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**Surface chemical analysis —  
Secondary-ion mass spectrometry —  
Repeatability and constancy of the  
relative-intensity scale in static  
secondary-ion mass spectrometry**

*Analyse chimique des surfaces — Spectrométrie de masse des ions  
secondaires — Répétabilité et constance de l'échelle des intensités  
relatives en spectrométrie statique de masse des ions secondaires*

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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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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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 23830 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 6, *Secondary ion mass spectrometry*.

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## Introduction

Static secondary-ion mass spectrometry (SIMS) is used extensively for the surface analysis of complex materials. Static SIMS is most often used for identification of materials at surfaces and for the quantification of mixtures, for example functionalized-surface polymer blends. Quantification is usually achieved through measuring relative peak intensities and by comparing these with appropriate peaks from reference materials. Repeatability is important for these measurements in order to understand if any variations observed are significant. The constancy of relative intensities is important for relating work historically to databases as well as establishing the instrument behaviour. Many static SIMS measurements are for the purpose of identification of an unknown material through the use of pattern matching with spectral libraries. The extent to which this is meaningful depends on the instrument repeatability and stability. There are two important instrumental contributions to the uncertainty of static SIMS intensity measurements that are addressed in this International Standard: (i) the repeatability of positive-ion relative-intensity measurements and (ii) the drift of the positive-ion relative intensities with time. With negative ions, the control of the surface potential for insulators is not yet sufficiently good for high repeatability. Consequently, negative ions are not included in this International Standard, although it is possible that the concept described will work for negative ions if the surface potential control problem is solved.

Repeatability is important for analysing the trends and differences between samples that are similar. Poor repeatability can erroneously give the conclusion that the samples are significantly different. The instrumental issues that limit the measurement repeatability include the stability of the ion source, charge stabilization, the settings of the detector, the sensitivity of the instrument to the sample placement, the data-acquisition parameters and the data-processing procedure.

This International Standard describes a simple method for confirming the repeatability and constancy of the relative-intensity scale of the instrument so that the instrument behaviour is characterized and that any remedial action, such as improving the operating procedure or resetting of the instrument parameters, may be made. This method should, therefore, be conducted at regular intervals and is most useful if the data include a period in which the instrument has been checked to be working correctly by the manufacturer or other appropriate body. This method uses a sample of poly(tetrafluoroethylene), which is abbreviated to PTFE, and is applicable to static SIMS spectrometers with charge stabilization.

This method does not address all of the possible defects of instruments since the required tests would be very time-consuming and need both specialist knowledge and equipment. This method is, however, designed to address the basic common problem of repeatability and of drift of the relative-intensity scales of static SIMS instruments.

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# Surface chemical analysis — Secondary-ion mass spectrometry — Repeatability and constancy of the relative-intensity scale in static secondary-ion mass spectrometry

## 1 Scope

This International Standard specifies a method for confirming the repeatability and constancy of the positive-ion relative-intensity scale of static secondary-ion mass spectrometers, for general analytical purposes. It is only applicable to instruments that incorporate an electron gun for charge neutralization. It is not intended to be a calibration of the intensity/mass response function. That calibration may be made by the instrument manufacturer or another organization. The present method provides data to confirm the constancy of relative intensities with instrument usage. Guidance is given on some of the instrument settings that may affect this constancy.

## 2 Symbols and abbreviations

$A_1$	average combined peak area for the $C_3F_3$ and $C_2F_5$ peaks
$A_2$	average combined peak area for the $C_7F_{13}$ and $C_8F_{15}$ peaks
$A_3$	average combined peak area for the $C_{14}F_{27}$ and $C_{15}F_{29}$ peaks
$d$	beam diameter ( $\mu\text{m}$ )
$e$	charge on the electron (C)
$F$	pulse repetition rate or frequency ( $\text{s}^{-1}$ ) (required for time-of-flight systems only)
$i$	index for the $i$ th of the 13 mass peaks
$I_{ij}$	matrix of peak intensities for the $i$ th mass peak and $j$ th spectrum
$\bar{I}_i$	average peak intensity over the seven spectra
$j$	index for the $j$ th of the seven spectra
$J$	total ion fluence ( $\text{ions}\cdot\text{m}^{-2}$ )
$n$	number of complete raster frames
$N_{ij}$	matrix of normalized peak intensities for the $i$ th mass peak and $j$ th spectrum
$p$	number of ion pulses delivered per pixel (required for time-of-flight systems only)
$P_{ij}$	matrix of relative peak intensities for the $i$ th mass peak and $j$ th spectrum
$\bar{P}_j$	average relative peak intensity over nine mass peaks of the $j$ th spectrum

$q$	time-averaged pulsed ion beam current (A) (required for time-of-flight systems only)
$Q$	ion beam current (A) (required for non-pulsed systems and for some time-of-flight systems)
$r$	relative-intensity repeatability
$R$	length of side of square raster ( $\mu\text{m}$ )
SIMS	secondary-ion mass spectrometry
$T$	total spectrum acquisition time (s)
ToF	time of flight
$u$	unified atomic mass unit
$U_{95}(A_1/A_2)$	uncertainty in $A_1/A_2$
$U_{95}(A_3/A_2)$	uncertainty in $A_3/A_2$
$w$	pulse width (s)
$X$	number of pixels along a line of the raster

### 3 Outline of method

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Here, the method is outlined so that the detailed procedure, given in Clause 4, may be understood in context. To evaluate a static SIMS spectrometer using this procedure, obtain a fresh reel of PTFE tape, of the type used for domestic plumbing, in order to measure the intensities of selected SIMS mass peaks with appropriate instrumental settings. This material is chosen because it is readily available, has low surface contamination and has been well characterized by static SIMS to have the surface uniformity to achieve excellent levels of repeatability. The peaks are chosen to exhibit no mass interference and so the procedure is relevant to instruments with both high and low mass resolution.

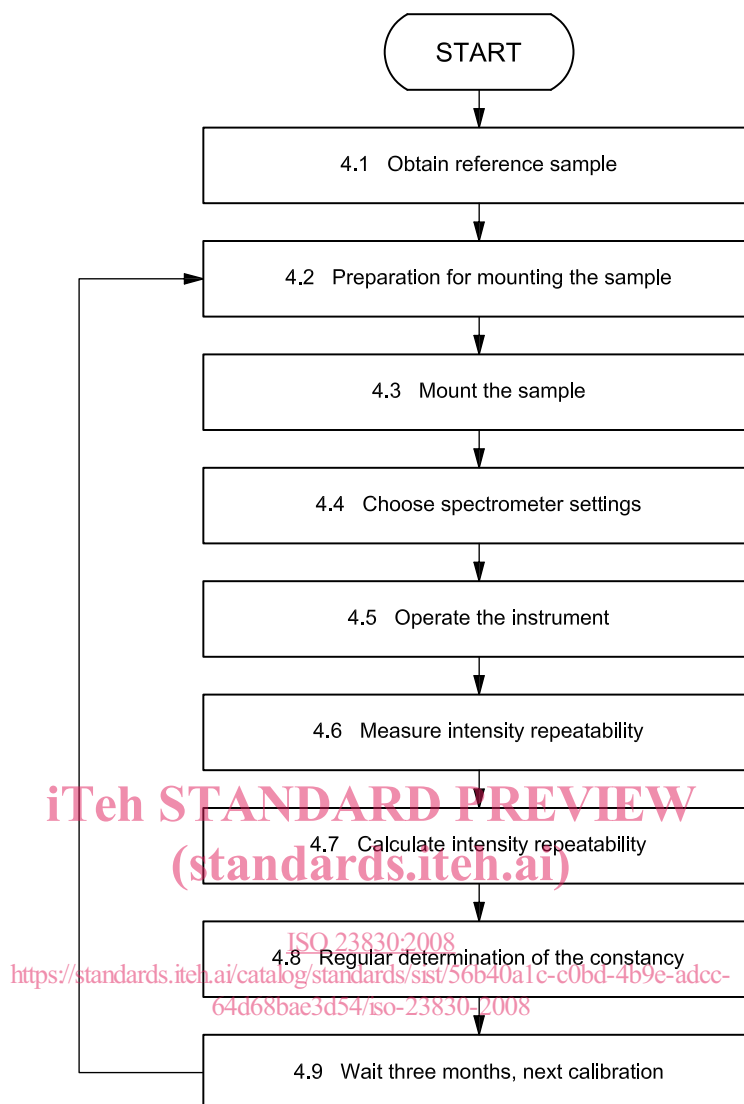
The initial steps of procuring the sample and setting up the instrument are described in 4.1 to 4.5 and shown in the flowchart in Figure 1 with the relevant paragraph headings paraphrased.

In 4.6, mass spectra are acquired in a sequence repeated seven times. These data give the repeatability standard deviations of the peak intensities. These repeatabilities have contributions from the stabilities of the ion source, the surface charge on the sample, the spectrometer detector and the electronic supplies, as well as from the sensitivity of the measured peak intensity to the sample position and from the statistical noise in the peak. In the method, conditions are defined to ensure that the statistical noise of the measured intensities is relatively small. The value of the repeatability standard deviation may depend on the sample-positioning procedure. In 4.6.1, the use of a consistent sample-positioning procedure is required and the final calibration is only valid for samples positioned using this positioning procedure.

Generally, for static SIMS, the relative peak intensities, rather than the absolute intensities, are most important for material identification. In this method, therefore, the scope is limited to determining the constancy of the relative intensities. These determinations are made in 4.7 and their calculation defined in 4.8, as shown in the flowchart in Figure 1.

In practice, the intensity/mass response function of spectrometers may change significantly with instrument use and especially between different operators of the same instrument. It may therefore be useful for each analyst to conduct this procedure. The interval between repeat assessments is given in 4.9. It is imperative that the instrument be operated using a documented procedure to help minimize variations in spectral response.





**Figure 1 — Flowchart of the sequence of operations of the method**  
(Subclause numbers are given with items for cross-referencing with the body of the text.)

## 4 Method for confirming the repeatability and constancy of the intensity scale

### 4.1 Obtaining the reference sample

For the calibration of static SIMS spectrometers, obtain a new reel of PTFE tape of the type used for domestic plumbing. Label this reel and keep it with your reference samples for later checks in 4.9.

**NOTE** The PTFE is usually in the form of a reel of tape of length 12 m and width 12 mm and approximately 0,075 mm thick.

### 4.2 Preparation for mounting the sample

Samples shall only be handled with clean metal tweezers held using powderless polyethylene gloves. Vinyl gloves, often used in clean rooms, are coated with a release agent from the moulding process and shall not be used. The release agent is very mobile and quickly contaminates the samples. This leads to poor measurement repeatability and poor-quality data.

### 4.3 Mounting the sample

Remove and discard the first 20 cm of material from the reel obtained in 4.1 and then cut appropriately sized samples from the subsequent material with clean scissors. As the reel is unwound, a fresh surface of PTFE is exposed and it is this surface that is analysed. Do not clean the sample. Mount samples on the sample holder to produce a flat, even surface using a mechanical clamping or fixing method. Do not use adhesive tape. Ensure that the reverse side of the sample is against a conducting surface, electrically connected to the sample holder. The PTFE shall not be placed over a hole.

NOTE The presence of a hole under the sample leads to poor mass resolution and repeatability in systems that use high extraction fields, such as time-of-flight and magnetic-sector systems.

Repeat measurements are required later in 4.9 at intervals of no longer than three months. For this, a fresh sample is required and, for consistency, the sample should be from the same reel.

### 4.4 Choosing the spectrometer settings for which intensity stability is to be determined

Choose the spectrometer operating settings for the positive secondary ions for which the intensity stability is to be determined. The method from 4.5 to 4.9 shall be repeated for each ion source for which confirmation is required.

NOTE The repeatability of the relative intensities varies with the combination of settings used. In general, the repeatability will be best when using an energy acceptance of the mass analyser of 50 eV or more.

### 4.5 Operating the instrument

4.5.1 Operate the instrument in accordance with the manufacturer's or local documented instructions. The instrument shall have fully cooled following any bakeout. Ensure that the operation is within the manufacturer's recommended ranges for ion beam current, counting rates, spectrometer scan rate and any other parameter specified by the manufacturer. Check that the detector multiplier settings are correctly adjusted.

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4.5.2 Centre the ion beam in the acceptance area of the mass spectrometer. Ensure that the ion beam focus diameter,  $d$ , is sufficiently defocused such that

$$d > \frac{2R}{X} \quad (1)$$

for a square raster with side of length  $R$  and where  $X$  is the number of pixels along a line of the raster. If it is found that the maximum achievable beam diameter in the instrument is too small to satisfy Equation (1), the number of pixels will need to be increased or the raster size reduced.

4.5.3 Select values of the time-averaged pulsed ion beam current,  $q$ , the total acquisition time,  $T$ , and the raster size,  $R$ , to give an ion fluence,  $J$ , of  $< 1 \times 10^{16}$  ions·m<sup>-2</sup>.  $J$  is given by

$$J = \frac{qT}{eR^2} \quad (2)$$

For non-pulsed instruments, such as magnetic-sector and quadrupole-based systems, use Equation (2) but with the pulsed current,  $q$ , substituted by the non-pulsed current,  $Q$ .

For some ToF instruments,  $q$  is not known and the non-pulsed current,  $Q$ , is recorded instead, in which case

$$J = \frac{FQwT}{eR^2} \quad (3)$$

where

$F$  is the frequency of the ion pulses;

$w$  is the ion beam pulse width (without pulse beam bunching).

**EXAMPLE** A 0,5 pA beam (either ToF instrument pulsed beam current or the non-pulsed beam current for a quadrupole or magnetic-sector instrument) with a 200  $\mu\text{m}$  square raster and 128 s acquisition time would need to be defocused to a diameter greater than 3,1  $\mu\text{m}$  for a 128  $\times$  128 pixel display. If it can only be defocused to 1  $\mu\text{m}$ , a 256  $\times$  256 pixel display needs to be used or the local fluence on a pixel, given by the  $1 \times 10^{16}$  ions·m<sup>-2</sup> limit, will be exceeded by a factor of more than 2.

The total acquisition time,  $T$ , is the total time that the ion beam impacts the sample surface. For pulsed systems, this includes the full cycle time, not just the time width of the ion pulse at the surface. Some instrument computers, especially for ToF instruments, report a “total time” which includes computer-processing time when the ion beam is blanked and an “acquisition time” which does not include the additional computer-processing time. In this case, use the “acquisition time” value. Terms used in commercial instruments may not be consistent with the terms used here.

**4.5.4** For magnetic-sector and quadrupole instruments, operate in a spectroscopy mode using a high-speed scan rate (typically at television rates) rather than the digital raster used for imaging purposes. If you need to use the digital raster, use an acquisition time to acquire an integer number of frames,  $n$ , above 20, if possible.

For ToF systems, acquire an integer number of frames,  $n$ , above 20, if possible. If you cannot acquire an integer number of frames,  $n$ , then, to ensure homogeneous sampling over the raster area,  $n$  should be kept above 20 provided the analysis is across a uniform area of spectrometer transmittance. If this is uncertain, use 100 frames. This ensures that the final frame, which most likely will not be a full frame, represents only a small fraction of the data. The number of frames,  $n$ , with  $p$  ion pulses per pixel is given by

$$n = \frac{TF}{X^2 p} \quad (4)$$

**NOTE** For ToF instruments, typically,  $p = 1$  for spectroscopy.

**4.5.5** Measure the ion beam current density,  $J$ , with the raster operational and using, if available, a suitable Faraday cup. If your system does not have a Faraday cup, a simple but accurate device may be made by drilling a small hole in a sample holder, aligned to the ion beam axis and with a depth greater than five times the diameter. The diameter shall be in the range 0,1 mm to 1 mm.

**NOTE** Details of Faraday cup behaviour may be found in Reference [1].

**4.5.6** At high primary-ion beam currents, a significant fraction of counts are lost through detector saturation. Ensure that the primary-ion beam current is below a level at which < 2 % of counts are lost through detector saturation.

Primary ions of mass > 70 u or primary ions composed of clusters may cause peak saturation even at low primary-ion currents. This is usually manifested by reduced or complete loss of intensities in the mass range following the saturated peak. For Bi<sup>+</sup>, a pulsed ion beam current of no more than 0,1 pA is recommended.

**4.5.7** For maximum efficiency of ion detection, set the impact energy of the detected ions at the detector surface to the maximum stable value that does not contribute significantly to the background noise.

**NOTE** High counting rates<sup>[2]</sup> or incorrect detector voltages<sup>[3]</sup> can cause peak distortions that lead to erroneous peak intensity measurements.

**4.5.8** A proper charge stabilization procedure is essential for obtaining good measurements. Use the manufacturer's procedure or a local documented procedure. Ensure that your procedure is clear and complete.

**NOTE** Guidance for charge stabilization is provided in References [4] and [5].