# INTERNATIONAL STANDARD

ISO 16308

First edition 2014-09-15

# Water quality — Determination of glyphosate and AMPA — Method using high performance liquid chromatography (HPLC) with tandem mass spectrometric detection

Qualité de l'eau — Détermination du glyphosate et de l'AMPA — Méthode par chromatographie en phase liquide à haute performance (CLHP) avec détection par spectrométrie de masse en tandem





# **COPYRIGHT PROTECTED DOCUMENT**

© ISO 2014

All rights reserved. Unless otherwise specified, 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 Case postale 56 • CH-1211 Geneva 20 Tel. + 41 22 749 01 11 Fax + 41 22 749 09 47 E-mail copyright@iso.org Web www.iso.org

Published in Switzerland

Contents		Page
Fore	eword	iv
Introduction		v
1	Scope	1
2	Normative references	1
3	Principle	1
4	Interferences	2
5	Reagents	2
6	Apparatus	4
7	Sampling	5
8	Procedure	
	<ul> <li>8.1 Pre-treatment (Suspended particular matter)</li> <li>8.2 Chelate break and derivatization</li> <li>8.3 Pre-concentration</li> <li>8.4 Chromatographic determination</li> <li>8.5 Identification and confirmation of the analytes</li> <li>8.6 Blank control monitoring</li> </ul>	
9	Calibration	
	9.1 Concentration ranges	
	9.3 Internal standard calibration	
10	Expression of results	10
11	Test report	10
Ann	nex A (informative) Performance data	11
Ann	nex B (informative) Examples of chromatographic conditions	14
Annex C (informative) Examples of chromatograms		15
Ann	nex D (informative) Analysis of gluphosinate	16
	nex E (informative) Pre-treatment of hard water samples	
Rihl	lingraphy	22

### **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 on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 147, Water quality, Subcommittee SC 2, Physical, chemical and biochemical methods.

## Introduction

Glyphosate [N-(phosphonomethyl)glycine] is a non-selective broad-spectrum herbicide. The efficiency of this compound makes it a top selling and one of the most widely used herbicides in the world since it entered the market in 1974. Together with its main degradation product, aminomethylphosphonic acid (AMPA), glyphosate is one of the most detected substances in water bodies in many developed countries. Note also that AMPA can be released during sewage treatment, e.g. due to breakdown of detergent formulations for textiles.

Glyphosate and AMPA belong to the aminophosphonate family and have specific physico-chemical properties that require the development of complex analytical methods for analysis and detection. The difficulty in analysis is mainly linked to the high solubility of glyphosate and AMPA and their chelating nature. To solve these problems, their pre-column derivatization with 9-fluorenylmethylchloroformate (FMOC-Cl) to form less polar derivatives allows a better separation using liquid chromatography.

Gluphosinate, another aminophosphonate, is less commonly subject to regulation and can be determined simultaneously, provided it can be demonstrated that there is no interference with the sample under analysis.

There is currently an International Standard for the determination by liquid chromatography and fluorometric detection; however, the determination by HPLC–ESI–MS/MS can be much more specific (unambiguous identification) and more sensitive (limits of quantification of approximately 30 ng/l for both glyphosate and AMPA). This International Standard is based on this analytical technique and is intended for laboratories involved in the regulatory control of the aquatic environment. Many such laboratories are now equipped with this kind of apparatus.