

Useful ion yields for Cameca IMS 3f and 6f SIMS: Limits on quantitative analysis

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Abstract

The useful yields (ions detected/atom sputtered) of major and trace elements in NIST 610 glass were measured by secondary ion mass spectrometry (SIMS) using Cameca IMS 3f and 6f instruments. Useful yields of positive ions at maximum transmission range from 10^{-4} to 0.2 and are negatively correlated with ionization potential. We quantified the decrease in useful yields when applying energy filtering or high mass resolution techniques to remove molecular interferences. The useful yields of selected negative ions (O, S, Au) in magnetite and pyrite were also determined. These data allow the analyst to determine if a particular analysis (trace element contents or isotopic ratio) can be achieved, given the amount of sample available and the conditions of the analysis. © 2005 Elsevier B.V. All rights reserved.

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1. Introduction

The ability to chemically characterize geological phases on the scale of a few microns has had a fundamental impact on the quantitative nature of modern geology. The analytical techniques used, such as

the electron probe, synchrotron X-ray analysis, secondary ion mass spectrometry, and laser ablation mass spectrometry have been described in several review articles (see for example Potts et al., 1995). The last two represent well-known techniques with expanding popularity, yet their limits of quantitative analysis have not been well described. This contribution presents new data on the useful ion yields of several elements determined on two secondary ion mass spectrometers. The useful yield is defined as the number of ions detected per atom of that species sputtered from the sample, and depends on the ionization probability as well as the transmission and detection efficiencies of the analytical instrument. A number of instrumental parameters, including the acceleration potential of the secondary ion mass spectrometer, the energy bandpass

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of the mass spectrometer, the energy and polarity of the secondary ions analyzed, and the mass resolving power of the instrument contribute to the useful yield. In this study, we investigated the dependence of the useful yield on each of these parameters. The results allow us to determine the most favorable analytical conditions for obtaining the lowest detection levels. Useful yields can be used to determine if signal sufficient for a meaningful analysis will be acquired given the concentration of the element to be analyzed and the volume of the phase available.

2. Samples studied

For this study, we selected one of the standard reference glasses synthesized at the National Institute of Standards and Technology, NIST 610. This is a soda-lime silicate glass doped with 61 trace elements nominally at 500 ppm (by weight) abundances. This glass and the related glasses NIST 612 and NIST 614 (nominally doped with 50 and 1 ppm, respectively, of these same trace elements) are commonly used as primary or secondary standards in microbeam trace element analysis (NIST 611, 613, and 615 have the same composition as NIST 610, 612, and 614, respectively. The odd numbered samples are sold as 14 mm diameter wafers 1 mm thick while the even numbered samples are 3 mm thick). The inductively-coupled plasma mass spectrometric (ICP-MS) analyses by Pearce et al. (1997) are given in Table 1. Three additional samples, Balmat pyrite (Crowe and Vaughan, 1996), a gold-implanted pyrite (Cabri and McMahon, 1995), and magnetite (Hervig et al., 1992) were also studied. All samples were embedded in epoxy and polished with a combination of SiC powder and diamond paste and cleaned by sonification. A carbon coat was applied to the pyrite sample (after implantation) and gold coats were applied to the magnetite and NIST 610 samples.

3. SIMS instrumentation

The design of the Cameca f-series secondary ion mass spectrometers (SIMS) has been described previously (e.g., Hervig, 1996; Ireland, 1995). Briefly, an energetic ion beam (O^- , O_2^- , O_2^+ or Cs^+ at 3 to 20 keV impact energy) is directed at a sample. As these primary ions strike the target, surface and near surface atoms are set in motion in a collision cascade. Some atoms are ejected from the surface as a result of this transfer of momentum, and a fraction of the ejected atoms (on the order of 1–10%) are ionized in this

Table 1
Composition of NIST 610 glass

	wt.%	Standard deviation		ppmw	Standard deviation
SiO ₂	69.975	0.391	Sr	505.3	9.2
TiO ₂	0.075	0.005	Y	452.6	19.1
Al ₂ O ₃	2.039	0.157	Zr	439.9	7.8
FeO	0.056	0.016	Nb	381.8	98.1
MnO	0.054	0.005	Mo	396.1	38.9
MgO	0.065	0.007	Ag	240.5	19.7
CaO	11.450	0.231	Cd	260.9	4.5
Na ₂ O	13.352	0.681	In	439.8	35.5
K ₂ O	0.059	0.002	Sn	390.0	19.8
Cl	0.047	0.000	Sb	388.3	5.1
P ₂ O ₅	0.120		Cs	351.1	26.8
			Ba	415.9	21.5
Sum	97.292		La	436.6	39.6
	ppmw	Standard deviation	Ce	443.3	15.0
Li	482.2	21.3	Pr	431.2	33.3
Be	460.7	15.5	Nd	430.0	41.0
B	357.6	4.5	Sm	450.6	22.6
Mg	465.3	26.6	Eu	429.5	38.3
P	342.5	53.1	Gd	422.9	31.4
Cl	470.0	0.0	Tb	446.0	32.3
K	463.1	8.1	Dy	434.8	21.5
Sc	439.3	10.9	Ho	453.0	36.8
Ti	432.8	12.1	Er	434.7	27.4
V	440.2	36.4	Tm	422.5	23.6
Cr	388.0	15.2	Yb	462.7	26.0
Mn	436.4	21.4	Lu	433.1	35.8
Fe	453.1	17.9	Hf	411.2	24.2
Co	400.9	22.6	Ta	361.0	71.6
Ni	439.1	15.8	Re	121.5	119.5
Cu	432.9	16.9	W	434.5	23.3
Zn	429.3	16.4	Au	22.9	3.7
Ga	438.0	12.2	Pt	61.3	1.9
Ge	431.0	7.1	Pb	419.1	9.4
As	317.6	13.9	Bi	373.8	28.7
Se	112.0	2.8	Th	463.1	11.9
Rb	430.2	7.0	U	462.9	6.2

Measurements from Pearce et al. (1997).

process. A high voltage ($\sim \pm 4500$ to ± 10000 V) is placed on the sample to accelerate these secondary ions into a mass spectrometer, where they are discriminated as a function of their energy and mass by electrostatic and magnetic analyzers, respectively. After mass analysis, the secondary ion beam is steered by a second electrostatic analyzer into a detector. Two detectors are available: an electron multiplier is used in an ion-counting mode for intensities from <1 to $\sim 10^6$ counts/s; whereas higher intensity signals are measured with a Faraday cup and electrometer. This study used both the Cameca 3f and 6f SIMS at Arizona State University. The major difference between the two instruments is that the mass spectrometer on the 3f operates at a secondary acceleration

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