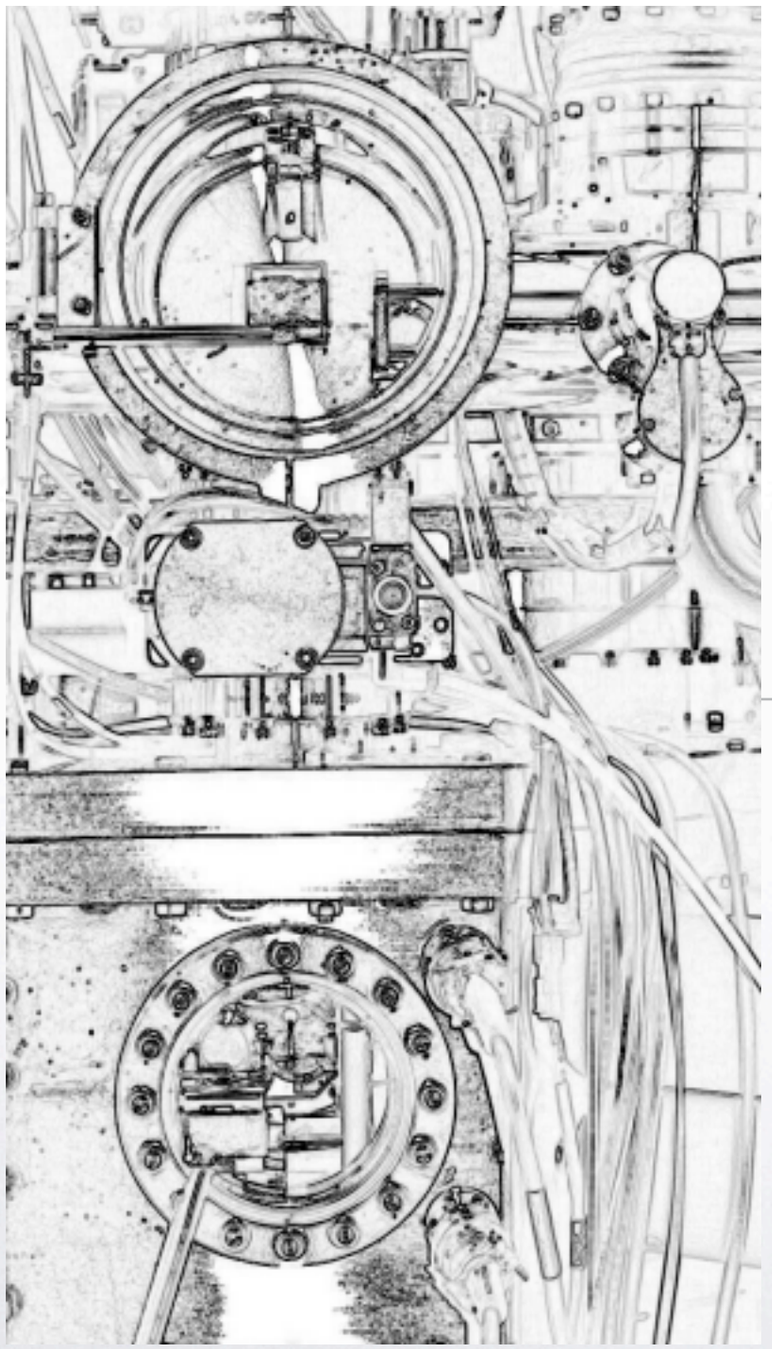

Towards SHRIMP SI: Developments in Stable Isotope Analysis with SHRIMP

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Research School of Earth Sciences

The Australian National University

Acknowledgments

- Ryan Ickert, Joe Hiess
- Peter Holden, Ian Williams
- John Foster, Peter Lanc, Ben Jenkins
- Electronics and Mechanical Workshops

Overview

- Historical
  - SIMS, SHRIMP, Stable Isotopes
- Current
  - SHRIMP II MC Oxygen isotopes
- Development
  - SHRIMP SI

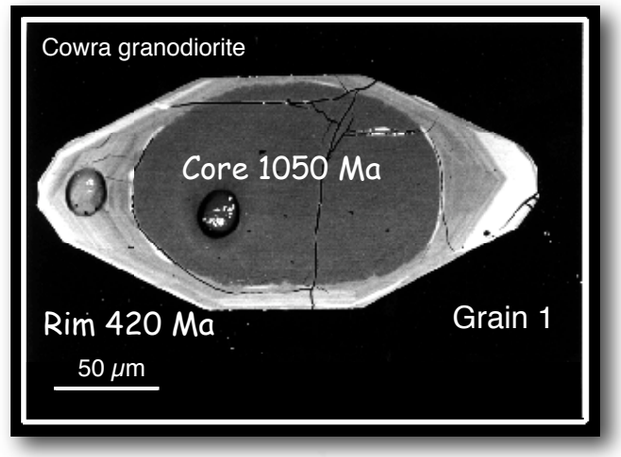
Stable-Isotope Analysis

- One of the first applications of SHRIMP
  - Coles et al. (1981; RSES Ann Rept)
    - Duoplasmatron with Ar^+ primary
    - PbS_2^- from Galena
      - combined Pb and S isotope analysis
    - S^- has higher yield
      - Faraday cup yields ± 0.3 ‰ in 20 minutes

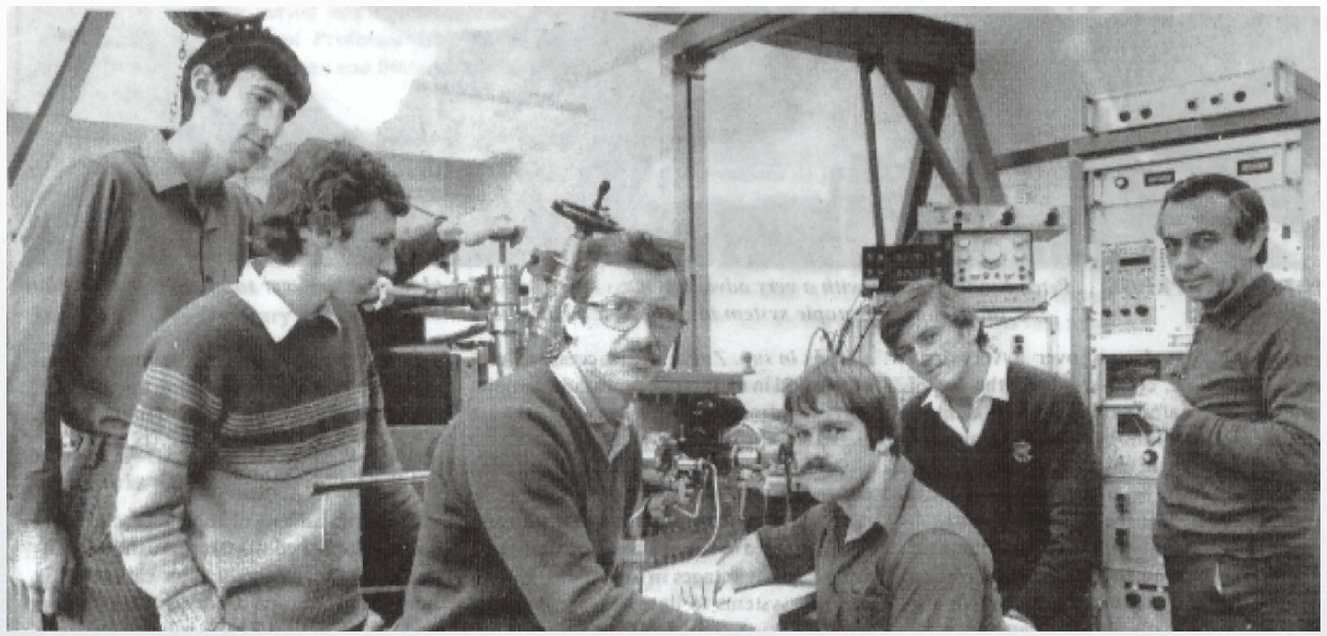
S-isotope Analysis

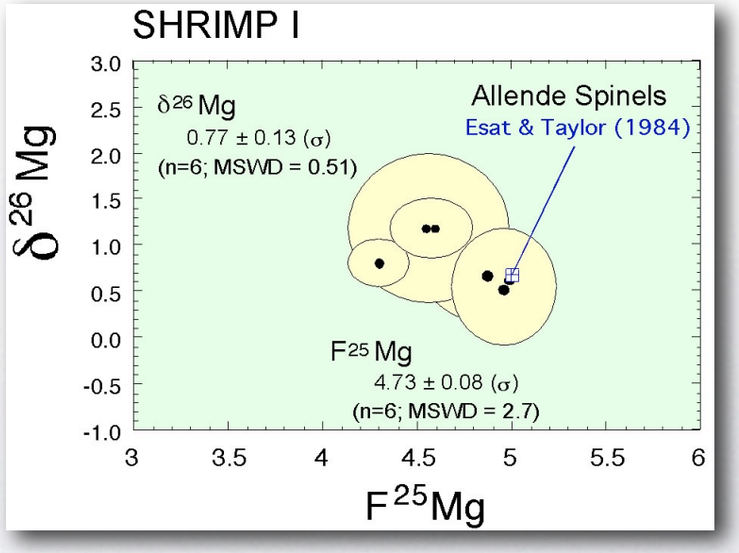
- Coles et al. noted:
  - "A primary Cs^+ beam will improve this(sensitivity)"
  - "further work has been deferred in the absence of an in-house sulphide specialist."
  - "ion probe has a fearfully short time interval between the decision to sample and getting a final isotopic result"

Positive secondaries



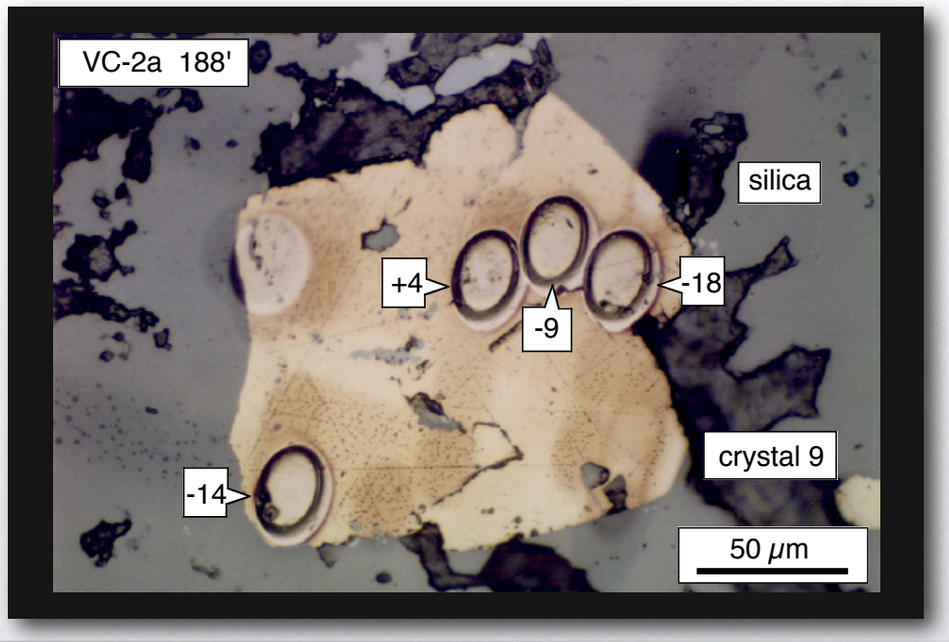
- U-Pb in zircon
- Ti, Mg isotopes in refractory inclusions





Stable isotope reprise: S^+

- Sulfide Specialist: Stewart Eldridge
- O_2^- primary; S^+ secondaries
  - few hundred kHz on $^{32}S^+$
  - electron multiplier yields ± 1 ‰
- First routine SIMS S-isotope analyses

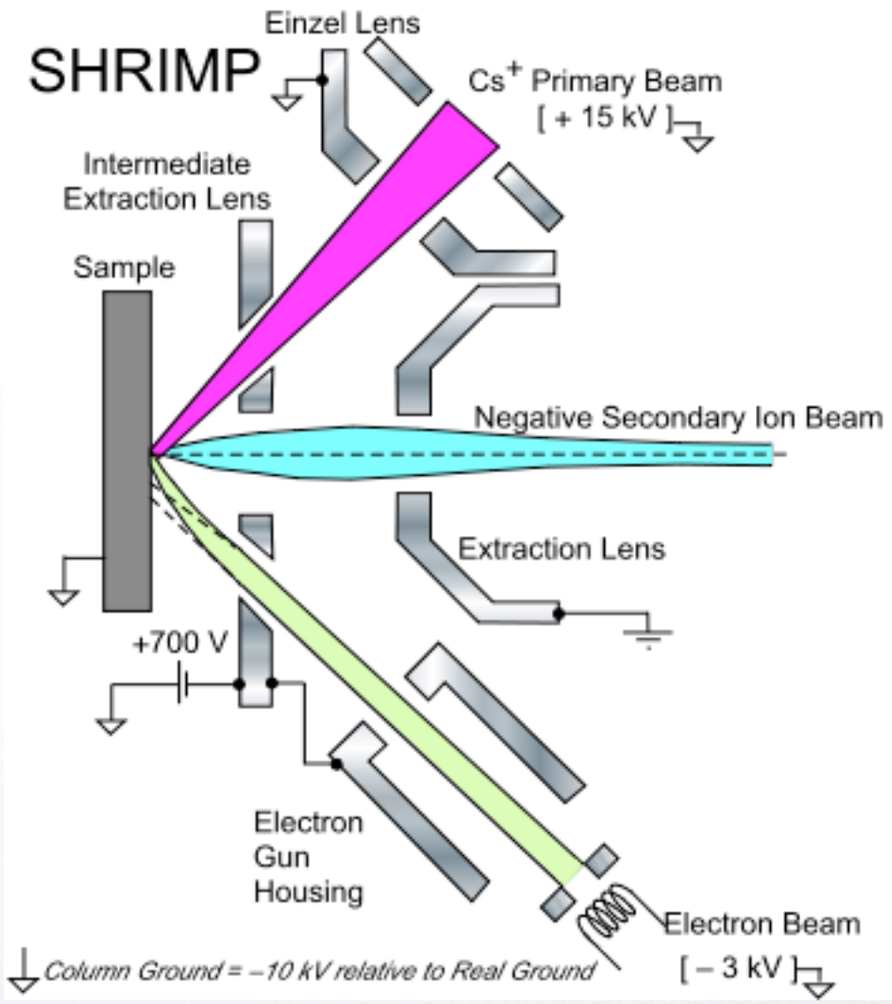


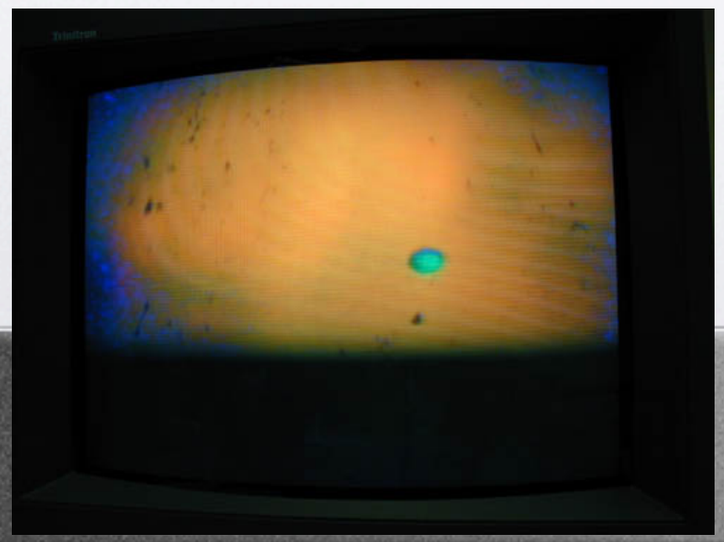
Stable Isotopes on SHRIMP II

- Cs Gun
- Kimball Physics IGS4
  - Cs zeolite

Electron Gun

- Electron beam
  - Energetic (1.5 keV)
  - Non-normal incidence
- Sustainable charge neutralization
  - no “settling”



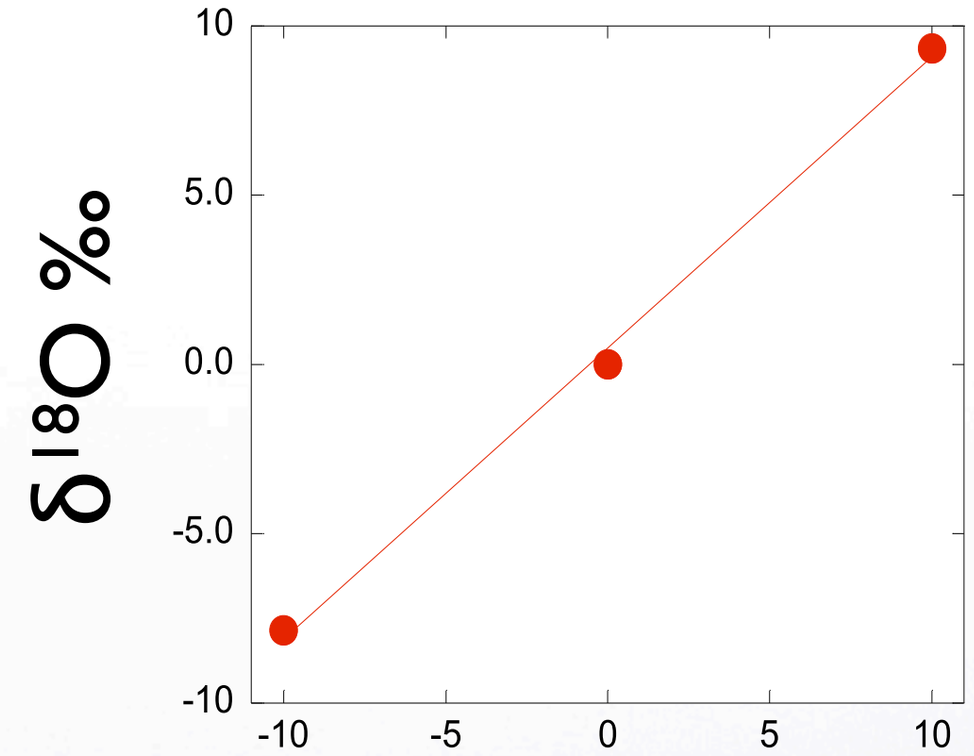


Stable isotope analysis

- Analytical Issues
  - Fractionation at source slit
    - Magnetic influence
    - Sensitivity to steering
      - Mount influence
  - Electron-induced secondary electrons

Fractionation

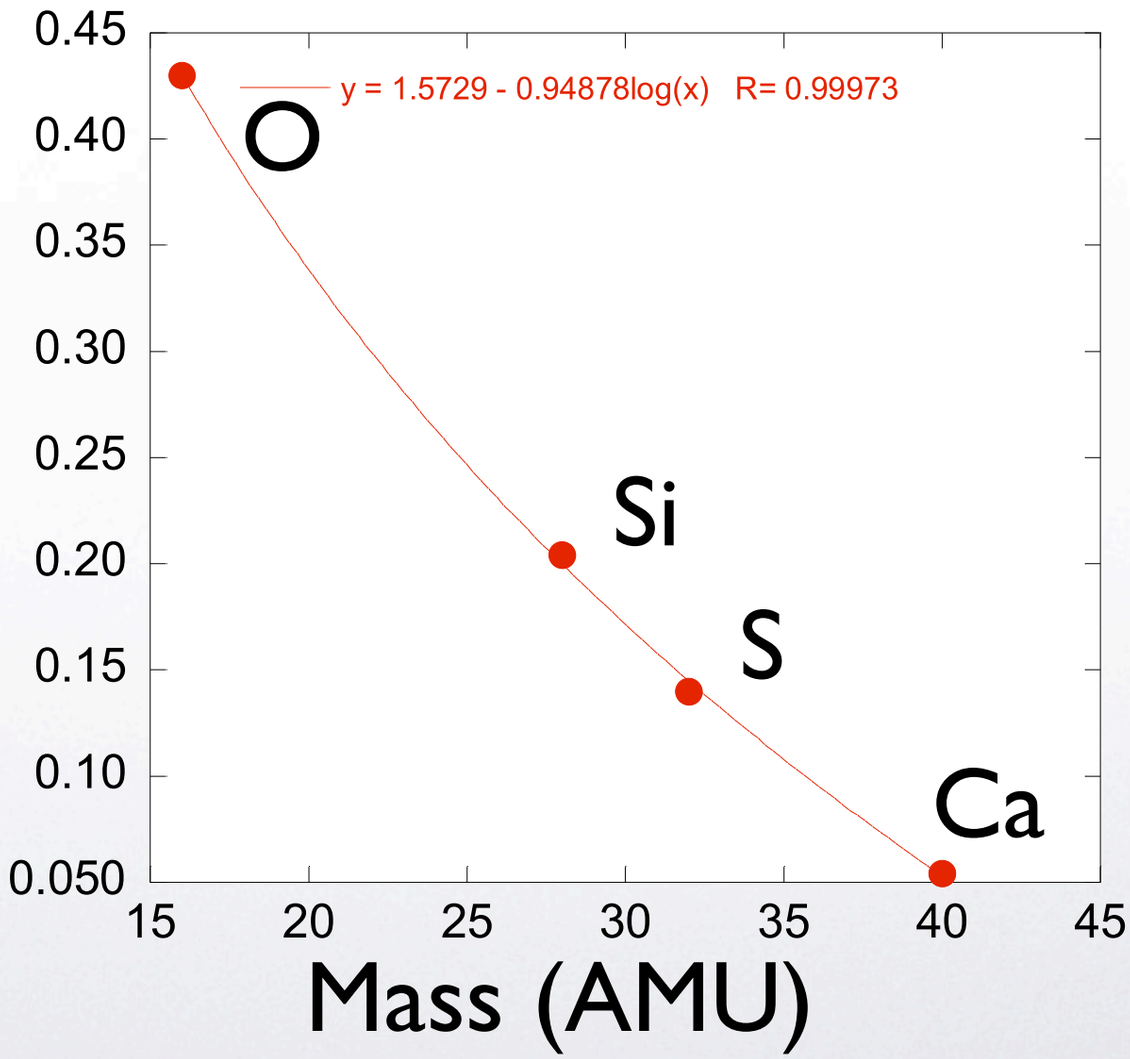
- Dependent on
  - steering
  - ion optics
  - beam dimensions



initial results in 2004

Mass bias at source slit

Fractionation (‰ per bit of lateral steering)

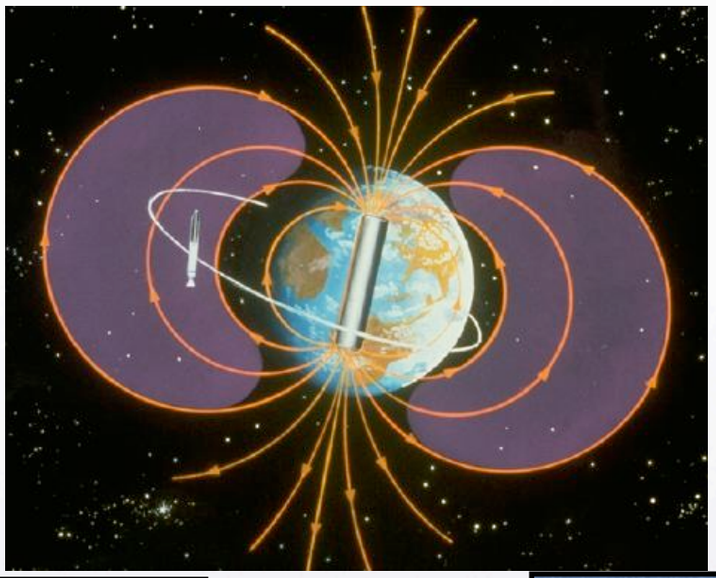


Source slit

^{28}Si

Source slit

^{28}Si



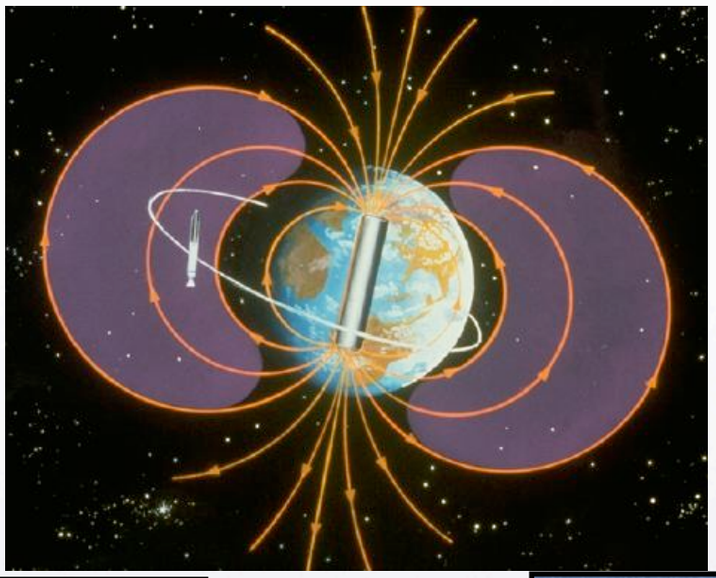
Canberra 35°S
Z -53,000 nT

St Petersburg 60° N
Z +49,800 nT

Source slit

^{28}Si

$^{30}Si^-$



Source slit

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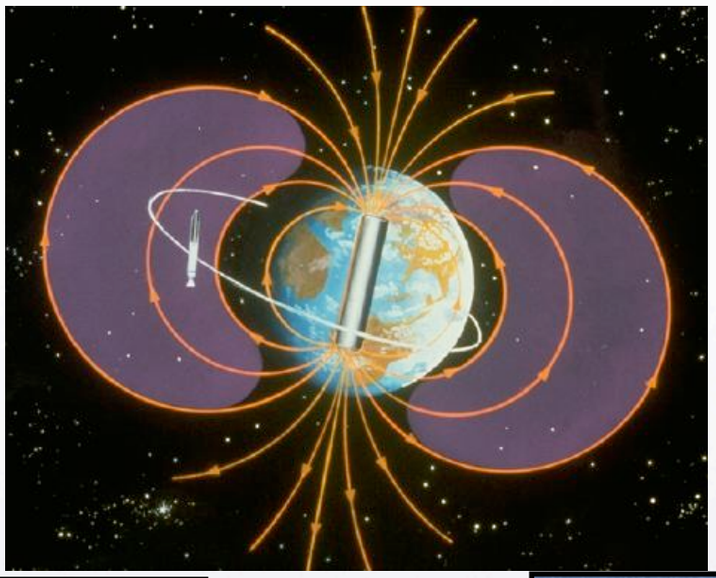


Canberra 35°S

Z -53,000 nT

Source slit

^{28}Si

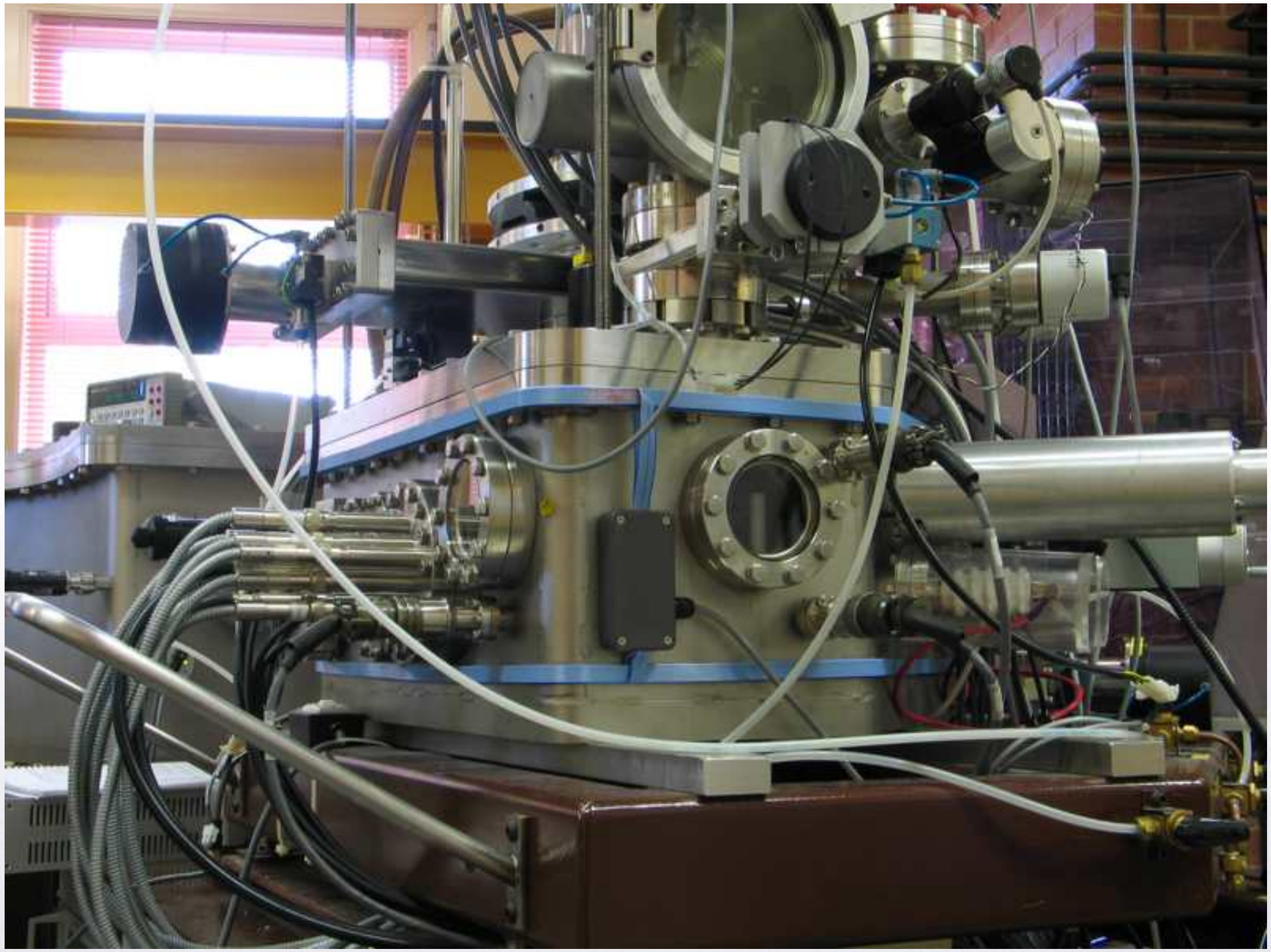
$^{30}Si^+$

$^{30}Si^-$

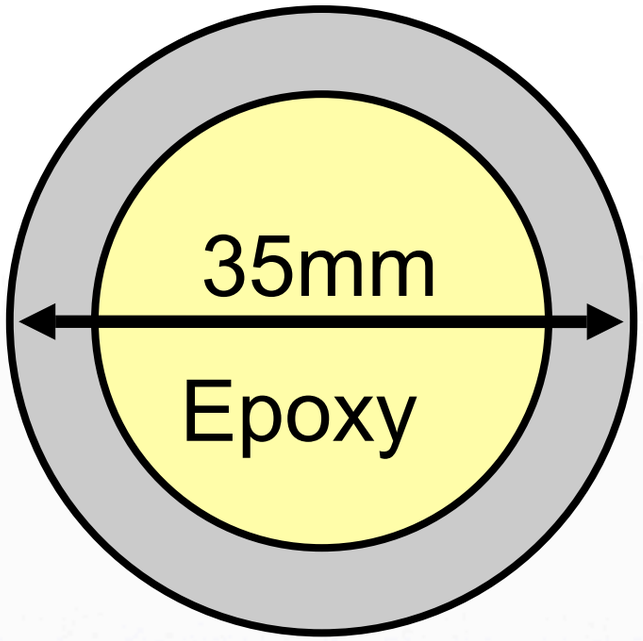
St Petersburg 60° N

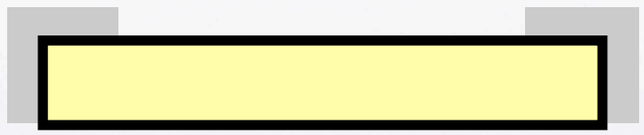
Z +49,800 nT

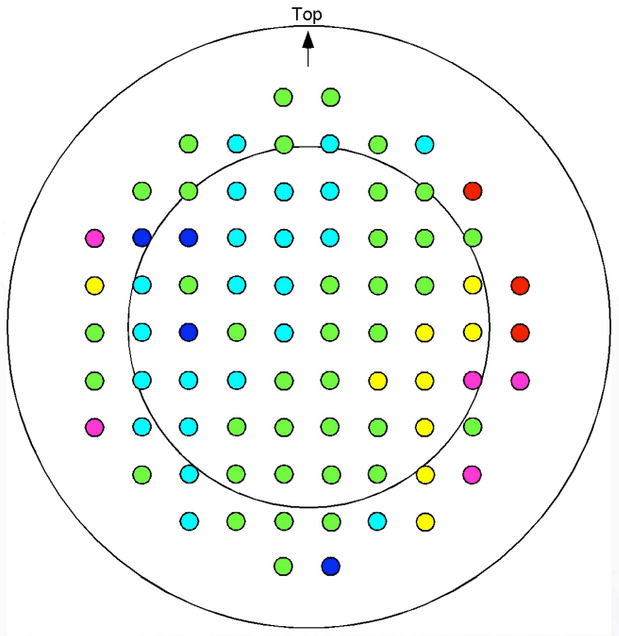
Helmholtz coils



Mount Design





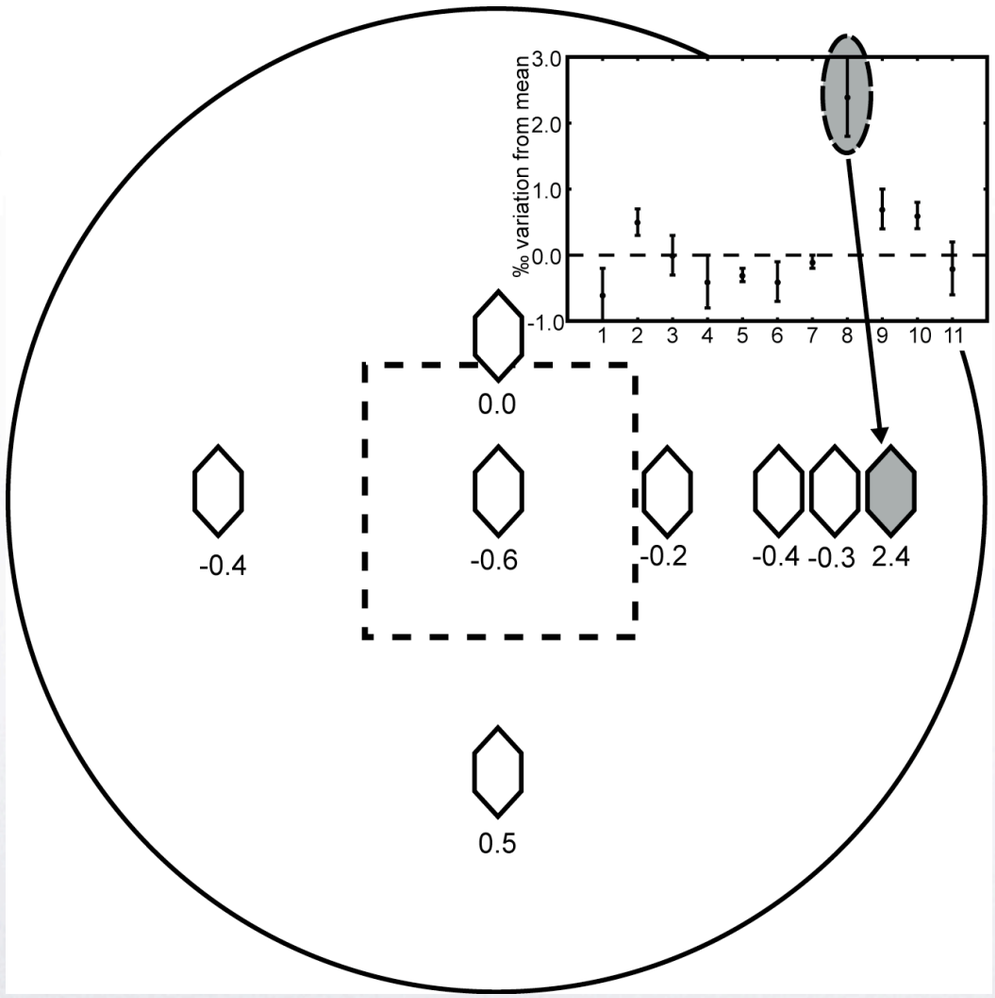


Oxygen isotopes affected by steel rim

Ickert and Hiess


Conventional Mount
Mega Mount

Mega Mount

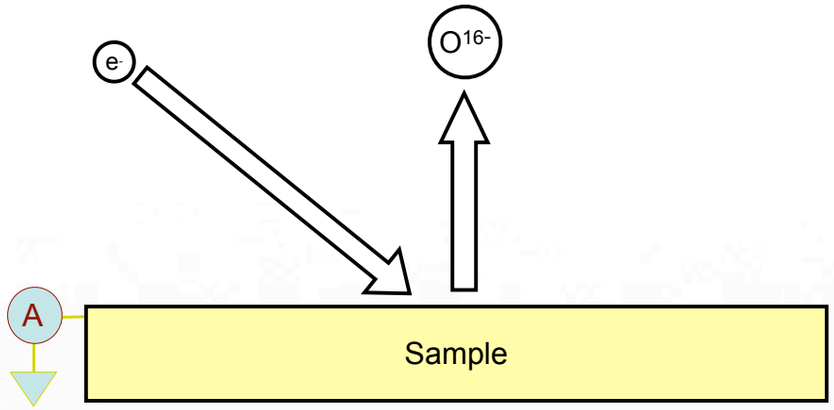


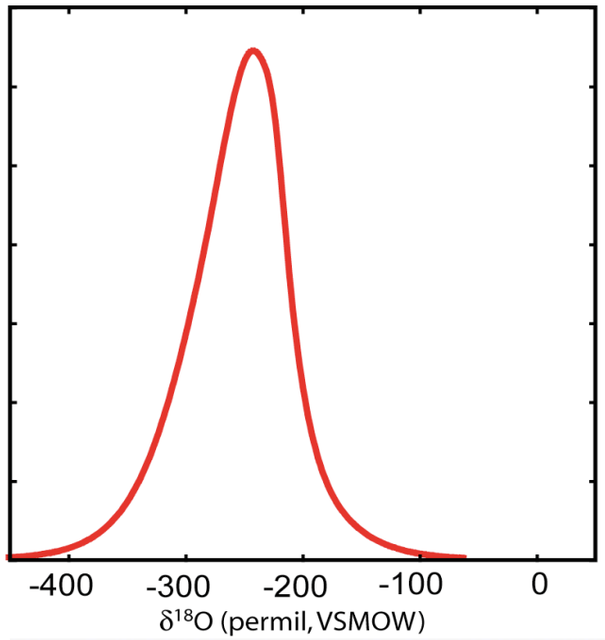
- No fractionation across mount until extraction aperture overlaps the mount edge

- Also favourable for U-Pb reproducibility

- Conductivity contrast on mounts an issue

Electron-Induced Secondary Ion Emission
EISIE

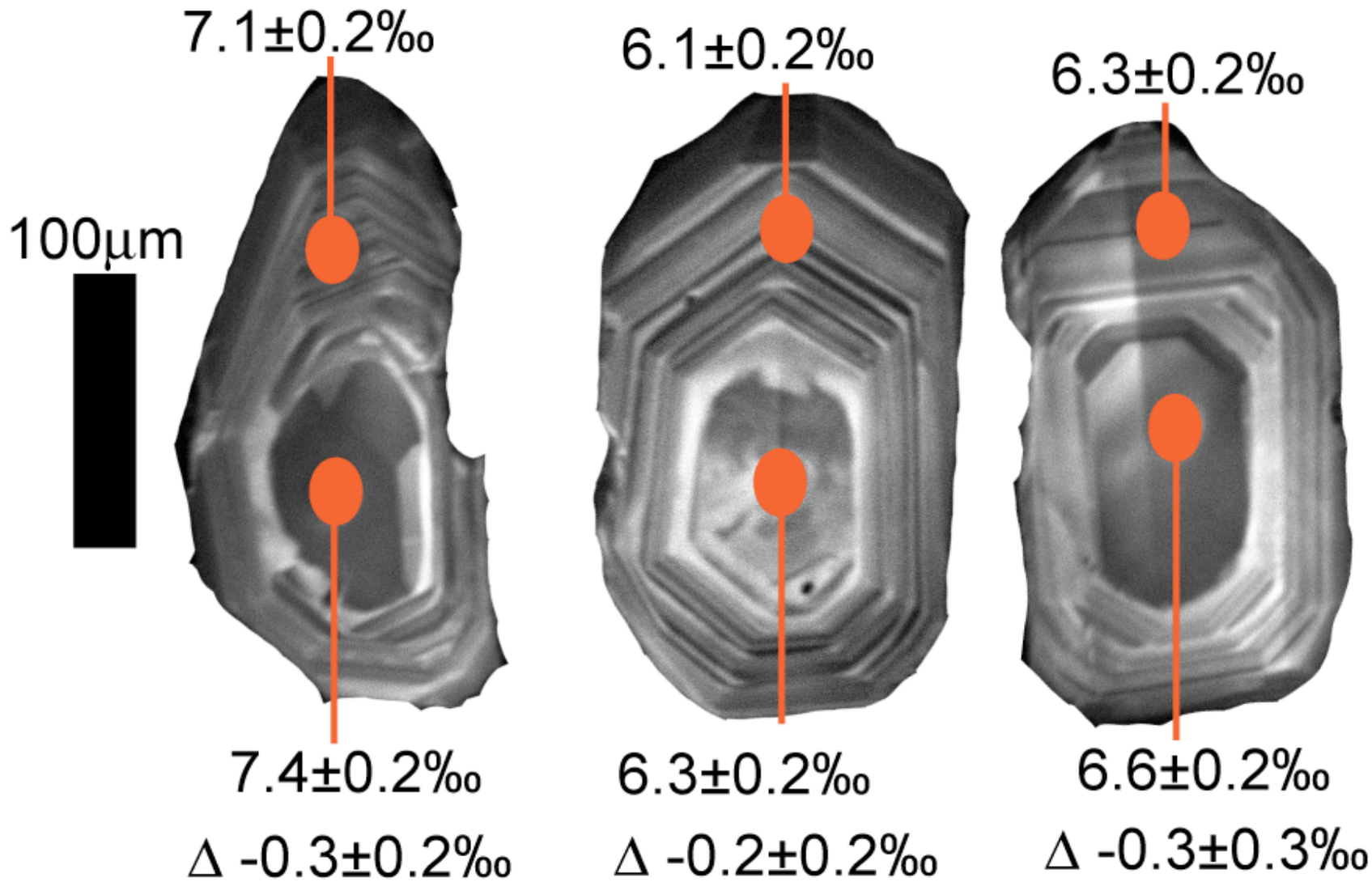




EISIE

- probably related to Cs bombardment
  - gettering vacuum water
  - issue for multiple spots
- much reduced on Al vs Au coats
  - factor of three reduced
  - typically ca. 0.1 ‰ correction

Intra-Grain Measurements



Other Improvements

- Specialised low-field magnet control
- Electrometers
- DVS distributed vacuum monitoring system
- Automation
- Flight tube

Al Fight Tube

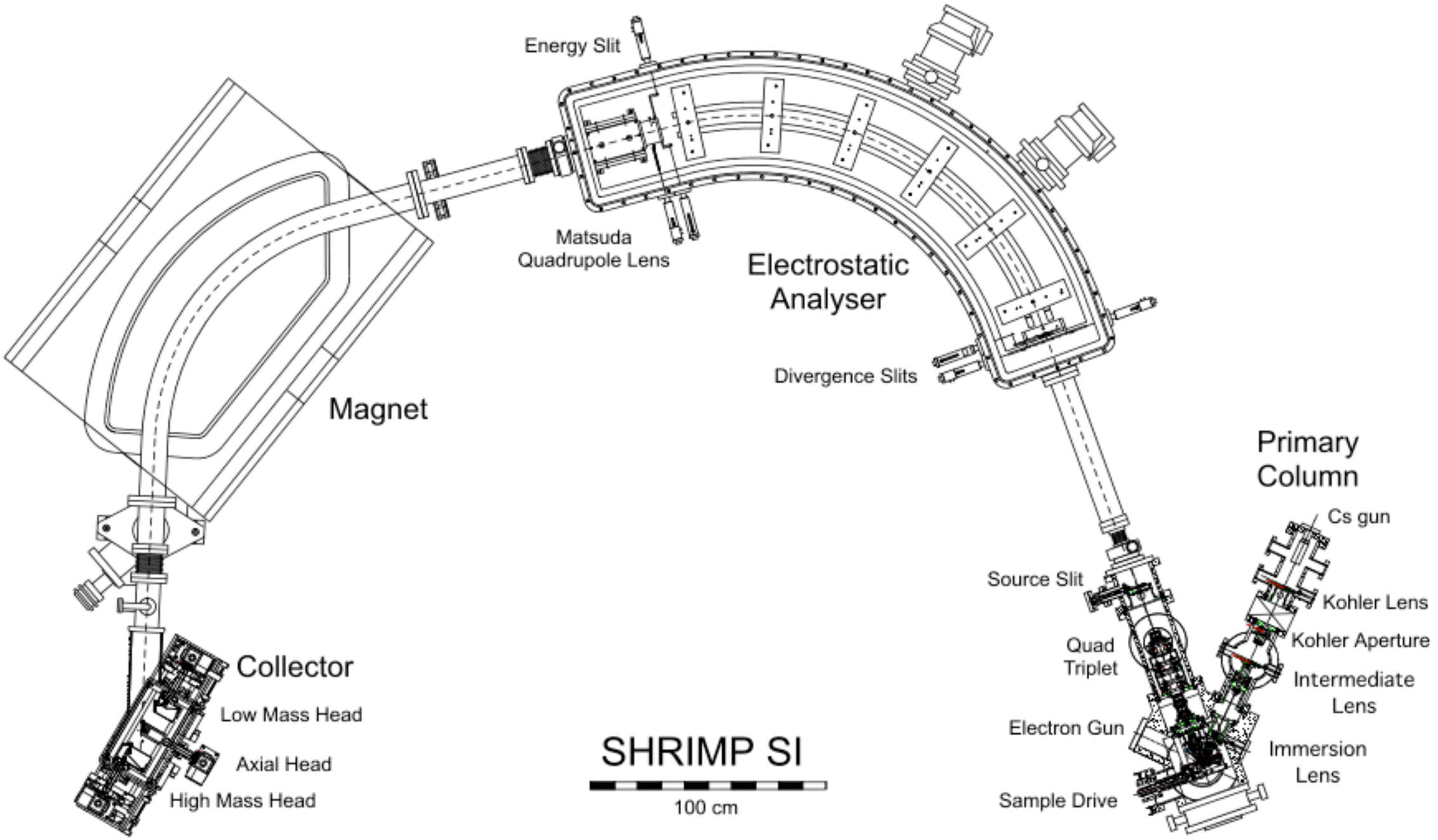
- Noted for some time that magnetic domains are present in 306 stainless steel
- ASI noted irreproducibility in beam refocus
  - tried an Al flight tube
- Al flight tube installed on SHRIMP II
  - greatly improved refocus (lower aberrations)
  - returned magnetic field to theoretical

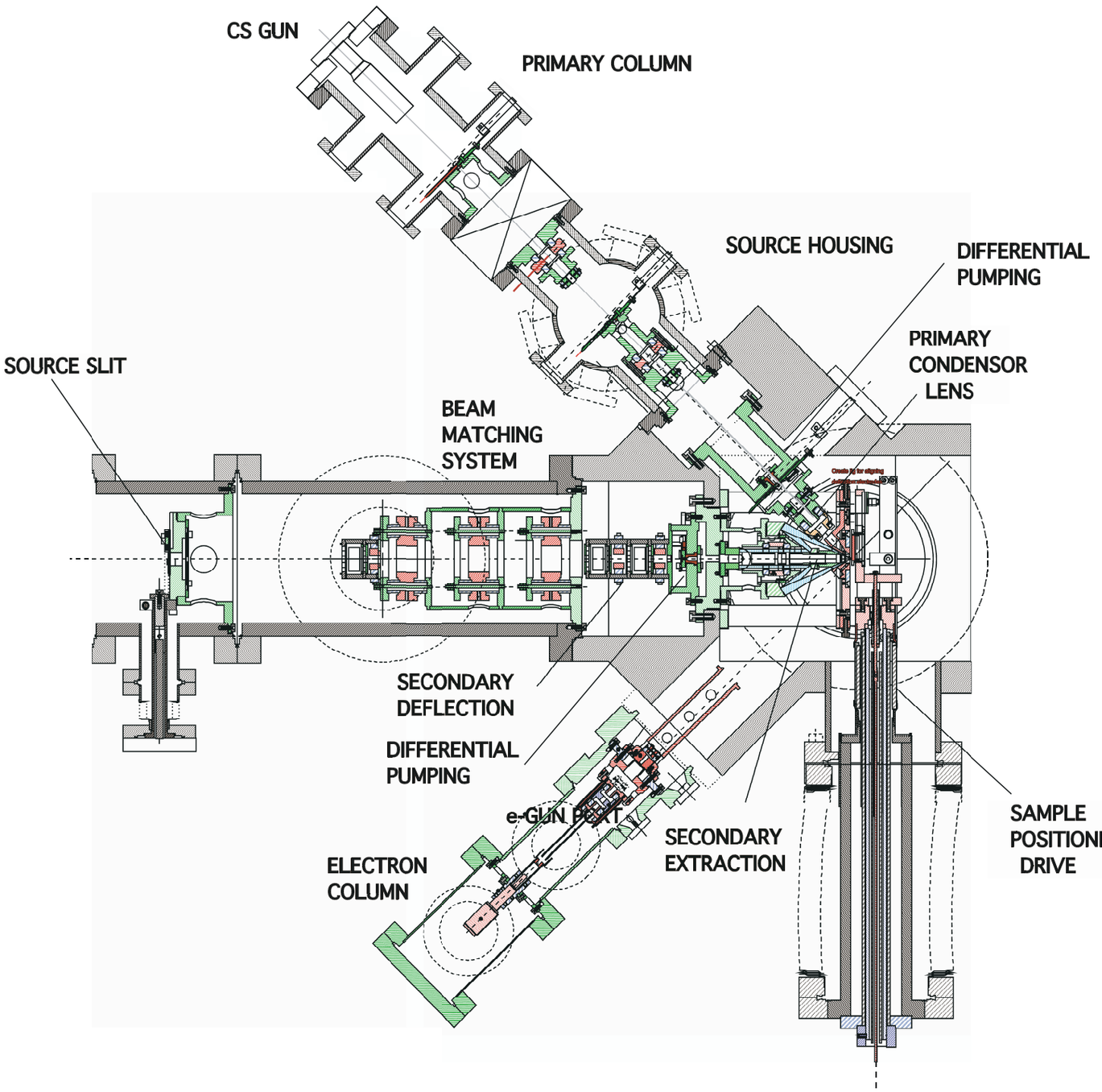
Current Status

- SHRIMP II typically achieves ca. 0.1‰ internal precision and <0.3‰ external, single spot precision for O, S, C
- April-May 2008
  - 4-week stable isotope session; 1 day lost
  - standard calibrations all better than 0.5 ‰
  - both user and automated operation

SHRIMP SI

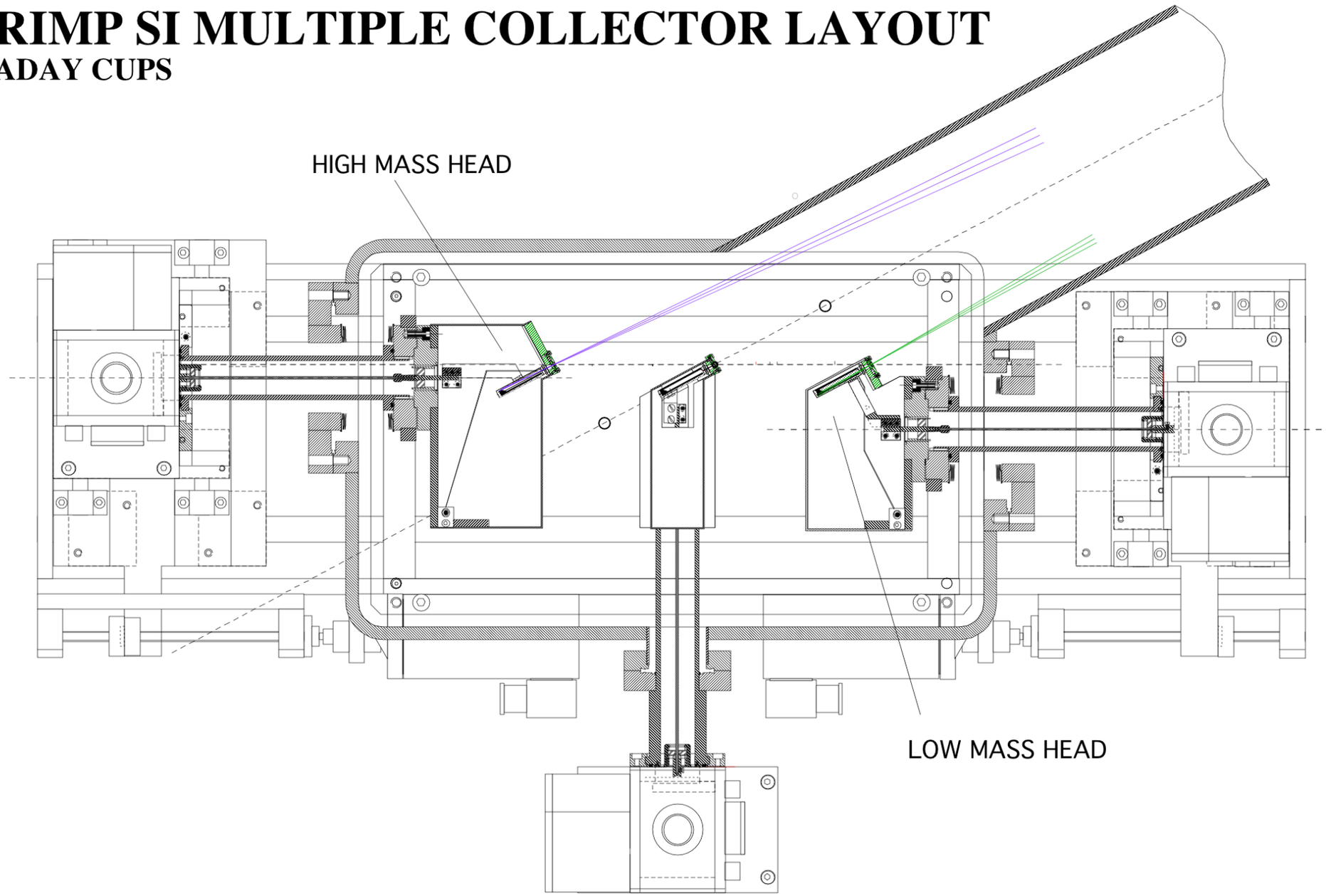
- Dedicated stable isotope instrument
- Improved vacuum
  - reduction in instrumental hydrides
- Smaller spot sizes
  - down to 1 μm
- Simpler multiple collector


Energy Slit
Matsuda
Quadrupole Lens
Electrostatic
Analyser
Divergence Slits
Magnet
Primary
Column
Cs gun
Source Slit
Kohler Lens
Kohler Aperture
Quad
Triplet
Intermediate
Lens
Electron Gun
Immersion
Lens
Sample Drive
Collector
Low Mass Head
Axial Head
High Mass Head
SHRIMP SI
100 cm


CS GUN
PRIMARY COLUMN
SOURCE HOUSING
DIFFERENTIAL PUMPING
PRIMARY CONDENSOR LENS
SOURCE SLIT
BEAM MATCHING SYSTEM
SECONDARY DEFLECTION
DIFFERENTIAL PUMPING
e-GUN PORT
SECONDARY EXTRACTION
SAMPLE POSITIONING DRIVE
ELECTRON COLUMN

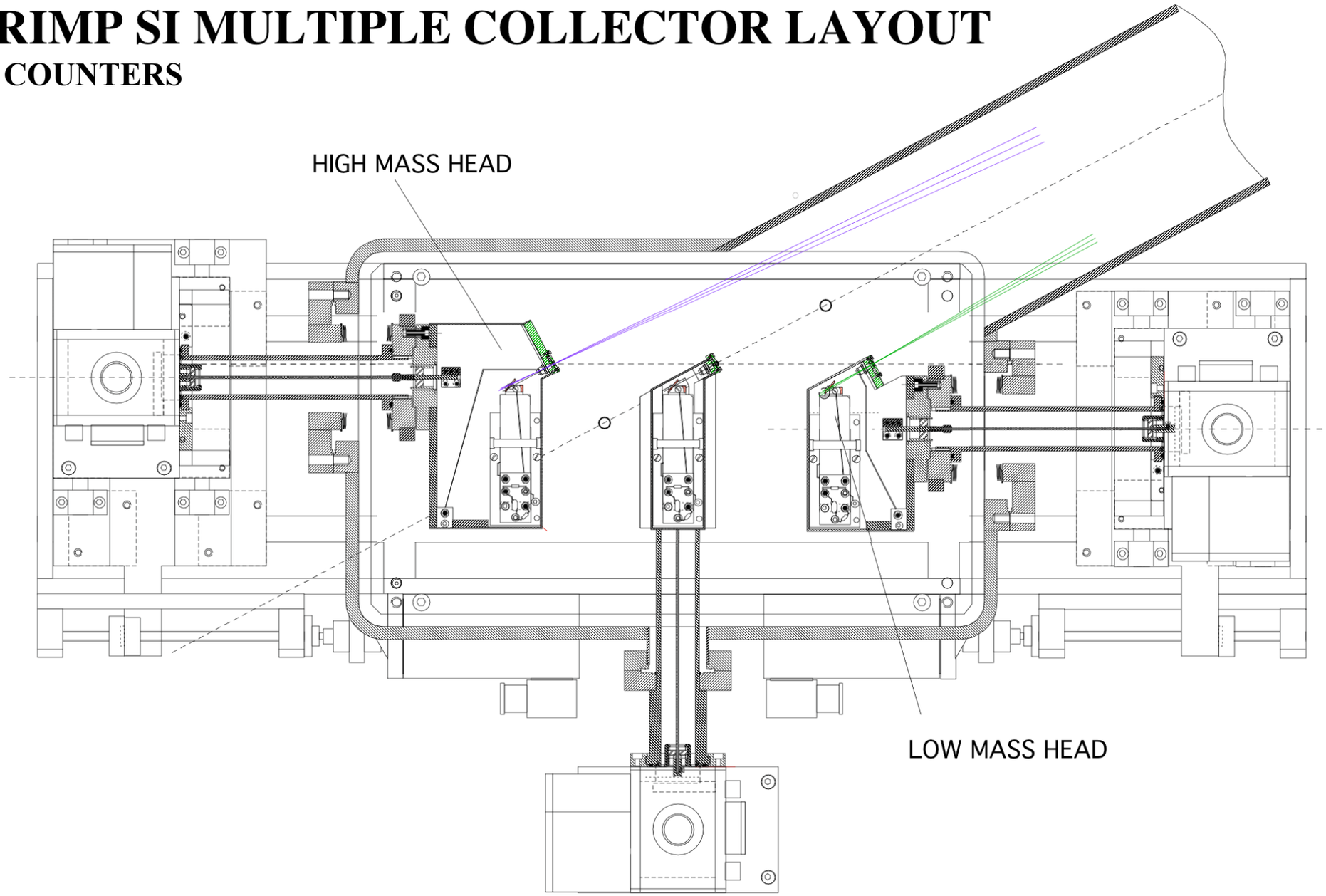
SHRIMP SI MULTIPLE COLLECTOR LAYOUT

FARADAY CUPS



SHRIMP SI MULTIPLE COLLECTOR LAYOUT

ION COUNTERS



SHRIMP SI

- Multiple collector fabrication complete
- Mass Analyzer fabrication complete
- Source and primary 60% complete
- Last machine drawings in by July 2008

Some assembly required

Calendar

- SHRIMP II Stable Isotopes ✔
- J5 Building extension ✔
- Build SHRIMP SI
- Move SHRIMP II from J3