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METALLURGY AND CERAMICS

UNITED STATES ATOMIC ENERGY COMMISSION

RESEARCH AND DEVELOPMENT IN THE FIELD OF THORIUM CHEMISTRY AND METALLURGY

Monthly Progress Report [for] March 15 thru April 15, 1955.

By
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Monthly Progress Report

RESEARCH AND DEVELOPMENT IN THE FIELD OF
THORIUM CHEMISTRY AND METALLURGY

For: Manager
U. S. Atomic Energy Commission
Savannah River Operations Office
P. O. Box A
Augusta, Georgia

Att: Mr. Paul J. Hagelston

Period: March 15 through April 15, 1955
Contract No. AT(30-1)-1335

RECEIPT NUMBERS
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By: Dr. James L. Wyatt
Project Director

[redacted]

DISCLAIMER

SUMMARY

The period ending April15 was host to a number of process changes in the development program under contract AT(30-1)-1335. A transition program, eliminating most of the wet chemical techniques for the preparation of cell feed and replacing these with a high temperature chlorination process, was begun. A complete metamorphosis cannot be achieved for several weeks, however, this being dependent upon the receipt of a large continuous filter.

A number of electrolytic runs of an experimental nature were completed in Mark II, XX and XXV electrolytic cells, and the latter unit was completely rebuilt in order to evaluate the use of ceramic components for cell lining applications.

At the end of the report period it developed that the technique utilized at Horizons for HCl-insolubles determination did not coincide with that in use at AEC installations, and that the lack of correlation of results possibly was due to improper equipment and process innovations.

Crushing tests on a Fitz mill were initiated and tentative conclusions are that this type of equipment is superior to a combination of jaw and roll crushing for producing more uniform metal product powder sizes and a minimum quantity of fines.

Small scale chlorination experiments have indicated the feasibility of pelletizing thorium oxycarbonate for feed to a continuous high temperature chlorinator. A medium-sized chlorinator is currently under construction which will evaluate thorium oxycarbonate as a feed material within the next report period.

A portion of the report period was devoted to an intensive clean-up and decontamination campaign, together with the relocation of certain pieces of equipment in order to place all operations, both experimental and pilot, in a restricted area of the company.

Summary (Cont.)

The program during the coming report period will have major emphasis on the preparation of a sizable quantity of high concentration cell feed in anticipation of a continuous cell campaign in the Mark VI cell in late May or early June.

RESEARCH AND DEVELOPMENT IN THE FIELD OF THORIUM

CHEMISTRY AND METALLURGY

INTRODUCTION

Continued efforts under contract AT(30-1)-1335 for the development of an economically and technically feasible method of producing thorium metal have continued during the present report period. Finite progress has been hampered somewhat by the decision to modify the process, install and place into operation new pieces of equipment, as well as an appreciable amount of time expended on decontaminating certain areas of the laboratory. An AEC health and safety laboratory report on operating conditions at Horizons Incorporated prompted Horizons' management to change the location of certain physical facilities being utilized by the project, inasmuch as they were not formerly within a restricted area. Because of the inherent radiation hazards involved, it was deemed necessary to relocate certain operations in the interests of health and safety of personnel both on and off this particular effort.

Insofar as possible the progress of research and development work has continued, the results of which will be described below.

Cell Feed Preparation

Preparation of Cell Feed by Wet Chemical Processes

The preparation of electrolytic cell feed by wet chemical techniques was continued throughout most of this period, primarily in order to complete the materials which were in progress during the early stages of the month. Current plans will involve shutting down this operation early in May if the alternative approach to cell feed preparation proves satisfactory. Certain aspects of the wet chemical method will still be utilized, inasmuch as the modified process involves the preparation of thorium oxycarbonate by chemical precipitation from a nitrate solution through the reaction with sodium carbonate.

A continuous filter is on order for inclusion in this process, and a continuous belt drier already has been completed and currently is going through its initial exploratory operations prior to actual small scale production scheduling. An experimental chlorinator of the internally heated variety patterned after a magnesium chloride reactor also is nearing completion and should be in operation within two to three weeks.

Some 650 kilograms of contained metal have been processed during the past month through various stages of the wet chemical process. At this writing approximately 70 kilograms of contained metal in the form of electrolytic cell feed were on hand and an additional quantity of 173 kilograms awaited sublimation treatment.

A proportionately large amount of time has been spent in the sorting and classification of radioactive scrap materials, to the extent that there now are on hand some 38 drums of material, most of which has been analyzed and evaluated for thorium content. A shipment to scrap storage will be made during the coming month.

Preparation of Cell Feed by Direct Chlorination Techniques

During the report period six chlorination runs were completed in the Mark XXIII chlorinator unit. The results of these reactions are presented in Table I (page 18).

Details of each of these chlorination efforts have been covered in previous weekly reports with the exception of run No. 19, which will be reported on here in some detail.

The nineteenth run in the Mark XXIII cell was an attempt to produce a 45% thorium cell feed, beginning with a 26% thorium product resulting from run No. 18. Thorium nitrate tetrahydrate was added in order to bring up the thorium content to the desired level.

One hundred and fifty pounds of run No. 18 material, together with 30 pounds of thorium nitrate tetrahydrate, were added initially into the furnace. An additional quantity of 25 pounds of TNT was added while the temperature was being raised to a level of about 650°C. Chlorination was started following eight hours of heat-up and melt-down time. Additional quantities of TNT were added in accordance with the schedule presented in Table II (page 19). Most of the remainder was added in five or ten pound increments during the latter stages of the run.

Phosgene consumption on this run was extremely high, and may have been associated somewhat with a new type sparger head which was considerably more porous than varieties formerly used. This coincides with small scale experimental work which indicated that the bubble size of gas is a critical factor on the over-all conversion rate of the chlorinating agent.

The exceptionally long time required for this operation, and difficulties in furnacing the melt during charging cycles, tend to substantiate further the fact that thorium nitrate tetrahydrate is not a suitable starting material for chlorination experiments at elevated temperatures. The oxycarbonate form will be used as the starting source material as soon as a sufficient quantity becomes available from the wet chemical processing.

Electrochemical Operations

Mark II Electrolytic Cell Operations

An experimental series of electrolytic runs in the Mark II cell has been completed, but as yet analytical data have not been made available. These heats have had a number of variations imposed upon them, including a determination of the effects of pre-sparging the bath with reducing and chlorinating agents, freezing the deposits in the bath, varying initial and final thorium concentrations in the salt matrix, determinations of thorium concentration gradients

within the deposit, the effects of quench cooling the deposit, results of intentionally inoculating the bath with impurities and many other variations. Evaluations of these heats will be held up until such time as an arc melting furnace, currently under construction, can be completed, installed and made ready for operation.

Mark XX Electrolytic Cell Operations

Earlier in the year a series of runs in the Mark XX electrolytic cell was initiated in an attempt to determine the effects of particle size and current density on the HCl insolubles of the resulting metal product. This series now has been completed, but again no analytical data are as yet available.

Additional runs in the Mark XX cell have been completed, primarily for the purpose of stockpiling materials for use in metal washing and recovery process variable evaluations. It is anticipated that this electrolytic cell will be removed from service on or about April 25, there being no further anticipated requirement for its use.

Mark XXV Electrolytic Cell Operations

The primary objective in the operation of the Mark XXV cell during the present report period has been to obtain operating data on heating elements and crucible design components. The experiments have included a number of modifications, such as the introduction of ceramic plates instead of graphite component parts for crucible lining and variations in heating element design, construction and positioning.

Variations in the technique for removing molten salt from the electrolytic cell also have been evaluated. In the thirtieth run a siphon tube consisting of two sections of metal tubing was joined by a tube union. It was found that if heat were applied to the union during the entire siphoning operation, no difficulty was encountered with freeze-up or interference in salt flow. It

therefore becomes possible to reclaim used tubes that have imperfections in them by sectioning and adjoining with unions.

Earlier experience has shown that the greatest amount of corrosion on nickel siphon tubes had occurred in the portion subjected to continuous exposure in the molten bath. An experiment therefore was tried in which graphitite replaced the nickel tube in this area of the siphon device. In one run the graphitite was spliced around the nickel through the medium of a standard 3/4" tube union specially machined to fit a threaded section inside the graphitite. A further attempt at splicing consisted of placing the nickel tubing directly inside the graphitite tube with a very tight fit, the joint being cemented. The latter was not successful, apparently as the result of differential expansion of the two materials.

Some additional data have become available on certain of the electrolytic runs made in the Mark XXV during previous report periods. In the twenty-third electrolytic run the cathode deposit was frozen in the bath under polarizing conditions after electrolysis. The initial thorium concentration of this bath was 6.4%, and current efficiency based on metal recovery was approximately 81%.

Table III (page 20) presents the results of a series of hardness tests on thorium metal produced in runs 14 through 21.

During the latter two weeks of the present report period the Mark XXV electrolytic cell was torn down completely for certain modifications and a general overhaul. A new graphite slab crucible has been constructed and installed in anticipation of an additional series of electrolytic experiments. It was of interest to note that salt penetration through the slab crucible which was removed was practically non-existent with the exception of a small leak at one end of the crucible where two slab joints came together.

Modifications in the cell and auxiliaries include the formation and installation of a heat shield directly on the heating element flange, changes in the design of the radiation heat shields in the upper portion of the cell, installation of a Hevi-Duty DC constant current regulator, relocation of the cooling system and fabrication of a hood to cover the upper portion of the Mark XXV cell unit.

Toward the later part of the report period an intensive clean-up campaign was initiated, including acid etching of the floor followed by painting and the general decontamination of the entire area. It is anticipated that this clean-up campaign will continue for an additional ten days to two weeks.

Metal Recovery

Major efforts in this department have continued toward the evaluation of factors affecting the HCl-insolubles content of Horizons' thorium metal. Difficulties in correlating results with process variations continued to be encountered, and tentatively it appears probable that the analytical procedures have been at fault.

The HCl-insolubles procedure utilized to date has duplicated that prescribed in the publication governing the chemistry of the Manhattan project of some three years ago. Recent test runs at Horizons, however, failed to give satisfactory duplicate analyses, and the procedure became suspect. Somewhat fortuitously the matter was brought up at a meeting in Oak Ridge and it was found that other AEC affiliates had experienced similar problems in the application of the HCl determination.

The Oak Ridge Laboratories appear to have resolved the problem in recent months by modifications in the technique and equipment used for the analysis, and reported reproducible results to within about 2%. Modifications include the use of concentrated HCl as the solvent, without dilution, and the application of reflux condensers to the solution step. Filtration on filter paper and subsequent ignition.

as opposed to Gooch filters and oven drying, also were recommended.

These modifications to the process have been instituted at Horizons, and first results indicate definitely improved results. It is unfortunate that these analytic techniques were not applied to Horizons' problems sooner, for much of the work on HCl-insolubles factor would seem to be subject to question.

While it may not be proper to draw final conclusions based on HCl-insolubles data, certain trends are sufficiently evident to warrant positive statements. The purity of the original metal deposits is a major factor in the amount of HCl-insolubles present in the melted pellet. Another interesting point was the fact that metal, dried at temperatures as high as 100°C. after washing procedures had been completed, was reported to have much lower insolubles contents than metal which had been vacuum dried in a dessicator at room temperature.

Effects of Pelletizing on HCl-insolubles

Almost without exception there has been a noticeable increase in HCl-insolubles content when going from the powder form to the melted form. So far this has not been attributable to oxidation during melting, based on results obtained from titanium of iodide grade in the same furnace melted under substantially identical conditions. It therefore appears to be inherent in the metal itself, and a major amount of investigation has been aimed at the resolution of just what causes this phenomenon. As a first step in this direction some pelletized samples, just prior to melting, were analyzed for HCl-insolubles. It was found that even in this form a major increase in insolubles content had already occurred, approximately to the same level as was normally found in the melted specimen. A series therefore was arranged in which the pressure utilized in pelletizing was made the major variable. Table IV (page 20) summarizes the results obtained in this experiment. With reference to Table IV, it is most interesting to note that on three different analyses of the powder, carefully sampled, the HCl-insolubles

reported ranged from .096% to 1.11%. This is typical of the results we have been getting during the last several months in trying to correlate the effects of various treatments on the metal powder. It is felt that these anomalies very largely are due to the improper method of running HCl-insolubles prior to the present date.

Crushing Tests

In the last Weekly Progress Report some data were presented which resulted from experimentally crushing a cathode deposit in a Fitz mill, the latter unit being manufactured by the Fitzpatrick organization. These data unfortunately did not include the weights of extremely fine material, possibly less than -325 mesh, which was carried over in the washing operation and collected in settling chambers.

Table V reports the corrected figures on the best run in the Fitz mill, as compared with a standard crushing operation using jaw crushers and roll crushers at Horizons. With reference to Table V it may be noted that results from the Fitz mill, at least insofar as sizing is concerned, are appreciably better than the results from utilizing the jaw crusher and the roll crusher combination. Inasmuch as the Fitz mill was not operated under the most optimum conditions because of a shortage of feed material, it is anticipated that even further improvement could be made in the sizing results providing sufficient experimentation were allowed. Based on these data, it would be our recommendation that a Fitz mill be considered for crushing of the cathode deposit in a pilot or production plant.

Facilities Modifications

Up until the present time most of the experimental washing operations, the melting and pelletizing facilities and a number of other related operations have been located outside the radiation restricted area. Because of the relatively high radiation contamination associated with these operations,

Horizons' management has decided that it is necessary to modify the building layout in order to enclose these operations completely. Inadequate dust collection and fume collection systems were available in the areas where these operations were being carried on, and from a straight health standpoint it seems improper to continue the operation any further in its present location. Accordingly, these phases of the program have now been completely removed from the front portion of building B and are set up on a temporary basis in the cell room until such time as certain major pieces of equipment can be removed from the cell operation area and a portion of the building walled off to house these operations. Personnel normally assigned to metal recovery processing have been utilized full time for decontamination operations, and have been very successful in eliminating almost completely the background alpha count in the rooms previously occupied by the washing department. It is hoped that this group can be gotten back into at least small scale operation within the coming week.

Experimental Program

Small Scale Experimental Chlorination Operations

Some additional work has been done during the past month in evaluations of the cascade system approach to higher utilization of chlorinating agents. In one such experiment three vycor tube furnaces were utilized, each being charged with 26 grams of KCl-NaCl eutectic mixture, some 40 grams of basic thorium oxycarbonate being added for chlorination purposes. Hydrogen chloride gas at a rate of four mols per hour was sparged into the No. 1 furnace, the off-gases from this passing through sulfuric acid drying towers, thence into furnace No. 2, and this operation repeated for furnace No. 3. It was found that chlorination was complete in furnace No. 1 after 200 minutes, in furnace No. 2 after 145 minutes and in furnace No. 3 in 285 minutes. These results

certainly show that exhaust gases can be passed through a series of furnaces, thus increasing the efficiency of hydrogen chloride gas as a chlorinating agent to a marked extent. Due to the unusual order of completion, this experiment will be repeated with some modifications. Instead of running all units to completion, the experiment will be stopped as soon as any one of the three furnaces has produced a clear product. Analyses will then indicate the relative efficiency of the other two in the series.

The second experiment duplicating the one described above has been run and the material submitted for analyses. As yet, no data are available.

In an attempt to product 100% thorium tetrachloride, 40 grams of basic thorium carbonate were charged into a vycor tube furnace and heated to 525°C. for one hour. Chlorine gas was bubbled through carbon tetrachloride, and the off-gases sparged through the dry carbonate for the entire period of time. This material has been submitted for analyses.

A series of runs has been completed in which a salt mixture containing two mols of sodium chloride and one mol of potassium chloride have been enriched successively with thorium to levels approximating 10% to 90% thorium tetrachloride in steps of 10%. These materials will be utilized to determine the liquidus and solidus lines in the ternary system $NaCl$-KCl-$ThCl_4$.

A further experiment was made in an attempt to chlorinate basic thorium carbonate in straight sodium chloride at 800°C. utilizing hydrogen chloride gas as the reactant. The rate of reaction proved to be extremely slow and the experiment was discontinued before it was completed.

A further series of experiments was run in which basic thorium carbonate was dried at 240°C. for 1-1/2 hours and served as a starting material for evaluations of pelletized products in the chlorination operations. A portion of the dried carbonate was ground in a pebble mill and pressed into one gram pellets for experimentation.

In one such run utilizing these pellets, a KCl-NaCl eutectic mixture held at 700°C. was enriched with the pelletized thorium oxycarbonate and sparged with hydrogen chloride gas. The chlorination reaction went to completion, indicating that a pelletized product is a satisfactory feed for chlorinating.

To check further the feasibility of a pelletized product, two simultaneous reactions were run in which thorium oxycarbonate in pellet form was charged to one furnace and thorium oxycarbonate in the powder form charged to another. Each was treated at the same temperature and with the same hydrogen chloride gas flow. There was no noticeable difference between the rates of chlorination in the two experiments.

In a still further experiment utilizing pellets and powder, it was found that even though the pelletized form is used, the oxycarbonate should be added incrementally. If too large a quantity is added at any one time, the mixture goes through a pasty stage and chlorinates only with difficulty. The operation of a large scale chlorinator would therefore be envisioned with a continuous or semi-continuous charging of pelletized basic thorium carbonate in small incremental quantities, rather than periodic charges of large quantities of this material.

Rates of Chlorination - HCl vs. $COCl_2$

In order to evaluate to a further extent the relative effects of hydrogen chloride and phosgene as chlorinating agents, two sets of duplicate experiments were run utilizing these two reactants on Lindsay oxycarbonate and pre-dried oxycarbonate made at Horizons. Table VI (page 21) compares the results of these tests, representing the number of minutes required to react each of a series of five-gram thorium oxycarbonate additions.

It is interesting to note in Table VI that apparently during the initial

stages of chlorination, before the thorium content becomes too high, hydrogen chloride gas is far more efficient than phosgene in the conversion to the chloride. However, it appears tentatively that the reverse is true for higher concentrations. The actual times represented in Table VI should perhaps be discounted, since under similar conditions 20 grams of wet process carbonate have in the past been chlorinated with hydrogen chloride gas in as little as 90 minutes, as compared with nearly 400 minutes in the case cited in the table. Apparently this reaction rate is strongly dependent upon the temperature to which the oxycarbonate has been dried, and possibly is related to the amount of CO_2 driven off of the oxycarbonate as a result of over-heating.

Qualitatively it has been noted that when utilizing hydrogen chloride gas as the chlorinating agent, the greater part of the thorium oxycarbonate appears to react to form the thorium tetrachloride in a relatively short period of time. However, in order to complete the chlorination the reaction generally has to be continued for an appreciable length of time, this being particularly true in a bath containing very high contents of thorium tetrachloride. In order to correlate the time of hydrogen chloride flow with the per cent conversion, a series of experiments was run in which 24 grams of a cell feed containing 40% thorium were melted in each of three vycor tube furnaces at 700°C. In one of these furnaces five grams of basic thorium carbonate were added and sparged for one hour with hydrogen chloride gas at a rate of four mols per hour. In the second unit ten grams of the carbonate were added and sparging was continued for two hours. In the third furnace 15 grams of carbonate were added and the reaction carried out for three hours. The products of these operations are currently in for analysis, but the results as yet are not available.

Preparation of Thorium Chloride with Carbon and Chlorine

Some further work has been done during the present report period in an attempt to produce thorium tetrachloride with carbon and chlorine as the reactants. For these experiments wet process basic thorium carbonate, oven-dried at 200°C. for two hours, was utilized for the starting material. This product analyzed 4.5% water and 14% carbon dioxide. It was milled in a ball mill with lampblack in a ratio ranging from 50% to 150% of the theoretical required for the reduction of thorium oxycarbonate. These mixtures then were pressed into pellets 1/2" in diameter and approximately 1/8" thick.

Sodium chloride-potassium chloride eutectics were utilized in the experiments, melting the bath at 700°C. and charging approximately 30 grams of pellets over a 15-minute period to 20 grams of the eutectic, sparging chlorine gas at a rate of two cubic feet per hour. In each experiment the gas flow was maintained for 1-1/2 hours following the final addition. The results of these experiments have been submitted for analyses, but as yet no data are available.

Evaluation of Ceramic Materials

For the past several months there has been a small scale effort in the ceramics department aimed at the evaluation of ceramic bodies for application in contact with molten salt systems. Some difficulties have been experienced in obtaining suitable crucible materials which will not contaminate salts, and furnace designs capable of maintaining high temperatures for months at a time in order to carry out these tests properly. As a result of building several types of furnaces a design finally has been found which seems to serve adequately, and three additional units are being constructed.

To date a number of determinations have been completed, and McDaniel Mullite, Star Porcelain No. 148 and U. S. Stoneware Elite all appear to be adequate for molten salt service. This experimental work will continue for several months in the future.

Extrusion of Thorium Metal

On April 5 a piece of lead was extruded in Horizons' experimental thorium extrusion equipment in order to determine the workability of the extrusion die and associated equipment. It was found that the lead extruded very nicely with no difficulties being encountered.

Fortified with the knowledge that there was nothing unworkable in the die design, a quantity of thorium metal powder containing 0.43% HCl-insolubles was obtained, compacted cold at a pressure of five tons per sq. in. and sintered at 1830°F. for one hour. The sintering was done under a vacuum averaging 2×10^{-5} mm. of mercury. Liquid nitrogen was maintained in a cold trap during the entire sintering operation.

The sintered product showed a density of 7.64 grams per cubic centimeter and an HCl-insolubles content of 1.38%. Metallographic examination showed a white layer on the outside of the specimen along with white particles inside the specimen fairly randomly distributed. Knoop microhardnesses with a 50-grams load were measured as follows: 54, 49, 57, 52.

Prior to initiation of the extrusion the inside of the container and the entire surface of the die were thoroughly coated with four layers of a graphite lubricant. The sintered slug also was coated with this dag material. The furnace was assembled and evacuated overnight. It pumped down to 28 microns as an ultimate vacuum prior to heating. The furnace was then heated to 230°F. for two hours for moisture elimination, after which an ultimate vacuum of 35 microns was obtained. The furnace and die assembly were flushed with argon twice and the temperature raised to 1275°F.

At a load of 44,000 lbs. per square inch the thorium began to extrude at a rate of 1/16" per minute. The load was held constant throughout the run, and resulted in extruding a slug 1-11/32" long into a rod 12-1/8" long, the

latter having a diameter of 0.255".

After completion of the extrusion the furnace and die assembly were cooled to room temperature and the specimen removed. The initial end of the extruded rod was black and pitted for a length of approximately 1-1/2". The appearance of the remaining portion of the rod was good and the rod showed adequate ductility by the qualitative test of bending it back and forth.

Knoop hardness values using a 50-gram load on the end of the extruded rod measured 65, 73, 62, 75, 69 and 66. HCl-insolubles analyses on the rod showed 1.68% insolubles.

Metallographic examinations of the longitudinal section of the rod showed a large number of white stringers which produced a somewhat banded structure. The sections appear to be fairly homogenous.

Considerable difficulty was experienced in removing portions of the die from the female section. As a result of this operation the die became very badly scored and could not be used again. In order to make it somewhat easier to remove the die from the container on the next run, the bottom of the hole in the female portion will be opened up for approximately 1/32" on the diameter for a height of 1" to facilitate removal of the internal component parts. Die modifications are expected to be completed within a week.

Metallographically the result of the extrusion test appeared somewhat confusing. Based on the area covered by the second and third phases present, one would have expected from 5% to 10% insolubles in the metal. In contrast to this, the insolubles content was less than 2%. Accordingly, a small quantity of the white phase, which had concentrated to a marked extent on the slug prior to extrusion, was scraped off and submitted to the physics department for X-ray analysis. While results are not completely proven, it appears tentatively that there are indications of two forms of thorium nitride

in this material. This result unfortunately has not been confirmed by the analytical chemistry group, which reports a very low nitrogen content. Additional investigations will be made in an attempt to explain the presence of this rather peculiar phase, which is separate and distinct from the gray-colored thorium oxide.

Future Plans

It is expected that the coming month will see major efforts applied to working out the bugs in processes for producing cell feed on moderate sized quantity scales, relocation of major equipment components in the electrolytic and metal recovery sections, further clarification of the HCl-insolubles problem and the initiation of construction of the Mark XXVI cell. It is hoped that the modifications in our HCl-insolubles techniques will eliminate the major variations and results which have been obtained during the past several months, and that it will be possible to correlate major process variables with the quality of the metal being produced.

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Cleveland, Ohio
April 26, 1955
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TABLE I

Summary of Direct Chlorination Runs

	Run No.					
	14	15	16	17	18	19
Total Weight of charge	285	325	336	336	336	425
Pounds of TNT charge	90	150	175	175	175	275
Pounds of Thorium charge	78	74	70	70	70	149
Time required to melt charge (hrs.)	16	15*	15	15	15	8
Pounds of phosgene used	517	551	560	750	880	1725
Time phosgene flowed	22	29	24	36	37	103
Ave. flow rate phosgene lbs./hr.	23	19	23	21	23	17
Pounds of salt recovered	208	264	248	217	260	-
Concentration of product	26.5	25	26	20	26	-
Thorium recovered in product	55	66	64	44	68	-

* Charge for Run 15 added to hot crucible after completion of Run 14.

TABLE II

Run No. 19 (291-20) Summary

Cumulative Hours	Cumulative Lbs. TNT	Cumulative Lbs. Th.	
0	30	51	Plus 150 lbs. of 26% Th feed
6	35	53	
6.5	40	55	
7	55	61	Started chlorinating after 8 hours.
22	85	73	
32	95	77	
34.5	125	89	30 lbs. TNT in 10 lb. incr.
47.5	130	91	Approx. 20 lbs. salt erupted from unit.
68	230	131	100 lbs. TNT in 5 lb. increments
70	235	133	approx. 5 lb./hr.
72	240	135	
76	245	137	
80	250	139	
81	263	144	
82	269	146	
83	275	149	
111			Siphoned.

Phosgene usage = 1725 lbs.

Recovered salt:

Siphoned = 145 lbs.
Crucible = 125 lbs.
Salt eruption = 20 lbs.

Total: 290 lbs.

TABLE III

Rockwell B Hardness of Mark XXV Electrolytic Products

Run No.	Hardness
14	23.5
15	15
16	38
17	39.5
18	37
19	35
20	30.5
21	20

* * * * * * *

TABLE IV

Effects of Pelletizing Pressure on HCl-insolubles Content in Thorium Metal

Sample No.	Pelletizing Pressure	% HCl-insolubles
1	Powder not compacted	0.096
2	300 psi compaction pressure	0.59
3	500 psi compaction pressure	0.38
4	800 psi compaction pressure	0.44
5	1200 psi compaction pressure	0.32
6	1600 psi compaction pressure	0.43
Recheck on starting material	Not compacted	1.11
Second recheck on starting material	Not compacted	0.62

TABLE V

Comparative Effects of Crushing Metal-Salt Deposits in Fitz Mill and Jaw and Roll Crushers

Particle Size	Roll Crushed Product % in specified size range	Fitz mill crushed product % in specified size range
-40 mesh +200 mesh	67.8	77.5
-40 mesh +160 mesh	50.4	65.0
-200 mesh	32.2	22.5

* * * * *

TABLE VI

Comparison of Reaction Rates of $COCl_2$ and HCl

Chlorinating Agent	Basic Th. Carbonate	Reaction Time (minutes) for Each Five Gram Addition									
		1st	2nd	3rd	4th	5th	6th	7th	8th	9th	10th
$COCl_2$	Lindsay	44	36	31	33	31	30	30	25	20	23
HCl	Lindsay	17	41	74	128	*					
$COCl_2$	Wet chemical process - pre-dried	52	61	37	38	47	26	29	33		
HCl	Wet chemical process - pre-dried	20	36	65	114	**					

* 5th addition incompletely reacted after 165 min.

** 5th addition was completely reacted in 117 min. by dropping the reaction temperature to 600°C.