
**Surface chemical analysis — Glow
discharge mass spectrometry (GD-MS) —
Introduction to use**

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

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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.

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- an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

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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.

ISO/TS 15338 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 8, *Glow discharge spectrometry*, based on Australian Standard AS 3685:1998.

Surface chemical analysis — Glow discharge mass spectrometry (GD-MS) — Introduction to use

1 Scope

This Technical Specification gives guidelines for the operation of glow discharge mass spectrometry (GD-MS) instruments and recommendations for the use of GD-MS. It is intended to be read in conjunction with the instrument manufacturers' manuals and recommendations.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 18115, *Surface chemical analysis — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115 and the following apply.

3.1

accuracy of measurement

closeness of the agreement between a result and the accepted reference value

3.2

elemental intensity

amount of ion current recorded for a particular element

3.3

pin cell

sample cell used for the analysis of wire and rod samples

3.4

precision of measurements

closeness of the agreement between independent test results obtained under stipulated conditions, normally reported as a standard deviation

3.5

pin, rod and wire samples

samples with cylindrical or square cross-section of nominal length typically 20 mm and not normally exceeding 10 mm across

3.6

transmission

ratio of the number of ions reaching the detector relative to the number of ions entering the mass analyser

4 Safety

4.1 Use of high-voltage power supply and connection of the instrument

Electrical connection should comply with the regulations in force. Particular care should be taken to ensure that connection of the instrument to ground is correct, and the efficiency of the ground connection should be checked.

4.2 Use and storage of compressed-gas cylinders

The compressed-gas cylinders should be regularly tested by the appropriate authorities. Cylinders should not be stored or used inside the laboratory. Rather, they should be located outside the laboratory in a place that is well ventilated, away from direct heat, and accessible to service and safety personnel. The cylinders should be provided with suitable pressure-reducing valves. If more than one cylinder is to be used or stored in close proximity, it is advisable to indicate in some way which cylinder or cylinders are currently in use.

4.3 Handling of cryogenic materials

Vessels containing cryogenic materials should be located so as to minimize the risk to personnel. Areas where cryogenic liquids are stored and used should be ventilated to prevent the accumulation of gas or vapour which could evaporate from the liquid. It is good practice to keep areas where cryogenic liquids are used very clean. All transfer operations should be in accordance with statutory requirements. When a cryogenic liquid is being transferred from one vessel to another, precautions should be taken to minimize any spills and splashing. The requirements of the relevant regulatory authorities should also be met.

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5 Principle

In a glow discharge source, electrical power is supplied between the sample (cathode) and the anode by a power supply typically operated in direct current (dc) at 0,5 kV to 2 kV and 1 mA to 300 mA. Argon (or another inert gas such as neon, krypton or helium) is introduced into the discharge cell. The pressure inside the discharge cell is typically a few hundred pascals (Pa). A potential difference is applied between the cathode and the anode and a glow discharge (plasma) is established. Sample material (single atoms and/or clusters) which is sputtered by ions and neutrals diffuses into the plasma.

Ions formed in the glow discharge are extracted from the ion source and pass into a mass analyser. The mass analyser is used to transmit ions of given mass-to-charge ratio to the detector(s). The ions reaching the detector(s) are measured as ion current or counted by a counting system. This information is stored in a computer system. Elemental mass fractions are typically calculated by the instrument software using the ion currents of isotopes, by normalizing the signal to the signal of a matrix element and subsequently comparing the normalized signals with those arising from the corresponding elements in calibration samples.

6 Materials

6.1 Deionized water, 18 M Ω -cm or better.

6.2 Argon gas, or other plasma support gases, of purity better than 99,999 9 % or in accordance with the recommendations of the instrument manufacturer.

6.3 Liquid nitrogen, for cryogenic cooling of the discharge cell.

6.4 Compressed gas, to operate pneumatic valves.

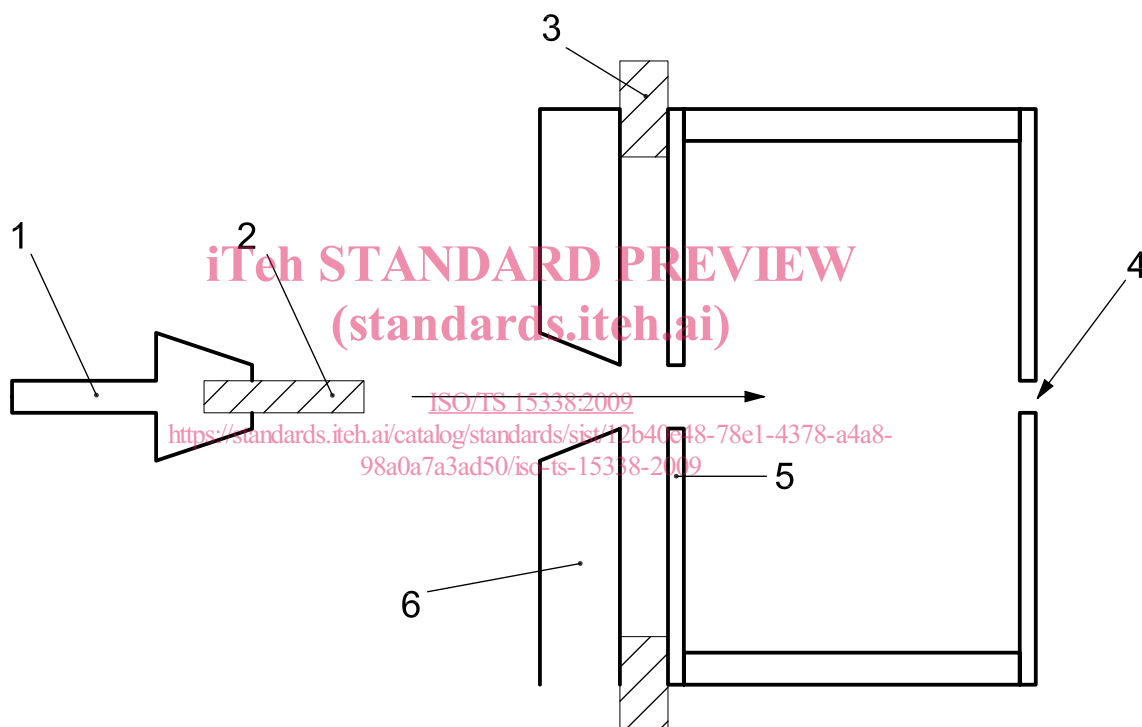
7 Apparatus

7.1 Ion source

7.1.1 General

A glow discharge ion source consists of a glow discharge cell and a power supply. In some designs, the ion source will also contain a series of focusing plates, external to the cell, whose function is to extract ions from the cell and focus these ions into the mass spectrometer.

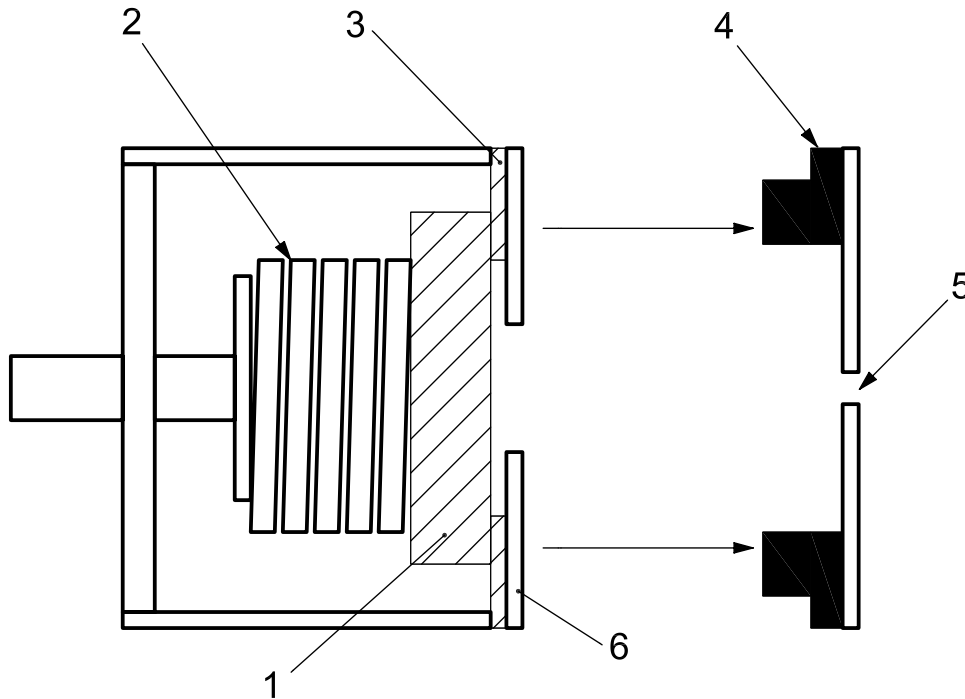
Typically the body of the discharge cell is connected to the anode output of the power supply. The sample serves in the glow discharge cell as a cathode and is connected to the cathode output of the power supply. Discharge cells have been designed to accommodate samples in the geometries recommended in 8.3, and examples of discharge cells are illustrated with the appropriate sample holders in Figures 1 and 2.



Key

- 1 sample holder
- 2 sample
- 3 insulator
- 4 ion exit slit
- 5 anode plate
- 6 cathode plate

Figure 1 — Example of a cell used for the analysis of pin samples



Key

- 1 sample cathode
- 2 spring
- 3 insulator
- 4 cell body
- 5 ion exit slit
- 6 anode plate

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Figure 2 — Example of a cell used for the analysis of flat samples

7.1.2 Source parameters

The source parameters are as follows:

- a) Electrical
 - potential difference between anode and cathode;
 - current;
 - power.
- b) Geometrical
 - dimension of sample exposed to plasma;
 - anode-to-sample distance;
 - cathode dimension;
 - mask dimension, where appropriate.

- c) Gas type and pressure.
- d) Cell temperature.
- e) Type of sample.

7.1.3 Operational modes

The direct-current source may be operated in different modes, including:

- a) constant current with potential difference adjusted by adjusting the plasma gas pressure;
- b) constant potential difference with current adjusted by adjusting the plasma gas pressure;
- c) constant current with plasma gas pressure adjusted by adjusting the potential difference.

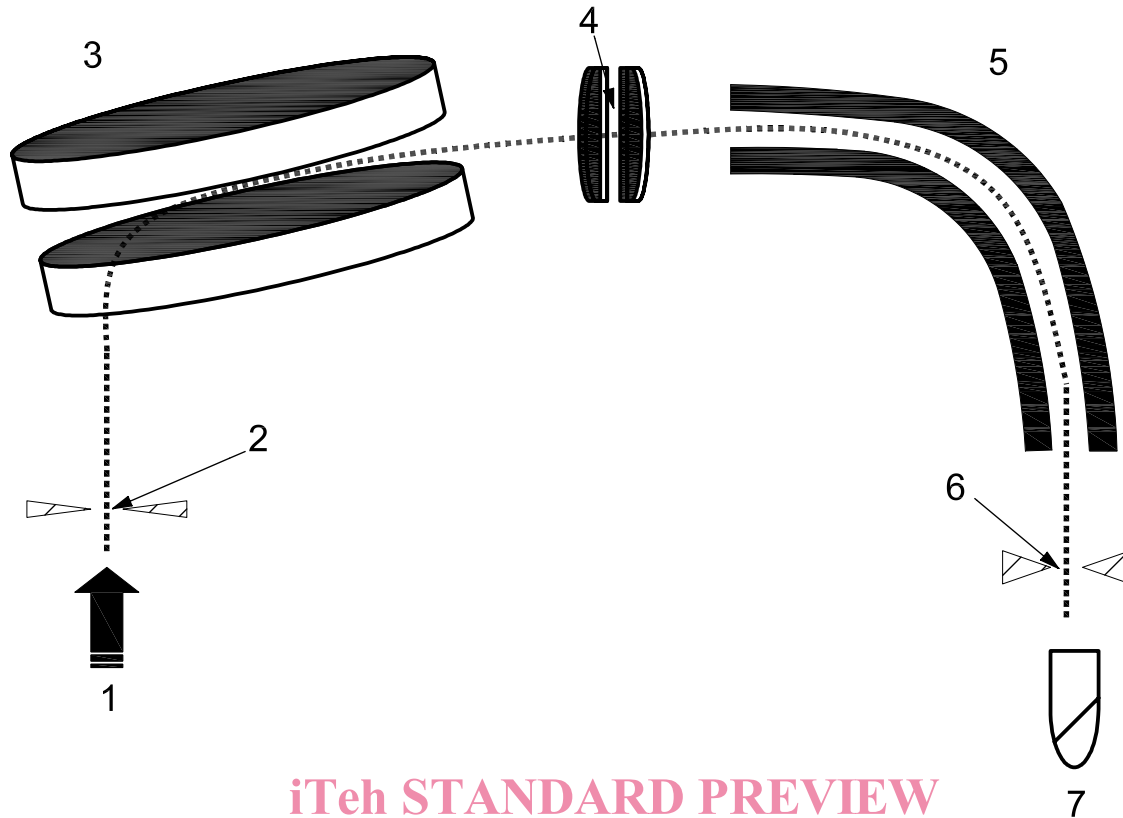
The discharge pressure may be regulated using a mass flow controller, needle valve or knife edge valve. For some types of GD-MS instrument, the high accelerating voltages encountered require a capillary in the gas line to prevent electrical breakdown through the gas line. Radio-frequency-powered GD sources are also being developed for GD-MS.

7.2 Mass analyser

There are two types of mass analyser commonly used for glow discharge mass spectrometry: double-focussing magnetic-sector instruments and quadrupole instruments. Other types, like time-of-flight instruments, are also becoming more common.

- a) **Sector mass spectrometer:** This type of instrument (see Figure 3) typically utilizes an electromagnet and an electrostatic analyser (ESA). The magnet achieves mass separation and, as the magnetic field is increased, ions with greater mass-to-charge ratio are transmitted. The ESA acts as an energy filter and transmits only those ions with the appropriate energies.

This arrangement permits high-transmission and high-resolution operation, giving accurate mass information advantageous with complex sample matrices where there is an increased possibility of interferences. A resolving power of 4 000 is sufficient to overcome most common interferences.



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Key

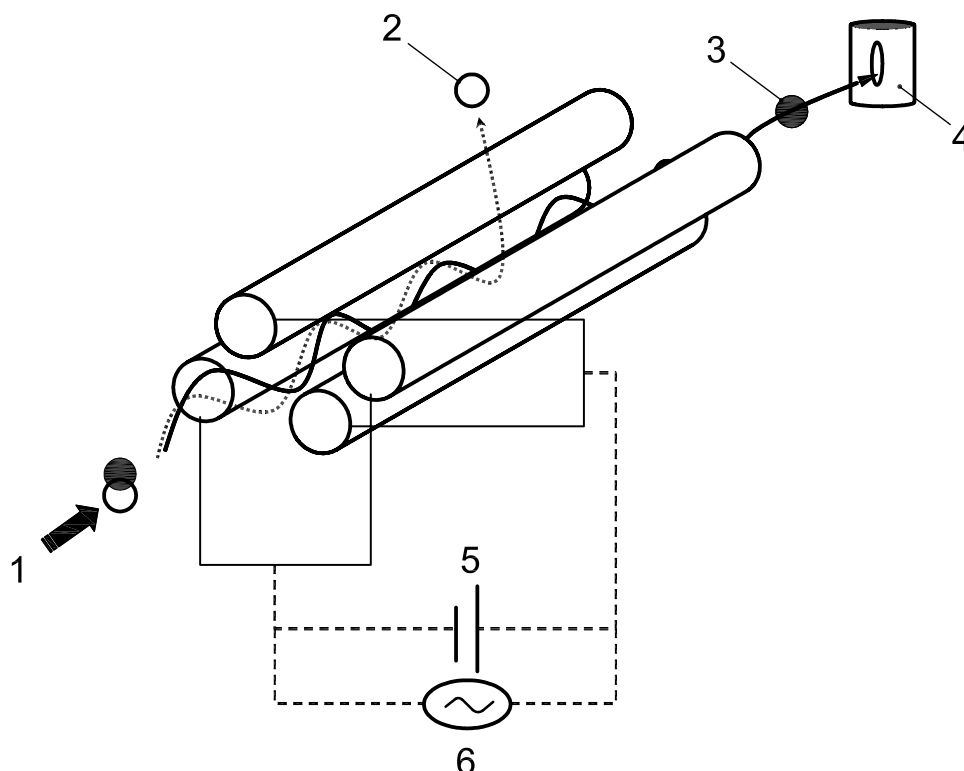
- 1 ion beam
- 2 source slit
- 3 magnet
- 4 intermediate slit
- 5 ESA
- 6 detector slit
- 7 detector

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Figure 3 — Schematic diagram of a magnetic-sector mass analyser

b) Quadrupole: This type of mass spectrometer consists of four parallel rods whose centres form the corners of a square and whose diagonally opposite poles are connected (see Figure 4). The voltage applied to the rods is a superposition of a static potential and a sinusoidal radio-frequency potential. The motion of an ion in the x and y directions is described by the Mathieu equation, the solutions of which show that ions in a particular m/z (mass-to-charge ratio) range can be transmitted along the quadrupole axis.

Whilst a quadrupole instrument does not have the high resolution of a magnetic-sector instrument (the resolving power at mass-to-charge ratio 100 is usually less than 200), it has a much faster scanning speed, is compact and is able to achieve low detection limits if interferences are kept low.

**Key**

- 1 from ion source
- 2 non-resonant ion
- 3 resonant ion
- 4 detector
- 5 d.c.
- 6 a.c.

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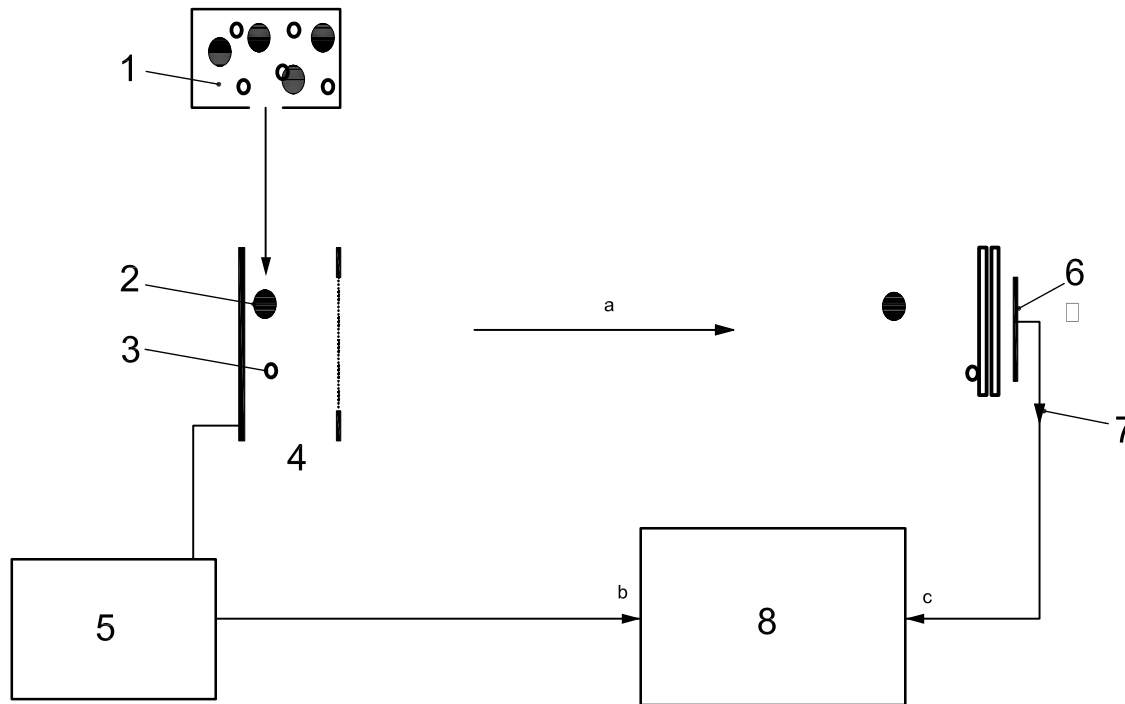
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Figure 4 — Schematic diagram of a quadrupole mass analyser

- c) TOF (time-of-flight) mass spectrometer:** In this type of mass spectrometer, an ensemble of ions is accelerated by an electric field and then allowed to drift a certain distance before impinging on an ion detector (see Figure 5). In the accelerating field, all ions receive the same kinetic energy, which can be equated to $\frac{1}{2}mv^2$. Therefore, ions with a lower m/z (mass-to-charge ratio) will have a higher velocity and will arrive at the detector earlier than ions with a higher m/z . By measuring the arrival time of ions, the m/z of those ions can be determined.

Whilst quadrupole instruments and sector instruments are m/z filters, TOF instruments do not have to scan in order to record a mass spectrum and therefore have the potential for a higher-duty cycle. TOF instruments intrinsically have a large mass range and good mass accuracy. They also have reasonably good resolving power, allowing them to overcome some interferences.



Key

- 1 ion source
 - 2 high-mass particle
 - 3 low-mass particle
 - 4 extractor
 - 5 high-voltage pulser (starts the ions)
 - 6 detector (generates stop signal)
 - 7 signal
 - 8 data-acquisition system (records time of flight)
- a Drift.
 b One start signal.
 c Multiple stop signals.

Figure 5 — Schematic diagram of a time-of-flight mass analyser

NOTE Other types of mass spectrometer (e.g. ion trap mass spectrometers) are being adapted for use with glow discharge ion sources.

7.3 Detector system

As a consequence of the wide mass fraction range measured using GD-MS, usually two detectors are required and/or needed, as follows:

- a) Ion signals from matrix and major trace elements present at mass fractions typically above 0,1 % are measured using a coarse detector such as a Faraday plate or an electron multiplier, operating in the analogue mode. These detectors simply measure the current generated by the ion beam striking the detector.
- b) Ion signals from trace elements present at mass fractions typically below 0,1 % are measured using ion-counting techniques. Examples of ion-counting detectors are the Daly detector and the Channeltron¹⁾, multi-channel plate and venetian-blind electron multipliers.

NOTE Any differences in the response of the detectors are reflected in the instrument calibration.

1) Channeltron[®] is the trade name of a product supplied by Burle. This information is given for the convenience of users of this Technical Specification and does not constitute an endorsement by ISO of this product.