IDENTIFICATION OF PRESOLAR SPINEL GRAINS FROM A MURRAY RESIDUE BY MULTI-DETECTION RASTER IMAGING. A. Nguyen[1], E. Zinner[1], and R. S. Lewis[2], [1]Laboratory for Space Sciences, and the Physics Department, Washington University, St. Louis, MO, 63130, USA (nguyen@levee.wustl.edu), [2]Enrico Fermi Institute, University of Chicago, Chicago IL 60637 USA.

Introduction: Grain size separate CG from the Murray CM2 carbonaceous chondrite contains mostly spinel grains of average diameter 0.5 μm [1]. Zinner et al. [2] found that approximately 1% of these spinel grains are of presolar origin as determined by their large O isotopic anomalies. These O isotopic measurements were made with the NanoSIMS on individual grains that were well separated from one another on a gold foil. The grains were selected for analysis from secondary electron and secondary $^{16}O^-$ images. The primary beam was then successively deflected onto these single grains for O isotopic analysis.

While single grain analysis on dispersed samples is effective for finding relatively abundant anomalous grains, ion imaging in a raster mode on tightly packed grains might be more efficient for locating few anomalous grains among predominantly isotopically normal grains. In fact, this was the analysis mode used by Messenger et al. [3] to discover presolar silicates in interplanetary dust particles. In an exploratory effort that is also geared toward establishing the optimum isotopic imaging technique in the search for presolar silicate grains in primitive meteorites, we measured O isotopic ratios in spinel grains from the Murray CG separate by raster imaging of areas with more or less tightly packed grains.

Experimental: Measurements were made on the same mount on which we had previously measured individual spinel grains [2]. CG grains had been deposited onto a gold foil from suspension in a liquid. While in most areas of the deposits the grains are well separated, some areas contain grains that are packed fairly tightly and almost completely cover the gold. Measurements were made with the NanoSIMS ion microprobe. A Cs^+ beam of ~100nm diameter was rastered over a 20x20 μm^2 area. Secondary ions of the three O isotopes as well as $^{24}MgO^-$ and $^{27}AlO^-$ ions were simultaneously counted in multi-detection in five small electron multipliers. MgO and AlO were detected for the identification of spinel grains. Either 20 or 40 sequential 256x256 pixel images were acquired as the primary ion beam removed less than 0.1 μm during a whole measurement, which lasted either 3.5 or 7 hours, respectively. We obtained six such images from different areas.

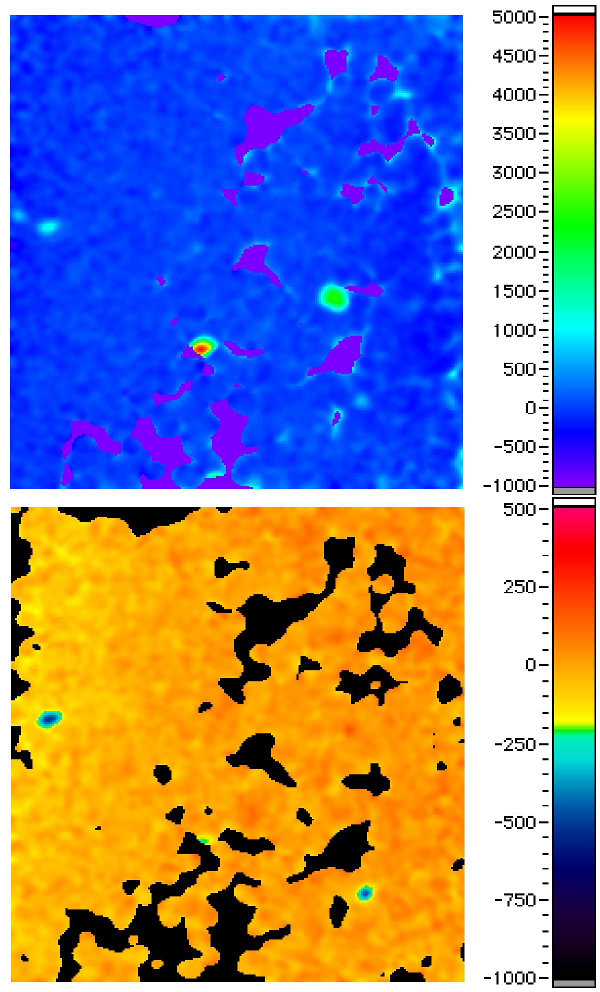

Figure 1. Images of the $^{17}O/^{16}O$ (top) and $^{18}O/^{16}O$ (bottom) ratios of a 20×20 μm^2 area partially covered with Murray CG grains. The ratios are expressed as δ-values, deviations from normal ratios in permil (‰). The images show small-scale variations of the ratios due to statistical fluctuations. However, several grains have much larger anomalies and can clearly be identified. Areas with δ-values of –1000‰ are regions with low (no) grain coverage that were excluded from analysis.

Isotopically anomalous grains were identified from isotopic ratio maps derived from the integrated images of all 20 (40) layers. These maps display the deviation of an isotopic ratio ($^{17}O/^{16}O$ or $^{18}O/^{16}O$) from the average ratio of a given image in permil. Figure 1 shows δ-value maps of the $^{17}O/^{16}O$ and $^{18}O/^{16}O$ ratios for one of the analyzed areas. Areas containing candidate grains were defined manually and the ratios and statistical errors for these grains computed. In addition to isotopically anomalous grains we also selected isotopically "normal" areas of approximately the same size of ~5-7 pixels diameter, corresponding to a diameter of 0.4-0.5 µm, for calibration. For this calibration we assumed these grains to have O isotopic compositions of $\delta^{17}O = \delta^{18}O = -50‰$, the O isotopic composition of spinel in CM2 carbonaceous chondrites [2, 4]. Grains were considered to be of presolar origin if their isotopic ratios were clearly distinct from those of the distribution of the "normal" grains that were used for calibration [2].

Results and discussion: In all six analyzed areas we identified a total number of 40 presolar grains. The isotopic ratios of the anomalous grains are shown in Fig. 2 together with those of isotopically normal CG grains used as standards and of the 9 presolar spinel grains from Murray CG that had previously been identified from the analysis of individual grains [2]. As can be seen, the isotopic ratios cover the range of the previously analyzed CG grains and are in general agreement with O isotopic ratios found in presolar spinel and corundum grains [2].

We estimate the average sample coverage in the six imaged areas to be ~49%. If we make the simplified assumption that all grains are 0.5µm in diameter and are uniformly tightly packed in the covered areas, we arrive at a number of ~4700 measured grains. This results in an abundance of 0.85% presolar oxide grains among all CG grains. In comparison, Zinner et al. [2] found the Murray CG residue to contain 1.2% presolar spinel. However, the previous result was based on 9 presolar spinels out of 753 analyzed CG grains and, within statistical uncertainty, the two results are in agreement with one another.

However, we should add that the two methods for identifying isotopically anomalous grains and determining their O isotopic ratios cannot necessarily be expected to yield identical results with respect to the fraction of anomalous grains and the exact isotopic ratios. Whereas during individual grain analysis only the measured grain contributes to the determined isotopic ratios, during raster imaging of tightly packed grains neighboring (normal) grains will unavoidably contribute to the isotopic ratios attributed to the anomalous grains and dilute the magnitude of their anomalies. We have not yet optimized the measurement conditions and the image processing methods for locating isotopically anomalous grains by raster isotopic imaging but even then there will be limitations in deriving absolute values for the abundance of presolar grains. However, it has already become clear that isotopic imaging of densely packed grains proves to be an effective means for analyzing large numbers of grains and for identifying presolar grains, especially if these presolar grains have very low abundance among the analyzed grains. A little more than a year ago only 7 presolar spinel grains had been identified; in contrast, in the present study we located another 40 such grains.

Acknowledgements: This work was supported by NASA grant NAG5-11545.

References: [1] Tang M. and Anders E. (1988) *Geochim. Cosmochim. Acta* 52, 1235-1244. [2] Zinner E. *et al.* (2003) *Geochim. Cosmochim. Acta,* submitted. [3] Messenger S. *et al.* (2002) *Science,* submitted. [4] Clayton R. and Mayeda T. (1984) *Earth Planet. Sci. Lett.* 67, 151-161.

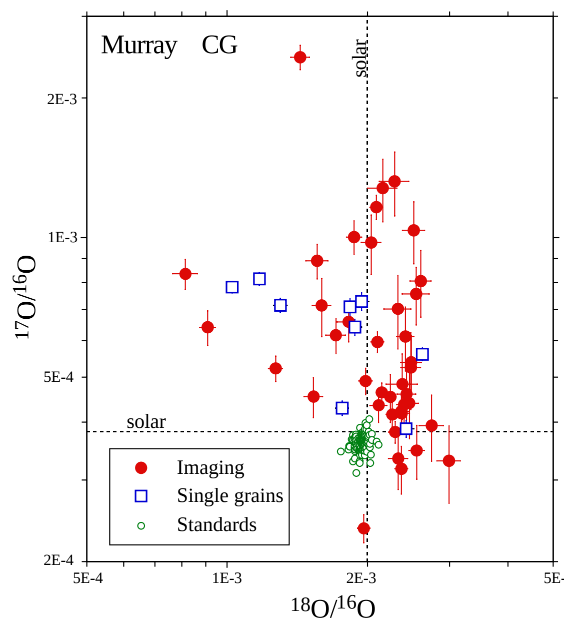

Figure 2. Oxygen isotopic ratios of presolar spinel grains from Murray residue CG located by raster imaging are compared with ratios from grains identified by single grain analysis. Also shown are the isotopic ratios of isotopically normal regions of one of the isotopic images that were used as calibration standards.