
Surface chemical analysis — Glow discharge mass spectrometry — Operating procedures

*Analyse chimique des surfaces — Spectrométrie de masse à décharge
luminescente (GD-MS) — Introduction à l'utilisation*

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Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Principle	1
5 Apparatus	1
6 Routine operations	6
6.1 Cleaning the system.....	6
6.2 Support gas handling.....	6
7 Calibration	7
7.1 Mass calibration.....	7
7.2 Detector calibration.....	7
7.3 Routine checks.....	8
8 Data acquisition	9
8.1 Sample preparation.....	9
8.2 Procedure setup.....	9
8.3 Data acquiring.....	10
9 Quantification	10
9.1 Element integral calculation.....	10
9.2 Ion beam ratios.....	11
9.3 Fully quantitative analysis.....	11
9.4 Semi quantitative analysis.....	12
9.5 Combination of semi quantitative and quantitative analysis.....	12
Bibliography	13

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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 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 www.iso.org/members.html.

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 <https://www.iso.org/obp>
- 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.

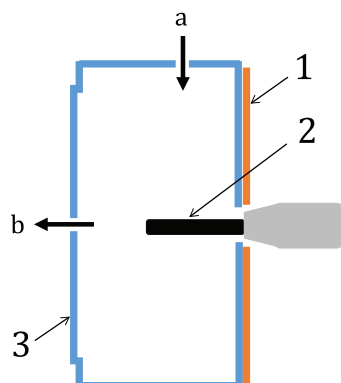
5 Apparatus

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

one sccm (standard atmosphere cubic centimetres per minute) or less, and the gas used, normally argon, should be of very high purity, six nines five or better. The power of the plasma is relatively low, typically 2 W or 3 W; the potential difference is typically 1 kV and the current 2 mA or 3 mA.



Key

- 1 insulator
- 2 sample (cathode)
- 3 anode (GD cell)
- a Gas inlet (0,3 sccm to 0,6 sccm)
- b To mass spectrometer

Figure 1 — Low flow source pin geometry

A schematic diagram of the low flow source in pin geometry is shown in [Figure 1](#). The gas is introduced into the cell through a metal pipe which forms a metal to metal seal with the cell body. On some systems an alternative of a PEEK tube with a ferrule seal to the cell body is used. The pin sample is held in a chuck which sits at cathode potential and the cell body is at anode potential, so the two are separated by an insulating disc. The chuck is actually located against a metal (tantalum) plate which also sits at cathode potential (not shown in the schematic diagram). The whole assembly forms a good gas seal while maintaining good electrical insulation. The only escape for the gas and any ions formed in the plasma is through a small slit or hole at the back of the cell, and this creates a pressure differential between the cell and the surrounding source vacuum chamber. It is normal to measure the pressure outside the cell in a low flow source rather than in the cell itself, the presence of a plasma making the measurement difficult. In this geometry, the potential difference between the anode and cathode “drops” in a small sheath approximately 1mm around the sample, thus leaving the main gas volume in the cell at the same potential. So any ions formed in the “plasma cloud” will not be electrically attracted back to the cathode.

It is standard practice in the low flow source to cool the plasma to near liquid nitrogen temperatures. This has been shown to reduce significantly the formation of molecular species associated with the matrix and plasma support gas combined as dimers or trimers, or with gas backgrounds such as hydrogen, nitrogen and oxygen. Cooling the sample in this way also allows for the measurement of low melting point materials such as gallium and indium, materials that would melt under normal plasma conditions.

Heat transfer between the components of the plasma cell needs to be considered. The whole cell assembly is floated up to the accelerating potential, so the anode will typically be around 6 kV to 8 kV while the sample (cathode) is at approximately 1 kV lower during operation. The design of the heat exchanger, or cooling assembly, means that it will be sitting at ground potential, and so it is connected to the cell insulating disc which is of a larger diameter than the cell body (not shown in [Figure 1](#)). Thus, it is necessary for the insulating disc to have a good coefficient of heat transfer at the same time as being electrically insulating; the material boron nitride is ideal for this and is used in most systems. It is important to consider heat transfer through all junctions, particularly from the cell body and cathode plate through the insulator to the heat exchanger. And in order to make the sample cold, it is