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THORIUM FUEL CYCLE DEVELOPMENT PROGRESS REPORT

APRIL 1970

NO. 9

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CHEMICAL TECHNOLOGY DIVISION
METALS AND CERAMICS DIVISION

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I. HEAD-END REPROCESSING DEVELOPMENT

R. E. Blanco, W. E. Unger, V. C. A. Vaughen, C. D. Watson

II. REFABRICATION DEVELOPMENT

R. G. Wymer, A. L. Lotts, R. E. Brooksbank, P. A. Haas
W. D. Bond, J. W. Snider, J. D. Sease

III. MATERIALS IRRADIATION

A. L. Lotts, T. N. Washburn, J. D. Sease, J. H. Coobs

IV. LWBR SUPPORT PROGRAM

J. R. Parrott, R. E. Brooksbank

V. URANIUM-233 REPROCESSING

R. E. Brooksbank, J. R. Parrott

JUNE 1970

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CONTENTS

		Page
INTRODUCTION		1
I.	HEAD-END REPROCESSING DEVELOPMENT	
	Studies with Irradiated Fuels	2
	Head-End Engineering Studies	2
II.	REFABRICATION DEVELOPMENT	
	Particle Preparation	3
	Fueled Graphite Fabrication Development	7
III.	MATERIALS IRRADIATION	
	HTGR Recycle Fuels Irradiation	16
IV.	LWBR SUPPORT PROGRAM	
	Storage Facilities	24
	Purification System	25
	Nitrate-to-Oxide Conversion Line	29
V.	URANIUM-233 REPROCESSING	
	Dissolution and Purification	31
	Storage and Distribution	31

THORIUM FUEL CYCLE DEVELOPMENT PROGRESS REPORT

APRIL 1970

NO. 9

Compiled by

The Staff of the Chemical Technology and
Metals and Ceramics Division

INTRODUCTION

The thorium fuel cycle development at ORNL is directed almost solely at HTGR fuels. These fuels consist of large blocks of graphite containing coolant channels and fuel and blanket holes. The fuel and blanket are made of microspheres of uranium or thorium compounds separately, or of mixtures of them in a single microsphere. The microspheres are coated with layers of pyrolytically deposited carbon and in some cases silicon carbide. The microspheres are retained in the holes in the graphite blocks in either unbonded or bonded states.

Development work on all aspects of HTGR fuel recycle is in progress at ORNL. In addition, a major recycle development facility, the Thorium-Uranium Recycle Facility (TURF), has been built at ORNL, and the Coated Particle Development Laboratory (CPDL) has been put into operation in Building 4508. TURF is intended to be used as a development facility for fuel recycle. The CPDL is for engineering development studies leading to design of the equipment to be used in TURF.

I. HEAD-END REPROCESSING DEVELOPMENT

(R. E. Blanco, W. E. Unger)

The objective of this program is to evaluate head-end processes for converting irradiated HTGR fuels to a form suitable for recovery and decontamination of the thorium and uranium by a solvent extraction process. Small samples of irradiated and unirradiated fuel are studied to determine effects of irradiation on fuel reprocessing steps and to correlate with metallographic studies. An important objective is the determination of the amount of breakage of coatings and the resultant amount of cross contamination of the fertile Th-^{233}U and fissile ^{235}U components in alternative reprocessing steps. Mechanical systems are being developed for degrading the fuels and providing a material suitable for use in studies of the burn-leach steps using fluidized bed for fixed-bed burners. The mechanical and burn-leach engineering development work is carried out using full-scale unirradiated fuel of the type to be used in PSC, and is designed to provide scale-up data for use in the design of pilot or full-scale processing plants.

1. Studies with Irradiated Fuels

(V. C. A. Vaughen, J. H. Goode, G. Davis)

During this month extensive negotiations were begun with the Dragon Reactor Project to obtain irradiated and control HTGR fuel compacts for use in hot cell studies in FY 1971.

2. Head-End Engineering Studies

(C. D. Watson, R. S. Lowrie)

The head-end engineering studies are comprised of two principal areas of investigation - mechanical and burn-leach. The general approach followed has entailed the mechanical dissection, by sawing, of full-sized graphitic blocks containing fuel sticks, followed by comminution to size fractions suitable for the burn-leach process. However, the recent GGA decision to use loose fuel particles rather than fuel sticks as the reference form has necessitated changes in the approach.

There is nothing to report in this area this month.

II. REFABRICATION DEVELOPMENT

1. Particle Preparation

(R. G. Wymer - Coordinator)

The fuel material of primary interest for ORNL recycle studies is the ThO_2-UO_2 particle, which has a thorium-to-uranium ratio of about 4.2. The uncoated fuel particles are to be microspheres 350 ± 100 μ in diameter, made by the sol-gel process. Fuel preparation includes development and demonstration of all process steps involved in making remotely the ThO_2-UO_2 microspheres. The steps include demonstrating reliable, remote processes for reproducibly mixing $Th(NO_3)_4$ and $UO_2(NO_3)_2$ solutions in the desired throium-to-uranium ratio, preparing the mixed, stable ThO_2-UO_3 sol in concentrations exceeding 1 g-mole of oxides per liter, forming ThO_2-UO_3 gel microspheres, and converting them to dense ThO_2-UO_2 in good yield.

1.1 Sol and Microsphere Preparation Development

(P. A. Haas)

Experimental engineering studies of processes and equipment for preparation of sols and microspheres are reported here. The present emphasis is on processes, procedures, and prototype equipment for preparation of oxide microspheres in the Thorium-Uranium Recycle Facility (TURF). Tests of flowsheets and procedures to be used for test materials preparation are also included.

Preparation of Gel Spheres Without Fluidization

As compared to fluidized-bed columns for preparation of gel spheres, non-fluidized columns are simpler to operate and are limited to smaller diameter of product. The sol droplet must be gelled before it settles to the bottom of the column. The size limitation can be calculated from a combination of mass transfer and particle settles correlations (see February report). Figure 1.1 shows calculated results for four alcohol conditions and experimental points for three of them. The curve I results correspond to conditions possible with recycle of 2EH. The curve II

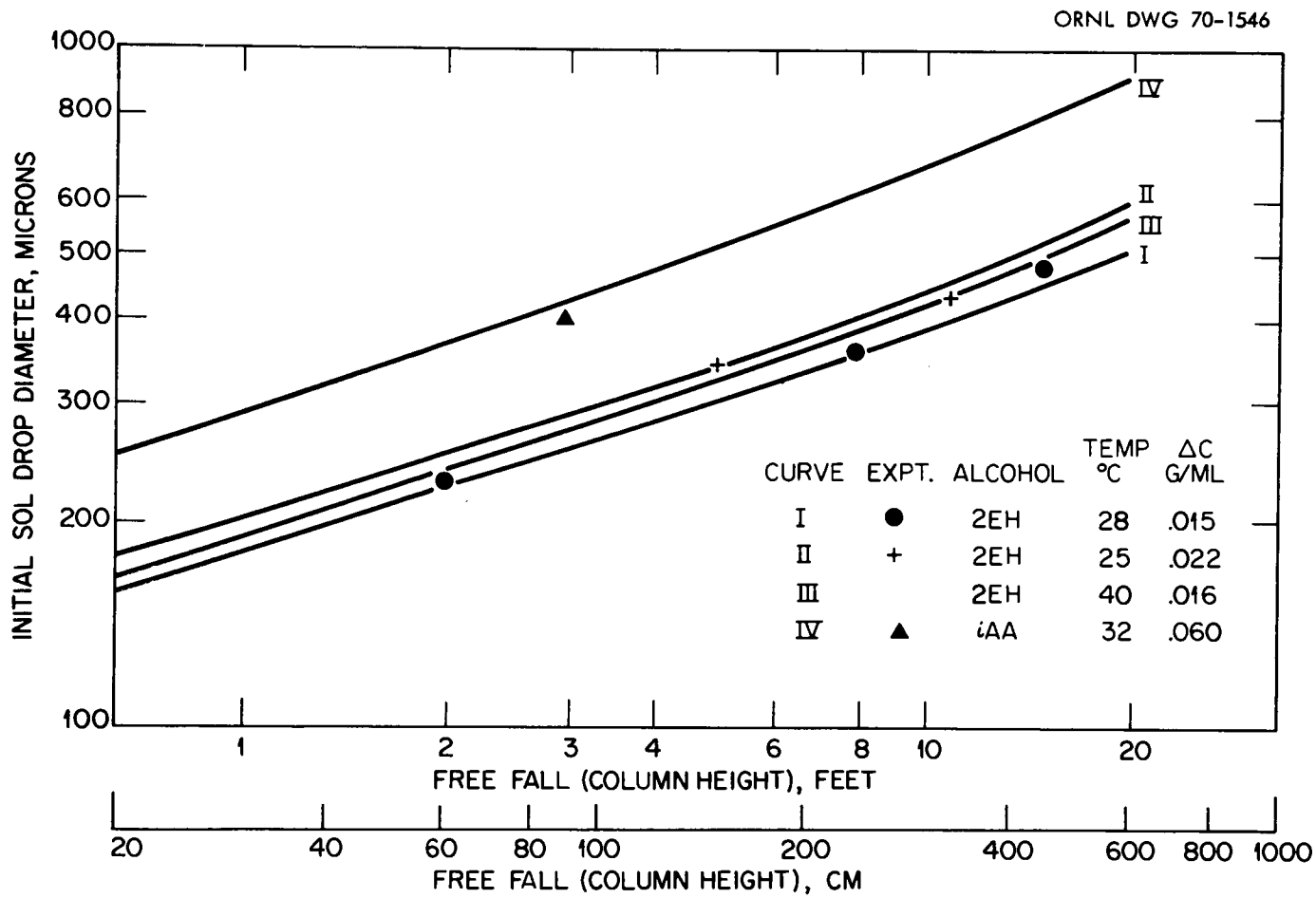

Fig. 1.1 Initial Sol Droplet Diameter vs Column Height Required for Gelation.

conditions were those checked experimentally in 5 and 11 foot columns with virgin 2EH. Curve III shows results for 2EH above room temperature. Curve IV conditions are those for recycle of iAA.

These results, along with other experimental results, show that sol droplets initially of 300 μ are easily gelled in non-fluidized columns while 500 or 600 microns are difficult. When 2EH is used, column heights must be increased over those heights required for columns using iAA. Increases of 4 to 6 feet are needed to form 300 μ diam spheres, and of 20 to 30 feet to form 600 μ diam spheres. For iAA, the difficulty is that sol droplets initially larger than 300 μ are almost certain to distort or crack as a result of the high rate of mass transfer. The corresponding sizes of fired product for 1 M and 3 M sols are 90 and 125 μ from 300 μ sol droplets and 145 and 210 μ from 500 μ sol droplets.

1.2 Preparation of Test Materials

(J. R. Parrott, F. L. Daley, R. J. Shannon)

In order to fabricate the eight recycle test elements (RTEs) that will be irradiated in the Peach Bottom Reactor we need several kilograms of ThO_2-UO_2 microspheres containing ^{235}U and thorium in the Th/U ratio of 2/1. Specifications call for the microspheres to be 350 ± 100 μ in diam with a BISO coating of pyrolytic carbon.

The preparation of this material continued through the month. Two batches (1000 g Th+^{235}U each) of sol were produced in the extraction equipment[1] using the standard flowsheet for extracting nitrate from previously mixed solution with Amberlite LA-2 amine in an n-paraffin diluent.

The sol was transferred batch-wise to the remotely-operated facility[2] in cell 4, Building 3019. As a result of development work reported in last month's report, good operation was attained. The important variables

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[1]Snider, J. W., The Design of Engineering-Scale Solex Equipment, ORNL-4256 (April 1969).

[2]Chem. Technol. Ann. Progr. Rept. May 31, 1969, ORNL-4422, pp. 185-6 (October 1969).

during sphere forming using 2-ethyl-1-hexanol as the drying agent appear to be the HNO_3 concentration and the water content. An analyzer which continuously determines water content as a function of the boiling temperature of the 2-ethyl-1-hexanol was installed and performed well. The water content was maintained at 1.6 volume percent. In addition, a pH meter was installed in the alcohol feed line and HNO_3 was added as necessary to hold the pH between 4.0 and 4.5. The only surfactant used was 0.2 v/o Ethomeen S/15. Product from the last run was screened and rounded, giving a yield of 64% (based on the feed) round spheres in the right size range.

2.0 Fueled Graphite Fabrication Development

(F. J. Furman, W. H. Pechin, C. B. Pollock, J. M. Robbins, and J. D. Sease)

We are developing processes and equipment for the refabrication of HTGR fuel as detailed in the National HTGR Recycle Development Program Plan. The fuel consists of microspheres of thorium and/or uranium as the oxide or carbide, coated with multiple layers of pyrolytic carbon and silicon carbide. These particles are loaded into hexagonal graphite logs which contain both fuel and coolant holes.

Our work is divided into particle coating, particle handling and inspection, particle blending, fuel element assembly, and fuel element inspection. This month we continued our work on particle handling and coating of fuel material to be used in the Recycle Test Elements (RTE's) being prepared for irradiation in the Peach Bottom Reactor.

2.1 Particle Handling

Prior to any coating operation the microspheres must be sintered to high density and processed to remove over- and undersized and nonspherical particles. We have set up a furnace to sinter to 1450°C in air and then reduce in Ar–4% H_2 all the mixed oxide particles which will be used in the RTE's. When we have finished sintering the material we will process it in a glove box by screening, shape separating, sampling, and batching for coating. The glove box is necessary because the material is contaminated by < 10 ppm ^{238}Pu. We have completed the sintering and processing of the (Th–20% U)O_2 particles and half of the (Th–33% U)O_2 particles required for the RTE's.

2.2 Particle Coating

We are coating four types of microspheres in the prototype remotely operated coating furnace for the RTE's: ThO_2, (Th–20% U)O_2, highly spherical (Th–33% U)O_2, and nonspherical (Th–33% U)O_2. In a laboratory-scale coater we are coating UO_2 and some additional ThO_2. We have successfully coated 2 kg of ThO_2. The analysis of this material

is presented in the next section of this report. The 2.4 kg of (Th–20% U)O_2 required for the RTE's have been coated and the results will be reported next month. This following month we plan to coat both types of (Th–33% U)O_2 particles.

The type of coating applied to the particles is the "BISO" coating, a low density pyrolytic carbon inner layer and a high density isotropic pyrolytic carbon outer layer. The coating parameters which are being used are shown in Table 1.

The (Th,U)O_2 particles are contaminated with < 10 ppm ^{238}Pu. The particles are adequately contained during the coating operation since they are pneumatically loaded from a glove box into the furnace and after coating, pneumatically transferred to another glove box through a sealed system. However, the contaminated furnace must be cleaned out and the graphite cones changed periodically. Contamination could be released during this operation. To prevent this from being a hazard the large plexiglas enclosure in which the furnace is contained was sealed and absolute filters placed on the air inlet and exhaust. The enclosure is maintained at 1 in. of water negative pressure with respect to the surrounding laboratory. The air flow through the chamber is 1000 cfm. A plastic airlock was installed over the enclosure entrance. Personnel are required to wear protective clothes and gas masks when they work in the enclosure.

To clean the furnace the top assembly with cone attached must be removed from the furnace shell. As it is removed it is drawn into a plastic bag. The bag and assembly are immediately transferred to a glove box where the cone is removed from the assembly. The assembly is cleaned, a new cone installed, and the assembly returned to the furnace. Meanwhile, the furnace shell is vacuumed and the "O" ring seals checked. Although we have performed this operation three times after coating runs with contaminated material, no contamination has been detected outside the furnace.

Table 1. Coating Parameters for RTE Fuel

Material	Diam (μm)	Initial Bed Size (g)	Temp[a] (°C)	Hydrocarbon Type	Flux[b]	Diluent Type	Flux[b]	Coating Rate[c] (μm)	Density[c] (g/cm^3)	Anisotropy[c] (BAF)
ThO_2	400	~ 1200	1100	Acetylene	2	He	0.8	7.6	1.30	——
Buffer coated 400μ ThO_2	500	~ 650	1350	Propylene	2	—	—	7.5	1.90	< 1.1
$(Th,U)O_2$	350	~ 1200	1100	Acetylene	2	He	0.8	9.6	1.20	——
Buffer coated 350μ $(Th,U)O_2$	510	~ 700	1420	Propylene	2	—	—	6.9	1.88	< 1.1

[a]Measured optically on cone wall 2 in. above inlet.

[b]Units are cm^3/min of gas per cm^2 of particle surface area.

[c]Estimated results.

2.3 Analytical Results on Material for Recycle Test Elements

Sufficient data are available to calculate the particle characteristics for the ThO_2 material prepared for the Recycle Test Elements. The various diameters for each batch of particles are listed in Table 2. The coating properties are listed in Table 3. The measured diameters are from carbon weight and particle density values using the equation

$$D_p^3 = \frac{\rho_k D_k^3 (Wc + 1)}{\rho_p}$$

where D_p = particle diameter, cm
D_k = kernel diameter, cm
ρ_p = particle density, g/cm^3
ρ_k = kernel density, g/cm^3
Wc = carbon weight g/g kernel

The buffer and isotropic coating densities were calculated from mercury displacement density of the coated particles and carbon weight. In addition, the density of the isotropic coating was measured directly in a gradient density column.

The agreement between calculated and measured diameters is excellent (0.4 to 0.8%) for the buffer coatings. For the final diameters, however, the agreement (0.2 to 2.5%) is not as good as we feel it should be. The reason for this is not known; but at this time we have more confidence in the calculated diameters which are based on larger samples and therefore should be less sensitive to sampling errors.

With one exception, the isotropic density calculated from mercury displacement agrees quite well with the measured densities when the expected bias of the gradient density method for this material is taken into account. The exception is run 49, for which the mercury displacement value is higher than the gradient density. This result would be expected if infiltration of the buffer coat occurred causing an increased buffer density. This explanation is supported by the

Table 2. Particle Diameters for ThO_2 Coated Materials for RTE's

Run Number	Kernel Diameter		Buffer Diameter			Final Diameter		
	Average (μm)	Standard Deviation	Measured Average (μm)	Standard Deviation	Calculated Average (μm)	Measured Average (μm)	Standard Deviation	Calculated Average (μm)
47/44	409.7	10.58	520.8	22.88	518.8	671.2	39.01	672.5
48/44	409.7	10.58	520.8	22.88	518.8	645.2	20.18	661.4
59/45	409.7	10.58	502.8	26.17	498.9	621.1	23.25	635.1
50/46	390.9	14.21	493.7	23.04	491.4	635.5	31.51	623.2
51/46	390.9	14.21	493.7	23.04	491.4	633.3	20.67	639.2

Table 3. Coating Properties of ThO_2 for RTE's

Run Number	Buffer Coating				Isotropic Coating			
	Thickness From Measured Diameters (μm)	Standard Deviation	Thickness From Calculated Diameters (μm)	Density From Mercury Displacement (g/cm^3)	Thickness From Measured Diameters (μm)	Thickness From Calculated Diameters (μm)	Density From Mercury Displacement (g/cm^3)	Density From Gradient Column[a] (g/cm^3)
47/44	55.6	10.1	54.6	1.26	75.2	76.9	1.905	1.935
48/44	55.6	10.1	54.6	1.26	62.2	71.3	1.914	1.940
49/45	46.6	12.0	44.6	1.23	59.2	68.1	1.949	1.890[b]
50/46	51.4	9.1	50.3	1.33	70.9	65.9	1.845	1.910
51/46	51.4	9.1	50.3	1.33	69.8	73.9	1.830	1.915

[a]These results are expected to be biased approximately 0.05 g/cm^3 high with respect to mercury displacement values.

[b]This value indicates mercury displacement density should be about 1.85. The value of 1.949 actually found is interpreted to mean that there was infiltration of the buffer coating.

metallography. Many of the particles in the polished section have buffer coatings whose density is almost the same as the isotropic coating density. Because of the infiltration, the material from run 49 has been rejected.

2.4 Development of Bonded Beds of Coated Particles for HTGR Fuel Elements

This report period has been devoted almost entirely to the scale-up of our facilities and the production of fuel for the Recycle Test Elements (RTE's). The RTE's are scheduled to be operated in the Peach Bottom Reactor starting in July of 1970. Our portions of the RTE's are being fabricated by bonding the mixed beds of various coated particles into sticks (OD = 0.49 in.; ℓ = 2.14 in.) with 15V pitch and 35 wt % Poco grade graphite flour. The final product is heat treated at 1800°C prior to shipping. Table 4 shows the requirement of RTE's from ORNL.

Table 4. ORNL's Fuel Stick Fabrication Requirements for Recycle Test Elements

Type Particles	Number of 2.14-in.-long Sticks Needed			
	1st Group	2nd Group	3rd Group	Total
$(4.2\ Th/U)O_2$	72	96	—	168
$(2\ Th/U)O_2 + ThO_2$	72	96	—	168
$(2\ Th/U)O_2 + ThC_2$	168	144	—	312
$(2\ Th/U)O_2 + ThC_2$	—	—	288	288
$UO_2 + ThO_2$	120	96	—	216
Total	432	432	288	1152

A total of 20 extra sticks will be required for control and test samples. The first group is for the RTE's 5–8 (the three-year elements), second group is for RTE's 1–4, and the third group is for FBTE. The first and third groups will contain 0.312 g U/in. of fuel stick. In the

second group, one half of each type will contain 0.312 g U/in. and one half will contain 0.381 g U/in. The first group is scheduled for completion about April 24, 1970. The remainder should be finished about the middle of June.

This job has increased from an originally requested 150 sticks to the present 1170. Several problems have been encountered, some because of the scaled-up operations and some that we are just now recognizing. One of the first difficulties encountered was simply that some equipment and procedures that are adequate for a small laboratory operation are not suitable for production operations. For instance, the threads on the screw-driven piston in our injection device had to be improved in order to increase the longevity of the device. Also, it was recognized that residual bonding material from the previous injection did not need to be removed from the mold end-caps each time. As the mold was reheated the residual bonding material remelted, thus causing no trouble. This saved much time in mold cleaning operations. An early problem was encountered when the bonding material containing 40 wt % Poco AXM graphite flour in 15V pitch could not be injected through the 2-in. fuel bed made from a blend of UO_2 and ThO_2 coated particles. The reason was determined to be a high volume fuel loading obtained with the two-sized particle system. Most previous work has been done with a single particle size or two particles fairly close to the same size, with a resultant particle volume loading of about 62 to 65%. The coated UO_2 particles used for the RTE's are 250 to 550 μ; the coated ThO_2 particles are 600 to 1000 μ. By weight there are about 1 1/2 parts coated ThO_2 particles to 1 part coated UO_2 particles in the RTE blend. With this blend of these two sizes of particles we get volume loadings as high as 70%, depending on regularity or irregularity in shape of the various particle batches. By trial and error we determined that we could use a mix containing 35 wt % of the graphite flour instead of 40 wt %.

Another problem with the bonding mix developed when we ran out of the first batch of graphite flour and started on another batch. An identical mix made with the second batch of graphite flour would inject

only about 1/2 in. into the bed of particles. We suspected that the problem was a different particle size distribution in the second batch of graphite flour than was in the first. We found that the first batch of graphite had been ground to pass through a 325-mesh screen and the second batch had been ground to pass a 200-mesh screen. Therefore, even though both flours were screened through a 40-μ precision screen prior to use, there were more of the fines or smaller particles in the first batch of flour than there were in the second. Also, since much more work is required to grind graphite to 325-mesh flour than to 200-mesh flour, one would expect the particle shapes to be different, perhaps elongated and oriented more in the finer flour. We solved the problem by screening and using the −27-μ particles from the second batch of flour. However, this is wasteful of graphite flour and it behooves us to do the necessary development work to be able to prepare a graphite flour with more control over the particle size distribution.

One problem that we have been unable to eliminate is the forming of a cap on one end of the fuel stick. This cap is formed on the injection end of the stick when the particles settle slightly during injection, leaving a thin layer or cap of bonding material without particles on the end of the stick. Although the particles are vibrated into the molds, the injection of bonding material through the restrained bed of particles must afford enough pressure, local turbulence, and lubrication to enable the particles to rearrange themselves into a smaller volume, thus leaving the void space to be filled with bonding material only.

A blending problem was encountered in making the UO_2 + ThO_2 sticks. This problem was solved by feeding the particles simultaneously from the vibratory feeders into a double funnel leading into the mold. The double funnel arrangement enables us to feed the particles directly into the bottom of the top funnel to prevent bouncing of the particles. The stem of the top funnel passes through a hole in a lid into the second funnel. The particles are directed against the angled wall of the second funnel, causing bouncing, swirling, and mixing of the particles. The lid on the second funnel contains the bouncing particles and keeps

them from being scattered. Radiographs show this method of mixing to give a consistently good blend. Unfortunately, many of the UO_2 + ThO_2 sticks were produced before this problem of blending was discovered. Consequently, several sticks will have to be rejected and others with less-than-ideal homogeneity will have to be accepted if we are to meet our schedule.

We have essentially completed the UO_2 + ThO_2 specimens for Group 1 (see Table 4). We expect to make the first shipment of these sticks to Gulf General Atomic by April 20. We have received the (4.2 Th/U)O_2 particles and should be able to fabricate the 72 sticks required in Group 1 by mid-April. Some of the coated (2 Th/U)O_2 particles are expected by April 15. That will leave about 1 1/2 weeks to produce from these particles the 240 sticks required in Group 1.

III. MATERIAL IRRADIATION

(A. L. Lotts, T. N. Washburn, J. D. Sease, and J. H. Coobs)

1.0 HTGR Recycle Fuels Irradiation

(T. N. Washburn, R. B. Fitts, and A. R. Olsen)

The irradiation tests on the HTGR recycle program have two main objectives: (1) to provide irradiated fuel for head-end process studies, and (2) to provide irradiation proof tests of the products of coated particle process development for the Thorium-Uranium Recycle Facility. The test conditions of interest include fuel temperatures between 600 and 1300°C, burnup to 20% FIMA in the (Th,U)O_2 particles, and fast fluence exposures up to 8×10^{21} neutrons/cm^2.

The first two stages in this program are being implemented this year. They are: (1) accelerated burnup rate capsule irradiations, and (2) eight test fuel elements to be irradiated in the Peach Bottom Reactor.

1.1 Capsule Irradiation

Detailed physics calculations for a fuel bed loaded with equal numbers of 4.2:1 $(Th,U)O_2$ and ThO_2 coated particles were completed. These calculations together with currently reported peak thermal flux of 5.67×10^{14} neutrons/cm^2-sec for an ETR-J8 position yield a calculated peak linear heat rate of 32 kw/ft. This heat generation rate is too high since the surface-to-center temperature differential, even for a bonded bed, would be greater than 900°C. Reducing the uranium content of the fuel bed by adding additional ThO_2 coated particles will lower the heat generation rate, but it will also eliminate the possibility of doing duplicate reprocessing tests. The necessity for doing duplicate tests is currently being reconsidered. We are also investigating the possibility of adding a thermal neutron absorber to the capsule to lower the thermal flux in the fuel without seriously disturbing the fast flux. A third possiblity is the use of a lower flux position in the ETR. This third possibility is the least desirable since the integrated fast fluence for coating damage will be decreased by the lower flux position in the scheduled irradiation time for these tests.

A final decision on these capsule tests will be made during the next month.

1.2 Large Scale Irradiation

Eight Recycle Test Elements (RTE's) will be operated in the Peach Bottom Reactor starting in June 1970. These fuel elements will contain samples of fuels proposed for use in 1100 Mw(e) HTGR's. The preparation of the ORNL portion of the fuels for RTE's is behind schedule, although not seriously, and is progressing well as outlined below and described in detail as indicated.

The ORNL specifications for kernels, coatings, and fuel sticks are being put in final form. The data sheets required to document the quality of the fuel have been prepared. Progress on defining the analytical techniques to be employed are reported under 2.3 of this report.

The coating of the ThO_2, 2/1 $(Th/U)O_2$, and 4/1 $(Th,U)O_2$ kernels (three of the four types to be coated) is described in Section 2.0 of this report, "Fueled Graphite Fabrication Development." The ThO_2 and 4/1 $(Th/U)O_2$ coating is complete. The coating of the UO_2 kernels is complete. The progress on bonded beds for the RTE's is given in Section 4.4.

During the last month an extensive final review of the RTE program was conducted by ORNL, GGA, and AEC representatives. The principal results and conclusions of this review are outlined below.

1. The emphasis in these tests was changed from loose to bonded beds as a result of recent irradiation test results at GGA and ORNL. The final RTE loading plan is presented in Tables 5 and 6.

2. The schedule for preparation of these test elements for installation in the Peach Bottom Reactor is very tight. Therefore, both ORNL and GGA will prepare all of the fuel and fuel elements for the three-year RTE's first. If the one- and two-year elements are not ready for the June 1970 Peach Bottom startings they can be loaded at a scheduled shutdown after 150 full power days of operation. The ORNL production schedule is attached as Table 7.

3. ORNL is to provide 2/1 $(Th/U)O_2$ coated particle fuel for one Fuel Bed Test Element. Two types of this fuel have been prepared—one containing a typical sol-gel spherical kernel and another containing roughly ellipsoidal kernels (potatoes). A decision was made to load the RTE's primarily with the spherical material and to use "potatoes" in the FBTE. The fabrication of this fuel will follow the preparation of the RTE fuel.

4. Gulf General Atomic cannot handle the mixed Th/U-containing particles in their fabrication facility because of the slight ^{238}Pu contamination of these particles. Therefore, ORNL will have to bond all fuel sticks containing the mixed oxide particles. The ORNL coated particle and fuel stick fabrication requirements are outlined in Tables 8 and 9.

Table 5

RTE Loading Combinations[a]

Position	℄ Temp Range (°F)	First Discharge		Second Discharge		Final Discharge			
		RTE 1 (Quartered)	RTE 2	RTE 3 (Quartered)	RTE 4	RTE 5 (Quartered)	RTE 6	RTE 7 (Quartered)	RTE 8
6 (Top of Reactor)	2050–1950	bdei	f	bdei	e*	bdei	g	acfg	f
5	2250–2050	bdeh	a*	bdeh	d	bdeh	f	acfg	i
4 (Max Flux)	2250–2300	acfg	e	acfg	f	acfg	f	bdeh	d
3 (Max Flux)	2050–2250	acfg	f*	acfg	a*	acfg	d	bdhi	e
2	1650–2050	acfg	d	acfg	f*	acfg	f	bdhi	i
1 (Bottom of Reactor)	1050–1650	acfg	e*	acfg	e	acfg	e	bdhi	f*

[a]See Table 6.

*Blended beds, all others bonded.

Table 6

RTE Loading Combinations

Combination	Fissile	Fertile
a	(4.2 Th,U)O_2	None
b	UO_2	ThC_2
c	(2.0 Th,U)O_2	ThO_2
d	(2.0 Th,U)O_2	ThC_2
e	UC_2	ThC_2
f	UC_2 TRISO	ThC_2
g	UO_2	ThO_2
h	UC_2	ThO_2
i	UC_2 TRISO	ThC_2 TRISO

All particles BISO unless otherwise stated.

Table 7

RTE Fuel Production Schedule

Date	March				April			
	6	13	20	27	3	10	17	24
Finish coating	UO_2	ThO_2	$(4\text{-}Th/U)O_2$	$(2\text{-}Th/U)O_2$				
Ship loose		UO_2	ThO_2	$(4\text{-}Th/U)O_2$	$(2\text{-}Th/U)O_2$			
Ship sticks				UO_2 + ThO_2			$(4\text{-}Th/U)O_2$ + ThO_2	$(2\text{-}Th/U)O_2$ + ThO_2

Table 8. Summary Tables for ORNL RTE's and FBTE Loading

A. Particle Fabrication

Particle	Amount to Coat,[a] g		Total Amount to Ship to GGA, g			
			Loose[b]		In Sticks[c]	
	Kernels	Finished Particles	Kernels in Coated Particles	Coated Particles	Kernels in Sticks	Coated Particles
ThO_2	2660	4165	442	680	1741	2681
UO_2	390	2250	135	780	193	1118
$(4.2\ Th/U)O_2$	1445	3690	498	1270	706	1800
$(2\ Th/U)O_2$ spheres	1545	3885	—	—	1189	3007
$(2\ Th/U)O_2$ potatoes	865	2185	—	—	659	1667
ThC_2	NA	4980[d]	NA	NA	2381	3786

[a]Includes 10% excess over projected project needs (32% over minimum for loading).

[b]Includes 20% excess for losses and sampling plus 50 g characterization sample.

[c]Includes 2 extra 2.14-in. sticks of each type for examination samples.

[d]Amount required from GGA.

Table 9. Summary Tables for ORNL RTE's and FBTE Loading contd.

B. Stick Fabrication[a]

Stick Type	Particle Content	Inches of Good Stick to Make[b]			
		First Group	Second Group	Third Group	Total
a	$(4.2\ Th/U)O_2$	153.84	205.12	———	358.96
c	$(2\ Th/U)O_2$ + ThO_2	153.84	205.12	———	358.96
d	$(2\ Th/U)O_2$ spheres + ThC_2	358.96	307.68	———	666.64
d	$(2\ Th/U)O_2$ potatoes + ThC_2	———	———	615.36	615.36
g	UO_2 + ThO_2	256.40	205.12	———	461.52
TOTAL	———	923.04	923.04	615.36	2461.44

[a]First Group is for RTE's 5–8; Second Group is for RTE's 1–4; Third Group is for FBTE. The First Group is to contain 0.312 g U per in. In the Second Group 1/2 of each type contains 0.312 g U per in. and 1/2 contains 0.381 g U per in. The Third Group all contain 0.312 g U per in.

[b]Make 4 extra sticks (8.56 in.) of each loading for ORNL and GGA samples.

IV. LWBR SUPPORT PROGRAM

J. R. Parrott, R. E. Brooksbank

ORNL participation in the Light Water Breeder Reactor Program, under contract with Bettis Atomic Power Laboratory, involves (1) the receipt and storage of approximately 640 kg of ^{233}U (400 kg as UNH and 240 kg as UO_2), (2) the purification of this material in small batches (~20 kg ^{233}U per week) to remove ^{232}U daughters, (3) the conversion of this UNH to ceramic-grade UO_2 powder at the rate of about 20 kg of ^{233}U per week, (4) the packaging and shipping of the UO_2 to BAPL for blending with thoria and pressing into pellets, and (5) the recovery of 175 to 225 kg of ^{233}U from an estimated 6 to 7 tons of urania-thoria scrap from the Bettis operation.

This program will require additional storage facilities for both oxide and solution, a new dissolver for recovering ^{233}U from the thoria-urania scrap, modifications to the present purification system, and a complete oxide conversion line. The facility is presently scheduled to begin production in October, 1971. Since this is the first contribution to the Thorium Fuel Cycle Monthly Report, an attempt will be made to bring the reader up to date with subsequent reports containing only the progress made during the particular month.

1. STORAGE FACILITIES

W. R. Whitson, G. E. Pierce, R. G. Nicol

1.1 Solution Storage

Five additional storage tanks, each with a capacity of 100 kg of ^{233}U at a concentration of 250 g/liter, will be required. Each tank will contain borosilicate-glass raschig rings (neutron absorbers).

Fabrication of the tanks by the ORNL shops is complete. The instrument transmitter modules have been installed in the penthouse, and the pneumatic tubing between the transmitters and control room is 90% complete. The CPFF contractor began installation of the tanks.

Analyses of samples of the borosilicate-glass raschig rings showed the rings to meet the specifications outlined in Nuclear Engineering Bulletin, Vol. 303 by the ANS-8 Standards Subcommittee.

1.2 Storage of Solids

Additional dry storage capacity is required to accommodate $^{233}UO_3$ received from the Savannah River Plant. Nine storage wells (or holes), each 4 in. in diameter and 15 ft deep, have been drilled in the shielding walls between cells 2 and 3. This work, which is being done by the CPFF contractor, is 90% complete. The scheduled completion date is April 10, 1970.

2. PURIFICATION SYSTEM

2.1 Process Development (R. H. Rainey)

In order to meet the specifications with regard to thorium concentration (500 ppm) in the $^{233}UO_2$, ORNL will be required to install a thorium removal step. Laboratory work to date, utilizing dissolved ^{233}U oxide from Savannah River as feed, indicates that a high pressure cation exchange column containing 200- to 400-mesh Dowex 50 (12% cross-linked) resin is capable of decreasing the activity in the product to less than 1% of the radiation intensity of ^{232}U in equilibrium with its daughters. The capacity was determined to be 5 kg of ^{233}U per liter of resin at a feed rate of 5 kg of ^{233}U per hour.

This system will be installed as a product-polishing step after solvent extraction; however, in the event the ^{233}U as received from the production plants meets specifications for ionic impurities, the solvent extraction system could be bypassed.

Resin reconditioning studies have shown that 1.5 M HNO_3 removes essentially all of the uranium and ^{212}Pb from the resin. The ^{224}Ra is readily removed with more concentrated acid. The removal of the thorium from the resin with nitric acid is much more difficult, and the amount removed has been difficult to determine accurately.

Optimum dissolution procedures for the ^{233}U oxide received from the production plants are being investigated. In order to feed the solution directly to the ion exchange system it is necessary to maintain the ^{233}U at approximately 100 g/liter and the HNO_3 concentration at 0.1 M for maximum thorium removal. Data indicate that the oxide dissolves within 2 hr in a stoichiometric volume of warm ($\sim$70°C) 4 M HNO_3. This indicates the need to dissolve to a high (400 g/liter) uranium concentration followed by dilution to the required ion exchange feed concentration.

2.2 Equipment Modifications (F. E. Harrington, W. R. Whitson)

The existing ^{233}U solvent extraction system will require modification and additions to ensure reliability and to meet specifications for feed to the oxide line. These changes and additions are as follows:

(1) addition of an efficient UO_2-ThO_2 scrap dissolver system;
(2) modification of existing equipment to provide a solvent wash system;
(3) replacement of worn instruments and pumps;
(4) addition of an efficient thorium removal system; and
(5) installation of tanks and piping to connect the purification system to the oxide line.

Table 1 summarizes the current status of the required design drawings.

Drawings 001 through 003 are related to solids storage installations reported in Sect. 1.2.

Drawings 004-008, 020, and 021 are flowsheets. Approval of these flowsheets must await completion of laboratory development work.

Drawings 009-012 are concerned with the scrap dissolver system. These drawings are in the process of being approved. The additional flowsheet drawings required for installation (piping) will be issued early in April.

The ion exchange system for removing thorium is covered by drawings 013-017, 020, and 021. The remaining item to be designed for this system is the waste resin disposal can (X 18 on engineering flowsheet 020D).

A formal Nuclear Safety Review is in progress for increasing the capacity of the system used for dissolving and adjusting UO_3 obtained from Savannah River. Drawings 018, 019, and 022 were prepared for this review.

The last two listed drawings refer to the tank and piping for the transfer of purified solutions to the UO_2 line, and to the removal of a condenser and a liquid vapor separator from the penthouse and subsequent relocation in cell 5 to improve operability.

Table 1. Status of Required Drawings

Drawing No.	Title	Status[a]
M-12229-CF-001-E	Solids Storage Arrangement	A
M-12229-CP-002-E	Core Drilling Details	A
M-12229-CP-003-E	Solids Storage Miscellaneous Details	R1
F-12229-CF-004-D	Chemical Flowsheet, Solvent Extraction	C
F-12229-CP-005-D	Chemical Flowsheet, Thorium Removal	C
F-12229-CP-006-E	Equipment Flowsheet, Sheet 1, Solvent Extraction	C
F-12229-CP-007-E	Equipment Flowsheet, Sheet 2, Solvent Extraction	C
F-12229-CP-008-E	Equipment Flowsheet, Sheet 3, Thorium Removal	C
M-12229-CP-009-E	Scrap Dissolver Assembly	C[b]
M-12229-CP-010-E	Scrap Dissolver Detail Sheet 1	C[b]
M-12229-CP-011-E	Scrap Dissolver Detail Sheet 2	C[b]
S-12229-CP-012-D	Scrap Dissolver Supports	C[b]
M-12229-CP-013-D	Vessels X11 and X14, Plan, Elevation, and Details	R1
S-12229-CP-014-D	Vessels X11 and X14, Support and Details	A
P-12229-CP-015-D	Vessels X11 and X14, Piping Details	A
M-12229-CP-016-D	X13 and X23, Ion Exchange Columns	A
M-12229-CP-017-D	X14 and X17, Ion Exchange Columns	A
D-34123	X14 and X17, Original Vessels	R2
F-12229-CP-018-C	S15 and R35 Equipment Flowsheet	A
P-12229-CP-019-C	R25, -26, -35, -36, -37 Piping	A
F-12229-CP-020-D	Engineering Flowsheet, IX System Sheet 1	C
F-12229-CP-021-D	Engineering Flowsheet, IX Sheet 2	C
F-12229-CP-022-D	^{233}U Dissolver Layout	A
M-12229-CP-023-D	X16 Fabrication	C
M-12229-CP-024-D	S6 Fabrication and Installation	C

[a]A = approved; R1 = revision 1 (approved); R2 = revision 2 (approved); C = check print.

[b]Approval in process.

Specifications were written for the purchase of instrumentation for the purification and the ion-exchange systems. Purchase orders will be issued upon receipt of approval from Westinghouse.

2.3 Facility Operations (R. G. Nicol, R. W. Horton, W. T. McDuffee, W. A. Shannon)

Three shipments of UO_2 powder, containing a total of 35.5 kg of ^{233}U, were received from Nuclear Fuels Services, Erwin, Tennessee, during March. Based on the shipper's analyses, this material will meet specifications as feed for the LWBR program. These analyses will be verified during dissolution and storage.

A total of 6.65 kg of ^{233}U from the above material was dissolved during the month. However, due to the nuclear safety limits applied to the dissolver, we could not attain both complete dissolution and a solution suitable as feed for the ion exchange system. This solution must be 0.1 N in HNO_3 and contain 100 g of ^{233}U per liter if maximum separation from the thorium is to be obtained.

Preliminary discussions with the ORNL Criticality Committee indicate that, with proper operating procedures, the mass limit of the oxide dissolver may be increased from 2.0 kg of 5.5 kg of ^{233}U and the final ^{233}U concentration may be increased from 150 g/liter to 400 g/liter. This solution, which will be 0.4 M in HNO_3, will then be diluted to 0.25 M HNO_3 and 250 g of ^{233}U per liter in the catch tank prior to transfer to solution storage. Final dilution to 0.1 M HNO_3 and 100 g of ^{233}U per liter will be performed immediately prior to feeding to the ion exchange system.

Dissolution will be discontinued until the modified limits and procedures are approved by the Criticality Committee. Approval is expected to be obtained in early April.

A draft of analytical requirements, including sampling frequency, analyses required, and analytical techniques, was prepared. This draft will be issued as a report after it has been edited.

3. NITRATE-TO-OXIDE CONVERSION LINE

3.1 Site Preparation (J. D. Sease, D. B. Allen, J. K. Dyer)

The UO_2 conversion line will be located in Building 3019, adjacent to the purification system. However, this area presently houses plutonium fuels fabrication equipment, which must be moved prior to the UO_2 equipment installation. The design necessary to renovate an area in Building 4508 to house the UO_2 equipment is complete. The order to the CPFF contractor to begin construction was issued on March 17. The removal of the equipment presently located in the proposed 4508 area is under way.

3.2 Flowsheet Development (J. D. Sease, C. R. Reese, J. M. Leitnaker, M. L. Smith)

Flowsheet development of the nitrate-to-oxide conversion line began in November, 1969. A series of runs, using 100 g of ^{238}U each, were made to determine the effects of various operating variables on the quality of the $^{233}UO_2$. The following flowsheet resulted from these studies:

(1) Dissolve U_3O_8 powder (as received) in 8 M HNO_3 (stoichiometric) and dilute to 100 g/liter ^{233}U; this gives a free HNO_3 concentration of 0.1 M.

(2) Circulate the uranium solution in a glass column 2-3/4 in. diam by 9-1/2 in. high.

(3) Precipitate the uranium from the solution by the addition of NH_4OH at a constant pH of 8 while recirculating at room temperature.

(4) Centrifuge the slurry to remove supernate.

(5) Dry precipitate (ammonium diuranate) in a microwave oven for 1 hr then screen through a 35-mesh sieve.

(6) Calcine dried ammonium diuranate in fused silica furnace boats placed in a batch furnace with a H_2 blanket using the following firing cycle:

(a) Load in at 200°C.

(b) 200 to 350°C at 100°C/hr.

(c) 350 to 500°C at 50°C/hr.

(d) 500 to 700°C at 100°C/hr.

(e) Soak at 700°C for 3 hrs.

(f) Cool furnace to room temperature.

(7) Stabilize the UO_2 in a dry ice bath.

The above flowsheet was used to prepare 2 kg (as metal) of $^{235}UO_2$ which was shipped to Bettis for evaluation. This material was blended by Bettis with ThO_2 in ratios of one wt % $^{235}UO_2$ in $^{235}UO_2$-ThO_2 and 9 wt % $^{235}UO_2$ in $^{235}UO_2$-ThO_2 and pressed into pellets. The fuel pellets made from this powder met LWBR fuel pellet specifications for structure and microhomogeneity.

Several revisions to the flowsheet have been made since the production of the $^{235}UO_2$. The major ones are the substitution of NH_3 for NH_4OH as the precipitating agent, the use of Ar-4% H_2 in place of pure H_2 during reduction, and the use of a vibrating "cold plate" in place of dry ice for the stabilization. Each of these revisions will result in a process more adaptable to continuous, production-type operation. The batch size will be increased to 1 kg of ^{235}U for future oxide for evaluation by Bettis.

3.3 Equipment Design (J. W. Snider)

The UO_2 conversion line will have a capacity of 22 kg of ^{233}U per week with a 5-day/week and 24-hr/day operating schedule. The line will be housed in alpha enclosures in the basement alpha lab, Bldg. 3019.

Conceptual drawings of the precipitation and centrifuging equipment, the furnace tray-loading equipment, the furnace, the furnace unloading station, the powder stabilization equipment, and the solid waste-burning equipment have been prepared. Preliminary operating flowsheets were prepared for each of the above items, as well as for the liquid waste-handling equipment. The design of the equipment for the liquid waste-handling enclosure is 50% complete. Fabrication of this enclosure is scheduled to begin May 1.

V. URANIUM-233 REPROCESSING

(J. R. Parrott, R. G. Nicol, W. A. Shannon)

ORNL serves as a national distribution center for ^{233}U. The facility, which contains a small batch leacher, a batch dissolver, and a single-cycle solvent extraction system, is capable of purifying ^{233}U at the rate of 25 kg per week. It includes storage systems for liquids and solids, with capacities of 500 and 120 kg of ^{233}U respectively.

1. Dissolution and Purification

Eight dissolvings of $^{233}UO_3$ received from Nuclear Fuels Services for the Light Water Breeder Reactor Program were made during the month. The results of this work are reported in Sect. IV, "LWBR Support Program."

Purification equipment was not operated during the month.

2. Storage and Distribution

The facility presently contains 261 kg of ^{233}U which varies in isotopic purity between 84 and 98% and ^{232}U content between 4 and 250 ppm. A total of 35,500 g of ^{233}U was received and 96 g of ^{233}U was shipped during the month.

We also have a facility (TRUST-Thorium Reactor Uranium Storage Tank) in which we store 1047 kg of highly enriched uranium (76.5% ^{235}U, 9.7% ^{233}U) in the form of a uranyl nitrate solution. This material is the uranium product from the Indian Point reactor fuel, which was purified by solvent extraction at the Nuclear Fuels Services Plant. The solution will be stored indefinitely since the ^{232}U content (120 ppm) prohibits its direct refabrication into fuel elements, and the low ^{233}U content makes it of little interest for reactors demonstrating the thorium fuel cycle.

The new vessel off-gas line for storage tank P-25 was placed in operation. Sparging of the solution in the tank (6 hours) produced a high radiation background at the off-gas filter (above the sample gallery and south of the penthouse). Other parts of the system showed only slight build-up of radiation. Before sparging for extended periods of time, a lead shield will be placed around the filter.

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