MASTER

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OAK RIDGE NATIONAL LABORATORY
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Post Office Box X
Oak Ridge, Tennessee

DATE: August 31, 1959

COPY NO. 71

SUBJECT: Molten Salt-Graphite Compatibility Test. Results of Physical and Chemical Measurements

TO: Distribution

R-9462

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ABSTRACT

The observed changes in dimensions, weight, and chemical composition of "impervious" graphite rods in contact for a year with flowing liquid LiF-BeF_2-UF_4 (62-37-1 mole %) in a pump loop at 1300°F are listed. On the average the change in diameter was less than 0.02%, and the change in weight was less than 0.03%. Chemical analyses of machine cuttings from the graphite rods show average uranium and beryllium concentrations of approximately 20 and 100 ppm respectively.

NOTICE

MOLTEN SALT-GRAPHITE COMPATIBILITY TEST

RESULTS OF PHYSICAL AND CHEMICAL MEASUREMENTS

In a graphite moderated-molten salt reactor, certain advantages are possible if the graphite is in direct contact with the circulating fluid. The feasibility of such a design, which depends to a large extent on the compatibility of the graphite-fused salt system, was tested by inserting 56 samples of an impervious graphite contained by INOR-8 in a flowing stream of $LiF-BeF_2-UF_4$ (62-37-1 mole %) for a period of one year at 1300°F.

The experimental assembly consisted of a pump loop constructed and operated by the Metallurgy Division and the Experimental Engineering section of the Reactor Projects Division as described elsewhere.[1]

Graphite Loop - Initial Preparations

Two sets of graphite specimens were obtained from the National Carbon Company. All rods were 11" long and either 1/2" or 3/16" in diameter. After calipering and weighing, the rods were packaged in an INOR-8 box as shown in Figures 1 and 2. Each of the larger was fitted with four spacing rings to permit the desired flow characteristics and to provide samples for carburization and metallographic examination.

Upon welding the cover plate in place, the assembly was swaged in the loop and the graphite degassed under vacuum for 24 hours at 1100°F. It was then repressured with argon before being contacted with salt. During operation, a total pressure of 13 psig was maintained on the graphite: 3 psig the contribution of the helium cover blanket and the remaining due to the head developed by the pump-rate of 1.1 gal/min. When the test was completed, the salt was drained from the loop and sampled for optical microscopy, X-ray and wet chemical analyses.

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1. J. L. Crowley, MSR Quar. Prog. Rep., January 31, 1958, ORNL-2474.

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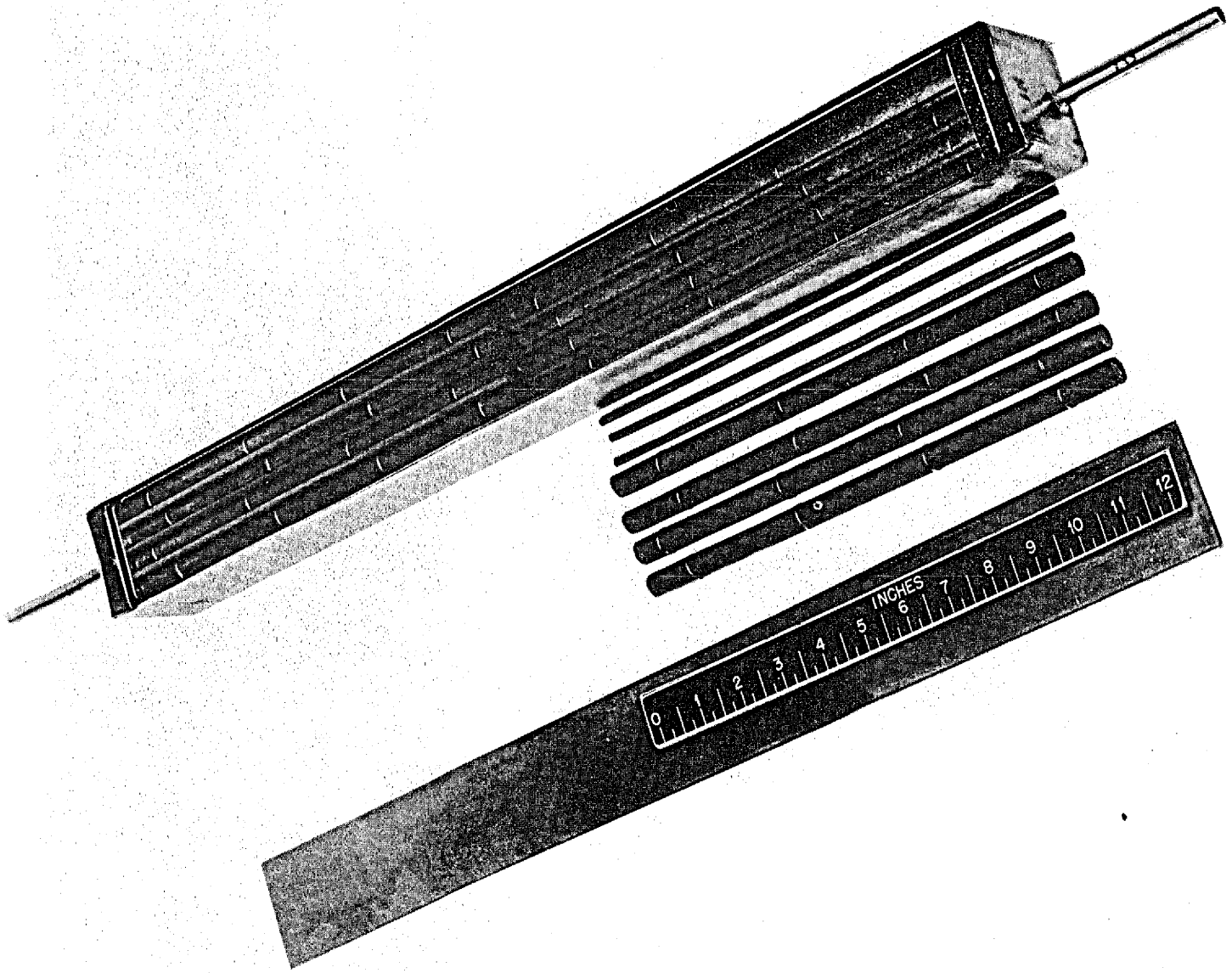

Fig. 1.

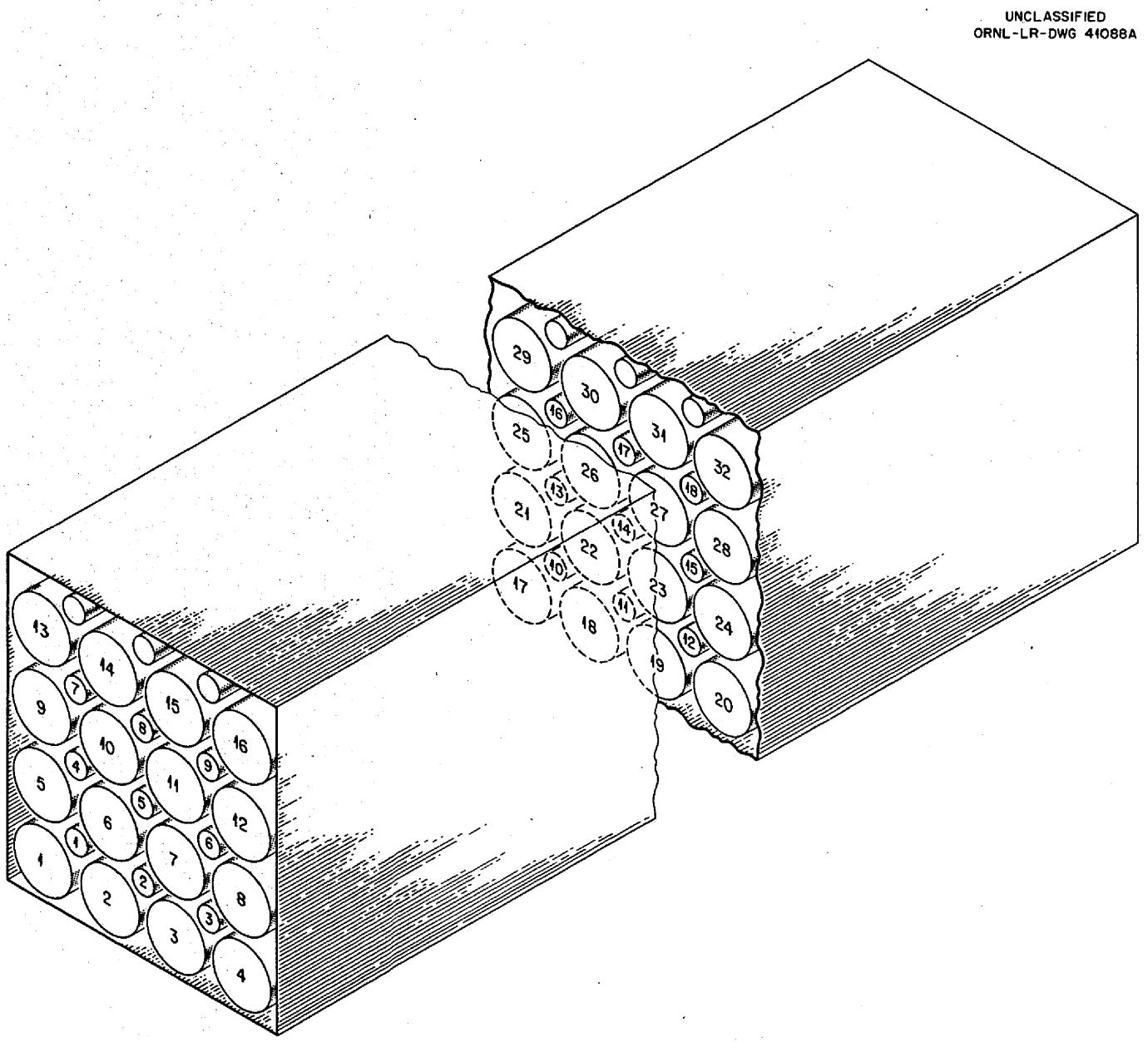

Fig. 2. Numbering Scheme for Positioning Graphite Rods.

The box containing the graphite was opened by grinding off the top of the container in the Y-12 Beryllium Shop. Figure 3 is a photograph of the opened vessel after exposure to molten fluorides for one year at 1300°F.

Post Test Examinations

A. Salt Condition. - The chemical analysis, given in Table I shows the chromium content of the salt increased from 135 ppm to 550 ppm. Petrographic and X-ray analyses did not reveal any oxidation products or dissolved carbon.

B. Graphite Condition

1. Chemical Analyses Data. Machined increments of graphite specimens were submitted for chemical analysis. Successive cuttings, 1/32" in depth were taken from two of the larger diameter rods until center portions of less than 3/16" diameter were left. These portions and "as received" impervious graphite "blanks" were then ground to -100 mesh in a mortar and pestle (thoroughly scoured with Ottawa Sand according to the recommendations of the Analytical Chemistry Division after each grinding). All graphite samples were submitted for an analysis of the uranium and beryllium concentrations. Two machine cuttings, 1/32" in depth, were taken from four additional rods. These results are given in Table II with the beryllium and concentrations graphed as a function of penetration depth in Figure 4. Only a very slight migration of salt to the center of the graphite is noted.

2. Physical Changes in Graphite. Macroscopically, there was no change in the rods. None of the samples were broken or distorted and except for the bottom layer of rods that was covered with solidified melt, the salt did not adhere to the graphite. The weight and dimensional changes observed after they had been contacted with circulating fluorides are listed in Table III. The dimensional changes for the 13-1/2" diameter rods corresponded to an average loss of less than 0.5 mil in diameter which approximates the probable error of the measurements. Otherwise, there was no evidence of erosion. Weight losses, which ranged from negligible to 0.05% and averaged 0.02% could be attributed to desorption of residual gases from the graphite. No statistically significant differences were noted in the 13-1/2" diameter rods as compared with the 8-3/16" diameter rods for which weight data were available.

Based on the loss in weight of the test samples and the chemical analyses of the machine cuttings, it appears that only minute quantities of salt permeated the graphite.

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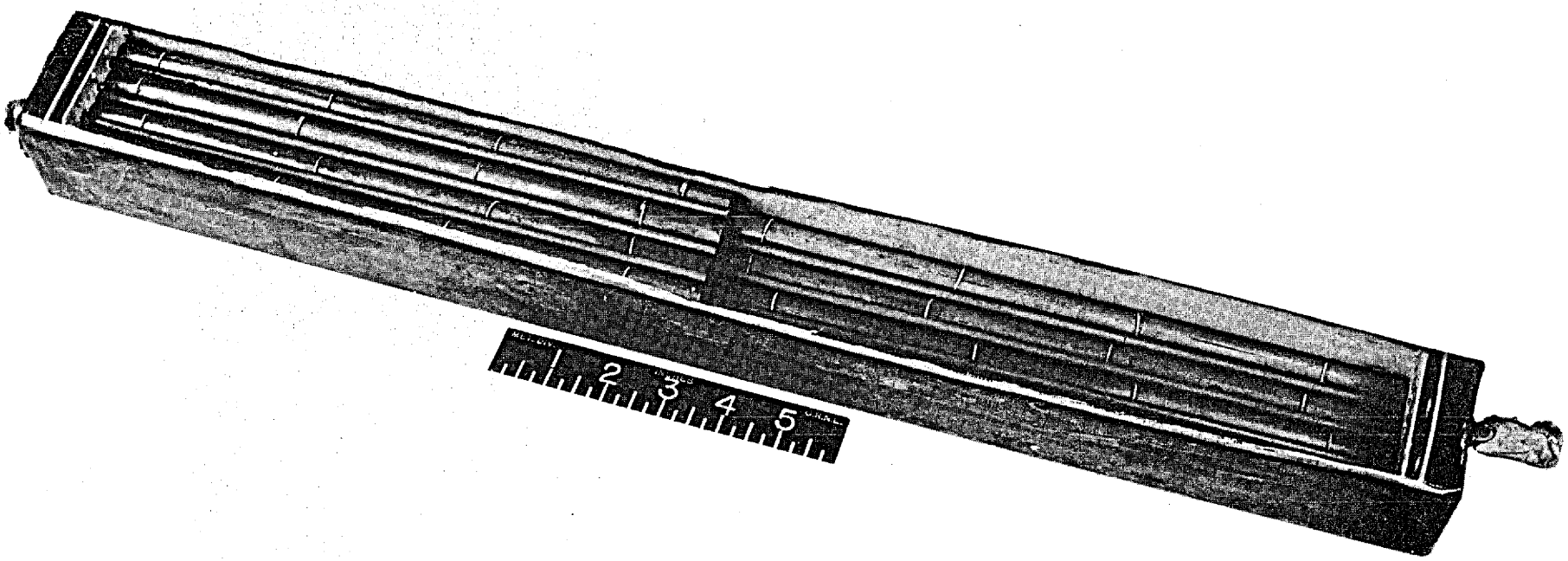

Fig. 3.

Table I

Chemical Analyses of Fuel*

	Wt. %			Theoretical**	PPM		
	U	Be	U/Be	U/Be	Fe	Cr	Ni
Charge	4.87	8.37	0.582	0.767	235	135	5
After operating 1 year	4.97	9.77	0.509		330	555	25

* G. J. Nessle, Personal Communication.
** Calculated for LiF-BeF_2-UF_4 (62-37-1 mole %).

Table II

Analyses of Machine Cuttings from Graphite Rods

Rod No.	Cutting No.	PPM U	PPM Be	Theoretical[1] U/Be	Actual U/Be
8	1	30	125	0.573	0.240
	2	9	175		0.051
	*	10	< 1		-
11	1	22	125		0.176
	2	10	110		0.091
14	1	24	75		0.320
	2	28	105		0.267
23	1	17	125		0.136
	2	<1	60		0.017
	*	5	< 1		-
18	*	8	< 1		-
	1	50	170		0.294
	2	15	130		0.115
	3	15	125		0.120
	4	12	100		0.120
	5	10	65		0.154
	6	13	105		0.124
	7	<1	50		0.020
	8	13	140		0.093
	9	5	165		0.030
	10	<1	< 1		1.000
	11	6	105		0.057
	12*	<1	< 1		-
	Center	100	125		0.800
31	*	5	< 1		-
	1	20	165		0.121
	2	18	140		0.129
	3	24	120		0.199
	4	20	85		0.235
	5	20	75		0.267
	6	20	80		0.250
	7	17	55		0.310
	8	<1	80		0.013
	9	<1	95		0.011
	10	<1	< 1		1.000
	11	<1	90		0.011
	Center	70	170		0.411
	*	<1	< 1		-

* Samples machined from "as received" material.

1. Based on chemical analysis at original salt batch, nominally $LiF-BeF_2-UF_4$ (62-37-1 mole %).

Table III

Impervious Graphite Rods (0.5" diameter)

Rod No.	Before Exposure Weight (gms)	Before Exposure Dia. (in.)	After Exposure Weight (gms)	After Exposure Dia. (in.)	Net Change (gms)	Percent Change
1	Lost					
3	Lost					
6	68.0555	0.498	68.0397	0.496	-0.0158	-0.02
8	68.0571	0.498	68.0438	0.497	-0.0133	-0.02
9	68.5709	0.502	68.5572	0.501	-0.0137	-0.02
11	68.4152	0.500	68.4096	0.500	-0.0056	-0.01
14	68.7779	0.499	68.7639	0.500	-0.0140	-0.02
16	67.7389	0.496	67.7205	0.495	-0.0184	-0.03
18	68.2650	0.498	68.2517	0.497	-0.0133	-0.02
20	Lost					
21	68.5801	0.500	68.5793	0.500	-0.0008	0.00
23	67.9828	0.497	67.9703	0.496	-0.0125	-0.03
26	68.2956	0.499	68.2911	0.500	-0.0045	-0.01
28	68.6806	0.501	68.6666	0.501	-0.0140	-0.02
29	67.8522	0.499	67.8352	0.498	-0.0174	-0.03
31	67.9389	0.499	67.9169	0.498	-0.0220	-0.04
	Impervious Graphite Rods (3/16" diameter					
2	9.1095		9.1082		-0.0013	-0.01
4	9.1236		9.1228		-0.0012	-0.01
6	9.4826		9.4810		-0.0016	-0.02
8	9.0329		9.0352		+0.0021	+0.02
10	9.3176		9.3126		-0.0050	-0.05
12	8.7251		8.7372		+0.0121	+0.14
14	9.0932		9.0930		-0.0002	0.00
16	9.5142		9.5098		-0.0044	-0.05
18	9.0149		9.0104		-0.0045	-0.05

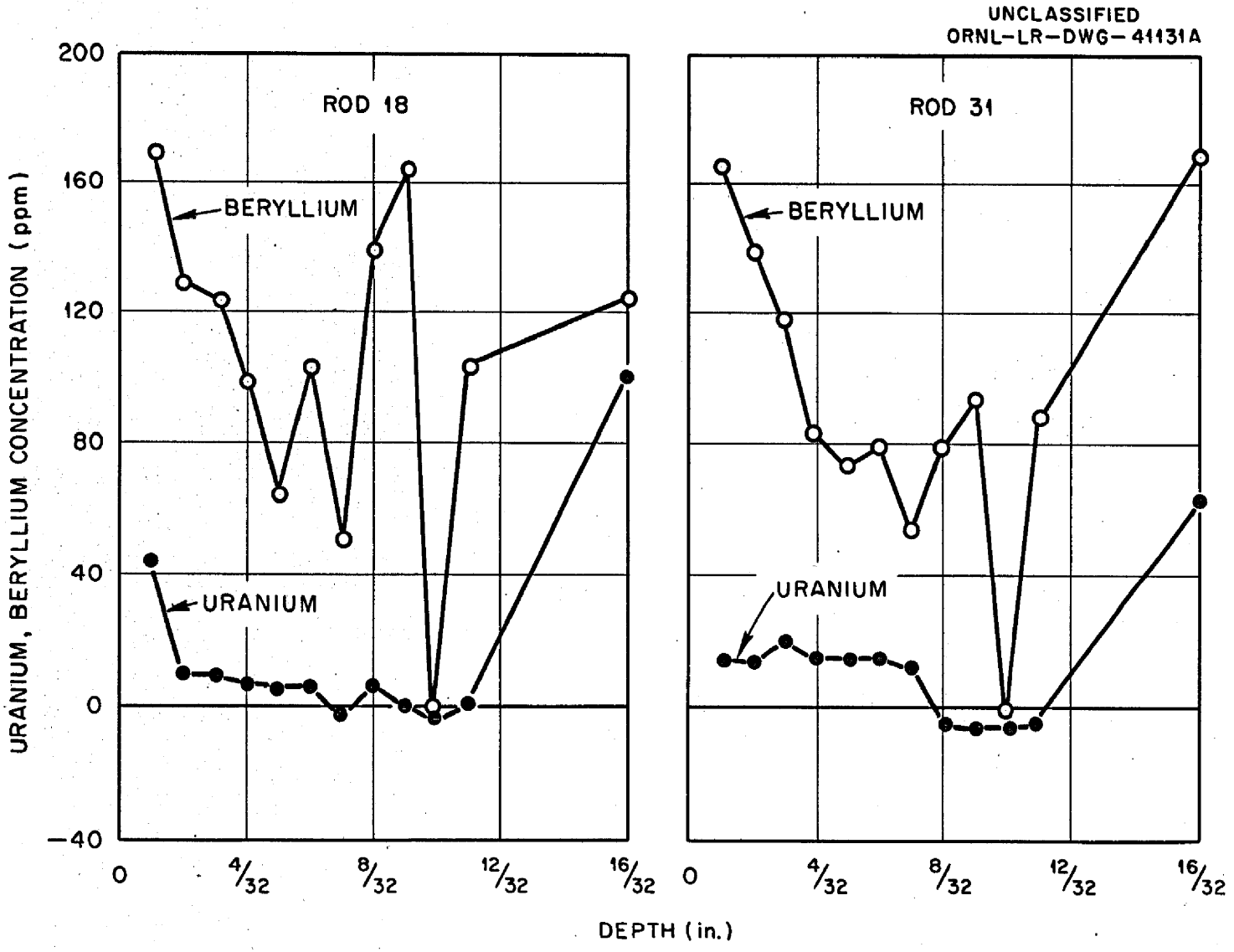

Fig.4. Penetration of an Impervious Graphite by $LiF-BeF_2-UF_4$.

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