BS EN 16801:2016

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Foodstuffs — Determination of elements and their chemical species — Determination of methylmercury in foodstuffs of marine origin by isotope dilution GC-ICP-MS

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee AW/275, Food analysis - Horizontal methods.

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

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Published by BSI Standards Limited 2016

ISBN 978 0 580 79937 2

ICS 67.120.30

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 April 2016.

Amendments/corrigenda issued since publication

Date	Text affected

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 16801

March 2016

ICS 67.120.30

English Version

Foodstuffs - Determination of elements and their chemical species - Determination of methylmercury in foodstuffs of marine origin by isotope dilution GC-ICP-MS

Produits alimentaires - Détermination des éléments et de leurs espèces chimiques - Détermination de la teneur en méthylmercure dans les produits alimentaires d'origine marine par dilution isotopique CG-ICP-SM

Lebensmittel - Bestimmung von Elementen und ihren Verbindungen - Bestimmung von Methylquecksilber in Lebensmitteln marinen Ursprungs mit Isotopenverdünnung GC-ICP-MS

This European Standard was approved by CEN on 8 February 2016.

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Ref. No. EN 16801:2016 E

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European foreword

This document (EN 16801:2016) has been prepared by Technical Committee CEN/TC 275 “Food analysis - Horizontal methods”, 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of an identical text or by endorsement, at the latest by September 2016, and conflicting national standards shall be withdrawn at the latest by September 2016.

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1 Scope

This European Standard describes a method for the determination of monomethylmercury (MMHg) in foodstuffs of marine origin. The method has been validated in an interlaboratory test on mussel tissue, squid muscle, crab claw muscle, dog fish liver, whale meat, cod muscle and Greenland halibut muscle (all freeze-dried) with mass fractions from 0,04 mg/kg to 3,6 mg/kg dry weight according to ISO 5725-2 [1].

Laboratory experiences have shown that this method is also applicable on fresh samples [2].

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 13804, *Foodstuffs — Determination of elements and their chemical species — General considerations and specific requirements*

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

3 Principle

The sample is spiked with an appropriate amount of Hg-isotope enriched MMHg and digested using tetramethylammonium hydroxide (TMAH). After pH adjustment, derivatisation and extraction, the organic phase is analysed using GC-ICP-MS. The GC separates the different mercury species before the derivatised species (ethylmethylmercury) is atomised and ionised in the high temperature by the ICP. The ions are extracted from the plasma by a set of sampler and skimmer cones and transferred to a mass spectrometer where the ions are separated by their mass/charge ratio and determined by a pulse-count and/or analogue detector. The result is calculated using the isotope dilution equation.

WARNING — The use of this method may involve hazardous materials, operations and equipment. This method does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

4 Reagents

4.1 General

The concentration of mercury species in the reagents and water used shall be low enough to not affect the results of the determination. When using a method of high sensitivity like ICP-MS, the control of the blank levels of water, acid and other reagents is very important. Generally ultra-pure water complying with ISO 3696 grade 1 (i.e. electrical conductivity below 0,1 µS/cm at 25 °C) and acid of high purity is recommended, e.g. cleaned by sub-boiling distillation. Reagents should be of minimum p.a. quality where possible. Special facilities can be used in order to avoid contamination during the steps of preparation and measurement (e.g. uses of laminar flow benches or comparable clean room facilities).

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