WSRC-RP--89-901

DE90 011239

TECHNICAL DIVISION
SAVANNAH RIVER LABORATORY

Key Words: MIIT, WIPP,
In-situ tests, SRL waste glass,
glass/metal interactions

Authorized Derivative Classifier:
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Retention Time: December 1992

November 5, 1989

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WIPP/SRL In-Situ Tests: MIIT Program - Glass/Metal Interfaces of SRS Waste Glass (U)

BACKGROUND

In 1986, approximately 2000 samples of waste glasses and proposed package components were buried in the salt formation at the Waste Isolation Pilot Plant (WIPP) in Carlsbad, New Mexico [1-3]. These samples are part of a five year study, called the Materials Interface Interactions Tests (MIIT), being managed by Savannah River Laboratory [SRL] and Sandia National Laboratory [SNL]. [4,5] MIIT involves the active participation of eight countries, including Belgium, Canada, France, Germany, Japan, Sweden, the United Kingdom, and the United States. The compositions of the 15 distinct waste glass and waste form systems used in MIIT are summarized in Table 1A and 1B. [6] All of the MIIT systems contain nonradioactive components and approximately half of the glass samples buried at WIPP are from SRL .

Waste glass samples were fabricated into the shape of 'pineapple slices' that were polished on one side. Proposed package components were also made into a similar configuration and the various glasses, metals, and geologic samples were then stacked onto heater elements within Teflon

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assemblies. This produced interactions of interest by creating glass/glass, glass/salt, and glass/metal interfaces. Overall, a total of 50 stacks or assemblies of pineapple slices were created in seven different stacking arrangements. Each individual assembly was then installed in an instrumented borehole at WIPP. Brine was then added to almost all of the boreholes and the assemblies heated and maintained at 90°C. This was achieved by energizing the central heating rod that traversed through the middle opening of each of the pineapple slices in each assembly. Due to the design of these units, samples could be removed at time intervals of 6 mos., 1 year, 2 years, and 5 years. Currently, all but the 5 year samples have been removed from test and are being evaluated in laboratories of MIIT participants.

OBJECTIVES

Many studies have been or presently are in progress on the 6 mos., 1 year, and 2 year samples. These studies are being conducted in eight countries and a variety of federal, national and university laboratories in the U.S. Some of these results have been presented in a special workshop in Cadarache, France sponsored by the Commission of European Communities [7] and a session dedicated to MIIT was held at the Fourth International Symposium on Ceramics in Nuclear Waste Management, sponsored by the American Ceramic Society. [8]

The main objective of the present study is to investigate the interactions of SRL Y (165/TDS) waste glass with proposed metal canister or overpack materials, and more specifically, to determine what effect these metals have on leaching of the SRL waste glass system. Metal systems studied include 304L stainless steel, TiCode-12, lead, and A216 carbon steel. Analyses were performed using scanning electron microscopy [SEM] along with complementary energy dispersive x-rays [EDX]. These results are then correlated to an earlier MIIT study which assessed the behavior of the same SRL waste glass system in WIPP but without metal present. [8]

SUMMARY

- There was no significant effect observed on leaching of SRL Y waste glass due to the presence of 304L S.S., the reference DWPF canister material. There were also no large effects observed on the leaching of SRL Y waste glass when leached in the presence of TiCode-12, lead, or even A216.
- Leached glass layers were characterized by two regions; a precipitated layer and an interaction zone. The depths of these regions were seen to increase with increasing time, from 6 mo., 1 year, to 2 years.
- The interaction zones were generally very consistent and uniform while the thicknesses of precipitated layers varied considerably, depending on the

interfaces studied.

• Interaction zones, which provide a measure of glass interaction and leaching, indicated that the SRL Y waste glass interacted only a very small amount with surrounding brine [<1-3 μ], even after being in test at WIPP for 2 years.

EXPERIMENTAL PROCEDURES

Six SRL Y waste glass samples from four MIIT assemblies were studied in the current investigation. The first group of samples were obtained from borehole #31 and represent a time dependent study involving SRL Y/304L S.S. interactions. These samples include Y-49 [6-mo.], Y-51 [1-yr.], and Y-53 [2-yr.] glasses. The remaining samples involve other metal/glass interactions evaluated after 2-yrs. of testing and include Y-69 [SRL Y/ TiCode-12, borehole #33, 2-yrs.], Y-85 [SRL Y/ lead, borehole #35, 2-yrs.] and Y-103 [SRL Y/ A216, borehole #37, 2-yrs].

A representative MIIT assembly involving glass/metal interactions is shown schematically in Figure 1, along with an enlarged and more detailed view of one of the four sub-assemblies in Figure 2. Samples in the current study were obtained from similar units. After the assemblies were removed from test at WIPP, the pineapple slices were cut into 1/8 wedges and studied in the laboratory. Four of the wedges were analyzed using a variety of analytical techniques as described elsewhere [8], while the rest of the pineapple slice was stored as a library sample [Figure 3]. A 1/8 wedge or smaller was analyzed in the present study using SEM/EDX. In Figure 4, locations of analyses and terminology that will be used in data analysis are summarized.

In order to assess the behavior of leached MIIT glasses, an integrated study approach is being used. This combines solution analyses with many detailed surface and bulk studies. The various analytical tools being used, which are summarized in Figure 5, provide different information on the composition or structure of elements of interest within the leached glass surface layers. [9] In the present study, SEM/EDX analyses were used to examine and characterize deposited salt precipitates, leached glass surfaces, and overall depths of leaching. The samples were prepared for analyses by cutting into segments 1/8 of a slice or smaller, which could then be easily mounted on a pedestal and studied in the SEM. The sample was mounted on the aluminum pedestal using double sided tape, so that it was standing on one of its edges. The edge that was standing up, and not stuck to the pedestal, was the edge studied. Next, a carbon coating was applied on the sample in order to minimize charging effects and improve electrical conductivity before placing it in the microscope. This method of sample preparation was the

least destructive to fragile surface layers that were formed during leaching.

Once the sample was in the scanning electron microscope, microstructures and leached layers were photographed and morphological features studied. The chemical composition of leached layers and interfaces of interest were then examined by energy dispersive x-rays. The side of the glass in contact with an adjacent glass was designated side 'A' while the side of the glass in contact with the adjacent metal designated side 'B'. (Figure 4) Since the outer diameter of the metal was smaller than the outer diameter of the glass, a lip was created which represented a glass/liquid interface and this region also was studied. EDX was performed over each of these surfaces and the chemistry of the glass/glass, glass/metal, and glass/liquid interfaces defined. Additional spectra were recorded on the cross-section of the glass, traveling from the outermost leached surfaces to the pristine bulk glass. (Figure 6)

RESULTS

Three layers or zones were clearly identified on all of the MIIT glasses. This observation is consistent with observations made in an earlier study which investigated the leaching behavior of a similar MIIT glass buried in the absence of metals.[8] The first or outermost layer is called the precipitated layer due to the fact that it was composed of precipitated salt phases deposited from the brine while in test underground. The next layer is located under the precipitates and referred to as the interaction zone . This region represents the leached glass surface and the amount of interaction or corrosion that occurred. The third and final zone is that of the bulk glass, and this represents the pristine or unreacted glass. The depth at which this zone starts represents the maximum depth of leaching. In Table 2 measured depths of precipitated layers and interaction zones for sides A and B of all six MIIT samples are summarized. Figures 7A through 7V detail morphological features and corresponding chemical analyses of precipitated and interaction zones along with interfaces and interactions of interest.

By examining the interaction zones of Y-49 [6-mo.], Y-51 [1-yr.], and Y-53 [2-yrs.], it is noted that the thickness of this zone increases consistently with increasing time. For side A, the zone starts off at less than one micron after six months, grows about 1 micron after one year, and becomes 1-2 microns after two years. For side B, the zone starts off at less than one micron after six months, grows about 2 microns after one year, and becomes 2-3 microns after two years. (Table 2) In an earlier study [8], SRL Y glass [G/G] leached in the absence of metal, was also seen to have an interaction zone of several microns after two years.

In general, interactions occurring near the outer diameter of the glass pineapple slice were slightly more extensive than those occurring near the inner diameter. The interaction zone near the outer diameter ranged from <1 to 2 microns, while near the inner diameter, this zone ranged from <1 to 1 micron.

Interaction zone measurements were very consistent and provided a reliable means to assess glass leaching. The precipitated layer thicknesses, however, varied considerably as a result of the accessibility of the brine to glass surfaces and the different amounts of salt precipitate deposited. It was also noted that the outermost precipitated salt layer was very friable and subject to damage from handling and storage operations.

CONCLUSIONS

After 2 years of burial, there was no significant effect on leaching of SRL Y waste glass when buried in the presence of 304L S.S. metal. Since this metal is the reference material for DWPF canisters, it is important that it does not adversely affect performance of the glass in a repository environment.

The leaching behavior of SRL Y waste glass is related to the interaction depth observed within the leached glass surface. Measurements performed on the glass after leaching for up to 2 years indicate that the SRL Y waste glass system is very durable and that it interacts only slightly with the surround geology/hydrology at WIPP.

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Table 1A (ref. 6)

Updated 11/12/87

MIIT WASTE FORM COMPOSITIONS {United States}

COUNTRY	*USA*	*USA*	*USA*	*USA*	*USA*	*USA*	*USA*	*USA*	*USA*
WASTE	SRL-Y	SRL/PNL-Y	SRL-Z	SRL-AGEDY	RHO	PNL	CU	MCC-Std	BASALT
FORMS	[165/TDS]		[131/35%TDS]		[HWVP-HW39]	[76-68]		[ARM-1]	
COMPONENTS									
SiO2 [Wt. %]	54.1	54.1	39.3	53.0	51.03	40.73	45.2	46.5	51.34
Na2O	10.3	10.3	12.0	10.1	11.26	11.3	11.0	9.66	4.42
B2O3	6.8	6.8	9.6	6.7	9.42	8.99	10.0	11.3	-
TiO2	-	-	0.8	-	0.34	3.04	1.0	3.21	-
Li2O	4.7	4.7	3.4	4.6	4.00	-	3.1	5.08	-
MgO	0.8	0.8	1.0	0.8	0.97	0.14	1.3	-	3.84
ZrO2	1.2	1.2	0.4	1.2	0.62	1.79	3.1	1.80	-
La2O3	-	-	0.2	0.4	0.52	4.33	-	-	-
Fe2O3	12.3	12.3	16.3	12.1	10.95	9.52	11.8	-	15.81
MnO2	2.9	2.9	4.8	2.8	0.23	0.04	1.7	5.59	0.33
Al2O3	4.1	4.1	3.3	4.0	4.75	0.63	3.3	-	17.68
NiO	0.9	0.9	2.0	0.9	-	0.22	0.7	-	-
CaO	1.5	1.5	1.2	1.5	2.93	2.22	0.6	2.24	6.08
CaNO3	0.1	0.1	0.1	0.5	-	-	-	-	-
Sr[NO2]2	0.1	0.1	0.1	0.5	-	-	-	-	-
Na2CO3	-	-	1.1	-	-	-	-	-	-
Na2CO4	-	-	0.2	-	-	-	-	-	-
Coal	-	-	0.8	-	-	-	-	-	-
Zeolite	-	-	3.5	-	-	-	-	-	-
BaO	-	-	-	-	0.33	0.54	0.6	0.66	-
CdO	-	-	-	-	-	0.05	-	-	-
CeO2	-	-	-	-	0.20	0.82[Ce2O3]	0.2	1.51	-
Cr2O3	-	-	-	-	1.23	0.47	0.2[CrO3]	-	-
Cs2O	-	-	-	-	0.20	1.14	0.1	1.17	-
Dy2O3	-	-	-	-	-	0.01	-	-	-
Cr2O3	-	-	-	-	-	0.01	-	-	-
Gd2O3	-	-	-	-	-	0.02	-	-	-
K2O	-	-	-	-	-	0.27	3.5	-	-
MoO3	-	-	-	-	0.27	1.96	-	1.66	-
Nd2O3	-	-	-	-	0.51	1.47	-	5.96	-
P2O5	-	-	-	-	-	0.66	2.5	0.65	-
SrO	-	-	-	-	0.10	0.42	-	0.45	-
ZnO	-	-	-	-	-	4.76	-	1.46	-
SeO2	-	-	-	-	-	-	-	-	-
Eu2O3	-	-	-	0.4	-	-	-	-	-
Yb2O3	-	-	-	0.4	-	-	-	-	-
Ag2O	-	-	-	-	-	-	-	-	-
SnO2	-	-	-	-	-	-	-	-	-
TeO2	-	-	-	-	-	-	-	-	-
Pr2O3	-	-	-	-	-	-	-	-	-
Sm2O3	-	-	-	-	-	-	-	-	-
RuO2	-	-	-	-	-	-	-	-	-
Rh2O3	-	-	-	-	0.03	-	-	-	-
PdO	-	-	-	-	-	-	-	-	-
CoO	-	-	-	-	-	-	-	-	-
Sb2O3	-	-	-	-	-	-	-	-	-
ThO2	-	-	-	-	-	-	-	-	-
CuO	-	-	-	-	0.17	-	-	-	-

Table 1B (ref. 6)

Updated 10/19/87

MIIT WASTE FORM COMPOSITIONS {International}

COUNTRY	*CANADA*	*CANADA*	*BELGIUM*	*BELGIUM*	*FRANCE*	*UNITED KINGDOM*	*JAPAN*	*GERMANY*
WASTE FORMS	[AECL-GC-NACTS 33]	[AECL-AS-C81]	[SAN 60-25-19-L3C2]	[WG 124]	[SON 68-18-17-L1-C2-A2-Z2]	[BNFL 209]	[JAERI-Z-20/JW-A]	[HMI-SM 513LW11]
COMPONENTS								
SiO2 [Wt. %]	48.00	50.22	43.41	60.7	45.48	51.0	38.27	52.154
Na2O	6.33	8.24	10.67	3.6	9.86	8.0	20.57	9.149
B2O3	-	-	17.00	-	14.02	12.0	12.99	13.083
TiO2	18.3	-	-	1.1	-	-	0.06	4.539
Li2O	-	-	5.00	-	1.98	4.0	-	4.183
MgO	-	-	-	2.9	-	6.04	1.49	2.047
ZrO2	0.37	0.33	0.144	-	2.65	1.14	1.73	0.810
La2O3	0.26	-	0.105	-	0.90	0.34	0.39	0.085
Fe2O3	0.14	0.13	-	12.5	2.91	2.60	3.18	1.712
MnO2	-	-	-	-	0.72	-	0.21[MnO]	0.764
Al2O3	8.05	20.48	18.09	2.9	4.91	4.87	4.29	3.615
NiO	0.12	0.11	-	1.1	0.74	0.34	0.18	0.432
CaO	14.32	11.72	3.50	4.1	4.04	-	6.60	4.539
CaNO3	-	-	-	-	-	-	-	-
Sr[NO2]2	-	-	-	-	-	-	-	-
Na2CO3	-	-	-	-	-	-	-	-
Na2CO4	-	-	-	-	-	-	-	-
Coal	-	-	-	-	-	-	-	-
Zeolite	-	-	-	-	-	-	-	-
BaO	0.17	0.15	0.052	4.4	0.60	0.46	0.49	0.100
CdO	-	-	-	-	0.03	0.02	0.02	-
CeO2	1.90	1.74	0.118	-	0.93[Ce2O3]	0.70	1.09	0.810
Cr2O3	0.04	0.04	-	2.2	0.51	0.54	0.22	0.282
Cs2O	0.32	0.29	0.215	-	1.55	0.63	0.76	0.130
Dy2O3	-	-	-	-	-	-	-	-
Cr2O3	-	-	-	-	-	-	0.05	-
Gd2O3	-	-	-	-	-	1.63	-	0.008
K2O	-	-	-	1.3	-	-	1.14	-
MoO3	0.75	0.69	0.161	2.1	1.70	1.20	1.35	0.264
Nd2O3	0.15	0.14	0.169	-	1.59	1.13	1.28	0.421
P2O5	-	-	-	-	0.28	0.23	1.21	0.006
SrO	0.09	0.08	0.056	-	0.33	0.23	0.26	0.046
ZnO	-	-	-	-	2.50	-	-	0.016
SeO2	-	-	-	-	-	0.02	0.02	-
Rb2O	-	-	-	-	-	0.09	0.09	0.020
Y2O3	0.06	0.05	-	-	0.20	0.14	0.16	0.045
Ag2O	-	-	-	-	0.03[Ag2O3]	0.02	0.02	-
SnO2	0.01	0.01	-	-	0.02	0.01	0.02	-
TeO2	-	-	-	-	0.23	0.14	0.18	0.041
Pr2O3	0.13	0.12	0.050	-	0.44	0.32[Pr6O11]	0.39[Pr6O11]	0.077
Sm2O3	-	-	-	-	-	0.19	0.25	0.058
RuO2	-	-	0.074	-	-	0.67	0.59[Ru]	0.148
Rh2O3	-	-	0.066	-	-	0.13	0.07[Rh]	-
PdO	0.49[Pd]	0.45[Pd]	-	-	-	0.40	0.19[Pd]	-
CoO	-	-	-	-	0.12	-	-	-
Sb2O3	-	-	-	-	0.01	-	-	-
ThO2	-	-	-	-	0.33	-	-	-
CuO	-	-	-	1.1	-	-	-	-

Table 2

OBSERVED REGION THICKNESSES OF WASTEGLASSES REACTING WITH DIFFERENT METALS

SAMPLE	TIME	LOCATION Side	Diameter	INTERFACE	PRECIP. LAYER	INTERAC. ZONE
Y49 (304L)	6 mos.	A	I.D.	G/G	<1μ	<1μ
			O.D.	G/G	<1μ	<1μ
		B	I.D.	G/M	<1-6μ	<1μ
			O.D.	G/L	<1-6μ	<1μ
Y51 (304L)	1yr.	A	I.D.	G/G	<1-2μ	1μ
			O.D.	G/G	<1-2μ	2μ
		B	I.D.	G/M	<1-3μ	2μ
			O.D.	G/L	<1-3μ	2μ
Y53 (304L)	2yr.	A	I.D.	G/G	<1-8μ	1-2μ
			O.D.	G/G	<1-8μ	1-2μ
		B	I.D.	G/M	<1-6μ	2-3μ
			O.D.	G/L	<1-6μ	2-3μ
Y69 (TiCode)	2yr.	A	I.D.	G/G	<1-5μ	<1μ
			O.D.	G/G	<1-5μ	1μ
		B	I.D.	G/M	<1-3μ	1μ
			O.D.	G/L	<1-3μ	2μ
Y85 (Pb)	2yr.	A	I.D.	G/G	<1-2μ	<1μ
			O.D.	G/G	<1-2μ	2μ
		B	I.D.	G/M	<1-2μ	1-2μ
			O.D.	G/L	<1-2μ	1-2μ
Y101 (A216)	2yr.	A	I.D.	G/G	<1-3μ	<1μ
			O.D.	G/G	<1-2μ	1-2μ
		B	I.D.	G/M	<1-3μ	<1μ
			O.D.	G/L	<1-1μ	<1μ

(G/G-glass/glass interface, G/L-glass/liquid interface, G/M-glass/metal interface)

Figure 1

WIPP MIIT Sample Assembly

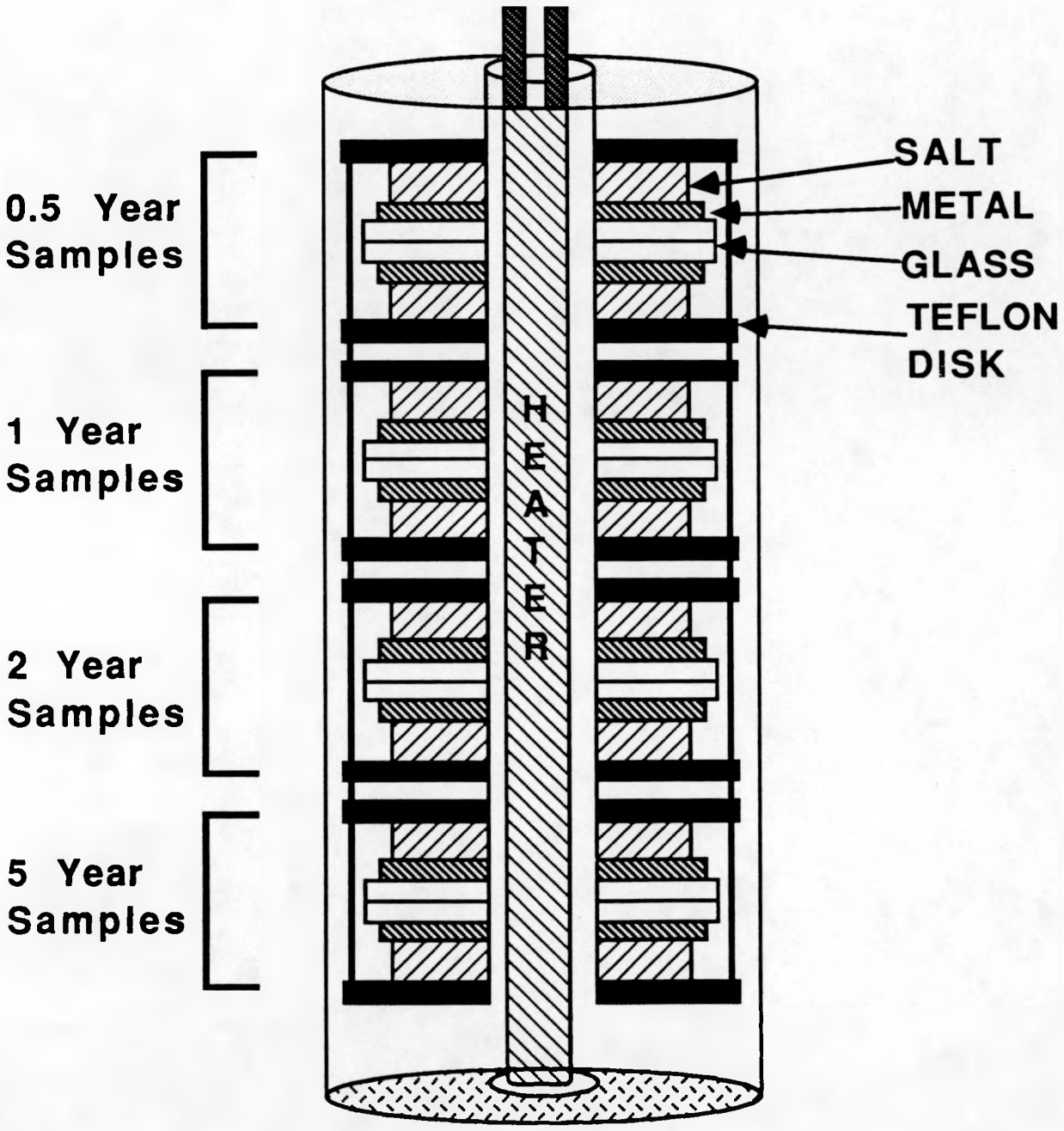
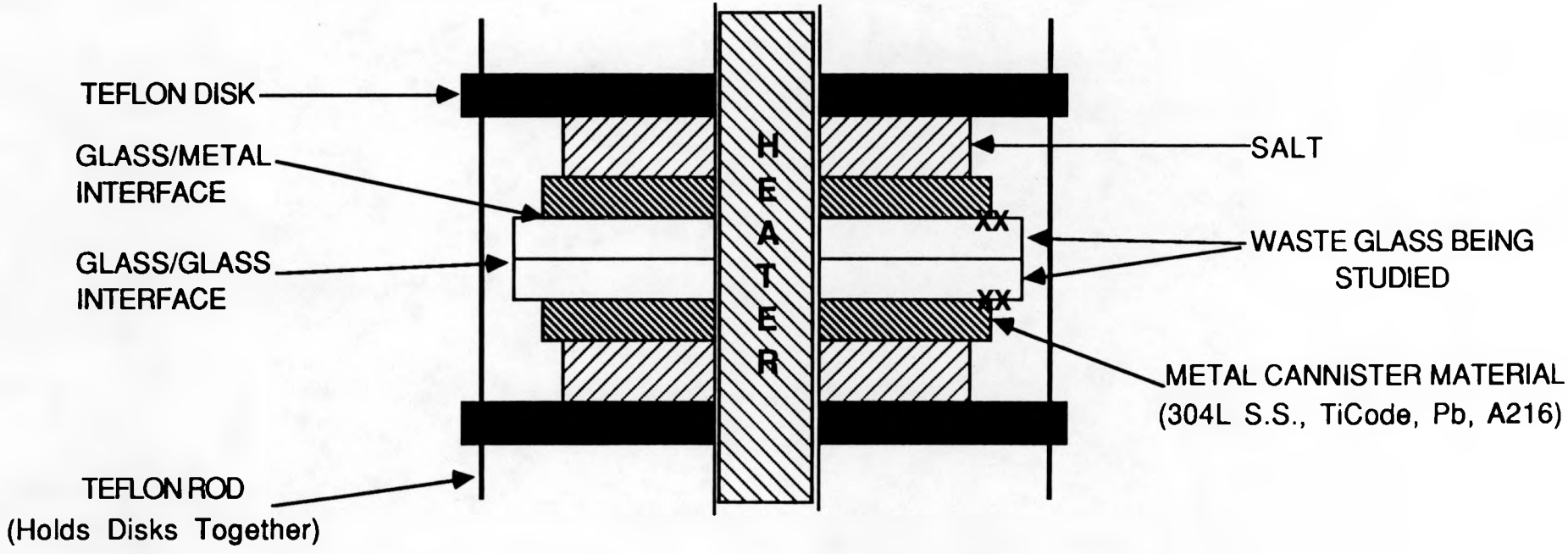


Figure 2

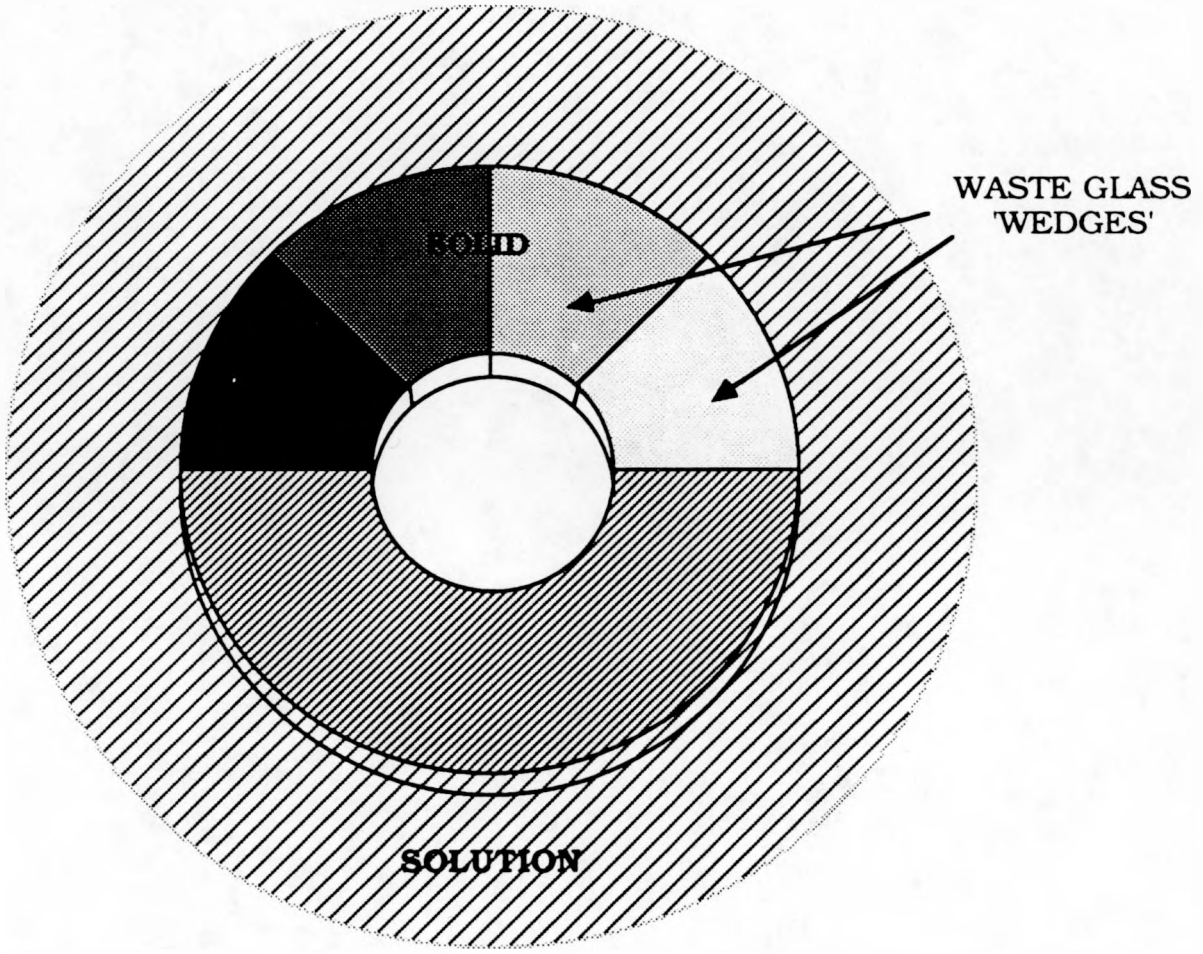
ENLARGED SECTION OF WIPP MIIT SAMPLE ASSEMBLY



XX - Denotes Polished Surfaces

Figure 3 (ref. 8)

SRL Y Glass 'Pineapple Slice'



SOLUTION ANALYSES

☑ ICP, AA, DCP

SOLID ANALYSES IN PROGRESS

■ WAXD
▩ SEM/EDX
□ FTIRRS
■ SIMS
▨ LIBRARY

Figure 4

TOP VIEW OF PINEAPPLE WEDGE

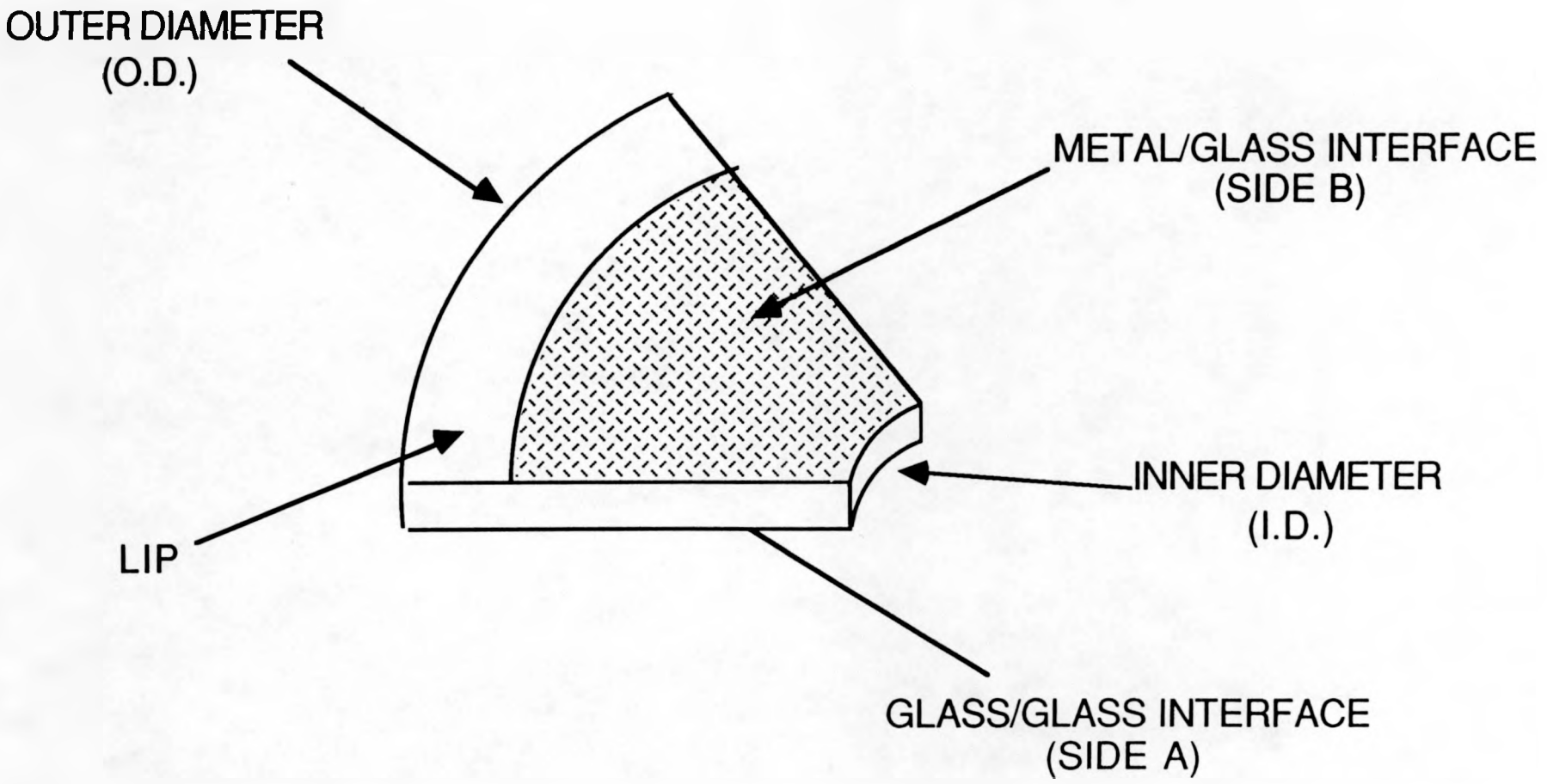


Figure 5 (ref. 9)

Surface Analyses of SRL Y Leached Glass Surface Layers

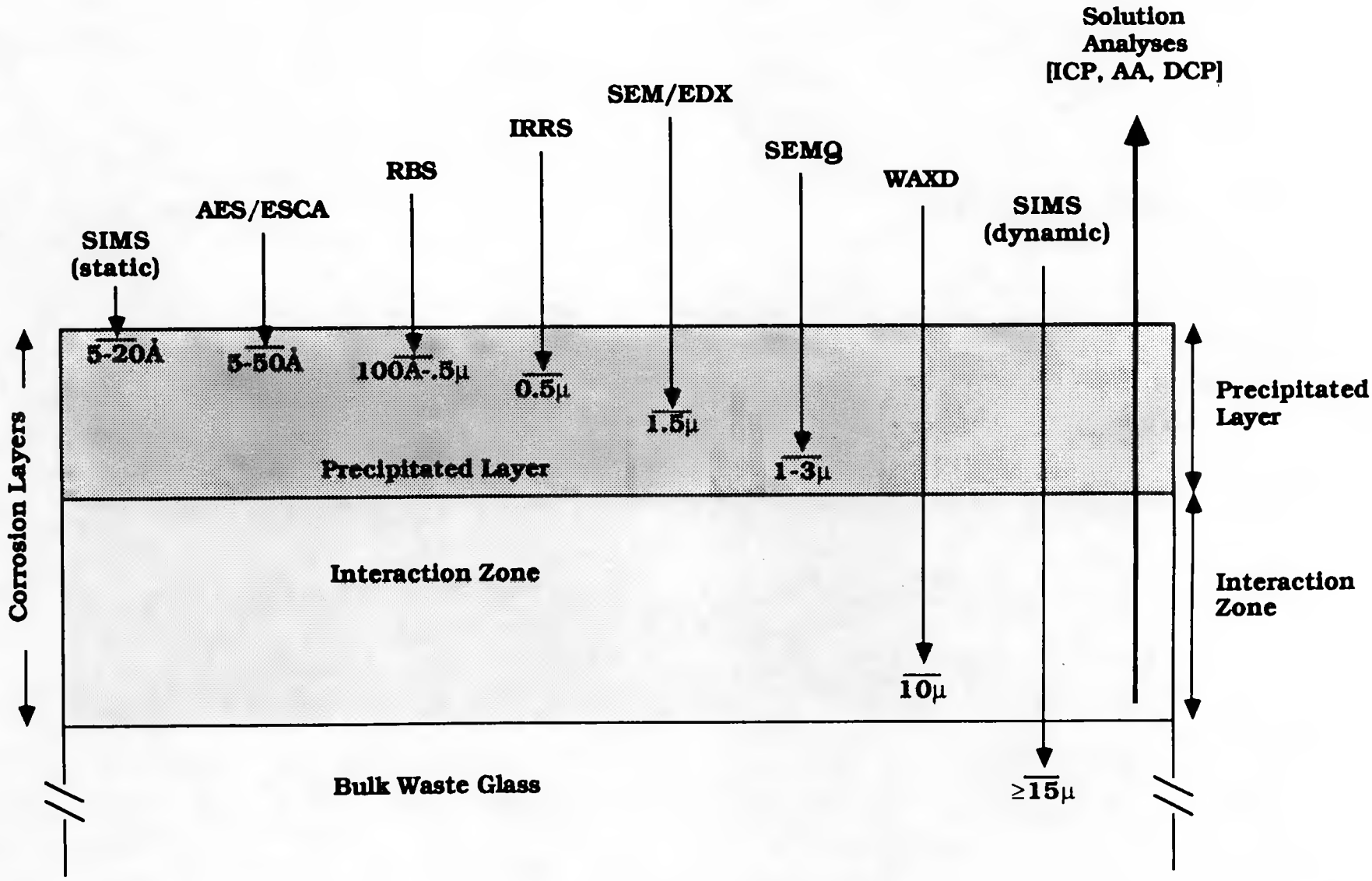


Figure 6

SIDE VIEW OF WASTE GLASS WEDGE

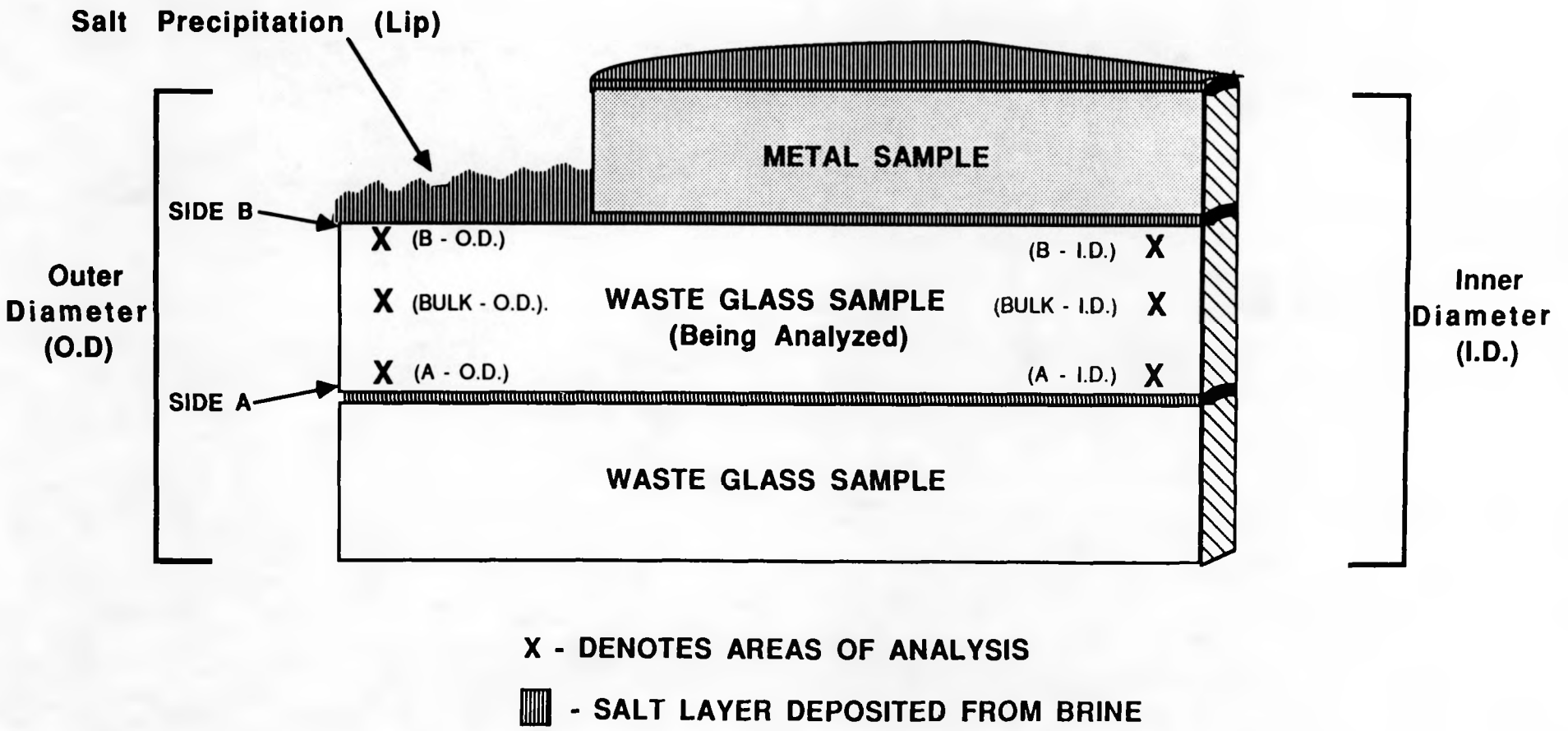


Figure 7A through Figure 7V

SEM/EDX ANALYSES OF MIIT GLASSES Y-49, Y-51, Y-53, Y-69, Y-85, AND Y-103

Figure 7A

SEM/EDX Cross Section
Sample Y-49 (Side A, O.D.)

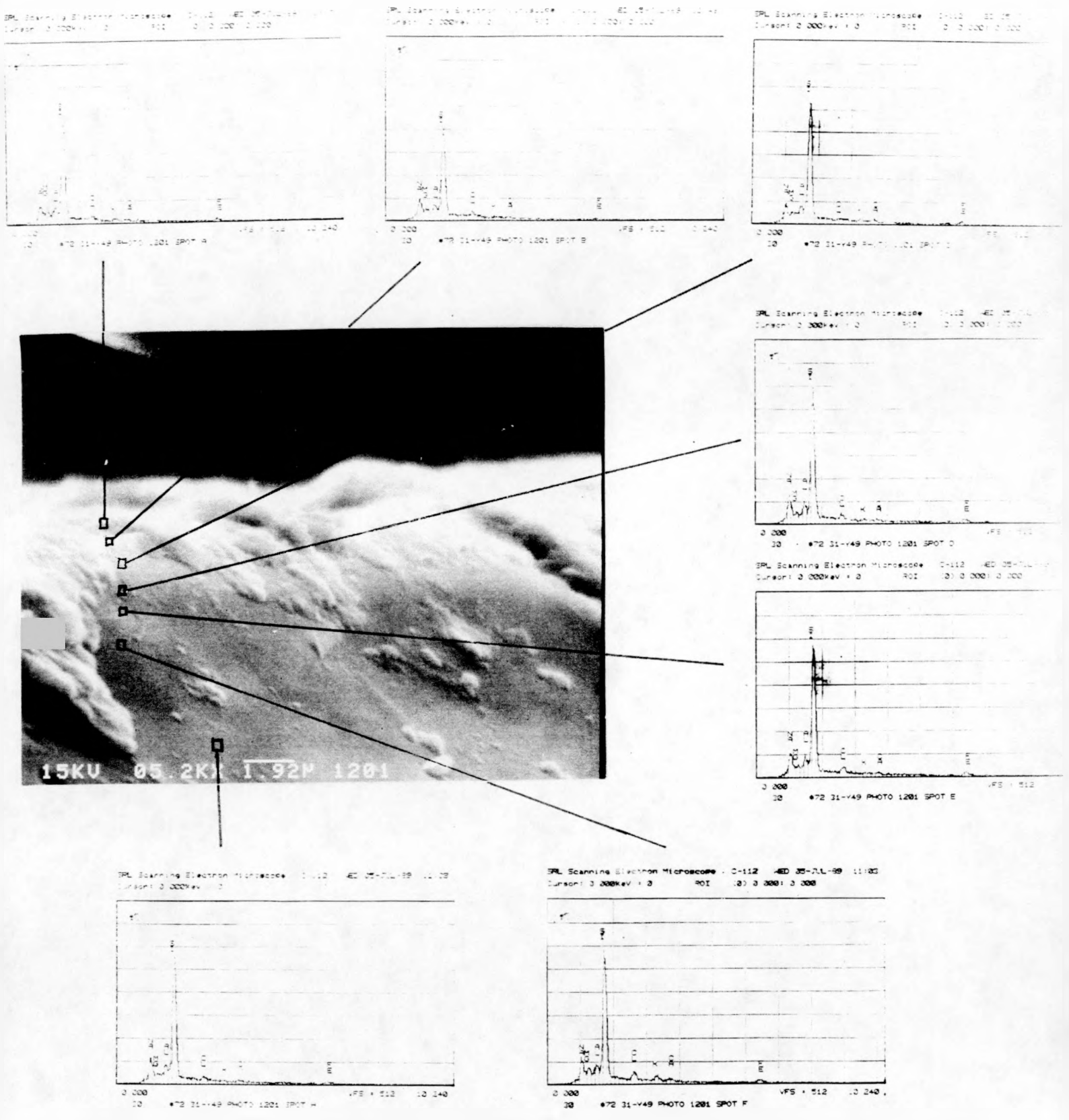


Figure 7B

SEM/EDX Cross Section
Sample Y-49 (Side B, O.D.)

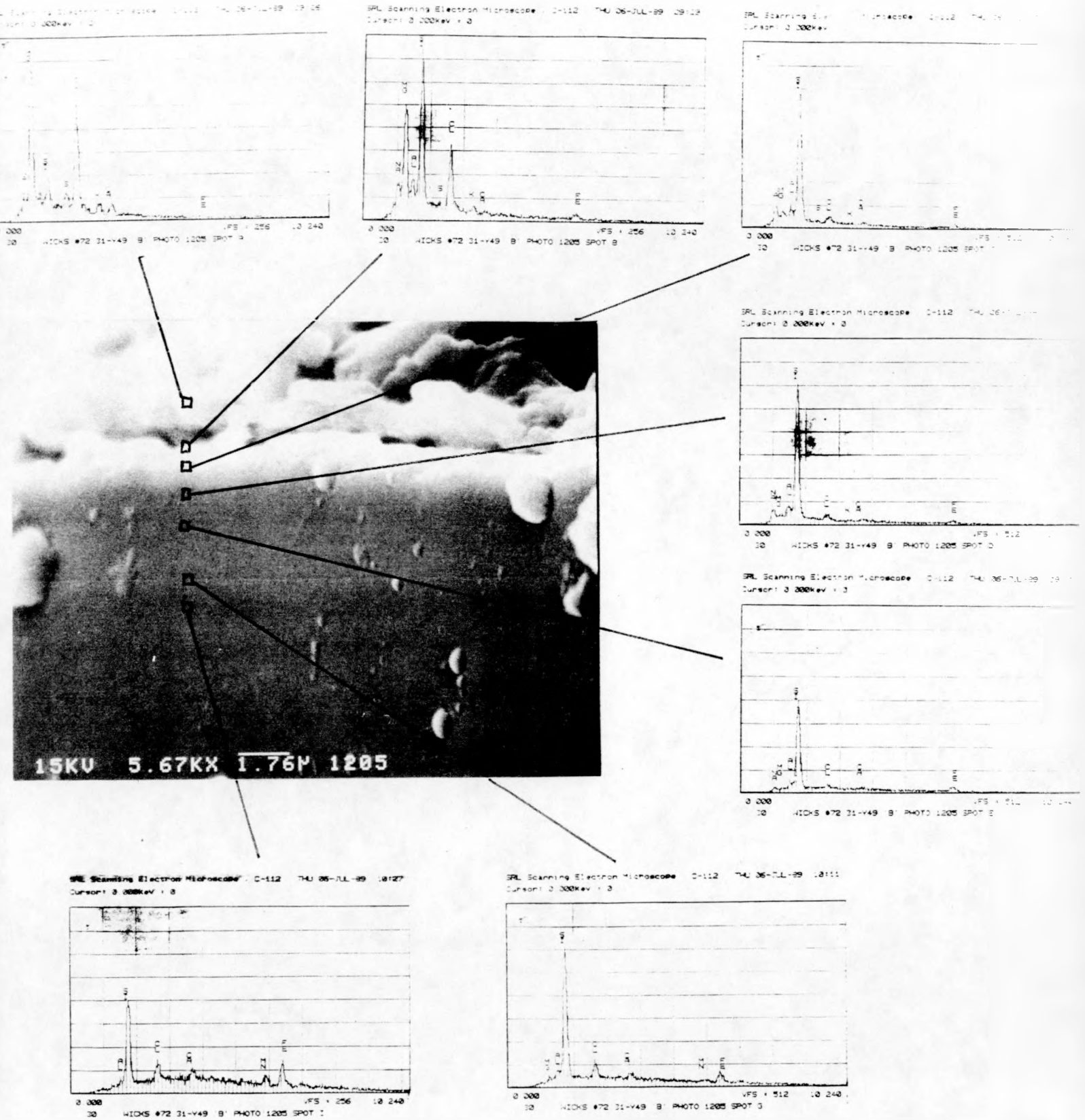


Figure 7C

SEM/EDX Cross Section
Sample Y-51 (Side A, O.D.)

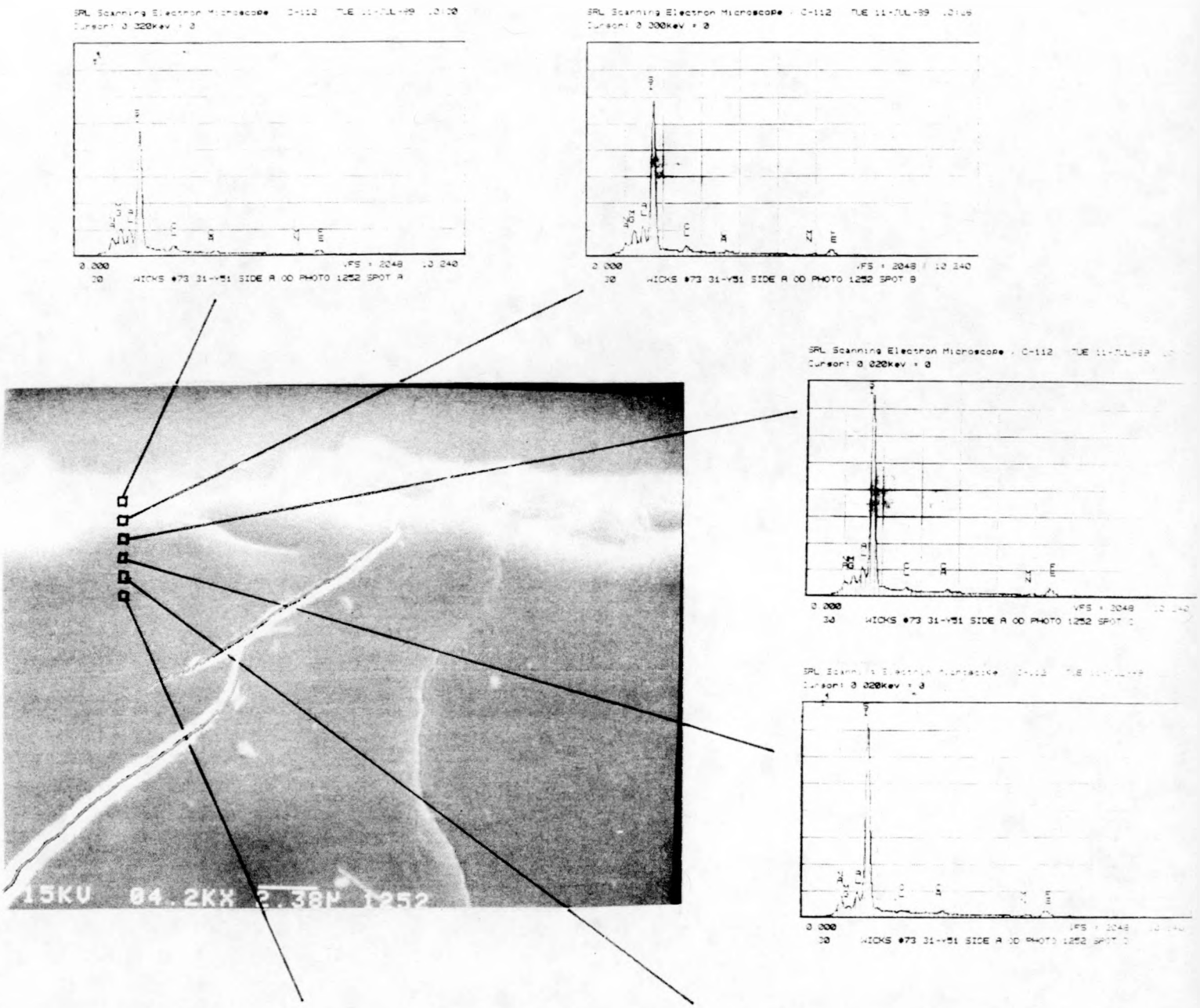


Figure 7D

SEM/EDX Cross Section
Sample Y-51 (Side A, I.D.)

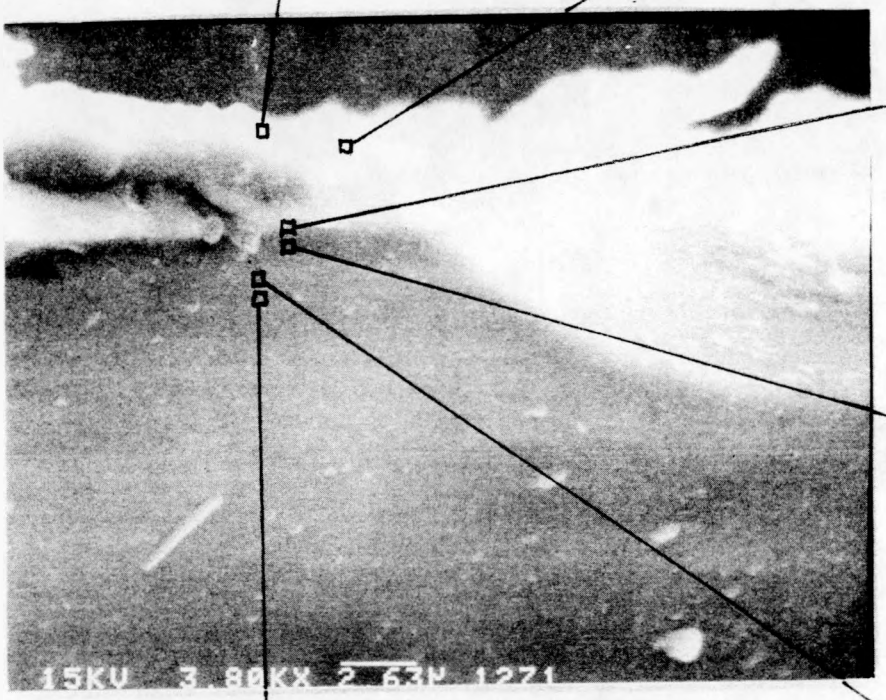
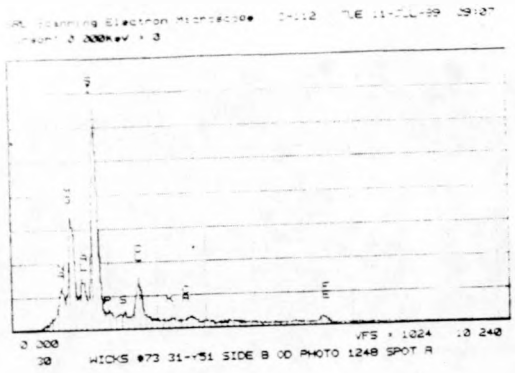
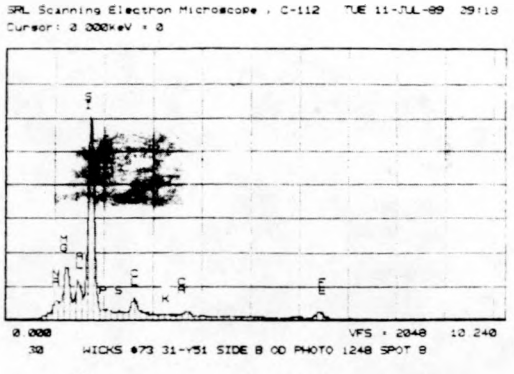
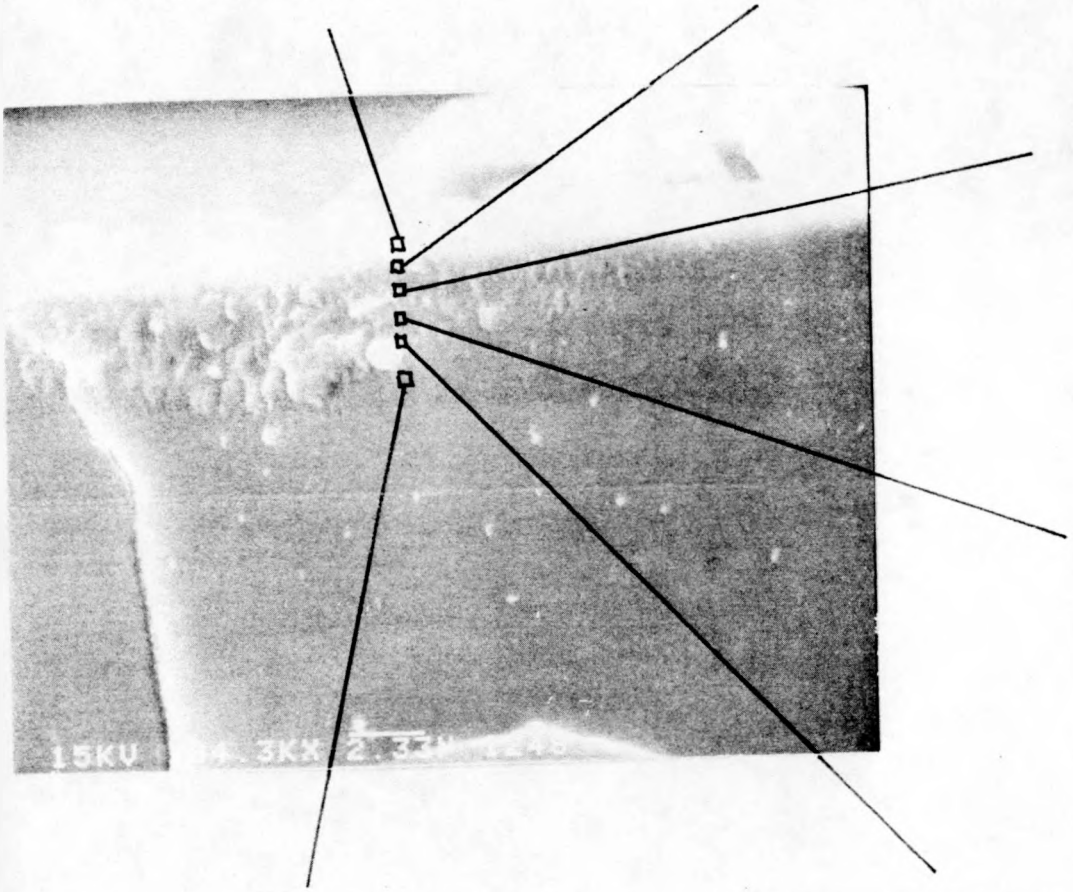


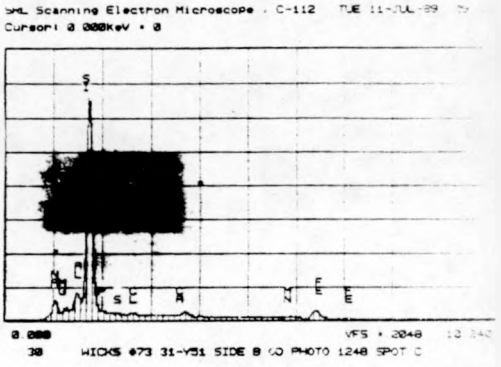
Figure 7E

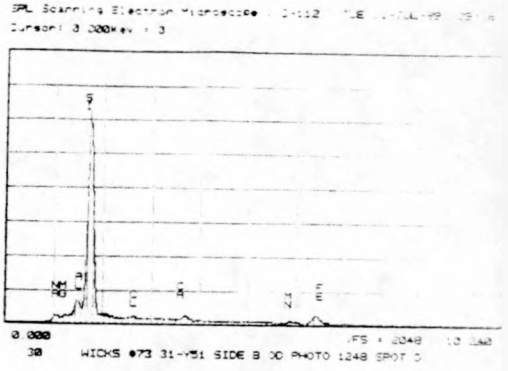
SEM/EDX Cross Section
Sample Y-51 (Side B, O.D.)

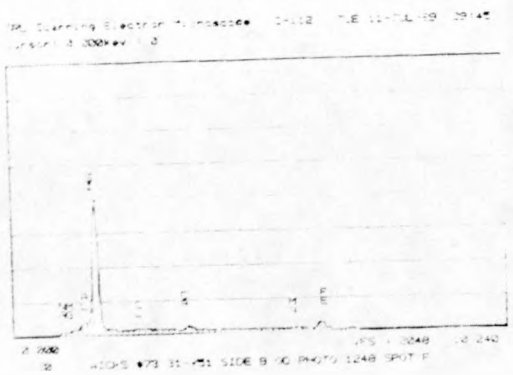












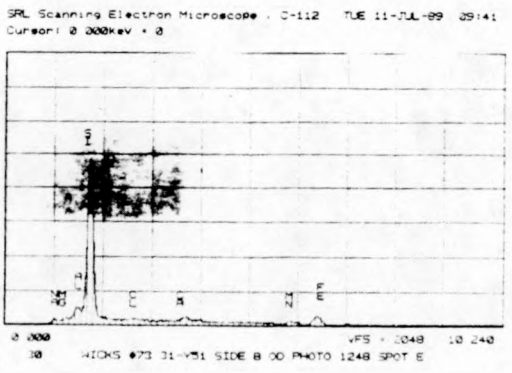
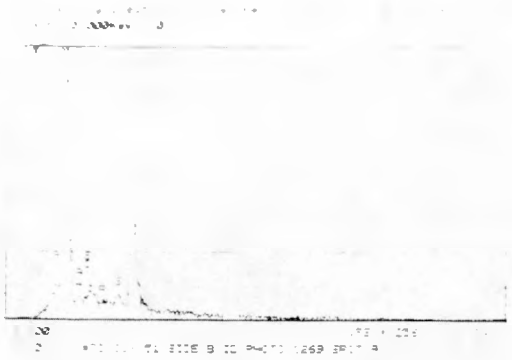
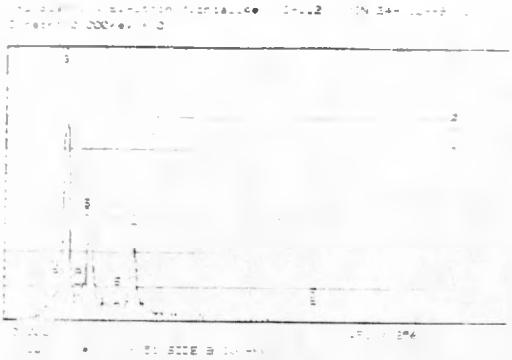
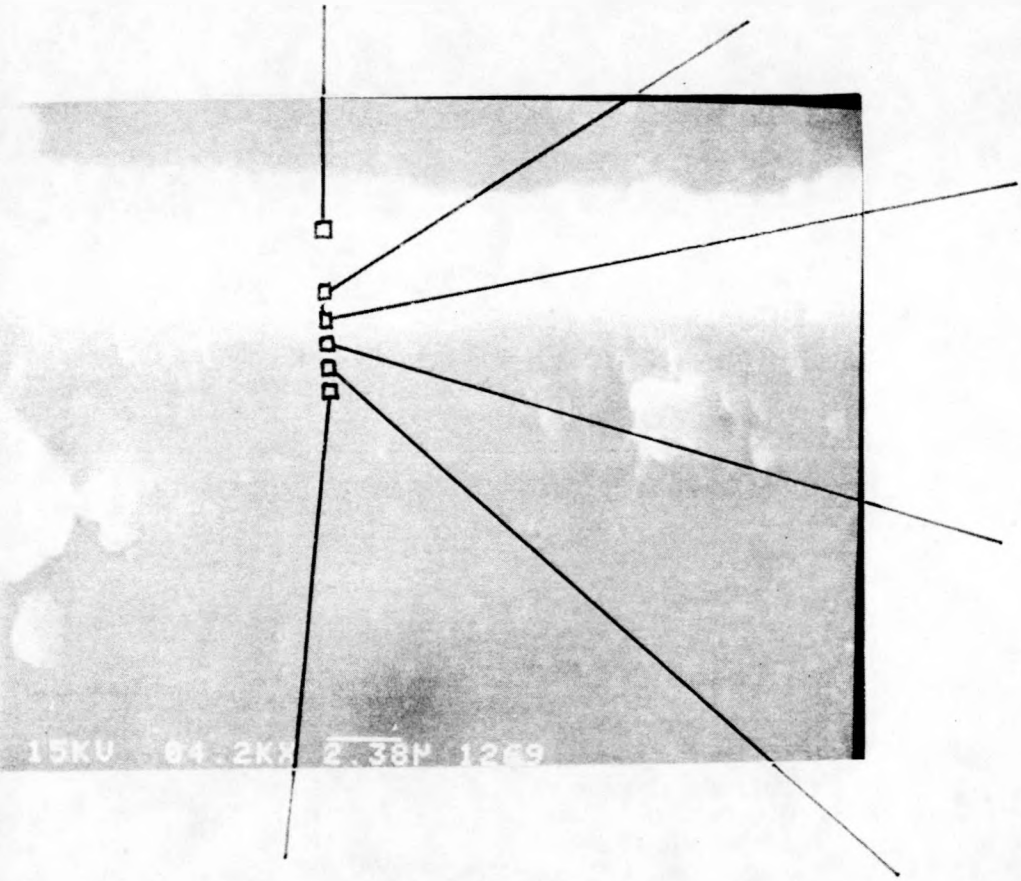


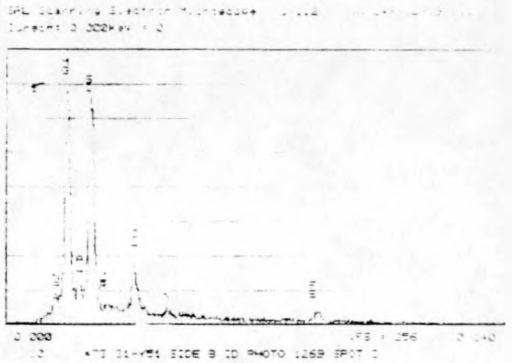
Figure 7F

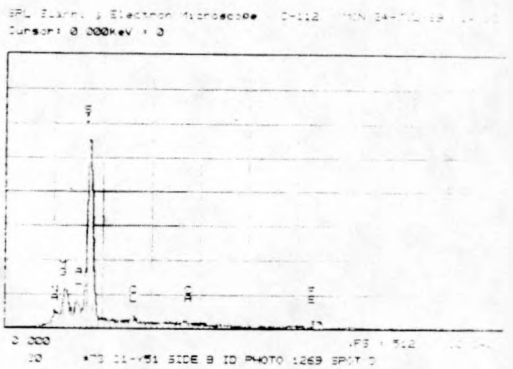
SEM/EDX Cross Section
Sample Y-51 (Side B, I.D.)

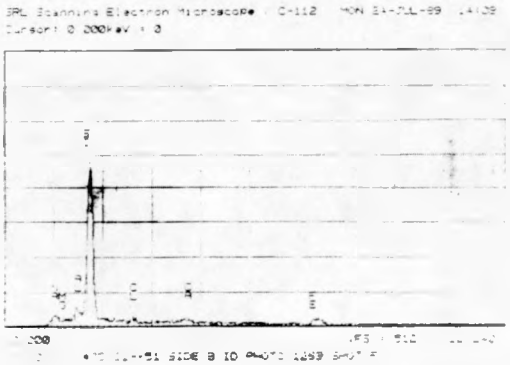












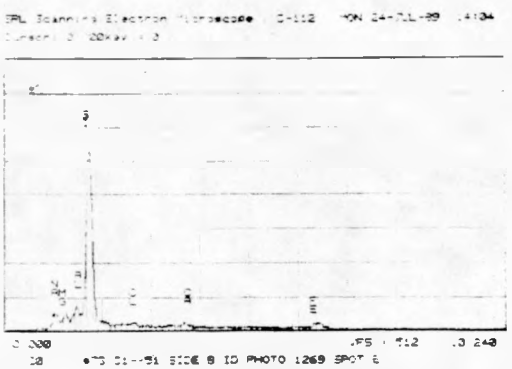


Figure 7G

SEM/EDX Cross Section
Sample Y-53 (Side A, O.D.)

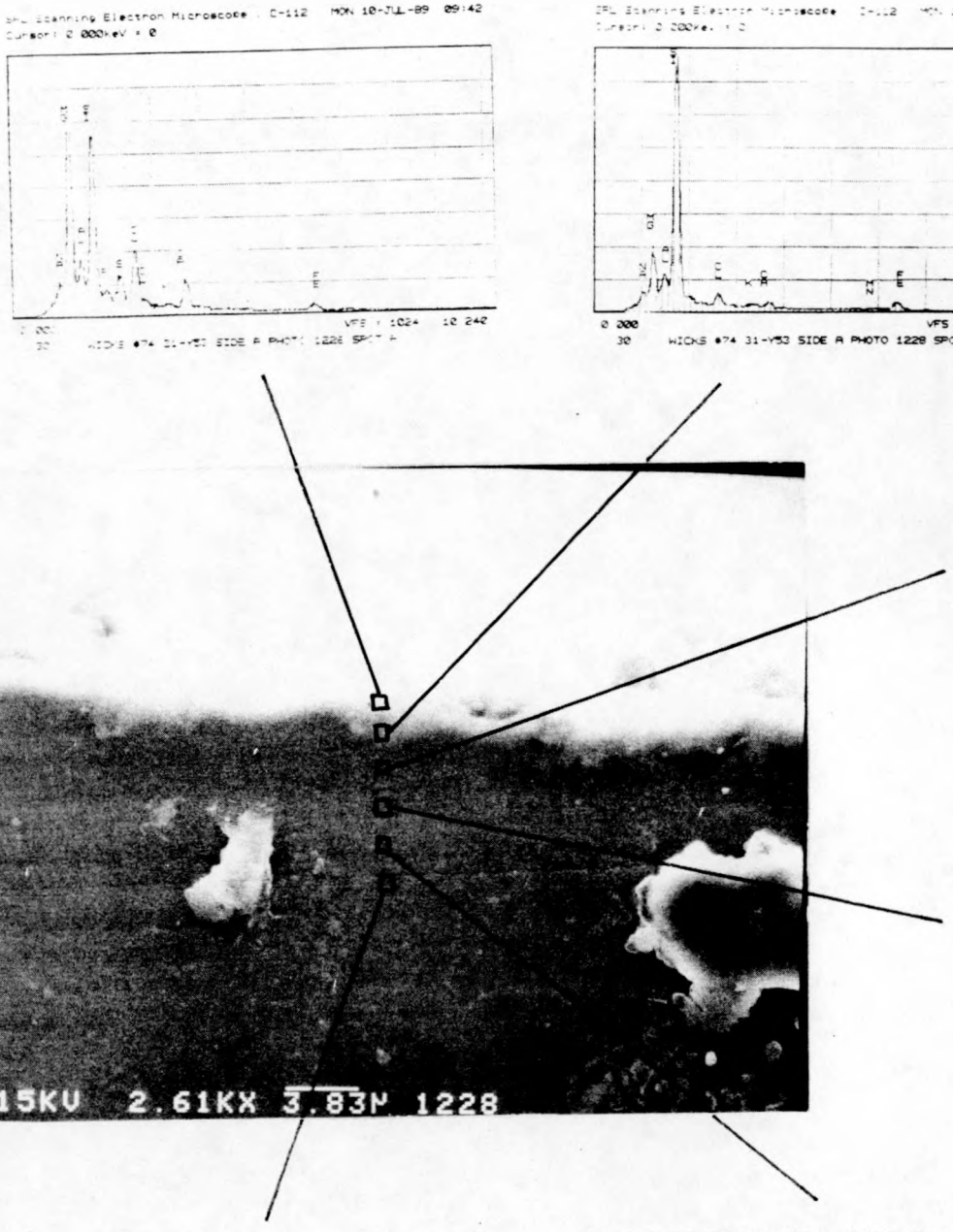
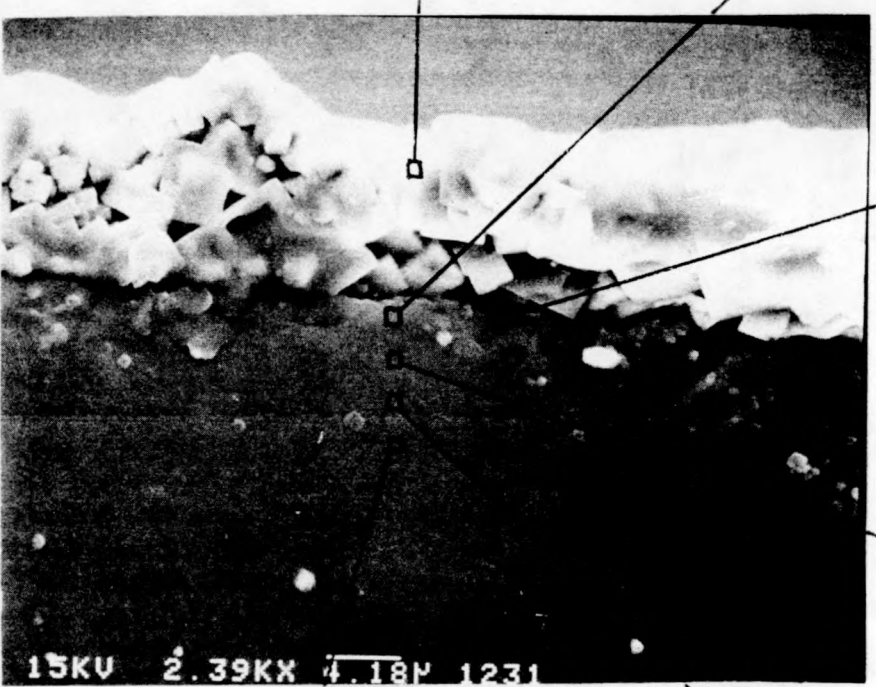
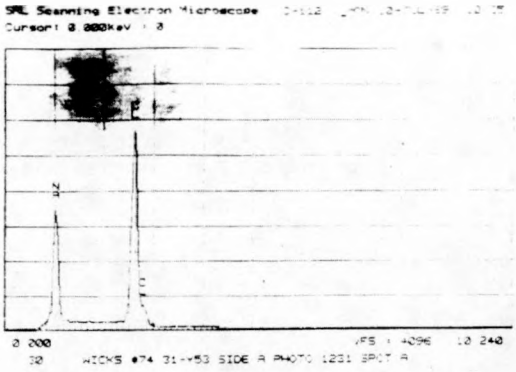
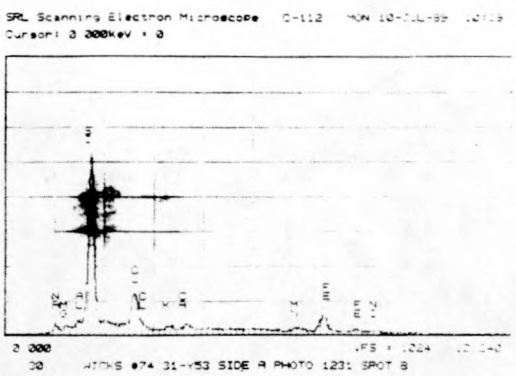


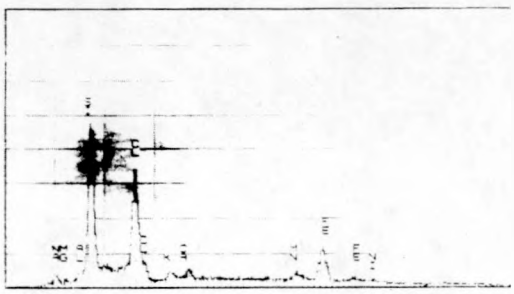
Figure 7H

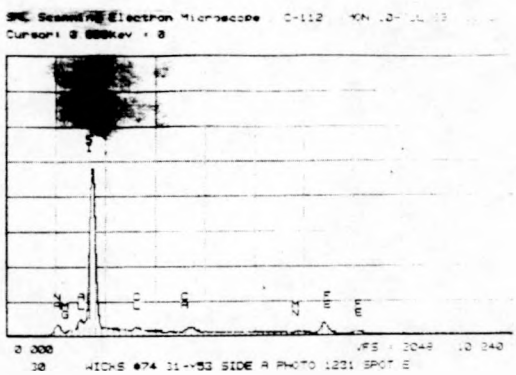
SEM/EDX Cross Section
Sample Y-53 (Side A, I.D.)

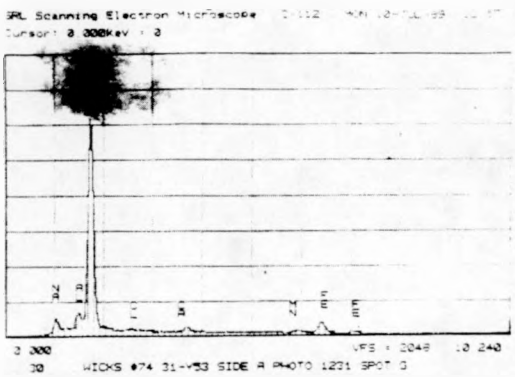












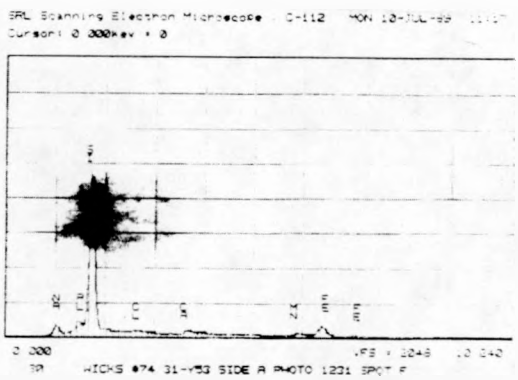
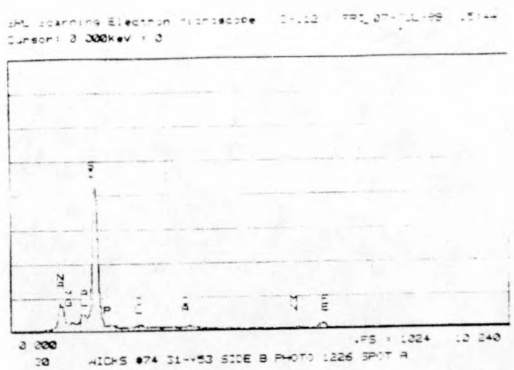
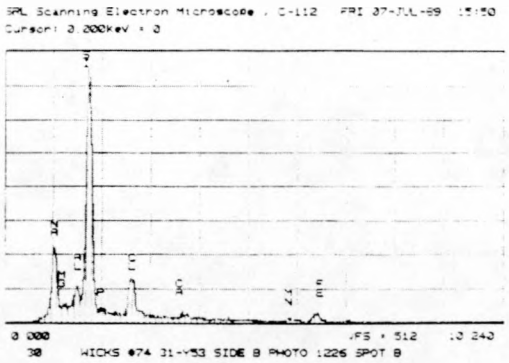
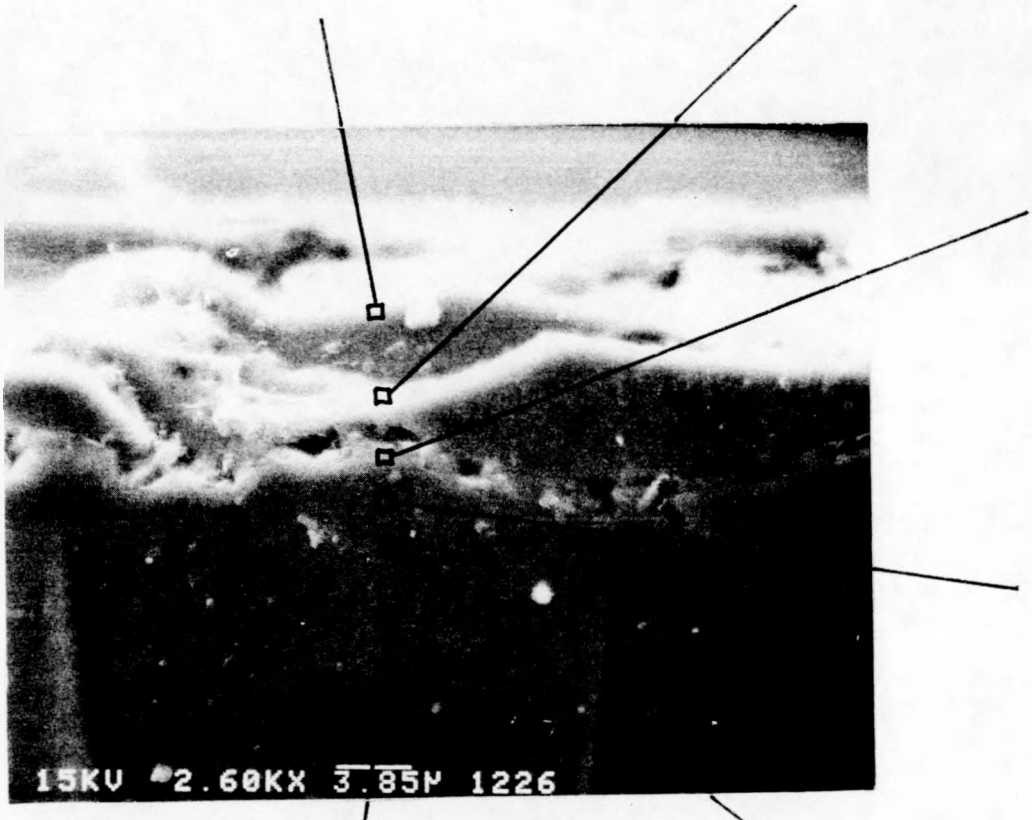


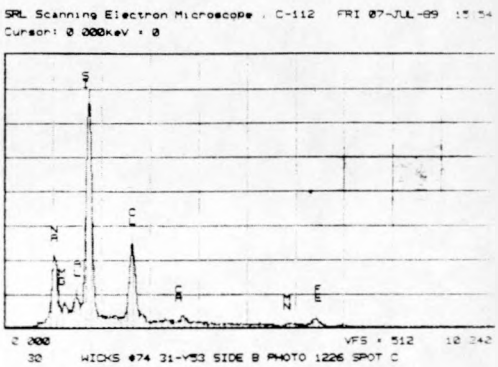
Figure 7I

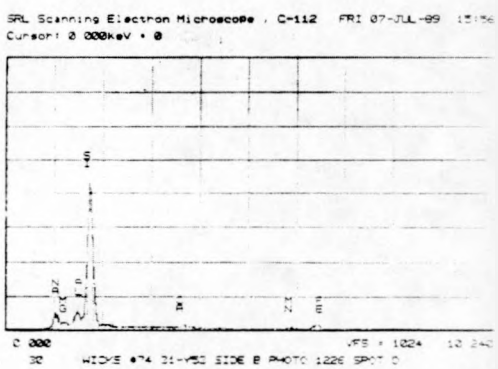
SEM/EDX Cross Section
Sample Y-53 (Side B, O.D.)

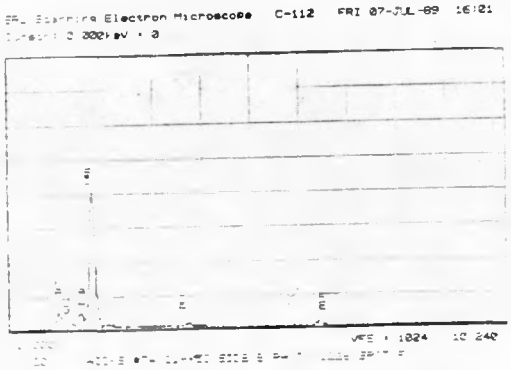












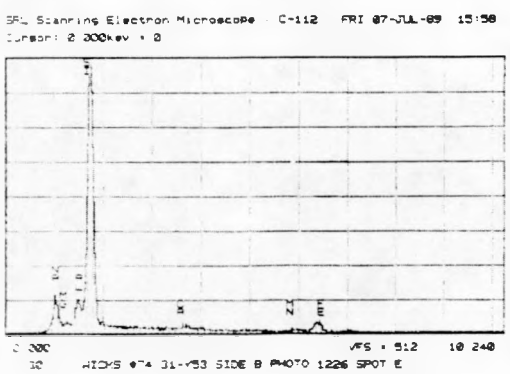


Figure 7J

SEM/EDX Cross Section
Sample Y-53 (Side B, I.D.)

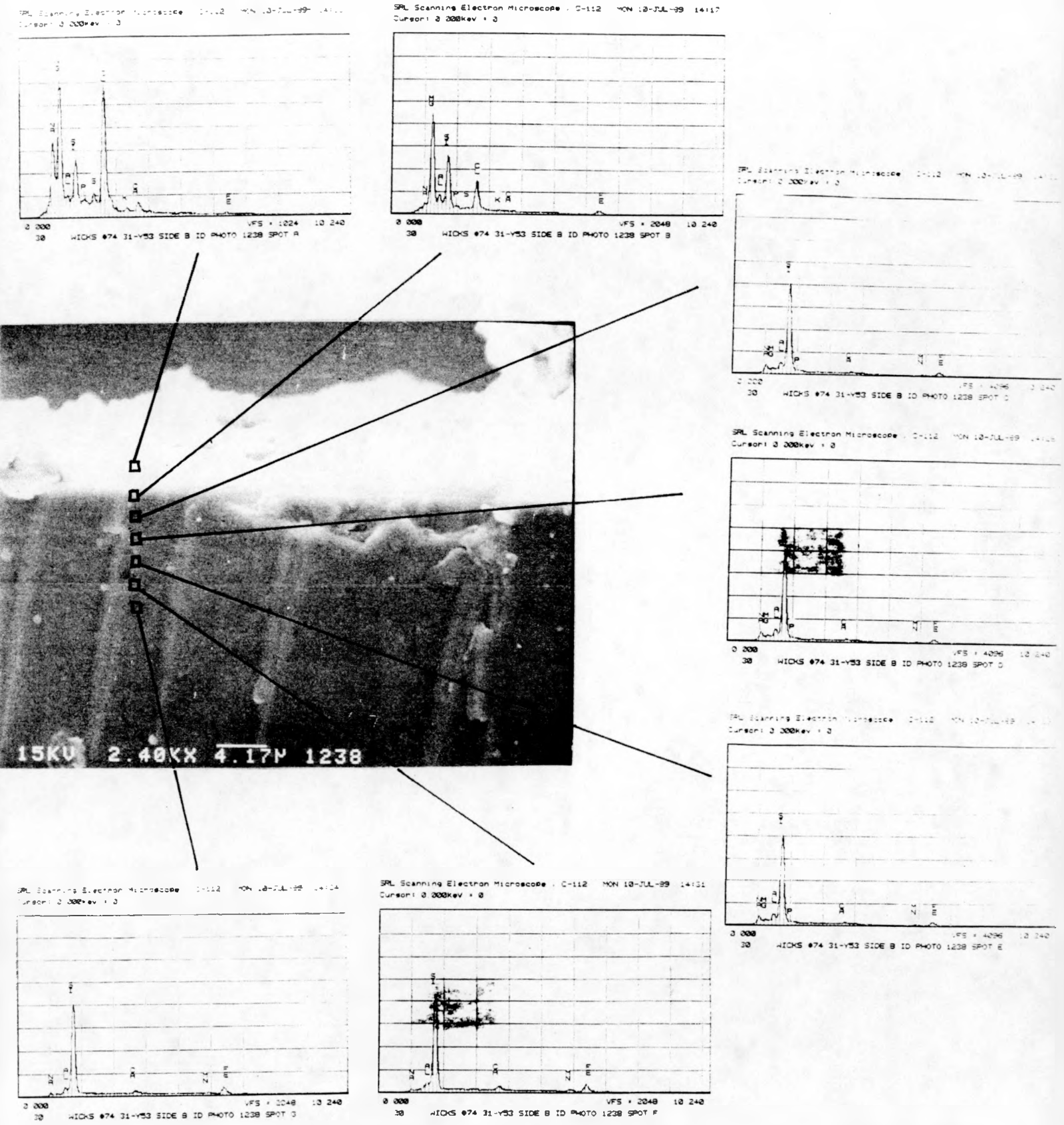


Figure 7K

SEM/EDX Cross Section
Sample Y-69 (Side A, O.D.)

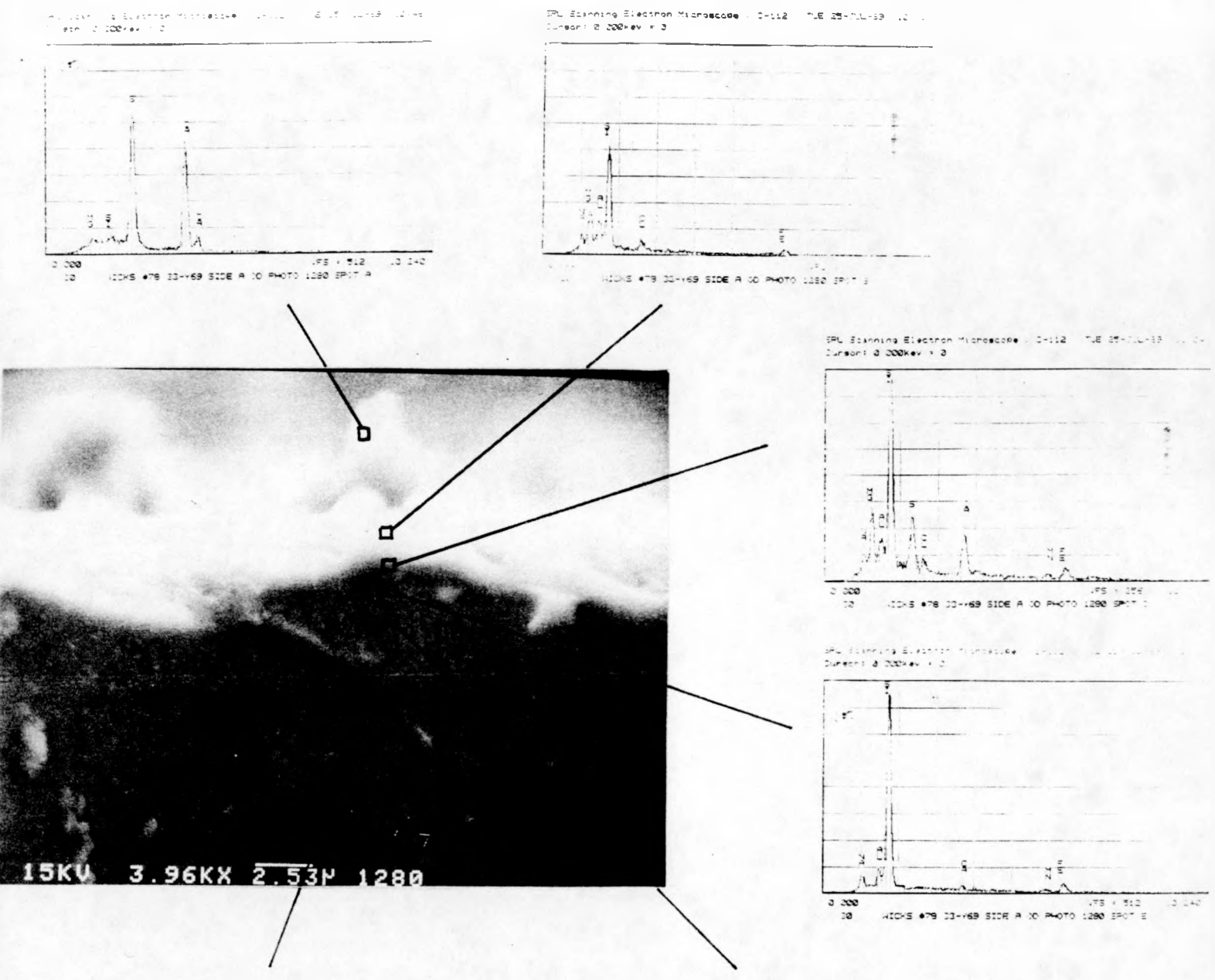


Figure 7L

SEM/EDX Cross Section
Sample Y-69 (Side A, I.D.)

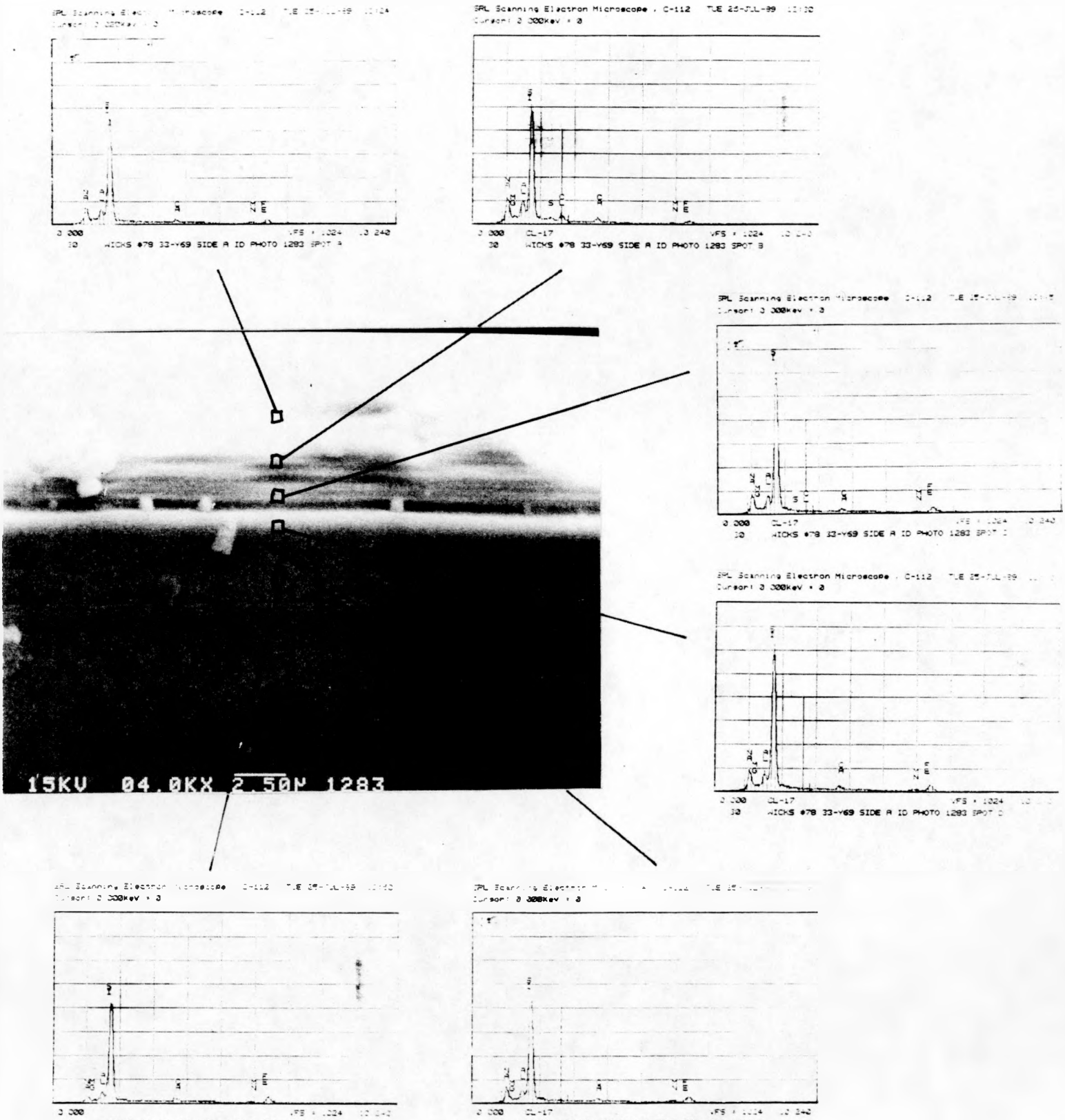


Figure 7M

SEM/EDX Cross Section
Sample Y-69 (Side B, O.D.)

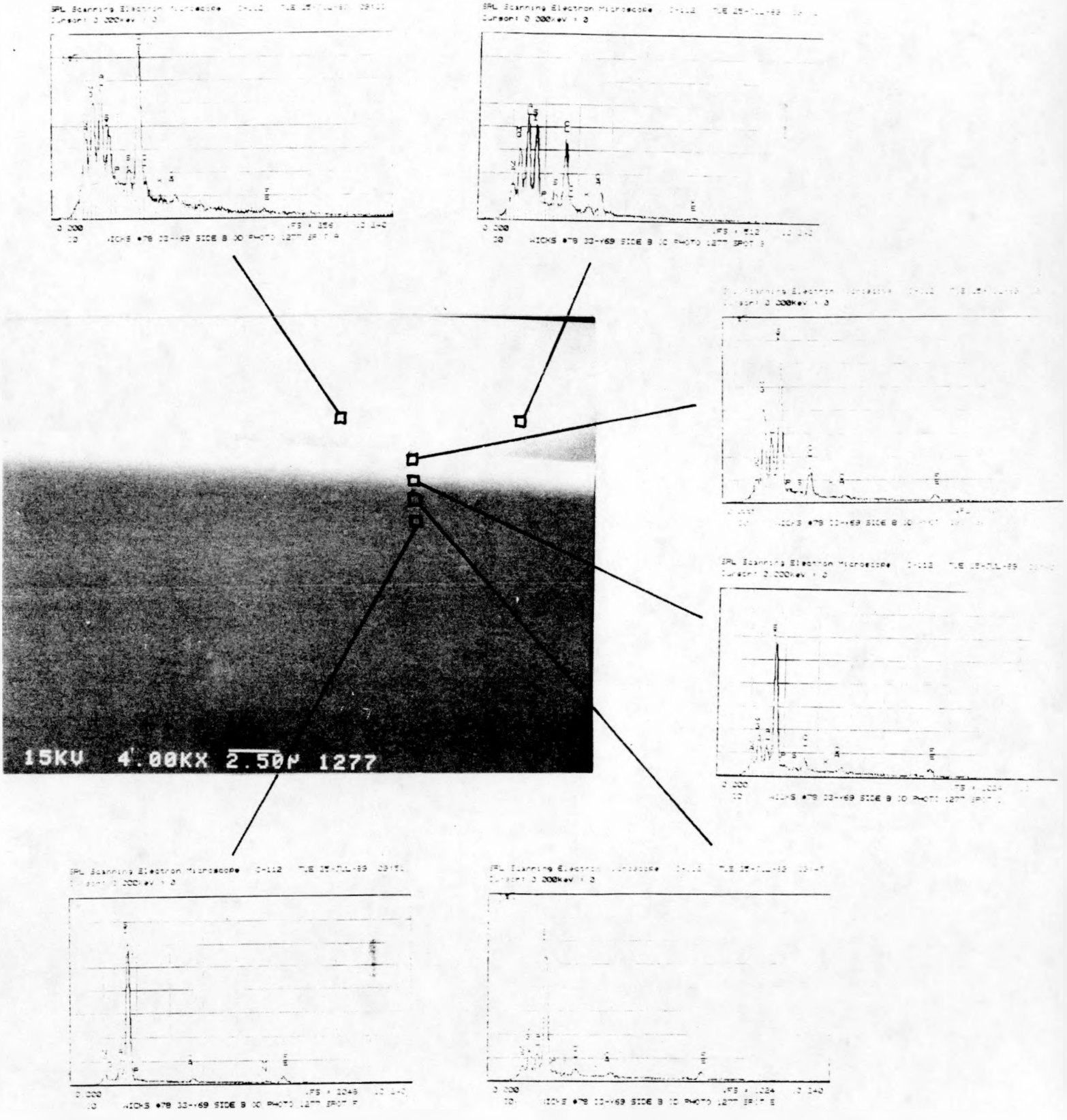


Figure 7N

SEM/EDX Cross Section
Sample Y-69 (Side B, I.D.)

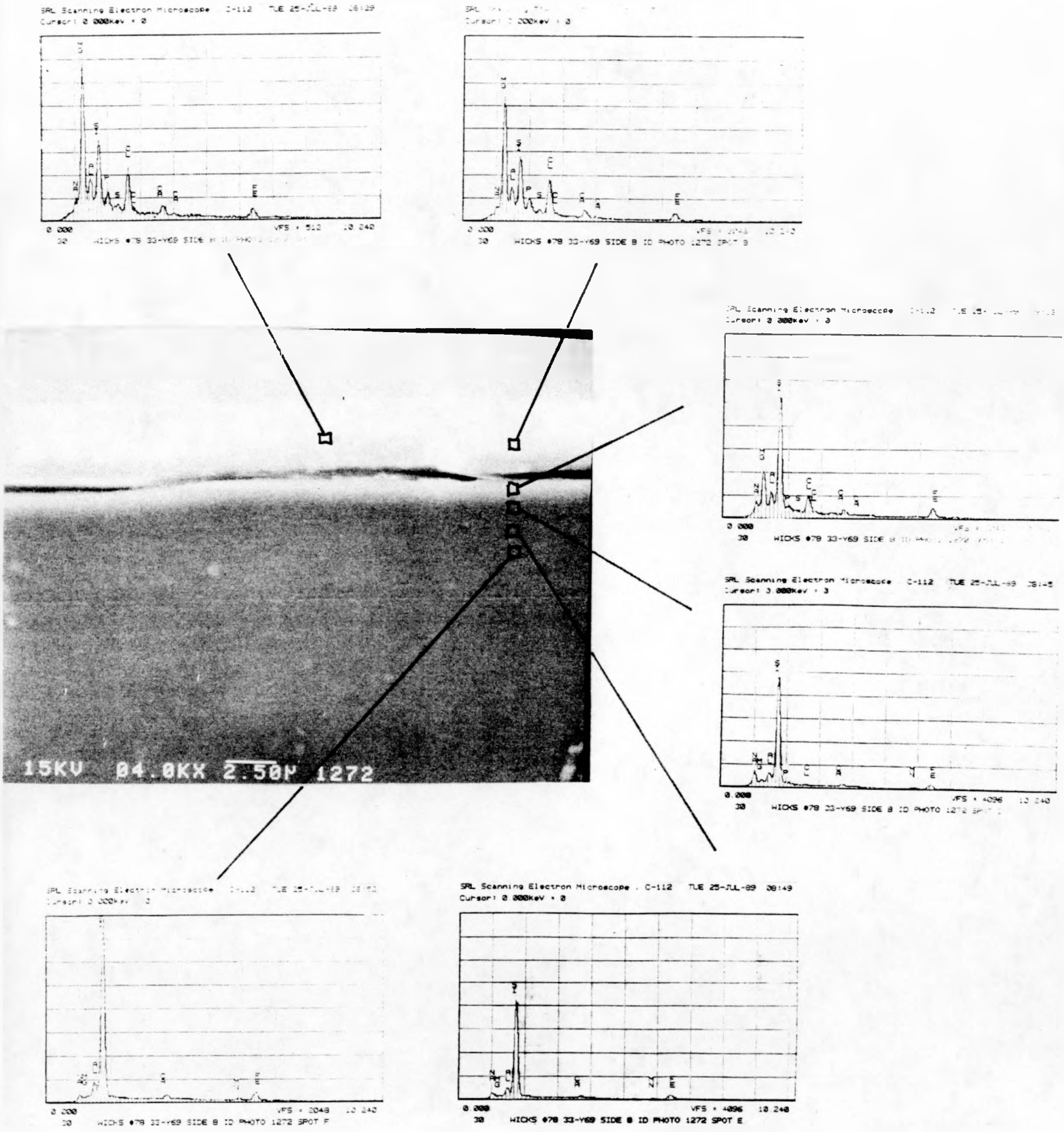


Figure 70

SEM/EDX Cross Section
Sample Y-85 (Side A, O.D.)

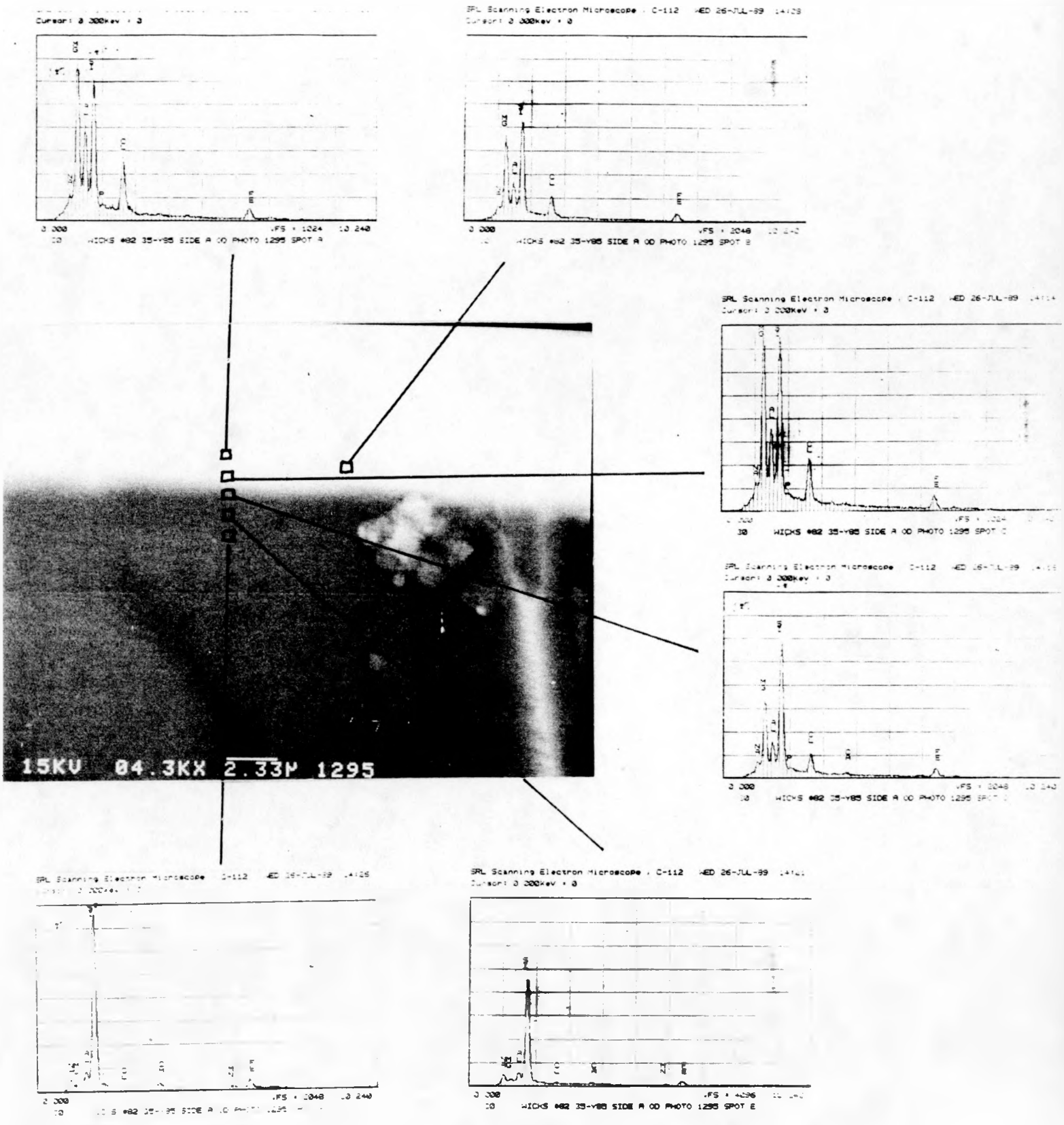


Figure 7P

SEM/EDX Cross Section
Sample Y-85 (Side A, I.D.)

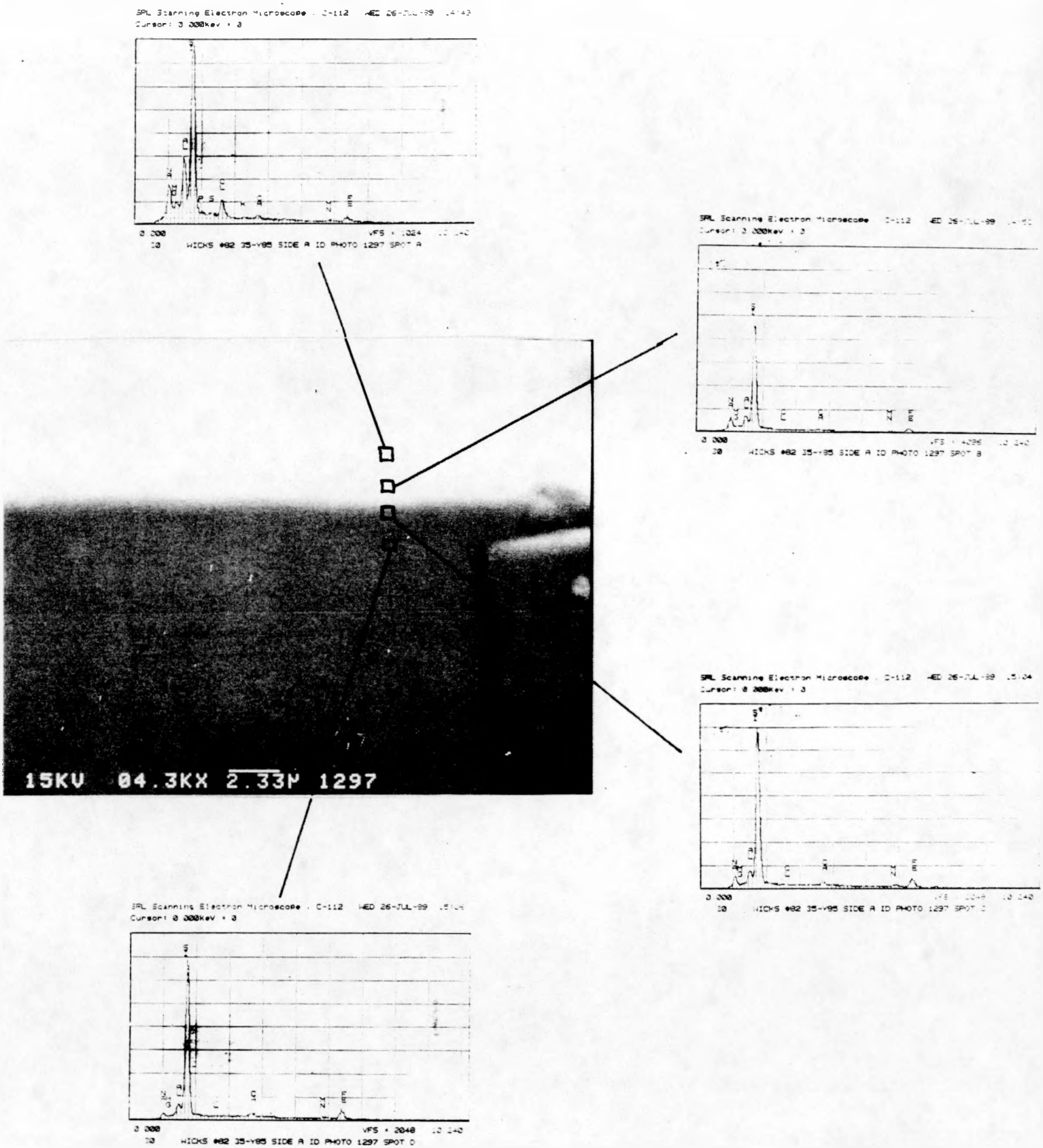
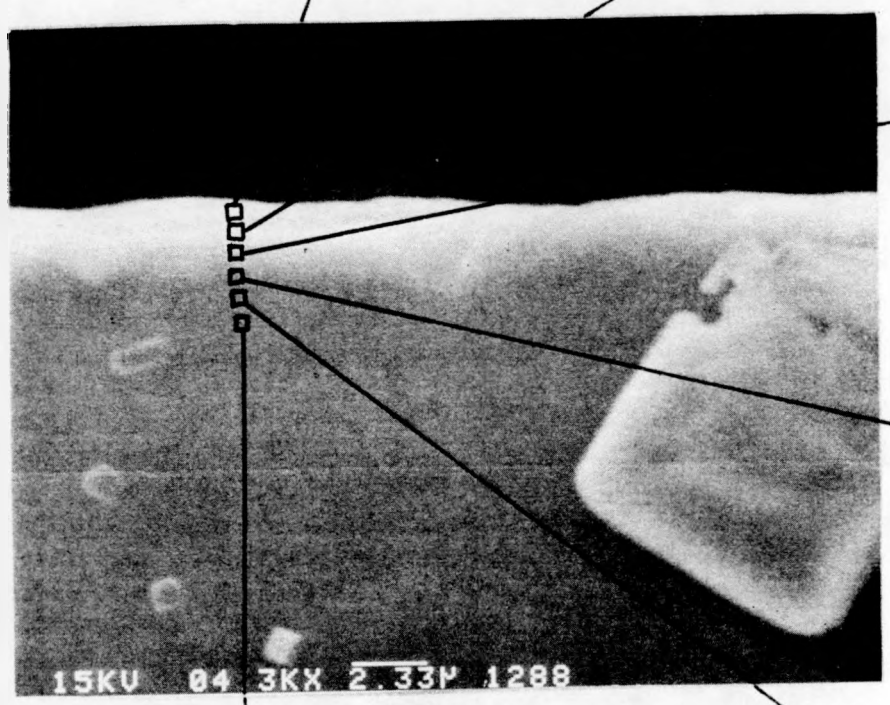
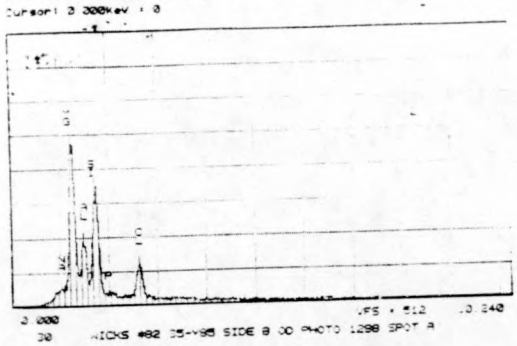
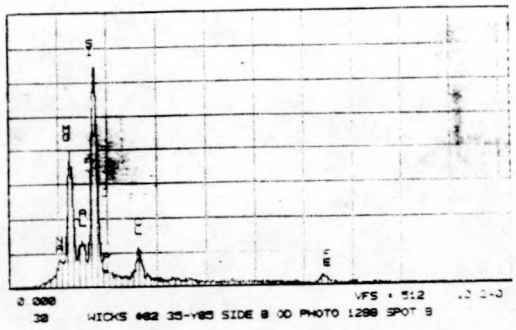


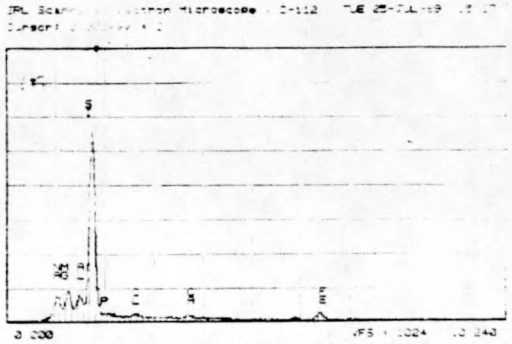
Figure 7Q

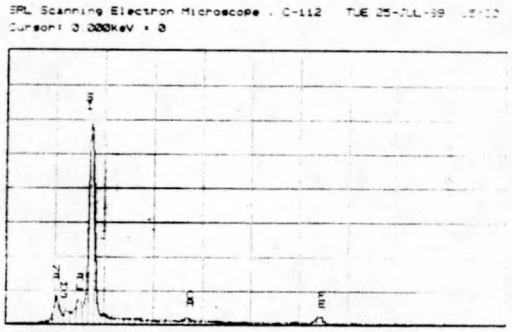
SEM/EDX Cross Section
Sample Y-85 (Side B, O.D.)

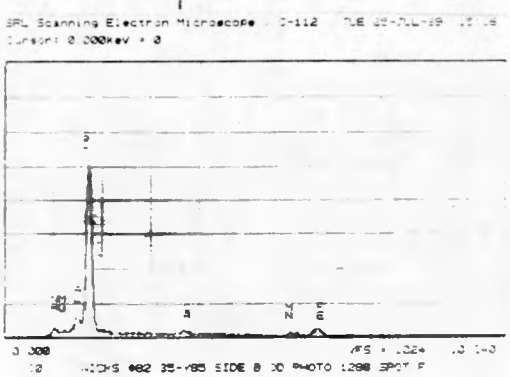












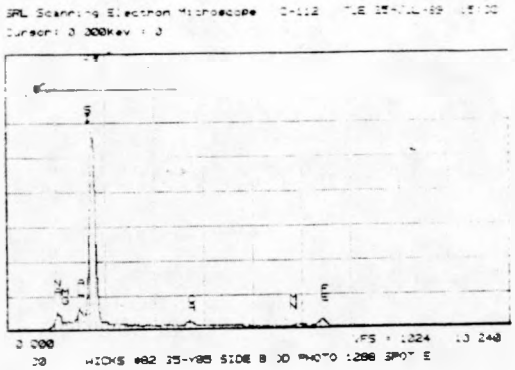


Figure 7R

SEM/EDX Cross Section
Sample Y-85 (Side B, I.D.)

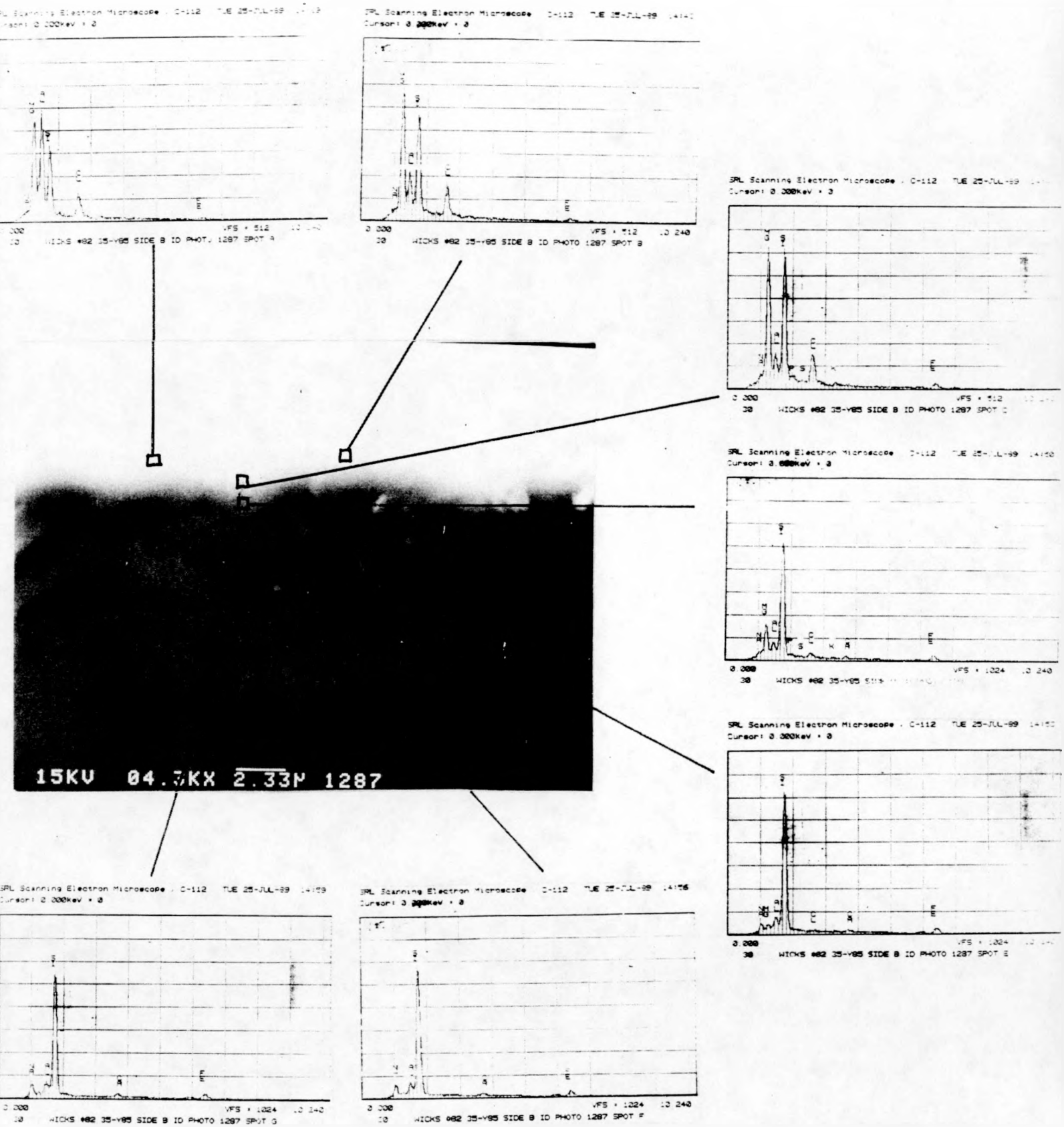


Figure 7S

SEM/EDX Cross Section
Sample Y-103 (Side A, O.D.)

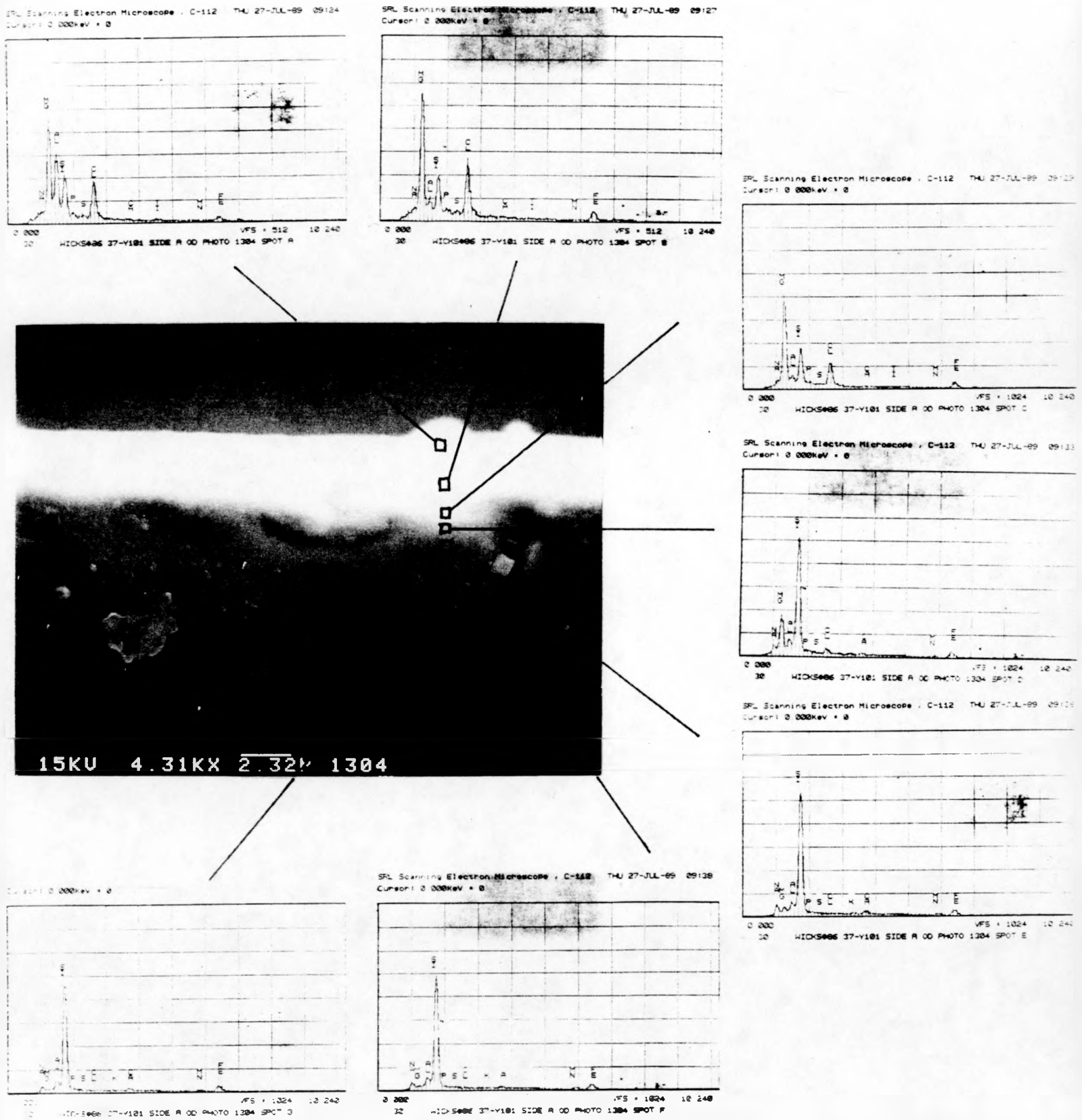
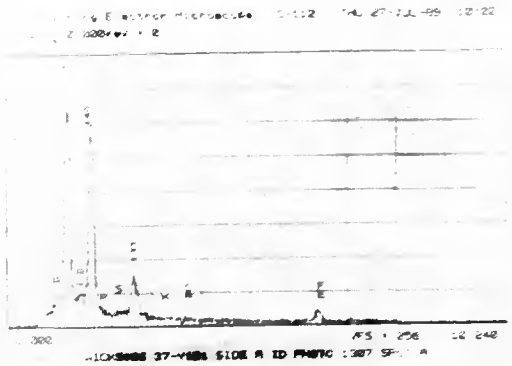
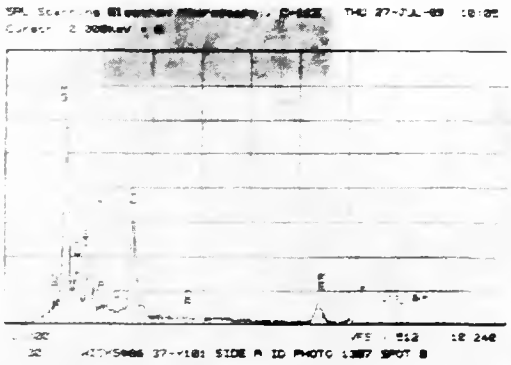
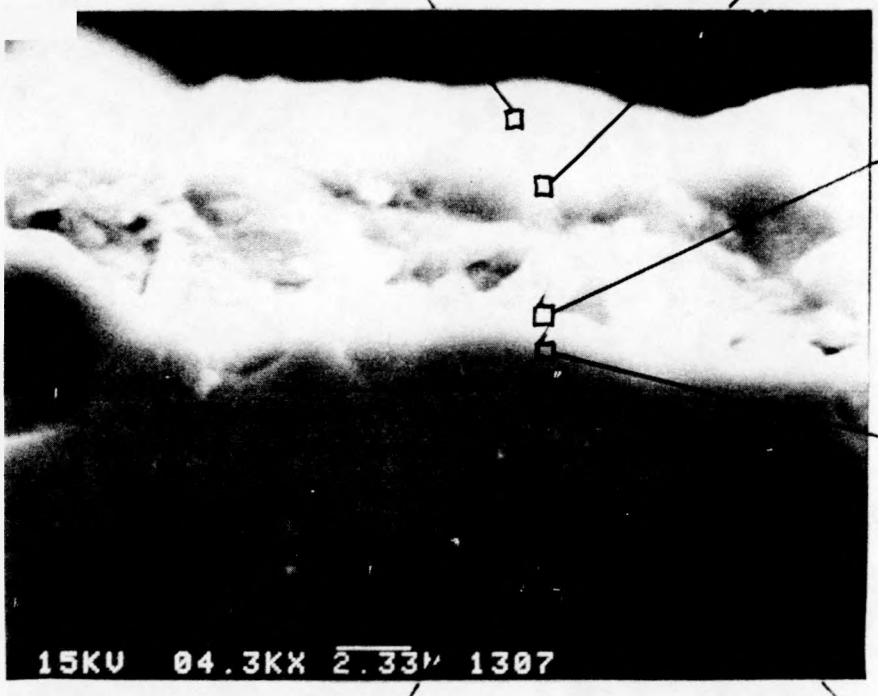


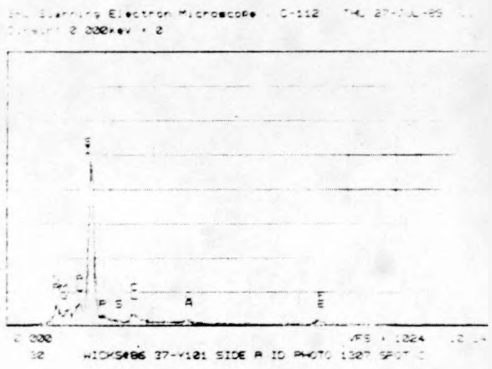
Figure 7T

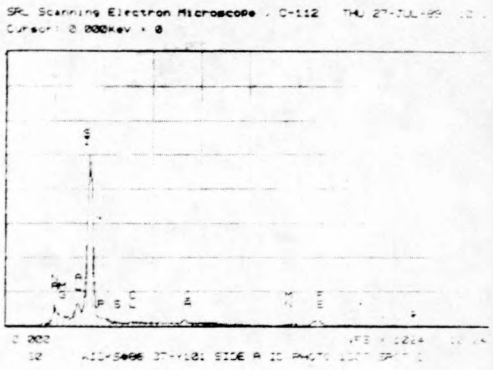
SEM/EDX Cross Section
Sample Y-103 (Side A, I.D.)

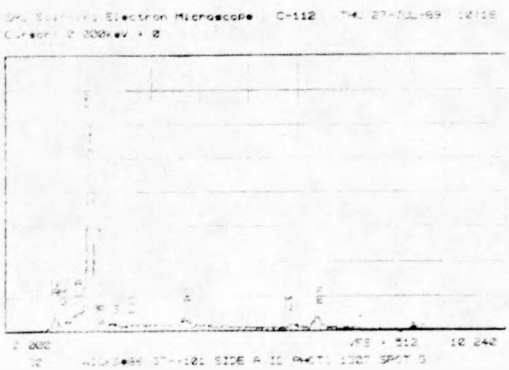












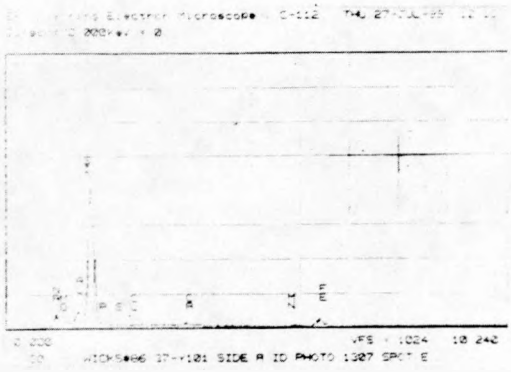


Figure 7U

SEM/EDX Cross Section
Sample Y-103 (Side B, O.D.)

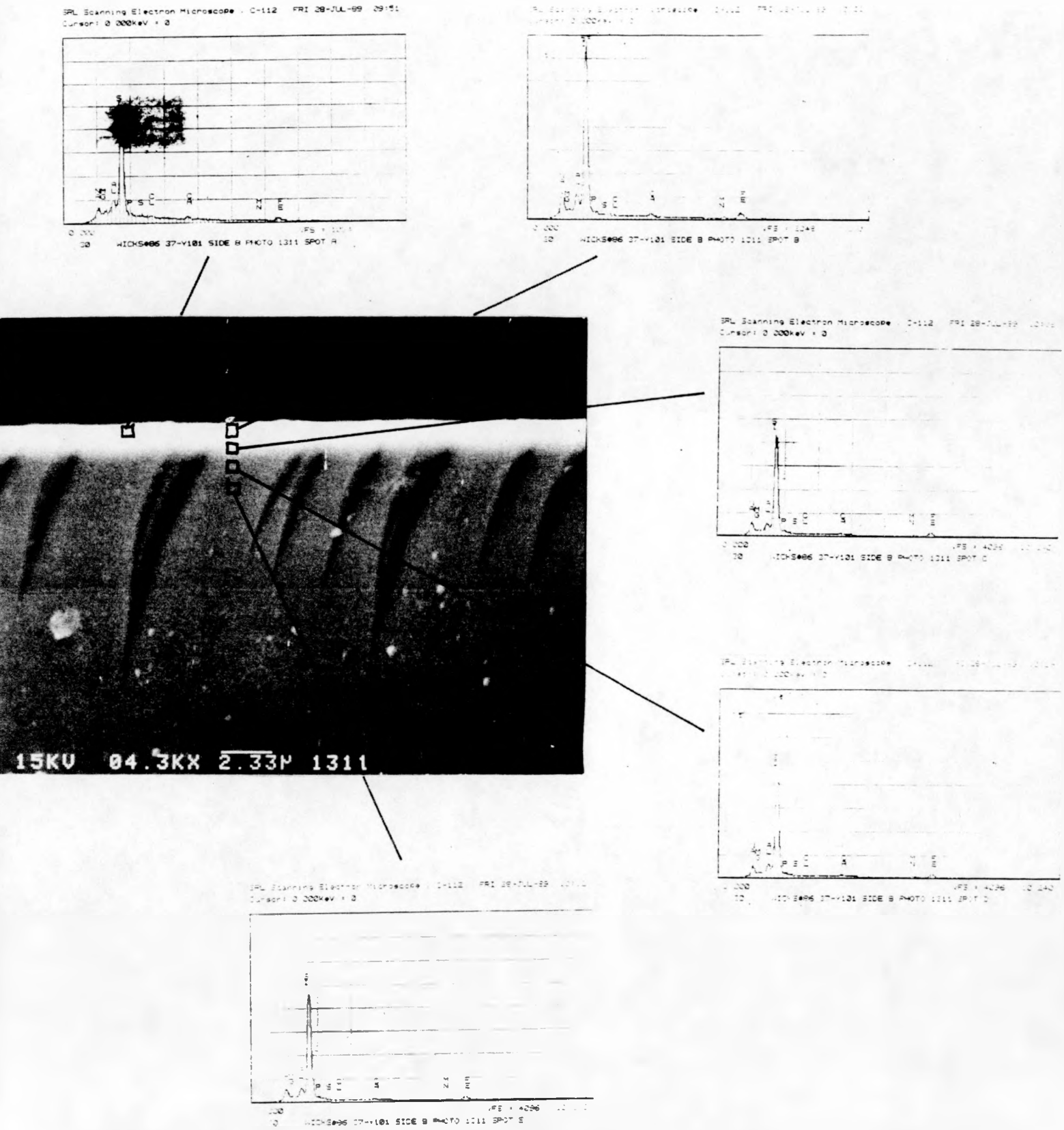


Figure 7V

SEM/EDX Cross Section
Sample Y-103 (Side B, I.D.)