### **TECHNICAL SPECIFICATION**

Second edition 2020-02

# Surface chemical analysis — Glow discharge mass spectrometry — **Operating procedures**

lyse i ninescen Analyse chimique des surfaces — Spectrométrie de masse à décharge



**Reference** number ISO/TS 15338:2020(E)



#### © 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 Fax: +41 22 749 09 47 Email: copyright@iso.org Website: www.iso.org

Published in Switzerland

Page

#### **Contents**

| For | eword  | iv |
|-----|--|----|
| 1   | Scope  | 1  |
| 2   | Normative references   | 1  |
| 3   | Terms and definitions  | 1  |
| 4   | Principle  |    |
| 5   | Apparatus  |    |
| 6   | Routine operations   |    |
|     | <ul><li>6.1 Cleaning the system</li><li>6.2 Support gas handling</li></ul>                       |    |
| 7   | Calibration  |    |
|     | 7.1 Mass calibration   | 7  |
|     | <ul><li>7.2 Detector calibration</li><li>7.3 Routine checks</li></ul>                            |    |
| 8   | Data acquisition   |    |
| 0   | 8.1 Sample preparation   |    |
|     | 8.2 Procedure setup  |    |
| 0   | 8.3 Data acquiring <b>Quantification</b>   |    |
| 9   | 9.1 Element integral calculation   |    |
|     | 9.2 Ion beam ratios  |    |
|     | <ul> <li>9.3 Fully quantitative analysis</li> <li>9.4 Semi quantitative analysis</li> </ul>      |    |
|     | 9.4 Semi quantitative analysis<br>9.5 Combination of semi quantitative and quantitative analysis |    |
| Bib | liography  |    |
|     |  |    |
|     |  |    |
|     |  |    |

#### 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 <a href="https://www.iso.org/directives">www.iso.org/directives</a>).

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 <a href="https://www.iso.org/patents">www.iso.org/patents</a>).

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 201, *Surface chemical analysis*, Subcommittee SC 8, *Glow discharge spectroscopy*.

This second edition cancels and replaces the first edition (ISO/TS 15338:2009), which has been technically revised.

The main changes compared to the previous edition are as follows:

- This document is more generic and covers not only the static, cryogenic cooled source, but also the fast flow high power source.
- This document no longer refers to calibration factors specific to one particular instrument type.

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 <u>www.iso.org/members.html</u>.

002 FT2

## Surface chemical analysis — Glow discharge mass spectrometry — Operating procedures

#### 1 Scope

This document gives procedures for the operation and use of glow discharge mass spectrometry (GD-MS). There are several GD-MS systems from different manufacturers in use and this document describes the differences in their operating procedures when appropriate.

NOTE This document is intended to be read in conjunction with the instrument manufacturers' manuals and recommendations.

#### 2 Normative references

There are no normative references in this document.

#### 3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <a href="https://www.iso.org/obp">https://www.iso.org/obp</a>
- IEC Electropedia: available at http://www.electropedia.org/

#### **4** Principle

In a glow discharge source, a potential difference is applied between the cathode (the sample to be analysed) and the anode, and a plasma is supported by the introduction of an inert gas, normally argon. This potential difference can be either direct current (DC) or radio frequency (RF), the advantage of RF being that electrically insulating materials can be analysed directly. Inert gas ions and fast neutrals formed within the plasma are attracted to the surface of the sample and their impact results in the production of neutrals by sputtering from surface.

These neutrals diffuse into the plasma where they are subsequently ionised within the equipotential area of the plasma and can then be extracted to a mass spectrometer for analysis. Both magnetic sector and time of flight spectrometers are available.

#### **5** Apparatus

#### 5.1 Ion source

There are two fundamental types of ion source used for the GD-MS, a low flow or "static" source, and a fast flow source. Both types can accept pin samples or samples with a flat surface. A typical pin would be 20 mm long with a diameter of 3 mm, and a typical flat sample would be 20 mm to 40 mm diameter. More details of these dimensions can be found later.

In the low flow source the plasma cell is effectively a sealed unit held within a high vacuum chamber, with a small exit slit or hole to allow the ions to exit the cell and enter the mass spectrometer. The cell body is at anode potential, the acceleration potential of the mass spectrometer, and the sample is held at cathode potential, typically 1 kV below anode potential. In this type of source, the argon flow is typically