade and the water used shall conform to the grade 3 in accordance with EN ISO 3696.

B.1.3.1 Sulfuric acid, H_2SO_4 concentrated, density $\rho = 1{,}84$ g/ml.

B.1.3.2 Phosphoric acid, H_3PO_4 concentrated, density $\rho = 1{,}71$ g/ml.

B.1.3.3 Barium diphenylamine sulfonate solution, $c(Ba(C_6H_5\text{-}NH\text{-}C_6H_4SO_3)_2) = 8$ mmol/l.

B.1.3.4 Potassium dichromate solution, $c(K_2Cr_2O_7) = 0{,}0166$ mol/l.

B.1.4 Apparatus

Ordinary laboratory apparatus and glassware.

B.1.5 Procedure

B.1.5.1 Test solution

In a 500 ml conical flask, pour 200 ml of water, add 10 ml of sulfuric acid (B.1.3.1) and 10 ml of phosphoric acid (B.1.3.2). Cool with water. Weigh to the nearest 0,1 mg, between 3 g and 10 g of the laboratory sample and transfer to the conical flask containing the acid solution.

B.1.5.2 Determination

Titrate the test solution with potassium dichromate (B.1.3.4) with a volumetric burette and add five drops of barium diphenylamine sulfonate (B.1.3.3) near the end-point.

End-point is achieved when a persistent purple colour appears, record the volume (V) of potassium dichromate required for the complete titration.

B.1.5.3 Expression of results

The iron (II) content, $C_{(II)}$ expressed as mass fraction, is given by the following equation:

$$C_{(II)} = \frac{V \times 0{,}558}{m} \tag{1}$$

where

V is the volume, in millilitres, of potassium dichromate required for the titration;

m is the mass, in grams, of the sample used for the test solution.

B.2 Determination of manganese

B.2.1 General

This method is applicable to iron salt with manganese content greater than a mass fraction of 0,05 %.

B.2.2 Principle

Iron salt is diluted in water. Manganese is determined in this diluted solution by direct injection of the solution into the flame of an atomic absorption spectrometer. Manganese is converted to the atomic state by thermal dissociation. The absorption of the atoms is measured in the light path of the FAAS at a wavelength specific to the element. The concentrations are calculated by means of a calibration curve.

B.2.3 Reagents

All reagents shall be of a recognized analytical grade, water shall conform to the grade 3 in accordance with EN ISO 3696 and ultra pure grade acid shall be used.

B.2.3.1 Hydrochloric acid (HCl), solution, mass fraction 30 %.

B.2.3.2 Manganese, standard stock solution, c_0 (Mn) = 1 g/l.

B.2.3.3 Manganese, intermediate standard solution, c_1 (Mn) = 100 mg/l : Pipette 10 ml of standard stock solution (B.2.3.2) into a 100 ml volumetric flask (B.2.4.1), add 3 ml of hydrochloric acid (B.2.3.1) and dilute to the mark with water .

B.2.3.4 Manganese, standard solution, c_2 (Mn) = 0,5 mg/l : Pipette 0,5 ml of intermediate standard solution (B.2.3.3) into a 100 ml volumetric flask (B.2.4.1) and dilute to the mark with water .

B.2.3.5 Manganese, standard solution, c_3 (Mn) = 1 mg/l : Pipette 1 ml of intermediate standard solution (B.2.3.3) into a 100 ml volumetric flask (B.2.4.1) and dilute to the mark with water .

B.2.3.6 Manganese, standard solution, c_4 (Mn) = 2 mg/l : Pipette 2 ml of intermediate standard solution (B.2.3.3) into a 100 ml volumetric flask (B.2.4.1) and dilute to the mark with water .

B.2.3.7 Manganese, standard solution, c_5 (Mn) = 3 mg/l : Pipette 3 ml of intermediate standard solution (B.2.3.3) into a 100 ml volumetric flask (B.2.4.1) and dilute to the mark with water .

B.2.3.8 Blank solution: Pipette 3 ml of hydrochloric acid (B.2.3.1) into a 200 ml volumetric flask (B.2.4.1) and dilute to the mark with water.

B.2.4 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

B.2.4.1 Volumetric flasks, capacity 100 ml and 200 ml.

B.2.4.2 Analytical balance, having an accuracy of ± 0,1 mg.

B.2.4.3 Flame atomic absorption spectrometer.

B.2.4.4 Electrode-less discharge or hollow cathode lamp for the determination of manganese.

B.2.5 Procedure

B.2.5.1 Sample solution

Weigh, to the nearest 0,1 mg, 20 g of the laboratory sample, transfer into a 200 ml volumetric flask (B.2.4.1) and dilute to the mark with water.

B.2.5.2 Spectrometer settings

The operating parameters of the flame atomic absorption spectrometer are adjusted in accordance with the operating manual issued by the manufacturer.

The spectrometer is operated at the wavelength specific for manganese: 279,5 nm. The flame composition is the following: Air: 13,5 l/min - Acetylene: 2,0 l/min.

B.2.5.3 Determination

B.2.5.3.1 Test solution

Pipette 5 ml of the sample solution (B.2.5.1) into a 100 ml volumetric flask (B.2.4.1), add 3 ml of hydrochloric acid (B.2.3.1) and dilute to the mark with water.

B.2.5.3.2 Measurement

Inject the test solution (B.2.5.3.1) directly into the flame by aspiration through the nebuliser, setting the spectrometer as described in B.2.5.2. Repeat the injection three times, record the absorbance and calculate the mean.

B.2.5.4 Calibration function

To provide the calibration function, measure the absorbance of the four standard solutions (B.2.3.7, B.2.3.6, B.2.3.5 and B.2.3.4) and the absorbance of the blank solution (B.2.3.8) respectively in the same way as the sample solutions (see B.2.5.3.2). Calculate calibration function with the absorbance means, expressed in absorbance unit, of each solutions on vertical axis and the concentrations, expressed in milligrams per litre, on the horizontal axis.

B.2.5.5 Expression of results

The manganese concentration (C_{Mn}) in the test solution is calculated with the calibration function (B.2.5.4).

The manganese concentration of the iron salts (C_s) expressed in milligrams per kilogram of iron product is calculated as following:

$$C_s = \frac{C_{Mn} \times 100 \times 200}{5 \times m} = C_{Mn} \times 4000 / m \qquad (2)$$

where

C_{Mn} is the manganese concentration in the test solution in milligrams per litre.

m is the mass , in grams, of the sample used for the test solution.

B.3 Determination of insoluble matters

B.3.1 General

This method is applicable to the determination of insoluble matters in iron based coagulants. The measuring range of insoluble matters is between a mass fraction of 0,002 % and 10 %.

B.3.2 Principle

The sample is diluted with hydrochloric acid, in order to maintain the pH value below 2, and filtered through a dried 0,22 µm pore size membrane filter. The filter is washed with diluted hydrochloric acid and dried at 105 °C. The mass of material retained by the filter is determined.

B.3.3 Reagents

B.3.3.1 Deionised water.

B.3.3.2 Hydrochloric acid, solution *c*(HCl) = 0,2 mol/l.

B.3.3.3 Hydrochloric acid solution. Dissolve 5,0 ml hydrochloric acid (B.3.3.2) in 100 ml water (B.3.3.1). The pH of the solution shall be less than 2.

B.3.4 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

B.3.4.1 Membrane filter pore size 0,22 µm.

B.3.4.2 Desiccator with dry silica gel.

B.3.4.3 Oven.

B.3.5 Procedure

Dry a 0,22 µm pore size membrane filter (B.3.4.1) in an oven (B.3.4.3) for 30 min at 105 °C. Transfer the membrane filter to a desiccator with dry silica gel (B.3.4.2) and let it cool to room temperature. Determine the mass of the dried filter on a balance weighing to an accuracy of 0,001 g.

Dilute about 60 g of the sample (m_0) with 100 ml diluted hydrochloric acid (B.3.3.3). Filter the diluted sample through the weighed dried filter. Wash the filter with three 50 ml portions of diluted hydrochloric acid (B.3.3.3).

Dry the filter for 2 h at 105 °C. Cool the filter in a desiccator. Weigh the filter.

B.3.6 Calculation

The insoluble matters (I_m) as mass fraction are given by the following equation:

$$I_m = \frac{(m_2 - m_1) \times 100}{m_0} \tag{3}$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the dry filter;

m_2 is the mass, in grams, of the dry filter cake with filter.

B.3.7 Precision

Under the repeatability conditions the coefficient of variation is 18 % with 10 measurements.

B.4 Determination of arsenic, antimony and selenium by hydride generation atomic absorption spectrometry (AAS)

B.4.1 General

This method is suitable for the determination of low concentrations of arsenic, antimony and selenium in iron salts.

The suitable concentration ranges of this method are:

As : 0,1 mg/kg to 0,5 mg/kg;

Sb : 0,1 mg/kg to 0,5 mg/kg;

Se : 0,1 mg/kg to 0,5 mg/kg.

For higher concentrations the sample solution shall be diluted.

B.4.2 Principle

Arsenic (As), antimony (Sb) and selenium (Se) are reduced to the elemental hydrides AsH_3, SbH_3 and SeH_4 by sodium tetrahydroborate. The gaseous hydrides are conveyed with argon gas to a heated quartz cuvette and thermally decomposed. The absorption of the atoms is measured in the light path of an atomic absorption spectrometer at a wavelength specific to the element. The concentrations are calculated by means of calibration curves.

B.4.3 Reagents

All reagents shall be of a recognized analytical grade and the water used shall conform to the grade 3 in accordance with EN ISO 3696.

B.4.3.1 Hydrochloric acid (HCl), solution, mass fraction 30 %.

B.4.3.2 L-ascorbic acid solution, 100 g/l.

B.4.3.3 Oxalic acid solution, 100 g/l.

B.4.3.4 Potassium iodide solution, 100 g/l.

B.4.3.5 Sulfuric acid, solution $c(H_2SO_4)$ = 1 mol/l.

B.4.3.6 Sodium tetrahydroborate solution, mass fraction of 5 % $NaBH_4$ in NaOH solution of mass fraction of 1%.

B.4.3.7 Element stock solutions, c_0 (As, Sb, Se) = 1 g/l.

B.4.3.8 Standard element solutions, c_1 (AS, Sb, Se) = 10 mg/l, to be prepared by dilution of B.4.3.7.

B.4.3.9 Standard element solutions, c_2 (As, Sb, Se) = 0,1 mg/l, to be prepared by dilution of B.4.3.8.

B.4.3.10 Element reference solutions, prepared by pipetting 1 ml, 2 ml, 4 ml, 8 ml and 10 ml of each of the standard element solutions (B.4.3.9) into a series of 100 ml volumetric flasks (B.4.4.3), adding 30 ml oxalic acid (B.4.3.3), 10 ml potassium iodide (B.4.3.4), 5 ml L-ascorbic acid (B.4.3.2) and 5 ml hydrochloric acid (B.4.3.1) for As and Sb. Then fill the volumetric flasks up to the mark with water. The concentrations of the reference solutions will be 1 µg/l, 2 µg/l, 4 µg/l, 8 µg/l and 10 µg/l.

B.4.3.11 Sample blank solution.

B.4.4 Apparatus

Ordinary laboratory apparatus and glassware together with the following:

B.4.4.1 Pipettes, capacity 5 ml, 10 ml and 20 ml.

B.4.4.2 Graduated cylinder, capacity 50 ml.

B.4.4.3 Volumetric flask, capacity 100 ml.

B.4.4.4 Atomic absorption spectrometer.

B.4.4.5 Electrode-less discharge lamps for the determination of As, Sb and Se.

B.4.4.6 Hydride system consisting of a heatable quartz cuvette, a reaction unit and a control unit.

B.4.4.7 Argon gas supply.

B.4.5 Procedure

B.4.5.1 Spectrometer settings

The atomic absorption spectrometer (B.4.4.4) and the hydride generator unit (B.4.4.6) are adjusted in accordance with the operating manual issued by the manufacturer. The spectrometer is operating at the wavelength for each element:

As : 193,7 nm;

Sb : 217,6 nm;

Se : 196,0 nm.

The quartz cuvette is heated to 900 °C.

B.4.5.2 Measurement of arsenic and antimony

Take 20 ml of the sample solution (5.2.3.2.5) with a pipette (B.4.4.1) and transfer to a 100 ml volumetric flask (B.4.4.3). Add with a graduated cylinder (B.4.4.2) 30 ml oxalic acid (B.4.3.3), 10 ml potassium iodide (B.4.3.4), 5 ml L-ascorbic acid (B.4.3.2) and 5 ml hydrochloric acid (B.4.3.1), then make up to the mark with water. The prepared solution shall be allowed to stand for 3 h to ensure complete reaction. Then transfer 5 ml of the solution with a pipette (B.4.4.1) to the reduction vessel, add 10 ml sulfuric acid (B.4.3.5) and start the hydride control unit. The solution is reduced with $NaBH_4$ and immediately AsH_3 and SbH_3 are transferred with the argon gas (B.4.4.7) to the 900 °C heated quartz cuvette. Measure the absorption of arsenic with an arsenic specific lamp (B.4.4.5) at wavelength 193,7 nm. Measure the absorption of antimony with an antimony specific lamp (B.4.4.5) at wavelength 217,6 nm.

B.4.5.3 Measurement of selenium

Transfer 20 ml of the sample solution (5.2.3.2.5) with a pipette (B.4.4.1) to a 100 ml volumetric flask. Add 30 ml oxalic acid (B.4.3.3) and 5 ml hydrochloric acid (B.4.3.1), then make up to the mark with water. Transfer 5 ml of the solution with a pipette to the reduction vessel, add 10 ml sulfuric acid (B.4.3.5) and start the hydride control unit. The solution is reduced with $NaBH_4$ and immediately SeH_4 is transferred with the argon gas (B.4.4.7) to the 900 °C heated quartz cuvette. Measure the absorption of selenium with a selenium specific lamp (B.4.4.5) at wavelength 196,0 nm.

B.4.5.4 Calculation and expression of results

The As, Sb and Se concentrations C_e in the sample solution expressed in micrograms per litre are read out from the calibration function or regression function:

$$C_e = \frac{A_e - A_B}{S} \tag{4}$$

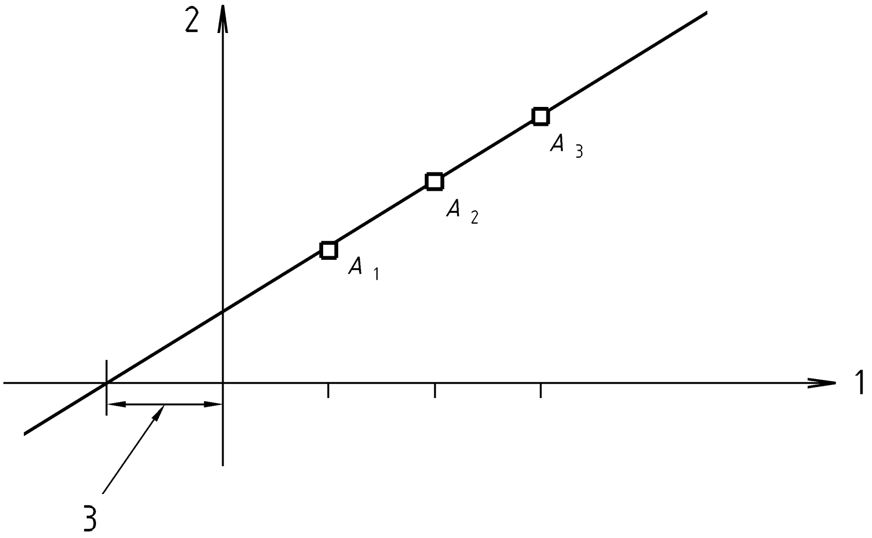
where

A_e is the absorbance of the sample solution;

A_B is the absorbance of the blank solution;

S is the slope of the calibration line in litres per microgram.

The calculation by the standard addition method can be carried out as show in Figure B.1:



Key

1 Concentration of added standard in micrograms per litre

2 Absorbance A

3 Concentration in the sample solution

A_1, A_2, A_3 Spiking

Figure B.1 — Example for the calculation of the element concentration in the sample solution by standard addition method

The concentration in the sample solution is the intersection of the extrapolated calibration curve of the spiked concentration versus absorbance and the horizontal axis on the left side.

The As, Sb and Se concentration (C_s) of the iron salt in milligrams per kilogram iron product are calculated using the following equation:

$$C_s = \frac{C_e \times 100 \times 200}{20 \times 1000 \times 20} = \frac{C_e}{20} \tag{5}$$

where

C_e is the element concentration, in micrograms per litre, in the sample solution.

NOTE The following equation gives the relation between the concentration C_f in milligrams per kilogram of Fe (II) and the concentration C_s in milligrams per kilogram of product: $C_f = C_s \times 100 / C_{(II)}$

B.5 Determination of mercury by cold vapour atomic absorption spectrometry (AAS)

B.5.1 General

This method is suitable for the determination of mercury in iron salts.

The suitable concentration range of this method is:

Hg: 0,01 mg/kg to 0,1 mg/kg.

For higher concentrations the sample solution shall be diluted.

B.5.2 Principle

Divalent mercury in the sample solution (5.2.3.2.5) is reduced to the element by tin (II) chloride or sodium tetrahydroborate solutions. Mercury is stripped from this solution in an inert gas stream and converted to the amalgam on a gold/platinum gauze for the purpose of enrichment. Mercury is released again by rapidly heating the absorber and conveyed to a quartz cuvette. The absorption of the mercury atoms is measured in a light path of an atomic absorption spectrometer at a wavelength specific to mercury (253,7 nm). The concentration is calculated by means of a calibration curve.

B.5.3 Reagents

All reagents shall be of a recognized analytical grade and the water used shall conform to the grade 3 in accordance with EN ISO 3696.

B.5.3.1 Hydrochloric acid (HCl) solution, mass fraction 30 %.

B.5.3.2 Sulfuric acid, solution, $c(H_2SO_4) = 1$ mol/l.

B.5.3.3 Tin (II) chloride solution, mass fraction of 10 % $SnCl_2$ in HCl solution of a mass fraction of 5 %.

B.5.3.4 Stabilizing solution (0,5 g $KMnO_4$ in 100 ml of water).

B.5.3.5 Mercury stock solution, c_0 (Hg) = 1 g/l.

B.5.3.6 Standard mercury solution, c_1 (Hg) = 10 mg/l to be prepared by dilution of B.5.3.5.

B.5.3.7 Standard mercury solution, c_2 (Hg) = 0,1 mg/l, to be prepared by dilution of B.5.3.6.

B.5.3.8 Mercury reference solution, to be prepared by pipetting 1 ml, 2 ml, 4 ml, 8 ml and 10 ml of the standard solution (B.5.3.7) into a series of 100 ml volumetric flasks (B.5.4.2), adding 1 ml of stabilizing solution (B.5.3.4), 5 ml of hydrochloric acid (B.5.3.1), and making up to the mark with water. The reference solutions contain 1 µg/l, 2 µg/l, 4 µg/l, 8 µg/l and 10 µg/l mercury.

B.5.3.9 Sample blank solution.

B.5.4 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

B.5.4.1 Pipettes, capacity 1 ml, 5 ml and 10 ml.

B.5.4.2 Volumetric flasks, capacity 100 ml.

B.5.4.3 Atomic absorption spectrometer.

B.5.4.4 Electrode-less discharge lamp for the determination of mercury.

B.5.4.5 Reduction system consisting of a reaction unit with tin (II) chloride, a quartz cuvette and a control unit.

B.5.4.6 Argon gas supply.

B.5.4.7 Attachment for the amalgam technique.

B.5.5 Procedure

B.5.5.1 Spectrometer settings

The atomic absorption spectrometer (B.5.4.3) and the reduction unit (B.5.4.5) are adjusted in accordance with the operating manual issued by the manufacturer. The spectrometer is operated at the wavelength 253,7 nm specific for mercury. The quartz cuvette is heated to 200 °C.

B.5.5.2 Measurement of mercury

Transfer 5 ml of the sample solution (5.2.3.2.5) with a pipette (B.5.4.1) to the reduction vessel (B.5.4.5). Add 10 ml sulfuric acid (B.5.3.2) and 1 ml stabilizing solution (B.5.3.4). The reaction with tin (II) chloride is started from the control unit and the mercury vapour is enriched at the gold/platinum gauze (B.5.4.7). After rapidly heating the absorber, mercury is transferred with the argon gas (B.5.4.6) to the 200 °C heated quartz cuvette. Measure and record the absorption of mercury with a mercury specific lamp (B.5.4.4) at wavelength 253,7 nm.

B.5.5.3 Calibration curve

To provide the calibration curve measure the blank solution and at least five reference solutions (B.5.3.8) in the same way as the sample solution, so that 5 ml of the reference solutions are also pipetted in the reduction vessel. The further procedure is the same as in B.5.5.2. The standard addition method is also possible to avoid matrix effects.

B.5.5.4 Calculation

The mercury concentration (C_{Hg}) in the sample solution is read out from the calibration curve and the calculation is the same as in B.4.5.4.

The mercury concentration (C_s) of the iron salts in milligrams per kilogram of iron product is calculated using the following formula:

$$C_s = \frac{C_{Hg} \times 200}{1000 \times 20} = \frac{C_{Hg}}{100} \qquad (6)$$

where

C_{Hg} is the mercury concentration in micrograms per litre in the sample solution.

NOTE: The following equation gives the relation between the concentration C_f in milligrams per kilogram of Fe (II) and the concentration C_s in milligrams per kilogram of product: $C_f = C_s \times 100/ C_{(II)}$

B.6 Determination of cadmium, chromium, nickel and lead by graphite furnace atomic absorption spectrometry (AAS)

B.6.1 General

This method is suitable for the determination of low concentrations of cadmium, chromium, nickel and lead in iron salts.

The suitable concentration ranges of this method are:

Cd : 0,1 mg/kg to 1,0 mg/kg;

Cr : 1,0 mg/kg to 10 mg/kg;

Ni : 1,0 mg/kg to 10 mg/kg;

Pb : 1,0 mg/kg to 10 mg/kg.

NOTE For higher concentration the sample solution should be diluted or inductively coupled plasma optical emission spectrometry (ICP/OES) and flame atomic absorption spectrometry (FAAS) can be used.

B.6.2 Principle

Cadmium, chromium, nickel and lead are converted to the atomic state by thermal dissociation in an electrically-heated graphite tube of an atomic absorption spectrometer. As the temperature is raised in steps, the stages of drying, thermal decomposition of the matrix and thermal cleavage into free atoms are passed through in that order. The absorption of the atoms is measured in the light path of the atomic absorption spectrometer at a wavelength specific to the element. Interferences due to background absorption resulting from high salt concentrations can be suppressed with a background correction system such as the Zeeman effect. The concentrations are calculated by means of calibration curves.

B.6.3 Reagents

Only reagents of a recognized analytical grade, water conform to the grade 3 in accordance with EN ISO 3696 and high-purity grade hydrochloric acid shall be used.

B.6.3.1 Hydrochloric acid (HCl), solution, mass fraction 30 %.

B.6.3.2 Element stock solutions, c_0 (Cd, Cr, Ni, Pb) = 1 g/l.

B.6.3.3 Standard element solutions, c_1 (Cd, Cr, Ni, Pb) = 10 mg/l, to be prepared by dilution of B.6.3.2.

B.6.3.4 Standard element solutions, c_2 (Cr, Ni, Pb) = 1 mg/l and c_3 (Cd) = 0,1 mg/l, to be prepared by dilution of B.6.3.3.

B.6.3.5 Element reference solutions, to be prepared by pipetting 1 ml, 2 ml, 4 ml, 8 ml and 10 ml of each of the standard solutions (B.6.3.4) into a series of 100 ml volumetric flasks (B.6.4.2), adding 3 ml hydrochloric acid (B.6.3.1) and filling up to the mark with water. The chromium, nickel and lead concentration of the reference solutions will be 10 µg/l, 20 µg/l, 40 µg/l, 80 µg/l and 100 µg/l. The cadmium concentration of the reference solutions will be 1 µg/l, 2 µg/l, 4 µg/l, 8 µg/l and 10 µg/l.

B.6.3.6 Sample blank solution.

B.6.4 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

B.6.4.1 Pipettes, capacity 1 ml, 5 ml and 10 ml.

B.6.4.2 Volumetric flask, capacity 100 ml.

B.6.4.3 Atomic absorption spectrometer equipped with graphite furnace and temperature controller.

B.6.4.4 Electrode-less discharge or hollow cathode lamps for the determination of cadmium, chromium, nickel and lead.

B.6.4.5 Background compensator (e.g. Zeeman).

B.6.4.6 Auto-sampler, capable of being adjusted to pipetting volumes from 5 µl to 20 µl.

B.6.5 Procedure

B.6.5.1 Spectrometer settings

Adjust in accordance with the operating manual issued by the manufacturer the operating parameters of the graphite furnace atomic absorption spectrometer (B.6.4.3). The spectrometer is operated at the wavelength specific for each element:

Cd : 228,8 nm;

Cr : 357,9 nm;

Ni : 232,0 nm;

Pb : 283,3 nm.

A suitable background correction (see B.6.4.5) is highly recommended. Optimized heat programs are used for each atomization of Cd, Cr, Ni and Pb, an example is given in B.6.5.2.

B.6.5.2 Measurement of cadmium, chromium, nickel and lead

Transfer 10 ml of the sample solution (5.2.3.2.5) with a pipette (B.6.4.1) to a 100 ml volumetric flask (B.6.4.2), add 1 ml of hydrochloric acid (B.6.3.1), and make up with water. Inject 10 µl of this sample solution in the graphite tube by means of an auto-sampler (B.6.4.6). Then start the heat programme, (e.g. see Table B.1).

Table B.1-Heat programme of the spectrometer

Cd, Pb	Ramp, in s	Hold, in s	Temperature in °C
Preheating	5	10	90
Drying	20	10	130
Ashing	30	10	850
Atomisation	0	3	1 800
Cleaning	1	4	2 650
Cr, Ni			
Preheating	5	10	90
Drying	20	10	130
Ashing	30	10	1 400
Atomisation	0	4	2 500
Cleaning	1	4	2 650

Carry out the measurements with gas-stop mode during the atomization phase. Use the L'vov platform for cadmium, nickel and lead. Wall atomization is preferred for chromium. Measure the absorption using the specific lamp (B.6.4.4) of the atomized elements cadmium, chromium, nickel and lead at the specific wavelength given in B.6.5.1.

B.6.5.3 Calibration function

To provide the calibration curve measure the blank solution and at least five reference solutions (B.6.3.5), in the same way as the sample solutions, so that 10 µl of the reference solutions are also injected in the graphite tube. The further procedure is the same as in B.6.5.2, the standard addition method is strongly recommended to avoid matrix effects.

B.6.5.4 Calculation

The cadmium, chromium, nickel and lead concentration in the sample solution are read out from the calibration curve and the calculation is the same as in B.4.5.4.

The cadmium, chromium, nickel and lead concentrations (C_s) for the iron salts in milligrams per kilogram of iron product are calculated using the following equation:

$$C_s = \frac{C_e \times 100 \times 200}{10 \times 1000 \times 20} = \frac{C_e}{10} \tag{7}$$

where

C_e is the element concentration in the sample solution in micrograms per litre.

NOTE 1 An alternative method for determination of cadmium, chromium, nickel and lead with the ICP optical emission spectrometry is described in annex C.

NOTE 2 The following equation gives the relation between the concentration C_f in milligrams per kilogram of Fe (II) and the concentration C_s in milligrams per kilogram of product: $C_f = C_s$ x 100/ $C_{(\text{II})}$

Annex C
(informative)

Determination of cadmium, chromium, nickel and lead (inductively coupled plasma optical emission spectrometry (ICP/OES))

C.1 General

This method is suitable for the determination of cadmium, chromium, nickel and lead in iron salts.

The suitable concentration ranges of this method are:

Cd : 1 mg/kg to 50 mg/kg;

Cr : 1 mg/kg to 50 mg/kg;

Ni : 1 mg/kg to 50 mg/kg;

Pb : 1 mg/kg to 50 mg/kg.

For higher concentrations the sample solution shall be diluted.

C.2 Principle

The basis of this method is the measurement of atomic emission by an optical spectroscope technique. Sample solutions are nebulized and the aerosol, that is produced with argon gas, is transported to the plasma torch where excitation occurs. Characteristic atomic line emission spectra are produced by an inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The concentrations are calculated by means of calibration curves.

C.3 Reagents

All reagents shall be of a recognized analytical grade and the water used shall conform to the grade 3 in accordance with EN ISO 3696.

C.3.1 Hydrochloric acid (HCl), solution, mass fraction 30 %.

C.3.2 Element stock solutions, c_0 (Cd, Cr, Ni, Pb) = 1 g/l.

C.3.3 Multi-element standard solution, to be prepared by pipetting 10 ml of each of the element stock solutions (C.3.2) to one 1 000 ml volumetric flask, adding 10 ml hydrochloric acid (C.3.1) and filling up to the mark with water. The concentration c_1 of the multi-element standard solution will be Cd: 10 mg/l, Cr: 10 mg/l, Ni: 10 mg/l and Pb: 10 mg/l.

C.3.4 Multi-element reference solutions, to be prepared by pipetting 1 ml, 5 ml, 10 ml, 20 ml and 50 ml of the multi-element standard solution (C.3.3) into a 100 ml volumetric flask, adding 25 ml hydrochloric acid (C.3.1) and filling up with water. The Cd, Cr, Ni and Pb concentrations of the multi-element reference solutions will be 0,1 mg/l, 0,5 mg/l, 1 mg/l, 2 mg/l and 5 mg/l.

C.3.5 Sample blank solution.

C.4 Apparatus

Ordinary laboratory apparatus and glassware together with the following.

C.4.1 ICP optical emission spectrometer.

C.4.2 Argon gas supply.

C.5 Procedure

C.5.1 Spectrometer settings

Adjust the operating parameters of the ICP optical emission spectrometer in accordance with the operating manual issued by the manufacturer. Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects shall be investigated and established for each individual analyte line on that particular instrument. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain quality control data confirming instrument performance and analytical results.

A background correction procedure is highly recommended. The analytical lines at wavelength:

Cd : 228,80 nm;

Cr : 357,87 nm;

Ni : 231,60 nm;

Pb : 220,35 nm.

are suitable for the determination of these elements in an iron matrix.

C.5.2 Calibration, measurement and calculation of the elements cadmium, chromium, nickel and lead

An analytical program is designed to calibrate and measure cadmium, chromium, nickel and lead in the iron salt solution. To provide the calibration curves measure the blank solution (C.3.5) and the five multi-element reference solutions (C.3.4). Subtract the emission intensities from the blank solution from the reference solutions. These corrected emission intensities versus the concentrations are the calibration functions which are calculated by means of regression analysis.

Spray the sample solution (5.2.3.2.5) with argon gas (C.4.2) in a well adjusted and calibrated ICP-optical emission spectrometer (C.4.1). Run the analytical program and record the results.

The cadmium, chromium, nickel and lead concentrations (C_s) of the iron salts in milligrams per kilogram iron product are calculated using the following equation:

$$C_s = C_e \times 10 \tag{8}$$

where

C_e is the element concentration in milligrams per litre in the sample solution.

NOTE: The following equation gives the relation between the concentration C_f in milligrams per kilogram of Fe (II) and the concentration C_s in milligrams per kilogram of product: $C_f = C_s \times 100 / C_{(II)}$

Bibliography

[1] 98/83/EC, *Council Directive of 3 November 1998 on the Quality of Water intended for Human Consumption.*

[2] 67/548/EEC, *Council Directive of 27th June 1967 on the approximation of the laws, regulations and administrative provisions relating to the classification, packaging and labelling of dangerous substances and its amendments and adaptations.*

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