

# PHYS 3081 Lab Report: Vacuum and Mass Spectrometer\*

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The objective of this experiment is to find the relative abundance of isotopes of xenon and argon within a vacuum pipe system. The report outlines the function and operation of the equipment used to bring the pipe down to a vacuum and analyze the composition of the system. The experiment examines the capabilities of a mass spectrometer to find the concentration of distinct particles in a system. Generally, the data matched accepted values for the abundance of known isotopes, although there was greater error for isotopes of smaller concentrations. We look only at the values for a single set of measurements from the second week of experimentation in this report.

## I. INTRODUCTION

The goal of this experiment is to find the relative abundance of isotopes of argon and xenon in a vacuum tube, as well as examine the composition of the gases within the system to analyze the background. To do this, a pipe is connected to a series of pumps through valves which can be opened and closed to control the system. The sources of xenon and argon are connected to the end of the system, where they can be introduced through the valves. A residual gas analyzer (RGA) mass spectrometer is connected to the opposite end of the system, which can analyze the composition of gases and output the data into its associated software on the computer.

The data is plotted in a graph where the AMU (atomic mass unit) of the particle is graphed against the pressure (in Torr) of the particle in the system. As part of the process of analyzing the particles in the system (which is explained in the theory section), the RGA must ionize incoming particles. When the water molecules within the system are ionized, chemical bonds are broken and new molecules are formed from the atoms in water, creating distinctive peaks of hydrogen, hydroxide, and water on the RGA software. These peaks are then used to determine the calibration of the system, as they were consistently off by the same amount from known mass values of the molecules.

Not all isotopes of xenon and argon are present in significant quantities. Thus, from the nine stable isotopes of xenon, only five were detected in the system. All three stable isotopes of argon were detected, although we only found small concentrations of the Ar-36 and Ar-38 isotopes.

## II. THEORY

Xenon and argon are used for their properties which make it easier to study them than other molecules. Xenon is a noble gas, a group of elements which do not interact with other gases. It is significant for having nine isotopes of AMU 124 to 136 - the largest number of stable isotopes of any element - five of which are of a significant concentration in the Earth's atmosphere. Xenon is produced through supernovae due to the large amounts of energy produced during these events, which allows for endothermic fusion processes to occur and produce heavier elements.

Argon is another noble gas, but unlike xenon it is abundant in Earth's atmosphere. Argon comprises 1% of atmospheric gases, being several times more abundant than other noble gases in Earth's atmosphere. Argon has three stable isotopes, the vast majority in the atmosphere being Ar-40. Argon is produced in the Earth's crust through the decay of Potassium-40.

The background measurement shows the composition of the pipe prior to introducing xenon or argon into the system. The composition is expected to contain the various oils at 40 to 70 AMU, as well as oxygen, nitrogen, and nitrogen ions, and possibly the isotopes carbon-13 and oxygen-17. Water will be over represented in the system - as it is difficult to pump out - and in the mass

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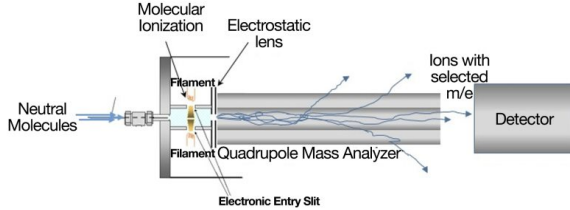
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spectrometer will ionize, producing hydrogen ions and hydroxide ions which show distinctive peaks.

A mass spectrometer detects the speed at which positively charged ions move through a vacuum chamber towards a negatively charged plate, which is determined by the mass of said ions. The mass spectrometer bombards the molecules with electrons, which ionize the molecule. The ion is repelled within the ion chamber, which accelerate according to their masses. A magnetic field deflects the ions, and the extent of the deflection is determined by their mass. Finally, the ions pass through the mass analyzer, where the charge is neutralized by an electron jumping from the metal plate to the ion. The electric current generated is proportional to the abundance of the ion.

The RGA (shown in Figure 1) is a form of mass spectrometer which can only operate at low pressures in the open ion source, below  $1^{-5}$  Torr. It is called open ion source as the ion source it uses - a filament wire and anode cage - are exposed directly to the system. Ions are then accelerated through the quadrupole (shown in Figure 2), which deflect ions as they pass through. Some ions are deflected out of the system, while others are moved towards the detector, which has the same function as a traditional mass spectrometer. The RGA measures the pressure of the molecules at a certain mass as determined by the detector.

FIG. 1. Residual Gas Analyzer Diagram, from mks.com



The quadrupole selects for ions which are directed through it. Two of the rods have a positive charge, and two have a negative charge. These rods are charged with radio frequency and direct current voltages. The DC voltage propels the ions down the quadrupoles. The radio frequency voltage creates an oscillating electric field which causes the particles to move with non-linear trajectories. The ions are filtered based on their mass-to-charge ratios. The computer translates the received data using the Mathieu function (equation 1), where  $u$  is the displacement,  $t$  is time,  $a$  is a parameter related to the radio frequency voltage,  $q$  is a parameter related to the electrostatic field, and  $\omega$  is the angular frequency of the radio frequency voltage.

$$\left\{ \frac{\delta^2 u(\omega)}{\delta t^2} + (a - 2q \cos(2\omega t))u(\omega) = 0 \right\} \quad (1)$$

The ion pump works by ionizing the gases which are in the vessel it is attached to and uses an electric field to

pull the ions into an electrode. The gasses are attached to the electrode and brought underneath it. The most common method of ionizing gasses in the chamber is by using an electron cloud which ionizes the atoms in the gas. The electron cloud is stored in a device called a Penning Trap, a device which uses a magnetic field and four swirling electric fields to store charged particles, as diagrammed in Figure 3.

FIG. 2. Quadrupole, from Wikimedia Commons

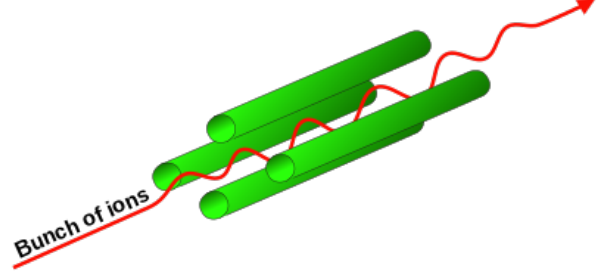
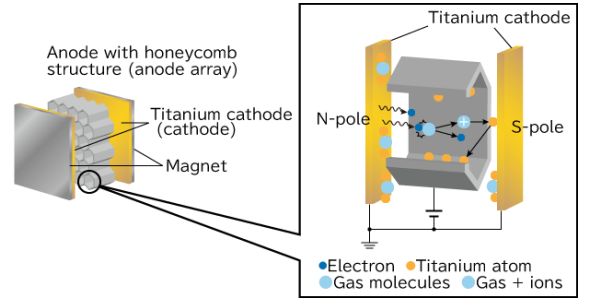


FIG. 3. Ion Pump Diagram, from matsusada.com



The electrons ionize incoming particles and are pushed with an electric field towards a cathode. The ions are either embedded within the cathode, or sputtered onto the cathode, which acts to evacuate the gas through chemisorption or physisorption (binding through chemical reactions or absorption onto the surface), which acts to pump the gases out of the system. Since the ion pump works by adding electrons to the valence shell of atoms in the gas, it is very poor at pumping noble gasses, as they have full valence shells and thus are not receptive to ionization.

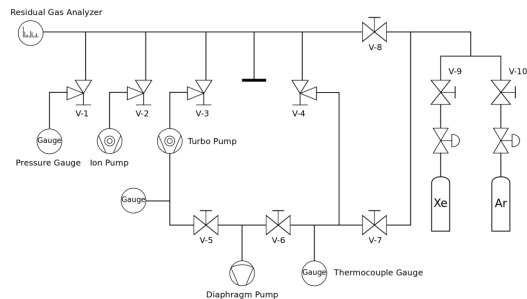
### III. METHODOLOGY AND DESCRIPTION

#### A. Equipment

The system is contained within a long metal tube (illustrated in Figure 4) which is connected to a series of equipment that alter the system. A series of valves are used to control the system labeled V-1 through V-10. The values are connected to the pumps which reduce the atmospheric pressure of the system, and the valves V-9 and V-10 are connected to the sources of argon and xenon. The valve V-1 is connected to the pressure gauge display;

V-2 connects to the ion pump; V-3 to the turbo pump; V-4 to the diaphragm pump. Connected to the end of the tube is the RGA.

FIG. 4. Valve System Diagram, from EKS lab manual



The diaphragm pump (Figure 5) is the first pump which is turned on to begin the process of building a vacuum in the chamber. The diaphragm pump uses a mechanical diaphragm which pushes air out of the system similar to human lungs. It pumps the system down sub-one Torr and maintains the upper bound of pressure for the system. The turbo (or turbomolecular) pump (Figure 6) is a small pump on the bottom of valve 3 which can only be turned on when the atmospheric pressure within the tube is less than 1 Torr. The turbo pump uses a fast moving fan which sucks air from the system. It reduces the pressure of the system down to  $10^{-5}$  Torr.

FIG. 5. Pfeiffer Diaphragm Membrane Vacuum Pump MVP 015-4, from highvacdepot.com



The ion pump (Figure 7) can only operate at low ( $10^{-5}$  Torr) pressures, as it collects atoms in the system and will fill up very quickly when there are many particles present, thus causing it to become unusable when subjected to higher pressure environments. The ion pump can bring the system down to  $10^{-10}$  Torr, however in this lab it was not necessary to bring it to this pressure so we did not use it.

The pressure gauge display shows the pressure in the system, measured in Torrs, and is connected to the V-1 valve. The turbo pump controller shows the frequency at which the turbo pump fan moves, and is used to adjust the turbo pump.

FIG. 6. Varian Turbo-V 81-T, ptbsales.com

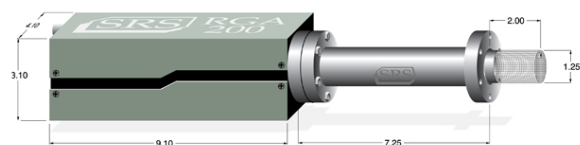


FIG. 7. Ion Pump, from idealvac.com



The SRS 200 Residual Gas Analyzer (Figure 8) is an open ion source mass spectrometer which measures the pressure of a system by detecting atoms as they pass through the quadrupole. It can only operate at pressure lower than  $10^{-5}$  Torr. Thus, both the diaphragm pump and turbo pump must be operating before it can be turned on. The RGA is attached to the vacuum chamber and is connected to the computer where the data is processed through the RGA software and output as a graph of AMU versus pressure in Torr.

FIG. 8. SRS 200 Residual Gas Analyzer, thinksrs.com



## B. Method

First the system must be rough-pumped to reduce the pressure to lower than one Torr. To do this, the valves V-1, V-3, V-4, V-5, V-6, and V-8 should be opened, and the values V-2, V-7, V-9, and V-10 should be closed. The diaphragm pump can then be switched on. The pressure

can be monitored from the pressure gauge adjacent to the diaphragm pump. When the pressure gauge reads lower than  $10^{-1}$  Torr, the values V-4 and V-3 should be closed and the turbo pump can be turned on from the turbo pump monitor. The turbo pump will spin until it reaches a certain frequency, after which Valve V-3 can be opened.

Once the system is at  $10^{-5}$  Torr, the RGA can be turned on and the RGA software can be loaded on the computer. The RGA must then be connected to the computer from the software, and then the program can be started. The information will be displayed on the screen as a graph of AMU versus pressure. This graph can be saved as an “ASCII” file to extract the numerical values.

The valves V-9 and V-10 control the introduction of xenon and argon into the system respectively. The RGA must be turned off prior to the introduction of gases into the system in order to not damage it. The system will quickly rise to  $10^{-3}$  Torr and must be reduced to  $10^{-5}$  Torr before the RGA can be turned back on and measurements can be performed again.

#### IV. DATA

##### A. Data Analysis

The background measurements were used to determine the calibration of the data. Since water and the various oils within the system are more difficult to pump out than other molecules, they created distinct peaks within the system. Water molecules (AMU 18) were ionized within the mass spectrometer, splitting the water into hydrogen (AMU 1) and hydroxide (AMU 17) molecules. These created distinctive peaks which were compared against known AMUs. These peaks were offset by 1.3 AMU, thus the data was translated over 1.3 AMU and the isotopes were selected from the known AMUs and then fit to a Gaussian function (Equation 2).

The relative abundance of the particles in the system was determined by summing the pressures of the individual isotopes to get the total pressure for xenon gas, and then dividing the individual pressures by the sum of pressure.

The data was translated into a graph which plotted AMU against the abundance of the atom within the system. From the known value of the AMUs of xenon and argon, we can infer what the abundances of the isotopes in the system are. The relative abundance was calculated by taking the abundance over the sum of the abundances, and these values were compared to the accepted values. The peaks of hydrogen and water were determined to be 1.3 AMU off from what was expected, so the graph was shifted accordingly. The peaks for each isotope were fit to a Gaussian function (Eq. 2) in Python, which took the maximum y- and x-values from each data set as arguments and an initial guess was provided for sigma,  $\sigma$ . A set of graphs were produced (Figures 9-16), from which

the maximum values and uncertainties were calculated.

$$\left\{ f(x) = \frac{1}{\sqrt{2\pi\sigma^2}} e^{-\frac{(x-\mu)^2}{2\sigma^2}} \right\}. \quad (2)$$

TABLE I. The five most abundant isotopes of Xenon and three isotopes of Argon listed with their relative abundances within the system, their errors compared to IUPAC measurements, and their uncertainties.

Isotope	Abundance (exp.)	Abundance (IUPAC)	Error	Uncertainty
Xe-129	27.89%	26.40%	5.793%	0.012
Xe-131	22.88%	21.23%	7.477%	0.030
Xe-132	20.24%	26.91%	24.75%	0.059
Xe-134	16.20%	10.44%	55.13%	0.018
Xe-136	12.80%	8.86%	44.63%	0.026
Ar-36	0.74%	0.33%	121.6%	0.009
Ar-38	0.22%	0.06%	239.2%	0.027
Ar-40	99.04%	99.60%	0.5662%	0.007

TABLE II. The five most abundant isotopes of Xenon and three isotopes of Argon listed with their pressures in the system.

Isotope	Pressure (Torr)
Xe-129	$4.01 \times 10^{-8}$
Xe-131	$3.28 \times 10^{-8}$
Xe-132	$2.91 \times 10^{-8}$
Xe-134	$2.33 \times 10^{-8}$
Xe-136	$1.84 \times 10^{-8}$
Ar-36	$3.76 \times 10^{-8}$
Ar-38	$1.10 \times 10^{-8}$
Ar-40	$5.02 \times 10^{-6}$

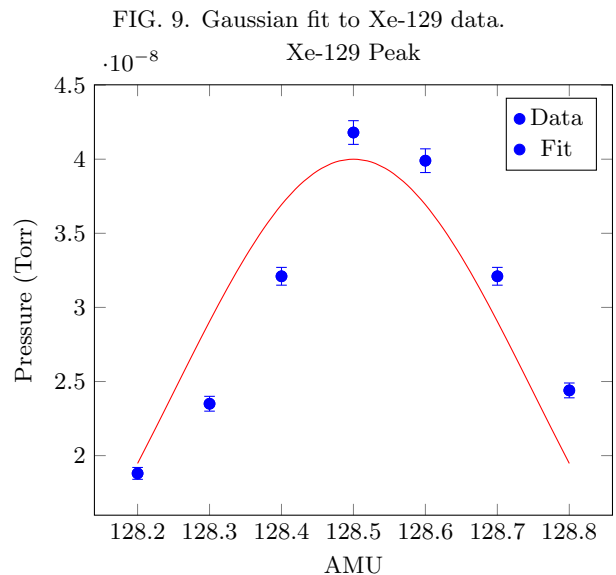


FIG. 10. Gaussian fit to Xe-131 data.

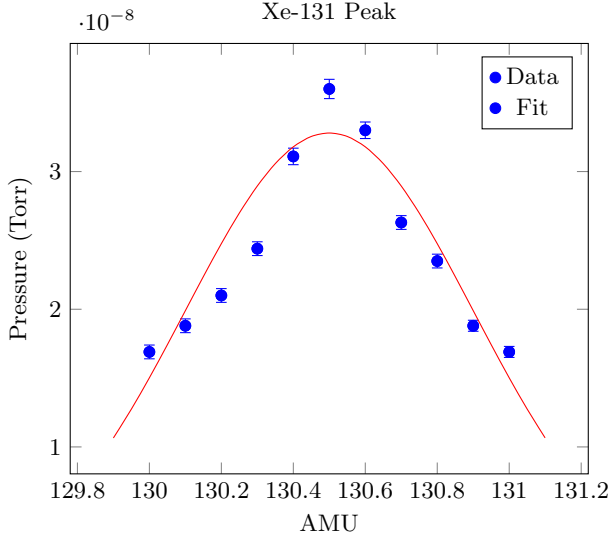


FIG. 12. Gaussian fit to Xe-134 data.

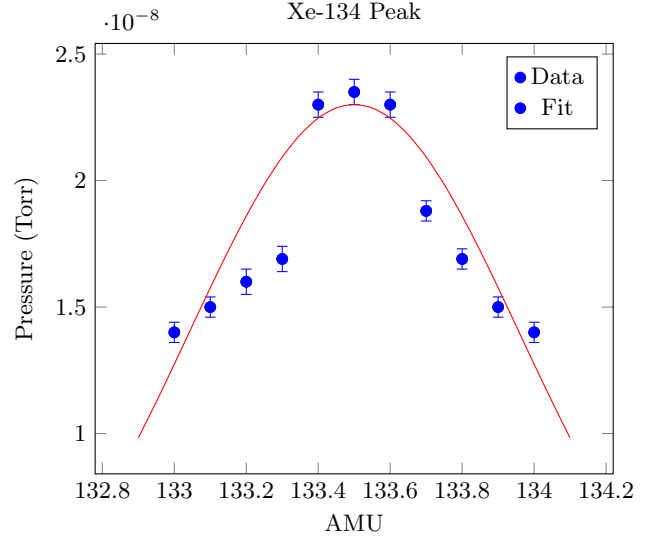


FIG. 11. Gaussian fit to Xe-132 data.

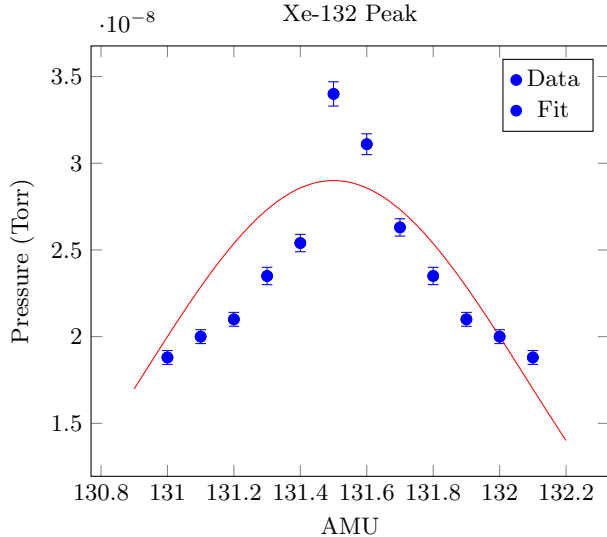
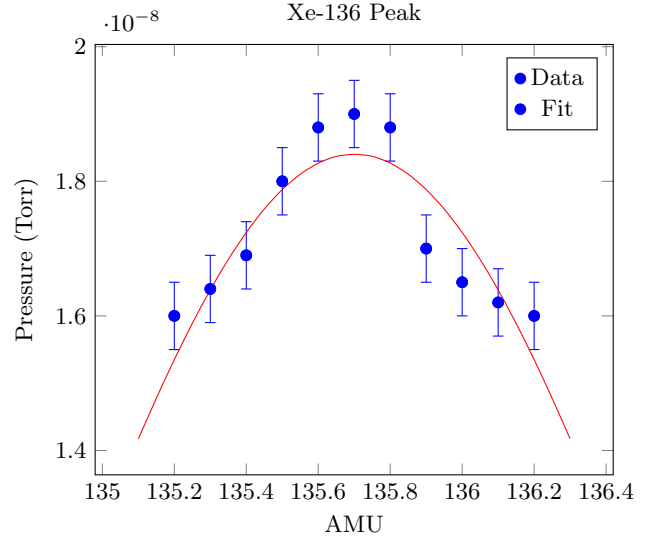


FIG. 13. Gaussian fit to Xe-136 data.



## V. DISCUSSION

The accepted values for the data are listed in Table I. Our error from these accepted values is listed in Table I. The percent error from the accepted values is acceptable for isotopes with larger abundances, but becomes larger for isotopes with smaller abundances. The error for Xe-134 and Xe-136 is around 50%, while the error for Ar-36 and Ar-38 is over 100%. This is likely due to the RGA being unable to effectively measure small concentrations with precision, as well as there being more background noise for molecules of small concentration to compete with.

The relative abundances of the atoms all fall within the values for the uncertainty and error bars for the data, although the error was far greater for the smaller values

of Argon and less abundant isotopes of Xenon (Ar-36 and Ar-38; Xe-134 and Xe-136). Figures 9-16 show the graphs for the Gaussian fits to the data sections determined by examining the ASCII files and selecting the for the peaks. A Gaussian was fitted to the data sets to determine the values of the peaks and the uncertainties of the fits were calculated and added to Table I.

### A. Errors

Due to the action of the pumps, the vacuum is not a constant environment. Gases from outside the system continuously seep in, causing small fluctuations in the pressure and gaseous composition of the system. This makes determining the composition of the system more

FIG. 14. Gaussian fit to Ar-36 data.

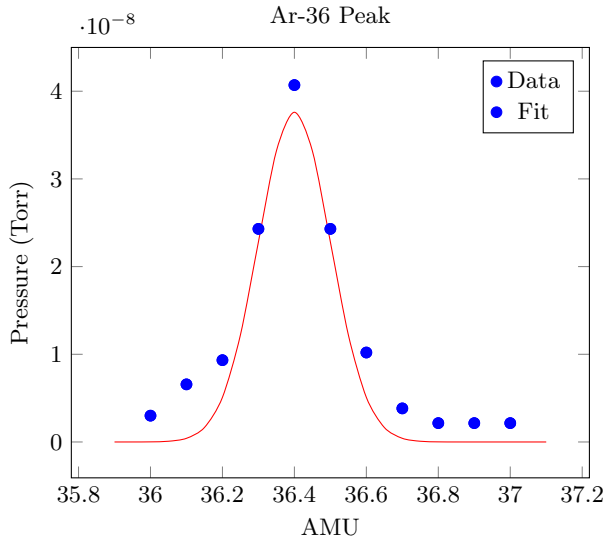
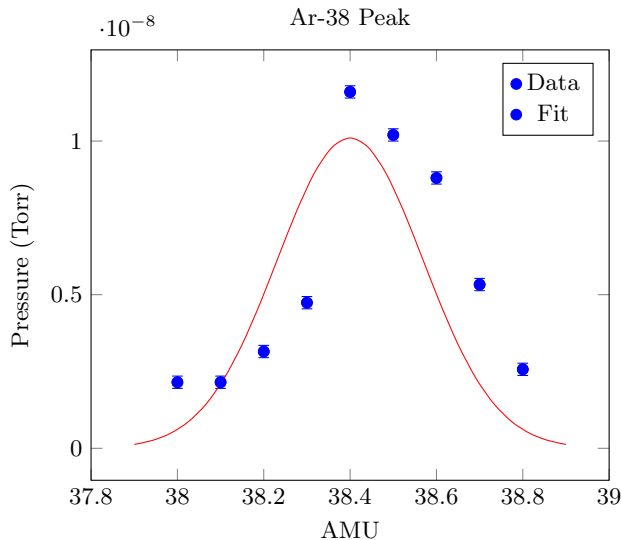


FIG. 15. Gaussian fit to Ar-38 data.



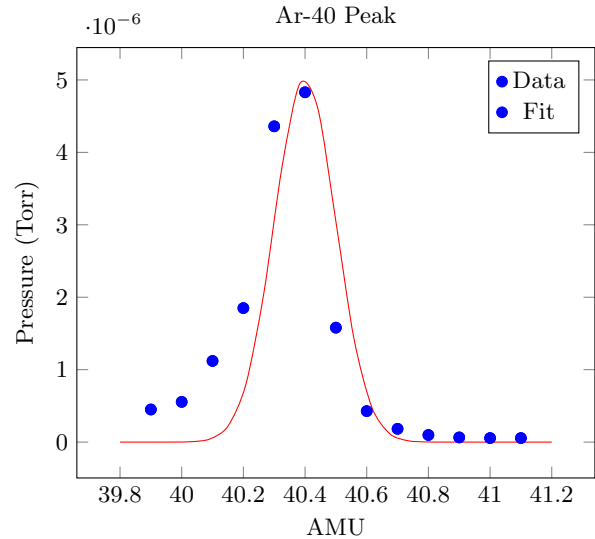
difficult, so multiple measurements have to be done and the background must be measured several times to check the composition of the system before and after the introduction of the gasses. This creates a small noise across the system which interferes with measurements. The measurement of less abundant molecules is difficult due to the precision of the RGA, which is unable to "sift out" less abundant particles from the background noise.

## VI. CONCLUSION

We learned from this experiment how to analyze the molecular composition of a system using a mass spec-

trometer, comparing known AMUs to the abundance peaks produced by the graphing software on the com-

FIG. 16. Gaussian fit to Ar-40 data.



puter. We additionally learned the mechanism behind the various pumps used to bring the system down to vacuum pressures necessary to prevent interference from molecules within the system which would give inaccurate peaks for the data. We learned how to use Python to fit data sets into Gaussian peaks and extract data using Python commands.

The lab could be expanded upon by using different particles - such as nitrogen oxide, methane, or carbon - and determining the abundance of the various isotopes of those particles. The ion pump could be used to reduce noise in the system and see if error can be reduced in the measurements. I would recommend this lab as it was useful for gaining insight into how the molecular composition of a system is found using a mass spectrometer.

## VII. REFERENCES

- Vacuum and Mass Spectrometry, E.K.A. Advanced Physics Laboratory Laboratory Manual*
- O'Hanlon, A User's Guide to Vacuum Technology, John Wiley Sons, 1989.*
- C. Rolfs and W. Rodney, Cauldrons in the Cosmos, University of Chicago Press, 1988.*

## Appendix A: Graphs

FIG. 17. Five peaks for xenon isotopes.

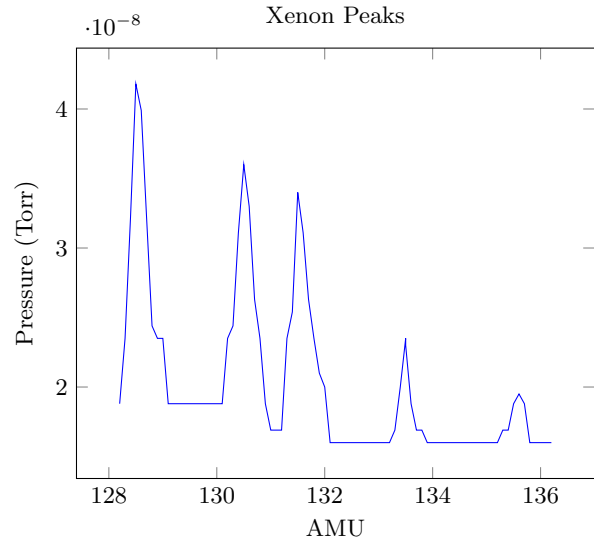


FIG. 18. The three peaks for the argon isotopes. Note the peaks for Ar-36 and Ar-38 aren't visible.

