



Task 2 INL Updated Test Matrix and Updated Milestones

June 2020

Changing the World's Energy Future

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Milestone Overview

Agreed milestones for FY 2020 with updated expected completion dates:

- **Milestone 2.6:** Complete round robin hydrogen gas analysis capability comparison.
- **Milestone 2.7:** Evaluation of techniques for the measurement of molecule hydrogen gas in helium matrices.
- **Milestone 2.8:** Complete additional radiolytic gas generation analysis including helium-backfilled samples. To achieve the due date of 09/30/2020, the following will be aimed for:
 - Pristine and blank irradiations in helium atmospheres
 - Pristine irradiations to a total dose 100 kGy for argon and nitrogen atmospheres
 - Corroded sample irradiations in helium (all doses), and remaining nitrogen and argon atmospheres to doses of 100, 250, 750, 1000, 2000, 5000 kGy.

Current Status and Proposed Test Matrix

Corrosion testing has been delayed due to iron contamination of corroded samples – investigations as to the source of this is underway and plans for mitigation for the next round in place.

Completed pristine Al1100 coupon irradiations under argon and nitrogen atmospheres of various relative humidities were undertaken and reported in December 2019. This work contributed to **Milestone 2.6** and **Milestone 2.8**. The agreed total absorbed doses for this work were 250, 500, 750, and 1000 kGy. However, the new proposed work to be undertaken at SRNL includes total doses of 50 and 100 kGy. Additional total gamma doses will also include pre-corroded samples, so thorough comparisons can be made. Samples in helium head spaces will be prioritized.

Update: With a focus on physiosorbed water, if time permits the effect of temperature on sample weight and hydrogen generation. Samples will be heated in a vacuum oven at 50, 150 and 250 °C overnight (approx. 16 hours), weight measurements will be taken pre and post heating. Samples will then be irradiated in argon environments with 0% relative humidity and the radiolysis induced hydrogen concentration will be measured using gas chromatography. Changes in oxide morphology and composition will be probed using SEM and XRD. The additional total doses to be undertaken initially are 2 MGy and 5 MGy to see if the systems can be forced to steady state. If samples and irradiation facilities are available dose point of to 100 kGy will be undertaken to overlap with data points at SRNL.

Table 1 shows the proposed irradiation test matrix with the highlighted rows to be undertaken after discussion with SRNL.

Table 1. Proposed test matrix for remaining FY20 work.

Fill Gas	% Relative Humidity	Desired Absorbed	Sample Description
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		Dose (kGy)	
Helium	0 approx.	250	No coupon, corroded and pristine
Helium	0 approx.	500	No coupon, corroded and pristine
Helium	0 approx.	750	No coupon, corroded and pristine
Helium	0 approx.	1000	No coupon, corroded and pristine
Helium	0 approx.	2000	No coupon, corroded and pristine
Helium	0 approx.	5000	No coupon, corroded and pristine

Helium	50 approx.	250	No coupon, corroded and pristine
Helium	50 approx.	500	No coupon, corroded and pristine
Helium	50 approx.	750	No coupon, corroded and pristine
Helium	50 approx.	1000	No coupon, corroded and pristine
Helium	50 approx.	2000	No coupon, corroded and pristine
Helium	50 approx.	5000	No coupon, corroded and pristine

Helium	100 approx.	250	No coupon, corroded and pristine
Helium	100 approx.	500	No coupon, corroded and pristine
Helium	100 approx.	750	No coupon, corroded and pristine
Helium	100 approx.	1000	No coupon, corroded and pristine
Helium	100 approx.	1000	No coupon, corroded and pristine
Helium	100 approx.	1000	No coupon, corroded and pristine

The lower helium, **argon and nitrogen** sample doses (e.g., 100 kGy and 50 kGy) time permitting.

Argon	0 approx.	250	Corroded
Argon	0 approx.	750	Corroded
Argon	0 approx.	2000	No Coupon, corroded and pristine
Argon	0 approx.	5000	No Coupon, corroded and pristine

Argon	50 approx.	250	Corroded
Argon	50 approx.	750	Corroded
Argon	50 approx.	2000	No Coupon, corroded and pristine
Argon	50 approx.	5000	No Coupon, corroded and pristine

Argon	100 approx.	250	Corroded
Argon	100 approx.	750	Corroded
Argon	100 approx.	2000	No Coupon, corroded and pristine
Argon	100 approx.	5000	No Coupon, corroded and pristine

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Nitrogen	0 approx.	250	Corroded
Nitrogen	0 approx.	500	Pristine
Nitrogen	0 approx.	750	Corroded
Nitrogen	0 approx.	2000	No Coupon, corroded and pristine
Nitrogen	0 approx.	5000	No Coupon, corroded and pristine
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Nitrogen	50 approx.	250	Corroded
Nitrogen	50 approx.	500	Pristine
Nitrogen	50 approx.	750	Corroded
Nitrogen	50 approx.	2000	No Coupon, corroded and pristine
Nitrogen	50 approx.	5000	No Coupon, corroded and pristine
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Nitrogen	100 approx.	250	Corroded
Nitrogen	100 approx.	500	Pristine
Nitrogen	100 approx.	750	Corroded
Nitrogen	100 approx.	2000	No Coupon, corroded and pristine
Nitrogen	100 approx.	5000	No Coupon, corroded and pristine
