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TO: F. R. Bruce

FROM: R. E. Blanco, H. K. Jackson, W. H. Lewis, E. L. Nicholson, J. W. Ullmann

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1.0 INTRODUCTION

At the request of F. R. Bruce, the authors as a committee reviewed the design of the HRT-Core Processing Plant. The purpose of this review was to obtain comments and recommendations which might result in increased effectiveness of the Chemical Plant phase of the HRT program.

The main items of interest to the committee were (1) the fate of solids generated in the reactor, (2) the design and mode of operation of the solids removal carrier, (3) the development information obtainable, (4) the maintenance techniques proposed, and (5) possible operational hazards and difficulties.

The committee met for a total of thirty hours with W. E. Unger, who coordinated the presentation of information by the individuals responsible for various phases of the design and development. The committee spent ten hours on background information on the reactor and basic chemistry, about six hours on the loop results and the remaining fourteen hours on discussion of individual equipment pieces and schemes of operation.

The committee arbitrarily limited its time and did not discuss components developed for the reactor and adopted by the Processing Plant, such as special valves and pumps. Since within the allotted time it was not possible to examine many details of layout, piping and auxiliaries, it was attempted to concentrate on items which might jeopardize either successful operation or a definitive analysis of reasons for failure.

2.0 CONCLUSIONS

The principal objectives of the HRT-Core Processing Plant can be grouped as follows:

- (1) Demonstrate rare earth removal from the reactor.
- (2) Demonstrate corrosion product removal from the reactor.
- (3) Evaluate equipment performance.
- (4) Determine the quantities of corrosion and fission products which reach the core processing plant.

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The conclusions of the committee on achievement of these objectives are:

Removal of Insoluble Fission Products from the Reactor — Rare earth solids which reach the processing plant will be removed, either by the hydroclone or by deposition on the chemical plant heater, but sufficient data are not available to predict the split. Rare earths will also deposit on the walls of the reactor equipment, but it is not known if they will continue to build up or reach a steady state. Solubilization of rare earths during reactor shutdown may permit some transfer of rare earths from the reactor surfaces to the processing plant. Quantitative predictions of solids deposition cannot be made from loop data because reactor conditions cannot be exactly simulated.

Removal of Insoluble Corrosion Products from the Reactor — Zirconium and iron oxide particles which reach the processing plant will be removed, principally by the hydroclone. The fraction of corrosion product solids which will collect in the reactor dump tank cannot be predicted at present. A portion of the corrosion products will deposit on the reactor system surfaces and it is not known whether these solids will flake off either during reactor operation or shutdown.

Demonstration of Hydroclone with Underflow Pot for Continuous Solids Removal from Reactor — The basic mechanics of the hydroclone have been adequately studied. The size, shape and density of the particles in the reactor system are not known and because of the experimental difficulties involved will not be determined during operation. If the degree of solids removal attained is unsatisfactory, only indirect evidence will be available, therefore, that the particles were of a type not removable by the hydroclone. Even if the effluent from the hydroclone is clear, it will be impossible to state what fraction of the solids remained in the reactor system without dismantling or descaling the reactor. Since the compositions of the streams to and from the hydroclone are almost identical, it is crucial to determine the weight and analysis of the solids collected. The quality of the solids sample will depend on how well the core processing system is descaled and drained of solids after each run and therefore conclusions from any particular run may be dubious. The whole problem of analyzing how much better

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off the reactor is by virtue of the processing plant is a formidable one and has not been completely solved.

Demonstration of Heater — The use of a heater for removing ions or solids from solution by deposition has been demonstrated but very little is known about the rates or mechanisms which are involved.

Determination of Reactor Core Corrosion Rate — Since some corrosion products will remain in the reactor system only a minimum estimate of Zr corrosion rate can be obtained.

Purging of Soluble Fission Products by Carrier Removal — The removal of evaporated core solution associated with the solids collected is a difficult operation as now designed. The operators must cut open the pressure membrane of the chemical plant cell, remotely disconnect a vessel, place the vessel into a shield, transfer the shielded unit to another processing site, remotely connect a new vessel and reweld the cell membrane.

Addition of a Fresh Fuel to Reactor — The design is workable but some members of the committee are concerned over the use of a line directly from an operating area to the high pressure system. Operation depends on valves and interlocks rather than a break, vented to the cell.

Demonstration of Underwater Maintenance — Underwater connections will be made and broken as part of the plant routine. Since no provisions have been made for equipment decontamination the shielding water will become contaminated. The system is not designed for handling radioactive water.

Analysis of Solids Removed — If the accuracies in remote equipment are of the same order as demonstrated on non-radioactive samples in conventional equipment, analytical results will be satisfactory.

3.0 RECOMMENDATIONS

The principal recommendations of the committee are to:

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(1) Attempt to provide a means to determine separately solids deposition on the chemical plant heater and screen.

(2) Restudy the system of removing collected solids from the cell to avoid the present handling problems. If possible, a clear solution rather than a slurry should be transferred for uranium recovery.

(3) Install a plant simulating the type of system which we would propose for a commercial power reactor, as soon as schedules permit. At present we remove undesirable solids from a solution and subsequently redissolve them, whereas it would be preferable to discard the solids. Solvent extraction equipment may not have to be included, but a different hydroclone arrangement and heavy water recovery system would be desired and a feed preparation step must be demonstrated.

(4) Devise a less corrosive system for heavy water recovery from sulfuric acid slurries. Addition of a neutralizer is one possibility.

(5) Provide a means for determining the weight or volume of the contents of the carrier.

(6) Design a means for reducing bumping and spattering during heavy water recovery.

(7) Check if it is advisable to change the fuel introduction system to avoid the connection between the cell and operating area.

(8) Increase the margin of safety of the overflow sampler by additional instrumentation.

(9) Check to see if unit shielding can be installed on the shielding water pump and if shielding water can be filtered or decontaminated.

(10) Try to prevent gross contamination of the shielding water and outside surfaces of equipment, perhaps by location of the suction of a pump close to the open flange.

(11) Consider alternate high pressure connectors or improve the flange and gasket arrangement to maintain a seal after the connection has been broken and reassembled.

(12) Revise the valve control system to prevent rupture of the high pressure system as a result of decay heat following a power or air failure. Emergency override of at least one of the valves to the reactor may be indicated.

- (13) Devise tests to determine the particle sizes of alkaline earth sulfates in loops and their efficiency of removal.
- (14) Determine more fully the degree of coprecipitation of rare earths, corrosion products, copper and uranium, possibly in the presence of coprecipitation agents such as aluminum.
- (15) Evaluate the worth of more positive means of decontaminating or unplugging lines and equipment.
- (16) Check bearing wear of a canned rotor pump with solids present during underflow pot recirculation.
- (17) Install a tee for future iodine removal.
- (18) Increase the amount of sampling in reactor and chemical plant mockup tests to determine the fate of solids more accurately.
- (19) Study the effect of flow and temperature difference on solids deposition on surfaces.
- (20) Change hydroclone retainer plug design to make remote removal easier.

4.0 BRIEF PLANT DESCRIPTION

Uranyl sulfate solution containing suspended solids is bled from the reactor main circulating loop into the processing plant where it flows through a heater and screen to a hydroclone with integral underflow collection pot. The heater decreases solubility of rare earths and retains part of them; the screen protects the hydroclone against plugging by large particles. The clarified stream from the hydroclone is sampled and returned to the reactor with a pump which makes up the pressure head lost in the processing plant. The pump rate is controlled by the pressure drop across the hydroclone. Solids centrifugally removed by the hydroclone are held in the underflow pot for the duration of a run.

At the end of a run (estimated to be two days each week) the processing plant is isolated by valves from the reactor and the pump is used to recirculate the slurry collected in the underflow pot through the heater and screen while pressure is being reduced by cooling. The slurry is recirculated through the pot and heater, sampled and dropped by gravity to a carrier.

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The carrier will be used for 10 to 20 solids batches with heavy water evaporation between batches. Decomposed heavy water is recombined and condensate is collected or returned to the carrier as desired. Non-condensable gases pass through a gas train for decay holdup and exhaust to the stack.

The carrier is periodically removed from the cell for sampling and recovery of uranium. The system is put on stream again by filling the underflow pot with fresh uranyl sulfate and opening the valves to the reactor.

5.0 SOLIDS REMOVAL FROM THE REACTOR

Table 1 summarizes the data presented to the committee on simulated HRT solids.

Rare earths will require about a month's operation of the HRT to reach their solubilities, corrosion products about a day. Since the production rate of fission products is low at HRT power, augmentation of this rate by injection of rare earths has been suggested. This does not appear to be a desirable procedure except possibly for non-radioactive shakedown runs because the injected material may precipitate differently from rare earths generated in the reactor.

Additional work is necessary to check the coprecipitation of copper, uranium, fission products and corrosion products as a function of temperature. The use of low cross-section additives to reduce rare earth solubility may be a promising approach.

A check should be made of the poison expected from the products of burned-out fission products.

Particle size determinations have been made on corrosion products by injecting Fe, Zr, and Cr into loops operating at simulated reactor conditions. Rare earth particle sizes could not be determined in the loops because the precipitates redissolve on cooling. Laboratory observations in static rare earth systems show a large variation in particle size, i.e., from 100 microns to mm size. Due to thermo-cycling in the

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Table 1

Simulated HRT Solids

<u>Solid</u>	<u>Production Grams/day</u>	<u>Solubility at 300°C, 2000 psi ppm</u>	<u>Loop Runs Observed</u>		<u>Temperature Effect</u>
			<u>Particle Size, μ</u>	<u>Concentration Factor</u>	
Iron Oxide	10	0.5 - 2	0.04 - 0.5	100	Insol. at low temp.
Zirconium Oxide	40	1 - 3	1 - 35	4500	Insol. at low temp.
Chromium Oxide	2	0.5 - 2	Unknown	--	Insol. at low temp.
Rare Earth Sulfates	1.3	65	Unknown	65 - 100*	Inverse Solubility
Alkaline Earth Sulfates	0.3	10	Unknown	--	Insol. at low temp.

*Nd used in test

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reactor system the rare earth particle size cannot be predicted and will probably never be known other than by reference to the performance data of the clone. Alkaline earth particle sizes have not been observed either in laboratory or loop tests.

The effect of radiation on particle size is not known. It is expected that very large particles will be chewed up by the reactor pump, and very fine rare earth particles will grow from thermal cycling in the reactor loop, although supporting data are not available.

Solids tend to deposit on surfaces which are above 100°C. The hotter the surface the greater is the deposition which has been observed, but it is not known if the surface becomes saturated or if buildup continues. It is therefore expected that a significant fraction of the solids will deposit on hot surfaces within the reactor system. Additional study of the mechanism of deposition and the effect of variables such as flow rate, fluid temperature, and temperature difference between fluid and surface is recommended.

It will not be possible to establish the quantities of solids accumulated in the reactor system without descaling the reactor and cleaning it out completely. Although corrosion specimens in the reactor and nickel analyses will provide some order of magnitude estimates, solids may reside in the reactor dump tank where they cannot be accurately sampled. During reactor operation the contents of the dump tank cannot be pumped to the processing plant. After shutdown, descaling is still required since there is no reason to expect that solids which were not pumped out during operation would be removed by the same pump after shutdown.

A mock-up test is in progress at Y-12 to determine the quantities and particle sizes of solids held up in the dump tank and centrifuged by the tee through which fluid is withdrawn from the reactor and routed to the processing plant. The use of monitors and tracer La^{140} , and Zr^{95} in the mock-up does not appear to be a sufficiently accurate method. Provision should be made to flush and sample the dump tank, clone underflow pot, and heater. Samples should be taken from the clone overflow for comparison with the equilibrium loop solids concentration.

Since large particles may collect on the screen, and deposition is expected on the chemical plant heater, it is recommended that a means of analyzing the performance of each unit be sought. The program calls for runs without the heater and without the hydroclone — this is the simplest suggestion but assumes that conditions have not changed between runs.

As a run progresses, the concentration of solids in the overflow stream approaches that of the feed to the hydroclone. The initial difference expected for rare earths is 30% and the results will probably not be significant. The initial difference for corrosion products may be as high as 95% and the comparison may have significance.

The best measure of the quantity and compositions of solids removed is by analysis of the material collected in the underflow pot. Slurrying and sampling of the solids is difficult and the quality of the sample will be checked by analyzing the entire contents of the carrier. Any carrier corrosion will obscure the check and must be avoided.

The analytical accuracy obtained in conventional equipment with non-radioactive samples is adequate to establish concentration factors across the hydroclone. The accuracies reported are $\pm 3\%$ for iron, zirconium and rare earths. The accuracy obtainable with remote manipulation has not been established. The accuracy of radiochemical analyses is estimated by analytical personnel to be $\pm 5 - 10\%$ after a decay period of one week. The effect of this decay will have to be considered in calculating performance data.

6.0 EQUIPMENT DESIGN

6.1 Heater

Pressure taps are not installed across the heater and plugging or partial plugging from solids deposition will have to be determined qualitatively and indirectly as shown in Table 2. The temperature of the exit stream could be recorded on a narrower range instrument to improve accuracy. Since the deposition of solids on a heated surface is of prime interest, the committee feels any instrumentation which can clarify the deposition

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question is justified. The effect of putting the heater after, instead of before, the hydroclone has not been studied. Failure of two of the four calrod heaters would not cause a shutdown. The effect of fission product decay heat is small compared to the electrical heat.

Table 2

Effect of Erosion or Partial Plug

<u>Event</u>	<u>Flow</u>	<u>Pump Frequency</u>	<u>Temperature Difference</u>	
			<u>Across Heater</u>	<u>Through Heater Block</u>
Clone Plugs	Decreases	Decreases	Increases	Decreases
Clone Erodes	Increases	Increases	Decreases	Increases
Heater Plugs	Same*	Increases	Same or Decreases	Decreases
Screen Plugs	Same*	Increases	Same*	Same*
Line Plugs	Same*	Increases	Same*	Same*
Pump Erodes	Same*	Increases	Same*	Same*

*Within limits of pump developed head.

6.2 Screen

Since pressure taps are not installed across the screen, and no temperature change occurs, no information can be collected on the performance of the screen. Loops have plugged between the heater and screen, and with the present instrumentation it will probably not be possible to determine if a plug occurs in the heater, in the screen, or between them. No back-wash system is installed for the screen, but in an emergency high pressure could be applied to free recalcitrant particles.

6.3 Hydroclone and Underflow Receiver

Loop runs have demonstrated concentration factors of about 50 with 0.7μ Th_2O_3 particles and about 100 with Fe_2O_3 particles smaller than 0.5μ . If the particles which reach the hydroclone are not much smaller than 1μ , as is expected for corrosion products, they should be easily removed.

The principal concern of the committee over hydroclone operation is the danger of a plug in the bottom drain from the underflow receiver caused at least partly by uranium peroxide precipitation in the neighborhood of the freeze valve. Further study of this point may be warranted because the underflow drain is one of the most likely places for a plug to occur.

6.4 Canned Rotor Pump

The pump has been placed at the end of the chemical plant high pressure system and during the solids removal part of a run receives clarified solution. At the conclusion of a run, however, the pump is used to try to reslurry all the collected solids (about 4% solids by weight, of unknown particle size) by recirculation. The committee is concerned over possible bearing troubles caused by the solids and perhaps a test run can be made to check this point.

6.5 Flanges, Freeze Plugs and High Pressure Valves

These units were designed by REED and the construction details were not checked. There is some danger that a tight seal will not be effected with the valves in the presence of solids and if the leakage exceeds a maximum rate of 5 cc/min a freeze plug cannot be effected. There is no simple way of determining if a valve is leaking prior to the point at which the input of heat from the leak exceeds the removal capacity of the freeze plug coolant.

Unit Operations' reports on the difficulty of making a tight seal between flanges after they have been broken lead to the recommendation that other high pressure connectors be examined for applicability. In the case of routinely broken low pressure connections, such as the carrier, the use of flanges to permit leak detection may be particularly undesirable.

6.6 Carrier-Evaporator

It is proposed to transfer the slurry from 10 to 20 runs (400 grams solids per run) into a carrier and ^{evaporate} heavy water between batches. For the first few runs only one batch will be transferred to the carrier in order to provide a check on the solids sampler.

To remove a carrier the operators must cut open the cell, fill with shielding water, remotely disconnect the carrier, place it in a shield, and remove the carrier. The next carrier is then remotely installed and the cell welded shut. The solids removed in the carrier must be dissolved for analysis and uranium recovery. The details of the recovery step have not been adequately worked out.

The committee recommends consideration of dissolution in the cell in a corrosion resistant boil-down tank with suitable instruments to determine volumes and obtain samples. D_2SO_4 would be used to dissolve solids prior to sampling. Solutions would be allowed to cool in storage tanks prior to jetting to outside carrier for removal to solvent plant.

The present system of carrier evaporation will probably lead to bumping and solids spattering and consequent contamination of shielding water when the connections are broken. There is no method of checking the tightness of the valves remotely. It is suggested that deflector baffles be incorporated into the vessel to minimize the effect of spattering. Termination of the inlet line several inches above the bottom of the vessel may minimize plugging.

It is proposed to distill all water from the carrier prior to removal. With the steam pressure available it will not be possible to concentrate to over 70% H_2SO_4 . It may, however, be preferable to lose some heavy water, and design for radiolysis rather than dissolve a dried mass.

The corrosion rate of the stainless vessel may be several hundred mils per year during the evaporation of water from sulfuric acid. Corrosion would add material which cannot be distinguished from solids removed from the reactor. It may be desirable to add a corrosion inhibitor such as copper sulfate to each empty carrier. Coupons should be installed to check the fraction of solids due to corrosion from boildown. The effect of pouring aluminum around stainless steel on corrosion should be checked.

Water vapor is required to dilute possibly explosive radiolysis gases, but no instrumentation is provided to determine directly the quantity of water in the carrier. The weight of water present is found from the heavy

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water inventory and checked by integration of the flow rate of steam distilled. The inventory check may lead to cumulative errors and the steam flowmeter is susceptible to corrosion by sulfur trioxide. It is recommended that remote weighing of the carrier be investigated. Since the solids content and therefore freeboard in the carrier prior to removal can only be estimated after analysis of the solids sample, weighing may indicate after how many batches the carrier should be removed.

A suggested alternate method of carrier operation which does not require redesign consists of adding an excess of K_2CO_3 or Na_2CO_3 (over that required to neutralize all acid in the anticipated number of slurry batches) to the carrier before it is connected to the system. The advantages of this procedure appear to be:

- (1) Neutralization of all free sulfuric acid and maintenance of an alkaline environment that will be less corrosive to the carrier. No non-noble material of construction appears to be completely satisfactory for the acid environment.
- (2) Elimination of stable hydrates ($H_2SO_4 \cdot 2H_2O$) permits the attainment of complete dryness in the carrier and eliminates any radiolytic gas evolution during short or long term storage.
- (3) Possible formation of a friable, easily re-slurried solid residue in the carrier.
- (4) Less possibility of thermal decomposition of $SO_4^{=}$ and evolution of SO_3 which would poison the recombiner catalyst.
- (5) Potential elimination of the necessity for dissolution of all solids in the carrier because it may be possible to remove slurry from the carrier, sample and dissolve the samples only. Uranium could be recovered from the diluted slurry by solvent extraction without a corrosive complete dissolution of the slurry. Thus the whole sampling and uranium recovery operation could be done with existing pilot plant equipment and a small hot cave on top of the cell block.

Disadvantages of this system are:

- (1) Foaming due to CO_2 evolution during slurry addition to the carrier.

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(2) Possible increase of bumping and splattering of the carrier contents upon evaporation to dryness.

(3) The possibility of contaminating the reactor with the neutralizing agent.

6.7 Off-Gas System

If solids are bumped from the evaporator, plugging of the jackleg on the entrainment separator may occur.

It is recommended that the possibility of sulfur trioxide poisoning of the recombiner catalyst be checked. A hot wire deuterium monitor is suggested to check the degree of recombination.

The cold trap is cooled with cooling water. Refrigerant cooling to prevent water vapor from the evaporator from entering the adsorption beds should be installed if sufficient excess bed capacity is not available.

6.8 Heavy Water Storage

Both weigh cells and thermo probes are used to check the volume of heavy water stored. Either is probably adequate but a float system may be simpler.

6.9 Fuel Charging System

Instrumental interlocks are installed to prevent an operator from opening the charging line if there is pressure within the cell equipment. When the charging line is open, one valve and one freeze plug separate the reactor from the operating area. Additional safety may be obtained by inclusion of a positive seating check valve or by the use of a vented funnel.

6.10 Sampling

The sampler is to be mocked up and tried. Analytical personnel report that liquid is spilled during cone removal. Provision should be made to be able to decontaminate the sampler housing. Interlocks should be added to prevent opening the sampler if there is high pressure between the isolating valves, and to prevent sampler operation if the valve in the discharge line to the dump tank is closed.

7.0 PLANT OPERATION

7.1 Maintenance

In order to perform any maintenance on cell equipment the steel membrane over the cell must be cut, and, after completion of the job, rewelded. Underwater removal and replacement of units is planned but weld or flange failures or repairs on equipment require contact maintenance. Layout and equipment have not been specifically designed to aid contact maintenance.

No provisions are made for decontaminating the shielding water. Maintenance of the shielding water pump may be difficult unless unit shielding can be added. It is recommended that a "vacuum sweeper" technique be used to reduce contamination of the water when performing maintenance. Light water will have to be removed from the system after some maintenance jobs; perhaps, by pressurization, out of the sampler.

If corrosion of the retainer plug or solids deposition in threads gives trouble in the removal of the hydroclone retainer plug, an auxiliary "O" ring gasket could be used to seal the threads from the solution. Acme instead of "V" threads may permit use of type 347 instead of type 316 stainless steel for the retainer. Annealing the type 316 stainless steel retainer after machining may reduce corrosion. Gold or MoS_2 lubrication may reduce galling.

7.2 Decontamination

Very little attention has been given to decontaminating the equipment. Solutions can be introduced via the sampler or fuel addition system if necessary.

7.3 Hazards

Careful attention has been given to this aspect of the plant design. Items discussed in previous sections which might increase the safety of the operation are:

Less direct fuel introduction (Section 6.9)

Bumping in carrier-evaporator (Section 6.6)

Recombiner gas monitoring (Section 6.7)
Carrier weight measurement (Section 6.6)
Sampler valve interlock (Section 6.10)
Shielding water contamination (Section 7.1)

In case of an air or power failure the main isolation valves between the reactor and chemical plant will close. The pressure of the system will increase from radioactive decay heating. Emergency override of the valves is suggested.

Quantitative investigation of the explosion hazard from radiolysis during carrier transport is recommended.

8.0 DESIGN REVIEW COMMITTEE CONCEPT

A review committee should point out weak spots rather than provide solutions to them. In practice, however, a clear distinction cannot be drawn since criticism of a design feature implies that a better solution can be found. Considerable time was spent by the committee checking alternate proposals with the designers to determine reasons why they had not been used.

Much time would have been saved if a status report had been previously prepared in which the basic data, objectives and limitations of the plant were listed.

The committee did not uncover areas of difficulty which had not been recognized — at least to some degree — by the designers. Since the opinions of people not intimately connected with a design may lead to a shift of emphasis, it is believed that the review would have been more useful if it had been instituted several months earlier.

J. W. Ullmann

J. W. Ullmann
Chemical Technology Division
(For the HRT-Core Processing
Plant Design Review Committee)

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HRT-CHEMICAL PLANT BLOCK SCHEMATIC FLOWSHEET

Figure 1

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