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MOUND LABORATORY-MONSANTO
Central File No. 54-7-57

Date: July 19, 1954

To: Dr. R. A. Staniforth

Subject: Trip to Fernald July 9, 1954

On July 9, 1954 J. J. Burbage, R. A. Staniforth, J. R. Wiesler, F. L. Halbach, F. C. Mead, Jr., and D. L. Timma visited FMPC at Fernald. Dr. Roberson and Mr. Walker also attended the meetings. The primary conversations were with the following members of the AEC staff at Fernald: Mr. Karl, Dr. Robinson, Mr. Stafford, Mr. Anderson and Mr. Hughes.

The AEC personnel outlined both the uranium and the thorium process at Fernald. They then proceeded to outline a series of problems which confront the Fernald operation and which they thought might prove of interest to Mound as problems in the Feed Materials area.

The final purified uranium is in the form of aqueous uranyl nitrate solution with a uranium concentration of about 70 g/l and about 0.03 N in HNO_3. This solution is concentrated by evaporation in 347 stainless steel tanks to a concentration of about 1600 g/l. In this process the iron contamination rises to about 30-70 ppm and nickel impurity to about 10-20 ppm. Both of these values are above specifications. This indicates the need for a rapid reliable method for iron and nickel determinations in these concentration ranges. The possibility that inhibitors might prevent the corrosion of the tanks should be investigated as an immediate solution.

A more acceptable solution was believed to be the development of a continuous denitration process which would take the 70 g/l OK liquor and convert it to UO_3 of acceptable density and purity. All plants are now using a batch process for denitration and this is thought to be a point where impurities are introduced as well as a holdup point in the overall process. Work in this field appears promising.

Trouble is being experienced in getting complete denitration of the AR raffinate from the first extraction column. This material is a complex mixture of metal nitrates which are spray calcined to recover the nitrogen oxides. Current operation leaves about 5-15 per cent nitrates with the metal oxides which is uneconomic and later presents a corrosion problem. They would like optimum operating conditions for the calciner. This appears to be a problem of developing proper operating conditions for an existing piece of plant equipment and the work could best be done at Fernald.

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NAME J.M. FLANAGAN
2ND REVIEW-DATE: 2/19/98
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The UO_3 produced must be in an "active" form before hydroflurination. Exact knowledge of what the active form may mean is unknown, however its existence is measured by the per cent conversion to UF_4. Oak Ridge has done some work on activation of UO_3 by making a slurry with water and heating.

The UO_3 should have a high density ($\rho = 2.5 - 3.0$) if the UF_4 produced is to have a high density. High density UF_4 is desired because it influences the quantity of uranium metal which can be produced per bomb reduction. Consequently problems of densification of UF_4, briquetting UF_4 and Mg, and the study of the kinetics of the UF_4 - Mg reaction were suggested.

The reactivity of uranium produced by both Fernald and Mallinckrodt is thought to be decreasing over the past few months. This might be arising from many sources, some of those suggested were; (a) rare earth contamination below the level now being detected by analytical procedures, (b) corrosion contamination from Carpenter series 20 stainless steels which might contain rare earths, and (c) current decrease in TDS. Our experience and interest in neutron work would indicate the desirability of work on the development of improved test methods of determining reactivity.

The remelt step to convert the small uranium derbies to larger ingots is now done by batch operation in small vacuum furnaces. This leads to difficulty in maintaining comparable purity figures on all the melts. Present thinking is to convert to multi-ton melts in air. Battelle is now melting up to 1,200 pound batches in air. A problem still remains of developing a flux or slag to prevent excessive pickup of oxygen and nitrogen from the air. The use of MgF_2 from the bomb liners has been suggested.

Fernald has a considerable problem in handling their scrap and recycle material. Of each 100 pounds of uranium feed to the plant 44 pounds goes through recycle. Presently they take dolomite material, magnesium fluoride liners, old crucibles, etc. and treat them with HCl. An extract is filtered off containing uranyl chloride which is precipitated from solution with ammonia. The ammonium uranate is then ignited to U_3O_8. Considerable trouble has been experienced in getting U_3O_8 which was low enough in fluoride ion concentration. They need some agent to complex chloride and fluoride ions. They now have hundreds of barrels of cutting oils, originating from uranium machining, which contain recoverable quantities of uranium. They also have quantities of contaminated clothing and shoes for which they need to have a method developed for uranium recovery.

The present method of thorium production is the co-reduction of ThF_4 and $ZnCl_2$ with metallic calcium. The calcium used is redistilled metal of high purity. There appears to be some question on whether the ultrahigh purity calcium is required to give thorium metal of sufficient purity. The calcium metal is very expensive and National Lead is trying to locate suppliers at a lower cost. (They indicated the Dominion Magnesium of Toronto had bid on supplying the calcium at $2.75 per pound).

Some trouble has been experienced in melting thorium since stabilized zirconia is the only known crucible material that can be used at the melting point of thorium. They suggested that arc melting might solve this difficulty and suggested work on this process.

Fernald has a contract with Horizons, Inc. of Cleveland for the development of an electrolytic process for production of thorium metal. Their proposed process depends on the electrolysis of $ThCl_4$ dissolved in a bath of molten NaCl. The early runs are encouraging as far as the electrolysis is concerned, however considerable difficulty has been experienced in developing a method for preparing $ThCl_4$. A cheap source of $ThCl_4$ might also make possible the development of a Kroll-process-type of reduction with magnesium. In scale up work Horizons is experiencing difficulty in finding crucible materials to hold the bath.

In general firm specifications on thorium nitrate tetrahydrate and on thorium metal have not been developed. This is primarily true because of the lack of good reliable control methods for impurities in thorium materials. Extensive work needs to be done in developing rapid process control methods.

Mr. Robinson of the AEC staff felt that if they were to look at the uranium metal production program now as a new program that direct fluorination of ores and concentrates would be seriously considered. Oak Ridge once studied this problem and made cost estimates for the process. However, since that time the chemical composition of the ores and concentrates being supplied have changed and the ores are considerably poorer in fluoride-consuming impurities. This change has been significant and he felt that a new economic study was justified based on the new materials. Argonne has done considerable work on the fluorination of materials using bromine trifluoride.

In the afternoon Mead, Haubach and Timma were taken on a tour of the thorium pilot plant area by Mr. Anderson.

A meeting was held with National Lead personnel in the afternoon. Those attending were Burbage, Staniforth, and Walker from Mound and Dr. G. W. Wunder, Plant Manager, Mr. Noyes, Assistant Plant Manager, and Arnold, Noyes and Ciborski from Fernald. At this meeting the National Lead people suggested the following problems.

1. Need for analytical methods for thorium.
2. Need for an analytical method for rare earths in OK liquor - preferably a flowline process.
3. Study of the continuous denitration of UNH.
4. Continuous reduction technique for U and Th.

5. Determination of the necessity for high purity specifications for calcium.
6. Study of thorium recycle to determine effects of impurities present.
7. Method for removing chloride ion from Brazilian sludges.

DL Timma

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D. L. Timma

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