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***Examination of SNAP-27
Heat Source FCA4 and
Preparation of Fuel for FCA8***

D. L. Prosser

July 23, 1971

AEC Research and Development Report

MOUND LABORATORY

Miamisburg, Ohio

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ABSTRACT

The capsule and fuel of SNAP-27 fuel capsule assembly (FCA)4 were examined for changes which may have occurred during its life of 2.25 yr. No significant effects were noted. The fuel was reprocessed and qualified for use in SNAP-27 FCA8.

INTRODUCTION

In early 1970 the U. S. Atomic Energy Commission authorized the fabrication of a SNAP-27 heat source for the Apollo 17 lunar mission. To provide the fuel for the source the plutonium-238 dioxide microsphere fuel was to be removed from the fuel capsule assembly (FCA)4 which had been made in May 1968 for qualification test purposes. Because FCA4 had been subjected to a more stringent vibration and shock testing program than the flight capsules and had seen an extensive and varied thermal environment, it was considered important to examine the fuel and capsule in detail to determine the effects of the physical and thermal exposures on them and to evaluate the suitability of the fuel for use in the new capsule.

The results of the examination would indicate the condition of the flight units. These units had not experienced as severe a physical or thermal regime as FCA4 and should be in at least as good condition as FCA4. The tests which were performed on the capsule assembly were:

I. Capsule Evaluation

1. Visual examination
2. Dimensional test
3. Leak test
4. Radiographic examination
5. Metallurgic examination

II. Fuel Evaluation

1. Dose rate measurements
2. Helium retention studies
3. Fuel fines determination
4. Helium removal studies

Following the test program, which began in July 1970, the fuel was baked out to remove the helium. The fuel then was processed and sealed into

the capsule in accordance with the SNAP-27 heat source specification MD1-11391.

EXPERIMENTAL

I. Capsule Evaluation

1. Visual Examination - The capsule was inspected primarily to determine the condition of the emissive coating on its surface. The unit was supported vertically in a holder and the coating was examined along the length of the capsule and at several locations around its circumference. A Questar telescope was used to study the coating. The coating was found to have only a few minor defects and appeared to be in good condition considering the thermal cycling and mechanical stresses¹ to which the capsule had been subjected.

2. Dimensional Test - The capsule was placed in the final assembly gage in air and passed both the straightness test for the bottom of the capsule and the closure weld diameter test. Any loss of straightness or increase in diameter was minimal, as the capsule was still within specification. On length it was found that the leaf gage at the bottom of the gage assembly would not pass completely under the capsule. It was estimated that the increase in length was of the order of a few thousandths of an inch. Because the capsule was coated, the assembly gage used at the General Electric Vallecitos plant was obtained for the test.

3. Leak Test - A helium leak test was performed to determine the integrity of the capsule and the welds. The leak rate was found to be 9.7×10^{-7} std cc helium/sec, 26 months after manufacture. The value at time of fabrication was 1.0×10^{-7} std cc helium/sec. The accuracy of the measurements was $\pm 20\%$.

4. Radiographic Examination - The clad closure welds were examined by tangential radiography. The welds were found to be free of defects as they were at the time of fabrication.

5. Metallurgic Examination - One-inch-square sections of the walls from both fueled liners along with a section from an unfueled liner were sent to the Los Alamos Scientific Laboratory (LASL) for metallographic analysis. The study was performed to determine whether any reaction had occurred between the liner material (Haynes alloy No. 25) and the

fuel. The sections were given the following designations:

- 1A - Outer tube of fueled liner A
- 1B - Inner tube of fueled liner A
- 2A - Outer tube of fueled liner B
- 2B - Inner tube of fueled liner B
- 3A - Outer tube of empty liner

The outer tube sections included a portion of the circumferential weld used to join the bottom cap to the outer tube. One of the inner tube sections, 2B, contained a portion of the seam weld used in forming the tubing. The approximate locations of the sections taken from the liners are shown in Figure 1.

The sections were mounted and polished at LASL and etched electrolytically in a solution of four parts HCl, one part HNO₃, one part HAc, and one part H₂O at a voltage of 1.5 V for 1-2 sec.

Preliminary photographs of the sections taken at LASL are shown in the following figures. All views were taken at a magnification of 250X after etching. Figure 2 illustrates the condition of the inner and outer surfaces of the inner tube of the fueled liner B. Views were taken both of the parent metal and of the longitudinal seam weld. No appreciable reaction between the fuel and the tube was noted as seen in views C and D. The reaction is minimal considering the age and thermal history of the capsule. In Figure 3 similar views are presented for both surfaces of the outer tube of liner B. No evidence of fuel-liner reaction was apparent. Figure 4 reveals the inner surface of the bottom cap which was in contact with the fuel. There is no significant indication of any reaction. It is interesting, however, to note that the grain size of the metal in the bottom cap is much larger than in the tubing (Views A and C, Figure 3). For comparison, views of the inside surface of the longitudinal seam weld of the outer tube of the unfueled liner and of the outer surface of the circumferential weld are given in Figures 5 and 6.

The five liner samples used for the metallographic study were also subjected to microhardness measurements at LASL to evaluate the characteristics of the metal. Results of microhardness measurements are given on page 12.

Figure 1 SNAP-27 Liner Assembly
Showing Location of Metallographic Sections

FIGURE 2: Metallographic section of inner tube of liner B as viewed along axis of tube. 250X-etched

FIGURE 3: Metallographic section of outer tube of liner B as viewed normal to axis of tube. 250X-etched

FIGURE 4: Metallographic section of inner surface of bottom cap of liner B normal to axis of liner. 250X-etched

FIGURE 5: Metallographic section of seam weld of outer tube of empty liner as viewed along axis of liner. 250X-etched

FIGURE 6: Metallographic section of outer surface of circumferential weld of empty liner normal to liner axis.
250X-etched

<u>Samples</u>	<u>Location</u>	<u>DPH (100-g load)</u>
1A	fine grained base metal	314, 336
1A	heat affected zone	319, 327, 333
1A	weld	363, 345, 354
1A	coarse grained base metal	283, 297, 297
2A	fine grained base metal	354, 330
2A	heat affected zone	360, 363, 380
2A	weld	360, 345, 357
2A	coarse grained base metal	294, 302, 292
1B	base metal location 1 location 2 location 3	325, 322, 319 299, 322, 342 336, 330
2B	base metal	383, 366, 383, 383
3A	base metal	366, 357, 360, 366
3A	weld	345, 383, 336, 342

The microhardness readings show no anomalous values which would indicate age embrittlement of the material.

To check for gross hardening effects, sample 1A was bent back and forth 90° over a 1/8 in. radius. The specimen survived two bends and broke at the third bend. Some indications of brittle failure were observed, but survival through two bends shows that any age-hardening was minimal.

The unexposed sample, 3A, was also bent and survived four bends. When failure occurred, it was ductile in nature.

Although slight age hardening of the Haynes-25 in FCA4 appears to have occurred when compared with the control sample, the effects are minimal.

II. Fuel Evaluation

1. Dose Rate Measurements - For these measurements the heat source

was held horizontally on a heat sink and cooled with two 12-in. fans. Both neutron and gamma dose rate measurements were taken at various distances from the side and each end of the source.

The measurements are listed in Table 1 and are compared with those made at time of encapsulation 26 months ago. The significant increase in the gamma radiation is due to the ingrowth of the thallium-208 isotope.

The neutron measurements were made using a Texas Nuclear Nemo spherical Neutron Dosimeter Series 9140, with a 10-in. diameter sphere. The gamma measurements were made with a Victoreen Radector III Gamma Survey Meter having an accuracy of $\pm 20\%$. Both instruments were calibrated before and after the measurements.

2. Helium Retention Studies - The study of the helium content of the capsule was performed in two parts. First the quantity of helium in the capsule which had escaped from the fuel was determined before the fuel was removed. Then the helium still retained by the fuel was evaluated.

To obtain the quantity of helium released by the fuel the apparatus shown in Figure 7 was used. Pressure measurements were made with a Model 100H MKS Baratron pressure transducer. Procedures for determining the volume of the system and the amount of helium is described in more detail in Appendix A.

A special fixture was designed to accommodate the SNAP-27 heat source capsule. It is shown schematically in Figure 8. Temperature measurements were made during the entire process by means of six thermocouples strapped to the capsule. The thermocouples were positioned to avoid contact with the wall of the drilling fixture. A multipoint recorder was used to continuously record the temperatures. The location of each thermocouple is shown schematically in Figure 9. From the thermocouple readings it was hoped to determine whether one or both of the burst discs on the liners had been ruptured. The condition of the discs would control the helium content of the capsule at time of drilling which in turn would affect the thermal conductivity characteristics of the capsule and the temperatures at the thermocouples. Drilling was accomplished with a hand drill and a carbide drill bit brazed to a steel shaft. O-ring seals around the shaft helped maintain the necessary vacuum during drilling. The dome end of the capsule was drilled first.

The gas data are summarized in Table 2. The values for the dome end are low because the drill was forced from the drilling fixture by the pressure of helium when the strength member was penetrated. As a result, some of the gas escaped. The excessive pressure was caused because only the 311.2 cc standard volume was open to the system. The pressure indicated that the burst disc on the dome end liner was ruptured. From a knowledge of the weight of the drill and of the volumes of the capsule, a minimum helium content of 445 cc was calculated for the dome end of the capsule. The dome end between the strength member and the liner was then evacuated

Table 1

NEUTRON AND GAMMA DOSE RATE MEASUREMENTS
FOR SNAP-27 FCA4

Side

Center of Source to Center of Detector (cm)	Neutron (mrem/hr)		Gamma (mrem/hr)		Total (mrem/hr)	
	5-27-68	7-10-70	5-27-68	7-10-70	5-27-68	7-10-70
20	1769		140		1909	
25	1225	1209	101	140	1326	1349
40	535	519	45	80	580	599
50	365	346	30	52	395	398
100	99	97	7.1	16	106.1	113
200	31	29.8	2.1	4	33.1	33.8

Curved End

40	404	396	20	40	424	436
50	213	204	9	21	222	225
60	137	137	5.5	12	142.5	149
100	45	45.1	1.9	3.3	46.9	48.4
120	33	31.9	1.3	2.2	34.3	34.1
200	14	13.9	0.6	1.0	14.6	14.9

Flanged End^a

40	294	304	12	28	306	332
50	157	156	6	14	163	170
60	99	97.2	3	8.5	102	106
100	35	33.2	1.3	2.5	36.3	35.7
120	25	24.1	1.0	1.7	26	25.8
200	13	12.4	0.6	0.8	13.6	13.2

^a Readings taken through 1/4 in. thick stainless steel handling plate bolted to flange.

FIGURE 7 - Schematic diagram of helium release measuring apparatus

FIGURE 6 - Schematic diagram of drilling fixture

FIGURE 9 - Schematic diagram of thermocouple locations

until the thermocouple readings stabilized. The dome liner was drilled and the volume of gas released was measured. The volume was calculated to be 90 cc as listed in Table 2. After further evacuation, the strength member of the flange end was drilled. No gas was collected. Presumably any gas which may have been present in this section of the capsule had been removed through the vents by prior pumpings on the dome end. Drilling through the liner produced 728 cc of helium indicating that the flange liner burst disc was still intact. This volume of gas is considered to be more accurate than those volumes for the dome end because there was no evidence that any of the gas collected from the flange end was lost.

The sequence of operations and the time required for each are illustrated in Figure 10.

The numerical values used for the void volume of the fuel annulus and the center cylindrical volume of the liner were calculated rather than measured because measurements of the volume of the system after drilling was complicated by several factors such as the complexity of the free volume of the system which in some cases included the free volume of the microspheres, the volume of the apparatus, the volume of the space between the filters, and the volume between the strength member and the liner. The volumes were determined using a water measurement method with representative capsule components. Adjustment for fuel volume was based on the particle density of the fuel.

A trace of the temperature profile of the various thermocouples is given in Figure 10, and the temperature drop for each thermocouple after each operation is given in Table 3. From this table it appears that the dome end burst disc was ruptured while the flange end burst disc was not. This conclusion is supported by the data in Table 2 because no gas was collected after drilling through the strength member at the flange end.

Table 2

SUMMARY OF HELIUM MEASUREMENTS ON SNAP-27 FCA4

<u>Region</u>	<u>Amount of Helium at STP (cc)</u>	
	<u>Dome End</u>	<u>Flange End</u>
Between liner and strength member	445	0
Fuel annulus and central void	90	728
Total	535	728

FIGURE 10 - Trace of temperature profiles on thermocouples in SNAP-27 heat source capsule during helium release measurements

Table 3
TEMPERATURE DATA ON SNAP-27 FCA4

DOME END^a

	Thermocouple Number					
	(flange end)			(dome end)		
<u>Drill Strength Member</u>	1	2	3	4	5	6
After pump down and before drilling (A)	427	415	510	440	425	590
After drilling (B)	96	265	175	~120	245	255
After pump down (C)	278	377	368	~300	400	440
ΔT (A-C)	-149	-38	-142	~-140	-25	-150
<u>Drill Liner</u>						
Before drilling (D)	278	377	368	~300	400	440
After drilling (E)	110	280	205	125	270	280
After pump down (F)	200	335	320	200	300	340
ΔT (D-F)	-78	-42	-48	-145	-100	-100
Total ΔT (A-F)	-227	-80	-190	-240	-125	-250

^a All values are given in °C.

Table 3 (Continued)

FLANGE END^a

	Thermocouple Number					
	(flange end)			(dome end)		
<u>Drill Strength Member and Liner</u>	1	2	3	4	5	6
Before drilling (G)	200	335	320	200	300	340
After drilling (H)	90	255	165	110	260	280
After pump down (I)	120	310	235	150	325	330
ΔT (G-I)	-80	-25	-85	-50	+25	-10
Total ΔT (A-I)	-307	-105	-275	-290	-100	-260

^a All values are given in °C.

When the strength member on the dome end was drilled, the temperature drop in both ends after pump down [$\Delta T(A-C)$ of Table 3] was about the same suggesting that the helium measured came from the void space between the liner and strength member in both halves of the capsules. The configuration of the capsule shows this to be true if the vents are considered to be open channels between the two halves. However, the volume of gas measured was quite high suggesting that one of the burst discs had ruptured. Drilling of the dome end liner produced a relatively small amount of helium and a much greater drop in temperature after pump down [$\Delta T(D-F)$ of Table 3] in the dome end thermocouples than in the flange end thermocouples. This suggests that the dome end burst disc ruptured as referred to previously in Table 2. The condition of the discs was confirmed visually when the strength member (clad component) was cut away from the liners after the fuel had been removed. The dome disc was ruptured whereas the flange disc appeared intact. The total temperature drop [$\Delta T(A-I)$ of Table 3] in the dome end was about the same as the total temperature drop in the flange end. This would be expected if all of the helium had been removed from the void volumes of each half.

Because it appeared that the burst disc on the flange liner had remained intact, it was interesting to calculate the pressure differential across the disc to determine what stresses it could withstand after being aged for 2.25 yr. The calculation was made after the dome liner had been evacuated, but before the flange liner was opened to give the maximum pressure which would exist across the disc.

Upon drilling through the flange liner the helium expanded into a total volume comprising the following components.

Large reservoir	3994.1 cc
Piping and drill fixture	277.6
Capsule	<u>674.1</u>
	4945.8 cc

The gas pressure was 11.2 cm Hg. The free volume of the flange liner as determined by water measurements and from the particle density of the fuel was 308.5 cc. Therefore, the pressure at room temperature in the liner before drilling was 179 cm Hg. As seen in Figure 10, the strength member temperature was 315°C (600°F) for about 15 min before opening the liner. From the data in the General Electric summary report² it was estimated that the bulk helium temperature at the disc would be about 900°F. Therefore, the pressure in the liner would be 462 cm or 6.08 atm at this temperature indicating the magnitude of the pressure differential across the disc.

² SNAP-27 Summary Report DIN, GEMS 402, General Electric Company, (July 15, 1970), pp. 6-17.

Following the determination of the volume of helium in the capsule released by the fuel, experiments were performed on fuel samples to estimate the amount of helium retained by the microspheres.

Representative samples of microspheres from each of the capsule liners and a composite (1:1) sample of both liners were used for this study. The data collected on each sample were corrected back to the day the capsule was opened. The information then could be combined with the data collected during the opening of the capsule to provide a value for the amount of helium present at that time.

Approximately 0.250 g of microspheres from Liner A (dome end) were used in the first experimental run. The sample was placed in the helium release experimental setup and heated to approximately 1980°C in vacuum. The sample was maintained at this temperature until a decrease in the helium release rate was noted. The sample was cooled and taken off test when it was determined that the helium release rate had decreased to the background rate of the measuring equipment. The results of this experiment are shown in Table 4.

Another portion of the same sample of microspheres was tested after it was determined that the results from the first portion did not correspond with the results from the other samples tested. The results for this sample are also shown in Table 4.

Two portions of the sample of microspheres from Liner B (flange end) were tested in exactly the same manner as those from Liner A. The results are given in Table 4.

The Composite A-B was prepared as a uniform mixture of microspheres from Liners A and B. One portion of the sample of the composite was tested and the results are listed in Table 4.

Table 4

HELIUM INVENTORY DATA FOR SNAP-27 FCA4

<u>Sample</u>	<u>Sample Weight (g)</u>	<u>Helium Inventory^a (cc/g)</u>	<u>Helium Inventory (expressed in months)</u>
Liner A-1	0.250	0.2429	5.62
Liner A-2	0.198	0.1979	4.58
Liner B-1	0.279	0.8350	19.33
Liner B-2	0.164	0.8674	20.08
Composite AB	0.248	0.7691	17.80

^aCorrected to date of capsule opening.

As seen in Table 4, there is an apparent anomaly in the experimental results. The data on the samples from Liner A do not correspond with the data on the other samples. When these data are combined with the capsule opening data, it becomes obvious that the sample from Liner A was not representative. It is assumed that the Liner A sample was from the stratified high temperature region of the liner and, as a consequence, contained much less helium.

Since the data from the Liner A sample are questionable, they were eliminated from further consideration. However, the data for the composite sample approximately equal the data for the Liner B sample. The composite was prepared as a uniform mixture of microspheres from Liners A and B; and if a sample ratio of 1:1 is assumed, it can be stated that the data collected on the composite sample should reflect the approximate amount of helium retained by the microspheres in both Liner A and Liner B since they were exposed to the same thermal history during encapsulation. With the above assumptions, it is now possible to average the helium inventory values obtained for the samples from Liner B and the composite sample. This average helium inventory value is 0.8236 cm³ of helium per gram of PuO₂ which is equivalent to a helium production of 19.06 months being retained by the fuel.

The above inventory value together with the gas volumes in Table 2, were used to determine the total amount of helium present in the SNAP-27 FCA4 just prior to opening. These data are presented in Table 5. The volume of helium retained by the fuel in each liner was determined by multiplying the average value of 0.8236 cm³ He/g PuO₂ by the fuel weight in each liner listed in the table.

Table 5

AMOUNT OF HELIUM PRESENT IN SNAP-27 FCA4
PRIOR TO OPENING

	<u>Liner A</u>	<u>Liner B</u>	<u>FCA4 Capsule</u>
Fuel loading (g PuO ₂)	1913.41	1884.8	3798.21
Amount of helium released on opening (cm ³)	535	728	1263
Amount of helium retained by the fuel (cm ³)	1575	1552	3128
Total amount of helium present (cm ³)	2111	2280	4391
Percent released of total		32	

The amount of helium which would have been generated during the encapsulation period of 2.25 yr was calculated using the theoretical helium generation rate of 1.67×10^{-6} cm³ of helium per gram of PuO₂ per second. The calculated values of gas based on the fuel loading of each liner are given in Table 6 along with the experimental values from Table 5.

Table 6

COMPARISON OF THE EXPERIMENTAL AND THEORETICAL
HELIUM INVENTORIES OF SNAP-27 FCA4

	<u>Liner A</u>	<u>Liner B</u>	<u>FCA4 Capsule</u>
Calculated helium inventory (cm ³)	2272	2235	4507
Experimentally determined helium inventory (cm ³)	2111	2280	4391

The data show a reasonable comparison between the theoretical values and the experimentally determined ones. Since the fuel had a prior encapsulation history before being loaded into FCA4, the experimentally determined values should be greater than the theoretical values listed in Table 6. This is true in the case of Liner B. In the case of Liner A, however, the amount of helium released on capsule opening was an estimated value rather than a measured value and the sample of microspheres from this liner used for the helium inventory measurement was not representative of the fuel. Actually the quantity of helium recovered from Liner A should have been greater than for Liner B for two reasons.

1. There was more fuel in Liner A.
2. The fuel in Liner A was made up of 84% of fuel from SNAP-19 capsules and 16% of bonded fuel whereas the fuel in Liner B consisted entirely of SNAP-19 fuel.

The SNAP-19 fuel had been subjected to a severe thermal environment which should have driven off much of the helium before being used for fueling FCA4. On the other hand, the bonded fuel which had not experienced such a severe thermal history should have contained more helium at time of encapsulation. Therefore Liner A should have released more helium when it was opened. The capsule should have contained at least twice the quantity of gas obtained from Liner B or 4560 cc. Therefore the volume of gas in the fuel at time of encapsulation of FCA4 should have been (4560-4507)cc or 53 cc. This amount is equivalent to the quantity of gas which would have been generated by the 3798.21 g of fuel in 10 days.

The gas pressure which could have existed in FCA4 at an assumed operating temperature of 1500°F (815°C) would be 23.56 atm. The value is based on the free gas volume of the capsule of 674.1 cc and a helium content of 4391 cc. This pressure would apply after the fuel had been encapsulated for 2.25 yr if all the helium was released in this period. However, only 32% of the helium was released by the fuel in Liner B. Therefore the pressure first created at 1500°F would be 7.54 atm. The pressure of 23.56 atm would not be reached until after the fuel had been at this temperature for several years. After several years though, the pressure would be somewhat higher due to the additional helium generated during this period which in turn would be offset to some extent by the decrease in temperature from the additional decay of the fuel and the possible increase in the capsule volume due to creep. At higher temperatures experienced during reentry the bulk of the gas would escape from the fuel in a matter of a few minutes. Again for FCA4 a maximum pressure of 30.87 atm would be reached at a temperature of 2085°F (1140°C). At a temperature of 2400°F (1315°C) all the gas would be driven from the fuel very rapidly reaching a pressure of 34.69 atm.

As noted previously the fuel in FCA4 had experienced a rather unusual thermal history. In addition the fuel, while in the SNAP-19 capsules, had been exposed to severe thermal conditions which would account in part for low calculated volume of retained helium of 53 cc at time of encapsulation in FCA4. A summary of the time-temperature data for the fuel from the four SNAP-19 capsules used is given in Table 7. Similar data for FCA4 during its life are given in Table 8. For FCA4 the temperatures for 850°F and above apply to the capsule while under test. For lower temperatures the capsule was in storage.

It can be seen from the data for Liner B in Table 5 that the helium retained by the fuel was 68% of the total in spite of the elevated thermal environment experienced by the fuel. The fuel in the prime capsules prior to launching should not normally be subjected to the thermal regime imposed on FCA4 and should be able to retain at least as much helium as the fuel did in FCA4. In addition because of the older fuel used in FCA4 there would be a greater tendency for helium release.

Table 7

THERMAL HISTORY OF SNAP-19 CAPSULES

Capsule Number	°F °C	Time at Temperature (hr)								
		350 177	380 193	650 343	825 441	900 482	1000 538	1100 593	1325 718	1425 774
307				1008	6192	2064	1488			
341,360								2400	672	288
346		192	240	936	6192	2064	1488			

Table 8

THERMAL HISTORY OF SNAP-27 FCA4

<u>Location</u>	°F	<u>Time at Temperature (hr)</u>								
	°C	<u>212</u>	<u>500</u>	<u>520</u>	<u>850</u>	<u>1050</u>	<u>1100</u>	<u>1200</u>	<u>1250</u>	<u>1350</u>
		<u>100</u>	<u>260</u>	<u>270</u>	<u>480</u>	<u>565</u>	<u>595</u>	<u>650</u>	<u>675</u>	<u>730</u>
Mound		2253 ^a	5 ^t	30 ^b						
GE			17616 ^c	93		39	3	20	121	166
TRW					<u>350</u>					
		<u>2253</u>	<u>17621</u>	<u>123</u>	<u>350</u>	<u>39</u>	<u>3</u>	<u>20</u>	<u>121</u>	<u>166</u>

^a Fuel in liners 216 hr, in uncoated capsule 353 hr, in coated capsule 1684 hr.

^t Fuel in uncoated capsule.

^b Includes total time for coated capsule in GSC at Mound, GE, and TRW.

3. Fuel Fines Determination Following the helium measurement study, an evaluation of the present condition of the fuel in the capsule was undertaken. As part of the radiographic examination of the capsule, cobalt radiography was used to determine whether the fuel had changed position. No slumping of the fuel was detected. After the helium had been removed from the capsule, the capsule was cut in half at the center section weld to facilitate the extraction of the fuel. Then the flanged and dome clad ends and the liner caps were cut away to expose the liner cap weld and the zee-ring. Each half was doubly encapsulated in sealed containers and transferred under water to the fuel removal facility. The dome end liner, now designated Liner A, was opened first. The assembly, including the cladding, was placed in a water-cooled chill block and left overnight. A circumferential cut was made below the liner cap weld with compound snips and the remainder of the zee-ring was removed. All six shims were found still resting on top of the fuel column in the position that they were placed originally. The shims were removed and the fuel was poured in increments of 200-300 g into a 6-in. diameter stainless steel screen pan. Each increment then was transferred to a stainless steel fuel container. The fuel flowed readily with about 10-15% of fuel (as estimated visually) consisting of agglomerates less than 1/2 in. x 1 in. x 1/8 in. in size. The agglomerates appeared to consist of thin layers which may have been formed in the hotter region of the fuel volume. The agglomerates broke up readily when shaken on the screen pan. The fuel was collected in seven fuel containers. Next the liner from the flange end, now called Liner B, was opened in the same manner as Liner A. The only difference was that

this liner was stored in the chill block for two days before the zee-ring was removed. Again, the eight shims originally placed in the liner were found on top of the fuel. The fuel was free flowing during removal and devoid of clumps. The fuel was distributed among seven fuel containers. The weight of fuel in each batch for both liners is shown in column 2 of Table 9. The zirconium dioxide filler in both liners was distributed throughout the fuel. The filler apparently was more evenly distributed throughout the fuel in the dome liner than in the flange liner. This difference will be discussed in more detail later.

After the fuel containers had been removed from the glovebox in which the fuel transfer was made the box was wiped down, and the wipings and the box filter were gamma-scanned to estimate the amount of plutonium remaining in the box. For the same reason the capsule metal components were also scanned. The results of the scan expressed in grams of isotope are as follows:

Dome Liner (A)

	Residue (g isotope)
Metal components	0.267
Wipes	<u>0.238</u>
	0.505

Flange Liner (B)

Metal components	0.151
Box filter	0.026
Wipes	<u>0.425</u>
	0.602

The metal components were sealed into a metal storage container to be held for future metallographic examination.

The elutriation column used originally for determining the fines content of microsphere fuel was modified in an attempt to measure the fines which existed in the FCA4 fuel at the time of its removal. The column was changed to obtain a continuous sample of the washings from each batch of fuel in the column. Normally only the last 500 ml of the 20 liters used for washing a batch was collected to determine the fines content of the batch. For this study, however, it was desirable to collect all the fines which were present in the fuel. After several batches of fuel from Liner B were washed, it became apparent that the fines measurements were on the low side. Accordingly another method for determining the fines content of the fuel was adopted. The method, in addition to providing more reliable data, allowed the fines to be

Table 9

WEIGHT OF FUEL FROM BOTH LINERS AFTER WASHING AND SCREENING^a

<u>Batch Identification</u>	<u>Weight Prior to Fines Determination</u>	<u>Weight Prior to Screening</u>	<u>Weight 53µm<x<250µm</u>	<u>Weight <53µm Particles</u>	<u>Wt % of <53µm Fines/ Batch wt</u>	<u>Weight >250µm Particles</u>	<u>Wt % of >250µm Fines/ Batch wt</u>	<u>Total Weight After Screening</u>	<u>Screening Difference</u>
27-4-B1	394.1	392.9	373.0	1.1	0.28	18.4	4.68	392.5	-0.4
27-4-B2	297.2	293.8	278.3	1.8	0.61	13.4	4.56	293.5	-0.3
27-4-B3	324.2	320.7	309.3	2.8	0.87	8.9	2.78	321.0	+0.3
27-4-B4	325.5	319.2	314.5	2.8	0.88	1.4	0.44	318.7	-0.5
27-4-B5	290.4	286.2	281.7	3.2	1.12	0.6	0.21	285.5	-0.7
27-4-B6 ^b	279.3	276.4	272.0	3.3	1.19	0.5	0.18	275.8	-0.6
27-4-B7	11.3								
Totals	1921.8	1889.2	1828.8	15.0		43.2		1887.0	-2.2
^b 27-4-B7 (11.1g) combined with 27-4-B6 prior to washing.									
27-4-A1	290.2	286.0	277.4	0.4	0.14	7.3	2.55	285.1	-0.9
27-4-A2	280.8	273.0	260.6	0.6	0.22	11.3	4.14	272.5	-0.5
27-4-A3	280.5	275.3	258.9	0.4	0.15	15.7	5.71	275.0	-0.3
27-4-A4	253.6	248.9	233.3	0.3	0.12	14.8	5.95	248.4	-0.5
27-4-A5	310.7	304.0	290.3	0.8	0.26	12.1	3.98	303.2	-0.8
27-4-A6	366.4	359.3	343.9	1.3	0.36	15.0	4.18	360.2	+0.9
27-4-A7	145.0	143.8	136.9	1.4	0.97	4.6	3.20	142.9	-0.9
Totals	1927.2	1890.3	1801.3	5.2		80.8		1887.3	-3.0
Capsule FCA 4									
FCA 4		3779.5	3630.1	20.2		124.0		3774.3	-5.2

^a All values given are grams of oxide except as noted.

segregated into several size ranges. The method consisted of placing a batch of fuel including the filler material on a 37 μm screen and washing with about 400 ml of washing solution. The fuel then in turn was placed on a 44 μm screen and a 53 μm screen. All the fuel from Liner A except batch 27-4-A7 was treated in this manner. Batch 27-4-A7 had been used previously for checking the elutriation column. In all cases the washings and sieve fractions were saved for analysis. The results of the analyses are listed in Table 10 and summarized in Table 11. The washings from the 37 μm screen were allowed to stand and the supernatant liquid was decanted. The values for the plutonium in the decanted liquid are given in row one of the table. The values for the residue are given in row two. The values for the washings after the fuel had been placed on a 44 μm screen and the 37-44 μm particles are shown in rows three and four. The values for the first and second washings from the 53 μm screen and the 44-53 μm particles are listed in rows five, six, and seven. Finally, each batch was washed in the elutriation column using 15 liters of washing solution. Samples of each solution were analyzed and the values are reported in the eighth row. A total of 22.496 g (isotope) of fines were removed by washing from six of the batches of fuel of the dome liner. To this value can be added the 0.505 g of fines detected on the capsule components and wipes by gamma scanning. The total fines content of 23.001 g is equivalent to $\frac{23.001}{0.6682} \times \frac{100}{1749.1}$ or 1.97% of the fuel in the six batches. The factor 0.6682 is the ratio of isotope to oxide weights for the fuel at time of removal from the liner. The weight of fuel (1749.1 g) is the weight of the first six batches of fuel removed from Liner A less the weight of the filler (33.1 g) added to the liner originally.

Figures 11, 12, and 13 are representative views of the shape of particles in the three size ranges of fines removed from one of the batches of fuel from the A liner. The larger pieces of material which are most noticeable in Figure 13 are assumed to be filler material.

The weight of particles $>250 \mu\text{m}$ given in Table 9 totalled 80.8 g for Liner A. Of this weight, 49.2 g consisted of PuO_2 as determined by calorimetry. Therefore, the difference in weight of 31.6 g was assumed to consist of filler material. This assumption appears reasonable because the amount of filler added to the liner was 33.1 g. For the B liner the weight of particles $>250 \mu\text{m}$ in size amounted to 43.2 g as compared with 42.4 g of filler which was added to the liner. For this liner it appears that very little of the material consisted of PuO_2 as contrasted with the findings for Liner A. Examination of the material from Liner A under a microscope revealed that the plutonium dioxide consisted of agglomerates in which two or more of the individual microspheres were bonded together. These findings coincide with the earlier observations which showed that the fuel when poured from Liner A had some large agglomerates whereas the fuel in Liner B had none.

After washing, the fuel was screened to segregate the material in the 53 to 250 μm range as required by the SNAP-27 fuel specification. The results of the screening operations are shown in Table 9. The screening

Table 10

FINES ANALYSIS OF FUEL FROM LINER A (DOME END)

<u>Fraction^a</u>	<u>Batch Number^a</u>						<u>Total</u>	<u>Fines (wt %)</u>
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>		
<37 μ m wash	0.060	0.080	0.052	0.039	0.084	0.155	0.470	2.09
<37 μ m fines	0.777	0.373	0.510	0.580	0.875	1.275	4.390	19.51
44 μ m wash	0.051	0.000	0.162	0.010	0.087	0.009	0.319	1.42
37-44 μ m fines	0.383	0.723	0.207	0.429	0.724	0.844	3.310	14.71
1st 53 μ m wash	0.034	0.003	0.112	0.085	0.034	0.059	0.327	1.47
2nd 53 μ m wash	0.052	0.000	0.025	0.086	0.017	0.051	0.231	1.02
44-53 μ m fines	1.218	2.423	1.508	0.823	1.621	1.475	9.068	40.31
Column Wash	1.840	1.545	0.120	0.006	0.135	0.735	4.381	19.47
Total	4.415	5.147	2.696	2.058	3.577	4.603	22.496	
wt % fines	19.63	22.88	11.98	9.15	15.90	20.46		

^a All sieving was done using U. S. Standard sieves having a permissible variation in average opening of 7%.

^b All values are grams isotope except as noted.

Table 11

SUMMARY OF FINES ANALYSIS OF FUEL FROM LINER A (DOME END)

<u>Size Range</u>	<u>Batch Number^a</u>						<u>Total</u>	<u>Fines (wt %)</u>
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>		
< 37 μm	2.677	1.998	0.682	0.625	1.094	2.165	9.241	41.08
37-44 μm	0.434	0.723	0.369	0.439	0.811	0.853	3.629	16.13
44-53 μm	<u>1.304</u>	<u>2.426</u>	<u>1.645</u>	<u>0.994</u>	<u>1.672</u>	<u>1.585</u>	<u>9.626</u>	<u>42.79</u>
Total	4.415	5.147	2.696	2.058	3.577	4.603	22.496	
wt %	19.63	22.88	11.98	9.15	15.90	20.46		

^a All values are grams isotope except as noted.

FIGURE 11 - Fines particles ($<37\mu\text{m}$) washed from fuel
in liner A. 98X

FIGURE 12 - Fines particles (37-44 μ m) washed from fuel
in liner A. 98X

FIGURE 13 - Fines particles (44-53 μ m) washed from fuel
in liner A. 98X

of Liner A produced 5.2-1.4 g or 3.8 g of particles with one dimension less than 53 μm in size from the six batches of fuel which were washed. If this weight is added to the total weight 23.001 g noted above, then the total weight percent of fines smaller than 53 μm is $(\frac{23.001}{0.6682} \times \frac{1}{1.7491} + 3.8) \times 100$ or 2.19.

The weights of particles greater than 250 μm in size are assumed to consist largely of zirconium dioxide filler because the filler size range used was nominally 300-590 μm , whereas the fuel was in the 50-250 μm size range. The fuel at the top of Liner B was poured into fuel container 27-4-B1 and the fuel at the bottom into 27-4-B7. The fuel from Liner A was transferred in the same manner into the containers. From the percentages of particles >250 μm diameter in each batch of fuel it can be seen in the table that these particles are concentrated in the upper part of the fuel in liner B, whereas in Liner A the particles are more evenly distributed throughout the fuel. If it is assumed that the capsule was suspended vertically by the flange through most of its life and knowing that Liner A was inverted in the capsule, it can be surmised that the >250 μm particles consisting mostly of zirconium dioxide which would be at the bottom of the fuel column in Liner A would tend to migrate up through the fuel because of its lower density. However, in Liner B the less dense filler which is on top of the fuel would remain near the top of the fuel column.

To replace FCA4 fuel lost during processing, batches of bonded fuel were selected. This fuel would be processed in the same manner as the FCA4 fuel before being used to load FCA8. For comparison one of the batches consisting of 253.8 g (isotope) of fuel was washed through the same screens as the FCA4 fuel to determine the fines distribution. The results are shown in Table 12. The total fines content amounted to 0.64% which is about one-third of that found in the FCA4 fuel. However, the size distribution is considerably different from that for the FCA4 fuel (Table 11). About 95% of the fines for the bonded fuel is in the 44-53 μm size range. The bonded fuel after fabrication had been in storage for 15-20 months before being processed. There is some evidence that the microspheres shrink with age. This characteristic plus possible fracturing caused by additional screening could account for the high percentage of particles in the 44-53 μm range.

After completing the analysis given in row one of Table 10 for the <37 μm wash, the remaining washes for each batch were combined and reduced to a volume of about 50 ml in 0.35N HNO_3 solution. The solution contained about 4 mg of plutonium as the oxide. The washes were sent to the Los Alamos Scientific Laboratory to obtain a distribution of the particle sizes less than 10 μm in size.

In reducing the volume of the solution at Mound, a quantity of white particulate matter was observed to form in the solution. This condition was reported to Los Alamos. Los Alamos agreed to try to determine the distribution of particle sizes in the solution in the presence of the precipitate rather than having Mound attempt to remove the

precipitate which might jeopardize the accuracy of the distribution data. The identity of the precipitate was not determined. At Los Alamos approximately one-half of the clear supernatant was carefully decanted off, and the remaining liquid was ultrasonically agitated to suspend the particles. A portion of the resulting slurry was spread on a clean microscope slide. The slurry was allowed to dry and the residue was used for an extraction replica. (Standard two-stage plastic/carbon replica with subsequent solution removal of the plastic leaving the residue suspended on a carbon film.) The replica was adversely affected by the presence of the unknown contaminant as seen in Figure 14. In the figure particles of PuO_2 of different sizes can be seen buried in the contaminant. The diffraction pattern in the figure shows the foreign material to be a dispersion of very fine crystallites.

Table 12

FINES ANALYSIS OF BONDED FUEL

<u>Size Range</u>	<u>Weight (g isotope)</u>	<u>Fines (wt %)</u>
<37 μm	0.043	2.65
37-44 μm	0.043	2.65
44-53 μm	<u>1.536</u>	<u>94.7</u>

1.622

Weight percent fines in batch = 0.64

Batch size = 253.8 g isotope

The remainder of the slurry was then diluted in water and agitated; the PuO_2 particles were allowed to settle; and the supernatant still containing the lighter, finer contaminant was partially withdrawn with a syringe. This separation step was repeated five times until the fines appeared to be thoroughly washed free of the contaminant. Presumably a considerable number of the smallest fines particles were lost during the separation procedure. Consequently, the weight distribution of particles in the less than 0.5 μm range was adversely affected. Therefore, all particles up to 0.3 μm in size were grouped together in Table 13. After the contaminant was removed from the slurry, it was possible to make replicas with good definition. Figures 15, 16, and 17 illustrate the shape and size of some of the particles. The particles were found to be PuO_2 by electron diffraction. A size-weight-fraction distribution for the particles are given in Table 13. The data are plotted in Figure 18. The distribution is based on the particles in the solution recovered during the washing of 1749 g of PuO_2 from capsule FCA4 on a 37 μm screen.

FIGURE 14 - $^{238}\text{PuO}_2$ particles from FCA4 fuel imbedded in
contaminant and corresponding diffraction pattern.
5000X

Table 13

SIZE - WEIGHT-FRACTION DISTRIBUTION OF PARTICLES IN <37 μm
 WASHINGS FROM 1728 g OF PuO_2 REMOVED FROM SNAP-27 FCA4^a

<u>Size (μm) (largest particle diameter)</u>	<u>Number</u>	<u>Weight-fraction</u>
up to 0.3	422	0.0036
" 0.4	45	0.0048
" 0.5	29	0.0061
" 0.6	28	0.0102
" 0.7	27	0.0156
" 0.8	14	0.0121
" 0.9	11	0.0135
" 1.0	5	0.0085
" 1.25	13	0.0428
" 1.5	11	0.0626
" 1.75	14	0.1266
" 2.0	10	0.1349
" 2.25	5	0.0961
" 2.5	7	0.1845
" 2.75	7	0.2455
" 3.0	6	0.2732
" 4.0	17	1.8351
" 5.0	12	2.5306
" 6.0	8	2.9145
" 7.0	10	5.7852
" 8.0	17	14.6805
" 9.0	7	8.6069
" 10.0 and over	37 ^b	62.4056

^a Both the low statistics and probable experimental loss of particles less than 0.3 μm make this distribution suspect.

^b Nine of these particles had all dimensions over 10 μm .

FIGURE 15 - $^{238}\text{PuO}_2$ particles taken from FCA4 fuel together with confirming diffraction pattern of large particle on the left.

FIGURE 16 - Typical large $^{238}\text{PuO}_2$ particles from FCA4 fuel.

FIGURE 17 - Typical smaller $^{238}\text{PuO}_2$ particles from FCA4 fuel.

FIGURE 18 - Particle number and weight fraction of particles as a function of particle diameter.

4. Helium Removal Studies Before the fuel form FCA4 was used to load FCA8, it was necessary to remove the bulk of the entrapped helium from the fuel. This step was necessary to comply with the requirement that the fuel contain no more helium than would be produced by the decay of the fuel over a six-month period. The method chosen to remove the helium was to bake the fuel in air at an elevated temperature. To determine whether the fuel would be adversely affected by the baking procedure, a sample of fuel from a SNAP-19 capsule was used for this study. The test sample would also demonstrate whether baking the fuel in air would remove the helium sufficiently. Previously, bakeout tests at Mound had been performed in vacuum.

The sample was obtained from SNAP-19 capsule #369/375, batch 2, and was screened to retain the 50 to 250 μm fraction. The 23-g sample was washed on a 50 μm screen with 250 ml of wash solution. The solution was discarded. The sample was again washed with 250 ml of solution which was retained for fines determination.

The sample was divided into three 6-g samples for the bakeout study and into two 2-g analytical samples. The stoichiometry, size, shape, density, fines content, and cation impurities of the fuel were determined from the analytical samples. The before bakeout data are reported in Table 14. Portions of the 6-g samples after being baked out were also taken and analyzed for the same attributes to determine what effects the baking out had on these characteristics.

The three bakeout samples each containing 5.7 g of fuel as oxide were identified as 19-27-1, 19-27-2, and 19-27-3. The first two were used for the air bakeout studies and the third one was used for vacuum bakeout. The vacuum sample was used in two ways. One portion was used to determine the total helium content retained by the sample, and another portion was treated in the same manner as the two air samples.

Previous work³ had demonstrated that vacuum bakeout involving fast thermal excursions to 1300°C effectively reduced the helium inventory of microspheres in 20 min at maximum temperature. However, the bakeout time and temperature for adequately reducing the helium content of microspheres baked in air was not known. Therefore, it was decided to bake both air samples at 1300°C with one being held for 1 hr and the other for 2 hr at temperature. Portions of these samples then would be baked in vacuum to remove any helium remaining to check the effectiveness of the air bakeout method.

The studies were initiated by taking 0.251 g of the vacuum sample 19-27-3 to determine the helium inventory for all three samples. The sample was placed in the helium release experimental setup and heated to approximately 1300°C in vacuum in less than 6 min. The sample was

³ P. Angelini, et al., Helium Release from $^{238}\text{PuO}_2$ Microspheres, ORNL-4507, Oak Ridge National Laboratory (March 1970).

Table 14

ANALYSIS OF SNAP-19 FUEL SAMPLES
FROM BAKEOUT TESTS

Cation Impurities	Amount Before Bakeout (ppm)	Amount After Bakeout (ppm)		
		1 hr air	2 hr air	vacuum
Al	100	50	50	200
Ba	150	< 50	ND	ND
Ca	350	<250	250	250
Co	ND	ND	ND	ND
Cr	400	50	600	130
Cu	50	50	< 50	50
Fe	600	>1000 <3000	>1000	>1000
Mg	80	< 50	< 50	70
Mn	<50	50	< 50	< 50
Na	ND	ND	ND	ND
Ni	250	< 50	50	< 50
Si	750	900	>1000	>1000
Zn	ND	ND	50	50
Total ppm	2780	5500	3150	2850
Total Percent	0.28	0.55	0.32	0.29
Stoichiometry	--	PuO ₂	PuO ₂	PuO ₂
Size & Shape	OK	OK	OK	OK
App. Density (g/cc)	9.71	--	10.6	10.2
Lattice A	5.3974	5.4095	5.4119	5.4069
Fines (wt %)	1.8×10^{-3}	7.0×10^{-3}	6.5×10^{-3}	3.3×10^{-3}

maintained at this temperature until a decrease in the helium release rate was noted, then further heated to approximately 1950°C to drive out any residual helium that could have been retained in the sample. The sample was cooled and taken off test when the helium release rate had decreased to the background rate of the measuring equipment. The results of this experiment are shown in Table 15.

Table 15

VOLUME OF HELIUM REMOVED BY BAKEOUT
IN AIR AND VACUUM AT 1300°C

<u>Sample</u>	<u>Helium Removed (cc/g)</u>	<u>Helium Removed (%)</u>
19-27-1	0.1231	83
19-27-2	0.1462	98.5
19-27-3	0.1484	100

The remaining 5.449 g of sample were heated in a vacuum furnace using the following thermal profile: 5 min from ambient temperature to 1300°C; 20 min at 1300°C; manual cool down to ambient temperature. The sample was then analyzed for the various attributes noted above. The results are reported under the vacuum column in Table 14.

Next the entire 19-27-1 sample was baked out in air. First the furnace was allowed to heat to and stabilize at 700°C. The sample in a high-fired Al_2O_3 crucible was placed in the furnace and the furnace temperature was increased to 1300°C in 45 min. The furnace was maintained at 1300°C for 1 hr. The sample was then removed and allowed to air-cool.

Approximately 0.533 g of this sample was later heated in vacuum at 1950°C to determine the amount of helium removed. The results are shown in Table 15. The balance of the sample was washed and analyzed. The results are listed in the column 1-hr air in Table 14.

Now the 19-27-2 sample was baked out in the same manner as sample 19-27-1 with the exception that this sample was removed from the furnace after 2 hr at 1300°C and air-cooled.

Approximately 0.185 g of this sample was used to determine the helium removed while the remaining portion was washed and used for analysis. The data are given in the column 2-hr air in Table 14. The amount of helium baked out is shown in Table 15.

The volumes are corrected for the time between bakeout at 1300°C and the residual helium determination. It was assumed that all the helium generated during this interval (6.5 days) was retained by the fuel.

At the time that the samples were taken the fuel had been in FCA4 about 2.25 yr. If the fuel had accumulated a six-month inventory of helium prior to being placed in the capsule then the fuel would have a total helium inventory of 2.75 yr assuming that all the helium was entrapped in the fuel. Calculations using the factor 1.67×10^{-8} cc He/g/sec, which is the generation rate for helium in microspheres, showed that 1.459 cc He/g would accumulate in 2.75 yr. If a 2-hr air bakeout reduces the helium to 1.45% of the original amount, then only 0.0211 cc He/g would remain which is equivalent to the helium generated in 1 g of microspheres in about 15 days. This value is well below the maximum helium inventory of six months allowed in the fuel at time of encapsulation. Of course, the release data were obtained from 5-g samples. If the bakeout parameters are as effective in treating large quantities of fuel, then the bakeout at 1300°C in air for 2 hr should remove an adequate quantity of helium to qualify the fuel for encapsulation in FCA8.

A review of the data in Table 14 shows that the attributes of the fuel were not significantly changed by the bakeout procedure. There was some increase in the iron impurity which may have been introduced during the baking. Nevertheless, the total cation impurity content of the samples were, as a maximum, about 0.5% which is appreciably less than the 2% maximum allowed for the fuel. The fines content for the samples after bakeout were higher. However, such additional fines as may be formed by baking can be removed by additional washing of the fuel.

With the conclusion of the bakeout study the removal of the helium from the FCA4 fuel was begun. A composite sample of the fuel from Liner A was analyzed to determine its characteristics before bakeout. The data are given in Table 16. The table also includes the original analysis of the fuel when it was placed in FCA4 some two years ago. The size of the fuel batches baked out were 400-600 g oxide. All batches were held at 1300°C in air. The first two of the 10 batches were heated for 4 hr, the others for 6 hr.

To determine whether the bakeout parameters were as effective in removing helium from large batches of fuel as from small ones, representative samples were taken from three of the heated batches and tested to obtain the residual helium value. For the test 0.25-g samples were held at 2000°C in vacuum. The amount of helium in each sample in terms of days required to generate the volume of helium found was 13, 66, and 24 days. Because the accuracy of the test method was estimated to be ± 20 days, the above values appear to agree with each other within the limits of experimental error. The results indicate that the bakeout parameters are effective in removing the helium from the fuel. They also show that the residual helium is well below the maximum allowable limit of 180 days.

A sample from one of the batches was analyzed to determine the fuel characteristics after bakeout. The values are given in the last column of Table 16.

Table 16

ANALYSIS OF SNAP-27 FCA4 FUEL
BEFORE AND AFTER BAKEOUT

Cation Impurities	Original Analysis (ppm)		Liner A (ppm)	
	Liner A	Liner B	Before Bakeout	After Bakeout ^a
Al	1200	500	200	>1000
B	100	400	ND	ND
Ba	ND	ND	100	ND
Ca	700	500	< 250	500
Co	300	ND	1000	>1000
Cr	600	400	780	800
Cu	300	ND	50	100
Fe	3400	4100	>1000 <3000	>1000 <3000
Mg	100	200	50	750
Mn	ND	ND	< 50	250
Na	ND	ND	ND	ND
Ni	600	300	80	350
Pb	400	ND	ND	ND
Si	2600	2100	>1000 <3000	>1000
Ti	100	ND	ND	ND
Zn	<u>100</u>	<u>100</u>	<u>ND</u>	<u>ND</u>
Total ppm	10500	8600	8560	8750
Total Percent	1.05	0.86	0.86	0.88
Stoichiometry	PuO ₂	PuO ₂		PuO ₂
Size & Shape	OK	OK		OK
App. Density (g/cc)	9.82- 10.45	9.61- 9.82	10.0	9.5
Lattice Å				5.4070

^a Sample from 535 g oxide.

Following the bakeout work the fuel was screened, segregated, and washed to comply with the SNAP-27 fuel specifications. The fuel was assembled into batches for calorimetry, and the thermal power was measured in preparation for loading the fuel in FCA8. To replace FCA4 fuel lost during processing sufficient fuel was removed from bonded storage and added to that of FCA4 to provide the required amount of fuel for FCA-8. The bonded fuel was also baked out, screened, segregated, washed, and calorimetered. The characteristics of the fuel prepared for FCA8 are given in Table 17.

In processing the fuel for FCA8, including the bonded fuel, the amount of fuel which could not be accounted for amounted to only 30.5 g (isotope) out of a total weight of 3726.3 g (isotope) handled. The percent loss then is 0.82. As the calorimetric method used for determining the quantities of fuel is estimated to have an accuracy of $\pm 0.6\%$ then the real loss could be even less than the 30.5 g indicated. The low loss attests to the careful handling practiced during the processing of the fuel.

Finally the amount of fuel from FCA4 which remained after all the processing and which qualified for use in FCA8 was 1176.11 W (th) as of March 1971.

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Table 17

ANALYSIS OF SNAP-27 FCA4 FUEL USED IN FCA8

<u>Cation Impurities</u>	<u>Analysis (ppm)</u>		
	<u>Lot A</u>	<u>Lot B</u>	<u>Lot C</u>
Ag	<< 50	ND	ND
Al	400	50	50
B	ND	ND	ND
Ba	70	< 50	ND
Be	<< 50	ND	ND
Ca	440	< 250	< 250
Co	1000	ND	ND
Cr	340	100	150
Cu	50	50	< 50
Fe	>1000 <3000	950	690
Mg	250	50	< 50
Mn	100	< 50	< 50
Na	ND	ND	ND
Ni	170	80	50
Pb	< 50	ND	ND
Si	>1000 <3000	500	< 50
Ti	500	ND	ND
Zn	ND	ND	180
Total ppm	9470	2130	1570
Total percent	0.95	0.21	0.16
Size & Shape	OK	OK	OK
App. Density (g/cc)	9.6	9.5	10.3
Fines (wt %)	5.0×10^{-4} 2.8×10^{-4} 5.2×10^{-4} 5.7×10^{-4}	6.9×10^{-5} 5.0×10^{-5} 3.8×10^{-5} 8.9×10^{-5}	5.0×10^{-4} 6.0×10^{-4} 4.5×10^{-4}

APPENDIX A

OPERATION OF HELIUM RELEASE MEASURING APPARATUS (FIGURE 7)

I. Check-out of Vacuum System

- A. Close valves 5, 9, and 13 - all other valves are open.
- B. Balance MKS sensor.
- C. Close valve 3 and main valve 1 and determine leak rate of system.
- D. Repeat step C with drill rotating.
- E. When satisfactory leak rate has been obtained, close valves 6 and 7.

II. Determine Volume of System

- F. Pressurize system to 10-20 torr by opening valve 5. Record stabilized pressure P_1 .
- G. Open standard volume valve 6. Record stabilized pressure P_2 .
- H. Calculate system volume V_1

$$\frac{P_1}{P_2} = \frac{V_1 + V_{std}}{V_1}$$

III. Helium Measurement

- I. Close valve 5. All other valves are open to the vacuum pumping system.
- J. Balance MKS sensor.
- K. Close valves 3, 6, 9, and the main valve 1.
- L. Drill open capsule. After opening, locate the drill shaft at the same position used in standardizing the system volume.
- M. Record the stabilized pressure P_3 (mm Hg).
- N. Calculate volume of gas in capsule $V_g = \frac{P_3}{760} V$

This assumes the temperature to be uniform within the system. For a capsule the size of the SNAP-27 this is not true and some error will be introduced by this method.