

Water quality — Determination of epichlorohydrin

The European Standard EN 14207:2003 has the status of a
British Standard

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National foreword

This British Standard is the official English language version of EN 14207:2002.

The UK participation in its preparation was entrusted by Technical Committee EH/3, Water quality, to Subcommittee EH/3/2, Physical chemical & biochemical methods, which has the responsibility to:

- aid enquirers to understand the text;
- present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- monitor related international and European developments and promulgate them in the UK.

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

This is one of a series of standards on water quality, others of which have been, or will be, published as Sections of BS 6068. The various Sections of BS 6068 are comprised within Parts 1 to 7, which, together with Part 0, are listed below.

- Part 0 *Introduction*
- Part 1 *Glossary*
- Part 2 *Physical, chemical and biochemical methods*
- Part 3 *Radiological methods*
- Part 4 *Microbiological methods*
- Part 5 *Biological methods*
- Part 6 *Sampling*
- Part 7 *Precision and accuracy*

NOTE The tests described in this British Standard should only be carried out by suitably qualified persons with an appropriate level of chemical expertise. Standard chemical procedures should be followed throughout.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the *BSI Catalogue* under the section entitled “International Standards Correspondence Index”, or by using the “Search” facility of the *BSI Electronic Catalogue* or of British Standards Online.

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Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 18, an inside back cover and a back cover.

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English version

Water quality - Determination of epichlorohydrin

Qualité de l'eau - Dosage de l'épichlorhydrine

Wasserbeschaffenheit - Bestimmung von Epichlorhydrin

This European Standard was approved by CEN on 17 January 2003.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

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Foreword

This document EN 14207:2003 has been prepared by Technical Committee CEN/TC 230 “Water analysis”, the secretariat of which is held by DIN.

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 October 2003, and conflicting national standards shall be withdrawn at the latest by October 2003.

Annexes A, B and C are informative.

WARNING — Persons using this standard should be familiar with normal laboratory practice. This standard does not propose to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard : Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

It is highly recommended that the test described in this standard be carried out by suitably qualified staff.

It should be investigated whether and to what extent particular problems will require the specification of additional marginal conditions.

1 Scope

This European Standard specifies a method for the determination of epichlorohydrin in drinking water and water used for drinking water processing. According to the given procedure, the limit of determination in routine analysis is about 0,5 µg/l ¹⁾. The limit of determination can be lowered to monitor 0,1 µg/l.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 25667-1, *Water quality — Sampling — Part 1: Guidance on the design of sampling programmes (ISO 5667-1:1980)*.

EN 25667-2, *Water quality — Sampling — Part 2: Guidance on sampling techniques (ISO 5667-2:1991)*.

1) This value was checked in an interlaboratory trial.

EN ISO 5667-3, *Water quality — Sampling — Part 3: Guidance on the preservation and handling of samples (ISO 5667-3:1994)*.

ISO 5667-5, *Water quality — Sampling — Part 5: Guidance on sampling of drinking water and water used for food and beverage processing*.

ISO 8466-1, *Water quality - Calibration and evaluation of analytical methods and estimation of performance characteristics - Part 1: Statistical evaluation of the linear calibration function*.

3 Principle

Solid phase extraction of epichlorohydrin from the drinking water sample followed by gas chromatography using a mass spectrometer (MS) as detector. Alternatively, an electron capture detector (ECD) can be used.

4 Interferences / Losses

4.1 Interferences during sampling

In order to avoid interferences, withdraw the sample according to clause 7, taking into account the information given in EN 25667-1, EN 25667-2 and EN ISO 5667-3.

In order to avoid losses due to the easy decomposition of epichlorohydrin, avoid unnecessary storage and analyse the sample as soon as possible after sampling. If storage is unavoidable, store between 2 °C and 5 °C until sample pretreatment.

4.2 Interferences during enrichment

The commercially available adsorbance materials are often of varying quality. Considerable batch-to-batch differences in quality and selectivity of these materials are possible. Perform calibration and analysis with one and the same batch of material. Make sure to avoid any losses when removing the residual water in the adsorbance material (8.1.2).

4.3 Interferences in the gas chromatography and mass spectrometry

Set the operational conditions in accordance with the manufacturer's instructions. Check these settings at regular intervals.

General interferences, caused by the injection system can be eliminated with the help of special laboratory experience and the instruments manuals.

The stability of the analytical system should be checked (for example by application of a measuring standard).

5 Reagents

Use reagents of the reagent grade “for residual analysis” or equivalent as far as available. Impurities in the reagents and in the water contributing to the blank shall be negligibly low. Check the blank regularly, especially prior to the use of a new batch.

5.1 Water

Use double-distilled water or water of comparable purity.

5.2 Operating gases for the gas chromatography / mass spectrometry / ECD, according to the manufacturer's instructions. The operating gases shall be of high purity.

5.3 Nitrogen, high purity, minimum 99,996 % (v/v), for removal of water in the sorbent packing after sample extraction.

5.4 Solvents

5.4.1 Diisopropyl ether, $C_6H_{14}O$.

5.4.2 Methanol, CH_3OH , as conditioning agent.

5.5 Reducing agents, e.g. sodium thiosulfate ($Na_2S_2O_3$).

5.6 Epichlorohydrin stock solution

Weigh 50 mg of epichlorohydrin (C_3H_5ClO) into a 100-ml volumetric flask containing diisopropyl ether (5.4.1) up to the neck and fill to mark with diisopropyl ether (5.4.1). Store the solution in a refrigerator between 2 °C and 5 °C. The shelf-life of the solution is limited (about 6 months). Check the concentration prior to analysis in order to make sure that no significant differences arise.

5.7 Internal standard stock solution

The internal standard shall not be present in the sample itself.

5.7.1 $^{13}C_3$ -epichlorohydrin stock solution for GC-MS

This solution may be purchased as certified solution (e.g. at a concentration of 100 µg/ml in nonane) or prepared from pure standard material according to 5.6. Never add more than 100 µl of a diluted internal standard stock solution in diisopropyl ether (5.4.1) to 100 ml of the water sample, a greater volume may result in poor recovery.

NOTE The peak area of the internal standard should be equivalent to that of 1 µg/l of the analyte. For example, if the concentration of the stock solution is 100 µg/ml of $^{13}C_3$ -epichlorohydrin in nonane, 100 µl, dissolved in 1 ml diisopropyl ether (5.4.1) is required to produce a 10 µg/ml spiking solution from which a volume of 10 µl can be injected directly into 100 ml of water (5.1).

5.7.2 Ethyl 2-chloropropionate ($C_5H_9ClO_2$) stock solution for GC-ECD (see 5.6)

5.8 Epichlorohydrin spiking solutions

Using stock standard solution (5.6), prepare spiking solutions by appropriate dilution in a 100-ml volumetric flask containing diisopropyl ether (5.4.1). Prepare the spiking solutions at concentrations such that the aqueous calibration solutions (5.9) will cover the working range of the analytical system. Store spiking solutions in a refrigerator between 2 °C and 5 °C. Let the solutions adjust to room temperature before preparing calibration solutions. The storage time shall not exceed one month.

5.9 Epichlorohydrin calibration solutions for the multipoint calibration

Prepare aqueous calibration solutions from the spiking solutions (5.8) by injection of an appropriate volume (e.g. 10 µl) of the spiking solution directly into 100 ml of water (5.1). However, do not use more than 100 µl of a spiking solution to produce the calibration solutions. Mix the aqueous calibration solutions thoroughly by inverting the flask several times. Prepare five different concentration levels. Prepare calibration solutions fresh daily.

Table 1 gives an example for a dilution scheme.