BRITISH STANDARD

BS EN 14805:2008

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

ICS 13.060.25; 71.100.80

British Standards

National foreword

This British Standard is the UK implementation of EN 14805:2008.

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

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Chemicals used for treatment of water intended for human consumption - Sodium chloride for on site electrochlorination using non-membrane technology

Produits chimiques utilisés pour le traitement de l'eau destinée à la consommation humaine - Chlorure de sodium pour la génération électrochimique de chlore utilisant des technologies non membranaires

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

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Contents

Foreword

This document (EN 14805:2008) 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of an identical text or by endorsement, at the latest by December 2008, and conflicting national standards shall be withdrawn at the latest by December 2008.

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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 non-membrane technology. 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 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 973:2002, *Chemicals used for treatment of water intended for human consumption — Sodium chloride for regeneration of ion exchangers*

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

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 degrees 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 Number[1)]

7647-14-5

3.1.7 EINECS Reference[2)]

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.

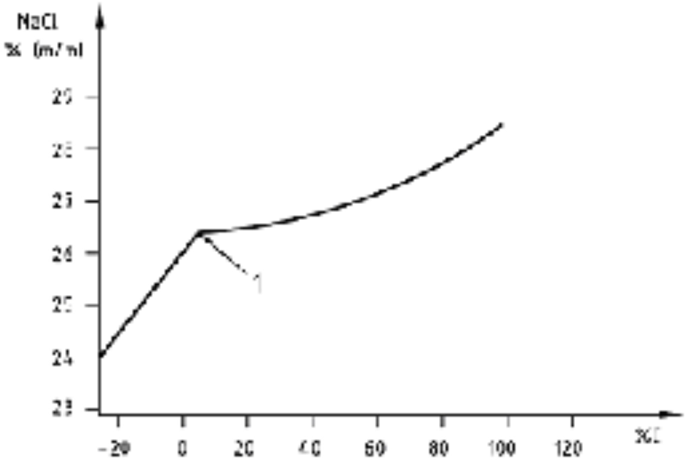
The bulk density depends on the particle size distribution.

[1)] Chemical Abstracts Service Registry Number.

[2)] European Inventory of Existing Commercial chemical Substances Reference.

3.3.3 Solubility (in water)

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



Temperature °C	NaCl solution Mass fraction in %
- 10	25,0
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

Key

1 transition point
NaCl → NaCl . $2H_2O$

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.

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3) 100 kPa = 1 bar

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.

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 and non-volatile, and aqueous solutions have good electrical conductivity.

Sodium chloride is decomposed by a number of acids. It reacts with sulfuric acid, phosphoric acid and strong oxidizing 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 non-membrane technology. As the treatment chemical produced by this process is sodium hypochlorite, limits have been calculated from the requirements of EN 901 (sodium hypochlorite) for those impurities commonly present in the product and the chemical parameters. Consideration has also been given to the requirements of EN 973 (sodium chloride) for regeneration of ion exchangers. 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 1 Users of this product should check the national regulations in order to clarify whether it is of appropriate purity for the 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 and not stated in this product standard.

NOTE 2 Using the sodium hypochlorite standard to define limits produces discrepancies with EN 973 regarding sodium chloride for the regeneration of ion exchange water softeners. This should not be taken to imply any real difference between the chemical purity between salt types meeting either specification.

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

4.2.1 Sodium chloride content

The content of sodium chloride in the dry product shall not be less than:

Type 1: mass fraction of 99,9 % of dry sodium chloride, NaCl;

Type 2: mass fraction of 98,5 % of dry sodium chloride, NaCl.

4.2.2 Anticaking agent

Subject to any local legislation in member states, an anticaking agent, sodium or potassium hexacyanoferrate, is allowed up to a maximum level in the final product of 15 mg/kg, expressed as the anhydrous hexacyanoferrate ion $[Fe(CN_6)]^{-4}$; for the determination, see B.3 of EN 973:2002. If gaseous free chlorine is produced as a stage in the electrochlorination process, the level of hexacyanoferrate ion present should be less than 3 mg/kg.

4.3 Impurities and main by-products

The product shall conform to the requirements specified in Table 1 and Table 2.

Table 1 — Impurities

Impurity	Limit in mass fraction in % of NaCl content		
Water-insoluble matter		**Type 1**	**Type 2**
	max.	0,05	0,35
Bromide	max.	0,025	0,05

Table 2 — Moisture content

Impurity	Limit in mass fraction in % of NaCl content		
Moisture content		Dry salt	Un-dried salt
	Max.	0,1	5

4.4 Chemical parameters

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

Table 3 — Chemical parameters

Parameter		Limits in mg/kg of commercial product	
		Type 1	Type 2
Arsenic (As)	max.	0,3	1,5
Cadmium (Cd)	max.	0,75	1,5
Chromium (Cr)	max.	0,75	1,5
Mercury (Hg)	max.	1,05	1,5
Nickel (Ni)	max.	0,75	3
Lead (Pb)	max.	3,5	4,5
Antimony (Sb)	max.	6	7,5
Selenium (Se)	max.	6	7,5

NOTE Other chemical parameters and indicator parameters are not relevant in sodium chloride, but there might be other parameter limits related to equipment performance, and some of these are listed in Annex A.

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 also 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 recognized 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, $\beta(NaClO) \approx 19$ g/l

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

NOTE 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 2 H_2O), 50 g of disodium hydrogen orthophosphate dodecahydrate (Na_2HPO_4 12 H_2O), 50 g of tetrasodium pyrophosphate decahydrate ($Na_4P_2O_7$ 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 standardize 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 95 % (volume fraction) 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.

NOTE 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. Neutralize 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.

NOTE The presence of oxidizing 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 a 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, $\omega_{(Br)}$, expressed as milligrams of bromine per kilogram of salt, is given by the following equation:

$$\omega_{(Br)} = 13{,}318 \times c_{(Na_2S_2O_3)} \times \frac{1\,000}{m} \times (V_1 - V_0) \qquad (5)$$

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);
$c_{(Na_2S_2O_3)}$	is the molar concentration of the sodium thiosulfate standard volumetric solution (5.2.2.3.4.7).

5.2.2.3.7.2 Repeatability and reproducibility

Analyses, carried out on 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.

5.2.3 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 B.1).

NOTE Alternatively the determination of contents of some chemical parameters can be carried out by atomic absorption spectrometry (AAS) and the analytical methods are given in Annex C and Annex D of EN 973:2002.

5.2.3.2 Mercury

The content of mercury shall be determined by cold vapour atomic absorption spectrometry according to B.2 of EN 973:2002.

5.2.3.3 Arsenic

The content of arsenic shall be determined using the silver diethyldithiocarbamate photometric method given in 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 Risk and safety labelling in accordance with EU directives

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

NOTE Annex I of the Directive 67/548/EEC [2] 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 that are not mentioned 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

Sodium chloride is not listed under a UN Number4). 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 for non-membrane equipment", “dry salt" or "undried salt", trade name and type;
- the net mass;
- the name and the address of supplier and/or manufacturer;
- and the statement "this product conforms to EN 14805".

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 so as 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) United Nations Number.

Annex A
(informative)

General information on sodium chloride for electrochlorination

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 recrystallization of the salt.

A.2 Chemical composition

A.2.1 The efficiency of all electrochlorination equipment can be impaired particularly by iron and manganese. Levels of both of these elements should be less than 10 mg/kg.

A.2.2 Other natural components of sodium chloride such as calcium, magnesium, sulfate and organic content might affect equipment efficiency. As these requirements can vary considerably, it is important that both equipment and salt suppliers are consulted when selecting salt for this application.

A.2.3 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.2.4 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 [3].

NOTE 1 Sodium chlorate is a by-product of the electrochlorination process and can be formed during storage (see EN 901:2007, 6.5.1).

NOTE 2 The treated water should not contain more than 700 µg/l chlorate.

A.3 Use

A.3.1 Function

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

A.3.2 Form in which the product is used

It is used in the form of a saturated solution. Some technologies require high purity brine. This will be achieved by including an ion exchange resin step following the brine production, before entering the electrolysis cell. The most important parameters that should be monitored are Ca, Mg, SO_4^{2-}, Br^-, total Fe and Mn. The equipment supplier should be consulted about the limits.

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 produced.

A.3.4 Means of application

The supplier of the electrochlorination equipment should provide documentation.

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

B.1 Determination of antimony, cadmium, chromium, iron, lead, manganese, nickel and selenium (inductively coupled plasma optical emission spectrometry (ICP/OES))

B.1.1 General

The present method specifies an inductively coupled plasma optical emission spectrometry method for the determination of eight 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
Se	1,0
Sb	2,0
Pb	2,5

B.1.2 Principle

The sample is dissolved in diluted nitric acid and the acid solution is directly nebulized 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.1.3 Reagents

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

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

B.1.3.1 Nitric acid, $\rho \approx$ 1,40 g/ml, mass fraction of 65 % solution.

B.1.3.2 Sodium chloride solution, β(NaCl) = 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.1.3.3 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.1.3.1) to a 1 000 ml volumetric flask. Make up to the mark with water and mix.]

B.1.3.4 Element, stock solution, β (each element) = 1 000 mg/l certified commercial solutions

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

B.1.3.5 Argon, pressure not less than 700 kPa. The argon used can be compressed or liquefied gas.

B.1.4 Apparatus

B.1.4.1 General

Ordinary laboratory apparatus.

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

B.1.4.2 Spectrometer ICP/OES

This is an inductively coupled plasma optical emission spectrometer fitted with a nebulizer 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 for most ICP/OES spectrometers [a]

Parameter	Unit	Specification
Argon flows		
— plasma	l/min	12 to 15
— auxiliary	l/min	≈ 1,5
— nebulizer	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

[a] Check for use with high salinity solutions.

B.1.5 Procedure

B.1.5.1 Test portion

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

NOTE The highest acceptable concentration of NaCl for the spectrometer should be used and the calibration and verification solutions should be corrected accordingly.

B.1.5.2 Test solution

Transfer the test portion (B.1.5.1) and water to a 100 ml volumetric flask and stir to dissolve. Add 1 ml nitric acid (B.1.3.1), *[5 ml of scandium solution (B.1.3.3)]*, make up to the mark with water and mix.

B.1.5.3 Calibration and verification solutions

Transfer 40 ml of sodium chloride solution (B.1.3.2), 1 ml of nitric acid (B.1.3.1), *[5 ml of scandium solution (B.1.3.3)]* and the volumes of each stock solution (B.1.3.4) 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 for the spectrometer and correct the calibration and verification solutions accordingly.

Table B.3 — Calibration solutions

Calibration solution No.	Concentration of NaCl g/l	Concentration of elements mg/l
1 [a]	0	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.1.5.4 Determination

B.1.5.4.1 Setting of the apparatus

Set all instrumental parameters of the optical emission spectrometer (B.1.4.1) in accordance with the operating manual of the instrument's manufacturer.

Prepare the analytical procedure including the wavelengths shown in Table B.4 (or wavelengths with similar sensitivity and free from interferences), with background correction and concentrations of calibration solutions 1 and 3 described in (B.1.5.3) *[and applying the technical reference]*.

Table B.4 — Wavelength per element

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

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

B.1.5.4.2 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.1.5.3).

Control and check the linearity of the calibration curve by measurement of the following calibration solutions (B.1.5.3) considered as unknown solutions:

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

NOTE 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.1.5.3);
- solution 1 (B.1.5.3);
- solution 1 (B.1.5.3);
- test sample solution(s) (B.1.5.2);
- solution 3 (B.1.5.3);
- solution 1 (B.1.5.3);
- solution 1 (B.1.5.3).

NOTE Five to ten test sample solutions can be analyzed consecutively, provided the stability of the measurements is sufficient.

B.1.6 Expression of results

B.1.6.1 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.1.6.2 Calculation

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

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

where

m is the mass in grams of the test portion (B.1.5.1);

β is the corrected concentration of element, in milligrams per litre, in the test solution (B.1.5.2).

B.1.6.3 Repeatability and reproducibility

Analyses carried out on five samples, containing the eight 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	Repeatability standard deviation	Reproducibility standard deviation	Repeatability limit	Reproducibility limit	Upper limit	Rounding interval "a"
	ω (mg/kg)	s_r (mg/kg)	s_R (mg/kg)	r (mg/kg)	R (mg/kg)	$B = 0,5 \times s_r$	
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 (*concluded*)

Sample	Element content	Repeatability standard deviation	Reproducibility standard deviation	Repeatability limit	Reproducibility limit	Upper limit	Rounding interval "a"
	ω (mg/kg)	s_r (mg/kg)	s_R (mg/kg)	r (mg/kg)	R (mg/kg)	$B = 0,5 \times s_r$	
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-off-recommendations of Table B.6.

B.1.6.4 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"	
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

B.2 Determination of Arsenic (Silver Diethyldithiocarbamate Photometric Method)

B.2.1 General

The present method describes a silver diethyldithiocarbamate photometric method for the determination of arsenic in sodium chloride. The method is applicable to products of arsenic content (As) equal to or greater than 0,1 mg per kilogram of salt.

B.2.2 Principle

The sample is dissolved in dilute hydrochloric acid, followed by treatment with zinc to form arsine (AsH_3). The arsine is absorbed in a solution of silver diethyldithiocarbamate in pyridine,and the resulting purplish-red colour of dispersed silver is measured photometrically at a wavelength around 540 nm.

NOTE Whilst nitrates, nitrites, antimony, mercury, chromium, cobalt, copper, molybdenum, nickel, platinum, silver and others can interfere in this determination, there is no interfering effect at concentrations usually present in salt.

B.2.3 Reactions

Zinc reacts with arsenic in acid media to yield arsine as follows:

$$Zn_3As_2 + 6\ H^+ \rightarrow 2\ AsH_3 + 3\ Zn^{2+}$$

Colloidal silver is formed according to the following equation:

$$AsH_3\ + 6\ Ag(DDTC) \longrightarrow 6\ Ag\ +\ 3\ H(DDTC)\ +\ As(DDTC)_3$$

B.2.4 Reagents

All reagents shall be of recognized analytical grade and the water used shall conform to grade 2 in accordance with EN ISO 3696. All the reagents, and the zinc in particular, shall be free from arsenic or have a very low arsenic content.

B.2.4.1 Pyridine, $\rho \approx 0{,}980$ g/ml.

WARNING — Because of the hazardous properties of pyridine, it is recommended to handle with care and in a well-ventilated fume cupboard.

B.2.4.2 Zinc pellets, 3 mm to 8 mm diameter or any other form of zinc.

NOTE When zinc is used in any other form than pellets, it needs to be ensured that the rate and time of evolution of hydrogen are sufficient to reduce and expel all arsenic present as arsine.

B.2.4.3 Hydrochloric acid, $\rho \approx 1{,}19$ g/ml, 37% (m/m).

B.2.4.4 Silver diethyldithiocarbamate, 5 g/l solution in pyridine

Dissolve 1 g of silver diethyldithiocarbamate [Ag(DDTC)] in pyridine (B.2.4.1) and dilute to 200 ml with pyridine.

This solution remains stable for about 2 weeks, if stored in a tightly sealed dark glass bottle and protected from light.

B.2.4.5 Potassium iodide solution, $\beta_{(KI)} \approx 100$ g/l

Store this solution in a dark bottle.

B.2.4.6 Tin (II) chloride solution, $\beta_{(SnCl_2.2H_2O)} \approx 400$ g/l

Dissolve 40 g of tin (II) chloride dihydrate in a mixture of 25 ml of water and 75 ml of hydrochloric acid (B.2.4.3).

B.2.4.7 Arsenic, stock solution I, $\beta_{(As)}$ = 1 000 mg/l, commercial standard solution or to be prepared as follows

Dissolve 1,320 g of arsenic trioxide, (As_2O_3), in about 20 ml of sodium hydroxide solution (50 g/l solution). Make up to 1 000 ml in a one-mark volumetric flask and mix.

B.2.4.8 Arsenic, stock solution II, $\beta_{(As)}$ = 2,50 mg/l

Transfer 2,50 ml of arsenic stock solution I (B.2.4.7.) into a 1000 ml one-mark volumetric flask, make up to the mark and mix.

Prepare this solution just before use.

B.2.4.9 Absorbent cotton wool, saturated with lead acetate

Dissolve 20 g of lead acetate trihydrate, $Pb(C_2H_3O_2)_2.3H_2O$, in 100 ml of water. Saturate the absorbent cotton wool with this solution, remove the excess solution by allowing it to drain and dry the cotton wool under vacuum at room temperature. Store it in an airtight container.

B.2.5 Apparatus

Usual laboratory equipment and:

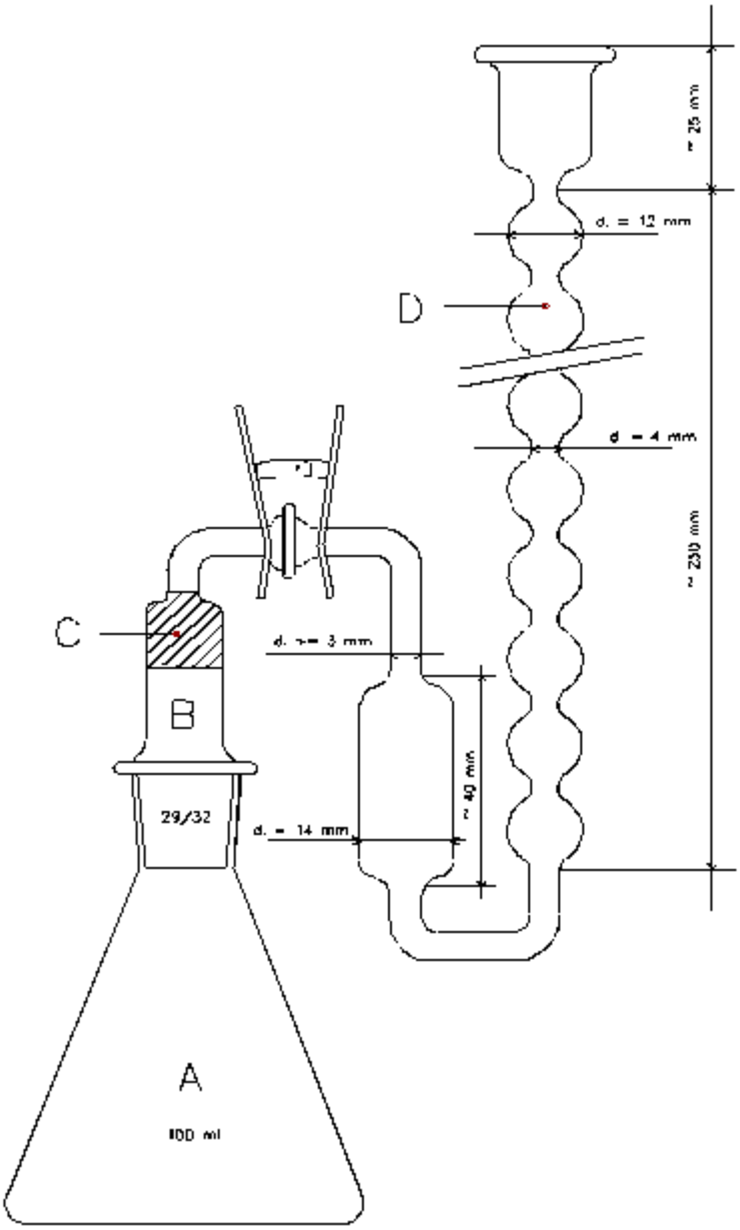
B.2.5.1 Glass apparatus, with ground glass joints, for the liberation and absorption of arsine

All the glass containers used for the determination of arsenic shall be washed with hot concentrated sulfuric acid, taking the necessary precautions, then rinsed thoroughly with water and dried in a drying cabinet. A suitable apparatus is shown in Figure B.1 and includes:

B.2.5.1.1 Conical flask, 100 ml capacity

B.2.5.1.2 Connecting tube

B.2.5.1.3 Absorption vessel with 15 bulbs



Key

A 100 ml conical flask

B connecting tube

C absorbent cotton wool saturated with lead acetate

D absorption vessel with 15 bulbs (total volume of bulbs ≈ 14ml)

Figure B.1 — Determination of arsenic - Silver Diethyldithiocarbamate Photometric Method - Typical apparatus

B.2.5.2 Spectrophotometer or

B.2.5.3 Photocolorimeter fitted with a filter ensuring maximum transmission between 520 nm and 560 nm

Specify the type of apparatus used (spectrophotometer or photocolorimeter), the path length and the wavelength (or type of filter).

B.2.6 Sampling

See 5.1.

Ensure that no trace of arsenic is introduced during the sampling operations (e.g. by apparatus) or during the analysis.

B.2.7 Procedure

B.2.7.1 Test portion

Weigh, to the nearest 0,1 g, about 10 g of the test sample containing a maximum of 20 µg of Arsenic (As)

NOTE In case of an arsenic content greater than 2 mg/kg of salt, the size of test portion (B.2.7.1) shall be reduced. Sodium chloride as a matrix does not influence the generation, evolution and determination of arsine.

B.2.7.2 Test solution

Transfer the test portion (B.2.7.1), 50 ml of water and 10 ml of hydrochloric acid (B.2.4.3.) into a 100 ml conical flask (B.2.5.1.1).

B.2.7.3 Blank solution

Transfer 50 ml of water and 10 ml of hydrochloric acid (B.2.4.3) into a 100 ml conical flask (B.2.5.1.1)

B.2.7.4 Calibration

B.2.7.4.1 Calibration solutions

These preparations are used for photometric measurement in cuvettes of 1 cm optical path length.

Transfer the volumes of arsenic stock solution II (B.2.4.8) indicated in Table B.7 into a series of six 100 ml conical flasks (B.2.5.1.1). Add 10 ml of hydrochloric acid (B.2.4.3) and the quantity of water necessary to make up to about 60 ml.

Table B.7 — Calibration solutions

Calibration solution No.	Arsenic stock solution II ml	Corresponding mass of arsenic µg
1 [a]	0	0
2	1,0	2,5
3	2,0	5,0
4	4,0	10,0
5	6,0	15,0
6	8,0	20,0

[a] Zero calibration solution.

B.2.7.4.2 Reduction and colour development

Proceed with each glass apparatus (B.2.5.1) as follows.

Place a little of the absorbent cotton wool (B.2.4.9) in the connecting tube in order to absorb any hydrogen sulfide which might be released with the arsine (see Figure B.1).

Transfer 5,0 ml of silver diethyldithiocarbamate solution (B.2.4.4) into the absorption vessel and attach the connecting tube to the absorption vessel by means of a safety clip.

Transfer 3 ml of potassium iodide solution (B.2.4.5) and 2 ml of tin (II) chloride solution (B.2.4.6) into the conical flask, swirl and allow to stand for 15 min.

Add 5 g of zinc (B.2.4.2), if necessary with the aid of a powder funnel, and rapidly assemble the apparatus as indicated in Figure B.1.

Allow approximately 45 min for the reaction to take place.

Detach the absorption vessel and swirl in order to disperse the formed red deposit and to mix.

The colour of the solution is stable for about 2 h in the absence of light; nevertheless, it is better to carry out the photometric measurements immediately.

B.2.7.4.3 Photometric measurements

Adjust the apparatus to zero absorbance with pyridine (B.2.4.1).

Carry out the photometric measurements using a spectrophotometer (B.2.5.2) set up at the maximum of absorption (wavelength around 540 nm) or a photocolorimeter (B.2.5.3) fitted with the appropriate filter.

B.2.7.4.4 Calibration curve

Subtract the absorbance of the zero calibration solution from that of each calibration solution and plot a graph with the quantities of arsenic (As), in micrograms, added to the calibration solutions (B.2.7.4.1) as the abscissa and the corresponding corrected absorbances as the ordinate.

B.2.7.5 Determination

B.2.7.5.1 Reduction and colour development

Proceed with the test solution (B.2.7.2) and the blank solution (B.2.7.3) according to the procedure for calibration solutions described in B.2.7.4.2.

B.2.7.5.2 Photometric measurements

Carry out the photometric measurements of the two solutions (B.2.7.5.1) according to the instructions given in B.2.7.4.3.

B.2.8 Expression of results

B.2.8.1 Calculation

The arsenic content of the sample, $\omega_{(\mathrm{As})}$, is given by the following equation:

$$\omega_{(\mathrm{As})} = \frac{m_1 - m_0}{m}$$

where

$\omega_{(\mathrm{As})}$ is the arsenic content, in milligrams per kilogram of salt;

m is the mass, in grams, of the test portion (B.2.7.1);

m_1 is the mass of arsenic, in micrograms, analysed in the test solution (B.2.7.2);

m_0 is the mass of arsenic, in micrograms, analysed in the blank solution (B.2.7.3).

B.2.8.2 Repeatability and reproducibility

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

Table B.8 — Results of the interlaboratory study

	Rock salt [a]	Vacuum salt [a]	Sea salt [a]
Number of laboratories after elimination of outliers	17	17	18
Results, mg As/kg salt			
Mean [b]	0,005	0,007	0,024
Standard deviation for:			
- repeatability (s_r)	0,021 5	0,023 6	0,021 0
- reproducibility (s_R)	0,034 0	0,040 1	0,050 6

[a] See A.1.

[b] Mean values are below Limit of Quantitation.

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 27 June 1967 on the approximation of laws, regulations and administrative provisions relating to the classification, packaging and labelling of dangerous substances and its amendments and adaptations*

[3] EN 901:2007, *Chemicals used for treatment of water intended for human consumption — Sodium hypochlorite*

[4] ISO 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method*

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