YELLOW COPY PROVIDED [redacted]

GEH-17,672

C-206-05

FILE'S CIRCULATING COPY
RETURN TO:
TECHNICAL INFORMATION FILES

Route List

1. R. H. Beaton
2. ~~[illegible]~~
3. F. Chapman
4. [illegible]
5. R.E. Atkinson 1853 326

FILE _____

DATE Undated

SUBJECT Specifications for Recovered UO_3

To File

FROM No Author Given

COPY NO. [redacted]

BEFORE READING THIS DOCUMENT, SIGN AND DATE BELOW:

ARB	12/1/50
HMP	12/1/50
CAR	2/15/51
DDR	8/14/53
FC	8/13/53
RET	4-10-56

[redacted] Act of 1946.

[redacted]

I

GEH 17672

[redacted]

SPECIFICATIONS FOR RECOVERED UO_3

34796-1

This document was transmitted by: GEH-17,671, copies 1,2, & 3-4, dated 11-30-50.

I. Radioactivity Contamination

In order to allow some process flexibility and to limit the amount of material requiring reprocessing, a maximum as well as an average specification for radioactivity contamination is stated. It is desirable that the average fission product activity of the recovered UO_3 be maintained as low as is practicable.

A. Beta Activity

1. Specifications. The maximum net beta activity due to fission products in the recovered UO_3 shall not exceed 20% of the beta activity of normal uranium* in equilibrium with its short-lived daughter products. The average net fission-product beta activity of the recovered UO_3 shipped during any one-month period shall not exceed 10% of the beta activity of normal uranium in equilibrium with its short-lived daughter products.

2. Measured Method. Samples containing equal quantities (approximately 50 mg.) of recovered UO_3 and normal UO_3 will be prepared for counting under identical conditions. The beta activity of these samples will be determined by counting with the same Geiger-Muller tube, alternating the recovered UO_3 and the normal uranium samples. The recovered UO_3 activity is corrected to zero time for UX_1 buildup (6) during the period from processing to counting.

3. Counting Apparatus. The counter consists of a Nuclear Instrument and Chemical Corporation scaling unit (Model 164) and a Radiation Counter Laboratories vertical lead shield or chamber. The Geiger-Muller tube rests upon a lucite rack, and the sample is placed on a shelf 14 mm. from the Geiger-Muller tube window.

The Geiger-Muller tube is the standard end-window tube filled to 10 cm. pressure with 1 cm. pressure of ethyl alcohol and 9 cm. of argon. The window is of Aquadag-coated mica with a density less than 3 mg./cm.^{2}.

B. Gamma Activity

1. Specifications. The maximum net gamma activity due to fission products in the recovered UO_3 shall not exceed 200% of the gamma activity of normal uranium in equilibrium with its short-lived daughter products. The average net fission-product gamma activity of the recovered uranium oxide shipped during any one-month period shall not exceed 100% of the gamma activity of normal uranium in equilibrium with its short-lived daughter products.

*Aged at least 6 months after processing.

[redacted]

2. Measurement Method. One hundred gram samples containing equal quantities of recovered UO_3 and normal UO_3 are measured alternately under identical conditions. The recovered UO_3 activity is corrected to zero time for UX_1 (6) buildup during period from processing to counting.

3. Measurement Apparatus. A high pressure chamber (7), vibrating reed electrometer, and recorder will be used.

C. Plutonium

1. Specifications. The concentration of plutonium shall not exceed ten parts plutonium per billion parts uranium.

2. Measurement Method. The plutonium is separated by lanthanum fluoride precipitations and determined by alpha counting.

3. Measurement Procedure.

a. Dissolve 0.5 to 1.0 grams UO_3 in 8N HNO_3 and dilute to 10 ml.

b. Pipette 1 ml. of sample into a 15 ml. centrifuge tube and make it acidic with 2N hydrochloric acid.

c. Add 2 ml. of a solution of 4N hydroxylamine hydrochloride and let the solution stand in an 80° C. water bath for twenty minutes.

d. Add 0.25 ml. of lanthanum nitrate solution (31.17 gms. $La(NO_3)_3 \cdot 6H_2O$ per liter), agitate, and then add 0.5 ml. of hydrofluoric acid.

e. Centrifuge and discard the supernate. Wash the residue with 1 ml. of an (1N HCl - 1N HF) acid wash solution.

f. Wash the precipitate with one ml. of water.

g. Dissolve the precipitate with 1 ml. of zirconyl chloride solution (35.32 gms. $ZrOCl_2 \cdot 8H_2O$ per liter) and dilute to 5 ml.

h. Add 4 drops of concentrated hydrofluoric acid and agitate, then add 4 more drops of concentrated hydrofluoric acid and centrifuge. Discard supernate.

i. Wash the precipitate with 1 ml. of acid wash solution (1N HNO_3 - 1N HF). Centrifuge and then wash with 1 ml. of water.

j. Make a slurry with water and mount on a stainless steel disc and cover with 6 drops of a 40:1 mixture of acetone and Wrap-Rax.

k. Make alpha count of sample on proportional counter with 50% geometry.

[redacted]

CONF

II. Chemical Purity of Uranium Oxide

A. Net purity desired - approximately 98.5 weight percent UO_3.

1. Impurities to be primarily H_2O and NO_3^-.

2. Concentration limits on tetravalent uranium are not yet available.

B. Specific impurity elements tolerances.

1. Analyses similar to those obtained for Redox material (1) should be satisfactory; however, a reduction in the sodium content (2500 p.p.m.) of the material would lessen the problem of filtering fine dust particles from the UF_6 gas produced.

2. Analyses similar to those obtained from TBP material (2) should be satisfactory; however, the high percentage of PO_4 (0.002 g./gU) probably would increase corrosion in some of the feed plant equipment (e.g., HF still, condenser, and pumps).

3. UO_3 received from the Mallinckrodt Chemical Works has proven satisfactory in feed plant pilot plant work. Specifications on this material are as follows:

Min. UO_3	-	97%
Max. Fe	-	.003%
Max. Cr	-	.01%
Max. Ni	-	.004%
NHO_3 insoluble	-	.01%

However, the material we have been working with analyses at least 98.5% UO_3 and this purity should be maintained if possible.

C. Material should be sufficiently low in water content to be free-flowing.

D. Adsorbed gas (such as NO_2) tolerance limit is not known.

III. Physical Properties of Uranium Oxide

A. Particle size distribution:

99.5 percent to pass 100-mesh screen.
99.8 percent to pass 60-mesh screen.

B. Bulk density:

The loosely packed product should have a density of 3.2 - 3.9 grams per cc.

C. Surface area per unit weight (by nitrogen adsorption) should be approximately ~~16~~ 1.5 or better square meters/gram. - method to be specified

D. Reactivity to hydrogen and/or fluorine - no specifications available. X

E. Solubility and dissolution rates in water and nitric acid - no specifications available. X

REFERENCES

1. Jacobson, H. K. and Sadowski, G. S., Summary Report for the ORNL Pilot Plant Investigation of the Redox Process, Oak Ridge National Laboratory, November 1, 1949, (ORNL-463)Page 149-150.

2. Nurmi, E. O., UNH from TBP Process, Memorandum to Mr. F. L. Steahly, Oak Ridge National Laboratory, June 26, 1950, (ORNL-50-6-172).

3. K-25 Plant Quarterly Report for Second Fiscal Quarter October 1 - December 31, 1949, Carbide and Carbon Chemicals Division, K-25 Plant, February 15, 1950, (K-560, Part I).

4. K-25 Plant Quarterly Report for Third Fiscal Quarter January 1 - March 31, 1950, Carbide and Carbon Chemicals Division, K-25 Plant, May 5, 1950, K-600, Part I).

5. K-25 Plant Quarterly Report for Fourth Fiscal Quarter April 1 - June 30, 1950, Carbide and Carbon Chemicals Division, K-25 Plant, August 15, 1950, (K-636, Part I).

6. Knight, G. B. and Macklin, R. L., "Half-Life of UX_1 (Th_{234})," Phy. Rev., 74, 1540-1541, 11-15 (1948).

7. Borkowski, E. J., "Instruments for Measuring Radioactivity," Anal. Chem. 21, 350-352 (March, 1949).