



Standard Guide for Interpretation of Mass Spectral Data Acquired with Time-of-Flight Secondary Ion Mass Spectroscopy¹

This standard is issued under the fixed designation E2695; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide provides time-of-flight secondary ion mass spectrometry (ToF-SIMS) users with a method for forms of interpretation of mass spectral data. This guide is applicable to most ToF-SIMS instruments and may or may not be applicable to other forms of secondary ion mass spectrometry (SIMS).

1.2 This guide does not purport to address methods of sample preparation. It is the responsibility of the user to adhere to strict sample preparation procedures in order to minimize contamination and optimize signals. See Guide E1078 and ISO 18116 for sample preparation guidelines.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

E673 Terminology Relating to Surface Analysis

E1078 Guide for Specimen Preparation and Mounting in Surface Analysis

E1504 Practice for Reporting Mass Spectral Data in Secondary Ion Mass Spectrometry (SIMS)

E1635 Practice for Reporting Imaging Data in Secondary Ion Mass Spectrometry (SIMS)

2.2 ISO Standards:³

ISO 23830 Repeatability and Constancy of the Relative Intensity Scale

¹ This practice is under the jurisdiction of ASTM Committee E42 on Surface Analysis and is the direct responsibility of Subcommittee E42.06 on SIMS.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, <http://www.iso.ch>.

ISO 18115:2007 Terminology Related to Surface Chemical Analysis

ISO 18116 Guidelines for Preparation and Mounting of Specimens for Analysis

3. Terminology

3.1 *Definitions*—See Terminology E673 and ISO 18115:2007 for definitions of terms used in SIMS.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *cationization*—the process by which a positively charged ion is formed, whereby a neutral molecule is combined with a cation, usually a metal ion; the resulting peaks are often referred to as cationized peaks.

3.2.2 *deprotonated molecular ion*—a molecule that has lost a proton to form a negative ion.

3.2.3 *exact mass*—measurement of the mass of an ion with sufficient accuracy to provide an unequivocal identification of its elemental and isotopic composition (**1**);⁴ for example, a peak located at m/z 43.0184 in a positive ion mass spectrum is most likely $C_2H_3O^+$ and cannot be $C_3H_7^+$ (m/z 43.0547).

3.2.4 *fragment ion*—the molecular ion, when struck by a primary ion beam, will typically break up into smaller pieces; this process is called fragmentation; a charged dissociation product arising from the fragmentation process is called a fragment ion.

3.2.5 *fragmentation pattern*—the fragmentation of molecular species results in a series of peaks that are characteristic of the molecule's underlying structure; this series of peaks is referred to as the fragmentation pattern of the molecule.

3.2.6 *m/z* —the X-axis of a mass spectrum is labeled as m/z , defined as the mass of the ion, in daltons, divided by the charge of the ion, where most ions are singly charged.

3.2.7 *mass defect*—when a peak's exact mass is located slightly below nominal mass.

3.2.8 *mass excess*—when a peak's exact mass is located slightly above nominal mass.

3.2.9 *mass resolution (ΔM)*—the measured full width at half maximum intensities of spectral peaks above their local backgrounds.

⁴ The boldface numbers in parentheses refer to a list of references at the end of this standard.

3.2.10 *molecular ion*—an ion formed by the removal of (positive ions) or addition of (negative ions) one or more electrons to a molecule, without fragmentation of the molecular structure.

3.2.11 *nominal mass*—particle mass, in daltons rounded to the nearest integer.

3.2.12 *protonated molecular ion*—a molecule that has gained a proton to form a positive ion.

3.2.13 *resolving power*—the ratio $M/\Delta M$ where ΔM is the full width at half-maximum peak height intensity for an ion peak of mass M ; in ToF-SIMS, the mass resolution depends on the time spread of the ions entering the detector.

4. Summary of Practice

4.1 This guide discusses the important issues that need to be considered during mass spectral data interpretation. It includes discussions of contamination, peaks commonly observed for certain organic functional groups, isotopic analysis, and differentiating between organic and inorganic peaks. This guide contains examples of several mass spectra including positive and negative mass spectra of commonly occurring contaminants.

5. Significance and Use

5.1 Interpretation of static SIMS mass spectral data can be complicated due to the complexity and density of data obtained and therefore, variability often occurs when users are not consistent in their methods of data interpretation. This guide is intended to help avoid these inconsistencies, by discussing the most commonly observed scenarios in static SIMS analysis and how to approach these scenarios.

5.2 This guide can be used as a training guide for employees or students, or both.

6. Procedure

6.1 *Tuning and Calibration*—For tuning and calibration, please refer to [ISO 23830](#).

6.2 *Important Factors in Mass Spectral Data Interpretation:*

6.2.1 *High Mass versus Low Mass Regions*—ToF-SIMS spectra are often divided into two separate regions: the low mass region and the high mass region. The low mass region extends from m/z 1 to approximately m/z 250. Within this range, peaks due to organic molecular fragments are mixed with peaks from atomic or inorganic clusters, or both. It is more unlikely for organic molecular ions to be found in this range, because without the use of a cold stage, most organic species with a molecular weight this low will volatilize in the vacuum system.

6.2.1.1 Above m/z 250 (the “high” mass range), organic peaks can represent either fragments or molecular ions. Also, in this range, there will be no more atomic ions and any inorganic ion will be a cluster. The region from m/z 100 to m/z 225 typically has more compound specific/unique organic fragment ions.

6.2.1.2 At very low mass ranges, (for example, below m/z 25), one will typically observe a series of background peaks characteristic of carbon (C), oxygen (O), and hydrogen (H) (except for fluorocarbon samples). The ammonium ion (NH_4^+)

and hydronium ion (H_3O^+) may also be present. An example of what is typically observed is depicted in [Fig. 1](#), which shows the positive and negative ion mass spectra (m/z 0 to m/z 27) taken from the surface of a silicon (Si) substrate.

6.2.2 *Nominal Mass*—In ToF-SIMS spectra which have a dynamic range of six orders of magnitude, most spectra have a peak at nearly every nominal mass, and with high mass resolution, multiple peaks may be found at the same nominal mass. Nonetheless, the analyst, at least initially, will focus on the more prominent peaks at any mass range.

6.2.2.1 *Mass Excess versus Mass Defect*—With a few exceptions, inorganic species in the low mass range will have a mass defect, which can clearly be observed in the mass spectrum. That is, the exact mass of these peaks will be less than the nominal mass. In contrast, organic species will have a mass excess, where the exact mass of the peaks will be greater than the nominal mass. For example, [Fig. 2](#) shows a mass spectrum taken from a poly(glycolic acid) (PGA), $(\text{CH}_2\text{COO})_n^-$, film on Si. In a spectrum covering a wide mass range, as depicted in [Fig. 2\(a\)](#), it would appear that there is only one peak at m/z 28. However, upon closer examination, it can be seen that there are actually three different peaks observed at the same nominal mass of m/z 28. The inorganic ion, Si^+ , is lower than m/z 28 (m/z 27.98), while the organic ion, C_2H_4^+ , is located above m/z 28 (m/z 28.04). CO^+ is also present in the mass spectrum and is located slightly below nominal mass.

(1) The exceptions to this rule include atomic H, lithium (Li), beryllium (Be), boron (B), C, and nitrogen (N) species, which have mass excesses. Above m/z 15, however, most inorganic ions will have a mass defect. In some specific situations, inorganic surfaces may give rise to high mass cluster series. At high enough mass, typically higher than m/z 500, the mass defect will become greater than 0.5 Daltons, and will enter a regime where the peaks can be comparable in fractional mass to highly saturated high mass organic fragments. In these situations, the peak identities should be apparent from the isotopic distributions, differences in mass between peaks in a series, and from calculations of possible empirical formulas.

6.2.2.2 *Odd versus Even Mass*—For a surface without nitrogen, the spectrum will be dominated by peaks with odd nominal masses. This is because SIMS organic fragments and molecular ions are dominated by even electron ions ($[\text{M} + \text{H}]^+$ or $[\text{M} - \text{H}]^-$), which for carbon-based species typically results in odd nominal masses. This is in contrast to electron impact ionization (EI) mass spectra, which more often have prominent odd electron ions, or free-radical ions ($\text{M}^{\cdot+}$ or $\text{M}^{\cdot-}$), which for carbon-based species typically results in even nominal masses (2). Therefore, in SIMS experiments, peaks with elevated intensities having even nominal mass are noteworthy, in that they either represent odd electron ions, or the presence of odd numbers of nitrogen atoms. Odd electron fragments typically have the odd electron stabilized within a pi system (resonance stabilized aromatic structures), which is an indication of unsaturation in the molecule. Aromatic species are more likely to produce odd electron ions, as are certain polymers, such as polyesters.

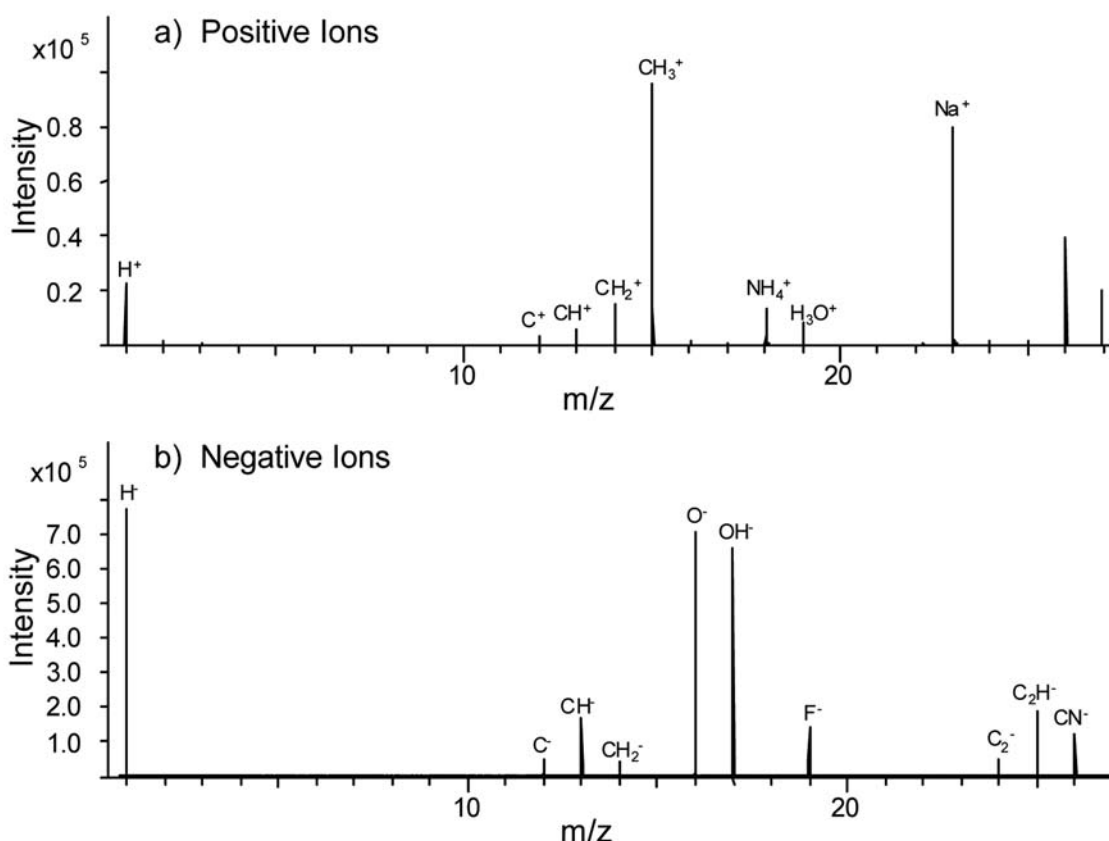


FIG. 1 Low Mass Range Mass Spectra Acquired From a Si Wafer with a Bi₃⁺ Ion Source: (a) Positive Ion Spectrum and (b) Negative Ion Spectrum

6.2.3 The Molecular Ion—All spectra should have a peak that is characteristic of the *molecular ion* or its protonated/deprotonated forms. An example of this is shown in Fig. 3, which shows a positive ion mass spectrum acquired from a cocaine deposit on Si (C₁₇H₂₁NO₄). The [M + H]⁺ ion is readily observed. In most spectra, the protonated [M + H]⁺ and deprotonated [M – H]⁺ molecular ions will have considerably higher intensities than the molecular ion [M]⁺. In the presence of strong ionizers, such as silver (Ag), sodium (Na), or gold (Au), one typically observes cationization. The cationized peaks will be of the form [M + Ag]⁺ or [M + Na]⁺. An example of this type of ionization is displayed in Fig. 4 and Fig. 5 (Na⁺ and Ag⁺ cationized peaks, respectively), which will be discussed later. It should be noted that the peak observed at the highest m/z is not necessarily the molecular ion in SIMS and that cluster ions are common (see 6.2.5).

6.2.4 Fragmentation Patterns—Other peaks in the spectra represent some fragment of the molecule. For example, in Fig. 3, the peak at m/z 182 represents the cocaine fragment after loss of (C₆H₅)COO⁻, while the peak at m/z 105 is associated with (C₆H₅)C=O⁺. Each molecule will have a unique fragmentation pattern associated with it, which can be useful for identification purposes.

6.2.5 Repeat Patterns in Polymers and Cluster Ions—Structures such as polymers typically yield mass spectra which have repeat patterns such as that displayed in Fig. 4 (3). In these repeat patterns, the peaks are separated by a mass that is

characteristic of the monomeric structure. Hence this can be very important in helping to identify structural components. In the particular example shown in Fig. 4, the peaks are separated by m/z 44, the monomer weight of the polyethylene oxide (PEO) chain, ⁻(CH₂CH₂O)⁻, (X or Y in the structure), and the distribution of intensities is representative of the polymer molecular weight distribution. This particular example shows a case where the polymer has a relatively low molecular weight compared to most polymers (<m/z 1000). More often than not, the polymer molecular weight will be much too high to be observed by SIMS (>m/z 10 000). We may however see a distribution of fragments.

6.2.5.1 The formation of inorganic or organic clusters also results in the formation of such repeat patterns, where the mass separating the peaks represents the mass of the element or compound that is forming the clusters.

6.2.6 Isotopic Patterns—Each element has a unique isotopic composition that should be evident in the mass spectrum, where all isotopes for a given element will be present with their expected, and in most cases natural, abundances. Therefore, definitive identification of unknown samples can be achieved with the help of isotopic analysis. An example of this is given in Fig. 6, which displays the measured isotopic pattern observed for ruthenium (Ru) in Fig. 6(a), with the theoretical distribution shown in Fig. 6(b). We can say definitively that this is Ru⁺ and not some organic fragment, such as CN₄O₂H₂⁺ because the isotopic distribution matches that of Ru⁺, not

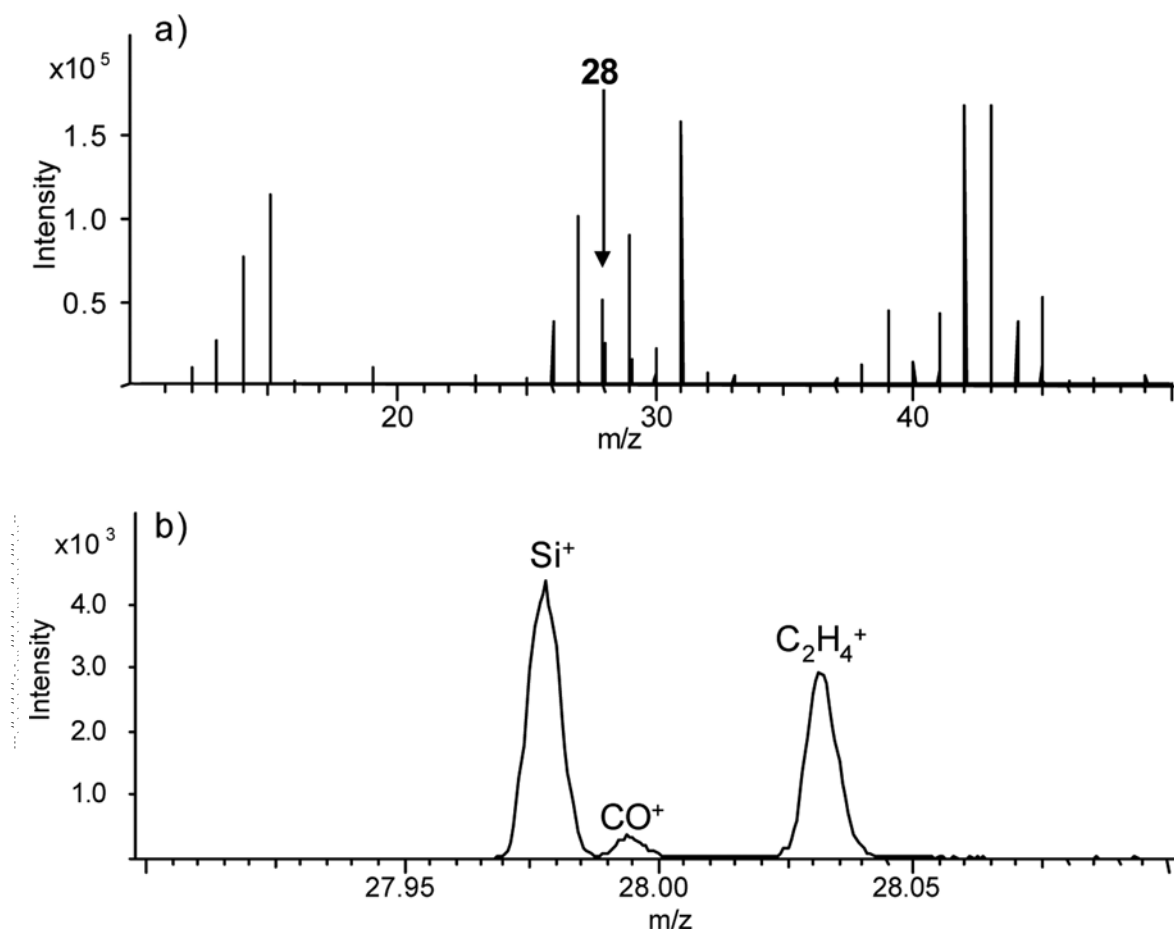


FIG. 2 Positive Secondary Ion Mass Spectrum of PGA on Si, Acquired with an Argon (Ar^+) Primary Ion Source: (a) Mass Range = (m/z 1 – m/z 50) and (b) Mass Range = (m/z 27.90 – m/z 28.10)

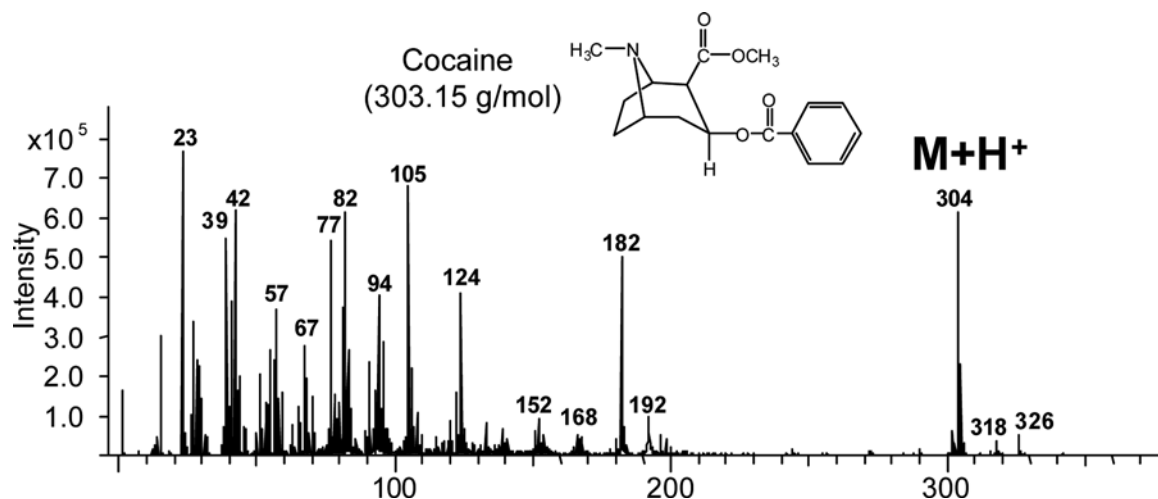
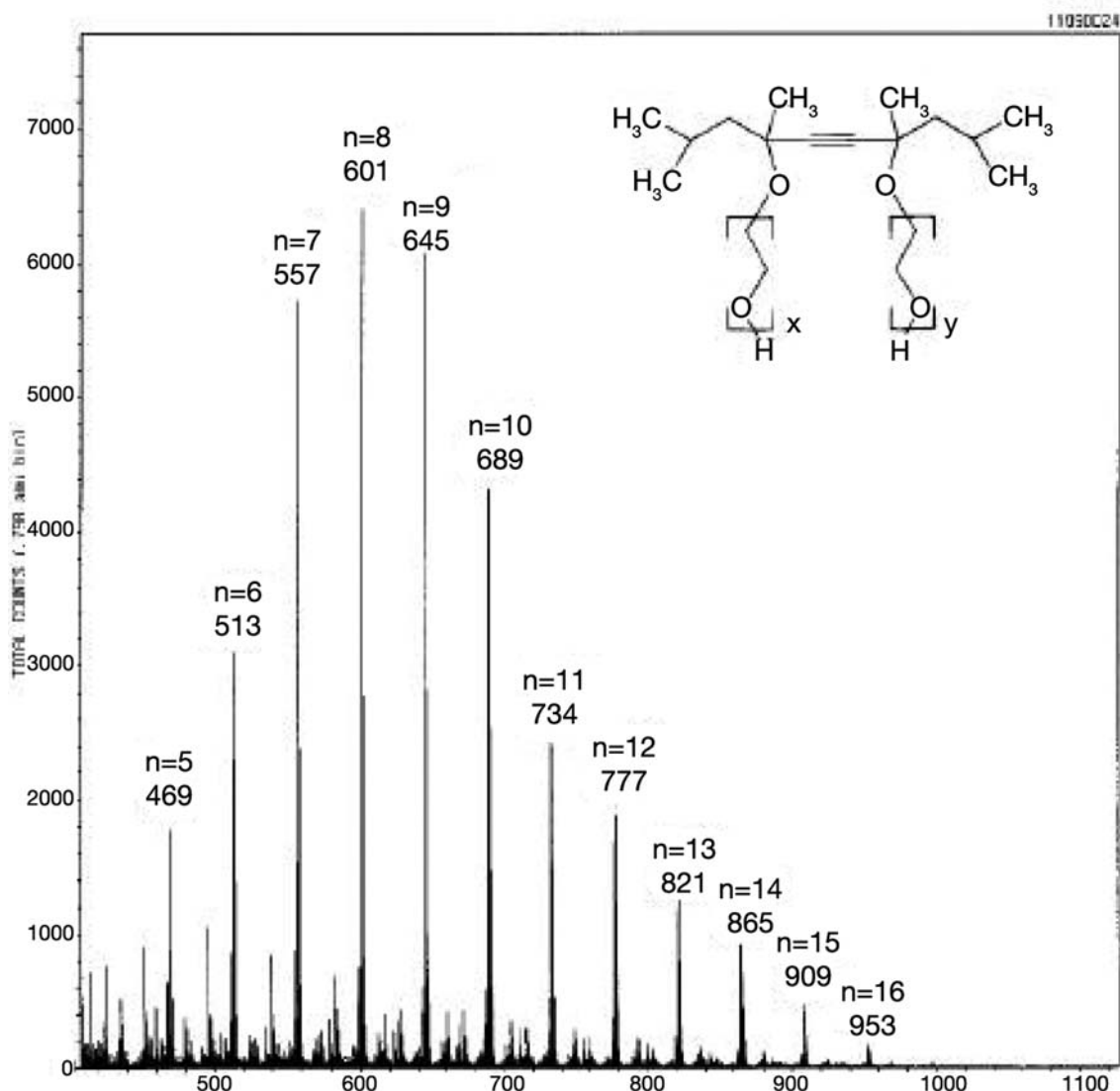


FIG. 3 Positive Secondary Ion Mass Spectrum of Cocaine Deposited on Si

$\text{CN}_4\text{O}_2\text{H}_2^+$, where 96.97 % of the intensity would be in the ^{102}Ru isotope, the m/z 102 peak, as opposed to 31.60 %. One should be aware that many atomic ions will have hydride peaks (especially for positive ions) that may be prominent 1 mass unit above the more abundant isotopes.

6.2.6.1 Groups of elements or molecular compounds also have unique isotopic patterns. These unique isotopic patterns

can help one to distinguish between compounds of near-identical masses, but with different empirical formulas. An example of this is given in Fig. 5(a), which shows the isotopic distribution observed at m/z 1047–1056, obtained from a sample of amine-terminated polydimethylsiloxane (PDMS) ($\text{NH}_2(\text{CH}_2)_3-(\text{Si}(\text{CH}_3)_2\text{O})_n-(\text{CH}_2)_3\text{NH}_2$) on Ag. One possible structure that is consistent with this distribution is given in Fig.



NOTE 1—Image taken from Cheng et al. (2000) (3), reprinted with permission.

FIG. 4 Repeat Pattern of a Na⁺ Cationized Surfnol Polymer, the Structure of Which is Indicated in the Top Right-Hand Corner

5(b). As can be seen, the experimentally observed and theoretical calculations match quite nicely for this particular structure. Fig. 5(c) shows another possible non-silver cationized fragment ion that could be observed. However the isotopic distribution is very different, and therefore we can conclude that the set of peaks in Fig. 5(a) is not consistent with the structure shown in Fig. 5(c). This example clearly demonstrates that isotope patterns can be particularly useful for identifying metal-containing ions.

6.2.6.2 One should use correlation plots to further verify the differences between the theoretical and predicted distributions, where the predicted intensities are plotted against the experimental intensities. If there is a correlation both the slope and the correlation coefficient should be near one. However, it is more than likely to be off somewhat due to the presence of hydrides, coupled with less than ideal mass resolution. For example, it is common for some elements to produce $[M + H]^+$ ions in addition to M^+ ions.

6.2.6.3 Isotope ratios can also be disturbed by the presence of inorganic cluster ions. For example, $^{65}\text{Cu}^+$ can be confused with $^{58}\text{Ni}_2\text{CH}_2^{2+}$. Even though this may be a small peak in the spectrum relative to $^{58}\text{Ni}^+$, it can complicate the detection of a trace of Cu. The transition elements in general are predisposed to making both singly and doubly charged clusters with O, CH_x , and N. Thus, the presence of transition elements in the spectrum can complicate the identification of elements with higher masses.

6.2.7 Negative versus Positive Ion Mode—Electronegative species will produce intense atomic ions in the negative ion mode and conversely with electropositive species. Thus, between positive and negative ion mode, the presence of most elements and molecular species can be determined. For example, the presence of halides and elements such as phosphorus (P), sulfur (S), and selenium (Se) should be determined from the negative ion spectra and will tend to form negative ions, while elements such as Na, potassium (K), Li, and

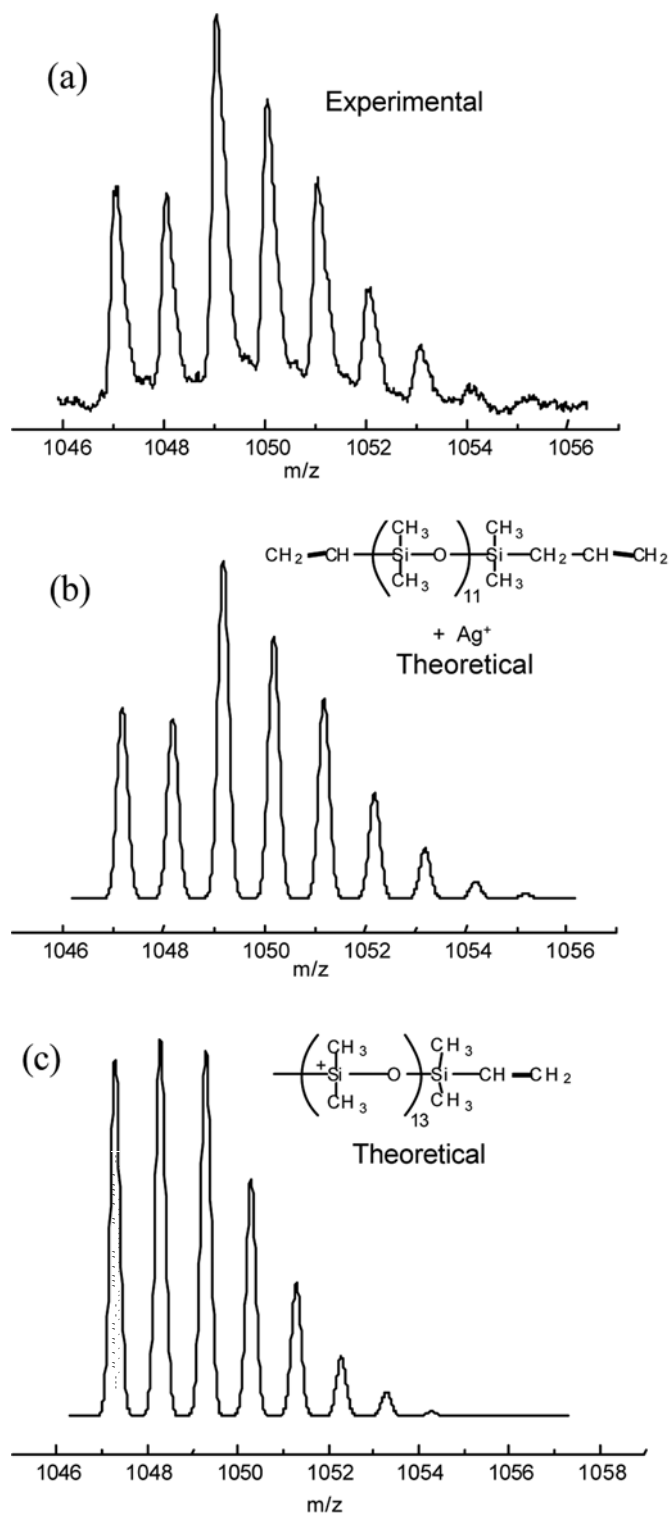


FIG. 5 Isotopic Distributions Acquired from a Sample of Amine-Terminated PDMS on Ag: (a) Measured Isotopic Distribution; (b) Theoretical Isotopic Distribution from a Possible Ag Cationized Molecule, and (c) Theoretical Isotopic Distribution from a Possible Protonated Fragment of PDMS

calcium (Ca) will tend to form positive ions.

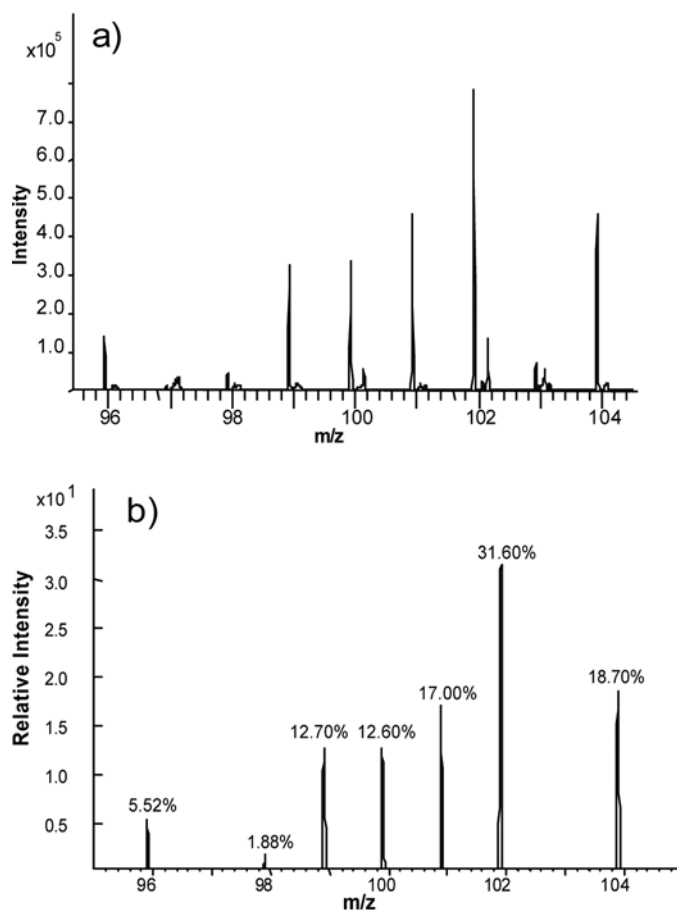


FIG. 6 Experimental (a) and Theoretical (b) Isotopic Distributions for Ru

6.2.8 SIMS Background—Below m/z 100, most ToF-SIMS spectra will contain a *hydrocarbon envelope* consisting of clusters of peaks separated by approximately m/z 14. The highest mass in each cluster will be the saturated ion with a given number of C atoms, the series CH_3^+ , C_2H_5^+ , C_3H_7^+ , C_4H_9^+ , etc. Each cluster will include one or more less saturated ions, with the main peaks having odd masses. For example, in the 2-carbon series, C_2H_5^+ will be accompanied by C_2H_3^+ , with a smaller peak for C_2H_4^+ . The hydrocarbon envelope can be absent or may not dominate this region of the spectrum for samples that are perfluorinated or have clean highly unsaturated materials (for example, polystyrene). For most surfaces, however, that have either an adventitious hydrocarbon contamination, or that contain molecules with significant hydrocarbon content, the hydrocarbon envelope will dominate this portion of the spectrum. The nature of the hydrocarbon envelope will be affected by instrumental parameters such as beam energy and primary ion type. For the same surface, a spectrum taken with a high energy gallium (Ga^+) ion beam, for example, will tend to produce a hydrocarbon envelope with more intense unsaturated ions, and will peak in a cluster with a lower number of C atoms when compared with a spectrum taken with a higher mass ion such as Au^+ or Bi^+ , or a cluster ion such as Au_2^+ or Bi_3^+ . Hydrocarbon envelope differences between samples with a given primary ion can be important to

an understanding of the surface. Surfaces with species containing O may also have added peaks that initially have the appearance of peaks associated with the envelope.

6.2.8.1 For most samples, introducing them into the system from air and without special treatment (etching or fracturing) will present a surface that is at least partially oxidized, and for such surfaces, atomic peaks are common for electropositive elements from Na and above.

6.2.9 Commonly Observed Functional Groups in SIMS:

6.2.9.1 *Nitrogen Containing Species*—As mentioned earlier, N containing species tend to result in the formation of even numbered peaks. N does not become an atomic ion in either positive or negative SIMS. In the positive spectra, N will present itself as NH_4^+ , which has a relatively large mass excess, while in the negative ion mass spectra, there will typically be a very large CN^- peak. An example of a mass spectrum acquired from a nitrogen containing species was illustrated in Fig. 3. As can be seen, the mass spectrum contains several even-numbered peaks.

6.2.9.2 *Phthalates*—Evidence of phthalates can be seen by a strong peak at m/z 149 in the positive mode, and intense peaks at m/z 121 in the negative ion mode.

6.2.9.3 *Sulfates*—Peaks at m/z 64, m/z 80, and m/z 97 present in the negative mode are indicative of sulfates. An example in Fig. 7, which shows the mass spectrum of sodium lauryl sulfate (C₁₂H₂₅NaO₄S), a common surfactant.

6.2.9.4 *Fluorocarbons*—Surfaces with fluorocarbons produce unique SIMS spectra and are readily characterized. These surfaces have low surface energies, so adventitious hydrocarbons tend not to adsorb, leaving the surface pristine and free of the typical hydrocarbon envelope described above. Fluorine (F) has a very small mass defect, so C_xF_y⁺ peaks are very close to nominal mass, falling between inorganic and organic species.

Perfluoropolyethers (PFPE), $-(\text{CF}_2\text{CFCF}_3\text{O})^-$, can be immediately distinguished from typical fluoropolymers such as polytetrafluoroethylene (PTFE), $-(\text{CF}_2\text{CF}_2)^-$, by the presence of the CFO^+ peak at m/z 47. Positive and negative ion mass spectra of PTFE are provided in Fig. 8 and Fig. 9.

6.2.9.5 *Phosphates (PO₄)*—Strong peaks at m/z 63 (PO_2^-) and m/z 79 (PO_3^-) in negative SIMS indicates the presence of a phosphate functional group.

6.2.9.6 *Polycyclic Aromatic Hydrocarbons*—Polycyclic aromatic hydrocarbons (PAH) are known to form in organic samples that are heavily damaged by ion beams (4). Their appearance indicates the formation of graphitic like species. They are also typically more prevalent in samples containing aromatic species (damaged or undamaged) and are indicative of the presence of benzene rings. PAH are observed in the positive ion mass spectra at the following masses: m/z 65, m/z 77, m/z 91, m/z 105, m/z 117, m/z 128, and m/z 165.

6.2.10 Commonly Observed Contaminants in SIMS:

6.2.10.1 *Poly(dimethylsiloxane)s (PDMS)* $-(\text{Si}(\text{CH}_3)_2\text{O})_n^-$ —One of the most commonly observed contaminants in surface analysis with SIMS is PDMS. PDMS is a low surface energy material that tends to spread over the surface of a material, so even a small amount can result in complete coverage of the sample, making surface analysis difficult. Positive ion and negative ion mass spectrum PDMS are displayed in Fig. 10 and Fig. 11.

6.2.10.2 *Fluorocarbons*—Another common contaminant in SIMS is fluorocarbons, found oftentimes in lubricants used during processing. An example of mass spectra from a fluorocarbon polymer is given in Fig. 8 and Fig. 9.

6.2.10.3 *Sodium and Other Salts*—Sodium is a very easily ionized material and therefore even if a small amount is present, it will be observed in the mass spectrum. In cases

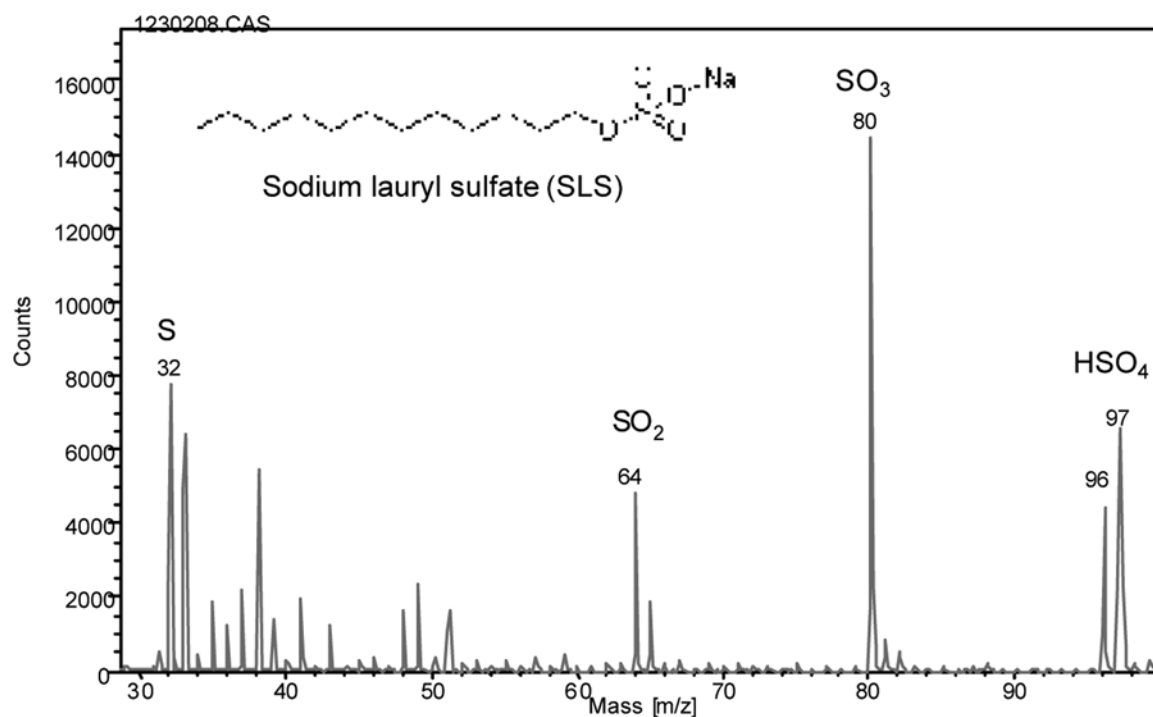


FIG. 7 Negative Ion Mass Spectrum of Sodium Lauryl Sulfate on Si

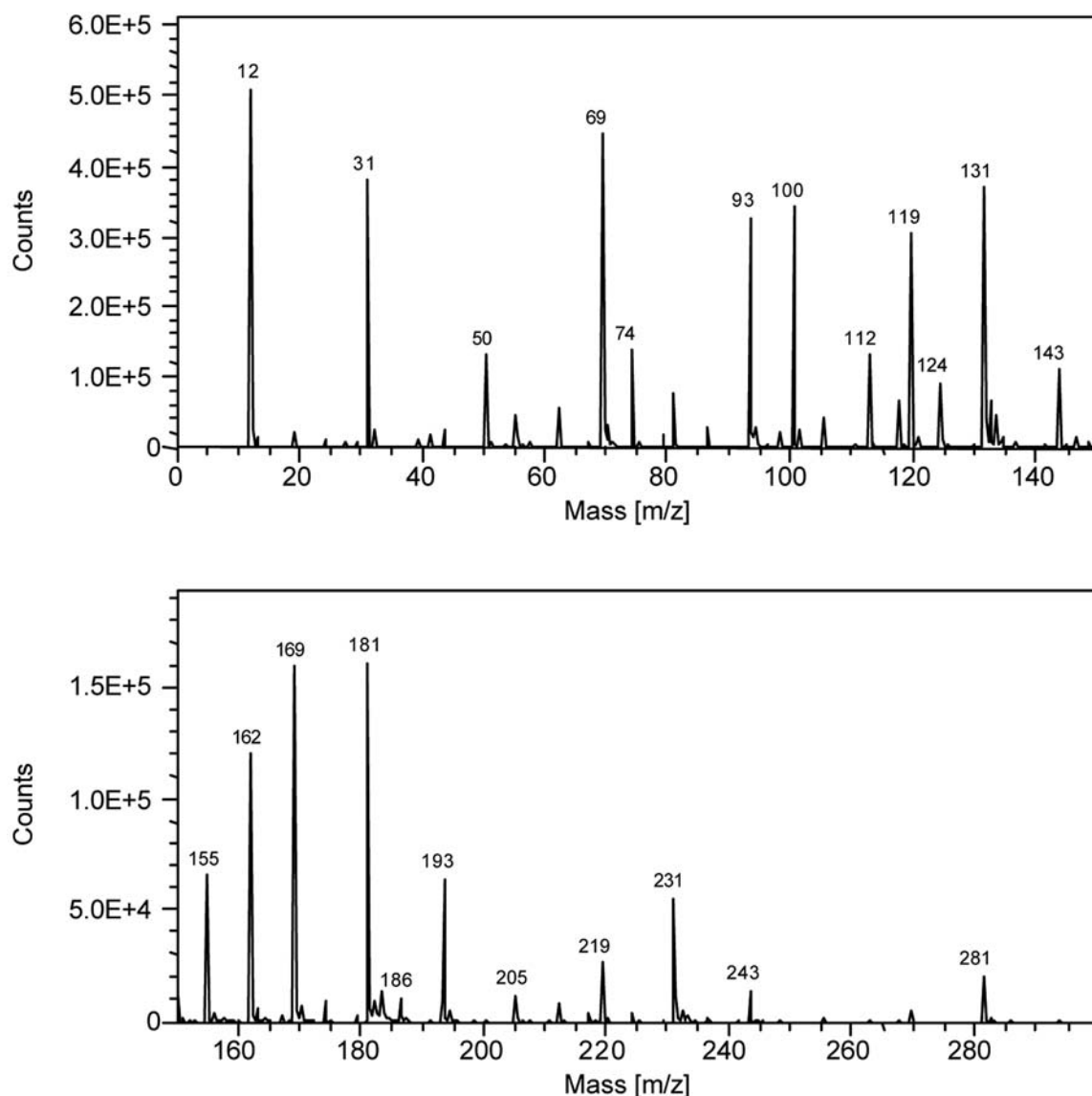


FIG. 8 Positive Secondary Ion Mass Spectrum of PTFE

where a lot of Na is present, it can dominate the spectrum and cause significant matrix effects.

6.2.11 Useful Tools for Data Analysis:

6.2.11.1 SIMS Database—For all mass spectral based methods, it is useful to have some form of mass spectral database from which to identify unknown constituents. These databases may be available through the software provided with the instrument. There is also a commercially available *Static SIMS library*. Unfortunately, there are still too many variables involved to obtain identical mass spectra from different instruments, even with similar samples, due to changes in ion beam parameters, such as source used, beam energy, and angle, etc. Currently no standardized database exists for static SIMS applications. However, one can obtain a general idea of the composition of a surface when comparing the experimental results to that obtained with a library, because while the relative intensities will vary significantly, in general, the same qualitative peaks will be present.

6.2.11.2 Use of a Molecular Weight Calculator—Molecular weight calculators are also a useful tool for identification of peaks in mass spectra. These tools may also be found with the software or there are several models available online for use. With this tool, one can calculate the exact mass and isotopic distribution associated with a particular molecule or element. One may also enter the mass of an unknown peak, and obtain several possible formulas for that mass. This is an essential tool for qualitative analysis.

6.2.11.3 Multivariate Statistical Analysis Methods—Principal Components Analysis (PCA) or Multivariate Curve Resolution (MCR) can be useful techniques to help rapidly characterize and compare complex mass spectral data. For more information about these techniques utilized for SIMS applications, see Eynde and Bertrand (1997) (5).

6.2.12 Commonly Observed Artifacts in SIMS:

6.2.12.1 Topography Effects—In imaging SIMS, the yield of a specific secondary ion species can vary across a sample

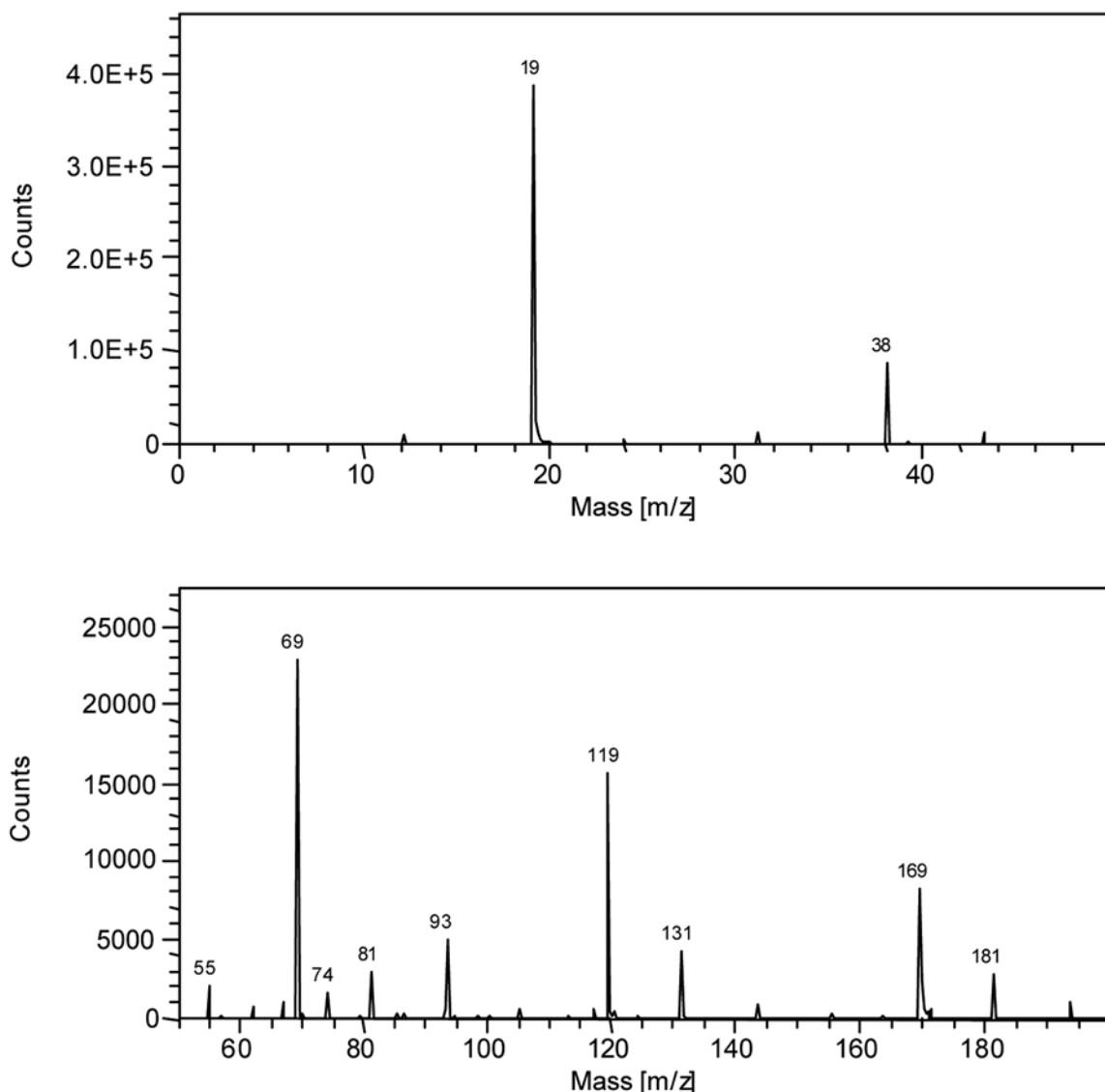


FIG. 9 Negative Secondary Ion Mass Spectrum of PTFE

surface because of true surface concentration differences and topographic effects. In terms of mass spectral data analysis, large topography features, will most likely result in peak broadening or possible double peak formation, or both.

6.2.12.2 *Charging*—In insulating samples, charging starts to play an important role. This may cause peak broadening, peak shifts, and in extreme cases, loss of signal.

6.2.12.3 *Incorrect Bunching*—If broad peaks or double peaks are observed, and the sample is relatively flat and

conducting, this is typically an indication that the beam is not being bunched correctly.

6.3 *Reporting of Mass Spectral Data*—See Practices E1504 and E1635 for proper reporting of mass spectral data.

7. Keywords

7.1 mass spectra; SIMS; interpretation; contaminants; ToF-SIMS

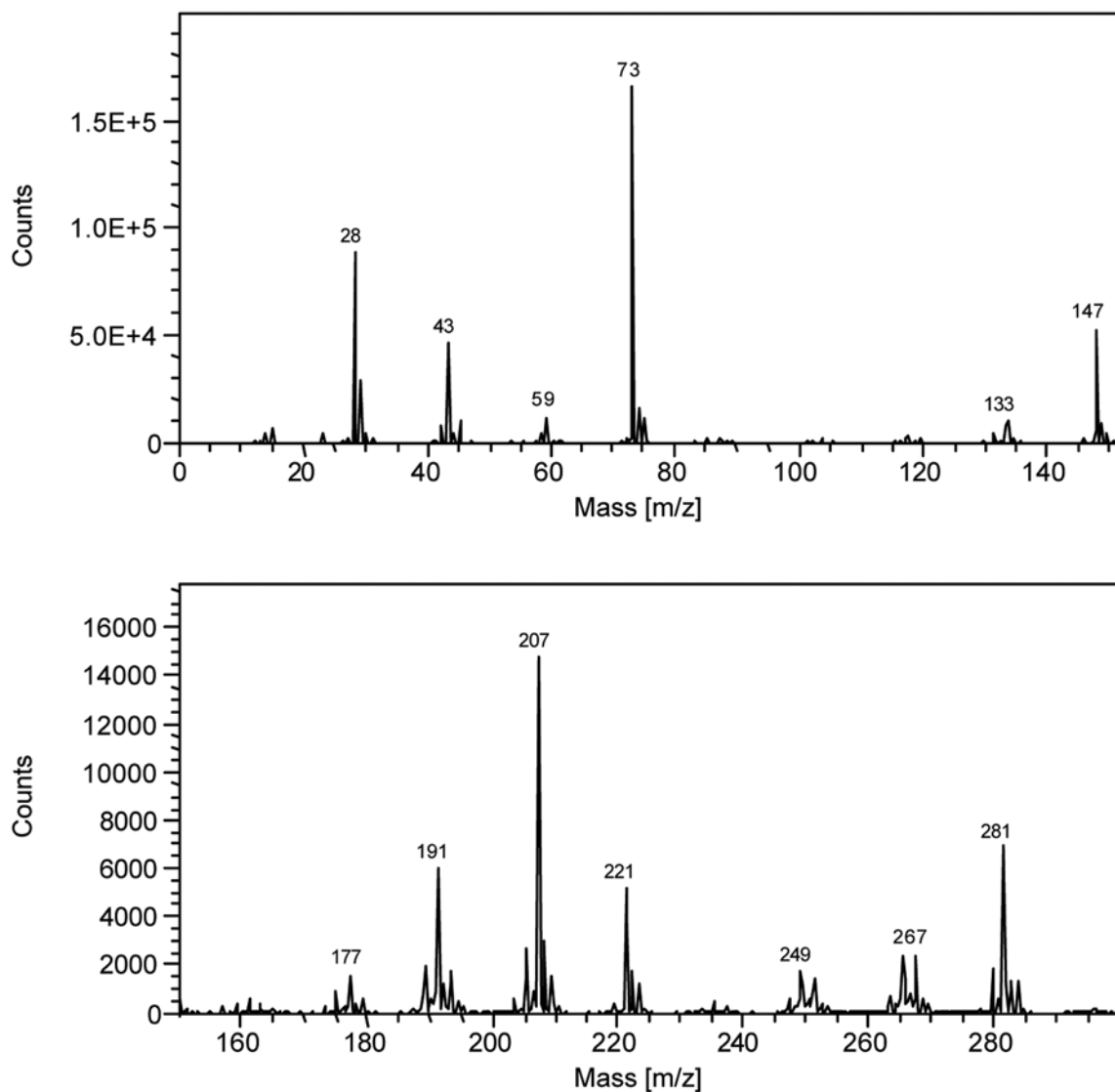


FIG. 10 Positive Ion Mass Spectrum of PDMS

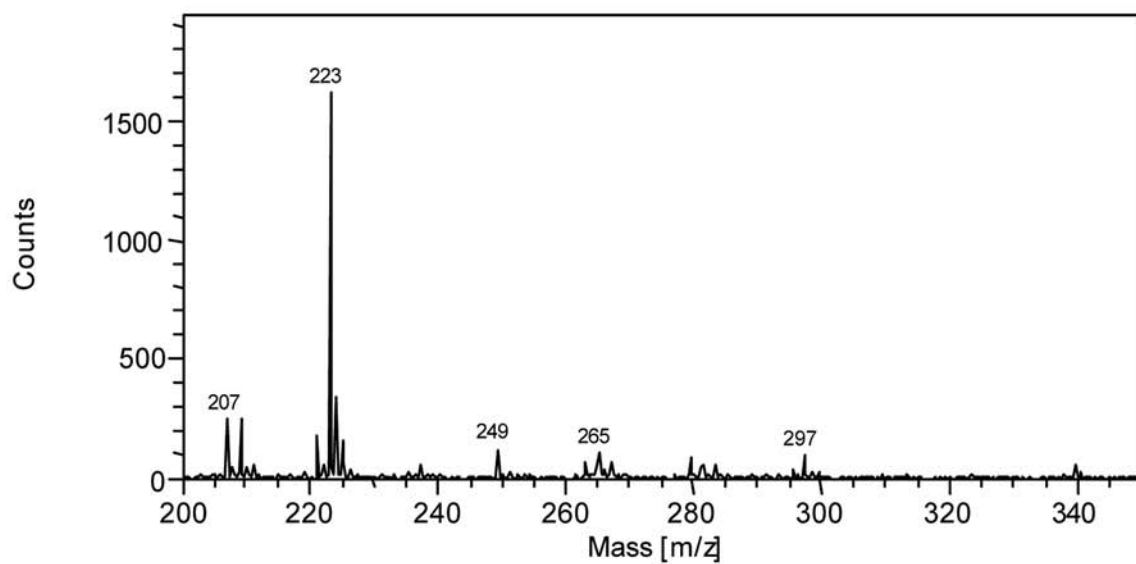
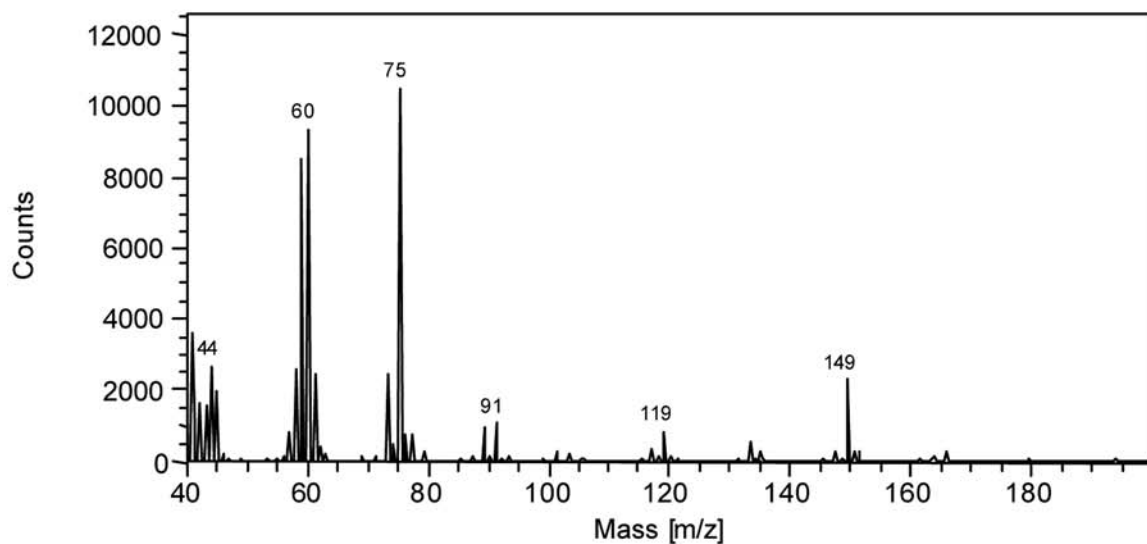
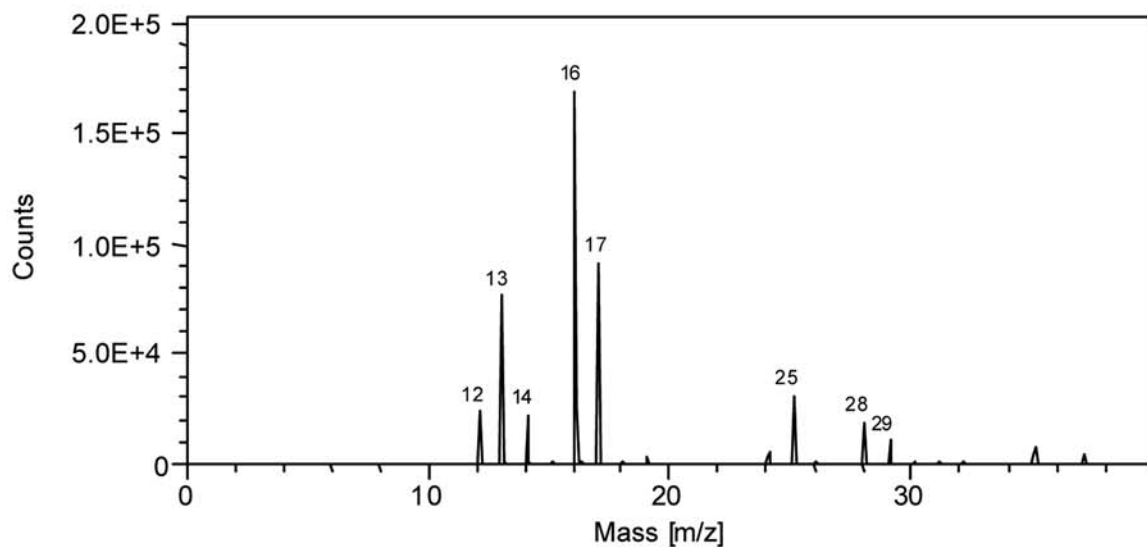


FIG. 11 Negative Ion Mass Spectrum of PDMS

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