

QUANTIFICATION OF SURFACE CONTAMINATION ON GENESIS SOLAR WIND SAMPLES

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Introduction: The Genesis mission was the only mission returning pristine solar material to Earth since the Apollo program up to date [1, 2]. Unfortunately, the return of the spacecraft on September 8, 2004 resulted in a crash landing shattering the solar wind collectors into small fragments and exposing them to desert soil and other debris at the landing site. To permit analysis of solar wind material embedded within a collector, surface cleaning is a necessity. Cleaning should remove surface contamination quantitatively, but leave the embedded solar wind intact. However, contamination varies from sample to sample and each fragment requires an individual cleaning approach. To ensure that cleaning is effective a sample has to be inspected carefully before and after cleaning was performed. This is done optically using a microscope and also spectroscopically by using total reflection X-ray fluorescence (TXRF) analysis. Total reflection X-ray fluorescence is a non-destructive surface sensitive multi element analysis method and has been applied to a number of Genesis solar wind samples before and after various cleaning methods. [3-5].

In case contaminants remain after cleaning, quantification of those is important to evaluate the effectiveness of the procedure itself and to provide information about a sample for other investigators. Quantification is typically done by addition of an internal standard for TXRF analysis, but this approach is not possible for Genesis samples as it would introduce additional contamination. In this study, external calibration curves were produced instead and the concentration of remaining elements determined for three flight samples after different cleaning procedures were applied.

Experimental: Three Genesis flight samples (61546, 30580 and 61052) were analyzed using TXRF after at least one cleaning step was performed. Flight sample 61546 (synthetic single-crystal sapphire) was measured after cleaning with ultra pure water (UPW), flight sample 30580 (synthetic single-crystal sapphire) was analyzed a first time after UPW cleaning and a second time after acid cleaning with a mixture of concentrated hydrochloric, concentrated nitric acid and ultrapure water in equal proportions, and flight sample 61052 (float zone silicon) was first measured after UPW cleaning and then after CO₂ snow application. [3-5]. Figure 1 shows micrographs of each flight sample after the UPW cleaning procedure was applied.

All samples were analyzed by TXRF (PicoFox, Bruker AXS, Madison, WI) using Molybdenum excitation (17.4keV) at 50kV and 600 μ A power. Data collection time was 3600 seconds.

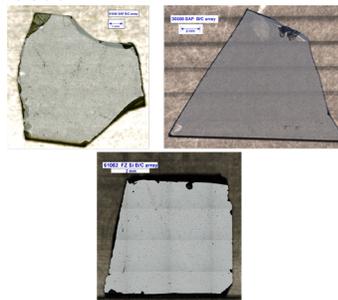


Figure 1: Micrographs of flight samples 61546 (top left, scale bar 1 mm), 30580 (top right, scale bar 2mm), 61052 (bottom, scale bar 2mm).

For quantification of contaminants, calibration curves were produced to obtain the relationship between concentration and fluorescence intensity. The measured fluorescence intensity of each element detected on the surface of a flight sample was then converted into concentration using these calibration curves. Limits of detection (LOD) and quantification (LOQ) were determined to ensure the validity of the results. When the values were below LOQ the data were disregarded. Both LOD and LOQ are shown in table 1 for elements commonly found as surface contaminants for Genesis samples. Please note that the data are shown in atoms for the area analyzed by TXRF. The area is 10mm² (2mm by 5mm) in size.

Element	LOD [atoms]	LOQ [atoms]
Cr	4E10	1E11
Mn	4E10	1E11
Fe	2E10	7E10
Ni	2E10	7E10
Zn	1E10	4E10
Ga	9E9	3E10
Ge	8E9	3E10

Table 1: Limits of Detection (LOD) and Quantification (LOQ) in atoms for the area analyzed (10mm²).

Results and Conclusion: Figure 2 shows the TXRF spectrum for sapphire sample 61546 and table 2 the data obtained for remaining contaminants using the external calibration method. Since contamination is not uniformly distributed over the surface as can be seen from the micrographs in figure 1, concentrations are shown for the area analyzed instead of the more common atoms/cm².

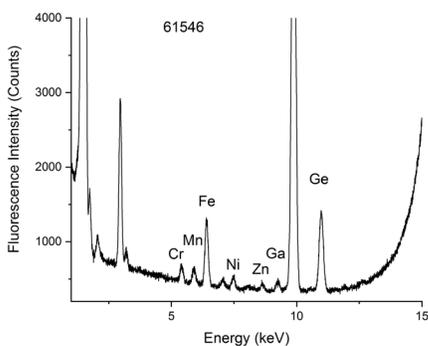


Figure 2: TXRF spectrum of sapphire sample 61546 after ultra pure water jet (UPW) cleaning.

Element	Concentration [atoms]
Cr	3E12
Mn	1E11
Fe	6E12
Ni	7E11
Zn	2E11
Ga	3E11
Ge	3E13

Table 2: Concentration of remaining contamination for sapphire sample 61546 in atoms for the area analyzed by TXRF (10mm²).

Figure 3 and table 3 show the data obtained with TXRF for sapphire sample 30580 after UPW cleaning (black spectrum, second column) and after additional acid cleaning was applied (red spectrum, third column).

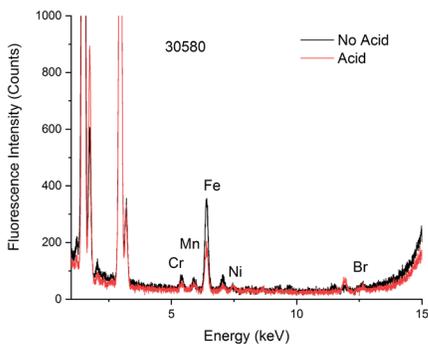


Figure 3: TXRF spectra of sapphire sample 30580 after ultrapure water (UPW) cleaning (black) and after additional treatment with concentrated acids (red).

Element	30580 No Acid Concentration [atoms]	30580 Acid Concentration [atoms]
Cr	7E11	3E11
Mn	2E11	2E11
Fe	2E12	1E12
Ni	1E11	9E10

Table 3: Concentration of remaining contamination for sapphire sample 30580 in atoms for the area analyzed by TXRF (10mm²) after UPW cleaning and after additional acid cleaning.

Figure 4 and table 4 display the results for silicon sample 61052 after UPW cleaning (black spectrum, second column) and after additional CO₂ cleaning is done (red spectrum, third column).

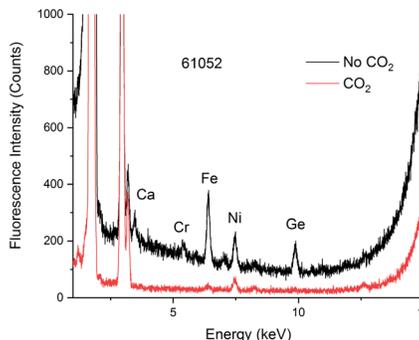


Figure 4: TXRF spectra of silicon sample 61052 after UPW cleaning (black) and after additional CO₂ cleaning (red).

Element	61052 no CO ₂ Concentration [atoms]	61052 CO ₂ Concentration [atoms]
Cr	6E11	
Fe	2E12	
Ni	5E11	2E11
Ge	2E11	5E10

Table 4: Concentration of remaining contamination for silicon sample 61052 in atoms for the area analyzed by TXRF (10mm²) after UPW cleaning and after additional CO₂ cleaning.

The data presented here show that TXRF spectra provide information about the remaining contaminants present on a sample and that it was possible to quantify those contaminants using external calibration.

References:

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