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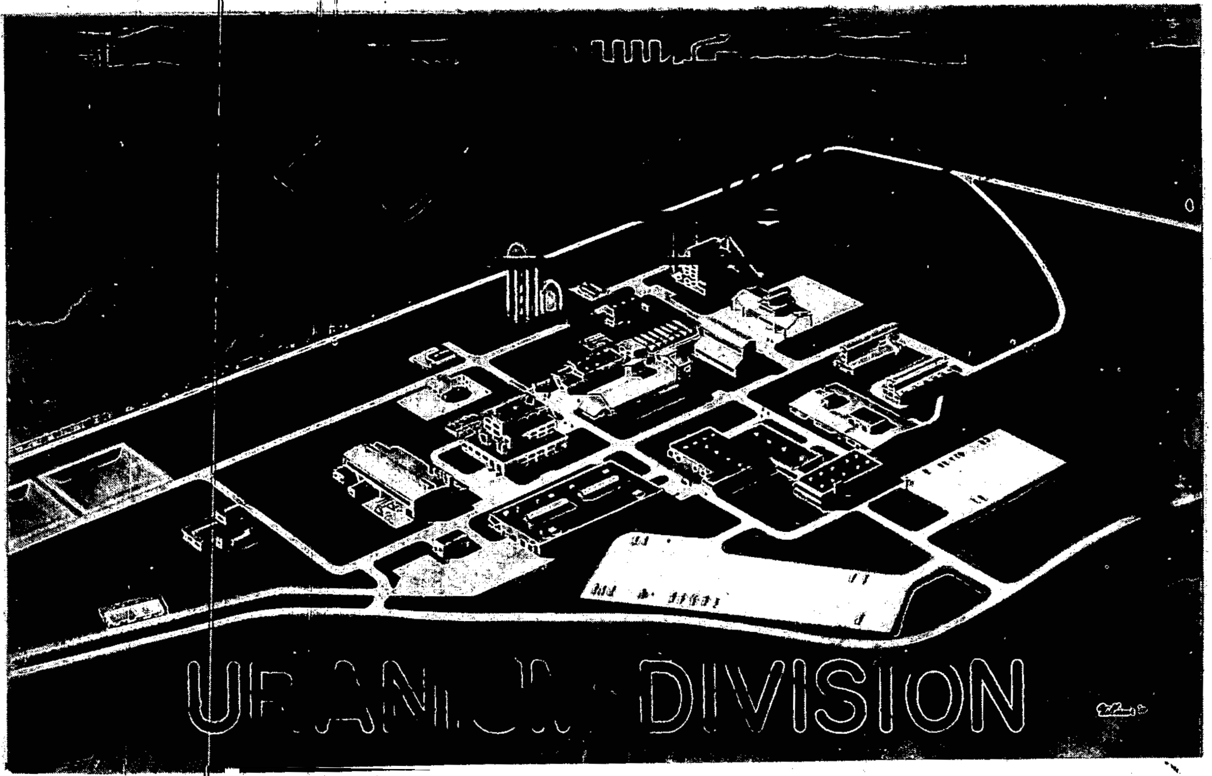

A.E.C. RESEARCH & DEVELOPMENT REPORT

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PROCESS DEVELOPMENT QUARTERLY REPORT
PART II - PILOT PLANT WORK

edited by Barbara Elliott

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Title: PROCESS DEVELOPMENT QUARTERLY REPORT, PART II - PILOT PLANT WORK

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GENERAL SUMMARY

During the second quarter of 1957 work in the MCW Process Development pilot plant has continued on the TBP-hexane extraction cycle of the Weldon Spring refinery with studies being concentrated on the low-acid flowsheet, process flow rates, and TBP removal from raffinate. The Metallurgical pilot plant has continued investigations on hydrogen in uranium metal, dingot forging, extrusion, and UO_2 fuel elements. Plant decontamination factors were determined for cerium (III), magnesium, and aluminum.

Specific studies are summarized as follows (Roman numerals refers to the section on which summary is based):

II. A study has been made in the pilot plant to determine the effects of several factors on digestion operations. It was determined that the concentration of nitric acid required for the digestion of the concentrates tested was limited by reaction stoichiometry rather than kinetics for 210° F digestion.

Temperature was a major variable with high temperatures favoring fast dissolution rates. As a result, it is predicted that the Weldon Spring digest capacity would not be hindered by using 35% acid for digestion rather than 60%.

III. Monticello A and Porter Brothers' ore concentrates were tested in the TBP-hexane extraction pilot plant at a 400 g. U/l, 1M nitric acid feed level. Monticello demonstrated excellent product purity but gave poor recovery. Porter Brothers' concentrate gave excellent recovery but relatively poor product purity.

IV. Theoretic calculations have been used to predict the tolerance variation in flow ratios in the Weldon Spring pumper decanters, wash columns, and reextraction columns. As a result of these calculations, it is predicted that the pumper decanter flow ratio must be maintained within ±5%, the wash column flow ratio as high as ±10%, and the reextraction columns ±5%.

V. Four methods for removing TBP from extraction column raffinates have been studied. Of the four methods tested, steam distillation of the TBP from the raffinate was the most effective.

VI. Flame fusions with an atomic hydrogen arc of UO_2 with small additions of Al_2O_3 encountered difficulties with trapped air bubbles. Improved mechanical controls should avoid this problem and give maximum density.

VII. Extrusion of micronized UO_2 has permitted sintering to densities above 96% of theoretical with excellent surface quality.

VIII. Successful reduction of hydrogen in dingot metal appears possible by three techniques:

1. The use of lower furnace control temperatures to prolong the preheat and raise the bomb center temperature at the time of firing;

2. Additions of barium peroxide or uranium trioxide introduced as a possible means of tying up hydrogen from the bomb atmosphere and to provide a potential source of extra heat;

3. The pre - treatment of the magnesium in HF at 500°C to outgas the chips of hydrogen and to provide a MgF_2 surface film that may be conjectured to be protective during bomb heating.

Of these three techniques, the HF filming of magnesium appears to be the most attractive for operation at Weldon Spring.

IX. The major effort in the forging of dingot metal was directed at preparation of low hydrogen materials for HAPO. An assortment of 10 inch diameter cast NLO ingots was forged to 7½ inch rounds for subsequent rolling.

X. Experimental extrusions in the gamma range at Adrian gave further evidence that glass lubrication eliminates die wear, whereas damage became immediately evident when "oil dag" was used; extrusion pressures were lower with glass lubrication but rod surfaces showed certain imperfections such as grooving and gouging; contrary to initial expectations, hydrogen pickup from salt bath preheating at 1900°F was negligible.

XI. Studies of bomb reduction practice have indicated that magnesium which has been surfaced - filmed in HF may provide a more satisfactory reductant for green salt having a high UO_2 content; bomb liner hardnesses are higher with material containing an excess of fines; maximum hardness appears to be reached at a constant jolting time regardless of particle size distribution.

XII. Plant tests in the Destrehan Refinery gave decontamination factors of >3 x 10^5 for cerium (III),10^5 for magnesium, and 10^5 for aluminum.

Laboratory work for this quarter has been reported in a separate volume as *Part I, MCW-1403.*

FACTORS AFFECTING THE DIGESTION OF ORE CONCENTRATES

by

J. T. Krieg
G. B. Wills

I. Summary

Nitric acid strength (1*N* *vs.* 3*N*) was a minor variable in the rate and completeness of feed material digestions. One exception was Uravan where excess acid (not initial acid) concentration was an important governing factor in digestion reaction kinetics. In all cases studied the concentration of stock nitric acid required for digestion was limited by reaction stoichiometry rather than kinetics for 210° F digestions. Temperature was a major variable with high temperatures favoring fast dissolution rates. Extended digestion periods at 210°F (in excess of one-half hour) were beneficial in dissolving Uravan and Vitro concentrates (in 1 and 3*N* excess acid) and Monticello A (in 1*N* excess acid).

These tests predict that the ability of the Weldon Spring Refinery to digest ore concentrates would not be hindered if it were decided to recover acid from raffinate at 35 instead of 60 weight percent, thereby, minimizing the chloride corrosion problem in the acid reconcentrator.

II. Introduction

The Weldon Spring refinery is capable of recovering excess nitric acid from raffinates and concentrating it to 60 weight percent for reuse in the digesters; however, serious corrosion problems are anticipated if halides are allowed to accumulate in the acid reconcentrator. Preventative measures are planned for halide control; however, it will be some months after plant startup before all of the necessary equipment will be available. One proposal for operation during the interim period is to produce 35 instead of 60 weight percent nitric acid in the reconcentrator which, together with other modifications, would reduce chloride accumulation in the unit. This proposal brings up the question of whether recovered acid at 35 weight percent (together with more concentrated absorber and makeup acid) would be adequate for feed material digestions. A digestion program was initiated by the pilot plant to study this problem.

III. Description of Equipment

Digestions were made in 1-liter stainless steel beakers over a hot plate. Extraction of the uranium from the digest liquors by 28% TBP in hexane was made in glass separatory funnels (shakeouts). All ore concentrates which were available at the time in the Destrehan refinery were tested (a total of eleven feeds).

IV. Experimental Procedure

A. Digestion Procedure

Measured quantities of concentrate, water, and nitric acid were brought together at a controlled temperature and digested for eight or more hours. Two methods of acid addition were used. In several of the tests the ore was added to an acid-water mixture, so that for the first few seconds of the digestion the ore was exposed to concentrated acid. During this short time interval most of the uranium dissolved and the acid strength very quickly diminished to flowsheet specifications (1*N* or 3*N* excess acid); therefore, for the majority of the digestion period, it was this latter acid concentration to which the insoluble uranium was exposed.

In most of the tests, however, the concentrate was first slurried in water after which the acid was slowly added. In this manner the ore was never exposed to an acid concentration in excess of 1*N* or 3*N*. This method was not intended to be a plant procedure but was used only to obtain digestion information at controlled acid concentrations. Samples were taken of the slurry at intermediate time intervals to determine the amount of undigested (inextractable) uranium. Digestions were made to 200 g U/l at 210°F, 1900° F or 75° F.

B. Analytical Procedure

Several methods for determining the insoluble uranium content in a slurry are available; however, in order to simulate actual plant environment as much as possible, the following procedure was used. Each sample was given a 4-stage shakeout with acidified (35 g HNO_3/l) TBP-hexane at a 5:1 organic to aqueous volume ratio in each stage. In all cases the low uranium content of the organic phase from the final shakeout indicated substantially complete extraction of the soluble uranium from the ore slurry. Raffinates from the shakeout procedure were analyzed for uranium content which was termed "the inextractable uranium".

C. Procedure for Determining Stoichiometric Acid Requirements

In addition to the experimental work on the kinetics and completeness of digestions at various temperatures and acid concentrations, a stoichiometric analysis of digest acid requirements was made. Nitric acid consumption data for each concentrate was obtained from the laboratory and the Destrehan Refinery. The method of correlating this data is described in the Appendix.

V. Results

The stoichiometric nitric acid concentrations required to digest feed materials to 200 g U/l, 3*N* excess acid are tabulated below in Table I. Stoichiometrically the minimum nitric acid concentration required to dissolve feed materials to flowsheet conditions is between 28 and 33 weight percent.

Table I

Concentration and Volume of Acid[a] Required for Feed Digestion

(200 g U/l, 3*N* Acid Flowsheet)

Feed Material	Assay %U	Acid Concentration Required %	Gal Acid/ lb Feed
Anaconda A	70	33	0.39
Anaconda C	69	28.8	.384
Monticello A	58	28.7	.318
Edgemont	61	27.9	.336
Kerr-McGee	66	29.6	.366
Rare Metals	62	28.3	.342
Rifle	71	28.6	.396
Moab	61	28.7	.336
Vitro	62	32.3	.342
Climax	68	30	.378
Durango	74	32.5	.414
Uravan	67	28.6	.372
Naturita	70	33.7	.390
Euxenite	69	30.8	.384

[a] Includes total amount of acid required to bring excess acid concentration up to flowsheet conditions.

The results of tests showing the kinetics and completeness of the digestions as a function of acid concentration are tabulated below in Table II.

Table II

Digestion Kinetics

Test No.	Feed Material	Free Acid[a] Concentration of Digest, N	Percent of Total Uranium[b] Inextractable at Noted Time of Digestion				
			½ hr	1 hr	2 hr	4 hr	8 hr
1	Rifle	1.2	0.06	0.08	0.08	0.08	0.15
2	Rifle	3.0	.09	.11	.10	.09	.11
3	Monticello-A	1.0	.14	.05	.03	.02	.01
4	Monticello-A	3.6	.01	.03	.01	< .01	.01
5	Vitro	1.3	.07	.06	< .01	.06	< .01
6	Vitro	3.0	.05	.04	---	.03	.02
7	Kerr-McGee	1.4	.02	.02	.02	.02	.02
8	Kerr-McGee	3.0	.02	.03	.02	.01	.02
9	Moab	1.4	.01	.02	.04	.01	.01
10	Moab	3.0	.01	.02	.02	.02	.01
11	Rare Metals	1.3	.01	.02	.02	.02	< .01
12	Rare Metals	3.2	---	< .01	---	< .01	< .01
13	Anaconda A	1.1	.01	.01	< .01	< .01	< .01
14	Anaconda A	3.0	< .01	< .01	< .01	< .01	< .01
15	Anaconda C	1.3	< .01	< .01	< .01	< .01	< .01
16	Anaconda C	3.0	< .01	< .01	< .01	< .01	< .01
17	Durango	1.0	< .01	.013	< .01	< .01	.012
17A	Naturita	1.0	.08	.01	.016	.008	.01
18	Naturita	3.0	.011	.007	.008	.006	.007
19	Uravan	1.4	---	---	1.6	1.0	.5
20	Uravan	3.3	---	---	.29	.28	.26
21	Uravan[c]	4.9	.3	.26	.21	.15	.12

[a] At no time during the digestion was the ore exposed to acid concentrations in excess of this value. (The acid was added to an ore-water mixture.)

[b] Total uranium was 200 g/l.

[c] In this test the ore was added to 50 weight percent HNO_3 and adjusted with water.

As may be seen in Table II, with most feeds the acid strength played a minor role in digestion kinetics, its effect being noticeable only with Uravan and Monticello A. The effect of acid concentration in Uravan digestions is illustrated in Figure 1. Since most of the digestion reaction takes place in the first few minutes, it appears that the prolonged contact with the remaining excess acid rather than the initial acid concentration is responsible for dissolving the small amount of insoluble uranium. Uravan, Rifle, and Vitro contained more inextractable uranium than the other feeds with Uravan being by far the worst offender regardless of the initial acid concentration (Table I). In other cases digestion was essentially complete within one-half hour.

As might be expected at very low uranium concentrations, there appears to be a problem of precision associated with the results listed in Table II. It would seem improbable that the samples taken later in the digestion would contain more inextractable uranium than the initial sample taken after one-half hour of digestion time, yet this would appear to be the case for a number of the tests, *i.e.*, Tests 1 and 2 (Table II).

Figure 2 illustrates the effect of temperature (210°F *vs* 190°F) on the kinetics of Uravan digestion. Two hours at 210°F was more effective than nine hours at 190°F. The effect of temperature was illustrated more dramatically by another test in which Uravan was digested for eight hours at 210°F in the presence of 4 to 5*N* excess acid. During this period the amount of inextractable uranium diminished with increasing time, a level of 0.12% inextractable uranium being reached at the conclusion of the test. The remaining slurry from this test was then bottled and stored for 25 days at room temperature. After this storage period the slurry was again heated to 210°F and digested at that temperature for an additional eight hours. The results of the previous test together with the results for the additional eight hours of digestion are shown in Figure 2. As may be seen, the second high temperature digestion period reduced the inextractable uranium from 0.12% to 0.043% while the 25 days at room temperature resulted in no significant change in the inextractable uranium content.

VI. Appendix

Correlation of stoichiometric digestion data is presented below:

A. Source of Data

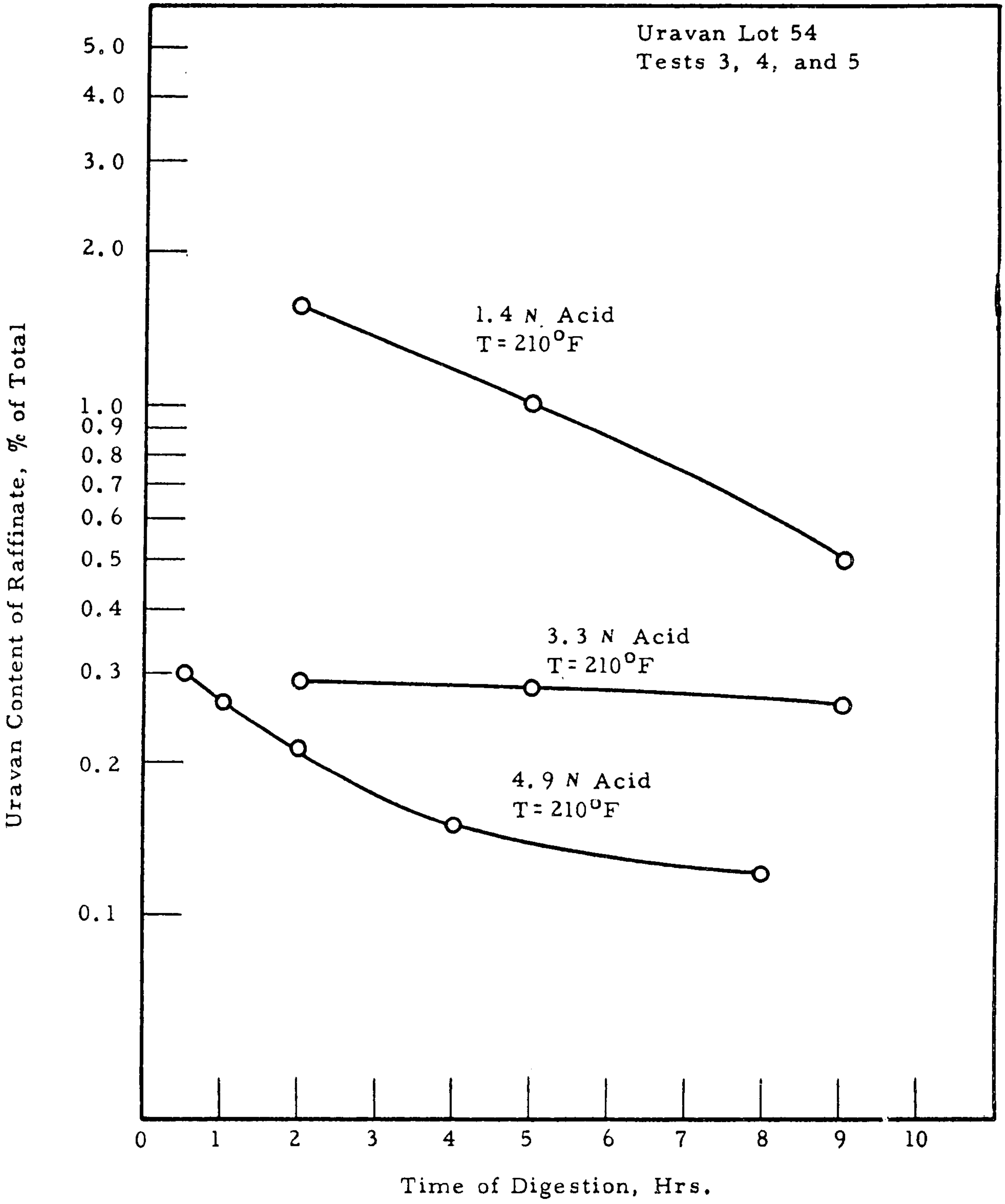
The assay and acid consumption[1] of recent feeds is shown in Table A-I. This data

---

[1] Pounds of HNO_3 to dissolve one pound of ore to zero excess acid.

FIGURE 1

THE EFFECT OF TIME AND EXCESS ACID ON DIGESTION OF URAVAN FEED



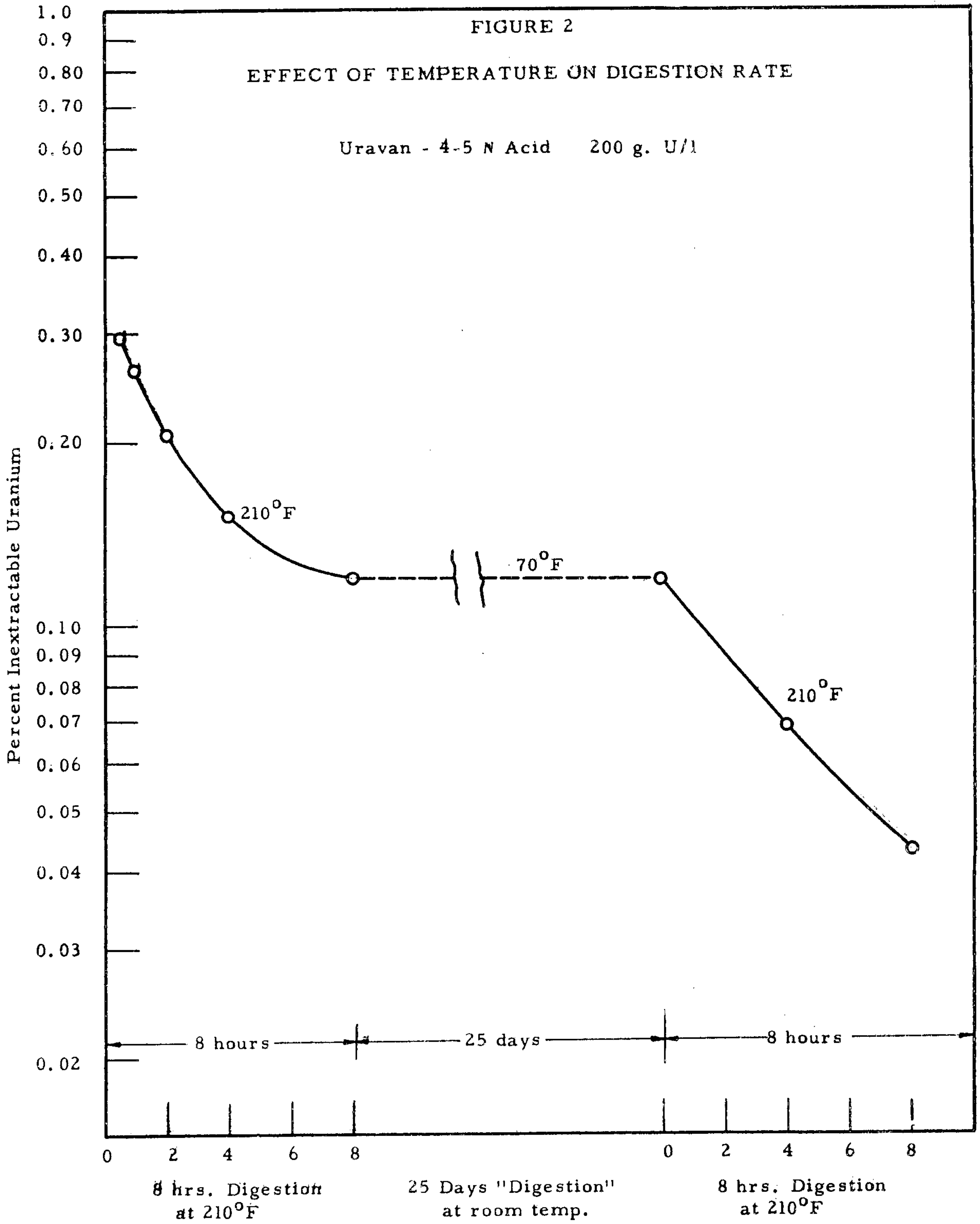


FIGURE 2

EFFECT OF TEMPERATURE ON DIGESTION RATE

Uravan - 4-5 N Acid 200 g. U/l

was obtained from the Research Laboratory and from Plant 6 refinery as noted in Table A-1.

Table A-1

Digestion Data

Feed	Assay %U	Acid Consumption[2] lb HNO_3/lb ore	Source of Data
Anaconda A	70	0.62	Laboratory
Anaconda C	69	.43	Laboratory
Monticello A	58	.34	Plant 6
Edgemont	61	.33	Plant 6
Kerr-McGee	66	.44	Plant 6
Rare Metals	62	.35	Plant 6
Rifle	71	.43	Plant 6
Moab	61	.36	Plant 6
Vitro	62	.51	Plant 6
Climax	68	.47	Plant 6
Durango	74	.64	Plant 6
Uravan	67	.40	Plant 6
Naturita	70	.65	Plant 6
Euxenite	69	.51	Laboratory

Measurements of the absolute density of feeds were made in the pilot plant. These results are shown in Table A-2.

---

[2] Pounds of HNO_3 to dissolve one pound of ore to zero excess acid.

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Table A-2

Absolute Densities of Feeds

Feed	Lot No.	Absolute Density g/cc
Anaconda C	C-788	4.35
Moab	47	4.00
Uravan	542	5.56
Rare Metals	18	3.00
Monticello A	A75	2.94
Kerr-McGee	116	3.33
Anaconda A	846	4.54

B. Assumptions

The following assumptions were made in the analysis of acid requirements:

1) The absolute density of the feed is 4 g/cc.
2) The volume of the acid-insoluble solids may be neglected.
3) The volume of the final slurry is equal to the absolute volume of the feed plus the volume of the acid required for digestion.

C. Sample Calculations

Calculate the acid concentration required to dissolve feed to 200 g U/l, 3*N* excess acid.

Data: Feed = Anaconda A
Density of feed = 4 g/cc
Assay = 70% U
Acid consumption = 0.62 lb HNO_3 / lb. ore

Basis: 100 gal of slurry

$$\text{Weight feed} = (\text{lb U/gal})\,(\text{gal slurry})\,\left(\frac{1}{\text{assay}}\right)$$

$$= \left(\frac{200}{100}\right)(100)\left(\frac{1}{0.70}\right)$$

$$= 238.6\ \text{lbs}$$

$$\text{Volume of feed} = (\text{weight feed})\left(\frac{1}{\text{density}}\right)\left(\frac{1}{8.34\ \text{lb/gal H}_2\text{O}}\right)$$

$$= 238.6\left(\frac{1}{4}\right)\left(\frac{1}{8.34}\right)$$

$$= 7.2\ \text{gal}$$

$$\text{Volume of acid} = \text{volume slurry} = \text{volume feed}$$

$$= 100-7.2 = 92.8\ \text{gal}$$

$$\text{Weight HNO}_3 = \text{HNO}_3\ \text{consumed} + \text{excess HNO}_3$$

$$= (0.62)(238.6) + \left(\frac{189}{120}\right)(100)$$

$$= 305\ \text{lbs}$$

$$\text{Lbs/gal HNO}_3 = \frac{305\ \text{lbs}}{92.8\ \text{gals}} = 3.29\ \text{lbs/gal}$$

$$= 33\%\ \text{HNO}_3$$

$$\text{Gal acid/lb feed} = \frac{92.8}{238.6} = 0.389\ \text{gal}\ 33\%\ \text{HNO}_3\text{/lb feed}$$

PILOT PLANT TESTING OF A LOW-ACID FLOWSHEET FOR THE TBP-HEXANE EXTRACTION PROCESS

by

J. T. Krieg
J. A. Soukup

I. Summary

Monticello-A and Porter Brothers' ore concentrates were tested on the low-acid extraction flowsheet. High uranium losses to the raffinate (1.0 to 5.2 g U/liter) were experienced in the Monticello tests; however, product (NOK) purity was excellent. The converse was true for the Porter Brothers' tests. Uranium in the raffinate was less than 0.02 g U/liter but product purity was poor with shotgun values ranging from 0.04% to 0.09%. Sodium was a principle metallic contaminant in the product in all tests, ranging from 13 to 300 ppm (U_3O_8 basis.)

II. Introduction

The original design of the TBP-hexane extraction process for Weldon Spring was based on a digest liquor (extraction feed) containing 200 g U/l and 3 M HNO_3. Pilot plant development of this process has been progressing since early 1955 and has been reported in previous Process Development Quarterly Reports.

During the past six months a modified flowsheet based on a digest liquor containing 400 g U/liter and 1 M HNO_3 has been tested in the pilot plant.[1] The purposes of the low-acid flowsheet are to reduce operating costs by lowering nitric acid requirements and to increase refinery capacity by increasing the amount of uranium processed per unit volume of feed. Comparison of the two flowsheets is made in Figure 1 and Figure 2. The flowsheets differ only in the extraction and wash steps.

Pilot plant tests of the low-acid flowsheet were made with Monticello-A and Porter Brothers' uranium concentrates during the past quarter and are described in this report.

III. Description of Materials and Equipment

A. Solvent

The solvent used in these tests was 25% to 30% v/v TBP in a hexane diluent.

---

[1] Krieg, J. T., Fariss R. H., Soukup., J. A., and Nerrow D. J., *Process Development Quarterly Report, Part II,* Mallinckrodt Chemical Works, *MCW-1402* (May 1, 1956) p. 55-72.

FIGURE I

LOW ACID FLOWSHEET FOR THE TBP-EXTRACTION CYCLE

(NOK WASH; FLOW RATES ARE RELATIVE)

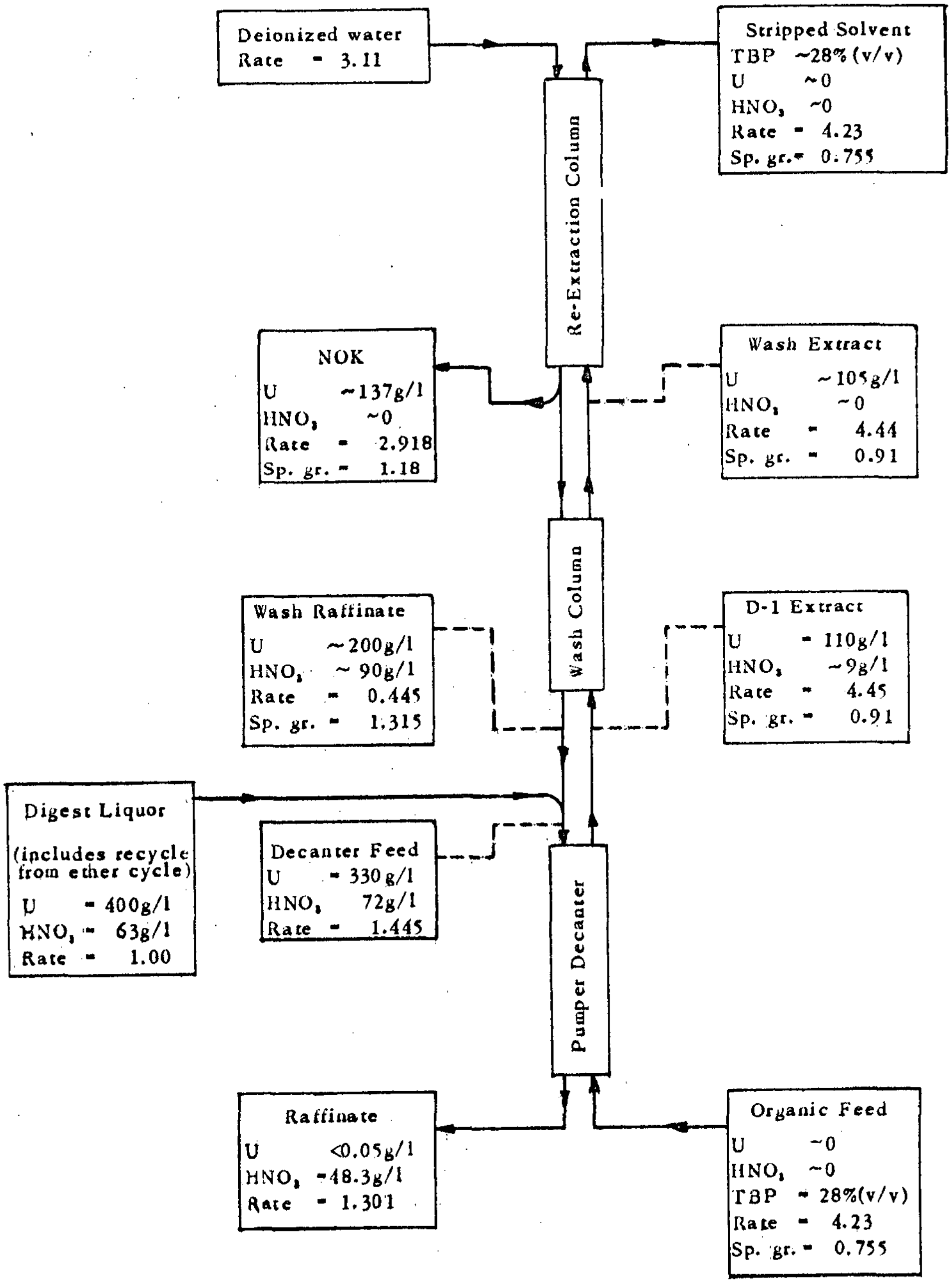
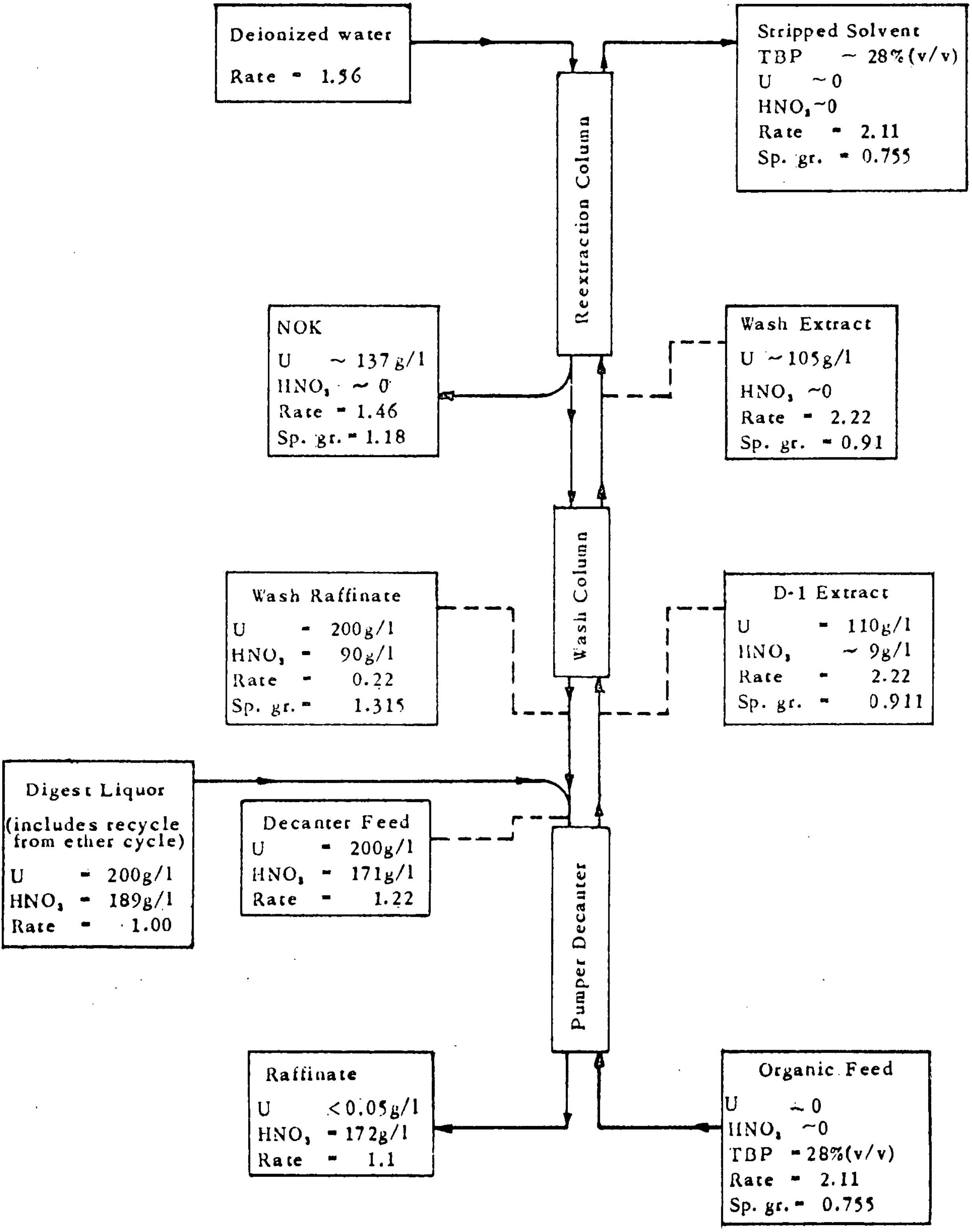


FIGURE 2
NORMAL FLOWSHEET FOR THE TBP EXTRACTION CYCLE
(NOK WASH; FLOW RATES RELATIVE)



B. Feed

Feed materials tested on the low-acid flowsheet this quarter were Monticello A, Lots 74 and 75; and Porter Brothers' concentrate (Euxenite), Lots PSO2, SRO3, and TMO4, (all sodium diuranates). Assays of these feeds are presented below in Table I.

Table I

Assay of Monticello-A and Porter Brothers' Concentrates

	Monticello A[a]		Porter Brothers'[a]		
	Lot 74 Percent	Lot 75 Percent	Lot PSO2 Percent	Lot SRO3 Percent	Lot TMO4 Percent
Cu	nil	nil	-	-	-
As	trace	nil	<0.01	<0.01	<0.01
F	0.03	0.01	0.026	0.008	0.004
Halogen less F	0.01	0.01	0.05	0.08	0.08
P_2O_5	1.51	1.35	<0.09	<0.09	<0.09
V_2O_5	0.02	nil	<0.036	<0.036	<0.036
Water	6.943	7.634	0.84	1.06	0.9
Fe	0.8	0.86	-	-	-
MoO_3	0.16	0.15	<0.01	<0.01	<0.01
SO_4	11.58	11.2	0.10	0.16	<0.04
U_3O_8	69.9	70.05	79.5	79.4	80.8
Ca[b]	0.3	0.78	-	-	-
SiO_2[b]	2.9	4.9	-	-	-
B	-	-	0.0006	0.0008	0.0004
NH_3	-	-	0.008	0.002	0.001
Pb	-	-	0.08	0.006	0.02
$RE_2O_3 + ThO_2$	-	-	4.55	5.65	4.3

[a] Sample on a dry U_3O_8 basis

[b] Sample on an "as received" basis.

Porter Brothers' concentrate is unusual because of its high thorium and rare earths content. The Monticello A contained noticeable amounts of phosphate, sulfate, and calcium which affect the operation of the extraction process.

C. Equipment

1. Pumper Decanters

Four pumper-decanter units were used for the extraction step. The pumper-decanter system is the standard extraction equipment in the pilot plant and is described in detail in previous reports.[2]

2. Pulse Columns

A schematic representation of the pilot plant pulse columns is shown in Figure 3. All columns contain 14 ft. of plate section and are operated with the interface in the top disengaging section. The first pulse column depicted in Figure 3 is a 4-in.-diameter glass column containing $\frac{1}{16}$-in.-thick teflon-coated stainless steel plates with $\frac{3}{8}$-in. holes triangular spaced to give 33% free area. The plates are spaced 4 inches apart. The second pulse column is also 4-in.-diameter glass pipe; however, the plates differ from the preceding column in that they are made of $\frac{1}{8}$-in.-thick teflon. The third column is identical to the first except it is 5-in.-diameter glass pipe. Pulse amplitudes in all columns were set at 2 inches. The columns were so interconnected that $\frac{1}{2}$, 1, 1$\frac{1}{2}$, or 2 columns could be used for washing, and the balance used for re-extraction. The pulse frequency in the wash column (or columns) was 40 to 45 cycles/min.

IV. Experimental Procedure

A. Digestion

Feed materials were digested in 40 wt.percent nitric acid for one to three hours at 200° F, after which they are sampled and adjusted to 1 *M* excess nitric acid and 400 g U/liter. Digest liquors were held for 1 to 24 hours before use in the pilot plant. Chemical requirements for digestions are tabulated below in Table II.

Table II

Chemical Requirements of Pilot Plant Ore Digestions

(400 g U/liter, 1 *M* excess HNO_3 Flowsheet)

Concentrate	Water gal/lb ore	HNO_3 (as 60 wt. percent acid) gal / lb. ore	Final Volume gal/lb ore
Monticello A	0.042	0.059	0.122
Porter Brothers	0.067	0.107	0.188

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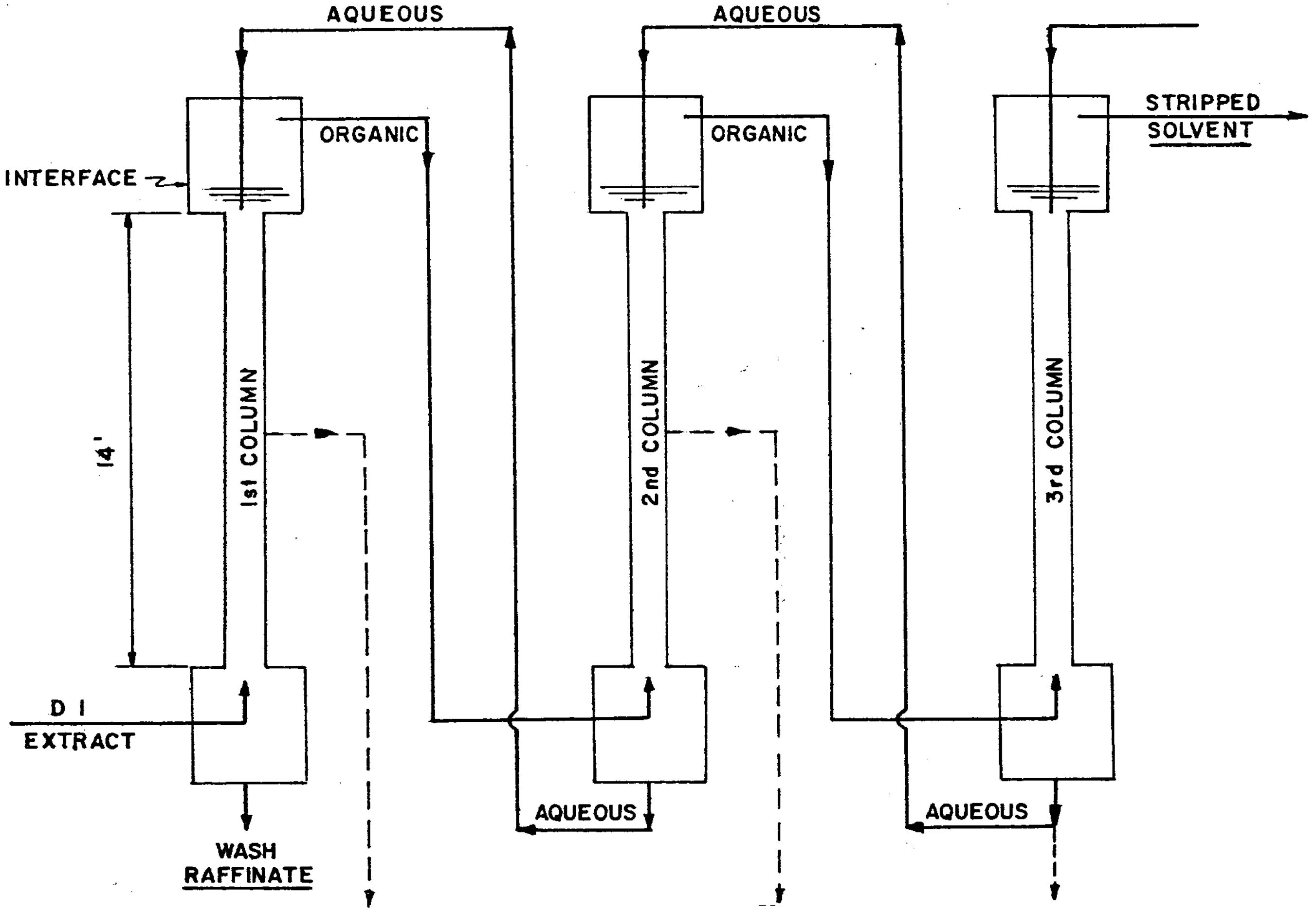
[2] Fariss, R. H., Hemkens, G. C., Krone, C. H., Paul, J. F., and Powell, C. S., *Process Development Quarterly Report, Part II*, Mallinckrodt Chemical Works, *MCW-1381*, (November 1, 1955) p 9-102.

FIGURE 3

PILOT PLANT PULSE COLUMN ARRANGEMENT



PRODUCT (NOK) MAY BE REMOVED
FROM ANY OF THE DASHED LINES

B. Operation of Extraction Equipment

The extraction equipment was operated at steady state for a period of 6 hours before equilibrium was assumed. Operation was extended for an additional 24 hours during which time both composite and spot samples were taken of the process flow streams to determine the uranium loss to the raffinate, extraction efficiency, and product purity. Equilibrium data was obtained by shakeouts of the decanter mixing-pump effluents. In addition to tests on the low-acid flowsheet several runs were made on the high-acid flowsheet for comparison. Uranium flow rates (lb. U/hr. - sq ft.) to the pumper decanters were varied between 1.1 and 2.25 times the Weldon Spring design rates.

V. Experimental Results and Discussion

A run summary of the low-acid flowsheet tests is presented in Table III:

Table III

Run Summary of Monticello-A and Porter Brothers Tests

						Wash Raffinate Contaminants, ppm		Product (NOK) Contaminants, ppm				
Run No.	Flow-sheet	Pumper Decanter Control[a]	Pumper Decanter Mixing Pump Ratio[b]	Percent Uranium Saturation of Pumper Decanter Extract	Uranium Loss to Raffinate g/l	Na (U_3O_8 basis)	K (U_3O_8 basis)	Na (U_3O_8 basis)	K (U_3O_8 basis)	Shot-gun[c]	Thorium Plus Rare Earth g/l	Rate Factor[d]
					Monticello-A							
75	Low Acid	D2, .83	20/1	85%	1.0	-	-	-	-	-	-	2.25
75A	Low Acid	D2, .82	10/1	74%	5.2	500	-	17	-	0.007	-	1.9
75D	Low Acid	D3, .82	20/1	97%	5.4	100	-	13	-	0.017	-	1.9
75E	Low Acid	D3, .82	20/1	97%	6.0	200	-	20	4	0.03	-	1.9
75F	Low Acid	D2, .82	20/1	86%	2.1	-	-	-	5	0.007	-	1.9
75G[e]	Low Acid	D2, .82	20/1	81%	1.5	-	-	-	-	0.03	-	1.9
75H	High Acid	D2, .82	20/1	73%	0.25	-	-	-	-	0.04	-	1.1
					Porter Brothers[g]							
76[f]	High Acid	D2, .82	10/1	77%	<0.025	-	-	80	-	0.03	<0.05	1.1
76A[f]	Low Acid	D2, .82	20/1	89%	<0.025	-	-	-	-	-	-	2.25
76D[f]	Low Acid	D2, .82	10/1	88%	-	2000	-	80	-	0.04	<0.02	1.9
76E	Low Acid	D2, .82	10/1	84%	<0.025	6000	-	300	-	0.09	0.85	1.9
76F[f]	Low Acid	D3, .82	20/1	86%	0.12	1000	8	250	2	0.07	<0.02	1.9
76G[e,f]	Low Acid	D3, .82	20/1	85%	-	5000	40	50	2	0.09	<0.02	1.9

Table III (continued)

[a] The aqueous/organic flow ratio to the pumper decanters is controlled by the density of the extract leaving the second or third decanter stage.

[b] Ratio of solvent to aqueous in the mixing pumps.

[c] A shotgun is the percent increase in neutron absorption cross section due to impurities in the product. The shotgun of 100% pure U_3O_8 is zero by definition.

[d] Rate factor is the ratio of actual rate (lb uranium/sq ft -hr) in the pumper decanter to the present Weldon Spring design rate.

[e] Solvent washing was made with a 5 weight percent K_2CO_3 solution. In all other runs solvent washing was with 5 weight percent Na_2CO_3.

[f] 1 ½ columns were used per washing. In all other runs one column was used for washing.

A. Equilibrium Data

Uranium equilibrium between Monticello-A slurries and the organic phase in the pumper decanters was not as favorable as with other feeds tested on the low-acid flowsheet as shown by Figure 4. This was probably due to the high phosphate and sulfate content of this feed. Not enough data was available to plot a similar equilibrium curve for Porter Brothers; however, uranium equilibrium appeared good as shown by the uranium profile in the pumper decanters tabulated below in Table IV. A thorium profile for the same run, also presented in the table, illustrates the large accumulation of this element.

Table IV

Uranium and Thorium Profiles in the Pumper Decanter[a]

(Extract from the Second Decanter Controlled at a Specific Gravity of 0.82)

Decanter	U g/l	Th[b] g/l	Normal Uranium Profile[c] g U/l
1	97	4	105
2	7.4	23.6	45
3	0.25	4.5	6
4	0.081	3	0.7

[a] Run H 76 E (Porter Brothers')

[b] Estimated from uranium concentration density of extract solution.

[c] In runs without the presence of thorium.

The above table illustrates the salting effect of thorium in the runs made with Porter Brothers' concentrate.

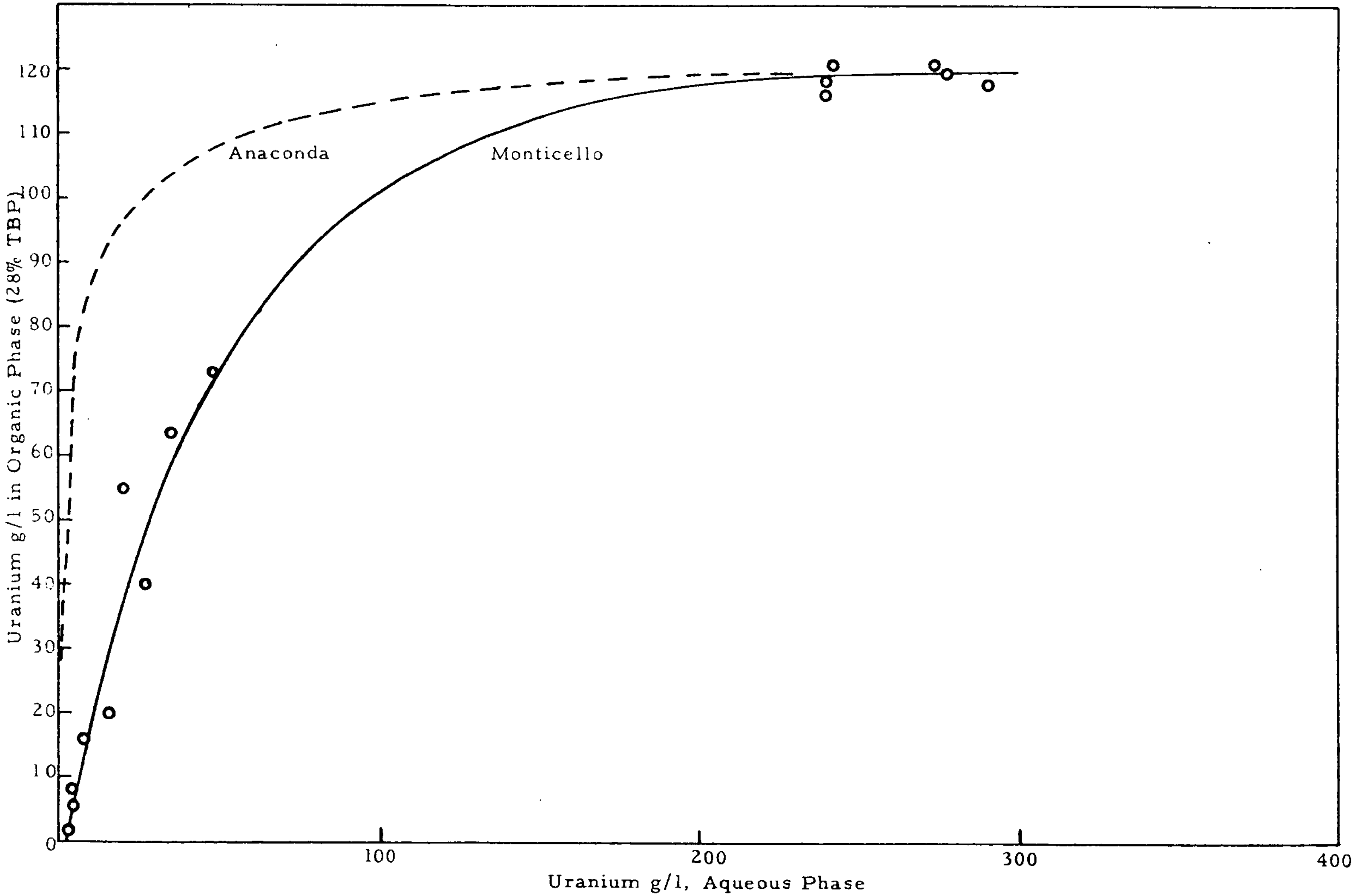
B. Mechanical Operation

1. Density Control

In normal pilot plant operation the aqueous-feed-to-organic-feed flow ratio is controlled by the density of the extract leaving the second or third decanter. In the runs made with Porter Brothers' concentrate, however, the control system did not

FIGURE 4

URANIUM EQUILIBRIUM DIAGRAM FOR PUMPER-DECANTERS



function properly because of the large accumulation of thorium mass at the density control point. Because the density-control system reads all mass as uranium, it called for an excessive solvent-to-aqueous flow ratio which reduced the solvent-phase uranium profile in the decanters. This effectively lowered the extract uranium saturation as well as the uranium loss to the raffinate.

2. Scaling

No scaling in the pilot plant equipment could be detected when processing Porter Brothers' concentrate; however, mild scaling occurred in the decanters when processing Monticello-A. More serious scaling occurred in the raffinate transfer lines, pumps, and steam stripper. On several occasions during the ten day series of Monticello tests, the pilot plant had to be shut down because of plugged raffinate lines. The scale is believed to have been calcium sulfate since this material was present in unusually large quantities in the feed material (Table I). In feed lot 75 the calcium and sulfate combination was above MCW tolerance.

3. Phase Dispersion

As with previous feeds[3] tested on the low-acid flowsheet, Monticello-A ran best at a 20:1 mixing-pump ratio (flow ratio of solvent to aqueous in the mixing pumps.) Increasing the mixing pump ratio from 10:1 to 20:1 increased the extract saturation from 74% to 85% (Table III, Run 75 and 75A). The same effect was observed with Porter Brothers' concentrates; however, at a 20:1 mixing-pump ratio the aqueous phase was too finely dispersed and would not disengage fast enough, causing incipient flooding of the decanters. This occurred at a superficial uranium flow rate of 2.25 times the corresponding Weldon Spring flow rate (present design basis).

Operation during the Monticello tests was unusual in that there was a conspicuous absence of crud on the column interfaces. In both the Monticello and Porter Brothers' tests no troublesome emulsions occurred at the decanter interface.

C. Extraction Performance

1. Raffinate loss

In the Monticello low-acid flowsheet tests uranium losses to the raffinate were high, ranging from 1 to 2.1 g U/liter at optiumum conditions (density control on second decanter and a mixing-pump phase ratio of 20:1 solvent to aqueous). In a

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[3] *Op. cit., MCW-1402.*

similar test on the high-acid flowsheet the uranium in the raffinate was 0.25 g U/l. The unfavorable uranium distribution coefficients (due to the complexing effects of phosphate and sulfate in the Monticello-A material) were responsible for the high loss to the raffinate. Uranium loss to the raffinate using Porter Brothers' concentrate with either flowsheet was extremely low (usually less than 0.025 g U/l). The salting action of the thorium together with the excessive solvent-to-aqueous flow ratio in the decanters, also the result of thorium in the system (Part B - Density Control), was responsible for the low uranium loss to the raffinate.

2. Purity

The product (NOK) purity of the Monticello-A tests on the low-acid flowsheet was good with shotgun values ranging from 0.007% to 0.03%. The high-acid-flowsheet yielded a product liquor having a shotgun value of 0.04%. The uranium saturation of the pumper-decanter extract ranged from 73% to 97% with the high-acid flowsheet yielding the 73% value; however, no general correlation is evident between the extract saturation and the product purity for this series of runs. Spectrographic analysis of the product liquor from Monticello-A tests are tabulated below in Table V.

Table V

Spectrographic Analyses of NOK from Monticello Tests

(Ppm Impurities U_3O_8 Basis)

	Low Acid Flowsheet			High Acid Flowsheet
Run	75A	75D	75E	75H
Ag	<.1	<.1	<.1	<.1
Al	<10	<10	<10	<10
As	<10	<10	<10	<10
B	<.1	<.1	<.1	.35
Be	<.1	<.1	<.1	<.1
Bi	<1	<1	<1	<1
Co	8	4	3	3.3
Cr	2	2	2	<1
Cu[a]	<1	<1	<1	1
Fe	6	11	11	–
In	<.5	<.5	<.5	<.5
K	-	2.	4	—
Mg	27	13	10	33
Mn	<10	<10	<10	<10
Mo	10	<10	<10	<10
Na	17	13	20	—
Ni	<1	<1	<1	2
P	<50	<50	<50	<50
Pb	<1	<1	<1	1
Sb	<1	<1	<1	<1
Si	18	15	<15	50
Sn	<1	<1	<1	<1
V	<15	<15	<15	<15
Zn	<15	<15	<15	18

[a] Chemical Determination

Sodium contamination is discussed under a separate heading below.

The purity of the product obtained from the Porter Brothers' tests on the low-acid flowsheet was poor with shotgun values ranging from 0.03% to 0.09%. The uranium saturation of the decanter extract ranged from 77% to 89%, the lowest saturation again occurring using the high-acid flowsheet. The low extract saturations were caused by the excessive solvent-to-aqueous flow ratio in the decanters (Part B). As with the Monticello tests, no correlation was evident between extract saturation and the product purity. Spectrographic analyses of the product liquor from the Porter Brothers'test are tabulated below in Table VI. Principle contaminants were thorium, sodium and possibly rare earths (a potential source of the high shotgun values).

Table VI

Spectrographic Analyses of NOK from Porter Brothers' Tests

(Ppm Impurities, U_3O_8 Basis)

	High Acid Flowsheet	Low Acid Flowsheet			
Run	76	76D	76E	76F	76G
Ag	.1	<.1	<.1	<.1	<.1
Al	<10	<10	<10	<10	<10
As	<10	<10	<10	<10	<10
B	.22	<.1	.14	.15	<.1
Be	<.1	<.1	<.1	<.1	<.1
Bi	<1	<1	<1	<1	<1
Co	<1	<1	15	3	2
Cr	<1	<1	<1	1	1
Cu	1	1	1	<1	1
Fe	15	<15	<15	<15	<15
In	<.5	<.5	<.5	<.5	<.5
K	-	-	-	2	2
Mg	30	10	<10	10	<10
Mn	<10	<10	<10	<10	<10
Mo	<10	<10	<10	<10	<10
Na	-	-	-	250	1000

Table VI (continued)

	High Acid Flowsheet	Low Acid Flowsheet			
Run	76	76D	76E	76F	76G
Ni	1	<1	1	<1	<1
P	<50	<50	<50	<50	<50
Pb	1	<1	<1	<1	<1
Sb	<1	<1	<1	<1	<1
Si	30	<15	<15	<15	<15
Sn	<1	<1	<1	<1	<1
Th + RE	-	<200	8400	<200	<200
V	<15	<15	<15	<15	<15
Zn	<15	<15	<15	<20	<15
Ce	-	<6	-	<5	<5
Dy	-	<13	-	<10	<10
Er	-	<3	-	<2.5	<2.5
Eu	-	<1.2	-	<0.8	<0.8
Gd	-	<4	-	<3.3	<3.3
Ho	-	<2	-	<1.6	<1.6
La	-	<5	-	<4	<4
Nd	-	<3	-	<2.5	<2.5
Pr	-	<3.0	-	<2.5	<2.5
Sc	-	<3.0	-	<2.5	<2.5
Sm	-	<1.0	-	<0.8	<0.8
Tb	-	<3.0	-	<2.5	<2.5
Yb	-	<7.0	-	<5	<5

3. Sodium Decontamination

Sodium is a predominant metallic impurity in most MCW feed materials, (sodium diuranates). Sodium likewise appears to be the most abundant metallic impurity in the product liquor, ranging from 13 to 20 ppm (U_3O_8 basis) in the Monticello tests, 20 to 133 ppm in Anaconda tests, and 50 to 300 ppm in Porter Brothers' tests. The deionized-water feed to the reextraction column cannot be blamed for all the sodium detected in the NOK liquor because copious quanties of the contaminant were detected in the wash raffinate (100 to 500 ppm in the Monticello tests, 700 ppm in Anaconda tests, and 1000 to 6000 ppm in the Porter Brothers' tests). Since recycled NOK liquor is used for washing, the sodium must have entered the wash column from the pumper decanters. The exact mechanism for this sodium transfer *via* the solvent phase is not known; however, it is believed to be sodium-bearing aqueous entrainment. Other sites[4,5] using TBP extraction have reported similar phenomena with sodium.

When potassium carbonate was substituted for sodium carbonate in the solvent treatment step, no adverse change in sodium or potassium decontamination could be detected as shown in Table VI, Runs 76F, 76G, 75F and 75G.

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[4] Verbal report from Hanford.

[5] Verbal report from National Lead of Ohio.

SENSITIVITY OF THE WELDON SPRING TBP-EXTRACTION SYSTEM TO PROCESS FLOW RATES

by

J. T. Krieg
J. A. Soukup
G. B. Wills

I. Summary

The maximum tolerable variation of flow ratio, A/O, (volume of aqueous feed to volume of organic feed) in the Weldon Spring pumper-decanters is predicted to be ± 5%. Beyond these limits of control either the uranium loss to the raffinate will be excessive or the product liquor (NOK) may not meet purity specifications. The optimum flow ratio is a function of the composition of the feed and the TBP content of the solvent.

The tolerable variation of wash-column A/O (flow ratio) is expected to be between ± 5 and ± 10%; however, the consequences of variations in flow ratios beyond these limits are not as severe as in the pumper-decanters. Too high a flow ratio will slightly decrease capacity by recycling excessive amounts of uranium and diluting the product liquor (NOK). Too low a flow ratio may decrease the purity of the product liquor.

The flow ratio in the reextraction columns should be controlled within ± 5% of the optimum value which is a function of the TBP and uranium content of the organic feed. Beyond these limits of control either the NOK product would be unnecessarily diluted (an economic disadvantage) or else the stripped solvent will contain excessive amounts of uranium, a factor which induces high uranium loss to the pumper-decanter raffinate.

II. Introduction

The operation of an extraction system, such as the TBP process at Weldon Spring is sometimes quite sensitive to variables such as process flow rates. If these variables are not controlled within definite limits, the extraction system will not function properly. The ratios of aqueous to solvent flows in the extraction equipment are such variables. This report analyzes the theoretical limits within which these ratios must be controlled for satisfactory operation of the process.

III. Description

The process flow scheme for the TBP extraction cycle is presented in Figure 1. Uranium is extracted out of the aqueous phase by counter-current contact with solvent (28% TBP in hexane) in the pumper-decanters. It is then washed for further purification with water in a pulse column. This is followed by a reverse extraction of the uranium back into an aqueous stream in a pulse column. The variation of flow ratios in these three operations are discussed herein.

IV. Discussion

A. Pumper-Decanters

The relationship between flow ratio (volume of aqueous feed to volume of solvent feed) and pumper-decanter performance is shown in Figure 2. This curve was constructed by calculating the uranium concentration in the raffinate and in the extract (the latter expressed as percent saturation) by a series of material balances at various flow ratios. In this analysis the uranium saturation of 28% TBP in hexane was assumed to be 113 g U/liter. For a pumper-decanter system containing an infinite number of stages, the curves in Figure 2 would intercept the "100% saturation" ordinate and the "0 g/l" ordinate; with the 5-stage system at Weldon Spring the curves become asymptotic at these ordinates. The values of these curves above about 98% saturation and below about 2 g U/liter are not known. They are functions of the mass transfer coefficient and equilibrium characteristics of each individual feed batch.

These curves demonstrate that an excessive flow ratio will result in high uranium loss to the raffinate; while an insufficient flow ratio will lower uranium saturation (thereby increasing the impurity level) in the extract. Recent pilot plant studies[1] have demonstrated that usually no significant variation of product purity can be detected within the range of 91% to 100% uranium saturation in the extract; however, somewhere below this range purity decreases with decreasing percent saturation. If unusually large amounts of rare earths or thorium are present in the feed material, it may be necessary to operate above 95% uranium saturation to maintain product purity.

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[1] Fariss, R. H., Krieg, J. T., Soukup, J. A., *Process Development Quarterly Report, Part II*, Mallinckrodt Chemical Works, *MCW-1402*, (May 1, 1957) p.21-53

FIGURE - I

TBP HEXANE EXTRACTION CYCLE

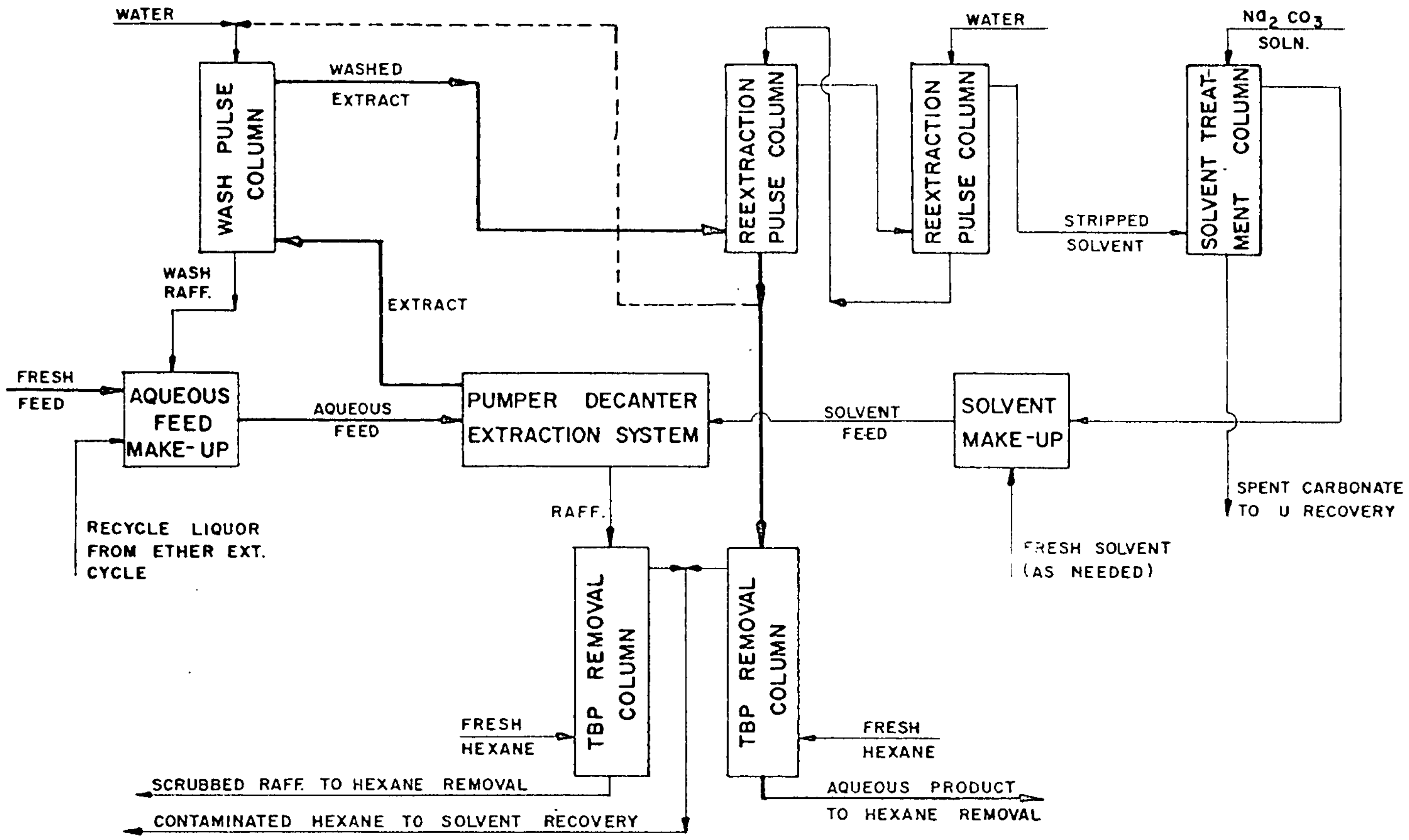
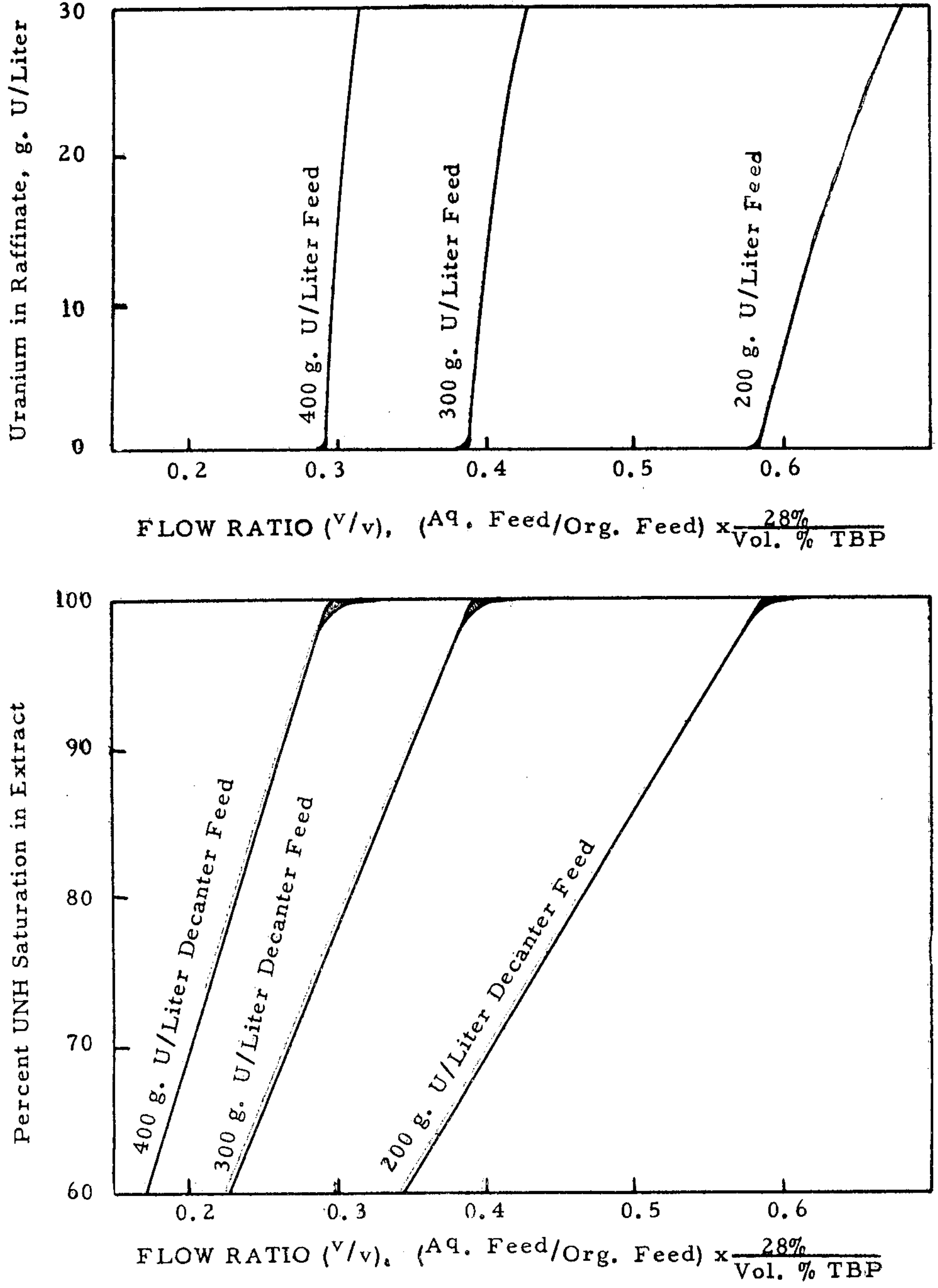


FIGURE 2

THE EFFECT OF FLOW RATIO ON PUMPER-DECANTER OPERATION



Based on the data in Figure 2, it is necessary to control the pumper-decanter flow ratio within the limits set in Table I. These limits would be narrower if difficultly extractable, or high-thorium and rare-earth feeds were used.

Table I

Tolerable Limits of Pumper-Decanter Flow Ratio
(Based on 91% minimum saturation of extract)

Feed Concentration g U/l	Flow Ratio, Aqueous Feed/Organic Feed Maximum	Minimum	Variation
200	0.59	0.53	± 5%
300	0.393	0.355	± 5%
400	0.295	0.265	± 5%

B. Wash Column

A study of the effect of wash column flow ratio on NOK purity was made recently in the pilot plant. Runs were made with flow ratios of 0.1 to 1, 0.175 to 1, and 0.25 to 1, NOK wash to extract. Anaconda C feed materials were used with the low acid flowsheet[2] and all process variables, with the exception of wash-column flow ratio, were held as constant as possible. With the exception of sodium and nitric acid contaminants, no significant change in NOK (product liquor) purity was detected as illustrated in Table II.

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[2] *Ibid,* p 55-75

Table II

NOK Purity as a Function of Wash-Column Flow Ratio
(NOK Wash)

NOK Assay	A/O = 0.1 to 1	A/O = 0.175 to 1	A/O = 0.25 to 1
U (g/l)	126	105	71
HNO_3/U w/w	0.25	0.1	0.2
Al ppm (U basis)	<10	10	<10
B	<.1	0.12	0.14
Co	<1	3	3
Cr	<1	<1	<1
Cu	<1	<1	1.5
Fe	3	9	3
Mg	<10	28	15
Na	25	175	157
Ni	1	<1	<1
P	<50	<50	<50
Pb	<1	<1	2
Si	<15	22	28
V	<10	-	<10
Zr	15	-	<10
Shotgun[a] (average of four)	0.005%	.015%	.007%

[a] A "Shotgun" is the percent increase in neutron capture cross-section due to impurities in the product. The shotgun of 100% pure U_3O_8 is zero.

As might be expected, the nitric acid and uranium concentrations of NOK decreased with increasing wash-column flow ratios. The increase of sodium contamination with flow ratio is unexplainable and contrary to known equilibrium data. A more detailed discussion of sodium contamination occurs elsewhere in this volume.[3]

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[3] *Cf.*, p.25

Since a higher uranium concentration in NOK liquor reduces costs by minimizing uranium recycle and conserving evaporation costs, the optimum wash-column flow ratio (A/O) for this series of runs was 0.1 to 1.

C. Reextraction Columns

The effect of reextraction flow ratio (water to stripped solvent) on the uranium content of the NOK (product) and stripped solvent is presented in Figure 3 which is based on the relationship.[4]

$$\text{Height of column} = \text{HTU} \int_{y_2}^{y_1} \frac{dy}{y - y^*}$$

where an HTU is the height of a transfer unit. Y_2 and Y_1 are the organic phase uranium concentrations at the top and bottom of the column and y^* is the organic phase uranium concentration in the equilibrium with the aqueous phase at some point in the column. The value of an HTU was assumed to be constant at 2.86 ft. over the range of flow ratios investigated. This value is based upon observed pilot plant efficiencies[5] ranging from 2.2 to 3.3 ft. The calculations further assumed a value of 28% V/V TBP and105 g U/l in the loaded solvent (Y_1). Equilibrium data for 130°F was obtained by a method outlined in a previous report[6] and is presented in the appendix. The two reextraction columns at Weldon Spring are situated such that they may be operated either in series or in parallel so Figure 3 was constructed for both cases.

A very low uranium concentration in the stripped solvent is necessary to minimize uranium losses to the pumper-decanter raffinate, while a high uranium concentration in the NOK (product stream) is desirable to conserve steam in the triple-effect evaporator. As demonstrated by Figure 3, with the columns operating in series the optimum flow ratio (water to stripped solvent) would be about 0.72:1 to 0.80:1 or 0.76 ±0.04 since this would result in low uranium loss to the stripped solvent (<0.05 g U/liter) without unnecessarily diluting the uranium in the NOK stream. With the columns in parallel, undesirable results are obtained regardless of flow ratios.

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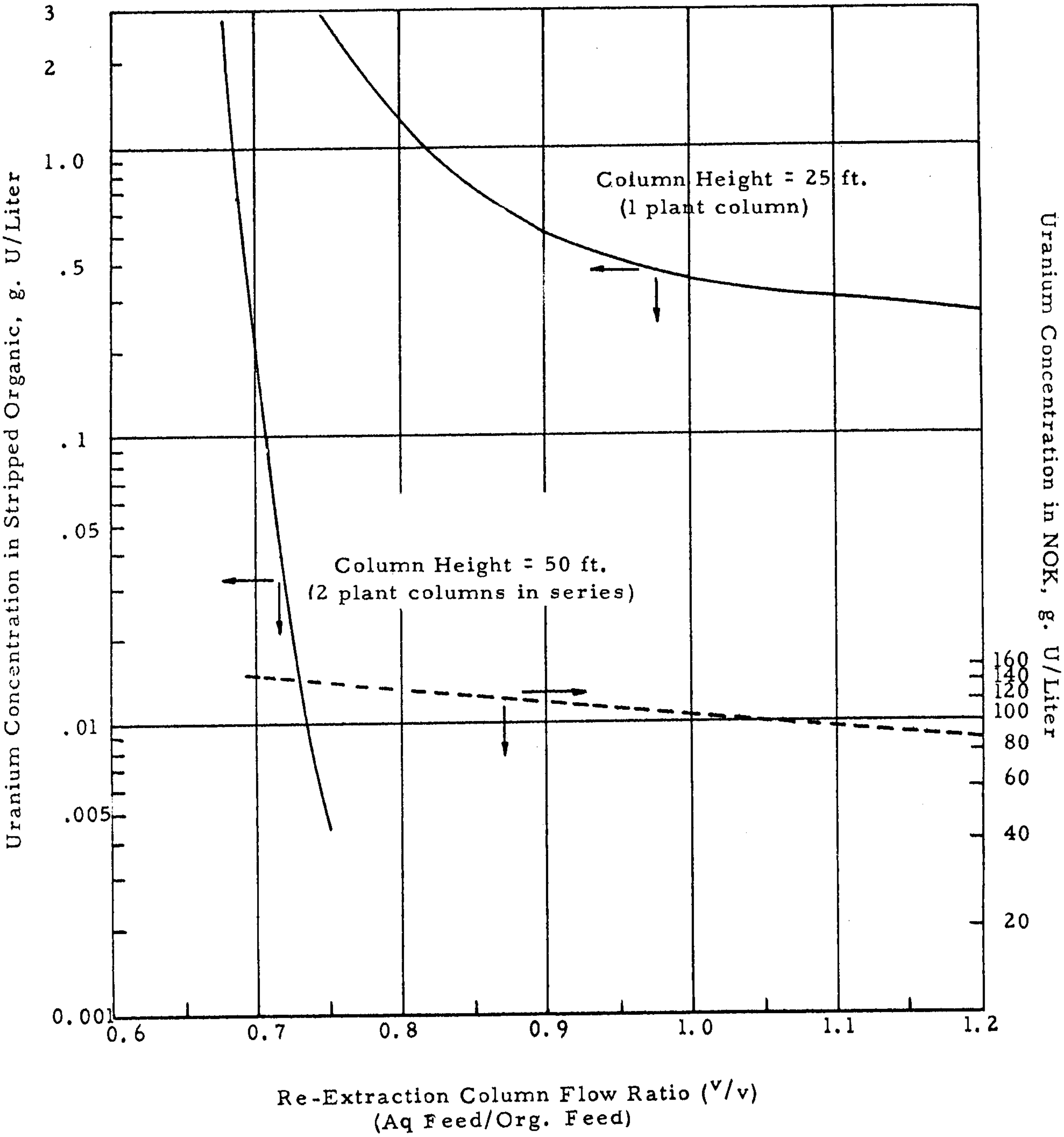
[4] Colburn, A. P., *Ind. Eng. Chem.*, *33* 459 (1941)

[5] Fariss, R. H. Krone, L. H. and Nerrow, D. J., *Process Development Quarterly Report, Part II*, Mallinckrodt Chemical Works, *MCW-1393* (August 1,1956) p. 37

[6] Fariss, R. H., *Process Development Quarterly Report, Part II*, Mallinckrodt Chemical Works, *MCW-1383* (February 1, 1956) p. 10-48

FIGURE 3

THE EFFECT OF RE-EXTRACTION COLUMN FLOW RATIO ON UNH CONCENTRATION OF NOK AND STRIPPED SOLVENT



V. Appendix

Equilibrium data for the reextraction columns at 130°F were calculated by the methods described in *MCW-1383*[7] and are presented below in Figure A-1.

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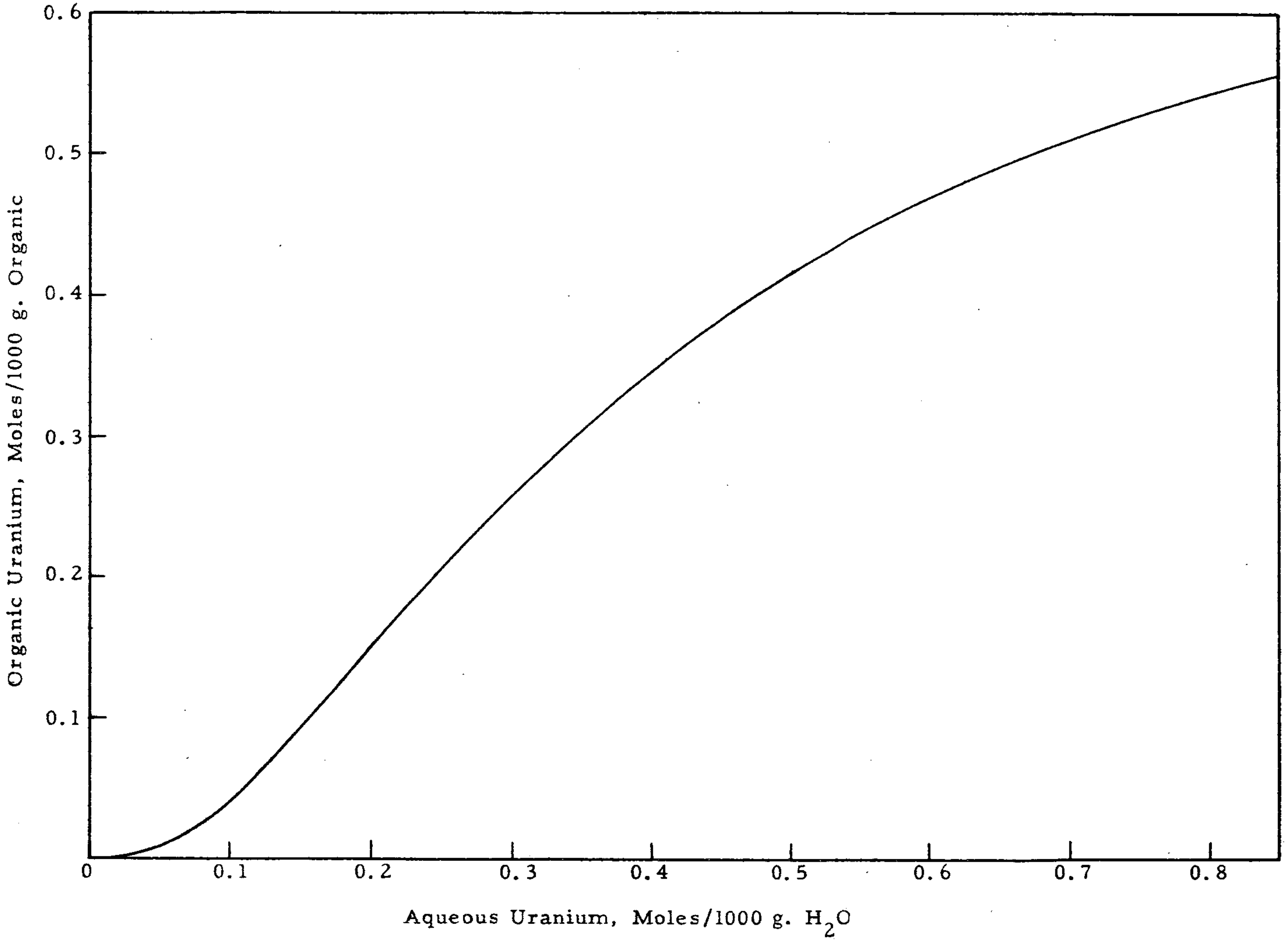
[7] Fariss, R. H., *Process Development Quarterly Report, Part II,* Mallinckrodt Chemical Works, *MCW-1383* (February 1, 1956) p. 10-48

FIGURE A-1

EQUILIBRIUM DATA FOR $UO_2(NO_3)_2$ - H_2O - 28% TBP IN HEXANE



TBP REMOVAL FROM RAFFINATE

by

J. A. Soukup

I. Summary

A series of pilot plant experiments are described for removing TBP from raffinate. Steam distillation of the raffinate slurries proved to be more effective than washing with hexane in a pulse column. No TBP decontamination was detected when thick raffinate slurries containing TBP were contacted with hexane in an open column.

II. Introduction

Because raffinate will be boiled down for acid recovery at Weldon Spring, it is important to remove all organic contaminants beforehand so as to minimize the opportunity for a pyrophoric nitrate-organic reaction to occur.

In view of the high TBP content of recent pilot plant raffinates various methods of TBP removal were studied. These included:

1. Hexane scrubbing of raffinate in an open column, either phase continuous.
2. Hexane scrubbing of raffinate in a pulse column, either phase continuous.
3. Steam distillation of TBP from raffinate.
4. Batch washing of raffinate with hexane.

III. Description of Equipment

The open column used in the experiment was a 4-inch-diameter Pyrex-glass column with 10 ft of straight pipe section. Hexane was continuously recycled from a tank (approximately 20 inches by 20 inches), through the column, and back into the tank.

The pulse column was a 4-inch-diameter Pyrex-glass column consisting of 15 ft of Teflon-coated-stainless steel plates. The plates contained 33% free-area on a four-inch spacing.

The vessel used for steam distillation was a 36-inch by 36-inch stainless steel tank equipped with coils supplied with 100 psi steam.

The vessel used in the batch washing operation was a 34 inch by 34 inch stainless steel tank equipped with a recycle line and an agitator.

IV. Experimental Procedure

Test were made using a synthetic raffinate; *i.e.*, a feed composed of Moab, Monticello A, and Porter Bros. raffinates concentrated to a slurry containing 1.5 lb. solids/gal. Approximately 1.0 g TBP/1 was present in the synthetic raffinate.

A. Open Column

The synthetic raffinate was fed to the open column at 125 gph (1440 gph/sq. ft.). This was equivalent to 1.1 times Weldon Spring superficial flow rates. Hexane scrub was continuously recycled countercurrent to the raffinate at a flow ratio of 1/1. Tests were made with the interface both at the top and bottom of the column. After one-half hour duration at steady state conditions five-liter samples were taken of the raffinate to determine how much TBP had been removed.

B. Pulse Column

Similar tests were made in a pulse column. The pulse mechanism to the column was set in such a manner that the pulse amplitude was two inches in the column and the pulse frequency 25 cpm. Flow rates were the same as those used in the open column. Trials were again made with the interface at the top and at the bottom. The sampling procedure and test duration was the same as in the open column test.

C. Steam Distillation

Distillation of the synthetic raffinate was made at a temperature between 115° - 120°F. Boiling was continued until the liquid volume was reduced to coil level. Samples were taken for TBP analyses at various intervals during the course of boiling.

D. Batch Washing

Synthetic raffinates containing 1.5, 1.0, and 0.5 lb solids/gal were agitated with hexane for one-half hour at a phase ratio of 1/1. Settling times for the raffinates were measured.

E. Analytical Procedure

TBP determinations in raffinate were made by hexane extraction. The extract was saturated with uranium which functioned as a tracer for the TBP. This procedure is described in detail in a previous quarterly report.[1]

V. Results

A. Open Column

No TBP decontamination could be detected across the column regardless of which phase was operated as the continuous one. The column was stable at the 125 gph feed rate.

B. Pulse Column

With the aqueous phase operated as continuous, a TBP decontamination factor of four was achieved across the column, leaving 0.25 g TBP/l in the scrubbed raffinate. No flooding of the pulse column was observed at the flow rates used. By visual estimation the best mixing occurred with the hexane as the continuous phase (interface at the bottom of the column).

C. Steam Distillation

Steam distilling was an effective means of removing TBP from raffinate. The TBP concentration is tabulated below as a function of the raffinate volume during the course of boiling.

Raffinate Volume Percent of original	Test 1 TBP g/l	Test 2 TBP g/l	Test 3 TBP g/l
100	0.8	0.8	0.8
83	.5	.8	.2
67	.3	.6	.08
50	.2	.2	.1
40	–	.14	–

By the mechanism of steam distillation the mole ratio of TBP/distillate should be equal to the vapor pressure of TBP divided by the vapor pressure of the distillate. If this relationship applied in the above test, all the undissolved TBP should have been distilled by the time the raffinate had only concentrated to 97% of its original volume; however the raffinate had been concentrated to between 50 and 80% of its

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[1] Fariss, R. H., Krieg, J. T., Powell, C. S., and Soukup, J. A., *Process Development Quarterly Report, Part II*, Mallinckrodt Chemcial Works, *MCW-1398* (February 1, 1957) p. 46.

original volume before all insoluble TBP had been distilled off. (The solubility of TBP in water at 100°C is about 0.2 g /l).

D. Batch Washing with Hexane.

No TBP decontamination data is available for this test, however, the settling times for the raffinate slurries are tabulated below.

Raffinate	Solid Content lb/gal	Settling Time
Synthetic	1.5	>8 hours
Synthetic	1.0	>8 hours
Synthetic	0.5	30 minutes

PROPERTIES OF URANIUM DIOXIDE

by

W. Hedley

Summary

During the last quarter an atomic hydrogen arc has been used to melt mixtures UO_2 and Al_2O_3. The rods formed had hardnesses between 9 and 10 on Moh's scale. Up to 6 w/o Al_2O_3 was used with theoretical densities of up to 91% being achieved. This decreased density (as compared with earlier work) is apparently caused by trapped air. The cure is believed to lie in better mechanical controls.

Introduction

The object of the flame fusion project is to melt UO_2 to form dense rods of relatively constant cross section. The heat source which was experimented with during this last quarter was the atomic hydrogen arc. Mixtures of Al_2O_3 and UO_2 were used as material from which to grow rods.

Experimental Results and Discussion

Previous experiments in flame fusion of UO_2 were combinations of C_2H_2, CH_4, and H_2 with oxygen as the source of heat. Mixtures of Al_2O_3 and UO_2 as well as pure UO_2 were melted. However, because it was difficult to produce the required high temperature uniformly over the cross section of the seed, the rods produced were irregular in shape.

The atomic hydrogen arc has since been used to grow rods of UO_2 and $UO_2Al_2O_3$ mixtures. A picture of three such rods is shown in Figure 1.

The rod containing 6 w/o of Al_2O_3 in UO_2 was ⅝ inch in diameter, 1 inch long, and weighed 53 grams. The rod containing 2 w/o of Al_2O_3 in UO_2 was ½ inch in diameter, 1½ inches long, and weighed 68 grams. The pure UO_2 rod was ½ inch in diameter, ⅝ inch long, and weighed 26 grams. Each of these rods had a hardness between 9 and 10 on Moh's scale. The bulk densities of these rods were measured by the mercury immersion method. These densities and the percent of the theoretical densities are reported in Table I.

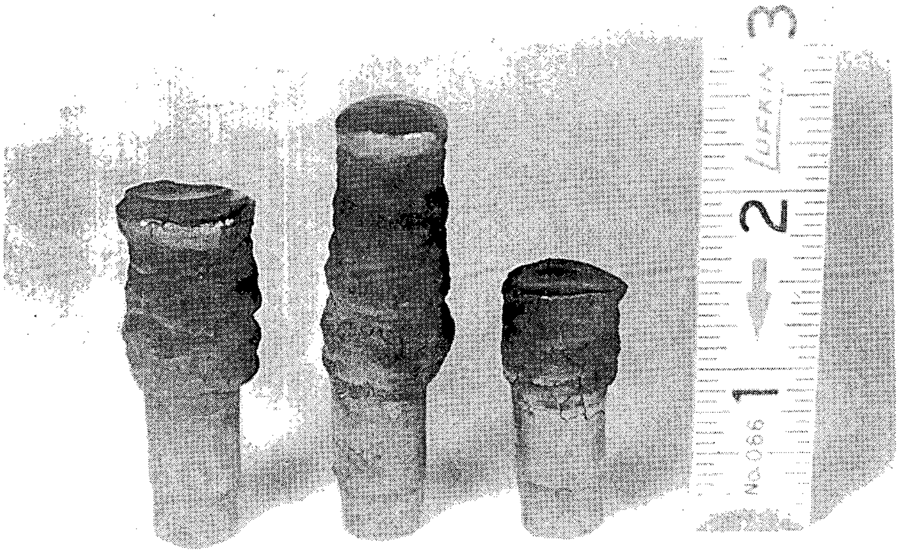

Figure 1

Flame Fused Rods

From left to right: (1) 6 w/o Al_2O_3 - 94 w/oUO_2
(2) 2 w/o Al_2O_3 - 98 w/oUO_2
(3) pure UO_2

Table I

Densities of Flame Fused Rods

Material	Measured Density g/cc	Percent of Theoretical	Percent of Pure UO_2
Pure UO_2	9.43	86.0	86.0
2 w/o Al_2O_3 in UO_2	9.69	91.4	88.3
6 w/o Al_2O_3 in UO_2	9.04	91.0	82.4

The theoretical densities of the mixtures were calculated by dividing the weight fraction of the components by their true particle densities and taking the reciprocal of the sum of these numbers.

The true particle densities used were 10.97 g/cc for UO_2 and 3.99 g/cc for Al_2O_3. This method of calculation assumed that the two constituents did not interact when they melted together. These densities are lower than the 100% of theoretical previously reported.[1]

When the samples were broken open, it became apparent that the reason for the lower densities of these rods was due to included air bubbles which were large enough to be seen with the naked eye. This air was apparently trapped under the small piles of UO_2 fed on to the top of the seed, the arc having melted the material on top and sealed the air underneath. It is believed that this can be avoided if the feed is continuous instead of intermittent. Uniform continuous feeding will require steadier growing conditions than have been achieved to date, particularly with respect to stability of the arc.

It is planned that work with the atomic hydrogen arc will continue. Efforts will be concentrated on the improvement of positioning mechanisms to insure that the powder is fed to the most favorable spot. Better arc control will be attempted with revised instrumentation.

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[1] Henderson, C. M., *Process Development Quarterly Report, Part II*, Mallinckrodt Chemical Works, *MCW-1398* (November 1, 1956), p. 121

UO_2 FUEL ELEMENTS

by

R.B. Wrinkle

Summary

Rods and tubes which were extruded from micronized UO_2 had excellent surface quality and sintered densities greater than 96% of theoretical. It was found that extrusion dies with 40° and 50° lead-in angles gave nearly equal results in extruding micronized UO_2 powder. Rods which were extruded from a blend of fuel-element-grade UO_2 and micronized UO_2, and rods which were extruded from UO_2 made from ammonium diuranate had lower sintered densities than those made from micronized UO_2 alone.

Introduction

Experimental work has been concentrated on an evaluation of highly sinterable types of UO_2 powder for possible use in extrusion processes. Extrusion experiments were performed with three types of UO_2 powder, both rod and tubular type fuel elements having been extruded. Sintered densities and surface qualities were the principle characteristics used to evaluate the extruded samples.

Experimental Results and Discussion

Micronized UO_2 was extruded into rods using dies with lead-in angles at 40° and 50° in an attempt to evaluate the effect of this variable on rod quality. Extruded samples were allowed to dry in air for at least one day and where then faced off at the ends. The densities of the air dried samples were calculated by dividing the weight of each sample by its volume as determined by micrometer measurements. The samples were then sintered at 1680°C for 17 hours in hydrogen after which the sintered densities of the samples were determined by a mercury displacement method. The extrusion data and densities of these rods are shown in Table I as Mix 1 and Mix 2. A representative rod from Mix 1 is illustrated in Figure 1. Micronized UO_2 was also extruded into tubes to determine if the extrusion technique used for rods would yield satisfactory tubes. The extrusion data and densities of these tubes are shown in Table I as Mix 3. A representative tube from Mix 3 is illustrated in Figure 2.

Table I

Data Sheet for UO_2 Extrusions

(Sintered at 1680°C for 17 hrs)

Mix No.	Type of UO_2 Powder	Plasticizer Added	Die Type	Average[a] Ram Pressure psi	Type of Sample	Quality of Sample	Density of Sample, Percent of Theoretical		
							Unfired	Fired	Difference
1	Fuel-element-grade MCW UO_2 ground in a rubber-lined micronizer to 0.83μ average particle size.	8.5 w/o of a 1.5 w/o methyl cellulose-H_2O solution.	50° Cone	3,500	½ inch diameter rod.	Excellent surfaces, moderate green strength	51.0	96.8	45.8
2	Fuel-element-grade MCW UO_2 ground in a rubber-lined micronizer to 0.83μ average particle size.	8.5 w/o of a 1.5 w/o methyl cellulose-H_2O solution	40° Cone	2,900	½ inch diameter rod	Excellent surfaces, moderate green strength	50.2	96.5	46.3
3	Fuel-element-grade MCW UO_2 ground in a rubber-lined micronizer to 0.83μ average particle size.	8.5 w/o of a 1.5 w/o methyl cellulose-H_2O solution	40° Cone	5,500	½ inch O.D. ¼ inch I.D. tube	Excellent surfaces, moderate green strength	47.0	96.6	49.6
4	40 w/o of fuel-element-grade MCW UO_2 and 60 w/o of fuel-element-grade UO_2 ground in a rubber-lined micronizer 0.83μ average particle size.	8.5 w/o of a 2.7 w/o methyl cellulose-H_2O solution	40° Cone	2,400	½ inch diameter rod	Fair surfaces, moderate green strength	53.5	91.5	38.0
5	Uranium dioxide produced from ammonium diuranate.	12.5 w/o of a 1.0 w/o methyl cellulose-H_2O solution	50° Cone	4,900	½ inch diameter rod	Fair surfaces, fair green strength	36.6	80.8	44.2

[a] Average Ram Pressure = Average ram thrust, lbs. divided by ram area, sq in.

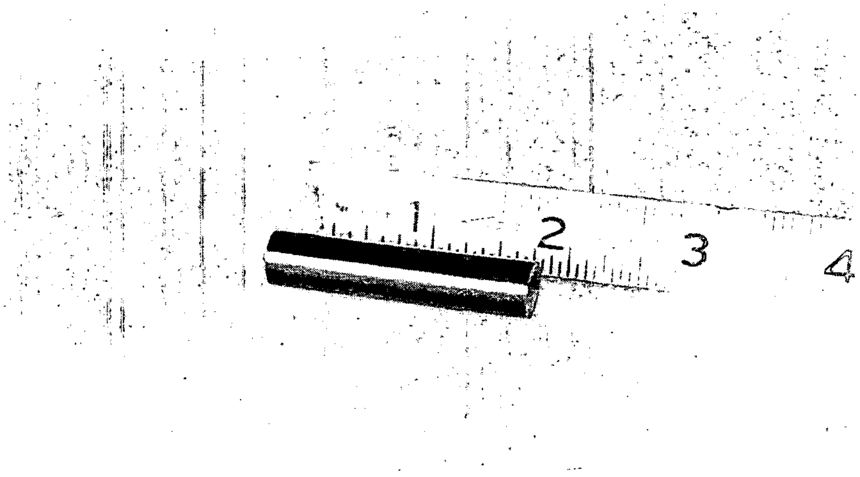

Figure 1

Extruded UO_2 Rod (Mix 1)

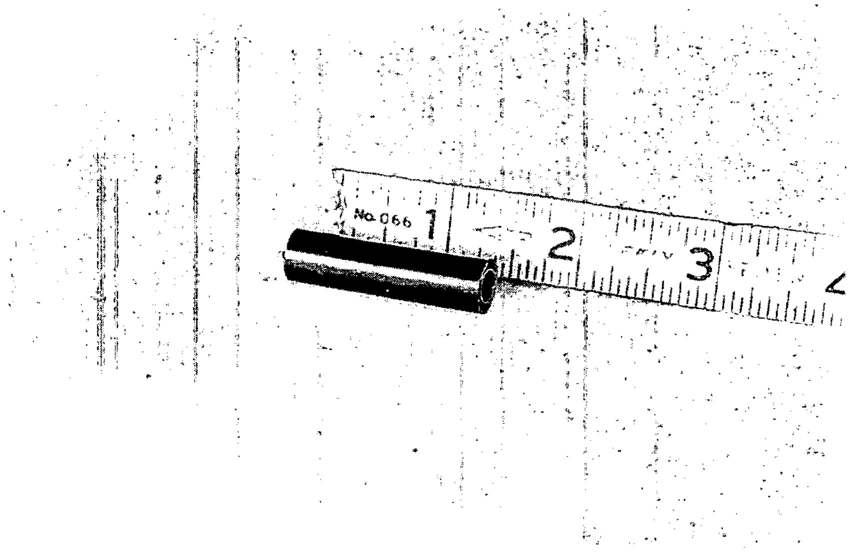

Figure 2

Extruded UO_2 Tube (Mix 3)

A mixture of 40 w/o fuel-element-grade MCW UO_2 and 60 w/o micronized UO_2 was extruded into rods to determine if higher sintered densities could be achieved. It was reasoned that better packing characteristics might result from a mixture of large (MCW UO_2) and small (micronized UO_2) particles. Finally, uranium dioxide from ammonium diuranate was extruded into rods to see if higher densities could be obtained from this highly sinterable powder. The extrusion data and densities of the latter extrusions are shown in Table 1 as Mixes 4 and 5 respectively.

It may be seen from Mix 1 in Table 1 that the rods extruded from micronized UO_2 with the 50° die had high densities and excellent surface quality while the rods from Mix 2 which were extruded from the 40° die, were just as good and extruded at a slightly lower pressure. The tubes extruded from Mix 3 also had high densities and excellent surface quality but required more ram pressure for extrusion. The tubes had slightly lower densities than the rods before sintering but gained more during the sintering operation. The rods of Mix 4 which were extruded from a blend of fuel-element-grade UO_2 and micronized UO_2 had slightly higher unfired densities than the preceding rods and tubes; however, the density increase during firing was insufficient to equal that of rods made entirely from micronized UO_2. In addition, the surface quality of these rods was considerably lower than that of the rods from micronized UO_2. The rods of Mix 5, which were extruded from UO_2 made from ammonium diuranate, had rather low densities in both the unfired and fired conditions; likewise the surface quality and green strength of these rods were not very good. However, it is possible that rods extruded from ammonium diuranate UO_2 would be of better quality if a more concentrated plasticizer solution were used.

Program

The movement of the UO_2 fuel element section to a new laboratory was started during the present quarter and is scheduled for completion in July, 1957. After the UO_2 fuel element laboratory is established in its new location, it is planned that the following projects will be pursued:

1. Further evaluation of sinterable UO_2 powders by the extrusion of prototype fuel elements.
2. Sintering studies on UO_2 and ThO_2 particles.

HYDROGEN IN DINGOT URANIUM METAL

by

L. L. Gidley
J. Howard
W. A. Taylor
W. G. Weber
H. J. Schaffer

I. Summary

1. Bomb-center temperatures of 500°F and higher prior to firing, obtained by increasing the firing time, tended to produce low hydrogen uranium. High bomb-center temperatures were effective with both dingot-MFL lined bombs and helium-purged, roasted slag-lined dingots with filter tips and vented caps.

2. A method for producing dingot uranium with an acceptable hydrogen content of 2 ppm or less has been developed. This method employs magnesium filmed with HF as the reductant.

3. A number of solid additions to the UF_4 - Mg bomb charge were investigated. Barium peroxide and uranium trioxide additions were found to be beneficial in lowering the hydrogen content of the reduced uranium.

II. Introduction

Previous dingot bomb experiments occasionally resulted in exceptionally long firing times. Evaluation of hydrogen results for a large number of dingot bomb experiments, including both short and long firing times, indicated that the latter tended to yield dingot metal with a low hydrogen content.[1] Also with small laboratory-sized bombs, an inverse relationship was found between the hydrogen content of the uranium and the bomb's central temperature, which in turn is related to the firing time (increased firing times having resulted in higher central temperatures in the bomb).[2] Finally, laboratory studies of gas evolution from UF_4 - Mg mixtures indicated that hydrogen evolution is both time and temperature dependent above 550°F.[3]

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[1] Becker, R., *et al.*, *Process Development Quarterly Report, Part II*, Mallinckrodt Chemical Works, *MCW-1402*, (May 1, 1957), p. 113-114

[2] Neumann, N. F., *et al.*, *Process Development Quarterly Report, Part I*, Mallinckrodt Chemical Works, *MCW-1401*, (April 1, 1957), p. 100-103.

[3] *Ibid*, p. 65-70.

These experimental results provided sufficient evidence to warrant concentration of the Pilot Plant efforts on increasing the firing time, and thus the charge temperature, of dingot bombs. Three suggested methods for increasing the firing time were:

1) Varying the furnace conditions to obtain longer firing times and higher charge temperatures.
2) Filming the magnesium to delay firing.
3) Additives to the charge to delay firing.

III. Experimental Procedures and Discussion

A. Variation of Dingot Furnace Conditions to Obtain Longer Firing Times

Past laboratory work has indicated that an inverse relationship exists between the hydrogen content in uranium metal and the center temperature of the bombs.[4] Above 500°F, hydrogen in uranium decreases more slowly[5] with increases in bomb-center temperature than below 500°F. The hydrogen content is above 3 ppm below 500°F.

Therefore, dingot firing schedules were altered so that a center temperature of 500°F could be reached. The dingot-center temperatures, prior to firing, were obtained for several Hevi-Duty furnace control settings. Only the bottom elements (zone-2)[6] were used in every case. The control temperatures were 800°F, 900°F, 1000°F, and 1100°F, for various groups of dingot runs.

Two types of dingots were fired with the above furnace conditions. These were: (1) dingots using dingot product slag for liners, and (2) helium bottom-purged, roasted slag-lined dingots employing slag derived from Plant 6E. In each type of dingot, thermocouples were placed at the center and at the radius of gyration of the large right cylindrical section, both being set at 20 inches below the top of the shell flange (see Figure 1). An igniter was placed in the center of the lower cavity, two inches from the bottom of the blend. These bombs were fired electrically when the center temperature reached 500°F, or in any event at the end of 20 hours so that the heating time for each dingot would be limited.

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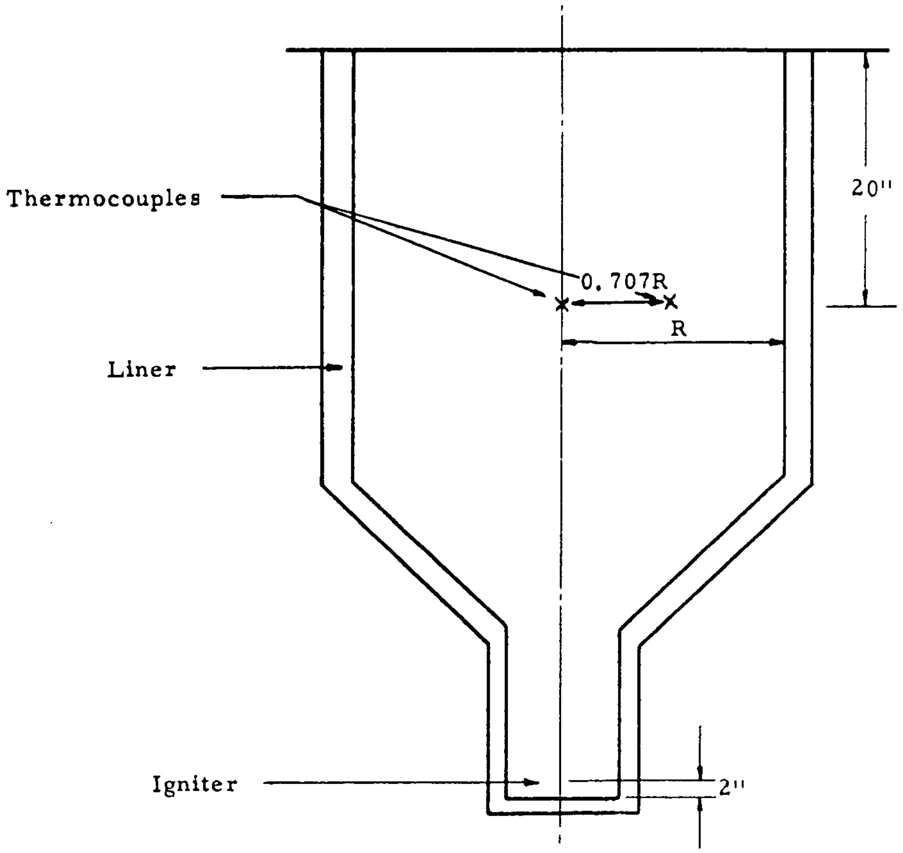
[4] *Op. cit., MCW-1401*, p 100-103

[5] *Ibid*, p 103

[6] Weber, W. G., *et al, Process Development Quarterly Report, Part II*, Mallinckrodt Chemical Works, *MCW-1381* (November 1, 1955) p 134.

FIGURE 1

POSITION OF THERMOCOUPLES IN 3300 LB. DINGOT BOMBS



The data obtained from these runs are shown in Tables I and II and Figures 2 through 5. Figure 2 is a plot of average center temperature, prior to firing, *versus*, average hydrogen content in uranium metal. It shows a trend toward lower hydrogen contents with increasing dingot-center temperature for both types of dingots. As shown in Tables I and II, several bombs fired prematurely, and these supplied low-firing time points for Figure 2.

Table I

Experimental Data for Helium Bottom–Purged, Roasted Slag-Lined Dingots

Run No	Furnace Temp °F	Firing Time mins	Elect. Firing	Total He in cu ft	Crude Yield %	Mach. Yield %	Hydrogen ppm	Center Temp °F
23142	900	980	No	291	96.07	72.96	1.7	477
23145	900	1260	No	Nil	99.55	71.24	2.9	390
23151	900	1040	No	325	97.11	76.22	2.0	382
23156	800	1540	Yes	510	96.95	scrap	0.7	502
23157	800	1540	Yes	510	93.37	67.39	0.7	503
23159	1000	1484	Yes	490	92.24	65.79	1.9	503
23160	1000	1235	Yes	390	96.10	63.33	3.8	275
23162	800	2225	Yes	740	82.30	64.82	2.8	484
23164	1100	660	No	220	91.30	scrap	2.7	233
23167	1100	1210	Yes	400	95.69	80.24	3.4	256
23168	1100	670	No	220	99.59	54.68	1.5	275
23170	1000	1205	Yes	300	90.06	55.92	2.3	267
23172	1000	675	No	225	91.73	54.8	2.2	169

Summary at Each Furnace Temperature[a]

Number of cases 3	900	1093	No	205.3	97.57	73.47	2.2	416
Number of cases 3	800	1768	Yes	586	90.87	66.10	1.4	496
Number of cases 4	1000	1149	3 of 4	351	92.53	59.96	2.5	303.5
Number of cases 3	1100	846	1 of 3	280	95.52	67.46	2.5	254

[a] Average values

Table II

Experimental Data for Dingot MFL-Lined Dingots

Run No.	Furnace Temp. °F	Firing Time mins	Elect. Firing	Crude Yield %	Mach. Yield %	Hydrogen ppm	Center Temp. °F
21830	900	720	No	95.39	79.46	4.8, 5.3	191
21833	900	630	No	95.07	79.61	6.6	373
21838	800	905	No	96.64	81.51	3.5	269
21851	1000	1085	No	98.16	80.83	1.6	500
21853	1000	1520	Tried	98.74	83.11	1.3	645
21857	1000	625	No	97.51	82.13	3.8	204
21861	1000	675	No	92.36	64.35	5.6	272
21865	1000	1215	Yes	96.93	79.85	3.3	358
21866	1000	1205	Yes	98.16	79.01	3.3	413
21867	1000	1205	Yes	97.57	76.98	2.2	440
Summary at Each Furnace Temperature[a]							
Number of cases 1	800	905	No	96.64	81.51	3.5	269
Number of cases 2	900	675	No	95.23	79.53	5.7	282
Number of cases 7	1000	1075	3 of 7	97.06	78.04	3.0	404

[a] Average values

The overall machined dingot yield data are shown in Figure 3. These yield data for helium-purged, roasted slag-lined dingots agree, in range, with previous data[7] for this type bomb. The overall machined dingot yields for dingot MFL-lined bombs are higher than for helium bottom-purged, roasted slag-lined dingots, but the hydrogen contents are also higher, as shown in Figure 4. The center temperatures appear to be about the same for both types of bombs at any given firing time, as shown in Figure 5.

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[7] Hansen, J.W., Kerr, G. E. and Schaffer, H.J., *Process Development Quarterly Report, Part II*, Mallinckrodt Chemical Works, *MCW-1398*, (November 1, 1956) p 81

FIGURE 2

AVERAGE CENTER TEMPERATURE *vs* AVERAGE HYDROGEN CONTENT

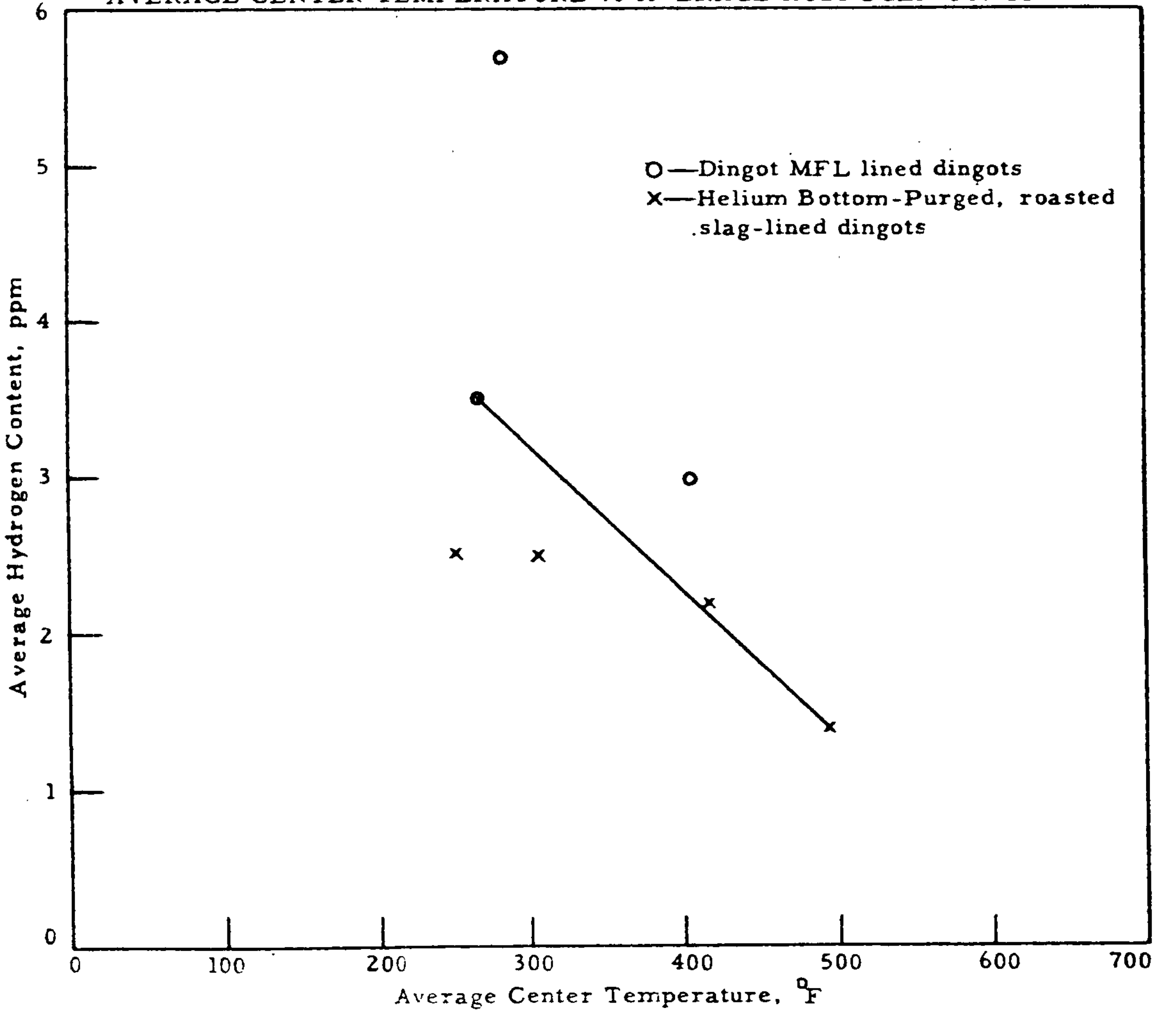


FIGURE 3

DISTRIBUTION OF PERCENT OVERALL MACHINED YIELDS

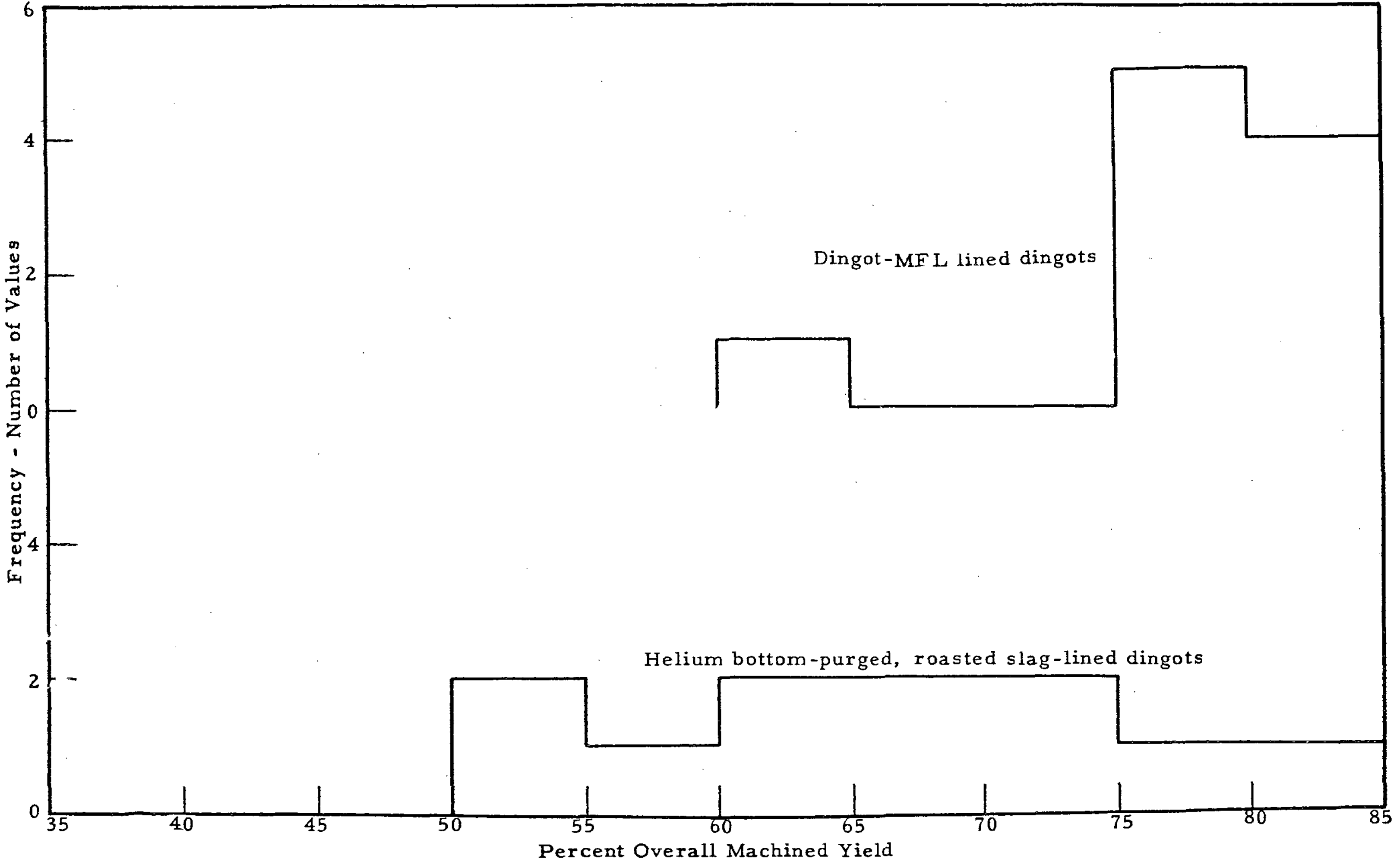


FIGURE 4

DISTRIBUTION OF DINGOT HYDROGEN CONTENTS

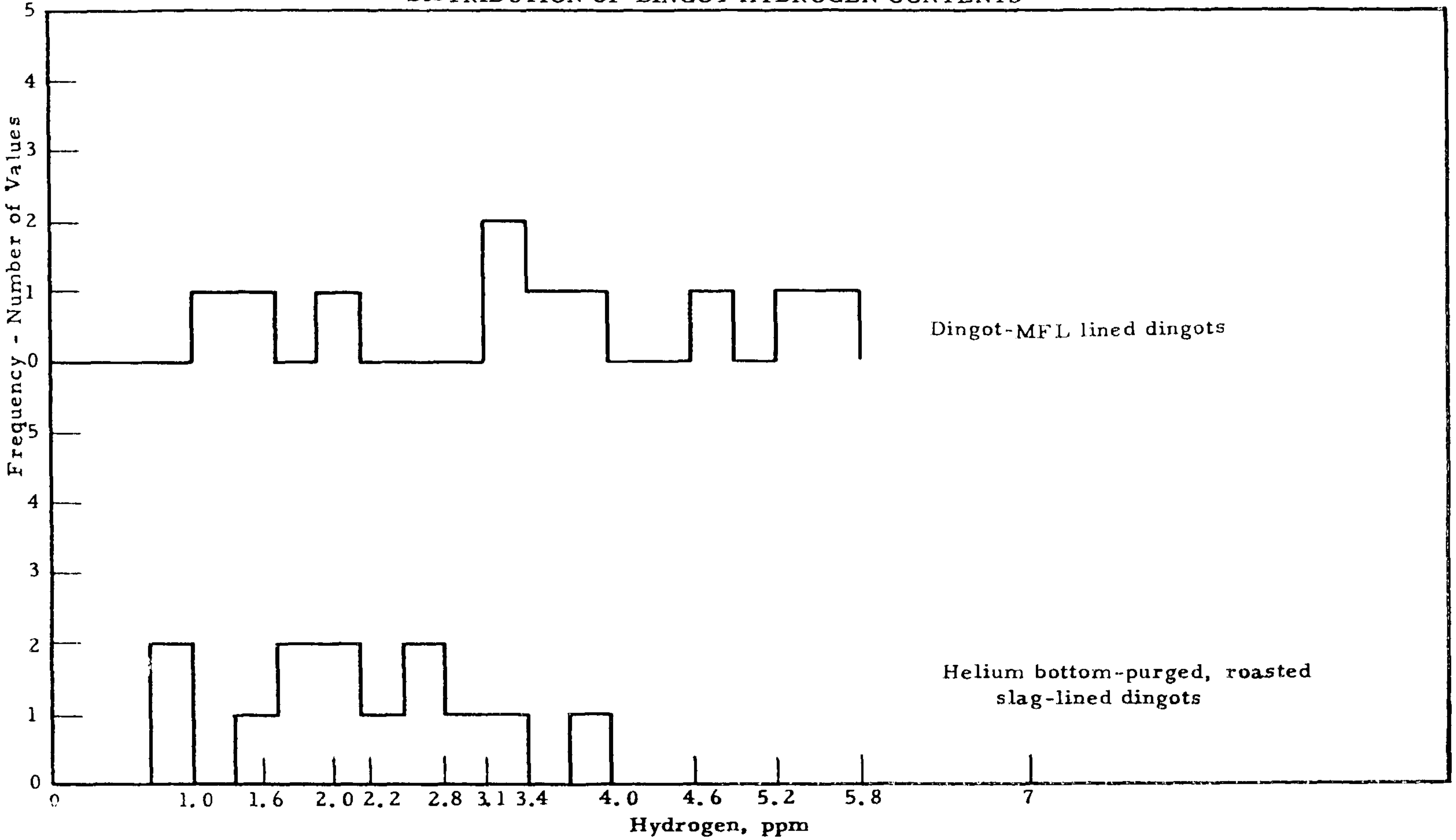
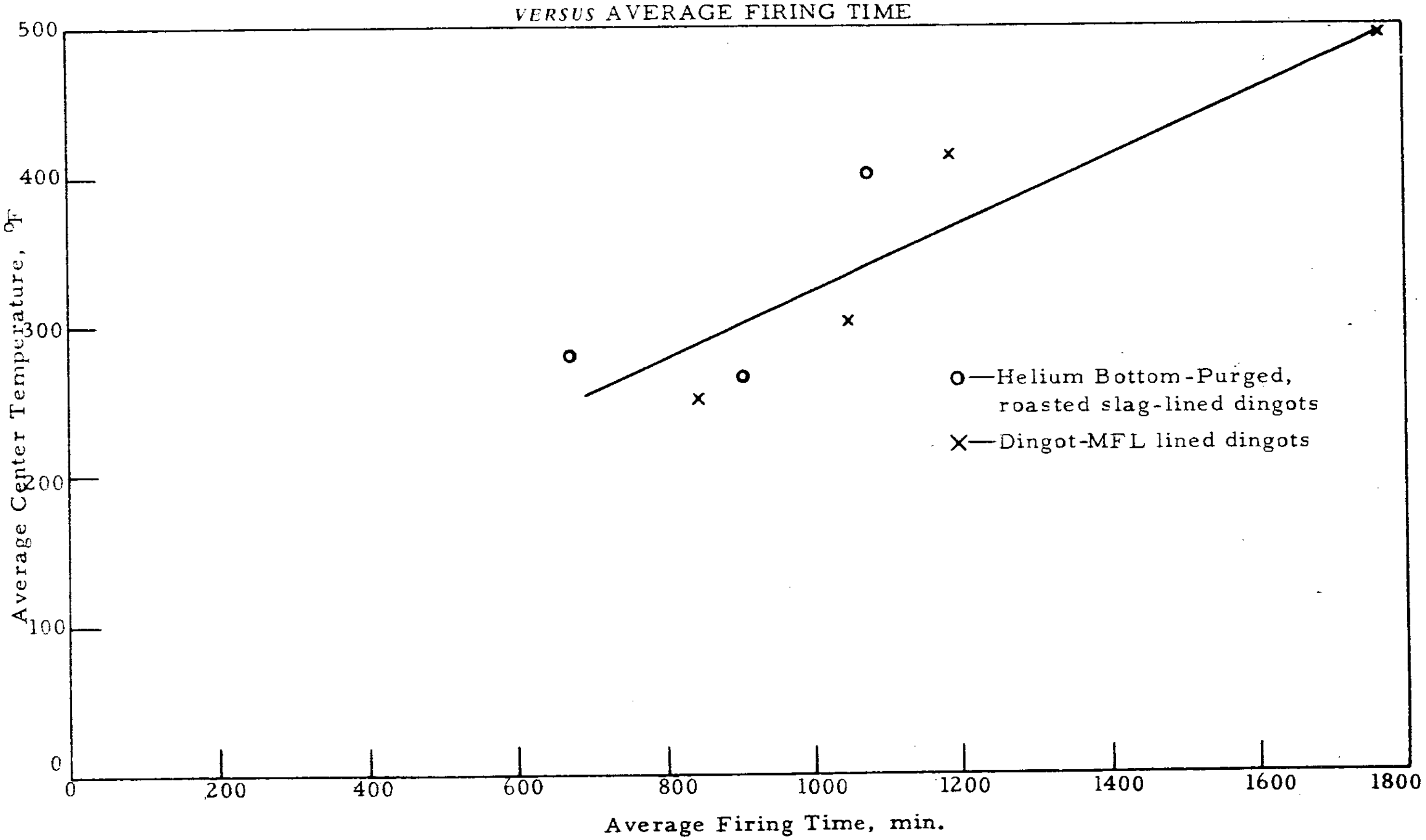


FIGURE 5

AVERAGE DINGOT CENTER TEMPERATURE AT FIRING

VERSUS AVERAGE FIRING TIME



B. HF-Filmed Magnesium

A suggested method for lengthening the firing time of bombs, which should result in a desired increase in the charge temperature at firing, was to coat the surface of the magnesium particles with a film. A tenacious film might delay magnesium volatilization (magnesium has the relatively low boiling point of 1107°C) which in turn might delay firing. The logical film for magnesium is magnesium fluoride, which is one of the two major bomb reaction products.

The Research Department performed the initial filming experiments by heating the magnesium to 400 – 630°C for 1 to 3 hours in HF or in an air and HF atmosphere. X-ray analysis of the film indicated the presence of only MgF_2.[8]

The laboratory hydrogen-gas evolution experiments, that were briefly mentioned in the introduction, were repeated with mixtures of uranium tetrafluoride and filmed magnesium. Hydrogen-gas evolution was less at all temperatures with the latter mixture.[9] Hydrogen analyses of the filmed magnesium revealed that on the average it contained $\frac{1}{6}$ as much total hydrogen as did production grade magnesium. Filming had even decreased the hydrogen content of the magnesium to a value lower than than obtained for redistilled milled magnesium, (the latter contained $\frac{1}{4}$ as much hydrogen as did production grade magnesium).[10]

Laboratory-sized bombs (500gram uranium derbies) that were fired with filmed magnesium generally yielded metal with hydrogen contents of 1.5 ppm. Helium purging with filmed magnesium yielded metal with 0.5 ppm hydrogen.[11] These small scale experiments were sufficiently promising to warrant Pilot Plant experiments.

1. Filming Procedure

The rerun reactor in Plant 7 (normally utilized for the retreatment of subgrade green salt)[12] was slightly altered to permit HF filming of magnesium on a large scale. This alteration consisted of removing the screws which were previously used for continuously moving material through the two monel tubes, and sealing off the entrance and

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[8] *Op. cit., MCW-1401,* p 105-106

[9] *Ibid,* p 65-70

[10] *Ibid,* p 83-87

[11] *Ibid,* p 105-110

[12] Gidley, L. L., *et. al., Process Development Quarterly Report, Part II,* Mallinckrodt Chemical Works, *MCW-1400,* (February 1, 1957) p 40-42

exit ports normally used in handling green salt. The resulting reactor was the equivalent to two electrically heated tube furnaces as shown in Figure 6. The cal-rod heating elements for each reactor zone were separately controlled. The purging, filming, or cooling gas entered the lower tube, passed through its entire length, exited into the second tube, passed through its entire length and then out. To implement handling of the magnesium, boats of magnesium alloy sheet were constructed so that each tube would hold four boats.

Approximately 100 pounds of magnesium were charged into each boat which was then pushed into the two reactor tubes. The control temperature for each zone was set for 850°F and a helium flow of 1.8 cfm was maintained over the magnesium particles. The helium purged air from the reactor and eliminated the danger of burning the magnesium. After 1 hour the helium flow was decreased to 0.2 cfm. This flow was maintained for approximately 6 hours which is the time required for the reactor to reach 850°F. Then the helium flow was stopped, and HF gas was introduced into the reactor for 1 hour at the rate of 40 pounds/hour. At the end of this hour, the HF gas flow was stopped, and the helium flow was resumed and maintained at 0.2 cfm until the magnesium cooled to 500°F. To increase the rate of cooling, a forced air draft was passed around the tube exterior. Cooling required about 2 hours, after which the boats were removed, and the filmed magnesium was placed in 33 pound capacity cans. Magnesium filmed in this fashion has been used for the Pilot Plant experiments which are discussed below.

2. Pilot Plant Experimental Procedure and Results

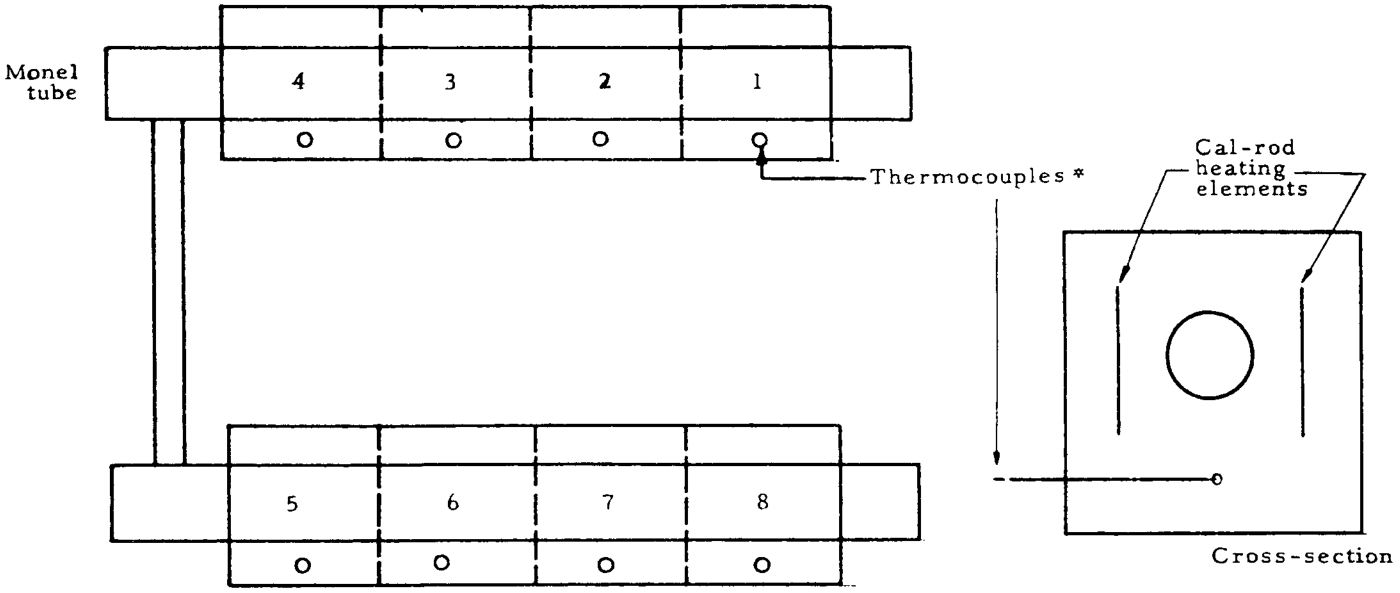
Pilot Plant experiments were initiated with 3300 pound dingot bombs. Each bomb was lined with standard magnesium fluoride liner material, and charged with a mixture of uranium tetrafluoride and filmed magnesium (1% excess). A large number of bombs have been fired.

a. Normal Firing of 3300 Pound Dingot Bombs

The first series of 3300 pound dingot bombs were fired in the Hevi Duty furnace with the following sequence of furnace conditions.

FIGURE 6

SKETCH ILLUSTRATING RERUN REACTOR USED FOR FILMING MAGNESIUM



*Thermocouples located at midpoint of zone, one inch below reactor tube

Control Temp.	Zones Activated	Heating Time
1150°F	All Furnace Elements	5 hours
1150°F	Bottom Furnace Elements	Until firing or for 9 hours (a total of 14 hours)
1250°F	Bottom Furnace Elements	Until firing or for 4 hours (a total of 18 hours)
1350°F[a]	Bottom Furnace Elements	Until firing or for 5 hours (a total of 23 hours)

[a] Used only if the bomb fails to fire in 18 hours.

Following are the experimental results:

Table III

Filmed Magnesium Experimental Results for
3300 Pound, Normal Fired Dingot Bombs

Run No.	Firing Time min	Crude Yield %	Overall[a] Machined Yield %	Dingot Hydrogen Content ppm
21806	905	96.31	----	1.6
21809	905	97.78	----	1.6
21813	730	98.09	----	1.4
21819	830	98.70	84.42	1.3
21820	735	97.02	----	1.4
21822	785	94.65	62.07	2.4
21826	840	93.89	72.85	1.5
21832	725	96.76	80.03	1.9
21834	715	96.55	69.24	1.7
21843	960	99.49	75.30	1.5
21844	975	97.56	79.30	1.7
21845	860	98.88	74.53	1.7
Averages	830	97.14	74.72	1.6

[a] Four crude dingots were either sawed for samples or machined to dimensions suitable for extrusion billets.

The desired increase in firing time was definitely obtained (an average of 830 minutes compared to a normal firing time of 600 minutes). The machined yield average of 74.72% was not satisfactory, and visual examinations of the crude dingot suggested that poor metal-slag separation caused the low yields. Dingot hydrogen contents were encouraging in that, with one exception, all were below 2.0 ppm.

b. Electrical Firing of 3300 Pound Dingot Bombs

After firing several of the above described bombs, the question arose as to whether the increase in firing time or the filming of the magnesium was actually the cause of the decrease in the hydrogen content of the dingot.

Five 3300 pound dingot bombs were therefore charged, placed in the Hevi-Duty furnace with the same furnace conditions as stated above, and fired electrically at the end of 600 minutes. The electrical firing was accomplished with the aid of a nichrome wire igniter[13] which was placed in the bottom of the dingot cavity before charging, and heated electrically at the end of approximately 600 minutes.

Following are the experimental results:

Table IV

Filmed Magnesium Experimental Results for 3300 Pound, Electrically Fired Dingot Bombs

Run No.	Firing Time min	Crude Yield %	Overall Machined Yield %	Dingot Hydrogen Content ppm
21814	628	93.62	62.52	1.4
21818	600	92.65	60.33	1.0
21821	685	97.55	78.85	1.6
21827	635	98.57	80.57	1.5
21831	610	90.21	80.03	1.6
Averages	632	94.52	72.46	1.4

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[13] *Op. cit., MCW-1402, p 115-116*

All of the bombs were successfully fired electrically. Both crude and machined yield averages were several percentage points below those values obtained with normally fired bombs. Slaggy tops (indicating incomplete metal-slag separation) were again observed on some of the crude dingots probably accounting for the 60% yields.

The dingot hydrogen contents were excellent averaging 1.4 ppm. This indicates that the filming of the magnesium, and not an increased firing time, was responsible for the low hydrogen levels. This also suggests a new hypothesis of elimination of hydrogen from the dingot metal; namely (1), that the hydrogen borne by the magnesium at the time of firing determines the contribution to the uranium; and (2), that HF filming not only reduces the initial hydrogen content of the magnesium but also protects the magnesium but also protects the magnesium particles from subsequent pick up.

c. Helium-Purged, 3300 Pound Dingot Bombs Fired Normally with Filmed Magnesium

A brief reference given above, concerning Research Department bomb experiments, stated that filmed magnesium resulted in derby hydrogen contents averaging 1.5 ppm, and that the addition of helium purging lowered the average hydrogen to 0.5 ppm. The possibility of some future need for dingot hydrogen contents of less than 1.0 ppm prompted the Pilot Plant to fire four 3300 pound dingot bombs with filmed magnesium plus helium purging from bottom to top. The helium purging practice has been discussed in detail in the past[14].

Following are the experimental results:

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[14] *Ibid*, p 89-102

Table V

Filmed Magnesium Experimental Results for
3300 Pound, Helium-Purged, Normal Fired Dingot Bombs

Run No	Total Helium[a] cu ft	Firing Time min	Crude Yield %	Overall Machined Yield %	Dingot Hydrogen Content ppm
23163	115	915	91.54	66.14	1.0
23165	40	890	96.98	71.05	0.9
23166	nil	685	89.88	65.86	1.1
23169	230	675	97.72	72.10	2.3
Averages		791	94.03	68.79	1.3

[a] An attempt was made to maintain a helium flow of 20 cfh but bomb resistance limited the flow to a varying amount in each experiment.

The small number of experimental runs limits any effective comparison of firing times and yields with previously discussed normally fired bombs. Dingot hydrogen contents were quite low in three out of four cases. If the deviation represented by the last experimental hydrogen result can be solved, the suggestion exists that filmed magnesium with helium purging might provide a consistent level of 1.0 ppm hydrogen in 3300 pound dingots.

3. Discussion

The hydrogen results presented above indicate that in magnesium filming a method has been developed to maintain the hydrogen content of dingot metal at an acceptable value of 2.0 ppm or less. Future Pilot Plant experiments will be directed toward

1. elimination of the infrequent dingot hydrogen contents above 2.0 ppm.
2. improvement of the green salt to machined dingot yield.

Successful termination of these experiments should complete the data required for using filmed magnesium in dingot production at Weldon Spring.

C. Additives

Another method considered for increasing firing times to obtain low hydrogen dingot metal was that of using solid additives in the UF_4-Mg bomb charge. The additives considered herein act in any one of several ways to aid in lowering the hydrogen level of the resulting dingot; *i.e.*, (1) filming, in which the additive causes the magnesium to become coated, thereby prolonging the firing time and allowing the bomb charge to become hotter before igniting, as in the case of sodium bifluoride; (2) direct flushing and dilution, in which the additive decomposes to liberate a gas, as with the action of basic magnesium carbonate; (3) indirect flushing and dilution, as in the action of water which first films the magnesium with a hydroxide coating, that in turn is released as water vapor again when the bomb triggers; and (4) oxidizing and flushing, as with the possible action of the oxygen of barium peroxide combining with hydrogen in the bomb to produce water vapor.

Table VI lists the additives tried in these experiments, with the decomposition product and decomposition temperatures of each and an indication of the type of action to be obtained in each case. Of course, the selection of the additives was restricted to those materials which were non-contaminating from the standpoint of forming alloys with uranium. Of necessity they had to have low neutron cross sections to maintain high reactivity in pile operations. They were selected so as to give a wide range of decomposition temperatures so that the actions would take place both during heating and also during firing of the bombs. The quantity of each used in a single bomb was limited so as to allow a normal liquation and slag-metal separation.

Table VI

Types of Additives

Additive	Decomposition Product	Decomposition Temperature °F	Filming	Direct Flushing and Dilution	Indirect Flushing and Dilution	Oxidizing and Flushing
BaO_2	O_2	1470				X
H_2O	OH^-	250	X	X	X	
UO_3	O_2	840	X			X
$NaClO_3$	O_2	900	X			X
$KClO_3$	O_2	750	X			X
$Ca(OH)_2$	H_2O	1070	X	X	X	
Limestone	CO_2	1350		X		
$NaHF_2$	HF	520	X	X		
$4MgCO_3 \cdot Mg(OH)_2 \cdot 4H_2O$	H_2O, CO_2	460-740	X	X		

Except as noted in the text, the make-up of the 300 pound derby bombs and the 3300 pound dingots was similar; (1) recycled magnesium fluoride slag liners and caps were used in shells with filter tip lids; (2) the caps were vented with a six-inch diameter cylinder of 20 mesh magnesium fluoride grog extending to the top of the charge to allow gases to escape through the filter tips; and (3) the uranium tetrafluoride, magnesium for reduction, and the additive were mixed intimately in a conical blender, a four percent excess magnesium was used in the derby bomb charges, while 0.5 percent was used for the dingots. The derby bombs were furnace-fired with a continuous control at 1250°F, while the dingots were heated for 5 hours with the controls at 1150°F and then the control setting was raised to 1250°F until firing occurred. Samples for hydrogen analyses were taken from the centers of the derbies, while those for the dingots were taken either from the top of the crude dingot after scalping, or from the center of the forged bar.

Data on the firings of both derby and dingot bombs are presented in Tables VII through XV. Run numbers which are bracketed were made with the same lot of UF_4. No improvement in hydrogen level in 300 pound derbies was obtained with additions of sodium chlorate (Table VII) or potassium chlorate (Table VIII). Increasing amounts of sodium chlorate and potassium chlorate tended to shorten firing times and produce derbies having bad tops that were heavy in slag.

Table VII

$NaClO_3$ Additions to 300 Pound Derby Bombs

Run No.	$NaClO_3$ lbs	Firing Time min	H_2 ppm	Comments
4917	0.44	220	2.7	
4916	0.88	250	5.8	Furnace power off 25 minutes
4915	1.32	215	3.0	
4914	1.76	220	2.2	
4850	2.0	--	6.7	
4851	4.0	210	2.7	Fired in Hevi-Duty furnace
4852	8.0	135	2.9	Fired in Hevi-Duty furnace
4853	12.0	145	4.1	

Table VIII

$KClO_3$ Additions to 300 Pound Derby Bombs

Run No.	$KClO_3$ lbs	Firing Time min	H_2 ppm	Comments
4913	0.44	200	4.4	
4912	0.88	195	3.5	Blowout through lid flange
4911	1.32	195	2.1	
4910	1.76	210	1.9	
4858	2.0	248	2.8	
4859	4.0	196	3.0	
4860	8.0	145	3.0	Blowout through lid flange

Table IX

H_2O Additions to 300 Pound Derby Bombs

Run No.	H_2O ml	Firing Time min	H_2 ppm	Comments
4942	none	215	5.7	Control bomb.
4943	none	247	2.5	Control bomb. Blowout through lid flange.
4944	50	220	2.3	
4945	100	298	1.9	
4946	200	270	2.3	
4947	400	285	2.2	
4948	800	300	2.4	
4949	1200	400	2.3	
4950	1600	410	2.6	
4951	2000	320	2.4	
4952	3000	305	3.7	
4953	4000	255	3.1	Blowout.

Table X

$Ca(OH)_2$ Additions to 300 Pound Derby Bombs

Run No.	$Ca(OH)_2$ lbs	Firing Time min	H_2 ppm	Comments
4854	2	370	1.8	Fired in Hevi-Duty furnace.
4855	4	248	2.6	
4856	8	285	2.8	
4857	12	265	2.4	

Table XI

Limestone Additions to 300 Pound Derby Bombs

Run No.	Limestone lbs	Firing Time min	H_2 ppm	Comments
4936	2	285	2.3	
4937	4	240	2.1	
4938	8	245	3.0	Furnace power off 50 minutes
4939	12	175	4.7	

Table XII

$4MgCO_3 \cdot Mg(OH)_2 \cdot 4H_2O$ Additions to 300 Pound Derby Bombs and 3300 Pound Dingots

Run No.	Bomb Size lbs	Additive lbs	Firing Time min	H_2 ppm	Comments
4787	300	1.6	275	0.44	
4788	300	1.6	230	3.5	
4791	300	1.6	243	1.0	
4793	300	1.6	352	1.7	1150°F furnace temp.
21780	3300	17.8	705	4.6-C[a]	
21796	3300	17.8	540	5.2-C[a]	
21797	3300	17.8	673	4.7-C[a]	

[a] C indicates sample taken from the top of the crude dingot after scalping.

Table XIII

$NaHF_2$ Additions to 3300 Pound Dingots

Run No.	$NaHF_2$ lbs	Firing Time min	H_2 ppm	Comments
21810	10	450	3.6-C[a]	1250°F furnace temp.
21811	10	490	7.4-C[a]	1250°F furnace temp.
21816	60	645	5.4-C[a]	1250°F furnace temp.

[a] C indicates sample taken from the top of the crude dingot after scalping.

Table XIV

BaO_2 Additions to 300 Pound Derby Bombs and 3300 Pound Dingots

Run No.	Bomb Size lbs	Extra Magnesium Added lbs	BaO_2 based on UF_4 %	Firing Time min	H_2 ppm	Comments
4813	300	none	0.49	205	4.9	
4814	300	none	0.99	245	2.1	
4815	300	none	1.48	290	2.0	
4816	300	none	1.98	362	1.4	
4825	300	none	1.48	290	1.9	
4826	300	none	1.98	-	1.0	
4827	300	none	2.47	355	0.9	
4828	300	none	2.96	430	-	Blowout
21824	3300	none	0.45	560	2.4-F[a]	1250°F furnace temp.
21812	3300	none	0.90	620	3.1-F[a]	1250°F furnace temp.
21815	3300	none	1.35	810	3.5-C[b]	1250°F furnace temp.
21829	3300	5.7	0.45	465	4.9-F[a]	1250°F furnace temp.
21828	3300	11.5	0.90	535	1.9-C[b]	1250°F furnace temp.
21846	3300	17.2	1.35	905	1.3-F[a]	1250°F furnace temp.
21847	3300	23.0	1.80	730	1.6-F[a]	1250°F furnace temp.

[a] F indicates sample taken from the center of the forged dingot.

[b] C indicates sample taken from the top of the crude dingot after scalping.

Table XV

UO_3 Additions to 300 Pound Derbies and 3300 Pound Dingots

Run No.	Bomb Size lbs	Extra Magnesium Added lbs	UO_3 based on UF_4 %	Firing Time min	H_2 ppm	Comments
4834	300	none	0.49	205	2.1	
4835	300	none	0.99	205	2.0	
4836	300	none	1.98	225	3.8	
4837	300	none	2.96	360	1.5	
4865	300	none	1.98	265	2.4	Fired in Hevi-Duty furnace
4866	300	none	2.96	365	1.9	Fired in Hevi-Duty furnace
4867	300	none	3.95	400	0.9	
4883	300	0.51	0.49	230	2.1	
4884	300	1.02	0.99	230	2.6	
4885	300	2.04	1.98	237	1.8	
4886	300	3.06	2.96	330	1.4	
4887	300	4.08	3.95	365	0.73	
4888	300	5.1	4.9	307	1.0	
21869	3300	10.2	0.90	700	2.0-F[a]	
21868	3300	15.3	1.35	795	1.8-F[a]	
21849	3300	20.4	1.80	935	1.4-F[a]	
21854	3300	20.4	1.80	1130	1.8-F[a]	
21850	3300	25.5	2.24	910	2.0-F[a]	
21855	3300	25.5	2.24	1225	1.8-F[a]	
21842	3300	30.6	2.69	1075	1.5-F[a]	

[a] F indicates sample taken from the center of the forged dingot.

No effective decrease in hydrogen values was obtained with additions of water, calcium hydroxide, limestone, sodium bifluoride, or basic magnesium carbonate. As was the case with all additives, the water additions (Table IX) were made directly to the UF_4-Mg mixture in the blender. Additions of greater than one liter in the 300 pound derby size bomb caused the mixture to become wetted to such a degree as to be difficult to discharge from the blender. The increased firing times encountered with increased water additions were evidently merely caused by water being evaporated from the charge before firing. Evidently these increased firing times did not cause a corresponding increase in central temperature and, thus, a decrease in hydrogen in the derbies.

Additions of calcium hydroxide (Table X) showed little effect on firing times or hydrogen in the metal. The derbies all had slaggy tops with the exception of 4854 which was fired in the Hevi-Duty furnace (in which heating is normally slower than when using the 30KW Rockwell furnace) and had a long firing time of 370 minutes.

The use of two to twelve pounds of limestone (Table XI) to provide a carbon dioxide flush caused decreased firing times as the additions were increased. Trials of basic magnesium carbonate (Table XII) in 300 pound derby bombs showed erratic results with some hydrogen values being very low. However, when scaled up to the 3300 pound dingot no lowering of the hydrogen was obtained with this addition and the resulting dingots were very slaggy and gave very poor overall machined yields.

The work with sodium bifluoride (Table XIII) was done directly on dingots. It showed little promise and was shelved in favor of barium peroxide and uranium trioxide.

A pronounced extension of firing times and effective reduction of hydrogen levels in both 300 pound derbies and 3300 pound dingots was obtained when barium peroxide (Table XIV) or uranium trioxide (Table XV) were used as additives to the bomb charge. As is shown in the tables, some of the initial experiments with both barium peroxide and uranium trioxide were done with no extra magnesium added to the charge to compensate for the oxygen liberated from the additives. The slag-metal separation of these runs was rather poor, producing slaggy tops. This condition was markedly improved by the addition of compensating magnesium in later bombs. The metal of several derbies and dingots was checked for barium content, which was found to be 5 ppm in all cases. Barium is not routinely checked in metal to be shipped, but the thermal neutron cross section of barium is low at 1.17 barns per atom. It appears therefore that the barium pick-up is sufficiently low to avoid any ill effects on pile operation.

It seems evident that a minimum addition of eight to twelve pounds in a derby size bomb and sixty to eighty pounds in a dingot is required of either barium peroxide or uranium trioxide to achieve an acceptable hydrogen level in the metal. These values are illustrated more clearly in the graphs derived from Tables XIV and XV which appear in Figures 7 through 10. These figures show the definite trend of increased firing times with increased additions and the corresponding decrease in hydrogen in the metal.

As would logically be expected, the prolonged firing times obtained with barium peroxide and with uranium trioxide allowed the internal temperature of the bombs to climb much higher than in normal bombs. In several of the bombs, thermocouples were installed, - - one in the center of the charge, and another at the radius of gyration, a position which would give approximately the average temperature of the charge. A typical plot of these data is presented in Figure 11 for 3300 pound dingot No. 21842 which was made with 120 pounds of uranium trioxide in the charge. Bombs with no additives normally fire in 8 to 10 hours, while this bomb fired in almost 18 hours. The increased heat content is apparent from the fact that the center temperature is approximately 360°F higher than in a normal bomb, and the radius of gyration temperature is about 270°F higher than a normal bomb.

The data of Tables XIV and XV have been combined and plotted in Figures 12 and 13 to show the effect of firing time on 300 pound derby hydrogen content and also on the hydrogen content of 3300 pound dingots. These data agree with previous results obtained in the laboratory investigation of the effect of firing on hydrogen content.

It is not known whether the reduction in hydrogen experienced with the barium peroxide and uranium trioxide additions is achieved solely because of the extension in firing time. This point will be clarified by firing some of the bombs with an internal hot wire igniter at predetermined firing times. Additional charge temperature data are also desired, and future work will include a study of various methods to improve yields.

FIGURE 7

EFFECT OF BARIUM PEROXIDE ADDITIONS ON 300 POUND DERBY

HYDROGEN CONTENT AND FIRING TIME

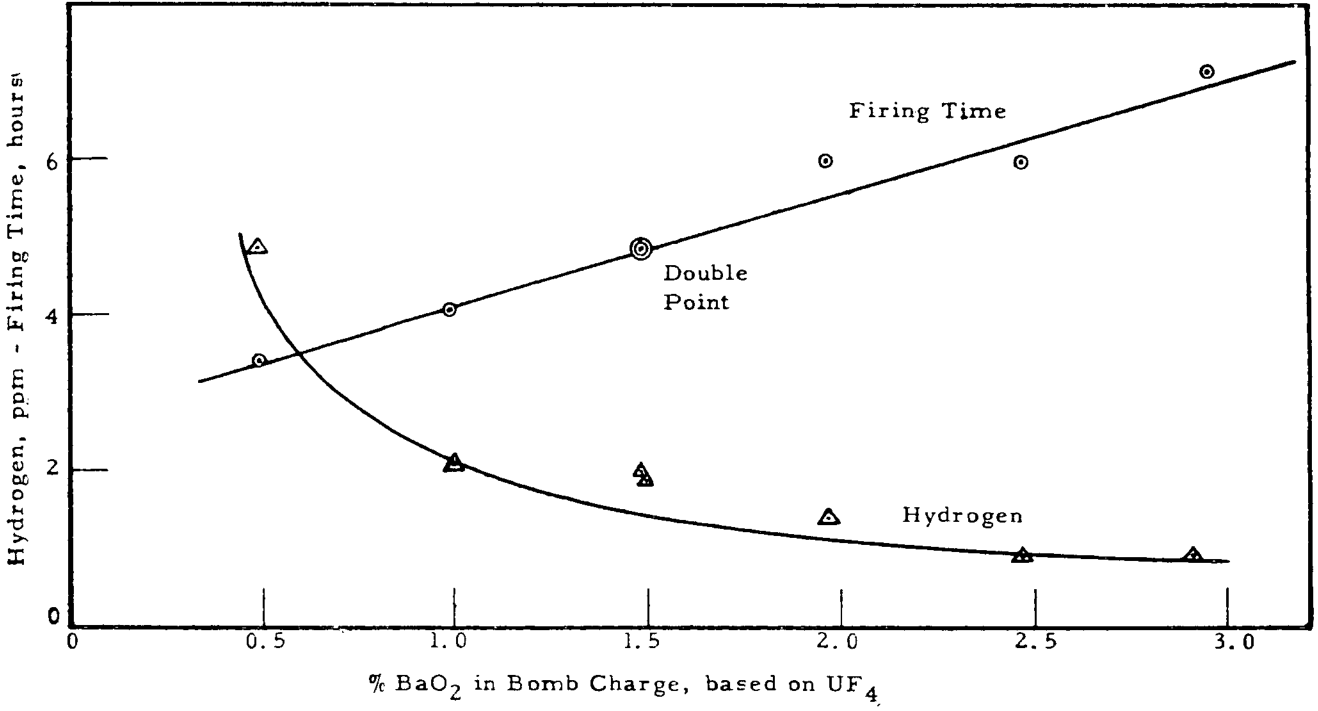


FIGURE 8

EFFECT OF BARIUM PEROXIDE ADDITIONS ON 3300 POUND DINGOT HYDROGEN CONTENT AND FIRING TIME

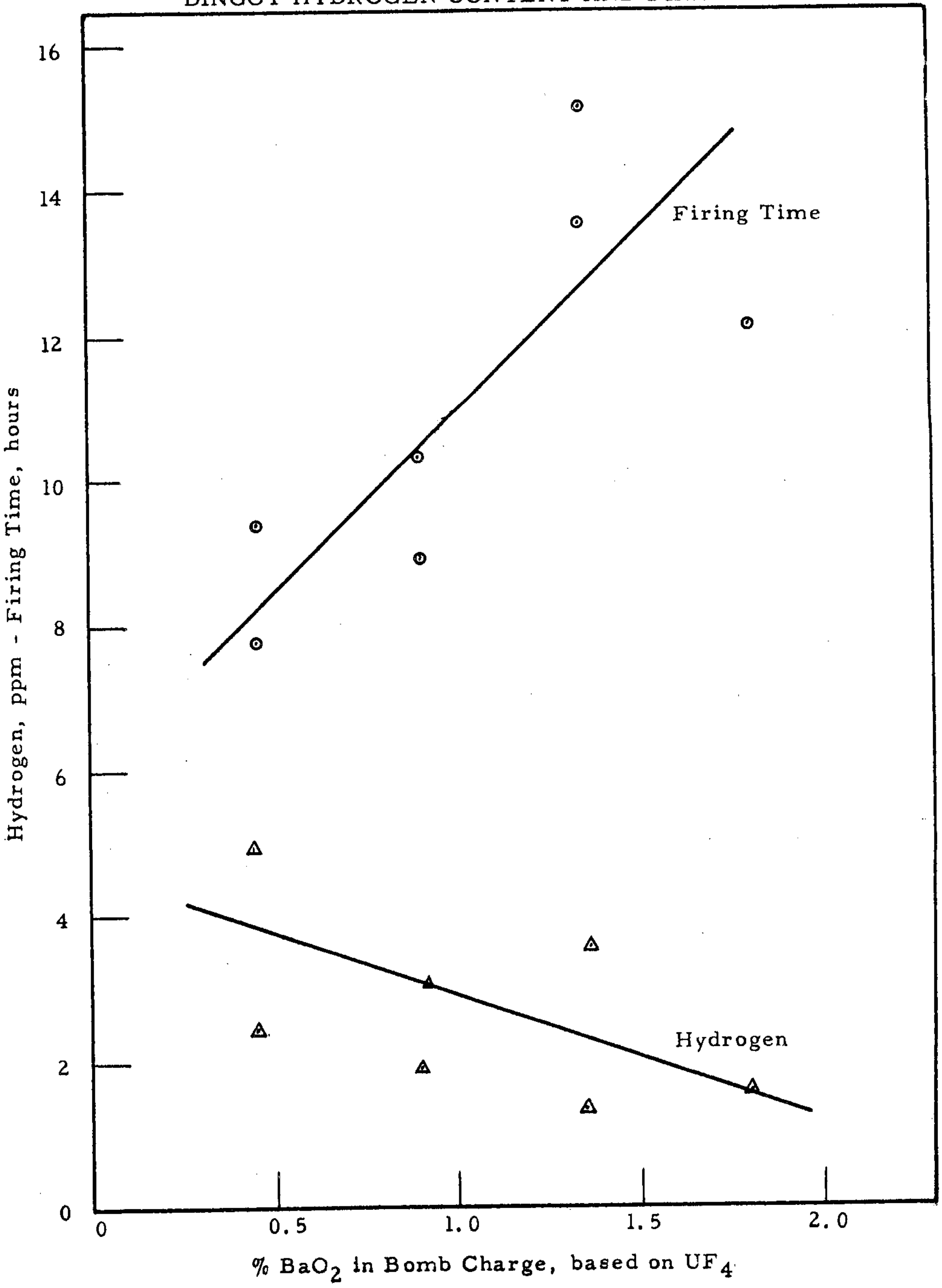
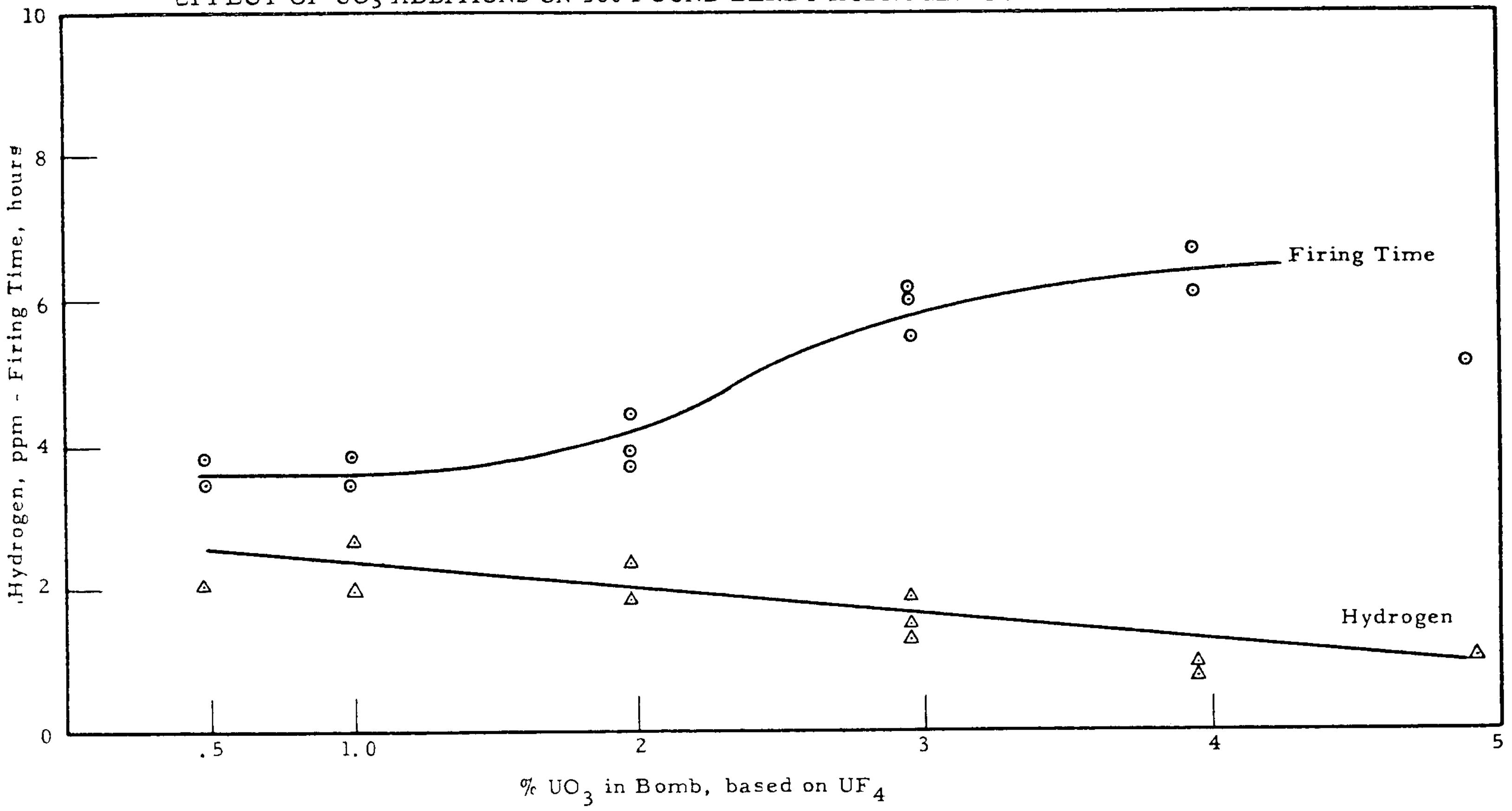


FIGURE 9

EFFECT OF UO_3 ADDITIONS ON 300 POUND DERBY HYDROGEN CONTENT AND FIRING TIME



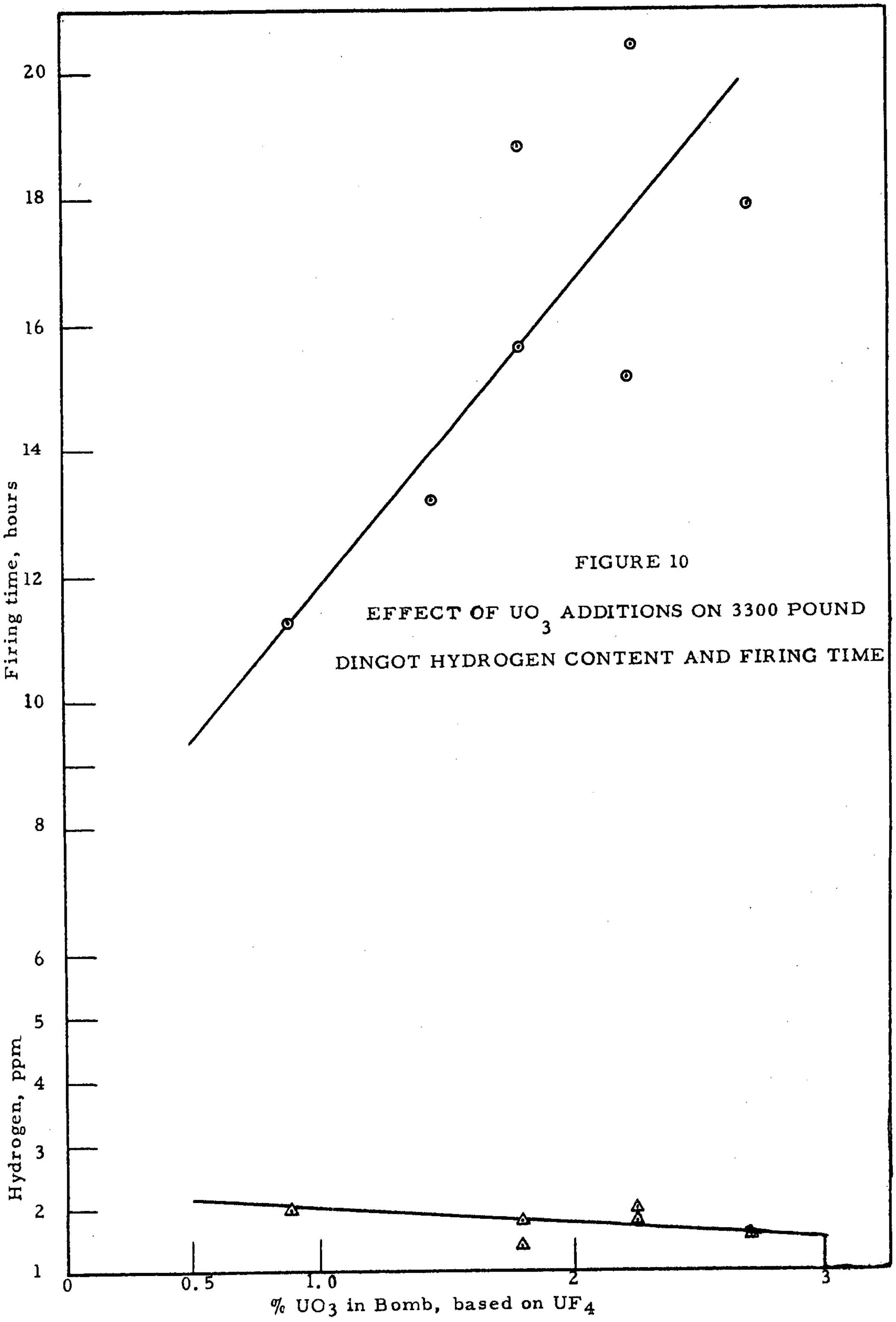


FIGURE 10

EFFECT OF UO_3 ADDITIONS ON 3300 POUND DINGOT HYDROGEN CONTENT AND FIRING TIME

FIGURE 11

PLOT SHOWING INCREASE IN INTERNAL BOMB TEMPERATURES WITH FURNACE HEATING TIME

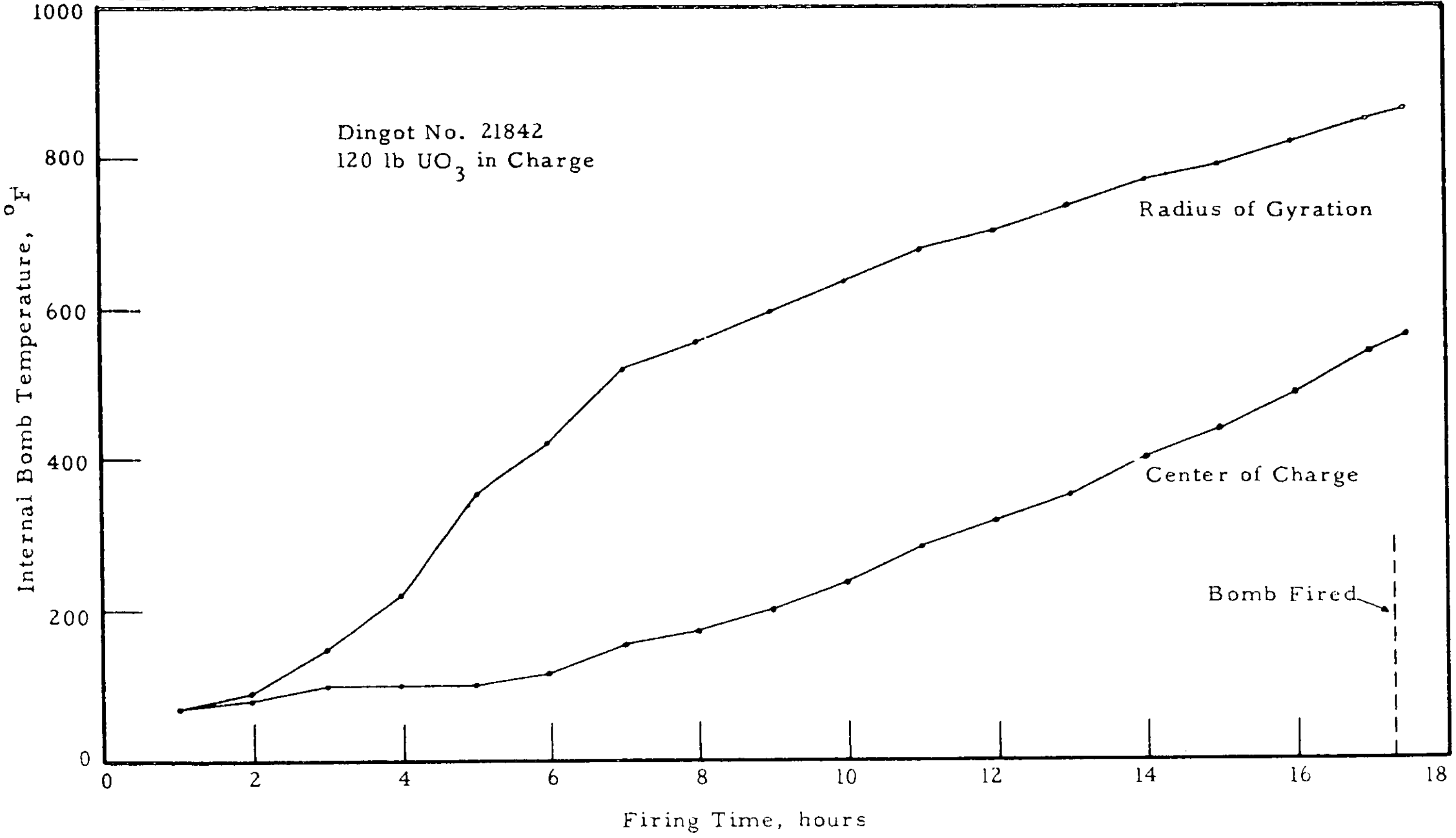


FIGURE 12

EFFECT OF FIRING TIME ON 300 POUND DERBY BOMB HYDROGEN CONTENT

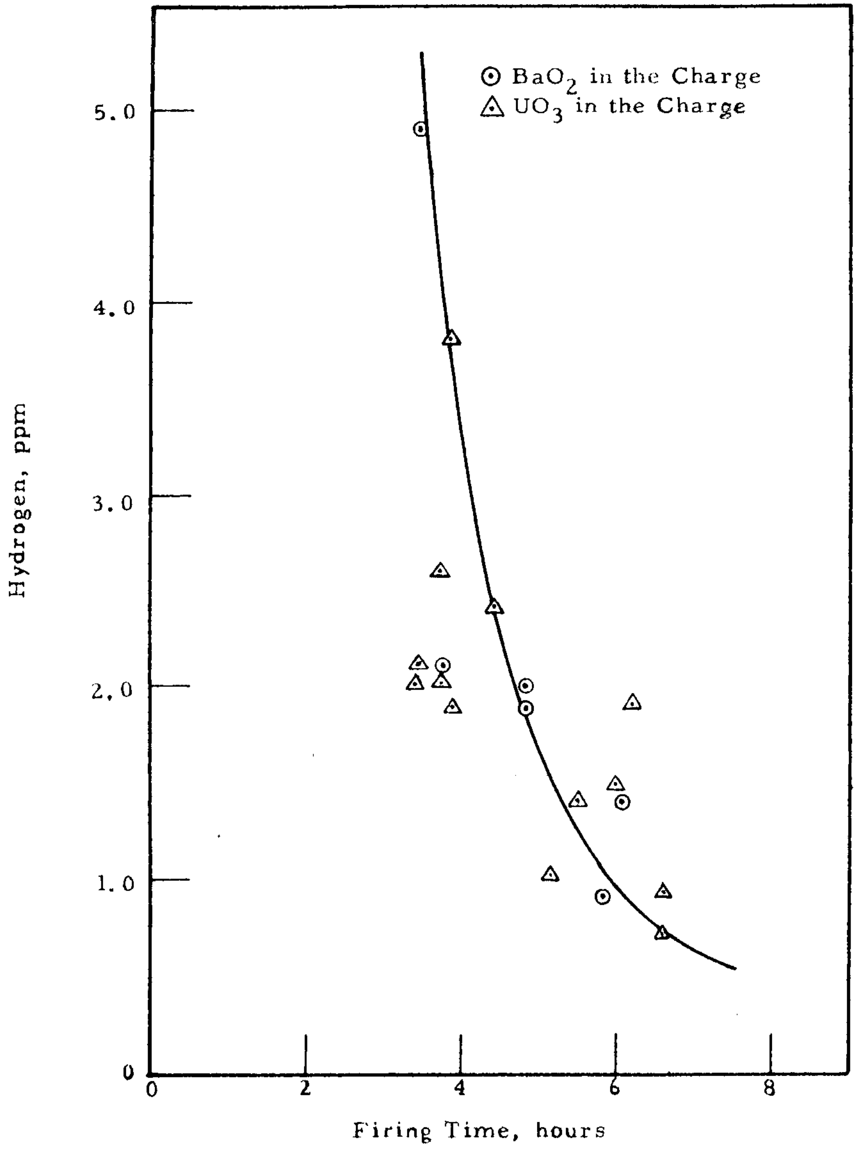
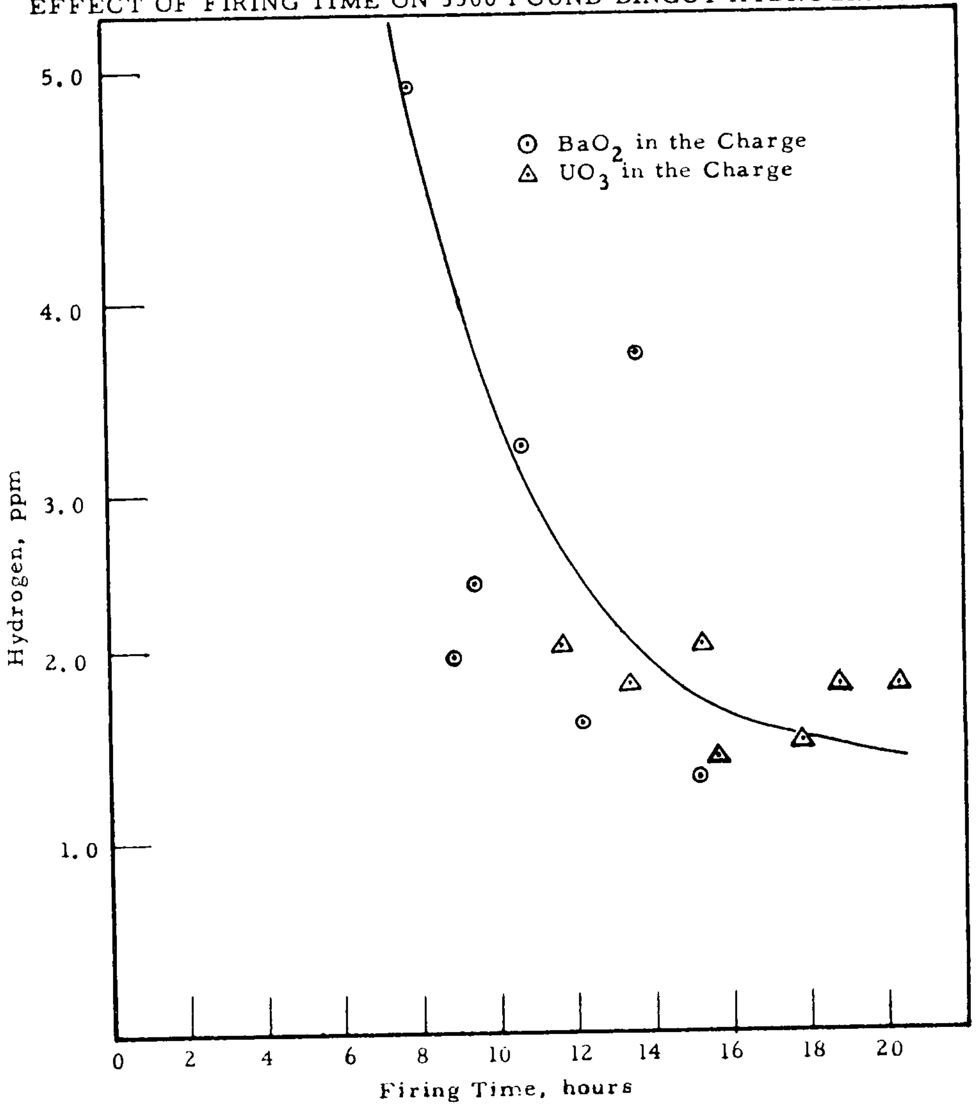


FIGURE 13

EFFECT OF FIRING TIME ON 3300 POUND DINGOT HYDROGEN CONTENT



DINGOT FORGING

by

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R. Becker
H.J. Schaffer

I. Summary

1. Production

During this quarter, the following quantities of forged bar stock were produced:

Routine dingot metal for SRO slugs	57 tons
Low hydrogen dingot metal for HAPO slugs	71 tons
Bars from NLO ingots	3 tons
Total	131 tons

2. NLO Ingots

Three tons of ingots from NLO, 10 inches in diameter and 17 to 20 inches long, were sucessfully swage-forged to 7½ inch diameter round bars with convex ends.

3. Yields

Overall yields for the dingot process have remained at the same level. The yields for the first six low H_2 shipments as well as normal dingot metal shipments are reported in Table I.

II. Introduction

Acceptance of dingot metal for pile usage has required the piloting of fabrication of fuel elements for irradiation tests. Early in the investigation of the dingot process it became apparent that the most economical shape of a dingot required preliminary forming to prepare it for successful rolling at NLO. Initial experiments indicated that press forging a dingot that had been heated in a salt bath would satisfactorily reduce the cylindrical dingot to a forged bar, which could easily be rolled. Work done in perfecting this forming technique has been reported in past quarterly reports.[1], and recent work is described below.

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[1] Williams, F.H., Schaffer, H.J., *Process Development Quarterly Report, Part II,* Mallinckrodt Chemical Works, *MCW-1398* (November 1, 1956) p. 83-88.

Table I

Individual and Overall Yields for the Dingot Process

Shipment Number	Date of Shipment	Type of Slug	Green Salt to Crude Dingot %	Green Salt to Scalped Dingot %	Crude Dingot to Scalped Dingot %	Green Salt to Forged Bar %	Scalped Dingot to Forged Bar %	Forged Bar to Good Rod %	Good Rod to Total Slug %	Total Slug to Good Slug %	Forged Bar to Good Slug %	Green Salt to Good Slug %
62	11-24-56	SRO	96.97	80.01	82.51	79.00	98.75	[a]	[a]	[a]	[a]	[a]
63	11-24-56	SRO	96.73	79.26	81.94	74.99	94.61	94.82	84.04	96.25	56.05	42.02
64	11-30-56	SRO	96.47	81.99	84.99	81.22	99.07	95.22	71.90	94.69	64.83	53.83
65	11-30-56	SRO	97.20	82.54	84.92	79.67	96.52	88.62[b]	70.77[b]	98.72[b]	61.91[b]	49.06[b]
66	12-14-56	SRO	97.18[c]	81.46	83.83[c]	79.91	98.11	92.83	75.11	96.54	67.32	53.79
67	12-26-56	SRO	97.50	84.65	86.81[c]	82.21	97.13	90.61	70.86	96.10	61.70	50.72
68	12-26-56	SRO	97.24	80.56	82.84[c]	79.47	98.68	81.57	57.84	93.30	44.10	34.99
69	1-10-57	SRO	97.18[c]	80.31	82.64	78.34	97.53	94.95[b]	69.81[b]	95.05[b]	63.00[b]	48.58[b]
70	1-16-57	SRO	96.68[c]	73.25	75.77	71.65[c]	97.80	90.08	67.64	97.49	59.40	42.47
71	1-23-57	HAPO	97.19	74.17	76.31	72.71	98.06	90.86	74.95	95.51	65.05	47.29
72	2-11-57	SRO	95.21	69.47	72.97[c]	68.49	98.60	93.42[b]	66.32[b]	70.67[b]	43.79[b]	34.88[b]
73	2-20-57	SRO	94.33[c]	67.50	71.56	63.93	94.79	94.41	66.62	84.95	53.43	34.16
74	2-19-57	HAPO	94.03	69.47	73.88	67.22	96.77	90.16	77.92	95.64	67.19	45.16
75	2-28-57	HAPO	92.28	68.20	73.91	64.03	93.91	92.14	73.96	96.56	65.81	42.13
[d] 76	2-28-57	SRO	93.49[c]	67.68	72.40	60.87	89.96	89.93	69.67	98.12	61.47	37.42
77	3-5-57	SRO	96.15	71.64	74.50	67.93	94.83	87.78	70.71	98.52	61.15	41.53
78	3-12-57	HAPO	96.34	72.81	75.58	71.34	97.97	90.00	71.27	97.84	62.75	44.76
79	3-18-57	SRO	93.25	68.25	73.19	61.40	89.96	90.55[b]	62.84[b]	89.70[b]	51.04[b]	27.80[b]
80	3-20-57	SRO	93.61	62.60	66.88	60.01	95.86	[a]	[a]	[a]	[a]	[a]
81	3-28-57	HAPO	95.93	79.33	82.70	77.15	97.25	93.11	74.77	96.60	67.24	51.79
82	4-4-57	SRO	93.65	71.07	75.89	70.06	98.59	[a]	[a]	[a]	[a]	[a]
83	4-18-57	SRO	94.36	70.03	74.22	68.55	97.89	94.45	71.24	97.29	65.46	44.86

Table I (continued)

Shipment Number	Date of Shipment	Type of Slug	Green Salt to Crude Dingot %	Green Salt to Scalped Dingot %	Crude Dingot to Scalped Dingot %	Green Salt to Forged Bar %	Scalped Dingot to Forged Bar %	Forged Bar to Good Rod %	Good Rod to Total Slug %	Total Slug to Good Slug %	Forged Bar to Good Slug %	Green Salt to Good Slug %
84	4-18-57	HAPO	95.61	73.39	76.76	71.49	97.40	89.38	75.66	97.20	65.73	47.00
85	5-7-57	HAPO	94.73	68.89	72.72	67.21	97.57	*e*	*e*	*e*	*e*	*e*
86	5-7-57	SRO	93.77	67.50	71.98	64.90	96.15	*e*	*e*	*e*	*e*	*e*
87	5-21-57	SRO	92.55	68.58	74.11	67.43	98.32	*e*	*e*	*e*	*e*	*e*
88	5-21-57	HAPO	94.71	69.98	73.90	69.12	98.77	*e*	*e*	*e*	*e*	*e*
89	5-24-57	HAPO	94.83	72.22	76.16	70.71	97.91	*e*	*e*	*e*	*e*	*e*
90	5-31-57	SRO	91.19	69.07	75.74	68.20	98.74	*f*	*f*	*f*	*f*	*f*
Average		HAPO	95.23	72.90	76.52	70.66	96.89	90.94	74.76	96.56	65.63	46.35
Average		SRO	95.22	73.87	77.48	71.41	96.59	91.37	69.67	93.39	59.07	42.58

[a] Rolled to WAPD ovals.

[b] Part of these shipments rolled to WAPD ovals; the yields for these shipments represent only those forged bars that were made into SRO slugs.

[c] Results corrected from those previously reported.

[d] Data above line reported previously.

[e] No rolling or slug data available.

[f] Rolled to ovals for the A.E.C. of Canada.

III. Discussion

A. Production

Press forging of machined dingots has continued as the primary fabrication process for the semi-works production of dingot metal.

During this quarter 57 tons of forged dingot bars containing more than 2 ppm hydrogen were produced for rolling to SRO fuel elements, and 71 tons of forged dingot bars with hydrogen below 2 ppm were produced for rolling to HAPO type fuel elements to be evaluated at that site. Three tons of forged bars were produced, by cross-forging or swage-forging, from 10 inch diameter NLO ingots, 17 to 20 inches long.

B. NLO Ingots

Six ingots, 10 inches in diameter and 17 to 20 inches long, were received from NLO, forged to flat-sided ovals, 5½ inches by 7 inches, and returned to NLO for rolling. These ingots were heated for forging in Houghton Liquid Heat No. 980 salt at 1200°F for a minimum of 45 minutes each. Each forging cycle continued for a maximum of 8 minutes before reheating.

Three ingots were upset until their heights approximated their diameters and then cross-forged in the conventional manner to produce bars for rolling at NLO. Eight reheatings, including the one for final contour forging, were necessary for production of these bars. Figure 1 shows the contour of these bars as they were shipped to NLO. Figure 2 is a closeup showing the surface obtained on these bars. Three ingots were swage-forged in "vee" dies to 7½ inch diameter rounds, then flattened, and finally contour forged to the proper contour for rolling. Only three reheatings, including those for flattening and contour forging, were necessary on these bars. Care was taken during swage-forging to align one end of the ingot with the working edges of the dies when forging at the ends of the ingots. This practice prevented dishing of the ends of the bars, which would result in excessive fishtailing of the rods in the NLO blooming mill. Figure 3 shows the convex ends of these bars after contour forging. Figure 4 is a closeup showing the surface obtained on these bars, which compares favorably with that obtained on the cross forged bars.

Rolling yields for these bars have not as yet been received from NLO.

Work on swage-forging of dingots is planned in the future to further investigate the possible economies which would result from fewer reheating cycles.

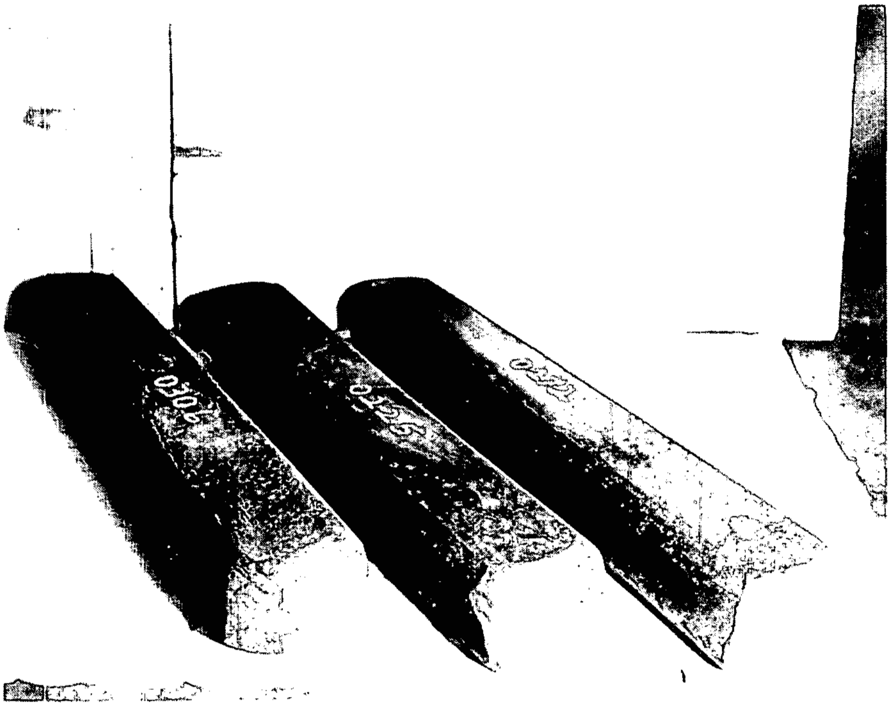

Figure 1

Bars Cross-Forged from NLO Ingots

Figure 2

Closeup Showing Surface of Cross-Forged Bar

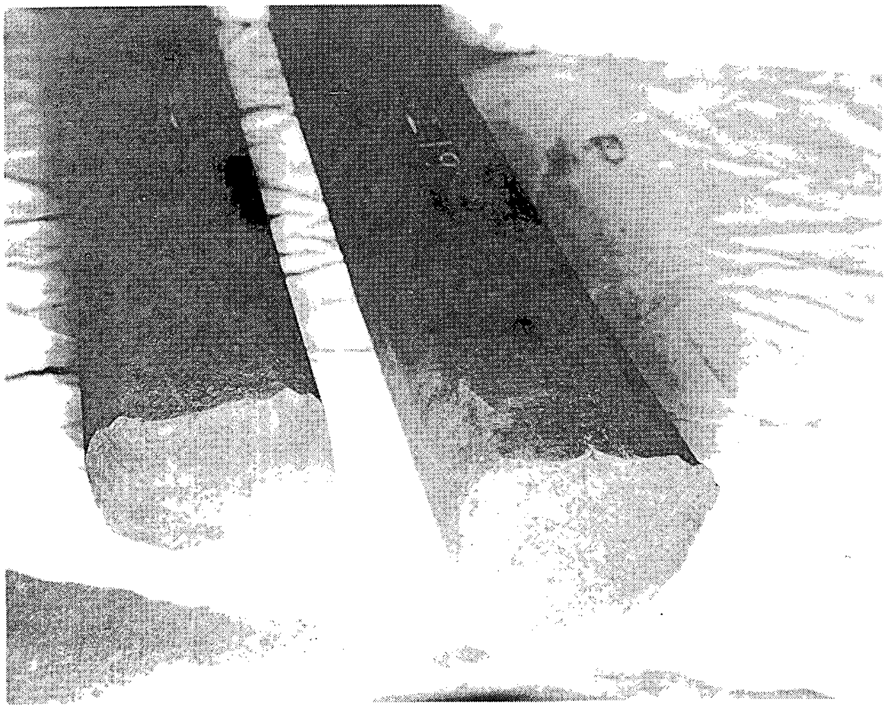

Figure 3

Bars Swage-Forged from NLO Ingots

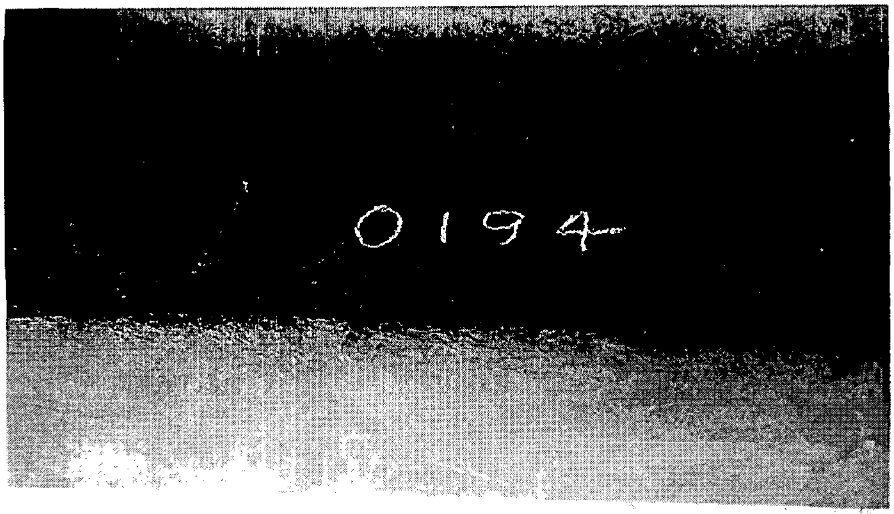

Figure 4

Closeup Showing Surface of Swage-Forged Bar

C. Yields

During the past quarter overall yields from the dingot process have remained at about the same level as those reported in the previous quarterly report.[2] The yields for the first six shipments of low hydrogen uranium metal which were rolled to HAPO slugs are reported in Table I and had an overall green salt to acceptable slug yield of 46.35%. The total slug to acceptable slug yield for this material is 96.56%.

Shipments 72, 73, and 79 had somewhat higher slug reject rates due to machining losses which lowered the overall yields for these shipments. The 68th shipment has a lower overall yield due to higher losses in the bar rolling and rod machining operations at NLO.

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[2] Becker, R.W., Schaffer, H.J., Hartmann, R.F., *Process Development Quarterly Report, Part II,* Mallinckrodt Chemical Works, *MCW-1402* (May 1, 1957) p. 127.

DINGOT EXTRUSION

by

W. E. Ellermann
R. W. Becker
H. J. Schaffer

I. Summary

Rods extruded in the third and fourth campaigns of the MCW- Bridgeport Brass Co. gamma extrusion development program at Adrian, Michigan, were evaluated. Findings of this evaluation were:

1. Glass lubrication resulted in minor gouging, or grooving, of the rods but only a few scabs and blisters were observed.
2. Oil dag lubrication resulted in numerous scabs and blisters, but otherwise the rods were smoother than those extruded with a glass lubricant.
3. Conditions which, individually, resulted in higher yields were glass lubrication, graphite follower blocks, and 1900°F billet temperatures.
4. An average linear shrinkage of 2.68%, due to oxidation and thermal contractions, was observed for rods extruded at 1800° and 1900°F.
5. Hydrogen pickup from heating billets to 1800° or 1900°F in a salt bath was negligible.
6. Metallographic examination of the extruded rods revealed only minor variations in structure. The macrostructure was found to be radially orientated consistent with the direction of heat flow while the microstructure was typical of metal quenched from the beta phase.

The fifth campaign of the above project was performed, and the following observations were made on completion of the campaign.

1. No noticeable die wear resulted from the extrusion of thirty rods through a die made of Rex AA high speed steel when glass was used as a lubricant on both the front end and periphery of the billet.

2. Grooving was noticeable on the inlet radius and land of a Rex AA die when "oil dag" was used as a lubricant.

3. No noticeable die wear resulted from the extrusion of three rods through a Rex AA die when only a glass disc in front of the billet was used for lubrication.

4. Grooving was noticeable on part of the inlet radius and land of a Rex AA die after extrusion of two billets having glass applied effectively only to their peripheries.

5. Extrusion constants, based on strain gauge measurements, for billets extruded with glass as a lubricant averaged 1805 psi, compared to an average of 2121 psi, for billets extruded with oil dag as a lubricant. This indicated that glass was superior to "oil dag" as a lubricant for the gamma extrusion of uranium.

II. Introduction

The geometry of dingots necessitates a forming operation prior to rolling at NLO. This primary forming will be accomplished by gamma phase extrusion at the new Weldon Spring Plant. The bases for this conclusion were discussed in previous quarterlies.[1,2]

A program for the development of gamma phase extrusion techniques has been conducted by the Bridgeport Brass Co., with the cooperation and assistance of MCW. The work has been performed at the Adrian, Mich., BBC plant and has been on a relatively small scale, due to the size limitations of the press being used. Preparations are nearing completion for the beginning of a program to be performed by Dow Chemical Co., as a subcontractor to MCW, at their Madison, Ill., plant. The press to be used at Dow will accommodate billets approximating the size of those to be produced from dingots at Weldon Spring. Progress on the BBC program is reported below.

Billets $6\frac{13}{16}$ inches in diameter and from 12 to 18 inches long have been extruded at Adrian. After some work with smaller orifices, a die orifice of 2.910 inches was chosen to give a reduction ratio of 6.6 to 1 with a $7\frac{1}{2}$" diameter container. This reduction ratio is equivalent to that proposed for Weldon Spring.

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[1] Williams, F, H., Schaffer, H. J., Hartmann, R. F., *Process Development Quarterly Report, Part II,* Mallinckrodt Chemical Works, *MCW-1400* (February 1, 1957) p 105-119

[2] Becker, R. W., Hansen, J. W., Schaffer, H. J., Hartmann, R. F., *Process Development Quarterly Report, Part II,* Mallinckrodt Chemical Works, *MCW-1402* (May 1, 1957) p 133-150

Previous work has indicated that:

1. Billet temperatures of 1800° and 1900°F permit extrusion without any particular billet handling or runout problems.
2. Relatively complete gamma phase extrusion is possible at billet temperatures of 1800° and 1900°F.
3. Glass lubricants seem to decrease die wear.
4. Partially extruding a graphite follower block results in practically no butt material, obviating the need for sawing or shearing for butt removal.
5. Rolling the extruded rods in Houghton Liquid Heat No. 980 salt immediately after extrusion lowers the effect of surface oxidation.

Performance of the third and fourth extrusions at Adrian was reported in the previous quarterly.[3] Evaluation of the rods from these third and fourth extrusions and the performance of the fifth extrusion are reported below.

III. Evaluation of Rods from Third and Fourth Campaigns

A. Experimental Work

Salt, glass, and oxides were removed from the rods by washing in water. After visual examination for surface defects, three micrometer measurements were made, 120° apart, at each of four locations on each rod: (1) 2 inches from the lead end, (2) the center of the rod, (3) 2 feet from the back end, and (4) 2 inches from the back end. The back ends of the rods were then cropped until free of "pipe", or extrusion defect. The good portion of each rod was weighed for yield determinations.

B. Experimental Results

1. Diameter of Rods

Averages of the three rod diameter measurements taken at various locations on each rod ranged from 2.713 in. to 2.941 in. About 80 percent of these measurements were were between 2.820 in. and 2.840 in. The frequency of these average measurements is indicated in Table I.

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[3] *Ibid*

Table I

Frequency of Average Rod Diameter Measurements

Location	Average Rod Diameter Measurements Less than 2.820 inches	 2.820 through 2.840 inches	 Greater than 2.840 inches
Back end	11	19	3
2 feet from back end	1	26	6
Middle of rod	2	30	1
2 inches from lead end	2	29	2

Surface oxidation was responsible, in part, for the large variations in diameter. Beta checking was responsible for the particularly large variations on the back ends.

Figure 1 shows graphically the frequency of individual rod diameter measurements taken 2 inches from the front ends of the rods. These measurements were chosen because they were generally more reliable than those taken at other locations. The peak of this curve at 2.832 inches indicates a mean linear shrinkage of 2.68 percent, due to oxidation and thermal contraction, from a 2.910 inches die diameter.

Only a very slight and inconsistent increase was observed in the average diameters of progressive rods extruded through each die. In order to determine wear on a Rex AA die in terms of rod diameter, it apparently will be necessary to extrude a considerably greater number of rods through one die than was done in these campaigns. Despite the obvious striations on the inlet radii and lands of the dies used with oil dag, rod diameters did not indicate any greater wear on these dies than on the one used with glass lubricant.

2. Rod Yields

Table II lists the conditions of extrusion for each rod, together with rod yields based on butt, pipe, and oxidation losses or on rod surface quality. Table III lists, for various extrusion conditions, average rod yields of both types from Table II, as well as average percentages of back end beta checking.

FIGURE 1

FREQUENCY OF ROD DIAMETER MEASUREMENTS

2 INCHES FROM LEAD END

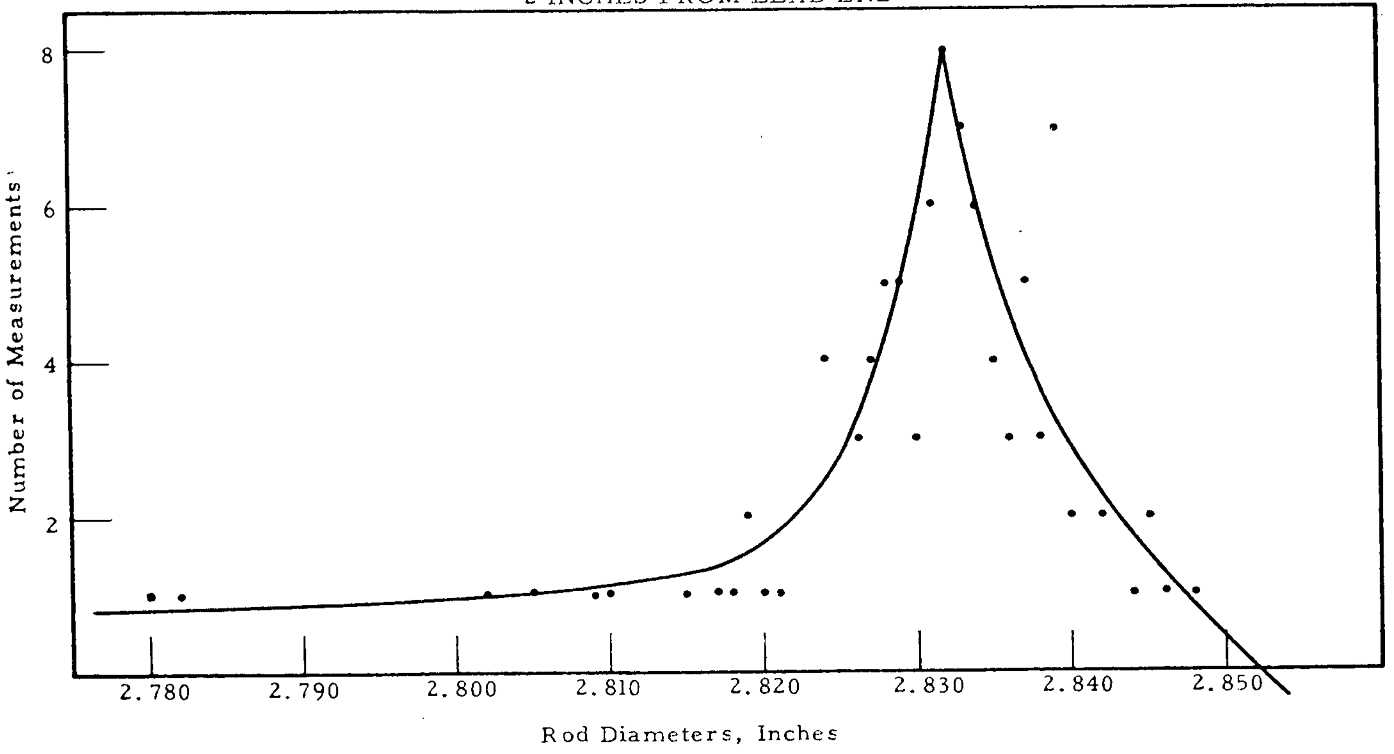

Table II

Individual Rod Yields for Third and Fourth Gamma Extrusion Campaigns

Billet Number	Die Number	Die Type	Lubrication		Follower Block Material	Billet and Follower Block Temperature °F	Yield Based on Butt and Pipe Loss[b] %	Yield Based on Rod Surface Quality[c] %
			Die & Liner	Billet[a]				
16	8	Flow without cone	Oil dag	Salt only	Graphite	1800	83.1	29
17	8	Flow without cone	None	Salt only	Steel	1800	82.7	0
18	8	Flow without cone	Oil dag	Salt only	Steel	1800	82.2	9
19	8	Flow without cone	None	Salt only	Graphite	1800	86.8	76
20	7	Flow with cone	None	Salt only	None	1800	84.3	13
21	7	Flow without cone	Oil dag	Salt only	Steel	1800	85.2	44
22	7	Flow without cone	Oil dag	Salt only	None	1800	88.3	38
23	7	Flow without cone	None	Salt only	Steel	1800	79.3	77
24	7	Flow without cone	None	Salt only	Graphite	1800	87.5	86
25	7	Flow without cone	Oil dag	Salt only	Steel	1800	85.1	61
26	7	Flow without cone	None	Salt only	None	1800	85.0	61
27	7	Flow without cone	Oil dag	Salt only	Graphite	1800	86.5	51

Table II (continued)

Billet Number	Die Number	Die Type	Lubrication Die & Liner	Lubrication Billet[a]	Follower Block Material	Billet and Follower Block Temperature °F	Yield Based on Butt and Pipe Loss[b] %	Yield Based on Rod Surface Quality[c] %
28	7	Flow without cone	None	Salt only	None	1900	88.2	95
29	7	Flow without cone	None	Salt only	Steel	1900	82.5	62
30	7	Flow without cone	Oil dag	Salt only	Steel	1900	74.2	86
31	7	Flow without cone	None	Salt only	Graphite	1900	90.5	93
32	7	Flow without cone	Oil dag	Salt only	None	1900	81.2	53
33	7	Flow without cone	Oil dag	Salt only	Graphite	1900	83.1	89
34	7	Flow without cone	Oil dag	Salt only	Steel	1900	84.6	92
35	7	Flow without cone	None	Salt only	Graphite	1900	91.4	41
36	7	Flow without cone	Oil dag	Salt only	None	1900	85.3	65
37	7	Flow without cone	None	Salt only	Steel	1900	82.9	89
38	7	Flow without cone	Oil dag	Salt only	Graphite	1900	87.2	89
39	7	Flow without cone	None	Salt only	None	1900	79.1	62
40	3	Flow with cone	Glass No.2	Glass No.2	Graphite	1800	84.8	68

Table II (continued)

Billet Number	Die Number	Die Type	Lubrication		Follower Block Material	Billet and Follower Block Temperature °F	Yield Based on Butt and Pipe Loss[b] %	Yield Based on Rod Surface Quality[c] %
			Die & Liner	Billet[a]				
41	3	Flow with cone	Glass No.2	Glass No.2	Steel	1800	87.6	69
42	3	Flow with cone	Glass No.4	Glass No.3	Graphite	1800	89.0	0
43	3	Flow with cone	Glass No.3	Glass No.3	Steel	1800	87.9	97
44	3	Flow with cone	Glass No.3	Glass No.3	Graphite	1800	90.9	98
45	3	Flow with cone	Glass No.4	Glass No.4	Graphite	1800	86.3	100
46	3	Flow with cone	Glass No.4	Glass No.4	Steel	1800	82.0	88
47	3	Flow with cone	Glass No.2	Glass No.2	Graphite	1800	91.1	68
48	3	Flow with cone	None	Salt only	None	1800	87.0	51

[a] Billets rolled in glass also covered with salt.

[b] Weight rod after cropping pipe (lbs.) × 100 ÷ weight billet

[c] Length of rod containing no surface defects (inches) × 4.4 lbs per inch of rod × 100 ÷ weight of billet (lbs.)

Table III

Average Rod Yields by Extrusion Conditions–Third and Fourth Gamma Extrusion Campaigns

Billet Temperature °F	Follower Block Material	Lubrication	Yield Based on Butt and Pipe loss[a] %	Average Back End Beta Checking[b] %	Yield Based on Rod Surface Quality[c] %
1800 and 1900	Graphite[d]	Glass or Oil Dag	87.6		
1800 and 1900	Steel[d]	Glass or Oil Dag	83.1		
1800 and 1900	None	Glass or Oil Dag	84.8		
1800	Graphite or Steel[d]	Glass	87.5	14	74
1800	Graphite[d]	Glass	88.4		
1800	Steel[d]	Glass	85.8		
1800	Graphite, Steel or None[d]	Oil Dag	84.8		
1800	Graphite or Steel[d]	Oil Dag	84.3	14	49
1800	Graphite[d]	Oil Dag	86.0		
1800	Steel[d]	Oil Dag	82.9		
1800	None	Oil Dag	86.2	6	41
1900	Graphite, Steel or None[d]	Oil Dag	84.2		
1900	Graphite or Steel[d]	Oil Dag	84.7	4	80
1900	Graphite[d]	Oil Dag	88.1		
1900	Steel[d]	Oil Dag	81.1		
1900	None	Oil Dag	83.5	2	69

Table III (continued)

[a] Weight rod after cropping pipe (lbs) × 100 ÷ weight billet (lbs.)

[b] Length back end beta checking (inches) × 4.4 lbs per inch of rod × 100 ÷ weight billet (lbs)

[c] Length of rod containing no surface defects (inches) × 4.4 lbs per inch of rod × 100 ÷ weight of billet (lbs)

[d] Follower block at same temperature as billet

Table III shows that:

a. Higher yields, based on either butt and pipe losses or rod surface quality, resulted when glass was used as a lubricant with 1800°F billet temperatures, regardless of the follower block material used.

b. Higher yields based on butt and pipe losses, were obtained with graphite follower blocks, regardless of the billet temperatures or lubricant used.

c. Higher yields, based on rod surface quality, were obtained with 1900°F billet temperatures, regardless of the follower block material used.

d. Beta checking was more pronounced with 1800°F billet temperatures in all cases.

e. Since back end beta checking caused fourteen of the twenty-six percent of surface defective rods extruded with glass as a lubricant, it is reasonable to assume that higher yields than any shown in Table III would be achieved by using glass as a lubricant with 1900°F billet temperatures.

The fact that higher yields, based on butt and pipe losses, were obtained with no follower blocks than with steel follower blocks is not readily explained. A possible explanation for the lesser amount of back-end beta checking when no follower blocks were used is that the billets spent a few seconds less time in the container and therefore had less opportunity to cool.

While the data is not shown in either Table II or Table III, billets 18 inches long had an average yield of 85.8 percent as opposed to 84.4 percent for those less than 18 inches long. This is due, of course, to the relatively uniform butt loss for all billets, which is a higher percentage of the shorter billets.

3. Hydrogen Pickup

Hydrogen analyses were performed at the center and edge of transverse slices taken from the middle of the good portion of each rod to determine whether any hydrogen pickup resulted from heating the billets in Houghton Liquid Heat 1450 salt for 40 to 85 minutes, at temperatures of 1800° and 1900°F. The hydrogen content of the billets before extrusion averaged 3.0 ppm. The extruded rods averaged 3.1 ppm and 3.2 ppm hydrogen at the edge and center respectively. Some of the hydrogen contents reported for the extruded rods were actually lower than those reported originally for the corresponding billets. This was probably caused by the limit of accuracy of the hydrogen analyses. The small average increases of 0.1 ppm to 0.2 ppm are considered negligible.

4. Surface Quality of Rods.

Rods extruded with glass lubrication exhibited shallow grooving, or gouging, on their entire surface, probably caused by incomplete fusion of the glass at the periphery of the die orifice. Figure 2 is a transverse section of a rod having such grooves, which were not considered serious enough to be included as surface defects in calculating the yields in Tables II and III. These rods exhibited relatively few scabs and blisters, although one rod, number 42, had a narrow row of blisters extending its entire length.

Rods extruded with oil dag lubrication exhibited numerous areas of scabs and blisters. These defects are believed to be different degrees or forms of a single defect. The exact cause of their occurrence is unknown at this time. Figure 3 and 4 are transverse sections of rods having blisters and scabs respectively. Those areas of the rods extruded with oil dag lubrication which were not scabbed or blistered were smoother than the rods extruded with glass lubrication.

Photographs of the outside surfaces of the rods shown in Figures 2, 3, and 4 appeared in the previous quarterly.[4]

5. Follower Block Material

The superiority of graphite follower blocks, as they affect rod yields, was discussed above. Advantages of graphite follower blocks are that they can be readily partially extruded through the die, leaving virtually no butt material in the container. Furthermore they form a regular, smooth, clean cone at the back end of the rod, as shown in Figures 5 and 6. The rough, irregular extrusion defects encountered when using hot steel follower blocks or no follower blocks at all are shown in Figures 7, 8, and 9.

6. Metallographic Examination

The appearance of the macrostructure and the microstructure was observed on nine of the rods in this campaign. Each rod was examined at the lead end, the middle, and adjacent to the butt end. At each of these locations, transverse and longitudinal photomacrographs and photomicrographs were obtained.

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[4] *Ibid.*

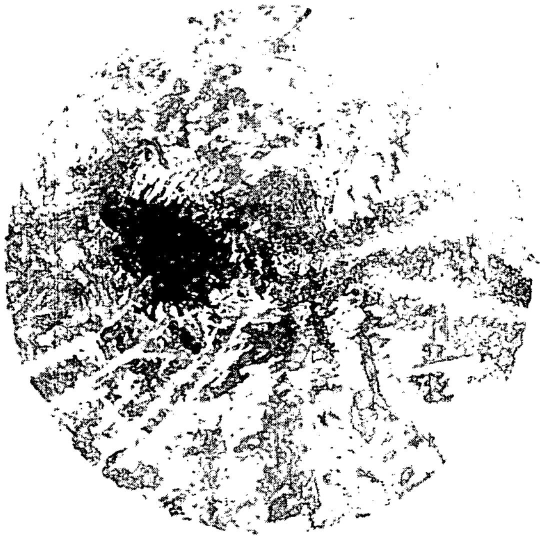


Figure 2

Transverse Section at Middle of Rod 47
Showing Grooving. (Glass Lubricant)
Approximately 1X

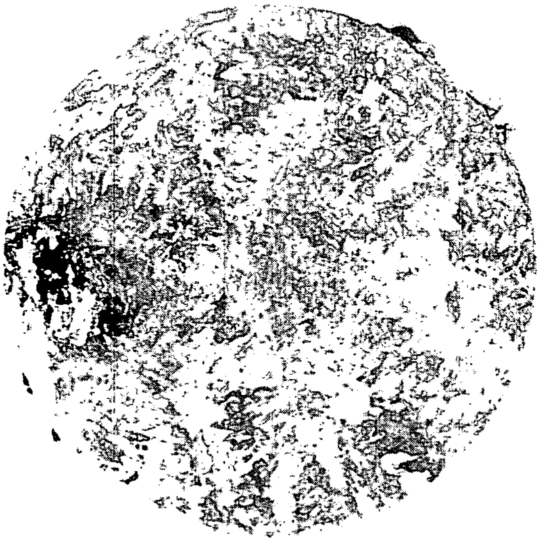
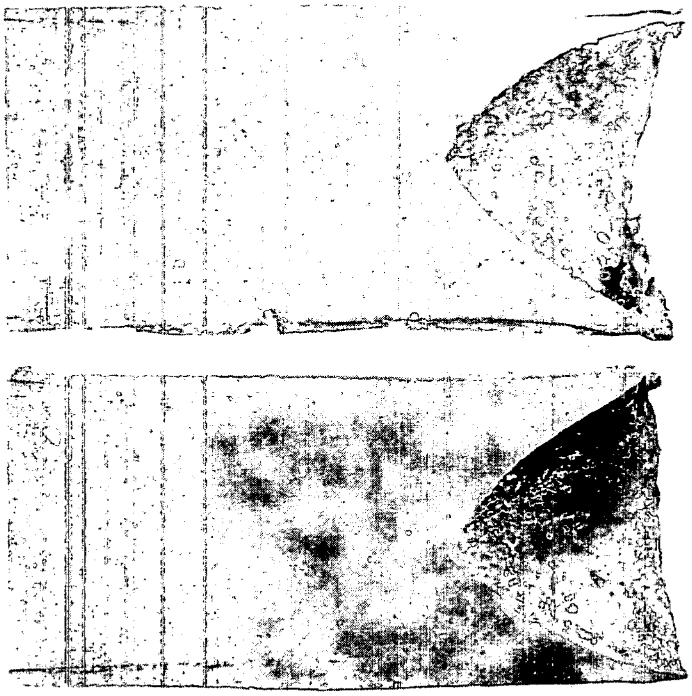


Figure 3

Transverse Section at Back End of Rod 36
Showing Blisters. (Oil Dag Lubricant)
Approximately 1X

Figure 4

Transverse Section at Back End of Rod 33 Showing Scab.
(Oil Dag Lubricant) Approximately 1X



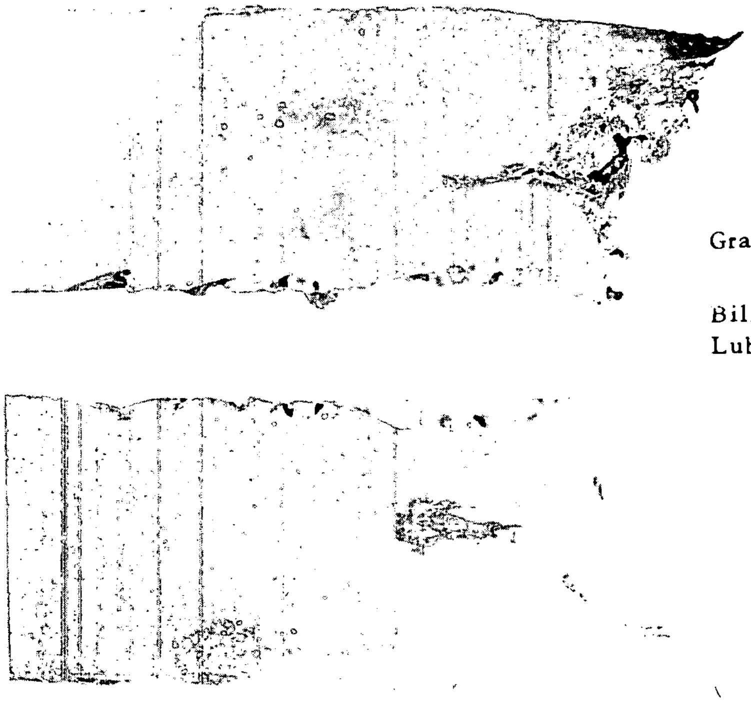
Graphite Follower Block

Billet Temp. - 1900°F

Lubricant - Oil Dag

Figure 5

Longitudinal Section of Crop from Back End of Rod No. 31



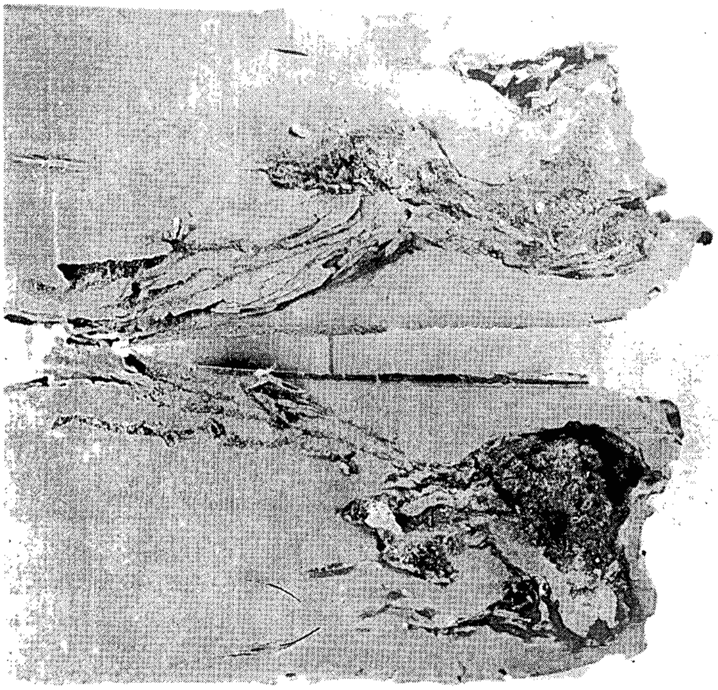
Graphite Follower Block

Billet Temp. - 1800°F

Lubricant - Glass

Figure 6

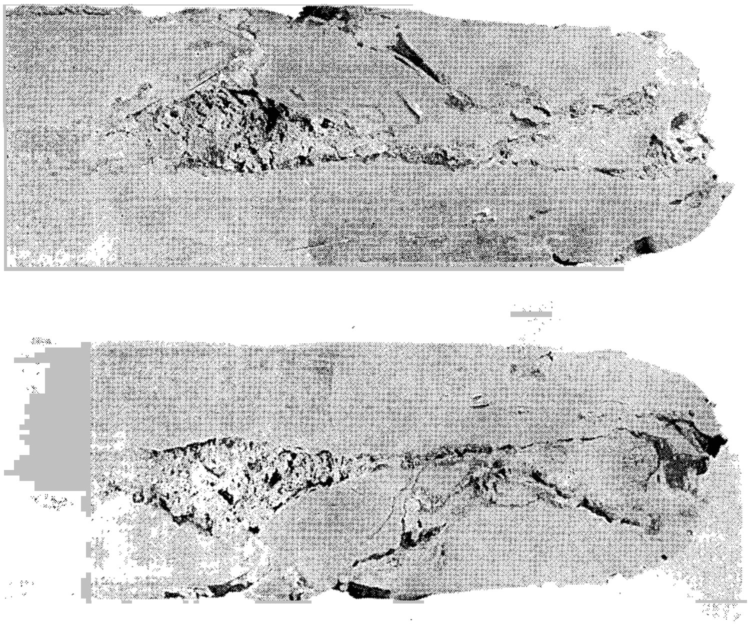
Longitudinal Section of Crop from Back End of Rod No. 44



Steel Follower
Block
Billet
Temp.-1900° F.
Lubricant
Oil Dag

Figure 7

Longitudinal Section of Crop from Back End of Rod No. 30



No.
Follower Block
Billet
Temp.- 1900° F
Lubricant
Oil Dag

Figure 8

Longitudinal Section of Crop from Back End of Rod No. 32

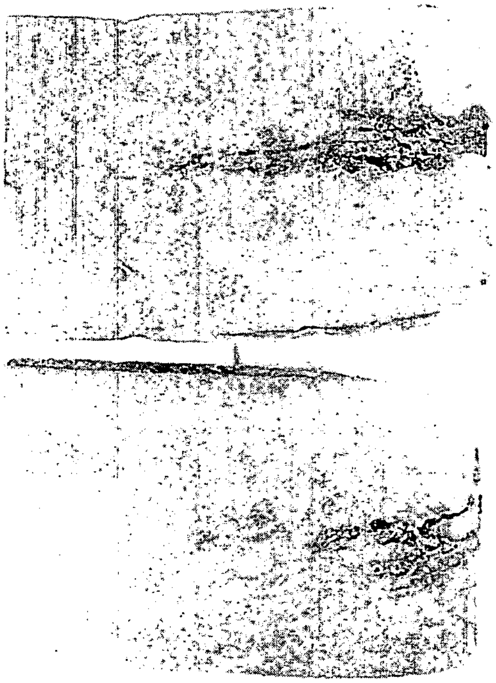


Figure 9

Longitudinal Section of Crop from Back
End of Rod No. 41

Steel Follower Block
Billet Temp. - 1800°F
Lubricant - Glass

The macrostructures of all of the rods were quite similar. Each photomacrograph presented a pattern of grains which indicated a strong radial orientation. The direction of grain growth is consistent with the direction of heat flow out of the rod upon cooling from the gamma extrusion temperature. Figure 10 and 11 show macrostructures which are typical of all of the rods studied.

The microstructures are again all quite similar. Each presents an appearance typical of a beta heat-treated and water quenched material. This indicates that the cooling of the rods from the extrusion temperature must have been rather rapid, otherwise the more rounded appearance of the alpha annealed structure would have been evident. A photomicrograph of a typical structure is shown in Figure 12.

Although there were minor variations in structure between samples there were no consistent differences that could be attributed to extrusion variables.

IV. Performance of Fifth Campaign

A. Purpose

The purpose of the fifth campaign, performed on May 1 and 2, 1957, was to evaluate the effect of glass and "oil dag" as lubricants on die wear, and to compare the grain structure of rods extruded from as-cast and forged dingot metal. Two variations of the accepted method of applying glass lubricant were also tried.

In order to minimize the variables involved, the conditions listed below were held constant. Knowledge gained from previous work indicated at the time, that these conditions would be desirable.

Billet temperature - 1800°F

Follower block temperature - 2000°F

Liner temperature - 800°F

Ram speed - approximately 250 inches/minute

Dies - 2.910 inches diameter, flow type as described in the previous quarterly[5], Rex AA high speed tool steel.

Follower block material - graphite

Follower block partially extruded behind each billet.

Each rod rolled in and sprinkled with Houghton Heat 980 salt immediately after extruding.

Table IV shows all of the data collected during the fifth campaign.

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[5] *Ibid*

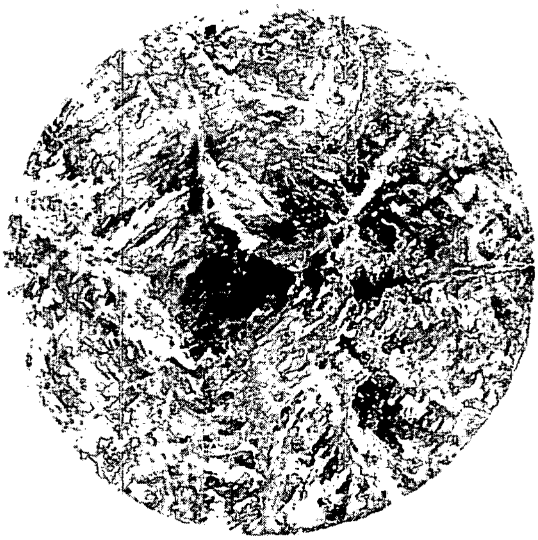

Figure 10

A Transverse Section Typical of the Rods in This Campaign. Approximately 1X

Figure 11

A Longitudinal Section Typical of the Rods in This Campaign. Approximately 1X

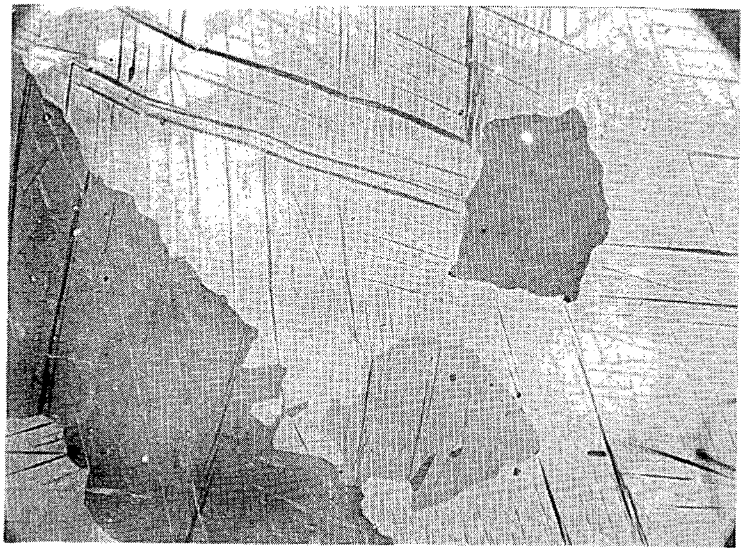

Figure 12

A Microstructure Typical of the Rods in This Campaign 100X - Polarized Light

Table IV

Data Sheet for Fifth Gamma Extrusion
at Bridgeport Brass Company
on May 1st and 2nd, 1957
Pages 128 to 133

Table IV

Data Sheet for Fifth Gamma Extrusion at Bridgeport Brass Company on May 1 and 2, 1957[a]

Time of Day	BBC Billet Number	MCW Billet Number	Billet Weight lb	Billet Length in	Salt Bath Time min	Transfer Time sec	Extrusion Time sec
May 1, 1957							
9:56 A.M.	49	21664-E	347	13 ⅞	55	97	7.4
10:12 A.M.	50	21664-D	338.5	13 ½	78	113	3.2
10:24 A.M.	51	21664-C	363	14 ½	95	105	3.3
10:35 A.M.	52	21706-B	448	17 ⅞	110	123	3.5
10:47 A.M.	53	21664-B	452	18	88	117	4.3
11:47 A.M.	54	21706-A	447.5	17 ⅞	145	104	4.3
1:50 P.M.	55	21664-A	450.5	18	227	107	4.3
2:01 P.M.	56	21706-C	450	18	220	119	3.4
2:09 P.M.	57	21706-D	328.5	13 ⅛	220	94	2.7
2:14 P.M.	58	21729-E	359.5	14 ⅜	210	97	3.8
2:21 P.M.	59	21706-E	353.5	14 ⅛	175	106	3.6
2:27 P.M.	60	21729-D	357	14 ¼	81	99	3.5
May 2, 1957							
8:17 A.M.	61	21734-A	450	18	80	104	4.8
8:30 A.M.	62	21729-A	449	18	92	107	4.8
8:41 A.M.	63	21729-B	449.5	18	100	98	5.4

[a] In all cases the butt consisted of a number of small pieces.

				"K" Extrusion Constant		
Ram Speed in/min	Die No.	Cone No.	Lubrication	Pressure Gauge psi	Strain Gauge psi	Remarks
113	1	4	Rolled in Glass Glass disc	1372		Die cold at start, tool oven not up to temperature. Cold follower block due to delay out of salt bath
253	1	2	Rolled in Glass Glass disc	2744		
264	1	1	Rolled in Glass Glass disc	3430		
309	1	3	Rolled in Glass Glass disc	3430		
251	1	5	Rolled in Glass Glass disc	2058		
251	1	4	Rolled in Glass Glass disc	4802		Salt bath out of order, billet not up to temperature.
251	1	2	Rolled in Glass Glass disc	2744	1767	
318	1	1	Rolled in Glass Glass disc	3430	1920	Good Glass coating
292	1	3	Rolled in Glass Glass disc	2744	1657	Good Glass coating
227	1	5	Rolled in Glass Glass disc	2744	1614	Good Glass coating
235	1	4	Rolled in Glass Glass disc	2744	1758	Good Glass coating
244	1	2	Rolled in Glass Glass disc	3087	1684	Good Glass coating
225	1	1	Rolled in Glass Glass disc	2744	2030	Follower Block at 1800°F
225	1	3	Rolled in Glass Glass disc	2744	1822	Follower Block at 1800°F
200	1	5	Rolled in Glass Glass disc	2744	1906	Follower Block at 2000°F

continued on next page

Table IV (continued)

Data Sheet for Fifth Gamma Extrusion at Bridgeport Brass Company on May 1 and 2, 1957[a]

Time of Day	BBC Billet Number	MCW Billet Number	Billet Weight lb	Billet Length in	Salt Bath Time min	Transfer Time sec	Extrusion Time sec
May 2, 1957							
8:51 A.M.	64	21729-C	447	17⅞	111	112	4.8
9:13 A.M.	65	21734-B	450.5	18	122	105	4.8
9:26 A.M.	66	21738-B	450	18	133	136	4.4
9:38 A.M.	67	21738-A	454.5	18⅛	120	109	3.6
9:52 A.M.	68	21738-C	452	18	75	100	3.6
10:02 A.M.	69	21734-C	356	14¼	75	113	3.1
10:15 A.M.	70	21738-D	334	13⅜	80	99	3.5
10:28 A.M.	71	21738-E	335	13⅜	69	110	3.0
10:39 A.M.	72	21734-D	362.5	14½	67	113	–
11:30 A.M.	73	21753-B	452	18	108	106	4.2
11:42 A.M.	74	21745-C	441.5	17⅝	105	103	4.2
11:51 A.M.	75	21753-A	450.5	18	102	104	3.6
12:02 P.M.	76	21753-D	440.5	17⅝	99	116	5.4
12:12 P.M.	77	21753-C	450.5	18	100	109	5.4
12:22 P.M.	78	21745-A	448.5	17⅞	100	111	4.8
12:38 P.M.	79	21800-C	392.5	15⅝	60	81	3.6
12:49 P.M.	80	21800-B	388.5	15½	60	182	4.0

[a] In all cases the butt consisted of a number of small pieces.

				"K" Extrusion Constant		
Ram Speed in/min	Die No.	Cone No.	Lubrication	Pressure Gauge psi	Strain Gauge psi	Remarks
223	1	4	Rolled in Glass Glass disc	2744	1894	
225	1	2	Rolled in Glass Glass disc	2744		Die measured 2.905 ±0.003
245	1	1	Rolled in Glass Glass disc	2744		
302	1	3	Rolled in Glass Glass disc	3430		Die measured 2.917 ±0.003 Same as new die
300	1	5	Rolled in Glass Glass disc	2744		
276	1	4	Rolled in Glass Glass disc	3430		
229	1	2	Rolled in Glass Glass disc	2744		
268	1	1	Rolled in Glass Glass disc	2744		
–	1	3	Rolled in Glass Glass disc	2744		Pushed in two steps, dummy block left out first time
257	1	5	Rolled in Glass Glass disc	2744		
252	1	4	Rolled in Glass Glass disc	2744		
300	1	2	Rolled in Glass Glass disc	2058		
196	1	1	Rolled in Glass Glass disc	4116		
200	1	3	Rolled in Glass Glass disc	4116		
223	1	5	Rolled in Glass Glass disc	4116		Die changed, still in good condition.
260	2	4	Glass disc only	3430	1880	
233	2	2	Glass disc only	5488	2657	Delay in entering press

continued on next page

Table IV (continued)

Data Sheet for Fifth Gamma Extrusion at Bridgeport Brass Company on May 1 and 2, 1957[a]

Time of Day	BBC Billet Number	MCW Billet Number	Billet Weight lb	Billet Length in	Salt Bath Time min	Transfer Time sec	Extrusion Time sec
May 2, 1957							
12:59 P.M.	81	21800-A	392.5	15 5/8	60	82	3.5
1:23 P.M.	82	21799-B	392	15 5/8	73	107	2.9
1:33 P.M.	83	21799-A	391	15 5/8	75	110	3.3
1:43 P.M.	84	21799-C	372.5	14 7/8	73	77	3.7
1:51 P.M.	85	21745-D	419.5	16 3/4	66	83	3.9
2:00 P.M.	86	21745-B	443.5	17 3/4	68	81	4.2
2:08 P.M.	87	21756-B	450	18	65	75	4.6
2:15 P.M.	88	21756-C	449.5	18	49	74	5.4
2:23 P.M.	89	21756-A	448	17 7/8	48	81	4.5
2:30 P.M.	90	21756-D	442	17 5/8	47	79	4.7
2:37 P.M.	91	21704-D	341	13 5/8	45	79	3.7

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[a] In all cases the butt consisted of a number of small pieces.

				"K" Extrusion Constant		
Ram Speed in/min	Die No.	Cone No.	Lubrication	Pressure Gauge psi	Strain Gauge psi	Remarks
268	2	1	Glass disc only	4802	1880	
323	2	3	Rolled in Glass	5488	2270	
270	2	5	Glass sprinkled on top only	6174		Die striated on one side
272	3	None	Oil Dag 170 Die and liner	5488	2157	
258	3	None	Oil Dag 170 Die and liner	5488	2226	Die began to striate on inlet radius and land
254	3	None	Oil Dag 170 Die and liner	5488		
235	3	None	Oil Dag 170 Die and liner	5488	1894	
200	3	None	Oil Dag 170 Die and liner	5488	2088	
238	3	None	Oil Dag 170 Die and liner	4802		
225	3	None	Oil Dag 170 Die and Liner	5488	2241	
221	3	None	Oil Dag 170 Die and Liner	6860		Die badly striated and oversize due to grinding

B. Experimental Work

1. Glass Lubricant

Thirty billets, $6\frac{13}{16}$ in. in diameter and $13\frac{1}{8}$ to 18 in. long were extruded through one die in an effort to determine the life of the die when using glass as a lubricant. These billets were rolled in −100 mesh Thomas C. Thompson No. 1852-Y2 glass[6] just before placing them in the container, and a disc, composed of −20, + 100 mesh glass of the same composition and water glass, was placed in the container in front of each billet. Flow cones having a 130° included angle were used with all of these billets. The die was examined frequently during the extrusion of these billets; the cones were changed and examined after the extrusion of each billet.

2. Oil Dag Lubricant

Eight billets, $6\frac{13}{16}$ in. in diameter and between $13\frac{5}{8}$ and 18 in. long, were extruded through one die in an effort to determine the life of the die when using oil dag as a lubricant. Oil dag No. 170 was sprayed in the container liner and on the die before each of these billets was extruded. No lubricant was applied to the billets, although, of course, each was covered with a layer of salt from the salt bath. No flow cones were used in extruding these billets. The die was examined after extruding each billet.

3. Glass Application

Five billets, $6\frac{13}{16}$ in. in diameter and $15\frac{1}{2}$ or $15\frac{1}{8}$ in. long, were extruded through one die in an effort to determine the effect of the method of glass application on die wear. Three were extruded with a glass disc in front of each, but without rolling in glass. Two were rolled in glass, but instead of using a glass disc in front of the billet, glass was sprinkled on the end of the billet just before rolling in glass. Unfortunately, practically all of the glass on the end of the billet was accidentally scraped off during the rolling operation in each case. Flow cones having a 130° included angle were used in extruding these billets. The die and cone were examined after extruding each billet.

4. Grain Structure

Thirty-seven of the forty-three billets used in this campaign were machined from $7\frac{1}{2}$ inch diameter round bars forged from dingots. Six billets were obtained by

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[6] *Ibid*

sawing two dingots into three 120° sectors each, and machining the billets from these sectors. These six billets, numbers 79 through 84 in Table IV, were prepared in this manner and extruded to study the effect of initial billet grain size and structure, either forged or as cast, on the grain size and structure of the extruded rod.

5. Extrusion Pressures

When extruding uranium in the gamma phase, the readings of the pressure gauge attached to the press used at Adrian were close to the bottom of the scale and unreliable. For the fifth campaign, strain gauges were attached to the ram (in such a manner that temperature changes would not affect the readings) and strain-time diagrams were made for the extrusion of each billet. The apparatus was calibrated by attaching strain gauges to a solid cylinder of the same diameter and material as the ram and subjecting this cylinder to know stresses in a compression testing machine.

C. Experimental Results

1. Die Life

When glass was used as a lubricant, the die showed no appreciable wear after thirty billets were extruded through it. This die is shown in Figure 13.

When oil dag was used as a lubricant, grooves developed on the inlet radius of the die after only three rods were extruded through it. After eight rods were extruded, serious grooves were present on the inlet radius and about half way through the land on the entire circumference of the orifice. However, the grooves did not extend entirely through the land at any point; about $\frac{1}{8}$ inch of the exit and of the land was undisturbed. This die is shown in Figure 14.

The die used in evaluating glass application methods showed no wear after extrusion of three billets with a glass disc in front of the billet but no glass on the periphery. After extruding two more billets, which were rolled in glass and had glass sprinkled on the end, this die had deep groves on the inlet radius and land over about 60° of the circumference. The balance of the orifice was undisturbed. The grooves on this die extended through the entire length of the land.

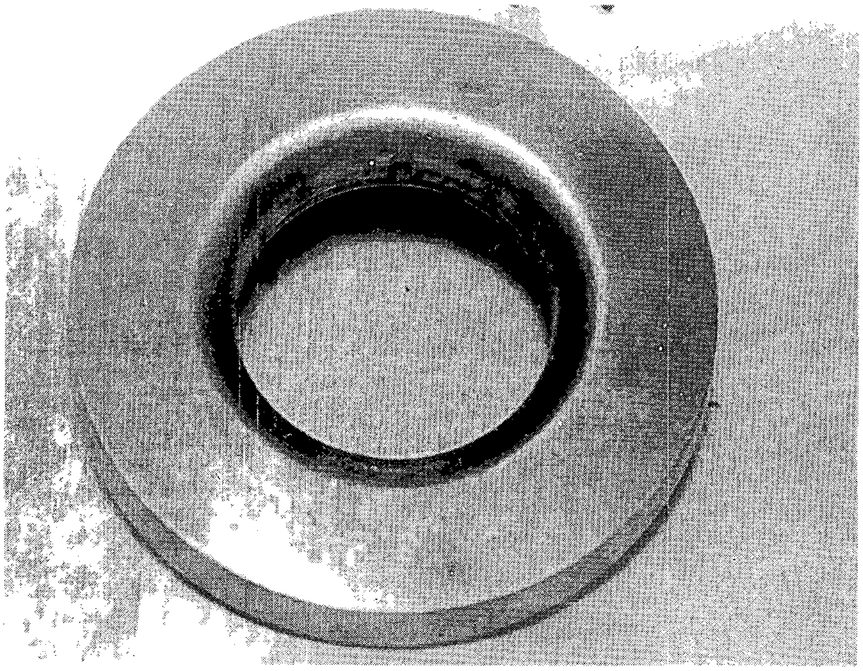

Figure 13

Die No. 1, Fifth Campaign, Used With Glass Lubricant

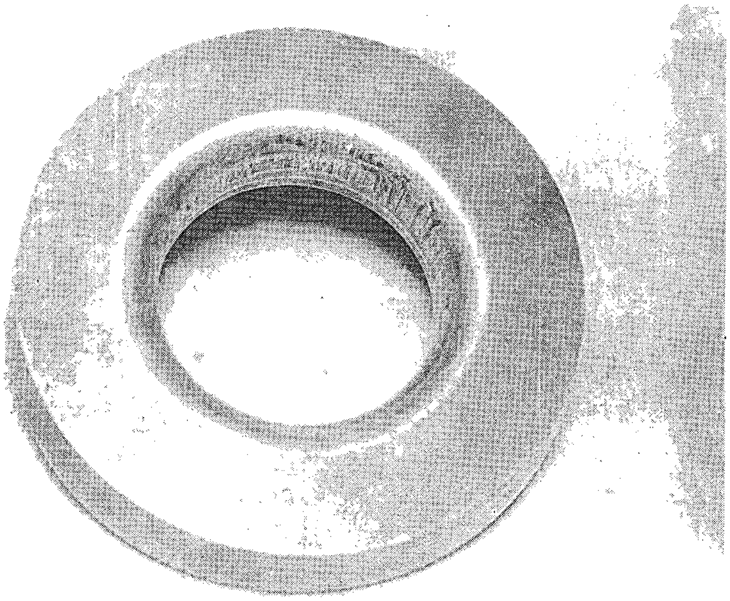

Figure 14

Die No. 3, Fifth Campaign, Used With Oil Dag Lubricant

2. Extrusion Pressures

Due to lack of experience in operating the strain recorder in this application, only nineteen of the forty-three strain diagrams made were considered reliable. These are not available for inclusion with this report, but the peak values were used for computing the extrusion constants "K", shown in Table IV. The extrusion constants based on pressure gauge readings, obtained for every billet, are also shown in Table IV for comparison. The higher extrusion constants obtained from calculations based on pressure gauge readings were probably due to the unreliability of the readings and the fact that the pressure required for overcoming friction between the parts of the press was included in the pressure gauge readings while it was not included in the extrusion pressures determined by the strain gauges.

The average of the ten extrusion constants calculated from strain gauge measurements for billets extruded with glass as a lubricant is 1805 psi, compared to an average of 2121 psi for the five billets extruded with oil dag as a lubricant. This, of course, indicated that the glass was a more effective lubricant for extrusion of uranium in the gamma phase than oil dag.

V. Future Work

Rods from the fifth campaign are being evaluated in generally the same manner as those from the third and fourth campaigns. Yields and rod diameters will be reported. A metallographic investigation will be conducted to determine the effect of initial grain size and structure on the grain size and structure of the extruded rod.

Other work in the immediate future will be directed toward improvement of yield and surface finish. Yield improvement will receive major emphasis. Specific items to be investigated in the next campaign at Adrian are listed below. To avoid beta checking on the back end of the rod as much as possible, all billets will be extruded at 1900°F in the next campaign.

a. Several billets having back ends contoured to the frustrum of a cone ("boat tails") will be extruded, in an effort to increase yield by decreasing "pipe". These contours will be made by:

   (1) Alpha forging in the extrusion press container, using a steel forging tool, and gamma extruding later.

   (2) Gamma forging in the extrusion press container, using a graphite follower block as a forging tool and extruding the billet immediately after forging.

(3) Making a 7 in. diameter, 20 in long dingot with the contour cast on the bottom end.

b. Several billets will be extruded through oval dies to obtain rods which can be used in the NLO continuous rod mill.

c. Several billets will be extruded through round shear type dies having chrome carbide inserts, using oil dag as a lubricant.

d. Spraying a suspension of glass ground in oil will be tried as a method of glass lubrication.

e. Several billets of uranium-chromium alloy and uranium-silicon alloy will be extruded to a size for machining directly to HAPO slugs.

VI. Acknowledgement

Metallographic work was completed and reported by H. Kloepper.

INVESTIGATION OF THE DINGOT PROCESS

by

L. L. Gidley
W. Taylor
H. Schaffer

I. Summary

The use of HF-filmed magnesium, rather than regular magnesium, appeared to result in higher bomb yields with high AOI green salt. This effect appeared to be the result of the longer firing times associated with filmed magnesium. Studies of the permeability of bomb liner materials have indicated that the particle size distribution of the liner material had a marked effect on the gas flow through the liner. Furthermore, it appeared that liner materials of various particle size fractions reached their maximum hardness after 400 jolts, irrespective of the size fraction. The finer size fractions reached higher jolted hardnesses than the coarse fractions.

II. Introduction

Uranium metal produced by the bomb reduction of uranium tetrafluoride with magnesium is obtained as a highly pure regulus. The dingot process was developed to produce a regulus which could be fabricated directly into fuel elements without a recasting operation and its attendent carbon contamination. Previous work has been described in past quarterly reports.[1] The production of 3300 lb dingots has been on a semi-production basis during all of 1956 and the first half of 1957. During the past quarter most of the experimental work on dingots has been devoted to the investigation of methods for reducing the hydrogen content of dingot metal and is reported in the section entitled "Hydrogen in Dingot Metal".[2] During the past quarter additional experiments were performed to investigate the effect of HF-filmed magnesium on the bomb yield when high AOI green salt is used as the charge and to investigate the effect of bomb liner material particle size and amount of jolting on the permeability and hardness of bomb liners.

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[1] Hansen, J. W., Schaffer, H. J., and Hartmann, R. F., *Process Development Quarterly Report, Part II*, Mallinckrodt Chemical Works, *MCW-1400* (February 1, 1957) p. 93

[2] *Cf.*, p. 65

III. Experimental Procedures and Discussion

A. Effect of Filmed Magnesium on Bomb Reduction with High AOI Green Salt

The purpose of this investigation was to determine whether a combination of filmed magnesium and green salt of greater than 3% AOI would produce satisfactory derby yields.

Previous AEC specifications have limited the maximum AOI to 2%. A relaxation of this specification would make possible an increase in the capacity of the green salt reactors without material increases in operating costs per unit time.

One derby bomb charged with 3.4% AOI green salt, and filmed Mg, had a firing time of 225 minutes and a crude yield of 95.28%. The appearance of the derby was slightly better than average for normal AOI green salt derbies. On the basis of this result, a more thorough investigation of filmed magnesium was inaugurated.

Twelve 12 inch derbies were fired from a lot of green salt having the following composition:

Assay: 95.01%
AOI: 3.56%
Water Soluble: 1.01%

Six of these derbies were made with magnesium fluorinated in the Plant 7 Rerun Reactor, and six were made with standard production grade magnesium. All of the derbies had MFL liners and vented caps. The bombs were fired with a 4% magnesium excess at a control temperature of 1250°F. The results of these runs are shown in Table I.

Table I

Comparison of Filmed and Regular Magnesium Fired with High AOI Green Salt in Twelve Inch Derbies

Run No.	Magnesium	Firing Time Mins.	Crude Yield %
4893	filmed	225	96.6
4894	filmed	245	97.6
4895	filmed	250	97.6
4896	filmed	270	97.9
4897	filmed	180	95.3
4898	filmed	227	97.9
4899	normal	155	92.0
4900	normal	135	86.8
4901	normal	185	93.5
4902	normal	165	96.6
4903	normal	150	90.1
4904	normal	165	94.0
Average	filmed	233	97.2
Average	normal	159	92.2

All the derbies fired with filmed magnesium, except 4897, had light gray product slag, a good slag - metal separation, and no entrapped slag in the metal. These derbies compared favorably with derbies from normal AOI green salt. Run 4897 had a lower firing time and yield.

Those derbies fired with normal magnesium had lower firing times and crude yields. The product slags were dark. There was a poor slag-metal separation and a great deal of slag entrapped in the metal. These derbies were very poor in appearance, as shown in Figures 1 thru 6.

Twelve more derbies, six with filmed magnesium and six with normal magnesium, were fired with the following furnace conditions:

Filmed magnesium	1300°F
Normal magnesium	1100°F

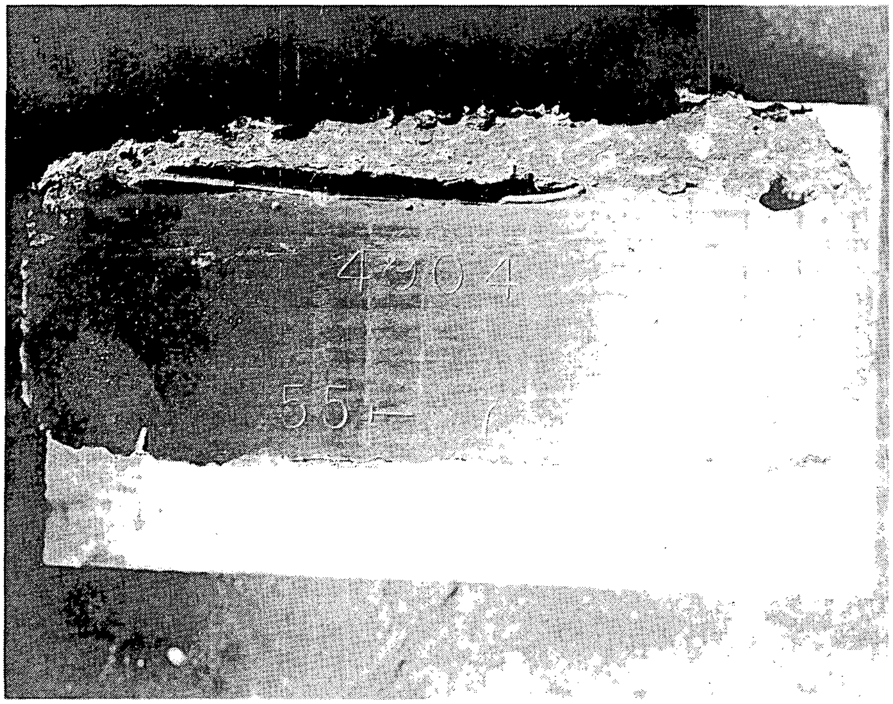

Figure 1

Cross Section of 12" Derby Made with Normal Magnesium

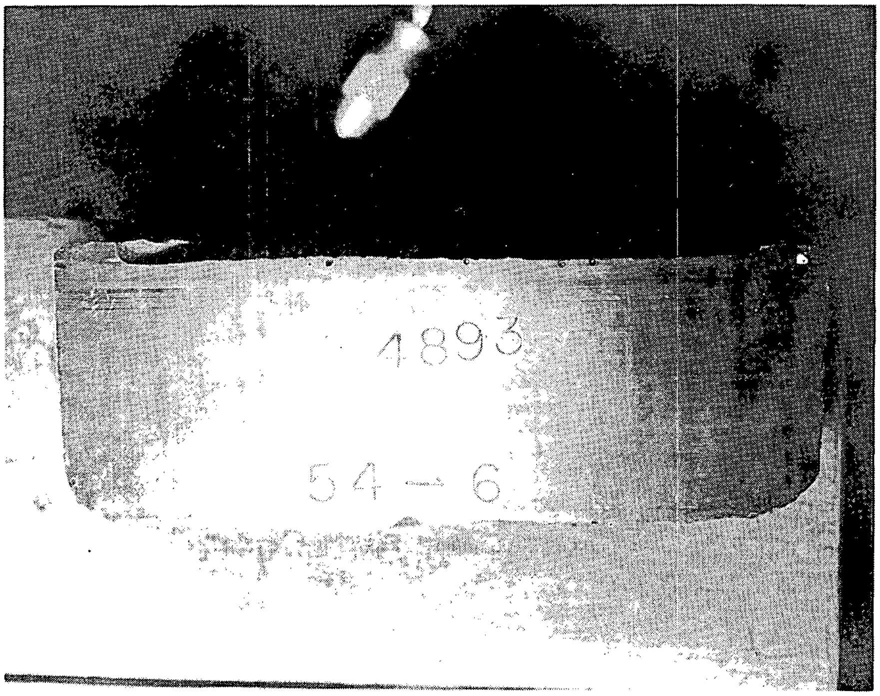

Figure 2

Cross Section of 12" Derby Made with Filmed Magnesium

Figure 3

Top View of 12" Derby Made With Normal Magnesium

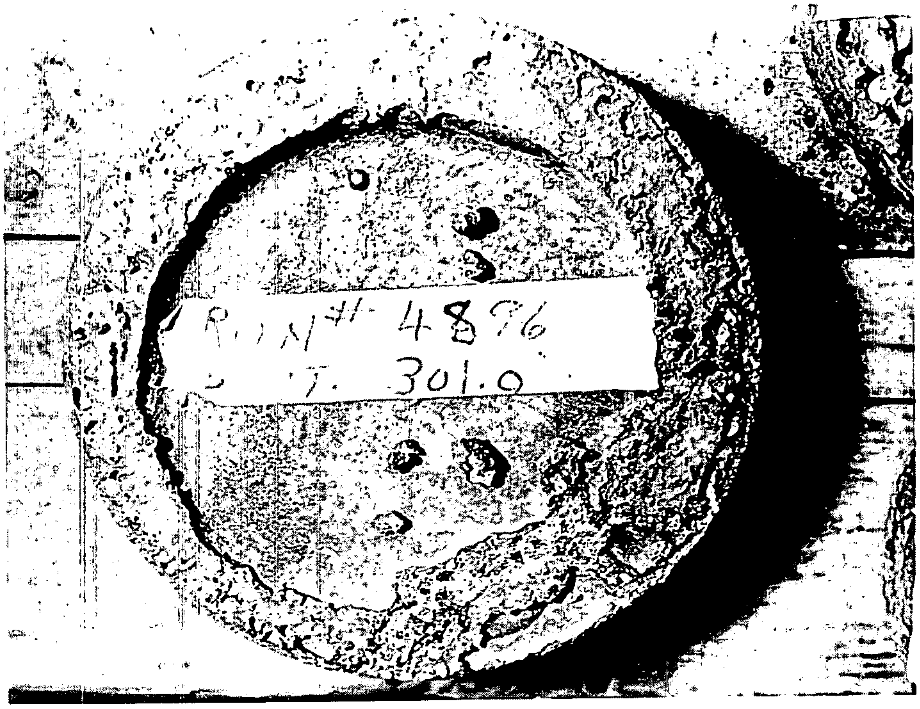

Figure 4

Top View of 12" Derby Made With Filmed Magnesium

Figure 5

12" Derbies Made With Normal Magnesium

Figure 6

12" Derbies Made With Filmed Magnesium

These derbies were fired to see whether the increased firing time or the filmed magnesium was directly responsible for giving good slag-metal separations and crude yields. Thus, the above temperatures were picked so that equalization of firing times could be realized. The green salt used had the following composition:

Assay: 95.74%
AOI: 3.06%
Water Soluble: 0.87%

The results are reported in Table II and the derby appearances are shown in Figures 7 through 16.

Table II

Comparison of 12-in Derby Results Obtained by Reacting High AOI Green Salt (3.06%) with Normal and Filmed Magnesium

Run No.	Magnesium	Firing Time Min.	Crude Yield %	Remarks
4918	Normal	265	96.2	good derby
4919	Normal	265	94.9	good derby
4920	Normal	225	92.0	slaggy derby
4921	Normal	350	95.2	good derby
4922	Normal	275	95.9	good derby
4923	Normal	210	90.6	slaggy derby
4924	Normal		96.2	slaggy derby (treated as fired).
4925	Filmed	225	91.0	good derby
4926	Filmed	160	86.5	fair derby
4927	Filmed	165	71.7	slaggy derby
4928	Filmed	295	83.9	very poor derby
4929	Filmed	190	94.3	good derby
Average	Normal	265	94.1	
Average	Filmed	207	90.6	

It appears that not the filmed magnesium, but the increase in firing time caused by the filmed magnesium may be responsible for good slag-metal separation when green salt above 3% AOI is used in bomb reduction.

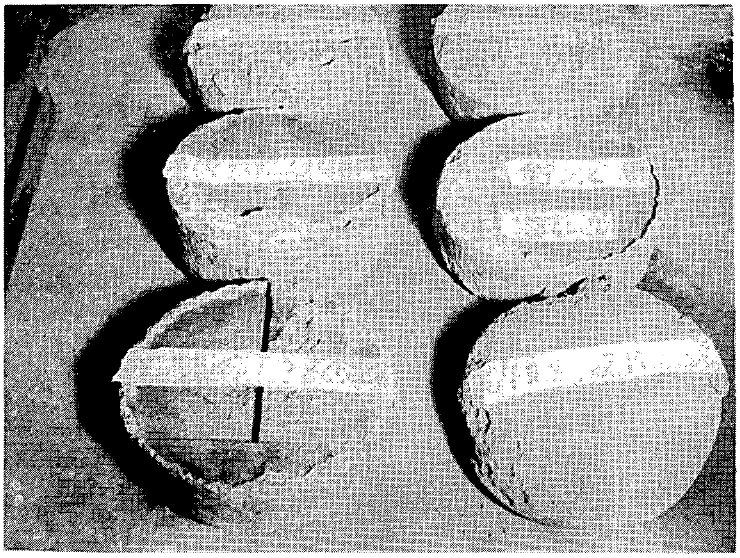

Figure 7

Appearance of Derby Tops

4927 4924
4929 4926
4925 4928

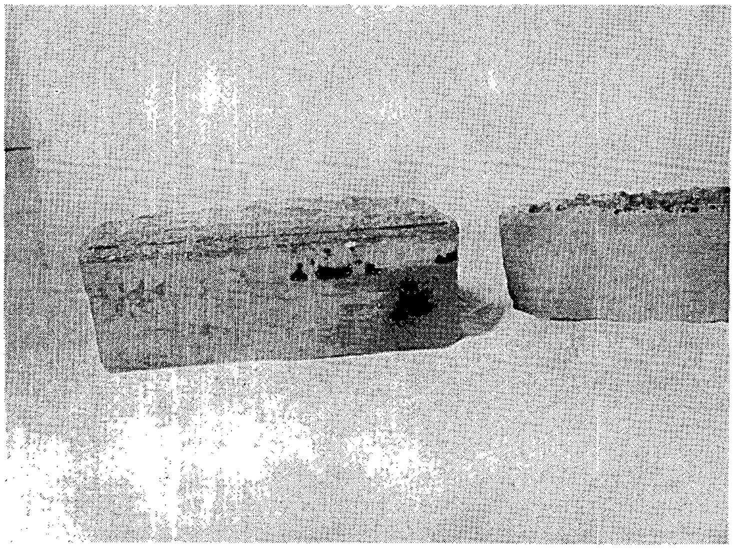

Figure 8

Sections Through Derbies

4924 4925

Figure 9

Appearance of Derby Tops

4920 4919
4921 4923
4922 4960

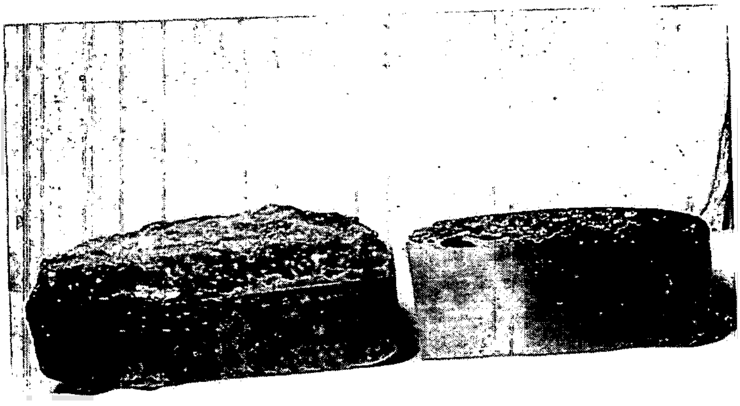

Figure 10

Sections Through Derbies

4923 4918

B. Bomb Liner Permeability and Hardness Studies

During this period, work hascontinued in obtaining relative permeabilities and hardness of various liner materials, so that an optimum liner, in terms of high resistance to gas flow and high liner hardness, may be achieved. A high pressure drop through the side liner was deemed necessary when gas purging of dingots was considered, so that the majority of the purge gas would travel through the charge. High liner hardness has been considered representative of good strength which is desired to avoid any mixing of liner with the charge during bomb filling. The procedure used for obtaining permeability data was described previously.[3]

The material investigated was blended to insure a more consistent particle size distribution.

To study the effect of particle size upon permeability, material "A" was formed by a partial sieving of the blended Vitro-roasted air-classified fines. Material "A" was taken from the fraction passing through the 200 (U.S.) mesh and retained on the 230 (U.S.) mesh screen. This material is, then a -200 (U.S.) mesh fraction of the original material. The experimental data for the blended Vitro-roasted air-classified fines, and material "A" are shown in Figures 11 and 12. These data indicated an increased gas velocity for increased pressure drop and a decreased gas velocity for an increasing number of jolts. From Figures 11 and 12, it is evident that the change in particle size distribution had a marked effect upon thegas flow through the liner. The same material was investigated with respect to liner hardness, the optimum liner, being one which gives a high hardness with a small number of jolts.

The procedure followed here was to load the glasspipe, shown in Figure 13, with a known weight of slag. The glass pipe was then placed on a jolting collar welded to a 12 inch bomb shell lid. A simulated iron mandrel was placed on top of the slag, and the apparatus was bolted down by means of a lid above the glass pipe, and four tie rods. A threaded mandrel shaft protruded through the lid. A bolt placed upon this shaft above the lid regulated the mandrel height, while a bolt below the lid regulated the pressure upon the slag. The slag was then given a controlled number of jolts. Hardnesses were measured after each jolt from zero to 10 jolts, after each 10 jolts

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[3] Becker, R. *et al, Process Development Quarterly Report, Part II,* Mallinckrodt Chemical Works, *MCW-1402,* (May 1, 1957) p. 118-119

FIGURE 11

ΔP/L *versus* FLOW FOR BLENDED VITRO ROASTED AIR CLASSIFIED FINES AND MATERIAL "A"

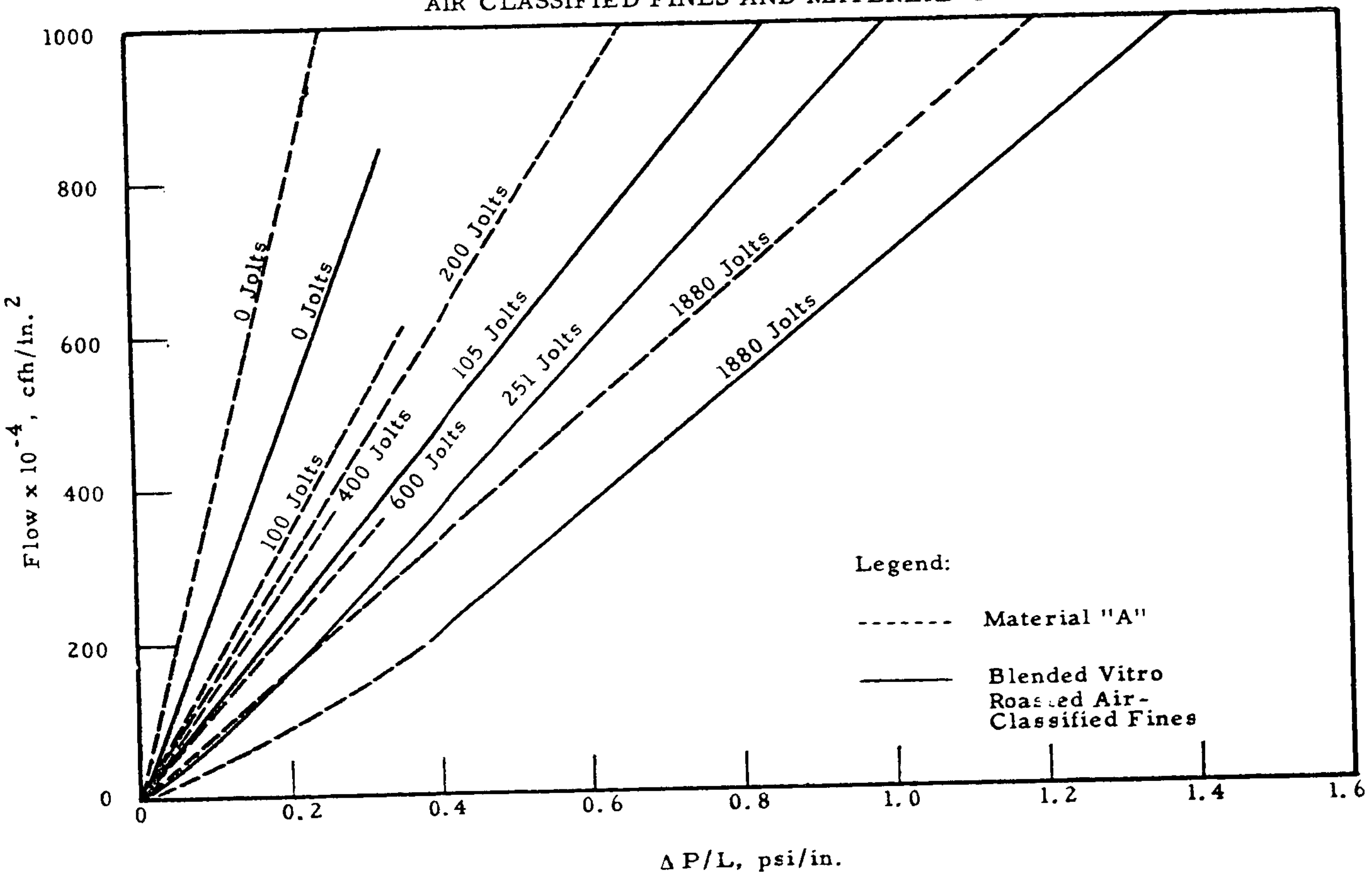


FIGURE 12

SIEVE ANALYSES FOR BLENDED VITRO ROASTED AIR CLASSIFIED FINES AND MATERIAL "A"

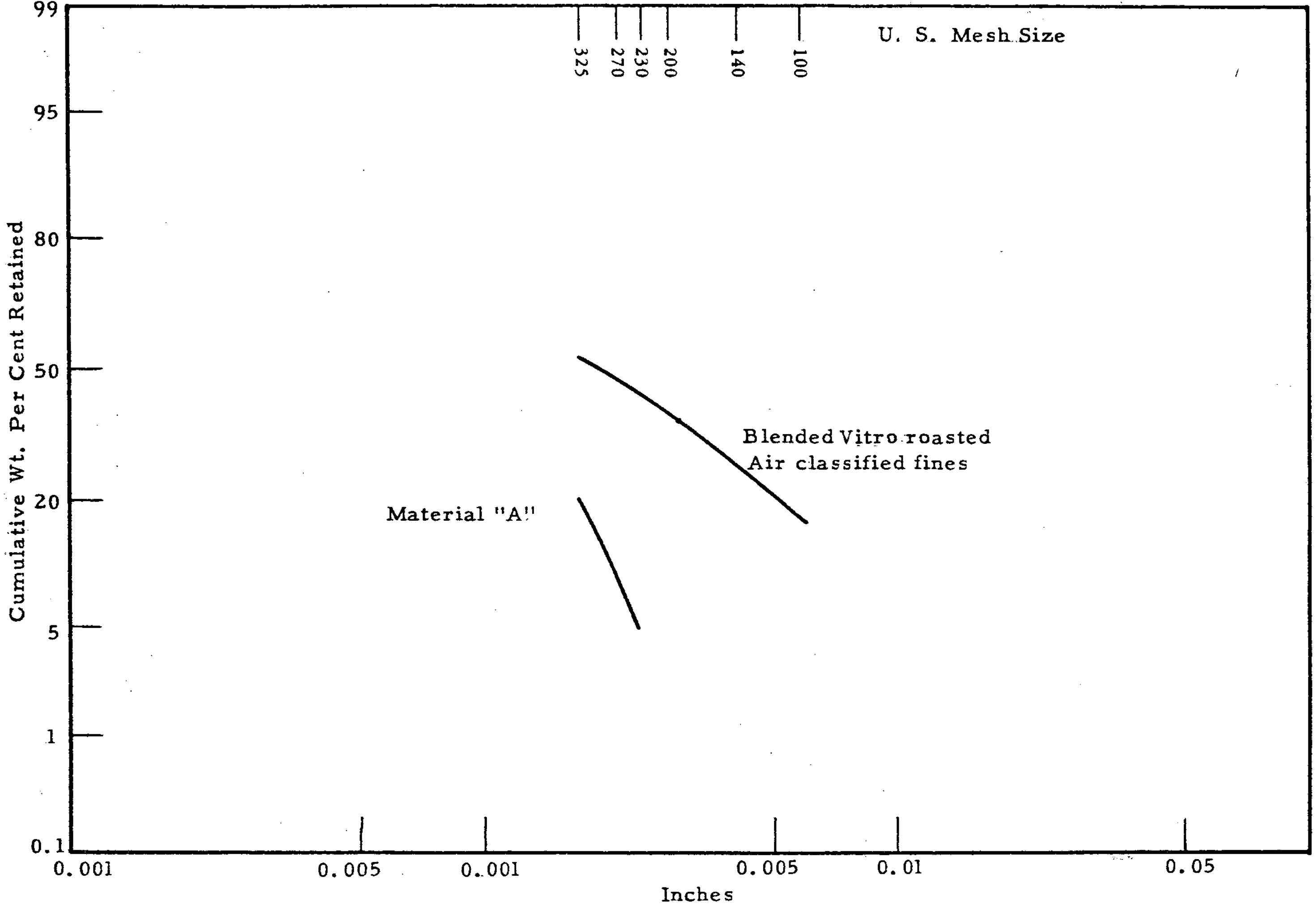
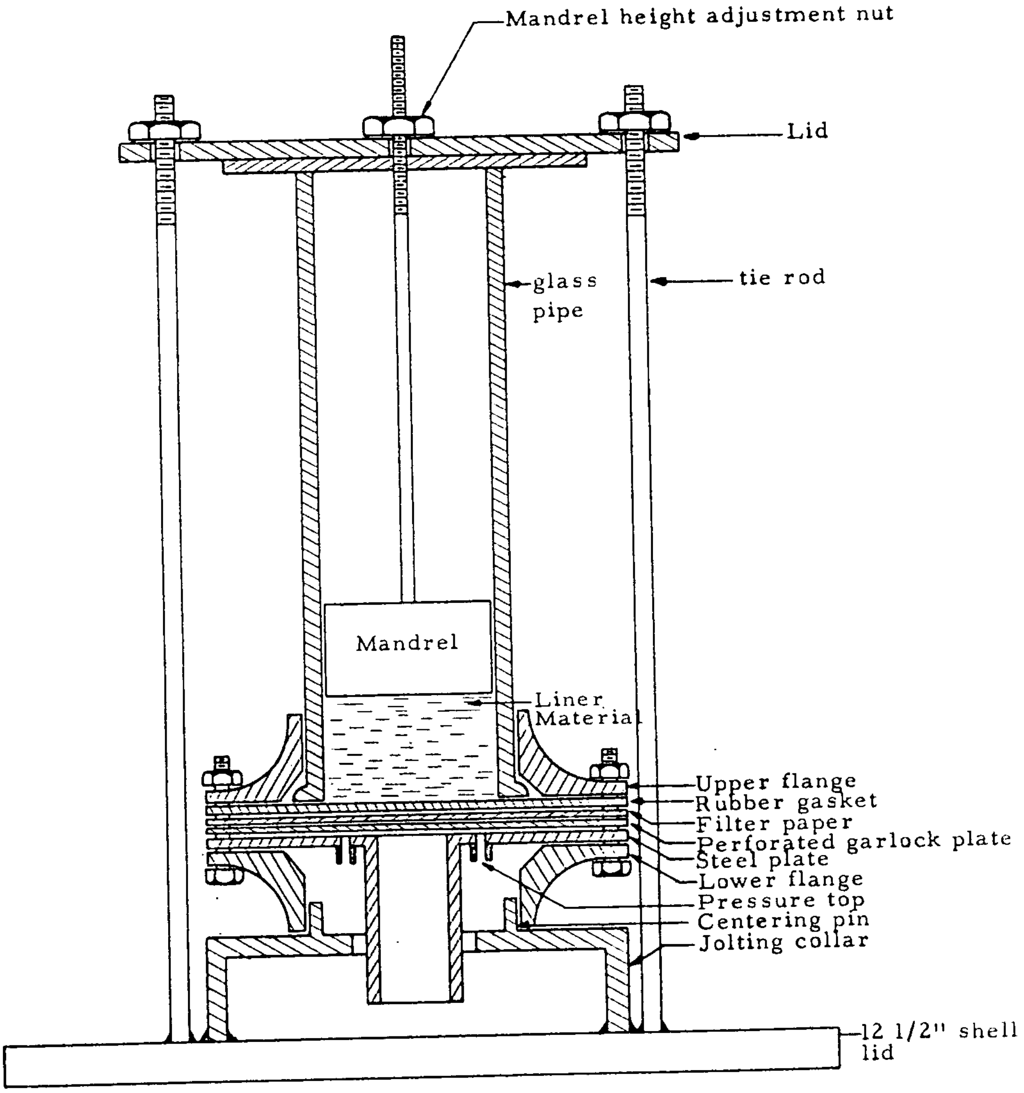


FIGURE 13

JOLTING APPARATUS



from 10 to 100 jolts, and after each 100 jolts, from 100 to 1880 jolts, by a "green" hardness gage.[4] The jolting rates were approximately 50 jolts/min and 250 jolts/min for slow and fast jolts, respectively, from 10 through 100 total jolts; however, between 100 and 1880 jolts, the slow jolting rate was 52 jolts/min while the fast rate was 167 jolts/min. After the hardness was measured, the mandrel and lid were replaced, and the jolting continued. Thus a total number of jolts was known for each hardness.

To study the effect of particle size distribution upon jolting, a sample of blended Vitro-roasted air-classified fines was partially sieved to give different particle size distributions. Slag was taken from these sieves to give the materials shown in Table III.

Table III

Description of Materials

Material	Taken From (U.S. Standard Mesh)
A	+ 80
B	- 80+100
C	-100+140
D	-140+170
E	-170+200
F	-200+230
G	-230+280
H	-270

The experimental data is shown in Figures 14 thru 17. Figures 14 through 16 show liner hardness as a function of the total number of jolts for the blended Vitro-roasted air-classified fines and materials "A" through "H". In most cases, above 1500 jolts, the hardness behaved erratically, and the liner cracked upon more jolting. This may be due to a small amount of grinding, since most of the sieve analyses of the materials

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[4] Green Hardness Gage – Number 473 Green Hardness Tester, manufactured by the Harry W. Dietert Co., 9338 Roselawn Ave., Detroit 4, Michigan. This device is used for obtaining definite hardness values for green molding sand and cores in foundries. This tester reads 100 for a flat metal surface.

FIGURE 14

NUMBER OF JOLTS *vs* HARDNESS FOR BLENDED VITRO ROASTED AIR CLASSIFIED FINES AND VARIOUS SCREEN FRACTIONS

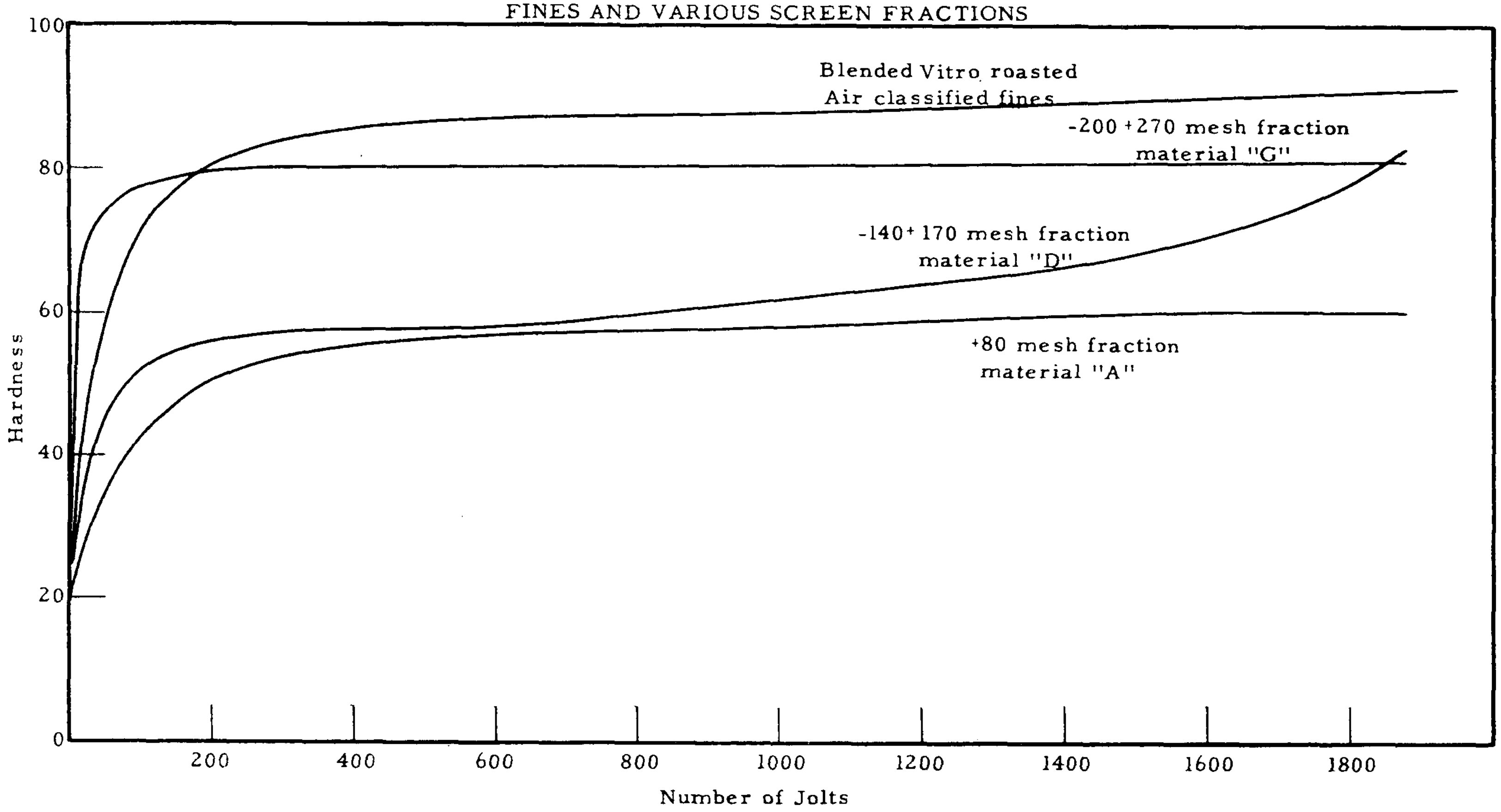


FIGURE 15

NUMBER OF JOLTS *vs* HARDNESS FOR VITRO ROASTED AIR CLASSIFIED FINES

AND VARIOUS SCREENED FRACTIONS

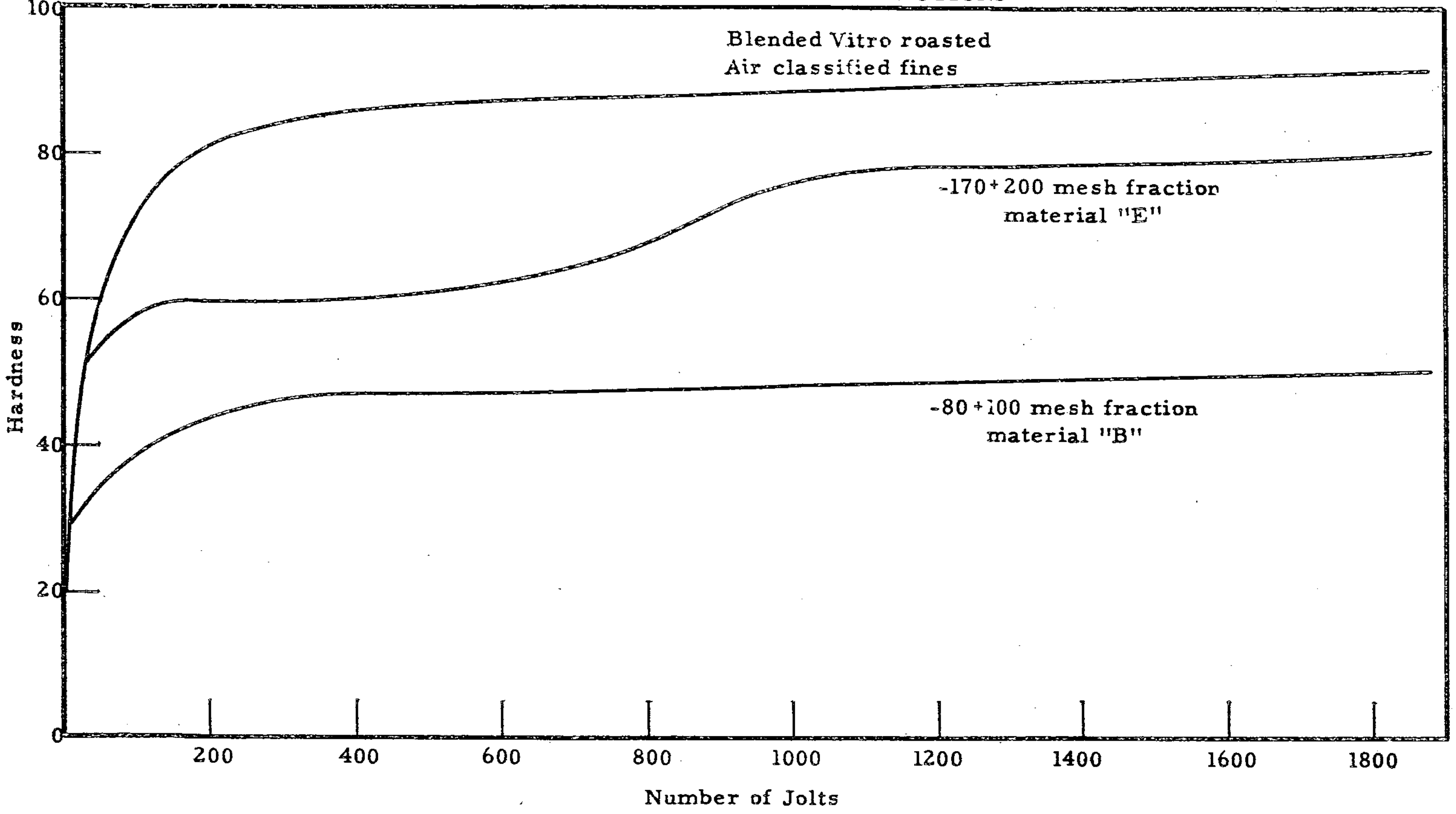


FIGURE 16

NUMBER OF JOLTS VS HARDNESS FOR BLENDED VITRO ROASTED AIR CLASSIFIED FINES AND VARIOUS SCREENED FRACTIONS

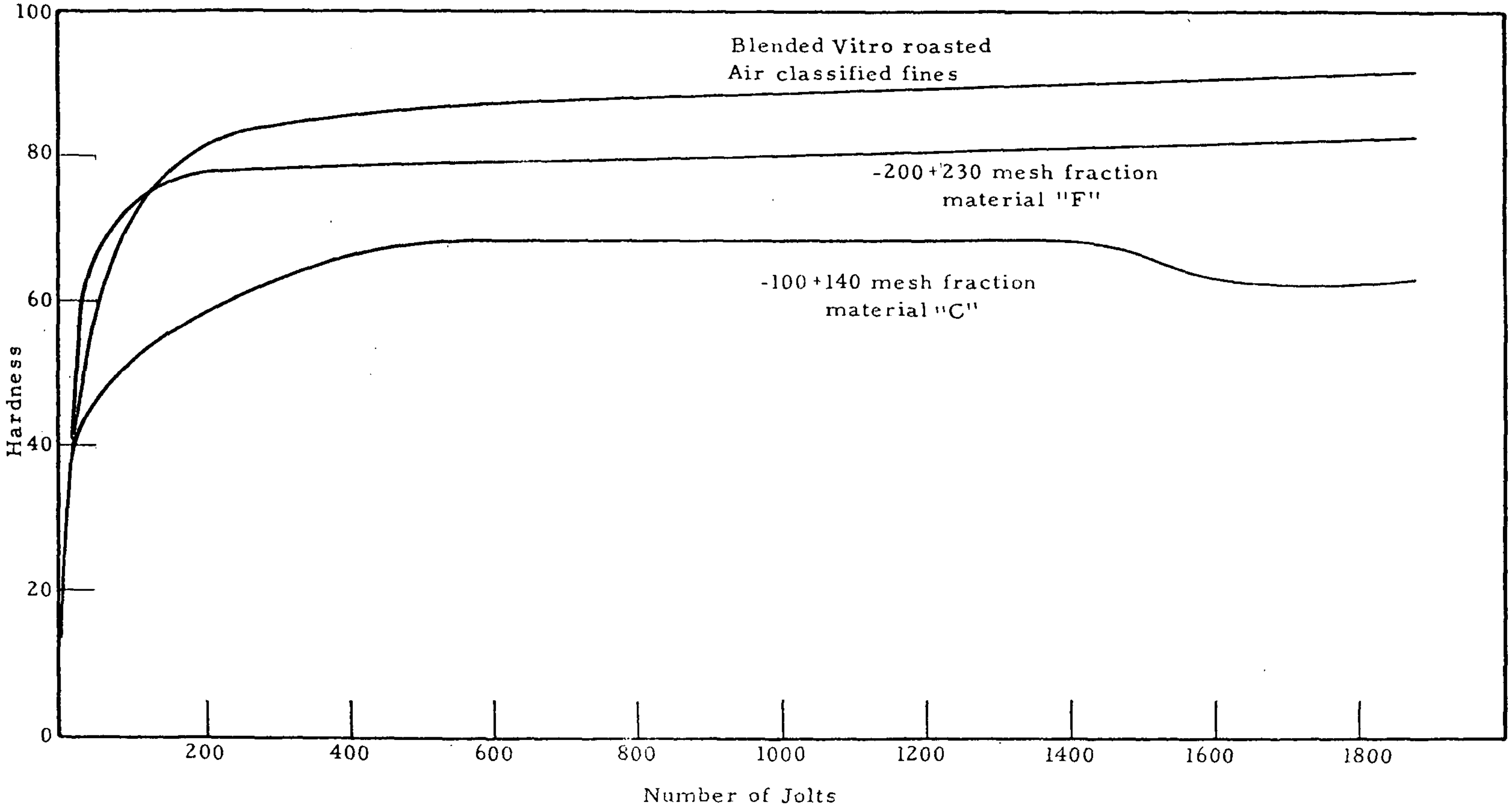
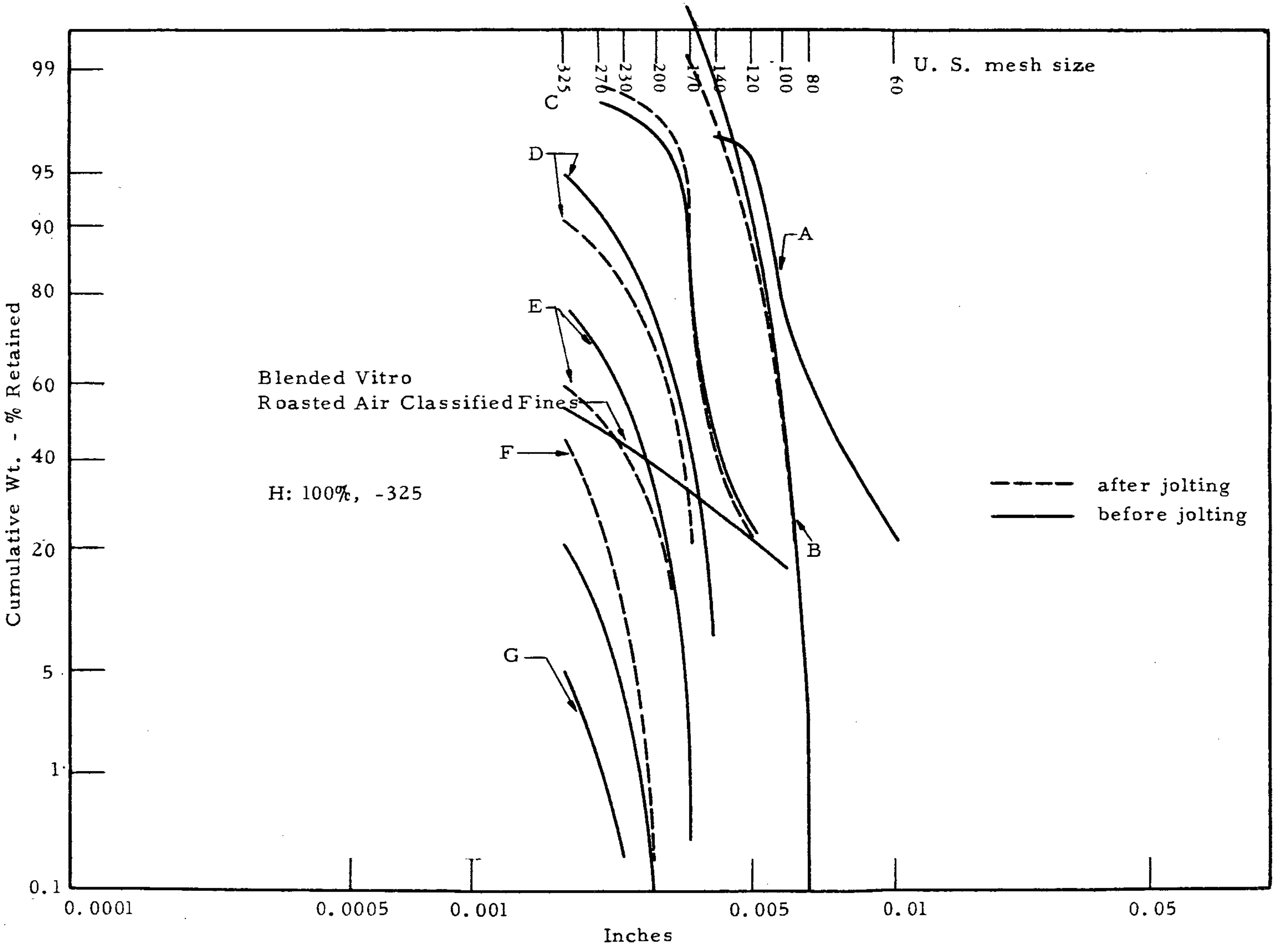


FIGURE 17

SIEVE ANALYSIS FOR BLENDED VITRO ROASTED AIR CLASSIFIED FINES AND MATERIALS A THROUGH H



before and after jolting, shown in Figure 17, indicate some decrease in particle size. Material "F", however, shows an increase in particle size. Since this material had been subjected to a permeability test, it may have absorbed some moisture.

To obtain the variation in hardness with the number of jolts on the dingot jolter, 150 lb of blended Vitro-roasted air-classified fines were placed in the bottom of a dingot shell. The mandrel was positioned, and the material jolted. After a known number of jolts, the mandrel was removed and hardness measured on the bottom and 1 inch from the bottom on the side liner. Figure 18 shows a comparison of this data and the data obtained from the experimental apparatus. For any given number of jolts, the dingot bottom liner hardness varies by no more than 2 from the experimental data. The side liner hardness, however, is consistently softer than that obtained from the experimental data. Figure 19 shows that essentially no grinding occurred for blended Vitro-roasted air-classified fines during jolting on the dingot jolter.

IV. Conclusions

In most cases, the blended Vitro-roasted air-classified fines and materials "A" through "H" reach their apparent saturation hardness after 400 jolts. Above this value, the hardness increases very slightly with increasing number of jolts. Above 100 jolts the blended material is harder, for any number of jolts, than any of materials "A" through "H".

More work will be done to find the optimum liner material with respect to liner hardness. Both MFL and dolomite will be subjected to hardness tests at various liner moisture contents.

FIGURE 18

JOLTS *vs* HARDNESS FOR BLENDED VITRO ROASTED AIR CLASSIFIED FINES

LINER MATERIAL IN A DINGOT SHELL AND IN THE EXPERIMENTAL APPARATUS

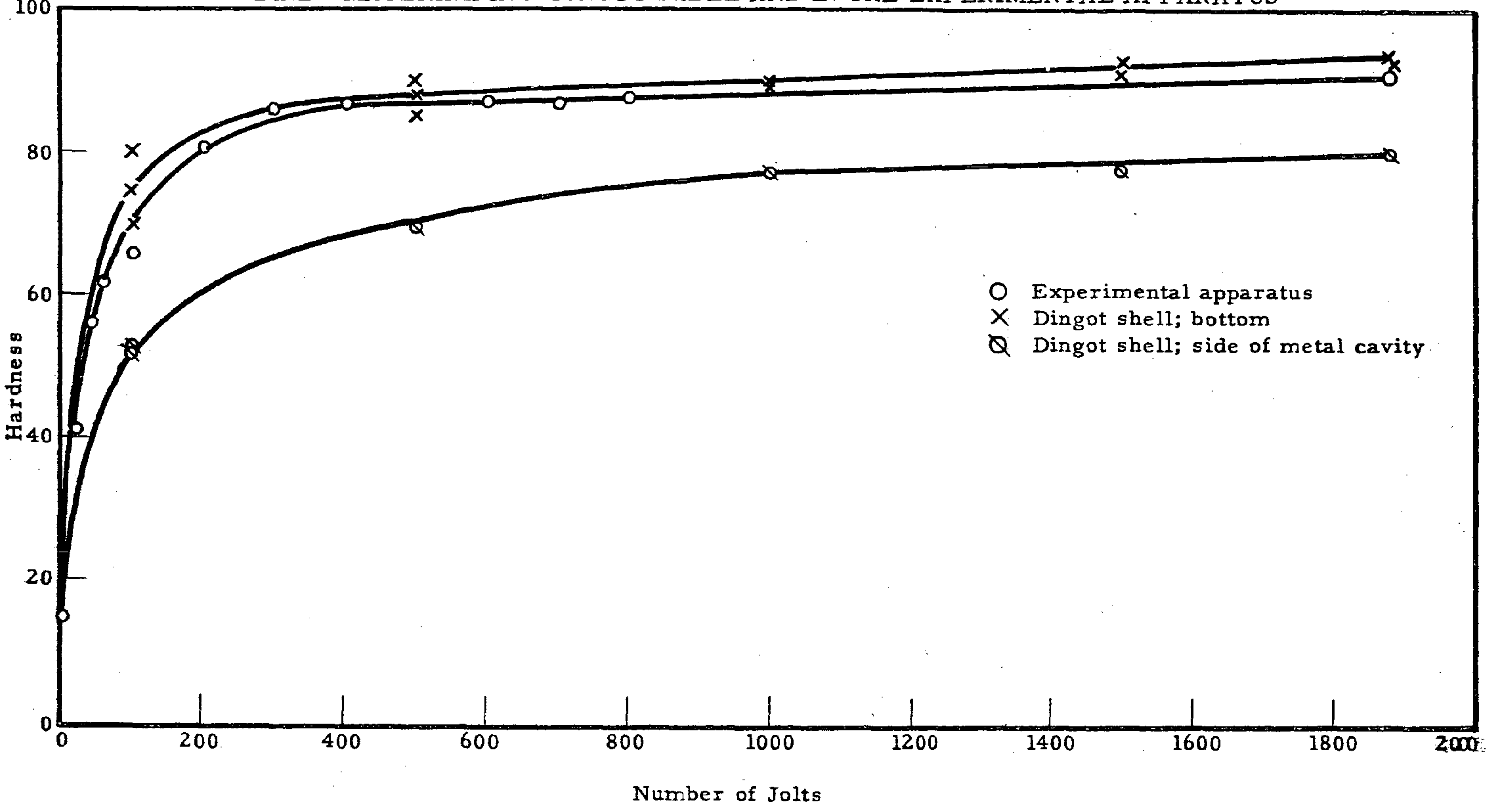
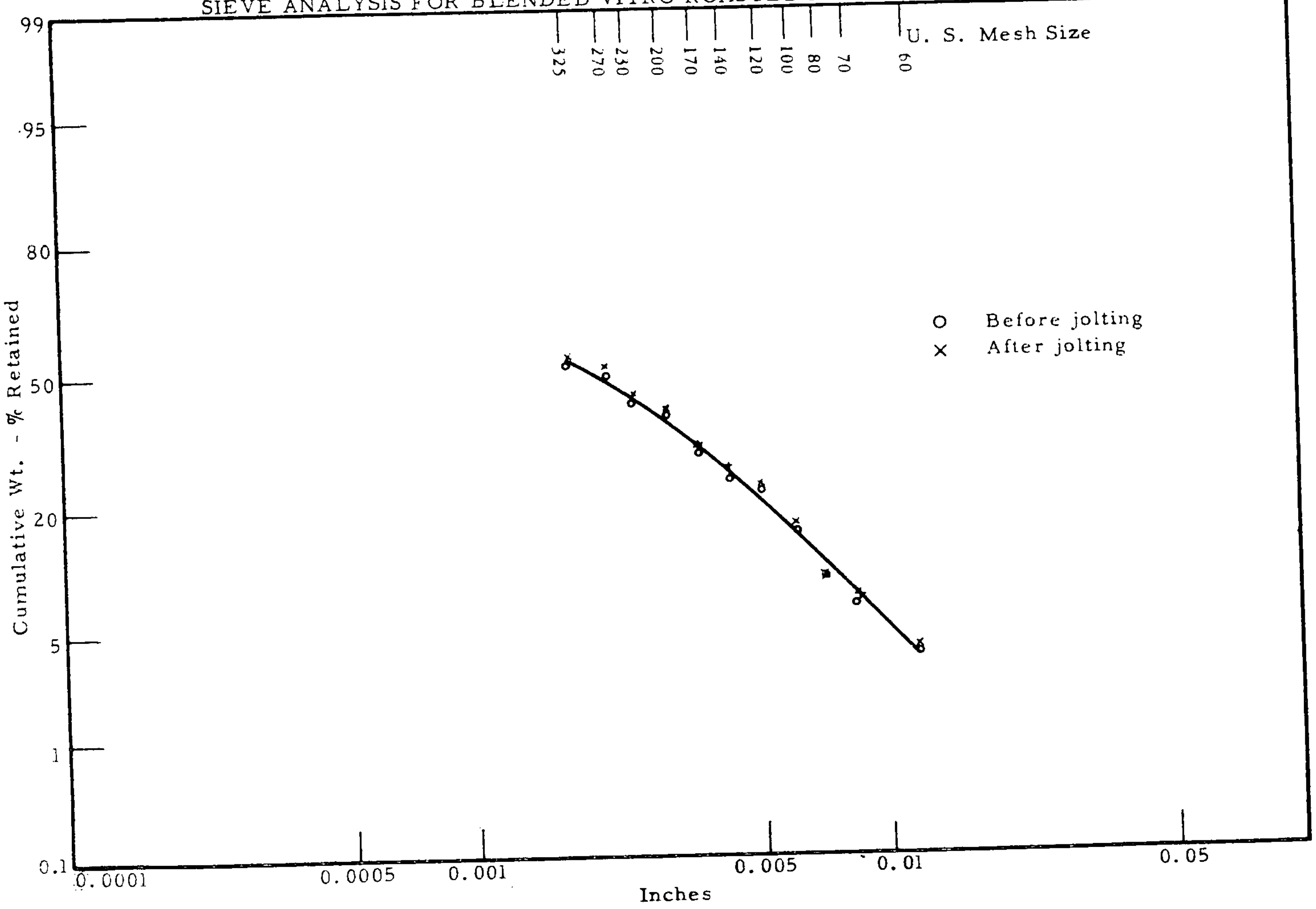


FIGURE 19

SIEVE ANALYSIS FOR BLENDED VITRO ROASTED AIR CLASSIFIED FINES



DETERMINATION OF PLANT DECONTAMINATION FACTORS FOR CERIUM (III), MAGNESIUM, AND ALUMINUM

by

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E. Monaco
H. F. Plagens

Summary

Results of a decontamination experiment in the refinery gave decontamination factors of 3×10^5 for Ce(III), 10^5 for Mg, 10^5 for Al. These results indicate that rare earths should be successfully separated from uranium when processing concentrates containing 0.2% rare earths (U_3O_8 basis) with the two cycle ether system at SLPC. Magnesium and aluminum also have high decontamination factors.

Introduction

In a previous report plant decontamination factors for thorium (IV), manganese (II), copper (II), and iron (III), were given.[1] Since the variable feed picture now indicates that SLPC will process Canadian concentrates, this work was extended to include cerium (III), a typical trivalent rare earth. Magnesium and aluminum were also studied.

Experimental

A large batch of metal dissolver feed salted with $Mg(NO_3)_3$ and Al $(NO_3)_3$ was spiked with about 2 g/l Ce(III) added as $Ce(NO_3)_3$. This feed was processed in the refinery using three columns for extraction. Previous laboratory work on a tracer scale indicated that the cerium (III) would not be oxidized under the conditions used for the run.

Results

Figure 1 is a simple flow sheet numbered to indicate sampling points. It should be noted that three extraction columns were in use at the time of this experiment rather than two

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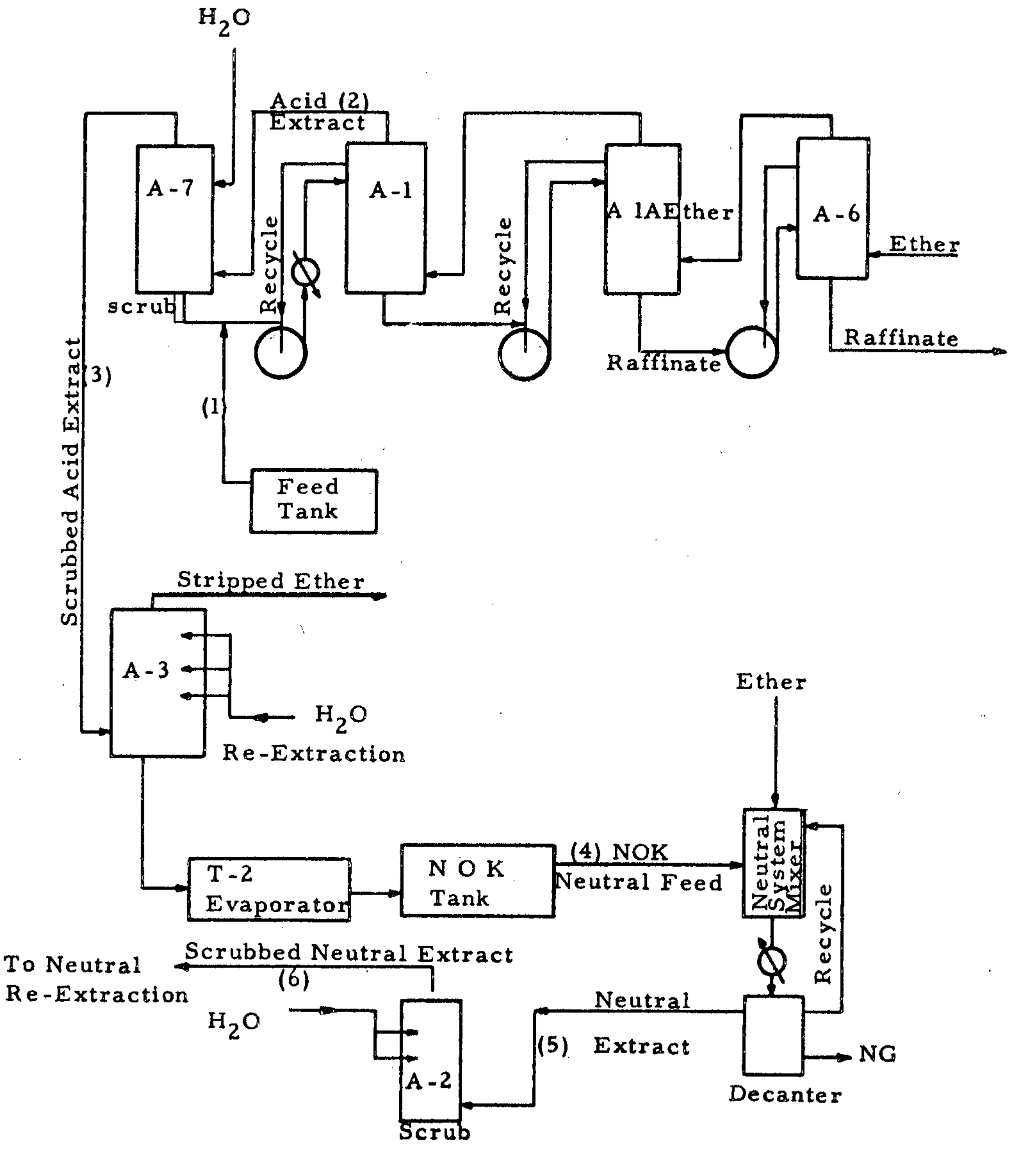
[1] Kennelley, J. A., Martin, G. L., Monaco, E., and Plagens, H. F., *Process Development Quarterly Report, Part II*, Mallinckrodt Chemical Works, *MCW-1400* (February 1, 1957) p 151

FIGURE 1

REFINERY FLOW SHEET INDICATING SAMPLING POINTS



extraction columns which were used when the decontamination factor for thorium was determined.[2]

Results are listed in Table I. The variability which usually occurs in a plant experiment is apparent in the data. In each case, however, overall decontamination was excellent. Cerium (III) is typical of the trivalent rare earths and the decontamination factor of $>10^5$ should apply to all rare earths. No difficulty will be encountered in processing concentrates containing 0.2% rare earths (U_3O_8 basis) with the ether system. The single exception to this is cerium (IV) which is so readily extracted into ether that only slight decontamination would be achieved.

Table I

Results of Plant Decontamination Experiment

Sample	Analysis[a] Ce (III) ppm	 Mg ppm	 Al ppm
1. Acid System Feed	8000	230,000	66,000
2. Acid System Extract	<0.23	14	6
3. Scrubbed Acid Extract	<0.12	1.8	0.3
4. Neutral System Feed	<9.11	71	0.35
5. Neutral Extract	<0.037	0.16	<0.079
6. Scrubbed Neutral Extract	0.026	1.8	0.26
Overall Decontamination Factor	$>3 \times 10^5$	10^5	10^5

[a] Analysis on a uranium basis

The decontamination factors reported from this and the previous experiment are somewhat less than would be predicted from laboratory distribution coefficients. These results indicate that the maximum decontamination which can be achieved in the plant is in the range of 10^5 to 10^6. Physical factors such as entrainment and carry over of solids probably limit decontamination to this range.

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[2] *Ibid*

Glossary of Specialized Terms

AOI	- ammonium oxalate insoluble
billet	- a bar of forged dingot uranium suitable for subsequent rolling
black oxide	- U_3O_8
brown oxide	- UO_2
derby	- the uranium metal product of the nominal 300, 100 and 50 lb. reduction bombs which is subsequently recast.
dingot	- (direct ingot) the uranium metal product of 1400 and 3300 lb. reduction bombs. This metal not recast
DMFL	- dingot magnesium fluoride liner
ESU	- easily soluble uranium
green salt	- UF_4
I & E slug	- (hollow) internally and externally cooled slug
ingot	- recast uranium metal
MFL	- magnesium fluoride liner
NOK	- a uranyl nitrate liquor which is fed to the neutral ether system for final purification
OK-liquor	- the uranyl nitrate liquor of highest purity used as a feed to the denitration pots
orange oxide	- UO_3
P - D	- pumper decanter

(continued on next page)

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Glossary continued

RMF	- reject magnesium fluoride
rod	- cylindrical length of uranium produced by rolling or extruding uranium billets and ingots
shotgun	- a percent increase of neutron absorption cross section due to impurities in the product; pure U_3O_8 has a shotgun of zero
slag	- magnesium fluoride, containing small quantities of uranium and magnesium formed in the thermite bomb reaction
slug	- rods of uranium machined to specific diameter and lengths. Slugs, when canned are used as fuel elements
soda-salt	- sodium diuranate; usually applied to raw materials of that composition
UNH	- uranyl nitrate hexahydrate
U-Con	- a product of the physical separations plant consisting of a mixture of fine metal particles, uranium oxides, and magnesium fluorides, screened to -10 mesh and assaying 70 - 75% uranium
U-Mag	- an intermediate product of the physical separations plant consisting of the +10 mesh oversize from the screening of ground RMF. It is further separated to 701-metal and C-701
701-metal	- the metal concentrate product of the physical separations plant consisting of uranium metal particles ranging in size from +10 mesh to ½ inch and assaying 95% uranium
C-701	- the reject product of the physical separations plant consisting chiefly of finely ground magnesium floride

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