Proposed Changes to Coors Porcelain Co. Recipe for Fueled Reactor Tubes (Memo to Mr. Bob L. Mornin)

T. C. Merkle

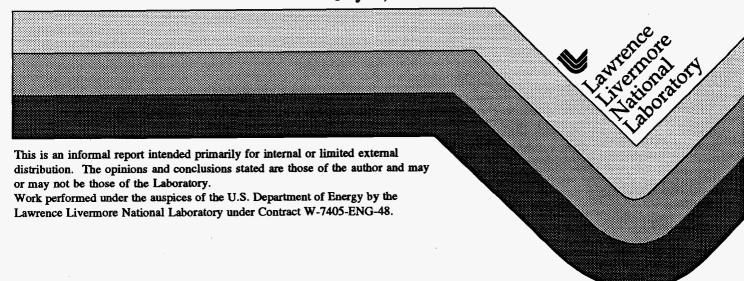
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July 27, 1961

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Mr. Bob L. Mornin Project Manager Coers Porcelain Company Golden, Colorado

July 27, 1961

Dear Bob!

The following information for use with fueled tubes is of a preliminary nature intended for guidance only. More definitive information will follow-as it becomes available.

Because of various evaluation studies on fuel stability, it is now proposed that Coors (Subcontract 165) change their recipe for fueled tubes. Specifically, zirconyl nitrate should be used in addition to uranyl nitrate. The detailed changes to be made in the contract at this time are listed below.

and Changed to:

Section III (COB-985, page 1)

Under 2. Materials

Add:

2.3 Zirconyl nitrate crystals or wet filter cate fill be supplied to the subcontractor by the University in accordance with a University Specification attached hereto.

Under 3.3 Density of Elements

A change in the calculation of the density of fueled tubes will be required. This information will be forwarded as the information becomes available.

Add a Section after Section II.

II.A. Zirconyl Nitrate Tentative Specification

This specification is tentative only. Zirconium nitrate will be procured of the highest purity practicable.

Tentative Specification: Zirconyl Nitrate

- 1. Scope
- 1.1 General: This specification defines the requirements for high purity Zirconyl Nitrate prepared by arbitrary processes.
- 2. Reference Documents

None

- 3. Requirements
- 3.1 Chemical Composition: Material shall be supplied in the form of a moist highly acid filter cake (ca. 35-40% acid) containing both soluble and insoluble zirconium. This cake contains 25 percent by wt. zirconium.

Crystalline material may be used if it becomes available.

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July 27, 1961

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: bbA

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3.2 Purity

3.2.1 Impurity Concentrations - Impurities in the samples of the solution shall not exceed the following limits on a dry basis as ZrO(NO₃)₂.

773	Maximum Concentration
Element	Dry Basis, ppm by wt
Fluorine	20
Magnesium	20
Vanadium	20
Barium	50
Copper	60
Calcium	100
Chromium	100
Nickel	100
Tin	100
Aluminum	200
Phosphorus	200
Potassium	200
Molybdenum	250
Silicon	300
Iron	300
Carbon	700
Beryllium	24.1000 (25.5)
Hafnium	100

3.2.2 C/q Summation - The summation of C/q, where C is the concentration in ppm by wt. on a dry basis (as ZrO(NO₃)₂) and q is the tabulated value, shall not exceed 50 for any sample.

Element	<u>. 9</u>
Gadolinium	1
Boron	4
Hafnium	30
Samarium	5
Cadmium	5 6 8
Europium	. 8
Cobalt	12
Lithium	20
Indium	20
Manganese	25
Gold	30
Rhodium	40
Sodium	50
Titanium	. 50
Silver	50

5.2.3 Other Impurities - No elements other than Uranium, Oxygen, Nitrogen, Zirconium and Hydrogen and those listed in paragraphs 5.2.1 and 3.2.2 shall be individually present in the finished filter cake to an extent greater than 100 ppm by wt. on a dry ZrO(NO₂) basis.

Changes in Process Specifications (VI.), COB-987

The incorporation of ZrO₂ in Tory IIC fuel elements can be covered by the following changes in Addendum No. 1 of Appendix A to Subcontract 165 between the University and Coors. (Document No. COB-987, Revision I):

VI. Process Specification

Introduction (p. 1)

Insert a new b) "adding zirconyl nitrate solution to the slurry".

Change b) to c) and add the phrase "and zirconium".

c) will be d) when relettered should read "filtering drying and calcining the solids."

Change subsequent letters to conform i.e. c) to d) etc.

Raw Material (p. 1)

Insert subheading "BeO powder" followed by existing paragraph.

Insert subheading "Zirconyl Nitrate" and the following paragraphs.

"Reactor grade zirconyl nitrate shall be dissolved in a 0.5 N nitric acid solution to a concentration of about 0.05 g.Zr0, per ml. The solution should be filtered prior to use to remove insoluble materials and analyzed for Zr0, content. Analysis shall consist of drying and igniting an aliquot of the solution at 1500°F for 3 hours. The residue shall be weighed and relegated as grams Zr0, per ml of solution."

"There is some question as to the stability of these zirconyl nitrate solutions. They tend to hydrolyze and precipitate on standing even with the nitric acid present. It is therefore necessary that the solution be made up and the concentration determined just prior to use. A test is underway to determine to what extent the solution is unstable. The results of this test will be forwarded in the near future."

Operations (p. 3)

1. Mixing - First paragraph unchanged. Second paragraph revised as follows:

"The BeO slurry shall be moved to the mixing tank and the proper amounts of Oy Nitrate and Zirconyl Nitrate solutions added to it with thorough mixing. After sufficient mixing to assure a uniform slurry, Ammonium Hydroxide solution will be added to the mixer to precipitate the Oralley and Zirconium. The completeness of precipitation will be checked by a pH meter. The final pH should be about 9."

Third paragraph unchanged.

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2. Filtering - (p. 3) First paragraph. The next to last sentence should read: "The filter cake will be washed with the D.I. water, containing about 0.2 percent NH₁OH, used to clean out the mix tank." The last sentence in the paragraph reads correct.

New 4. Calcining - Should read as follows:

"The dried filter cake shall be calcined at a maximum temperature of 1000° F for approximately 10 hours. This may be accomplished by loading a 1/2 mix batch (ca.900g) in a covered stainless steel can (4° x 8° x 6° high). The firing may be done in a tunnel kiln on a continuous basis, by pushing on the 4 x 6 face of the can. The calcined cake from each batch shall then be combined and placed in polyethylene bags and suitable containers on a safe storage rack until used for the next operation."

5. Pugging - (p. 4) First paragraph. Delete first two sentences. Paragraph should start: "The cake is poured....."

Second paragraph. Second sentence should read: "Upon completion of mixing a sample will be taken for quantitative determination of the Oralloy, ZrO2, water and binder content." The remaining portion of the paragraph reads correct.

Change subsequent numbers under "Operations" to conform, i.e. (5) to (6) etc.

Additional

The ZrO₂ to UO₂ weight ratio in each fueled tube should be 0.692/1.000. Modification to a ZrO₂/UO₂ weight ratio as high as 1.077/1.000 may be made in the near future.

Yours truly,

T. C. Merkle

Associate Director (Pluto)

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