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Surface chemical analysis — Secondary ion mass spectrometry — Linearity of intensity scale in single ion counting time-of-flight mass analysers

Analyse chimique des surfaces — Spectrométrie de masse des ions secondaires — Linéarité de l'échelle d'intensité des analyseurs de masse à temps de vol pour comptage des ions individuels

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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

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 17862:2013), which has been technically revised.

The main changes are as follows:

— the procedure has been simplified by removing the informative background (including Annexes B to D);

— all figures have been fixed to adhere with ISO standards.

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.

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Introduction

For the quantitative analysis of materials using secondary ion mass spectrometry (SIMS), measurements are made of the spectral intensities. Nonlinearity in the instrument intensity scale, unless corrected, leads directly to errors in the relative amounts of material determined at surfaces and in-depth profiles. In general, intensity scales are linear at very low count rates, or more correctly low counts per pulse, but become progressively nonlinear as the count rates rise. Measurements of intensity rely on the measurement system delivering an intensity signal fixed in proportion to the intensity being measured. In counting systems, this proportionality is expected to be unity. If this proportionality varies with the signal level or counting rate, the measurement system is said to be nonlinear. It is rare for nonlinearities below 1 % to be treated as significant. The intensity scale nonlinearity can exceed 1 % for count rates that exceed 5 % of the maximum permissible count rate[2]. For many instruments, the nonlinearity behaviour will not vary significantly from month-to-month, provided the detection system is correctly set. For these instruments, the count rate can be corrected, using the relevant relationship, so that the corrected intensity is then linear for a greatly extended fraction of the maximum obtainable count rate. This correction to the intensity scale might or might not already be available in the instrument's data capture or processing computer. In this document, a simple test of linearity is provided for the intensity lost in systems in which secondary ions arrive at a detector based on a microchannel plate or scintillator and photomultiplier followed by a time-to-digital converter. If this test is shown to be valid, a correction is provided that, for suitable instruments, can extend the intensity scale by up to a factor of more than 50. For some instruments, the nonlinearity cannot be predictable nor described by any simple relationship. For these instruments, this document allows the extent of the nonlinearity to be measured and a maximum count rate for an acceptable limit of divergence from linearity to be defined. In some cases, adjustments to the instrumental settings can improve the situation so that the required correction is then valid. The limit of divergence from linearity is set by the user appropriately for the analyses to be conducted.

Although there are a number of causes of nonlinearities in TOF-SIMS instrumentation, the most significant is intensity saturation caused by the effective dead-time of the detector system. This arises since only one secondary ion count per primary ion pulse can be detected within a dead-time interval τ, regardless of the actual number of secondary ions arriving at the detector. Nonlinearity can also be aggravated by unwanted background in the spectra.

This document provides, and can only provide, a correction to the dead-time nonlinearity for a somewhat ideal situation and not for all cases. Nevertheless, the significantly enhanced dynamic range or rate of working can be very important. Suggestions are included to optimize the instrument to provide the best measurement capability and to diagnose simple instrumental defects such as detector faults, e.g. a low detector efficiency or a detector not providing single ion counting. Then, a dead-time Poissonian correction is established to correct the measured counts within certain limits set by the analyst. This establishes an upper value for c_M, the count per pulse, either before or after correction. This upper limit is generally applicable to peaks where the signal is constant with both time and spatial distribution, where there is only one peak within the dead-time interval, and where the background intensities are negligible (these conditions are not always satisfied in practice). This is explored and explained in detail in Reference [2]. The results from applying this document relate to a “best-case scenario” and the linearity achievable with Formula (1) can be lower in real cases where it is not practical to use a wide peak integration limit of ± the dead-time. More advanced dead-time correction routines should be sought in these cases and their effectiveness can be tested using the methodology here.

This document requires technical skills that may go beyond everyday operation and should be used when characterizing a new spectrometer so that it can be operated in an appropriate intensity range. It should then be repeated after any substantive modification to the detection circuits, after replacement of the microchannel plate (MCP), or at approximately 1 year intervals.

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Surface chemical analysis — Secondary ion mass spectrometry — Linearity of intensity scale in single ion counting time-of-flight mass analysers

1 Scope

This document specifies a method for determining the maximum count rate for an acceptable limit of divergence from linearity of the intensity scale in single ion counting time-of-flight (TOF) secondary ion mass spectrometers using a test based on isotopic ratios in spectra from poly(tetrafluoroethylene) (PTFE). It also includes a method to correct for intensity nonlinearity arising from intensity lost from a microchannel plate (MCP) or scintillator and photomultiplier followed by a time-to-digital converter (TDC) detection system caused by secondary ions arriving during its dead-time. The correction can increase the intensity range for 95 % linearity by a factor of up to more than 50 so that a higher maximum count rate can be employed for those spectrometers for which the relevant correction formulae have been shown to be valid.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 13084, *Surface chemical analysis — Secondary ion mass spectrometry — Calibration of the mass scale for a time-of-flight secondary ion mass spectrometer*

3 Terms, definitions, symbols and abbreviated terms

3.1 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological 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/

3.2 Symbols

The term intensity is used below and elsewhere. This refers to a measurement of peak area in the spectrum.

c_M measured counts per pulse intensity

c_P corrected counts per pulse intensity

F_M shorthand for $F_M(i,j)$

$F_M(i,j)$ ratio of measured intensities for the ith $^{12}C_xF_y^+$ and $^{13}C^{12}C_{x-1}F_y^+$ secondary ions in Table 1

F_P shorthand for $F_P(i,j)$

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