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SNC-3693-5 RADIOISOTOPES AND RADIATION APPLICATIONS TID 4500 (55TH ED.) UC-23

THULIUM OXIDE FUEL CHARACTERIZATION STUDY

PART III - PROCEDURES

Prepared By

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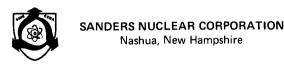
Prepared For

DIVISION OF ISOTOPES DEVELOPMENT U.S. ATOMIC ENERGY COMMISSION

CONTRACT NUMBER: AT-(40-1)-3693

JUNE 1970

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SECTION 1

THULIUM SESQUIOXIDE - YTTERBIUM SESQUIOXIDE
PSEUDO - BINARY PHASE DIAGRAM TEST PROCEDURES

SANDERS NUCLEAR CORPORATION

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1.0 Introduction

The following are the test procedures for the determination of the thulium sesquioxide - ytterbium sesquioxide quasi-binary phase diagram. The diagram will include compositions from 100% ${\rm Tm_2O_3}$ to 100% ${\rm Yb_2O_3}$ and temperatures through the melting points. The melting point of ${\rm Tm_2O_3}$ has been reported to be about 2400°C; however, the melting point of ${\rm Yb_2O_3}$ is not known. From theoretical considerations it is expected that the diagram will show complete miscibility. One solid state phase transformation has been reported for ${\rm Tm_2O_3}$ at 2280°C. ¹ Such information is not available for ${\rm Yb_2O_3}$.

The phase diagram will be determined primarily by thermal analysis. If required, the thermal analysis will be supplemented by other tests such as metallography and X-ray diffraction analysis. The work will be performed as part of Phase II of Contract Number AT-(40-1)-3693.

A tungsten crucible shall be arranged within an induction coil to provide temperatures beyond the melting point of the oxide sample composition. The induction power supply shall be programed to give a constant thermal increase and then decrease to permit study of thermal arrests of

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^{1.} M. Foex & J. P. Traverse. "High-Temperature Study of the Allotropic Transformations of the Sesquioxides of Yttrium, Erbium, and Thulium." Compt Rend 261, 2490-3 (1965).



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the samples. The temperatures shall be recorded by use of an automatic optical pyrometer focused on a black body target.

2.0 Objectives

The objective of this study will be to determine the quasi-binary phase diagram of the ${\rm Tm}_2{\rm O}_3$ -Yb2O3 system. The composition limits of the diagram will be 100% ${\rm Tm}_2{\rm O}_3$ and 100% Yb2O3 and the temperatures will be through the melting points. Data will be obtained for the solidus and liquidus temperatures, allotropic phase transformations and possible intermediate compound formation. The condition of reversibility shall be noted and previous work reported by others shall be reviewed for correlation.

It will be necessary to obtain data about crucible materials for ${\rm Tm_2O_3\text{--}Yb_2O_3} \ {\rm at\ temperatures\ through\ the\ melting\ point.}$

For this study only stable isotopes of Tm and Yb shall be used.

3.0 Test Requirements

3.1 Applicable Documents

As per contract AT-(40-1)-3693.

3.2 Raw Materials

All materials used shall be from single production batches. The material will be controlled as follows:

3.2.1 Impurities not to exceed 0.1% including all rare earths and other impurities.

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3.2.2 High purity shall be specified; certified copies of the vendors' reports will be reviewed for compliance and filed. The analysis will be for such elements at Ytterbium, Thulium, Lutetium, Erbium, Silicon, Iron, Aluminum and Calcium.

3.3 Sample Preparation

Various processes are being evaluated in Phase I of Contract AT(40-1)-3693 for fabrication of $Tm_2O_3-Yb_2O_3$ thermal analysis samples. The process that yields the best product shall be selected as the method for fabrication of the samples. A slug about 3/8 inch diameter by 1/2 inch length shall be pressed and sintered at $1455^{\circ}C$ for densification and homogenization. Selected samples will be checked by X-ray diffraction to determine the extent of the reaction of $Tm_2O_3+Yb_2O_3 \longrightarrow (Tm, Yb)_2O_3$. If it is found the reaction has gone to completion (to the right), the samples could then be used directly for thermal analysis testing. However, if any Tm_2O_3 and Yb_2O_3 remain, additional homogenization treatment (either sintering or melting) may be required.

- (a) Samples fabricated by the process shall be analyzed by such means as emission spectrography and chemical analysis to assure maintenance of purity during fabrication and to check the Tm and Yb content.
- (b) The sample density and physical data including size and

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surface condition shall be recorded.

- (c) The process techniques and equipment used with operational variations shall be recorded.
- (d) All other materials within the powder mixture such as binders or lubricants shall be recorded.

3.4 Compositions Scheduled

The initial determination of the phase diagram shall include the following compositions:

After the thermal analysis and substantiating test data has been reviewed from these compositions, other compositions may be tested if better definition of specific areas of the diagram is required.

3.5 Sample Preparation for Furnace

Suitable samples prepared as above will have a hole drilled in the end surface to simulate a black body target for optical pyrometry during thermal analysis. The material removed by drilling shall be

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submitted for analysis by such means as emission spectrography and chemical analysis for Tm/Yb content and impurity check.

3.6 Low Temperature Analysis

The portion of the diagram from room temperature to about 1200°C that will overlap the portion of the temperature range being monitored by optical pyrometry shall be determined by Differential Thermal Analysis using Fisher Co. Model 260 Differential Thermalyzer with the Model 360 Programer. The procedures used will be as recommended by the manufacturer. The samples will be heated and cooled at programed rates to detect possible allotropic transformation by use of thermocouples and strip chart recorders.

Prior testing using known samples will be run to assure calibration of the unit.

3.7 Thermal Analysis

3.7.1 Programed Thermal Change

The portion of the diagram above 750°C to the melting point will be determined by controlled heating and then cooling in a cold wall induction furnace. This Thermal Analysis Test will be performed by slowly increasing the furnace power at a steady rate as determined electrically with a tap coil on the inductive loop. Simultaneously but independently, the sample temperature shall be measured with an automatic optical pyrometer. Inflec-

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tions or arrests observed on the heating curve would be indications of allotropic or phase transformations. The furnace power supply shall be programed by use of a ramp generator with a power control to signal a saturable core reactor of the induction power supply. The thermal input will be free of detectable superimposed fluctuations.

Prior to actual thermal analysis the furnace power control device will be checked with dummy samples to demonstrate the smoothness of control. Both the heating and the cooling rates will be determined empirically with these trial samples.

3.7.2 Planned Rate Change

The optimum rate at high temperature would be one giving greatest sensitivity with a minimum amount of reaction with the crucible and decomposition. At lower temperatures, slower rates would be used to allow for slower rates of transformation.

Selected samples will be run at various rates as a check on this procedure.

3.7.3 Furnace Construction

The construction of the crucible and other interior parts of the high temperature furnace shall be of materials that will have low susceptibility to surface contamination and minimal

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gaseous absorptive capabilities. A preliminary furnace cycle, using a dummy sample, will be programed for vacuum atmosphere with temperatures beyond the anticipated melting points of the sample. This will assure that the parts of the furnace operating at high temperature shall be thoroughly out-gased prior to setting up the test sample in the crucible.

The sample shall be placed in a crucible within the inductive field of a water cooled susceptor coil. Initially, tungsten will be used as a crucible material. Limited data indicates that this material is suitable at temperatures near the melting point of ${\rm Tm}_2{\rm O}_3$; however, no data is available on compatibility at the melting point. The behavior of tungsten will be closely observed and if found unsatisfactory, substitute

3.7.5 Furnace Atmosphere

materials, such as oxides, will be sought.

3.7.4 Crucible Selection

The crucible and susceptor coil will be placed inside the pyrex glass envelope. A dynamic flow of argon at about one atmosphere will be maintained within the glass envelope to retard vaporization and to flush impurities. Use of argon will serve several purposes; (1) to retard selective evaporation of the sample, (2) minimize the vapor deposition on sight ports of

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the optical pyrometer, (3) protect reactive metal components (crucible) and (4) to allow determination of the phase diagram at a selected one atmosphere pressure. The argon gas used will be welding quality (99.99% pure).

3.7.6 Temperature Readout

The inductive field coils of the power supply shall be placed around the glass envelope. The optical pyrometer will be placed outside the glass envelope and will observe the sample through a suitable sight port. The temperature shall be read by focusing on the black body cavity in the sample that has a length to diameter ratio of at least fine.

The recording optical pyrometer has calibrations traceable to the National Eureau of Standards that shall be maintained. But additional calibration of the unit will be made as it is assemble with the high temperature induction furnace by measurement of melting points of pure metals such as copper and nickel. This will permit a correction for error due to transmission losses at the sight port. Such calibration checks will be repeated as necessary to assure temperatures with an accuracy of +1%.

The readout of the pyrometer shall be presented on a strip chart recorder that will be adjusted in the various temperature

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ranges for an optimum presentation of thermal change per unit of time.

3.7.7 Power Supply Control

The ramp generator and power control system of the power supply shall be monitored with a reference coil on the induction power supply output coil to assure steadily increasing and decreasing slopes.

3.8 Test Failure Criteria

A particular test will be deemed void if the composition of the sample was altered significantly by either selective volitilization or by contamination. The failure criteria will be based on the appearance of the sample, the relation of the data to companion samples, duplicate tests and chemical analyses of selected samples. The validity of a particular test will be determined by the cognizant engineer.

3.9 Test Data Review

The thermal analysis tests will be guided by the ASTM Recommended Practice for Thermal Analysis for Metals and Alloys (E14-63). For each test, the following information will be obtained and recorded.

- 1) Composition of the sample
- 2) Complete thermal and processing history

3) Weight o	of the	e sa r	mple		
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- 4) Atmosphere
- 5) Crucible material
- 6) Rate and direction of temperature change
- 7) Method of analysis (i.e., direct thermal analysis, DTA, etc.)
- 8) Maximum temperature obtained and time at maximum temperatures
- 9) Temperature of sample at identified inflection points (start and finish of transformation)
- 10) Direction of change at inflection point
- 11) Rate of temperature change of furnace at time of inflection
- 12) Maximum and minimum temperature of decalescence and recalescence if either is observed.
- 13) Any departure from expected tehavior

3.10 Reports

The test reports will be summaries presented in the monthly letter reports as theeffort progresses. At termination of the programed tests all data will be reviewed and correlations made to assure a useful presentation. The final phase diagram will be presented with substantiating data one month after completion of the final laboratory effort.

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4.0 Procedure

- 4.1 Receipt of Materials
 - 4.1.1 Each material received shall be from a single production batch maintained by the vendor. both the Tm₂O₃ and Yb₂O₃ shall be analyzed for impurities which shall not exceed 0.1%. A certified quantitative analysis shall be provided by the vendor at time of delivery to include the amount in ppm of such elements as Ytterbium, Thulium, Lutetium, Erbium, Silicon, Iron, Aluminum and Calcium.
 - 4.1.2 The argon gas for the furnace shall be welding grade with certification of 99.99% pure at time of delivery.
 - 4.1.3 All other materials shall be traceable to batches or manufacturers lots and information recorded.
- 4.2 Sample Preparation
 - 4.2.1 Process selection shall be based on the maintenance of the purity of the product.
 - 4.2.2 The compositions scheduled are as follows:

4.2.2.1	100% 1	.m203	-	0%	4p503
	90%	11	-	10%	ff
	70%	11		30%	11
	50%	11	-	50%	rt.
	30%	11	-	70%	11

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10% Tm₂0₃ - 90% Yb₂0₃
0% " - 100% "

4.2.3 Further compositions may be scheduled as required to complete the phase diagram.

4.2.4 Compaction data for the ${\rm Tm}_2{\rm O}_3$ - ${\rm Yb}_2{\rm O}_3$ samples shall include binder and lubricant used, the charge per cycle, compacting pressure and ejection condition. The size of the sintered sample shall be (3/8 inch diameter by 1/2 inch length) recorded to the nearest 0.001 inch. The weight shall be reported to the nearest 0.01 gram.

4.2.5 The sintering cycle of time and temperature together with atmosphere used shall be recorded. The density, dimensions and weight shall be recorded together with the visual appearance of the surface. A black body hole with an L/D of at least five shall be drilled in the end for optical pyrometer readings. The chips from the hole shall be saved for submittal to chemical or emission spectrographic analysis as necessary to assure maintenance of purity of the process. Chips from selected samples shall be submitted for X-ray Diffraction and/or metallographic analysis to assure complete homogenity of the (Tm-Yb)₂O₃.

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4.3 Furnace Operation

4.3.1 The furnace control system shall be checked for smooth changes in temperature during increasing and decreasing power cycles. The crucible will be run empty with the ramp generator set for 50°C in excess of the programed sample temperature.

Cooling rate shall be checked by use of the ramp generator control. The optical pyrometer readout presented on the strip chart shall be studied for acceptable heating and cooling rates.

4.3.2 Furnace outgassing shall be performed when new parts are added or when the system has been reworked. While maintaining a vacuum of 10°4 torr the temperature shall be raised to 50°C above the melting point of the programed sample melting point. This condition shall be maintained for 10 minutes at the temperature after the vacuum has stabilized and then the furnace can be cooled.

- 4.3.3 Normal furnace operation shall be with flowing argon past first the optical sight port and then the crucible while maintaining a pressure of approximately one atmosphere.
- 4.3.4 The optical pyrometer shall be maintained on a yearly cycle for calibration traceable to the National Bureau of

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Standards. Periodically, the pyrometer will be calibrated to the furnace system by checking the melting points of pure metals.

4.4 Test Operation

4.4.1 The low temperature analysis shall be checked using the Fisher Differential Thermalyzer Model 260 with the Model 360 Programer to 1200°C. Readout shall be by thermocouple with a strip chart recorder. Various rates of temperature change shall be programed.

4.4.2 The high temperature thermal analysis shall be programed from 750°C to beyond the liquidus point. The ramp generator shall be set at the selected temperature change rates.

4.4.3 The temperature readout shall be suitable for interpretation of the following items:

4.4.3.1 Inflections or arrests of the temperature may indicate allotropic phase transformation or intermediate compound formation. As the temperature continues through the melting point, the solidus and liquidus temperatures shall be detected by thermal arrests in the heating curve.

4.4.3.2 Reversibility shall be demonstrated during cooling by ramp generator control.

4.4.4 Samples shall be reviewed for stability of the composition

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by correlation of weights before and after testing. Periodically an analysis shall be made to assure purity and composition.

4.5 Test Data Compilation

- 4.5.1 During testing the data will be reviewed and plotted to determine requirements for additional compositions to substantiate the phase diagram.
- 4.5.2 Data will be used to plot the phase diagram. All supportive data will be compiled as part of the written report describing the method and variations noted.

4.6 Cautions

- 4.6.1 Anticipate a melt point of Tm_2O_3 at 2400°C.
- 4.6.2 Melt point of Yb_2O_3 is unknown but assumed to be slightly lower than Tm_2O_3 .
- 4.6.3 Check for solid state phase transformation of pure Tm_2O_3 at $2280^{\circ}C$.

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SECTION 2 COMPATIBILITY TESTS - TEST PROCEDURES

SANDERS NUCLEAR CORPORATION

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1.0 Introduction

The following test procedures have been prepared to assess the compatibility of thulium oxide (Tm_2O_3) containing ytterbium oxide (Tm_2O_3) with candidate container materials. Wafers of Tm_2O_3 containing various amounts of Tm_2O_3 are to be placed in contact with the candidate materials for times and temperatures ranging to 4000 hours and Tm_2O_3 . The compatibilities of the tested materials are to be determined by metallographic examination and such other diagnostic techniques as are required to determine the depths of penetration by diffusion and other interaction mechanisms. The compatibility studies will be performed as part of Phase II of contract number Tm_2O_3 .

2.0 Objectives

The compatibility of Tm_2O_3 Yb_2O_3 with candidate container materials will be assessed by measurement of solid state reaction rates as a function of temperature. Supplemental information pertaining to the mode of penetration (i.e., bulk, intergranular or attack of selected phases) and the degree of bonding will also be obtained. The oxide wafers are to contain four levels of Yb_2O_3 concentration, one of which would be zero. The other three levels of Yb_2O_3 have not been specified and are referred to as A, B, and C. The candidate container materials will be: Hastelloy X, Haynes 25, T-222, TZM (Mo.5Ti) and tungsten.

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3.0 Test Requirements

3.1 Applicable Documents

Contract Number AT-(40-1)-3693.

3.2 Test Conditions

Compatibility tests will be performed for the oxide-metal samples as listed below. After examination of these tests, additional tests may be run to better define the reaction rates. All tests will be performed in evacuated containers as described in Section 4.1.

Oxide Samples	Metallic Samples*	Test Temperature	Exposure Time
Tm ₂ 0 ₃	1,2,6 & 7	<u>600</u> °c	500,2500,4000 hrs.
Tm ₂ 0 ₃ -A%Yb ₂ 0 ₃	11	11	11 11 11
-B%Yb ₂ 03	tt.	11	11 11 11
-c%Yb ₂ 0 ₃	11	. "	11 11 11
·Tm203	1,2,3,6,7	1000°c	500,2500,4000 hrs.
Tm203-A%Yb203	11	tt	11 11 11
-B %Y b ₂ 0 ₃	11	11	11 11
-c%yb ₂ 03	11	11	11 11
Tm203	3,4,5,6,7	<u>1600</u> °c	500,2500,4000 hrs.
Tm203-B%Yb203	ti .	11	11 11 11
Tm203	4,5,6,7	5000 ₀ C	500 hours
Tm ₂ 0 ₃ -B%Yb ₂ 0 ₃	11	n	"
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*Metallic Samples

- Hastelloy X
- 2. Haynes 25
- T-222
- TZM
- 5. Tungsten
 6. Blank-oxide (produced by SRL process) to be tested by itself for comparison.
- Sanders Nuclear Proprietary Process Tm203

3.3 Test Measurements

The tested oxide-metal samples will be examined as follows:

- visual examination check for gross reaction, possible contamination and degree of bonding.
- metallographic examination of a cross section of each sample observe nature of reaction and measure depth (width) of reaction zone. The metallic samples will be examined pefore and after etching.
- Concurrent with the metallographic examination a portion of the samples will be examined by X-ray flourescence, by chemical analysis or by electron microprobe analysis.

3.4 Test Failure Criteria

A given compatibility test will be deemed void if the oxide-metal interface becomes contaminated during the test. The validity of all tests will be determined by the cognizant engineer.

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3.5 Reports

Monthly status letter reports and a final report will be prepared as stated in the contract.

4.0 Procedure

4.1 Sample Preparation

wafers of the Tm₂O₃-Yb₂O₃ will be prepared by compacting and sintering the required mixtures of the oxides. The oxides will be prepared by the method developed at the Savannah River Laboratory, excepting those prepared by the Sanders proprietary process. The sintered wafers will be about 0.60 inches in diameter and about 0.06 inches thick. The faces of the wafers will be ground nominally flat and parallel to allow maximum surface contact with the inner surface of the metallic capsule. The metallic capsules will be cut from commercially available stock, bearing vendor certified analyses, with interior dimensions similar to those of the oxide wafer. Prior to testing, the metal surfaces will be machined to remove possible surface contaminants (i.e., scale), washed with solvents and thoroughly rinsed, the final rinsing being with alcohol.

The sample test procedure will use two methods, one for the 600 and 1000°C tests and the other for the 1600 and 2000°C tests. For the 600 and 1000°C tests, oxide wafers will be encapsulated (by EB welding)

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in appropriate metallic capsules. The individual metal capsules will be wrapped loosely with tantalum foil or other sacrificial metals. The wrapped capsules will then be sealed in quartz ampules containing a partial pressure of pure argon. The sacrificial foil will serve as a barrier between the quartz and the capsules, as well as serving as a gas getter. The group of encapsulated capsules will then be heated in a resistance furnace for the required times at 600 and 1000°C. The furnaces to be used will be Blue M "Stabil Glow" Box Furnace or equivalent. The temperature will be maintained within 12% by means of an automatic controller.

For the 2000°C tests, the appropriate oxide discs will be encapsulated as were the 600 and 1000°C samples. However, instead of encapsulating the samples in quartz ampules, the samples will be held in a dynamic vacuum of 10⁻⁵ torr or less while being heated in a brew Model 466 vacuum furnace. The heating element will be a brew Weave tungsten heater with tungsten radiation shields. The temperatures will be measured by a micro-optical pyrometer, or equivalent.

For the 1600° C tests, the samples will be prepared as for the 2000° C tests. The high temperature vacuum furnace (Brew Model 466) will be used.

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4.2 Sample Examination

The exposed capsules will be carefully sectioned as as to preserve any bonding between the oxide and metal. Half of the sectioned capsule will then be mounted in a transparent mounting materials such as epoxy resin. Mounting materials requiring the use of pressure (i.e., lucite) will be avoided to prevent breaking of interfacial bonds.

The method of metallographic sample preparation of the oxide will be based on those developed for Tm₂O₃, Thulium Oxide Compatibility Study, SNC 3, 4 December 1967. Depending on the effects of Yb₂O₃ and possible reactions, the procedure will be modified as deemed necessary by the cognizant engineer. (The procedure for polishing Tm₂O₃ includes wet grinding on successively finer SiC to 600 grit, rough polishing with 6 micron diamond and vibratory polishing with submicron alumina. Between the various grinding and polishing operations, the specimen is ultrasonically cleaned in first a detergent solution and then tap water to remove polishing debris).

The polished samples will be examined microscopically by the cognizant engineer to determine the extent of reaction between the metal and oxide. The results will be compared with the respective blank oxide samples. Representative micrographs at suitable magnifi-

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cations will be prepared as required. The micrographs will be taken at calibrated magnification to allow accurate measurement of reaction zone.

The remaining portion of each capsule will be examined by the diagnostic techniques (such as X-ray flourescence, chemical analysis or electron microprobe analysis) as are required to determine the depths of penetration by diffusion and other interaction mechanisms.

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SECTION 3

THULIUM 170 OXIDE DOSE RATE MEASUREMENTS - TEST PROCEDURES

SANDERS NUCLEAR CORPORATION

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1.0 Introduction

The radiation dose rate measurements described in this procedure will be made on thulium-170 to determine the shielded dose rates from thulium oxide and to reliably evaluate calculated effectiveness of candidate shield materials. Wafers of thulium-170 oxide $(Tm_2^{170}O_3)$ will be placed behind various shield materials and the transmitted radiations will be allowed to impinge upon thermoluminescent dosimeters for specific time durations. The resulting dose rates will be determined optically and electronically in special "reading" equipment and compared with calculated values.

Specifically, the radiation dose rates from thulium-1/0 sources through lead, stainless steel, tungsten, aluminum and depleted uranium absorbers will be determined by measurement of the luminescence of lithium fluoride phosphors at high temperatures. The energy imparted to this material as a result of interactions with incident radiation is released as luminescence at elevated temperatures. The particular dosimeter used for these measurements will consist of precise quantities of LiF powder metered into special holders. Three thulium-170 sources in strengths of 5, 10 and 20.7 thermal watts will be utilized. Three different thicknesses of each shield material (lead, tungsten, stainless steel, aluminum and depleted uranium) will be used varying from 0.250 to 1.000 inches. The dose rate measurements will made at the shield surface and one meter.

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Representative exposure periods for lead, tungsten and uranium to obtain measurement precision of 5% standard deviation at one meter are tabulated below:

Shield Thickness	Exposure Time (Minutes)					
(inches)	Lead	Tungsten	Uranium			
1	4	6	50			
2	9	17	75			
1.0	45	133	834			

2.0 Objective

The dose rate measurements described herein are to be performed to reliably evaluate the calculated effectiveness of various absorber materials as radiation shields for thulium-170.

3.0 Test Requirements

3.1 Applicable Documents

The measurements described in this procedure will be carried out in accordance with the Statement of Work (Article II) of Contract No. AT-(40-1)-3693: Section b - Phase I, part 2-C and Phase II, part 1-C.

3.2 Measurement Conditions

Radiation dose rate measurements will be performed for each of three thulium sources and each of three different thicknesses of five

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shield materials. Each measurement will <u>utilize five</u> separate dosimeters in accordance with the matrix outlined in Table 3.2.1. The use of <u>five</u> separate TLD dosimeters for each measurement is expected to assure high reliability of test results.

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3.3 Dosimeter Readout

After exposure the thermoluminescent dosimeters will be read by placing them in a specially designed heating device which raises them to a specified temperature at which light is emitted proportional to the amount of radiation absorbed by the dosimeter. This light emission is detected by a photomultiplier tube, the integrated output current of which is electronically converted into a dose reading.

TLD dosimeters utilizing LiF (TLD-700) are available either in preformed geometrical configurations or as loose powder; the ranges of measurement precision vs. exposure dose for each is tabulated below: 1

TABLE 3.3.1

PRECISION AS A FUNCTION OF EXPOSURE FOR
L1F POWDER (TLD-700) AND L1F-TEFLON DOSIMETERS

Exposure Dose (roentgens)	LiF-Teflon Discs	Standard Deviation (%) TLD-700 Powder
0.01	- -	± 20
0.05	±15	
0.30	±10	
0.10		± 5
1.0	± 5	± 2
3.0	± 3	± 2
10.0	± 3	‡ 2
50 to 10 ⁵	± 3	± 5

^{1.} Ejarngard, B.E., et al. Lithium fluoride - teflon thermoluminescence dosimeters. Proceedings: International Conference of Luminescence Dosimetry. Stanford University, Stanford, California. June 1965.

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3.4 Measurement Failure Criteria

A given dose rate measurement result will be deemed void if the value thereof deviates from the group meas value by a factor greater than the standard deviation listed in Table 3.3.1.

3.5 Test Results

Dose rates will be calculated for each test measurement using computed values of source specific activity and published values of bremsstrahlung emission spectra appearing in the literature.

Computed dose rates will be compared with experimental test results and recommendations drafted respecting how anamolies, if any, might be resolved.

3.6 Reports

Monthly status letter reports and a final report will be prepared as stated in the contract.

4.0 Procedure

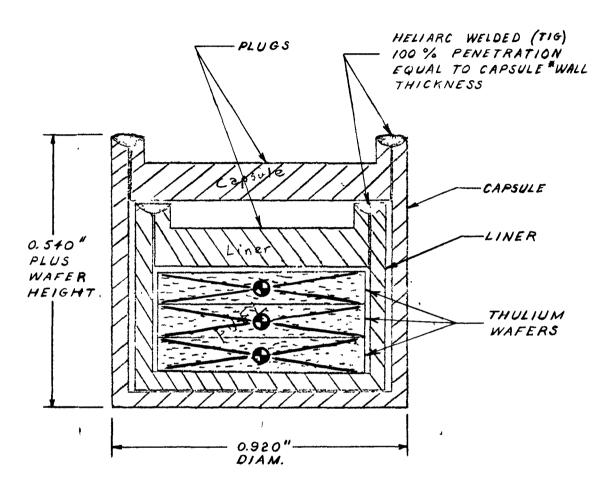
4.1 Receipt of Thulium-170 Sources

It is anticipated that the sources upon which these measurements are to be performed will arrive at Sanders Nuclear Corporation in

^{1.} Arnold, E.D. Handbook of Shielding Requirements and Radiation Characteristics of Isotopic Power Sources for Terrestrial, Marine and Space Applications. ORNL-3576. 1964.

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* note: capsule mat'l to be HASTELLOY C
capsules to be scribed - SNC and numbered according
to source strength as

TM-170
No. 1

FIGURE 4.1.1 REPRESENTATIVE THULIUM SOURCE CAPSULE

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appropriately shielded transportation casks. They will be deposited inside the hot cell where any necessary preliminary operations for source removal will be performed. The hot cell will then be closed and secured. Using remote manipulators in conjunction with a travelling hoist, the casks will be uncapped and the sources removed. Once removed, the sources will be visually inspected and wipe tests will be made on their exterior surfaces. If necessary, weight and dimensional measurements will be made on each source to assure their compatibility with the test fixtures. The radiation level in the hot cell and at the operators station will be continuously monitored and recorded during these operations.

The individual radiation sources will consist of doubly encapsulated stacks of thulium-170 oxide wafers as depicted in Figure 4.1.1.

As noted, both the exterior capsule and the lever will be of Hastelloy C to the dimensions indicated. Since each wafer will comprise approximately 5 watts of thermal energy, it is anticipated that the three sources strengths of 5, 10 and 20.7 watts will consist of 1, 2 and 4 wafers, respectively.

4.2 Dosimeter Exposure Fixture

Having completed preliminary operations with the sources, they will then be individually placed into position on a special radiation

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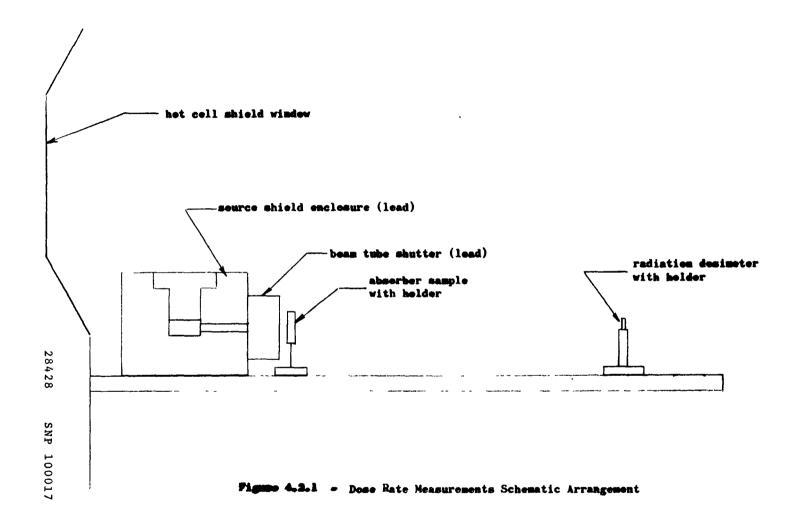


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exposure measurement fixture. During all operations with any one source, all other radioactive sources will be stored inside their shipment casks or storage pigs to reduce extraneous radiation.

The measurement fixture will consist of a rectangular base platform upon which will be mounted: (1) a shielded enclosure for the radiation sources, (2) a fixed holder for the various absorber specimens and (3) another fixed holder for the thermoluminescent dosimeters. This fixture is schematically depicted in Figure 4.2.1. The shield enclosure will incorporate lead walls of adequate thickness that with the strongest anticipated thulium-170 source contained within it, the dose rate at one meter from the enclosure will be no greater than 10 mr/hr. In addithon, the enclosure will have a removeable (or hinged) lead cover over the source cavity of likewise appropriate thickness. The cavity itself will be so constructed and aligned with a radiation beam "tunnel" that with the thulium capsule(s) in place, only the axial radiation emission will be allowed to exit through the tunnel and be incident upon the absorber specimen. The source shield enclosure and the absorber holder are depicted in Figure 4.2.2. Both the absorber and dosimeter holders will be axially aligned with the source capsule/ beam tunnel centerline. In addition, the dosimeter holder (shown in Figure 4.2.3) will be located so that the dosimeters may be positioned at various distances from the vertical source centerline. Furthermore,

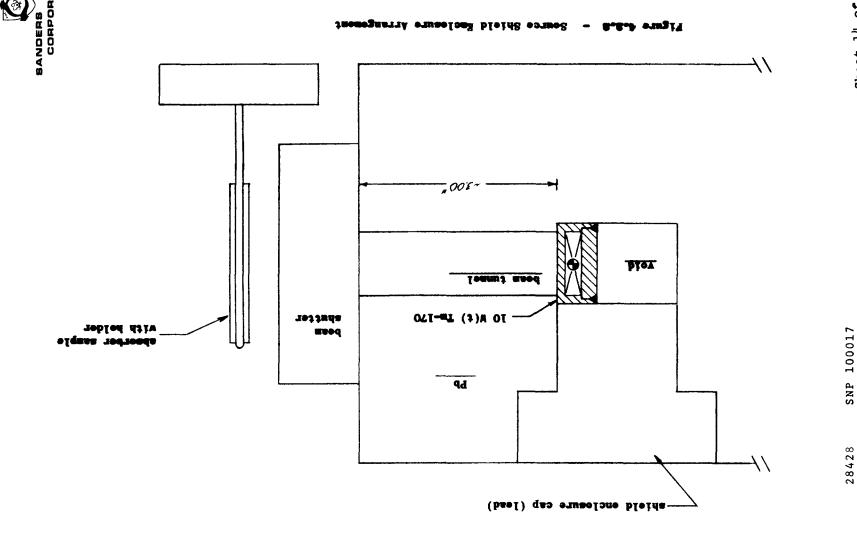
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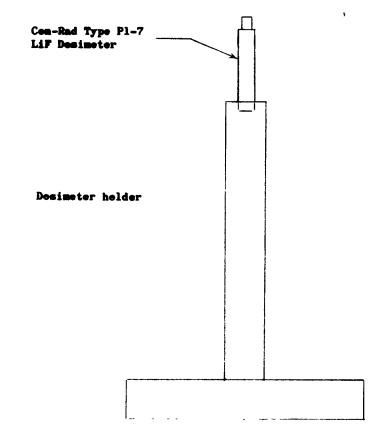


Figure 4.2.3 - Desimeter Mounting Arrangement

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the shield enclosure will incorporate a removeable (or hinged) lead shutter in front of the collimator slit of appropriate thickness that the prededing dose rate specification has maintained.

4.3 Preliminary Test Operations

Prior to commencement of any exposure procedures, the following operations will be performed.

4.3.1. Radiation Absorber Acceptability Criteria

Each of the various absorbers to be used in the measurements will be visually inspected for cleanliness and freedom from surface defects and/or blemishes. These absorbers will then be arranged in special trays (equipped with hinged covers capable of being opened and closed with the hot cell remote manipulators) which will then be placed next to the measurement fixture inside the hot cell within easy reach of the manipulators.

4.3.2. Dosimeter Acceptability Criteria

Each dosimeter to be used in the measurement experiment will be visually inspected for cleanliness and freedom from surface defects and/or blemishes. These dosimeters will then be arranged in special trays (equipped with opaque covers) in groups of eight dosimeters per tray. Three of these trays (24 dosimeters, total) will then be placed within easy reach of the remote manipulators for each dosimeter exposure sequence with a particular absorber

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material. (See Table 3.2.1). In addition, the dosimeters will be located as far as practicable from the stored radiation sources and will be protected by sufficient auxiliary radiation shielding that they will receive minimal spurious radiation exposure during source transfer operations and during purposeful source exposure procedures.

4.3.3 Dosimeter Reader Calibration

The TLD dosimeter reader will be calibrated against a traceable, vendor-supplied standard. This calibration will be repeated at appropriate intervals throughout the performance of measurements.

4.3.4 Dosimeter Geometry Calibration

To assure reproducibility of the geometrical configuration exposure of calibrated TLD dosimeters will be repeated after each change in configuration and/or experiment interruption.

4.4 Test Operations

Having completed the preliminary operations described in Section

- 4.3, the dosimeter exposure operations will be initiated.
 - 4.4.1 The first radiation source to be measured will be removed from its storage cask and transferred into the uncovered shield enclosure, the beam-hole shutter of which shall be closed. The shield enclosure cover will then be replaced.

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- 4.4.2 The particular thickness of absorber to be utilized shall be removed from its storage tray and positioned in its special holder in front of the beam-hole shutter.
- 4.4.3 The first tray of dosimeters to be exposed shall be opened and each dosimeter shall be individually subjected to the following procedures:
 - a) each dosimeter to be placed in position in the dosimeter holder which will then be positioned in the configuration being measured (as required).
 - b) the shutter in front of the radiation beam-hole to be opened for a predetermined length of time.
 - c) the shutter to be closed and the dosimeter to be removed from its holder, the dosimeter being replaced into its original position in its tray. (At all times the tray will be positioned and shielded such as to minimize non-intentional exposure of dosimeters).
- 4.4.4 Succeeding trays of dosimeters will be opened and the sequence of Section 4.4.3 will be repeated, for the successive absorber thicknesses until the transmitted radiation has been monitored with five dosimeters at each thickness for a total of fifteen exposure readings.

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4.4.5 The radiation source will be replaced into storage and the exposed dosimeters will be removed from the hot cell. Additional dosimeters required for the next exposure will be brought in.

4.4.6 The source will be returned to the shield enclosure and the first thickness of the next absorber material placed into the special holder. The complete exposure sequence of Section 4.4.3 and 4.4.4 will be repeated for a total of fifteen exposures, after which the procedure of 4.4.5 will be repeated.

4.4.7 The sequence described in paragraph 4.4.6 will be repeated for the remaining absorber materials, resulting in a total of 75 exposures for the five absorber (candidate shield) materials.

4.4.8 Sections 4.4.3 through 4.4.6 will be repeated for the remaining two sources for a total of 225 exposures.

4.4.9 Having completed the procedures of Sections 4.4.3 through 4.4.8 for each of the three thulium sources and for all absorber specimens, these procedures will then be repeated for each of the remaining geometrical configurations for a total of 450 exposures.

4.4.10 The exposed dosimeters will be inserted into appropriate reader equipment and their absorbed doses measured as detailed above (Section 3.3). The resulting data will be recorded on data sheets, a sample of which is presented in Figure 4.4.10.

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4.4.11 These dosimeters will be annealed after reading to eliminate any thermoluminescence remaining after the irradiation and readout process. For loose LiF powder, this consists of heating it to 400°C for one hour.

4.5 Confirmatory Measurements

Selected configurations of source/absorber/geometry will be duplicated and dose rate measurements repeated for comparison with original data to assure reproducibility of results.

4.6 Data Analysis Operations

The data obtained above will be statistically corrected to yield a single representative value of absorbed dose from each group of five measurements. Comparison of this value of integrated dose with the exposure time in each case will yield the dose rate at a given separation distance due to a known strength of thulium-170 behind a known thickness of absorber material. This result may then be compared with calculated values for identical conditions.

4.7 Measurement Termination

Having completed all the exposures detailed in Sections 4.4 and 4.5 the measurement operations will be terminated by removal of all sources to their storage casks. These will be securely closed and identified. Procedures for ultimate disposal of these sources will

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SECTION 4

PROCEDURE FOR PREPARATION OF Tm₂O₃ WAFERS USAEC SAVANNAH RIVER LABORATORIES

- 1. Dissolve as-received ${\rm Tm_2O_3}$ powder in 5M nitric acid at $80^{\circ}{\rm C}$ with continuous stirring. Do not boil. Batch size is $80^{\circ}{\rm C}$ gms ${\rm Tm_2O_3}$, $600^{\circ}{\rm mil}$ distilled water, and 275 mil concentrated ${\rm HNO_3}$.
- 2. Digest for one hour at 80° C with continuous stirring. Maintain pH = 1-2.
- 3. Dilute with additional 600 ml ${\rm H}_2{\rm O}$. Then cool to room temperature.
- 4. With slow stirring, rapidly add 1 liter of 5M NaOH to precipitate thulium hydroxide. If necessary, cool the beaker during precipitation to keep solution at room temperature. Continue stirring for 30 minutes.
- 5. Filter through glass frit Buchner funnel using suction.
 Wash precipitate at least five times with distilled water,
 breaking up to filter cake and stirring each time the funnel
 is filled.
- 6. Dry precipitate overnight in vacuum oven at $100-110^{\circ}$ C.
- 7. Grind precipitate and screen through 100-mesh sieve.
- 8. Place precipitate in calcining dishes allowing a maximum depth of 1/4 inch precipitate.
- 9. Place calcining dishes into furnace maintained at 750° C. Remove after one hour at 750° C and cool in dessicator.



- 10. Screen Tm₂O₃ through 100-mesh sieve again.
- 11. Place material in plastic bag and add 3% to 5% by weight (based on dry weight of oxide) of binder solution to powder. Binder solution consists of 0.2 gms "Carbowax"* 4000/8 cc H₂0. Seal bag and hand mix for minimum of 30 minutes to obtain uniform blending of binder and oxide.
- 12. Place damp powder into die, using spatula to break up any large air pockets. Press at 30,000 psi. If laminations occur, reduce pressure to 20,000 psi. Mold releases such as "Sterotex"** may be used, but silicone-based releases are specifically excluded.
- 13. Place part on a sprinkling of fired ${\rm Tm}_2{\rm O}_3$ coarse powder on an alumina (99.5% or higher) setter. Fire in air as follows:

200°C/hr to 1000°C, hold 1 hour at 1000°C 200°C/hr to 1750°C, hold 4 hours at 1750°C (Firing Cycle) 200°C/hr to room temperature (Cooling Cycle)

- 14. Grind specimens to appropriate dimensions.
- 15. Check for laminations or other defects. Measure dimensions.

 Determine density.

NOTE: Use plastic beakers during the reprocessing to avoid silicon contamination.

^{*}Union Carbide's polyethylene glycol.

^{**}Capital Cities Products Company, Columbus, Ohio



SECTION 5

SRL THULIUM AND/OR YTTERBIUM OXIDE POWDER REPROCESSING FOR SINTERING

SANDERS NUCLEAR CORPORATION

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1.0 INTRODUCTION

This procedure is based on the wafer production process used by Savannah River Laboratory with modifications for our application. The reprocessing of the as received powder provides a uniformly small particle that is conducive to a reasonable sintering cycle after the green compact is formed. The powder dissolution, precipitation, recovery and calcining is in step by step operation with material and equipment detailed sufficiently to assure repeatability.

2.0 OBJECTIVE

The objective is to provide laboratory personnel with specific direction to produce a powder that is uniform and suitable for use in a forming die. The resultant powder shall produce a high density sintered product with minimal cracking, spalling and warppage.

3.0 PROCESS REQUIREMENTS

- 3.1 APPLICABLE DOCUMENTS
- 3.1.1 Fabrication and Irradiation Behavior of Thulium Sesquioxide by Keski and Smith (SRL) DP-MS-68-28.
- 3.1.2 Savannah River Laboratory Process Sheet, October 12, 1967 (furnished through USAEC-DID).
- 3.2 EQUIPMENT AND MATERIALS
- 3.2.1 Calcining Furnace, normal atmosphere, 1380°F
- 3.2.2 Drying Oven, Vacuum 25 inch @ 212°F.
- 3.2.3 Balance, Analytical to 0.0001 gram accuracy
- 3.2.4 Hot plate with magnetic stirrer
- 3.2.5 Agate mortar and pestle, 85 mm top, well used to reduce silica pick-up during grinding.

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- 3.2.6 Measure Polyethylene Graduate, 500 ml
- 3.2.7 Funnel. Buchner Polyethylene, with 15 cm dia
- 3.2.8 Filter disc Whatman No. 50, 15 cm dia
- 3.2.9 Digest Beaker 600 ml Nalgene
- 3.2.10 Extraction Flasks, Polyethylene Ernlynmeyer side arm 1000 ml
- 3.2.11 Glazed Evaporation Dish, 100 mm dia
- 3.2.12 Harvest Dish, Nalgene (shallow pan)
- 3.2.13 Weighing Dish, Aluminum
- 3.2.14 Cover, Saran Vrap
- 3.2.15 And laboratory tools such as stainless steel spatula, etc.
- 3.3 DEFINITIONS
- 3.3.1 ml milliliter a cubic centimeter
- 3.3.2 5M five molar
- 3.3.3 H₂O Distilled Water
- 3.3.4 HNO3 Nitric Acid, Reagent Grade
- 3.3.5 NaOH Sodium Hydroxide, Reagent Grade
- 3.3.6 Tm₂O₃ thulium sesquioxide, 99.9% purity
- 3.3.7 $$^{\text{Yb}}_{2}$ 03 · Ytterbium Sesquioxide, 99.9% purity

NOTE: All glass units used in process must be seasoned by mechanical abrasion to remove microscopic glass particles prior to use to reduce the contamination of silica to the product.

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- 3.4 LABORATORY PREPARATION
- 3.4.1 Mixing 5M Nitric Acid
- 3.4.1.1 Measure 600 ml of H₂0 into a 3000 ml glass beaker
- 3.4.1.2 Measure 275 ml of concentrated HNO_3 and add to H_2O in beaker and stir several times with plastic rod and allow to cool to room temperature.
- 3.4.1.3 Measure 328 ml of the stirred 5M $\rm HNO_3$ solution into a 500 ml glass beaker and heat to $176^{\rm o}\rm F$.
- NOTE: Do not store in glass container for prolonged period to reduce possibility of silica contamination.
- 3.4.2 Water bath for digest beaker.
- 3.4.2.1 Install support for 600 ml digest (nalgene) beaker into 3000 ml glass beaker to separate bottoms of beakers with $\frac{1}{2}$ inch clearance.
- 3.4.2.2 Add hot tap water, boiling chips, to large beaker.
- 3.4.2.3 Install the covered digest beaker with teflon covered stirring bar and adjust water level to one inch below the edge of the empty beaker.
- 3.4.2.4 Place assembly on magnetic stirring hot plate to reach moderate boil to maintain liquid in digest beaker at 176°F.
- 3.4.3 Powder Weighing
- 3.4.3.1 Determine ratio of Tm_2O_3 and Yb_2O_3 of as received powder for total weight of 30.0000 grams.
- 3.4.3.2 Check balance for zero set, record tare weight of clean weighing dish and add Tm_2O_3 powder per scheduled weight and set covered powder asside. Then weigh Yb_2O_3 powder and set aside.
- 3.4.3.4 Secure balance.
- 3.4.4 Mixing 5M Sodium Hydroxide.
- 3.4.4.1 Measure 600 ml of H₂O into a glass 1000 ml beaker.

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- 3.4.4.2 Check balance for zero, record tare weight of clean weigh dish and add 200.0 grams of NaOH pellets to dish and set aside and secure balance.
- 3.4.4.3 Add pellets to beaker of H_2O and stir into solution to promote reaction (warm temperature) and set aside.
- 3.4.4.4 After solution has cooled pour in glass volumetric flask, add $\rm H_2O$ to the 1000 ml mark.

NOTE: Do not store in glass container for a prolonged period of time to reduce possibility of silica contamination.

- 3.4.5 Precipitate Washing Apparatus
- 3.4.5.1 Install two sets of Buchner Funnels with filter disc in ring stands with extraction flasks.
- 3.4.5.2 Connect vacuum pump to side arm of extraction flasks capable of maintaining 25 inch vacuum during extraction.
- 3.4.6 Material Identity
- 3.4.6.1 Traceability to the vendor and his batch number shall be recorded in the lab notebook by the operator at the time of the wafer batch number assignment. The wafer batch number shall be successive number related to the various compositions.
- 3.4.6.2 Compositions of Tm_2O_3 and/or Yb_2O_3 shall be recorded in the Lab notebook as actual weights to the nearest 0.0001 grams and listed by the following letter code

Composition	Tm203	Yb203
A	100%	0%
В	95%	5%
С	90%	10%
L	80%	20%
F	70%	30%
H	50%	50%
G	30%	70%
J	10%	90%
K	0%	100%

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4.0 PROCESS

4.1 DISSOLUTION OF POWDER

- 4.1.1 Pour heated 5M HNO3 into digest beaker mounted in water bath and await stabilization of 176°F temperature (five minute minimum).
- 4.1.2 Lift digest beaker and add weighed powders. Replace to maintain temperature and keep covered. Start magnetic stirrer and maintain for one hour.
- 4.1.3 Remove digest beaker with diluent from water bath.
- 4.2 FORMING THE THULIUM AND/OR YTTERBIUM HYDROXIDE
- 4.2.1 Pour diluent of digest beaker into the hydroxide beaker (3000 ml nalgene) together with teflon coated magnetic stirring bar.
- 4.2.2 Measure 225 ml of H_2O and add to hydroxide beaker, cover and set aside to cool to room temperature.
- 4.2.3 Set hydroxide beaker on magnetic stirrer (no heat) for slow stir and add 375 ml of 5M NaOH solution rapidly to diluent and recover to stir for 30 minutes.

4.3 PRECIPITATE RECOVERY

- 4.3.1 Carefully pour hydroxide suspension into two funnels (Buchner) to extract filtrate. Then refill the empty hydroxide beaker with 500 ml of H₂O and rapidly stir for five minutes to salvage residue of the hydroxide suspension.
- 4.3.2 Carefully stir the hydroxide suspension with a plastic rod (do not damage filter) until solids become visible and the hydroxide becomes tacky. Add the wash liquid from the hydroxide beaker (4.3.1 above) to wash the precipitate.
- 4.3.3 Continue to add additional H_2O as needed when material in funnel becomes tacky. Periodically interrupt the process to pour off the filtrate in storage containers until a total of $4\frac{1}{2}$ liters of H_2O has been used. This is generally about ten water additions to the funnel. (NOTE: The filtrate must be retained until powder harvest is complete and indicates acceptable loss).

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- 4.3.4 Continue extraction after last wash to form precipitate cake. Scrape funnel and filter disc into a nalgene harvest dish using a stainless steel spatula and plastic stirring rod. Cover the harvest dish with loose fitting teflon sheet as a cover.
- 4.4 DRYING AND GRINDING THE PRECIPITATE
- 4.4.1 Load vacuum oven with covered harvest dish and dry 3 100°C for 15 hours at 25 inch vacuum. Remove from oven and store in aluminum desicator using Dririte as desicant until required for use.
- 4.4.2 Use agate mortar and pestle to grind small lots to pass 100 mesh stainless steel sieve. Recover densified material from grinding tools using plastic stirring rod. Regrind powder that does not pass sieve.

4.5 CALCINING

- 4.5.1 Spread screened powder to $\frac{1}{4}$ inch depth in glazed evaporation dish and load in preheated drying furnace at 700°F. Increase temperature to 1380° F in 30 minutes and maintain for one hour. Remove dishes of powder and set aside on fire brick for five minutes to cool before placing in desicator to complete cooling to room temperature in about two hours. Leave in desicator until required.
- 4.5.2 Again sieve powder through 100 mesh stainless steel sieve. Weigh powder recovered and determine recovery.

NOTE: Typical recovery is 96% of the original 30 grams of as received powder. If loss is in excess of 15% the retained filtrate shall be reworked to increase the harvest.

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SECTION 6

COLD PRESSING AND SINTERING THULIUM OXIDE WAFERS

SANDERS NUCLEAR CORPORATION

SNP-100026



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1.0 INTRODUCTION

This procedure is based on empirical evaluation at Sanders Nuclear of the Savannah River Laboratory process to form green compacts that are suitable for a furnace sinter at normal atmosphere. The detailed process includes techniques bordering on craftsmanship that are now resolved in specific instruction but which nevertheless are continually being improved as observations are confirmed.

The powder is conditioned, pressed as a green compact and sintered by conventional techniques in this process.

2.0 OBJECTIVE

The objective is to present a laboratory operation which has been proven to be reliable on a small scale. We shall continue to use these techniques as the productive output is increased. Gradually mechanized methods shall be integrated in this system to assure a transitional change which shall remain in control.

3.0 PROCESS REQUIREMENTS

- 3.1 APPLICABLE DOCUMENTS
- 3.1.1 Fabrication and Irradiation Behavior of Thulium Sesquioxide by Koski and Smith (SRL) DP-MS-68-28.
- 3.1.2 Savannah River Laboratory Process Sheet dated October 12, 1967 (furnished through USAEC-DID).
- 3.2 EQUIPMENT AND MATERIALS
- 3.2.1 Balance, Analytical to 0.0001 gram accuracy
- 3.2.2 Die Cavity, Split Wall for 5/8 diameter wafer
- 3.2.3 Press Vertical 29 Ton Capacity
- 3.2.4 Sinter Furnace, normal atmosphere, 2650°F

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- 3.3 LABORATORY PREPARATION
- 3.3.1 Mixing the Binder Additive
- 3.3.1.1 Measure 8 ml of H_20 in a small glass screw cap container.
- 3.3.1.2 Add. 0.20 grams of "Carbowax 4000" and agitate to liquify.

 R Union Carbide's Polyethylene glycol
- 3.3.2 Prefired Tm_2O_3
- 3.3.2.1 Retain sintered wafers that are damaged.
- 3.3.2.2 Pulverize in an agate mortar and pestle to 100 mesh size.
- 3.3.3 Material Identity
- 3.3.3.1 Retain the wafer batch identity of the calcined material with Lab Notebook traceability to Vendor and vendor batch number.
- 3.3.3.2 As the wafers are sintered select a numerical identity from one to the total quantity completed of that one batch.
- 3.3.3 Material as calcined powder or damaged cold pressed wafers maintain the wafer batch identity until reused.
- 3.3.3.4 Sintered wafers that are not suitable for further study do not receive a wafer number but are maintained with the wafer batch identity number for possible sacrificial use.
- 4.0 PROCESS
- 4.1 BINDER ADDITIVE
- 4.1.1 Note the recorded weight of the recovered calcined powder.
- 4.1.2 Determine binder weight on basis of 4.0% of calcined powder to 0.01 gram accuracy. Place polyethylene bag on balance and record tare weight and then add binder material to bag as scheduled and secure balance.

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- 4.1.3 Add the blended powder to the bag and secure by three successive $\frac{1}{4}$ inch folds and apply masking tape to assure closure of folded area.
- 4.1.4 Knead the bagged contents thoroughly for 30 minutes and then store in a desicator to age at least two hours before use.
- 4.2 COLD PRESSING OF WAFERS
- 4.2.1 Check the balance for zero set, record tare of clean weighing dish and then add 2.08 grams of the blended powder with binder for each wafer to be pressed and set aside. Replace the balance of the blended powder in the desicator and secure balance (weighing dish of aluminum foil).
- 4.2.2 Add the weighed powder to the split wall die cavity pellet jig (Bernier #SK 30, SK 31, SK 32) assembled with bottom piston in place. Assure powder distribution in the die cavity by use of a stainless steel spatula to break up the air pockets and/or lumps. Assemble the top piston into the die cavity with set plates.
- 4.2.3 Position die and plates in press. Increase pressure to 29 tons and maintain pressure five seconds before securing motor and initiate pressure bleed off. This bleed off should be gradual over a one minute time span to assist in forming a satisfactory green compact.
- 4.2.4 Reposition die assembly with cylinder ejector to lift die cavity free of the die body. Gently open the die cavity to recover the green pressed wafer. Visually examine the wafer surfaces for laminations, cracks or other irregularities.

NOTE: If wafer is rejected at this point, store in desicator with identity and schedule for regrinding with agate mortar and pestle and rescreening using 100 mesh stainless steel sieve with no additional binder.

4.2.5 Place green pressed wafers on alumina setter plate that has been covered with a thin layer of prefired Tm₂O₃ powder and cover and store in desicator until required.

4.3 FURNACE SINTER

4.3.1 Load sinter furnace with green pressed wafers on setter plates while at room temperature with normal atmosphere. Start furnace temperature increase and reset as necessary at rate of 200°F maximum in 30 minutes until plateau of 1850°F is attained (using Blue M Globar

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progressively change from tap one through five). Maintain temperature for one hour.

- 4.3.2 Reset temperature for 2650°F. Typically the Blue M Globar furnace requires 8 hours to increase to this temperature. Maintain temperature for 16 hours.
- 4.3.3 Shut down furnace to initiate cooling to 700°F. This normally requires 15 hours. Open furnace door at this point to increase cooling rate to 300°F which usually takes an additional hour.
- 4.3.4 Remove the setter plates with sintered wafers from the furnace.
- 4.4 DATA RECORD OF EACH WAFER
- 4.4.1 Assign an identity to each wafer. Record the following.
- 4.4.2 Diameter to 0.001 inch
 minimum to determine average for acceptance criteria
- 4.4.3 Thickness to 0.001 inch
 minimum aximum to determine average for acceptance criteria
- 4.4.4 Weight to nearest 0.0001 gram
- 4.4.5 Visual inspection for chips, cracks, laminations, or other irregularities. Record using sketch as necessary.
- 4.4.6 Color variations from standard wafers.
- 4.4.7 Calculate density from dimensions and weight.
- 4.4.8 Store wafers, maintaining identity.

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SECTION 7

PREPARATION OF THULIUM AND/OR YTTERBIUM OXIDE POWDER VIA PRECIPITATION WITH OXALIC ACID, AMMONIUM OXALATE, UREA AND METHYL OXALATE

7.1 PRECIPITATION OF THULIUM WITH OXALIC ACID

- 1. A quantity of thulium oxide was dried for 30 minutes $(2105)^{\circ}$ C under vacuum.
- 2. 20.0019 gms of the dried thulium oxide (previously cooled to room temperature) were weighed into a 400 ml beaker.
- 3. The oxide was dissolved in 80 ml of 1:1 nitric acid. After the water and acid were added, the solution temperature was raised to about 82° C and held for two hours with frequent hand swirling.
- 4. After two hours the solution temperature was raised to the boiling point and held for five minutes, cooled to room temperature, (diluted to 300 ml with distilled water), and gravity filtered through a Whatman #5 filter paper.
- 5. The filtered solution of thulium nitrate was placed on the stirr-plate and heated to 93° C. A teflon coated stirring bar was used for agitation through the precipitation and digestion steps.
- 6. A solution of 21.56 gms of oxalic acid dihydrate in 100 ml of distilled water was added to the hot thulium nitrate solution at an average rate of five ml per minute. Solution temperature at end of addition: 77°C.



- 7. The mixture was allowed to digest at 93° C for fifteen minutes with continued stirring.
- 8. After the digestion period the mixture was allowed to cool to room temperature, filtered in a buchner funnel with vacuum, washed five times with distilled water, and dried in the funnel under vacuum for thirty minutes.
- 9. The product was then dried at 100° C in a vacuum oven overnight.

7.2 PRECIPITATION OF THULIUM WITH AMMONIUM OXALATE

- 1. A 20.0026 gms quantity of thulium oxide was dissolved in 1:1 nitric acid as in 7.1 (1-4) except that the solution was diluted to 200 ml with distilled water.
- 2. The pH of the resulting thulium nitrate solution was raised to 5.0 (meter) using concentrated and then dilute ammonium hydroxide.
- 3. A magnetic stirring bar was placed in the solution and a solution of 24.3 grams of ammonium oxalate in 1000 ml of distilled water was added at an average rate of 41.7 ml per minute.
- 4. The solution was stirred for an additional fifteen minutes, filtered through a standard 8 micron Millipore filter and filter holder assembly, washed five times with distilled water and dried for 30 minutes in the filter apparatus with vacuum applied.
- 5. The product was then dried at 100° C in a vacuum oven overnight.

7.3 PRECIPITATION OF THULIUM WITH UREA

1. A 20.0275 gms quantity of thulium oxide was dissolved in 1:1 nitric acid as in 7.1 (1-4) except that the solution was diluted to 200 ml with distilled water.



- 2. The pH of the resulting thulium nitrate solution was raised to 5.0 (meter) using concentrated and then dilute ammonium hydroxide.
- 3. A solution of 50 grams of urea in 100 ml of distilled water was added to the thulium nitrate.
- 4. The resulting solution was transferred to a round bottom flask fitted with reflux condenser and refluxed for 1.5 hours.
- 5. The solution was cooled to ambient temperature, filtered through Whatman #5, washed five times with water and dried on the filter paper with vacuum for thirty minutes.
- 6. The product was then dried at 100°C in a vacuum oven overnight.

7.4 PREPARATION OF THULIUM OXALATE VIA METHYL OXALATE

- 1. Dissolve 25 grams of methyl oxalate in 506 ml of distilled water.
- 2. Place 250 ml of thulium stock solution (Section 7.1, 1-4 pH adjusted to 3.0 with ammonium hydroxide) and 220 ml of methyl oxalate solution in a round bottom flask fitted with a reflux condenser.
- 3. Reflux for 2.5 hours at $\sim 95^{\circ}$ C. Slight bumping was observed after 45 minutes of refluxing.
- 4. Allow solution to reach ambient.
- 5. Filter the solution through an 8 micron Millipore filter under vacuum.
- 6. Wash five (5) times with distilled water and dry under vacuum for thirty (30) minutes.
- 7. Dry the product overnight at 100° C in a vacuum oven.



SECTION 8 DETERMINATION OF THE TOTAL SURFACE AREA OF RARE EARTH OXIDE POWDERS

SANDERS NUCLEAR CORPORATION

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DETERMINATION OF THE TOTAL SURFACE AREA OF RARE EARTH OXIDE POWDERS

1.0 SCOPE

1.1 Introduction

This procedure is used in the determination of the total surface area of a refractory rare earth oxide such as thulium sesquioxide (${\rm Tm}_2{\rm O}_3$).

The method of Brunauer, Emmett, and Teller has been employed to measure the surface area of this oxide. A powder sample of known weight is brought to liquid nitrogen temperature. Metered quantities of gaseous nitrogen are then added to the helium flushed internal system, and a mixture of known concentrations of helium and nitrogen is obtained. The reduction of pressure resulting from the adsorption of gaseous nitrogen to a monomolecular layer by the liquid nitrogen cooled sample together with the vapor pressure of nitrogen at dewar temperature are all that are required to obtain a BET plot for determination of total surface area of the powder.

1.2 Objective

This procedure is used in the determination of the actual surface area of the thulia and ytterbia powders produced in our laboratory by variation of process parameters. Extrapolation permits establishment of "particle size" to assist in determining the effect on the ceramic body of varying physical and chemical process parameters.

The objective of this procedure is to provide a technique which will allow reproducible results within $\pm 5\%$ accuracy.

2.0 APPLICABLE DOCUMENTS

2.1 Contract

Statement of Work for Contract AT-(40-1)-3693, Contract Change Proposal for Phase III Modification, Thulium Oxide Fuel Characterization Study, July 1969.

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2.2 <u>Instruction Manuals</u>

American Instrument Co., Inc. Instruction Manual No. 889-A for the 5-7300 Aminco Sor-BET.

2.3 <u>Technical Reports</u>

- 2.3.1 Kremen, Jerome, "A New Approach to Surface Area Determinations by Selective Gas Adsorption in a Nitrogen-Helium System," Paper No. 110 Delivered at the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, (March 1-5, 1965).
- 2.3.2 <u>Howard, M.</u>, "Test Report Comments on Aminco Order No. A9-2924", Materials Technology Laboratory, American Instrument Co., Inc., (June 1969).
- 2.3.3 Keski, J. R. and Smith, P. K., "Fabrication and Irradiation Behavior of Thulium Sesquioxide (SRL, DP-MS-68-29).

3.0 PROCEDURE

- 3.1 Equipment is the basic system of the American Instrument Co., Inc., (Sor-BET).
- 3.1.1 <u>Installation</u> (See referenced instruction manual)
- 3.1.2 Controls and Valving (See Figures 1 and 2 and referenced manual.)
- 3.1.3 Leak Testing the Sor-BET (See referenced instruction manual.)
- 3.1.4 Equipment Start-Up (Refer Figures 1 and 2.)
- 3.1.4.1 All valves shall be closed on the Sor-BET
- 3.1.4.2 Gas regulators shall be backed off on both the helium and the nitrogen cylinders.
- 3.1.4.3 Loosen the plug on the front of the Differential Pressure gauge to vent the case to atmospheric pressure.

3.1.5 Shutdown

3.1.5.1 Back-off the regulators on both the helium and the nitrogen cylinders.

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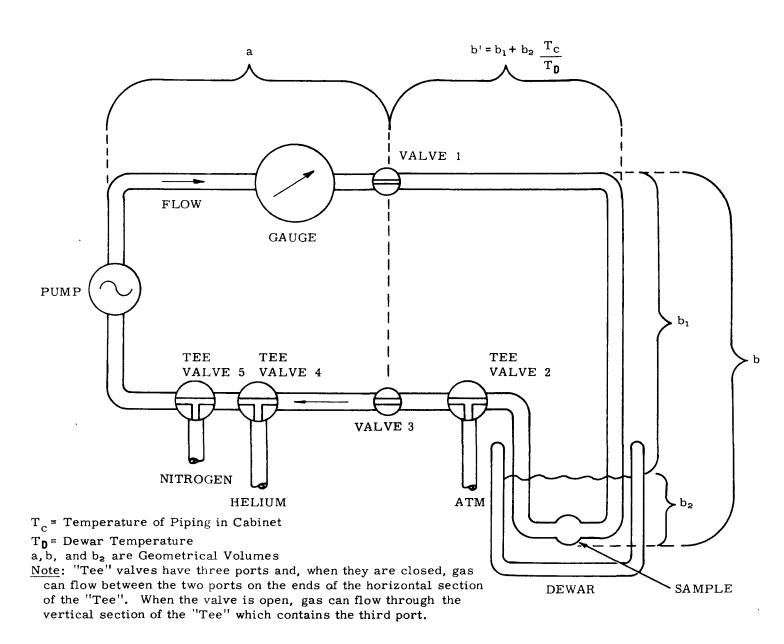


Figure 1. Simplified Flow Diagram

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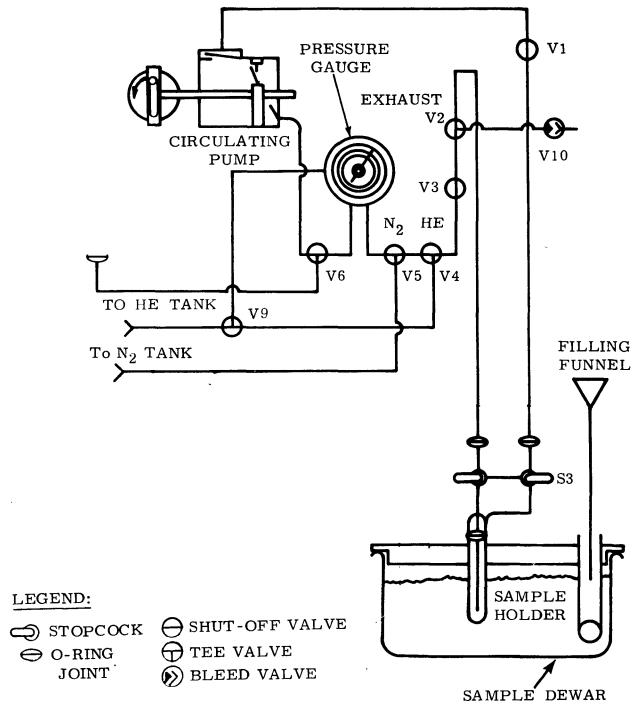


Figure 2. Sor-BET Flow Schematic Diagram

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- 3:1.5.2 Close the main tank valves and the needle valves.
- 3.1.5.3 Close all the valves on the Sor-BET.
- 3.1.6 <u>Sample Holder Modification</u> is necessary to restrain the extreme fines of the thulium oxide powder.
- 3.1.6.1 A three micron metallic filter (Regimesh fabricated by Aircraft Porus Media Inc.) with a diameter of 0.313 inch is joined by flush contact to the bottom of the inner tube of the empty sample holder using an epoxy cement ("Torr Seal" produced by Varian Associates).
- 3.1.6.2 Allow the epoxy cement to cure 24 hours before use of the holder to assure all volatiles are removed.
- 3.2 <u>Pre-Conditioning</u> of sample holder and then the powder sample by heat and a flow of helium entrains other gasses and volatiles from the glassware and the powder sample prior to attachment to the Analyzer. This permits obtaining a constant weight powder sample.
- 3.2.1 <u>Flushing the sample holder</u> is necessary to obtain an initial fixture weight.
- 3.2.1.1 Assemble the empty five cubic centimeter (cc) sample holder together with its 0-ring gasket and clamp.
- 3.2.1.2 Attach the holder (stopcock handles opposed, Refer Figure 3) to the pre-conditioning apparatus by attaching valve S3 to the helium manifold.
- 3.2.1.3 Open valves S4 (handle facing down) and then S3 (handle facing down) and purge the sample bulb with helium at a flow rate of one cubic foot/hour (CFH) and at a temperature of 150° C for twenty minutes.
- 3.2.1.4 Close valves S4 (handle facing out) and then S3 (handle facing out).
- 3.2.1.5 Remove the sample holder and weigh it on an analytical balance reading to the nearest milligram. Be certain not to include the O-ring and wipe joint clean of visible traces of vacuum grease which may adhere to the top of the holder. Record this weight on Form 1.

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SANDERS NUCLEAR CORPORATION 95 Canal Street Nashua, New Hampshire

Sor BET #1

SAMPLE MASS DETERMINATION SHEET

PRECONDITIONING:		START	STOP	TOTAL TIME	
TEMPERATURE: /50 °C	<u></u>	1600 8/2	4 1000 8/25	18 hours	
PRESSURE	11				
ATMOSPHERE <u>He</u>	III				
FLOW / CFH	IV				

SAMPLE IDENTITY	MASS OF FLUSHED	MASS OF PRE-	SAMPLE MASS
٠,	SAMPLE HOLDER	CONDITIONED SAMPLE HOLDER & SAMPLE (2)	(2 MINUS 1)
I Amineo Reference Std. Sample#5	157.0346 g.	154.2466 g	2,2/209
II			
III			

BY m &T	DATE 8-25-69
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FORM 1

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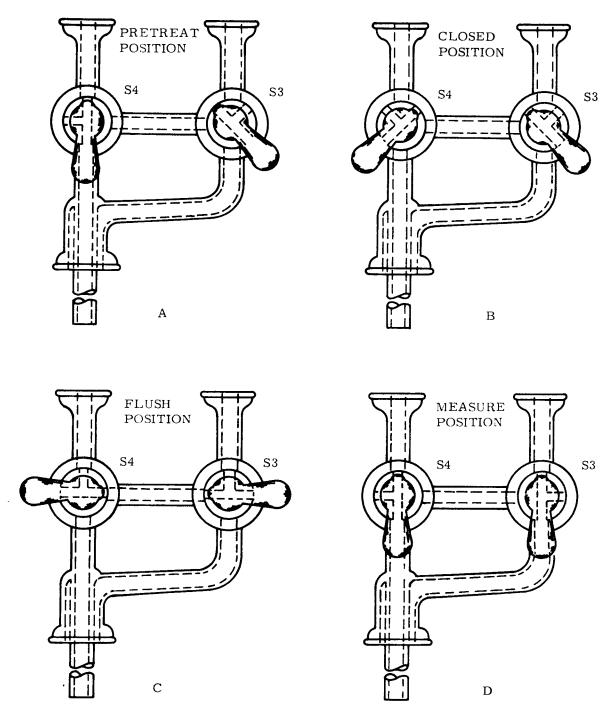


Figure 3. Stopcock Positions

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- 3.2.2 <u>Flushing the powder sample</u> cycle is of sufficient duration to remove contaminating constituents from the processed powder.
- 3.2.2.1 Disassemble the holder and add about eight grams of sample powder and assemble the holder together with its O-ring gasket and clamp making certain to use the same components as a set.
- 3.2.2.2 Reattach the holder to the preconditioning apparatus and outgas the sample for four hours at 150° C with a flow rate of one CFH helium. (Refer PARA 3.2.1.3 and 3.2.1.4)
- 3.2.2.3 Remove the sample holder, weigh and record the weight on Form 1.
- 3.2.2.4 Calculate sample mass utilizing data available. Perform subtraction on Form 1 to obtain the powder sample weight.
- 3.3 Surface Area Determination is by a series of pressure readings of a helium purged system with a partial addition of gaseous nitrogen. As sections of the system are isolated and pressures are regulated the sample holder is immersed in liquid nitrogen with an internal pressure change attributed to monomolecular layer adsorption of the gaseous nitrogen in a known volume. Variations of this pressure change are in direct relation to the total surface area of the powder sample.
- 3.3.1 Install the Sample Holder to the Sor-BET analyzer.
- 3.3.1.1 Close valves V1 and V3, if not already closed. Remove empty sample holder, if attached. (Refer PARA 3.1.4.1)
- 3.3.1.2 Attach the filled preconditioned sample holder to the O-ring connectors on the right side of the Sor-BET cabinet.
- 3.3.1.3 Confirm valves S3 and S4 of sample holder to flush position (arms opposed).
- 3.3.2 <u>Flushing the Nitrogen Line</u> from the gas cylinder to the Sor-BET analyzer will assure positive gas mixture control.

Note: If a previous sample has just been tested, it is unnecessary to flush the nitrogen lines again. Proceed to "Flushing the Helium Line."

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- 3.3.2.1 Open valves V5, V3 and V2. (Sample holder valves S3 and S4 remain in flush position arms opposed.)
- 3.3.2.2 Open the regulator needle valve and adjust the nitrogen regulator for a differential pressure gauge indication of 350 mm of Hg. Flush for one minute.
- 3.3.2.3 Then close valves V2 and then V5.
- 3.3.2.4 Open valve V2 and bleed the excess nitrogen to a differential pressure gauge indication of 10 mm of Hg and then close valve V2.
- 3.3.3 <u>Flushing the Helium Line and the Sample Loop</u> from the gas cylinder will assure the Sor-BET lines are purged.
- 3.3.3.1 Adjust the regulator on the helium cylinder to 8 pounds per square inch (psi).
- 3.3.3.2 Open vlaves V4, V3 and V2. Adjust the regulator for a differential pressure gauge indication of 350 mm of Hg, and flush for one minute.
- 3.3.3. Open valve VI and close valve V3 and flush for one minute.
- 3.3.3.4 Open valve V3 and close valve V1 and flush for one minute.
- $^\circ$ 3.3.3.5 Close valves V2, V3 and V4 simultaneously so as not to exceed 380 mm of Hg on the differential pressure gauge.
- 3.3.4 <u>Determining the "b₂" Space</u> as shown in Fig. 1 is the system that is immersed in the dewar liquid nitrogen bath.
- 3.3.4.1 Open valves V1 and V3 and then open valve S3 slowly (handle down) and then valve S4 (handle down) to admit gas to the sample holder.
- 3.3.4.2 Open valve V2 slightly to allow excess helium to escape until a reading of 200 mm of Hg (P_r), is obtained. Then close valve V2 and record the pressure on Form 2.
- 3.3.4.3 Close valves V1 and V3 and record the pressure, (P_1) , on Form 2.

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SANDERS NUCLEAR CORPORATION 95 Canal Street .
Nashua, New Hampshire

Sor BET #2

FREE SPACE and b SPACE

SAMPLE AMINCO Reference Sample No.5	DATE	8-25-69
Weight, $w = 2.2/20$ g.	ву	MLT
a-SPACE, a = 65.20 cc.		
CABINET TEMPERATURE, $T_C = 296.4$ °K. (°K = °C + 273.2°)		
DEWAR TEMPERATURE, T _D = 77.4°K.		

GEOMETRIC VOLUME AT LN2 TEMPERATURE (b2)

1	2	3	4	5	6 4 - 5	7 4 - 1	8 3 - 2	9	10
FLUSH Pn	CLOSE a	FILL a	EXPAND P3	IMMERSE P	P3 • P4	P3 - P	P2 - P1	T _c /T _D	T _c /T _D - 1
340	344	380	361	11	350	21	36	3,8294	2.8294
			<u> </u>			<u> </u>			
			 						

11 6 - 7	12 8 ÷ 10	13 5 + 760	14 a - 13	15 11 × 12	16 14 × 15	17
$\frac{(P_3 \cdot P_t)}{(P_3 \cdot P_t)}$	$\frac{(P_2 - P_1)}{(T_c/T_D - 1)}$	P ₄ + 760	(P ₄ + 760)		$b_2 = \frac{(P_3 - P_4)(P_2 - P_1) a}{(P_3 - P_1)(T_c/T_D - 1)(P_4 + 760)}$	AVERAGE VALUE OF b ₂
16.666	12,725	77)	.0846	212.07	17.9411	17.944
				•		

FREE SPACE (a + b')

1	2	3	4	5 3 • 2	6 4 - 1	7 5 - 6	8 7 × a	9
FLUSH R	CLOSE a	FILL a	EXPAND P4	P • P 2	P P.	$\frac{(P_2 \cdot P_1)}{(P_+ \cdot P_L)}$	$a + b^{1} = \frac{(P_{2} - P_{1}) a}{(P_{1} - P_{1})}$	AVERAGE VALUE OF (a + b')
20	2/	350	150	329	130	2.53	164,9560	164.9560

FORM 2

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- 3.3.4.4 Open valve V4 slightly until the differential pressure gauge indicates approximately 380 mm of Hg and then close valve V4 and record the pressure (P_2) on Form 2.
- 3.3.4.5 Open valves V1 and V3 and record the pressure (P_3) on Form 2.
- 3.3.4.6 Partially fill the dewar and place it on the dewar platform. Raise the dewar part way and, when the boiling subsides, place the styrofoam cap on the dewar. Then raise the dewar as high as possible.
- 3.3.4.7 Insert the flexible tube from the funnel into the lucite tube in the styrofoam cap so that the end of the tube comes to a point just above the reference mark on the dewar. Fill the dewar until the liquid nitrogen level comes to, or slightly above, the reference mark.
- 3.3.4.8 Allow the dewar temperature to stabilize as indicated by the differential pressure gauge. A monomolecular layer of nitrogen will form on the particles of the powder sample. Refill the dewar if necessary to raise the liquid level of the dewar. Record the stabilized pressure (P₄) on Form 2.
- 3.3.4.9 Calculate the " b_2 " space using the formula on Form 2.

Note: For maximum precision and to prevent error repeat 'Determination of "b $_2$ " Space' (refer PARA 3.3.4.1 through 3.3.4.9).

- 3.3.5 <u>Determination of the Free Air Space</u> as shown on Fig. 1 includes the total Sor-BET piping system (a + b') including the sample holder and the liquid nitrogen immersed section.
- 3.3.5.1 Open valve V4 slightly until a reading of 20 mm of Hg is obtained on the differential pressure gauge and then close valve V4 and then record the pressure (P_r) under Free Space on Form 2.
- 3.3.5.2 Close valves V1 and V3 and record the pressure (P_1) on Form 2.
- 3.3.5.3 Open valve V4 slightly until the differential pressure gauge indicates approximately 350 mm of Hg and then close valve V4 and record the pressure (P_2) on Form 2.

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- 3.3.5.4 Open valves V1 and V3 and record the pressure (P,) on Form 2.
- 3.3.5.5 Calculate the Free Space using the formula on Form 2.

Note: For maximum precision and to prevent error repeat Determination of Free Air Space (Refer PARA 3.3.5.1 through 3.3.5.5)

- 3.3.6 Testing of the Sample is accomplished by admission of a measured gas sample to the sample holder volume. As seen on Fig. 1, the higher pressure of area "a" is admitted into the lower known pressure system area "b" with the attendent pressure equalization that may be related to the sample condition.
- 3.3.6.1 Refill the dewar if necessary to bring the liquid nitrogen to the reference mark.
- 3.3.6.2 Open valve V2 slightly and vent the helium to a pressure of approximately 20 mm of Hg. Close V2 and record the pressure (P_r) on Form 3.
- 3.3.6.3 Close valves V1 and V3 and record the pressure (P_c) on Form 3.
- 3.3.6.4 Open valve V5 and allow the nitrogen gas to enter the "a" space until the differential pressure gauge indicates 250 mm, close valve V5 and record the pressure (P_d) on Form 3.
- 3.3.6.5 Open valves V1 and then V3.
- 3.3.6.6 Adjust the black hand on the gas circulating pump timer to 10 minutes and press the red inner button on the timer to start circulation of the gas mixture through the sample.
- 3.3.6.7 When the sample has equilibrated, record the pressure (P_{α}) on Form 3.
- 3.3.6.8 Repeat PARA 3.3.6.3 through 3.3.6.7 two additional cycles to obtain data for the next BET points with further additions of nitrogen gas.
- 3.3.6.9 Record cabinet temperature of Sor-BET on Form 3.
- 3.3.6.10 Calculate the Surface Area using the formula on Form 3. (Figure 4)
- 3.3.7 Venting the Sample After Testing
- 3.3.7.1 Close valve V1 and open valve V2 to vent the gas gradually.

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SANDERS NUCLEAR CORPORATION 95 Canal Street Nashua, New Hampshire SOR BET #3
SURFACE AREA DATA

SAMPLE AMINEO Reference Sample NO. 5	DATE	8-25-69
Pre-treatment He 150°C 18 hours	ву	MILT
Weight, w = 2,2120 g.		
a-SPACE, a = 65.20 cc.		
FREE SPACE, (a + b') = 164.9560 cc.		
GEOMETRIC VOLUME AT LN, TEMPERATURE, b, = 17.941/ cc.		
CABINET TEMPERATURE, $T_{C} = 296.4^{\circ} \text{K}$. (°K = °C + 273.2°)		
DEWAR TEMPERATURE, T _D = 77.4°K.		
N ₂ SATURATION PRESSURE, P ₀ = <u>758</u> mm. of Hg		

1	2	3	4	5 3 - 2	6 5 × a	Σ 6	8.	9 4 • 1	10 * 8 × 9	11 * (a+b') + 10	12 9 × 11
FLUSH P.	CLOSE a	DOSE .	EXPAND Pe	P.P.	D = (P _d -P _c)a	ΣD = Σ(P - P) =	$K_a = \frac{5 \times 10^{-5} T_c b_2}{T_D}$	P= Pe-P	ν _α = κ _α (P _e • P _n)	(a+b') + V _a	V _f =(P _e -P _r () `^(a+b'+V _α)
20	22	250	54	228	14866	14866	$\frac{5 \times 10^{-5} \text{ T}_{c}^{b}}{77.4} =$	34	-1156	165.072	56/2
	56	250	120	194	12649	275/5	7	100	3400	165.296	16530
	122	280	176	158	10302	37817		156	15304	165.486	25816
	178	280	212	102	6650	44467	Ka=.0034	192	.6528	165,609	3/797
···											

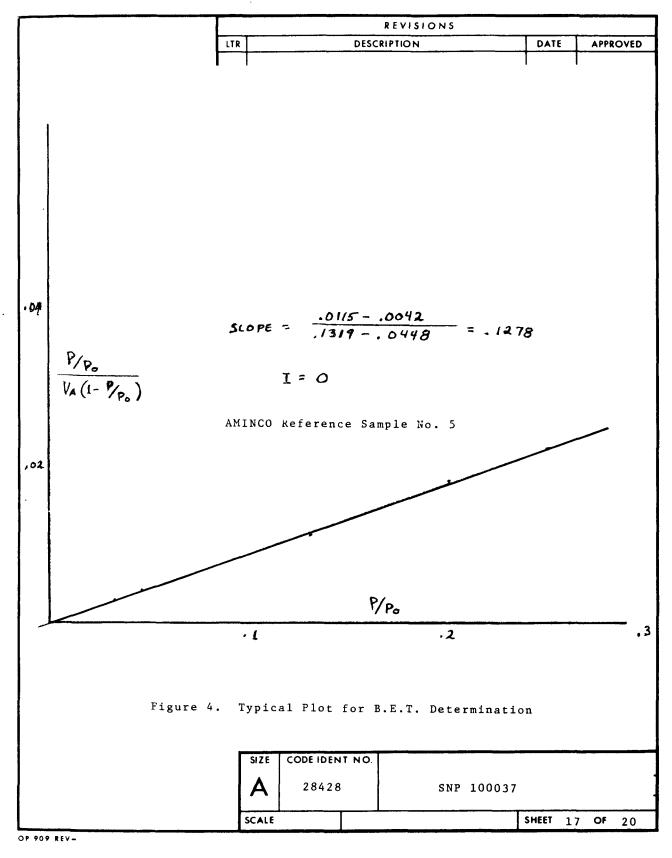
13	14 7 - 12	15 13 × 14	16 9 ÷ P _o	17 1.0 - 16	18 15 × 17	19 16 ÷ 18	20	21	22 1.0-(20+21)	23
$K_2 = \frac{273.2}{760 \times T_c}$	ΣD·V _f	V_A , cc, STP = $(\Sigma D \cdot V_f) K_2$	P/P _o	1 - P/P _o	V _A (1-P/P _o)	P/P _o V _A (1 • P/P _o)	(GRAPH) S	(GRAPH) I	$V_m = \frac{1}{S+1}$	$A = \frac{4.38 V_m}{W}$
$\frac{359.5 \times 10^{-3}}{T_c} =$	9254	11.1048	.0448	9552	10.607	.0042			S+1=	SURFACE AREA,
Tc	10975	13.170	.1319	.8681	11.433	.0115	.0-0		.0838	M ² /G
	12001	14.400	.2058	.7942	11.436	,0179	.0838	0	V _m =	A =
K2=,00/2	12670	15.204	.2533	.7467	11.353	,0223			,	23.63
·										

[•] OMIT IF FREE SPACE ONLY IS DETERMINED.

FORM 3

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- 3.3.7.2 Lower the dewar and remove it from the platform. Cover the dewar to prevent condensation.
- 3.3.7.3 When the sample bulb has defrosted, close V2.

4.0 QUALITY ASSURANCE

The condition and status of all materials used in this testing shall be established at the time of receipt and the identity maintained and recorded at time of the operation.

- 4.1 The history of the material submitted to test shall be available with identification to handling and/or processing. Traceability of such identification shall be maintained by use of the referenced forms, Engineering Notebooks, Process Logs and data sheets.
- 4.2 <u>Testing materials</u> shall be visually reviewed on receipt for conformance to available procurement data with reference to certification when available. Identity shall be established and maintained by use of referenced forms, Engineering Notebooks, Process Logs and data sheets.
- 4.3 <u>Calibration of equipment</u> shall be by the Sanders Associates Calibration Lab and laboratory personnel as applicable.
- 4.3.1 <u>Specification MIL-Q-9858A</u>, when applicable, shall use an annual frequency per Specification MIL-C-45662.
- 4.3.2 <u>Functional Calibration</u> of the BET is an operational function as relates to this procedure. Review shall be periodic to assure conformance to this procedure. Necessary deviations should be proposed as revisions to this procedure and noted on the test forms.
- 4.3.2.1 Use Section 3 of this procedure for operational calibration.
- 4.3.2.2 Repeat calibration at least once a day using AMINCO Reference Sample No. 5.
- 4.3.2.3 Surface area calibration standards shall be "sole source supplied" by American Instrument Co., Inc.

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- 4.4 <u>Laboratory techniques</u> used shall be commensurate with good laboratory practice.
- 4.5 <u>Records</u> shall be maintained in the laboratory available for review by Quality Assurance personnel.
- 4.6 <u>Precautions</u> should be taken to minimize handling of the equipment.

 Minute leakage causes drastic changes in the test results.
- 4.6.1 Valve V9 should be closed at all times.
- 4.6.2 The differential pressure gauge plug should be left loose to vent trapped air for helium carrier operation.
- 5.0 PREPARATION FOR DELIVERY (not applicable)
- 6.0 NOTES
- 6.1 Glossary of Terms (Refer to Figures 1, 2 and 3.)
- 6.1.1 "a" space The geometrical volume of the piping to which the gases have been added.
- 6.1.2 "b₂" space The geometrical volume of the filled sample bulb at liquid nitrogen temperature.
- 6.1.3 Dewar 1250 milliliter, silvered, containment vessel for the liquid nitrogen.
- 6.1.4 Differential Pressure gauge A non absolute gauge referenced to atmosphere indicating in mm of mercury.
- 6.1.5 Free space The total effective volume of the system.
- 6.1.6 Helium manifold Connection on the preconditioning apparatus used to connect the sample holder for a helium purge.
- 6.1.7 Preconditioning apparatus Used to outgas a sample at elevated temperature.
- 6.1.8 Pump timer Controls the length of time during which a gas mixture is circulated through the sample.
- 6.1.9 Reference mark Corresponds to one inch above the constriction on the sample holder in the dewar flask to show liquid nitrogen level.

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- 6.1.10 Regulator Used on both the helium and the nitrogen cylinders to reduce the cylinder pressure to the required pressure.
- 6.1.11 Sample holder Containment vessel for the powder sample. This procedure is limited to 5cc size.
- 6.2 <u>Materials and/or Measurements</u>
- 6.2.1 Calibration Standards Reference adsorbent samples from the National Bureau of Standards Bone Char Project.
- 6.2.2 Abbreviations
 - (a) Oc = degrees Centigrade
 - (b) CFH = cubic foot/hour
 - (c) g = grams
 - (d) mm of Hg = millimeters of mercury
 - (e) psi = pounds per square inch

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SECTION 9

PROCEDURE FOR DETERMINING OXYGEN IN THULIA THULIA/YTTERBIA FOR THE PURPOSE OF DETERMINING METAL-TO-OXYGEN RATIOS

SANDERS NUCLEAR CORPORATION
SNP-100036

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Determination of the Oxygen Content in Thulia Wafers

1. [ntroduction

This procedure is used in the determination of the metal to oxygen ratio in a refractory rare earth oxide, thulium sesquioxide (Tm_2O_3) , having a nominal 12.5 w/o oxygen.

An inert gas fusion method has been employed for the direct determination of oxygen in this oxide. A sample of known weight is dropped into a heated graphite crucible where the oxides are reduced in the presence of excess carbon. The resulting carbon monoxide, which contains all of the oxygen in the sample, is then swept over an oxidizing agent, copper oxide, by an inert gas, helium, where the carbon monoxide is converted to carbon dioxide. This gas is then isolated in a collection trap and after a preset time the gas is released and carried into a packed column of silica gel. The CO₂ then passes over a thermal conductivity cell and the change in resistance in the Wheatstone bridge is integrated and read directly on the digital voltmeter as percent oxygen. Standard oxygen samples are used in calibrating this apparatus thus eliminating the need for preparation of a graph.

2. Objective

The objective of this procedure is to provide a technique which will allow reproducible results using standards that are traceable to other techniques or laboratories for comparative purposes. In particular, this procedure is used in the determination of the actual oxygen content in thulia wafers as produced in our laboratory.

3. Applicable Documents

3.1 Contract

Statement of Work for Contract AT-(40-1)-3693, Contract Change Proposal for Phase III Modification Thulium Oxide Fuel Characterization Study, July 1969.

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3.2 Instruction Manuals

- 3.2.1 Laboratory Equipment Corporation Instructions and Operation Manual (Form 176B) for the 734-300 Rapid Oxygen Analyzer Package and the 589-600 Rapid Oxygen and Low Carbon Analyzer.
- 3.2.2 Laboratory Equipment Corporation Instruction
 Manual for the Operation of the Leco Induction
 Furnace No. 537.

3.3 Technical Reports

- 3.3.1 Doherty, P.E., "Precise Determination of Oxygen in Refractory Oxides by Inert Gas Fusion Gas Chromatography," Savannah River Laboratory, (July 1967). (SRL DP 1115).
- 3.3.2 MacDonell, H. L., et al, "Determination of Oxygen in Glasses, Refractories, and Refractory Oxides by the Inert Gas Fusion Method," Analytical Chemistry, Vol 35, pp. 579-82 (April 1963).

4. Procedure

- 4.1 Installation (see referenced instruction manuals).
- 4.2 Testing the Analyzer for Leaks (see referenced instruction manuals).
- 4.3 Testing the Induction Furnace for Leaks (see referenced instruction manuals).
- 4.4 Equipment Start-up.

4.4.1 Analyzer

- 4.4.1.1 Remove the plug from the end of the purge hosing.
- 4.4.1.2 Set the helium cylinder regulator for 20 pounds per square inch (psi).

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- 4.4.1.3 Adjust the helium rotometer to an indicated flow of "24" (no units).
- 4.4.1.4 Turn the AC switch on. Red light glows.
- 4.4.1.5 Allow the resistance furnace to heat.

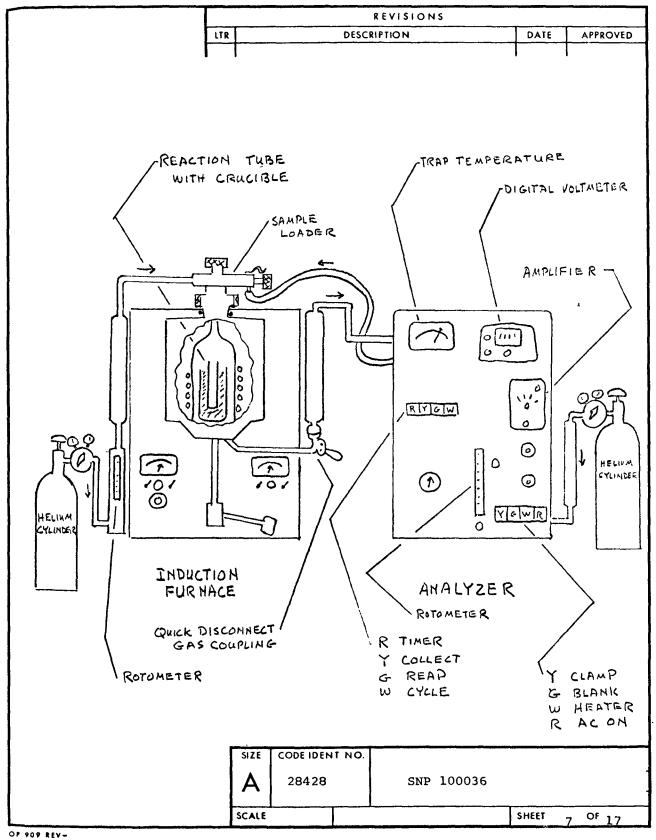
 Normal cycle requires four hours until stable.
- 4.4.1.6 Turn the selector switch of the digital voltmeter (DVM) to the NORM position.

4.4.2 Induction Furnace

- 4.4.2.1 Turn the water valve on. The pressure gauge on the wall should read in excess of 20 psi.
- 4.4.2.2 Set the helium cylinder regulator for 10 psi.
- 4.4.2.3 Adjust the helium rotometer for a flow of 2 liters per minute.
- 4.4.2.4 Turn the filament switch on. Green light glows and meter indicates current flow.
- 4.4.2.5 Turn the catalyst furnace switch on.
- 4.5 Balancing the Digital Voltmeter (DVM).
 - 4.5.1 Turn the selector switch of the DVM to the REV position.
 - 4.5.2 Place the range switch of the DVM in the 1.000 position.
 - 4.5.3 Turn the amplifier select control to the DVM zero position and adjust the DVM to zero using the zero adjust.

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- 4.5.4 Turn the amplifier select control to the Amplifier Balance position and adjust the DVM to zero using the Amplifier Balance adjust.
- 4.5.5 Turn the amplifier select control to the System Balance position and adjust the DVM to zero using the System Balance adjust. Lock in position.
- 4.5.6 Turn the amplifier select control to Operate.
- 4.6 Calibrating the Analyzer
 - 4.6.1 Turn the Timer and Clamp switches on.
 - 4.6.2 Run a blank on the outgassed crucible by depressing the Cycle switch. This reading should be less than 15 parts per million (any reading on the DVM would indicate a reading in excess of 15 ppm). (Refer Paragraph 4.7).
 - 4.6.3 Turn the Blank switch on. Green light glows.
 - 4.6.4 Run the 0.198 mg oxygen calibration standard using the Sample Analysis Procedure: (Refer Para. 5.2).
 - 4.6.4.1 When a stable reading is obtained on the DVM adjust the DVM to indicate .00943 by changing the blank adjust dial. After the Read light comes on, the DVM is clamped and if an additional 20 seconds is needed to complete adjustment, the Clamp switch must be turned off. Lock the dial in position.
 - 4.6.5 Run the 0.298 mg oxygen calibration standard using the Sample Analysis Procedure: (Refer Para.5.2).
 - 4.6.5.1 When a stable reading is obtained on the DVM adjust the DVM to indicate .01420 by changing the slope adjust dial. After the Read light comes on, the DVM is clamped and if an additional 20 seconds is needed to complete the adjustment, the Clamp switch must be turned off. Lock the dial in position.

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- 4.6.6 Verify the calibration by running both standards with the Clamp switch on. The correct values should appear on the DVM when the Read light comes on.
- 4.7 Packing and Outgassing the Crucible
 - 4.7.1 Screen the carbon black through a 20 mesh sieve.
 - 4.7.2 Place 2 inches of the screened carbon black in the thimble and insert the crucible so that the top of the crucible is at the same level as the top of the thimble.
 - 4.7.3 Place the spider over the crucible to keep it clean and fill the remaining voids to within 1/4 inch of the top of the crucible. Tap the thimble regularly to insure removal of any air pockets in the carbon black. Take care not to pack the crucible either too loosely or too tightly. Remove the spider.
 - 4.7.4 Review Equipment Start-up Induction Furnace Procedure. (Refer Para. 4.4.2).
 - 4.7.5 Open the Quick Disconnect gas coupling.
 - 4.7.6 Readjust the helium rotometer to a flow of 2 liter per minute.
 - 4.7.7 Remove the pedestal tray holding device and lower the raising pan mechanism and remove the expended parts.
 - 4.7.8 Place the packed crucible on the raising pan and lift into the reaction tube.
 - 4.7.9 Secure with the pedestal tray holding device and lock into position using the handle on the raising pan mechanism post.

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- 4.7.10 Turn the High Voltage switch on. Note light and meter indication.
- 4.7.11 Gradually heat the crucible by the following schedule by rotating the phase shift knob clockwise:

200MA - 5 minutes and then increase to 400MA - 5 minutes and then increase to 600MA - 5 minutes and then increase to 800MA - 30 minutes

NOTE: While bringing the temperature up watch for "hot spots" on the thimble indicative of uneven packing. If these are observed repack the crucible.

- 4.7.12 Lower the temperature until 2100°C is measured using an optical pyrometer. This setting will vary with each crucible assembly.
- 4.7.13 Close the quick disconnect gas coupling to the analyzer.
- 4.7.14 Readjust the helium rotometer on the furnace to a flow of 2 liters per minute.
- 4.8 Daily Shutdown
 - 4.8.1 Analyzer
 - 4.8.1.1 Leave the AC switch and the DVM on to maintain the resistance furnace at temperature.
 - 4.8.1.2 Turn all other switches OFF.
 - 4.8.1.3 Readjust the helium rotometer to an indicated "5" (no units).

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4.8.2 Induction Furnace

- 4.8.2.1 Rotate the phase shift knob counterclockwise.
- 4.8.2.2 Turn off the catalyst furnace switch.
- 4.8.2.3 Readjust the helium rotometer on the Induction Furnace to a minimum positive flow.

4.9 Prolonged Shutdown

4.9.1 Analyzer

- 4.9.1.1 Complete the daily shutdown
- 4.9.1.2 Turn off the AC switch and the DVM to start cooling the resistance furnace.
 This typically requires 3 hours.
- 4.9.1.3 After the furnace is cool secure the helium flow by halting the helium supply at the cylinder valve.
- 4.9.1.4 Cap the gas outlets (two) to prevent backstreaming of air into the Analyzer System.

4.9.2 Induction Furnace

- 4.9.2.1 Complete the daily shutdown
- 4.9.2.2 When the catalyst furnace has cooled (typically this requires 3 hours) secure the helium flow by halting the helium supply at the cylinder valve.
- 4.9.2.3 Turn off the filament switch.
- 4.9.2.4 Secure the water valve.

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5. Oxygen Determination

5.1 Sample Preparation

- 5.1.1 Thoroughly clean any foreign material from the surface of the sample.
- 5.1.2 Wash the sample in reagent grade acetone using an ultrasonic cleaner.
- 5.1.3 Dry under vacuum at 35°C overnight (minimum 12 hours).
- 5.1.4 Pulverize the dried sample using a clean agate mortar and pestle.
- 5.1.5 Weigh out 2.1 mg of sample on an analytical balance.
- 5.1.6 Place the weighed sample in a tin capsule, flatten the capsule, and fold over to seal.

5.2 Sample Analysis Procedure

- 5.2.1 Pull the sample plunger out to the load position and insert the sample.
- 5.2.2 Attach the purging hose to the bottom of the plunger and flush with helium for 10-15 seconds.
- 5.2.3 Push the sample plunger in and disconnect the purging hose.
- 5.2.4 Wait for 20 seconds to permit adequate flushing of the chamber.
- 5.2.5 Simultaneously rotate the plunger 180° and depress the Cycle switch.
- 5.2.6 When the Read light comes on Record the DVM Indication on the supplied data sheet.

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- 5.2.7 Run a duplicate sample as soon as the Read light comes on.
- 5.3 Calculations Previous calibration of the Analyzer has preadjusted the DVM to indicate directly in percent oxygen as compared to the two standards (See Figure 2).
 - 5.3.1 Record the DVM read out on the Oxygen Analysis Data Sheet.
 - 5.3.2 Repeat using duplicate samples.
 - 5.3.3 Determine the arithmetic average and record on the data sheet.
- 6. Quality Assurance
 - 6.1 Calibration Maintenance Refer to Balancing the DVM and Calibrating the Analyzer. (Refer Para. 4.5 and 4.6).
 - 6.1.1 Repeat daily or after each crucible packing and outgassing as required.
 - 6.1.2 Oxygen calibration standards shall be sole source supplied by Laboratory Equipment Corporation.
 - 6.1.3 Reference Sources fabricated for unusual samples (beyond the range of available LECO standards) shall be confirmed by reference to Laboratory Equipment Corporation Standards.
 - 6.2 Operational Maintenance Refer to Sample Preparation and Sample Analysis Procedure (Refer Para. 5.1 and 5.2).
 - 6.2.1 All materials used shall have traceability to a vendor.
 - 6.2.2 Techniques used shall be commensurate with good laboratory techniques.

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Figure 2 OXYGEN ANALYSIS DATA SHEET

Sample Identity	Oxy	gen	%	Avg % oxygen	calcd oxygen	Deviation %
1.typical Tm ₂ 0 ₃	12.00	12.38	12.97	12.45	12.46	-0.1
2.						
3.						
4.						
5.						
6.						
7.						
8.						
9.						
10.						
STANDARD						
11.						
12.						
13.						
14.						
15.						
16.						
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18.						
19.						
20.						

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- 6.2.3 Discrepancies shall be noted on the Oxygen Analysis Data Sheet.
- 6.3 Records Refer to Calculations and Oxygen Analysis
 Data Sheet. See Figure 2.
 - 6.3.1 Records shall be maintained in the laboratory available for review by Quality Assurance.

6.4 Precautions

- 6.4.1 Maintain the helium flow and water cooling until the furnaces have cooled to room temperature. Typically this requires three hours.
- 6.4.2 Do not remove the housing covers from the Induction Furnace or Analyzer. Refer to the Instruction Manuals.

7. Glossary of Terms

- 7.1 Equipment (Refer to Figure 1).
 - 7.1.1 Analyzer Composed of a collection trap, a
 Wheatstone bridge circuit with one
 thermal conductivity cell, and a
 digital voltmeter.
 - 7.1.2 Catalyst Furnace Converts the carbon monoxide to carbon dioxide.
 - 7.1.3 Crucible A graphite container used as the electrical susceptor for the induction furnace coil and as the reaction vessel for the sample.
 - 7.1.4 Digital Voltmeter Presents a four digit direct readout of precent oxygen.

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- 7.1.5 Induction Furnace Generates a high-frequency electrical field in the center of an induction coil to heat the sample.
- 7.1.6 Quick Disconnect Gas Coupling Joins the induction furnace system to the Analyzer.
- 7.1.7 Reaction Tube A quartz enclosure in the induction furnace which funnels the carbon monoxide to the copper oxide.
- 7.1.8 Regulator Used on both helium cylinders to reduce the cylinder pressure to the required pressure.
- 7.1.9 Resistance Furnace Located in the analyzer and heats the molecular sieve trap to facilitate the release of carbon dioxide.
- 7.1.10 Rotometer Flow indicator
- 7.1.11 Spider A cover for the crucible to prevent the carbon black from falling in while packing.
- 7.1.12 Sample Loader A mechanical device which both isolates a new sample for a preliminary gas purge and introduces the sample into the crucible.
- 7.1.13 Thimble A holding device for the graphite crucible.
- 7.1.14 Oxygen Analysis Data Sheet (Refer to Figure 2).
- 7.2 Materials and/or Measurements
 - 7.2.1 Calibration Standards High purity compounds encased in tin capsules.

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	AC - alte	ernating curre gital voltmete	ent		
	My - mil	liamp electric	er eel ourront		
	ma = mil	ligram weight	di cullenc		
	nom - nai	rts per millio	on (1 ppm = 0.000	120/1	
	psi - po	unds per squar	re inch pressure	11%)	
	w/o - we:	ight percent	re men pressure		
	, -	-3c pozodc			
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SECTION 10

DETERMINATION OF THE IMPACT RESISTANCE TO FINES GENERATION OF SINTERED RARE EARTH OXIDE

SANDERS NUCLEAR CORPORATION

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DETERMINATION OF THE IMPACT RESISTANCE TO FINES GENERATION OF SINTERED RARE EARTH OXIDES

1. SCOPE

1.1 Introduction

This procedure was developed for the determination of particle fines of the respirable range of ten microns that may occur during an accidental impact of a radioactive substance.

During the development and subsequent application of isotopic heat sources fabricated from sintered rare earth oxides and other materials used in extra terrestrial missions, questions had been raised about fuel behavior in launch abort, controlled or uncontrolled re-entry and re-entry impact conditions. The procedure is intended for use with materials prior to irradiation and their subsequent isotopic conversion such as thulium sesquioxide (Tm $_2$ 0 $_3$), but is also adaptable to a hot cell schedule at a future time.

1.2 Objective

The procedure is an analytical method for a semi-quantitative measurement of fines, generated by a consistent impact technique of a ceramic wafer. The standard sieve sizes of less than 10 micron, 10-44 micron, 44-149 micron and larger than 149 micron include the respirable range of 10 microns and are indicative of the total wafer condition.

2. APPLICABLE DOCUMENTS

2.1 Contract

Statement of Work for Contract AT-(40-1)-3693, Contract Change Proposal for Phase IV Modification, Thulium Oxide Fuel characterization Study, July 1969.

3. PROCEDURE

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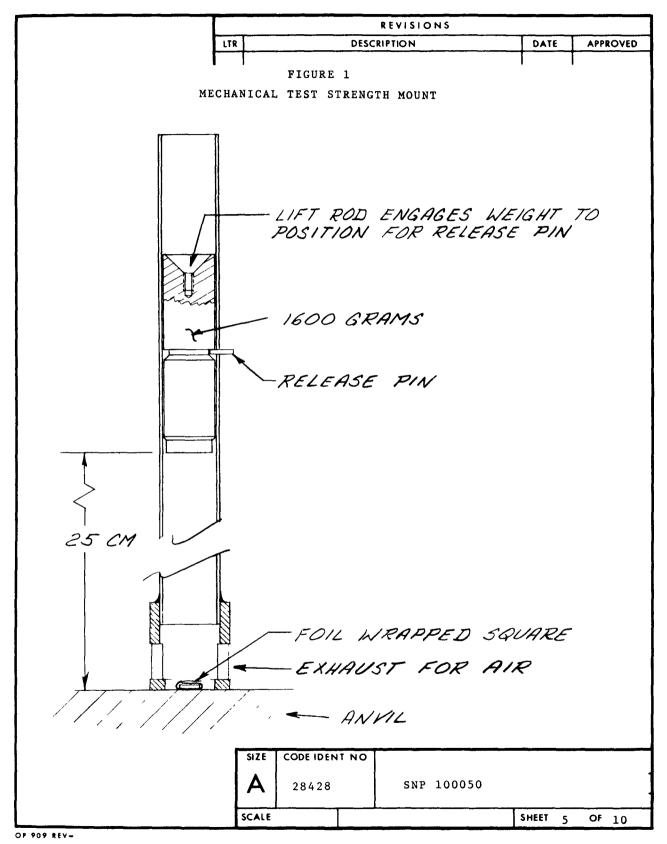
- 3.1 Equipment was devised by Sanders Nuclear Corporation while using standard sieves for analysis of the particle size.
- 3.1.1 Mechanical strength test mount consisting of a drop tube vertically mounted complete with 1600 gram free fall weight with release device (see Figure 1) at 5 cm increments and impact anvil. The threaded lift rod as a positioning device for the free fall weight is an accessory.
- 3.1.2 Sieves fabricated by Buckbee Mears Co., are 3 inch diameter stainless steel mesh.
- 3.1.2.1 Nominal 10 micron calibrated 8-12 micron.
- 3.1.2.2 Nominal 44 micron calibrated 42-46 micron.
- 3.1.2.3 Nominal 149 micron calibrated 147-151 micron.
- 3.1.2.4 Cover and pan set for 3" diameter sieves.
- 3.1.3 Analytical balance, sensitivity 0.0001 gram (Ainsworth Type 21N).
- 3.1.4 Ultrasonic vibratory source equivalent to Buehler Ltd Ultramet sonic cleaner with water treated with Alconox detergent to form a couplent.
- 3.1.5 Expendable supplies needed such as 15 ml disposable polyethylene beaker, aluminum foil equivalent to Reynolds Wrap Super X, measuring 0.0015 inch thick, liquid Freon type TF in a squeeze bottle dispenser and methyl alcohol deagent Grade.

3.2 Sample Preparation

3.2.1 The sieve assembly, consisting of 10, 44 and 149 micron sieves, pan and cover, is first washed with methyl alcohol and individually placed into a drying oven maintained at 80° C for 30 minutes.

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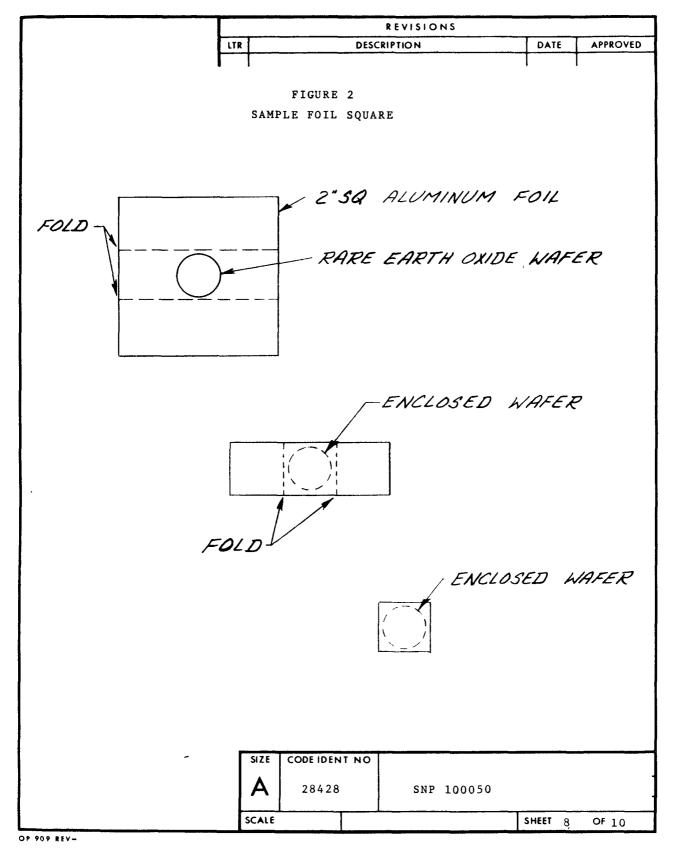
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- 3.2.2 After drying, the parts are removed from the oven using tongs and allowed to stand in air for 5 minutes; the set is then assembled and placed into a desiccator for one hour. The five individual parts of the sieve assembly are then weighed empty using the analytical balance with weights recorded to tenth milligram on the Impact Analysis Data Sheet. (See Form 1). As the weighings proceed, the individual parts of the assembly are put back together with the top sieve the largest mesh (149μ) . NOTE: Tongs are used in all handling steps.
- 3.2.3 A two inch (nominal) square of aluminum foil is then cut and weighed.
- 3.2.4 The sample is then weighed, and its weight and identification number is recorded on the Impact Analysis Data Sheet.
- 3.2.5 The sample is placed in the middle of the foil square, (see Figure 2). The top and bottom of the foil is then folded over the sample. The ends of the foil are then folded, one end on each side of the sample. Final size of the wrapped sample is approximately a 5/8 inch square.
- Impact and Fines Measurement utilizes a falling weight to shatter the enveloped ceramic wafer and a system of sieves with a strict routine of handling and weighing to determine the actual particle size distribution and in particular the percentage of fines of ten microns or less.
- 3.3.1 The foil wrapped sample is visually centered on the base of the Mechanical Strength Test Mount, equidistant from the walls at the base of the drop tube.
- 3.3.2 The weight is lowered into the drop tube until it is 25 cm above the sample and is held at that point by a release pin and the lifting rod is then removed. After checking that the drop tube is in contact with the base, the release pin is removed and the weight is allowed to free fall for impact with the sample.

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			PRE	IMPAC	T WEIG	HTS (G	MS)						PACT W					PE	RCENT	FINES	
ATE	SET	Wgt. Wafer	Wgt. Foil	Wgt. Cover	Wgt. Pan	Wgt. 10بر Sieve	Wgt. 4444 Sieve	Wgt. 149 u Sieve			Wei: Foil	ht Po Cover	vder R Pan	emaini 10 X Sieve	ng In 44 4 Sleve	149 Sieve				44-1494	4714 5
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3.3.3 The weight is extracted from the drop tube by again attaching the lift rod and the impacted sample removed.

A beaker is placed in the center of a 5 inch square piece of aluminum foil. The foil around the sample is then carefully unwrapped over the beaker in a reversed manner, allowing the remains of the sample to fall into the beaker. Any particles on the foil beneath the beaker are deposited into the beaker. The now opened foil envelope, which had contained the impacted sample, is flushed with a liquid stream of highly volatile Freon TF with the residue collected in the same beaker.

- 3.3.4 The sieve assembly is placed into a large dry polypropelene beaker which in turn is placed into the operating ultrasonic cleaner to utilize the vibratory motion using the Alconox treated water as a couplent for increased cavitation.
- 3.3.5 Remove the cover to the sieve assembly and add the contents of the small beaker containing the impacted specimen and residue of the Freon TF on the 149u sieve. Particles adhering to the beaker are carefully washed into the sieve with additional Freon TF and the cover is replaced on the sieve stack.

NOTE: Approximately 30 ml of Freon TF is used in washing the foil envelope and beaker in an attempt to secure all particles.

- 3.3.6 Continue the ultrasonic operation for five minutes after which the empty beaker with the sieve stack is removed and the ultrasonic unit shut down.
- 3.3.7 The complete sieve assembly is removed from the plastic beaker and the assembly is dried for 45 minutes in an oven maintained at 80°C along with the foil used to enclose the sample. Upon removal from the oven, the bottom pan is checked for the complete evaporation of the Freon TF. After 5 minutes in air for cooling, the assembly and foil is placed into a desiccator for one hour.

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- 3.3.8 The sieve stack assembly is removed from the desiccator and the 5 component parts of the set are individually weighed. The foil envelope in which the sample was impacted is also weighed, with all weights being recorded on the Data Sheet.
- 3.3.9 The pre and post impact weights are compared, with the increase in weight of each assembly component and the foil envelope recorded on the Data Sheet, indicating the amount of fines attributable to each component screen size. An increase in the weight of the foil envelope is considered to be an indication of fines < 10 microns. The percentage of fines less than 10 microns, 10 to 44 microns, 44 to 149 microns, and > 149 microns is then determined on the basis of the original weight of the sample as follows:

weight fraction fines $(10-44\mu)$ x 100 = % fines $(10-44\mu)$ total weight original sample

3.3.10 After the determination of the weights of the assembly components and the foil envelope, the contents of the 3 sieves and the pan are placed into a plastic bag, identified and sealed. The foil envelope is discarded.

4. QUALITY ASSURANCE

- 4.1 Review of data by laboratory supervision shall be sufficient for the existing programs.
- 4.2 Calibration of the Analytical Balance shall be maintained in the Quality Assurance established frequency.
- 5. PREPARATION FOR DELIVERY

Not Applicable

6. NOTES

Not applicable

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