SLOVENSKI STANDARD
SIST EN 1302:2025

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Nadomešča:
SIST EN 1302:2000/AC:2002

Kemikalije, ki se uporabljajo za pripravo pitne vode - Koagulanti na osnovi aluminija - Analitske metode

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

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Flockungsmittel auf Aluminiumbasis - Analytische Methoden

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

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EUROPEAN STANDARD
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English Version

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

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

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Flockungsmittel auf Aluminiumbasis - Analytische Methoden

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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (EN 1302:2024) 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of an identical text or by endorsement, at the latest by June 2025, and conflicting national standards shall be withdrawn at the latest by June 2025.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 1302:1999.

In comparison with the previous edition, the following technical modifications have been made:

— Replacement of the withdrawn aluminium product standards EN 881 and EN 883 by EN 17034.

— Replacement of the withdrawn mercury analysing standard ISO 5666-1:1983 by ISO 12846:2012.

— Removal of the withdrawn standard ISO 6382:1981. As there was no replacement, the analysing method for silicate content based on this standard was removed. To analyse silicon this standard applies ICP-OES as given in Annex B.

— Removal of routine methods used to determinate arsenic. Method: Silver diethyldithiocarbamate spectrophotometric method based on standard ISO 6595:1982 as this standard has been withdrawn without replacement. This standard applies ICP-OES as given in Annex B.

— Removal of routine methods used to determinate iron. Method: 1,10-Phenanthroline spectrophotometric method based on standard ISO 6685:1982 as this standard has been withdrawn without replacement. This standard applies ICP-OES as given in Annex B.

— Removal of a method for determination of iron (total and Fe^{2+}) (volumetric method) as the method uses potassium dichromate and there are other options for iron determination. This standard applies ICP-OES as given in Annex B.

— Update of the clause titles to correspond the CEN Standard form.

— Update of Table 1 and 2 to correspond the analytical methods available in the standard.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

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1 Scope

This document is applicable to aluminium-based coagulants used for treatment of water intended for human consumption. It specifies analytical methods to be used for products described in EN 878, EN 882, EN 885, EN 886, EN 887, EN 935 and EN 17034.

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 878, *Chemicals used for treatment of water intended for human consumption — Aluminium sulfate*

EN 882, *Chemicals used for treatment of water intended for human consumption — Sodium aluminate*

EN 885, *Chemicals used for treatment of water intended for human consumption — Polyaluminium chloride hydroxide silicate*

EN 886, *Chemicals used for treatment of water intended for human consumption — Polyaluminium hydroxide silicate sulfate*

EN 887, *Chemicals used for treatment of water intended for human consumption — Aluminium iron (III) sulfate*

EN 935, *Chemicals used for treatment of water intended for human consumption — Aluminium iron(III) chloride (monomeric) and aluminium iron(III) chloride hydroxide (monomeric)*

EN 17034, *Chemicals used for treatment of water intended for human consumption — Aluminium chloride anhydrous, aluminium chloride basic, dialuminium chloride pentahydroxide and aluminium chloride hydroxide sulfate*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

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

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 6206:1979, *Chemical products for industrial use — Sampling — Vocabulary*

ISO 6227:1982, *Chemical products for industrial use — General method for determination of chloride ions — Potentiometric method*

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

3 Terms and definitions

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

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

— ISO Online browsing platform: available at https://www.iso.org/obp/

— IEC Electropedia: available at https://www.electropedia.org/

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

[SOURCE: ISO 6206:1979]

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

[SOURCE: 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

[SOURCE: ISO 6206:1979]

4 Methods of analysis

The methods of analysis are used to analyse the product quality. Table 1 lists the methods which shall be used for analysis of aluminium-based coagulants composition as commercial product and the principles of each method. The normative methods which shall be used are described in full in Annexes A and B. Other methods can be used for quality control. Examples of other methods are mentioned in Annex C.

Table 2 lists the methods which shall be used for analysis of other chemical parameters. These are applicable to all aluminium based coagulants (EN 878, EN 882, EN 885, EN 886, EN 887, EN 935 and EN 17034). The relevant limit value for each coagulant is available in the product standard.

Table 1 — Methods of analysis of the composition of commercial product

Determination	Reference method	Informative method	EN 878	EN 882	EN 885	EN 886	EN 887	EN 935	EN 17034	Principle	Annex
Aluminium	x		x		x	x	x	x	x	EDTA complexometric titration[a]	A.1
Aluminium		x	x		x	x	x	x	x	CDTA complexometric titration	C.8.
Aluminium	x			x						Gluconate	A.2
Iron	x		x	x	x	x	x	x	x	ICP-OES	B.7
Iron		x	x	x	x	x	x	x	x	FAAS	C.4
Iron		x	x	x	x	x	x	x	x	ICP-MS [b]	C.2
Sodium	x		x		x	x	x	x	x	ICP-OES	B.7
Sodium		x			x	x				FAAS	C.5
Sodium		x		x						Gluconate	A.2
Calcium		x			x	x			x	FAAS	C.6
Calcium	x				x	x			x	ICP-OES	B.7
Chloride	x				x		x		x	Potentiometric titration	B.1
Sulfate	x		x			x	x		x	Barium sulfate gravimetric titration	B.2
Silicon	x		x	x	x	x	x	x	x	ICP-OES	B.7
Free acid			x				x	x	x	Acidimetric titration	B.3
Basicity	x		x	x			x	x	x	Acidimetric titration, oxalate method	B.4
Basicity	x				x	x				Acidimetric titration, KF method	B.5
Basicity		x								Calculation method	C.7

Key
FAAS: Flame Atomic Absorption Spectrometry
AFS: Atomic Fluorescence Spectrometry
ICP-OES: Inductively Coupled Plasma Optical Emission Spectrometry
ICP-MS: Inductively Coupled Plasma Mass Spectrometry

[a] If the concentration of iron is higher than 0,1 %, the calulation needs to be corrected due to interferences of iron.
[b] This document does not give a description for this method of analysis.

Table 2 — Applicability methods of analysis to other parameters

Determination	Reference method	Routine method	Principle
Insoluble matter	B.6		Gravimetry
Arsenic	B.7		ICP-OES
		C.3	ICP-MS
		C.2	AAS
Cadmium	B.7		ICP-OES
		C.3	ICP-MS
		C.2	AAS
Chromium	B.7		ICP-OES
		C.3	ICP-MS
		C.2	AAS
Mercury	B.9		AAS (cold vapour)
		B.8	AFS
Nickel	B.7		ICP-OES
		C.3	ICP-MS
		C.2	AAS
Lead	B.7		ICP-OES
		C.3	ICP-MS
		C.2	AAS
Antimony	B.7		ICP-OES
		C.3	ICP-MS
		C.2	AAS
Selenium	B.7		ICP-OES
		C.3	ICP-MS
		C.2	AAS

Key
AAS: Atomic Absorption Spectrometry
AFS: Atomic Fluorescence Spectrometry
ICP-OES: Inductively Coupled Plasma Optical Emission Spectrometry
ICP-MS: Inductively Coupled Plasma Mass Spectrometry

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.

From the laboratory sample, prepare the test sample by grinding the solid samples until the particle size is below 2,5 mm and homogenizing, and homogenize the liquid samples.

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 Aluminium content

The aluminium content shall be expressed as aluminium mass fraction in % or as Al in grams per kg of product.

6.2 Iron

The iron content shall be expressed as iron mass fraction in % or ar Fe in grams per kg of product depending on analytical method.

6.3 Sodium, calcium, chloride, sulfate and silicate

The sodium, calcium, chloride or sulfate content shall be expressed as grams of substance per kilogram of product or as mass fraction in % depending on analytical method.

6.4 Free acid

Free acid shall be expressed as in grams of sulfuric acid per kilogram of product (H_2SO_4 g/kg) or in grams of hydrochloric acid per kilogram of product (HCl g/kg) or as mass fraction in %.

6.5 Basicity

The basicity shall be expressed in grams of hydroxide per kilogram of product (OH^- g/kg) or as the relative basicity expressed in moles of OH^- per moles of Al (OH/Al) or as in % (OH/AL *100/3).

6.6 Insoluble matters

Insoluble matters shall be expressed in mass fraction in % or in grams per kilogram of product.

6.7 Chemical parameters

The chemical parameters mentioned in Table 2 shall be expressed as milligrams of element per kilogram of product.

6.8 Repeatability

Each laboratory shall calculate the repeatability of the method under their laboratory conditions according to the procedure defined in ISO 5725-2.

Annex A
(normative)

Analysis of aluminium content

A.1 Determination of aluminium (EDTA complexometric method)

A.1.1 General

This method shall be used for the determination of aluminium in aluminium-based coagulants used for treatment of water intended for human consumption, as a reference method for products described in EN 878, EN 885, EN 886, EN 887, EN 935 and EN 17034.

If iron concentration is lower than 0,1 % Fe, the calculation needs to be corrected due to interference of Fe (see A.1.2)

A.1.2 Principle

Dissolution in water, in the case of solid products, or dilution with water in the case of products in solution, of a test sample.

Complexation of aluminium, in a hot acidic medium with an excess of ethylenediaminetetraacetic acid (EDTA) solution. Titration of the excess EDTA with a standard volumetric solution of zinc in the presence of xylenol orange as indicator.

$Al^{3+} + EDTA^{4-} \rightarrow AlEDTA^{-}$

$Zn^{2+} + EDTA^{4-} \rightarrow ZnEDTA^{2-}$

If the Fe concentration is > 0,1 %, the concentration X_1 will be corrected, due to interferences of Fe, using the calculation mentioned in A.1.6.

A.1.3 Reagents

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

A.1.3.1 Sodium acetate solution, 80 g/l.

A.1.3.2 Sodium hydroxide solution, 100 g/l.

A.1.3.3 Hydrochloric acid solution 1:1

Dilute one volume of hydrochloric acid (P = 1,19 g/ml) with one volume of water.

A.1.3.4 Hydrochloric acid, 36,5 g/l, c(HCl) = 1 mol/l.

A.1.3.5 Disodium ethylenediaminetetraacetate dihydrate (NaEDTA), standard volumetric solution, $c(C_{10}H_{14}N_2O_8Na_2{\cdot}2H_2O)$ = 0,05 mol/l. Weigh to the nearest 0,000 1 g, 18,625 g of NaEDTA. Dissolve in water, transfer the solution quantitatively to a 1 000 ml volumetric flask. Dilute to volume with water and homogenize.

NOTE Commercial standard volumetric solution could be used.

A.1.3.6 Zinc, standard volumetric solution, c(Zn) = 0,05 mol/l. Weigh to the nearest 0,000 1 g, 6,537 g (m)of pure zinc (minimum content 99,9 % (m/m)).

Dissolve in 60 ml of hydrochloric acid solution (A.1.3.3). During the reaction, cover the beaker with a watch-glass. At the end of the reaction, boil the solution for 10 min, then cool to room temperature. Dilute to about 500 ml with water and add sodium acetate solution (A.1.3.1) until a pH of 5,5 ± 0,1 is obtained. Transfer the solution quantitatively to a 2 000 ml volumetric flask. Dilute to volume with water and homogenize.

NOTE 1 If the mass of zinc is not exactly that stated above, the zinc concentration is given by the equation:

$$c\left(\mathrm{Zn}\right) = \frac{m}{2 \times 65,37}$$

where

m is the mass in grams of zinc weighed;

65,37 is the relative molecular mass of zinc;

c(Zn) is the concentration of zinc solution, in moles per litre, calculated to the fourth significant figure.

NOTE 2 Commercial standard solution could be used.

A.1.3.7 Buffer solution, pH 5,5.

Weigh 50 g of sodium acetate trihydrate ($CH_3COONa{\cdot}3H_2O$). Dissolve in 500 ml of water and add glacial acetic acid (CH_3COOH) until a pH of 5,5 ± 0,1 is obtained.

A.1.3.8 Xylenol orange.

Grind 1,0 g of xylenol orange with 99 g of potassium nitrate in a mortar until a homogeneous mass is obtained.

A.1.4 Apparatus

Ordinary laboratory apparatus and glassware, and optionally:

A.1.4.1 Automatic titrator and photometer with fibre optic probe.

A.1.4.2 Microwave equipment.

A.1.5 Procedure

A.1.5.1 Preparation of the test solution

Weigh, to the nearest 0,001 g, about 25 g of the test sample (m_0) into a 400 ml beaker.

Add approximately 150 ml of water at 80 °C to 90 °C. Stir until dissolved, using a glass stirrer.

Transfer quantitatively to a 500 ml volumetric flask. Dilute to volume with water and homogenize. Filter if necessary through a filter paper (particle retention size 2,5 µm) (test solution V_0).

Place V_1 ml of this solution (see Table A.1) into a 200 ml volumetric flask. Dilute to volume with water and homogenize (diluted test solution V_2).

Table A.1 — Aliquot portion V_1 for Al determination (EDTA method)

Expected content Al g/kg	Volume V_1 ml
< 27	100
27 to 66	50
66 to 133	20
133 to 265	10

A.1.5.2 Blank test

Perform a blank test following the same procedure and using the same quantities of all the reagents as indicated in A.1.5.3. Record the volume used for the titration (V_5).

A.1.5.3 Determination

Transfer 100,0 ml of solution (A.1.5.1) (V_3) to a 250 ml beaker and adjust to pH 5 to 6 with hydrochloric acid solution (A.1.3.3) or sodium hydroxide solution (A.1.3.2). Add 5 ml of hydrochloric acid solution (A.1.3.4) and 50,0 ml of the standard volumetric solution of EDTA (A.1.3.5). Cover with a watch glass. Heat the solution at 80 °C to 90 °C for at least 20 min. Cool to room temperature. Rinse the watch glass with water into the beaker. Neutralize with sodium acetate solution (A.1.3.1). The pH value shall be between 7 to 7,5 and add 10 ml of the buffer solution (A.1.3.7).

Add 30 mg to 50 mg of xylenol orange mixture (A.1.3.8). Titrate with the standard volumetric zinc solution (A.1.3.6) until the indicator changes from yellow to definite red or determine the equivalence point using an automatic titrator. Record the volume (V_4) used.

If microwave equipment (A.1.4.2) is used, the volume V_1 and the volume of the aliquot portion (A.1.5.1) can be different from those indicated above. Transfer the test portion to a 250 ml conical flask and adjust to pH 5 to 6 with hydrochloric acid (A.1.3.3) or sodium hydroxide solution (A.1.3.2). Add 5 ml of hydrochloric acid solution (A.1.3.4) and the suitable volume of the standard volumetric solution of EDTA (A.1.3.5). Transfer to the microwave equipment. Operate the microwave equipment at a power setting to achieve a temperature at 80 °C to 90 °C for 15 min. Then cool to room temperature. Transfer quantitatively to a 250 ml beaker or to the automatic titration cell.

If an automatic titrator (A.1.4.1) is used, the volume of the aliquot portion (A.1.5.1) and the volumes of the reagents can be different from those indicated above. They should be such that the required precision is achieved.

A.1.6 Expression of results

The aluminium content, X_1 expressed in grams of aluminium per kilogram of product (Al g/kg) is given by the equation:

$$X_1 = 0{,}026\ 98 \times \left(V_5 - V_4\right) \times c \times \frac{V_2}{V_3} \times \frac{V_0}{V_1} \times \frac{1\ 000}{m_0}$$