



Physical & Theoretical Chemistry University of Wuppertal

A Plasma/Electron Ionization Calibration Source for **Absolute Ion Counting and Ion Density Measurements**

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Introduction

This work presents the concept of a "calibration" source which is used for comprehensive determination of ion transport efficiencies throughout the mass spectrometer as well as ion density measurements. The MS system includes a 170 cm hexapole transfer stage, enabling long-distance ion sampling. A key application for such a system is in-situ ultra-trace element plasma analysis in hydrogen matrices as occurring in semiconductor manufacturing environments.

Two different ionization methods for calibration are established: I) ionization via DC plasma and II) ionization with a commercial EI source from Pfeiffer Vacuum GmbH (Crossbeam IQ, Asslar, Germany). In both approaches, the total ion count (TIC) is determined on an electrode of defined geometry at the ion entrance of the MS system.

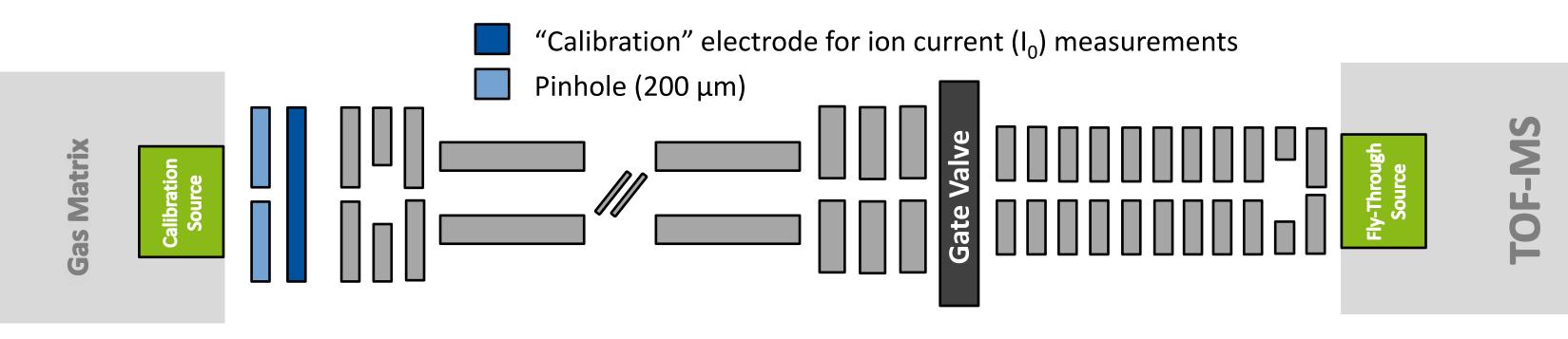
The calibration unit consists of a pinhole (200 μ m), which has the same geometry as the first lens of the transfer stage and the electrode where the ion current (I_0) is detected. The small pinhole prevents the voltages applied downstream from influencing the region upstream of the hexapole, while also acting as a pressure restriction. After removing the calibration unit, the total ion count is measured at the MS multichannel plate detector, providing mass resolved ions-per-area data for calibration purposes.

Besides ion transfer determination, this calibration method is used to obtain further data, including the ion density distribution in front of the transfer stage. This offers insights in plasma dynamics and enables direct plasma characterization regarding mass resolved ion current densities, which are significant input parameters for plasma simulations.

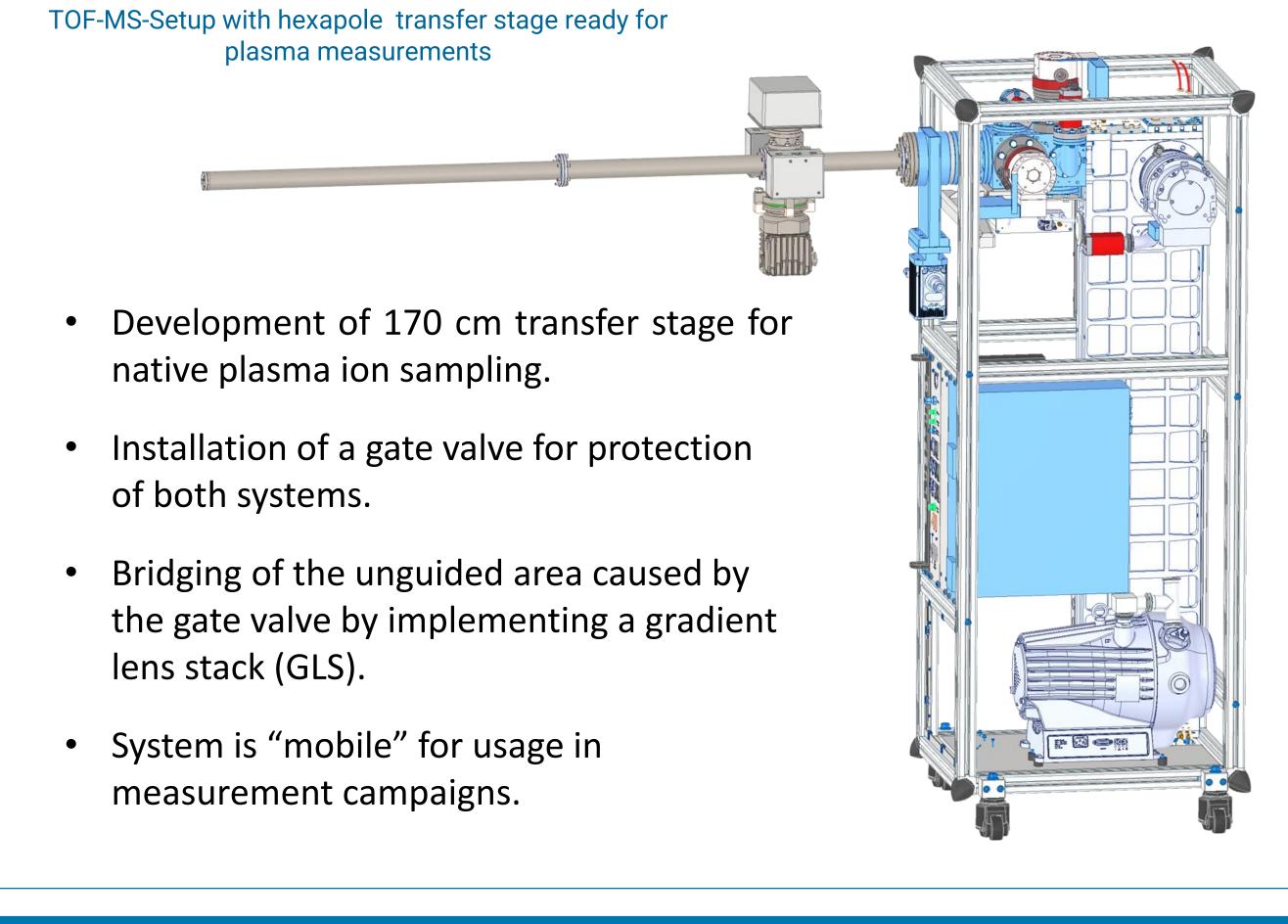
Concept

Setup for Plasma Measurements

- Double calibration electrode stack in front of the first transfer stage lens at the MS-system entrance.
 - System needs to be vented for placing or removing the calibration unit.
- Ion current (I_0) per time and area is measured at the "calibration" electrode with defined geometry.
 - Due to the same geometry of the first transfer stage lens and the first calibration optic lens, the number of ions entering the MS-system through the transfer stage can be deduced.
- When the calibration unit is removed, the total ion count is measured at the TOF-MS detector.
 - Mass resolved ion current per area and time provides insights in transport efficiencies through transfer and vacuum stages as well as plasma ion densities.



Schematical illustration of the calibration source concept including the transfer stage



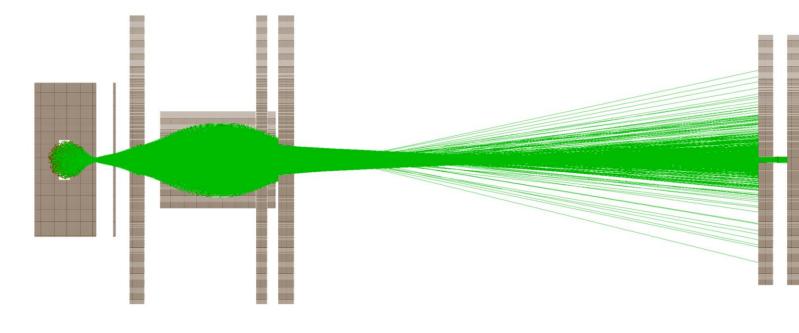
El Approach

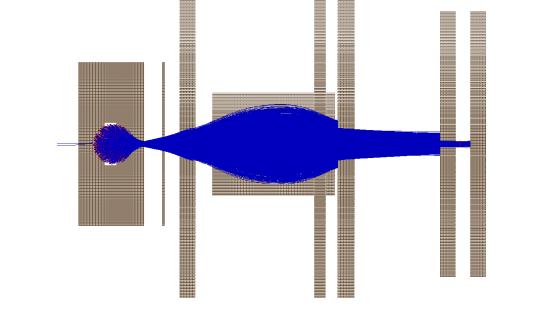
Commercial El source from Pfeiffer Vacuum GmbH (El Crossbeam IQ, Asslar, Germany)

Plasma Approach

Native plasma ions from a RF plasma have already been detected with this setup.

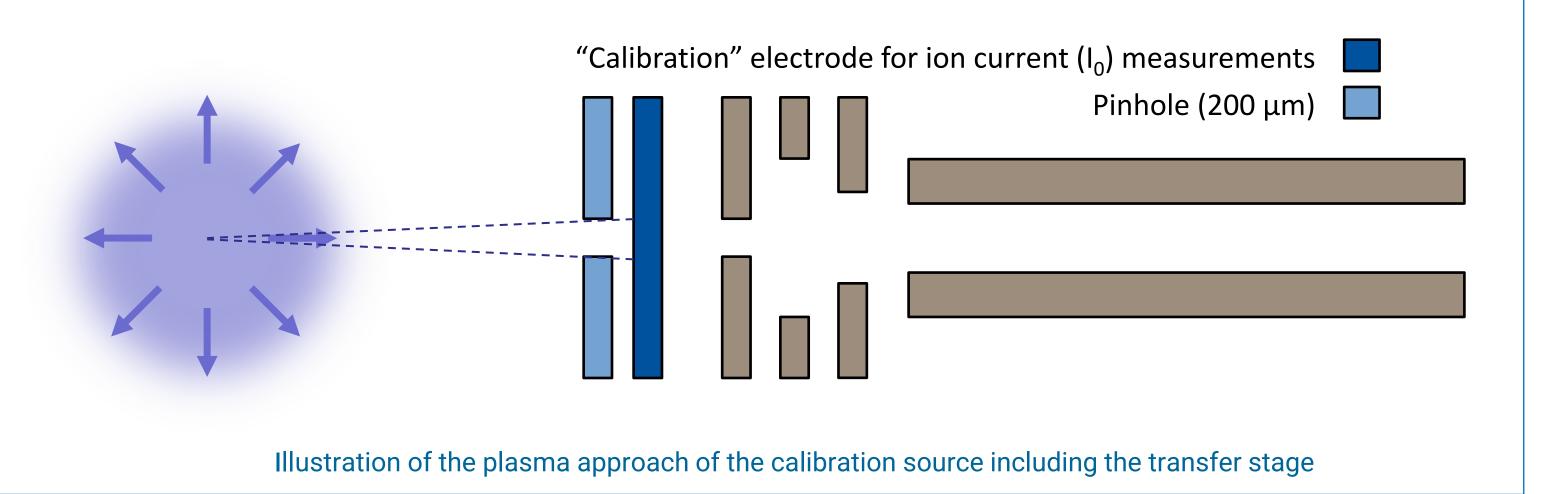
- Produces more ions than plasma use case.
- Confirmation of the calibration validity is required.
- Variable distance of the calibration unit and the last electrode of the El source.
 - Ideal configuration still needs to be figured out.
- SIMION simulations illustrate a correlation between the distance from ion source to calibration unit and the ions reaching the calibration electrode.
 - Green (5 cm distance): 6 % of generated ions reach the calibration electrode through the pinhole.
 - Blue (2 cm distance): 9 % of generated ions reach the calibration electrode through the pinhole.





SIMION simulations of the EI source and the calibration unit with two different distances

- - RF interferes with measuring electronics.
 - DC approach for more homogenous ion distribution in reaction chamber.
- Assumption: spherical expansion of a DC plasma in all spatial directions.
 - Only few ions with a certain solid angle reach the calibration electrode behind the pinhole of the calibration unit.
- Knowing the distance of the plasma sphere from the calibration electrode, the solid angle can be used to determine the total number of ions in the plasma sphere.



Conclusion & Outlook

The concept of the calibration ion source is still under development and further research is required to refine its applicability and reliability. A key aspect that remains to be addressed is the reproducibility of the measurements, particularly considering the necessity of fully venting the MS system each time the calibration unit is installed or removed. This process introduces potential variations in system conditions, which impact measurement consistency. A possible solution to this challenge is the implementation of a vacuum-compatible mechanism that enables the placement and removal of the calibration unit without breaking the vacuum.

Additionally, the assumption of a homogenous ion distribution on the calibration electrode needs further investigation, and whether it is required with this setup.

Regarding the EI approach, experiments need to be conducted to validate whether the simulation results agree with the measured ion currents on the calibration electrode. Additionally, the voltages applied to the electrodes of the ion source must be optimized and the impact of the calibration optics distance needs to be determined.

In terms of the plasma approach, it needs to be discussed whether a spherical ion distribution is realistic in this setup. Further experimentation is required, especially regarding the calibration optics geometry and its capability to detect low ion currents. Future studies should also focus on characterizing the properties and reproducibility of the calibration source with both approaches and optimizing the ion distribution to ensure robust and reliable calibration data for absolute quantification.

Acknowledgement References Generous support from the institute for pure and applied mass spectrometry (Wuppertal, Germany), as well "Probing ions from deeply embedded plasmas: From simulation to as financial support within the 14AMI project funded by the BMBF (16MEE0370) and the EU-CHIPS JU realization Investigation of a novel long distance ion transfer unit "; Poster (101111948) is gratefully acknowledged. presentation, Sanna Benter et al., Bergische Universität Wuppertal, 2023.