OXYGEN ISOTOPE MEASUREMENTS OF STARDUST INTERSTELLAR ANALOGS A. J. Westphal[1], S. M. Jones[2], R. C. Ogliore[3], K. Nagashima[4], G. R. Huss[4], A. L. Butterworth[1], C. E. Jilly-Rehak[1], J. Vasquez[1]. [1]Space Sciences Laboratory, University of California Berkeley, Berkeley, CA 94720, USA, [2]Jet Propulsion Laboratory, Pasadena, CA 91220,[3]Laboratory for Space Sciences and Physics Department, Washington University in St. Louis, St. Louis, MO 63117, USA, [4]Hawai'i Institute of Geophysics and Planetology, University of Hawai'i at Mānoa, Honolulu, HI 96822, USA.

Introduction: With the discovery of extraterrestrial materials of possible interstellar origin in the Stardust Interstellar Dust Collector, NASA now has an eighth returned sample collection[1], after the Apollo, Antarctic meteorite, Cosmic Dust, Genesis, Stardust Cometary, Hayabusa and Exposed Space Hardware collections. ***Samples from the Stardust Interstellar Collector present unprecedented challenges, and are vastly more difficult to prepare and analyze than Stardust cometary samples.*** Specifically:

- Particles are of order 1 pg in mass, $<10^{-3}$ of the mass of typical particles in the other small-particle collections (Stardust Cometary, Cosmic Dust, and Hayabusa).
- The total number of interstellar particles near 1 μm in size is probably <10.
- Unlike the Stardust cometary collection, the background is large: for each track containing extraterrestrial material, there are $\sim$30 tracks due to secondary ejecta.
- Distinguishing signal from background nondestructively requires synchrotron-based x-ray microprobes, which are heavily oversubscribed.

During the Stardust Interstellar Preliminary Examination (ISPE)[1, 2], we identified three impacts in aerogel and four impacts in the aluminum foils which were consistent either with an origin in the interstellar dust stream, or with an origin as secondary ejecta from impacts on the spacecraft. Two of the projectiles in aerogel were captured at sufficiently low speed that the projectiles survived: olivine and spinel were identified in one, and olivine was identified in the other. An origin as secondary ejecta or as interplanetary dust is statistically unlikely, but isotopic measurements are required to definitively test for interstellar origin. These measurements were not allowed under the rules of the ISPE.

Methods: Interstellar dust particles in the 1 μm size range penetrate approximately 100 μm into the aerogel collectors. To simulate aerogel collectors which have captured interstellar dust particles, we synthesized several aerogel tiles in which a layer of spinel separates from the Allende meteorite were included in a single layer several hundred μm below the surface. Most of these spinel grains are ^{16}O-rich, and lie on the CCAM line at approximately (δ^{18}O, δ^{17}O) = (-40‰, -40‰). Sample preparation and systematic analytical errors during isotope measurements may cause measured isotope ratios to differ from the true values in a mass-dependent or mass-independent way. The accuracy and precision of analog spinel separate measurements will help us to identify our sources of error, which can then be addressed in the next iteration of analog sample preparation and analysis, in order to achieve the ideal protocol to measure the Stardust interstellar candidates.

We extracted spinel particles in aerogel keystones[3]. We embedded one of these keystones in epoxy on an epoxy bullet, and microtomed the keystone using a diamond knife, exposing one of the spinel grains. We then mounted the bullet on a standard "buckler" mount[4], and analyzed the sample on the Cameca ims 1280 ion microprobe at the University of Hawai'i. In parallel, we pressed another keystone into In, using a teflon-coated press, at sufficiently high pressure that the resulting compressed aerogel was conductive after a carbon coat (Figure 1). (This procedure was also used for the sample preparation on the bulb of track 184, reported in these proceedings[5].)

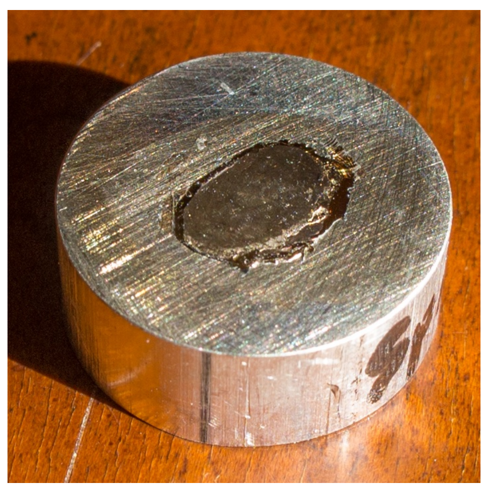

Figure 1: Spinel keystone pressed into indium, mounted in a 1-inch aluminum round.

We used a <3 pA Cs^+ primary beam focused to $\sim$250 nm, and acquired 10$\times$10 μm, 128$\times$128 pixel scanning ion images of the spinel potted butt (Figure 2). An electron flood gun was used for charge compensation. We simultaneously collected $^{16}O^-$, $^{17}O^-$, and $^{18}O^-$ on electron multipliers. We used a mass-resolving power of 5500 on $^{17}O^-$ to minimize the interference from $^{16}OH^-$. We collected 400 total frames (220 minutes of measurement). We measured San Carlos olivine under identical conditions, and from this measurement calculated a correction that was applied to the $\delta^{17}O$ and $\delta^{18}O$ measured values for the spinel grain.

The keystone containing spinels pressed into indium was measured under similar conditions, except $^{24}Mg^{16}O^-$, $^{27}Al^{16}O^-$, and $^{56}Fe^{16}O^-$ were also collected by magnetic-field peak jumping in order to easily identify the spinel grains embedded in aerogel. We used a larger measurement raster collected for a longer time. The grain we measured was 3.9 μm in size. The raster was increased to 25×25 μm and 872 frames were collected for this particle (a total measurement time of 14.5 hours).

Figure 2: Scanning ion image of ^{16}O from the spinel grain. Image is 10×10 μm.

Results: The spinel grain mounted in the potted butt was measured to have O isotopic composition: $\delta^{18}O$ = 20.3±3.3, $\delta^{17}O$ = 10.0±7.5 (all errors are 2σ). The spinel grain in aerogel pressed into indium was measured to have O isotopic composition: $\delta^{18}O$ = 20.9±12.7, $\delta^{17}O$ = 14.3±28.8. The larger uncertainties for the spinel mounted in indium was due to the larger raster used in this measurement. A larger raster was used to find spinel grains more efficiently; a smaller raster was used on the potted butt because the location of the spinel grain was precisely known.

Conclusions: Both particles were found to have oxygen isotopic compositions consistent with terrestrial spinel. Some spinels have been reported in ordinary chondrites that are close in composition to terrestrial spinel ($\delta^{18}O$, $\delta^{17}O$) = (+22.3‰, +11.6‰) [6] (presumably from ^{16}O-poor chondrules), but we found no reports in the literature of such spinels separated from Allende (which are mostly from ^{16}O-rich CAIs).

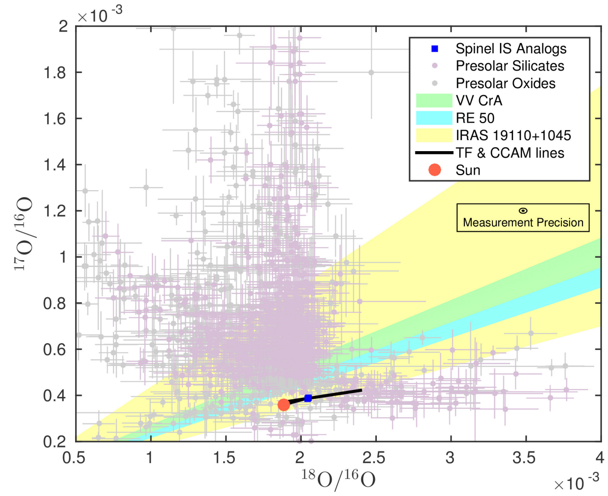

Figure 3: Oxygen isotope ratios of IS analog spinels reported here compared to presolar silicates and oxides, three young stellar objects in the Sun's galactic neighborhood, the Sun, and the range of Solar System materials. Our 2σ measurement precisions of the two spinels reported here are shown by the two black ellipses.

Although a measurement of oxygen isotopic composition is probably necessary to confirm an interstellar origin for particles captured in aerogel in the Stardust Interstellar Dust Collector, the measurement is not required to have high accuracy or precision. It is known from astronomical observations that the Solar System is strongly anomalous in oxygen: $^{18}O/^{17}O$ = 5.2 in the Solar System, while $^{18}O/^{17}O$ = 4.1 ± 0.1 in the local galaxy[7] (Figure 3). This difference in $\Delta^{17}O$ of about 250‰ should be a smoking gun for interstellar origin. Even if the actual composition of two spinels that we measured here were ($\delta^{18}O$, $\delta^{17}O$) = (−40‰, −40‰), which we think is unlikely, the corresponding systematic error in $\Delta^{17}O \approx 25$‰ is about an order of magnitude smaller than the difference expected between the local interstellar and the Solar System values. Nevertheless, care must be taken to be sure that the signal is not diluted significantly by surrounding aerogel (and epoxy, in the case of the epoxy embedding process), particularly since the statistical precision of the measurement will be compromised by small counting statistics. We conclude that further measurements are necessary to establish the reliability of either technique.

References: [1] A. J. Westphal, et al. (2014) *MAPS* 49(9):1720. [2] A. J. Westphal, et al. (2014) *Science* 345:786. [3] A. J. Westphal, et al. (2004) *MAPS* 39(8):1375. [4] A. J. Westphal, et al. (2011) *MAPS* 74:5274. [5] R. C. Ogliore, et al. (2016) *47th LPSC* #1721. [6] K. Makide, et al. (2009) *40th LPSC* #2079. [7] J. Wouterloot, et al. (2008) *A&A* 487(1):237.