Fluorescence Imaging of the Stardust Collector Trays

Discovery of Aerogel Fluorescence
During the PE period it was noted that aerogel keystones removed from the Stardust collectors showed varying degrees of visible fluorescence when exposed to UV light. This was first noticed on an aerogel keystone that contained a track that had been examined by beam analysis techniques.

Several zones of fluorescence were noted. First, portions of the keystone that represented the exposed surface of the aerogel tile showed increased fluorescence (see above). Second, portions of the aerogel exposed to ionizing radiation showed induced fluorescence.

Data were taken from several keystones (previous irradiated and not previously irradiated) by Andrew Westphal and Scott Sandford using a fluorescent microscope at UC Berkeley that could measure fluorescent yield as a function of excitation wavelength. These studies failed to yield conclusive results. The biggest differences between different samples and different locations in the same sample were largely a matter of fluorescent intensity. The spectral nature of the fluorescence appeared to be fairly consistent from sample to sample, and was similar for both the radiation induced fluorescence and the fluorescence seen on the original keystone surfaces.

The presence of this fluorescence caused concern within the Organics PET since it was possible that it was due to exposure of the surface to an organic contaminant with conjugated bonds and/or that radiation exposure during beam analyses was changing some of the carbon original to the flight aerogel (which is largely in the form of Si-CH₃) into more complex, fluorescent forms.

Measurement of the “Global” Aerogel Fluorescence in the Cometary Tray
In order to understand whether this fluorescence was a more global issue on the collectors, Simon Clemett used UV illumination to examine the collector trays at JSC. The imaging conditions were not ideal, but it was possible to obtain fluorescence images of the cometary collector tray using a 254 nm fluorescence excitation source held at a distance of about 12 inches from the front (i.e. comet exposed) surface of the tray. A digital camera was position about 2 inches above the UV source and at a distance of about 14 inches from collector. The camera had a zoom lens that was adjusted so that an individual image would cover
approximately 1/4 of the collector surface. Under these conditions the imaged area had a relatively uniform UV flux.

Figure 1(A): Envisioned setup with the UV source located directly below the digital camera and at sufficient distance to provide an approximately uniform UV flux on the imaged region of the collector tray; Figure 1(B) Practical implementation in the Stardust clean room.

These images were then combined into composite images of the entire tray. Each of these images uses the green channel as a sort of crude spectrometer, so that the intensity variations represent the fluorescence emission principally in the 500~540 nm range. Each image was then processed by a de-speckling algorithm to remove CCD hot spots (which are inevitably since each image required a 30 second exposure) and then corrected for lens barrel distortion and made into a montage using an autocorrelation routine. The first images (below) use a simple grayscale.
Since in general the human eye can only really differentiate about 32 shades of grey, the same image using a color table that helps to better show the variations is also provided.

It is clear from these images that the fluorescence is not uniformly or smoothly spread across the collector tray, strongly suggesting that the source of the fluorescence is not a contaminant to which the entire tray has been exposed. A histogram plot of the rough individual tile ‘bulk’ fluorescence intensities is shown below.
Correlation of Fluorescence with Aerogel Production Batch Number

Comparison of the images above with a map of the flight aerogel batch placement of the tiles in the tray demonstrated that the fluorescence correlates strongly with production batch number. A somewhat subjective analysis made by Scott Sandford of the relationship of cell fluorescence with aerogel Batch Number in which each tile is categorized (by eye) as having low, low-medium, medium, medium-high, and high fluorescent intensity is summarized in the table below. It is clear that the intensity of the fluorescence is batch-related, although there are a few cells that fall off the trends (see below).

### Number of Cells vs. Intensity of Fluorescence

<table>
<thead>
<tr>
<th>Batch</th>
<th>Low</th>
<th>Low-Med</th>
<th>Medium</th>
<th>Med-High</th>
<th>High</th>
<th>Unknown Cells</th>
</tr>
</thead>
<tbody>
<tr>
<td>227</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>232</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>3</td>
<td>044 (probably High)</td>
</tr>
<tr>
<td>234</td>
<td>14</td>
<td>6</td>
<td>1</td>
<td>-</td>
<td>-</td>
<td>027, 054, 086, 092 (probably Low)</td>
</tr>
<tr>
<td>235</td>
<td>17</td>
<td>9</td>
<td>5</td>
<td>-</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>236</td>
<td>-</td>
<td>10</td>
<td>15</td>
<td>-</td>
<td>-</td>
<td>009, 115 (probably Medium)</td>
</tr>
<tr>
<td>237</td>
<td>24</td>
<td>3</td>
<td>-</td>
<td>-</td>
<td>1</td>
<td>038, 052 (probably Low)</td>
</tr>
<tr>
<td>239</td>
<td>11</td>
<td>1</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>126 (probably low)</td>
</tr>
<tr>
<td>246</td>
<td>1</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td></td>
</tr>
</tbody>
</table>

Batches 227 and 232 are responsible for all the brightest cells but one. Fortunately, these batches contributed relatively few tiles to the tray. Unfortunately, Tile C2044 is from Batch 232 and we should remember this when looking at the data from any samples from that tile. The tiles from these two batches are: C2075 (trapezoidal cell), C2028, C2035, C2044, C2085.

Batch 236 shows uniformly moderate fluorescence – data from samples taken from Tiles C2009 and C2115 should be considered in this light (pun intended).

Batches 234 and 235 show generally low fluorescence, but some Tiles grade up to the Medium fluorescence level. Thus, pulled Tiles C2027, C2054, C2086, and C2092 probably have low fluorescence, but possibly a little higher.

Batches 237, 239, and 246 consistently show very little fluorescence, with the exception that Tile C045 is listed as being from Batch 237 and shows very high fluorescence (appears the brightest of all the tiles). Given the uniformity of the other cells from this batch, this suggests that Tile C045 may be mislabeled. Samples from Cells C2038, C2052, and C2126 are probably not severely complicated by the fluorescence issue.
**Current Thinking on the Nature of the Fluorescence**

The fact that adjacent cells can show greatly different fluorescence intensities indicates that the source is not organics or other fluorescent materials (cometary or contaminant) added to the samples after tray assembly. So far, there has been no noted correlation between the intensity of the fluorescence and the nature and abundance of organic materials detected using a variety of analytical techniques in the samples, suggesting that organics may not be responsible at all.

NMR measurements made by George Cody on preflight aerogel samples from both a low and a high fluorescence batch suggest the fluorescence may be due to Q₃ defect sites, e.g. Si-O•, which are known to be a potential source of photo-luminescence [Nishikawa et al. 1996, J. Appl. Phys. 80, 3513]. The density of these sites correlates in a qualitative sense with fluorescent intensity of the batches in the samples examined so far. However, only a few samples have been examined in this manner, so the correlation is not rigorously established.

Fluorescence due to defects is consistent with the observations made to date. More intense fluorescence at aerogel surfaces could be due to induced defects caused by solar particle irradiation. Increased fluorescence induced by beam analysis techniques could also be due to production of such defects. This would also explain why the spectral properties of the ‘original’ surface and volume induced fluorescence look very similar.

Scott Sandford    October 3, 2006