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Operated by
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Division of Union Carbide Corporation

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Oak Ridge, Tennessee

External Transmittal
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ORNL
CENTRAL FILES NUMBER

59-1-101

DATE: January 22, 1959
SUBJECT: CONTROL OF THORIUM OXIDE PARTICLE SIZE
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COPY NO. 119

MASTER

ABSTRACT

By carefully controlling the agitation, temperature, concentrations, and reagent addition rate it has been possible to produce a thorium oxide product within 0.3μ of any selected size between 2.0 and 3.0μ . The rate of addition of oxalic acid to a thorium nitrate solution was the variable used in the current series of tests.

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Introduction

In order to gain better control over the particle size of the thorium oxide prepared by the Thorium Oxide Facility for the Homogeneous Reactor Project, operating conditions have been standardized for the past several months. This program has resulted in greatly improved consistency of a desired size thorium oxide product.

Equipment

All the runs tabulated herein were made in the regular Thorium Oxide Facility equipment located in Building 3019. The equipment is essentially as described in ORNL-2509.

Run Conditions

Since particle size is known to be dependent on solution concentration, precipitation temperature, reagent mixing rate, and degree of agitation (and possibly others) it was desirable to fix all of the known variables except one. For simplicity of operation the rate of addition of oxalic acid to the thorium nitrate solution was chosen as the variable. The fixed conditions were:

Oxalic acid solution:	257 gallons, 0.7 ± 0.02 M
Thorium nitrate solution:	117 gallons, 0.7 ± 0.02 M
Precipitation temperature:	$30^\circ \pm 1^\circ\text{C}$

Agitation: 431 rpm, 8 in. semiturbine type impeller in draft tube having side slots completely open and bottom area 50% open (see p. 12 ORNL-2509)

Precipitation of the thorium oxalate was made by pumping the oxalic acid solution at a steady rate into an agitated tank containing the thorium nitrate solution. Both solutions were adjusted to 30°C before mixing and the precipitation tank was maintained at $30^\circ \pm 1^\circ\text{C}$ during the acid addition by means of refrigeration. After precipitation the oxalate slurry was digested for either 48 hours at 85°C or 6 hours at 95°C . The slurry was then filtered and the thorium oxalate cake converted to the oxide by calcination at either 650°C or 800°C depending on the end use for the particular run.

Results

The geometric mean particle size and geometric standard deviation for various flow rates are given below. These data are also plotted on the attached graph.

Run No.	Oxalic Acid Flow (gpm)	Mean Size (microns)	Standard Deviation
30	1.6	3.7	1.29
31	3.1	2.5	1.34
32	3.1	2.8	1.36
33	3.6	3.1	1.29
34	3.5	2.6	1.37
35	4.3	2.5	1.27
36	5.5	2.2	1.30
37	4.1	2.7	1.30
38	Special run, oxalic acid added as crystals		
39	4.7	2.8	1.40
40	6.6	2.1	1.39
41	6.5	2.0	1.42
42	6.4	2.0	1.35
43	6.6	2.1	1.40
44	6.0	2.1	1.43
45	6.0	2.2	1.34
46	5.9	2.1	1.28
47	5.9	2.0	1.30
48	5.8	2.0	1.30
49	6.3	1.9	1.37
50	6.0	2.1	1.28
51	6.0	2.1	1.31

Conclusions

The present operating procedure and controls used in the Thorium Oxide Facility can produce 185 pound batches of thorium oxide having a geometric mean particle diameter within ± 0.3 microns of any chosen size between 2 and 3 microns. The geometric standard deviation of the product will be approximately 1.35. It is believed that the present data can be successfully extrapolated to cover the size range of 1.5 to 4.0 microns using only oxalic acid flow rate as the variable.

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