Lattice Energy Converter (LEC) Progress Report

Frank Gordon and Harper Whitehouse, Rob Duncan and Peyton Thorn

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1. Introduction

Greetings from Frank Gordon and Harper Whitehouse and Rob Duncan, Peyton Thorn, and the Texas Tech team.

Several weeks ago, Harper and I took two working LEC cells and two samples of the original gas in those cells to Texas Tech to be analyzed by Rob Duncan and his team. They have outstanding facilities and people who know how to use them. Four samples of gas were tested. They were:

A sample of H_2 that was initially loaded into a glass jar cell;

A sample of D_2 gas that had been loaded into the brass pipe cell, as well as the gas in the operating cells.

The working electrode in glass jar cell was graphite with Pd-H particulate dried on it and the counter electrode was Al. This cell has been working continuously operating for since June 2023. We connected a DVM in the Texas Tech lab and it was still producing a voltage and current, indicating that it survived the trip. A sample of gas was transferred into a 100 ml bottle and tested. In addition, we took a sample of the H_2 gas that had initially been loaded into the cell. Results from this cell were presented at ICCF-25.

The second cell was concentric pipes with the inner pipe having been codeposited with Pd-D and a brass outer pipe as the counter electrode. This cell has been producing a voltage and current for several years and is normally operated with an external potential applied between the electrodes. We took a sample of the D_2 gas using the same technique that we used several years ago. A sample of gas from the cell was collected for testing. Results from this cell have been reported in numerous presentations.

Below is a report from Texas Tech that documents the test results. As you will see, neither cell had ^{4}He in the initial gas, but both cells contained some ^{4}He after operation. However, the concentration of ^{4}He in the cells was less than the concentration of ^{4}He in the atmosphere. ^{4}He will diffuse through most glass and would also diffuse through the nylon bushing in the pipe cell. Therefore, it is inconclusive if the 4He was produced in the cell or if it was there because of diffusion. No ^{3}He was detected in either cell after operation or in the original gas. The team at Texas Tech is interested in testing for tritium. So far, they have determined there is no more than 10^{-14} mole of tritium in the original gas. They will look at additional samples soon. Also, we are looking into how to perform the tests so that we can identify if the ^{4}He was produced in the cell or if it diffused into the cell or a combination of both.

Thanks again to Rob Duncan, Peyton Thorn, and the whole team involved in developing and operating test facility.

Best regards,

Frank and Harper

2. InovL, Inc. Helium Analysis Report

Performed by the Scientific Staff of the Center for Emerging Energy Research at Texas Tech University, Lubbock, Texas

November 2023

Two different gas samples from the InovL, Inc. LEC cells were submitted to Texas Tech CEES for the quantitative analysis of both helium-3 and helium-4 by the Quantra 3 and Quantra 6 FT-ICR mass spectrometers, respectively. Also, three different feedstock gas samples were submitted for a composition determination using the SRS residual gas analyzer (RGA) mass spectrometer. On November 16, 2023 the helium-3 analysis was performed on Quantra 3, and the helium-4 analysis was performed on Quantra 6.

Compositional Determination of Hydrogen and Deuterium Feedstock Gases using the RGA

Two different deuterium (D_2) feedstock bottles were analyzed on Wednesday, November 15th for purity using the RGA mass spectrometer. Also, a hydrogen (H_2) feedstock bottle was analyzed on Thursday, November 16, 2023

RGA Operating Parameters and Background Measurements

Prior to the analysis of injected samples, instrument and manifold background scans were performed. As with every mass spectrometer enclosed in an ultra-high vacuum chamber, hydrogen was present in the background scans. Also, after the injection of the deuterium samples on Wednesday, the background measurements on Thursday showed the presence of deuterated hydrogen, which was due to the isotopic exchange reaction 1.

$$D_2 + H_2 \rightarrow 2\ HD \qquad (1)$$

Deuterated hydrogen was not initially present within the RGA system on Wednesday or Thursday morning before the injection of the D_2 sample gases from InovL. The typical scan parameters used for all scans were: 1.) an ionization energy of 70 eV, 2.) a scan speed of 2 (slow, 1 sec/amu), 3.) 1 mA electron emission current, and 4.) a scan range of $1 < m/z < 35$ amu. The results are the averaged values of the 10 scans performed. The background signal levels in units of Torr for masses of interest are shown in Table 1 below and the values shown are typical for this system. The instrument background measurements were obtained with the inlet valve to the RGA fully closed, while the manifold background measurements had the inlet valve open, and the manifold evacuation valve closed. The manifold background measurements were obtained immediately before the gas samples were injected. The RGA level of detection (LOD) is about 5E−11 Torr.

Note that the resolution of the RGA is only 1 amu and any signal measured at $m/z = 4$ will include the sum of signals from both ^4He and D_2. Similarly, any signal measured at $m/z = 3$ will

include the sum of signals from both ^{3}He and HD. No "heavy" water, D_2O, at m/z = 20 or its isotoptomer (isomer with isotopic atoms), HDO, at m/z = 19 was detected in any of the background measurements nor in any of the InovL samples.

Table 1: RGA Background Signal Levels in Torr

Date	Background	Hydrogen	H-D (and ^{3}He)	Deuterium (and ^{4}He)	Water	Nitrogen
Nov. 15th	Instrument	8.55E−9	--	--	4.96E−11	--
Nov. 15th	Manifold #1	8.56E−9	--	--	4.82E−11	1.69E−11
Nov. 15th	Manifold #2	8.62E−9	--	--	--	--
Nov. 16th	Instrument #1	8.58E−9	7.09E−11	--	3.80E−11	3.66E−11
Nov. 16th	Instrument #2	8.53E−9	9.03E−11	--	8.69E−11	3.50E−11
Nov. 16th	Instrument #3	8.62E−9	1.09E−10	--	4.96E−11	3.51E−11
Nov. 16th	Manifold #1	8.68E−9	1.26E−10	--	1.05E−10	7.02E−11

Hydrogen is typically present in all ultra-high vacuum systems such as those required for operation of RGA mass spectrometers, and the values shown in Table 1 are typical. Only a negligibly small amount of nitrogen was detected in the manifold background measurements, as the LOD is about 5E−11 Torr.

D_2 and the H_2 Feedstock Gas Analysis

The bottles containing the D_2 and the H_2 feedstock gases were directly connected to the inlet manifold of the RGA with a short ¼ inch transfer line using Swagelok compression fittings. The RGA manifold contained a MKS 910 DualTrans Micro Pirani Piezo pressure transducer capable of measuring pressure from 1 E-5 to 1000 Torr (https://www.mks.com/f/910-micro-pirani-vacuum-transducer). The RGA manifold and the transfer line was completely evacuated to P < 1 E−5 Torr prior to transferring the feedstock gas samples. Gas sample scans were performed after the manifold background scans. Two gas samples were taken from each bottle; one sample was from the inter-valve volume, and one was from the bottle. Table 2 below shows the net peak heights (the manifold background was subtracted) in units of Torr.

Table 2: Net Peak Height Signals for the D_2 and H_2 Gas Feedstocks in Torr

Sample	Pressure, Torr*	Hydrogen	H-D (and ^{3}He)	Deuterium (and ^{4}He)	Water	Nitrogen
D_2 Control Inter-valve	196	2.25E-08	3.14E-08	1.62E-08	1.28E-11	9.81E-11
D_2 Control Bottle	155	1.76E-08	2.58E-08	1.28E-08	0	0
D_2 Receptor Inter-valve	152	1.83E-08	2.52E-08	1.23E-08	0	8.26E-11
D_2 Receptor	118	1.45E-08	1.92E-08	9.30E-09	1.9E-11	4.82E-11
H_2 Inter-valve	83.7	8.50E-08	4.46E-10	0	0	0
H_2 Gas Bottle	205	2.15E-07	3.57E-10	0	0	0

* Pressure measure at manifold with MKS 910 DualTrans transducer.

Except for the hydrogen gas bottle sample, the calibrated leak valve (flow control on RGA inlet) was ½ turn open for all samples. As expected, the largest signal in the D_2 bottles was from the deuterated hydrogen, HD, at m/z = 3, rather than the injected deuterium gas at m/z = 4. As mentioned above, the HD resulted from the isotopic exchange reaction (eqn. 1) and is not considered a contaminate in either of the D_2 feedstock samples. The presence of hydrogen in the RGA instrument reduced the injected D_2 signal due to reaction (1) above. The hydrogen signal at m/z = 2 was the largest signal seen from the two H_2 gas feedstock samples. No other atomic or molecular gas species were observed in these samples.

A nitrogen gas signal was seen on the inter-valve gas samples for D_2 feedstock gases, which indicates a very small amount of air intrusion due to leakage in the first valve. If there was significantly greater air intrusion, an argon and an oxygen signal would have been also seen. The very small amount of nitrogen seen in the D_2 receptor bottle was near the LOD for nitrogen. The small amount of water signal present in two of the D_2 gas samples is insignificant, as it is below the LOD of 5E−11 Torr. However, as mentioned above, there was not any “heavy” water detected in either of the D_2 feedstock gases.

To enhance the signal, the calibrated leak valve was 1 ½ turns open for the hydrogen gas bottle, which allowed a larger sample size into the RGA. Thus, the hydrogen peak height for this gas was more than twice that for the H_2 inter-valve sample. Despite a larger sample size, no other species were detected.

Helium Quantification of LEC Gases using the Quantra FT-ICR Mass Spectrometer

Two different LEC gases, named Bunny Cell and Pd Particular, were analyzed on Thursday, November 16th for both helium isotopes using the Quantra FT-ICR mass spectrometers. Quantra 3 was used for the ^{3}He analysis and Quantra 6 was used for ^{4}He quantification.

LEC Gas Samples Transfer to Stainless Steel Bottles

Two stainless steel 150 ml gas sample bottles with 2 bellows valves were completely evacuated while heated to P < 1.4E−6 Torr on a Pfeiffer Vacuum HiCube pumping station with a HiPace 80 turbomolecular pump. The pressure was measured with a Pfeiffer Digiline Pirani-cold cathode combination MPT 200 gauge. This combination gauge covers the pressure range from 1,000 to 5×10^{-9} hPa.

After evacuation of each bottle, the Bunny and the Pd Particular LEC gas bottles were separately connected to the pumping station inlet for the gas transfer process. The pressure of each bottle after the gas transfer was above the highest measurement available on the MPT 200 gauge, which was 1000 hPa or 750 Torr. Because the valve packing nut seal on the Pd Particular LEC cell was leaking, a small amount of air was transferred into the 150 ml sample bottle.

Quantra Helium Background Levels

Before the calibrations and sample measurements were performed, background ^{3}He and ^{4}He measurements were performed to ensure that both FT-ICR instruments (Quantras 3 & 6) and its manifolds were helium-free. Helium background measurements were also performed on the fully evacuated U-tube, which is used as a cold trap. No helium background (both isotopes) was seen inside the Quantra ICR cell, or inside the combined manifold and U-tube cold trap.

Similarly, background argon-40 measurements were also performed (a proxy gas to indicate air intrusion) and the signal levels were typically extremely low, indicating the hermeticity of the manifold and the ICR cell. Typically, a Quantra mass spectrometer has a higher sensitivity to the presence of argon-40 than to helium and thus, it makes an excellent tracer to indicate any air intrusion. Thus, the manifolds of both Quantra 3 and 6 were hermetic.

Quantra 3 ^{3}He Calibrations

A ^{3}He calibration curve was performed on Q3 on Tuesday, November 14th using a gas mixture of helium-3 in a hydrogen matrix. The calibration curve was linear, and the result is given in the table below. This calibration result, shown in Table 1, is consistent with previous results and indicates the instrument is performing normally.

LOD = Level of Detection and LLOQ = Lowest Level of Quantification. These 2 metrics are expressed in picomoles. The LOD is based on a 95% confidence level, and the LLOQ uses an International Conference on Harmonisation (ICH) factor of 10.

Table 1: Quantra 3 ^{3}He Calibrations

Date	Calibration Gas	No. of Points	Slope, Ct./pmole	He LOD, pmol	He LLOQ, pmol
November 14th	21.28 ppm ^{3}He/D2	18	0.369	0.744	3.508

Quantra 6 ^{4}He Calibration

A ^{4}He calibration curve was performed on Q6 on November 16th using a gas mixture of helium in a deuterium matrix. It was also calibrated on November 17th and 21st using a 3 component calibration gas containing 5.20 ppm He and 18.23 ppm Ne in a H_2 matrix. The 3 component calibration is used whenever an analysis of the ambient air is performed as Ne is a component of ambient air. Any Ne present is not condensed in the cold trap which uses liquid nitrogen as a cryostat nor is it absorbed by the CapaciTorr getter. Thus, it is injected into Quantra along with the other uncondensable gases. The ^{4}He calibration curve was linear, and the results are given in the table below. The calibration results are consistent with previous results and indicate the instrument is performing normally. Table 2 shows the calibration results.

Table 2: Quantra 6 ^{4}He Calibrations

Date	Calibration Gas	No. of Points	Slope, Ct./pmole	He LOD, pmol	He LLOQ, pmol
November 16th	4.996 ppm ^{4}He & D2	39	0.336	0.081	0.398
November 17th	5.20 ppm ^{4}He, 18.23 ppm Ne & H_2	48	0.397	0.099	0.491
November 21st	4.996 ppm ^{4}He, 18.23 ppm Ne & H_2	38	0.220	0.233	1.148

Quantra Helium Analysis Procedure

After a complete evacuation of the transfer line, a sample of the gas was admitted to the front-end manifold and the pressure was measured with a MKS 910 Dual-Trans pressure gauge. The sampled gas was pretreated to remove the air matrix before injection into the Quantra FT-ICR mass spectrometer. The sample pretreatment consisted of using a U-tube cold trap at liquid nitrogen temperatures to condense (liquify) any high boiling point gases such as nitrogen, oxygen, and argon. The resulting gas in the headspace of the trap was essentially free of these components and consisted of only helium and hydrogen (or deuterium). After the analysis of each gas sample, the U-tube cold trap was fully evacuated and baked out to remove any residual helium. Next, the sample gas passes through the sample loop to measure the amount of gas and then contacts a fully regenerated CapaciTorr (SAES Group) non-evaporative getter (metal alloy) to remove any hydrogen (or deuterium) present. The removal of these species before the electron impact ionizer is necessary to avoid the loss of helium ions due to charge exchange reaction. Helium has the first highest ionization potential of any atom or molecule at 24.59 eV and thus will lose its positive charge via reaction with any other species with a lower ionization potential.

Helium-3 Analysis Results

Analysis for helium-3 was performed on the 2 different samples. For the Pd Particular LEC gas sample the sample size was P = 48 Torr. After immersion of the U-tube in liquid nitrogen, the manifold pressure measured 4.0E−5 Torr, indicating complete absorption of the higher boiling point components. 2 aliquots were injected into Quantra 3. As usual, to get a larger signal using a larger sample size, Valve 3 was kept open during the sampling. No helium-3 signal was seen on

either aliquot. Next, the Bunny Cell LEC gas sample size was analyzed with a sample size of P = 24.9 Torr. 2 aliquots were injected, but no helium-3 signal was seen. The ^{3}He results are presented in Table 3 below. Because the concentration of ^{3}He in the ambient air is extremely low, it can be assumed to be zero. Thus, a control experiment to measure the ^{3}He content of the air wasn't needed or performed.

Table 3: Helium-3 Determintation Results

Sample	Sample Pressure, Torr	No. of Aliquots	Initial Amount, μmol	^{3}He Amount, pmol	[^{3}He], ppb
Inovl Pd Particular Gas	48.0	2	40.8	0.0	0.0 ±17.7
Inovl Bunny Cell Gas	24.9	2	21.1	0.0	0.0 ±16.7

Helium-4 Analysis Results and Sensitivity Determinations

Analysis for ^{4}He was performed on both gas samples submitted on November 16th. The Bunny LEC cell gas sample size was P = 9.26 Torr and the Pd Particulate LEC cell gas sample size was larger at P = 20.8 Torr. After the analysis of this sample a control experiment using an ambient air sample was performed the next day to determine the helium-4 sensitivity. This air sample size was almost the same at P = 12.0 Torr. The accepted helium-4 concentration in air is 5.24 ppm.

For consistency, both LEC cell gas samples were analyzed on Quantra 6. After injection of the LED cell gases into the manifold, it was exposed to a U-tube cold trap for the condensation of nitrogen, oxygen, argon, water, and carbon dioxide. When the U-tube trap was immersed in liquid nitrogen, the manifold pressure measured 2.88E−3 Torr for the Bunny gas sample and 4.20E−4 Torr for the Pd Particular gas sample. These very low pressure values indicated almost complete removal of all components in the gas except for some deuterium and helium. After the cold trap, 40 or 45 aliquots of the gas were injected into Quantra 6, respectively. The air samples used for the helium sensitivity determination required 40 or 45 aliquots. The results are presented in Table 4 below.

The helium sensitivity value was based upon an average value of the two ambient air sample determinations shown below. A value of 3.37 counts/Torr was used.

Table 4: ^{4}He Analyses Results for Bunny Cell and Pd Particular Cell Gases using Quantra 6

Sample	Sample Pressure, Torr	No. of Aliquots	Initial Amount, μmol	Total ^{4}He Signal, Counts	^{4}He Sensitivity, Cts Torr^{-1}	[^{4}He], ppm
Bunny Cell	9.26	40	26.3 ±2.7	19.72 ±010	3.37 ±0.03	3.31 ±0.17
Pd Particular	20.8	45	59.1 ±5.3	62.55 ±0.11	3.37 ±0.03	4.68 ±0.05
Air Sample 1	12.0	40	34.1 ±3.1	40.34 ±0.11	3.36 ±0.03	5.24 ±0.004
Air Sample 2	13.2	45	37.4 ±3.4	44.63 ±0.11	3.38 ±0.03	5.24 ±0.004

Conclusion

The measured ^{4}He concentration in the Bunny and Pd Particular Cell samples are below the accepted air (control) sample of 5.24 ppm. It is unknown what the initial ^{4}He concentration of the D_2 feedstock gas to either of the LEC cells.

3. Resources at Texas Tech used in this research

By Robert Duncan, Texas Tech

January 2024

We are working with Frank and Harper collaboratively, but they are clearly leading the LEC effort, and we are supporting them with this 4He and 3He assay work, and hopefully soon a tritium assay.

Here is some information on the instruments and experiments we bring to this research.

We are in the Department of Physics and Astronomy, Center for Emerging Energy Sciences

https://www.depts.ttu.edu/phas/cees/

Our lab capabilities are described here:

https://www.depts.ttu.edu/phas/cees/Lab_Capabilities/

Here are some of our publications:

https://www.depts.ttu.edu/phas/cees/PublicationsPatents/

The mass spec used in the LEC study is described in detail under lab capabilities, and publications. This paper describing the mass spec is available under open access (free-of-charge) from the International Journal of Mass Spectrometry:

Thorn, R.P., et al., *A quantitative light-isotope measurement system for climate and energy applications.* International Journal of Mass Spectrometry, 2021. **464**: p. 116574.

https://www.sciencedirect.com/science/article/pii/S1387380621000543

I think that the LENR community may be interested in this calorimetry paper in particular:

Lacouture, S., et al., *A solid-state, open-system, differential calorimeter.* Review of Scientific Instruments, 2020. **91**(9).

https://doi.org/10.1063/5.0013591 (not open access)

In this paper we show that a systematic exists in the design of the open-air calorimeter that consists of an active and dummy sample chamber that ride on top of a large, passive heat sink. The conceptual model that the aluminum base plate remains isothermal and that it sluffs all waste heat that flows down into the heat sink is inaccurate. Part of this heat from the active cell flows laterally across the heat sink and up the dummy cell, and this creates a heat flowing backwards in the Peltier device at the base of the dummy cell. Since the active cell Peltier signal is subtracted from the dummy cell, this reverse heat flow up the dummy cell results in the appearance of excess heat that does not really exist. We solved this problem by actively cooling the baseplate to a set temperature well below the cell and dummy temperatures, so that all heat

the flows into the baseplate heat sink (now that is actively cooled) is removed without reverse heat flow into the dummy cell from the baseplate. Other systematic errors are modeled and removed as well. Please see this paper for more details.

ARPA-E presentation: Advanced Materials Characterization and Nuclear Product Detection for LENR. Slides presented at ARPA-E, September 8, 2023

https://arpa-e.energy.gov/sites/default/files/2023-09/05_Duncan_Performer.pdf

A copy is here:

https://lenr-canr.org/acrobat/DuncanRadvancedma.pdf