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THORIUM FUEL CYCLE DEVELOPMENT PROGRESS REPORT

January 1970

No. 6

Compiled by

The Staff of the Chemical Technology

and

Metals and Ceramics Divisions

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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

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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. URANIUM-233 REPROCESSING

R. E. Brooksbank, J. R. Parrott

JANUARY 1970

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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 forms.

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 pilot plant for fuel recycle. The CPDL is for engineering development studies leading to design of the pilot plant equipment.

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 the solvent extraction process. Small samples of irradiated and unirradiated fuel are processed 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 or fixed bed burners. The mechanical and burn-leach engineering development work is carried out using full-scale unirradiated fuel, 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)

Evaluation of the data from the crush-burn-leach experiment on Dragon fuel is being carried out as time permits.

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 present general approach involves the mechanical dissection by sawing of full-sized graphitic blocks containing fuel sticks followed by comminution to size fractions suitable for the downstream burn-leach process. However, recent decisions leading to the possible use of loose fuel particles rather than fuel sticks may alter this approach quite drastically in future work.

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 $\pm$ 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 thorium-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 in a Column Without Fluidization

The three-foot high column and cone bottom settler-tank which has been most used for preparing spheres when fluidization was not employed was operated to prepare ThO_2 spheres of 10 to 15 micron mean diameter. This ThO_2 product was dried and fired with less sticking during firing than was experienced with UO_2 spheres of the same mean diameter.

A new system was installed for the preparation of gel spheres without fluidization (Fig. 1). The disperser forms sol droplets in the

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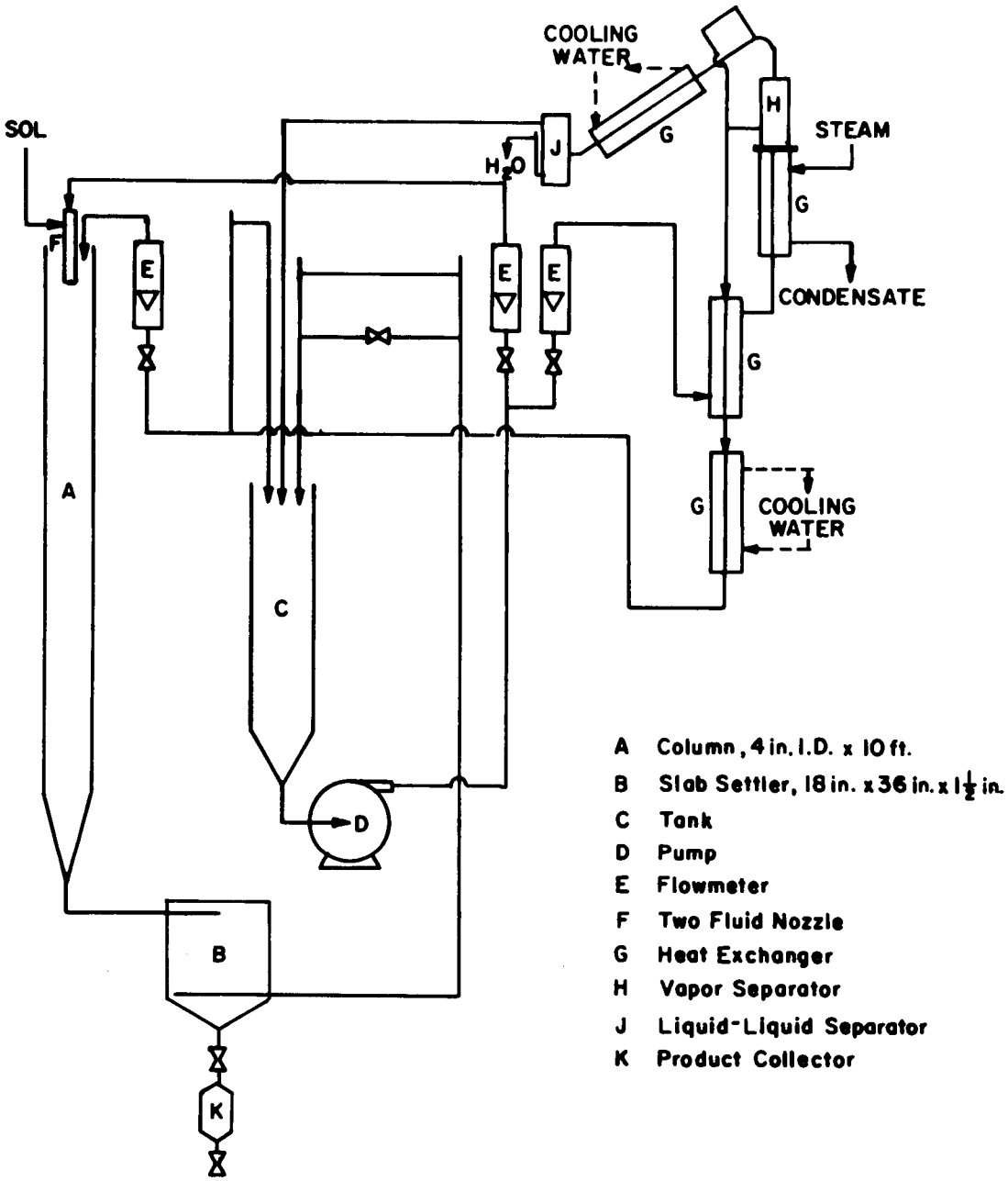

Fig. 1. Schema of Unfluidized Column for Ultra-Fine Microsphere Preparation.

top of a 4-in. ID glass column. Water is extracted to form gel spheres as the sol droplets and the organic liquid flow cocurrently down the 10-ft length of column. The organic liquid has been either 2-ethyl-1-hexanol (2EH) or isoamyl alcohol (iAA). The sol droplets or gel spheres settle through the alcohol and so have a shorter average residence time than the alcohol. Water is removed from the alcohol in a single-stage distillation, and the dry alcohol is returned to the column. The gel spheres are separated in an inclined slab settler and collected in a combination product receiver and gel sphere dryer. This receiver is valved off from the system, and the gel spheres are dried batchwise. The system was operated to form UO_2 and UO_2-C gel spheres using 2EH and UO_2 gel spheres using iAA. The maximum sol droplet size which can be gelled in the ten-foot height of 2EH appears to be about 400 microns, in agreement with previous results. UO_2 sol droplets of 350 microns in iAA gave gel particles with a dimple distortion. Sol droplets as large as 500 microns gave dry gel spheres using about 60 v/o 2EH--40 v/o iAA.

1.2 Preparation of Test Materials

(J. R. Parrott, F. L. Daley, P. A. Haas, R. J. Shannon)

The sol-forming equipment in the Solex Development Laboratory and the Cell 4 Microsphere Facility were operated during the month to make $^{235}UO_2$-ThO_2 microspheres containing a thorium-to-uranium ratio of 2 to 1. This material (350 $\pm$ 100 μ in diam.) will be used in the fabrication of 8 recycle test elements which will be irradiated starting in the fall of 1970 in the Peach Bottom Reactor as a part of the National HTGR Recycle Program. A Th/U ratio of 2/1 is lower than we have had any large-scale experience with, and we have reason to believe its preparation to be difficult by solvent extraction.

Two runs (UX-3 and UX-4) were made during the month. The sols were produced by mixing nitrate solutions of thorium and uranium and extracting the nitrate with an amine (Amberlite LA-2 in n-paraffin diluent) using the standard flowsheet which was successfully used to prepare materials having a Th/U ratio of 3/1 and 4/1. The first sol prepared (UX-3) was concentrated to 1.37 M in total metal and contained a NO_3^-/metal ratio

of 0.124. The second sol was concentrated to 1.3 M in metal and contained a NO_3^-/metal ratio of 0.107. Each run contained approximately 1 kg of total metal.

An attempt to form microspheres from the UX-3 sol using 2-ethyl-1-hexanol (2EH) which had been used previously, was only partially successful. The yield of good spheres was estimated to be between 30 and 40% with cracking the prime contributor to rejection of the material. In an attempt to increase the yield, the 2EH was transferred out of the system and virgin 2EH was added. This solution was adjusted to 0.5% water, 0.008 N HNO_3, and 0.05 v/o ethomeen. Efforts to dry the last half of UX-3 sol with this agent were even less successful; cracking became more severe and clustering in the column was observed. Previous experience with sols having a higher Th/U ratio indicated that acidity of the 2EH should be increased. However, an increase of HNO_3 content of the 2EH to 0.010 N did little to help, and the addition of Pluronic L-92 surfactant proved of little value.

The second sphere-forming run (UX-4) was started using the same 2-ethyl-1-hexanol with the acid increased to 0.014 N. This additional acid cleared up much of the clustering and breaking problem; however, the dried gel spheres were dimpled. This problem was attributed to traces of Span 80 remaining in the equipment from the previous program. The system was flushed exhaustively and new 2-ethyl-1-hexanol was added. This solution was adjusted to 1% Pluronic L-92, 0.014 N HNO_3, 0.5% H_2O, 0.05% Ethomeen, and 5% octanol. This drying agent was used during the latter part of Run UX-4 and resulted in about a 50% yield of good microspheres. An additional run will be made early next month in an effort to improve the yields.

In addition to the above material, 300 g of $^{235}UO_2$ microspheres with diameters of 100 $\pm$ 30 microns are needed for the recycle test elements. Two 300-g batches of UO_2 sol were produced by the precipitation technique and the first was formed into satisfactory microsphere product. A $^{235}UO_2$-ThO_2 product having a 4/1 Th/U ratio has already been prepared for use in fabricating these elements, as has a ThO_2 product.

2. Fueled Graphite Fabrication Development

(F. J. Furman, J. D. Sease, and R. A. Bowman)

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 to emphasize particle coating.

2.1 Particle Coating

We are working with the prototype remote coating furnace, an 8-in.-diameter cone furnace designed to be used in the TURF for coating ^{233}U-Th oxide, to both develop the equipment for its intended purpose and to obtain sufficient information about the operating parameters to coat large batches of material for irradiation testing as described in the Materials Irradiation section of the National HTGR Recycle Development Program Plan.

We are experimenting with buffer coating using acetylene and helium. Nine runs have been made. After comparing our data with that generated using the 5-in.-diameter coater in the Coated Particle Development Laboratory (CPDL),[1] we find that we cannot go quite as high in cone temperature due to clogging of the cone inlet. Apparently the geometry of the heating element in the prototype is sufficiently different from the CPDL coater so that the temperature is higher at the base of the cone, causing premature cracking of the acetylene at high cone temperatures (1250°C or greater). However, this situation poses no problem because completely successful buffer coatings are

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[1] R. B. Pratt, J. D. Sease, W. H. Pechin and A. L. Lotts, "Pyrolytic-Carbon Coating in an Engineering-Scale System," Nucl. Appl. 6, 241–255 (March 1969).

possible in the temperature range of 1000 to 1200°C as read by optical pyrometer at the hottest region on the cone. We also found that the densities and coating rates were predictable by using the data previously derived for the CPDL coating furnace. Specifically, the density can be controlled between 0.6 g/cm^3 and at least 1.1 g/cm^3 by means of helium dilution. The coating rate with an 1100°C wall temperature, using an acetylene flux of 2 cm^3/cm^2-min (cm^3/min gas per cm^2 particle surface area) and a helium flux of 1 cm^3/cm^2-min, is 8 μ/min and the resulting density is 1.1 g/cm^2.

At the coating rate of 8 μ/min, a coating time of 10 min will be required for specification thickness. To maintain this rate, the gas flux must remain constant. The prototype system has a programmed gas controller which can vary the gas flux at any predetermined rate. Our next step will be to produce multiple batches of microspheres coated with the buffer coatings as specified for reference recycle particles using a constant gas flux generated by our programmed gas supply.

During initial coating runs, we discovered that our pneumatic unloading system would not reliably transfer the buffer coated particles. Although this type of system had been tested before,[2] the system was installed on the prototype with a number of comparatively sharp bends in the transfer line. These bends apparently impeded the flow of particles. We have modified the transfer system using a technique being developed by the British Ministry of Technology's Warren Spring Lab, Stevenage, Hertfordshire, England. This technique consists of pulsing air into the transfer line near the material inlet. These pulses of air separate the material into plugs to minimize the tendency of the material to jam over long distance transfers or on getting around sharp bends. Our initial tests using this technique have been completely successful.

During the next month we plan to complete the experiments on buffer coatings and begin experiments on isotropic coatings produced from propylene.

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[2] F. J. Furman, J. D. Sease and J. M. Meador, Microsphere Handling Techniques, ORNL-TM-2782, to be published.

III. MATERIALS IRRADIATION

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

1. Irradiation of Prototype Recycle Fuels

(R. B. Fitts, T. N. Washburn, 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 Irradiations

There has been no significant progress during this month.

1.2 Large Scale Irradiations

Eight test fuel elements, known as Recycle Test Elements (RTE's), will be operated in the Peach Bottom Reactor starting in June of 1970. These fuel elements will contain samples of fuels proposed for use in 1100 Mw(e) HTGR's. The test will serve three purposes.

1. Provide irradiated fuel in gram quantities for head-end reprocessing development studies.
2. Provide irradiated fuel blocks to test engineering-scale recycle process equipment.
3. Provide irradiation proof tests of recycle fuel particles prepared with TURF processes and prototype equipment.

In cooperation with Gulf General Atomic, a final plan has been prepared for the loading and utilization of the RTE's. This plan

is summarized below with an explanation of the details of loading and postirradiation utilization of the fuels.

Figure 2 is a cross-section view of an RTE, and six such bodies, each 15 in. long, are stacked vertically to form an element. The RTE's are to be discharged in pairs. Of these pairs, one element will contain fuel bodies each filled with a single particle mixture, while the other element will contain bodies each filled with four different particle mixtures. The multiple mixture bodies will be quartered to separate these mixtures after irradiation. The single-mixture bodies will be used primarily for the larger scale reprocessing studies while the multiple-mixture elements will provide material for hot cell reprocessing studies and fuel performance characterization.

Fuel Mixtures

The fuel particles of interest to the HTGR Recycle Program are listed in Table 1, along with the possible uses of each. Of the various particle combinations which could be formed with these particles, the most promising are listed in Table 2, along with the HTGR use for each.

Table 1. Particles of Interest to the HTGR Recycle Program

Particle	Coating	Possible Uses
$(4.2\ Th,U)O_2$	BISO	Reference recycle fissile particle
$(2.0\ Th,U)O_2$	BISO	Stand-in for above to provide faster accumulation of burnup
UO_2 (buffer diluted)	BISO	Alternate recycle fissile particle Alternate make-up fissile particle
ThO_2	BISO	Alternate recycle fertile particle Alternate make-up fertile particle
UC_2	BISO	Reference make-up fissile particle
UC_2	TRISO	Alternate make-up fissile particle
ThC_2	BISO	Reference recycle fertile particle Reference make-up fertile particle

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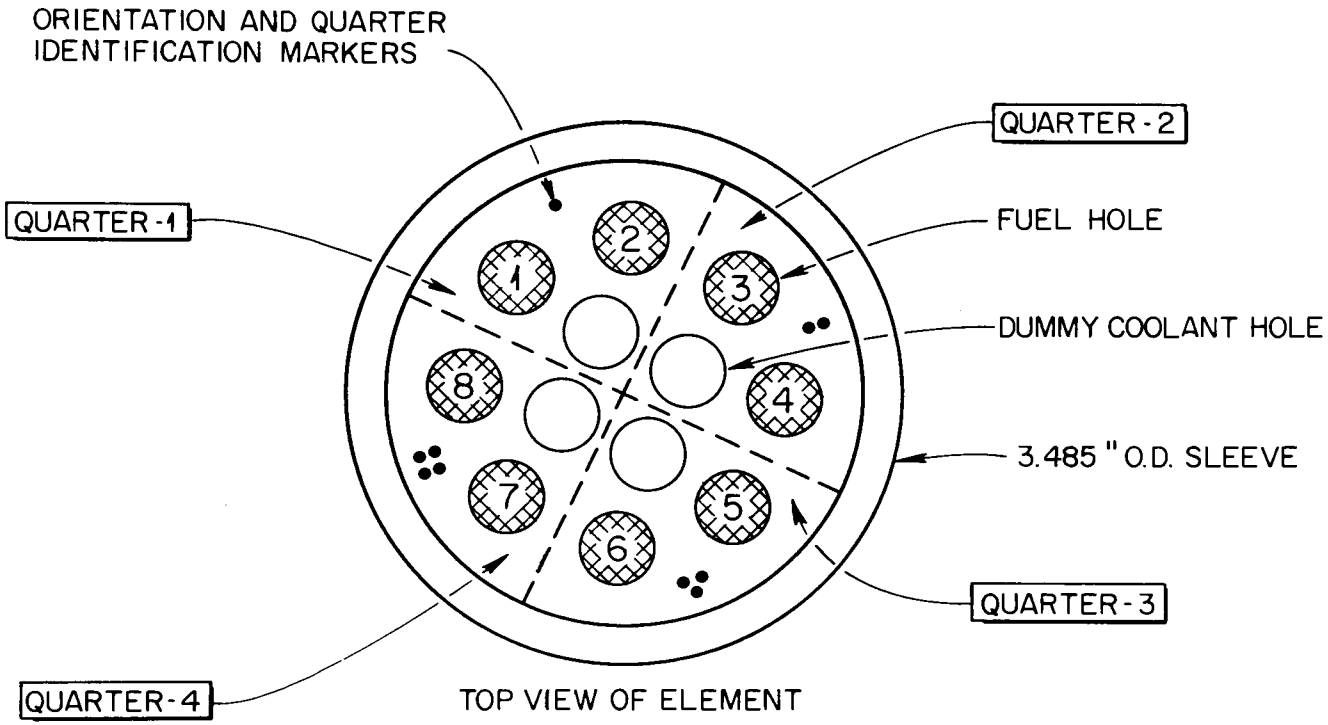

DASHED LINES INDICATE CUTS TO BE MADE AFTER IRRADIATION

Fig. 2. Recycle Test Element.

Table 2. Particle Combinations of Interest

Combination	Fissile	Fertile	HTGR Application
a	$(4.2\ Th,U)O_2$	ThO_2 [a]	Reference recycle
b	UO_2	ThC_2	Advanced recycle or advanced make-up
c	$(2.0\ Th,U)O_2$	ThO_2	Alternate recycle with accelerated burnup
d	$(2.0\ Th,U)O_2$	ThC_2	Reference recycle with accelerated burnup
e	UC_2 BISO	ThC_2	Reference make-up
f	UC_2 [b]	ThC_2	Alternate make-up
g	UO_2	ThO_2	Alternate make-up Alternate recycle
h	UC_2	ThO_2	Alternate make-up

[a] The RTE's containing $(4.2\ Th,U)O_2$ will actually contain no ThO_2 particles in order to maximize the uranium content of the particle bed.

[b] TRISO coating; all other are BISO.

Fuel Element Loading

Each element is composed of six 15-in.-long blocks, and each block contains eight 1/2-in.-diam holes to be loaded with fuel. These holes are identified in Fig. 2. The particle loading for each RTE is shown in Table 3, and the amounts of kernels and coated particles required to provide this loading are given in Table 4.

These test elements are to be discharged in groups of two. This fact, combined with the ability to quarter the elements axially at ORNL after irradiation, permits us to define eight pairs of holes at each axial location in each pair of elements. Thus, we can test the most likely particle combinations under all the available conditions of

Table 3. Loading of RTE's

Position of Fuel Body in Element	℄ 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	abdf[a]	e	abdf[a]	f	abdf[a]	e	aceg	f
5	2250–2050	aceg	a	bdfh	d	bdfh	f[a]	aceg	e
4 (Max flux)	2250–2300	bdfh	f	aceg	e	bdfh	f	aceg	e
3 (Max flux)	2050–2250	aceg	f[a]	aceg	a	aceg	d	bdfh	e
2	1650–2050	aceg	d	aceg	f[a]	aceg	e	bdfh	f
1 (Bottom of reactor)	1050–1650	aceg	e	aceg	e	aceg	f[a]	bdfh	d

[a] Fuel beds in this body are bonded; all others are loose packed beds.

NOTE: a, b, c, d, e, f, g refer to particle combinations of Table 2.

Table 4. Kernel and Coated Particle Requirements for RTE's

Particle	Grams of Kernel Required[a]	Grams of Coated Particles Required
4/1 $(Th,U)O_2$	1377[b]	3515
2/1 $(Th,U)O_2$	1000[c] + 1367	6000
UO_2	273	2058
ThO_2	1392	2140
UC_2 BISO	629	4350
UC_2 TRISO	507	4000
ThC_2	5025	7975

[a] Based on 240 g of U per element.

[b] This is a loading rate of only 224 g/element. This is the maximum loading that can be obtained with this type of particle.

[c] 1000 g required for loading of one FBPTE.

temperature, time, and burnup. Positions are also available for tests of bonded particle beds and of 15-in. blocks containing only one combination of particles.

The overall loading pattern of the pairs of elements is illustrated by the first discharge pair in Table 3. The odd numbered elements (1, 3, 5, 7) contain blocks which will be quartered, and the even numbered elements (2, 4, 6, 8) contain blocks with only one particle mixture in a block. Thus, the odd numbered elements will provide tests of four different mixtures of particles at each axial position and furnish quarter blocks for reprocessing studies. The even numbered elements will provide single-fuel blocks for larger scale reprocessing studies and help in evaluating the performance of prototypic single-fuel blocks. These even numbered elements will also furnish backup samples if more material is

needed than is available in the quarter blocks to study the particles or reprocessing techniques.

The first three odd numbered blocks are loaded with emphasis on particle combinations a, c, e, and g (see Table 2). These combinations, containing all of the particles of interest except the alternate make-up fissile particle (TRISO coated UC_2), are used as the key combinations and are generally placed in the lower four positions in each odd numbered element. These positions, as can be seen in Table 3, cover all of the available temperatures and they also cover the full range of available fast and thermal flux and burnup.

The upper positions in the first three odd numbered elements are loaded with the remaining four candidate particle mixtures and with the combinations of most interest for use in bonded sticks. The four particle mixtures b, d, f, and h will occupy Positions 1 through 4 in RTE-7. This will permit the exposure of all eight mixtures to the full range of temperature and flux; but b, d, f, and h will only be examined for all temperatures at maximum burnup.

The single-fuel blocks emphasize the make-up type fuels. The recycle of these fuels requires separation of the particles containing make-up ^{235}U from those containing bred ^{233}U, and therefore requires more extensive development of the head-end recycling process procedures.

This arrangement of the eight RTE's will meet all three of the test objectives quite well. The head-end crushing and separation development studies can be adequately conducted on quarter blocks and the dissolution studies require only 1/20 of the material in a quarter block. Larger quantities of fuel will be available from the even numbered elements, as needed for engineering-scale work. The prime candidate fuels will be tested under all available conditions so that, in the event of unexpected particle failure, an analysis of the failure regime and cause may be undertaken. In addition, all of the candidate particle mixtures will be tested to full test burnup and fast fluence (4×10^{21} neutrons/cm^2) in RTE's 5 and 7. This arrangement allows early characterization of fuel and development studies using only the odd numbered elements with storage of the even numbered elements until larger quantities of fuel are required.

IV. 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

The dissolution and purification equipment were not operated during the month. Except for 9 kg of uranium (84% ^{233}U) remaining in the vault, all the scrap has been dissolved and accounted for.

2. Storage and Distribution

The facility presently contains 226 kg of ^{233}U which varies in isotopic purity between 84 and 98% and ^{232}U content between 4 and 250 ppm. Receipts and shipments consisted of 105 and 204 g ^{233}U, respectively, 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 pressure control valve, purchased for use in the new vessel off-gas line for storage tank P-25, was received but not installed because of a shortage of manpower. The contents of the tank will not be resampled until the off-gas line is completed so sparging tests may precede the sampling, thus reducing the radiation exposure to personnel caused by repeated sampling.

3. LWBR Support Program

Funding was received from Bettis Atomic Power Laboratory to support ORNL participation in the Light Water Breeder Reactor Program. ORNL will

receive approximately 650 kg of ^{233}U from the production plants, store the material as a solid or solution, purify and convert to ceramic grade UO_2 powder at a rate of 25 kg ^{233}U per week. Operation is scheduled to begin in January, 1971.

Development programs to optimize the purification as well as the oxide forming flowsheets are underway. Detailed design of the additional storage space and modifications to the solvent extraction facility have begun.

The present dissolver is to be replaced with an improved design. To date, the charging facility in the penthouse has been removed.

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