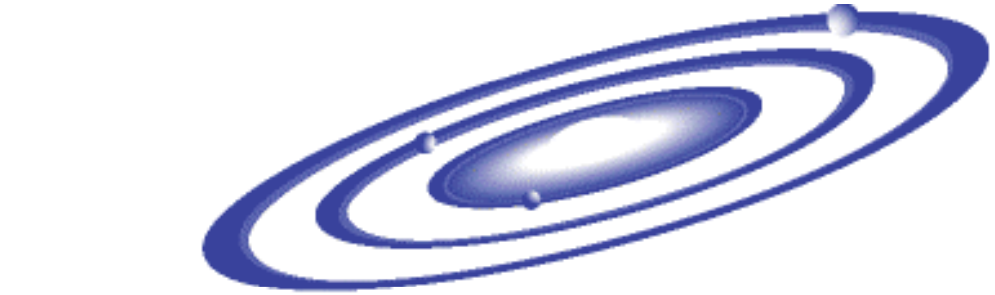
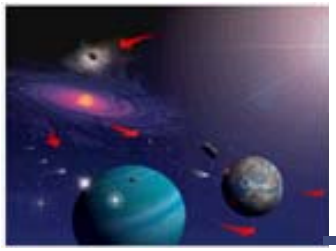


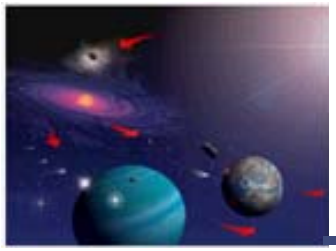
SIMS Update

D.S. Burnett, Amy Jurewicz, R. Hervig, K. Rieck, M.Wadhwa,Y. Guan, D. Woolum, H. Ngo, D. Papanasstssiou, L. Nittler, J. Wang.

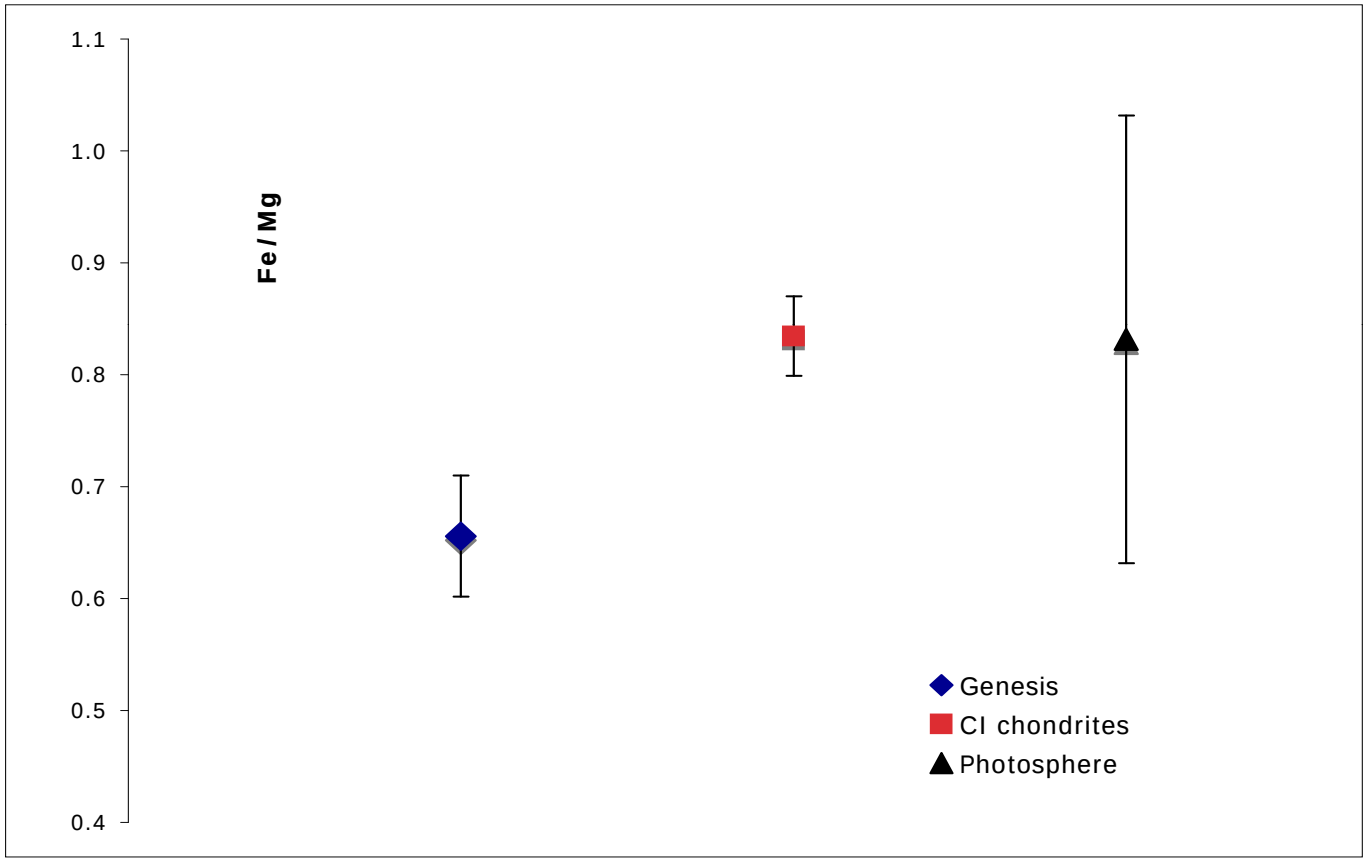


Status: Fe/Mg by SIMS (Jurewicz, Guan, Hervig)

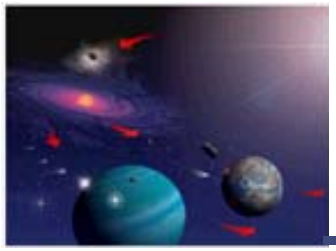
- Details of analyses in Nov. 2007 GPMC.
- Major discrepancy in Mg fluence between Si and DOS (Sandia) when “external” implant standard used.
- Discrepancy eliminated by implanting known fluence of ^{25}Mg as internal standard into flight samples.
- Unlike Mg, good agreement for Fe fluence obtained between two materials.



Compare with CI chondrites

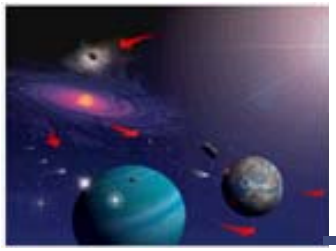


Genesis Fe/Mg, at present, distinct from CI ratio, but systematic errors in implant fluences must be assessed before final conclusions drawn. Goal will be to maintain precision as on figure, but Fe/Mg value could change.



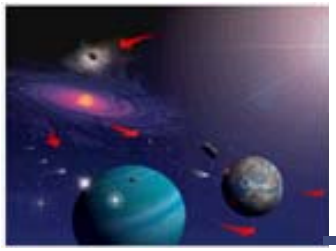
Calibration of Fe implant fluences

- SW fluence data on Sandia (diamond-like-C), SoS, and Si based on 4e13/cm^2 implant.
- Fluence can be independently analyzed on 5e15/cm2 Si implant by isotopic dilution on samples of known area. (ICPMS; FSU; in progress).
  - Measurement of high fluence implant in one material applies to all materials in same implant.
- Measurement of *relative* fluence measurement by SIMS of 5e15 and 4e13 Sandia implants is possible (ASU, CIW, in progress).
- High fluence implant also calibrated by RBS (ASU).
  - RBS data agree well with ion implanter fluence.
  - RBS shows fluence uniformity to better than 1%.



Mg Implant Calibration Plan.

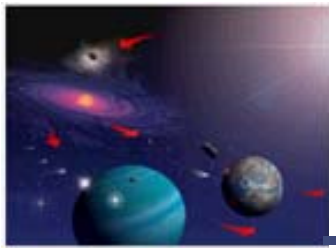
- Mg fluences based on Si and Sandia using K7A (1e12/cm2) 2007 internal standard implants into flight sample.
- Control pieces of Si included in K7A.
- 2007 implant has high fluence 25Mg implant (K7C). Analyze by isotopic dilution. Independent calibrations by TIMS (JPL) and ICPMS (FSU).
- *Relative* K7C and K7A fluences for other implants measured by SIMS
  - Replicate measurements at ASU and CIW have been carried out
  - Complications with dead time in high fluence samples; transient sputtering effects presumably under control.
  - Precision of profilometry pit depths has been an issue, but apparently resolved (following slide)



Resolved issue: Precision of sputter pit depths.

- SIMS solar wind fluences based on concentration depth integral.
- Depth scale requires post-analysis measurement of depth of sputter pit.
- Discrepancy on some of the CIW K7C implant pit depths:
  - Four different instruments have been used. Interferometers (UCLA, CIT) and stylus profilometers (ASU, NIST).
  - Two separate intercomparisons of ASU and UCLA on other samples have given good agreement.
  - A subset of the CIW K7C pits show a systematic 10% difference between the interferometer and stylus profilometer depths.
  - Two different instruments of each kind agree with each other, but the inter-instrument difference remains!
  - When the K7C/K7A fluence ratio calculated using only interferometer or only stylus pits, good agreement is obtained.
    - saved by internal consistency !

	K7A/K7C fluence *ratio* ($\times 10^{-4}$) CIW profiles
Stylus (NIST)	3.51
Interferometer (UCLA)	3.47



ASU-CIW comparison of K7A/K7C relative fluence

- Comparison on previous page involved only different depth scale calibrations for SIMS profiles of implants measured at CIW.
- Comparison of relative fluences from ASU and CIW:

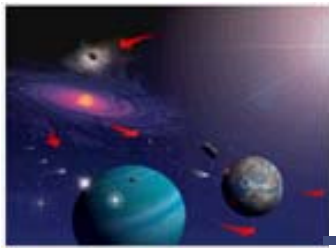
	K7A/K7C relative ^{25}Mg fluence ($x10^{-4}$)
CIW	3.49 ± 0.07
ASU	3.06 ± 0.21

"Error bars" represent total range.

It would be nice to have better agreement than this.

Part of the problem is trying to accurately measure fluences that differ by a factor of 3000.

Plan: Isotopic dilution calibrations of K7C have no blank issues. Isotopic dilution measurement of K7A difficult but if blanks good, may be possible to measure K7A directly.



Calibration of K7C (3e15) implant with TIMS

Ngo, Papanasstassiou, JPL/CIT

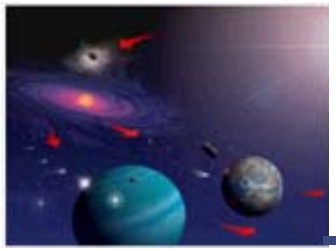
Plan: Strip off implanted layer (0.5 μ for K7C) with HNO3/HF.

Obtaining uniform, controlled etching difficult, but

(HNO_3 / HF / H_2O) = (125 / 5 / 80)

gives good results.

(see us for details which are important)



K7C TIMS: Procedures are important

Approx 1 cm^2 K7C samples (Jurewicz)

areas measured accurately in A. Westphal lab (Woolum)

1. **uvO3 cleaning at JSC to remove implant hydrocarbons.**
2. **Dilute (5%) HF to remove most SiO_2.**

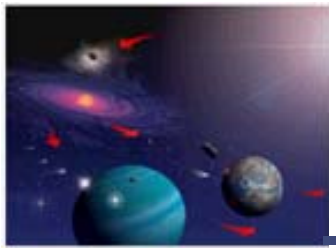
Steps 1 and 2 to make HNO_3/HF etching uniform.

3. **HNO3/HF etching**

   follow weight loss; few microns on average removed.

4. **Repeat step 3 to check for complete extraction.**
5. **Spike etches and rinses with ^{26}Mg.**

No implant ^{25}Mg measured in three separate, step 4 re-extracts.

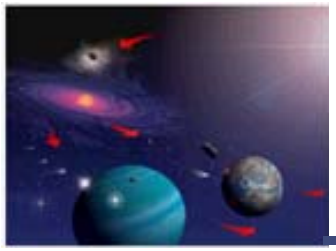


Results

Nominal K7C fluence: 2.99 e15/cm^2

Sample	^{25}Mg fluence(e15)	Percent Recovered
K7C-5B	2.1	69
K7C-5C	1.9	62

5B - 5C fluence difference probably significant.



Interpretation

Replating ? $Si + Mg^{++} \rightarrow Mg + Si^{+4}$

Contradictory previous data on whether this happens.

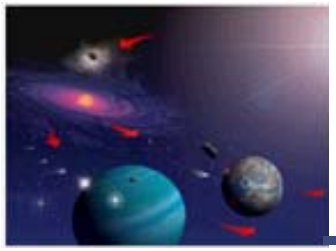
Not expected from EMFs (Munir)

Second re-extract of 5C with spike added to HNO_3 / HF; three times longer etch:

- **No measurable ^{25}Mg.**
- **No evidence for replating.**

?????? Hard to believe that fluence low by 40%

Fe looks OK from RBS.

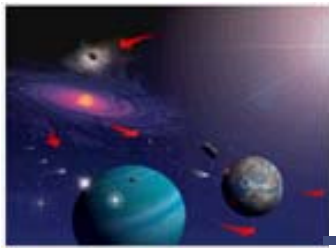


Another Complication

HF etch of 5C: 0.03 e15 $^{25}Mg/cm^{2}$.

Only 0.08% but shouldn't happen.

- **K7C implant is deep; could be more important for flight samples.**
- **Needs to be checked out.**



Solar Wind Mg Isotopes (Jurewicz, Hervig, Rieck, Wadhwa)

Mg isotopes provide a better test for isotopic differences between photosphere and solar wind if an assumption is made:

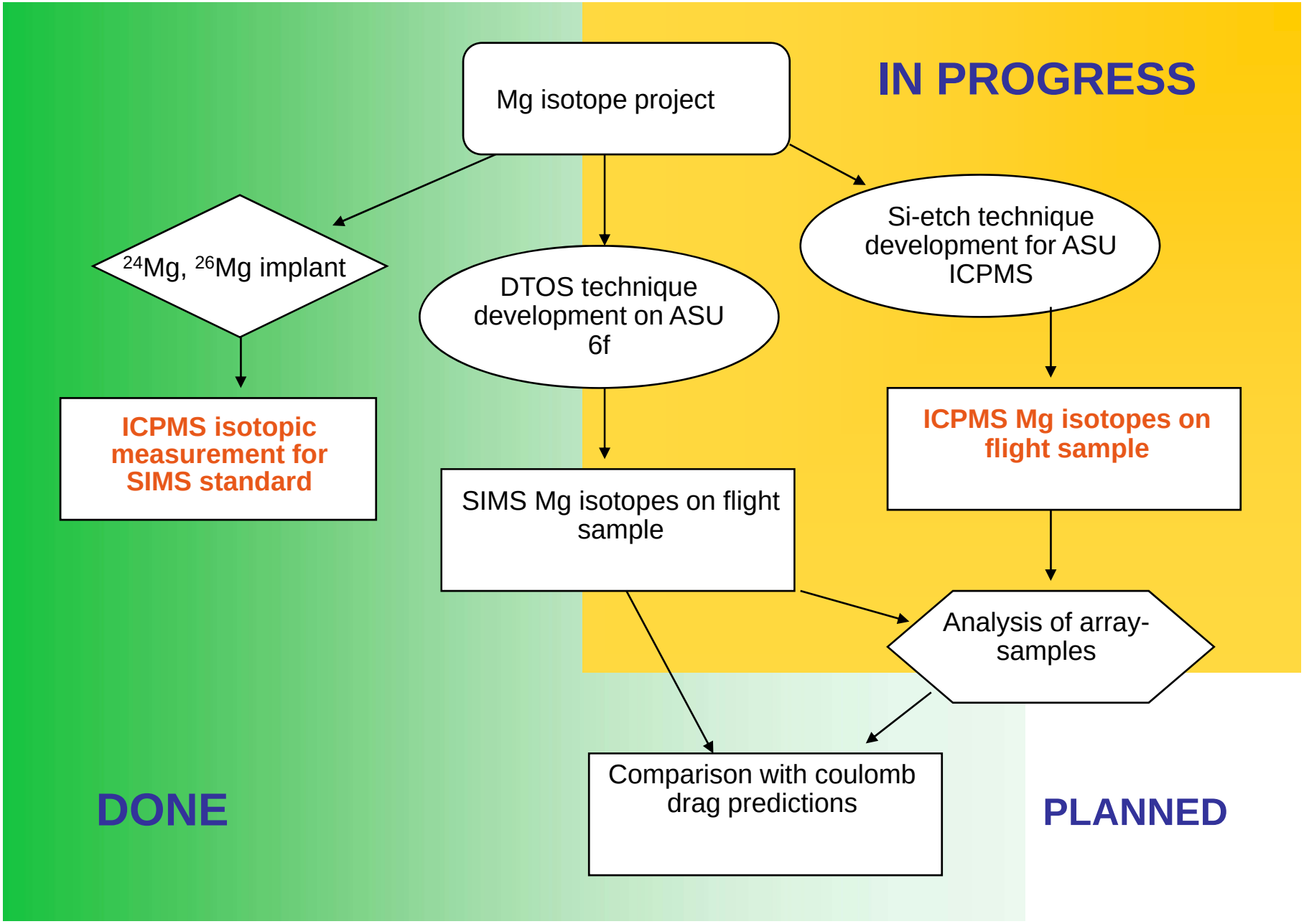
With exception of evaporation effects in CAIs, no systematic mass dependent fractionations for non-volatile elements greater than about 1 per mil / amu.

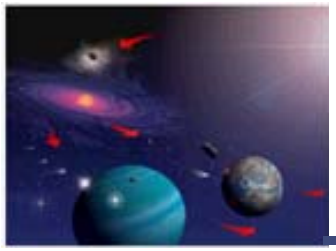
If this assumption valid for Mg, then comparison of Earth and Solar Wind is comparison between photosphere and solar wind.

For noble gases, have no apriori knowledge of photospheric isotopic compositions.

Bochsler Coulomb Drag model predicts light isotope enrichment: -19 permil/amu for Mg.

ASU Experimental Approach and Progress





SIMS mass fractionation correction

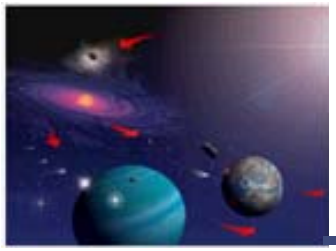
"Srni" implant: separate ^{24}Mg, ^{26}Mg implants with 26/24 =0.100

High 24 fluence, 3e15/cm^2

ICPMS calibration (Wadhwa) of $(26/24)_{implant}$ possible:

0.09989, in good agreement with implanter intgrated current ratio.

- **Kroko relative fluences appear good.**



Dead time corrections

For SIMS, high ^{24}Mg fluences in Srni implant creates dead time problems:

Correction procedure (Hervig):

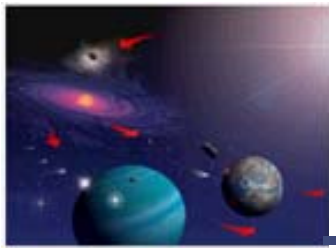
Take data using large raster (250 microns) and dynamic transfer gating: 60% of raster size.

Using low current and ^{28}Si, measure ratio (2.2) of cps in 60% gate compared to total cps (100% gate).

For cps 24 in SRNI implant, use 2.2 factor to scale measured cps to total cps, then calculate deadtime using known (37 ns) instrument dead time constant.

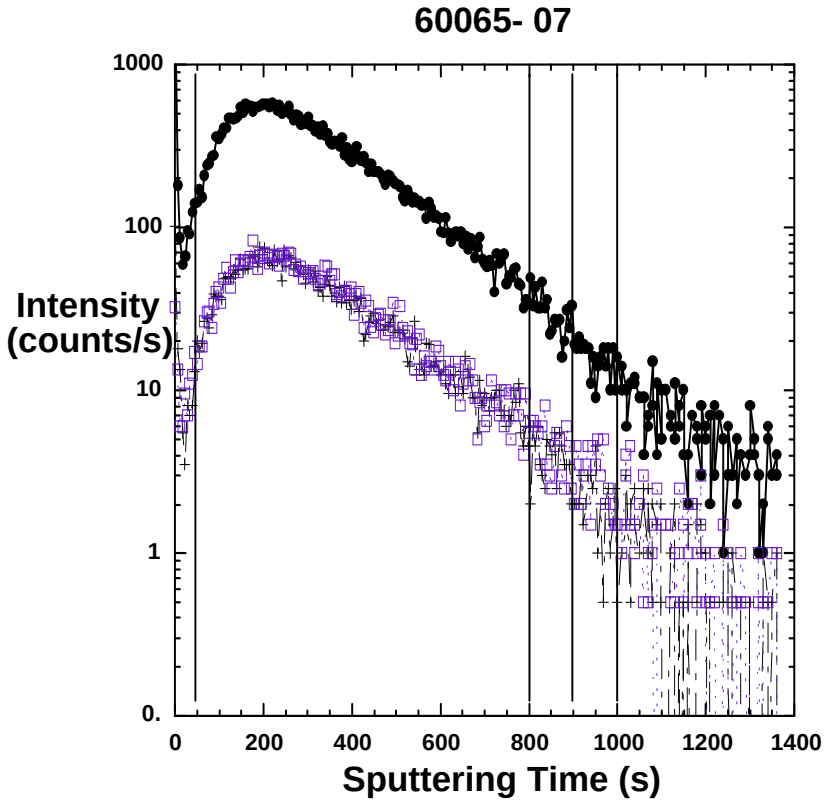
At peak of 24 implant, dead time corrections around 8%; average over profile much less.

Good agreement (< 1 permil/amu) in mass fractionation from separate profiles.



Sandia Flight Sample 60065

Si analysis requires O_2 flood which defocuses beam, so use diamond-like-C where transient sputtering effects small and O_2 flood not necessary.

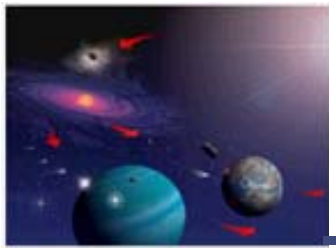


Sufficient mass resolv. power to rule out contributions from C_2^+

Solar wind = cons. v ions; Implant = cons E ions.

In both cases, depth profiles differ for isotopes; calculate ratios of integrated profiles.

Effect of choice of ranges for integration not important.



Very Preliminary Results for 60065

- Making estimates for errors in corrections, precision for both δ^{25}Mg and δ^{26}Mg around 6 per mil (1 sigma)

- δ^{25}Mg and δ^{26}Mg are both negative outside of error with a constant permil/amu mass fractionation.
- No evidence for ^{26}Al effects at mass 26.
- Sign and magnitude of Mg variations consistent with Bochsler Coulomb Drag Model.
- Fractionations between photosphere and solar wind would be bigger for O.
  - Bochsler predicts 60 per mil.