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8. TW Hauff
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FROM: R. B. Richards RBR
Separations Technology Unit
Technical Section

MEETING NO. 1 WITH CARBIDE & CARBON (K-25) PERSONNEL
ON HANFORD URANIUM OXIDE PERFORMANCE

BACKGROUND

Beginning February 27, 1952 the K-1131 feed plant at the Carbide and Carbon K-25 installation processed 26,200 pounds of Hanford oxide (Lots 003 through 006). This amount of material was adjudged sufficient to test the efficiency of conversion to UF_4 (green salt) under process conditions known to be suitable for Mallinkrodt UO_3 and Harshaw UO_3 . On March 17 and 18, R. B. Richards, A. R. Maguire, F. W. Woodfield and W. N. Mobley of the General Electric Company, together with J. T. Christy of the HOO-AEC met with representatives of Carbide and Carbon to discuss the performance of the Hanford product and to assist in the formulation of development programs directed toward the eventual

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realization of satisfactory UF_6 produced at a suitable rate through the K-1131 feed plant. The following discussion describes briefly the performance of the Hanford oxide in the K-1131 feed plant together with agreements on future proposals directed toward improving the product quality and K-1131 production rate.

PERFORMANCE OF HANFORD UO_3

The K-1131 feed processing plant will eventually have three lines, A, B, and C, each consisting of a 15-foot vibrating reduction tray ($UO_3 \longrightarrow UO_2$), 40-foot and 30-foot vibrating trays in series for hydrofluorination ($UO_2 \longrightarrow UF_4$), and 6-inch diameter fluorination tower ($UF_4 + F_2 \longrightarrow UF_6$).

During Part I of the processing, 8721 pounds of Hanford oxide were processed through the 15-foot reduction tray (normal 95-98% conversion) and the 40-foot hydrofluorination tray under conditions of temperature, retention time, and vibration frequency that proved suitable for Mallinkrodt and Harshaw UO_3 . At a product rate of 101 pounds per hour, the conversion efficiency to UF_4 was 76%. At similar product rates the Harshaw and Mallinkrodt oxides gave 85% and 91% conversion respectively.

During Part II of the processing, the remaining Hanford material was processed at an average rate of 234 pounds per hour through the 40-foot and 30-foot hydrofluorination trays in series to give a UF_4 conversion efficiency of 70%. At similar throughputs, Harshaw and MCW oxides were 90% converted.

Subsequent fluorination of UF_4 , because of the high fraction of unconverted UO_2 , consumed a greater than normal amount of fluorine with an accompanying build-up of solids on the walls of the fluorination tower. These solids analyzed 75% U, 21% F^- , 0.9% Na, 0.34% R_2O_3 . Such solid build-up had been noticed previously when relatively large amounts of unconverted UO_2 were fluorinated in the tower. At the conclusion of the test, a small amount of white fluffy powder was removed from the UF_6 exit line from the fluorinator. The powder was composed of 42.2% U, 29% F^- , 20.5% Na, 0.5% Al-Fe, 0.015% H₂, 0.04% Cr, and 48 ppb Pu. K-25 personnel felt that this was a greater than normal amount of entrained powder and attributed this to the sintering of the sodium impurity during fluorination. The UF_6 exit line contains a cooler and a filter. Some solids were recovered from the filter but no analyses were available at the time of the meetings. The K-25 personnel admitted that entrainment of fluffy solids through the UF_6 exit line could be reduced by design modifications to direct these solids to the ash pit at the base of the fluorination tower.

DEVELOPMENT PROGRAM FOR HW AND K-25

Following a review by Hanford personnel of the Redox flowsheet together with a discussion of the difficulties involved in modifying the flowsheet to reduce impurity content, it was agreed that both sites should

study process modifications to improve the conversion of Hanford product to UF₆. The agreed program is summarized briefly below:

1. K-1131 Feed Plant Process Modifications. It was agreed that all Hanford oxide (including lot 001 and 002) produced up to the point of where lag storage for U-237 decay becomes necessary would be accumulated at K-25 to provide one feed batch for the K-1131 plant. This material will probably total at least 80-100,000 pounds. Runs will then be made through the feed plant with varying temperature, retention time, and vibration frequency during the hydrofluorination step. K-25 personnel felt that some improvement could certainly be realized but that duplication of MCW behavior would not be realized so long as the Hanford material contained the 0.5% of metallic impurities.
2. Calcination of Mallinkrodt UNH in Hanford Oxide Plant. In order to establish that the below-normal behavior of the Hanford oxide is due to metallic impurities rather than process or operational conditions, it was agreed that a 5000 pound test with pure MCW UNH material in the Hanford pots would be desirable. Mallinkrodt indicated a willingness to provide thirty (30) drums of UNH solution and K-25 personnel agreed to test such a 5000 pound lot in their pilot plant which will be completed May 1. The AEC is currently taking steps to secure approval for such a shipment. It was agreed that the period during the lag for 237 decay would be an appropriate time to conduct such a test at Hanford.
3. Effect of Heat Cycle on Hanford UO₃ Reactivity. Mallinkrodt ventured the belief that prolonged heat cycles during calcination might affect oxide reactivity adversely. Recent studies of ORNL Purex product at MCW where calcination periods had to be extended, gave evidence of low reactivity when tested at K-25. In order to obtain a preliminary estimation of this effect, three samples were taken from a Hanford oxide pot at half-hour intervals following the completion of the mastic stage. These were forwarded to K-25 for examination of flow characteristics and a laboratory evaluation of relative reactivity.
4. Effect of Particle Size and Surface Area on Hanford UO₃ Reactivity.

It was the considered opinion of the group that any further reduction of the particle size would probably not affect oxide reactivity appreciably. Hanford will examine possibilities for achieving some reduction easily and perhaps prepare an experimental lot for pilot plant testing at K-25.
5. Reduction in Impurities in the Redox Process. Various possibilities for impurity reduction were discussed. It was pointed out that flow-sheet modifications to improve purity were under laboratory study by Separations Technology but that it might be some time before application to plant operation could be realized. It was emphasized that

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the operation of the third uranium extraction cycle for purification purposes only would be a costly operation and that other alternatives should be emphasized. Hanford nevertheless agreed to pursue development and production testing directed toward improving purity.

6. Specifications. Discussions were confined mainly to "suitability of product" rather than to detailed numerical specifications. Laboratory personnel at K-25 are developing a laboratory static test for the evaluation of relative reactivity of UO_3 powder. Such a test, if successful, might be a key specification determination and lead to the elimination of the many physical and spectrographic determinations now comprising the tentative specification. Such a test will be set up at Hanford shortly by Separations Technology.

It was agreed that the next meeting would probably be held at this site following the next plant-scale run in the K-1131 plant. It is expected that this run will be conducted during April.

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