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THORIUM FUEL CYCLE DEVELOPMENT STUDIES PROGRESS REPORT

AUGUST 1969

No. 1

Compiled by

The Staff of the Chemical Technology

and

Metals and Ceramics Divisions

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

IV. URANIUM-233 REPROCESSING

R. E. Brooksbank, J. R. Parrott

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ABSTRACT

This is the first in a series of monthly reports which will give the progress on programs relating to the thorium-^{233}U fuel cycle. The programs, which are almost solely directed toward application in HTGR's include head-end processing, refabrication development, and irradiation testing of fuels. This series of reports continues the reporting previously done in the ORNL Fuel Cycle Studies Report.

HIGHLIGHTS

Cold tests on a 5-in. prototype remote coater for coating particles with pyrolytic carbon and SiC were begun.

Detailed planning of the irradiation program for prototype recycle fuels was begun. Two stages are to be implemented in FY-1970:
(1) accelerated capsule tests in the Advanced Test Reactor, and
(2) eight test rods to be irradiated in the Peach Bottom Reactor.

INTRODUCTION

The thorium fuel cycle development studies at ORNL are 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 separately, or of mixtures of them in a single microsphere. The microspheres are coated with layers of pyrolytically deposited carbon and silicon carbide and are bonded in an organic matrix to form a stick which is carbonized. The bonded microsphere sticks are retained in the holes in the graphite blocks.

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 irradiation effects which affect fuel reprocessing steps and to provide a comparison with metallographic studies. An important objective is the determination of the amount of breakage of silicon carbide 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 reported last month is continuing. An elementary computer program has been written for rapid reduction and compilation of analytical data. The program is being verified using existing run data. Current and future experiments will be handled by this method. The output will provide a concise and uniform summary of run data for easier future reference and cross comparisons.

1.2 Sphere-Forming Column Chemistry
(W. D. Bond, J. W. Snider, P. A. Haas)

Our previous studies have shown that pH, surfactant concentration and water content of 2-ethyl-1-hexanol (2EH) are important variables in the microsphere forming process. We are presently examining methods for improving the control of pH and surfactant concentration during continuous operation of sphere forming columns. Our present system for controlling the water content is adequate. For water content control, a portion of the used solvent is heated at 150-160°C to remove most of the water, and the desired water content is obtained by mixing this relatively dry 2EH with the remaining wet solvent.

In the past, sphere forming columns have been operated continuously by making periodic additions of surfactants to off-set the losses of surfactant caused largely by reaction of the nitric acid (extracted from the sol) with the surfactants during removal of water by distillation. In addition to reactions during distillation, a relatively small amount of reaction occurs in the forming column as evidenced by slow discoloration of the solvent when the forming column is batch operated without a distillation system. The pH or acid contents of the 2EH that have been employed in continuous operation, have been mainly those governed by the amount of acid extracted from the sol which can subsequently be maintained in the 2EH with the distillation system operating. When higher acid contents are required, nitric acid must be added to the 2EH, and reactions with surfactants and 2EH are increased.

Laboratory studies have shown that reactions of nitric acid with 2EH and surfactants during distillation could essentially be eliminated by removal of the acid prior to distillation step by contacting the 2EH with alkaline solutions. The acid content of the alochol could then be maintained by adding nitric acid to the alcohol after the removal of water by distillation. We are presently evaluating this procedure as a means of controlling the pH and surfactant concentration of the engineering development sphere forming column during continuous operation. We are also studying a total 2EH purification method in which the 2EH is recovered by distillation; the used surfactants are discarded, and the 2EH is recycled to the forming column after surfactants, water and nitric acid are added. Studies are continuing on the effect of surfactant and water content, and the pH of 2EH on sphere forming using different sols in batch operated laboratory forming columns.

Effect of pH of 2EH in Forming ThO_2-UO_3 Microspheres

Span 80 is an effective anti-clustering surfactant in concentrations as low as 0.003% for use in the 2EH in the sphere forming column operating ThO_2-UO_3 sols prepared by solvent extraction, but at the pH of the sols (~ 4.5) it causes pitting. The pitting is diminished by the addition of Ethomeen S/15. Pitting also decreases as the 2EH pH is raised; this led to bead-forming experiments in which the 2EH was made basic by addition of NH_4OH. Not only did pitting cease entirely, but also it was found possible to form larger microspheres than could be formed in the acidified column. For example, at about pH 9.0, some good spheres were made which had diameters after firing of over 600 μ.

Studies With Engineering Development Microsphere Forming Column

The sphere forming system was modified by addition of two spray columns for scrubbing the 2EH prior to distillation and one scrubbing column for control of the solvent pH. Various alkaline solutions have been tested for scrubbing the 2EH. Good results in sphere forming have been obtained for UO_2 gel spheres (see Sect. II, 2.3 of LMFBR Fuel Cycle Studies Progress Report for August 1969) using this system, and we are hopeful that it will be useful in microsphere forming of ThO_2-UO_3 sols. Laboratory studies are being carried out on the determination of distribution coefficients of various species between the 2EH and the scrubbing solutions. Results on extraction of HNO_3, HCOOH, Na_2CO_3 and NH_4OH are reported in the August LMFBR Fuel Cycle Studies Progress Report. Determination of losses of surfactant to the scrubbing solutions are in progress.

Recovery of 2EH by Distillation

A continuous distillation unit for recovery of 2EH has been constructed for laboratory studies. Preliminary tests using synthetic solutions of 2EH, surfactants, and water confirmed our previous batch distillation results, in which 95-99+ % of 2EH was recovered. Tests are now in progress on 2EH that has been used during continuous operation of the engineering development microsphere forming column.

1.3 Equipment Development and Design
(J. W. Snider)

Particle size estimation of hydrosol droplets during initial column startup has been accomplished visually during developmental studies. Operation of microsphere forming columns in high radiation fields will require techniques other than direct vision. An optical system will be tested in the CPDL for possible use in the TURF.

An alternate method of estimating the size of hydrosol droplets might be accomplished by measuring the unsteady-state water concentration of the microsphere forming column. For a given hydrosol concentration and feed rate to the column, the number of droplets formed per unit time is related to the cube of the droplet diameters. The time required for a hydrosol droplet to gel is proportional to its initial diameter.

Experiments are currently underway to develop an in-line instrument for water determination in 2-ethyl-1-hexanol. An unsteady-state computer code has been written to calculate the material balance for a microsphere forming column. The results of this code should provide a good estimate of the accuracy required on the water concentration measurement for it to be used to estimate the initial distribution of hydrosol droplets.

The rate of change in diameter of a hydrosol droplet, using the measured mass transfer film coefficient, is given by Clinton[1] as:

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[1] S. D. Clinton, Mass Transfer of Water from Single Thoria Sol Droplets Fluidized in 2-Ethyl-1-Hexanol, ORNL-TM-2163 (Thesis) (June 1968).

$$-\frac{dD_p}{d\theta} = \frac{0.18}{\rho_w}(C_s - C_o)(0.238\,\underline{M}_p + 0.17)^{0.38}$$

where

D_p = diameter of hydrosol droplet, cm,
θ = time, min,
C_s = concentration of water in 2EH at the hydrolsol droplet interface, g/cc,
C_o = concentration of water in the bulk 2EH stream, g/cc,
$\underline{M}_p$ = molarity of hydrosol droplet, moles/liter,
ρ_w = density of water, g/cc.

The computer code written solves the above equation for five different hydrosol droplet diameters and sums them for all droplets admitted to the column which have not gelled. The concentration of water in the 2EH is calculated by material balance assuming plug flow through the external column system.

Calculations were made for a 1-in.-diam microsphere forming column being fed 5 cc/min of a 2 $\underline{M}$ hydrosol. For an initial system concentration of 0.006 mg H_2O/cc and with the 2EH still effluent at the same concentration, calculations have been made on the unsteady-state microsphere forming column water concentration. The maximum difference in water concentration was 0.00061 and 0.00066 mg H_2O/cc for test cases 1 and 2, respectively (see Table 3-1). These maxima occurred at eight and ten minutes after startup for cases 1 and 2, respectively.

For this technique to be applicable as an in-line estimation of hydrosol droplets which will produce a calcined product with 75% within a $\pm$ 100 μ size range, it will require an instrument that is accurate to $\pm$ 0.00001 mg H_2O/cc.

Table 3-1. Particle Size Distributions of Case 1 and Case 2.

Hydrosol Droplet Diameter, cm	Case 1 Distribution No. 4, %	Case 1 Distribution No. 2, %
0.160	3.73	3.00
0.116	41.15	6.17
0.080	31.39	9.38
0.056	2.16	14.71
0.040	21.57	66.74

	Case 2 Distribution G, %	Case 2 Distribution B, %
0.147	0.56	0.22
0.107	67.56	0.41
0.080	30.71	40.02
0.053	0.18	58.54
0.040	0.99	0.82

1.4 Preparation of Test Materials
(R. E. Brooksbank)

A campaign to prepare over 32 kg of $^{233}UO_2$-ThO_2 microspheres for use in physics experiments in the HTLTR at Pacific Northwest Laboratory has just been completed. We are about to begin the preparation of about 6 kg of $^{235}UO_2$-ThO_2 for use in preparing irradiation test elements for insertion in the Peach Bottom Reactor. The sols for these tests are prepared in alpha-contained, critically safe solvent extraction equipment in the Pilot Plant. The spheres are dried and fired in Cell 4 of the Pilot Plant.

2. HEAD-END ENGINEERING STUDIES

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. The burn-leach program will remain relatively unchanged.

In this report period, the extent of fissile and fertile particle breakage from crushing prototype Ft. St. Vrain fuel in a hammer mill was measured and leaching tests to evaluate the particle breakage contribution in a fluidized bed burner were made. However, an analysis of the particle breakage from fluidized bed burning is incomplete and is not reported this month.

Reportable accomplishments include:

2.1 Mechanical Head-End
(C. D. Watson, R. S. Lowrie)

Scoping studies are being made to determine the extent of particle breakage in prototype Ft. St. Vrain fuel sticks during a head-end crushing or comminution step of hammer milling. Two pie-shaped fuel sections (each 1/6 of a hexagonal fuel block) containing TRISO[1] coated UO_2 and ThO_2 particles were crushed in a hammer mill[2] with the grid bars set to produce minus 3/4-in. material. The crushed material was sized, and duplicate samples riffled from each size fraction which contained fuel particles. These samples were leached for 17 hours with boiling acid (12 M HNO_3, 0.04 M F^-, 0.1 M Al^{+++}), washed and the solutions and residue analyzed for U + Th. The results are shown below:

HTGR Run No.	wt % of Particles with Broken Coatings	
	UO_2 (fissile stand-in)	ThO_2 (fertile stand-in)
11	2.2	1.1
14	0.1	1.5

For the worst possible case when the largest breakage values for either shape are assumed to be the values of the ^{235}U (UO_2 particles) and the ^{233}U (ThO_2 particles) cross mixed into the appropriate recycle or retired uranium process stream, the cost penalty incurred would be ~ 0.007 mill/kw-hr, well below the acceptable value of 0.015 mill/kw-hr.[3]

2.2 Burn-Leach Head-End
(C. D. Watson, R. S. Lowrie)

Studies are under way to determine the extent of particle coating breakage as a function of the operating parameters when crushed Ft. St. Vrain prototype fuel is burned in a fluidized bed reactor. Leaching tests to ascertain particle coating breakage of the TRISO coated UO_2 and ThO_2 particles in the burner residues from three burning tests reported last month have been completed and the data are being evaluated. Data analyses will be reported next month.

Fluidized bed burning tests will also be made next month to determine the effect of using CO_2 instead of N_2 as the diluent in the oxygen stream.

References

1. TRISO refers to three layers: the first two layers are carbon consisting of a porous buffer layer overlaid with a thicker non-porous pyrolytic protective layer; the third outer layer is pyrolytic carbon containing a middle layer of SiC.

2. Jeffrey Swing Type Model A Swing Hammer Pulverizer - 900 rpm, 10 hammers 3, 2, 3, 2 array, 90° apart rotating in a chamber 14-in.-diam x 8-in.-wide, -3/4 in. grating.

3. Based on data shown in Fig. B-6 National HTGR Recycle Development Program Plan - 29 May 1969 (in rough draft at ORNL).

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 4.25. 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 a reliable, remote method for reproducibly mixing $Th(NO_3)_4$ and $UO_2(NO_4)_2$ 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, and forming ThO_2-UO_3 gel microspheres 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.

CPDL Microsphere Preparation (C. C. Haws)

Over 100 kg of ThO_2-UO_3 sol was prepared in an engineering demonstration run. This sol has been used in sphere forming, drying, and firing studies in engineering equipment being developed to provide guidance for the design of equipment for TURF. Forty kg of ThO_2-UO_3 gel spheres prepared during May were dried and half of them were fired. The driers, which have been modified following earlier tests, operated without significant breakage of spheres, although small temperature excursions in the gel sphere beds occurred near the ends of drying cycles. Some uranium reduction occurs during drying. The dried gel spheres heat up if exposed to air. This behavior has been observed before, and is due to hydration of the porous, dry gel, and to uranium oxidation. The Th-U material balance for this batch is about 95%; about 5% of the fired product was rejected by shape and size classification operations.

Inspection of three 6 in. ID pyrex pipe sections used to hold sol feed revealed serious defects or stresses. Therefore, we have fabricated stainless steel sol feed tanks to replace the two Pyrex pipe tanks previously used.

The sphere forming column system is being modified to provide spray columns to scrub and control the pH of the 2EH (see Sect. 1.2).

2. Fueled Graphite Fabrication Development

F. J. Furman, J. D. Sease,
R. A. Bowman, and J. M. Robbins

We are developing processes and equipment for the refabrication of HTGR recycle fuel, as detailed in the National HTGR Recycle 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 typically made into a fuel stick which is then inserted into a hexagonal graphite log, which contains both fuel holes and coolant holes.

Our work is divided into particle coating, particle handling and inspection, particle blending, fuel stick molding and inspection, fuel element assembly, fuel stick carbonization, and fuel element inspection. Currently, we are concentrating on the areas of particle coating, particle handling, and particle inspection. Some limited work is being done on fuel stick making.

2.1 Particle Coating

Particle coatings are intended to limit the spread of fission products in the reactor. The types of coatings used are high-density pyrolytic carbon, formed from the decomposition of propylene, low-density pyrolytic carbon, formed from the decomposition of acetylene, and silicon carbide formed from the decomposition of methyltrichlorosilane in the presence of hydrogen. We have extensive experience in operating a 5-in.-diam laboratory coater, which is sufficiently large for production work. Because of the high radioactivity associated with recycle fuel, we have constructed a prototype of a remotely operated 5-in.-diam coating furnace. This furnace incorporates many improvements over our laboratory coater. The most important improvement is the modular construction of the furnace, which allows remote maintenance to be performed.

All modules are joined by disconnects which can be handled by a manipulator. Other improvements are our exhaust system, which can safely handle the hydrogen gas effluent of two furnaces; the control system which allows all coating gas adjustments to be made from one location some distance from the actual operation; and the safety interlock system which prevents furnace operation if hazardous conditions exist.

The writing of operating and maintenance procedures for the furnace is now completed. Also, initial cold check-out of the equipment has begun. The furnace power supply was found to be incorrectly wired; and this problem has been corrected. Tests on the furnace cooling system have begun.

2.2 Particle Handling

Particle handling involves the transfer of particles between equipment and the processing of the particles after particle forming, drying and firing, and before particle coating. These processes included sizing the particles, eliminating non-spherical particles, sampling, and weighing. Our particle handling development has two objectives: (1) to develop processes and equipment which will be used in a remotely operated facility, and (2) to set up and operate equipment for processing material to be used in coating development operations.

For particle sizing, we are currently modifying a commercial SWECO particle sizer that separates by gyrating 18-in.-diam screens. We are currently testing the SWECO unit to determine screening efficiency and amount of screen blinding.

For shape separation, that is elimination of non-spherical particles, we use a flat plate which vibrates in one direction and slopes in a direction of 90° to the vibration. Spherical particles tend in the direction of the slope, and the non-spherical particles move with the vibration, thus achieving a separation. We are currently setting up four vibrators, each having two shape-separator plates. Each plate uses a particle feeder which is designed to minimize maintenance requirements.

2.3 Particle Inspection

To economically produce coated particles, the size of the particles must be accurately known immediately before and after coating. To achieve this, we are developing a particle size analyzer which measures the size of individual particles by the amount of light a particle blocks as it passes between a light source and a photo-cell. To aid in the calibration of this instrument, we have sized reference batches containing approximately 1000 particles. These batches vary in size from 250 to 700 μ. By sizing the batches with the instrument, we can cross-check the instrument calibration.

Normally, we convey the particles through the analyzer measurement cell by an air stream. However, low-density buffer coated particles abrade slightly and deposit a layer of carbon dust on the surface of the cell, thereby blocking the light source. To prevent this, we have developed a water transport system which minimizes the abrasion and prevents the abraded material from depositing on the window. The change from air to water transport requires only a few minutes and dows not change the calibration of the instrument.

2.4 Coated Particle Bonding Technique for HTGR Fuels

The results from irradiation experiments show that the stability of particle bonding needs to be improved for use in HTGR fuels.[1] The objective of this program is to develop a fuel body that will withstand irradiation to the exposures required for Fort St. Vrain and larger HTGR's, i.e., 20% burnup at temperatures of 600 to 1250°C and fast neutron fluence up to 8×10^{21} neutrons/cm^2 (> 0.18 Mev).

We are waiting on the furnace for heating our hot injection device when fabricating bonded bed specimens using thermoplastic binders. In the interim we are trying to use a heating tape for heating the injection device and mold.

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[1] J. H. Coobs *et al.*, "Irradiation of Loose and Bonded Coated Particles in HFIR Targets," pp. 28–54 in *GCRP Semiann. Progr. Rept. Sept. 30, 1968*, ORNL-4353.

Currently, specimens are being fabricated for the HT-4 experiment. Specimens have been prepared containing PyC-coated carbon particles bonded with a mixture of furfuryl alcohol, Poco graphite flour (29 wt %) and Thermax carbon black (29 wt %). Remaining to be made are the specimens with the same type particles bonded with 15V pitch, Poco graphite flour, and Thermax carbon black. These specimens are scheduled for reactor insertion in late July.

III. MATERIALS IRRADIATION

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

1. Irradiation of Prototype Recycle Fuels

A. R. Olsen and F. J. Furman

The HTGR recycle program requires irradiation tests. The twofold objectives of these tests are: (1) to provide irradiation fuel for head-end process studies, and (2) to provide irradiation proof tests of the products of the various stages of process development for the Thorium Uranium Recycle Facility. The test conditions have been defined as fuel temperatures between 750 and 1300°C with fast fluence exposures from 4 to 8 $\times 10^{21}$ neutrons/cm^2 and burnup levels up to 20% FIMA.

A multi-stage program has been proposed with two stages to be implemented during FY-1970. These two stages involve accelerated capsule tests in the Advanced Test Reactor and eight test rods to be irradiated in the Peach Bottom Reactor. Preliminary design parameters for the Peach Bottom test rods have been proposed. Information required for the design of the ATR capsules has been obtained from Idaho Nuclear Company.

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

A total of eight dissolutions were made during the month. The material dissolved consisted of waste UO_2-ThO_2 microspheres from the Sol Gel Development Pilot Plant, scrap metal, UO_2, and analytical waste (4,311 g ^{233}U).

2. STORAGE AND DISTRIBUTION

The facility presently contains 228 kg of ^{233}U which varies in isotopic purity between 91% and 98% and contains ^{232}U quantities between 4 ppm and 250 ppm.

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 contents of this storage tank (P-25) were not analyzed this month. Tests are being made to determine how to reduce the radiation background in the vessel off-gas line during sparging, preparatory to sampling. An aqueous sample of material collecting at the pressure control valve in the off-gas line was analyzed and found to contain daughters of ^{232}U (^{208}Tl, ^{212}Bi and ^{212}Pb). During a long term sparging test, a portion of the off-gas stream was passed through paper filters, silica gel, and charcoal in separate runs to determine the efficiency of removal of the highly radioactive elements. The results of these tests are incomplete.

There were no shipments during the month.

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