

# Two-Way Vacuum Transfer Device for the Thermo Fisher Scientific X-Ray Photoelectron Spectroscopy Systems

A senior design project report submitted in partial fulfillment of the requirements for the degree of Bachelor of Science in Mechanical Engineering at Harvard University

Matthias Fischer  
S.B. Degree Candidate in Mechanical Engineering

Advisor: Kevin Hauser  
Thesis Reader: Clowes Professor of Science Jennifer Hoffman

Harvard John A. Paulson School of Engineering and Applied Sciences  
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**Figure 36.** a) The VTM consistently achieved less than 1% oxidation rates for the K-Alpha+ XPS (a) and for the Nexsa XPS (b)). The outliers were most likely caused by incomplete exfoliation prior to the test run, and were more than 2 standard deviations away from the mean of the non-oxidized population. Indicated in blue is the sample mean of the population below 1% oxidation. Indicated in black is the oxidation level that is 3 sample standard deviations above the sample mean, based on the sample standard deviation of the population with <1% oxidation.

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# Abstract

Currently, air-sensitive samples analyzed with the Thermo Fisher X-Ray Photoelectron Spectroscopy (XPS) systems will be degraded following a measurement, because samples cannot be removed from the XPS without exposure to air. The Hoffman lab at Harvard synthesizes thin-film materials and studies the physics at their interfaces, the electronic properties of which critically depend on an environment free of oxygen and other reactants [1]. For the development of synthesis recipes, characterization systems like scanning tunneling microscopy (STM) and XPS are used. To allow for contamination-free back-and-forth sample transfer between growth and characterization systems, a two-way vacuum transfer module (VTM) is required.

I have developed and manufactured a two-way (2-way) VTM compatible with the Thermo Fisher K-Alpha+ and Nexsa XPS systems at the Harvard Center for Nanoscale Studies (CNS). The 2-way VTM protects samples during transfer between a glove box and the XPS systems. When transferring oxygen-sensitive  $WTe_2$ , I could show that less than <1% oxidation after a full transfer cycle can be achieved, below the measurement accuracy of the XPS. The VTM accommodates sample thicknesses up to 9 mm, including all sample holders used at the Hoffman lab. The VTM can be manufactured for less than \$1800, while a one-way transfer device from Thermo Fisher is sold for \$9000.

## 1. Introduction

### 1.1. Background and Motivation

Novel 2D atomic layer structures are expected to exhibit exceptional electronic, optoelectronic and magnetic characteristics [2]. This includes transition metal dichalcogenides (TMDs), which in different chemical compositions yield superconductors, semi-metallic materials or semiconductors [3]. The Hoffman lab researches a TMD,  $WTe_2$ , by growing samples and subsequently analyzing these to improve and validate the growth methods used. Growth methods include molecular-beam epitaxy (MBE), which enables layer-by-layer growth of crystalline thin films on substrates [4]. Because MBE is conducted under ultra-high vacuum (UHV), and defects and contaminations can be minimized with this method, it is the preferred method for thin-film growth. For growth-process development, films are analyzed with regards to their chemical composition, film morphology and electronic structure. Each of these characteristics requires a different type of measurement system, including STM and XPS. It is important to transfer samples without contamination between these measurement systems, otherwise it is impossible to determine whether defects and contaminations were introduced in the growth process or during transfer. Once a sample is exposed to air it cannot be used for further analysis. Since the growth of films is time-expensive – up to 8h per sample – it is desirable to re-use samples after measurements for further synthesis and analysis in a multi-stage process using MBE, STM and XPS. A multi-stage process could consist of synthesis in the MBE chamber, then analysis with the XPS, analysis with the STM and finally additional synthesis in the MBE chamber. For this multi-stage process, two XPS systems are available at the Harvard Center for Nanoscale Systems (CNS), the Thermo Fisher K-Alpha+ and Nexsa XPS. The Hoffman lab has its own customized MBE and STM systems. To enable multi-stage synthesis and analysis, contamination-free, two-way transfer options are required between the MBE, STM and XPS systems. In the case of  $WTe_2$ , samples should not be exposed to air during transfer, because  $WTe_2$  is oxygen-sensitive when synthesized as a monolayer structure [1]. As

shown in Figure 1, a UHV suitcase exists for two-way sample transport between MBE chamber and STM, described in detail under *Previous work*. The only commercial vacuum transfer device for the XPS enables one-way transfer only from a glove box under an oxygen-free environment to the XPS, but not back. Therefore, after an XPS analysis, an as-grown film like  $WTe_2$  is contaminated, and a new sample needs to be grown for further analysis.

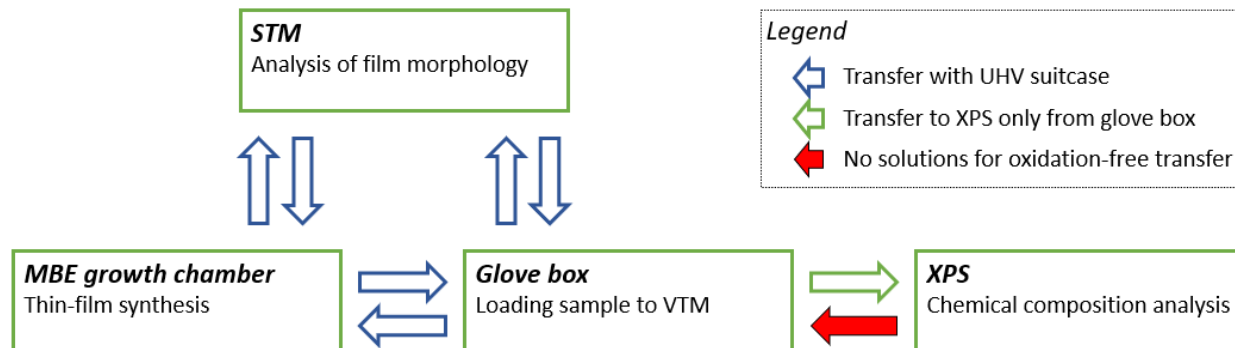


Figure 1. To analyze as-grown thin films, samples need to be transferred without oxidation between MBE, STM and XPS systems. Samples can be transferred between MBE chamber, STM and glove box with a UHV suitcase. Thermo Fisher offers a VTM for transfer only from glove box to XPS. Samples will be contaminated after XPS analysis.

## 1.2. Prospective clients

My immediate client will be Kevin Hauser and the Hoffman lab. The key problem my client is facing is a lack of a reliable 2-way VTM for their XPS systems, leading to contamination of samples and therefore additional time needed to manufacture new samples. The Hoffman lab will be able to draw value from a contamination-free transfer solution, because it will help accelerate their research.

The Thermo Fisher K-Alpha+ and Nexsa XPS systems are commercial products and are used by researchers worldwide, who could be further users of the end product. Like the Hoffman lab, they may be conducting research on air-sensitive samples that are time- and cost-expensive to manufacture. A successful solution could help them prevent sample degradation after XPS measurements to reduce sample waste and accelerate their research.

## 2. Previous work

### 2.1. UHV suitcase from Ferrovac GmbH

The Hoffman lab uses a vacuum transfer device from Ferrovac, to transport samples to and from the STM or MBE without contamination [5]. This system is capable of maintaining UHV, down to  $10^{-11}$  mbar. The device has an ion pump and batteries ensuring UHV conditions for up to 60 hours (limited by battery life). It also uses a passive system, a so-called non-evaporable getter, which can absorb particles at its surface. To interface with measurement systems it requires a standardized ConFlat (CF) flange, in the case of the Hoffman lab this is the DN40CF standard. This flange is capable of maintaining UHV conditions [6]. The knife-edge seals used in these flanges consist of a sharp metal edge pressing down on a softer metal like copper, creating seals up to  $10^{-13}$  mbar [6]. Flanges utilizing knife-edge seals are referred to as ConFlat or CF flanges as in DN40CF [6]. Sufficient contact pressure is ensured with bolts [7]. While the

K-Alpha+ and Nexsa XPS used at Harvard have CF flanges built in, these are not accessible for sample loading under vacuum conditions. Components of the XPS system would need to be removed to make a flange available. Since the XPS systems are managed by CNS and used by various users, such a disassembly on a regular basis is not practical. Therefore, the UHV solutions from Ferrovac and any CF flanged transfer devices can not be used to transfer samples to the XPS.

## 2.2. Thermo Fisher Vacuum Transfer Device

Thermo Fisher, the manufacturer of the K-Alpha+ and Nexsa systems, offers a vacuum transfer module that fits the load lock of both XPS systems [8]. The Thermo Fisher vacuum transfer module (TF VTM) is unsuitable for multi-stage sample characterization because it allows only for one-way transfer without contamination. It is not UHV rated because it is an O-ring-sealed device without a pump or a non-evaporable getter that could absorb particles to sustain ultra-high vacuum [9]. Thermo Fisher recommends this device for the transfer of samples to avoid exposure to air [9]. It is designed for transfer between an inert environment like a glove box and the XPS system [9]. The device is not designed to reseal after an XPS measurement is completed, and therefore is not capable of contamination-free two-way transfer between XPS and glove box [10].

The commercial TF VTM consists of three parts, as seen in Figure 2. When in the glove box, the sample is loaded onto and attached to the sample base [9]. Samples are attached to a plate in the base using clamps as shown in Figure 3. This plate can be set to different heights, based on the sample thickness [9]. Next, the sample cover is placed on the sample base and held in place by attaching the retaining plate to the legs of the cover [9]. Now the device needs to be evacuated either with a vacuum pump or in a vacuum chamber as they may be fitted to glove boxes, to ensure that base and cover stick together due to the pressure difference between the inside and outside of the TF VTM [9].

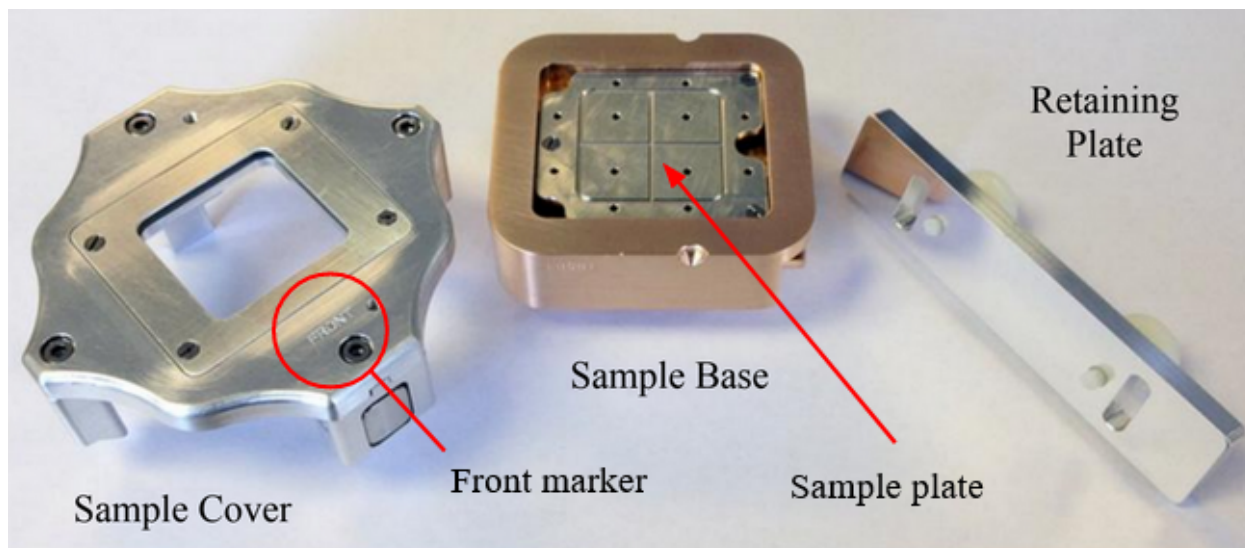


Figure 2. The commercial one-way Thermo Fisher VTM consists of a base and a cover which is held together by the retaining plate. Adapted from [10]

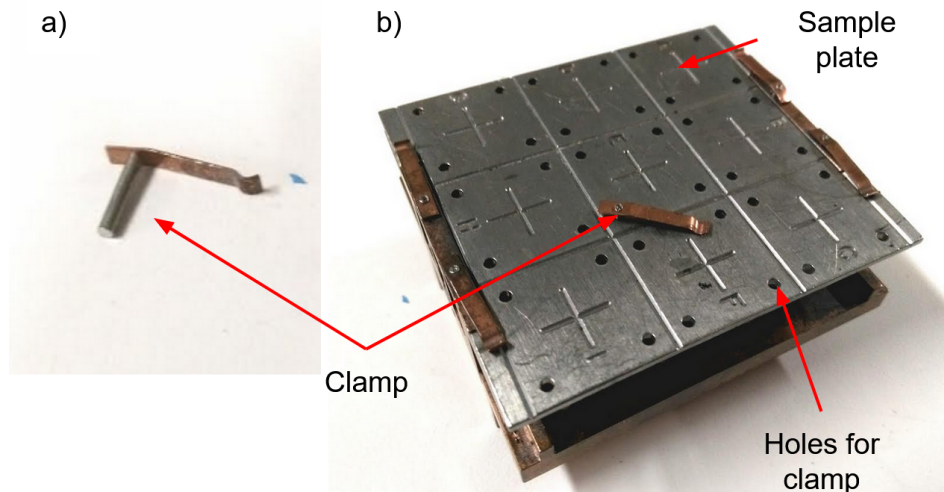


Figure 3. a) A typical clamp that can be pushed through holes on the sample plate to clamp a sample, as demonstrated in b) on a regular commercial sample stage. The clamping mechanism is equivalent to that used in the TF VTM

After evacuation is completed and the device is moved to ambient pressure, the cover and base are held together by the pressure difference between inside the TF VTM and ambient pressure [9]. Now, the device needs to be transferred to the XPS load lock within 30 minutes to prevent the O-ring from sticking. Increased O-ring stickiness inhibits a smooth cover-base separation in the XPS, causing the base to drop into the XPS (explained in more detail in section 4.3.1.2) [9]. The retaining plate is removed and then the transfer device is placed in the load lock of the XPS, as shown in Figure 4.

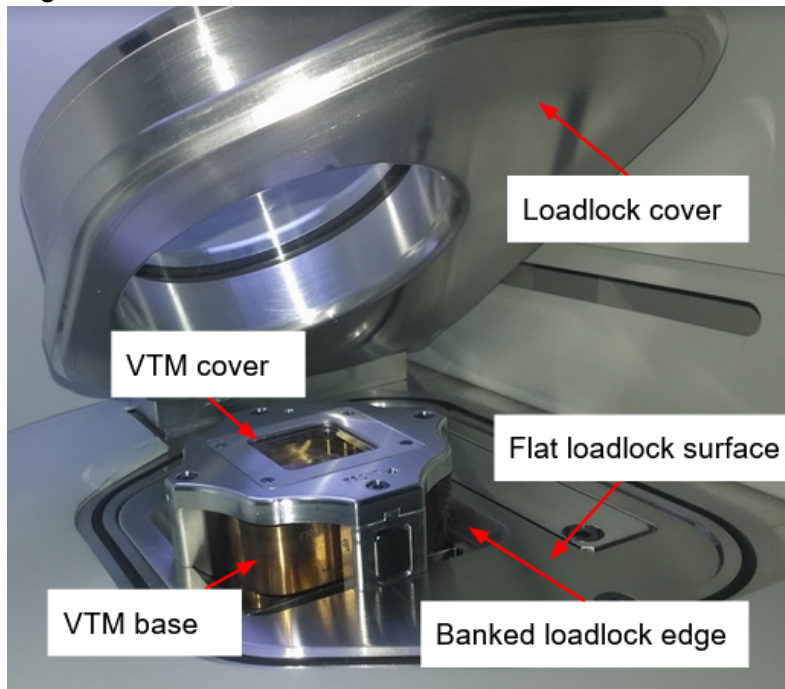


Figure 4. The sealed TF VTM is placed in the XPS loadlock, such that the legs of the TF VTM cover rest on the loadlock surface and the banked loadlock edge. Next, the loadlock cover is closed, and as the loadlock is evacuated, the TF VTM base and cover separate.

Once the TF VTM is placed in the loadlock, the load lock is closed and evacuated. As soon as the pressure in the load lock drops below the pressure within the transfer device, the sample base will separate from the cover and drop down less than 5 mm onto a transport platform, as shown in Figure 5 [10]. The cover remains loosely on top of the edge around the load lock throughout the entire measurement procedure. The area on the sample to be analyzed is determined with a camera system. The camera takes a top view image of the loadlock, such that the sample on the TF VTM base can be seen through the glass load lock cover and the viewport of the TF VTM.

Once a sufficiently low pressure is reached in the loadlock, the carrying mechanism will move the sample down and into the analysis chamber of the XPS and the analysis is completed. The main analysis chamber is pumped down to at least  $8 \times 10^{-8}$  mbar. After the complete analysis, the sample base will be moved back and vertically upwards to the loadlock [10]. However, Thermo Fisher did not design their VTM to be moved up far enough for base and cover to touch and re-seal [10]. The O-ring in the cover will not touch the base, as shown in Figure 5. This prevents the TF VTM from resealing. This is due to the gap that previously caused the base to drop down onto the transport platform before the transport platform is lowered.

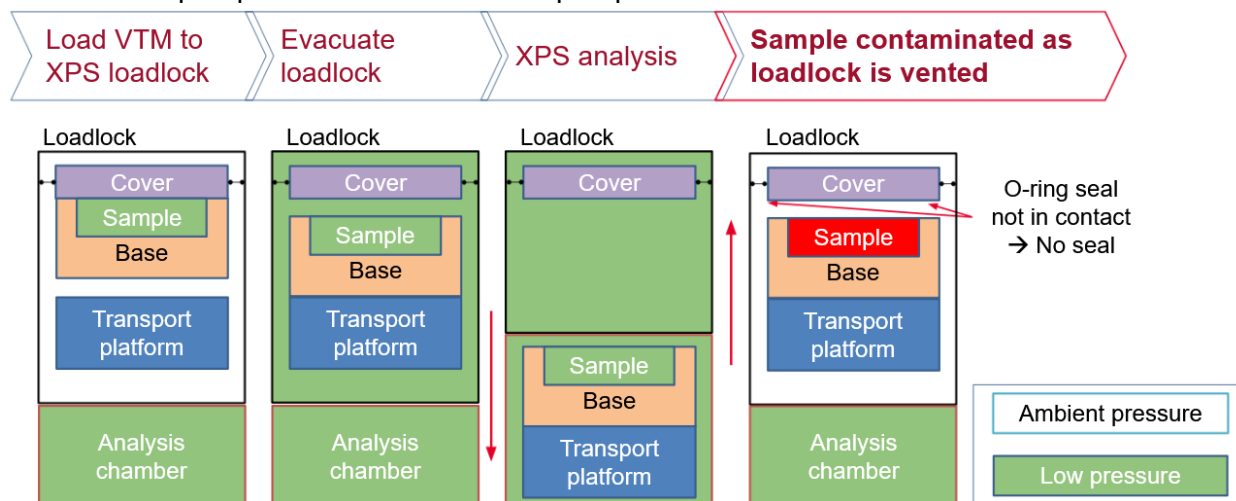


Figure 5. When the TF VTM is loaded to the XPS loadlock, cover and base stick together due to the pressure difference between the inside of the TF VTM and the loadlock. A gap exists between the bottom of the base and the transport platform. Once the loadlock is evacuated, the base drops down, and can be transferred to the analysis chamber. When the transport platform returns the base to the loadlock, the base does not seal with the cover, because the O-ring in the cover is not in contact with the base.

Thermo Fisher states that their VTM is not designed to reseat because the transport platform return height after a measurement varies between Thermo Fisher XPS systems and between XPS systems of the same model [10]. This means the gap between base and cover shown in Figure 5 will vary. Thermo Fisher told me that this return height varies by a few millimeters, and that this prevented them from finding a consistently effective solution to ensure the separation between cover and base is bridged [10]. It appears possible that the gap is an intentional design feature. The base may be designed to drop down slightly onto the transport platform to prevent an unsafe separation of base and cover in the loadlock. During late-stage testing I noticed that O-rings in my design would become sticky after sealing. If the base sticks to the O-ring in the cover when the transport platform is lowered, the base will initially remain in the loadlock, even as the transport platform is lowered. It may then drop into the loadlock and damage the XPS. It is possible that Thermo Fisher designed the base to drop down before the transport platform is lowered, because this prevents an unsafe drop of the base due to a sticky O-ring.

### 3. Technical Specifications

#### 3.1. Project Goals

My objective is to build a vacuum transfer device capable of transferring air-sensitive samples like monolayer  $WTe_2$  from a glove box with an inert-gas environment to the K-Alpha+ and Nexsa XPS systems; allow for an XPS measurement on the sample to be completed; and then transfer the sample back to the glove box. The device should prevent a chemical reaction of the sample with oxygen or other compounds in the air during back-and-forth sample transfer.

Requirement	Reason
1. No detectable sample contamination during transfer between glove box and XPS	To protect air-sensitive samples the solution needs to eliminate detectable sample contamination during back-and-forth transfer between a glove box and the K-Alpha+ and Nexsa XPS systems.
2. Secure sample loading & unloading in glove box	The solution needs to allow for secure loading and unloading of samples fixated on sample holders typical at the Hoffman lab, without damage to samples. Loading and unloading must be possible in a glove box environment.
3. Compatibility with K-Alpha+ and Nexsa XPS systems at Harvard CNS	The solution needs to allow for an analysis in the K-Alpha+ and Nexsa XPS systems at Harvard. The solution needs to seamlessly interface with the sample transportation system within the XPS system.
4. Manufacturability in Harvard ALL facilities	Manufacturing of the solution needs to be feasible with equipment available at the Harvard Active Learning Lab (ALL) facilities.

Table 1. Overarching design requirements need to be fulfilled to satisfy client needs.

#### 3.2. Resulting specifications

##### 3.2.1. No detectable sample contamination during transfer between glove box and XPS

###### *Quantitative value*

The solution needs to allow for two-way transfer between a glove box and the K-Alpha+ and Nexsa XPS systems with no detectable contamination with oxygen. To quantify oxygen contamination through air exposure, surface oxidation will be measured with the K-Alpha+ and Nexsa XPS. The detected presence of W bound to O should be less than 1% of W bound to Te after the equivalent of one transfer from the glove box to each XPS system and back to the glove box. Further details on the test procedure are provided in the *Measurement* paragraph below.

###### *Justification*

The Hoffman group aims to validate that their growth methods of air-sensitive samples including but not limited to  $WTe_2$  yield samples of high purity. To accurately measure contamination that occurred during growth with the XPS or STM, sample transfer to and from the XPS should not

add further contamination. Sample oxidation will be used to estimate contamination with chemical compounds in air in general. The XPS system can determine the relative quantity of W bound to O relative to W bound to Te. The CNS staff estimates the XPS measurement accuracy to be 1% [11]. Therefore, the quantity of oxidized W relative to non-oxidized W to be expected after the equivalent of one full transfer cycle from the glove box to the XPS and back should be less than 1%.

#### *Measurement*

To estimate oxidation to be expected after a full transfer cycle between glove box and XPS, I will first transfer samples from the glove box to the XPS in 20 minutes, and transfer them into the analysis chamber. I will then remove the device from the XPS again. To prevent contamination of samples, it has to reseal. To simulate contamination during transfer from the XPS back to the glove box, the sealed transfer device with the sample inside will be held in ambient conditions for 20 mins, and then a second XPS measurement will be performed.

Testing procedure:

- Load samples into solution in glove box
- Transfer solution to XPS within 20 minutes
- Transfer solution to XPS analysis chamber and return transfer to XPS loadlock
- Remove solution from XPS
- Hold solution in ambient conditions for 20 minutes to simulate transfer back to glove box
- Quantification of atomic percentage of W bound to O relative to W bound to Te, check that the oxidation rate is <1%

### 3.2.2. Secure sample loading and unloading in glove box

To fulfill this overarching requirement, it must be possible to load the 2-way VTM to the glove box, the 2-way VTM must be ventable in the glove box, sample holders as typical at the Hoffman lab need to be clamped securely to the solution, and evacuation of the solution needs to be possible with and without an evacuation chamber.

#### 3.2.2.1. Solution needs to fit into glove box antechamber

##### *Quantitative value*

The solution needs to fit in a diameter of 12”.

##### *Justification*

The glove box available at CNS for sample transfer between the Ferrovac suitcase and my device has a large antechamber with a diameter of >12”.

##### *Measurement*

This requirement can be tested by attempting to transfer it into the CNS glove box.

#### 3.2.2.2. Solution should be ventable by hand

##### *Quantitative value*

It must be possible to vent the solution without any actuation other than a hand and a simple tool like an allen key or screwdriver. For example, if a screw is used to force the solution components apart, the required torque should be below 14 Nm.

##### *Justification*

A potential separation mechanism would consist of a screw pushing cover and base apart. The required torque to actuate such a mechanism with an allen key or another tool should be achievable by hand. Hand-held screwdrivers with preset torques can be bought for example

with 14 Nm [12]. This appears to be a useful benchmark, even though slightly higher torques could likely also still be accommodated by hand, especially if tools with longer levers like allen keys were used. It is expected that much less torque will be needed in this case.

#### *Measurement*

It is possible to verify fulfillment of this requirement using a torque screwdriver or torque wrench. Additionally, if the device can easily be vented by hand with an allen key, this requirement is considered fulfilled. The exact torque is not central to the device functionality.

### 3.2.2.3. Sample heights of typical Hoffman lab sample holders to be accommodated

#### *Quantitative value*

The solution needs to accommodate samples and sample holders with a height up to 7 mm. It also needs to ensure that each sample holder can be mounted such that the top of the sample is at a height of 27 mm - 35 mm above the bottom of the solution base. The sample holders to be accommodated are shown in Figure 7.

#### *Justification*

Thermo Fisher recommends setting the sample plate such that the top of the sample is between 0 - 3 mm below the top of the TF VTM base, corresponding to a height of 27 mm - 30 mm, as shown in Figure 6. Thermo Fisher also provides a device that can be used to check the height of a sample holder and ensure that the sample is at the correct height. Based on this device, the top of the sample can also be at a height of up to 35 mm. It is unclear what would happen if this height constraint is exceeded, but most likely there would be an internal collision between XPS and the base. If the sample is at a height of <27 mm, the XPS may not be able to focus the X-ray beam on the sample. For all sample holders shown in Figure 7, sample mounting needs to ensure that the top of the sample is at 27 mm - 35 mm.

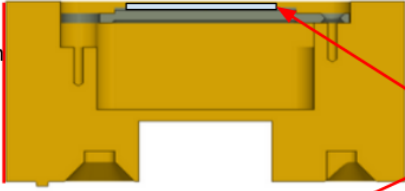
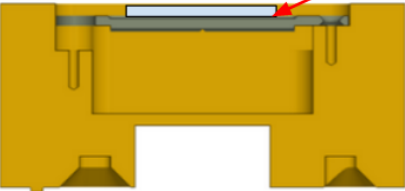


<b>Position 1</b> <b>Raised side up</b>		Sample thickness range 0 - 1 mm  Top of sample should be in this area
<b>Position 2</b> <b>Flat side up</b>		Sample thickness range 0 - 2 mm
<b>Position 3</b> <b>Raised side up</b>		Sample thickness range 2 - 5 mm  Top of sample should be in this area
<b>Position 4</b> <b>Flat side up</b>		Sample thickness range 3 - 6 mm

Figure 6. Thermo Fisher recommends that samples should be between 0 and 3 mm below the top of their VTM. The commercial TF VTM can accommodate sample thicknesses between 0 and 6mm. Adapted from [9]

Since the Hoffman lab's sample holders as shown in Figure 7 have total heights up to 7 mm, the solution should accommodate sample holders up to at least 7 mm high.

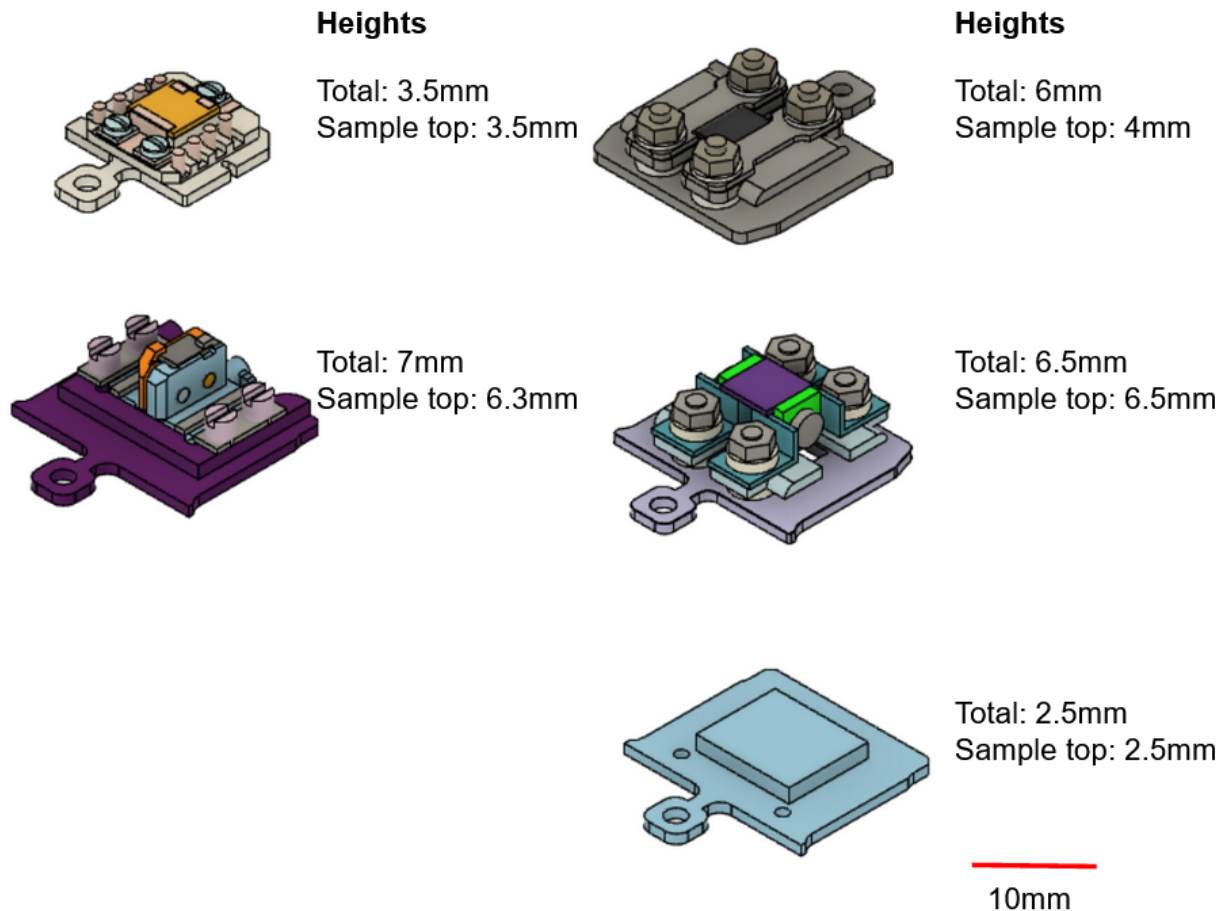


Figure 7. Sample holders to be accommodated by the solution. For each sample holder the solution needs to ensure that the top of the sample is at a height of 27 mm - 35 mm above the bottom of the solution base. Provided by the Hoffman lab.

**Measurement**

Fulfillment of this requirement can be verified in CAD, and using calipers after manufacturing.

3.2.2.4. Sample widths and lengths of typical Hoffman lab sample holders to be accommodated

**Quantitative value**

The solution base needs to accommodate sample holders of at least dimensions 19 mm x 21 mm.

**Justification**

The largest of the Hoffman lab sample holders shown in Figure 7 have widths and lengths of 19 mm x 21 mm. The sample holders are based on a common design, as sold for example by Ferrovac, which may be adapted for specific purposes [13].

**Measurement**

Fulfillment of this requirement can be verified in CAD, and using calipers after manufacturing.

### 3.2.2.5. Accommodation of standard Thermo Fisher sample clamping

#### *Quantitative value*

To allow for secure loading and unloading of samples in a glove box, and sufficient sample fixation during transfer within the XPS, the clamps from commercial XPS sample stages will be used as demonstrated in Figure 3. The clamps shown are available at CNS. To mount the samples with clamps, the solution base needs a plate with holes of diameter 0.063" - 0.064".

#### *Justification*

The sample holders to be accommodated as defined in 5.2.2 can be mounted to the commercial sample stages with the clamping method shown in Figure 3. To improve compatibility for other potential users, Thermo Fisher's established method will be used. The clamp bolts have diameters of between 0.061 - 0.062". The holes should be slightly larger, with a clearance of about 0.001" for loose fits of this basic diameter [14], resulting in diameters of around 0.063". A slightly larger clearance of 0.064" is also expected to be acceptable, as some of the holes on a commercial sample stage had a diameter of 0.064". This clamping method does not accommodate sample loading and unloading under UHV conditions.

#### *Measurement*

The hole diameter in the sample plate can be verified using calipers. If clamps can be inserted into each and fastened, this requirement is fulfilled.

### 3.2.2.6. Evacuation capability

#### *Quantitative value*

The solution needs to have a connection for an evacuation line. It should have an outer diameter of 5.3 mm.

#### *Justification*

It must be possible to evacuate the solution in the absence of a glove box vacuum chamber, e.g., in case such a chamber had a technical failure. The commercial TF VTM has a connection piece similar to that shown in Figure 8. Such a connection piece should be included in my solution. Its diameter should be slightly larger than typical pipe internal diameters available at the Hoffman lab. By attempting to push tubing with 4.5 mm inner diameter from the Hoffman lab onto the flat end of drill bits of different sizes, I was able to determine that for a diameter of about 5.3 mm, it was still possible to push the tubing on the drill bit, but it also expanded around the drill bit, indicating that it would create a sufficient seal to evacuate the 2-way VTM.

#### *Measurement*

Fulfillment of this requirement can be tested using calipers.

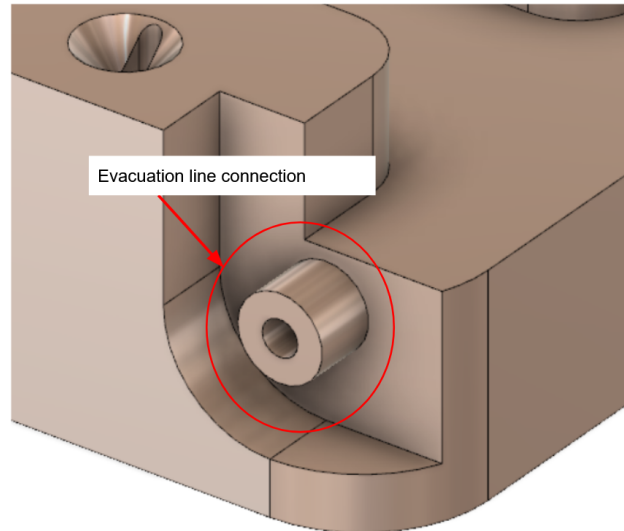


Figure 8. The solution needs a connection piece for an evacuation tube, similar to that on the commercial TF VTM shown here.

### 3.2.3. Compatibility with K-Alpha+ and Nexsa XPS systems at Harvard CNS

To fulfill this overarching requirement, the solution must fit in the XPS systems. Outgassing of the solution needs to be low enough to ensure UHV compatibility of the base. The solution base needs to be sufficiently conductive to prevent a charge buildup on the sample during an XPS measurement. A viewport needs to be available to define the analysis area on the sample prior to transfer of the solution base to the analysis chamber.

#### 3.2.3.1. Needs to fit in XPS systems

##### *Quantitative value*

The dimensions of the solution base should have dimensions  $W \times L = 66 \text{ mm} \times 60 \text{ mm}$ , as shown in Figure 9a. As shown in Figure 9b, the solution cover width and length must be within a circle of 94 mm to fit in the XPS loadlock. The overall height is already constrained by requirement 3.2.2.3 to 35 mm.

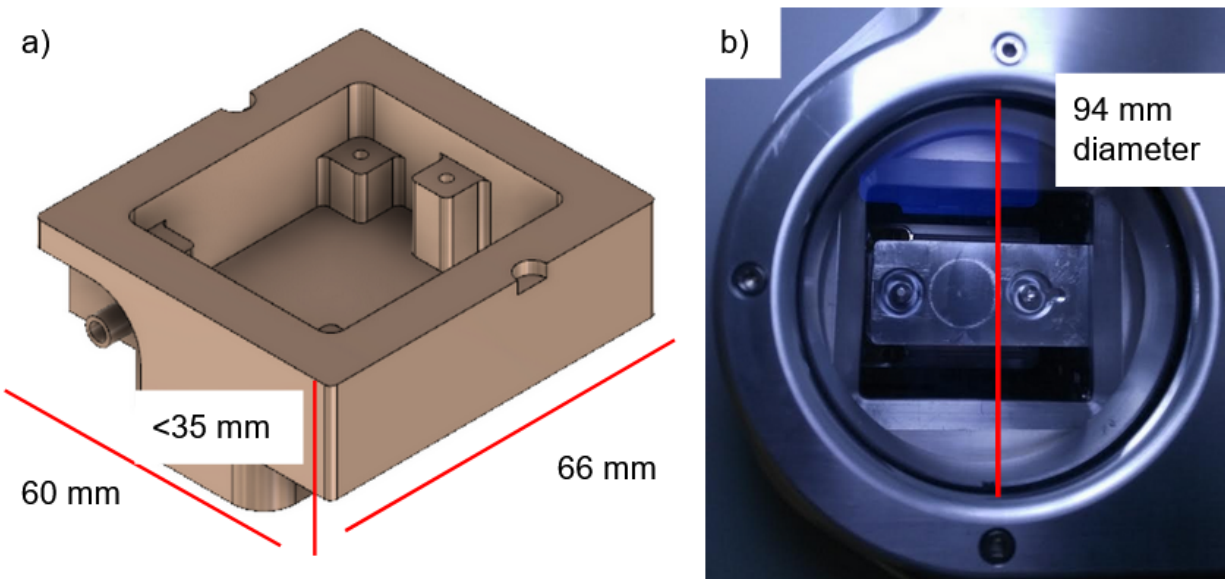


Figure 9. a) The dimensions of the solution base should be the same as for the commercial TF VTM base depicted here ( $W \times L = 66 \text{ mm} \times 60 \text{ mm}$ ). b) Top view of the empty XPS loadlock with closed cover. The solution base needs to fit into the closed loadlock of  $\text{Ø}94 \text{ mm}$ .

#### *Justification*

The solution must fit in the K-Alpha+ and Nexsa XPS to enable an XPS measurement. By using the dimensions of the TF VTM, I can ensure compatibility with the XPS transfer mechanism. It is not possible to directly access these components of the XPS, to establish a range of acceptable dimensions. Therefore,  $W \times L = 60 \text{ mm} \times 66 \text{ mm}$  for the solution base, like the commercial TF VTM base. The diameter of the XPS loadlock has  $\text{Ø}94 \text{ mm}$ .

#### *Measurement*

Fulfillment of this requirement can be tested using calipers and using the XPS systems. If transfer of the base to the XPS analysis chamber and back can be conducted without damage to the XPS, these requirements have been fulfilled.

### 3.2.3.2. Compatibility with XPS sample transportation platform

#### *Quantitative value*

The transport platform of the XPS has a specific groove pattern to hold sample stages in place. The solution base needs to fit onto these grooves shown in Figure 10a. This requires the same groove pattern as the commercial sample stages have, shown in detail in Figure 10b and c.

#### *Justification*

This requirement is needed to ensure the solution base does not move unintentionally during transfer between XPS loadlock and analysis chamber, which may result in damage to the XPS, or measurement inaccuracies if the base moves during a measurement. The grooves shown keep the sample stage in the correct position as it is transferred to the analysis chamber, and indicate to the XPS analysis chamber that a sample stage in the correct orientation is ready for measurement. Ideally, tolerances on these critical dimensions could be specified, but this would require access to the analysis chamber of the XPS, which would require XPS disassembly.

#### *Measurement*

If it is possible to transfer the solution base from the loadlock to the analysis chamber, conduct an analysis and transfer the solution base back to the loadlock, this requirement is fulfilled.

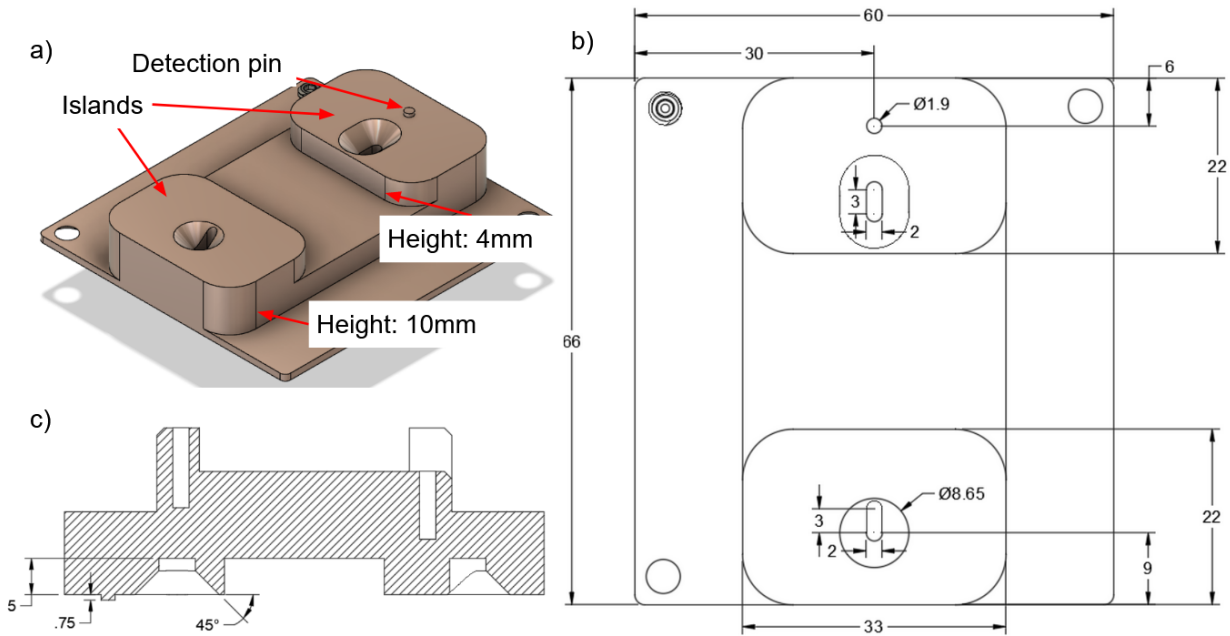


Figure 10. To ensure the solution base can be transported safely from the loadlock to the analysis chamber, and to ensure the XPS can detect the presence of the base in the analysis chamber, the bottom of the base needs to have the islands and detection pin as indicated. a) Isometric view of bottom of base with required feature heights indicated. b) Bottom view of base indicating dimensions of groove pattern on bottom of base. c) Section view of base, with depth of key features indicated.

### 3.2.3.3. Solution resealing in XPS loadlock on K-Alpha+ and Nexsa XPS systems

#### Quantitative value

To achieve the contamination minimization targets set out in 3.2.1, the solution needs to reseal in both XPS systems before the loadlock is vented.

#### Justification

If the 2-way VTM does not reseal, the  $WTe_2$  sample will be exposed to air, resulting in surface oxidation.

#### Measurement

If the contamination targets in 3.2.1 can be reached for the K-Alpha+ and Nexsa XPS at CNS, the requirements in 3.2.3.3 are fulfilled.

### 3.2.3.4. Compatible outgassing of materials used

#### Quantitative value

The loadlock of both the K-Alpha+ and Nexsa XPS system needs to be pumped down to a pressure where the base can be transferred to the analysis chamber, so the cover needs to be made of materials with sufficiently low outgassing rates. Additionally, the base needs to consist of materials with low enough outgassing rates to ensure that the analysis chamber can be pumped down to  $8e-8$  mbar with the base inside.

#### Justification

It is not clear at what pressure exactly the XPS system will allow for the base to be transferred from the loadlock to the analysis chamber, but it appears to be above  $5e-6$  mbar. The CNS

standard operating procedure for the XPS systems recommends that the analysis chamber is pumped down to at least  $8\text{e-}8$  mbar with the sample inside.

*Measurement*

If the base can be transferred to the analysis chamber and if the pressure of  $8\text{e-}8$  mbar can be achieved in the analysis chamber, this requirement is fulfilled.

3.2.3.5. Electrical conductivity between sample and contact point with XPS transport platform

*Quantitative value*

The resistance between where the sample is attached to the solution base, and the bottom of the solution base should be similar to the respective resistance for the commercial TF VTM, less than  $0.7\Omega$ .

*Justification*

The solution base needs to be sufficiently conductive, to prevent a charge build-up on the sample as electrons are ejected from the sample during the XPS measurement. An exact resistance limit could not be determined, but one of the commercial sample stages has a resistance of  $0.7\Omega$ . The resistance in the base of my solution should have a similar value.

*Measurement*

The resistance between the bottom of the solution base and where the sample is attached to the solution base can be measured using a multimeter.

3.2.3.6. Viewport in solution cover to define analysis area

*Quantitative value*

A transparent viewport is needed in the solution cover, with minimum dimensions of  $1'' \times 1''$ .

*Justification*

The viewport is needed to define the analysis area on the sample after the solution has been loaded to the loadlock, before the solution base and sample are transferred to the analysis chamber. The viewport should be at least as large as the viewport of the commercial TF VTM, which is  $1'' \times 1''$ . This is also larger than the largest sample holder's length and width.

*Measurement*

Fulfillment of this requirement can be tested with calipers.

3.2.4. Manufacturability with Harvard ALL equipment and McMaster-Carr tools

*Quantitative value*

It needs to be possible to manufacture the solution using CNC-mills, drills, and a water jet. Additionally, feature sizes and depths must be such that corresponding end mills and drill bits are easily available from McMaster-Carr or a similar vendor.

*Justification*

The solution needs to be manufacturable with the equipment at Harvard ALL. Machines available include a CNC-mill, drills, and a water jet. Additionally, any special tools needed to

manufacture the solution must be purchased easily and be delivered in a timely fashion, for example from McMaster-Carr.

*Measurements*

If a solution fulfilling the oxidation requirement 3.2.1. can be manufactured, this requirement is fulfilled.

### 3.2.5. Summary of technical specifications

	<b>Overarching requirement</b>	<b>Description</b>
<b>3.2.1</b>	No detectable sample contamination during transfer between glove box and XPS	< 1% W bound to O relative to W bound to Te as measured by the K-Alpha+ and Nexsa XPS at Harvard CNS
<b>3.2.2.1</b>	Secure sample loading and unloading in glove box	Overall diameter < 12" to enable loading to glove box
<b>3.2.2.2</b>		< 14 Nm torque required to vent solution
<b>3.2.2.3</b>		Accommodate sample heights >7 mm, ensuring top of sample is at a height of 27 - 35 mm above the bottom of the solution base
<b>3.2.2.4</b>		Accommodate sample widths and lengths of at least 19 mm x 21 mm
<b>3.2.2.5</b>		Sample plate with clamping holes of diameter 0.063" - 0.064"
<b>3.2.2.6</b>		Connection for evacuation line with .21" diameter
<b>3.2.3.1</b>	Compatibility with K-Alpha+ and Nexsa XPS systems at Harvard CNS	Solution base dimensions: 66 mm x 60 mm Solution cover dimensions: Width and length within Ø94 mm.
<b>3.2.3.2</b>		Compatibility with XPS transport platform (see Figure 10 for detailed specification set)
<b>3.2.3.3</b>		Ensure resealing of the 2-way VTM after XPS analysis to achieve requirement 3.2.1
<b>3.2.3.4</b>		Loadlock can be pumped down sufficiently to transfer the base to the analysis chamber Analysis chamber can be evacuated to 8e-8 mbar
<b>3.2.3.5</b>		Resistance between sample mounting plate and bottom of solution base <1Ω
<b>3.2.3.6</b>		Viewport with dimensions > 1" x 1"

<b>3.2.4</b>	Manufacturability on ALL facilities	Manufacturable with CNC-mills, lathes, water jet; all required tools available from McMaster-Carr
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Table 2. Summary of technical specifications.

## 4. Design approach

In the following I describe the final design fulfilling the requirements above. Throughout the project, Fusion360 and Solidworks CAD software was used to design, analyze and simulate the design before manufacturing components.

### 4.1. System diagram

Shown in Figure 11 is the system diagram for the final 2-way VTM. The 2-way VTM consists of a cover and a base. Within the cover, a valve enables evacuation of the 2-way VTM in the glove box. The cover seals with the base using an O-ring in the cover. When the sealed 2-way VTM is loaded to the XPS loadlock, the cover can be removed using a cover separation tool, which uses magnets to pull up on a steel plate attached to the cover. When the base is returned to the loadlock after an XPS analysis, the 2-way VTM reseals, ensured by a spring system on the cover compressing the O-ring between cover and base in the XPS loadlock.

The base allows for sample clamping using the standard Thermo Fisher clamps, compatible with all Hoffman lab sample holders. To ensure the 2-way VTM can be vented, a screw is used to push apart the cover and base.

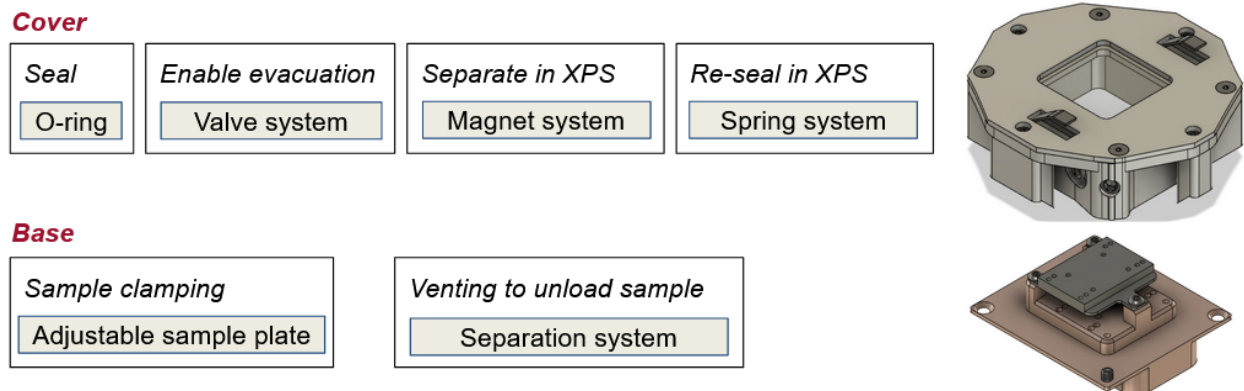


Figure 11. The 2-way VTM consists of a cover and a base. The cover hosts an O-ring to seal base and cover, a valve to enable evacuation, a magnet system to separate base and cover in the XPS loadlock, and a spring system to re-seal base and cover. The base enables sample clamping of all Hoffman lab sample holders. A vent mechanism in the base is used to separate the sealed cover and base.

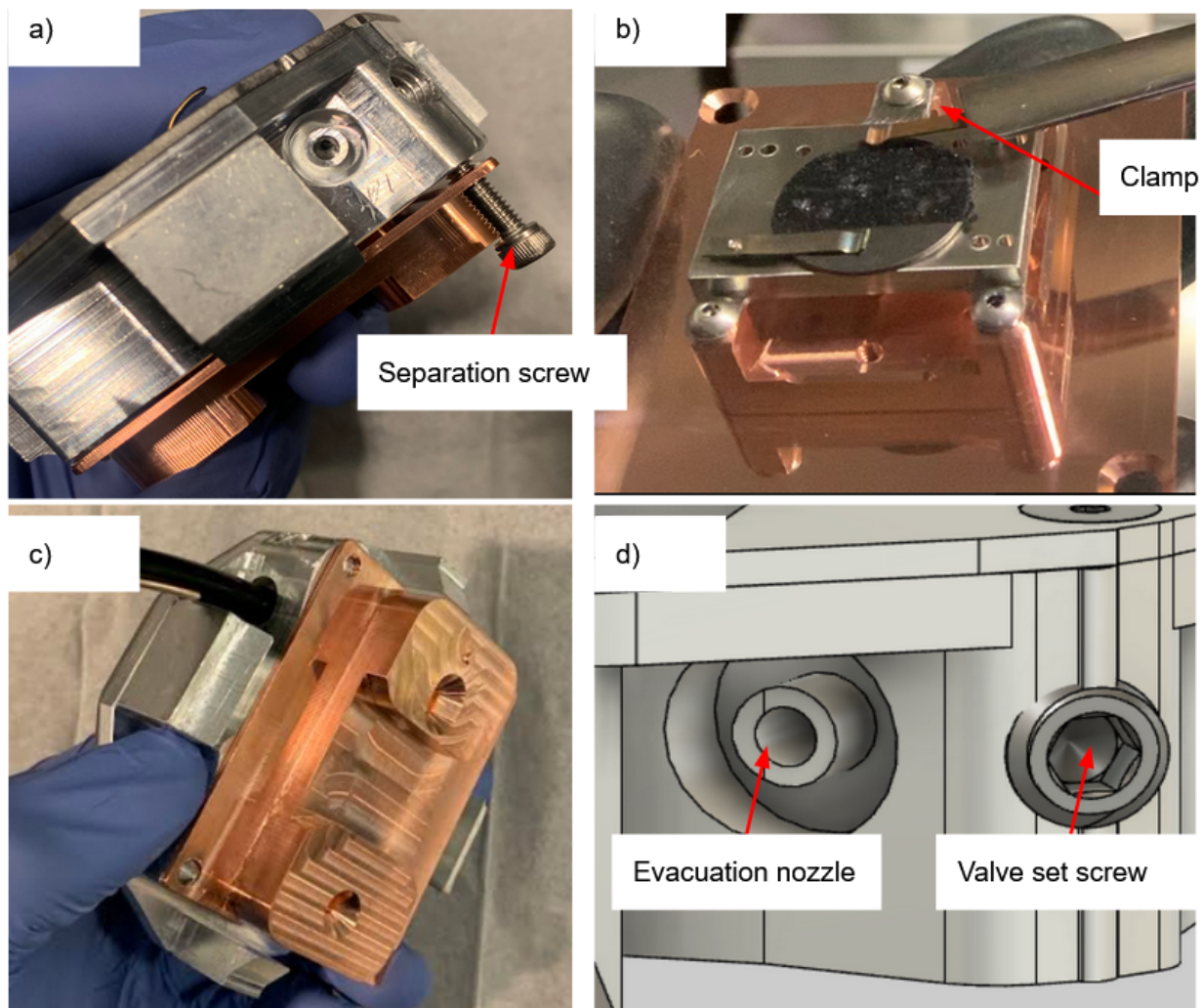
### 4.2. Usage and functional workflow

Before describing the design components in detail, I will outline how the 2-way VTM is used.

## 4.2.1. Workflow for two-way sample transfer

### 4.2.1.1. Sample loading

Initially, the 2-way VTM is moved into a glove box filled with an inert gas like nitrogen or argon. The sample or sample holder is transferred into the glove box, e.g. with a Ferrovac suitcase. Next, the 2-way VTM is vented by tightening the separation screw, as shown in Figure 12a.



*Figure 12. a) The 2-way VTM is vented by inserting and tightening a screw into a bore on one of the corners of the base. b) Samples are clamped using the standard Thermo Fisher sample clamps described in Figure 3. c) If using a roughing pump to evacuate the 2-way VTM, an evacuation tube is connected to the valve nozzle (shown in d)), and the valve set screw is loosened. The 2-way VTM can be evacuated. Then the set screw is closed to seal the 2-way VTM.*

The sample or sample holder is placed on the sample plate, and clamped in place with one of Thermo Fisher's standard clamps, as shown in Figure 12b. In the glovebox, this is easiest with a spatula to accurately place the clamps. When placing on the cover, check that the sample and the sample clamps do not collide with the viewport glass or the inside of the cover.

#### 4.2.1.2. Evacuation

The 2-way VTM can be evacuated through a roughing vacuum pump or in the glove box antechamber. Using the glove box antechamber is more convenient but I have not tested which method is preferable to reduce oxidation. Evacuation in the antechamber is sufficient to prevent oxidation of  $WTe_2$  above the measurement accuracy of the XPS, as explained in detail in section 6. After evacuation check again that the sample and the sample clamps do not collide with the viewport glass or the inside of the cover. As the O-ring compresses, the gap between the sample clamps and the cover will decrease.

##### *Evacuation in antechamber*

Fully tighten the valve set screw, shown in Figure 12d. Place the cover on the base. There is only one configuration that fits. The corner of the cover with the valve system needs to be aligned with the bore for the separation set screw as visible in Figure 12a. Place the 2-way VTM in the antechamber of the glove box, preferably on a tray or some other flat surface. Evacuate the antechamber. From the inside of the glovebox, remove the 2-way VTM. Ensure it is sealed, by checking that base and cover do not separate when lifting up the 2-way VTM cover. Then, the 2-way VTM can be transferred out, without evacuating the glovebox. If the evacuation was not successful, refer to section 4.5.1 regarding possible failure modes.

##### *Evacuation with roughing pump*

Alternatively, evacuate the 2-way VTM using a roughing pump as shown in 12c. Connect a 4.5mm inner diameter plastic tube to the nozzle indicated in Figure 12d. Use an allen key to loosen the set screw such that it is at least 5mm out of the bore. After about 30 seconds of pumping, tighten the set screw completely to seal the 2-way VTM. Wait for 1 minute and check that cover and base do not separate when the cover is lifted. If the evacuation was not successful, refer to section 4.5.1 regarding possible failure modes. Move the sealed 2-way VTM out of the glove box. Do not evacuate the antechamber while transferring the 2-way VTM out.

#### 4.2.1.3. XPS analysis

Transfer the 2-way VTM to the XPS within 20 min. Vent the XPS loadlock. Place the 2-way VTM in the loadlock, such that the valve is on the right-hand side of the loadlock, as shown in Figure 13a. Alternatively, check that the pin on the bottom of the base is on the right-hand side. Evacuate the loadlock.

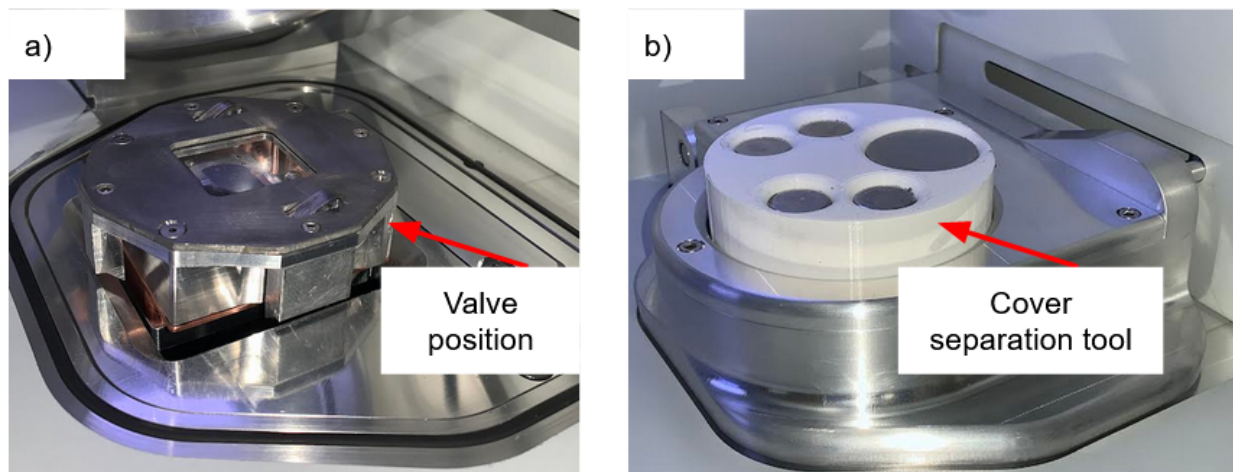


Figure 13. a) Place the 2-way VTM in the XPS loadlock in the orientation shown. b) Place the cover separation tool on the closed loadlock cover once the loadlock reaches  $1.0e-4$  mbar.

Evacuate the loadlock. When the load lock pressure reaches approximately  $1.0 \times 10^{-4}$  mbar, place the removal tool on top of the load lock cover in the orientation indicated in Figure 13b. This pressure is below the pressure that the 2-way VTM will typically hold, which is estimated to be in the 1-10 mbar range. At this point the pressure inside the 2-way VTM is expected to be higher than in the loadlock. The cover separation tool needs to be left on the loadlock cover for at least 10 min to ensure the cover lifts off from the base completely. This ensures that the O-ring between cover and base does not cause the base to stick to the cover, when it is supposed to be transferred to the analysis chamber. It is also possible to visually confirm the separation of cover and base. Closely watch the 2-way VTM cover in the loadlock, as the separation tool is placed on the loadlock. If the cover moves up, but the base remains stationary as the magnet approaches the cover, the O-ring no longer sticks, and the base can be transferred. The separation tool needs to be on the loadlock cover during transfer. After transfer, remove the separation tool from the loadlock. Conduct your analysis.

After the completed analysis, transfer the base back to the load lock. The base is moved upwards and slots into the cover. The base compresses the leaf springs on the cover as it is being moved up into the loadlock. This ensures that the O-ring between cover and base is compressed and creates a seal. When the loadlock is vented, the vacuum inside the 2-way VTM is preserved. The 2-way VTM stays sealed due to the pressure difference when the loadlock cover opens.

Now the 2-way VTM can be transferred back to the glove box, opened, and the samples can be used for further analysis.

### 4.3. Solution design

The solution consists of three components: a cover, a base, and the cover separation tool. I decided against purchasing a TF VTM and adapting it, because it is not compatible with all Hoffman lab sample holders. Additionally, this would have made adaptations to ensure two-way transfer more difficult and potentially less reliable. Thermo Fisher's VTM also exceeds the budget set by ES100 and the Hoffman group, it costs about 9,000\$ [15].

#### 4.3.1. Design of solution cover

The solution cover shown in Figure 14 consists of 6061 aluminum, with six key features and subassemblies integrated, discussed in detail below. Aluminum 6061 is listed as an ultra-high vacuum compatible material [16].

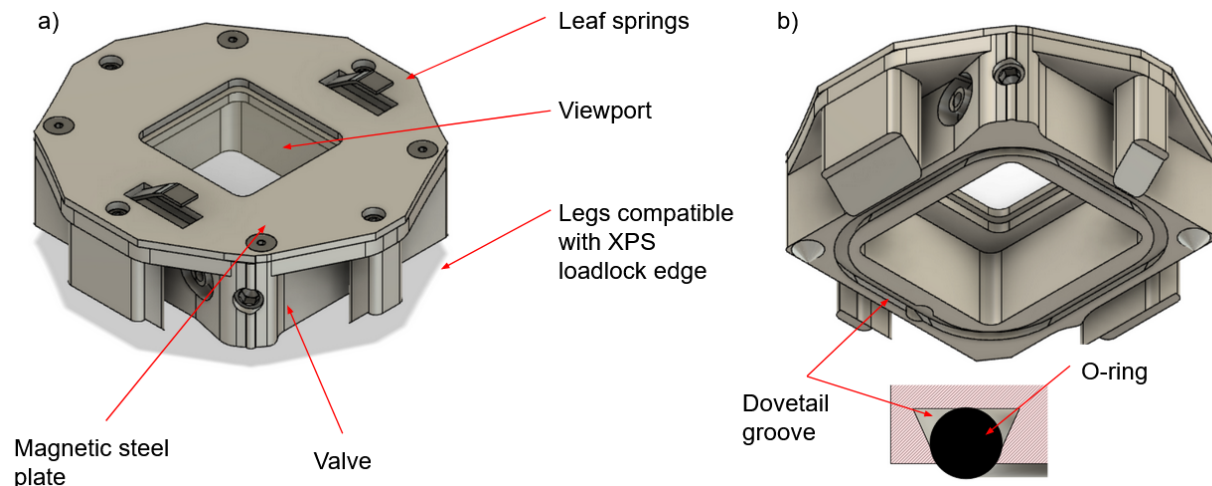


Figure 14. a) Top view of solution cover with main features indicated. Further explanation of each follows below. b) The O-ring sealing cover and base is mounted in a dovetail groove to prevent it from falling out, whenever the cover is lifted off the base.

#### 4.3.1.1. O-ring seal in solution cover

Base and cover are sealed with an O-ring. The O-ring is mounted in a dovetail groove, to ensure it does not fall out when cover and base are separated as shown in Figure 14b. The O-ring has a cross-sectional diameter of 0.139". Even though scratches or dirt should not be present on the O-ring sealing surface on the base, O-rings with a larger cross-sectional diameter are preferable, because they can cope with surface irregularities of greater magnitude [17]. However, the bending radius of any O-ring in a non-circular groove should not be lower than 3x the O-ring cross-sectional diameter [18]. The 0.139" O-ring was found to be the largest diameter that could be accommodated easily taking design constraints on the dovetail groove curvature into account. The groove dimensions were designed in compliance with the Parker O-ring handbook. McMaster sells a dovetail end mill specifically designed for O-rings of this diameter [19] [20]. The O-ring is made from Viton™, a typical material used in vacuum applications, e.g. in KF flanges [21]. It has size number 228, with an inner diameter of 2.234".

#### 4.3.1.2. Cover separation tool

To ensure cover and base separate in the loadlock, an external separation tool is used which does not enter the loadlock. The base is transferred from the loadlock to the analysis chamber with a transport platform, as shown in Figure 15. It is important that the base and cover separate in the loadlock before the transport platform is lowered, because otherwise the O-ring in the cover will stick to the base, and prevent the base from moving down with the transport platform. In some cases, the base will drop into the XPS loadlock shortly after.

I was not able to determine the root cause of O-ring stickiness. The TF VTM's base and cover separate before the transport platform is lowered. As shown in Figure 5, there is a gap between base and transport platform in the loadlock after the TF VTM is loaded into the XPS loadlock. After the pressure inside the TF VTM and the loadlock are equal, the force from gravity pulling down on the TF VTM base will separate the base from the O-ring in the cover. After the base has dropped onto the transport platform, a gap exists between cover and base. This gap prevents resealing of the TF VTM.

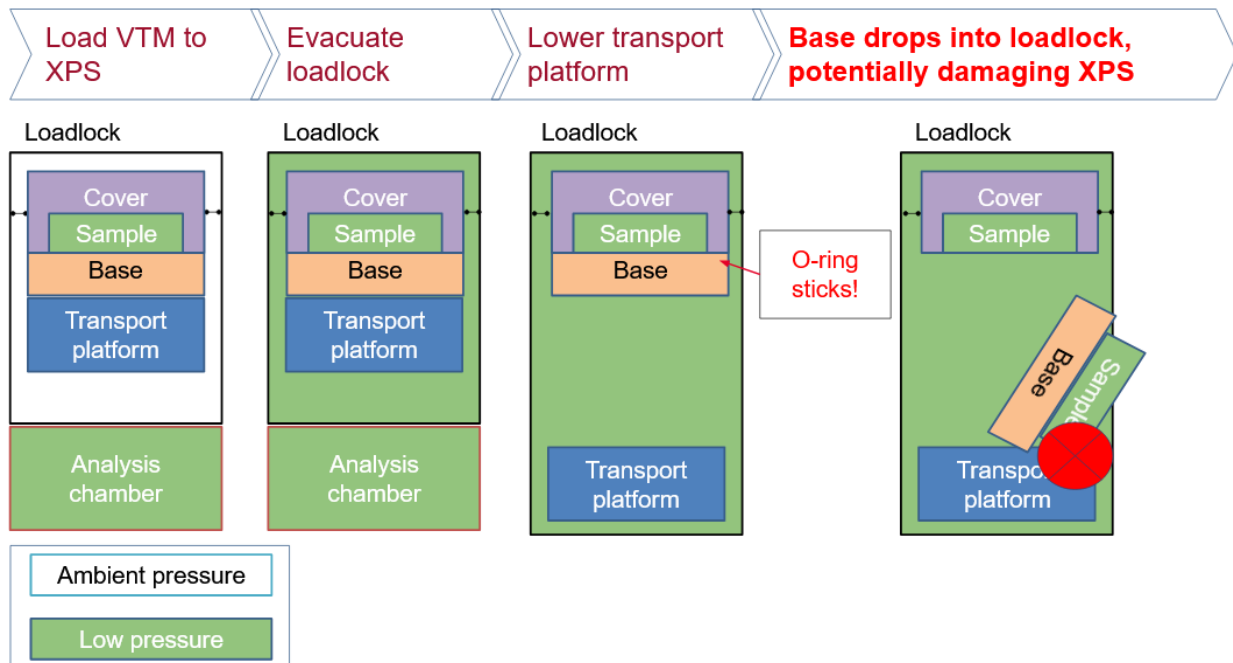
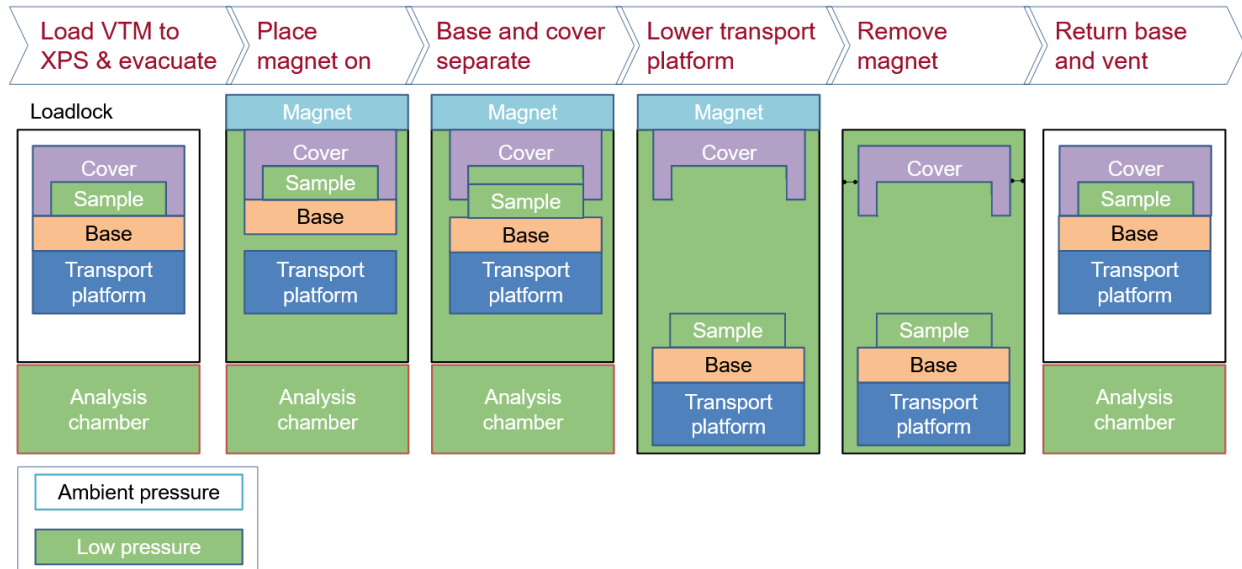


Figure 15. The 2-way VTM base is transferred to the analysis chamber with a transport platform. The O-ring between base and cover can cause the base to stick to the cover, as the transport platform is lowered. This can cause the base to drop into the loadlock, and damage the XPS.

To ensure resealing of the 2-way VTM there must not be a gap between cover and base after the base is returned following an analysis, as explained in Figure 5. However, a gap between cover and base prior to an analysis, before the transport platform is lowered, will prevent the base from sticking to the cover unsafely.

To create a gap between cover and base prior to the analysis, I use a cover separation tool with magnets to pull up the 2-way VTM. The adapted transfer process is shown in Figure 16. When the 2-way VTM is loaded to the XPS loadlock, the base sits directly on the transport platform, there is no gap like for the TF VTM. The loadlock is evacuated, and the user places the cover separation tool with magnets onto the loadlock from the outside. This pulls up the entire 2-way VTM. Because the base is not magnetic, it drops onto the transport platform, once gravity has overcome the stickiness of the O-ring. This step is considered in more detail in Figure 17. The transport platform can be lowered safely into the analysis chamber. The user removes the cover separation tool from the loadlock. The 2-way VTM cover remains in the loadlock. This is ensured by legs on the cover that sit on the loadlock edge, explained in detail in section 4.3.1.5. When the base is returned to the loadlock after an analysis, the O-ring in the cover will seal onto the base, such that the 2-way VTM reseals.



*Figure 16. By using magnets to pull up the 2-way VTM cover, it is possible to separate base and cover before the transport platform is lowered. Initially the base will stick to the cover, but it will then drop down onto the transport platform. It will safely move down with the transport platform when the platform is lowered. Removing the magnet and returning the base to the XPS loadlock will eliminate the gap between cover and base, ensuring the 2-way VTM reseals.*

The separation tool shown schematically in Figure 17 consists of magnets, which pull up on the 2-way VTM cover in the XPS loadlock against the spring system on the cover. A 3D-printed fixture holds five Neodymium magnets of thickness 1", four of these with  $\frac{3}{4}$ " diameter, and one with 1.5" diameter [22] [23]. The magnets have a rated total pull force of 330 lbs, if attached directly to a thick steel plate. In my application they pull through the loadlock glass on a magnetic steel plate on the 2-way VTM cover. The steel plate has a thickness of 0.075" and consists of low carbon steel, likely grade 1008-1010 low carbon steel. While it was not possible to confirm the UHV compatibility of the steel, I experienced no problems evacuating the XPS loadlock to pressures below 1e-6 mbar. The steel plate does not enter the analysis chamber, and thus does not enter UHV pressures. It is mounted to the cover using three 4-40 vented flat head screws and one non-vented flat head screw, made from UHV-compatible 18-8 stainless steel [24][25][26]. The non-vented flat head screw sits directly above the valve system, with the bore ending in the valve system. This ensures there is no air trapped under the screw, so no vented screw is needed. Once the loadlock is pumped down the magnets pull up the cover separating the cover from the base and overcoming any stickiness in the O-ring, as shown in Figure 16.

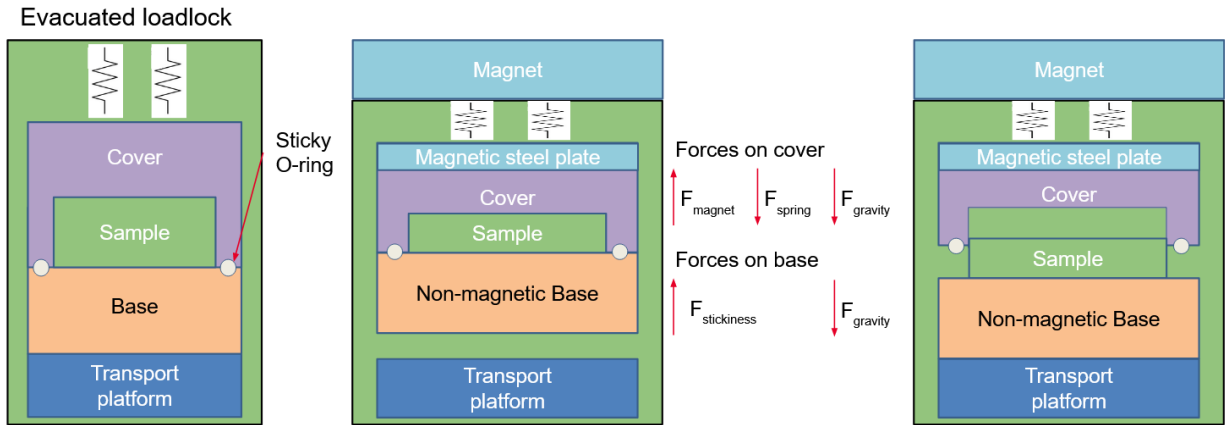


Figure 17. Magnet placed on the loadlock cover pulls up the 2-way VTM in the loadlock, overcoming gravity and the spring forces. The base will initially stick to the cover and move up. As gravity pulls down on the base, the force from the sticky O-ring diminishes. The base drops onto the transport platform, once gravity is stronger than the force from stickiness. When the transport platform is subsequently lowered, the base moves down safely.

#### 4.3.1.3. Valve system

I found three possible design implementations to ensure evacuation of the 2-way VTM. First, a check valve allows air flow, when the pressure outside the 2-way VTM is lower than the inside and blocks air flow with an opposite pressure gradient. However, this only allows evacuation to a certain threshold pressure difference, when the check valve closes. Secondly, a gate valve can be used, which is manually opened to allow evacuation and closed to seal. Third, evacuation in the glove box antechamber is possible without a valve. Each option will be explained in more detail below.

##### *Thermo Fisher check valve*

The commercial Thermo Fisher VTM uses an integrated check valve, which allows air to be pumped out of the TF VTM, but blocks air from flowing back in. A cross-sectional view of this valve type is shown in Figure 18. It consists of a ball which is pushed on an O-ring by a spring. Using a set screw, the spring force can be adjusted. The force acting on the O-ring  $F_{O-ring}$  consists of the press-on force resulting from the spring, and the pressure difference between the inside and outside of the TF VTM, such that  $F_{O-ring} = F_{spring} + F_{pressure}$ . To allow for air flow out of a check valve, a certain minimum pressure difference is required, called cracking pressure [27]. The cracking pressure depends on the spring force that needs to be overcome to move the ball off the O-ring, which depends on the pre-tensioning of the spring using the set screw. To minimize the cracking pressure, and still ensure sufficient force on the O-ring to seal the valve, the contribution to  $F_{O-ring}$  by the pressure difference should be increased. This is achieved by increasing the area enclosed by the O-ring. In my final design I used a 4.5 mm inner diameter O-ring in the check valve. Incorporation of a larger O-ring was not possible due to the design's wall thickness.

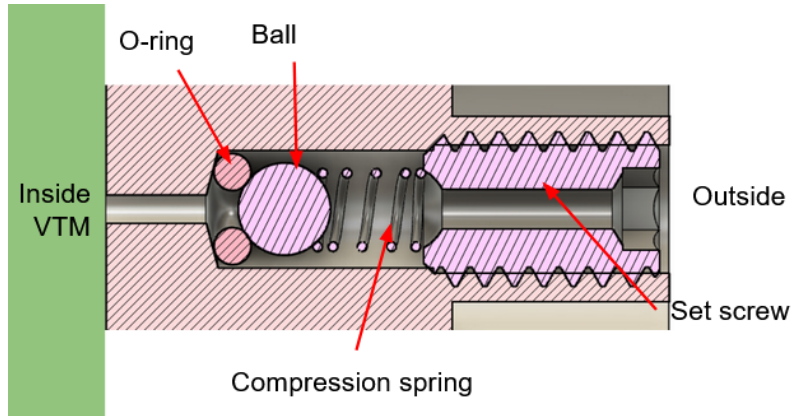


Figure 18. The Thermo Fisher check valve consists of a ball that is pressed onto an O-ring by a compression spring to allow air flow, when the outside pressure is lower than the inside pressure, and if not, creates a seal.

### 2-way VTM valve

Relative to the check valve in the Thermo Fisher VTM, I increased the inner diameter of the sealing O-ring, and I added a separate evacuation channel to increase flexibility of the valve. The valve consists of a steel ball pushed onto an O-ring by a spring, pre-tensioned by a set screw, as shown in Figure 19. I used a 4.5 mm inner diameter, 1 mm cross-sectional diameter O-ring, and a 1/4" diameter ball made from 52100 steel [28]. I was not able to integrate an O-ring with a larger inner diameter due to the limited wall thickness of the 2-way VTM. No sources could be found regarding UHV-compatibility of 52100 steel. However, I experienced no issues evacuating the XPS loadlock with the cover and steel ball inside. The compression spring has 1/4" length and .18" outer diameter made of UHV-compatible 302 stainless steel, and a 5/16"-18 non-vented set screw of 3/8" length made of UHV-compatible 18-8 stainless steel [29] [16] [30]. The 2-way VTM is not evacuated through the set screw, but through an evacuation channel on the side. An evacuation tube can be pushed over a nozzle around the evacuation tube to enable evacuation as required in 3.2.2.6.

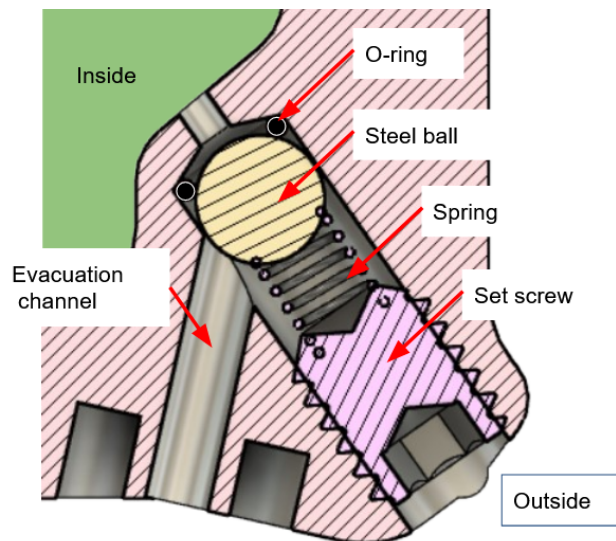
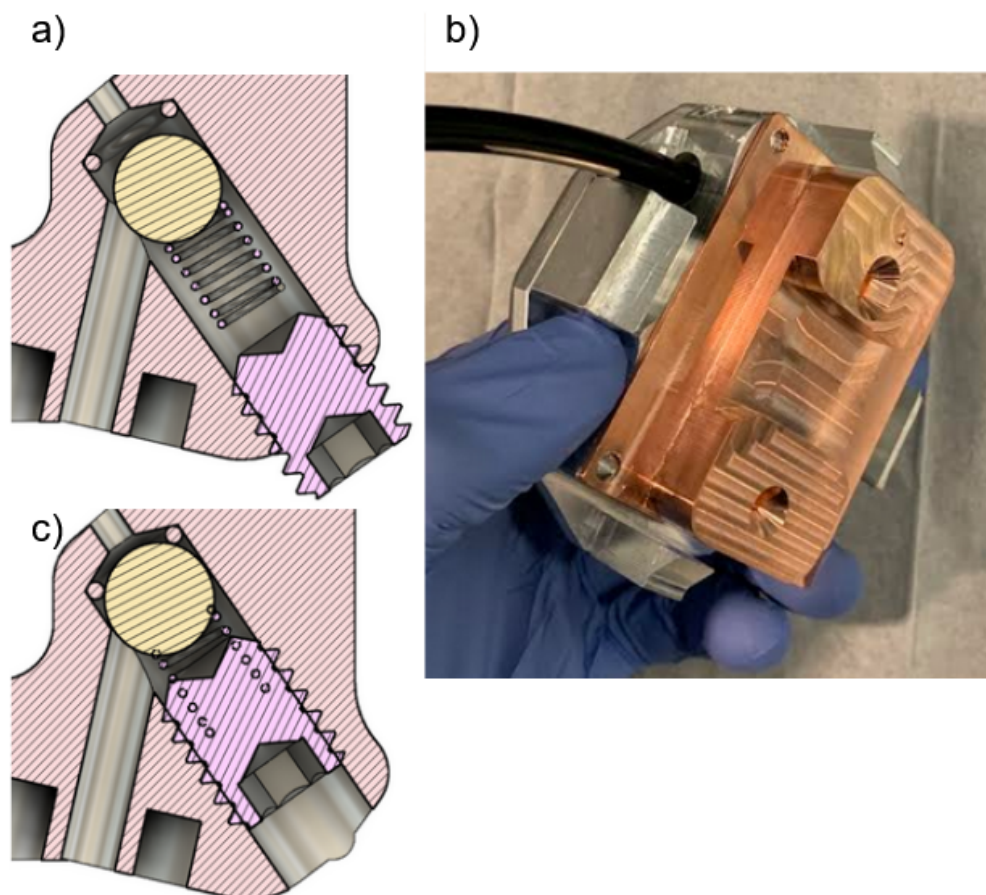


Figure 19. The 2way VTM valve system consists of a steel ball sealing onto an O-ring with an additional evacuation channel to allow a gate-valve operation mode. The ball is compressed onto the O-ring with a spring, which is pre-tensioned by a set screw. Evacuation occurs through a separate evacuation channel, not through the set screw as in the Thermo Fisher design.

To use the valve as a check valve, the set screw can be set to a position where it enables sealing and evacuation. During testing, this position was difficult to find, so I do not recommend use as a check valve.

The valve can be used as a gate valve. If a vacuum pump is connected to the evacuation channel using a rubber tube, and the set screw is loosened, the ball can come completely off the O-ring, as shown in Figure 20a. After evacuation through the tube as shown in Figure 20b, the set screw can be tightened, as shown in Figure 20c. This creates a higher compression force on the O-ring than in the case of a check valve, ensuring a tighter seal. This setup requires a vacuum pump with an evacuation line in the glove box.



*Figure 20. The valve can be used as a gate valve. a) To evacuate the 2-way VTM the set screw is loosened, so that the steel ball can separate from the O-ring. b) Then, the 2-way VTM can be evacuated using a vacuum pump and a tube connected to the evacuation channel. c) Once the 2-way VTM is evacuated, the set screw can be tightened to seal the 2-way VTM.*

When the set screw is fully tightened, the 2-way VTM can still be evacuated in the glove box antechamber. When the antechamber is evacuated, the remaining air inside the 2-way VTM will push up the cover and escape until the pressure difference is too low to push up the cover, as shown in Figure 21 below. When the antechamber is subsequently vented, the 2-way VTM will seal, maintaining vacuum inside. This is the most convenient method of evacuating the 2-way VTM, because the valve does not have to be opened or closed. I used this evacuation procedure during my oxidation tests. No additional compression force is needed for the O-ring to seal, as the leaf springs would provide in the XPS loadlock. My hypothesis is that this is caused by a significant difference in venting speeds of the antechamber and the loadlock. The

antechamber of the glove box I used in testing could be vented in less than 10 seconds, while the XPS loadlock will take at least 30 seconds. Venting speeds cannot be reduced on the XPS system. When the glove box antechamber is vented more slowly, to take more than 20 seconds, the 2-way VTM will not always seal.

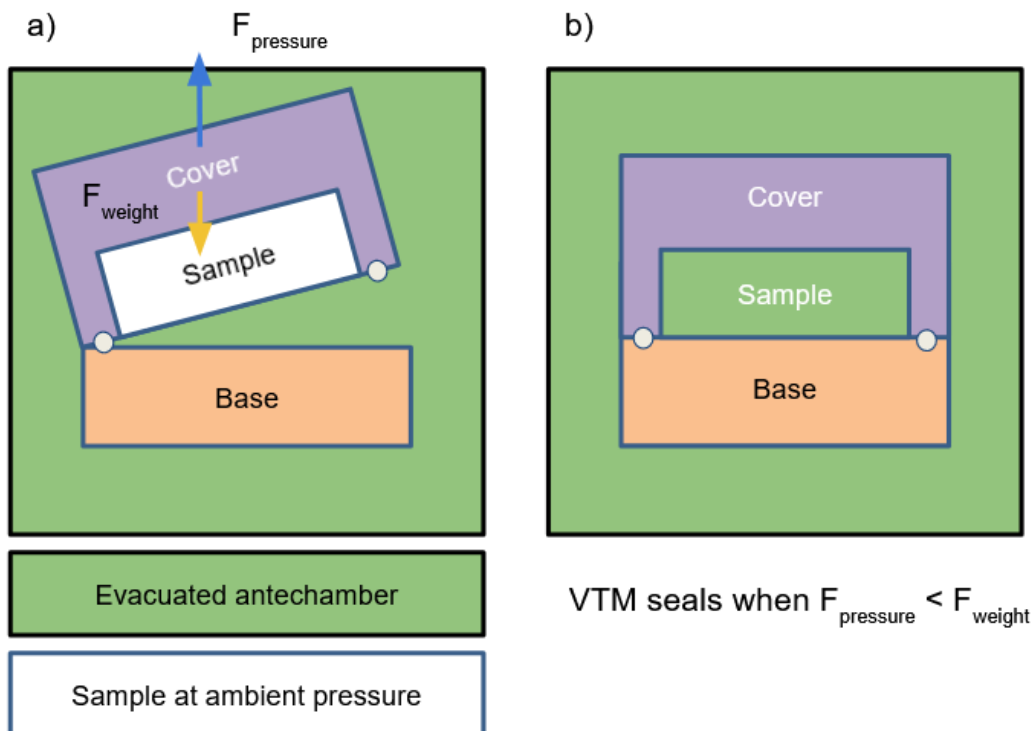
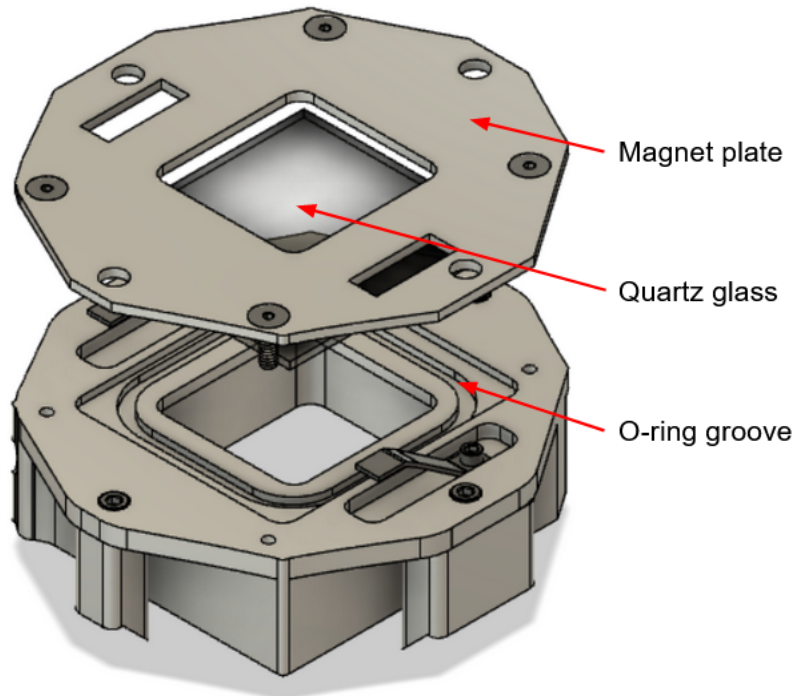


Figure 21. When the valve is closed fully, it is still possible to evacuate the 2-way VTM. When placed in the glove box antechamber, and the antechamber is evacuated, the pressure inside the 2-way VTM will push up the cover. Once the pressure difference can no longer lift the cover, and the antechamber is vented, the 2-way VTM seals.

#### 4.3.1.4. Viewport

The viewport consists of a glass plate, which is pressed onto an O-ring by a cover plate to ensure a seal, as shown in Figure 22. The viewport has a width of 1.05" and length of 1", fulfilling requirement 3.2.3.6. Glass is chosen over acrylic, which like other polymers is more permeable to gas molecules, by up to 3 orders of magnitude [19, p. 301-309]. The glass plate consists of fused silica glass (quartz glass), which is typically used in vacuum applications [19, p. 302]. A 1/16" thickness was used. The glass plate is pressed onto an O-ring by the steel plate of the cover separation system. An O-ring with a cross-sectional diameter of .103" was used. The O-ring groove depth and width was designed in accordance with the Parker O-ring handbook [19].



*Figure 22. The viewport consists of a stainless steel plate, which presses a quartz glass plate onto an O-ring to create a seal.*

#### 4.3.1.5. Cover legs

To ensure the cover remains in the loadlock after the base is lowered, the cover has legs, which sit on the loadlock edge as shown in Figure 23b. The dimensions of the legs were initially based on the dimensions of the TF VTM. To ensure the base and cover are in contact after an XPS analysis to allow for re-sealing, they were shortened. Additionally, after placing initial prototypes in the XPS loadlock, the width indicated in Figure 23a was adapted to ensure the legs aligned well with the loadlock surface and slanted edge of both the K-Alpha and Nexsa XPS at CNS. They consist of 6061 aluminum, which is UHV-compatible. The legs are screwed to the cover plate using 4-40" vented socket head screws from McMaster-Carr made of 18-8 stainless steel, which is UHV-compatible [24] [32].

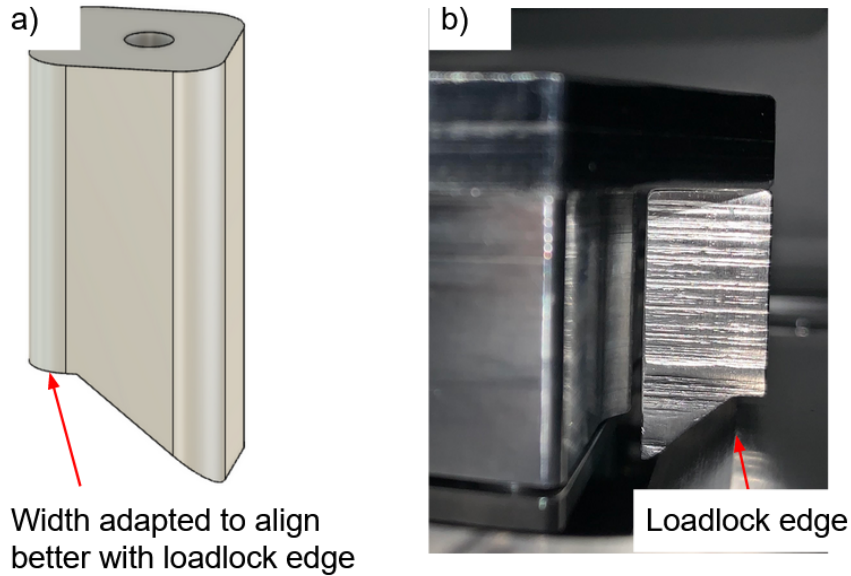


Figure 23. The width of the horizontal part of the legs (indicated in a)) was adapted to align closely with the XPS loadlock edge (indicated in b)).

#### 4.3.1.6. Spring system

The spring system on top of the cover pushes against the closed loadlock cover of the XPS to push the cover onto the base and establish a seal, as the loadlock is vented after an XPS analysis. The final spring design consists of two strips of 1095 spring steel bent to form leaf springs as shown in Figure 24. No sources were found regarding UHV-compatibility of 1095 spring steel, but I experienced no issues evacuating the loadlock below  $1e-6$  mbar, sufficiently low for the XPS software to allow transfer of the base to the analysis chamber. Each strip has a thickness of 0.021". Leaf springs were chosen over coiled springs, because coil springs were found to be more prone to buckling during initial prototyping. The springs are screwed onto the cover using 2-56" vented socket head screws from McMaster Carr, made from 18-8 stainless steel, a common UHV material [33] [24].

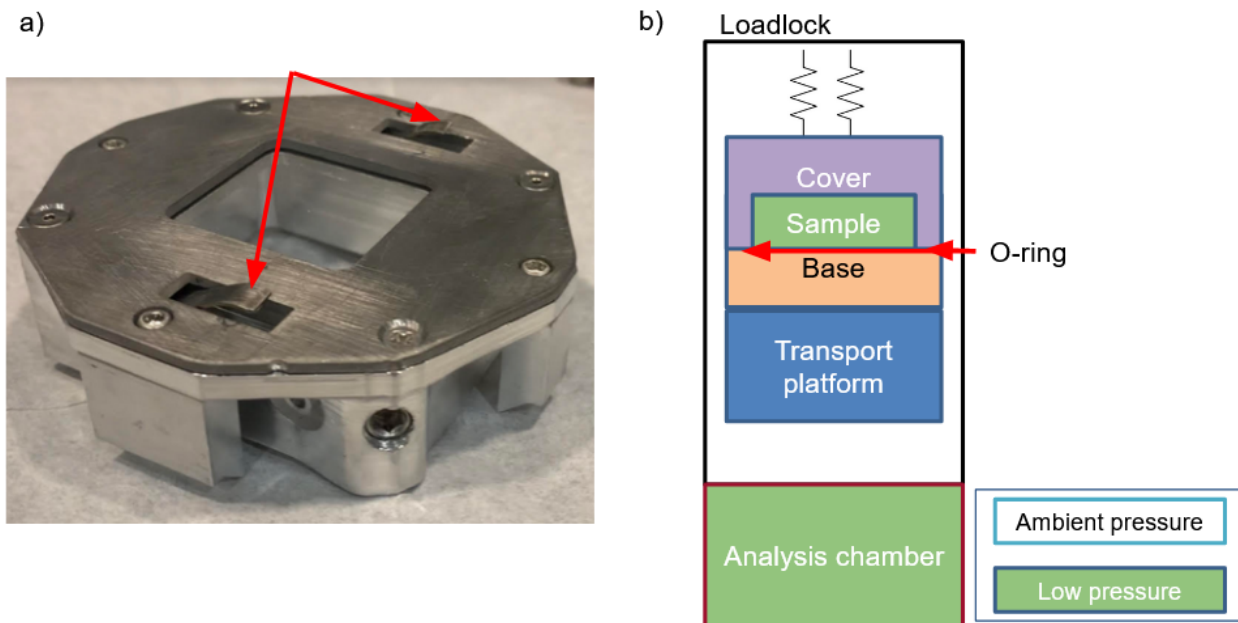


Figure 24. Leaf springs are placed on the cover as shown in a). They press against the XPS loadlock cover as shown in b), pressing the 2-way VTM cover on the base and sealing the 2-way VTM as the XPS loadlock is vented.

#### 4.3.1.7. Centering cones

The cover also has centering cones in two corners on the bottom, as shown in Figure 25. These ensure that the cover is centered on the base. They have a base diameter of 0.27", taper at a 45° angle, and slot into countersunk holes with base diameter 0.28".

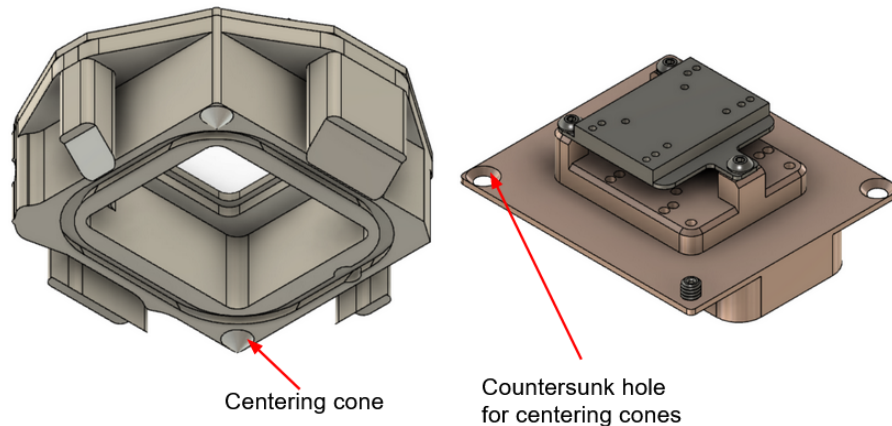
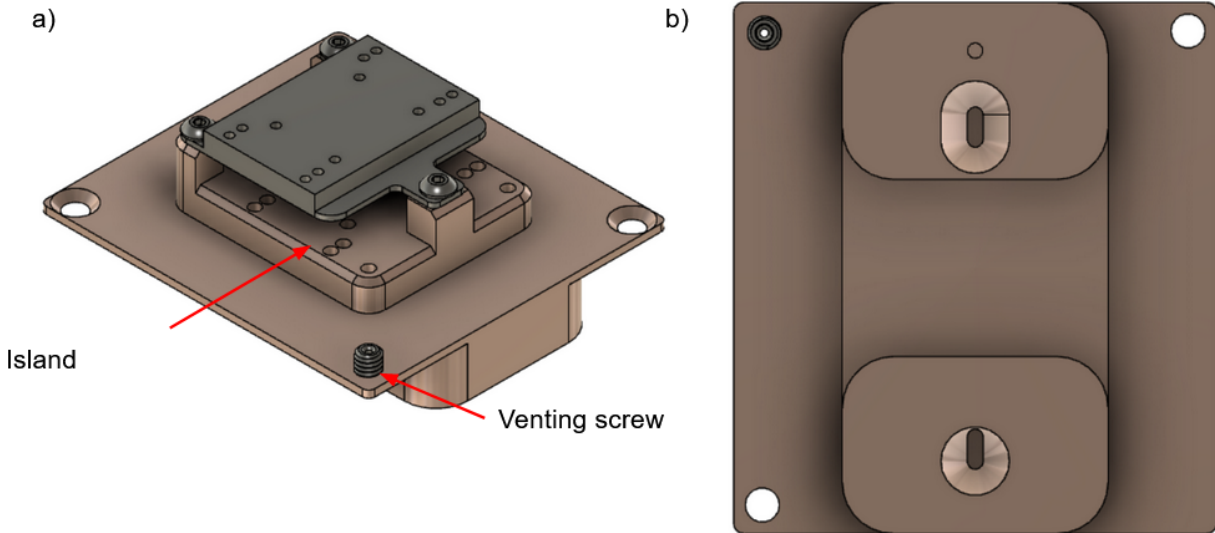


Figure 25. Centering cones help ensure the cover is centered on the base.

#### 4.3.2. Design of solution base

The base shown in Figure 26 consists of a copper platform. I used copper alloy 145, which is easier to machine than regular copper. I could not find specific data regarding UHV-compatibility for this copper alloy, but I experienced no issues evacuating the XPS analysis chamber to the pressure required, which is  $8 \times 10^{-8}$  mbar. The overall dimensions of the base are 66 mm x 60 mm x 25 mm, compliant with requirement 3.2.3.1. The bottom of the base has a groove pattern as required by 3.2.3.2, shown in Figure 26b. On top of the platform, pillars of different heights with threaded holes allow for the attachment of a sample plate. The sample plate can be set to different heights to accommodate sample heights up to 9 mm. Samples can be fixed using the clamps Thermo Fisher manufactures for their sample stages, as described under 3.2.2.5 and as shown in Figure 3. A screw can be used to vent the 2-way VTM by separating base and cover. Additionally, two countersunk holes are included to receive the centering cones on the cover. An island between the pillars increases the volume and weight of the base, which aids separation from the cover O-ring in the XPS loadlock.



*Figure 26. a) The base consists of a copper platform onto which a sample plate can be screwed. A venting screw can separate the cover and base when sealed. An island between the pillars of the base increases the weight of the base to aid separation of cover and base in the XPS loadlock before the transport platform is lowered. b) The bottom of the base has a groove pattern as required in 3.2.3.2.*

#### 4.3.2.1. Standard sample clamping

The sample plate is shown in Figure 26a. It consists of 304 stainless steel, which is UHV-compatible [16]. The sample plate has holes to accommodate the commercial Thermo Fisher sample clamps, with the diameters as required in 3.2.2.5. There are three different height settings to accommodate sample heights up to 9 mm, as shown in Figure 27. Using this plate, each Hoffman lab sample holder can be loaded to the solution, such that the top of the sample is at a height of at most 31 mm, and the sample is at a height between 27 mm and 31 mm, as required in section 3.2.2.3. The inner section of the sample plate has a width of 1.3" x .88" to fulfill requirement 3.2.2.4, the width and length of sample holders to be accommodated. The plate is screwed to the base using 4-40" vented button head screws, made from 18-8 stainless steel, a common UHV material [16].

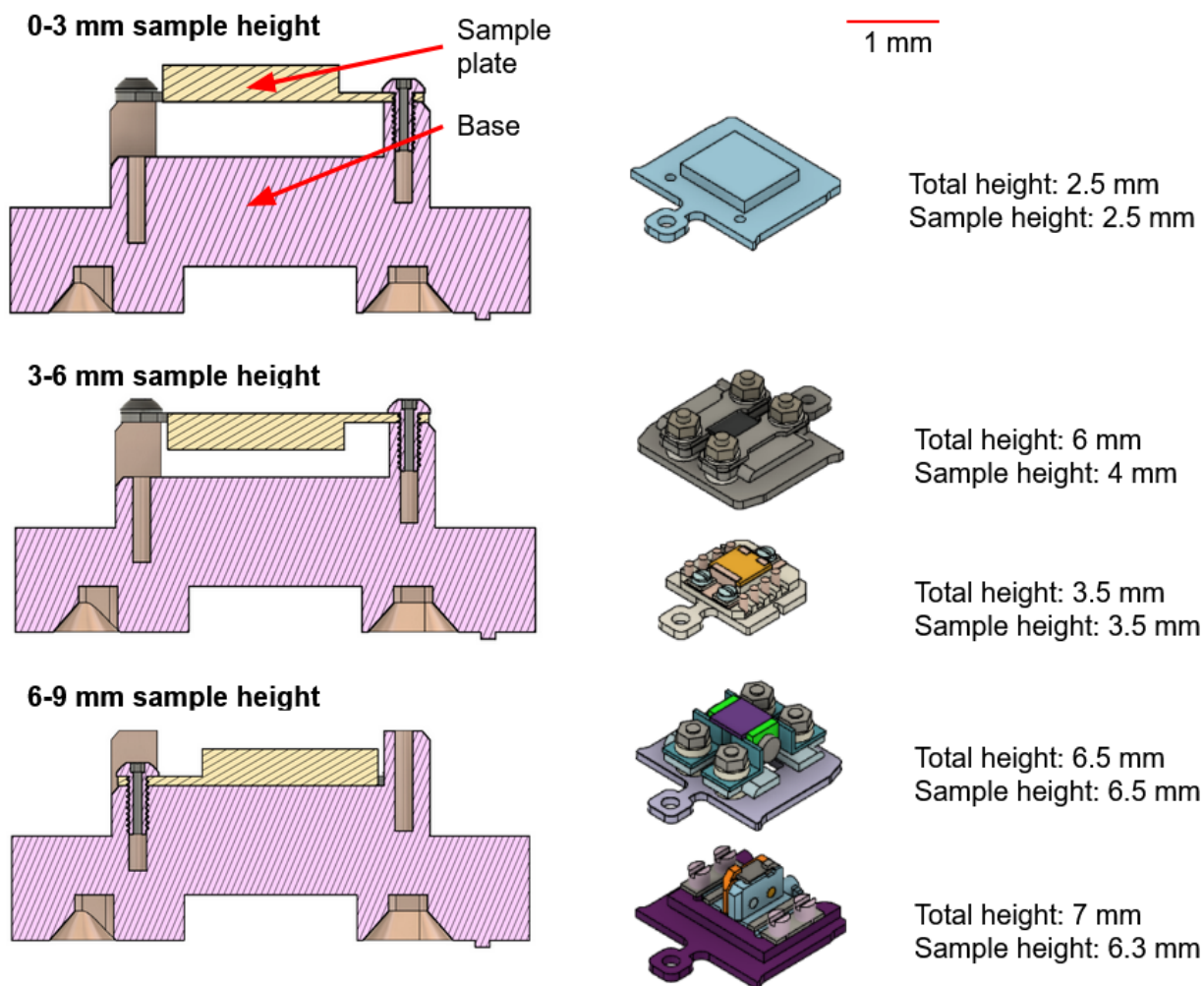
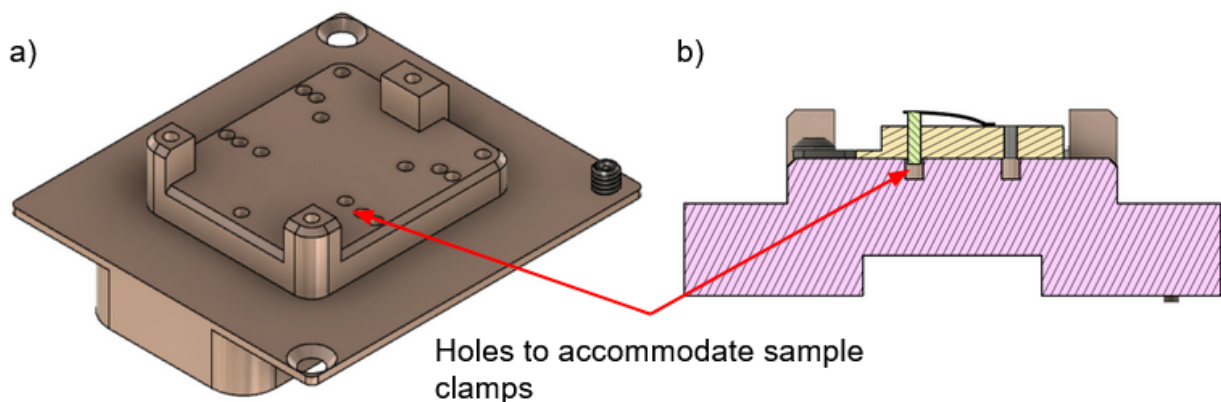


Figure 27. Solution base with standard sample plate. The plate can be set to four different height settings to accommodate samples up to 9 mm. This accommodates all Hoffman lab sample holder heights.

I included holes on the top of the base, to ensure the Thermo Fisher sample clamps can be inserted in the sample plate, even when the sample plate is in the lowest position, as shown in Figure 28.



*Figure 28. a) The island between the base pillars has holes, such that the Thermo Fisher sample clamps can be accommodated. b) The clamps can be inserted, even when the sample plate is set to the lowest setting.*

#### 4.3.2.2. Venting screw

Any 8-32" screw can be used to vent the 2-way VTM, to fulfill requirement 3.2.2.2, separation of base and cover under ambient pressure conditions. The required torque can be estimated from the pressure difference and the area enclosed by the large O-ring in the dovetail groove. The area enclosed by the O-ring is 26.4cm<sup>2</sup>. So at a pressure difference of 1 bar, 264N will be required to separate base and cover. The required torque for this bolt tension force will be  $T = cDF$  where  $c$  is the coefficient of friction of the thread material,  $D$  is the diameter of the bolt and  $F$  is the compression force in the bolt [34]. Assuming a coefficient of friction of dry steel of at most 1 and a bolt diameter of 4mm, the required torque will be about 1.1Nm, which can be unscrewed by hand.

## 4.4. Failure modes: Evacuation and venting

### 4.4.1. Failure modes during evacuation

If evacuation is not possible, this may be caused by four reasons: The sample or sample clamp obstruct the cover; the valve does not open; the valve does not seal; the O-ring between cover and base or in the valve is not clean.

#### 4.4.1.1. Sample or sample clamp obstruct cover

Ensure that the sample clamps and sample do not collide with the viewport glass or the inside of the cover, as shown in Figure 29. In extreme cases this can prevent the O-ring in the cover from touching the base completely, preventing evacuation in the antechamber. Even if evacuation in the antechamber is possible, the sample or sample clamp will create an upwards force on the cover, reducing the compression force on the O-ring. This can prevent resealing in the XPS loadlock. To prevent a collision of the sample, ensure that the sample plate is set to the correct position based on your sample height, as shown in Figure 27. To prevent a collision of the sample clamps, ensure the clamps are pushed down fully, or place the clamp closer to the center of the sample plate where there is more space between clamp and viewport, as shown in Figure 29.



*Figure 29. If the sample or sample clamps collide with the cover as shown in red, this may prevent evacuation and resealing. Either push down the clamp fully, or use a different hole to insert the clamp, as shown in green.*

#### 4.4.1.2. Valve does not open

This applies only to gate valve use. When using the gate valve to evacuate the 2-way VTM, evacuation may not be possible if the spring is caught in the threads for the set screw. In this case, the steel ball may not be able to separate from the O-ring. If this occurs, the set screw should be removed, and the allen key used to remove the set screw can be used to push on the spring. This will remove it from the threads. This can be done in the glove box. If the spring is visibly bent or deformed, it should be replaced. The risk can be avoided by fully tightening the set screw and evacuating the 2-way VTM in the glove box antechamber.

#### 4.4.1.3. Valve does not seal

The valve set screw needs to be fully tightened to seal the 2-way VTM. This applies to gate valve evacuation and to evacuation in the glove box antechamber.

#### 4.4.1.4. Dirty O-ring

Additionally, if the O-ring inside the valve or the O-ring between cover and base is excessively dirty it may prevent sealing. They can be cleaned with a wipe and methanol. Disassembly of the valve needs to be done outside of the glove box, to allow for accurate handling of the small components. If aluminum or copper flakes are visible, all components should be sonicated in acetone and methanol for at least 10 min each.

### 4.4.2. Failure modes during venting

Repeated venting may cause damage to the cover, as scratches in the cover may be caused by the screw end. These scratches will not impede functionality, unless aluminum protrudes from the bottom surface of the cover indicated in Figure 30, which may prevent the O-ring in the cover from touching the base and seal. The protruding aluminum can simply be filed off.

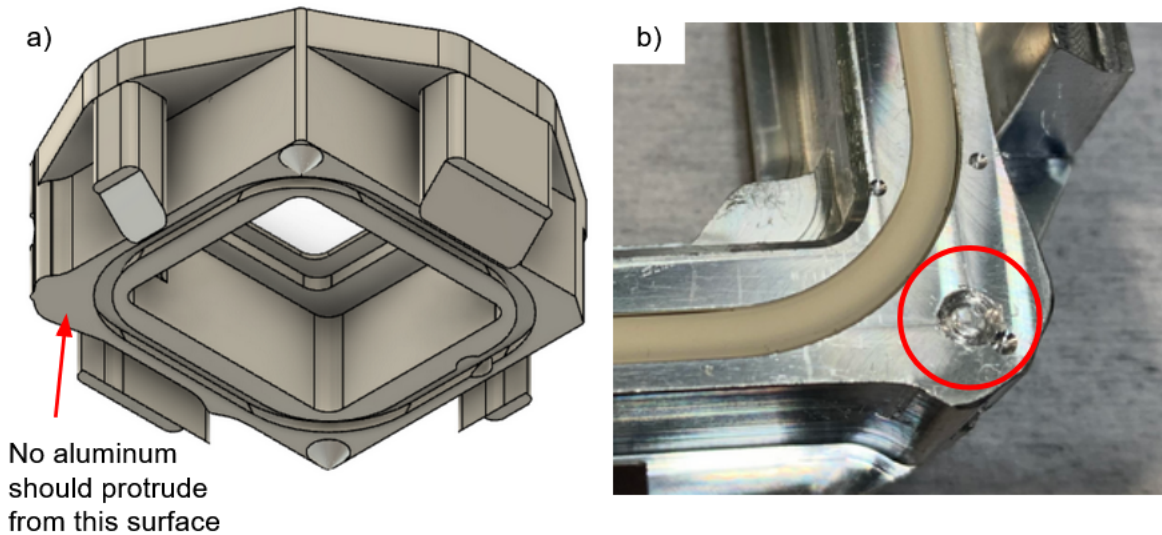


Figure 30. a) If aluminum protrudes from the surface indicated, this may prevent the base from fully touching the O-ring in the cover, preventing a seal. b) Harmless scratches were visible after a total of more than 20 separations.

## 4.5. Failure modes: XPS compatibility

### 4.5.1. Closing of loadlock

The 2-way VTM should not prevent the XPS loadlock from closing fully. If the springs prevent closing of the loadlock, they need to be bent down such that they exert less force on the loadlock cover. The loadlock cover must not be pushed down manually, as this may damage the transport platform.

### 4.5.2. Transfer of base to the analysis chamber

#### 4.5.2.1. O-ring causing base to stick to cover

The base may stick to the O-ring in the cover when the transport platform is lowered, and then fall into the XPS. By using the cover separation tool as described above, this risk can be reduced. The tool should be left on the loadlock cover for at least 10 minutes. In testing, less than 5 minutes were enough to separate base and cover, but no accurate estimate of the required duration to definitively ensure separation was possible. O-Ring stickiness depends on various factors including at least transfer duration, leaf spring force, distance between the steel plate on the cover and the magnets, the weight of the base and sealing surface condition on the base. The user can also visually ensure the cover and base are separated. If the cover separation tool is placed on the loadlock and the user can see the cover move up, while the base stays in place, this indicates the O-ring is no longer sticking. To prevent excessive sticking, the 2-way VTM should be transferred to the XPS within 20 min.

#### 4.5.2.2. Magnet handling

The cover separation tool needs to be handled very carefully to prevent injury or damage due to the strength of the magnets. The table top surface on the XPS has magnetic steel underneath, so the magnet should not be placed anywhere on the XPS other than on the loadlock glass. If the magnet is brought too close to a magnetic surface there is a high risk of pinching your finger.

### 4.5.3. Return of base to loadlock after analysis

When the base is returned to the loadlock after an analysis, it needs to slide back into the VTM cover, as shown in Figure 31a. I expect that if this positioning is incorrect as shown in Figure 30b, damage would occur to the transport platform or the XPS loadlock. In testing, my prototypes the base always slotted back into the cover. To prevent a collision and guide the cover into place as the base is moved up, I used three design features: a 0.1" gap between the pillars on the base and the inside walls of the cover; a tapered top edge of the pillars; button head screws with a tapered head to mount the sample plate, as shown in Figure 32. I tested the reinsertion more than 40 times on the Nexsa and K-Alpha+ each, experiencing no problems related to reinsertion of the base in the cover.

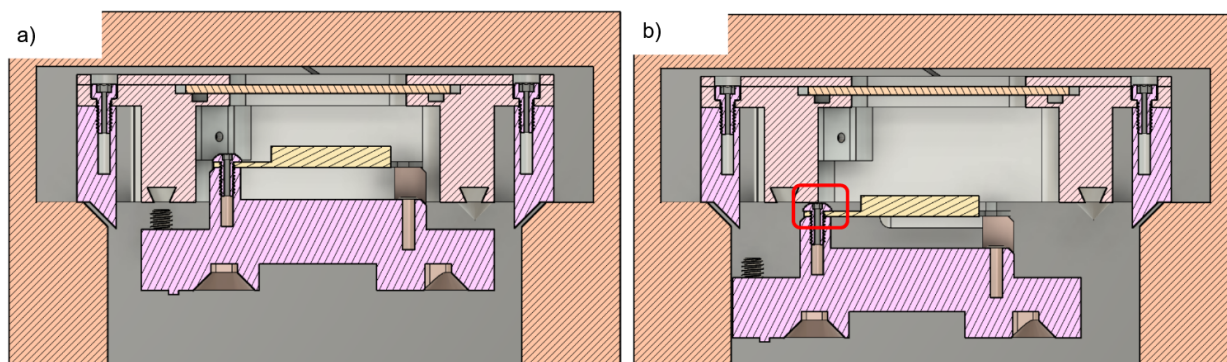
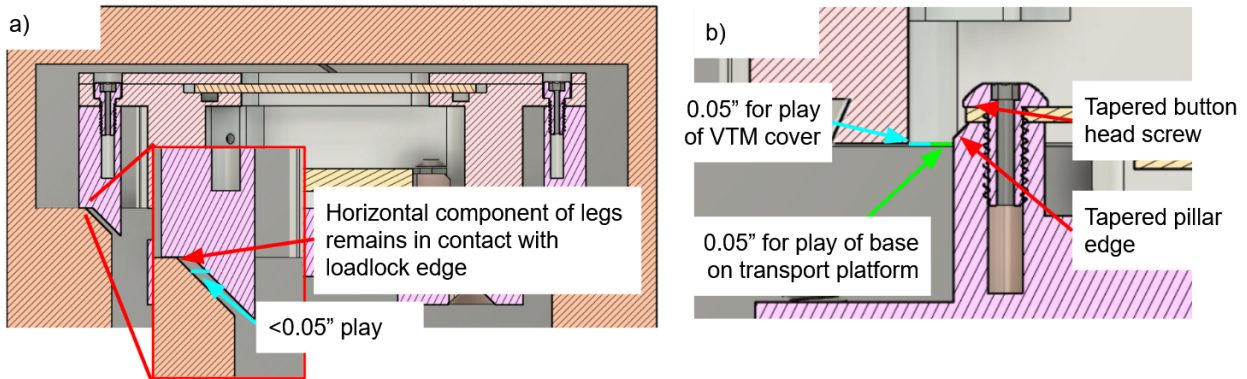


Figure 31. a) When the base is returned to the loadlock after the XPS analysis, it needs to slide into the cover without collisions. b) If the cover is substantially misplaced relative to the base, the pillars on the base may collide with the cover, damaging the XPS.

If the horizontal component of the legs remains in contact with the loadlock edge and does not slide off the loadlock edge as shown in Figure 32a, the lateral play of the cover in the loadlock in any direction for both the K-Alpha+ and Nexsa XPS is less than 0.05". The tapered part of the legs should ensure that the horizontal component of the legs remains in contact with the loadlock edge. When I tried to displace the cover in the loadlock with the magnets in the cover separation tool, the horizontal part of the legs initially slid off the loadlock edge, but the cover fell back into place when the cover separation tool was removed. The base can move laterally on the transport platform. While accurate measurement of this play is difficult, it is estimated to be less than 0.05". So the cumulative play is less than the 0.1" gap between the pillars and the inside wall of the cover, as shown in Figure 32b. The tapered top edge of the pillars and the tapered button head screws further guide a misplaced cover into place. Based on these design considerations and the high number of test runs without issues I conclude that it is very unlikely that there will be a collision between cover and base during reinsertion of the cover. However, for other XPS systems the cover may be able to move laterally by more than 0.1". This could make the 2-way VTM incompatible with that XPS system.



*Figure 32. a) The horizontal leg component remains in contact with the loadlock edge, such that the lateral play of the cover is limited to 0.05". b) Safe reinsertion of the cover is ensured with a 0.1" gap between base pillar and wall of the cover. This is sufficient to absorb the 0.05" of lateral play of the cover in the loadlock, and the 0.05" of play of the base on the transport platform. Additionally, a tapered top edge of the pillar and a tapered button head screw can guide a misplaced cover over the base.*

#### 4.5.4. Failure modes during resealing in the XPS

If the 2-way VTM could be evacuated, and the base is correctly reinserted into the cover after an XPS measurement, the 2-way VTM may still not reseal. This can be caused by three errors: Insufficient leaf spring force; the sample or sample clamp may collide with the cover; the O-ring between base and cover may be dirty. The errors are ordered from most likely to least likely to cause failed resealing.

##### 4.5.4.1. Insufficient leaf spring force

If the compression force exerted by the leaf springs is too low, the 2-way VTM may not reseal in the loadlock. This can be the case, even if the 2-way VTM was successfully evacuated in the glove box antechamber, due to the lower venting rate of the XPS loadlock. Depending on the exact dimensions of the XPS loadlock and the transfer mechanism, the distance between the 2-way VTM cover and the loadlock glass may vary. This will change the compression of the springs, and the force with which the springs compress the O-ring between cover and base. The springs may also plastically deform over time, reducing the spring force exerted. Refer to section 10.5 for a description of how to ensure the springs exert sufficient force to ensure resealing.

##### 4.5.4.2. Sample or sample clamp obstruct 2-way VTM cover

As described in 4.4.1.1 the sample and sample clamps may collide with the cover, preventing resealing in the XPS.

##### 4.5.4.3. Dirty O-ring between base and cover

If the O-ring between base and cover is dirty, this may prevent sealing. Refer to section 4.4.1.4 for details.

## 4.6. Engineering standards

No applicable engineering design standards were found. However, as described above, engineering guidelines were used in designing the O-ring seals, the Parker O-ring handbook [19].

# 5. Manufacturing

In this section, I will discuss each component as outlined in the Design section, including manufacturing and critical iterations.

### 5.1.1. Manufacturing of solution cover

Initially, I used 3D-printed prototypes to test compatibility with the XPS loadlock, but key prototypes for testing were milled using the 2op TRAK CNC-mills at the Active Learning Labs (ALL). It is recommended to use flood coolant if available, to increase feeds, improve surface finish and avoid broken end mills. Before placing the milled prototypes into the XPS, I cleaned them in an ultrasound bath, using a soap-water mixture for at least 10 min, then with acetone for 10 min, and finally with methanol for 10 min. This removed remnants of coolant, oil and chips.

#### 5.1.1.1. O-ring seal in solution cover

The dovetail groove for the O-ring sealing cover and base was manufactured with a dovetail end mill [20]. Initially, the groove had corners with a smaller corner radius, resulting in a bulging O-ring. To seal an O-ring with bulging corners requires more force, so an increased corner radius was needed. The corner radius was increased from 3x the cross-sectional diameter to about 4.3x.

#### 5.1.1.2. Cover separation tool

Initially, I tried using O-rings with a higher durometer to reduce the stickiness of the O-rings. However, even with a hard O-ring (90 durometer), I could not fully prevent the base from sticking unsafely to the cover. I created a basic testing setup to test the O-ring stickiness without needing to test directly on the XPS. This setup is represented in Figure 33 below. To simulate the O-ring compression, I first placed a high mass on the cover, to simulate the loading from the pressure difference once the 2-way VTM is evacuated and transferred to the XPS. I then removed some weight and left on a low mass to simulate the load from the springs once the XPS loadlock is evacuated. I used 25 kg for the high mass, since the pressure difference results in approximately 250 N of force on the O-ring between cover and base. I used between 5 and 10 lbs to simulate the force from the springs, resulting in 20 to 45 N. I left the heavy mass on for 20 mins to simulate the duration of the transfer between glove box and XPS, and the spring mass for 2 - 10 min to simulate the evacuation in the XPS loadlock. I would then remove the weights and lift up the cover, to see whether the base would lift off with the cover. Even with the 90 durometer O-ring I could not consistently ensure the base would not lift up.

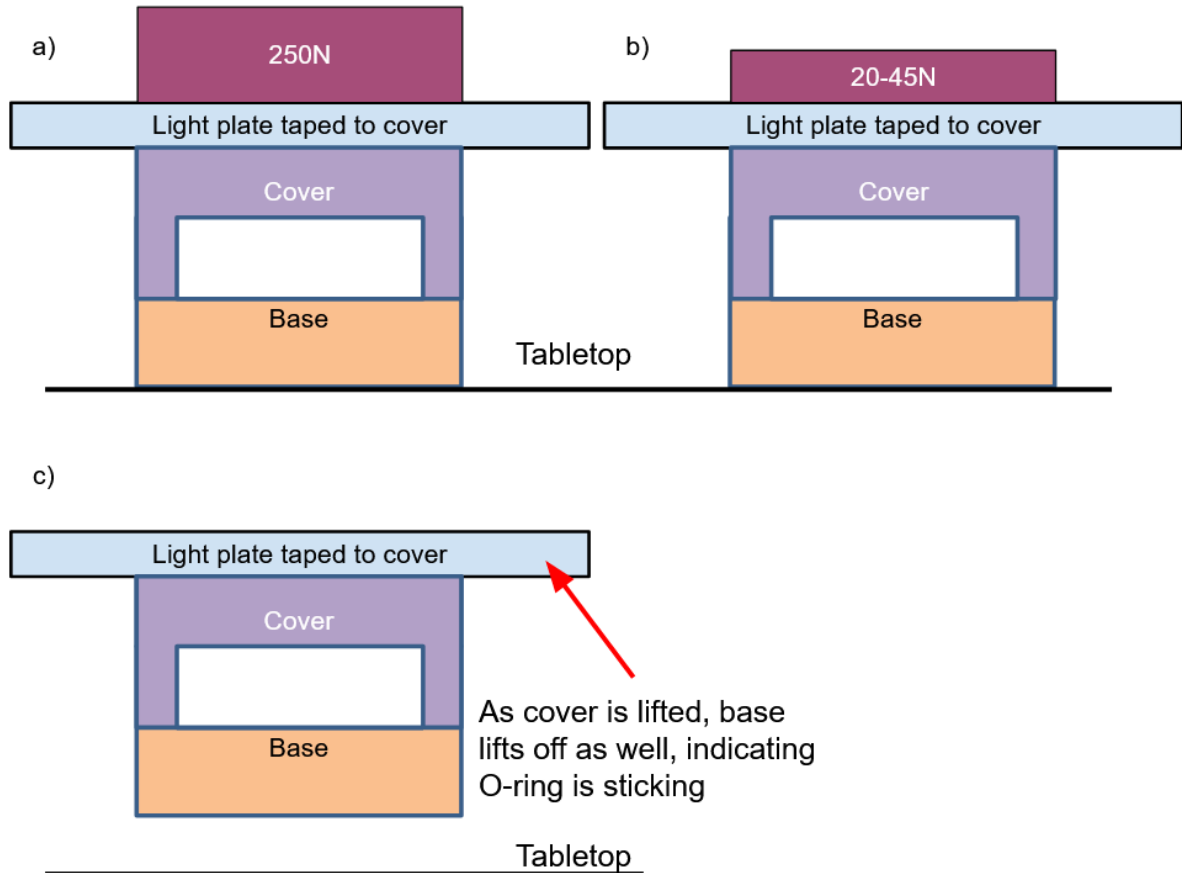


Figure 33. a) I placed weights on the 2-way VTM cover to simulate O-ring compression from the pressure difference outside the XPS loadlock. b) Then I placed lower weights on to simulate the force from the leaf springs inside the XPS loadlock. c) Finally, I lifted the cover and checked with a camera whether the base was lifting up.

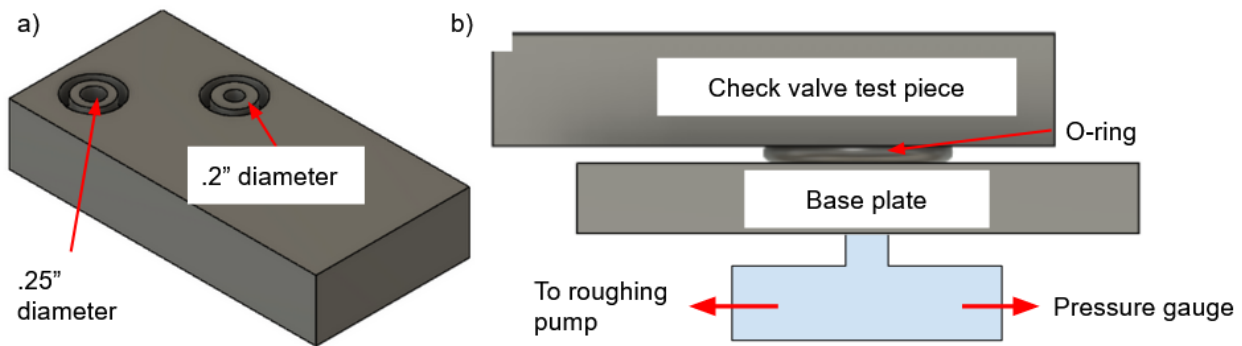
To ensure the base does not stick to the cover when the transport platform is lowered, the base and cover need to be separated in the loadlock, before the transport platform is lowered. One option is to use a magnet to lift the cover in the loadlock. The magnet needs to be external to the XPS, to ensure that it can be removed from the XPS far enough to prevent any effect on the electrons ejected during the XPS measurement. To test which combination of magnets and steel plate on the cover could lift the cover off the base, I simulated the separation between 2-way VTM cover and magnet due to the loadlock glass with a wooden plate of similar thickness. With the setup described in section 4.3.1.2 consisting of a steel plate with thickness 0.075" and five magnets I could lift the 2-way VTM and compress the springs against the wooden plate simulating the cover. When I tested this cover separation tool with the 2-way VTM in the loadlock, I could see the cover move up while the base remained stationary when I placed the tool on the loadlock cover, indicating that the O-ring had separated from the base. During testing, I used this cover separation tool more than 45 times on the Nexsa, and more than 47 times on the K-Alpha successfully. During testing, I typically left the tool on for less than 5 min. Therefore, I expect that if the cover separation tool is left on the loadlock for 10 min, consistent separation of base and cover is ensured.

### 5.1.1.3. Valve

Initially, I tried using a check valve in a single channel as shown in Figure 18 with an internal diameter of 0.136". However, I could not adjust the spring of this check valve to a setting where

the check valve would work, where it would allow evacuation and sealing with the same set screw position. As mentioned in section 4, no valve is needed to evacuate the 2-way VTM in the glove box antechamber. However, I was not aware of this at this stage of the project.

To test other valves easily, I set up a roughing vacuum pump to evacuate the 2-way VTM and various valve subassemblies with design adaptations. This setup is shown in Figure 34. It consists of an aluminum base plate that is connected to a roughing pump and a pressure gauge using KF-flanges. I could place the 2-way VTM cover or an aluminum plate with check valves of different diameters on the plate, and measure the leak rate of the system. This helped understand which check valve diameter could enable evacuation and sealing in the same position. I sealed the check valve plate to the base plate with an O-ring.



*Figure 34. a) To test the required check valve diameter to improve check valve performance, I manufactured a test plate with different check valve diameters. b) The check valve plate was evacuated using a roughing pump and the pressure increase after pump disconnection was measured with a pressure gauge.*

Using this setup, I determined that a check valve based on a 5/16"-18 set screw can be adjusted to a setting where both evacuation and sealing can be achieved. I also tested a 1/4"-20 check valve. For this valve I could find a setting where evacuation and sealing were feasible, but it was more difficult to reproduce the setting.

When I tested the check valve functionality on my final prototype, I was again unable to consistently find the correct setting of the set screw to enable both sealing and evacuation. This may be due to manufacturing inaccuracies.

To further improve robustness and avoid the need to find a correct set screw setting, I decided to have an evacuation channel separate from the bore for the set screw. This setup was shown and explained in section 4.3.1.3.

#### 5.1.1.4. Viewport

The glass plate of the viewport was manufactured using a water jet.

#### 5.1.1.5. Legs on the cover

The legs were milled using flat end and taper end mills. Initial leg iterations had more lateral play in the loadlock. By reducing the length of the horizontal component of the leg as indicated in Figure 23 from 0.13" to 0.095", I could ensure that reinsertion of the base into the cover is robust because lateral play of the cover in the loadlock is minimized, as explained in section 4.4.3.

#### 5.1.1.6. Spring system

In my final design I use leaf springs to compress base and cover. When I tested coil springs, I found these were prone to buckling. I used 1095 spring steel for the leaf springs. Stainless steel tested, because it is a common UHV material, but was found to deform plastically more easily than 1095 steel. The springs were plastically deformed by the closing loadlock cover, before the base was even transferred to the analysis chamber. Ensuring reliable compression force on the O-ring and resealing after the base was returned was difficult. I switched to 1095 spring steel, which may rust when exposed to water. In general, vacuum systems should be kept in a dry environment, to reduce outgassing from air humidity once introduced into vacuum. Regular XPS sample stages are stored in a dry box with reduced humidity to minimize moisture on the stage. Nevertheless, there is a risk of the spring steel rusting over time due to humidity in the air. This rust could be sanded off. To prevent setting of the springs I heat-treated them. I heated them to 230°C for 30 minutes and then left the oven to cool down for several hours. It would have been possible to test Beryllium Copper springs, which are UHV-compatible. However, thicker BeCu plates are more expensive and less readily available, so for my application 1095 steel is overall more suitable.

To prevent damage to the XPS, it is important to avoid excessively high spring forces. During testing with stainless steel springs I tested very high spring forces, by bending the springs upwards. I did not quantify the exact spring force, but the K-Alpha+ XPS loadlock did not close by itself. I pushed down the loadlock until it would make contact with the O-ring for the loadlock and seal. This bent the screws attaching the transport platform and this subsequently damaged the gate valve between loadlock and analysis chamber during the sample transfer into the analysis chamber. It would make intuitive sense that if the loadlock cover can close by itself, the spring force is low enough to avoid damaging the transport platform. While this is pure speculation, it seems reasonable that Thermo Fisher considered that users might place the regular sample stages into the loadlock incorrectly, such that they prevent the loadlock from closing. If the loadlock cover was strong enough to bend the screws of the transport platform, such a seemingly common user error would also damage the gate valve. To prevent damage in this failure mode, Thermo Fisher may have limited the maximum closing force of the loadlock to prevent damage to the transport platform. I expect that as long as the loadlock cover closes by itself, the springs on the 2-way VTM cover will not damage the transport platform.

#### 5.1.1.7. Manufacturing of solution base

The solution base was also milled using the 2op TRAK mills at the ALL. I manufactured early prototypes from aluminum to avoid wasting more expensive copper. The primary design changes to the base ensured the base is detected in the analysis chamber.

The XPS system uses the small pin on the bottom of the base to detect the presence of a base in the analysis chamber as explained in requirement 3.2.3.2. To ensure successful detection of the base in the analysis chamber, this pin needs to be in contact with a receptacle on the XPS analysis stage. I found that the gap between the two islands on the bottom of the cover is important to ensure detection in the XPS, as indicated in Figure 35. Standard Thermo Fisher stages have gears between the two islands, which allow rotation of the sample in the analysis chamber. The gap between the islands is necessary, to avoid interference with the gear driving system in the analysis chamber. Prototypes with the gap were successfully detected in the analysis chamber. Shown in Figure 39 is the final base prototype.

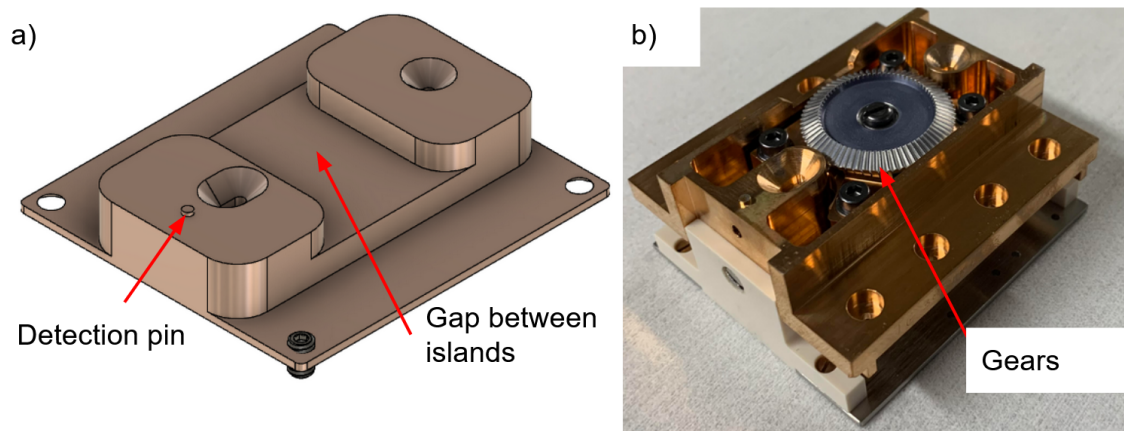


Figure 35. a) The gap between the two islands on the bottom of the base is necessary to ensure the base is detected in the XPS loadlock. b) Thermo Fisher's standard stages have gears on the bottom. If the gap in a) does not exist, the bottom of the base will collide with the gear drive system, so the pin in a) is not detected.

## 6. Performance testing and validation

The final prototype was tested against the specifications described in section 3.

### 6.1. No detectable oxidation after one transfer cycle between glove box and XPS

According to specification 3.2.1, the 2-way VTM should protect air-sensitive  $WTe_2$  samples, such that the relative quantity of W bound to O is less than 1% relative to the quantity of W bound to Te. This measurement is to be conducted on the K-Alpha+ and Nexsa XPS systems at CNS. First, I will describe the functionality of the XPS system.

#### 6.1.1. XPS measurement method

XPS measurement method utilizes the photoelectric effect, the ejection of photons from a material exposed to radiation of sufficient energy. The XPS directs x-rays at the sample, which will eject electrons from the sample surface. Knowing the energy of the x-ray beam, and measuring the kinetic energy of the ejected electron, the binding energy of the bond that the ejected electron was engaged in can be calculated. The binding energy can be used to determine the bond and the respective element. Shown in Figure 36 is a typical spectrum that can be generated with the XPS. The plot shows the number of electrons counted per second by the XPS for different binding energies. For each bond present in the material, the spectrum will show a peak. The relative quantity of oxidized and unoxidized W can be estimated by comparing the area under the peaks corresponding to oxidized and non-oxidized W. This area can be determined by fitting a mix of a Lorentzian and a Gaussian function to the peaks and determining the area under the peak from the width and height of the fitted peaks. These areas are scaled using tabulated relative sensitivity factors. Shown in Figure 36a is a typical spectrum of a non-oxidized  $WTe_2$  sample. On the right of the spectrum we see a peak pair which corresponds to the  $W4f\ 7/2$  and  $W4f\ 5/2$  shell, for W bound to Te. Additionally, we see the  $W5p$  peak. Shown in Figure 36b, we see the spectrum of an oxidized  $WTe_2$  sample. In addition to the peaks we see in Figure 36a, there is a peak pair to the left of the peaks corresponding to W

bound to Te. This peak pair also corresponds to the W4f 7/2 and 5/2 shells, but in this case the W is bound to O. Due to the difference in electronegativity between Te and O, the binding energy for this peak pair is higher. We can fit each of these peaks using Thermo Fisher's fitting software.

#### *Thermo Fisher Advantage fit parameters*

Regarding fit parameters. Since the measured electron count is non-zero at every binding energy, it is necessary to subtract the background electron count from the measured spectrum. To calculate the background I used the "Smart" background algorithm, which is recommended by Thermo Fisher [35]. I subtracted the background from the spectrum between 39.5 eV and 29 eV. The upper boundary is just below the Te4d peaks, and the lower boundary is below the W4f peaks. I then fitted the peaks. The doublet corresponding to W bound to Te is located between 30.9 and 31.6 eV, with the second peak between 2.15 and 2.19 eV above the lower peak [36]. In my fitting, the peak was sometimes located at up to 31.65 eV. I used an energy step size of 0.05 eV, so in my fits I used an allowable range for the W4f 7/2 peak of 30.9 - 31.7 eV. The peak areas should have a ratio of 0.75, in my measurements it was typically around 0.70 - 0.76 [37]. The doublet corresponding to WO<sub>3</sub> has the W4f 7/2 peak at 35.7 - 36.1, again with doublet separation of 2.15 - 2.19 eV [36]. For the location of the W5p peak, a wide range of reported locations can be found [36]. Out of the 27 reported locations, the majority were in the range of 35.7 - 36.1 with the other reported peak locations more than 0.4 eV away. Therefore, I used the range of 35.7 - 36.1 eV. The W5p peak is irrelevant for oxidation, so the peak area does not have to be included in the oxidation calculation [37]. However, it overlaps with the oxidized W4f peaks, so it needs to be accounted for in the fit. To ensure the peak area under the W5p peak is not incorrectly attributed to oxidized W, I fixed the area ratio between the W4f 5/2 and W4f 7/2 peaks to 0.75 [37]. The full width at half maximum of the W5p peak should be around 1.87 eV, in my fits it was typically only around 1 eV [37]. I decided not to fix the FWHM, because this increased fit residuals. I did not fix the FWHM of the W4f peaks either, again to minimize residuals.

#### *Calculating atomic percentage*

The electron intensity measured at a given binding energy depends on the actual atomic percentage  $n$ , the X-ray flux  $F$ , the instrument Transmission Factor  $T$ , the angular distribution factor for the electron flux  $\phi$ , the mean free path length  $\lambda$  (some electrons may not reach the detector), the photoionization cross-section  $\sigma$  (describing how likely an electron is to be ejected by a photon), and the fraction of electrons remaining,  $\gamma$  [38]. The fraction of electrons remaining describes the interaction between the hole left behind by the electron ejected by the photon and the surrounding electrons. If only a few electrons remain, this will significantly change the binding energy strength of the electrons remaining. This provides us with the following formula for the electron intensity measured:  $I = nFT\phi\lambda\sigma\gamma = n * SF$ . Rather than having to determine each of the factors other than  $n$ , we can use empirically determined relative sensitivity factors, to determine the atomic percentages from the intensity measured. I used relative sensitivity factors of 6.181 for the W4f 7/2 peak and 4.877 for the W4f 5/2 peak [38]. To calculate the atomic percentage of oxygen I used:

$$n = \frac{A_{WO_3 4f 7/2} / 6.181 + A_{WO_3 4f 5/2} / 4.877}{A_{WO_3 4f 7/2} / 6.181 + A_{WO_3 4f 5/2} / 4.877 + A_{WTe_2 4f 7/2} / 6.181 + A_{WTe_2 4f 5/2} / 4.877}$$

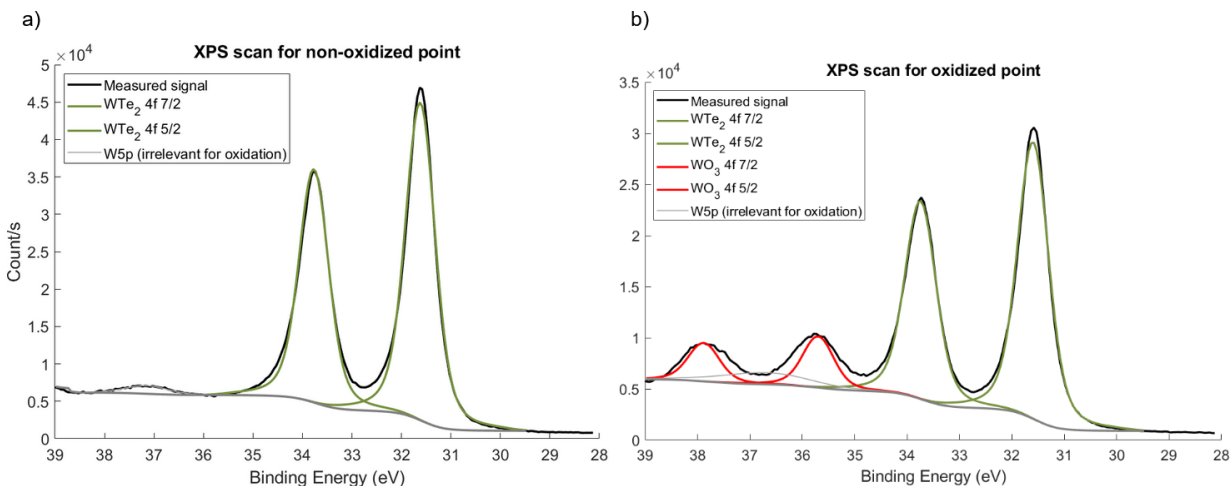


Figure 36. a) W4f spectrum of non-oxidized WTe<sub>2</sub> sample on K-Alpha+ XPS system. The WTe<sub>2</sub> 4f 7/2 and 5/2 peaks are visible, as well as the W5p peak. No WO<sub>3</sub> 4f peaks are visible. b) W4f spectrum of oxidized WTe<sub>2</sub> sample on K-Alpha+ XPS system. In addition to the WTe<sub>2</sub> W4f 7/2 and 5/2 peaks the WO<sub>3</sub> 4f 7/2 and 5/2 peaks are visible.

### 6.1.2. Testing specification 3.2.1 using the XPS

To test whether the final solution fulfills the technical specification 3.2.1, the testing procedure described in section 3.2.1 was used:

- Loading of bulk WTe<sub>2</sub> sample into 2-way VTM in glove box
- Transfer of 2-way VTM to XPS within 20 minutes
- Transfer of 2-way VTM to XPS analysis chamber and return transfer to XPS loadlock
- Removal of 2-way VTM from XPS
- Holding of solution in ambient conditions for 20 minutes to simulate transfer back to glove box
- Quantification of atomic percentage of W bound to O relative to W bound to Te, and check whether this is <1%.

For all tests, I used bulk WTe<sub>2</sub> crystals provided by Efrén Moratalla from the Crystalline Inorganic Solids Lab at the University of Valencia, Spain [39]. To ensure the samples had no oxidation prior to being loaded to the 2-way VTM, the top layer of each sample was removed by exfoliation using sticky tape in the glove box. The newly exfoliated layer was exposed to at most 0.5 ppm of oxygen in the glove box. This exfoliation was not always successful on the entire sample surface, leading to some areas showing oxidation. The exfoliated sample was then loaded to the base. I evacuated the 2-way VTM with the valve set screw fully tightened in the glove box antechamber. I transferred the 2-way VTM to the XPS in 20 mins and transferred the base with the WTe<sub>2</sub> samples to the analysis chamber and back to the loadlock. After loadlock venting, I waited for 20 mins to simulate the transfer back to the glove box. Next, I measured the oxidation on the sample by acquiring W4f spectra as shown in Figures 36. I then fitted the peaks using the fit parameters explained above, and quantified the W oxidation. I conducted measurements on both the K-Alpha+ and Nexsa. I conducted three measurement runs measuring the oxidation on 14 points of the sample each cycle for the K-Alpha+. For the Nexsa I also conducted three measurement runs measuring oxidation in at least 10 different points each cycle. I collected a total of 73 data points, of which 7 points were oxidized by more than 1%. Shown in Figure 37 is the distribution of oxidation rates measured for the K-Alpha+ system and for the Nexsa system. The outliers are more than 3 standard deviations away from the mean of the non-oxidized population. They are most likely caused by incomplete exfoliation of the

sample surface prior to the test runs. I used the same  $WTe_2$  crystal for all measurements, so with each exfoliation, less material remained. This made it more difficult to ensure that all areas on the sample were exfoliated. This is reflected in an increasing number of outliers for the Nexsa measurements, which I conducted after the K-Alpha+ measurements. Given that most points on the sample surface were not oxidized in each measurement run, and assuming that oxidation should not be a local phenomenon, the incomplete exfoliation explanation makes sense. I can conclude that the 2-way VTM ensures  $<1\%$  oxidized W in a  $WTe_2$  sample after the equivalent of one transfer cycle between glove box and XPS, fulfilling the technical specification.

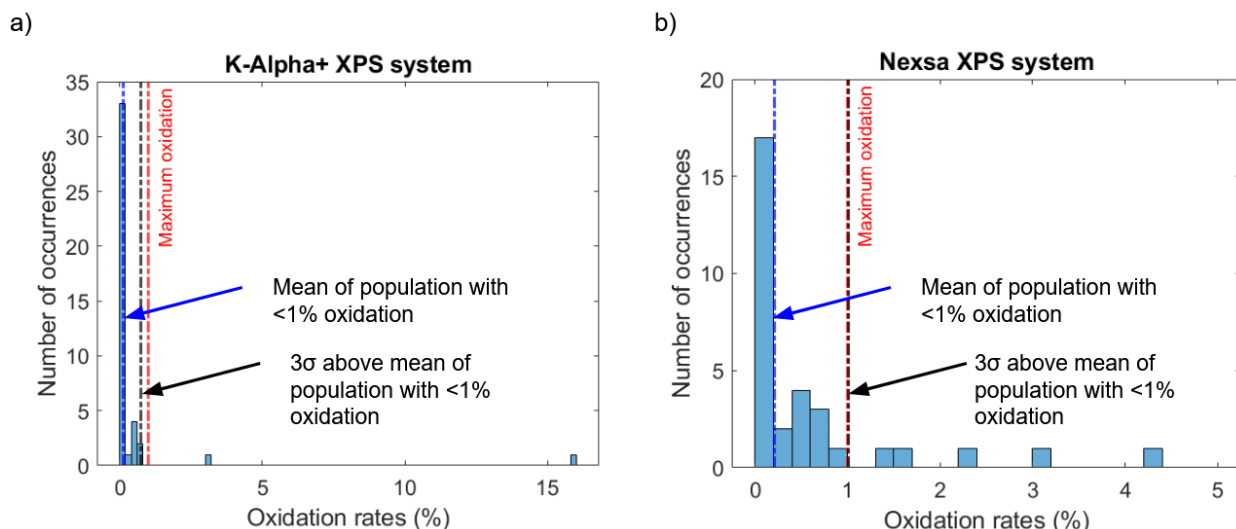


Figure 37. a) The 2-way VTM consistently achieved less than 1% oxidation rates for the K-Alpha+ XPS (a) and for the Nexsa XPS (b). The outliers were most likely caused by incomplete exfoliation prior to the test run, and were more than 2 standard deviations away from the mean of the non-oxidized population. Indicated in blue is the sample mean of the population below 1% oxidation. Indicated in black is the oxidation level that is 3 sample standard deviations above the sample mean, based on the sample standard deviation of the population with  $<1\%$  oxidation.

## 6.2. Enable loading and unloading of samples

According to specification 3.2.2, the solution needs to fulfill the following requirements shown in Table 3.

<b>3.2.2.1</b>	Secure sample loading and unloading in glove box	Overall diameter of 2-way VTM $< 12''$ to fit in glove box antechamber
<b>3.2.2.2</b>		$< 14$ Nm torque required to vent solution
<b>3.2.2.3</b>		Accommodate sample heights of at least 7mm, ensuring top of sample is at a height of 27 - 35 mm above the bottom of the solution base for all Hoffman lab sample holders
<b>3.2.2.4</b>		Accommodate sample widths and lengths of at least 19 mm x 21 mm

<b>3.2.2.5</b>		Sample plate with clamping holes of diameter 0.063" - 0.064"
<b>3.2.2.6</b>		Connection for evacuation line

Table 3. Sub-requirements for specification 3.2.2

### 6.2.1. Specification 3.2.2.1: Fit into CNS glove box

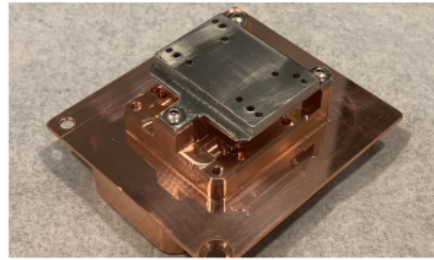
The solution needs to fit into the antechamber of the glove box available at Harvard CNS. This antechamber has an inner diameter of at least 12". I confirmed this requirement through live-testing. I transferred the 2-way VTM into the glove box using the smaller antechamber which has an internal diameter of only about 6".

### 6.2.2. Specification 3.2.2.2: Venting by hand

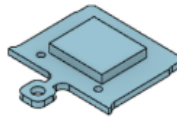
According to specification 3.2.2.2, the solution must be easily ventable by hand, using a screwdriver or allen key. I did not measure the exact torque to vent the 2-way VTM, but was able to vent it with an allen key easily. With nitrile gloves, I could even tighten the separation screw by hand, without an allen key.

### 6.2.3. Specification 3.2.2.3: Compatible with height of Hoffman lab sample holders

To ensure that the height of each of the Hoffman lab's sample holders could be accommodated, I measured the height of the standard sample plate in each of the three configurations shown below in Figure 38 using calipers. I did not load the actual sample holders to the 2-way VTM, but I could confirm that each sample holder would fit within the specification, with the top of the sample holder at a height of <35 mm, and the sample at a height of 27 mm - 35 mm. I measured the height of the sample plate using calipers in each corner of the sample plate.

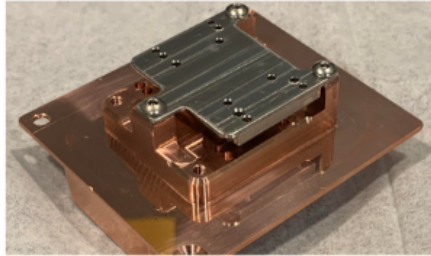


Sample plate height: 27.8 mm

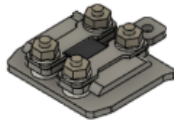


### Heights

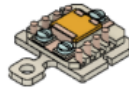
Total: 30.3mm  
Sample top: 30.3mm



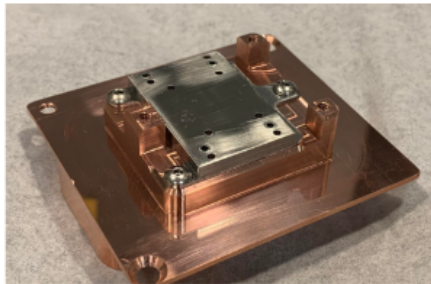
Sample plate height: 24.7- 24.8 mm



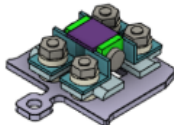
Total: 30.7-30.8mm  
Sample top: 28.7-28.8mm



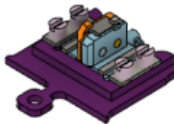
Total: 28.2-28.3mm  
Sample top: 28.2-28.3mm



Sample plate height: 21.8 mm



Total: 28.3mm  
Sample top: 28.3mm



Total: 28.8mm  
Sample top: 28.1mm

Figure 38. The left column shows the sample plate at each height position with the height of the top of the sample plate. The right column shows the resulting total height of each Hoffman lab sample holder. If applicable, the height of the sample top is listed. Each Hoffman lab sample holder can be loaded to the solution at the correct height, with the top of the sample holder at a height of <31 mm and the sample at a height of 28 mm - 31 mm.

### 6.2.4. Specification 3.2.2.4: Compatible with width and length of Hoffman lab sample holders

To ensure that the width and length of each sample holder could be accommodated, I measured the width and length of the island on the standard sample plate as indicated below using calipers. The minimum width and length required is 19 mm x 21 mm. The actual width and length is 1.29" x .88". I measured the width and length in two positions respectively, in case the island is not exactly rectangular.

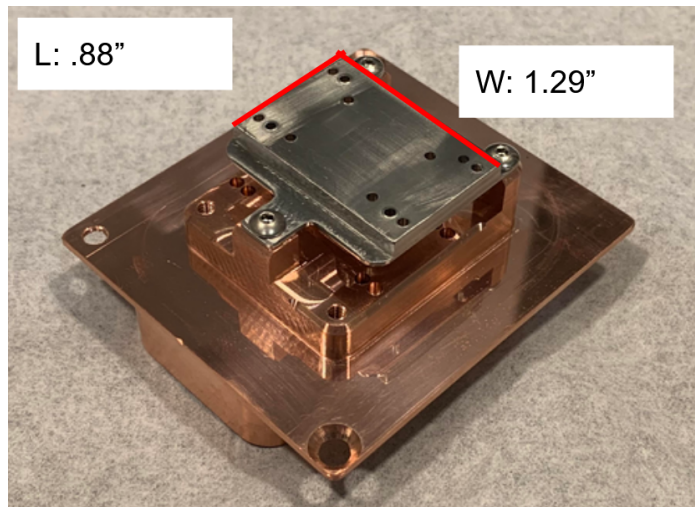


Figure 39. The island on the sample plate has a size of 1.29" x .88", within specification.

### 6.2.5. Specification 3.2.2.5: Holes for Thermo Fisher sample clamps

The solution needs a sample plate with holes for the standard Thermo Fisher clamps. This requires holes with a diameter of .063" to .064". The holes on the sample plate were drilled with a drill of diameter 0.063". The sample clamps could be inserted and tightened well, so the diameter was not increased. I tried increased hole diameters on an older sample plate, which resulted in the clamps becoming loose.

### 6.2.6. Specification 3.2.2.6: Connection for an evacuation line

The solution has an adapter for an evacuation line with diameter 0.207", measured with calipers. During testing it was possible to evacuate the 2-way VTM using this adapter. The adapter is compatible with tubes of internal diameter of 4.5 mm.

## 6.3. Compatibility with XPS systems at Harvard CNS

According to specification 3.2.3, the 2-way VTM needs to be compatible with the K-Alpha+ and Nexsa XPS system at Harvard CNS. The specifications are summarized in Table 4. Refer to the respective sections in 3.2.3 for details.

<b>3.2.3.1</b>	Compatibility with K-Alpha+ and Nexsa XPS systems at Harvard CNS	Solution base dimensions: 66 mm x 60 mm Solution cover dimensions: Width and length within Ø94 mm.
<b>3.2.3.2</b>		Compatibility with XPS transport platform (see Figure 10 for detailed specification set)

<b>3.2.3.3</b>		Ensure resealing of the 2-way VTM after XPS analysis
<b>3.2.3.4</b>		Loadlock can be pumped down sufficiently to transfer the base to the analysis chamber Analysis chamber can be evacuated to 8e-8 mbar
<b>3.2.3.5</b>		Resistance between sample mounting plate and bottom of solution base <1Ω
<b>3.2.3.6</b>		Viewport with dimensions > 1" x 1"

*Table 4. Solution needs to be compatible with XPS systems at Harvard CNS.*

### 6.3.1. Specification 3.2.3.1 and 3.2.3.2: Dimensions compatible with XPS systems

To confirm this requirement, the 2-way VTM was placed in the loadlock, the base was transferred to the analysis chamber, the sample contained was analyzed with the XPS, and then returned to the XPS loadlock multiple times. To confirm requirement 3.2.1 at least three repetitions of loading the 2-way VTM, transferring the base to the analysis chamber and back were conducted on the K-Alpha+ XPS and at least four measurements were conducted on the Nexsa XPS, including three to quantify oxidation performance. I also tested the potentially error-prone lowering of the base with the transport platform and the reinsertion of the base in the cover separately. In total, I tested reinsertion 47 times on the K-Alpha+ and 45 times on the Nexsa XPS. These tests were with the final prototype iteration and the second to last iteration, which was nearly equivalent to the final prototype. The second to last iteration did not have centering cones. No problems occurred during these tests. Therefore, the solution must be sufficiently compatible with the dimensions of the XPS.

### 6.3.2. Specification 3.2.3.3: Resealing in the XPS loadlock

The 2-way VTM needs to reseal in the XPS loadlock as it is vented. This requirement is closely tied to the oxidation performance requirement. Since the oxidation rate is less than 1% during a full transfer cycle, the resealing requirement is also fulfilled. Additionally, I checked that the base and cover did not separate by hand after venting the loadlock.

### 6.3.3. Specification 3.2.3.4: Compatible outgassing of all materials

As specified in section 3.2.3.4, all materials used in the 2-way VTM need to have a maximal outgassing rate such that the XPS loadlock can be evacuated to a pressure where transfer of the base to the analysis chamber is possible, and a pressure of <8e-8 mbar can be achieved in the analysis chamber. While conducting my oxidation tests I had no issues reaching a sufficiently low pressure in the loadlock to transfer the base. I was able to reach <8e-8 mbar in the analysis chamber of both XPS systems. The XPS analysis is typically conducted at 3e-7 mbar. As the x-ray guns turn on, the analysis chamber pressure typically increases.

#### 6.3.4. Specification 3.2.3.5: Low resistance between sample plate and bottom of base

The resistance between the top of the sample plate and the bottom of the base should be  $<1\Omega$ . To confirm this requirement I used a Fluke 179 multimeter to measure the resistance between the top of the sample plate and the bottom of the base as shown below. The final prototype had a resistance between the bottom of the base and the sample plate of  $0.4\Omega$ . This is within specification, the Thermo Fisher stages have about  $0.7\Omega$  of resistance.

#### 6.3.5. Specification 3.2.3.6: Viewport with dimensions of at least 1" x 1"

I measured the size of the 2-way VTM's viewport using a caliper in the positions indicated in Figure 40 below. The width and length are equal to or higher than 1", as required. This does not include the curved section of the viewport corners.

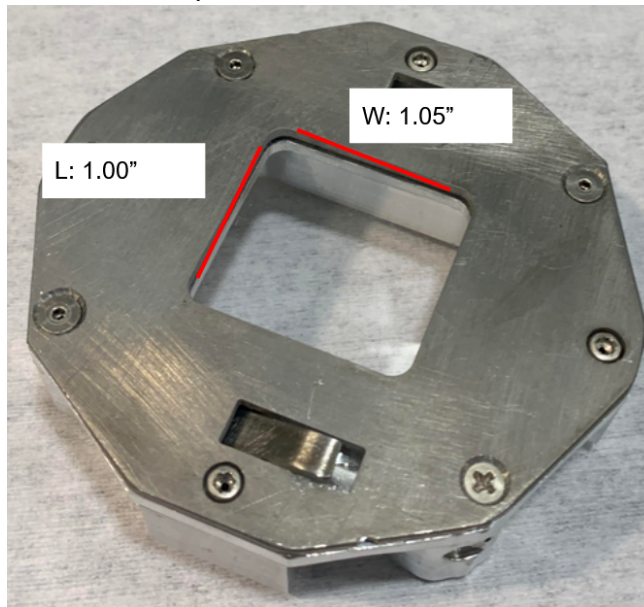


Figure 40. The size of the viewport is 1.00" x 1.05", larger than required.

### 6.4. Manufacturability with ALL equipment

Since all prototypes were manufactured with equipment available at the ALL labs, this requirement is fulfilled.

## 7. Budget

The project including all prototype iterations cost \$1065. One prototype is estimated to cost about \$300, with a detailed breakdown of parts in the Appendix 10.3.

No specific design decisions to reduce cost were made, because this was not a specification. However, in prototyping, only the later versions of the base were manufactured with copper, early iterations were made from aluminum or stainless steel to reduce costs.

If this device were introduced into the market, manufactured by a machinist, a prototype would likely cost around \$1800. Stock materials would cost around \$300 per 2-way VTM. At an estimated 30h of machining time for a full prototype, and a speculated rate of \$50/h, this would result in labor costs of \$1500. Alternatively, the key CNC-machined parts could be ordered for less than \$500 from Xometry, an online CNC-machining service [40]. Smaller parts like the springs or the glass plate would likely need to be manufactured by a machinist. For reference, Thermo Fisher sold their TF VTM to the Li group at Harvard for \$9000 [15]. Note that this price also includes R&D, distribution and overhead costs.

## 8. Conclusions and future work

I developed, built, and tested a two-way vacuum transfer device compatible with the K-Alpha+ and Nexsa XPS system at Harvard CNS. Based on testing with oxygen-sensitive  $WTe_2$ , the transfer device achieved no detectable oxidation during the equivalent of a full transfer cycle between glove box and the XPS system. The device is ready for use by the Hoffman lab. The Hoffman lab and other potential clients will benefit from using the solution, because air-sensitive samples can be reused after XPS analysis. Additionally, the new device can be built at a lower cost than the commercial one-way solution from Thermo Fisher.

Moving forward, it would be interesting to further assess the oxidation performance using a more air-sensitive sample. For example, this would make it possible to assess which evacuation method minimizes sample oxidation, evacuation in the antechamber or using a roughing pump. To improve the value of the 2-way VTM to potential users, user feedback should be gathered to make the 2-way VTM easy and robust to use. To make the 2-way VTM usable on XPS systems other than those at CNS, further testing should be conducted on non-Harvard XPS systems. Considerations regarding societal, cultural and economical factors are included in Appendix 10.2.

## 9. Acknowledgements

I would like to acknowledge the help from my advisors, Harvard CNS, and ALL. Kevin Hauser provided outstanding mentorship, insights, and ideas throughout the project. He had a large part in helping me find a functional solution, resolve obstacles through structured testing, and present my work effectively. Thank you to Prof. Jennifer Hoffman, and the Hoffman lab who provided important advice and lab resources. Greg H. Lin from Harvard CNS was a key enabler for the project, because he supported my testing on the Harvard XPS systems. Without his consistent support, even when I was damaging the XPS and my work was resulting in frequent down-time of the XPS in January and February, I could not have found a functional design. Thank you also to Austin Akey, who provided me with a glove box and a custom roughing pump setup for the project. Thank you to CNS for waiving facility usage fees for the project. Importantly, thanks to the Active Learning Labs, including Elaine Kristant, Alejandro Lopez, Steve Cortesa and Hanna Quigley for supporting my extensive machining work. Thanks to the ES100 staff.

## 10. Appendix

### 10.1. Engineering standards considered in design

As outlined previously, I did not find any engineering standards applicable to the design challenges in this project. However, standards exist to support the XPS analysis of samples. Specifically, ISO standard 10810 is a useful standard to validate a robust oxidation quantification method [41].

After the introductory chapters 1 through 5, chapter 6 describes the specimen characterization and considerations to be made before the actual XPS analysis. I analyzed an exfoliated thin layer of bulk  $WTe_2$ , and section 6.2.3 specifies that quantitative analysis of such segregated layers is generally possible. While no specific section on analysis considerations for TMDs is provided, the section on metals is applicable to  $WTe_2$ . It confirms that metals may develop an oxide layer, preventing accurate analysis. The standard recommends sputtering, removing the top surface layer through ion bombardment. In my case I want to determine how much oxidized  $WTe_2$  is at the surface, so I did not conduct any sputtering.

Section 7 of the ISO report discusses how the XPS system should be set up and calibrated. CNS ensures a functional and calibrated XPS system.

Section 8 describes how a wide-spectrum XPS scan should be performed. My analysis focuses on a narrow band spectrum, to focus on the W and their respective oxidized states.

Section 9 describes how a narrow band spectrum should be performed. The report mentions the importance of correctly choosing the binding energy step width to be used for the narrow scan. It depends primarily on the full width at half of the maximum value (FWHM) of the narrowest peak to be analyzed. A specific step size based on the FWHM is not provided, but at most 0.1 eV should be used. The report refers to a different report to determine the dwell time at each binding energy [42]. Unfortunately, I was not able to find recommendations of dwell times to use in this referenced report. The report also recommends generally recording the C1s and O1s regions along with other regions of interest, for me that is the W4f region. In some of my tests I did not record the C1s peak. Once the narrow band spectra are recorded, the standard recommends using databases to identify chemical states corresponding to the peaks measured. I used Thermo Fisher's knowledge base and outside sources as explained previously [43]. To then quantify the sample composition, the standard recommends that the background spectrum is subtracted from the peaks first. Thermo Fisher's software has algorithms built in to calculate the background. The standard then recommends using the software's built in sensitivity factors to quantitatively determine the sample composition based on the peaks detected. Avantage also has such features built in. The standard warns that this analysis is only an approximation. As described previously, CNS staff has estimated the accuracy for this method to be about 1%, such that it seems reasonable to use this quantification approach. The rest of the section relates to analysis methods irrelevant to my project, like composition quantification varying with depth.

In designing the O-ring seals of the 2-way VTM, I relied on the Parker O-ring handbook as an engineering guideline [19]. Among other information it provides specific dimensions for the sizing of O-ring grooves for different O-ring cross-sections. I used this guidance to design the rectangular O-ring groove to seal the viewport glass, the dovetail groove to seal cover and base and the O-rings in the plug valve. It also provides recommendations for the compression force of O-rings with different diameters and durometers, which helped inform my decision to use a larger O-ring diameter for the check valve as described above.

## 10.2. Design considerations regarding public health, social, cultural, environmental, global and economic factors

### 10.2.1. Public health and welfare

In general, usage of the 2-way VTM does not appear to result in any health hazards, as no toxic or harmful materials are used. Manufacturing using milling can result in sharp edges, so all sharp edges were rounded off on a grinder. This also helps prevent tearing of glove box gloves, which could contaminate the glove box.

### 10.2.2. Global factors

There appear to be few potential global factors that would need to be considered. Procurement of parts was found to be straightforward despite global supply chain disruptions due to COVID-19.

### 10.2.3. Cultural factors

No specific cultural factors were considered in the design. However, a successful product would be used not just by the Hoffman lab, but could also be used by other groups, which can further strengthen the relationships between these labs.

### 10.2.4. Social factors

Along the lines of the argument made under cultural factors, a successful product can be used across other Physics labs at Harvard, or potentially even undergraduates, depending on the needs of the Hoffman lab. This can allow other groups to analyze air-sensitive samples on the XPS without each sample being oxidized after an XPS analysis.

### 10.2.5. Environmental factors

No major environmental damage is done in the manufacturing of this product. Ideally, recycled stock materials should be used, as available.

### 10.2.6. Economical factors

A successful device may have substantial economic effects, by making a 2-way VTM accessible to more research institutions and companies. While Thermo Fisher's VTM costs about \$9000, the required investment for my solution is less than \$500 in material costs and about 30h in machining time. This may be more affordable for some users meaning my solution may enable more research institutions to conduct insightful analysis of air-sensitive samples using the Thermo Fisher XPS systems.

## 10.3. Bill of materials

Below is a breakdown of the cost needed to manufacture the final prototype. For stock materials, the price of the stock actually used is given. So if a piece of stock metal costs 100\$, but only 25% is used in one prototype, the cost of stock is listed as 25\$.

Item	Function description	Total Cost (\$)
Bar of copper (4" x 3" x 1")	Used to mill the 2-way VTM base.	58\$ [44]
1095 spring steel strip (0.02" thick)	Used to create springs on cover	5\$ [45]
Bar of aluminum 1" x 3/8" x 1ft	Used to create the 2-way VTM cover and legs	5\$ [46]
Aluminum sheet (1 1/4" x 4" x 4")	Used to create cover plate	13\$ [47]
1x Mild steel plate 4" x 4" x 0.075"	Used to create steel plate for cover separation	3\$ [48]
1x Viton O-ring Dash 228 Durometer 60	Used to establish seal between 2-way VTM cover and base	5\$ [49]
1x Viton O-ring 2mm CD, 46mm ID	Used to establish seal between viewport and cover	1\$ [50]
1x Viton O-ring 1mm CD, 4.5mm ID	Used in check valve	1\$ [51]
3x Vented flat head screws	Used to fixate steel plate on cover	7\$ [25]
1x Regular flat head screw	Used to fixate steel plate on cover	1\$ [26]
4x Vented socket head screws 4-40	Used to attach legs to cover plate	9\$ [32]
6x Vented Button Head Screw	Used to attach standard sample plate to base	10\$ [52]
2x Vented Socket Head Screw 2-56	Used to attach springs to cover plate	3\$ [53]
5/16-18 Set screw	Used for check valve (sold in pack of 100)	1\$ [30]
1x Spring, 0.18" OD	Used for check valve	5\$ [29]
1x Steel balls .25"	Used in check valve (sold in pack of 100)	1\$ [28]
1x Viewport glass 2" x 2" x 1/16"	Used to manufacture viewport	15\$ [54]
1x Steel sheet 2.5" x 1.3" x 1/4"	Used in standard sample plate	10\$ [55]
1x Stainless Steel Socket Head Screw 8-32"	Used to separate base and cover	4\$ [56]
4x Neodymium magnets 3/4" OD, 1" thick	Used to pull up cover in XPS loadlock	82\$ [22]
1x Neodymium magnet 1.5" OD, 1" thick	Used to pull up cover in XPS loadlock	57\$ [23]
Total for final solution		296\$

*Table 5. Bill of materials for one prototype.*

Additionally, I created a [spreadsheet](#) with all components purchased over the course of my project, and expenses. I have exceeded the ES100 budget due to the large number of prototypes needed to achieve my technical specifications, but have communicated with Anas Challah to ensure a budget extension.

## 10.4. Technical resources

CAD models for all manufactured components can be found [here](#).

It would not make sense to upload technical drawings here, because my components have complex shapes, especially the cover block and the base platform. This was aligned with ES100 staff. No specification sheets are included, because no complex component was purchased.

## 10.5. Maintenance procedure: Ensuring sufficient leaf spring force for resealing

This paragraph illustrates how to calibrate the 2-way VTM springs, such that resealing of the 2-way VTM in the XPS load on the K-Alpha+ and Nexsa at CNS is guaranteed. This calibration is needed when new springs are manufactured, or if the 2-way VTM fails to reseal. If new springs are manufactured, the correct bending geometry needs to be ensured. Existing springs can plastically deform after repeated measurement cycles. Since the effective spring force is dependent on the XPS loadlock geometry, the calibration procedures outlined below are slightly different for the K-Alpha+ and Nexsa system at CNS.

Initially, the springs should be nearly flat, such that they are not compressed when the loadlock is closed. Progressively, the springs should be bent upwards more and more, until the 2-way VTM reseals. On the K-Alpha+ and Nexsa XPS systems I used the setup below to quantify the spring force at a certain bending geometry. I placed weights on a light wooden plate onto the 2-way VTM cover as shown in Figure 41a until there was no gap between the cover and the wooden plate visible. Then I removed 2.5 lbs, and checked that there was a gap. From this, I could quantify the spring force at full compression within that range. To prevent spring setting during this test, the springs should be deformed as little as possible. I ensured the springs were compressed by less than 0.1", but more may be acceptable. Since the Nexsa XPS at Harvard CNS has a larger gap between 2-way VTM cover and loadlock glass, I placed a second 0.075" steel plate on top of the cover as shown in Figure 41b. This reduced the compression of the springs preventing the springs from plastically deforming.

To allow reliable resealing on the K-Alpha+ I found that the springs should be bent upwards far enough that I could place between 5-7.5 lbs on top to fully compress both springs onto one steel plate (as illustrated in Figure 41a). For the Nexsa, at least between 5-7.5 lbs are needed, compressing onto a second 0.075" steel plate placed on the cover.

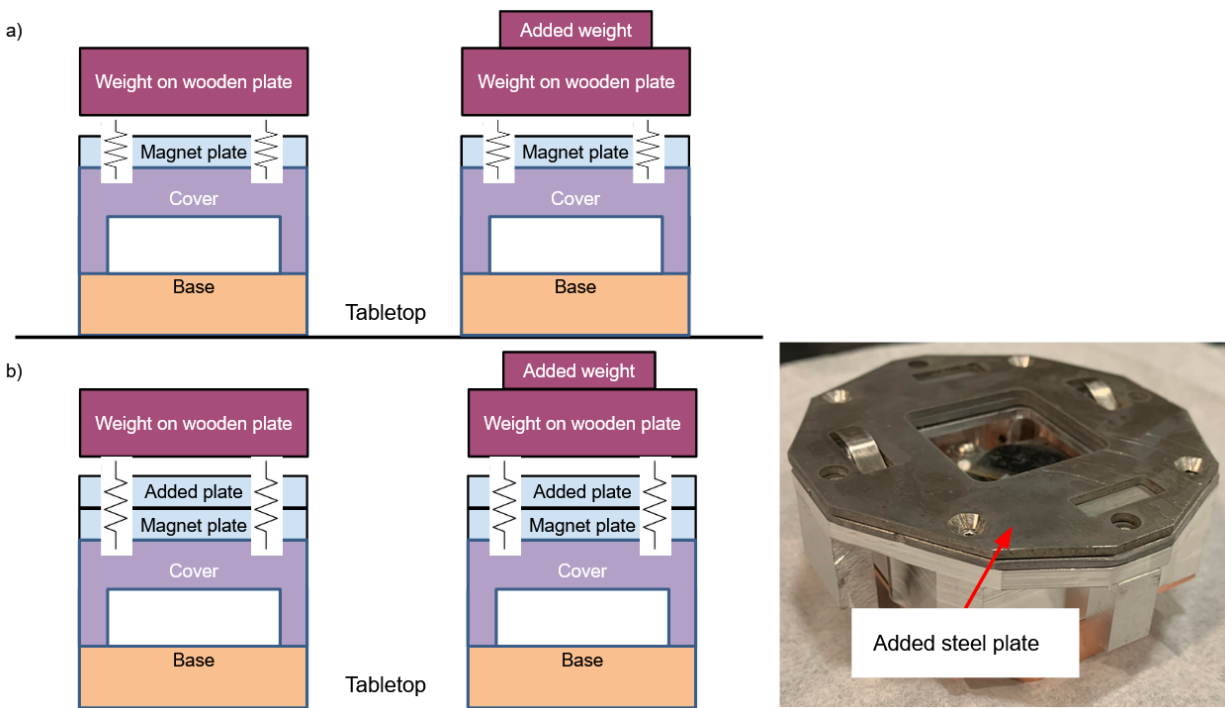


Figure 41. a) To ensure sufficient spring force to reseal, weights should be placed on the cover to test the force needed to fully compress the springs onto the magnetic steel plate on top of the cover. b) For the Nexsa XPS at Harvard CNS, the springs have to be bent up more to ensure resealing, because the distance between the 2-way VTM cover and the loadlock glass is larger. Compressing these springs until they are level with the top of the magnet plate would plastically deform the springs, so I placed a second steel plate on the cover, and quantified the weight needed to fully compress the springs to this level.

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