CELL DESIGN FOR LASER-INDUCED BREAKDOWN SPECTROSCOPY MEASUREMENTS ON REACTIVE GAS SAMPLES

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Abstract

Uranium hexafluoride (UF_6) is the primary material used for the enrichment of uranium in the production of light water nuclear reactor fuels worldwide. Accurate, rapid quantification of uranium isotopic composition in nuclear materials is required for safeguards programs and nonproliferation purposes. One potential technique for isotopic measurements in uranium species in the field is laser induced breakdown spectroscopy (LIBS). Safe and effective application of LIBS to UF_6 for enrichment measurements is uniquely challenging due to the chemical and physical properties of UF_6, which necessitate specific handling procedures. The objective of this work is to design a cell for isotopic analysis of UF_6 that is (1) compatible with chemical and physical properties of UF_6, (2) compatible with LIBS laser, and (3) portable-sized for nuclear safeguards applications. Along with cell design, initial testing of the cell for basic performance and chemical compatibility is performed. As designed and constructed, the portable gas cell was gas-tight, chemically compatible with UF_6, and withstood long-duration laser exposure. The cell has proven capability for handling reactive gases, such as UF_6, with specification application to isotopic analysis.

1. Introduction

Nuclear safeguards programs seek to verify declared uranium enrichment at nuclear facilities worldwide for nonproliferation purposes, requiring accurate and rapid quantification of uranium isotopic composition in nuclear materials. Uranium hexafluoride gas ($UF_{6(g)}$) is used for isotopic separation and enrichment of uranium by gaseous diffusion or centrifugation [1], making it a key compound in the nuclear fuel cycle and critical for safeguards measurements. Non-destructive analysis (NDA) is a prevalent method for monitoring the enrichment of uranium because it can be performed in field settings without having to send samples to a laboratory [2]. The most commonly used NDA technique for monitoring uranium enrichment is gamma spectroscopy [2]. The advent of field-portable and handheld gamma spectrometers has led to rapid onsite enrichment verification for safeguards applications.

However, gamma spectrometers can have limitations on precision of measurements. Destructive analysis techniques, such as mass spectrometry, can achieve precise isotopic measurements for uranium enrichment verification and are the most sensitive analytical method available. Techniques such as thermal ionization mass spectrometry (TIMS) and multi-collector inductively coupled plasma mass spectrometry (MC-ICP-MS) are used to analyze uranium isotopic composition for safeguards applications [2]. Mass spectrometry techniques are extremely sensitive, but they are also costly and time consuming, and samples must be sent off site to a certified laboratory for processing and analysis. New shipping regulations make it difficult to transport UF_6. Laser induced breakdown spectroscopy (LIBS) is a potential technique for field-portable measurement of isotopic composition, with noteworthy application in nuclear safeguards work. LIBS is an atomic optical emission spectroscopic technique that utilizes pulsed lasers to generate a plasma and vaporize the sample; optical emissions are recorded by spectrometer [3]. LIBS can be used for both elemental and isotopic analysis. Because LIBS does not require sample preparation, it is potentially an excellent option for field measurements of elemental and isotopic composition. Over the last two decades, the technology has been developed to make portable LIBS systems for accurate field measurement of isotopic composition [4-6], advancing the applications of this technique beyond the laboratory setting and making it attractive for safeguards work. Methods for isotopic measurements of uranium in nuclear materials [7-10] and uranium in soil [11] have been established for LIBS, but these methods only focus on measurements in the solid state. One such method that has recently received attention from the International Atomic Energy Agency (IAEA) for the isotopic analysis of UF_6 is the Cristallini method[12]. Named after its inventor, the Cristallini method works by sorption of UF_6 gas onto alumina pellets and subsequent LIBS analysis of pellets for U isotopics, therefore removing the issues of having to transport or handle the gaseous UF_6. However, this method still requires on site sample preparation using a gas manifold and operators must ensure that there is no cratering on the sample which could lead to inaccurate results, making it beneficial to develop a potential method for *direct analysis* of gaseous UF_6 via LIBS. An approach for measurement of the isotopic composition of UF_6 in its gaseous state was recently developed [13], expanding the relevance of LIBS beyond the solid state for uranium

isotopic measurements. Previous studies by Chan *et al*.[13] provide detailed analytical characterization of the LIBS system for gaseous uranium, including isotopic analysis, detector settings, and laser parameters.

Specialized handling techniques and materials of construction are required to safely handle UF_6, adding a unique challenge to the application of LIBS for UF_6 enrichment measurements. UF_6 is a white crystalline solid at room temperature, but with a significant vapor pressure at room temperature [1, 14] and with the triple point occurring at 64.02°C and 1137.72 Torr [15]. While UF_6 does not readily react with dry air, in the presence of water vapor [16], UF_6 will hydrolyze [17-21] to generate hydrogen fluoride (HF) and UO_2F_2 according to the series of reactions presented below [20]:

$$UF_6 + H_2O \rightarrow UOF_4 + 2HF \quad (1)$$

$$UOF_4 + H_2O \rightarrow UO_2F_2 + 2HF \quad (2)$$

$$UO_2F_2 + H_2O \rightarrow UO_3 + 2HF \quad (3)$$

The reactions presented in Eqs. (1) and (2) occur very rapidly at normal atmospheric conditions, and the reaction given in Eq. (3) occurs very slowly, except at high temperatures. The HF formed in these hydrolysis reactions may be in the form of HF(g), or it may be an HF fog consisting of miniscule droplets of HF–water solution: the form it takes is dependent on humidity and temperature [16]. HF is acutely toxic [22] and presents significant human health risks if released. Another consequence of UF_6 hydrolysis and HF formation is the subsequent reaction of HF with glass and fused silica [17]:

$$4HF + SiO_2 \rightarrow SiF_4 + 2H_2O \quad (4)$$

This reaction generates a new supply of water, which can, in turn, feed the reactions presented in Eqs. 1–3, resulting in a constant cycle of HF generation. Over time, this reaction etches the glass and can eventually cause breakdown of glass vessels, leading to potential release of stored UF_6. To prevent these types of reactions, UF_6 samples must be kept under vacuum conditions in completely sealed systems [15, 16, 22], and careful precautions must be taken in handling UF_6 samples to (1) prevent any moisture from

entering the system and (2) to prevent leaks of UF_6 samples into the atmosphere. UF_6 samples can be stored in glass, although it is recommended that vessels be degassed and flamed prior to use [17]. Moreover, a thoroughly dried potassium or sodium fluoride salt "getter" [1, 17] should be included to prevent HF buildup. UF_6 is also chemically compatible with fluorinated materials such as Teflon [1]. Besides its potential reactivity with water and glass, UF_6 is a strong fluorinating compound, and as such, it can attack metals [1, 16, 18]. Therefore, it is recommended that UF_6 be handled in stainless steel or other metals alloyed with nickel [1, 18].

Best practices for UF_6 handling in a laboratory or industrial setting are well documented in the literature [15, 16, 22]. However, no design or practices for a UF_6 handling and measurement system with specific applicability to LIBS have been reported. The cell under development in this effort must (1) meet the various chemical and physical challenges posed by UF_6 handling related to reactive gas chemistry, (2) be compatible with the low-energy laser and resultant plasma, and (3) retain a portable size to be relevant for the desired uranium enrichment monitoring needed for nuclear safeguards applications. The objectives of the present work are (1) to design a cell for isotopic analysis of UF_6 using LIBS and (2) to test the cell for basic performance, chemical compatibility, and potential sample carryover between measurements. For this work, the emphasis is on portability of the gas cell, not the entire measurement setup. Besides UF_6 measurements, the cell conceived in this study, along with the associated handling techniques, have potential applicability to other reactive gases such as fluorine and chlorine compounds, which have physical and chemical properties, as well as handling challenges, that are similar to UF_6.

2. Results & Discussion

The finalized design for a self-contained LIBS cell for use with UF_6 is shown in Figure 1, with full details of cell design and components described in Experimental section. The cell consists of a Kimball Physics spherical cube vacuum chamber 6.985 cm wide with 3 sapphire viewports (2.0 mm thickness) mounted into a 6.985 cm conflat flange (MDC Precision), a custom-designed flange with gas

inlet/outlet valves (Swagelok), a pressure transducer (902B MKS instruments), and a custom-designed reservoir for excess solid UF_6 (Accu-Glass Products, Inc.). All conflat flanges were sealed to the spherical cube using copper gaskets.

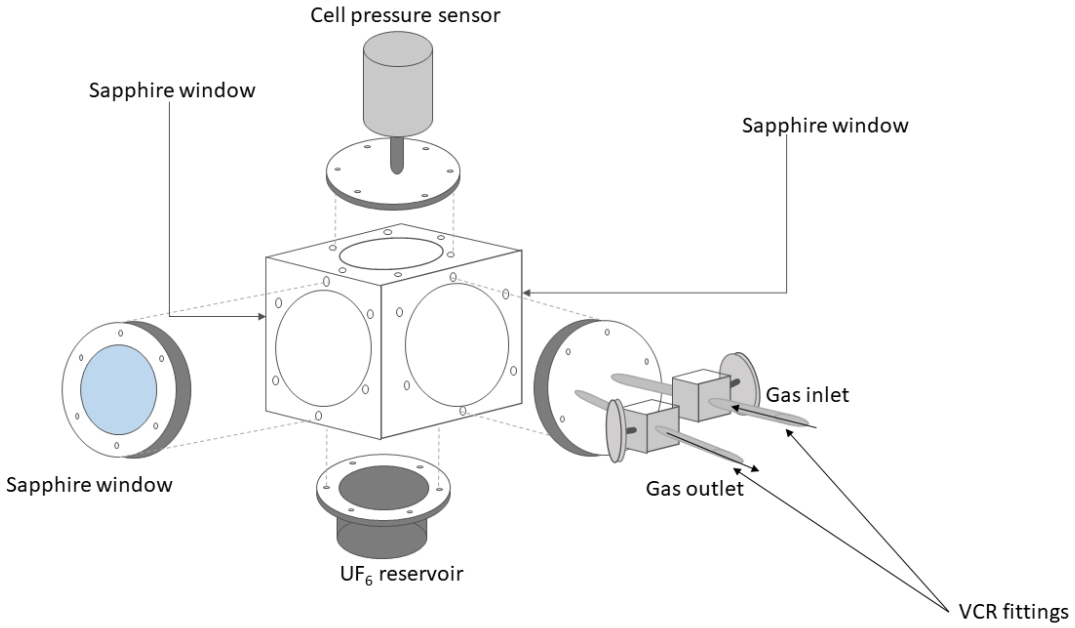

Figure 1. CAD drawing of the final cell design with three sapphire windows.

During initial testing in a cube-like chamber at the Lawrence Berkeley National Laboratory (LBNL), a laser-induced plasma was generated in 50–100 Torr air with a gaseous surrogate analyte, (methylcyclopentadienyl) manganese tricarbonyl (MMT), at <0.1 Torr. The optical emission from the plasma was collected at 90° from the laser with a focusing lens. Figure 2a depicts the measured emission from the Mn present in MMT at 257.610, 259.372, and 260.568 nm. However, during this experiment, several other atomic emission lines grew in that did not belong to Mn, especially when a higher laser pulse energy was used. Figure 2b depicts an example emission spectrum recorded when the laser pulse energy was increased to 134 mJ. These additional peaks were identified as being associated with Fe and Cr. Upon examination of the cell (Figure 3), it became apparent that the Fe and Cr peaks originated from the stainless-steel backstop of the surrogate cell. This finding resulted in the third sapphire window being installed in the

cell that was to be used for UF_6 so that the laser light could exit the cell without damaging it and a backstop installed outside of the chamber.

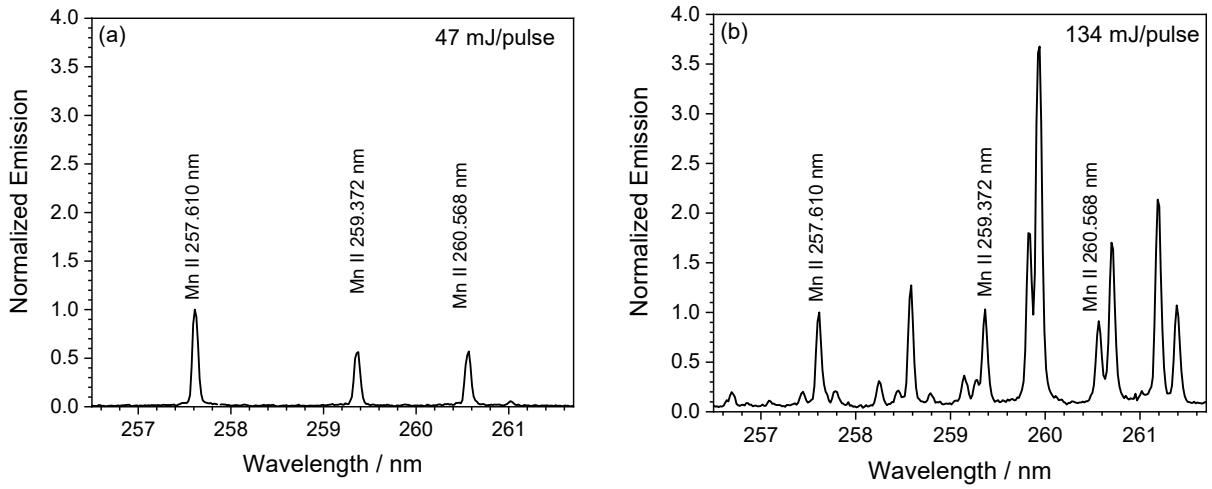

Figure 2. Measured LIBS emission spectra with low-pressure gaseous surrogate (methylcyclopentadienyl) manganese tricarbonyl (MMT) under laser pulse energies of (a) 47 mJ and (b) 134 mJ.

Figure 3. Laser ablation mark on the surrogate cell backstop plate.

142

143 Cell leak tests using pressure measurements indicated a leak-tight system. The measured pressure
144 change was acceptable (Table 1), indicating that the cell was gas tight, preventing UF_6(g) escaping the
145 cell or in-leakage of air. The diminishing rate of increase in the pressure change is only indicative of
146 outgassing from the inner surface of the cell rather than in-leakage. As a precaution, a long-term
147 experiment was initiated in which the cell was filled with 69.96 Torr UF_6, sealed, and left for a long-term
148 exposure test. The pressure of this cell was monitored with the MKS pressure transducer for 100 days,
149 and no statistically significant pressure changes were observed during this time. This result is consistent
150 with the measurements presented in Table 1.

151

152 **Table 1**. Pressure over time for 3 cell leak tests.

Test 1		Test 2		Test 3	
Time (days)	Pressure (Torr)	Time (days)	Pressure (Torr)	Time (days)	Pressure (Torr)
0	4.70E-04	0	7.10E-05	0	4.20E-05
1	9.60E-04	1	1.40E-04	2	1.00E-04
2	1.10E-03	2	1.80E-04	3	1.20E-04
3	1.30E-03	3	2.00E-04	4	1.30E-04
4	1.40E-03	4	2.20E-04	5	1.40E-04
5	1.60E-03	5	2.40E-04	6	1.60E-04

The cell material's compatibility with the laser system was determined through extended laser testing on a cell made at the Oak Ridge National Laboratory (ORNL) that was filled with 60 Torr N_2 and shipped to LBNL for testing. When the cell was returned to ORNL, there was no observable optical damage to the sapphire windows, indicating that the sapphire withstood the repetitive laser firings at full pulse energy (~130 mJ). After the visual inspection, the cell was connected to a manifold, and UF_6 was circulated through it. Fourier-transform infrared (FTIR) measurements of the cell after UF_6 addition showed no reaction with UF_6. Based on these results, it was concluded that firing the laser through the cell had no detrimental effect to the inner surfaces that would make them reactive towards UF_6.

To further probe the chemical compatibility of the cell's interior components and verify that the cell was leak-tight, a cell was constructed with ZnSe windows so that it could undergo FTIR analysis for an extended period of time. Figure 4, which depicts the FTIR spectra recorded over 8 days of measurements, shows little change in the intensity of the UF_6 peaks, a peak at ~1030 cm^{-1} due to trace SiF_4, and only small peaks consistent with HF. If there had been a leak in the cell, the UF_6 would have reacted with water to produce an increasing quantity of HF. The lack of ingrowth of HF over the 8-day period indicates that there was no significant leakage or permeation of water or water vapor into the cell. Overall, the FTIR data indicate that UF_6(g) was chemically stable in the cell as designed and corroborate the conclusion from the pressure measurements that the cell is leak-tight. During the 8-day timeframe of this experiment there was no evidence of resublimation of UF_6 on any of the windows of the cell. Indeed, the only visible UF_6 solid was that depicted in Figure 5 in the cell reservoir. It is worthy of note that this well could be cooled by a Peltier plate, thereby desubliming UF_6 in a harmless location and lowering the

174 UF_6 vapor pressure throughout the cell if necessary during a measurement (i.e., preventing desublimation
175 on the optical windows).
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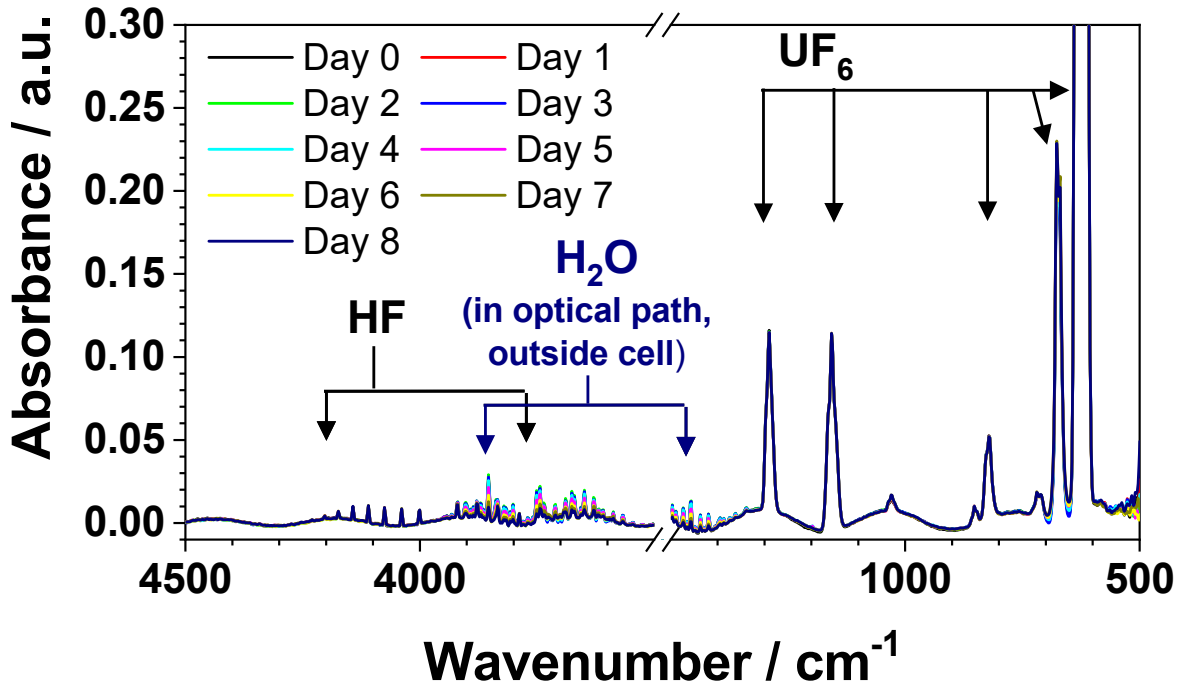

177

178 **Figure 4**. Time-resolved Fourier-transform infrared (FTIR) spectra of UF_6(g) sample in cell.

179

180 After the compatibility testing, a cell containing 1.13 g of natural enrichment UF_6 (0.711 wt-%)
181 was sent to LBNL for testing. This allowed for a saturated vapor of UF_6 to be present in gaseous form
182 inside the cell, along with solid UF_6 in the reservoir at the bottom of the cell (Figure 5). The reserve
183 amount of solid UF_6 was added to the cell because it was assumed that the generation of the plasma inside
184 the cell would destroy the UF_6 molecule entirely to its constituent atoms through an atomization reaction
185 such as depicted in Equation (5).

186 $$UF_6 \xrightarrow[laser\ induced\ plasma]{} U + 6F \qquad (5)$$

Therefore, additional solid UF_6 in the reservoir could be used to replenish the saturated vapor phase if UF_6 were destroyed by the reaction in Equation (5). Another concern for the destruction of UF_6 molecules in the vapor phase is photo-dissociation reactions. The photo-dissociation of gaseous UF_6 to solid UF_5 is shown in Equation 6 and is feasible with the laser light, or with the emission from the laser-induced plasma, as discussed in previous work [13]:

$$UF_6 \xrightarrow{light} UF_{5(s)} + \frac{1}{2} F_2 \qquad (6)$$

To arrest and partially reverse the forward photo-dissociation reaction shown in Equation (6), 20 Torr of F_2 was also added to the cell prior to shipping. Because the volume of the plasma could not be accurately measured, approximate values were utilized to calculate a conservative number of laser shots—60,000 [13]—that would result in the consumption of all the UF_6.

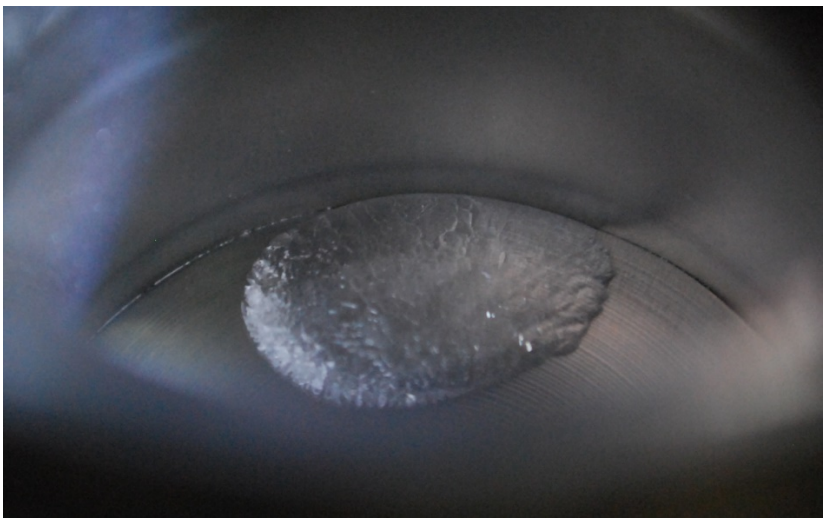

Figure 5. Excess UF_6 solid in the reservoir of the cell.

The initial experiments performed with this mixture of UF_6 and F_2 in the headspace of the cell did not lead to any visible deposition of solid materials on the interior surfaces of the cell. In addition, no significant change of pressure was noted beyond what could be explained by day-to-day fluctuations of

room temperature. According to the reaction shown in Eq. (6), if UF_6 is photo-dissociated to UF_5, then a change of pressure could be expected, along with precipitation of solid UF_5. This suggests that most of the uranium and fluorine atoms in the plasma recombined to form UF_6 molecules. As such, additional samples that were sent to LBNL for testing only contained UF_6 of the desired assay. The second and third cells sent for long-term analysis contained natural and 4.62 wt-% enriched UF_6 from ORNL stocks. During the course of testing with these samples, it was determined that there was no notable degradation of UF_6 caused by firing the laser and subsequent plasma generation into the chamber. This suggests that, moving forward, it would not be necessary for the samples to have a reserve in the bottom of the cell. The cell could simply contain UF_6 in the headspace at approximately 70 Torr.

3. Conclusions

A cell was designed for isotopic analysis of UF_6 using LIBS. The cell design required careful attention to and testing of the chemical compatibility of UF_6 with materials of construction, as well as combability of materials with the laser and the LIBS plasma. Testing of the cell revealed a gas-tight system, excellent chemical compatibility of parts with UF_6, as well as good performance of sapphire windows under long-duration laser exposure. The analytical performance of the LIBS system requires detailed analysis and optimization of the various LIBS parameters, which is beyond the scope of the present manuscript. However, analytical performance of this LIBS system is published separately in a spectroscopy-focused journal[13]. As constructed and tested, the cell has proven capability for handling and subsequent isotopic analysis of reactive gases such as the UF_6 used in this study. This capability can likely be extended to testing of other fluorine and chlorine compounds with comparable chemical handling complications, thus opening the door for enhanced isotopic analysis of challenging reactive gas systems using LIBS techniques.

4. Experimental

228 Caution! UF_6 is radioactive and forms highly toxic hydrogen fluoride in the presence of water. Safe
229 handling requires appropriate facilities and qualified personnel. All handling and testing were performed
230 in sealed manifolds and/or cells.

231 *4.1 Cell Design*

232 The initial cell design was adapted from one used to study low pressure LIBS of (methyl
233 cyclopentadienyl) manganese tricarbonyl (MMT) at LBNL. The preliminary design for a self-contained
234 cell for use with UF_6 is shown in Figure 1. The initial cell consisted of a spherical cube (Kimball Physics)
235 6.895 cm. wide with 2, 2.0 mm thick sapphire windows at right angles to each other. However, as a result
236 of issues identified during testing with the MMT at LBNL, a third sapphire window was added in place of
237 a blank stainless-steel plate opposite the laser entry window. The laser light that was used to form the
238 plasma entered and exited through the two sapphire windows that were opposite to each other, and the
239 sapphire window that was perpendicular to these windows was used to collect the light emitted from the
240 plasma. The remaining three faces of the spherical cube consisted of a custom-designed flange with gas
241 inlet/outlet valves, a pressure transducer (MKS 902B), and a custom-designed reservoir (Accu-Glass
242 Products, Inc.) for excess solid UF_6.

243 Sapphire was chosen as the material of construction for the windows because of its excellent
244 corrosion resistance to UF_6 [23] and its large optical transmission window (0.15 -–4.5 μm) [24] for
245 analytical measurements. The primary reason for the choice of the MKS 902B pressure transducer for use
246 in the cell was the chemical resistivity of the wetted surfaces to UF_6. Prior to any testing, the inner cell
247 surfaces were dried and then passivated. The passivation process included holding the cell under vacuum,
248 filling it with dry N_2 gas, evacuating the cell, and finally, filling the cell with F_2 gas (>99% purity) and
249 exposing for 72 hrs. The cell was subsequently evacuated for use and testing with UF_6.

250 To develop of a smaller, self-contained system, a light-proof enclosure was designed to house the
251 LIBS system and the UF_6 cell. The container not only served to decrease the footprint of the system, but it

also enclosed the Class 4 laser (Nd:YAG laser, wavelength 1064 nm) that was used for the LIBS measurements. With a well-engineered interlock system, the operator outside the enclosure has no exposure to the laser, the whole system, by definition, is a Class-1 laser product, which is laser safe for the operator. Class-1 laser products are the lowest hazard class, thus making the system safer and easier to operate. The laser, a laser beam-directing mirror, the UF_6 cell, the beam stop, and the optical lenses and fibers (Figure 6) are all contained inside the enclosure. Also, a Peltier cooler was situated beneath the UF_6 cell to maintain the UF_6 at a constant partial pressure of 15 Torr during measurements.

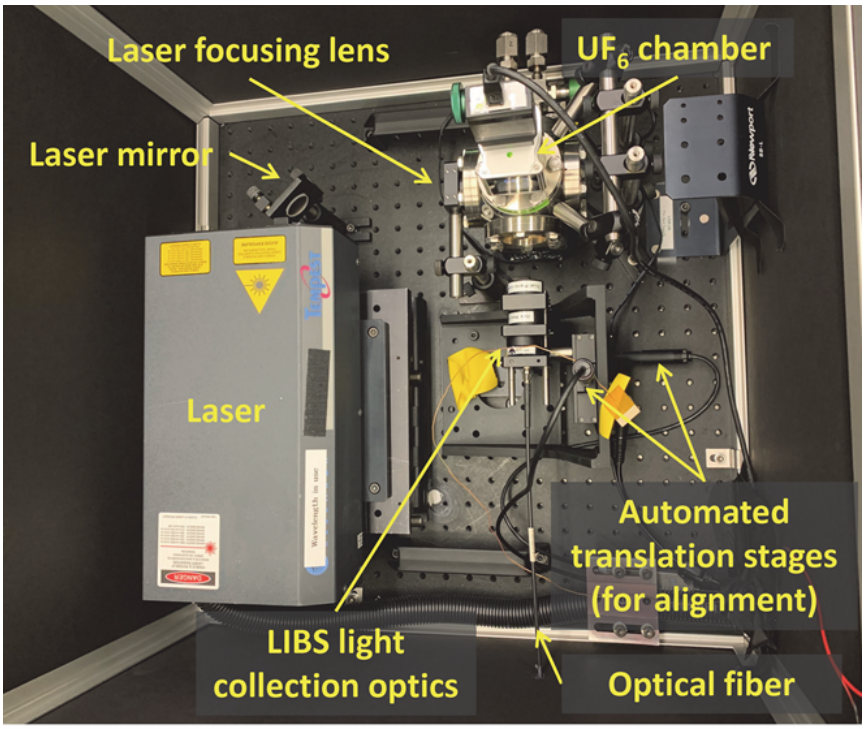

Figure 6. Photograph of interior of enclosure for laser (left) and UF_6 cell (top right).

4.2 Cell Testing

To leak test the cell, it was attached to a low-volume vacuum manifold, where it was evacuated and filled with dry N_2 three times to remove water from the interior surfaces. This pressure cycling also served as an internal check for the pressure sensor mounted directly to the cell. Cell volumes were determined by performing gas expansion tests through various sections of known volumes in the vacuum manifold and by applying the following:

$$\frac{P_1 V_1}{P_2} = V_2 \qquad (7)$$

Where P is pressure and V is volume of the cell. Average cell volumes were found to be approximately 270 cm^3. The cells were evacuated to below 10^{-5} Torr on a custom manifold fabricated at ORNL (measured from the test loop equipment *Granville-Phillips ion gauge*), isolated, and removed from the loop. As a result of minor differences in manufacturing processes, the volume for each cell must be determined individually; the approximate cell volume of the final cell was determined to be ~272.48 cm^3.

Over the course of 7 days, the pressure was monitored on the sensor attached to the cell, and no increase was observed. Following these tests, the interior of the cells was fluorinated to passivate the interior surfaces. The cells were filled with F_2 and evacuated three times to pressures of 10.2 Torr, 30.2 Torr and 100.3 Torr. After the final evacuation, both cells were filled with dry N_2. After this treatment, a cell was filled with 99.4 Torr dry N_2 (at 296.76 K / 23.61 °C) and was shipped to LBNL for testing with repetitive laser firings under the maximum pulse energy (~ 130 mJ) of the laser. It should be noted that the typical laser pulse energy for UF_6 enrichment assay is much less than this maximum energy and should be 40 mJ or less [13]. Extended laser testing with maximum pulse energy was performed to determine the materials' compatibility with laser pulses. The extended laser testing lasted for a total of 130 hours (i.e., 130 hr × 3,600 s/hr × 10 laser pulses/s = 4.7 million laser pulses), and no damage on the window was found.

After the repetitive laser-firing test, the cell was shipped back to ORNL for UF_6 compatibility testing. On receipt of this cell at ORNL, it was attached to a manifold and evacuated. The UF_6 manifold

was set up in a configuration that circulated UF_6 through the cell and through a gas cell attached to an ABB MB3000 FTIR spectrophotometer. The manifold was filled with ~30 Torr UF_6, and this was circulated through the system for several days. Infrared spectra (4 cm^{-1} resolution, 8 scans from 500 to 5,000 cm^{-1}) were recorded periodically during the 5-day run to measure for UF_6 and for the presence of HF or other degradation products. This experiment was repeated twice.

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Author Contributions

The manuscript was written through contributions of all authors. All authors have given approval to the final version of the manuscript.

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