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CHARACTERIZATION OF EPICOR II PREFILTER LINER 16

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ABSTRACT

As part of the overall TMI-2 Information and Examination Program, EPICOR II Prefilter Liner 16 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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CHARACTERIZATION OF EPICOR II PREFILTER 16 LINER

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, EPICOR II Prefilter Liner 16 (PF-16) was examined at Battelle Columbus Laboratories (BCL) to aid in the development of technology for safely processing contaminated ion-exchange media and to gain information on the shelf life of these media and liners.

Among the events that occurred during and immediately following the March 1979 accident at TMI-2 was the transfer of more than 500,000 gal of contaminated water to the Auxiliary and Fuel Handling buildings. The principal sources of this water were the makeup and letdown purification system and the containment building sump. One of the early tasks in the recovery operation was processing this water.

The contaminated water was processed through an ion-exchange system designated EPICOR II. The contaminated water passes through three stages of ion-exchange media. The first is designated the prefilter and the second and third stages are called the demineralizers. A cross-sectional view of a typical EPICOR II liner is shown in Figure 1. During operation, the majority of the activity is deposited in the first stage prefilters.

The EPICOR II prefilter liner is a 5-ft-high right circular cylinder 4 ft in diameter, containing $\sim 30 \text{ ft}^3$ 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 liners are believed to contain zeolites and three 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.

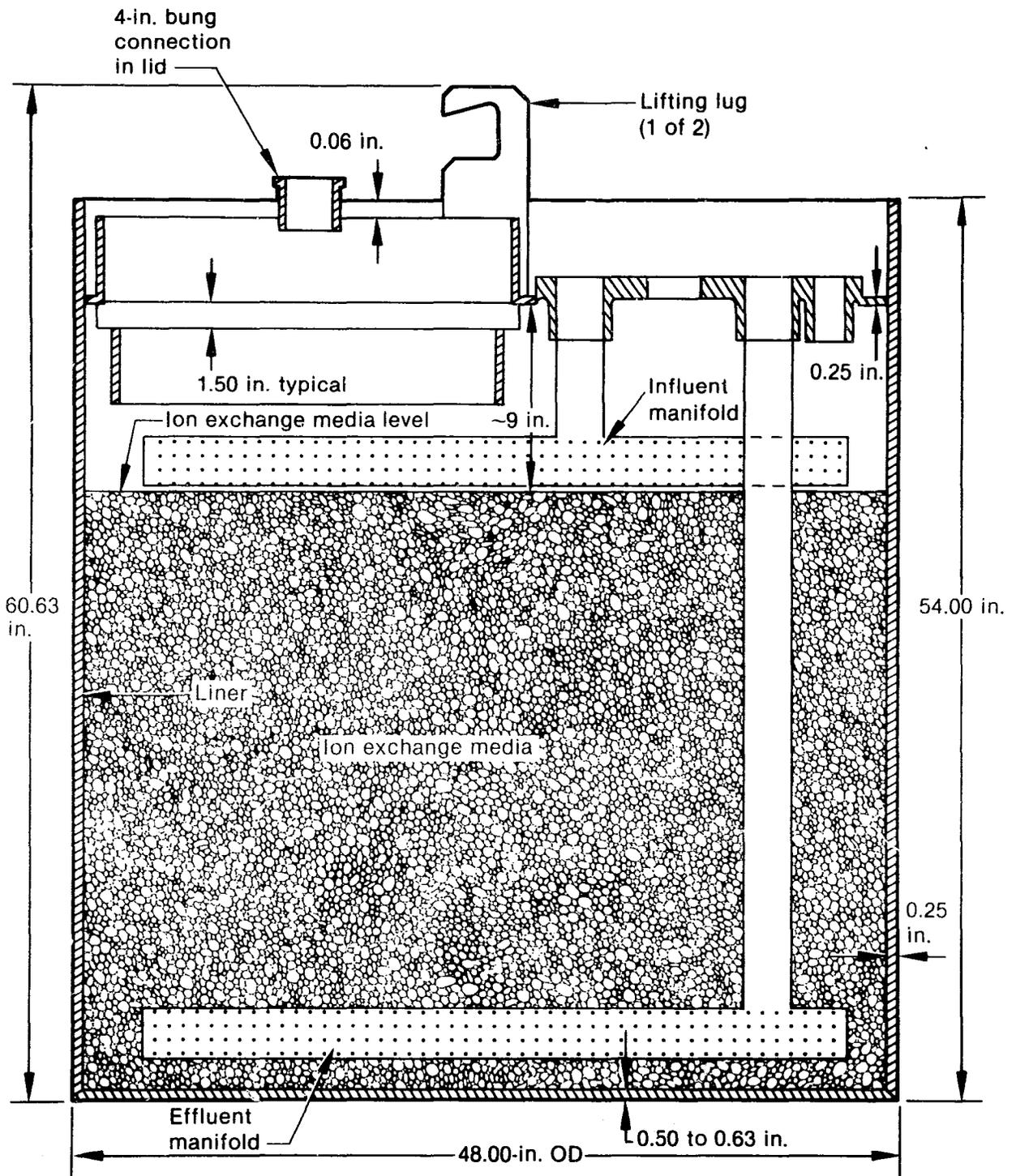


Figure 1. Cross-sectional view of a typical EPICOR II liner.

The liner selected for characterization is EPICOR II Prefilter Liner 16 (PF-16). Schematic views showing the configuration of the liner lifting lugs and penetration plugs are presented in Figure 2. This liner was in service during March 3 and 4, 1980, and processed a total of 8,250 gal of water removed from Reactor Coolant Bleed Tank A. On the basis of analyses of the influent water, it was estimated that the curie loading of PF-16 was ~1250 Ci of mixed fission products. A summary of the chemical and radiochemical analyses of the influent water is presented in Table 1. From an inspection of the radiochemistry data, it is clear that the primary nuclide (~80% of the total activity) was ^{137}Cs . During final operations by General Public Utility Nuclear Corporation (GPUNC) personnel, an effluent pH of 2.79 was measured.

The PF-16 characterization includes receipt of the liner, in-cell examinations, sampling and analysis of liner contents, and shipment of the liner to the Idaho National Engineering Laboratory (INEL).

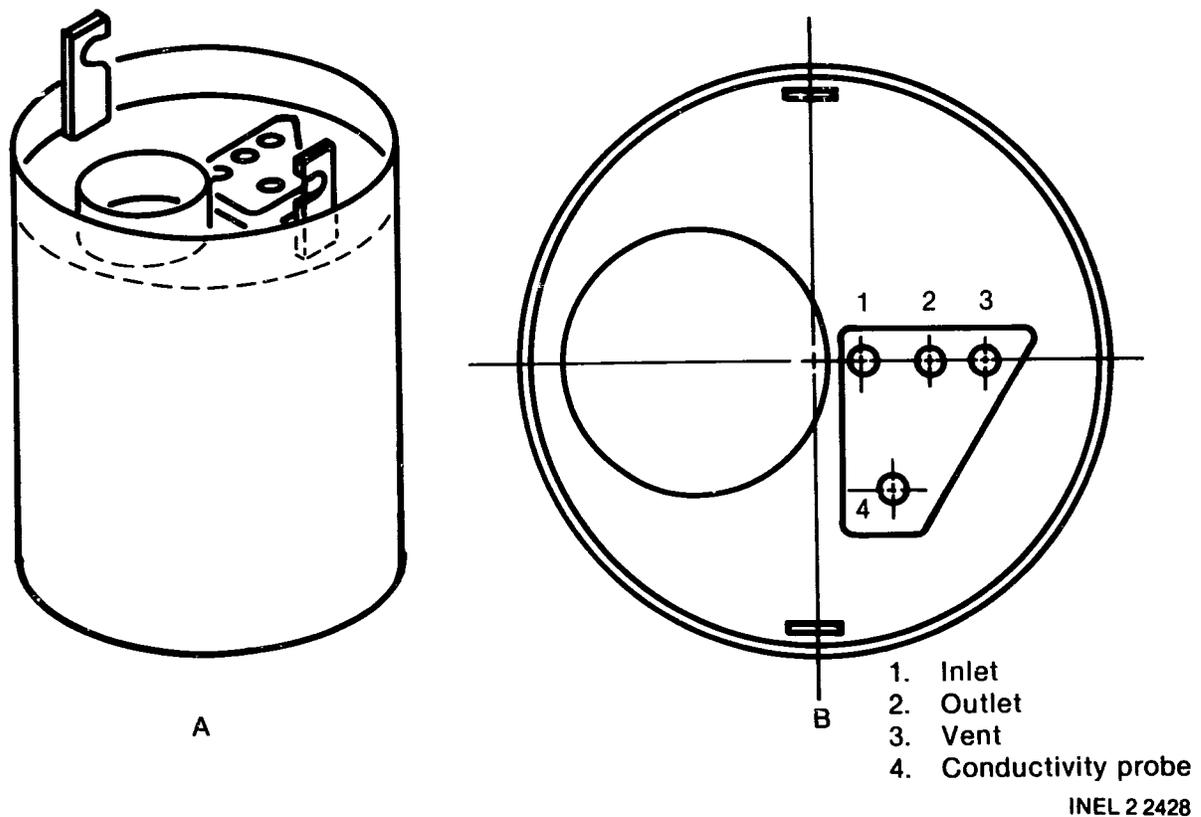


Figure 2. Schematics showing (a) orientation of PF-16 lifting lugs and (b) configuration of PF-16 penetration plugs.

TABLE 1. CHEMISTRY AND RADIOCHEMISTRY OF PF-16 INFLUENT WATER

Chemistry	
pH	7.93
Conductivity	1200 $\mu\text{mho/cm}$
Boron	1341.7 ppm
Sodium	220 ppm
Radiochemistry	
	<u>($\mu\text{ Ci/ml}$)</u>
Gross $\beta\gamma$	4.01 E+1
Gross α	5.89 E-4
^{137}Cs	3.176 E+1
^{134}Cs	6.068 E+0
^{140}La	<2.016 E-1
^{140}Ba	<1.4 E-1
^{131}I	<5.908 E-2
^{58}Co	<8.55 E-2
^{60}Co	<7.27 E-3
$^{89,90}\text{Sr}$	\sim 1.43 E+0

SAFETY ANALYSIS

The BCL Operational Practice calls for 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 accordance with this practice, a detailed description of the operations associated with the receipt, handling, and examination of the PF-16 liner was submitted to the RSC for review and approval. This document addressed the potential hazards associated with each of the major operations and defined procedures and administrative steps to be taken to minimize the probability of problems or their consequences.

It was presumed that a combustible gas mixture might have been generated inside the liner during storage at TMI, the result of radiation-induced degradation of the resins and the radiolysis of the contaminated water. It was also conservatively assumed that the gas mixture could combust if ignited by sparks generated under appropriate conditions during gas sampling operations. However, in subsequent communications from EG&G/TIO, BCL was informed that the liner would be vented (vent plug removed for >1 h) prior to shipment from TMI and, therefore, would not contain combustible gas mixtures at the time of arrival at BCL. It was further assumed that the gas sampling operations could be performed within two weeks of the date of liner venting and still be below the lower flammability limit. This information was presented to the RSC.

Another hazard associated with the liner handling operations was exposure of operating personnel to excessive amounts of radiation. It was pointed out that the use of a transfer device during cask unloading operations would significantly reduce personnel exposure. In addition, continuous monitoring of the radiation levels in the work area by the Health Physics staff would provide information to further reduce personnel exposure to radiation.

Based on a review of the information presented, the RSC approved all operations involving receipt, handling, examination, and storage of the liner.

LINER PREPARATION AND SHIPMENT

On the basis of a Brookhaven National Laboratory (BNL) report¹ concerning the effects of radiation on ion-exchange resins, it was believed that resin degradation had, through radiolysis, produced combustible gases within the liner. A GPU combustible gas source term evaluation² indicated that the predominant combustible species present would be hydrogen, with the worst-case concentration of the order of 13 volume %. Because the lower combustible limit of hydrogen is 4 volume %, it was felt that venting the liner prior to shipment was necessary to minimize hazards. It was concluded by EG&G Idaho that venting the liner and shipment to BCL within 15 days of recapping the vent penetration would ensure that hydrogen generated during transit would not result in a combustible configuration. Based on GPU's worst-case combustible source term, it was concluded that the hydrogen generation in 15 days would not lead to a level in excess of 0.54 volume %. This value is well below the lower flammable limit and makes the risk of combustion during shipment negligible (see Reference 2).

On May 16, 1981, the liner was vented at TMI in the Chemical Cleaning Building. At 2:00 p.m. on May 16, 1981, the liner vent plug was partially removed (unscrewed four turns). At this time a combustible mixture of gas was detected and personnel were evacuated from the area around the liner. The detection of a combustible gas mixture continued for ~5 h. The vent plug was completely removed at 5:30 a.m. on May 17, 1981, and a new plug inserted at 6:45. Attempts to remove the effluent plug were unsuccessful.

The liner was loaded into the lead/steel transfer device for subsequent transfer into Cask CNS 8-120 (ATCOR LL-50-100) leased from Chem Nuclear, Inc. The cask, a licensed Type B, meets all applicable Department of Transportation (DOT) and Nuclear Regulatory Commission (NRC) requirements. The cask shielding consists of two layers of steel, each ~1 in. thick, separated by 3-1/2 in. of lead. The cask lid and gasket are designed to withstand 0.5 atmospheres of internal pressure. The actual transfer occurred on May 17, 1981, and subsequent shipment of the liner and cask to BCL occurred on May 19, 1981.

TRANSFER/STORAGE DEVICE

A transfer and storage device was designed at BCL and fabricated by local contractors to (a) transfer the liner from the shipping cask into the hot cell and (b) provide a shielded storage container for the 24-month storage and monitoring of the liner outside of the hot cell.

The transfer/storage device is a cylindrical lead-steel shield with removable top and bottom shielding cover plates. The design of the device was based on shielding calculations made with the objective of a maximum outer surface radiation reading of ~ 50 mR/h. The inner and outer walls of the cylindrical body are steel. The 3-in. space between these two concentric steel cylinders is filled with lead. The lead provides the majority of the shielding, and the steel provides the necessary structural strength. The inner and side surfaces of the top and bottom plates are 1/4-in. thick steel and the outer surface is 1/2-in. thick steel. A 2-in. space between the inner and outer surfaces in each of the cover plates is filled with 2 in. of lead. As in the case of the main body, the lead provides the primary shielding and the steel provides the structural strength. The overall height, not including lifting lugs, is 67-1/2 in., the inside diameter is 54 in., and the outside diameter is 62 in. The transfer device is shown in Figure 3.

Before the PF-16 liner arrived at BCL, the transfer/storage device was tested using a dummy liner to ensure the device would function properly. Figure 4 shows the device resting on top of the shipping cask containing the liner.

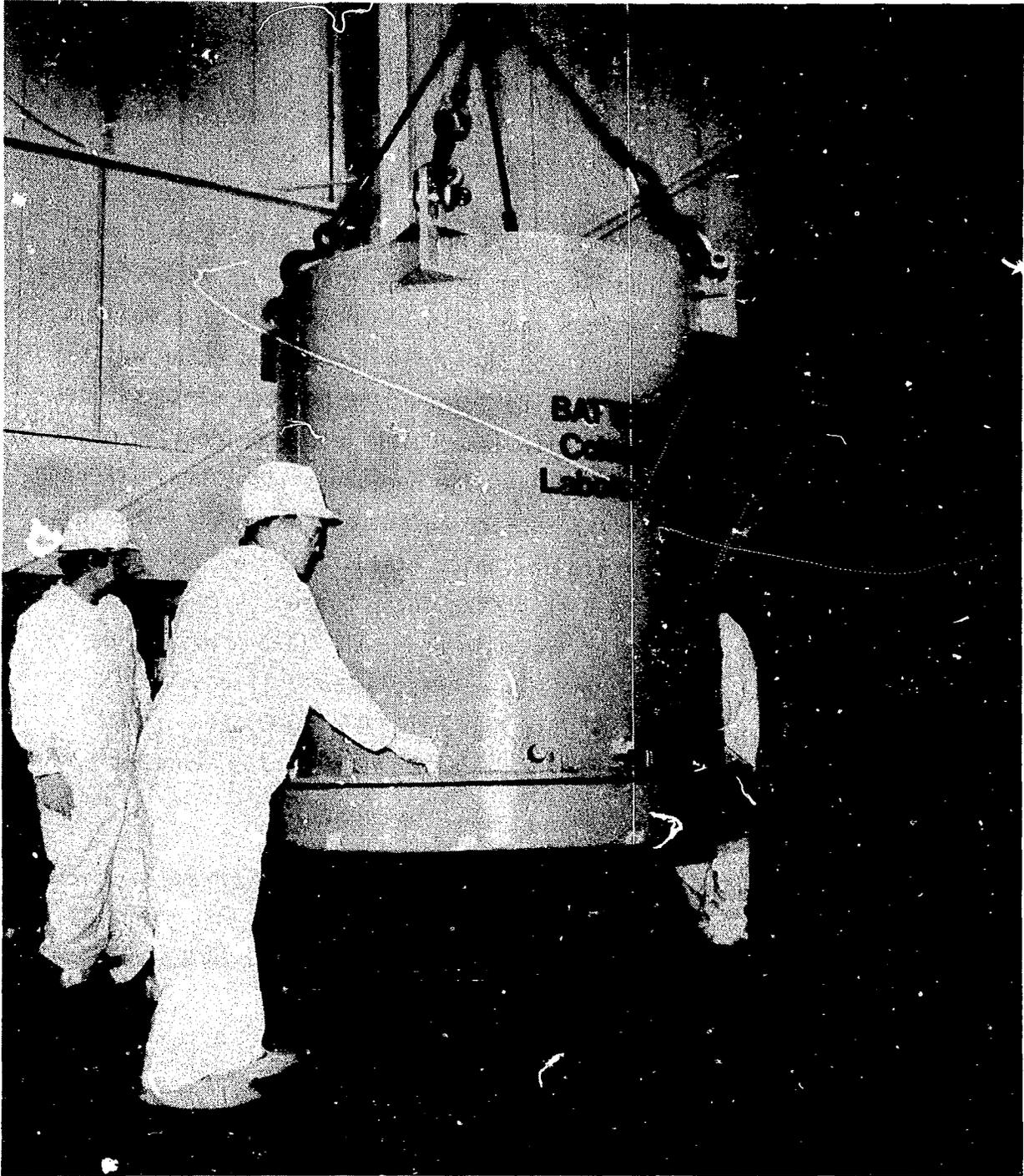


Figure 3. BCL designed liner transfer/storage device.

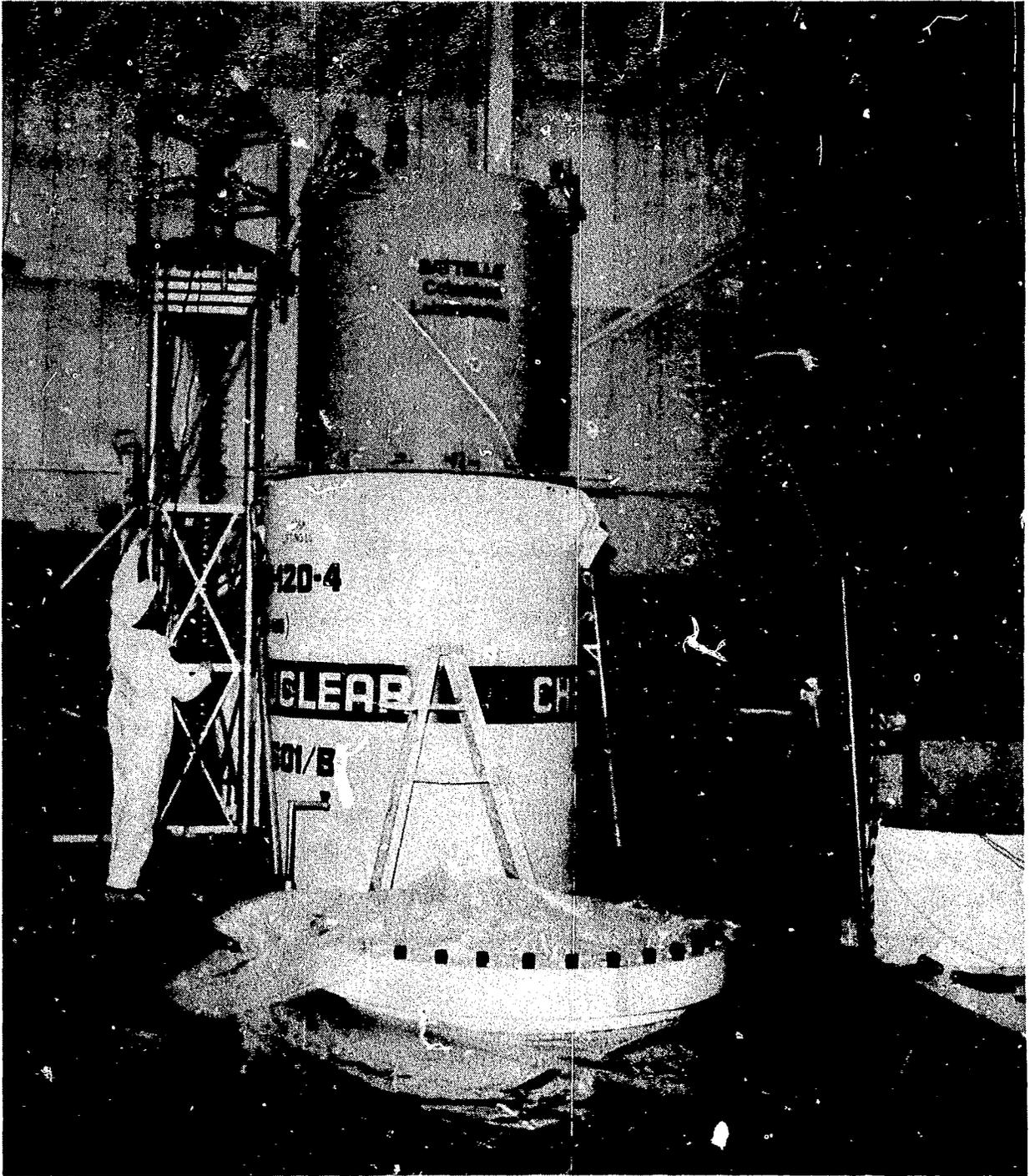


Figure 4. Transfer/storage device resting on top of the shipping cask.

LINER RECEIPT AT LABORATORY AND TRANSFER INTO HOT CELL

Liner PF-16 was received May 19, 1981, at Battelles Nuclear Center, West Jefferson, Ohio. Preliminary radiation and smear surveys were conducted by BCL Health Physics personnel. Preliminary readings were taken before the rain shield was removed and while the cask was on the trailer. The maximum radiation reading at the cask surface was 7 mR/h($\beta\gamma$) and the readings fell to a maximum of 3.5 mR/h($\beta\gamma$) in the vertical plane at the trailer edge nearest the cask. The radiation reading at the truck cab was <2 mR/h($\beta\gamma$). The smear survey indicated the contamination levels were well below the 2200 dpm/100 cm²($\beta\gamma$) and 220 dpm/100 cm²(α) limits.^a These readings represent primarily the radiation and contamination of the cask sides and were the basis for accepting the shipment. The low-boy trailer was backed into the Hot Laboratory building truck dock area and the tractor was uncoupled.

The cask rain shield was removed the next day, May 20, 1981, and the tiedown bars detached. It was found that the rain shield binders were not tight and the rain shield was slightly off center. Also, the cask tiedowns were loose and could be moved slightly by hand. After removal of the rain shield and tiedowns, more detailed radiation and smear surveys were conducted by the BCL Health Physics personnel. A maximum radiation reading of 70 mR/h($\beta\gamma$) was registered at the cask lid. The smear survey yielded a contamination level of <200 dpm/100 cm²($\beta\gamma$) and <4 dpm/100 cm²(α). After completing the surveys, the cask was removed from the trailer using a 50-ton crane.

A bubble solution (SnoopTM) was applied around each of the six cask lid plugs and no leakage of gas from the cask was detected. A gas sampling chamber had been constructed using the cask drawings.^b This chamber was designed to mate with the top of the cask. However, the cask lid plugs had

a. 49-CFR, 173.393 (j).

b. ATCOR Inc. 1042-D0021, Rev. C, and 1042-B-0005.

raised, 1-1/4 in. square, male fittings rather than the drilled holes and handles shown in the reference drawings; therefore, a new chamber handle had to be fabricated.

The sampling chamber was then attached over one of the cask lid plugs, the chamber was evacuated, the plug was loosened, and the internal air was sampled. An area gas monitor was operated during the sampling operation; no release of activity was observed and no activity was detectable from within the sample bulb. Therefore, the sample chamber was removed and the cask plug removed completely. A radiation and contamination survey was conducted. The radiation readings were >500 R/h inside the plug hole near the liner, 100 R/h inside the plug hole about 2 ft above the liner, 50 R/h at the top of the cask plug hole, and 2 R/h at 3 ft above the cask plug hole. A smear taken from inside the plug hole exhibited contamination of <500 dpm/100 cm² (βγ). The cask drain plug was removed and found to be dry and clean. The drain and cask lid plugs were reinserted and tightened. Because there was no detectable pressure (detection limit = ±0.25 psi) or radioactive air in the cask, purging was not required.

Following the radiological surveys, succeeding operations were removal of the cask lid, and preparation of the liner for transfer into the hot cell. During these operations, radiation from the cask was continuously monitored by the BCL Health Physics personnel. The cask lid was raised about 6 in. and a radiation reading of about 190 R/h was obtained in the opening between the cask body and the raised lid near the outside edge of the cask. A reading was taken to determine the radiation streaming directly from the liner through the opening between the cask body and lid. The monitor was raised and lowered at a distance of about 2 ft from the cask. A maximum reading of 200 R/h was obtained at a height of about 2 ft above the top edge of the cask body. With the cask lid fully removed, the radiation level 2 ft above the top of the cask centerline was also 200 R/h. A contact radiation reading at the side of the cask a few inches below the top was only 200 mR/h.

The transfer/storage device was used for the transfer of the cask into the hot cell. Using the adaptor plate to distribute the weight around the

cask top opening, the transfer/storage device was positioned on top of the cask (see Figure 4). Radiation readings were taken with the device on top of the cask, and before the liner was raised into the device. With the device top shield in place, the maximum radiation through the shield was 100 to 150 mR/h. A contact radiation reading at the mating surface of the transfer/storage device and the cask was 10 to 20 R/h. The radiation reading fell to 2 R/h at a distance of 1 ft from the cask in the same horizontal plane at the top of the cask. The device top shield was removed and the radiation reading at the open top (about 7 ft above the unshielded liner) was 50 R/h. The liner was then raised into the transfer/storage device, properly positioned, locked into place, and the device cover shield installed. The contact radiation readings were generally 1 to 1.5 R/h, with a maximum of 2 R/h through the device top shield. The transfer/storage device, with the liner inside, was then lifted from the cask and the bottom shield attached. The contact radiation through the side of the transfer/storage device, with the liner inside, was 35 mR/h near the top, 150 mR/h about half the way down, and 20 mR/h near the bottom. The maximum contact radiation reading was 150 mR/h through the side. The contact radiation reading through the bottom shield was 50 mR/h.

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 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 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 unnecessary contamination of the liner surface.

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 May 29, 1981. The general approach was first to affix a sampling chamber (see Figure 5) to the top of the liner using an electromagnetic clamping device. The chamber was then evacuated and the liner plug removed using a T-bar wrench penetrating the sampling chamber through an elastomer seal. The chamber was fitted with a vacuum/pressure gauge calibrated in inches of mercury and readable to within 0.5 in. of mercury. While the vent plug was being removed, the vacuum/pressure gauge was being continuously monitored. As the gas was

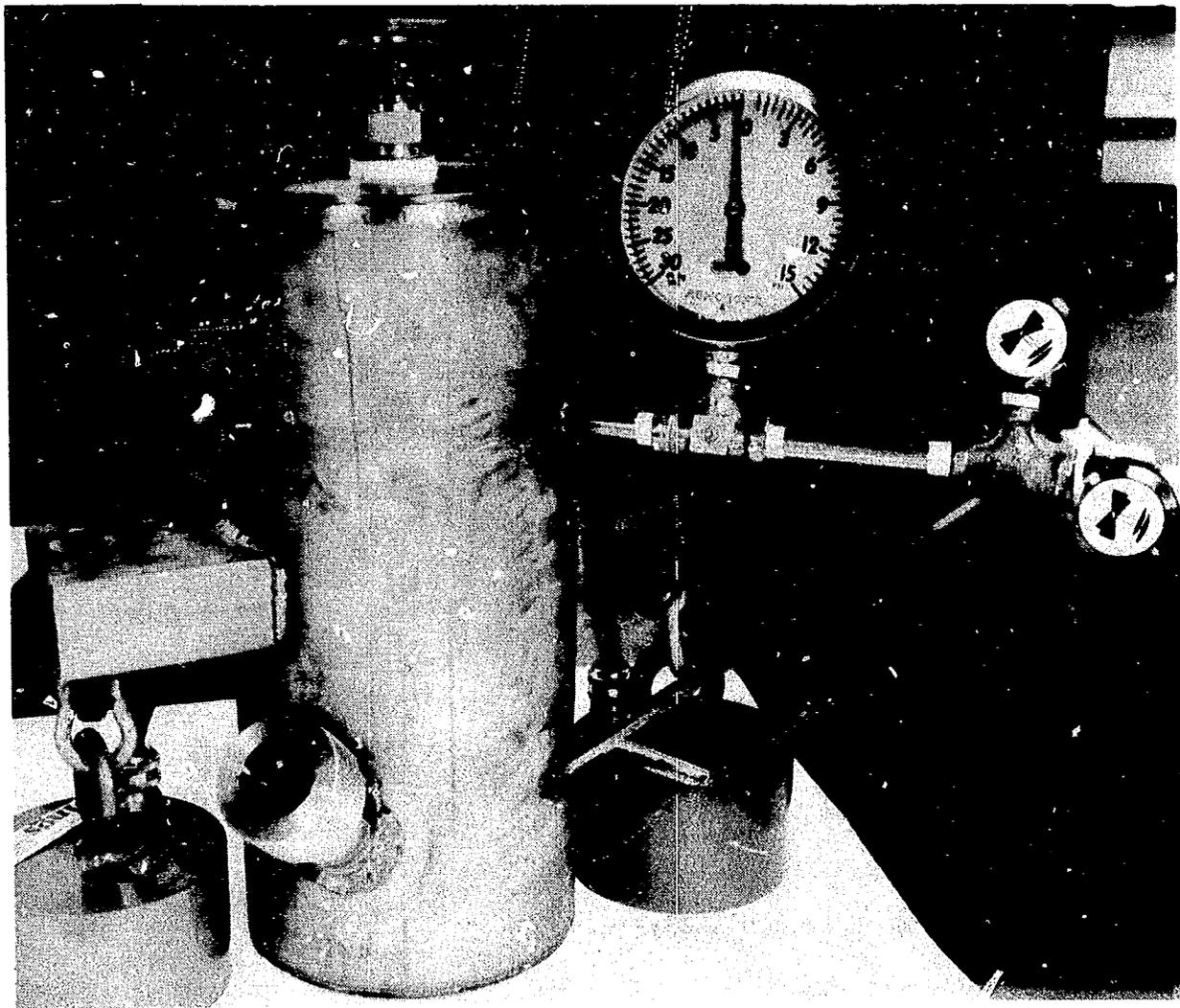


Figure 5. Gas sampling device.

released into the evacuated sampling chamber, the gauge indicated that the internal pressure did not exceed atmospheric pressure. After the chamber had filled, an evacuated one-liter gas bottle that was coupled to the system was opened to collect a sample. Following the first sample collection, the vent plug was closed, and the chamber evacuated before another one-half-liter sample was collected.

The gas samples were submitted for analysis using mass spectrometry followed by gas chromatography. The one-liter sample is designated Sample 1 and the 500-ml sample is designated Sample 2. The results of the analyses are shown in Table 2, which clearly indicate the gas is highly enriched in

TABLE 2. PF-16 GAS ANALYSIS

	Volume Percent		
	Light Fraction		Heavy Fraction
	Sample 1	Sample 2 ^a	
Carbon dioxide	5.52 ± 0.06	5.27 ± 0.06	0.30 ± 0.03
Argon	0.96 ± 0.05	0.96 ± 0.05	0.94 ± 0.05
Oxygen	0.20 ± 0.02	0.30 ± 0.05	20.2 ± 0.2
Nitrogen	80.6 ± 0.4	81.2 ^b ± 0.5	78 ± 0.4
Carbon monoxide	0.2 ± 0.02	--	0.004 ± 0.001
Hydrogen	12.4 ± 0.2	12.2 ± 0.02	0.5 ± 0.05
	Volume (ppm)		
Methane	500 ± 2.5		45 ± 5
Ethylene and acetylene	0.7 ± 0.1		0.1
Ethane	42 ± 4		4 ± 1
Propylene	0.1		0.1
Propane	6 ± 1		1 ± 0.2
Iso-butane	0.6 ± 0.1		0.4 ± 0.1
N-butane	0.1		0.1
Hydrogen sulfide	20		20
Carbonyl sulfide	10		10
Sulfur dioxide	10		10
Unknown compounds	20		20

a. Not subjected to detailed analysis.

b. Includes Co.

hydrogen and carbon dioxide while depleted in oxygen as compared to air. The samples also exhibit slightly higher concentrations of nitrogen and carbon monoxide than is expected in air. Several hydrocarbons were observed in small quantities, the most abundant concentration being 500-ppm methane. The 500-ml sample was analyzed using only mass spectrometry and not subjected to the same detailed analysis as the primary sample.

It was speculated that a gas layering effect might have resulted in a concentration of a heavy combustible gas near the bottom of the liner. A gas sample was drawn from the bottom of the effluent tube on June 2, 1981. The results of the analysis (designated as heavy fraction) are shown in Table 2. The composition appears to be very close to that of air, with no indication of a large concentration of any heavy gases. However, the effluent plug had been removed for ~7 h before an attempt was made to draw a heavy gas sample. In addition, attempts had been made during this 7-h period to obtain a liquid sample. Thus, there may have been considerable mixing of any gas fractions originally in the effluent tube with the cell air.

GAS GENERATION RATES

In order to better define the gas environment and the rate of evolution or depletion of gases, a gas generation rate experiment (limited to the gases listed in Table 3) was designed. The liner vent plug was replaced with a plug fitted with a sampling valve and a pressure gauge. All other penetration plugs were left in place. The remains of the manway cover gasket were liberally coated with silicone rubber cement, and the cover secured in place with the liner clamping ring. Immediately following the sealing, a baseline gas sample was drawn to provide a basis for the evaluation of subsequent samples. A total of five gas samples were drawn during the reporting period. All the samples were subjected to mass spectroscopic analysis, with the last three samples also being analyzed on a gas chromatograph to determine the carbon monoxide content. The results of the analyses are shown in Table 3.

TABLE 3. PF-16 GAS GENERATION RATES
(volume %)

Component	Days after Closure						Uncertainty
	Baseline	4	11	17	21	25	
N ₂	78. ^a	79. ^a	79.6	80.4	80.8	80.9	±0.2
H ₂	<0.1	<0.1	0.3	0.3	0.3	0.4	±0.08
O ₂	20.9	19.8	18.6	17.6	17.2	16.7	±0.2
CO ₂	0.08	0.25	0.43	0.62	0.76	0.84	±0.05
Ar	0.97	0.96	1	1.02	1.01	1.03	±0.03
CO			0.07	0.08	0.12	0.12	±0.02

a. Includes Co.

It is clear from inspection of the data that the percent oxygen content of the gas is decreasing while all the other components appear to be increasing in concentration. It should be noted that the argon/nitrogen ratio is remaining constant within the limits of the experimental

uncertainty. This indicates that the nitrogen is being neither generated nor consumed, though this does not preclude the possibility of a leak in the liner.

The decision was made, in consultation with EG&G Idaho and GPU Nuclear personnel, to postpone a leak check of the liner until the initial gas data were obtained. This decision was due to the extreme interest in the data and due to the change in gas composition observed in the sample drawn four days after the closure of the liner. It was felt that this change in composition was an indication that the liner was well sealed. The original pressure gauge could indicate only positive relative pressures to atmosphere. During the course of the experiment, no pressure change was observed.

As the oxygen depletion indicated that there might be a negative relative pressure in the liner, a pressure transducer capable of measuring ± 15 psig was installed on the atmosphere side of the sampling valve so that the internal gas composition and pressure would not be disturbed. The pressure check indicated there was no pressure differential between the liner interior and the atmosphere to within 0.1 psi. Following the pressure check, the liner was to be leak tested using a pressure decay method with helium as the pressurizing gas. The general approach of this method is to pressurize the liner to a given pressure and observe the pressure decay for a period of time. Once the liner volume is determined, the leak rate can be calculated from the decay rate. When Liner PF-16 was tested, it was found that it had a sufficiently high leak rate so that it would not hold pressure once the gas supply was removed. It was estimated that the leak rate was $> 1 \text{ ft}^3/\text{min}$ at STP. It is possible, however, that this leak did not occur until the liner was pressurized.

Although, due to the high leak rate, the gas data shown in Table 3 cannot be used to determine any quantitative gas generation information, it clearly demonstrates that there are processes occurring within the liner that are causing a depletion of oxygen and a generation of hydrogen. If one considers that a leak may have been present, the changing gas concentration is somewhat surprising and indicates the processes, particularly oxygen depletion, are quite rapid. It is also interesting to note that the liner had been exposed to an essentially infinite supply of oxygen for approximately six months prior to closure and the oxygen depleting processes had not exhausted themselves.

VISUAL EXAMINATION

Visual examinations were performed on the liner's interior and 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. 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, though painted over, 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. The plug that was removed and replaced at TMI could easily be identified. In addition to the three plugs, the top also contained the conductivity probe. All of the operations involving exterior visual examination were videotaped with verbal comments by the staff performing the examination.

The liner internal examination was initially performed manually using a fiber optics viewer. Apparently, the fiber bundle was damaged by the high radiation field and further examination was no longer possible. The interior of the liner was examined using an in-cell TV camera. The manway cover was removed and the camera was positioned inside the liner and panned to view the entire inner surface. All of the internal examination was videotaped for permanent record.

The underside of the manway cover was heavily rusted, with black deposits on the surface. This surface had not been sealed with a protective coating as had the rest of the liner interior. The under surface of the cover was wet when the liner was first opened, apparently as a result of

condensation of water vapor from within the liner. It would be expected that the condensation from liquid splashing onto the cover during operation of the EPICOR II system would lead to some corrosion of the uncoated surface.

Figure 6 shows the underside of the manway cover. The corrosion of the cover may be clearly seen. Although it is not appropriate to draw any definite conclusions concerning the condition of the cover, several observations were made:

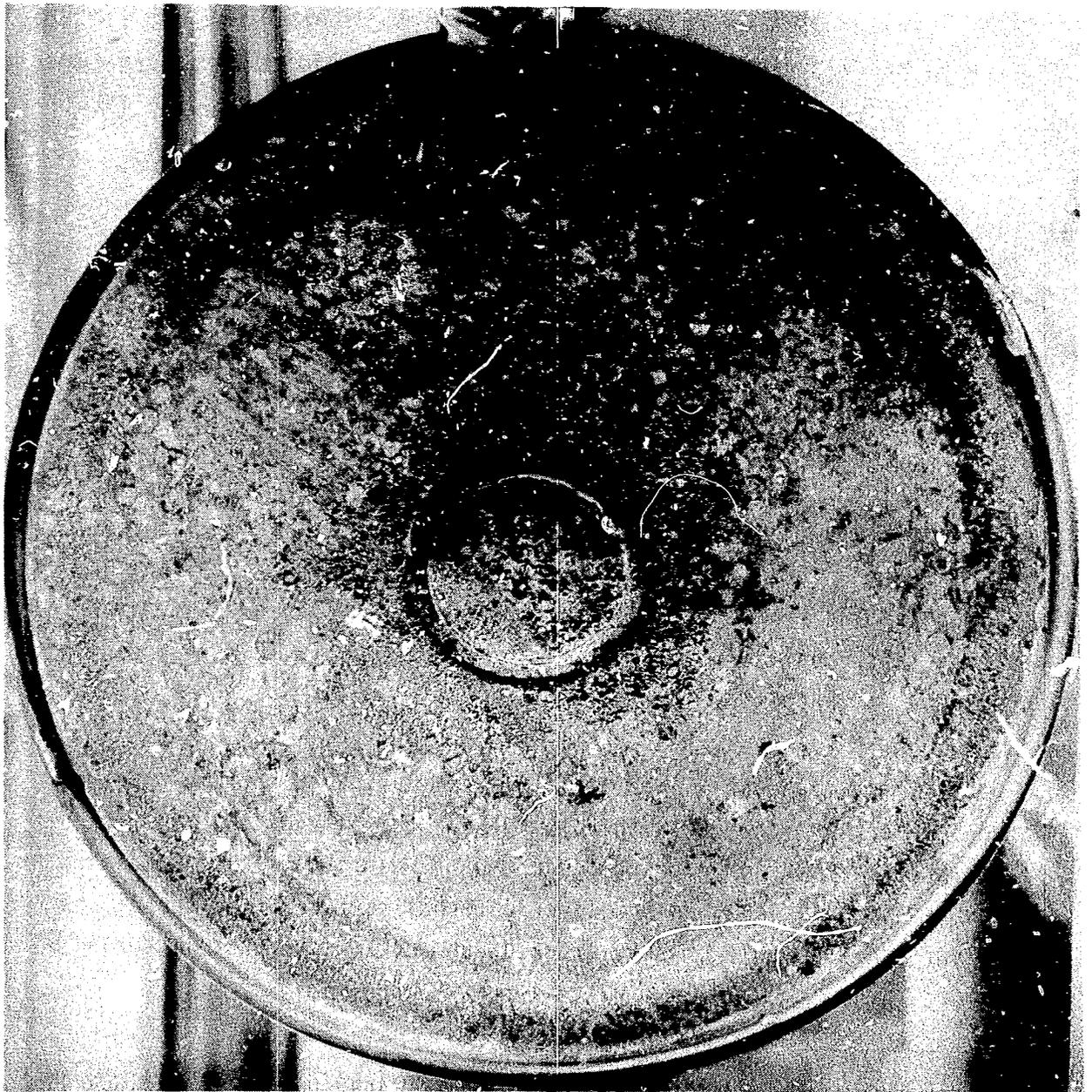


Figure 6. Corroded underside of manway cover.

- The corrosion deposits on the cover are reddish-brown, indicating the presence of oxygen, leading to FeO_2
- General corrosion is present
- The pitting does not appear severe, but there is clearly some pitting present
- The amount of pitting cannot be determined without chemical descaling
- Metallographic examination of the pits would be necessary in order to determine the pit depth and, consequently, the likelihood that the cover integrity has been damaged
- Round areas are observed, perhaps indicating that H_2O had condensed and remained on the cover
- Barnacle-like deposits have built upon the bung closure and surrounding area.

Some of the protective coating on the manway cover lip appeared to have peeled off and rust spots (1/4 to 1/2 in. in diameter) were observed in this area. Figure 7 shows the appearance of the area where the coating had peeled off. The protective coating on the inner surface of the liner appeared to be blistered; however, it seemed intact, with only occasional rust spots (\sim 1/8 in. in diameter).

One small area (\sim 4 x 4 in.) of the liner interior surface appeared to be uncoated and covered with rust.^a The underside of the liner top plate appeared to be in good condition, with relatively less blistering of the protective coating than the liner internal surface. The top of the resin surface was dark, crusty, cracked, and caked with white material

a. This area was purposely scraped, prior to liner use in the EPICOR II system, to provide a better ground for the conductivity probe.

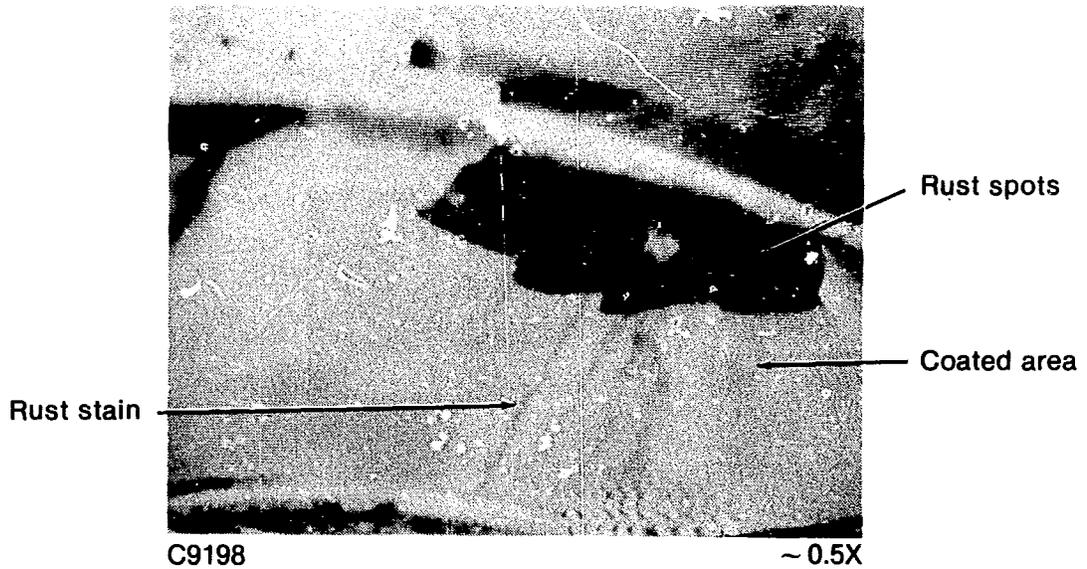


Figure 7. Corroded area on manway cover lip where protective coating was peeled off.

believed to be boric acid deposits (see Figures 8 and 9). The portions of the influent and effluent pipes and other braces that could be seen showed no visible evidence of corrosion. The areas around the welds joining the top plate to the shell, and the shell axial weld, showed no visible corrosion.

A small stainless steel spade was used to remove a portion of the ion-exchange media in contact with the liner so that the liner ion-exchange media interface area could be examined. No visible corrosion of the liner surface was evident. Figure 10 shows the area of the liner that was in contact with the ion-exchange media.

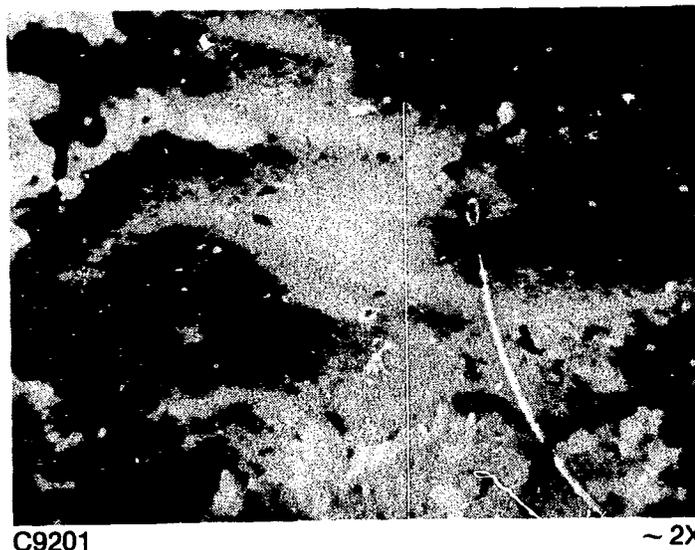


Figure 8. Appearance of the top of the resin bed.

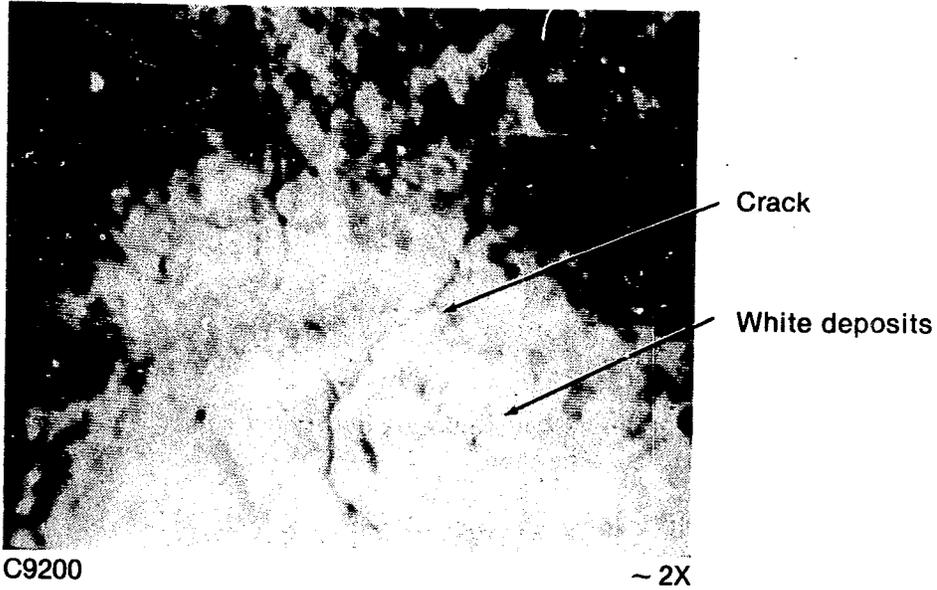


Figure 9. Top of the resin bed showing white patchy deposits.

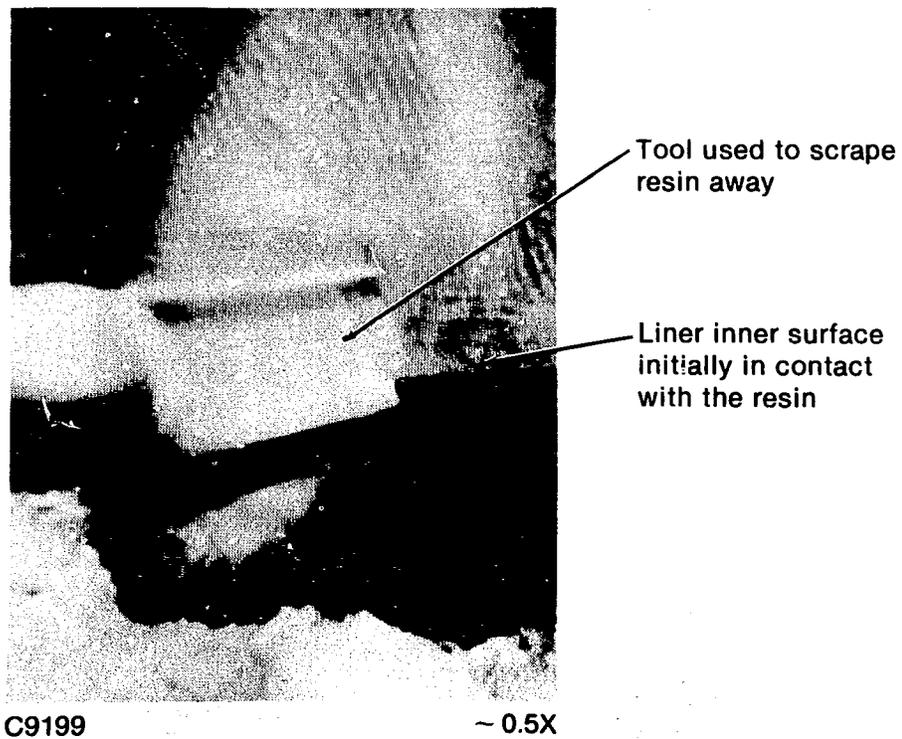


Figure 10. Appearance of the liner interior in contact with the resin.

ION-EXCHANGE MEDIA SAMPLING AND EXAMINATION

An attempt to remove a core sample of ion-exchange media was made using a technique based on an ASTM procedure (D2687-77), but due to the consistency of the media bed, a core sample could not be obtained. A new coring device was constructed that consisted of a 2-in. ID Lucite tube with a plastic basket-type retainer and a stainless steel cutting tip. The coring device was inserted into the media bed as close to the centerline as was practical. A 2.5-in. ID Lucite caisson tube was inserted to encase the sample tube so that when the sample was withdrawn the media bed would not collapse. The caisson tube also allowed insertion of a probe to determine the internal dose rate profile.

The redesigned coring device performed satisfactorily when tested in a wet, compacted sand pile; however, it was found that the behavior of the coring device in the ion-exchange media itself was quite unlike that in the sand, and a complete core could not be obtained. The cutting tip separated from the Lucite tube as a consequence of the force needed to seat the sampling tube and the core hole caisson tube. The basket retainer and the bottom 3 in. of the core sample were lost. It is suspected that one of the reasons for loss of the bottom of the core sample is that there is an accumulation of liquid at the bottom of the liner, resulting in a liquid/resin mix having a soupy consistency. Such a mix could not be retained without a positive closure at the bottom of the sampling tube.

Using the hot cell stereomicroscope at $\sim 30\times$ magnification, a preliminary visual examination of the core sample was made with ion-exchange media remaining encased within the sampling tube. Some general observations may be made concerning the media bed. The total length of the actual core sample is ~ 16 in. The total depth of the bed is ~ 31 in., and the shorter core sample may be an indication of compaction during sampling, or failure to collect a complete core sample. Approximately 3 in. of media were clearly lost, but this does not account for all the length difference from the full 31 in. of penetration.

Three well defined regions were observed in the core. The top region (Region I) appeared to consist of light-colored, free-flowing, dry, granular, irregular shaped particles. This had a length of ~ 5 in. A photomicrograph of this material is shown in Figure 11.

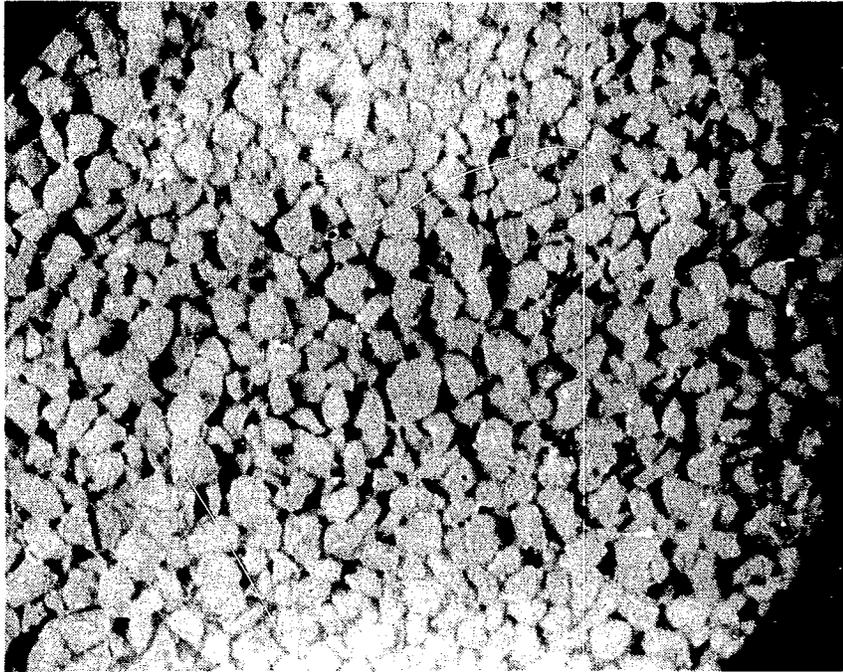


Figure 11. Photomicrograph of core sample, Region I.

Region II was ~ 8 in. long and consisted primarily of regular-shaped, spherical particles that appeared translucent. A few broken particles were observed. There was some observed mixing with the top layer, and the moisture content increased with increasing depth. However, no real difference in color or shade was observed. Near the bottom of this region, there appeared to be some mixing, with occasional opaque, spherical particles. Figure 12 is a photomicrograph of this region.

Region III appeared extremely moist and consisted of both opaque and translucent particles, with the opaque particles constituting approximately half of the particles. There was some appearance of agglomeration, but that may be an artifact resulting from the high moisture and the particles adhering to the side of the tube. The length of this region was 3 in., but apparently some of the core bottom had been lost during sampling. A contact pH of Region III material was made using pH paper, which exhibited an approximate pH of 2. The photomicrograph of this region is shown in Figure 13.

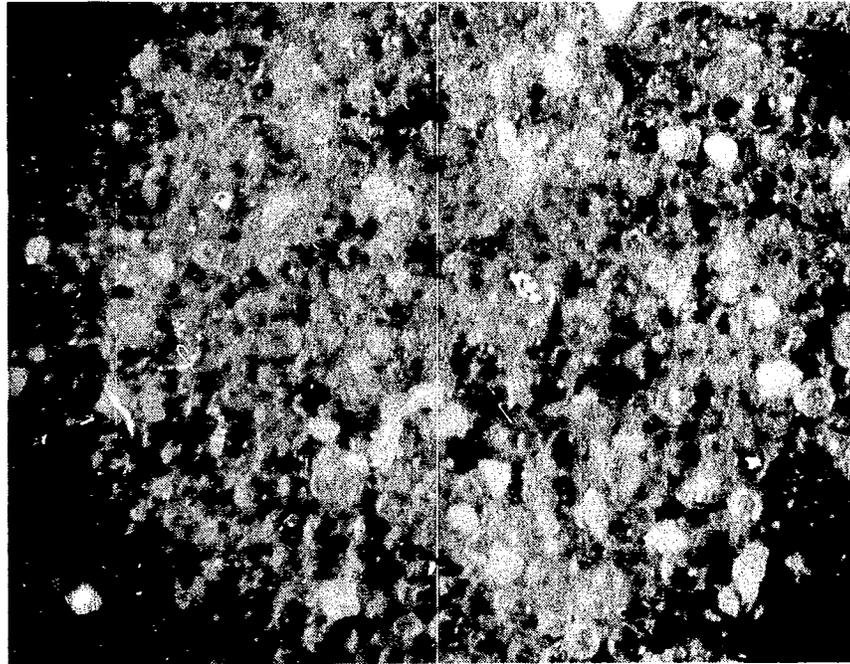


Figure 12. Photomicrograph of core sample, Region II.

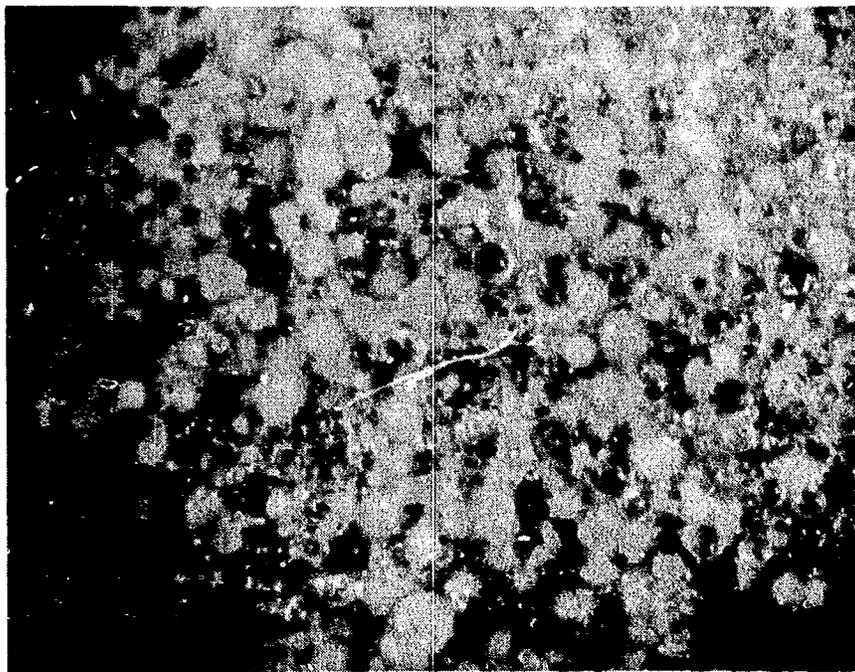


Figure 13. Photomicrograph of core sample, Region III.

The general observation made on the basis of examinations of the bulk material is that the ion-exchange media do not exhibit any evidence of significant degradation.

The material from the core sample was removed from the sampling tube to allow chemical analyses and detailed microscopic examination. Each of the two distinct organic layers was removed from the tube in two separate segments--bottom and top. These correspond to Regions II and III of the core. Region I was removed as one segment, as it was dry and free-flowing and believed to be thoroughly mixed by previous handling of the core sample. Some media samples were removed from each of the segments for microscopic examination. Larger subsamples were removed for radiochemical and chemical characterizations.

MICROSCOPIC EXAMINATION OF ION-EXCHANGE MEDIA

Samples of ion-exchange media from Liner PF-16 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 five segments of core and selected for examination; one segment from Region I, two from Region II, two from Region III. The particles were then mounted on a graphite cylinder using a graphite paste. The mounts were then coated with a thin film of carbon to provide an electrically conductive surface. 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.

Region III, Bottom Segment

Figure 14 shows the typical appearance of the ion-exchange particles from this segment. Most of the particles appear spherical. Occasionally, cracking and spalling of the surface layer was observed. Figures 15 and 16 show examples of surface defects. EDAX analysis of the particle shown in Figure 16 indicated sulphur as the only detectable element [elements of atomic numbers below sodium (11), including carbon and hydrogen, are not detectable with the EDAX system]. Figure 17 shows the X-ray spectrum obtained on this particle.

Region III, Top Segment

Particles in this segment were all spherical, with little or no evidence of surface defects. Figure 18 shows the appearance of the particle magnified 20X. Figure 19 shows one of the particles at a higher magnification, 130X. EDAX analysis showed predominantly sulphur, with very small amounts of calcium and iron.

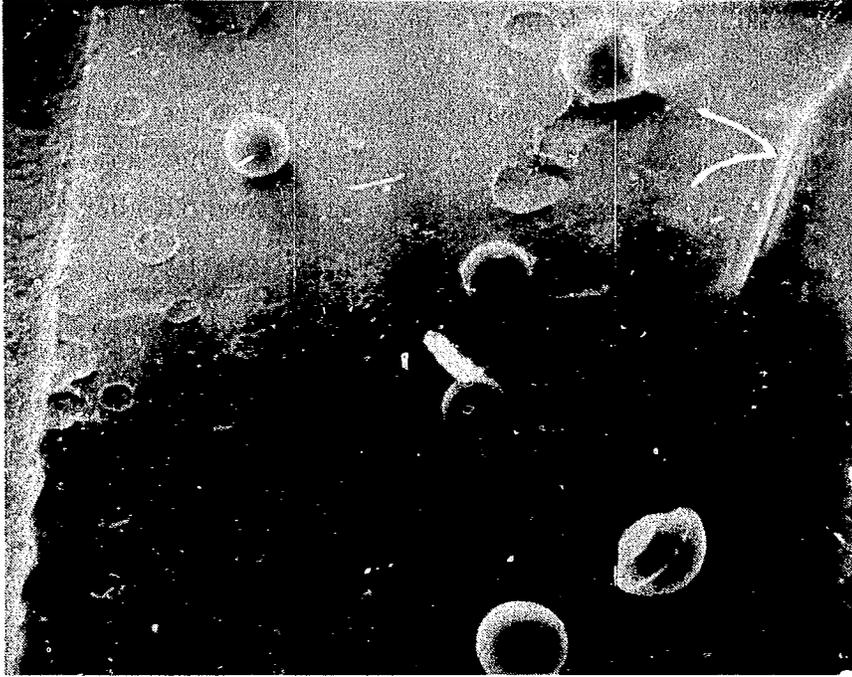


Figure 14. SEM photograph of ion-exchange media, Region III, bottom segment, 20X.

xx

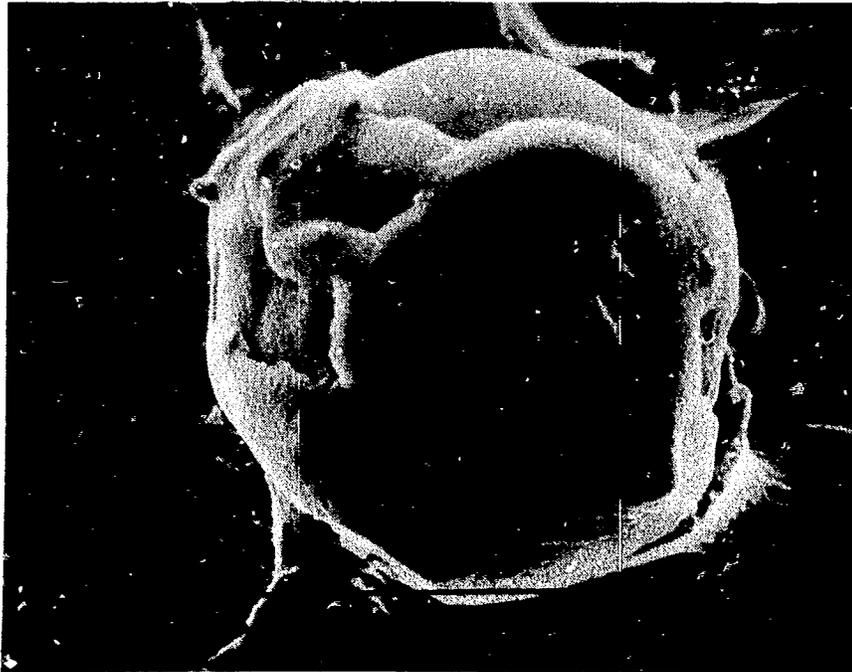


Figure 15. SEM photograph detailing surface defects in media particle, Region III, bottom segment, 130X.

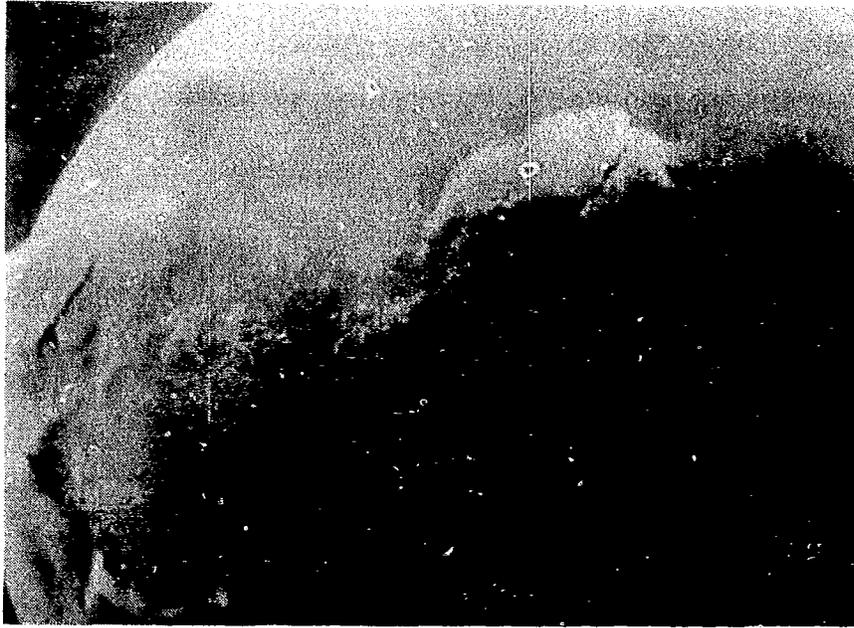


Figure 16. SEM photograph showing surface defects of media particle, Region III, bottom segment, 220X.

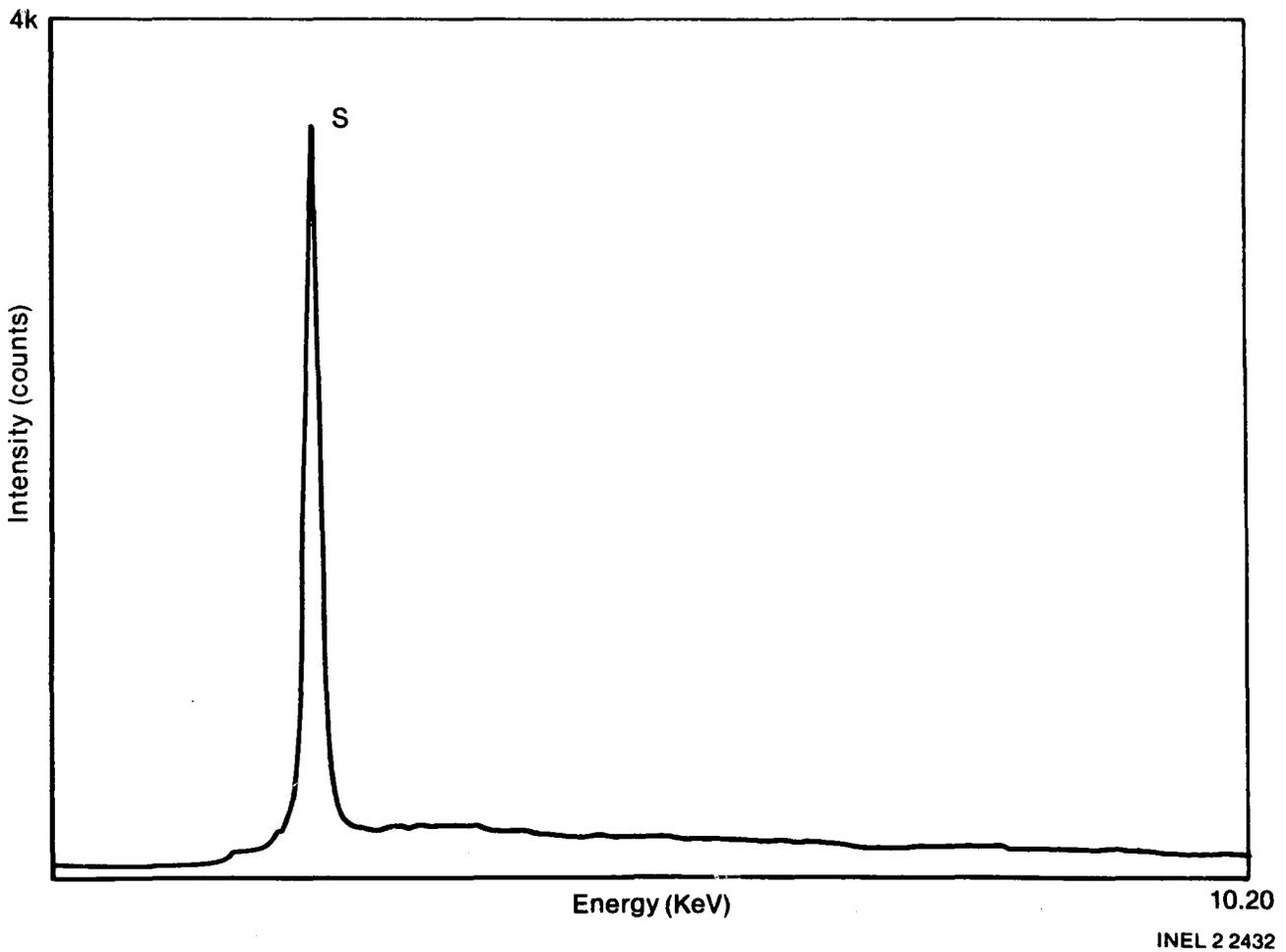


Figure 17. EDAX scan of media particle from Region III, bottom segment.

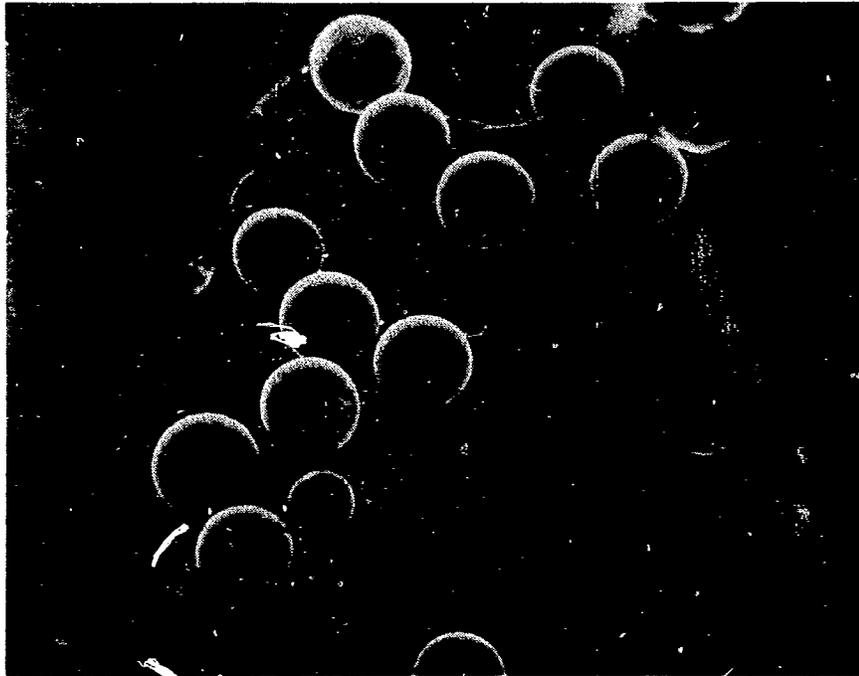


Figure 18. SEM photograph of ion-exchange media particles, Region III, top segment, 20X.

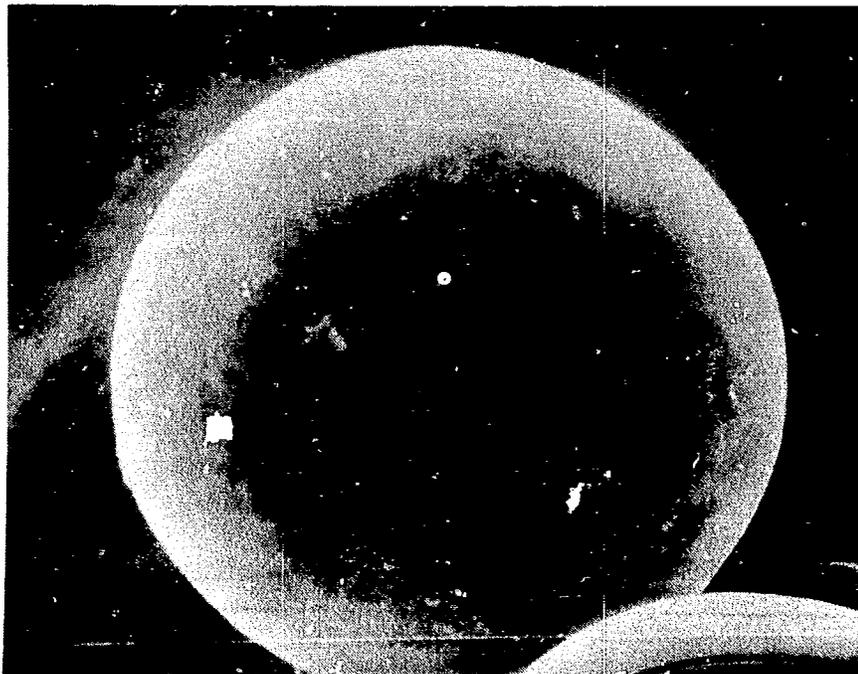


Figure 19. SEM photograph of media particle, Region III, top segment 130X.

Region II, Bottom Segment

The particles in this segment were also spherical and, again, no surface defects were evident. Figures 20 and 21 show the typical appearance of the particles. X-ray analysis of one of the particles showed a relatively smaller amount of sulphur. A small amount of potassium was observed in addition to calcium and iron. Figure 22 shows the X-ray spectrum obtained on this particle.

Region II, Top Segment

The particles in this segment were acicular with rough "rocklike" surfaces. Figure 23 shows the appearance of the particle at 20X. A higher magnification view (130X) is shown in Figure 24. The surfaces, when examined at higher magnification, showed the presence of crusty deposits. Analysis of the deposits showed high concentrations of calcium and iron. Figure 25 shows the X-ray spectrum obtained. Elements of mass numbers below potassium could not be detected because of the radiation level of the particles.

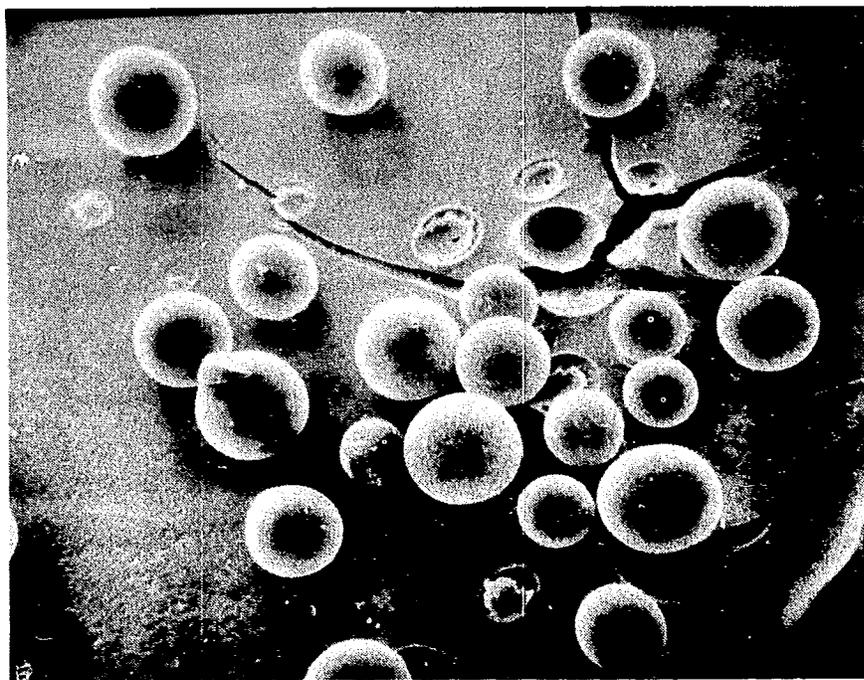


Figure 20. SEM photograph of ion-exchange media, Region II, bottom segment, 20X.

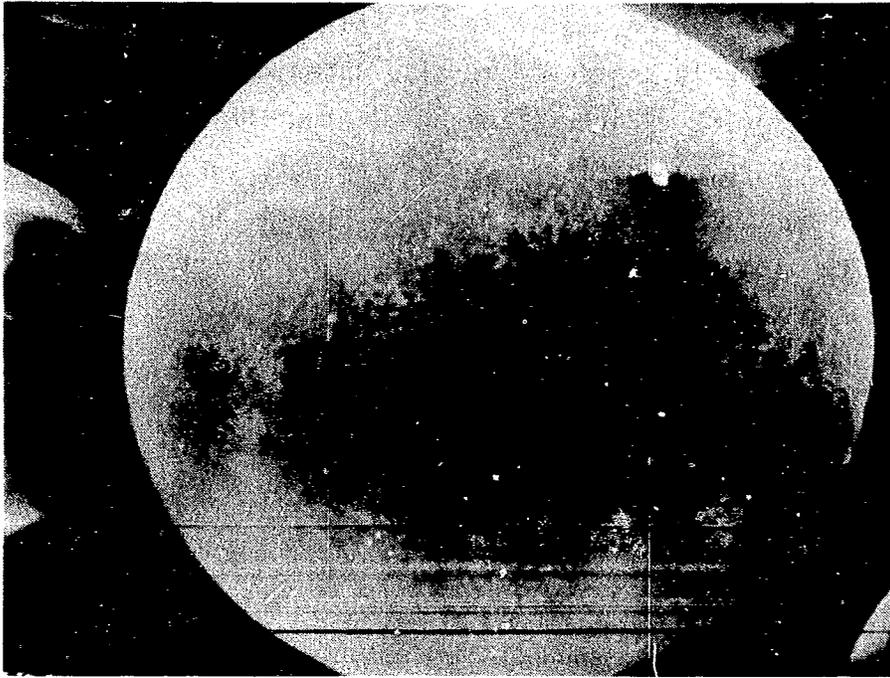


Figure 21. SEM photograph of media particle, Region II, bottom segment 130X.

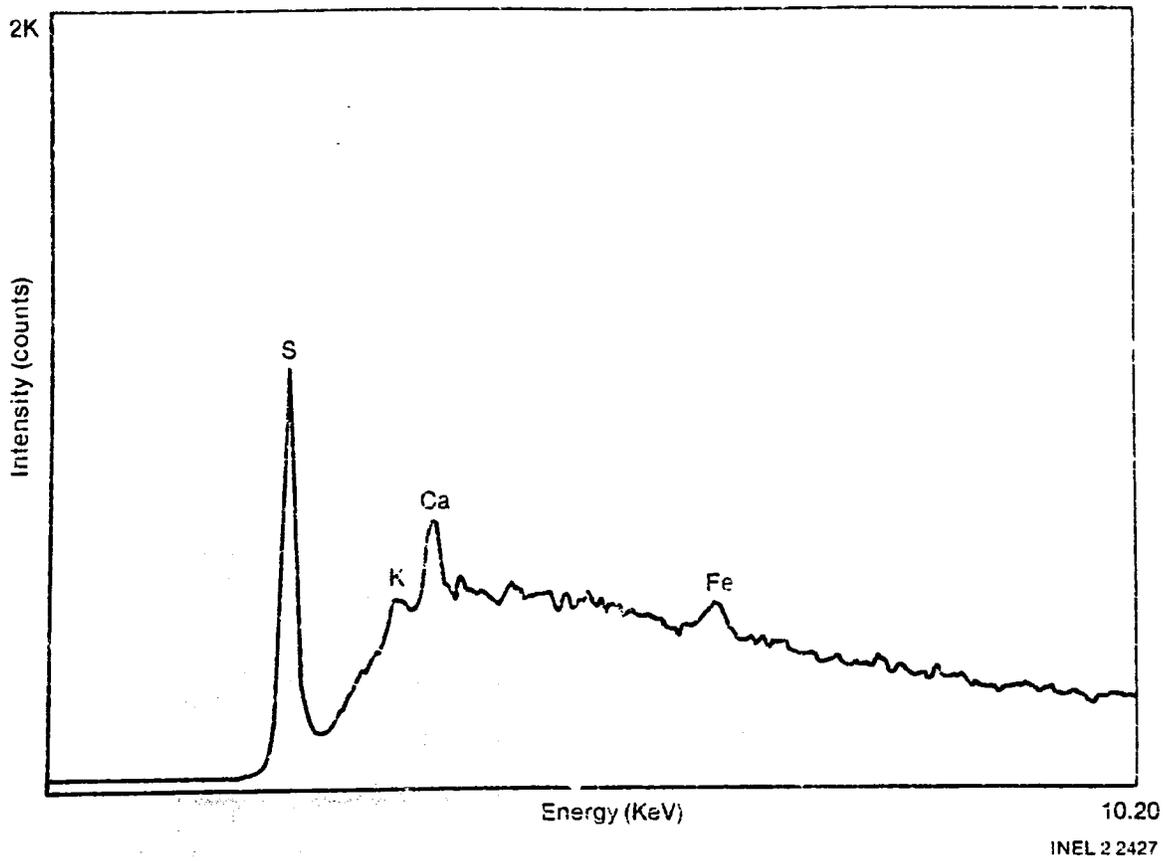


Figure 22. EDAX scan of media particle from Region II, bottom segment.

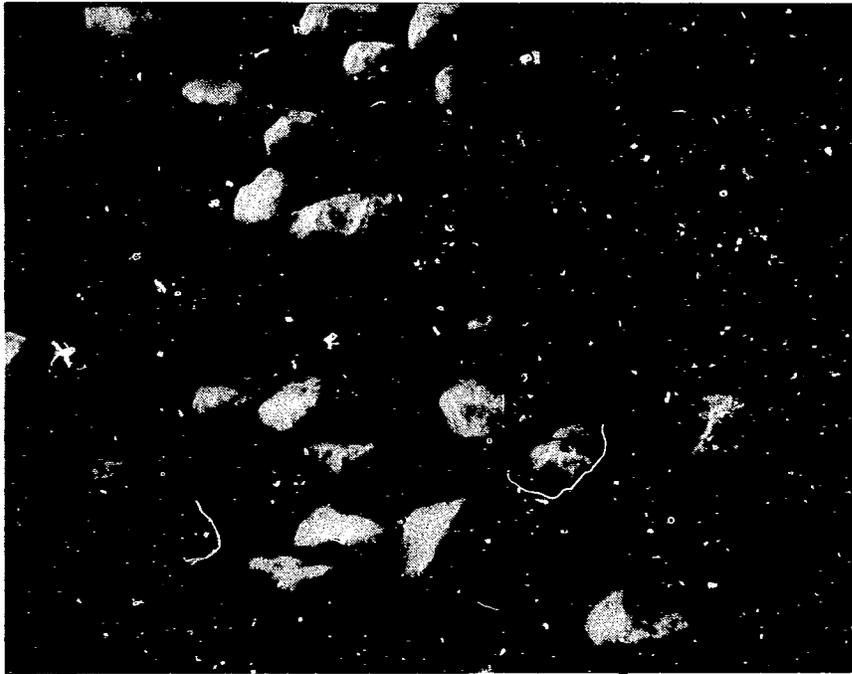


Figure 23. SEM photograph of media particles, Region II, top segment 20X.

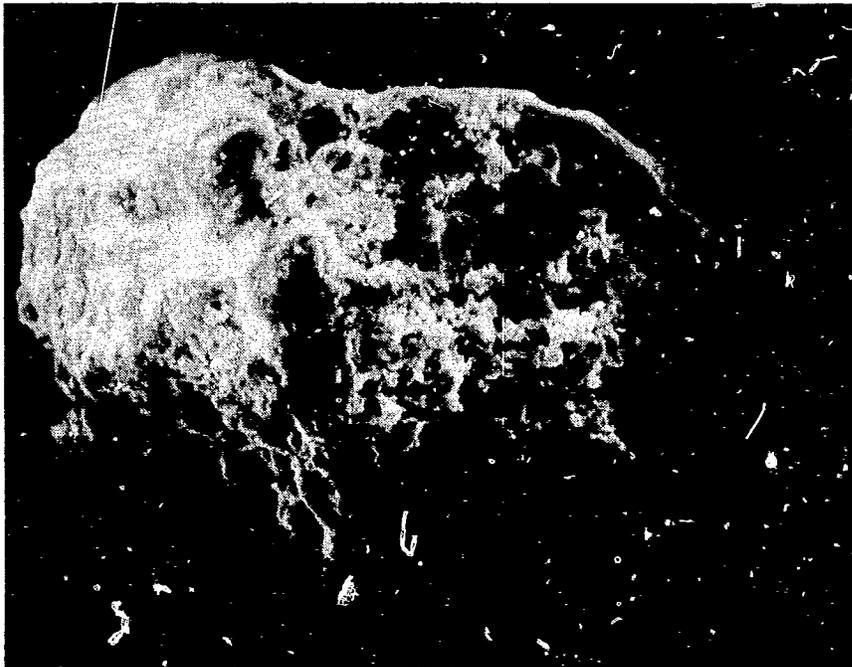


Figure 24. SEM photograph of media particle, Region II, top segment 130X.

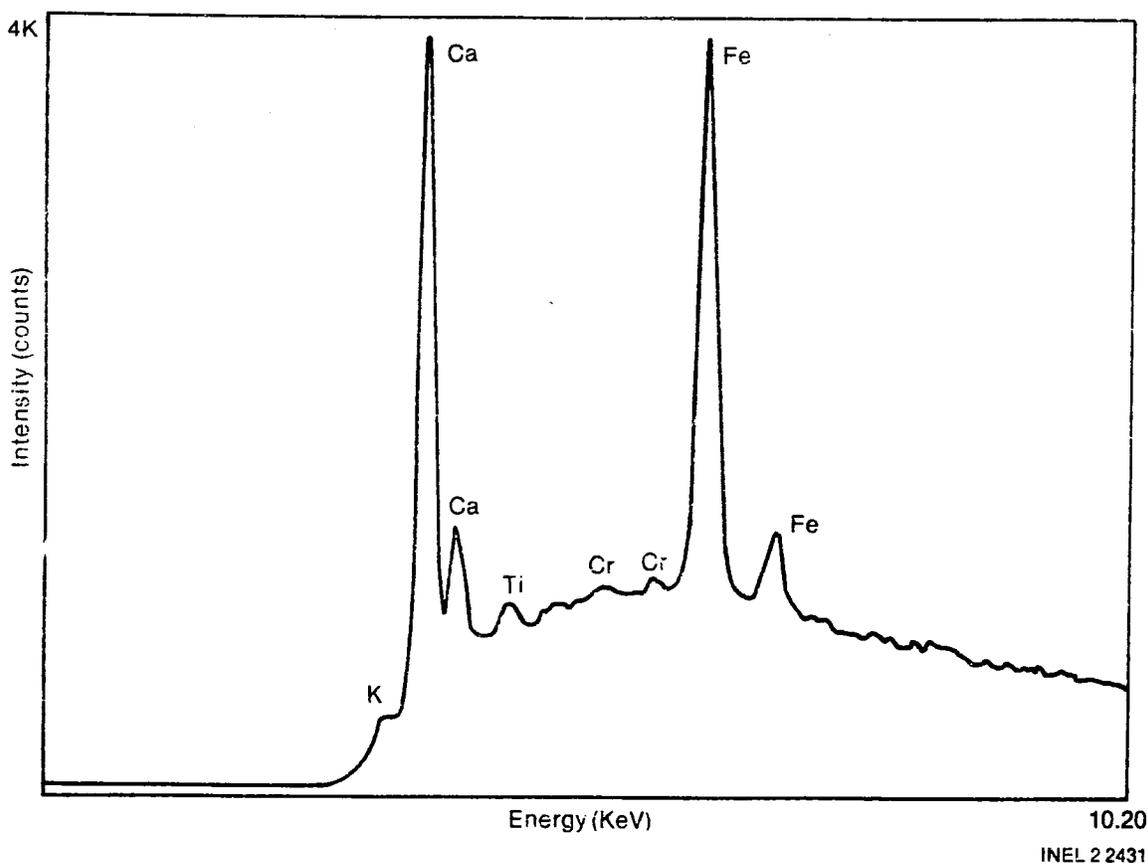


Figure 25. EDAX scan of media particle from Region II, top segment.

Region I

The particles in this segment were very similar to those in the previous segment. Figure 26 shows the appearance of the particle. Figure 27 shows the presence of deposits on the surface of the particle. X-ray analysis of the surface showed relatively less calcium and more iron. Small amounts of titanium were observed, as well as potassium as seen in the particles from Region II, bottom segment. None of the particles appeared to have been affected significantly by radiation.

In summary, deterioration of the ion-exchange media due to radiation exposure appears to be minimal. Surface cracking and spalling were observed only in one segment, the bottom layer of the core sample. As this is the region the farthest from the highest activity loading, it is likely that the observed degradation is an effect of the high moisture content of the region or chemical attack.

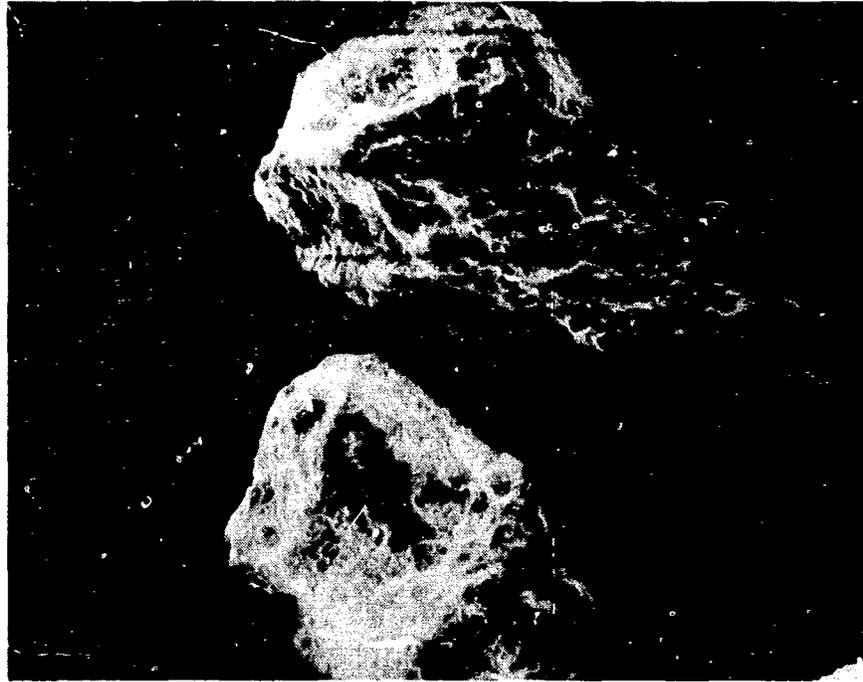


Figure 26. SEM photograph of media, Region I, 65X.

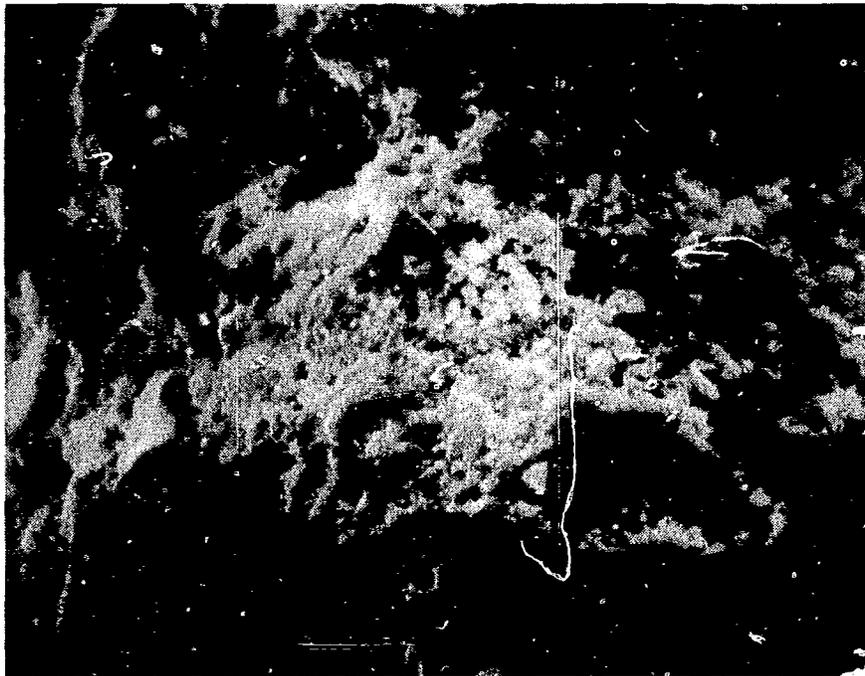


Figure 27. SEM photograph of media, particle, Region I, 455X.

RESIDUAL LIQUID SAMPLING AND ANALYSES

Upon removal of the liner manway cover, some droplets of liquid were observed on the cover's interior surface. The total volume of liquid present was estimated to be no greater than 10 ml and was presumed to be condensation from vapors inside the liner. As the presence of the liquid was not anticipated, no provisions had been made to allow its collection before evaporation. A strip of pH paper was inserted into the cell to provide a crude determination of the liquid pH. Although an accurate determination was not possible, the liquid appeared to have a pH of between 2 and 3.

The presence of liquid droplets with a pH of 2 to 3 on the manway cover raised questions concerning the nature of the liquid and the vapor/gas environment within the liner. It was not clear whether the low pH liquid was the result of condensation of a low pH vapor or condensation of water vapor with a dissolution of deposits or corrosion products from the surface of the manway cover. To resolve these questions, a stainless steel witness plate was installed in the liner to collect condensation. Several milliliters of condensate were collected in this manner. The measured pH of this liquid was 5.3. On this basis, it was concluded that the low pH observed earlier was due to the condensed liquid dissolving either corrosion products or materials splashed onto the cover during operation of the liner or its handling.

Following gas sampling, the liner effluent plug was removed to allow an examination for the presence of liquid. The plug could not be removed using a T-bar wrench, but was removed using a 1000 ft-lb impact wrench with no damage to the plug threads or the liner. An attempt was made to sample for liquid in accordance with the applicable BCL procedure. A stiff metal rod with strips of pH paper attached was inserted into the effluent tube to detect the level and approximate pH of any residual liquid. Due to bends in the tube, it was not possible to insert the rod more than about 2-1/2 ft. In order to negotiate the tube bends, a length of polypropylene tubing was inserted into the tube and connected to an evacuated collection flask. A length of ~5-1/2 ft of tubing was inserted with relative ease before an obvious barrier was encountered. The valve to the evacuated collection

flask was opened but no sample obtained. With the tubing remaining in place, the valve between the collection flask and the vacuum pump was opened for several minutes to allow any liquids to be collected through aspiration. Again, no liquid was obtained. The tubing was withdrawn and examined for the presence of moisture. No moisture was detected. It was then concluded that insufficient free liquid was available in the effluent manifold to be detected using this technique.

After the first core sample was taken, a liquid sample was obtained from the bottom of the caissoned hole. 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 ~500 ml of liquid was collected. The liquid was obtained with relative ease and it is suspected that several liters of liquid may remain at the bottom of the liner.

The liquid sample was clear and colorless, with a small quantity of solid material present. 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 indicator 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, $^{89,90}\text{Sr}$, Pu and U by alpha spectroscopy. The results of these analyses are presented in Tables 4 and 5.

Inspection of the data indicates that the water has low ion content. The 30 $\mu\text{mho/cm}$ 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

TABLE 4. RESIDUAL LIQUID CHEMISTRY ANALYSIS

pH	5.3 ± 0.1 at 27°C
Conductivity	30 µmho/cm at 27°C
Acidity	1.2 meq/ml at pH 7.0
Total residue upon evaporation	3.1 ± 0.1 mg/ml
	<u>(ppb)</u>
Sodium	<2000
Iron	34
Phosphorus	<110
Zinc	88
Magnesium	<20
Calcium	100
Aluminum	110
Boron	1.12 E+6
	<u>(µg/ml)</u>
NH ₄	0.8
SO ₄	5.2
NO ₃	<0.3
Chlorine	3
Total organic carbon	61
Total kjeldahl nitrogen (TKN)	0.48

TABLE 5. RESIDUAL LIQUID RADIOCHEMISTRY ANALYSIS
(µCi/ml)

Gross beta/gamma	1.77 ± 0.01E-2
Gross alpha	5.9 ± 0.01E-4
89,90Sr	5.2 ± 0.1E-4
125Sb	7.94 ± 0.42E-4
134Cs	1.32 ± 0.02E-3
137Cs	1.308 ± 0.005E-2
238,238,240Pu	<1.0 E-4
238U	<1.0 E-4

products. The only significant chemical species in the liquid was the 1.12×10^6 ppb of boron which is not effectively removed by the ion-exchange media.

Initially, there had been some concern that the residual liquid might present a corrosive environment at the bottom of the liner, as the pH of the liquids collected during final processing of PF-16 was found to be 2.79.

The observed residual liquid pH of 5.3 (weak acid) indicates that the materials in the liner provide a buffering action for the liquid. This weak acid would not be expected to present a corrosion hazard to the liner steel.

It is clear from a direct comparison of the influent liquid analysis and the residual liquid analysis that there is no significant release of the radionuclides from the resin matrix into the residual liquid. The residual liquid has three orders of magnitude less activity than the influent. This compares well with the decontamination factors expected for an ion exchanger that has processed 3×10^4 gal of liquid. When it is further considered that the liner currently contains ~1250 Ci of activity, it is apparent that no significant release of radionuclides has occurred.

DETERMINATION OF MEDIA pH

Media samples were obtained from each of the distinct layers of media in the core sample. A 2-g specimen of each sample was removed and mixed with 10 ml of deionized water (pH = 6.40). The pH of each solution was determined using a digital pH meter with a combination probe calibrated against certified buffer solutions. The measurements were conducted in duplicate for each of the three observable layers. The observed pH values are shown in Table 6.

The total absorbed dose for the PF-16 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-16 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 initial 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.

TABLE 6. EPICOR II LINER PF-16 MEDIA pH VALUES

<u>Layer</u>	<u>Media</u>	<u>Sample</u>	<u>pH</u>
Bottom	Organic	1	2.25 ± 0.05
		2	2.22 ± 0.05
Middle	Organic	1	3.05 ± 0.05
		2	3.16 ± 0.05
Top	Inorganic	1	8.20 ± 0.05
		2	8.22 ± 0.05

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 radiation damage and which of the two effects predominates. Experiments conducted at the Department of Energy's Rocky Flats plant^{3,4} indicated an increase in the water content of both cation and anion resins that were irradiated in seven normal nitric acid (7N HNO₃).

Approximately 10-g samples were drawn from each of the three distinct layers of ion exchange media observed in the 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 h. 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 7. 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-16 are considered proprietary, these values are not available.

TABLE 7. ION-EXCHANGE MEDIA WATER CONTENT

<u>Core Region</u>	<u>Media</u>	<u>Water Content^a (%)</u>
Bottom	Organic	15.0
		14.7
Middle	Organic	9.3
		9.2
Top	Inorganic	6.7
		7.8

a. Percent water content = $\frac{\text{grams air dried} - \text{grams oven dried}}{\text{grams air dried}}$.

EPICOR II INTERNAL DOSE RATE

After removal of the manway cover, a single core hole was made in the PF-16 liner resin bed, which utilizes a transport plastic tube to maintain an undisturbed resin bed outside the core hole. Lithium fluoride (LiF) thermoluminescent dosimeters (TLDs) were used as the gamma dose indicators. The TLDs were placed at 1-in. intervals along ~30 in. of a wooden holder. A plastic sheath was used to protect the TLD cards from contamination while in cell and allowed the cards to be read immediately upon removal from the hot cell.

The gamma profiling procedure using TLDs included an initial calibration of the reader instrument by placing several TLDs in a known gamma field for varying periods. The TLDs were then placed in the reader unit and a curve of dose versus TLD reading (in coulombs) was constructed. A single card was put into the cell to the level of the top of the resin bed and immediately brought out to determine the dose a TLD chip would be exposed to during the loading and unloading operation. This chip received ~1 rem dose.

Fifteen cards containing two chips on each card at a spacing of 1 in. were taped to a wooden holder. A plastic sheath was used to cover the holder and cards. The holder was placed in cell and into the caisson until the holder rested on the resin at the bottom of the caisson. The holder was left in the caisson for 1 h and then removed from the cell. Because this measured only the gamma, the shielding from the plastic was minimal. The TLDs were removed and counted. The results are shown in Table 8, and a plot is shown in Figure 28.

It was noted that the gamma dose decreased rather smoothly from a relatively level plateau between 33 and 23 in. to a minimum at 8 in. from the liner bottom. The dose rises again to the last reading taken at the 4 in. level. The smooth change is expected since each TLD is bombarded by radiation from all directions and the radiation field is a smooth gradient from the high levels at the top of the liner to lower levels at the bottom of the liner. The rise seen at the bottom of the liner was not expected since

TABLE 8. PF-16 INTERNAL GAMMA DOSE, FIRST MEASUREMENT

Distance From Liner Bottom (in.)	Dose (rem)
4	618
5	535
6	344
7	275
8	253
9	280
10	330
11	389
12	475
13	499
14	565
15	566
16	674
17	683
18	758
19	777
20	828
21	854
22	933
23	1067
24	1073
25	1068
26	1055
27	1018
28	1039
29	971
30	1005
31	1065
32	999
33	984

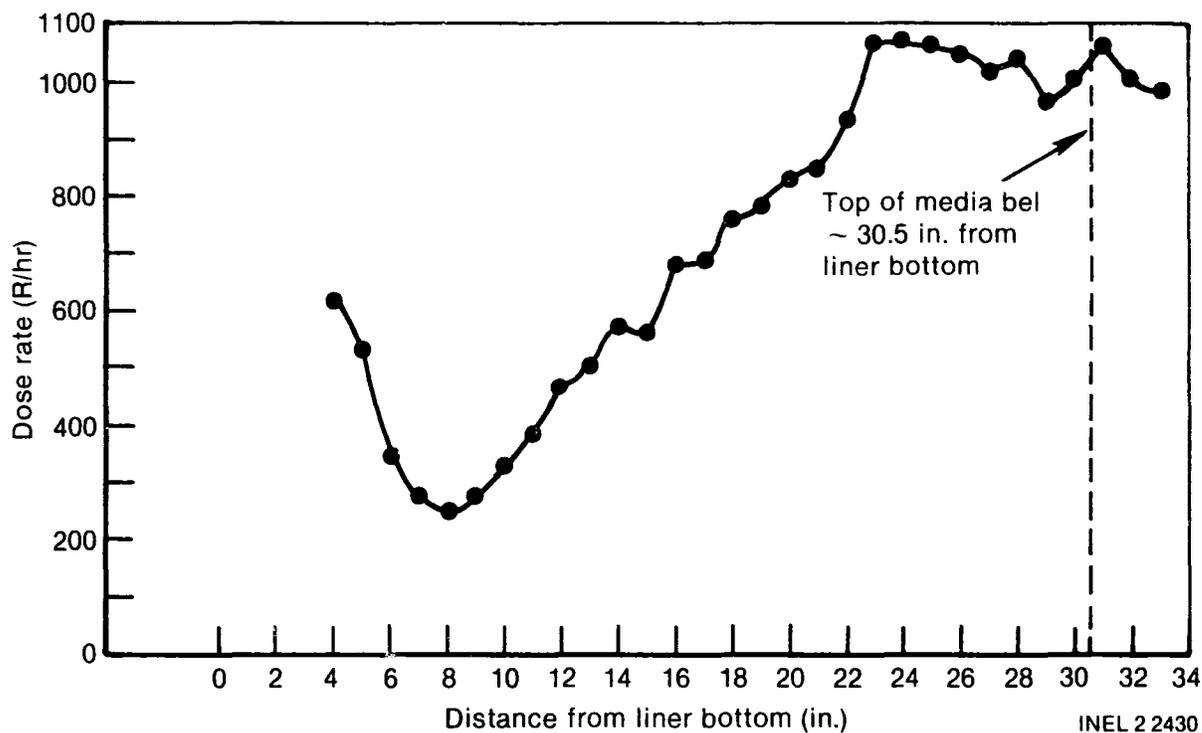


Figure 28. Internal dose rate profile, first measurement.

neither the direct probe axial profile nor the axial gamma scan taken outside the liner showed a higher activity at the bottom of the liner.

To verify the first profile, a second set of 15 TLDs were inserted into the liner caisson for 1 h. The results of this second test are given in Table 9 and plotted in Figure 29. As can be seen, both plots (see Figures 28 and 29) show a rise at the lower end of the liner. It is felt that the bottom TLD is partially imbedded in the resin at the open end of the caisson and the close proximity of the lower two TLDs (bottom 4 in.) caused these to receive a higher dose. Once the distance from the exposed resins reached about 4 in., the radiation contribution was primarily from the resins surrounding the caisson. Therefore, it is felt that the increased dose observed from the TLDs is geometric or related to the distance from the resins, and in reality the dose rate at the lower end of the resin liner is relatively constant at about 300 rem/h.

TABLE 9. PF-16 INTERNAL GAMMA DOSE, SECOND MEASUREMENT

Distance From Liner Bottom (in.)	Dose (rem)
4	556
5	439
6	327
7	270
8	288
9	280
10	315
11	374
12	451
13	471
14	574
15	579
16	697
17	757
18	733
19	757
20	830
21	880
22	974
23	1088
24	1103
25	1133
26	1098
27	1067
28	1065
29	1066
30	1040
31	1029
32	1052
33	1024

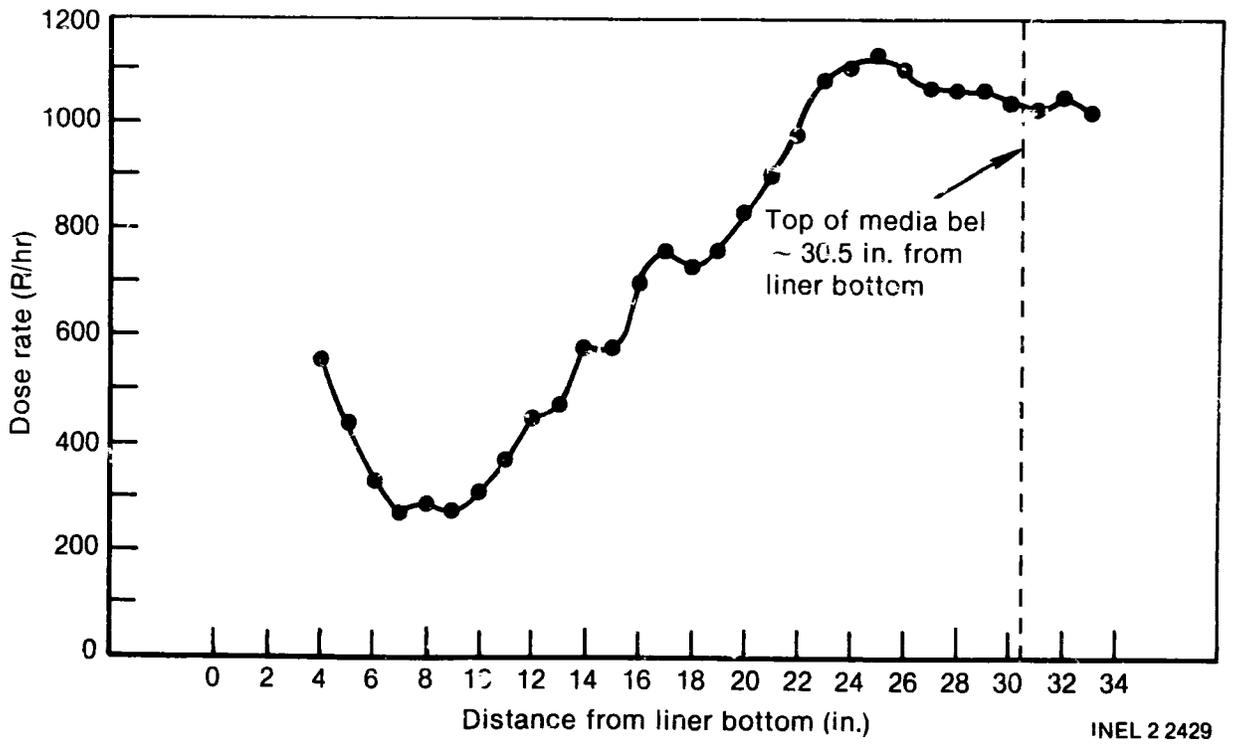


Figure 29. Internal dose rate profile, second measurement.

EXTERNAL GAMMA SCANS

External gamma scans of the liner were performed to determine the relative depositions of gamma-emitting radionuclides. First the liner was positioned in front of the in-cell slit collimator containing a horizontal Ge(Li) detector. The instrumentation was calibrated to detect ^{137}Cs gamma activity, using a mixed radioactive standard.

The liner was moved vertically in front of the slit collimator using the in-cell crane, and an axial profile of ^{137}Cs activity was obtained. This data was then used to get the gain and span, and to chart the speed of the instrumentation. With the instruments appropriately adjusted, a total of eight axial gamma activity profiles were obtained, along with azimuthal orientations. The scans for the eight orientations were identical within the uncertainty of the technique.

To obtain the axial position along the length of the liner, a gamma source was placed at the bottom of the liner during scanning. In addition, the location of the top of the ion-exchange media bed was measured with respect to the top of the liner.

Figure 30 shows a typical gamma activity profile along the length of the liner. Gamma spectroscopy performed at the location of the peak activity showed that >90% of the gamma activity is ^{137}Cs and ^{134}Cs . The gamma scans also showed that most of the activity is concentrated in the top 3 to 6 in. of the ion-exchange media bed. The peak at the bottom of the liner was caused by a gamma source attached to the outside of the liner for axial liner position reference, and is not indicative of any activity in the media bed.

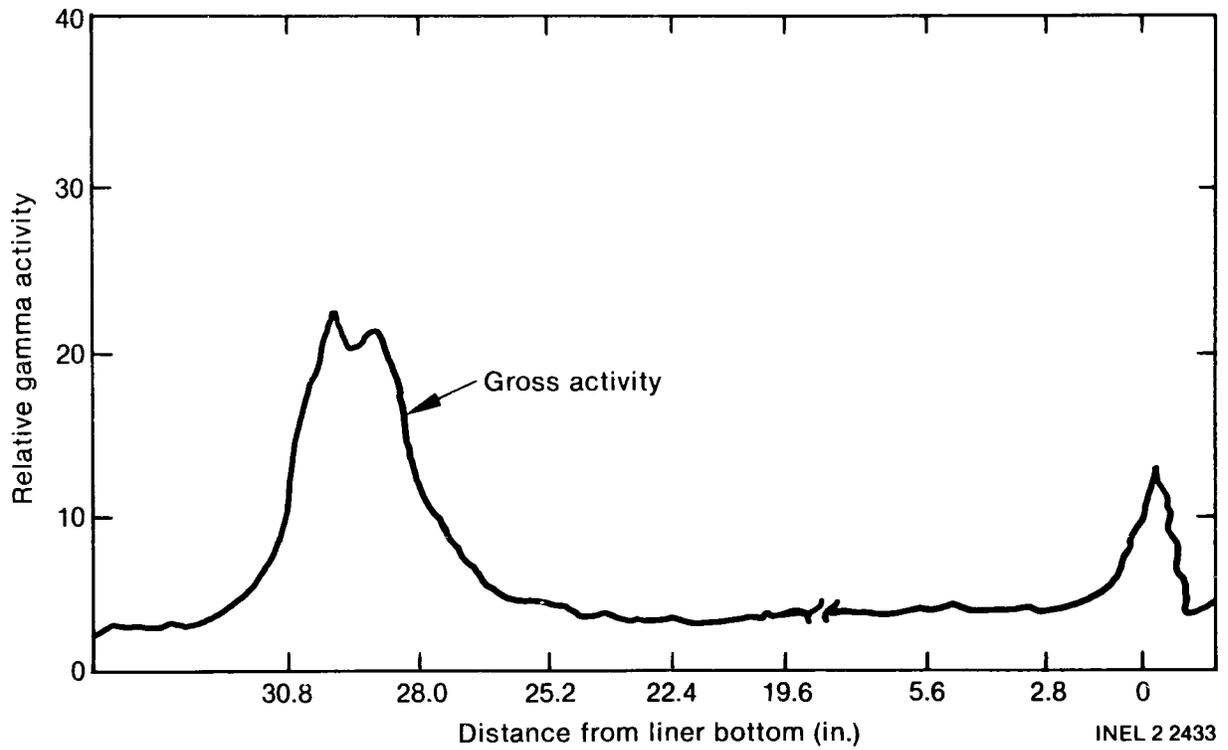


Figure 30. Gamma activity profile along outside of liner.

EXTERNAL RADIATION LEVEL PROFILE

Only limited information had been available concerning the radiation dose levels at contact with Liner PF-16. Such information was of interest for safe handling of the liners and development of high integrity container technology.

A profile of the gamma dose rate was determined by measuring the dose rate at intervals of 3 in. along the axis of the liner, using an ion-chamber probe. This was accomplished by securing a radiation level probe in a fixed position and moving the liner vertically, using an in-cell crane. The probe used was the Technical Associates Model CPMU High Range Underwater Monitor with a high range probe. Calibration of the instrument was provided by the manufacturer and verified by BCL personnel. The profile was measured at contact, 1 and 3 ft from the liner surface. The results of the profile are shown in Table 10. The cell background radiation level at the location of the profile measurement was between 30 and 60 R/h, depending upon the exact location being measured. It should be noted that the liner itself may have contributed to the background, as it could not be completely removed from the cell for this measurement.

Inspection of the data reveals higher level radiation readings at certain positions, at 1 and 3 ft, than observed at contact. It is suspected that this anomaly may be due to reflection of liner gamma radiation from the hot cell walls. Such reflection may contribute to any of the values observed, due to the proximity of the walls, and is not necessarily uniform. It is further recognized that there is an apparent inconsistency between the internal and external dose reading. However, it was not feasible within the scope of these measurements to design a measurement system that would completely eliminate reflected radiation; the numbers should, therefore, be considered approximate.

TABLE 10. LINER RADIATION LEVEL PROFILE

Distance From Top of Liner (in.)	Radiation Level (R)		
	Contact	1 ft	3 ft
0	590	620	400
3	720	740	410
6	960	890	410
9	1400	1000	410
12	1600	1000	370
15	1900	1000	240
18	2100	1000	300
21	2600	950	280
24	2800	780	250
27	2300	680	240
30	1100	610	230
33	620	550	220
36	350	460	210
39	200	380	200
42	140	310	180
45	110	250	180
48	90	210	160
51	60	170	150
53	50	150	140

LINER TEMPERATURE PROFILE

A temperature profile of the ion-exchange media was determined as close to the liner centerline as practical. A metal tube was inserted at a point near the centerline, positioning it such that it would not strike the effluent tube or the under-drain system. The tube was allowed to remain in place long enough to achieve thermal equilibrium with the media bed. A chromel-alumel thermocouple was inserted into the tube, positioned at the proper predetermined depth, allowed to reach thermal equilibrium, and the temperature recorded. This process was repeated for a total of 10 positions. The results of this measurement are shown in Table 11. The measurements indicate no significant temperature differences between media strata.

TABLE 11. LINER TEMPERATURE PROFILE^a

<u>Distance from Top of Media Bed (in.)</u>	<u>Temperature (°F)</u>
29	72.0
23	72.4
15	73.2
13	73.7
11	73.9
9	74.0
7	74.0
5	74.0
3	73.7
1	73.4

a. Cell ambient temperature is 73.8°F.

ION-EXCHANGE MEDIA RADIOCHEMISTRY

Ion-exchange media from each of the segments removed from the core sample were thoroughly blended to produce a homogeneous mixture. Samples of the media were removed from each of the segments corresponding to a given identifiable region of ion-exchange media. These were then blended together to form a single sample for each region. The radiochemical analyses were carried out on the blended samples, with all analyses being conducted in duplicate. The initial phases of the analyses were conducted in a small hot cell due to their high gamma activity.

The desired approach was to ash the organic ion-exchange media and then take the radionuclides in the solution through a pyrosulfate fusion. It was found, however, that the media did not ash sufficiently to enable fusion. The analytical procedure was then modified to substitute an acid leach for the ashing and fusion. The leaching consisted of heating ~10 g of ion-exchange media in a nitric acid and hydrochloric acid mixture at a temperature just below boiling for a period of 8 h.

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 also removed from the hot cell for the remainder of the analytical work.

The leachate was analyzed for the content of $^{238,239,240}\text{Pu}$, and for the content of ^{238}U by ion exchange separation followed by alpha spectroscopy. The strontium analysis was carried out using a nitration technique.

For each region of ion-exchange media, ~10-g samples were removed for analysis. These samples were leached in the nitric acid/hydrochloric acid solutions. Following the cesium removal and transfer from the hot cell, the leachates from these samples were split into two portions. One portion was used for the strontium analysis, the second for the uranium-plutonium

analyses. The results of all the analyses are summarized in Table 12. The analyses show that 80% of the observed gamma-emitting radionuclides are in Region I of the core sample. While Regions II and III contain ~20 % of the gamma activity in nearly equal proportions, these values may not be indicative of the distribution of activity within the liner, since there had been some degree of mixing between the layers during the coring operation. This appeared to be particularly true between Regions I and II, although some of the inorganic media from Region I were also observed in Region III as well. The distribution of the plutonium and uranium was more uniform within the bed; ~50% of the activity in Region I and the remainder distributed equally between the other two regions. The uranium activity was less than the minimum detectable limits in all three layers. The $^{89,90}\text{Sr}$ content was distributed relatively uniform through the media, with Region I having slightly more activity than the others.

On the basis of these analyses, it appears that Region I (the inorganic layer) has been quite effective in removing the cesium from the contaminated water; however, the strontium has been less effectively removed by that region and has been uniformly distributed throughout the media. Similarly, the plutonium has not been as efficiently removed by the inorganic media and has also been distributed throughout the other two regions.

TABLE 12. RADIONUCLIDE CONTENT OF EPICOR PF-16 ION-EXCHANGE MEDIA (Ci/g)

	Regions		
	I	II	III
$^{239,240,242}\text{Pu}$	$3.1 \pm 0.3\text{E-}10$	$1.1 \pm 0.1\text{E-}10$	$1.2 \pm 0.1\text{E-}10$
^{235}U	$<5 \text{ E-}12$	$<5 \text{ E-}12$	$<5 \text{ E-}12$
$^{89,90}\text{Sr}$	$1.6 \pm 0.1\text{E-}6$	$1.4 \pm 0.1\text{E-}6$	$9.9 \pm 0.2\text{E-}7$
^{137}Cs	$4.9 \pm 0.4\text{E-}3$	$7.3 \pm 1.5\text{E-}4$	$5.5 \pm 0.9\text{E-}4$
^{134}Cs	$6.4 \pm 0.4\text{E-}4$	$1.3 \pm 0.2\text{E-}4$	$4.4 \pm 0.8\text{E-}5$
^{60}Co	$4.8 \pm 1.7\text{E-}6$	$1.4 \pm 0.2\text{E-}6$	$5.5 \pm 1.4\text{E-}7$

LINER PREPARATION AND SHIPMENT FROM BATTELLE

Following the completion of the PF-16 examination, the liner was sealed, decontaminated, and shipped from Battelle Columbus Laboratories to the Idaho National Engineering Laboratory.

As the initial closure of the manway opening cover did not provide a sufficient seal, the original cover was removed and replaced with another cover fitted with a new but precompressed gasket. The liner manway extension was thoroughly cleaned to remove all corrosion and scaling. The gasket was coated with silicone rubber cement and secured in place using a clamping ring. The cement was allowed to cure for 24 h, and the liner leak tested, using helium gas and a pressure decay technique. Some small leaks were immediately detected and corrected. Further testing revealed a leak resulting in a pressure drop of 0.01 psi/h from an initial pressure of 1.5 psig. All the liner penetrations were tested using a liquid leak detector (SnoopTM), but no further leaks could be found. It was concluded that it would not be practical to attempt to make the liner more leak tight.

The liner was purged with nitrogen gas to remove any combustible gases from the liner prior to shipment. This was accomplished by flowing ~200 ft³ of dry nitrogen gas into the liner through the vent penetration and out the effluent penetration. This forced the gas through the media bed and the under-drain system into the effluent line. The purge was completed at 9:00 a.m. March 25, 1982.

The liner was decontaminated in the hot cell, using pressurized water, soap solutions, and methanol. Following the cleaning, the liner surfaces were smeared to determine the levels of transferable contamination. All surfaces were well below the limits of 250 dpm/100 cm²(α) and 10,000 dpm/100 cm²($\beta\gamma$).^a

a. Radioactive Contamination Control Program, Safety Manual 5040, EG&G Idaho, Inc., January 30, 1978.

The liner was covered with plastic sheets to avoid additional contamination and was transferred from the hot cell using the Battelle designed transfer/storage device. The transfer was completed March 26, 1982.

The shipping cask, CNS 8-120, leased from Chem Nuclear, Inc., was received at Battelles Hot Laboratory on March 29, 1982. The cask exterior was surveyed for residual activity, which was found to be well below the limits of 220 dpm/100 cm²(α) and 2200 dpm/100 cm²($\beta\gamma$).^a Following the surveys, the cask was unloaded from the trailer and the cover removed. Smear surveys revealed the activity inside the cask was well below the limits.

The cask was prepared for shipment by inserting the wooden dunnage and by fitting the transfer/storage device support ring to the cask. Prior to its insertion, the dunnage was painted with epoxy to facilitate decontamination. Metal facings were also removed in order to avoid metal-to-metal abrasion of the liner.

The liner was transferred into the cask on March 30, 1982. After the lid was secured, the cask was purged with \sim 300 ft³ of dry nitrogen. The gas in the cask was sampled and analyzed to ensure the purge was complete.

The cask was loaded onto the trailer, radiation surveys completed, and the trailer removed from the Hot Laboratory truck loading area. The shipment departed Battelle at 6:35 a.m., April 6, 1982.

a. 49-CFR, 173.393 (j).

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