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Chemicals used for treatment of water intended for human consumption — Sodium chloride for on site electrochlorination using membrane cells

National foreword

This British Standard is the UK implementation of EN 16370:2013.

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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 - Sodium chloride for on site electrochlorination using membrane cells

Produits chimiques utilisés pour le traitement de l'eau destinée à la consommation humaine - Chlorure de sodium pour la génération électrochimique de chlore au moyen d'électrolyseurs à membrane

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Natriumchlorid zur elektrochemischen Erzeugung von Chlor vor Ort mittels Membranzellen

This European Standard was approved by CEN on 5 July 2013.

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Contents

Foreword

This document (EN 16370:2013) 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of an identical text or by endorsement, at the latest by February 2014, and conflicting national standards shall be withdrawn at the latest by February 2014.

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Introduction

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

a) this European 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 this European 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 European Standard is subject to regulation or control by National Authorities.

1 Scope

This European Standard is applicable to sodium chloride intended for on site electrochlorination of water intended for human consumption using membrane cells. It describes the characteristics and specifies the requirements and the corresponding test methods for sodium chloride (see Annex B). It gives information on its use in water treatment.

2 Normative references

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

EN 901:2013, *Chemicals used for treatment of water intended for human consumption — Sodium hypochlorite*

EN 973:2009, *Chemicals used for treatment of water intended for human consumption — Sodium chloride for regeneration of ion exchangers*

EN 14805:2008, *Chemicals used for treatment of water intended for human consumption — Sodium chloride for on site electrochlorination using non-membrane technology*

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

ISO 2479, *Sodium chloride for industrial use — Determination of matter insoluble in water or in acid and preparation of principal solutions for other determinations*

ISO 2480, *Sodium chloride for industrial use — Determination of sulphate content — Barium sulphate gravimetric method*

ISO 2482, *Sodium chloride for industrial use — Determination of calcium and magnesium contents — EDTA complexometric methods*

ISO 2483, *Sodium chloride for industrial use — Determination of the loss of mass at 110 °C*

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

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

ISO 6227, *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 Description

3.1 Identification

3.1.1 Chemical name

Sodium chloride.

3.1.2 Synonym or common name

Salt.

3.1.3 Relative molecular mass

58,45.

3.1.4 Empirical formula

NaCl.

3.1.5 Chemical formula

NaCl.

3.1.6 CAS Registry Number1)

7647-14-5.

3.1.7 EINECS Reference2)

231-598-3.

3.2 Commercial forms

The product is available as rock salt, sea salt or evaporated salt, and it is supplied as free-flowing crystals or their compacted forms.

3.3 Physical properties

3.3.1 Appearance

The product is white and crystalline.

3.3.2 Density

The density of the solid crystal is 2,16 g/cm^3 at 20 °C.

The bulk density depends on the particle size distribution.

3.3.3 Solubility (in water)

The solubility of the product depends on the temperature as given in Figure 1.

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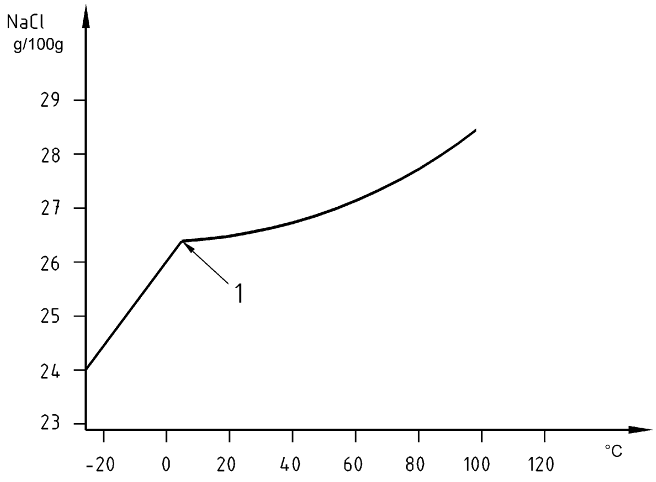
1) Chemical Abstract Service Registry Number.

2) European Inventory of Existing Commercial Chemical Substances.



Key

1 Transition point NaCl → NaCl · 2 H_2O

Temperature °C	NaCl solution Mass fraction in g/100 g
- 10	25,00
0	26,34
10	26,35
20	26,43
30	26,56
40	26,71
50	26,89
60	27,09
70	27,30
80	27,53
90	27,80
100	28,12

Figure 1 — Solubility curve for sodium chloride in water

3.3.4 Vapour pressure

Not applicable.

3.3.5 Boiling point at 100 kPa[3)]

Not applicable.

3.3.6 Melting point

802 °C.

3.3.7 Specific heat

Approximately 850 J/(kg · K) at 25 °C for the solid.

3.3.8 Viscosity (dynamic)

The viscosity of the saturated solution at 20 °C is approximately 1,9 mPa · s.

3.3.9 Critical temperature

Not applicable.

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3) 100 kPa = 1 bar.

3.3.10 Critical pressure

Not applicable.

3.3.11 Physical hardness

The hardness of solid salt is given as 2 to 2,5 on the Mohs' scale of hardness.

3.4 Chemical properties

Sodium chloride is stable, non-volatile and aqueous solutions have good electrical conductivity.

Sodium chloride reacts with a number of acids (e.g. sulfuric acid, phosphoric acid) and strong oxidising agents. The reactions are often complex and require heat for completion.

NOTE Under certain conditions, a sodium chloride solution can cause corrosion of metallic surfaces.

4 Purity criteria

4.1 General

This European Standard specifies the minimum purity requirements for sodium chloride for on site electrochlorination of water intended for human consumption using membrane cells. The basic quality of the applied salt shall meet the requirements of Codex Alimentarius for Food Grade Salt, because all components of the electrolysed salt are added to drinking water. As the treatment chemical produced by this process is chlorine or sodium hypochlorite, limits have also been calculated from the requirements of EN 901 and of EN 973 under consideration of the demand of minimisation for those impurities commonly present in the product and the chemical parameters. 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.

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 lead 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 composition of the commercial product shall conform to Table 1.

Table 1 — Composition/Additives

Composition	Mass fraction of dry product
NaCl	≥ 99,9 %
$[Fe(CN)_6]^{4-}$ [a)]	absent
[a)] Analytical method of $[Fe(CN)_6]^{4-}$: see EN 973:2009, B.3.”	

4.3 Impurities and by-products

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

Table 2 — Impurities and by-products

Impurity/by-product	Limit in mass fraction of NaCl content (mg/kg)	
	Grade 1	Grade 2
Moisture, dry product	≤ 1 000	≤ 1 000
Moisture, wet product	≤ 50 000 [a]	≤ 50 000
Water-insoluble matter	≤ 500	≤ 1000
Bromide	≤ 50	≤ 250
Iron	≤ 2	≤ 2
Manganese	≤ 0,5	≤ 1
Calcium + magnesium	≤ 25	≤ 300
Sulfate	≤ 400	≤ 600
[a] The flowability of the products is reduced by a high moisture content; further information is available from the supplier of the product.		

The grade should be selected based on the requirements of the electrochlorination equipment depending on the susceptibility of the membrane to fouling. The electrochlorination equipment manufacturers will specify which grade can be used to meet the requirements of the Drinking Water Directive or national regulations.

4.4 Chemical parameters

The products shall conform to the requirements specified in Table 3.

Table 3 — Chemical parameters

Parameter	Limits in mg/kg of commercial product
Arsenic (As)	≤ 0,3
Cadmium (Cd)	≤ 0,5
Chromium (Cr)	≤ 0,5
Mercury (Hg)	≤ 0,1
Nickel (Ni)	≤ 0,5
Lead (Pb)	≤ 2
Antimony (Sb)	≤ 2
Selenium (Se)	≤ 2
NOTE 1 The limits result from requirements of Codex Alimentarius (C.A.) for Food Grade Salt and EN 901 and EN 973 with respect to the demand of minimisation. NOTE 2 Other chemical parameters and indicator parameters as listed in EU Directive 98/83/EC (see [1]) are not relevant in sodium chloride.	

5 Test methods

5.1 Sampling

A test sample of about 500 g shall be taken for analysis, ensuring that it is representative of the whole batch, and taking account of ISO 3165 and ISO 6206. Prepare the laboratory sample(s) required in accordance with ISO 8213.

5.2 Analyses

5.2.1 Main product

The mass fraction in % of sodium chloride (NaCl) shall be determined by calculation, on the basis of the results of the determinations of sulfate (according to ISO 2480), halogens (according to ISO 6227), calcium and magnesium (according to ISO 2482), and loss of mass on drying (according to ISO 2483). Convert sulfate to calcium sulfate and unused calcium to calcium chloride, unless sulfate in sample exceeds the amount necessary to combine with calcium, in which case convert calcium to calcium sulfate and unused sulfate to first to magnesium sulfate and the remaining sulfate to sodium sulfate. Convert unused magnesium to magnesium chloride. Convert unused halogens to sodium chloride. Report the sodium chloride contents on a dry matter basis, multiplying the mass fraction in % of sodium chloride by 100/(100 - P), where P in the percentage of the loss of mass on drying (see 5.2.2.2).

5.2.2 Impurities

5.2.2.1 Water-insoluble matter

The content of water-insoluble matter shall be determined in accordance with ISO 2479.

5.2.2.2 Moisture content

The loss of mass at 110 °C shall be determined in accordance with ISO 2483.

5.2.2.3 Bromide

5.2.2.3.1 General

The present method describes a titrimetric method with sodium thiosulfate for the determination of total bromine and iodine in sodium chloride. The method is applicable to products of bromine and iodine content (expressed conventionally as bromide, Br^-) equal to or greater than 3 mg bromine per kilogram of salt. Bromine or bromide are equivalent for the expression of the results.

5.2.2.3.2 Principle

The sample is dissolved in water. Oxidation of iodide to iodate and bromide to bromate is achieved with hypochlorite in a buffered medium and the excess of oxidant is eliminated with formic acid. Free iodine, equivalent to the amount of the present iodate and bromate, is formed by addition of hydrochloric acid and potassium iodide. The free iodine is titrated with sodium thiosulfate using starch as indicator.

5.2.2.3.3 Reactions

$$I^- + 3\ ClO^- \rightarrow IO_3^- + 3\ Cl^- \quad (1)$$

$$Br^- + 3\ ClO^- \rightarrow BrO_3^- + 3\ Cl^- \quad (2)$$

Titration of the iodate and bromate follows the reactions:

$$IO_3^- + 6\ I^- + 6\ H^+ \rightarrow 3\ I_2 + I^- + 3\ H_2O \quad (3)$$

$$BrO_3^- + 6\ I^- + 6\ H^+ \rightarrow 3\ I_2 + Br^- + 3\ H_2O \quad (4)$$

NOTE Reactions (1) and (2) require for completion the presence of chloride ions that are added into the buffer solution.

5.2.2.3.4 Reagents

Unless otherwise stated, use only reagents of recognised analytical grade and only water conforming to grade 3 in accordance with EN ISO 3696.

5.2.2.3.4.1 **Calcium carbonate**, precipitated.

5.2.2.3.4.2 **Hydrochloric acid**, $c(HCl) \approx 4$ mol/l.

5.2.2.3.4.3 **Formic acid**, $c(HCOOH) \approx 3$ mol/l.

5.2.2.3.4.4 **Potassium iodide**, $\beta(KI) \approx 100$ g/l.

Prepare this solution on the day of use and store it in a dark bottle.

5.2.2.3.4.5 Sodium hypochlorite, $\beta(\text{NaClO}) \approx 19$ g/l.

Prepare this solution by dilution of a concentrated technical sodium hypochlorite solution. Renew the solution each week.

Commercial household solutions which can contain interfering additives should not be used.

5.2.2.3.4.6 Buffer solution

Dissolve 50 g of sodium dihydrogen orthophosphate dihydrate, ($NaH_2PO_4 \cdot 2\ H_2O$), 50 g of disodium hydrogen orthophosphate dodecahydrate ($Na_2HPO_4 \cdot 12\ H_2O$), 50 g of tetrasodium pyrophosphate decahydrate ($Na_4P_2O_7 \cdot 10\ H_2O$) and 150 g of sodium chloride (see note) in 650 ml of water.

NOTE As the same quantity of buffer solution is used for the sample and for the blank test solution, it is not necessary to use products completely free of iodine and bromine.

5.2.2.3.4.7 Sodium thiosulfate, standard volumetric solution, $c(Na_2S_2O_3) = 0{,}01$ mol/l.

Prepare this solution by dilution of a standard volumetric solution $c(Na_2S_2O_3) = 0{,}1$ mol/l and standardise with a potassium iodate solution [$c(1/6\ KIO_3) = 0{,}01$ mol/l].

5.2.2.3.4.8 Methyl red, 0,5 g/l solution in volume fraction 95 % ethanol.

5.2.2.3.4.9 Starch solution, 2 g/l solution.

Prepare this solution at the time of use from soluble starch.

5.2.2.3.5 Apparatus

Ordinary laboratory apparatus and:

5.2.2.3.5.1 Burette allowing the distribution and measurement of 0,01 ml.

5.2.2.3.6 Procedure

5.2.2.3.6.1 Test portion

Weigh, to the nearest 0,1 g, about 40 g of the test sample.

For bromine plus iodine content greater than 200 mg per kilogram of salt, the test portion should be reduced accordingly.

5.2.2.3.6.2 Test solution

Transfer the test portion (5.2.2.3.6.1) and 150 ml of water into a 500 ml conical flask, stir to dissolve.

5.2.2.3.6.3 Blank test solution

Transfer 150 ml of water into a 500 ml conical flask.

5.2.2.3.6.4 Determination

Proceed with the conical flasks prepared in (5.2.2.3.6.2) and (5.2.2.3.6.3) in the following way.

Add 4 drops of methyl red (5.2.2.3.4.8) and hydrochloric acid (5.2.2.3.4.2) until the solution turns red. Neutralise by addition of calcium carbonate (5.2.2.3.4.1) to a permanent slight cloudiness.

Add 6,0 ml of buffer solution (5.2.2.3.4.6) and 5,0 ml of sodium hypochlorite solution (5.2.2.3.4.5). Heat to (90 ± 2) °C with agitation to avoid local overheating and keep at this temperature for 20 min.

Add 10 ml of formic acid (5.2.2.3.4.3) and swirl. When CO_2 ceases to evolve, cool to about 20 °C, add 2,0 ml of potassium iodide solution (5.2.2.3.4.4) and 25 ml of hydrochloric acid (5.2.2.3.4.2). Swirl and allow to stand for 1 min.

Titrate with the sodium thiosulfate standard volumetric solution 0,01 mol/l (5.2.2.3.4.7) using a burette (5.2.2.3.5.1). When the solution is nearly discoloured, add 1 ml of starch solution (5.2.2.3.4.9) and continue the titration until the blue colour disappears during at least 30 s.

The presence of oxidising agents can lead to inaccurate results. The Fe^{3+} interference can be avoided by complexation with EDTA. An automatic titrator provided with a platinum electrode and an Ag/AgCl reference electrode can be used. In this case, the addition of starch solution (5.2.2.3.4.9) during the determination should be avoided.

5.2.2.3.7 Expression of results

5.2.2.3.7.1 Method of calculation

The bromine plus iodine content of the sample, $w_{(\mathrm{Br})}$, expressed as milligrams of bromine per kilogram of salt, is given by the following formula:

$$w_{(\mathrm{Br})} = 13{,}318 \times c(\mathrm{Na_2S_2O_3}) \times \frac{1000}{m} \times (V_1 - V_0) \qquad (5)$$

where

m	is the mass, in grams, of the test portion (5.2.2.3.6.1);
V_1	is the volume, in millilitres, of sodium thiosulfate (5.2.2.3.4.7) used for the titration of the test solution (5.2.2.3.6.2);
V_0	is the volume, in millilitres, of sodium thiosulfate (5.2.2.3.4.7) used for the titration of the blank test solution (5.2.2.3.6.3);
$c(\mathrm{Na_2S_2O_3})$	is the concentration in moles per litre of the sodium thiosulfate standard volumetric solution (5.2.2.3.4.7). $c(\mathrm{Na_2S_2O_3})$

5.2.2.3.7.2 Repeatability and reproducibility

Analyses, carried out on the three samples by 14 laboratories, have given the following statistical results, each laboratory having furnished results obtained by the same operator performing two analyses per sample.

Table 4 — Precision data

	Rock salt[a]	**Vacuum salt**[a]	**Sea salt**[a]
Number of laboratories	14	13	12
Results, Br mg/kg salt			
Mean	141	85	135
Standard deviation for:			
— repeatability (s_r)	4	1	1
— reproducibility (s_R)	14	4	8

[a] See A.1.

5.2.2.4 Iron

The content of iron shall be determined by inductively coupled plasma optical emission spectrometry (ICP-OES) (see Annex B).

5.2.2.5 Manganese

The content of manganese shall be determined by inductively coupled plasma optical emission spectrometry (ICP-OES) (see Annex B).

5.2.3 Chemical parameters

5.2.3.1 Cadmium, chromium, nickel, lead, antimony, selenium

The contents of chemical parameters, except for arsenic and mercury, shall be determined by inductively coupled plasma optical emission spectrometry (ICP-OES) (see Annex B).

Alternatively, the determination of contents of some chemical parameters may be carried out by atomic absorption spectrometry (AAS) and the analytical methods are given in EN 973:2009, Annex C and Annex D.

5.2.3.2 Arsenic

The content of arsenic shall be determined by silver diethyldithiocarbamate photometric method (see EN 14805:2008, B.2).

5.2.3.3 Mercury

The content of mercury shall be determined by cold vapour atomic absorption spectrometry (see EN 973:2009, B.2).

6 Labelling – Transportation – Storage

6.1 Means of delivery

Sodium chloride shall be delivered in bulk or in bags.

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 Labelling according to the EU legislation[4)]

Sodium chloride is not subject to labelling regulations at the date of the publication of this European Standard.

The regulation [2], and its amendments for the purposes of its adaptation to technical and scientific progress contains a list of substances classified by the EU. Substances not listed in this regulation should be classified on the basis of their intrinsic properties according to the criteria in the regulation by the person responsible for the marketing of the substance.

6.3 Transportation regulations and labelling

Sodium chloride is not listed under a UN Number[5)]. Sodium chloride 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 "Sodium chloride, salt for electrochlorination by membrane cells", "dry salt" or "undried salt" and trade name;
- the net mass;
- the name and the address of supplier and/or manufacturer;
- the statement "this product conforms to EN 16370".

6.5 Storage

6.5.1 Long term stability

Sodium chloride is stable during long-term storage, providing it is kept in a dry place.

6.5.2 Storage incompatibilities

Sodium chloride shall be stored in covered and dry conditions to avoid any risk of contamination.

The product shall not be allowed to come into contact with strong acids, e.g. sulfuric acid or phosphoric acid.

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4) See [2].

5) United Nations Number.

Annex A
(informative)

General information on sodium chloride for electrochlorination by membrane cells

A.1 Origin

a) Rock salt: Salt produced by mining salt deposits of different geological formations derived from ancient seas.

b) Sea salt: Salt produced by sea water evaporation via the action of sun and wind.

c) Evaporated salt: Salt produced by evaporating water from a salt solution in a special evaporator leading to the recrystallisation of the salt.

A.2 Chemical composition

A.2.1 Organic components of sodium chloride can also affect equipment efficiency. As these requirements can vary considerably, both equipment and salt suppliers should be consulted when selecting salt for this application.

The organic content should be limited, as it can form halogen-organic compounds. If necessary, it should be explained which type of salt is concerned, especially rock salt and sea salt.

A.2.2 It should also be noted that the quality of water used in preparing salt solutions for this application might also need to be taken into account when selecting the salt to be used.

A.2.3 The sodium chlorate ($NaClO_3$) content of the sodium hypochlorite produced should not exceed a mass fraction of 5,4 % of available chlorine in line with the requirements of EN 901.

NOTE Sodium chlorate is a by-product of the manufacturing process and can be formed during storage (see EN 901:2013, 6.5.1).

The treated water should not contain more than 700 µg/l chlorate.

A.3 Use

A.3.1 Function

Salt to be used to produce active chlorine (Cl_2 or NaClO) by electrolysis of the brine for disinfection of the water.

A.3.2 Form in which the product is used

It is used in the form of a saturated solution.

A.3.3 Consumption of salt for electrochlorination

The consumption is variable and depends on the applied technique, and is typically in the range of 1,7 kg to 3 kg of NaCl per kilogram of chlorine.

A.3.4 Means of application

To be documented by the equipment supplier.

A.3.5 Secondary effects

- Increase of the chloride content;
- in some cases formation of halomethanes.

A.3.6 Removal of excess product

Not applicable.

A.4 Rules for safe handling and use

No particular precaution is necessary.

A.5 Emergency procedures

A.5.1 First aid

Not applicable.

A.5.2 Spillage

The product should be collected, then the area should be rinsed with plenty of water.

A.5.3 Fire

Sodium chloride is not combustible.

Annex B
(normative)

Analytical methods — Determination of antimony, cadmium, chromium, iron, lead, manganese, nickel and selenium (inductively coupled plasma optical emission spectrometry (ICP-OES)

B.1 General

The present method specifies an inductively coupled plasma optical emission spectrometry method for the determination of 9 elements soluble in diluted acid in sodium chloride. The limit of quantification (LOQ) for each element is given in Table B.1:

Table B.1 — Limits of quantification (LOQ)

Element	**Limit of quantification (LOQ)**
	(mg/kg of salt)
Mn	0,05
Cr	0,1
Cd	0,15
Fe, Ni	0,25
As, Se	1,0
Sb	2,0
Pb	2,5

B.2 Principle

The sample is dissolved in diluted nitric acid and the acid solution is directly nebulised into an inductively coupled argon plasma sustained by a high frequency. The radiation emitted at a specific wavelength of each element is measured using background correction.

NOTE The use of reference element (internal standard) such as scandium, yttrium, cobalt ... can improve the quality of the results especially using a simultaneous spectrometer. Every mention of this optional reagent (here scandium) is stated in italic and in brackets.

B.3 Reagents

All reagents shall be of recognised analytical grade and the water used shall conform to grade 2 in accordance with EN ISO 3696.

Store all prepared solutions in polyethylene (PE) or polytetrafluorethylene (PTFE) flasks to prevent contamination.

B.3.1 **Nitric acid**, $\rho \approx$ 1,40 g/ml, mass fraction of 65 % solution.

B.3.2 Sodium chloride solution, β = 250 g/l.

Dissolve 250 g of very pure NaCl with water and transfer to a 1 000 ml volumetric flask. Make up to the mark and mix.

NOTE A very pure salt is a salt with element contents at least ten times lower than the limit of quantification for the respective element.

B.3.3 Scandium (reference element) solution, β(Sc) = 50 mg/l.

Transfer 50 ml of a scandium stock solution ($\beta_{(Sc)}$ = 1 000 mg/l and 10 ml nitric acid (B.3.1) to a 1 000 ml volumetric flask. Make up to the mark with water and mix.

B.3.4 Element, stock solution, β (each element) = 1 000 mg/l certified commercial solutions.

The stock solution shall be certified not only for the element itself but also for the other analyte elements (impurities).

B.3.5 Argon, pressure not less than 700 kPa. The argon used can be compressed or liquefied gas.

B.4 Apparatus

Ordinary laboratory apparatus and the following:

All vessels (glassware, polyethylene, polypropylene and PTFE flasks) should be washed successively with hydrochloric acid (≈ 6 mol/l) and water.

B.4.1 Spectrometer ICP-OES

Inductively coupled plasma optical emission spectrometer fitted with a nebuliser and torch for high salt concentrations and with an argon humidifier filled with water. This instrument can measure simultaneously and/or sequentially. The specifications and operating conditions used with most spectrometers are given in Table B.2:

Table B.2 — Typical operating conditions ICP-OES spectrometers[6)]

Parameter	Unit	Specification
Argon flows		
— plasma	l/min	12 to 15
— auxiliary	l/min	≈ 1,5
— nebuliser	l/min	≈ 0,7
Sample flow	ml/min	≈ 1,5
RF power	W	1 000 to 1 250
Integration time	s	
— simultaneous		≈ 10
— sequential		≈ 3

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6) Check for use with high salinity solutions.

B.5 Procedure

B.5.1 Test portion

Weigh, to the nearest 0,1 g, about 10 g of the test sample (*m*).

The highest concentration of NaCl accepted by the spectrometer should be used and the calibration and verification solutions should be corrected accordingly.

B.5.2 Test solution

Transfer the test portion (B.5.1) and water to a 100 ml volumetric flask and stir to dissolve. Add 1 ml nitric acid (B.3.1), *[5 ml of scandium solution (B.3.3)]*, make up to the mark with water and mix.

B.5.3 Calibration and verification solutions

Transfer 40 ml of sodium chloride solution (B.3.2), 1 ml of nitric acid (B.3.1), 5 ml of scandium solution (B.3.3) and the volumes of each stock solution (B.3.4) to obtain the concentrations indicated in Table B.3 into a series of four 100 ml volumetric flasks. Make up to the mark with water and mix.

Use the highest concentration of NaCl acceptable by the spectrometer and correct the calibration and verification solutions accordingly.

Table B.3 — Calibration solutions

Calibration solution No.	**Concentration of NaCl**	**Concentration of elements**
	(g/l)	**(mg/l)**
1[a]	100	0
2[b]	100	2,5
3	100	5,0
4[c]	100	5,0

[a] Blank calibration solution.

[b] Linearity verification solution.

[c] Control calibration solution prepared with different pipettes, flasks and if possible with different stock solutions.

B.5.4 Determination

B.5.4.1 Setting of the apparatus

Set all instrumental parameters of the optical emission spectrometer (B.4.1) in accordance with the operating manual of the instrument's manufacturer.

Prepare the analytical procedure including the lines shown in Table B.4 (or lines with similar sensitivity and free from interferences), with background correction and concentrations of calibration solutions 1 and 3 described in (B.5.3) *[and applying the reference technique]*.

Table B.4 — Wavelength per element

Element	Wavelength (nm)
As	189,042
	193,696
Cd	214,438
	228,802
Cr	267,716
Fe	238,204
	259,940
Mn	257,610
Ni	221,647
	231,604
Pb	168,220
	220,353
Sb	217,581
Se	196,026
Sc (reference element)	*424,683*
	361,384

Other lines with similar sensitivity and free from interferences may be used.

B.5.4.2 Spectrometric measurements

Repeat the measurements for at least five integration periods.

If necessary, rinse with the blank calibration solution (solution 1) after each solution.

Calibrate the instrument with the calibration solutions 1 and 3 (B.5.3).

Control and check the linearity of the calibration curve by measurement of the following calibration solutions (B.5.3) considered as unknown solutions:

- solution 3;
- solution 1;
- solution 1;
- solution 2;
- solution 4;
- solution 3.

If the calibration function does not appear to be a linear one, the corresponding calibration range should be decreased.

Continue the measurements in the following order:

- solution 3 (B.5.3);
- solution 1 (B.5.3);
- solution 1 (B.5.3);
- test sample solution(s) (B.5.2);
- solution 3 (B.5.3);
- solution 1 (B.5.3);
- solution 1 (B.5.3).

Five to ten test sample solutions may be analysed consecutively, provided the stability of the measurements is sufficient.

B.6 Expression of results

B.6.1 Evaluation

If necessary, correct for drift the results obtained with the test sample solution:

- for baseline drift by interpolating in time between both second measurements (the first can be cross-contaminated) of the blank calibration solution (solution 1);
- for sensitivity drift by interpolating in time between the corrected measurements for baseline drift of the control solution 3.

B.6.2 Calculation

The element content of the sample, $w_{\text{(Element)}}$ in milligrams per kilogram of sodium chloride is given by the following formula:

$$w_{\text{(Element)}} = \frac{100}{m} \times \beta_{\text{(Element)}}$$

where

m is the mass in grams of the test portion (B.5.1);

$\beta_{\text{(Element)}}$ is the corrected concentration of element, in milligrams per litre, in the test solution (B.5.2).

B.6.3 Repeatability and reproducibility

Analyses carried out on five samples, containing the 9 elements in different concentrations, by 16 laboratories, gave the following statistical results, each laboratory performed three replicates under repeatability conditions:

Table B.5 — Results of the interlaboratory study

NOTE Grey shaded values are below LOQ.

Sample	**Element content** w(mg/kg)	**Repeatability standard deviation** s_r (mg/kg)	**Reproducibility standard deviation** s_R (mg/kg)	**Repeatability limit** r (mg/kg)	**Reproducibility limit** R (mg/kg)	**Upper limit** b = 0,5×s_r	**Rounding interval "*a*"**
Arsenic							
1	0,08	0,124	0,216	0,35	0,61	0,062	
2	0,84	0,106	0,181	0,30	0,51	0,053	
3	2,04	0,079	0,166	0,22	0,47	0,040	0,01
4	20,76	0,312	1,120	0,88	3,17	0,156	0,1
5	8,16	0,210	0,577	0,59	1,63	0,105	0,1
Cadmium							
1	0,02	0,009	0,021	0,02	0,06	0,005	
2	0,40	0,018	0,023	0,05	0,05	0,009	0,005
3	8,00	0,107	0,196	0,30	0,55	0,054	0,05
4	2,08	0,030	0,106	0,08	0,30	0,015	0,01
5	15,48	0,318	0,822	0,90	2,33	0,159	0,1
Chromium							
1	0,00	0,010	0,028	0,03	0,08	0,005	
2	0,39	0,014	0,033	0,04	0,09	0,007	0,005
3	7,96	0,080	0,258	0,23	0,73	0,040	0,01
4	2,00	0,031	0,074	0,09	0,21	0,016	0,01
5	15,44	0,378	0,971	1,07	2,75	0,189	0,1
Iron							
1	0,01	0,024	0,034	0,07	0,10	0,012	
2	0,44	0,024	0,030	0,07	0,09	0,012	0,01
3	8,20	0,113	0,282	0,32	0,80	0,057	0,05
4	2,08	0,026	0,062	0,07	0,18	0,013	0,01
5	15,88	0,271	0,619	0,77	1,75	0,136	0,1
Manganese							
1	0,00	0,001	0,004	0,004	0,01	0,001	
2	0,08	0,002	0,003	0,004	0,01	0,001	0,001
3	0,40	0,007	0,027	0,02	0,04	0,004	0,001
4	2,08	0,022	0,068	0,06	0,19	0,011	0,01
5	7,80	0,170	0,405	0,48	1,15	0,085	0,05

Table B. 5 *(continued)*

Sample	**Element content** w(mg/kg)	**Repeatability standard deviation** s_r (mg/kg)	**Reproducibility standard deviation** s_R (mg/kg)	**Repeatability limit** r (mg/kg)	**Reproducibility limit** R (mg/kg)	**Upper limit** b = 0,5×s_r	**Rounding interval "*a*"**
Nickel							
1	0,04	0,026	0,132	0,08	0,38	0,013	
2	0,37	0,022	0,108	0,06	0,30	0,011	0,01
3	8,04	0,058	0,251	0,16	0,71	0,029	0,01
4	2,00	0,039	0,146	0,11	0,41	0,020	0,01
5	15,64	0,296	0,628	0,84	1,78	0,148	0,1
Lead							
1	0,07	0,315	0,315	0,89	0,89	0,158	
2	0,80	0,162	0,240	0,46	0,68	0,081	
3	1,92	0,261	0,289	0,74	0,82	0,131	0,1
4	20,36	0,342	0,596	0,97	1,69	0,171	0,1
5	7,76	0,260	0,513	0,74	1,45	0,130	0,1
Antimony							
1	0,01	0,100	0,100	0,28	0,28	0,050	
2	0,76	0,169	0,212	0,48	0,60	0,085	
3	2,20	0,233	0,840	0,66	2,38	0,117	0,1
4	18,96	0,529	1,503	1,50	4,25	0,265	0,1
5	7,92	0,338	1,327	0,96	3,76	0,169	0,1
Selenium							
1	0,02	0,070	0,082	0,20	0,23	0,035	
2	0,76	0,094	0,106	0,26	0,30	0,047	
3	1,96	0,107	0,113	0,30	0,32	0,054	0,05
4	20,28	0,315	0,710	0,89	2,01	0,158	0,1
5	7,88	0,266	0,370	0,75	1,05	0,133	0,1

The rules for rounding off the observations:

- calculation of the upper limit $b = 0,5 \times s_r$;
- choosing the rounding off interval. Select from the sequence 10; 1; 0,5; 0,1; 0,05; 0,01; 0,005; etc. the first value that is less than or equal to *b*. This value is the rounding-off-interval “*a*”. Applying these rules gives the rounding of recommendations of Table B.6.

B.6.4 Limit of quantification (LOQ)

The limit of quantification (LOQ) - the lowest concentration of the element which can be determined with a coefficient of variation of 10 % - is calculated based on the repeatability standard deviation s_r of each element near the estimated LOQ.

Table B.6 — LOQ and rounding off recommendations for each element

Concentration range	Report
Concentrations below LOQ are to be reported as "< LOQ"	
ARSENIC	LOQ = 1,0 mg/kg
LOQ – 2 mg/kg	to the nearest 0,01 mg/kg
> 2 mg/kg	to the nearest 0,1 mg/kg
CADMIUM	LOQ = 0,15 mg/kg
LOQ – 2 mg/kg	to the nearest 0,01 mg/kg
2 mg/kg – 10 mg/kg	to the nearest 0,05 mg/kg
> 10 mg/kg	to the nearest 0,1 mg/kg
CHROMIUM	LOQ = 0,1 mg/kg
LOQ – 10 mg/kg	to the nearest 0,01 mg/kg
> 10 mg/kg	to the nearest 0,1 mg/kg
IRON	LOQ = 0,25 mg/kg
LOQ – 2 mg/kg	to the nearest 0,01 mg/kg
2 mg/kg – 10 mg/kg	to the nearest 0,05 mg/kg
> 10 mg/kg	to the nearest 0,1 mg/kg
MANGANESE	LOQ = 0,05 mg/kg
LOQ – 2 mg/kg	to the nearest 0,01 mg/kg
2 mg/kg – 10 mg/kg	to the nearest 0,05 mg/kg
NICKEL	LOQ = 0,25 mg/kg
LOQ – 10 mg/kg	to the nearest 0,01 mg/kg
> 10 mg/kg	to the nearest 0,1 mg/kg
LEAD	LOQ = 2,5 mg/kg
> LOQ	to the nearest 0,1 mg/kg
ANTIMONY	LOQ = 2,0 mg/kg
> LOQ	to the nearest 0,1 mg/kg
SELENIUM	LOQ = 1,0 mg/kg
LOQ – 2 mg/kg	to the nearest 0,05 mg/kg
> 2 mg/kg	to the nearest 0,1 mg/kg

Bibliography

[1] 98/83/EC, Council Directive of 3 November 1998 on the quality of water intended for human consumption

[2] Regulation (EC) No 1272/2008 of the European Parliament and of the Council of 16 December 2008 on classification, labelling and packaging of substances and mixtures, amending and repealing Directives 67/548/EEC and 1999/45/EC, and amending Regulation (EC) No 1907/2006 (REACH)

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