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Report On Hydrogen Content Measurements of Yttrium Hydrides

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SUMMARY

This report describes the hydrogen content measurements of yttrium samples as irradiated at Advanced Test Reactor as part of the Microreactor Program. Irradiated samples were prepared at the facilities of Analytical Research Laboratory (ARL) of Materials and Fuels Complex (MFC). Hydrogen content measurements were performed on reduced size specimens using an inert gas fusion analyzer. From a single specimen, replicate samples were prepared and tested. Uncertainty analyses were conducted using two approaches to determine the hydrogen content variations in the samples. The hydrogen content of irradiated specimens was both lower and higher than the expected values, indicating stoichiometry variations. Hydrogen was detected in all samples, including the cracked capsule's specimens. Results also suggested the potential presence of hydrogen redistribution between samples inside the capsules.

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ACRONYMS

ARL	Analytical Research Laboratory
CLGB	Casting Laboratory Glove Box
DOE	Department of Energy
GUM	Guide To The Expression Of Uncertainty In Measurement
FY	Fiscal Year
H	hydrogen
IGF	Inert Gas Fusion
MFC	Materials and Fuels Complex
PIE	post-irradiation examination
YH	yttrium hydride

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REPORT ON HYDROGEN CONTENT MEASUREMENTS OF YTTRIUM HYDRIDES

1. INTRODUCTION

The Department of Energy's (DOE) Microreactor Program is investigating the use of solid neutron moderators that enable the deployment of transportable compact nuclear reactors (microreactors) [1, 2]. Owing to high moderating power, light-elements, such as hydrogen (H), beryllium, and carbon, are suitable for use as neutron moderators. As the lightest element, H has the highest moderating power. Thus, H-bearing materials such as metal hydrides are candidate materials for a neutron moderator. Amongst all metal hydrides, yttrium hydride has superior high-temperature stability at temperatures greater than ~800°C with improved hydrogen retention compared to other candidate moderators [3-5]. While use of yttrium hydride is beneficial, chronic hydrogen loss at high temperature (> 600°C) may degrade the long-term moderating power and consequently the neutronic performance of the reactor. Furthermore, the effect of neutron irradiation on hydrogen retention is unknown. For the development of a safe operation window, hydrogen retention needs to be determined for such hydride-moderator reactors [6]. Therefore, H content measurements on irradiated yttrium hydride are essential to understanding the evolution of hydrogen in yttrium hydride, and predicting H inventory of a moderator within a nuclear environment.

Inert gas fusion (IGF) is one of several techniques used to measure the hydrogen content in materials. Idaho National Laboratory (INL) has an IGF hydrogen analyzer configured for irradiated samples. A typical IGF analyzer is set to measure H content lower than 500 wt. ppm for a standard sample mass of 100 mg. Above that hydrogen concentration, the IGF's detection instruments can saturate and become insensitive to the hydrogen content. Noting that, the lack of traceable certified calibration specimens to bound hydrogen content range challenges the use of typical samples. Because a solid substoichiometric yttrium dihydride ($YH_{1.89}$) contains ~21000 ppm of H, IGF measurements require smaller samples that will evolve hydrogen below the detectable level via either diluting samples with an inert compound or reducing the specimen mass. Challenges are associated with both methods. For example, an inert material used as a diluent can absorb moisture, which influences the hydrogen measurements. Reducing the specimen mass can drastically affect the uncertainties of the mass measurements which can deleteriously propagate. Both methods can introduce biases due to sampling errors as caused by potential contamination and non-homogeneity.

During the fiscal year 2022 (FY-22), fresh yttrium hydride samples were pulverized and diluted with yttria powder. The hydrogen content measurements were in good agreement with the expected value. To validate the accuracy of the dilution method additional experiments were carried out using a titanium hydride reference material in FY-23. The accuracy of the dilution method diverged for one reference sample. Graphite was used as a second diluent and results looked promising. However, with both diluents, inconsistencies were observed in reverence unirradiated yttrium hydride samples over time. An alternative method of reducing the sample mass was explored and postulated to provide more consistent results with reasonable uncertainties.

The irradiated samples were tested by reducing sample mass to the range of 2.8–3.1 mg which was 30 times lower than ones used with the dilution approach. This significant change increased the uncertainty of mass measurements due to the sensitivity of the balance. To minimize the total uncertainty in hydrogen content measurements, replicate measurements were performed and summed.

This report describes the hydrogen content measurements of yttrium samples irradiated as part of the Microreactor Program. Irradiated samples were prepared at the facilities of Analytical Research Laboratory (ARL) at INL's Materials and Fuels Complex (MFC). Hydrogen content measurements were performed on reduced size specimens using an IGF analyzer. For a single sample, multiple sub-specimens were prepared and tested to improve statistics of results. Uncertainty analyses were conducted to determine the hydrogen content variations in the samples.

2. MATERIALS AND METHODS

2.1 Irradiated Samples

Six capsules, each containing 17 yttrium hydride samples, were irradiated in INL's Advanced Test Reactor. Table 1 shows the specimen layout within each capsule, and samples that were subject to IGF for hydrogen content measurements are highlighted. For IGF analysis, six samples at the same axial location were selected from each capsule. Two additional samples from the capsules 700-2B (cracked) and 800-6 were tested to study the impact of pressure boundary loss and elevated temperature on the hydrogen retention. In total, eight irradiated specimens, shown in Figure 1, were prepared at ARL. Samples were stored in an inert atmosphere as long as possible prior to examinations[a].

Table 1. Locations of the hydrogen content measurement specimens (shaded with blue) in the capsules.

Specimen name	Capsule identifications					
	600-1	700-2B	800-3A	600-4	700-5	800-6
RUS1	Intact	Intact	Intact	Intact	Intact	Intact
RUS2	Intact	Intact	Intact	Intact	Intact	Intact
RUS3	Intact	Intact	Intact	Intact	Intact	Intact
GDOES1	Intact	Intact	Intact	Powdered	Intact	Intact
GDOES2	Intact	Intact	Intact	Powdered	Intact	Intact
GDOES3	Intact	Intact	Intact	Powdered	Bonded to TZM	Intact
GDOES4	Intact	Intact	Intact	Powdered	Intact	Intact
GDOES5	Intact	Intact	Broken	Powdered	Intact	Intact
TEM1	Intact	Intact	Intact	Intact	Intact	Intact
LFA1	Intact	Intact	Broken	Intact	Intact	Intact
LFA2	Intact	Intact	Intact	Intact	Intact	Intact
DSC1	Intact	Intact	Bonded	Intact	Intact	Intact
DSC2	Intact	Intact		Intact	Intact	Intact
DSC3	Intact	Intact		Intact	Intact	Intact
DSC4	Intact	Intact	Bonded together	Intact	Intact	Intact
DSC5	Intact	Intact		Intact	Broken	Intact
DSC6	Intact	Intact	Intact	Intact	Intact	Intact

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[a] Yttrium hydride samples were transferred in several batches from the Hot Fuels Examination Facility to ARL beginning in April 2022 and spanning until July 2022. While in the ARL, samples were kept in aluminum or steel containers. Exposure to oxygen and nitrogen was minimized by storing the containers under argon atmosphere using an airtight "gas box" in the hot cells and the argon atmosphere Casting Laboratory Glove Box (CLGB). However, all samples did spend some time in an air atmosphere during the physical analysis and measurements which were performed in the radiochemistry glovebox. The metal containers for storing the samples could not be purged with inert gas after being opened in air unless they were reopened and closed in the CLGB. Samples for hydrogen analysis were not transferred into the CLGB until December 2022, and they were exposed to air atmosphere for much of the time after their physical analysis which was performed through July, August, and September of 2022. While waiting for hydrogen analysis, the samples were returned to the hot cells and kept in the argon-filled gas box to minimize air exposure.

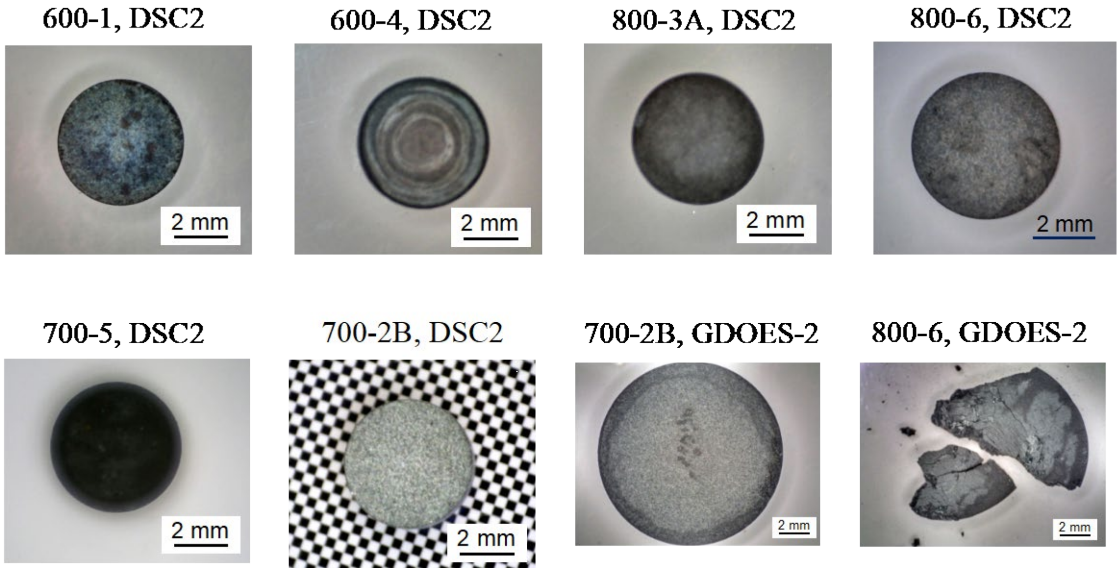
600-1, DSC2
600-4, DSC2
800-3A, DSC2
800-6, DSC2
700-5, DSC2
700-2B, DSC2
700-2B, GDOES-2
800-6, GDOES-2

Figure 1. Pictures of the selected samples that were subject to IGF analysis.

Samples were sheared into halves. One half of each sample was designated and secured for another post-irradiation examination (PIE) activity. The other half of each sample was used for hydrogen measurements. Selection was performed randomly to determine which half of the samples were used for hydrogen measurements. An additional sectioning was performed for the 800-3A DSC2 specimen to separate a free disc from two other disks fused to its top and bottom, as shown in Figure 2. The 800-6 GDOES sample degraded significantly during storage. Much of the sample had turned into a powder with a few large chunks. A chunk with a weight of 0.1026 g was chosen for hydrogen analysis. Each sample then was crushed and sub-sampled six times. The results of the replicates were used to calculate the average hydrogen concentration for the yttrium hydride disk from which they were taken. For reference, the percentage of the sample taken for analysis varied from approximately 15% for DSC samples to under 2% for GDOES samples. Samples taken from the crushed material were large enough to pick up with tweezers, but never weighed more than 3.1 mg.

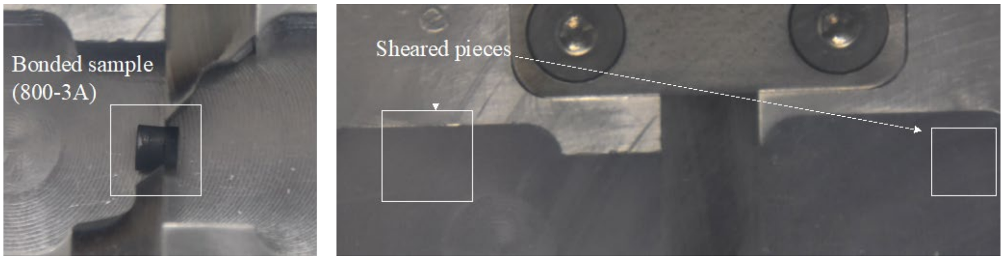

Figure 2. Picture of the bonded sample (800-3A) and shearing set-up (left). Material came apart cleanly and no degradation to the surface of either sample was observed with visual inspection (right).

2.2 Mass Measurements

All masses were measured using a calibrated analytical balance with assigned uncertainty of ±0.3 mg. The balances were calibrated annually by the Standards and Calibration Laboratory and assigned an uncertainty for their measurement range. Daily checks were performed on these balances using both internal and external calibration weights to confirm their functionality and accuracy before use. Results from the external calibration weight checks were recorded on a control chart so that long-term bias was evaluated. The high degree of confidence in the mass measurements was accomplished through comparative measurements of certified reference weights that were similar in mass to the sample. Three weights totaling 60 mg were kept in the balance enclosure to minimize any thermal variance. Between each sample measurement they were weighed, and the results recorded. Any deviation in the mass was applied to the mass measurements as a correction. Two weights were measured in a narrow mass range for each sample. Bounding the range reduces the effect of balance non-linearity on the resulting mass measurement. The first measurement for each sample was a tare weight of the tin capsule container (Alpha Resources, Part#AED4131, Lot#BN358089). This weight varied between 56–59 mg. Capsules outside of this weight band were discarded and a new capsule was chosen. Sample material was added into the capsule until it contained 2.8–3.1 mg, then the total mass was recorded. This rigor ensured that the balance responded in the correct manner and was accurate to the level required by the project.

2.3 Inert Gas Fusion Analysis

An ELTRA ONH-2000 IGF analyzer was used for hydrogen measurements. Prior to reference standard and irradiated specimen tests, blank runs with empty tin capsules were performed to evaluate and correct for the hydrogen content of the capsules during the data analysis. The instrument was calibrated with National Institute of Standards and Technology (NIST) traceable and certified reference standards (see Table 2) having hydrogen contents bounding those predicted for the sample. For this analysis, a powdered standard material was used, and the mass of the standard analyzed was varied to bound the expected range. The calibration was then checked by analyzing an independent NIST traceable certified reference standard with hydrogen content near the middle of the calibrated range. This process was repeated for each range of interest in the analysis. After calibration check[b], the samples were tested. An additional check standard run was applied at the end of the sample batch and at least every 1 hour during the analysis to evaluate the detector drift[c].

The hydrogen concentration of each sample ($[\mathrm{H}]_{\mathrm{sample}}$) was calculated using Equation 1:

$$[\mathrm{H}]_{\mathrm{Sample}} = \frac{\mathrm{K_H}\left(\mathrm{A_{Sample}} - \mathrm{A_{Background}}\right)}{\mathrm{M_{Sample}}} \tag{1}$$

where $\mathrm{K_H}$ is the sensitivity constant of the instrument for hydrogen determined through the analysis of a known standard, $\mathrm{A_{Sample}}$ is the sample peak area, $\mathrm{A_{Background}}$ is the sum of all background contributions to the peak area determined experimentally, and $\mathrm{M_{Sample}}$ is the net weight of the sample subtracting out the weight of the capsule. The average hydrogen content is then calculated as the arithmetic mean of a set of individual samples as shown in Equation 2 where $[\mathrm{H}]_i$ is the calculated hydrogen concentration for a given sub-sample using Equation 1, and n is the number of replicates analyzed.

$$[H]_{Average} = \frac{\sum_{i=1}^{n}[H]_i}{n} \tag{2}$$

Table 2. Standard reference materials used for H analysis of irradiated yttrium hydride samples.

Vendor	Part#	Lot#	H content (ppm)	Uncertainty (± ppm)
LECO	502-506-HAZ	1000	279	10

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[b] A check standard passed the analysis when its results were within the 2σ certified value on its certificate.

[c] Calibration runs were repeated once the sample results were outside of the calibration range or a check standard sample failed.

LECO	502-507-HAZ	1000	276	10

2.4 Uncertainty Evaluations

Uncertainty of the mass and hydrogen content measurements were evaluated using the Guide to the expression of Uncertainty in Measurement (GUM) Workbench Professional Version 2.4.1.406 software (http://www.metrodata.de/ver24_en.html). The total uncertainty of the measurements was calculated by propagating the individual uncertainties of parameters such as mass measurement and outputs of the IGF analyzer. The total uncertainty was derived by solving partial differential and model equations embedded in the GUM software.

Furthermore, two methods for total uncertainty of hydrogen analysis with small sample sizes were developed. For both methods, a larger sample was sub-sampled, and the replicates were analyzed for hydrogen content. Method A followed the conventional measurement analysis process where each replicate's concentration was measured and averaged together to obtain the sample's concentration. For the Method B, the concentration of the replicates was not calculated. Instead, the total hydrogen content from each individual measurement was calculated by summation of the sub-sample's hydrogen content and divided by the sample's change in mass during sub-sampling. This new approach (Method B) avoided the large uncertainties relative to the small sample mass measurements for individual small samples of 3.1 mg with ±0.3 mg uncertainty.

To overcome small mass measurement challenge, the sum of these hydrogen content measurements performed for a set of replicates can be divided by the total mass change during sub-sampling to calculate the average hydrogen content among the replicates, see Equation 3 below.

$$[H]_{Average} = \frac{\sum_{i=1}^{n} K_H\left(A_i - A_{Background}\right)}{M_{start} - M_{end}} \qquad (3)$$

The numerator term (starting with summation symbol) on the right hand-side of Equation 3 represents the sum of the cumulative signal output of each replicate of a large specimen. M_{start} is the initial mass of the material taken before sub-sampling, and M_{end} is the mass of the material taken after sub-sampling. This method of the bulk hydrogen content calculation can avoid the uncertainty contribution from small mass measurements. The denominator term of Equation 3 ($M_{start} - M_{end}$) assumes no mass is lost during the sub-sampling process, and it represents the total mass of replicates. The mass loss can occur from small particulates (i) sticking to sample handling equipment or (ii) being dropped from the sample dish in which it is ground up. This error has not been corrected in this work, but the results reported in this analysis have been calculated using both methods for comparison.

3. RESULTS AND DISCUSSION

3.1 Uncertainty Results of Replicates

To improve the statistics of the hydrogen content measurements, the minimum required number of replicates taken from a single large specimen was predicted using GUM software for a mass balance where the measurement uncertainty may vary from ±0.1, 0.3, and 1.2 mg., as shown in Figure 3. Such values were based on the balance used in this work, where ±0.1 mg was the best possible value that could be obtained by the balance. An uncertainty of ±0.3 mg was assigned for masses below 1g; and ±1.2mg was the assigned uncertainty for measurements above 1g. The estimates assumed a uniform hydrogen distribution in the large specimen (250 mg) and sampled replicates (3.1 mg). For measurements where the mass uncertainty would be ±1.2mg, the total uncertainty of the hydrogen content measurement would be

50%, as shown in Figure 3. This level of total uncertainty was determined to be unsuitable for this project. As uncertainty of the balance decreased to ±0.3 mg, the total uncertainty of the hydrogen content measurement decreased to 13% and 12% for number of replicates 4 and 9, respectively. For ±0.1 mg balance, the total uncertainty of the hydrogen content measurement decreased to 6% for three replicates and was unaffected after that. Results showed that the balance uncertainty, and so for the mass measurement, had a significant impact on the uncertainty of the hydrogen content measurements. Based on these results, and considerations toward the potential for sample loss and data outliers, six replicates from a single large specimen were tested, and the balance uncertainty was certified as ±0.3mg for masses less than 1.0g.

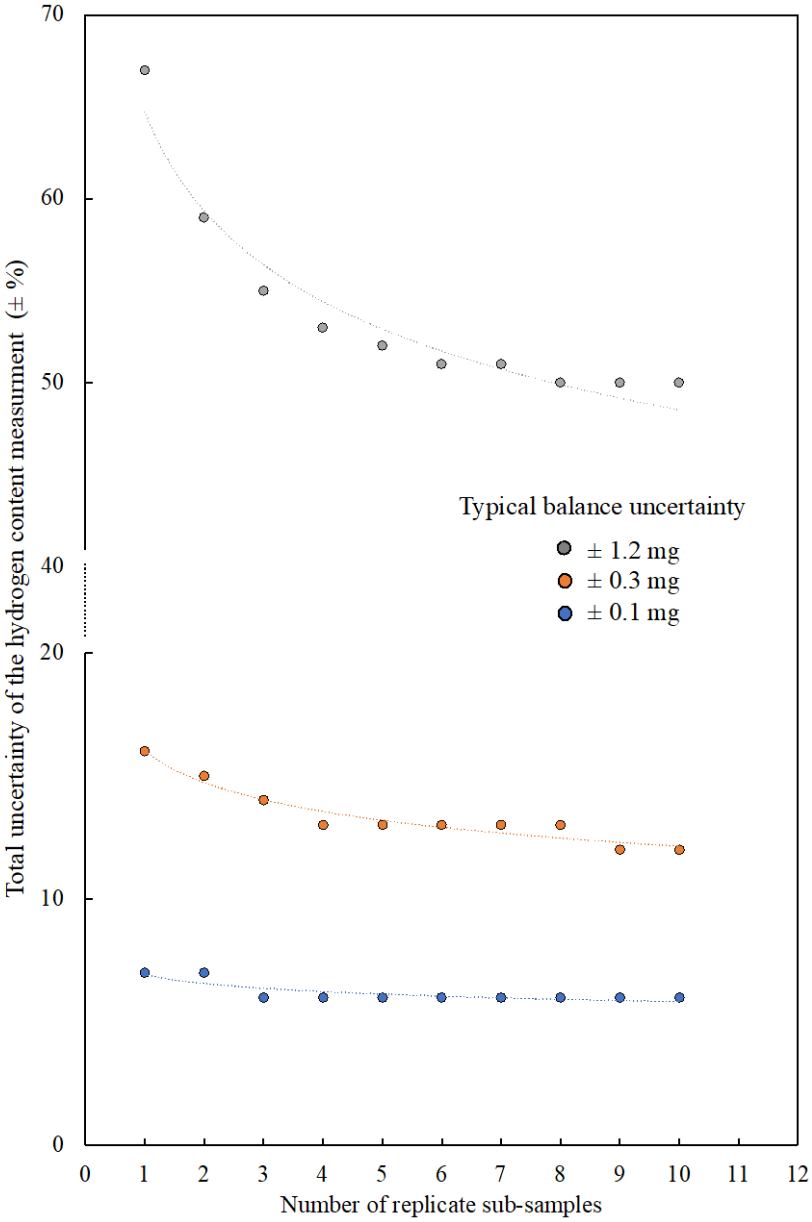

Figure 3. Impact of the number of replicates from a large specimen on the total uncertainty of the hydrogen content measurement for different balance mass uncertainties 0.1, 0.3, 1.2 mg. The GUM software was used for the uncertainty prediction.

3.2 Mass and Hydrogen Content Measurements

Table 3 shows the total mass measurements of the large specimens using uncertainty calculation Methods A and B. Mass measurements showed a nonnegligible difference between uncertainties calculated through Methods A and B. This mass measurement difference indicated that material lost during specimen preparation was clear for Method B since Method A was a direct mass measurement. Comparison of the two methods indicated that the use of Method A (the conventional method where the concentration was measured for replicate measurements and averaged together to obtain the sample's concentration) was suggested since Method A was a direct mass measurement while Method A had higher measurement uncertainty than Method B.

Table 3. The mass of large specimens (not replicates) is based on Methods A and B. For Method A, the mass was the sum of the individual sample masses. For Method B, it was the difference of the initial and post replicate sampling weights.

Capsule	Sample	Method A Mass (mg)	Uncertainty Method A (± mg)	Method B Mass (mg)	Uncertainty Method B (± mg)
700-2B	DSC2	17.8	0.2	17.9	1.7
600-1	DSC2	17.8	0.2	22.4	1.7
600-4	DSC2	17.7	0.2	22.4	1.7
800-3A	DSC2	18.0	0.2	17.6	1.7
700-5	DSC2	17.5	0.2	17.5	1.7
800-6	DSC2	17.6	0.2	20.2	1.7
800-6	GDOES2	17.9	0.2	18.2	1.7
700-2B	DSC2	17.7	0.2	19.5	1.7

Table 4 shows the average hydrogen contents of each specimen where six replicates were sampled and concentrations were calculated using Methods A and B. Hydrogen contents of the irradiated samples showed values both lower and higher than the initial value of ~21000 ppm. The hydrogen content of capsules 600-1, 600-4, 700-5, and 800-6 were higher than expected, indicating a stoichiometry increase for those yttrium hydride specimens. The sample capsules were fabricated and sealed with a 10 torr overpressure of hydrogen. This provided an initial temperature-pressure-composition equilibrium condition that would discourage hydrogen desorption from the yttrium hydride samples. It is possible that some the hydrogen from this overpressure could have absorbed onto some of the yttrium hydride samples; however, it is unclear why this behavior would not be consistent for all samples. This would also imply that hydrogen did not permeate from the TZM capsule in significant quantities. For the 700-2B (cracked) capsules, DSC2 and GDOES1 specimens had 15400 and 18200 ppm of hydrogen (Method A). For the cracked capsule's specimens, the hydrogen contents were low, as expected, due to hydrogen outgassing from the crack region. The same behavior was also determined for the 800-6 capsule while hydrogen contents of GDOES2 and DSC2 sample were determined to be 21200 and 16300 ppm using Method A.[d] It is unclear what would cause the hydrogen content in samples within the same capsule to vary so significantly. These results might suggest that some hydrogen was lost either during irradiation or post-irradiation activities. Thus, hydrogen content measurements of yttrium hydride were based on what was

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[d] Note that the initial material's pedigree, including the hydrogen content, was insufficient. We considered the expected average hydrogen content of samples was around 21000 ppm, based on targeted stoichiometry.

present after the capsule opening, sample storage, and specimen preparations, as well as the cooling period after irradiations.

Table 4. Average hydrogen content measurements after IGF for methods A and B. Six replicates from each specimen were tested. The total number of IGF runs was 36.

Capsule	Sample	Method A H (ppm)	Uncertainty Method A (± %)	Method B H (ppm)	Uncertainty Method B (± %)
700-2B	DSC2	18200	11	18100	11
600-1	DSC2	22400	12	17800	10
600-4	DSC2	22100	10	17500	10
800-3A	DSC2	17000	13	17400	11
700-5	DSC2	22400	6	22400	7
800-6	DSC2	16300	11	14200	5
800-6	GDOES2	21200	14	20900	7
700-2B	GDOES1	15400	14	14000	5

4. CONCLUSIONS

During FY-23, the hydrogen content of irradiated specimens was measured using INL's IGF analyzer. Modifications to the standard IGF process were required due to the very high hydrogen content of the yttrium hydride. These modifications included a dilution approach and the use of smaller-than-normal mass specimens with increased uncertainty. Because the dilution method did not provide reliable data, this approach was abandoned. Instead, smaller-than-normal specimen replicates ranging from 2.9–3.1 mg were used determine the hydrogen concentration. Two different calculation methods were applied for mass measurements (conventional and alternative) to reach lower uncertainty. The first approach (Method A) directly used the mass value of each replicate (~3.1 mg) to determine the hydrogen content while the second approach (Method B) used the accumulative mass of the replicates by subtracting the initial mass of the large specimen (~250 mg) from mass after sampling the replicates. The second approach was able to lower the total uncertainty of the hydrogen content measurements for half of the samples. However, the uncertainty of mass measurements increased for the Method B due to loss of material during the process. Thus, Method A was used to calculate the hydrogen content.

Hydrogen contents of the irradiated samples showed values both lower and higher than the initial value of ~21000 ppm. The hydrogen content of samples from capsules 600-1, 600-4, 700-5, and 800-6 were higher than expected, indicating a stoichiometry increase for those yttrium hydride specimens. An increase in hydrogen content was unexpected. Because the capsules were sealed with a 10 torr hydrogen overpressure to discourage hydrogen loss, it is possible that select yttrium hydride samples absorbed that hydrogen. However, the cause of this selective behavior is unclear. For the cracked capsule's specimens, the hydrogen contents were low, as expected due to hydrogen outgassing from the crack region. Interestingly, another sample from the capsule 800-6 had low hydrogen content which suggested a potential hydrogen redistribution between samples inside the capsule.

In general, hydrogen content measurements using analytical measurement tools are quantitative and informative on hydrogen loss and redistribution. Hydrogen content results need to be carefully understood by acknowledging the activities that occurred up until analytical hydrogen measurements commenced. On the pathway to such measurements, other hydrogen loss mechanisms could have been present, although without clearly identified physical non-thermal driving forces, such as internal gas release due to capsule opening, sample storage, and specimen preparations. Including such parameters is critical, especially the release of the internal gas in the capsules. Furthermore, performing additional activities such as x-ray diffraction and metallography can provide supportive information related to hydrogen loss and redistribution. The main takeaways are (i) uncertainty of the hydrogen content measurements (ii) hydrogen redistribution inside the capsule is likely present. Both may have an impact on the reactor design. In addition, an explicit sample pedigree is essential to understand post-irradiated sample behavior.

For future work, a short-term but high-temperature irradiation of single yttrium hydride specimens can be beneficial where the pressure of the capsule is measured as operando or after the irradiation prior to analytical measurements. The effect that irradiation fluence, especially approaching an end-of-life fluence, has on hydrogen concentration should be examined to support microreactor development.

5. REFERENCES

[1] A microreactor program plan for the Department of Energy INL/EXT-20-58191, Idaho National Laboratory, Idaho Falls, 2020, p. 23.
[2] M.N. Cinbiz, PLN-6268 Yttrium Hydride Post Irradiation Examination Plan, United States, 2022, p. Medium: ED.
[3] J.P. Blackledge, Chapter 10 - Yttrium and Scandium Hydrides, in: W.M. Mueller, J.P. Blackledge, G.G. Libowitz (Eds.), Metal Hydrides, Academic Press1968, pp. 441-489.
[4] W.M. Mueller, Chapter 2 - Hydrides in Nuclear Reactor Applications, in: W.M. Mueller, J.P. Blackledge, G.G. Libowitz (Eds.), Metal Hydrides, Academic Press1968, pp. 21-50.
[5] A.P. Shivprasad, T.E. Cutler, J.K. Jewell, V.K. Mehta, S.W. Paisner, C.A. Taylor, C.N. Taylor, H.R. Trellue, D.W. Wootan, E.P. Luther, Advanced Moderator Material Handbook, Los Alamos National Laboratory, United States, 2020, p. Medium: ED; Size: 66 p.
[6] M.N. Cinbiz, C.N. Taylor, E. Luther, H. Trellue, J. Jackson, Considerations for Hydride Moderator Readiness in Microreactors, Nuclear Technology (2022) 1-10.