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IN-PILE LOOP IRRADIATION OF AQUEOUS THORIA-URANIA SLURRY AT ELEVATED TEMPERATURE. DESIGN AND IN-PILE OPERATION OF LOOP L-2-27S.

> H. C. Savage E. L. Compere J. M. Baker V. A. DeCarlo A. J. Shor



OAK RIDGE NATIONAL LABORATORY

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REACTOR CHEMISTRY DIVISION

IN-PILE LOOP IRRADIATION OF AQUEOUS THORIA-URANIA SLURRY

AT ELEVATED TEMPERATURE. DESIGN AND IN-PILE

OPERATION OF LOOP L-2-275.

H. C. Savage, E. L. Compere, J. M. Baker, V. A. DeCarlo, and A. J. Shor

DATE ISSUED

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IN-PILE LOOP IRRADIATION OF AQUEOUS THORIA-URANIA SLURRY AT ELEVATED TEMPERATURE. DESIGN AND IN-PILE OPERATION OF LOOP L-2-27S.

H. C. Savage, E. L. Compere, J. M. Baker, V. A. DeCarlo, and A. J. Shor

ABSTRACT

An in-pile pump loop, designed to fit within horizontal beam hole HB-2 of the Low-Intensity Test Reactor (LITR), was used to circulate an aqueous thoria-urania slurry while exposed to reactor irradiation. The total loop volume was about 1600 ml, including pump and pressurizer, but the slurry was confined to the 900-ml volume of the main loop stream by means of a sintered stainless steel filter. The filter was an important feature of the loop design in that it provided a thoria-free filtrate as a purge stream to the pressurizer and pump bearings to prevent entry and accumulation of thoria in these two regions.

Corrosion-test specimens of Zircaloy-2, titanium, and type 347 stainless steel were placed in the loop at three different locations for exposure to three different levels of irradiation. Duplicate sets of specimens in each position were exposed to flow velocities of 8 and 22 fps, respectively.

The loop was connected to auxiliary equipment at the reactor face by capillary tubes. This equipment was used to remove slurry samples and to make additions of D_2O and gas to the loop while operating in-pile and to drain and purge the loop before it was removed from the reactor.

For the in-pile irradiation, thorium oxide containing 0.43 w/o enriched uranium, based on thorium, was used. This thoria-urania was produced by air calcination at 1225°C of coprecipitated oxalates and had a mean particle size of 1.7 μ . A palladium catalyst was dispersed in the slurry for liquid-phase recombination of the radiolytic gas.

The loop was operated in beam-hole HB-2 of the LITR from July 19 to October 19, 1960. During this period slurry was continuously circulated at 280°C for 2220 hr without incident; 1839 hr were at full reactor power (3 Mwt), at which the estimated average thermal flux over the 300-ml volume core section was 5×10^{12} neutrons/cm²·sec.

At the start of in-pile operation the loop was charged to a concentration of 979 g of Th and 3.83 g of fully enriched U per liter (at 280°C) which was reduced by sampling to 748 g of Th and 2.74 g of U per liter at the end of the irradiation period based on the assumption that no losses had occurred. Samples of slurry were withdrawn at intervals for analyses to determine the effects of radiation on the thoria-urania slurry.

When the experiment was terminated, the loop was drained, rinsed, and removed from the reactor for dismantling and examination in hot-cell facilities.

1. INTRODUCTION

Thorium oxide slurries are of interest as a fluid, fertile material in an aqueous homogeneous thorium-breeder reactor. Extensive out-of-pile studies of thorium oxide slurries have been carried out at ORNL since 1955, ^{1,2} and in-pile experiments have been conducted in static and rocking autoclaves during the past several years.³ Since a pump loop capable of circulating the slurry more nearly duplicates the dynamic conditions which would exist in the reactor, a pump loop capable of circulating a thorium oxide slurry while exposed to reactor irradiation has been a major goal of the aqueous homogeneous reactor development program.

A 5-gpm in-pile loop capable of circulating a thorium oxide slurry was developed^{4,5,6} and was operated in an experimental facility of the Low-Intensity Test Reactor (LITR) from July 19 to October 19, 1960, to determine the effect of radiation on the physical properties of the thorium oxide and on the corrosion-erosion of containment materials.

The thorium oxide, containing 0.43 wt % U²²⁵ based on thorium, dispersed in D₂O, was circulated at a temperature of 280°C and at concentrations to 1350 g of Th per kg of D₂O while in-pile. A "sol-prepared" palladium oxide catalyst⁷ was added to the slurry for internal recombination of the radiolytic gas (D₂ + 1/2 O₂) formed under irradiation. Samples of the circulating slurry were removed from the loop periodically for chemical analysis and determination of physical properties.

Corrosion test specimens of Zircaloy-2, type 347 stainless steel, and titanium were exposed to the slurry at two different velocities (8 and 22 fps) and at three neutron-flux levels for information on the effect of reactor radiation on corrosion/erosion.

This report describes the design features of the loop, the experimental facilities, and operation of the loop in-pile. Results of analyses of slurry samples removed during in-pile operation and dismantling of the loop and examination of the corrosion test specimens and loop components have not been completed and are not reported here.

2. DESCRIPTION OF LOOP AND EXPERIMENTAL FACILITIES

2.1 Loop

The in-pile slurry loop was similar to the pump loops used to obtain radiation-corrosion information with uranyl sulfate solutions⁸ and was designed to be operated with the experimental facilities already in existence at the LITR. Only those modifications were made to the solution loop which were found necessary to ensure that the thoria remained in suspension during circulation. These modifications involved redesign of the loop core section to increase the fluid velocity in that region from two to six fps (velocity in the 3/8-in. sched-40 main loop piping was nine fps) and the use of a filter to provide thoria-free filtrate. This filtrate was used as a pressurizer feed stream to prevent entry and accumulation of thoria in the low-velocity pressurizer (<0.1 fps) and as a purge stream for the canned-rotor pump bearings.

The loop was designed to operate at temperature and pressures to 300°C and 2000 psia and was constructed entirely of type 347 stainless steel with the exception of the pump bearings (aluminum oxide) and impeller (Zircaloy-2). Total loop volume was ~1630 cc, which included ~520 cc for the pressurizer and ~900 cc for the main loop stream and core section. The remaining 210 cc was taken up by the

pump-rotor cavity (160 cc) and the various interconnecting tubing. By virtue of the filter all solids (thoria-urania, Pd catalyst, etc.) were confined to the 900-cc volume of the main loop stream and core section. A schematic diagram and an assembly drawing of the loop are shown in Figs. 1 and 2, respectively.

Tables Al, A2, and A3 in the appendix may be referred to for a tabulation of the drawing numbers, material identifications, volumes, and internal surface areas for all loop components.

2.1.1 Core

Figure 3 is a photograph of the loop core section which was designed to have a fluid-flow velocity of 6 fps (at 5 gpm) to prevent thoria dropout and deposition within the core. One-half-in. sched-40 pipe was used for the core section because of its appropriate cross-sectional area. The particular configuration used was the result of several considerations: (1) to provide sufficient volume (300 cc) to expose an appreciable fraction (35%) of the slurry to maximum neutron flux, (2) to provide space for corrosion-test specimens, and (3) to fit the existing loop container and beam-hole dimensions.

Thirty-two corrosion test specimens were exposed to the circulating slurry in the core section and Co-Al flux monitors, encased in stainless steel, were placed on the outside of the core for determination of the thermal flux by counting after termination of the experiment. A graphite reflector and moderator was installed to the rear of the core as shown in Fig. 2.

2.1.2 Pump

An ORNL 5-gpm canned-rotor pump (Fig. 4) equipped with aluminum oxide bearings and journal bushings was used to circulate the thoria slurry. Except for the bearings and impeller, the pump was fabricated entirely of type 347 stainless steel. Zircaloy-2 was chosen for use as the impeller material because this alloy had been found to be more resistant than stainless steel to slurry corrosion-erosion in highly turbulent regions. The pump-bearing and rotor region was continuously purged with the thoria-free filtrate obtained from the sintered-metal filter. This prevented entry and accumulation of thoria in the pump-rotor cavity with resultant loss of thoria from the main loop circulating stream and also minimized the possibility of excessive bearing wear by the thoria solids. Since this filtrate was routed through the pressurizer (295°C) before entering the pump, it was necessary to cool it to prevent pump overheating. Thus the purge stream was cooled to about 40°C before entering the pump.

Pump-characteristic curves are shown in Fig. 5, and the effect of thorium concentration on pump power is plotted in Fig. 6. During in-pile operation, pump-power measurements were used as a qualitative indication of the quantity of thorium in circulation.

2.1.3 Filter

A sintered-metal filter of type 347 stainless steel was a part of the main loop circuit and provided a thoria-free filtrate as a pressurizer feed stream and pump-bearing purge. The filter element was in the form of a cylinder 9/16 in. OD by 11/32 in. ID and 11 1/4 in. long. Mean pore openings were $4.0-\mu$ radius, with maximum-size openings of $6.2-\mu$ radius. The circulating slurry flowed axially through the cylinder, and the filtrate was collected in an annulus between it and the loop piping as shown in Fig. 7. For a pressure differential across the filter of 40 ft of water, an initial flow rate of approximately 7 cc/sec of thoria-free filtrate was obtained with the slurry at 280°C. An expansion bellows of type 321

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Fig. 1. Flow Diagram, In-Pile Slurry Loop L-2-27S

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Fig. 2. Slurry In-Pile Loop, L-2-27S.

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Fig. 3. Coiled-Pipe Core for the In-Pile Slurry Loop.

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Fig. 4. Canned Rotor Pump.



Fig. 5. Characteristics of 5-gpm Pump (Serial No. 219), Water (H₂O) at 25° C.

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Fig. 6. Pump Power vs Thorium Concentration.

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Fig. 7. Diagram of Filter Assembly Showing Flow of Slurry and Filtrate

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stainless steel was attached to one end of the filter element to protect it against fracture due to differential thermal expansion between the filter and its housing. Figure 8 is a photograph of the filter and bellows assembly.

2.1.4 Pressurizer and Pressurizer Heater

The pressurizer was fabricated from 1 1/4-in. sched-80 pipe with a volume of 521 cc at operating temperature (295°C), and was mounted in a horizontal position (refer to Fig. 2). The thoria-free filtrate from the sintered-metal filter was circulated to the pressurizer and returned to the loop through the rear of the pump. Before entering the pressurizer, the filtrate passed through a pressurizer heater where it was heated to a temperature above that in the loop main stream to provide steam overpressure.

Fluid from the pressurizer heater entered the pressurizer at one end and exited at the opposite end. Both inlet and exit lines were of 1/4-in. x 0.049-in. wall tubing connected to the bottom of the pressurizer (refer to Fig. 1). In order to maintain, as nearly as possible, equilibrium thermal conditions throughout the pressurizer, a pressurizer heating jacket was used to minimize heat losses. Two tubes of capillary dimensions, connected to the upper vapor space of the pressurizer, were used for pressure measurement and oxygen additions.

2.1.5 Loop Heater

Operating temperature of the loop was maintained and controlled by means of a loop heater which consisted of Calrod-type electric heaters cast in an aluminum matrix around the main loop piping. Heater capacity was 7500 w. This relatively large capacity was required because the pump purge was cooled to about 40°C after leaving the pressurizer and required reheating to 280°C upon re-entry into the main loop stream. Dependent on the pump-purge flow rate, 5 to 6 kw of heat were required to maintain the loop operating temperature of 280°C.

2.1.6 Corrosion Test Specimens

A total of 48 corrosion test specimens of type 347 stainless steel, Zircaloy-2, and titanium were exposed to the circulating slurry in the loop. These specimens were mounted in six holders; each holder contained an array of eight specimens as shown in Fig. 9. Two types of holders were used; in one the flow channel was designed to give a velocity of 22 fps past the coupons (high velocity), and in the other 8 fps past the coupons (low velocity). High- and low-velocity holder pairs were mounted in three different locations in the loop; one set, in the main loop stream, removed from the neutron flux; a second set, in the rear of the core section, for an intermediate flux exposure; and a third set at the foremost position in the core for the highest neutron-flux exposure. Each corrosion test coupon, 0.250 in. by 0.637 in. by 0.62 in. thick, was photographed and weighed before installation in the loop. The machined and sanded surfaces of all specimens were replicated for comparison with postirradiation observation. Initial weights of all specimens and identification of the material from which they were fabricated are tabulated in Table A4 of the appendix.

2.2 Experimental Facilities and Auxiliary Equipment

The in-pile slurry loop package, consisting of the pump loop, shield plug, and container can, was designed to operate in beam hole HB-2 of the LITR. This horizontal beam hole extends from a point adjacent to the reactor lattice back some 13 ft to the face of the reactor shielding. Only that portion of the loop, the core section, which is close to the reactor lattice, was exposed to appreciable neutron flux, since the flux decreases rapidly with distance from the lattice as



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Fig. 9. Core Corrosion Specimen Holders

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shown in Fig. 10. The fuel-loading pattern used in the LITR during in-pile operation of the loop is shown in Fig. 11.

Two interconnected equipment chambers were located at the face of the reactor. These equipment chambers contained all the tanks, valves, and capillary tubing necessary to remove slurry samples from the loop and to make additions of fluid and gas during in-pile operation. The equipment chambers were sealed and vented to the reactor off-gas system to prevent accidental release of radioactive material. Lead shielding surrounded the equipment chambers to reduce the level of radioactivity from the storage tanks in the chambers. A cutaway drawing of the loop facility at beam hole HB-2 is shown in Fig. 12, and a process flowsheet is shown in Fig. 13.

From the standpoint of obtaining information on the effect of reactor radiation on thorium oxide slurries, the chemical analysis and physical-property measurements of samples of slurry routinely removed from the circulating stream of the loop were of primary importance. The equipment and procedures for removing samples of the highly radioactive slurry from the loop are described below.

2.2.1 Sampling Equipment and Procedures

With the exception of the lead-shielded container used to transfer the slurry samples to hot-cell facilities, the entire sampling system was located inside the shielded equipment chambers. This system is shown schematically in Fig. 14.

The initial withdrawal from the loop was into the 15-cc-volume tank, called the standard drain volume (SDV), and after venting, the contents of the standard drain volume were transferred by displacement with low-pressure gas (~30 psia) to the evacuated external sample tank. Briefly, the entire procedure consisted of evacuating the SDV to off-gas pressure through valves 12, 59, and 56. A sample was then withdrawn into the SDV through valves 3 and 7. Valves 3 and 7 were then closed and part of the pressure in the SDV was relieved by opening valve 20, which allowed expansion into the short length of capillary tube between values 20 and 67. The SDV was then further vented to the slurry dump tank by opening valve 6. The first 15-cc slurry volume obtained was discarded to the slurry dump tank and was thus used to flush residual slurry (from previous samples) from the sampling lines. A second 15 cc was then withdrawn from the loop into the SDV and, after venting, ~30 psia of gas pressure from the low-pressure oxygen header was used to transfer the slurry to the external sample tank. All lines through which slurry had passed were then back-flushed with water from the wash-tank header, and these lines were subsequently dried by flushing with oxygen gas from the low-pressure oxygen header.

A mockup of the sampling system was tested during out-of-pile operation of the loop, and analysis of samples obtained in this manner gave results which compared favorably with thoria concentrations expected from the book inventory (see Table 1, Sec. 3.4).

2.2.2 Addition System

For proper operation, the fluid volume in the loop must be maintained within fairly close limits. Thus it was necessary to replace the volume of slurry removed in sampling. This was done by means of an addition system located in the equipment chambers. The addition system is shown schematically in Fig. 15.

For an addition to the loop, the 50-cc slurry-injection tank was evacuated by means of an aspirator. The injection tank was then filled with D_20 or slurry from the slurry reservoir. Pressure was then applied to the slurry-injection tank from the high-pressure oxygen-metering tank such that the pressure of the metering-tank - injection-tank system was several hundred pounds above that in the loop. By opening



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Fig. 11. LITR Fuel Lattice Pattern

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Fig. 12. Cutaway Drawing of In-Pile Loop Facility.

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Fig. 14. Sampling System, In-Pile Slurry Loop.

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Fig. 15. Addition System, In-Pile Slurry Loop.

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values 66 and 3, material in the injection tank was forced into the loop, and from the pressure-volume relationship the quantity of material forced into the loop was controlled by closing value 3 when the pressure in the injection tank reached the predetermined value. An exact measure of the quantity added to the loop was then obtained by venting the injection tank, evacuating with the aspirator, and measuring the volume of fluid (from the reservoir) required to refill the injection tank.

2.2.3 Slurry Dump Tank

The slurry dump tank (Fig. 16) was fabricated from 6-in. sched-80 pipe and had an internal volume of 4 liters. This compared with the loop volume of 1.5 liters. A sintered-metal filter tube was installed in the dump tank and was attached to the discharge opening. The filter was used to prevent loss of thoria-urania solids by discharge out the drain line. A cylindrical metal tube placed around the filter served a dual purpose. First, it served as a dip tube through which supernatant water could be discarded from time to time to ensure that there was always adequate space in the tank (at least 2 liters) to contain the entire loop contents (~1.5 liters) in case of an emergency dump. Second, the tube provided structural support for the filter and minimized plugging by shielding the surface from spattering when slurry was dumped into the tank.

2.3 Instrumentation

Instrumentation associated with the in-pile slurry loop experiment was designed to perform several different functions: (1) maintain loop operating conditions within prescribed limits, (2) provide information about conditions existing throughout the loop and its associated auxiliary equipment, and (3) take corrective action automatically when abnormal or unsafe conditions were reached. In order to perform these functions adequately, the instruments and control circuits must be extremely reliable, particularly with regard to the reactor safety interlocks, and must have a high degree of precision for measurement of the more critical operating variables. Some of the more important features are discussed below.

2.3.1 Temperature Measurement and Control

Temperatures of the main loop stream and pressurizer were maintained by the use of automatically controlled air-operated Variacs which supplied power to the loop and pressurizer heaters. Fourteen thermocouples were located around the loop for temperature monitoring and control as shown in Fig. 17. Thermocouple 13, attached to the loop piping at the pump discharge, was used to control the loop heater-power supply; thermocouple 8, attached to the 1/4-in. line between the pressurizer heater and pressurizer, controlled the pressurizer heater power for pressurizer temperature control.

For precision measurements of the pressurizer temperature, three thermocouples, two of iron-Constantan and one of Chromel-Alumel, were placed in a thermocouple well in the pressurizer. Readings from these thermocouples were made by means of a precision potentiometer and on expanded-scale temperature recorders.

2.3.2 Pressure Measurement

Three strain-gage pressure cells were used to indicate the total loop pressure. Loop pressure consisted of the saturated-steam pressure plus excess oxygen gas pressure plus the radiolytic gas pressure generated when the reactor was in operation. For a precise determination of the oxygen and radiolytic gases existing in the loop, a precision pressure indicator was used which, in conjunction with the precision pressurizer-temperature measurements, provided a means of calculating the gas partial pressures.



Fig. 16. Slurry Dump Tank

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Fig. 17. L-2-27S In-Pile Slurry Loop Thermocouple Locations

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The steam pressure was determined from the precision temperature measurements of the D_2O in the pressurizer. Radiolytic gas pressure was determined from the increase in pressure which resulted when the loop was placed in the neutron flux, and this pressure was assumed constant throughout each period of irradiation. The radiolytic gas pressure was rechecked at each reactor startup and/or shutdown. Excess oxygen pressure was calculated by subtracting steam and radiolytic pressures from the precision measurements of total loop pressure.

2.3.3 Heater-Power Measurements

A knowledge of the total quantity of electrical heat supplied to the various loop heaters and to the pump provided a great deal of information about the operation of the experiment. For example, the difference between the summation of the loop and pressurizer-heater powers with the reactor shut down and with the reactor at power was used to determine the amount of fission and gamma heat generated within the loop. The pressurizer heater power also provided a qualitative measure of the rate of fluid flow through the pressurizer, since a constant temperature difference was maintained between the loop and pressurizer. The pump power provided information about the performance of the pump and was also used to estimate the quantity of thorium in circulation (Sec. 2.1.2). All heater-power measurements were monitored by means of indicating and recording instruments, and further, watt-hour meters were used for precise measurements.

2.3.4 Radiation Monitors

Six radiation monitors were located at various points in the loop facility to detect the escape of radioactive material. These detectors also monitored the radiation level in the small and large equipment chambers during operations such as the removal of radioactive slurry samples from the loop. Radiation levels measured by all six of the monitors were continuously indicated throughout the inpile loop operation. Four of the detectors were ion-chamber gamma monitors located in the small equipment chamber, one being close to each of the four capillary tubes that were connected directly to the loop. The fifth monitor was a scintillation gamma detector used to detect any leakage of material from the loop by monitoring a continuous loop-container air sweep. A BF₃ thermal-neutron detector was mounted in the closure door of the small equipment chamber, directly in line with the reactor beam hole, in order to detect any leakage of thermal neutrons.

2.3.5 Reactor Safety Interlocks

All reactor safety interlocks and alarm circuits were designed primarily to minimize the possibility of uncontrolled release of radioactive material. This hazard is increased when the loop operation deviates from prescribed conditions. Figure 18 is a diagrammatic presentation of the reactor safety interlocks which would be actuated either by a service failure or a loop component failure or malfunction. These interlocks are discussed briefly below.

Rupture of the loop with resultant discharge of the entire loop contents into the loop container or equipment chambers was considered one of the most serious hazards. This hazard would be increased if the design temperature and pressure were exceeded, or if concentrations of radiolytic gas in the loop reached explosive proportions. Where operating limits were exceeded, the safety circuit was designed to reduce reactor power, the primary benefit of which was to stop the generation of radiolytic gas.

Loss of flow in the main loop circuit with resultant overheating of the core section would be caused by pump malfunction or failure. For this reason the safety circuit was designed to reduce reactor power if the pump power became excessively high or low.

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Fig. 18. Schematic Diagram of Reactor Safety Interlocks.

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Excessive concentrations of radiolytic gas in the pressurizer vapor space could detonate and could cause rupture of the loop. Steam diluent is effective in preventing this reaction, and for the heavy-water system in the slurry loop (saturated D_2O vapor, $D_2 + O_2$) no reaction occurs if the mol % steam is ~80% or greater.⁹ Thus the safety circuits were designed to prevent the formation of potential detonation mixtures in the loop and/or pressurizer by (1) loss of pressurizer temperature with resultant loss of steam diluent, and (2) increased radiolytic gas pressure in the pressurizer and loop main stream. This was accomplished by automatic reactor power reduction in the event of (1) failure of the pressurizer heater, (2) decrease in the pressurizer and/or loop temperature, and (3) increase in the loop pressure. This action stopped formation of the radiolytic gas produced by decomposition of the heavy water under reactor irradiation.

In addition to the automatic controls and safety interlooks, the experiment was continuously manned by experienced personnel on a 24-hr, 7-day-per-week basis. Each reactor power-reduction signal was preceded by an alarm, and reactor setback occurred only when the loop did not respond to corrective action by the operating personnel.

3. LOOP TESTS AND OPERATION, OUT-OF-PILE

Before the loop package was considered acceptable for in-pile operation, it underwent extensive testing in a mockup of the in-pile facility. The loop was also operated for 905 hr at temperatures and pressures corresponding to those proposed for the in-pile experiment. Tests of the various operations, procedures, and components were carried out both with water and the thoria-urania slurry projected for in-pile test. Significant aspects of this out-of-pile test program are discussed below.

3.1 Pressure and Leak Tests

After final assembly the loop was subjected to a hydraulic pressure test of 2300 psia at room temperature (operating pressure in-pile = ~1400 psia at 280°C in the loop, 295°C in the pressurizer). Following this a helium leak test was performed by charging the loop with 350 psia of helium and probing with a Consolidated Engineering Corporation leak detector, Model 24-101A. No leaks were found.

3.2 Temperature-Pressure Calibration

In order to establish the temperaturé-pressure relationship of the thermocouples and pressure cells used to measure the pressurizer temperature and pressure, a "steam calibration" was made by charging the loop, under vacuum, with D_2O and heating up to operating conditions (280°C in the loop, 295°C in the pressurizer). The indicated pressurizer pressure and temperature were compared with the data for D_2O .¹⁰ This calibration was used to establish saturation data from which oxygen and radiolytic gas partial pressures could be calculated when operating in-pile. The following results were obtained and the indicated corrections were applied for all subsequent operating periods, both out-of-pile and in-pile.

Pressurizer Temperature as Indicated by:

C-A thermocouple, TC-9 = 296.07°C I-C thermocouple, TC-10 = 294.59°C Indicated pressurizer pressure = 1196.8 psia Saturation temperature of D_pO corresponding to 1196.8 psia = 295.14°C Correction:

TC-9, -0.93°C TC-10, +0.55°C

3.3 Flow Rates

The flow rate in the loop was determined by measuring the pressure drop across the loop core section and line-sample holder. The pressure drop - flow relationship for each of these components was established before final assembly of the loop.

The pressurizer flow rate was determined from a heat balance around the pressurizer, pressurizer-heater circuit. Results are shown below.

Loop flow	5.6	gpm (CO ()
Pressurizer flow	0.11	gpm (6.8 cc/sec)
Velocities:	•	
Core (1/2-in. sched-40 pipe)	5.9	fps
Loop (3/8-in. sched-40 pipe)	9.4	fps
Filter (0.406 in. ID) 1	.3.8	fps
Test specimens:		
High-velocity 2	2.4	fps
Low-velocity	8.2	fps
		-

3.4 Loop Operation with Slurry

The loop was charged initially with D_2O and ~100 psi of oxygen gas and operated at temperatures of 280°C in the main stream and 295°C in the pressurizer. After 50 hr of operation with D_2O and oxygen, sufficient thoria-urania (batch DT-22) was added to bring the concentration of thorium in circulation to 1239 g of Th per kg of D_2O .

Loop operation with slurry was then continued for a total of 855 hr under these conditions of temperature and pressure. During this period additions of thorium oxide were made to the loop to test the addition system proposed for in-pile operation, and 10 samples of the slurry were taken to determine circulating inventory and to test the sampling system and procedures to be used in-pile. The concentration of the thorium oxide in the loop throughout this period is tabulated in Table 1 and plotted in Fig. 19. In both Table 1 and Fig. 19 values obtained from the 10 samples are compared with those calculated from book inventory. The results indicate that essentially all thoria charged to the loop was being circulated.

Over-all corrosion of the stainless steel surfaces of the loop exposed to the circulating slurry and to the thoria-free filtrate was calculated from the amount of oxygen consumed in the corrosion process and also from analyses of the slurry samples for corrosion-product iron, chromium, and nickel. The increase in the corrosion products of stainless steel in the slurry was at a steady rate as shown in Fig. 20.

The loop main-stream area exposed to slurry represented about one half the total area exposed to slurry and filtrate combined. The area exposed to slurry only was used to calculate corrosion from iron and nickel oxides which were insoluble, since it was assumed that the insoluble iron and nickel oxides formed in the thoria-free pressurizer circuit would not be transferred to the slurry in the loop main stream where the samples were taken. Conversely, the oxygen consumption and soluble chromium were assumed to represent corrosion of the total loop and pressurizer areas since they would be readily dispersed throughout the system.

	То †а]	Cć					
Sample Number	Circulating Time* (hr)	Bcok Inventory at Start cf Sample	Bas Sample Density	ed on Analysis Analytical	Deviation of Analytical from Book (%!	$\frac{Particl}{(\bar{d}_g)}$	Le Size (σ _g)
3 4 5 6** 7** 8 9** 11 12 15	98.7 204.7 235.0 256.5 328.0 377.0 497.0 518.8 547.4 835.6	1239 1209 1221 1149 1102 1056 1006 927 1443 1538	1112 1224 1260 1192 1210 1176 1077 1009 1413 1559	1346 1219 1254 1191 1131 1030 967 1406 1530	+8.7 +8.2 +2.5 +3.7 +2.1 +7.1 +2.5 +4.3 -2.5 -0.5	1.7 1.5 1.5 1.7 1.6 1.7 1.7 1.4 1.7	1.4 1.4 1.3 1.4 1.4 1.4 1.4 1.6 1.5 1.4

Table 1. Thorium Concentration and Particle Size During Out-of-Pile Operation

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*First slurry addition was at 50 hr. **Samples removed by means of a mcckup of the in-pile sampling system.

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Fig. 19. Thorium Concentration vs Circulation Time During Out-of-Pile Operation.

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Fig. 20. Accumulation of Corrosion Froducts in Slurry During Out-of-vile Operation.

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Because of the many special tests such as measurements of the activity of the Pd catalyst used for recombination of radiolytic gas (Sec. 3.5), it was not possible to determine the total oxygen consumption throughout the entire period of mockup operation. However, oxygen consumption measurements and corresponding corrosion rates for some limited periods of operation were obtained.

On these bases the corrosion rate for the 855 hr of operation with slurry was estimated to be between 0.5 to 1.0 mpy (mil per year) for the stainless steel surface area exposed to circulating slurry at 280°C and at a flow rate of ~9 fps. Further details of the corrosion by the thoria-urania slurry (in D_2O) in the inpile pump loop will be discussed further in a report to be issued after examination of the loop components and corrosion test specimens has been completed.

Particle-size degradation of the thoria-urania during the mockup run was slight. The initial and final mean particle sizes were 1.7 μ , and values ranged between 1.7 and 1.4 with no particular trend.

The rate of thoria-free filtrate flow to the pressurizer, measured periodically throughout the run, is shown in Fig. 21. It can be seen that this flow stayed fairly constant in the range of 6 to 7 cc/sec until a relatively large quantity of palladium oxide catalyst (790 mg) was added to the loop. This addition resulted in a substantial pressurizer flow decrease from 6.5 to 3.5 cc/sec. The palladium oxide catalyst mean particle size ($<0.1 \mu$) was less than that of the circulating thoria, and this small particle-size material probably caused the decrease in flow by plugging of the filter.

3.5 Catalyst-Activity Measurements

Some 20 measurements of the activity of the Pd catalyst for recombining radiolytic gas were made, and the results are plotted in Fig. 22. All activity values were calculated from the rate of pressure decay following a batchwise addition of D_2 to the pressurizer vapor space in which an excess amount of oxygen was maintained. For the slurry in-pile loop, recombination presumably occurred only in the main loop, which contained the slurry and catalyst. Previous calculations had indicated that the flow rate between loop and pressurizer was sufficient so that recombination kinetics were controlling when D_2 gas was introduced into the pressurizer vapor space. However, an induction period of several minutes was required before steady-state conditions were reached.

The catalytic activity was calculated as follows: the rate of pressure-decay, following an induction period of several minutes, was graphed on semilog paper and a straight line could readily be drawn through the data. A first-order system rate constant, k_{π} , was determined from the slope:

$$k_{\pi} = \frac{2.3 \,\mathrm{d} \log \,\mathrm{p}}{\mathrm{d}t} \,, \,\mathrm{hr}^{-1} \,.$$
 (1)

The catalytic activity was then calculated from the conversion relationship:

Catalytic activity =
$$\frac{\binom{n/p}{P_{ref}}}{V}$$
 (2)





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Fig. 22. Catalytic Activity of Pd in Slurry Loop at 280° C vs Pumping Time

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where:

Catalytic activity = catalytic activity expressed as moles of D₂ recombined per hr per liter of slurry at 100 psi D₂ pressure;

n/p = mole-pressure ratio; i.e., moles of gas required to raise the loop pressure l psi;

 $P_{ref} = constant reference pressure (100 psi of D₂ gas);$

 $k_{\pi} = (1)$ above; and

V we volume of slurry in loop at operating conditions (liters).

Equations (1) and (2) assume that first-order kinetics are controlling.

As shown in Fig. 22, the rate of recombination increased (as expected) each time Pd catalyst was added to the loop. A similar (and unexpected) increase in the recombination rate also was seen when an addition of thoria-urania was made.

The data indicated that the Pd catalyst was sufficiently active for recombination of the radiolytic gas generation expected in-pile (i.e., the concentration of gas could be held below detonation proportions). However, because of the decrease in activity with pumping time ($t_{1/2}$ of ~260 hr), additions of fresh catalyst would probably be necessary for long-term in-pile operation (i.e., 1000 hr or more).

4. LOOP OPERATION, IN-PILE

After satisfactory completion of the mockup testing, the loop was shut down, disconnected from the auxiliary equipment and instrumentation, and transported to the LITR HB-2 facility. The slurry circulated in the loop in the mockup tests was not removed.

4.1 Loop Test and Operation at the Reactor Prior to Reactor Installation

Before installation in the reactor beam hole the loop was connected to the instrument panel and auxiliary equipment and operated at temperature and pressure for 85 hr as a check of the in-pile facility instrumentation and equipment. During this period two samples were taken to test the sampling and addition systems and procedures. The volume removed in sampling was replaced with D₂O. Another catalyst addition (0.09 g of Pd) was also made at this time; this brought the total quantity of catalyst in the loop to 0.02 m Pd (1.4 g total Pd). The catalyst activity was again measured by batchwise additions of D₂ gas to the pressurizer vapor space (refer to Sec. 3.5). The catalyst activity obtained in this manner was 0.00043 moles of D₂ per hr per liter per g of Th and was calculated to give a steady-state partial pressure of radiolytic gas (D₂ + 1/2 O₂) of 11 psi at the expected level of irradiation; well below detonation limits.

The loop was installed in beam hole HB-2 of the LITR on July 18, 1960, and slurry circulation at 280°C was initiated.

4.2 Slurry Circulation

At the start of in-pile operation in beam hole HB-2, the loop contained 1008 g of solids (881 g of Th), 3.9 g of UO₂ (3.4 g of U, 91% U²³⁵), 1.4 g of Pd, and 1226 g of D₂O. The total loop volume at operating temperature and pressure was 1674 cc, but by virtue of the filter the solids were confined to the 900-cc volume of the loop main stream (refer to Fig. 1 and Table A3). Thus the slurry concentration in the loop main stream was initially at 1337 g of Th per kg of D₂O (979 g of Th per high-temperature [280°C] liter of slurry).

Slurry was continuously circulated in-pile until October 19, 1960, when the slurry remaining in the loop was drained into the slurry dump tank. Loop operation is summarized below.

*Slurry contained 1.4 g of Pd as a radiolytic-gas combination catalyst.

During in-pile operation the quantity of solids was reduced in a step-wise fashion by sampling, since the volume of slurry removed was replaced with D₂O.

The inventory concentration of thorium throughout the 3160 hr of loop operation for both out-of-pile and in-pile periods is shown in Fig. 23. For comparison, the circulating concentration determined from chemical analyses of slurry samples removed is also indicated. It can be seen that there is good agreement between the concentration based on inventory and sample analyses for the out-of-pile period in the mockup run as previously indicated (Sec. 3.4, Table 1, and Fig. 19). However, for the operation at the reactor site, both prior to and after installation in the experimental beam hole, analyses of the slurry samples indicated that the circulating concentration of thorium was less than that predicted by book inventory.

There were other criteria used to determine the circulating concentration of thoria. These were (1) pump power and (2) fission and gamma heat. The dependence of pump power on thorium concentration at a loop temperature of 280°C is shown in Fig. 6 and indicates an increase of 100 w (900 to 1000 w) as the thorium concentration was increased from zero to 1550 g of Th per kg of D₂O. The data of Fig. 6 (Sec. 2.1.2) were obtained with the pump power recorder in the mockup facility. For the pump power recorder at the LITR, a 65-w difference in absolute pump power as compared to the mockup recorder was found, but the range of 100 w (965 to 1065 w) for a concentration range of 1550 g of Th per kg of D_2O was found to hold. Although the pump power was considered to be only a qualitative indication of the concentration of thoria in circulation, it indicated that some of the thoria present in the loop was not circulating during the in-pile operation. A comparison of the thoria concentration estimated from the pump power and calculated from inventory for the in-pile period is shown in Fig. 24. Based on pump power, it appeared that the circulating concentration was at or near inventory when the loop operation was first started at the LITR during the out-of-pile test period prior to sample L-2-27S-lP. However, after this first sample the pump power indicated a concentration less than inventory and thus the major loss of thoria-urania from circulation, based on pump

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Fig. 24. Comparison of Thorium Concentration Based on Inventory and Estimated from Pump Power (Loop at 280° C).

power, may have occurred at this time, which was before reactor irradiation commenced. Since factors other than the slurry density can affect pump power, this evidence alone could not be considered conclusive regarding the quantity of solids in circulation.

The combined fission and gamma heat generated in the loop core section was also followed throughout the run. All measurements were in the range of 950 to 1150 w and could not be correlated with other observations of changes in the circulating concentration of Th. Since only about 150 w of this could be attributed to fissioning of the U^{235} , only very gross effects could be seen; i.e., if all the thoria and its associated uranium deposited in the core cr, conversely, none of the thoria circulated through the core. Thus no meaningful conclusions regarding the quantity of thoria in circulation could be drawn from the fission and gamma heat measurements.

The data from sample analyses and pump power indicate that part of the thoriaurania was not in circulation. However, as discussed later (Sec. 4.8.2) there appeared to be no deposits or cakes formed in the loop piping--at least none which could not be readily rinsed out. It may be that a part of the solids migrated to, and were retained in, the pump-rotor cavity (~160 cc volume). The pump purge was designed to prevent this from happening, but the low pump purge flow existing for most of the in-pile period may have been insufficient for this purpose. It is hoped that additional information concerning any deposits of thoria within the loop will be found when the loop is dismantled. Such information is to be reported later.

4.3 Sampling and Additions

Fourteen samples of the slurry circulating in the loop main stream were removed from the loop; two samples were removed before loop irradiation commenced; six were removed during the period of reactor irradiation; and six terminal samples were taken just prior to removal of the loop from the beam hole. In the normal sampling operation, 38.8 cc of slurry per sample was removed from the loop. Most of this material was discarded to the slurry dump tank in purging the sample lines, and only about 12 cc of slurry was transferred to the external sample tank for chemical analysis. Two samples of larger volume, taken at the end of the run, contained about 70 cc of slurry. To obtain these samples required a total withdrawal of 174.6 cc of slurry from the loop.

Following each sampling operation the volume of slurry removed from the loop was replaced with D_2O by means of the addition system. At the end of the period of reactor irradiation, three tracer compounds (i.e., beryllium oxalate, yttrium oxide, and a thorium oxide slurry, batch $MO-5^4$) were added to the loop. Three of the six terminal samples were taken after the addition of all of these tracers. These tracer additions with subsequent sampling were an attempt to obtain better information on the quantity of thorium in circulation. Concentration of thorium based on sample analyses is indicated in Fig. 23 along with that based on inventory.

When the loop was transferred to the X-10 site, it contained llll g of solids. The two samples taken prior to installation in beam hole HB-2 reduced this to 1008 g at the start of in-pile operation. The subsequent 12 samples further reduced the quantity of solids to 377 g at the time the loop was dumped. Of the 734 g of solids removed during sampling, 455 g were sent to the slurry dump tank, and the 377 g remaining in the loop at the end of the experiment were also sent to the slurry dump tank. Thus 832 g of solids were transferred to the slurry dump tank, and 289 g were sent to the laboratory for analysis.

Table A5 of the appendix summarizes all sampling and addition operations, along with the corresponding loop inventory.

4.4 Oxygen Inventory and Generalized Loop Corrosion Rate

During in-pile operation oxygen was consumed by corrosion of the loop surfaces, which resulted in a decrease in the quantity (and pressure) of oxygen in the loop. A total of 4563 cc (STP) of oxygen was added to the loop in eight oxygen additions to replace that consumed in corrosion and to maintain an oxygen overpressure in the loop. In order to relate the decrease in oxygen partial pressure to the quantity of oxygen consumed, it was necessary to know the volume of oxygen equivalent to a given change in partial pressure. For this purpose an "oxygen factor" was used. The "oxygen factor" is defined as the cc's (STP) of oxygen required to change the oxygen pressure in the pressurizer by 1 psi. It was obtained by dividing the total volume of oxygen in the loop by the oxygen pressure at operating temperature. The oxygen factor was rechecked at each oxygen addition by dividing the quantity of oxygen added to the loop by the oxygen-pressure increase which resulted. The exact amount of oxygen added to the loop was obtained by careful metering from the metering tank.

The oxygen factor is, of course, dependent upon the quantity of liquid in the loop, the solubility of oxygen in the liquid, and the volume of the pressurizer vapor space. Meaningful measurements can only be obtained under equilibrium conditions of temperature and pressure. If all these conditions remain constant throughout the run, no change in the oxygen factor should be observed. However, due to changes in the loop inventory (e.g., from sampling and additions) the factor changed from time to time, and this change was taken into account in calculating the oxygen consumed. Oxygen factors calculated for each oxygen addition during the in-pile operation are shown in Fig. 25. The value shown for each period of time is an average of all factors measured in the period. The oxygen consumption throughout the in-pile operation as computed from these factors is plotted against operating hours in Fig. 26. The slope of the plot is an indication of the over-all loop corrosion rate, and a reference slope for a rate of one mil per year is indicated on the graph.

The rate of oxygen consumption during in-pile operation is shown in Fig. 26 and indicates a generalized loop corrosion rate of 0.7 mpy. It is to be noted in Fig. 26 that the initial corrosion rate was higher than that observed during the latter part of the run.

4.5 Mechanical Performance

For both out-of-pile and in-pile operating periods the loop was operated for a total of 3265 hr at 280°C, during which slurry was circulated for 3118 hr. No mechanical difficulties of any description were encountered during these operating periods with the exception of the decrease in filtrate flow to the pressurizer observed in-pile. The loop remained leak-tight throughout its operating history, and no escape of radioactive material was observed from the loop itself or during the removal of slurry samples and additions of D_2O , tracer compounds, and oxygen gas.

4.5.1 Pump

Pump operation was without incident, and there was no evidence of excessive bearing friction and wear or malfunction of the electrical stator.

4.5.2 Sintered-Metal Filter

For the out-of-pile operating period the sintered-metal filter exhibited very little, if any, tendency to plug and reduce the pressurizer flow rate except for the flow loss encountered when the last two additions of palladium catalyst were



Fig. 25. Oxygen Factor (accumulative)

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Fig. 26. Oxygen Consumed During In-Pile Operation of Slurry Loop L-2-27S

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made. However, immediately upon exposure to reactor irradiation, it was noted that the flow rate of filtrate began decreasing and was reduced from a rate of 3.5 cc/sec to 1 cc/sec during the first 10 hr of reactor irradition. Thereafter the flow rate drifted downward for the next 150 hr to 0.7 cc/sec, and for the remainder of the run remained at about 0.5 cc/sec as shown in Fig. 27. The reason for the rapid reduction of pressurizer flow under irradiation is unknown; however, it is speculated that it is due to either (1) corrosion-product accumulation in the filter pores or (2) particle-size degradation of the thoria with resultant plugging of the sintered-metal pores with the smaller thoria particles. In any event, this decrease in pressurizer flow did not preclude continued loop operation.

4.5.3 Sampling System

No difficulty was encountered with the slurry sampling system. A substantial quantity of slurry was obtained in each of the standard-size samples (~12 cc). Larger tanks were used for two samples, and ~70 cc of slurry was obtained in each of these. All slurry samples removed from the loop were successfully transferred to hot cells and readily removed from the sample tank by remote procedures.

4.5.4 Addition System

The addition system and procedures proved to be highly satisfactory, and not only was D_2O added to the loop, but three additions of tracer materials (solids . dispersed in D_2O) were made to the loop while operating at temperature and pressure.

4.5.5 Valves

Fifty-two valves designed for operation at pressures to 30,000 psi were incorporated in the loop facility to perform the various operations such as slurry sampling, oxygen additions, loop draining, etc. All valves were identical except that in some a nonrotating stem design was used, and in others the stem rotated in opening and closing. Based on previous experience, valves of the nonrotatingstem type were specified for dry-gas service, while the rotating-stem design was used in slurry service. All valve bodies and seats were of austenitic stainless steel (type 304); however, three different stem materials were used: type 420 stainless steel partially hardened, type 17-4 PH stainless steel (cond. H-1000), and Stellite-6. Only one valve failure, valve 40, in the line used for oxygen additions, occurred during in-pile operation; this failure probably resulted from the fact that a rotating-stem design was inadvertently used in lieu of the preferred nonrotating type for the dry-gas service for which valve 40 was used. This did not preclude making further oxygen additions to the loop but did require special care to balance the pressures across the valve seat to minimize leakage of oxygen during each addition. A tabulation of all valves incorporated in the in-pile slurry loop facility and their operating history is given in Table A6 of the appendix.

4.5.6 Thermocouples and Instrumentation

Thirteen iron-Constantan glass-insulated thermocouples and one Chromel-Alumel glass-insulated thermocouple were attached to the loop to measure and control operating temperatures of the main loop stream and the pressurizer. There were no thermocouple failures during the run. The three thermocouples (two iron-Constantan and one Chromel-Alumel) located in the thermocouple well in the pressurizer were initially calibrated as described in Sec. 3.2. Six iron-Constantan thermocouples were attached to the main loop piping; three of these were located on the loop core section and were subjected to the highest level of reactor radiation. No direct calibration of these thermocouples was made except to compare the six thermocouple readings with the loop at elevated temperature.







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Before installing the loop container all six of these loop thermocouples agreed to within $\pm 1^{\circ}$ C. After installing the container, the core-inlet thermocouple (TC-4) decreased about 4° C, and this was attributed to its close proximity to the water-cooled container. As a check on the reliability and stability of these thermocouples, temperature readings obtained at several different periods throughout the in-pile operation are shown in Table 2, and from these data it can be seen that the thermocouple readings did not change significantly during the 2220 hr of in-pile operation. For example, a comparison of several thermocouple readings made just prior to reactor startup and at termination of the experiment show a maximum deviation of initial and final readings of $\pm 2.1^{\circ}$ C (core inlet temperature) as shown in Table 3.

Steady-state operating conditions in the loop were maintained by controlling the temperature of the main loop stream and the pressurizer. Control of these temperatures was achieved by the use of air-operated Variacs which supply power to the loop and pressurizer heaters. Instruments of various types were also used to indicate temperatures around the loop circuit and to indicate and record variables such as pressure, pump power, heater power, and temperatures and pressures of the auxiliary equipment. For safety protection, the more critical variables were interlocked with the reactor in such a manner that a reactor setback would occur if safe operating limits were exceeded. The performance of the loop control instrumentation was exceptionally good. Operating conditions within the loop were maintained at the desired levels throughout the 2305 hr of continuous in-pile operation. Reliability of the instruments was achieved by frequent inspection and servicing, particularly during the periods when the reactor was shut down. Although 17 instruments, including six radiation monitors, were interlocked with the reactor, no reactor setbacks occurred as a result of instrument malfunction.

4.5.7 Unscheduled Reactor Shutdowns

There were three unscheduled reactor shutdowns caused by the operation of the in-pile slurry loop, and these are described below:

1. A fast setback and reactor down period of 0.3 hr occurred because of low flow rate of coolant water to the loop shield plug. The low flow rate was caused by an accumulation of rust and dirt in the coolant water outlet line. After the plug was cleared, additional filters were installed in the water line, and no further difficulty was encountered.

2. A momentary building power fluctuation actuated a relay in the pump electrical circuit which caused a reactor setback which resulted in 2.5 hr of reactor down-time.

3. One setback was caused by one of the radiation detectors when power to one of the detectors was accidentally turned off. Reactor down-time was 0.4 hr.

4.6 Radiation Effects

Several effects of reactor radiation on the loop and circulating slurry are readily measurable while the experiment is operating in-pile, and continuous knowledge of these effects is useful in following the progress of the experiment. The major effects which were continuously measured were (1) radiation-induced fission and gamma heat in the loop-core section, (2) oxygen consumption, (3) the concentration of radiolytic gas, and (4) radiation intensity of the slurry as measured when removing a slurry sample from the loop.

· · · · · · · · · · · · · · · · · · ·	Temperatures with Reactor at Zero Power (°C)				ower (°C)	Temperatures with Reactor at 3000 kw (°C)				
Hours Circulation:	260-290	940-990	2260-2290	972-975	1810-1829	160-260	Pump at 6 800-900	2020-2220	1752-1754	1804-1806
Pressurizer										
Htr. Temp. Cont., TC-8	295.5	292.8	297.1	293.0	292.1	295.1	292.9	296.9	295.3	295.2
Htr., TC-6	294.7	292.4	296.4	292.3	291.3	294.8	292.5	296.4	294.8	294.8
Temp. (I-C), TC-10	294.8	295.0	296.3	295.0	296.6	295.2	295.1	296.5	296.6	296.6
Temp. (I-C), TC-7	294.4	294.9	294.7	294.9	295.0	294.6	294.9	295.0	295.1	295.0
Jacket Cont., TC-11	292.0	291.7	292.0	291.5	291.7	291.9	291.7	292.0	292.0	291.5
Temp. (C-A), TC-9	295.7	295.9	295.7	295.9	296.1	295.8	295.9	295.9	296.2	296.1
Loop Temp. Cont., TC-13	280.1	280.1	280.0	280 . 0	280.1	280.1	280.0	279.9	279.8	280.0
Core Temperature										
TC-5	281.6	281.5	282.L	281.4	282.0	234.9	285.1	287.5	285.6	285.6
TC-12	277.7	277.3	277.8	277.5	277.6	235.8	286.6	287.3	286.2	286.5
TC-2	278.3	278.4	278.9	278.0	278.8	233.3	284.2	285.4	284.4	. 284.5
Inlet, TC-4	. 276.0	273.8	274.1	273.8	271.3	276.9	275.9	274.1 .	274.7	274.6
Outlet, TC-3	278.9	277.1	277.4	277.0	277.7	230.0	279.0	277.0	279.0	278.4
Pump Temperature, TC-1	56.2	54.0	58.1	62.0	63.3	55.4	60.3	58.5	65.0	66.0
Pump Power										
Watts	1016.0	1029.4	1007.6	1780.0	1726.0	1046.1	1024.5	995.1	1743.0	1740.0
Volts	220	220	220	294	294	220	220	220	294	294
Amps/Phase	5.3	5.3	5.3	5•7	5•7	5.3	5.3	5.3	5.7	5.7

Table	2.	Typical	Operating	Temperatures,	L-2-27S*
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*Refer to Fig. 17 for thermocouple locations.

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Auxiliary Equipment Cooling Water and Air Flow

Flow	
Plug coolant	l.l gpm process H ₂ O
Pump and liner coolant	1.4 gpm demineralized H ₂ O
Equipment Chambers	
Air flow	0.4 scfm filtered air
Vacuum	3.0 in. Hg
Loop Container	
Pressure	~10 psig
Air-sweep rate (RD-5)	0.28 scfm filtered air
Continuous air bleed	0.33 scfm process air

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Table	3.	Comparison	of t	he T	empe	erati	ire H	Reading	gs of	the	Six
	Th	ermocouples	Atta	iched	to	the	Mair	n Loop	Pipi:	ng	
Before and After In-Pile Operation											
			(F	React	or I) own))				

	Hours Circ. In-Pile	Temperature (°C)							
of Operation		Loop Temp. Control TC-13	Core Temp. TC-12	Core Temp. TC-5	Core Temp. TC-2	Core Inlet TC-4	Core Outlet TC-3		
Prior to reactor startup	11	279.9	276.8	281.0	277.9	274.2	277.8		
Prior to slurry draining	2220	279.8	277.5	282.0	278.0	273.5	276.9		
After slurry draining	2222	280 . ¼	278.0	282.5	278.7	276.3	278.5		
Maximum deviation		+0.5	+1.2	+1.5	+0. 8	+2.1	+0.7		

4.6.1 Fission and Gamma Heat

The fission and gamma heat generated in the loop core section was obtained by making heat balances around the loop before and after each reactor shutdown. All measurements were made with the loop and pressurizer at a constant temperature. Eleven measurements of the fission and gamma heat were made during the in-pile operating period and are tabulated in Table 4.

The total fission and gamma heat varied between 950 and 1150 w during the in-pile period as compared to a calculated value of 900 w based on the initial inventory of thorium and uranium. The calculated value of 900 w (fission and gamma heat) was made up of the following:

Gamma heating of core metal = 550 w Gamma heating of thoria-urania = 150 w Fission heat = 200 w

TOTAL = 900 w

Although the fission and gamma heat did not appear to correlate directly with the decrease in thoria and urania inventory by sampling, the total generation of fission and gamma heat was in the range expected and was interpreted to indicate that there was no major loss or accumulation of slurry in the loop core section.

4.6.2 Radiolytic Gas

The slurry contained a palladium catalyst for recombination of the radiolytic gas generated under reactor irradiation. Sufficient catalyst was added such that the pressure of the radiolytic gas in the pressurizer vapor space was expected to be about 10 psi at the start of in-pile operation. Based on out-of-pile measurements of the catalyst activity and the observed decay of activity with pumping time at temperature (refer to Sec. 3.5), the radiolytic gas pressure was expected to rise to about 50 psi after 1000 hr of in-pile operation. The expected rise did not occur, and the pressure of radiolytic gas remained below 10 psi during the entire run. Recombination as a result of in-pile gamma radiation¹¹ was estimated to be

Test	Circulating Time	Thorium*	T	Heater	-Power Me	asurement	ts (w)	7. Mar	<u>Total Heat (w)</u> Reactor Reacto		- Fission
No. I	In-Pile (hr)	-Pile In Loop nr) (g)	Loop	Press.	Jacket	Loop	Press.	Jacket	Down (1+2+3)	at 3 Mw (4+5+6)	Gamma (w)
	· · · · · · · · · · · · · · · · · · ·		1	2	3	4	5	6	7	8	7 - 8
l	124.5	881	1878.4	396.4	117.7	746.2	377.2	115.7	2392.5	1239.1	1150
2	261.6	881	1658.3	365.4	126.2	553.2	357•9	125.8	2152.9	1036.9	1116
3	449.1	839	1670.0	380.1	123.4	667.3	367.2	120.6	2173.5	1155.1	1018
4	597.6	839	1674.8	381.2	122.6	701.4	369.0	121.5	2178.6	1191.9	986
5	933.6	759	1510.0	350.3	125.1	576.7	334.5	124.5	1985.4	1035.7	950
6	1102.7	759	1660 . C	366.0	121.4	661.4	353.2	120.9	2147.4	1135.5	1011
7	1293.7	759	1623.0	362.5	118.0	635.5	350.8	118.5	2103.5	1104.8	998
8	1437.7	723	1680.6	364.9	119.3	699.1	353.2	119.7	2164.8	1072.0	1092
9	1604.6	687	1657.4	358.4	120.3	690.9	345.6	120.2	2136.1	1156.7	979
11	2250.6	654	1608.8	345.3	124.1	645.0	331.0	122.2	2078.2	1098.2	980

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Table 4. Fission and Gamma Heat from Total Lcop Heater-Power Measurements

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*Contained 0.5 w/o U^{235} , based on thorium.

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sufficient to account for the estimated production rate of 0.08 mole/liter-hr, using a G value¹² of 5 molecules of D_2 per 100 ev. This eliminated the necessity of making further additions of catalyst to the loop.

4.6.3 Radiation Intensities

The loop shield plug and shielding around the equipment chambers were more than adequate in preventing any significant radiation leakage from the facility. During normal operating periods, radiation intensities at the outside surface of the equipment chamber shielding were no more than background.

The major source of activity inside the equipment chambers was encountered when the highly radioactive slurry samples were removed from the loop into the equipment chambers. A measure of this activity was obtained by means of the ion chambers located inside the equipment chamber. One of these detectors, RD-3, was in contact with the 90-mil-OD (50-mil-ID) tubing through which slurry passed during sampling. After sampling, most of the slurry and associated activity was removed from this tubing by back-flushing with water. The activity level as measured by RD-3 is shown in Table 5 for two different sampling operations; one reading was taken for a sample removed about 4 hr after reactor shutdown, and the other was for a sample removed 1 min after reactor shutdown. For reasons of safety, all samples were removed only when the reactor was down.

Sample Number	Hours In-Pile	Radiation Hours at 3 Mw	Time Between Reactor Shutdown and Sample Removal	Maximum Radiation Intensity of Sample Line at Contact (RD-3)
L-2-275-4	516.4	447.5	4.4 hr	78 r/hr
L-2-27S-5	682.6	607.5	1.0 min	390 r/hr

Table 5. Radiation Intensity at Sample Line During Sampling

Radiation levels at the outside surface of the equipment chambers during sampling were negligible except for one small area. This area, representing an area approximating a 4-in. square, was at a point between the large and small equipment chambers where the lead shielding thickness had been reduced to about 1 in. (as compared to the normal 8- and 6-in. thicknesses surrounding the large and small chambers, respectively). This reduced shielding resulted from modifications necessary to install a sampling tube in the existing facility. A maximum reading of 200 mr/hr was obtained on the outside surface of the 1-in.-thick shielding when the highly radioactive slurry samples passed through the capillary tube. However, because of the small area and geometry of this section and the short residence time of slurry in the capillary tube (less than 5 min), personnel exposures were negligible.

After back-flushing the sample line, the radiation level at RD-3 dropped to about 600 mr/hr with no apparent decay for the succeeding week. When the in-pile operation was terminated and the thoria was removed from the loop by flushing, the radiation background inside the small equipment chamber was about 300 mr/hr from the radioactive material contained in the slurry dump tank.

4.7 Special Operations and Observations

Several operational phenomena occurred during in-pile operation of the slurry loop which had not been observed in the uranyl sulfate solution loop experiments previously operated,⁸ and in this respect were unique to the slurry loop.

4.7.1 Oxygen Pressure

Throughout the period of in-pile operation an oxygen overpressure was maintained in the pressurizer. Normally a gradual decrease in the oxygen pressure occurred as oxygen was consumed in corrosion. For the in-pile slurry loop this rate of decrease (from corrosion) was in the range of 1 to 3 psi/24 hr. A stepwise decrease of about 20 psi in oxygen pressure also occurred when a sample was removed from the loop because of the decrease in the loop liquid inventory. However, most of this loss was recovered when the volume removed in sampling was replaced.

Unexpectedly large decreases (40 to 100 psi) in oxygen pressure occurred during in-pile operation when samples were removed from the loop and occasionally when the loop temperature was lowered 5°C (from 280 to 275°C) to measure the pressurizer flow rate. However, the oxygen overpressure returned to its normal value after sampling when the volume removed by sampling was replaced (by a D₂O addition) and after a flow check when the loop temperature was brought back to 280°C. This unexpectedly large oxygen pressure loss was observed when the first sample, L-2-27S-3, was taken after irradiation commenced (265.9 hr of circulation) and during all subsequent sample removals unless a D_pO addition to the loop was made just prior to sampling. This excessive loss of oxygen pressure was also observed during some of the pressurizer flow checks which involved lowering of the loop temperature from 280 to 275°C and was larger than could be accounted for by the increase in vapor volume in the pressurizer. The pressure was always recovered when the temperature was returned to 280°C. This loss of oxygen pressure was not seen during the first 11 flow checks made but was encountered during flow checks 12, 13, 14, and 15 (773.7 to 948.9 hr of in-pile circulation).

Because this excessive loss occurred during operations which reduced liquid volume in the pressurizer (by sampling or lowering of loop temperature), it raised the question as to whether or not the liquid level in the pressurizer was adequate (discussed in Sec. 4.7.2). Thus it was decided to increase the D_2O inventory in the loop by about 50 cc. This was done after 968 hr of circulation and temporarily relieved the problem. No further excessive losses of oxygen pressure were observed during the pressurizer flow checks until pressurizer flow check 26 (after 1306 hr of circulation). However, subsequent to this, the loss of partial pressure was encountered for all remaining flow checks.

Although the reason for this phenomenon is not clear at present, it is thought to be related to the low flow rate in the pressurizer (Sec. 4.4.2) which substantially decreased the rate of transfer of gas between the loop and pressurizer and also made it difficult to maintain thermal equilibrium in the pressurizer. This could have caused erroneous pressure and temperature measurements¹³ when the loop temperature and pressure were upset by the sampling and flow-check operations.

Under steady-state operating conditions the low pressurizer flow rate had no apparent effect on oxygen pressure measurements. Thus it appeared that the low pressurizer flow rate affected the ability of the loop to recover from transient upsets which caused erroneous oxygen partial-pressure values during these operations. "This assumption was supported by the fact that the excessive loss of oxygen pressure during pressurizer flow checks could be prevented by increasing the pressurizer flow rate. This was done on occasion by increasing the pump speed (by operation on 80-cps power supply) which increased both the flow in the main loop stream and the pressurizer.

4.7.2 Liquid Volume in the Pressurizer

The volume of the pressurizer was 521 cc as compared to the total loop volume of 1620 cc. During in-pile operation the liquid level in the pressurizer was determined by difference between the known loop volume and slurry inventory. For proper pressurizer operation it is necessary to maintain at least 100 cc of liquid in the pressurizer at elevated temperature. This is required in order to submerge the thermocouple well in liquid for accurate temperature measurement. Further, the pressurizer outlet line must be submerged in liquid so that gas will not be pulled into the rear of the pump and cause cavitation.

Accurate knowledge of the pressurizer vapor volume is also necessary to determine the total quantity of oxygen in the loop from the oxygen pressure measurements. A direct measure of the liquid level in the pressurizer cannot be made; but in addition to the value calculated from the loop volume and slurry inventory, the vapor volume (and corresponding liquid level) was calculated each time oxygen was added to the loop.

At the start of in-pile operation the vapor volume in the pressurizer was calculated to be 212 cc (309 cc liquid volume) at operating temperature. Based on loop inventory calculations, the pressurizer vapor volume varied between 29 and 212 cc throughout the period of in-pile operation. These changes occurred as a result of sampling and additions.

Eight oxygen additions were made during the experiment from which the vapor volume in the pressurizer was calculated and compared with inventory values. A comparison of the pressurizer vapor volume determined from inventory and that calculated for each oxygen addition is shown in Table 6. It can be seen that considerable discrepancy sometimes existed between the two values thus obtained; however, it should be noted that all values show that the pressurizer contained sufficient liquid for proper loop operation (>100 cc).

Date	Circulation	Oxygen	Liquid Volume in F	Pressurizer (cc) ^{a,b}
	Time	Addition	from	from
	(hr)	Number	Oxygen Addition	Slurry Inventory
7-1-60	8.2	1	296 (225)	309 (212)
7-5-60	25.7	2	319 (202)	362 (159)
7-26-60	272.5	3	191 (330)	368 (153)
8-24-60	929.6	4	c	426 (95)
8-31-60	1120.1	5	414 (107)	429 (92)
9-30-60	1840.2	6	254 (267)	440 (81)
10-17-60	2258.3	7	427 (94)	384 (137)
10-17-60	2269.9	8	271 (250)	493 (29)

Table 6. Pressurizer Liquid Volume and Vapor Volume as Determined from Slurry Inventory and Oxygen Additions

^aTotal pressurizer volume was 521 cc. At least ~100 cc liquid required for proper operation.

Values in parentheses indicate corresponding vapor volume.

^CNot determined; leak in valve 40.

The only known source of error in the loop inventory values was the uncertainty in the volume of slurry removed in sampling. Since this volume was based on removing an amount exactly equivalent to the standard drain volume, any leakage past valves into the dump tank (not believed to have occurred) would have affected the inventory calculations. Values of pressurizer liquid level determined from oxygen additions depended on a knowledge of the amount of oxygen added, oxygen solubility in the slurry, the quantity of slurry in the loop, and measurements of the oxygen pressure in the loop. Of these, the most likely source of error was measurements of the oxygen pressure in the loop before and after each oxygen addition. As noted in Sec. 4.7.1, these oxygen pressure measurements could have been in error because of the low pressurizer flow rate.

4.7.3 Core Inlet and Outlet Temperatures

During in-pile operation the temperature difference across the core section was continuously recorded. At the start of in-pile operation the core Δt was about 4.5°C, but gradually decreased to ~1.5°C after 1200 hr of in-pile operation. This decrease was a result of a decrease in the core outlet temperature rather than an increase in the core inlet temperature. This latter temperature stayed fairly constant throughout the in-pile run.

With the reactor operating at power, the core Δt could be brought back to its initial value by increasing the slurry flow rate (by increasing the pump speed) as shown in Fig. 28. Decreasing the reactor power (shutdown) also had the same effect. This phenomenon was not completely understood, but one possible explanation is that some deposit of solids occurred in the pipe at the core exit, where there was a change in the cross section of the piping (decreasing from 1/2-in. sched-40 pipe to 3/8-in. sched-40 pipe). A deposit of solids at this point may have decreased the transfer of heat to the thermocouple located on the bottom side of the pipe. However, if slurry deposition occurred, such incremental loss from circulation was too small to be observed from the measurements of pump power, fission and gamma heat, or sampling. After return to initial values, a substantial period of time (up to one week of operation) was required before the core inlet and outlet temperatures converged again.

4.8 Loop Removal

4.8.1 Normal Slurry Draining and Flushing of Loop

At the termination of the experiment the slurry remaining in the loop was dumped into the slurry dump tank in the small equipment chamber. This was done by opening the valve in the loop drain line while the loop was operating at temperature and pressure. The pump was allowed to operate until cavitation occurred, at which time the pump was shut off, but loop draining was continued through the loop drain line (at this point the valves in the pump drain line were opened to drain any material held up in the pump-rotor cavity) until all material that could be removed was transferred to the dump tank. The loop was then refilled with D_2O , the pump turned on, and again the loop contents were dumped with the pump running. This flushing operation was repeated until the loop had been rinsed five times, at which time radiation monitors on the loop and pump drain lines indicated that no more radioactive material was being removed from the loop as shown in Fig. 29. Although no radioactive slurry was removed through the pump drain line during sampling, the radiation monitor, RD-2, on the pump drain line was close enough to the loop drain and sample line to detect radiation during sampling. The radiation intensities at RD-2 are included with those from RD-3, loop drain and sample line, as additional evidence that additional rinsing operations were not required.





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Fig. 29. Radiation Intensities at Drain Lines During Sampling, Draining and Rinsing Operations.

4.8.2 Loop Removal

No difficulty was encountered in removing the loop from the beam hole into its shielded container. After the loop was removed from the beam hole, a radiation survey of the loop was made through a 1.3/8-in. x 1.5/8-in. rectangular opening in the shielded carrier. Radiation readings were made at a number of points on the loop and indicated that substantially all slurry and associated fission products had been removed. Figure 30 is a schematic diagram of the loop showing the radiation intensities at various locations along the length of the loop. Except for the radiation intensity at the core section (>l00 r/hr), which resulted from the induced activity in the stainless steel piping, radiation levels were below 6.5 r/hr, well below the 78 r/hr readings observed for the radioactive slurry during sampling operations.

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APPENDIX

	Drawing	Area		
Component	Number	$(in.^2)$	(cm ²)	
<u>Circulating Pump</u> Scroll Impeller (Zircaloy-2) Impeller screw (Zircaloy-2) Thermal shield Rotor Housing assembly Bearing holder (front) Bearing holder (rear) Bearing spacer	TD-D-2769 TD-D-2768a TD-C-2375b TD-B-2372 TD-D-2766 TD-D-2765a TD-E-2764a TD-C-4913 TD-C-4913 TD-C-4913	36.6 33.3 0.5 31.5 66.4 94.5 23.3 22.1 34.1	236.0 214.8 3.2 203.5 428.0 610.0 153.3 142.8 220.0	
	Total	342.3	2211.6	
347 stainless steel pump area exposed to slurry at 280°C (including scroll, front of thermal shield, and section of housing from scroll weld to front of thermal shield)		56.4	364.0	
Core piping 54.8 in. of 1/2-in. sched-40	. ID-D-4983	107.5	694.0	
*Coupon holder (two, high velocity) *Coupon holder (two, low velocity)	TD-D-4983 TD-D-4983	6.5 10.5	41.8 67.7	
	Total	124.5	803.5	
Loop Piping 3/8-in. sched-40 pipe (55.5 in.) 1/2-in. sched-40 pipe (32 in.) *In-line coupon holder (high velocity) *In-line coupon holder (low velocity) Pump discharge fitting Filter unit (slurry side)	TD-F-4982 TD-F-4982 TD-D-4983 TD-D-4983 TD-C-2365 TD-C-4978	86.0 63.2 3.2 5.3 5.0 18.9	555.0 408.0 20.9 33.9 32.4 121.5	
	Total	181.6	<u>1171.7</u>	
Pressurizer Circuit Wetted surface (assuming 200 ml vapor space) Unwetted surface (assuming 200 ml vapor space) Calculated volume at pressure and	TD-E-4375 TD-E-4375	67.2 42.2	435.0 273.0	
temperature = 521.2 ml Filter unit (filtrate side)	TD-C-4978	28.3	182.5	

Table Al. Tabulation of Surface Areas of Loop Components Exposed to Slurry and $\mathrm{D}_2\mathrm{O}$

*Does not include corrosion specimens.

		Drawing	Area		
Component		Number	$(in.^2)$	(cm^2)	
Tubing					
1/4 in. x 0.049 in. wall,	126.5 in.	TD-F-4982	79.2	512.0	
$0.090 \text{ in. } \times 0.020 \text{ in. wal}$	1, 455 in.	TD-F-4982	71.6	462.0	
(Lines 2, 5, 4, and 5) 0.060 in. x 0.020 in. wal (Line 1)	l, 138 in.	TD-F-4982	8.7	56.1	
		Total	159.5	1030.1	
	SUMMARY*				
347 SS Area Exposed to Slurry			re h	7610	
Pump Core Loop piping			124.5 181.6	803.5 1171.7	
		Total	362.4	<u>2339.2</u>	
347 SS Area Exposed to D ₂ O Onl Pump Pressurizer (wetted area) Filter unit (filtrate sid Tubing (pressurizer circu discharge to rear of pu	<u>y</u> e) it from filter mp)		252.1 67.2 28.3 82.5	1629.6 435.0 182.0 535.0	
	Total 347 SS .	Area	430.1	2781.6	
Zircaloy-2 Area Exposed to Slu Pump impeller Impeller screw	rry		33•3 <u>0•5</u>	214.8 <u>3.2</u>	
	Total Zircalo	y-2 Area	<u>33.8</u>	218.0	

Table Al (continued)

*Does not include corrosion test specimens.

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Component	Drawing Number	Material	Source and/or Identification No.
Pump Assembly Housing End piece Drain nozzle Flange Bail Purge fitting	ID-D-2769 ID-E-27640	347 SS 347 SS 347 SS 347 SS 347 SS 347 SS 347 SS	1182 1247 1225 1050 1148 1195
Rotor shaft Stub shaft Journal bushings (2) (pure, fine-grained al Rotor can End piece Clamp rings Plug	TD-D-2765 umina)	347 SS 347 SS Al ₂ 0 ₃ 347 SS 347 SS 347 SS 347 SS 347 SS	1247 1040 Kearfott Co. 1179 1247 1189 414
Rear bearing housing Insert	TD-C-4913	347 SS Al ₂ 0 ₃	1247 American Lava Co. AlSiMag 652 P.O. 63V-57983-3
Front bearing housing Insert	TD-C-4913	347 SS 99.97% Al ₂ 0 ₃	1247 Kearfott Co.
Bearing spacer Thermal shield Impeller	ТД-В-2703б ТД-Д-2766 ТД-С-2375б	347 SS 347 SS Zircaloy-2 (commercial grade)	l193 1247 Oregon Metallurgical Ht. No. OMC39001
Impeller screw Scroll	TD-B-23 72 TD-D-2768A	Zircaloy-2 347 SS	1206 1150
Discharge piece Body Ends (2) Nipple	ID-C-2365	347 SS 347 SS 347 SS	1194 1195 4 <u>1</u> 4
Loop Piping 3/8-in. sched-40	TD-F-4982	347 SS	1243
Filter End, item 2 Housing, item 1 Housing, item 3 End, item 4 Bellows, item 7 Filter, item 5 Adapter, item 6	TD-C-4978	347 SS 347 SS 347 SS 347 SS 321 SS 347 SS 347 SS 347 SS	1268 HRP-200-2 1183 1245 Unknown P.O. 70Y-77013 1243

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Table A2. Tabulation of Loop Drawings and Materials

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Component	Drawing Number	Material	Source and/or Identification No.
Sample Holders			
Low velocity	<u>10-0-490)</u>	347 55	1266
High velocity		347 SS	1245
Reducer		347 SS	1125
Core			
1/2-in. sched-40 pipe	TD-D-4983	347 SS	1266
Pressurizer	TD -E- 4375		
Body		347 SS	1021
Ends (2)		347 SS	1189
Nipples		347 SS	414
Connecting tubing (1/4 in. by 0.045 in. wall)		347 SS	1261
Capillary Line	TD-F-4982		
Line 1: 0.060 in. OD by 0.020 in. ID capillary		347 SS	1259
Line 4: 0.080 in. OD by		347 SS	Unknown
Lines 2 3 and 4 num		347 SS	1222
purge line 0.090 in. capill	arv	J# 00	
Purge-cooler line: (1/4 in.	OD OD		,
by 0.035 in. wall tube)	TD-F-4986	304 SS	1256
water flow channel only			
Pressure taps: $(1/4 \text{ in. OD})$	TD-F-4982	347 SS	1261
by 0.045 in. wall) (cut	•		
off and seal-welded after	•		· .
initial flow measurements)			

4

Table A2 (continued)
Component	Volume at 28°C (cc)	Operating Co Temperature (°C)	Volume (cc)
Pump	160.0	90 [·]	160.4
Main stream	888.5	280	900.1
Pressurizer (inlet line)	17.7	287	17.9
Pressurizer (outlet line)	22.5	295	22.7
Filter Housing (effluent side	1 ⁾ +•O	280	15.2
Pressurizer	514.0	295	521.1
(liquid, nominal)		(295)	(321.1)
(vapor, nominal)		(295)	(200.0)
Circulating loop total	1616.7		1637.4
· · · · · · · · · · · · · · · · · · ·			
Pressure cell No. 1	7•7	28	7.7
Pressure cell No. 9	8.3	28	8.3
Pressure cell No. 10	7.9	28	7•9
Line 1	0.7	28	0.7
Line 2	4.1.	28	4 <u>.1</u>
Line 3	4.1	28	4.1
Line 4	3.5	. 28	3.5
Line 5	0.5	28	0.5
Total for Lines and Pressure Cells	36.8		36.8
Total Loop Volume	1653.5		1674.2

Table A3. Tabulation of Volumes of Loop Components

(Core volume measured at 28°C = 304.0)

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Holder Number, Velocity	Location	Position	Coupon Number	Material	Material Item No.	Weight (g)
l Low	Line	1 2 3 4 5 6 7 8	ZB1 TD1 SA1 ZB2 ZB3 SA2 TL1 ZB4	Zr-2 Ti-110AT 347 SS Zr-2 Zr-2 347 SS Ti-45A Zr-2	60 1176 1149 60 60 1149 122 60	1.0362 0.7072 1.1998 1.0172 1.0274 1.2012 0.7122 1.0210
2 High	Line	1 2 3 4 5 6 7 8	ZB5 TD2 SA3 ZB6 ZB7 SA4 TL2 ZB8	Zr-2 Ti-110AT 347 SS Zr-2 Zr-2 347 SS Ti-45A Zr-2	60 1176 1149 60 60 1149 122 60	1.0169 0.7129 1.1969 1.0180 1.0245 1.2024 0.7085 1.0204
3 Low	Core, high-flux	1 2 3 4 5 6 7 8	ZB9 TD-3 SA5 ZB10 ZB11 SA6 TL3 ZB12	Zr-2 Ti-110AT 347 SS Zr-w Zr-2 347 SS Ti-45A Zr-2	60 1176 1149 60 60 1149 122 60	1.0318 0.7072 1.2134 1.0124 1.0284 1.2164 0.7103 1.0286
ц High	Core, high-flux	1 2 3 4 5 6 7 8	ZB13 TD4 SA7 ZB14 ZB15 SA8 TL4 ZB16	Zr-2 Ti-llOAT 347 SS Zr-2 Zr-2 347 SS Ti-45A Zr-2	60 1176 1149 60 60 1149 122 60	1.0325 0.7063 1.2021 1.0287 1.0232 1.2160 0.7050 1.0281
5 Low	Core, low-flux	1 2 3 4 5 6 7 8	ZB17 TD5 SA9 ZB18 ZB19 SA10 TL5 ZB20	Zr-2 Ti-110AT 347 SS Zr-2 Zr-2 347 SS Ti-45A Zr-2	60 1176 1149 60 60 1149 122 60	1.0328 0.7070 1.2154 1.0200 1.0116 1.2142 0.7150 1.0221
6 High	Core, low-flux	1 2 3 4 5 6 7 8	ZB21 TD6 SA11 ZB22 ZB23 SA12 TL6 ZB24	Zr-2 Ti-110AT 347 SS Zr-2 Zr-2 347 SS Ti-45A Zr-2	60 1176 1149 60 60 1149 122 60	1.0227 0.7031 1.2169 1.0243 1.0109 1.2098 0.7083 1.0046

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Table A4. Identification and Initial Weights of Corrosion-Test Specimens

Circulation	n'	Room- Temperatur	Loo	Loop Inventory			
(hr)	Operation	Volume (cc)	D20	Total Solids	Th		
0	Received from Y-12		1175	1111	971		
8.2	D ₂ O exp. No. 1	+ 1.7	1177	1111	971		
19.2	Sample No. 1	-πS°O	1 1 37	1053	921		
. 21.8	Catalyst add. No. 1	+41.0	1182	1060	926		
25.7	D ₂ O add. No. 2	+36.1	1222	1060	926		
28.5	D ₂ 0 exp. No. 2	+ 1,4	1223	1.060	926		
47.1	D ₂ O exp. No. 3	+ 1.0	1224	1060	926		
64.6	Sample No. 2	-40.7	1186	1008	881		
66.7	D_20 add. No. 3	+35.0	1224	1008	881		
71.2	D ₂ O exp. No. 4	+ 1.2	1226	1008	881		
85.6	Loop inserted in beam	-hole HB-2		_	_		
265.9	Sample No. 3	-38.8	1189	960	839		
267.9	D_2O add. No. 4	+ 41.0	1234	960	839		
272.5	D ₂ O exp. No. 5	+ 0.9	1235	960	839		
602.0	Sample No. 4	-38.8	1197	913	798		
604.0	D_2O add. No. 5	+40.2	1242	913	798		
768.1	Sample No. 5	-38.8	1204	869	759		
770.1	D ₂ O add. No. 6	+36.8	1245	869	759		
968.5	D_2O add. No. 7	+37.5	1286	869	759		
976.0	D ₂ O exp. No. 6	+ 1.1	1287	869	759		
1120.1	$D_2 O e_{X} p$, N_{O} , 7	+ 1.0	1288	869	759		
1299.9	Sample No. 6	-38.8	1250	827	723		
1309.9	D ₂ O add. No. 8	+40.2	1295	827	723		
1440.9	Sample No. 7	-38.8	1256	787	688		
1445.6	D_2O add. No. 9	+35.5	1296	787	688		
1806.8	Sample No. 8	-38.8	1257	748	654		
1809.0	D_20 add. No. 10	+44.0	1306	748	654		
1840.2	$D_2O exp. No. 8$	+ 1.0	1307	748	654		
2258.2	Sample No. 9	-38.8	1268	712	622		
2260.2	D_20 add. No. 11	+38.6	1311	712	622		
2269.9	D ₂ O add. No. 12	+35.6	1350	712	622		
2273.8	Sample No. 10	-38.8	1311	677	592		
2275.8	D_2O add. No. 13	+40.4	1356	677	592		
2280.0	Sample No. 11-1*	-58.2	1297	626	540		
2281.1	D ₂ O add. No. 14	+50.5	1353	626	540		
2281.7	Sample No. 11-2	-58.2	1294	579	500		
2282.6	D ₂ O add. No. 15	+50.0	1349	579	500		
2283.0	Sample No. 11-5	-50.2	1290	500	460		
	Be oxalate) 10.0					
	J ₂ U rinse						
0095 0	Tracer 1 ₂ U ₃ slurry		86 0 1 775	c).c)175		
2205.0	Addition (D ₂ U rinse	1 10.0	00.0 12(2	242	417		
	NO. 17.7** MU74						
		20.0					

Table A5. Summary of Sample Volumes, Addition Volumes, and Loop Inventory, Loop L-2-27S

Circulation Time (hr)	Operation	Room- Temperature Volume (cc)	Lo D ₂ 0	oop Inventor (g) Total Solids	ry . Th
2289.5 2291.5 2291.9 2292.5 2293.0 2294.7 2297.7 2299.0 2300.5 2305.2	Sample No. 12-1* D ₂ O add. No. 16 Sample No. 12-2 D ₂ O add. No. 17 Sample No. 12-3 D ₂ O add. No. 18 Sample No. 13 D ₂ O add. No. 19 Sample No. 14 Loop drained to dump	-77.6 +50.0 -58.2 +50.0 -58.2 +46.5 -38.8 +50.0 -38.8 o tank	1296 1351 1291 1346 1286 1337 1296 1352 1311	489 489 453 453 417 417 396 396 377	427 427 395 365 365 365 346 346 328

Table A5 (continued)

*The large quantity of slurry removed for samples 11 and 12 required extra additions of D_2O to maintain the required liquid level in the pressurizer. **Tracers added = 0.195 g Y_2O_3 • .

0.072 g BeO 8.812 g ThO₂ + UO₂ $(6.77 \text{ g Th} + 0.88 \text{ g U}^{238})$

Valve	Type Stem		Type Material		Exposed to			Number of Times
Number	Ra	Иp	Body	Stem	Slurry	D ₂ 0	0 ₂	Operated
l	x		316	420		X	Χ.	23
2	Х		316	420	Х	х	Х	12
3	Х		304	420	X	х	Х	65
4		Х	304	17-4		X	. Х	
5		Х	304	17-4		\mathbf{X}_{\cdot}		
6	X		316	420	X	X	X	70
7	X		316 701	420	Х	X	X	62
9	X		304 31.6	17-4	v	X. V	X	. 10
	A V		20)	420 17 h	х	N V	N V	1.2
10	A V		316	71-4	v	X Y	л У	155
15	x X		301	420 400	A	x	л Х	±))
16	x		316	420		x	x	
17	x		316	420	х	x	x	131
īģ	X		304	17-4			X	
19		Х	304	17-4		X	х	145
20	Х		304	420	Х	х	х	112
25	Х		304	17-4		Х	х	
26	Х		316	420	,	•	х	
37		X	304	Stellite		•	X	77
38 70		X	304 701	Stellite			X	58
29	v	X	204 216	1(-4)			X	12
40),1	A V		316 316	420		v	x v	. 90
41 h2	л Y		316	420 420		л	л У	38
43	X		316	420		x	x	
44	x		30 ¹	420		x	x	65
45	x		304	420			x	43
46	Х		303	420			х	65
49	X		316	420		х		14
50 55	X X		316 316	420 420		Х	X X	38
56 57	X X		316 316	420 420			X X	123
58	Х		304	420		х	X	38
59	Х		316)+20			X	58
60	Х		304	420		х	х	42
66	Х		316	420		х	х	56
67	Х		316	420	Х	Х	Х	42
70	X		316	420		Х	Х	38
89	X		316	420		Х		14
100	X		316	420			Х	56
TOT	X	v	516 716	⊥′(-4 Ct-123+		X	v	15
102		X V	216 216	Stellite			X v	QT QT
102	v	A	510 316			v	Y	90
T04	•		010	⊥(- 4		Λ		

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Table A6. Tabulation of Valves Used in the In-Pile Slurry Loop Facility

-66-

Valve	Type	Stem	Type M	Material	Expos	ed to	02	Number of Times
Number	R ^a	N ^D	Body	Stem	Slurry	D ₂ 0		Operated ^C
124 126 127 510 511 512	X X X	X X X	316 304 316 304 304 304 304	420 420 Stellite Stellite Stellite 420		х	X X X X X	73 67 19

Table A6 (continued)

a Rotating.

^bNonrotating.

^cValves not shown operated infrequently (normally less than 10 times).

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