BS ISO 17862:2013

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

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National foreword

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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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Foreword

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The committee responsible for this document is 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), 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.[1] 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 can or cannot already be available in the instrument's data capture or processing computer. In this International Standard, 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 International Standard 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 exacerbated by unwanted background in the spectra.

This International Standard 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 [1]. The results from applying this International Standard 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 International Standard 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 six monthly 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 International Standard 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. This International Standard can also be used to confirm the validity of instruments in which the dead-time correction is already made but in which further increases can or cannot be possible.

2 Symbols and abbreviations

2.1 Abbreviated terms

For the purposes of this International Standard, the following abbreviations are used:

MCP microchannel plate

PTFE poly(tetrafluoroethylene)

SIMS secondary ion mass spectrometry

TDC time-to-digital converter

TOF time-of-flight

2.2 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 $F_P(i,j)$

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

i index number for ion pair listed in Table 1

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i_{max} highest primary ion current used for the saturation analysis

I_M integrated measured secondary ion intensity of a specified SIMS peak

$I_M(X)$ integrated measured secondary ion intensity of the SIMS fragment X

I_P integrated corrected secondary ion intensity of a specified SIMS peak

$I_P(X)$ integrated corrected secondary ion intensity of the SIMS fragment X

j index number for spectrum in the measurement series

k index number for setting the different primary ion currents

L_P shorthand for $L_P(i,j)$

$L_P(i,j)$ ratio of $F_P(i,j)$ to the product $\alpha(i)$ and $\beta(i)$

L_M shorthand for $L_M(i,j)$

$L_M(i,j)$ ratio of $F_M(i,j)$ to the product $\alpha(i)$ and $\beta(i)$

L_M^T theoretical ratio of measured and corrected intensities per pulse

n number of raster frames used to generate each SIMS intensity

N total number of primary pulses used to generate the SIMS spectrum

R length of the raster side used to generate each SIMS intensity

V_{E} mass analyser desired energy acceptance, in eV

V_{R} mass analyser reflector voltage referred to the sample potential

V_{T} mass analyser reflector voltage referred to the sample potential for a secondary ion intensity to fall to half the maximum intensity

$\alpha(i)$ expected isotope ratio of the ith $^{12}C_xF_y^+$ and $^{13}C^{12}C_{x-1}F_y^+$ secondary ions in Table 1

$\beta(i)$ scaling factor to correct $\alpha(i)$ for the measured data, found by fitting

τ detection system dead-time

x number of ^{13}C or ^{12}C atoms in the characteristic PTFE secondary ion

y number of F atoms in the characteristic PTFE secondary ion

3 Outline of method

The method is outlined by the flow chart shown in Figure 1. In this method, secondary ion spectra are measured for PTFE tape analysed in the "as received" condition with no in-house cleaning and no further sample preparation as described from 4.1 to 4.3. The analytical conditions are chosen by the analyst as described in 4.4 to provide secondary ion intensities per pulse in the linear and nonlinear ranges of detector ion counting. This is established using 16 test spectra for a test sample to define the correct range of primary ion beam current settings and 16 data spectra are then measured for the analysis sample to provide data for the linearity establishment. In order to ensure that the instrument is operating in the best condition for linearity, considerations for setting the ion beam, the mass analyser, the charge compensation, and the ion detection system are described from 4.5.2 to 4.5.5. PTFE is a bulk insulator and requires charge neutralization.

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The spectrometer should be operated under conditions that give the most stable performance. It is recommended that analysts use ISO 23830[2] to confirm the repeatability of their instrument. The protocol described in this International Standard is closely aligned with that in ISO 23830 and those using that International Standard are already familiar with much of the procedure given here.

The acquisition of data is described in 4.5 and details of the peaks to be measured are given in Table 1. The behaviour expected is described in 4.6 with relevant formulae. If the linearity is adequate, either for the data directly or for the data after correction using the instrument's data capture computer, the work is complete until, through changes to the instrument or the passage of time, a repeat of this International Standard is required. If the linearity is inadequate, and if the instrument follows the predicted behaviour, a correction can be made as described in 4.6.5 which can extend the linear range by a factor of more than 50. The work is now complete until, through changes to the instrument or the passage of time, a repeat of this International Standard is required. Finally, if the linearity is inadequate and if the instrument does not follow the predicted behaviour, annexes are provided to indicate how to improve the matters. These can improve the linearity range to the extent that this is possible with the equipment being used. This can or cannot be the full range expected for ideal equipment but should lead to some improvement.

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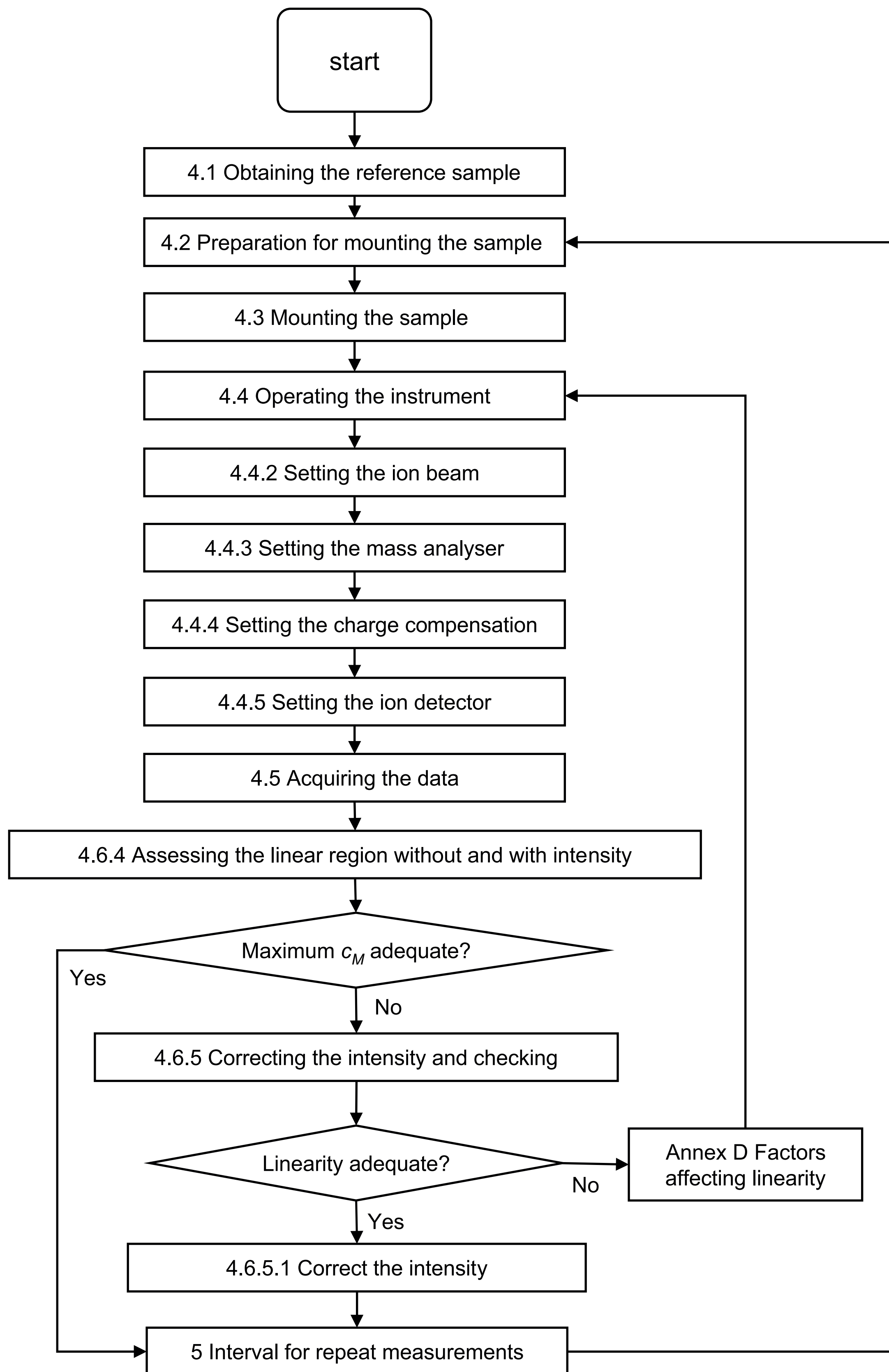

NOTE The numbers refer to the relevant subclauses.

Figure 1 — Flow chart of the work

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4 Procedure for evaluating the intensity linearity

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 and keep this reel with your reference samples.

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

4.2 Preparation for mounting the sample

Samples shall only be handled with clean, uncoated stainless steel 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.

NOTE This International Standard uses the intensity ratio of natural ^{12}C and ^{13}C isotopes to determine the linearity. For the isotope ratio method to be successful, it is important that the isotope peaks can be measured without any significant background from peak interferences. Since most TOF-SIMS instruments do not have sufficient resolution to completely separate between a fragment with ^{13}C and the peak interference with ^{12}CH, it is important to have no hydrogen in the reference material and a low surface energy so that hydrocarbon contamination is minimized. PTFE has both of these crucial attributes and, importantly, is very easy to obtain and use.

In selecting gloves, care should be taken to avoid those with talc, silicone compounds, or similar contaminants. “Powder-free” gloves have no talc. Coated stainless steel or other tweezers can cause unwanted contamination.

4.3 Mounting the sample

4.3.1 To manipulate the samples, the gloves are used to hold the tweezers and not the sample. Avoid any wiping materials, sometimes used to handle samples, as they can result in unwanted contamination of the sample surface. Unnecessary contact of the sample with the gloves shall be avoided. Sample mounts and other materials used to hold samples shall be cleaned regularly whenever there is a possibility of cross-contamination of samples. The use of tapes containing silicones and other mobile species shall be avoided.[3]

4.3.2 Remove and discard the first 20 cm of the 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 1 Common mounting systems include metal plates with holes of various sizes and metal grids. The grid often helps if severe charging is experienced.

NOTE 2 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.

A repeat of this International Standard is required later in Clause 5. For this, a fresh sample is required and for consistency, the sample should be from the same reel.

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4.4 Operating the instrument

4.4.1 General

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 (see 4.4.5). In some instruments, the issue of count rate nonlinearity is dealt with by providing the user with a warning that a certain count rate should not be exceeded. This can limit the count rate to below, say 0,1 counts per pulse. It can be possible to correct the nonlinearity to significantly higher count rates using the present procedure such that a much higher dynamic range is possible, enabling the work of a higher quality to be achieved in a shorter analytical time. Doing so can require this warning to be ignored. If the warning is ignored, ensure by checking in the instrument manual or by contact with the manufacturer that it is safe to do this.

4.4.2 Setting the ion beam

4.4.2.1 In this International Standard, the primary ion current is to be varied to provide secondary ion intensities per primary ion pulse that range from the linear regime to highly nonlinear regime. A measurement of the ion beam current is not required since ^{12}C and ^{13}C isotope ratios are used. If many ion sources are available, use the one generating the highest intensities for the $^{12}CF_3^+$ peak from PTFE. It does not matter if it is an atomic primary ion or a cluster primary ion, so long as the beam current can be adjusted to give a large range of secondary ion intensities and detector saturation as discussed in 4.5.4.

4.4.2.2 In setting the primary ion beam to provide a range of currents on the target sample, it is important that the following conditions are satisfied.

(a) The pulse width and peak shape do not change drastically at different current settings.

(b) The peak width is much smaller than the dead-time of the detector (determined following 4.5.10 but is usually –50 ns).

(c) The selected C_xF_y peaks (Table 1) do not suffer from mass interference.

The primary ion current can be easily adjusted by altering the alignment of the ion beam through the internal apertures of the ion beam column using deflection voltages. Other methods can cause changes to the pulse timing or mass calibration.

4.4.2.3 The ion beam should be centred in the acceptance area of the mass spectrometer as well as possible. To do this, first, centre all the alignments for the mass analyser and then increase the ion beam raster area to image the entire mass analyser acceptance area. Using the ion beam controls, centre the mass analyser acceptance area in the imaged area. In some cases, the maximum field of view cannot be large enough to observe the acceptance area. If the software restricts it, it can be possible to change the calibration scaling or "sensitivity" to access a larger raster area and then return to the calibrated conditions after centring.

4.4.2.4 A maximum ion fluence of 1×10^{16} ions/m^2 is recommended for each measurement. A typical ion beam raster area for this work is 200 µm × 200 µm but can be increased to 400 µm × 400 µm to satisfy the fluence requirements given generically by Formula (A.2) with illustrative numbers and where R is the length of the raster side. For example, a 0,5 pA pulsed beam and a 200 µm square raster requires 128 s acquisition time. This beam would need to be defocused to a diameter greater than 3,1 µm for a 128 × 128 pixel display. If it can only be defocused to 1 µm, a 256 × 256 pixel display shall be used or the local fluence maximum on a pixel will be exceeded by more than a factor of 2. During a digital raster scan, an over-focused ion beam will drill a matrix of small holes. For this reason, a large diameter defocused beam is required. The precise minimum beam size depends on the instrument used but can be evaluated using Formula (A.1).

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4.4.2.5 The number of frames acquired, *n*, should be kept above 20, as described in Formula (A.4), so that the final frame, which most likely will not be a full frame, represents only a small fraction of the data. It is recommended that a random raster pattern is used to minimize local sample charging, if available.

4.4.2.6 For good statistics, the integrated spectrum over the whole analysis area is required.

4.4.3 Setting the mass analyser

4.4.3.1 Choose the spectrometer operating settings which provide high intensities and for which the linearity is to be determined. Select settings to measure positive secondary ions.

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

4.4.3.2 In general, the mass analyser should be operated in the condition that gives the most stable and repeatable performance with good, high mass sensitivity. A suggested method for analyser deflector alignment, if required, is as follows. With the primary ion beam centred in the analysis area on the PTFE sample, reduce the raster area to 50 µm × 50 µm. The analyser deflectors should now be systematically adjusted, one set at a time, to maximize the mass resolution. This alignment process will need to be iterated by the number of analyser deflector pairs. If the analysis area moves over the sample surface during this process, re-centre the image using the primary ion beam controls.

4.4.3.3 For reflection instruments, a systematic method for determining the surface potential, in order to set the reflector voltage, is required. The effect of reflector voltage on the peak intensity of the CF_2^+ ion from PTFE is shown in Figure 2. As the reflector voltage is made more positive, the peaks move down the apparent mass scale, because the ions are reflected earlier and their flight time is reduced. The reflector potential at which the ion signal begins to increase rapidly is approximately equal to the sample potential. In the example shown in Figure 2, the sample potential is approximately −79 V and depends on the sample thickness and dielectric constant. As the reflector voltage is increased positively, more of the secondary ion energy distribution is reflected to the detector and the signal rises to a plateau. The reflector voltage that gives an ion signal of half the maximum peak intensity, V_T, is accurately and quickly measured. The operating reflector voltage, V_R, is then set in a reproducible way by adding a further V_E volts, where V_E is the desired energy acceptance. Here the half maximum intensity occurs at −75 V and a reflector voltage of −55 V is effective for an energy acceptance of 20 eV.

4.4.3.4 After setting the reflector potential as that in 4.4.3.3, the reflector potential might need further minor adjustment to ensure that strong metastable peaks do not overlap the weaker ^{13}C isotopic peaks identified later in Table 1.

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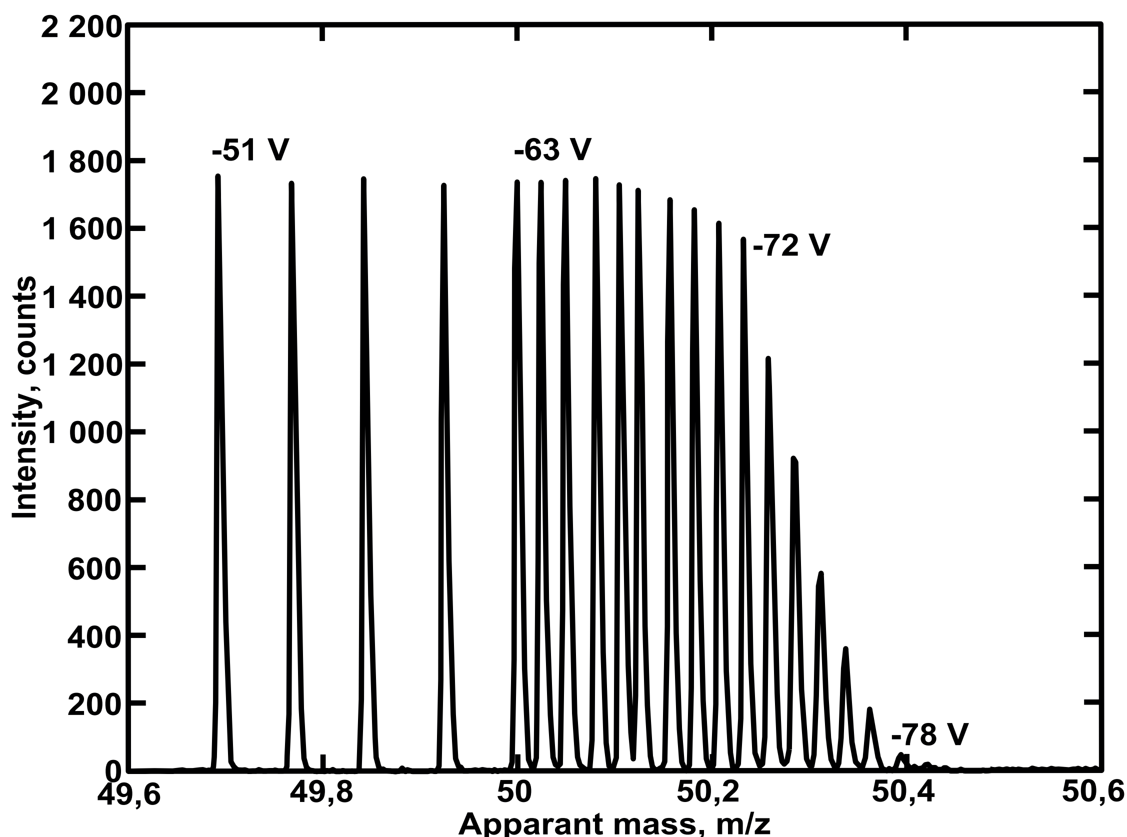

Figure 2 — The effect of reflector voltage on the CF_2^+ peak intensity from PTFE

4.4.4 Setting the charge compensation

Charge compensation is necessary for this study. A detailed study of electron beam damage[4] provides recommendations which are presented in Annex B in order to be able to set the electron flood neutralizer sufficiently low in current not to cause electron damage but sufficiently high to be effective.

NOTE Insufficient charge compensation can affect the linearity as described in Annex D.

4.4.5 Setting the ion detector

A detailed study of ion detection efficiencies for microchannel plates[5] provides recommended operating conditions that are presented in Annex C. Use the detector post-acceleration voltage that is customary for the work. Set the voltage of the microchannel plate using usual procedures; further guidance is given in Annex C and Reference [5].

NOTE Inadequate detector efficiencies can affect the linearity as described in Annex D.

4.5 Acquiring the data

4.5.1 Be prepared to analyse a fresh area of material each time with a total fluence of less than 1×10^{16} ions/m^2 (1×10^{12} ions/cm^2) from an array of 16 measurement positions as shown in Figure 3. Here, the example is for a sample holder with an 8 mm × 8 mm square aperture. All analysis areas should be at distances greater than 1,5 mm from the edge of the sample holder. For a square raster of side R, the recommended repeat distance (centre to centre) is 2,5 R. With the recommended raster area R = 200 µm, the minimum gap between each raster is 300 µm. Record these settings.

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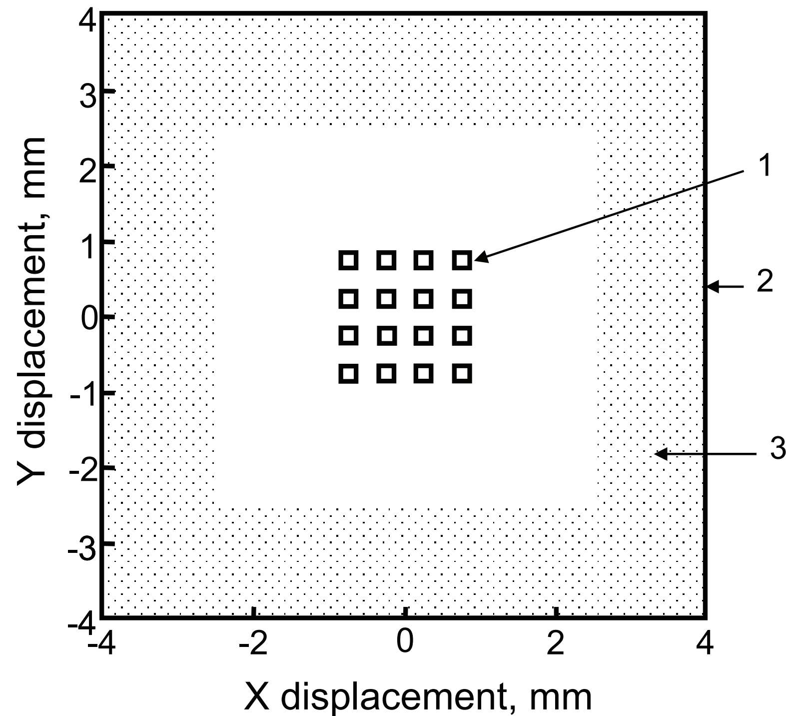

Key

1 example array

2 sample holder aperture

3 avoid this zone

NOTE A zone 1,5 mm from the perimeter is identified (dotted zone) where data should not be acquired.

Figure 3 — Schematic of 4 × 4 array of measurement positions for a sample holder with a square aperture of side 8 mm

4.5.2 Mount two PTFE samples as described in 4.3. One is for setting the range of primary ion currents required called the “test sample”, the other, for the analysis using those settings, called the “analysis sample”.

4.5.3 Switch on the charge neutralization. The charge neutralization system can comprise an electron (flood) gun or a combined electron gun and low energy ion gun. Use whatever system is recommended by the instrument manufacturer or the instrument operating manual.

4.5.4 From the test sample, acquire a positive ion spectrum using the primary ion current that would usually be used for analytical conditions. Next, establish 16 settings of the primary ion current that provide spectra that, at the highest current, i_{max}, give ion intensities that lead to strong saturation of the detector. The remaining 15 currents are each at approximately $(k/16)i_{max}$, where k = 1 to 15. Three example spectra for the CF_3^+ peak, covering this range, are shown in Figure 4. The spectra shown are normalized to a constant intensity for the $^{13}CF_3^+$ peak.

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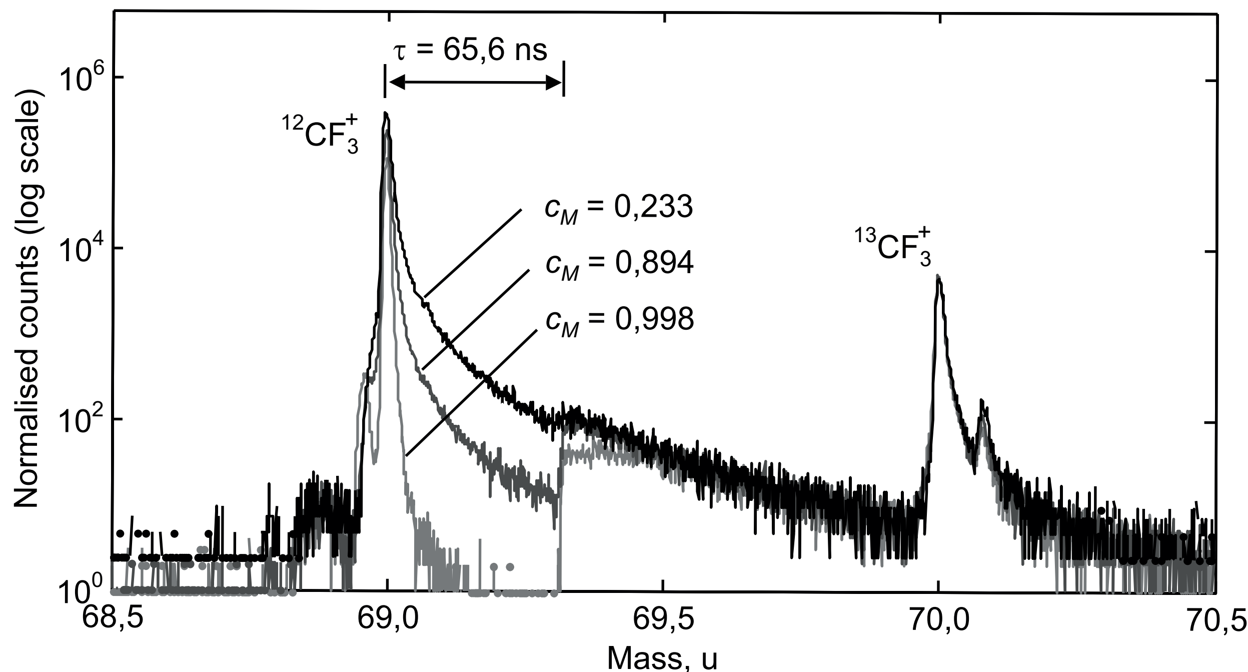

Figure 4 — Details of the $^{12}CF_3^+$ peak and $^{13}CF_3^+$ peak for spectra obtained using a range of primary ion currents

NOTE The line identifiers in the boxes show the integrated intensity of the $^{12}CF_3^+$ peak in units of c_M (secondary ion counts per primary ion pulse). The intensity of the most saturated (highest c) spectrum is presented in its measured counts on the logarithmic intensity scale, and the other spectra are normalized to the same $^{13}CF_3^+$ intensity. The dead-time, τ, of 65,6 ns is marked for the saturated peak.

4.5.5 The secondary ion peak intensities to record are given in Table 1. For the low ion beam currents, it might be necessary to increase the acquisition time so that the weakest peaks (i.e. those containing ^{13}C isotopes) have > 1 000 counts. The weakest peak is likely to be the $^{13}C^{12}CF_5^+$ peak.

Table 1 — Characteristic PTFE isotopic secondary ion intensities to be measured, showing their exact masses and the expected isotope ratios $\alpha(i)$ calculated using the natural abundance of ^{13}C

i	$^{12}C_xF_y^+$ ion	Mass u	$^{13}C^{12}C_{x-1}F_y^+$ ion	Mass u	Expected isotope ratio $\alpha(i)$
1	$^{12}CF^+$	30,998 4	$^{13}CF^+$	32,001 8	90
2	$^{12}CF_3^+$	68,995 2	$^{13}CF_3^+$	69,998 6	90
3	$(^{12}C)_3F_3^+$	92,995 2	$^{13}C(^{12}C)_2F_3^+$	93,998 6	30
4	$(^{12}C)_2F_5^+$	118,992 0	$^{13}C^{12}CF_5^+$	119,995 4	45
5	$(^{12}C)_3F_5^+$	130,992 0	$^{13}C(^{12}C)_2F_5^+$	131,995 4	30

NOTE The expected isotope ratio varies with the number of carbon atoms in the secondary ion fragment. The natural abundance of ^{13}C is around 1,1 % of that of ^{12}C.

4.5.6 For the analysis of the linearity of the intensity scale later in this International Standard, it is the measured counts per primary ion pulse, c_M, that is important rather than the total ion counts. Use Formula (A.5) or (A.6) to confirm that the selected primary ion currents provide a range of values of c_M, for the CF_3^+ peak, between approximately 0,1 and 0,99 counts per pulse. If this is not the case, adjust the primary ion beam current and repeat 4.5.4 until the condition is satisfied.

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4.5.7 Check that no major metastable peak overlaps with the peaks in Table 1. If there are, adjust the reflector voltage to remove them.

4.5.8 Move to the analysis sample. For each of the 16 settings of primary ion current determined in 4.5.4, acquire a positive ion spectrum from a fresh area of sample. Select the acquisition time to ensure the counts for the $^{13}C(^{12}C)F_5^+$ peak exceed 1 000 and the maximum dose is < 1×10^{16} ions/m^2.

4.5.9 Calibrate the mass scale for each spectrum using the instrument calibration system or ISO 13084. [6]

4.5.10 Measure the dead-time, τ, of the spectrometer from an overlay plot of the $^{12}CF_3^+$ peak. If your software allows, it is helpful to normalize the spectra to the peak height of the $^{13}CF_3^+$ peak. Identify a spectrum similar to the one labelled in Figure 4 with c_M value of 0,894. Here, the signal is reduced after the peak but does not go to zero and, after some time, τ, recovers. Measure the value of τ. It is usually possible, in the software, to switch between a mass scale, a time scale, and sometimes a channel number. To convert from channel number to time, simply multiply the channel number by the channel width (usually around 200 ps). In this example τ = 65,6 ns. Record the value of τ.

4.5.11 For some instruments, the background signal can be too low to determine the dead-time of the detector using the method in 4.5.10. If this is the case, proceed by using the manufacturer's specified dead-time value.

4.5.12 Integration limits are now set for measuring the peak intensities. This is usually done by establishing a peak list in the instrument analysis software. For each peak in Table 1, identify the masses equivalent to τ ns less than, and τ ns more than, the peak centre. Set these as the peak integration limits. Some weak metastable peaks and metastable background intensity is included in the integration. This generally does not matter if they are relatively weak. It is important that the intensity is summed for at least τ ns after the peak.

4.5.13 Measure the intensities of the 10 peaks listed in Table 1 for the 16 spectra without any software dead-time correction.

4.5.14 Measure the intensities of these 10 peaks with instrument analysis software dead-time correction, if available.

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4.6 Checking the linearity

4.6.1 The relation of corrected and measured counts

For a single SIMS peak where the peak width is smaller than the dead-time τ (usually around 30 ns to 70 ns), and where the peak is not preceded by another peak within the time τ, the intensity correction simplifies to[7]

$$\frac{I_P}{N} = -\ln\left(1 - \frac{I_M}{N}\right) \tag{1}$$

or

$$c_P = -\ln(1 - c_M) \tag{2}$$

where

I_M is the integrated measured intensity of the SIMS peak;

I_P is the corrected intensity;

N is the total number of primary ion pulses used to generate the spectrum;

$c_M = I_M/N$ is the measured count per pulse intensity;

$c_P = I_P/N$ is the corrected count per pulse intensity.

Note that with these assumptions, c_M has a maximum value of unity.

Since Formula (1) is not a function of the dead-time τ, it should properly be called a "Poissonian correction". However, to be consistent with common usage, in this International Standard, this is referred to as "dead-time correction". Formula (1) is deduced for a Poissonian flux in which I_M is the measured intensity and I_P is the true intensity. It scales the measured secondary ion count rate per pulse, c_M, so that the corrected count rate per pulse c_P is proportional to the actual number of secondary ions impinging the detector. It is routinely applied to SIMS data and has been demonstrated to be very successful for many data sets.[7][8][9] From Formula (1), dead-time correction is required to maintain 95 % linearity between the measured intensity and the true (corrected) intensity when the value of c_M exceeds 0,1.

NOTE Formula (1) is for an intensity measured under steady-state conditions in which the counts follow pure Poissonian statistics. If the intensity is summed or averaged over a period in which the beam is rastered, deviations can occur arising from one of many instrumental or sample related factors that affect the constancy of the signal. Examples include sample heterogeneity, charging, variation in instrumental sensitivity with position in the raster, beam current fluctuations and sample damage, and those that are discussed in Annex D.

4.6.2 The measured ratios for isotopes

Calculate the linearity of the intensity scale as follows. Where applicable, the subscript M denotes quantities calculated using measured intensities and subscript P denotes quantities calculated using dead-time corrected intensities. The linearity, L, is defined such that L is exactly unity for an unsaturated spectrum, and $L < 1$ for a spectrum where the more intense $^{12}C_xF_y^+$ peak begins to saturate relative to

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the much weaker $^{13}C^{12}C_{x\text{-}1}F_y^+$ peak. For measured peak intensities without dead-time correction, the theoretical linearity L_M^T assuming single ion counting theory[7] is from Formula (1).

$$L_M^T = \frac{I_M}{I_P} = \frac{c_M}{-\ln(1-c_M)} \tag{3}$$

Next are defined as the isotope ratios for measured intensities, F_M, and the isotope ratios for corrected intensities, F_P.

$$F_M = \frac{I_M(^{12}C_xF_y)}{I_M(^{13}C^{12}C_{x-1}F_y)} \tag{4}$$

$$F_P = \frac{I_P(^{12}C_xF_y)}{I_P(^{13}C^{12}C_{x-1}F_y)} \tag{5}$$

For each pair of isotope ions (index i, identified in Table 1) and each spectrum acquired (index j), the linearity before and after dead-time correction, L_M and L_P, are then calculated from F_M and F_P, as follows

$$L_M(i,j) = \frac{F_M(i,j)}{\alpha(i)\beta(i)} \tag{6}$$

$$L_P(i,j) = \frac{F_P(i,j)}{\alpha(i)\beta(i)} \tag{7}$$

Here, for each isotopic pair i, $\alpha(i)$ is the expected isotope ratio and $\beta(i)$ is a scaling factor that is determined by fitting.

NOTE 1 The true isotopic ratio of the sample does not need to be known since any difference is accounted in the β scaling parameter.

NOTE 2 The scaling factor β is necessary because experimental SIMS intensities might not accurately reflect the expected isotope ratio α for three reasons. First, α varies naturally depending on the source of the carbon. Second, the existence of background intensities, caused by metastable ions or contamination, can contribute differently to different isotopic peaks. Finally, the sputtering and ionization efficiencies are generally not constant for different isotopes of the same element and the degree of difference can vary with experimental conditions.

4.6.3 Fitting the data

For each pair of isotope ions i, $\beta(i)$ is calculated by the least squares fitting of L_M to the theoretical ratio L_M^T. Plot L_M from Formula (6) and L_M^T from Formula (3) versus c_M as shown in Figure 5(a). The fitting is carried out over the range of c_M where the data are in good agreement with the theory, thus, data with very low c_M are often excluded due to the low signal-to-noise ratio, and data with c_M approaching unity are often excluded as they can begin to deviate from the theory. The range of c_M used to calculate $\beta(i)$ is determined separately for each instrumental condition (if more than one is being studied) but this range is the same irrespective of the fragment i. For consistency, the same value of $\beta(i)$ is then used in the calculation of dead-time corrected linearity L_P shown in Figure 5(b).

NOTE 1 In using a constant value of $\beta(i)$, it is assumed that the fractional contribution of background intensities is different for each peak i, but for the same peak i, it is the same regardless of the spectrum j.

NOTE 2 In an interlaboratory study,[1] $\beta(i)$ averaged 1,06 and, for all participants, was between 0,9 and 1,2.

4.6.4 Assessing the linear region without and with any instrumental intensity correction

4.6.4.1 On a plot of L_M from Formula (6) versus c_M for assessment without the instrumental intensity correction, and L_M^T from Formula (3) versus c_M, draw horizontal bounds representing the linearity tolerance. Note the range of c_M acceptable within this tolerance.

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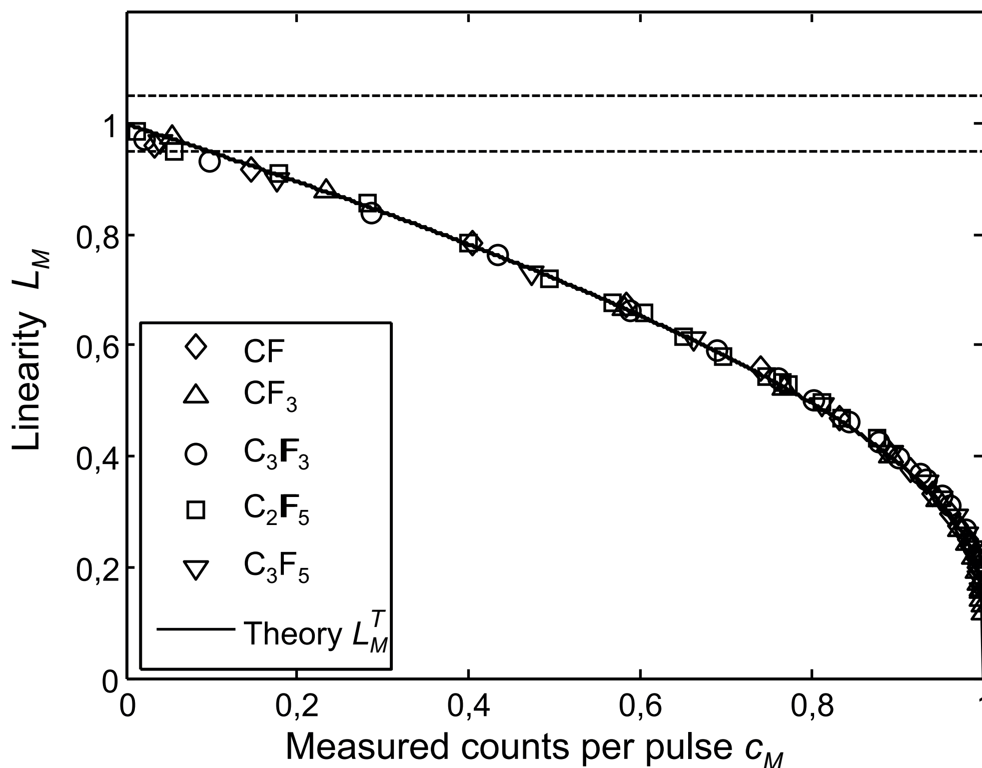
NOTE An example plot is shown in Figure 5(a) with the tolerance set at 95 %. The acceptable range is up to c_M = 0,098.

4.6.4.2 If the instrument has an intensity correction capability, plot the instrumental value of L_P versus c_M and draw horizontal bounds representing the linearity tolerance. Note the range of c_M acceptable within this tolerance.

NOTE An example plot is shown in Figure 5(b) where the lines are shown for 95 % linearity and the acceptable range is up to c_M = 0,999 8. However, in the uncorrected state, the maximum value of c_M is only 0,098 for this level of linearity.



a) The linearity L_M calculated using measured intensities

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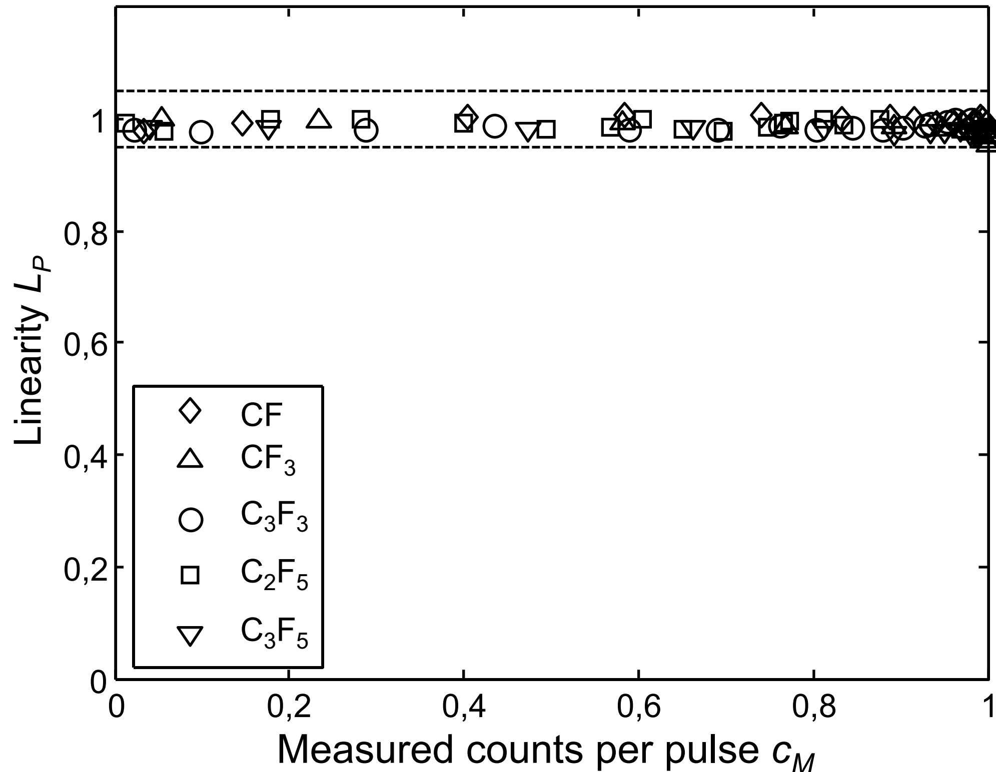

b) The intensity linearity L_P calculated using dead-time corrected intensities

NOTE 1 The theoretical linearity L_M^T using single ion counting statistics is plotted as a solid line.

NOTE 2 The dashed lines show the limits for 95 % linearity for the corrected intensities.[1]

Figure 5 — Example results showing the linearity plotted against the measured counts per pulse c_M of the $^{12}C_xF_y^+$ peak

4.6.4.3 If the value of c_M from 4.6.4.1 or 4.6.4.2 is adequate, go to Clause 5. If not, or if the instrumental correction software is to be compared with the correction available in this International Standard to validate it, proceed to 4.6.5.

4.6.5 Correcting the intensity and checking the validity of any instrumental correction

4.6.5.1 The intensity is corrected using Formula (1) and is valid over the range for which L_M and L_M^T agree and the extent to which they agree. Figure 5a) shows excellent agreement such that the useful measured counts per pulse c_M for 95 % linearity can be increased by a factor of 10. The corrected intensity at the highest limit is then 8,5 counts per pulse, showing an 85-fold improvement over the uncorrected result. Often, agreement between L_M and L_M^T is not this good as shown in Figure 6. In this case, the useful measured counts per pulse for 95 % linearity can only be increased by a factor of 4. The corrected intensity at the highest limit is then 0,5 counts per pulse, showing only a fivefold improvement over the uncorrected result. Annex D shows some of the factors that affect linearity and which can be adjusted to increase the effective operating range of the instrument significantly.

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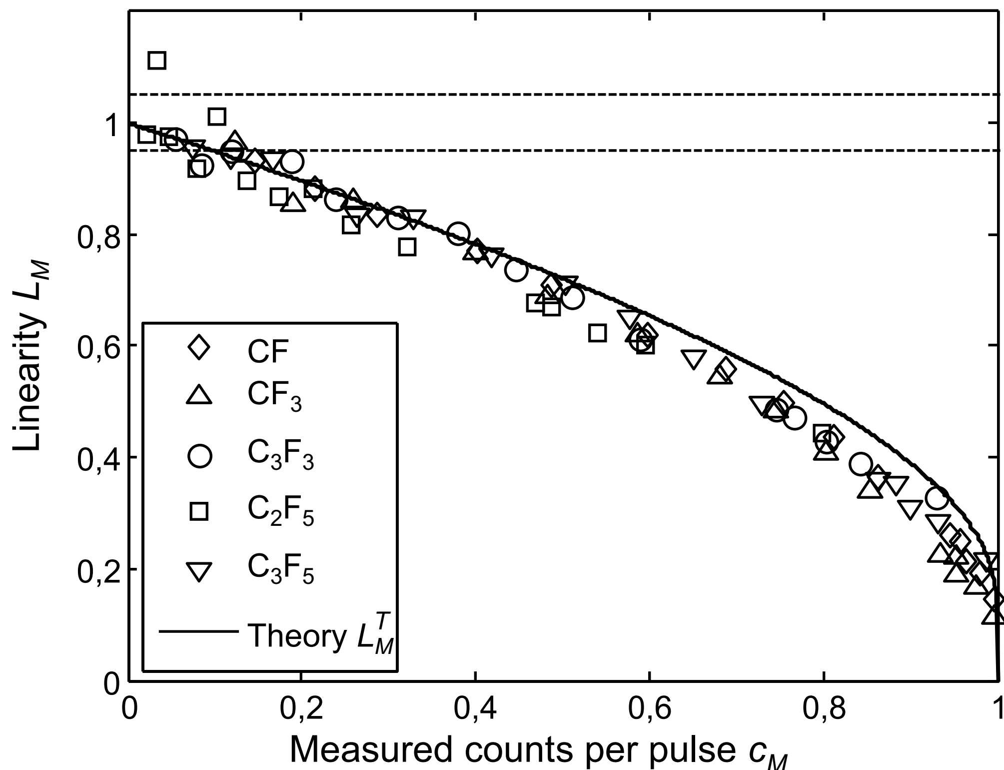
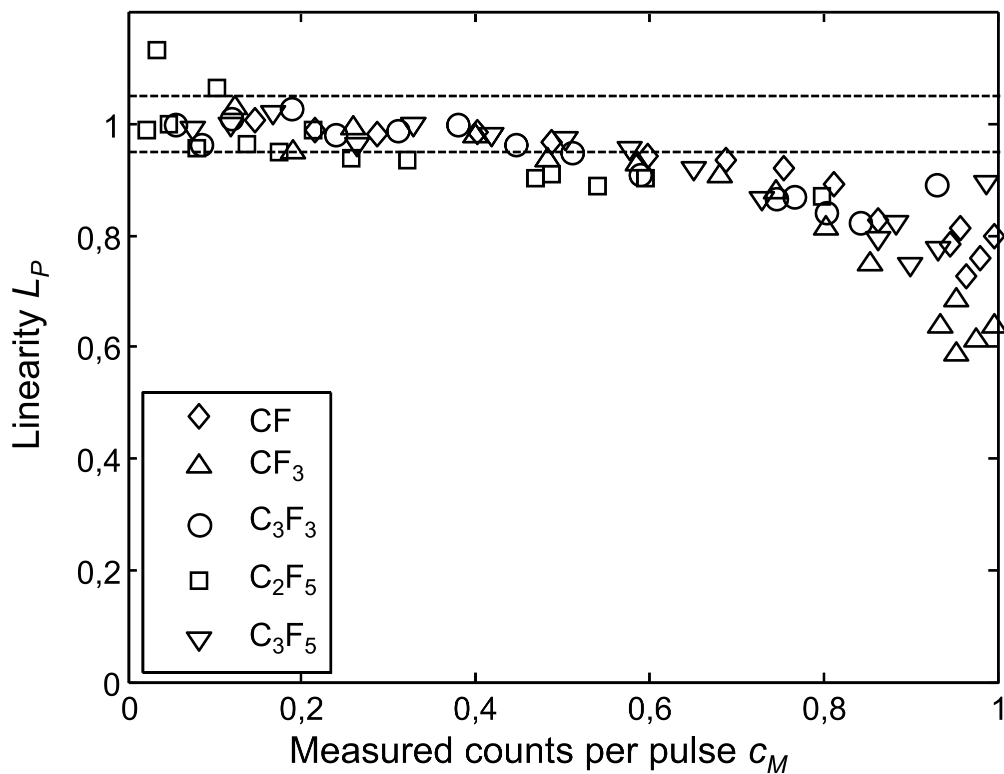


a)



b)

Figure 6 — As Figure 5 but for a poorly adjusted instrument as discussed in Annex D[1]

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4.6.5.2 If adjustments are made, prepare a fresh PTFE sample and repeat 4.5.2 to 4.6.5.1 until an adequate linearity is achieved or until no further improvement is obtained.

4.6.5.3 If the instrument has correction software that is to be compared with the correction available in this International Standard in order to validate it, plot both sets of results in the form of Figure 5b) and note the upper limit of c_M, valid for the level of linearity required, achieved in each case. If the two values are comparable or if the value for the instrumental software exceeds that from this International Standard, the instrumental software is valid.

4.6.5.4 If c_M from the instrumental software is not significantly lower than that generated from this International Standard, the convenience of using the instrumental software usually outweighs the value of these separate calculations; however, the user needs to check, where quantitative data are required, that whatever limit of c_M that is actually valid is not exceeded.

5 Interval for repeat measurements

This International Standard shall be repeated after any substantive modification to the detection circuits, after replacement of the MCP, scintillator, photomultiplier, or other detector components, or at approximately six monthly intervals.

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Annex A
(normative)

Computation of raster size, ion beam current, number of frames for analysis, and counts per pulse

A.1 Symbols

A peak intensity of a selected peak (counts)

c secondary ion counts per primary ion pulse (counts)

d beam diameter (m)

e charge on the electron (C)

F pulse repetition rate or frequency (s^{-1})

i_P pulsed ion beam current (A) (i.e. the time-averaged current with pulsing on)

I DC ion beam current (ions/s) (i.e. the current with the pulsing off)

J total ion fluence (ions/m^2)

M number of pixels along a line of the raster

P total number of primary ion pulses in the acquisition time T (no units)

Q total number of incident ions

R raster size (m)

T total spectrum acquisition time (s)

w pulse width (s)

A.2 Computation

(a) Condition that the beam is sufficiently defocused

$$d > 2R/M \quad \text{(A.1)}$$

(b) Total dose

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$$J = \frac{i_P T}{eR^2} \tag{A.2}$$

For some instruments, i_P is not known and the DC current, I, is recorded instead, then

$$J = \frac{FIwT}{eR^2} \tag{A.3}$$

For non-pulsed instruments, such as magnetic-sector and quadrupole-based systems, use Formula (A.2) but with the pulsed current, i, substituted with the non-pulsed current, I.

(c) Number of frames

The number of complete frames, n, in the acquisition is

$$n = \frac{JeR^2F}{i_P M^2} \tag{A.4}$$

It is recommended that the ion beam current and/or the acquisition time are adjusted so that $n > 20$.

(d) Counts per primary ion pulse

$$c = \frac{A}{P} \tag{A.5}$$

if the total number of pulses is not given by the software use

$$c = \frac{A}{FT} \tag{A.6}$$

A.3 Example values for static SIMS

For a reasonable quality spectrum, $Q = 4 \times 10^8$ incident ions. Then, if the maximum dose, J, is < 10^{16} ions/m^2, from Formula (A.2)

$$R \geq 200\ \mu\text{m} \tag{A.7}$$

To ensure that mass resolution is not too degraded, $R = 200\ \mu$m is recommended. If better counting statistics are required, R is needed to be increased. This can be at the cost of reduced mass resolution. For a 128 × 128 pixel raster, from Formula (A.1)

$$d \geq 3{,}1\ \mu\text{m} \tag{A.8}$$

If the beam cannot be defocused to this extent, M shall be increased. Typically, many systems use a 0,5 pA pulsed ion beam current (i), a repetition rate F of 10 KHz, a square raster of 128 pixels (M), then for the recommended conditions, where $J = 10^{16}$ ions/m^2 and $R = 200\ \mu$m:

$$T = 128\ \text{s} \tag{A.9}$$

and

$$n = 78 \tag{A.10}$$

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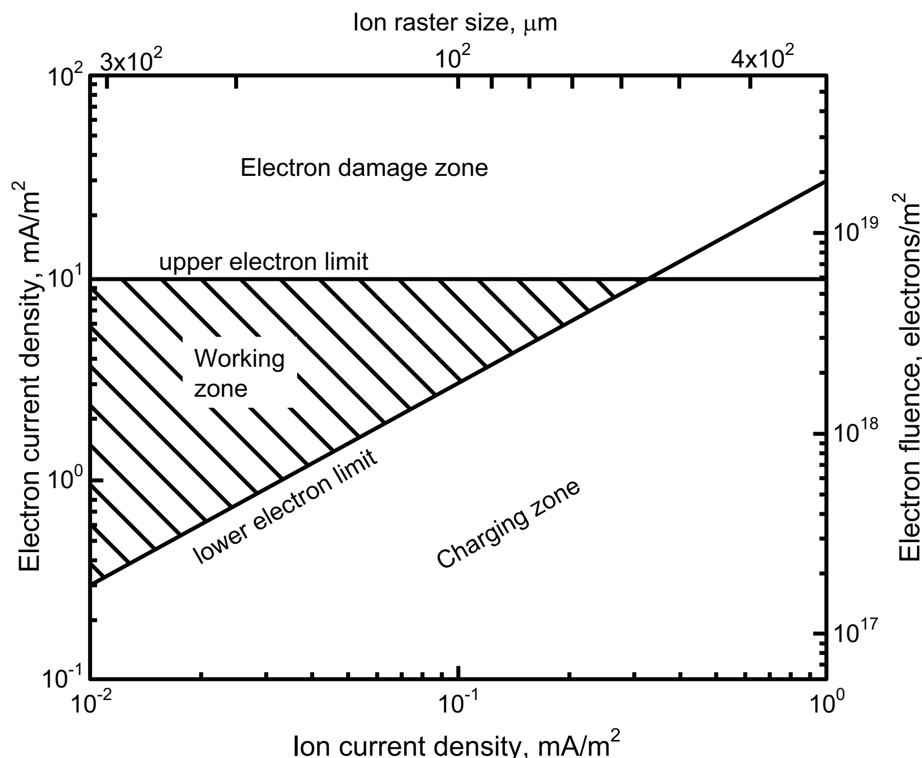
Annex B
(normative)

Charge compensation setting

In general, time-of-flight (TOF) SIMS experiments are considered to be non-damaging to the analysed material. Fluence limits for ion beam damage in polymers have been reported in Reference [10] and most modern equipment readily operates below these guidelines. This limit is typically 1×10^{16} ions/m^{-2}. However, damage caused by low energy electrons from flood guns used for charge stabilization is often overlooked. In a routine static SIMS spectrum of an insulator, an electron fluence of approximately 2×10^{20} ions/m^{-2} can be delivered. While these electrons usually have low energy and a small cross section for damage, there are some 10^4 electrons for every ion per unit area.

A detailed study of the effect of damage on organics and polymers by low energy electrons from flood guns used for charge stabilization in static SIMS is reported in References [4] and [11]. Analysis of the molecular fragmentation for the polymers poly(styrene), poly(vinylchloride), poly(methylmethacrylate), and poly(tetrafluoroethylene) shows that an upper limit of 6×10^{18} electrons/m^{-2} can be defined to retain ion intensities within 1 % of their true levels. At a fluence of $7{,}5 \times 10^{20}$ electrons/m^{-2}, relative signal intensities, for some polymers, have changed by over a factor of four. Recommendations are provided to ensure high repeatability and reproducibility of static SIMS spectra which also allow effective G-SIMS[12][13] spectra to be calculated. These results are summarized in Figure 1. The hatched zone indicates the working range that gives spectra with insignificant charging or damage.



NOTE The upper electron limit is defined by the maximum fluence without significant damage and the lower limit is that required for adequate neutralization. The fluence scale and the dimensions of the square raster are calculated for a 100 s acquisition time with a 1 pA ion beam current.

Figure B.1 — Map of parameters that define conditions to acquire spectra without charging and with low electron damage

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Annex C
(normative)

Ion detector setting

The effect of ion energy, mass, and composition on the detection efficiency of a microchannel plate (MCP) have been studied in detail,[5] using a time-of-flight mass spectrometer. This spectrometer is used for static SIMS although the data are relevant to any ion detection system. A theoretical model is developed which shows how the efficiency falls with increased mass and decreased ion impact energy at the front of the MCP. At an impact energy of 20 keV, the efficiency for the detection of cationized poly(styrene) oligomers of mass 10 000 dalton is approximately 80 % whereas, at 5 keV, it has fallen to around 5 %. The model is extended to estimate the effect of ion composition on the detection efficiency.

Figure C.1 illustrates that ions with high hydrogen content have a lower efficiency than those which consist of a cluster of high atomic number atoms. The spread of detection efficiencies arising from both the effect of composition and mass can be reduced by increasing the ion impact energy, so that, up to a mass of 4 000 dalton, the spread of 100 % that is observed for 5 keV ion impact energy is reduced to a negligible spread for ions of 20 keV impact energy, where the efficiency is approximately unity, independent of the composition. A simple method is developed to determine the correct voltage to operate the MCP for a given efficiency. The transition voltage[14] V_T, which gives 50 % of the plateau efficiency, can be quickly and accurately determined because of the steep efficiency gradient. Values of V_T and the corresponding voltage V_N, to achieve 98 % efficiency, are shown in Figure C.2. The operating voltage, V, is now defined as $1,3(V_N - V_T) + V_T$. For the values in Figure C.2:

$$V = V_T + 29,1 + 0,057\ 2\ \mathrm{m} \tag{C.1}$$

This operating voltage should be determined for the highest mass ions in the required range.

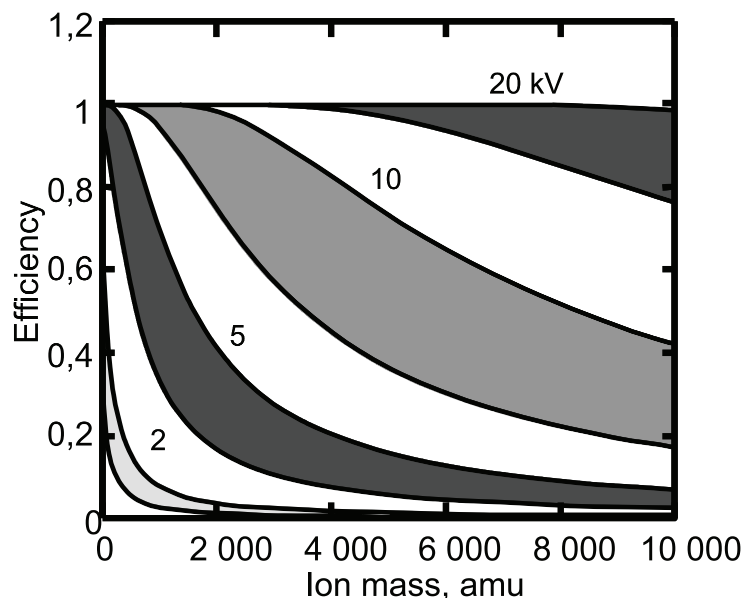

Figure C.1 — Estimated detection efficiency for Cr cluster ions (upper curve) and hydrocarbons (lower curve) for post-acceleration voltages between 2 kV and 20 kV[5]

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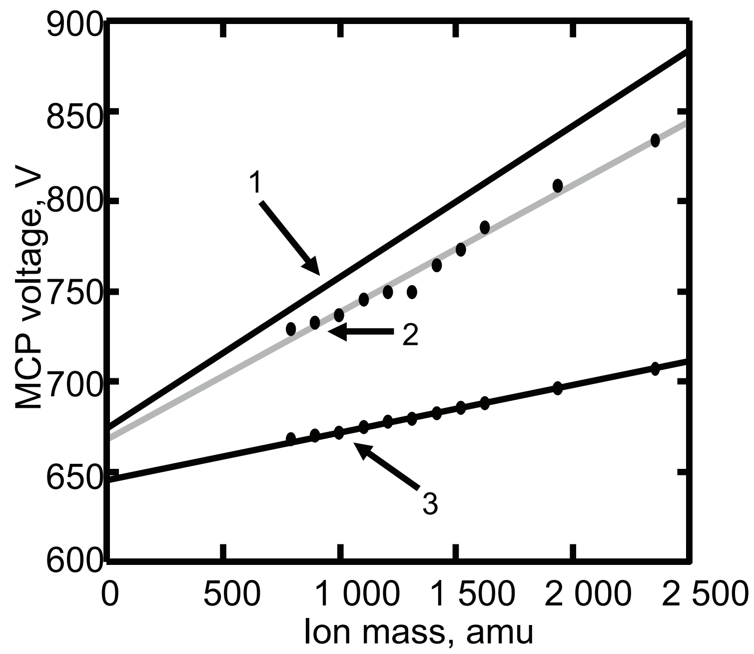

Figure C.2 — MCP operating voltage together with V_T and V_N

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Annex D
(informative)

Instrumental factors affecting linearity

D.1 General

Many instruments achieve excellent linearity of the intensity scale when the correction is applied. However, this is not uniformly the case, and some instruments of the same design and operating parameters show significantly poorer linearity. In an interlaboratory study, no strong correlations were observed between the linearity performance and the instrument model, analysis parameters (i.e. primary ion species, energy, and dose), repeatability of the spectra, or the level of carbon contamination observed (as measured by the relative intensity of the $^{12}CH_3^+$ peak).[1] A number of reasons for the deviations from linearity have been identified, and they are briefly outlined below. This is a non-exhaustive list on the common causes of nonlinearities in order to help analysts with instruments showing poor linearity to diagnose the causes. More details are provided in Reference [1].

D.2 Signal inhomogeneity

Since the dead-time correction using Formula (1) is a nonlinear function of the average counts per pulse c_M, the simple correction needs refinement if the secondary ion intensities vary spatially (e.g. due to an inhomogeneous sample) or temporally (e.g. as a result of sample damage or beam current fluctuations).[7] While a beam current fluctuation of 5 % would give rise to a maximum error of around 2 % in the corrected intensities, the error could be much larger with inhomogeneous samples where the intensities at different pixels can vary by orders of magnitude. For a spatially inhomogeneous sample with a constant signal with time, the proper way to calculate the dead-time corrected intensity for a particular ion is to apply Formula (1) to the ion intensity at each pixel and summing the results. Because of the functional form of Formula (1), this always gives higher corrected intensities than the common (but incorrect) method of applying Formula (1) to the total measured intensity disregarding spatial variations.

One common cause of signal inhomogeneity for the PTFE reference sample is ineffective charge compensation, which can be caused by poor electron gun alignment.[1] This causes a peripheral zone of low intensity in the ion beam rastered area, arising from the difference in the surface potential between the analysed area (which is charging) and the outside areas. This distorts the extraction field which deflects secondary ions from the periphery of the rastered area and prevents them from entering the analyser,[15] causing the secondary ion intensities to be strongly spatially inhomogeneous. As the surface charge builds up, the secondary ion intensities are also likely to be time-dependent. Therefore, as charge compensation becomes inadequate at higher primary ion currents, the apparent $^{12}C_xF_y^+/^{13}CC_{x-1}F_y^+$ isotope ratios calculated for Figure 6 reduces due to the dead-time correction being applied to the total measured intensity rather than to the intensity at each pixel or each scan. This is the cause of the lower linearity at higher primary ion currents shown in Figure 6. To optimize the performance, the electron beam was realigned using the procedure provided by the instrument manufacturer. New data were acquired and the linearity was calculated by applying Formula (1) to the total measured spectral intensity. Before realignment, the linearity was poor at < 0,50 counts per pulse for 95 % linearity after dead-time correction, and after realignment, a much-improved result of 0,90 counts per pulse was obtained. With the data analysis restricted to a region of interest covering the central 25 % of the rastered area, the lower intensity areas at the periphery of the rastered area caused by minor charging were removed, and the linearity improved such that >98 % linearity was achieved with 0,97 counts per pulse.

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D.3 Detector dark counts

Some instruments show deviations from linearity at very low c_M values where detector saturation is not expected to be too significant. These deviations are usually towards lower values of L_M. This can occur when spectra are obtained using low primary ion currents with longer acquisition times. These spectra are prone to low signal-to-noise ratios, current fluctuations, and increased background intensities. While the first two have a random effect on the measured $^{12}C_xF_y^+/^{13}C^{12}C_{x-1}F_y^+$ ratios (and therefore do not affect the linearity measured), the latter effect (increased background counts on both peaks) causes a systematic reduction in the peak ratios, leading to lower calculated values of L_M. Increased background counts can be caused by sporadic dark counts at the MCP detector if, for example, the discriminator voltage is set too low.

D.4 Detector efficiency

The detector efficiency has an effect on the linearity in SIMS. It is recognized that the probability of ion detection (assuming constant analyser transmission) is a function of ion mass and composition, and depends strongly on detector settings, including MCP voltage, secondary ion post-acceleration voltages and pulse counting discriminator voltages.[5] However, the effect of secondary ion intensity on detection probability is perhaps less established. For example, simultaneous impact by multiple secondary ions onto the MCP can lead to a higher pulse height than a single impact. If the discriminator voltage is set too high, then it is possible that the detection efficiency for single ion impact would be lower than for multiple ion impacts. For a particular SIMS secondary ion, the number arriving at the detector after each primary ion pulse follows a Poissonian distribution, and weaker peaks therefore consist of a larger proportion of single ion impact events than stronger peaks. For incorrectly adjusted systems, the detector efficiency can therefore be a function of ion intensity, leading to intensity nonlinearity.

An example is that at low MCP and high discriminator voltages, the detector efficiency is sub-optimal and the isotope ratios increase significantly from those for the correct settings, since the weaker $^{13}C^{12}C_{x-1}F_y^+$ peaks are detected with lower efficiency. This effect is very similar to signal nonlinearities observed in electron spectrometers when the discriminator is set too high.[16] Fortunately, although this can be a significant issue, it is not very common. The problem can be diagnosed effectively by comparing the measured isotope ratios to the expected ratios α given in Table 1. To ensure optimal detector efficiency and maintain intensity linearity for weak peaks, the MCP and discriminator voltages should be set correctly as demonstrated in the manufacturer's handbook or following the method given in Reference [5]. The discriminator should be operated at the lowest voltage that does not introduce significant detector dark counts. These counts can be measured by monitoring the background intensities in the mass spectrum with the primary ion beam switched off.

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