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REACTIVITY OF UO_3 PRODUCED AT HAP0

INTRODUCTION

The reactivity of UO_3 is expressed by the ratio of the percent UF_4 produced in the test sample under a given set of conditions, to the percent UF_4 produced in a standard UO_3 sample under the same conditions, and thus represents the relative rate of reduction and hydrofluorination of a given UO_3 sample in comparison with a UO_3 standard. This reactivity ratio is a measure of one of the characteristics which determine the rate of processing UO_3 in the Oak Ridge K-25 operation.

The routine measurement of the reactivity of UO_3 produced at HAP0 was undertaken by the Process Chemistry group in June, 1952, while K-25 was experiencing difficulty in obtaining satisfactory feed rates with Hanford material. The reactivity determination, patterned after that used at K-25, was therefore adopted both as a measure of plant UO_3 quality, and as a laboratory tool to assist in determining the factors affecting UO_3 reactivity and in a search for possible methods for its control and improvement.

During the course of the development and use of this determination in the Process Chemistry laboratory, several different samples of UO_3 were used as reactivity standards. However, the present report expresses all the available data on plant UO_3 in terms of a single reactivity standard to facilitate less ambiguous, longer range comparisons. The results are reported here for lots and carloads up to and including 072 at which point the Analytical Services group, Manufacturing Department, accepted responsibility for the routine determination of the reactivity

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of plant UO_3 lots. The results of the research and development work, which was carried on in connection with this study, will be summarized in a separate report.

METHOD OF DETERMINATION

The determination of the reactivity or conversion ratio of a given sample of UO_3 is carried out by a procedure based on a method previously developed largely by Sidney Katz at K-25 in Oak Ridge. The test samples and the UO_3 standard are placed in separate boats in a combustion tube in an electric furnace. Hydrogen is introduced during a half hour warm-up period and for one hour longer at $600^\circ C$. at approximately 1600 ml. per minute. Then hydrogen fluoride gas at about 500 ml. per minute is passed over the samples for one hour at $600^\circ C$., and the HF flow is continued while the samples are cooled below $100^\circ C$. as rapidly as possible. The samples are then analyzed for total tetravalent uranium and for UO_2 (so-called AOI or ammonium oxalate-insoluble). The percentage of UF_4 in each of the samples is then calculated by the following relationship:

$$\% UF_4 = \frac{\text{Mol. Wt. of } UF_4}{\text{At. Wt. of U}} \times \left[\% U^{+4} - \left(\frac{\text{At. Wt. U}}{\text{Mol. Wt. } UO_2} \times \% AOI \right) \right]$$

The reactivity of the given sample is then expressed by the ratio of the UF_4 percents determined in the same run, thus:

$$\text{Reactivity or Conversion ratio} = \frac{\% UF_4 \text{ in unknown}}{\% UF_4 \text{ in standard}}$$

RESULTS

The reactivity data presented in this report are all expressed in terms of the Mallinckrodt T-268 standard. This material was prepared at K-25 by blending a quantity of "good" Mallinckrodt UO_3 and shipping a portion of the blended material to Hanford for use as a standard. This material is considered by K-25 personnel to react at an acceptable rate in the feed preparation plant; therefore any UO_3 with a conversion ratio of 1.0 or better would show a satisfactory processing rate in the K-25 feed plant. The same standard will be used by the Analytical Services group in their reactivity determinations.

In the data as reported, the third digit of each ratio is probably not significant. They have been included here merely to give an indication of the significance of the second digit, and to illustrate that extremely good reproducibility has been achieved in some cases. Where more than one reactivity ratio is quoted for a given UO_3 lot, the different ratios represent separate determinations generally made on different dates.

Table I summarizes the reactivity data for all the determinations made on individual plant lot samples. These lot samples were composites of 8 drum samples prepared by the Analytical Services group and were the same samples used for the specification analyses of the production lots. Table II summarizes all the available reactivity data on carload lots. These determinations were likewise made on composites prepared by the Analytical Services group from the lot composites of the 12 lots making up

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each shipment. It is possible that the difficulty experienced in getting reproducible results with some samples may have been due to the inability to carry such an extensive compositing operation to the point of complete homogeneity with a material as difficult to mix as UO_3 . Also the reactivity of UO_3 is very sensitive to the presence of moisture which it readily picks up on exposure to the atmosphere.

The following table compares the reactivity of some earlier carload composite samples with the average of the reactivities of the individual lots making up the carload. Only for these five carloads are the reactivity data available for all the individual lots making up each carload.

<u>Carload Number</u>	<u>Carload Reactivity</u>	<u>Lot Numbers</u>	<u>Average of Lot Reactivities</u>
003	0.92	77 thru 88	0.86
004	0.80	89 thru 100	0.78
005	0.84	101 thru 112	0.86
012	0.89	161-163, 192-200	0.93
023	0.98	318 thru 329	0.98

It is expected that in the future all data on current plant production will be obtained and reported by the Analytical Services group.

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TABLE I

REACTIVITIES OF HANFORD UO₃ LOTS

Lot No.	Conversion Ratio	Lot No.	Conversion Ratio	Lot No.	Conversion Ratio	Lot No.	Conversion Ratio
005T	0.771	93	0.763	159	0.745	328	0.995
6T	0.604	94	0.813	161	0.906	329	0.994
7T	0.830	95	0.889	162	0.855	330	1.01
8T	0.757	96	0.721			331	0.996
9T	1.02	97	0.746	163	0.958	333	0.974
"	1.03			180	0.924		
"	1.03	98	0.784	181	0.924	335	1.00
"	1.06	99	0.797	189	0.950	337	0.970
		"	0.837	190	0.924	339	1.00
11	0.647	100	0.788			341	0.982
"	0.610	101	0.803	191	0.924	343	0.980
12	0.651	102	0.950	192	0.924		
63	0.472			193	0.976	345	0.988
65	0.839	103	0.984	194	0.941	347	1.02
"	0.831	104	1.01	195	0.967	349	1.01
66	0.936	105	1.07			351	1.03
		106	0.794	196	0.967	353	1.02
67	0.780	107	0.717	197	0.932		
68	0.837			198	0.915	373	0.975
70	0.822	108	0.742	199	0.924	375	0.972
"	0.780	109	0.794	200	0.889	377	0.965
75	0.822	110	0.751			379	1.02
77	0.861	111	0.820	204	0.858	419	0.991
		112	0.780	"	0.862		
78	0.864	"	0.839			420	1.00
79	0.889	"	0.825	206	0.950	425	0.998
80	0.981	"	0.841	"	0.958	426	0.997
"	1.01			209	0.958	431	1.01
81	0.864	113	0.820	"	0.977	441	1.04
82	0.920	114	0.768	"	0.993		
"	0.915	115	0.820	"	0.999	451	1.02
		"	0.837	214	0.950	512	0.974
83	0.838	116	0.794	"	0.924	"	0.96
84	0.813	"	0.803	244	1.00	515	0.953
85	0.822	117	0.780	"	0.984	524	0.966
86	0.947					527	0.994
87	0.696	118	0.787	318	0.962		
"	0.721	126	0.661	319	0.974	539	0.976
"	0.729	"	0.681	320	1.01		
"	0.717	"	0.530	321	0.992	611	0.966
"	0.739	"	0.588	322	1.00	614	0.991
"	0.763	"	0.599			632	0.962
		131	0.848	323	0.994	638	0.986
88	0.780	132	0.889	324	0.984		
89	0.771	134	0.783	325	0.987	675	1.00
90	0.746			326	0.941		
91	0.771	139	0.889	327	0.951		
92	0.847	140	0.915				

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TABLE II

REACTIVITIES OF HANFORD CARLOAD LOTS OF UO₃

Carload No.	Conversion Ratio	Carload No.	Conversion Ratio
003	0.922	049	0.980
004	0.803	050	0.992
005	0.837	051	0.968
012	0.872	052	1.04 (1.02)
"	0.892	053	0.969
013	0.974	054	0.976
015	0.943	055	0.988
016	0.976	056	0.972
"	0.993	057	0.995
017	1.01	058	0.965
018	0.986	059	0.974
019	0.970	060	0.969
020	0.972	061	0.969
021	0.978	062	0.996
022	0.970	063	0.985
023	0.975	064	1.00
033	0.977	"	1.01
034	1.00	055	0.963
"	1.01	"	0.962
"	1.02	066	0.975
"	1.02	"	0.971
035	1.01	"	0.970
036	1.00	067	0.991
037	0.969	"	0.967
038	0.959	"	0.952
039	1.00	"	0.977
040	1.01	068	0.970
041	1.01		0.940
042	1.00	069	1.01
043	0.989	"	1.00
044	1.02	070	0.982
045	0.972	071	0.975
046	0.983	072	0.967
047	1.01		
"	0.99		
048	1.00		

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