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WANL-TME-1859
23 September 1968

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Westinghouse Astronuclear Laboratory

FINAL REPORT

WFDD - TS - 3005

PERMEABILITY STUDIES

(Title Unclassified)

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Westinghouse Astronuclear Laboratory

FINAL REPORT

WFDD - TS - 3005

PERMEABILITY STUDIES

(Title Unclassified)

Classification cancelled ~~(or changed to~~ ______
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ABSTRACT

(CRD) This experiment was designed to test methods for lowering the leak rate of high-loaded elements. Three different methods were tried: 1) processing variations such as extended green mix state holds, 2) cure atmosphere variations, and 3) matrix particle size variations. All three methods were effective to various degrees in lowering the leak rate of NERVA fuel elements.

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1.0 INTRODUCTION (U)

(CRD) Element permeability, or body leak rate, is used by Matrix Engineering as a parameter in pre-corrosion or reactor test evaluation of fuel elements. Basically, this test involves pressurizing the bores of NERVA fuel elements with either air or helium at 10 psig and measuring the rate of gas release through the element webs. The test is somewhat inaccurate in that it measures release from the outer webs, and the measurement tends to vary with operator and equipment, but it does provide an indication of the integrity or permeability of the matrix.

(CRD) It is known that the leak rate of a NERVA fuel element tends to increase with increased loading of pyrocoated uranium carbide fuel beads (Figure 1). Also, there is a design limitation of 170 cc/min leak rate of air from the matrix. The maximum limit was established as a result of the post-operational evaluations of the NRX-A4 and NRX-A5 reactors and the NRX-A6 development programs which indicated that elements with high leak rates tended to exhibit excessive hydrogen corrosion weight losses.

(CRD) The design loadings of the PEWEE II reactor call for fuel loadings in excess of 550 mg U/cc element. An extrapolation of Figure 1 indicated that elements made with these loadings would, on the average, exceed the maximum allowable leak rate, causing an excessive ratio of extruded to deliverable fuel elements.

(U) Because of these data, it was felt that some work must be done to modify the matrix or fabrication processes and, thereby, reduce the element leak rate. This report summarizes the data obtained from that work.

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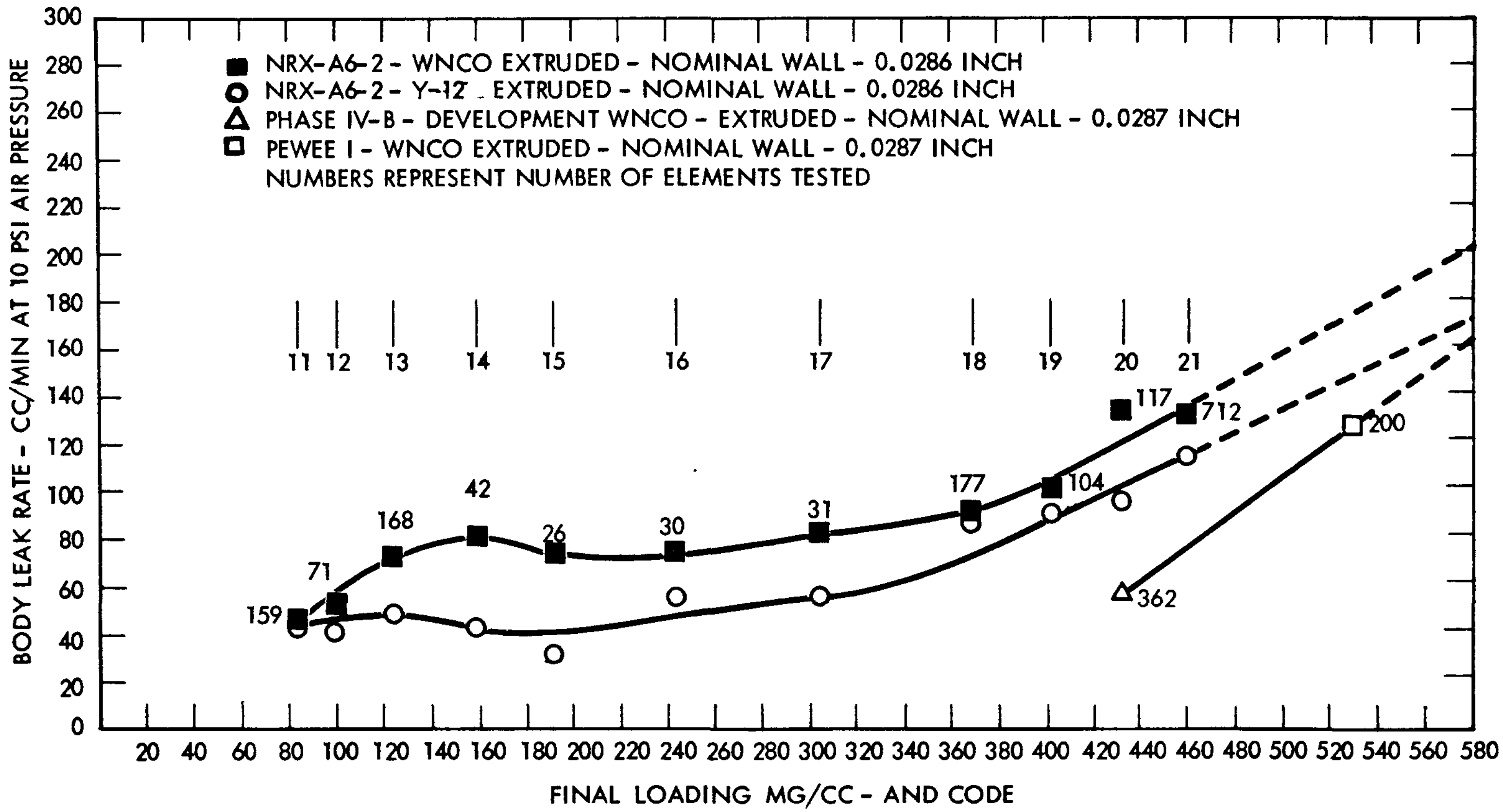

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Figure 1.(CRD)Body Leak Rate Versus Element Loading (U)

2.0 EXPERIMENTAL APPROACH (U)

(CRD) The desired procedure would have been to fabricate elements with loadings of 550 mg U/cc and check the validity of the extrapolation mentioned in Section 1.0. However, a shortage of time dictated that the extrapolation be postulated and that it was necessary to proceed on to the next step, i.e., determining matrix or fabrication changes which would result in lower leak rates. Standard elements were made at the same time both to check the validity of the assumption and to act as control elements.

(CRD) Three methods were proposed for lowering leak rates. All of these methods had some related experimental background to substantiate their validity. First, both Union Carbide at Y-12 and the Los Alamos Scientific Laboratory were successful in lowering the leak rate of central support elements by holding the mix in the green state for extended time periods before extrusion. Second, WANL was able to lower the permeability of fuel element shapes by curing the extruded stock under flowing argon, rather than the standard air purged cycle. Finally, WANL had achieved encouraging results toward lowering element leak rates by varying the particle size distribution of the graphite flour.

(CRD) The experiment was designed to test all three methods. Nine different types of extrusion batches were planned. They were as follows:

1) Extrude two batches after holding the mixes in the green state for 24 hours at 38°C, with normal processing to be used otherwise.

2) Extrude two batches after holding the mixes in the green state for 48 hours at room temperature, with normal processing to be used otherwise.

3) Extrude two batches after holding the mixes in the green state for 48 hours at 38°C with normal processing to be used otherwise.

4) Extrude two batches by normal procedure, cure under flowing argon, with normal processing to be used otherwise.

5) Extrude two batches, using graphite flour with a modified particle size distribution and standard processing. (See Figure 2 for specific size fractions.)

6) Extrude two batches, using a different modified flour distribution and standard processing. (See Figure 2 for specific size fractions.)

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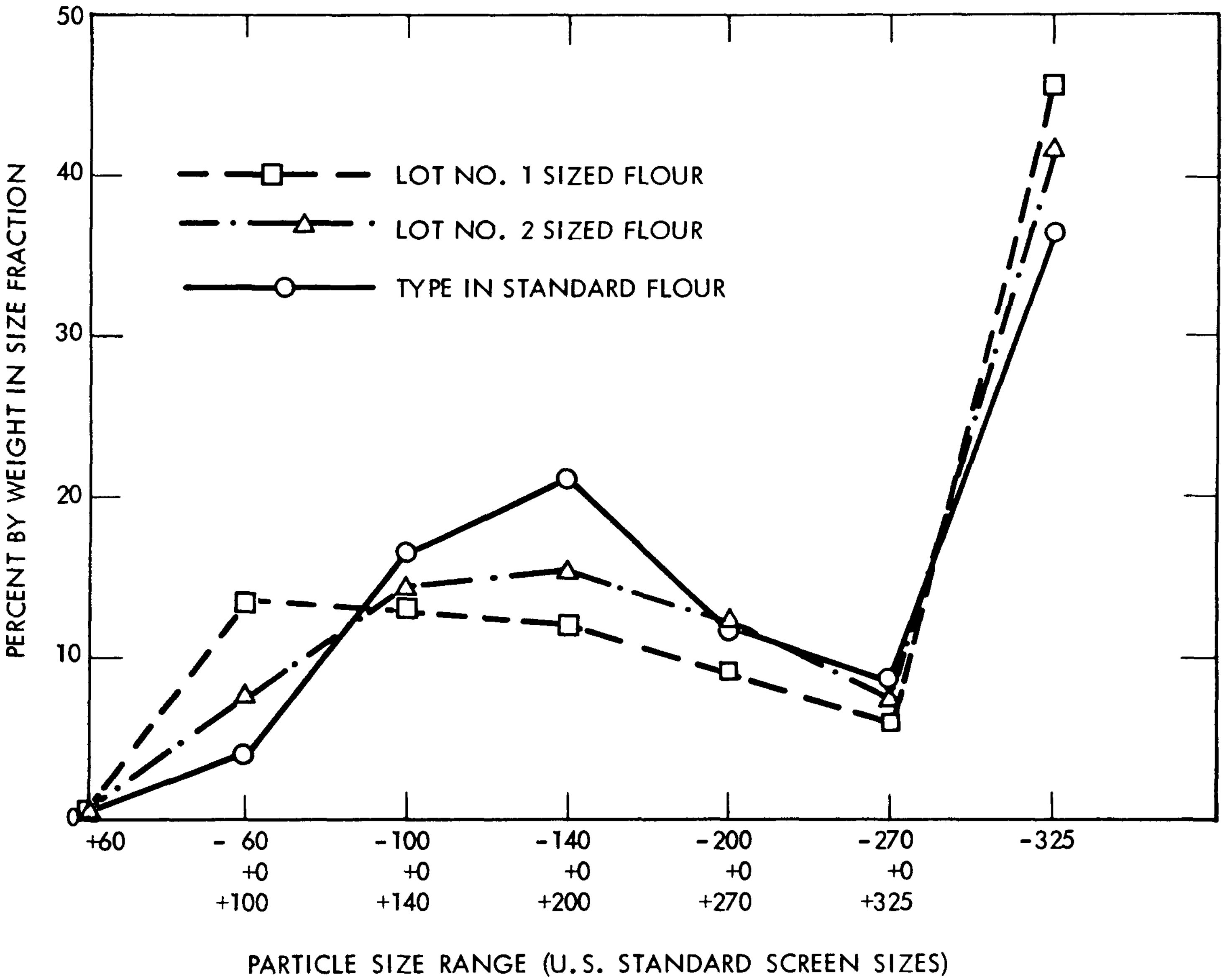

Figure 2. (CRD) Particle Size Analysis of TS-3005 Flour (U)

[redacted]

7) Extrude two batches using 18 percent thermax and standard processing.

8) Extrude two batches using 21 percent thermax and standard processing.

9) Extrude two standard batches to act as control.

All loadings to be 600 mg U/cc aim. All element cooling channels to be reamed to 0.102 $^{+001}_{-000}$ inch diameter.

3.0 EXPERIMENTAL PROCEDURE (U)

3.1 RAW MATERIALS (U)

(CRD) All starting materials used were standard. Fuel particles were typical General Atomic pyrocoated uranium carbide beads. Standard thermax and Great Lakes 89772 lot No. 2 flour were used. The binder was standard varcum. The binder catalyst was maleic anhydride and was production standard. The two lots of sized flour were screened at WEFF from standard 89772-2 flour.

3.2 MIXING (U)

(CRD) Varcum and DCI preparation were accomplished at WNCO. Mixing and blending were accomplished by standard WNCO procedures except on the four raw material variation batches. On these four batches, the raw materials were added to the Vee blender separately. Mixing time, binder addition time, binder addition pressure, and the 8-quart Patterson-Kelley Vee blender were all standard.

(CRD) After mixing, the six delayed extrusion batches were dropped into loosely-closed plastic bags and either heated to 38°C or held in open storage areas until the designated time had passed. Holding at 38°C was accomplished in the WNCO curing ovens because of safety restrictions.

(CRD) Two passes were made through the chopper immediately before extrusion. All processing from this point forward was standard.

3.3 EXTRUSION (U)

(CRD) Extrusion was accomplished at WNCO on April 10-11, 1968. Extrusion conditions were normal. The standard three-cycle extrusion process was used. An attempt was made to hold the extrusion rate constant at about 100 in./min while the tonnage was allowed to vary.

(U) Each pair of batches was extruded together, followed by the tailings for that group. The extrusions were considered normal. The extrusion parameters are presented in Table 1.

3.4 CURE (U)

(CRD) Curing was accomplished in the WNCO curing ovens, using the standard thermal cycle and air flow. The two argon cured batches were placed in the

[redacted]

TABLE 1 (CRD)

EXTRUSION SHEET (U)

Batch	Serial Numbers	Chop Temp. 1 °C 2	Force (Tons)	Rate (In/Min)	Chop Temp. (°C)	Force (Tons)	Rate (In/Min)	Chop Temp. (°C)	Force (Tons)	Rate (In/Min)	Remarks
5706	550-1046114 to 46117	32/45	100	100	34	99	100	37	96	98	Standard Formulation, Cured Under Argon
5707	46118 to 46122	33/37	105	100	37	104	98	38	101	94	Standard Formulation, Cured Under Argon
5707T	46123 to 46124	- - -	- - -	- - -	- - -	- - -	- - -	33	108	100	Tailings Cured Under Argon
5708	46136 to 46140	34/41	115	100	41	108	106	43	109	100	Standard Formulation and Processing
5709	46161 to 46165	33/36	113	100	43	106	98	43	108	98	Standard Formulation and Processing
5710	46188 to 46192	38/42	91	100	39	91	106	42	95	103	Lot #1 Specially Blended Flour
5711	46193 to 46197	37/43	95	100	39	94	103	42	95	103	Lot #1 Specially Blended Flour
5711T	46198	- - -	- - -	- - -	- - -	- - -	- - -	34	108	100	Tailings From Batches 5710 and 5711
5712	46199 to 46202	38/42	99	98	40	95	103	40	97	103	Lot #2 Specially Blended Flour
5713	46203 to 46207	31/42	98	98	41	93	106	43	94	106	Lot #2 Specially Blended Flour
5713T	46208 to 46209	- - -	- - -	- - -	- - -	- - -	- - -	32	103	106	Tailings From Batches 5712 and 5713
5714	46141 to 46145	44/47	117	106	42	119	106	45	123	100	48 Hour Hold at 38°C

[redacted]

TABLE 1 (CONTINUED) (CRD)

Batch	Serial Numbers	Chop Temp. 1 °C 2	Force (Tons)	Rate (In/Min)	Chop Temp. (°C)	Force (Tons)	Rate (In/Min)	Chop Temp. (°C)	Force (Tons)	Rate (In/Min)	Remarks
5715	46146 to 46150	35/45	135	85	43	134	106	46	136	100	48 Hour Hold at 38°C
5715T	46151	- - -	- - -	- - -	- - -	- - -	- - -	38	138	110	Tailings From Batches 5714 and 5715
5716	46152 to 46155	33/39	CRACKS		43	103	106	43	105	106	48 Hour Hold at Room Temperature
5717	46156 to 46160	34/38	105	107	41	104	106	40	106	103	48 Hour Hold at Room Temperature
5718	46125 to 46129	33/37	105	98	41	100	109	43	100	100	24 Hour Hold at 38°C
5719	46130 to 46134	42/43	103	103	44	101	106	45	104	100	24 Hour Hold at 38°C
5719T	46135	- - -	- - -	- - -	- - -	- - -	- - -		115	100	Tailings From Batches 5118 and 5119
5720	46166 to 46169	31/36	90	100	35	89	103	38	98	100	82/18 Ratio of Flour To Thermax
5721	46170 to 46174	35/38	97	100	39	98	103	39	100	103	82/18 Ratio of Flour To Thermax
5721T	46175 to 46176	- - -	- - -	- - -	- - -	- - -	- - -	34	105	98	Tailings From Batches 5720 and 5721
5722	46177 to 46180	34/40	99	100	37	103	103	40	110	98	79/21 Ratio of Flour To Thermax
5723	46181 to 46185	33/39	107	98	37	105	100	38	112	100	79/21 Ratio of Flour To Thermax
5723T	46186 to 46187	- - -	- - -	- - -	- - -	- - -	- - -	36	130	103	Tailings From Batches 4722 and 5723

loosely sealed WEFF curing box and the box was then placed in the oven. Argon flow in the coffin was set at approximately 3 ft^3/min for the first half-hour to purge the coffin. The flow was then lowered to approximately 1 ft^3/min for the rest of the run. The air cured elements were placed in the curing ovens with their ends wrapped in foil. No unusual occurrence was noted during the runs.

3.5 BAKE (U)

(CRD) All elements were baked at WNCO, using standard WNCO bake cycles and conditions. The thermal cycles were as follows:

1) Rapid heat to 250°C under argon.
2) 250°C to 610°C under argon at a ramp of 10°C/hr.
3) 610°C to 850°C under argon at a ramp of 25°C/hr.

The bake runs were considered normal.

3.6 GRAPHITIZATION (U)

(CRD) Eighty-eight of the elements were graphitized to the standard WNCO conditions which are ramp to 2200 $\pm$ 25°C with a 3-hour hold at temperature under flowing argon.

(CRD) Four elements were graphitized at 2300°C for 1 hour and four were graphitized at 2300°C for 2 hours. The two- and four-element runs will be discussed later in Section 4.0. The standard graphitization runs were considered normal.

3.7 TIPPING (CRD)

(CRD) Only even-numbered elements were tipped. It was considered expedient to process half of the elements ahead to get data quickly while the remaining elements were held for other studies or for supplemental work. Therefore, the odd-numbered elements were not tipped. Subsequently, eighteen of the even-numbered elements were tipped with PEWEE I, composite carbide tips and twenty-four even-numbered elements received non-fuel tips. Tipping was accomplished by the standard WNCO molybdenum brazing technique and was considered normal.

3.8 MACHINING (U)

(CRD) Machining was accomplished at WNCO. The elements were reamed to $0.103^{+0.000}_{-0.001}$. Some difficulty was encountered in reaming due to the size of the as-graphitized bores. The elements had been extruded using intermediate diameter pins (0.104 inch diameter), and the thermal shrinkage was less than anticipated. However, since this problem was encountered on all elements, it was felt that this would normalize the final results. Across-flats machining was accomplished on the WNCO diamond mill without incident.

3.9 LEACHING (U)

(CRD) Leaching was accomplished at 2100°C. Two different types of leaching cycles were used. The odd-numbered elements were leached using the following cycle:

1) Rapid heat to 1700°C under flowing argon.
2) Leach gases turned on at 1700°C, heating continued to 2100°C.
3) Hold at 2100°C for 1 hour with leach gases flowing.
4) Rapid cool to 1700°C under leach gases.
5) Hold at 1700°C for 1 hour under leach gases.
6) Cool under argon to room temperature.

(CRD) Leach gases and gas ratios were standard. The even-numbered elements were leached in a 17-hour, 2100°C cycle. These two cycles are displayed graphically in Figures 3 and 4. The elements, matrix-type, leach run, and weight loss are listed in Table 2.

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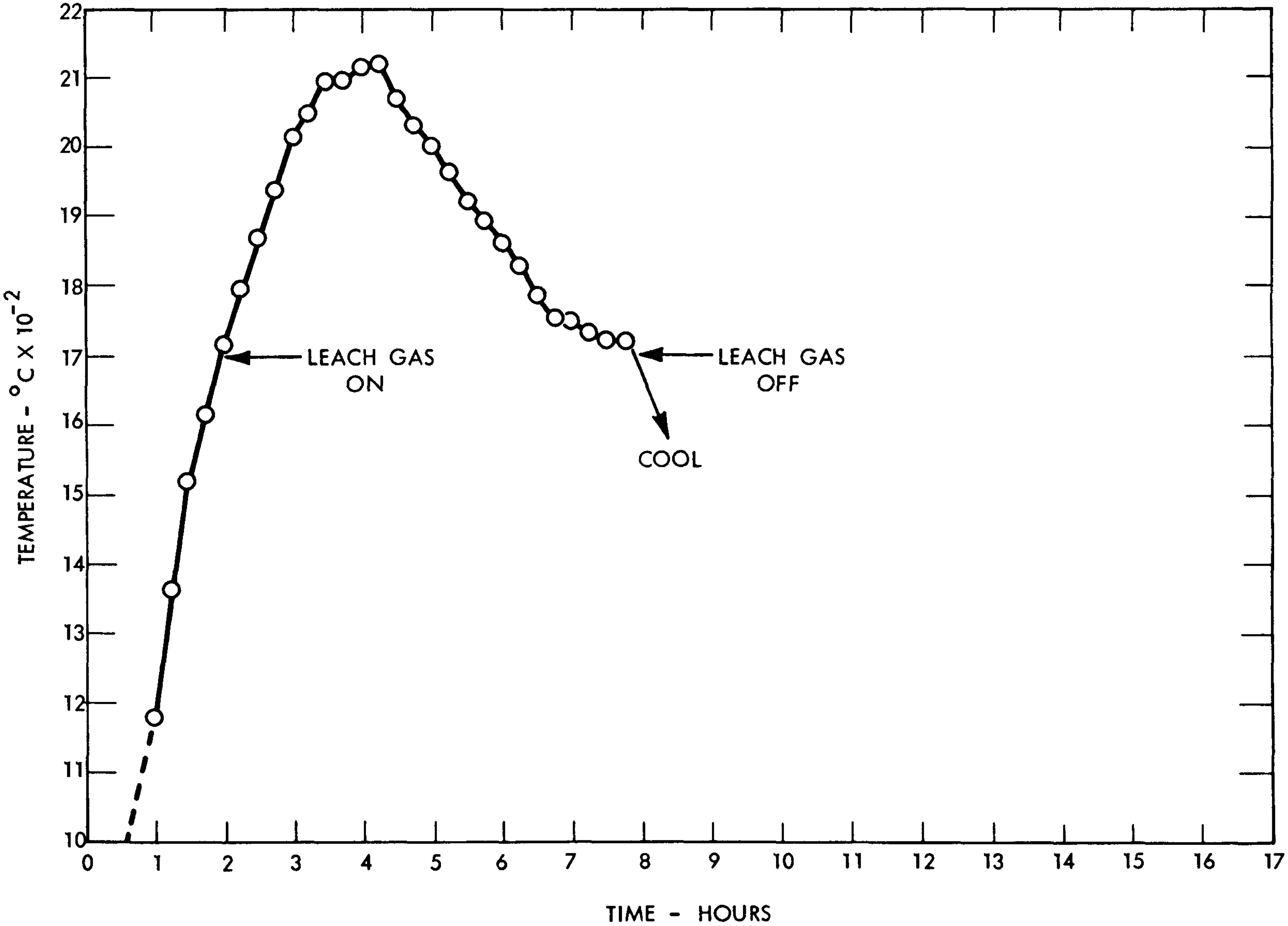

Figure 3. (CRD) Time-Temperature Profile of Leach Run S-133 TS-3005 (U)

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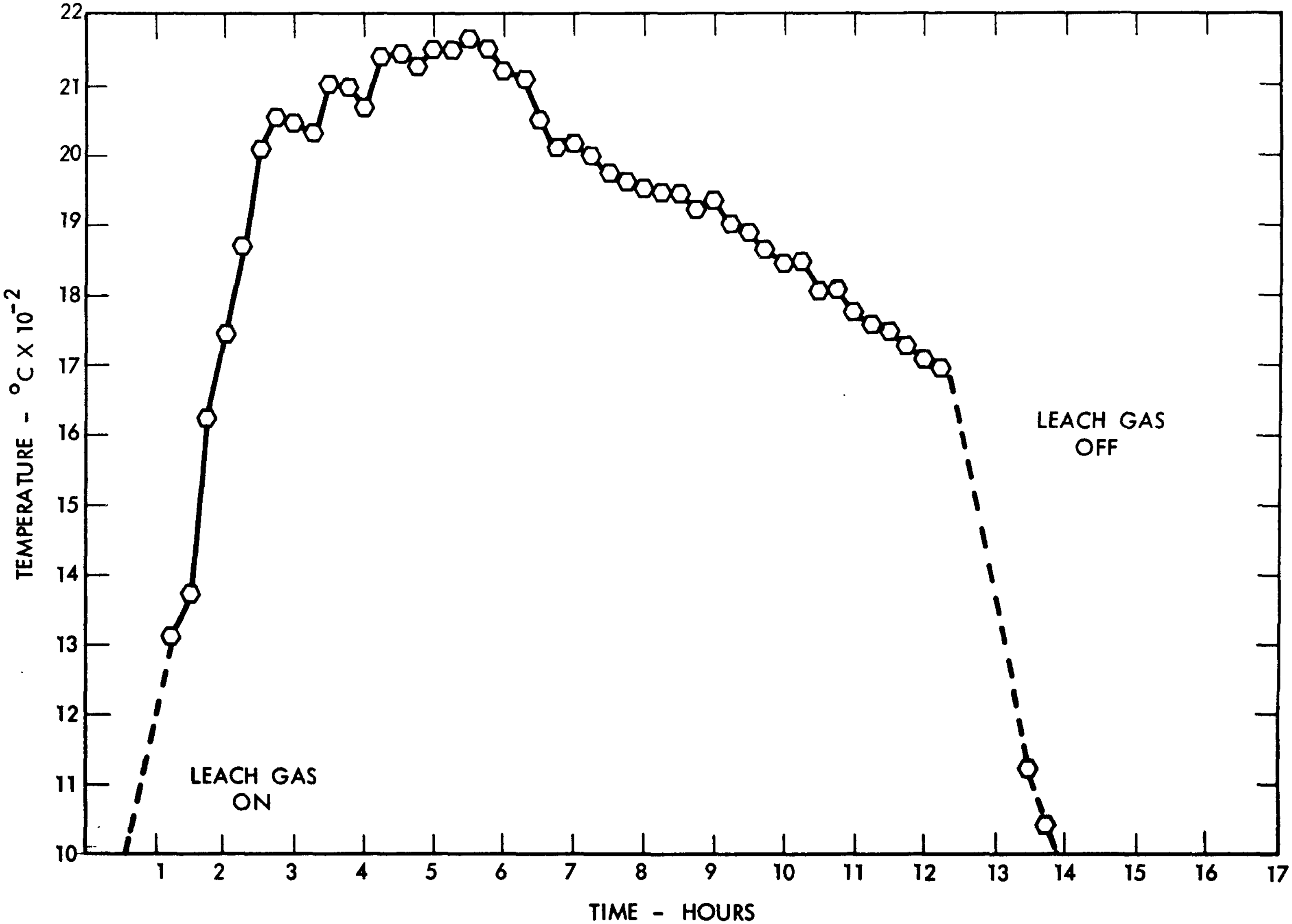

Figure 4.(CRD) Time-Temperature Profile of Leach Run D-617 TS-3005 (U)

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TABLE 2 (CRD)

LEACH WEIGHT LOSSES OF TS-3005 ELEMENTS (U)

S/N	Matrix Type	Leach Run	Weight Loss (g)
550-1046114	Standard, Argon Cure	- - -	- - -
550-1046115		- - -	- - -
550-1046116		- - -	- - -
550-1046117		S-133	14.5
550-1046118		- - -	- - -
550-1046119		S-133	11.6
550-1046120		D-615,D-616	15.0
550-1046121		- - -	- - -
550-1046122		D-617	14.3
550-1046123		S-133	12.5
550-1046124	Standard, Argon Cure	D-617	15.2
550-1046125	Standard, 24 Hr. Hold at 38°C	S-133	11.0
550-1046126		D-617	13.4
550-1046127		S-133	13.0
550-1046128		D-615,D-616	13.8
550-1046129		- - -	- - -
550-1046130		D-617	14.2
550-1046131		S-133	11.2
550-1046132		D-615,D-616	14.6
550-1046133		S-133	10.8
550-1046134		D-617	13.9
550-1046135	Standard, 24 Hr. Hold at 38°C	S-133	12.0
550-1046136	Standard	D-615,D-616	14.8
550-1046137		- - -	- - -
550-1046138		D-615,D-616	12.5
550-1046139		S-133	10.8
550-1046140	Standard	D-617	14.0
550-1046141	Standard, 48 Hr. Hold at 38°C	S-133	11.1
550-1046142		D-615,D1616	14.0
550-1046143		S-133	12.0
550-1046145		- - -	- - -
550-1046146		D-617	13.2
550-1046147		S-133	11.5
550-1046148		D-615,D-616	14.4
550-1046149		S-133	11.7
550-1046150		D-617	14.5
550-1046151	Standard, 48 Hr. Hold at 38°C	S-133	12.0

TABLE 2 (CONTINUED) (CRD)

S/N	Matrix Type	Leach Run	Weight Loss (g)
550-1046152	Standard, 48 Hr. Hold at R. T.*	D-617	14.1
550-1046153		S-133	11.8
550-1046154		D-615, D-616	13.4
550-1046155		S-133	11.2
550-1046156		D-617	14.1
550-1046157		- - -	- - -
550-1046158		D-615, D-616	14.3
550-1046159		S-133	12.0
550-1046160	Standard, 48 Hr. Hold at R. T.*	D-617	13.9
550-1046161	Standard	S-133	11.2
550-1046162		D-615, D-616	14.5
550-1046163		S-133	11.6
550-1046164		- - -	- - -
550-1046165	Standard	- - -	- - -
550-1046166	18% Thermax	D-617	14.3
550-1046167		- - -	- - -
550-1046168		D-615, D-616	14.6
550-1046169		- - -	- - -
550-1046170		D-617	14.7
550-1046171		S-133	11.3
550-1046172		D-615, D-616	14.6
550-1046173		S-133	12.1
550-1046174		D-617	14.7
550-1046175		S-133	12.3
550-1046176	18% Thermax	D-617	14.7
550-1046177	21% Thermax	S-133	12.9
550-1046178		D-615, D-616	14.7
550-1046179		- - -	- - -
550-1046180		D-617	14.9
550-1046181		S-133	12.5
550-1046182		D-615, D-616	14.9
550-1046183		- - -	- - -
550-1046184		D-617	14.8
550-1046185		S-133	11.2
550-1046186		D-617	14.9
550-1046187	21% Thermax	S-133	11.6

* R. T. - Room Temperature

TABLE 2 (CONTINUED) (CRD)

S/N	Matrix Type	Leach Run	Weight Loss (g)
550-1046188	Lot #1, Sized Flour	D-617	12.5
550-1046189		- - -	- - -
550-1046190		D-615, D-616	13.4
550-1046191		S-133	11.8
550-1046192		D-617	12.8
550-1046193		S-133	13.7
550-1046194		D-615, D-616	16.6
550-1046195		S-133	13.3
550-1046196		D-617	16.2
550-1046197		S-133	13.1
550-1046198	Lot #1, Sized Flour	D-617	14.4
550-1046199	Lot #2, Sized Flour	S-133	15.0
550-1046200		D-615, D-616	16.8
550-1046201		- - -	- - -
550-1046202		D-617	16.9
550-1046203		S-133	11.0
550-1046204		D-617	16.8
550-1046205		S-133	13.1
550-1046206		D-615, D-616	17.0
550-1046207		S-133	14.8
550-1046208		D-617	16.3
550-1046209	Lot #2, Sized Flour	S-133	12.9

4.0 DATA AND RESULTS (U)

4.1 PHYSICAL PROPERTIES (U)

(U) Resistivity, flexure strength, compressive strength, and longitudinal coefficient of thermal expansion were measured on each type of matrix. The data obtained are presented in Table 3. Since these measurements are somewhat statistical in nature, the small sample size cannot be considered conclusive. However, a few trends can be noted. First, the high thermax elements tend to be stronger and have higher resistivities than the standard elements. The CTE's tend to be about equal.

4.2 ANALYSIS OF HIGH TEMPERATURE GRAPH RUNS (U)

(CRD) Two graph runs, EA-49 and E-1068, were made at WNCO using a 2300^{o}C graphitizing temperature. EA-49 was held at 2300^{o}C for 2 hours while E-1068 was held at 2300^{o}C for 1 hour. These elements were sectioned for metallographic and radiographic bead damage. The results indicate that extensive bead damage (8 to 9 percent totally migrated) will result with both time cycles. Sister elements run in standard WNCO cycles, i.e., 2200^{o}C for 3 hours, showed less than 1 percent migration. Migration was uniform along the element. It appears that the temperature control is somewhat critical at these temperatures and beyond the ability of the present control equipment. This 2300^{o}C cycle has subsequently been dropped from the WEFF procedures.

4.3 EXTRUSION PARAMETERS (U)

(CRD) An attempt was made to control extrusion rate to 100 ± 10 in./min. In order to do this and maintain somewhat equivalent extrusion forces, it was necessary to adjust binder levels in several of the extrusion batches. Regular thermax was used in all batches. Since this material is oily ($\sim$1% by weight benzene-type oil), it acts as an extrusion lubricant. With the four high thermax batches it was necessary to drop the binder level to 26 pph with the 18 percent thermax batch and to 25 pph with the 21 percent thermax batch. The batches that were held in the green state required 28 pph binder due to either an absorption of the binder into the dry ingredients or to an evaporation of the volatiles. Even with this correction, the 48-hour hold at 38^{o}C batches required $\sim$30 percent

TABLE 3 (CRD)

PHYSICAL PROPERTIES OF TS-3005 ELEMENTS (U)

Matrix Type	Flexural Strength (lb/SN^2)	Compressive Strength (lb/in.^{2})	CTE % Elong. @2000°C	Resistivity (milliohm/in.)
Standard	4210 3940	9,570	0.74 0.715	1.91
Standard With Argon Cure	4330 4550	10,950	0.69 0.76	1.88
24 Hr. Hold at 38°C	4610 4130	11,630	0.72	1.91
48 Hr. Hold at 38°C	4170 4080	11,150	0.72	1.87
48 Hr. Hold at R.T.*	4210 4510	10,980	0.715	1.88
18% Thermax	4550 4430 4290 4360	11,450 11,490	0.70 0.70	2.01
21% Thermax	4330 4240	11,430	0.73	2.11
Lot #1, Sized Flour	4560 4160	11,820	0.72	1.95
Lot #2, Sized Flour	4120 4170	10,380	0.73	1.91

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* R.T. - Room Temperature

higher pressures for extrusion. The parameters for the three passes on each batch are presented in Table 1.

4.4 LEACHING (U)

(CRD) The TS-3005 elements were leached in one of three leach runs: S-133, D-615-616, or D-617. The elements, their respective leach runs, and weight losses are presented in Table 2. Run S-133 was experimental using standard gas flows, a 2100°C maximum temperature, and a short-time cycle (see Figure 3). The D-615-616 combination was an interrupted run using the same time temperature cycle used in D-617 (see Figure 4).

(CRD) The average weight loss for S-133 was 12.2 grams with a range of 10.8 to 15.0 grams. The average for the D-615-616 combination was 14.7 grams with a range of 12.5 to 17.0 grams. The D-617 average weight loss was 14.5 grams with a range of 12.5 to 16.9. It is readily apparent that the longer cycle is more effective in leaching uranium out of the matrix than the short cycle.

(CRD) A problem was noted in the leaching of the carbide tipped elements. There seems to have been some chemical attack on the carbide tip itself by the leach gases leaving what appears to be a chip on the hot end corners. Some attack was also noted on the molybdenum braze material. Initially, it was felt that this attack was caused by allowing the leach gases to enter the chamber at 1000°C. The cycle was later modified to permit gas entry only about 1600°C, thereby eliminating leaching in a carbide sensitive temperature range. The effects of this modification have not, as yet, been analyzed.

4.5 LEAK RATE (U)

(CRD) All elements were subjected to whole body leak tests at 10 psig in the as-machined condition; most elements were retested after leach. Incremental leak tests were requested in the as-leached condition on all elements whose leak rate exceeded 70 cc/min. The data for the individual elements are presented in Table 4 and summarized in Table 5.

(CRD) The data show that all methods tried did significantly lower element permeability. The most effective method was to extrude after a 48-hour hold at

[redacted]

TABLE 4 (CRD)

AS-LEACHED LEAK RATE FOR THE TS-3005 ELEMENTS (U)

S/N	Matrix Type	Incremental (cc/min)																	Whole Body (cc/min)	
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	As-Mach.	As-Leach.
550-1046114	Standard and Argon	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	(A)	---
550-1046115	↓	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	94	---
550-1046116	↓	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	(A)	---
550-1046117	↓	17	22	25	28	29	30	32	31	32	29	30	29	26	24	33	35	20	180	200
550-1046118	↓	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	(A)	---
550-1046119	↓	9	10	10	11	12	14	14	15	15	15	15	14	13	11	10	10	10	61	74
550-1046120	↓	10	10	10	11	11	12	13	13	12	12	12	11	10	11	10	9	10	56	90
550-1046121	↓	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	74	---
550-1046122	↓	23	27	18	20	16	18	16	20	15	16	15	14	12	13	27	27	66	83	90
550-1046123	↓	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	47	58
550-1046124	Standard and Argon	6	11	12	12	13	13	13	13	13	11	11	11	10	10	9	9	1020	70	78
550-1046125	24-hr. Hold at 38°C	10	11	12	15	15	15	14	14	18	18	17	16	19	19	18	17	18	79	150
550-1046126	↓	16	22	25	25	26	24	23	21	21	20	21	20	18	16	13	13	500	125	145
550-1046127	↓	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	120	180
550-1046128	↓	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	145	225
550-1046129	↓	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	195	---
550-1046130	↓	13	21	23	25	23	25	24	26	25	24	24	22	17	17	18	18	23	140	155
550-1046131	↓	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	180	180
550-1046132	↓	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	130	200
550-1046133	↓	14	18	19	20	20	21	22	24	24	24	23	24	22	19	18	16	16	135	150
550-1046134	↓	21	35	37	49	37	37	40	40	32	32	27	27	27	24	21	21	26	200	220
550-1046135	24-hr. Hold at 38°C	11	16	20	23	26	27	27	27	27	27	26	26	26	25	26	26	26	160	180

[redacted]

TABLE 4 (CONTINUED) (CRD)

S/N	Matrix Type	Incremental (cc/min)																	Whole Body (cc/min)	
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	As-Mach.	As-Leach.
550-1046136	Standard	24	25	27	30	32	37	37	40	40	41	41	45	960	45	45	41	43	380	700
550-1046137		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	280	---
550-1046138		21	27	33	39	42	43	43	42	42	42	42	42	42	39	31	29	26	205	300
550-1046139		16	22	24	28	30	31	31	30	31	31	31	31	26	22	20	19	19	210	180
550-1046140	Standard	44	44	43	43	43	44	43	44	44	43	43	43	43	40	33	30	24	260	285
550-1046141	48-hr. Hold at 38°C	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	50	48
550-1046142		9	10	16	11	11	13	13	13	13	12	14	13	13	10	11	8	12	67	105
550-1046143		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	46	51
550-1046145		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	58	---
550-1046146		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	36	36
550-1046147		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	40	40
550-1046148		0	0	6	6	6	9	9	9	9	9	9	10	9	9	9	8	10	31	56
550-1046149		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	42	41
550-1046150		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	53	61
550-1046151	48-hr. Hold at 38°C	0	0	9	13	105	86	14	12	11	10	11	11	10	10	11	10	9	115	98
550-1046152	48-hr. Hold at R. T.	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	50	62
550-1046153		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	50	57
550-1046154		10	10	9	10	10	11	11	11	11	11	11	11	11	12	10	11	11	54	87
550-1046155		9	11	11	11	12	13	15	13	13	13	13	14	16	16	12	11	11	74	75
550-1046156		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	44	50
550-1046157		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	49	---
550-1046158		7	9	10	11	10	13	16	16	16	16	13	13	13	12	11	10	11	60	100
550-1046159		11	15	15	18	18	21	23	05	22	21	21	20	19	18	16	13	13	120	96
550-1046160	48-hr. Hold at R. T.	16	20	20	20	2700	05	2700	2150	24	37	175	05	05	455	25	25	05	05	05
550-1046161	Standard	9	10	11	13	16	16	15	16	16	16	18	22	24	25	22	22	20	125	92
550-1046162		10	11	12	16	17	17	17	16	15	15	15	15	15	14	11	10	10	60	99
550-1046163	Standard	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	52	63

[redacted]

[redacted]

TABLE 4 (CONTINUED) (CRD)

S/N	Matrix Type	Incremental (cc/min)																	Whole Body (cc/min)	
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	As-Mach.	As-Leach.
550-1046164	Standard	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	(A)	---
550-1046165	Standard	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	60	---
550-1046166	18% Thermax	27	49	20	40	32	37	23	27	23	32	46	30	20	20	18	18	27	100	115
550-1046167		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	71	---
550-1046168		11	11	12	13	17	18	18	16	16	16	17	19	19	18	16	15	16	70	100
550-1046169		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	97	---
550-1046170		10	9	9	10	13	13	13	11	11	12	19	20	20	20	20	20	210	82	92
550-1046171		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	53	63
550-1046172		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	56	91
550-1046173		8	8	9	16	10	11	11	11	11	13	13	16	16	16	16	16	14	64	100
550-1046174		10	11	11	13	11	13	12	20	20	20	16	16	14	27	13	13	16	75	87
550-1046175		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	38	49
550-1046176	18% Thermax	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	50	58
550-1046177	21% Thermax	0	6	8	0	8	8	9	10	12	18	20	19	18	19	14	15	14	54	---
550-1046178		0	0	8	7	9	9	9	9	17	11	13	21	21	10	10	9	10	32	61
550-1046179		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	33	---
550-1046180		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	34	40
550-1046181		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	31	43
550-1046182		9	7	8	8	8	8	9	9	9	9	9	9	9	11	10	10	13	26	47
550-1046183		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	47	---
550-1046184		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	58	66
550-1046185		19	11	23	16	13	15	16	24	20	18	13	18	15	20	17	13	12	59	72
550-1046186		30	16	13	13	16	16	18	18	41	20	20	19	20	43	44	66	2700	100	105
550-1046187	21% Thermax	10	11	12	12	12	12	12	16	15	14	14	14	15	15	15	16	16	64	77

[redacted]

[redacted]

Astronuclear Laboratory

[redacted]

TABLE 4 (CONTINUED) (CRD)

S/N	Matrix Type	Incremental (cc/min)																	Whole Body (cc/min)	
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	As-Mach.	As-Leach.
550-1046188	Lot #1 Sized Flour	32	46	49	49	46	49	32	46	47	66	66	27	25	23	46	46	410	120	140
550-1046189		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	75	---
550-1046190		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	62	99
550-1046191		10	10	25	14	15	14	15	16	17	17	16	18	16	18	17	17	17	68	80
550-1046192		250	25	44	43	66	250	175	41	40	20	49	44	58	46	46	58	30	100	120
550-1046193		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	85	98
550-1046194		12	13	15	17	22	21	23	23	23	23	23	23	25	24	23	22	30	95	160
550-1046195		13	17	17	17	23	19	25	22	22	19	20	19	21	24	23	23	22	86	140
550-1046196		19	20	25	21	23	41	25	44	32	26	27	37	24	25	23	23	24	120	140
550-1046197		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	92	145
550-1046198	Lot #1 Sized Flour	10	19	250	330	250	44	53	44	330	35	250	330	35	35	35	25	49	74	81
550-1046199	Lot #2 Sized Flour	11	12	13	11	13	13	13	14	14	14	15	17	18	20	22	26	24	64	90
550-1046200		10	11	13	16	16	18	18	18	18	19	19	19	20	20	20	20	21	82	140
550-1046201		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	84	(A)
550-1046202		37	66	66	250	210	210	210	30	27	44	44	470	32	32	20	20	330	95	100
550-1046203		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	54	66
550-1046204		44	35	46	41	13	23	32	35	18	35	18	13	14	13	13	11	11	64	73
550-1046205		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	51	57
550-1046206		9	10	10	10	10	11	11	11	11	12	13	13	14	14	14	14	16	65	100
550-1046207		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	60	70
550-1046208		--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	52	59
550-1046209	Lot #2 Sized Flour	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	53	60

[redacted]

TABLE 5 (CRD)

SUMMARY OF LEAK RATES
OF THE TS-3005 ELEMENTS (U)

Process	As-Leached Leak Rate (cc/min)		
	Average	Range	Sample Size
Standard	246	63 to 700	7
Standard With Argon Cure	98	58 to 200	6
24-Hour Hold at 38^{o}C	178	145 to 225	10
48-Hour Hold at 38^{o}C	60	36 to 105	9
48-Hour Hold at Room Temperature	75	50 to 100	7
18% Thermax	83	49 to 115	9
21% Thermax	66	40 to 105	9
Lot #1 Flour	120	80 to 160	10
Lot #2 Flour	82	57 to 140	10

elevated temperature, while the least effective method was a 24-hour hold. Also, the extrapolation of Figure 1 proved to be a conservative estimate.

(U) One anomaly was noted. One of the two standard batches had unusually low permeabilities. This batch, 5709, received the same treatment as its sister batch 5708. As yet the distinct difference between the two standard batches is unexplained. However, this kind of batch-to-batch variation is not outside the range of previous WNCO experience for standard formulations.

5.0 DISCUSSION OF RESULTS (U)

(U) The objectives of this experimental program were achieved to a surprising extent. All of the proposed methods were effective in lowering the permeability of the elements. While permeability is somewhat statistical in nature, it is felt that enough elements were tested to give reasonable confidence in the results.

(CRD) It must be remembered that this was only the first try at a controlled experiment and does not represent the limit that can be obtained. The obvious extension would be to try combinations of methods such as a 48-hour hold and an argon cure. Also, there is the possibility of custom-blending graphite flour so that a particular particle size distribution is used for each fuel loading.

(CRD) The results dictate a change from standard processing if elements with fuel loadings in excess of 525 mg U/cc are to be made with whole-body leak rates less than 170 cc/min. The results also show that several methods can be used. The method differing the least from previous practice is to add thermax. However, this method is made less attractive by the fact that it adds a poorly graphitizable material to the matrix. Custom-blending flour is somewhat expensive and time consuming. Extended holding periods are awkward from a criticality standpoint. It was recommended, therefore, that argon curing be incorporated in the standard process procedures. Subsequent to TS-3005, a number of extrusions,using the argon curing step,have successfully met permeability requirements, which confirms the validity of the recommendation.