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SUBJECT: HRP-CP: Sampler Operation and Uranium Peroxide
Precipitation in the HRT Chemical Processing Plant

TO: W. E. Unger

FROM: W. L. Carter

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OAK RIDGE NATIONAL LABORATORY

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DATE MAR 14 1957

April 11, 1956

For The Atomic Energy Commission

H. R. Cancee
Chief, Declassification Branch TC

TO: W. E. Unger

FROM: W. L. Carter

SUBJECT: HRP-CP: Sampler Operation and Uranium Peroxide Precipitation
in the HRT Chemical Processing Plant

It has been observed that under radiative conditions water can be decomposed and that the uranyl ion will unite with oxygen to form uranium peroxide. At normal reactor operating temperatures this compound is soluble; however, at lower temperatures in a radioactive environment it should be determined whether or not uranium peroxide will come out of solution and interfere with operations. Such a situation presents itself in the sampling operations of the HRT chemical processing plant where plugging of small apertures could seriously restrict flow.

Because of design restrictions on the sampler mechanism, the chemical plant samples have to be taken at low pressure and temperature. Volume holdup requirements dictate the use of small lines and passages in the unit making conditions favorable for flow restriction or stoppage if precipitates lodge in the system. This memorandum presents the results of a study of the chemical plant sampling system with respect to uranium peroxide precipitation.

Figure 1 presents a flow diagram of the sampling system to assist the reader in becoming oriented.

Underflow Sample

The underflow sample is taken after the completion of a two-day chemical plant run in order to obtain an analysis of the contents of the underflow receiver. Although it might be desirable to obtain samples simultaneously from the hydroclone underflow and overflow to get an instantaneous reading of the unit's performance, the construction of the hydroclone and underflow receiver is such that this operation would be very difficult. Therefore only the sample at the end of the run will be taken.

The underflow receiver contains solids of ZrO_2 , Fe_2O_3 , Cr_2O_3 and $(RE)_2(SO_4)_3$ at a concentration of about 49 gm solids per kg D_2O . Securing a representative sample of a slurry is difficult, and it must be assured that complete agitation of the tank contents is provided before a sample is withdrawn. In the underflow sample this is provided by circulating with the main circulating canned rotor.

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FIGURE 1
HRT CHEMICAL PROCESSING PLANT
FLOW DIAGRAM FOR SAMPLING OPERATIONS

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pump through the sampler and tank in series for a period of time long enough to provide homogeneity. After the chemical plant run is completed and the two valves to the reactor system have been closed, the underflow receiver will be cooled by passing air at ambient temperature through the cooling jacket. Almost immediately the vessel will lose its overpressure and drop to 1250 psia; cooling will continue until the temperature has been lowered to 85° - 90° C. During all of this cycle the circulating pump has been running to circulate the slurry through the underflow receiver. The temperature of the underflow receiver will be maintained in the range 85° - 90° C, and the pump discharge will be routed through the sampler as indicated by the heavy line on Figure 1 by freezing the pump drain line at FP-11. It is not possible to pump through the sampler before the temperature of the tank has been lowered to at least 116° C because a pressure interlock actuated by sampler pressure will not allow the valve in the sampler feed line to remain open at sampler pressures greater than 10 psig.

Circulation is continued through the circuit of underflow receiver, pump and sampler for 0.5 - 1 hour to obtain homogeneity of solution and solids throughout the system. The act of sampling is simply that of closing valves on either side of the isolation chamber which is inside the sampler, thereby trapping a small portion of the flowing stream which is subsequently drained to a sample flask.

It has been noticed that under radiative conditions and at moderately low temperatures uranium peroxide will precipitate from fuel solutions. The conditions existing in the underflow sampling operation have been examined to determine if this is a serious factor influencing sampler design especially from the viewpoint of adding a warm water coil to the isolation chamber. Previous calculations* on this same subject for the reactor samplers were made by P. M. Wood, and the data developed by him were used in these calculations.

It has been calculated that a minimum of 2 hours after isolation from the reactor system will be required to cool the underflow receiver from 300° C to 85° - 90° C. At this time the activity of the contents is estimated to be 1700 watts, or, since there are 2 gal. of solution present, the

$$\begin{aligned}\text{total power density} &= 1700/2 \times 3.785 \\ &= 225 \text{ watts/liter}\end{aligned}$$

It might be safely assumed that since the lines to the sampler are quite small - 1/4-inch Sch 80 pipe - there will be practically no gamma attenuation in these lines and that only the beta energy will contribute to peroxide formation. The ratio of beta energy to total fission product energy is 0.526; therefore,

$$\begin{aligned}\text{beta energy} &= 225 \times 0.526 \\ &= 118 \text{ watts}\end{aligned}$$

* Wood, P. M., Possibility of Peroxide Precipitation in HRT Dump and Sampler Lines, ORNL CF 54-11-172, Nov. 29, 1954.

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The graph on Figure 2 of 54-11-172 shows that the temperature at which peroxide precipitation will occur is 56°C . This is the case of the reactor operating at 5 MW; even for 10 MW operation where the power density is twice this figure, the peroxide precipitation temperature is only 62°C .

If the underflow receiver is considered large enough to permit absorption of the total beta and gamma energy, i.e., 225 and 450 watts/liter at 5 MW and 10-MW operation respectively, the respective peroxide precipitation temperatures would be 61°C and 68°C . Undoubtedly a certain fraction of the gamma energy will escape from the underflow receiver which has an inside diameter of 5.189 inches and not be available for peroxide formation. In this case the temperatures mentioned above will be slightly lower.

Since the uranium peroxide precipitation temperature depends upon the quantity of beta and gamma energy that is absorbed, this temperature will depend upon the location of the fluid and its previous history in the reactor. In any case the range should be $56^{\circ} - 68^{\circ}\text{C}$. Calculations show that heat transfer from the underflow receiver is slow enough that it is improbable that the vessel can be cooled to lower than $85^{\circ} - 90^{\circ}\text{C}$. Hence it appears that the underflow receiver itself should be free of peroxide precipitate. However a slightly different condition exists in the lines to the sampler. It is conceivable that as flow begins in the sampler line the first volume of fluid will be cooled by the cold wall of the pipe and reach a temperature low enough to precipitate peroxide. Nevertheless this should not cause plugging of the sampler or any of the lines. The entire system has been designed to handle solids in much greater concentration than a few milligrams of uranium peroxide per kilogram of D_2O , so the precipitate should flow along with other solids. It will require only a short time for the fluid to heat the entire system to underflow receiver temperature at which the peroxide will redissolve.

Overflow Sample

The hydroclone overflow sample is taken at anytime during the two-day operating period by by-passing part of the hydroclone overflow stream through the sampler. The overflow stream is at 2000 psi pressure and 300°C ; whereas, the sampler internals are designed for low pressure operation. Therefore the sampler feed must be cooled and lowered in pressure. These are accomplished by means of a cooler on the line and a throttling valve having a low coefficient ($C_v = 0.005$ at one-half open), allowing the sample stream to enter the sampler at a slight positive pressure and at approximately 90°C . Cooling water temperature can be adjusted in the range $20^{\circ} - 100^{\circ}\text{C}$ for control of the sample stream temperature. The flowrate cannot be allowed to exceed 0.75 gpm which is the flowrate through the hydroclone because at rates greater than this figure there would be back flow from the reactor and the sample would no longer be representative of the hydroclone overflow.

For taking the overflow sample the sampler housing contains a second isolation chamber similar to the one used for the underflow sample. This isolation chamber is equipped with a cooling coil through which warm water is circulated to preclude too much cooling of the sample and possible precipitation of uranium

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peroxide. The sample stream is allowed to flow through the isolation chamber for a period of time to flush the system and to assure representativeness of the sample before a 5 ml portion is isolated by closing valves around the isolation chamber. This portion is the sample which is transported for analysis via a shielded carrier. The circulating stream discharges from the sampler into the reactor dump tanks (See flow diagram Fig. 1) from which it is pumped back into the core by the fuel feed pump. The sample stream is expected to be of the order of 0.2 gpm and should not approach the capacity of the fuel feed pump.

The question of uranium peroxide precipitation in the reactor sample lines has been examined by P. M. Wood and reported in ORNL CF memorandum 54-11-172. The chemical plant overflow sample is not significantly different from the reactor fuel sample to warrant additional treatment. Using Wood's data for 100 sec decay, which is about the time it would take the overflow sample to reach the sampler, it is seen that the precipitation temperatures are 58°C and 64°C for 5 MW and 10 MW operation respectively. This also assumes that only beta energy is contributing to peroxide formation and that the lines are so small that gamma radiation escapes without causing peroxide formation.

Some of the clearances, e.g., the throttling valve and isolation chamber openings, in the overflow sample system are smaller than those in the underflow sample system, and if peroxide precipitation causes trouble it may first occur here. However there is no reason to believe that the temperature of the sample stream cannot be maintained above 58°C - or 64°C if at 10 MW - and trouble be avoided. The overflow sample cooler covers about 80% of the line from the sample tap to the sampler, and the coolant temperature in this unit can be controlled above the peroxide precipitation temperature. Likewise the isolation chamber can be held above 58°C or 64°C by adjusting the temperature of the coolant circulating around it. Downstream of the sampler, the lines are 1/2-inch Sch 40 pipe and should be in no danger of plugging.

Conclusions

Neither the underflow or overflow sampler lines should be in danger of plugging from uranium peroxide precipitation. The following reasons are offered in support of this statement:

1. The rate of heat transfer in cooling the underflow receiver is low, and, therefore, it is improbable that the vessel can be cooled below 85°C - 90°C . The peroxide precipitation temperatures are 56°C for 5 MW operation and 62°C for 10 MW operation.
2. All openings in the underflow sample system are designed against plugging for slurry operation and a few additional peroxide solids should behave just like the other solids.

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3. Temperature control on the overflow sample can be maintained to keep this sample above the peroxide precipitation temperatures since the sample line and isolation chamber coolers are cooled with hot water. Peroxide precipitation temperatures are 58° C and 64° C for 5 MW and 10 MW operation respectively.

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