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INTRODUCTION

Gas Chromatography – Mass Spectrometry (GC-MS) is a powerful technique widely used in chemical and bioanalysis methods. GC provides separation of compounds essential for working with complex mixtures, while MS provides ultimate specificity to the method. GC-MS is currently considered the gold-standard technology to be used for the analysis of volatile and semi-volatiles compounds in complex mixtures, and commercial units are further enhanced with software operable databases of mass-spectral and retention index libraries. GC-MS is thus not surprisingly among the first MS-based field-deployable techniques, and a number of portable GC-MS systems are available commercially.

This work reports a further development of our miniature MEMS (Micro-ElectroMechanical Systems) GC-MS technology to include MEMS-based sample collector/pre-concentrator device with a goal to develop a portable GC-MS system for fieldable applications. Total GC-MS system volume is 1.25 cu.ft. @ 22 lb weight and it is capable of up to 8 hrs of battery operation with performance comparable to desktop GC-MS instruments.



A general view of μ GC/MS instrument in a waterproof enclosure of 16.9 x 9.6 x 13.4" dimensions

MEMS Pre-Concentrator

Advantages of MEMS Pre-Concentrator

- ❑ Compact integrated system including a heater and temperature sensor
- ❑ Lower power consumption
- ❑ Can be available with different sorbent phases
- ❑ Replaceable with a miniature programmable temperature vaporizing (PTV) inlet for analysis of standard liquid and solid-phase micro-extracted (SPME) samples

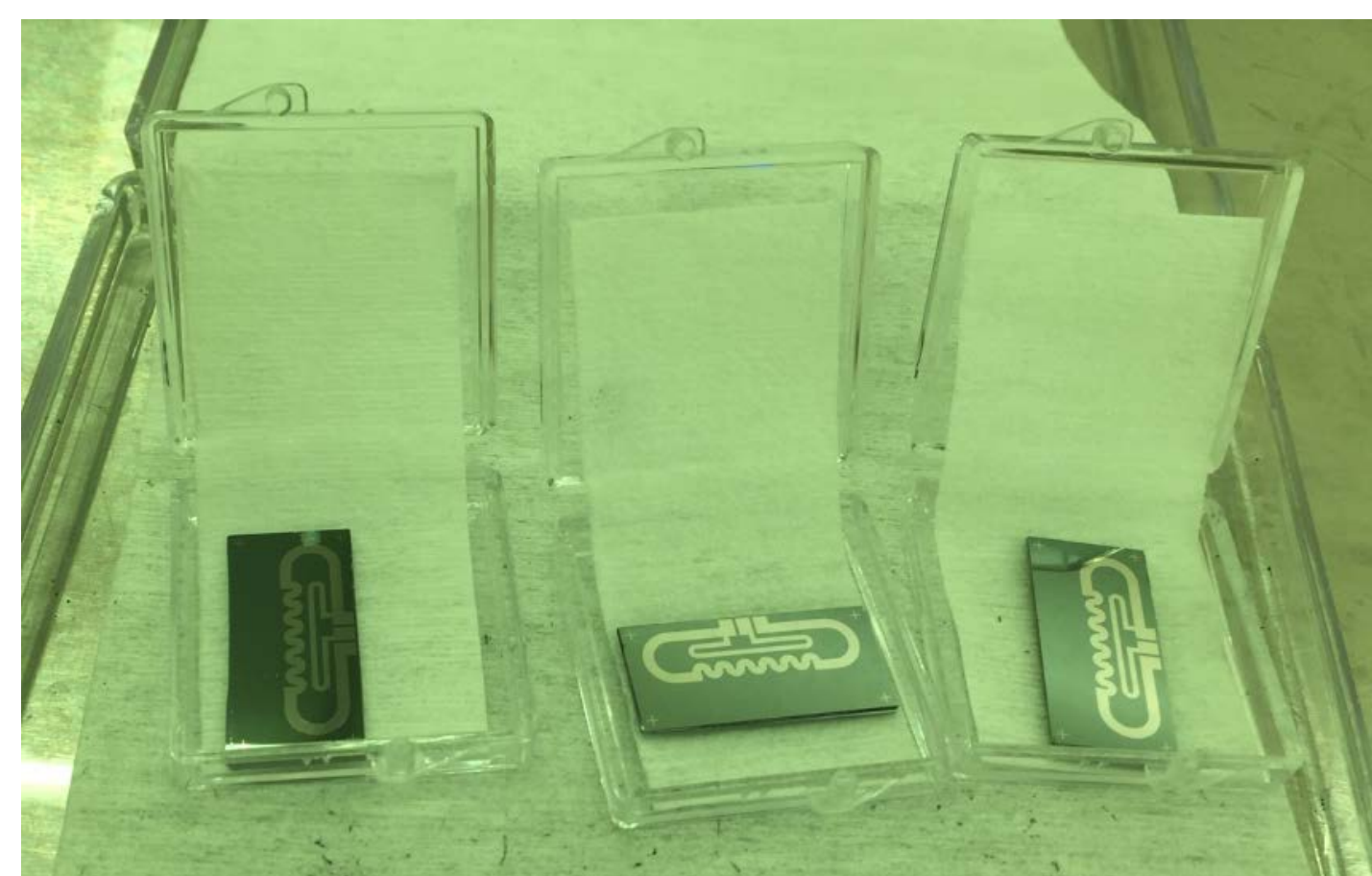
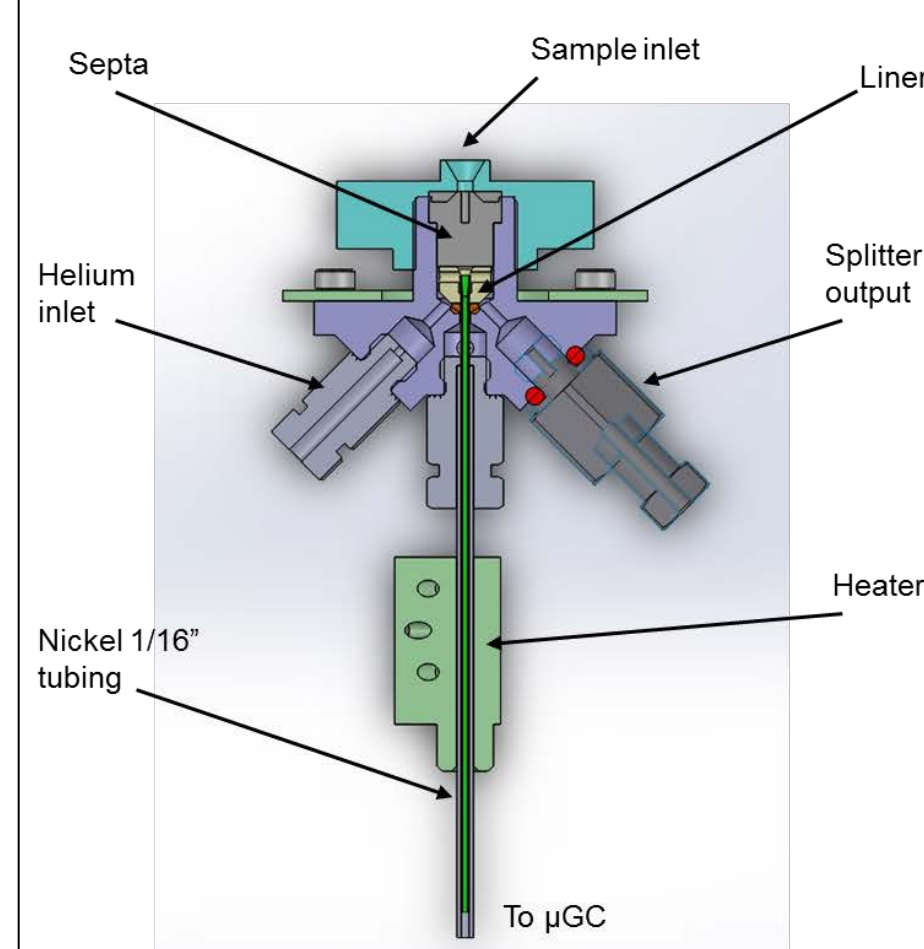
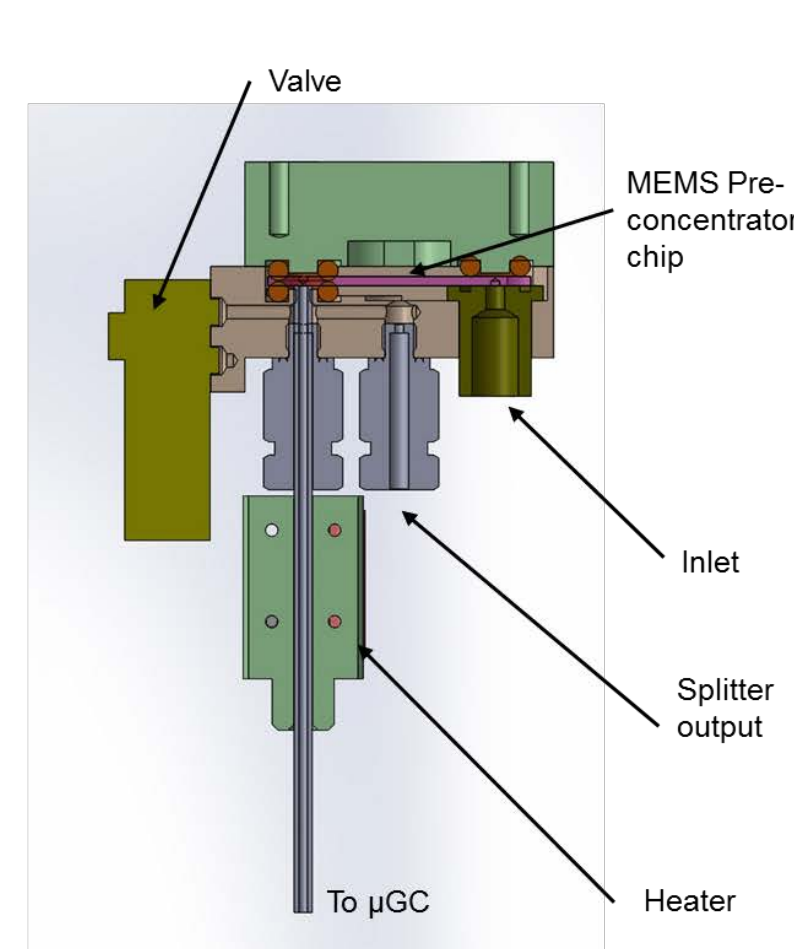


Image of Pre-concentrator chip (23 x 11 mm) filled with HayeSep D sorbent

PTV Inlet assembly



Pre-concentrator assembly



Replaceable PTV inlet and Pre-concentrator units

MEMS μ GC design

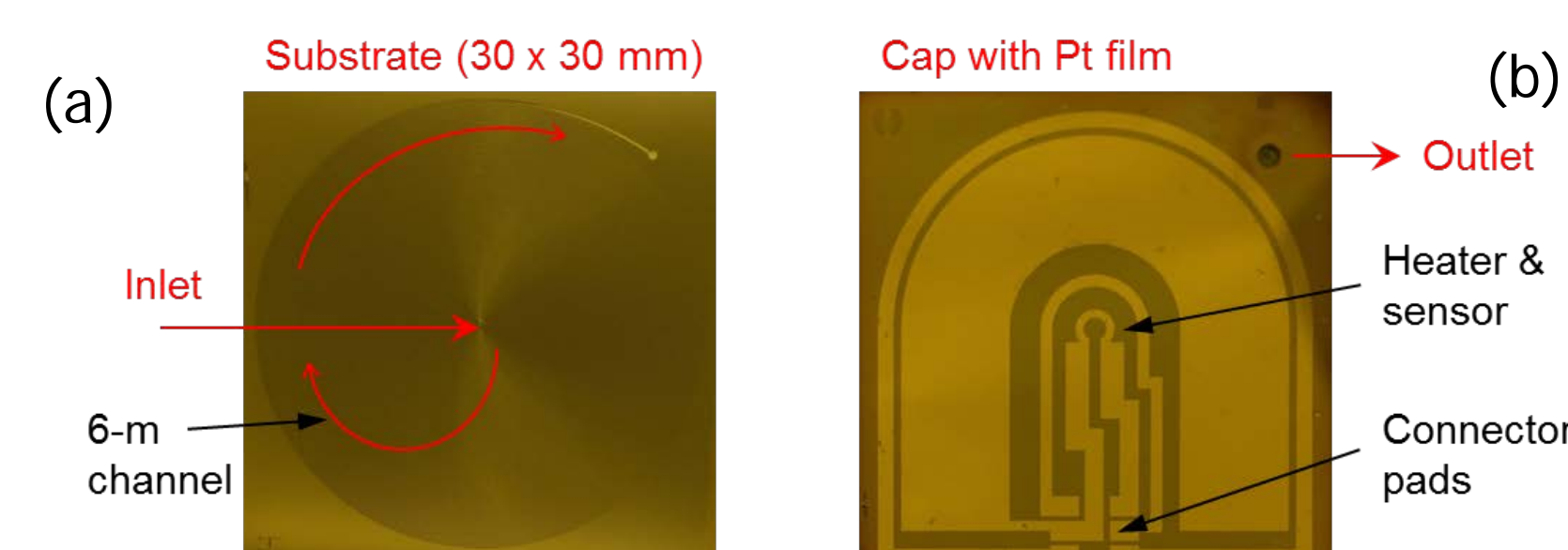
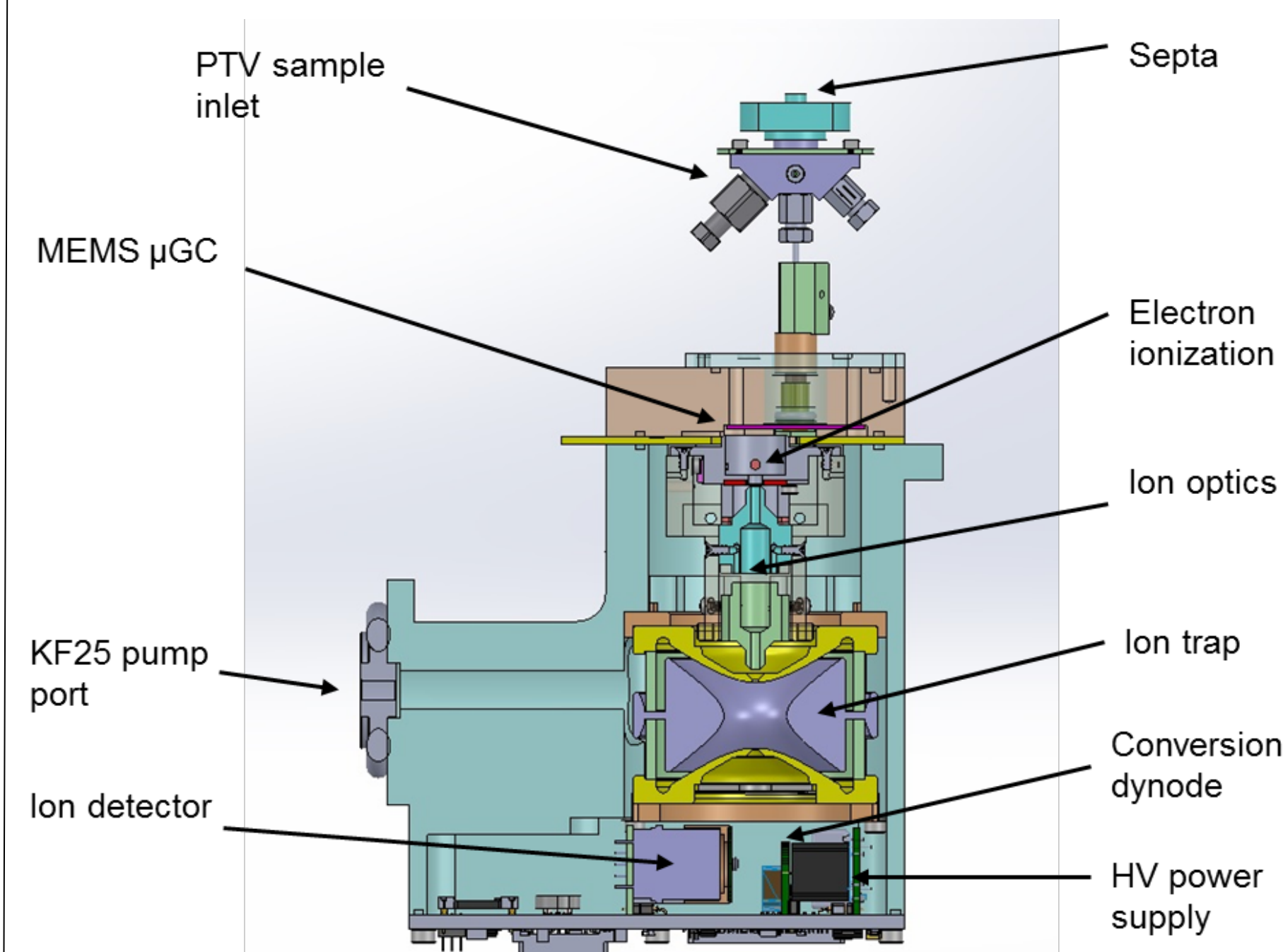


Image of a MEMS GC chip with an etched 6-m long channel before (a) and after bonding with a silicon cap with integrated Pt film heater and temperature sensor (b)

Miniature μ GC-MS

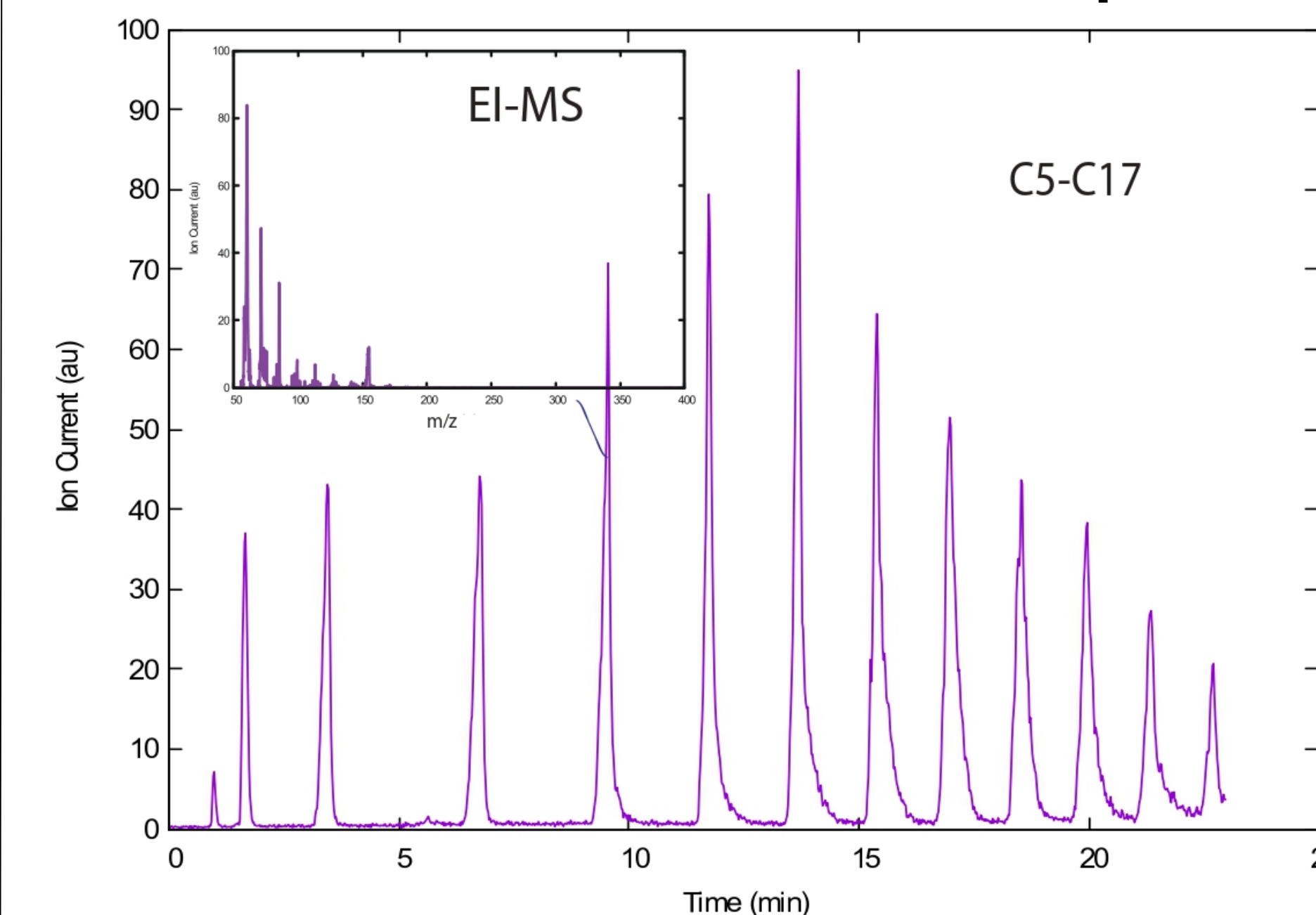
Main features of the ITMS design

- ❑ A standard size 3D ion trap cell is used without sacrificing effective storage capacity and performance
- ❑ Mass range is 30-500 da
- ❑ Better than unit mass resolution
- ❑ Electronic PCBs are used as vacuum walls to eliminate electrical feedthroughs
- ❑ All HV PS modules are inside a vacuum chamber
- ❑ RF generator with ferrite-based RF coil to reduce size and power consumption

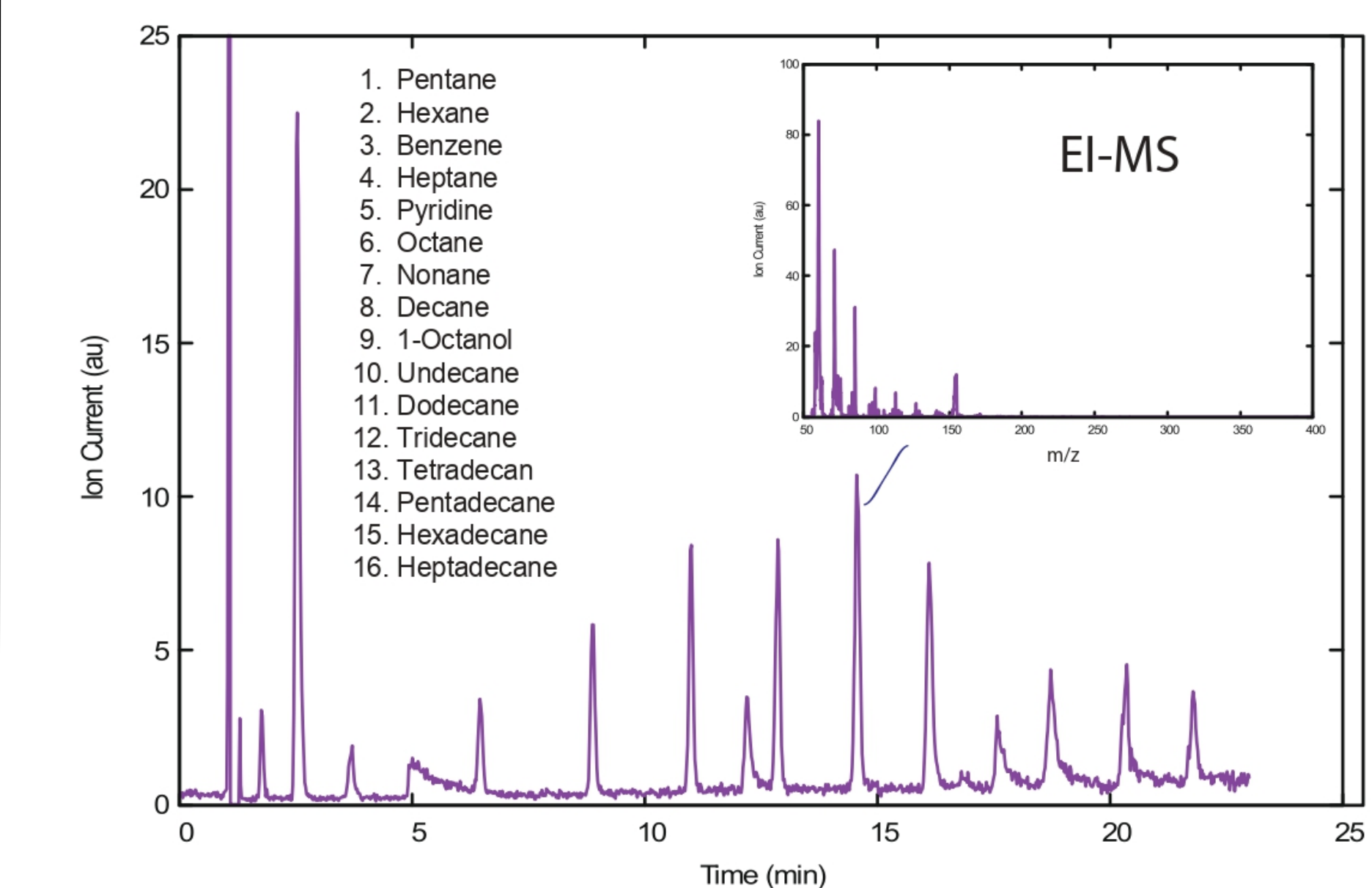


RESULTS

MEMS μ GC-MS performance



MEMS μ GC-MS TIC chromatogram for a equivolumetric 13-component mixture of Alkanes - C5 to C17

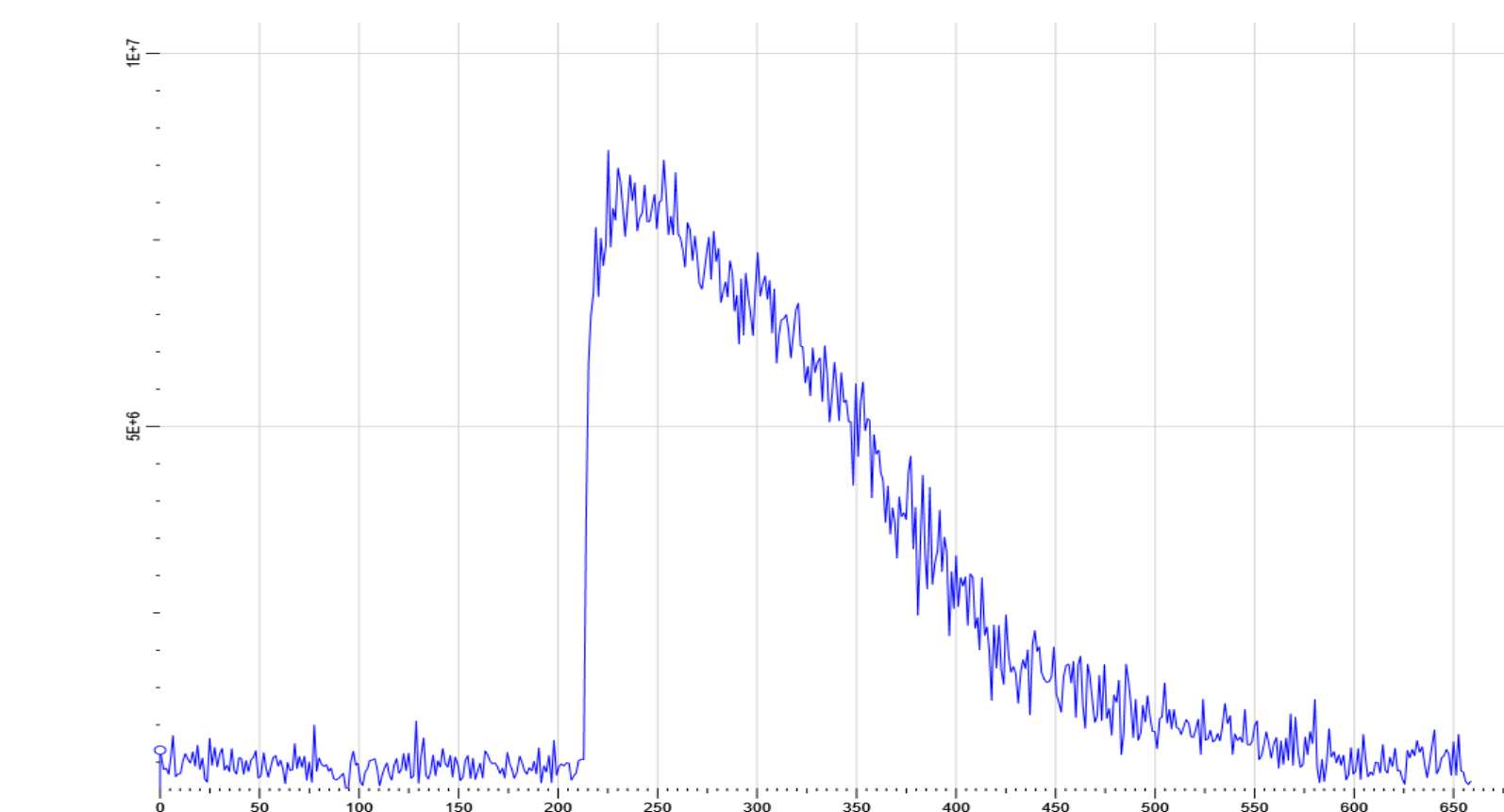


MEMS μ GC-MS TIC chromatogram for a equivolumetric 16-component mixture made of 10% volumetric solutions in Dichloromethane

MEMS μ GC column: 6-m long, 30- μ m wide channel, OV-1 stationary phase;
37 psi He inlet pressure (52 psia total GC pressure drop);
Split ratio 1:150;
0.5 μ L of mixtures injected;
GC run: 120 sec pre-run @ 40°C + 10°C/min ramp to 180°C + post-run @ 180°C;
MS scans: from 45 to 400 Da

Pre-concentrator performance evaluation

Pre-concentrator sorbent: HayeSep D;
0.5 μ L of pyridine injected outside GCMS;
Desorption temperature: step from 22 to 180°C;
20 psi He inlet pressure (35 psia total GC pressure drop);
Split ratio 1:150;
GC run: 60 sec pre-run @ 40°C + 20°C/min ramp to 180°C + post-run @ 180°C;
MS scans: from 45 to 400 Da



μ GC-MS chromatogram of pyridine sample obtained from MEMS pre-concentrator