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HW-19932

CONVERSION OF UNH TO UO_3 - TERMINAL REPORT

by

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INTRODUCTION

Batchwise calcining of aqueous uranyl nitrate to UO_3 is a well established process. The investigation of continuous processing has been underway at several sites to determine the feasibility of cost reduction, (4, 5) greater dust control, and better control of the quality and uniformity of the product. A continuous operation should also be capable of greater production capacity for a specific application of plant facilities in the form of space and equipment.

Continuous production of UO_3 by spray decomposition and by drum drying has been previously reported.(7,8,9,13) The feasibility of a continuous calcining process was outlined in a laboratory progress report(2) the data from which are incorporated in this report. Continuous denitration has been reported at Mallinckrodt(11, 12) for the addition of molten nitrate to a bed of hot trioxide in a furnace-heated trough-type reactor. A similar reactor and a fluidized bed process are also under study at Hanford.(4, 5)

OBJECTIVES

This work was undertaken to determine the feasibility of producing UO_3 by the continuous addition of concentrated uranyl nitrate solution to hot, agitated UO_3 . The study sought to determine:

- a. Optimum operating conditions for the process,
- b. The extent of corrosion,
- c. Heat transfer coefficients,
- d. Heat consumption as a function of feed concentration,
- e. The fate of phosphate ion in the conversion process.

SUMMARY AND CONCLUSIONS

It has been found feasible to convert uranyl nitrate to uranium trioxide by the continuous addition of concentrated uranyl nitrate solution to hot, agitated UO_3 powder. Batchwise rather than continuous removal of the oxide from the conversion pot was utilized in order to simplify the experimental requirements.

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Satisfactory product was obtained with feed concentrations greater than 78 weight percent uranyl nitrate. Minimum lumping occurred with 80 to 84 percent uranyl nitrate in the feed and a decomposition temperature of 300°C. The decomposition was complete in less than 5 minutes at 250°C. In the laboratory reactor, high agitation rates (100 rpm optimum) and agitation during cooling tended to favor the production of fine powder. Feed addition was effected successfully at rates varying from 1.4 to 15.9 weight percent of the initial UO_3 charge per minute.

For the conversion of solid $UO_2(NO_3)_2 \cdot xH_2O$ to UO_3 , the heat consumption was calculated to vary from 300 BTU/lb. of UO_3 produced for $x = 0$, to 850 BTU/lb. for $x = 6$. For a feed containing 88 percent uranyl nitrate (boiling point 160°C.) 1.43×10^7 BTU would be required for processing 10 tons of uranium. With a heat transfer coefficient of 10 BTU/hr.(ft.²)(°F.) and a temperature differential of 800°F., 93 square feet of heat transfer surface would be required to achieve and maintain the reaction temperature, and denitrate an initial feed of 88 percent uranyl nitrate, to produce the 10 tons of uranium.

Phosphate ion was found to remain with the uranium in the conversion process. Corrosion of the stainless steel calcining pot was negligible. Analysis of the product from all runs gave an average of 83.0 weight percent uranium, 0.35 percent nitrate, less than 0.01 percent U_3O_8 , and less than 3 percent loss on ignition. The bulk density (not packed) varied from 2.7 to 3.5 g./cc.

DISCUSSION

Equipment

The calcination runs were carried out in a 4-inch x 8-inch stainless steel pot with a hemispherical bottom. The pot was heated by a variac-controlled resistance furnace. Thermocouples were used to obtain the temperatures at the heating elements, in the pot wall, and in a well inside the pot. An anchor type agitator with a 1/16-inch pot-wall clearance was driven by a one-quarter horsepower, thymotrol-controlled motor operating through a 20-to-1 gear reduction. The available agitation rates ranged from 20 to 150 rpm.

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Experimental

A known weight of UO_3 was charged to the pot and brought to the required temperature by means of the furnace. After setting the desired rate of agitation, aqueous uranyl nitrate feed was added to the reactor at a controlled rate from a heated feed vessel through a heated feed line. The denitration off-gases were passed through a trap and then through a scrubbing tower. On completion of a run, the reactor and contents were cooled as rapidly as possible (15 to 20 minutes) by means of an air blast directed against the outside of the pot wall after removal of the heating furnace. Trap and scrubbing tower samples were analyzed at the completion of a run to determine the off-gas uranium losses. The latter were generally less than 0.5 percent.

Laboratory Calcination Variables

A total of 18 runs was completed under a variety of conditions summarized in Table I along with the results. An initial trial run (I) was made in the equipment utilizing a straight charge of UO_3 with no feed added. Runs II through V were made using a feed prepared by continuous laboratory concentration of simulated TBP-process RCU solution, and runs VI through XVIII were made using a feed concentrated by the Equipment Development group of the Chemical Development Unit in a stainless steel tube-type evaporator.

The composition of the feed varied from 75 to 84 percent $\text{UO}_2(\text{NO}_3)_2$. In run XVIII a lumpy product was formed with a feed concentration of 75 percent uranyl nitrate but under otherwise optimum conditions; nitrate analysis showed that decomposition was essentially complete in this run. Run XVI, which was made under similar conditions but with a feed containing 78 percent uranyl nitrate, produced a fine powder. Increasing the feed concentration permitted a higher denitration rate for a given set of heat transfer conditions, and the higher rate decreased the tendency for lump formation. Attempts to add a feed more concentrated than 84 percent $\text{UO}_2(\text{NO}_3)_2$ failed in the laboratory equipment due to freezing in the feed line.

Decomposition time was studied as a function of temperature in runs X through XIII and in run XV. In these runs, 50 g. of feed were added rapidly to 300 g. of agitated UO_3 which had been preheated to the required temperature. After a specified time interval the furnace was turned off and removed from the pot. A jet of air cooled the pot and contents below the decomposition temperature in approximately 15 minutes. Nitrate analysis of the product showed that decomposition was complete in each case tried, down to a minimum 5-minute reaction time at 250°C .

It may be seen by reference to Table I that reaction temperatures below about 250°C . tended to give a lumpy product. Higher temperatures generally yielded powder. Run VII did not give powder because in

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addition to the use of a low agitation rate during feed addition, the agitation was shut off and cooling was begun within five minutes of completion of the feed addition. In all other cases agitation was continued during the cooling period.

The effect of the rate of agitation is illustrated by runs III and IV, which were made under otherwise similar conditions. The reactor contents in run III were agitated at 40 rpm during the period of feed addition and at 130 rpm during the drying period. A lumpy product resulted. Run IV employed an agitation rate of at least 130 rpm throughout the run and produced a fine powder. As noted above, run VII was made under conditions of slow agitation, but the absence of both a drying period and agitation during cooling probably contributed to the lumpy product which was formed. An agitator speed of 100 rpm appeared to be optimum for the equipment used.

The rate of feed addition is of necessity limited by the rate of heat transfer available. For an estimated heat transfer coefficient of 10 BTU/(hr.)(ft.²)(°F.) and a temperature differential of 800°F., the heat flux available is 8000 BTU/(hr.)(ft.²). This requires approximately 93 sq. ft. of heat transfer surface for calcining 88 percent UN at a rate of 10 tons of U/day. Feed addition rates in the laboratory apparatus were varied from 1.4 to 15.9 percent of the initial UO₃ charge per minute, the latter value being much higher than that required for plant operation at a 10 ton per day rate.

For a given uranyl nitrate hydrate composition, the heat of decomposition may be calculated from the heats of formation of the reactants and products. This heat of formation of uranyl nitrate as a function of the degree of hydration is plotted in Figure 1 as calculated from data in CL-697. For 88 percent uranyl nitrate (100 percent UNT; boiling point 160°C.) it may be seen that its heat of formation is 544 kilocalories per mole. The ΔH at 25°C. for the calcination may then be calculated from the various heats of formation according to the following equation:

For this reaction, as written, ΔH at 25°C. equals 190 kilocalories. From such calculated values, the heat of decomposition of UO₂(NO₃)₂·xH₂O has been plotted as a function of degree of hydration, and is shown in Figure 2 in terms of BTU per pound of UO₃ produced. At the operating temperature, about 10 to 15 percent NO exists in equilibrium with NO₂. Insufficient heat capacity data are available to calculate accurately the heat of reaction at 300°C., but a difference of only plus or minus 10 percent is estimated. For 88 percent uranyl nitrate feed, approximately 600 BTU/lb. of UO₃ produced are required for calcination. This is equivalent to 1.43 x 10⁷ BTU for processing 10 tons of uranium. The

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dependence of the heat of calcination on feed composition is shown in Figure 3 for a production rate of 10 tons of uranium per day.

To determine the rate of heat transfer through dry UO_3 the decomposition pot was charged with UO_3 and preheated to 150°C . Full voltage was then applied to the furnace. Temperatures in the pot and around the heating elements were recorded periodically with the rate of agitation being held constant. From the data obtained, an apparent heat transfer coefficient of $2.3 \text{ BTU}/(\text{hr.})(\text{ft.}^2)(^\circ\text{F.})$ was calculated for the laboratory equipment at a temperature differential of 700°F . Much higher heat transfer coefficients were experimentally determined (3,6) for the Mallinckrodt plant calcining pots under conditions similar to those described in this report. For the continuous processing reaction mixture, the coefficient was calculated to be $13.9 \text{ BTU}/(\text{hr.})(\text{ft.}^2)(^\circ\text{F.})$, and for dry UO_3 powder it was reported as $7.8 \text{ BTU}/(\text{hr.})(\text{ft.}^2)(^\circ\text{F.})$.

Feed Concentration

Heat requirement considerations led to the study of further concentration of feed before its denitration. From the data in Figure 1, it may be seen that by concentrating the feed from 83 to 88 percent uranyl nitrate (100 percent UNT), an 18 percent reduction of the heat required during denitration was realized. This presents the advantage, previously mentioned, of reducing the tendency to produce a lumpy product.

In order to obtain information as to the physical properties and stability of uranyl nitrate solutions at relatively high boiling points, a UNH solution was concentrated in a system which allowed either evaporation or total reflux. The solution volume was reduced step-wise to give increasing uranyl nitrate concentrations held under reflux for relatively long periods of time. The data obtained are tabulated below and plotted in Figure 4.

<u>CONCENTRATION OF UNH SOLUTION (TBP Free)</u>			
<u>Boiling Point,</u> <u>$^\circ\text{C.}$</u>	<u>Freezing Point,</u> <u>$^\circ\text{C.}$</u>	<u>$\text{UO}_2(\text{NO}_3)_2$,</u> <u>Wt. %</u>	<u>Observations</u>
120	60	78.5	Clear; refluxed 4 hours
140	108	83.8	Clear; refluxed 2 hours
160	146	87.8	Clear; refluxed 4 hours
178	167	92.0	Clear; refluxed 72 hours
188	172	94.0	NO_2 evolved slowly; solution slightly turbid in 2 hours

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The stability of the above solutions differed greatly from that reported in HW-18406 (1) which describes the continuous concentration of $\text{UO}_2(\text{NO}_3)_2$ solution containing TBP (tributyl phosphate). For this reason the experiment was repeated using a uranyl nitrate solution containing TBP. The data obtained are tabulated below and indicate oxidation of TBP by nitrate ion with the accompanying formation of UO_3 . Qualitative tests showed the amount of UO_3 produced to be proportional to the TBP concentration. No settling of UO_3 from the heated solutions was noted indicating satisfactory calcination feed.

CONCENTRATION OF UNH SOLUTION CONTAINING 0.2 g./l. TBP

Boiling Point, °C.	Observations
120	Foaming
140	Foaming; trace of NO_2 evolved; solution amber
145	One-half-inch layer of foam; NO_2 evolution increased
150	Three-quarter-inch layer of foam; NO_2 evolution constant
156	Foaming and NO_2 evolution decreased
162	Foaming and NO_2 evolution ceased
174	Solution turbid

Path of Phosphate Ion

Dissolved TBP is present in RCU to the extent of about 0.02 volume percent plus a varying amount of entrained solvent. Consequently the path of PO_4^{3-} through the denitration process was determined on the assumption that no organic was stripped from the uranium solution prior to calcination. In run XVII, H_3PO_4 was added to the feed to produce a PO_4^{3-} concentration of 0.94 percent; analysis after calcination showed 85 percent of the total PO_4^{3-} originally present in the feed remained in the product. In run XIV, the feed contained 0.20 percent PO_4^{3-} produced from the decomposition of TBP, and of this total PO_4^{3-} present in the feed, 90 percent remained in the product. If the TBP is completely stripped from the RCU stream, then the only phosphate reaching the denitration pots will be from TBP hydrolysis products, e.g., dibutyl phosphate, which are not volatile with steam.

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Corrosion

Some of the concentrated feeds and UO_3 products were analyzed spectrographically to estimate the corrosive effect on stainless steel of the RCU concentration process and the $\text{UO}_2(\text{NO}_3)_2$ decomposition (see Table II). The wet assays listed in Table I for total iron are probably more accurate than the spectrographic analyses; however, good wet assays for chromium and nickel under the existing conditions were not available. The results indicate little pick-up of the stainless steel components by the process materials either during the RCU concentration stage or during the decomposition of the uranyl nitrate. Two of the samples show relatively high values for sodium, boron and phosphorus, which probably resulted from the use of glass equipment for the RCU concentration runs carried out in the laboratory. The abnormally high values for iron in the product from runs XV, XVI, and XVII, are believed to be due to corrosion of the cast iron packing gland follower in the reactor cover plate.

UO_3 Product Quality

Chemically, the product from all runs was satisfactory. Nitrate analyses, which are a measure of the degree of decomposition, ranged from 0.17 to 0.83 weight percent with an average value of 0.35. Uranium varied from 82.3 to 83.7 weight percent with an average value of 83.0 (theoretical composition of pure UO_3 is 83.2 percent U). At the temperatures employed, no correlation between the various analyses and the temperatures or heating times could be made. The material which was HCl-insoluble (a measure of the amount of U_3O_8 formed) averaged less than 0.01 weight percent, and in many cases was not detectable. The significance of the loss-on-ignition test is doubtful due to the hygroscopic nature of UO_3 , but the average loss on ignition was approximately 3 weight percent, as found.

The results obtained by the determination of particle size distribution in laboratory-produced UO_3 samples are summarized below. The samples were not ground after calcination. The determinations were made with the Fisher-Dotts particle size apparatus which utilizes differentiation of particle sedimentation rates in a liquid dispersion medium⁽¹⁴⁾. The size distribution was determined on the fines obtained by screening the samples through a 325-mesh screen. Some difficulty was encountered in finding a suitable dispersing medium, and the results were not entirely reproducible. In general, 50 to 75 percent of the product was retained on a 325-mesh screen while most of the fines were found to range from 10 to 20 microns in diameter.

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UO₃ PARTICLE SIZE DISTRIBUTION

Diameter, Microns	Distribution, Percent				
	Run I(2)	Run III(3)	Run IV(3)	Run V(3)	Run VI(3)
44.0 (1)	53.2	70.3	78.9	48.8	62.5
30.0 - 44.0	1.0	6.5	5.5	8.4	11.1
25.0 - 30.0	4.2	1.8	1.8	3.1	5.4
20.0 - 25.0	10.8	1.1	2.8	4.9	6.6
15.0 - 20.0	20.8	9.5	7.5	23.4	6.1
10.0 - 15.0	10.0	8.2	3.3	9.6	4.9
10.0	<u>0.0</u>	<u>2.6</u>	<u>0.2</u>	<u>1.8</u>	<u>3.4</u>
	100.0	100.0	100.0	100.0	100.0

- Notes: (1) Percent retained on 325 mesh screen.
 (2) Dispersed in 100 parts of glycerine diluted with 75 parts of methanol.
 (3) Dispersed in 100 parts of glycerine diluted with 90 parts of water.

Data on the distribution of the coarse particles are summarized in the following table of sieve test results. The results are rather widely divergent, even for runs made under similar conditions, and would probably have been profoundly influenced by even coarse grinding of the UO₃ product.

UO₃ PRODUCT SIEVE TESTS

Run No.	Classification, Percent			Bulk Density, G./Cu.Cm.
	Retained on 80 Mesh	Retained on 100 Mesh	Through 100 Mesh	
IV	2.9	2.9	94.2	—
V	20.6	3.7	75.7	2.7
VI	14.3	9.4	76.3	3.5
X	9.0	5.0	86.0	3.1
XI	23.2	5.8	71.0	3.1
XII	36.6	3.6	59.8	—

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FUTURE WORK

No additional work on the production of UO_3 by the continuous (or semi-continuous) calcination of uranyl nitrate solutions is planned by the Process Chemistry group of the Chemical Development Unit. Development studies of the fluid bed and continuous screw techniques, however, are current elsewhere within the Unit. See references (4) and (5), below.

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TABLE I

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CONTINUOUS CONVERSION OF $UO_2(NO_3)_2$ TO UO_3

Run No.	Uranyl Nitrate Feed Solution (1)						Reaction Temperature, °C.					UO ₃ Product							
	UO ₃ Initially Charged to Reactor, Grams	Amount Added, Grams	Addition Rate, Grams/Minute	UN, Wt. Percent	HNO ₃ , Wt. Percent	Fe, (2) Wt. Percent	At Start of Addition	At End of Addition	At End of Drying Period	Agitator Speed, RPM	Length of (3) Drying Period, Minutes	Yield, Grams	Quality of Powder	Uranium, Wt. Percent	Nitrate, Wt. Percent	HCl -Insoluble, Wt. Percent	Loss on Ignition, Wt. Percent	Fe, (2) Wt. Percent	UO ₃ Material Balance
I	400.2	—	—	—	—	—	—	—	—	40	—	380.8	Fine	83.36	0.06	0.03	2.52	—	95.2
II	400.0	99.6	9.96	82.37	ca. 1	0.021	215	195	205	40	60	438.2	Lumpy	83.34	0.19	0.026	3.18	—	95.5
III	400.2	176.8	7.07	82.37	ca. 1	0.021	220	200	260	41-130	120	480.7	Lumpy	82.47	0.22	0.068	3.01	—	95.0
IV	600.0	135.7	4.85	82.37	ca. 1	0.021	237	197	266	130-150	60	647.2	Fine	83.25	0.20	0.023	2.79	—	95.4
V	—	284.4	7.48	82.37	ca. 1	0.021	274	248	289	139-166	30	162.5	Fine	83.12	0.83	0.014	3.85	0.036	95.8
VI	—	1315.4	7.52	83.85	0.87	0.007	254	276	315	76-146	60	706.4	Fine	83.67	0.42	0.0013	2.90	0.001	90.4
VII	321.2	248.5	4.50	79.56	0.86	0.015	302	246	—	58	5	444.1	Lumpy	83.17	0.26	0.004	—	0.016	95.6
VIII	348.8	417.3	14.4	79.56	0.86	0.015	254	174	210	120	30	566.4	Fine	82.75	0.48	Not Detected	—	0.012	96.8
IX	350.0	548.9	23.8	79.56	0.86	0.015	196	163	224	110	150	659.6	Chunk	82.58	0.39	0.011	3.34	0.014	98.9
X	300.0	50.0	857.0	78.42	0.64	0.010	318	318	300	100	10	309.6	Fine	83.16	0.30	0.003	2.57	0.026	94.6
XI	300.0	50.0	857.0	78.42	0.64	0.010	318	318	310	100	15	313.2	Fine	82.75	0.44	0.004	2.74	0.006	95.6
XII	300.0	50.0	857.0	78.42	0.64	0.010	318	318	293	100	5	309.9	Fine	82.82	0.22	Not Detected	2.91	0.016	94.5
XIII	300.0	50.0	857.0	78.42	0.64	0.010	275	275	267	100	10	306.2	Fine	83.68	0.20	Not Detected	3.02	0.008	93.6
XIV	300.0	455.7	25.3	78.42	0.64	0.010	300	200	250	100	30	536.3	Fine	82.61	0.51	Not Detected	3.04	0.01	97.2
XV	300.0	50.0	857.0	78.42	0.64	0.010	250	250	225	100	5	326.4	Fine	82.32	0.45	Not Detected	3.01	0.11 (4)	99.5
XVI	300.0	360.5	25.8	78.42	0.64	0.010	296	277	400	100	30	484.9	Fine	83.15	0.17	Not Detected	2.53	0.11 (4)	97.6
XVII	150.0	188.0	12.0	83.70	—	—	300	270	290	100	60	245.2	Fine	82.96	0.34	< 0.01	—	0.15 (4)	96.1
XVIII	153.0	360.9	24.2	75.12	—	—	300	245	240	100	30	344.3	Lumpy	83.20	0.41	—	—	—	99.7

NOTES: (1) The feed was prepared by concentrating simulated TBP - Process RCU solution (see text).
(2) Wet chemical analysis.
(3) An additional 15 to 20 min. were required to cool the reaction mixture below the decomposition temperature.
(4) These abnormally high values are due to corrosion of the cast iron packing follower in the pot cover plate.

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TABLE II
SPECTROGRAPHIC ANALYTICAL RESULTS⁽¹⁾

<u>Element</u>	<u>Uranyl Nitrate Feed for Run V</u>	<u>UO₃ Product from Run V</u>	<u>UO₃ used for Synthesis⁽²⁾ of Uranyl Nitrate Concentrator Feed</u>	<u>Uranyl Nitrate⁽³⁾ Feed for Run VI</u>	<u>UO₃ Product from Run VI</u>
Ag	20	50	0.1	L 0.1	0.1
As	L 5	L 5	L 5	L 5	L 5
B	20	20	L 0.2	0.5	0.2
Be	L 0.1	L 0.1	L 0.1	L 0.1	L 0.1
Bi	2	L 1	L 1	L 1	L 1
Cd	100	100	L 1	L 1	L 1
Co	L 100	L 100	L 100	L 100	L 100
Cr	50	50	L 10	10	10
Cu	200	100	L 20	L 20	L 20
Fe	50	50	L 20	20	50
K	50	50	L 50	L 50	L 50
Li	L 5	L 5	L 5	L 5	L 5
Mg	50	100	L 10	20	20
Mn	10	10	L 5	L 5	L 5
Mo	L 20	L 20	L 20	L 20	L 20
Na	G 1000	G 1000	L 5	L 5	20
Ni	20	20	L 10	L 10	L 10
P	100	100	L 5	50	L 5
Pb	20	20	L 5	L 5	L 5
Sb	L 2	L 2	L 2	L 2	L 2
Sn	L 5	L 5	L 5	L 5	L 5
Zn	200	L 100	L 100	L 100	L 100

- Notes: (1) In parts per million parts of U₃O₈. L indicates "less than";
G indicates "greater than".
- (2) This synthetic RCU solution was prepared by the Equipment Development group of the Chemical Development Unit for use in the tube-type evaporator studies.
- (3) This UO₂(NO₃)₂ feed for the calcination run was prepared in the Equipment Development tube-type evaporator (see Note (2), above).

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HEAT OF DECOMPOSITION OF $\text{UO}_2(\text{NO}_3)_2 \cdot x\text{H}_2\text{O}$

EFFECT OF DEGREE OF HYDRATION

FIGURE 3
CALCINATION HEAT REQUIREMENTS
EFFECT OF FEED COMPOSITION

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