INTERNATIONAL STANDARD

ISO 21863

First edition 2020-11

Water quality — Determination of alkylmercury compounds in water — Method using gas chromatographymass spectrometry (GC-MS) after phenylation and solvent extraction

Qualité de l'eau — Détermination des composés alkyl mercure dans l'eau — Méthode par chromatographie gazeuse et spectrométrie de masse (CG-SM) après phénylation et extraction par solvant





COPYRIGHT PROTECTED DOCUMENT

© ISO 2020

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office CP 401 • Ch. de Blandonnet 8 CH-1214 Vernier, Geneva Phone: +41 22 749 01 11 Email: copyright@iso.org Website: www.iso.org

Published in Switzerland

Contents			Page
Forev	word		iv
Intro	ductio	n	v
1	Scop	е	1
2	Norn	native references	1
3	Terms and definitions		
4		rinciple	
5	Interferences		
J	5.1 5.2	Interferences with sampling, sample storage and sample preparation Interferences with GC-MS	2
6	Reag	leagents and standards 2	
7	Appa	ratus and materials	4
8	Samı	ole collection, preservation and storage	5
9	Procedure		
	9.1	Sample preparation	5
		9.1.1 pH-adjustment of water sample	
		9.1.2 Phenylation and solvent extraction	
	9.2	Preparation of samples for GC-MS	
	9.3	Optimization of operating condition for GC-MS	6
	9.4	Identification of individual substances with GC-MS	6
	9.5	Blank tests	7
10	Calibration		
	10.1	General requirements	
	10.2	Performance test of GC-MS	
	10.3	Calibration with internal standard 10.3.1 General requirement	
		10.3.2 Procedure of calibration	
	10.4	Spike recovery test of target substances	
11	Calculation		9
	11.1	Calculation of results after calibration with internal standards	
	11.2	Treatment of results lying outside the calibration range	
	11.3	Quality checks for internal standardization	
12	_	Expression of results10	
13	Test	report	11
Anne	x A (in	formative) Example of operating condition of GC-MS	12
Anne	x B (in: alkyl	formative) Examples of mass chromatograms and mass spectra of phenylated mercury by GC-MS	13
Anne	nex C (informative) The use of sodium tetrapropylborate as an alternative derivatizing agent ^[4]		
Anne	x D (in	formative) The use of GC-AFS as an alternative detector	17
Anne	x E (inf	Formative) Performance data	20
Bibliography			

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 147, *Water quality*, Subcommittee SC 2, *Physical, chemical and biochemical methods*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document specifies a method for the determination of alkylmercury compounds in water by gas chromatography-mass spectrometry (GC-MS) after phenylation and solvent extraction.

Alkylmercury has high toxicity that causes Minamata disease in the heavy exposure as discovered at Minamata City in Japan in 1956. Methylmercury in wastewater from an acetaldehyde acetic acid manufacturing plant was identified as a causative substance. Subsequent investigation revealed that ethylmercury poisoning has a similar toxic effect as methylmercury. Japanese government set an effluent standard and an environment standard for alkylmercury.

Minamata Convention on Mercury was adopted by over 140 countries in 2013 for prevention of global environmental pollution and health damage caused by mercury, and entered into force in 2017. The convention states that each party shall identify the relevant point source categories and take measures including the set of release limit values and the use of best available techniques and best environmental practices. It should be noted that the released inorganic mercury is partially converted to alkylmercury by biochemical processes of microorganism in water and sediment. Alkylmercury is concentrated in biota through food chain, and consequently the risk to higher organism increases.

This document will be beneficial to evaluate the risk of alkylmercury from water and to control the anthropogenic releases of alkylmercury from the relevant point sources.