OAK RIDGE NATIONAL LABORATORY

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ORNL/TM-2011/109

Data Compilation for AGR-3/4 Designed-to-Fail (DTF) Fuel Particle Batch LEU03-07DTF

Andrew K. Kercher, Brian C. Jolly, Fred C. Montgomery, G. W. Chinthaka M. Silva, and John D. Hunn

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United States Department of Energy -
Office of Nuclear Energy
under the
Next Generation Nuclear Plant –
Advanced Gas Reactor Fuel Development Program

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Oak Ridge National Laboratory

This document is a compilation of coating and characterization data for the designed-to-fail (DTF) fuel particle batch LEU03-07DTF. LEU03-07DTF was fabricated for insertion into fuel compacts designed for the Advanced Gas Reactor Fuel Development and Qualification (AGR) program's third irradiation test (AGR-3/4). This document also includes coating and characterization data for the associated qualification DTF fuel particle batch, LEU12-03DTF. LEU12-03DTF was fabricated using the same coating parameters as LEU03-07DTF, as a final check on the processing conditions prior to coating the LEU03-07DTF fuel batch.

The DTF coating is a high density, high anisotropy pyrocarbon coating of nominal 20 μm thickness that is deposited directly on the kernel. This coating is designed to fail early in the AGR-3/4 irradiation test, resulting in a controlled release of fission products which can be analyzed to provide data on fission product transport. In the AGR-3/4 irradiation experiment, twenty DTF particles will be included along the centerline of each 12.3 mm diameter, 12.5 mm long cylindrical compact. The DTF will be surrounded by standard tristructural isotropic (TRISO) "driver fuel" particles. The driver fuel particles were fabricated in 2007 and coating and characterization data can be found in ORNL/TM-2007/019, "Data Compilation for AGR-3/4 Driver Fuel Coated Particle Composite LEU03-09T." Concentric cylinders of matrix and graphite will surround the compact. During irradiation, fission products will migrate out through the compact into these surrounding matrix and graphite rings.

Both the qualification batch, LEU12-03DTF, and the final AGR-3/4 DTF material, LEU03-07DTF, were inspected and determined to meet the product specifications for DTF particles in section 3 of the AGR-3/4 DTF Fuel and Capsule Component Material Specifications (SPC-1214, Rev. 0). Table 1 provides a summary of key properties of the DTF layer for both the qualification and the final AGR-3/4 DTF material.

Table 1. Summary of DTF coating properties in comparison to product specifications.

Specified Parameters		LEU12-03DTF (qualification run)	LEU03-07DTF (AGR-3/4 DTF fuel)
Mean coating thickness (μm)	20 ± 5	20.1	20.0
Mean sink/float coating density (g/cc)	1.95 ± 0.05	1.961	1.988
Mean anisotropy (true BAFo)	≥ 1.151	1.183	1.243
Mean anisotropy (diattenuation)	≥ 0.070	0.084	0.108

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1 Fabrication of DTF to replace LEU04-02DTF

The LEU03-07DTF particle batch was fabricated as a replacement for a previously fabricated batch of DTF particles, LEU04-02DTF. Coating and characterization data for this previous batch can be found in ORNL/TM-2008/193, "Data Compilation for AGR-3/4 Designed-to-Fail (DTF) Fuel Particle Batch LEU04-02DTF." LEU04-02DTF was fabricated using 425 μm diameter 14% enriched uranium oxide/uranium carbide (UCO) kernels and passed all criteria of the then-applicable AGR-3 & 4 Fuel Product Specification (EDF-6638, Rev. 1), except for an approved deviation in the coating gas fraction used to fabricate the coating. Subsequently, it was recommended by the AGR Technical Coordination Team (TCT) that DTF particles be fabricated using higher enrichment UCO kernels (19.78% U-235). In addition, improvements were made in the fabrication process for the LEU03-07DTF to address observations of anomalies in the LEU04-02DTF pyrocarbon layer and indications that the DTF coatings may not survive the 1800°C heat-treatment in the final step of the compact fabrication process.

Previous studies at ORNL have shown that UCO kernels coated with only buffer and a standard TRISO inner pyrocarbon layer (no SiC) exhibit uranium dispersion through the pyrocarbon layers when heated for 1 hour at 1800°C to simulate the standard heat-treatment process used in the fabrication of AGR compacts. These prior observations prompted a test of the LEU04-02DTF particles, where particles were heated for 1 hour at 1800°C followed by visual inspection and nitric acid leaching. The acid leaching showed around 20% of the total uranium was no longer contained by the DTF layer after heat-treatment, compared to about 0.1% uranium exposure in as-deposited particles. The particles were examined after leaching and many particles had cracks or holes in them (Figure 1 and Figure 2).

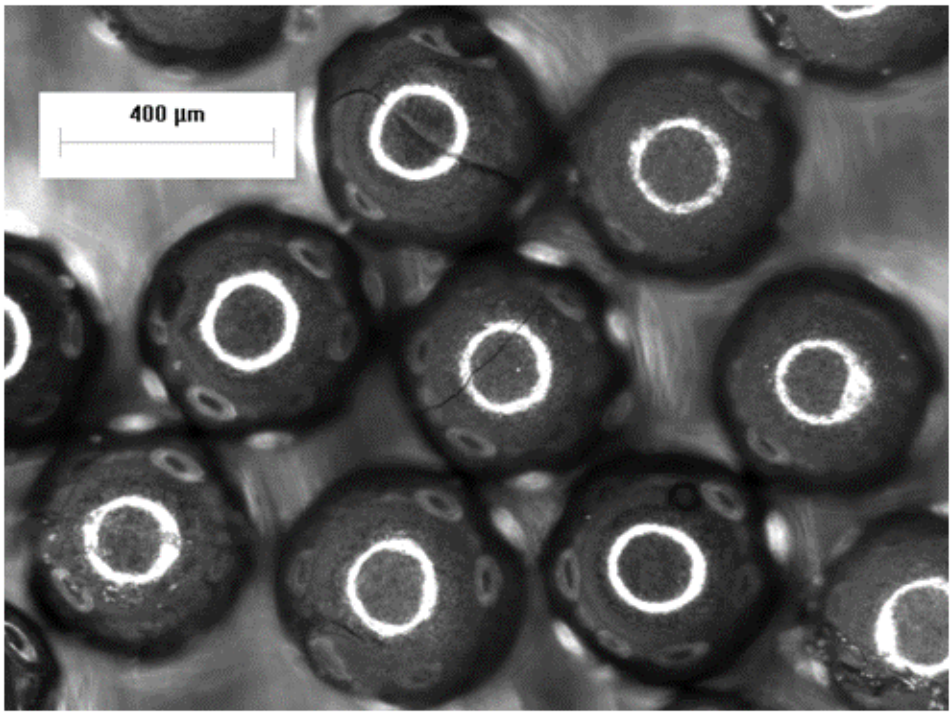

Figure 1: Cracked particles were found in heat-treated LEU04-02DTF after leaching. White circles in image are reflection of stereoscope ring light.

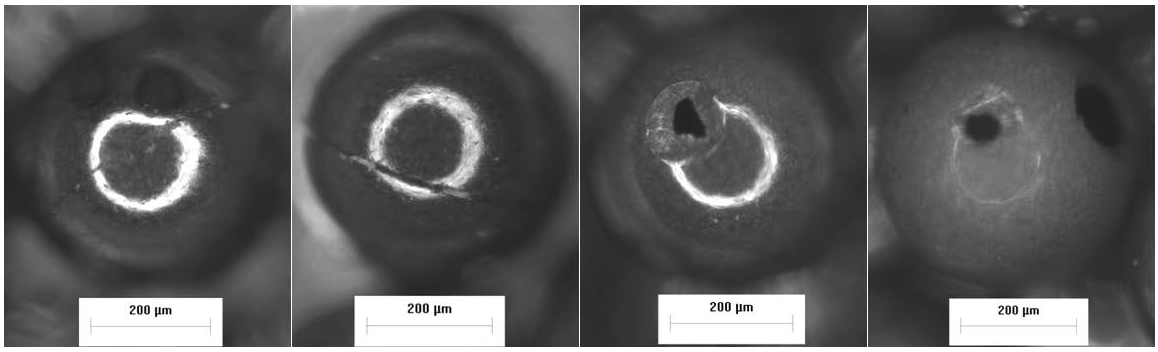

Figure 2: Cracks and holes in the DTF pyrocarbon could be found in stereoscope views of individual LEU04-02DTF particles after leaching. White circles in image are reflection of stereoscope ring light.

It was hypothesized that the failure of the LEU04-02DTF particles to contain the uranium after heat-treatment may have been related to the very high anisotropy of the DTF layer (average diattenuation of 0.1426), reaction of the layer with uranium released from the kernel by conversion of UC to $UC_{1.86}$ during the heat-treatment, or several microstructural anomalies observed in the LEU04-02DTF coating. Microstructural anomalies observed in the LEU04-02DTF coating were reported in ORNL/TM-2008/193. A gap was observed between the kernel and the coating. This had some impact on the image analysis for coating thickness and on the measurement of open porosity. In addition, what appeared to be low density soot inclusions were observed in the DTF layer. Two distinct types of these soot inclusions were observed. Thin soot inclusions were seen on about 85% of the population. More severe, thicker inclusions were observed on a smaller number of particles, about 5% of the population.

Figure 3 shows a typical cross-sectioned particle from LEU04-02DTF. A gap between the kernel and DTF layer of 1-2 µm was observed on all the cross-sectioned particles. Compression of the DTF to close the gap during measurement in the mercury porosimeter made it impossible to determine the open porosity of the layer. This was also discussed in the ORNL/TM-2008/193 report. Figure 3 also shows a thin soot inclusion within the DTF layer, which was observed on most of the particles. This appears as a dark band close to the kernel/coating interface and indicates an interruption in the pyrocarbon coating.

Thicker inclusions were observed on several particles from the LEU04-02DTF batch (Figure 4). A sample of GA archive DTF particles (batch 8662-133) also showed soot inclusions in the DTF layer, many of them much more severe than those observed in the ORNL particles (Figure 5). Therefore, it is not surprising that these anomalies, which are related to fluidization problems, were observed in the LEU04-02DTF particles, which were intentionally coated using conditions close to those used by GA. In both cases, helium was used as the primary fluidization gas. For AGR TRISO materials, pyrocarbon layers are deposited with a primary fluidization gas of argon. Helium and argon behave very differently as fluidization gases. For the single nozzle, 50 mm diameter ORNL coater, the argon fluidization gas can produce a bed with particles usually confined to a limited height range in the coating chamber. A helium fluidization gas in the same coater produces large violently exploding bubbles which often result in some particles dropping out of the fluidization stream while others bounce off the upper walls and roof of the coating chamber.

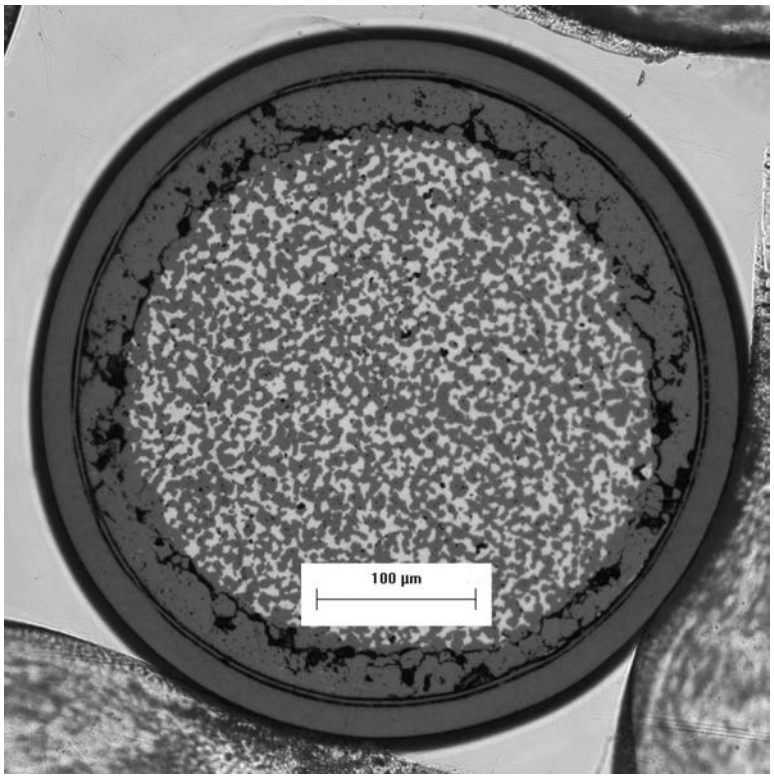

Figure 3. Cross-section of a typical LEU04-02DTF particle.

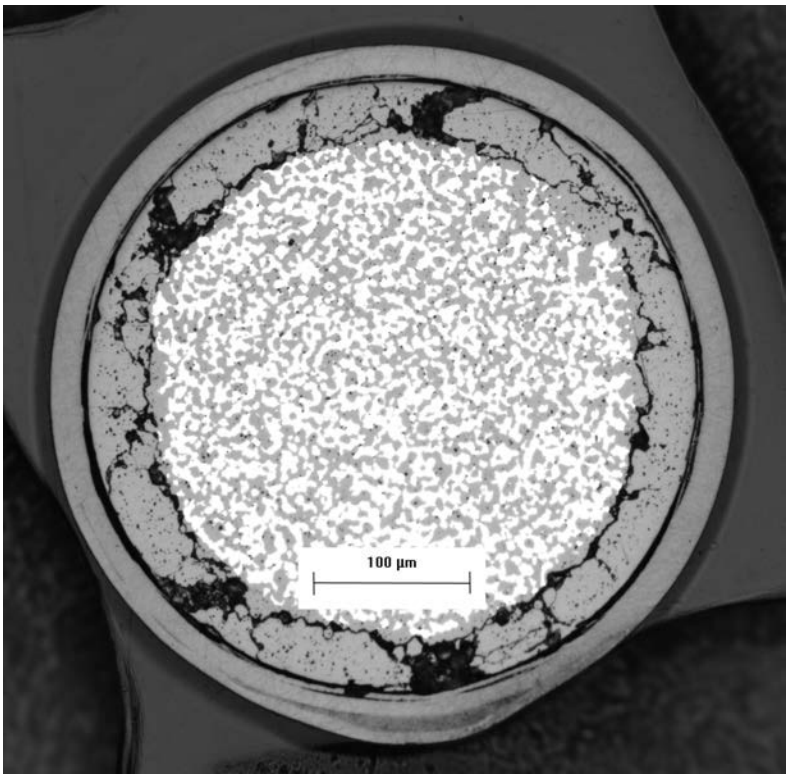

Figure 4. LEU04-02DTF particle cross section showing a thick inclusion in the DTF layer.

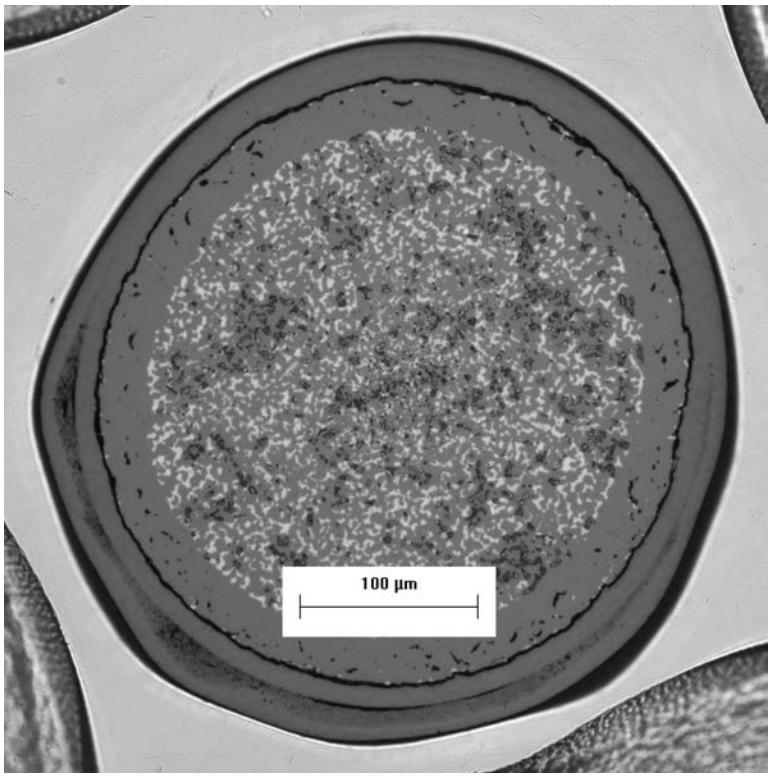

Figure 5. GA DTF with a thick soot inclusion.

A study was conducted to investigate the impact of changes to the coating process conditions on the observed defects and uranium retention after heat-treatment. The fluidization gas was changed from pure helium to either pure argon or a mixture of argon and helium. This improved the particle fluidization, eliminating the thin soot inclusions and reducing both the population and severity of thick soot inclusions (Table 2). Argon was ultimately chosen as the fluidization medium and temperature, coating gas fraction, and total gas flow were adjusted to produce the desired density, anisotropy, and coating rate.

Table 2. Reduction of soot inclusions by change from He to Ar fluidization

	Number of particles analyzed	Percentage of thin soot inclusions	Percentage of thick soot inclusions
GA 8662-133 (He fluidization)	80	0%	16%
LEU04-02DTF (He fluidization)	240	85%	5%
LEU12-03DTF (Ar fluidization)	240	0%	3%
LEU03-07DTF (Ar fluidization)	240	0%	0.8%

Introduction of a thin buffer layer between the kernel and DTF layer was also investigated. The buffer provided a compliant layer to allow for thermal expansion mismatch and provided sacrificial carbon for reaction with uranium freed by the conversion of UC to $UC_{1.86}$. However, buffer was not used in the final process for LEU03-07DTF because of the unknown effect it might have on delaying coating failure during irradiation.

The coating conditions used to produce LEU03-07DTF also resulted in a reduced anisotropy in the DTF layer, compared to LEU04-02DTF (Table 3). The pyrocarbon anisotropy in the DTF layer is higher than a typical TRISO pyrocarbon layer by design, in order to increase the likelihood of failure early in the irradiation. However, it is possible that the very high anisotropy in the LEU04-02DTF pyrocarbon layer contributed to the observed cracking after heat-treatment (Figure 1).

Table 3. Reduction in pyrocarbon anisotropy with modified coating conditions

	Description	Diattenuation	True BAFo
GA 8662-133	Archive GA DTF particles	0.0835	1.1822
LEU04-02DTF	Rejected AGR-3/4 DTF particles	0.1426	1.3328
LEU12-03DTF	Qualification DTF particles	0.0836	1.1825
LEU03-07DTF	New AGR-3/4 DTF particles	0.1084	1.2432

Uranium retention was dramatically improved using the revised coating process. The exposed uranium was reduced from ~20% after heat-treatment to ~2%. This level of coating failure corresponds to less than one particle per compact. Results of heat-treatment tests on particles from the LEU03-07DTF and LEU12-03DTF are discussed in section 8.

2 Material Flow

Table 4 and Table 5 identify the material used to produce the two DTF particle batches. A characterization summary for the LEU03 kernels used for the AGR-3/4 DTF can be found in ORNL/TM-2006/552, "Results from ORNL Characterization of Nominal 350 μm LEUCO Kernels (LEU03) from the BWXT G73V-20-69303 Composite." These kernels were also used for the fabrication of the AGR-3/4 driver fuel. The LEU02 kernels used for the qualification DTF came from the same original kernel composite, G73D-20-69302, as the LEU01 kernels used to produce TRISO particles for the AGR-1 irradiation capsule. Characterization of samples taken from the LEU01 kernel composite can be found in ORNL/TM-2005/517, "Results from ORNL Characterization of Nominal 350 μm LEUCO kernels from the BWXT G73D-20-69302 Composite." Due to a low supply of available LEU03 kernels, LEU02 kernels were upgraded by hand-tabling and renamed LEU12 kernels for use in the final stages of AGR-3/4 DTF development.

Table 4. Material flow for qualification DTF fuel particle batch LEU12-03DTF

Sample ID	Parent Material	Notes
NP-B6700	G73D-20-69302	19.74% U-235 enriched UCO kernel composite shipped from B&W to ORNL 3/23/06
LEU02	NP-B6700	Kernels renamed LEU02
LEU02-63K LEU02-64K LEU02-65K	LEU02	Riffled samples from LEU02
LEU12	LEU02-63K LEU02-64K LEU02-65K	Kernels upgraded by hand tabling to remove 0.72 g of debris and irregularly shaped kernels, recomposited and named LEU12
LEU12-03K	LEU12	Riffled coating charge from LEU12 kernels
LEU12-03DTF	LEU12-03K	DTF coated particles

Table 5. Material flow for AGR-3/4 DTF fuel particle batch LEU03-07DTF

Sample ID	Parent Material	Notes
BP-388475	G73V-20-69303	19.78% U-235 enriched UCO kernel composite shipped from B&W to ORNL 7/13/06
LEU03	BP-388475	Kernels upgraded by hand tabling to remove 0.37 g of debris and irregularly shaped kernels and renamed LEU03
LEU03-07K	LEU03	Riffled coating charge from LEU03 kernels
LEU03-07DTF	LEU03-07K	DTF coated particles

3 Fabrication of LEU12-03DTF and LEU03-07DTF

A designed-to-fail pyrocarbon coating was deposited on nominally 350 μm diameter UCO kernels according to AGR-COAT-SOP-03 Rev. 0, "Standard Operating Procedure: Fluidized Bed Chemical Vapor Deposition System." A series of coating runs were performed to determine the appropriate process conditions. Once the desired layer properties were achieved, a final coating process qualification batch, LEU12-03DTF, was fabricated as a final check prior to coating the LEU03-07DTF fuel batch. Table 6 gives a summary of the process conditions. The LEU12-03DTF qualification run was also used to allow for a quality assurance surveillance of the procedure and supporting infrastructure.

Table 6. Summary of DTF coating conditions for LEU12-03DTF and LEU03-07DTF in comparison to conditions recommended in SPC-1214 Rev. 0.

Parameter	Recommendation	LEU12-03DTF	LEU03-07DTF
Coating gas	Propylene	Propylene	
Fluidization gas	Helium or argon	Argon	
Coating Gas Fraction	0.015 ± 0.0015	0.0160	
Average coating rate	~0.19 μm/min	0.142 μm/min	0.141 μm/min
Coating temperature	1285°C ± 25°C	1200°C	
Total Gas Flow	Not specified	8.13 L/min	
Coating time	Not specified	142 min	

The coating temperature and average coating rate did not lie within the recommended processing window for DTF particles listed in section 3.2 of SPC-1214. The DTF recommended coating parameters of SPC-1214 were based on the conditions used by General Atomics (GA) to produce DTF particles using a helium fluidization gas (batch # 6450-00-0100). The coating parameters used to make LEU03-07DTF were based on process development for an argon fluidization gas in the ORNL 50-mm coater, where the goal was a DTF layer with the specified properties and optimized microstructure.

Both LEU03-07DTF and LEU12-03DTF particles were sorted on an inclined table to remove debris and severely aspherical particles according to AGR-TABLE-SOP Rev. 2, "Standard operating procedure: Tabling to remove highly aspherical particles." The same tabling conditions were used for both particle batches. Only one feeder trough was used with a trough angle of 0.6° and a vibration amplitude of 2.0. The tabling plate had horizontal and vertical angles of 1.5° and a vibration amplitude of 4.0. All but the contents of the collection bin with the most spherical particles were discarded.

The coating run summary sheets, gas certification sheets, and tabler worksheets for LEU12-03DTF and LEU03-07DTF are provided at the end of this section.

Appendix G: Run Summary

Run Number	LEU12-03DTF	
Description	DTF fuel LEU kernels 350 µm	
Particle batch description	LEU kernels 350 µm Ø - See pg	
Particle initial batch wt. (g)	64.2998	
Coated particle batch wt. (g)	66.5195g	
	Target Parameters	As-Processes
Buffer		
Coating gases		
TGF		
CGF		
Temperature (°C)		
Time (min)		
IPyC		
Coating gases	C3H6	C_3H_6
TGF sccm	8130	8130
CGF	.016	.016
CGR	N/A	N/A
Temperature (°C)	1200	1200
Time (min)	142	142
SiC		
Coating gases		
TGF		
CGF		
Temperature (°C)		
Time (min)		
OPyC		
Coating gases		
TGF		
CGF		
CGR		
Temperature (°C)		
Time (min)		
Comments	DTF run - Only Protocarbon layer made	
Operator/Date	Bill Jay 9-23-10	
Verify/Date	Jason Miller 11-1-10	

Appendix D: Precursor Summary

Run Number	LEU12-03DTF		
Gas	**Cylinder number (stamped on cylinder)**	**Tracking number (bar code number)**	**Batch analysis or material cert. (logbook and page number)**
Argon	TX 34636	017334677	~~Pg 140 Coating log Vol 6~~ Invalid BCJ 4-20-11 See note
Argon	TX 18764	036145108	Pg-11 Coating log Vol 7
Propylene	M9057	APR000D54CA	Pg 16 of Coating Log Vol. 6
Comments			
Operator/Date	Bin Jolly	9-23-10	
Verify/date	Jeremy F. Mulloy	11-1-10	

Ar bottle TX 34636 filled with same batch # as TX 18764 – 286OAK0395A

USE same gas cert as TX 18764

Bin Jolly 4-20-11

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2B-ALPCYL-OPS-0028-F
Rev. 0
Effective Date:06/15/2008

CERTIFICATE OF ANALYSIS

3A-3365
SN 036145108

Certification Of Cylinder #: TX18764

Product: Argon **Grade: Ultra High Purity**

Customer:		**Batch/Lot #:**	236OAK0395A
Date of Certification:	8/27/10	**Item Number:**	0014-1400
P.O. Number:		**Valve:**	580
Document Number:		**Cylinder Size:**	49

ANALYSIS REPORT

Major Component	Specification	Purity
Argon Ultra High Purity	99.9990%	>99.9990%

Impurities	Specification	Actual Analysis
Moisture	<3 ppm	0.2 ppm
Oxygen	<2 ppm	1.0 ppm
Total Hydrocarbons	<0.5 ppm	0.1 ppm

Notes:

Certified By: *Jamie Gilmore*
Jamie Gilmore

Air Liquide America Specialty Gases LLC
1001 Alvin Weinberg Dr. Oakridge, TN 37830-8012
Phone: 865-482-7046

MANDATORY DOCUMENT

9-29-10

AIR LIQUIDE™

CERTIFICATE OF ANALYSIS

Customer : Ala Cyl Oak Ridge
P.O. Number :
Document # : 18469552-1C
Mix/Lot # : LPX130325
Item Number : FLAMAL 57
Valid Until : 6 December, 2007

Specification : CERTIFIED
Phase : GAS
Cyl. Size : 55 Valve : CGA 510
Pressure : 240 psia
Volume : 31 SCF

Cylinder Number: **M9057**

Component	Requested Concentrations MOLE	Actual Concentration MOLE	% Analytical Uncertainty	Equipment Used		
				Scale	Analyt. Inst.	Calibration Standard
PROPYLENE		99.61 %				
IMPURITIES:						
PROPANE		0.385 %			3028	1631

This mixture was certified by analysis using one or more calibration standards prepared with scales certified against weights traceable to N.I.S.T.

Comments:

Improper storage or use may affect the accuracy of this standard.
Dewpoint calculated to 40 degrees F, unless otherwise stated.

Certified by _____ Date: 6-Dec-2005

11426 Fairmont Pkwy -- LaPorte, TX 77571
Phone (281) 474-8400 **Fax** (281) 474-8419 **USA** (800) 248-1427
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103 | PROJECT NAME _____ NOTEBOOK NO. _____

Surface Processing and Mechanics Group
Materials Science & Technology Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

AGR-TABLE-SOP-01 Rev. 2
Issue Date 09/08/08
Expire Date 09/08/11

Standard Operating Procedure
Tabling to Remove Highly Aspherical Particles

APPENDIX C: Tabler Worksheet

Particle Batch Identification:	LEU12-03DTF		
Batch Weight:	66.5201 g		
Equipment			
Inclinometer:	PRO360	Verification:	by user

Process Variables

Variable	Setting	Confirmed	Date
Plate Horizontal Angle	1.5°	AKK	9/27/10
Plate Vertical Angle	1.5°	AKK	9/27/10
Plate Vibration Amplitude	4.0	AKK	9/27/10
Feeder #1 Trough Angle	0.6°	AKK	9/27/10
Feeder #1 Steady-State Vibration Amplitude	~~1.5~~ 2.0 AKK 9/27/10	AKK	9/27/10
Feeder #2 Trough Angle	N/A	One feeder used.	
Feeder #2 Steady-State Vibration Amplitude	N/A		

Results – Bins from left to right

Bin #1 (g)	Bin#2 (g)	Bin#3 (g)	Total (g) AKK 9/27/10	Loss (g)
0.1018g	3.2888g	61.2873g	~~64.2873g~~ 64.6779g	1.8422g AKK 10/14/10

Comments/Notes:
Particles were lost into the containment tray (1.6486g), and the table, and the floor.

Operator:	Andrew K. Kercher	Date:	9/27/10
Verified by:	James H. Miller	Date:	11-1-10
QAS:	M. C. [illegible]	Date:	4/21/11

SIGNATURE Andrew K. Kercher DATE 9/27 20 10
READ AND UNDERSTOOD [illegible] DATE 11-10 20 10

Appendix G: Run Summary

Run Number	LEU03-07DTF	
Description	DTF run - pyrocarbon only	
Particle batch description	LEU03-07K 350 μm kernels	
Particle initial batch wt. (g)	65.8019	
Coated particle batch wt. (g)	68.8670	
	Target Parameters	As-Processes
Buffer		
Coating gases		
TGF		
CGF		
Temperature (°C)		
Time (min)		
IPyC		
Coating gases	C3H6	C_3H_6
TGF	8130	8130
CGF	.016	.016
CGR	N/A	—
Temperature (°C)	1200	1200
Time (min)	142	142
SiC		
Coating gases		
TGF		
CGF		
Temperature (°C)		
Time (min)		
OPyC		
Coating gases		
TGF		
CGF		
CGR		
Temperature (°C)		
Time (min)		
Comments	DTF coating run - pyrocarbon only	
Operator/Date	Ben [signature] 10-13-10	
Verify/Date	Jeremy Miller 11-1-10	

24

Appendix D: Precursor Summary

Run Number	LEU03-07DTF		
Gas	**Cylinder number (stamped on cylinder)**	**Tracking number (bar code number)**	**Batch analysis or material cert. (logbook and page number)**
Argon	H1007677	011695503	pg 21 vol 7
Argon	TX18764	036145108	pg 11 vol 7
Propylene	M9057	APR000D54CA	Pg 16 of Coating Log Vol. 6
Comments			
Operator/Date	[signature]		10-13-10
Verify/date	[signature]		11-1-10

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CERTIFICATES FOR PURE SPECIALTY GASES

2B-ALPCYL-OPS-0028-F
Rev. 0
Effective Date:06/15/2008

21

CERTIFICATE OF ANALYSIS

Certification Of Cylinder #: **H1007677**

Product: Argon **Grade: Ultra High Purity**

Customer:	UT Battelle	**Batch/Lot #:**	215OAK0395A
Date of Certification:	10/7/10	**Item Number:**	0013-1300
P.O. Number:	4800636980	**Valve:**	580
Document Number:	39220283	**Cylinder Size:**	44

ANALYSIS REPORT

Major Component	Specification	Purity
Argon Ultra High Purity	99.9990%	>99.9990%

Impurities	Specification	Actual Analysis
Moisture	<3 ppm	0.2 ppm
Oxygen	<2 ppm	1.2 ppm
Total Hydrocarbons	<0.5 ppm	0.1 ppm

Notes:

Certified By: *Jamie Gilmore*
Jamie Gilmore

Air Liquide America Specialty Gases LLC
1001 Alvin Weinberg Dr. Oakridge, TN 37830-8012
Phone: 865-482-7046

MANDATORY DOCUMENT

10-13-10

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Rev. 0
Effective Date:06/15/2008

CERTIFICATE OF ANALYSIS

3A-3365
SN 036145108

Certification Of Cylinder #: TX18764

Product: Argon **Grade: Ultra High Purity**

Customer:		**Batch/Lot #:**	236OAK0395A
Date of Certification:	8/27/10	**Item Number:**	0014-1400
P.O. Number:		**Valve:**	580
Document Number:		**Cylinder Size:**	49

ANALYSIS REPORT

Major Component	Specification	Purity
Argon Ultra High Purity	99.9990%	>99.9990%

Impurities	Specification	Actual Analysis
Moisture	<3 ppm	0.2 ppm
Oxygen	<2 ppm	1.0 ppm
Total Hydrocarbons	<0.5 ppm	0.1 ppm

Notes:

Certified By: *Jamie Gilmore*
Jamie Gilmore

Air Liquide America Specialty Gases LLC
1001 Alvin Weinberg Dr. Oakridge, TN 37830-8012
Phone: 865-482-7046

MANDATORY DOCUMENT

9-23-10

AIR LIQUIDE™

CERTIFICATE OF ANALYSIS

Customer : Ala Cyl Oak Ridge
P.O. Number :
Document # : 18469552-1C
Mix/Lot # : LPX130325
Item Number : FLAMAL 57
Valid Until : 6 December, 2007

Specification : CERTIFIED
Phase : GAS
Cyl. Size : 55 Valve : CGA 510
Pressure : 240 psia
Volume : 31 SCF

Cylinder Number: **M9057**

Component	Requested Concentrations MOLE	Actual Concentration MOLE	% Analytical Uncertainty	Equipment Used		
				Scale	Analyt. Inst.	Calibration Standard
PROPYLENE		99.61 %				
IMPURITIES:						
PROPANE		0.385 %			3028	1631

This mixture was certified by analysis using one or more calibration standards prepared with scales certified against weights traceable to N.I.S.T.

Comments:

Improper storage or use may affect the accuracy of this standard.
Dewpoint calculated to 40 degrees F, unless otherwise stated.

Certified by _____ Date: 6-Dec-2005

11426 Fairmont Pkwy -- LaPorte, TX 77571
Phone (281) 474-8400 **Fax** (281) 474-8419 **USA** (800) 248-1427

ISO: 9001-2000

ORNL/TM-2011/109

Surface Processing and Mechanics Group
Materials Science & Technology Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831

AGR-TABLE-SOP-01 Rev. 2
Issue Date 09/08/08
Expire Date 09/08/11
Page 7 of 7

Standard Operating Procedure
Tabling to Remove Highly Aspherical Particles

APPENDIX C: Tabler Worksheet

Particle Batch Identification:	LEU03-07DTF		
Batch Weight:	68.8672g g		
Equipment			
Inclinometer:	Pro360	Verification:	by user

Process Variables

Variable	Setting	Confirmed	Date
Plate Horizontal Angle	1.5°	AKK	10/14/10
Plate Vertical Angle	1.5°	AKK	10/14/10
Plate Vibration Amplitude	4.0	AKK	10/14/10
Feeder #1 Trough Angle	0.6°	AKK	10/14/10
Feeder #1 Steady-State Vibration Amplitude	2.0°	AKK	10/14/10
Feeder #2 Trough Angle	N/A	One feeder used	
Feeder #2 Steady-State Vibration Amplitude	N/A		

Results – Bins from left to right

Bin #1 (g)	Bin#2 (g)	Bin#3 (g)	Total (g)	Loss (g)
0.2169g	3.2661g	65.1100g	~~68.593g~~ 10/15/10 AKK 68.5930g ~~0.2742g~~	0.2742g

Comments/Notes: My previous DTF experience (p. 103 9/27/10) had a major loss problem. I did 2 fixes: #1 there is an Al foil wall preventing particles from going uphill and falling off the tabler and #2 I put foil enclosures between the bins and the funnels. #2 fix seems most important.

Operator:	Kathleen K. Kvarda	Date:	October 15, 2010
Verified by:	Jeanette Miller	Date:	11-1-10
QAS:	M. C. Lee	Date:	4/21/11

SIGNATURE Kathleen K. Kvarda October 15, 2010
READ AND UNDERSTOOD Neal Bowden DATE 11-1-10 2010

4 Characterization of LEU12 Kernels

This section contains data on the kernel composite used for LEU12-03DTF. The data was obtained according to product inspection plan AGR-CHAR-PIP-01R1. This was only a partial analysis of the kernel composite. Additional characterization of the kernel composite is provided in the BWXT data package for the kernel composite G73D-20-69302.

Because the supply of LEU03 kernels was limited, LEU12 kernels were used for development of the DTF coating process. The LEU12 kernels came from the same original kernel composite, G73D-20-69302, as the LEU01 kernels used to produce TRISO particles for the AGR-1 irradiation capsule. Characterization of samples taken from the LEU01 kernel composite can be found in ORNL/TM-2005/517, "Results from ORNL Characterization of Nominal 350 μm LEUCO Kernels from the BWXT G73D-20-69302 Composite." The LEU12 kernels were originally labeled as LEU02 and were shipped to ORNL about one year after the LEU01 kernels were shipped. The LEU02 kernels appeared to have degraded over that one year period due to exposure to air and were not used for any irradiation test articles. LEU02 kernels were upgraded by hand-tabling and renamed LEU12 kernels for use in the final stages of AGR-3/4 DTF development. Additional safety measures were employed when performing characterization methods which exposed LEU12 kernels because of the higher friability.

The following pages show the inspection report form (IRF-01) for the LEU12 kernel composite. Following IRF-01 are the individual data report forms for the measurements that were performed.

Inspection Report Form IRF-01: B&W Kernel Composite G73D-20-69302 (LEU12)

Procedure: AGR-CHAR-PIP-01 Rev. 1

Property	Measured Data: Mean (x)	Measured Data: Std. Dev. (s)	Measured Data: # measured (n)	Measured Data: k or t value	Specification: INL EDF-4380	Acceptance Criteria	Acceptance Test Value	Data Records
Average kernel diameter (μm)	349.9	8.9	3620	1.645	mean 350 ± 10	A = x - ts/√n ≥ 340	349.7	DRF-06 DRF-09
						B = x + ts/√n ≤ 360	350.1	
				2.380	dispersion ≤0.01 < 300 ≤0.01 > 400	C = x - ks > 300	328.7	
						D = x + ks < 400	371.1	
Kernel ellipticity (Dmax/Dmin)	1.019		3620		dispersion ≤0.10 ≥1.05	≤1 in 50 or ≤7 in 142	69	DRF-06 DRF-09
Kernel envelope density (Mg/m^3)	11.181	0.007	4	2.353	mean ≥10.4	A = x - ts/√n ≥ 10.4	11.2	DRF-15 DRF-22

Comments

69 kernels with ellipticity ≥1.05 out of 3620 kernels measured passes the dispersion specification acceptance criteria of ≤332 in 3620.
This composite would pass an ellipticity control limit of ≥1.035 at 10% tolerance limit with a 95% confidence level.
This composite would pass an ellipticity control limit of ≥1.05 at 2.4% tolerance limit with a 95% confidence level.

QC Supervisor — Date: 1-31-11

QA Reviewer — Date: 2/3/11

Data Report Form DRF-06: Imaging of Kernel Diameter and Ellipticity Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-06 Rev. 1
Operator:	Andrew K. Kercher
Sample ID:	LEU12-B01
Sample Description:	B&W kernel composite G73D-20-69302
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P10080901\

DMR Calibration Expiration Date:	11/2/10
Stage Micrometer Calibration Expiration Date:	2/10/14
Measured Value for 760 µm in Stage Micrometer Image:	759.4 µm

Operator

August 9, 2010

Date

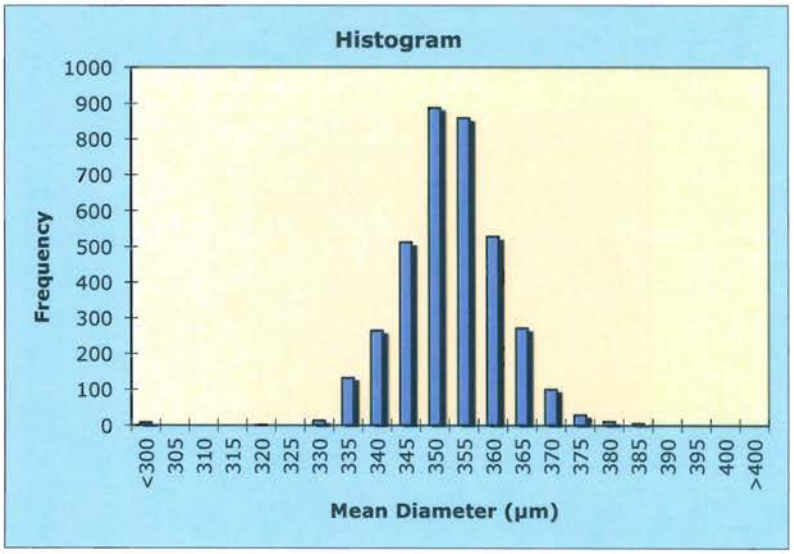
Data Report Form DRF-09A: Measurement of Kernel Diameter

Procedure:	AGR-CHAR-DAM-09 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P10080901\
Sample ID:	LEU12-B01
Sample Description:	B&W kernel composite G73D-20-69302
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P10080901_output\

Number of kernels analyzed:	3620
Mean of the average diameter of each kernel (μm):	349.9
Standard deviation in the average diameter of each kernel (μm):	8.9

Distribution of the average particle diameter (top binned)

Mean Diameter (μm)	*Frequency*
<300	8
305	0
310	0
315	0
320	1
325	0
330	13
335	132
340	264
345	512
350	887
355	859
360	528
365	271
370	100
375	29
380	11
385	5
390	0
395	0
400	0
>400	0



Andrew K. Kercher
Operator

August 11, 2010
Date

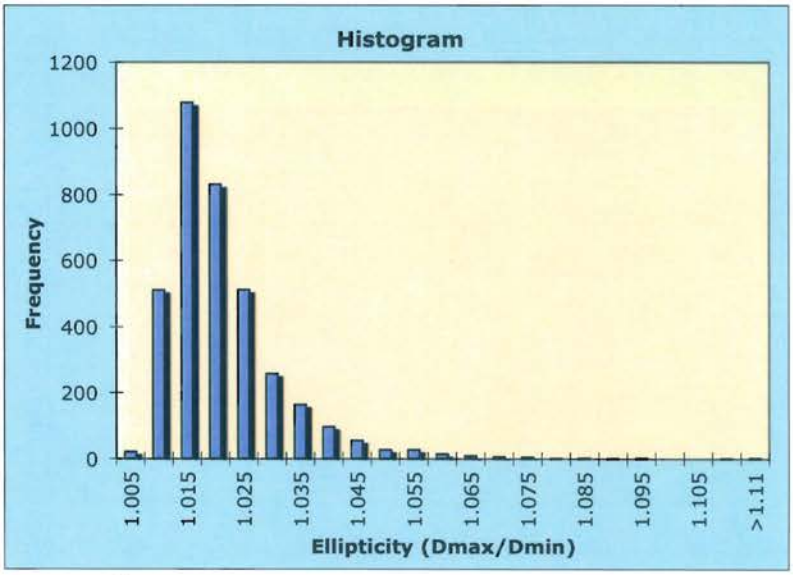
Data Report Form DRF-09B: Measurement of Kernel Ellipticity (Dmax/Dmin)

Procedure:	AGR-CHAR-DAM-09 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P10080901\
Sample ID:	LEU12-B01
Sample Description:	B&W kernel composite G73D-20-69302
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P10080901_output\

Number of kernels analyzed:	3620
Number of kernels with ellipticity > 1.05	69
Average kernel ellipticity:	1.019

Distribution of the ellipticity (top binned)

Ellipticity (D)	*Frequency*
1.005	21
1.010	511
1.015	1078
1.020	831
1.025	512
1.030	257
1.035	163
1.040	96
1.045	55
1.050	27
1.055	27
1.060	14
1.065	8
1.070	6
1.075	5
1.080	1
1.085	2
1.090	1
1.095	2
1.100	0
1.105	0
1.110	1
>1.11	2



Operator

August 11, 2010

Date

Data Report Form DRF-15: Measurement of Average Kernel Envelope Density using a Mercury Porosimeter

Procedure:	AGR-CHAR-DAM-15 Rev. 3
Operator:	C. Silva
Kernel Lot ID:	LEU12-C01, D01, E01, F01
Kernel Lot Description:	B&W kernel composite G73D-20-69302
Thermocouple Expiration Date:	3/25/11
Penetrometer Expiration Date:	7/20/11
Completed DRF Filename:	\\mc-agr\AGR\Porosimeter\S10080501\S10080501_DRF15R3.xls

Mean average weight/kernel (g):	2.42E-04
Standard error in mean average weight/kernel (g):	5.10E-07

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Porosimeter data file number:	S10080501	S10080502	S10080503	S10080504	
Weight of kernels (g):	13.4070	13.3827	13.3207	13.1177	
Approximate number of kernels:	55332	55232	54976	54138	
Uncertainty in number of kernels:	116	116	116	114	
Envelope volume of sample (cc):	1.200	1.196	1.191	1.174	
Average envelope volume/kernel (cc):	2.17E-05	2.17E-05	2.17E-05	2.17E-05	
Sample envelope density (g/cc):	11.174	11.190	11.184	11.178	

Mean average envelope volume/kernel (cc):	2.167E-05
Standard error in mean envelope volume/kernel (cc):	6.4E-09
Mean sample envelope density (g/cc):	11.181
Standard deviation in sample envelope density (g/cc):	0.007

Comments
Sample 5 porosimeter run failed.

G.W. Chinthaka — Operator

08/05/2010 — Date

Data Report Form DRF-22: Estimation of Average Particle Weight

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Dixie Barker
Particle Lot ID:	LEU12
Particle Lot Description:	B&W kernel composite G73D-20-69302
Filename:	\\mc-agr\AGR\ParticleWeight\W10080901_DRF22R1.xls

	Sample 6	Sample 7	Sample 8	Sample 9	Sample 10
Weight of particles (g):	0.0789	0.0576	0.1091	0.0749	0.1087
Number of particles:	327	239	449	309	446
Average weight/particle (g):	2.413E-04	2.410E-04	2.430E-04	2.424E-04	2.437E-04

Mean average weight/particle (g):	2.423E-04
Standard error in mean average weight/particle (g):	5.10E-07

Dixie Barker
Operator

8-9-10
Date

5 Characterization of LEU03 Kernels

This section contains data on the kernel composite used for LEU03-07DTF. The data was obtained according to product inspection plan AGR-CHAR-PIP-01R1. This was only a partial analysis of the kernel composite and was not used for product acceptance. Characterization of the kernel composite for acceptance according to the specific requirements listed in section 3.1 of INL EDF-6638, "AGR-3 & 4 Fuel Product Specification," is documented in the BWXT data package for G73V-20-69303. The requirements of EDF-6638 are identical to those in SPC-1214, "AGR-3/4 DTF Fuel and Capsule Component Material Specifications." The BWXT kernel lot G73V-20-69303 was riffled into sublots for characterization and coating by ORNL and identified as LEU03-## (where ## was a series of integers beginning with 01).

The following pages show the inspection report form (IRF-01). Following IRF-01 are the individual data report forms for the measurements that were performed.

Inspection Report Form IRF-01: BWXT LEUCO Kernel Composite 69303

Procedure: AGR-CHAR-PIP-01 Rev. 1

Property	Measured Data				Specification	Acceptance Criteria	Acceptance Test Value	Data Records
	Mean (x)	Std. Dev. (s)	# measured (n)	k or t value	INL EDF-4380			
Average kernel diameter (μm)	357.3	10.5	3847	1.65	mean 350 ± 10	A = x - ts/√n ≥ 340	357.0	DRF-06 DRF-09
						B = x + ts/√n ≤ 360	357.6	
				2.38	dispersion ≤0.01 < 300 ≤0.01 > 400	C = x - ks > 300	332.3	
						D = x + ks < 400	382.3	
Kernel ellipticity (Dmax/Dmin)	1.016		3847		dispersion ≤0.10 ≥1.05	≤1 in 50 or ≤7 in 142	11	DRF-06 DRF-09
Kernel envelope density (Mg/m³)	11.098	0.025	3	2.920	mean ≥10.4	A = x - ts/√n ≥ 10.4	11.1	DRF-15 DRF-22

Comments

11 kernels with ellipticity ≥1.05 out of 3847 kernels measured passes the dispersion specification acceptance criteria of ≤353 in 3847.
This composite would pass a control limit of ≥1.026 at 10% tolerance limit with 95% confidence level.
This composite would pass a control limit of ≥1.05 at 0.48% tolerance limit with 95% confidence level.

John Hunn — QC Supervisor

2-20-07 — Date

M. C. Fox — QA Reviewer

2/20/07 — Date

Data Report Form DRF-06: Imaging of Kernel Diameter and Ellipticity Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-06 Rev. 1
Operator:	Andrew K. Kercher
Sample ID:	LEU03-01K-B01
Sample Description:	BWXT kernel composite 69303
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P06080902\

DMR Calibration Expiration Date:	9/8/2006
Stage Micrometer Calibration Expiration Date:	2/17/2007
Measured Value for 760 µm in Stage Micrometer Image:	759.4 µm

Andrew K. Kercher — August 9, 2006

Operator — Date

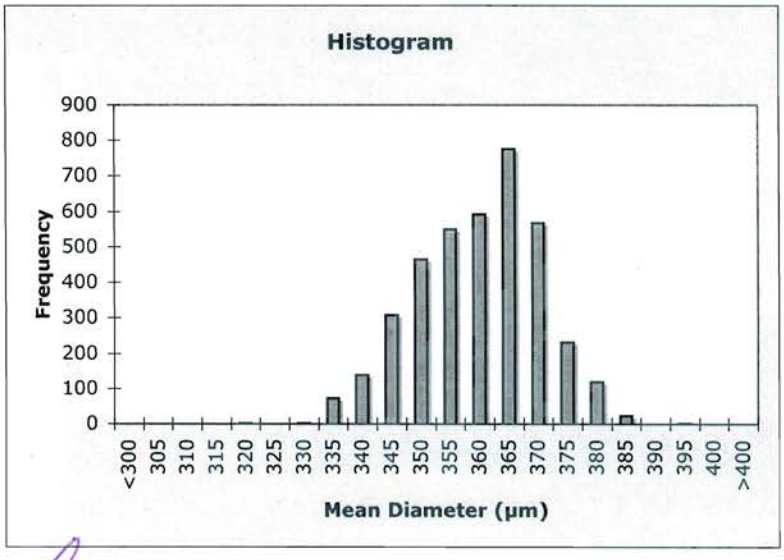
Data Report Form DRF-09A: Measurement of Kernel Diameter

Procedure:	AGR-CHAR-DAM-09 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P06080902\
Sample ID:	LEU03-01K-B01
Sample Description:	BWXT kernel composite 69303
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P06080902_output\

Number of kernels analyzed:	3847
Mean of the average diameter of each kernel (μm):	357.3
Standard deviation in the average diameter of each kernel (μm):	10.5

Distribution of the average particle diameter (top binned)

Mean Diameter (μm)	*Frequency*
<300	0
305	0
310	0
315	0
320	1
325	0
330	2
335	73
340	138
345	308
350	465
355	550
360	592
365	775
370	567
375	232
380	120
385	23
390	0
395	1
400	0
>400	0



Andrew K. Kercher
Operator

August 10, 2006
Date

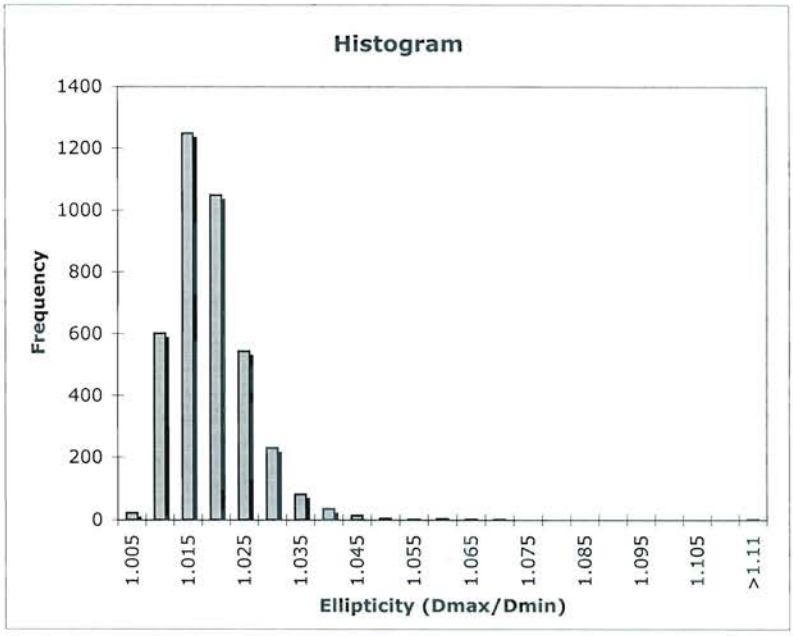
Data Report Form DRF-09B: Measurement of Kernel Ellipticity (Dmax/Dmin)

Procedure:	AGR-CHAR-DAM-09 Rev. 2
Operator:	Andrew K. Kercher
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P06080902\
Sample ID:	LEU03-01K-B01
Sample Description:	BWXT kernel composite 69303
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P06080902_output\

Number of kernels analyzed:	3847
Number of kernels with ellipticity > 1.05	11
Average kernel ellipticity:	1.016

Distribution of the ellipticity (top binned)

Ellipticity (D)	*Frequency*
1.005	23
1.010	602
1.015	1249
1.020	1049
1.025	544
1.030	230
1.035	83
1.040	36
1.045	15
1.050	5
1.055	2
1.060	4
1.065	2
1.070	1
1.075	0
1.080	0
1.085	0
1.090	0
1.095	0
1.100	0
1.105	0
1.110	0
>1.11	2



Operator

August 10, 2006
Date

Data Report Form DRF-15: Measurement of Average Kernel Envelope Density using a Mercury Porosimeter

Procedure:	AGR-CHAR-DAM-15 Rev. 3
Operator:	S. D. Nunn
Kernel Lot ID:	LEU03-01K
Kernel Lot Description:	BWXT kernel composite 69303
Thermocouple Expiration Date:	5/19/07
Penetrometer Expiration Date:	5/25/07
Completed DRF Filename:	\\mc-agr\AGR\Porosimeter\S06081001\S06081001_DRF15R3.xls

Mean average weight/kernel (g):	2.628E-04
Standard error in mean average weight/kernel (g):	8.48E-07

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Porosimeter data file number:	S06081001L	S06081002L	S06081003L		
Weight of kernels (g):	12.7870	12.8001	12.7347		
Approximate number of kernels:	48657	48707	48458		
Uncertainty in number of kernels:	157	157	156		
Envelope volume of sample (cc):	1.1502	1.1563	1.1467		
Average envelope volume/kernel (cc):	2.36E-05	2.37E-05	2.37E-05		
Sample envelope density (g/cc):	11.117	11.070	11.106		

Mean average envelope volume/kernel (cc):	2.368E-05
Standard error in mean envelope volume/kernel (cc):	3.0E-08
Mean sample envelope density (g/cc):	11.098
Standard deviation in sample envelope density (g/cc):	0.025

Comments
Only 3 samples were measured due to the limited amount of kernels available for AGR-3/4 fabrication.

S. D. Nunn
Operator

8/15/06
Date

Data Report Form DRF-22: Estimation of Average Particle Weight

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	John Hunn
Particle Lot ID:	LEU03-01K
Particle Lot Description:	BWXT kernel composite 69303
Filename:	\\mc-agr\AGR\ParticleWeight\W06080901_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	6.15E-02	7.35E-02	6.83E-02	6.50E-02	6.68E-02
Number of particles:	236	279	259	249	252
Average weight/particle (g):	2.61E-04	2.63E-04	2.64E-04	2.61E-04	2.65E-04

Mean average weight/particle (g):	2.628E-04
Standard error in mean average weight/particle (g):	8.48E-07

John Hunn
Operator

8-9-06
Date

Data Report Form DRF-22: Estimation of Average Particle Weight

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Dixie Barker
Particle Lot ID:	LEU03-01K
Particle Lot Description:	BWXT kernel composite 69303
Filename:	\\mc-agr\AGR\ParticleWeight\W06080902_DRF22R1.xls

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
Weight of particles (g):	6.15E-02	7.35E-02	6.83E-02	6.50E-02	6.68E-02
Number of particles:	236	278	259	250	252
Average weight/particle (g):	2.61E-04	2.64E-04	2.64E-04	2.60E-04	2.65E-04

Mean average weight/particle (g):	2.628E-04
Standard error in mean average weight/particle (g):	1.03E-06

Dixie Barker
Operator

8-9-06
Date

6 Characterization of LEU12-03DTF

Product inspection plan AGR-CHAR-PIP-08 Rev. 2 was used to characterize the LEU12-03DTF qualification batch, except for measurement of uranium loading which was unnecessary since this DTF material will not be used in compacts for irradiation. This section contains the associated inspection report form (IRF-08) and the data report forms from that analysis. The LEU12-03DTF particle batch was found to conform to all specifications for DTF particles in section 3.3 of the AGR-3/4 DTF Fuel and Capsule Component Material Specifications (SPC-1214, Rev. 0). Figure 6 shows a polished cross-section of a typical LEU12-03DTF particle.

Characterization samples of LEU12-03DTF were riffled according to the product inspection plan. The measurements of pyrocarbon thickness, density, and optical anisotropy were required for testing conformance to the DTF product specifications in SPC-1214. Additional characterization was also performed for information only. Exposed uranium content was measured by acid leaching as-deposited and heat-treated DTF particles and is discussed in section 8. Open porosity of the DTF layer was measured, but the value may be affected by a slight compression of the DTF layer. The old LEU04-02DTF particles had an obvious gap between the DTF layer and the kernel that resulted in a measurable compression when pressure was applied during analysis with a mercury porosimeter. This effect was explained in detail in ORNL/TM-2008/193, "Data Compilation for AGR-3/4 Designed-to-Fail (DTF) Fuel Particle Batch LEU04-02DTF." There was some indication for the existence of a slight compression during porosimetry in the LEU12-03DTF particles, but it could not be clearly resolved. A small compression component in the porosimetry data would result in a slight over estimate of the open porosity reported on DRF-31.

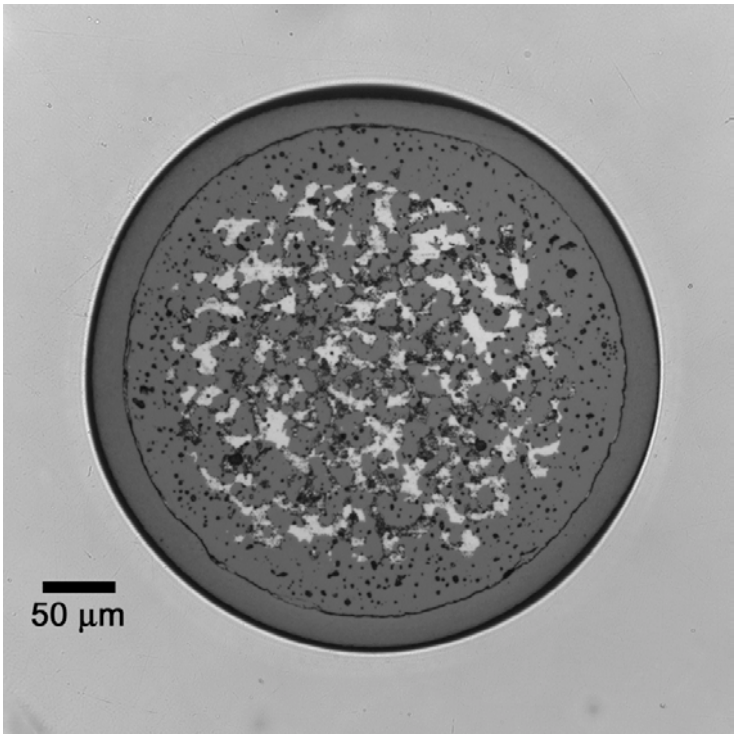


Figure 6. Cross-sectional optical micrograph of a typical LEU12-03DTF particle.

Inspection Report Form IRF-08: AGR-3/4 Designed to Fail Coated Particle Batches

Procedure:	AGR-CHAR-PIP-08 Rev. 2
Coated particle batch ID:	LEU12-03DTF
Coated particle batch description:	DTF Qualifier Run

Property	Measured Data				Specification	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
	Mean (x)	Std. Dev. (s)	Measurements (n)	k or t value	INL SPC-1214 Revision 0				
Average DTF thickness for each particle (μm)	20.1	0.9	199	1.653	mean 20 ± 5	A = x - ts/√n ≥ 15	20.0	pass	DRF-33 DRF-34
						B = x + ts/√n ≤ 25	20.2	pass	
				2.569	dispersion ≤0.01 ≤ 8	C = x - ks > 8	17.8	pass	
DTF sink/float density (Mg/m³)	1.961	0.047	42	1.683	mean 1.95 ± 0.05	A = x - ts/√n ≥ 1.90	1.949	pass	DRF-03
						B = x + ts/√n ≤ 2.00	1.973	pass	
DTF anisotropy (True BAFo)	1.183	0.011	7	1.943	mean ≥ 1.151	A = x - ts/√n ≥ 1.151	1.175	pass	DRF-18
Particle diameter	392.2	7.8	2896	1.645	Not Applicable				DRF-06 DRF-09
Average uranium per particle (g)	-	-	-	-	Not Applicable				DRF-35
Average particle wt (g)	2.618E-04	1.1E-06	5	2.132	Not Applicable				DRF-22
Average particle envelope volume (cm³)	3.17E-05	-	1	-	Not Applicable				DRF-31
DTF open porosity (ml/m²)	0.046	-	1	-	Measurement Only				DRF-31

Comments

The average diameter aspect ratio was measured to be 1.028.

QC Supervisor — 1-31-11 — Date

Accept coated particle batch (Yes or No):	N/A

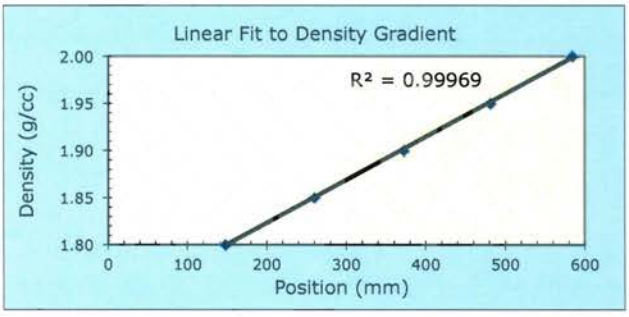
QA Reviewer — 2/3/11 — Date

Data Report Form DRF-03: Measurement of PyC Density using a Density Gradient Column

Procedure:	AGR-CHAR-DAM-03 Rev. 3
Operator:	Dixie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D10092901_DRF03R3.xls
Sample ID:	LEU12-03DTF-C01
Sample description:	DTF qualifier run: 1200C PyC - in Ar - CGF 0.016
Float expiration date:	01/2014
Gauge expiration date:	08/2011
Bath temperature:	23.0 °C

Calibrated Floats			
Density	Top of Float	Bottom of Float	Center of Mass
1.800	141.91	151.52	146.72
1.850	254.41	262.92	258.67
1.900	368.25	374.48	371.37
1.950	476.80	483.34	480.07
2.000	579.94	586.53	583.24

Linear Fit			
slope	StDev	intercept	StDev
4.57E-04	2.68E-06	1.73E+00	9.92E-04



Sample Density								
Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	484.38	1.9531	26	543.37	1.9801	51		
2	492.20	1.9567	27	543.37	1.9801	52		
3	53.41	1.7563	28	551.14	1.9836	53		
4	503.41	1.9618	29	554.42	1.9851	54		
5	504.44	1.9623	30	555.43	1.9856	55		
6	504.73	1.9624	31	560.50	1.9879	56		
7	505.63	1.9629	32	583.39	1.9984	57		
8	506.25	1.9631	33	554.58	1.9852	58		
9	506.51	1.9633	34	543.82	1.9803	59		
10	507.29	1.9636	35	533.50	1.9756	60		
11	507.00	1.9635	36	527.79	1.9730	61		
12	67.00	1.7625	37	526.91	1.9726	62		
13	508.58	1.9642	38	526.88	1.9726	63		
14	514.67	1.9670	39	524.88	1.9716	64		
15	515.62	1.9674	40	518.77	1.9689	65		
16	515.72	1.9675	41	515.51	1.9674	66		
17	514.44	1.9669	42	514.48	1.9669	67		
18	518.14	1.9686	43			68		
19	518.95	1.9689	44			69		
20	523.93	1.9712	45			70		
21	524.86	1.9716	46			71		
22	524.86	1.9716	47			72		
23	527.88	1.9730	48			73		
24	529.73	1.9739	49			74		
25	533.36	1.9755	50			75		

Average density of PyC fragments:	1.9613
Standard deviation in density of PyC fragments:	0.0466
Uncertainty in calculated density of PyC fragments:	0.0019

Dixie Barker
Operator

9-29-10
Date

Data Report Form DRF-06: Imaging of Small Particle Diameter and Ellipticity Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-06 Rev. 1
Operator:	G. W. C. Silva
Sample ID:	LEU12-03DTF-I01
Sample Description:	DTF qualifier run: 1200C PyC - in Ar - CGF 0.016
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P10101801\

DMR Calibration Expiration Date:	11/2/10
Stage Micrometer Calibration Expiration Date:	2/10/14
Measured Value for 760 µm in Stage Micrometer Image:	760. µm

G.W. Chinthaka — Operator

10/18/2010 — Date

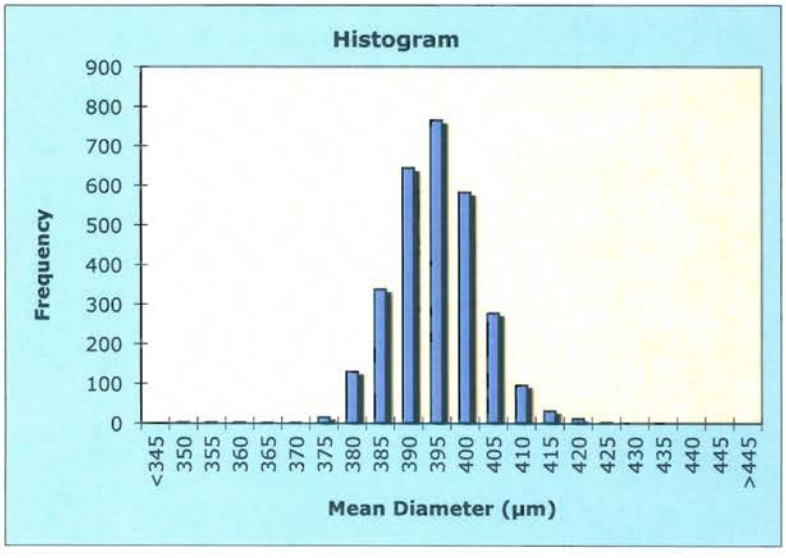
Data Report Form DRF-09A: Measurement of Small Particle Diameter

Procedure:	AGR-CHAR-DAM-09 Rev. 2
Operator:	G. W. C. Silva
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P10101801\
Sample ID:	LEU12-03DTF-I01
Sample Description:	DTF qualifier run: 1200C PyC - in Ar - CGF 0.016
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P10101801_output\

Number of small particles analyzed:	2896
Mean of the average diameter of each small particle (μm):	392.2
Standard deviation in the average diameter of each small particle (μm):	7.8

Distribution of the average particle diameter (top binned)

Mean Diameter (μm)	*Frequency*
<345	0
350	2
355	2
360	2
365	1
370	1
375	15
380	129
385	337
390	644
395	765
400	582
405	277
410	95
415	31
420	11
425	2
430	0
435	0
440	0
445	0
>445	0



G. W. C. Silva
Operator

10/19/2010
Date

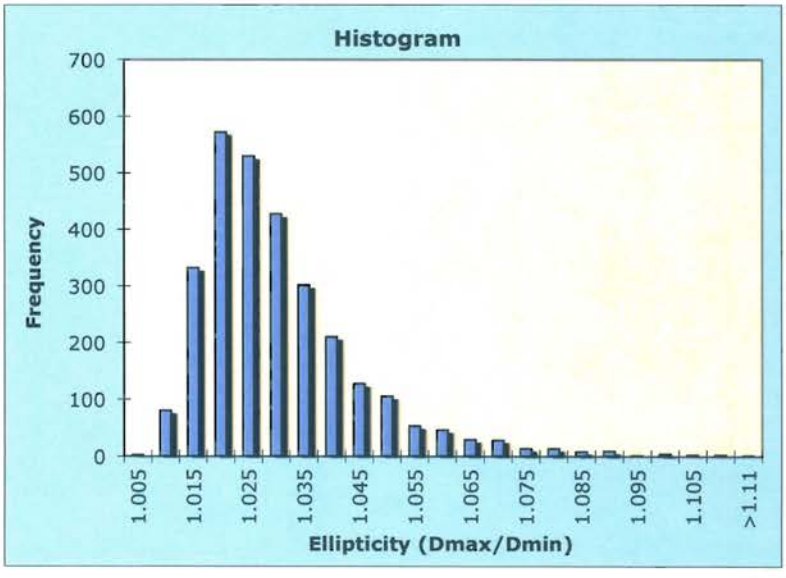
Data Report Form DRF-09B: Measurement of Small Particle Ellipticity (Dmax/Dmin)

Procedure:	AGR-CHAR-DAM-09 Rev. 2
Operator:	G. W. C. Silva
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P10101801\
Sample ID:	LEU12-03DTF-I01
Sample Description:	DTF qualifier run: 1200C PyC - in Ar - CGF 0.016
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P10101801_output\

Number of small particles analyzed:	2896
Number of small particles with ellipticity > 1.05	209
Average small particle ellipticity:	1.028

Distribution of the ellipticity (top binned)

Ellipticity (D)	*Frequency*
1.005	2
1.010	80
1.015	332
1.020	572
1.025	530
1.030	427
1.035	302
1.040	210
1.045	127
1.050	105
1.055	53
1.060	46
1.065	29
1.070	28
1.075	13
1.080	13
1.085	8
1.090	9
1.095	1
1.100	4
1.105	2
1.110	2
>1.11	1



G.W. Chinthaka — Operator

10/19/2010 — Date

Data Report Form DRF-18A: Measurement of Pyrocarbon Anisotropy using the 2-MGEM - IPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M10092801
Sample ID:	LEU12-03DTF-B01
Sample Description:	DTF qualifier run: 1200C PyC - in Ar - CGF 0.016
Folder containing data:	\\mc-agr\AGR\2-MGEM\R10101201\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.0821	0.0123	0.0018	1.1789	0.0292	0.0043
2	4,5	0.0829	0.0120	0.0019	1.1808	0.0285	0.0045
3	4,6	0.0795	0.0140	0.0017	1.1727	0.0330	0.0040
4	5,4	0.0903	0.0190	0.0016	1.1985	0.0459	0.0039
5	5,5	0.0776	0.0143	0.0018	1.1683	0.0336	0.0042
6	5,6	0.0857	0.0146	0.0017	1.1875	0.0349	0.0041
7							
8							
9	6,6	0.0870	0.0140	0.0018	1.1906	0.0336	0.0043
10							
Average		0.0836	0.0143	0.0018	1.1825	0.0341	0.0042

Mean of average BAFo per particle:	1.1825
Standard deviation of average BAFo per particle:	0.0105

Comments

Only 7 particles analyzed, according to laboratory supervisor instruction.

G. E. Jellison — Operator

11/2/2010 — Date

Data Report Form DRF-22: Estimation of Average Particle Weight

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Andrew K. Kercher / Dixie L. Barker
Particle Lot ID:	LEU12-03DTF-D00
Particle Lot Description:	DTF qualifier run: 1200C PyC - in Ar - CGF 0.016
Filename:	\\mc-agr\AGR\ParticleWeight\W10093001_DRF22R1.xls

	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Weight of particles (g):	0.0830	0.0967	0.1467	0.1420	0.1097
Number of particles:	319	369	558	541	420
Average weight/particle (g):	2.602E-04	2.621E-04	2.629E-04	2.625E-04	2.612E-04

Mean average weight/particle (g):	2.618E-04
Standard error in mean average weight/particle (g):	4.85E-07

Operator

September 30, 2010

Date

Data Report Form DRF-31: Measurement of Open Porosity using a Mercury Porosimeter

Procedure:	AGR-CHAR-DAM-31 Rev. 1
Operator:	G. W. C. Silva
Coated particle batch ID:	LEU12-03DTF-E01
Batch Description:	DTF qualifier run: 1200C PyC - in Ar - CGF 0.016
Thermocouple Expiration Date:	3/25/11
Penetrometer Expiration Date:	8/16/11
Completed DRF Filename:	\\mc-agr\AGR\Porosimeter\S10100601\S10100601_DRF31R1.xls

Mean average weight/particle (g):	2.62E-04
Standard error in mean average weight/particle (g):	4.85E-07

Weight of particles (g):	13.0551
Approximate number of particles:	49867
Uncertainty in number of particles:	92
Total envelope volume of sample (cc):	1.578
Average envelope volume/particle (cc):	3.17E-05
Sample envelope density (g/cc):	8.272

Average particle diameter (microns):	3.92E+02
Average surface area/particle (cm2):	4.84E-03
Total sample surface area (cm2):	2.41E+02
Intruded mercury volume from 250-10,000 psia (cc):	1.10E-03
Open porosity (ml/m2):	4.56E-02

Comments

G. W. C. Silva — Operator

10/06/2010 — Date

Data Report Form DRF-33: Imaging of Small Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-33 Rev. 0
Operator:	G. W. C. Silva
Sample ID:	LEU12-03DTF-B01
Sample description:	DTF qualifier run: 1200C PyC – in Ar – CGF 0.016
Mount ID number:	M10092801
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P10093001\P1009300101

DMR calibration expiration date:	11/2/10
Calibrated pixels/micron:	4.4767
Stage micrometer calibration expiration date:	2/10/14
Measured value for 300 μm in stage micrometer image (μm):	300.9

Polish-down distance n,m (μm)			
2,2	2,8	8,2	8,8
180	191	190	195

Approximate layer width in polish plane (μm)				
Kernel radius	Layer 1	Layer 2	Layer 3	Layer 4
173	20			

G. W. C. Silva
Operator

10/01/2010
Date

Data Report Form DRF-33: Imaging of Small Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-33 Rev. 0
Operator:	G. W. C. Silva
Sample ID:	LEU12-03DTF-B01
Sample description:	DTF qualifier run: 1200C PyC – in Ar – CGF 0.016
Mount ID number:	M10092802
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P10093001\P1009300102

DMR calibration expiration date:	11/2/10
Calibrated pixels/micron:	4.4767
Stage micrometer calibration expiration date:	2/10/14
Measured value for 300 µm in stage micrometer image (µm):	300.9

Polish-down distance n,m (µm)			
2,2	2,8	8,2	8,8
205	189	221	174

Approximate layer width in polish plane (µm)				
Kernel radius	Layer 1	Layer 2	Layer 3	Layer 4
178	20			

G.W.C. Silva
Operator

10/01/2010
Date

Data Report Form DRF-33: Imaging of Small Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-33 Rev. 0
Operator:	G. W. C. Silva
Sample ID:	LEU12-03DTF-B01
Sample description:	DTF qualifier run: 1200C PyC – in Ar – CGF 0.016
Mount ID number:	M10092803
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P10093001\P1009300103

DMR calibration expiration date:	11/2/10
Calibrated pixels/micron:	4.4767
Stage micrometer calibration expiration date:	2/10/14
Measured value for 300 μm in stage micrometer image (μm):	300.9

Polish-down distance n,m (μm)			
2,2	2,8	8,2	8,8
187	213	193	229

Approximate layer width in polish plane (μm)				
Kernel radius	Layer 1	Layer 2	Layer 3	Layer 4
177	20			

G.W. Chinthaka — Operator

10/01/2010 — Date

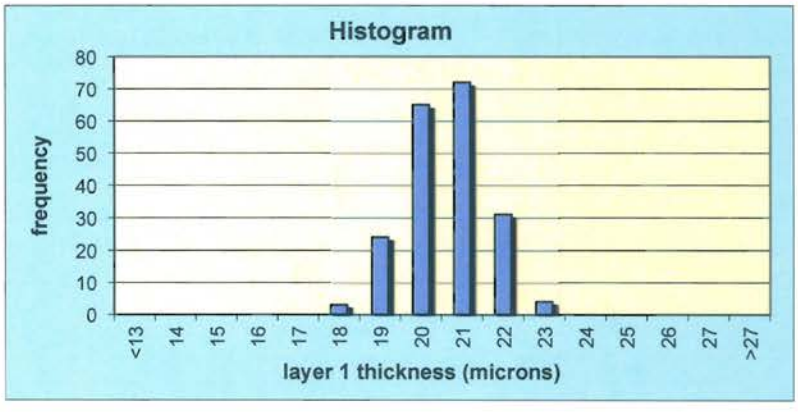
Data Report Form DRF-34A: Measurement of Layer 1 Thickness

Procedure:	AGR-CHAR-DAM-34 Rev. 0
Operator:	Chinthaka Silva
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P10093001\
Sample ID:	LEU12-03DTF
Sample Description:	DTF qualifier run: 1200C PyC - in Ar - CGF 0.016
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P10093001_output\

Number of layers analyzed:	199
Mean of the average layer 1 thickness of each particle (μm):	20.1
Standard deviation in the average layer 1 thickness of each particle (μm):	0.9

Distribution of the average layer 1 thickness (top binned)

Layer 1 Thickness (μm)	*Frequency*
<13	0
14	0
15	0
16	0
17	0
18	3
19	24
20	65
21	72
22	31
23	4
24	0
25	0
26	0
27	0
>27	0



G.W. Chinthaka — Operator

10/04/2010 — Date

7 Characterization of LEU03-07DTF

Product inspection plan AGR-CHAR-PIP-08 Rev. 2 was used to characterize the LEU03-07DTF AGR-3/4 DTF fuel batch. This section contains the associated inspection report form (IRF-08) and the data report forms from that analysis. The LEU03-07DTF particle batch was found to conform to all specifications for DTF particles in section 3.3 of the AGR-3/4 DTF Fuel and Capsule Component Material Specifications, SPC-1214 Rev. 0. Figure 7 shows a polished cross-section of a typical LEU03-07DTF particle.

Characterization samples of LEU03-07DTF were riffled according to the product inspection plan. The measurements of pyrocarbon thickness, density, and optical anisotropy were required for testing conformance to the DTF product specifications in SPC-1214. The mean particle diameter, average uranium content per particle, average particle weight, and average particle envelope volume were measured for later use in compacting. Additional characterization was also performed for information only. Exposed uranium content was measured by acid leaching as-deposited and heat-treated DTF particles and is discussed in section 8. Open porosity of the DTF layer was measured, but the value may be affected by a slight compression of the DTF layer. The old LEU04-02DTF particles had an obvious gap between the DTF layer and the kernel that resulted in a measurable compression when pressure was applied during analysis with a mercury porosimeter. This effect was explained in detail in ORNL/TM-2008/193, “Data Compilation for AGR-3/4 Designed-to-Fail (DTF) Fuel Particle Batch LEU04-02DTF.” There was some indication for the existence of a slight compression during porosimetry in the LEU03-07DTF particles, but it could not be clearly resolved. A small compression component in the porosimetry data would result in a slight over estimate of the open porosity reported on DRF-31.

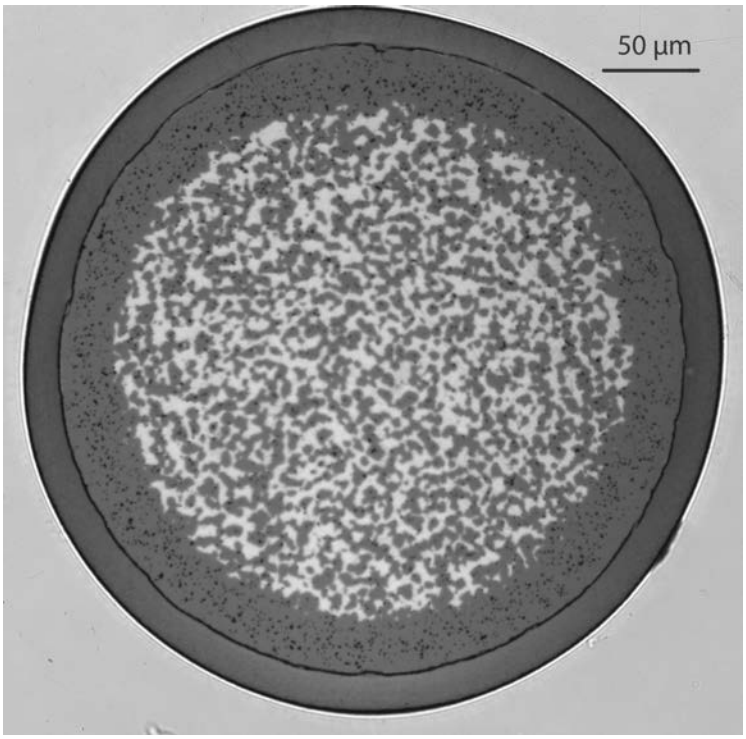


Figure 7. Cross-sectional optical micrograph of a typical LEU03-07DTF particle.

Inspection Report Form IRF-08: AGR-3/4 Designed to Fail Coated Particle Batches

Procedure:	AGR-CHAR-PIP-08 Rev. 2
Coated particle batch ID:	LEU03-07DTF
Coated particle batch description:	AGR-3/4 DTF on kernel composite 69303

Property	Measured Data				Specification	Acceptance Criteria	Acceptance Test Value	Pass or fail	Data Records
	Mean (x)	Std. Dev. (s)	Measurements (n)	k or t value	INL SPC-1214 Revision 0				
Average DTF thickness for each particle (μm)	20.0	0.9	188	1.653	mean 20 ± 5	A = x - ts/√n ≥ 15	19.9	pass	DRF-33 DRF-34
						B = x + ts/√n ≤ 25	20.1	pass	
				2.576	dispersion ≤0.01 ≤ 8	C = x - ks > 8	17.7	pass	
DTF sink/float density (Mg/m³)	1.988	0.009	40	1.685	mean 1.95 ± 0.05	A = x - ts/√n ≥ 1.90	1.986	pass	DRF-03
						B = x + ts/√n ≤ 2.00	1.990	pass	
DTF anisotropy (True BAFo)	1.243	0.019	10	1.833	mean ≥ 1.151	A = x - ts/√n ≥ 1.151	1.232	pass	DRF-18
Particle diameter	400.0	9.2	2709	1.645	Not Applicable				DRF-06 DRF-09
Average uranium per particle (g)	2.353E-04	4.0E-07	3	2.920	Not Applicable				DRF-35
Average particle wt (g)	2.803E-04	7.3E-07	5	2.132	Not Applicable				DRF-22
Average particle envelope volume (cm³)	3.35E-05	-	1	-	Not Applicable				DRF-31
DTF open porosity (ml/m²)	0.079	-	1	-	Measurement Only				DRF-31

Comments

The average diameter aspect ratio was measured to be 1.024.

QC Supervisor — 1-31-11 — Date

Accept coated particle batch (Yes or No):	Yes

QA Reviewer — 2/3/11 — Date

Data Report Form DRF-03: Measurement of PyC Density using a Density Gradient Column

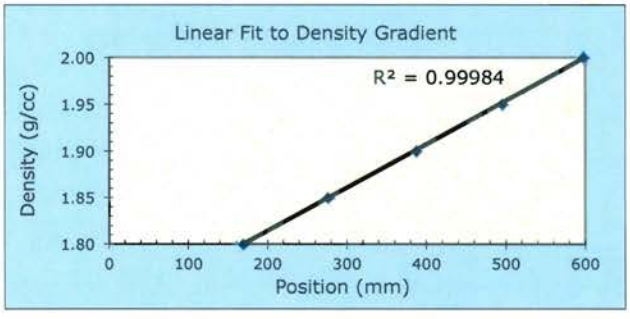
Procedure:	AGR-CHAR-DAM-03 Rev. 3
Operator:	Dixie Barker
Filename:	\\mc-agr\AGR\DensityColumn\D10102501_DRF03R3.xls
Sample ID:	LEU03-07DTF
Sample description:	AGR-3/4 DTF on kernel composite 69303
Float expiration date:	01/2014
Gauge expiration date:	08/2011
Bath temperature:	23.4 °C

Calibrated Floats

Density	Top of Float	Bottom of Float	Center of Mass
1.800	164.57	172.43	168.50
1.850	271.50	279.61	275.56
1.900	383.45	389.26	386.36
1.950	490.57	497.89	494.23
2.000	593.39	600.39	596.89

Linear Fit

slope	StDev	intercept	StDev
4.65E-04	2.85E-06	1.72E+00	1.10E-03



Sample Density

Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density	Fragment Number	Fragment Position	Calculated Density
1	564.09	1.9836	26	579.73	1.9908	51		
2	565.52	1.9842	27	573.55	1.9880	52		
3	565.44	1.9842	28	571.71	1.9871	53		
4	568.42	1.9856	29	569.43	1.9861	54		
5	468.85	1.9393	30	569.03	1.9859	55		
6	571.14	1.9868	31	566.53	1.9847	56		
7	571.44	1.9870	32	564.44	1.9837	57		
8	571.98	1.9872	33	569.83	1.9862	58		
9	572.89	1.9877	34	572.55	1.9875	59		
10	569.45	1.9861	35	569.74	1.9862	60		
11	569.45	1.9861	36	573.38	1.9879	61		
12	572.48	1.9875	37	574.29	1.9883	62		
13	573.05	1.9877	38	576.35	1.9893	63		
14	573.93	1.9881	39	576.40	1.9893	64		
15	575.97	1.9891	40	573.32	1.9879	65		
16	579.03	1.9905	41			66		
17	580.58	1.9912	42			67		
18	585.50	1.9935	43			68		
19	588.76	1.9950	44			69		
20	591.29	1.9962	45			70		
21	592.23	1.9967	46			71		
22	598.61	1.9996	47			72		
23	604.45	2.0023	48			73		
24	606.64	2.0034	49			74		
25	581.00	1.9914	50			75		

Average density of PyC fragments:	1.9880
Standard deviation in density of PyC fragments:	0.0092
Uncertainty in calculated density of PyC fragments:	0.0020

Dixie Barker
Operator

10-25-10
Date

Data Report Form DRF-06: Imaging of Small Particle Diameter and Ellipticity Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-06 Rev. 1
Operator:	G. W. C. Silva
Sample ID:	LEU03-07DTF
Sample Description:	AGR-3/4 DTF on kernel composite 69303
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P10101802\

DMR Calibration Expiration Date:	11/2/10
Stage Micrometer Calibration Expiration Date:	2/10/14
Measured Value for 760 µm in Stage Micrometer Image:	760. µm

G.W. Christmas — Operator

10/18/2010 — Date

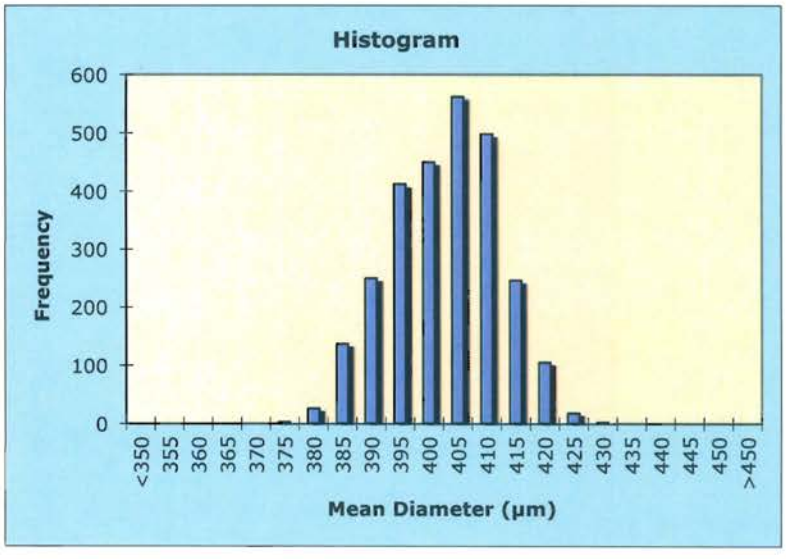
Data Report Form DRF-09A: Measurement of Small Particle Diameter

Procedure:	AGR-CHAR-DAM-09 Rev. 2
Operator:	G. W. C. Silva
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P10101802\
Sample ID:	LEU03-07DTF
Sample Description:	AGR-3/4 DTF on kernel composite 69303
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P10101802_output\

Number of small particles analyzed:	2709
Mean of the average diameter of each small particle (μm):	400.0
Standard deviation in the average diameter of each small particle (μm):	9.2

Distribution of the average particle diameter (top binned)

Mean Diameter (μm)	*Frequency*
<350	0
355	0
360	0
365	0
370	0
375	3
380	26
385	137
390	250
395	412
400	450
405	562
410	498
415	246
420	105
425	18
430	2
435	0
440	0
445	0
450	0
>450	0



G.W.C. Silva
Operator

10/19/2010
Date

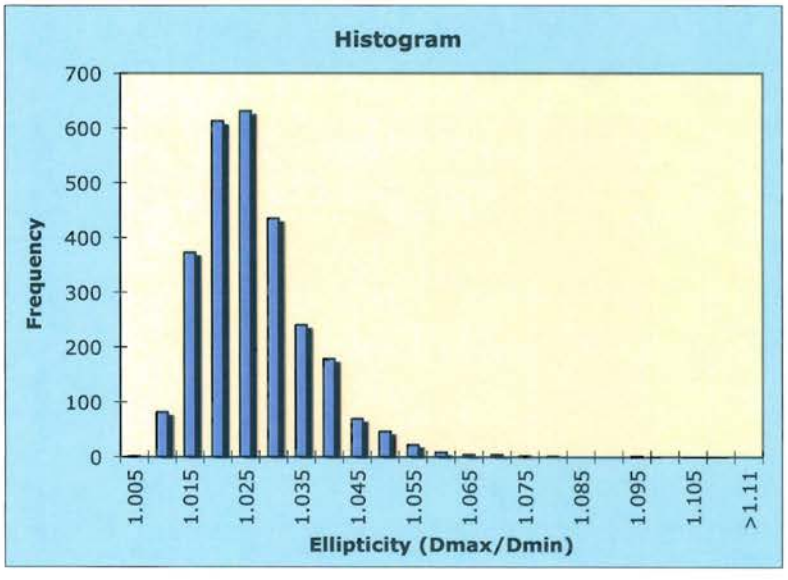
Data Report Form DRF-09B: Measurement of Small Particle Ellipticity (Dmax/Dmin)

Procedure:	AGR-CHAR-DAM-09 Rev. 2
Operator:	G. W. C. Silva
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P10101802\
Sample ID:	LEU03-07DTF
Sample Description:	AGR-3/4 DTF on kernel composite 69303
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Shadow\P10101802_output\

Number of small particles analyzed:	2709
Number of small particles with ellipticity > 1.05	42
Average small particle ellipticity:	1.024

Distribution of the ellipticity (top binned)

Ellipticity (D)	*Frequency*
1.005	1
1.010	81
1.015	373
1.020	613
1.025	631
1.030	435
1.035	240
1.040	178
1.045	69
1.050	46
1.055	22
1.060	8
1.065	4
1.070	4
1.075	2
1.080	1
1.085	0
1.090	0
1.095	1
1.100	0
1.105	0
1.110	0
>1.11	0



G.W.C. Silva — Operator

10/19/2010 — Date

Data Report Form DRF-18A: Measurement of Pyrocarbon Anisotropy using the 2-MGEM - IPyC

Procedure:	AGR-CHAR-DAM-18 Rev. 1
Operator:	G. E. Jellison
Mount ID:	M10101501
Sample ID:	LEU03-07DTF
Sample Description:	AGR-3/4 DTF on kernel composite 69303
Folder containing data:	\\mc-agr\AGR\2-MGEM\R10102201\

Particle #	Grid Position	Diattenuation			True BAFo = (1+N)/(1-N)		
		Average	St. Dev.	Ave. Error	Average	St. Dev.	Ave. Error
1	4,4	0.0922	0.0147	0.0009	1.2031	0.0357	0.0022
2	4,5	0.1047	0.0170	0.0007	1.2339	0.0424	0.0017
3	4,6	0.1142	0.0176	0.0007	1.2578	0.0449	0.0018
4	5,4	0.1091	0.0181	0.0007	1.2449	0.0456	0.0018
5	5,5	0.1114	0.0219	0.0006	1.2507	0.0555	0.0015
6	5,6	0.1136	0.0180	0.0007	1.2563	0.0458	0.0018
7	6,4	0.1100	0.0174	0.0006	1.2472	0.0439	0.0015
8	6,5	0.0998	0.0199	0.0006	1.2217	0.0491	0.0015
9	6,6	0.1101	0.0217	0.0006	1.2474	0.0548	0.0015
10	5,7	0.1185	0.0258	0.0007	1.2689	0.0664	0.0018
Average		0.1084	0.0192	0.0007	1.2432	0.0484	0.0017

Mean of average BAFo per particle:	1.2432
Standard deviation of average BAFo per particle:	0.0191

Comments

G. E. Jellison — Operator

11/2/2010 — Date

Data Report Form DRF-22: Estimation of Average Particle Weight

Procedure:	AGR-CHAR-DAM-22 Rev. 1
Operator:	Dixie L. Barker / Andrew K. Kercher
Particle Lot ID:	LEU03-07DTF-D00
Particle Lot Description:	AGR-3/4 DTF on kernel composite 69303
Filename:	\\mc-agr\AGR\ParticleWeight\W10102801_DRF22R1.xls

	Sample 6	Sample 7	Sample 8	Sample 9	Sample 10
Weight of particles (g):	0.1022	0.0980	0.1108	0.1032	0.1135
Number of particles:	365	349	394	369	406
Average weight/particle (g):	2.800E-04	2.808E-04	2.812E-04	2.797E-04	2.796E-04

Mean average weight/particle (g):	2.803E-04
Standard error in mean average weight/particle (g):	3.25E-07

Dixie Barker
Operator

10-26-10
Date

Data Report Form DRF-31: Measurement of Open Porosity using a Mercury Porosimeter

Procedure:	AGR-CHAR-DAM-31 Rev. 1
Operator:	G. W. C. Silva
Coated particle batch ID:	LEU03-07DTF-E01
Batch Description:	AGR-3/4 DTF on kernel composite 69303
Thermocouple Expiration Date:	3/25/11
Penetrometer Expiration Date:	8/16/11
Completed DRF Filename:	\\mc-agr\AGR\Porosimeter\S10102001\S10102001_DRF31R1.xls

Mean average weight/particle (g):	2.80E-04
Standard error in mean average weight/particle (g):	3.25E-07

Weight of particles (g):	13.4140
Approximate number of particles:	47856
Uncertainty in number of particles:	55
Total envelope volume of sample (cc):	1.601
Average envelope volume/particle (cc):	3.35E-05
Sample envelope density (g/cc):	8.379

Average particle diameter (microns):	4.00E+02
Average surface area/particle (cm2):	5.02E-03
Total sample surface area (cm2):	2.40E+02
Intruded mercury volume from 250-10,000 psia (cc):	1.90E-03
Open porosity (ml/m2):	7.91E-02

Comments
Open porosity was determined using HP plot rather than Merge plot.

G.W. Silva
Operator

11/01/2010
Date

Data Report Form DRF-33: Imaging of Small Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-33 Rev. 0
Operator:	G. W. C. Silva
Sample ID:	LEU03-07DTF
Sample description:	AGR-3/4 DTF on kernel composite 69303
Mount ID number:	M10101801
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P10102201\P1010220101\

DMR calibration expiration date:	11/2/10
Calibrated pixels/micron:	4.4767
Stage micrometer calibration expiration date:	2/10/14
Measured value for 300 μm in stage micrometer image (μm):	300.2

Polish-down distance n,m (μm)			
2,2	2,8	8,2	8,8
189	218	199	227

Approximate layer width in polish plane (μm)				
Kernel radius	Layer 1	Layer 2	Layer 3	Layer 4
186	20			

G.W. Chinthaka — Operator

10/22/2010 — Date

Data Report Form DRF-33: Imaging of Small Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-33 Rev. 0
Operator:	G. W. C. Silva
Sample ID:	LEU03-07DTF
Sample description:	AGR-3/4 DTF on kernel composite 69303
Mount ID number:	M10101802
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P10102201\P1010220102\

DMR calibration expiration date:	11/2/10
Calibrated pixels/micron:	4.4767
Stage micrometer calibration expiration date:	2/10/14
Measured value for 300 µm in stage micrometer image (µm):	300.2

Polish-down distance n,m (µm)			
2,2	2,8	8,2	8,8
227	196	233	206

Approximate layer width in polish plane (µm)				
Kernel radius	Layer 1	Layer 2	Layer 3	Layer 4
179	21			

G.W. Chidthab
Operator

10/22/2010
Date

Data Report Form DRF-33: Imaging of Small Particle Cross-sections Using an Optical Microscope System

Procedure:	AGR-CHAR-DAM-33 Rev. 0
Operator:	G. W. C. Silva
Sample ID:	LEU03-07DTF
Sample description:	AGR-3/4 DTF on kernel composite 69303
Mount ID number:	M10101803
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\P10102201\P1010220103\

DMR calibration expiration date:	11/2/10
Calibrated pixels/micron:	4.4767
Stage micrometer calibration expiration date:	2/10/14
Measured value for 300 μm in stage micrometer image (μm):	300.2

Polish-down distance n,m (μm)			
2,2	2,8	8,2	8,8
208	236	209	232

Approximate layer width in polish plane (μm)				
Kernel radius	Layer 1	Layer 2	Layer 3	Layer 4
182	21			

G. W. C. Silva
Operator

10/22/2010
Date

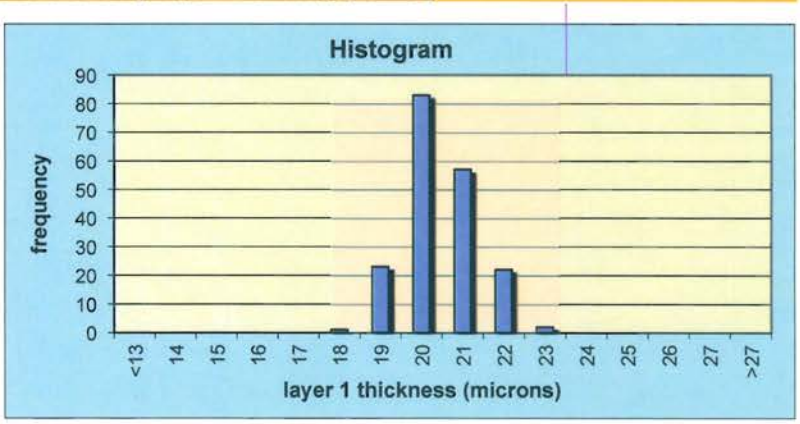
Data Report Form DRF-34A: Measurement of Layer 1 Thickness

Procedure:	AGR-CHAR-DAM-34 Rev. 0
Operator:	G. W. C. Silva
Folder name containing images:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P10102201\
Sample ID:	LEU03-07DTF
Sample Description:	AGR-3/4 DTF on kernel composite 69303
Folder name containing processed data:	\\mc-agr\AGR\ImageProcessing\Completed_Layers\P10102201_output\

Number of layers analyzed:	188
Mean of the average layer 1 thickness of each particle (μm):	20.0
Standard deviation in the average layer 1 thickness of each particle (μm):	0.9

Distribution of the average layer 1 thickness (top binned)

Layer 1 Thickness (μm)	*Frequency*
<13	0
14	0
15	0
16	0
17	0
18	1
19	23
20	83
21	57
22	22
23	2
24	0
25	0
26	0
27	0
>27	0



G. W. C. Silva
Operator

10/22/2010
Date

Data Report Form DRF-35: Fuel Particle Uranium Loading

Procedure:	AGR-CHAR-DAM-35 Rev. 0
Operator:	Fred Montgomery
Particle lot ID:	LEU03-07TDTF
Particle lot description:	AGR-3/4 DTF on kernel composite 69303
Filename:	\\mc-agr\AGR\UraniumLoading\LEU03-07DTF_DRF35R0.xls

Mean average weight per particle (g):	2.803E-04
Standard error in mean average weight per particle (g):	3.3E-07

	Sample 1		Sample 2		Sample 3	
	Leach 1	Leach 2	Leach 1	Leach 2	Leach 1	Leach 2
Particle sample ID:	LEU03-07DTF-F01		LEU03-07DTF-G01		LEU03-07DTF-H01	
Weight of particles:	3.8980		4.0361		3.9844	
Approximate number of particles:	13907		14399		14215	
Uncertainty in number of particles:	16		17		16	
Acid leach sample ID:	U10110101	U10110201	U10110102	U10110202	U10110103	U10110203
Radiochemical laboratory analysis number:	3346-001	3346-004	3346-002	3346-005	3346-003	3346-006
Weight U in leach (mg):	3271	0.146	3383	0.088	3351	0.095
Uncertainty in weight U in leach (mg):	6	0.015	4	0.009	11	0.010
Total weight U in sample (mg):	3271		3383		3351	
Average weight U per particle (mg):	0.2352		0.2349		0.2357	
Uncertainty in average weight U per particle (mg):	0.0005		0.0004		0.0008	

Mean average uranium loading per particle (g):	2.353E-04
Standard error in mean average uranium loading per particle (g):	2.3E-07

Comments

Leach 1 was analyzed by Davies-Gray titration method. Leach 2 was analyzed by ICP-MS, due to low U concentration.
Davies-Gray: initial known U recovery = 100.7%; final known U recovery=100.3%
Uncertainty in Davies-Gray based on standard deviation from 3 titrations.
Data checked by FCM against official results of analyses for RMAL3346 on 1/13/2011.

Fred C. Montgomery
Operator

1-20-2011
Date

8 Analysis of Uranium Retention Before and After Heat-treatment

During AGR-3/4 fuel compact fabrication, compacts were heated from room temperature to 1800°C at approximately 20°C/min, held at 1800°C for 1 hour, and then cooled back to room temperature at no more than 20°C/min (ORNL/TM-2011/124, "Data Compilation for AGR-3/4 Designed-to-Fail (DTF) Fuel Compact Lot (LEU03-10T-OP2/LEU03-07DTF-OP1)-Z"). After a sample of particles from the old LEU04-02DTF particle batch were heated in vacuum under these conditions, 20% of the uranium in the kernels was no longer contained by the DTF pyrocarbon coating (see Section 1). One of the goals for the DTF particles fabricated to replace these original DTF particles in the AGR-3/4 fuel irradiation test was to minimize this observed coating failure. The DTF coating is designed to fail early in the irradiation test to produce a controlled release of fission products. Failure of the coating during compact fabrication could result in undesirable dispersion of uranium out of the fuel kernel prior to the start of the irradiation.

Samples of LEU12-03DTF and LEU03-07DTF were tested for uranium retention by the DTF pyrocarbon layer. Particles were immersed in boiling nitric acid to dissolve any exposed uranium. Two 24 hour dissolutions were performed, after which the acid solutions were decanted off for uranium analysis by mass spectrometry. Particles were tested in the as-coated state and after heat-treatment. Heat-treatment was performed by distributing the particles in a bed of graphite powder inside a clean graphite crucible and heating to 1800°C in vacuum using the ramp rates and hold times described above to simulate thermal processing during compact fabrication (Figure 8).

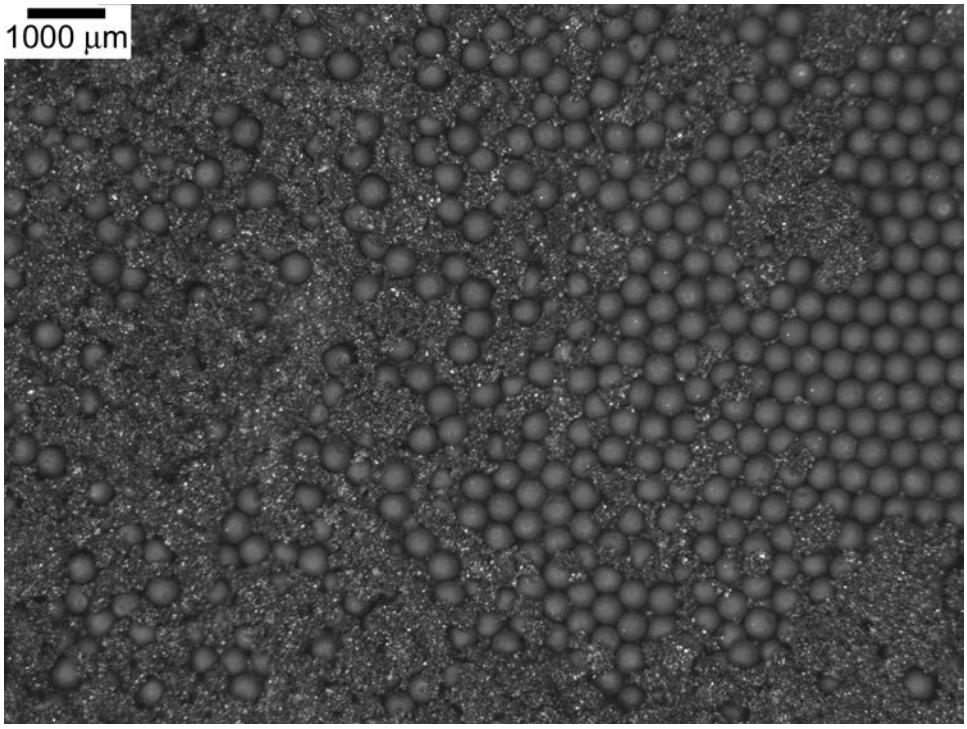


Figure 8. Particles from LEU03-07DTF after heat-treatment in a graphite powder bed.

Table 7 summarizes the results of the uranium retention study; data report forms from the analyses are attached to the end of this section. The equivalent number of exposed kernels in each sample was determined by dividing the total amount of uranium dissolved in the nitric acid by the average uranium content of one kernel. As discussed in Section 1, modifications to the coating process used to fabricate LEU03-07DTF resulted in fewer defects in the coating layer, lower anisotropy, and dramatically improved uranium retention after heat treatment. The exposed uranium after heat-treatment was reduced from ~20% in the original DTF batch to ~2% in the new DTF batch. In an additional informal test, approximately 2000 DTF particles from a development batch were overcoated, compacted, heat-treated, electrolytically deconsolidated, and nitric acid leached. At the conclusion of this test, the amount of exposed uranium was equivalent to only ~0.2% of the sample (4 equivalent exposed kernels out of ~2000 particles). This suggests that the DTF particles may perform even better than predicted by the uranium retention results for the particles heat-treated in a bed of graphite powder.

Table 7. Results of analysis of uranium retention by the DTF coating.

	Condition	Equivalent number of exposed kernels	Number of particles analyzed	Percent failure
LEU04-02DTF Original DTF Fuel	as coated	3	5125	0.06%
	heat-treated	1011	5121	20%
LEU12-03DTF New DTF qualification batch	as coated	7	11683	0.06%
	heat-treated	293	11827	2.5%
LEU03-07DTF New DTF Fuel	as coated	1	10611	0.009%
	heat-treated	191	10839	1.8%

Data Report Form DRF-40: Measurement of U Contamination and Impurities of Loose Particles by Leach Testing

Procedure:	AGR-CHAR-DAM-40 Rev. 0
Operator:	Fred Montgomery
Particle lot ID:	LEU12-03DTF-P01
Particle lot description:	DTF qualifier run, as-coated
DRF filename:	\\mc-agr\AGR\LeachBurnLeach\LEU12-03DTF-P01_DRF40R0.xls

Mean average weight uranium per particle (g):	2.178E-04
Uncertainty in mean average weight uranium per particle (g):	5.37E-07
Mean average weight per particle (g):	2.618E-04
Uncertainty in mean average weight per particle (g):	4.85E-07
Weight of particles in sample (g):	3.0585
Approximate total number of particles in sample:	11683
Uncertainty in total number of particles in sample:	22

		First extraction	Second extaction	Total
	Leach solution ID:	L10110801	L10111001	
	Total volume of leach solution (ml):	36.0	33.8	
	Radiochemical laboratory analysis number:	3352-001	3352-003	
	Measured uranium concentration (µg/ml):	4.05E+01	3.18E-01	
	Uncertainty in uranium concentration (µg/ml):	4.05E+00	3.18E-02	
	Weight uranium leached (g):	1.46E-03	1.07E-05	1.47E-03
	Uncertainty in weight uranium leached (g):	1.48E-04	1.09E-06	1.48E-04
	Effective number of exposed kernels:	6.7	0.0	6.7
	Uncertainty in effective number of exposed kernels:	0.7	0.0	0.7
Fe	Measured concentration of impurity in sample (µg/ml):			Fe
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Cr	Measured concentration of impurity in sample (µg/ml):			Cr
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Mn	Measured concentration of impurity in sample (µg/ml):			Mn
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Co	Measured concentration of impurity in sample (µg/ml):			Co
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Ni	Measured concentration of impurity in sample (µg/ml):			Ni
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Ca	Measured concentration of impurity in sample (µg/ml):			Ca
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Al	Measured concentration of impurity in sample (µg/ml):			Al
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Ti	Measured concentration of impurity in sample (µg/ml):			Ti
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
V	Measured concentration of impurity in sample (µg/ml):			V
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			

Comments

Data checked against the official results of analyses for RMAL3352 by FCM on 1/05/2011.
Uranium loading of kernel estimated from average weight of kernels (2.418E-4 ± 5.96E-7 g) multiplied by 90.06 wt% U reported by B&W for kernel composite 69302.

Fred C. Montgomery
Operator

5-2-2011
Date

Data Report Form DRF-40: Measurement of U Contamination and Impurities of Loose Particles by Leach Testing

Procedure:	AGR-CHAR-DAM-40 Rev. 0
Operator:	Fred Montgomery
Particle lot ID:	LEU12-03DTF-T01
Particle lot description:	DTF Qualifier Run, heat-treated
DRF filename:	\\mc-agr\AGR\LeachBurnLeach\LEU12-03DTF-P01_DRF40R0.xls

Mean average weight uranium per particle (g):	2.178E-04
Uncertainty in mean average weight uranium per particle (g):	5.37E-07
Mean average weight per particle (g):	2.618E-04
Uncertainty in mean average weight per particle (g):	4.85E-07
Weight of particles in sample (g):	3.0962
Approximate total number of particles in sample:	11827
Uncertainty in total number of particles in sample:	22

		First extraction	Second extaction	Total
	Leach solution ID:	L10110802	L10111002	
	Total volume of leach solution (ml):	28.8	35.5	
	Radiochemical laboratory analysis number:	3352-002	3352-004	
	Measured uranium concentration (µg/ml):	1.61E+03	4.91E+02	
	Uncertainty in uranium concentration (µg/ml):	1.61E+02	4.91E+01	
	Weight uranium leached (g):	4.64E-02	1.74E-02	6.38E-02
	Uncertainty in weight uranium leached (g):	4.75E-03	1.77E-03	5.07E-03
	Effective number of exposed kernels:	212.9	80.0	292.9
	Uncertainty in effective number of exposed kernels:	21.8	8.1	23.3
Fe	Measured concentration of impurity in sample (µg/ml):			Fe
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Cr	Measured concentration of impurity in sample (µg/ml):			Cr
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Mn	Measured concentration of impurity in sample (µg/ml):			Mn
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Co	Measured concentration of impurity in sample (µg/ml):			Co
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Ni	Measured concentration of impurity in sample (µg/ml):			Ni
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Ca	Measured concentration of impurity in sample (µg/ml):			Ca
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Al	Measured concentration of impurity in sample (µg/ml):			Al
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Ti	Measured concentration of impurity in sample (µg/ml):			Ti
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
V	Measured concentration of impurity in sample (µg/ml):			V
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			

Comments

Data checked against the official results of analyses for RMAL3352 by fcm on 1/05/2011.
Uranium loading of kernel estimated from average weight of kernels (2.418E-4 ± 5.96E-7 g) multiplied by 90.06 wt% U reported by B&W for kernel composite 69302.

Fred C. Montgomery
Operator

5-2-2011
Date

Data Report Form DRF-40: Measurement of U Contamination and Impurities of Loose Particles by Leach Testing

Procedure:	AGR-CHAR-DAM-40 Rev. 0
Operator:	Fred Montgomery
Particle lot ID:	LEU03-07DTF-P01
Particle lot description:	AGR-3/4 DTF on kernel composite 69303, as-coated
DRF filename:	\\mc-agr\AGR\LeachBurnLeach\LEU03-07DTF-P01_DRF40R0.xls

Mean average weight uranium per particle (g):	2.353E-04
Uncertainty in mean average weight uranium per particle (g):	2.30E-07
Mean average weight per particle (g):	2.803E-04
Uncertainty in mean average weight per particle (g):	3.25E-07
Weight of particles in sample (g):	2.9744
Approximate total number of particles in sample:	10611
Uncertainty in total number of particles in sample:	12

		First leach	Second leach	Total
	Leach solution ID:	L10251001	L10261001	
	Total volume of leach solution (ml):	35.5	36.0	
	Radiochemical laboratory analysis number:	3320-001	3320-003	
	Measured uranium concentration (µg/ml):	6.24E+00	1.88E-02	
	Uncertainty in uranium concentration (µg/ml):	6.24E-01	1.88E-03	
	Weight uranium leached (g):	2.22E-04	6.77E-07	2.22E-04
	Uncertainty in weight uranium leached (g):	2.25E-05	6.88E-08	2.25E-05
	Effective number of exposed kernels:	0.9	0.0	0.9
	Uncertainty in effective number of exposed kernels:	0.1	0.0	0.1
Fe	Measured concentration of impurity in sample (µg/ml):			Fe
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Cr	Measured concentration of impurity in sample (µg/ml):			Cr
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Mn	Measured concentration of impurity in sample (µg/ml):			Mn
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Co	Measured concentration of impurity in sample (µg/ml):			Co
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Ni	Measured concentration of impurity in sample (µg/ml):			Ni
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Ca	Measured concentration of impurity in sample (µg/ml):			Ca
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Al	Measured concentration of impurity in sample (µg/ml):			Al
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Ti	Measured concentration of impurity in sample (µg/ml):			Ti
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
V	Measured concentration of impurity in sample (µg/ml):			V
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			

Comments

Data checked against the official Results of Analyses for RMAL3320 by FCM on 12/15/2010.

Fred C. Montgomery
Operator

5-2-2011
Date

Data Report Form DRF-40: Measurement of U Contamination and Impurities of Loose Particles by Leach Testing

Procedure:	AGR-CHAR-DAM-40 Rev. 0
Operator:	Fred Montgomery
Particle lot ID:	LEU03-07DTF-T01
Particle lot description:	AGR-3/4 DTF on kernel composite 69303, heat-treated
DRF filename:	\\mc-agr\AGR\LeachBurnLeach\LEU03-07DTF-T01_DRF40R0.xls

Mean average weight uranium per particle (g):	2.353E-04
Uncertainty in mean average weight uranium per particle (g):	2.30E-07
Mean average weight per particle (g):	2.803E-04
Uncertainty in mean average weight per particle (g):	3.25E-07
Weight of particles in sample (g):	3.0383
Approximate total number of particles in sample:	10839
Uncertainty in total number of particles in sample:	13

		First leach	Second leach	Total
	Leach solution ID:	L10251002	L10261002	
	Total volume of leach solution (ml):	33.8	35.8	
	Radiochemical laboratory analysis number:	3320-002	3320-004	
	Measured uranium concentration (µg/ml):	1.27E+03	5.50E+01	
	Uncertainty in uranium concentration (µg/ml):	1.27E+02	5.50E+00	
	Weight uranium leached (g):	4.29E-02	1.97E-03	4.49E-02
	Uncertainty in weight uranium leached (g):	4.37E-03	2.00E-04	4.38E-03
	Effective number of exposed kernels:	182.4	8.4	190.8
	Uncertainty in effective number of exposed kernels:	18.6	0.9	18.6
Fe	Measured concentration of impurity in sample (µg/ml):			Fe
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Cr	Measured concentration of impurity in sample (µg/ml):			Cr
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Mn	Measured concentration of impurity in sample (µg/ml):			Mn
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Co	Measured concentration of impurity in sample (µg/ml):			Co
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Ni	Measured concentration of impurity in sample (µg/ml):			Ni
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Ca	Measured concentration of impurity in sample (µg/ml):			Ca
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Al	Measured concentration of impurity in sample (µg/ml):			Al
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
Ti	Measured concentration of impurity in sample (µg/ml):			Ti
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			
V	Measured concentration of impurity in sample (µg/ml):			V
	Uncorrected weight of impurity in sample (µg):			
	Weight of impurity in blank (µg):			
	Minimum corrected weight of impurity in sample (µg):			
	Maximum corrected weight of impurity in sample (µg):			

Comments

Data checked against the official Results of Analyses for RMAL3320 by FCM on 12/15/2010.

Fred C. Montgomery
Operator

5-2-2011
Date