Design and Operation of an Oven for Supersonic Molecular Beams

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December 19, 2017

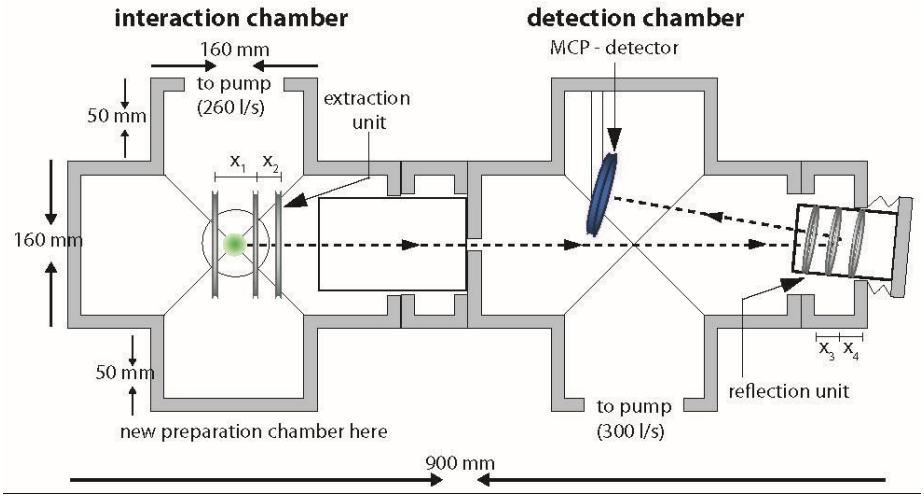

Figure 1: Main vacuum chamber before addition of the molecular beam.

1 Background

Molecular beams have had a major impact on many fields of study since their development in the 1920's. As you can probably imagine, having a collimated beam of molecules is a useful tool for many experiments. For over twenty years the vacuum chamber in lab 5 has been providing many students a workspace to pursue their Ph.D and masters studies, so it is only fitting that we outfit it with the latest and greatest technology.

Before this revamp, the machine had an effusive beam source at the bottom of the interaction chamber where it reads "new preparation chamber here" in fig 1. An open valve would allow one of several specified samples to evaporate into the chamber which would then be bombarded by laser pulses. Although this method works, it wastes large amounts of sample and fills the chamber with gas. A molecular beam, on the other hand, provides several benefits.

1. The sample is concentrated along the centerline of the beam.
2. Less sample is ultimately used.
3. The pressure in the interaction chamber will be lower.
4. We will be able to study many more substances.

Unfortunately, this project also brings many technical challenges. This paper is intended to walk through said challenges and how we ultimately overcame them.

1.1 How Molecular Beams Work

A molecular beam is created by allowing a moderately pressurized gas - 1-10 Bar usually - to pass through a very small aperture into vacuum conditions. The act of passing through this opening and expanding into a space with a pressure around 10^{-4} mBar cools the gas down to a few Kelvin. This expansion also forces the configuration of the molecules to be identical; as these supercooled molecules will bump against each other during the expansion until they are all in a somewhat steady state [1]. Furthermore, any seed gas being carried in the expansion will be forced along the centerline, due to the overwhelming amount of surrounding carrier gas molecules bumping them inwards [4]. These three factors are extreme incentive for using molecular beams, and why they are used so extensively in research all over the world.

Over the next few sections we will look through how all the components of the Molecular Beam Setup were chosen and why they were chosen. This will allow you to see the whole picture as it came together and achieve a deep understanding of how the machine operates.

2 Components of the Machine

2.1 Choosing a Valve

At first we were heavily leaning towards a high repetition rate piezo-actuated valve [2] to deliver molecular beam pulses to the interaction chamber. After conferring with the company that offered this product, and soliciting advise from Dr. Fischer we determined that it was not suitable for this application. There are several reasons for this, but only two really stand out. First of all, the current valve model can not be heated and passing our warm seed gas through the small aperture would cause deposition of sample and eventual clogging of the valve. Secondly, the aperture was not small enough. At a minimum diameter of 100 microns, even with a very efficient duty cycle and operating at 1000 Hz the chamber would be under too much pressure causing the molecular beam to deteriorate.

A second, simpler idea was suggested by Dr. Fischer: a continuous beam from a smaller aperture. This was an attractive solution as it removed much of the complexity involved with the electromechanical valve. After some calculations to determine the required diameters, we ordered several sizes ranging from 1 to 100 microns from Lenox Laser.

The analysis is quite simple and I will outline it below. In these calculations we consider Argon, Neon and Helium as they are the candidate carrier gases which will make up most of the beam. The following equations are from Cyril Bernard Lucas in his book *Atomic and Molecular Beams: Production and Collimation* [3].

The density of the gas is given by, $n = 7.24 \times 10^{13} p/T \quad \text{mm}^{-3}$

The mean velocity of the gas is given by, $\bar{c} = 145.5\sqrt{T/M}$ $\quad \mathrm{ms}^{-1}$

Where p is the pressure in the chamber, T is the temperature in the chamber and M is the molecular mass of the carrier gas.

If A is the area of the orifice that the gas is escaping through, then the number of atoms through the orifice per second is given by,

$$N = nA\bar{c}/4$$

The following table shows gas loads for various carrier gases and nozzle diameters in mBar·L/s.

gas\nozzle diameter (um)	5	50	100
He	0.025	2.5	9.9
Ne	0.011	1.1	4.4
Ar	0.0079	0.79	1.8

With these numbers we can move on to our next section.

2.2 Choosing a Vacuum Pump

In order to maintain the integrity of a molecular beam, the expansion chamber must have a maximum pressure of about 10^{-3} mBar. As we will be using multiple nozzle sizes, it is best to be conservative with the expected gas load. After communicating with Pfeiffer Vacuum they suggested that we use a 2000 L/s turbo-molecular pump with a separate backing pump from the one we have now. With this setup and our expected gas loads, the pressure in the expansion chamber should be around 10^{-4} mBar or lower.

Unfortunately, this pump weighs upwards of 60 kg. This massive piece of equipment will require a solid support system.

2.3 The Chamber

Part of the support for the large pump will come from the chamber to which it is fastened. However, this chamber has many more duties to fulfill. It must provide a suitable geometry for setting up the molecular beam oven and transmitting it into the main chamber, and it must allow for auxiliary systems to be attached. For example, we need an ion gauge to monitor the pressure inside this chamber.

At first, I was imagining a cubical design with various port sizes on each of the six faces. This seemed reasonable, but I was quickly diverted by an expert at Pink Vak who told me that the stresses on the system would be too high. The required thickness of the chamber to avoid buckling at the center of these flat faces would be superfluous. Instead, using a cylindrical design, the stresses are evenly distributed around the chamber and therefore it will be thinner, lighter and cheaper.

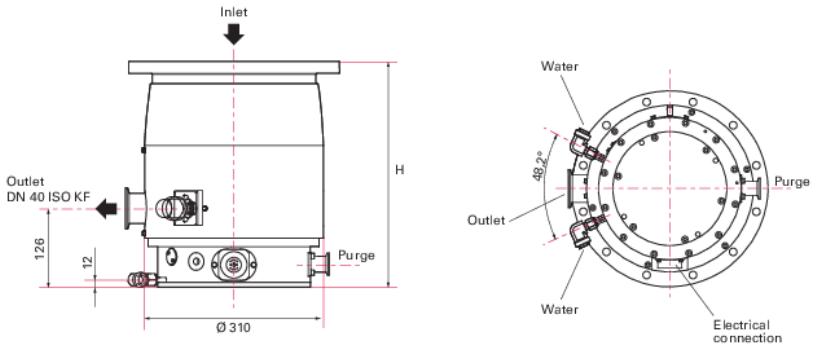

Figure 2: Dimension of the ATH 2300 from Pfeiffer vacuum. It's huge!

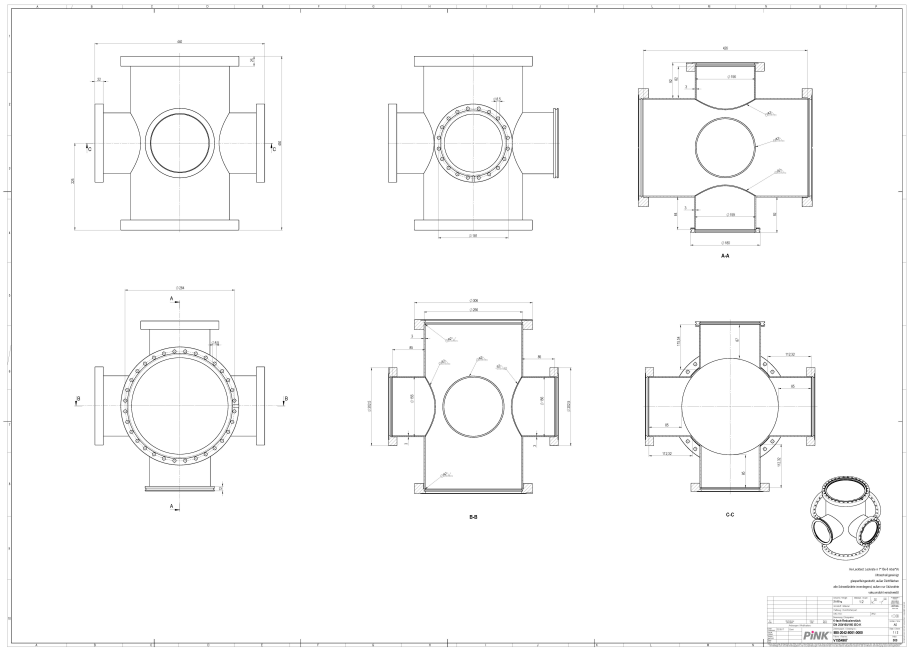

Figure 3: Dimension of the vacuum chamber.

We then ordered from Pink Vak a 6-way cross with two opposing DN250 CF flanges, three DN160 CF flanges and one DN160 Iso-F flange. The most important characteristic of this chamber, aside from the molecular beam, is the window port at the front. This high vacuum resealable window port will provide easy access to the molecular beam oven and the skimmer flange as well as any other equipment inside the chamber. This is extremely useful because the skimmer must be adjusted ever so slightly in order to line up perfectly with the molecular beam.

2.4 The Molecular Beam Flange and Skimmer

As shown in fig 6 the oven is fastened to an L-shaped support that is attached to the accompanying DN160 flange. This flange is equipped with gas and electrical feed-throughs which will supply the carrier gas and heating for the molecular beam respectively. This Molecular Beam Flange will sit on top of the Iso-F port and held in place by gravity and the vacuum itself. In order to optimize the throughput of the beam, we will have a system of screws that can nudge the flange ever so slightly which will effectively move the oven.

The oven is essentially a square block of aluminum. However, it is the most important part of the entire project because it is the single difference from the previous setup. Before, where sample was simply evaporating, now it is being evaporated. This subtle change allows for so many more interesting species to be investigated. To be fair, it was by far the most intricate piece, and required a lot of thought and discussion on how to make it work. I will discuss its design in the next section.

The gas exits the nozzle as a supersonic expansion. Roughly 10 cm downstream a skimmer is mounted to another DN160 flange which selects the center of the expansion to be transmitted into the main chamber. This skimmer flange is fastened in between the bottom facing port of the chamber and the Gate Valve. The skimmer itself has a ring clamp that will hold it in place and is designed to provide a bit of wiggle room. This wiggle room allows us to fine tune the positioning of the beam and skimmer opening so that we get a maximum beam throughput.

The skimmers themselves are made in house through an electroplating procedure. A conical electrode is dipped into a nickel plating solution at 50 degrees Celsius. The voltage applied to this electrode is raised periodically over several days. The newly formed nickel skimmer is periodically raised from the mixture and rinsed to avoid abrasion on the surface. It is essential that the skimmer surface is smooth for the integrity of the molecular beam.

2.5 The Oven

The oven design has had many iterations that began when we first wanted to use a pulsed valve. As the project progressed this has been the most challenging part to complete. The oven must achieve three important goals. One is to evaporate the substance we wish to study - these will be organic compounds that do not

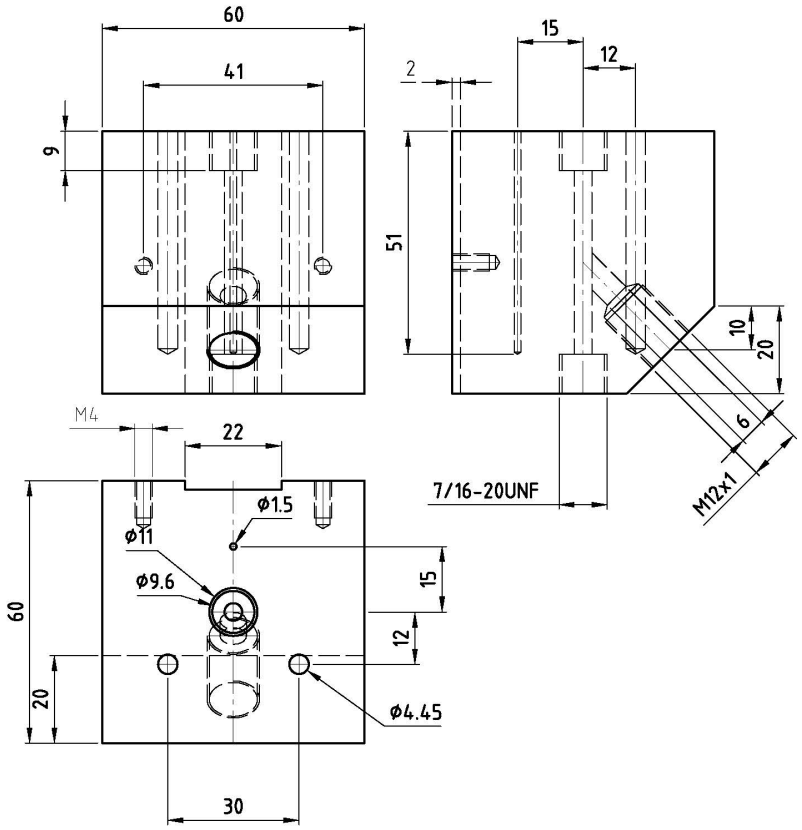

Figure 4: CAD drawing of the oven.

require a temperature higher than 400° Celsius. Second, the evaporated sample must be mixed with a seed gas that will constitute the majority of the molecular beam. Lastly, this mixture needs to be expelled from the oven as a supersonic expansion. I believe with this design we have met these tasks in an elegant manner.

As you can see from the right-most drawing in fig 4, there is a extrusion on the 45° cut. This will house a sample holder that screws in for a leak tight seal. this connects to the main channel which extends from the top of the oven to the bottom. Two channels are bored out for heating elements to be inserted which will control the temperature of the oven. Another thinner channel is for the thermocouple that will monitor the oven's temperature. At the top and bottom of the main channel are connections for 1/4" Swagelok pipe fittings. On the top a flexible vacuum tube will be attached and at the bottom, one of the several nozzles we discussed earlier.

The oven will be suspended downwards from the top of the chamber by an L-shaped metal support. There are two screw holes that will allow us to fasten the oven and various heights above the skimmer.

3 Method and Operation

In this section I will describe how each part works together in order to deliver the molecular beam.

3.1 System Architecture

The way that the parts are assembled is critical to this project. The first piece to be attached to the interaction chamber is the gate valve. This will sit atop the DN160 flange and serve as a seal for the interaction chamber when the vacuum in the preparation chamber must be broken. Next, on top of the valve is the skimmer flange which will select the center of the molecular beam. These two pieces create a sort of neck in between the interaction and preparation chambers. Each connection is sealed with a copper gasket as these seals will not be broken often.

The preparation chamber sits above the skimmer flange. It has two DN250 CF flanges and three DN160 flanges free for attachments. The frontal DN160 flange will house the high-vacuum resealable window that serves as a view-port and an avenue to adjust parts inside the chamber. The top DN160 flange is penetrated by the molecular beam flange and oven setup as shown in fig 6. The final DN160 flange currently has no planned usage and will be sealed with a blank. The DN250 flange on the left will be occupied by an ion gauge to monitor the pressure inside the chamber. Finally, the right DN250 flange will hold the large turbomolecular pump. This pump requires its own backing pump as well as a liquid chiller. The latter is the main reason we have to wait to begin building since it arrives in February 2018! As I said earlier, this pump will be secured by a custom support system.

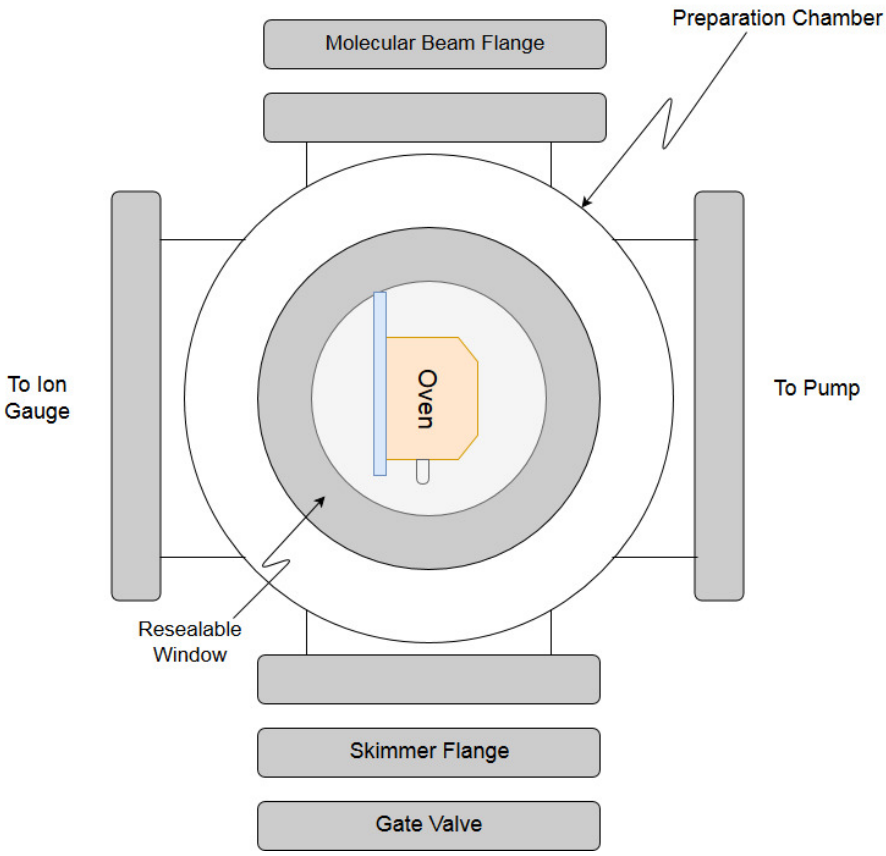

Figure 5: The preparation chamber architecture.

Two heating elements and a thermocouple are embedded within the oven - which is orange in fig 6. These are controlled via heater box on the outside of the chamber and connect through the electrical feed-through on the molecular beam flange. With these, the oven can reach a temperature of 400° Celsius. The seed gas - for most purposes Argon - will be directed into the oven via gas feed-through also on the molecular beam flange. The gas feed-through will be equipped with a valve near the flange in order to limit the volume the pump must evacuate when not running the gas.

The sample is held in a small container that screws into the oven on the 45° angle cut, shown in gray in fig 6. The oven is thermally insulated from the metal chamber with a ceramic block between the L-shaped support and itself, but this is not shown in the drawing. It can be moved to various heights by removing the screws on the left of the support.

3.2 Theory in Practice

With all the parts in place, the following is how the beam should operate:

1. A vacuum is achieved through pumping down the entire system.
2. The Argon is turned on to create an equilibrated gas flow.
3. The heating elements are set to the evaporation temperature of the sample.
4. The seed gas mixes with the carrier gas.
5. Our gas mixture expands into the preparation chamber and the center of the expansion is selected by the skimmer downstream.
6. The collimated beam enters the interaction chamber and crosses the laser path.

After entering the interaction chamber, a strong laser pulse excites the molecules. This is followed by a second delayed pulse which interrogates the dynamics initiated by the first. This generates ions which are analyzed via mass spectrometer. This structure can be seen in fig 7.

4 Conclusion

When I first arrived, we had little to no idea how a molecular beam was generated and propagated. After eight months of research, failed ideas and lots of coffee, we now have a stockpile of vacuum parts and highly customized apparatus that will perform that very task. Although I will not be here for the physical build, I hope this manual will help with the process.

I would like to thank my colleagues Sebastian Roeding and Hans-Peter Solowan for guiding me through this process. I learned so much from you guys and I hope that this addition works out for Hans-Peter's work! I would also like

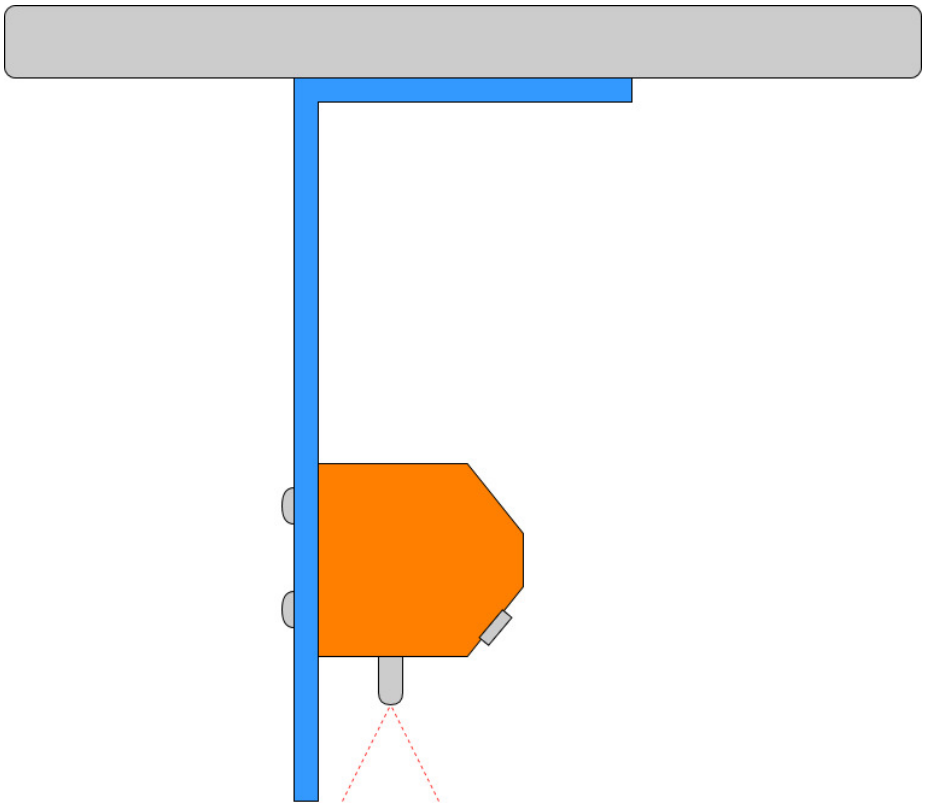

Figure 6: The molecular beam flange architecture.

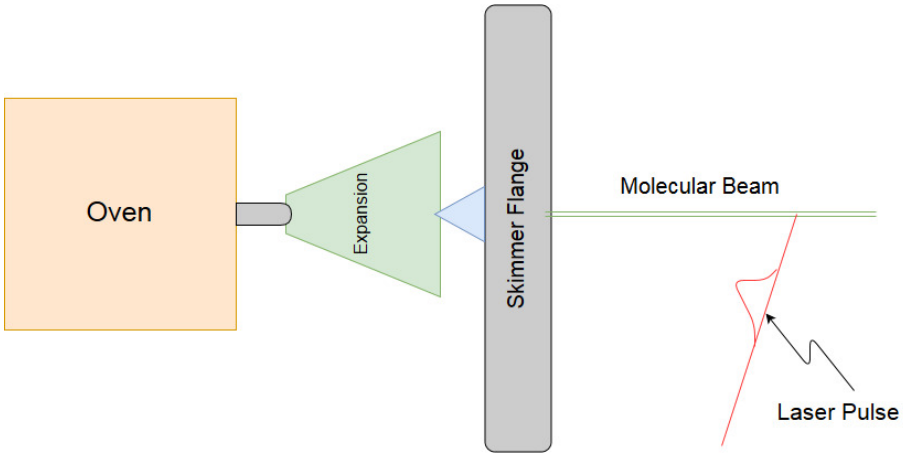

Figure 7: Expansion of the molecular beam (horizontal view).

to thank Prof. Dr. Tobias Brixner and Dr. Matthias Hensen for inviting me to work for their chair. This has truly been one of the greatest experiences in my life.

It seems like a lot of planning and work for such a small interaction, and it was. Hopefully, with the ability to investigate heavier molecules some new exciting data will be uncovered.

References

[1] H. C. W. Beijerinck and N. F. Verster. Absolute intensities and perpendicular temperatures of supersonic beams of polyatomic gases. *Physica B+C*, 111(2):327 – 352, 1981.

[2] Daniel Irimia, Dimitar Dobrikov, Rob Kortekaas, Han Voet, Daan A. van den Ende, Wilhelm A. Groen, and Maurice H. M. Janssen. A short pulse (7 us FWHM) and high repetition rate (dc-5khz) cantilever piezovalve for pulsed atomic and molecular beams. *Review of Scientific Instruments*, 80(11):113303, 2009.

[3] Cyril Bernard Lucas. *Atomic and molecular beams: production and collimation.* Taylor and Francis, Hoboken, NJ, 2013.

[4] Michael D. Morse. 2 - Supersonic Beam Sources. In F. B. Dunning and Randall G. Hulet, editors, *Atomic, Molecular, and Optical Physics: Atoms and Molecules*, volume 29 of *Experimental Methods in the Physical Sciences*, pages 21 – 47. Academic Press, 1996. DOI: 10.1016/S0076-695X(08)60784-X.