INL/MIS-17-43272-Revision-2

ECAR-3873 Acceptance of Nonconforming Fuel for the AGR-5/6/7 Irradiation Experiments

November 2020

Changing the World's Energy Future

D. W. Marshall

Idaho National Laboratory

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Idaho Falls, Idaho 83415

http://www.inl.gov

Prepared for the
U.S. Department of Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

INL/MIS-17-43272
Revision 2

SIGNATURES

	(Name)	(Organization)	(Signature)	(Date)
Performer:	D. W. Marshall	C620		11/4/2020
Checker[1]:	M. A. Plummer	C220		11/4/2020
Independent Peer Reviewer[2]:	N/A			
CUI Reviewer: Yes ☐ or No[5] ☒				
Manager[3]:	T. R. Mitchell	C640		11/4/2020
Owner[4]:	P. A. Demkowicz	C600		11/4/2020
Nuclear Safety[4]:	N/A			
Cognizant Engineer[4]:	N/A			
Quality Assurance:	Michelle T. Sharp	H330/C020		11/4/2020

1. Confirmation of completeness, mathematical accuracy, and correctness of data and appropriateness of assumptions.
2. Concurrence of method or approach. See definition, LWP-10106.
3. Concurrence of procedure compliance. Concurrence with method/approach and conclusion.
4. Concurrence with the document's assumptions and input information. See definition of Acceptance, LWP-10200.
5. Does the document contain CUI material please check either yes or no.

REVISION LOG

Rev.	Date	Affected Pages	Revision Description
0	09/26/2017	All	New document
1	05/13/2020	All	Major rewrite to incorporate new characterization data.
2	11/04/2020	6-8	Revised TRISO lot defect data in Table 1. Appended revision information to the Prologue, corrected a reference callout, and added URL information to TCT references.

1. Quality Level (QL) No.	QL-3	**Professional Engineer's Stamp** N/A See LWP-10010 for requirements.
2. QL Determination No.	NA	
3. Engineering Job (EJ) No.	NA	
4. SSC ID	NA	
5. Building NA	X	
6. Site Area NA	X	

7. Objective/Purpose:

Document acceptance of graphitic fuel compacts fabricated for the Advanced Gas Reactor (AGR) irradiation experiments, AGR-5/6/7, despite non-conformance with three fuel specifications.

8. If revision, please state the reason and list sections and/or pages being affected:

Since issuance of the original document, additional characterization data have been collected that warrant a change in the reported mean defect values.

9. Conclusions/Recommendations:

The fuel compacts, although not fully conforming to fuel specifications, are of sufficient quality that useful and meaningful data can be collected from the AGR-5/6/7 experiment irradiations. The fuel compacts are found to be acceptable for the purposes of the irradiations.

CONTENTS

PROJECT ROLES AND RESPONSIBILITIES

Project Role	Name	Organization	Pages Covered (if applicable)
Performer	D. W. Marshall	C620	
Checker[a]	M. A. Plummer	C220	
Independent Reviewer[b]	N/A		
CUI Reviewer[c]	H. R. Creasey	M310	
Manager[d]	T. R. Mitchell	C601	
Requestor[ef]	P. A. Demkowicz	C100	
Nuclear Safety[f]	N/A		
Document Owner[f]	P. A. Demkowicz	C100	

Responsibilities:

a. Confirmation of completeness, mathematical accuracy, and correctness of data and appropriateness of assumptions.

b. Concurrence of method or approach. See definition, LWP-10106.

c. Concurrence with the document's markings in accordance with LWP-11202.

d. Concurrence of procedure compliance. Concurrence with method/approach and conclusion.

e. Authorizes the commencement of work of the engineering deliverable.

f. Concurrence with the document's assumptions and input information. See definition of Acceptance, LWP-10200.

NOTE: *Delete or mark "N/A" for project roles not engaged. Include ALL personnel and their roles listed above in the eCR system. The list of the roles above is not all inclusive. If needed, the list can be extended or reduced.*

PROLOGUE

Revision 0 of ECAR-3873 was written based on pooled data sets from defect analyses performed by BWX Technologies Nuclear Operations Group (BWXT-NOG) and the first set of "confirmatory" analyses performed by Oak Ridge National Laboratory (ORNL). The BWXT-NOG data exhibited some anomalous results, such as subsequent leaches recovering more uranium than previous leaches, that called the data set into question. Furthermore, the levels of the defect fractions were unexpected. A sample of fuel compacts were sent to ORNL to confirm the BWXT-NOG results and reduce statistical uncertainties. The ORNL data, however, did not confirm the BWXT-NOG data for all defects and were insufficient to allow the rejection of either data set. Revision 0 used all available data from both sources that could not be rejected as outliers. A second round of analyses were conducted by ORNL, using slightly revised methods to assure a good separation of the supernate from the graphite sludge. These results largely confirmed the previous ORNL analyses and provided a statistical basis to reject the results from the BWXT-NOG defect analyses. Revision 1 results and conclusions for the defect fractions are based on the ORNL analyses. Revision 2 replaces defect fraction data for the TRISO particle lot, J52R-16-98005 with values derived from BWXT data.[2]

SCOPE AND BRIEF DESCRIPTION

Prismatic Very-High Temperature Reactor (VHTR) fuel was fabricated at two nominal packing fractions (PFs) of 25% and 40% for the AGR irradiation experiments, AGR-5/6/7. The fuel did not meet all fuel specifications. The mean thickness of the outer pyrocarbon (OPyC) for the tristructural isotropic (TRISO) fuel particles was below the specified range, the as-measured dispersed uranium fraction (DUF) for the 25% PF compacts was above the specified maximum at 95% confidence, and the specified maximum exposed kernel fraction (EKF) was exceeded by the 40% PF compacts.

Furthermore, because the impurities outside of the silicon carbide (SiC) layer, as measured by BWXT-NOG all measured below the detection limit for the method on a clutch of five compacts, the results were reported with units "µg/5-compact clutch" and could not be reported with the required units of "µg/compact."

This document describes the reasons why the fuel compacts were deemed acceptable to use in the AGR-5/6/7 irradiation experiment despite the nonconforming properties.

The original (Rev. 0) version of this document was written before the second round of "confirmatory defect analyses" had been completed by ORNL on the fuel compacts, which improved the data statistics and enabled the rejection of questionable DUF, EKF, and SiC data. BWXT-NOG data are generally rejected because of anomalies (subsequent leaches recovering more U than the first leach, no U recovered on several concurrent leaches, etc.). Data reported for the 25% PF in the table below includes ORNL generated data for J52R-16-14156C & D, but no data were collected for J52R-16-14157C. The quality of the latter is inferred from the former because the compacts were produced from the same TRISO particle lot, overcoated with resinated-graphite powder and compacted using the same equipment and process parameters.

DESIGN OR TECHNICAL PARAMETER INPUT AND SOURCES

None

RESULTS OF LITERATURE SEARCHES AND OTHER BACKGROUND DATA

Table 1. Fuel characterization data and specifications.

Property	Mean (N)		Mean at 95% Confidence [a]	Dispersion 0.95/0.99
OPyC Thickness Specification [1]	---		36 - 44 µm	≤ 20 µm [b]
TRISO lot J52R-16-98005 [2]	35.03 µm		≥ 34.75 µm (F)	30.76 µm
Dispersed U Fraction Specification [1]	---		≤ 1.0E-5	---
TRISO lot J52R-16-98005 [2,c]	1.04E-5		---	---
25% PF Compacts [3,4]	5.02E-6		≤ 5.59E-6	---
40% PF Compacts [3,4]	4.95E-6		≤ 5.68E-6	---
Overcoated TRISO, 40% PF [3,4]	5.25E-6		≤ 5.37E-6 (N)	
Exposed Kernel Fraction Specification [1]	---		≤ 5.0E-5	---
TRISO lot J52R-16-98005 [2,c]	9.40E-6		≤ 2.43E-5 (N)	---
25% PF Compacts [3,4]	7.27E-6		≤ 3.45E-5	---
40% PF Compacts [3,4]	5.39E-5		≤ 8.30E-5 (F)	---
Overcoated TRISO, 40% PF [3,4]	0.00		≤ 1.42E-5 (N)	---
SiC Defect Fraction Specification [1]	---		≤ 1.0E-4	---
TRISO lot J52R-16-98005 [2,c]	1.89E-5		≤ 4.88E-5 (N)	---
25% PF Compacts [3,4]	2.18E-5		≤ 5.64E-5	---
40% PF Compacts [3,4]	4.67E-5		≤ 7.43E-5	---
Overcoated TRISO, 40% PF [3,4]	9.47E-6		≤ 2.99E-5(N)	---

a.
b. (N) Property calculated, but not specified, and (F) fails to conform to the fuel specification.
c. Not more than 1% of the population may be less than 20 µm at 95% statistical confidence.
d. Calculated from data in the BWXT J52R-16-98005 certification package, pp. 16, 413-418.

ASSUMPTIONS

Not Applicable

COMPUTER CODE VALIDATION

Not Applicable

DISCUSSION/ANALYSIS

TRISO OPyC Thickness

The OPyC layer performs multiple functions. 1) It serves as a final barrier to fission product releases, specifically for gaseous fission products. 2) It shrinks under fast neutron irradiation and is presumed to compressively load the SiC layer; thereby preventing the silicon carbide layer from failing due to hoop stresses caused by internal pressurization. 3) The OPyC layer provides mechanical protection of the brittle SiC layer during subsequent handling.

The United States AGR Program used a mixed-ceramic fuel kernel that is composed of uranium carbides and oxides (UCO). UCO kernels evolve far less CO than urania kernels employed by other countries. Consequently, the internal pressures formed during irradiation are far less and hoop stress failures are effectively prevented. Furthermore, post-irradiation-examinations (PIE) on AGR-1 and AGR-2 fuels show some tendency for the OPyC to shrink away from the SiC vs. towards the SiC as conventionally imagined. Therefore, the OPyC layer may not impose a significant compressive load on the SiC as assumed.

The fuel specification [1] for the OPyC thickness was based on all three functions, collectively. A major driver for the specification was an adequate thickness to enable the OPyC to compress the SiC layer without cracking. Since the OPyC layer may not compress the SiC, as envisioned, and because thinner OPyC layers can adequately perform the functions of retaining gaseous fission products and protecting the SiC layer from mechanical damage, an OPyC thickness of ~ 35 µm will be sufficient and no degradation of the TRISO in-pile performance is expected due to the mean thickness being slightly below the specification at 95% confidence.

The narrowly distributed OPyC thicknesses keep the lower tail of the distribution well above the critical limit of 20 µm, which further supports the argument that the thickness will be adequate for the functions it performs.

The AGR Technical Coordination Team (TCT) was consulted[5] before the TRISO batches were selected for composing the TRISO lot, knowing that conformance to the OPyC thickness specification could be jeopardized by the selection of batches. The TCT recommendation was: "BWXT TRISO coating batches J52O-16-93165, 93168 and 93169 should be included in the coated particle composite to be used for AGR-5/6/7 compact formation," with full knowledge that inclusion of TRISO batch J52O-16-93165 would result in the composite mean OPyC thickness failing the fuel specification.

INL instructed BWXT-NOG to compose the TRISO lot from TRISO batches J52O-16-93165, 93168, 93169, and 93170 [6]. BWXT-NOG issued a Quality Control Deficiency Notice (QCDN) [7] documenting that the composited TRISO lot did not meet the mean OPyC specification. The QCDN was accepted by INL with the disposition of "Approve as-is."

Compact Defect Fractions

The three defects that are quantified by the compact deconsolidation-leach-burn-leach (DLBL) method are the DUF, EKF, and the SiC defect fraction (SDF). The total contribution of all defects (DUF, EKF, and SDF) is to be kept below an effective 2E-4 defect level in a VHTR reactor to enable maintenance on heat transfer units and prevent excessive off-site releases during accident events. The total allowable defect level is subdivided between the three defect fractions. Fission product releases from DUF are thought to be an order of magnitude higher, for a given mass of uranium, than releases from dense kernels with either cracked layers or porous SiC, thus the allowable defect level for DUF has been assigned a lower limit than the other two fractions.

The particulars of the three defect fractions are discussed individually below. The levels of the defects, which are higher than intended, were evaluated by INL and the TCT prior to accepting the fuel for use for the AGR-5/6/7 experiment irradiation [8]. The reasons for accepting the fuel as-is include: 1) The fuel specifications were written for fuel to be used in a future VHTR reactor and not specifically written to safeguard data collection for an experiment irradiation. 2) The defect levels are high enough to complicate the analysis of fuel performance data but are not so high as to preclude “seeing” in-pile TRISO particle failures and collecting valuable data from the irradiation of the fuel. 3) Commercial VHTR fuel would need to undergo a “proof” test and not rely solely on AGR-5/6/7 data. 4) Refabricating the fuel would not guarantee conforming attributes. The required time to investigate the causal factors for the measured defect levels during overcoating and compaction, in addition to that needed for fuel refabrication, would have resulted in lost access to the Advanced Test Reactor Northeast flux trap, which is essential for irradiating a test train of the size designed for AGR-5/6/7.

Samples of overcoated TRISO particles for 40% PF compacts and compacts of both packing fractions were analyzed at ORNL for the three defect fractions to confirm or refute the compact defect data obtained by BWXT-NOG and to get a metric for the damage done during the deposition of the resinated-graphite overcoat on the TRISO. All “confirmational” data were not available when this document was originally written and issued. Revision 1 of this document incorporates the ORNL data. BWXT-NOG measured data are discounted because of anomalous results during the DLBL analyses and because the BWXT-NOG results for the defect fractions were statistically dissimilar from the ORNL results. Whereas ORNL developed the analytical methods and has more experience with the methods, the ORNL results are considered to be more accurate.

Dispersed Uranium Fraction

The DUF is a variable property used to estimate the quantity of uranium outside of the SiC coating layer in the TRISO and compact. It is postulated that uranium can be incorporated into the OPyC layer from contamination on the coater internal surfaces and frangible kernels or introduced as a contaminant in the resinated-graphite matrix. Impurities analyses of the components for the resinated-graphite matrix preclude incorporation of significant natural uranium, leaving the interior surface contamination and frangible kernels as the remaining sources.

DUF is quantified by assuming any quantity of uranium, leached from the liberated TRISO particles, amounting to less than a one-half kernel equivalent in the deconsolidating acid and in the preburn leachates or postburn leachates is from contamination and that anything greater than this is from exposed kernels in cracked or broken particles or from porous SiC layers. An intact OPyC layer is impervious to the acid used to deconsolidate the compact or leach prior to burning back the OPyC, so not much more than surface contamination should be detected with the preburn leaches. Burning back the OPyC liberates the remainder of the uranium embedded within the OPyC and makes it accessible to the postburn leach acids. The DUF is reported as the sum of the uranium recovered from the combined preburn and postburn leaches that is not otherwise attributed to either broken or porous coating layers.

Leach-burn-leach tests on the TRISO lot indicated that the compacts could fail the DUF as the preburn leach uranium fraction was in excess of the compact DUF specification. The TRISO DUF value has a wide uncertainty band because there was no means of segregating the dispersed uranium from exposed kernels in the sample. Because the estimated EKF and SiC defects for the TRISO were well below the compact specification by a wide margin, the TRISO was deemed acceptable for AGR-5/6/7 irradiation experiments. If the method for determining the DUF is accurate, then DUF should be unaffected by fuel compaction. Comparison of the DUF values calculated for the TRISO lot (98005

Preburn) and the "Confirmatory" analyses by ORNL did not measure an increase for either PF (Figure 1). After exclusion of suspect data and outliers, the DUF values were a little more than one-half of the specified maximum at the 95% confidence limits. The original conclusion (Revision 0 of this document) was that the upper 95% confidence limit for the 25% PF compacts exceeded the specification.

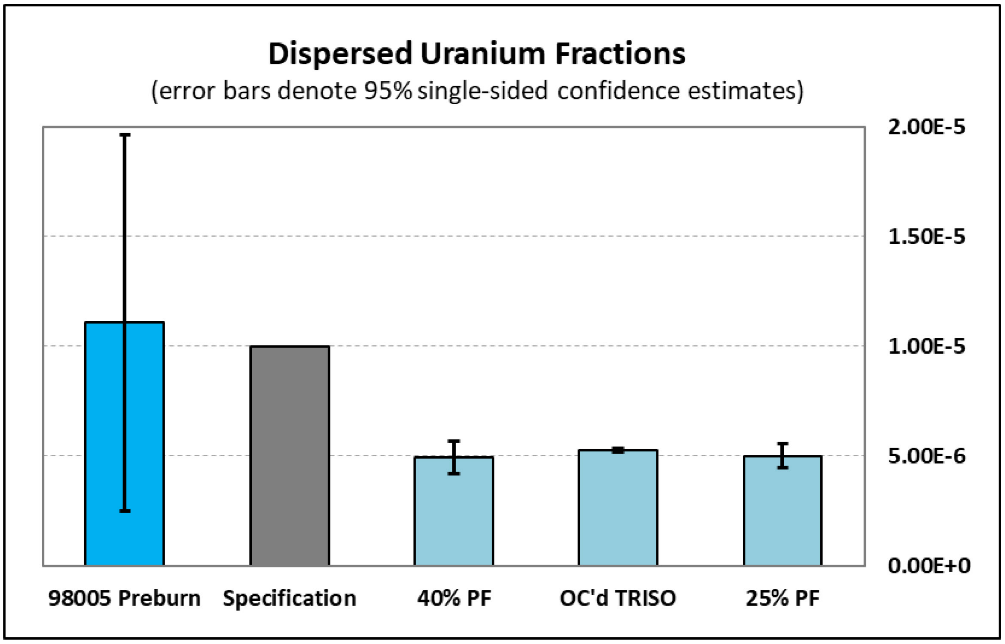

Figure 1. Dispersed uranium fraction results.

Exposed Kernel Fraction

The EKF is an attribute property estimating the fraction of damaged TRISO particles with cracked or broken coating layers such that the kernel is partially or totally exposed to the leachant. Even severely cracked coating layers and the fuel kernel, itself, retard the release of some fission products better than dispersed uranium. The TRISO particles are thought to incur damage during vacuum unloading of the TRISO, while overcoating particles with the resinated-graphite powder, and during fuel compact formation. The latter being the most significant source of damage and the process most likely to exacerbate minor defects from previous handling steps.

TRISO particle lot (J52R-16-98005) preburn leach data was about one-fifth of the EKF specification; leading to an expectation that the compacted fuel would conform to the specification even with modest damage during compaction. The measured EKF data for the 25% PF compacts passed the specification requirement as expected (see Figure 2). The EKF for the 40% PF compacts, however, failed to meet the specification with the upper 95% confidence limit. The data indicate that the process of overcoating the TRISO particles with resinated-graphite powder did not measurably contribute to the EKF defect. Compacting to a 25% PF also had little to no impact on the defect fraction, but that compacting at a 40% PF increased the EKF defect and was averse to compact quality. It is noted that the compacting process parameters used for AGR-5/6/7 fuel fabrication may not be fully optimized to reduce or prevent TRISO particle damage at the higher packing fraction.

Capsule 1 at the bottom of the test train and Capsule 5 at the top of the test train have compacts with 40% PF, collectively holding 114 fuel compacts. Capsules 2, 3, and 4 hold, collectively, 80 compacts at 25% PF. Irradiation of the 25% PF compacts is not impacted by the high EKF of the 40% PF compacts, provided capsule effluents remain independent. Capsule 1, with 90 compacts, is the most affected by the high EKF value. An assessment of the defect levels in Capsule 1 indicates that in-pile particle failures would still be detectable, and that valuable data can be obtained during the irradiation period and PIE. Based on combined ORNL data, one would expect 23^{+10}_{-8} exposed kernels in Capsule 1, which is similar to a single AGR-3/4 compact with 20 designed-to-fail particles.

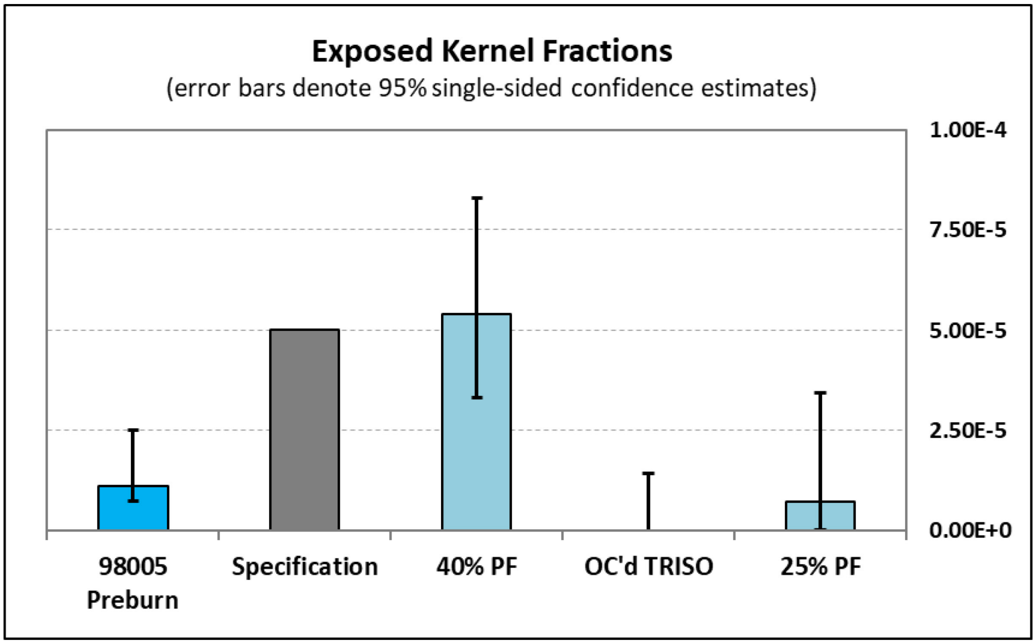

Figure 2. Exposed kernel fraction results.

Silicon Carbide Defect Fraction

The SDF is an attribute property estimating the fraction of particles with a porous or permeable SiC layer that formed during the deposition of that layer. Half of the allowable defect fraction for the reactor core is allocated to this SiC defect.

The fraction of truly porous SiC layers should not increase during overcoating, compaction, or thermal treatment and should be independent of the EKF. ORNL data show, however, that the mean SiC defect fraction may have increased for the 40% PF compacts relative to the TRISO particle (98005) defect level (see Figure 3). This phenomenon is yet to be explained. It's conceivable that weak SiC layers, in some particles, cracked under the mechanical stress of compaction without the OPyC layers breaking. Nonetheless, the defects are attributed (by the method) to a SiC defect and seem to correlate in some way with the EKF values (see Figure 2).

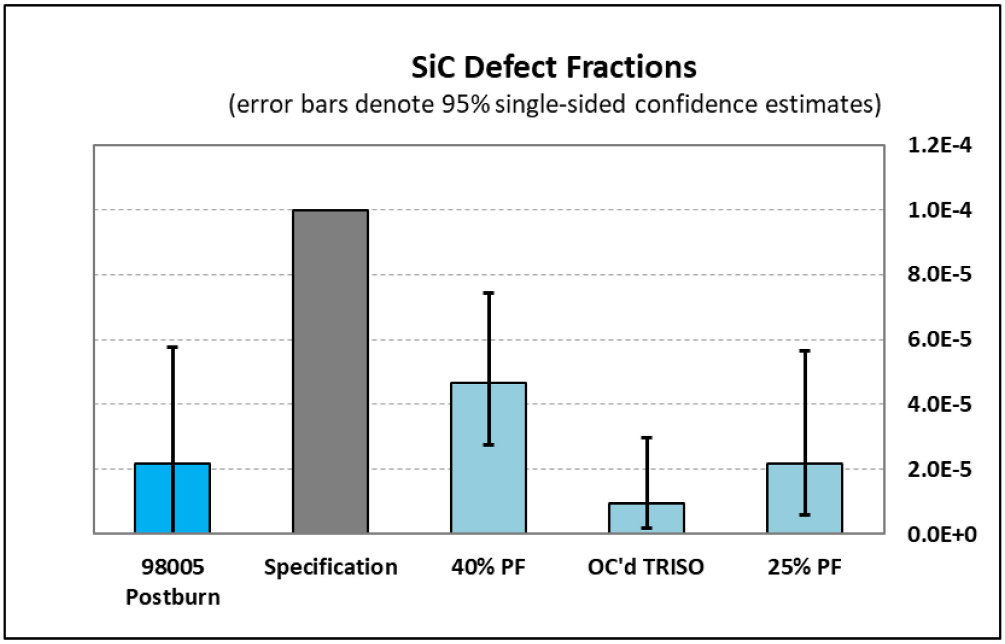

Figure 3. Silicon carbide defect fraction results.

Compact Impurities Units of Measure

The fuel specification [1] and the sampling plan [9] state that the units for impurities in the compacts be micrograms of the metal impurity outside of the SiC layer per compact. These analyses are performed on leachates generated during the compact DLBL procedure for quantifying the DUF, EKF, and SiC defect fractions. A five-compact "clutch" is deconsolidated and leached for this method, so the measured impurities apply to the clutch and not to the individual compacts. The measured impurities for all clutches were below the established detection limits for all elements of concern. BWX Technologies wrote: [10]

> "If the result reported by the analysis was greater than the LDL [lower detection limit], then dividing the result by five would produce the desired unit. However, for cases where the reported result was "less than LDL", then dividing the LDL value by five would not be appropriate. That is because the LDL had been determined by the reproducibility of the ppb [parts per billion] calibration curve, which was independent of the number of compacts under test. Therefore, for those samples with elemental results reported at the LDL value, the minimum division possible is the clutch. For those situations, the desired unit of µg/compact cannot be achieved."

INL accepts reporting the compact clutch results, in lieu of compact averages, for this case. The maximum possible impurity per compact is, arguably, no more than the values reported for the clutch, and thereby passing the specification requirement with adequate assurance.

CONCLUSIONS

After a statistical comparison of DLBL data from the BWXT-NOG and ORNL, it was determined that the data were dissimilar. Given that the ORNL developed the DLBL method and their data showed fewer anomalies, the BWXT-NOG DLBL data was rejected for the purpose of determining the defect fractions.

The BWXT-NOG impurities analyses, that had no anomalies, are accepted as valid for the purpose of demonstrating conformance with the related fuel specifications.

The consensus is that the OPyC thickness being below the specified range for the mean will not significantly contribute to in-pile particle failures, because the distribution is narrow and sufficiently high that the probability of having particles with less than a 20 µm thickness is very, very remote. Additionally, the specification for OPyC thickness was partially based on the assumption that the shrinking OPyC would compress the SiC layer. Mounted particles of irradiated fuel from the AGR-1 and AGR-2 fuels show that some OPyC layers may shrink radially outward and de-bond from the SiC layer.

The higher than expected EKF defects is undesirable and might not be acceptable for use in a VHTR reactor. The defect levels will make detecting in-pile particle failures more difficult during the AGR-5/6/7 experiment irradiations, but the judgement of subject matter experts is that our instrumentation will be able to discern the few particle failures that may occur and that valuable data will be obtained from the irradiation of the as-fabricated AGR-5/6/7 fuel compacts.

Despite the nonconforming properties of the TRISO particles and the compacted fuel, the fuel is of sufficient quality that useful and meaningful data can be collected during the AGR-5/6/7 irradiations and it is preferable to accept the fuel, as-is, rather than attempting to refabricate the fuel (with no guarantee of improvement) and risk losing access to the Advanced Test Reactor's northeast flux trap for the irradiation. The fuel compacts are acceptable for the purposes of the irradiations.

EPILOGUE

Data from the early irradiation showed that the AGR-5/6/7 fuel compacts were performing well and had similar fission gas release-to-birth ratios as the AGR-2 fuel during irradiation for compacts of both PFs. Subsequent fission product monitoring data for Capsule 1 indicate that a multitude of in-pile failures have occurred while the fuel compacts in Capsule 5 have seen very few observed failures in fuel with the same PF. The causes for the failures in Capsule 1 cannot be identified until a PIE is performed.

REFERENCES

1. SPC-1352, "AGR-5/6/7 Fuel Specification," Rev 8, March 2017.

2. BWXT Advanced Gas Reactor Program (AGR) Contract No. 107790, J52R, Industrial Fuel Fabrication and Development, Lot J52R-16-98005, Book 1 (BWXT N-74 Document Transmittal Form, "Revised Statistical Report on AGR 98005 Blend," Signed June 13, 2017.

3. ORNL/TM-2018/774, "Confirmatory LBL Analysis of AGR-5/6/7 Compacts and Overcoated Particles," Rev. 0., February 2018.

4. ORNL/TM-2019/1154, "Additional Confirmatory LBL Analysis of AGR-5/6/7 Compacts and-Overcoated Particles," Rev. 0., November 2019.

5. Technical Coordination Team February 1, 2017 videoconference summary issued February 8, 2017, "Advanced Gas Reactor Fuel Development and Qualification Program Technical Coordination Team – Videoconference Summaries, December 2016 - … ", pp. 170-192, https://htgr.inl.gov/htr/agrSite/TCT/SitePages/Technical%20Coordination%20Team.aspx

6. BWXT N-74 Document Transmittal Form, “Permission to Blend Mixture to Supply Material for AGR Compacting,” signed February 20, 2017 (Appendix B).

7. BWXT N-74 Document Transmittal Form, “QCDN for missing OPyC thickness,” signed March 21, 2017 (Appendix C).

8. Technical Coordination Team June 5, 2017 videoconference summary issued June 8, 2017“Advanced Gas Reactor Fuel Development and Qualification Program Technical Coordination Team – Videoconference Summaries, December 2016 - …”, pp. 193 – 241, https://htgr.inl.gov/htr/agrSite/TCT/SitePages/Technical%20Coordination%20Team.aspx

9. PLN-4352, “Statistical Sampling Plan for AGR-5/6/7 Fuel Materials,” Rev. 5, May 2016.

10. BWXT N-74 Document Transmittal Form, “Supplemental Information to the AGR 5/6/7 Compact Certification Package,” signed September 12, 2017, Re: Impurities analyses and reported units (Appendix D).

APPENDICES

A. Deleted

B. BWXT N-74 Document Transmittal Form, “Permission to Blend Mixture to Supply Material for AGR Compacting,” signed February 20, 2017.

C. BWXT N-74 Document Transmittal Form, “QCDN for missing OPyC thickness,” signed March 21, 2017.

D. BWXT N-74 Document Transmittal Form, “Supplemental Information to the AGR 5/6/7 Compact Certification Package,” signed September 12, 2017, Re: Impurities analyses and reported units.

Appendix A

Deleted

Appendix B

BWXT N-74 Document Transmittal Form, "Permission to Blend Mixture to Supply Material for AGR Compacting," signed February 20, 2017.

BWXT

DOCUMENT TRANSMITTAL FORM

N-74 Rev. 2 (02/26/16)
(CR-1044961-01) Page 1 of 1

CUSTOMER CONTRACT:	107790	CUSTOMER PROJECT:	Advanced Gas Reactor	BWXT PAC / NPN NO.	J52R

*** only ONE APPROVAL document per form ***

DESCRIPTION:	Permission to Blend Mixture to Supply Material for AGR Compacting		
BWXT DOCUMENT NUMBER:	NA	REVISION:	00
DATE: (Note to CDC: when FAX'd or delivered to BWXT mail room)	2 / 20 /17	TIME / # Pgs: (FAX only)	3

☒ FOR APPROVAL – NEED DATE 2/21/17 ☐ FOR INFORMATION

TO: company name: Idaho National Laboratory
address 1: PO Box 1625
address 2
city, state, country, zip: Idaho Falls, ID 83415
phone number: 208-526-3857
fax number: 208-526-2930
in care of (c/o)

FROM: (see originator's name, phone, mail code below)
BWXT NOG-L
USPS P.O. Box 785 Lynchburg, VA, USA 24505-0785
courier *Mt. Athos Road Lynchburg, VA, USA 24504*
FAX 434/522-5410

ATTN: Doug Marshall

REMARKS:

Last Friday (2/17/17) I reported to you the leach burn leach results for AGR run J52O-16-93170B. The results were a significant improvement compared to run 93170A and showed that the resieving was successful. Please confirm that it is acceptable to use the 93170B material as part of the TRISO lot blend to be used for AGR compacting.

BWXT ORIGINATOR:

NAME:	EXT / MAIL CODE:	SIGNATURE / DATE:
Joseph Keeley	6177 / 61	Joseph Keeley / 2-20-17

BWXT MANAGEMENT:

NAME:	EXT / MAIL CODE:	SIGNATURE / DATE:
David Navolio	6450/61	D.W. Nav[illegible] 2/20/17

CUSTOMER DISPOSITION: (customer may substitute equivalent form)

☒ ACCEPTED ☐ ACCEPTED AS NOTED ☐ NOT ACCEPTED

COMMENTS:

NAME:	EXT / MAIL CODE:	SIGNATURE / DATE:
Douglas W. Marshall		DW Marshall 2/20/2017

ADMINISTRATIVE REVIEW: [illegible signature] 2/20/2017

Keeley, Joseph T (Joe)

From: Douglas Marshall <opamarshall@gmail.com>
Sent: Friday, February 17, 2017 10:36 AM
To: Keeley, Joseph T (Joe); Marshall, Douglas W
Cc: Navolio, David W; Niedzialek, Scott E; Richardson, W C (Clay); Jones, Aaron C; Mulreany, Robert E
Subject: EXTERNAL:Re: FW: AGR Burn Leach results

Joe, et al.,

The data look far better than I had hoped for. Both leaches pass the compact specifications at 95% confidence. We saw, as you stated earlier, more than 50% reduction of the pre-burn leach and nearly 75% reduction in the post-burn leach calculated at 95% confidences.

	Pre Burn Leach				Post Burn Leach			
	Total				Total			
	Failed Part	# Particles	Failure Fraction	95% Conf.	Failed Part	# Particles	Failure Fraction	95% Conf.
93165A	2.21	330000	6.70E-06	1.91E-05	2.78	165000	1.68E-05	4.70E-06
93166RA	1.57	318000	4.94E-06	1.98E-05	0.92	159000	5.79E-06	2.98E-05
93168A	3.71	324000	1.15E-05	2.83E-05	2.27	162000	1.40E-05	3.89E-05
93169A	10.37	324000	3.20E-05	[illegible]	2.88	162000	1.77E-05	4.79E-05
93170A	[redacted]	[redacted]	2.15E-05	4.06E-05	[redacted]	[redacted]	2.28E-04	[illegible]
93170A extra	[redacted]	[redacted]	1.06E-04	[illegible]	[redacted]	[redacted]	1.67E-04	[illegible]
	[redacted]	[redacted]	4.26E-05	[illegible]	[redacted]	[redacted]	2.08E-04	[illegible]
93170B	[illegible]	[illegible]	1.06E-05	3.89E-05	[illegible]	[illegible]	2.49E-05	3.65E-05
93170B	[illegible]	[illegible]	1.48E-05	3.89E-05	[illegible]	[illegible]	6.22E-05	[illegible]
93170B total	[redacted]	[redacted]	1.27E-05	2.83E-05	[redacted]	[redacted]	4.35E-05	6.75E-05

Please use 93170 as part of the LEU TRISO lot blend.

Douglas Marshall

On Fri, Feb 17, 2017 at 7:18 AM, Keeley, Joseph T (Joe) <jtkeeley@bwxt.com> wrote:

I made an error and corrected it in the table. I added the failure fractions and what I should have totaled all failed parts and divided by the total number of particles. The conclusion is still the same, we made significant improvements.

From: Keeley, Joseph T (Joe)
Sent: Friday, February 17, 2017 8:49 AM
To: 'opamarshall@gmail.com'
Cc: Navolio, David W; Niedzialek, Scott E; Richardson, W C (Clay); Jones, Aaron C; Mulreany, Robert E
Subject: AGR Burn Leach results

Doug,

I've updated the results table with the burn leach results for 93170B. We made significant improvements to both the pre-leach and post-leach results and are now with specification. I know Dave set a teleconference for 11 am, but if the results look good to you and we can go ahead processing, please call me sooner (434-522-6177). I'd like to try and get our Operations group working on this as soon as possible.

Thanks,

Joe

Appendix C

BWXT N-74 Document Transmittal Form, "QCDN for missing OPyC thickness," signed March 21, 2017.

BWXT

DOCUMENT TRANSMITTAL FORM

N-74 Rev. 2 (02/26/16)
(CR-1044961-01) Page 1 of 1

CUSTOMER CONTRACT:	107790	CUSTOMER PROJECT:	Advanced Gas Reactor	BWXT PAC / NPN NO.	J52R

*** only *ONE* APPROVAL document per form ***

DESCRIPTION:	QCDN for missing OPyC thickness
BWXT DOCUMENT NUMBER:	NA
REVISION:	00
DATE: (Note to CDC: when FAX'd or delivered to BWXT mail room)	2 / 20 /17
TIME / # Pgs: (FAX only)	7:55 / 2

☒ FOR APPROVAL – NEED DATE 3/22/17 ☐ FOR INFORMATION

TO:
company name: Idaho National Laboratory
address 1: PO Box 1625
address 2
city, state, country, zip: Idaho Falls, ID 83415
phone number: 208-526-3857
fax number: 208-526-2930
in care of (c/o)

FROM: (see originator's name, phone, mailcode below)
BWXT NOG-L
USPS: P.O. Box 785, Lynchburg, VA, USA 24505-0785
courier: *Mt. Athos Road, Lynchburg, VA, USA 24504*
FAX: 434/522-5410

ATTN: Doug Marshall

REMARKS:
Attached is the QCDN for missing the OPyC thickness on the AGR coating blend J52R-16-98005 (approved by INL on February 8, 2017). The violation was noted in the Met Lab samples that were split from Containers #1 and #2 of the blend. FYI, the weighted average I reported in the QCDN came from the batch weights used in the blend and the thickness data from the "A" material. We did not collect thickness data on the "B" material (after the resieve). If you have any questions please contact Dave or me.

BWXT ORIGINATOR:

NAME:	EXT / MAIL CODE:	SIGNATURE / DATE:
Joseph Keeley	6177 / 61	Joseph Keeley 3-21-17

BWXT MANAGEMENT:

NAME:	EXT / MAIL CODE:	SIGNATURE / DATE:
David Navolio	6450 / 61	D. W. Navolio 3/21/17

CUSTOMER DISPOSITION: *(customer may substitute equivalent form)*

☐ ACCEPTED ☒ ACCEPTED AS NOTED ☐ NOT ACCEPTED

COMMENTS: Recent post-irradiation examination observations from previous AGR experiments suggest that the measured OPyC thickness on TRISO batches composing lot J52R-16-98005 will not have a deleterious effect on in-pile performance. J52R-16-98005 is accepted for use as is.

NAME:	EXT / MAIL CODE:	SIGNATURE / DATE:
Douglas Marshall		D W Marshall 3/21/17

ADMINISTRATIVE REVIEW: KA Hartless 3/21/17
KA Hartless

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Quality Control Deficiency Notice (U)

Page 1 of 1
Q11-127
Rev 01
March 11, 2016
CR-1044961-01

QCDN No. J52-004 Dept / Section UPRR QC

Material ID: J52R-16-98005	Stage of Processing: Blend
Contract No. : 107790	Contract Name: AGR
Requirement Violated: OPyC thickness	Data Attached ____YES X NO

Route Card Seq. No.	Defect Description	Actual Requirement
N/A	Sample Data 1 = 35.1	36 - 44
N/A	Sample Data 2 = 34.5	36 - 44

Correction Action Required Applicable________ Not Applicable X

Responsible Area: Engineering________ Manufacturing________ QC________

Corrective Action Comments________ A

CA # N/A Other ________

Corrective Action By________ Date ________

QA Engr.________ Date________

____ Reject
____ Continue Process, No Further Evaluation Necessary
X Submit for Customer Disposition
____ Continue Process, Evaluate at________ RC________ RC Step________
____ Repair / Rework Via RC________ OP________ Other________
____ Hold (By________ Date________) ____ Release (By________ Date________)

B

Engr. Comments / Actions:
The blend was made from four batches (93165B, 3057.1 g, 30.3 μm; 93168B, 2995.8 g, 38.5 μm; 93169B, 2747.6 g, 36.0 μm an 93170B, 2845.4g, 35.6 μm). The thickness of 93165B and 93170B were low. The weighted average thickness was calculated to be 35.1 μm.

Process Engr. [signature] Date 3/20/17 QA Engr. [signature] Date 3/20/17

Customer Disposition:

Approve as is X

Reject____

Approve with the following Conditions ________

C

Customer Signature [signature] Date 3/21/2017

Process Engr.________ Date________ QA Engr.________ Date________

Appendix D

BWXT N-74 Document Transmittal Form, "Supplemental Information to the AGR 5/6/7 Compact Certification Package," signed September 12, 2017, Re: Impurities analyses and reported units.

BWXT

DOCUMENT TRANSMITTAL FORM

N-74 Rev. 2 (02/26/16)
(CR-1044961-01) Page 1 of 1

CUSTOMER CONTRACT:	107790	CUSTOMER PROJECT:	Advanced Gas Reactor	BWXT PAC / NPN NO.	J52R

*** only *ONE* APPROVAL document per form ***

DESCRIPTION:	Supplemental Information to the AGR 5/6/7 Compact Certification Package
BWXT DOCUMENT NUMBER:	N/A
REVISION:	
DATE: (*Note to CDC:* when FAX'd or delivered to BWXT mail room)	09/11/2017
TIME / # Pgs: (FAX only)	10:20am / 3

☒ FOR APPROVAL -- NEED DATE 9/12/2017 ☐ FOR INFORMATION

TO: Idaho National Laboratory
P. O. Box 1625
Idaho Falls, ID 83415
phone number 208-526-3657
fax number 208-526-2930

FROM: (see originator's name, phone, mail code below)
BWXT NOG-L
USPS P.O. Box 785
Lynchburg, VA, USA 24505-0785
courier *Mt. Athos Road*
Lynchburg, VA, USA 24504
FAX 434/522-6410

ATTN: Doug Marshall

REMARKS:

The attached memorandum clarifies BWXT's impurities testing approach. Please note that the outstanding statistical calculation for iron and transition metals will be reported as micrograms per clutch. These calculations will begin as soon as BWXT has INL approval to proceed.

BWXT ORIGINATOR:

NAME:	EXT / MAIL CODE:	SIGNATURE / DATE:
Alex Tilton	5394/ 061	Alexander Tilton 9/11/2017

BWXT MANAGEMENT:

NAME:	EXT / MAIL CODE:	SIGNATURE / DATE:
Dave Navolio	6450/ 061	D.W. Navolio 9/11/2017

CUSTOMER DISPOSITION: *(customer may substitute equivalent form)*

☒ ACCEPTED ☐ ACCEPTED AS NOTED ☐ NOT ACCEPTED

COMMENTS:

NAME:	EXT / MAIL CODE:	SIGNATURE / DATE:
Douglas W. Marshall		DW Marshall 9/13/17

ADMINISTRATIVE REVIEW: Kelly A. Hartless 9/11/17

BWX Technologies, Inc.

Testing of the impurities present in the AGR 5/6/7 Compacts was specified in INL Documents SPC-1352 and PLN-4352. The limits for each metallic impurity are listed in the respective table in the two documents. To summarize the process that is used for the impurity testing, a set of randomly sampled compacts from each lot are selected. The number of compacts selected for each lot is determined by the number of TRISO particles per compact and the statistical confidence required for the reported results. Once the compacts have been removed from the lot, they are placed into one container and transferred to the BWXT Chemistry Laboratory. The compacts are randomly removed from the container into groups of five compacts, known as a clutch of compacts, or clutch. The compacts in each clutch are deconsolidated into TRISO particles and residual solids using an electrochemical process with nitric acid. Following deconsolidation, the solution is leached at just below the boiling point of the acid. The solids and the TRISO particles are separated from the solution and all the solids (including the TRISO particles) are burned to remove the excess carbon. Following the burn, the residual solids are again leached in hot nitric acid. Samples of the solution prior to the burn and post-burn are retained for impurity testing. The impurity testing is done using an ICP-MS (Inductively Coupled Plasma Mass Spectrometer). Each sample (pre-burn and post-burn by clutch) is first diluted using a dilution factor of 200 to minimize the corrosive effects of the acid inside the torch. The diluted sample is injected into the torch of the ICP-MS (Nu Instruments Attom HR ICP-MS, Figure 1) and the analyzer is tuned to the specified elements. The mass spectrometer records the counts associated with each specified elements. The conversion of the counts to concentration is done using a parts-per-billion (ppb) calibration curve obtained using standards. The ppb concentration corresponding to the accumulated counts is further corrected for the dilution factor prior to being reported by the unit. A lower detection limit (LDL) for each element is established by the chemist based upon the precision and confidence found at the lower concentration levels found in the samples. If the elemental value for a given sample is reported to be less than the LDL, the LDL value is used. If the elemental value is greater than the LDL, then the reported value is used. Note that during the previous discussion, the sample under analysis was obtained from a clutch of five compacts. The requirement in the two INL documents referenced above was that the results be reported in micrograms per compact (µg/compact). If the result reported by the analysis was greater than the LDL, then dividing the result by five would produce the desired unit. However, for cases where the reported result was "less than LDL", then dividing the LDL value by five would not be appropriate. That is

Figure 1: Attom High Resolution ICP-MS

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BWX Technologies, Inc.

because the LDL had been determined by the reproducibility of the ppb calibration curve, which was independent of the number of compacts under test. Therefore, for those samples with elemental results reported at the LDL value, the minimum division possible is the clutch. For those situations, the desired unit of µg/compact cannot be achieved.

Table 1 below demonstrates that the results for iron and the Transition Metals were all below the LDL for all samples. Note also in the table that the results are listed as µg/clutch. The results in the table will be forwarded to the BWXT Statistical Department for the remaining statistical calculations, as defined in the INL Specifications.

Lot	Sample #	MS Detection Limit (µg/clutch)					
		Fe	Co	Cr	Mn	Ni	Sum Co, Cr, Mn, Ni
J52R-16-14154C	1	<5	<10	<25	<10	<10	<55
J52R-16-14154C	2	<5	<10	<25	<10	<10	<55
J52R-16-14154C	3	<5	<10	<25	<10	<10	<55
J52R-16-14154C	4	<5	<10	<25	<10	<10	<55
J52R-16-14154C	5	<5	<10	<25	<10	<10	<55
J52R-16-14154C	6	<5	<10	<25	<10	<10	<55
J52R-16-14155C	1	<5	<10	<25	<10	<10	<55
J52R-16-14155C	2	<5	<10	<25	<10	<10	<55
J52R-16-14155C	3	<5	<10	<25	<10	<10	<55
J52R-16-14155C	4	<5	<10	<25	<10	<10	<55
J52R-16-14155C	5	<5	<10	<25	<10	<10	<55
J52R-16-14155C	6	<5	<10	<25	<10	<10	<55
J52R-16-14156C	1	<5	<10	<25	<10	<10	<55
J52R-16-14156C	2	<5	<10	<25	<10	<10	<55
J52R-16-14156C	3	<5	<10	<25	<10	<10	<55
J52R-16-14156C	4	<5	<10	<25	<10	<10	<55
J52R-16-14156C	5	<5	<10	<25	<10	<10	<55
J52R-16-14156C	6	<5	<10	<25	<10	<10	<55
J52R-16-14157C	1	<5	<10	<25	<10	<10	<55
J52R-16-14157C	2	<5	<10	<25	<10	<10	<55
J52R-16-14157C	3	<5	<10	<25	<10	<10	<55
J52R-16-14157C	4	<5	<10	<25	<10	<10	<55
J52R-16-14157C	5	<5	<10	<25	<10	<10	<55
J52R-16-14157C	6	<5	<10	<25	<10	<10	<55

Table 1: Summary Impurity Data