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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*
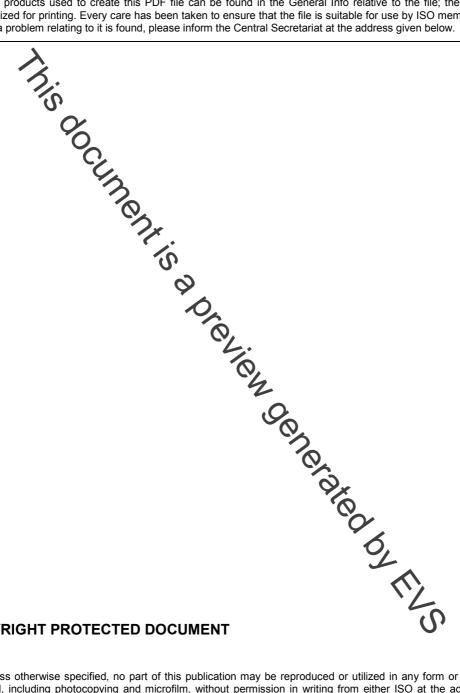
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# Foreword

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

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

# 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.

# 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 (µm) |
| e | charge on the electron (C) |
| $F$ | pulse repetition rate or frequency ($s^{-1}$) (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 (ions·$m^{-2}$) |
| $n$ | 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 |

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