## Lab 7: Residual Gas Analyzers

## Vacuum System Overview: Residual Gas Analyzers.

A <u>**R**</u>esidual <u>**G**</u>as <u>**A**</u>nalyzer (RGA) allows the scientist/engineer to measure the gases present in a low-pressure environment. This can be an extremely valuable measurement since it allows one to know the chemical species involved in gas phase reactions and can help one to determine which reactions are most important. Further, it can allow one to monitor the stability of the gas environment and determine when some aspect has changed. For examples: One could easily determine when a leak to the outside has developed, the gas bottle on the system was incorrectly installed, the gas itself was contaminated or the mass flow controllers (that set the flow of gas into the chamber) have gone out of calibration. This kind of information may be difficult to determine by other methods, but can easily be found using a RGA.

The operation of a RGA is conceptually quite simple although the mathematics of the quadrupole mass analyzer section can become too complex for many undergraduates to appreciate. This operation is diagrammed in Figure 1. First, an "ionizer" converts many neutral gas molecules into positive ions in a well-controlled region at a specified electric potential. These ions are next accelerated by a series of electrostatic "lenses" and formed into a beam that has about 20 eV of energy. The ion beam is subsequently passed into the quadrupole mass analyzer region. This region acts as a filter. It will very nicely pass through ions with a user chosen mass to charge ratio (M/e), but all the other ions get pushed aside into walls where they neutralize and become undetectable. The ions that are passed through this filter are detected as

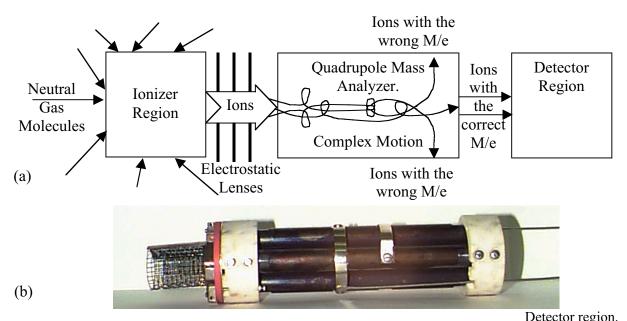


Figure 1: **Thanbasic** layout of a Residual Gas Analyzer. (a) Schematic diagram identifying thed.) **anajorns**omponents of an RGA and showing typical ion motion. (b) Picture of an old, half disassembled RGA showing the ionizer region (left) and the quadrupole rods. The detector region has been removed from the right hand side end. current either at a "Faraday cup" or using a secondary electron multiplier (also known as a "channeltron"). The channeltron gives a large amplification of the signal from ions and consequently is used to enhance the sensitivity of the RGA. By choosing a mass to charge ratio and making a measurement of the signal obtained, one can immediately find out the number of those molecules present in the ionizer region of the RGA. By sweeping through a whole range of M/e ratios, one can find a whole range of molecules that are present and begin to understand the full range of chemical components in the gas.

Such a spectrum is simulated in Figure 2. (This was not taken with an RGA. It is only a simplified example of what one could obtain.) There one can see that ions with mass to charge ratios of 14, 20, 28 and 40 amu are present. (An "amu" is an "atomic mass unit" and corresponds roughly to the mass of one proton.) These M/e ratios are characteristic of nitrogen and argon. 14 amu is  $N^+$ , 20 amu is  $Ar^{2+}$ , 28 amu is  $N_2^+$ , and 40 amu is  $Ar^+$ . The argon signals at 20 and 40 amu demonstrate that argon is present in substantial quantities. The RGA baseline rests at  $10^{-10}$  Torr in this case, but approximately  $5 \times 10^{-7}$  Torr signal is present. We will have to discuss further what this signal level means, but it is sufficient to point out here that the signal from argon is both clearly above the minimum detectable limit and therefore unambiguous. The signal at 28 amu corresponds to molecular nitrogen in this case, but other molecules might also have contributed to this signal (or any other signal for that matter). In particular, carbon monoxide,  $CO^+$ , has the same M/e ratio as  $N_2^+$  to within the detection capability of any ordinary RGA and consequently is always detected at 28 amu with nitrogen. The differences between N<sub>2</sub> and CO come in the total spectrum of CO versus N<sub>2</sub>. N<sub>2</sub> will produce signal peaks at primaily 28 and 14 amu while CO will produce peaks at 28 (CO), 16(O) and 12(C) amu along with lesser peaks at 29( $C^{13}O$  and COH) and 13 amu ( $C^{13}$ ). These characteristics of the molecular break-up as well as of isotope effects are important for analyzing RGA data and we will spend some time in both learning them and using them in this lab.



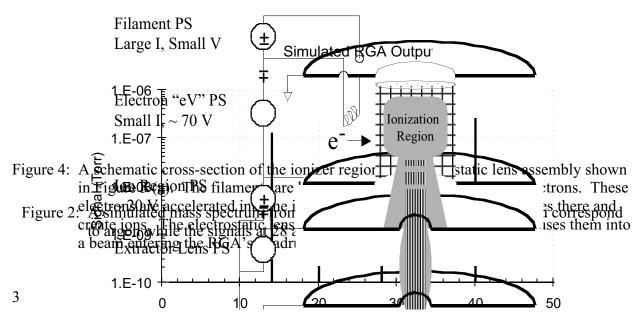
Figure 3: Three views of the ionizer-region and electrostatic lens assembly of the MKS RGA.
(a) A side view. The upper-most barrels are electrical connectors for the filaments. The top-most disk holds the two filaments (one on each side.) This top-most disk is comprised of two interlocking half-disks shown in (b) and (c). The filament is clearly visible in the picture of the underside of a half-disk shown in (c).

Let's go through and discuss the various sections of RGAs in more detail next.

1. "Ionizer" Region:

The ionizer region of the RGA we will use in this lab is shown in Figure 3. It consists of four primary elements: [1] Two filaments. (These are hidden in Fig. 3(a), but one is shown in Fig. 3(c) connected to the topmost half-disk.) [2] An electrostatic basket made of wire mesh (for setting up a constant electrostatic potential inside the ionization region.) [3] Insulating holders to give the whole thing a proper shape, and [4] an electrostatic lens assembly to be discussed in the next section. There is also a Faraday shield "basket" around the entire assembly to keep any external electrostatic potential from affecting the ionizer. These can all be seen in Figure 3. Figure 4 is a schematic representation of the equipment shown in Figure 3.

The filament wire hangs below the topmost disk as can be seen in Figures 3(c) and 4. This allows the filament wire to be heated white-hot by flowing a current through it. The Filament Power Supply (PS) provides this current and controls the temperature of the filament by it. A hot wire allows electrons to escape and this process is called "thermionic emission." The thermionic emission of electrons becomes ever more important as the filament is made hotter (>1000 C). The increasing thermal energy ( $k_BT$ ) of the electrons inside the filament as the temperature of the filament is increased, allows much larger numbers of electrons to overcome the metal work function and escape from the filament wire into the vacuum surrounding it.



These escaping electrons are then accelerated toward the wire basket of the ionization region by a potential difference setup between the filaments and the ion-region basket using the "electron eV" PS. The electrons gain as much energy (in eV) as the power supply Voltage. Once these electrons are accelerated to large enough energies, they can collide with any neutral gas molecule and ionize it. Since the electrons are accelerating between the ionization region basket and the filaments, most of the ionization occurs inside the basket. (The electrons only reach their largest energies AFTER they have been fully accelerated which is when they have reached the inside of the basket.) As a result, the ionizer is simply a device used to create ions at a known rate, at a known location (inside the basket) and at a known potential.

Several things can happen when an electron collides with a molecule. For example: the molecule can simply bounce the electron away in a different direction. This is called an "elastic collision" since it is nearly like a rubber ball (electron) colliding with a Cadillac. The energy of each remains about the same, but they are now moving in different directions. (Well, the rubber ball is at least.) The word "elastic" reminds us that an insignificant amount of energy went toward deforming either the electron or molecule. A second possibility is that the molecule can be ionized. The chemical equation for such an inelastic collision looks something like:  $e^- + M \Rightarrow$ 

 $2e^{-} + M^{+}$ . This ionization reaction requires significant energy (the "ionization potential" of the molecule M) and that energy must be supplied by the electron. Table I gives the energy required to ionize a variety of molecules into the specified ions. Other kinds of ionization reactions can also occur. For example a process called dissociative ionization can occur. The chemical equation for such a reaction would look something like:  $e^{-} + MX \Rightarrow 2e^{-} + M^{+} + X$  (or)  $\Rightarrow 2e^{-} + M^{-} + X$ 

 $M + X^+$ . Clearly, things can get complex quickly. Indeed, an electron collision with a nitrogen molecule (N<sub>2</sub>) can have well over a dozen possible outcomes! We only measure 2 using an RGA: The formation of N<sub>2</sub><sup>+</sup> and N<sup>+</sup>. Can you write down these chemical reaction equations?

Scientists use a concept called "cross-section" to describe the probability of an electron reacting with a molecule. The simplest form of the idea is relatively simple: an electron has to collide with a molecule to initiate a reaction. Since the electrons move at least 100x faster than molecules, we can often treat those molecules as if they were standing still. If the molecule has a LARGE radius (and therefore a large cross-section) the probability of an electron colliding with it is relatively large too. If the molecule is small, its' cross-section is small as well. This is far too simple a description, since cross-sections also account for various quantum mechanical effects during the collision as well as energy thresholds, but it gives one the basic idea. (Note that energy has to be conserved, so cross-sections are always equal to zero below the threshold energy required for the reaction to occur.) These cross-sections are generally measured as a function of energy of the electron and an example for the Argon atom is plotted in Fig. 5a, while an example for CF<sub>4</sub> is plotted in Figure 5b.

Table 1. The minimum Ionization Potentials (IP) of selected atoms and molecules. Atomic data from the Handbook of Chemistry and Physics, 66<sup>th</sup> edition, CRC Press (1985). CF<sub>4</sub> data from R. Bonham and M. Bruce, Argonne National Labs. N<sub>2</sub> data from Itakawa et al. J. Phys. Chem. Ref. Data 15, 985 (1986). Cl<sub>2</sub> data from Christophorou and Olthoff, J. Phys. Chem. Ref. Data 28, 131 (1999). O<sub>2</sub> data from Lieberman and Lichtenberg, Principles of Plasma Discharges and Materials Processing, Chap. 8, Wiley Interscience 1994.

Atom	1 <sup>st</sup> IP (eV)	2 <sup>nd</sup> IP (eV)	Molecule	Ion formed	IP (eV)
Н	13.598		CF <sub>4</sub>	$\mathrm{C}^+$	34.5
Не	24.587	54.416		$CF^+$	27
В	8.298	25.154		$\mathrm{CF_2}^+$	22
С	11.260	24.383		$CF_3^+$	16
N	14.534	29.601	$N_2$	$N_2^+$	15.6
0	13.618	35.116		$\mathbf{N}^+$	24.3
F	17.422	34.970		$N_2^{2+}$	~ 43
Ne	21.564	40.962	Cl <sub>2</sub>	$\operatorname{Cl}_2^+$	11.5
Si	8.151	16.345		$\mathrm{Cl}^+$	15.5
Р	10.486	19.725	O <sub>2</sub>	$O_2^+$	12.2
Cl	12.967	23.81		$O^+$	18.7
Ar	15.759	27.629	CO <sub>2</sub>	$\mathrm{CO_2}^+$	
Kr	13.999	24.359		$\mathrm{CO}^+$	

Note that all of the cross sections tend to have a maximum value near 70 eV to 200 eV! That is why we use about 70eV electrons to ionize molecules for the RGA. The ionization process is relatively efficient at that energy. Note also that the cross section for forming  $CF_3^+$  is much larger than for forming other ions like  $CF^+$  or  $C^+$ . Consequently, we should expect that the ionizer will produce much more  $CF_3^+$  from  $CF_4$  than  $CF^+$  or  $C^+$  and that the  $CF_3^+$  signal will be the largest as a result.

Many RGAs come with information on how their ionizer produces ions from various gases. For example: The MKS Instruments RGA that we are using in this lab comes with a small "cracking pattern" table describing the major mass peak and up to 3 minor mass peaks for each gas. Nitrogen has a major peak at 28 amu and minor peaks at 14 amu (5%) and 29 amu (1%).

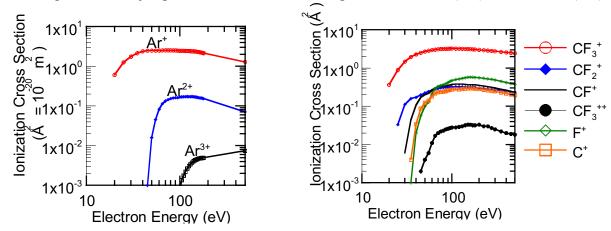


Figure 5: Electron impact ionization cross-sections for (a) Argon and (b) CF<sub>4</sub>.

The 29 amu peak could be due to <sup>15</sup>N (about 0.37% natural abundance) or to  $N_2H^+$ . RGAs also often come with a "Relative ionization sensitivity" table that describes the number of ions produced from a given molecule compared to an equal number of nitrogen molecules. For example: Argon produces 1.2 times as many ions as nitrogen and helium only 0.14. Consequently, one should expect smaller signals from helium at a given pressure than from either argon or nitrogen. The cracking pattern table and relative ionization sensitivity table for our MKS Instruments RGA is attached to the back of this lab section.

## 2. Electrostatic Lens Assembly:

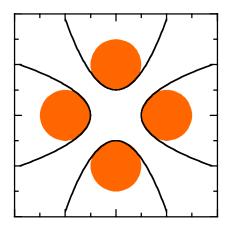
Once ions have been produced in the ionization region, an RGA is designed to accelerate these ions into the quadrupole mass filter. Electrostatic lenses are used to accelerate the ions out of the ionization region and focus them into a beam appropriate for the mass filter. Each lens is a simple disk with a hole in the center, a donut in shape. When a potential is placed on such a donut structure, electric fields are formed that can accelerate ions through it as well as push them toward the center of the donut hole. (You can solve Laplace's equation to show what this electric field looks like.)

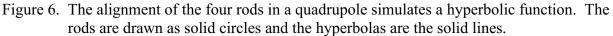
The potentials placed on the various lenses determine the efficiency with which ions are accelerated into the mass filter as well as the beam quality. The ion-region basket (the basket around the ionization region shown in Fig. 4) is generally set to a potential above ground by the ion-region Power Supply. This is often around 10 to 20 V, but not always. Setting the ion region basket at a potential above ground ensures that all the ions produced there have a significant potential energy. This is important because the mass filter region is usually referenced to a ground electrostatic potential. Any ion produced at a positive electrostatic potential, which then moves to a ground potential region, will have significant kinetic energy. Indeed, assuming the ion made no collisions, the kinetic energy would simply be the potential difference times the ion charge.  $KE = Q^*(V_{ion\_region} - V_{filter})$ . Thus, ions produced in the ion region basket will accelerate into the grounded mass filter region and travel through it at a reasonably well-defined velocity (and KE) simply by setting the potential on the ion region basket.

The lens nearest to the ion region basket is usually called an "extractor" lens. (See Figure 4.) Its' purpose is to encourage ions produced in the ion region to move toward the quadrupole mass filter. As such, it is generally biased at a potential that attracts positive ions. (Negative potential with respect to the ionization region.) The ions would move in all directions nearly equally without this lens, since the ion region basket is supposed to form a region with a nearly constant electrostatic potential. The extractor lens disturbs this constant potential by a small amount, enough to pull most ions produced there toward the mass filter region. As such, it can greatly increase the signal from a RGA. Some RGAs have only the extractor lens and that is all. Others will have a 2nd or even 3rd and 4th lens for focusing the ion beam and directing it into the mass filter with optimal efficiency.

## 3. Mass Filtering: (Quadrupole mass filters and magnetic sector filters.)

Quadrupole mass filters consist of 4 ("quad") rods that are electrically biased (poles). No magnetic fields are required to filter out different mass ions for this arrangement. A picture of the quadrupole region is shown in Fig. 1 where 3 of the 4 rods can clearly be seen. The 4th rod is hidden from view in that picture. These rods are extremely carefully placed so that they approximate a hyperbolic configuration in the center of the filter. (See Fig. 6 where the solid circles represent the rods and the lines are hyperbolic functions.) This hyperbolic configuration





of rods, when biased with both dc and rf voltages, produces fields that confine very small ranges of M/e (mass to charge ratio) to the central region. All other M/e ions are accelerated right into the rods where they are neutralized and become undetectable. The mathematics describing the motion of ions in this configuration includes the "Mathieu equations" and would require at least 4 pages of intense differential equations and calculus to explain. We will consider it beyond the scope of this discussion. A useful reference, however, is the report by P. H. Dawson and N. R. Whetten from the General Electric Research and Development Center in Dec. 1968 entitled "Mass Spectroscopy using radio-frequency quadrupole fields." (Report No. 68-C-418.) 4. Ion Detection System.

Once specific M/e ions have been passed through the filter, the RGA must detect them and count them. There are two ways an RGA can detect ions passing through the filter. The first makes use of a very simple electrical structure called a "Faraday cup" detector that is present in nearly all RGAs. This is the only detector we have present on our RGA. The second makes use of an advanced amplifier technology called a "Channeltron<sup>®</sup> Multiplier" or "Electron Multiplier." This was an option on our RGA, which we did not choose to purchase, but can allow one to measure trace gases with much improved signals.

A schematic representation of a Faraday cup is shown in Fig. 7 (a). It is simply a piece of metal biased at an appropriately negative potential ( $\sim -50$  V) so that the positive ions passing

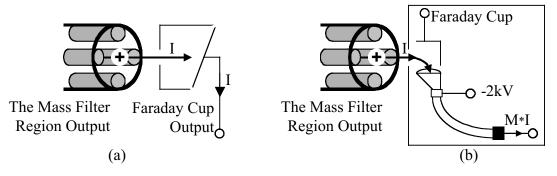


Figure 7. Two methods of ion detection: (a) using a Faraday cup to measure the ion current (I) directly and (b) using an electron multiplier to measure the ion current after amplification by a factor of M. The value of M can be on the order of 10<sup>8</sup> for a good electron multiplier.

through the mass filter are attracted to it and neutralized. The current (I) flowing through the Faraday cup is measured using a fast electrometer and this current is the relative signal recorded by the RGA. The larger the current, the larger the signal and the more ions must have been present. Since current is simply the net flow of charge across a unit area plane per unit time, and since a multiply ionized molecule carries more charge than a singly ionized molecule, one should expect multiply ionized molecules to produce a larger signal for a given number of ions. For example: each  $Ar^{++}$  ion carries twice the charge of an  $Ar^{+}$  ion; consequently the  $Ar^{++}$  signal should be twice as large as the  $Ar^{+}$  signal if there are the exact same numbers of ions. This can cause the signal of multiply charged ions to be larger than that predicted by just using the ionization cross section in Fig. 5.

A schematic representation of an electron multiplier is shown in Fig. 7 (b) as well. Electron multipliers are simply bent glass tubes with a special interior coating and a wide "mouth" at the front end. The back end has a Faraday cup installed on the tube that collects the electrons produced inside the tube. The front end has a collar that is connected to a low current power supply and is biased to a very large and negative voltage ( $\sim -2 \text{ kV}$ ). The back end is kept very close to ground potential and connected to a fast electrometer again. In some cases, the back-end can be connected to a pulse counting circuit instead so that the current pulse from each individual ion is counted rather than the time integrated current measured. This measuring procedure is rare for RGAs however. Most often, the current is simply measured. The advantage of an electron multiplier is that it amplifies the current of each ion by a very large factor. As a result, the current exiting the multiplier is M times larger than the ion current entering the front end. The value of the amplification factor, M, can be on the order of  $10^4$  to  $10^9$  depending upon the construction of the multiplier, the voltage used and the age of the device. Electron multipliers with amplification  $\sim 10^7$  are typical and much less than  $10^6$  indicates that a new part is needed.

Electron multipliers are extraordinary amplifiers and so we will take a moment to describe their operation even though our RGA does not have one. Figure 8 shows a schematic cutaway view of the interior of such a device. The glass walls are coated with a special material (usually an oxidized metal such as Pb or Bi) that emits several electrons every time it is hit by either an ion or an electron. This is an interesting phenomenon called "secondary electron emission" and in this case results in several charges leaving the surface every time one charge hits it. When an

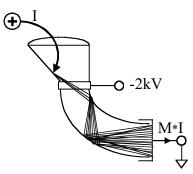


Figure 8. A schematic drawing of amplification in an electron multiplier. An ion enters the front-end "mouth" of the tube carrying current, I. The large negative voltage near the mouth causes the ion to strike a wall inside. The ion impact shown here released 3 electrons (three lines on the picture) from the wall. The voltage causes these electrons to move down the tube striking the wall further down. The process repeats until M electrons are collected at the back-end giving rise to M\*I current.

ion reaches the front-end "mouth" of the electron multiplier then, it collides with this surface. As a result, several electrons are emitted from the surface and into the vacuum. Those electrons see the ground potential at the backend as the most positive potential around and therefore accelerate towards the back-end. They are unable to curve with the tube however, so as they accelerate, they also collide with the walls. Each time they collide, they produce several more electrons doing the same thing! The net result is that M electrons arrive at the back end instead of just one ion and the ion signal is greatly amplified.

- 5. Differential Pumping System. Calibrated Leaks and Pinholes. We aren't using these during this semester, so we won't discuss them in this manual.
- 6. Control Software.

See the attached experimental procedure.

7. Example of analyzing RGA data.

Figure 9 is a plot of the first RGA scan that was ever made in the "Indy" reactor. The gate valve over the turbo-molecular pump was full open and the mass flow controllers were all turned off so that this scan showed the baseline components of the gas in the chamber. Note that there are major peaks at  $18(H_2O)$ , 17(OH),  $28(N_2 \& CO)$  and  $44(CO_2)$  amu. The next largest peaks occur at  $32(O_2)$  and 16(O) amu along with peaks at  $2(H_2)$ ,  $29(N_2 \& N_2H)$ ,  $41(hydrocarbons perhaps C_3H_5)$ ,  $43(hydrocarbons perhaps C_3H_7)$ , and 14(N) amu. The spectrum demonstrates that there is serious out-gassing of water vapor continuing in this chamber. Indeed, to stop this water vapor out-gassing would likely require us to bake the chamber for several hours as well as replace several of our viton gasket seals with copper metal seals.

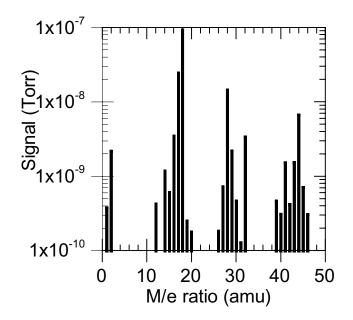


Figure 9. The first mass spectrum ever obtained from Indy. The pump was fully open and there was no intentional gas flow, so this spectrum describes the background gases present in the system. The background pressure was approximately  $2x10^{-7}$  Torr and the spectrum shows that there is significant water vapor present along with N<sub>2</sub> and CO<sub>2</sub>.

Homework Problems:

- 1) Describe the reasons an ionizer is almost always used with an RGA.
- 2) Describe the advantages and disadvantages of using a channeltron detector.
- 3) Find the isotopes of xenon and their natural abundances. Using this information, draw the mass spectrum you would expect to obtain for xenon at 10<sup>-6</sup> Torr. (Assume that the ionizer will cause 10% of the available xenon to doubly ionize [Xe<sup>2+</sup>].)
- 4) Find the isotopes of chlorine and their natural abundances. Using this information:
  - a. Make a table of all possible masses for atomic (Cl) and molecular (Cl<sub>2</sub>) chlorine and the relative signal abundances for each. (Hint: The sum of the atomic abundances should = 1 and the sum of the molecular abundances should = 1 as well.)
  - b. Draw the mass spectrum you would expect to obtain for chlorine gas at 10<sup>-6</sup> Torr. Assume that the total signal due to atomic chlorine is half that due to molecular chlorine.
- 5) Do you expect to see signal due to Ar<sup>3+</sup> from a RGA with 70 eV electrons in the ionizer? Why or why not. Also, what M/e ratio would you expect to find Ar<sup>3+</sup> signal?
- 6) Analyze the mass spectrum shown in Figure P1.
  - a. Figure out which neutral atoms and molecules are producing the spectrum.
  - b. Figure out what fraction of the signal at 28 amu is due to  $CO_2$ .
  - c. Is pump oil back-streaming into the chamber to any significant degree?
- 7) The signals at 72, 28 and 16 amu are monitored as a function of time for an etch reactor running a chlorine based etch of aluminum. The presence of oxygen could severely impact the etch process while nitrogen is routinely used as a buffer gas for loading new wafers into the reactor. The results are shown in Figure P2.
  - a. Identify the 72, 28 and 16 amu signals. What molecules do they result from?
  - b. Explain what occurs just after 15 seconds. Why should that process step cause an increase in the 28 amu signal and decreases in 16 and 72 amu signals?
  - c. Explain what occurs just after 60 seconds. Why should that process step cause an increase in the 72 amu signal along with the 28 and 16 amu signals?
  - d. Why does the 72 amu signal decrease when the plasma is turned on?
- 8) 70 eV electrons are generally produced in the ionizer of RGAs because most ionization cross sections are largest for 70 eV electrons (or thereabouts.) Each molecule has a smallest

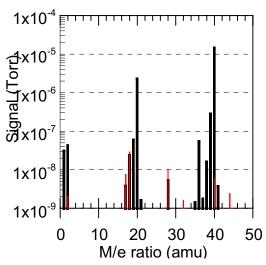


Figure P1. Comparison of mass spectra obtained from Indy under no-flow (thin red lines) and 1.5 sccm gas flow conditions (Thick black lines). You have to figure out the gas.

energy at which it will ionize called its "ionization threshold energy", however, so that the molecule will not be ionized if an electron below this threshold hits it. We can use this to our advantage in analyzing signals. By lowering the electron energy (but not the electron current) and measuring the decrease in the signal we can estimate the ionization threshold energy of the molecule producing our signal and thereby determine the identity of the molecule in cases where several molecules might be contributing to a single signal. Find the ionization threshold energy for  $CO^+$  from  $CO_2$  and  $N_2^+$  from  $N_2$ . Are they significantly different? If we can control electron energies to approximately 0.1 eV, can we use this technique to separate signals from CO and  $N_2$ ?

9) An ion is produced in the ion-region basket. The electrostatic potential in the ion-region is known to be 20V positive with respect to ground. What kinetic energy will that ion have if it subsequently travels to a region where the electrostatic potential is a negative 5 volts with respect to ground? Give your answer in both Joules and eV and assume the ion is singly charged argon.

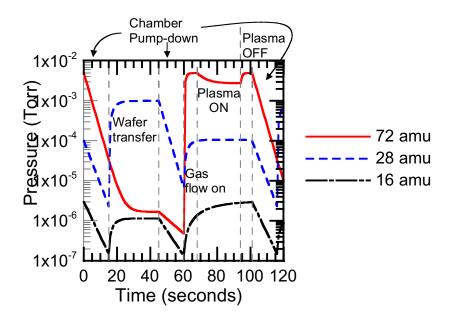


Figure P2. The time histories of signals at 72, 28 and 16 amu. Chambre pump-down begins at t=0 and ends at t=15 s. Wafer transfer occurs between 15 and 45 seconds after which the chamber is again pumped down. The process gases begin flowing at 60 sec and the plasma is turned on at 67 seconds for the etch. The plasma is turned off again at 94 seconds and the whole procedure begins again (with a chamber pump-down) at 101 seconds.

Lab Procedure:

- 1. Follow the procedure you learned to pump down Indy. Use the ionization gauge to measure the chamber pressure and make sure that the pressure is below  $10^{-4}$  Torr. You can start the RGA program once you are sure that the pressure is below  $10^{-4}$  Torr.
  - a. Make sure that the external RGA unit is plugged in and has power. The external unit is attached to a 2\_" CF flange at the rear left of Indy. It has a red Power LED on it that should be lit. It should have a cable to the computer as well.
  - b. Start the RGA program by going within the start menu to "Start/Programs/Spectra/RGA." Do NOT run the RGA-reset program by accident!
- 2. The first thing you will do is measure the mass spectrum with no gas flow. (Measure the pressure as well.) Look to see if there is any evidence of oil back-streaming from our mechanical pump. Follow the steps below to do this.
  - a. Record the total pressure measured using the ionization gauge.
  - b. Turn on the RGA filament by clicking on the filament (No. 1) button at the top of the RGA program window.
  - c. Choose the bar-chart display mode by clicking on the bar-chart button. (Note: the action caused by clicking each button can be found by watching the bottom yellow window as you position the mouse pointer over each button.)
  - d. Choose a logarithmic axis.
  - e. Choose accuracy setting 2
  - f. Set the top of the log axis to the pressure range measured using the ionization gauge. (Example: If the pressure is  $5 \times 10^{-7}$  Torr, make the log axis top to be  $10^{-6}$  Torr.) You can set the axis limits by pressing the "set first mass" button at the left hand corner of the second toolbar.
  - g. Set the bottom of the log axis to the pressure range / 1000. (Example: If the pressure is  $5x10^{-7}$  Torr, make the log axis bottom to be  $10^{-11}$  Torr { $5x10^{-10}$  rounds down to  $1x10^{-11}$ }.)
  - h. Let the RGA scan through all the decades of pressure at least once. (You will see the x-axis title alternate with the pressure decade being scanned.)
- 3. Save the no-flow data for future plotting.
  - a. Copy the data to the clipboard. Go to the file menu and choose "copy data to clipboard."
  - b. Paste the data to notepad. (Open an instance of notepad by going to the start menu and choosing Start/Programs/Accessories/Notepad.) Paste the data to the notepad.
  - c. Save the notepad document, being sure that the data has pasted into notepad correctly. This is your background scan. It will tell you what the residual gases in the chamber were before you began flowing gas. An example spectra is shown in Fig. 9.
- 4. Turn the bar-chart mode off by clicking on the bar-chart button.
- 5. <u>Turn off the RGA filament</u> to protect it against over-pressure in the next steps. <u>Turn off</u> <u>the Ionization gauge</u> to protect it against over-pressure during the next steps. Watch the chamber pressure using the capacitance manometer.
- 6. Enable gas flow on Channel 1 (Argon).
  - a. Make sure the argon gas bottle has been opened so that gas is in the line to the mass flow controller.
  - b. Enable gas flow by flipping on the gas enable switch. Allow the chamber to return to base pressure.
  - c. Enable gas flow by using the MKS 247 mass flow controller to allow 1.5 sccm of argon flow. Make the channel 1 set-point be 1.5 sccm and turn on gas flow.

- d. Let the chamber reach steady state. The pressure should remain below  $2.5 \times 10^{-4}$  Torr on the capacitance manometer.
- 7. Flow 1.5 sccm of Argon Measure the pressure and the new spectrum.
  - a. Measure pressure using the ionization gauge. Record the new pressure. It should be below  $2.5 \times 10^{-4}$  Torr.
  - b. Provided the pressure is below  $2.5 \times 10^{-4}$  Torr, turn on the RGA filament.
  - c. Measure the spectrum as before by setting up the top of the logarithmic axis to match the chamber pressure, but leave the log axis bottom the same as before.
  - d. Note any substantive differences between the two spectra (no-flow and Ar-flow.) Save the data to your disk.
- 8. Flow 3 sccm of Argon Measure the pressure and the new spectrum.
  - a. Measure pressure using the ionization gauge. Record the new pressure. It should be below  $2.5 \times 10^{-4}$  Torr.
  - b. Provided the pressure is below  $2.5 \times 10^{-4}$  Torr, turn on the RGA filament.
  - c. Measure the spectrum as before by setting up the top of the logarithmic axis to match the chamber pressure, but leave the log axis bottom the same as before.
  - d. Note any substantive differences between the two spectra (1.5 sccm and 3 sccm.) Save the data to your disk.
- 9. Turn the bar-chart mode off by clicking on the bar-chart button.
- <u>Turn off the RGA filament</u> to protect it against over-pressure in the next steps. <u>Turn off</u> <u>the Ionization gauge</u> to protect it against over-pressure during the next steps. Watch the chamber pressure using the capacitance manometer.
- 11. Enable gas flow on Channel 2 (Nitrogen).
  - a. Make sure the Nitrogen gas bottle has been opened so that gas is in the line to the mass flow controller.
  - b. Enable gas flow by flipping on the gas enable switch. Allow the chamber to return to base pressure.
  - c. Enable gas flow by using the MKS 247 mass flow controller to allow 1.5 sccm of  $N_2$  flow. Make the channel 2 set-point be 1.5 sccm and turn on gas flow.
- 12. Let the chamber reach steady state. The pressure should remain below  $2.5 \times 10^{-4}$  Torr on the capacitance manometer.
- 13. Flow 1.5 sccm of  $N_2$  Measure the pressure and the new spectrum.
  - a. Measure pressure using the ionization gauge. Record the new pressure. It should be below  $2.5 \times 10^{-4}$  Torr.
  - b. Provided the pressure is below  $2.5 \times 10^{-4}$  Torr, turn on the RGA filament.
  - c. Measure the spectrum as before by setting up the top of the logarithmic axis to match the chamber pressure, but leave the log axis bottom the same as before.
  - d. Note any substantive differences between the two spectra (no-flow and  $N_2$ -flow.) Save the data to your disk.
- 14. Flow 3 sccm of  $N_2$  Measure the pressure and the new spectrum.
  - a. Measure pressure using the ionization gauge. Record the new pressure. It should be below  $2.5 \times 10^{-4}$  Torr.
  - b. Provided the pressure is below  $2.5 \times 10^{-4}$  Torr, turn on the RGA filament.
  - c. Measure the spectrum as before by setting up the top of the logarithmic axis to match the chamber pressure, but leave the log axis bottom the same as before.

- d. Note any substantive differences between the two spectra (1.5 sccm and 3 sccm.) Save the data to your disk.
- 15. Turn the bar-chart mode off by clicking on the bar-chart button.
- 16. <u>**Turn off the RGA filament</u> to protect it against over-pressure in the next steps. <u><b>Turn off**</u> <u>**the Ionization gauge**</u> to protect it against over-pressure during the next steps. Watch the chamber pressure using the capacitance manometer.</u>
- 17. Enable gas flow on Channel 3 (Carbon Tetrafluoride).
  - a. Make sure the CF<sub>4</sub> gas bottle has been opened so that gas is in the line to the mass flow controller.
  - b. Enable gas flow by flipping on the gas enable switch. Allow the chamber to return to base pressure.
  - c. Enable gas flow by using the MKS 247 mass flow controller to allow 1.5 sccm of CF<sub>4</sub> flow. Make the channel 3 set-point be 1.5 sccm and turn on gas flow.
- 18. Let the chamber reach steady state. The pressure should remain below  $2.5 \times 10^{-4}$  Torr on the capacitance manometer.
- 19. Flow 1.5 sccm of  $CF_4$  Measure the pressure and the new spectrum.
  - a. Measure pressure using the ionization gauge. Record the new pressure. It should be below  $2.5 \times 10^{-4}$  Torr.
  - b. Provided the pressure is below  $2.5 \times 10^{-4}$  Torr, turn on the RGA filament.
  - c. Measure the spectrum as before by setting up the top of the logarithmic axis to match the chamber pressure, but leave the log axis bottom the same as before.
  - d. Note any substantive differences between the two spectra (no-flow and CF<sub>4</sub>-flow.) Save the data to your disk.
- 20. Flow 3 sccm of  $CF_4$  Measure the pressure and the new spectrum.
  - a. Measure pressure using the ionization gauge. Record the new pressure. It should be below  $2.5 \times 10^{-4}$  Torr.
  - b. Provided the pressure is below  $2.5 \times 10^{-4}$  Torr, turn on the RGA filament.
  - c. Measure the spectrum as before by setting up the top of the logarithmic axis to match the chamber pressure, but leave the log axis bottom the same as before.
  - d. Note any substantive differences between the two spectra (1.5 sccm and 3 sccm.) Save the data to your disk.
- 21. Turn the bar-chart mode off by clicking on the bar-chart button.
- 22. <u>**Turn off the RGA filament.**</u> <u>**Turn off the Ionization gauge.**</u> Watch the chamber pressure using the capacitance manometer.
- 23. Exit the RGA program.
- 24. Data Analysis:
  - a. Plot the 7 spectra obtained in this experiment.
  - b. Identify as many of the mass peaks as you can in each of the 7 spectra. Identify major peaks first. What gases are present? What are the major components of the gas present in the chamber in each case?
  - c. Identify the major isotopes of each atom present in the spectra. Do the less-abundant isotopes affect the spectra at all?
  - d. Identify any substantive differences between the spectra as noted in the lab procedure above.
    - i. Compare the no-flow spectra to the 1.5 sccm spectra for each gas.
    - ii. Compare the 1.5 sccm spectra for each gas to the 3 sccm spectra.

e. Compare the spectra of Argon and CF<sub>4</sub> to that predicted using the cross sections given in this manual. Do they follow expectations?