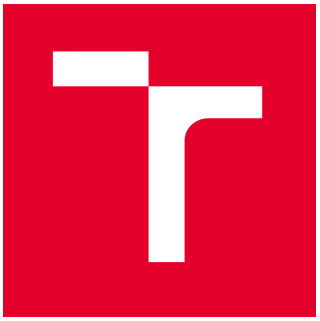

BRNO UNIVERSITY OF TECHNOLOGY

VYSOKÉ UČENÍ TECHNICKÉ V BRNĚ

FACULTY OF MECHANICAL ENGINEERING

FAKULTA STROJNÍHO INŽENÝRSTVÍ

INSTITUTE OF AEROSPACE ENGINEERING

LETECKÝ ÚSTAV

DEVELOPMENT OF INFRASTRUCTURE FOR TESTING MATERIALS UNDER VACUUM – OUTGASSING

VÝVOJ INFRASTRUKTURY PRO TESTOVÁNÍ MATERIÁLŮ PŘI PŮSOBENÍ VAKUA – OUTGASSING

MASTER'S THESIS

DIPLOMOVÁ PRÁCE

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VEDOUCÍ PRÁCE

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Assignment Master's Thesis

Institut:	Institute of Aerospace Engineering
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As provided for by the Act No. 111/98 Coll. on higher education institutions and the BUT Study and Examination Regulations, the director of the Institute hereby assigns the following topic of Master's Thesis:

Development of infrastructure for testing materials under vacuum – outgassing

Brief Description:

Outgassing is one of the material properties crucial for space applications. Low outgassing requirements are encountered practically in every satellite design, where the limits set in space standards must be met. However, the outgassing characteristics of, for example, 3D printed materials are often lacking and these materials need to be qualified. Outgassing testing is carried out in specialised thermo–vacuum chambers. What are the design requirements for these chambers and what might such a chamber for the IAE BUT look like?

Master's Thesis goals:

– describe and analyse outgassing testing approaches.
– present conceptual designs of sample fixture for degassing chamber.
– test rig/device specification and test procedures.

Recommended bibliography:

ASTM International. 1993. "ASTM E595-93: Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment." West Conshohocken, PA: ASTM International.

ECSS-Q-ST-70-02C: Space Product Assurance - Materials, mechanical parts and processes. 15 November 2008. Paris, France: European Space Agency.

Deadline for submission Master's Thesis is given by the Schedule of the Academic year 2023/24

In Brno,

L. S.

doc. Ing. Jaroslav Juračka, Ph.D. Director of the Institute	doc. Ing. Jiří Hlinka, Ph.D. FME dean

Abstract

This master thesis deals with the design of a testing device outgassing properties of materials. In the research part, standards dealing with this topic are described and research of existing devices from publicly available sources is carried out. The output of the theoretical part is a list of a total of seventeen requirements that the resulting device must meet.

In the practical part, a list of all systems entering the device is created and then a detailed model of the device is created. The device consists of a vacuum chamber and an inner apparatus in which the test takes place. Lastly, the fulfillment of all set requirements is evaluated, and a test plan is presented.

Keywords

Outgassing, TML, CVCM, outgassing testing, vacuum testing, vacuum chamber, design, space, vacuum

Abstrakt

Tato diplomová práce se zabývá návrhem testovacího zařízení pro testování odplyňování (outgassing) materiálů. V teoretické části jsou popsány standarty zabývající se danou tématikou a je provedena rešerše existujících zařízení z veřejně dostupných zdrojů. Výstup teoretické části je seznam celkem sedmnácti požadavků, které musí výsledné zařízení splňovat.

V praktické části je vytvořen seznam všech systémů, vstupujících do zařízení a následně je vytvořen detailní model zařízení. Zařízení se skládá z vakuové komory a vnitřního aparátu, ve kterém probíhá daný test. Na závěr je vyhodnoceno splnění všech stanovených požadavků a je představen testovací plán.

Klíčová slova

Odplyňování, TML, CVCM, testování odplyňování, testování ve vakuu, vakuová komora, design, vesmír, vakuum

Rozšířený abstrakt

Odplyňování (*outgassing*) je proces, při kterém se z materiálů samovolně uvolňují plynné částice nebo molekuly. Tento proces je pak urychlen, pokud je materiál vystaven vakuu nebo zvýšené teplotě. Pokud je odplyňování při návrhu vesmírného prostředku zanedbáno, může dojít ke zkondenzování odplyněních částic na kritických součástech prostředku a k ohrožení celé mise. Proto je to pro vesmírný průmysl důležitý parametr, který je potřeba u materiálů sledovat. Materiály, které jsou schopny vlivem odplyňování ztratit více než 1 % své původní hmotnosti nejsou vhodné pro vesmírné mise.

Již od roku 1977 existují standardizované testy, které mají usnadnit výběr materiálu. Podle standardů ESA a NASA existují dva testy, původní, statický test, kde se testuje celková ztracená hmotnost a druhý novější, kde se testuje kinematika outgassing.

Tato diplomová práce se zabývá návrhem testovacího zařízení podle původního standardu, protože pro něj již existuje databáze materiálů, se kterými se dají nové výsledky porovnat. Navíc je tento typ testu bohatě dostačující pro většinu misí včetně misí s CubeSaty.

Tento test probíhá tak, že je vzorek materiálu vystaven vakuu a zvýšené teplotě 125 °C během 24 hodin. Zároveň s tím, je naproti materiálu umístěno sběrné sklíčko, které je udržováno na 25 °C. Po uběhnutí 24 hodin je zváženo, kolik hmotnosti vzorek materiálu ztratil a kolik hmotnosti sběrné sklíčko nabralo.

V první části této práce jsou tedy prostudovány relevantní standardy a veřejně dostupné informace o již existujících zařízeních tohoto typu, a z toho je následně vytvořen soupis požadavků, které musí výsledné zařízení splňovat. Dále je přidáno několik požadavků vyplývajících z již používaných zařízení na VUT, se kterými musí být toto zařízení schopno pracovat.

Po finalizaci požadavků, kterých je celkem 17, se přešlo k výběru konkrétních podsystémů, které by splňovali dané požadavky. Byl proveden výběr systému zahřívání vzorků a chlazení sběrných sklíček, systém sledování úrovně vakua uvnitř vakuové komory, systém vpuštění čistého dusíku do komory, a další.

Po vybrání všech potřebných podsystémů bylo možné začít s návrhem zařízení. Navržené testovací zařízení má místo na 13 vzorků a bylo navrženo tak, aby bylo co nejjednodušeji vyrobitelné. Kromě samotného zařízení byla navržena také vakuová komora, ve které bude test probíhat, a která obsahuje všechny potřebné konektory pro zapojení všech systémů testovacího zařízení.

Pro navržené zařízení byl proveden cenový rozbor, jehož výsledkem je cena přibližná cena minimálně 720 000 Kč.

Nakonec byl prezentován také podrobný postup testování v navrženém zařízení a bylo shrnuto splnění všech požadavků.

Bibliography

VEČEŘA, Tomáš. *Development of infrastructure for testing materials under vacuum – outgassing* [online]. Brno, 2024 [cit. 2024-02-25]. Available at: https://www.vut.cz/studenti/zav-prace/detail/158044. Master´s thesis. Brno University of Technology, Faculty of Mechanical Engineering, Institute of Aerospace Engineering. Supervisor: Václav Lazar.

Declaration of authenticity

I declare that I have written my master's thesis on the topic of Development of infrastructure for testing materials under vacuum – outgassing independently under the supervision of Ing. Václav Lazar using literature listed in the bibliography section at the end of the thesis.

place, date

Tomáš Večeřa

Acknowledgement

I would like to thank the supervisor of my master thesis, Ing. Václav Lazar, for his guidance, and valuable advice.

Contents

Introduction

Outgassing is a phenomenon where molecules trapped in or on a material are slowly released when the material is exposed to a vacuum. Several mechanics can be at play during this process [1; 2]. Namely desorption, evaporation, diffusion, and permeation.

Desorption is a process where molecules absorbed on top of materials are released to vacuum due to their random thermal movement. Evaporation is a process where liquids or solid contaminants undergo a phase change to a gas phase, thus escaping to vacuum. Diffusion is the random movement of molecules trapped deep inside a material. Those that reach the surface of a material can be further desorbed to vacuum. Finally, permeation is a process where gas molecules trapped in a closed container permeate through the walls. This is most notable for fuel containers. A schema of these processes can be seen in figure 1.

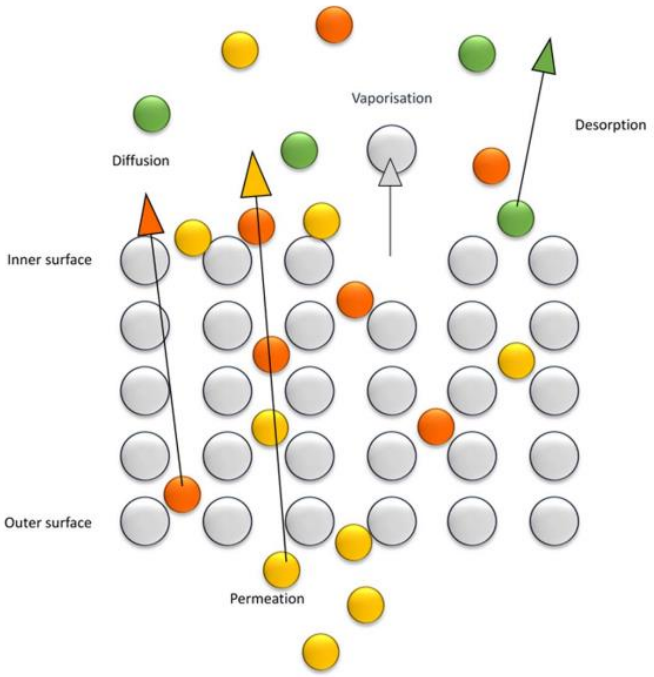

Figure 1: The mechanisms of outgassing visualized. [3]

The process of outgassing is not instantaneous, so the impact of it is not immediately visible. The rate and amount of outgassing varies greatly based on the material in question and the environmental conditions it is subjected to. The outgassed molecules can then condense on other surfaces. This can be a huge problem in space exploration if left unaccounted for, as condensation can occur on mission-critical surfaces, greatly affecting the mission outcome.

The condensation on spacecraft surfaces can affect anything from the optical imaging apparatus to the energy distribution or thermal management system of a spacecraft [4]. Condensation can reduce the effectiveness of solar panels or radiators, thus possibly shortening the spacecraft's lifetime. Condensation on mission-critical optical hardware can reduce the mission's success by reducing the imaging quality or introducing unwanted artifacts on the images. This happened on the NASA Cassini spacecraft, where a condensation on one of the cameras left the images with a halo around bright objects (figure 2).

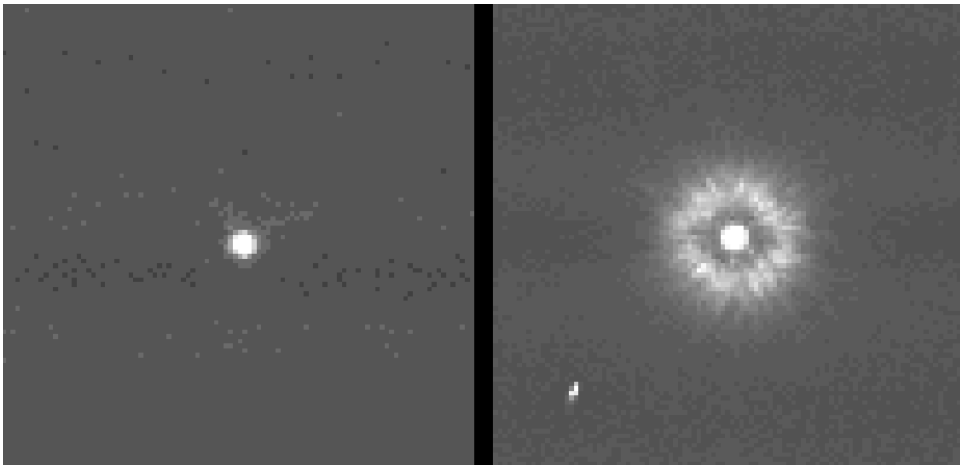

Figure 2: An image of a star taken with the Cassini Narrow Angle Camera before (left) and after (right) contamination. [5]

Due to these risks, several tests were created to review the outgassing properties of materials before using them on spacecraft. The most common and the oldest is a test, to see how much mass a material subject to vacuum loses. To speed up the test, the samples are usually heated up as well, as this gives the gas particles more energy. This is called a *Total mass loss* test, or TML for short [6]. However not all the released particles will stick to surrounding surfaces and create a potential hazard for the mission. To measure the deposition characteristics of the released gas particles for each material, a second value is measured during the test. This value is the mass of a cooled collector plate gained during the TML test. It is called *Collected volatile condensable materials*, or CVCM for short [6]. Typically, materials that lose more than 1 % of their initial mass or condense more than 0.1 % of their initial mass are deemed unfit for use in space applications, however this can differ by mission.

The TML test provides simple to understand and easily comparable data for tested materials and is still in use today [7]. Lately, however, this test has been shown to not be enough for certain applications and for computer simulations of material deposition. Therefore, a new test that measures the outgassing in time and with varying sample and collector plate temperatures has been developed called *Kinetic outgassing* [8]. This test does not replace the TML test, it is rather aimed at supplementing it for applications where more detailed outgassing and deposition characteristics are required.

Today, it is common practice to use in all space applications only materials that have been tested by the TML test. Some more advanced and expensive missions will even require the measurement of outgassing in time. However, with the advent and mass adoption of 3D printed materials, a lot of new materials that are tempting to be used in space applications lack this data. Therefore, this thesis aims to design an outgassing testing apparatus for BUT, where these materials could be tested and subsequently compared to existing databases, without the need to have these materials tested by external contractors.

1. Testing standards and guidelines

As mentioned before, today there are multiple tests that can be used to determine the outgassing characteristics of a material. These were developed after the end of the space race, as the space industry stopped being about being first, and more time could be spent developing standards regarding all domains of spaceflight. A timeline of the release dates of the most used standards regarding outgassing in space is in figure 3.

Starting in the seventies, both NASA and ESA were looking into standardizing the outgassing properties of materials so that they could be compared and that the selection of materials for space application would become easier.

The first standard to come out was from NASA, called *Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment* with the designation *ASTM E-595-77*. This standard came out in 1977 and has been regularly updated with the latest version being ASTM E-595-15R21 from 2021 [6].

The first ESA equivalent standard *ESA-PSS-01-702* came out in 1985 with the name *A thermal vacuum test for the screening of space materials* [9]. This standard was later replaced by a new standard in the ECSS framework, formed to unify the European standards for European space activities [10]. The up-to-date version of the standard is the *ECSS-Q-ST-70-02C*, called *Thermal vacuum outgassing test for the screening of space material* [11].

All of the above standards measure the two main characteristics of a material, TML and CVCM. However, these properties do not describe the evolution of outgassing in time, for this a new test was designed. Originally developed by the U.S. Air Force Materials Laboratory, the *Standard Test Method for Contamination Outgassing Characteristics of Spacecraft Materials* designated *ASTM E-1559*, is a test that can measure the outgassing rate in time and based on different sample and collector temperatures [8]. This however means, that this test is incompatible with the previous standards, and a sample measured by this kinetic outgassing test cannot be easily compared to existing databases of TML and CVCM data.

While the ECSS does not have an equivalent standard to the *ASTM E-1559*, they have released a technical memorandum *ECSS-Q-TM-70-52A*, a non-normative document aimed at providing useful information about the kinetics of outgassing, its testing and modelling, specifically referencing the *ASTM E-1559* standard [1].

All of these four up-to-date documents are described in more detail in the following chapters.

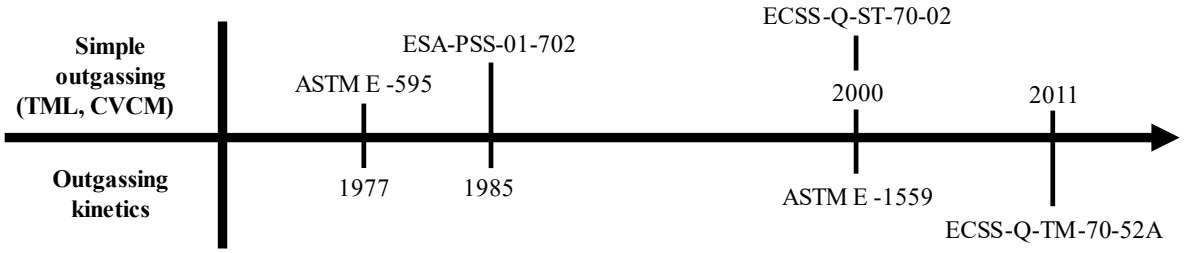

Figure 3: Timeline of first releases of each document.

1.1. ASTM E-595

This document was the first to standardize the test method and evaluation of outgassing and deposition characteristics of materials. The method aims to measure three material properties, defined in the document.

As stated before, the two main measured properties are total mass loss (TML), defined as the reduction of mass of the sample during the test, and collected volatile condensable material (CVCM), defined as the mass of condensed material on a collector that is maintained at a specific temperature. If there are empty compartments in the system during the test, the CVCM value can be corrected for ambient outgassing by subtracting the CVCM value of the empty compartment. The third property is water vapor regained (further referred to as WVR), defined as the mass the sample has regained after a given time after the test in a controlled environment. All of these values are represented as a percentage of the initial sample mass. The formulas of each of these properties are shown in table 1.

The test apparatus, a drawing of which can be seen in figure 6, is comprised of a specimen compartment, called an effusion cell. The effusion cell has an orifice that is pointed in the direction of a collector plate, used to measure the CVCM value of the sample. The effusion cell and the collector plate are separated by a separator plate, that also has a larger opening in front of the effusion cell orifice. The separator plate is there to limit cross-contamination between adjacent collector plates.

Table 1: Formulas defined by the ASTM E-595 standard. [6]

Property	Formula	Definition
Total Mass Loss	$\frac{S_I - S_F}{S_I} \cdot 100 = TML$ [%]	Where: S_I [g] … initial specimen mass S_F [g] … final specimen mass
Collected Volatile Condensable Material	$\frac{C_I - C_F}{C_I} \cdot 100 = CVCM$ [%]	Where: C_I [g] … initial collector mass C_F [g] … final collector mass
Water Vapor Regained	$\frac{S_F{}' - S_F}{S_I} \cdot 100 = WVR$ [%]	Where: C_I [g] - initial collector mass C_F [g] - final collector mass C_F' [g] - reconditioned mass of specimen after 24 h at 50% relative humidity

The test procedure starts by preconditioning the samples in a controlled environment. This is to ensure repeatability of the test. The ambient temperature during the preconditioning is 23 °C and the relative humidity is 50 %. The preconditioning lasts 24 hours. The sample is then weighted using a balance with a 1 μg sensitivity. The sample is then placed in the effusion cell and the system must be brought to a vacuum of at least $7 \cdot 10^{-3}$ Pa within the next hour. Within

the first hour of pump-down the collector temperature of 25 °C must be reached and subsequently held during the whole procedure. Once the required level of vacuum is reached, the sample compartment is heated to 125 °C within one hour. The status of the apparatus is then maintained for 24 hours. After 24 hours, a vent valve is opened and the apparatus is filled with clean and dry nitrogen gas, to a pressure of 2 to 4 psi, to rapidly cool the heating bars. Once the heater bar has reached a temperature below 50 °C, the pure nitrogen atmosphere is brought to room pressure and the test chamber is opened. The collector plates are weighed immediately after the test chamber is opened. The sample is immediately placed in a desiccator (figure 5) using active silica gel desiccant. Once the sample has reached room temperature, but no later than 30 minutes, the sample is removed from the desiccator and immediately weighted. Optionally, the sample can be placed back into the preconditioning chamber for 24 hours, to determine the WVR value. The procedure is summarized in a flowchart in figure 4.

The required values of the test apparatus properties like temperature and pressure with their respective tolerances are in table 2.

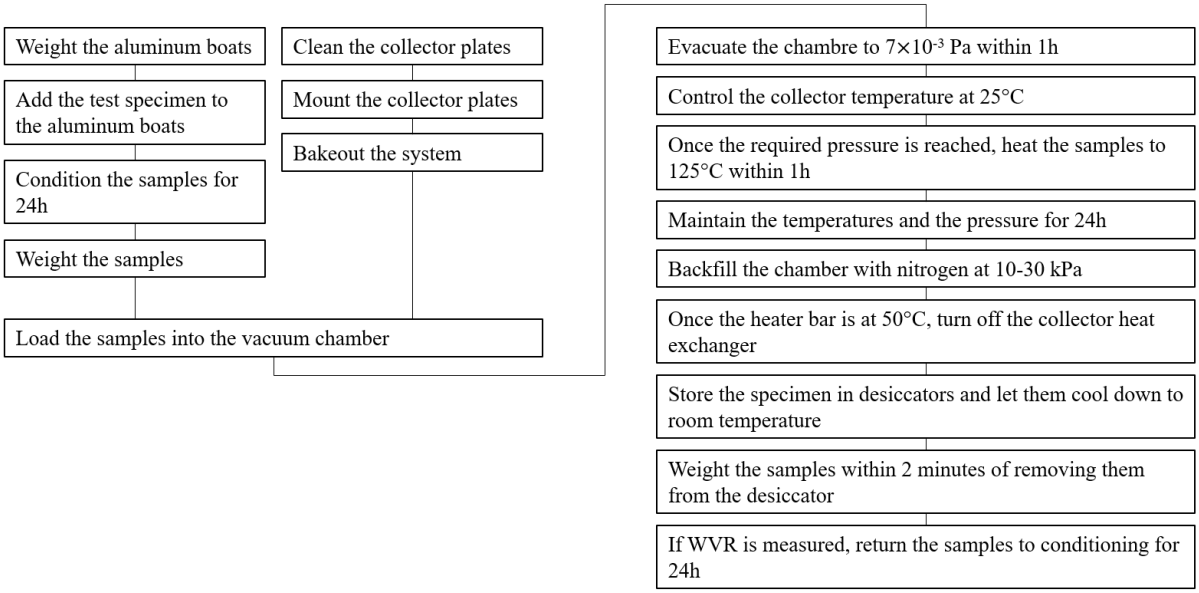

Figure 4: Flowchart of the test procedure.

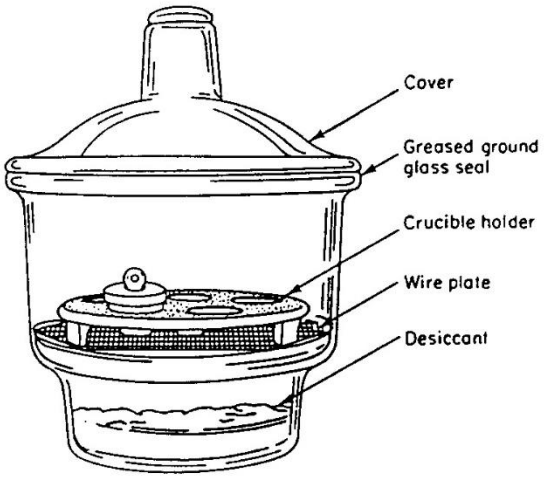

Figure 5: A desiccator. [12]

Table 2: Measured physical properties during the test and their corresponding values. [6]

Physical property	Units	Required value
Boat temperature	°C	125 ± 1
Collector temperature	°C	25 ± 1
Vacuum during test	Pa	$< 7 \cdot 10^{-3}$
Precondition room temperature	°C	23 ± 2
Precondition room relative humidity	%	50
Sample mass	mg	100-300
Microbalance resolution	g	10^{-6}
TML recommended acceptance limit	%	1.0
CVCM recommended acceptance limit	%	0.1

Aside from the described conditions, some key dimensions of the test apparatus are also prescribed. These mainly relate to the dimensions of the effusion cell, the collector plate, and the openings connecting these features. Some of the dimensions that are concerned are shown in figure 6, the specific values of these dimensions can be found in the ASTM standard.

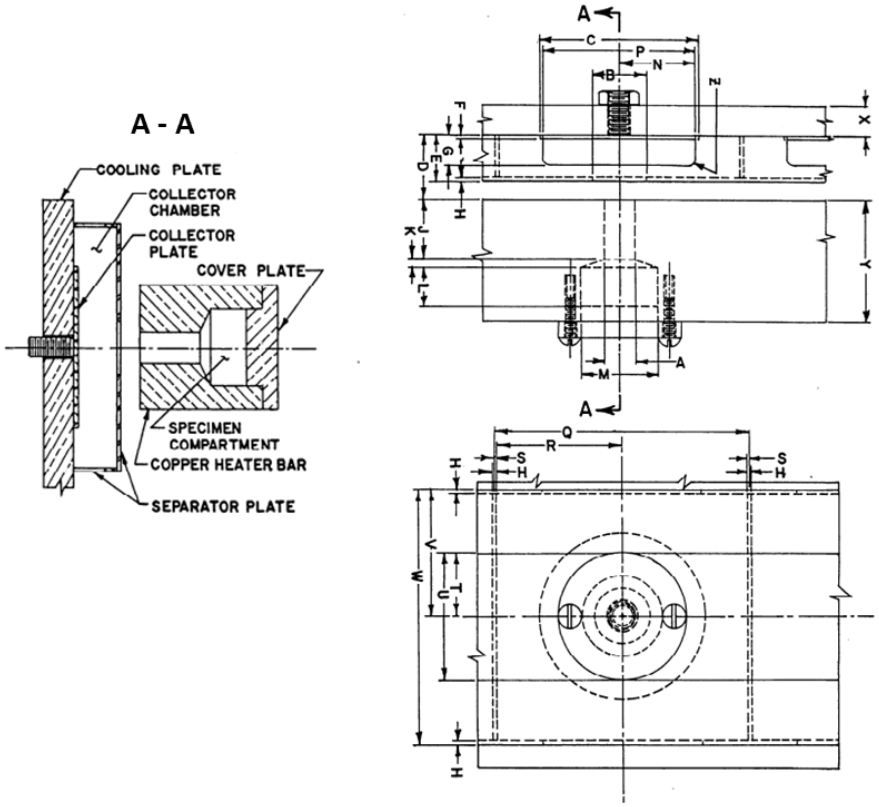

Figure 6: A drawing of the standardized part of the testing apparatus. [6]

Because the standard has been in place since 1977, an extensive database of tested materials exists already [7]. To give an example of usually obtainable values for this test, a selection of materials can be seen in table 3 below.

Table 3: Example values for different materials for TML, CVCM and WVR. [7]

Material	TML [%]	CVCM [%]	WVR [%]
Fiberglass reinforced, ceramic filled, high TG epoxy resin composite	0.49	0.01	0.25
[1]WINDFORM LX 2.0	0.42	0.00	0.16
Velcro hi-gard hooks stainless steel	0.02	0.01	-
Nylon 6 polyamide	3.40	0.17	-

[1]*Glass fiber filled composite material polyamide based. Selective Laser Sintering*

1.2. ECSS-Q-ST-70-02C

This standard was created after the ASTM standard and was therefore made very similar to allow an easy way to certify for both standards at the same time, and not to unnecessarily create separate test data from a slightly different test method. This resemblance can be seen at first glance from the flowcharts describing the sequencing of the test in the document (figure 7).

Because of the similarities, the whole test method will not be repeated here. Only additional representations, descriptions, or requirements will be noted.

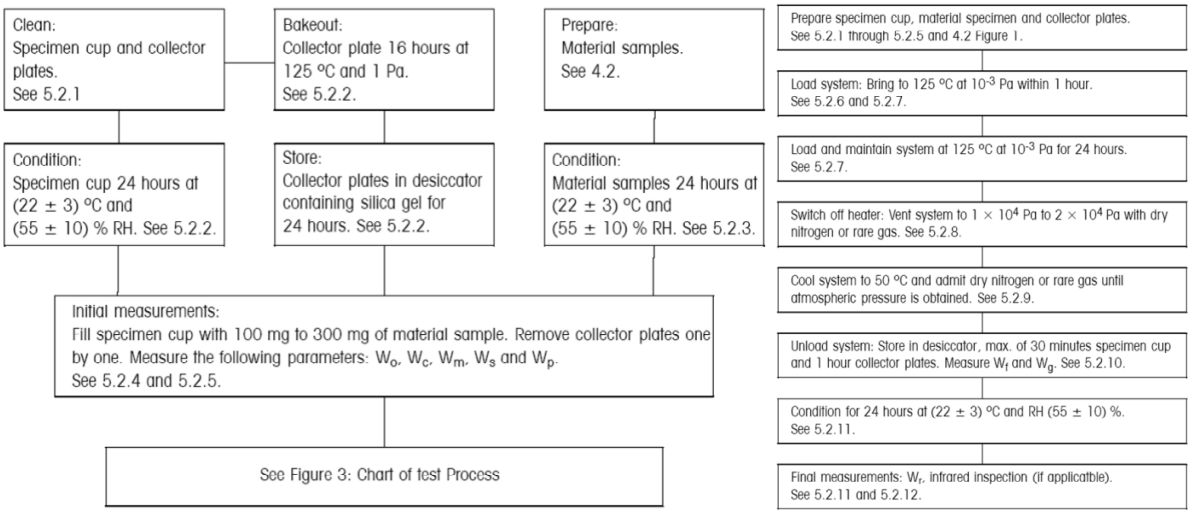

Figure 7: Flow chart of the preparation (left) and of the actual test procedure (right). [11]

The measured values are TML, CVCM, and WVR, the same as in the ASTM standard. Then there is a new calculated value *Recovered Mass Loss* (further referred to as RML), that can be calculated as $RML = TML - WVR$. All the measured values are illustrated in figure 8.

This standard prescribes the required precisions of the measuring devices used during the test. The thermometers in both the vacuum chamber and the preconditioning chamber must be capable of measuring the temperature from 10 °C to 130 °C with an accuracy of ± 1 °C. The humidity sensors must be capable of measuring from 40 % to 80 % relative humidity with an accuracy of ± 1 %. The vacuum sensor must be capable of measuring at 10^{-4} Pa with an accuracy of ± 10 %. As for the microbalance, it must have a resolution of at least 10^{-6} g.

The dimensions of the sample compartment and the collector plate are prescribed and can be seen in figure 9. Also prescribed are the properties of the test apparatus during the test procedure and their values. To ensure the compatibility of this test data with test data from the ASTM standard, the values are very similar. They can be seen in table 4.

The basic representation of how the outgassed particles behave can be seen in figure 10. A portion of the particles will condense on the collector plate while the rest will leave to the vacuum pump. It can also be seen from this schema, that the cup with the sample doesn't have to point towards the cell orifice.

Table 4: Measured physical properties during the test and their corresponding values. [11]

Physical property	Units	Required value
Boat temperature	°C	125
Collector temperature	°C	25
Vacuum during test	Pa	10^{-3}
Precondition room temperature	°C	22 ± 3
Precondition room relative humidity	%	55 ± 10
Sample mass	mg	100-300
Microbalance resolution	g	10^{-6}
TML recommended acceptance limit	%	1.0
CVCM recommended acceptance limit	%	0.1

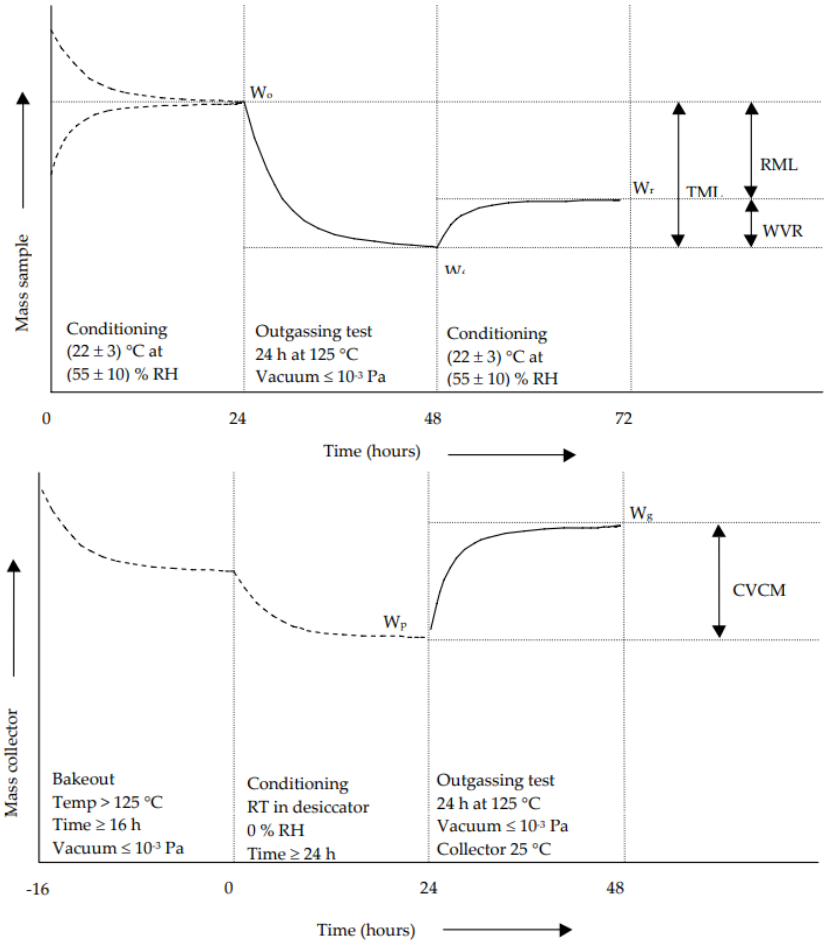

Figure 8: Representation of the different measured values in the test. [11]

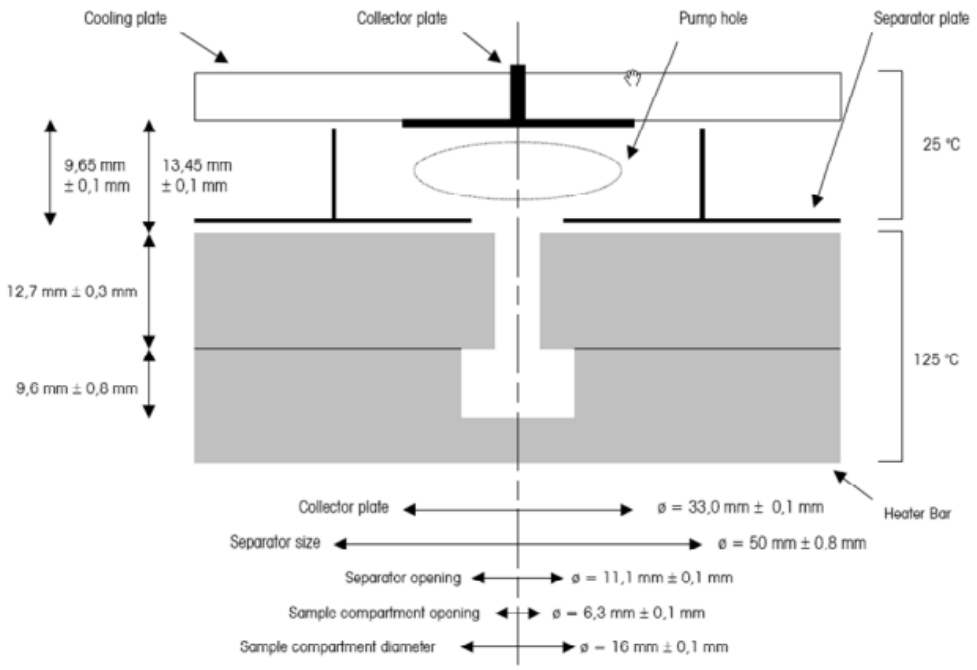

Figure 9: A schematic drawing of the required dimensions in the test apparatus. [11]

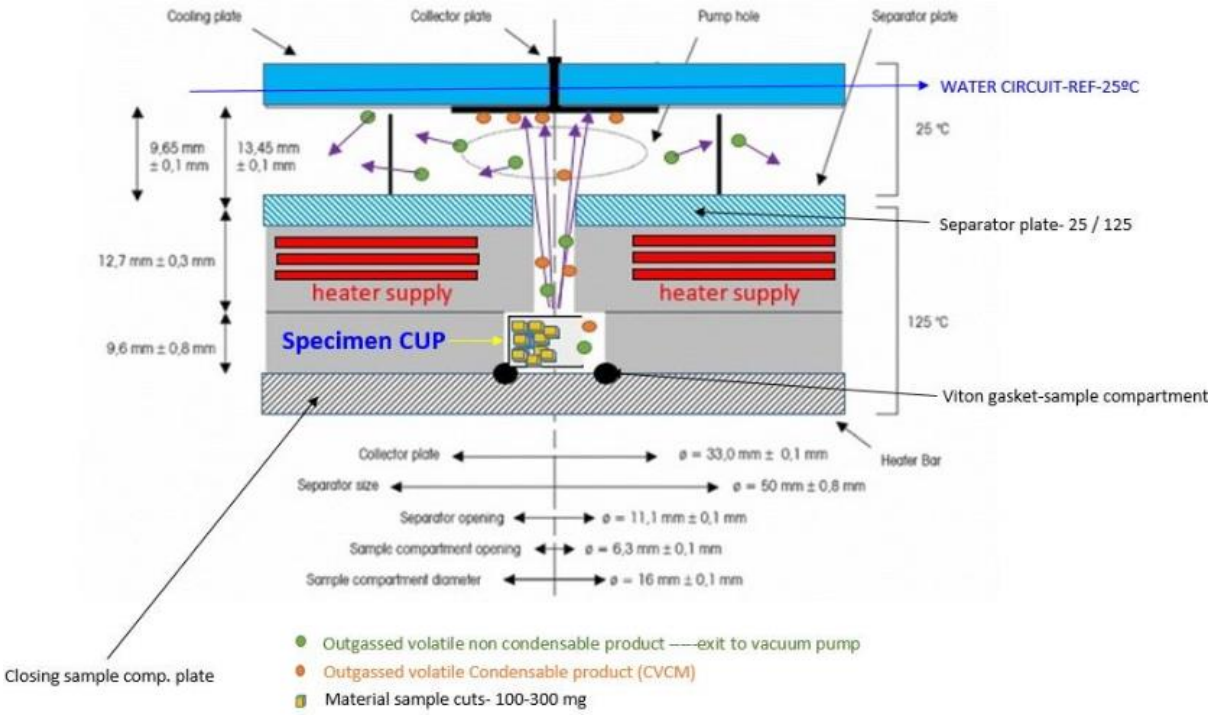

Figure 10: A schematic drawing of the test apparatus with outgassed particles representation. [2]

1.3. ASTM E-1559

This is a newer standard developed with the aim of being able to evaluate the outgassing properties in time and with varying temperature. This is done, so as to be able to accurately simulate the deposition mechanics on different components with varying temperatures. Unlike the previous methods, this test method measures only one sample at a time. The measured values are TML (same as in the tests before) and the evolution of mass in time of multiple collectors, all at varying temperatures. To be able measurement of these properties, the collectors are Quark Crystal Micro balances (QCM). Because the collectors are not the same dimensions and temperature as the one in the ASTM E-595, the results of CVCM cannot be compared with the results of this test.

Same as in the preceding ASTM E-595 test, the material sample is placed in an effusion cell, that is temperature controlled. By placing the orifice of the cell in the direction of the QCMs, the outgassed material will condense on them (figure 11).

The initial isothermal outgassing test is run three times at three different temperatures of the sample. The first two are 398 K (125.85 °C) and 348 K (75.85 °C) respectively. The thirst test is run at either 323 K (55.85 °C) or 373 K (105.85 °C), depending on the results of the first two tests. This is explained in more detail in the standard itself. The QCMs during this test are at 90 K (-182.15 °C), 160 K (-115.15 °C) and 298 K (25.85 °C).

After this test, a QCM thermogravimetric analysis (QTGA) is done. This is done by slowly heating the QCMs from their base temperatures up to 398 K in order to evaporate the collected species. Since every species has a different evaporation characteristic, they will leave the collectors at different temperatures. This helps better characterize the outgassing flux which helps better model this process in simulations. A QTGA test is also a good way to clean the QCM.

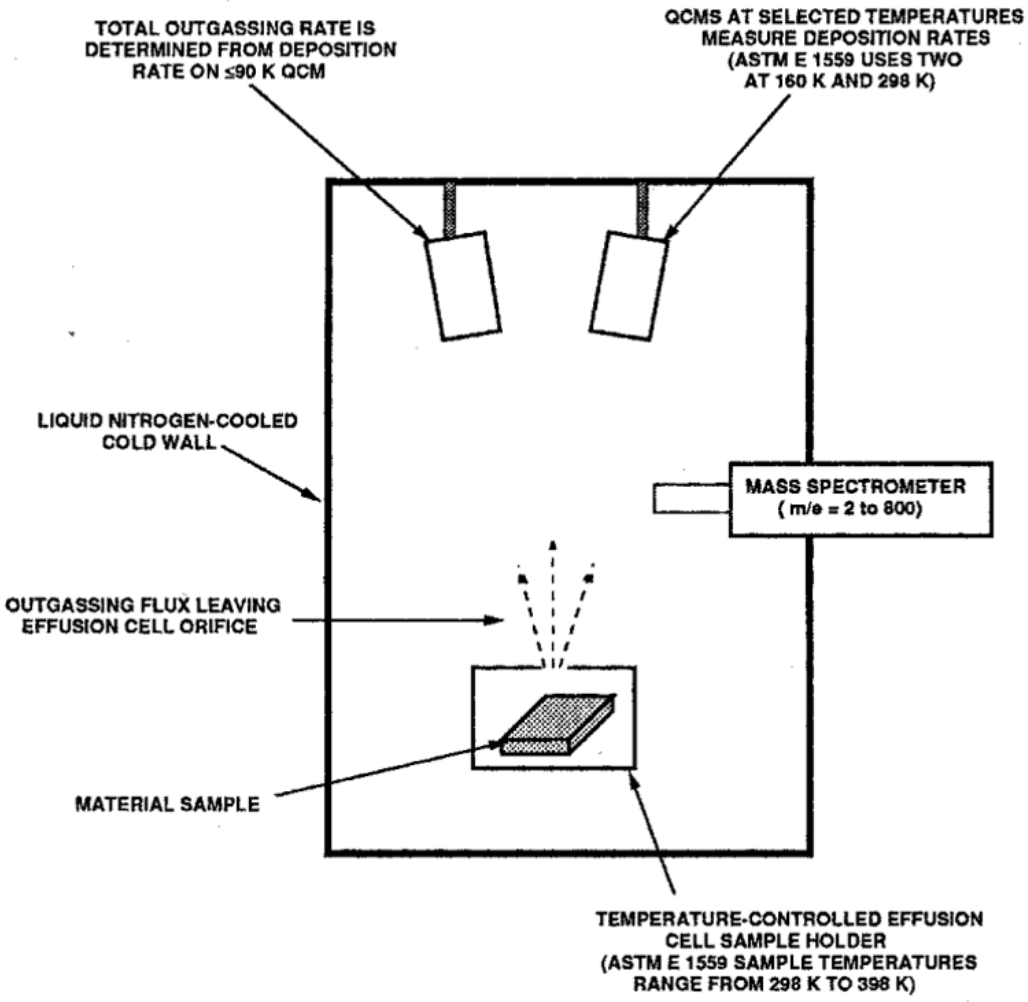

Figure 11: Schematic of the QCM measurement method. [8]

As a model test apparatus, this standard uses a Lockheed device, which was used during the development of this testing method (figure 12). The standard requires that at least three QCMs are present, each consisting of two crystals (one for mass collection and one for reference). The required sensitivity is at least 10-8 g/cm² Hz at 298 K. To minimize the molecular contamination from sources other than the effusion cell, a nitrogen cooled shroud is used around the walls of the apparatus. Additionally, this test uses bigger samples than the original ASTM E-595 method. The samples are around 30 grams, so the balance accordingly requires less precision. The required precision of the balance is ± 10μg.

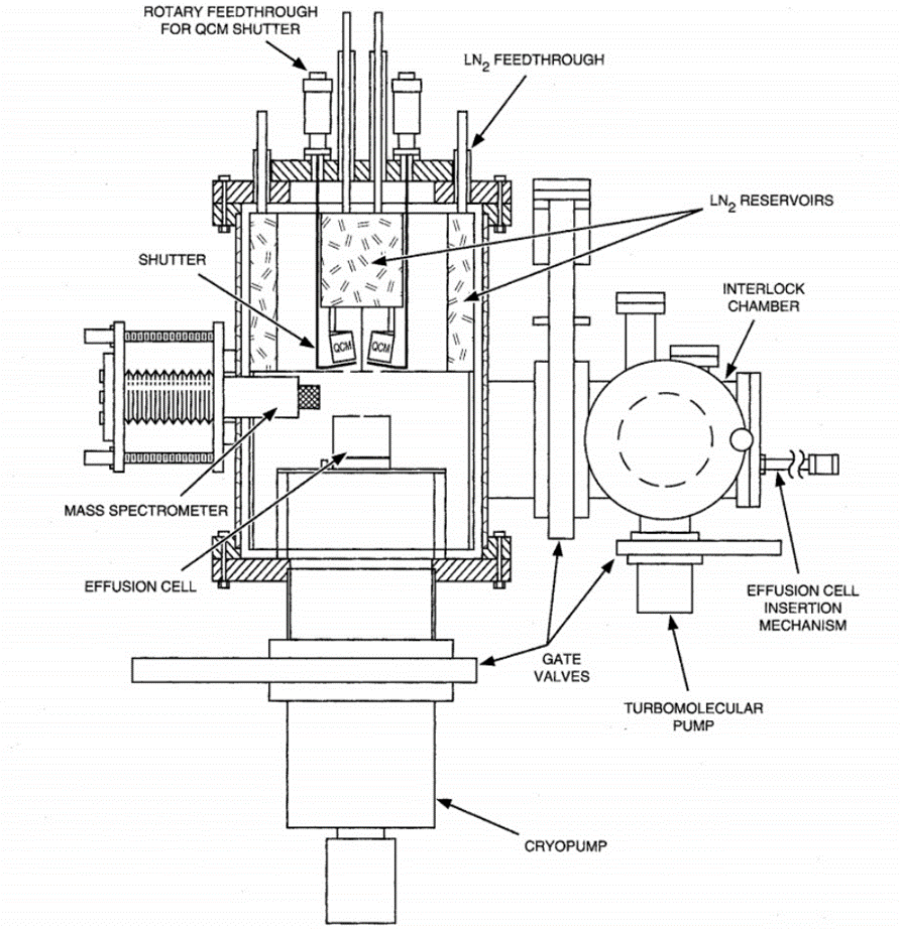

Figure 12: Lockheed vacuum outgassing kinetics apparatus. [8]

1.4. ECSS-Q-TM-70-52A

This document is not a standard. It is a technical memorandum, therefore the information in this document is non-normative and is only provided as additional and possibly useful information for people interested in outgassing testing and modelling. The paper includes a quick description of everything from the physics of outgassing and testing methods through to available modelling tools. But most importantly, the paper describes two additional methods used for measuring the outgassing and deposition rate, the CNES method and the ESTEC method.

1.4.1. CNES method

The CNES method is interesting in that it uses a vacuum microbalance to measure the TML value directly rather than indirectly as it is done in ASTM E-1559. However, this test does not measure the CVCM value or any other deposition characteristics of the material. An image of the testing apparatus can be seen in figure 13.

Four tests are performed on each material, three of which consist of raising the sample temperature from ambient up to 125 °C during 12, 18 and 30 hours subsequently, while during the fourth test, the sample temperature increases by a step of 25 °C every 24 hours, until it reaches 125 °C. An example of the resulting data is in figure 14.

Figure 13: Overview of the CNES test apparatus. [1]

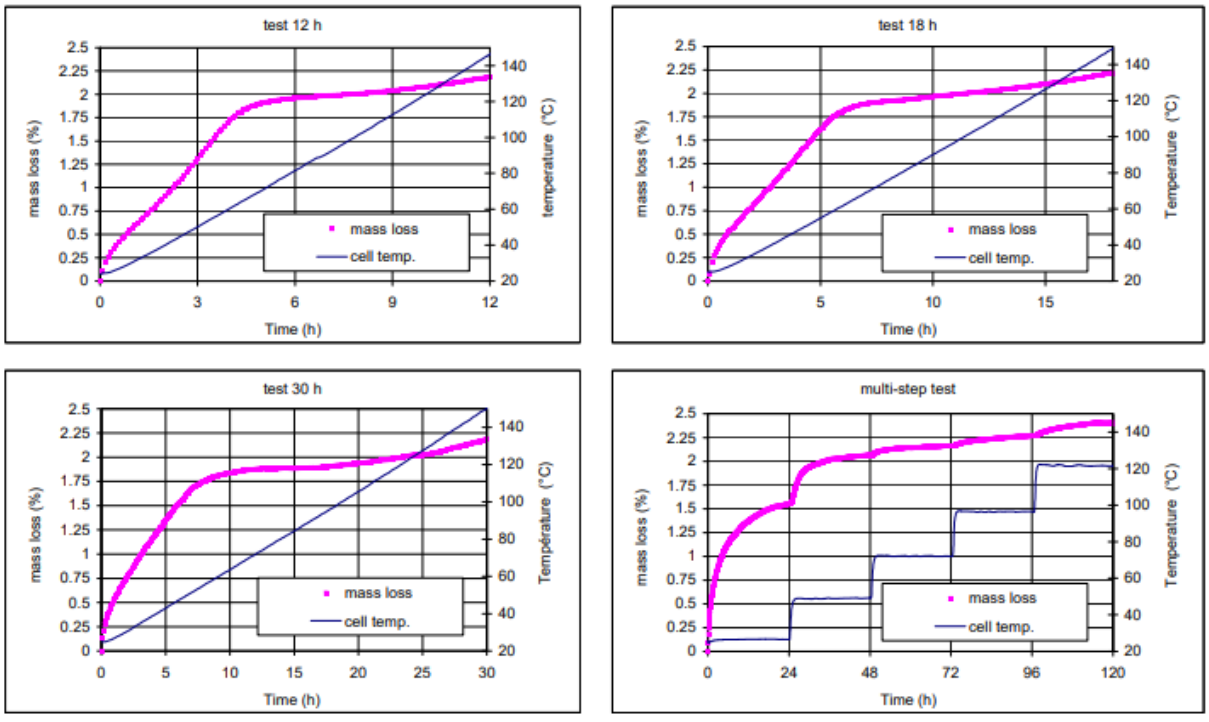

Figure 14: CNES outgassing test for HYSOL EA 9321. [1]

1.4.2. ESTEC method

This method uses two different apparatuses, the VBQC (Vacuum Balance Quartz Crystal) and the DOK (Dynamic Outgassing Knudsen cell). Both systems use QCMs to measure the CVCM of a sample, while the TML is measured either by another QCM (for the DOK) or by a vacuum microbalance (for the VBQC). The standard test procedure is the same as the fourth test of the CNES system. The sample temperature is increased every 24 hours by 25 °C until it reaches 125 °C.

Both systems are in principle similar to the system used in ASTM E-1559, as the CVCM is measured directly using three QCMs. The difference is that in the ASTM test, TML is measured indirectly by the CVCM value and the initial and final weight of the sample, while in the VBQC and DOK tests, TML is measured directly.

In the VBQC system, the sample is heated by radiating panels along the chamber walls. Additionally, this system does not use and interlock chamber, as is required by the ASTM standard, thus making it noncertifiable (figure 15). The DOK system on the other hand has been built in accordance with the ASTM standard, so it has an interlock system and the effusion cell in which the sample is placed provides the heat to the sample (figure 16).

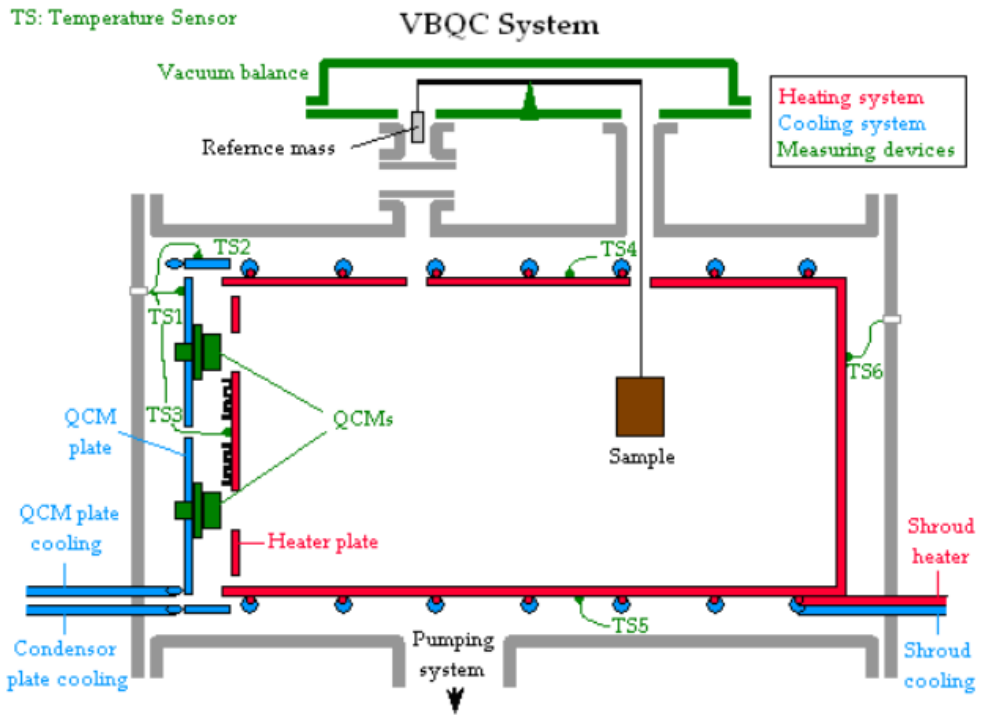

Figure 15: VBQC system schematics. [1]

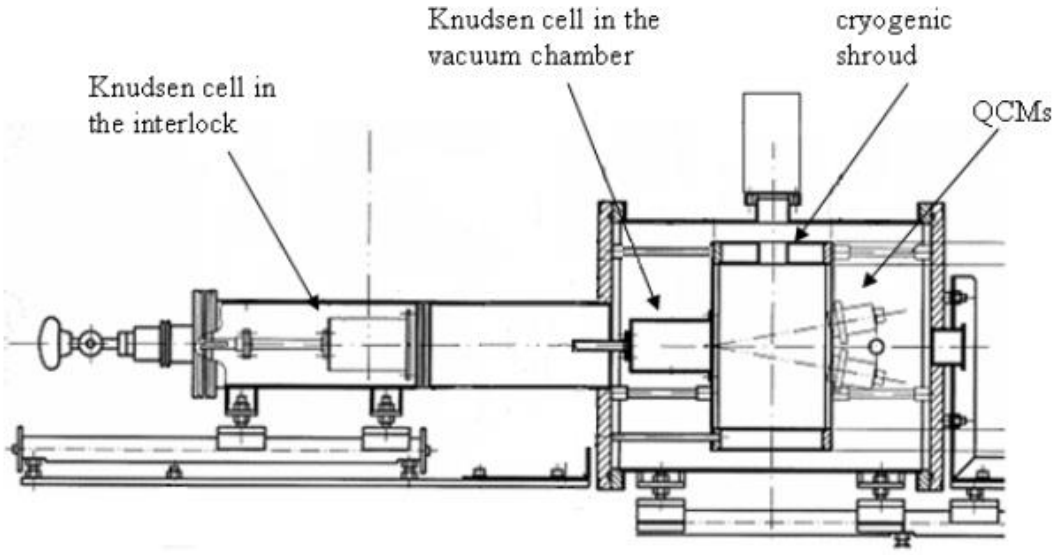

Figure 16: DOK system schematics. [1]

1.5. Overview of standards and their differences

There are 3 standards and 1 technical memorandum that apply to outgassing in space applications. An overview of basic information regarding these documents is in table 5. Even though there are 4 documents in total, they only describe two separate test procedures, as two of them are American and two are European. The first set of standards, ASTM E-595 and ECSS-Q-ST-70-02C, came out in 1977 and 1985 respectively. The other two documents, ASTM E-1559 and ECSS-Q-TM-70-52A, came out much later in 2000 and in 2011 respectively. The most up-to-date versions are ASTM E-595-15R21 from 2021, ECSS-Q-ST-70-02C from 2008, ASTM E-1559-09E22 from 2022, and ECSS-Q-TM-70-52A from 2011.

Table 5: A summary of the 4 main outgassing documents.

	ASTM E-595	**ECSS-Q-ST-70-02C**	**ASTM E-1559**	**ECSS-Q-TM-70-52A**
Measured properties	TML, CVCM, WVR	TML, CVCM, WVR, RML	Kinetic outgassing	Kinetic outgassing
Ordered by	NASA	ESA	NASA	ESA
First version release year	1977	1985 (under different regulation body called ESA-PSS-01-702	2000	2011
Last update	2021	2008	2022	2011
Reference	[6]	[11]	[8]	[1]
	Subject of this thesis		More advanced testing	

The ASTM E-595 and the ECSS-Q-ST-70-02C are almost identical in both the scope and the requirements of the testing system. They both measure multiple samples at once, while simultaneously measuring one or two empty effusion cells to correct the CVCM result for the ambient outgassing of the apparatus. The procedure of the test is identical, the samples are preconditioned for 24 hours, tested in vacuum for 24 hours, and then optionally conditioned for another 24 hours.

In the parameters of the test, there are some minor differences. The maximum pressure required is lower in the ECSS standard. The conditions during the preconditioning are slightly different as well. The temperature sees a 1 °C difference in the median value, but both are within the tolerances prescribed by the other standard. The relative humidity also sees a 5 % difference in value, but while the ASTM standard does not give an interval field for this value, the ECSS does, and its interval includes the required ASTM value. The rest of the test parameters, including the base geometry of the effusion cell and the collector plate are identical between these two standards. A comprehensive overview can be seen in table 6.

Table 6: An overview of the test parameters comparing the ASTM and ECSS standards.

Physical property	Units	ASTM E-595	ECSS-Q-ST-70-02C
Boat temperature	°C	125 ± 1	125
Collector temperature	°C	25 ± 1	25
Vacuum during test	Pa	$< 7 \cdot 10^{-3}$	10^{-3}
Precondition room temperature	°C	23 ± 2	22 ± 3
Precondition room relative humidity	%	50	55 ± 10
Sample mass	mg	100-300	100-300
TML recommended acceptance limit	%	1.0	1.0
CVCM recommended acceptance limit	%	0.1	0.1

The ASTM E-1559 cannot be easily compared to the two original standards, as the test is very different. It only measures one sample at a time, and the result is not a single number describing the mass loss and deposition after 24 hours, rather it is a discrete dataset that describes the mass loss and deposition with time, sample temperature, and collector temperature.

To be able to measure these properties in time, these devices use a QCM (Quark Crystal Microbalances) as their collectors of the outgassed material. By monitoring their frequencies during the test, they are able to accurately measure the mass deposition characteristics of the sample. The mass loss in time can then be measured indirectly, by measuring the initial and the

final mass of the sample. However, some devices use a special microbalance to measure the mass loss directly.

Since this thesis aims to create a conceptual design of a device measuring the TML, CVCM, and WVR values, thus conforming to the two original standards, the ASTM E-1559 standard along with the ECSS-Q-TM-70-52A technical memorandum serve only as an inspiration for some of the technical solutions used in their measurement devices.

2. Existing measurement apparatuses

Most, if not all existing devices used for measuring either TML and CVCM or kinetic outgassing are operated by private companies. This unfortunately means that only very limited information is available about these devices, usually only photos. Because of this, this chapter will mostly look at top level design features, like sample orientation, number of samples and the design of the vacuum chamber.

Exceptions are the few test apparatuses that have been used to develop the standards. The standards usually provide at least a simple conceptual schematic of these devices.

2.1. TML and CVCM measurement

The requirements for the layout of the apparatus defined in the two standards are very loose, only some of the most crucial dimensions regarding the effusion cell, collector plate and the path between them are defined precisely. Because of this looseness, there are great differences in the existing devices used around the world.

The first apparatus with which the original ASTM E-595 standard was developed, is a conceptually simple device, that uses a glass vacuum bell, in which the apparatus is mounted (figure 18). A total of 24 samples can be tested at once, meaning there are 24 effusion cells and collector plates. The effusion cells are mounted with the orifice pointing horizontally. The collector plates are chromium plated and are all towards the center of the device to minimize the volume which must be cooled. The cooling of the collector plates is done using a heat exchanger. The heating of the effusion cells is done by a resistance heated copper bar. All weighting of the collector plates and samples is done outside of the apparatus on a microbalance.

A very similar if not the same concept is used by NuSil Technology's outgassing apparatus. The main difference being that it only has one column of stacked effusion cells, thus making the vacuum bell narrower and taller. (figure 17)

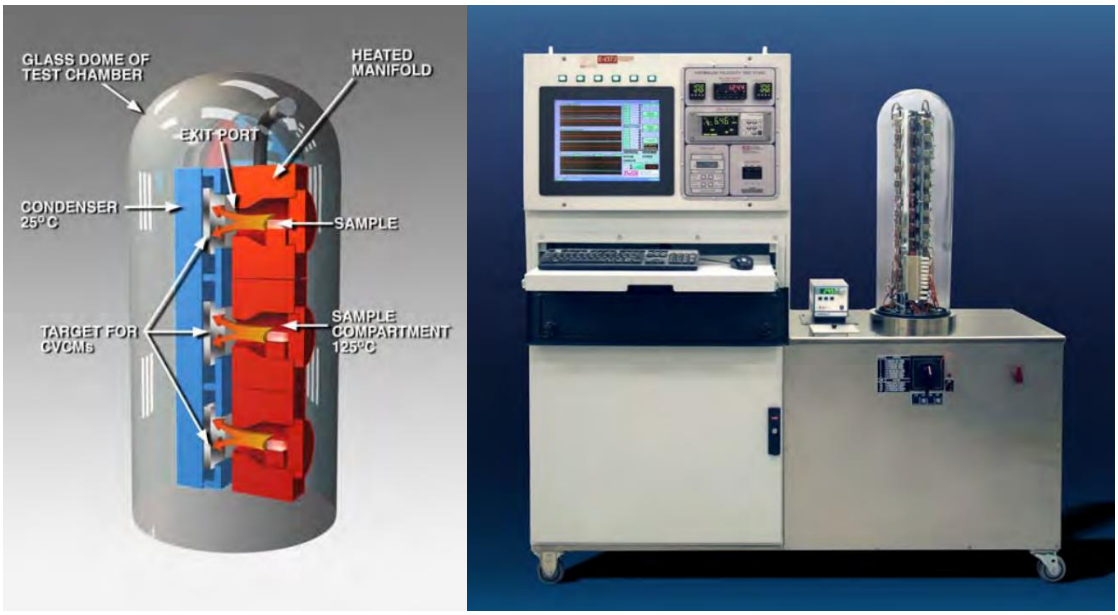

Figure 17: NuSil Technology outgassing apparatus. [13]

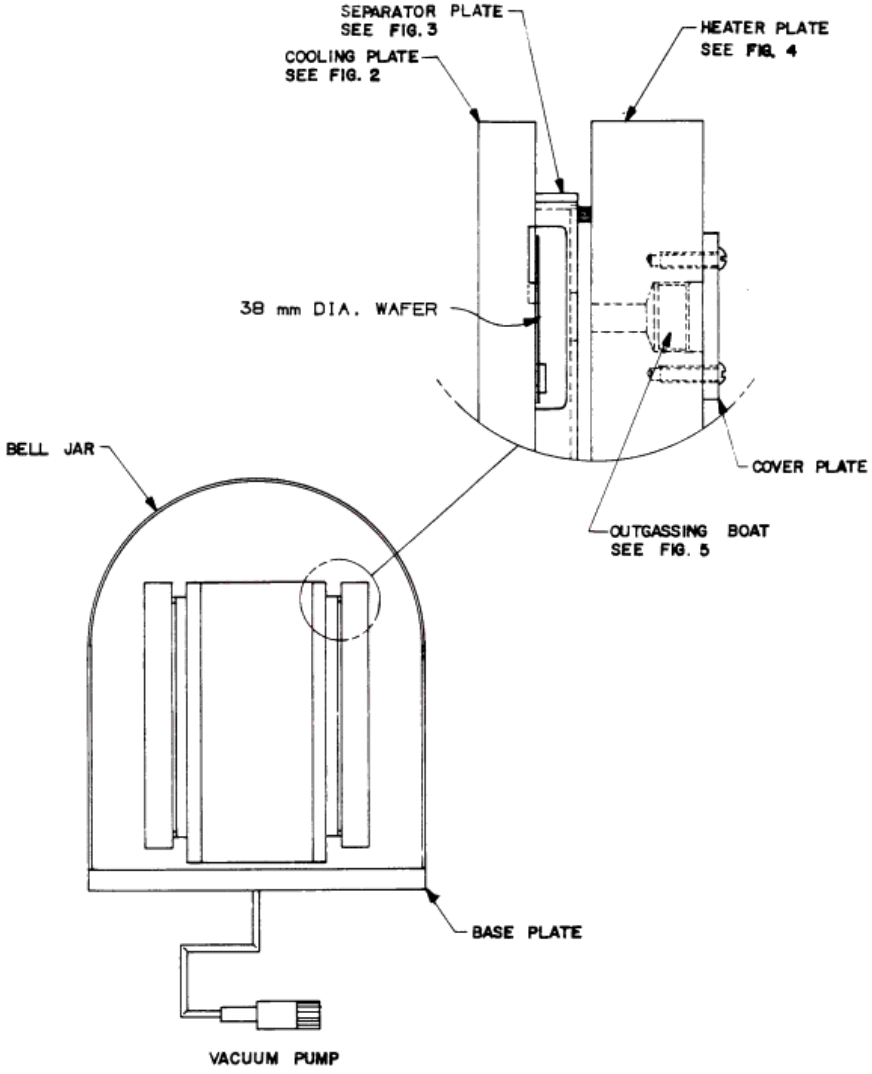

Figure 18: Schematic of the outgassing apparatus used in ASTM F-1227. [14]

A different way to arrange the effusion cells is arranging them horizontally, with the orifice pointing upwards and the collector plates above them. This layout can be seen in figure 19. This is an apparatus used by NYE Lubricants. It only has 6 effusion cells, but that is still enough to test up to two different materials at once. Instead of the glass vacuum bell used in the original standard, this uses a steel cylinder to withstand the atmospheric pressure. The cylinder opens from the top with the collector plate assembly attached to the lid. Additionally, it can be seen that the separator plate is not a thin sheet of metal, but rather a milled piece of aluminum.

There are multiple ways to load the sample into the effusion cell. This can be done either from the orifice side or from the back side of the compartment. In the ASTM standard, it is done from the back, with a cover plate closing the sample inside (figure 6). A different solution can be seen in the figure 19. The sample is first placed on the bottom of the effusion cell and is then closed from the top with a cover plate with the effusion cell orifice. Ergonomically, this seems to be the more comfortable solution, as multiple sample compartments are covered by one cover plate.

To close the effusion cell after the sample is inserted, the cover plate is usually secured by two screws, which is also hinted at in the ASTM standard, however this solution is not required. Instead of the screws, a hold down clamp can be used to hold the cover plate down (figure 20). This is a faster and less tedious solution, especially with a larger number of samples.

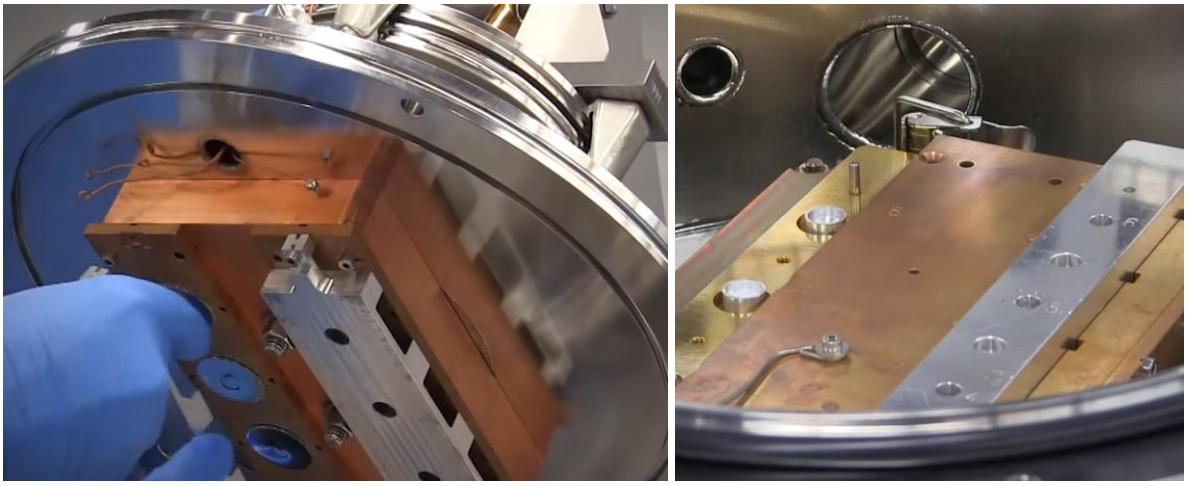

Figure 19: NYE Lubricants outgassing apparatus design. [15]

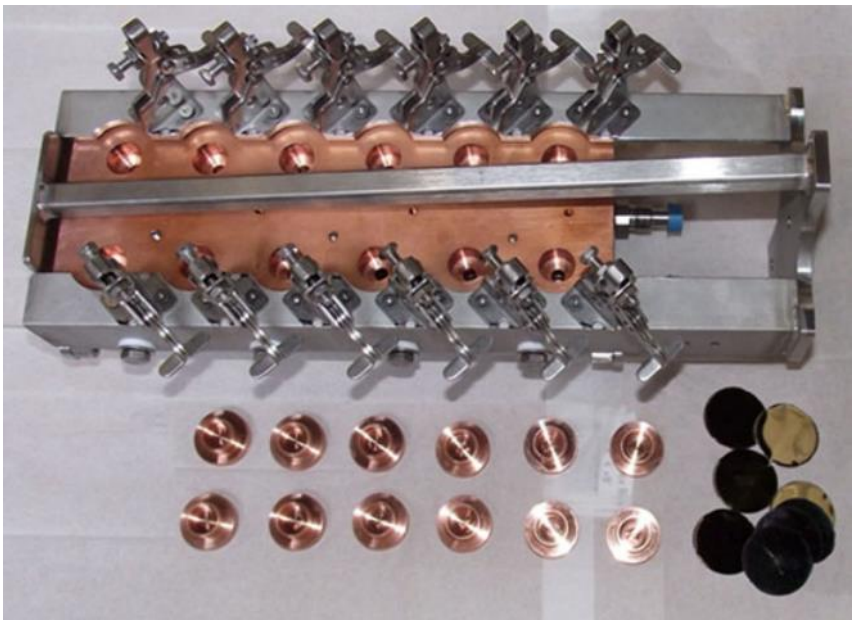

Figure 20: Effusion cell assembly with hold down clamps. [16]

In the INTA outgassing apparatus (figure 21), the sample compartments are arranged circularly, allowing for a better use of space in the vacuum chamber. This apparatus, while having similar vacuum space, can house up to 24 samples while the NYE lubricants can only house 6. What can also be seen, is that the separator plate ring and the effusion cell ring have handles, to permit easier manipulation with these presumably not very light components, especially because they need to be removed to get to collector plates.

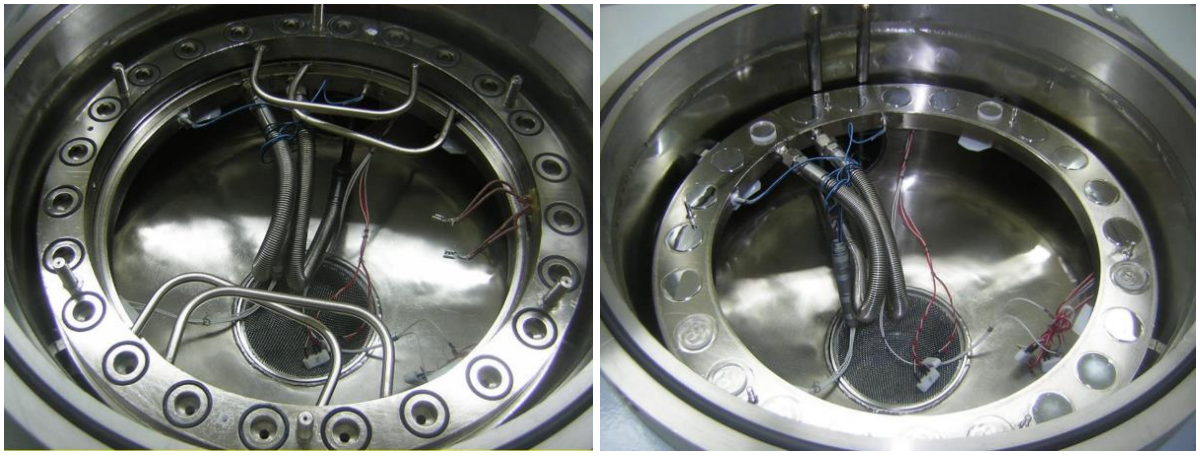

Figure 21: INTA horizontal outgassing apparatus. [2]

Figure 22: Securing mechanism of a collector plate. [2]

In Figure 22 a possible mounting mechanism for the collector plates can be seen. Additionally, this image shows that a part of the collector plate can be covered by the holding mechanism, as long as the uncover area is in accordance with the standards.

Unfortunately, not a lot of information is publicly available about these devices aside from a few photos. However, by the analysis of said photos, a pattern already emerges for vacuum chamber design and effusion cell orientation. This is evident in table 7.

Table 7: Basic design characteristics of each of the testing devices.

	Lockheed Martin [6; 14]	**NuSil Technology [13]**	**NYE Lubricants [15]**	**INTA [2]**
Cell stacking direction	Vertical	Vertical	Horizontal rectangular	Horizontal circular
Vacuum chamber design	Glass bell	Glass bell	Metal cylinder	Metal cylinder
Number of samples	24	12	6	24

2.2. Kinetic outgassing

Testing equipment for kinetic outgassing is even more loosely defined than the testing for TML and CVCM, that said, most of the testing equipment around the world follows the general philosophy of the Lockheed outgassing kinetics apparatus, with which the ASTM E-1559 standard was developed.

The main features of these devices are the use of two chambers with an interlock system between them and the use of QCMs to measure the condensed material mass (figure 23). Usually, these devices are not used to test the classic TML and CVCM values, but some devices have this capability and use the interlock chamber for this (figure 24).

Because this thesis is mainly centered around the classic TML and CVCM measurement, these more complex devices will not be further analyzed, as they refer to a different test set.

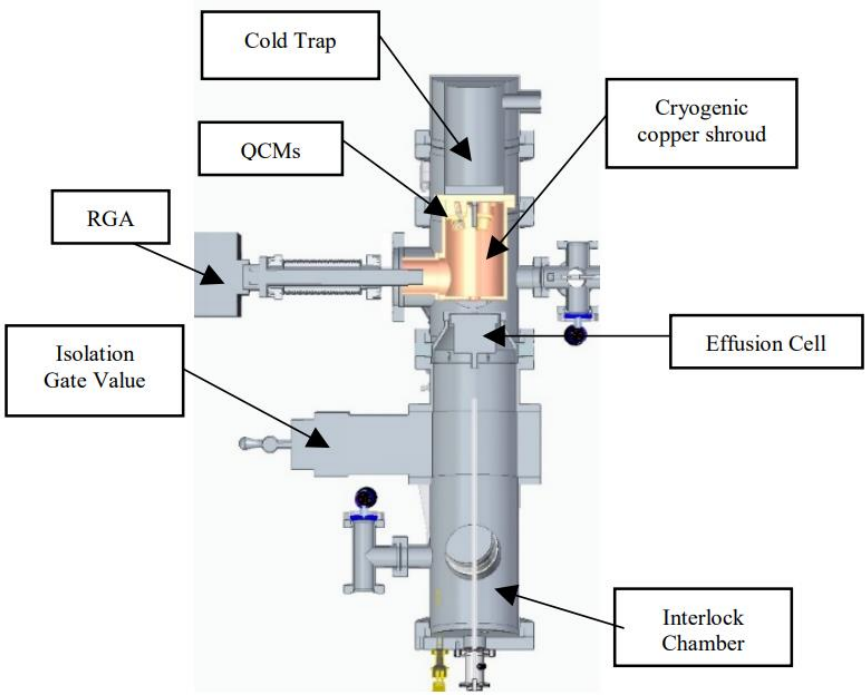

Figure 23: Simplified section view of an outgassing kinetics testing device. [17]

Figure 24: ASTM E-595 and ASTM E-1559 testing device. [16]

3. Aim of the thesis

The aim of this thesis is to propose a design for a outgassing testing apparatus that would fulfil specified requirements. The requirements come from multiple sources. An overview of their sources as well as the structure of this chapter is in figure 25.

Most of the requirements understandably come from the standards, as they define the testing conditions and procedures. Some requirements come from BUT, as there is already some equipment present at their testing facility and to reduce costs, this testing apparatus should make use of these. Finally, some requirements are self-imposed, as they are the result of chapter 2.

Throughout this chapter, each requirement will be presented and at the end, a table will summarize them in a comprehensive way.

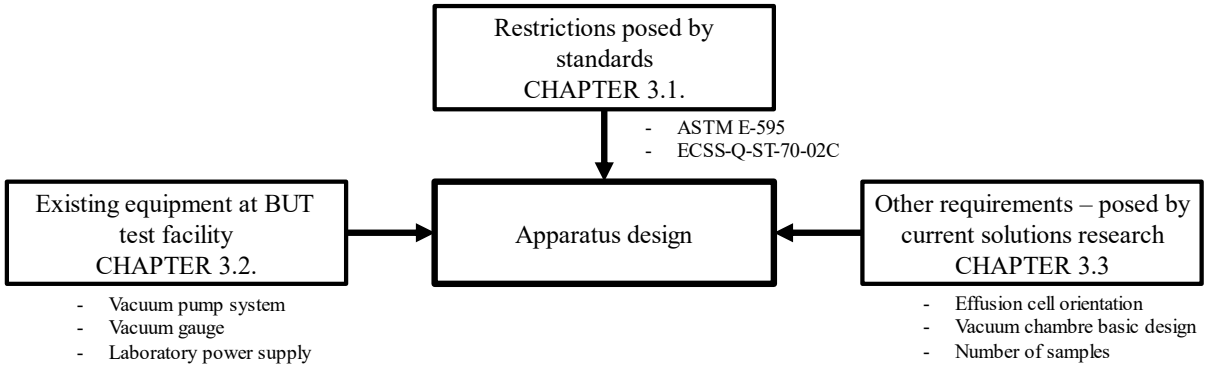

Figure 25: Conceptual design inputs.

3.1. Restrictions posed by standards

As mentioned previously, this device will fall only under the ASTM E-595 and ECSS-Q-ST-70-02C standards, testing only the TML, CVCM and WVR properties. Nonetheless, these two standards impose a multitude of constraints regarding the test conditions, evaluation criteria, and the geometry of the test apparatus.

The required test conditions for the two standards are very similar but differ in a few small details. However, test conditions that are compatible with both standards are possible. Boat temperature must be 125 $\pm$ 1 °C (requirement R1). Collector plate temperature must be 25 $\pm$ 1 °C (requirement R2). Vacuum during the test must be 10^{-3} Pa (requirement R3). Preconditioning room temperature must be 23 $\pm$ 2 °C (requirement R4). Preconditioning room humidity must be 50 $\pm$ 5 % (requirement R5). And sample mass before the test must be 100-300 mg (requirement R6).

Additionally, the device must be capable of monitoring the sample boat and collector plate temperatures during the whole duration of the test (requirement R7).

In addition, the device must incorporate a mechanism to backfill the chamber with pure nitrogen at a pressure ranging from 10 to 30 kPa. This pressure must be regulated so as not to invalidate the results. This is requirement R8.

The standards also dictate certain critical geometrical dimensions like the effusion cell and the collector plate (requirement R9) to ensure the comparability of results across different facilities.

These can be seen in figure 9 in chapter 1.2. with other dimensions being up to the apparatus designer to choose.

As for the evaluation of results, the main restriction is that the balance, with which the samples and the collectors are weighted must have a 1 μg sensitivity. This is requirement R10.

3.2. Existing equipment at BUT

The BUT test facility already has some equipment that is used for other vacuum related tests and that can be leveraged for this test as well. First is a laboratory power supply DPS-3005D (figure 26), that can supply up to 32 V and 5 A [18]. This will be used for the heating system of the effusion cells (requirement R11). Second is a vacuum gauge WRG from Edwards, that can measure pressure from atmospheric to 10^{-7} Pa [19] and will be used to measure the pressure in the vacuum chamber (requirement R12).

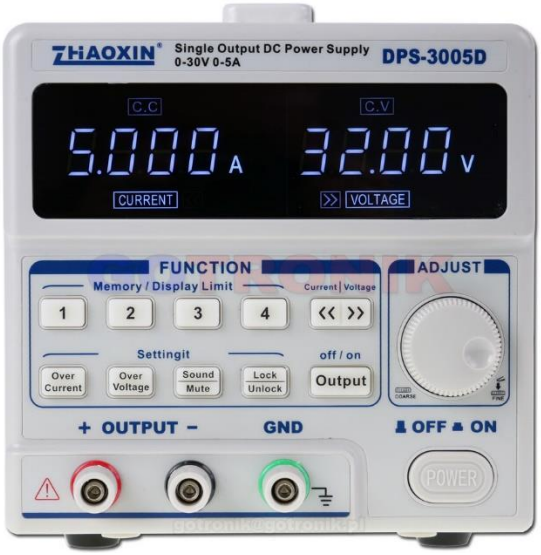

Figure 26: Laboratory power supply DPS-3005D. [18]

Third and last component is a vacuum pump system, that this system will use to create vacuum in the chamber (requirement R13). The vacuum pump system is the Edwards Vacuum nEXT Turbomolecular pumping station. The exact configuration is a EXT75DX turbomolecular pump, capable of up to 66 l/s [20], backed by a nXDS10i backing pump, capable of up to 11.4 m^3/h [21]. While the turbomolecular pump can theoretically reach a vacuum of $5 \cdot 10^{-7}$ Pa, this is in a chamber that has no leakage and no desorption from walls. In real life, the larger the chamber the more leakage and desorption there will be. This implies that a maximum volume exists for which the pump will no longer be able to reach the required pressure of 10^{-3} Pa or will take more than 60 minutes to pump down. This maximum volume is requirement R14.

3.2.1. Maximum chamber volume determination

To aid the calculation of the volume, an online calculator from Pffeifer Vacuum [22]. Because the online calculator only allows for systems from Pffeifer Vacuum, an equivalent system to the one at BUT was chosen for the calculation. The basic characteristics of the two systems are in table 8. A turbomolecular pump HiPace 80 Neo was chosen as an equivalent to the EXT75DX turbomolecular pump, as it has a pumping speed of 67 l/s [23] compared to the 66 l/s of the Edwards System. For the backing pump, the Duo 11 M was chosen, having a pumping speed

of 10.5 m^3/h [24] compared to the 11.4 m^3/h of the Edwards System. An overview of the characteristics in in table 8, while an overlay of the pumping characteristics based on chamber pressure of the two systems used for the calculation is in figure 27.

As mentioned before, the system will necessarily have some leaking and some desorption from the walls. Because these values are not yet determined for the final chamber, they have to be chosen from expected values for this type of chamber. As for desorption, this system will be baked before every test, so this value is very low. For baked systems, the desorption is usually between $5 \cdot 10^{-10}$ and $1 \cdot 10^{-11}$ mbar·l/(s·cm^2) [22]. To remain on the save side, the value of $5 \cdot 10^{-10}$ mbar·l/(s·cm^2) was used. For leakage, the value for high vacuum chambers is usually between $7 \cdot 10^{-8}$ and $2 \cdot 10^{-10}$ mbar·l/s [22]. To remain on the save side, the value of $7 \cdot 10^{-8}$ mbar·l/s was chosen.

The results of the pump down time and minimum pressure reached are in figure 28. The first limit reached is not the pressure, rather the pump down time. It can be seen that a chamber of 1400 liters is dangerously close to the 60 minutes limit. Because some of the vacuum volume is taken up by the pipes connecting the vacuum pump and the chamber, and to remain on the save side, the requirement R12 will reflect a maximum chamber volume of 1200 liters.

Table 8: Equivalent pump system specifications.

		Edwards Vacuum System (at BUT) [20; 21]	**Pffeifer Vacuum system [23; 24]**
Backing pump	Minimum pressure [Pa]	1	0,3
	Pumping speed [m^3/h]	11,4	10,5
Turbomolecular pump	Maximum pressure [Pa]	80	50
	Minimum pressure [Pa]	$5 \cdot 10^{-7}$	$1 \cdot 10^{-5}$
	Pumping speed [l/s]	66	67

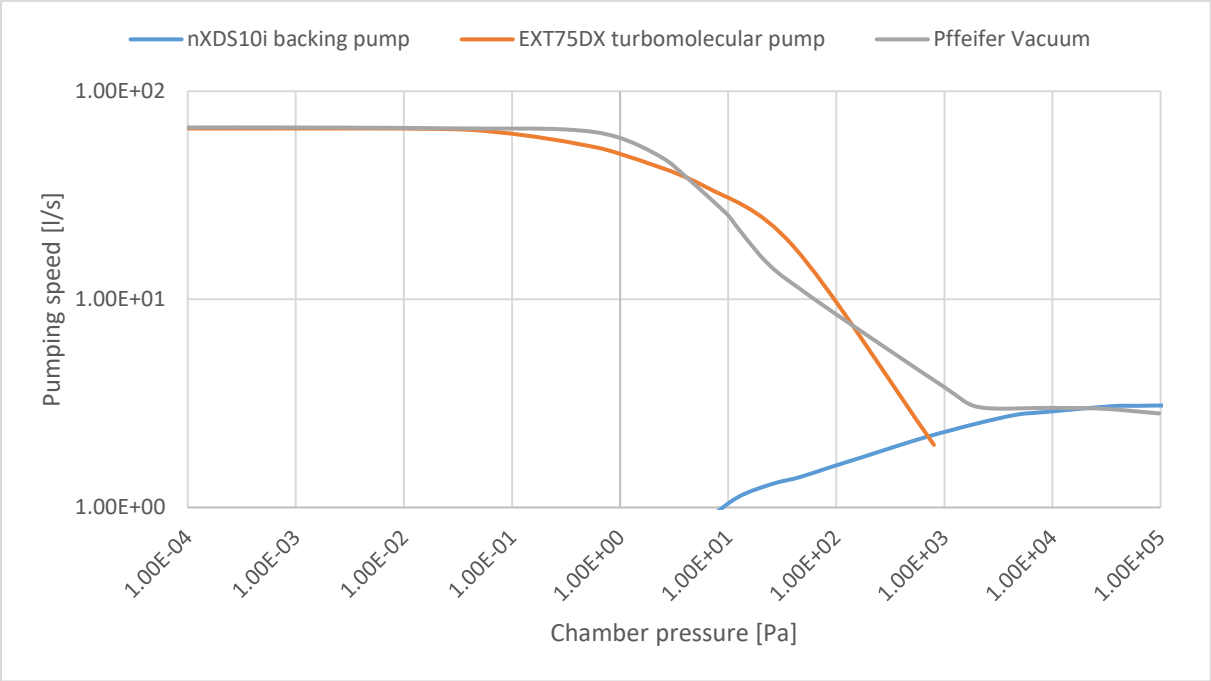

Figure 27: Vacuum system pumping speed vs. chamber pressure. [20; 21; 22]

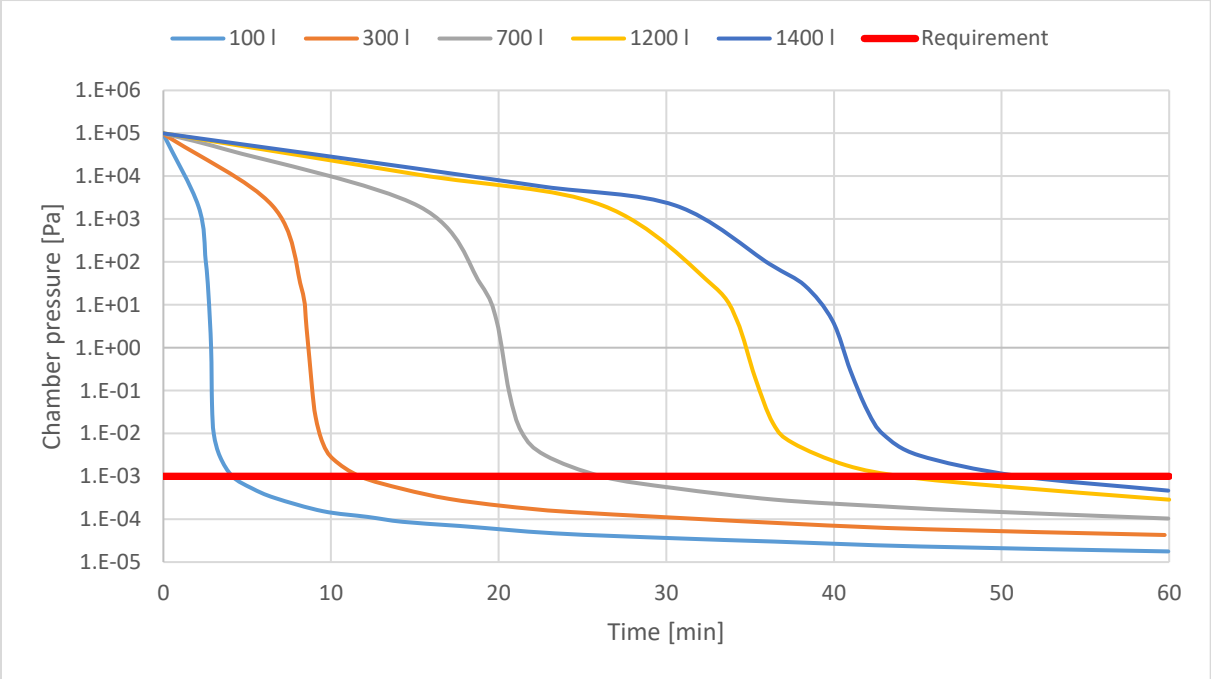

Figure 28: Pump down time for different chamber volumes overlayed over the required chamber pressure. [22]

3.3. Other requirements

The final set of requirements are self-imposed as they are the result of the chapter. From the research in chapter 2, the choice of the vacuum chamber is between two basic philosophies. One option is using a glass bell as part of the vacuum chamber and the second option is using a full metal encloser.

The disadvantage of the glass vacuum bell is that it has to be fully removed to get to the apparatus. This can be quite an undertaking as the glass bell gets taller and heavier the more samples there are. The steel cylinder has this problem mitigated by using one side of the chamber as a lid that can be opened and closed. The design in this thesis will thus use a metal vacuum chamber. This is requirement R15.

From table 7, it is apparent, that there is a correlation between the vacuum chamber design and the effusion cell stacking orientation. While it is possible to use a metal vacuum chamber and stack the effusion cells vertically, most designs stack the cells horizontally as it is more practical. The design in this thesis will not break this pattern and also use horizontal layout. This is requirement R16.

Again, from table 7, it can be seen that the number of samples ranges anywhere from 6 to 24. Having 6 samples allows only for very limited testing, where to have the best possible outcome, only one material can be tested in a configuration of 4 samples and 2 empty effusion cells for correction. Alternatively, two materials can be measured, with 2 samples of each. However, having only two samples of any given material can lead to statistical errors.

On the other range of the spectrum, having 24 effusion cells is an overkill for BUT, as this allows for testing of up to 7 different materials with 3 samples each and with 3 empty cells for correction.

The ability to test 3 or 4 different materials at once is enough for this thesis. The lowest number of effusion cells that this can be achieved with is 10, which allows for configurations of 1 empty cell and 3 materials in 3 cells each or of 2 empty cells and 4 materials in 2 cells each. This means that having at least 10 effusion cells is requirement R17.

3.4. Requirements overview

This chapter has summarized all the requirements, the final design will have to fulfil to be considered a success. All the requirements are written out in table 9. Most of the requirements are imposed from the two standards talking about TML testing. These requirements (R1 – R10) touch on anything from effusion cell geometry to sample preparation and test conditions. Requirements R11 to R14 come from the existing equipment already used at BUT and with which this test apparatus must be compatible to save costs. The last set of requirements (R12 – R14) comes from lessons learned from existing solutions. These speak mainly to vacuum chamber material design and to effusion cell orientation.

Table 9: List of requirements.

	Requirement	Description
Restrictions posed by standards	R1 – Boat temperature	The apparatus must achieve and maintain the sample boat temperature of 125 °C.
	R2 – Collector temperature	The apparatus must achieve and maintain the collector temperature of 25 °C.
	R3 – Vacuum during test	The apparatus must achieve and maintain the chamber pressure at below 10^{-3} Pa.
	R4 – Precondition room temperature	A conditioning room/chamber must be able to achieve and maintain its temperature at 23 °C.
	R5 – Precondition room relative humidity	A conditioning room/chamber must be able to achieve and maintain its humidity at 50 %.
	R6 – Sample mass	The measured material sample must have a total mass between 100 and 300 mg before the test.
	R7 – Temperature monitoring	The apparatus must be able to monitor the temperature of the effusion cell and the collector plate in real time.
	R8 – Nitrogen backfill	The apparatus must be able to backfill at a regulated pressure of 10 to 30 kPa with pure nitrogen.
	R9 – Critical apparatus dimensions	The effusion cell and collector plate geometries as well as their distances are dictated by the standards.
	R10 – Microbalance	The microbalance for evaluation of results must have a sensitivity of less than or equal to 1 μg.
Existing equipment at BUT	R11 – Existing laboratory power supply	The effusion cell heating system must be compatible with a laboratory power supply already present at BUT.
	R12 – Existing vacuum gauge	The chamber pressure must be monitored using a vacuum gauge already present at BUT.
	R13 – Existing vacuum pump system interface	The test apparatus must be designed to interface with the vacuum pump system already present at BUT.
	R14 – Maximum volume	The test apparatus must have a volume smaller than 1200 liters.
Other requirements	R15 – Vacuum chamber material	The main material of the vacuum chamber must be metal.
	R16 – Effusion cell stacking direction	The stacking direction of the effusion cells must be horizontal.
	R17 – Number of effusion cells	The number of effusion cells must be at least 10.

4. Conceptual design

With the requirements from the previous chapter, multiple conceptual designs can be crafted. Because the design must comply with those requirements, these designs will mostly differ in criteria not covered by the requirements. This is mostly the physical layout of the effusion cells and resulting design solutions and limitations.

Proposed designs will be evaluated based on 5 different criteria, each with a different weight, ranging from 1 to 3. The overview of these criteria is in table 11. For each criterium, the designs can earn from 1 to 3 points. The guidelines to the rating are in table 10. This will result in a final score for each design. The design with the highest score will be chosen.

Table 10: Rating guidelines for the conceptual designs.

Rating	Description
★ ★ ★	This solution has no inherent disadvantages.
★ ★ ☆	This solution is not ideal but can be used. The problems created stay constant with the increasing size of the test apparatus (more samples).
★ ☆ ☆	This solution either presents serious problems or is worsening with the increasing size of the apparatus (more samples).

The first criterion and the most important one is the **accessibility of the effusion cells**. This means not only how many steps have to be taken to be able to open the chamber, load the samples and close the chamber again. This also means that once the chamber is open, how accessible the effusion cells are for the loading process. Because this process is undertaken two times during each test, this clearly has a weight of 3.

The second criterion, **cleanness of the loading and unloading process**, is how big a chance there is that during the loading or unloading of samples, any part of the inner apparatus will be contaminated. Because the cleanness of the inner apparatus is critical to the test procedure, and because the apparatus is opened twice during each test, this criterion also has a weight of 3.

To be able to fulfil the requirements R1 and R2, the inner apparatus must have a connection to the outside of the chamber. The **technical feasibility of outside connections** is the third criterion. While this will only be delt with once during the design phase, it is still an important criterion to consider, as designing something more complicated unnecessarily can result in a more expensive solution or a solution that is more prone to failure. Because of that, this has a weight of 2.

The fourth criterion is the **ability of the apparatus to be dismantled and manually cleaned**. This is important if a particularly high outgassing material was tested and left a lot of residuals or if, for example, an oil was tested. Because this does not have to be performed after every test, this has a weight of 2.

The last criterion is the **vacuum chamber volume efficiency**. While an important thing to consider during design, the maximum volume of requirement R11 is rather big. Therefore, this criterion only has a weight of 1.

Table 11: Conceptual design evaluation criteria overview.

Criteria	Weight	Description
Effusion cell accessibility	3	The ease of access to the effusion cells for loading and unloading of samples.
Cleanness of the loading and unloading process	3	Risk of contamination during loading and unloading of samples.
Outside connections technical feasibility	2	Technical feasibility of electrical cables and coolant pipes outside connections.
Easy to clean (dismantlability)	2	Ability of the apparatus to be dismantled to allow an easy way to clean any residuals.
Chamber volume efficiency (for 10 samples)	1	Wasted vacuum space in the vacuum chamber.

4.1. Rectangular layout

The first possible layout is using a rectangular, 2-by-5, effusion cell layout. To save space and not use an unnecessarily large vacuum chamber, the test apparatus is loaded into the chamber from its side. This means that the test apparatus must slide into the chamber already closed as shown in figure 29.

This has the advantage of allowing the operator to load the samples outside of the chamber. This means that the effusion cells are more accessible, and the process is easier. This also allows for easier manual cleaning of the inner if it is required. However manual cleaning of the vacuum chamber itself is more challenging, especially if a design with more than 10 samples is introduced.

Having the apparatus outside the chamber also has its disadvantages. When loading the samples, none of the parts of the apparatus can touch any outside surface, as it would contaminate them (before loading samples, the apparatus is baked in the vacuum chamber). This can however be easily solved with the proper procedures.

Another disadvantage is that the cables connecting the heating and the coolant must be able to slide with the apparatus as well, while their connection to the outside of the chamber is fixed. So, some rollup mechanism for these would have to be designed. This mechanism would be more and more complicated with the increasing number of effusion cells.

The lid of the vacuum chamber will either rotate out, as shown or alternatively, the vacuum chamber lid could be attached to the rails and would slide back and forth with the test apparatus. The fact that the lid is not opened vertically has its advantages. The force necessary is much lower, as the operator does not have to lift the whole weight of the lid.

The vacuum chamber itself can be either a cylindrical design, as that is the best shape to withstand the atmospheric pressure, or it could also be a rectangular design, as that would provide the best use of space inside the chamber and would facilitate the mounting of the test apparatus.

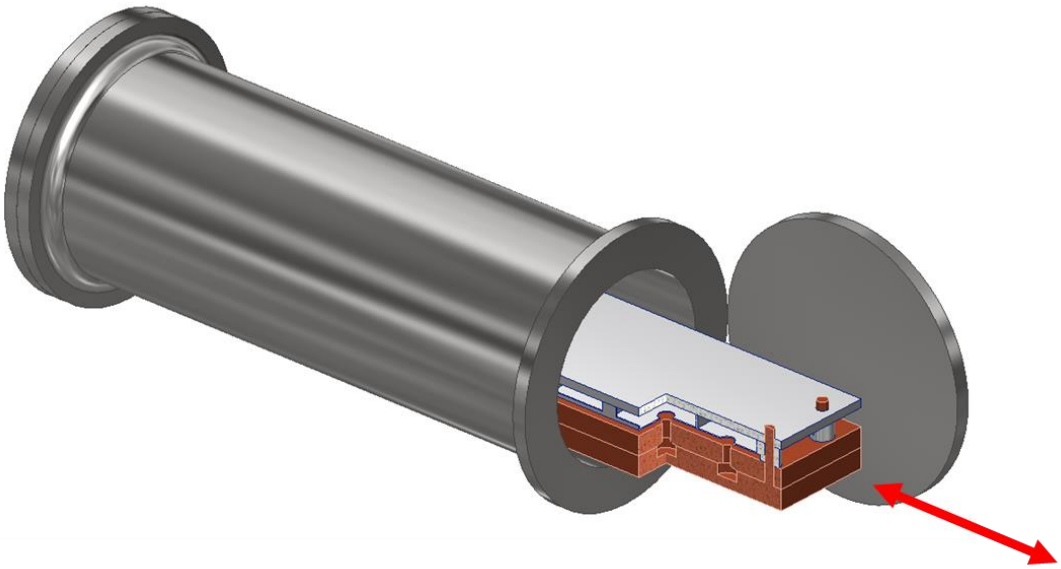

Figure 29: A rectangular layout in a tube-like design.

4.2. Circular layout

The second possible layout is a circular one, that would see the effusion cells in a one cell wide circle around the whole vacuum chamber. The chamber is opened from the top (figure 30), as this would be the best use of space. Unfortunately, because the effusion cells are around the wall of the chamber, the more effusion cells there are, the more wasted space in the center there is.

While the inner apparatus will not slide out, but rather rotate up similar to the chamber lid, this design is still easily accessible for the loading and unloading of samples. Because when opened, the apparatus will not be able to touch any outside surfaces, the whole loading process is clean.

The opening of the apparatus also allows for easy manual cleaning if necessary. An advantage is that unlike the rectangular design, this vacuum chamber design does not create any difficulties with manual cleaning of the chamber itself.

This design also does not introduce problems with outside connections. That's because the inner apparatus will only rotate, so if the connections are at the place of the pivot, they too, will only have to pivot, unaffected by how large the apparatus is.

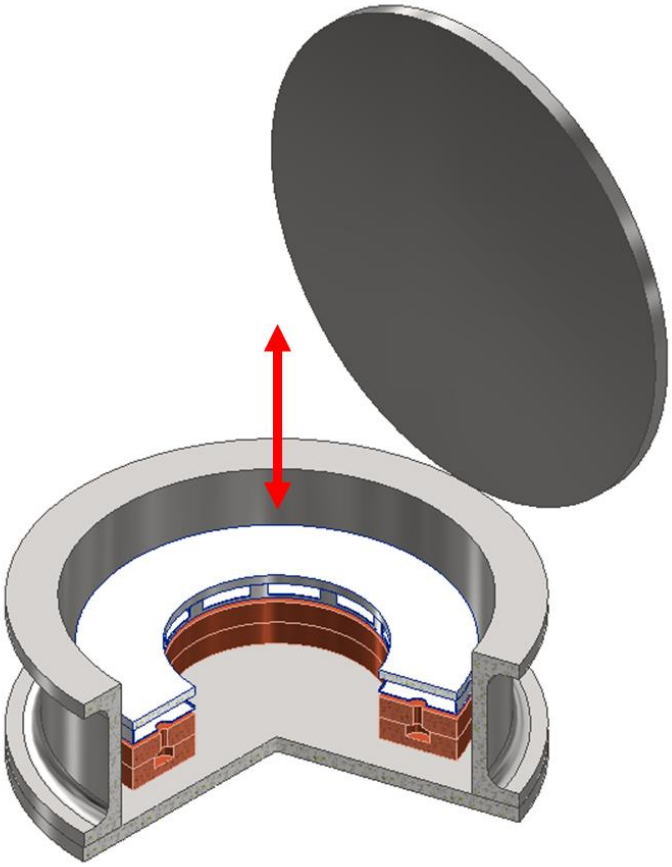

Figure 30: A circular layout in a cylindrical vacuum chamber.

4.3. Comparison

When comparing the two designs with one another, there are some criteria that both are very good at, and either one would be a good choice. These are namely the effusion cell accessibility and, where both designs are easily accessible, and the loading process inherently flowed in any way. This means that both designs have scored maximum points in the two most important criteria.

Where things start to differ are the lower tier criteria. The rectangular design has a big problem with the technical feasibility of the coolant and heating connections to the inner apparatus and additionally this chamber design is difficult to clean because it is very narrow. For this reason, in both criteria, the rectangular layout has only scored one point. The circular layout does not have any serious problems in any of the two criteria and has scored 3 and 2 points respectively.

For the last criterion, the chamber volume efficiency, the rectangular layout has scored all 3 points, as it can be very compact, even if more effusion cells are added. This cannot be said about the circular layout, as it can become very inefficient if more effusion cells are added. However, because the maximum volume is very high, this is a criterion with the lowest weight.

The points and the final score for each layout are in table 12. The circular layout has been found to be better and will therefore be used in the final design.

Table 12: Concept design criteria rating.

Criteria	Weight	Rectangular layout	Circular layout
Effusion cell accessibility	3	★ ★ ★	★ ★ ★
Cleanness of the loading and unloading process	3	★ ★ ★	★ ★ ★
Outside connections technical feasibility	2	★ ☆ ☆	★ ★ ★
Easy to clean (dismantlability)	2	★ ☆ ☆	★ ★ ☆
Chamber volume efficiency	1	★ ★ ★	★ ☆ ☆
Total score (higher is better)		25	29

5. System specifications

With the conceptual layout chosen, it is now high time to determine what specific components will be used to fulfill all the requirements like effusion cell heating, collector plate cooling, temperature monitoring and so on. The schematic overview of all systems and connections along with the relevant chapters are in figure 31. External devices used in the test, like microbalance and conditioning room, will also be mentioned in this chapter.

The output of this chapter will therefore be a list of all the components that have to be incorporated into the final design, both the vacuum chamber and the inner apparatus.

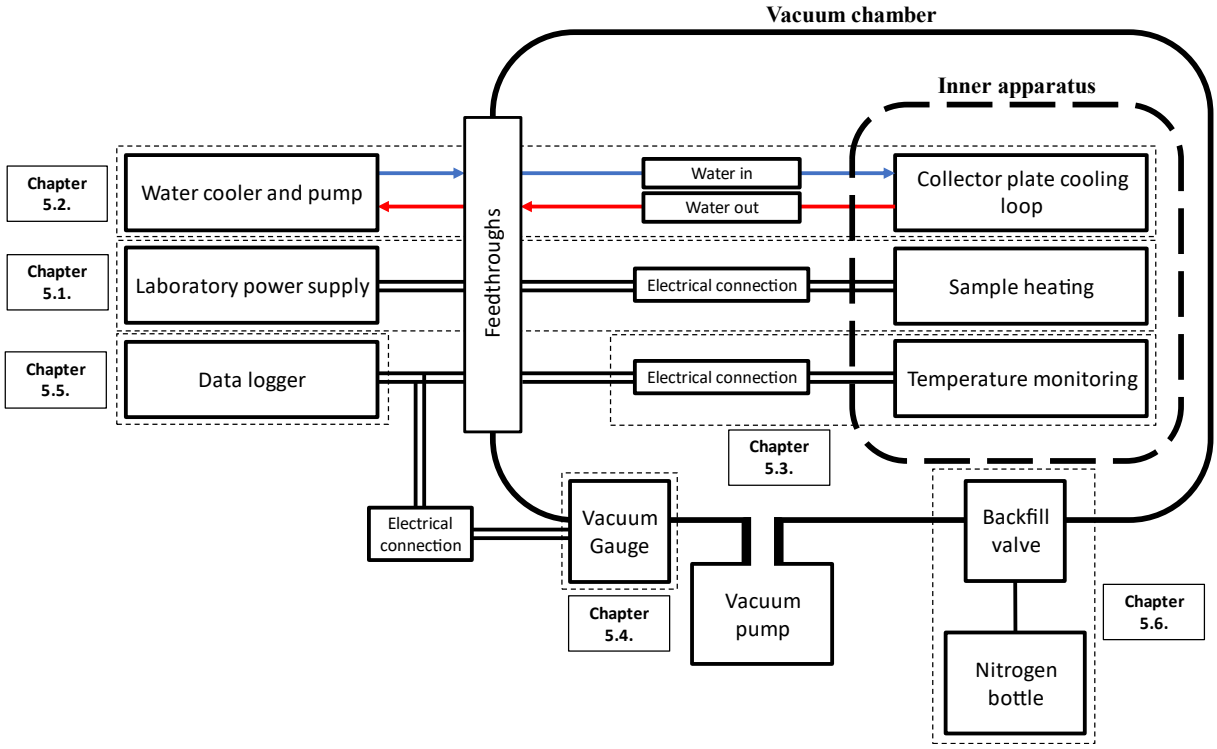

Figure 31: Schematic overview of system connections.

5.1. Effusion cell heating

The bottom part of the apparatus, in which the sample is placed during the test, must be heated to 125 °C, as required by the standards (requirement R1). A cross section of the heated part of the system is in figure 32.

This part will be entirely made from copper, as it is a great heat conductor with thermal conductivity of 398 $W \cdot m^{-1} \cdot K^{-1}$ [25] and it has a low specific heat capacity of 0.385 $J \cdot g^{-1} \cdot K^{-1}$ [26]. This means that the part will heat up quicker to the desired temperature and the heat will quickly spread to all of the part, ensuring an even heat up process.

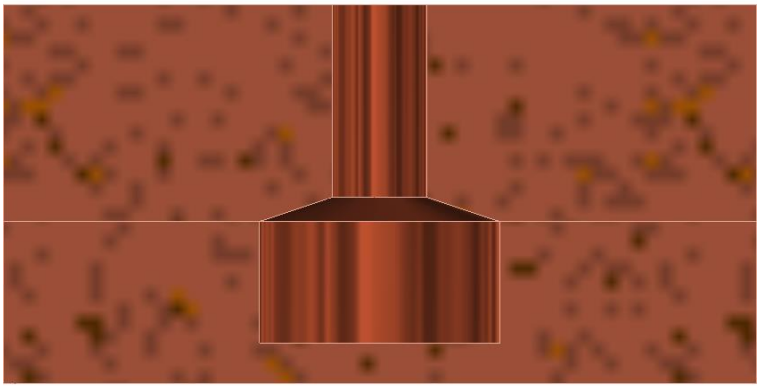

Figure 32: Effusion cell – heated part.

From a preliminary CAD design with room for 13 samples, the total volume of the effusion cells is approximately 1 000 cm^3. Since this part of the apparatus will be made out of copper, the weight is approximately 2 700 g, as seen in the calculation below (equation 1):

$$m = \rho \cdot V \tag{1}$$
$$2.7 \cdot 1\,000 = 2\,700\ g$$

where:

m [g]	mass
$\rho \left[\frac{g}{cm^3}\right]$	density
V [cm^3]	volume

To heat the effusion cell from a room temperature of approximately 25 °C to the required temperature of 125 °C, the heating element will need to produce at least 101 950 J of heat, as seen in the calculation below (equation 2):

$$Q_E = m \cdot c \cdot \Delta T \tag{2} [27]$$
$$2\,700 \cdot 0.385 \cdot 100 = 101\,950\ J$$

where:

Q_E [J]	thermal energy
m [g]	mass
$c \left[\frac{J}{g \cdot K}\right]$	specific heat capacity (copper)
ΔT [K]	temperature difference

The effusion cell is required to reach the temperature in less than an hour. Therefore, to account for all the uncertainties in this calculation, the heating element will be sized to reach the temperature in 30 minutes. This means that the heating element must produce at least 56.6 W of heat energy, as seen in the calculation below (equation 3):

$$Q = \frac{Q_E}{t} \tag{3}$$

$$\frac{101\,950}{1\,800} = 56.6\ W$$

where: Q [W] thermal power
Q_E [J] thermal energy
t [s] time

This means, that with one heating pad under each effusion cell (figure 34), each heating pad needs to produce at least 4.4 W of heat. To fit under the effusion cell, the pads must not be bigger than 45x45 mm. Both requirements are met by for example the Thermo TECH 7.2 W and 45x25 mm heating pad [28]. This pad is shown in figure 33.

Figure 33: Thermo TECH 7.2 W, 45x25 mm [28].

Because of the great thermal conductivity of copper, individual control of each pad is not necessary, as there should not be a large temperature difference between any parts of the effusion cells. This means that the heater pads can be connected to one power supply output, to reduce the number of connections running outside of the vacuum chamber. However, to have the same voltage on all, they will be connected in parallel. The parallel branching of the wires from the power supply will happen in the vacuum chamber itself, as this will limit the number of necessary feedthroughs in the chamber.

The thermal pad requires a 24 V connection and 13 of them will draw a total of 93.6 W at full power, therefore, the laboratory power source supply at BUT can be used for this (requirement R11), as it can supply up to 32 V and 160 W.

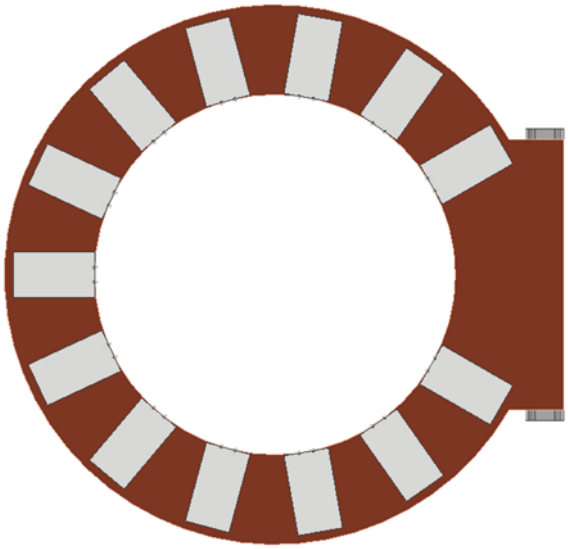

Figure 34: Heater pad layout under each effusion cell.

Using these thermal pads under each effusion cell is therefore a viable option for the heat up process. Even if the final design of this part is more massive than the preliminary design, this solution will still be viable. Not only is there still a 30-minute reserve in the calculation for the heat up process, but this heating pad is also 60 % more powerful than is required.

Additionally, because the pad is under every effusion cell, this solution is also easily scalable, if a bigger apparatus with more effusion cells is desired. Alternatively, a custom designed circular pad of similar power can be used from companies like Alper [29].

5.2. Collector water cooling

Because the collector plate must be maintained at 25 °C (requirement R2), the part that is in contact with it must be temperature controlled, specifically it must be cooled. Because 25 °C is not an unusually low temperature, water cooling will suffice.

Because the apparatus will be in a vacuum, most heat will come from radiation from the heated part of the apparatus. From the preliminary design, the total area of the heated part is approximately 120 000 mm^2. Copper has emissivity between 0.03 (polished) and 0.2 (roughed) [30]. To calculate the worst-case scenario, this calculation will take the value 0.2 for emissivity. Therefore, the total radiation heat output of the heated part given by the Stefan-Boltzmann law of radiation is approximately 34.2 W, as seen below (equation 4):

$$Q = \sigma \cdot e \cdot A \cdot T^4 \qquad (4)\ [31]$$

$$= 5.67 \cdot 10^{-8} \cdot 0.2 \cdot 0.12 \cdot 398.15 = 34.2\ W$$

where:

Q [W]	…	thermal power
σ [$W \cdot m^{-2} \cdot K^{-4}$]	…	Stefan-Boltzmann constant
e [−]	…	emissivity
A [m^2]	…	area of the part
T [K]	…	temperature of the part

For the purposes of this chapter, radiation will be the only heat transfer in the apparatus. Any possible physical connections between the parts will not be accounted for, as they are not yet designed. Instead, to create a reserve factor, the calculation will be done, as if all the heat radiated will be absorbed by the cooled part. In reality, this will likely not be the case, as some radiation will be absorbed by the vacuum chamber walls, and the direct line of sight between the heated and cooled parts will be mostly shielded by an aluminum separator plate (figure 35), which has a great reflectivity in infrared wavelengths (figure 36).

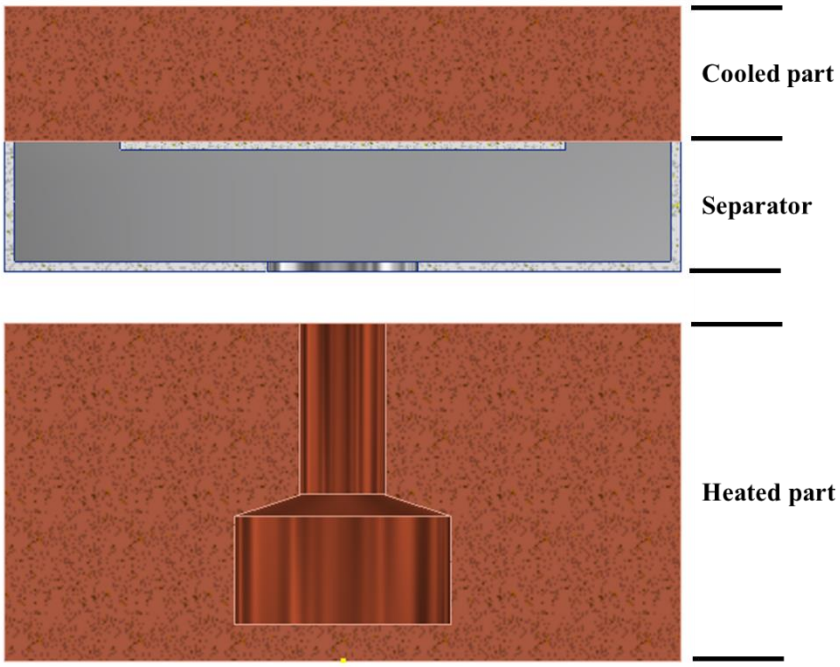

Figure 35: Separator shielding – cross-section.

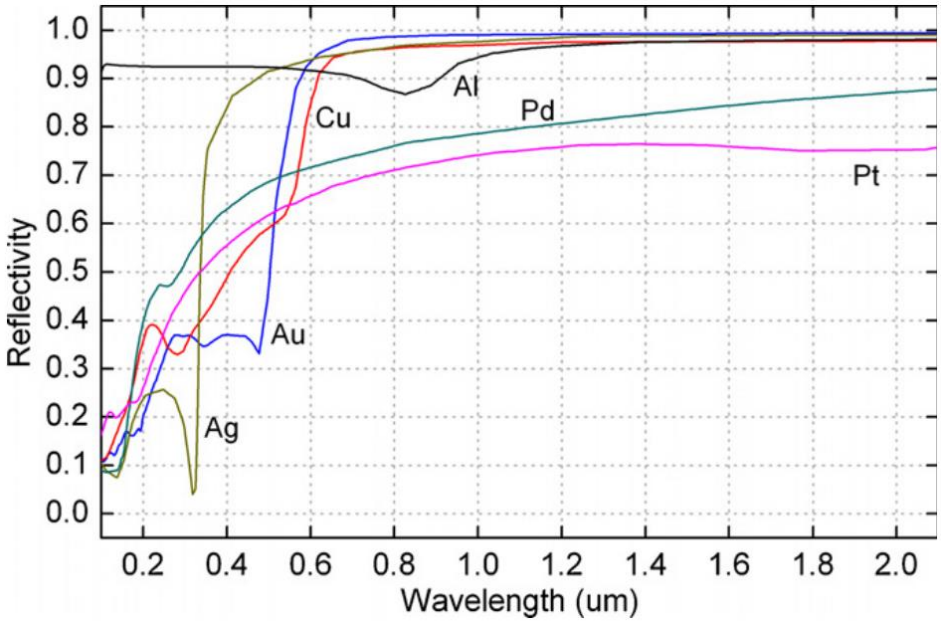

Figure 36: Reflectivity of chosen metals in different wavelengths. [32]

Therefore, the cooler will have to dissipate at least 34.2 W of heat. The water will flow in a copper tubing, as it has a high heat transfer coefficient anywhere between 340 and 455 $W \cdot m^{-2} \cdot K^{-1}$ [33]. Again, to be on the safe side, the calculation will use 340 $W \cdot m^{-2} \cdot K^{-1}$. The heat exchanger area and the water flow rate are calculated from the two equations below (equation 5, 6):

$$A = \frac{Q}{U \cdot \Delta T} \quad (5)\ [34]$$

where:
- $A\ [m^2]$ — heat exchanger area
- $Q\ [W]$ — thermal power
- $U\ \left[\frac{W}{m^2 \cdot K}\right]$ — heat transfer coefficient
- $\Delta T\ [K]$ — temperature difference between water and required temperature

$$m = \frac{Q}{c \cdot \Delta T} \quad (6)\ [34]$$

where:
- $m\ [g \cdot s^{-1}]$ — water flow rate
- $Q\ [W]$ — thermal power
- $c\ \left[\frac{J}{g \cdot K}\right]$ — specific heat capacity (copper)
- $\Delta T\ [K]$ — temperature difference between water and required temperature

As seen in the two formulas, to calculate the two values, the temperature difference is required. This value is determined by looking at resulting flow rates and heat exchanger area for different values. These values are in table 13.

Table 13: Resulting cooling layout and flow rate based on initial water temperature.

Coolant temperature	**21 °C**	**22 °C**	**23 °C**	**24 °C**
Flow rate	1.33 l/min	1.78 l/min	2.66 l/min	5.33 l/min
Heat exchanger area	25 147 mm^2	33 529 mm^2	50 294 mm^2	100 588 mm^2
Number of loops based on cooling tube diameter				
⌀ 3 mm	3.4	4.5	6.8	13.6
⌀ 4 mm	2.6	3.4	**5.1**	10.2
⌀ 5 mm	2.1	2.7	4.1	8.2

For the final design, the water will be running at 23 °C in the cooling system, the cooling layout will be 5 loops of 4 mm tubing and the flow rate will be 2.7 liters per minute. This is because 5 loops should provide a nice and even distribution of the coolant. However, other configurations could also be used if deemed necessary.

To feed the cooled water into the loop, one possible water cooler is an industrial 9-liter cooler from Stamos [35], shown in figure 37. With a maximum flow rate of 8.5 liters, an advertised cooling power of 1 700 W and water temperature from 20 °C to 60 °C, this cooler is more than capable of cooling the system. Unfortunately, lower powered coolers are difficult to find.

Figure 37: A 9-liter water cooler from Stamos. [35]

Due to the shape of the cooled part, it will have to be a custom manufactured part. As mentioned previously, the heat exchanger area is equal to approximately 5 loops of a 4 mm diameter tubing. Therefore, the cooling channels will be as shown in figure 38. They will copper tube, inserted in a prepared metal part, as shown in figure 39. Because it uses standard tubing, the intake and output ends of the tubing will also use standard headers. Specifically, the CEL header [36] will be used to connect to a flexible hose, as this part will be able to rotate up, so the connection must be flexible. The hose will then be connected to the vacuum chamber interface wall.

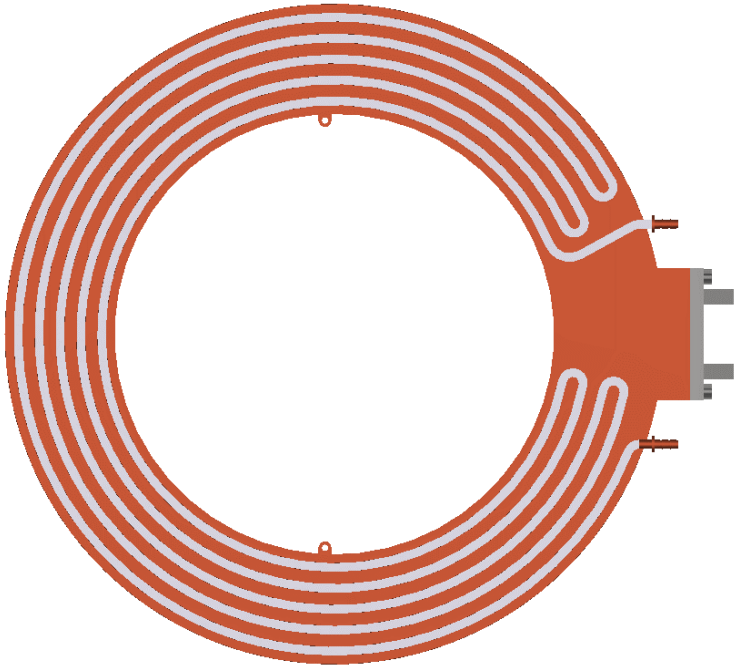

Figure 38: Cooling channels layout.

Figure 39: An example of cooler manufacturing technology. [37]

5.3. Temperature monitoring

Both the heated effusion cell and the cooled collector plate temperatures have to be monitored during the test (requirement R7). For this, thermocouples are the ideal solution. Thermocouples from IST [38] will be used. They provide multiple classes with varying deviations from the real temperature.

The highest temperature these thermocouples will monitor is 125 °C of the effusion cell. The deviation at this temperature should be no less than 0.5 °C, to remain usable. This means that

the IEC60751 F 0.15, with a temperature deviation of 0.15 + 0.002 · T [°C] or 0.4 °C at 125 °C will be used.

Because the thermocouples cannot be placed directly at the collector plate and the sample because it would interfere with the test, they will be placed as close as possible (figure 40). Unfortunately, there will always be a small inaccuracy in the real temperature versus the measured temperature. This can be partially eliminated by using one empty compartment unused during the test and measuring the temperature directly in the sample boat and on the collector plate (figure 41).

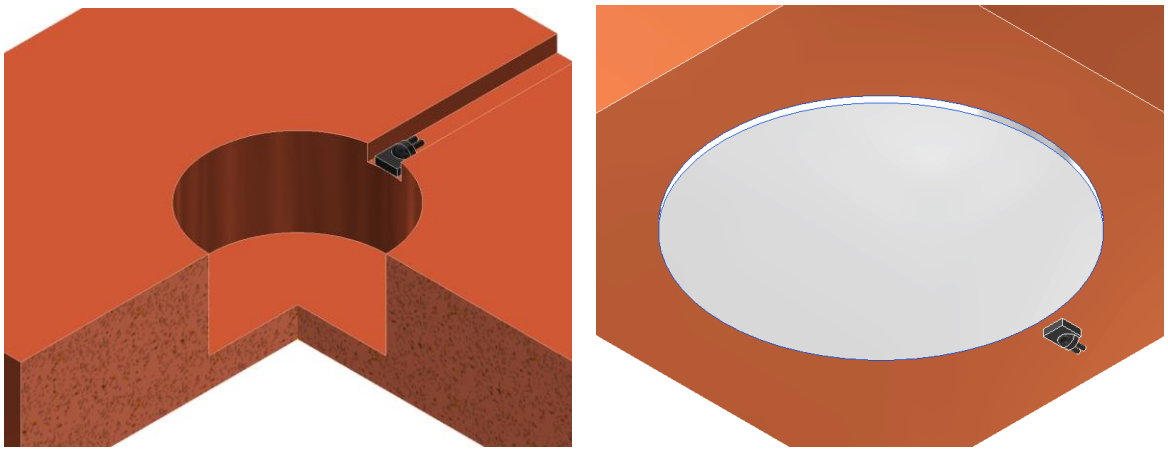

Figure 40: Thermocouple placement for effusion cell (left) and collector plate (right) – temperature measurement during test.

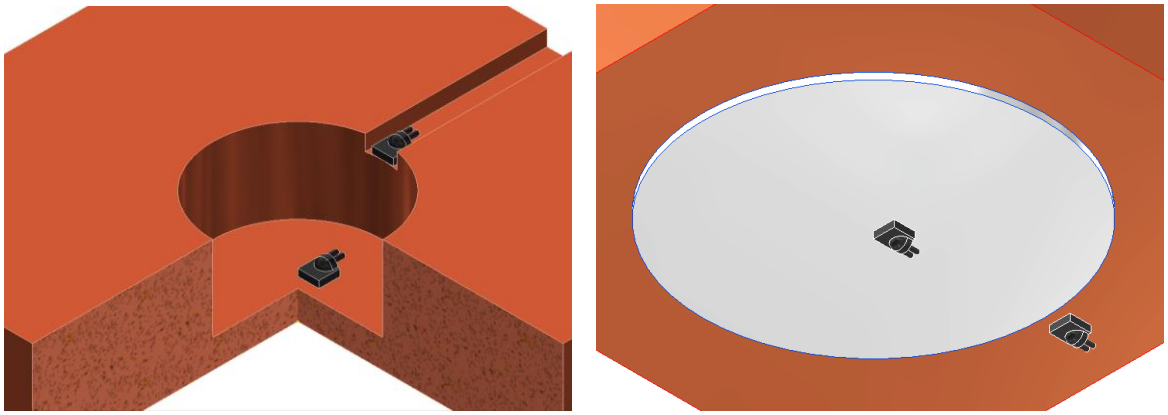

Figure 41: Thermocouple placement for effusion cell (left) and collector plate (right) – temperature correction.

5.4. Vacuum gauge

Just like the pump system, this system will utilize a vacuum gauge that is already at the BUT test facility (requirement R12). It is the Edwards Vacuum Active Wide Range Gauge [19], or WRG for short. It can measure from atmospheric pressure all the way down to 10^{-7} Pa. This makes it a good fit for this test apparatus.

Figure 42: Edwards Vacuum WRG. [19]

The connection to the chamber is created with a KF25 vacuum flange (figure 43), so the vacuum chamber will have to incorporate it. The output signal is a DC voltage from 1.8 to 10.2 V.

Figure 43: KF255 vacuum fitting. [39]

5.5. Data logger

This data logger will take the data from all the thermocouples and the vacuum gauge. There are 28 thermocouples in total, if the correction thermocouples are included. With the additional input from the vacuum gauge, this data logger must have at least 29 ports.

Possible solution is a Hioki LR8400 (figure 44) with 30 discrete channels [40]. Each channel can receive an input of up to 50 V, more than enough for the vacuum gauge that is sending a signal of up to 10.2 V. It can also read signal from devices that output 0-20 mA as signal, meaning that thermocouples can also be connected.

Figure 44: Hioki LR8400 data logger. [40]

5.6. Nitrogen backfill

Backfilling the system with nitrogen after each test is part of the standardized procedure (requirement R8). The required backfill pressure is 14-28 kPa. The backfilling is done with a valve connecting the vacuum pump closed.

To backfill gas into a vacuum chamber controllably, a mass-flow transducer or meter is usually used [41]. These can not only monitor the flow of the gas into the chamber, but often also regulate it. A possible solution is a thermal flow meter and controller, like the one in figure 45 from Voegtlin Instruments. This would sit outside the vacuum chamber and control the flow. As for the source of the nitrogen, because of the small size of the apparatus, an 8-liter nitrogen bottle (figure 46) is sufficient for multiple tests.

To ensure that no nitrogen is leaking into the chamber during the test, a separate valve will be place between the flow controller and the vacuum chamber, that will be closed during the test. A manual valve from Lesker Company (figure 47) can be used. It will interface with the chamber with a standard KF16 flange.

Figure 45: Thermal mass flow meters and Mass flow controllers for gases. [42]

Figure 46: Nitrogen bottle (8 liters) [43]

Figure 47: A manual angled valve. [44]

5.7. Mass measurement - microbalance

A microbalance is one of the most essential parts of the test system, as all the results will be measured on it. This means that it must be chosen properly, otherwise it will not be possible to evaluate the test.

There are three things that the microbalance will be weighing. The sample boat, the sample boat with the sample inside and the collector plate. All of these will be measured before and after the test.

The collector has a minimum dimension of ⌀33x0.65 mm. Usually being made out of aluminum, the minimum mass is 1.5 grams. This is without any overhangs to secure the collector to the apparatus. However, even with a significant increase in volume, this collector is very unlikely to surpass 5 grams in total weight.

The sample boat has no strict dimensions, however it must fit in the effusion cell. Also being made from aluminum, the boat alone will weigh approximately 1 gram. With a sample inside, it will be 1.3 grams.

Therefore, the microbalance should have a maximum capacity of more than 5 grams. The biggest object that will be measured is the collector plate with a diameter of at least ⌀33 mm. Therefore, the minimum dimension of the weighing chamber must be 40x40 mm. The sensibility, as per requirement R10, must be at least 1 µg (requirement R10).

In table 14, a list of possible solutions is presented. All these candidates fulfill the requirements of maximum capacity and sensitivity. All candidates can also accommodate the collector plate within their weighing chamber, as the chamber is always bigger than the weighing pan. The main deciding factor is therefore the price, with a secondary factor being repeatability, as that influences the result's confidence level.

Table 14: Microbalance comparison.

Microbalance	Maximum capacity	Sensitivity	Repeatability	Weighing pan dimensions	Quote
Radwag MYA 5.5Y [45]	5.1 g	1 µg	0.6 µg	⌀26 mm	343 170 CZK [1]
Radwag MYA 11.5Y [46]	11 g	1 µg	0.9 µg	⌀26 mm	389 000 CZK [2]
Radwag MYA 21.5Y [47]	21 g	1 µg	1 µg	⌀26 mm	429 000 CZK [3]
Radwag XA 6/21.5Y.M [48]	6 g (21 g)	1 µg (2 µg)	1.3 µg	⌀30 mm	279 000 CZK [4]
Sartorius MCA10.6S-2S00-M [49]	10.6 g	1 µg	5 µg	⌀30 mm	-
Mattler Toledo XPR36DR [50]	8,1 g (32 g)	1 µg (10 µg)	1 µg	40x40 mm	910 700 CZK[5]

[1]Quoted to 22.4.2024
[2]Quoted to 22.4.2024
[3]Quoted to 22.4.2024
[4]Quoted to 22.4.2024
[5]Quoted to 3.5.2024

5.8. Conditioning room

Both standards require that all samples are preconditioned before the TML test. Postconditioning is also a requirement to obtain the WVR value. For this, a special conditioning chamber is used. The chamber must be able to regulate the temperature and the humidity inside

to fulfil the imposed requirements. The required temperature that it must hold is 25°C (requirement R4) and the humidity is 50% (requirement R5).

The BUT test facility already has a controlled atmosphere chamber. It has the ESPEC ARS-680. This chamber can control temperatures between -75 °C and +180 °C and relative humidity between 10 % and 98 %. The chamber volume is 680 liters, so it satisfies all the requirements of this test.

5.9. System specification overview

Sample heating power and design and collector plate cooling power and design, along with several other components and subsystems were reviewed and their overview and how they will be implemented is in table 15. A schematic view of all subsystems is also in figure 48.

Table 15: Components to be incorporated to the final design.

Component	Implementation
Heating pads	A power connection must be run through a feedthrough into the vacuum chamber.
Cooling loop	A water loop must be run though a feedthrough into the vacuum chamber.
Thermocouples	28 separate thermocouple connections must be run through a feedthrough into the vacuum chamber.
Vacuum gauge	A KF16 flange must be incorporated into the vacuum chamber.
Data logger	Outside of the vacuum chamber.
Nitrogen valve	A valve connecting a nitrogen bottle must be incorporated into the vacuum chamber.
Microbalance	Outside of the vacuum chamber.
Conditioning room	Outside of the vacuum chamber.

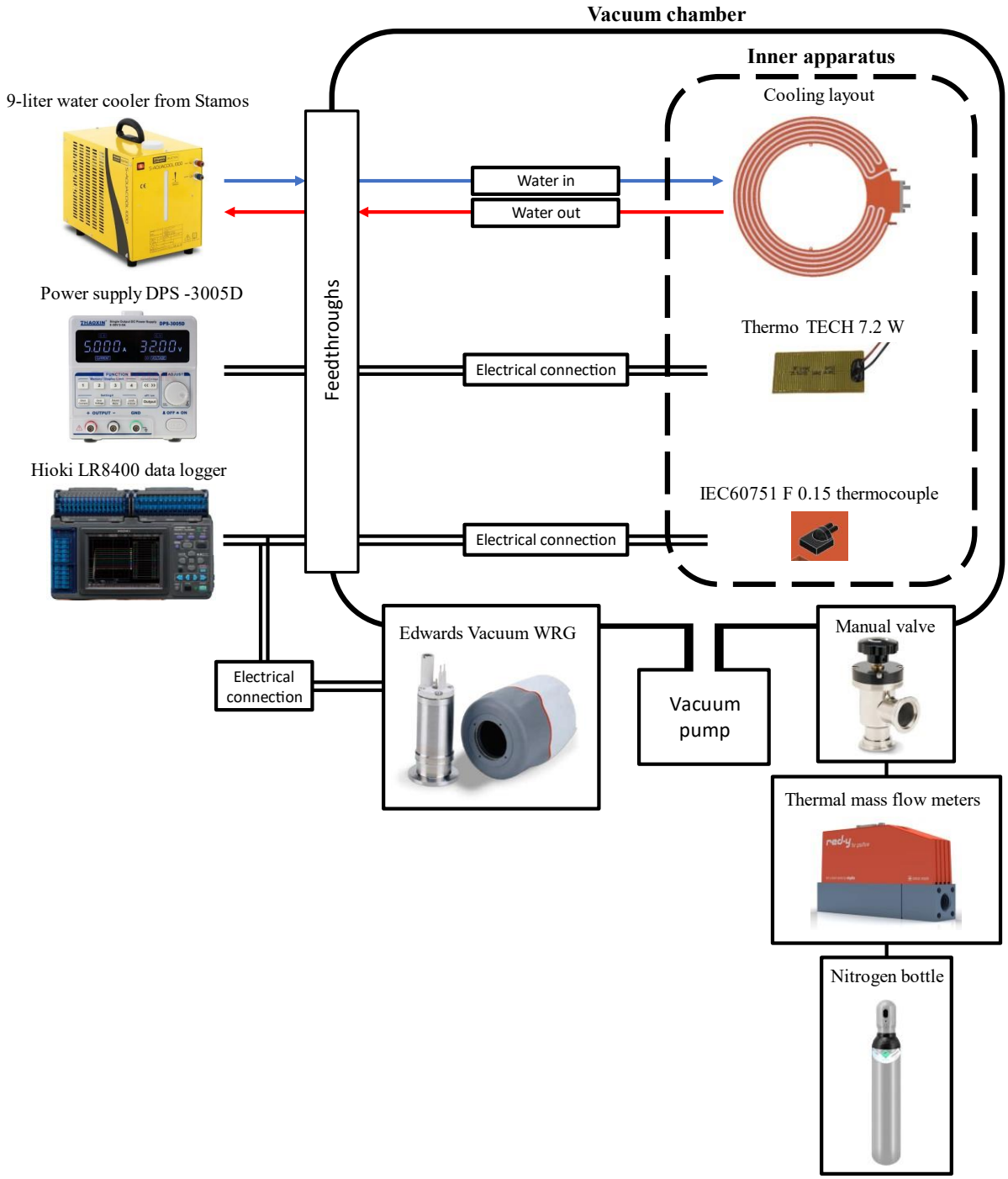

Figure 48: Schematic connections with specific components.

6. Test apparatus design

With the inputs from the previous chapter, the apparatus can be designed. The design will feature 13 sample compartments to conform to the requirement R17. Most of the dimensions will therefore be derived from the number of samples.

Similarly, the inner apparatus dimensions will dictate the dimensions of the vacuum chamber. Therefore, the design will begin with the inner apparatus (chapter 6.2.) and then follow up with the vacuum chamber design (chapter 6.2.). An overview of the complete system will also be provided in chapter 6.3.

To prevent describing every single dimension from the design, only significant design features will be presented in this chapter. The complete model will then be available as the annex of this thesis.

6.1. Inner apparatus design

The basis of the inner apparatus design comes from the requirement R9, the critical dimensions prescribed by the standards. These can be seen in figure 49.

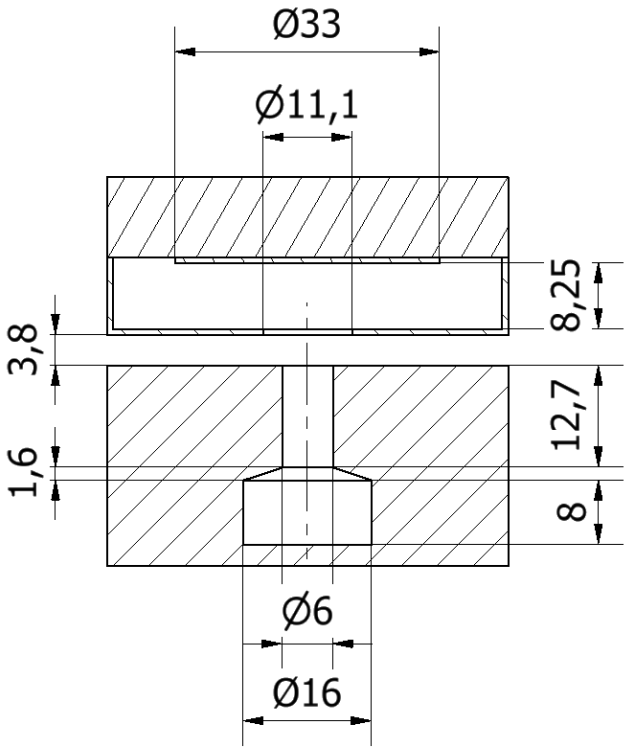

Figure 49: Effusion cell, separator and collector plate with critical dimensions implemented (requirement R9).

To make the design clearer, it will be separated into 3 assemblies, the base, the middle assembly and the top assembly (figure 50). The division comes from the necessity to access the samples that are in the base and the collector plates that are in the top assembly. The base, as the name suggests, will be the stationary part where the samples will be placed during the test. The middle assembly is the top of the effusion cell and the separator plate. Finally, the top assembly are the collector plates and cooling.

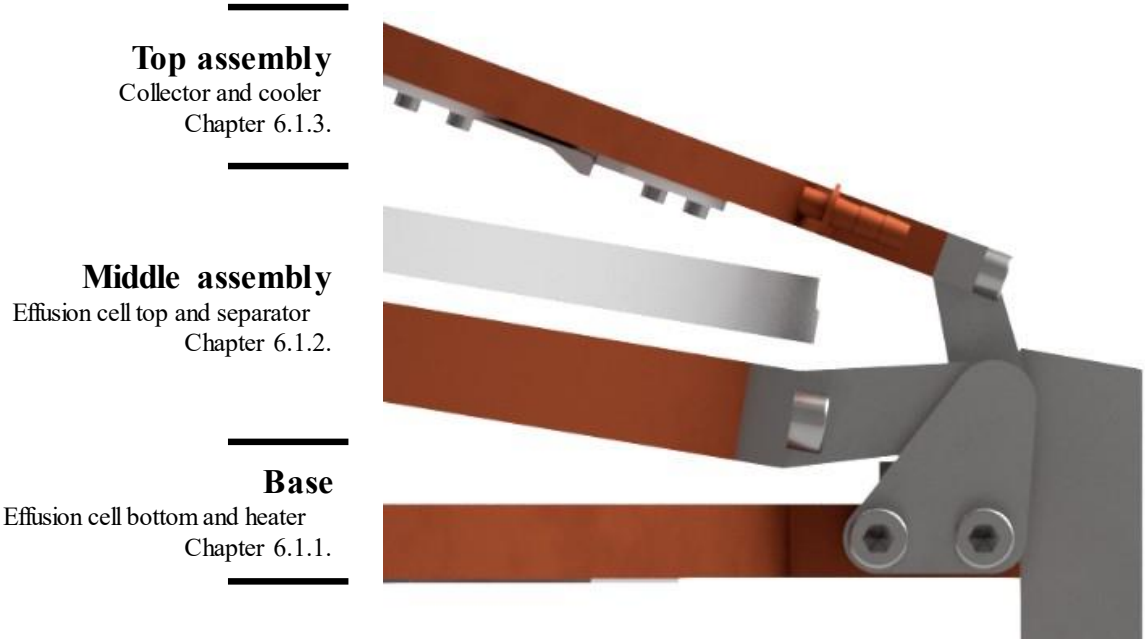

Figure 50: Inner apparatus basic division.

6.1.1. Base

As mentioned previously, the base is a stationary part of the system that will house the material samples during the test. To allow the loading of the samples into the effusion cells, the base consists only of the bottom part of the effusion cell. Once the sample is placed in the effusion cell, the top part of the effusion cell, that is part of the middle assembly, will enclose the sample (figure 51).

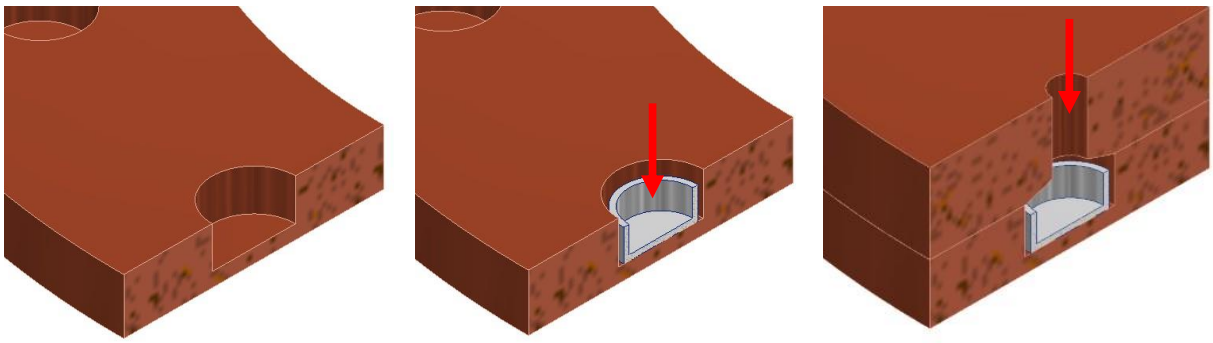

Figure 51: Sample loading process.

Some design features of this part were discussed in previous chapters. This includes the grooves for the thermocouples that lead to the sample compartments (chapter 5.3) and heater pads under each effusion cell (chapter 5.1). These features can be seen in figure 52.

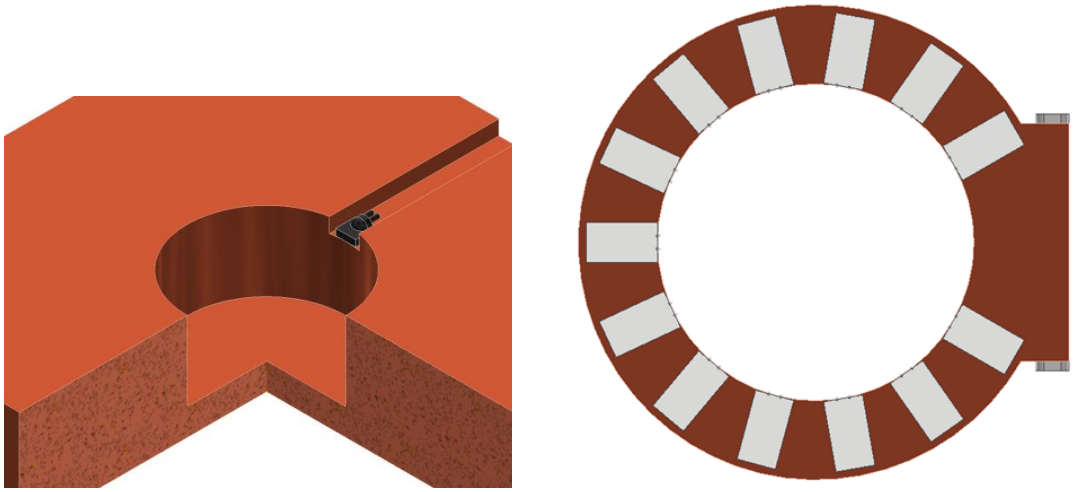

Figure 52: Groove for thermal couple (left) and heater pads layout (right).

The hinge main axis is also connected to this part. To facilitate manufacturing it is a separate part connected with 4 screws. Additionally, to lift the whole assembly higher, three legs are connected to the base (figure 53). This is done because most of the feedthroughs will be located on the bottom of the vacuum chamber, therefore a clearance for the cables must be created below the assembly. The feet of the legs will be lined with Teflon pads, to lower the heat conduction to the vacuum chamber. Teflon is used because it has a very low outgassing, comparable to metals [7].

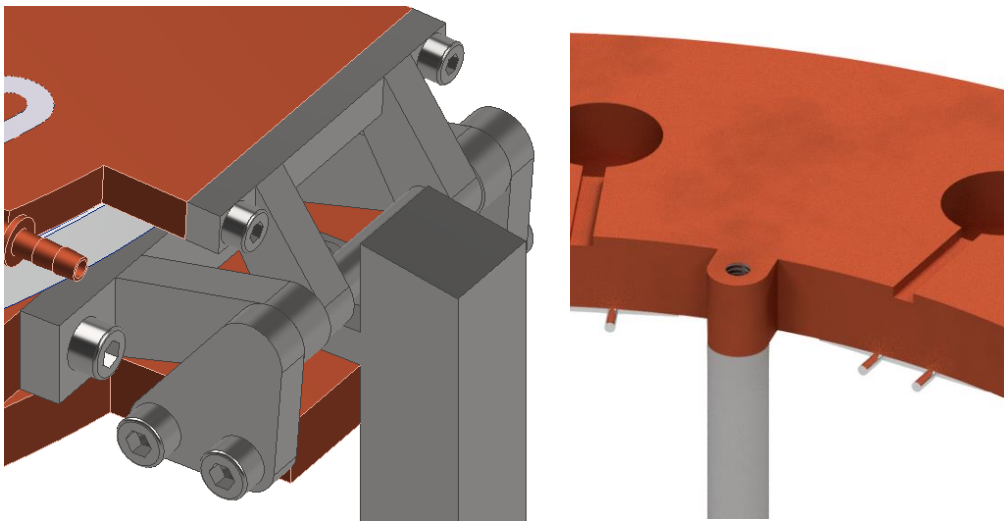

Figure 53: Hinge mechanism (left) and one of the legs of the apparatus (right).

6.1.2. Middle assembly

The middle part is composed of two main parts, the top of the effusion cell and the separator plate. This part must be able to rotate away from the base to allow access to the sample compartments, but it also must be able to rotate independently of the top part, as access to the collector plates is also required.

To allow good thermal conductivity between the bottom of the effusion cell and the top, two screws are introduced that will be tightened once the samples are loaded. A second function of these screws is the correct positioning of the middle assembly on the base, to prevent the parts being of center from each other. This can be seen in figure 54.

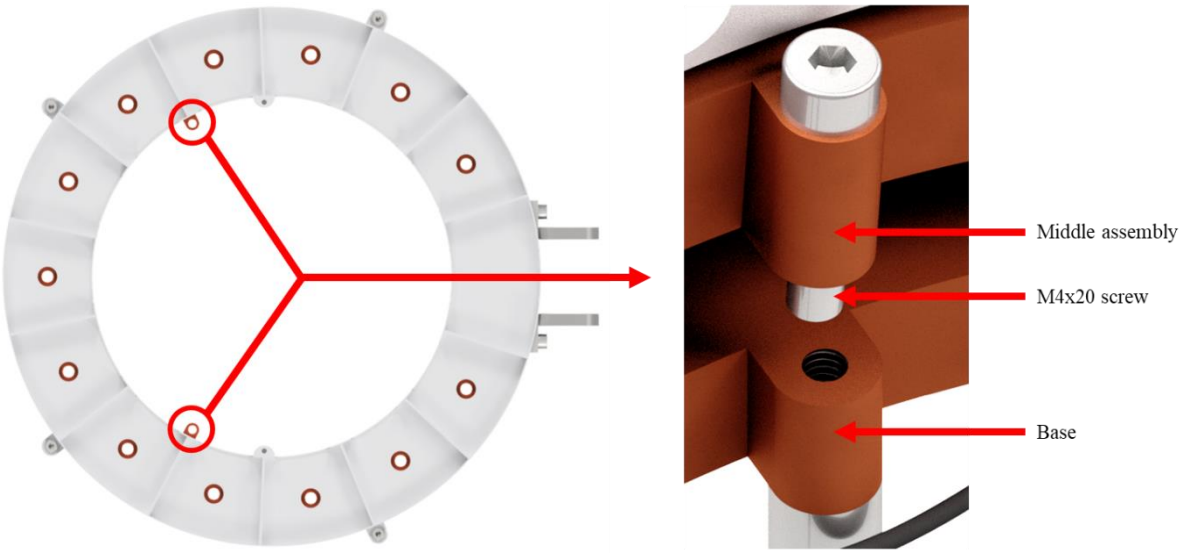

Figure 54: Base-to-Middle parts positioning and tightening.

While the top of the effusion cell is in close contact with the heated base, the separator plate is in contact with the cooled top assembly. Therefore, the connection of these two should be minimal to limit the heat transfer through conduction. For this reason, only four M4 screws are used. To limit the use of metal with high thermal conductivity in this connection, the distancing washer will be made from Teflon. On of these four connection points is in figure 55.

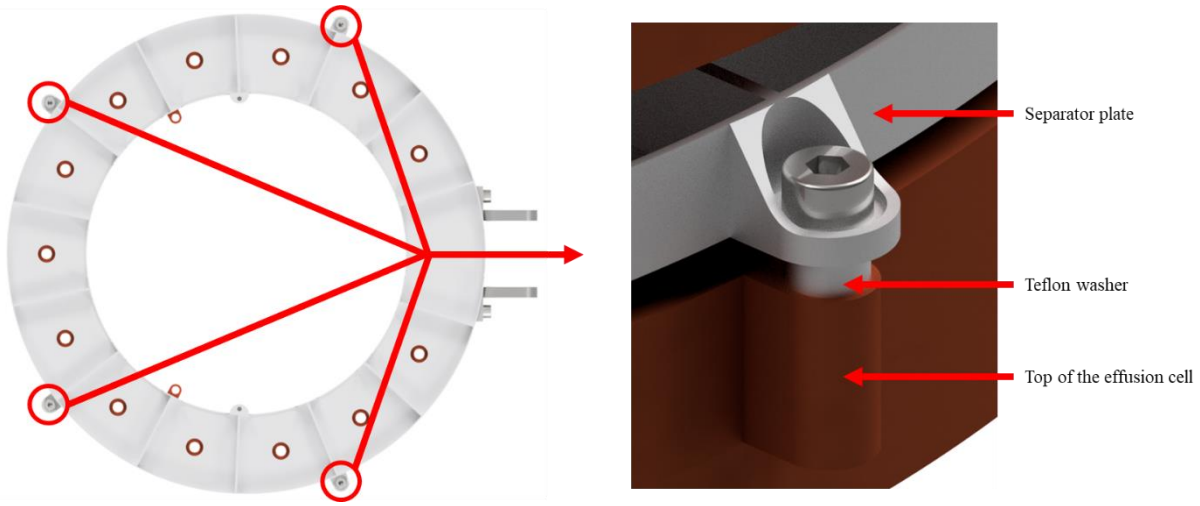

Figure 55: Separator plate and effusion cell connection.

6.1.3. Top assembly

The main features of the top assembly are the collector plates and the cooling system. The basics of the cooling system were described previously in chapter 5.2. Important to note is the location of the inflow and outflow connections. They will be located as close as possible to the hinge axis (figure 56), to limit the change in distance the tubing must cover from closed position to open position of the apparatus (figure 57).

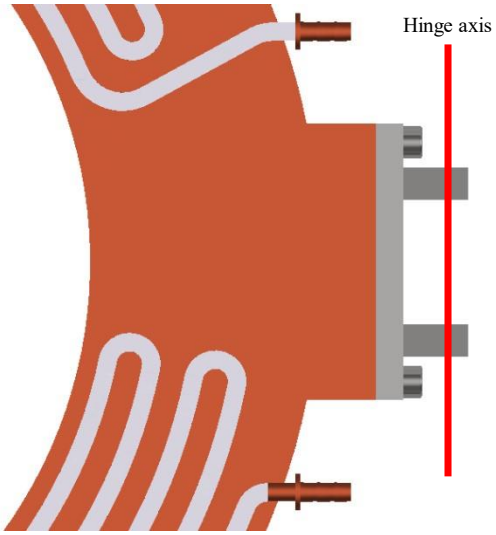

Figure 56: Cooling tubing inflow and outflow connections.

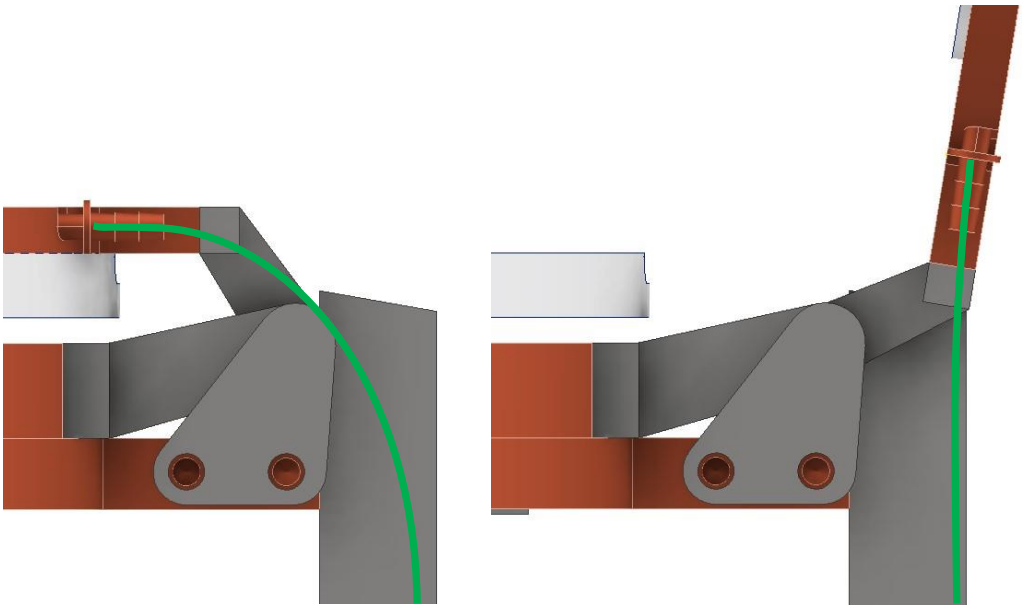

Figure 57: Tubing route (green) in closed (left) and open (right) position.

As for the collector plates, they will be loaded and unloaded when the apparatus is opened. This means that gravity can be used to position them correctly in the holder before securing them down. The only problem is that the securing screws must not penetrate the cooling system that is just 2 mm below the surface. This means that every collector holder has the tapped holes for the screws in a slightly different position. To prevent having to manufacture 13 different collector holders with slightly different screw patterns, the holder design has a universal pattern, that allows the use of only one design for all 13 collectors. The holder and how it works is in figure 58.

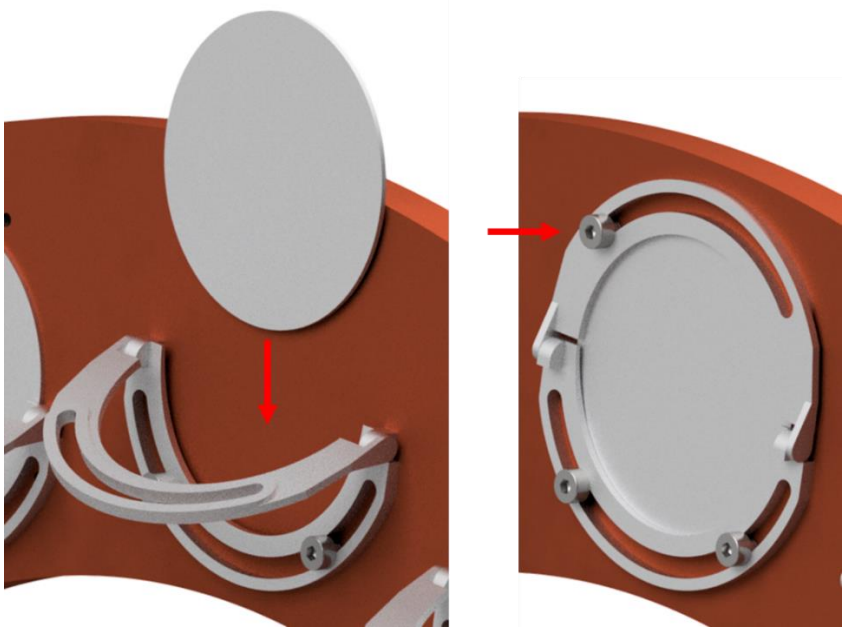

Figure 58: Collector holding mechanism.

Additionally, a positioning mechanism between the middle assembly and the top assembly is implemented. Unlike the one between the base and the middle assembly, this one is only two pins, as no tight contact is required here. Having tight contact here would be contra productive, as it would allow for easier heat transfer from the separator to the top assembly. One of these pins is in figure 59.

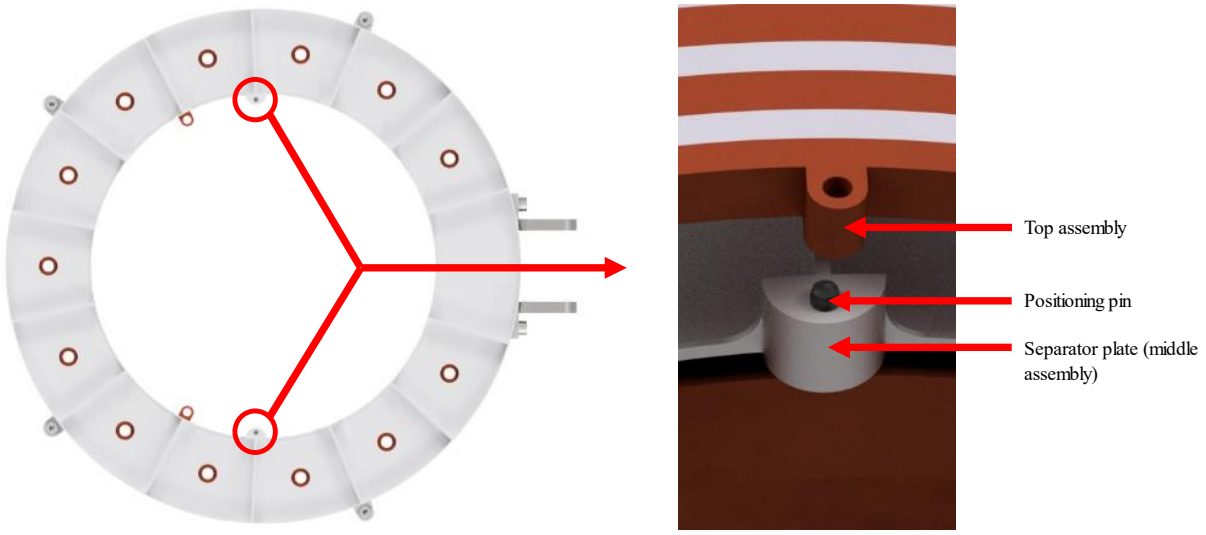

Figure 59:Middle-to-Top assembly positioning.

6.1.4. Inner apparatus overview

With all individual parts designed, the complete apparatus can be assembled. The apparatus has 3 basic positions. Closed during the test, top assembly opened, when working with collector plates, and both top and middle assemblies opened, when working with samples. All three states are shown in figure 60.

A notable feature is that there is a maximum position how far the apparatus can be opened (figure 61). This is to facilitate operations with the collector plates. The maximum position is 105° from horizontal, then it is blocked by one of the legs.

As mentioned previously, for more details, refer to the complete CAD model in the annex of this thesis.

Figure 60: CAD model of the inner apparatus in all three positions.

Figure 61: Side view in open position.

6.2. Vacuum chamber design

The vacuum chamber will be designed around the inner apparatus, to accommodate it in both closed and opened positions. Along with that, all outside connections, shown in figure 62, where incorporated to the design.

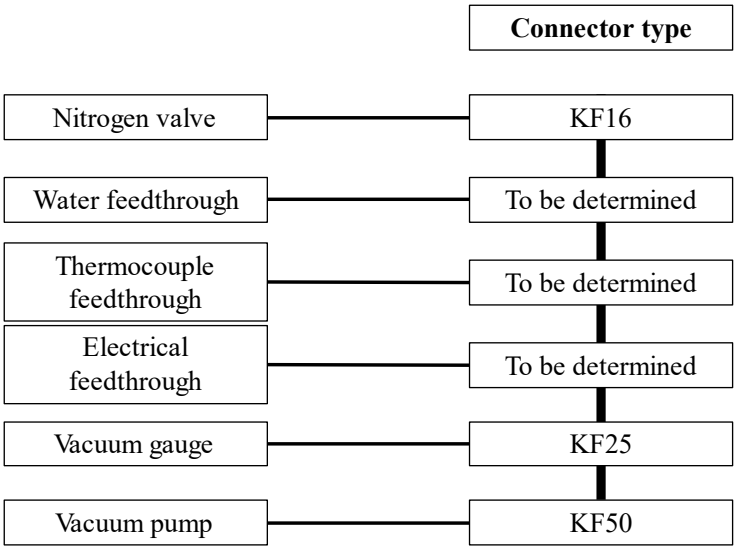

Figure 62: Outside connection types determined in previous chapters.

First, all feedthroughs that are not yet chosen will be chosen so that the complete chamber design can then be done.

Similarly to the inner apparatus design, only important features will be presented here. For more details, refer to the complete CAD model in the annex of this thesis.

First the basic design of the vacuum chamber, meaning walls, will be presented. Then all outside connections will be shown in detail, followed by an overview of the assembled vacuum chamber.

6.2.1. Feedthroughs

Three feedthroughs, fluid for cooling, electrical to power the heaters and electrical for thermocouples, must first be chosen. All will be chosen from Lesker Company, as they along with a comprehensive portfolio of vacuum accessories provide an extensive CAD catalogue for all parts. This will facilitate the modeling of the chamber.

The fluid and the electrical power feedthroughs will be both going through a CF16 flange. The specific parts are in figure 63 and figure 64. For the thermocouples, a feedthrough with a CF35 flange will be used figure 65. This feedthrough has connections for 5 thermocouples, so a total of 6 will be implemented to the design.

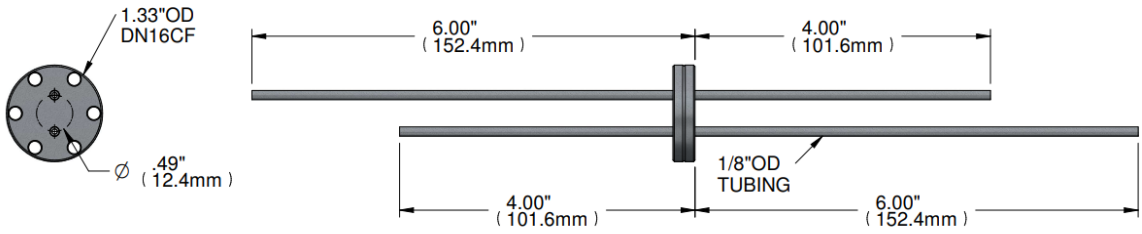

Figure 63: Fluid feedthrough with a CF16 flange. [51]

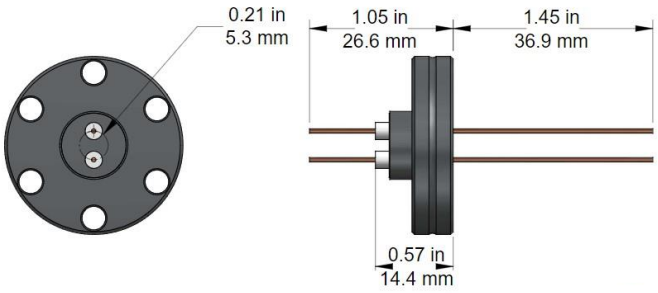

Figure 64: Electrical feedthrough with a CF16 flange. [52]

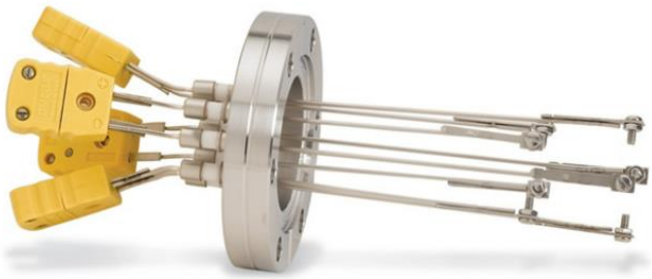

Figure 65: Thermocouple feedthrough with a CF35 flange. [53]

6.2.2. Vacuum chamber walls

With all necessary connections determined, the wall of the chamber can be designed and dimensioned. To accommodate the inner apparatus with some clearance, the inner diameter of the chamber is 400 mm. To facilitate the manufacture as much as possible, the bottom and the top of the chamber are flat surfaces. From the strength point of view, this is obviously not ideal for a vacuum chamber, so a FEM analysis was done to verify the design.

The analysis was done with a simplified model, without any bolts or feedthroughs modelled. As the load, atmospheric pressure was applied to all outside surfaces. The most critical part of the design is the top lid, which is a 10 mm thick sheet of stainless steel. The maximum stress in the model is 32 MPa, which for stainless steel that has a tensile strength of at least 200 MPa results in a reserve factor RF = 6.25. The maximum displacement in the center of the lid is 0.17 mm, so nothing would somehow interfere with the inner apparatus. The results for

maximum displacement and maximum stress are in figure 66. If required, these results could be used to optimize the design and reduce the total weight of the system.

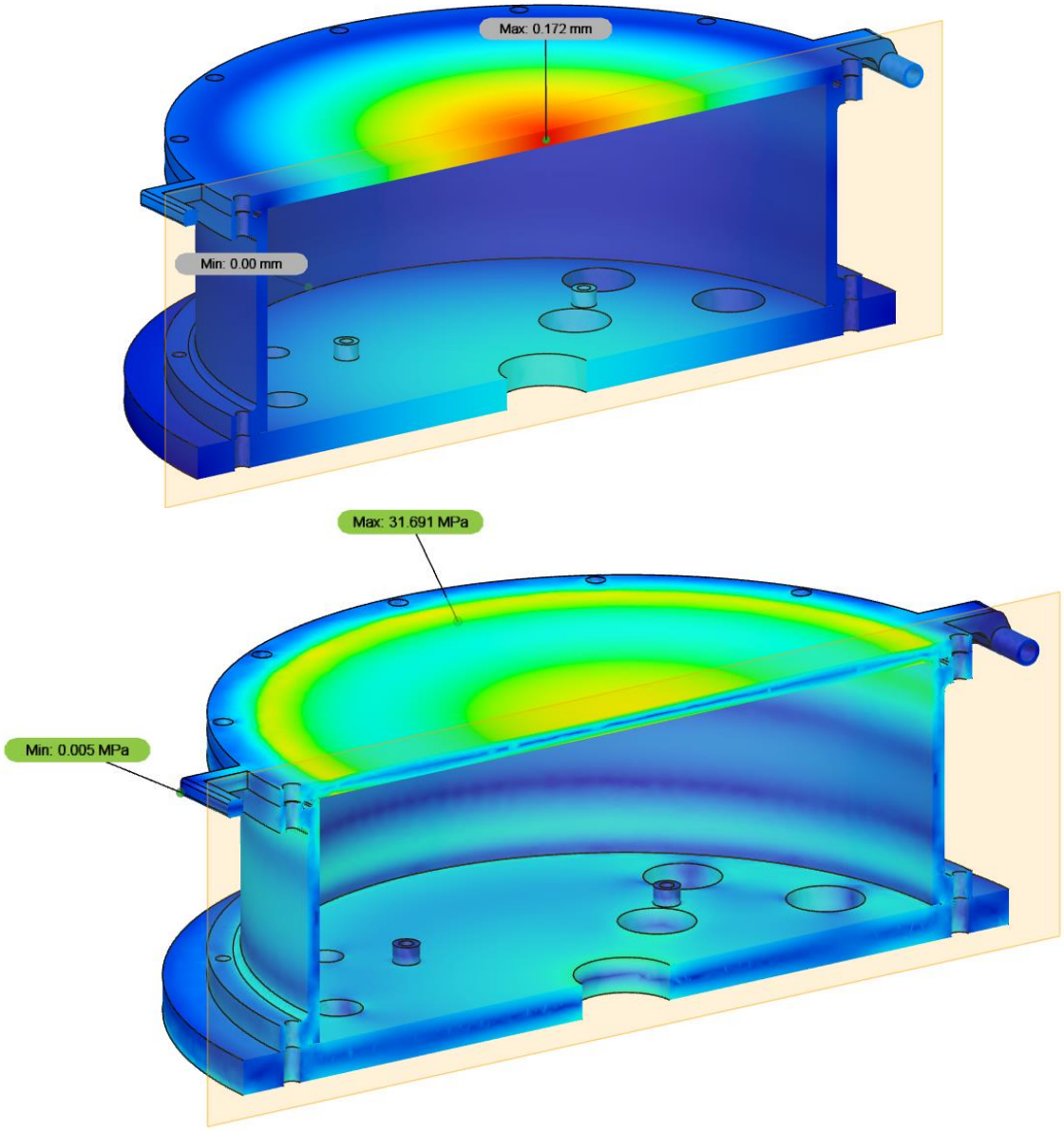

Figure 66: Vacuum chamber walls FEM analysis under atmospheric pressure – displacement (top) and stress (bottom).

Being made from three parts, there are two sealing that need to be designed. The bottom sealing will be a standards copper gasket, as metal to metal contacts provide the best seal and this assembly is not going to be regularly dismantled. The top sealing, however, will be opened for every test, so a rubber O-ring must be used. For the housing of the O-ring, a Half Dovetail Groove was chosen with an O-ring with a 5 mm diameter (figure 67).

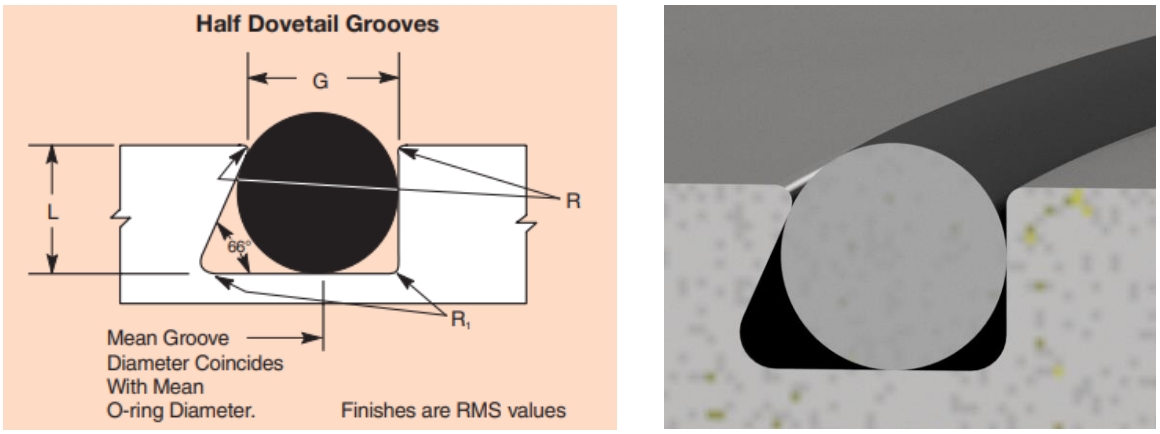

Figure 67: Half Dovetail Groove standardized (left) [54] and implemented (right).

6.2.3. Vacuum chamber overview

With the chamber wall designed and the feedthroughs chosen, the two can be implemented. To prevent creating a complicated part that is difficult to manufacture, all feedthroughs will be coming out the bottom of the chamber. The layout can be seen in figure 68 and the connections from below can be seen in figure 69.

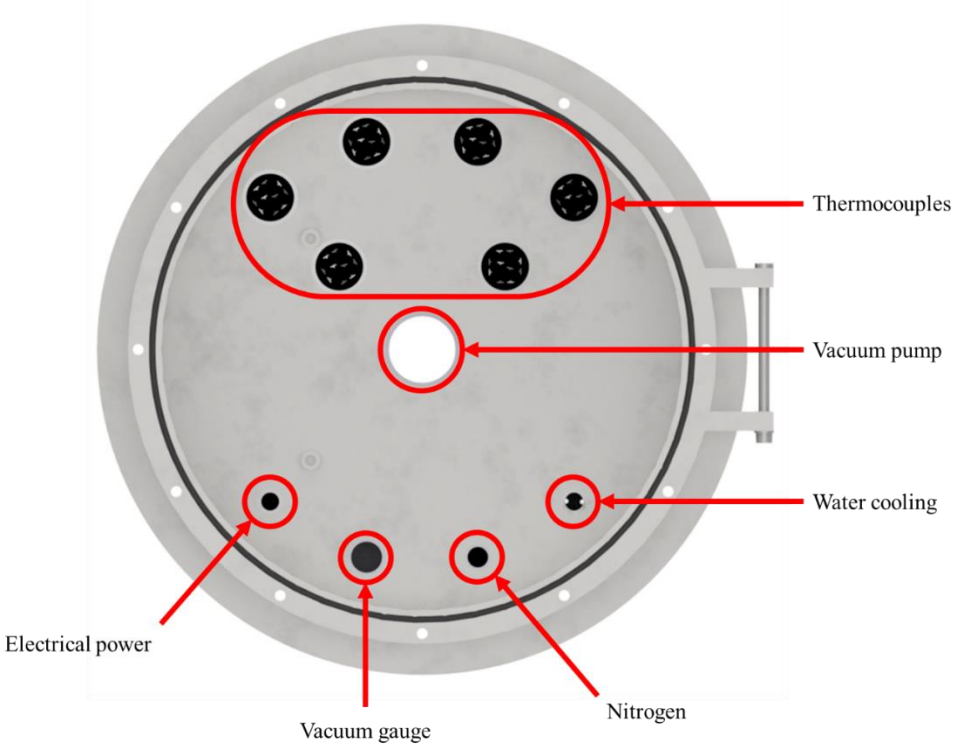

Figure 68: Vacuum chamber feedthroughs layout.

Figure 69: Bottom of the vacuum chamber will all connections modeled.

6.3. Complete system overview

Because all the outside connections from the vacuum chamber are running downwards, an aluminum frame was added to create a clearance below the chamber. The complete system can be seen in figure 70. For more details, refer to the complete CAD model in the annex of this thesis.

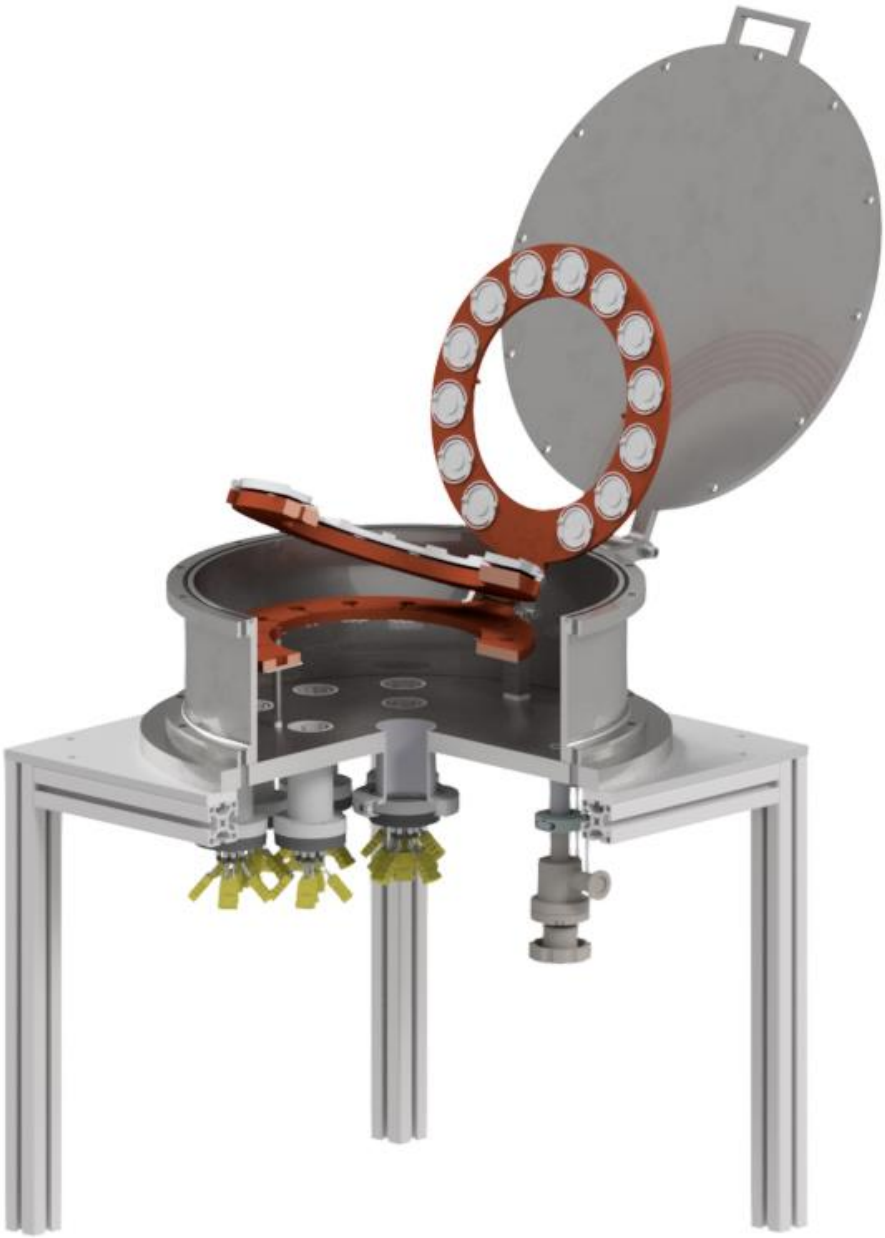

Figure 70: Complete system render.

7. Price breakdown

The price breakdown is split into two tables. One is for commercially available and off-the-shelf components, for which the prices are usually available (table 17). The second one is for custom designed parts, that will have to be custom manufactured (table 16). For the custom-made parts, the prices are only an estimate, as these will differ by manufacturer and possibly other factors.

The total price of the off-the-shelf components is 429 611 CZK and the estimated total price of the custom parts is 289 758 CZK. The total estimated price is therefore **719 369 CZK**. The final price will likely be higher, as both tables are missing prices for some components/parts.

Table 16: Estimated prices of custom components manufactured using CNC.

	Part	Reference	Pieces	Estimated price*
Inner apparatus	Base (bottom of the effusion cells) - Copper C101	[55]	1	19 368 CZK
	Top of the effusion cells - Copper C101	[55]	1	20 471 CZK
	Separator plate - Aluminum 6061	[55]	1	4 364 CZK
	Cooler layout - Copper C101	[55]	1	16 301 CZK
	Cooler tubing		1	-
	Collector plate holder - Aluminum 6061	[55]	13	31 308 CZK
	Base leg - Aluminum 6061	[55]	2	2 491 CZK
	Hinge leg - Stainless steel SUS303	[55]	1	3 052 CZK
	Hinge – base - Stainless steel SUS303	[55]	1	2 477 CZK
	Hinge – middle - Stainless steel SUS303	[55]	1	2 511 CZK
	Hinge – top - Stainless steel SUS303	[55]	1	1 494 CZK
Vacuum chamber	Top lid - Stainless steel SUS303	[55]	1	37 346 CZK
	Chamber wall - Stainless steel SUS303	[55]	1	144 790 CZK
	Chamber bottom - Stainless steel SUS303	[55]	1	-
Table	Table top - Aluminum 6061	[55]	1	3 785 CZK

*EUR-CZK exchange rate used was 24.74 CZK and EUR-USD exchange rate used was 22.72 CZK

Table 17: Prices for off-the-shelf components.

	Part	Reference	Pieces	Total price*
Outside systems	Microbalance Radwag XA 6/21.5Y.M	[48]	1	279 000 CZK
	A 9-liter water cooler from Stamos	[35]	1	12 990 CZK
	Hioki LR8400 data logger	[40]	1	47 700 CZK
Inner apparatus	Heating pad Thermo TECH 7.2 W, 45x25 mm	[28]	13	7 778 CZK
	Thermocouple IEC60751 F 0.15	[38]	28	2 290 CZK
	M5x14 screw	[56]	3	125 CZK
	M5x20 screw	[56]	4	211 CZK
	M3x14 screw	[56]	2	72 CZK
	M5 washer	[56]	7	29 CZK
	M3 washer	[56]	2	4 CZK
	M4x16 screw	[56]	6	84 CZK
	M4x18 screw	[56]	2	84 CZK
	M4 washer	[56]	2	8 CZK
	M2x4 screw	[56]	39	2 184 CZK
	Pin 4x8	[56]	2	110 CZK
Vacuum chamber	A manual angled valve	[44]	1	5 987 CZK
	KF16 Half Nipple	[57]	1	506 CZK
	KF25 Half Nipple	[57]	1	937 CZK
	KF50 Half Nipple	[57]	1	1 753 CZK
	KF16 Centering Ring	[58]	1	114 CZK
	KF16 Machined Clamps	[59]	1	990 CZK
	LFT/LNFT Fluid Feedthrough	[51]	1	4 993 CZK
	Electrical feedthrough	[52]	1	3 550 CZK
	CF16 Weldneck	[60]	2	17 318 CZK
	M4x20 screw	[56]	12	382 CZK
	M4 washer	[56]	24	100 CZK
	M4 nut	[56]	12	69 CZK
	Thermocouple feedthrough	[53]	6	83 700 CZK
	CF35 Weldneck	[60]	6	92 775 CZK
	M6x30 screw	[56]	36	2 592 CZK
	M6 washer	[56]	72	914 CZK
	M6 nut	[56]	36	375 CZK
	O-ring 415x5	[61]	1	194 CZK
	M8x16 screw	[56]	1	91 CZK
	M8 washer	[56]	1	18 CZK
	M10x25 screw	[56]	12	3 222 CZK
	M10x35 screw	[56]	12	2 670 CZK
	M10 washer	[56]	24	240 CZK
Table	Profile 40x40x455	[62]	8	-
	Angle fix 40x40	[62]	8	-
	T-nut	[62]	24	-
	M8x16	[56]	24	2 184 CZK

*EUR-CZK exchange rate used was 24.74 CZK and EUR-USD exchange rate used was 22.72 CZK

8. Test procedure

This procedure must be followed, to ensure good and repeatable results from the test. The procedure covers everything from test preparation, step-by-step test procedure and results evaluation. The preparation and the evaluation of data is taken directly from the two standards [6; 11], while the procedure is specific for this system. The two standards can also be consulted for additional information.

Before every test, the specimen cups, collector plates, separator plate and the collector plate holder must be cleaned. The specimen cups and the collector plates are to be vapor degreased using a 1:1:1 by volume chloroform-acetone-ethanol solvent. The separator plate and the collector plate holders must be cleaned with a 1:1 by volume acetone-ethanol solution.

Additionally, the rest of the apparatus must be visually inspected, if there is not any residual contamination. If there is any, the contamination must also be cleaned with a suitable solvent, like a 1:1 by volume acetone-ethanol solution.

Once cleaned, the whole apparatus must be baked out at 150 °C and 10^{-3} Pa for at least 4 hours. This is done without the cooling system turned off and the manual valve connecting the nitrogen system closed. Once the bake out is complete, the system must be allowed to cool down to room temperature before the test.

During all operations with the samples or the collector plates, nitril gloves must be used. The prepared samples must be 100-300 mg in weight and must be able to fit in the sample boat (diameter of 12 mm, figure 71). In case the test has to be repeated, a total of 12 g of the tested material should be initially prepared in the conditioning chamber.

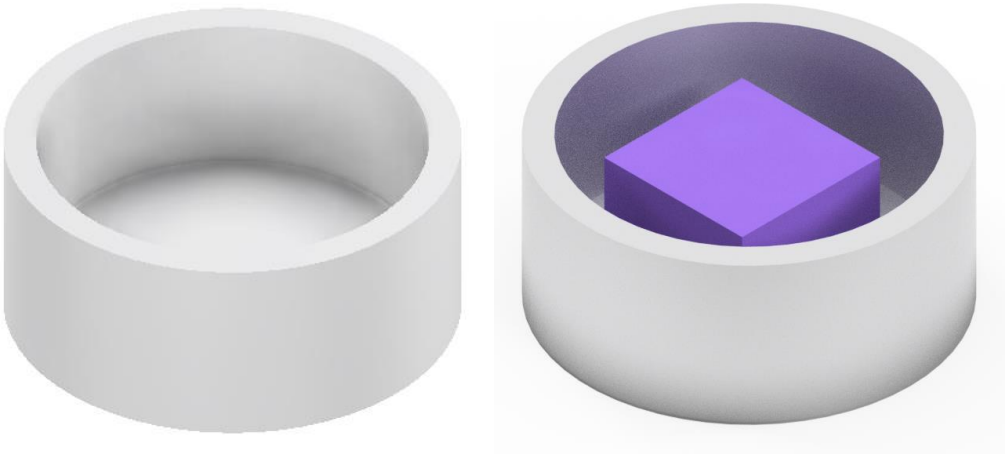

Figure 71: Aluminum sample boat with (right) and without (left) a sample.

Test procedure:

1. The sample boat is weighed.
2. The material sample is placed in the sample boat and is conditioned at 23 °C and 50 % relative humidity for 24 hours.
3. After 24 hours, the material sample is weighed on a microbalance.
4. The material sample with the sample boat is placed in the sample compartment of the apparatus.
5. The sample compartment is closed and the two positioning screws of the middle assembly of the inner apparatus are tightened.
6. The collector plate is weighed and placed in the apparatus.
7. The screw holding the collector plate is tightened.
8. The inner apparatus is closed.
9. The vacuum chamber is closed, and all screws are tightened.
10. The system is evacuated to below 10^{-3} Pa.
11. The heater pads and the cooling system are turned on.
12. The sample is heated up to 125 °C while the collector plate temperature is controlled at 25 °C.
13. Once the sample reaches 125 °C, the temperature of the sample and the collector are maintained for 24 hours.
14. After 24 hours, the heating pads are turned off.
15. The manual valve connecting the vacuum pump is closed.
16. The manual valve connecting the nitrogen is opened.
17. Using the thermal mass flow meter, the system is backfilled to 10-30 kPa with nitrogen.
18. Once the sample reaches 50 °C, the cooling system is turned off.
19. Using the thermal mass flow meter, the system is backfilled to atmospheric pressure with nitrogen.
20. The manual valve connecting the nitrogen is closed.
21. The chamber is opened, and the sample and the collector plate are stored in a desiccator using active silica gel descant.
22. After 30 minutes, the sample and the collector plate are weighed on a microbalance.

If WVR is required:

23. The sample with the sample boat is conditioned at 23 °C and 50 % relative humidity for 24 hours.
24. After 24 hours, the sample is weighed on a microbalance.

From the gathered weights, TML, CVCM and WVR are calculated with formulas from table 1. Historically, materials with TML higher than 1 % and CVCM higher than 0.1 % are deemed not suitable for flight hardware [6], however the acceptance limit can differ based on the specific mission and application.

9. Design requirements overview and fulfilment

As seen in table 18, all the imposed requirements from chapter 3.4. were fulfilled and the final design can thus be deemed a success. The table provides for each of the requirements a simple explanation of how it is fulfilled along with a chapter reference, where it is discussed in more detail.

Table 18: Status of each of the requirements – verification matrix.

	Requirement	Description
Restrictions posed by standards	R1 – Boat temperature	Chapter 5.1. – Temperature will be attained using resistance heating.
	R2 – Collector temperature	Chapter 5.2. – Temperature will be attained using a liquid cooling loop.
	R3 – Vacuum during test	Chapters 3.2.1. and 6.2. – Chamber pressure will be attained using existing pump system at BUT and by not exceeding the chamber volume limit.
	R4 – Precondition room temperature	Chapter 5.8. – Preconditioning room temperature will be attained by using existing BUT climate chamber.
	R5 – Precondition room relative humidity	Chapter 5.8. – Preconditioning room humidity will be attained by using existing BUT climate chamber.
	R6 – Sample mass	Chapter 8. – Sample mass is dictated by the procedure.
	R7 – Temperature monitoring	Chapter 5.3. – Monitoring will be done using thermocouples inside each effusion cell and next to each collector plate.
	R8 – Nitrogen backfill	Chapter 5.6. – A nitrogen bottle is connected to the test apparatus.
	R9 – Critical apparatus dimensions	Chapter 6.1. – Critical dimensions are implemented in the design.
	R10 – Microbalance	Chapter 5.7. – The chosen microbalance fulfils all requirements.
Existing equipment at BUT	R11 – Existing laboratory power supply	Chapter 5.1. – The thermal pads require 24V and 90W and are therefore compatible with the power supply.
	R12 – Existing vacuum gauge	Chapter 5.4. and 6.2. – An interface for the existing vacuum gauge is implemented.
	R13 – Existing vacuum pump system interface	Chapter 6.2. – An interface for the existing vacuum pump is implemented.
	R14 – Maximum volume	Chapter 6.2. – The final volume is well below the required 1200 liters.
Other requirements	R15 – Vacuum chamber material	Chapter 6.2. – The chamber will be made out of stainless steel.
	R16 – Effusion cell stacking direction	Chapter 6.1. – The stacking direction of the effusion cells is horizontal.
	R17 – Number of effusion cells	Chapter 6.1. – The design is for 13 samples.

Conclusion

Outgassing is a serious problem in the space industry, so a standardized testing procedure is in place since 1977. Therefore, an extensive database of materials exists. However, with the widespread adoption of 3D printing, a new wave of materials that could possibly be suitable for space has not yet been tested. The aim of this thesis was therefore to design a testing apparatus for BUT, so that these new materials could be tested in-house.

First, all relevant standards had to be studied, so that the device would comply with them. Four documents (three standards and one technical memorandum) in total were studied in the beginning of this thesis.

This was followed up by a quick overview of existing systems. These are unfortunately usually in private possession, so only a little information is available about them, mostly pictures.

With the standards and existing solutions studied, a set of requirements for the design was created. Some requirements also came from existing equipment that is already in use at BUT. A total of 17 requirements were created.

With the final requirements, a design process was then started. First, all the subsystems, like the heating of samples and the cooling of the collector plates were analyzed, and a diagram of what needs to connect where was created, to facilitate the following design.

This was then followed by a detailed design of the inner apparatus, including all connections and the hinge mechanism that opens the apparatus. With the inner apparatus designed, the vacuum chamber housing the apparatus was designed, including all feedthroughs connecting outside systems.

A price breakdown of all off-the-shelf parts and an estimation of the custom-made parts was presented, to give an idea of the cost of the system. This amounted to approximately 720 000 CZK.

Finally, a complete, step-by-step, procedure of the test and evaluation of results was presented.

The final design that was proposed is complete with all parts and a real system can be assembled following this thesis. The system also fulfils all previously established requirements.

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List of abbreviations

ASTM	American Society for Testing and Materials
BUT	Brno University of Technology
CAD	Computer aided design
CVCM	Collected volatile condensable materials
DOK	Dynamic Outgassing Knudsen cell
ECSS	European Cooperation for Space Standardization
ESA	European Space Agency
FEM	Finite element method
NASA	National Aeronautics and Space Administration
QCM	Quartz crystal microbalance
QTGA	Quartz crystal microbalance thermogravimetric analysis
RML	Recovered mass loss
TML	Total mass loss
VBQC	Vacuum Balance Quartz Crystal
WVR	Water vapor regained

List of symbols

A	$[m^2]$	Area
ρ	$\left[\frac{g}{cm^3}\right]$	Density
e	[-]	Emissivity
U	$\left[\frac{W}{m^2 \cdot K}\right]$	Heat transfer coefficient
m	[g]	Mass
c	$\left[\frac{J}{g \cdot K}\right]$	Specific heat capacity
σ	$[W \cdot m^{-2} \cdot K^{-4}]$	Stefan-Boltzmann constant
T	[K]	Temperature
Q_E	[J]	Thermal energy
Q	[W]	Thermal power
t	[s]	Time
V	$[cm^3]$	Volume
m_s	$[g \cdot s^{-1}]$	Water flow rate

Appendix

A1 - Complete Apparatus Assembly.iam – CAD model (Autodesk Inventor 2024)