MASTER

LABORATORY AND PILOT PLANT EVALUATION
OF
STANLEIGH URANIUM CONCENTRATE

By

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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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CONTENTS

	Page No.
ABSTRACT	5
INTRODUCTION	7
CONCLUSIONS	7
LABORATORY AND PILOT PLANT TESTS	9
Chemical Analyses	9
Laboratory Digestion Tests	10
Laboratory Extraction Tests	10
Pilot Plant Two-Inch-Column Extraction Test	11
DISCUSSION	17
ACKNOWLEDGMENT	19
REFERENCES	19
APPENDIX I – Impurity Tolerance Limits for FMPC Feed Materials	21
APPENDIX II – Material Balance Calculations for Stanleigh Concentrate, Test No. 11	23

ABSTRACT

The laboratory and pilot plant evaluation of Stanleigh concentrate is presented in this report. This concentrate meets all FMPC impurity tolerance limits except those for thorium and chloride. The thorium limit can be met by the addition of phosphate as required for thorium-containing uranium concentrates or by blending with low thorium concentrates. The high chloride content of this material will necessitate the blending of the material with a good quality low-chloride-content 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 FMPC Refinery.

INTRODUCTION

The Stanleigh Uranium Mining Corporation¹ started producing uranium concentrate in March of 1958. The mine and mill are located in the Blind River area, north of Lake Huron in Ontario, Canada. The concentrate is a sodium precipitate of an ion exchange liquor. The present mill capacity is rated at 3000 ore tons per day.

The National Lead Company of Ohio has begun to receive significant quantities of Stanleigh concentrate to be processed in its Refinery. Thus, a complete evaluation of the concentrate is necessary to determine possible refinery processing difficulties and to compare impurity concentrations with FMPC impurity tolerance limits.

The Stanleigh 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

Stanleigh 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 Stanleigh 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 Stanleigh concentrate, Lot S-1.

CHEMICAL ANALYSES

Samples of Stanleigh 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 given in Table I; the spectrochemical analyses are presented in Table II; and the rare earth analyses are listed in Table III.

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

Constituent	Results (%)	Constituent	Results (%)
Uranium (U)	60.11*	Iron (Fe)	2.58
Ammonia (NH ₃)	0.013	Phosphate (PO ₄ ⁻³)	0.20
Calcium (Ca)	0.38	Sodium (Na)	9.97
Carbonate (CO ₃ ⁻²)	0.15	Sulfate (SO ₄ ⁻²)	4.21
Chloride (Cl ⁻)	0.17	Thorium (Th)	0.50
Fluoride (F ⁻)	<0.05	Total Organic	0.01

* Per cent uranium, as-received basis.

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

Constituent	Results (ppm)	Constituent	Results (ppm)
Aluminum (Al)	660	Magnesium (Mg)	580
Arsenic (As)	<170	Manganese (Mn)	83
Bismuth (Bi)	66	Molybdenum (Mo)	<17
Boron (B)	13	Nickel (Ni)	<33
Cadmium (Cd)	<330	Phosphorus (P)	<660
Chromium (Cr)	<170	Silicon Dioxide (SiO ₂)	<660
Cobalt (Co)	<170	Tin (Sn)	<17
Copper (Cu)	<170	Vanadium (V)	<170
Iron (Fe)	>1,700	Zinc (Zn)	<330

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

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

Constituent	Results (ppm)	Constituent	Results (ppm)
Dysprosium (Dy)	52	Samarium (Sm)	58
Erbium (Er)	22	Terbium (Tb)	15
Europium (Eu)	<7	Thulium (Tm)	<7
Gadolinium (Gd)	37	Yttrium (Y)	17
Holmium (Ho)	11.3	Ytterbium (Yb)	270

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

TABLE IV Laboratory -Scale Digestion of Stanleigh Concentrate

Digestion Characteristics

Apparent Assay (wt % U)	61.5
Acid Consumption*	0.0075
Volume Change Factor**	0.980
Reactivity of HNO_3 Dissolution	The reaction proceeded with very mild fuming and a slight temperature rise.

Slurry Characteristics

Solids (% by wt)	<0.1
Digestion Insolubles (% by wt of insolubles in concentrate)	<0.1
Digestion Efficiency***	>99.99

* Acid consumption is reported in gram equivalents of HNO_3 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 per Final Volume of Slurry.

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

LABORATORY DIGESTION TESTS

Laboratory-scale digestions⁴ were conducted with Stanleigh concentrate to produce a slurry which met refinery specifications in regard to uranium and nitric acid concentrations. The results are listed in Table IV.

LABORATORY EXTRACTION TESTS

Extraction tests were made with feed slurries prepared from Stanleigh concentrate to determine the uranium distribution curve⁵ of the material in a 33.5 per cent TBP-66.5 per cent kerosene system. The uranium distribution curve for Stanleigh concentrate is compared with the uranium distribution curve for pure uranyl nitrate hexahydrate (UNH) in Figure 1.

FIGURE 1 Uranium Distribution Curve for Stanleigh Concentrate

PILOT PLANT TWO-INCH-COLUMN EXTRACTION TEST

A pilot plant two-inch-column extraction test⁶ was conducted, utilizing a feed slurry prepared from Stanleigh concentrate, Lot S-1. Because of the high thorium content of this concentrate phosphate was added to the feed slurry for thorium complexing.^{2,3} Therefore, the test was designed to determine extraction column performance with a feed slurry containing 10 per cent phosphate on a uranium basis. Test conditions and resulting stream analyses are listed in Tables V through VIII.

TABLE V Pilot Plant Two-Inch-Column Test Conditions,
Stanleigh Concentrate; Test No. 11

Feed Material

Aqueous Feed (AF) — Stanleigh concentrate slurry (Lot S-1); 200 ± 10 g/l U; $3.0 \pm 0.2N$ HNO_3 ; $100^\circ - 110^\circ F$

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

Scrub Water (BS) — Deionized water, ambient temperature.

Test Flowratios*

AX/AF = 1.98/1; AX/BS = 6.32/1

Test Flowrates*

AF = 6.20 gph AR = 7.68 BS = 1.95 gph BP = 12.75 gph

AX = 12.26 gph AP = 12.88 gph BR = 2.055 gph

Test Column Throughput*

A-Column = 1004 qsfh; B-Column = 712 qsfh

A-Column Flooding Throughput*

>1500 qsfh

Continuous Phase

A-Column — Organic; B-Column — Aqueous

Pulse Conditions

A-Column: Frequency = 65 cpm; Amplitude = 0.75 inch

B-Column: Frequency = 34 cpm; Amplitude = 0.60 inch

Length of Test

21 hours

* Flowratios were held constant throughout the entire 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,
Stanleigh Concentrate; Test No. 11

	Hour of Test		
	9½	10½	11
<u>A - Column Streams (1004 gsfh)</u>			
<u>Aqueous Feed (AF)</u>			
U (g/l)	193	194	196
HNO ₃ (N)	3.3	3.3	3.3
Th (g/l)	1.09	1.09	1.07
PO ₄ (g/l)	14.4	14.2	13.8
<u>Organic Feed (AX)</u>			
U (g/l)	0.24	0.20	—
HNO ₃ (N)	<0.01	—	—
TBP (%)	34.0	—	—
<u>Organic Product (AP)</u>			
Density and Temperature	0.992 at 84°F	0.993 at 82°F	0.995 at 80°F
U (g/l)	109.9	111.0	111.3
HNO ₃ (N)	0.22	0.22	0.22
Th (g/l)	0.044	0.047	0.053
<u>Aqueous Raffinate (AR)</u>			
U (g/l)	0.012	0.008	0.009
HNO ₃ (N)	2.56	2.58	2.61
Th (g/l)	0.72	0.72	0.74
<u>B - Column Streams (712 gsfh)</u>			
<u>Aqueous Recycle (BR)</u>			
U (g/l)	104.9	105.5	106.8
HNO ₃ (N)	1.33	1.37	1.32
Th (g/l)	0.41	0.40	0.44
<u>Scrubbed Organic Product (BP)</u>			
Density and Temperature	0.966 at 83°F	0.968 at 80°F	0.969 at 78°F
U (g/l)	94.2	94.6	95.1
HNO ₃ (N)	0.024	0.023	0.024
Th (g/l)	0.003	0.002	0.002

TABLE VII Spectrochemical Analyses of Pilot Plant Two-Inch-Column Scrubbed Organic Product (BP)*, Stanleigh Concentrate; Test No. 11
(All values are ppm, U basis)

Element	Hour of Test		
	9½	10½	11
Al	<6.0	<6.0	<6.0
Bi	<1.0	<1.0	<1.0
Cd	<0.2	<0.2	<0.2
Cr	<10.0	<10.0	<10.0
Cu	4.0	6.0	5.0
Dy	<0.02	<0.02	<0.02
Fe	<10.0	29.0	<10.0
Gd	<0.02	<0.02	<0.02
Mg	8.0	7.0	6.0
Mn	<4.0	<4.0	<4.0
Mo	6.0	<4.0	<4.0
Ni	<10.0	<10.0	<10.0
P	100.0	20.0	40.0
Pb	<1.0	2.0	<1.0
SiO ₂	240.0	30.0	<20.0
Sn	<1.0	<1.0	<1.0
V	<40.0	<40.0	<40.0
Zn	<20.0	<20.0	<20.0

* 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.

NOTE: Accuracy of these results is estimated to be ±50%.

TABLE VIII Pilot Plant Two-Inch -Column Organic Stream Analyses,
Stanleigh Concentrate; Test No. 11

Stream	Hour of Test		
	8¼	9¼	9¾
<u>Organic Feed (AX)</u>			
TBP (%)	34.0	—	—
Coalescence Time* (sec) ⁷	145	155	—
Aqueous (%)	—	—	—
Solids (%)	—	—	—
<u>Organic Product (AP)</u>			
Solids (%)	—	0.01	0.01
Aqueous (%)	—	<0.01	<0.01
<u>Scrubbed Organic Product (BP)</u>			
Solids (%)	—	<0.01	<0.01
Aqueous (%)	—	<0.01	<0.01

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

NOTE: All percentages are volume per cent.

DISCUSSION

Contaminant concentrations of Stanleigh concentrate show that the material meets all FMPC impurity tolerance limits except the limits for chloride and thorium (Appendix I). The chloride concentration of this concentrate is 0.17 per cent (U basis), compared with the tolerance limit of 0.125 per cent. The material will have to be blended approximately one to two with a high-quality low-chloride-content concentrate to meet this tolerance limit. The thorium level, which is 5000 ppm (U basis) compared to the primary thorium tolerance limit of 5000 ppm, can be met by handling the material as a thorium containing uranium concentrate² (i.e., 10 per cent PO₄ addition on a uranium basis) or by blending with a low thorium feed.

Digestion of Stanleigh concentrate to give a satisfactory feed slurry for Refinery use should present no problems. The reactivity of HNO₃ dissolution parallels feed materials presently used in the Refinery. The resulting slurry is very low in nitric acid-insolubles and should present no slurry handling difficulties.

The uranium distribution curve for Stanleigh concentrate compares favorably with that for pure UNH. Good extraction characteristics are to be expected in pulse column operation. No emulsification tendencies were noted.

The primary extraction column performance of a feed slurry prepared from Stanleigh concentrate (with an addition of 7 per cent PO₄ on a uranium basis for thorium complexing) was satisfactory. Although an error resulted in the addition to the feed slurry of only 7 per cent PO₄ (on a uranium basis), the thorium contamination of the scrubbed organic product was within the desired level. With normal uranium saturation of the organic product (AP), raffinate losses were within specification. Although intermediate sections of the scrub column (B) were emulsified during the entire test, over-all performance of the column was good, and the scrubbed organic product (BP) contamination levels were well within the desired limits. The A-column flooding capacity exceed 1500 gsfh for this concentrate.

ACKNOWLEDGMENT

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APPENDIX I - IMPURITY TOLERANCE LIMITS FOR FMPC FEED MATERIALS

NOTE: The following 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	40% (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 (U Basis) x %SO ₄ (U Basis) < 40*	Causes column scaling
4. Phosphate (see Item 10)	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 (0.1% on U ₃ O ₈ basis)	Corrosion
7. Fluorides	0.125% on U basis (0.1% on U ₃ O ₈ basis)	Corrosion
8. Gadolinium, Dysprosium, Samarium, Europium	175 ppm each on U basis	Reduces product purity
9. Thorium, no Phosphate	0.5% (max) on U basis	Reduces product purity
10. Thorium, plus Phosphate	2% (max) on U basis with PO ₄ added	Reduces product purity
11. Iron	40% (max) on U basis	Reduces product purity
12. Vanadium	5% (max) on U basis	Reduces product purity
13. Boron	0.7% (max) on U basis	Reduces product purity
14. Copper	4% (max) on U basis	Reduces product purity
15. Organic Material	0.12% (max) on U basis (0.1% on U ₃ O ₈ basis)	Possible explosion hazard with HNO ₃ and reduced extractability due to addition of surfactants
16. Arsenic	Unknown (feed containing 7% As on U basis has been processed satisfactorily)	Reduces product purity
17. Molybdenum	Unknown (feed containing 1.5% Mo on U bases has been processed satisfactorily)	Reduces product purity
18. Cadmium, Lead, Zinc, Chromium, Zirconium, Titanium, Niobium, Hafnium, Beryllium, Tin	Data on which to base limiting conc. of these elements in feed materials are not yet available	Reduces product purity

* This value for a feed slurry which contains 200 g/l U and 3.0N HNO₃. This value changes with uranium concentration and/or HNO₃ normality.

APPENDIX II – MATERIAL BALANCE CALCULATIONS FOR STANLEIGH CONCENTRATE, TEST NO. 11

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

$$\begin{aligned}
 &\text{In} && \text{Out} \\
 &(6.20 \times 194) + (12.26 \times 0.22) + (2.055 \times 105.7) = (12.88 \times 110.7) + (7.68 \times 0.01) \\
 &1422.7 = 1425.8 \\
 &\text{Per Cent Error} = \frac{1425.8 - 1422.7}{1425.8} \times 100 \\
 &= +0.22\%.
 \end{aligned}$$

HNO₃ Balance

$$\begin{aligned}
 &\text{In} && \text{Out} \\
 &(6.20 \times 3.3) + (12.26 \times 0) + (2.055 \times 1.34) = (12.88 \times 0.22) + (7.68 \times 2.58) \\
 &23.21 = 22.64 \\
 &\text{Per Cent Error} = \frac{22.64 - 23.21}{23.21} \times 100 \\
 &= -2.46\%.
 \end{aligned}$$

Thorium Balance

$$\begin{aligned}
 &\text{In} && \text{Out} \\
 &(6.20 \times 1.083) + (12.26 \times 0) + (2.055 \times 0.42) = (12.88 \times 0.048) + (7.68 \times 0.733) \\
 &7.57 = 6.25 \\
 &\text{Per Cent Error} = \frac{6.25 - 7.57}{7.57} \times 100 \\
 &= -17.45\%.
 \end{aligned}$$

*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

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

$$\begin{aligned} & \begin{array}{cc} \text{In} & \text{Out} \end{array} \\ & (12.88 \times 110.7) + (1.94 \times 0) = (2.055 \times 105.7) + (12.75 \times 94.9) \\ & 1425.8 = 1427.2 \\ & \text{Per Cent Error} = \frac{1427.2 - 1425.8}{1425.8} \times 100 \\ & = +0.10\%. \end{aligned}$$

HNO₃ Balance

$$\begin{aligned} & \begin{array}{cc} \text{In} & \text{Out} \end{array} \\ & (12.88 \times 0.22) + (1.94 \times 0) = (2.055 \times 1.34) + (12.75 \times 0.024) \\ & 2.83 = 3.06 \\ & \text{Per Cent Error} = \frac{3.06 - 2.83}{3.06} \times 100 \\ & = +8.13\%. \end{aligned}$$

Thorium Balance

$$\begin{aligned} & \begin{array}{cc} \text{In} & \text{Out} \end{array} \\ & (12.88 \times 0.048) + (1.94 \times 0) = (2.055 \times 0.42) + (12.75 \times 0.002) \\ & 0.62 = 0.89 \\ & \text{Per Cent Error} = \frac{0.89 - 0.62}{0.89} \times 100 \\ & = +30.34\%. \end{aligned}$$

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

In

Out

$$(6.20 \times 194) + (12.26 \times 0.22) + (1.94 \times 0) = (7.68 \times 0.01) + (12.75 \times 94.9)$$

$$1205.5 = 1210.0$$

$$\text{Per Cent Error} = \frac{1210.0 - 1205.5}{1210.0} \times 100$$

$$= +0.37\%.$$

HNO₃ Balance

In

Out

$$(6.20 \times 3.3) + (12.26 \times 0) + (1.94 \times 0) = (7.68 \times 2.58) + (12.75 \times 0.024)$$

$$20.46 = 20.12$$

$$\text{Per Cent Error} = \frac{20.12 - 20.46}{20.46} \times 100$$

$$= -1.71\%.$$

Thorium Balance

In

Out

$$(6.20 \times 1.083) + (12.26 \times 0) + (1.94 \times 0) = (7.68 \times 0.733) + (12.75 \times 0.002)$$

$$6.72 = 5.67$$

$$\text{Per Cent Error} = \frac{5.67 - 6.72}{6.72} \times 100$$

$$= -15.63\%.$$