

Shtojca 20



FORMULARI I ANKESËS PRANË AUTORITETIT/ENTIT KONTRAKTOR DHE
KOMISIONIT TË PROKURIMIT PUBLIK

Ankesë drejtuar: Autoriteti/Entit Kontraktor dhe Komisionit të Prokurimit Publik

Seksioni I. Identifikimi i ankimuesit

Ankimuesi mund të jetë një ofertues ose ofertues i mundshëm (p.sh. individ, operator ekonomik, shoqatë, bashkim operatorësh ekonomike)

METANI

Emri i plotë i ankimuesit (ju lutem shtypeni)

METANI

Nuis/Nipt

L11312046V

Adresa

**Rruga "Xhorxhi
Martini", godina 33**

SHQIPERI

Shteti

10001

Kodi Postar / Posta

Qyteti

TIRANE

N/A

Numri i telefonit (përfshirë kodin e zonës)

Numri i faksit (përfshirë kodin e zonës)

0697007522

E-mail

info@metani-ddd.co

Emri dhe titulli i zyrtarit të autorizuar për lëshimin e ankesës (ju lutemi shkruani)

Albert METANI

Nënshkrimi i zyrtarit të autorizuar

00355697007522

Data (viti/muaji/dita)

2023-02-15

Numri i telefonit (përfshirë kodin e zonës)

Numri i faksit (përfshirë kodin e zonës)

Seksioni II. Informacion mbi procedurën

1. Numri i referencës së procedurës/Lotit

Blerje hipoklorit kalciumi dhe natriumi, REF-58347-02-08-2023 “

2. Lloji i Procedurës

Plotësoni llojin e procedurës së përdorur për prokurimin në fjalë.

Procedurë e hapur <input type="checkbox"/>	Procedurë e hapur e thjeshtuar X
Procedurë e kufizuar <input type="checkbox"/>	Procedurë konkurruese me negociim <input type="checkbox"/>
Partneritet për inovacion <input type="checkbox"/>	Dialog konkurrues <input type="checkbox"/>
Procedurë me negociim me shpallje paraprake të njoftimit <input type="checkbox"/>	Procedurë me negociim pa shpallje paraprake të njoftimit të kontratës <input type="checkbox"/>
Shërbim Konsulence <input type="checkbox"/>	
Kontratë e lidhur pa zhvilluar ndonjë nga procedurat e prokurimit të parashikuara në LPP <input type="checkbox"/>	

3. Autoriteti /Enti Kontraktor

Emri i autoritetit/entit kontraktor që administron procesin e prokurimit.

Shoqëria Rajonale Ujësjellës Kanalizime Berat SH.A

4. Vlera e përlogaritur e prokurimit

(Vlera e përlogaritur e kontratës/marrëveshjes kuadër) (shuma në shifra dhe fjalë)

8,725,667 (Tetëmiljon e shtatëqinde e njëzet e pesëmije e gjashtëqinde e gjashtëdhjetë e shtatë) leke pa tvsh

5. Objekti i kontratës/Marrëveshjes Kuadër



(Përshkrimi i shkurtër i punëve / mallrave / shërbimeve objekt kontrate/marreveshje kuader).

Blerje hipoklorit kalciumi dhe natriumi

6. Afati i fundit për paraqitjen e ofertës

(Data (viti/muaji/dita)

2023-02-20

7. Data e publikimit të Njoftimit të Fituesit

(Data (viti/muaji/dita) nëse është e zbatueshme)

8. **Data e nënshkrimit të kontratës**

(Data (viti/muaji/dita) në rastet e kërkesave për pavlefshmërinë e kontratës)

Seksioni III. Përshkrimi i ankesës

1. Baza ligjore (Shkelje ligjore, bazuar në vendime, akte, dokumente, etj.)

Ligji 162/2020 date 23.12.2020 nenet: 2,3,4,18,36,73,77,80,98,110,112,113,.

Vendim i Keshillit të Ministrave Nr. 285, date 19.05.2021 ndryshuar me Vendimin 710, date 24.11.2022, VKM nr. 933, date 25.11.2020, kreu III, pika 1, dh), kreu IV, 1, a) dhe ç).

Vendim i Keshillit të Ministrave Nr. 379, datë 25.5.2016,

Ligj nr. 10 448, datë 14.7.2011” Për lejet e mjedisit”

Ligj nr. 105/2014 “Për barnat dhe shërbimin farmaceutik”

2. Objekti i ankesës

- Modifikim i dokumentave të tenderit

- Kundërshtim i vendimit të Komisionit të Vlerësimit të Ofertave lidhur me skualifikimin e ofertës tuaj.

(Citoni këtu arsyet e skualifikimit)

- Kundërshtim i vendimit të Komisionit të Vlerësimit të Ofertave lidhur me kualifikimin e ofertës



të një/disa operatori/ve ekonomik pjesëmarrës në procedurën e prokurimit.

(Citoni operatorin/ët ekonomik për të cilin keni pretendime)

- Pavlefshmëri kontrate

(Citoni kontratën për të cilën kërkon pavlefshmërinë)

- Tjetër

(Anulim i procedurës së prokurimit bazuar në nenin 98, pika 1, c dhe d,)

3. Rrethanat dhe faktet

Përshkruani rrethanat e faktit.

Shoqëria METANI është njoftuar nepermjet sistemit elektronik të prokurimit publik (SPE) mbi publikimin e procedurës me Autoritet Kontraktor (AK) **Shoqëria Rajonale Ujësjellës Kanalizime Berat S.H.A me objekt:** Blerje hipoklorit kalciumi dhe natriumi dhe fond limit: 8,725,667 (Tetëmiljon e shtatëqinde e njëzet e pesë mijë e gjashtëqinde e gjashtëdhjetë e shtatë) leke pa tvsh , numer reference **REF-58347-02-08-2023.**

Ne shtojcën 5, Specifikimet Teknike, dhe shtojca 7, pika 2.3.3 ndryshuar me shtojcën mbi Ndryshimin e Kriterëve të tenderit, dokumentave standarte të tenderit (DST) publikuar në SPE, **OE Metani** është njohur me kërkesat e specifikimeve teknike si mëposhte:

Specifikimet teknike Pika 1,.

Specifikimi i Materialeve:

Informacioni në lidhje me specifikat kryesore për Hipokloritin e Natriumit

Hipokloriti i Natriumit duhet të plotësojë kushtet e mëposhtme

PH < 8.0

Koha e nevojshme e kontaktit > 30 minuta

Turbullira < 0.5 NTU

Specifikimet Teknike

Hipokloriti I natriumit (NaOCl), “ Cilësia e Ujit të Pijshëm “ nga VKM nr 379 datë 25.05.2016

Nr.	Parametrat	Njësia e matjes	Specifikat Kimike
1	Gjëndja	-	Lëng i kthjellët
2	Ngjyra	-	E Verdhë e çelët

3	Densiteti në 20°C	gr/ml	1.13 - 1.28
4	Titulli i Cl ₂	%	12 - 15
5	Substanca Toksike	mg/kg	Limitet në mg/kg të klorit të lirë
6	Kadmium (Cd)	mg/kg	max 2.5
7	Krom (Cr)	mg/kg	max 2.5
8	Nikel (Ni)	mg/kg	max 2.5
9	Plumb (Pb)	mg/kg	max 15

Shtojca 7, Pika 2.3.2,

Operatori Ekonomik duhet të paraqesë Certifikaten e prodhuesit që verteton konformitetin e produktit me standardin SSH EN 901: 2013 ‘Kimikate që përdoren për trajtimin e ujërave të destinuara për konsum njerëzor –**Calcium hypochlorite (Ca(OCl)₂) dhe Sodium hypochlorite**’, shoqeruar me:

- Lejen e importit të lëshuar nga Komisioni i Liçensave pranë Ministrisë së Shëndetësisë për llojin e kimikatit, në rast se operatori ekonomik është importues.
- Leje mjedisore e tipit A të lëshuar nga Ministria e Mjedisit për kimikatin e kërkuar nga AK.

Shtojca 7, pika 2.3.3

2.3.3 Operatori ekonomik duhet të paraqesë në sistemin elektronik certifikatën e analizës nga Instituti i Shëndetit Publik ose nga laboratore të certifikuar, për analizë toksikologjike për produktet që prokurohet (certificate analize brenda 3 muajve të fundit).

Hipoklorit Kalciumi (në mg/kg të klorit aktiv 60-70 %) Cilësia e Ujit të Pijshëm “ nga VKM nr 379 datë 25.05.2016

Nr.	Parametrat	Njësia e matjes	Specifikat Kimike
1	Gjëndja	-	Granular
2	Ngjyra	-	E bardhe e hapur
3	Titulli i Cl ₂	% m/m	60 - 70
4	Kadmium (Cd)	mg/kg	max 5

5	Krom (Cr)	mg/kg	max 2.5
6	Nikel (Ni)	mg/kg	max 20
7	Plumb (Pb)	mg/kg	max 20

Hipokloriti I natriumit (NaOCl), “ Cilësia e Ujit të Pijshëm “ nga VKM nr 379 datë 25.05.2016

Nr.	Parametrat	Njësia e matjes	Specifikat Kimike
1	Gjëndja	-	Lëng i kthjellët
2	Ngjyra	-	E Verdhë e çelët
3	Densiteti në 20°C	gr/ml	1.13 - 1.28
4	Titulli i Cl ₂	%	12 - 15
5	Substanca Toksike	mg/kg	Limitet në mg/kg të klorit të lirë
6	Kadmium (Cd)	mg/kg	max 2.5
7	Krom (Cr)	mg/kg	max 2.5
8	Nikel (Ni)	mg/kg	max 2.5
9	Plumb (Pb)	mg/kg	max 15

Shtojca 7 pika 2.3.4,

2.3.4 Operatori ekonomik duhet të ketë në pronësi ose me qera/Huapërdorje 1 mjet të autorizuar për transport mallrash te rrezikshme sipas standartit të BE

Për të vërtetuar disponimin e mjeteve të kërkuara më lart operatori ekonomik duhet të paraqesë dokumentacionin e mëposhtëm:

Certifikate Pronesie , Leje qarkullimi, certifikaten e kontrollit teknik dhe siguracionin e mjetit (te vlefshme)Dhe per mjetet e mara me qera, kontraten perkatese te Qerese/Huapërdorje te shoqeruar me Certifikate Pronesie leje qarkullimi, certifikaten e kontrollit teknik dhe siguracionin e mjetit,(te vlefshme), per te gjithë periudhen e realizimit te kontrates objekt i ketij prokurimi.



Shtojca 7, Pika 2.3.6.,

2.3.6 Operatori Ekonomik Ofertues duhet te paraqesi Certifikatat ISO Si me poshte: Ofertuesit duhet te paraqesin Çertifikate Personale ISO 9001:2015, te standartit nderkombetar te menaxhimit te cilesise dhe /ose te njevlefshme, te cilat do te garantonin cilesine e produktit ose standart ISO me e avancuar, e perkthyer e noterizuar. Çerfikata duhet te jete e vlefshme dhe e leshuar nga organizma te vleresimit te konformitetit, te cilet jane akredituar nga DPA ose nga organizmat nderkombetar akreditues të njohur nga Republika e Shqiperisë.

Ne Çertifikate personale ISO 9001:2015 duhet te jete e perfshire Objekti i prokurimit.

Te nderuar Anetare te Komisionit.

Shoqeria METANI shpk. Ne cilesine e ofertuesit te mundshem ne perputhje me nenin 110,112,113, te ligjit 162/2020, i eshte drejtuar me Ankesa per Kriteret e Tenderit, Autoritetit Kontraktore **Shoqëria Rajonale Ujësjetllës Kanalizime Berat S.H.A, dhe Komisionit te Prokurimit Publik** ne ankesen tone per kriteret e tenderit duke argumentuar shkeljet ligjore te Autoritetit kontraktore si meposhte:

I. Ne Dokumentat Standarte te Tenderit ne shtojcen 7, pika 2.3.3, kapaciteti teknik specifikimet teknike shtojca 5 pika 1, te ndryshuara me date 13.02.2023, me njoftim ne SPE bashkelidhur shtojcen perkatese, Autoriteti Kontraktues kerkon si kriter kualifikues:

Operatori Ekonomik duhet te paraqesë Certifikaten e prodhuesit qe verteton konformitetin e produktit me standardin SSH EN 901: 2013 ‘Kimikate që përdoren për trajtimin e ujërave të destinuara për konsum njerëzor –**Calcium hypochlorite (Ca(OCl)₂) dhe Sodium hypochlorite**’, shoqeruar me:

- Lejen e importit të lëshuar nga Komisioni i Liçensave pranë Ministrisë së Shendetësisë për llojin e kimikatit, në rast se operatori ekonomik është importues.
- Leje mjedisore e tipit A të lëshuar nga Ministria e Mjedisit për kimikatin e kërkuar nga AK në rast se operatori ekonomik është prodhues vendas (vetdeklarim ne shtojcen nr.8).

Kontratë furnizimi me 1(një) importues të mallrave për kimikatin e përcaktuara në DST ose Kontratë furnizimi ose autorizim me prodhuesin në rast se operatori ekonomik është tregtues vendas.

I.1 Duke u ndalur ekzaktesisht ne kerkesen per paraqitjen e Certifikates se prodhuesit ku vertetohet konformiteti i standartit SSH EN 901:2013, i sjellim ne vemendje te Autoritetit Kontraktor se standarti SSH EN 901:2013, eshte pershtatja e Standartit europian EN 901:2013,. standart i aplikueshem vetem per cilesine e Hipokloratit te Natriumit percaktuar ne piken 1, faqe 6, dhe piken 3.1.6, faqe 7, ku percaktohet ne menyre te pa gabueshme numri CAS:7681-52-9, konfirmuar edhe ne Agjencine Europiane te Chimikateve <https://echa.europa.eu/substance-information/-/substanceinfo/100.028.790> si Hipoklorat Natriumi. Autoriteti Kontraktor kerkon Certifikaten e Prodhuesit jo vetem per Hipoklorat Natriumi por edhe per Hipoklorat Kalciumi ne kundershtim me dispozitat ligjore te neni 40, pika 3, te rPP, ku percaktohet: *Kërkesat e veçanta të kualifikimit duhet të përfshijnë të gjitha kriteret specifike, në përputhje me nenin 77, të LPP-së. Kriteret duhet të jenë në përpjesëtim dhe të lidhura ngushtë me aftësinë zbatuese, natyrën dhe vlerën e kontratës. Në çdo rast, autoriteti/enti kontraktor duhet të përcaktojë dokumentet*

konkrete që duhet të paraqesin operatorët ekonomikë për përmbushjen e këtyre kritereve. Ne kerkesen e shtojces 7, pika 2.3.2 Autoriteti kontraktor jo vetem qe nuk ka percaktuar ekzaktesisht dokumentet konkrete por kerkon per vertetimin e cilesise se Hipokloratit te Kalciumit nje dokument i cili nuk ekziston. Kerkesa per certifikaten e per standartin eshte jo vetem ne kundershtim me nenin 40, pika 3, te RPP por tregon paqartesi dhe mungese njohurie te specifikimeve teknike te kerkuara duke kerkuar certifikate standarti bazuar ne nje standart i cili nuk i perket mallit te kerkuar. Standartet e njesuara nga Drajtoria e Pergjithshme e Standarteve por edhe standartet e njesuara nderkombetare nuk jane objekt leshimi certifikate por udhezime te cilat percaktojne nje certifikim.

Nga parashtrimi i sipercituar Auoriteti Kontraktor ka deshtuar ne percaktimin e kritereve specifike si rrjedhoje e pa-afetise per te njohur karakteristikat dhe specifikat e mallit te kerkuar duke kerkuar dokument provues i cili nuk ekziston dhe ne te njejten kohe kerkon dokument provues i cili nuk i perket mallrit objekt kontrate. Pa aftesia profesionale ne njohjen e specifikimit teknik duke kerkuar dokumentacion bazuar ne nje standart kombetar/nderkombetar te ndryshem nga mallrat objekt kontrate eshte ne kundershtim me nenin 2, pika dh): *Qëllim i këtij ligji është: dh) të sigurojë integritet, besim publik dhe transparencë në procedurat e prokurimit publik.* Ne Procesverbalin per argumentimin dhe miratimin e specifikimeve teknike dhe kritereve per kualifikimin, Autoriteti kontraktor argumenton ne piken 2.3.2, te ketij procesverbali se baza ligjore e ketij kriteri eshte standarti SSH EN 901: 2013 ‘Kimikate që përdoren për trajtimin e ujërave të destinuara për konsum njerëzor’ I cili eshte nje standart vetem per mallin Hipoklorat Natriumi dhe si standart nuk percakton nje certificate per konformitetin e standartit dhe ligjin nr. 105/2014 “Për barnat dhe shërbimin farmaceutik” i cili eshte nje ligj i cili nuk rregullon apo percakton ne asnje dispozite te ketij ligji karakteristikat apo standartet per perdorim te mallrave objekt kontrate cka verteton se Autoriteti Kontraktor ne asnje moment nuk ka qene informuar lidhur me karakteristikat e mallarave objekt kontrate duke iu refereruar dispozitave te gabuara ne percaktimin e kerkeses se shtojces 7, pika 2.3.2.

Nga sa parashtruar mesiper kerkojme heqjen e ketij kriteri si nje kriter ne kundershtim me ligjin 162/2020 dhe rregullave te prokurimit publik.

II. Ne Dokumentat Standarte te Tenderit ne shtojcen 7 pika 2.3.6, kerkon :

2.3.7 Operatori Ekonomik Ofertues duhet te paraqesi Certifikatat ISO Si me poshte: Ofertuesit duhet te paraqesin Çertifikate Personale ISO 9001:2015, te standartit nderkombetar te menaxhimit te cilesise dhe /ose te njevlefshme, te cilat do te garantonin cilesine e produktit ose standart ISO me e avancuar, e perkthyer e noterizuar. Çerfikata duhet te jete e vlefshme dhe e leshuar nga organizma te vleresimit te konformitetit, te cilet jane akredituar nga DPA ose nga organizmat ndërkombetar akreditues të njohur nga Republika e Shqiperisë.

Ne Çertifikate personale ISO 9001:2015 duhet te jete e perfshire Objekti i prokurimit.

Certifikata e ISO 9001:2015 eshte certifikate e cila sipas edhe drejtorise se pergjitheshme te standartizimit garanton vetem sistemin e menaxhimit administrativ te subjekteve te Certifikuara duke garantuar vetem aspektin administrativ duke mos u lidhur ngushtesisht me procese prodhimi, furnizimi apo menaxhimit te mjedisit. Nisur nga specifika e pergjitheshme e stadartit te percaktuar ne ISO 9001:2015, perfshirja e emerteses se objektit te prokurimit eshte e pa baze pasi nese nje Operator Ekonomik ka ne fokusin e tij disa aktivitete tregtare nuk mund te pajset me certifikata per secilin aktivitet kur Certifikimi iso eshte nje standart i lidhur ngushte vetem me menaxhimin administrativ. Veprime te cilat administrata e



operatorit ekonomik i kryen ne menyre te njejte pa asnje ndryshim thelbesor pavaresisht objektit te aktivitetit.

Nga sa parashtruar mesiper kerkojme heqien e ketij kriteri si nje kriter ne kundershtrim me ligjin 162/2020 dhe rregullave te prokurimit publik.

Nga sa parashtruar ne piken 1 te kesaj Ankese duke vertetuar gabimet thelbesore ne hartimin e dokumentave te tenderit kerkojme Anullimin e Kesaj procedure prokurimi duke hartuar serisht ne perputhje me dispozitat ligjore Dokumentat e Tenderit ne zbatim te nenit 98, pika 1,c dhe d.

- **Bashkelidhur ne kete Ankese Standarti SSH EN 903:2013 i certifikuar nga Drejtoria e Pergjitheshme e Standartizimeve.**

5. Kërkesë për ekspertizë të posacme

Po

Jo

(Nëse po, specifikoni llojin e ekspertizës që kërkoni)

6. Kërkesë për përjashtim të zyrtarëve që do të merren me shqyrtimin e ankesës:

7. Lista e informacionit konfidencial:



Përcaktoni se cili informacion është konfidencial, nëse ka. Shpjegoni pse informacioni është ose një version i dokumenteve përkatëse me heqjen e pjesëve konfidenciale dhe një përmbledhje të përmbajtjes.

Kujdes :Ankimuesi duhet t'i bashkëlidhë ankimit, që do të paraqesë në autoritetin/ entin kontraktor dhe Komisionin e Prokurimit Publik, dokumentin bankar që vërteton pagesën e tarifës përkatëse për ankesën pranë Komisionit të Prokurimit Publik

Dërgojeni formularin e plotësuar të ankesës së prokurimit, të gjitha shtojcat e nevojshme dhe kopjet shtesë, pranë **Autoritetit /Enti Kontraktor dhe Komisionit te Prokurimit Publik.**

Shënim: Ankimuesi duhet ta dërgojë njëkohësisht ankesën në autoritetin/ entin kontraktor dhe Komisionin e Prokurimit publik

Nr. i faksit:

E-mail:

Nënshkrimi dhe vula e Ankuesit

Administratori/ Përfaqësuesi i autorizuar

Alisa Metani

Alisa



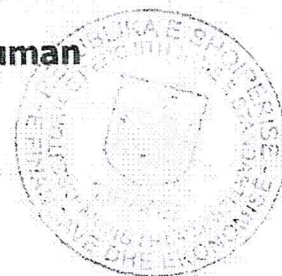
Alisa

STANDARD SHQIPTAR

SSH EN 901:2013

Kimikate që përdoren për trajtimin e ujrave të destinuara për konsum njerëzor - Hipokloriti i natriumit

Chemicals used for treatment of water intended for human consumption - Sodium hypochlorite



Rruga: Reshit Çoliaku (godina e ILCKPKI, kati VI)
Kutia Postare 98
1001 Tiranë – Shqipëri
Tel: + 355 (0) 4 222 62 55
E-mail: info@dps.gov.al
Web: <https://www.dps.gov.al>

Drejtoria e Përgjithshme e Standardizimit (DPS) është Organi Kombëtar i Standardizimit në Republikën e Shqipërisë.

DPS harton, miraton dhe publikon standardet e publikon standardet evropiane dhe ndërkombëtare, duke ju dhënë atyre statusin e Standardeve Shqiptare (SSH). Te njetat kompetenca ka edhe për Dokumentet e Standardizimit (DS), Miratimi formal i tyre bëhet nga Drejtori i Përgjithshëm i DPS.

Standardet Shqiptare hartohen dhe adoptohen nga Komitetet Teknike (KT), Anëtarët e Komiteteve Teknike janë specialistë të subjekteve shtetërore dhe private nga fusha të ndryshme të ekonomisë, që angazhohen vullnetarisht në këtë proces.

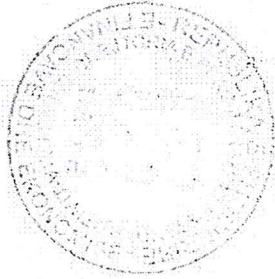
Standardi SSH EN 901:2013 është adoptuar nga DPS/KT 230 dhe miratuar nga DPS, si Standard Shqiptar (SSH), më 24.09.2013.

Standardi SSH EN 901:2013 botohet për herë të parë.

Teksti i standardit SSH EN 901:2013 është i njëjti me tekstin e standardit EN 901:2013 dhe riprodhohet me lejen e:

Komiteti European për Standardizim (CEN),
Avenue Marnix 17
B-1000 Brussels

Të gjitha të drejtat e shprehura nga kompanitë ndërkombëtare, në gdo lloj forme, në të gjithë botën, i takojnë kompanitë ndërkombëtare. Asnjë nisim për riprodhimin e tyre nuk mund të marrë vend në asnjë formë dhe në asnjë mënyrë nga CEN, CENELEC, ETSI, ISO dhe IEC, nëpërmjet të tyre.



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English Version

Chemicals used for treatment of water intended for human consumption - Sodium hypochlorite

Produits chimiques utilisés pour le traitement de l'eau destinée à la consommation humaine - Hypochlorite de sodium

Produkte zur Aufbereitung von Wasser für den menschlichen Gebrauch - Natriumhypochlorit

This European Standard was approved by CEN on 21 March 2013.

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

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Direktoria e Përgjithshme e Standardizimit

Foreword

This document (EN 901:2013) 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 November 2013, and conflicting national standards shall be withdrawn at the latest by November 2013.

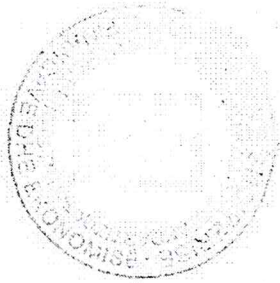
Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 901:2007.

Significant technical differences between this edition and EN 901:2007 are as follows:

— replacement of warning and safety precautions notes by labelling according to Regulation (EC) No 1272/2008.

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



Introduction

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

- a) this European Standard provides no information regarding 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. Use of the product covered by this European Standard is subject to regulation or control by National Authorities.

This product is a biocide and should comply with the relevant legislation in force. In the European Union, at the time of publication, this legislation is Directive 1998/8/EC [1].

1 Scope

This European Standard is applicable to sodium hypochlorite used for treatment of water intended for human consumption. It describes the characteristics of sodium hypochlorite and specifies the requirements and the corresponding test methods for sodium hypochlorite. It gives information on its use in water treatment. It also determines the rules relating to safe handling and use of sodium hypochlorite (see Annex B).

NOTE While this standard is not applicable to sodium hypochlorite generated in-situ (see bibliographic reference [7]), the limits for impurities and chemical parameters apply.

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 1233, Water quality — Determination of chromium — Atomic absorption spectrometric methods

EN ISO 3696:1995, Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)

EN ISO 12846, Water quality — Determination of mercury — Method using atomic absorption spectrometry (AAS) with and without enrichment (ISO 12846)

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

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

ISO 8268:1986, Water quality — Determination of cobalt, nickel, copper, zinc, cadmium and lead — Flame atomic absorption spectrometric methods

3 Description

3.1 Identification

3.1.1 Chemical name

Sodium hypochlorite.

3.1.2 Synonym or common names

Liquid bleach, soda bleach, bleach, bleach lye.

3.1.3 Relative molecular mass

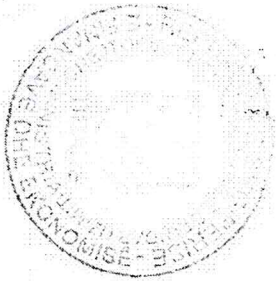
74,44.

3.1.4 Empirical formula

NaClO.

3.1.5 Chemical formula

NaClO.



3.1.6 CAS Registry Number ¹⁾

7681-52-9.

3.1.7 EINECS reference ²⁾

231-668-3.

3.2 Commercial form

The product is supplied as an aqueous solution with an available (active) chlorine concentration up to a mass fraction of 18 %.

3.3 Physical properties**3.3.1 Appearance and odour**

The product is a clear yellowish-green solution with a faint chlorinous odour.

3.3.2 Density

The density of the product varies between 1,13 g/ml and 1,30 g/ml at 20 °C.

3.3.3 Solubility in water

The product is capable of being mixed with water in any proportion.

3.3.4 Vapour pressure

Approximately 2,5 kPa at 20 °C.

3.3.5 Boiling point at 100 kPa ³⁾

Not applicable.

3.3.6 Crystallisation and freezing point

At about - 10 °C crystallisation of NaOCl · 6 H₂O starts.

Freezing of the concentrated product takes place between - 20 °C and - 30 °C.

3.3.7 Specific heat

The specific heat is 3,43 kJ/(kg.K) for a solution with an available active chlorine concentration of mass fraction between 14 % and 15 %.

3.3.8 Viscosity (dynamic)

2,6 mPa.s at 20 °C.

1) Chemical Abstracts Service Registry Number.

2) European Inventory of Existing Commercial Chemical Substances.

3) 100 kPa = 1 bar.

3.3.9 Critical temperature

Not applicable.

3.3.10 Critical pressure

Not applicable.

3.3.11 Physical hardness

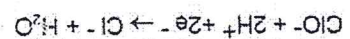
Not applicable.

3.4 Chemical properties

The product is an alkaline solution with a pH value greater than 11 at 20 °C.

It reacts with acids and acidic salts to form chlorine.

Vigorous reactions occur with reducing chemicals. It is a strong oxidant (E°_{red} for $ClO^- = 0,89 V$).



4 Purity criteria

4.1 General

This European Standard specifies the minimum purity requirements for sodium hypochlorite used for treating water intended for human consumption. Limits are given for impurities commonly present in the product. Depending on the raw material and the manufacturing process, other impurities may be present and, if so, the user, and when necessary the relevant authorities, shall be notified.

Users of the product should check the national regulations to clarify whether it is of appropriate purity for treating water intended for human consumption, taking into account raw water quality, required dosage, contents of other impurities and additives used in the product that are 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 the raw materials bring about the presence of significant amounts of impurities, by-products or additives, the user shall be notified.

4.2 Composition of commercial product

Sodium hypochlorite is available only in solutions with concentrations up to 10 % active chlorine at the time of delivery by the producer. Common concentrated products contain a minimum of 12 % active chlorine. Diluted solutions are also available.

The concentration of sodium hypochlorite shall be equal to or greater than the value specified by the manufacturer.

4.3 Impurities and main by-products

The product contains sodium chloride ($NaCl$) in equimolar amounts at minimum, and a small portion of sodium hydroxide ($NaOH$) which keeps the product alkaline. Thus a little amount of sodium carbonate (Na_2CO_3) can be present, too.

The sodium chlorate (NaClO_3) content shall not exceed a mass fraction of 5,4 % of available chlorine at the time of delivery by the producer. The product shall be visibly-free from deposits or suspended matter.

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

4.4 Chemical parameters

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

Table 1 — Chemical parameters

Parameter		Limit in mg/kg of available chlorine	
		Type 1	Type 2
Arsenic (As)	max.	1	5
Antimony (Sb)	max.	20	25
Cadmium (Cd)	max.	2,5	5
Chromium (Cr)	max.	2,5	5
Lead (Pb)	max.	15	15
Mercury (Hg)	max.	3,5	5
Nickel (Ni)	max.	2,5	10
Selenium (Se)	max.	20	25
		Limit in g/kg of available chlorine	
Sodium bromate ^a	max.	2,5	5,0
^a Sodium bromate is a by-product of the manufacturing process.			
NOTE Cyanide, which does not exist in a strong oxidising medium such as sodium hypochlorite, is not a relevant chemical parameter. Pesticides and polycyclic aromatic hydrocarbons are not by-products of the manufacturing process. For parametric values of sodium hypochlorite on trace metal content in drinking water, see bibliographic reference [1].			

5 Test methods

5.1 Sampling

Observe the general recommendations of ISO 3165 and take account of ISO 6206.

5.2 Analysis

5.2.1 Determination of available chlorine content (main product)

5.2.1.1 General

This method applies to all commercial products with available chlorine contents within the range of 70 g/l to 170 g/l.

NOTE It detects all oxidising agents being active in weak acidic solutions, i.e. hypochlorite/chlorine, iodate, and partially chloramines, Fe(III), etc. Bromate and chlorate are not covered under these conditions.

5.2.1.2 Principle

Sodium hypochlorite reacts with potassium iodide to release iodine in the presence of acetic acid. The iodine is titrated with sodium thiosulfate standard volumetric solution in the presence of starch indicator solution. The titration may also be carried out potentiometrically by the aid of titration automates, in which case the addition of soluble starch is unnecessary.

5.2.1.3 Reagents

All reagents shall be of a recognised analytical grade and the water used shall conform to grade 3, as specified in EN ISO 3696:1995 (de-ionised water for common laboratory purposes).

5.2.1.3.1 Potassium iodide solution, mass fraction 10 %

Weigh, to the nearest 0.1 mg, 100 g of potassium iodide, iodate-free, and dissolve in water and dilute to 1 l.

5.2.1.3.2 Acetic acid concentrated, of purity at least of mass fraction 99 %

5.2.1.3.3 Sodium thiosulfate standard volumetric solution, c(Na2S2O3.5H2O) = 0,1 mol/l

Standard volumetric solutions are commercially available, which might have to be diluted. Alternatively, a standard volumetric solution may be prepared by the following procedure:

Dissolve 24,8 g Na2S2O3.5H2O in a 1 000 ml one-mark volumetric flask in 0,75 l of water. After the temperature has equalised, make up to the mark with water and mix thoroughly.

To standardise: Weigh, to the nearest 0,1 mg, 3,600 g (m) of dry potassium iodate. Dissolve in water in a 1 000 ml one-mark volumetric flask, make up to the mark with water and mix (standard reference solution c(1/6 KIO3) = 0,1 mol/l). Place 200 ml of water in a 500 ml stoppered conical flask, add (2 ± 0,5) g of potassium iodide and stir to dissolve. Then introduce, by means of a pipette, 10,0 ml of sodium thiosulfate solution for standardization, add (15 ± 1) ml of hydrochloric acid solution (diluted 1 + 1 by volume) and (5 ± 1) ml of starch solution (5.2.1.3.4). Titrate immediately with the potassium iodate standard reference solution until the appearance of a blue coloration that persists for 30 s. Record the volume (V1) of iodate used. The actual concentration, c, of the sodium thiosulfate standard volumetric solution (Na2S2O3.5H2O), expressed in moles per litre is given by Formula (1)

$$c = \frac{A}{V_1 \times c_1} \quad (1)$$

where

c1 is the concentration, expressed in moles per litre, of the potassium iodate standard reference solution c(1/6 KIO3) = 0,1 mol/l;

V1 is the volume, in millilitres, of the sodium thiosulfate standard volumetric solution used for the standardization (V = 10 ml);

V2 is the volume, in millilitres, of potassium iodate standard reference solution used in the titration.

5.2.1.3.4 Starch solution, mass fraction 1 %

Make a slurry with (1 ± 0,1) g of starch and (2 ± 1) ml of water. Add (30 ± 5) ml of boiling water to the slurry. Stir to dissolve it and cool the solution. This solution needs to be filtered to avoid the decomposition of the starch which results in a vague end point. Keep the solution cool and use it within one week.



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Commercial indicators for iodine titration exist and may be used in place of the described starch solution, provided that their efficiency has been previously tested.

5.2.1.4 Apparatus

Ordinary laboratory apparatus and glassware.

5.2.1.5 Procedure

5.2.1.5.1 Test solution

Weigh to the nearest 0,1 mg 1 g of the laboratory sample (record mass m_1 in grams) into a 250 ml conical flask and dilute with water up to 100 ml.

5.2.1.5.2 Determination

Add 10 ml of the potassium iodide solution (5.2.1.3.1) and 5 ml of concentrated acetic acid (5.2.1.3.2).

Titrate at once with the sodium thiosulfate standard volumetric solution (5.2.1.3.3) until the iodine colour is nearly gone. Add 3 ml of the starch indicator solution (5.2.1.3.4) and complete the titration until the disappearance of the blue-black colour. Record the volume V_1 of the sodium thiosulfate standard volumetric solution added.

5.2.1.6 Expression of results

The available chlorine (Cl_2) content, C_2 , expressed in mass fraction in %, is given by Formula (2):

$$C_2 = (V_1 \times c \times 3,545) / (m_1) \quad (2)$$

where

V_1 is the volume, in millilitres, of the sodium thiosulfate standard volumetric solution (5.2.1.3.3);

c is the concentration in moles per litre, of the sodium thiosulfate standard volumetric solution (see 5.2.1.3.3);

3,545 is the mass in milligrams of chlorine (Cl_2) corresponding to 1 ml of sodium thiosulfate solution of $c(\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}) = 0,100 \text{ mol/l}$;

m_1 is the mass in grams of the laboratory sample used to prepare the test solution (5.2.1.5.1).

5.2.2 Impurities

5.2.2.1 Determination of sodium chlorate content (NaClO_3)

5.2.2.1.1 General

This method is used to determine the chlorate content, in the range between 3,75 g/l and 15 g/l, in sodium hypochlorite solutions for commercial use; it is specific for these species.

5.2.2.1.2 Principle

Direct determination of chlorate ions in a diluted solution of sodium hypochlorite by ion chromatography with suppressed conductimetric detection.

5.2.2.1.3 Reagents

All reagents shall be of a recognised analytical grade and the water used shall have a conductivity of $0,056 \mu\text{S}/\text{cm}$ (conform to grade 1, as specified in EN ISO 3696:1995).

5.2.2.1.3.1 Sodium carbonate and sodium hydrogen carbonate, eluant solution.

Mix one volume of sodium carbonate solution $c(\text{Na}_2\text{CO}_3) = 2 \text{ mmol/l}$ with one volume of sodium hydrogen carbonate solution $c(\text{NaHCO}_3) = 0,75 \text{ mmol/l}$.

5.2.2.1.3.2 Sulfuric acid solution $c(\text{H}_2\text{SO}_4) = 0,025 \text{ mol/l}$ regenerant solution.

5.2.2.1.3.3 Helium gas, high purity, for degassing eluant and regenerant solutions.

5.2.2.1.3.4 Sodium chlorate stock solution 1 g/l .

Weigh, to the nearest $0,0001 \text{ g}$, $0,2551 \text{ g}$ of NaClO_3 . Dissolve in 200 ml of the water (grade 1, EN ISO 3696:1995).

5.2.2.1.4 Apparatus

Ordinary laboratory apparatus and glassware, together with the following.

5.2.2.1.4.1 Ion chromatograph

5.2.2.1.4.2 Chemical suppressed conductivity detector

5.2.2.1.4.3 Anionic column and pre-column

Resin composed of $15 \mu\text{m}$ polystyrene/divinylbenzene substrate agglomerated with anion exchange latex that has been aminated.

5.2.2.1.4.4 Data logger/plotter, able to record and display the chromatographic peak heights.

5.2.2.1.4.5 Generator of water (grade 1, EN ISO 3696:1995).

5.2.2.1.5 Chromatographic conditions

— Eluant flow rate: 2 ml/min ;

— Regenerant flow rate: $2,5 \text{ ml/min}$;

— Full scale of conductivity: 30 mS ;

— Residual conductivity: $< 18 \text{ nS}$.

5.2.2.1.6 Procedure

5.2.2.1.6.1 Preparation of calibration solutions

Prepare calibration solutions in volumetric flasks by diluting accurately measured volumes of the chlorate stock solution (5.2.2.1.3.4) with the eluant solution (5.2.2.1.3.1), in accordance with Table 2.

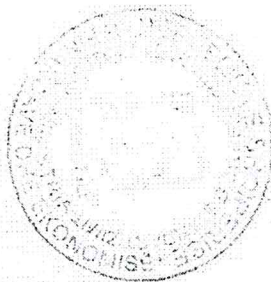


Table 2 — Calibration solutions for determination of chlorate content

Solution	ClO ₃ ⁻ in mg/l	NaClO ₃ in mg/l
1	3,75	4,78
2	7,5	9,57
3	11,25	14,35
4	15	19,13

NOTE Calibration is linear for concentration of chlorate ion between 3,75 mg/l and 15 mg/l in the diluted solution.

5.2.2.1.6.2 Preparation of test solution

Weigh to the nearest 0,1 mg 250 mg of the laboratory sample (record mass m_2 in milligrams) into a 100,0 ml measuring flask, fill up with eluant solution (5.2.2.1.3.1) and homogenise the test solution by shaking the sealed flask carefully.

5.2.2.1.6.3 Measurement of calibration and test solutions

Measure each calibration and test solution three times using a constant injection volume according to the requirements of the respective ion chromatograph. For each solution, the relative repeatability standard deviation shall be lower than 0,5 %.

5.2.2.1.7 Expression of results

The sodium chlorate content of the test solution (y in mg/l) is calculated from the calibration line which is obtained by linear regression with the five levels (four chlorate solutions and one blank solution) in the calibration procedure.

The sodium chlorate (NaClO₃) content of the laboratory sample, C_2 , expressed in grams per kilograms is given by Formula (3):

$$C_2 = (y \times V_2) / m_2 \quad (3)$$

where

y is the concentration of the test solution in milligrams per litre obtained via the regression line of the calibration;

V_2 is the volume, in millilitres, of the test solution (5.2.2.1.6.2);

m_2 is the mass of the laboratory sample, in milligrams (5.2.2.1.6.2).

The sodium chlorate (NaClO₃) content of the laboratory sample, C_3 expressed in mass fraction in % of available chlorine is given by Formula (4):

$$C_3 = \frac{(C_2 \times 10)}{C_1} \quad (4)$$

where

C_2 is the sodium chlorate (NaClO₃) content in g/kg of the laboratory sample;

C_1 is the available chlorine (Cl₂) content in mass fraction in % (5.2.1.6).

5.2.2.1.8 Repeatability limit

The absolute difference between two single test results, obtained under repeatability conditions, shall not be greater than the repeatability value, r , as calculated from Formula (5):

$$r = 0,001 z$$

where

z is the mean of the two results, expressed in mass fraction in %.

NOTE Repeatability conditions are conditions where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

5.2.2.2 Determination of sodium bromate content (NaBrO_3)

5.2.2.2.1 General

This method is used to determine the bromate content, in the range between 2 mg/l and 1 000 mg/l, in sodium hypochlorite solutions for commercial use.

5.2.2.2.2 - Principle

Direct determination of bromate ions in a diluted solution of sodium hypochlorite by ion chromatography with suppressed conductimetric detection.

The range of measurement can be limited by analytical column capacity. A dilution of the sample in the measurement range can be necessary. A convenient pre-treatment of the sample might be required (for example to remove any chloride, sulfate, carbonate or metals).

Direct determination of bromate by separation and indirect UV detection may also be carried out. The complete analytical procedure is described in Annex D.

5.2.2.2.3 Reagents

All reagents shall be of a recognised analytical grade and the water used shall have a conductivity of 0,056 $\mu\text{S}/\text{cm}$ (conform to grade 1 as specified in EN ISO 3696:1995).

5.2.2.2.3.1 Sulfuric acid solution (H_2SO_4) = 0,025 mol/l regenerant solution.

5.2.2.2.3.2 Helium gas, high purity, for degassing eluent and regenerant solutions.

5.2.2.2.3.3 Stock sodium carbonate solution, $c(\text{Na}_2\text{CO}_3) = 0,5$ mol/l.

Dissolve 53,0 g of anhydrous sodium carbonate in 800 ml of water (grade 1, EN ISO 3696:1995) in a 1 000 ml volumetric flask and make up to the volume with water (grade 1, EN ISO 3696:1995).

Store the solution at 2 °C to 6 °C in a polyethylene or glass bottle and renew it every 6 months.

5.2.2.2.3.4 Eluent carbonate solution, $c(\text{Na}_2\text{CO}_3) = 0,009$ mol/l.

Introduce 36 ml of the stock sodium carbonate solution 0,5 mol/l (5.2.2.2.3.3) into a 2 000 ml volumetric flask and make up to the volume with water (grade 1, EN ISO 3696:1995).

Store the solution at 2 °C to 6 °C in polyethylene or glass bottle and renew it every week.

Degas the solution with helium (5.2.2.2.3.2) before use according to the instructions given by the manufacturer.

Eluent should be maintained under an inert helium atmosphere to avoid carbonate contamination.

5.2.2.2.3.5 Stock standard bromate solution, $w(\text{BrO}_3) = 1\ 000\ \text{mg/l}$

Dry 1,5 g of potassium bromate for at least 1 h at 105 °C. Store the dried product in a desiccator. Weigh, to the nearest 0,0 001 g, 1,3 057 g of the dry potassium bromate. Introduce into a 1 000 ml volumetric flask. Dissolve in 800 ml of water (grade 1, EN ISO 3696:1995) and make up to the volume with water (grade 1, EN ISO 3696:1995). Store the solution at 2 °C to 6 °C in polyethylene or glass bottle and renew it every 12 months.

A commercial standard stock solution at required concentration may be also used.

5.2.2.2.3.6 Bromate standard solution, $w(\text{BrO}_3) = 10\ \text{mg/l}$

Introduce 1 ml of stock standard bromate solution (5.2.2.2.3.5) into a 100 ml volumetric flask. Make up to the volume with water (grade 1, EN ISO 3696:1995).

Store the solution at 2 °C to 6 °C in polyethylene or glass bottle and renew it every 3 months.

5.2.2.2.4 Apparatus

Ordinary laboratory apparatus and glassware, together with the following.

5.2.2.2.4.1 Ion chromatograph

5.2.2.2.4.2 Chemical or electrochemical suppressed conductivity detector

5.2.2.2.4.3 Anionic column and pre-column

Resin composed of 15 μm polystyrene/divinylbenzene substrate agglomerated with anion exchange latex that has been aminated.

5.2.2.2.4.4 Pre-treatment cartridges

- Cationic exchangers as Ag-form (Silver) (cartridge);
- cationic exchangers as H-form (Hydrogen) (cartridge).

If necessary, cationic exchangers as Ba-form (Barium) (cartridge) may be used to suppress sulfate interferences.

5.2.2.2.4.5 Generator, of water (grade 1, EN ISO 3696:1995).

5.2.2.2.5 Chromatographic conditions

- eluant flow rate: 1 ml/min;
- regenerant flow rate: 2,5 ml/min or electrochemical regeneration;
- recommended sampling loop size: 50 μl to 100 μl .

5.2.2.2.6 Procedure

5.2.2.2.6.1 Preparation of calibration solutions

Prepare calibration solutions in volumetric flasks by diluting accurately measured volumes of the bromate stock solution (5.2.2.2.3.5) with the eluant carbonate solution (5.2.2.2.3.4) in accordance with Table 3.

Solution	BrO ₃ ⁻ in mg/l	NaBrO ₃ in mg/l
1	0,2	0,236
2	0,5	0,59
3	1	1,18
4	2	2,36

Table 3 — Calibration solutions for determination of bromate content

NOTE 1 Calibration is linear for concentration of bromate ion between 0,2 mg/l and 2 mg/l in the diluted solution.

NOTE 2 The calibration solutions will be prepared freshly on the day of use.

5.2.2.2.6.2 Sample pre-treatment (if required)

If sample pre-treatment is necessary (presence of chloride and/or carbonate in large amount in the sample that can interfere with the bromate determination), calibration solutions and diluted test solutions shall be treated as follows:

- connect the cationic exchangers (cartridges) in series in order of Ag-H;
 - using a 10 ml syringe, flush the cartridges with 10 ml of water;
 - fill a 10 ml syringe with sample and attach it to the cartridge exit;
 - flush it with 5 ml of sample;
 - continue to inject sample through the cartridges and collect the next 5 ml of treated sample for analysis;
 - purge the treated sample with helium gas for 5 min to remove the remaining carbonate.
- If necessary, a Ba-form cartridge may be added to remove sulfate; the three pre-treatment cartridges should then be attached in the following order: Ba-form, Ag-form, and H-form.

5.2.2.2.6.3 Preparation of test solutions

Weigh to the nearest 0,1 mg, 250 mg of the laboratory sample (record masses *m*_s in milligrams) into a 100,0 ml measuring flask, fill up with eluant solution (5.2.2.2.3.4) and homogenise the test solution by shaking the sealed flask carefully.

5.2.2.2.6.4 Measurement of calibration and test solutions

Measure each calibration solution and test solution three times using a constant injection volume according to the requirements of the respective ion chromatograph. For each solution, the relative repeatability standard deviation shall be lower than 0,5 %.

5.2.2.2.7 Expression of results

The sodium bromate content of the test (y_1 in mg/l) is calculated from the calibration line which is obtained by linear regression with the five levels (four bromate solutions and one blank solution) in the calibration procedure. The sodium bromate (NaBrO_3) content of the laboratory sample, C_4 , expressed in milligrams per kilogram is given by Formula (6):

$$C_4 = (y_1 \times V_3 \times 10) / m_3 \quad (6)$$

where

y_1 is the concentration of the test solution in mg/l obtained via the regression line of the calibration;

V_3 is the volume in millilitres of the test solution (5.2.2.2.6.3);

m_3 is the mass of the laboratory sample in milligrams (5.2.2.2.6.3).

The sodium bromate (NaBrO_3) content of the laboratory sample, C_5 , expressed in mass fraction of available chlorine is given by Formula (7):

$$C_5 = (C_4 \times 10) / C_1 \quad (7)$$

where

C_4 is the sodium bromate (NaBrO_3) content in mg/kg of the laboratory sample;

C_1 is the available chlorine (Cl_2) content in mass fraction in % (5.2.1.6).

5.2.2.2.8 Repeatability limit

The absolute difference between two single test results, obtained under repeatability conditions, shall not be greater than the repeatability value, r , as calculated from Formula (8):

$$r = 0,005 z \quad (8)$$

where

z is the mean of the two results, expressed in mass fraction in %.

NOTE Repeatability conditions are conditions where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

5.2.3 Chemical parameters

5.2.3.1 Determination of antimony (Sb), arsenic (As), cadmium (Cd), chromium (Cr), lead (Pb), nickel (Ni) and selenium (Se)

5.2.3.1.1 Principle

The elements arsenic, antimony, cadmium, chromium, lead, nickel and selenium are determined by atomic absorption spectrometry.

5.2.3.1.2 Reagents

All reagents shall be of a recognised analytical grade and the water used shall conform to grade 2 as specified in EN ISO 3696:1995.

5.2.3.1.2.1 Nitric acid, concentrated, density $\rho = 1,42$ g/ml.

5.2.3.1.3 Procedure

5.2.3.1.3.1 Test portion

Weigh, to the nearest 0,001 g, 20 g (m_2) from the laboratory sample into a glass beaker.

5.2.3.1.3.2 Test solution

Evaporate until a wet residue is obtained, cool, add 1 ml of nitric acid (5.2.3.1.2.1), dilute with a few millilitres of water, transfer quantitatively to a 100 ml volumetric flask, make up to the volume with water and mix. Carry out the evaporation carefully and not to dryness in order to avoid possible losses of arsenic and selenium.

5.2.3.1.3.3 Determination

Determine the content of elements in the test solution (5.2.3.1.3.2) in accordance with the following methods:

— Cd, Ni and Pb: in accordance with ISO 8288:1986, Method A;

— Cr: in accordance with EN 1233;

— As, Se and Sb: in accordance with the method given in Annex C.

These methods will give an interim result (y) expressed in milligrams per litre which needs to be converted to give the final concentration according to the formula in 5.2.3.1.3.4.

5.2.3.1.3.4 Expression of results

From the interim result (y) determined (see 5.2.3.1.3.3), the content, C_0 , of each element in the laboratory sample, expressed in milligram of available chlorine is given by Formula (9):

$$C_0 = y \times \frac{100}{V} \times \frac{m_2}{C_1} \quad (9)$$

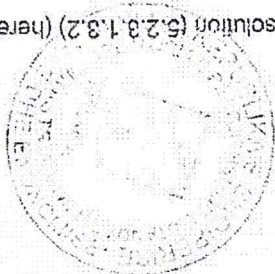
where

y is the interim result (5.2.3.1.3.3);

V is the volume, expressed in millilitres, of the test solution (5.2.3.1.3.2) (here $V_1 = 100$ ml);

m_2 is the mass, expressed in grams, of the test portion;

C_1 is the available chlorine (Cl_2) content in mass fraction in % (5.2.1.5).



5.2.3.2 Determination of mercury content (Hg)

5.2.3.2.1 Principle

The element mercury is determined by flameless atomic absorption spectrometry in accordance with EN ISO 12846.

5.2.3.2.2 Reagents

All reagents shall be of a recognised analytical grade and the water used shall conform to the grade 3 as specified in EN ISO 3696:1995.

5.2.3.2.2.1 Nitric acid, mass fraction 65 %.

5.2.3.2.2.2 Sulfuric acid, mass fraction 30 %.

5.2.3.2.2.3 Hydroxylammonium chloride solution (NH₂OH.HCl), mass fraction 10 %.

5.2.3.2.2.4 Tin(II) chloride solution, mass fraction 10 %.

5.2.3.2.3 Procedure

5.2.3.2.3.1 Test portion

Pipette 10 g (*m_s*) of the laboratory sample and transfer to approximately 70 ml of water taking care to avoid sputtering.

5.2.3.2.3.2 Test solution

Quantitatively transfer the test portion to a gas washing flask, capacity 250 ml, with the gas inlet equipped with a porous glass frit. Dilute the contents of the gas washing flask with water to obtain a total volume of 100 ml. Transfer to a volumetric flask (solution A).

Pipette accurately 5 ml of the sodium hypochlorite (solution A) and dilute in a 250 ml graduated gas washing flask. During gentle shaking, add 30 ml of hydroxylammonium chloride solution (5.2.3.2.2.3), 5 ml of nitric acid (5.2.3.2.2.1) and 2 ml of tin(II) chloride solution (5.2.3.2.2.4) successively. Close immediately the gas washing flask with the gas inlet equipped with a porous glass frit.

5.2.3.2.3.3 Determination

Proceed as described in EN ISO 12846.

5.2.3.2.3.4 Expression of results

The interim result for mercury content (*y*) expressed in milligrams per litre is given by the following formula:

$$y = y_A \times \frac{V_T}{10} \quad (10)$$

where

y_A is the result obtained, for the concentration of mercury in solution A, expressed in milligrams per litre;

V_T is the volume in millilitres of the test solution.

The content of mercury, *C₇*, in milligrams per kilogram of available chlorine is given by Formula (11):

$$C_1 = y \times \frac{m}{100} \times \frac{10}{G} \quad (11)$$

where

m_s is the mass expressed in grams, of the test portion;

C_1 is the available chlorine content in mass fraction in % (5.2.1.6).

6 Labelling - transportation - storage

6.1 Means of delivery

Sodium hypochlorite shall be delivered:

- a) in containers of polyethylene or polyvinyl chloride (PVC) with external glass fibre reinforcement (GFR);
- b) in steel tank wagons lined with rubber or coated with a suitable plastic.

The containers shall be closed so that no pressure can build up inside and no liquid can escape. The closure shall be protected from unintentional opening.

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

6.2 Labelling according to the EU legislation ⁴⁾

The following labelling requirements shall apply to sodium hypochlorite at the date of publication of this European Standard.

— Signal word:

Danger

— Hazard statements:

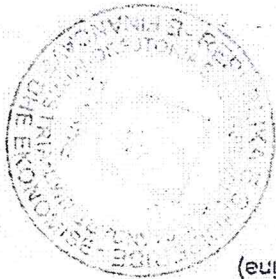
H314: Causes severe skin burns and eye damage.

H400: Very toxic to aquatic life.

EUH031: Contact with acids liberates toxic gas (chlorine)



Figure 1 — GHS 05



4) See [3].

h3



Figure 2 — GHS 09

The regulation [3] contains a list of substances classified by the EU. It contains only a single entry for classification and labelling of the substance which is however not appropriate for the various solutions placed on the market, since the hazards vary at different concentrations depending on the individual content of free chlorine and free alkali; therefore no classification for diluted solutions can be given here. Diluted solutions and 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 hypochlorite solution is listed as UN Number ⁵⁾ 1791.

- RID ⁶⁾ ADR ⁷⁾: class 8, classification code C9, packing group II or packing group III.
- IMDG ⁸⁾: class 8.
- IATA ⁹⁾: class 8.

6.4 Marking

The marking shall include the following:

- name "sodium hypochlorite solution", trade name, grade and type;
- net mass;

5) United Nations Number.

6) Regulations concerning International carriage of Dangerous goods by rail.

7) European Agreement concerning the international carriage of Dangerous goods by Road.

8) International Maritime transport of Dangerous Goods.

9) International Air Transport Association.

- name and address of supplier and/or manufacturer;
- statement "this product conforms to EN 901".

6.5 Storage

6.5.1 General

Sodium hypochlorite shall be protected against light, and particularly direct sunlight. It shall be stored in cool rooms in containers made from metal with internal coating or suitable plastics materials. To protect metal containers from corrosion, they shall be either rubber-lined or plastics-coated.

Pressure build-up in the containers should be prevented by suitable venting.

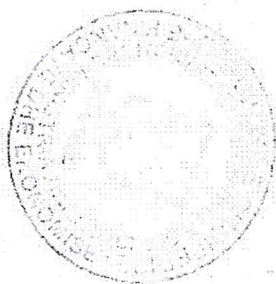
6.5.2 Long term stability

The stability is greatly affected by heat, light, pH, and the presence of heavy metal ions. The solution gradually decomposes resulting in the reduction of the concentration of the active chlorine and in the liberation of oxygen gas. The concentration of chlorate increases.

6.5.3 Storage incompatibilities

In the presence of acids or acidic salts: formation of chlorine

In the presence of high concentrations of ammonia, ammonium salts and derivatives, and isocyanurate derivatives: vigorous reaction with the formation of highly explosive nitrogen trichloride.



Annex A (informative)

General information on sodium hypochlorite

A.1 Origin

A.1.1 Raw materials

Sodium hypochlorite is manufactured from chlorine (Cl_2) and sodium hydroxide (NaOH).

A.1.2 Manufacturing process

It is produced by reacting gaseous chlorine with a solution of sodium hydroxide.

A.2 Use

A.2.1 Function

Sodium hypochlorite functions in water treatment are, removing ammonium compounds, oxidising sulfides and oxidising iron(II) to iron(III) and as a disinfectant.

A.2.2 Form in which it is used

It is used as delivered or diluted if necessary, depending on the required dose.

A.2.3 Treatment dose

The treatment dose depends on the composition of the raw water. Care should be taken not to exceed a maximum concentration of active chlorine in the water supply, usually a few tenths of 1 mg/l.

A.2.4 Means of application

It is applied using a metering-pump, an injection feeder or a constant head gravity feeder.

A.2.5 Secondary effects

The secondary effects include the following:

- slight increase in pH value;
- slight increase in the chloride content;
- oxidation of organic compounds; formation of halogenated organic substances, especially trihalomethanes, is possible;
- local precipitation of carbonate at the injection point.

A.2.6 Removal of excess product

The most practical method is using a reducing agent such as sulfur dioxide gas or an aqueous solution of a sulfite compound. Other methods can use activated carbon or hydrogen peroxide.

Annex B
(normative)

General rules relating to safety

B.1 Rules for safe handling and use

The supplier shall provide current safety instructions.

B.2 Emergency procedures

B.2.1 First aid

In case of contact with the skin rinse with copious amounts of water and remove any contaminated clothing.

In case of contact with the eyes, rinse immediately with copious amounts of water for at least 15 min and consult a doctor.

B.2.2 Spillage

Do not drain into waste water piping. Collect any leaking liquid in plastic containers. Contain spilled liquid and absorb with absorbing material such as kieselguhr, universal absorbent, etc. - do not use sawdust. Collect contaminated residues in plastic containers. Clean contaminated tools by rinsing with plenty of water.

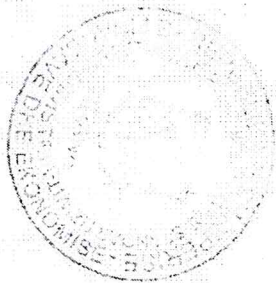
Dilute with water and reduce the solution with either sodium sulfite, sodium hydrogen sulfite, hydrogen peroxide or sodium thiosulfate. Disposal shall be carried out in accordance with the local regulations.

B.2.3 Fire

Non-combustible, but poses an extra hazard with fires.

Cool fire-endangered containers with water.

Use appropriate means to extinguish fires.



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Annex C (normative)

Determination of arsenic, antimony and selenium (atomic absorption spectrometry hydride technique)

C.1 General principle

SAFETY PRECAUTIONS: Arsenic, antimony and selenium and their hydrides are toxic. Handle with care.

Arsenious acid, antimonous acid and selenious acid, the As(III), Sb(III) and Se(IV) oxidation states of arsenic, antimony and selenium, are instantaneously converted by a sodium borohydride reagent in an acid solution to their volatile hydrides. The hydrides are continuously purged by argon or nitrogen into an appropriate atomiser of an atomic absorption spectrometer and converted to gas-phase atoms. The sodium borohydride reducing agent, by rapid generation of the elemental hydrides in an appropriate reaction cell, minimises dilution of the hydrides by the carrier gas and provides rapid, sensitive determinations of arsenic, antimony and selenium.

The sample is digested to solubilise particulate As, Sb and Se. The digested solutions are treated separately for determining As, Sb and Se to convert them to As(III), Sb(III) and Se(IV) oxidation states.

C.2 Interferences

Interferences are minimised because the As, Sb and Se hydrides are removed from the solution containing the most potential interfering substances. Slight response variations occur when acid matrices are varied. Control these variations by treating standards and samples in the same manner. Low concentrations of noble metals (approximately 100 µg/l of Ag, Au, Pt, Pd, etc.) concentrations of Cu, Ni and Pb at or greater than 1 mg/l, and concentrations between 0,1 mg/l and 1 mg/l of hydride-forming elements (Bi, Sn and Te) can suppress the response of As, Sb and Se hydrides due to the formation of mixed metal: As-Sb or -Se compounds. The presence of As, Sb and Se in each other's matrices can cause similar suppression. Reduced nitrogen oxides resulting from HNO₃ digestion and nitrite also can suppress instrumental response for all elements. Large concentrations of iodide interfere with the Se determination by reducing Se to its elemental form. Do not use any glassware for determining Se that has been used with iodide reduction of As(V).

C.3 Reagents

All reagents shall be of a recognised analytical grade and the water used shall conform to grade 3 in accordance with EN ISO 3696:1993.

C.3.1 Sodium tetrahydroborate (sodium borohydride).

Dissolve 8 g NaBH₄ in 200 ml of NaOH, c(NaOH) = 0,1 mol/l. Prepare fresh daily.

C.3.2 Sodium iodide, pre-reductant solution.

Dissolve 50 g NaI in 500 ml of water. Prepare fresh daily.

C.3.3 Sulfuric acid, solution c(H₂SO₄) = 9 mol/l.

C.3.4 Sulfuric acid, solution c(H₂SO₄) = 1,25 mol/l.

— stock Se(VI) solution: Dissolve 2,393 g of sodium selenate Na_2SeO_4 in water containing 10 ml of nitric acid (C.3.5). Transfer quantitatively to a 1 000 ml one-mark volumetric flask and make up to the mark with water and mix; 1,00 ml contains 1,00 mg Se(VI);

C.3.13 Selenium(VI) solutions:

— standard Se(IV) solution: Dilute into a 1 000 ml one-mark volumetric flask 10 ml of intermediate Se(IV) solution to the mark with water containing the same concentration of acid used for sample preservation (2 ml to 5 ml nitric acid (C.3.5)) and mix. Prepare the solution daily when checking the equivalent of instrument response for Se(IV) and Se(VI); 1,00 ml contains 0,100 μg Se(IV).

— intermediate Se(IV) solution: Dilute into a 1 000 ml one-mark volumetric flask 10 ml of stock Se(IV) solution to the mark with water containing 10 ml of hydrochloric acid (C.3.7) and mix; 1,00 ml contains 10,0 μg Se(IV);

— stock Se(IV) solution: Dissolve 2,150 g of sodium selenite, Na_2SeO_3 in water containing 10 ml hydrochloric acid (C.3.7) and transfer quantitatively to a 1 000 ml one-mark volumetric flask and make up to the mark with water and mix; 1,00 ml contains 1,00 mg Se(IV);

C.3.12 Selenium(IV) solutions:

— standard As(V) solution: Prepare as for As(III) above; 1,00 ml contains 0,100 μg As(V).

— intermediate As(V) solution: Prepare as for As(III) above; 1,00 ml contains 10,0 μg As(V);

— stock As(V) solution: Dissolve 1,534 g of arsenic pentoxide, As_2O_5 in water containing 4 g NaOH. Transfer quantitatively to a 1 000 ml one-mark volumetric flask and make up to the mark with water and mix; 1,00 ml contains 1,00 mg As(V);

C.3.11 Arsenic(V) solutions:

— standard As(III) solution: Dilute into a 1 000 ml one-mark volumetric flask 10 ml intermediate As(III) solution to the mark with water containing the same concentration of acid used for sample preservation (2 ml to 5 ml nitric acid (C.3.5)) and mix; 1,00 ml contains 0,100 μg As(III). Prepare diluted solutions daily.

— intermediate As(III) solution: Dilute into a 1 000 ml one-mark volumetric flask 10 ml stock As(III) solution to the mark with water containing 5 ml hydrochloric acid (C.3.7) and mix; 1,00 ml contains 10,0 μg As(III);

— stock As(III) solution: Dissolve 1,320 g of arsenic trioxide, As_2O_3 in water containing 4 g of NaOH. Transfer quantitatively to a 1 000 ml one-mark volumetric flask and make up to the mark with water and mix; 1,00 ml contains 1,00 mg As(III);

C.3.10 Arsenic(III) solutions:

C.3.9 Hydrogen, commercial grade.

C.3.8 Argon (or nitrogen), commercial grade.

C.3.7 Hydrochloric acid, density (ρ) = 1,16 g/ml.

C.3.6 Perchloric acid, density (ρ) = 1,66 g/ml.

C.3.5 Nitric acid, density (ρ) = 1,42 g/ml.

Cautiously add 35 ml sulfuric acid, density (ρ) = 1,84 g/ml to about 400 ml of water, allow to cool and adjust the volume to 500 ml.

- intermediate Se(VI) solution: Prepare as for Se(IV) above; 1,00 ml contains 10,0 µg Se(VI);
- standard Se(VI) solution: Prepare as for Se(IV) above; 1,00 ml contains 0,100 µg Se(VI).

C.3.14 Antimony solutions:

- stock Sb solution: Dry 2 g of potassium antimonyl tartrate hemihydrate (antimony potassium tartrate) ($C_4H_4O_7SbK \cdot 0,5 H_2O$) at 100 °C for 1 h. Dissolve 1,669 g in water, transfer quantitatively to a 1 000 ml one-mark volumetric flask and make up to the mark with water and mix; 1,00 ml contains 1,00 mg Sb;
- intermediate Sb solution: Dilute into a 1 000 ml one-mark volumetric flask 10 ml of stock Sb solution to the mark with water containing 10 ml of hydrochloric acid (C.3.7) and mix; 1,00 ml contains 10,0 µg Sb;
- standard Sb solution: Dilute into a 1 000 ml one-mark volumetric flask 10 ml of intermediate Sb solution to the mark with water containing the same concentration of acid used for sample preservation (2 ml to 5 ml nitric acid (C.3.5)) and mix; 1,00 ml contains 0,100 µg Sb. Prepare the diluted solutions daily.

C.4 Apparatus

Ordinary laboratory apparatus and glassware, together with the following:

C.4.1 Atomic absorption spectrometer, equipped with gas flow meters for argon (or nitrogen) and hydrogen, As, Sb and Se electrodeless discharge lamps with background correction at measurement wavelengths and an appropriate strip-chart recorder.

NOTE Certain atomic absorption atomisers and hydride reaction cells are available commercially for use with the sodium borohydride reagent.

C.4.2 Atomiser

Use one of the following:

- Boling-type burner¹⁰⁾ head for argon (or nitrogen)-air entrained-hydrogen flame;
- cylindrical quartz cell, 10 cm to 20 cm long, electrically heated by external Ni-Cr wire to 800 °C to 900 °C;
- cylindrical quartz cell with internal fuel rich hydrogen-oxygen (air) flame.

The transparency of quartz cells deteriorates over several months of use. The transparency can be restored by treatment with 40 % hydrofluoric acid (HF).

SAFETY PRECAUTIONS: Be careful while handling HF, which is toxic and corrosive and avoid prolonged contact of quartz with HF.

C.4.3 Reaction cell for producing As, Sb or Se hydrides

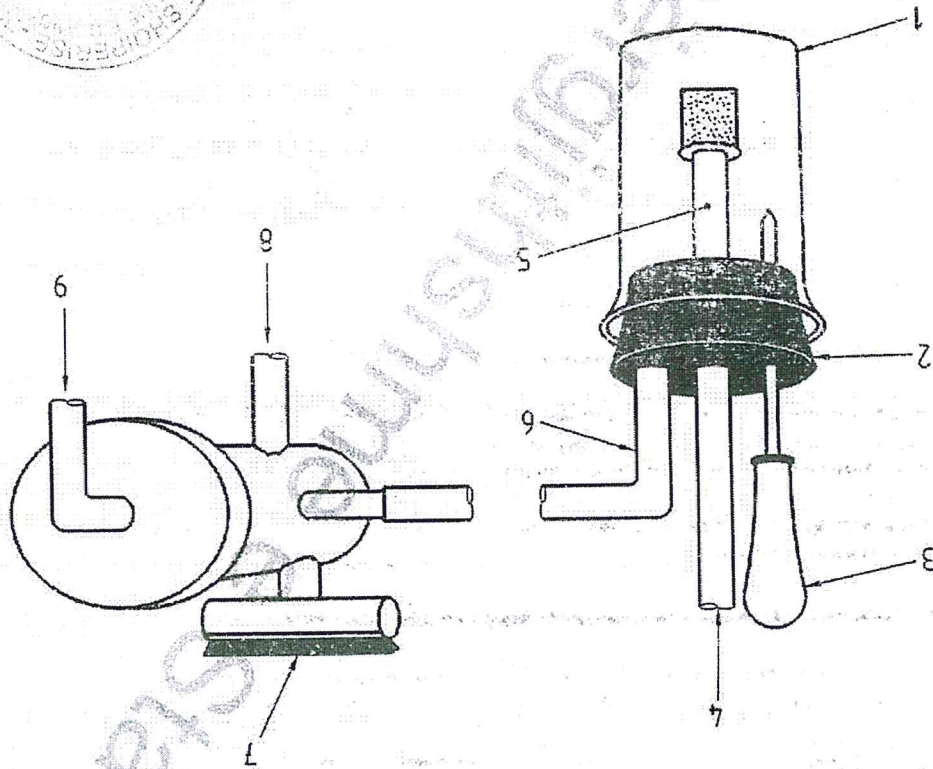
An example of a reaction cell is given in Figure C.1.

A commercially available system is acceptable if it utilises liquid sodium borohydride reagents; accepts samples digested in accordance with C.5.3, accepts between 4 mol/l to 6 mol/l (HCl); and is efficiently and precisely stirred by the purging gas and/or a magnetic stirrer.

Irrespective of the hydride reaction cell-atomiser system selected, it shall meet the following quality-control considerations:

¹⁰⁾ Boling is the name of the inventor of this type of burner for rapid combustion of the hydrides.

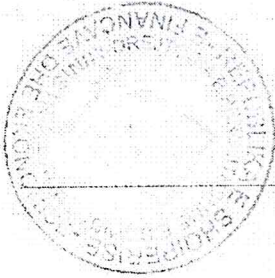
- a) It shall provide a precise and reproducible calibration curve between 0 µg/l and 20 µg/l As, Sb or Se and a detection limit between 0,1 µg/l and 0,5 µg/l As, Sb or Se.
 - b) When carried through the entire procedure, oxidation state couples (As(III) - As(V) or Se(IV) - Se(VI)) shall cause equal instrument response.
 - c) Sample digestion shall yield 90 % or greater recovery of added As(III), As(V), Se(VI), Se(IV) or Sb.
- C.4.4 Dropper and syringe capable of delivering 0,5 ml to 3,0 ml sodium borohydride reagent:**
- Exact and reproducible addition is required so that the production of hydrogen gas does not vary significantly between determinations.



Key

1	beaker 250 ml
2	rubber stopper
3	dropper
4	auxiliary nitrogen
5	gas dispersion tube
6	outlet tube
7	burner
8	hydrogen
9	nitrogen

Figure C.1 — Reaction cell for producing As, Sb or Se hydrides



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C.5 Procedure

C.5.1 Preparation of the apparatus

Connect the inlet of the reaction cell with the auxiliary purging gas controlled by a flow meter. If a drying cell between the reaction cell and atomiser is necessary, use only anhydrous CaCl_2 and not CaSO_4 , as it can retain SeH_2 . Before using the hydride generation/analysis system, optimise operating parameters. Aspirate the aqueous solutions of As, Sb and Se directly into the flame to facilitate atomiser alignment. Align quartz atomisers for maximum absorbance. Establish a purging gas flow, concentration and rate of addition of sodium borohydride reagent, solution volume, and stirring rate for optimum instrument response for the chemical species to be analyzed. If a quartz atomiser is used, optimise the cell temperature. If sodium borohydride reagent is added too quickly, rapid evolution of hydrogen will unbalance the system. If the volume of solution being purged is too large, the absorption signal will be decreased. Recommended wavelengths are 193,7 nm, 196,0 nm and 217,6 nm for As, Se and Sb.

C.5.2 Preparation of calibration solutions

Transfer 0,00 ml; 1,00 ml; 2,00 ml; 5,00 ml; 10,00 ml; 15,00 ml and 20,00 ml of standard solutions of As(III), Se(IV) or Sb to 100 ml volumetric flasks and make up to volume with water containing the same acid concentration used for sample preservation (commonly 2 ml to 5 ml nitric acid (C.3.5)). This yields calibration solutions of 0 $\mu\text{g/l}$, 1 $\mu\text{g/l}$, 2 $\mu\text{g/l}$, 5 $\mu\text{g/l}$, 10 $\mu\text{g/l}$, 15 $\mu\text{g/l}$ and 20 $\mu\text{g/l}$ As, Se or Sb. Prepare fresh daily.

C.5.3 Preparation of test solutions and standard solutions

Add 50 ml of the sample or As(III), Se(VI) or Sb standard solution to a 250 ml beaker. Alternatively, prepare standard solutions by adding aliquots of solutions containing 5 μg of As, Se or Sb directly to the beaker and dilute to 50 ml in this beaker, achieving a concentration of 100 $\mu\text{g/l}$ of the respective solutions. Add 7 ml of sulfuric acid $c(\text{H}_2\text{SO}_4) = 9 \text{ mol/l}$ (C.3.3) and 5 ml of nitric acid (C.3.5). Add a small boiling chip or glass beads if necessary. Evaporate to SO_3 fumes. Maintain oxidising conditions at all times by adding small amounts of nitric acid, to prevent solution from darkening.

Maintain an excess of nitric acid until all organic matter is destroyed. Complete digestion is usually indicated by a light-coloured solution. Cool slightly, add 25 ml of water and 1 ml of perchloric acid (C.3.6) and again evaporate to SO_3 fumes to expel oxides of nitrogen.

Monitor effectiveness of digestion procedure used by adding 5 ml of a standard arsenic solution, 5 ml of a standard selenium solution or 5 ml of a standard antimony solution to 50 ml of the sample and measuring recovery. Average recoveries shall be greater than 90 %. Alternatively, use 100 ml micro-Kjeldahl flasks for the digestion of total recoverable arsenic, selenium or antimony, thereby improving digestion effectiveness. After final evaporation of SO_3 fumes, dilute to 50 ml for arsenic measurements or 30 ml for selenium and antimony measurements.

C.5.4 Determination of arsenic with sodium borohydride

Add 5 ml of hydrochloric acid (C.3.7) to 50 ml of the digested standard solution or test solution in a 250 ml beaker (see Figure C.1) and mix. Add 5 ml sodium iodide pre-reductant solution (C.3.2), mix and wait 30 min.

NOTE 1 The sodium iodide has not been found necessary for certain hydride reaction cell designs if a 20 % to 30 % loss in instrument sensitivity is not important and variables of solution acid conditions, temperatures, and volumes for production of As(V) and arsine can be strictly controlled. This can require an automated delivery system.

Attach one beaker at a first time to the rubber stopper containing the gas dispersion tube for the purging gas, the sodium borohydride reagent inlet, and the outlet to the atomiser. Turn on a strip-chart recorder and wait until the base line is established by the purging gas and all air is expelled from the reaction cell. Add 0,5 ml of sodium borohydride reagent (C.3.1). After the instrument absorbance has reached a maximum and returned to the base line, remove the beaker, rinse the dispersion tube with water, and proceed to the next test solution or standard solution. Periodically compare the standard As(III) and As(V) curves for response consistency.

NOTE Repeatability conditions are conditions where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

z is the mean of the two results, expressed in mass fraction in %.

where

r = 0,001 z

The absolute difference between two single test results, obtained under repeatability conditions, shall not be greater than the repeatability value, r, as calculated from the following formula:

Determine the calibration curve by plotting peak heights of standard solutions versus concentration. Measure peak heights of samples and read concentrations from the calibration curve. If sample was diluted (or concentrated) before sample digestion, apply an appropriate factor.

C.6 Calculation

To 30 ml of the digested standard solution of the test solution, or to 30 ml of the undigested standard solution, or the test solution in a 250 ml beaker, add 15 ml of hydrochloric acid (C.3.7) and mix. Heat for a predetermined period (between 5 min and 60 min) at a temperature between 90 °C to 100 °C. After the predigestion of Sb, attach beakers, one at a time, to the purge apparatus. For each, turn on the strip-chart recorder and wait until the base line is established. Add 0,50 ml sodium borohydride reagent (C.3.1). After the instrument absorbance has reached a maximum and returned to the base line, remove beaker, rinse dispersion tube with water and proceed to the next test solution or standard solution. Check for a presence of chemical interferences that suppress antimony hydride instrument response by treating a digested sample with 10 µg/l Sb. Average recoveries shall be not less than 90 %.

C.5.6 Determination of antimony with sodium borohydride

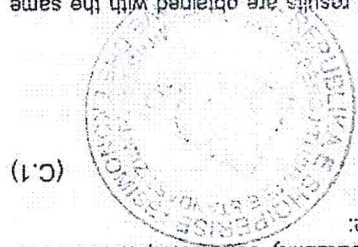
NOTE See also bibliographic reference [5].

Add 15 ml of hydrochloric acid (C.3.7) to 30 ml of the digested standard solution or test solution, or to 30 ml of the undigested standard, or the sample in a 250 ml beaker, and mix. Heat for a predetermined period at a temperature between 90 °C to 100 °C. Alternatively, autoclave at 121 °C in capped containers for 60 min, or heat for a predetermined time in open test tubes using a 90 °C to 100 °C hot water bath or an aluminium block digester. Check the effectiveness of the selected heating by demonstrating equal instrument responses for calibration curves prepared either from standard Se(IV) or from Se(VI) solutions. Effective heat exposure for converting Se(VI) to Se(IV), with no loss of Se(IV), ranges between 5 min to 60 min when open beakers or test tubes are used. Do not digest standard Se(IV) and Se(VI) solutions used for this check of equivalency. After predigestion of Se(VI) and Se(IV), attach beakers, one at a time, to the purge apparatus. For each, turn on the strip-chart recorder and wait until the base line is established. Add 0,50 ml sodium borohydride reagent (C.3.1). After the instrument absorbance has reached a maximum and returned to the base line, remove beaker, rinse dispersion tube with water and proceed to the next test solution or standard solution. Check for presence of chemical interferences that suppress selenium hydride instrument response by treating a digested sample with 10 µg/l Se(VI). Average recoveries shall be not less than 90 %.

C.5.5 Determination of selenium with sodium borohydride

NOTE 2 See also bibliographic reference [4].

Check for the presence of chemical interferences that suppress instrument response for arsenic by treating a digested sample with 10 µg/l As(III) or As(V) as appropriate. Average recoveries shall be not less than 90 %.



(C.1)

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Annex D (normative)

Determination of bromate ion content in sodium hypochlorite by liquid chromatography of ions and UV detection

D.1 General

This method specifies a method for determining dissolved bromate (BrO_3^-) in sodium hypochlorite in the measurement range of 2 mg/l to 1 000 mg/l.

This method does not require any sample pre-treatment.

The rate of sample dilution avoids all problems of saturation of the column ion capacity as well as an excessive depletion of the reduction capacity of the post-column reactant.

D.2 Interferences

The presence of chlorite concentration found in sodium hypochlorite does not interfere with the bromate determination.

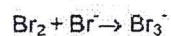
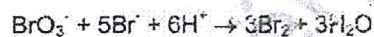
No anion normally found in sodium hypochlorite hinders the bromate determination.

D.3 Principle

A sample of sodium hypochlorite is diluted 10 000 times.

The sample is eluted through an analytical column using a sodium carbonate solution.

The eluted bromate ion is converted into tribromide ion (Br_3^-) by a post-column reaction with bromide and nitrite under acidic condition at a temperature of 60 °C.



Determination of the tribromide ion is carried out using a UV spectrometric detection at 267 nm.

This device is added in series to a classic ion chromatography system.

D.4 Reagents

All reagents shall be of a recognised analytical grade and the water used shall conform to grade 1 in accordance with EN ISO 3696:1995.

D.4.1 Potassium bromate KBrO_3

D.4.2 Sulfuric acid, H_2SO_4 , 18 mol/l

Store the solution at 2 °C to 6 °C in polyethylene or glass bottles and renew it every 6 months.
 Dissolve 53,6 g of anhydrous sodium carbonate in 800 ml of water in a 1 000 ml volumetric flask and make up to volume with water.

D.4.13 Eluent solution carbonate c(CO₃²⁻) = 0,5 mol/l

Prepare on the day of use.

Mix and make up the volume with water.

Pipette 4 ml of the nitrite solution into the same volumetric flask.

Dissolve 102,9 g of sodium bromide into 500 ml of water in a 2 l volumetric flask.

D.4.12 Post-reaction solution c(Br⁻) = 0,5 mol/l, c(NO₂⁻) = 0,0 0043 mol/l

Prepare on the day of use.

Dissolve 1,5 g of sodium nitrite in 80 ml of water in a 100 ml volumetric flask and make up the volume with water.

D.4.11 Nitrite solution at 10,0 g/l

Make up to the mark with water.

Carefully mix 210 ml of sulfuric acid with water in a 5 l volumetric flask.

D.4.10 Post-reaction chemical acid solution, c(H₂SO₄) = 6,750 mol/l

Mix 7,0 ml sulfuric acid with 5 l water.

D.4.9 Regenerant solution for the suppressor, c(H₂SO₄) = 0,025 mol/l

Store the solution at 2 °C to 6 °C in polyethylene or glass bottles and renew it every 3 months.

Make up to the mark with water.

Pipette 1,00 ml of standard stock solution (D.4.7) into a 100 ml volumetric flask.

D.4.8 Bromate standard solution, 10 mg/l bromate

Store the solution at 2 °C to 6 °C in polyethylene or glass bottles and renew it every 12 months.

Dissolve (1,3 057 ± 0,01) g of the dried potassium bromate in 800 ml of water in a 1 000 ml volumetric flask and make up the volume with water.

Dry 1,5 g of potassium bromate for 1 h at 105 °C. Store in a desiccator.

D.4.7 Standard stock solution 1 000 mg/l bromate

D.4.6 Helium 99,999 %

D.4.5 Sodium nitrite, NaNO₂

D.4.4 Sodium bromide, NaBr

D.4.3 Sodium carbonate, Na₂CO₃

D.4.14 Eluent solution carbonate $c(\text{CO}_3^{2-}) = 0,009 \text{ mol/l}$

Pipette 36 ml of the sodium carbonate solution 0,5 mol/l into a 2 000 ml volumetric flask and make up to volume with water.

Store the solution at 2 °C to 6 °C in polyethylene or glass bottles and renew it every week.

Filter the eluent on a sintered glass filter with a pore size of 0,45 µm.

Purge with helium before use for 5 min at a flow rate of 3 l/min.

D.5 Apparatus

Ordinary laboratory apparatus and glassware, together with the following.

D.5.1 Ion chromatography system

- Eluent reservoir;
- pump for isocratic technique;
- sample delivery device, including a sample injection system incorporating a sample loop of appropriate volume (e.g. 750 µl);
- column switching valves including a device for timing and controlling the valves and pump;
- separator column high capacity, specially designed for determining oxyhalides;
- pre-column to protect the separator column;
- conductivity detector with an anion suppressor device assembly (detector not in use for determining bromate), UV detector.

D.5.2 Post column device

- Reagent mix reservoir and pump to feed the reactor through a chemical suppressor;
- post column reactor;
- UV detector (267 nm).

D.6 Procedure**D.6.1 Preparation of calibration solutions**

The calibration range is dependent on the expected bromate concentration in the sample. In most cases the concentration in sodium hypochlorite is less than 1 000 mg/l.

Prepare the calibration solutions, for example according to Table D.1:

Table D.1 — Calibration solutions for determination of bromate content

Solution	BrO ₃ ⁻ in µg/l	NaBrO ₃ in µg/l
1	10	11,8
2	20	23,6
3	40	47,19
4	60	70,78
5	80	94,37
6	100	118

Prepare the calibration solutions on the day of use.

D.6.2 Preparation of test solutions

Due to the extreme sensitivity of the method, the sample shall be diluted 10 000 times.

If the concentration of the sodium hypochlorite exceeds 150 g/l chlorine, use an extra two times dilution to avoid an excessive consumption of the bromide nitrite reagent.

D.6.3 Measurement of calibration and test solutions

Measure each calibration solution three times; for each solution, the relative repeatability standard deviation shall be lower than 0,5 %.

Measure each test solution twice in order to keep the relative repeatability standard deviation at no more than 0,5 %.

D.6.4 Expression of results

The bromate content of the test solution is obtained from the regression line obtained with the calibration results in the sodium hypochlorite solution.

The sodium bromate (NaBrO₃) content of the laboratory sample, C₉, expressed in mg/l is given by

Formula (D.1):

$$C_9 = yd$$

where

y is the concentration (in mg/l NaBrO₃) obtained from the calibration curve;

d is the dilution factor of the sample to be analysed.

The sodium bromate (NaBrO₃) content, C₉, expressed in mass fraction in % of available chlorine is given by

Formula (D.2):

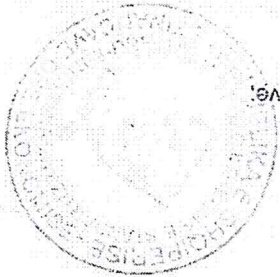
$$C_9 = \frac{C_8 \times 10}{C_7}$$

where

C₇ is the available chlorine (Cl₂) content expressed in g/l (C.2.1.6),

(D.2)

(D.1)



D.6.5 Repeatability limit

The absolute difference between two single test results obtained under repeatability conditions shall not be greater than the repeatability value, r , as calculated from Formula (D.3):

$$r = 0,005 z \quad (D.3)$$

where

z is the mean of the two results, expressed in % (m/m).

NOTE Repeatability conditions are conditions where mutually independent test results are obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within short intervals of time.

Annex E
(informative)

Results of inter-laboratory tests on sodium bromate determination in sodium hypochlorite commercial solutions

An inter-laboratory test was carried out in 2002 with laboratories from France, Belgium, Norway and Netherlands. The sodium bromate content has been determined by ion chromatographic techniques (with conductimetric or UV detection).

Sample matrix is described in Table E.1 (see also bibliographic reference [6]).

The statistical treatment of results, according to ISO 5725-2, is described in Table E.2.

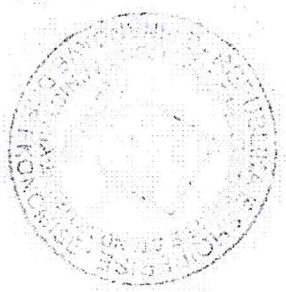
Table E.1 — Description of samples matrix

Sample #	Active chlorine (g/l)	Sodium chloride (g/l)	Sodium bromate (mg/l)
1	≈ 160	130 to 140	blank
2	≈ 160	130 to 140	500 (spike)
3	≈ 160	130 to 140	1 180 (spike)

Table E.2 — Statistical treatment for sodium bromate determination

Sample	n	K ₁	X _{int}	s _R	s _r	R _r	K ₂	n	number of analytical values (without outliers)	number of participating laboratories	K ₃	X _{int}	s _R	s _r	R _r	K ₄	X _{int}	s _R	s _r	R _r	K ₅	X _{int}	s _R	s _r	R _r	K ₆	X _{int}	s _R	s _r	R _r	K ₇	X _{int}	s _R	s _r	R _r
1	27	10	3,6	blank	35,9	-	9,4	24	3,1	7,9	2	42	16	8,7	500	620	98,4	26	4,4	11,8	2	3	42	15	8,7	1 180	1 217	99,8	63,7	5,4	20	1,7			

n: number of analytical values (without outliers)
 l: number of participating laboratories
 K₁: percentage of outliers
 X_{int}: theoretical bromate content of the sample
 X: mean of inter-laboratory test
 R_r: recovery
 s_R: reproducibility standard deviation
 s_r: reproducibility relative standard deviation
 R_r: repeatability relative standard deviation
 s_r: repeatability standard deviation
 K₂: repeatability relative standard deviation



Annex F (informative)

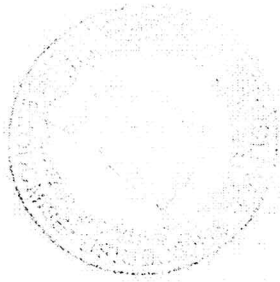
Environmental, health and safety precautions within chemical laboratories

When preparing the analytical methods for application of this document, consideration was given to minimising environmental impacts caused by using these methods of analysis.

It is the users' responsibility to use safe and suitable techniques when handling materials in the methods of analysis specified in this document.

The following list is not exhaustive but users of the analytical methods referred in this document may use it as a guide to the use of safe and proper techniques. They should:

- investigate if European Directives, transposed European legislation and national laws, regulations and administrative provisions apply;
- consult manufacturers/suppliers for specific details such as material safety data sheets and other recommendations;
- use safety equipment and wear protective clothing (usually goggles and coats), appropriate for the test product and the test chemicals, in all laboratory areas, to ensure the safety of the operator;
- be careful about flammable materials and substances that are toxic and/or human carcinogens and generally take care during transportation, decanting, diluting and dealing with spillages;
- use a fume cupboard during preparation of organic solvent solutions;
- store, handle and dispose of chemicals in a safe and environmentally satisfactory manner: including chemicals for laboratory test, test specimens, unused solvents and reagents that have to be disposed of.



- [1] Directive 98/8/EC of the European Parliament and of the Council on the placing on the market of biocidal products
- [2] 98/83/EC, Council Directive of 3rd November 1998 relating to the Quality of Water intended for Human Consumption
- [3] 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)
- [4] EN ISO 11969, Water quality — Determination of arsenic — Atomic absorption spectrometric method (hydride technique) (ISO 11969)
- [5] ISO 9965, Water quality — Determination of selenium — Atomic absorption spectrometric method (hydride technique)
- [6] ISO 5725-2, Accuracy (trueness and precision) of measurement results and results — Part 2: Basic method for the determination of repeatability and reproducibility of standard measurement method
- [7] EN 14905, Chemicals used for treatment of water intended for human consumption — Sodium chloride for on site electrochlorination using non-membrane technology

EN 901:2013 (5)