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TO: M. T. Kelley

FROM: W. L. Carter

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O A K R I D G E N A T I O N A L L A B O R A T O R Y

June 7, 1956

TO: M. T. Kelley

FROM: W. L. Carter

SUBJECT: HRP-CP: Analytical Requirements for the
HRT Chemical Processing Plant

CLASSIFICATION CANCELLED
DATE MAR 16 1957 *RET*
For The Atomic Energy Commission
H. F. Canale
Chief, Declassification Branch

A previous memorandum, ORNL CF 56-4-101, on this subject detailed an analytical schedule for the HRT core processing plant. However, since its publication, slight modifications to the plant and its function as well as valuable comments from project personnel have indicated a need for supplementing the original schedule. It is the purpose of this memorandum to point out several additional analyses that will be required and to make suggestions which will work toward minimizing the possibility of contaminating the reactor.

Cl, F, NO₃, S, AND SO₄ IN HYDROCLONE OVERFLOW: Table I of the reference memorandum schedules the analyses of the hydroclone overflow stream, and at any particular instant this sample is representative of the reactor fuel solution. It is suggested that, in addition to the requested analyses, chloride, fluoride, nitrate, sulfide and sulfate ion concentrations be obtained. These ions--except SO₄--are corrosive and/or incompatible to reactor operation, and their concentrations should be checked. The SO₄ ion is a necessary ingredient of the HRT fuel, and in this case the uranium to sulfate ratio is desired. It will not be necessary to analyze all of the hydroclone overflow samples each week for these anions; however, at least one sample per week should be checked. Table II of ORNL CF 56-4-101 requests analyses on the hydroclone underflow similar to those of Table I; however, it is not believed necessary to assay the hydroclone underflow for these anions.

MANGANESE AND MOLYBDENUM: It is further recommended for Tables I and II that in the analyses included under CORROSION COMPONENTS the element manganese be added. Also, molybdenum should be included in the portion of these tables under the heading FISSION COMPONENTS. Both analyses should be made from the viewpoint of quantitative information for material balance purposes.

NICKEL AND TIN: It has been the experience¹ with the system UO₂SO₄ - CuSO₄ - NiSO₄ - H₂SO₄ - H₂O that under certain conditions nickel is precipitated with uranium as a yellow or orange-yellow solid. The remarks in ORNL CF 56-4-101 concerning nickel analysis imply that this element will probably be found in solution;

¹ Secoy, C. H., A Study of Dilute Solutions in the System UO₂SO₄ - CuSO₄ - NiSO₄ - H₂SO₄ - H₂O, ORNL CF 56-5-114.

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however, it should be remembered that a portion of the nickel can be present as a precipitate. Under proper conditions of temperature and excess acid concentration, a nickel precipitate can occur at nickel molalities as low as 0.02. A further anomaly in the original schedule has been observed. The behavior of tin in the system will be similar to zirconium and will be present as the precipitated oxide. Therefore, attempts to follow core tank corrosion by tin analysis must include a thorough analysis of the hydroclone underflow solids and solution for this element.

ISOTOPIC PURITY OF RECOVERED D₂O: With the advent of a dissolving system for the HRT core processing plant, the desirability of periodic checks on the purity of recovered D₂O has been introduced. Before each addition of recovered D₂O from vessel T-2 to the reactor system, a sample will be taken for isotopic purity analysis. The frequency of such samples would be one per week, and isotopic purity should be of the order of 99.95+% D₂O.

ISOTOPIC PURITY OF STOCK D₂O: As purchased, D₂O is around 99.98% isotopic purity. It is recommended that before any stock D₂O is added to the plant its isotopic purity be checked to preclude H₂O contamination. A maximum of one sample per week is anticipated.

Cl, F, NO₃, S IONS IN D₂O: Further suggestions for analytical procedures are concerned with securing thorough checks on all ingredients added to the chemical plant or to the reactor via the chemical plant. Having such assays will greatly assist in determining the source of any unexpected contamination that might be observed in the system. In this respect all heavy water that is used for fresh fuel make-up solutions or flush solutions should be analyzed for chloride, fluoride, nitrate, and sulfide ion before addition to the plant. These anions, if present, would be in trace quantities, and a maximum of one sample per week would be submitted.

U²³⁵ ANALYSIS: After each fresh fuel make-up, it would be well to check the isotopic purity of the uranium and its thorium content when the solution is analyzed for chemical composition. A maximum of one sample per week would be submitted. It has not been decided whether this sample will be submitted by REED laboratory personnel or by the core processing plant operator, since the procedure for make-up of fresh fuel has not been specified. If the laboratory prepares the fuel solution and submits a sample, it should not be necessary for the chemical plant operator to submit a second sample. However, a definite procedure should be formulated so that a sample will be taken.

CuSO₄ AND H₂SO₄ MAKE-UP: The usual ionic contaminants of CuSO₄ are Cl, N, Ni, and Fe and of H₂SO₄ are Cl, NO₃, As, Pb, and Fe. The purity of these chemicals should be checked before each addition to the plant.

DESCALING OR DECONTAMINATING SOLUTIONS: The object in obtaining analyses on these solutions is to have a record on all atoms going into the system.

LIQUID AND SOLID PHASE ANALYSES: In Tables I and II of ORNL CF 56-4-101, it was pointed out that both the liquid and solid phases of the samples should be

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tested for U and Cu. However, it was not thought necessary to analyze both phases for all of the rare earths and other fission components, the contention being that an analysis for these in the supernate would suffice. Nevertheless, it is now believed that valuable data on the behavior of fission products in the system could be found by analyzing both supernate and precipitate for these atoms. It is reasonable that fission atoms might plate out on the reactor surfaces and become interstitial with corrosion product solids and thereby be overlooked in an analysis of the supernate alone. More information would be obtained by analyzing the supernate and precipitate separately than by putting everything into solution and making one assay; an analysis on the one dissolved phase would give only the total and not the ratio of fission products in solids to fission products in solution. It might be that certain atoms are selectively adsorbed, and this would be valuable information for future process design. In view of this, it is recommended that both supernate and solids from the hydroclone overflow and hydroclone underflow samples be tested routinely for all atoms. However, if it is observed that the precipitate is not effective at retaining fission product atoms, there would be no reason to continue these analyses.

DUPLICATE SAMPLE STORAGE: It has been suggested that two samples of all materials added to the plant be held in storage for future check analyses. These would include samples of each fuel addition, D₂O addition, decontaminating or descaling solution, and anything else that finds its way into the reactor system. These would be cold solutions and storage would be no problem; it is somewhat impractical to attempt such a program on samples removed from the plant. With such an inventory any doubtful points could be checked, and there would always be a record of everything entering the system through the core processing plant. It is recommended, therefore, that the operator of the core processing plant set up such a storage.

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