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Surface chemical analysis — Mass spectrometries — Calibration of the mass scale for a time-of-flight secondary ion mass spectrometer

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Foreword

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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 document 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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This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*, Subcommittee SC 6, *Mass spectrometries*.

This third edition cancels and replaces the second edition (ISO 13084:2018), which has been technically revised.

The main changes are as follows:

— addition of informative Annex B.

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

Secondary ion mass spectrometry (SIMS) is a powerful technique for the analysis of organic and molecular surfaces. Over the last decade, instrumentation has improved significantly so that modern instruments now have very high repeatability and constancy[2]. A growing need is for the identification of the chemical composition of complex molecules from accurate measurements of the mass of the secondary ions. The relative mass accuracy to do this and to distinguish between molecules that contain different chemical constituents, but are of the same nominal mass (rounded to the nearest integer mass), is thus an important parameter. A relative mass accuracy of better than 10 ppm is required to distinguish between C_2H_4 (28,031 30 u) and Si (27,976 92 u) in a parent ion with total mass up to 1 000 u, and between CH_2 (14,015 65 u) and N (14,003 07 u) in parent ions with total mass up to 300 u. However, in a recent interlaboratory study[3], the average fractional mass accuracy was found to be 150 ppm, which is significantly worse than the accuracy needed for unambiguous identification of ions. A detailed study[4] shows that the key factors degrading the accuracy include the large kinetic energy distribution of secondary ions, non-optimized instrument parameters and extrapolation of the mass scale calibration.

This document describes a simple method, using locally sourced material, to optimize the instrumental parameters, as well as a procedure to ensure that accurate calibration of the mass scale is achieved within a selectable uncertainty.

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Surface chemical analysis — Mass spectrometries — Calibration of the mass scale for a time-of-flight secondary ion mass spectrometer

1 Scope

This document specifies a method to optimize the mass calibration accuracy in time-of-flight secondary ion mass spectrometry (SIMS) instruments used for general analytical purposes. This document is only applicable to time-of-flight instruments but is not restricted to any particular instrument design. This document gives guidance for some of the instrumental parameters that can be optimized using this procedure and the types of generic peaks suitable to calibrate the mass scale for optimum mass accuracy.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology 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/

4 Symbols and abbreviated terms

4.1 Symbols

m	mass of interest
m_1	calibration mass 1
m_2	calibration mass 2
M	mass (u)
M_0	peak centre (u)
ΔM	mass accuracy (u)
M_P	measured peak mass (u)
M_T	true mass (u)
$U(m)$	mass uncertainty for a mass, m, arising from calibration
U_1	uncertainty in the accurate mass measurement of m_1
U_2	uncertainty in the accurate mass measurement of m_2

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U_0	average uncertainty in an accurate mass measurement
V_R	reflector or acceptance voltage (V)
W	relative mass accuracy
x	number of carbon atoms
y	number of hydrogen atoms
G_0	scaling term
α	asymmetry term
$\sigma(\Delta M)$	standard deviation of the mass accuracy for a number of peaks
σ_M	average of the standard deviations of ΔM for each of the four $C_xH_y^+$ cascades with 4, 6, 7 and 8 carbon atoms

4.2 Abbreviated terms

MEMS	micro-electromechanical system
PC	polycarbonate
ppm	parts per million
r/min	revolutions per minute
SIMS	secondary ion mass spectrometry
THF	tetrahydrofuran
ToF	time-of-flight

5 Outline of method

Here, the method is outlined so that the detailed procedure, given in Clause 6, can be understood in context. First, to optimize a time-of-flight mass spectrometer using this procedure, obtain a thin film of polycarbonate (PC) on a conducting substrate (silicon). The optimization procedure is achieved by carrying out the procedures in 6.3 to 6.5 iteratively; it uses 19 specific C_xH_y peaks in the PC positive-ion mass spectrum. In 6.6, a general calibration procedure is given which provides the rules by which calibrations for inorganics and organics may be incorporated. This leads to a new generic set of ions for mass calibration that can improve the mass accuracy from some often-used calibrations by a factor of five. The effects of extrapolation beyond the calibration range are discussed and a recommended procedure is given to ensure that accurate mass is achieved, within a selectable uncertainty, for large molecules. Therefore, the procedure has two parts, optimization and calibration. Subclauses 6.1 to 6.5 are only required as part of the regular maintenance of the instrument as defined by the testing laboratory. Subclause 6.6 is required for all calibrations of the mass scale. This is summarized in the flowchart in Figure 1.

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