

National Aeronautics and
Space Administration
Goddard Space Flight Center
Greenbelt, MD 20771



Reply to Attn of 440

April 18, 1995

TO: 440/Associate Director of Flight Projects
for Hubble Space Telescope

FROM: 442/SI Optical Interface Manager

Subject: WFPC-1 Pickoff Mirror Contamination Failure Review
Report

Enclosed please find a copy of the final report of the WFPC-1
Pickoff Mirror Contamination Failure Review Board. Included in
this report is an executive summary describing what the board has
concluded is the likely source of the contaminant. Additionally,
the report includes a set of recommendations which the board
believes would be prudent actions to take to minimize the risk to
the current and future scientific instruments inside of HST.

Lee Feinberg

Lee Feinberg

Enclosure

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April 15th, 1995

WIDE FIELD/PLANETARY CAMERA-I PICKOFF MIRROR
CONTAMINATION
FAILURE REVIEW BOARD REPORT

Hubble Space Telescope Project
Goddard Space Flight Center
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References:

1. Optical Components Degradation Assessment and On-Orbit Implications, HST Returned Hardware Evaluation Symposium, December 15th, 1994
2. GCMS and Pyrolysis Testing Report, LMSC, 4/95
3. Auger Analysis of HSP Filters, 3/95, 5/95
4. Laboratory Experiments to Study Surface Contamination and Degradation of Optical Coatings and Materials in Simulated Space Environments, G. Hass and W. R. Hunter, Applied Optics, 9/70
5. Evans East XPS Report Summaries

1. Executive Summary

In December 1993, the space shuttle Endeavor and its crew of 7 astronauts rendezvoused with the Hubble Space Telescope in order to repair its faulty optical system and other components. As part of this mission, the Wide Field Planetary Camera-I (WFPC-I) scientific instrument was replaced by the Wide Field Planetary Camera-II (WFPC-II) and the High Speed Photometer (HSP) was replaced by the Corrective Optics Space Telescope Axial Replacement (COSTAR) instrument in order to correct the spherical aberration that was known to be incorrectly polished in the primary mirror of the telescope. The success of this mission allowed the WFPC-1 and HSP to be returned to earth. Once on the ground, the payload bay of the space shuttle was purged within 45 minutes of landing, thus keeping the instruments in a relatively clean environment.

On the ground, plans were already under way to remove and study all of the returned hardware in order to understand space environment effects on HST hardware. One important study that was planned involved reflectivity tests of the WFPC-1 pickoff mirror. Since this mirror was mounted on an arm that stuck out from the instrument into the central "hub" region of the Hubble Space Telescope Optical Telescope Assembly (OTA), it was thought that understanding degradation to the mirror would give an indication of how other instruments in Hubble would and should perform. The decision was made to test the pickoff mirror completely in an ambient environment in order to minimize the chance that vacuum exposure could change the condition of the mirror. Since the Hubble optical system performs at wavelengths as short as Lyman-alpha (1216 angstroms) and below, it was decided to initially test the mirror in a nitrogen purged tent to obtain ultra-violet measurements.

The initial reflectivity measurement of the pickoff mirror indicated a strong dropoff in reflectance at wavelengths near Lyman-alpha. Subsequent vacuum reflectance measurements confirmed a dropoff noticeable at 1600 angstroms that resulted in less than 1% reflectivity at 1216 angstroms. Post-thermal vacuum test measurements from JPL indicated that the mirror had a 72% Lyman-alpha reflectivity before launch.

At this point, the team studying the problem decided that the aperture window which was located on WFPC-1 next to the pickoff mirror should also be evaluated. The window also showed a degradation at the same wavelengths on its hub facing side, though not quite so drastic. The team then had X-ray Photo-electron Spectroscopy performed to the surface of the mirror and both surfaces of the window. These results, along with additional testing requested by the Failure Review Board indicated an approximately 450 angstrom thick uniform organic contaminant on the pickoff mirror, and an approximately 150 angstrom uniform

organic contaminant on the aperture window only on the side which faced the HST hub. Additionally, an organic contaminant similar to that found on the pickoff mirror and on the hub-facing side of the aperture window was found on HSP filters that directly faced the hub. The amount of organic on the HSP filters was similar to the amount found on the Hub-facing side of the WFPC-1 aperture window.

Additional testing using Time-of-Flight Secondary Ion Mass Spectroscopy (TOF-SIMS) on the contaminant confirmed an additional XPS result that the surface contained polydimethyl siloxane (silicones). This was found largely in the very first molecular layers, as TOF-SIMS is extremely sensitive to the top few monolayers of contaminants. Because there is only a few layers, these silicones are not likely to be the predominant cause of the optical degradation.

Additional testing using Gas Chromatography/Mass Spectroscopy (GC/MS) of the soluble organics from the aperture window indicated the presence of di-octyl phthalate (plasticizer), di-ethyl phthalate (plasticizer), tri-phenyl phosphine oxide (a fire retardant), and aliphatic acids (fatty acids). This initial analysis did not identify any of the polymeric portion of the contaminant. However, Solids Probe Mass Spectrometry and Pyrolysis Gas Chromatography/Mass Spectrometry on the Hub facing side of the WFPC aperture window and on the pickoff mirror did indicate the presence of high molecular weight material that is thought to be polymeric, and traces of phthalate esters and caprolactam. This data suggested that a partially polymerized contaminant comprised of more than one type of molecule likely was on the pickoff mirror.

Additional returned hardware surfaces were identified to help determine whether the contamination occurred in HST or during the First Servicing Mission. The team had scrape samples taken on hub facing and non-hub facing locations of the High Speed Photometer. The analysis of these scrape samples qualitatively indicates that only the Hub facing surfaces had the high molecular weight contaminant that came off at high temperature in the Pyrolysis MS analysis. Additionally, there were qualitative similarities between the HSP polymer and what was seen on the WFPC-I aperture window and pickoff mirror.

Since witness samples indicated the contamination did not occur on the ground, the FRB has concluded the likely contamination mechanism is the photopolymerization of outgassing hub contaminants from low level earth ultra-violet albedo over the three and a half years in orbit. This is possible because Hubble sees the bright earth during orbits where it is observing a target which is occulted by the Earth. The theory is supported by the fact that the reflectivity degradation curve is consistent with contaminants that have been photo-polymerized. Additional evidence of polymerization is the contaminant did not come off in vacuum or when washed in Hexane. The team considered it unlikely

that charged particles were the energy sources for the mechanism; this was partially substantiated based on results from Hass and Hunter which indicated that fast electrons and protons have no effect on the 1216 Angstrom reflectivity of MgF1 on aluminum.

The team does not believe that the servicing mission was responsible for the large portion of contaminant for a number of reasons. The most convincing arguments were the HSP data, the fact that there was 450 angstroms on the pickoff mirror which was exposed in space during the servicing mission for less than 2 hours, and the correlation of FGS outgassing products to what was found on the pickoff mirror.

The specific outgassing source or sources of the contaminant is difficult to identify. However, the team has concluded that the source must have a path to the HST hub and is probably not from the science instruments because of their small apertures and stringent certification requirements. This leaves the Optical Telescope Assembly and the Fine Guidance Sensors as the likely candidates. Neither of these assemblies were certified to the same requirements as the scientific instruments.

A vacuum test on the spare FGS has been performed and the results indicate outgassing levels higher than the science instruments. Additionally, Gas Chromatography Mass Spectroscopy (GCMS) and Fourier Transform Infra-red Mass Spectroscopy (FTIR) analysis of cold finger contaminants from the test showed high levels of caprolactam and aliphatic hydrocarbons. Initial analysis of these results indicates they are consistent with the findings from the pickoff mirror, aperture window, and HSP filters, but do not rule out other sources. A test to better understand the efficiency of photopolymerization from Earth albedo UV is ongoing.

These findings indicate that bright earth UV induced polymerization is a particular concern during regular HST operations, and also a concern during servicing missions. Therefore, the Failure Review Board has the following recommendations:

1. If the spare Fine Guidance Sensor is flown, clean it to the same cleanliness level as the original Fine Guidance Sensors are at now after five years of outgassing.
2. If the spare Fine Guidance Sensor is flown, perform UV monitoring of STIS, WFPC-II, and the remaining COSTAR channel as part of the Servicing Mission Orbital Verification (SMOV). The monitoring should occur frequently enough to assess build up of contaminants soon after the mission is complete. This would allow a determination of deposition rate and would allow for time to decide if operational constraints should be imposed.

3. Minimize exposure of UV optics to the bright earth during servicing mission instrument changeout. For the 1997 mission it is important that the open STIS instrument aperture not be unnecessarily pointed at the bright earth.
4. If an FGS is removed, its pickoff mirror should be preserved both during the servicing mission and during ground processing. This includes minimizing exposure of this mirror to the bright earth during the servicing mission.
5. Characterize, as well as possible, the environment to which the science instruments are subjected during a servicing mission with witness mirror(s) and/or with onboard QCM measurements. The time resolution of the QCM measurements would provide considerably more information regarding potential contamination sources.

2.0 Vacuum and Ambient Reflectance and Transmission Measurements

A detailed description of the reflectance and transmittance measurements performed by the Optics Branch on the HST returned hardware is given in Appendix A and contains plots of reflectivity versus wavelength. A brief description follows here.

The items measured in connection with the pickoff mirror investigation were:

- °The flight pickoff mirror
- °The spare pickoff mirror, which was identical to the flight mirror in every way except that it did not go into space
- °A reference mirror which had the same coating as the two pickoff mirrors, and whose absolute reflectance was known. This was used as a standard from which to calculate the absolute reflectance of the pickoff mirrors.
- °The aperture window

Reflectance measurements were initially performed in a tent under a continuous dry nitrogen purge. The purge was needed to displace air which absorbs the shorter wavelengths. The normal procedure is to perform UV reflectance measurements in vacuum; however in this case there was a concern that exposing the pickoff mirror and aperture window to vacuum might cause the optics to change (by pumping off a contaminant, for example). Later, after the measurements under nitrogen were completed and after the flight pickoff mirror had been exposed to vacuum for XPS analysis with no ill effects, the reflectance and transmittance measurements were repeated in vacuum to verify the accuracy of the results. There were some minor disagreements between the vacuum and nitrogen measurements due to a problem with wavelength selection in the nitrogen-purged reflectometer. Once this problem was corrected, the vacuum and nitrogen measurements agreed well.

The key result is shown in Figure 1. Note that measurements were made at two angles of incidence: normal incidence and 45 degrees. The normal incidence measurements are standard Optics Branch procedure, and the 45 degree measurement is shown because the pickoff mirror was used at a 45 degree angle during the HST mission. The flight pickoff mirror shows a dramatic loss of reflectance in the far ultraviolet compared to the spare pickoff mirror and compared to preflight measurements of both the flight and spare mirrors.

The aperture window has a transmittance loss in the ultraviolet. Some of this roll-off is normal for a thick magnesium fluoride window, however some degradation is apparent compared to the preflight data.

3.0 Microphotography/Visual Analysis

The observed reflectance degradation in the UV was cause for great concern, so further testing was pursued to find the cause of the degradation. First, nondestructive tests (visual inspection, microscopic inspection, micro-photography) were performed, then minimally destructive testing (XPS and TOF-SIMS for example) was done, and finally the destructive tests (GC/MS of solvent rinses, etc.) were run.

A microphotograph of the WFPC-1 Pickoff Mirror and the spare pickoff mirror is shown on Figure 2 on the next page. Visually, a blue haze was evident on the flight pickoff mirror, but not on the spare pickoff mirror or the aperture window. The haze was not uniform; it was more pronounced near the center and less visible in a narrow region at the edges of the pickoff mirror.

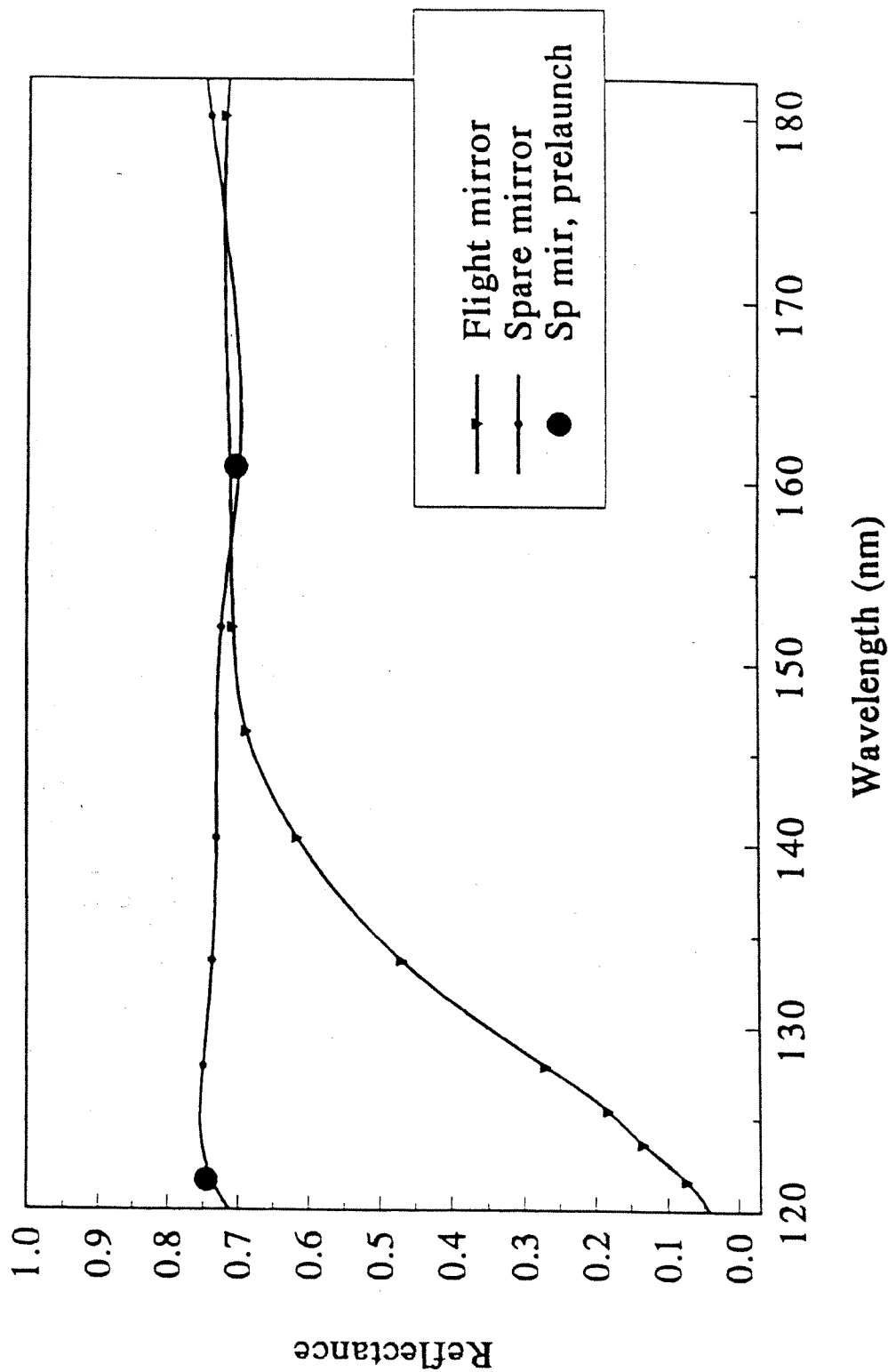
The flight pickoff mirror and aperture window were then examined and photographed under a high-power phase-contrast microscope using the Automated Image Analysis Facility run by the Contamination Engineering Section. No significant contamination or damage could be seen on the aperture window. The window did have a crystalline defect in the exact center; consultation with JPL revealed that this defect was present before launch.

The pickoff mirror contained numerous circular features about 1 to 2 microns in diameter (see attached photographs). This led us to suspect contamination, since contamination does not generally deposit as a uniform film, but rather forms small droplets or droplets on top of a film. However, the photographs alone could not allow us to rule out the possibility that the reflectance degradation was caused by damage to the optical coating, since the circular features could just as easily be blisters or pits in the coating. Surface chemistry analyses such as XPS were necessary to verify conclusively that the problem was contamination, not coating damage.

Figure 1

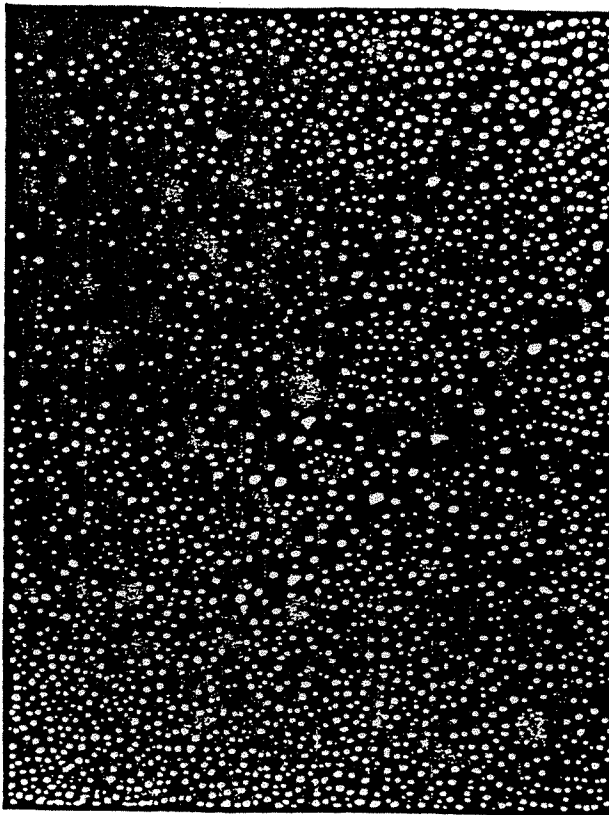
UV Reflectance at 45 degrees

Flight and Spare Pickoff Mirrors



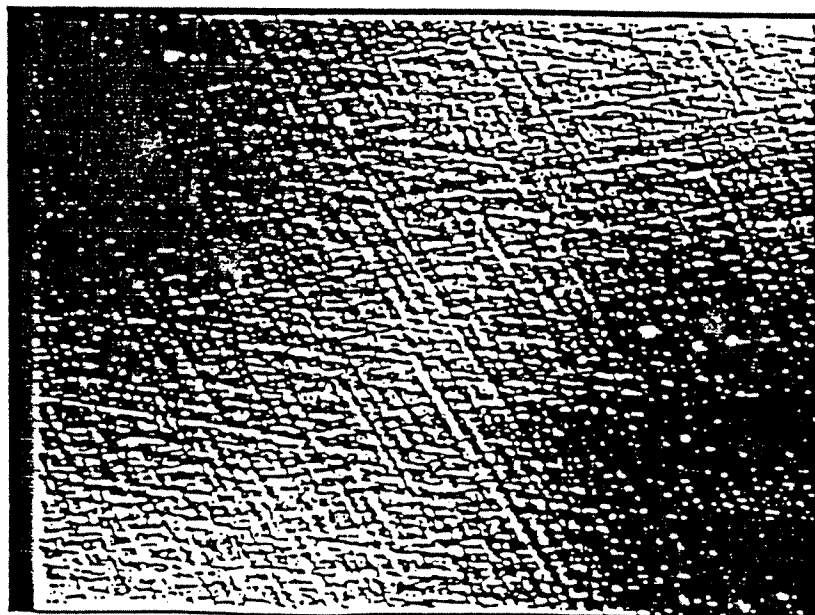
- 10 A.M.

Figure 2



WF/PC-1 P.O.M.
close up of blue haze

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268x



SPARE MIRROR
(WFPC-1 SPARE P.O.M.)

4.0 Evans East XPS Results

Evans East performed a number of X-ray Photoelectron Spectroscopy analyses on the pickoff mirror, aperture window (both sides), HSP filters, WFPC multi-layer insulation, and spare and reference samples. The results indicated that surfaces which faced the HST hub while in orbit were contaminated with an organic (carbon containing) contaminant. This included the pickoff mirror, hub facing side of aperture window, hub facing side of HSP filters. WFPC MLI and a metallic ring around the HSP aperture window gave inconclusive results as to whether there was an organic because the MLI is carbon filled and the ring has a black dye used in its blackening.

A summary of the pickoff mirror, aperture window, and filter data are shown on Figure 3. The data indicates that carbon was a major contributor (63-67%) on all hub facing surfaces, followed by oxygen (27-30%). Evans East was able to sputter through the contaminant until it saw the optical substrate (Magnesium Fluoride for the aperture window and pickoff mirror). However, the sputtering could not be correlated to contaminant thickness at first because the sputter efficiency of the contaminant layer was unknown. The team got around this problem by sputtering a square well through the contaminant and then using Atomic Force Microscopy (AFM) to measure the depth. The data was consistent with the equivalent thicknesses Evans East was already estimating. The pickoff mirror was estimated to have 450-500 angstroms of organic and the aperture window was estimated to have 150-160 angstroms. Analysis of bond energies for atoms excited from the surface by XPS indicated the presence of phthalates and esters.

5.0 GSFC XPS Results

Various space flight articles were examined by X-ray Photoelectron Spectroscopy (XPS) with Code 313 facilities. Included in the list of items measured by Code 313 is an optical filter element from the High Speed Photometer (HSP) and several pieces of the top layer of the Multi Layer Insulation (MLI) from the Wide Field and Planetary Camera I (WF/PC-I). The Pick Off Mirror itself was not analyzed in Code 313 facilities because the mirror is too large for the sample chamber.

The filter element was in the light path for the HSP instrument. The MLI samples from the WF/PC-I were not directly in the light path, but were in close proximity to the POM. A reference MLI sample was made available for the analysis. This reference was assembled the same time as the flight MLI, but was never attached to the WF/PC-I. The relative surface elemental percentages of the HSP filter element and MLI samples are given in the following table.

Figure 3
Evans East XPS Results Comparison
Atomic Concentration of Elements Detected

		Depth								
	Sample	Depth (Å)	O	N	C	Si	Sn	F	Mg	
HSP FILTER	Inner Surface	0	36.2	0.7	40.7	22.3	-	-	-	
	Outer Surface	0	24.2	0.9	65.8	7.9	0.1	1.1	-	
	Outer Surface	50	15.7	1.9	66.6	15.4	0.3	0.0	-	
	Outer Surface	100	34.0	2.1	35.9	27.7	0.3	0.0	-	
	Outer Surface	150	50.6	1.3	12.7	35.3	0.1	0.0	-	
WF/PC-I APERTURE WINDOW	Outside Position 1	0	27.7	1.6	66.5	2.8	-	1.4	-	
	Outside Position 2	0	28.2	1.7	66.4	2.5	-	1.3	-	
	Outside Position 2	-100	12.8	0.3	51.5	-	-	21.8	13.7	
	Outside Position 2	-150	16.8	0.0	17.6	-	-	39.1	26.5	
	Outside Position 3	0	27.9	1.7	66.5	2.7	-	1.3	-	
	Inside Position 1	0	6.4	0.5	31.8	-	-	38.3	23.0	
	Inside Position 2	0	6.7	0.5	32.6	-	-	37.3	22.9	
	Inside Position 2	-50	6.1	0.0	8.8	-	-	60.0	25.1	
	Inside Position 2	-100	7.5	0.0	7.3	-	-	56.5	28.7	
	Inside Position 2	-150	11.0	0.0	3.1	-	-	56.4	29.5	
WF/PC-I PICKOFF MIRROR	Hazy Area	0	30.0	2.6	63.4	2.3	-	1.6	-	
	Scratched Area	0	30.5	1.9	64.0	2.5	-	1.1	-	
	Referenced Area	0	31.5	2.4	62.5	2.4	-	1.2	-	

* Atomic Concentration by percent (%) of weight

Figure 3
Evans East XPS Results Comparison (cont'd)

Summary of Carbon Curve Fits

	Sample	C-(C,H)	C-(O,N)	C=O	O-C=O
WF/PC-I PICKOFF MIRROR	Hazy Area	60.2	20.6	8.3	11.0
	Scratched Area	58.9	21.1	9.3	10.7
	Referenced Area	60.3	21.2	8.8	9.7
WF/PC-I APERTURE WINDOW	Outside Position 1	61.3	21.9	7.7	9.1
	Outside Position 2	61.0	21.5	7.9	9.6
	Outside Position 3	60.5	22.6	9.3	7.6
	Inside Position 1	86.9	8.4	2.5	2.2
	Inside Position 2	87.1	8.8	2.5	1.6

*Concentration by percent (%) of weight

Sample	Carbon	Oxygen	Nitrogen	Fluorine	Silicon	Titanium	Aluminum
HSP Filter Element	68	21	1	0	10	0.3	0
WF/PC-I MLI Samples *	61	27	**	5	5	0	0
WF/PC-I MLI Reference	54	32	0	0	7	0	7

Atomic Percentages of the Elements

Table 1

* The values listed for the WF/PC-I MLI samples are averaged from 6 samples.

** One sample of MLI contained 2 atomic percent nitrogen. The remainder of the samples had zero atomic percent.

It is not known where the aluminum material originated on the MLI reference sample. It is thought to have originated by a rubbing action against the back surface of other MLI material.

The next process in the chemical analysis was the determination of carbon functional groups. This identification will determine if the chemical composition of the contamination layers is the same for all surfaces. The following table lists the surface functional groups for the analyzed samples.

Sample	Hydrocarbon (C-H, C-C)	Alcohol or Ether (C-O)	Aldehyde or Ketone (C=O)	Ester or Carboxylic Acid (O-C=O)
HSP Filter Element	57	31	12	0
WF/PC-I MLI Samples* **	73	15	0	14
WF/PC-I MLI Reference	71	14	0	16

Functional Group Percentages

Table 2

*** The values listed for the WF/PC-I MLI samples are averaged from 6 samples.

XPS results show the contaminant on the MLI to be different than the contaminant on the filter element. When comparing the three sample groups, the functional group values indicate a greater occurrence of hydrocarbons on the MLI samples, and a greater occurrence of alcohols or ethers on the filter element. Also shown is that aldehydes or ketones are present on the filter element, and are not present on the MLI, and ester or carboxylic acids are present on the MLI, and are not present on the filter element.

6.0 Time-of-Flight SIMS Result

Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) is an extremely sensitive technique for detecting and characterizing organic and inorganic materials deposited on surfaces. Like other mass spectrometry techniques, TOF-SIMS basically involves two phases - excitation of molecules and molecular fragments from the surface in question, (generating both ions and neutrals), and mass separation and detection of those ions.

The first phase, exciting material off of the surface, can be done by a variety of methods (laser desorption/ablation, pulsed or continuous ion beams, rastered ion beam), each using various intensities. Those techniques which use incident ion beam intensities equivalent to less than approximately 10^{12} ions/cm² are generally referred to as "Static" SIMS and result in the production of relatively large neutral and ionized fragments from a fraction of the surface monolayer, with less fragmentation than

other techniques. This lessens the problem of identifying the parent molecules, as larger fragments (or whole ionized molecules) are available for mass separation rather than similar or identical fragments from possibly different sources (i.e. C₈H₅O₃ phthalate fragments from both benzyl butyl terephthalate and diethylhexyl phthalate parent molecules).

The second phase of analysis, mass separation and detection, is performed by a mass spectrometer. Time-of-Flight refers to the type of mass spectrometer which uses a static magnetic field to turn or deflect the ionized molecules from their original paths after leaving the surface, and measuring their time of flight from the surface to the detector. The time taken to reach the detector, and the location on the detector of the fragments impact, are determined by the mass to charge ratio of the ionized fragment excited from the surface. The general principle is the same as for other types of mass spectroscopy (i.e. quadrupole MS) but mass separation is produced by the static magnetic field and fragment identification is aided by measuring the fragments time of flight. This technique allows the detection of molecules and fragments which are too large for other types of mass filter/ion detectors to see. Both positively and negatively charged fragments can be detected, generating positive and negative mass spectra.

The TOF-SIMS analysis of the WF/PC-1 aperture window initially used a Cesium ion source, and further analysis used a pulsed Gallium beam (Gallium Liquid Metal Ion Gun) as the primary ion source to excite material from the surface of the aperture window. In both cases, the beam intensity was equivalent to roughly 10¹² ions/cm², thus retaining the high surface sensitivity of Static SIMS. The instrument was specifically configured for high mass resolution to emphasize molecular identification. A TRIFT Time-of-Flight mass spectrometer was used to perform the ion mass separation and detection, generating both positive and negative ion mass spectra.

The data from the initial analysis showed high levels of polydimethyl siloxane (PDMS) or silicone on the sample surface. Silicones are relatively mobile molecules and are generally found at the surface of contaminant layers of which they are a constituent. For this reason, they tend to dominate (when present) the data from a Static SIMS, due to its extreme sensitivity to the top monolayer(s) of a sample. To minimize this problem, the sample was rinsed with hexane to remove silicone from the surface monolayer, and further data was then collected. The positive ion spectra showed sodium, silicon, adventitious carbon fragments, tin, cesium from the initial analysis, phthalate (C₈H₅O₃) and some higher mass species (288 and 316 AMU) which are likely nitrogen containing compounds. The negative ion spectra showed mostly oxygen and low mass carbon species, with some fluorine, chlorine, bromine and iodine, and SiO₃ and SiO₃H (probably from the silica substrate).