



Approved as an American National Standard
ANSI Approval Date: February 11, 2022

ANSI/NEMA 62321-3-2-2020

*Determination of Certain Substances in Electrotechnical Products
Part 3-2: Screening—Fluorine, Bromine and Chlorine in Polymer and Electronics by
Combustion-Ion Chromatography (C-IC)*

Published by:

National Electrical Manufacturers Association

1300 North 17th Street, Suite 900
Rosslyn, Virginia 22209

www.nema.org

© 2022 National Electrical Manufacturers Association. All rights including translation into other languages, reserved under the Universal Copyright Convention, the Berne Convention for the Protection of Literary and Artistic Works, and the International and Pan American Copyright Conventions.

These materials are subject to copyright claims of IEC and NEMA. No part of this publication may be reproduced in any form, including an electronic retrieval system, without the prior written permission of NEMA. All requests pertaining to the ANSI/NEMA 62321-3-2-2020 standard should be submitted to NEMA.

NOTICE AND DISCLAIMER

The information in this publication was considered technically sound by the consensus of persons engaged in the development and approval of the document at the time it was developed. Consensus does not necessarily mean that there is unanimous agreement among every person participating in the development of this document.

NEMA standards and guideline publications, of which the document contained herein is one, are developed through a voluntary consensus standards development process. This process brings together volunteers and/or seeks out the views of persons who have an interest in the topic covered by this publication. While NEMA administers the process and establishes rules to promote fairness in the development of consensus, it does not write the document and it does not independently test, evaluate, or verify the accuracy or completeness of any information or the soundness of any judgments contained in its standards and guideline publications. NEMA disclaims liability for any personal injury, property, or other damages of any nature whatsoever, whether special, indirect, consequential, or compensatory, directly or indirectly resulting from the publication, use of, application, or reliance on this document.

NEMA disclaims and makes no guaranty or warranty, express or implied, as to the accuracy or completeness of any information published herein, and disclaims and makes no warranty that the information in this document will fulfill any of your particular purposes or needs. NEMA does not undertake to guarantee the performance of any individual manufacturer or seller's products or services by virtue of this standard or guide.

In publishing and making this document available, NEMA is not undertaking to render professional or other services for or on behalf of any person or entity, nor is NEMA undertaking to perform any duty owed by any person or entity to someone else. Anyone using this document should rely on his or her own independent judgment or, as appropriate, seek the advice of a competent professional in determining the exercise of reasonable care in any given circumstances. Information and other standards on the topic covered by this publication may be available from other sources, which the user may wish to consult for additional views or information not covered by this publication.

NEMA has no power, nor does it undertake to police or enforce compliance with the contents of this document. NEMA does not certify, test, or inspect products, designs, or installations for safety or health purposes. Any certification or other statement of compliance with any health or safety-related information in this document shall not be attributable to NEMA and is solely the responsibility of the certifier or maker of the statement.

CONTENTS

FOREWORD	iv
INTRODUCTION	vi
1 Scope	1
2 Normative references	2
3 Terms, definitions and abbreviated terms	2
3.1 Terms and definitions	2
3.2 Abbreviated terms	4
4 Principle	4
5 Reagents and materials	5
6 Apparatus	6
7 Sampling	7
8 Procedure	7
8.1 Combustion	7
8.2 IC analysis	8
8.3 Blank test	8
8.4 Cleaning and recalibration	8
8.5 Calibration	8
8.6 Measurement of the sample	9
8.7 Interference	9
9 Calculation	9
10 Precision	10
11 Quality assurance and control	10
11.1 General	10
11.2 Limits of detection (LOD) and limits of quantification (LOQ)	11
12 Test report	12
Annex A (informative) Screening for fluorine, chlorine, bromine and iodine in polymers and electronics by oxygen bomb combustion-ion chromatography	13
A.1 General	13
A.2 Principle	13
A.3 Reagents and materials	13
A.4 Apparatus	14
A.5 Sampling	14
A.6 Procedure	15
A.6.1 General	15
A.6.2 Choice of the absorption solution	15
A.6.3 Preparation of the bomb	16
A.6.4 Combustion	16
A.6.5 Collection of halides	16

A.6.6	Cleaning procedure	17
A.7	Ion chromatographic analysis	17
A.8	Calculation	17
A.9	Quality assurance and control and test report.....	18
Annex B (informative)	Screening for fluorine, chlorine, bromine and iodine in polymers and electronics by oxygen flask combustion-ion chromatography	19
B.1	General	19
B.2	Principle	19
B.3	Reagents and materials.....	19
B.4	Apparatus.....	19
B.5	Sampling	20
B.5.1	General.....	20
B.5.2	Solid and paste-like samples	20
B.5.3	Liquid samples	20
B.6	Procedure.....	20
B.6.1	General.....	20
B.6.2	Choice of the absorption solution	21
B.6.3	Combustion.....	21
B.7	Ion chromatographic analysis, calculation, quality assurance and control and test report.....	21
Annex C (informative)	Example of a combustion device and IC system.....	23
Annex D (informative)	Screening for iodine in polymers and electronics by combustion-ion chromatography (C-IC)	24
D.1	General	24
D.2	Principle	24
D.3	Reagents and materials.....	24
D.4	Apparatus.....	26
D.5	Sampling	27
D.6	Procedure.....	27
D.6.1	Combustion	27
D.6.2	IC analysis	27
D.6.3	Blank test.....	28
D.6.4	Cleaning and recalibration.....	28
D.6.5	Calibration.....	28
D.7	Measurement of the sample.....	28
D.8	Interference.....	29
D.9	Calculation	29
Annex E (informative)	Results of international interlaboratory study (IIS 4A and IIS 3-2)	30
Annex F (informative)	Additional results of TG 3-2 test	33
Annex G (informative)	Additional validation data.....	35
Annex H (informative)	Additional IC data	37
Bibliography	39

Figure A.1 – Example of oxygen bomb combustion device.....	18
Figure B.1 – Example of oxygen flask combustion device	21
Figure B.2 – Example of wrapping of sample	22
Figure C.1 – Example of a combustion device connected to an ion chromatograph (IC).....	23
Figure C.2 – Example of ion chromatographic system.....	23
Figure H.1 – Example of a chromatogram of the standard solution (4 mg/l of each standard) by IC.....	37
Table 1 – Tested concentration ranges for fluorine by C-IC in various materials	1
Table 2 – Tested concentration ranges for chlorine by C-IC in various materials	1
Table 3 – Tested concentration ranges for bromine by C-IC in various materials	1
Table 4 – Fluorine results of international inter-laboratory study (IIS 4A)	10
Table 5 – Chlorine results of international inter-laboratory study (IIS 4A).....	10
Table 6 – Bromine results of international inter-laboratory study (IIS4A and IIS 3-2).....	10
Table 7 – Acceptance criteria of items for quality control	11
Table 8 – Student’s t values used for calculation of method detection limit ($MDL = t \times s_{n-1}$)	12
Table E.1 – Mean results and recovery rates for bromine obtained in the IIS4A study using C-IC	30
Table E.2 – Statistical bromine data for IIS 4A results using C-IC.....	30
Table E.3 – Mean results and recovery rates for fluorine obtained in the IIS 3-2 study using C-IC	31
Table E.4 – Statistical fluorine data for IIS 3-2 results using C-IC	31
Table E.5 – Mean results and recovery rates for chlorine obtained in the IIS 3-2 study using C-IC	32
Table E.6 – Statistical for chlorine data for IIS 3-2 results using C-IC	32
Table F.1 – Mean results and recovery rates for bromine obtained in the TG 3-2 internal test study by using C-IC	33
Table F.2 – Mean results and recovery rates for bromine obtained in the TG 3-2 internal test study by using oxygen bomb-IC	34
Table G.1 – General conditions for the combustion furnace and the absorption solution	35
Table G.2 – Additional information – Difference in sample sizes and measured bromine values in solder paste with burning aid (WO_3 powder).....	35
Table G.3 – Additional information – Difference in combustion temperatures and measured bromine values in solder paste with burning aid (WO_3 powder).....	35
Table G.4 – Additional information – Difference in recovery rate of iodine according to adsorbents (H_2O_2 , hydrazine)	36
Table H.1 – Typical operating conditions for IC	37
Table H.2 – Example of fluorine calibration solutions for IC	38
Table H.3 – Example of chlorine calibration solutions for IC	38
Table H.4 – Example of bromine calibration solutions for IC	38

INTERNATIONAL ELECTROTECHNICAL COMMISSION

DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS –

Part 3-2: Screening – Fluorine, chlorine and bromine in polymers and electronics by combustion-ion chromatography (C-IC)

FOREWORD

- 1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
- 2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.
- 3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
- 4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.
- 5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
- 6) All users should ensure that they have the latest edition of this publication.
- 7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
- 8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
- 9) Attention is drawn to the possibility that some of the elements of this IEC Publication may be the subject of patent rights. IEC shall not be held responsible for identifying any or all such patent rights.

International Standard IEC 62321-3-2 has been prepared by IEC technical committee 111: Environmental standardization for electrical and electronic products and systems.

This second edition cancels and replaces the first edition published in 2013. This edition constitutes a technical revision.

This edition includes the following significant technical changes with respect to the previous edition:

- a) In the previous edition, a screening test method for bromine (Br) content only was provided. In this edition, a screening test method by C-IC for fluorine (F), chlorine (Cl) and bromine (Br) has been added to the normative part of the document.
- b) A screening test method by C-IC for iodine (I) has been added in Annex D (informative).

The text of this International Standard is based on the following documents:

FDIS	Report on voting
111/573/FDIS	111/577/RVD

Full information on the voting for the approval of this International Standard can be found in the report on voting indicated in the above table.

This document has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts in the IEC 62321 series, published under the general title *Determination of certain substances in electrotechnical products* can be found on the IEC website.

The committee has decided that the contents of this document will remain unchanged until the stability date indicated on the IEC website under "<http://webstore.iec.ch>" in the data related to the specific document. At this date, the document will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

Note: The user's attention is called to the possibility that compliance with this standard could require use of an invention covered by patent rights.

By publication of this standard, no position is taken with respect to the validity of any such claim(s) or of any patent rights in connection therewith. If a patent holder has filed a statement of willingness to grant a license under these rights on reasonable and non-discriminatory terms and conditions to applicants desiring to obtain such a license, then details may be obtained from the Secretary.

INTRODUCTION

The widespread use of electrotechnical products has drawn increased attention to their impact on the environment. In many countries all over the world this has resulted in the adaptation of regulations affecting wastes, substances and energy use of electrotechnical products.

The use of certain substances (e.g. lead (Pb), cadmium (Cd), polybrominated diphenyl ethers (PBDEs) and phthalates) in electrotechnical products is a source of concern in current and proposed regional legislation.

The purpose of the IEC 62321 series is therefore to provide test methods that will allow the electrotechnical industry to determine the levels of certain substances in electrotechnical products on a consistent global basis.

The first edition of IEC 62321-3-2 (2013) was published to address screening for total bromine.

This document (revised edition of IEC 62321-3-2) describes the test methods to quantify halogen (fluorine, chlorine and bromine) in polymers and electronics by C-IC in the normative section and to quantify iodine (I) in an informative Annex D.

In addition, information on oxygen bomb combustion-ion chromatography and oxygen flask-ion chromatography is provided in Annex A (informative) and Annex B (informative).

WARNING – Persons using this document should be familiar with normal laboratory practice. This document does not purport 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.

DETERMINATION OF CERTAIN SUBSTANCES IN ELECTROTECHNICAL PRODUCTS –

Part 3-2: Screening – Fluorine, chlorine and bromine in polymers and electronics by combustion-ion chromatography (C-IC)

1 Scope

This part of IEC 62321 specifies the screening analysis of fluorine, chlorine and bromine in polymers and electronics using combustion-ion chromatography (C-IC). A C-IC screening analysis procedure for iodine can be found in Annex D.

This test method has been evaluated for ABS (acrylonitrile butadiene styrene), EMC (epoxy moulding compound), PE (polyethylene) and PC (polycarbonate) within the concentration ranges as specified in Table 1, Table 2 and Table 3. (Detailed results are shown in Table E.1 to Table E.6, and in Annex F (Table F.1 and Table F.2).

The use of this method for other types of materials or concentration ranges outside those specified below has not been evaluated.

Table 1 – Tested concentration ranges for fluorine by C-IC in PC

Substance/element	Fluorine	
Polymer	Unit of measure mg/kg	PC
Concentration or concentration range tested		575

Table 2 – Tested concentration ranges for chlorine by C-IC in PE

Substance/element	Chlorine	
Polymer	Unit of measure mg/kg	PE
Concentration or concentration range tested		102,2

Table 3 – Tested concentration ranges for bromine by C-IC in various materials

Substance/element	Bromine			
Polymer	Unit of measure mg/kg	ABS	EMC	PE
Concentration or concentration range tested		124 to 890	195 to 976	96

This horizontal standard is primarily intended for use by technical committees in the preparation of standards in accordance with the principles laid down in IEC Guide 108.