Mass Spectrometers and Gas Chromatographs for Planetary Exploration

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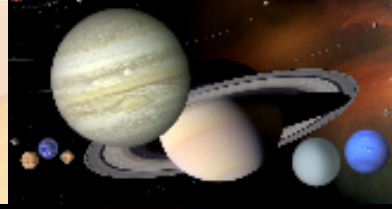
IPPW9 Short Course – Mass Spectrometers & Gas Chromatography



Science questions addressed by mass spectrometers and gas chromatograph mass spectrometers

Mass spectrometer types and how they work

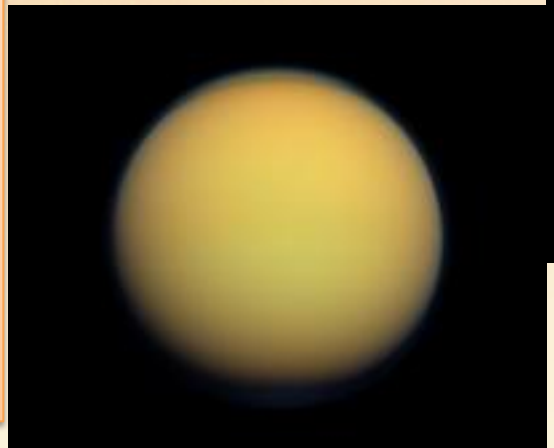
Chemical separation with chromatography

Diverse chemical and physical environments

Examples – past, present, future exp's

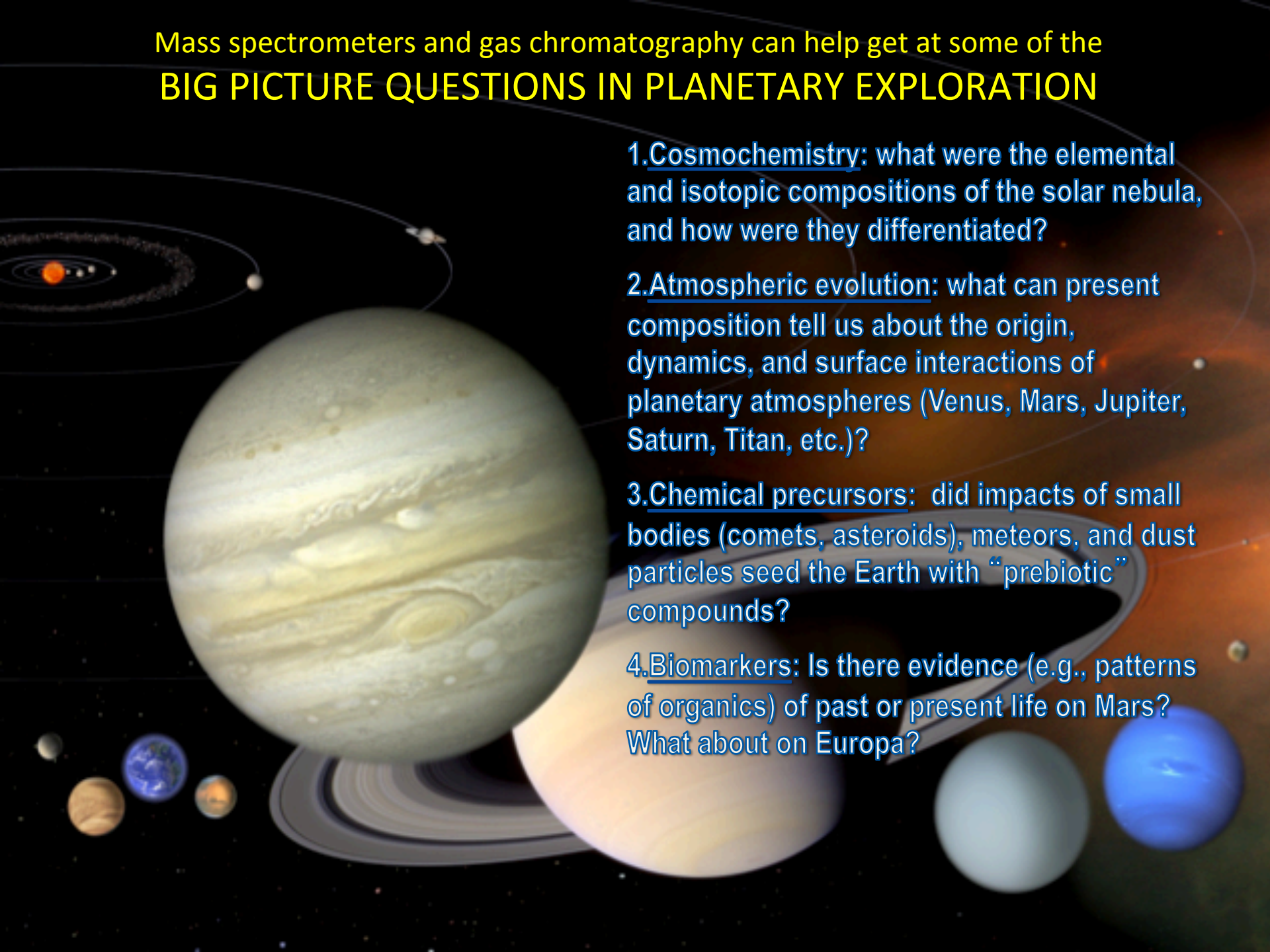
- Galileo Probe Neutral Mass Spectrometer
- Mars Science Laboratory Sample Analysis at Mars Instrument Suite
- Mars Organic Molecule Analyzer developed for the ExoMars Mission
- Two-Step Laser Tandem MS for Organics detection
- Ultra-high mass resolution MS

Probes with MS or GCMS instruments sent to Venus, Jupiter, Mars, & Titan



Mass spectrometers and gas chromatography can help get at some of the

BIG PICTURE QUESTIONS IN PLANETARY EXPLORATION



1. Cosmochemistry: what were the elemental and isotopic compositions of the solar nebula, and how were they differentiated?

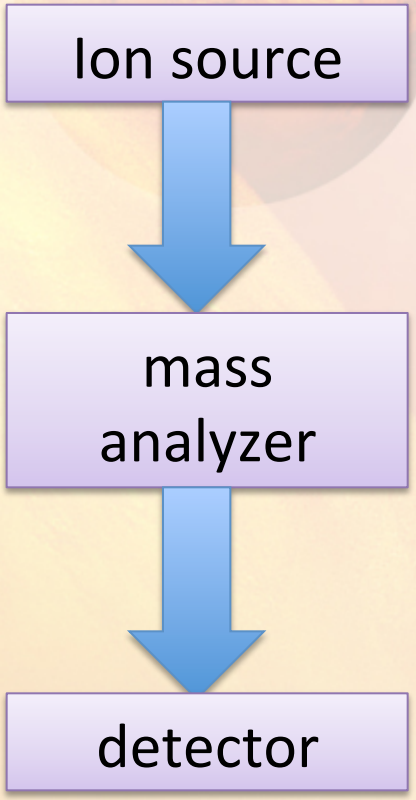
2. Atmospheric evolution: what can present composition tell us about the origin, dynamics, and surface interactions of planetary atmospheres (Venus, Mars, Jupiter, Saturn, Titan, etc.)?

3. Chemical precursors: did impacts of small bodies (comets, asteroids), meteors, and dust particles seed the Earth with "prebiotic" compounds?

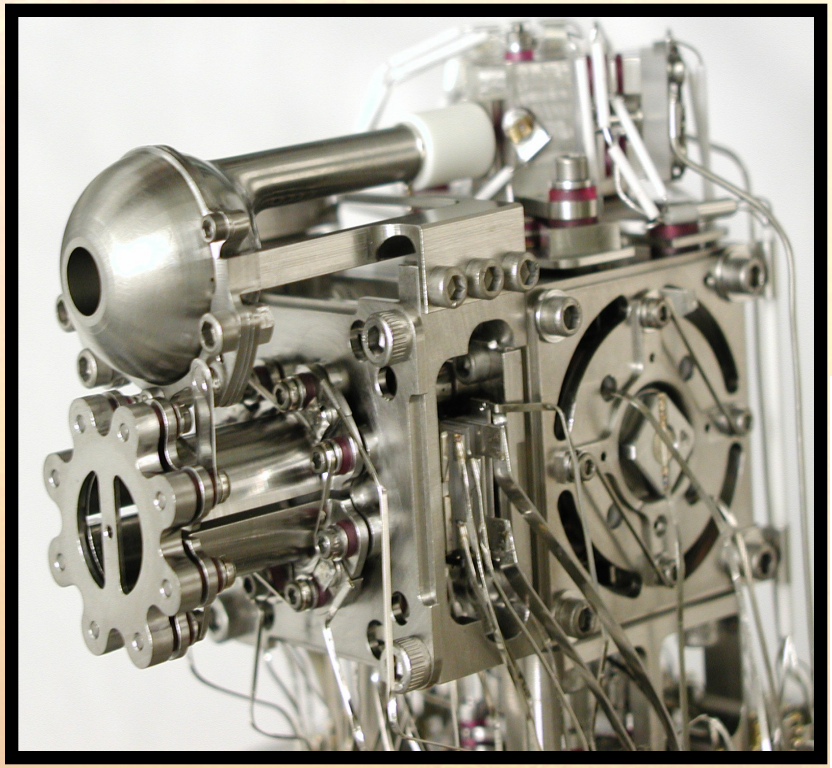
4. Biomarkers: Is there evidence (e.g., patterns of organics) of past or present life on Mars? What about on Europa?

Mass spectrometry – the basics

- **A mass spectrometer measures the mass-to-charge ratio (m/z) of atoms and molecules in the gas phase**
- **If your sample is not in the gas phase, you have to do something to volatilize it**
  - heat, sputter, laser desorb, spray, even scratch! Also, pressure.
- **If your sample is not yet ionized you need to ionize it**
- **Value of m/z used to identify atoms and molecules**
  - At "unit" mass resolution you can distinguish any two species separated by 1 amu (or 1 Da), such as ^{12}C and ^{13}C
  - At higher mass resolution you can distinguish species with the same "nominal" mass, such as CO and N_2 (~28 Da)
  - Peak intensities (I) ~ relative abundances; "Pattern" of *I* vs. *m/z*
  - The pattern of isotopes is a "fingerprint" of a species
  - The pattern of fragments is another "fingerprint"



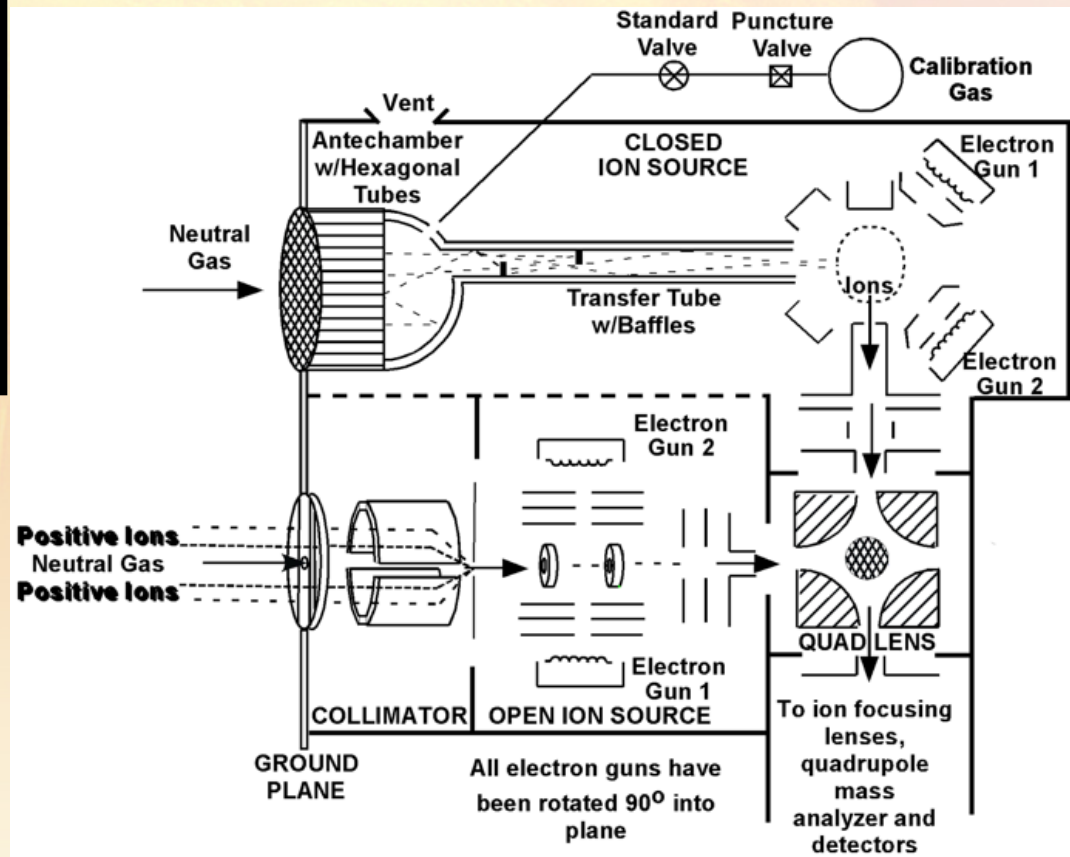
Example dual ion source with redundant W/3%Re filaments



CONTOUR NGIMS with both OPEN and CLOSED source

OPEN → for surface reactive species

CLOSED → for high sensitivity for surface inert species



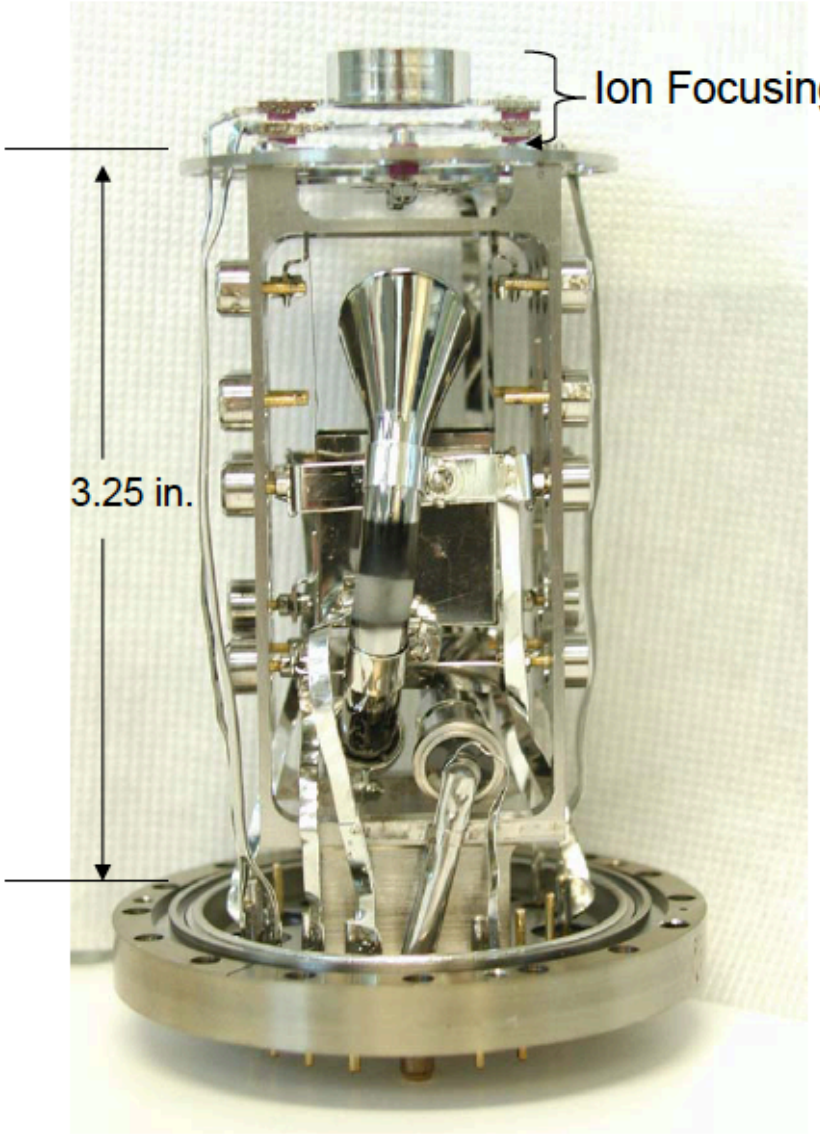
Developed at NASA Goddard for

- Comet Rendezvous Asteroid Flyby Mission
- Cassini INMS
- CONTOUR NGIMS
- LADEE NMS
- MAVEN NGIMS

Example - redundant channeltron pulse counting detector

Gain ~ 50 million

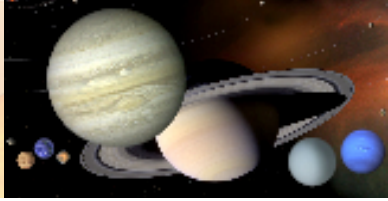
Channeltron Secondary Electron Multipliers

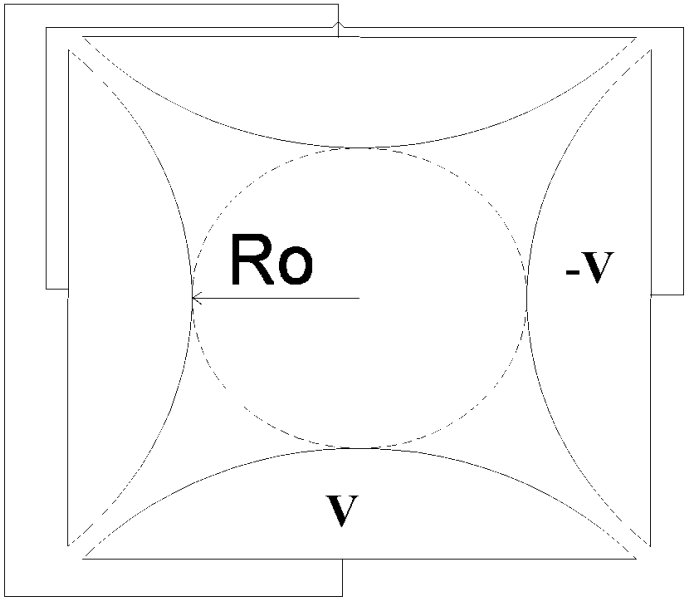


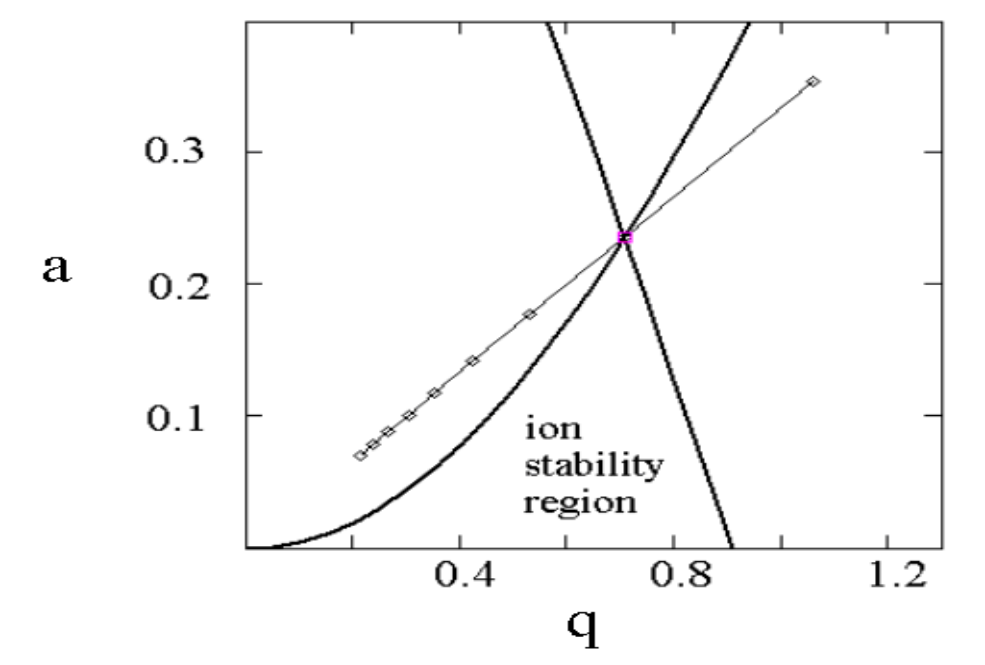
Great Variety in Mass Analyzer Types

Type → ↓Feature	Quadrupole (QMS)	Ion Trap (ITMS)	Time-of-Flight (TOF-MS)	Sector MS
Scan Type	**Filter**: one m/z at a time stable in AC field, passes to det.	**Spectrograph**: Trap all ions in AC field, then scan out to det.	**Spectrograph**: All ions reach det. at t ~ $(m/z)^{1/2}$	**Filter**: one (or more) m/z in E-B field pass to det.
Ionization Type(s)	Mostly continuous (EI, ESI, ICP)	Continuous (EI/ESI) or pulsed (laser)	Mostly pulsed (laser, pulsed EI)	Mostly continuous (EI, ICP)
Mass Range	Lab: 1-2000+; Mini/ Flight 1-500	Lab: 50-4000; Mini/Flight: 50-1000	Infinite (practically 1 to ~10 kDa)	Several hundred (and up)
Mass Res. (typical)	Typically unit (very low crosstalk)	Typically unit (can have high mass acc)	From 10^2 to 10^5 (path length & V)	Typically unit (very low crosstalk)
MS/MS?	no	yes (MS^n)	yes (pseudo)	no
Planetary Examples	Galileo/GPMS, Cassini/INMS, Huygens/GCMS, MSL/SAM, MAVEN/ NGIMS	Rosetta/Ptolemy, ExoMars/MOMA	Rosetta/ROSINA-ROTF, Rosetta/ COSAC, Phobos/ LIMA-D (Phobos Grunt/LASMA)	Viking GCMS, Rosetta/ROSINA-DFMS, Beagle II/ GAP, Phoenix/ TEGA-MS

Quadrupole MS







Hyperbolic Electrodes give field of form
$\Phi(x, y) = (V_{dc} + V_{ac} \cos(\Omega t)\,(x^2 - y^2)/R_o^2$

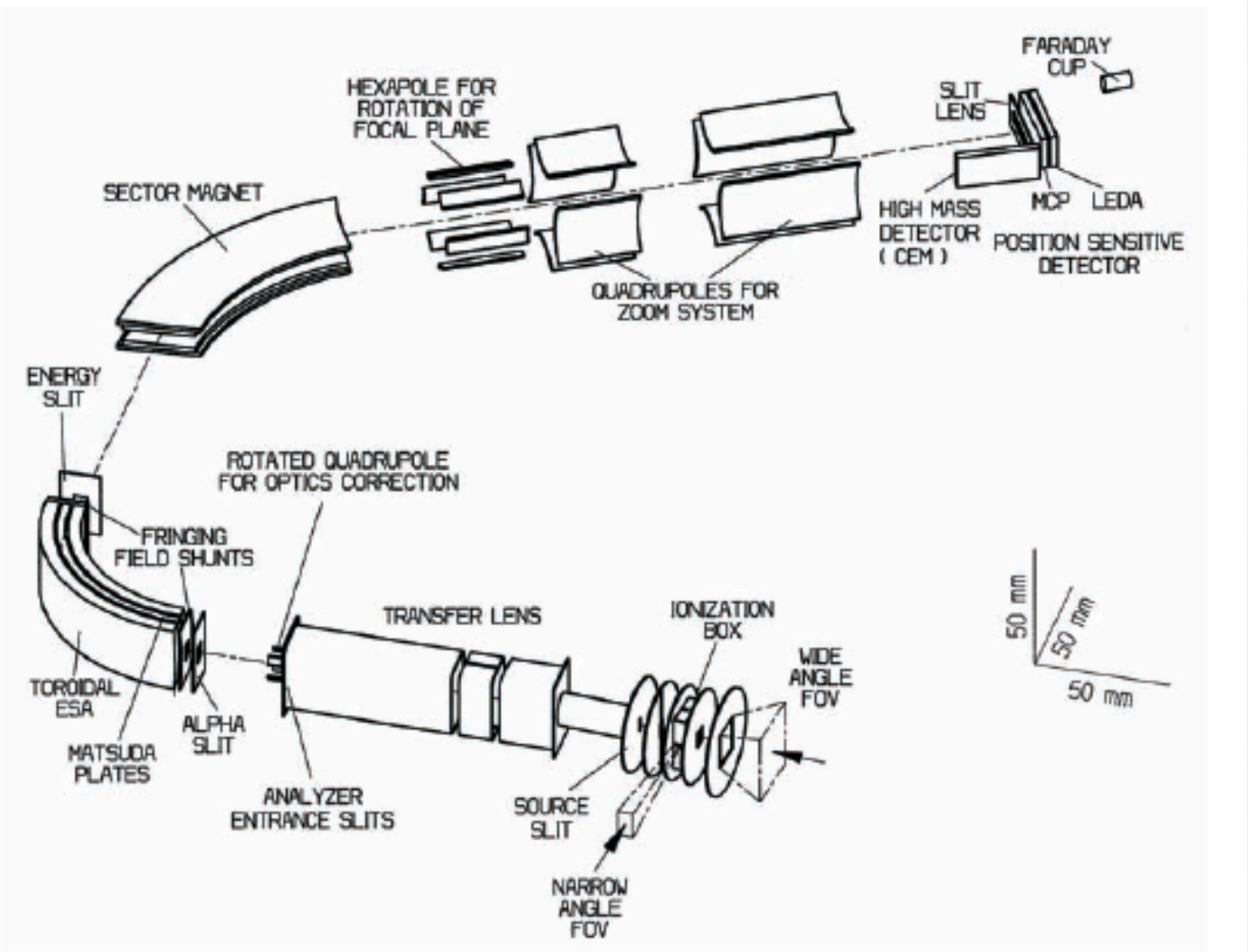
Dimensionless parameters
$a = 8eV_{dc}/mR_o^2\Omega^2$
$q = 4eV_{ac}/mR_o^2\Omega^2$.

Shown by Paul and Steinwedel [1953] that solutions of the equations of motion

$$d^2x/dt^2 + [V_{dc} + V_{ac} \cos(\Omega t)\, 2ex/mR_o^2] = 0$$
$$d^2y/dt^2 - [V_{dc} + V_{ac} \cos(\Omega t)\, 2ey/mR_o^2] = 0$$

of a charged particle with mass m and charge e in this device -> to two types of solutions **stable** and **not stable**. May use MULTIPLE frequencies.

Example of double focusing MS Balsiger et al. (2007)



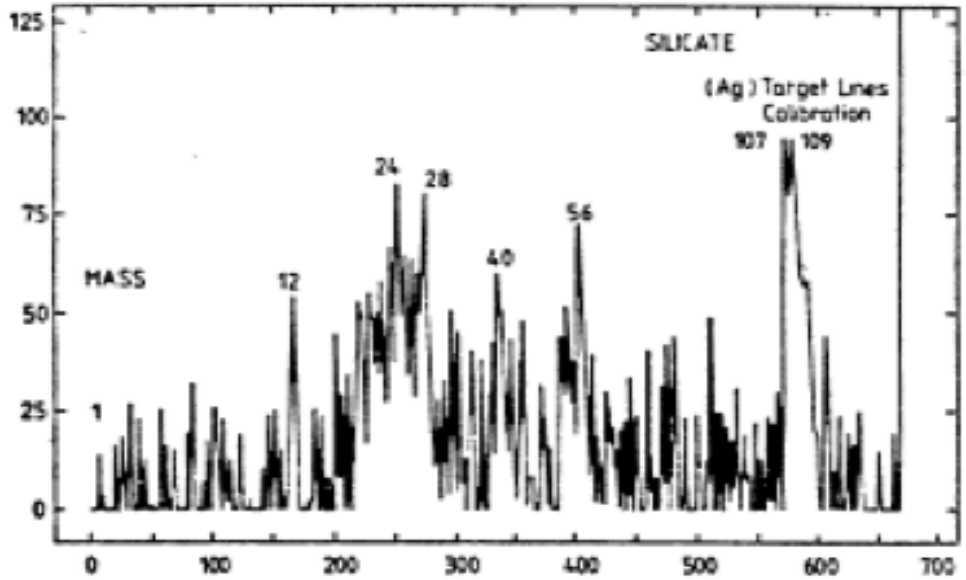
With ion path perpendicular to magnetic field B

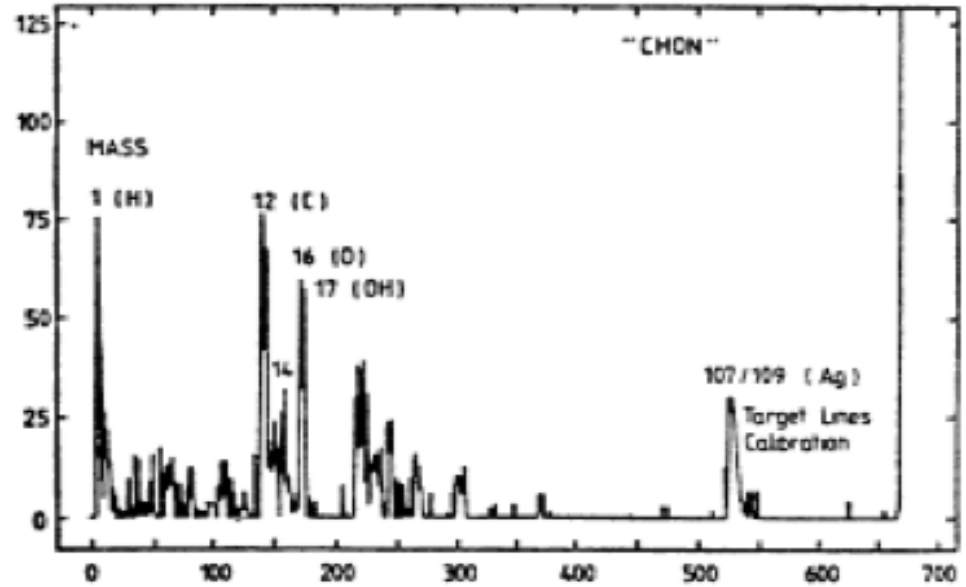
$m/q = B^2 r^2_M/(2U)$

Magnet can DISPERSE ions on focal plane

One could change B or U but for flight application U is easier to change and a permanent magnet used to create B

Examples of time of flight MS

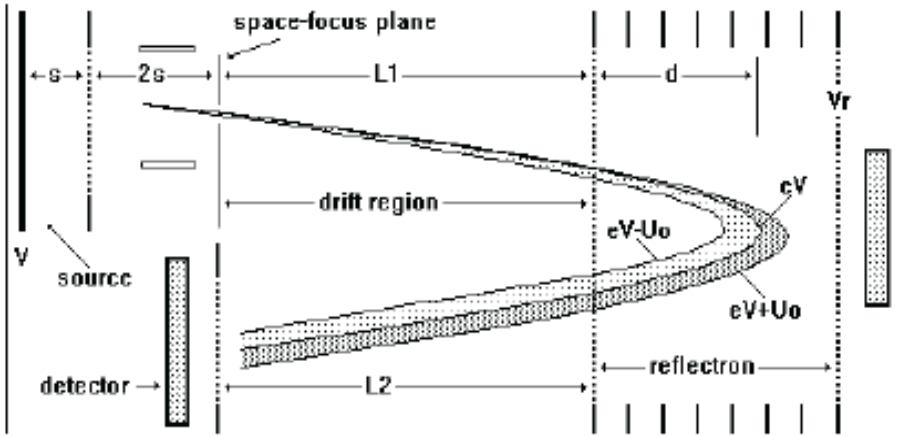




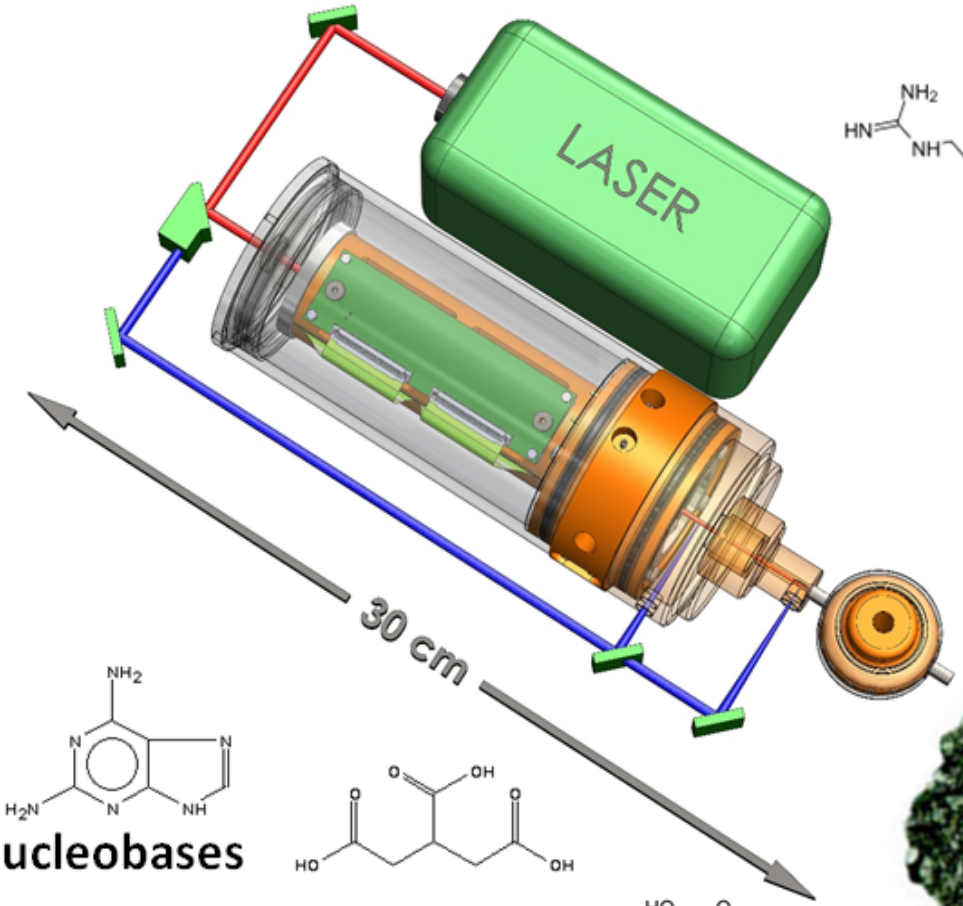
Giotto Particulater Impact Analyzer (PIA) secured molecular and elemental composition measurements at comet Halley in 1986.

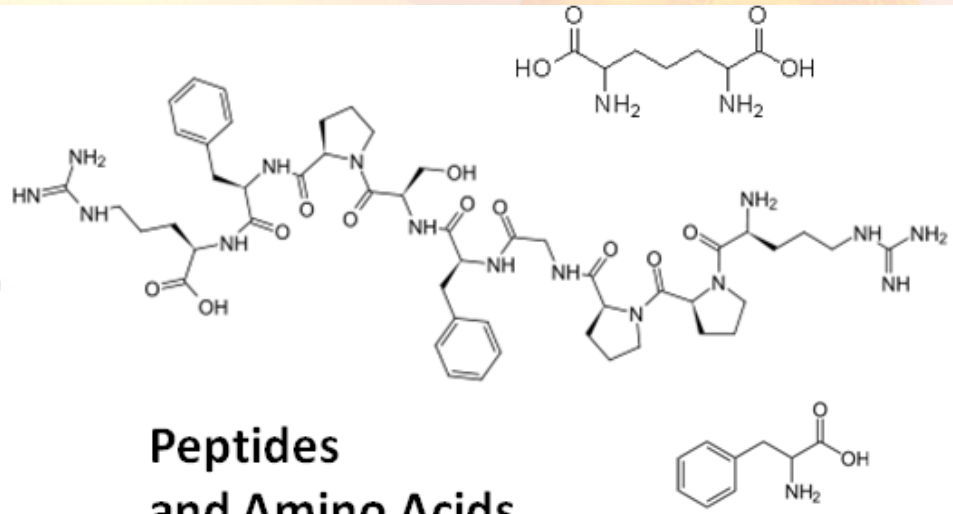
Laser desorption resonance ionnization mass spectrometer under development for Rb-Sr in situ dating on the moon or Mars. S. Anderson et al.

Reflectron figure – Cotter et al., 1999

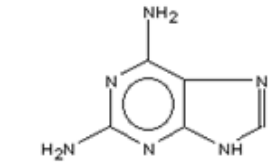


Two-Step Laser Tandem MS for *In Situ* Organics Detection

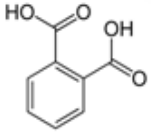




Peptides and Amino Acids



Nucleobases



Carboxylic Acids

Polycyclic Aromatic Hydrocarbons

11-PIDD11-0044 - Getty, Brinckerhoff, Cornish, Ecelberger, Callahan, Elsila, Zare, Yu, Trainer

Two-Step Laser Tandem MS for *In Situ* Organics Detection

Development of a highly capable *two-step laser tandem mass spectrometer* (L2MS) optimized to study the diversity and structure of nonvolatile organics in solid samples on missions to outer planetary satellites and small bodies. The L2MS combines multiple laser pulses and a precision ion gate to enable both post-ionization of organics and tandem analysis of dissociation products *to achieve tunable selectivity and sensitivity*.

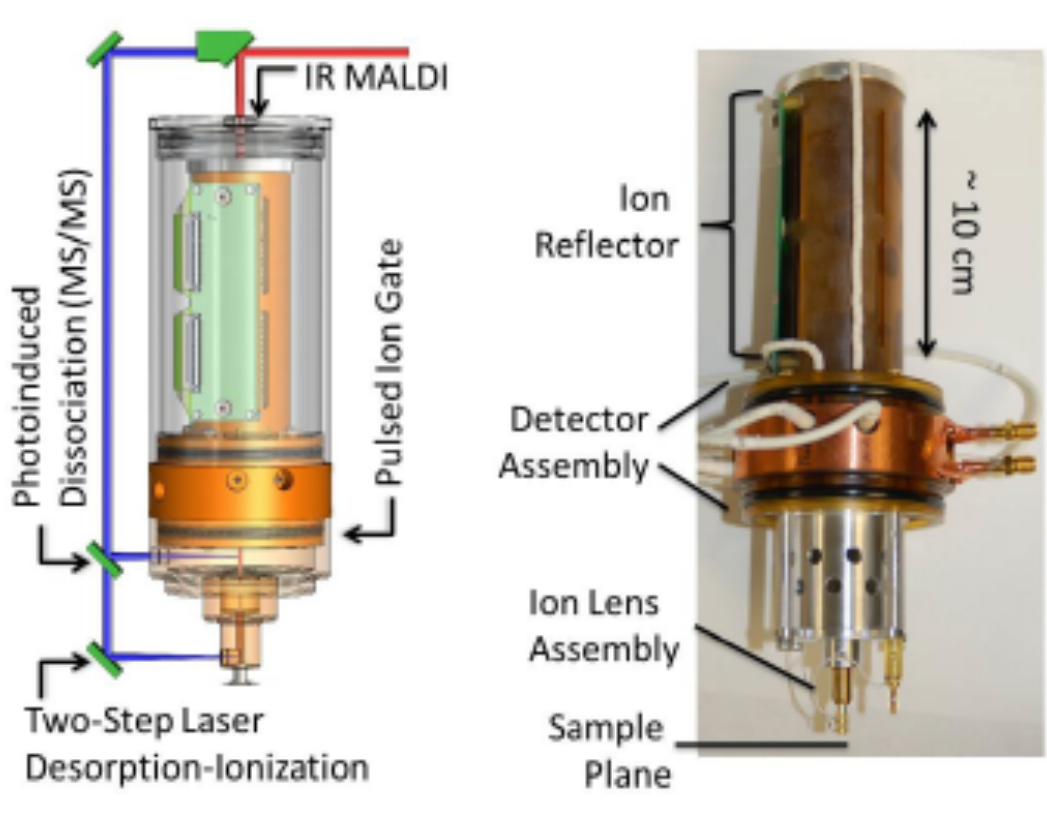
1. Infrared matrix assisted laser desorption/ionization (IR MALDI) to significantly increase the sensitivity to the parent ions of high molecular weight organics in complex samples, especially those with water ice matrices where sample preparation would not be required;

2. Two-step laser desorption/ionization with maximal detection selectivity for aromatic organics via ultraviolet resonance-enhanced multiphoton ionization (UV REMPI); and

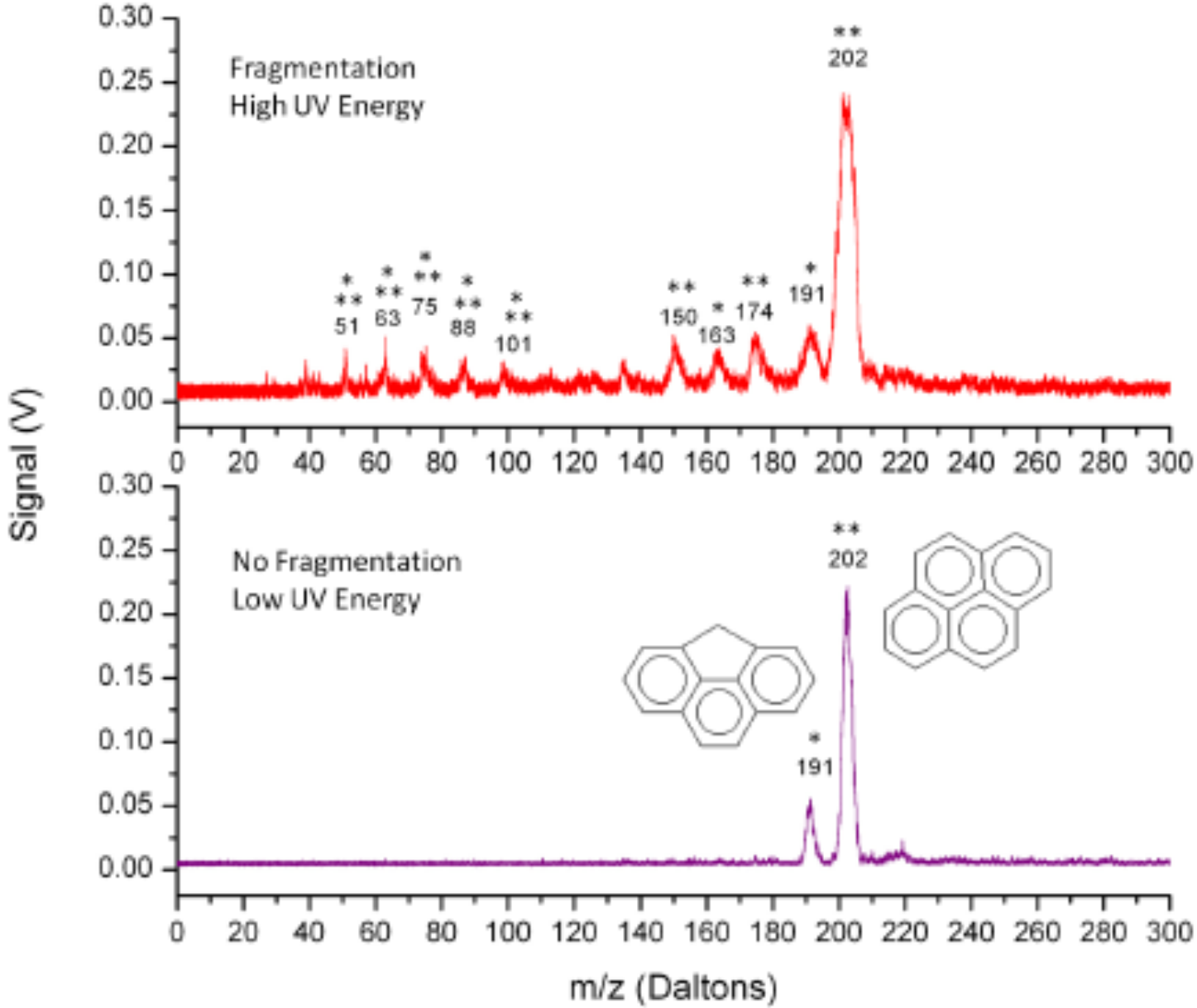
3. Tandem mass spectrometry, or MS/MS, to reduce the intrinsic complexity of indiscriminate laser ionization by selectively isolating and fragmenting parent ions in the TOF-MS flight tube with photo-induced dissociation (PID) and precision gating.

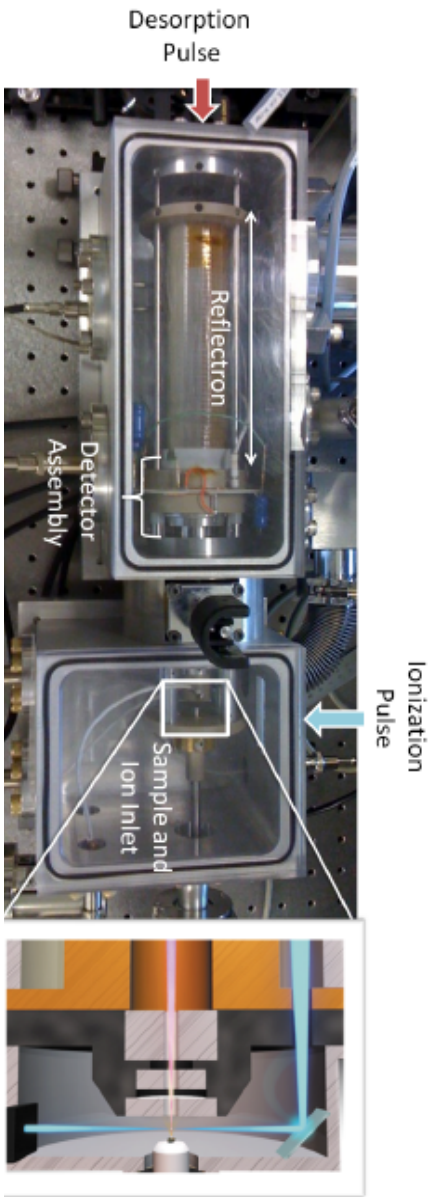
L2MS Instrument Design

- Leverage reversible-polarity TOF-MS development
- Additional features/ capabilities:
  - 3 um pulsed primary laser (OPO)
  - 266 nm ionization pulse
  - 266 nm photodissociation pulse
  - Cooled sample stage for measuring doped ices

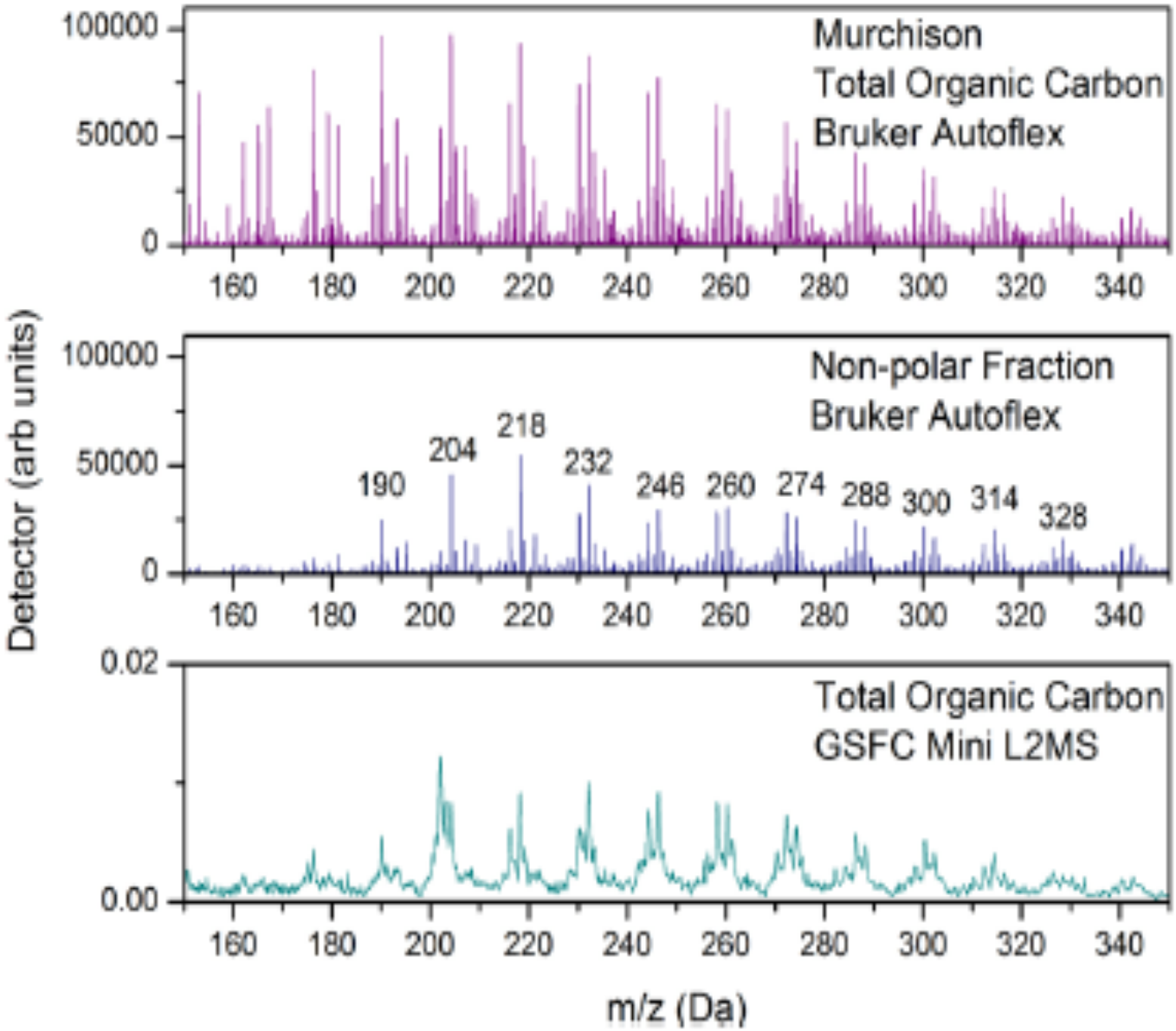


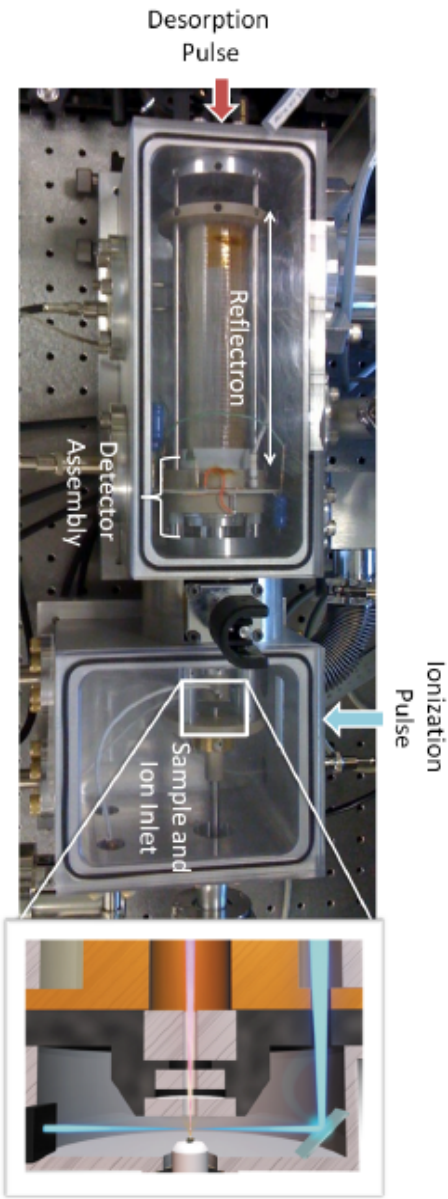
Preliminary L2MS Demonstration: Pyrene Standard



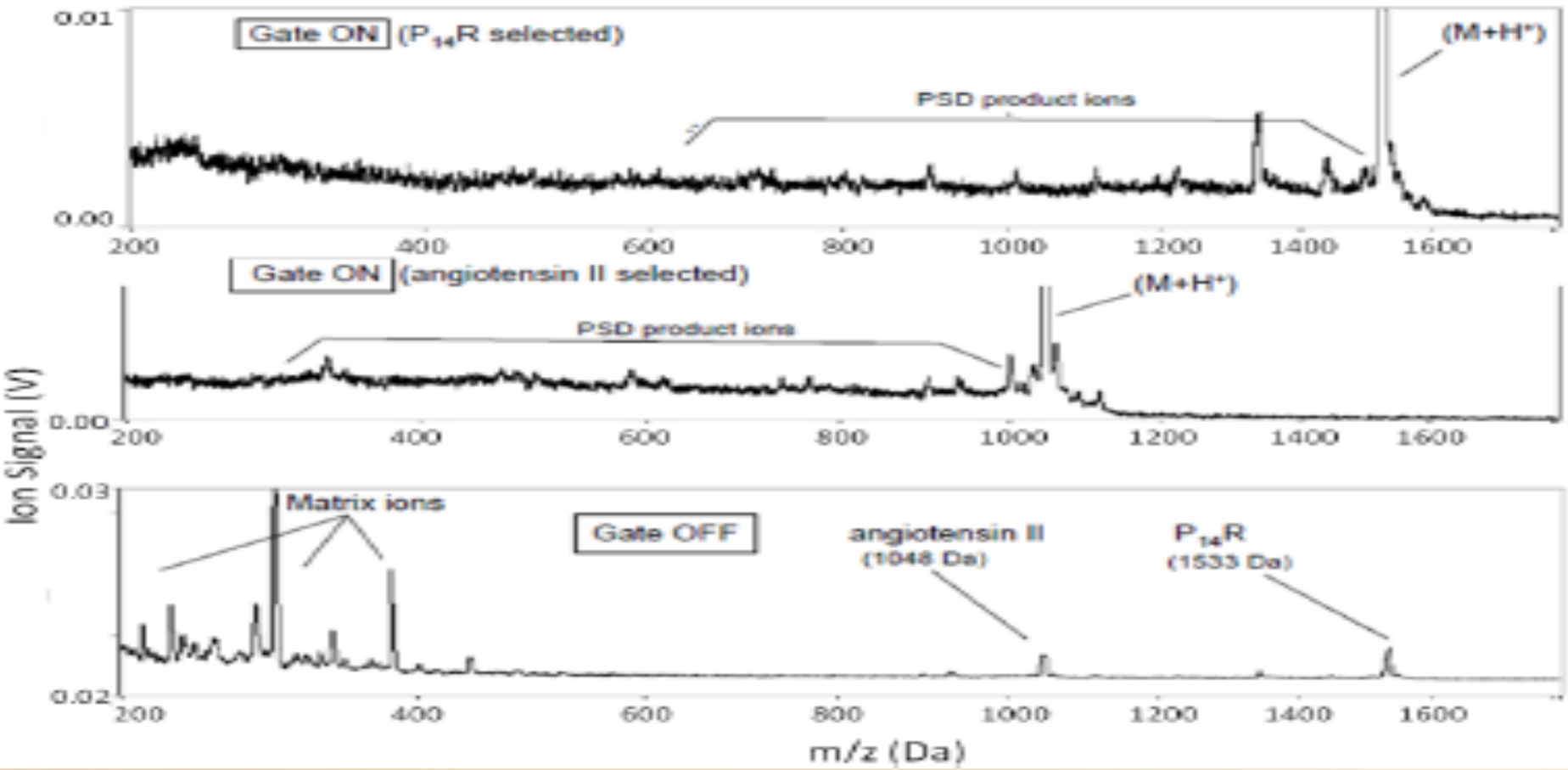


Preliminary L2MS Demonstration: Meteoritic Extracts





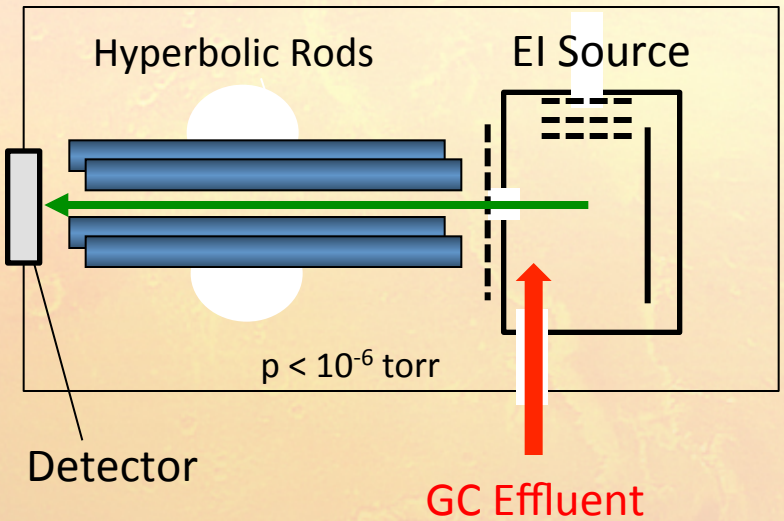
Precision Ion Gating



- Pulsed ion gate can select a peak of interest, rejecting dominant ions that obscure minor species
- For parent species that dissociate easily, the parent mass and its product ions can be detected in one measurement in pseudo-tandem MS

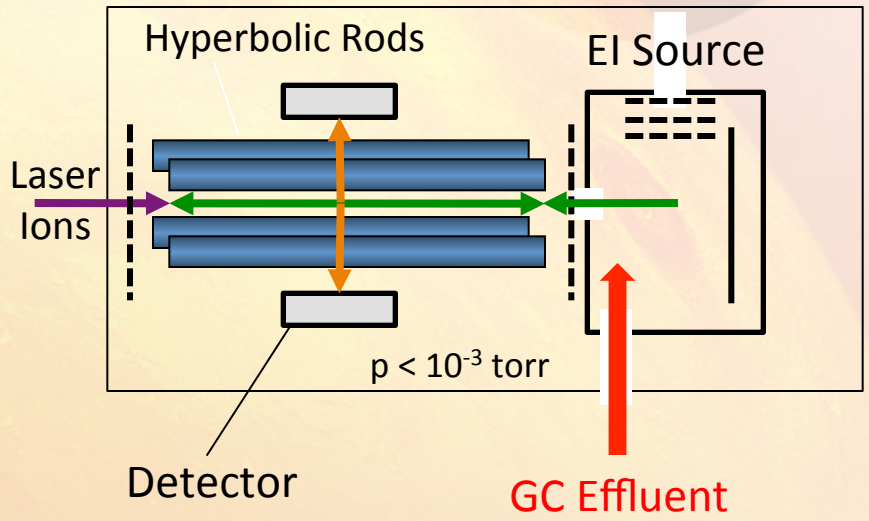
Quadrupole and Ion Trap MS

SAM
Quadrupole MS



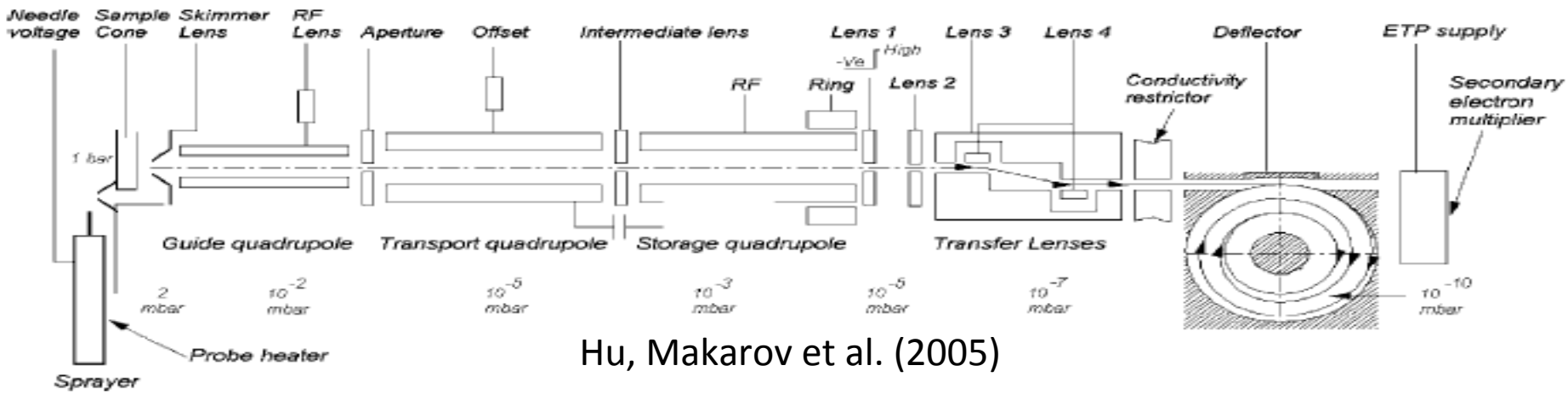
One m/z value passes through rods to detector at given set of rod voltages; spectrum formed by scanning voltages.

MOMA
Linear Ion Trap MS

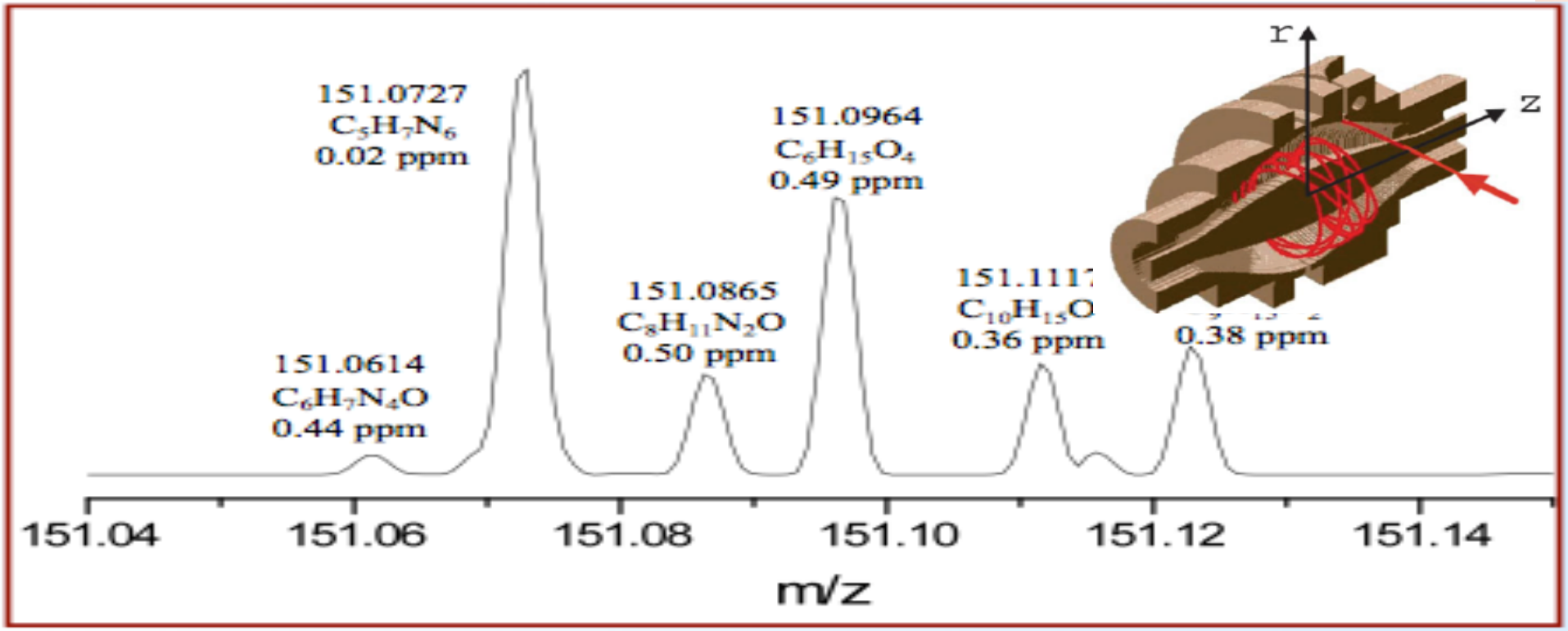


All ions over m/z range are trapped in field of rods; spectrum formed by exciting ions radially to detector by scanning voltage.

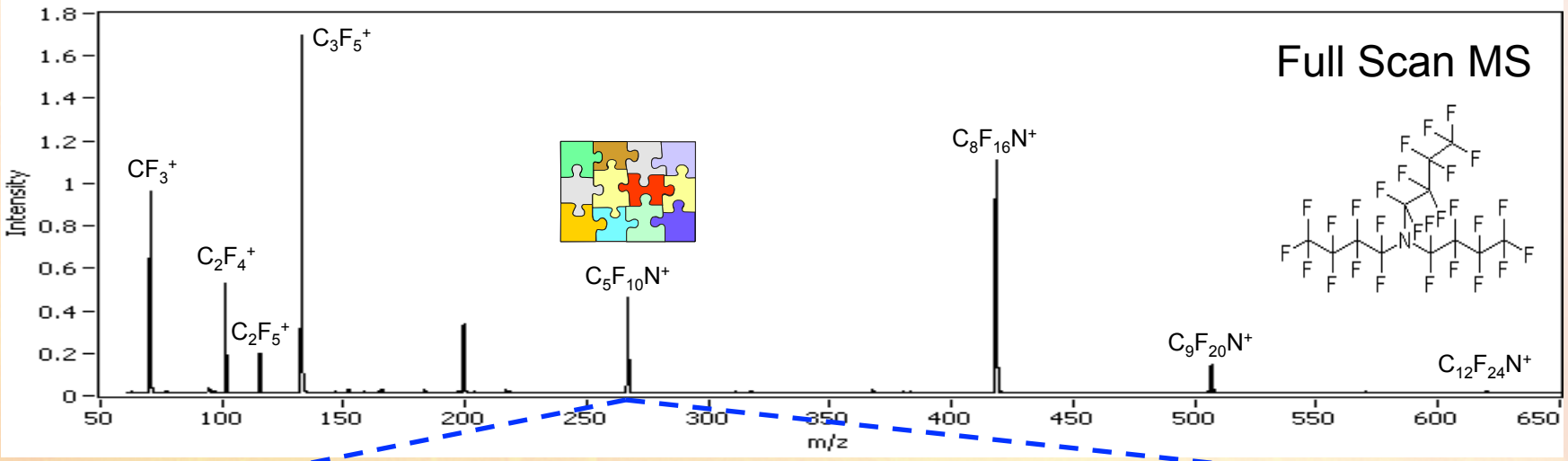
Ultra-high resolution mass spectra gives exact chemical composition

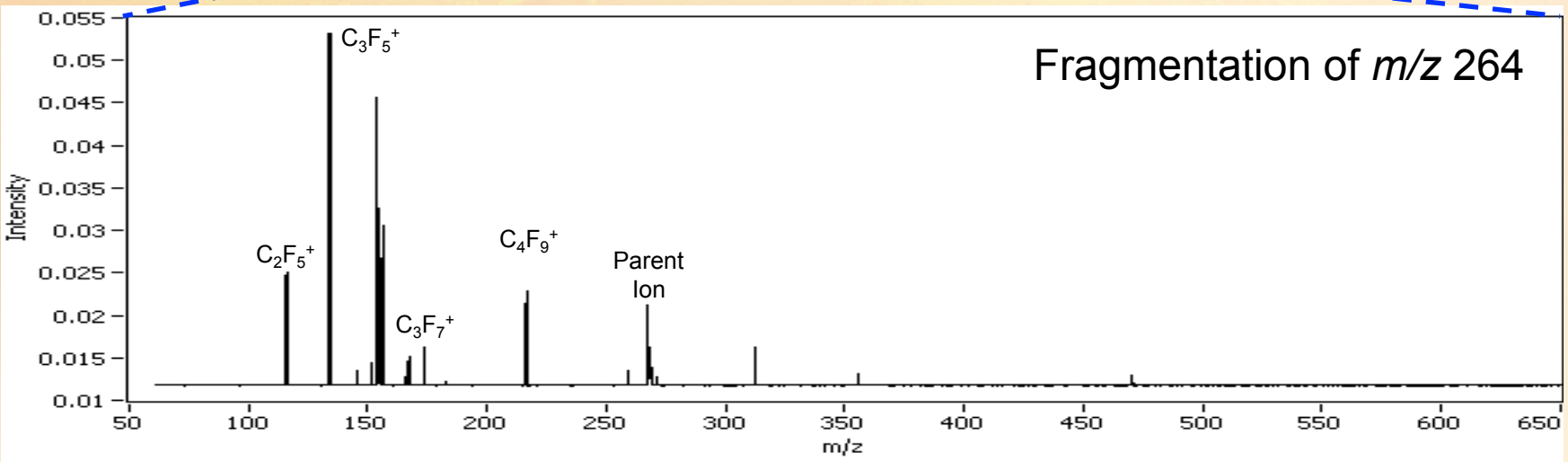


Hu, Makarov et al. (2005)

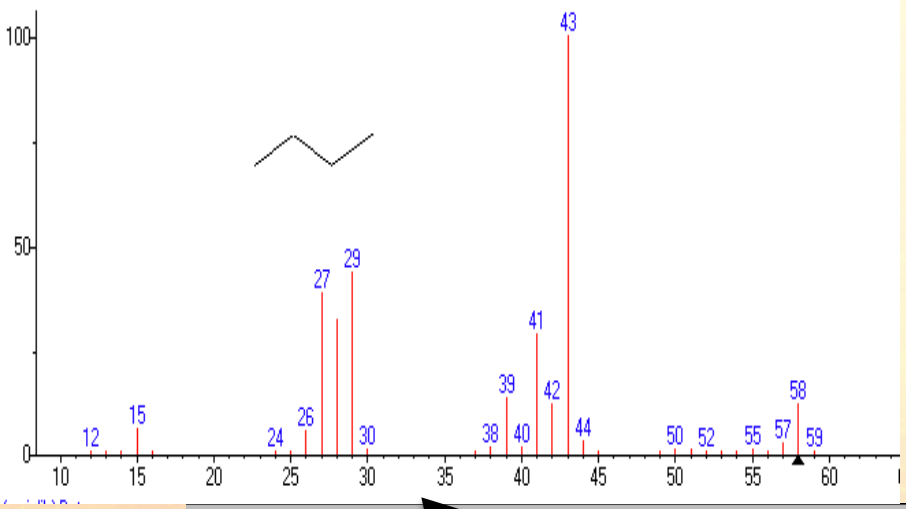


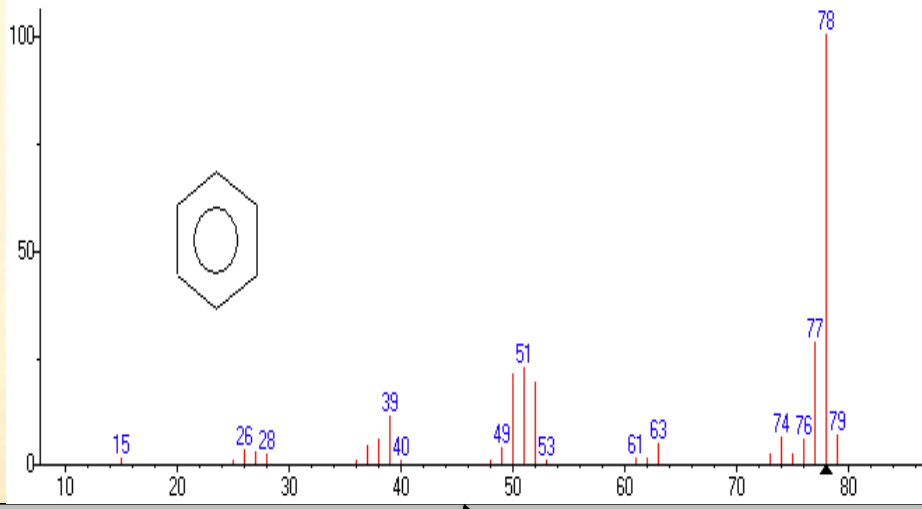
Example mass spectra

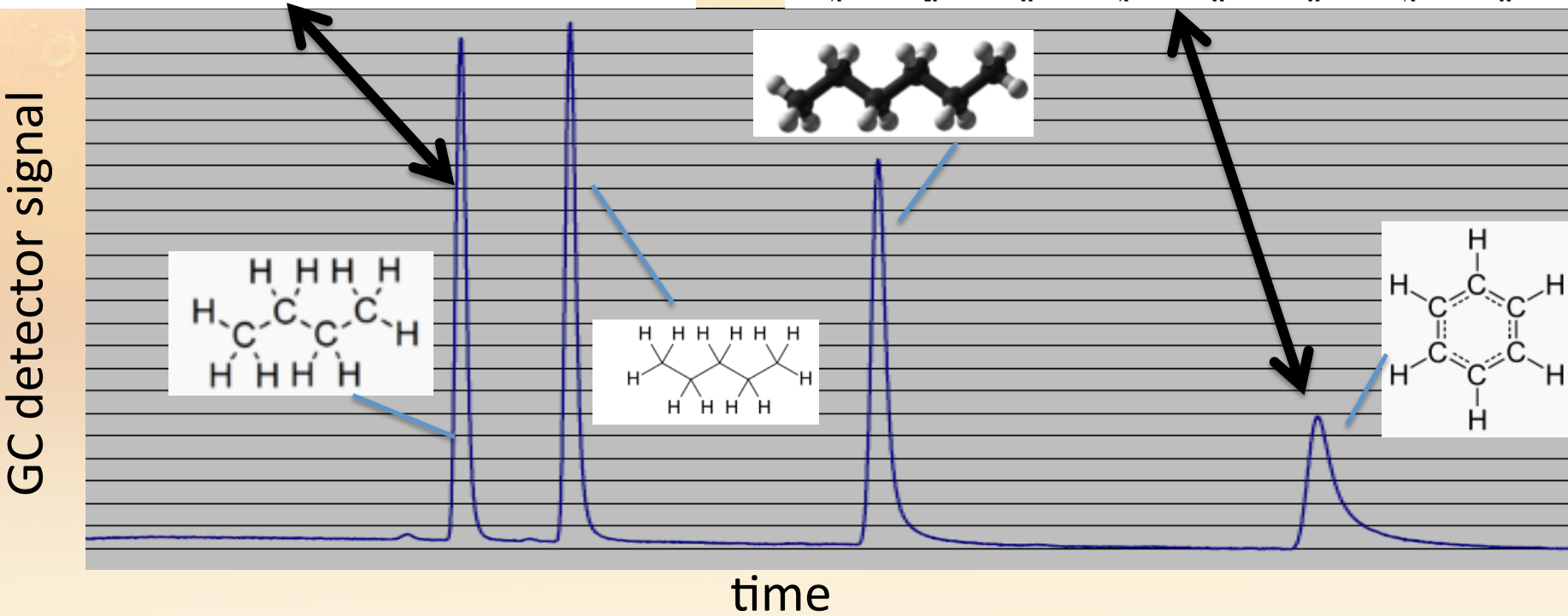




GCMS separation in time provides definitive molecular identification







Harsh environments encountered at and in transit to planetary systems

Temperature

- Venus surface 735 K
- Mars surface 214 K
- comet interior temperature 140 K

Pressure

- to 100 bar in the giant planets in future probes
- Venus surface pressure 92 bar
- Mars surface pressure 6.4 millibar

Radiation

- magnetospheric heavy ions and electrons
- cosmic ray protons and heavy ions
- solar flare protons and heavy ions

Vibration and transient loads

- harsh launch environment
- entry of Galileo Probe 107,000 miles/hour (230 g)

Lifetime

- several months to many years (1960's and 1970's planetary spacecraft are still operating

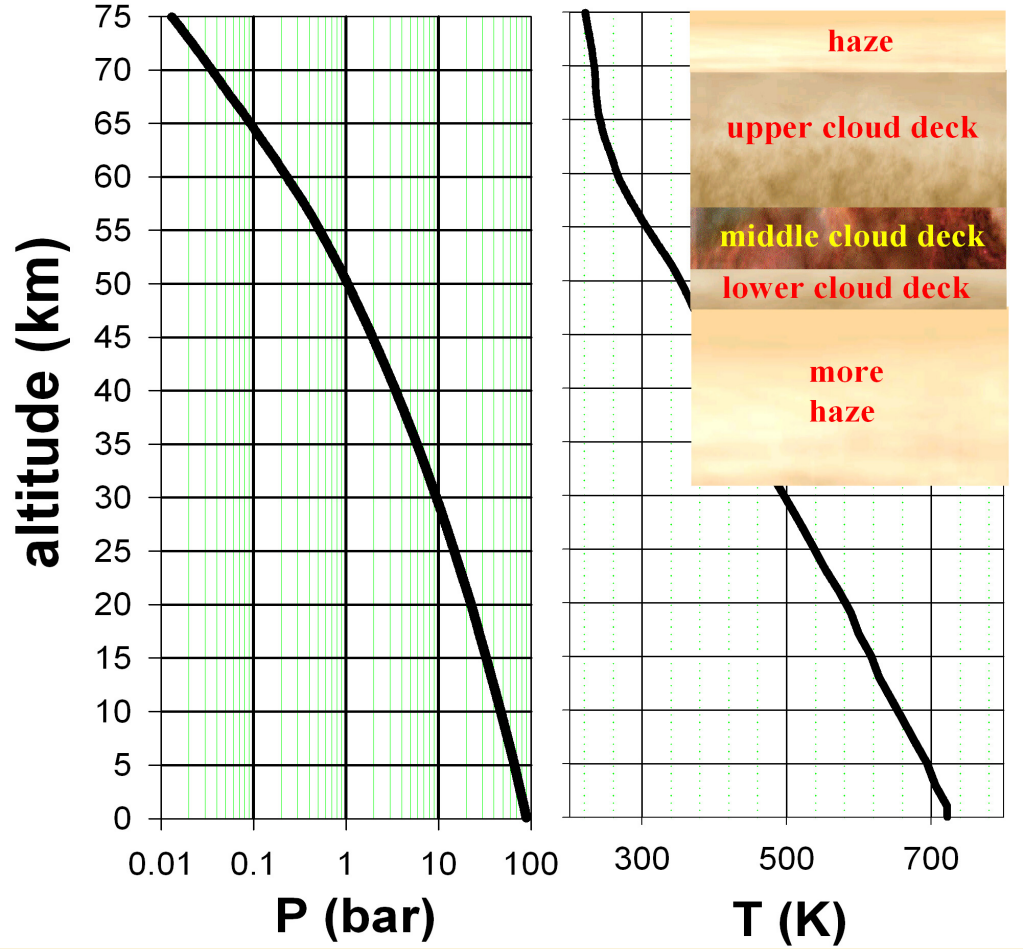
Motivation for improved mass spectrometer measurements at Venus is to address fundamental issues of terrestrial planetary formation and evolution

The assignment

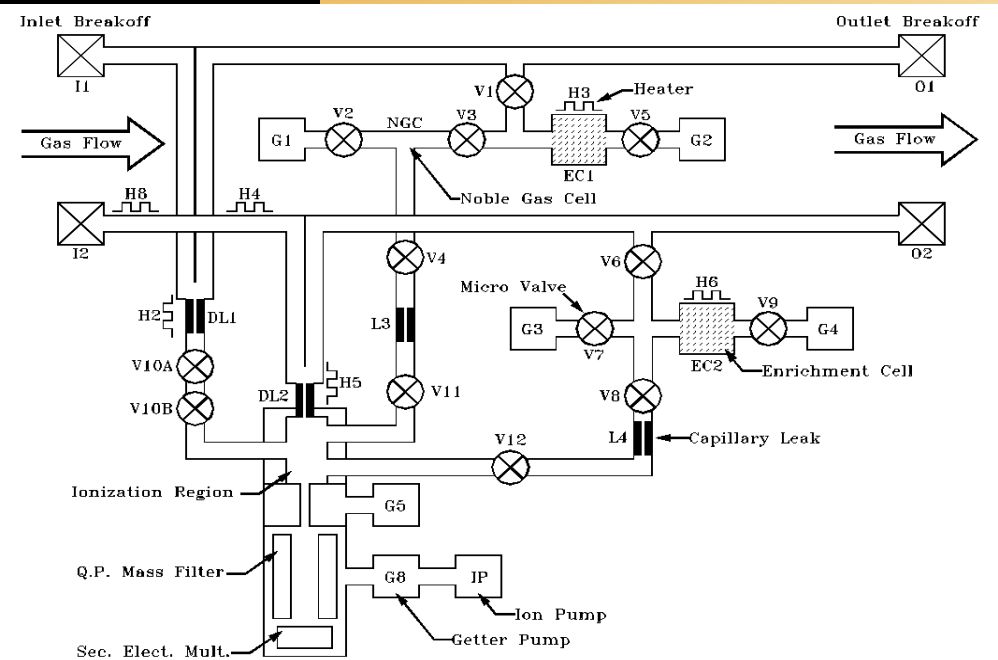
- to make precise (better than 1 %) measurements of isotope ratios and accurate (5-10%) measurements of abundances of noble gas
- to obtain vertical profiles of trace chemically active gases from above the clouds all the way down to the surface

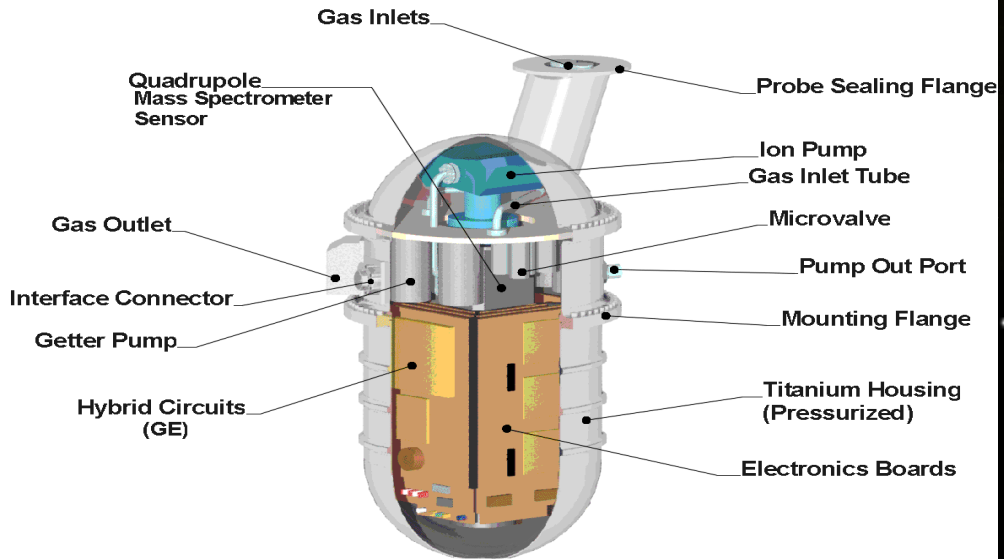
The challenge for Venus probe mass spectrometry

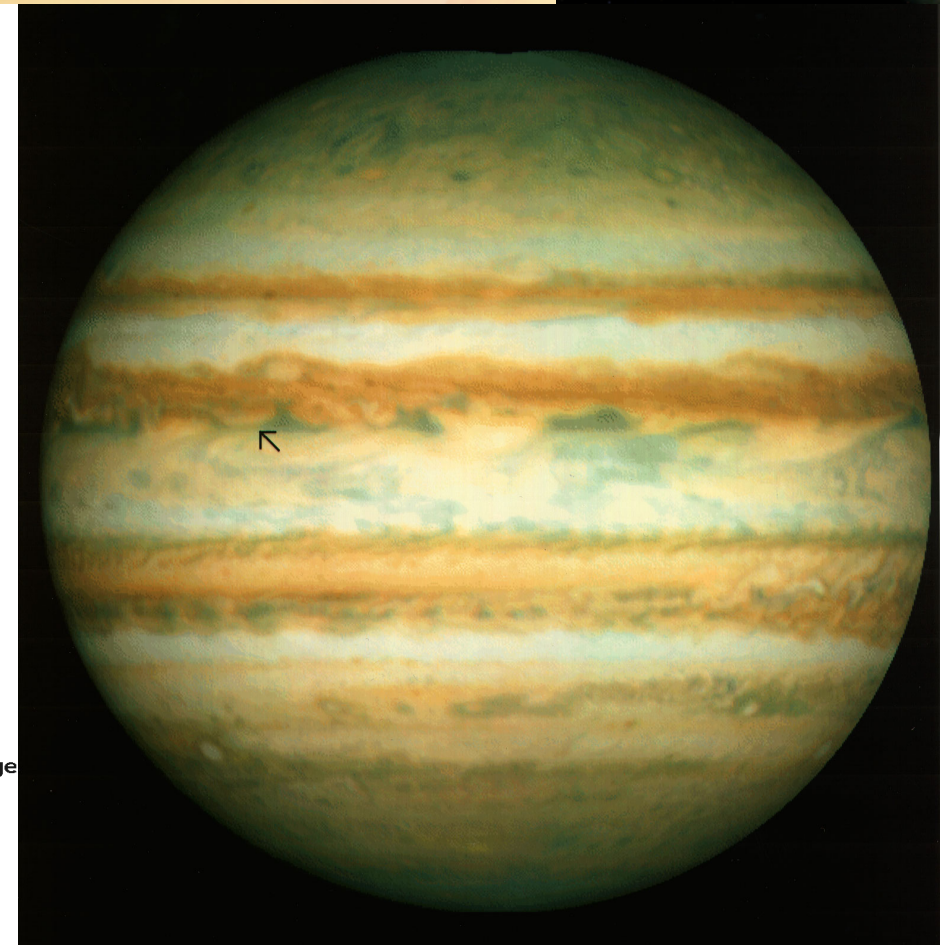
- 4 orders of magnitude pressure differential on track from above clouds to surface
- trace species measured to parts per billion
- 9 orders of magnitude difference between atmospheric pressure at surface and ion source pressure in mass spectrometer
- 500 degree temperature gradient from atmosphere above clouds to surface
- cloud droplets and aerosols that can clog mass spectrometer inlet systems and mask real vertical variations due to their condensation on surfaces
- a fast ride to the surface with an entry probe

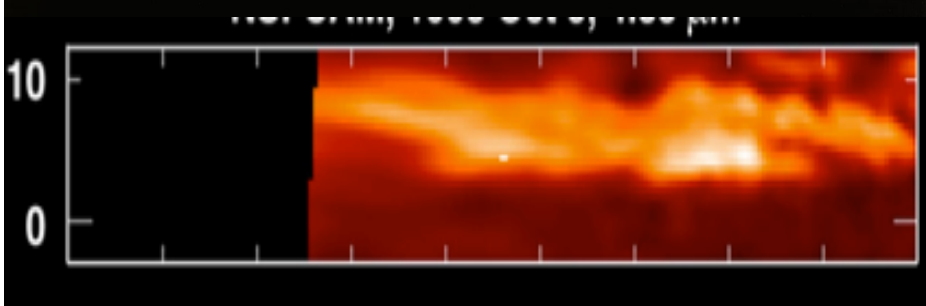


Galileo Probe Neutral Mass Spectrometer

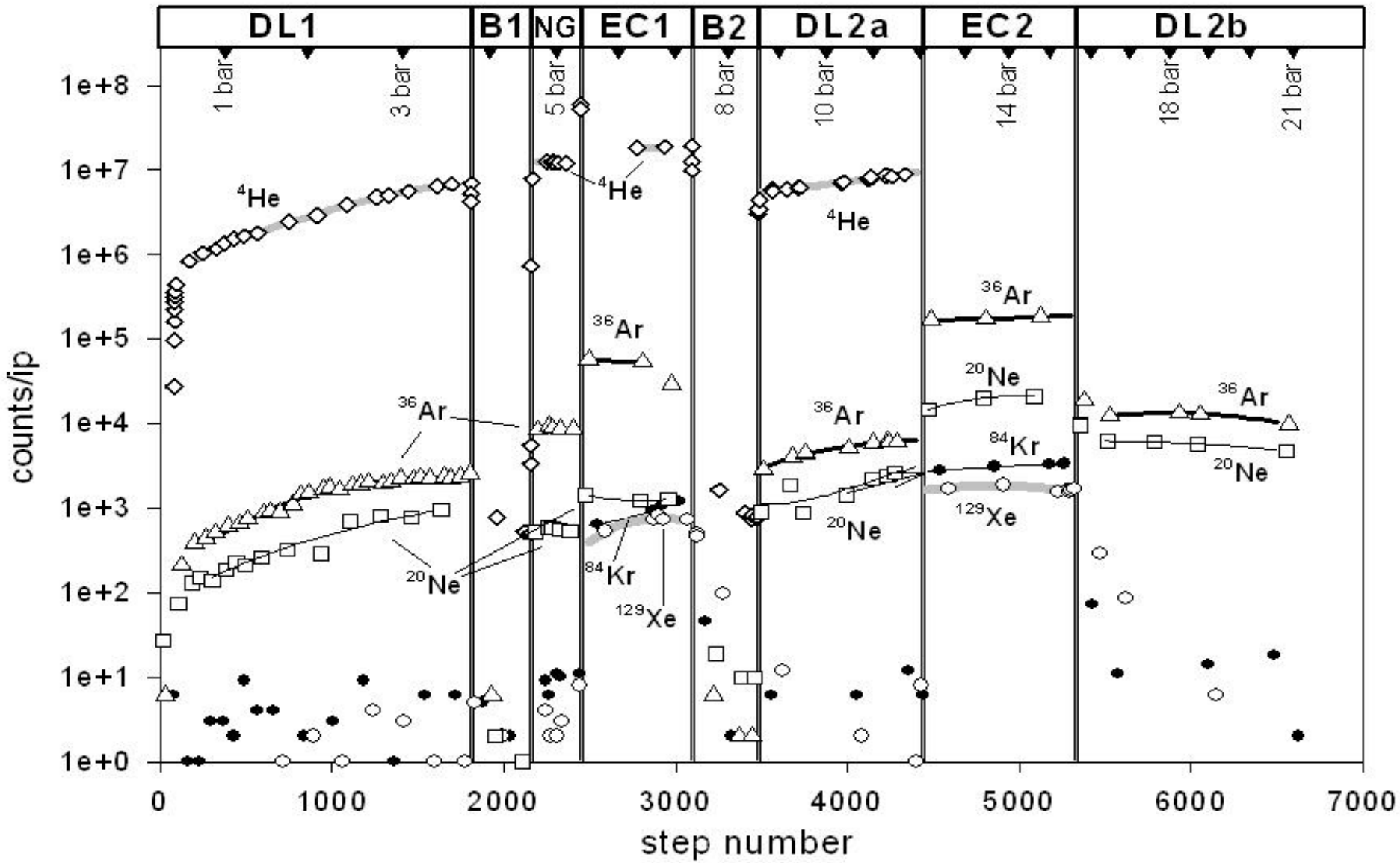






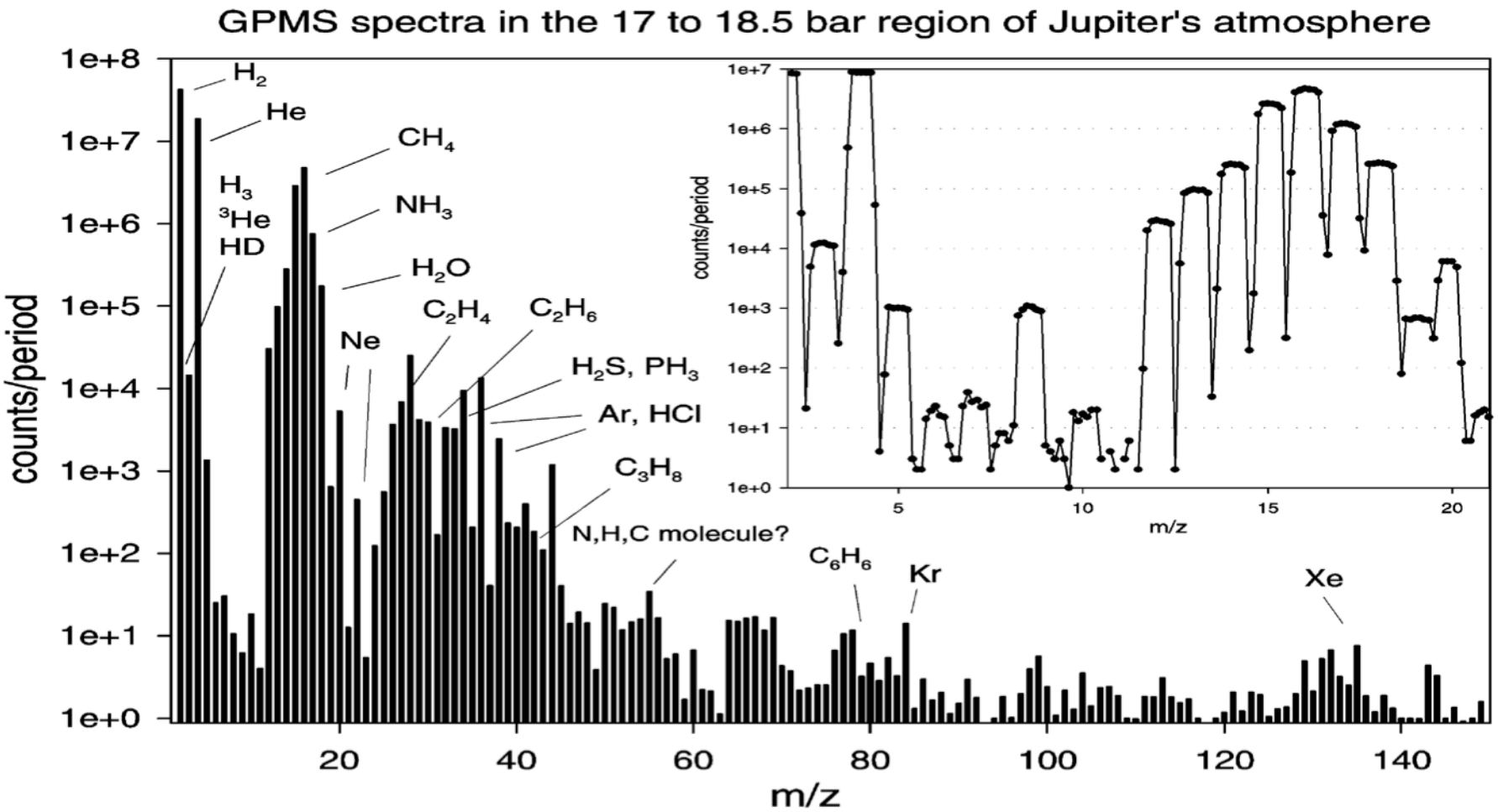


GPMS – each m/z tracked during ~ 1 hour descent to 21 bar



GPMS spectrum from deep in Jupiter's atmosphere

GPMS @ Jupiter



Features of the quadrupole mass spectrometer

- Dynamic range
- Minimal crosstalk between unit mass channels

Multiple elemental and isotope ratios measured by the GPMS

Elements	Sun (protosolar)	Jupiter/Sun	Saturn/Sun
He/H	0.09705	0.807±0.02	0.56–0.85 (2)
Ne/H	2.10×10^{-4} (3)	0.059±0.004	?
Ar/H	1.70×10^{-6}	2.2±0.5	?
Kr/H	2.14×10^{-9}	2.03±0.38	?
Xe/H	2.10×10^{-10}	2.11±0.40	?
C/H	2.75×10^{-4}	3.82±0.66	9.3±1.8 (CIRS)
N/H	6.76×10^{-5}	4.90±1.87	2.6–5
O/H	5.13×10^{-4}	0.48±0.17 (hotspot)	?
S/H	1.55×10^{-5}	2.88±0.69	?
P/H	2.57×10^{-7}	1.21	5–10

Isotopes	Sun	Jupiter	Saturn
D/H	$2.1\pm0.5\times10^{-5}$	$2.6\pm0.7\times10^{-5}$	$2.25\pm0.35\times10^{-5}$
$^{3}He/^{4}He$	$1.5\pm0.3\times10^{-4}$	$1.66\pm0.05\times10^{-4}$	
$^{15}N/^{14}N$	$\leq2.8\times10^{-3}$	$2.3\pm0.3\times10^{-3}$	

Table extracted from Atreya, S.K., "Saturn Probes: Why, Where, How?" Proc. 4th Int. Planetary Probe Workshop, NASA JPL Document, 2007 with Ar correction from table at right.

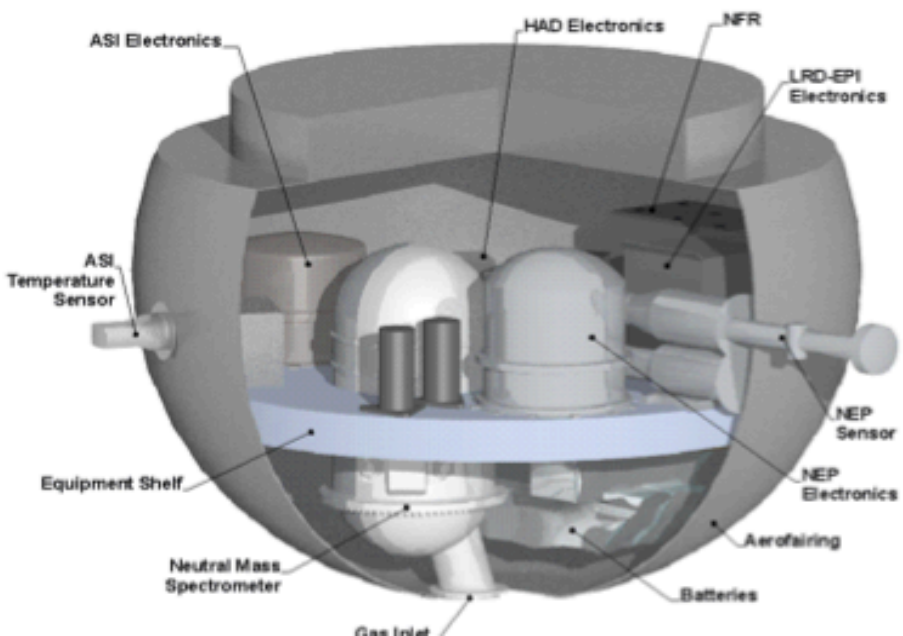
(2) Conrath et al., Icarus, 144, 124 (2000).

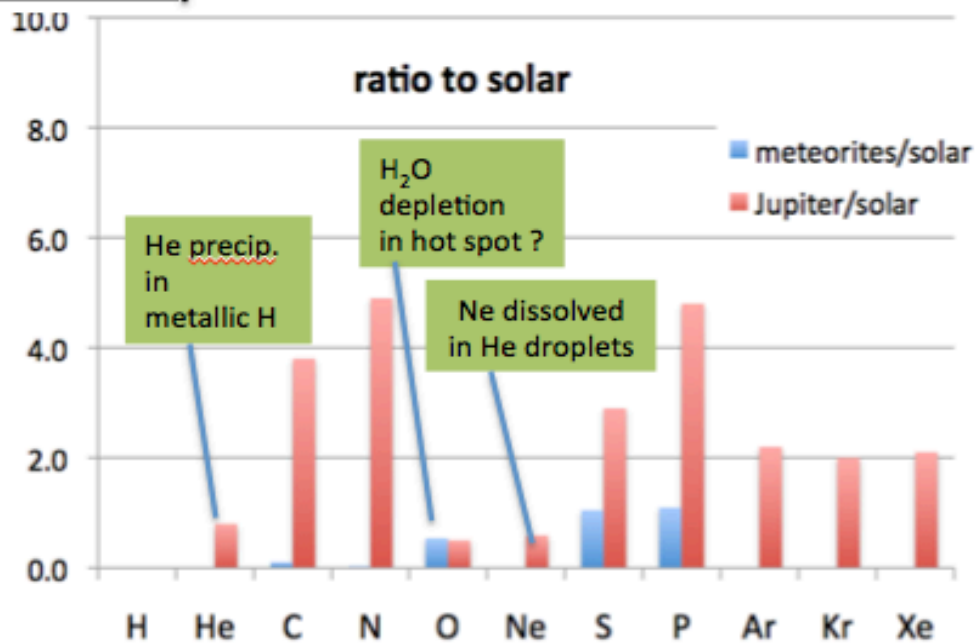
(3) Drake & Testa, Nature 436, 1038 (2005).

Jovian phosphine - Fletcher, L.N., G. Orton, N. Teanby, and P. Irwin, "Phosphine on Jupiter & Saturn from Cassini/CIRS," Icarus 202 (2), 543–564, 2009.

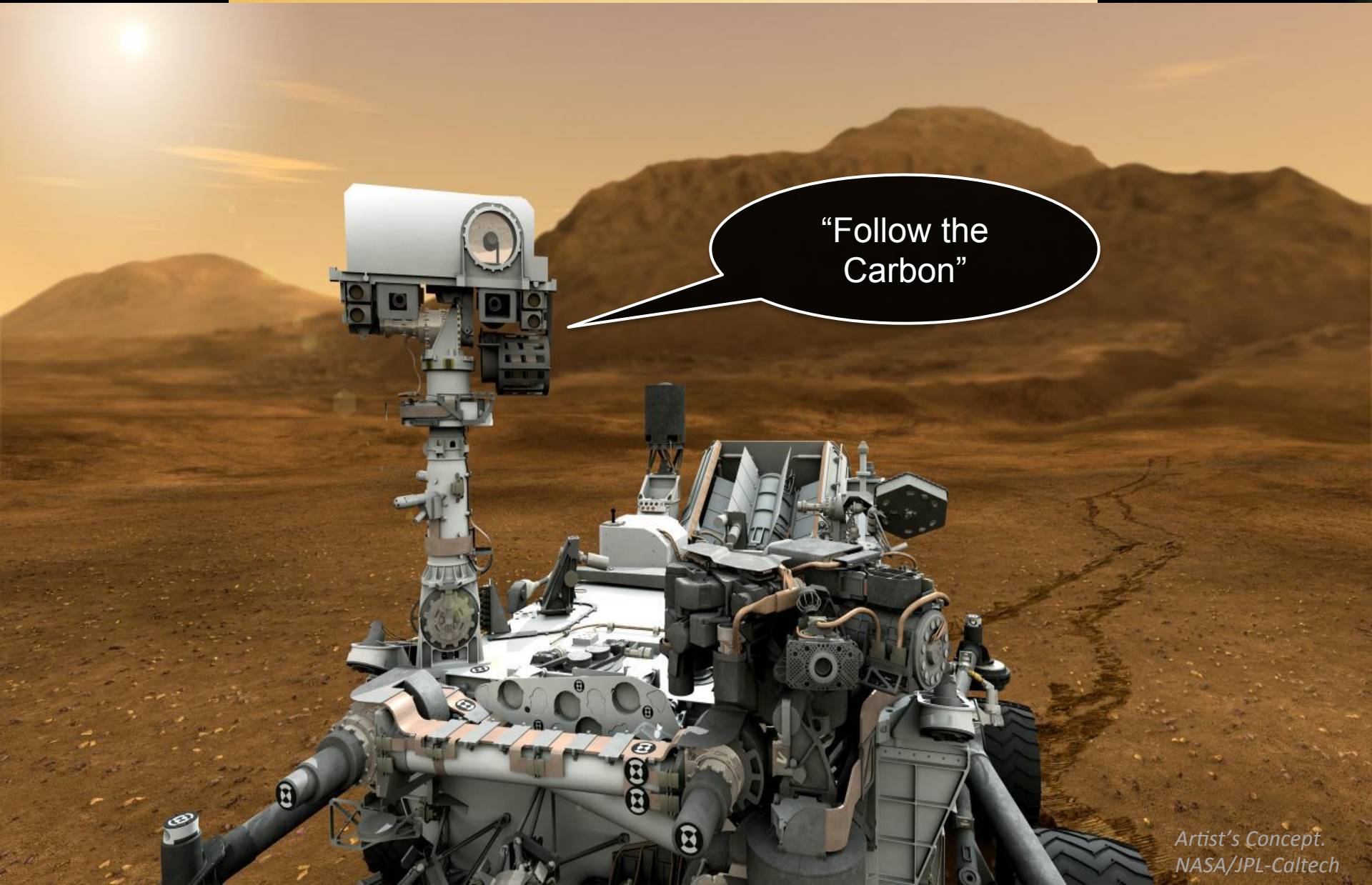
	New Solar	Old Solar
H	1.00E+00	1.00E+00
He	8.51E-02	9.75E-02
C	2.45E-04	3.62E-04
N	6.03E-05	1.12E-04
O	4.57E-04	8.53E-04
Ne	6.92E-05	1.23E-04
S	1.38E-05	1.85E-05
P	2.29E-07	3.73E-07
Ar	1.51E-06	3.62E-06
Kr	1.78E-09	1.61E-09
Xe	1.74E-10	1.68E-10

New Solar Values: Grevesse, N., et al., Space Sci. Rev. 130 (1), 105–114, 2007.





Curiosity continues the assessment of Mars Habitabili
Beyond signs of water, the rover will look for signs of organics, the chemical building blocks of life



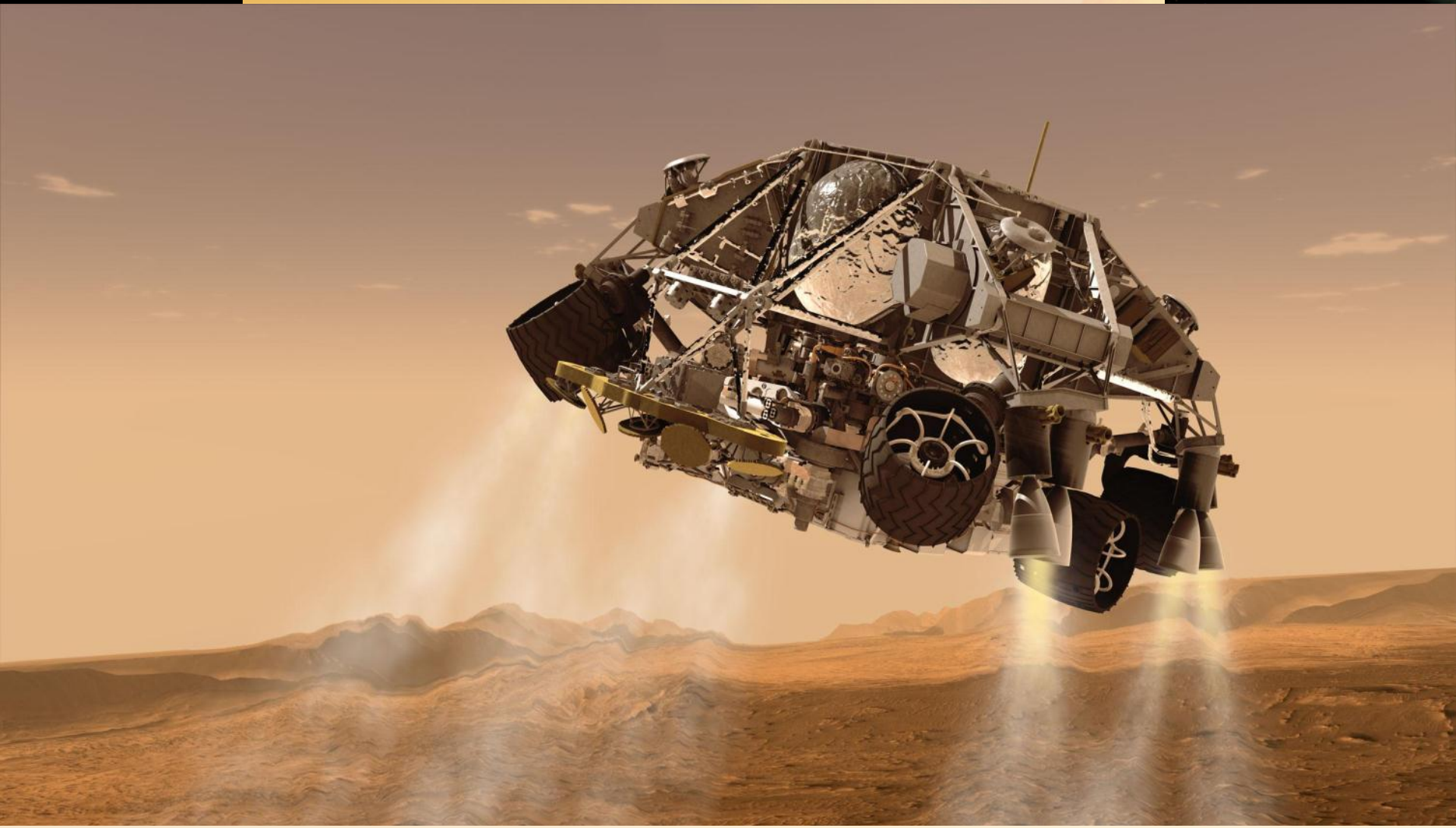
Artist's Concept. NASA/JPL-Caltech

While slowing down using the parachute, the heat shield is popped off, exposing the rover to the Martian atmosphere.

The rover's descent camera begins taking a movie of the remaining five-mile flight to the ground.

Artist's Concept. NASA/JPL-Caltech

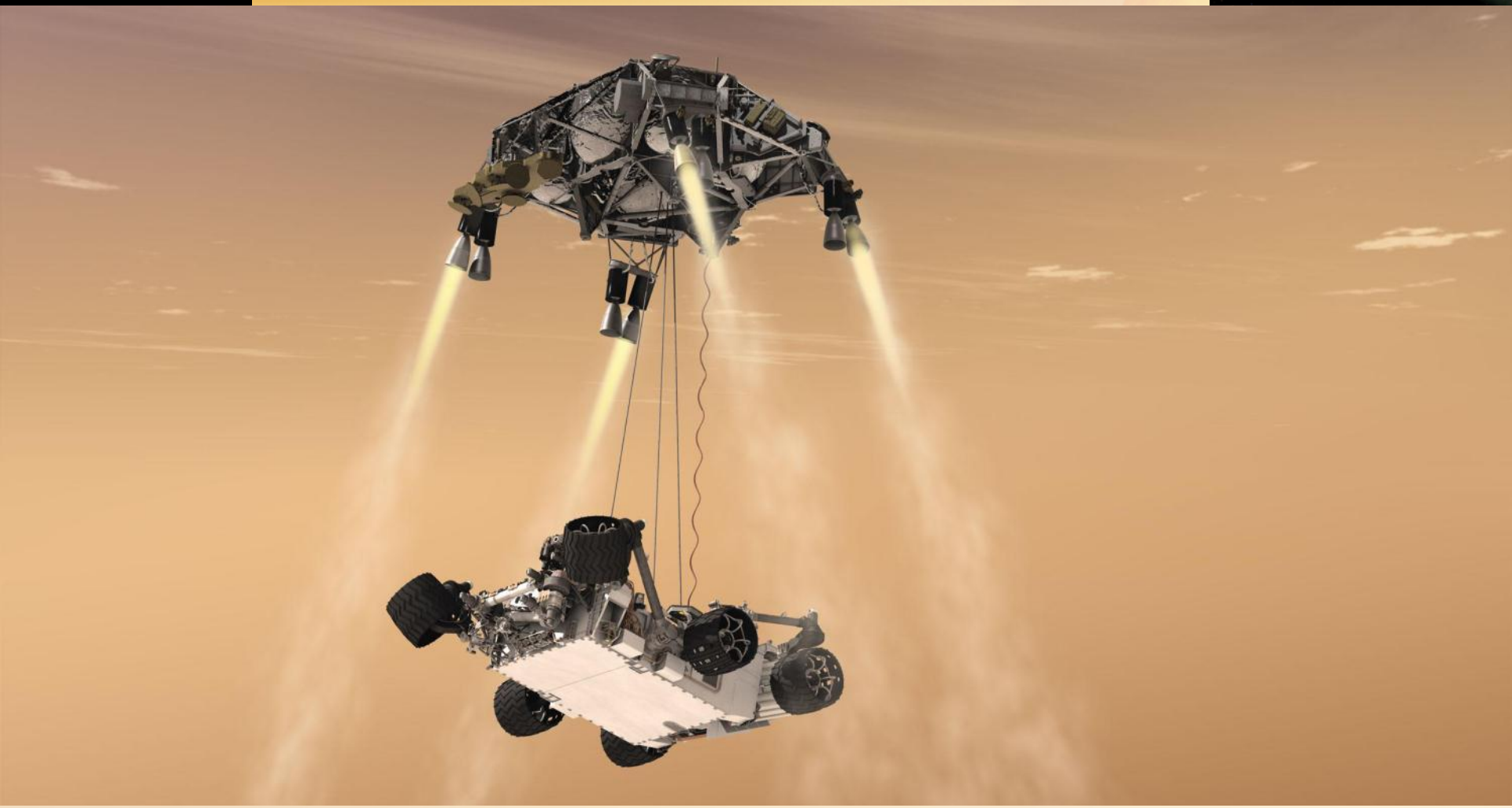
The engines on the descent stage roar to life and fly the rover down the last mile to the surfac



As it descends, the rover uses radar to measure its speed and altitude, which it uses to land safely.

Artist's Concept. NASA/JPL-Caltech

The hovering descent stage lowers the rover on three nylon ropes called bridle.



Coiled electronics and communications cables also unspool from the descent stage. This configuration is known as the "Sky Crane."

Artist's Concept. NASA/JPL-Caltech

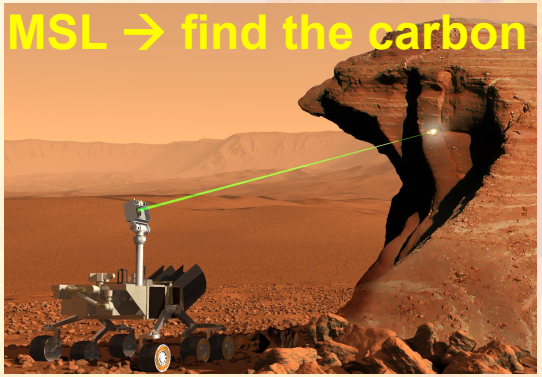
When the sky crane "senses" that Curiosity has touched down, the cables are cut.

The sky crane flies a safe distance away from the rover before crash-landing.

Artist's Concept. NASA/JPL-Caltech

Curiosity being tested in the JPL clean room

MSL mission goals

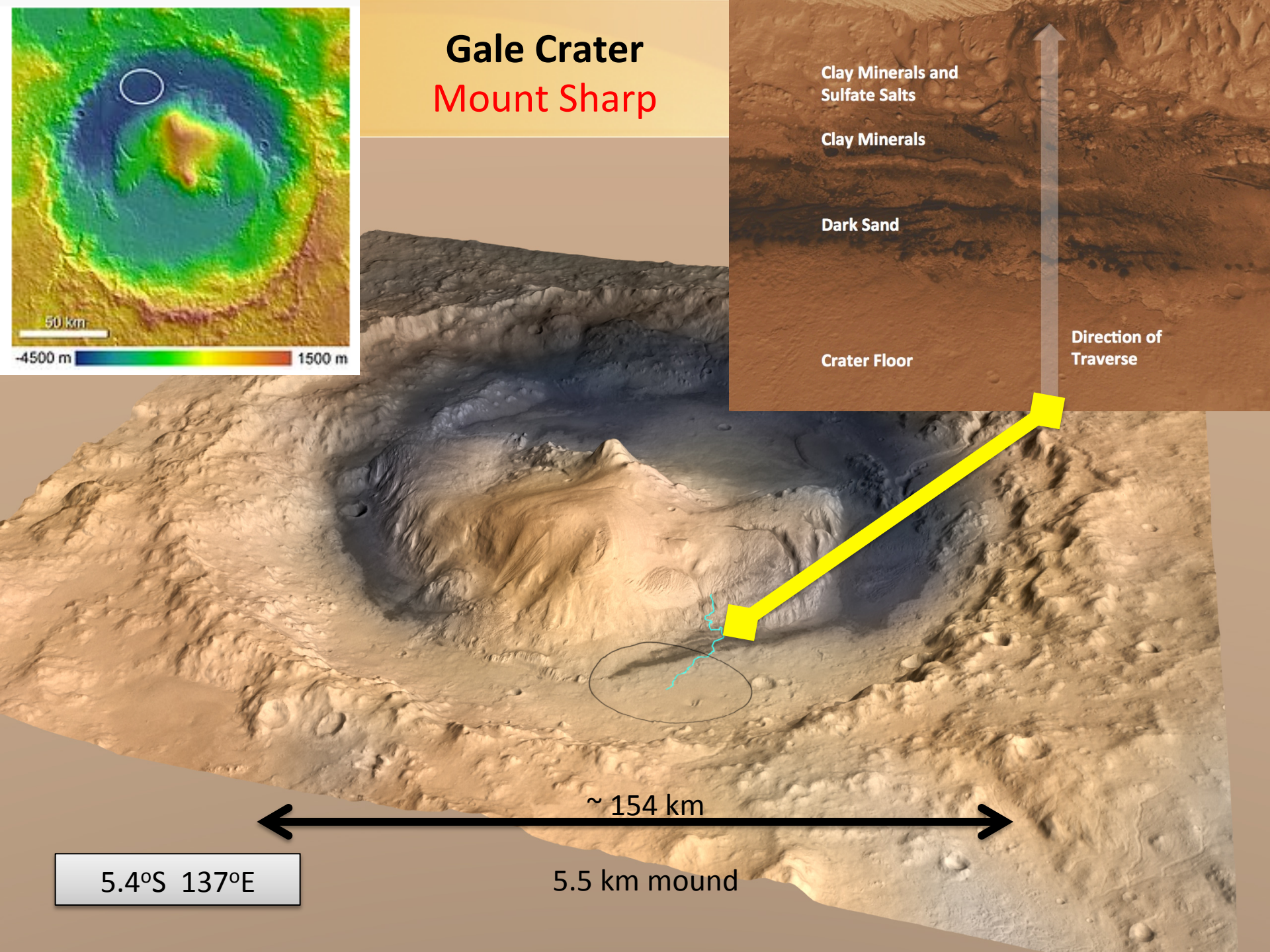


Curiosity's primary scientific goal is to explore and quantitatively assess a local region on Mars' surface as a potential habitat for life, past or present

Objectives include:

- Assessing the biological potential of the site by investigating any organic and inorganic compounds and the processes that might preserve them
- Characterizing geology and geochemistry, including chemical, mineralogical, and isotopic composition, and geological processes
- Investigating the role of water, atmospheric evolution, and modern weather/ climate
- Characterizing the spectrum of surface radiation

Gale Crater
Mount Sharp

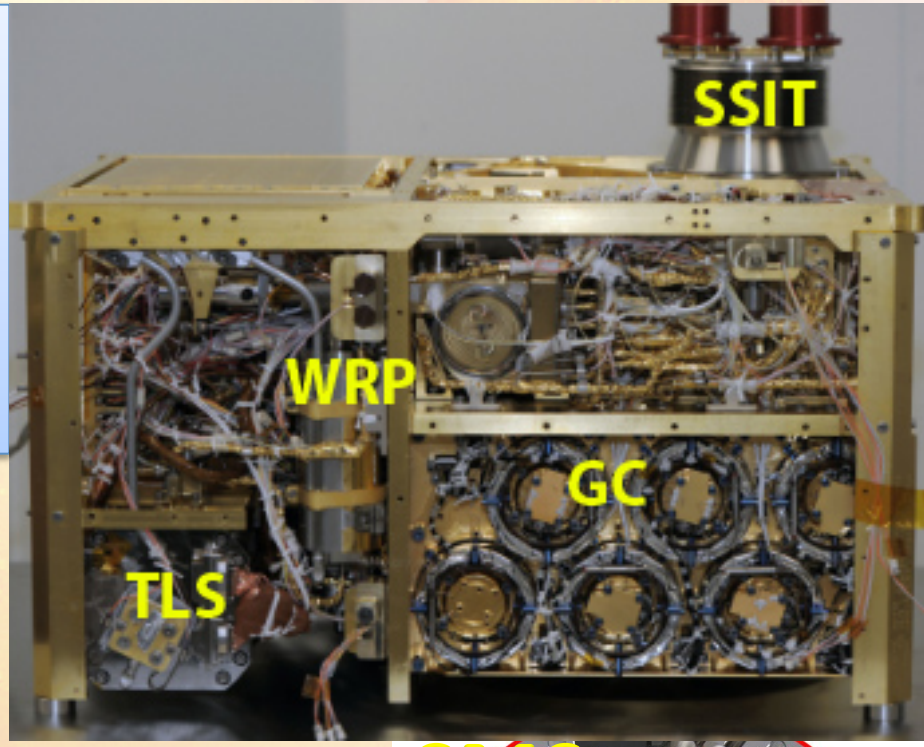


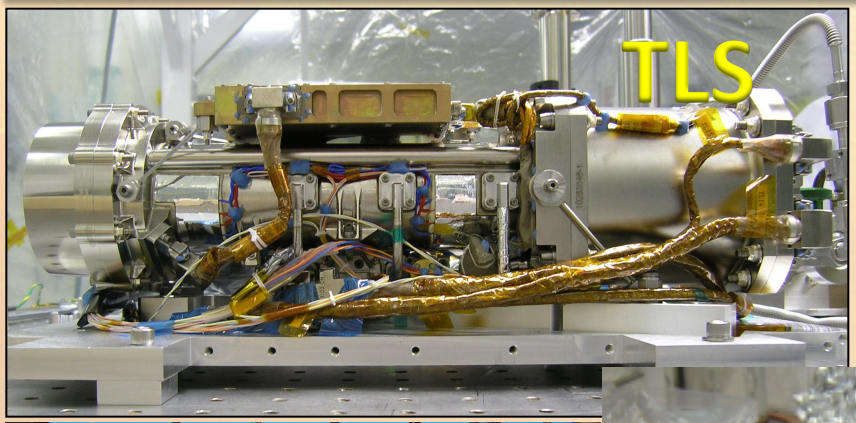
5.4°S 137°E

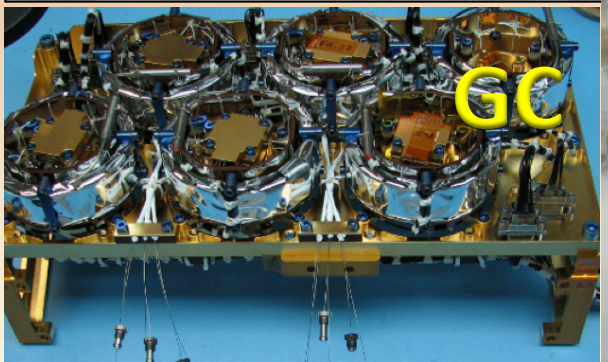
Sample Analysis at Mars Instrument Suite – including GCMS

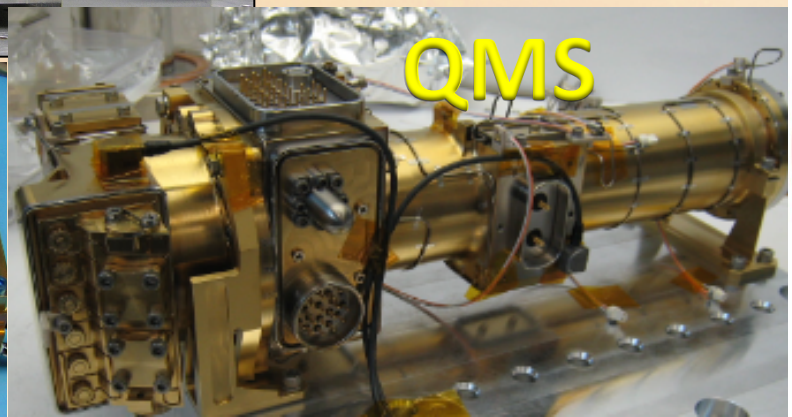
SAM suite instruments and major subsystems

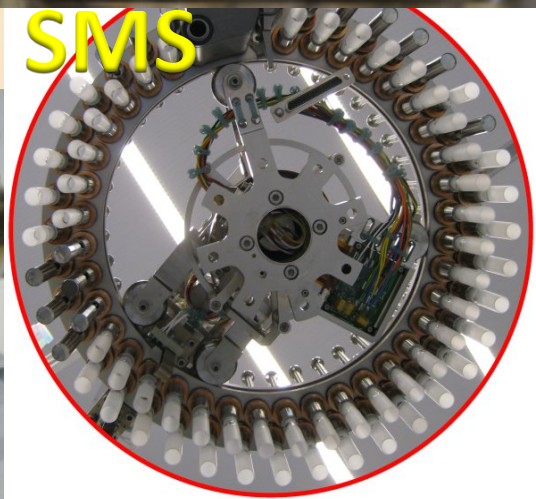
- Quadrupole Mass Spectrometer
- 6-column Gas Chromatograph
- 2-channel Tunable Laser Spectrometer
- Gas Processing System
- Sample Manipulation System











SAM Science Goal	SAM Measurement

PAST OR PRESENT HABITABILITY OF MARS

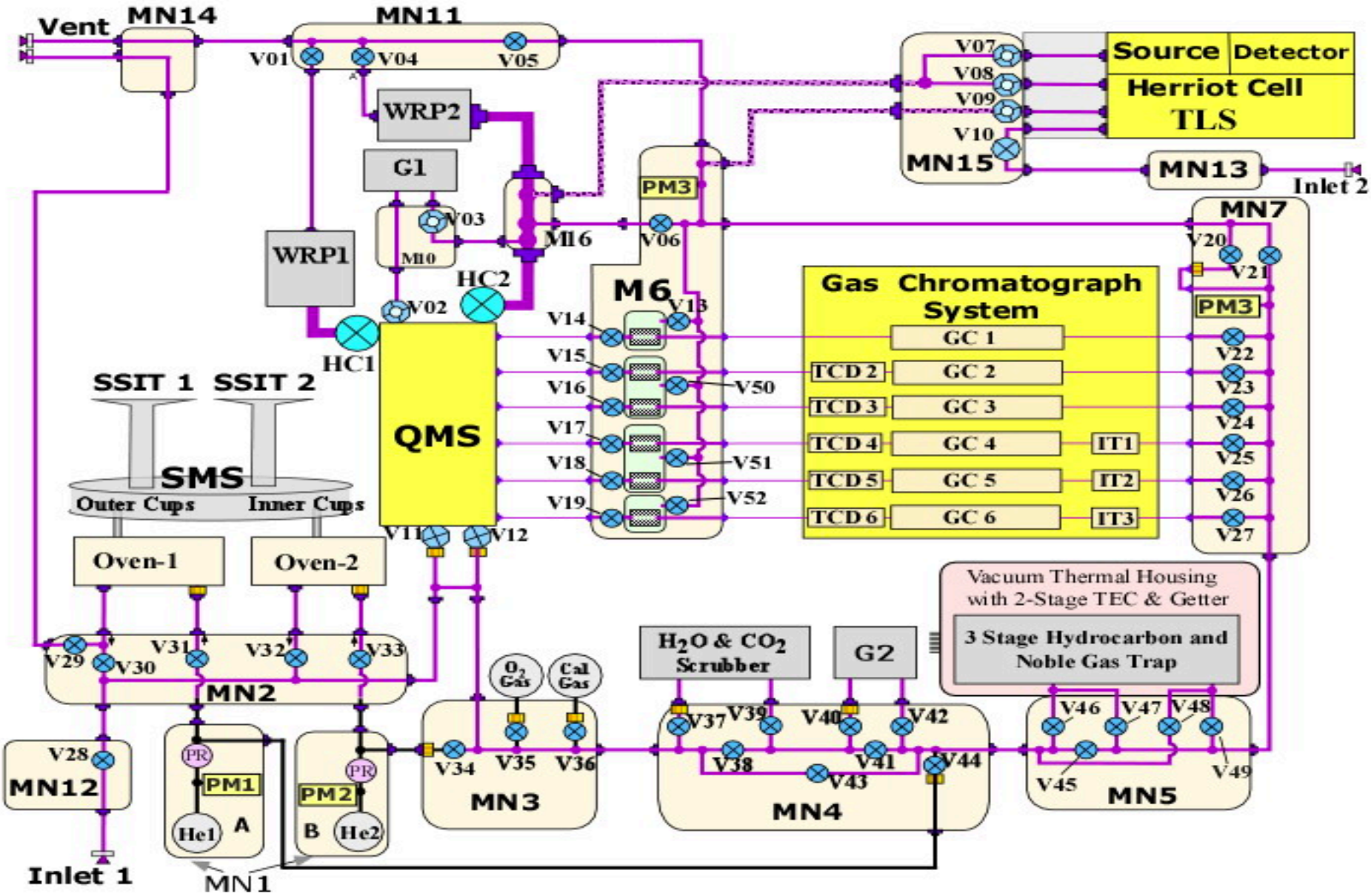
assessment includes

INVENTORY OF ORGANIC COMPOUNDS

CHEMICAL, ISOTOPIC, & MINERALOGICAL COMPOSITION

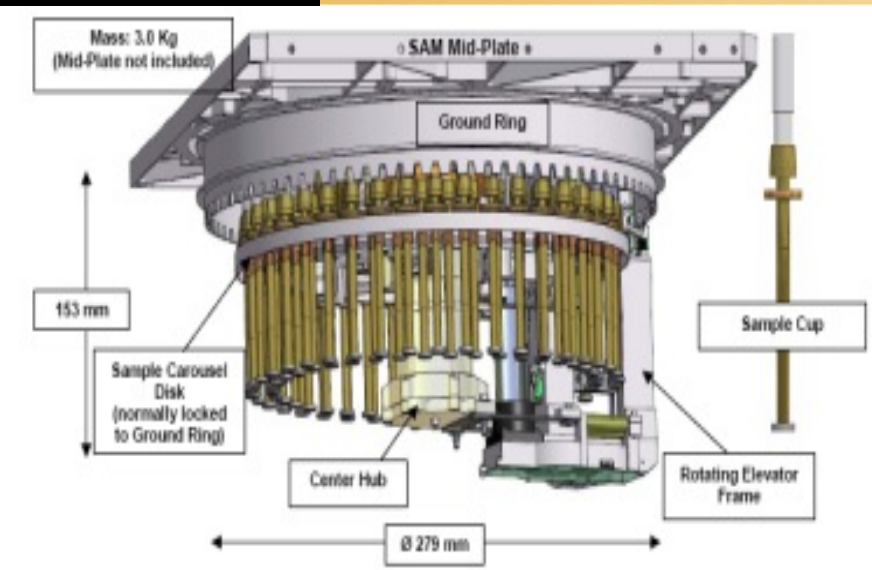
- Sources & destruction paths for organics
- Organic compound inventory
- Chemical state of light elements
- Evolved gas analysis (CO_2, SO_2, H_2O etc.)
- Atmospheric evolution
- Noble gas abundance & isotopes
- Trace species & atmos/surf ace interactions
- CO_2, CH_4 and H_2O variability & isotopes
- Organic compounds of biotic relevance
- Targeted organics (amino acids etc.)

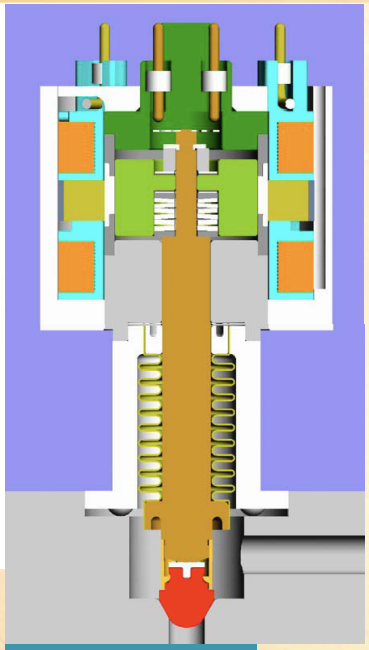
Gas flow diagram - simplified



SAM INSTRUMENT SUITE OVERVIEW

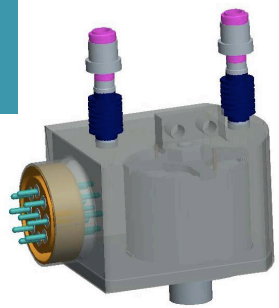
Gas and solid sample manipulation

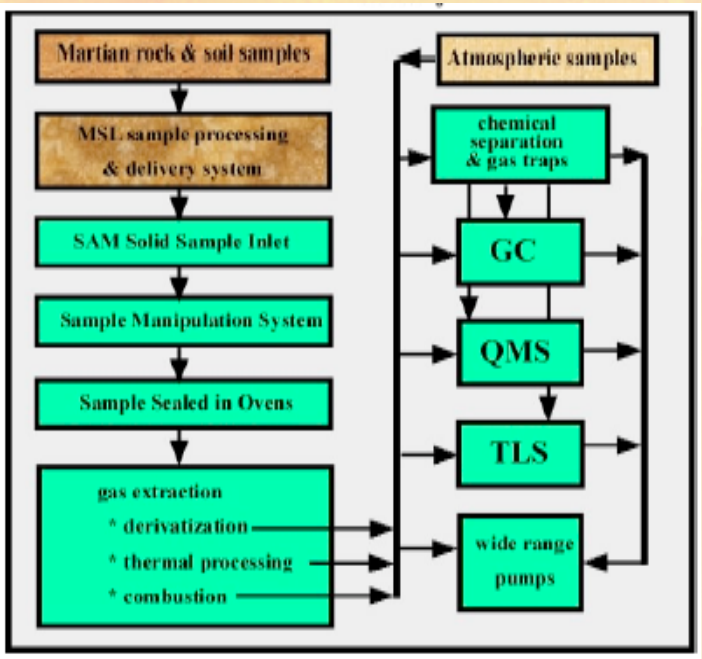




microvalve

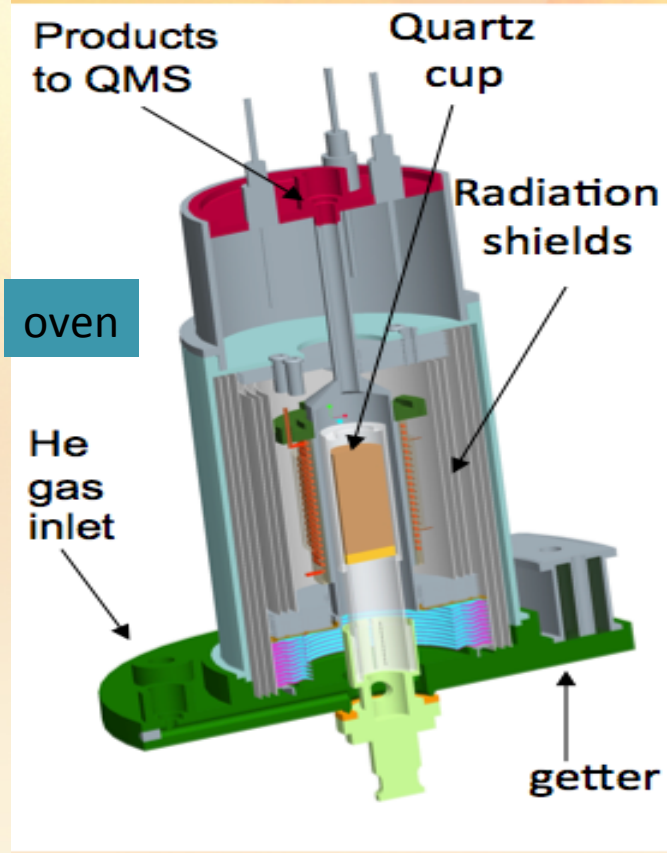
chemical getter



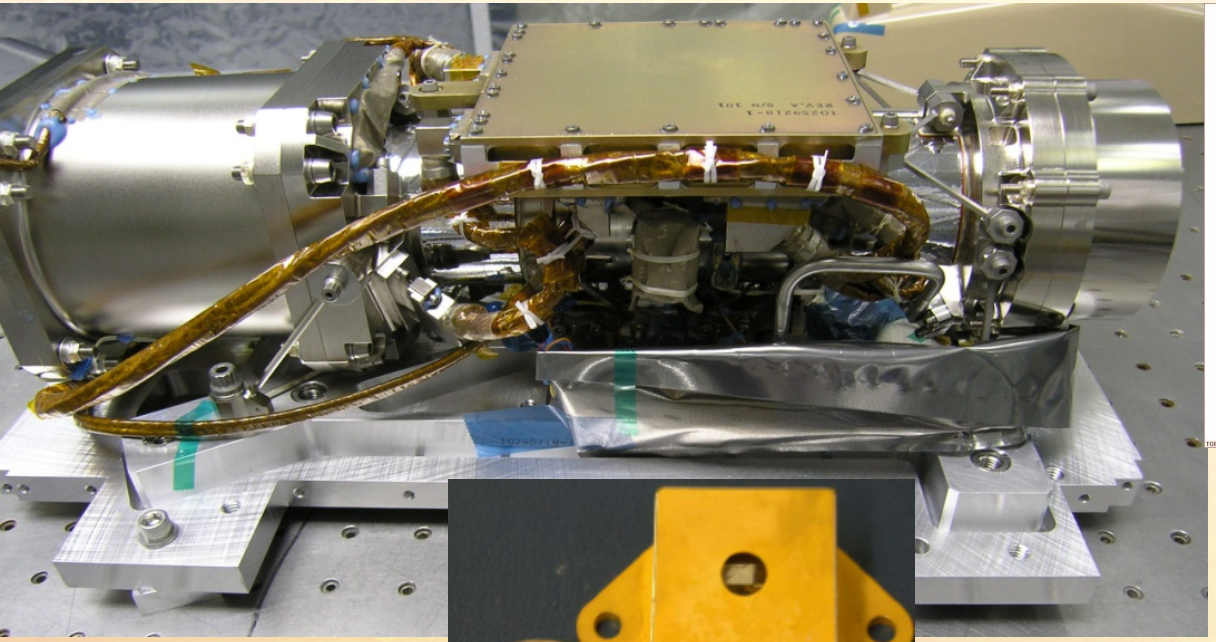


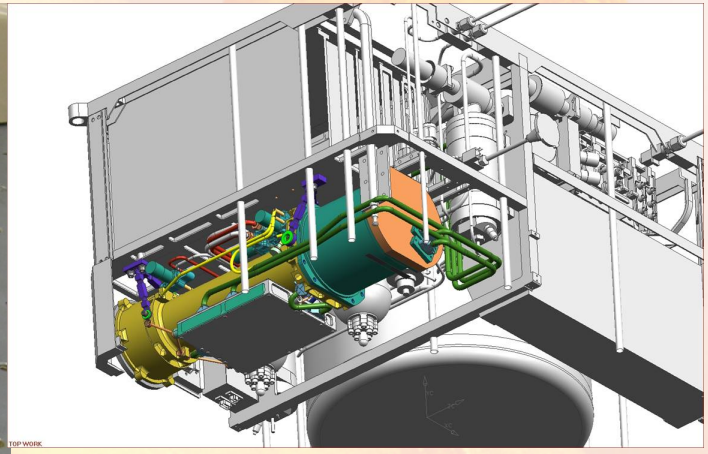
Sample manipulation

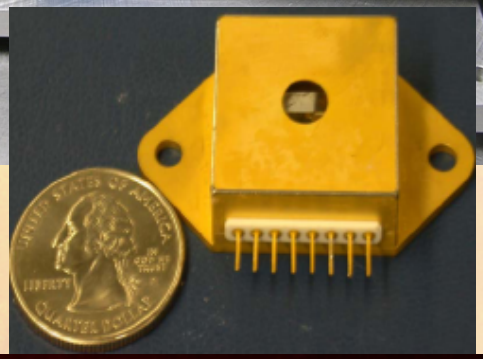
oven

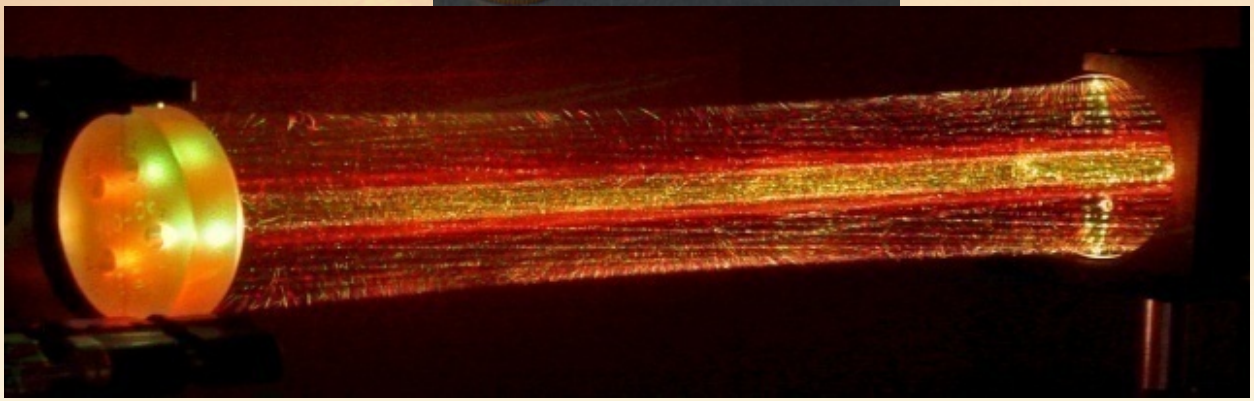


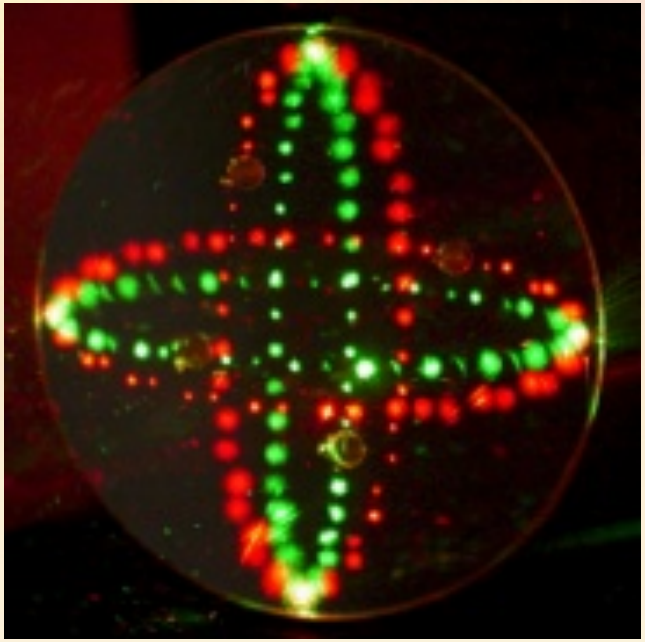
The SAM Tunable Laser Spectrometer developed by Webster/JPL





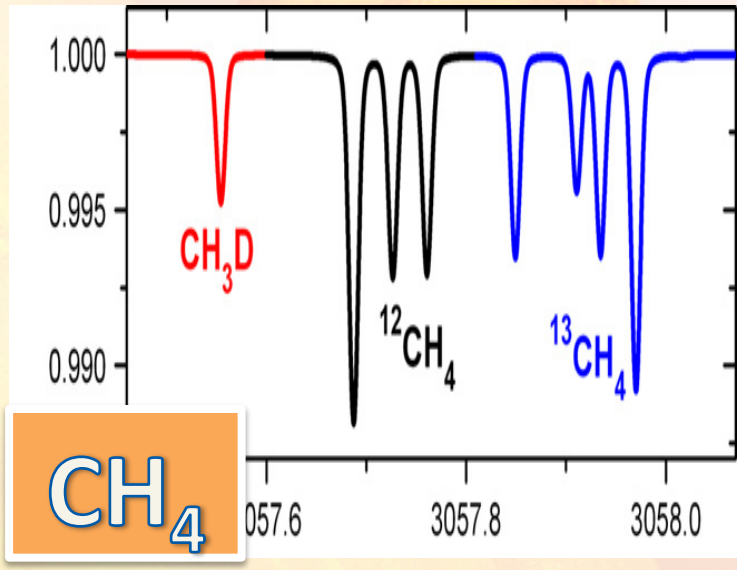


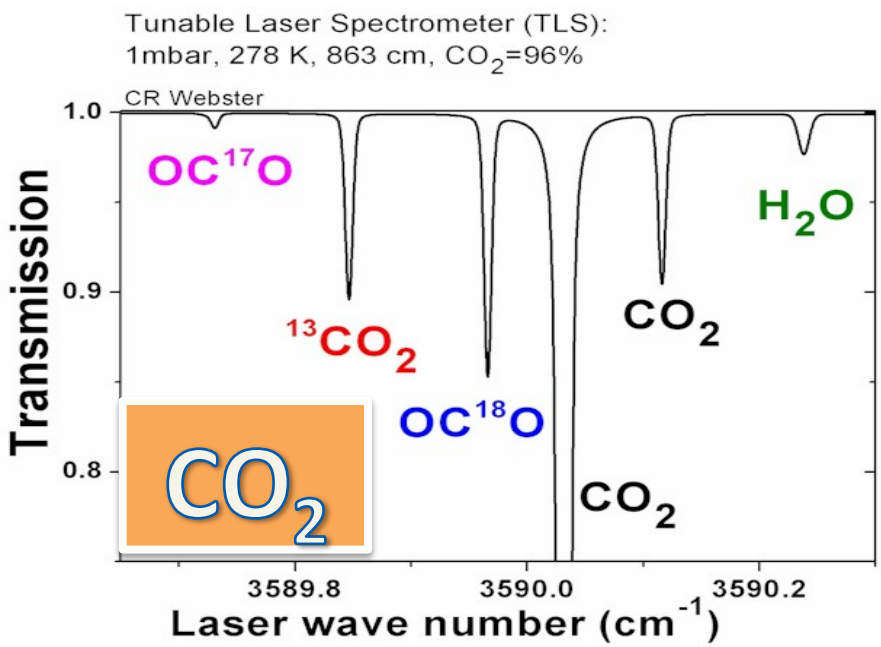


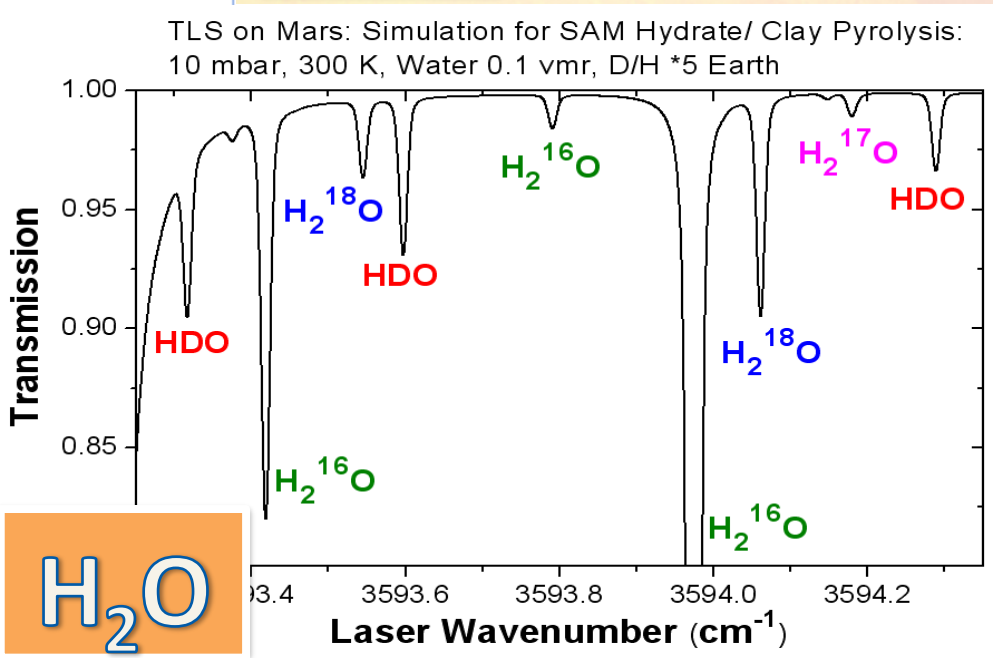


The SAM Tunable Laser Spectrometer line selections

TLS measurement capability			
Channel	**Wavelength**	**Scan name**	**15 minute predicted capability**
1- IC laser	3.3 μm	Methane	to 0.3 ppbv δ ^{13}C to 2 per mil
2-Near IR laser	2.785 μm	Carbon dioxide	to 0.2 ppmv in CO_2 and H_2O δ ^{13}C to 2 per mil δ ^{18}O to 3 per mil δ ^{17}O to 5 per mil
	2.783 μm	Water	H2O to 0.1 ppmv δD to 2 per mil δ ^{18}O to 3 per mil δ ^{17}O to 5 per mil



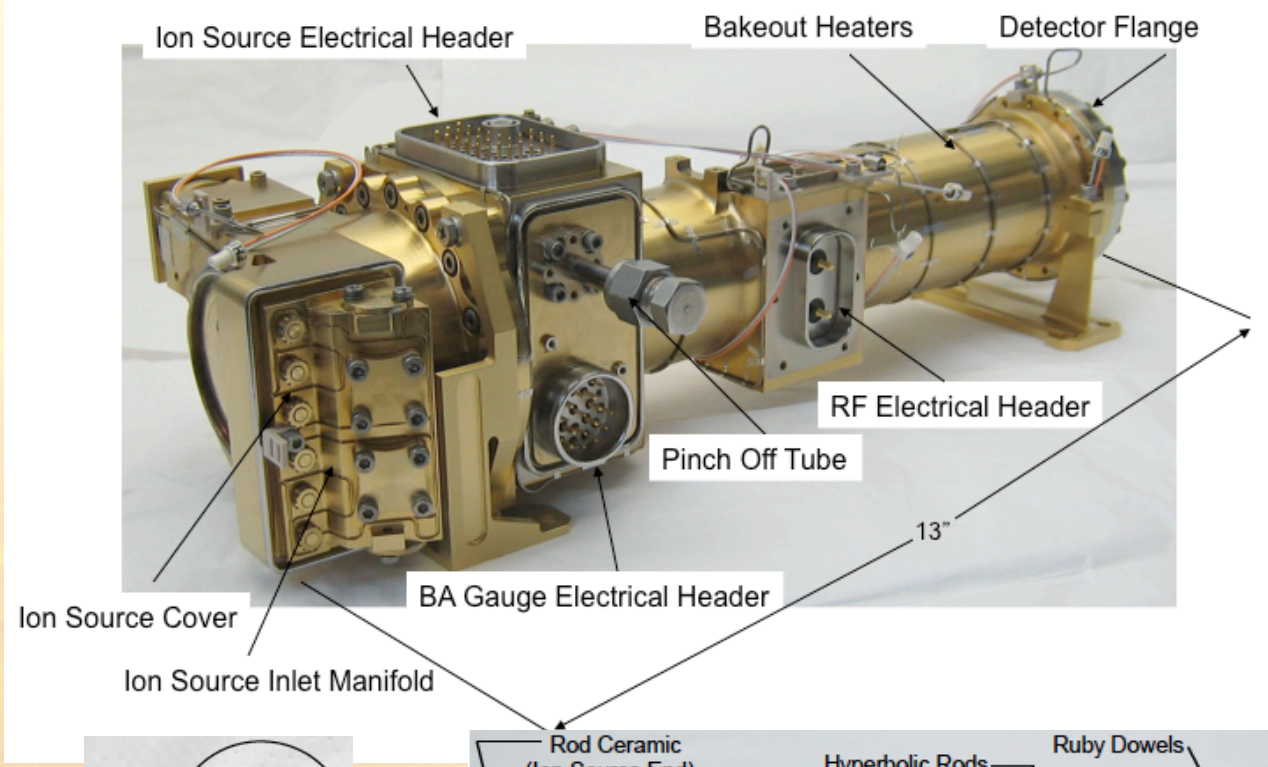


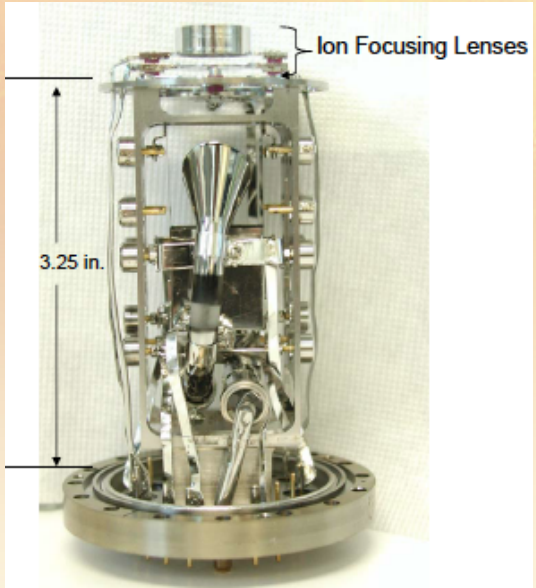


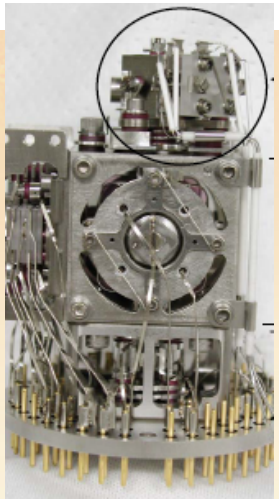
SAM Quadrupole Mass Spectrometer (QMS)

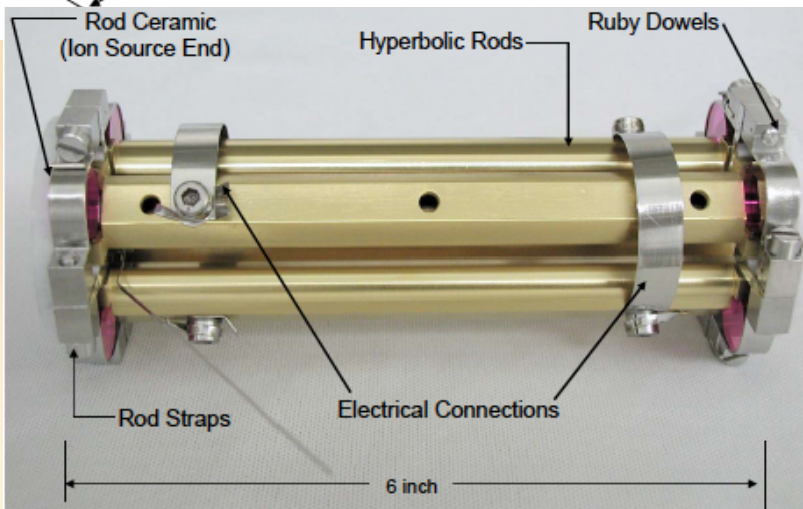
QMS characteristics

- precision assembly hyperbolic rods
- titanium alloy housing
- in situ bakeout
- 3 frequency RF
- pressurized enclosure for RF electronics
- 2-535 Da
- dual electron guns
- dual detectors
- 2 direct atmosphere inlets
- 6 gas chromatograph inlets









SAM 6 column gas chromatograph

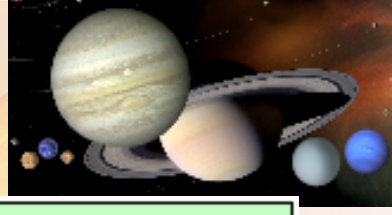
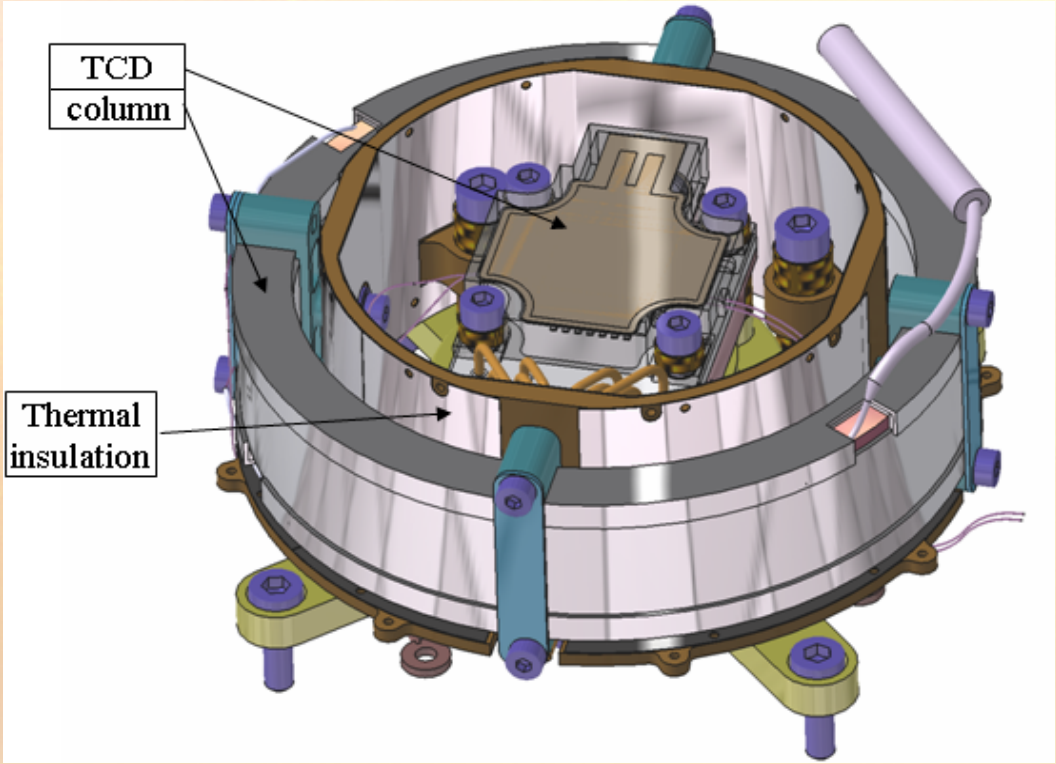
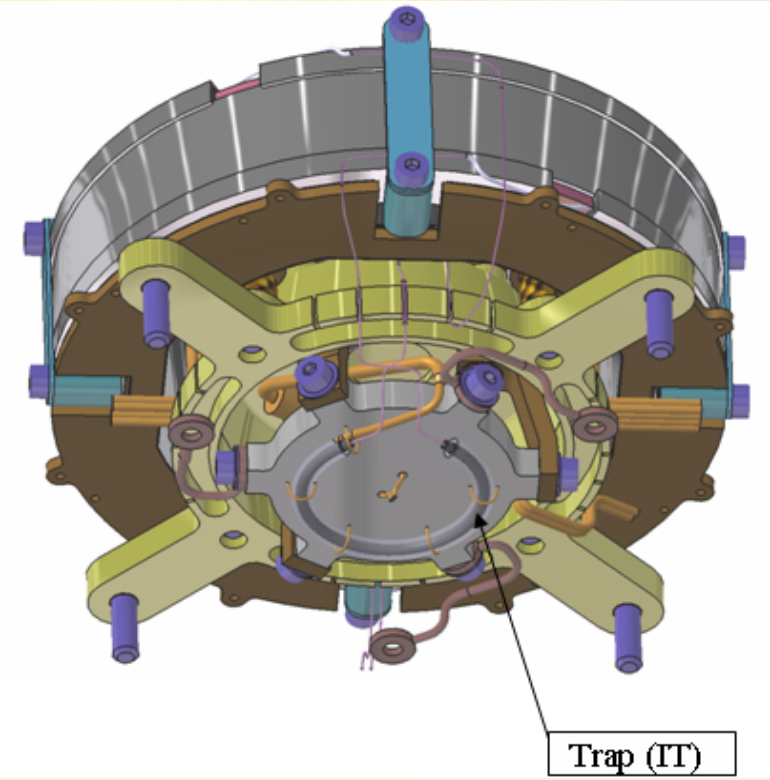


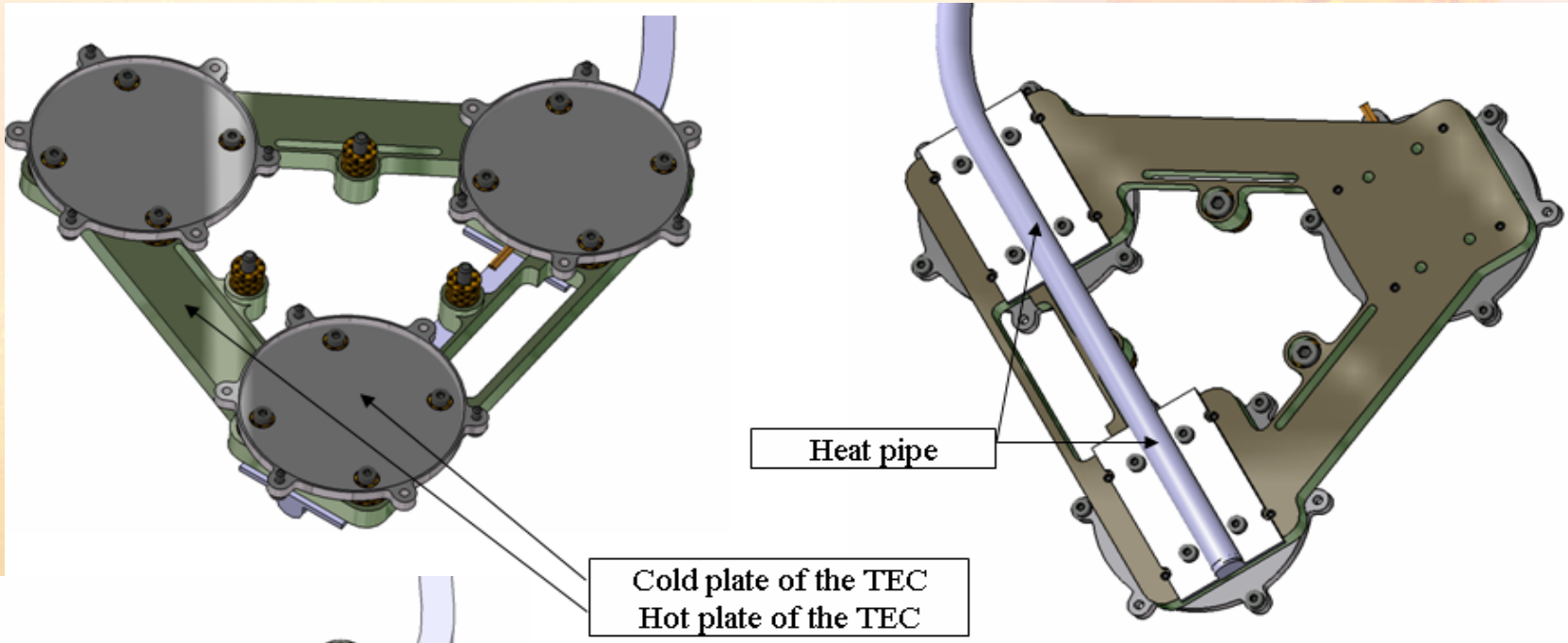
Table IX. Gas chromatograph columns		
Column	**Stationary Phase**	**Species targeted**
GC1–MXT 20 WCOT	polydimethylsiloxane with 20% of phenyl	Medium molecular weight organics (C5-C15 organics)
GC2-MTX 5 (WCOT)	polydimethylsiloxane with 5% of phenyl	High molecular weight VOCs including >C15 chemical derivatives
GC3-Carbobond (PLOT)	Carbon molecular sieve	Permanent gases and C1-C2 HCs
GC4-Chirasil-β Dex CB	β cyclodextrin	Enantiomers of VOCs
GC5-MXT CLP (WCOT)	polydimethylsiloxane with phenyl and cyanopropyle	Medium molecular weight organics (C5-C15 organics)
GC6-MXT Q (PLOT)	Divinylbenzene or substituted divinylbenzene	C1-C4 VOCs NH_3, S containing compounds
PLOT = porous layer open tubes; **WCOT** = wall coated open tublar **VOC** = volatile organic compounds		

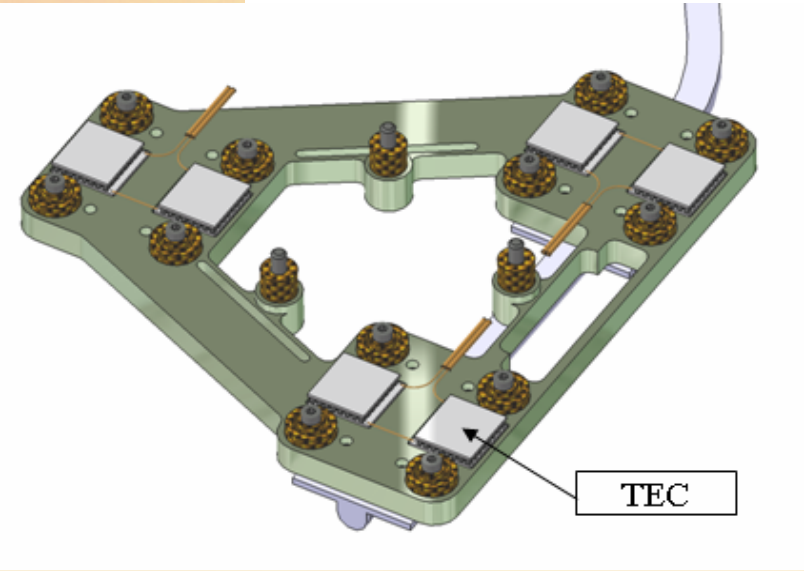
SAM 6 column gas chromatograph





SAM 6 column gas chromatograph

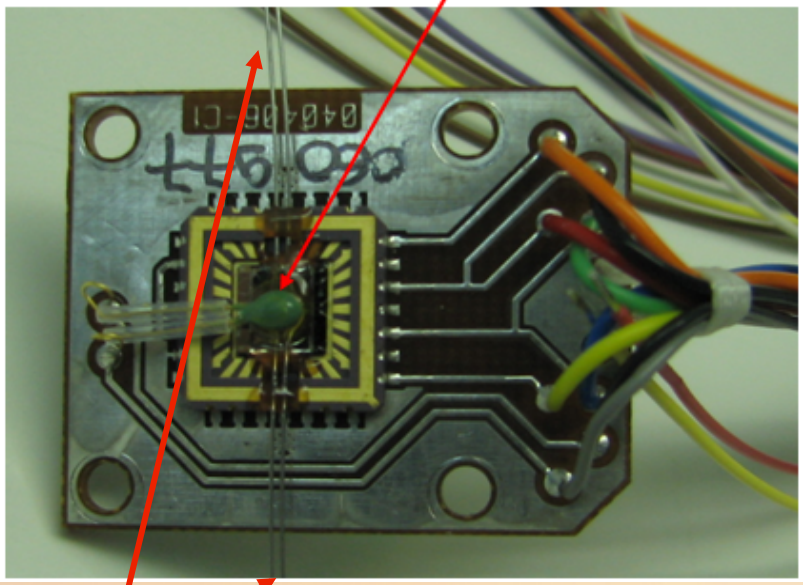


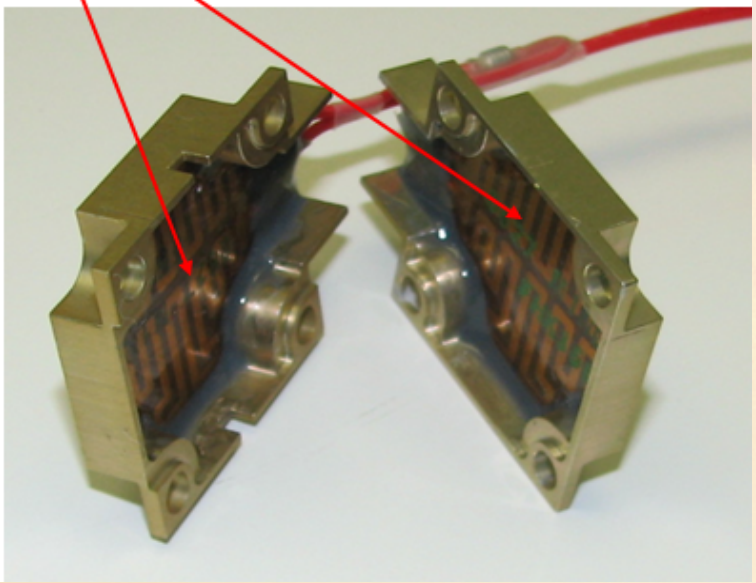


Injection trap and heat pipe

SAM 6 column gas chromatograph

Integration of the YSI thermistor and Minco heaters on the Aluminium covers.



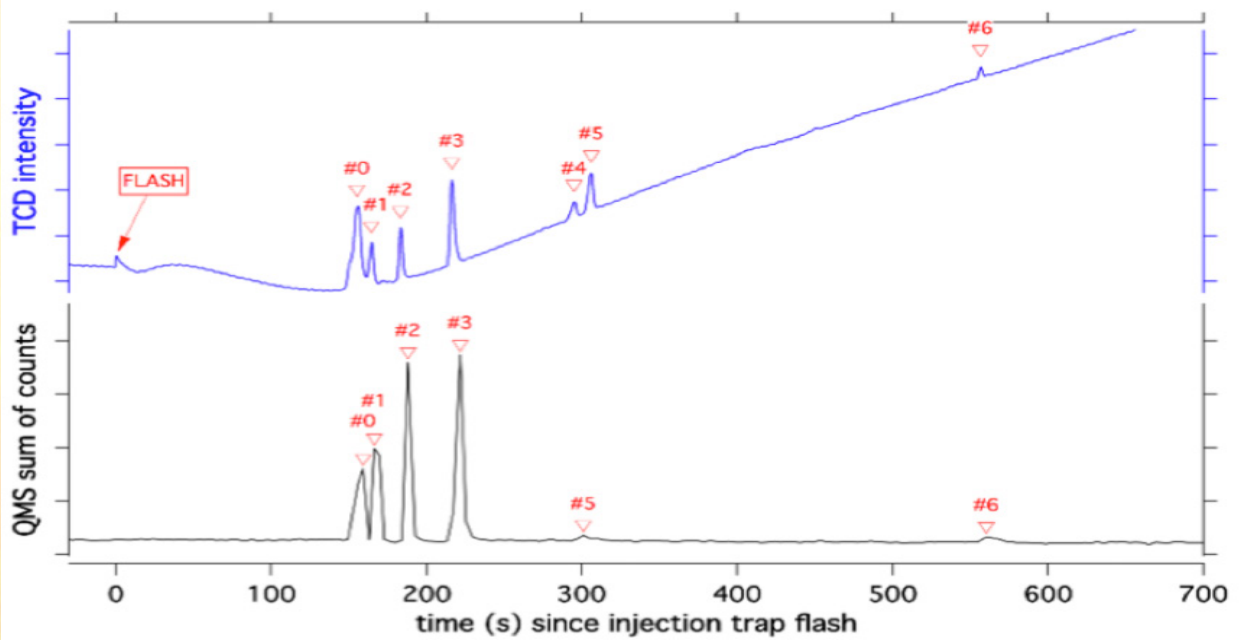


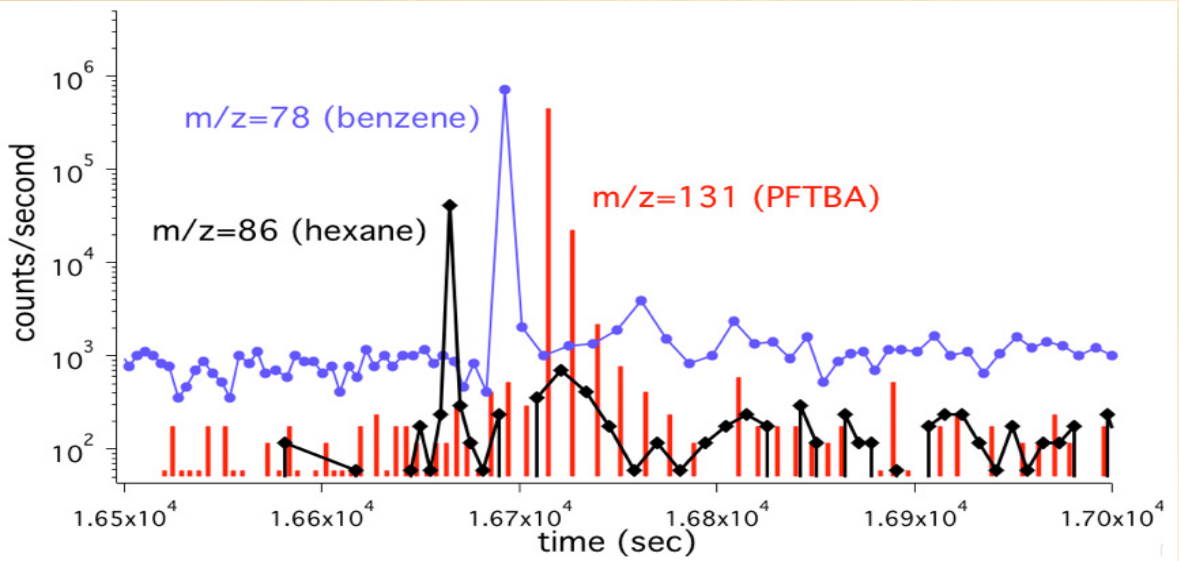
Inlet and outlet Capillaries

Thermal conductivity detectors

SAM 6 column gas chromatograph

GC TCD detectors obtained on the SAM flight unit during calibration and environmental testing

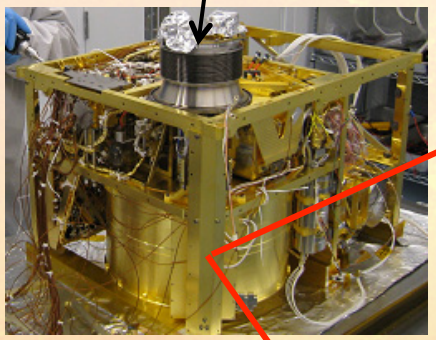




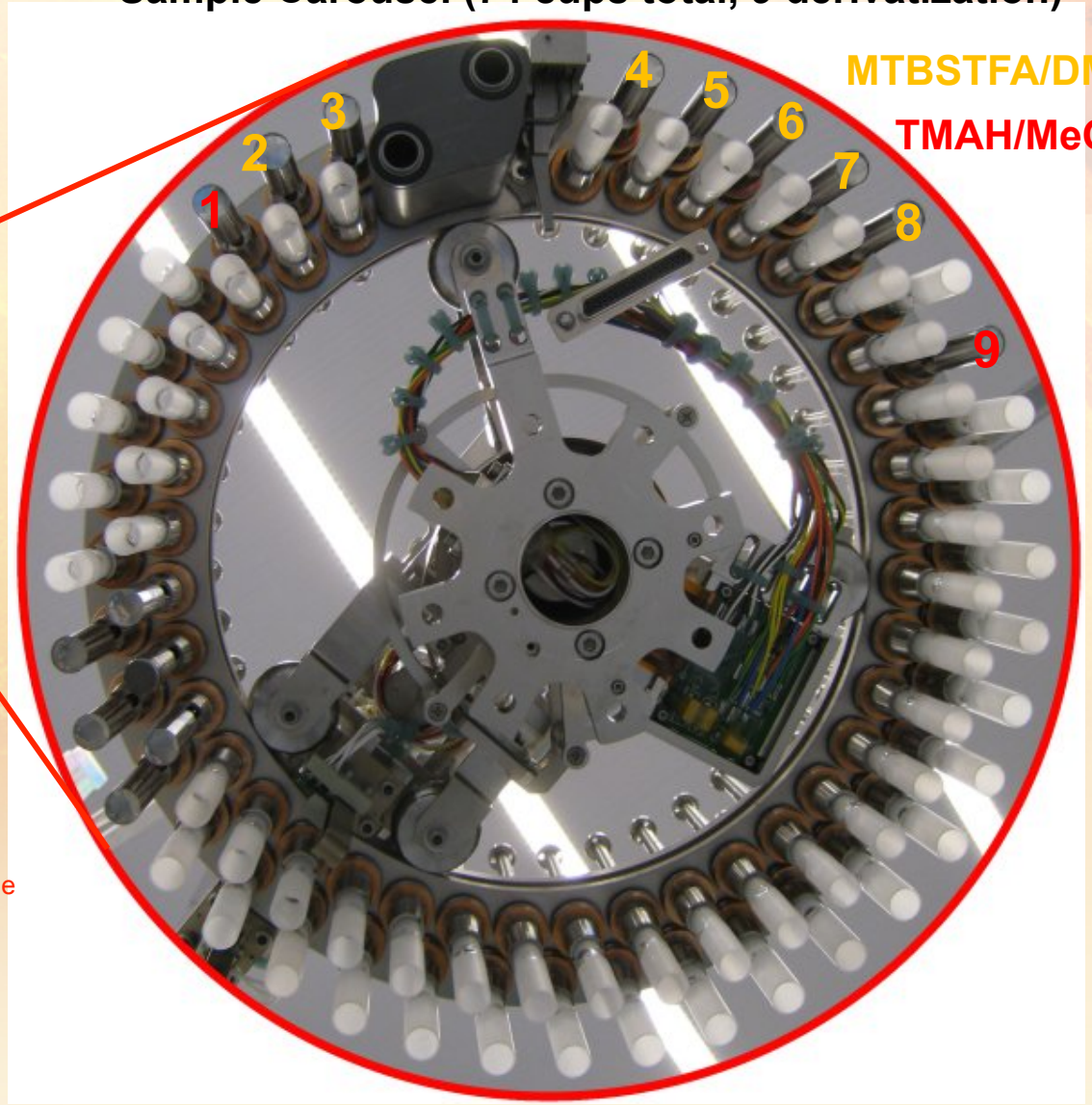
GCMS spectra obtained on the SAM flight unit during calibration and environmental testing

SAM Derivatization Experiment

Solid Sample Inlet Tube
(~ 0.5 g soil dropped to cup)



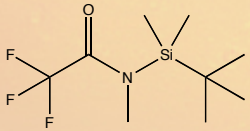
Sample Carousel (74 cups total, 9 derivatization)



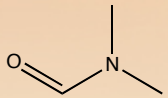
MTBSTFA/DMF (7 cups)

TMAH/MeOH (2 cups)

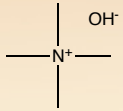
MTBSTFA: N-(*tert*-butyldimethylsilyl)-N-methyltrifluoroacetamide



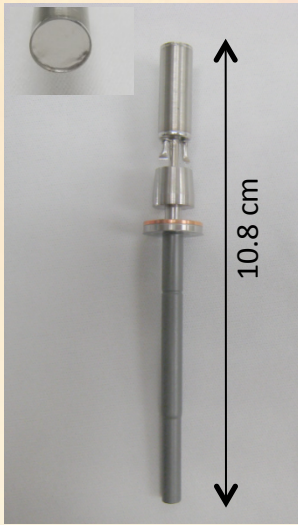
DMF: Dimethylformamide



TMAH: Tetramethylammonium hydroxide

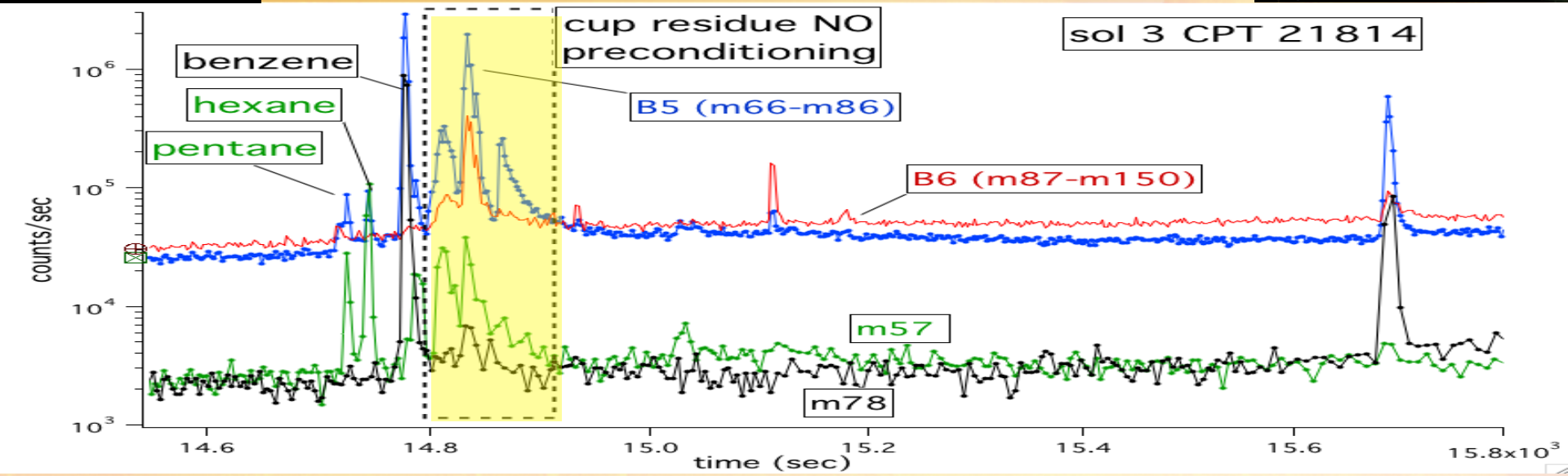


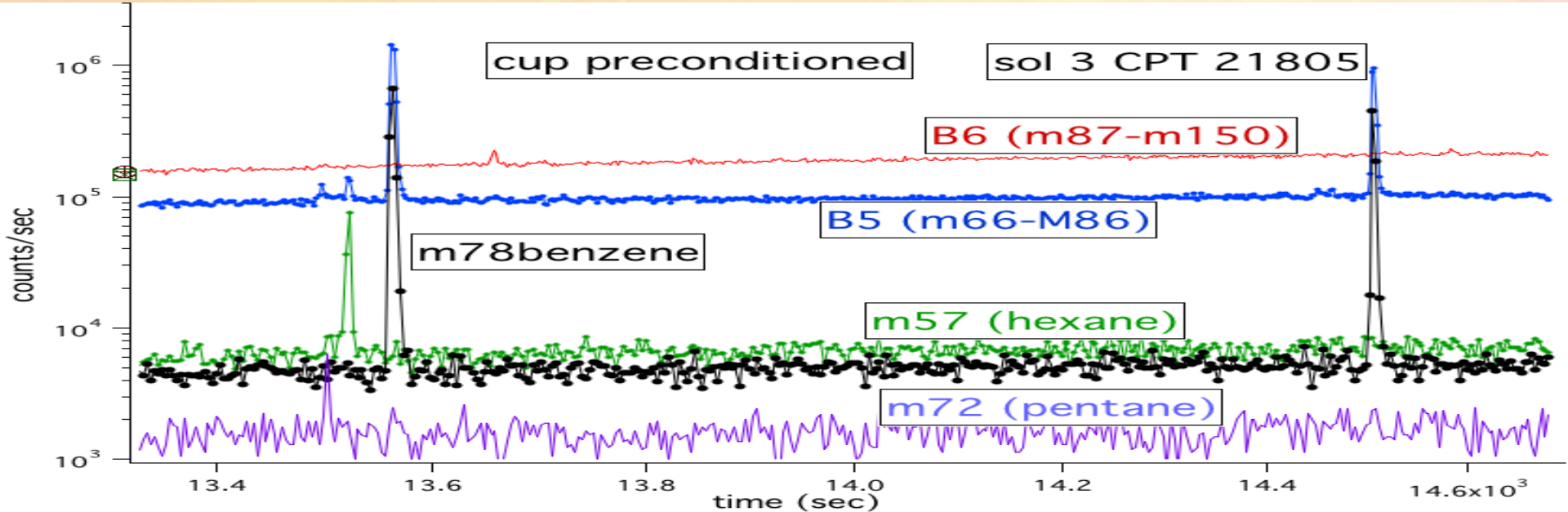
MeOH: Methanol



SAM test, qualification, and calibration

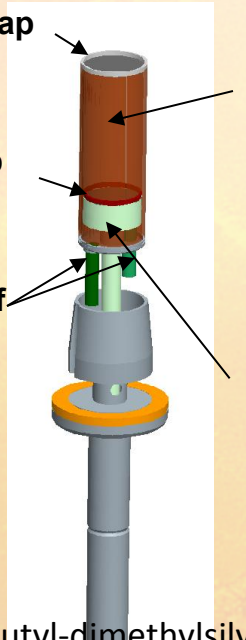
Runs with and without cup preconditioning gives residual contamination level in SMS





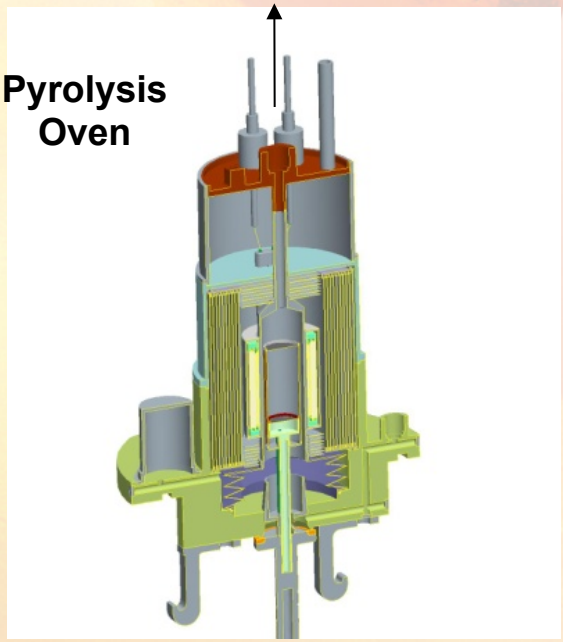
Derivatization Chemistry

Contents of Derivatization Cups:



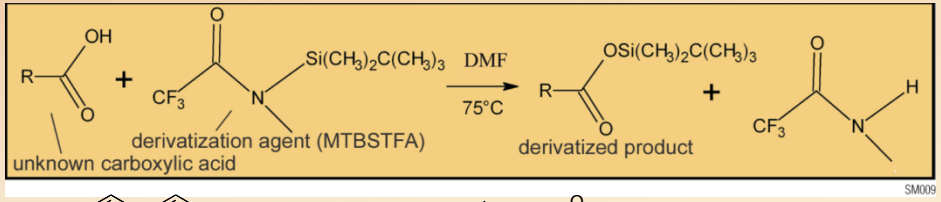
Outer volume:
0.5 ml MTBSTFA/DMF (4:1) or TMAH/MeOH (1:3)
25 nmol pyrene (in solution)
Solvent degassed by 3 freeze-pump-thaw cycles

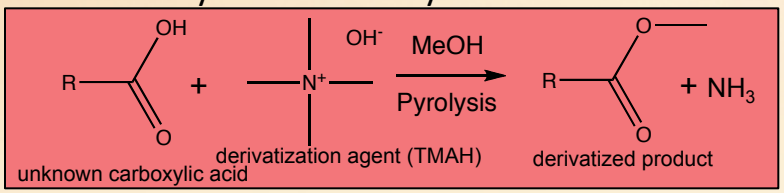
Inner volume (Internal standard):
40 nmol 3-Fluoro-DL-valine (MTBSTFA) or 13 nmol Nonanoic acid (TMAH)

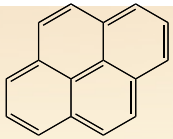


tert.-butyl-dimethylsilyl-N-methyltrifluoroacetamide (MTBSTFA) and dimethylform-amide (DMF)

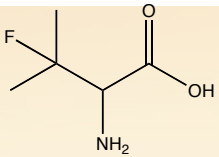
tetramethylammonium hydroxide



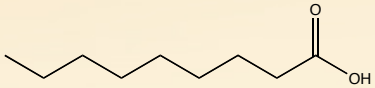




pyrene



3-Fluoro-DL-valine



Nonanoic acid

Organic Check Material

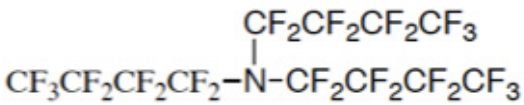
OCM Bricks

Organic check material

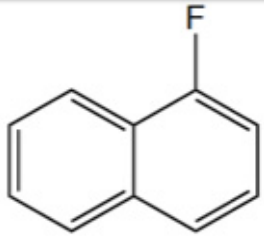
- Amorphous silica with about 30% porosity
- Compressibility about 12 MPa
- Doped with fluorinated compounds

In flight calibrants

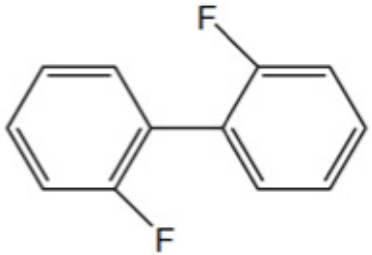
- Fluorinated compounds present both in calibration cell and in selected sealed cups



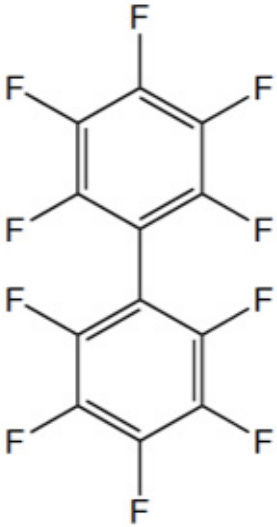
PFTBA



1-FN



DFBP



PFBP

SAM measurement modes

On board command SCRIPTS control each sequence

Evolved Gas Analysis

Solid Sample Quartz Cup → Heat in He (to 950-1100°C) → EGA of volatiles with QMS → Isotope & composition with TLS → Hydrocarbons trapped at selected T → GCMS analysis

Combustion Analysis

Solid Sample Quartz Cup → Combust in O_2 (T > 800°C) → CO_2 transfered to TLS → Isotope analysis of CO_2 with TLS

Wet Chemistry Analysis

Solid Sample Metal Cup → Derivatization of amine, acid, and alcohol groups TMAH/MeOH or MTBSFA/DMF → Thermal desorption (~350°C) → Hydrocarbons trapped → GCMS analysis

Rock Sample

Direct Atmospheric Analysis

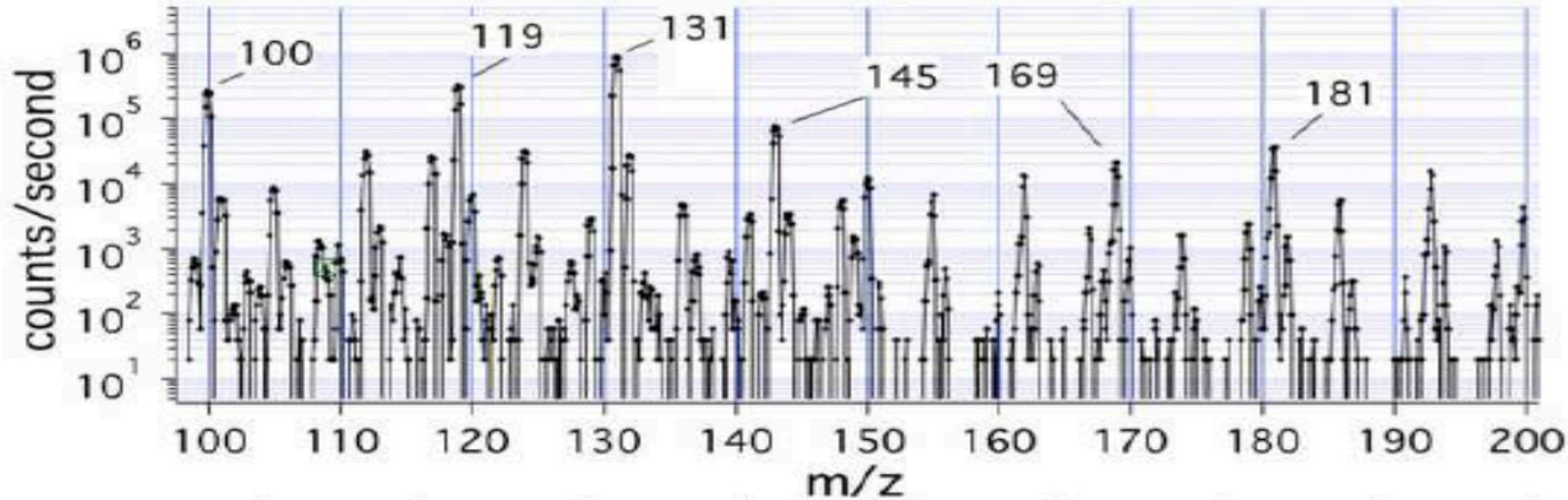
Atmospheric Sample → Composition & isotopes with QMS → Composition & isotopes with TLS

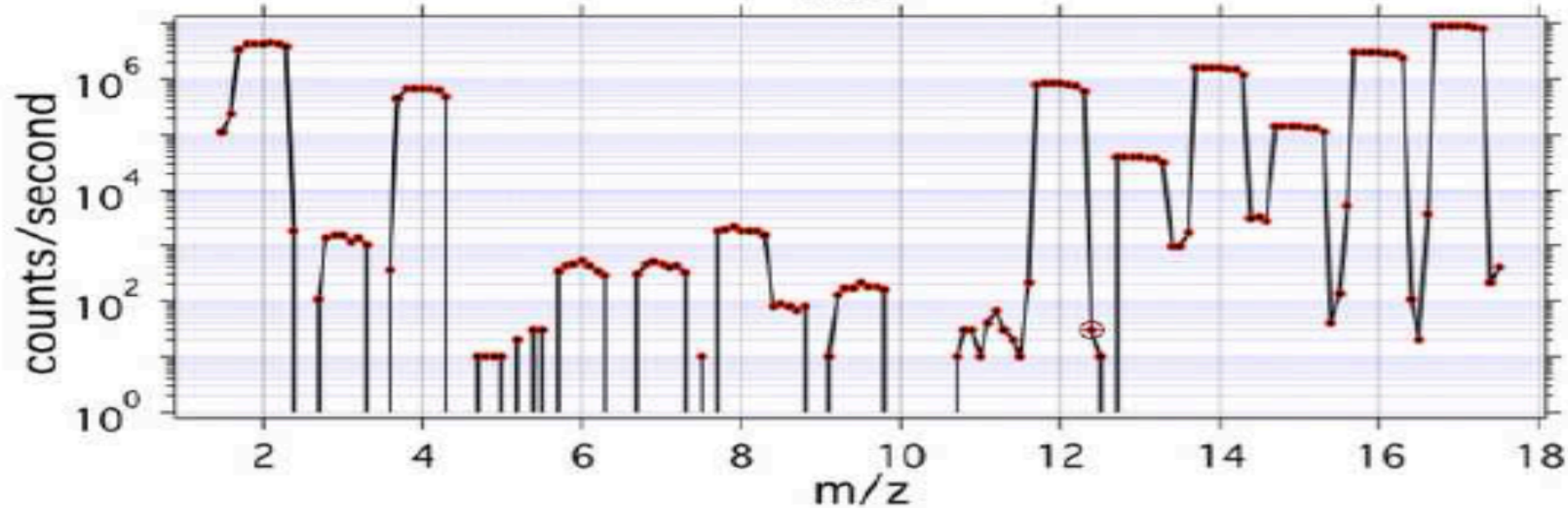
Atmospheric Analysis/Enrichment

Atmospheric Sample → Methane enrichment → Noble gas enrichment → Trace species enrichment → Isotopes with static QMS → Composition & isotopes QMS/GCMS → Methane abundance & isotopes/TLS

Air Sample

Calibration of the SAM Flight Model



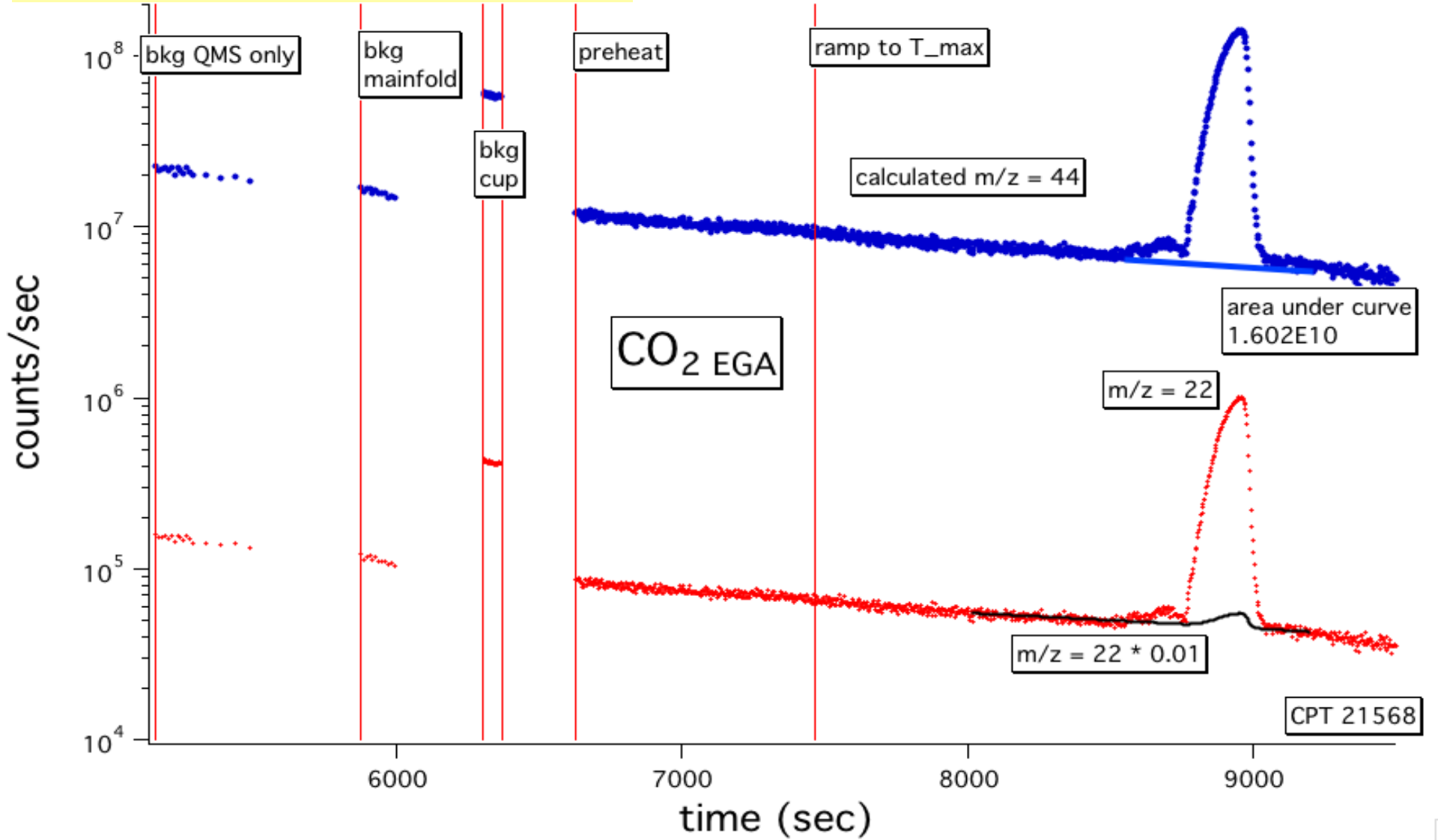


SAM test, qualification, and calibration

During thermal qualification core sequences were exercised with mixtu
of calcite, heptahydrate melanterite, and fluorocarbons on inert silica gla
or for volatile species introduced to the SAM hydrocarbon trap.

$CaCO_3$ (s) → CaO (s) + CO_2 (g)

Detection limit estimated to be ~10 microgram



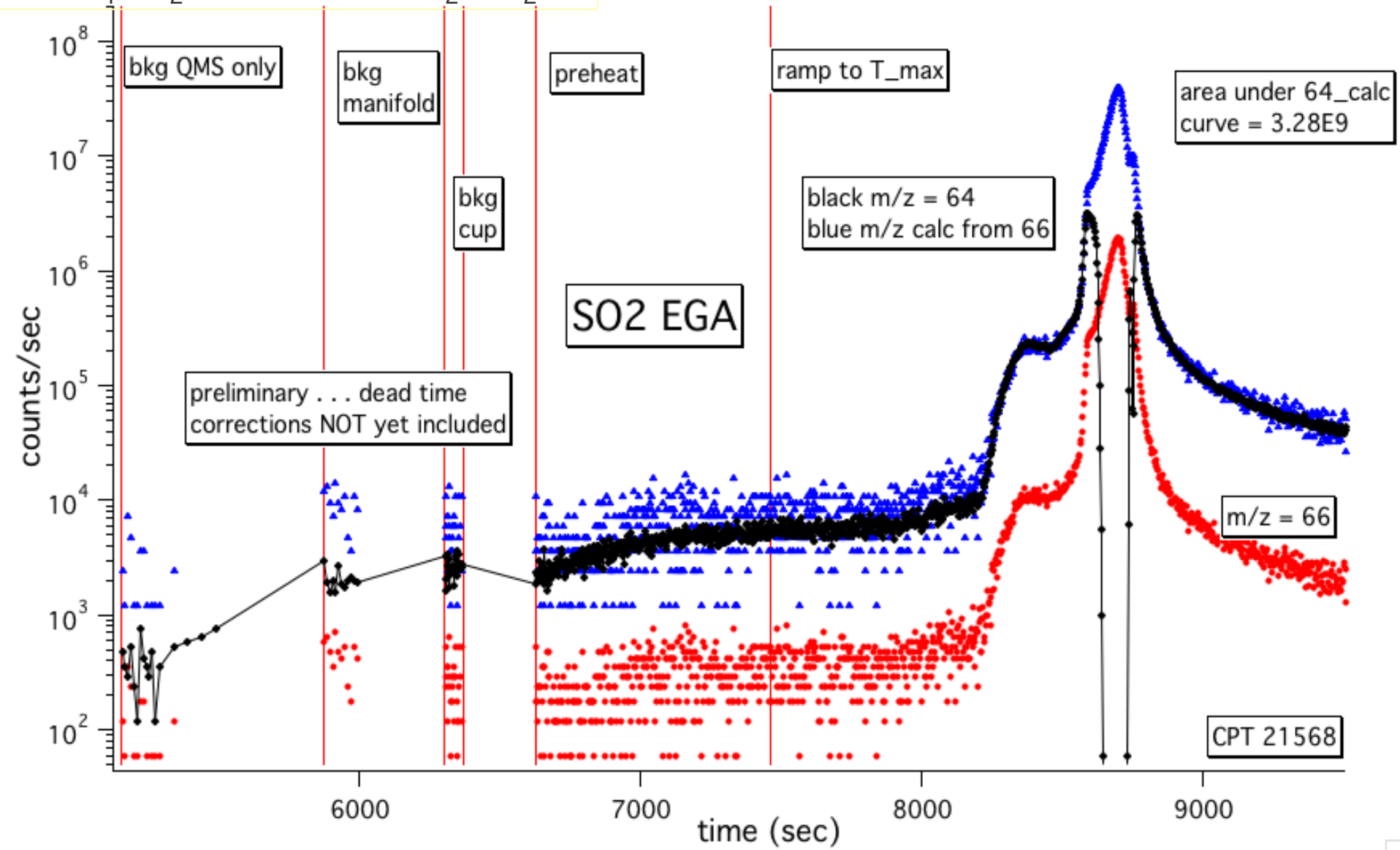
SAM test, qualification, and calibration

During thermal qualification core sequences were exercised with mixture of calcite, heptahydrate melanterite, and fluorocarbons on inert silica glas

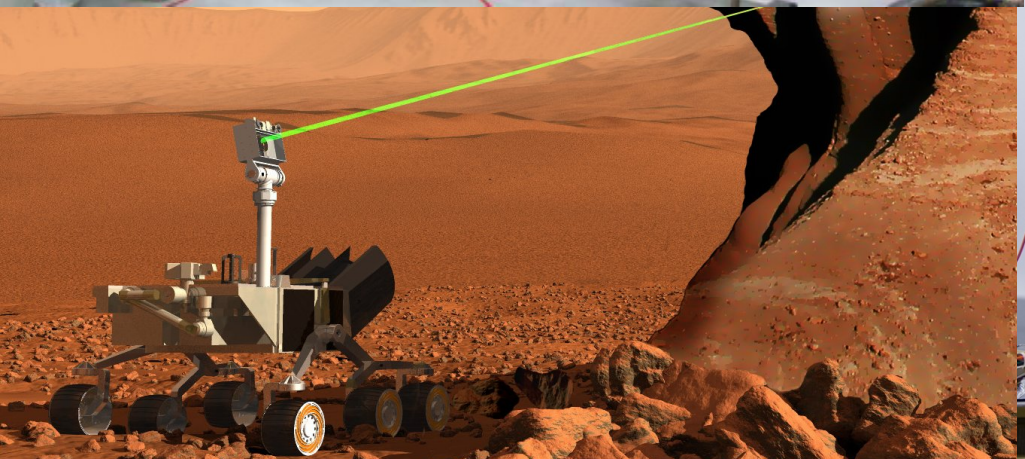
CONTINUED DATA ANALYSIS ON-GOING

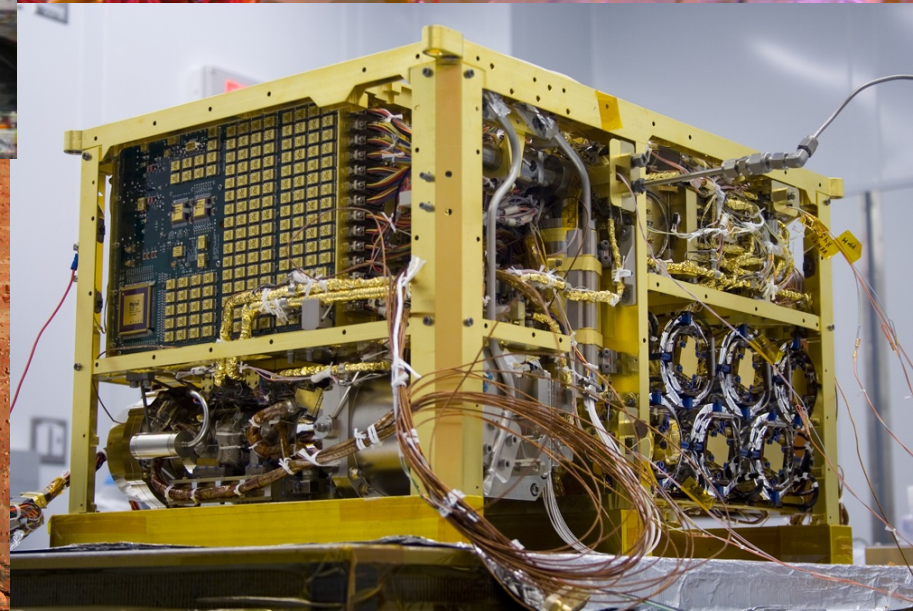
$FeSO_4.7H_2O \rightarrow$ evolved $SO_2 + H_2O$

Detection limit estimated to be ~200 nanogram

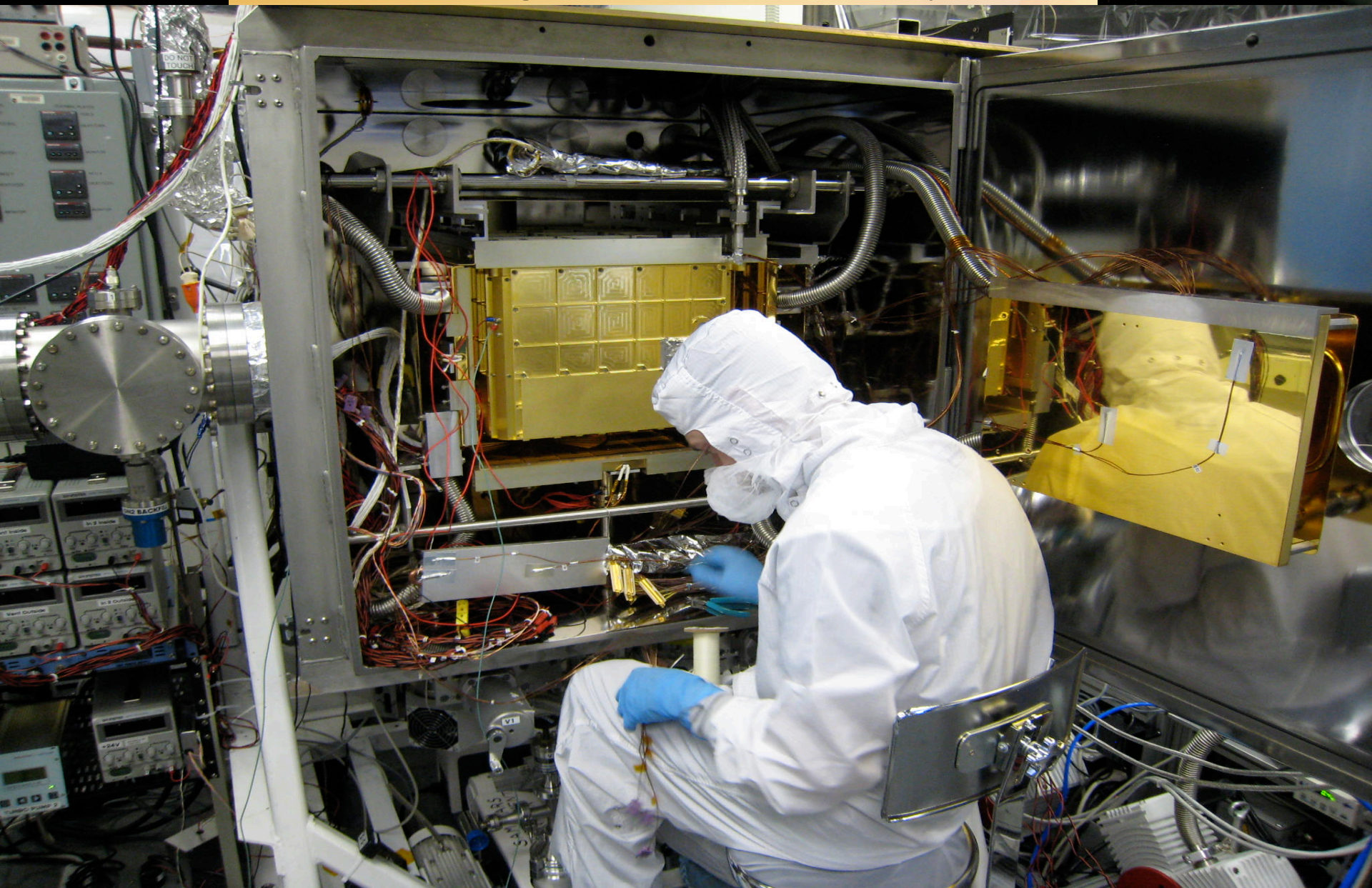


Bringing a Chemical Laboratory Named Sam to Mars on the 2011 Curiosity Rover





High fidelity SAM testbed used in high fidelity environment to test measurement sequences prior to radiating them to SAM on Curiosity



Mars Organic Molecule Analyzer - MOMA

2018 ExoMars Rover Mission

Goal: Seek signs of life on Mars

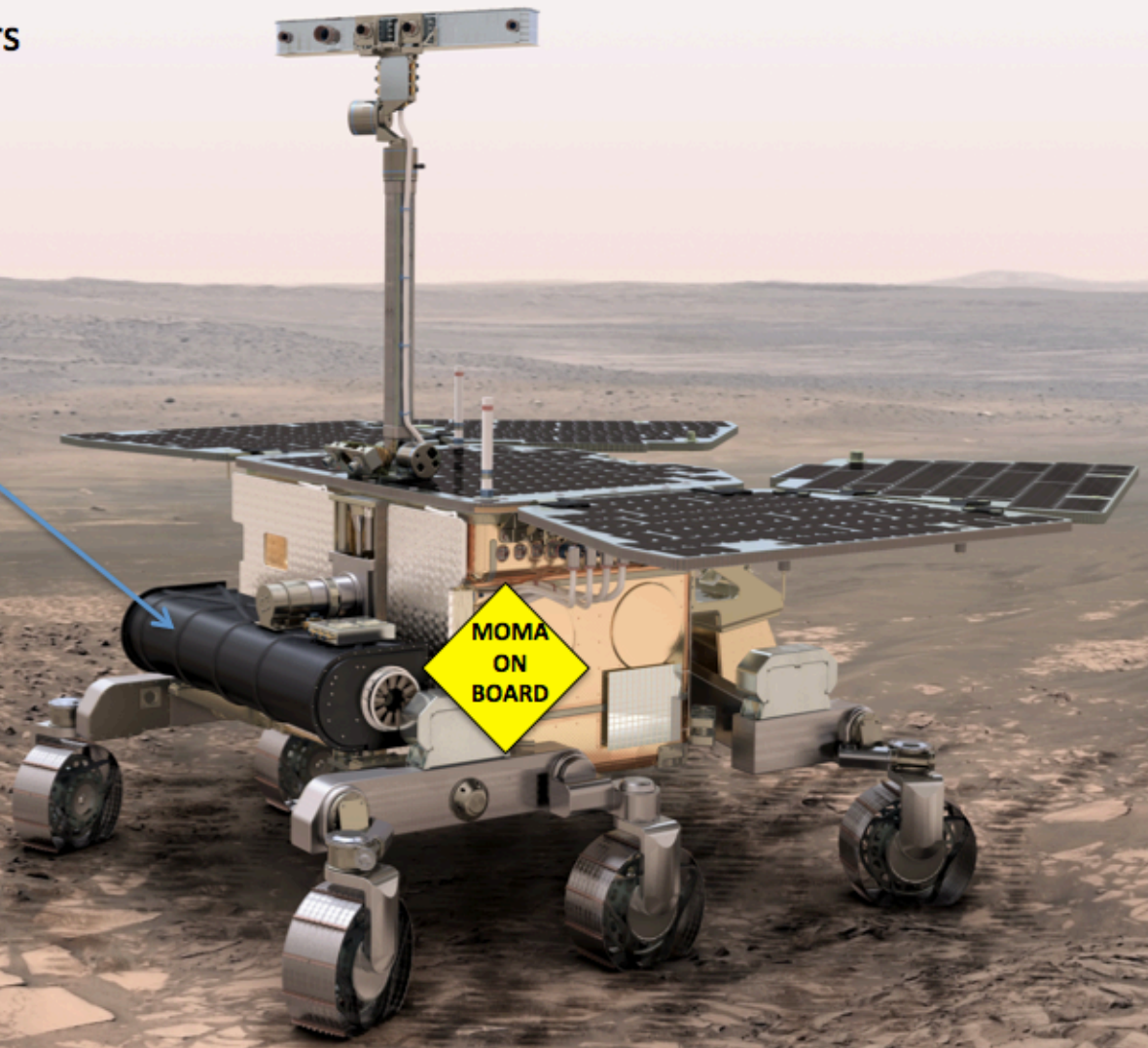
300 kg "MER Plus" solar rover

2 m deep coring drill!

a bunch of great instruments

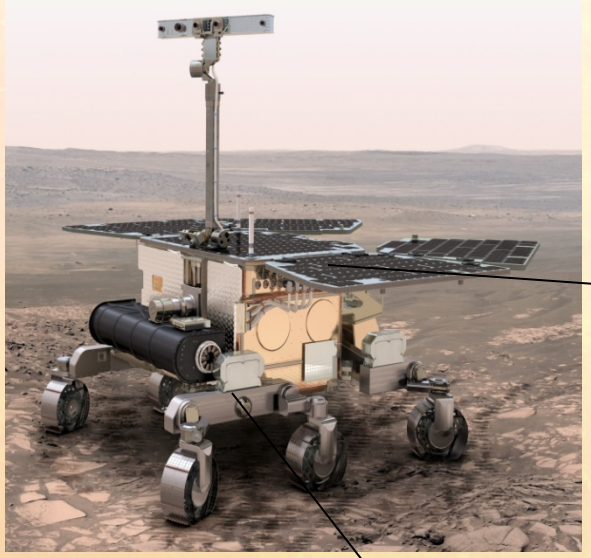
including one very, very cool mass spectrometer ...

MOMA!



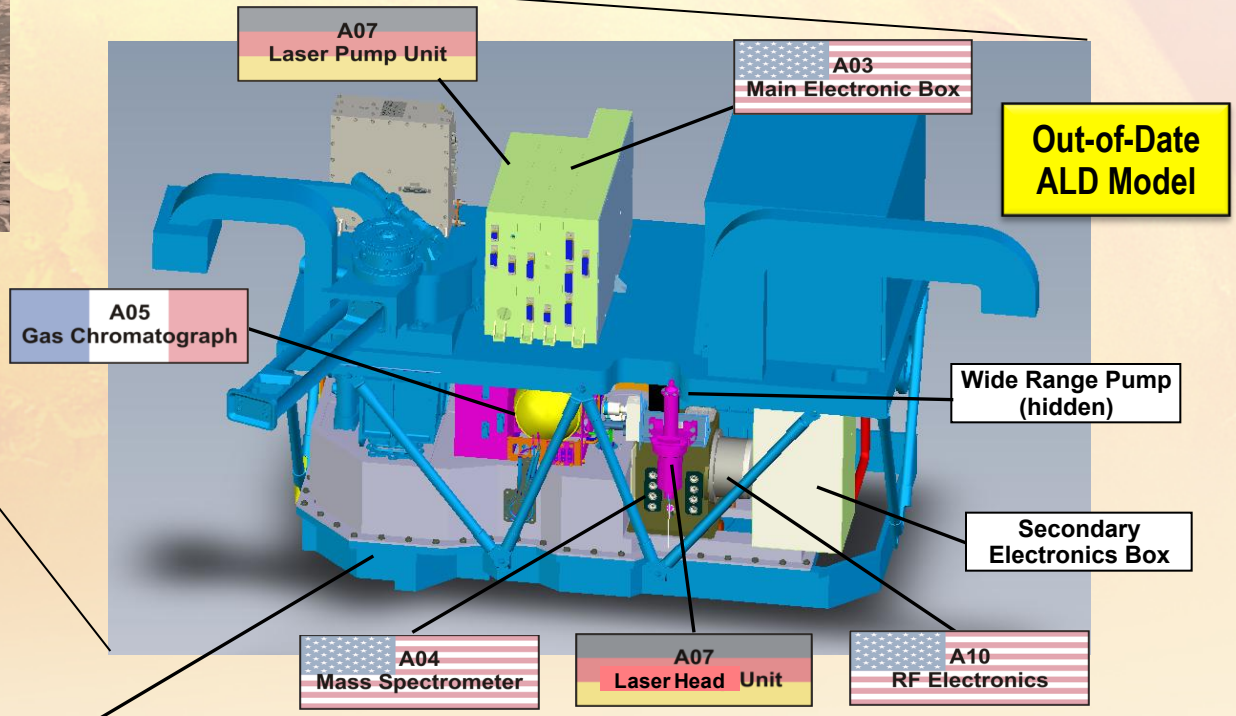
MOMA Investigation on ExoMars 2018

Principal Investigator: Fred Goesmann, MPS



MOMA directly addresses the top in situ science goal of ExoMars to seek the signs of past or present life on Mars.

MOMA is a dual-source mass spectrometer-based investigation of organic molecules in Mars samples. The concentration, structure, and distribution patterns of organics can be indicators of life.



MOMA-MS is the Goddard-provided central mass spectrometer and electronics subsystem of MOMA.

A09
Oven

A08
Tapping Station

ExoMars Analytical Laboratory Drawer (ALD) includes MOMA

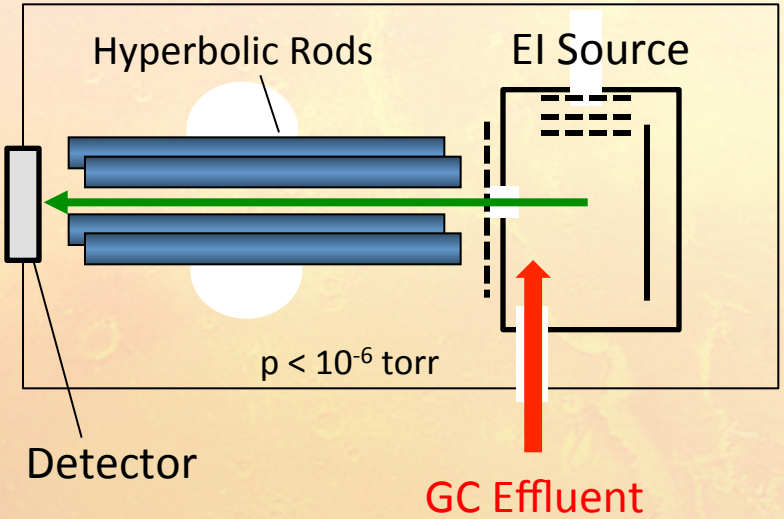
Why an ion trap for MOMA?

<u>Selection of the ion trap for the MOMA project</u>:

1. ability to interface with two **external ionization** sources including one at elevated (Mars) pressures (5-7 Torr)
2. ability to carry out **tandem mass spectrometry** or MS/MS
   a. provide definitive ID of organic compounds
   b. distinguish structural isomers
   c. peak in MS spectrum used to designate the precursor mass in subsequent MS/MS spectrum

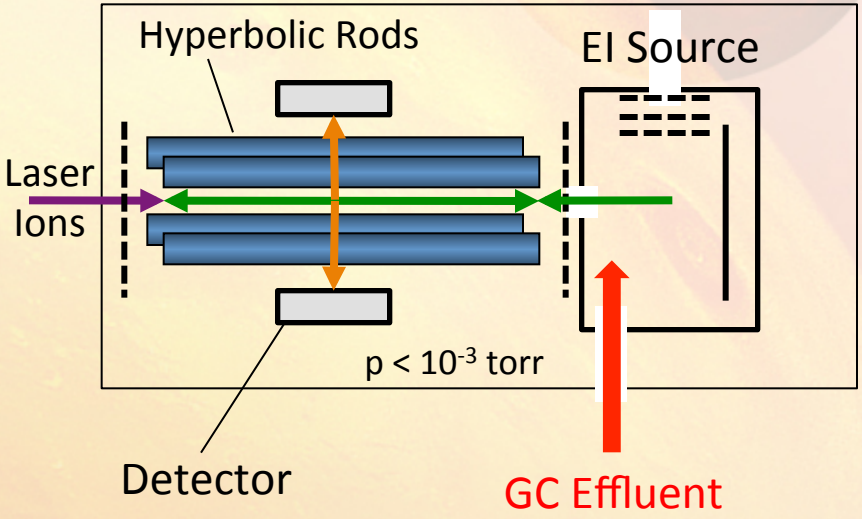
Separated at Birth

SAM
Quadrupole MS



One m/z value passes through rods to detector at given set of rod voltages; spectrum formed by scanning voltages.

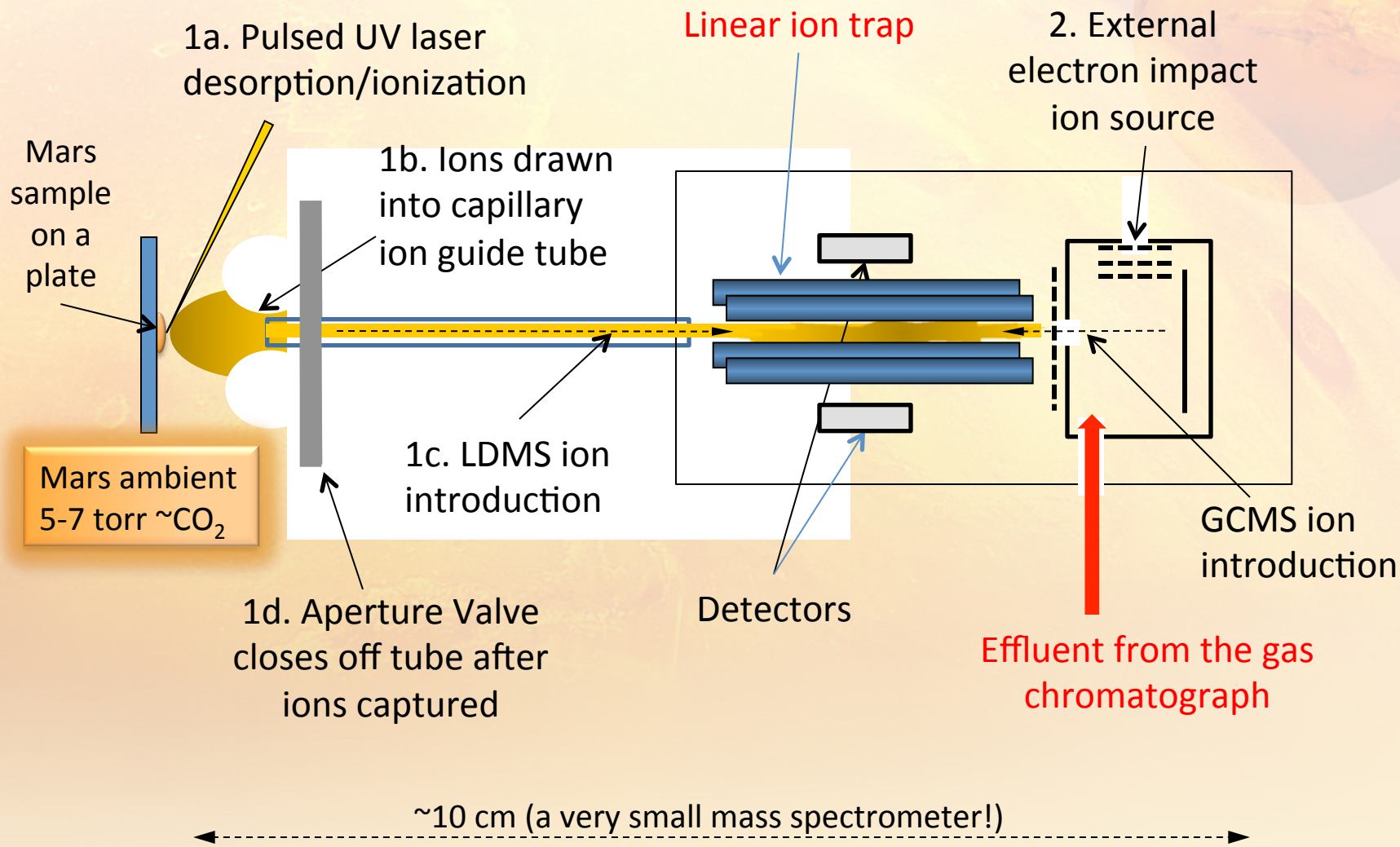
MOMA
Linear Ion Trap MS



All ions over m/z range are trapped in field of rods; spectrum formed by exciting ions radially to detector by scanning voltage.

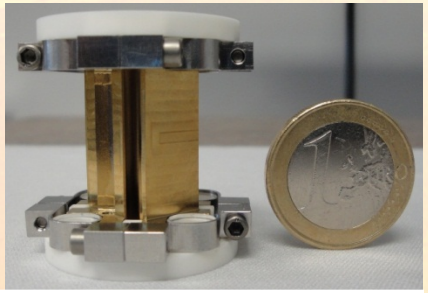
Heritage spaceflight QMS design readily translates to linear ion trap!

MOMA-MS:
Dual Source Ion Trap Mass Spectrometer

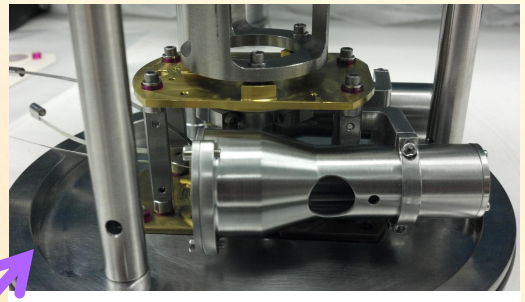


~10 cm (a very small mass spectrometer!)

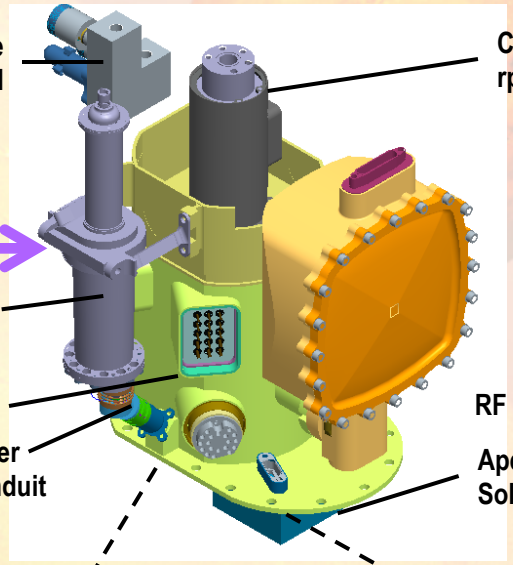
MOMA-MS Breadboards and Flight Design

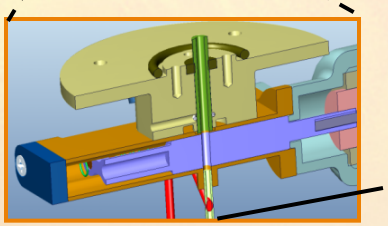


FLT-scale GB2 linear ion trap

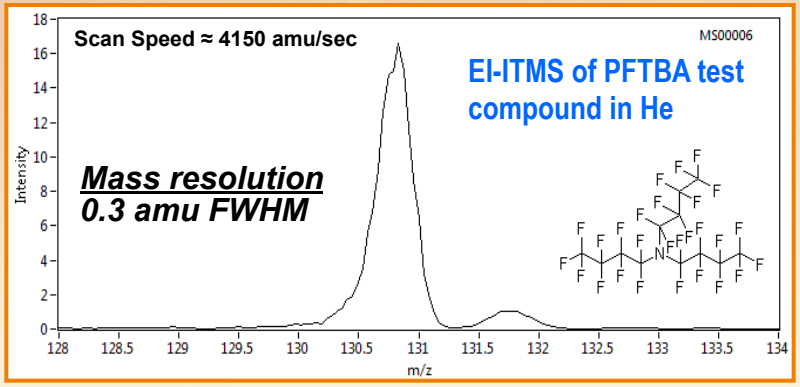


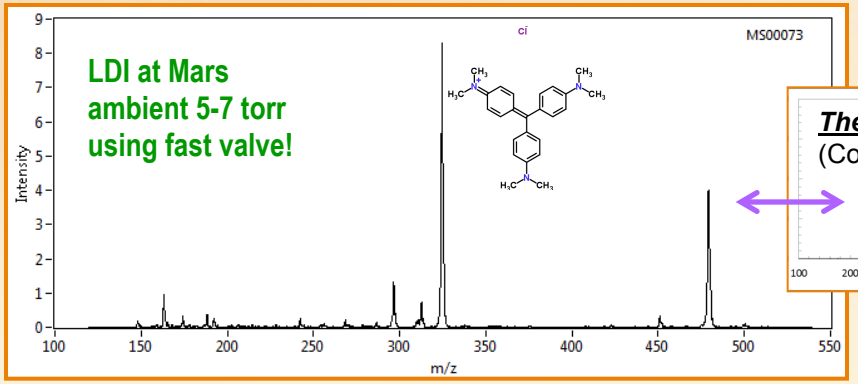
Mounted ion trap and detectors

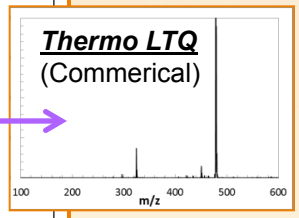




- **Latest breadboard demonstrates dual LIT performance at miniaturized Flight Scale**
- **Fast aperture valve enables LDI in the discontinuous API (DAPI) regime**
- **Ion trap analysis from French GC and German laser: end-to-end MOMA science**







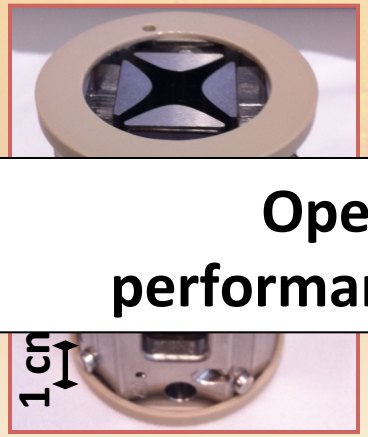
Trap Designs Under Test

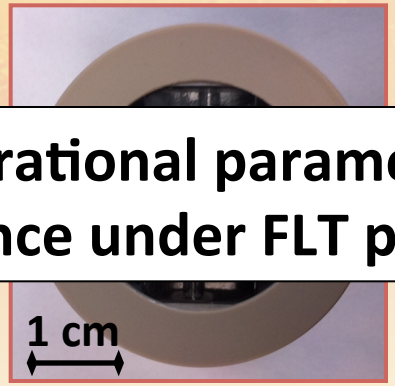
Breadboard "GB1" using Commercial LIT (LXQ or LTQ)

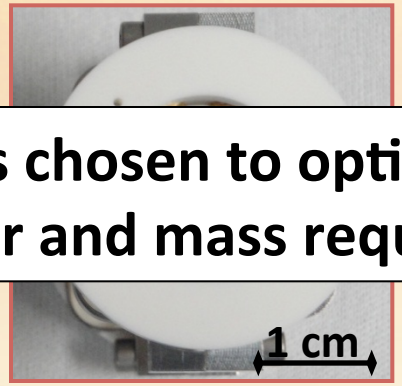
- r_0 = 4 mm
- Length = 63 mm
- RF frequency = 700-900 kHz
- Max RF voltage = 2000 Vpp

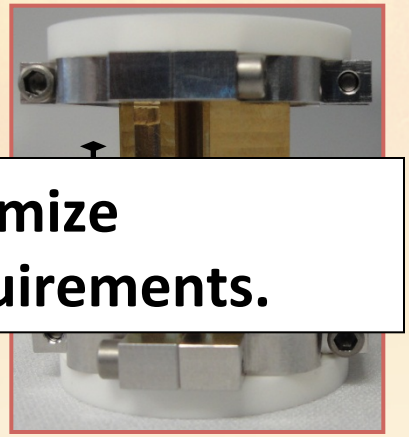
Breadboard "GB2" using custom/miniature LIT

- r_0 = **3mm**
- Length = **28 mm**
- **25%** of the LTQ volume
- RF frequency = 700 kHz
- Max RF voltage = 1000 Vpp





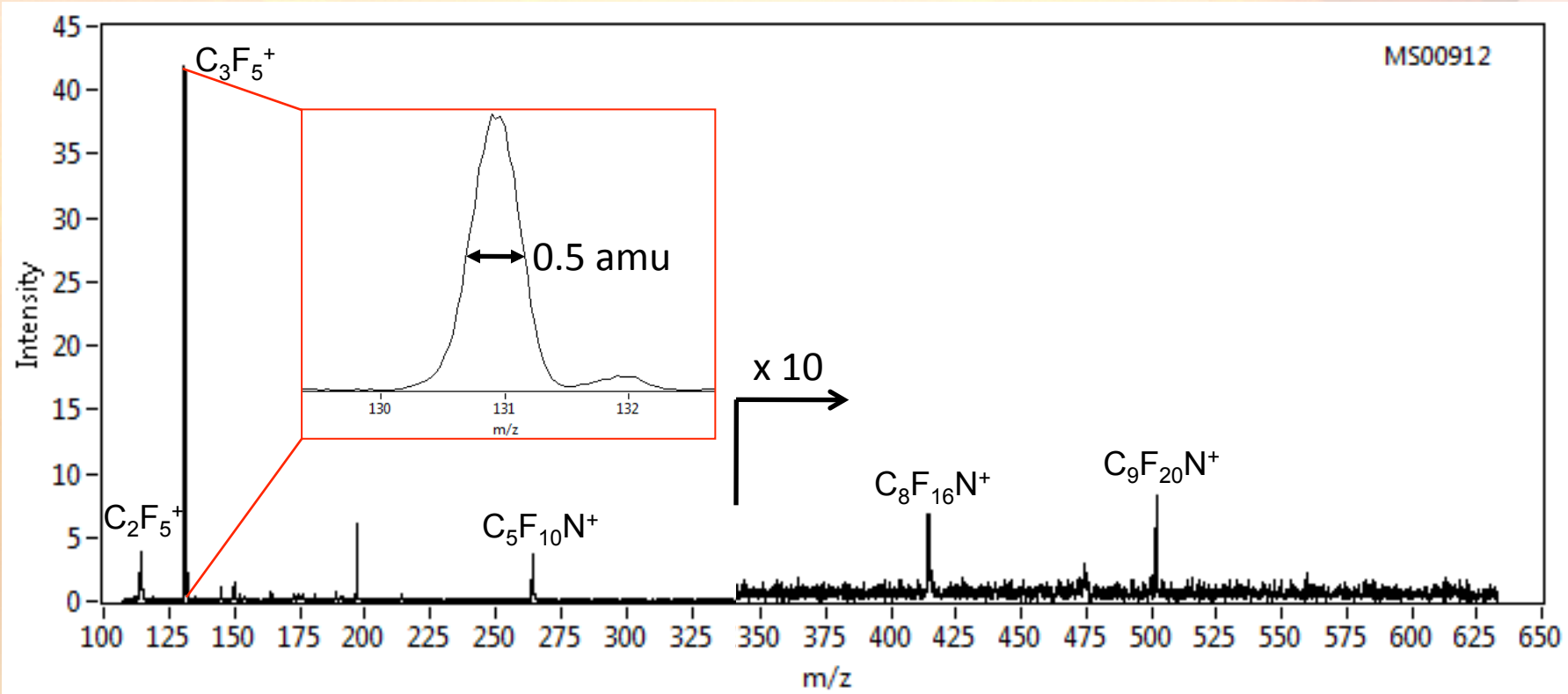




Operational parameters chosen to optimize performance under FLT power and mass requirements.

Resolution Performance
GB1 Breadboard (LTQ electrodes)

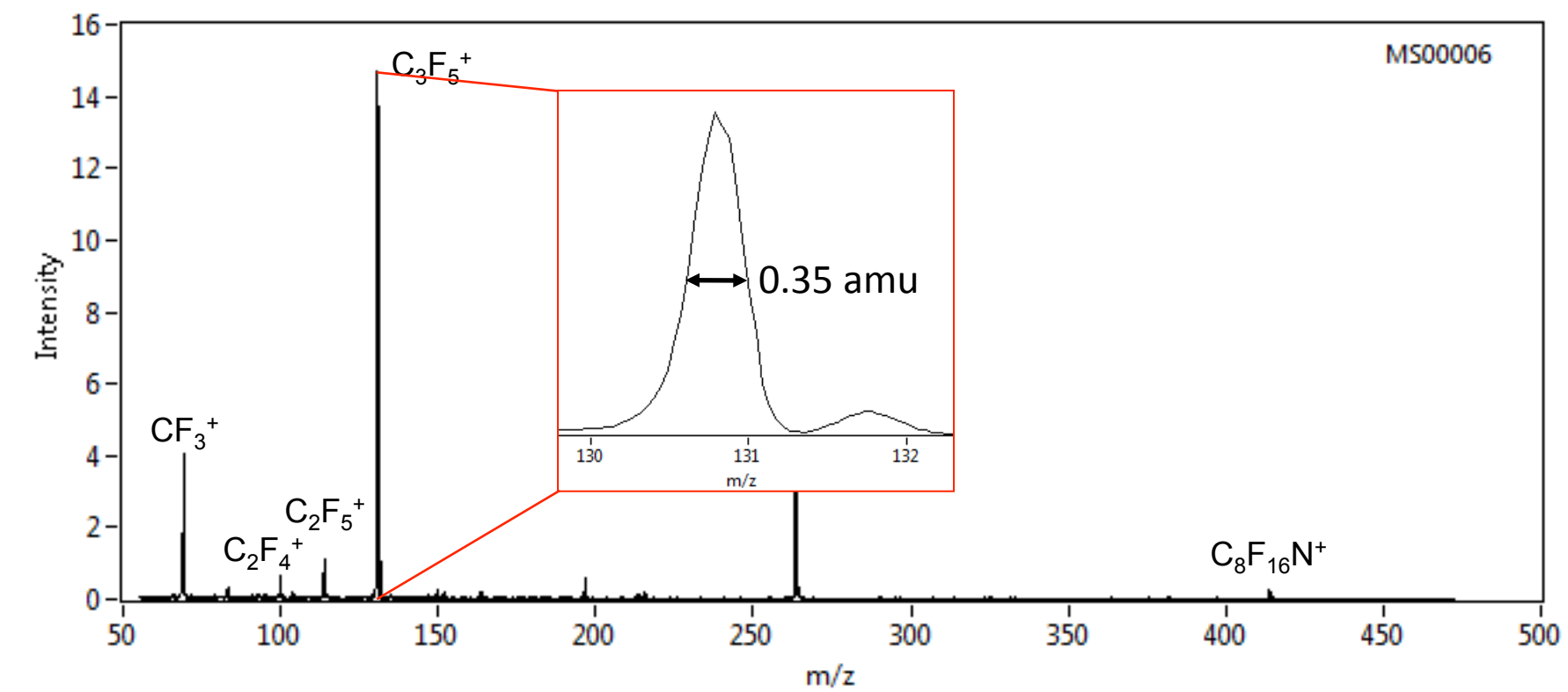
EI - PFTBA



GB1 with RF Breadboard Supply

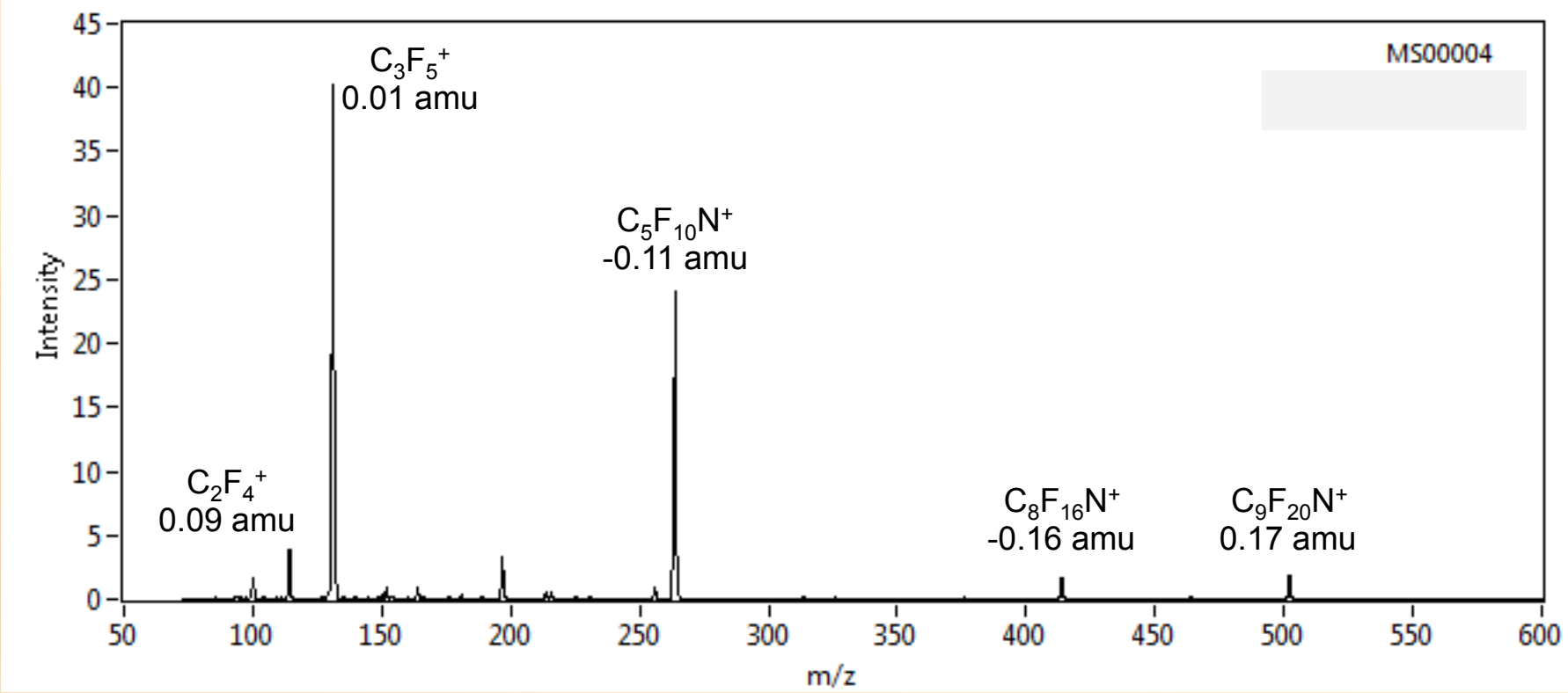
Resolution Performance – GB2

EI - PFTBA



EI Mass Range and Calibration

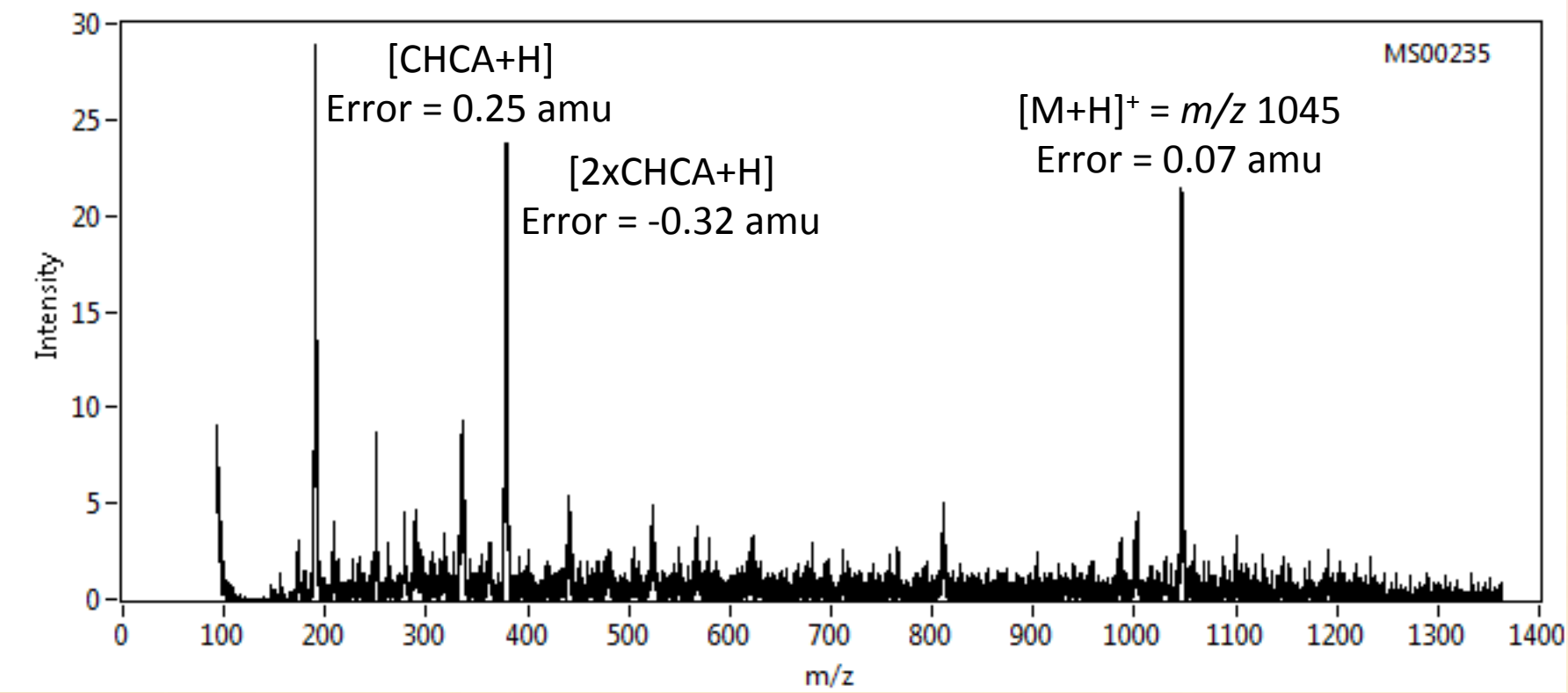
EI - PFTBA



Average Error = 0.12 amu

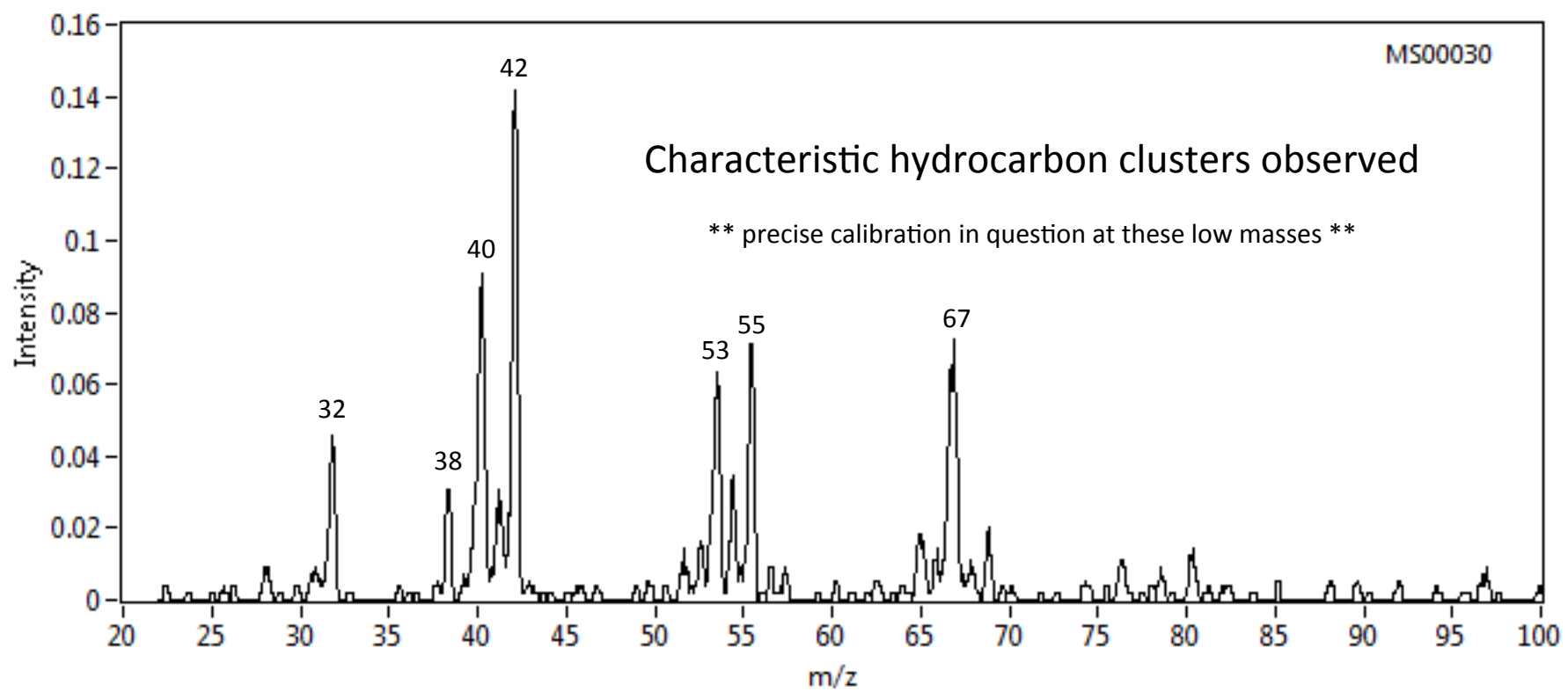
LDI Mass Range and Calibration– GB2

LDI – Angiotensin II



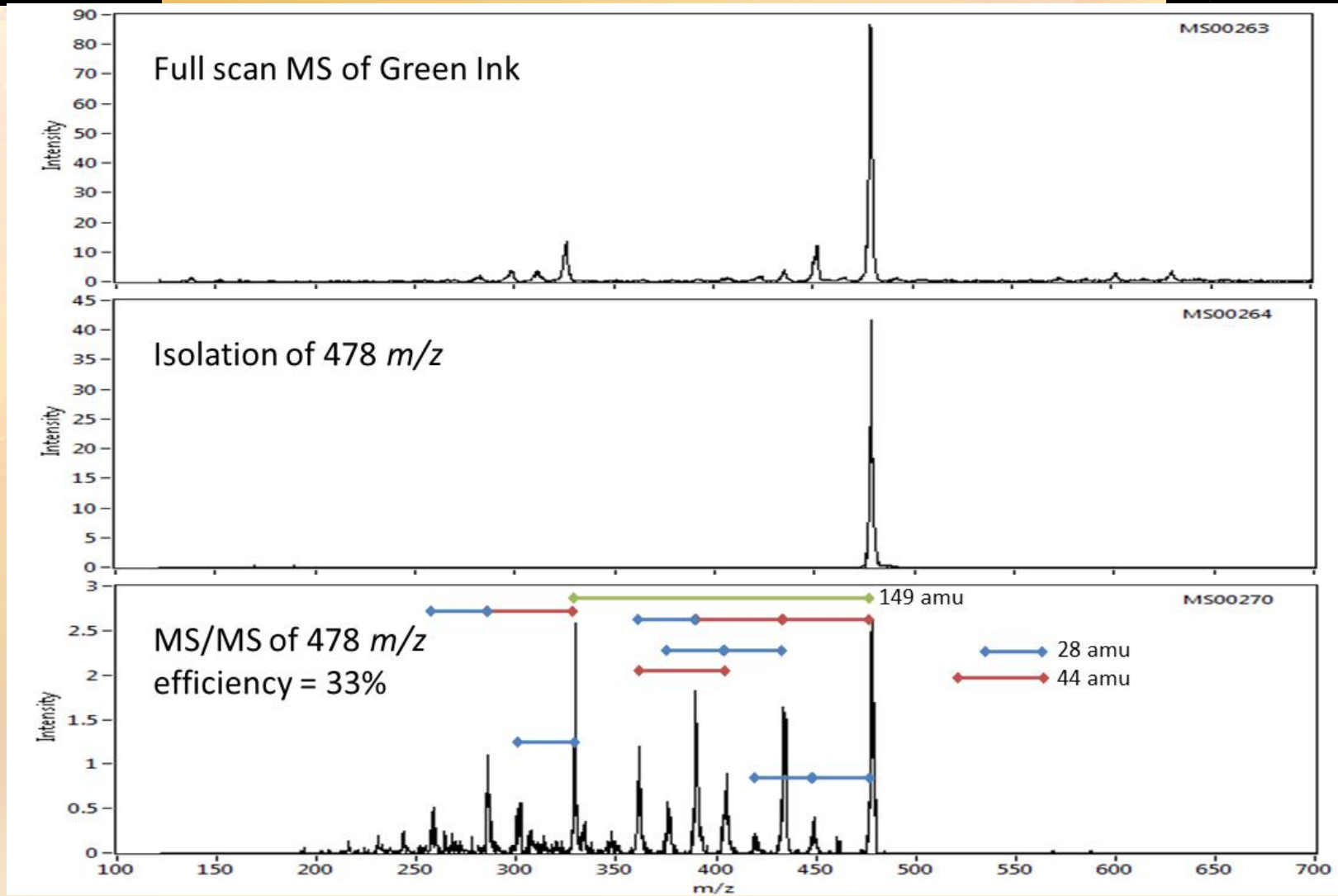
Average Error = 0.24 amu

EI Low Mass – GB2



Trapping of low masses possible with stable low RF voltages from RF breadboard

MS & MS/MS - ink standard



Future Opportunities for Planetary Probe Mass Spectrometers

Saturn: 2011 NRC Planetary Decadal Study

Key science themes:

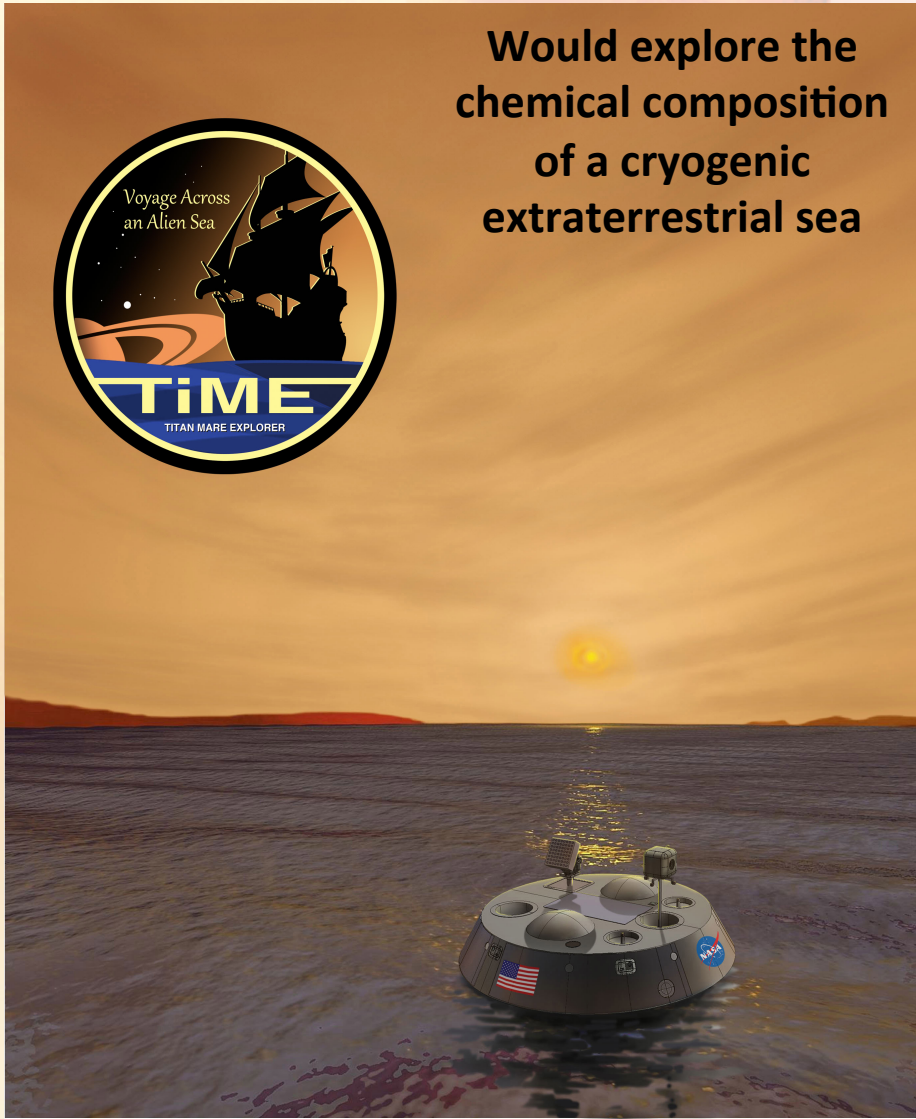
– Constrain models of solar system formation and the origin and evolution of atmospheres

– Provide a basis for comparative studies of the gas and ice giants

– Provide a link to extrasolar planetary systems

"The Saturn Probe mission will make important contributions to addressing giant planet goals in the period 2013-2022 by providing measurements of noble gas abundances that can be obtained in no other way and, thus, placing Saturn into context relative to Jupiter and the Sun." Vision and Voyages for Planetary Science in the Decade 2013-2022.

Titan Mare Explorer – TiME



Would explore the chemical composition of a cryogenic extraterrestrial sea