ORNL/TM-2017/407

Vanderbilt University Gamma Irradiation of Nano-modified Concrete (2017 Milestone Report)

Kory Linton
Aaron Selby
Yonathan Reches
Geoffrey Deichert
Kurt Terrani

August 14, 2017

Approved for public release.

OAK RIDGE NATIONAL LABORATORY
MANAGED BY UT-BATTELLE FOR THE US DEPARTMENT OF ENERGY

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Materials Science and Technology Division

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ACKNOWLEDGMENTS

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ACRONYMS

EABD	Experiment Authorization Bases Document
FY	fiscal year
HFIR	High Flux Isotope Reactor
NE	Office of Nuclear Energy
NSUF	Nuclear Science User Facility
ORNL	Oak Ridge National Laboratory
QA	quality assurance
UNF	used nuclear fuel
USQD	Unreviewed Safety Question Determination
XRF	x-ray fluorescence

EXECUTIVE SUMMARY

This document outlines the irradiation of concrete specimens in the Gamma Irradiation Facility in the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL). Two gamma irradiation runs were performed in July of 2017 on 18 reference mortar bar specimens, 26 reference cement paste bar specimens, and 28 reference cement paste tab specimens to determine the dose and temperature response of the specimens in the gamma irradiation environment. Specimens from the first two gamma irradiations were surveyed and released to Vanderbilt University. The temperature and dose information obtained informs the test parameters of the final two gamma irradiations of nano-modified concrete planned for FY 2018.

1. INTRODUCTION

1.1 BACKGROUND

In 2016, Vanderbilt University principal investigator Dr. Florence Sanchez and graduate student Yonathan Reches successfully proposed to study the gamma radiation microstructural and mechanical response of dry state concrete under a Nuclear Science User Facility (NSUF) access call. By studying the effect of gamma irradiation on nano-modified concretes, Dr. Sanchez hopes to fill gaps in the fundamental understanding of the phyisco-chemical processes and contribute to the Office of Nuclear Energy (NE) research goal to deploy radiation-resistant materials that extend service life and performance of used nuclear fuel (UNF) dry storage systems.

This award provided funded access to Oak Ridge National Laboratory (ORNL) staff engineers and operations to develop the appropriate experimental design, write the safety documentation, and ultimately perform the experiment in the High Flux Isotope Reactor (HFIR) using irradiation baskets filled with control and nano-modified concrete samples. The samples, after survey and release from HFIR, will be characterized at Vanderbilt University and US Army Engineer Research and Development Center. This report documents the preparations, irradiation experiments, and release of specimens back to Vanderbilt in successful completion of a PICS Milestone M3UF-17OR0207182 – “Complete Gamma Irradiation of Two Holders and Release Specimens to VU.”

1.1.1 HFIR Gamma Irradiation Facility

To expose test specimens to a total dose of 40 MGy in less than 1 month, Dr. Sanchez proposed using the HFIR Gamma Irradiation Facility, which accommodates gamma dose rates up to 1.8E+08 Rad/hr. This facility is a stainless-steel chamber designed for insertion into spent fuel elements located in the HFIR pool (Figure 1). Instrumentation required for the experiment is provided through an “umbilical,” providing inert gas in the chamber and temperature monitoring using multiple thermocouples along the vertical axis of the experiment canister.

Figure 1. HFIR Gamma Irradiation Facility. (Stock Photo: https://neutrons.ornl.gov/hfir/gamma-irradiation.)

2. IRRADIATION

2.1 SAFETY DOCUMENTATION AND APPROVALS

Prior to scheduling the experiment, Vanderbilt provided the elemental analysis documentation of all test specimens, design drawings of the canister, and related purchase history of all parts and specimen supplies. HFIR Quality Assurance also performed XRF (X-ray fluorescence) measurements, as shown in Figure 2, on all parts of the custom canister, confirming that 300-series stainless steel was used throughout. HFIR engineers developed the Experimental Authorization Bases Document (EABD) and Unreviewed Safety Question Determination (USQD) Document, giving approval to proceed with the experiment, as provided in Appendix A and B, respectively.

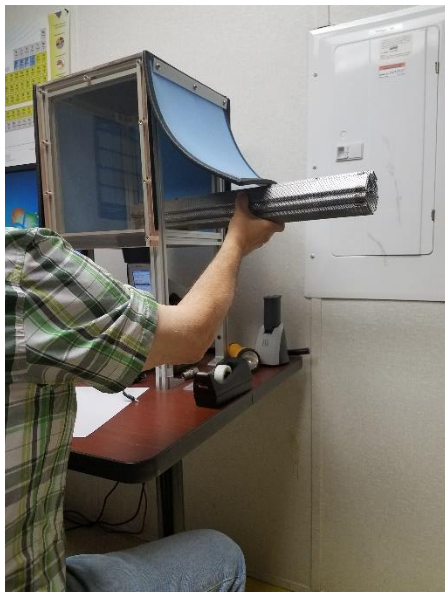

Figure 2. XRF Measurement of custom canister by HFIR QA.

2.2 ASSEMBLY OF THE IRRADIATION CANISTER

For the irradiation planned in July 2017, Yonathan Reches provided 26 reference cement paste and 18 reference mortar bar specimens (Figure 3) and 28 reference cement paste tab specimens (Figure 4). The custom-designed irradiation canister (Figure 5) was configured with six Omega type K thermocouples for temperature measurement (Figure 6) and six Far West Technology, Inc. radiachromic dosimeters (Figure 7).

Figure 3. Reference cement paste and reference mortar block specimens.

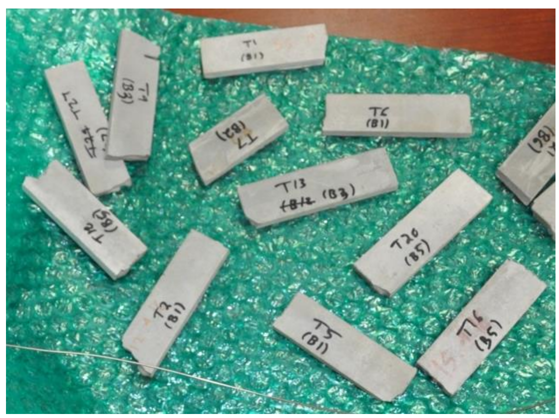

Figure 4. Reference cement paste tab specimens.

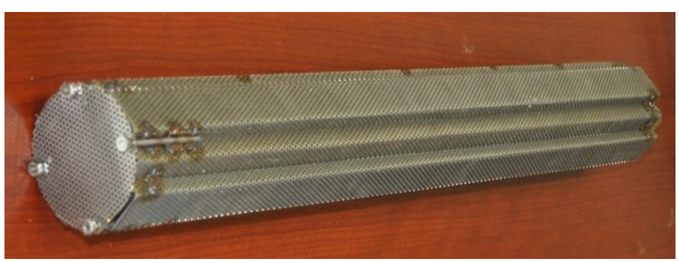

Figure 5. Custom-designed irradiation canister.

Figure 6. Omega type K thermocouple.

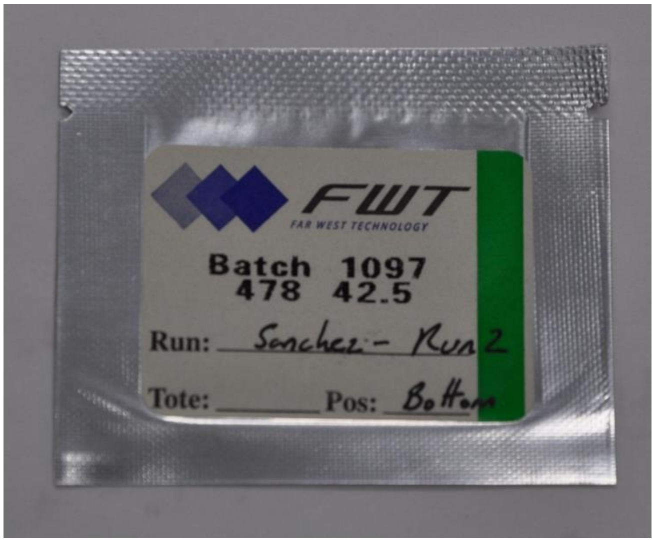

Figure 7. Far West Technology Inc. radiachromic dosimeter.

The bar specimens were loaded into the inner chamber of the canister (Figure 8), and the tab specimens loaded into the outer peripheral chambers (Figure 9). Figure 10 shows the top of the canister after all the specimens were loaded. The thermocouples were inserted through the holes in the canister (Table 1) to measure the temperature at predefined points and held in place with wire (Figure 11). Five radiachromic dosimeters were placed on the outside of the canister at heights of 1.5, 3.3, 11, 15, and 20.5 in. from the bottom. One dosimeter was placed inside of the top samples in the canister. The entire assembled canister was placed into a holder. Figure 12 shows graduate student Yonathan Reches standing in the HFIR Reactor Bay Area with the assembled canister.

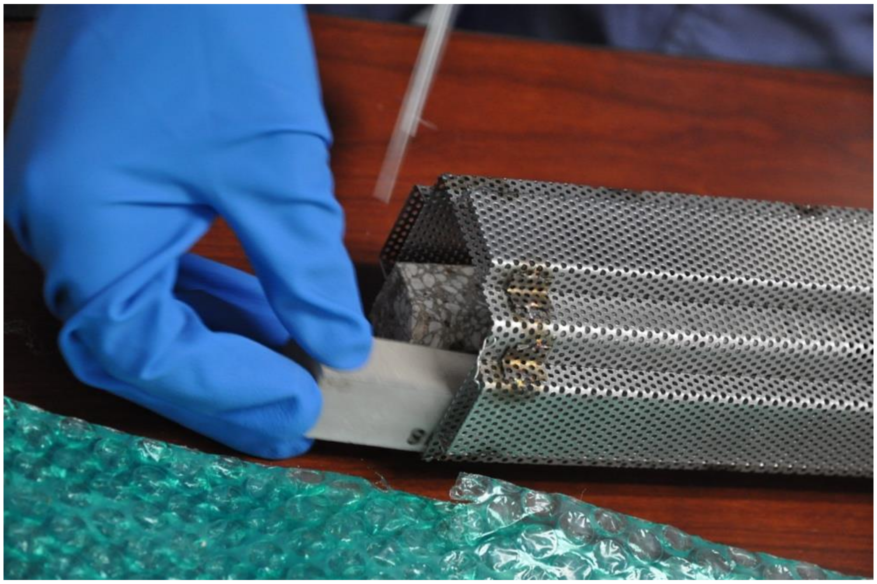

Figure 8. Bar specimens were loaded into the inner chamber of the canister.

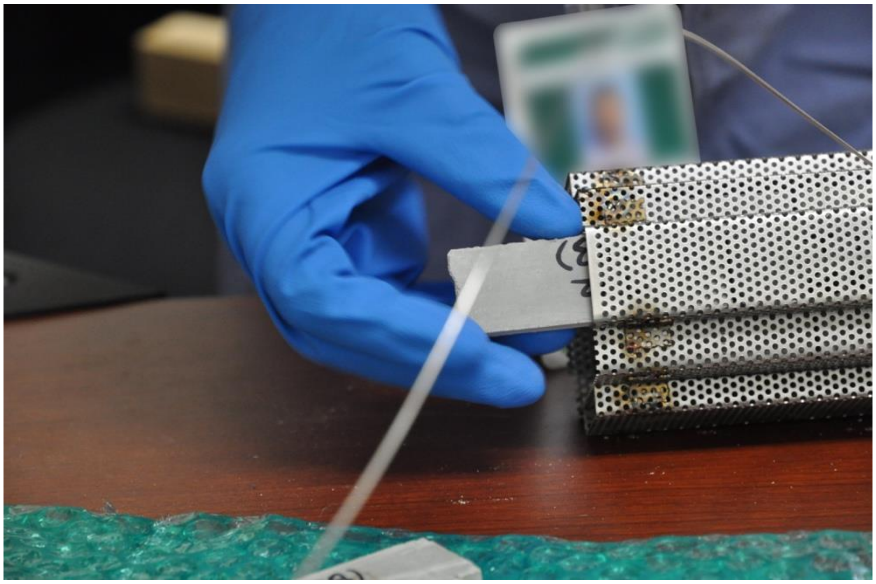

Figure 9. Tab specimens were loaded into the outer peripheral chambers of the canister.

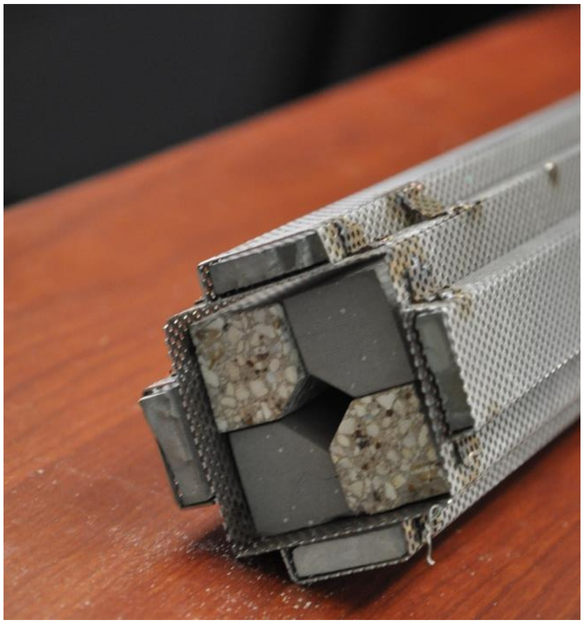

Figure 10. View of the top of the canister after all specimens were loaded.

Figure 11. A thermocouple was inserted into specimen assembly through a hole in the canister.

Table 1. Location of thermocouples

Thermocouple Number	Location
1	Bottom Inside
2	Middle Inside
3	Middle Outside
4	Top Inside
5	Top Middle
6	Top Outside

Figure 12. Graduate student Yonathan Reches in the HFIR Reactor Bay Area with his nano-modified concrete experiment ready for insertion into the gamma facility.

2.3 FIRST IRRADIATION

The assembled canister was taken out of the holder to load into the HFIR spent fuel irradiation chamber, as shown in Figure 13. The top sections of the irradiation chamber were then assembled, and the thermocouples and flow gas tube were connected to the umbilical cord that runs to the control area. This assembly procedure is shown in Figure 14.

Figure 13. The canister was loaded into the HFIR spent fuel irradiation chamber.

Figure 14. The assembly of the HFIR spent fuel irradiation chamber.

Once the HFIR spent fuel irradiation chamber was fully assembled, the chamber was pressurized with nitrogen flow gas and leak tests were performed, after which the irradiation chamber was lowered down into the HFIR spent fuel element 473, as shown in Figure 15.

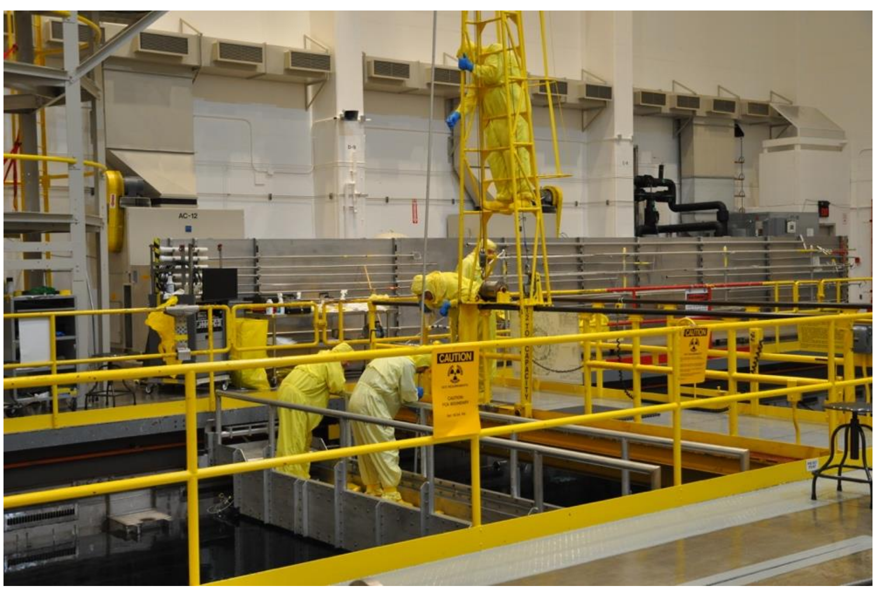

Figure 15. The irradiation chamber was lowered down into the spent fuel element.

For the first irradiation, with the intended dose of 10 MRad, the chamber was lowered into the spent fuel element 473 at 16:12 07/19/2017 and was removed at 16:43 07/19/2017 for a total irradiation time of 31 minutes. The temperatures were monitored in the control area and relayed to the experiment team in the reactor bay by radio. During the first irradiation run, one thermocouple failed and another delivered suspicious values. After removing the irradiation chamber from the spent fuel assembly, it was determined that the first thermocouple was faulty and the second thermocouple had an external short.

2.4 SECOND IRRADIATION

Dosimetry from the first irradiation was removed and the faulty thermocouple was replaced prior to loading the canister into the irradiation chamber. Once the top sections were loaded, the thermocouples were tested, confirming that five were functioning and one had an external short. After pressurizing the chamber and performing the gas flow leak checks, the irradiation chamber was lowered into spent fuel element 473.

The second irradiation began at approximately 18:40 07/19/2017. The irradiation chamber was removed from the spent fuel element at 02:30 07/26/2017 for a total irradiation time of 6 days, 7 hours, and 50 minutes (received an approximate dose of 2.49×10^{9} Rad). The irradiation chamber was pulled out of the pool, placed poolside (Figure 16), and after an appropriate cooling period, was depressurized, opened, and the canister extracted (Figure 17). The thermocouples were removed (Figure 18), and the canister was taken by a radiological control technician to check for activity and contamination. The radiological technician determined the samples to be free of radioactivity and contamination, and a green tag, as shown in Figure 19, was issued so that the samples could be removed from ORNL and taken back to Vanderbilt by Reches.

Figure 16. The irradiation chamber was removed from the pool and placed poolside.

Figure 17. The canister was removed from the irradiation chamber.

Figure 18. The thermocouples were removed from the canister.

Figure 19. A green tag was issued so that the samples could be taken back to Vanderbilt for analysis.

3. RESULTS

3.1 FIRST IRRADIATION RESULTS

During the first irradiation, temperatures were monitored by a technician and radioed into the HFIR bay for recording. The temperatures after 13 minutes of irradiation are shown in Table 2. It was determined that the temperature had not reached the maximum temperature by the end of the run as temperatures continued to climb until the end of the 31 minute irradiation.

Table 2. Temperatures from the first run after 13 minutes of irradiation

Temperature 1	Temperature 2	Temperature 3	Temperature 4	Temperature 5	Temperature 6
NA	39.0 ºC*	141.8 ºC	89.2 ºC	86.6 ºC	84.7 ºC

* It was determined after the run that Temperature 2 was faulty.

After the radiachromic dosimeters were removed, they were opened and inserted into the radiachromic reader for dosimetry measurement (Figure 20). The measured dose can be seen in Table 3.

Figure 20. Far West Technology Inc. Radiachromic Reader for dosimetry measurements.

Table 3. Dosimetry information from the first run

Dosimeter	Distance from bottom of canister	Radial location	Dose (MRad)	Average dose rate (R/hr)
1	20.5 in	Approximately centered	4.44	8.59×10^{6}
2	20.5 in	Exterior of canister	8.69	1.68×10^{7}
3	15 in	Exterior of canister	4.18	8.09×10^{6}
4	9.25 in (fuel centerline)	Exterior of canister	2.38	4.60×10^{6}
5	5.5 in	Exterior of canister	10.83	2.10×10^{7}
6	1.5 in	Exterior of canister	16.05	3.11×10^{7}

The dosimetry showed higher doses were received at the top and bottom and lower dose were received in the centerline, where the highest dose was expected. These unexpected results will require further analysis.

3.2 SECOND IRRADIATION RESULTS

Figure 21 shows the temperatures for the second run as a function of time and a trend of a rapid increase in temperature in the first hours of irradiation followed by a stabilization and then a slow decrease in temperature over a much longer time frame. The maximum temperature found at the centerline was recorded as 354.6ºC, with the temperatures decreasing away from the center.

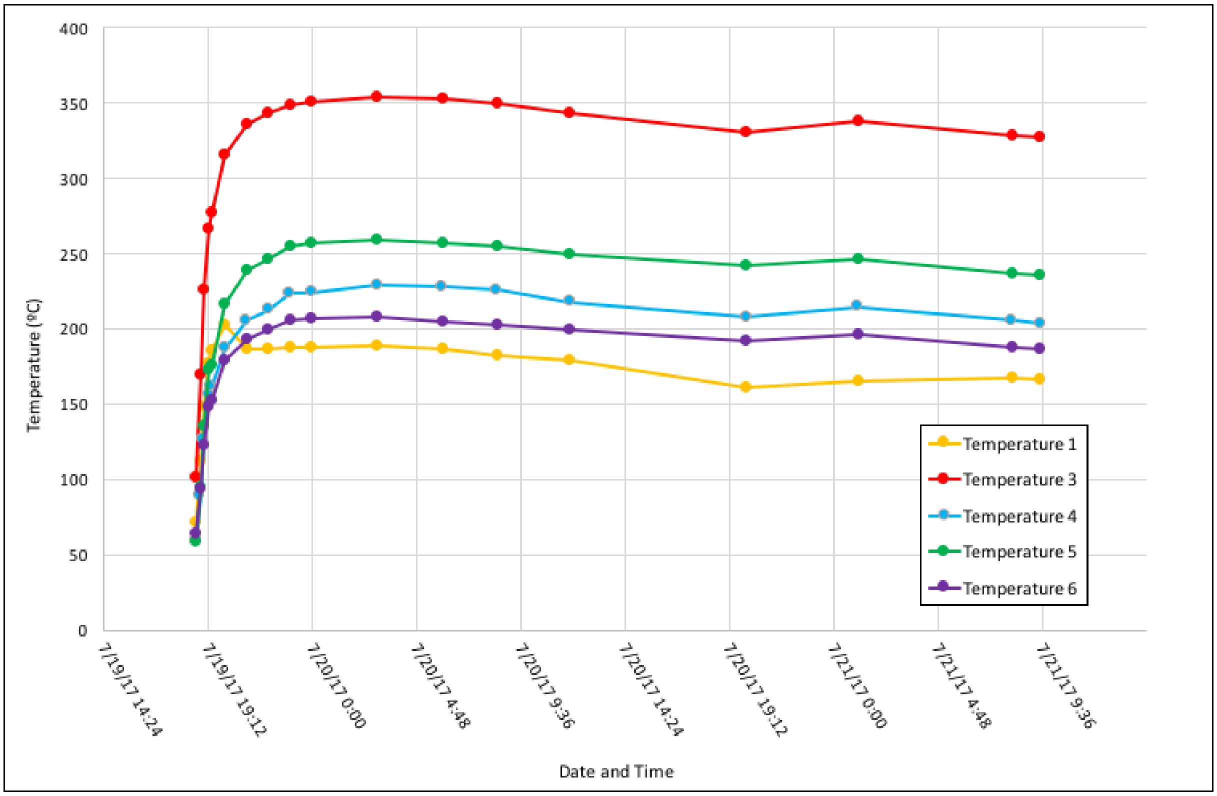

Figure 21. Temperature as a function of time for the second run.

4. CONCLUSION AND PATH FORWARD

The dosimetry and temperature data from these preliminary irradiations will aid in determining the conditions for the final two irradiations. The proposed work calls for two unique temperatures at the same total dose. To achieve the two temperatures the third irradiation containing the highest pedigree nano-modified samples will be performed using a spent fuel element with a similar decay as the first two runs already performed. After irradiation, the holder will be removed, new specimens loaded and the lower temperature for the final irradiation will be achieved by using an older fuel element over a longer period. The higher temperature run will be performed on a spent fuel element with a decay time of approximately 15 days, and the samples will be irradiated for approximately 17 days before removal from the spent fuel bundle. After reviewing the temperatures from the 17-day irradiation, two options will be available: (1) perform the final irradiation using the same spent fuel element after approximately 32 days of total decay or (2) move to a spent fuel element with a much longer decay time as there is a direct correlation between the decay time and the irradiation temperature.

Each irradiation will proceed according to the following procedure:

1) A dose run with no samples to find the dose profile of the spent fuel element before the 4000 MRad

2) A dose run with samples to find the dose profile of the spent fuel element with the loaded irradiation chamber before the 4000 MRad

3) The full 4000 MRad irradiation with the samples

4) A dose run with no samples to find the dose profile of the spent fuel element at the end of the 4000 MRad irradiation

These steps will be performed for both the high-temperature and the cool-temperature runs.

APPENDIX A. EXPERIMENT AUTHORIZATION BASES DOCUMENT

APPENDIX A. EXPERIMENT AUTHORIZATION BASES DOCUMENT

Experiment Authorization Bases Document: EABD-HFIR-2008-003	Rev 7
Title: HFIR Gamma Irradiation Facility Sample Irradiations Prepared By: C. S. Robison Date: 12/21/2016	Page 1 of 10

Purpose: The Experiment Authorization Bases Document is used to 1) define the approved experiment design and operating bases and 2) to accept the as-built experiment for installation and irradiation.

Section 1: Approved Experiment Configuration:
Note: Section 1 defines the approved experiment design.

This EABD is applicable to sample material irradiations conducted in the HFIR Gamma Irradiation Facility (reference DCM-HFIR-2005-083, MM-HFIR-2008-197, MM-HFIR-2009-123-R0).

Specific material configurations approved for irradiation in the chamber are listed in table 1.

This EABD is also applicable to sample material configurations other than those listed in table 1 provided all of the following conditions are met:

1. The irradiation of explosives or materials which could credibly detonate during irradiation or if contacted by water is prohibited.
2. Irradiation of pressurized sample containers which in the failed state could lead to sudden over pressure (pressure >75 psig) of the gamma irradiation chamber is prohibited.
3. Irradiation of sample material in sealed containers that could become pressurized due to radiolysis of the sample material, off gassing, phase changes or other means and potentially result in sudden over-pressure (>75 psig) of the gamma irradiation chamber is prohibited.
4. Irradiation of materials in a quantity that pose a credible corrosive threat to the HFIR fuel, pool liner or reactor components in pool storage is prohibited.
5. Sample material weight is restricted to $\leq$50 lbs to limit the potential for physical damage to the spent fuel due to inadvertent dropping of the chamber.
6. Sample material configurations employing electrical heaters, shall be equipped with thermocouples to monitor and ensure local pool coolant temperature on the chamber exterior remains below the saturation temperature (111 C).
7. Irradiation of fissionable sample material shall be approved by the HFIR Nuclear criticality safety officer and HFIR Material Balance Area coordinator.
8. All sample irradiations shall be reviewed and verified to meet applicable requirements contained in the latest revision of the nuclear criticality safety (NCS) approval documentation applicable to the storage of the HFIR spent fuel.
9. If a non-inert gas (i.e. other than helium, argon or nitrogen) is used as the gamma irradiation chamber sweep gas, then the sample material and quantity will be evaluated to ensure buildup of an ignitable mixture of radiolysis gases within the gamma irradiation chamber capable of causing damage to the spent fuel storage racks or HFIR fuel is not credible.

To ensure compliance with the above criteria, a USQD evaluation or USQD screening evaluation shall be performed for each new sample material irradiation configuration to verify the conduct of the sample material irradiation meets the above conditions. The USQD evaluation applicable to the sample material irradiation is to be listed in section 2 and 6. Upon completion of the USQD evaluation, table 1 may be amended without additional approval.

Section 2 : Requirements to be Met Prior to Experiment Installation
Section 2 defines requirements that are to be documented and verified prior to acceptance of the sample material for irradiation. Section 2 is to be completed (initialed/dated) **after** completion of the design safety review (section 4), and **prior** to sample material delivery to HFIR. Initials indicate that as-built sample configuration meets the requirement or has been appropriately dispositioned via a deviation or non-conformance report that has been approved by the RRD.

Req. No.	Requirements	Lead Exp.	Lead QA	RRD QA
	List Applicable Sample Material Configuration: Nano-modified Concrete	GGD 7/3/17	MAQ 7/13/17	SEP 7/14/17
1	Drawings/Sketches: The sample material configuration shall be defined on a drawing or sketch approved by the RRD Experiment Manager and RRD QAS. If sketches are used, the sketch should contain sufficient detail to define all applicable material, dimensional, cleaning, weld and other test/inspection requirements. For simple sample material configurations, a written description including sweep gas to be used approved by an EF&I staff member may be used in lieu of drawings or sketches. List applicable drawing/sketches below and attach copy of all sketches to EABD : **Or** List applicable written description and attach copy to EABD: List sweep gas to be used: Nitrogen	GGD 7/7/17	MAQ 7/13/17	SEP 7/14/17
2	USQD evaluation: A USQD or USQD screening evaluation applicable to the sample material configuration and sweep gas defined by the drawing/sketches or written description listed in item 1 shall be approved. The evaluation shall address all requirements listed in section 1. List the applicable evaluation # below and obtain signature of the EF&I staff member to verify evaluation is applicable to sample material and sweep gas configuration: List Applicable USQD/USQDSW evaluation: USQDSW - HFIR-2017-151 Rev. 0 RRD EF&I staff : [signature] Date: 7/16/17	GGD 7/16/17	MAQ 7/17/17	SEP 7/18/17
3	Fissionable Materials : The irradiation of fissionable material shall require the approval of the HFIR Criticality Safety Officer and the HFIR MBA representative. The non-fuel form fissionable inventory in the facility shall be limited to less than 700g ^{235}U fissionable equivalent mass. If applicable obtain signatures below. If no fissionable materials are present indicate N.A : HFIR MBA: ____________ Date: ________ HFIR NCS : ____________ Date: ________	N/A GGD 7/3/17	MAQ 7/13/17	SEP 7/14/17
4	Dimensions: Dimensions shall be per the requirements defined on the drawings/sketches or written description listed in item 1.	GGD 7/7/17	MAQ 7/13/17	SEP 7/14/17
5	Materials: Materials and materials inspection shall be per the drawings/sketches or written description listed in item 1.	GGD 7/7/17	MAQ 7/13/17	SEP 7/14/17
6	Cleaning: All components shall be cleaned in accordance with RRD-JS-31 or as specified on the drawings/sketches, or written description listed in item 1.	GGD 7/7/17	MAQ 7/13/17	SEP 7/18/17
7	Welding: Weld, weld inspection and weld report documentation shall be in accordance with drawing/sketch requirements or written description listed in item 1. Material certs are required for welding rod material.	N/A GGD 7/11/17	MAQ 7/13/17	N/A SEP 7/14/17

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8	Leak and Pressure Testing: Leak and Pressure testing shall be per the requirements of the drawings/sketches or written description listed above.	N/A GGD 7/3/17	[illegible] 7/13/17	N/A [illegible] 7/14/17
9	Nonconformances and Deviations: All nonconformances and deviations to the above requirements shall be listed in attachment B and dispositioned in accordance with applicable RRD procedures.	N/A GGD 7/16/17	[illegible] 7/17/17	N/A [illegible] 7/18/17
10	Auditable documentation sufficient to establish the as-built condition of the sample material holder configuration shall be on-file and retrievable.	GGD 7/3/17	[illegible] 7/13/17	N/A [illegible] 7/14/17
11	For experiments conducted with a non-inert sweep gas (air) the sponsor shall provide a hydrogen gas generation (radiolysis) constant for the sample material being irradiated, a maximum dose rate, and demonstrate that the sample mass is within the limits established for this combination as shown in table 5.1 of C-HFIR-2016-002. This requirement is not applicable to experiments that utilize inert sweep gases.	N/A GGD 7/3/17	[illegible] 7/13/17	N/A [illegible] 7/14/17

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Section 3: Administrative Constraints Imposed on Sample Irradiations

Sample material irradiations shall be conducted in accordance with PWP-1176 "Operation of the Gamma Irradiation Experiment Facility for Un-Instrumented Experiments" or PWP-1176A "Operation of the Gamma Irradiation Experiment Facility for Instrumented Experiments"

Bases : The above procedure ensures:

1) the chamber remains vented with sweep gas flow when performing sample irradiations
2) the chamber is provided with over-pressure protection (pressure relief valves)
3) the chamber will be removed if a leak to pool is detected
4) If electrical heaters are used, the chamber will be removed if the surrounding pool water temperature exceeds the saturation temperature (111C).
5) NCSA approval requirements are met.

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Section 4: Approval of Sample Material Configuration Design Requirements and Operating Bases
Note: This section is used to document approval of the sample material design requirements (Section 1), audit requirements (Section 2), and administrative constraints (Section 3). See notes for explanation of signatures.

C. S. Robison Lead Experimenter (print name)	Caleb Robison Lead Experimenter (signature)	12/21/16 Date
W.G.Askew Lead QA (print name)	W. George Askew Lead QA (signature)	12/21/2016 Date
C. S. Robison RRD E&FI Staff (print name)	Caleb Robison RRD E&FI Staff (signature)	12/21/16 Date
L. C. Smith RRD QA (print name)	[signature] RRD QA (signature)	1/6/17 Date
B. E. Fuller HFIR Operations (print name)	Brian E Fuller HFIR Operations (signature)	12/27/2016 Date
C. S. Robison USQD Preparer (print name)	Caleb Robison USQD Preparer (signature)	12/21/16 Date
C. D. Carathers USQD Reviewer (print name)	[signature] USQD Reviewer (signature)	01/19/17 Date
N.O. Huff RRD Criticality Safety Officer (print name)	Caleb Robison FOR PER TELECOM RRD Criticality Safety Officer (signature)	12/28/16 Date
FULLER, BRIAN E HFIR MBA Representative (print name)	Brian E Fuller HFIR MBA Representative (signature)	12/27/2016 Date
Not Required RRC Chair (print name)	RRC Chair (signature)	Date

Other:

Geoff Deichert for Florence Sanchez Lead Experimenter (print name)	[signature] Lead Experimenter (signature)	7/13/17 Date
N/A GGD 7/18/17 Lead Experimenter (print name)	N/A GGD 7/18/17 Lead Experimenter (signature)	Date
CHRIS BRYAN RRD Nuclear Safety and Experiment Manager	[signature] RRD NS and E Manager(signature)	1/19/2017 Date

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Section 5: Acceptance of QA Program
Note: This section is used to document acceptance of the QA program/plan governing sample material configuration fabrication, assembly, inspection, and testing.

Applicable QA Program/Plan: ORNL SBMS : Program Description : Quality Assurance Program

W.G.Askew Lead QA	W. George Askew L.E. Lead QA (signature)	7/18/2017 Date
L.C. Smith RRD QA	L C Smith L.E. RRD QA (signature)	7/18/2017 Date

Section 6: Acceptance for Use of As-Built Experiment Capsule
Note: This section is used to document acceptance of the as-built experiment for reactor installation and irradiation. This section is completed **after** completion of Section 2. See notes for explanation of signatures.

Applicable Sample Material Configuration : Nano-modified Concrete (See project description)

Approved Sweep Gas : Nitrogen

Applicable USQD/USQDSW Evaluation: USQDSW-HFIR-2017-151 Rev. 0

Geoff Deichert for Florence Sanchez Lead Experimenter (print name)	[signature] Lead Experimenter (signature)	7/18/2017 Date
W.GEORGE ASKEW Lead QA (print name)	W. George Askew Lead QA (signature)	7/18/2017 Date
Geoff Deichert RRD E&FI Staff (print name)	[signature] RRD E&FI Staff (signature)	7/18/2017 Date
Geoff Deichert for Nathan Huff per telecom RRD Criticality Safety Officer (print name)	[signature] RRD Criticality Safety Officer (signature)	7/18/2017 Date
N/A for MBA impact D.G. Blanchard for BEF Per telecom 7/18/17 HFIR MBA Representative (print name)	D.G. Blanchard for BEF Per telecom 7/18/17 HFIR MBA Representative (signature)	7/18/17 Date
L.C. Smith RRD QA (print name)	L C Smith RRD QA (signature)	7/18/17 Date
D.G. Blanchard HFIR Operations (name)	D.G. Blanchard HFIR Operations (signature)	7/18/17 Date

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Notes: Section 4 Signatures

Lead Experimenter: acknowledges acceptance the sample material configuration requirements listed in Section 1 and acknowledges acceptance of the acceptance requirements and conditions set forth in Sections 2 and 3.

Lead QA: acknowledges review and acceptance of the documentation requirements imposed on the as-built assembly in Section 2.

RRD E&FI Staff: acknowledges review and acceptance of the sample material configuration requirements in Sections 1,2 and 3.

RRD QA: acknowledges review and acceptance of the documentation requirements imposed on the as-built assembly in Section 2.

HFIR Operations Manager: acknowledges that required reactor operating procedures, reactor operator training or M.O.U needed to conduct sample irradiations are in place or listed as requirements in Section 2 or 3.

USQD Preparer: acknowledges that the USQD preparation was based on the configuration described in Section 1, and that design requirements and/or administrative constraints relied upon in the USQD are contained on the drawings or listed in Sections 2 and 3.

USQD Reviewer: acknowledges that the USQD preparation was based on the configuration described in Section 1, and that design requirements and/or administrative constraints relied upon in the USQD are contained on the drawings or listed in Sections 2 and 3.

RRD Criticality Safety Officer: acknowledges that nuclear criticality safety requirements that require documentation and verification prior to installation of the experiment are listed in Section 2, and that nuclear criticality safety requirements imposed on the operation of the experiment are listed in Section 3.

HFIR MBA Representative: acknowledges that material accountability requirements that require documentation and verification prior to installation of the experiment are listed in Section 2, and that nuclear material accountability requirements imposed on operation of the experiment are listed in Section 3.

RRC chair: acknowledges RRC review and acceptance and closure of all RRC recommendations.

Signature by the RRD NS&E Manager: acknowledges closure of the RRD safety review.

Notes Section 6 Signatures:

Lead Experimenter: certifies that a review of the as-built documentation has been conducted to verify compliance with each requirement initialed in section 2, that nonconformances and deviations have been appropriately documented and approved by the RRD and that the as-built assembly is ready for installation.

Lead QA: certifies satisfactory completion of independent review verifying compliance with each requirement initialed in section 2 and that documented evidence demonstrating compliance with each listed requirement is in place.

RRD E&FI Staff: acknowledges that sketches/drawings and USQD evaluation listed in section 2 have been approved and are applicable to the subject irradiation. Acknowledges that all deviations and nonconformances listed in ATT B have been approved by RRD.

RRD Criticality Safety Officer: signifies review and acceptance of the documentation or other evidence used to establish compliance of the as-built experiment assembly with the nuclear criticality safety requirements set forth in sections 2 and 3.

HFIR MBA Representative: signifies review and acceptance of the documentation or other evidence used to establish compliance of the as-built experiment assembly with the nuclear material accountability requirements set forth in sections 2 and 3.

RRD QA: certifies that the QA reviews specified in EG-1 have been conducted. Initials in section 2 document items checked.

HFIR Operations Manager: acknowledges that required reactor operating procedures, reactor operator training or M.O.U needed to interface experiment operations are in place and that appropriate experiment information has been entered into the HFIR Experiment Information Manual.

Revision Log for EABD-HFIR-2008-003 : HFIR Gamma Irradiation Facility Sample Irradiations

Revision Level	Description
0	Original Issue approved 12/16/08. Applicable USDQ : USQD-E-HFIR-2008-051
1	Revised 7/30/2009. Applicable USQD: USQD-E-HFIR-2008-051-Rev4 Revision 1 of the EABD extends the list of materials that may be irradiated in the facility to include: a) Gamma Dosimeters. The dosimeters will be irradiated concurrently with any planned experiment to verify the sample exposure and also used separately to verify facility dose rates in the empty chamber. b) Liquid covered resin samples in support of Savannah River waste disposition programs. The resins will be irradiated in both unsealed glass jars and in sealed stainless steel containers equipped with a rupture disc. Revision 1 also makes the following editorial changes: a) Adds reference to MM-HFIR-2009-123-R0 (addition of inline filter to gas supply station) b) Updates ATTA reference safety documentation to include Rev 3 and 4 of USQD-HFIR-2008-051
2	Revised 11/19/2009. Applicable USQD : USQDSW-HFIR-2009-262 Revision 2 of the EABD extends the list of materials that may be irradiated in the facility to include commonly used neutron flux monitors (Ag, Au, In, Mn, Cu, Co, Dy, Ni, Cd, Fe, Nb). Irradiation of materials in the gamma facility has shown that small levels of neutron induced activity are detectable in some of the sample irradiations. The monitors will be used to measure the neutron flux levels in the gamma chamber to allow calculations of induced activity. The flux monitors will be supported on aluminum or stainless steel stands and may also be attached to any approved sample material configuration to monitor the neutron exposure.
3	Revised 9/27/20010. Applicable USQD : USQDSW-HFIR-2010-321 Revision 3 of the EABD extends the list of materials that may be irradiated in the facility to include coaxial cable and teflon insulated coaxial cable connectors exposed to nitrogen, air and helium cover gas environments. The connectors and cable are being considered for use in the RRD safety channel ion chambers and the irradiation is intended to support the dedication process for the components. The cable and connectors will be enclosed inside two sealed stainless steel containers (one to provide an air cover gas, and the second to provide a nitrogen cover gas). A few cables and connectors will also be strapped to the outside of the containers and exposed to the normal helium gas flow used to purge the chamber. The sealed stainless steel containers used to provide the nitrogen and air sample cover gas environment are nearly identical to the canisters used in previous experiments (irradiation of liquid covered resin samples for SRL). The containers (160 ml volume) have been internally pressure tested at 500 psig and are equipped with a rupture disc having a burst pressure of 300 psig.
4	Revised 11/17/2011.-Applicable USQD:USQDSW-HFIR-2011-224 1. Editorial changes to reflect organizations changes and procedure additions. 2. Revised EABD to remove ATT C and ATTD (Table 1) from the body of the EABD to allow addition of sample material configurations without revising the EABD. 3. ATTC and ATTD will be revised with the approval of .additional material configurations. The new configurations will be approved with USQD/ USQDSW approval. 4. Irradiation approvals will still include approval of Sections 2, 4, 5 and 6 and ATTB(as applicable) of the EABD and will be filed with the as-built documentation for the irradiation.

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5	Revised 11/12/2012 –Applicable USQD HFIR-2008-051-Rev 5 Changes made in revision 5 of EABD-HFIR-2008-003 are to allow the use non-inert sweep gas (sweep gas other than helium, argon or nitrogen) to conduct sample irradiations provided a documented safety evaluation specific to the sample material/ sweep gas combination is completed to ensure that buildup of an ignitable mixture of radiolysis gases within the gamma irradiation chamber capable of causing damage to the spent fuel storage racks or HFIR fuel is not credible. Specific changes to the EABD are listed below 1. Section 1 : Added new requirement #9 If a non-inert gas (i.e. other than helium, argon or nitrogen) is used as the gamma irradiation chamber sweep gas, then the sample material and quantity will be evaluated to ensure buildup of an ignitable mixture of radiolysis gases within the gamma irradiation chamber capable of causing damage to the spent fuel storage racks or HFIR fuel is not credible. 2. Section 2 Revised Requirement 1 to require explicit listing of sweep gas. Revised Requirement 2 to reflect USDQ/USQDSW evaluation is to address sample material/sweep gas combination and to to address compliance with all design criteria set forth in section 1 3. Section 3 Revised bases statement # 1 to change inert sweep gas to sweep gas. 4. Section 6 Added "Approved Sweep Gas: ________________ " to clarify the approved sweep gas that may be used in conducting the irradiation and to ensure communication with reactor operations. 5. Revised the format of Table 1 "HFIR Gamma Irradiation Facility Approved Sample Configurations" to add a column entitled "Approved Sweep Gas" adjacent to the table column that lists the approved sample test configuration. 6. Updated Attachment A to include reference to revision 5 of USQD-E-HFIR-2008-051
6	Revised 4/27/2016 – Applicable USQ HFIR-USQDSW-2016-088, Rev 0 The change made in revision 6 of EABD-HFIR-2008-003 is to add an additional requirement for experiments that utilize a non-inert sweep gas. The requirement is found in section 2 and requires the sponsor to specify the conditions during irradiation and to prove that the sample mass is less than the limits derived in C-HFIR-2016-002.
7	Revised 12/21/2016 – Applicable USQ HFIR-USQDSW-2016-266 Requirement 5 of section 2 was changed so that the material is inspected and not verified. Verification denotes requirements per NQA-1 that are beyond what is necessary when the graded approach is applied. Inspection allows for a receipt inspection of materials which can vary in rigor depending on the particular circumstances of the experiment.

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ATT A - RELEVANT SAFETY REVIEW AND APPROVAL DOCUMENTATION

Applicable to : HFIR Gamma Irradiation Facility Sample Irradiations

Note: ATT A provides a list of references that constitute the bases for the safety review and approval. ATT A is completed by the RRD Research and Experimental Manager after completion of the design and safety review.

Attachment A may be amended without re-approval of the EABD to reflect safety documentation applicable to specific sample material irradiation configurations.

1. USQD-E-HFIR-2008-051-R0, R1, R2, R3 , R4 and R5 " DCM-HFIR-2005-083, PWP-1176 and EABD-HFIR-2008-003"

   These USQD's evaluate the use of the gamma irradiation facility hardware (DCM-HFIR-2005-083, MM-HFIR-2008-197, and MM-HFIR-2009-123-R0), the facility operating procedure used to conduct sample material irradiations (PWP-1176) and the requirements imposed on sample material configurations that may be irradiated in the facility (EABD-HFIR-2008-003).

   With respect to sample irradiations, six specific sample configurations (items 1 through 6 in table 1) are approved.

   Although the USQD is specific to the sample configurations listed in table 1, the USQD is intended to provide a basis for subsequent USQD screenings of future sample material configurations. Alternately the USQD may be revised to include future sample material configurations.

2. DCM-HFIR-2005-083 " Design Gamma Irradiation Facility"

   This DCM defines the design/design requirements established for the gamma irradiation facility hardware and approves the facility for conducting sample material irradiations. The original hardware approved in the DCM allows irradiation of un-instrumented samples only.

3. MM-HFIR-2008-197 " Add Instrument Capability to Gamma Irradiation Facility"

   This minor modification upgrades the gamma irradiation facility described in DCM-HFIR-2005-083 to accommodate instrumented experiment irradiations such as those planned for NASA

4. MM-HFIR-2009-123 " Add an in-line filter to the inert gas return on the gamma irradiation experiment facility"

   This minor modification adds an in-line filter on the gas supply station return line to reduce the potential for blockage of the gas station flow controller.

5. PWP-1176 and PWP-1176A- "Conduct of Gamma Irradiation Experiments Using the HFIR Gamma Irradiation Facility"

   This procedure provides the instructions for installation and removal of the chamber from the spent fuel and the operating instructions used to ensure a inert gas flow through the chamber during sample irradiations. The procedure specifies leak testing of the chamber and verification of relief valve set-point calibrations prior to capsule installation. If capsule pressure cannot be maintained above 10 psig a value above pool pressure (7 psig) or if capsule pressure increases above 25 psig or helium vent flow cannot be maintained above 100 sccm, the procedure requires removal of the chamber from the spent fuel. The procedure also verifies that for electrically heated experiments that thermocouples are attached to the exterior of the chamber to monitor pool water temperature and that the chamber is removed if pool water temperature exceeds the saturation temperature (111C).

6. Drawings M-11461-OH-248, -250, -251, -251A, -260 and 261A

   Detailed drawings that define the gamma irradiation facility hardware

7. Fabrication File : HFIR-ME-FF-475

   Contains Fabrication records associated with the gamma irradiation facility hardware

8. Listing of USQD/USQD Screening Evaluations Applicable to Specific Sample Material Configurations will be provided on ATT D and configuration shown in ATTC if necessary.

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ATT B - List of NCRs and Deviations

Applicable to: EABD-HFIR-2008-003 Rev. 5 : HFIR Gamma Irradiation Facility Sample Irradiations

Note: This section is completed only after completion of the fabrication of the sample holder configuration .

None. SPS 7/18/17

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Table 1
HFIR GAMMA IRRADIATION FACILITY APPROVED SAMPLE CONFIGURATIONS

Sample Test Configuration	Approved Sweep Gas	Applicable USQD	USQD/USQDSW Description	Sketch or Written Description	Comments
RRD Fission Chamber Epoxy	He, Ar,N	USQD-E-HFIR-2008-051-R0	Irradiation of insulated electrical leads inside a steel tube filled with epoxy to doses on the order of 10 mega-rad. The test is to qualify the epoxy as a sealer/spacer material for use in the HFIR fission chambers	See Attached Figure 1	The epoxy sample may or may not include embedded electrical leads or the steel tube enclosure. The specimens will be mounted to a center tube (aluminum or steel) using aluminum or steel set screws, wires or hose clamps.
RRD Pony Motor Fiberglass	He, Ar,N	USQD-E-HFIR-2008-051-R1	Irradiation testing of fiberglass/epoxy composite specimens to doses on the order of 10 megarad. The test is to qualify the fiberglass composite for use in HFIR pony motor electrical insulation applications.	See Attached Figure 2	The specimens will be mounted to a center tube (aluminum or steel) using aluminum or steel set screws, wires or hose clamps.
RRD RTD	He, Ar,N	USQD-E-HFIR-2008-051-R1	Irradiation of a resistance temperature detector (RTD) and associated phenolic terminal strip and wires to doses on the order of 10 mega-rad. The test is for dedication of the RTD circuit components intended for use in monitoring HFIR primary coolant pump bearing temperatures.	To be provided and approved per section 2 – requirement #1 prior to irradiation.	
NASA – Epoxy bonded coupons, O-Rings, Electrical wires. Xylan coated coupons	He, Ar,N	USQD-E-HFIR-2008-051-R1	Metal coupons (aluminum, steel, titanium alloys) glued together with epoxy, various elastomeric o-rings and insulated electrical wires and Xylan (a fluoropolymer coating similar to Teflon) coated coupons to doses on the order of 5 to 100 mega-rad. The irradiations will be used to qualify the potential use of these components in power converter applications associated with fission reactors being studied by NASA. The samples will be mounted to stainless steel or aluminum holders and incorporate additional thermocouples and electrical heaters to maintain component temperatures in the intended service range of 150 to 200C. Several irradiations of these components will be made.	To be provided and approved per section 2 – requirement #1 prior to irradiation.	

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Table 1
HFIR GAMMA IRRADIATION FACILITY APPROVED SAMPLE CONFIGURATIONS

<table>
<tr><th>Sample Test Configuration</th><th>Approved Sweep Gas</th><th>Applicable USQD</th><th>USQD/USQDSW Description</th><th>Sketch or Written Description</th><th>Comments</th></tr>
<tr>
<td>Irradiation of liquid covered resin samples to doses of 300 mega rad in support of Savannah River waste disposition programs.</td>
<td>He, Ar,N</td>
<td>USQD-E-HFIR-2008-051-R4</td>
<td>
The resin (resorcinol-formaldehyde: $C_7O_2H_2$) will be covered with one of 4 different liquids [water, 0.5 molar nitric acid, SRS Simulant or Hanford Simulant (see Table1)]. The resin will constitute nominally 60% of the volume and the liquid nominally 40%. The sample material will be irradiated in both unsealed glass jars and in sealed stainless steel containers equipped with a rupture disc.
Table 1: Composition of the Hanford Simulant and the Savannah River Simulant
<table>
<tr><th></th><th>SRS Tanks 1, 2, and 3</th><th>Hanford Tank AP-101</th></tr>
<tr><td>Chemical Compound</td><td>(g/L)</td><td>(g/L)</td></tr>
<tr><td>$NaNO_3$</td><td>336.7</td><td>154.2</td></tr>
<tr><td>$NaNO_2$</td><td>16.6</td><td>49.0</td></tr>
<tr><td>NaOH</td><td>58.4</td><td>81.6</td></tr>
<tr><td>$NaAlO_2$</td><td>10.7</td><td>21.3</td></tr>
<tr><td>Na_2CO_3</td><td>8.5</td><td>47.7</td></tr>
<tr><td>Na_2SO_4</td><td>4.3</td><td>5.7</td></tr>
<tr><td>NaCl</td><td>0.82</td><td>2.3</td></tr>
<tr><td>NaF</td><td>1.2</td><td>0.0</td></tr>
</table>
The unsealed glass jars (nominally 500 ml volume) will contain nominally 400 ml of the Hanford simulant resin solution and up to three sample jars may be irradiated at a single time in the un-instrumented gamma irradiation chamber configuration. The jars will be closed with plastic lids that have a small diameter hole (nominally 1/16") to allow venting of the radiolysis gases to the chamber interior. The inert sweep gas flow through the chamber interior will prevent accumulation of the radiolysis gases and pressure buildup within the chamber. Irradiations of similar material (see USQD-M-HFIR-1999-028) in glass jars in this configuration have been previously conducted.
The sealed stainless steel containers (≈160 ml volume) will contain nominally 130 ml of either one of the four liquid covered resin solutions and will be irradiated one at a time within the un-instrumented gamma irradiation chamber configuration. Although the steel container is designed to handle pressures (1800 psig) much higher than that expected during irradiation, the container has been provided with a rupture disc (tantalum or inconel in a 316 SST housing) having a burst pressure of 300 psig. If the rupture disc were to fail, the radiolysis gas would expand into the larger gamma irradiation chamber volume and the gas pressure would be reduced. Due to the large expansion volume provided by the gamma chamber (≈3000 ml with the sample installed) relative to the sample container volume (≈160 ml), the pressure inside the gamma irradiation chamber would remain well below the 100 psig value
</td>
<td>To be provided and approved per section 2 – requirement #1 prior to irradiation.</td>
<td>The sample jars or stainless containers are axially positioned within the gamma irradiation chamber using a steel or aluminum sample holder frame.</td>
</tr>
</table>

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Table 1

HFIR GAMMA IRRADIATION FACILITY APPROVED SAMPLE CONFIGURATIONS

Sample Test Configuration	Approved Sweep Gas	Applicable USQD	USQD/USQDSW Description	Sketch or Written Description	Comments
			conservatively assumed for the gamma irradiation chamber pressure rating (the gamma chamber has been hydro tested at 500 psig). The sealed steel containers are also fitted with an all metal SST valve at the upper end to allow gas extraction after irradiation and may also optionally include a direct reading bourdon pressure gage to determine sample container pressure after removal from the gamma chamber.		
Irradiation of Gamma Dosimeters	He, Ar,N	USQD-E-HFIR-2008-051-R4	The radiachromic dosimeters were procured from Far West Technology and consist of hexa paraosaniline nitrile dye (63.7% C, 12%% N, 9.5% H, and 14.8% O) dispersed in a nylon matrix. Changes in the optical density of the dosimeters after irradiation may be used to measure exposures in the range of 5.0E4 to 2.0E7 Rads. The dimensions of the dosimeters are nominally 1 cm square by 10um thick. Since the dosimeters are sensitive to humidity changes, each dosimeter is enclosed and irradiated inside a sealed aluminized pouch provided by the manufacturer. The dosimeters will be used to verify past measurements of the gamma dose rates in the facility and will be placed alongside other sample irradiations as requested by the experimenter to verify sample doses received.	See previous column.	May be incorporated as a component of any sample irradiation to verify accumulated exposure.
Neutron Flux Monitors	He, Ar,N	USQDSW-HFIR-2009-062	Revision 2 of the EABD extends the list of materials that may be irradiated in the facility to include commonly used neutron flux monitors (Ag, Au, In, Mn, Cu, Co, Dy, Ni, Cd, Fe, Nb). Irradiation of materials in the gamma facility has shown that small levels of neutron induced activity are detectable in some of the sample irradiations. The monitors will be used to measure the neutron flux levels in the gamma chamber to allow calculations of induced activity. The flux monitors will be supported on aluminum or stainless steel stands and may also be attached to any approved sample material configuration to monitor the neutron exposure.	SketchTo be provided Prior to irradiation	May be incorporated as a component of any sample irradiation to verify accumulated neutron exposure.

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Table 1

HFIR GAMMA IRRADIATION FACILITY APPROVED SAMPLE CONFIGURATIONS

Sample Test Configuration	Approved Sweep Gas	Applicable USQD	USQD/USQDSW Description	Sketch or Written Description	Comments
Electrical Connectors and cables in Support of RRD safety Chambers	He, Ar,N	USQDSW-HFIR-2010-321	Commercially available coaxial cable and teflon insulated coaxial cable connectors. The connectors and cable are being considered for use in the RRD safety channel ion chambers and the irradiation is intended to support the dedication process for the components. The cable and connectors will be enclosed inside two sealed stainless steel containers (one to provide an air cover gas, and the second to provide a nitrogen cover gas). A few cables and connectors will also be strapped to the outside of the containers and exposed to the normal helium gas flow used to purge the chamber. The sealed stainless steel containers used to provide the nitrogen and air sample cover gas environment are nearly identical to the canisters used in previous experiments (irradiation of liquid covered resin samples for SRL). The sample containers (160 ml volume) have been internally pressure tested at 500 psig and are equipped with a rupture disc having a burst pressure of 300 psig.	SketchTo be provided Prior to irradiation	The stainless containers are axially positioned within the gamma irradiation chamber using a steel or aluminum sample holder frame.
Rexolite@ dielectric material for use in RRD Safety Ion Chambers	He, Ar,N	USQDSW-HFIR-2011-223	Irradiation testing of Rexolite® thermosetting resin specimens to doses on the order of 1200 Megarad with a dry nitrogen cover gas. The test is to qualify the thermosetting resin for use in HFIR ionization chamber use.	Sketch to be provided	The specimens will be mounted to a center tube (aluminum or steel) using aluminum or steel set screws, wires, locking collars or hose clamps.
Ultra High Molecular Weight Polyethylene (UHMWPE)	He, Ar,N	USQDSW-HFIR-2012-125	Irradiation Testing of UHMWPE samples for the Food and Drug Administration (FDA) to evaluate the mechanical properties of UHMWPE for use in medical applications after irradiation up to 110KGy.	Photos to be provided to document the configuration.	The specimens will be positioned vertically along a stainless steel center tube to achieve a nominal dose rate of 27.5 KGy/ hr. The samples will be contained in a stainless steel can.
ASC Organics Testing for NASA	He, Ar,N	USQDSW-HFIR-2012-147	Materials irradiation for the NASA Fission Surface Power Program includes specifically ETFE shrink tube and insulation, Kapton, Xylan coated aluminum and inconel, Tra-Cast (Epoxy), and Loctite (anaerobic sealant applied as a thread lock). These materials are mounted to aluminum holders mounted and secured to the central heater cartridge (previously used central arrangement).	Sketch to be provided	The specimens will be mounted to a center tube as previously indicated and will be purposely assisted by internal heat source to a temperature of 150°C.

Table 1

HFIR GAMMA IRRADIATION FACILITY APPROVED SAMPLE CONFIGURATIONS

Sample Test Configuration	Approved Sweep Gas	Applicable USQD	USQD/USQDSW Description	Sketch or Written Description	Comments
Graphene and Graphite Nanotube on Silicon Oxide and Silicon	Compressed Air	HFIR-USQDSW-2012-247	Gamma Irradiations to determine the damage from gamma exposure to Graphene and Carbon Nanotubes on Silicon Oxide and Silicon Nitride Substrates for comparison to damage to samples exposed to Gamma and Neutron irradiation in the GPNAA facility. Work being done as part of Theses for Virginia Tech Students.	Written Description and Sketches	Irradiations restricted to: 1. Irradiations to be performed in an SFE more than 20 days (LT 3.0 X E07 R/hr) from shutdown.
Nitride Substrates					2. Sample/holder weight less than 30 grams 3. Free fillable volume in the chamber less than 1.0 liter.
Gamma Irradiation of Piezo valve for ITER design qualifications	Argon	HFIR-USQDSW-2013-045	Gamma Irradiation of Westlock Piezo Valve Assembly for Evaluation of the use of valve in the ITER operating environment. The valve materials include stainless stell, bras, epoxy and common plastics (nylon, polyester, polypropylene, polyvinyl acetate) and metals (copper and copper alloy, tin-silver-copper alloys) used in circuit board construction and in O-ring seals (Buna-N, Viton, EPDM).	Provided in Written Description and Photos	
Gamma Irradiation of common Electrical Insulation Materials Containing Nanocomposite Dielectrics	Argon, Compressed Air	HFIR-USQDSW-2013-080 HFIR-USQDSW-2016-088-R0	Conduct Gamma Irradiation of common Electrical Insulation Materials (such as cross linked polyethylene and polyvinyl alcohol) containing Nanocomposite Dielectrics, such as SiO_2, TiO_2, MgO, and GdO.	Provided in Written Description and Photos	May be performed in either instrumented or un-instrumented configurations.
Gamma Irradiation of Mineral (MgO) Insulated Cables for ITER	Nitrogen	HFIR-USQDSW-2013-122	Conduct Gamma Irradiation of Mineral (MgO) Insulated Cables for ITER	Provided in Written Description and Photos	May be performed in either instrumented or un-instrumented configurations.

Table 1

HFIR GAMMA IRRADIATION FACILITY APPROVED SAMPLE CONFIGURATIONS

Sample Test Configuration	Approved Sweep Gas	Applicable USQD	USQD/USQDSW Description	Sketch or Written Description	Comments
Gamma Irradiation of metal alloys in Uranyl Sulfate/Sulfuric acid and/or water	Nitrogen	HFIR-USQDSW-HFIR-2014-273	Corrosion testing of structural metal alloys in uranyl sulfate/sulfuric acid solution and/or water undergoing gamma irradiation to investigate the effects solution has on corrosion rates of the material.	Provided in Written Description and Photos	May be performed in either instrumented or un-instrumented configurations.
Gamma Irradiation of EPDM immersed in water	He, Ar, N	USQDSW-HFIR-2015-039	Gamma irradiation of Ethylene Propylene Diene Monomer (EPDM) rubber plugs immersed in water at doses up to nominally 5.0E9. An aluminum bar (3.25" O.D.) with an upper hole cavity (3" I.D. * 5.5 length) is used to secure and position up to 16 plugs about the spent fuel mid plane. Testing is for La Salle Nuclear Power Plant. The EPDM plugs were lost in the primary coolant system during a maintenance evolution. The hope is that radiation exposure will embrittle the plugs to the extent they break apart under expected hydraulic forces.	See description section of USQDSW-HFIR-2015-039	Performed in un-instrumented experiment configuration
Gamma Irradiation of Fiber Optics	He, Ar, N	USQDSW-HFIR-2015-190	Gamma irradiation of Fiber Optic cable to determine survivability in a high radiation/high temperature environment, up to 1.0 E8 rad. The fiber optic cable will be wrapped around a PVC pipe with fiber optic adapter connections at the top. The assembly will be covered in sheet metal duct pipe.	See description section of USQDSW-HFIR-2015-190	Performed in un-instrumented experiment configuration
Gamma Irradiation of Ion Exchange Resins	He, Ar, N	USQDSW-HFIR-2015-192	Gamma irradiation of Ion Exchange resins to identify leachable compounds. This work is sponsored by RevV program through the state of Tennessee in cooperation with ABT Molecular Imaging. Total Accumulated Doses of 25, 50, 75 kGy are desired.	See description section of USQDSW-HFIR-2015-192	Performed in un-instrumented experiment configuration
Gamma Irradiation of Carbon Based	He, Ar, N	USQDSW-HFIR-2015-259	Gamma irradiation of Carbon Nanotube Based Radiation Dosimeter material, e.g. fused silica and Poly(methyl methylacrylate) composites. The fused silica dosimeters consist of a substrate patterned with copper, titanium and nickel electrodes, coated in a layer of nanotubes and sealed with a layer of silicon dioxide. The composites are PMMA, boron, and carbon nanotubes.	See description section of USQDSW-HFIR-2015-259	Performed in un-instrumented experiment configuration

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Sample Test Configuration	Approved Sweep Gas	Applicable USQD	USQD/USQDSW Description	Sketch or Written Description	Comments
Gamma Irradiation of Polyurethane plugs immersed in water	He, Ar, N	USQDSW-HFIR-2015-039 Rev. 1	Gamma irradiation of Polyurethane plugs immersed in water. A stainless steel spacer (3.125" O.D. * 7.3125" length) with an upper holder (2-7/8" I.D. * 5.6875" length) is used to secure and position up to 9 plugs about the spent fuel mid plane. Testing is for La Salle Nuclear Power Plant. The Polyurethane plugs will be tested to determine what radiation exposure will embrittle the plugs to the extent they break apart under expected hydraulic forces.	See description section of USQDSW-HFIR-2015-039 Rev. 1	Performed in un-instrumented experiment configuration
Gamma irradiation of NIST and Landauer nanodot OSL dosimeters	He, Ar, N	USQDSW-HFIR-2016-163 Rev. 0	Evaluates the proposal to irradiate dosimeters from NIST as well as Landauer nanodot OSL dosimeters. The dosimeters are shown in photographs attached at the bottom of this USQDSW. The NIST dosimeters are alanine and are held in cylindrical clear plastic sample vials approximately 1.3 cm in diameter and 2.9 cm in length. The nanodot dosimeters are 1 cm in length and height and 1 mm in thickness and are made of Al_2O_3 in a plastic holder. The irradiations will be conducted with helium, nitrogen, or argon as the sweep gas.	See description section of USQDSW-HFIR-2016-163 Rev. 0	
Gamma irradiation of molded recombiner spheres	N	USQDSW-HFIR-2016-165 Rev. 0	Allow gamma irradiation of ¼" diameter recombiner spheres that are 85 wt% high consistency rubber silicone with the remaining 15 wt% a blend of 10% Pd on a carbon black catalyst.	See description section of USQDSW-HFIR-2016-165 Rev. 1	
Gamma irradiation of silicone and polyurethane discs	N	USQDS-HFIR-2016-256, Rev. 0	Allow gamma irradiation of 2.25" diameter discs of silicone and polyurethane. The discs are very thin and are testing for the degradation of medical implants in a gamma ray field.	See description section of USQDSW-HFIR-2016-256, Rev. 0	
Gamma irradiation of nano-modified concrete	N	USQDSW-HFIR-2017-151, Rev. 0	Allow gamma irradiation of nano-modified cement pastes and concrete-equivalent mortars (C.E.M.s). Material consists of Cement paste bars (1" x 1" x 2"), Cement paste tabs (1/4" x 1" x 3"), and C.E.M. Bars (1" x 1" x 2") as well as sacrificial samples of material. Use existing heater to control temperature at 200 °C and monitor with thermocouples.	See description section of USQDSW-HFIR-2017-151, Rev.0	May be performed in either instrumented or un-instrumented experiment configurations

Table 1

HFIR GAMMA IRRADIATION FACILITY APPROVED SAMPLE CONFIGURATIONS

Revision Log Summary for EABD

Revision	Date	Description	Reference Approval Documentation
0	12/16/08	Irradiation of insulated electrical leads inside a steel tube filled with epoxy to doses on the order of 10 mega-rad. The test is to qualify the epoxy as a sealer/spacer material for use in the HFIR fission chambers	USQD-E-HFIR-2008-051
1		Irradiation testing of fiberglass/epoxy composite specimens to doses on the order of 10 megarad. The test is to qualify the fiberglass composite for use in HFIR pony motor electrical insulation applications. Irradiation of a resistance temperature detector (RTD) and associated phenolic terminal strip and wires to doses on the order of 10 mega-rad. The test is for dedication of the RTD circuit components intended for use in monitoring HFIR primary coolant pump bearing temperatures. Metal coupons (aluminum, steel, titanium alloys) glued together with epoxy, various elastomeric o-rings and insulated electrical wires and Xylan (a fluoropolymer coating similar to Teflon) coated coupons to doses on the order of 5 to 100 mega-rad. The irradiations will be used to qualify the potential use of these components in power converter applications associated with fission reactors being studied by <u>NASA.</u> The samples will be mounted to stainless steel or aluminum holders and incorporate additional thermocouples and electrical heaters to maintain component temperatures in the intended service range of 150 to 200C. Several irradiations of these components will be made. Irradiation of liquid covered resin samples to doses of 300 mega rad in support of Savannah River waste disposition programs. Irradiation of FWT Gamma Dosimeters	USQD-E-HFIR-2008-051R1 USQD-E-HFIR-2008-051R4
2		Revision 2 of the EABD extends the list of materials that may be irradiated in the facility to include commonly used neutron flux monitors (Ag, Au, In, Mn, Cu, Co, Dy, Ni, Cd, Fe, Nb). Irradiation of materials in the gamma facility has shown that small levels of neutron induced activity are detectable in some of the sample irradiations. The monitors will be used to measure the neutron flux levels in the gamma chamber to allow calculations of induced activity. The flux monitors will be supported on aluminum or stainless steel stands and may also be attached to any approved sample material configuration to monitor the neutron exposure.	USQDSW-HFIR-2009-062
3		Commercially available coaxial cable and teflon insulated coaxial cable connectors. The connectors and cable are being considered for use in the RRD safety channel ion chambers and the irradiation is intended to support the dedication process for the components. The cable and connectors will be enclosed inside two sealed stainless steel containers (one to provide an air cover gas, and the second to provide a nitrogen cover gas). A few cables and connectors will also be strapped to the outside of the containers and exposed to the normal helium gas flow used to purge the chamber. The sealed stainless steel containers used to provide the nitrogen and air sample cover gas environment are nearly identical to the canisters used in previous experiments (irradiation of liquid covered resin samples for SRL). The sample containers (160 ml volume) have been internally pressure tested at 500 psig and are equipped with a rupture disc having a burst pressure of 300 psig.	USQDSW-HFIR-2010-321

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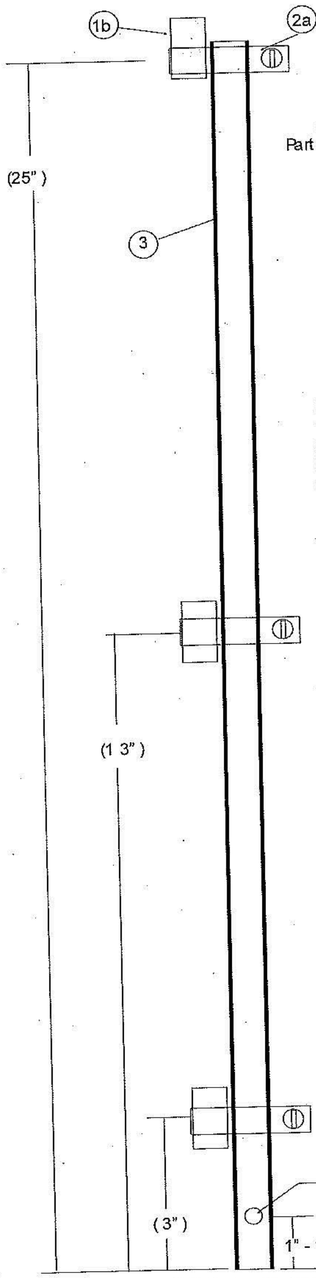
ATTACHMENT C (EABD-HFIR-2008-003) Table 1 HFIR GAMMA IRRADIATION FACILITY APPROVED SAMPLE CONFIGURATIONS	REV. 20	ATT C Page 9 of 9

Revision	Date	Description	Reference Approval Documentation
4	11/21/2011	Rexolite® and similar thermosetting resin material exposed to a dry nitrogen cover gas environment.	USQDSW-HFIR-2011-223
5	5/24/2012	Irradiation Testing of UHMWPE samples for the Food and Drug Administration (FDA) in Argon	USQDSW-HFIR-2012-125
6	7/20/2012	Materials irradiation for the NASA Fission Surface Power Program in Argon	USQDSW-HFIR-2012-147
7	11/12/2012	Gamma Irradiation of Single-Walled Carbon and Graphene Nanotubes with PTFE and polypropylene holders (SWNTs) in Laboratory Compressed Air	USQD-HFIR-2012-163
8	3/14/2013	Gamma Irradiation of the Westlock Piezo Valve Assembly in Argon sweep gas to evaluate for use in the ITER operating environment	USQDSW-HFIR-2013-045
9	5/6/2013	Conduct Gamma Irradiation of common Electrical Insulation Materials (such as cross linked polyethylene and polyvinyl alcohol) Containing Nanocomposite Dielectrics, such as SiO_2, TiO_2, MgO, and GdO	USQDSW-HFIR-2013-080
10	07/01/2013	Conduct Gamma Irradiation of Mineral (MgO) Insulated Cables for ITER	USQDSW-HFIR-2013-122
11	10/06/2014	Corrosion testing of structural metal alloys in uranyl sulfate/sulfuric acid solution and/or water undergoing gamma irradiation to investigate the effects solution has on corrosion rates of the material.	USQDSW-HFIR-2014-273
12	3/4/2015	Gamma irradiation of Ethylene Propylene Diene Monomer (EPDM) rubber plugs immersed in water.	USQDSW-HFIR-2015-039
13	8/31/2015	Gamma irradiation of Fiber Optics	USQDSW-HFIR-2015-190
14	9/9/2015	Gamma irradiation of Ion Exchange Resins	USQDSW-HFIR-2015-192
15	10/12/2015	Gamma irradiation of Carbon Nanotube Based Radiation Dosimeter materials	USQDSW-HFIR-2015-259
16	1/7/2016	Gamma irradiation of Polyurethane plugs immersed in water.	USQDSW-HFIR-2015-039 R1
17	4/28/2016	Add compressed air to the allowed sweep gasses for Gamma Irradiation of common Electrical Insulation Materials Containing Nanocomposite Dielectrics.	USQDSW-HFIR-2016-088-R0
18	9/7/2016	Added irradiation of NIST and Landauer nanodot OSL dosimeters as well as recombiner spheres	USQDSW-HFIR-2016-163 Rev. 0 USQDSW-HFIR-2016-165 Rev. 0
19	12/8/2016	Gamma irradiation of silicone and polyurethane discs and update USQDSW-HFIR-2016-165 to revision 1.	USQDSW-HFIR-2016-256, Rev. 0 USQDSW-HFIR-2016-165 Rev. 1
20	7/13/2017	Effect of Gamma Irradiation on the Microstructure and Mechanical Properties of Nano-modified Concrete.	USQDSW-HFIR-2017-151 Rev. 0

Table 1 Revision 20 Approval

RRD Experiment and Analysis [signature] Date: 7/16/17

ATTACHMENT D (EABD-HFIR-2008-003) **Sample Gamma Irradiation Sample Configuration Sketches**	REV. 5	ATT D Page 1 of 2



PARTS LIST

Part No.	Required	Description	Material
1b	3	Epoxy Specimen 9/16"O.D * 1.25" L	Modified Bisphenol A Epoxy (Huntsman - Araldite GY 502) Modified Amine Epoxy Hardner (Hunstman - Aradur 956-2-US)
2A	3	Hose Clamp ½" * 2"	Any Grade Steel Zinc Plating allowed.
3	1	Support Tube 3/4"O.D. * 25.25" L.	Any Grade Steel

Notes:
1. Clean Parts 2 and 3 with acetone and alchol per RRD-JS-31
2. Clean Specimens per Task Leader Instructions
3. Scribe Specimens "T" , "M", "B" for top, middle and bottom
4. Verify Centerline Location of Vent Holes is 1-1/8" ± 1/16" from bottom of Tube
   Verify Vent Hole Dia. Is 1/8" ± 1/16"

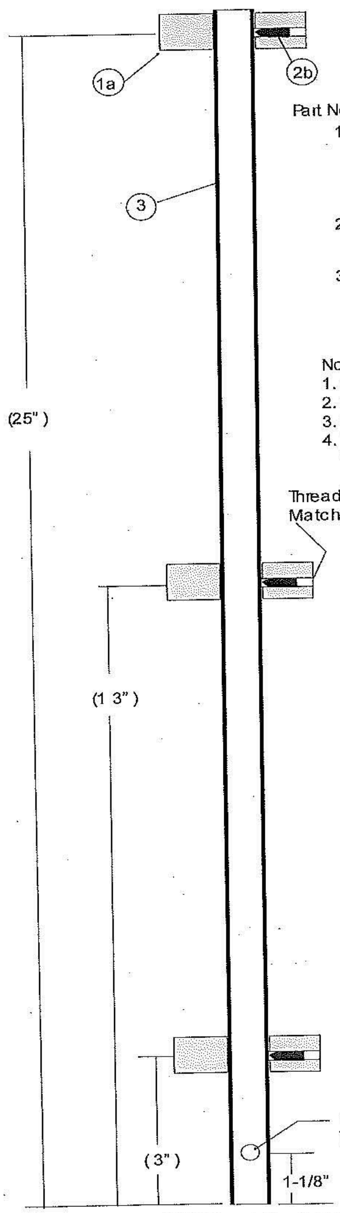
Approved BY: ______________________
Task Leader

Approved BY: ______________________
Nuclear Materials and Experiment Analysis

Approved BY: ______________________
RRD Quality Assurance

Figure 1: RRD Fission Chamber
Epoxy Specimen Gamma Irradiation Fixture

ATTACHMENT D (EABD-HFIR-2008-003) Sample Gamma Irradiation Sample Configuration Sketches	REV. 5	ATT D Page 2 of 2



PARTS LIST

Part No.	Required	Description	Material
1a	3	Fiberglass Reinforced Epoxy Specimen 2"square * ½"thick with 3/4" Center Hole	Rigid Epoxy Polymer Composite Reinforced with Fiberglass Fabric RESITEN GRADE G10FR4 (TEN INDUSTRIES)
2b	3	Set Screw 1/4" dia. * ½"L	Any Grade Steel
3	1	Support Tube 3/4"O.D. * 25.25" L	Any Grade Steel

Notes:
1. Clean Parts 2 and 3 with acetone and alchol per RRD-JS-31
2. Clean Specimens per Task Leader Instructions
3. Scribe Specimens "Top" , "Middle", "Bottom"
4. Verify centerline of Vent Holes is 1- 1/8" ± 1/6" from bottom of tube
   Verify Vent Hole dia. Is 1/8" ± 1/16"

Approved BY: ______________________
Task Leader

Approved BY: ______________________
Nuclear Materials and Experiment Analysis

Approved BY: ______________________
RRD Quality Assurance

Figure 2: RRD Primary Coolant Pump Motor Rocker Board Epoxy/ Fiberglass Specimen Gamma Irradiation Fixture

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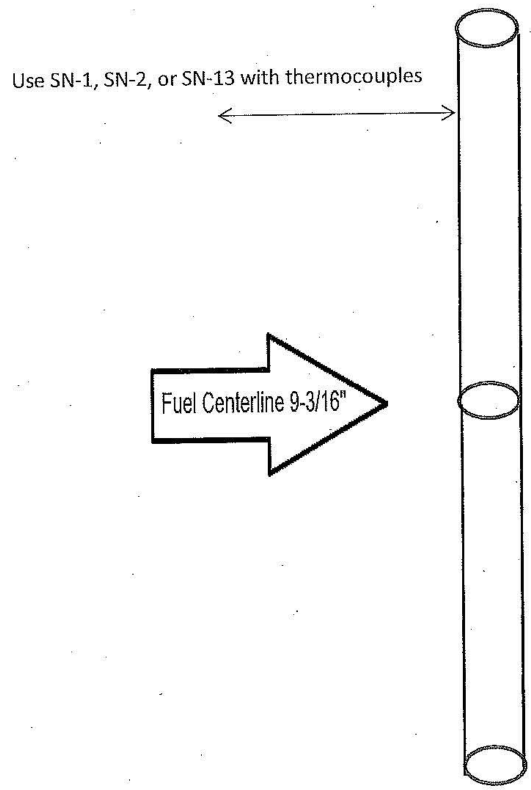
Experiment configuration A – (Dosimetry and Temperature evaluation only)



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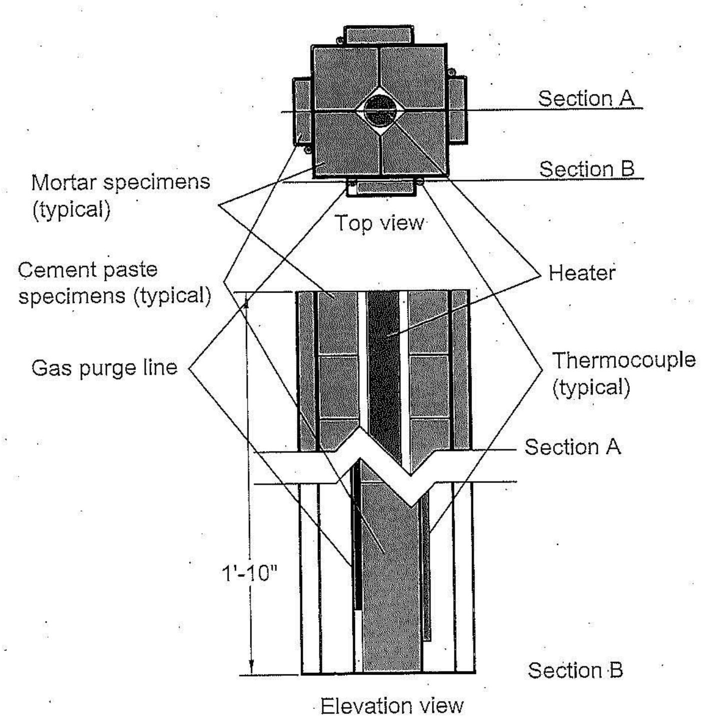
Experiment Configuration B (Sample irradiations)



Canister schematic

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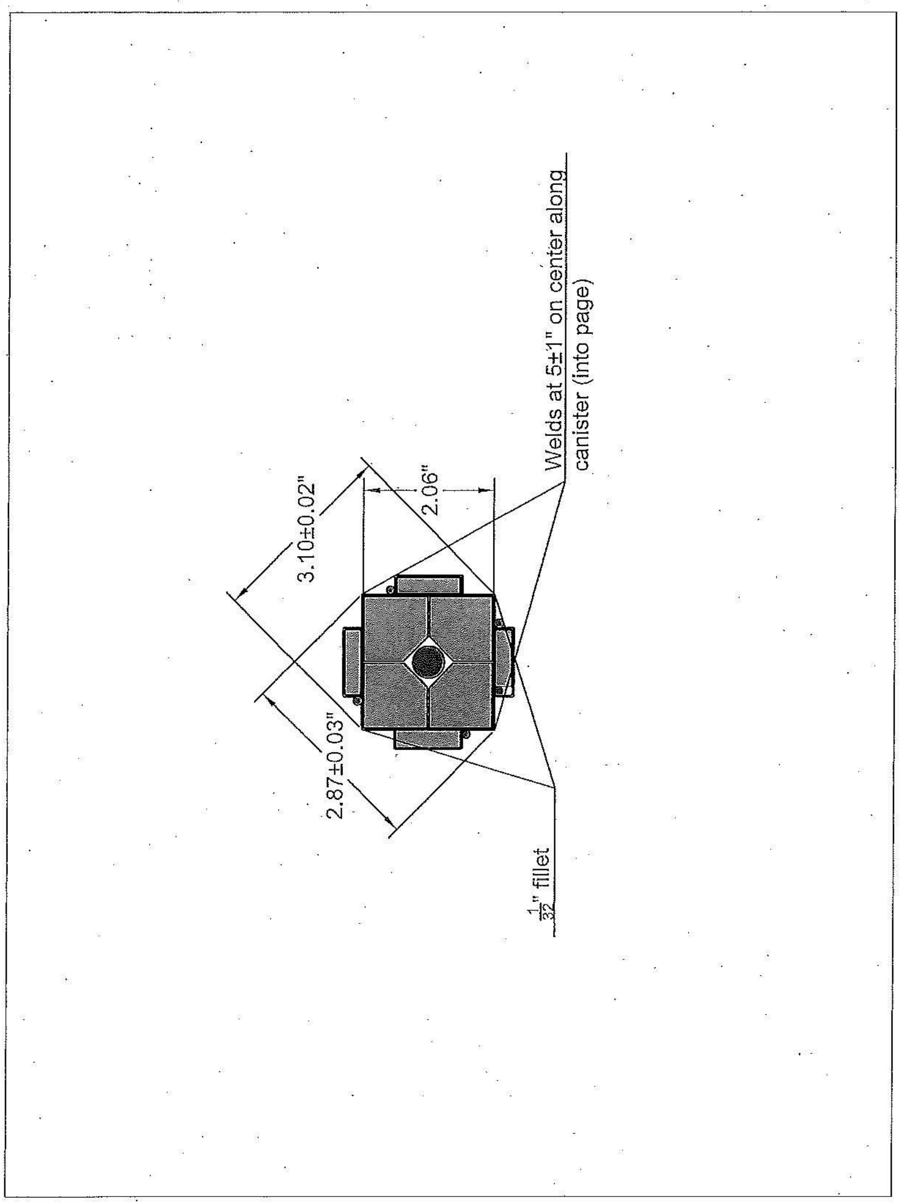
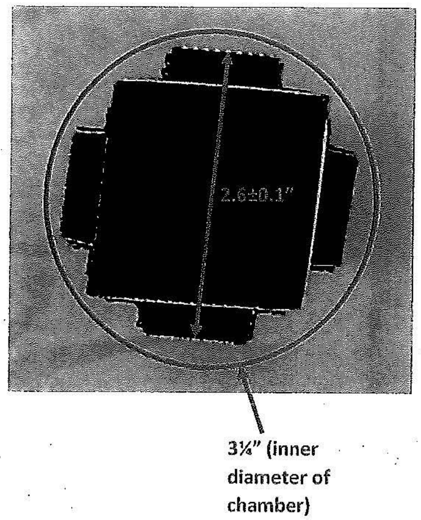
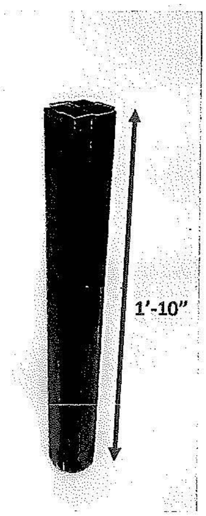


Canister cross-sectional dimensions

A-27





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APPENDIX B. HFIR USQD DOCUMENT

APPENDIX B. HFIR USQD DOCUMENT

USQDSW-HFIR-2017-151 Rev 0

UNREVIEWED SAFETY QUESTION DETERMINATION (USQD) SCREENING WORKSHEET

Part I - Introduction

1. **Facility/Activity:** HFIR

2. **Subject of Evaluation:**

   Irradiation of nano-modified cement pastes and concrete-equivalent mortars (CEMs) in the gamma irradiation facility.

3. **Description of the change:**

   Allow gamma irradiation of nano-modified cement paste and concrete-equivalent mortar (CEM) samples (Figure 1) in open stainless steel holders using inert sweep gas (He, Ar, or N2) in both the instrumented and un-instrumented configurations of the HFIR Gamma Irradiation Facility (GIF). Note that the current intent is to test in the instrumented configuration with heaters and thermocouples to monitor temperatures, but the un-instrumented configuration without heaters is also included in this USQD screening worksheet. The samples are composed of Al_2O_3, BaO, CaO, Fe_2O_3, K_2O, MgO, Na_2O, SiO_2, SO_3, and TiO_2. The term "nano-modified" refers to some of the cement components being supplied in powders with nanometer range particle sizes as opposed to micrometer or greater particle sizes that might be typical. These particle sizes affect the resulting microstructure of the cement, but after cement formation there are no longer nanometer sized free particles. There is no limit on sample size/geometry other than fitting within the gamma can and the overall sample weight restriction of 50 lbs per irradiation.

   This change includes the addition of a corresponding entry in Table 1 Rev 20: "HFIR Gamma Irradiation Facility Approved Sample Configurations" found in EABD-HFIR-2008-003 Rev 7, Attachment C.

4. **Primary safety basis documents:**

   1) ORNL/HFIR/SAR/2344, HFIR Safety Analysis Report, Revision 16
   2) ORNL/RRD/INT-138, HFIR Cold Neutron Source Documented Safety Analysis, Revision 10
   3) ORNL/TM-12841, HFIR Technical Safety Requirements, Revision 21
   4) Justification for Continued Operation for Potential Inadequacy of the Documented Safety Analysis Related to Error in Buoyancy Model in Effective Stack Height Calculation

   Other pertinent documents:

   5) USQD-E-HFIR-2008-051 Rev5, "Gamma Irradiation Facility Sample Irradiations" (11/15/2012)
   6) EABD-HFIR-2008-003 Rev 7, "HFIR Gamma Irradiation Facility Sample Irradiations" (02/20/2017)
   7) Table 1 Rev 20, "HFIR Gamma Irradiation Facility Approved Sample Configurations" (07/14/2017, found in EABD-HFIR-2008-003 Rev 7, Attachment C)

5. **Safety Analysis:**

   EABD-HFIR-2008-003 "HFIR Gamma Irradiation Facility Sample Irradiations" governs irradiation of material in the HFIR gamma irradiation facility. The EABD is based on the original USQD performed for the gamma irradiation facility (USQD-E-HFIR-2008-051). The requirements that must be met are delineated in Section 1 of the EABD (found on page 1) and are repeated here:

   1. **The irradiation of explosives or materials which could credibly detonate during irradiation or if contacted by water is prohibited.**

      -The materials used in the cement/concrete samples are all oxides which are not explosive either by themselves or in the presence of water. Note that this includes the alkali metals which are all present as oxides and therefore are not explosive by themselves or in the presence of water.

   2. **Irradiation of pressurized sample containers which in the failed state could lead to sudden over pressure (pressure >75 psig) of the gamma irradiation chamber is prohibited.**

      -The cement/concrete samples are not pressurized.

   3. **Irradiation of sample material in sealed containers that could become pressurized due to radiolysis of the sample material, off gassing, phase changes or other means and potentially result in sudden over-pressure (>75 psig) of the gamma irradiation chamber is prohibited.**

      - The cement/concrete samples are not enclosed in a sealed container.

   4. **Irradiation of materials in a quantity that pose a credible corrosive threat to the HFIR fuel, pool liner or reactor components in pool storage is prohibited.**

      -The cement/concrete samples do contain components which in the presence of water could conceivably generate alkaline (BaO, CaO, K_2O, MgO, Na_2O) and/or acidic (SO_3) solutions. However, exposure of the samples to water would require a leak in the gamma irradiation can, and if a leak is detected the can is immediately removed from the fuel element and the pool, thus removing any corrosive threat. If such a leak went undetected, any release of alkaline or acidic solution from the samples would occur slowly over time and would be diluted in the pool water such that it still would not pose a credible corrosive threat to the HFIR fuel, pool liner, or reactor components in pool storage. Note that this type of potential for corrosive threat falls well within the scope of USQD-E-HFIR-2008-051 Rev 5 (see page 15-16).

5. **Sample material weight is restricted to ≤50 lbs to limit the potential for physical damage to the spent fuel due to inadvertent dropping of the chamber.**

   -The cement/concrete samples will be limited to ≤ 50 lbs per irradiation.

6. **Sample material configurations employing electrical heaters, shall be equipped with thermocouples to monitor and ensure local pool coolant temperature on the chamber exterior remains below the saturation temperature (111 C).**

   -The cement/concrete samples will only employ electrical heaters in the instrumented configuration of the GIF where thermocouples will also be included for temperature monitoring.

7. **Irradiation of fissionable sample material shall be approved by the HFIR Nuclear criticality safety officer and HFIR Material Balance Area coordinator.**

   -The cement/concrete samples do not contain fissionable material.

8. **All sample irradiations shall be reviewed and verified to meet applicable requirements contained in the latest revision of the nuclear criticality safety (NCS) approval documentation applicable to the storage of the HFIR spent fuel.**

   -The cement/concrete sample experiments meet the requirements of Nuclear Criticality Safety Approval (NCSA) 119 revision 5. Specifically: the experiments will be performed in the target region of a stored fuel element in the planar fuel storage arrays (FSAs) or the loading station, the experiments will remain vented via inert sweep gas, and the experiments will be removed from the stored fuel element prior to moving that element.

9. **If a non-inert gas (i.e. other than helium, argon or nitrogen) is used as the gamma irradiation chamber sweep gas, then the sample material and quantity will be evaluated to ensure buildup of an ignitable mixture of radiolysis gases within the gamma irradiation chamber capable of causing damage to the spent fuel storage racks or HFIR fuel is not credible.**

   -Inert gas (typically N2) will be utilized as the sweep gas. Concrete is known to break down slowly over time in irradiation fields, and so any release of radiolysis gases will occur slowly over time and any potential for accumulation of ignitable mixtures of gas will be mitigated by the use of inert sweep gas. Note that this type of potential for ignitable gas accumulation falls well within the scope of USQD-E-HFIR-2008-051 Rev 5 (see page 14-15).

Although the safety basis documents have changed since USQD-E-HFIR-2008-051 Rev5 was written, the responses to the USQD questions have not changed and is therefore bounding.

6.	**Does the proposed change require revision of TSR or affect a DOE Condition of Approval? If Yes, then DOE approval is required.**	☐ Yes	☑ No
7.	**Does the change make any non-editorial changes to the DSA or an editorial change to TSR Bases? If Yes, a USQD is required. If an editorial change to the DSA is being made, identify the change.**	☐ Yes	☑ No

Part II - USQD Screening

1. **USQD Primary Screening Criteria: Is the proposed change:**

a.	**A temporary or permanent modification to a structure, system or component explicitly or implicitly described in the documented safety analysis?**	☐ Yes	☑ No
b.	**A completely new structure, system, or component that is of the type that would be described in the documented safety analysis?**	☐ Yes	☑ No
c.	**A temporary or permanent change in a procedure described in the documented safety analysis?**	☐ Yes	☑ No
d.	**A completely new procedure that is of the type that would be described in the documented safety analysis?**	☐ Yes	☑ No

e. A test, experiment, new operation or new activity not described in the documented safety analysis? ☑ Yes ☐ No

If the responses to questions 1.a through 1.e in Part II are all "No," then no further screening is necessary, a USQD is not required, and proceed to Part III. If any responses 1.a through 1.e are "Yes," then a USQD is required unless excluded by the additional screening in Item 2.

2. USQD Secondary Screening Criteria: Is the proposed change:

a. Fully within the scope of a DOE-approved categorical exclusion? ☐ Yes ☑ No
If "Yes," identify the exclusion: ______

b. Of an editorial nature and not a technical change? ☐ Yes ☑ No

c. Fully within the scope of a previous USQD? ☑ Yes ☐ No
If "yes," identify the USQD: USQD-E-HFIR-2008-051 Rev5

d. A common commercial practice? (e.g., office type change) ☐ Yes ☑ No

e. Limited to directly implementing a DOE-approved change? ☐ Yes ☑ No

If any answer to questions 2.a through 2.e is "Yes," then no USQD is required. Otherwise, a USQD is required if not screened out by question 1.

Part III – Conclusion and Approval

A USQD is not required ☑ A USQD is required ☐

Crowell, MW (Online)	*07/13/2017*
Prepared By	Date
Carathers, CD (Online)	*07/14/2017*
Nuclear Facility Manager (or designee)	Date

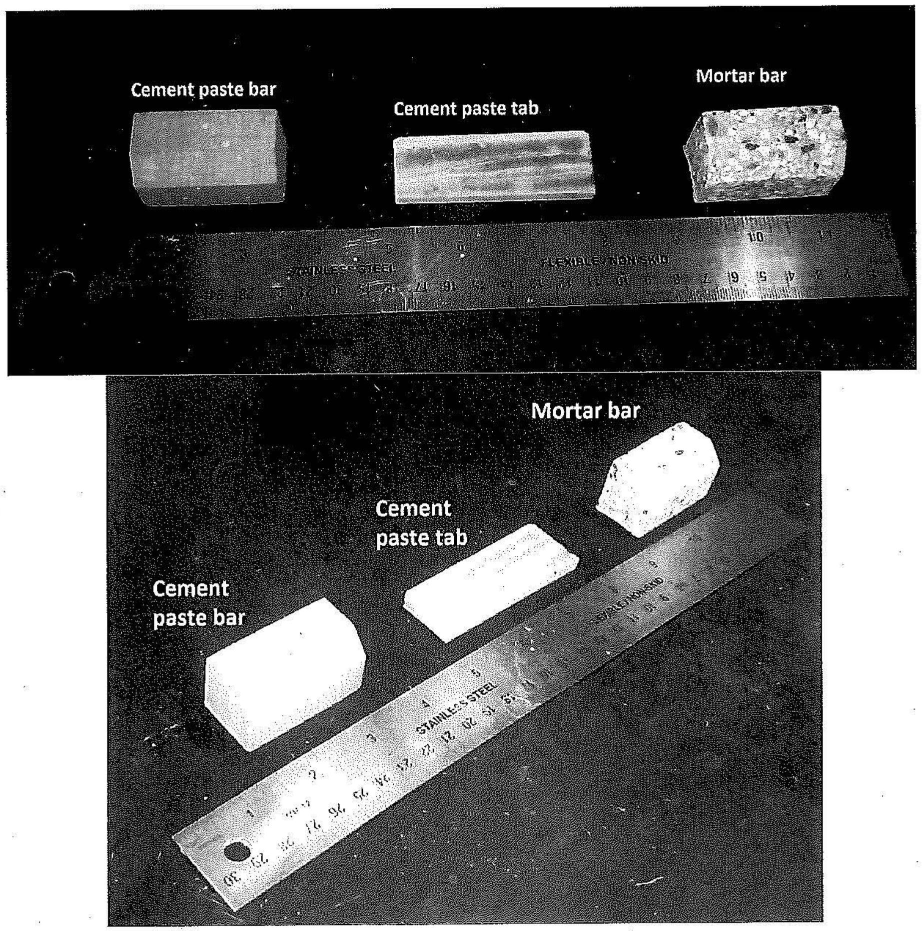

Figure 1. Cement paste and concrete equivalent mortar samples.

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