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ORNL
CENTRAL FILES NUMBER

58-10-114

COPY NO.

29

DATE: October 22, 1958

SUBJECT: HRP-CP: An Evaluation of the Design Features of Blanket Processing Loop P-1

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ABSTRACT

The design features and the performance of UO_2SO_4 blanket processing Loop P-1 are evaluated from an engineering viewpoint. This unique experimental development loop was operated with pump heating to study the behavior of plutonium in 1.4 M UO_2SO_4 at 250°C and was designed for mixed $\text{O}_2\text{-H}_2$ gas pressurization. The canned loop and the feed and sampling systems in glove boxes completely contained the plutonium throughout the experimental program.

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CONTENTS

1.0	SUMMARY.....	3
2.0	INTRODUCTION.....	3
3.0	LOOP FLOWSHEET DESCRIPTION.....	4
4.0	EVALUATION OF LOOP COMPONENTS.....	6
4.1	Canned Rotor Circulating Pump.....	6
4.2	Flow Restrictors.....	12
4.3	Hydroclone.....	12
4.4	Pressurizer.....	15
4.5	Bomb Sampling and Solids Addition System.....	17
4.6	Coupon Holder.....	18
4.7	Instrumentation.....	19
4.8	Valves.....	20
5.0	LOOP SAMPLING.....	21
5.1	High Pressure Generator.....	22
6.0	PLUTONIUM HANDLING.....	23
6.1	Glove Boxes.....	24
6.2	Glove Box Procedures.....	24
6.3	Plutonium Transfers.....	26
7.0	CORROSION.....	26
8.0	DESCALING OPERATIONS.....	28
9.0	LOOP HEAT BALANCE.....	29
10.0	REFERENCES.....	33
11.0	APPENDIX.....	34
11.1	O ₂ Overpressure Calculations.....	34
11.2	P-1 Loop Drawings.....	35
11.3	Tabulated Run Conditions.....	39

864 002

1.0 SUMMARY

The development studies to be made in loop P-1 required novel design features; an engineering evaluation of this loop and its operation is presented. The design temperature of 260°C with 1.4 M UO_2SO_4 was easily obtained and smoothly maintained with all heating due to degradation of mechanical energy added by an A-150-D Westinghouse canned rotor pump. Operation throughout 1500 hours was not seriously hampered by any equipment limitations or malfunctions.

Small, removable bombs with high circulation rate/volume ratios were found to be a dependable sampling and feed system. The original system for sampling and feeding was not operable due to failure of "O" ring seals on a high pressure generator.

The canned loop, and the feed and sampling system completely contained the plutonium throughout the experimental program. Glove boxes were used with an arrangement for sealing samples in plastic without exposing any of the glove box to the air or exposing the outsides of the plastic bags to the glove box. This arrangement was found superior to air locks.

Evaluation of a pressurizer design to contain both H_2 and O_2 has been reported separately. The behavior of plutonium and the performance of the hydraulic cyclone on precipitated plutonium will appear in a later report.

The loop corrosion rate was about 2 mpy during preliminary operation and during experiments with addition of $\text{Zr}(\text{SO}_4)_2$ or ZrO_2 . The corrosion rate increased to 18 to 23 mpy during studies with soluble plutonium and decreased to 10 mpy during studies with PuO_2 . The plutonium precipitated on surfaces was removed by chromous acid descaling solution with apparent corrosion rates of about 25 mpy.

2.0 INTRODUCTION

One method proposed for recovery of plutonium from a UO_2SO_4 aqueous homogeneous reactor blanket is to collect precipitated PuO_2 by use of a hydraulic cyclone. The P-1 loop was designed to study this processing method and the behavior of plutonium in 1.4 M UO_2SO_4 solution at 250°C. This report evaluates the design, the operability, and some possible design improvements for the P-1

loop and its components. Evaluation of the hazards of using a combination of O_2 and H_2 overpressure in this loop has been reported separately, and the chemical data and results will appear in a later report to be co-authored by Unit Operations and Chemical Development Section A personnel.

3.0 LOOP FLOWSHEET DESCRIPTION

The P-1 loop was designed to study the feasibility of recovering plutonium from homogeneous reactor blanket solution with a hydroclone and the solubility of plutonium in the blanket solution. In order to eliminate the effects of hot wall precipitation, the loop heat was supplied as mechanical energy by an A-150-D Westinghouse pump.

The P-1 loop system consisted of the main loop stream and three auxiliary streams (see Figure 1). The main loop stream was 110 gpm flow rate with a 200 ft pressure drop through two flow restrictors. The liquid temperature was above the wall temperature to prevent hot wall precipitation. Air cooling coils were welded to the periphery of the loop to control the operating temperature.

One auxiliary stream was connected across the pressure differential of the first flow restrictor. It contained the coupon holder and the hydroclone with a flow rate of approximately 0.6 gpm. A plug developed in this circuit near the end of operation. All attempts to remove it failed, and its exact location was never determined.

The second auxiliary stream was connected across the second flow restrictor, and it contained the solids addition bomb and valves. This stream only had flow while the loop samples were being taken.

The third auxiliary stream contained the pressurizer. The oxygen was injected into the pressurizer, and the overpressure was maintained in the vapor space. The bottom of the pressurizer was found to contain a large fraction of the corrosion products.

Three differential pressure cells were used within the loop system. One measured the total pressure differential across the pump, a second the pressure differential across the hydroclone, and a third the pressure differential across the second flow restrictor. A pressure element was located between the two flow restrictors.

FIG. 1. P-I LOOP FLOWSHEET

864 005

The P-1 loop system had two cooling circuits. A stream of air passed through the cooling coils of the main loop and controlled the loop operating temperature. The second cooling circuit was an enclosed circulating water stream to remove heat from the canned rotor pump. The circuit was enclosed to prevent contamination from leaking into the building process water stream should an internal pump leak develop.

The loop, canned rotor pump, pressurizer and part of the coupon holder were within the tank can which was maintained at 5 psia or less. This low pressure served several purposes. It provided the insulation to limit heat losses, it would reduce the pressure developed within the can if a loop leak occurred, it provided a check on loop or can leakage, and it directed can leakage inward to prevent the release of contamination.

4.0 EVALUATION OF LOOP COMPONENTS

The special requirements for the individual loop P-1 components are pointed out along with the effectiveness with which these requirements were met or the changes needed. The design was modified during construction and test operation to meet changing requirements indicated by laboratory studies and fuel solution loop operation. Three changes made were:

(a) The addition of H_2 was found to have little effect when stainless steel corrosion products were present. Therefore, the pressurizer was changed to remove the Raschig rings with their large surface area and the blast shield was left off the pressurizer.

(b) Coupons were found to be very valuable indicators of plutonium behavior; therefore, a coupon holder arrangement was added.

(c) Fuel solution loop studies indicated that bomb samples were the only dependable method of sampling a hot, high pressure loop. Also, the high pressure generator feed system appeared undependable. Therefore, a combination sampling-solids addition by use of high pressure bombs was devised and added.

4.1 Canned Rotor Circulating Pump

The A-150-D Westinghouse pump was used for two purposes: to circulate liquid through the P-1 loop system and to furnish all of the loop heating requirements.

The fluid circulated by the pump varied in concentration from demineralized water up to 1.4 molal uranyl sulfate solution. The pump maintained a 110 gpm flowrate at a 200 ft head, and the loop operating temperature of 250 to 260°C was obtained by the work input of the pump impeller.

The pump safety features were as follows: (1) a temperature element on the cooling H₂O line from the loop pump was connected to an alarm system which would shut off the loop pump power if the cooling water reached a temperature of 125°F; (2) a pressure element in the loop pump cooling water line was connected to an alarm system which would shut off the loop pump power if the pressure from the cooling H₂O centrifugal pump dropped below 15 psi.

Sealing difficulties were encountered with the two A-150-D Westinghouse pump gaskets. The pump was equipped initially with two hollow stainless steel O-rings between the thermal barrier and stator assembly, and the pump casing and the stator assembly. From previous pump experience the hollow O-rings were believed to be unsatisfactory. The thermal barrier O-ring was replaced successfully with a teflon gasket, and the pump casing O-ring was replaced with a gold wire, 80 mils in diameter. After thermal cycling the gold wire gasket continued to flatten allowing the torque on the pump casing bolts to drop from 300 to approximately 50 ft-lb. The gold wire was removed and replaced with a gold plated (3 mils thick gold plate) stainless gasket. Then after cleaning and lubricating the bolts with a copper base high temperature lubricant and applying a torque of 300 ft-lb to the bolts, there was no further evidence of leakage between the pump casing and stator assembly.

After operating the P-1 loop for 43 hr using H₂O and O₂ overpressure at approximate conditions of 200°C and 950 psi, it was necessary to disassemble the loop pump and replace the upper and lower Graphitar bearings and corresponding Stellite journals. The inner surface of the Graphitar radial bearings was deeply scored, and the Stellite journals were discolored and contained several axial stress cracks ranging from 1/2 to 3/4 in. long (see Fig. 2 and 3). During operation there was no evidence of any damage to the pump bearings except for the presence of finely divided graphite particles in the solution bled from the back of the pump.

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FIG. 2. UPPER GRAPHITAR RADIAL BEARING AND PLUG ASSEMBLY FROM THE A-150-D WESTINGHOUSE PUMP. Cumulative pump operating time before bearing failure was 43 hr. Conditions: 220°C, 950 psi, H₂O and O₂ overpressure. Scale: inside diameter of bearing before use was 1.5025 in. Note the wear on the bearing surface of the graphitar.

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FIG. 3. UPPER STELLITE JOURNAL (ON ROTOR SHAFT) FROM THE A-150-D WESTINGHOUSE PUMP. Cumulative pump operating time before failure was 43 hr. Conditions: 220°C, 950 psi, H₂O and O₂ overpressure. Scale: outside diameter of journal before use was 1.5000 in. Note the discoloration and stress cracks on the surface of the stellite.

The reason for the early bearing failure in the pump was attributed to the loop operating conditions of temperature, pressure, gas concentration, and to the steady continual exchange of the solutions being circulated in the loop and pump housing. Using a partial gas pressurization system, oxygen gas was believed to have been released in the pump bearing region because of the difference in the equilibrium solubility of O_2 at the loop temperature and an estimated pump temperature of 50 to 100°C (see calculations in Appendix).

After replacing the Graphitar bearings and Stellite journals, the Westinghouse pump was assembled with a titanium impeller and thermal barrier and operated successfully for approximately 1500 hr. A view of the scroll case at the end of operation is shown in Fig. 4. Note the pitting which occurred around the periphery, at the end of the loop pipe and at the wear ring screws.

Recommendations for successful operation of the A-150-D Westinghouse pump are as follows:

1. A careful procedure should be followed for starting the pump in order to remove all of the gas initially present in the pump casing and stator assembly. The pump should be turned on for 3 or 4 seconds, shut off, and then bled slowly through the back of the pump casing until approximately 10 ml of liquid are removed. This starting, stopping, and venting procedure should be repeated until there is no indication of gas being removed from the back of the pump.
2. After following the above starting procedure, it was found that any succeeding bleeding of the pump was unnecessary for successful operation. If a sample is desired of the solution in the pump casing, the pump should first be stopped, and then the sample bled from the back of the pump as slowly as possible. Bleeding the pump during operation creates a pressure unbalance and thus abnormal thrust bearing loadings.
3. In order to have sufficient net pump suction head to permit normal operation of the secondary impeller, the loop should be under a pressure of at least 50 psi before starting the pump.
4. In order to keep gas from forming in the pump housing during operation, the equilibrium solubility of noncondensable gas in the pump liquid

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FIG. 4. P-1 LOOP SCROLL CASE AT TERMINATION OF OPERATION OF LOOP

must be greater than the equilibrium solubility in the loop. This condition may be stated by the following equation (see Appendix):

$$[P_L(t)] [S(t_p)] > [P_g(t)] [S(t)]$$

where:

P_L = total static loop pressure, psia

P_g = partial pressure of noncondensable gases, psia

S = solubility of gas in liquid, ml/ml · 100 psia

(t) = loop temperature

t_p = pump temperature

4.2 Flow Restrictors

Two flow restrictors (ORNL Dwg. No. D-25096) were used in the loop to provide the pressure drops necessary in the short length of loop body. Each restrictor operated with 100 feet of pressure drop across it when the loop was at 250-260°C. The titanium lining and the tapered contraction were expected to reduce corrosion due to turbulence and to reduce trapping of solids.

The flow restrictors (Fig. 5,6) were made with a 40° reduction section, a 1 3/4" straight section and a rapid enlargement section. The metal used was titanium-55.

A stainless steel test restrictor was fabricated with dimensions calculated to give 100 ft of head drop for 110 gpm flow and was tested with water at room temperature (1). For 100 ft of head, the observed flows were almost exactly the calculated values of 106 gpm for the design direction of flow and 87 gpm for reverse flow. The enlargement coefficient is essentially constant while the contraction coefficient increases due to the sharp edge contraction for reverse flow.

4.3 Hydroclone

The hydroclone was added to the loop system to collect corrosion products and plutonium. The hydroclone appeared to be quite effective in removing particles which reached it. However, only a small fraction of the corrosion products was circulated through the hydroclone circuit.

A 0.4 in. diameter hydroclone with a 25 ml underflow pot was used (ORNL Dwg. D-25078). The body insert and vortex finder (Fig. 6) were made of Ti-55 and the remaining parts of 347 stainless steel.

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FIG.5 LOOP P-1 FLOW RESTRICTOR ASSEMBLY

FIG. 6. LOOP FLOW RESTRICTORS AND HYDROCLONE COMPONENTS

The hydroclone operated for approximately 450 hr at normal conditions. During run 33 a plug formed somewhere in the hydroclone circuit while PuO_2 was being injected in 1 gram quantities every eight hours. The plugging occurred between the first and second PuO_2 spike. All efforts to free the plug failed, and due to health physics hazards, the hydroclone was not cut open for observation. The plug was probably comparable to a partial flow restriction since the temperature of the hydroclone overflow only decreased a few degrees from its normal operating temperature.

4.4 Pressurizer

A special pressurizer was designed with two basic safety features to minimize the dangers of $\text{O}_2 + \text{H}_2$ reactions causing the release of plutonium into the operating area. First, there were no unmixed volumes in the pressurizer vapor space in which hydrogen and oxygen could segregate from water vapor. Solution passing through the pressurizer to be saturated with hydrogen and oxygen entered through a spray nozzle placed to thoroughly swirl and stir material in the vapor space (Fig. 7). The space below the normal liquid level (at 250°C) was packed with 1/4-in.-dia 3/8-in.-long rings to reduce the detonation concentration and break up detonation waves. These features, plus control of H_2 and O_2 concentrations, were intended to prevent concentrations above detonation limits.

The pressurizer and loop designs provided two containments for severe overpressures such as would occur from a $\text{O}_2 - \text{H}_2$ detonation. The 6-in. IPS schedule 160 pipe and caps used for the pressurizer body would be a safe working design for 4,000 psi and would probably contain 20,000 psi. The entire loop, including the pump and hydroclone, was canned in a pressure vessel designed to contain the entire loop contents discharged at 250°C . A blast shield was installed around the pressurizer to contain shrapnel that might be produced by a pressurizer rupture and to thus prevent puncturing of the container can. This blast shield was prestressed for resistance to shock and was vented through numerous holes to release the vapor into the loop can.

Results from test of a prototype pressurizer (2) indicated that spontaneous $\text{H}_2 - \text{O}_2$ explosions would not occur frequently to seriously hamper experimental operations and that they would not result in hazardous equipment failures. Subsequent laboratory results in stainless steel systems indicated that the presence

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FIG. 7. LOCP P-1 PRESSURIZER COMPONENTS BEFORE ASSEMBLY

of stainless steel corrosion products controlled the behavior of plutonium and the addition of hydrogen to stainless steel development loops was not effective for reducing Pu(VI) to Pu(IV). Therefore the Raschig rings were removed to reduce the surface area exposed to loop solution and the blast shield was not re-installed. The Raschig ring free pressurizer without blast shield and thermal level probe was used during the remaining 700 hr of operation. For details of these components see ORNL Dwg. No. E-25077, D-25082, Q-1771-9D, and Q-1771-8D.

Only two difficulties were encountered with the pressurizer. First, the octagonal ring gasket was impossible to seat until a 3 mil gold plating was applied. Second, the pressurizer bottom accumulated a thick layer of refractory type corrosion product scale which plugged both gas inlet lines to the Raschig ring filled pressurizer. Another gas inlet line became plugged after approximately 600 hr of operation with the Raschig ring free pressurizer. To avoid this plugging, the H_2 and O_2 should be injected into the gas phase due to the corrosion product build up in the liquid phase. A vortex mixer could be used to mix the H_2 and O_2 to prevent a detonation. If no H_2 is to be used in the pressurizer, then the problem of gas mixing is eliminated. The use of Raschig rings is not required for O_2 overpressure only, thereby, reducing the loop area considerably.

4.5 Bomb Sampling and Solids Addition System

The solids addition bomb was used for injecting fluids into the loop and for getting representative samples of the loop circulating medium. The procedure used was to fill the bomb with either spike or feed solution. After installing the filled bomb on the bomb train the valves were fully opened for five minutes. The flow rate was approximately 700 bomb volumes per minute or a total of 3500 per sample insuring that any spike was washed out of the bomb very thoroughly or that any loop sample was representative of the circulating medium.

The bomb was a conical frustum shaped chamber with a dip leg extending to within $3/32$ " of the bottom. The flow was in at the top and out through the dip leg; for details see ORNL Dwg. D-26400 and D-26402. The chamber held approximately 120 ml.

The bomb was cooled by circulating H_2O through cooling coils which were welded to the circumference of the bomb. By cooling in this manner it was possible to take bomb samples as frequently as every 15 min. The bomb valve train consisted of six Autoclave Engineering Inc. valves in series designed to

give minimum hold-up between valves. The bomb was fabricated of type 347 stainless steel, and the cooling coils were of type 304 stainless steel. The valves were made of 304 type stainless steel, and one set of titanium valves was used while waiting for replacements. All lines were of type 347 stainless steel.

One difficulty encountered with the bombs was lack of clearance between the inlet and outlet ports. This clearance should be great enough to allow the jaw of an open end wrench to be inserted between the two lines and rotated without binding against the opposite leg gland while tightening the bomb. The controlling clearance is the distance between the glands.

It would be preferable to use only two valves on each side with a looped pipe section connecting them. The short lengths of tubing used were too rigid for proper valve utilization. This rigidity resulted in thermal stresses caused by quick heating and cooling and in mechanical stress transmission. The valve-holding assembly should be designed to withstand at least 150 ft lb torque and to withstand approximately 100 lb of shear force applied from any horizontal direction.

4.6 Coupon Holder

The coupon holder was designed to hold 12 metal coupons each 0.5 in. x 0.562 in. x 0.03 in. in either a vertical or horizontal position. The coupons were used for obtaining corrosion and adsorption data on stainless steel, zircaloy-2, and titanium metal coupons. The coupons proved to be useless for corrosion data, but adsorption data was obtained. The liquid velocity over the coupons varied from 1 to 3 feet/second.

The coupon holder assembly consisted of a head, cap assembly, and thrust ring as manufactured by Superpressure Division, American Instrument Company, for vessel catalog No. 406-3504. The body, retainer plug, head gasket, and the coupon holder were fabricated in ORNL shops. The details of these assemblies are given by ORNL Dwg. No. D-26390.

The retainer plug was made of 17-4 PH stainless steel and designed for 5 mil clearance between the plug and body assembly. An O-ring type seal was provided on the retainer plug to hold the pressure and relieve the head assembly of having to be pressure tight. The head gasket was made of 304 type stainless steel with a thickness of 0.065 in. $\pm$ 0.015 in. This later proved to be inadequate tolerance

for withstanding the loop operating pressure.

The coupon holder was a split chamber with a rectangular inside cross section having three different tapered sections. The two holder halves originally were to be held together with clamp rings made from 20 gauge sheet stainless steel type 304. The resilience of the clamps was not sufficient, and the clamps remained in a somewhat open position after placing them over the holder halves. The coupon holder halves were held together for insertion and removal by means of a wire spiral instead of the clamp ring. The spiral was made of 20 mil dia stainless steel type 304 wire wound around a 3/8 in. bolt thread and rewound over the coupon holder halves.

The clearance of 5 mils between the retainer plug and the body assembly was not sufficient. After several runs with 10 gm U/kg H₂O the cylindrical shoulder of the retainer plug galled with the coupon holder body. The plug had 5 mils machined from it after removal; however, this later proved to be insufficient as a second galling occurred after the first Pu run. The plug had 15 mils machined from it after this second galling for a total clearance of 25 mils. The galling was enhanced by the large amount of corrosion products formed in the now oxygen-depleted retainer plug area. The 25 mil clearance appears to be sufficient to allow enough liquid exchange between the loop medium and this area to prevent an oxygen deficient solution from being formed.

The plug was designed with an O-ring to contain the loop pressure and prevent the UO₂SO₄ solution from contacting the cylindrical retainer plug surface. Silicone rubber and teflon O-rings were tried but both proved to be inadequate. The silicone rubber dissolved in the 1.4 M UO₂SO₄, and the teflon failed to seal due to its extrusion at loop temperature.

The O-ring failure caused the loop pressure to be transmitted to the head gasket. The tolerance of +15 mil for the gasket was not sufficient for a seal. A 20 mil gold disk was inserted between the coupon body head and head gasket; no further leakage occurred.

4.7 Instrumentation

The instrumentation for the loop consisted mainly of standard, commercially available instruments, however, a new type of 0 to 100 psig differential pressure cell was used. This stainless bellows sensing element, inductance type cell

uses a 1,000 cycle excitation supplied from a Foxboro Dynalog instrument. It is rated for a process pressure of 2,500 psi and has an electrical output of 0 to 1 millivolt per volt input. Initial difficulties were encountered in properly sealing the cell chambers with the bolt construction supplied by the manufacturer (the Foxboro Company). However, by using a higher cross section Teflon sealing O-Ring, these difficulties were eliminated. The body of the cell is so made as to permit seal welding if required. Service difficulties were not encountered after the installation. The cell appears to work quite satisfactorily over protracted periods of time and under repeated let down and repressurizing conditions. An earlier version of this type of cell, with a 0 to 55 psi differential range was found applicable on the loop. This type of cell had previously been installed in the HRT Chemical Plant.

A thermal level probe of the design used in the HRT Chemical Plant was inserted in the loop pressurizer. The thermal level probe was modified so that only three temperature points were recorded or indicated over its length. No difficulties arose in the operation of the probe.

A novel continuous H_2 analyzer and alarm system installed on the loop can was not used because H_2 pressurization was not used.

4.8 Valves

The valves used for P-1 Loop were Autoclave Engineers, Incorporated, 15,000 and 30,000 series. The most frequently used valves were 304 type stainless steel with 17-4PH valve stems and teflon packing. In one instance a set of six titanium valves were used in the bomb valve train.

Valve failures were of three types. The failure of either the valve stem tips or the valve stem seats were major causes of valve failure. The valve seats and tips would become scored due to corrosion product particles being left between these two surfaces whenever the valve was closed. After several deep scores the surfaces would fail to seat properly with a resulting leaky valve. In two cases three valves in series failed due to scored seats and stem tips.

Another valve stem tip failure was due to the valve tip shearing off. This was probably due to normal shearing forces associated with opening and closing. A second and very important cause was that if a valve was closed while hot, then an unusual amount of torque was required to reopen the valve after cooling.

This excess torque was applied to the valve stem tip in the form of a shearing force.

About one-half of the valve failures was due to faulty packing. Valve stem packings of teflon would extrude while hot and result in a leaky valve. The valves were initially assembled with a packing made of copper foil and graphite. It was found upon inspection that the valve stems had galled to the valve body with graphite particles between the two. The packings were removed and replaced with Teflon packings prior to any runs. Not a single case of galling occurred with the Teflon packings.

The valve to tubing connections held very well. Only one case of a bad fitting was encountered. This resulted from using a titanium valve with stainless steel tubing which was badly scored. This particular connection leaked only while the connection was heating or cooling during bomb sampling. A strip heater was applied to the valve and tubing to keep them both at 205°C , and at this temperature the valve sustained 338 hr of satisfactory operation. The ultimate failure of this valve was the shearing off of the stem tip.

The water cooled sample valve (ORNL Dwg. E-26375) sustained approximately 1000 hr of operation without replacement of the packing. The valve seat became scored and would not reseal after approximately 400 hr of operation, representing some 100 samples. Another more accessible valve was placed in series with the water cooled valve and used as a safety valve. The packing in the water cooled sample valve held for the remainder of the runs. This was about three times the life of valve packings in similarly located positions with no cooling.

The only recommendation to be made for future design concerning valves is that all tubing connections should be accessible to a coning tool. At high temperature and pressure using UO_2SO_4 solutions it will be necessary from time to time to replace valves. When replacing valves the coned surface of the tubing will inevitably become scored and must be reconed. If it is necessary to locate a valve connection in a location where the standard coning tool will not operate, then a special coning tool should be designed for such places.

5.0 LOOP SAMPLING

Four methods of getting samples were incorporated into the loop design.

They were hydroclone underflow pot samples, solids addition bomb samples, metal coupon samples, and the high pressure generator feed system.

The underflow pot sample volume was 25 ml, usually taken every 4 or 8 hours. These samples consisted of the solids removed by the hydroclone plus accompanying loop solution. From the amount of corrosion products collected in the hydroclone underflow pot one would surmise that the bulk of the corrosion products never entered the hydroclone circuit. The same holds true for the plutonium collected in the underflow pot.

The solids addition bombs were used to inject spikes into the loop and also to get representative samples of the loop circulating medium. The bomb was opened for five minutes allowing approximately 3500 bomb volumes to be rinsed through. This insured that the bomb solution was representative of the circulating loop medium. Bomb samples were taken at every 50°C rise in temperature while heating up and every 4 or 8 hours while at loop temperature. It was possible to obtain a sample every 15 minutes at a loop temperature of 250°C with the H₂O cooling coils on the bomb.

Metal coupon samples were used in the coupon holder for corrosion and adsorption data. Twelve metal coupons could be suspended in the loop medium at varying velocities from 1 to 3 ft/sec. The coupons were only removed at the end of a run.

The bomb sampling was the most accurate, dependable, and consistent indication of conditions in the loop. The coupons and the hydroclone samples were intended for specific conditions and were adequate for these purposes.

5.1 High Pressure Generator

The high pressure generator modified for cooling coils was never used due to repeated failure of the "O" ring seals. This equipment (See Drawing E-26378) was designed for the "O" rings to seal while sliding during operation of a threaded plunger.

The high pressure generator (See Boder Scientific Co. drawing no. 6-107-1) was originally designed to withdraw a 30 ml sample from the circulating liquid in the P-1 loop and to replace the sample volume against the loop pressure. The high pressure generator was water cooled and isolated from the main loop

flow by two autoclave valves connected in series on both the inlet and the outlet generator lines. A sample was taken by withdrawing the generator piston through 6 in. of travel. The piston and generator housing had been machined with 14 threads per in., so that it required 84 revolutions to move the piston from a closed to an open position. The sample volume was replaced by filling the generator with the piston in the withdrawn position and then closing the piston forcing the liquid into the loop.

The high pressure generator operated successfully until leakage occurred between the piston and generator housing. Upon disassembly of the sampling equipment, the rubber O-rings were found to be soft and apparently worn smaller than the original size. The rubber O-rings were replaced with Teflon, but the same leak recurred after removing 8 samples. Since the solids addition bomb was working properly, the high pressure generator was isolated from the rest of the loop system and was not used after the first ZrO_2 run (P-17).

In order to successfully operate the high pressure generator under conditions as encountered in the P-1 loop operations, it would be necessary to install a more resistant and effective type seal between the piston and housing assembly. The high pressure generator could possibly be used as it was originally designed, if a more efficient cooling system was installed in the vicinity of the rubber O-rings.

6.0 PLUTONIUM HANDLING

Extreme care is required for handling plutonium. The loop design features made possible numerous transfers of plutonium to and from the loop without any detectable amount of plutonium being discovered within the loop operating area. A lifetime dosage of Pu^{239} is 0.04μ curie (6.5×10^{-7} grams) with a biological half-life of 120 years.

Several safety features were incorporated into the loop design: glove boxes with double layer glass fronts, plastic bags for removing items from the glove boxes, a decontamination system for both the glove boxes and the loop container, an underground slurry tank for rapid let down, and a negative pressure within the loop container to confine the plutonium in the event of a line rupture.

6.1 Glove Boxes

The glove boxes were designed to confine any Pu activity and to permit entrance and removal of items to the boxes by means of 12 ft long polyethylene bags (Fig. 8) without any contact between the room atmosphere and the inside of the box. A sealing machine (built by Plastic Welding Corporation) was used to seal the bags. The sealing unit makes three seals 1/8" apart. The center seal was melted to the point that it could be torn with the fingers, leaving both ends sealed. There was no detectable amount of Pu lost from the bags during any of the runs.

The boxes were fabricated from 347 stainless steel. One box (Glove Box A) contained the hydroclone with underflow pot, soup addition tank, high pressure generator, gas addition bottles, pump bleed sight glass, and differential pressure cells. One (Glove Box B) contained the sample coupon assembly, gas analysis port, and the solids addition bomb. A third was added later for the purpose of charging the loop and for filtering the loop drain; however, this box was not used except during the first Pu run.

Each glove box was equipped with a sink that emptied into an underground tank located beneath the floor, and a decontamination system. Two external tanks of 12.5 liters capacity were connected to a decontamination hose located in each dry box. Various decontamination solutions could be sprayed into each dry box under N₂ pressure.

6.2 Glove Box Procedures

Each glove box had a 10 mil polyethylene bag, (supplied by Crystal-X Corporation) to transport items to and from the glove boxes. The bags were 12 ft long with a Buna-N O-ring for securing the bags to the glove box ports (ORNL Dwg. E-25118).

The plastic bags were sealed with a plastic welder (Weldotron Model 5-2B-R5, manufactured by Plastic Welding Corporation) which made three simultaneous seals. This permitted the bag to be torn leaving both ends sealed without exposing any of the inner surface to the room atmosphere..

The only difficulty experienced with the glove boxes was with holes in the gloves by puncturing during use. Punctures were discovered before any contamination was released so no serious contamination problem ever existed.

FIG. 8. P-1 LOOP SHOWING GLOVE BOXES

In almost all cases the operator realized when the puncture was made, and a new glove was installed immediately.

The plastic bag method of transporting material to and from dry boxes containing α material is much superior to the air lock type of transport. The one chief advantage with the bag lies in the fact that there is never any contact between the inside of the bag and the operating area atmosphere.

6.3 Plutonium Transfers

All underflow pot samples were transported in polyethylene sample bottles which were sealed in a plastic bag. The plastic bag and its contents were transported in a stainless steel carrier which was checked for contamination at the end of each transport. The solids addition bomb, feed, and coupon holder were transported in a similar manner.

7.0 CORROSION

Corrosion rates for the P-1 loop were calculated from soluble nickel analyses and checked by O_2 consumption calculations. The calculated rates varied from less than 2 mpy during preliminary operation with $1.4 \text{ M } UO_2SO_4$ up to 23 mpy during studies with soluble plutonium. The corrosion rates indicated by O_2 consumption were within $\pm 25\%$ of those from nickel analyses.

Corrosion rates from nickel analyses assume that all the stainless steel corroded releases its nickel into the solution as soluble ions. The iron and chromium corrosion products are not soluble. Corrosion rates calculated from oxygen consumptions are less consistent than nickel analyses. The O_2 is only a small fraction of the total pressure; the gas volume varies from run to run, and O_2 is lost by sampling and leakage. The $\pm 25\%$ check between nickel analyses and oxygen consumption as measures of corrosion is as good as could be expected. The area/volume ratio was $4 \text{ ft}^2/\text{liter}$ for the first 800 hr of operation and $1 \text{ ft}^2/\text{liter}$ for the final 700 hr of operation. This compares with $1.2 \text{ ft}^2/\text{liter}$ for HRE-2.

As shown in Table 1 the corrosion rate is unaffected by zirconium additions. It remained less than 2 mpy with or without the zirconium in $1.4 \text{ M } UO_2SO_4$.

The corrosion rate increased to 18-23 mpy when PuO_2SO_4 was added to the blanket solution. The upper limit occurred when the PuO_2SO_4 began to precipitate and remained at that rate through the remainder of the run. The corrosion

Table 1. Loop P-1 Corrosion Rates

Conditions: 1.4 M UO_2SO_4 at 250°C

Material Added to Loop	Pu Concentration (mg/liter)	Generalized Corrosion Rate ^a (mpy)
None		1.5
$\text{Zr}(\text{SO}_4)$ or ZrO_2		1.4
$\text{Pu}(\text{SO}_4)_2$	1.7	18.1
PuO_2SO_4	13-70	23.2
PuO_2SO_4	70 to ppt ^b	22.8 ^c
PuO_2	74 ^d	11.3
PuO_2SO_4	10 to ppt ^e	9.9
CrSO_4		25.0 ^f

^a Values listed are from nickel concentrations; oxygen consumptions gave checks within $\pm 25\%$.

^b Sufficient plutonyl sulfate was added to increase the plutonium concentration to 342 mg/liter. Plutonium precipitated at concentrations > 100 mg/liter.

^c Based on oxygen consumptions; nickel analyses were erratic.

^d 65 mg of plutonium per liter as PuO_2 plus 9 mg of plutonium per liter in solution.

^e Sufficient plutonyl sulfate was added to increase the plutonium concentration to 580 mg/liter. Plutonium precipitated at concentrations > 100 mg/liter.

^f 0.36 N chromous and 0.77 N acid as sulfuric; temperature cycled between 75 and 90°C.

rate decreased to approximately 10 mpy during subsequent operation with PuO_2 . The rate remained at this value when PuO_2SO_4 was added to the blanket solution containing PuO_2 .

A descaling solution of chromous sulfate was used in an attempt to remove the adsorbed plutonium. The chromous sulfate solution was 0.36 N chromous and 0.77 N acid as sulfuric acid. The corrosion rate during the three descaling runs based on nickel analysis of the drain solution was about 25 mpy.

Visual inspection indicated that only a small amount of pitting type corrosion occurred during P-1 operation. Pitting was observed around the scroll case periphery and at the end of the loop pipe (Figure 4). The titanium appeared to have undergone a negligible amount of corrosion. The impeller and all other Ti parts appeared to be unaffected by the blanket solution.

8.0 DESCALING OPERATIONS

Chromous sulfate solution (0.36 N chromous and 0.77 N sulfuric acid) was used in an attempt to remove the adsorbed plutonium from the loop. A series of these runs were made using the chromous sulfate solution. The amount of plutonium removed was 8.62 grams whereas only 8.0 grams were present at the start of the descaling operation based on an overall material balance.

The loop preparation for the CrSO_4 descaling was a thorough rinse to remove uranium followed by two nitrogen pressurization-evacuation cycles. The CrSO_4 was drawn into the loop by means of a vacuum with a mineral oil layer on the feed bottle to keep the CrSO_4 from being oxidized by the air during transportation.

Three chromous sulfate runs were made. The first, 76 hr in duration, consisted of three thermal cycles between room temperature and 90°C . The amount of Pu recovered during this run was 4.06 grams. The second, 44 hr in duration, was thermal cycled between 75 and 95°C for 2.5 hr. The amount of Pu recovered was 3.68 grams. The third, 24 hr in duration, was made with the loop at 90°C for 4 hr. The amount of Pu removed during this run was 0.88 grams. A total of 8.62 grams was removed during the three runs from a precalculated amount of 8.0 grams charged to the loop. The chromous sulfate was very effective in removing adsorbed plutonium from the P-1 loop.

9.0 LOOP HEAT BALANCE

A typical heat balance at steady state conditions accounts for 98% of the heat with 2% heat loss (Fig. 9). The major portion of the heat, 61.5%, was removed by the air through the cooling coils (Fig. 10) welded to the loop periphery. The remaining 36.5% was removed by cooling water flowing through the double pipe heat exchanger of the Westinghouse pump.

The cooling coils shown in Fig. 10 are 1/2" OD x 16 ga stainless tubing as shown by ORNL Dwg. D-25103. The amount of heat removed by the cooling coils as a function of air flow rate is shown in Fig. 11. Heat removal by this welded on coil design was demonstrated by a prototype heat exchanger (3).

The loop temperature was easily controlled. The time to bring the loop up to temperature was approximately 2 1/2 hr. The heating up time was independent of room temperature due to the insulation offered by the vacuum surrounding the loop (0.5 psia).

Table 2. P-1 Heat Balance Data

Pump Electrical Input	=	48,800 BTU/hr*
Air Flow Rate	=	36 SCFM
Heat Removed by Air	=	30,000 BTU/hr**
H ₂ O Flow Rate	=	1,780 lb/hr
H ₂ O ΔT	=	10°F
Heat Removed by H ₂ O	=	17,800 BTU/hr
Heat Loss	=	1,000 BTU/hr

* Loop pump and H₂O circulating pump heat input.

**Taken from Fig. 11.

FIG. 9 -- LOOP P-1 HEAT BALANCE

FIG. 10. P-1 LOOP AIR COOLING COILS

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FIG.11 P-1 LOOP HEAT REMOVED BY AIR FLOW

10.0 REFERENCES

- (1) CF-56-2-154 "Chemical Technology Division - Unit Operations Section Monthly Progress Report - February, 1956", p. 70-76.
- (2) P. A. Haas, "HRP Blanket Processing Studies of O_2H_2 Hazards in a Loop Pressurizer", ORNL-2568, Sept. 25, 1958.
- (3) CF-56-5-197, "Chemical Technology Division - Unit Operations Section Monthly Progress Report - May, 1956", p. 51-62.

11.0 APPENDIX

11.1 O_2 Overpressure Calculations

A failure of the pump bearing (Sec. 4.11) necessitated solubility calculations of O_2 in UO_2SO_4 to determine proper operating conditions of gas overpressure. A simple material and volume balance for the P-1 loop system yields the following equations which relate loop pressure, volume and temperature. These calculations are similar to those made for studies of O_2 - H_2 hazards in the loop P-1 pressurizer (2).

$$V_1(T) = \frac{\rho_1^0 V_1^0 - \rho_s(T) [V_t - V_p^0] - V_p^0 [\rho_1(100^\circ C) - \rho_1^0]}{\rho_1(T) - \rho_s(T)}$$

$$P_g(T) = \frac{\frac{V_g^0 P_g^0(273)}{14.7(273 + T)} + (V_1^0 + V_p^0) P_g^0 [S(21^\circ C)] - P_s V_p^0 [S(100^\circ C)]}{\frac{V_g(T) 273}{14.7(273 + T)} + V_1(T) S(T) + V_p^0 S(100^\circ C)}$$

where

- V_1 = volume of liquid phase, ml
- V_t = total volume of loop ($V_p^0 + V_1 + V_g = 16,000$ ml)
- V_p = volume of pump housing = 3,000 ml
- V_g = volume of gas phase, ml
- V_1^0 = volume of liquid phase at room temperature, 10 liters
- V_g^0 = volume of gas phase at room temperature, 3 liters
- P_g = partial pressure of noncondensable gases, psia
- P_g^0 = P_g at room temperature, 65 psia
- P_s = steam pressure obtained from steam tables, 680 psia
- T = temperature of loop contents, $260^\circ C$
- ρ_1 = density of liquid from Fig. 12, g/ml
- ρ_s = density of steam from steam table, g/ml
- S = solubility of gas in liquid from Fig. 13, ml/ml, 100 psia
- $P_t = P_s + P_g$

In order to keep gas from forming in the pump housing during operation, the equilibrium solubility of noncondensable gas in the pump liquid must be

greater than that in the loop. Assuming the liquid in the pump to be at 100°C, this condition may be stated

$$P_{(T)} S_{(pump)} > P_{O_2} S_{(T)}$$

$$\frac{P_{(T)}}{P_{O_2}} > \frac{S_{(T)}}{S_{(pump)}}$$

$$\frac{P_{(S)}}{P_{O_2}} + 1 > \frac{S_{(T)}}{S_{(pump)}}$$

Using the above conditions and equations and data from Fig. 12 and 13, the curve of Fig. 14 is obtained. The region above the curve is the safe operating region and that below is the danger area.

The operating procedure as outlined in Sec. 4.1 for proper loop overpressure was determined from Fig. 14.

11.2 P-1 Loop Drawing

<u>Drawing No.</u>	<u>Title of Drawing</u>	<u>Rev. No.</u>
E-25077	Pressure and Shield Assembly	2
D-25078	Hydroclone	3
D-25082	Pressure Assembly	-
E-25087	Pump and Container Supports	2
E-25088	Schematic Flow Diagram	8
D-25089	Hydroclone Chamber	3
D-25096	Flow Restrictors	3
E-25102	Container Details	3
D-25103	Piping Details-Pump Loop	3
C-25104	Service Supply Vessels	2
C-25109	Heat Exchanger Test	-
D-25114	Soup Addition Tanks	2
E-25118	Dry Box Details	3
E-25119	Dry Box Details	-
D-25123	Gas Supply Equipment	1
C-25124	Gas Supply Support Equipment	1

FIG.12 DENSITY OF 1.4 M UO_2SO_4 SOLUTION VERSUS TEMPERATURE

- 37 -

FIG.13 OXYGEN SOLUBILITY IN 1.4 M UO_2SO_4 VERSUS TEMPERATURE

FIG.14 DANGER PLOT FOR O₂ OVERPRESSURE IN P-1 LOOP

864 038

<u>Drawing No.</u>	<u>Title of Drawing</u>	<u>Rev. No.</u>
C-26375	H ₂ O Cooling Valve	-
E-26376	Test Pressure and Shield Assembly	-
E-26378	H.P. Gen. Control Arrangement	-
E-26381	Container Spray System	-
E-26389	General Layout	2
D-26390	Coupon Holder Assembly and Details	2
D-26391	Coupon Holder Installation and Mis.	1
E-26392	Elevation of Operating Equipment	4
C-26394	Pump Vent and Flame Arrestor	-
D-26395	Off-Gas Entrainment Sep. and Filter	-
E-26396	General Piping Arrangement	4
D-26397	Monorail for P-1 Loop	-
D-26399	Miscellaneous Installation Details	2
D-26400	Sample Bomb Assembly and Details	1
D-26402	Sample Bomb-Parts Details	-
D-26407	Sample Bomb Service Piping	1
D-26408	Sample Bomb Dry Box	-

11.3 Tabulated Run Conditions

<u>Run No.</u>	<u>Operating Time</u>		<u>Type Run</u>	<u>Remarks</u>
	<u>hr</u>	<u>min</u>		
P-1	6	50	H ₂ O	
P-2	5	15	3% Na ₃ PO ₄	
P-3	31	35	H ₂ O	
----- -Bearing Failure-----				
P-4	46	10	5% HNO ₃	
P-5	60	35	H ₂ O	
P-6	13	20	10 g U/liter	
P-7		25	H ₂ O	
P-8	52	02	10 g U/liter	
P-9	14	07	10 g U/liter	
P-10	13	40	45 g U/liter	
P-11	13	00	45 g U/liter	
P-12	10	15	45 g U/liter	
P-13	24	00	45 g U/liter	

Run No.	Operating Time		Type Run	Remarks
	hr	min		
P-14	15	38	295 g U/liter	
P-15	97	15	310 g U/liter	
P-16	60	10	300 g U/liter	
P-17	96	02	310 g U/liter	1 g ZrO ₂
P-18	77	41	312 g U/liter	2 g ZrO ₂
P-19	20	00	312 g U/liter	
P-20	39	05	312 g U/liter	
P-21	35	45	312 g U/liter	1 g ZrO ₂
P-22	102	45	312 g U/liter	10 g ZrO ₂
----- Pressurizer Change -----				
P-23	28	15	312 g U/liter	
P-24	31	00	312 g U/liter	
P-25	83	25	312 g U/liter	
P-26	13	45	312 g U/liter	
P-27	101	45	312 g U/liter	Pu(SO ₄) ₂
P-28A	8	30	312 g U/liter	P-27 Drain
P-28B	12	24	H ₂ O	
P-29	182	00	312 g U/liter	PuO ₂ SO ₄
P-30	76	10	312 g U/liter	PuO ₂ SO ₄
P-33	82	15	312 g U/liter	PuO ₂ (hydroclone plugged)
P-34	1	36	H ₂ O	
P-35	4	15	H ₂ O	
P-36	3	15	H ₂ O	
P-37	1	30	CrSO ₄	
P-38		51	H ₂ O	
P-39		27	CrSO ₄	
P-40	2	00	H ₂ O	
P-41	3	50	CrSO ₄	
P-42	2	30	H ₂ O	
TOTAL	1542 hr	48 min		

864 040

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