

---

---

**Water quality — Determination of  
alkylmercury compounds in water —  
Method using gas chromatography-  
mass 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](mailto:copyright@iso.org)  
Website: [www.iso.org](http://www.iso.org)

Published in Switzerland

# Contents

Page

Foreword	iv
Introduction	v
<b>1 Scope</b>	<b>1</b>
<b>2 Normative references</b>	<b>1</b>
<b>3 Terms and definitions</b>	<b>1</b>
<b>4 Principle</b>	<b>2</b>
<b>5 Interferences</b>	<b>2</b>
5.1 Interferences with sampling, sample storage and sample preparation	2
5.2 Interferences with GC-MS	2
<b>6 Reagents and standards</b>	<b>2</b>
<b>7 Apparatus and materials</b>	<b>4</b>
<b>8 Sample collection, preservation and storage</b>	<b>5</b>
<b>9 Procedure</b>	<b>5</b>
9.1 Sample preparation	5
9.1.1 pH-adjustment of water sample	5
9.1.2 Phenylation and solvent extraction	6
9.1.3 Dehydration of toluene extract	6
9.2 Preparation of samples for GC-MS	6
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
<b>10 Calibration</b>	<b>7</b>
10.1 General requirements	7
10.2 Performance test of GC-MS	7
10.3 Calibration with internal standard	8
10.3.1 General requirement	8
10.3.2 Procedure of calibration	8
10.4 Spike recovery test of target substances	9
<b>11 Calculation</b>	<b>9</b>
11.1 Calculation of results after calibration with internal standards	9
11.2 Treatment of results lying outside the calibration range	10
11.3 Quality checks for internal standardization	10
<b>12 Expression of results</b>	<b>10</b>
<b>13 Test report</b>	<b>11</b>
<b>Annex A (informative) Example of operating condition of GC-MS</b>	<b>12</b>
<b>Annex B (informative) Examples of mass chromatograms and mass spectra of phenylated alkylmercury by GC-MS</b>	<b>13</b>
<b>Annex C (informative) The use of sodium tetrapropylborate as an alternative derivatizing agent<sup>[4]</sup></b>	<b>16</b>
<b>Annex D (informative) The use of GC-AFS as an alternative detector</b>	<b>17</b>
<b>Annex E (informative) Performance data</b>	<b>20</b>
<b>Bibliography</b>	<b>21</b>