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DATE: July 21, 1958
 SUBJECT: Core Solution System, Run 22

TO: Distribution
 FROM: P. H. Harley 7770

Summary

The system was operated for 2623 hours at 1400 psi and 280°C. The fuel was 0.042 m WO_2SO_4 solution containing 0.025 m H_2SO_4 and 0.025 m CuSO_4 .

The generalized corrosion rate with a titanium letdown heat exchanger in the system was 0.80 mpy as compared to runs with a 347 SS heat exchanger in which the corrosion rate was 1.5 mpy. This corresponds to a 2.9 mpy generalized corrosion rate for the stainless steel heat exchanger.

A preliminary solution inventory study was made which showed that the variance in the dump tank inventory was 180 times as large as in any other part of the system. A more accurate method to measure the contents of the dump tank will be required for a good system inventory.

Precipitation of uranium between the pressurizer and the main loop was shown to depend upon the pressurizer purge rate and excess acid concentration. At 60% excess acid uranium would precipitate with the purge flow above 3 gph and at 55% excess acid uranium would precipitate above 2 gph purge flow.

The fuel mixing rate into the motor end of the circulating pump was 2.7 to 3 liters per hour for 2200 hours of the run and then dropped steadily to 0.04 liters per hour during the last 400 hours. No explanation has been found for this change.

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To: Distribution
From: P. H. Harley
Date: July 21, 1958
Subject: Core Solution System, Run 22

Introduction

Prior to starting Run 22, the 347 SS letdown heat exchanger was replaced by one constructed of A-55 titanium. The main objective of this run was to compare the loop generalized corrosion rate with the corrosion in similar runs in which stainless steel letdown heat exchangers were used.

Several other tests were made during this run. A preliminary continuous inventory study was made to locate any equipment design or operating and sampling procedures which needed to be improved to obtain an accurate system inventory.

Previous runs had indicated that uranium was being precipitated between the pressurizer and the main loop. The reason for this precipitation and methods of preventing it were investigated.

During the end of the run, the uranium content of the liquid and the motor end of the circulating pump decreased steadily but the reason for the change has not been identified.

General Operation

The operating conditions for this run were 1400 psi and 280°C. The fuel composition was 0.042 m UO_2SO_4 , 0.021 m H_2SO_4 and 0.005 m CuSO_4 for the first 1300 hours. For the rest of the run 0.023 m H_2SO_4 and 0.025 m CuSO_4 were used. The total operating time for the run was 2,523 hours. The run was terminated 48 hours before the scheduled shutdown date by a leak in a screwed fitting at the dump tank evaporator sample point.

Very few mechanical difficulties were encountered during the run and only one 48 hour shutdown was made. This shutdown came when the condensate return line between the high pressure loop heat exchanger and the 80 kw boiler was thought to be plugged. However, the subsequent investigation showed that the line was open and a shift in the boiler level indicator had caused the boiler to be filled too full. This caused flooding and loss of heat transfer surface in the loop heat exchanger.

Other equipment failures which were repaired without shutting down were: eleven packing gland failures in the O_2 compressor intermediate system, a weld leak in a spool piece to the feed pump suction, and a flange leak at the feed pump pulsator housing in the Scott and Williams oil system.

The total operating time on the major system components are:

1. Feed Pump, North Head	4890 hours
2. Feed Pump, South Head	9378 hours
3. Purge Pump Head	5637 hours
4. Ti Letdown Heat Exchanger	2623 hours
5. Loop Heat Exchanger	2623 hours
6. Circulating Pump	2623 hours
7. Letdown Valve	2700 hours

The letdown valve with 17-4 ph stainless steel trim had a leak rate of 190 cc/min with a 6 psi instrument air setting and 2000 psi water pressure under the seat when tested after the run. This valve was too large and cycled from closed to 15% open several times a minute during the entire run. A smaller valve has been fabricated which should have better throttling characteristics.

Corrosion Rates

The corrosion rate during the run was based on an inventory of the nickel in solution. Since no direct means of determining the exact quantity of solution in the system was available the amount charged minus the quantity removed in sampling was used to determine the volume of solution.

Over the first four days the generalized corrosion rate was 1.0 mpy. Following this startup and film forming period the corrosion rate decreased to 0.80 mpy for the remainder of the run. This value compares with 1.5 mpy in Runs 17 and 18 and 1.3 mpy in Runs 20 and 21. Since the letdown heat exchanger comprises 33.7% of the surface area in the high pressure fuel system, the generalized corrosion rate in the 347 stainless steel letdown heat exchanger (Runs 17 and 18) was calculated to be 2.9 mpy. W. J. Werner and T. M. Kegley, Jr. reported the results of the metallographic examination of the heat exchanger removed at the end of Run 18.* They showed that most of the corrosion was localized in a few areas in the heat exchanger.

Continuous Uranium Inventory

For a breeder type reactor to demonstrate breeding, a method to adequately demonstrate that more fuel has been produced than the amount that has been burned would be required. This could be done from a continuous inventory provided that the inventory was accurate. Also an accurate inventory procedure would help in indicating when precipitation of uranium from solution occurred in the system.

* W. J. Werner, et al., Metallographic Examination of HRT Mock-up Letdown Heat Exchanger, HRP-MET-10 (March 28, 1958).

With these ideas in mind, a preliminary study was made during the first five weeks of Run 22 for the purpose of setting up inventory control procedures, and correcting any variables which introduced excessive errors into the inventory.

During the first part of the study, the variance in the samples was examined. Six 2-Oz specimens were taken from the high pressure system and 6 from the low pressure system in each sample. Three specimens from a stock solution comprised the control sample. The standard deviations for the different samples are plotted in Fig. 1. After calculating the high deviation in the control sample on February 13, the control was increased to 6 specimens for samples taken starting February 24 to get better results.

The average of the points plotted in Fig. 1 represents a deviation of $\pm 5.4\%$ of the sample average in the low pressure system, -2.54% in the high pressure system and -2.05% in the control samples. A better sampling procedure was needed in the low pressure samples and should be developed in the next run.

For calculating the uranium inventory, the fuel system was divided into seven sections according to fuel concentrations and solution temperatures. The seven sections were: (1) the high pressure loop, (2) the dump tank and evaporator, (3) the letdown stream in the letdown heat exchanger, (4) the letdown stream from the heat exchanger to the dump tank (5) the feed stream from the dump tank through the feed cooler, (6) the cool feed from the cooler to the letdown heat exchanger, and (7) the feed stream in the letdown heat exchanger to the high pressure loop. The seven sections are shown in the flow sheet, Fig. 2.

The inventory in each section is a function of the section volume, uranium analysis and fluid density. The liquid volumes of all seven sections were constant except the dump tank and all seven sections were represented by two samples, a low pressure system sample and a high pressure system sample. The total uranium inventory was a summation of the inventory of each section:

$$I = A_1 V_1 D_1 + A_2 V_2 D_2 + \text{-----} \text{ etc.}$$

The inventory variance, σ_I^2 , is the summation of the variances of each section:

$$\sigma_I^2 = (A_1^2 V_1^2) \sigma_{D_1}^2 + (D_1^2 A_1^2) \sigma_{V_1}^2 + (V_1^2 D_1^2) \sigma_{A_1}^2 + (A_2^2 V_2^2) \sigma_{D_2}^2 \text{ ---- etc.}$$

I = total uranium inventory

$$\sigma = \text{deviation of the mean} = S(\bar{P}) = \frac{S(P)}{N}$$

A = analysis, average for N specimens

V = volume of solution--average for N readings

D = density (a function of the temperature) average for N temperatures

1,2,.....7 subscript for 7 sections

$\bar{P}$ = average of specimens in a sample, either A, V, or D

$S(P)$ = standard deviation of a sample

N = number of specimens in a sample

The variance for the seven sections in one inventory were:

Section 1, $\sigma^2 = 0.54$

Section 2, $\sigma^2 = 180.41$

Section 3, $\sigma^2 = 0.001$

Section 4, $\sigma^2 = 0.0003$

Section 5, $\sigma^2 = 0.014$

Section 6, $\sigma^2 = 0.050$

Section 7, $\sigma^2 = 0.026$

The total variance = $\sigma^2 = 181.06$

The total standard deviation = $\sigma_I = 13.42$

From the "t" table for $N = 6$, 13.42×2.57 gave the deviation in the inventory at the 95% confidence level of ± 34.6 g uranium in an inventory of 1550 g of uranium which is $\pm 2.23\%$ of the inventory. However the actual uranium in the system was about 1420 g so that the calculated value was biased high by 9%.

If the variance of the dump tank, Section 2, could be lowered by a factor of 100 the standard deviation would be ± 4 g of uranium or $\pm 0.27\%$ of the inventory, which would be very good. A weigh system with a 1% accuracy should lower the variance in the dump tank section by a factor of 100 (the present dump tank level is accurate to $\pm 10\%$).

The chemical analyses used in the above example had standard deviations of 0.076 in the low pressure system and 0.041 in the high pressure system which are lower than the average in Fig. 1. However these indicate that an accurate inventory could be obtained by lowering the one excessive variance and the 9% bias. This large bias could possibly be lowered some by more accurately determining the volume of each section and possibly better calibration of the thermocouples in the system.

M. W. Rosenthal has studied the requirements for experimental determination of the HRE-3 breeding ratio.² From corrosion product samples from the HRT

² M. W. Rosenthal, Experimental Determination of HRE-3 Breeding Ratio, ORNL CF-58-5-79 (May 27, 1958).

Mockup, it would appear that Cases II or III of the reference is likely. These cases assume an inventory accuracy of 0.5% and 1% uranium in the pipe film and corrosion products respectively. Case II gave a $\pm 5.7\%$ deviation in the breeding ratio after 100 days of operation and Case III gave a 5.1% deviation after one year of operation. This would be too much deviation to determine the breeding ratio accurately. Nevertheless breeding could be demonstrated in 100 days of operation.

The generation time³, which is defined as the time to generate one fuel inventory for the HRE-3, would be about 200 days. In 100 days a 50% inventory could be generated. Assuming a breeding ratio of 1.05, 47.5% of the inventory would be burned up in this time. This difference of 2.5% should be detectable with a $\pm 1\%$ accuracy in the inventory determination.

With the 3% accuracy obtained in the mock-up inventory estimate (at the 95% confidence level), precipitation of uranium at a rate of 1.6% of the inventory per day was easily detected in two days. The same precipitation rate (24 g/day) in the HRT would be equivalent to 0.4% of the inventory. It would take 7.5 days to precipitate 3% of the HRT inventory at this rate and possibly 10 days to be certain. An inventory determination accurate to $\pm 1\%$ could reveal precipitation in possibly 3 days. However the change in inventory would be revealed most quickly by the shift in critical temperature or power level.

Uranium Precipitation Tests

In Run 21 precipitation of uranium was observed with steam pressurization and 10 gph of purge water flowing through the pressurizer. This precipitate was believed to be caused by the hydrolysis of the uranium in the fuel solution as it mixed with the hot purge water in the horizontal header between the pressurizer and the main loop, shown in Fig. 2. Under gas pressurization with no purge flow the precipitate redissolved.

In Run 22 the uranium was precipitated and dissolved at various purge rates, at will. For these tests a 10 g/l UO_2SO_4 solution was used with a normal excess acid of 55 mol %. The results of these tests are shown in Fig. 3. The uranium was first precipitated with a 10 gph purge and then the purge flow was lowered to various rates and the uranium concentration was followed as it redissolved or precipitated. As shown by the wings for each point in Fig. 3 there was considerable spread in the results. The circled numbers indicate the starting and ending percent excess acid for each purge rate. At a 3 gph purge rate uranium precipitated when the excess acid was below 60% and redissolved when the acid was above 59%. At a 4 gph purge rate the precipitation appeared to stop at 72% excess acid. This point, however, was not approached from a higher percentage excess acid so it is not certain that the precipitation had stopped entirely or merely been reduced to an undetectable rate. At a 2 gph purge rate all the precipitated uranium was redissolved and the excess acid returned to the normal value of 55%.

³ M. W. Rosenthal et al., Nuclear Computations for HRE-3 Design: Equilibrium Results, ORNL CF-57-7-23 (July 10, 1957).

In the HRT some of the insulation on the horizontal pipe connecting the loop to the pressurizer had been removed and so a similar amount was removed from the mock-up to duplicate conditions, as shown in Fig. 2. This lowered the header temperature in the mock-up from 10° to 30°C depending on the purge rate. Nevertheless at a 10 gph purge rate the pipe temperature was 5° to 10°C below the main loop temperature. The effect of this lower temperature was not investigated but the removal of insulation did not prevent the precipitation of uranium.

Fuel Mixing Rate into the Circulating Pump Motor

During the first three months of Run 22, the uranium concentration in the motor end of the circulating pump was high compared to results reported by H. A. Rundell et al.⁴ In Run 22 the uranium analyses at the back of the pump varied from 1.4 to 0.55 g/l. These concentrations correspond to mixing rates of 2.7 to 3.1 l/hr for purge flows of 5 gph to 13 gph. The sample analyses and corresponding mixing rates are shown in Fig. 4. The mixing rates were calculated by assuming ideal mixing in the motor end of the pump and knowing the circulating fuel concentration, the uranium in the pump and the pump purge rate. The liquid in the back of the pump was well mixed by a secondary impeller used to circulate the liquid to keep the motor cool.

On May 9 the uranium concentration had dropped to 50% of the May 7 concentration at the same purge rate. During the last 400 hours of the run the mixing rate dropped from 3.6 l/hr to 0.04 l/hr as shown in Fig. 5. The circled numbers on the graph indicate the purge rate at the time of sampling. The mixing rates in Fig. 5 were based on uranium concentrations which decreased from 0.7 g/l to 0.015 g/l. No conclusions have been reached to explain the change in mixing rates. This study will be continued during the next run.

⁴ H. A. Rundell, D. H. Roy and J. C. Moyers, Effect of Seal Configuration on Mixing Flow, ORNL CF-57-10-48, p 6 (Oct. 10, 1957).

Fig. 1. Variation in Standard Deviation of Samples in Process Analysis

- 1 - 5 for 100% sample
- 2 - 1 for control 1.00 to 0.90
- 3 - 2 for control 0.90 to 0.80
- 4 - 3 for control 0.80 to 0.70

(2 is the number of samples in a sample)

* Values used for sample deviation

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Fig. 2. HRT Mockup Fuel Inventory Sections

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Fig. 3. Reaction of Temperature on Range Factor

(5-72) Standardized Range Factor, 10000
Range 1000 - 10000

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Fig. 5

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