



# Calibration of relative sensitivity factors for impact ionization detectors with high-velocity silicate microparticles



Katherina Fiege<sup>a,b,c,\*</sup>, Mario Trieloff<sup>b</sup>, Jon K. Hillier<sup>b</sup>, Massimo Guglielmino<sup>b</sup>, Frank Postberg<sup>b,c</sup>, Ralf Srama<sup>c,e</sup>, Sascha Kempf<sup>d</sup>, Jürgen Blum<sup>a</sup>

<sup>a</sup> Institut für Geophysik und extraterrestrische Physik, Universität Braunschweig, Mendelssohnstr. 3, 38106 Braunschweig, Germany

<sup>b</sup> Institut für Geowissenschaften, Universität Heidelberg, Im Neuenheimer Feld 236, 69120 Heidelberg, Germany

<sup>c</sup> Institut für Raumfahrtssysteme, Universität Stuttgart, Pfaffenwaldring 31, 70569 Stuttgart, Germany

<sup>d</sup> LASP, University of Colorado at Boulder, Boulder, CO 80303, USA

<sup>e</sup> Baylor University, Waco, TX 76706, USA

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## ABSTRACT

Impact ionization mass spectrometers, e.g., the Cosmic Dust Analyzer (CDA) onboard the Cassini spacecraft can quantitatively analyze the chemical composition of impacting particles, if the ionization efficiencies of the elements to be quantified are appropriately calibrated. Although silicates are an abundant dust species inside and outside the Solar System, an experimental calibration was not available for elements typically found in silicates. We performed such a calibration by accelerating orthopyroxene dust of known composition with a modified Van de Graaff accelerator to velocities of up to  $37.9 \text{ km s}^{-1}$  and subsequent analyses by a high resolution impact ionization mass spectrometer, the Large Area Mass Analyzer (LAMA). The orthopyroxene dust, prepared from a natural rock sample, contains  $\sim 90\%$  orthopyroxene and  $\sim 10\%$  additional mineral species, such as clinopyroxene, spinel, amphibole, olivine and glasses, which are present as impurities within the orthopyroxene, due to inclusion or intergrowth. Hence, the dust material can be regarded as a multi-mineral mixture. After analyses, we find that most particle data cluster at a composition ascribed to pure orthopyroxene. Some data scatter is caused by stochastic effects, other data scatter is caused by the chemically different mineral impurities. Our data indicate that these minor mineral phases can be recognized within a multi-mineral mixture. Here, for the first time, we present experimentally derived relative sensitivity factors (RSFs) for impact ionization mass spectroscopy of silicates, enabling the quantitative determination of the composition of cosmic dust grains. Orthopyroxene data were used to infer RSFs for Na, Mg, Al, Si, Ca, Ti, Fe and K, for particles with radii ranging from  $0.04 \mu\text{m}$  to  $0.2 \mu\text{m}$  and velocities between  $19$  and  $37.9 \text{ km s}^{-1}$ , impacting on a Rh-target.

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## 1. Introduction

Interplanetary space in our Solar System is populated by dusty debris resulting from collisions or internal activities of larger bodies like asteroids, comets, or planetary moons. Streams of dust from outside of our Solar System, i.e., interstellar dust (ISD) have also been detected. The use of space-based in situ dust detectors for compositional analysis of cometary (e.g., Kissel, 1986; Kissel et al., 1986, 2004, 2007), interplanetary and interstellar dust (e.g., Kempf et al., 2004; Grün et al., 2004; Hillier et al., 2007a;

Altobelli et al., 2003, 2006) and circumplanetary dust (e.g., Kempf et al., 2005; Hillier et al., 2007b; Hsu et al., 2009; Postberg et al., 2008, 2009b) enables us to gain insights into the composition and dynamical properties of individual dust grains and thus their origins and formation environments.

Most space-based dust detectors utilize impact ionization time-of-flight mass spectrometry (II-TOF-MS) for the determination of the chemical composition of cosmic dust grains. A particle impacting on a target will be partially or wholly vaporized and/or ionized, resulting in the development of a plasma cloud. The plasma cloud contains neutral and charged species from the projectile and from the target which may interact with each other. However, the energetics and composition of this plasma cloud depend strongly on a variety of conditions, such as the composition, mass and velocity of the impactor, its shape and orientation, as well as the properties

\* Corresponding author at: Institut für Geowissenschaften, Universität Heidelberg, Im Neuenheimer Feld 236, 69120 Heidelberg, Germany.

E-mail address: [katherina.fiege@geow.uni-heidelberg.de](mailto:katherina.fiege@geow.uni-heidelberg.de) (K. Fiege).

of the target, e.g., composition, surface roughness, and contamination state. Hornung and Drapatz (1981) and Hornung and Kissel (1994) have modeled the impact ionization process, but still many open questions remain.

Simple II-TOF-MS instruments consist of a source region, a drift region, and an ion detector. The ions are accelerated through a potential difference and are then drifting through a field-free region. The velocities of the particles are inversely proportional to the square root of their masses, i.e., lighter ions will have higher velocities than heavier ions, assuming they have the same charge. Thus, ions with lower masses will reach the ion detector earlier than heavier ions. The resulting spectrum from ions measured at the ion detector is the detected output charge as a function of time. Assuming all ions with different masses have the same initial energy distribution, a mass scale can be generated using, e.g.,  $t_i = b + a \cdot \sqrt{q_i/m_i}$ , with  $a$  being the *stretch factor*, a proportionality constant, determined by the set up of the instrument, and  $b$  being the *shift parameter*, representing any time offsets between the triggering point, and the start of the spectrum.  $t_i$ ,  $m_i$  and  $q_i$  represent the arrival time, mass, and charge of a specific ion species. The parameters  $a$  and  $b$  have to be determined for the conversion of the detector output, representing the relative intensity of the signal as a function of time, into a mass spectrum. Linear II-TOF-MS instruments, such as the Cosmic Dust Analyzer (CDA) (Srama et al., 2004) onboard the Cassini spacecraft, have a rather low resolution ( $\frac{m}{\Delta m} \sim 20\text{--}50$ ), limited by the length of the free drift region and due to the initial energy distribution. To obtain higher resolutions, we used an instrument utilizing a reflectron for this study, which provides longer ion flight times and mitigates the effects of the initial energy spread of the ions (e.g., Mamyrin et al., 1973; Mamyrin, 2001; Doroshenko and Cotter, 1999).

Our study aims at calibrating the detection efficiencies of II-TOF-MS detectors in order to allow quantification of the concentration of certain elements in silicate particles. II-TOF-MS measures directly the ionized species originating from a hypervelocity impact, either as atomic ions, as newly formed cluster ions, or as molecular ions that are extracted from the plasma via an electric field. The intensities of certain mass lines are mainly determined by the ion/neutral ratio, i.e., the ease of ionization of a specific element. For example, alkali elements with a low ionization potential should develop stronger lines than, e.g., Si. To translate line intensities into element concentrations, we need to establish relative sensitivity coefficients (RSFs). This is common use in similar geochemical methods, e.g., secondary ion mass spectrometry (SIMS) (e.g., Stephan, 2001). Here, ions are formed in a rock target by bombardment with a primary ion beam, and secondary ions are detected by sector field or time of flight secondary ion mass spectrometry (TOF-SIMS). In SIMS, peak heights are translated into actual compositions by the use of relative sensitivity factors (RSFs), which are a measure for detection efficiency. These detection efficiencies are usually normalized, for example, to a common rock-forming element like Si.

While qualitative calibration of II-TOF mass spectra has been performed for years using conductive material like metals, carbon and certain polymers (e.g., Stübiger et al., 2001; Goldsworthy et al., 2002; Goldsworthy et al., 2003), the detailed study of silicates (e.g., quartz, anorthite, see Hillier et al., 2009, 2012), became only recently possible due to a relatively new coating technique (Hillier et al., 2009). E.g., for the calibration of the PUMA/PIA instruments, regarding data from Comet Halley, only Fe, Ni, carbon and coated glass particles were available for electrostatic acceleration onto metal targets (e.g. Krueger, 1996).

This paper reports impact experiments with orthopyroxene dust onto a high resolution impact ionization mass spectrometer, the Large Area Mass Analyzer (LAMA) (Srama et al., 2005) at the Heidelberg dust accelerator facility.

## 2. Experimental setup

### 2.1. Impact ionization dust detector

Mass spectra were generated by the LAMA. The LAMA was developed for dedicated dust astronomy space missions. It is a reflectron-type II-TOF mass spectrometer, and its large target area of 0.1 m<sup>2</sup> and wide field-of-view of >50° are especially suitable for the detection of low dust fluxes, e.g. interstellar dust, in interplanetary space. It achieves a typical mass resolution of up to  $\frac{m}{\Delta m} \sim 300$ . The LAMA has a cylindrical symmetry and six annular disc electrodes, as well as six ring electrodes generate the electrostatic field of the reflectron. The impact target is held at +5 kV potential with a grounded acceleration grid 50 mm in front of it. Further, the LAMA has a field-free-drift region in front of the impact detector and two parabolic shaped grids at potentials of 0 V and +5800 V. With the LAMA, it is possible to operate in either positive or negative ion mode. In this work, we exclusively recorded cation spectra, using rhodium foil as the target material. The Rh foil is especially a good analog for the target material used in the CDA onboard the Cassini spacecraft.

The signals of the LAMA ion detector and the beam line sensor were recorded by a 4 channel 1 GHz digital oscilloscope. Finally, the data were processed with an in-house software package, developed for analyzing impact mass spectra.

### 2.2. Van de Graaff accelerator

For our experiments we used the modified 2 MV Van de Graaff accelerator (Mocker et al., 2011), operated by the University of Stuttgart and located at the Max-Planck-Institut für Kernphysik. Here, positively charged dust grains, with sizes from 50 nm up to 5 μm, are accelerated via an electrostatic field of ~2 MV to hypervelocity speeds between 1 and 100 km s<sup>-1</sup>. The kinetic energies of the particles arise from the conservation of energy  $1/2 \cdot m \cdot v^2 = q \cdot U_{pot}$ , with  $U_{pot} = 2$  MV,  $v$  being the particle's velocity,  $q$  its charge, and  $m$  its mass. The speeds and masses of the accelerated particles are derived from the durations and amplitudes of the charge signals induced by the grains during their flight through a shielded cylindrical sensor in the accelerator beam line (Srama and Auer, 2008).

### 2.3. Cosmic dust analog material

Minerals of the pyroxene group are amongst the most abundant ferromagnesian silicates in the Solar System. They belong to the group of chain silicates and occur either as orthorhombic or monoclinic crystals. Orthopyroxenes essentially consist of the simple chemical series of (Mg,Fe)<sub>2</sub>SiO<sub>6</sub>, whereas monoclinic species show a broader range of composition with also sodium, calcium, and aluminum accommodated in their structures (Deer et al., 1992). For our experiments we used an orthopyroxene separate (Table 1), obtained from a spinel-lherzolite, designated SA84-132, from Har-rat Uwayrid in Saudi Arabia (Kaliwoda, 2004). Spinel-lherzolites are representative of the terrestrial lithospheric mantle.

The orthopyroxene was prepared by standard mineral separation techniques, involving crushing, sieving and washing, hand-picking of coarse grained orthopyroxene grains, magnetic separation, and further cycles of the aforementioned procedures to aid in the purification of the orthopyroxene separate. The purity of such mineral separates is usually of the order of 90%. It is particularly time-consuming and difficult to remove remaining impurities, e.g. because of the fine-grained nature of mineral intergrowths or inclusions. During the final preparation step, grains

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