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# A technical note on a compact inert-gas suitcase for optical measurements of air-sensitive 2D materials

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We have designed and manufactured a low-cost, easy-to-use, inert-gas atmosphere over-pressurized compact suitcase for storing air-sensitive 2D materials or any other air-sensitive samples, as well as performing optical microscopy, Raman, and photoluminescence measurements. We demonstrate that the leaking rate of this suitcase is  $1e-7$  mbar.l/s, a low enough rate to protect an air-sensitive sample stored inside for at least 4.5 days. With Raman microscopy measurements, we further demonstrate that our inert-gas suitcase can protect  $\text{Cr}_2\text{Ge}_2\text{Te}_3$ , a well-known air-sensitive 2D material for at least 8 days from degradation.

## I. INTRODUCTION

The discovery of graphene in 2004, and subsequent discoveries of other 2D materials such as hBN,  $\text{MoS}_2$ ,  $\text{NbSe}_2$  has opened a new frontier of quantum materials research. Many of these materials host new, interesting phases such as 2D superconductivity, 2D ferroelectricity, 2D magnetism which are interesting for both fundamental physics as well as technological applications. However, some of these materials are extremely sensitive to air, thus making it very difficult, to impossible to access their intrinsic material properties. The experimental research groups working on these materials mitigate this issue by exfoliating these materials in an inert-gas glove box and then encapsulating them with hBN before taking them out of the glove box to perform further characterization and measurements. Although this encapsulation process is time-consuming and cumbersome, sometimes this may not be very efficient [1], or can induce additional strain [2]. In this technical note, we demonstrate an instrumentation solution to facilitate the protection of these air-sensitive 2D materials while transporting and storing them outside the glovebox. Moreover, our inert-gas

suitcase has an optical window that facilitates optical measurements of air-sensitive materials with equipment kept in inert conditions. We demonstrate the efficacy of the box through optical micrograph, and Raman spectroscopy.

The sections below describe the usage (Section II), performance parameters (Section III), structural components (Section IV), and measurement and testing (Section V) of this box.

## II. USAGE OF THE INERT-GAS SUITCASE

The operation of the inert-gas suitcase is demonstrated in Figure 1. After the 2D material is fabricated inside an inert glove box filled with  $\text{N}_2$  or Ar, it is loaded inside the inert-gas suitcase. The suitcase is then sealed inside the glove box. In a usual commercial glove box, the internal pressure is maintained at a slightly higher pressure ( $\simeq 110$  Pa) compared to the atmosphere to prevent leakage of  $\text{H}_2\text{O}$  or  $\text{O}_2$  into the glove box. This ensures that the 2D material is sealed in an inert-gas atmosphere at a slightly higher pressure than the atmosphere. Next, the inert-gas suitcase is carried out of the glove box through the mini antechamber. After this, the suitcase can be carried to other labs and equipment where further measurements can be performed. As the suitcase is equipped with

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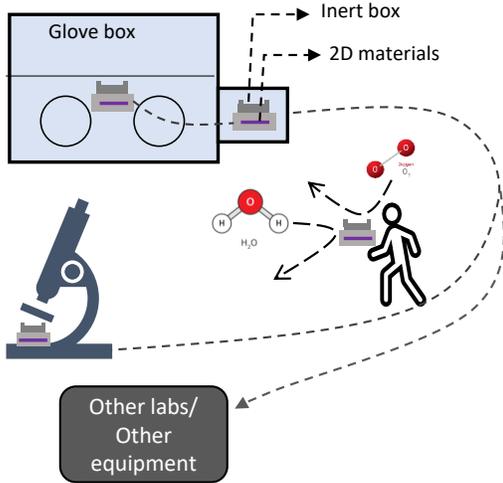


FIG. 1: A schematic demonstrating the operation of the inert-gas suitcase with an optical window. The sensitive 2D material is loaded into the inert-gas suitcase inside the glove box. Naturally, at this point, the suitcase is filled with inert gas from the glove box. Next, the inert-gas-filled suitcase containing the 2D material is taken out of the glove box through the mini-antechamber attached to the glove box. Following this, the 2D materials can be taken to optical equipment such as a Raman or photo-luminescence set up for further measurements, or other equipment and even other labs. While transporting and performing measurements the 2D material remains in the inert atmosphere of the suitcase and is protected from environmental moisture or Oxygen.

an optical window, optical measurements can be directly performed without opening the suitcase, and without exposing the sample to air. The pictures of the inert-gas suitcase are shown in figure 2. The maximum lateral dimension of this suitcase is 110 mm, i.e. the maximum dimension of the KF 50 flange. While inserting the sample the suitcase is opened and closed with the ISO-KF Vacuum Flange Wing Nut

Clamps, making the process quick and easy. In the subsections below we will describe the structural components and the materials used in the glovebox.

### III. PERFORMANCE PARAMETERS

The performance parameters of the inert-gas suitcase are given in table I. The details of the performance parameters are discussed in the following sections.

### IV. STRUCTURAL COMPONENTS

The structural components of the suitcase as shown in figure 3. are the following - 1. screws used to clamp the cover of the glass-window and the O-ring in between, 2. cover for the optical glass-window, 3. optical glass-window, 4. O-ring for clamping the glass window, 5. the lid that is closed on the base using the centering ring. 6. centering ring with O-ring, 7. the spacer used to raise the substrate to a height where the substrate is within the working distance of the objective. This is also used as the sample stage. 8. base, 9. the clamp used to seal for sample loading and unloading. In the subsections below we will elaborate on some of these components, for which special considerations had to be made. The technical drawings for the homemade parts (2,5,7,8) are given in the “Technical Drawing” zip folder. The parts 1, 4, 6, and 9 are commercially available, and their specifications are given in the table II. The optical glass-window (3) with a diameter of 50 mm and thickness 2mm was commercially bought. This optical glass-window costs 260 USD and the most expensive part. All the other parts are usually available in standard solid state lab or can be easily manufactured by a mechanical department.

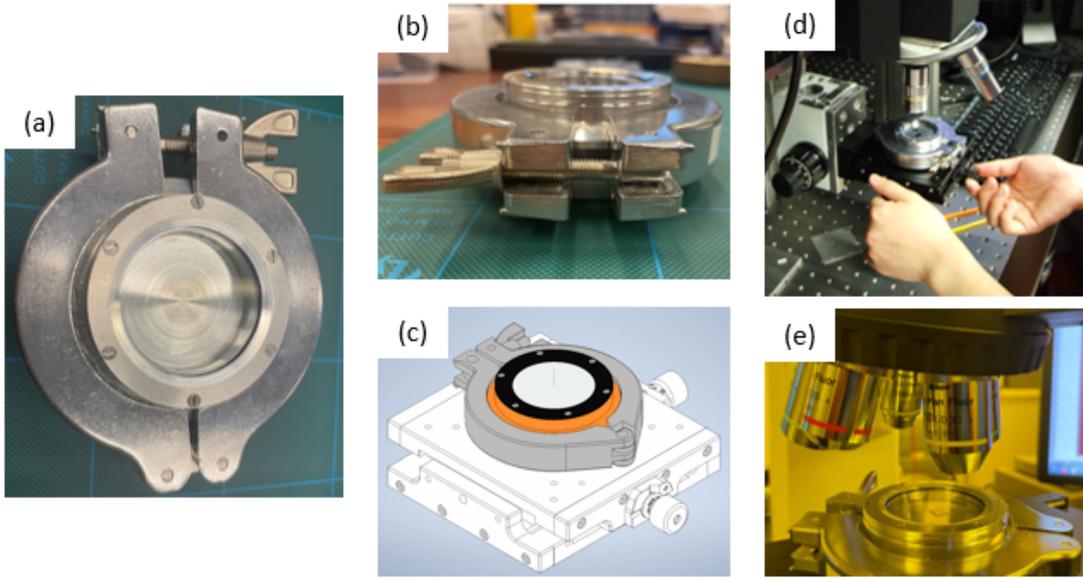


FIG. 2: The inert suitcase (a) top view, (b) sideview, (c) schematic drawing on top of XY stage, (d,e) inert-atmosphere box loaded on the sample stage of the Raman microscope. The sample is loaded into the inert-gas suitcase.

#### A. O-Ring (4)

The O-ring is an essential part of the design. It separates the outside environment from the inside, and its design determines the degree to which this separation is made and sustained. Multiple factors in the design influence the sealing. These factors are the material of the O-ring, the amount of pressure load, the type of groove, and the material it seals against.

An O-ring can be made of butyl rubber or Viton. The O-ring groove is defined by its width, depth, and shape. While in use, the O-ring is compressed between two materials. The level of compression determines the seal of the container and is given by equation: 1. The ideal compression of an O-ring in vacuum conditions is between 20 and 30 %. [3]

$$CR = \frac{\varnothing \text{ O-Ring} - \text{Groove depth}}{\varnothing \text{ O-Ring}} \cdot 100 \quad (1)$$

It is known that butyl rubber O-rings that are cycled between atmosphere and vacuum on a daily basis harden over many years and then, because of the compression and ageing, develop micro cracks. According to our experience Viton does not degrade in this manner. Because of this mechanical property and low out-gassing level we chose to work with a Viton O-ring.

The gland fill of the O-ring is determined by the O-ring cross sectional area (CSA) and gland CSA (equation 2).

$$\text{Gland fill [\%]} = \frac{\text{O-ring CSA}}{\text{Gland CSA}} \cdot 100 \quad (2)$$

Given these considerations, we chose a Viton O-ring (designated as 031) with a diameter of 1.78 mm. The inner diameter of the O-ring was 44.17 mm. The Gland depth is 1.2 mm; with these dimensions, a compression ratio of 25% is achieved. The width of the gland is 2.4 mm. With all these dimensions, the volume of the

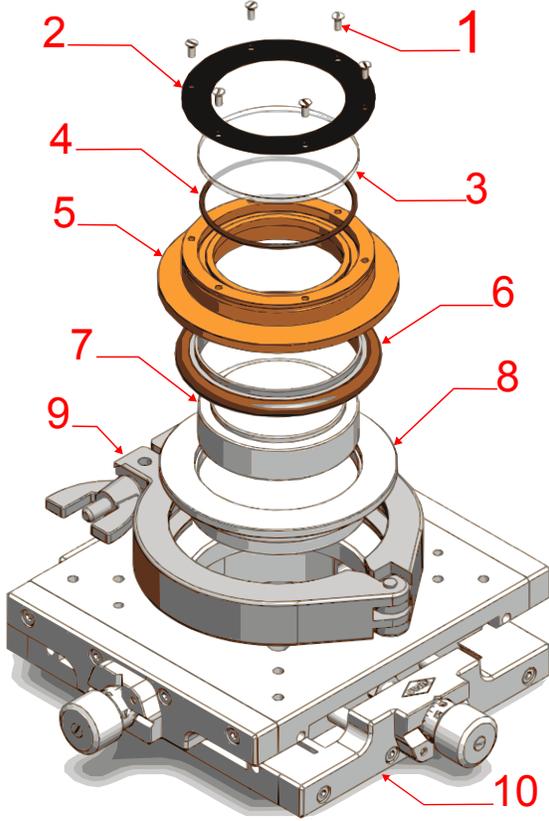


FIG. 3: An exploded view of the container. The golden part is made of aluminium and is colored for contrast. 1. screws used to clamp the cover of the optical glass-window and the O-ring in between, 2. cover for the optical glass-window, 3. optical glass-window, 4. O-ring for clamping the optical glass-window, 5. the top lid that is closed on the base using the centering ring, 6. centering ring with O-ring, 7. the spacer used to raise the substrate to a height where the substrate is within the working distance of the objective. This is also used as the sample stage. 8. base, 9. the clamp used to seal for sample loading and unloading. 10. the xy-stage is a part of the Raman spectroscope and has a hole that we use to secure the base of the suitcase.

gland filled by the O-ring is 86%, ensuring a

good seal.

### B. Optical glass-window (3)

The Raman microscope used in this project is a homemade system, interfaced with a commercial spectrometer/ EMCCD camera (Princeton Instruments IsoPlane 160, ProEM 1600) and an OptixCam Summit K2 for the optical images. Raman spectroscopy measurement is performed with two lasers with wavelengths 532 nm and 457 nm. This corresponds to a frequency-doubled Nd:YAG laser and a frequency-double Nd:YVO4 Laser. [4] Pairing a suitable optical glass with the laser for optimal results is essential.

When an optical glass-window is introduced to a converging beam, the focal length is modified due to the difference in refractive index of glass and air. According to small angle approximation, the shift in focal length can be calculated from, 3,

$$\Delta f = \frac{n-1}{n} \cdot d \quad (3)$$

where the  $\Delta f$  is the shift in focal length,  $n$  is the refractive index, and  $d$  is the thickness of the optical glass-window. Therefore, it is essential to choose a thin glass. In contrast, the glass needs to be thick enough to support the pressure difference, between the outside and inside of the suitcase. While the suitcase is placed in ambient condition, this pressure difference is relatively low,  $\sim 110$  Pa. We estimated the the minimum required thickness with equation 4 [5]

$$t = r \cdot \sqrt{\frac{P \cdot K \cdot SF}{M}} \quad (4)$$

Here,  $r$  is the radius of the unsupported glass,  $t$  is the thickness of the glass,  $P$  is the pressure difference,  $K$  is an empirical constant based on whether the glass is clamped or not (0,75 or 1,125, respectively),  $SF$  is the safety factor ranging from 1.5 to 7 and an ideal value of 4,  $M$  is the modulus of rupture of the glass. The modulus of rupture also plays an essential role in

the ability to withstand the clamping forces between the O-ring and the metal clamp.

Multiple types of glasses are optically transparent to 532 nm laser light. However, transmission losses will occur at the interface between the optical glass-window and the air. Therefore, using an optical flat with an anti-reflective (AR) coating is important. For roughly collimated light this is called the normal incidence reflection coefficient (R) and is given by  $\text{pow}((n_1-n_2)/(n_1+n_2), 2)$ . For a typical piece of glass with  $n_1=1.5$  and  $n_2=n_{\text{air}}=1$  this is 4%. This loss also occurs for the Raman-shifted light. The total loss is therefore well below 10%. This is not negligible but for some a lower-cost piece of glass without AR coating might still be a consideration.

Another consideration regarding the optical flat is the flatness of the glass piece. This is important in two aspects of the design. The optical glass-window needs to be flat enough not to cause aberrations. There are also mechanical design considerations, such as the optical flat not being compressed by two metal pieces.

In the design, the choice of fused silica was made. Fused silica has a modulus of rupture of 70 MPa. The thickness of the chosen glass window is 2 mm, and according to equation 4 is strong enough to sustain 110 Pa pressure difference. The refractive index of fused silica is 1.4585. The optical glass-window has an anti-reflective coating to reduce the reflections in the 532 nm range.

## V. MEASUREMENT AND TESTING

The inert-gas suitcase was tested with two different techniques. The first of these is the leak-testing and the second is the Raman spectroscopy. The results of those tests are further elaborated below.

### A. leak test

After the assembly of the Raman container, tests were conducted to determine the instru-

ment's efficacy. Inside the glove box, the suitcase is sealed at a pressure 110 Pa higher than atmospheric pressure. When the suitcase is taken out, it first enters the mini-antechamber which is pumped and purged 3 times. Hence, while the mini antechamber is being pumped, the inside of the suitcase stays at 101.1 kPa pressure higher than its surroundings. Naturally, the first test was to determine whether the optical glass-window is able to resist the pressure difference of the vacuum of the antechamber.

A Pfeiffer SmartTest HLT 570 was used to test the leak rate of the optical glass-window as the leak-rate of the other seal with the ISO-KF flange is already known ( $1e-9$  mbar.l/s). After pressurizing the container to a pressure of 400 Pa with Helium gas, the window design was tested to a leak rate of  $10^{-7} \frac{\text{mbar.l}}{\text{s}}$ .

As the suitcase is closed in the glovebox environment with an overpressure of 110 Pa, it will maintain that overpressure in the laboratory environment. It is important to remember that, as long as there is an overpressure in the suitcase, the diffusion rate of oxygen and moisture into the suitcase should be very low. With a volume of  $3.88 \times 10^{-5} \text{m}^3$ , the leak rate will cause the overpressure inside the Raman container to decay in 4.5 days. Typical Raman or photoluminescence experiments require the sample to be kept inside the suitcase for a few hours without being exposed to air. Hence leak rate of 4.5 days far exceeds the requirements.

### B. Optical micrography

Figure 4 demonstrates the quality of the optical image taken through the optical window of the suitcase. Figure 4a) is a direct image of an exfoliated 2D flake without any optical window in between. Figure 4b) is captured through the optical window while the sample is mounted inside the inert suitcase. Even though the images were taken with different microscopes, it is quite evident that the optical window degrades the quality of the image. However, it is still good enough for relocating 2D flakes, especially after



FIG. 4: Image of a 2D flake of  $\text{Cr}_2\text{Ge}_2\text{Te}_3$  with (a) the microscope inside the glovebox (The objective is 100x) (b) Raman microscope (OptixCam Summit K2 sensor and The objective is 50x) outside the glovebox through the glass-window. The scale bar in (a) is 15  $\mu\text{m}$ .

they have been located without an optical flat in our microscope housed inside the glove box. We attribute this degradation to chromatic aberrations [6]. If needed, these aberrations can be reduced with a corrective lens or a specially-made microscope objective. However, we do not think this was necessary for our purpose.

### C. Raman: Graphene

Our final aim is to develop methods for performing Raman measurement on air-sensitive van der Waals materials with ambient-condition Raman microscope. We chose graphene as our first control sample as it does not degrade and has a very clear Raman signature.

Figure 5 shows Raman measurement on graphene. The first peak is the  $I_G$  peak at  $1584 \text{ cm}^{-1}$  and the second peak  $I_{2D}$  at  $2671 \text{ cm}^{-1}$ . This measurement demonstrates that we could successfully detect the two characteristic Raman peaks of graphene while the sample was mounted inside the suitcase. The loss in intensity is approximately 73 percent at the peaks. We find that the ratio between the intensities ( $I_{2D}/I_G$ ) is not affected by the optical flat (table III). This is important as this ratio is utilized to determine the number of layers in a graphene flake.

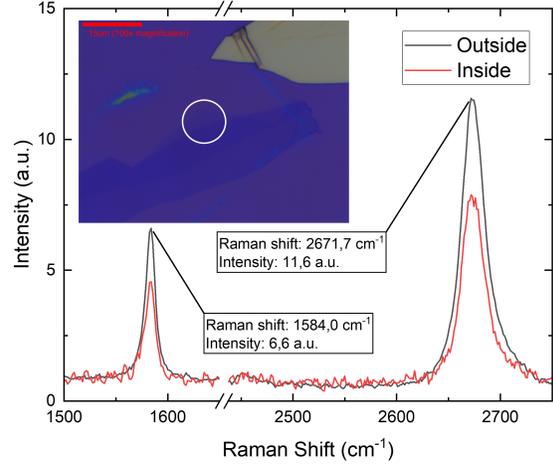


FIG. 5: Raman spectroscopy performed on a known monolayer graphene sample outside (black) and inside (red) the suitcase. The inside data is taken through the optical window. The inset panel shows a white circle to indicate the spot for Raman measurement. The graph shows two characteristic Raman peaks in graphene-  $I_G$  peak at  $1583 \text{ cm}^{-1}$  and  $I_{2D}$  at  $2671 \text{ cm}^{-1}$ .

### D. CGT degradation

Having proven that the container has a leak rate that guarantees an overpressure of at least 4.5 days and that it is possible to perform a Raman measurement with the container, we proceed to perform actual use cases on the container.

The second measurement was a Raman spectroscopy measurement of  $\text{Cr}_2\text{Ge}_2\text{Te}_3$  (CGT), a well-known 2D magnetic insulator that is sensitive to air. We measured 3 samples of CGT. The first sample was kept inside the glovebox to avoid air exposure and then encased in the inert suitcase just for the time of the measurement. The second sample was kept outside the glovebox in air, thus degrading its quality. The last sample was kept outside the glovebox inside the inert-gas suitcase for eight days. The goal of the experiment was to see if the spectrum of the third sample would look like the spectrum

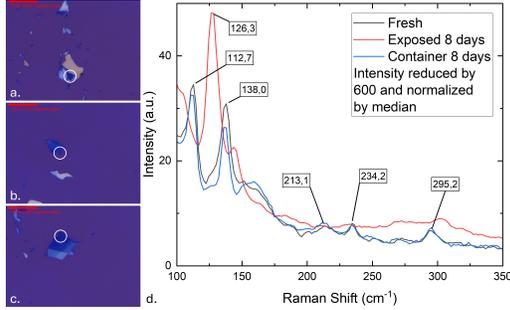


FIG. 6: Optical micrograph and Raman spectra of  $\text{Cr}_2\text{Ge}_2\text{Te}_3$  flakes freshly exfoliated and kept inside the suitcase (fresh, grey), exposed in air for 8 days (exposed 8 days, red), and stored inside the suitcase but in lab environment for 8 days (container 8 days, blue). The respective optical images are organized from top to bottom. The smaller the deviation of the blue line from the grey line, the better the performance of the suitcase.

of the sample that was exposed to air or like the sample that was kept inside the glovebox to avoid air exposure.

In figure 6, the Raman spectroscopy measurements on the first exfoliation CGT are shown. It is very clear that the sample kept inside the container has an identical Raman signature as the un-exposed, fresh sample, whereas the air-exposed sample has a very different spectrum.

During a second measurement of CGT, the results from the first measurements were replicated in a shorter time scale. Figure 7 shows that the sample kept inside the inert-gas suitcase for 22 hours does not show signs of degradation. In contrast, the air-exposed sample shows clear signs of degradation after 22 hours [7]. The difference in intensity between the samples kept outside the container and the samples inside the container is caused by the transmission losses due to the optical flat.

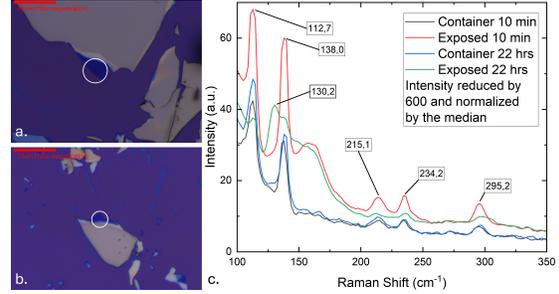


FIG. 7: The second exfoliation of CGT was interesting but also verified the results of the first Raman measurements on CGT. The grey and blue lines are measurements of the CGT inside the suitcase (10 minutes and 22 hours outside the glovebox). The red and green lines show the sample kept in ambient conditions measured after 10 minutes and 22 hours outside the glovebox, respectively. The data shows that CGT does not show degradation in the first 10 minutes, but after 22 hours, the first and second peaks (red line) have disappeared (green line).

## VI. CONCLUSION

To conclude, in this technical note, we share the designs of a low-cost, easy-to-use, compact inert-gas atmosphere over-pressurized suitcase that can be used to for storing air-sensitive 2D materials or any other samples, as well as performing optical microscopy, Raman, and photoluminescence measurements. We demonstrate that the leaking rate of this suitcase is  $1\text{e-}7$  mbar.l/s. According to this leaking rate, a sample stored inside this suitcase will be protected for a minimum duration of 8 days. Using Raman microscopy measurements, we further demonstrate that, our inert-gas suitcase can protect  $\text{Cr}_2\text{Ge}_2\text{Te}_3$ , a well-known air-sensitive 2D material for at least 4 days from degradation.

## VII. ACKNOWLEDGEMENTS

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TABLE I: The performance parameters of the inert suitcase

Parameter	Value	Comment
Sample dimension	10 mm*10 mm*0.5 mm	It is guaranteed that at least one sample with such dimension will be accommodated in the suitcase under the optical window. If strategically placed, several samples can be accommodated.
Maximum allowed dimension of the container	140 mm	This is constrained by the diameter of the mini-antechamber of the glovebox. The maximum dimension for this suitcase is the outer diameter of the KF 50 clamp and this is 110 mm.
The minimum working distance of the objective that can focus on the sample inside	13 mm	Our Raman microscope is equipped with mitutoyo M Plan APO 10X/28 and M Plan Apo 50x/0.55 objectives
Most suitable wavelength of laser	532 nm	The optical flat has an anti-reflective coating to reduce the reflection at 532 nm.
leaking rate of the glass window	1e-7 mbar.l/s	This is measured by the the leak tester Pfeiffer SmartTest HLT 570. The instrument is designed to capture an overpressure of 100 Pa. With a volume of $3.88 \times 105\text{m}^3$ , this leak rate will cause the overpressure inside the Raman container to decay in 4.5 days.

TABLE II: Parts list  
The numbers refer to the numbers in figure3

Number	Name	Specifications	Amount
1	Screws	m2 x 4mm	6
2	Top	See Technical drawing	1
3	Optical glass-window	Edmund optics 50 mm x 2mm optical flat AR coating VIS-EXT (350-700nm) $\lambda/10$	1
4	O-ring	CS: 1,78mm, ID: 41mm, norm: 030	1
5	Lid	See Technical drawing	1
6	Centering ring	ISO-KF DN 50 CR Aluminium	1
7	Spacer	See Technical drawing	1
8	Base	See Technical drawing	1
9	Clamp	ISO-KF Flange Size NW-50 Aluminium	1

Raman Shift	Inside	Outside
$\text{cm}^{-1}$	(a.u.)	(a.u.)
$I_G$ (1584,0)	4,6	6,6
$I_{2D}$ (2671,7)	7,9	11,6
Ratio:	1.7	1.7

TABLE III: This table shows the intensities of the peaks shown in figure 5. Then, a ratio is calculated to see if the window influences the ratios between the peaks. The intensities are reduced by 600 and then normalized by the median.