SLOVENSKI STANDARD
SIST EN 17215:2019

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Kemikalije, ki se uporabljajo za pripravo pitne vode - Sredstvo za strjevanje na osnovi železa - Analitske metode

Chemicals used for treatment of water intended for human consumption - Iron-based coagulants - Analytical methods

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Flockungsmittel auf Eisenbasis - Analysenverfahren

Produits chimiques utilisés pour le traitement de l'eau destinée à la consommation humaine - Coagulants à base de fer - Méthodes d'analyse

Ta slovenski standard je istoveten z: **EN 17215:2019**

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13.060.20	Pitna voda	Drinking water
71.100.80	Kemikalije za čiščenje vode	Chemicals for purification of water

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EUROPEAN STANDARD

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English Version

Chemicals used for treatment of water intended for human consumption - Iron-based coagulants - Analytical methods

Produits chimiques utilisés pour le traitement de l'eau destinée à la consommation humaine - Coagulants à base de fer - Méthodes d'analyse

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Flockungsmittel auf Eisenbasis - Analysenverfahren

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

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European foreword

This document (EN 17215:2019) 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of an identical text or by endorsement, at the latest by September 2019, and conflicting national standards shall be withdrawn at the latest by September 2019.

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1 Scope

This document is applicable to iron-based coagulants used for treatment of water intended for human consumption. It specifies analytical methods to be used for products described in EN 888 (Iron (III) chloride), EN 889 (Iron (II) sulfate), EN 890 (Iron (II) sulfate, solution), EN 891 (Iron (III) chloride sulfate) and EN 14664 (Iron (III) sulfate, solid).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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*

ISO 5790:1979, *Inorganic chemical products for industrial use – General method for determination of chloride content – Mercurimetric method*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

3.1
laboratory sample
sample as prepared for sending to the laboratory and intended for inspection or testing

Note 1 to entry: in accordance with ISO 8213.

3.2
test sample
sample prepared from the laboratory sample and from which test portions will be taken

Note 1 to entry: in accordance with ISO 6206:1979.

3.3
test portion
quantity of material drawn from the test sample (or from the laboratory sample if both are the same) and on which the test or observation is actually carried out

Note 1 to entry: in accordance with ISO 6206:1979.

4 Methods of analysis

The methods to analyse the main product of standards EN 888, EN 889, EN 890, EN 891 and EN 14664 are listed in Table 1.

The methods to be used for analysis of iron-based coagulants and the principles of each method are listed in Table 1 and described in full in Annex A (normative methods) and in Annex B (informative methods) for analysis of iron content. The methods to be used for other parameters are listed in Table 2 and described in Annexes C, D and E.

Table 1 — Determination of the iron content (normative and informative method)

Standard number	Main product	Test method annex and principle	
EN 888	Iron (III) chloride	A.3 B.2	Iron (III) chloride is determined as iron (III) content in the test sample. Iron (III) content is determined as the difference between total iron content and iron (II) content.
EN 889	Iron (II) sulfate	A.1 B.1	Iron (II) sulfate is determined as iron (II) content in the test sample by titrimetry with cerium sulfate solution.
EN 890	Iron (III) sulfate, solution	A.3 B.2	Iron (III) sulfate is determined as iron (III) content in the test sample. Iron (III) content is determined as the difference between total iron content and iron (II) content.
EN 891	Iron (III) chloride sulfate	A.3 B.2	Iron (III) chloride sulfate is determined as iron (III) content in the test sample. Iron (III) content is determined as the difference between total iron content and iron (II) content.
EN 14664	Iron (III) sulfate, solid	A.3 B.2	Iron (III) sulfate is determined as iron (III) content in the test sample. Iron (III) content is determined as the difference between total iron content and iron (II) content.

NOTE 1 An alternative method for determination of iron (II) and iron (III) content is described in Annex B.

Table 2 — Determination of parameters (normative and informative methods)

Parameter	EN 888	EN 889	EN 890	EN 891	EN 14664	Method	Principle
Insoluble matters	X	X	X	X	X	C.2	Weighted mass of the filtered sample
Iron (II)	X		X	X	X	A.1 B.1	Determination of iron (II) by titration against cerium sulfate Determination of iron (II) by titration against potassium dichromate
Free acid	X		X	X	X	C.1	Titration with sodium hydroxide
Antimony	X	X	X	X	X	C.4 D E	hydride generation atomic absorption spectrometry ICP/OES ICP/MS

Parameter	EN 888	EN 889	EN 890	EN 891	EN 14664	Method	Principle
Arsenic	X	X	X	X	X	C.4 D E	hydride generation atomic absorption spectrometry ICP/OES ICP/MS
Cadmium	X	X	X	X	X	C.6 D E	graphite furnace atomic absorption spectrometry ICP/OES ICP/MS
Chromium	X	X	X	X	X	C.6 D	graphite furnace atomic absorption spectrometry ICP/OES
Manganese	X	X	X	X	X	C.3 D E	flame atomic absorption spectrometry (FAAS) ICP-OES ICP-MS
Mercury	X	X	X	X	X	C.5	cold vapour atomic absorption spectrometry
Lead	X	X	X	X	X	C.6 D E	graphite furnace atomic absorption spectrometry ICP/OES ICP/MS
Nickel	X	X	X	X	X	C.6 D	graphite furnace atomic absorption spectrometry ICP/OES
Selenium	X	X	X	X	X	C.4 D E	hydride generation atomic absorption spectrometry ICP/OES ICP/MS

NOTE 2 An alternative method for determination of arsenic, antimony, cadmium, lead and selenium with the ICP mass spectrometry is described in Annex E. An alternative method for analysis on arsenic, antimony, cadmium, chromium, manganese, nickel, lead and selenium with the ICP/OES is described in Annex D.

5 Sampling

5.1 General

Observe the general recommendations in ISO 3165 and take into account ISO 6206.

5.2 Solids

Prepare the laboratory sample required by the relevant procedure described in ISO 8213.

5.3 Solutions

5.3.1 Sampling from drums and bottles

5.3.1.1 General

Mix the contents of each container to be sampled by shaking the container, by rolling it or by rocking it from side to side, taking care not to damage the container or spill any of the liquid.

If the design of the container is such (for example, a narrow-necked bottle) that it is impracticable to use a sampling implement, take a sample by pouring after the contents have been thoroughly mixed. Otherwise, proceed as described in 5.3.1.3.

Examine the surface of the liquid. If there are signs of surface contamination, take samples from the surface as described in 5.3.1.2. Otherwise, take samples as described in 5.3.1.3.

5.3.1.2 Surface sampling

Take a sample using a suitable ladle. Lower the ladle into the liquid until the rim is just below the surface, so that the surface layer runs into it. Withdraw the ladle just before it fills completely and allow any liquid adhering to the ladle to drain off. If necessary, repeat this operation so that, when the other selected containers have been sampled in a similar manner, the total volume of sample required for subsequent analysis is obtained.

5.3.1.3 Bottom sampling

Take a sample using an open sampling tube, or a bottom-valve sampling tube, suited to the size of container and the viscosity of the liquid.

When using an open sampling tube, close it at the top and then lower the bottom end to the bottom of the container. Open the tube and move it rapidly so that the bottom of the tube traverses the bottom of the container before the tube is filled. Close the tube, withdraw it from the container and allow any liquid adhering at the outside of the tube to drain off.

When using a bottom-valve sampling tube, close the valve before lowering the tube into the container and then proceed in a similar manner to that when using an open sampling tube.

5.3.2 Sampling from tanks and tankers

A representative sample should be taken as appropriate:

a) from the surface of the liquid, using a ladle as described in 5.3.1.2;

b) from the bottom of the tank or tanker, using a sampling tube as described in 5.3.1.3 or using specially designed bottom-sampling apparatus;

c) from one or more positions, depending on the overall depth, between the bottom and the surface using a weighted sampling can.

6 Expression of results

6.1 Iron content

The iron content shall be expressed as iron (II) or iron (III) mass fraction in %.

6.2 Free acid

Free acid shall be expressed as mass fraction in %.

6.3 Insoluble matters

Insoluble matters shall be expressed as mass fraction in %.

6.4 Impurities

Impurities shall be expressed as mg/kg.

6.5 Repeatability

Each laboratory shall calculate the repeatability of the method under their laboratory conditions according to the procedure.

Annex A
(normative)

Analysis of the iron content

NOTE The strengths of iron salt solutions are measured on the basis of the iron (II) and iron (III) concentration alone.

A.1 Determination of iron (II) concentration by titration against cerium (IV) sulfate

A.1.1 General

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

A.1.2 Principle

Iron (II) is directly titrated with cerium (IV) sulfate solution, which oxidises it to iron (III).

A.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.

A.1.3.1 Hydrochloric acid solution (HCl) 5,0 mol/l

A.1.3.2 1,10-Phenanthroline ferrous complex solution (ferroin, ferrous indicator) ([Fe(o-phen)$_3$]SO$_4$) 0,025 mol/l

A.1.3.3 Cerium (IV) sulfate solution, in 10 % w/v sulfuric acid, (Ce(SO$_4$)$_2$) 0,1 mol/l

A.1.4 Apparatus

Ordinary laboratory apparatus and glassware.

A.1.5 Procedure

A.1.5.1 Test solution

Weigh to the nearest 0,01 g, between 5 g and 10 g (m) of the laboratory sample and transfer to a conical flask. Dilute with 50 ml ± 5 ml of water.

A.1.5.2 Determination

With mixing, add 0,5 ml of hydrochloric acid (A.1.3.1) to the conical flask. A clear deep yellow solution is formed.

Add 2 to 4 drops (only) of the ferrous indicator (A.1.3.2). The solution will turn red.

Titrate the solution with cerium (IV) sulfate solution (A.1.3.3) from a volumetric burette. The end point is achieved when a colour change from red to blue-green occurs and is sharp. Record the volume (V) of cerium (IV) sulfate required for the titration.

A.1.6 Calculation and expression of results

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

$$C_{(II)} = \frac{V \times 0,5585}{m} \quad \text{(A.1)}$$

where

V is the volume, in millilitres, of cerium (IV) sulfate required for the titration;

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

A.2 Determination of total iron

A.2.1 General

This method applies to the products at the supply concentration.

A.2.2 Principle

Iron is reduced by tin (II) chloride and is subsequently titrated with cerium (IV) sulfate.

A.2.3 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.

A.2.3.1 Hydrochloric acid, HCl concentrated 1,19 g/ml

A.2.3.2 Tin (II) chloride solution ($SnCl_2 \cdot 2H_2O$) 0,5 mol/l

Dissolve 22,6 g of $SnCl_2 \cdot 2H_2O$ with 20 ml of hydrochloric acid (A.2.3.1) and dilute with water to 200 ml. Keep this solution in the dark.

A.2.3.3 Mercury (II) chloride, saturated solution ($HgCl_2$) 0,27 mol/l

A.2.3.4 1,10-Phenanthroline ferrous complex solution (ferroin, ferrous indicator) ($[Fe(o\text{-}phen)_3]SO_4$) 0,025 mol/l

A.2.3.5 Cerium (IV) sulfate ($Ce(SO_4)_2$) 0,1 mol/l

A.2.4 Apparatus

Ordinary laboratory apparatus and glassware.

A.2.5 Procedure

A.2.5.1 Test solution

Weigh to the nearest 0,01 g, between 5 g and 10 g of the laboratory sample (m) and transfer to a 200 ml volumetric flask, dilute to the mark with water. Pipette 10 ml ± 0,2 ml of this solution into a 500 ml conical flask.

A.2.5.2 Determination

Add 3 to 4 drops of hydrochloric acid (A.2.3.1) and heat until boiling while stirring.