BS ISO 14707:2015

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Surface chemical analysis — Glow discharge optical emission spectrometry (GD-OES) — Introduction to use

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National foreword

This British Standard is the UK implementation of ISO 14707:2015. It supersedes BS ISO 14707:2000 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee CII/60, Surface chemical analysis.

A list of organizations represented on this committee can be obtained on request to its secretary.

Published by BSI Standards Limited 2015

ISBN 978 0 580 83086 0

ICS 71.040.40

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2015.

Amendments/corrigenda issued since publication

Date	Text affected

INTERNATIONAL
STANDARD

ISO
14707

Second edition
2015-03-15

Surface chemical analysis — Glow discharge optical emission spectrometry (GD-OES) — Introduction to use

Analyse chimique des surfaces — Spectrométrie d'émission optique à décharge luminescente — Introduction à son emploi

Reference number
ISO 14707:2015(E)

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Published in Switzerland

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Foreword

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The committee responsible for this document is ISO/TC 201, *Surface chemical analysis, Subcommittee* SC 8, *Glow discharge spectroscopy*.

This second edition cancels and replaces the first edition (ISO 14707:2000), which has been technically revised.

Annex A of this International Standard is for information only.

Introduction

Glow discharge optical emission spectrometry (GD-OES) is used to determine the elemental composition of solid samples. GD-OES can be used for either bulk or depth profile analysis. In bulk analysis, changes in elemental composition with depth into the specimen are assumed to be negligible. In contrast, the main goal of depth profile analysis is usually to gain information concerning such changes of composition. Layer thicknesses amenable to GD-OES depth profiling range from a few nanometres to approximately one hundred micrometres. An average of the concentration within the crater will be obtained and therefore the lateral resolution of GD-OES corresponds to the inner diameter of the anode.

As is true for any instrumental analysis method, the quality of a GD-OES analysis depends markedly on the correct optimization and operation of the instrumentation. This International Standard provides guidelines of practice that are to be followed to ensure that GD-OES analyses are of the highest possible quality.

Surface chemical analysis — Glow discharge optical emission spectrometry (GD-OES) — Introduction to use

1 Scope

This International Standard provides guidelines that are applicable to bulk and depth profiling GD-OES analyses. The guidelines discussed herein are limited to the analysis of rigid solids, and do not cover the analysis of powders, gases or solutions. Combined with specific standard methods which are available now and in the future, these guidelines should enable the regulation of instruments and the control of measuring conditions.

Although several types of glow discharge optical emission sources have been developed over the years, the Grimm type with a hollow anode accounts for a very large majority of glow discharge optical emission devices currently in use both for dc and rf sources. It should be noted, however, that the cathode contact is often located at the back of the sample, in e.g. the Marcus type source, rather than at the front as in the original Grimm design. It should be clearly understood that the guidelines contained herein are equally applicable to both and other source designs and that the Grimm type source is used only as an example.

2 Normative references

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

ISO 3497:1990, *Metallic coatings — Measurement of coating thickness — X-ray spectrometric methods*

ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ISO 5725-3:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 3: Intermediate measures of the precision of a standard measurement method*

ISO 5725-4:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 4: Basic methods for the determination of the trueness of a standard measurement method*

ISO 6955:1982, *Analytical spectroscopic methods — Flame emission, atomic absorption, and atomic fluorescence — Vocabulary*

ISO 11505, *Surface chemical analysis — General procedures for quantitative compositional depth profiling by glow discharge optical emission spectrometry*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3497, ISO 5725-1, ISO 5725-2, ISO 5725-3, ISO 5725-4, and ISO 6955 apply.

4 Principle

Analysis by GD-OES involves the following operations:

a) preparation of the sample to be analysed, generally in the form of a flat plate or disc of dimensions appropriate to the instrument or analytical requirement (round or rectangular samples with a width of more than 3 mm, generally 20 mm to 100 mm, are suitable);

b) atomization and excitation of the analytes to be determined by means of ion sputtering and inter-particle collisions occurring in the glow discharge plasma;

c) measurement of the emission intensities of characteristic spectral lines of the analytes (for depth profiling, emission intensities are recorded as a function of time);

d) determination of the analyte concentrations contained in the sample by calibration with reference materials of known composition (for depth profiling, the sputtered depth as a function of time is also determined by calibration with reference materials of known composition and sputtering rates).

A diagram of a typical GD-OES system is presented in Figure 1. GD-OES is based on the use of a glow discharge device as an optical emission source. The glow discharge device consists of a vacuum chamber filled with a supporting gas, usually argon. The glowing plasma, from which the discharge takes its name, is maintained by a controlled high voltage of 200 V to 2 000 V applied between the anode and cathode in the plasma gas. The solid sample to be analysed serves as the cathode.

Atomization of sample material in the glow discharge is the result of cathode sputtering, the destruction of the negative electrode (cathode) in a gas discharge due to the impact of fast charged and neutral particles. Ions formed in the plasma are accelerated toward the cathode surface by the electric field in the plasma. When an ion or neutral atom collides with the surface, its kinetic energy may be transferred to atoms on the surface, causing some of these surface atoms to be ejected into the plasma. Once in the plasma, these sputtered sample atoms may be ionized and excited through inelastic collisions with electrons or other species. The majority of these excited analyte atoms and ions then emit characteristic optical emission upon relaxing into the lower electronic state. The optical emission is analysed by an optical spectrometer containing a dispersive element, normally a diffraction grating. The intensities of element-specific spectral lines are translated to electrical signals by means of appropriate detectors. A polychromator is commonly employed, so that many elements can be quantified simultaneously. Spectral lines that are not contained in the line set of the polychromator can be accessed by means of a scanning monochromator, if one is available. Also CCD instruments exist, where a spectrum over a wide spectral range can be measured continuously. In practice almost all elements in the periodic table can be determined, including metals, metalloids and non-metals.

5 Apparatus

At a minimum, the apparatus consists of the following:

5.1 Glow discharge optical emission source

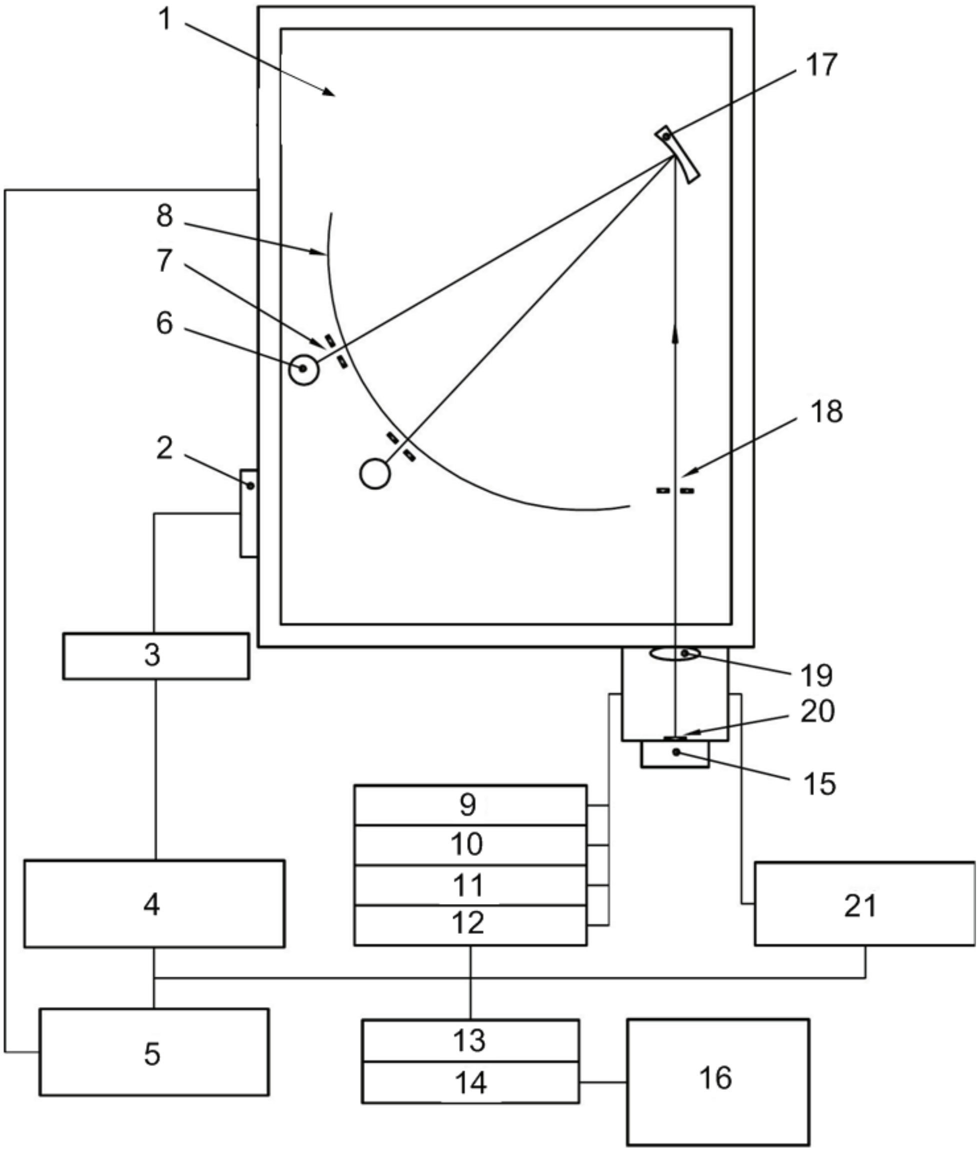
A diagram of a Grimm type glow discharge optical emission device is shown in Figure 2. Several modifications in the device have been introduced by instrument manufacturers. As noted in Clause 4, the sample effectively serves as the cathode. The anode takes the form of a tube with an inner diameter of 1 mm to 10 mm, typically 4 mm. The distance between the front face of the anode and the surface of the cathode is usually between 0,1 mm and 0,3 mm. As a result, ion sputtering is confined to a circular region of the sample surface with a diameter approximately equal to the inner diameter of the anode.

The glow discharge device requires several peripheral pieces of equipment for its operation. These include an electric power supply, one or two vacuum pumps, a source of plasma gas, a means of delivering that gas into the device in a controlled manner and a vacuum gauge. A cooling device, such as a metal block with circulating cooling liquid, is sometimes necessary for thin samples.

a) Source parameters

Glow discharge devices may be operated in either direct current (dc) or radio-frequency (rf) mode. Combinations of these two modes, such as the use of an rf voltage superposed onto a dc voltage, have also been reported. In both the dc and rf power modes, a pulsed discharge is also employed for switching the applied power periodically.

1) For dc operation, the pertinent electrical parameters are discharge current (1 mA to 200 mA) and voltage (200 V to 2 000 V). In addition to the electrical parameters, other parameters are important for the characteristics of the device. These include the inner diameter of the anode (1 mm to 10 mm), gas type and purity (for example, argon, > 99,999 %), gas flow rate or gas pressure introduced (100 ml/min to 500 ml/min, 100 Pa to 1 500 Pa, see note below) and physical characteristics of the sample material (for example, secondary electron emission yield and sputtering yield). The combined effects of all of these factors determine the spectrochemical character of the glow discharge plasma. Generally, it is recommended that the gas flow rate or the gas pressure be varied in real time, in order to achieve constant voltage and cur- rent. As an example, typical operating conditions for dc GD-OES bulk analysis of low-alloy steels are 250 ml/min argon flow rate, 600 V to 1 000 V discharge voltage and 20 mA to 60 mA discharge current, for an anode of 4 mm inner diameter. The sputtering rate in this case is typically 100 nm/s varying from 20 nm/s to 160 nm/s for the conditions given in the example.



Key

1	spectrometer	8	rowland circle	15	sample
2	electrical lead-in	9	cooling system	16	personal computer system
3	preamplifier	10	gas control system	17	grating
4	light intensity measuring circuit	11	RF power supply	18	entrance slit
5	vacuum/gas flush control system	12	DC power supply	19	lens
6	photomultiplier	13	control circuit	20	glow discharge source
7	exit slit	14	local computer	21	glow discharge control system

Figure 1 — Schematic diagram of a GD-OES system

NOTE It should be understood that the discharge gas flow rate is the practical means of controlling the source operating pressure, and so suggested ranges are very much source specific, and are provided here only with reference to the Grimm lamp example.

2) Typical conditions for rf glow discharge include the same range of argon flow rate and similar discharge power and voltage as in dc operation. In terms of the electrical parameters, it is important to note that GD-OES instruments usually measure the forward and reflected power, the applied

RMS (root mean square) voltage, or the peak voltage/current of the rf waveform. If conductive samples are sputtered with rf also the average voltage, called dc bias, can be measured. In the power measurement, it must be understood that the incident power is the forward power delivered to the glow discharge system by the rf power supply. Because of power losses in cables, the cooling system, etc., and the reflected power, caused by an incomplete matching of the generator and plasma impedance, the effective power is lower than the incident power, meaning that the effective power actually dissipated within the glow discharge itself. It is possible to employ a system to automatically measure the power loss as a function of voltage without plasma (under vacuum), prior to the discharge ignition. This value is then subtracted from the incident power during the measurement, providing a rather accurate measure of the effective plasma power. Similarly, in the measurement of voltage/current, the measured values may not be an accurate representation of the effective discharge voltage/current in the plasma. The rf power supply may be of either the fixed or the variable frequency type. In either case, the rf frequency is usually between 3 MHz and 41 MHz. Common fixed frequencies are 6,78 MHz or 13,56 MHz, in order to comply with national and international regulations.

In addition to conductive samples, rf GD-OES allows the analysis of non-conductive samples. For non- conductive samples, the rate at which ion sputtering erodes the sample surface is usually in the range of 1 nm/s to 50 nm/s. Non-conductive layers on conductive substrates may also be examined. For these samples, the applied rf voltage becomes higher than for conductive samples depending on the equipment used.

3) In both the dc and rf power modes, a pulsed discharge may be employed to control the sputtering rate and to reduce the damage of surface coatings such as a polymer film. The frequency and the duty cycle of the pulsed discharge should be determined depending on the sample analysed. Typical values for the frequency and the duty cycle are 100 Hz to 300 Hz and 20 %, respectively.

b) Source/spectrometer interface

The optical radiation emitted from excited sample atoms is guided by lenses or mirrors to the entrance slit of the spectrometer. If spectral lines with wavelengths below 200 nm (i.e. vacuum UV) must be used, the entire optical path from the glow discharge source to optical detector must be sufficiently free of molecular oxygen. This is because the oxygen molecule has very strong absorption bands below 200 nm. Oxygen gas can be eliminated from the optical system by flushing the system with a suitable pure gas such as nitrogen (or argon), or by evacuating the optical path to a pressure of ca 1 Pa. The window separating the source and the spectrometer must be cleaned periodically.

5.2 Optical unit

The most common instruments are equipped with a simultaneous spectrometer (for example, polychromator) with several fixed channels corresponding to analyte elements. It is also common to combine this with a sequential spectrometer (i.e. monochromator). With either simultaneous or sequential spectrometers, the spectral bandpass, and thus the effective spectral resolution, is determined by the dispersion of the instrument and the geometrical slit widths.

5.3 Photoelectric detectors and measuring devices

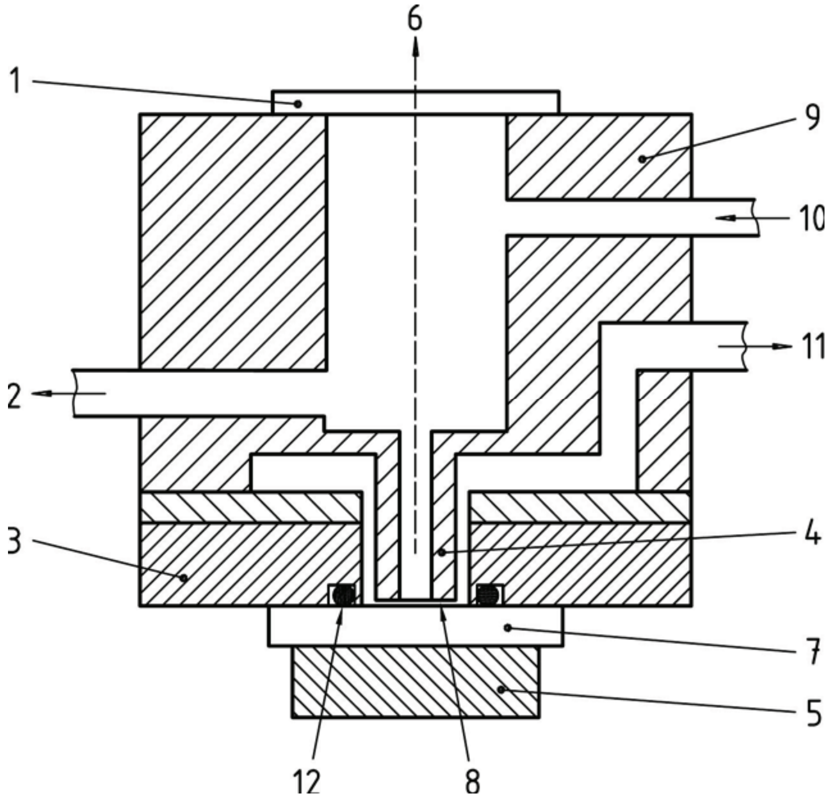
Most glow discharge spectrometers are equipped with photomultiplier tubes for signal detection. To achieve optimum performance (i.e. signal intensity, sensitivity and power of detection), photomultipliers with low dark current and high quantum efficiency are required. The photomultiplier gain shall be selected properly to avoid nonlinear response and saturation. This is accomplished by measuring selected samples containing different analyte concentrations and sputtering rates and adjusting the gain in such a way that sufficient sensitivity at the lowest intensities is ensured, without saturation of the detector at the highest intensities. An array-type detector, such as a charge coupled device (CCD) or a charge injection device (CID), is also used for simultaneous detection to cover a wide spectral range of the analytical lines. The amplified detector output is digitized by means of analog-to-digital converters and transferred to a computer for data storage and further evaluation.

6 Procedure

6.1 Verification tests of apparatus

6.1.1 General

In order to obtain analytical data of the highest possible quality, it is necessary to verify the performance of the spectrometer and all devices connected to the system. If such verification was not performed by the instrument manufacturer at the time of installation, then it should be done by the user. Verification should be also performed periodically. The principal components to be checked are the glow discharge source, the optical unit and the electric measuring devices.



Key

1 window/lens
2 vacuum line B
3 cathode block as a front contact or ceramic insulator as a back contact
4 interior wall of anode
5 cooling block and used as a cathode contact block in some types such as the Marcus type
6 to spectrometer
7 specimen (cathode)
8 front face of anode
9 anode body
10 gas inlet
11 vacuum line A
12 gas-tight vacuum sealing (O-ring)

Figure 2 — An example of a glow discharge source (Grimm hollow type anode)

6.1.2 Glow discharge source

The following should be examined using adequate source parameters [see 5.1 a)] and a suitable sample (for example, iron):

a) the stability of sputtering and plasma conditions while the discharge is running, using intensity ratios of spectral lines of interest to either the background continuum or to discharge gas lines;

b) the sputtering rate of the chosen sample at the selected discharge conditions;

c) the gas quality and tightness of the vacuum system;

d) the condition of the anode (for example, the spacing between its front face and the sample surface).

6.1.3 Optical unit and electric measuring device

The following should be verified.

a) The alignment of the spectrometer entrance slit with respect to the image of the glow discharge projected onto the entrance optics.

b) The spectral resolution of the spectrometer and the accuracy and stability of the wavelength adjustment over the useful spectral range, using a suitable sample (for example, low-alloy steel).

c) The stability of the detector readout with the glow discharge on and off, measured over a suitable period of time.

6.2 Determination

6.2.1 General

In order to achieve the full analytical potential of GD-OES in terms of limit of detection, accuracy and reproducibility, the measurement shall be performed under suitable discharge conditions, which are determined for the particular sample groups to be analysed in accordance with the following instructions.

Quantitative analysis by GD-OES involves the following steps

6.2.2 Preparation of the required calibration specimens

The reliability of the calibration procedure largely determines the accuracy of the analytical results obtained. It is necessary to use calibration specimens of well-known composition for optical emission spectrometric analysis. CRMs (certified reference materials) should be used, but RMs (reference materials) are also acceptable. The calibration specimens should, to the largest possible extent, resemble the samples under measurement with respect to chemical composition and metallurgical pretreatment. The calibration specimens are used to determine the emission intensity of analyte elements as a function of concentration and sputtering rate for depth profiling.

For the quantification of depth profiles, the calibration specimens need not to be very similar to the surface layer materials in composition but they shall have sputtering rates which are well determined and reproducible (see ISO 11505 for details).

Requirements for calibration specimens shape and size are determined by the design of the source. The specimens shall be flat and large enough to cover the contact surface of the sealing medium for vacuum. Moreover, the surface of the specimens shall be sufficiently smooth to form a suitably tight vacuum seal and to position the sample in the right distance to the anode. In order to maintain a suitable discharge, sputtered sample material that is deposited onto the interior wall and front face of the anode shall be regularly removed by means of a cleaning device. A reamer is commonly employed for this purpose.

To resolve problems that may occur during measurement, the following points should be checked using suitable calibration specimens:

a) the range of concentration for each element to be determined;

b) the presence or absence of interfering spectral lines of species associated with the sample or the discharge gas;

c) the contribution of the background emission and its temporal fluctuation;

d) adequate sputtering and data acquisition rates for depth profiling.

6.2.3 Setting up of measuring conditions and analysis of specimens

Usually, the operating instructions supplied by the instrument manufacturer state the operations to be carried out to measure calibration specimens The operations typically specified are as follows:

a) mount a test sample on the glow discharge device and evacuate the source to a vacuum of the order of 1 Pa in the source;

b) set the appropriate conditions of measurement(for example, gas flow rate or pressure);

c) select the wavelengths of the elements to be determined;

d) adjust the measuring parameters, such as the gain of detectors and/or the integration time, as well as the discharge parameters, such as the discharge voltage/current (dc mode) and the power (rf mode), to obtain the sufficient intensity;

e) ignite the glow discharge and analyse specimen under the selected conditions.

For each spectral line and element, calculate calibration functions based on the calibration measurements. These calibration functions are to be calculated according to the application (bulk or depth profile analysis) and the appropriate calibration models recommended by the instrument manufacturer. In case of severe nonlinearity due to self-absorption, a different spectral line should be selected. Line interference from other analyte elements should be corrected in the calibration functions according to the method given by the instrument manufacturer.

6.2.4 Quality check of results

The quality of the GD-OES results should be checked as follows.

a) One or more certified reference materials should be analysed as check standards. Analyte concentrations determined with GD-OES should agree with the certified concentrations within appropriate statistical bounds (see ISO 5725-1, ISO 5725-2, ISO 5725-3 and ISO 5725-4).

b) Whenever practical, analyte concentrations in selected unknown samples determined with GD-OES should be compared to the concentrations obtained by means of a second analytical method. Preferably, the second method should be one that is known to provide high-quality determinations for the analytes and matrices under measurement. In comparing the results from the two methods, particular attention should be given to their relative repeatability and reproducibility (see ISO 3497 and ISO 5725-1, ISO 5725-2, ISO 5725-3 and ISO 5725-4).

6.2.5 Test report

It is recommended that the following parameters, where applicable, be recorded and reported in conjunction with presentation of analytical results:

a) type of excitation (dc or rf);

b) for dc operation, discharge voltage and current;

c) for rf operation, forward and reflected power and frequency (if available also the effective power, effective voltage and dc bias voltage, if conductive samples are measured);

d) gas type and purity;

e) gas flow rate;

f) gas pressure (including type and location of pressure gauge);

g) anode shape and dimension (particularly the inner diameter);

h) measuring or sputtering time;

i) wavelengths of analytical lines;

j) analyte concentrations;

k) limits of detection;

l) repeatability of measurements;

m) sputtered mass and depth.

Annex A
(informative)

Safety

A.1 General

Although no official text has yet been published concerning the problems of safety in GD-OES, and although no generally recognized rules or recommendations relate specifically to this technique, a certain number of precautions should be taken to ensure the safety of operators and their environment. These precautions relate to the following specific aspects:

a) the use of a high-voltage power supply and grounding of the instrument;

b) the use of radio frequencies;

c) the use and storage of compressed-gas cylinders

d) safety checks before and during operation.

A.2 Use of a high-voltage power supply and grounding of the instrument

Electrical connections should comply with the regulations in force. Particular care should be taken to ensure that the connection of the instrument to ground is correct, and the efficiency of the ground connection should also be checked. The high voltage between samples and anode must be off when samples are being handled. Discharging of power supply capacitors should be done before touching source or sample.

A.3 Use of radio frequencies

Lacking certain knowledge of harm caused by radio frequencies, all radiation should be as low as possible. The Electromagnetic Compatibility Directive (Directive 89/336/EEC) was adopted in May 1989, and came into force on 1 January, 1992. This applies to nearly all electrical and electronic equipment. It requires that equipment neither cause excessive electrical interference nor be unduly susceptible to it.

A.4 Use and storage of compressed-gas cylinders

The compressed-gas cylinders should regularly be tested by the appropriate authorities. Preferably, cylinders should never be stored or used inside the laboratory. Rather, they should be located at all times outside the laboratory in a place that is well ventilated, away from direct heat, and accessible only 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. If a single cylinder supplies two or more users, the identities of the users in operation at any particular time should also be provided. A general inspection should be performed each time a gas cylinder is used in order to ensure that there are no obvious safety concerns. Each day or working shift, concerning about safety, the last person to leave the laboratory should check all cylinders and make sure that they are in proper status.

A.5 Safety checks before and during operation

Ensure that all safety interlocks function properly prior to using the instrument. Before the glow discharge is ignited and after the ignition for a while, the stability of the gas flow rate and pressure should be checked to be sure that adequate vacuum condition is being kept.

Bibliography

[1] ISO 18115-1, *Surface chemical analysis — Vocabulary — Part 1: General terms and terms used in spectroscopy*

[2] Council Directive 89/336/EEC of 3 May 1989 on the approximation of the laws of the Member States relating to Electromagnetic Compatibility OJ L 139 of 23 May 1989

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