LABORATORY AND PILOT PLANT EVALUATION
OF
DYNO URANIUM CONCENTRATE

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AEC RESEARCH AND DEVELOPMENT REPORT

FEED MATERIALS PRODUCTION CENTER
NATIONAL LEAD COMPANY OF OHIO

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TECHNICAL DIVISION
NATIONAL LEAD COMPANY OF OHIO

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ABSTRACT

The laboratory and pilot plant evaluation of Dyno concentrate is presented in this report. This concentrate meets all FMPC impurity tolerance limits except thorium and chloride limits. The thorium limit can be met by the addition of phosphate as required for thorium-containing uranium concentrates. The high chloride content of this material will necessitate blending of the material with a good quality low-chloride-containing concentrate in the National Lead Company of Ohio refinery process. Laboratory digestion and extraction tests indicate that this material should present no other problems in pulse column processing. A pilot plant pulse column test indicated that this material should present no operational problems in the National Lead Company of Ohio Refinery.

INTRODUCTION

The Canadian Dyno Mines, Limited,¹ started to produce a uranium concentrate in May, 1958. The mine and mill are located in the Bancroft area, which is approximately 160 miles from Toronto, Ontario, in Canada. The concentrate is a magnesium precipitate of an ion-exchange liquor. The company expects to process 1100 to 1200 tons of ore daily when maximum production is reached.

The National Lead Company of Ohio has started to receive significant quantities of Dyno concentrate to be processed in its Refinery. Thus, a complete evaluation of the concentrate is necessary to determine (1) possible refinery processing difficulties, and (2) impurity concentrations in comparison with FMPC impurity tolerance limits (Appendix I).

Dyno concentrate is to be processed by standard Refinery procedures, which are as follows:

The uranium concentrate feed materials are digested in a nitric acid solution, producing a slurry. The uranium in the feed slurry is separated and purified by a process wherein the uranium is extracted, scrubbed, and re-extracted in pulse-type perforated-plate columns. The uranium first is extracted by a tributyl phosphate-kerosene solvent, leaving a uranium-depleted raffinate. Then the uranium-containing solvent (extract) is scrubbed to remove impurities. Finally, the uranium is re-extracted by water. The final product of the Refinery process is thus an aqueous solution of uranyl nitrate hexahydrate (UNH).

CONCLUSIONS

Dyno concentrate will require blending with a good quality low-chloride-containing concentrate to lower the chloride content of the feed slurry to an acceptable level. It can be further concluded from this feed material evaluation that Dyno concentrate should present no other refinery processing problems, if handled as a thorium-containing uranium concentrate.^{2,3} These conclusions are based on the assumption that the material investigated is indicative of future receipts.

LABORATORY AND PILOT PLANT TESTS

The material used in the following tests was the official assayer's sample from Dyno concentrate, Lot DM-1.

CHEMICAL ANALYSES

Samples of Dyno concentrate were analyzed by the National Lead Company of Ohio Analytical Department for comparison of impurity concentrations with FMPC impurity tolerance limits (Appendix I). The wet chemical analyses of the concentrate are presented in Table I; the spectrochemical analyses are given in Table II; and the rare earth analyses are listed in Table III.

TABLE I Wet Chemical Analyses of Dyno Concentrate
(Results are in weight per cent on a uranium basis)

Constituent	Results (%)	Constituent	Results (%)
Uranium (U)	62.11*	Iron (Fe)	1.87
Ammonia (NH ₃)	0.005	Phosphate (PO ₄ ⁻³)	0.24
Calcium (Ca)	0.92	Sodium (Na)	4.03
Carbonate (CO ₃ ⁻²)	0.43	Sulfate (SO ₄ ⁻²)	<0.16
Chloride (Cl ⁻)	0.144	Thorium (Th)	0.84
Fluoride (F ⁻)	<0.05	Total Organic	0.003

* Per cent uranium, on an as-received basis.

TABLE II General Spectrochemical Analyses of Dyno Concentrate
(Results are in parts per million on a uranium basis)

Constituent	Results (ppm)	Constituent	Results (ppm)
Aluminum (Al)	<640	Magnesium (Mg)	40,000
Arsenic (As)	<160	Manganese (Mn)	190
Bismuth (Bi)	<32	Molybdenum (Mo)	<16
Boron (B)	130	Nickel (Ni)	<32
Cadmium (Cd)	<320	Silicon Dioxide (SiO ₂)	2,900
Chromium (Cr)	<160	Tin (Sn)	<16
Cobalt (Co)	<160	Vanadium (V)	<160
Copper (Cu)	<160	Zinc (Zn)	<320
Lead (Pb)	80		

NOTE: The accuracy of these results is estimated to be $\pm 50\%$.

TABLE III Spectrochemical Rare Earth Analyses of Dyno Concentrate
(Results are in parts per million on a uranium basis)

Constituent	Results (ppm)	Constituent	Results (ppm)
Dysprosium (Dy)	<6	Samarium (Sm)	<6
Erbium (Er)	<6	Terbium (Tb)	<6
Europium (Eu)	<6	Thulium (Tm)	<6
Gadolinium (Gd)	<6	Yttrium (Y)	34
Holmium (Ho)	<6	Ytterbium (Yb)	<6
Lutetium (Lu)	<6		

NOTE: The accuracy of these results is estimated to be $\pm 30\%$.

TABLE IV Digestion of Dyno Concentrate

Digestion Characteristics	Results
Apparent Assay (Wt % U)	60.6
Acid Consumption*	0.103
Volume Change Factor**	0.990
Reactivity of HNO ₃ Dissolution	Reaction proceeded with mild fuming and foaming and a slight temperature rise.
Slurry Characteristics	
Solids (% by wt)	1.57
Digestion Insolubles (% by wt of insolubles in concentrate)	6.48
Digestion Efficiency***	>99.99

* Acid consumption is reported in gram equivalents of HNO₃ (consumed in digesting to 200 g U/l and 0.0N free acid) per gram of concentrate.

** Volume Change Factor = Initial Volume of Dilute Acid/Final Volume of Slurry

*** Efficiency (%) = (Soluble U/Total U) $\times$ 100

LABORATORY DIGESTION TESTS

Laboratory-scale digestions⁴ were run on Dyno concentrate, producing a slurry which met Refinery specifications in regard to uranium and nitric acid concentrations. Observed and calculated results are listed in Table IV.

LABORATORY EXTRACTION TESTS

Laboratory extraction tests⁵ were made on slurries prepared from Dyno concentrate, to determine the uranium distribution curve for the material in a 33.5 per cent TBP-66.5 per cent kerosene system. The uranium distribution curve for Dyno concentrate is compared with that for pure UNH in the same system in Figure 1.

FIGURE 1 Uranium Distribution Curve for Dyno Concentrate and UNH. The normality of Aqueous in each case was $3.0N \pm 0.2N$. Room Temperature ($25^{\circ}C \pm 2^{\circ}C$). The organic phase was 33.1 Per Cent TBP in kerosene.

PILOT PLANT TWO-INCH-COLUMN EXTRACTION TESTS

A pilot plant two-inch column extraction test⁶ was conducted, utilizing a feed slurry prepared from Dyno concentrate, Lot DM-1. The high thorium content of this concentrate necessitated the addition of phosphate to the feed slurry for thorium complexing;^{2,3} therefore, the test was designed to determine extraction column performance with a feed slurry containing ten per cent phosphate on a uranium basis. Test conditions and resulting stream analyses are listed in Tables V through VIII. The material balance calculations are given in Appendix II.

TABLE V Pilot Plant Two-Inch Column Test Conditions
Dyno Concentrate - Test No. 17

Feed Material

Aqueous Feed (AF) Dyno Concentrate Slurry (Lot DM-1); 200 ± 10 g/l U; $3.0 \pm 0.2N$ HNO_3 ; 100° to $110^\circ F$.

Organic Extractant (AX) 33.5% TBP in kerosene; nonacidified; ambient temperature.

Scrub Water (BS) Deionized water, ambient temperature.

Test Flowratios*

AX/AF = 1.96/1; AX/BS = 6.21/1

Test Flowrates*

AF = 6.19 gph	AR = 7.70 gph	BS = 1.95 gph	BP = 12.67 gph
AX = 12.10 gph	AP = 12.80 gph	BR = 2.07 gph	

Test Column Throughput*

A-Column = 996 gsfh; B-Column = 708 gsfh.

A-Column Flooding Throughput*

>1500 gsfh.

Continuous Phase

A-Column - Organic; B-Column - Aqueous

Pulse Conditions

A-Column - Frequency = 65 cpm;	Amplitude = 0.75 inch
B-Column - Frequency = 36 cpm;	Amplitude = 0.60 inch

Length of Test

13½ hours

* Flowratios were held constant throughout the test. The flowrates and column throughputs listed are those which prevailed during sampling. A-column flooding studies were conducted after steady-state sampling had been completed.

TABLE VI Pilot Plant Two-Inch-Column Stream Analyses;
Dyno Concentrate – Test No. 17

	Hour of Test		
	11	11½	12
<u>A - Column Streams (996 gsfh)</u>			
<u>Aqueous Feed (AF)</u>			
U (g/l)	200	200	200
HNO ₃ (N)	3.3	3.1	3.2
Th (g/l)	2.18	2.18	2.20
PO ₄ (g/l)	18.6	19.1	19.1
<u>Organic Feed (AX)</u>			
U (g/l)	3.5	2.7	2.7
HNO ₃ (N)	0.0	0.0	0.0
TBP (%)	34.4	34.5	34.4
<u>Organic Product (AP)</u>			
Density and Temperature	0.994 at 87°F	0.994 at 85°F	0.994 at 85°F
U (g/l)	116*	113	113
HNO ₃ (N)	0.21	0.21	0.22
Th (g/l)	0.65	0.66	0.69
<u>Aqueous Raffinate (AR)</u>			
U (g/l)	0.14	0.15	0.14
HNO ₃ (N)	2.4	2.4	2.4
Th** (g/l)	1.35	1.33	1.23
<u>B - Column Streams (708 gsfh)</u>			
<u>Aqueous Recycle (BR)</u>			
U (g/l)	117	115	116
HNO ₃ (N)	1.4	1.3	1.3
Th (g/l)	0.255	0.249	0.255
<u>Scrubbed Organic Product (BP)</u>			
Density and Temperature	0.966 at 83°F	0.967 at 82°F	0.967 at 81°F
U (g/l)	96.5	97.6	98.0
HNO ₃ (N)	0.012*	0.023	0.021
Th (g/l)	0.0044	0.0032	0.0043

* Not used in material balance calculations (Appendix II).

** Very poor thorium balances resulted from this test, and a discussion of this problem is presented in Reference 6.

TABLE VII Spectrochemical Analyses of Pilot Plant Two-Inch-Column
Scrubbed Organic Product (BP)*; Dyno Concentrate – Test No. 17
(All values are ppm, U basis)

Element	Hour of Test		
	11	11½	12
Al	<6.0	<6.0	<6.0
Ba	<1.0	<1.0	<1.0
Bi	<1.0	<1.0	<1.0
Ca	1.0	<1.0	<1.0
Cd	<0.2	<0.2	<0.2
Cr	<10.0	<10.0	<10.0
Cu	<1.0	<1.0	<1.0
Fe	<10.0	<10.0	<10.0
Li	<3.0	<3.0	<3.0
Mg	6.0	10.0	4.0
Mn	<4.0	<4.0	<4.0
Mo	<6.0	<6.0	<6.0
Na	<1.0	<1.0	<1.0
Ni	<10.0	<10.0	<10.0
P	20.0	30.0	20.0
Pb	3.0	1.0	<1.0
SiO ₂	40.0	40.0	40.0
Sn	<1.0	<1.0	<1.0
Sr	<6.0	<6.0	<6.0
V	<40.0	<40.0	<40.0
Zn	<20.0	<20.0	<20.0

NOTE: The accuracy of these results is estimated to be $\pm 50\%$.

*The contamination level of the scrubbed organic product (BP) will normally be equivalent to the contamination level of a final aqueous uranyl nitrate product (CP) produced by stripping the scrubbed organic with hot deionized water.

TABLE VIII Pilot Plant Two-Inch-Column Organic Stream Analyses;
Dyno Concentrate – Test No. 17

Stream	Hour of Test		
	11	11½	12
<u>Organic Feed (AX)</u>			
TBP (%)	34.4	34.5	34.4
Coalescence Time* (sec) ⁷	150	—	—
Solids (%)	0.01	0.01	—
Aqueous (%)	<0.01	<0.01	—
<u>Organic Product (AP)</u>			
Solids (%)	0.01	0.01	0.01
Aqueous (%)	0.03	0.03	0.03
<u>Scrubbed Organic Product (BP)</u>			
Solids (%)	0.01	0.01	0.01
Aqueous (%)	0.01	0.07	0.09

NOTE: All percentages are volume per cent.

* A discussion of the effect of solvent condition upon A-column flooding capacity is presented in Reference 6.

DISCUSSION

Chemical analyses of Dyno concentrate reveal that the material meets all FMPC impurity tolerance limits except those for chloride and thorium (Appendix I). The chloride concentration is 0.144 per cent (U basis), compared with the FMPC tolerance limit of 0.125 per cent. Thus, the concentrate will have to be blended approximately one to two with a low-chloride-containing uranium concentrate to meet the chloride tolerance limit. The thorium concentration in this material is 8400 ppm, compared with the primary FMPC tolerance limit of 350 ppm. However, when treated as a thorium-containing uranium concentrate,² the secondary tolerance limit (20,000 ppm) is met.

Digestion of Dyno concentrate to give a satisfactory feed slurry for Refinery use should present no problems. The reactivity of HNO_3 dissolution parallels that of the feed materials presently used in the Refinery. The resulting slurry is moderately low in nitric acid insolubles and should present no difficulties in slurry handling.

The uranium distribution curve for Dyno concentrate in a 33.5 per cent TBP-66.5 per cent kerosene system compares favorably with that for pure UNH in the same system. It could be expected, then, that the material would perform favorably in pulse column operation. No emulsification tendencies were noted during the laboratory extraction tests.

The primary extraction column performance of a feed slurry prepared from Dyno concentrate (with an addition of 10 per cent PO_4 on a uranium basis for thorium complexing) was satisfactory. A higher-than-normal aqueous content of the scrubbed organic product (BP) was detected during the test, and may have been caused by an emulsion prevalent in intermediate sections of the scrub column. Despite this emulsification and aqueous content of the scrubbed organic product, over-all scrub column operation was satisfactory. The contamination levels of the scrubbed organic product (BP) were well within the desired limits. The A-column flooding capacity exceeded 1500 gsfh for this concentrate.

ACKNOWLEDGMENT

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APPENDIX I - IMPURITY TOLERANCE LIMITS FOR FMPC FEED MATERIALS

NOTE: These are values for operable limits based on available data. A Feed Materials Evaluation Program is being conducted on all new feeds received at this site. The data obtained, with those developed in various Refinery process development projects, will be used to enlarge and improve this table. In addition to data on operable limits, data are being developed for definition of limits on the basis of economic factors.

Constituent	Tolerance Limits	Effect of Deviation Beyond Limits
1. Uranium	30% (min) of feed materials (as received)	Reduces plant capacity
2. Sulfate	30% (max) on U basis	Reduces extractability of U from feed slurry
3. Calcium x Sulfate	$\%Ca \times \%SO_4 < 40$ on U basis	Causes column scaling
4. Phosphate (see Item 16)	7% (max) on U basis	Reduces extractability of U from feed slurry
5. Phosphate plus Sulfate	$5(\%PO_4) + \%SO_4 < 35$ on U basis	Reduces extractability of U from feed slurry
6. Chlorides	0.125% on U basis	Corrosion
7. Fluorides	0.125% on U basis	Corrosion
8. Iron	40% (max) on U basis	Reduces product purity
9. Vanadium	5% (max) on U basis	Reduces product purity
10. Boron	0.7% (max) on U basis	Reduces product purity
11. Copper	4% (max) on U basis	Reduces product purity
12. Organic Material	0.12% (max) on U basis	Possible explosion hazard with HNO_3 and reduced extractability due to addition of surfactants
13. Arsenic	Unknown (feed containing 7% As on U basis has been processed satisfactorily)	Reduces product purity
14. Molybdenum	Unknown (feed containing 1.5% Mo on a U basis has been processed satisfactorily)	Reduces product purity
15. Thorium, with no Phosphate	350 ppm (max) on U basis	Reduces product purity
16. Thorium, plus Phosphate	20,000 ppm (max) on U basis with 10% PO_4 added on U basis	Reduces product purity
17. Rare Earths, Cadmium, Lead, Zinc, Chromium, Zirconium and Titanium	Data on which to base limiting conc. of these elements in feed materials are not yet available. Tolerance limits on Gd, Dy and Zr are being developed.	Reduces product purity

APPENDIX II MATERIAL BALANCE CALCULATIONS; DYNO CONCENTRATE - TEST NO. 17

A-COLUMN MATERIAL BALANCES*

In: (AF flowrate $\times$ AF concentration) + (AX flowrate $\times$ AX concentration) + (BR flowrate $\times$ BR concentration)

Out: (AP flowrate $\times$ AP concentration) + (AR flowrate $\times$ AR concentration)

Uranium Balance

$$\begin{aligned}
 &\text{In} && \text{Out} \\
 &(6.19 \times 200) + (12.10 \times 2.96) + (2.07 \times 116) = (12.80 \times 113) + (7.70 \times 0.143) \\
 &1513.9 = 1447.5 \\
 &\text{Per Cent Error} = \frac{1447.5 - 1513.9}{1513.9} \times 100 \\
 &= -4.39\%.
 \end{aligned}$$

HNO₃ Balance

$$\begin{aligned}
 &\text{In} && \text{Out} \\
 &(6.19 \times 3.2) + (12.10 \times 0) + (2.07 \times 1.33) = 12.80 \times 0.212 + (7.70 \times 2.4) \\
 &22.56 = 21.19 \\
 &\text{Per Cent Error} = \frac{21.19 - 22.56}{22.56} \times 100 \\
 &= -6.07\%.
 \end{aligned}$$

Thorium Balance

$$\begin{aligned}
 &\text{In} && \text{Out} \\
 &(6.19 \times 2.19) + (12.10 \times 0) + (2.07 \times 0.253) = (12.80 \times 0.0663) + (7.70 \times 1.303) \\
 &14.08 = 10.88 \\
 &\text{Per Cent Error} = \frac{10.88 - 14.08}{14.08} \times 100 \\
 &= -22.73\%.
 \end{aligned}$$

B-COLUMN MATERIAL BALANCES*

In: (AP flowrate $\times$ AP concentration) + (BS flowrate $\times$ BS concentration)

Out: (BR flowrate $\times$ BR concentration) + (BP flowrate $\times$ BP concentration)

Uranium Balance

$$\begin{aligned} & \begin{array}{cc} \text{In} & \text{Out} \end{array} \\ & (12.80 \times 113) + (1.95 \times 0) = (2.07 \times 116) + (12.67 \times 97.33) \\ & 1446.4 = 1473.3 \\ & \text{Per Cent Error} = \frac{1473.3 - 1446.4}{1473.3} \times 100 \\ & = +1.83\%. \end{aligned}$$

HNO₃ Balance

$$\begin{aligned} & \begin{array}{cc} \text{In} & \text{Out} \end{array} \\ & (12.80 \times 0.212) + (1.95 \times 0) = (2.07 \times 1.33) + (12.67 \times 0.022) \\ & 2.71 = 3.03 \\ & \text{Per Cent Error} = \frac{3.03 - 2.71}{3.03} \times 100 \\ & = +11.22\%. \end{aligned}$$

Thorium Balance

$$\begin{aligned} & \begin{array}{cc} \text{In} & \text{Out} \end{array} \\ & (12.80 \times 0.0663) + (1.95 \times 0) = (2.07 \times 0.253) + (12.67 \times 0.00396) \\ & 0.85 = 0.57 \\ & \text{Per Cent Error} = \frac{0.57 - 0.85}{0.85} \times 100 \\ & = -32.94\%. \end{aligned}$$

SYSTEM MATERIAL BALANCES*

In: (AF flowrate $\times$ AF concentration) + (AX flowrate $\times$ AX concentration) + (BS flowrate $\times$ BS concentration)

Out: (AR flowrate $\times$ AR concentration) + (BP flowrate $\times$ BP concentration)

Uranium Balance

$$\begin{aligned}
 &\text{In} && \text{Out} \\
 &(6.19 \times 200) + (12.10 \times 2.96) + (1.95 \times 0) = (7.70 \times 0.143) + (12.67 \times 97.33) \\
 &1273.8 = 1234.3 \\
 &\text{Per Cent Error} = \frac{1234.3 - 1273.8}{1273.8} \times 100 \\
 &= -3.10\%.
 \end{aligned}$$

HNO₃ Balance

$$\begin{aligned}
 &\text{In} && \text{Out} \\
 &(6.19 \times 3.2) + (12.10 \times 0) + (1.95 \times 0) = (7.70 \times 1.303) + (12.67 \times 0.00396) \\
 &19.81 = 18.76 \\
 &\text{Per Cent Error} = \frac{18.76 - 19.81}{19.81} \times 100 \\
 &= -5.30\%.
 \end{aligned}$$

Thorium Balance

$$\begin{aligned}
 &\text{In} && \text{Out} \\
 &(6.19 \times 2.19) + (12.10 \times 0) + (1.95 \times 0) = (7.70 \times 1.303) + (12.67 \times 0.00396) \\
 &13.56 = 10.08 \\
 &\text{Per Cent Error} = \frac{10.08 - 13.56}{13.56} \times 100 \\
 &= -25.66\%.
 \end{aligned}$$

**NOTE: All flowrates are given in gph. The units of concentrations used in these calculations are: Uranium: (g/l); Nitric Acid: (N); Thorium: (g/l)*