

ADDENDUM

DATE: January 27, 1999

OBJECT: To determine if there are any significant differences found between the surface analysis of the Delphos control samples and the actual Ring samples.

PROCEDURE:

Samples: Control Center
Control A-9
Control C-8
Ring D-3
Ring C-3
Ring A-2

All of the samples were analyzed using X-ray Photoelectron Spectroscopy (XPS), also known as ESCA. XPS is a non-destructive, surface technique which can be used to determine the composition of the outermost atomic layers of a solid material. The average depth of analysis is approximately 30 Å. Both elemental identification and chemical speciation are possible with careful analysis of the obtained binding energy information.

A sampling of each of the Delphos samples was lightly dusted on a piece of silver tape and placed in the XPS vacuum chamber with a 2x1 0-10 torr base pressure. The samples were then irradiated with a Monochromated Al K α x-ray beam.

RESULTS:

From the XPS data, there appears to be a significant difference between the Delphos control samples and the ring samples. The ring samples show almost twice as much carbon present on the surface compared to the control samples, along with an increase in the amount of nitrogen. The carbon region also indicates the presence of more highly oxidized carbon species present, indicated by the shoulder at 289 eV binding energy in the carbon spectrum of each of the ring samples. Ring sample D-3 also shows a higher concentration of phosphorus and calcium present. The calcium in the ring samples appears to be shifted to a slightly lower binding energy, indicating a different oxidation state. There is also evidence for a lower oxidation state of iron being present in the ring samples (shoulder at 710 eV). Most of the iron is present as Fe⁰.

The sulfur region was also interesting. All of the samples show the presence of some type of sulfate species. However, only the ring samples contain a small amount of a sulfide/mercaptan type compound.

