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**Surface chemical analysis —  
Secondary ion mass spectrometry  
— Correction method for saturated  
intensity in single ion counting  
dynamic secondary ion mass  
spectrometry**

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*Analyse chimique des surfaces — Spectrométrie de masse des ions  
secondaires — Méthode de correction de l'intensité de saturation en  
SIMS dynamique à comptage d'ions individuel*

ISO 20411:2018

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# Contents

	Page
Foreword.....	iv
Introduction.....	v
<b>1 Scope.....</b>	<b>1</b>
<b>2 Normative references.....</b>	<b>1</b>
<b>3 Terms and definitions.....</b>	<b>1</b>
<b>4 Symbols and abbreviated terms.....</b>	<b>2</b>
<b>5 Outline of method.....</b>	<b>3</b>
<b>6 Procedure for evaluating intensity linearity.....</b>	<b>5</b>
6.1 Obtaining reference sample.....	5
6.2 Setting the sample.....	5
6.3 Operating the instrument.....	5
6.3.1 Setting the ion beam and the mass analyser.....	5
6.3.2 Setting the charge compensation.....	5
6.3.3 Setting the ion detector.....	5
6.4 Acquiring the data.....	5
6.5 Assessing the linearity without and with intensity correction.....	6
6.5.1 Interpolating the minor isotope intensity.....	6
6.5.2 Correcting the ratio of the isotope abundance to the instrument transmittance.....	7
6.5.3 Assessing the linearity of intensity.....	8
6.5.4 Correcting the saturated intensity.....	8
6.5.5 Assessing the uncertainty of the data.....	11
<b>7 Reporting the results.....</b>	<b>12</b>
<b>8 Correcting saturated intensity to the measurement results of the analysis samples.....</b>	<b>12</b>
<b>Annex A (informative) Effect of the dead time correction with various instruments for VAMAS study.....</b>	<b>13</b>
<b>Bibliography.....</b>	<b>15</b>

## 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](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

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For an explanation on 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 the following URL: [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html). (standards.iteh.ai)

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

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## Introduction

For the quantitative analysis of materials using secondary ion mass spectrometry (SIMS), the abundance of a species should be a linear function of its measured signal intensity. However, this relationship can become nonlinear as the count rates rise because of saturation of the detector, which uses a pulse counting system. This causes underestimation of an abundance of high-count-rate species. The nonlinearity of the counting system can be corrected using the relevant method, so that the corrected count rate gives a wider linear range.

This document explains how to assess the linearity in the high-count-rate region. The document also provides a correction method for the saturated intensity in a certain situation. It uses a test based on depth profile analysis of two isotopes in a reference material which has a gradual concentration change between low and high concentration regimes. The correction method is based on the approximate intermediate extended dead time model.

This document should be used when characterizing a new spectrometer so that it may be operated in an appropriate intensity range or when applying a new analysis condition. It should then be repeated after any substantive modification to the detection circuits, such as the multiplier voltage or the threshold, after replacement of their multiplier, or at approximately 6-monthly intervals.

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# Surface chemical analysis — Secondary ion mass spectrometry — Correction method for saturated intensity in single ion counting dynamic secondary ion mass spectrometry

## 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 pulse counting magnetic sector-type secondary ion mass spectrometers or quadrupole secondary ion mass spectrometers. It uses a test based on depth profile analysis of two isotopes in a reference material which has a gradual concentration change between low and high concentration regimes. It also includes a correction method for saturated intensity caused by the dead time of the detector. The correction can increase the intensity range for 95 % linearity so that a higher maximum count rate can be employed for those spectrometers for which the relevant correction equations have been shown to be valid.

This document does not apply to time of flight mass spectrometers.

This document is only applicable to elements with minor isotopes. It is not applicable if the element is monoisotopic or contains isotopes with equal abundances.

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

ISO 18115-2, *Surface chemical analysis — Vocabulary — Part 2: Terms used in scanning-probe microscopy*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18115-1 and ISO 18115-2 and the following apply.

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 <http://www.electropedia.org/>

### 3.1

#### major isotope

isotope whose abundance is more than others of the same element

### 3.2

#### minor isotope

isotope whose abundance is less than a *major isotope* (3.1)

**3.3 pulse counting system**

ion detector system which counts an ion as a single pulse

Note 1 to entry: When an ion reaches the detector it generates some electrons, which are multiplied whenever they collide with the multiplier or channeltron. Sufficient current is then produced so that the ion can be counted as a single pulse.

**3.4 medium intensity region**

intensity region where the major isotope intensity is still linear and the minor isotope intensity is sufficiently higher than background

**3.5 saturation**

phenomenon when too many ions reach the detector at the same time, their pulses are overlapped and the detector cannot separate individual pulses

**3.6 approximate intermediate extended dead time model a-IED**

model for detector *saturation* (3.5)

**4 Symbols and abbreviated terms**

The term “intensity” is used below and elsewhere. This refers to a measurement of count rate in the depth profiles.

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$I_M(t)$	measured intensity at the time $t$ , expressed in counts per second (cts·s <sup>-1</sup> )
$I_{M_{\max}}$	maximum measured intensity, expressed in counts per second (cts·s <sup>-1</sup> )
$I_{M-J}(t)$	measured intensity of the major isotope at time $t$ , expressed in counts per second (cts·s <sup>-1</sup> )
$I_{M-N}(t)$	measured intensity of the minor isotope at time $t$ , expressed in counts per second (cts·s <sup>-1</sup> )
$I_{M-N-P}(t')$	$I_{M-N}(t)$ interpolated at the measurement time of $I_{M-J}(t')$ , expressed in counts per second (cts·s <sup>-1</sup> )
$I_{M-\max}$	maximum reliable intensity without saturated intensity correction, expressed in counts per second (cts·s <sup>-1</sup> )
$I_C(t)$	corrected intensity at time $t$ , expressed in counts per second (cts·s <sup>-1</sup> )
$I_{C-J}(t)$	corrected intensity of the major isotope at time $t$ , expressed in counts per second (cts·s <sup>-1</sup> )
$I_{C-N}(t)$	corrected intensity of the minor isotope at time $t$ , expressed in counts per second (cts·s <sup>-1</sup> )
$I_{C-N-P}(t')$	$I_{C-N}(t)$ which is interpolated at the measurement time of $I_{C-J}(t')$ , expressed in counts per second (cts·s <sup>-1</sup> )
$I_{C-\max}$	maximum reliable intensity after saturated intensity correction, expressed in counts per second (cts·s <sup>-1</sup> )
$I_{C-m-\max}$	maximum reliable measured intensity without intensity correction at $I_{C-\max}$ , expressed in counts per second (cts·s <sup>-1</sup> )
$R_J$	natural abundance of the major isotope
$R_N$	natural abundance of the minor isotope
$R$	practical isotope ratio; isotope ratio ( $R_J/R_N$ ) multiplied by the transmittance differential efficiency, also known as isotope ratio corrected for mass-fractionation
$t_{MM}$	maximum time within the medium intensity region, expressed in seconds (s)
$t_{ML}$	minimum time within the medium intensity region, expressed in seconds (s)
$t_{CM}$	time at $I_{C-\max}$ , expressed in seconds (s)
$N_{Mid}$	number of data points in the medium intensity region
$N_{Cor}$	number of data points for the intensity from the $I_{M-\max}$ up to the $I_{C-\max}$
$\rho$	parameter corresponding to the degree of dead time extension; extension parameter



$I_M(t)$	measured intensity at the time $t$ , expressed in counts per second (cts·s <sup>-1</sup> )
$\tau$	time per pulse for which a pulse-counting system is unavailable for further counting, expressed in seconds (s)
$u(I)$	measurement uncertainty at intensity $I$ , expressed in counts per second (cts·s <sup>-1</sup> )
$v(I)$	relative measurement uncertainty at intensity $I$

## 5 Outline of method

The method is outlined by the flow chart in [Figure 1](#). In this method, depth profiles are measured for a reference material which has two or more isotopes with a gradual concentration change between low and high concentration regimes. The analytical conditions are chosen by the analyst at Step 6.3 to provide secondary ion intensities in a given period of time in the linear and nonlinear ranges of detector ion counting. The depth profiling is performed at Step 6.4. For the depth profiles,  $R$  and  $I_{M-\text{Max}}$  are determined at Steps 6.5.2 and 4.5.3. Then the saturated intensities are corrected with an approximate intermediate extended dead time (a-IED) model at Step 6.5.4 and the uncertainty of the corrected intensities is evaluated at Step 6.5.5.

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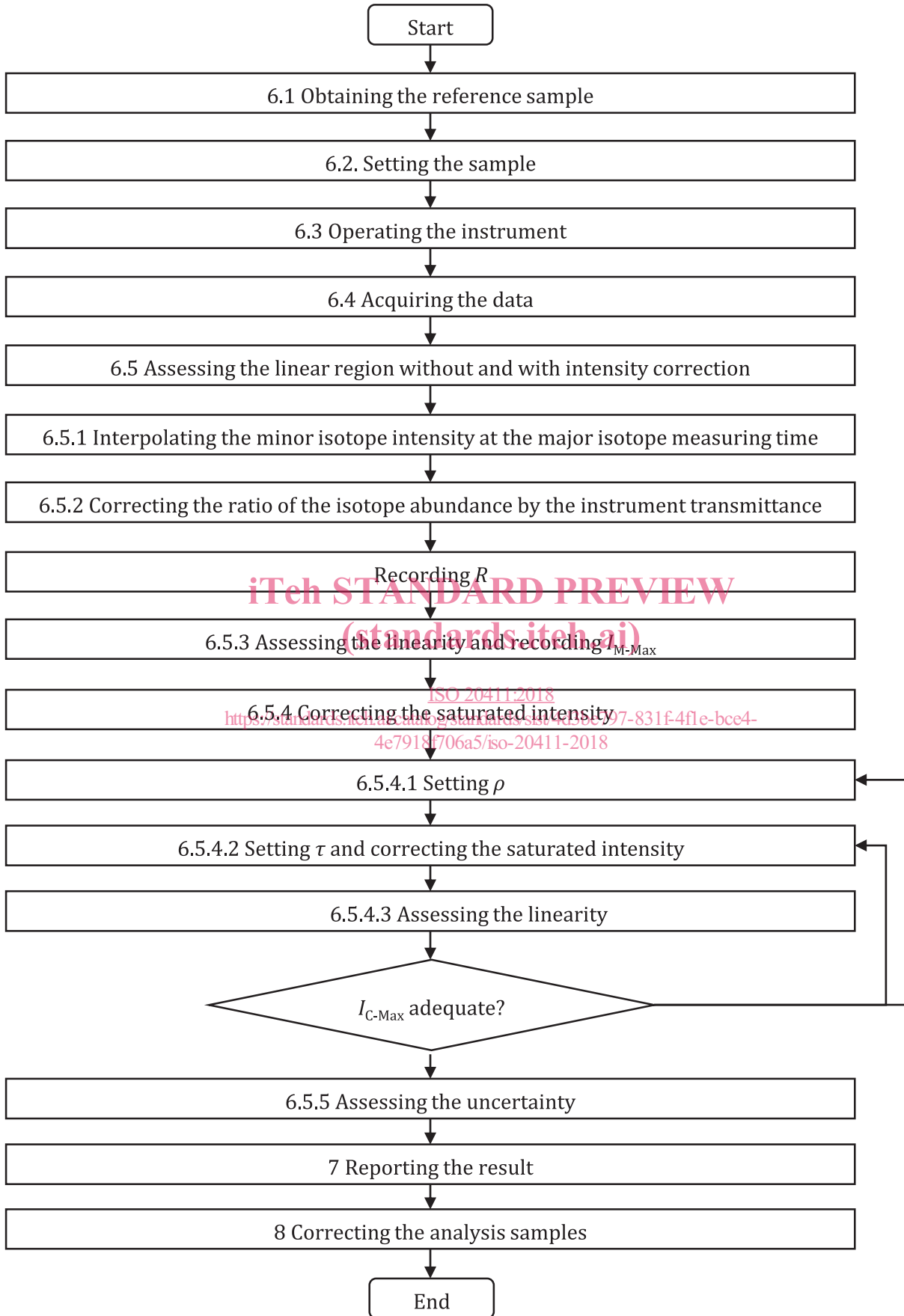


Figure 1 — Flow chart of the work. The numbers refer to the relevant subclauses

## 6 Procedure for evaluating intensity linearity

### 6.1 Obtaining reference sample

For the calibration of dynamic SIMS spectrometers, obtain a reference material whose signal level for the species of interest shall change gradually in a wide range from sufficiently high to low at the same analysis condition for the specimen. The reference material shall have a concentration gradient such that more than 10 data points can be obtained while the minor isotope intensity changes by a factor of 10 in a region where the major isotope intensity is getting saturated. The reference material shall contain two or more detectable isotopes whose intensities shall differ by a factor of 4–500. The analysed surface shall be as flat as that of a Si wafer. The concentration shall be uniform laterally.

### 6.2 Setting the sample

The reference material shall be set onto the sample holder with clean tweezers as described in the manufacturer's instructions or a local documented procedure. If there are some particles on the surface, these shall be blown off with dry nitrogen or clean air. Then the sample holder with the reference material shall be loaded into the analysis chamber.

### 6.3 Operating the instrument

#### 6.3.1 Setting the ion beam and the mass analyser

The condition of the ion beam and the mass analyser shall be set to be able to measure depth profiles in accordance with the manufacturer's instructions or local documented instructions.

In case the analysis area is limited by the selected area apertures, the gross intensity shall be corrected by the ratio of the analysis area to the sputtering area, because the nominal detecting time is affected by this ratio.

It is recommended that the analysis condition for the reference material be the same as that of the species of interest. Even though the intensity is corrected by the analysis/sputtering area, the dead time could change as the analysis condition, especially the analysis area or primary ion diameter, changes. This is considered to be due to the non-uniformity of transmittance in plane.

#### 6.3.2 Setting the charge compensation

If charge compensation is needed, the charge compensation condition shall be set in accordance with the manufacturer's instructions or local documented instructions.

#### 6.3.3 Setting the ion detector

Check that the detector system settings are correctly adjusted. The multiplier gain shall be set at the plateau on the detector gain versus the intensity curve, and the threshold of the amplifier shall be set so that the background is sufficiently low. The optimizing procedure of the detector system shall be done according to the manufacturer's instructions or local documented instruction.

### 6.4 Acquiring the data

At least two different isotopes of the reference material shall be depth profiled in accordance with the manufacturer's instructions or local documented instructions. The isotope ratio shall differ by a factor of 4–500. Each isotope to be analysed shall have sufficiently low background to avoid any mass interferences. If the major isotope intensity does not reach a saturation condition, another reference material with a higher concentration shall be prepared or the analysis condition shall be changed so that the intensity of the major isotope reaches a saturation condition.

More than 10 data points shall be obtained while the minor isotope changes by a factor of 10 in a region where the major isotope intensity is becoming saturated. If the number of data points is not sufficient,