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ISO/TS 15338

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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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 8, *Glow discharge spectroscopy*.

This third edition cancels and replaces the second edition (ISO/TS 15338:2020), which has been technically revised.

The main changes are as follows:

- additional technical information have been added to the principle, apparatus and routine operations
- minor editorial changes.

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 specifies 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 terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://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, but other inert gases can be used. 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. The impacts of inert gas ions and fast neutrals formed within the plasma on the surface of the sample result in the production of neutrals by sputtering from surface.

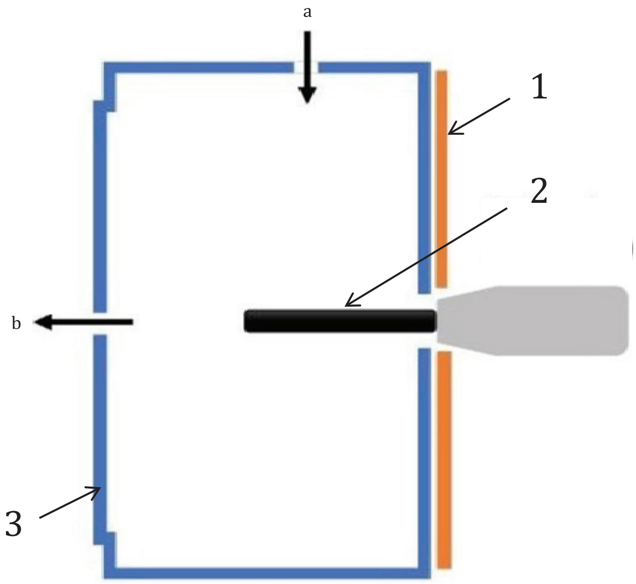
These neutrals diffuse into the plasma where they are subsequently ionised within the equipotential area of the plasma and can then be extracted to a mass spectrometer for analysis. Both magnetic sector and time of flight spectrometers are available.

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. It must be big enough to cover the hole in the chosen anode plate and provide a good gas seal. 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. If a metal pipe is used, then an insulating material must be included in the gas line as the cell is at anode potential. This is normally a piece of quartz with a very small diameter hole through which the gas passes. 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 1 mm 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