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Atomic Energy Division,  
SYLVANIA ELECTRIC PRODUCTS INC.  
Bayside, N.Y.

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May 15, 1956

SPECIAL REREVIEW  
FINAL DETERMINATION  
UNCLASSIFIED

Manager of Operations,  
Savannah River Operations Office,  
U.S. Atomic Energy Commission,  
P.O. Box A,  
Aiken, So. Carolina

By: *John L. Zambrow 10/6/80*  
Date: *John L. Zambrow 10/6/80*

Attn: Mr. Paul Hagelston

Dear Mr. Hagelston:

Attached is a statement of progress on the thorium reduction program  
for the period April 1, 1956 to May 1, 1956.

Very truly yours,

SYLVANIA ELECTRIC PRODUCTS INC.

*J. L. Zambrow*  
John L. Zambrow, Manager  
Development Engineering III

JLZ:MD

cc: See Distribution (attached)

Special Rereview  
Final Determination  
Unclassified  
By: K. A. Walter  
Date: 1830  
T. F. Davis

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*[Handwritten signature]*

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The hopper was loaded with 77 lb. of NLO production grade (FP-125)  $\text{ThO}_2$  containing 320 ppm carbon. Before equilibrium was reached, irregular feeding was observed and corrected by installation of a Syntroon vibrator on the hopper. At equilibrium the discharge rate was 0.25 lb/min, and a total of 40 lb. was collected under equilibrium conditions. This calcined oxide was discharged into a polyethylene bag held in a 5 gal. drum. The time of passage through the hot zone was calculated to be 20 min. The weight of oxide in the hot zone was 5 lb. The carbon content of the continuously calcined oxide was 120 ppm which is about the same obtained when the raw  $\text{ThO}_2$  is fired batchwise in boats for 40 hrs. or more.

### III. REDUCTION

Seven reductions were made on the 11 lb. scale in Type 446 stainless steel reactors. Reduction and leaching conditions as well as analytical results are summarized in Table I. The materials used were NLO production grade  $\text{ThO}_2$ ; Baker ACS spec.  $\text{CaCl}_2$  and Helco redistilled calcium. In each run the thorium was calcined by one of the following three methods:

1. Batch firing in a stainless steel boat at  $800^\circ\text{C}$  for 40 hours or more.
2. Heating of the charge (containing raw  $\text{ThO}_2$ ) in situ under vacuum at  $700^\circ\text{C}$  for 5 hours before reduction.
3. Continuous calcining in Bartlett-Snow unit.

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By: K. A. Walter  
Date: 1980  
T. F. Davis

Except for Run #79 the reduction product was crushed further in a jaw crusher after primary size reduction was carried out in the usual manner with a hydraulic press. In Runs #80-83 the jaws were adjusted to comminute the cake to a powder; whereas, in Runs #84-85 the jaws were opened to produce average particles of about  $1/\mu$ .

Leaching was carried out in the tumbling barrel in Runs #79-82. For subsequent runs the impeller agitated leaching apparatus was placed back in operation. The accompanying improvement in metal quality as demonstrated by button hardness suggests that the tumbling barrel was contributing some impurity when it was used. The impurity which has yet to be identified is probably the cause of the unusual phase appearing on the surface of arc-melted buttons. Moreover, this phase has never been observed on buttons made from powder processed by impeller agitation. It is possible, therefore, that the tumbling barrel method of agitation is satisfactory, in principle, despite the difficulty with this particular apparatus.

It is believed that the poor yield in Run #83 was caused, at least partly, by the small particle size of the crushed cake thrown into the leaching tank. Excessive comminution of the powder with impeller agitation had been encountered in earlier investigations.

### IV. RECYCLING OF THORIUM FINES

As described in last month's statement of progress, the fines from Runs #67-73 were collected by a procedure which incorporated the dissolved

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MONTHLY STATEMENT OF PROGRESS  
Reduction of Uranium Oxide by Carbon  
May 1 to June 1, 1956

I. SUMMARY

In this period, the final large scale leachings have been completed. The product will be evaluated. Work has continued on the terminal report. The uranium in unsatisfactory biscuits is being recovered while good biscuits and sponge are being stored.

II. REDUCTION

No reductions were carried out during this period, but a dense UO<sub>2</sub> crucible (9.8 g/cc) was prepared by hydrostatic pressing and sintering at 2200°C. This is to be used in an attempt to prepare a carbon-free product by reduction of a charge containing a slight excess of carbon.

III. LEACHING

Final leachings were completed this month. Still incomplete, however, is the evaluation of the sponge by spectrochemical analysis, and by preparation of buttons.

Results of these leaches are indicated in Table I.

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Date: 1980  
T. F. Davis

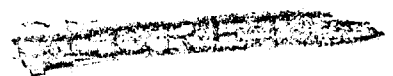
TABLE I

Leach No.	Biscuit No.	Wt. Biscuit Leached (#)	Est. of Metal in Biscuit (by density)	% Yield Metal Sponge	Est. % Leach Yield
L-10	221	38.00	45.2	34.8	77
L-11	222	22.55	39.0*	35.4	90.8
			36.0		98.4

\* Metal content by hydriding

The low yield of L-10 is possibly the result of over-heating during pickling when the temperature inadvertently rose to above 70°C.

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By: K. A. Walter  
Date: 1980  
T. F. Davis



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MONTHLY STATEMENT OF PROGRESS  
Reduction of Uranium Oxide by Carbon  
March 1 to April 1, 1956

Special Review  
Final Determination  
Unclassified

By: K. A. Walter  
Date: 1960  
T. F. Darr

I. SUMMARY

During this period, six reductions have been carried out in the 30-liter furnace under a variety of conditions. Several experiments on continuous addition were carried out in the 1-liter furnace.

By a combination of a slight alteration of the large leaching tank, and by leaching well fused biscuits, yields of bright sponge have been increased. The highest yield to date, i.e., 41.65% yield or 92.5% leaching yield, occurred with a biscuit prepared from stoichiometric pellets.

II. REDUCTION

A. Thirty-liter Furnace

During this period, six reductions were completed in the 30-liter furnace. The mechanical booster pump recently installed permits a final pressure of less than 1 mm to be attained during a reduction. In the first five runs, the charge contained a carbon excess corresponding to 60% U - 40% UC in the biscuit. Run #220 corresponds to 80% U - 20% UC. The results of these runs are given in Table I.

In the first high-carbon-excess runs, i.e., #214, #215, and #217, very little erosion of the crucible was found. In spite of the observation of fusion at the biscuit surface, a cross-section usually showed much unreacted oxide and large gas voids, together with a dispersed metal phase. Only the bottom of the biscuit showed the usual dendritic structure.

In these experiments, the graphite crucible had been lowered 4" below the induction coils to reduce bottom erosion. However, this apparently resulted in overheating of the top of the biscuit, and in trapping CO in the melt.

To reduce the temperature gradient, the crucible was raised into the susceptor sleeve for subsequent reductions.

For reduction #218, a charge of stoichiometric pellets, together with enough pelletized acetylene black covering the crucible bottom to correspond to a 60% U - 40% UC biscuit, was melted at 2240°C. There was little attack on the crucible at the biscuit surface level. The top and outside of the biscuit was fused, but the core consisted of a porous sintered mass, yielding little metal, together with some pellets of unreacted acetylene black.

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In Run #219 the charge was so fluid that part of it ran through the joint in the crucible liner. The entire biscuit was well fused, yielding a dendritic pattern in cross-section.

In the last reduction #220 using a low excess of carbon, the biscuit was collected in a heavy crucible liner with a replaceable graphite pipe insert. This yielded a well fused biscuit that fell out of the crucible easily. Erosion again appeared at the level of the top of the biscuit requiring disposal of the graphite pipe insert.

In order to obtain a biscuit in high yield and with large dendrites of uranium, it appears to be necessary to make the whole melt fluid. The temperature throughout the melt should be uniform. As a result, a certain amount of crucible erosion will have to be accepted.

B. Three-Liter Furnace

Investigation of continuous addition has continued in the three-liter furnace. For these experiments, the inner 5" graphite crucible is fitted with a pellet entry tube and a sight hole. The vacuum head for the furnace tube carries a sight window with a wiper and shutter, an addition tube with a window, and a graphite-tipped poke rod which can be inserted into the sight hole of the crucible. However, this position of the poke rod does not free pellet hang-up in the adjoining addition tube.

As a result of each of the three experiments carried out this month in the apparatus, a semifused crust of pellets formed on the top of the crucible, blocking the pellet addition tube and also preventing observation of the temperature. This has led to overheating and carbiding of the biscuit in most cases.

A change in the position of the poke rod is expected to improve the addition of pellets. On a larger scale many of these difficulties would be expected to disappear.

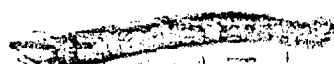
III. LEACHING

Several large scale leaching experiments were carried out this past month with the results tabulated below:

TABLE II

Run No.	Biscuit No.	Wgt. Biscuit Leached (#)	Est. % Metal	Sponge Yields (g)	Est. % Leach Yield
L-4	208	45.4	44	36.85	83.3
L-5	207	42.5	40	34.8	87
L-6	-	47	47.2	43.65	92.5
L-7	207	30.3	40	34.7	86.5

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By: K. A. Walker  
Date: 1990  
T. F. Lamb



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MONTHLY STATEMENT OF PROGRESS  
Calcium Reduction of Thorium Oxide  
February 1 to March 1, 1955

I. Summary

The study of process variables is continuing. Thorium powder of satisfactory quality can be prepared by reducing charges with a  $\text{CaCl}_2$  content of 20 <sup>w</sup>/<sub>o</sub> of the  $\text{ThO}_2$  and a 25% excess of Ca metal.

Small-scale runs were made to investigate the recycling of thorium fines.

II. Reduction

Five reduction runs were made in the eleven pound-scale type 446 stainless steel reactor. The composition of the charge, soaking time and yield for these runs are presented in Table I. The charges consisted of  $\text{ThO}_2$ , HLO production grade (fired at 800°C for at least 48 hours, except for Run 71), redistilled calcium metal (Domag or Melco) and reagent grade  $\text{CaCl}_2$  (Baker).

In Runs 67 and 68 the  $\text{CaCl}_2$  content of the charges was 20 <sup>w</sup>/<sub>o</sub> of the  $\text{ThO}_2$  and the calcium excess was 25%. The soaking times were 5 and 6-1/2 hrs. respectively. The crushed reduction cake was leached in the impeller-agitated 55 gallon leaching tank. After leaching, the resulting powder was acid-washed and dried in the usual manner. Chemical analyses of the resulting powders, hardness and density of arc-melted buttons are given in Table I.

The yield and particle size of the thorium powder in these runs were satisfactory and almost identical. This fact together with the increase in chromium content of the powder resulting from Run 68 indicate that the soaking period should not be greater than 5 hrs. with this charge composition.

In Runs 69, 70 and 71 the charges were blended in the double-cone stainless steel blender. The usual reduction and leaching procedures were employed. Chemical analyses of the resulting powders, hardness and density of arc-melted buttons are given in Table I. The poor yields and the non-uniform reduction cakes indicate that blending is not as efficient as tumbling the charge in jars.

The charge for Run 71 was prepared with unfired  $\text{ThO}_2$ . The charge was heated under vacuum for 2 hrs. at 700°C in an attempt to remove carbon by calcining the oxide in the reactor before reduction. The carbon content was reduced from 0.070% in the  $\text{ThO}_2$  to 0.036% in the Th powder. The crushed reduction cake was leached in the newly acquired stainless steel tumbling barrel. The barrel is 12-sided with a base of 36 in. across, a

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T. E. Galt

depth of 28 in and a top opening of 30 in. diameter. Agitation is achieved by rotating the barrel at 30 rpm on its axis which is 45° from the horizontal.

Since the poor blending may result from overcrowding in the blender, a run is currently being made in which the charge was blended in two portions.

### III. Recycling of Thorium Fines

#### A. Conversion of Fines to Oxide

Approximately 100 gms. of thorium fines from Runs 30 and 31 were converted to oxide by burning with a torch. The resultant oxide was ground in a mortar and pestle and sieved using a 60 mesh screen. The +60 mesh fraction contained some highly refractory oxide flakes which could not be pulverized. The -60 mesh fraction was fired at 750° to 800°C for approximately 16 hrs. and analysed 0.011% carbon.

The charge for Run 50 consisting of 22.3 g. Ca metal (Nalco, redistilled), 19.6 g. CaCl<sub>2</sub> (Baker) and 49.1 g. pre-fired ThO<sub>2</sub> was heated to 950°C in a type 446 stainless steel boat and soaked for 1/2 hr. The reduction cake was processed in the usual manner. The yield of coarse thorium powder was 94%. Chemical analysis of the thorium powder and the hardness of a button arc melted from this powder are presented in Table II.

On the basis of these experiments conversion of thorium fines to oxide for use in reduction is not considered a satisfactory recycling method. Firing of the fines results in a refractory oxide which is not completely reduced as indicated by the high insolubles content of the powder prepared from recycled oxide.

#### B. Use of Thorium Fines "As Is"

The use of thorium fines "as is" in the reduction charge was investigated as an alternative method of recycling. In Runs 60 and 70 the amount of thorium fines added to the reduction charge was varied. The charge for Run 60 contained 85% pre-fired NLC ThO<sub>2</sub>, 41.7 gms., and 15% ThO<sub>2</sub> added as thorium fines from runs 30 and 31, 6.5 gms., and for Run 70 50% pre-fired NLC ThO<sub>2</sub>, 24.6 gms., and 50% ThO<sub>2</sub> added as thorium fines from Runs 30 and 31, 21.5 gms. In addition each charge contained 22.3 g. Ca metal (Nalco, redistilled) and 19.6 g. CaCl<sub>2</sub> (Baker). The charges were heated in the type 446 stainless steel boat and soaked for 1/2 hour. The reduction cakes were processed in the usual manner. The yield of coarse thorium powder was 92% for Run 60 and 89% for Run 70. Chemical analyses of the resultant thorium powder and the hardness of buttons arc melted from these powders are presented in Table II.

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By: K. A. Weller  
Date: 1990

T. F. Sills

The effect of soaking time on the quality of the product was investigated. The charge for these runs contained 85% pre-fired NLO ThO<sub>2</sub>, 41.7 gms., and 15% ThO<sub>2</sub> added as thorium fines from Runs 30 and 31, 6.5 gms. The amount of excess calcium was reduced to 25%, 18.7 gms. (Melsco, redistilled). In addition each charge contained 19.6 gms. CaCl<sub>2</sub> (Baker). The charges were heated to 950°C and soaked for various times. In Run 80 the soaking time was 1/2 hr., in Run 90, 2 hrs., and in Run 100, 1 hr. The yield of coarse thorium powder was 91% for Run 80, 79% for Run 90 and 87% for Run 100. Chemical analyses of the resulting thorium powder and the hardness of buttons are melted from these powders are presented in Table II. These data suggest that recycling the thorium as fines is a satisfactory procedure.

C. Conversion of Fines to Oxalate

A sample of thorium fines from Runs 30 and 31 was dissolved in a 1:1 HNO<sub>3</sub> solution containing a trace of hydrofluoric acid. The addition of oxalic acid precipitated thorium oxalate which was filtered and dried. The oxalate was heated at 850°C for approximately 17 hours converting it to thorium oxide. The oxide analyzed 0.012% carbon.

The charge for Run 110 containing 49.1 g. ThO<sub>2</sub>, prepared from oxalate, 22.3 g. Ca metal (Melsco redistilled) and 19.6 g. CaCl<sub>2</sub> (Baker) was heated to 950°C in a type 445 stainless steel boat and soaked for 1/2 hour. A small portion of the charge did not fuse and was separated from the reduction cake prior to leaching. Chemical analysis of the thorium powder and the hardness of a button are melted from this powder are presented in Table II.

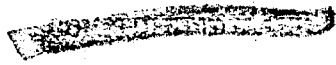
IV. Future Work

Calcining of the ThO<sub>2</sub> in situ in the reactor prior to reduction will be studied further. Recycling fines "as is" on the eleven pound scale is planned essentially duplicating the conditions of Run 80. Work is proceeding on the installation of the laboratory calciner.

Proj. Engineer N. Fuhrman  
N. Fuhrman

Section Head C. Whitman  
C. Whitman

CIM:MT:HS



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Unclassified  
By: K. A. Walter  
Date: 1980  
T. F. Davis



TABLE I

Data for Thoria Oxide Reduction - Runs 67, 68, 69, 70, 71.

No	Charge			Folding Time (hrs.)	Yield (%)	Thorium Powder Analysis (%)						Button Density g/cc	Button Hardness Rockwell C
	UO <sub>2</sub> (g)	CaF <sub>2</sub> (g)	U <sub>2</sub> O <sub>7</sub> (g)			Nitrogen	Carbon	HCl Insol.	Perchloric Acid Insol.	Iron	Uranium		
7	4000	1400	2654	3	86	0.004	0.019	0.12	0.67	0.0012	0.0017	11.67	56
8	4000	1400	2654	5	85	0.004	0.020	0.17	0.71	0.0039	0.0028	11.68	59
9	4000	2275	3029	6	78	0.005	0.017	0.056	0.14	0.0028	0.0026	11.67	40
0	4500	2100	2654	5	83	0.004	0.015	0.13	0.75	0.0058	0.0022	11.66	55
1	4000	1750	2654	3	71	0.004	0.036	0.075	0.016	0.0062	0.0019	11.67	62

TABLE II  
Recycling Studies

Run No.	Thorium Powder Analysis (%)				Hardness Rockwell C
	Carbon	Nitrogen	HCl Insol.	Perchloric Acid Insol.	
50	0.024	0.061	0.36	0.64	77
60	0.016	0.049	0.27	0.73	81
70	0.024	0.014	0.22	0.78	72
80	0.019	0.009	0.54	0.46	55
90	0.026	0.013	0.16	0.81	66
100	0.023	0.012	0.15	0.82	66
110	0.030	0.010	2.76	0.014	83

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R. K. A. Walker  
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I. F. Davis  
1990

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MONTHLY STATEMENT OF PROGRESS  
Reduction of Uranium Oxide by Carbon  
February 1 to March 1, 1956

I. Summary

During this period, a reduction run was completed in the thirty-liter furnace. The biscuit yield was 85.6% and the metal content 45%, according to density measurements. The thirty-liter furnace has been modified by the incorporation of a high-speed booster pump into the vacuum system. The investigation of continuous addition of charge to the reduction crucible is continuing. Leaching of the biscuit crushed by pressing has been completed. Forging has been investigated as a method of crushing the biscuit.

See view  
Final Determination  
Unclassified

II. Reduction

By K. A. Walter  
Date 1959  
T. E. Davis

A. Thirty-Liter Furnace

Prior to the installation of the booster pump, a reduction run was completed in the 30-liter furnace. In Run 210 a charge corresponding to 70% U, 30% UC was heated to a final temperature of 2235°C, at which point the CO pressure was 3.2 mm of Hg. The biscuit contained unfused areas at the bottom, but the top of the biscuit had eroded through the 5/8" thick liner, penetrating about 1/4" into the crucible wall. The data for this run and the averages obtained for this and the five other runs in the series are given in Table I.

The furnace has been modified by the incorporation of a high-speed booster pump into the vacuum system. The pumping system now consists of a 1200 cfm Kinney mechanical booster pump backed by a 220 cfm rotary pump. Leak testing of the complete assembly has been satisfactory, and an ultimate vacuum of 20 microns (McLeod) has been attained. The booster pump, which functions only when the system pressure is below 15 mm of Hg, increases the pumping speed by a factor of about five. It is intended to utilize this added pumping speed primarily toward the end of a reduction. By removing the evolved CO more rapidly, and thus reducing its pressure above the charge, it should be possible to determine the effect of low CO pressure on the quality of the biscuit, as well as to see how this variable affects fusion temperature, power consumption and time needed for fusion. To this end, a series of runs will be made on the one hundred-pound scale, using a charge equivalent to a 60% metal biscuit. This will afford a direct comparison with similar runs reported last month. The first of this series of reduction runs is now in progress.

B. Three-Liter Furnace

In one run in the 3-liter furnace, raw charge was added to the crucible during reduction. The crucible originally contained 3760 gms. of UO<sub>2</sub> and carbon, in a ratio to give a 60% metal product. The charge was heated to about 1800°C, and raw charge was added. A total of 3600 gms of raw charge was added in two portions. Clouding of the sight window prior to fusion prevented observation of the final temperature, and a carbided biscuit was obtained. The run will be repeated using a head with a means for cleaning the sight glass.

### III. Leaching

The leaching of three segments of Run 205 biscuit which were subjected to various pressing procedures has been completed. The biscuit density was 15.86 g/cc, corresponding to a metal content of 49.5%. The segments were leached in 40°C water containing detergent.

The first biscuit quarter, L-1, (8163 gms.) was pressed with a pointed ram which resulted in a number of small pieces. Most of the biscuit fell to sponge after 28 hours in the leaching medium. The heel was leached for an additional 65 hours in room temperature water. The yield of sponge plus heel was 28.8% based on the initial weight of the biscuit. The sponge from the first two taps analyzed 300 to 450 ppm carbon and from subsequent taps 70 to 200 ppm carbon.

The second biscuit quarter, L-2, (9100 gms.) pressed with a rounded ram was water-leached for a 28 hour period - the heel for an additional 24 hours. The total yield of sponge plus heel was 30.9%. Sponge from the first two taps contained 370 to 800 ppm carbon and from subsequent taps 90 to 550 ppm carbon.

Leaching of the third quarter of the biscuit, L-3, (9850 gms.) pressed with a flat ram was carried out over a 29 hour period; the heel was leached for an additional 70 hours at room temperature. The total yield of sponge was 33.8%. The first two taps analyzed 220 to 600 ppm and 1200 ppm carbon, respectively. The carbon contents of taps 3 and 4, which comprised the bulk of the product, were satisfactory, i.e., 70 to 470 ppm but the carbon content of the last tap was also high, 2200 ppm. Thus, the actual yield of carbide free metal should be less than the 33.8% observed. The high carbon contents may be attributed to insufficient cold work and to insufficient time in the leaching medium.

The leaching yields for this biscuit have been considerably lower than those in the past. This is possibly due to incomplete fusion of the biscuit during reduction which results in finely-divided uranium metal which dissolves during leaching. Another possibility is that the density is not giving a true value of metal content (see below).

Since it is difficult to subject all parts of a U-UC biscuit to uniform amounts of cold work by pressing in a 300 ton press, the biscuits from Runs 206, 207, and 210 were forged in a 3000 pound forge (total weight of hammer and anvil was 3000 pounds). Each biscuit was pre-leached for 3 hours before canning in a steel forging case. The biscuit segments will be leached in 40°C water containing detergent and the yields of pickled sponge will be determined.

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Final Determination  
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### IV. Additional Studies

By E. A. Miller  
Date: 1953

To determine more exactly the per cent of metal in a biscuit, an analytical method based on hydriding is being studied. A weighed sample is hydrided, then decomposed. The volume of hydrogen evolved is measured with a wet-test meter. The results of this method may then be checked with metal contents as determined by density and carbon content. Two samples have been tested. In the first, a metal content of 48.5% was

obtained based on density measurements; hydriding showed 43.2% metal. In the second, density indicated 42.9% metal, while hydriding showed 53.2%. The method will be checked further.

TABLE I

Data for Reduction in Thirty-Liter Furnace

	<u>Run 210</u>	<u>Average of 6 Runs</u>
Fusion Temperature (°C)	2235	
Pressure at fusion (mm Hg)	3.12	
CO volume (cu.ft.) calculated	294	
CO volume (cu. ft.) observed	296	
Product weight (lbs.) calculated	88.5	
Product weight (lbs.) observed	88.5	
Material balance (%)	100.0	99.8
Biscuit weight (lbs.)	75.75	
Biscuit Yield (%)	85.6	86.4
Power consumption (KWH)	313	
KWH per pound of biscuit	4.14	3.91
KWH per pound of charge	2.86	2.68
Archimedes' density (g/cc)	15.64	
Metal Content of biscuit (%)	45.0	43.4

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 Unclassified  
 By: K. A. Walter  
 Date: 1980  
 T. P. Lewis

Project Engineers:

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CKW:HB

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C. I. Whitman  
 C. I. Whitman



NY 7

MONTHLY STATEMENT OF PROGRESS  
Calcium Reduction of Thorium Oxide  
December 1 to January 1, 1956

I. SUMMARY

During this period five reduction runs were made in the eleven pound-scale type 446 stainless steel reactor. In all runs the reduction cake was removed without difficulty and there was very little pickup of iron and chromium.

Evaluation runs of production grade thorium oxide from National Lead of Ohio indicate that it is a satisfactory raw material. Thorium powder produced in these runs was arc melted to give buttons of hardnesses below Rockwell F 44. The HCl insolubles content of the powder averaged 0.17%.

II. Evaluation of NLO Production Grade Thorium

Three evaluation runs of production grade thorium oxide from National Lead of Ohio were made in the eleven pound-scale type 446 stainless steel reactor. The charge for Runs 60, 61 and 62 consisted of 7000 gms.  $\text{ThO}_2$  (NLO production grade, fired at  $800^\circ\text{C}$  for at least 20 hrs.), 2800 gms.  $\text{CaCl}_2$  (Baker), 3184 gms. Ca metal (Nelco redistilled or Donag). The charge was fired at  $950^\circ\text{C}$  and soaked for 2 hrs. The cooled reduction cake was removed without difficulty, crushed between flat plates in a hydraulic press, and then leached in the 55 gallon leaching apparatus. Agitation was maintained for two hours and then the cake was leached statically. The resulting powder was processed in the usual manner. The yield of coarse thorium powder was 83% for Run 60, 78% for Run 61 and 80% for Run 62. Chemical analysis of these powders is presented in Table I and hardnesses and densities in Table II.

III. Evaluation of Rare Earth Inc. Thorium

Another evaluation run of Rare Earth Inc. thorium was made in the eleven pound-scale type 446 stainless steel reactor. The oxide was crushed and passed through a 100 mesh sieve before the pre-firing treatment. A charge of 4200 gms.  $\text{ThO}_2$  (Rare Earth Inc., fired at  $800^\circ\text{C}$  for 20 hrs.), 1680 gms.  $\text{CaCl}_2$  (Baker) and 1910 gms Ca metal (Nelco redistilled) was heated to  $950^\circ\text{C}$  and soaked at temperature for 1.5 hrs. After cooling, the cake was dropped out by inverting the reactor and jolting about three times. The reduction cake was crushed between flat plates in a hydraulic press and then leached in the 55 gallon leaching apparatus. The unleached cake was agitated for approximately two hours and then allowed to disintegrate statically overnight. The powder was processed in the usual manner and the yield of coarse thorium powder was 85%. Chemical analysis of the powder is presented under Run 59 in Table I. Hardnesses and densities of buttons arc melted from this powder are given in Table II.

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Submitted  
A. Walker  
1956  
12/15/56

The oxide content (HCl insolubles) of the powder prepared in Run 59 (0.48%) from crushed  $\text{ThO}_2$  is not appreciably lower than in Run 57, from uncrushed  $\text{ThO}_2$ . Another evaluation run is planned using another type of Rare Earth Inc. thorium.

#### IV. Reduction

In Run 63 a charge of 7000 gms.  $\text{ThO}_2$  (NLO, production grade, fired for 68 hours at  $500^\circ\text{C}$ ), 2654 gms. Ca metal (Dowag) and 2800 gms  $\text{CaCl}_2$  (Baker) was soaked for 3.5 hrs. at  $950^\circ\text{C}$ . The calcium excess was 25% and the soaking time was increased to insure complete reduction. The reduction cake was removed without difficulty and then crushed prior to leaching in the normal manner. The yield of thorium powder was 91%. Chemical analysis of the +80 mesh and -80 mesh fractions is presented in Table I. The hardness and density of buttons arc melted from these powders is given in Table II.

TABLE I  
Thorium Powder Analyses

Run No.	Impurity, %						Yield
	Nitrogen	Carbon	HCl Insol.	Perchloric Acid Insol.	Iron	Chromium	
59	0.004	0.014	0.48	0.67	0.0038	0.0010	85
60	0.004	0.016	0.16	0.15	0.0011	0.0011	83
61	0.005	0.014	0.19	0.10	0.0035	0.0013	78
62	0.004	0.021	0.15	0.04	0.0046	0.0008	80
63+80 mesh	0.004	0.026	0.16	0.11	0.0065	0.0019	91
63-80 mesh	0.004	0.020	0.13	0.11	0.0044	0.0010	

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TABLE II

Density and Hardness of Arc Melted Buttons

Button No.	Density g/cc	Hardness Rockwell F
59-1	11.68	33
59-2	11.69	31
60-1	11.69	40
60-2	11.70	40
61-1	11.69	35
61-2	11.67	43
62-1	11.67	44
62-2	11.68	41
63-1 +80 mesh	11.66	41
63-1 -80 mesh	11.69	36
63-2 +80 mesh	11.70	40
63-2 -80 mesh	11.70	38

V. Future Work:

Now that the breakout problem has been solved, the emphasis of the process development will be shifted toward a more thorough investigation of process variables such as calcium excess, soaking time and calcium chloride quality. Equipment will also be procured to put the process on a firm engineering basis.

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Final Determination  
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Date: 11/15/83  
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