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CHARACTERIZATION OF EPICOR II PREFILTER LINER 3

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ABSTRACT

As part of the overall TMI-2 Information and Examination Program, EPICOR II Prefilter Liner 3 was examined to provide information to aid in the development of technology for safely processing highly loaded ion-exchange media. The characterization program included sampling and analyses of the liner contents, including ion-exchange media, liquids and gases, as well as examinations of the liner interior and exterior. This report details the handling of the liner, sampling and analysis of the contents, and the examinations of the liner.

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INTRODUCTION

The Department of Energy is conducting a TMI-2 Information and Examination Program to gain information that will be of generic benefit to the safety of all light-water reactors and to aid in accident cleanup and decontamination of other civilian nuclear plants. As part of this program, a second EPICOR II Prefilter Liner (PF-3) was examined at Battelle Columbus Laboratories (BCL). This liner and the one examined earlier (PF-16) had been two of the 50 prefilter liners used for processing contaminated water from the Auxilliary and Fuel Handling Building of TMI-2. Results of PF-16 examinations are documented in Reference-1.

Primary objectives of the PF-3 characterization program were:

- To gain information on the characteristics of the internal conditions of the liner
- To determine the extent of degradation of the ion-exchange media due to radiation
- To develop data to aid in the selection of methods for safe disposal of the liners.

The EPICOR II prefilter liner is a 5-ft-high right circular cylinder 4 ft in diameter, containing 30 ft^3 of ion-exchange media. The walls and top are 1/4 in. thick and the bottom is 1/2 to 5/8 in. thick. The liner is fabricated of A36 carbon steel and is of welded construction. The interior surfaces of

the liner are coated with Phenoline 368 to retard corrosion. EPICOR II PF-3 liner is believed to contain various types of organic ion-exchange media--cation, anion, and mixed bed. However, the actual composition of the media as well as additional details concerning the internal structure of the liners is considered proprietary and not available for this document. A cross section of a typical EPICOR liner is shown in Figure 1. Schematics of the liner lifting lugs and penetration plugs are shown in Figure 2. Note, however, that the penetration plugs configuration varies from liner to liner.

The characterization program conducted on the liner PF-3 included the following tasks:

- Receipt of the liner at Battelle
- Liner exterior visual examination
- Liner internal gas sampling and analysis
- Liner interior visual examination
- Ion-exchange media sampling by coring
- Examination of ion-exchange media
  - Core gamma scan
  - Core visual examinations
  - pH determination
  - Water content
  - Radiochemical analyses
  - Microscopic examinations
- Sampling and analysis of residual liquid
- Preparation of liner for shipment
- Conclusions obtained from the results.

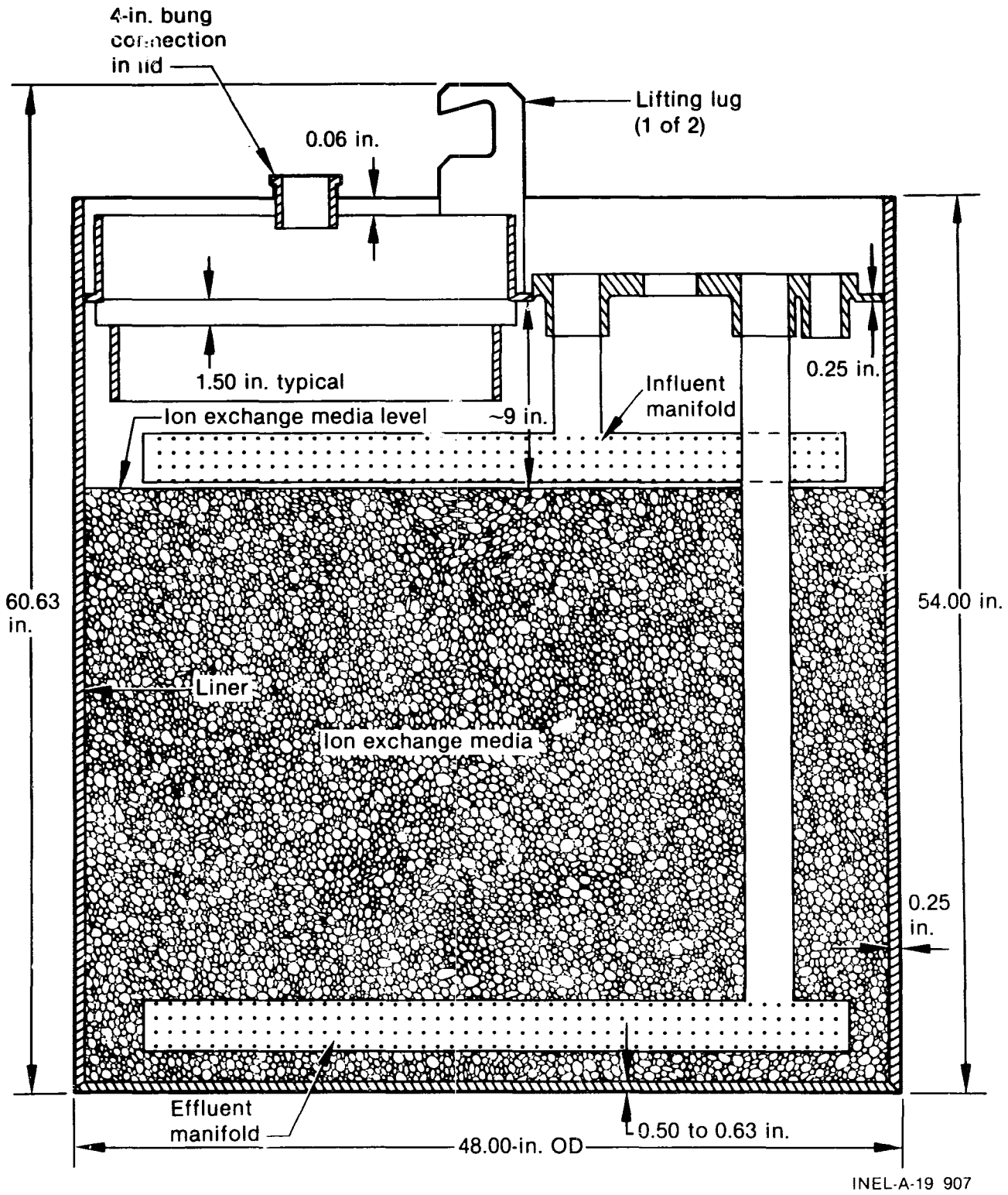


Figure 1. Cross-sectional view of a typical EPICOR II liner.

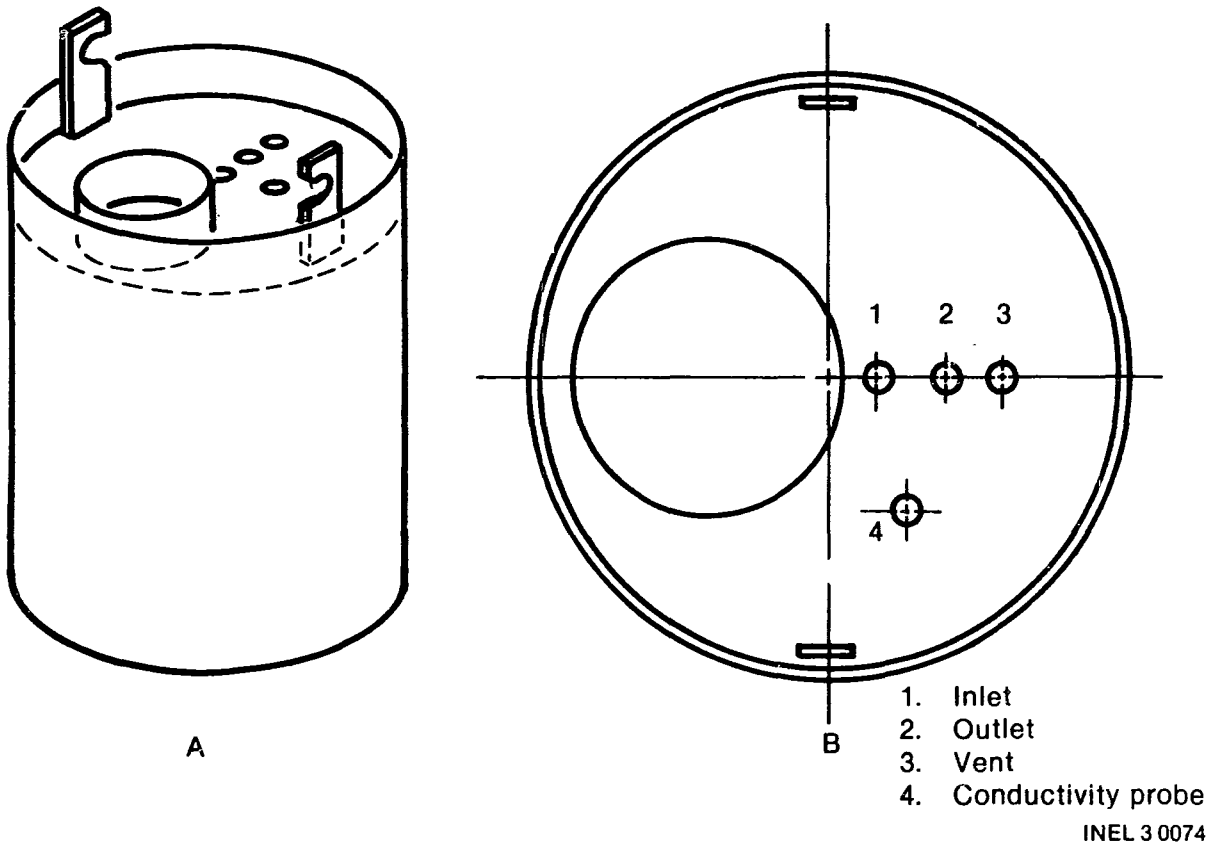


Figure 2. Schematics showing (a) orientation of PF-3 lifting lugs and (b) configuration of PF-3 penetration plugs.

SAFETY ANALYSIS

The BCL operational practice calls for a review and approval of certain operations by the Radiological Safety Committee (RSC), an independent group of BCL staff members. Operations or experiments that are judged to be off-normal or pose unique operational problems are reviewed by this committee. In general a detailed review of the operations, hazards associated with each of the major operations and procedures are conducted. In accordance with this practice, planned examinations were reviewed in detail. On the basis of the information presented and on the basis of the experience gained from the examinations of liner PF-16, RSC approval was granted to conduct all operations involving receipt, handling, examination and shipment of the liner.

LINER RECEIPT AT LABORATORY AND TRANSFER INTO HOT CELL

Liner PF-3 was received August 18, 1982, at Battelle's Nuclear Center, West Jefferson, Ohio. Preliminary radiological surveys were conducted by BCL Health Physics personnel and on the basis of these the shipment was accepted. The trailer and cask were transferred into the high bay area of the hot laboratory.

The gas sampling chamber originally designed for the characterization of EPICOR liner PF-16, (see Figure 3) was attached over one of the cask vent plugs, the chamber was evacuated, and the plug was loosened. The pressure gauge on the sampling chamber showed no detectable pressure with respect to atmospheric pressure (detection limit $\pm$ 0.25 psi). Two independent gas samples were taken for analysis, with the vent plug being replaced and the

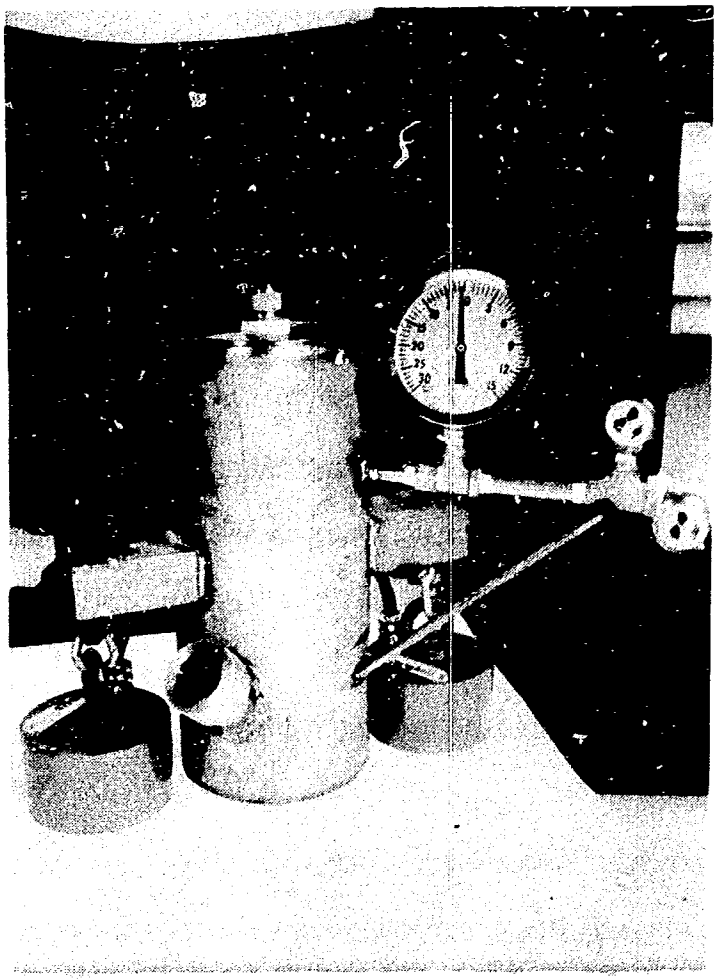


Figure 3. Gas sampling device.

sampling chamber evacuated between the samples. One sample was analyzed for gross gamma activity using portable survey instrumentation. No activity was detected. The first sample was analyzed by mass spectrometry and gas chromatography. The second sample was meant to be a confirmatory sample and was analyzed by mass spectrometry only. The results, shown in Table 1, are indicative of air. After the samples had been taken and the sampling chamber had been removed, the cask vent plug was loosened and a bubble solution (Snoop™) applied around it. A slight amourt of pressure in the cask was detected. The cask was then vented to the heavy element cell for approximately one hour before the cask lid was loosened.

A preliminary radiation survey was conducted before transfer of the liner from the cask. The radiation readings were 15 R/hr at the cask vent plug and 425 R/hr at the top surface of the liner. During the succeeding operations to remove the cask lid and transfer the liner to the hot cell, radiation from the cask was monitored continously by BCL Health Physics personnel. With the cask lid lifted, background around the cask was 60 mR/hr. A radiation reading of 100 R/hr was obtained at the horizontal plane of the cask lid with the cask lid completely removed and the general background dropped to 30 mR/hr. The reading at contact of the liner lid was 410 R/hr.

The transfer/storage device designed for use with liner PF-16 was used for the transfer of the cask into the hot cell. The transfer device, shown in Figure 4, was lifted into position on the open cask. A radiation reading of 60 mR/hr was obtained at the mating surface of the transfer device and the cask. The device top shield was removed and the reading at the opening (approximately 7 feet above the liner) was 25 R/hr. The liner was then raised into the transfer/storage device. After all support pins were in place, the

Table 1. Shipping Cask Gas Analysis

	Volume Percent		
	Sample 1	Sample 2	Uncertainty
Nitrogen	80.9	80.6	± 0.4
Oxygen	18.3	18.5	± 0.2
Carbon dioxide	0.05	0.05	± 0.01
Argon	0.84	0.84	± 0.02
Hydrogen	<0.1*	<0.1*	
Carbon monoxide	<0.002*	--	
Methane	<0.002*	--	

*Below detectable limits.

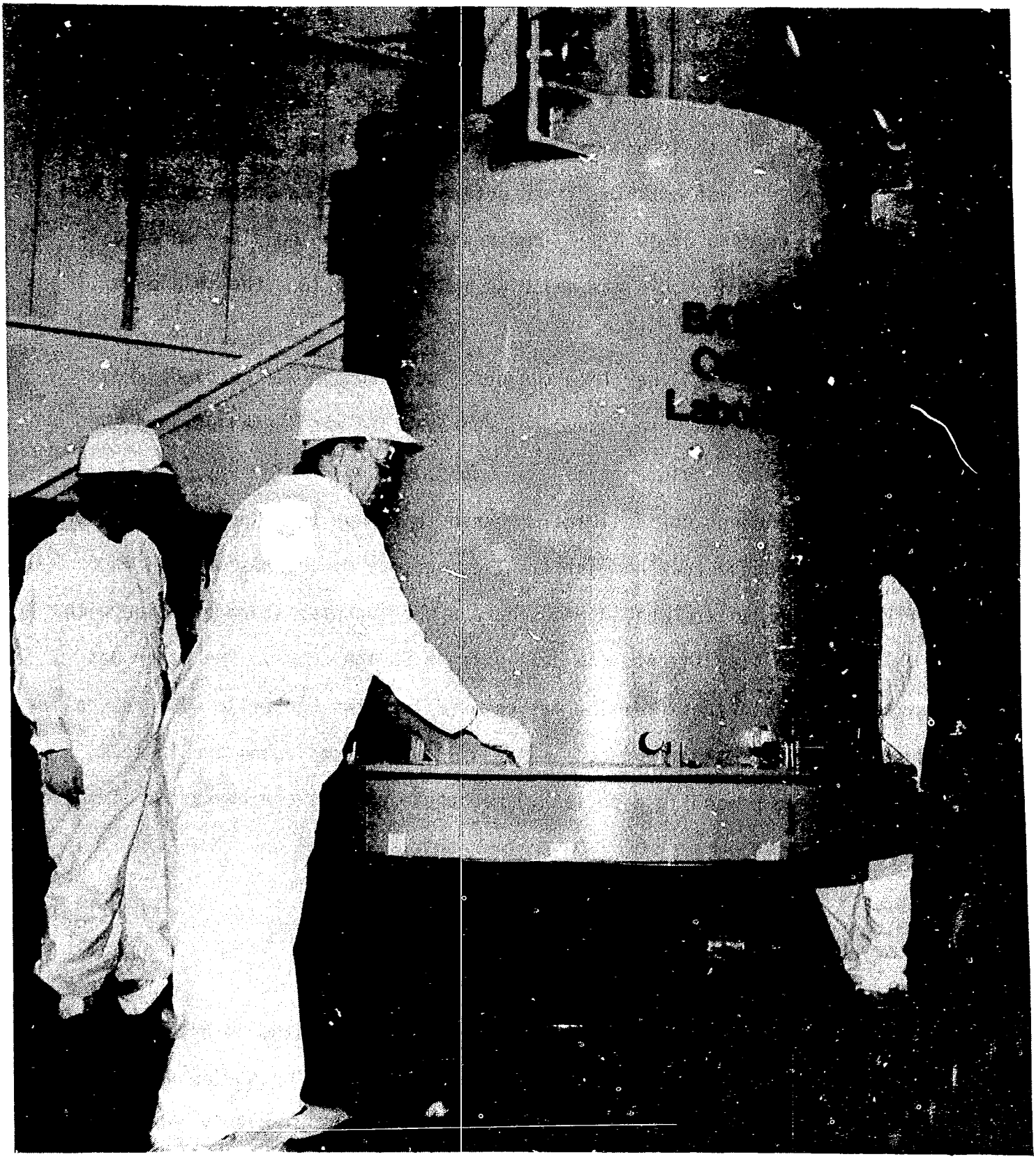


Figure 4. BCL designed liner transfer/storage device.

liner was lowered to rest on them and the top shield replaced. The contact radiation reading through the top shield was 250 mR/hr and through the sides, 60-70 mR/hr. As the transfer device was lifted away from the shipping cask, the readings at contact with the liner bottom generally ranged from 15-30 R/hr with a maximum reading of 40 R/hr. The transfer device and liner were positioned on the lower shield plate and pins installed to lock the shield in place.

After removing the liner from the cask, the dunnage was removed, radiation and smear surveys were conducted, the cask lid was installed, and the cask was prepared for shipment.

The heavy element hot cell was prepared to accept the liner by cleaning and removing the cell ceiling plates. The transfer device was wrapped in polyethylene, the bottom shield removed, and the transfer/storage device with the liner inside lowered into the cell using a 50-ton crane. The liner was unlatched and the transfer device was removed from the hot cell. The hot cell ceiling plates were then replaced. The liner was raised using the in-cell, 5-ton crane, and placed in a polyethylene bag to prevent contamination of the liner surface.

LINER EXTERIOR VISUAL EXAMINATION

A detailed visual examination was performed on the liner's exterior surfaces. The external visual examination was performed by both viewing the liner directly through the cell window and through the in-cell TV camera with an out-of-cell monitor.

The liner lifting bar was attached to the lifting lugs and to the 5-ton, in-cell crane hook. The liner was positioned in front of the cell window. The in-cell TV camera was positioned with the manipulator. The liner was then raised in front of the cell window and visually examined through the window and on the TV monitor. After the first pass, the liner was lowered, rotated 90 degrees, and examined again. A total of six passes were made. In general, the liner appeared clean and in good condition similar to PF-16. Paint on the surface was intact except on the bottom rim where a few rust spots (1/2 to 1 in. in diameter) were observed. Some surface scratches on the liner shell, with occasional rust spots were clearly visible but appeared to be superficial. The top of the liner was examined with the TV camera. The plugs and the manway cover were in good condition. Three penetrations plugs, influent, effluent and off-gas, could be clearly identified. In addition, the top also contained a conductivity probe and a pipe with a cap. The presence of the capped pipe was not anticipated as it is not shown in the design drawing. Purpose of this pipe is not clear.

The weld areas at the penetrations contained some black deposits as well as rust spots. The source of the black deposits is not clear. It is possible that these were deposits of dirt and grime collecting around the weld regions. Appearance of this deposit around the weld regions of the conductivity probe and other penetrations is shown in Figure 5.

The top surface of the manway cover was dirty with deposits that could be scraped off. This deposit presumably comes from spilled liquid which has dried off. The surface of the manway cover also showed rusting in areas with paint blistering and significant amounts of debris.

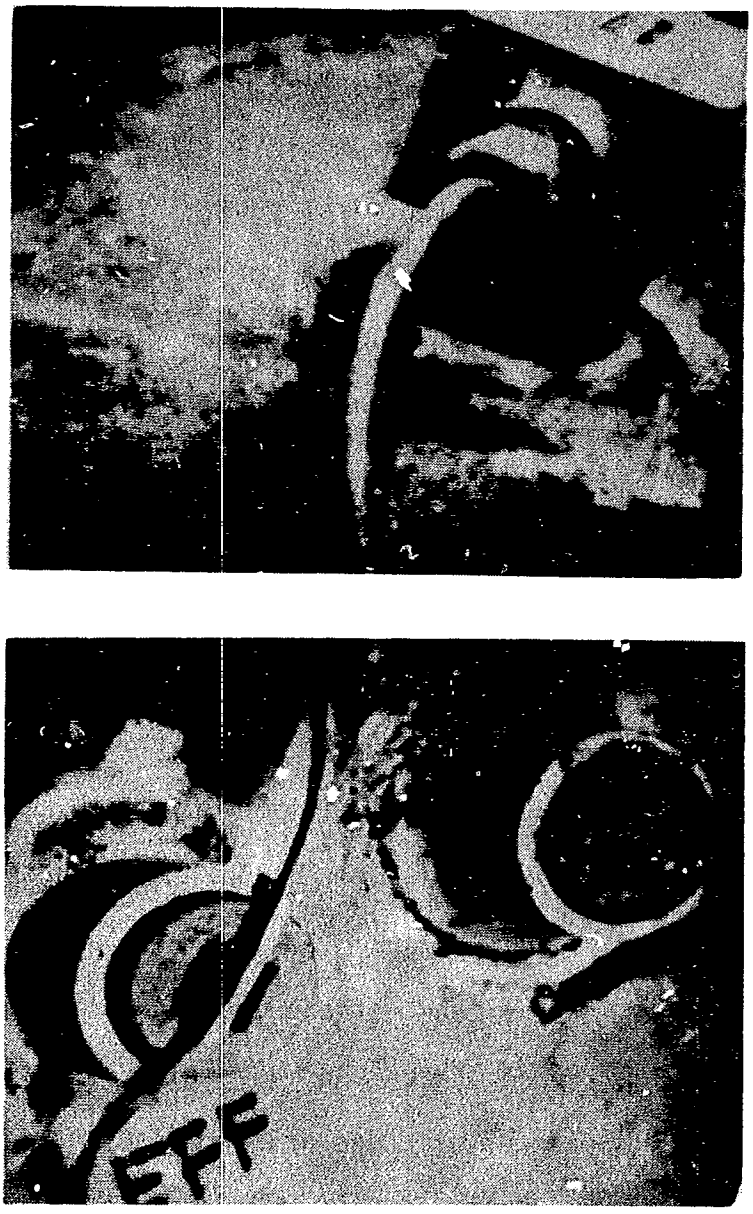


Figure 5. Appearance of black deposit around the weld regions of the penetrations. (a) conducting probe (b) effluent and off-gas penetrations.

The liner bottom surface had some areas of rust where the paint had been scraped off.

All of the operations involving the liner exterior visual examination were video-taped with verbal comments by the staff performing the examinations.

GAS SAMPLING AND ANALYSIS

One of the early tasks following the transfer of the liner into the hot cell was to obtain a sample of gas from the free space above the ion-exchange media. This was accomplished on August 23, 1982. The gas sampling device was modified for use with liner PF-3. The chamber was fitted with a vacuum/pressure gauge calibrated in psig and readable to within 0.1 psi. The general approach to the gas sampling was the same as that used for the cask sampling. Again, two independent samples of gas were drawn from the liner and liner internal gas pressure was measured during each sampling operation. The results of these measurements are given in Table 2 and indicate that the liner was at atmospheric pressure. Both samples were subjected to both mass spectrometric and gas chromatographic analyses. The results of the gas analyses are given in Table 3. There is a small discrepancy between the analyses for the first and the second samples. This is most apparent for the oxygen composition. It is suspected that the somewhat higher oxygen content in the first sample may be due to a small quantity of air trapped within the sampling device prior to the sampling. However, it is clear that the predominant constituent of gas is nitrogen.

Table 2. EPICOR Liner PF-3 Internal Pressure

	psia
Measurement 1	14.75
Measurement 2	14.67

Table 3. EPICOR Liner PF-3 Gas Analyses

	Volume Percent		
	Sample 1	Sample 2	Uncertainty
Nitrogen	94.7	95.0	± 0.4
Oxygen	0.86	0.35	± 0.05
Carbon dioxide	3.47	3.56	± 0.04
Argon	0.17	0.15	± 0.02
Hydrogen	0.8	0.9	± 0.1
Carbon monoxide	0.05	0.05	± 0.01
	Parts per Million by Volume		
Methane	82	90	± 10
Ethane	101	105	± 10
Propane	2	2	± 0.5
Iso-butane	0.7	0.5	± 0.1
Unknown	0.4	0.4	± 0.1
Ethylene	< 0.1	< 0.1	
Propylene	< 0.1	< 0.1	
Butene	< 0.1	< 0.1	
Acetylene	< 0.1	< 0.1	

LINER INTERIOR VISUAL EXAMINATION

The interior of the liner was examined with an in-cell TV camera and monitor. As a prelude to performing this examination, the liner manway cover was removed and examined. The bottom side of the manway cover showed a significant amount of rusting. The extent of corrision was about the same as that observed in the case of PF-16. In addition to corrosion, the bottom surface of the cover was also found to be coated with dried up splattered material. The splattered material appeared to contain flaky particles of yellowish brown color presumably from the turbulance of water flow through the ion-exchange media. Figure 6 shows the appearance of the corroded surface.

Subsequent to the examination of the manway cover, the interior of the liner was examined in detail. The support framework and the influent spreader pipe appeared to be in good condition. Liner interior walls above the resin bed also appeared clean over most of the visible surface. In some areas, however, the surface contained small bumps. It is not clear if these bumps were due to blistering of the coating or due to poor surface finish during liner fabrication.

The liner interior wall also contained one large area (approximately 8" by 2" area) where the coating had been scraped off. This area, located just above the resin bed, was presumably intended for the water to make a good ground with the liner so the water level can be determined by the conductivity probe. Significant amount of rusting was evident in this area. Figure 7 shows the appearance of this region.

The resin bed surface had the appearance of dried up mud with cracks. There were areas of white deposits with black particles which looked like

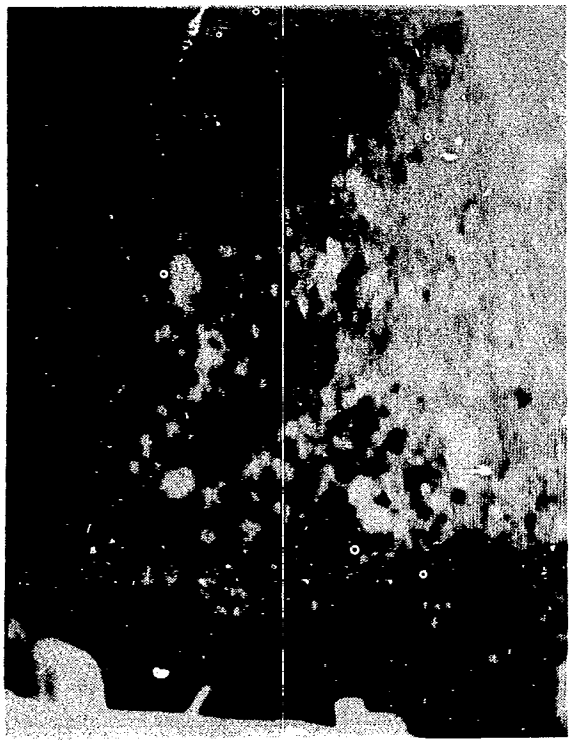


Figure 6. Appearance of the bottom surface of the manway cover.

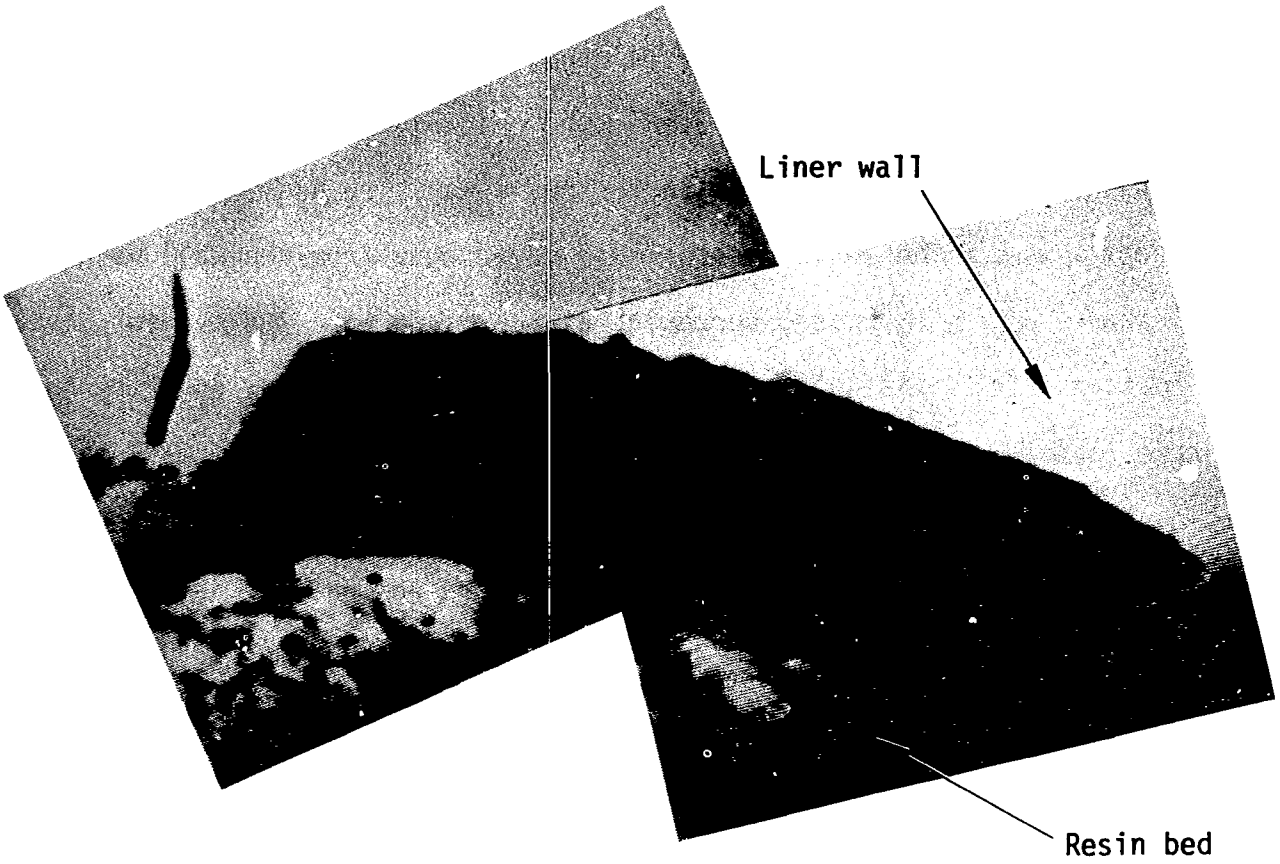


Figure 7. Appearance of the corroded area just above the resin bed surface. (Coating in this area appeared to be scraped off presummably to provide a contact surface for the conductivity probe).

pieces of charcoal. These particles were irregular shaped with sharp edges and were very few in number. They were not analyzed for composition. Typical appearance of the resin bed surface is shown in Figure 8.

The liner wall in contact with the resin was also examined in two different areas. The resin material was moved away from the wall using a small spade. In both cases, corrosion of the liner wall was evident beneath the coating. The coating was blistered and appeared loosely adherent on the surface (see Figure 9a). It could be removed easily with the small spade. In comparision, PF-16 showed little or no additional corrosion at the interface of liner wall and resin bed. Figure 9 shows the resin-liner interface before, during, and after removal of resin media in contact with the wall.

ION-EXCHANGE MEDIA SAMPLING AND EXAMINATION

Three core samples of ion-exchange media were taken using a core sampling device designed at BCL. Components for this core sampler are shown in Figure 10. The core sampling operation is illustrated in Figure 11. The core sampler was inserted into the media using a small air hammer (Figure 11a). The air hammer was then used to slide the shutter into place, closing the core sampler (Figures 11b and 11c). In a similar manner, a caisson and shutter were then inserted to encase the sample so that when the sample was withdrawn the media bed would not collapse. Figure 11d shows the caisson being inserted around the core sampler. Figure 11e shows the caisson and shutter in place and an eye-bolt attached to the top of the core sampler, ready to be lifted from the media bed.

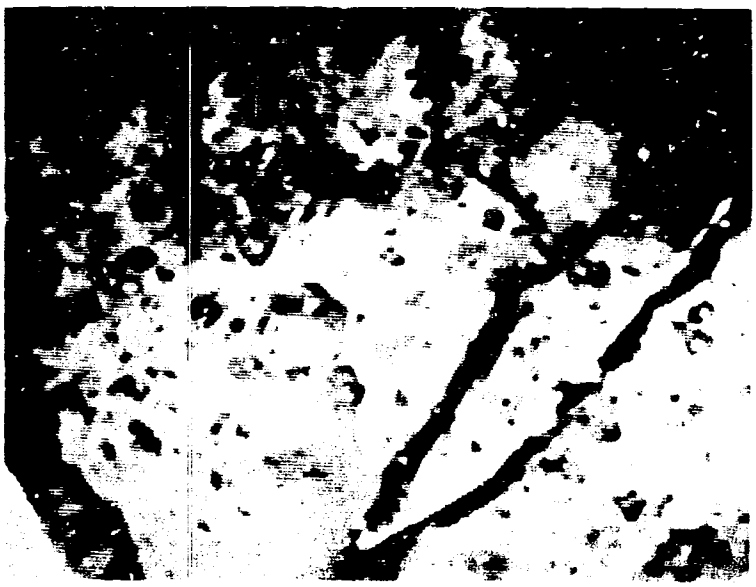


Figure 8. Resin bed surface showing white deposits, cracks and black charcoal-like particles.

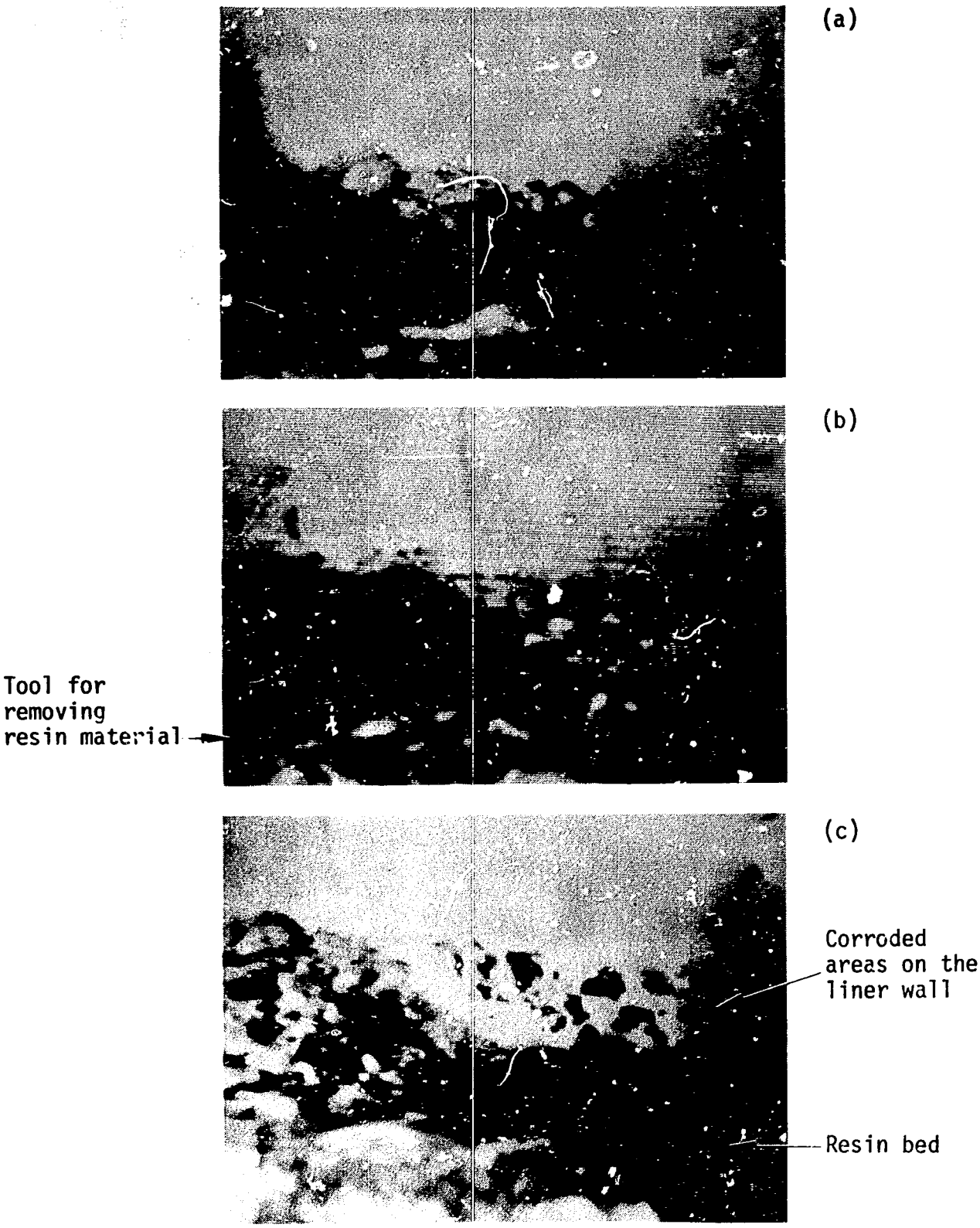


Figure 9. Resin bed liner wall interface (a) before (b) during and (c) after removal of the resin material in contact with the wall.

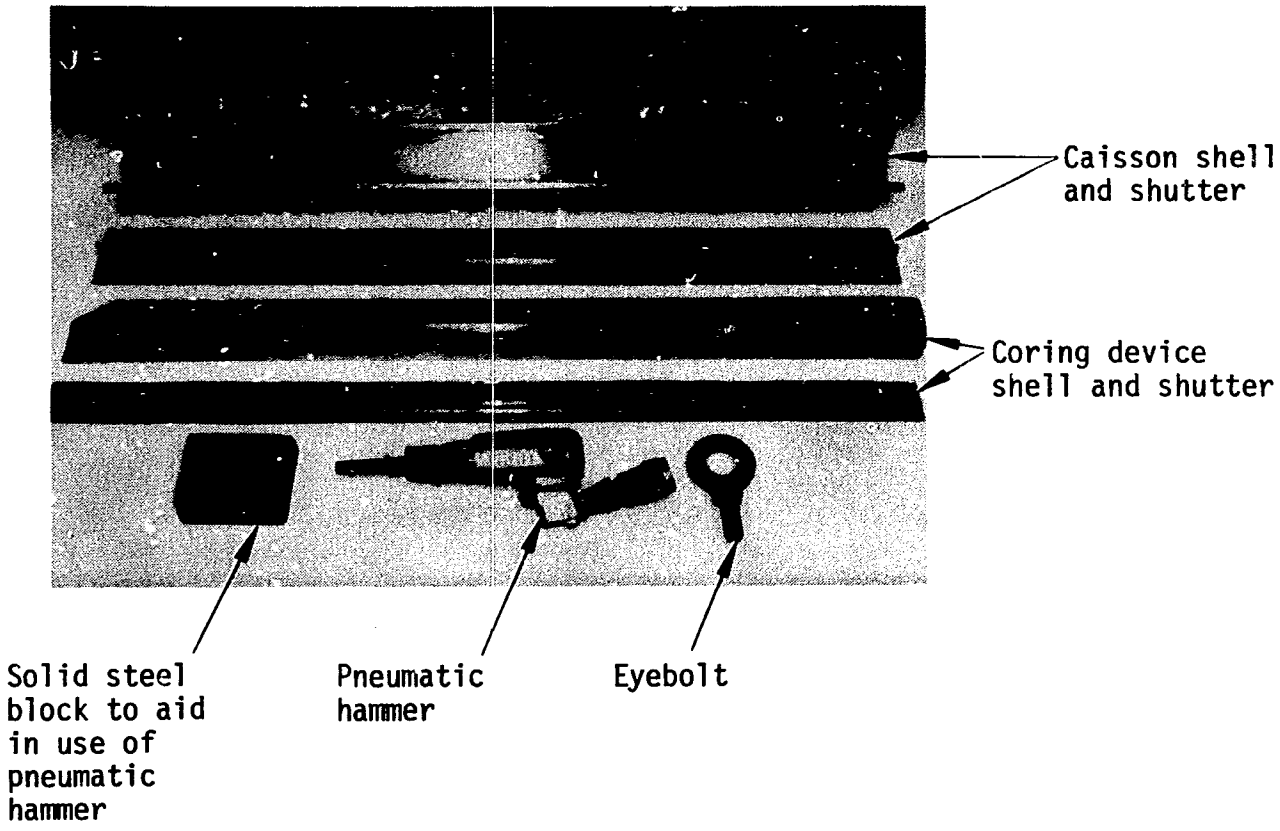
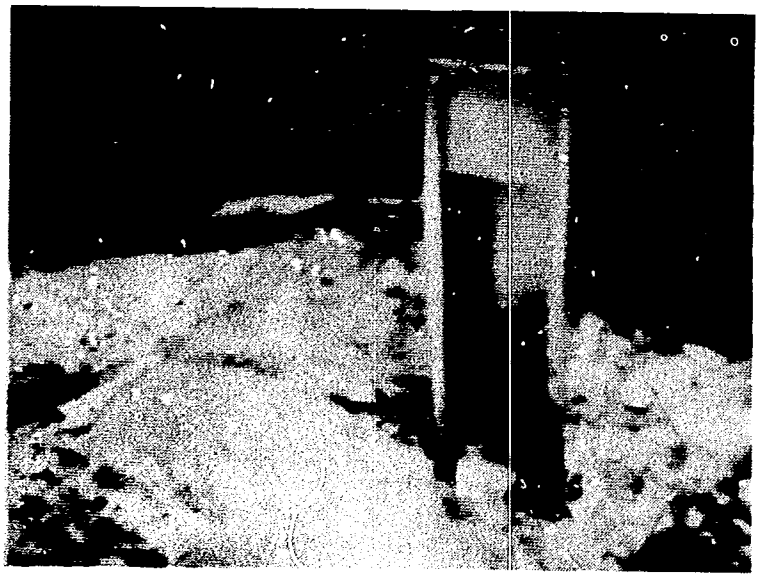
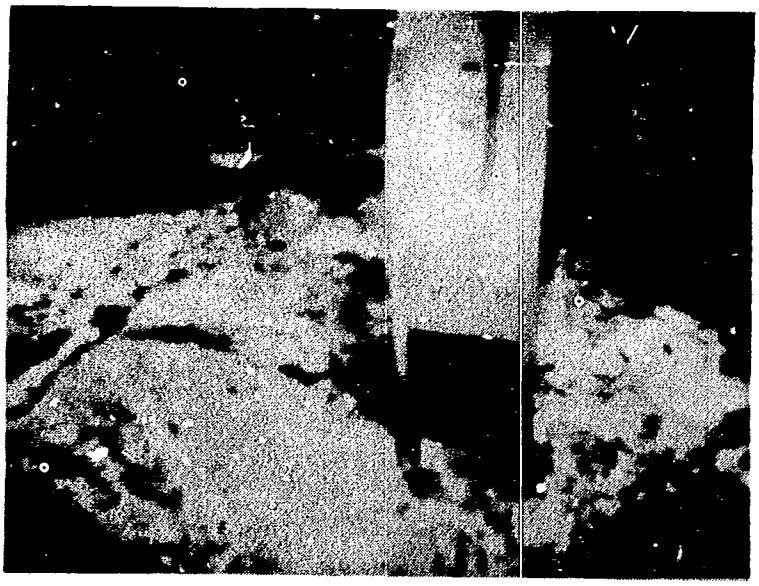


Figure 10. Core Sampling Device Components



(a)

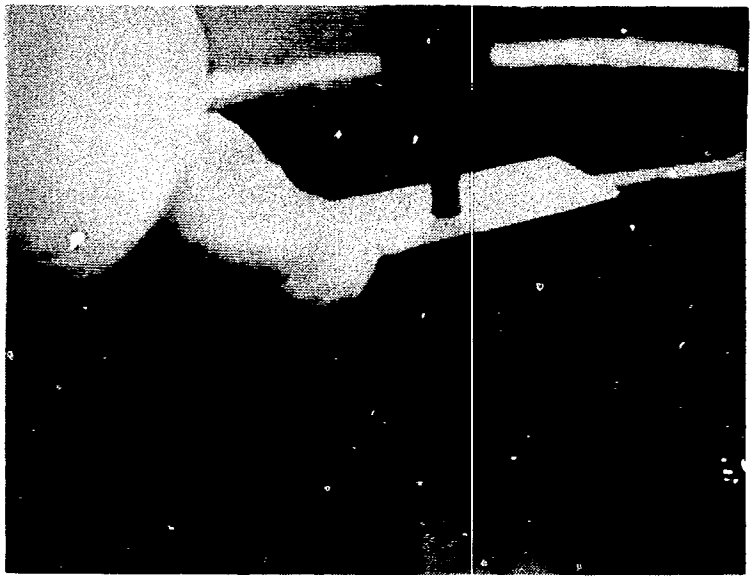
Coring device inserted in resin bed



(b)

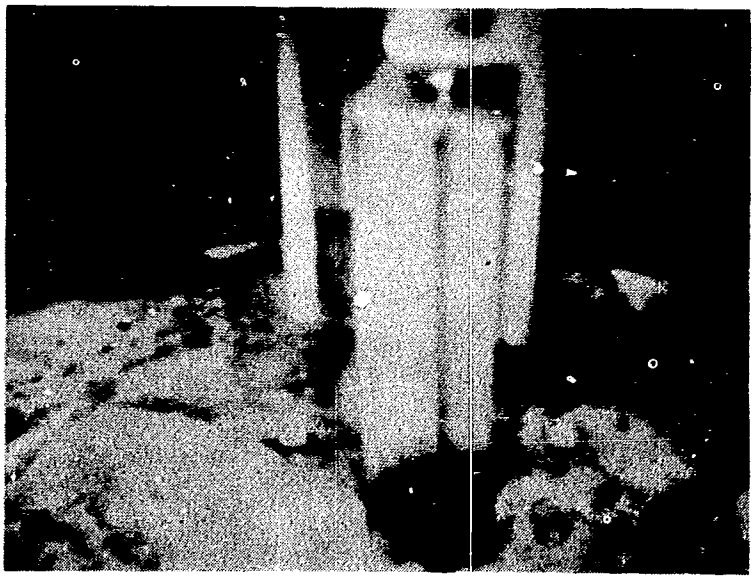
Inserting shutter in coring device

Figure 11. Core Sampling Operation.



(c)

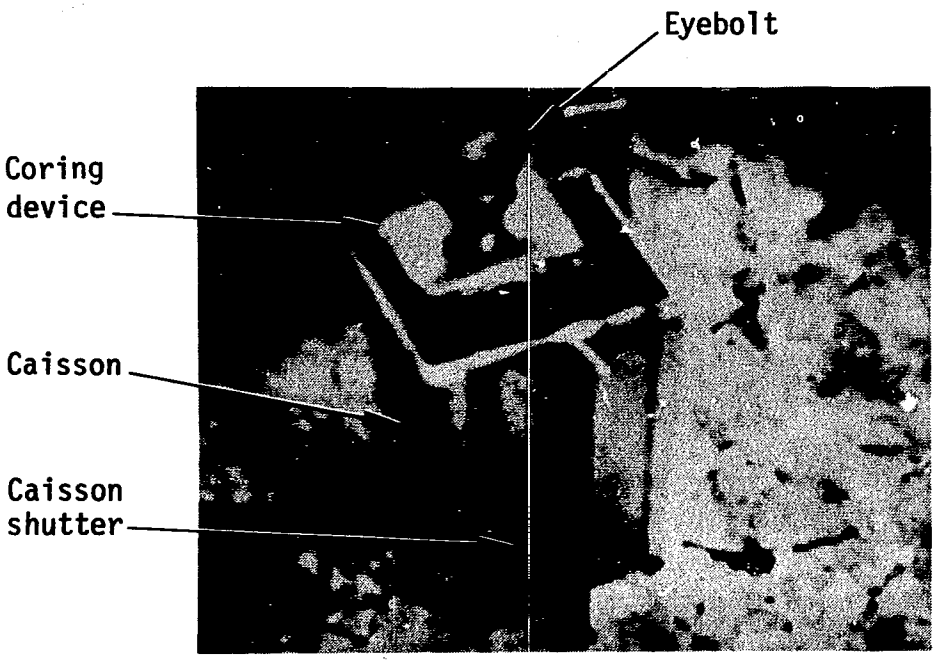
Closing shutter with pneumatic hammer



(d)

Inserting caisson around closed coring device

Figure 11. (Continued).



(e)

Coring device with caisson and eyebolt in place

Figure 11. (Continued)

During the first core sampling operation, the core sampler apparently struck the underdrain system after being inserted 26 inches into the approximately 30-inch resin bed. Removal of the shutter exposed a 24-inch core sample. After thoroughly probing the bed to determine the location of the underdrain system, a second core sample was taken. The sampler was 28 inches into the bed and an approximately 24 1/2-inch core sample was obtained. A third core sample, approximately 29 inches in length, was taken and shipped to Brookhaven National Laboratories. Figure 12 shows the relative locations of the three core samples obtained.

Core Gamma Scan

An external gamma scan of core sample #2 was performed to determine the relative depositions of gamma-emitting radionuclides. The gamma spectrometer with a horizontal Ge(Li) detector was calibrated using a mixed radioactive standard. The core sample was then positioned in front of the in-cell slit collimator containing the horizontal Ge(Li) detector. The core sample was moved vertically in front of the slit collimator using the in-cell crane, and an axial profile of ^{137}Cs activity and gross gamma activity was obtained. The gamma activity profile is shown in Figure 13. The maximum activity was observed at the 23-inch level of the core sample (total core length is 24 1/2 inches) and most of the activity was concentrated in the top 5 to 6 inches of the media bed. Gamma spectroscopy was performed at the location of the peak activity. The spectrum and a background spectrum acquired for the same time period are shown in Figures 14 and 15, respectively. The only isotopes detectable above background activities are ^{137}Cs and ^{134}Cs.

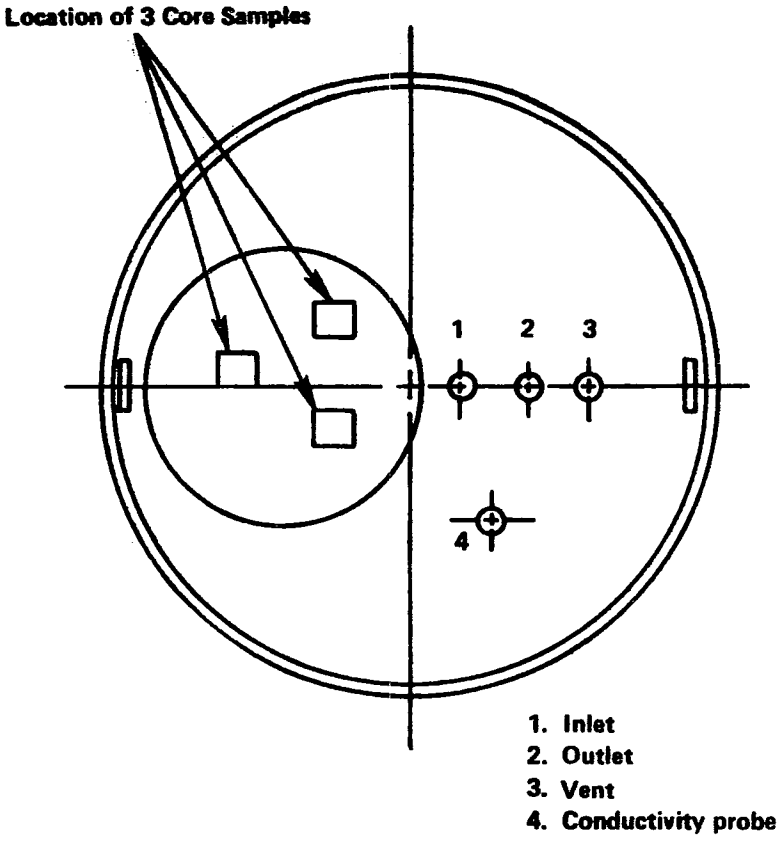


FIGURE 12. SCHEMATIC OF TOP OF LINER PF-3 SHOWING RELATIVE LOCATION OF 3 CORE SAMPLES TAKEN.

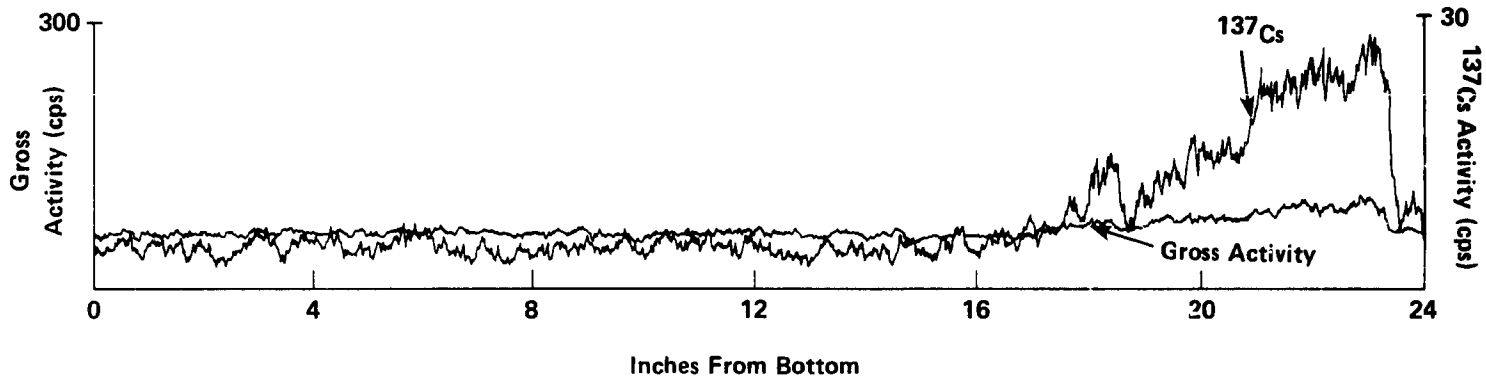


FIGURE 13. EPICOR LINER PF-3 CORE GAMMA SCAN (VERTICAL SCALE FOR GROSS ACTIVITY = 300 COUNTS PER SECOND (CPS), FOR ^{137}Cs = 30 CPS).

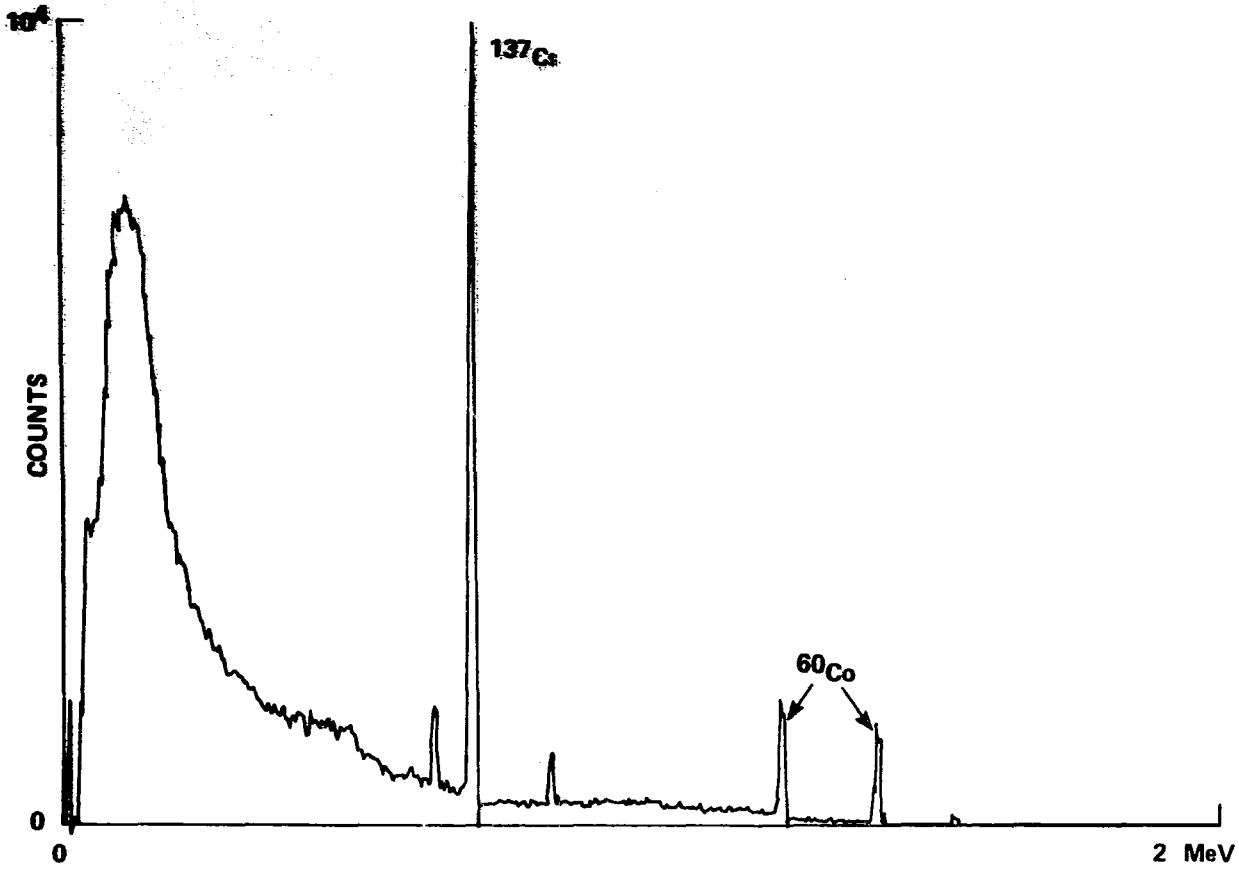


FIGURE 14. EPICOR LINER PF-3 CORE GAMMA ANALYSIS AT 24-1/4" LEVEL (4 MINUTE COUNT).

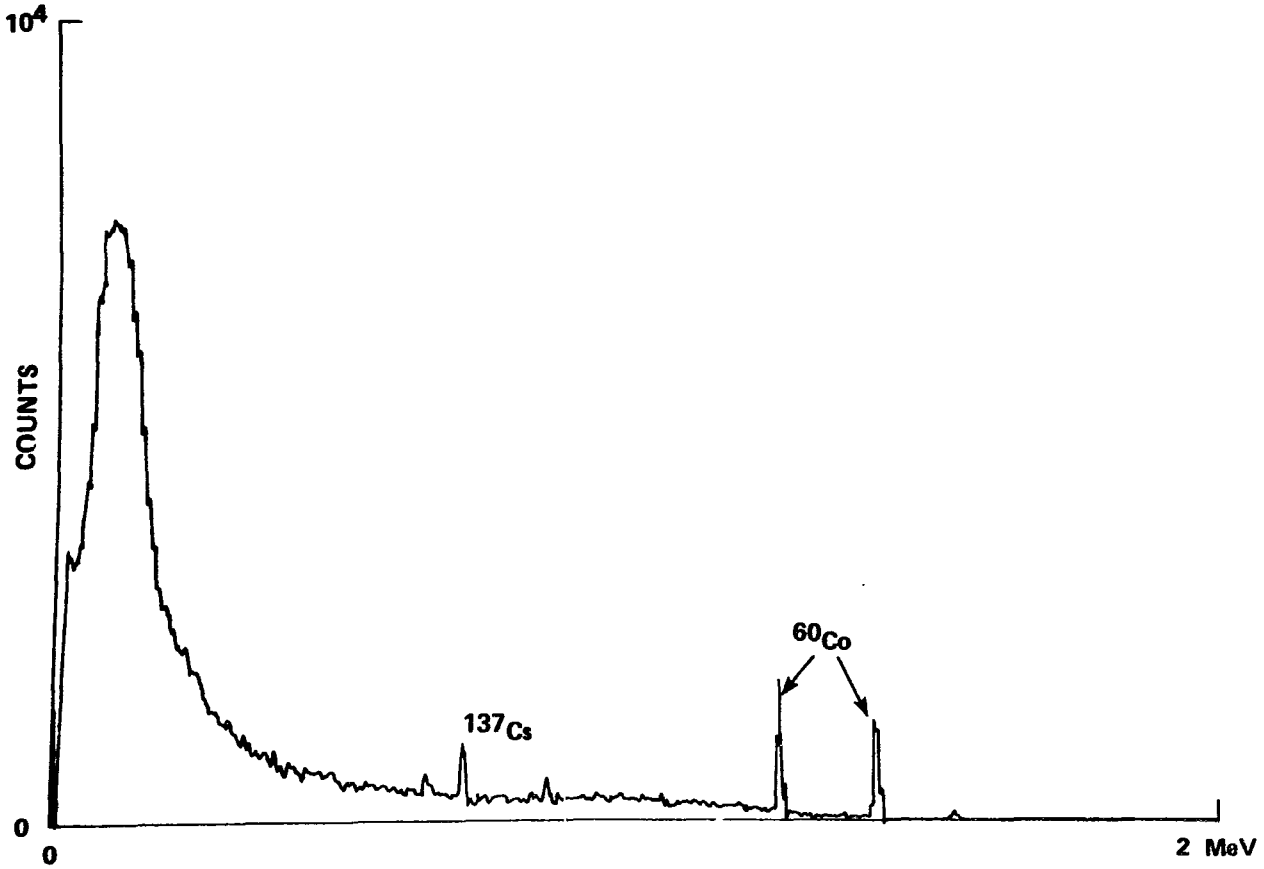


FIGURE 15. 4 MINUTE BACKGROUND COUNT.

Core Visual Examination and Sectioning

Using the hot cell stereomicroscope at ~30X magnification, a preliminary visual examination of core sample #2 was made with ion-exchange media exposed by removing the shutter from the core sampler. A schematic of the core sample is given in Figure 16. Two distinct regions were observed. The bottom region, approximately 16 inches in length, consisted of regular-shaped, light-colored, spherical particles that appeared translucent. These particles were yellow or orange in color. Figure 17 is a photomicrograph of this region. The top 8 inches of the core sample appeared darker than the bottom region. This region consisted of translucent, spherical particles in varying shades of dark and light and irregular shaped particles which were dark brown or black and looked like charcoal. Because the identity of the material used in liner PF-3 is considered proprietary and is not available for use in this project, it is not possible to determine the reason why some of the spherical particles are darker in color than others. A photomicrograph of this region is shown in Figure 18. Figure 19 is a micrograph of the light, powdery material at the top of the media bed.

The ion-exchange media was sampled by dividing the core into eight segments. The bottom region was removed from the sampler in 4-inch sections and the top region in 2-inch sections. The white powdery material on top of the core was removed as a separate section. Media samples were taken from the following sections for chemical and radio chemical characterizations: 0-4 inch, 8-12 inch, 18-20 inch, and 22-24 inch. All measurements given are in inches from the bottom of the core sample. Samples were taken from each of 8 ion-exchange media sections for microscopic examination.

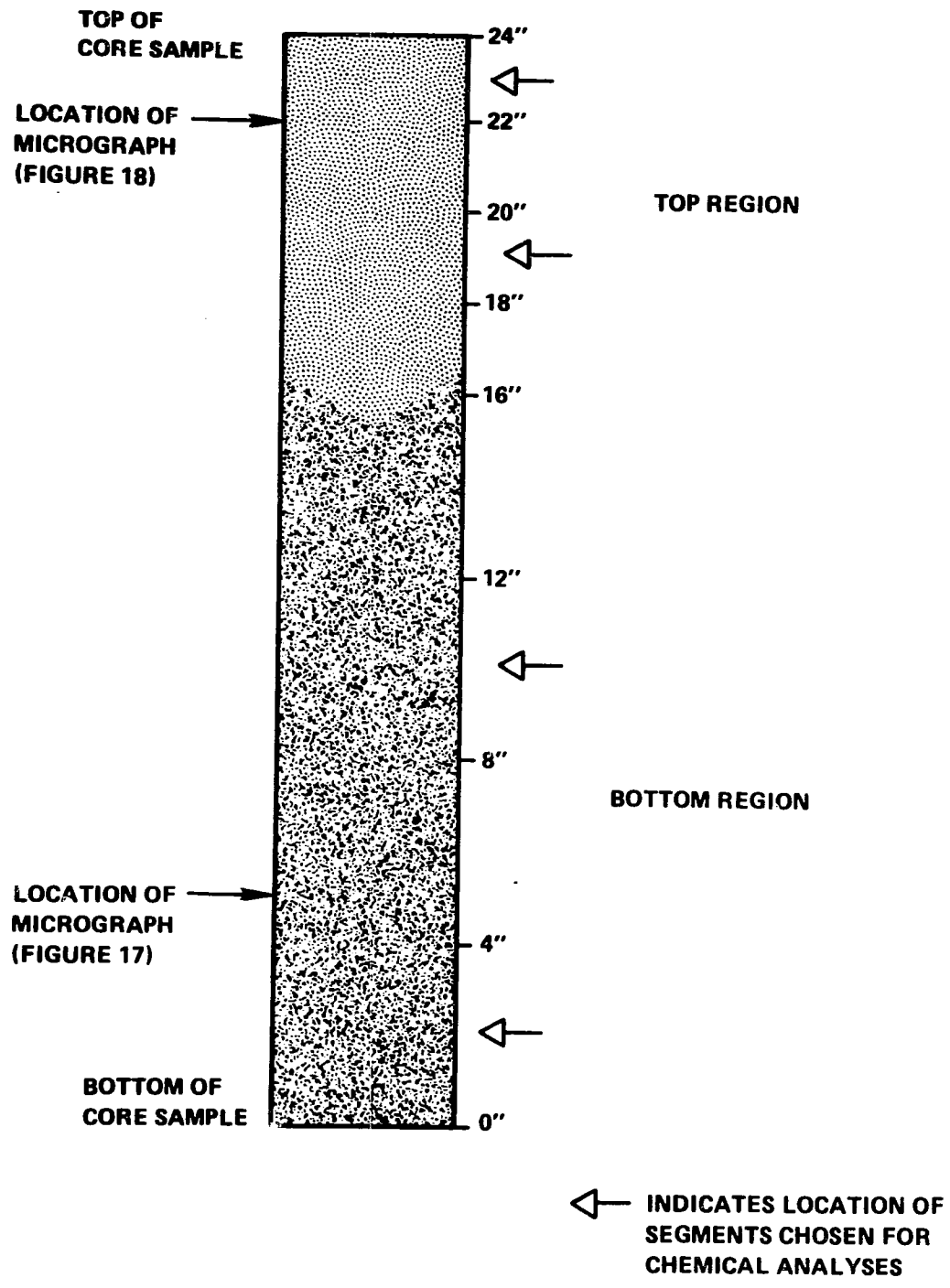


FIGURE 16. SCHEMATIC OF CORE SAMPLE NO. 2.

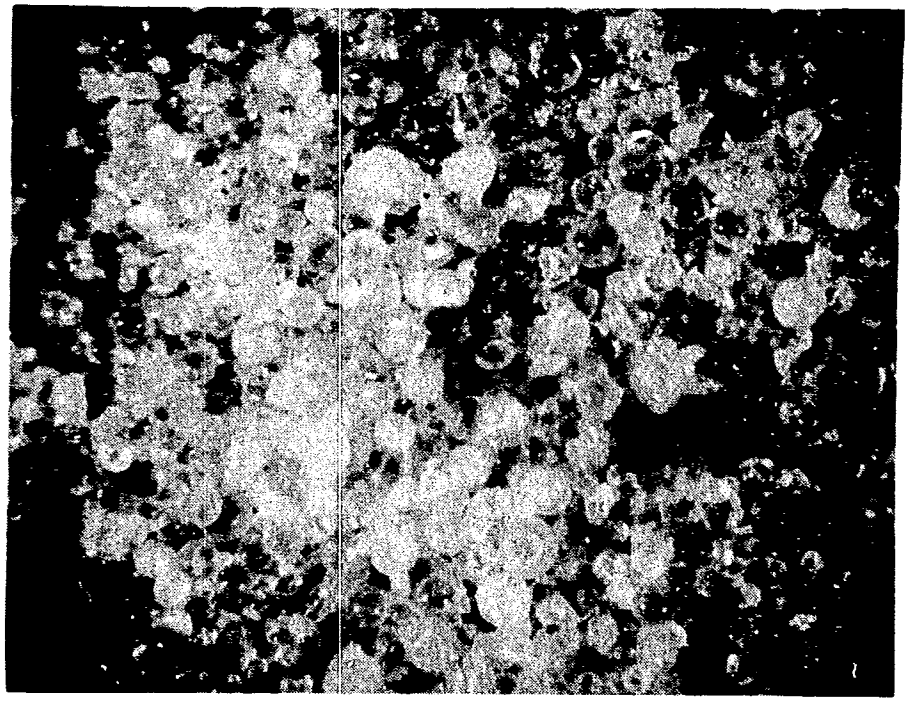


Figure 17. Micrograph of Core Sample, Bottom Region, 5 inches from bottom of core sample.

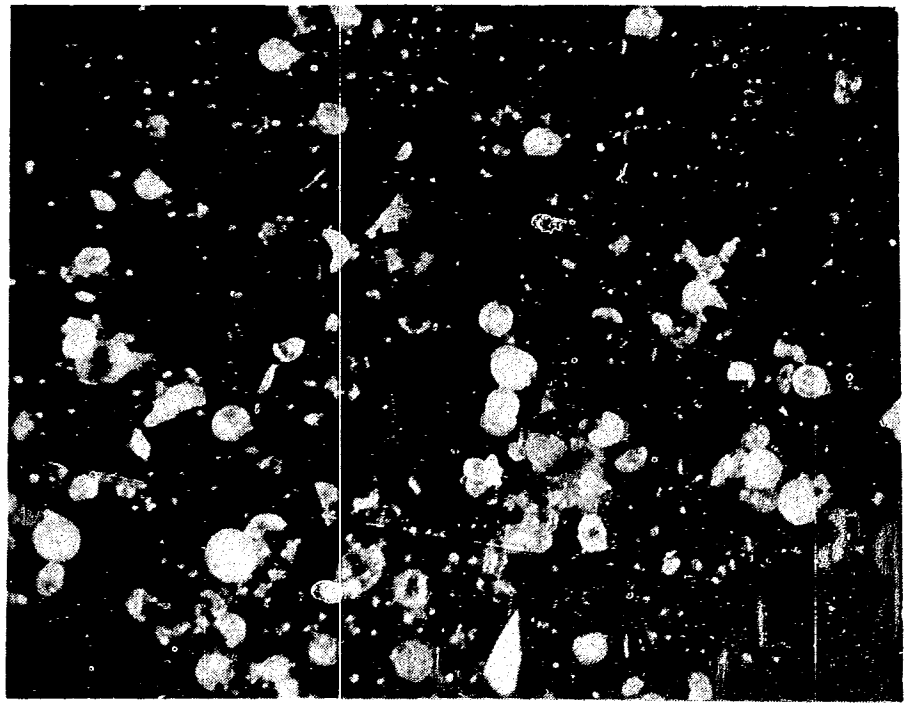


Figure 18. Micrograph of Core Sample, Top Region, 22 inches from bottom of core sample.

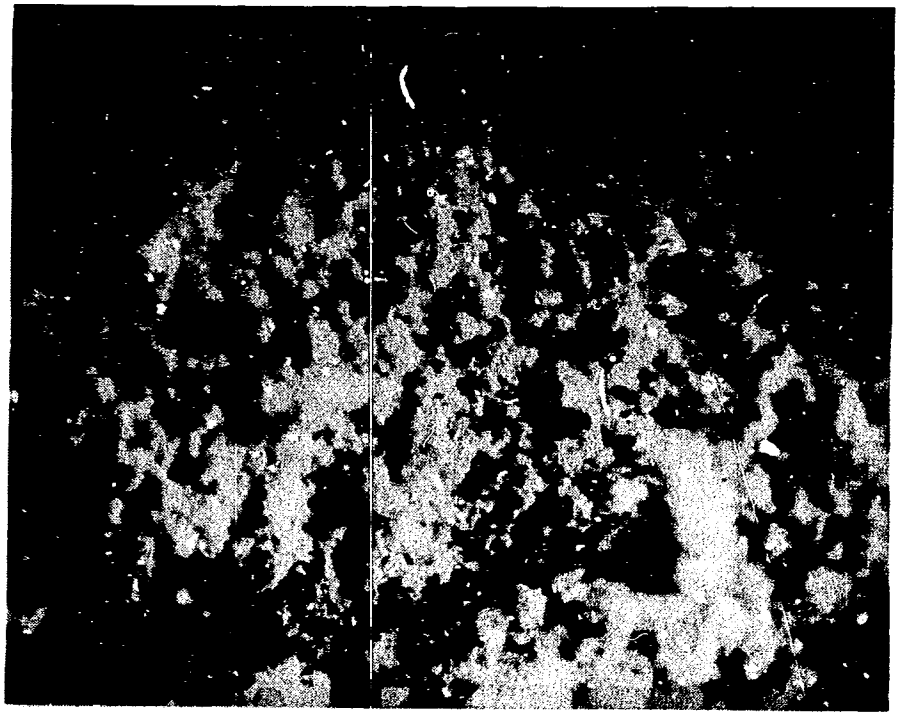


Figure 19. Micrograph of Light, Powdery Material at Top of Core Sample.

Determination of Media pH

Media samples were obtained from the following sections of the core sample: 0-4 inch, 8-12 inch, 18-20 inch, 22-24 inch. A 2-gram specimen of each sample was removed and mixed with 10 milliliters of deionized water (pH = 5.80). The pH of each solution was determined after the resin had settled using a digital pH meter with a combination probe calibrated against certified buffer solutions. The measurements were conducted in duplicate for each of the sections sampled. The observed pH values are shown in Table 4.

The total absorbed dose for the PF-3 resins is likely to be about 10^7 rads at the point of maximum dose. As the identity of the materials used in liner PF-3 is considered proprietary and not revealed for the purpose of this work, it is not possible to quantify any pH changes resulting from irradiation. The inital pH depends on both the type of resin (chemical form) and its ionic form, so it is not feasible to assume any given pH for comparisons.

Media Water Content

The water or moisture content of an organic ion-exchange medium varies directly with the porosity of the resin bead, which is itself inversely related to the degree of cross-linking. Gamma irradiation of an ion-exchange resin is known to result in both enhanced cross-linking and degradation of the ion-exchange materials, the predominant effect being a function of the total dose. The measurement of the water content and comparison with the water content of the unirradiated material provides an indication of the extent of

TABLE 4. EPICOR LINER PF-3 MEDIA pH VALUES

Section[a]	Sample	pH
0- 4"	1	5.16 ± .01
	2	5.24 ± .01
8-12"	1	3.08 ± .01
	2	2.92 ± .01
18-20"	1	3.01 ± .01
	2	2.94 ± .01
22-24"	1	2.80 ± .01
	2	2.75 ± .01

a Given in inches from the bottom of the core sample.

radiation damage and which of the two effects predominates. Experiments conducted at the Department of Energy's Rocky Flats plant[2,3] indicated an increase in the water content of both cation and anion resins that were irradiated in seven normal nitric acid (7N HNO_3).

Samples, approximately 10 grams each, were drawn from each of four sections of the media core sample. The water content measurements were conducted in duplicate. The specimens were allowed to air dry and then accurately weighed. They were then placed in a drying oven at 110°C for 16 hours. They were then allowed to cool and were reweighed. The percent water content is calculated from the difference between the grams of air-dried material and the grams of the oven-dried material divided by grams of the air-dried material. The calculated moisture contents of the media samples are shown in Table 5. In order to draw any conclusions concerning the radiation induced degradation or change in cross-linking, it would be necessary to compare these results with those for unirradiated materials. As the materials used in EPICOR II Liner PF-3 are considered proprietary, these values are not available.

Media Radiochemistry

The desired approach was to ash the organic ion-exchange media and then take the radionuclides in solution through a pyrosulfate fusion. As with the EPICOR liner PF-16, however, it was found that the media did not ash. The analytical procedure as modified for liner PF-16 was used in which an acid leach was substituted for the ashing and fusion. The leaching consisted of heating 10 grams of ion-exchange media in a nitric acid and hydrochloric acid mixture for a period of 8 hours.

Table 5. ION-EXCHANGE MEDIA WATER CONTENT

Section[a]	Water Content[b] (%)
0-4"	44
	46
8-12"	31
	30
18-20"	18
	17
22-24"	18
	18

a Given in inches from bottom of core sample.
b Percent water content = $\frac{\text{grams air dried - grams oven dried}}{\text{grams air dried}}$

A sample of the leachate was removed from this solution, diluted, and transferred out of the hot cell for gamma spectroscopic analysis. In order to remove the remainder of the leachate from the hot cell for analyses, it was necessary to first reduce the activity by removing ^{137}Cs and ^{134}Cs. This was accomplished by precipitating the cesium with silicotungstic acid. Following the precipitation, these samples were removed from the hot cell for the remainder of the analytical work.

The leachate was analyzed for the content of ^{239}Pu, ^{240}Pu, and ^{241}Pu, and for the content of ^{235}U and ^{238}U by ion-exchange separation followed by mass spectrometry. The strontium analysis was carried out using a nitration technique.

For each of the four core sections selected for radiochemical analysis, duplicate samples of approximately 10 grams each were leached in nitric acid/hydrochloric acid solutions. Following the cesium removal and transfer from the hot cell, aliquots were taken from each sample for analysis for strontium, uranium and plutonium content. The results are summarized in Table 6.

On the basis of these analyses it appears that the top region was very effective at removing the strontium and cesium from the contaminated water. Plutonium and uranium, however, were less effectively removed, having been distributed uniformly throughout the media bed.

TABLE 6. Radionuclide Content of EPICOR PF-3 Ion-Exchange Media (Ci/g)

Section	^{235}Ua	^{238}Ua	^{239}Pua	^{240}Pua	^{241}Pua	^{90}Sra,b	^{137}Csc	^{134}Csc	^{60}Coc
22-24"	4.8 E-14	4.6 E-13	2.8 E-10	4.0 E-10	5.8 E-8	2.0 E-4	5.2 E-3	3.1 E-4	Not detected
	5.6 E-14	7.8 E-13	4.9 E-10	3.8 E-10	<5.7 E-8	2.2 E-4	5.5 E-3	3.2 E-4	Not detected
18-20"	1.3 E-14	7.9 E-13	2.8 E-10	2.8 E-10	4.5 E-8	1.0 E-5	4.8 E-3	2.8 E-4	1.2 E-6
	2.0 E-14	1.4 E-13	3.5 E-10	4.3 E-10	6.5 E-8	1.0 E-5	4.8 E-3	2.9 E-4	1.4 E-6
8-12"	<1.5 E-14	1.7 E-13	2.3 E-10	2.4 E-10	<7.4 E-8	2.7 E-7	5.2 E-6	3.0 E-7	3.8 E-8
	<1.1 E-14	1.8 E-13	2.3 E-10	2.8 E-10	<5.3 E-8	2.9 E-7	4.9 E-6	3.3 E-7	2.3 E-7
0- 4"	4.2 E-14	6.5 E-13	4.0 E-10	5.6 E-10	9.1 E-8	3.2 E-7	4.0 E-6	4.6 E-7	6.3 E-8
	1.5 E-14	3.4 E-13	2.3 E-10	2.8 E-10	4.5 E-8	2.1 E-7	1.9 E-6	1.8 E-7	7.6 E-8

a Activity on 12/16/82.
b ^{89}Sr was not detected.
c Activity on 12/14/82.

Microscopic Examination of Ion-Exchange Media

Samples of ion-exchange media from Liner PF-3 were examined by scanning electron microscopy. The primary objective of the examination was to determine the integrity of the ion-exchange media and to characterize the extent of radiation induced degradation.

Particles of the ion-exchange media were extracted from each of the eight segments of core and selected for examination. (See Figure 16 for the location of the segments in the core sample.) The particles were then mounted on a graphite cylinder using a colloidal silver liquid. The mounts were then coated with a thin film of gold to provide an electrically conductive surface. (Coating with a thin film of carbon was tried initially but an adequate coating could not be obtained.)

The specimens were examined at various magnifications starting at 20X. Selected particles from each segment were subjected to a semiquantitative analysis using the Energy Dispersion Analysis X-ray (EDAX) attached to the scanning electron microscope.

Bottom Region

The particles in this region (the lower 16" of the media bed) were all spherical in nature. Many of the particles were fragmented and on all intact particles surface defects were observed. Figure 20 shows the typical distribution of intact particles and particle fragments in this region of the media core sample. Figures 21 through 24 show examples of surface defects and fragmented media particles. EDAX analysis was performed on several particles

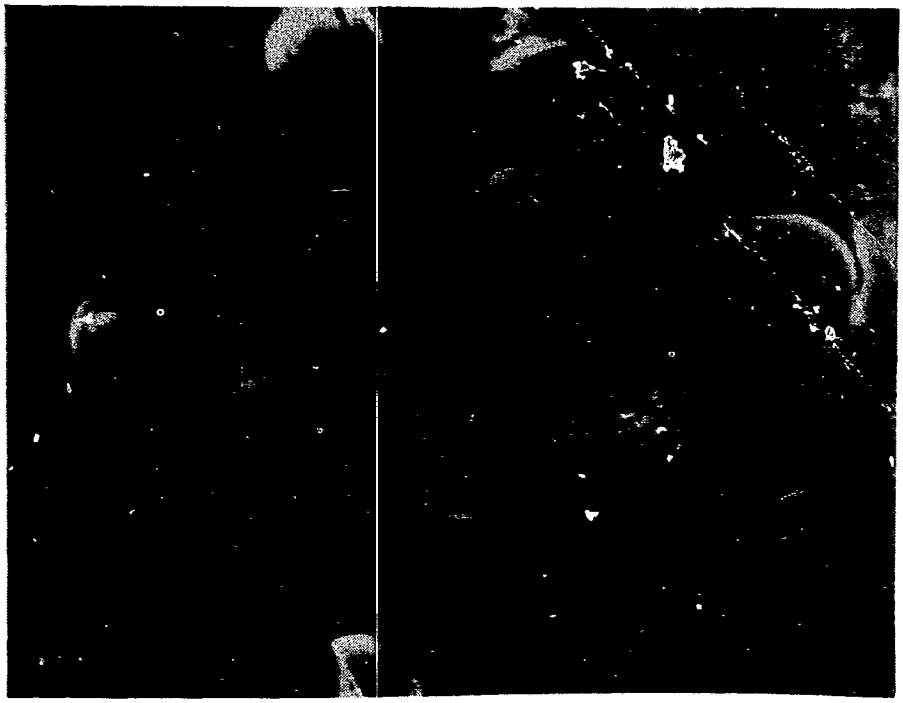


Figure 20. SEM photomicrograph of ion-exchange media, Bottom Region, 8-12" segment, 30X.

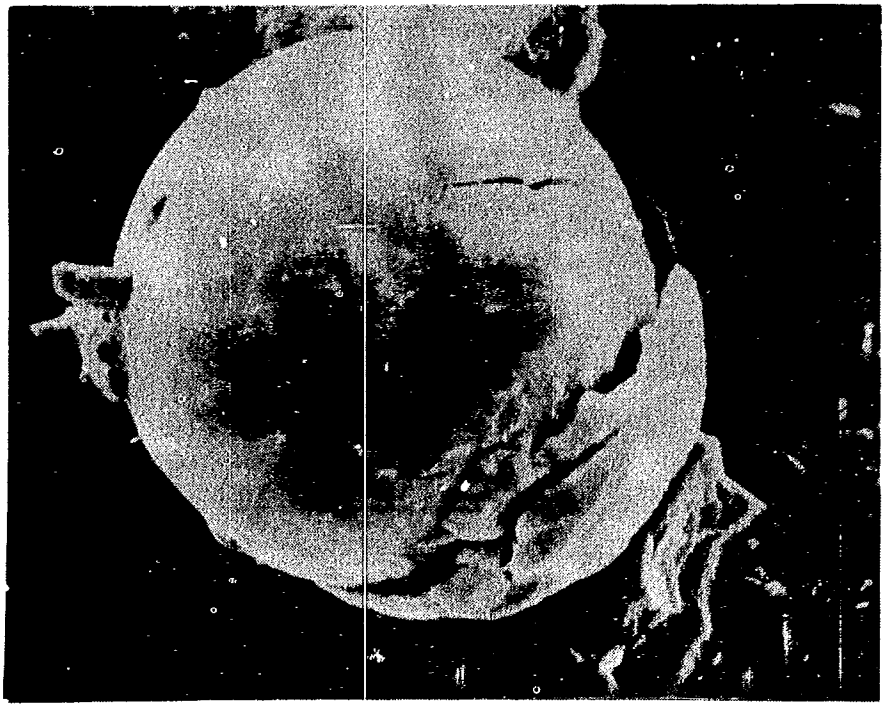


Figure 21. SEM photomicrograph detailing fractured media particle, Bottom Region, 0-4" segment, 150X.

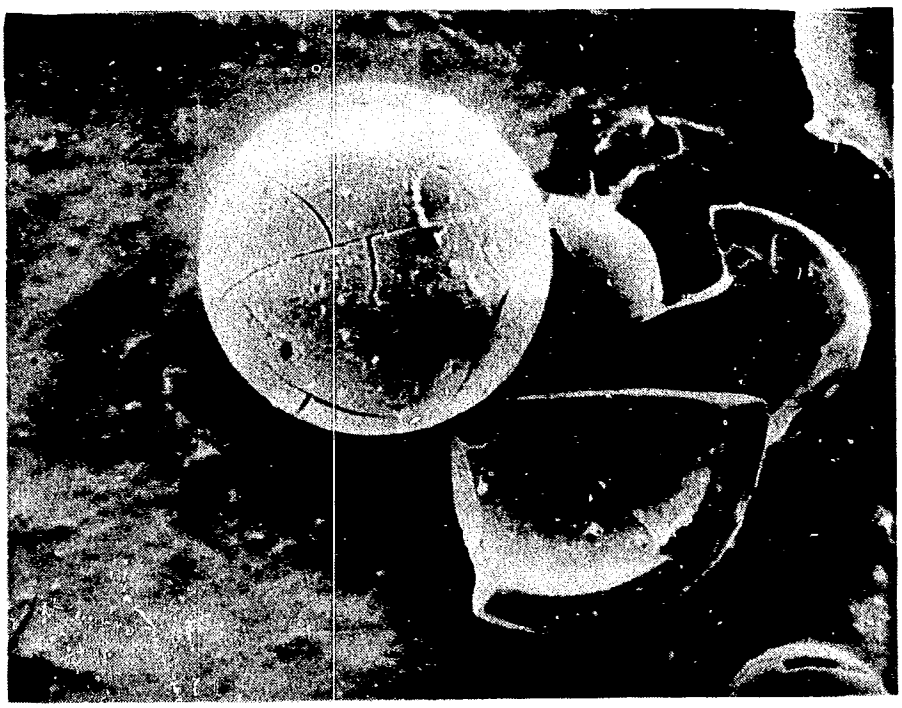


Figure 22. SEM photomicrograph detailing surface defects in media particle, Bottom Region, 4-8" seg,emt. 70X.

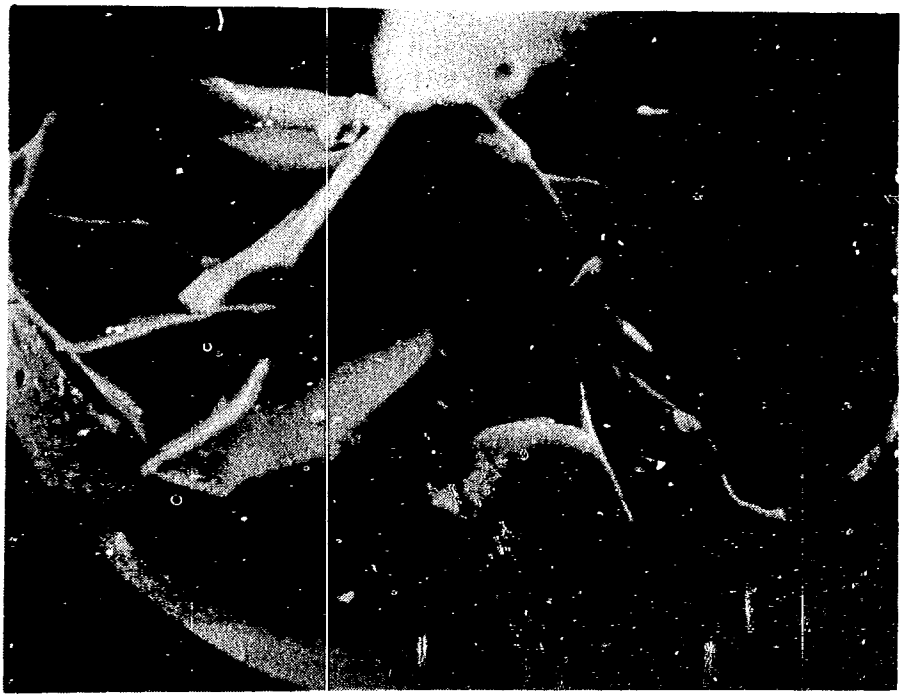


Figure 23. SEM photomicrograph detailing fragmented media particle, Bottom Region, 8-12" segment, 200X.

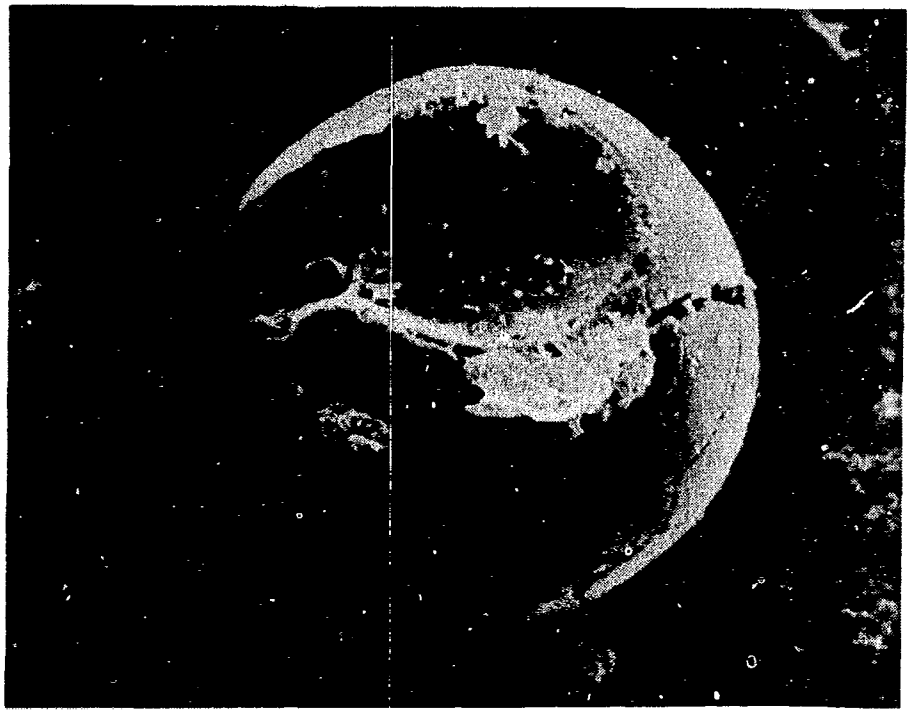


Figure 24. SEM photomicrograph detailing surface defects in media particle, Bottom Region, 12-16" segment, 100X. (White deposits on the media particle are silver mounting material.)

in this region. Two typical spectra are shown in Figures 25 and 26 and indicate sulfur (the predominant constituent), sodium, silicon, and calcium as the only detectable elements (elements of atomic numbers below sodium (11), including carbon and hydrogen, are not detectable with the EDAX system).

Top Region

In this region (the top approximately 8 inches of the media bed), two distinct layers were noticeable during the SEM examination. In the lower four inches of the region (16-20" from the bottom of the core sample), two types of particles were observed: spherical particles and granular, irregular particles. The spherical particles in this layer showed little or no evidence of surface defects and no fragmented particles were found. Examples of typical particles from this layer are shown in Figures 27-29.

An EDAX spectrum from a typical particle is shown in Figure 30. Sulfur is again the predominant constituent with smaller amounts of sodium, aluminum, silicon, tin, calcium and iron detected.

In the top four inches of this region (20-24" from the bottom of the core sample), only spherical particles, showing little or no evidence of surface defects were found. Two particle fragments were found and are shown in Figures 31 and 32. These fragments appear to be from cleanly split particles. Neither fragment showed any evidence of cracking or spalling on its outer face. Figure 33 shows a typical particle from this top layer of the core sample. An EDAX spectrum from a typical particle is shown in Figure 34. Sulfur is again the predominent constituent with smaller amounts of sodium, silicon, and calcium.

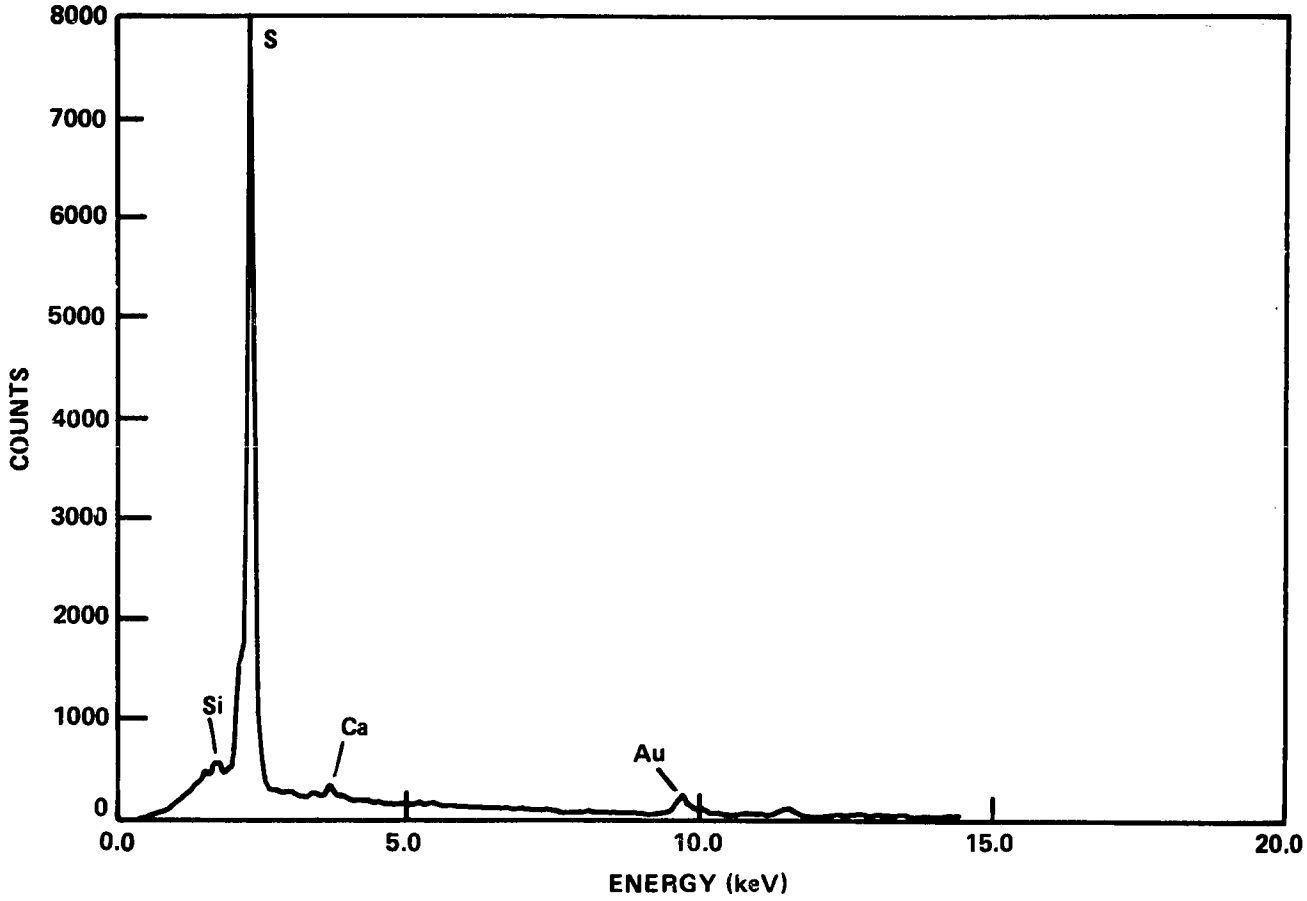


FIGURE 25. EDAX SCAN OF A MEDIA PARTICLE FROM BOTTOM REGION, 4-8" SEGMENT (Au IS COATING MATERIAL).

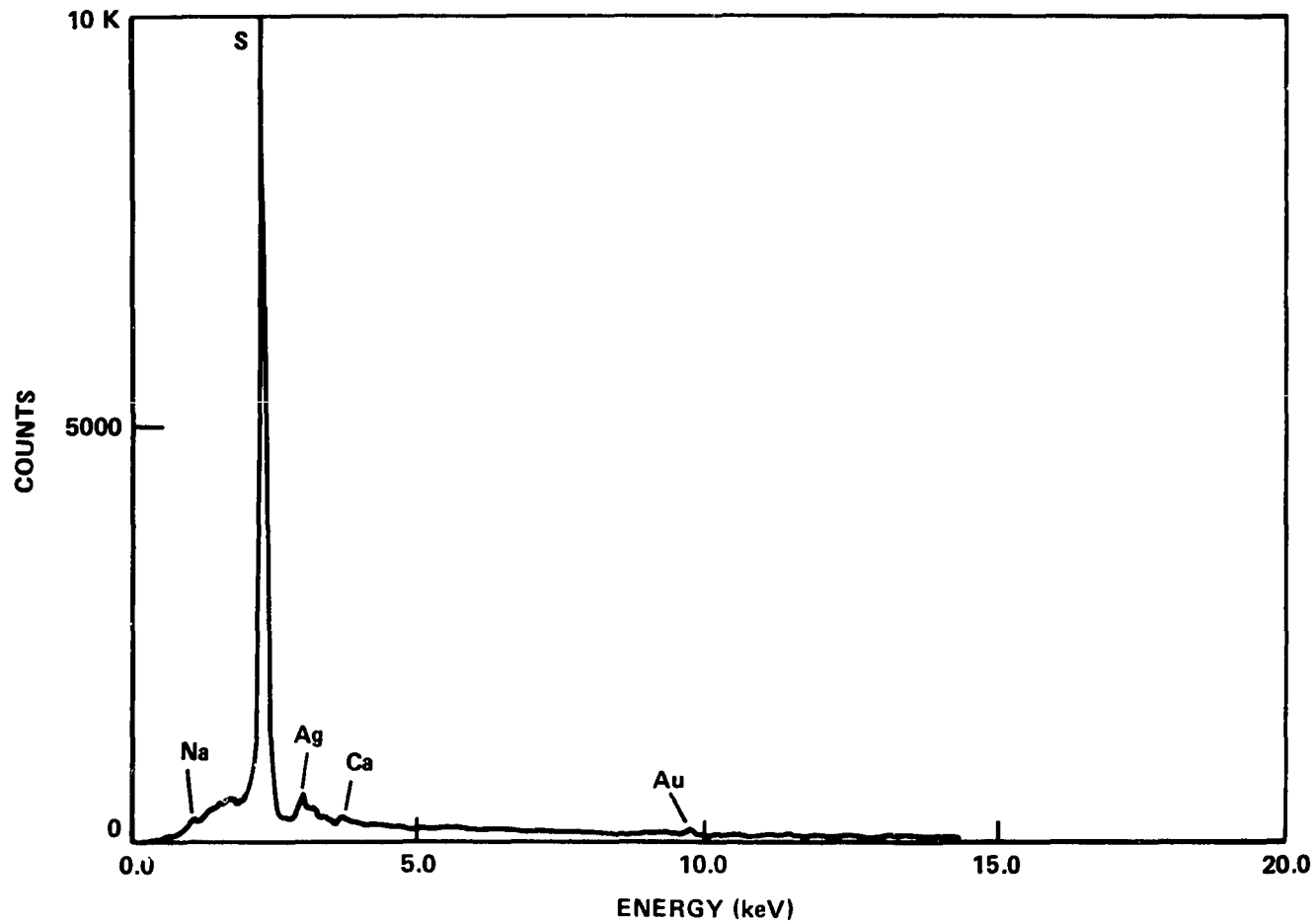


FIGURE 26. EDAX SCAN OF A MEDIA PARTICLE FROM BOTTOM REGION, 8-16" SEGMENT (Ag AND Au ARE MOUNTING AND COATING MATERIALS).

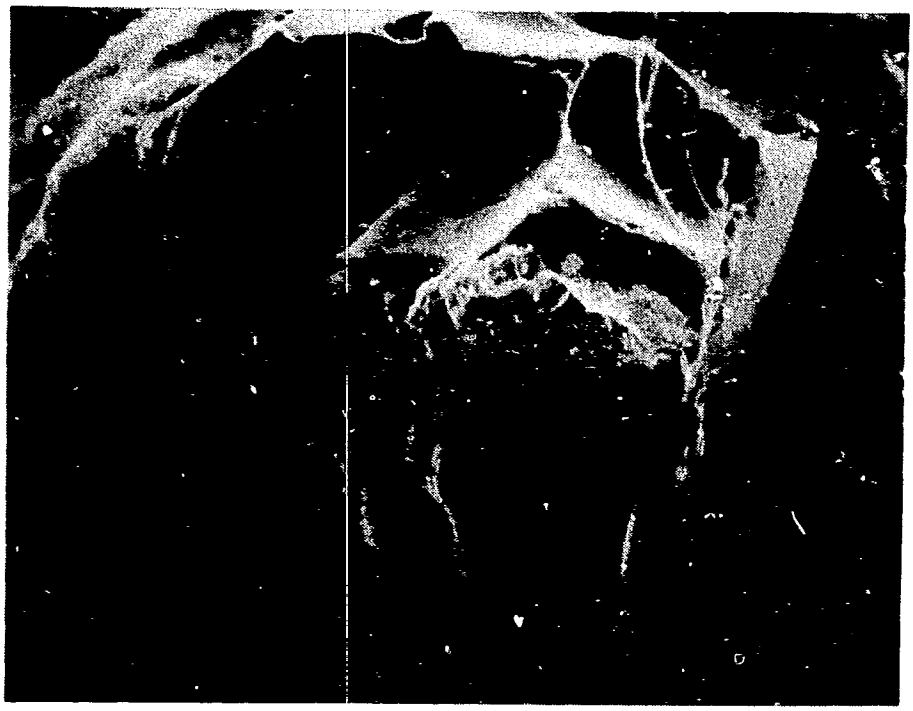


Figure 27. SEM photomicrograph of a typical granular particle, Top Region, 16-18" segment, 150X.

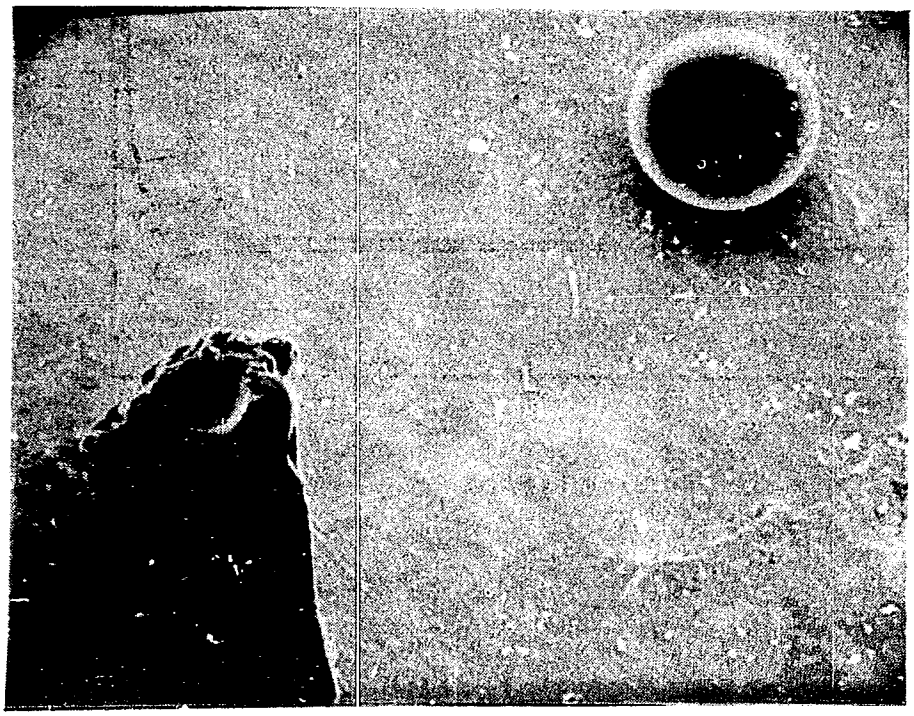


Figure 28. SEM photomicrograph of typical particles in the lower layer of the Top Region, 18-20" segment, 30X.

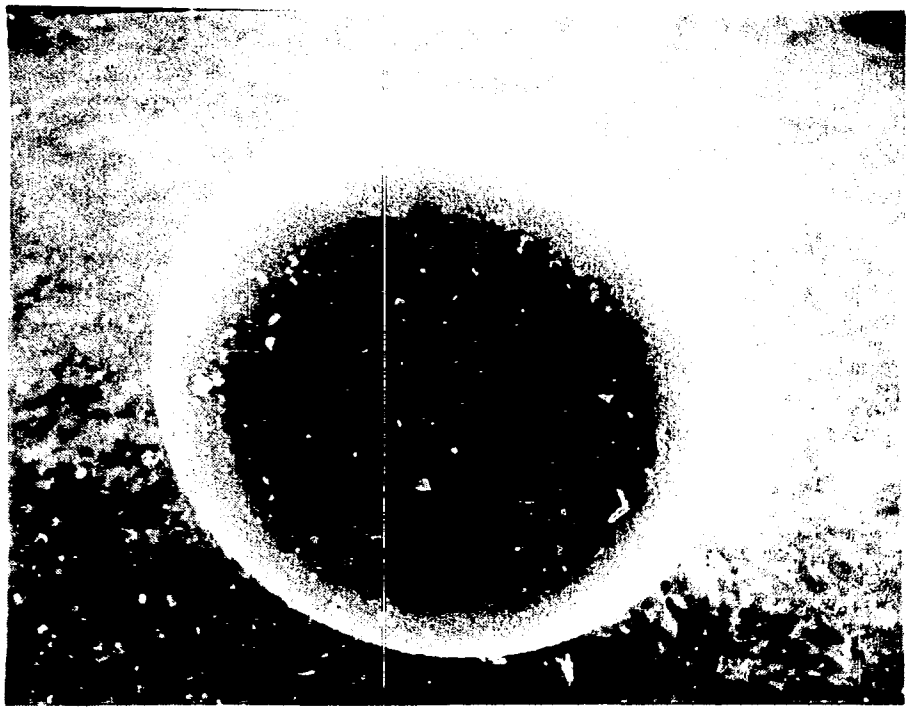


Figure 29. SEM photomicrograph of a typical spherical particle in the lower layer of the Top Region, 18-20" segment, 150X.

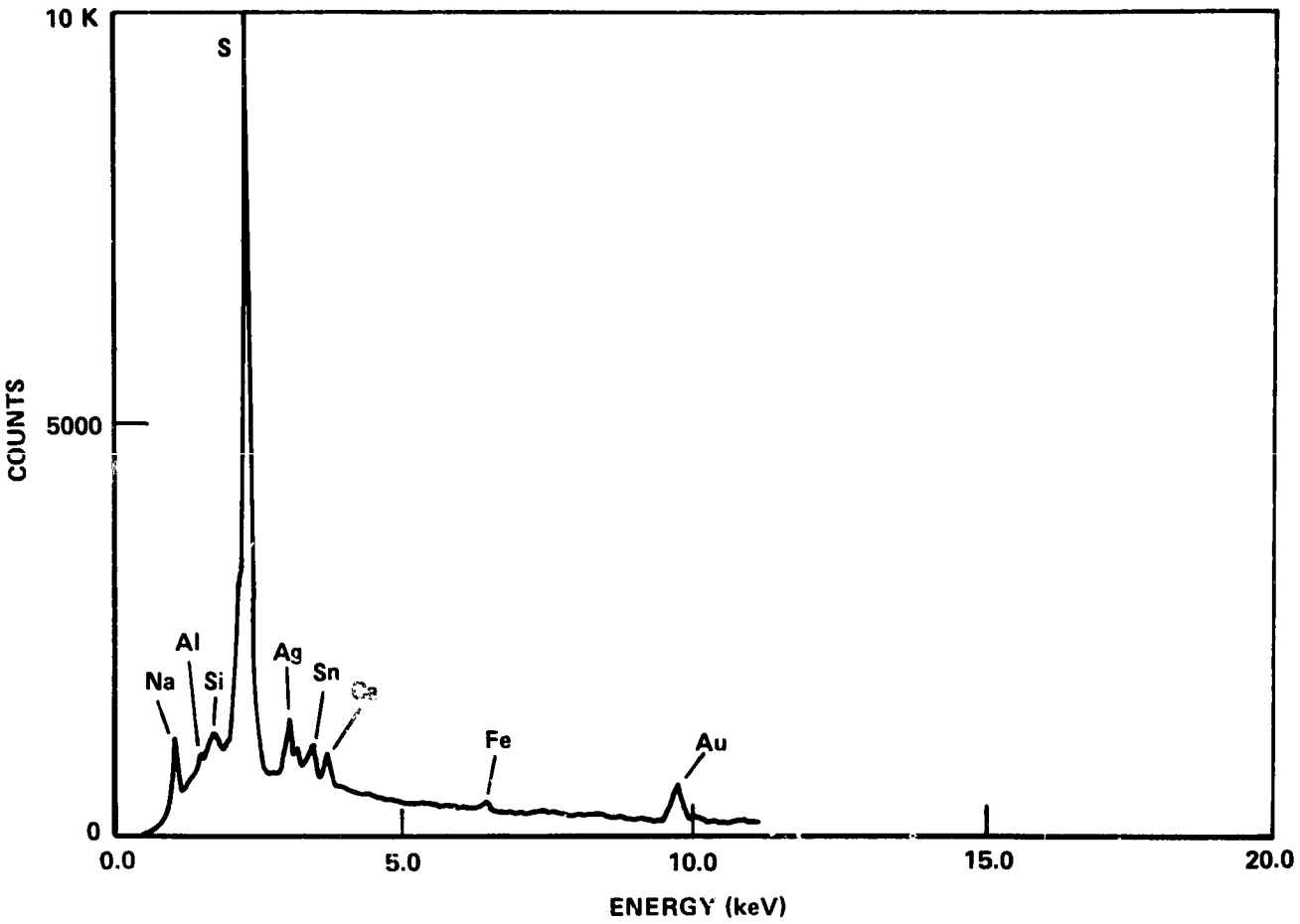


FIGURE 30. EDAX SCAN OF A MEDIA PARTICLE FROM TOP REGION, 16-20" SEGMENT (Ag AND Au ARE MOUNTING AND COATING MATERIALS).

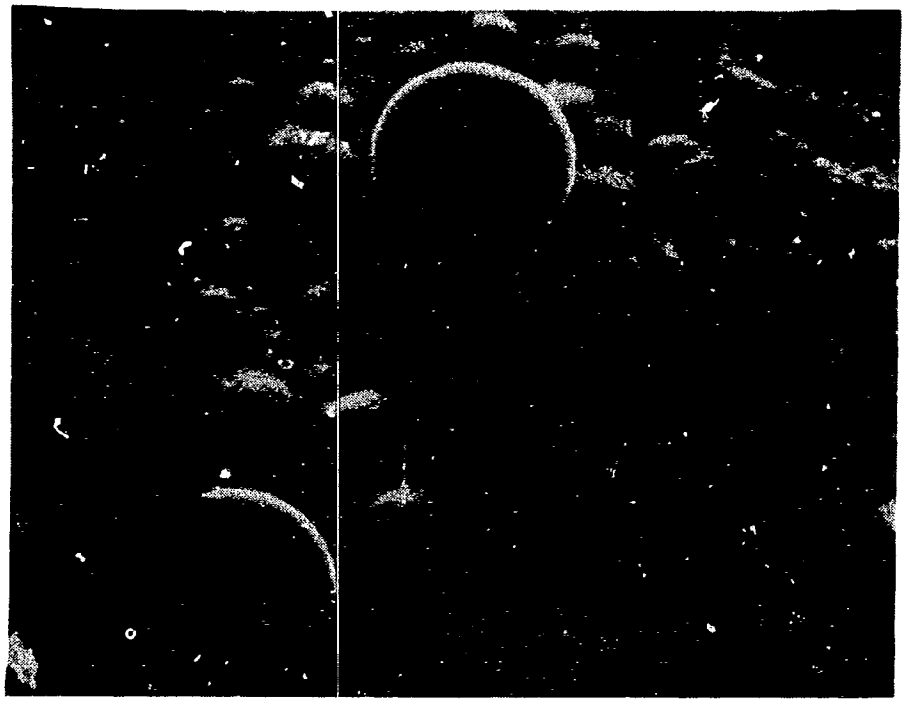


Figure 31. SEM photomicrograph of media particles, Top Region, 20-22", 30X.

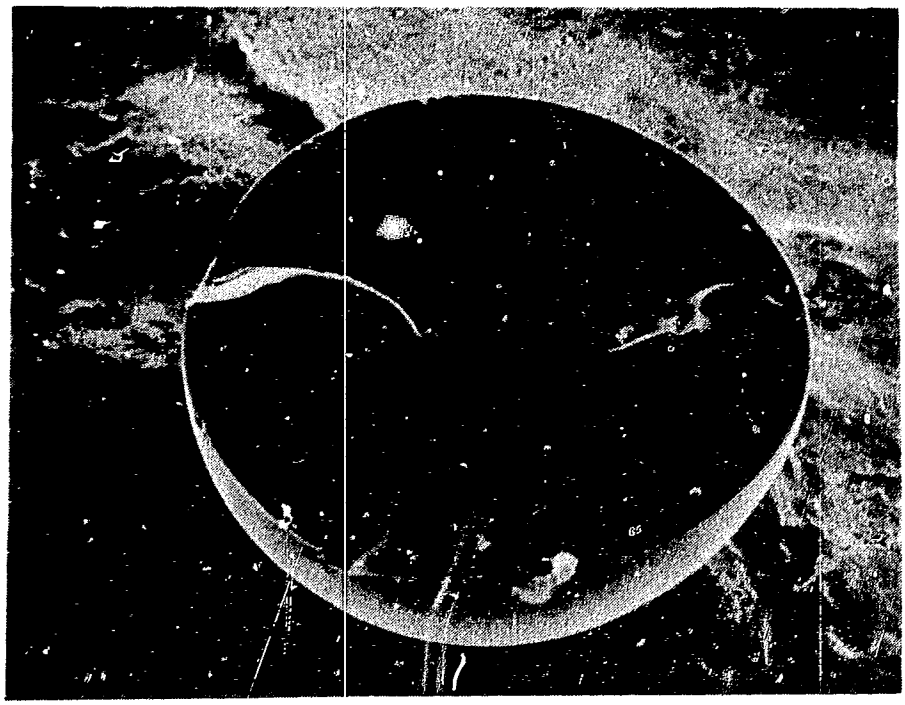


Figure 32. SEM photomicrograph detailing surface of fragmented particle, Top Region, 20-22" segment, 100X.

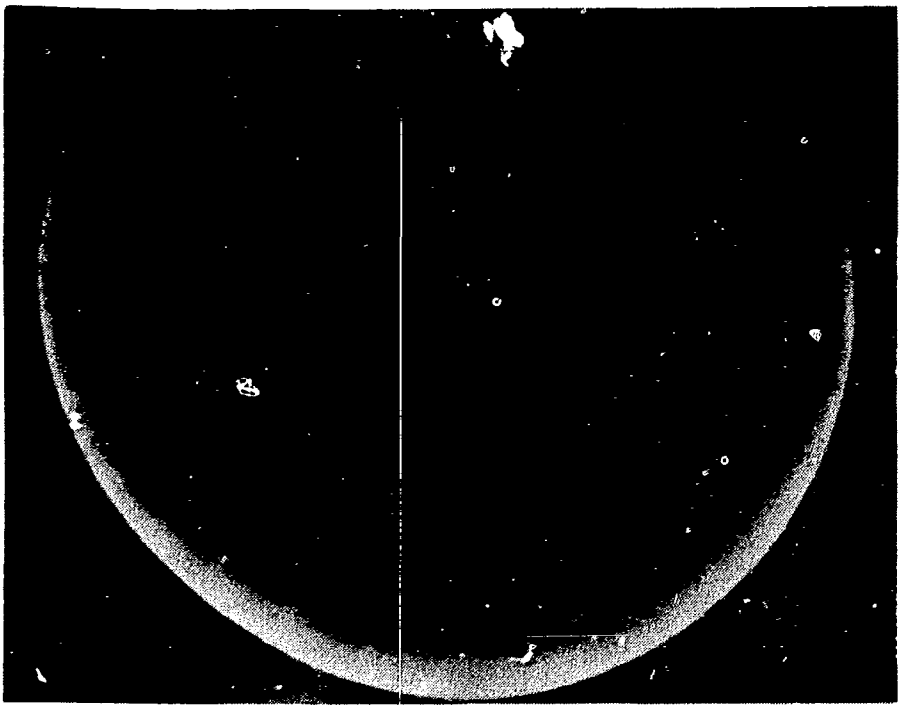


Figure 33. SEM photomicrograph of a typical particle in the upper layer of the Top Region, 22-24" segment, 150X.

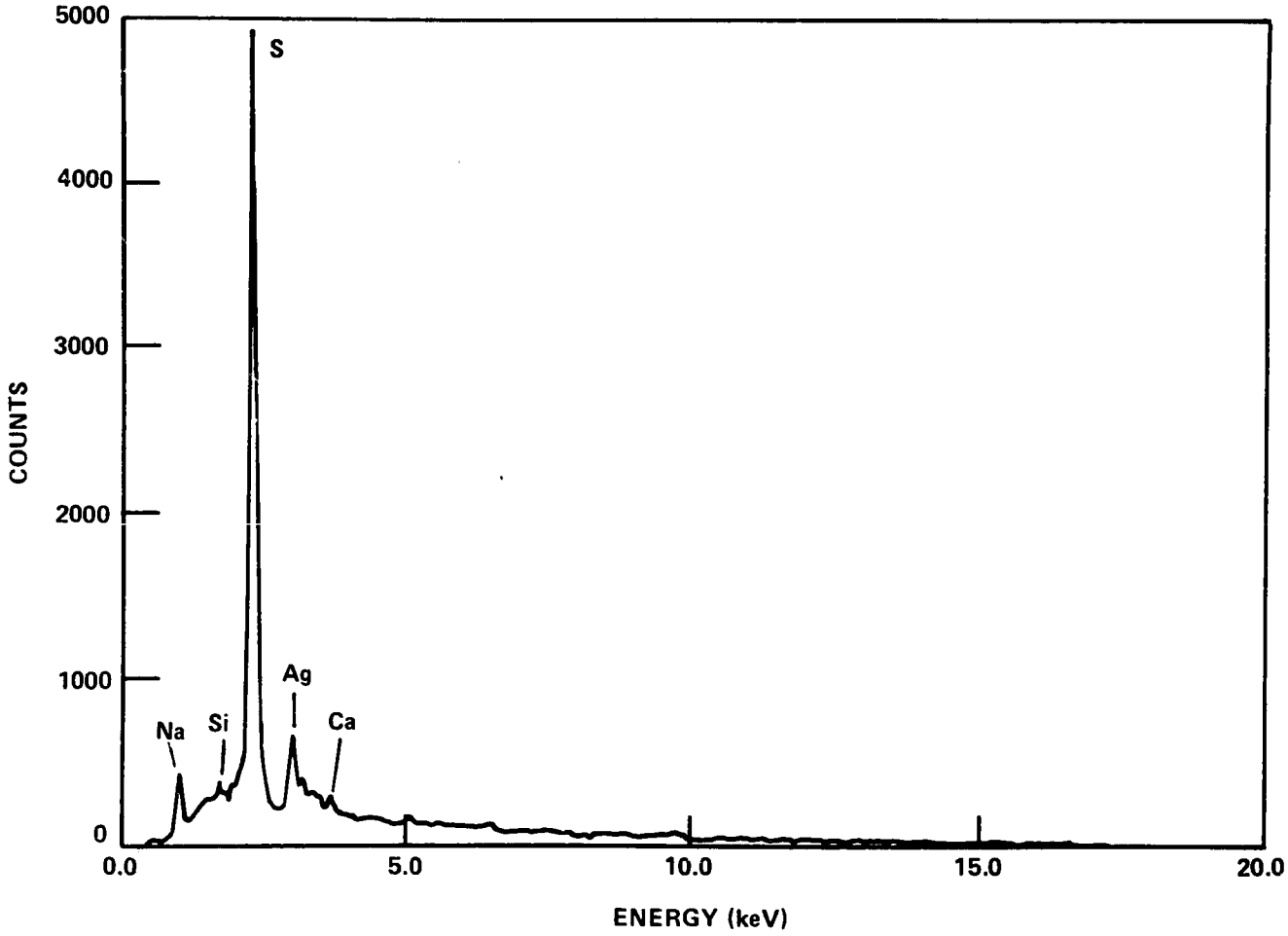


FIGURE 34. EDAX SCAN OF A MEDIA PARTICLE FROM TOP REGION, 20-24" SEGMENT (Ag IS MOUNTING MATERIAL).

In summary, deterioration of the ion-exchange media due to radiation exposure appears to be minimal, if any. Fragmented particles and surface cracking and spalling were observed only in the bottom region of the core sample, which is the region farthest from the highest activity loading.

RESIDUAL LIQUID SAMPLING AND ANALYSES

A liquid sample was obtained from the bottom of one of the caissoned holes from which a core sample had been removed. A 1/4-in. ID metal tube with a screen covering the top was connected to an evacuated collection vessel, using a length of polypropylene tubing. A total volume of 2 liters of liquid was collected. The first liter of liquid was obtained with relative ease, but the second liter was collected a few hundred milliliters at a time with time allowed for the caissoned hole to refill before attempting to collect more. It is suspected that very little liquid remains at the bottom of the liner.

The liquid sample was clear and colorless, with a small quantity of fines from the ion exchange media settled to the bottom of the sample container. Prior to analysis, the liquid was filtered through a 0.2 μm pore size filter in order to remove suspended materials. Specimens of the liquid sample were drawn and subjected to chemical and radiochemical analysis. The liquid was analyzed for cation content using an inductively coupled argon plasma spectroscopy technique, and for anion content using ion chromatography. An ion-selective electrode technique was used to determine the NH_4^+ content. An indication of the amine content was obtained by a total Kjeldahl nitrogen

measurement. Measurements were also made for the total organic carbon content, the liquid pH, acidity, conductivity, and total residue upon evaporation. Radiochemical analyses included gamma spectroscopy, gross alpha, gross beta-gamma, $^{89,90}Sr$, and Pu and U by mass spectrometry. The results of these analyses are presented in Tables 7 and 8.

Inspection of the data indicates that the residual water has low ion content. The 16 μmho/cm conductivity is indicative of relatively clean water. No significant concentration of corrosion products has been found and there is no evidence of large concentrations of ion-exchange media degradation products. The only significant chemical species in the liquid was the 2200 ppm of boron which is not effectively removed by the ion-exchange media.

The observed residual liquid pH is 5.3. This weak acid would not be expected to present a corrosion hazard to the liner steel.

PREPARATION OF LINER FOR SHIPMENT

Because of problems associated with cask availability, shipment of the liner could not be made for several months after completion of the liner examination. It was decided to remove the liner from the hot cell to prevent contamination of the liner.

Prior to removal of the liner from the cell, the exposed surfaces were decontaminated using damp cloth and paper towels. Smear samples were taken and counted to confirm that the smearable contamination was below acceptable limits. Another requirement imposed on the liner prior to shipment was that it be purged with nitrogen within eight days of the shipment date to remove oxygen and hydrogen from the liner. To accomplish the nitrogen purging step

TABLE 7. RESIDUAL LIQUID CHEMISTRY ANALYSIS

pH	5.3 ± 0.1 at 20°C
Conductivity	16 μmho/cm at 20°C
Acidity	4.2 meq/l at pH 7.0
Total residue upon evaporation	7.5 ± 0.1 mg/ml
	ppm
Sodium	5.7
Iron	0.35
Phosphorus	<0.1
Zinc	0.19
Magnesium	0.095
Calcium	0.53
Aluminum	<0.05
Boron	2200
NH_4	9 E-5
SO_4	1.44
NO_3	0.31
Chlorine	1.25
Total organic carbon	6.45
Total Kjeldahl nitrogen (TKN)	0.5

TABLE 8. RESIDUAL LIQUID RADIOCHEMISTRY ANALYSIS (μCi/ml)

Gross beta/gamma	1.36 ± .01 E-2
Gross alpha	7.5 ± .1 E-4
^{90}Sr[a,b]	1.04 ± .05 E-3
^{60}Co[c]	9.3 E-5
^{134}Cs[c]	2.1 E-3
^{137}Cs[c]	4.2 E-2
$^{239, 240}Pu$[b]	6.9 ± .7 E-6
^{241}Pu[b]	<2 E-4
^{238}U[b,d]	8.5 E-9

[a] ^{89}Sr was not detected.
[b] Activity on 12/14/82.
[c] Activity on 12/16/82.
[d] ^{235}U was not detected.

with the liner out of cell a special fitting had to be installed. This involved removal of the off-gas vent plug and replacing it with a fixture shown in Figure 35. Design of this fixture was such that a "quick-connect" type fitting can be attached to backfill the liner with nitrogen. The fixture also has a valve that can be closed off after the purging operation. Design of the fixture was reviewed and approved by EG&G-Idaho.

After the installation of the remote purging fixture, the liner was removed with the aid of the transfer/storage device. The top plate of the transfer/storage device was modified so that a hose can be attached to the purging fixture. The liner was stored with the vent valve open to avoid buildup of a combustible gas mixture.

The liner was shipped to EG&G-Idaho on January 6, 1983. Prior to shipment the liner was purged with nitrogen a number of times. Analysis of a sample of gas obtained from the liner showed it to contain more than 98% N_2. It was suspected that there was a leak in the manway cover seal preventing an efficient purging operation.

SUMMARY OF OBSERVATIONS

1. Characterization of EPICOR Liner PF-3 showed that the liner in general was in good condition. Visual examination of the exterior showed only surface scratches and occasional rust spots.

2. Liner interior visual examination suggested that the corrosion of the liner inner wall was somewhat more than that observed in liner PF-16. Liner wall in contact with the resin media near the top surface also

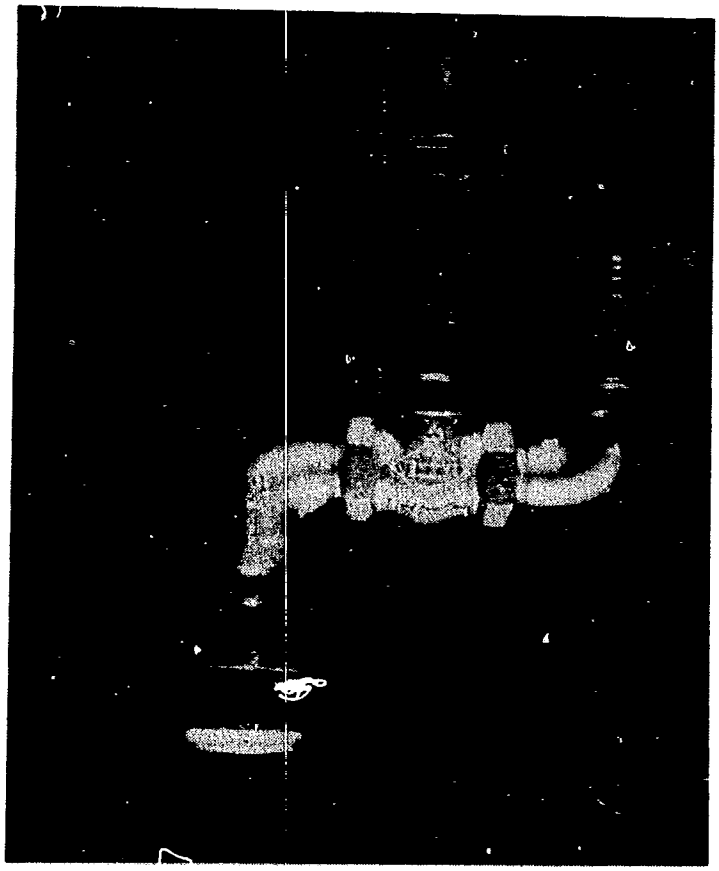


Figure 35. Remote purging fixture installed on liner PF-3 in place of the off-gas vent plug.

appeared to corrode more than in liner PF-16. The protective coating appears to be loosely adherent in this area with corrosion of the wall underneath the coating.

3. Degradation of the media due to radiation appeared to be minimal, if any. The only evidence of cracking and fragmentation of the media particles was found, not in the top region of the media bed where most of the gamma-emitting radionuclides were deposited, but in the bottom of the media bed. In this bottom region, the resin particles showed significantly more cracking, spalling and fragmentation than in liner PF-16.

4. The media pH determinations showed that the media is acidic in nature, with the pH decreasing from top to bottom in the media bed.

5. The top region of the media bed was very effective in removing both cesium and strontium from the contaminated water; plutonium and uranium however were uniformly distributed throughout the media.

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