

Sample schedule - Mass spec, vacuum

Opening discussion + framing: What is mass spec/ what does it have to do with vacuum?

Traditional magnetic (sector?) spectrometer

Board schematic - hook into phys 200

Discussion of energy/length scales

Have students calculate cyclotron radius w/ thermal speeds.

Motivate accelerating ions and/or velocity selector.

Tour real device (demo unit from leak checker) - pros + cons

Quad - Intro + tour of apparatus

Mini lecture on operating principles

Brief intro to SimIon - have students fiddle, test effects of various parameter changes

Example scan

Discussion of cracking patterns

Ion mass spec - Tour of apparatus

Brief discussion of operating principle

Parallel sessions (switch + repeat as time allows)

I) Tear down portions of quad rig. Diagram + ask questions.  
Possibly swap in/out leak valve, gauge, etc  
Reassemble, pump down, leak check  
Mass spec scans + interpretation  
Possibly do injection of air, He, Ar

II) Same as above but for ion rig.  
Pump down more involved; change out fewer parts.

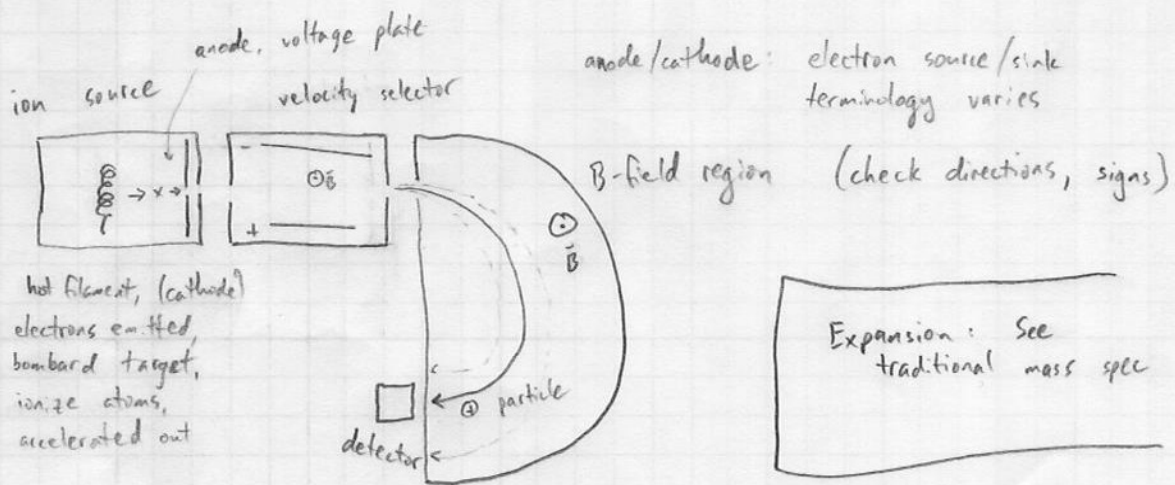
As time allows: mini-lectures on hot/cold cathode ion gauges  
(insert anywhere in schedule)

## Mass spec notes

Opening discussion question: Given an unknown substance, how do you figure out what it is?

Mass spectrometry uses electric & magnetic forces to separate charged ions. Basic method introduced in phys 200.

- 1) Ionize sample v.a electron bombardment
- 2) Accelerate particles through some potential
- 3) Steer ions w/known B-field - radius of trajectory depends on  $q/m$
- 4) Scan output & collect/count ions (or  $m/q$ )



B field region: Magnetic force acts as centripetal force

$$\vec{F}_B = q\vec{v} \times \vec{B}$$

$$qvB = mv^2/r$$

$$qB = m/r \Rightarrow r = \frac{m}{q} \frac{v}{B}$$

$v$  known from velocity selector, so net force when

$$qE = qvB$$

$$E = vB, v = E/B$$

$$E = \nabla/d \quad (d \text{ plate separation})$$

Ultimately,  $r \propto m/q$ .

Basic idea behind sector mass spectrometer, original scheme.

Still in use, can have great resolving power just by making larger

"Quick" cyclotron radius calculation:

$$r = \frac{m}{q} \frac{v}{B}$$

B from permanent magnets  $\sim 0.1$  T  
m for He  
q fundamental charge  
v from  $\frac{1}{2}kT = \frac{1}{2}mv^2$ , room temp

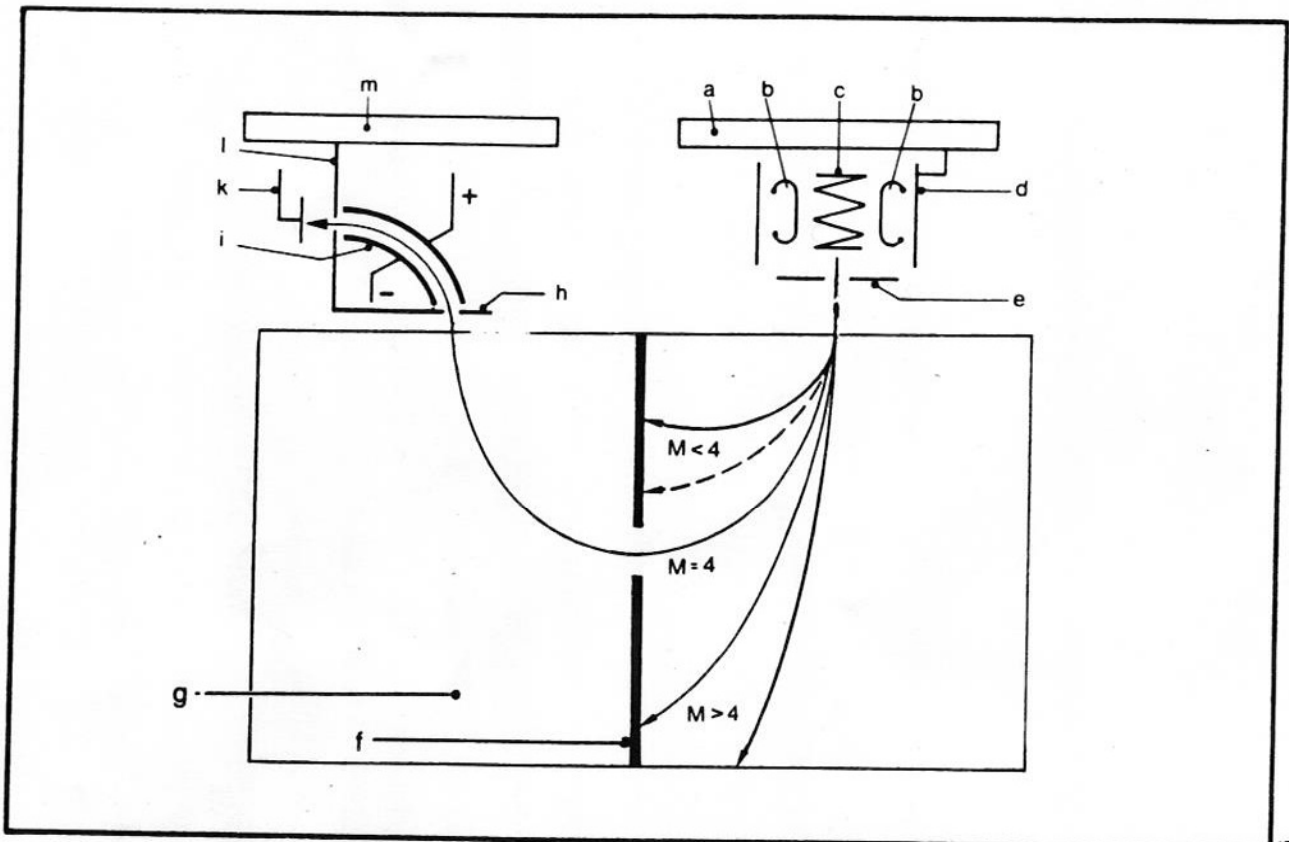
$$v \approx 1360 \text{ m/s}$$

$$r \approx 5.7 \times 10^{-4} \text{ m}$$

Distance between successive radii even smaller. Suggests increasing v by accelerating through potential difference.

Resolution can be improved (somewhat) arbitrarily by scaling device.

Big devices w/stray magnetic fields not always desirable -  
leads to development of other methods



- |   |   |
|---|---|
| a - Ion-Source Flange   | g - 180° Magnetic Deflection Field        |
| b - Cathodes (thoriated iridium)                                      | h - Inlet Slit to 90° Electrostatic Field |
| c - Anode (heated)  | i - Electrostatic Deflection Plates       |
| d - Ground Shield   | k - Ion Collector                         |
| e - Extractor Plate and Ion Collector for Pressure Measurement (PIII) | l - Ground Slit                           |
| f - Intermediate Slit   | m - Ion Collector Flange                  |

Figure 7-5 - Diagram of the Mass Spectrometer

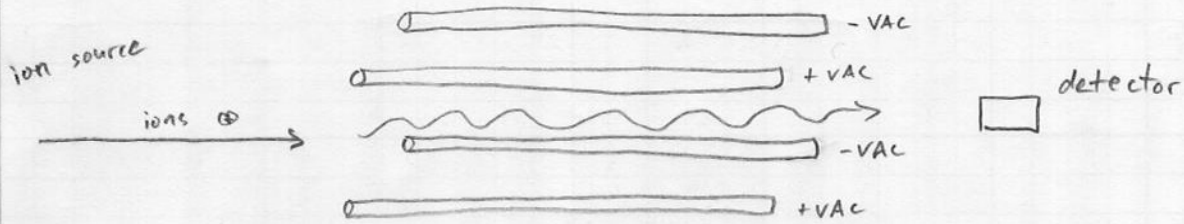
7-6-1-2 Ion Collector and Preamplifier - The deflection plates (i)/ion collector (k) assembly is flange-mounted (m) onto the mass spectrometer (see Figure 7-1A and 7-5). The positive helium ions striking the ion collector (k) cause it to have a positive charge. Negative electrons flowing toward this positive-charge collector (k) constitute a minute current. This current is proportional to the number of positive ions striking the collector (k). The current is amplified in the preamplifier and the signal appears on the meter as the leak rate (see Section 4-1-2).

There are two version of the preamplifier currently in use. One version has the changeover switch (see Figure 7-1A), and the other version does not have this switch (see Figure 8-4).



# Quad mini-lecture: Potentials + fields

Quadrupole mass spectrometer basic structure:



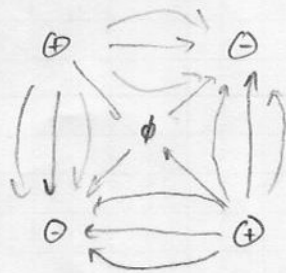
Four conducting poles, apply alternating voltage, only ions that have just the right  $q/m$  make it through for a certain AC frequency. The rest get thrown clear before reaching the detector. Either too heavy or too light is no good.

Let's think about fields and potentials involved.

Draw a dipole. If appropriate, invite someone to draw field lines, then equipotential lines.

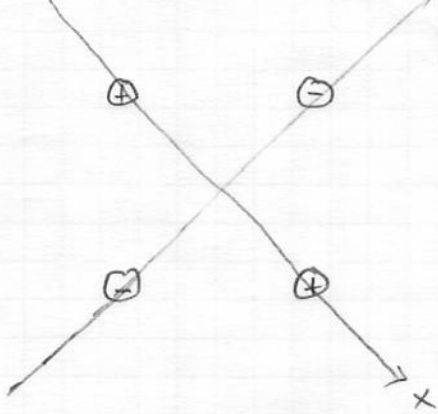


Quadrupole is two dipoles back to back

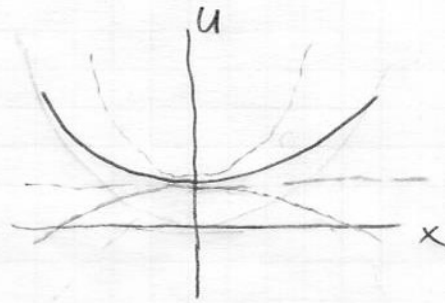


Saddle point at center

Add axes, consider energies on axes (for positive charge)

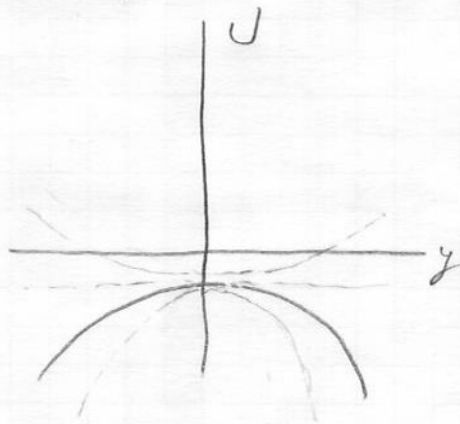


Get displaced along  $x$ :  
 Restoring force back.  
 Generally stable equilibrium



note asymmetry

Get displaced along  $y$ : Get attracted farther away  
 Unstable equilibrium

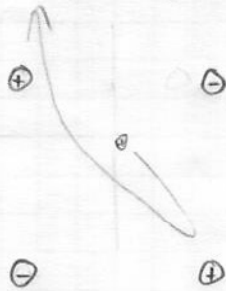


First step in constructing filter: Apply DC bias of this sort to the rods. By itself, insufficient. Particles all eventually crash into negative rods.

Add AC to all rods so that they flip back & forth.  
 Alternating stable/unstable equilibria along  $x, y$  axes.

Why this combo AC/DC? Treats heavy & light ions differently.  
 Heavy ions respond slowly, dominant effect is from DC, AC washes out. Get directed into overall-negative poles.

Light ions respond quickly, get flung out along  $x$  axis



Ions with just right  $g/m$  oscillate and never quite get thrown out in either direction.

Scan either frequency or (most likely) AC amplitude to count different species

## SimIon tour

Load quad sim.

Tour various views. Voltage settings.

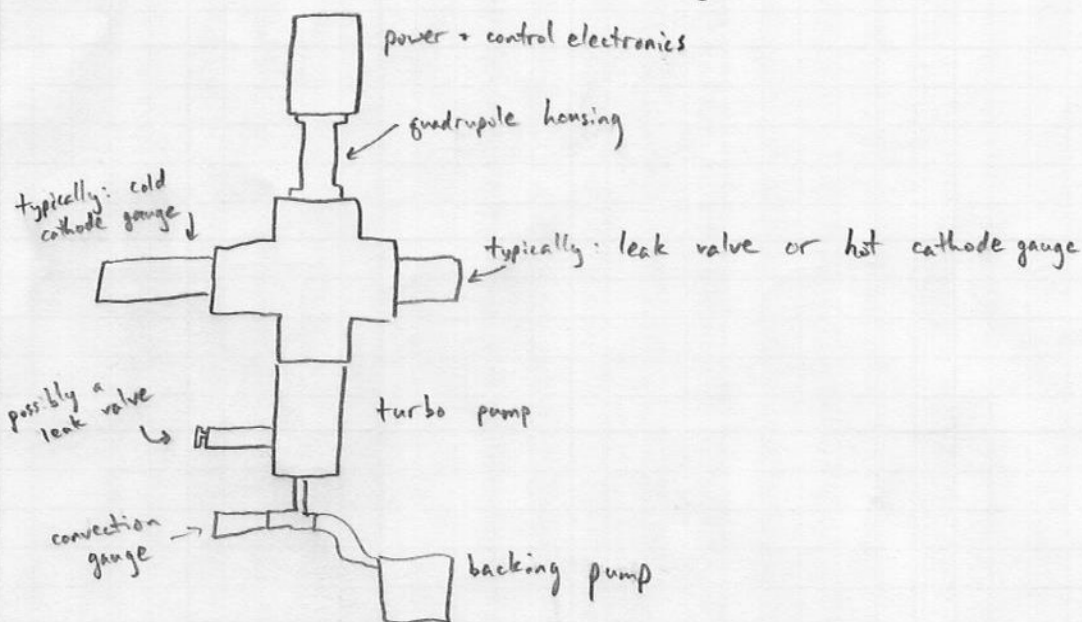
Start with 100 amu, various particles.

Swap to lower/higher mass particles. Check out departing trajectories.  
Hover over electrodes to check out high/low mass/voltage conjectures  
(do light ones go out + axis, heavy out - ?)

Settings to color code high/low masses.

Note two periods on motion of surviving species.

## Tour: Quadrupole system + scanning



## TalkStar software

Open

Right click Prisma 80 - connect

Right click - emission on

\* Don't turn on filament until under vacuum.

\* Manually turn off emission before venting \*

Ion source is hot cathode

Scan: Standard scan. Right click on screen to access various display options

Trend/Leak Test: Tracks set of user-definable species

If using sapphire leak valve: Turn slowly. Do not tighten past zero. Expect to unscrew 1.5<sup>+</sup> turns before seeing effects. Beyond that, go slow.



## Cracking patterns

Mass spectrometers function by sorting molecules according to  $m/z$ . Superficially, we have a problem: Many different species have same  $m/z$ .

Consider: methane,  $\text{CH}_4$ , 16 amu

Gaseous oxygen,  $\text{O}_2$ , 16 amu

These would be indistinguishable if nothing else were going on.

Fortunately, the ionization process tends to rip up the molecules involved (cracking)

$\text{O}_2$  becomes a mix of  $\text{O}_2$  (16) and  $\text{O}$  (8)

$\text{CH}_4$  becomes a mix of  $\text{CH}_4$  (16),  $\text{CH}_3$  (15),  $\text{CH}_2$ ,  $\text{CH}$ ,  $\text{H}$

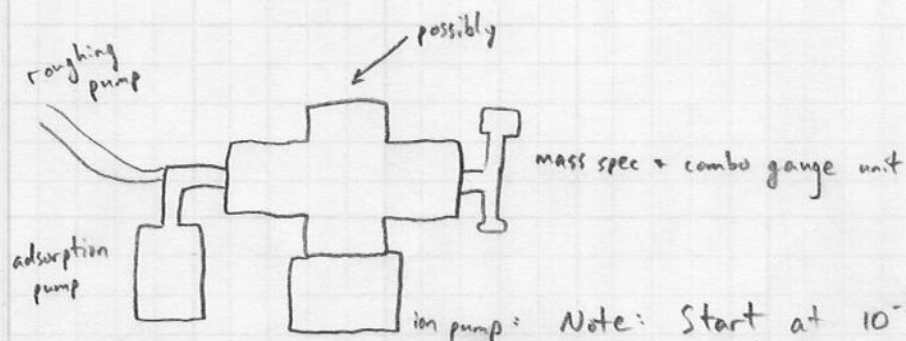
These characteristic distributions, or cracking patterns, give us vastly more information than we'd otherwise have

(browse charts, graphs)

Eventually note periodicities - hydrocarbons lead to patterns periodic in 12 (carbons) and 1 (hydrogens)

Tricky bit: Not everything is singly ionized  
14 can be a mix of singly ionized  $\text{N}$  and doubly ionized  $\text{N}_2$   
Possible to get a handle on expected proportions from ionization potentials (IPs)

# Ion trap apparatus + basic principle

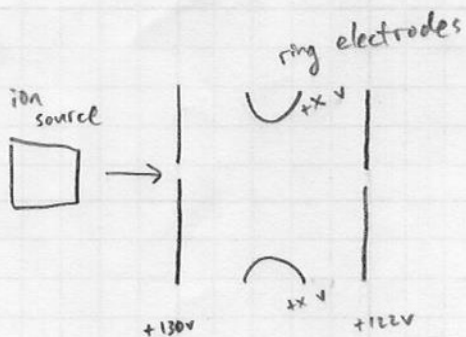


Note: Start at  $10^{-5}$  Torr, expect current overload warnings, ignore, should go away as pressure drops (maybe  $10^{-4}$ )

Roughing  $\rightarrow$  adsorption  $\rightarrow$  ion pump

Combo gauge can run starting from atmosphere

Mass spec interlock - won't turn on till combo gauge reads  $1 \times 10^{-5}$   
 Note odd discontinuity in pressure readings around  $10^{-6}$   
 (jumps to  $10^{-8}$ )



Ion trap mass spec video on youtube:  
 "trap mass spectrometer"

Collect ions, inject into chamber, trap in center region

Apply AC to left/right plates. Drive ions back + forth. For one m/z, get resonance. Ions ejected + counted fairly quickly.

## Ion trap mass spec scanning

Open VQM software.

Note blue box: Combined power supply, control electronics. Not very large. Lower voltage & frequency demands, compared to quad.

Note combo gauge: hot cathode, thermo, and piezo  
Has auto cutoff at high pressure

Connect device via menus.

Pull down menu → Standby. Filament ion source turns on, pressure goes up.

Scanning: Fast compared to quad. Multiple species tracked effectively simultaneously. Software can use cracking patterns to bundle peaks and automatically ID species.

Note that a particular mass peak may contribute to more than one species, and some may get categorized as 'unknown'.

Check out histogram mode.

Possible experiment: Neon injection, two stable isotopes.  
Downside: Ion pumps don't remove noble gases well.  
Atoms driven into trap walls, don't bond, pop back out  
Some neon still in system after years pass!

\* Don't change any voltages in the settings. But note voltage asymmetries.

Note sharpness of peaks.

Ion trap mass spec fast, good resolution compared to quad.  
Downside: smaller operating range.

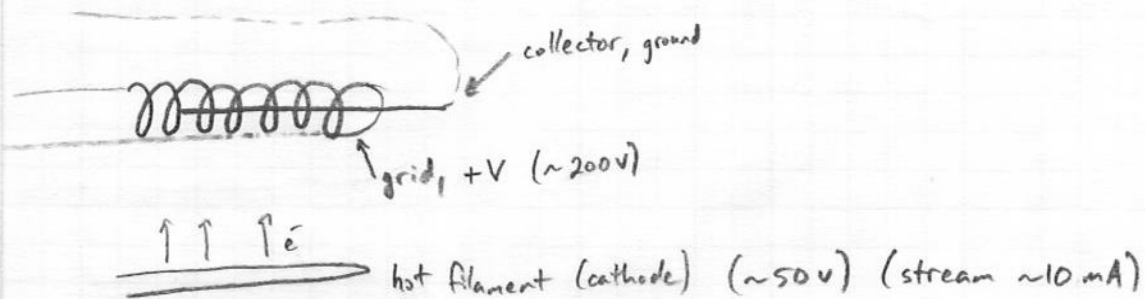
Resonant frequency proportional to  $\sqrt{m}$ .  $\sqrt{2}$  very different from  $\sqrt{4}$ , H very resolvable from He.  $\sqrt{156} \approx \sqrt{157}$ , resolution falls apart at high mass number.

Ion smaller (good), but can't be made better by being bigger, the way a quad can get better resolution by being longer.

Manual fig 7-9 and nearby for relevant schematics  
8-15 for cross-section



## Hot cathode ion gauge



Heat up filament. Electrons stream off (thermionic emission).

Grid accelerates electrons into cylindrical enclosed region. Electrons impact molecules + ionize them. Lower-voltage collection filament attracts ions, screens off electrons (counting both would yield zero)

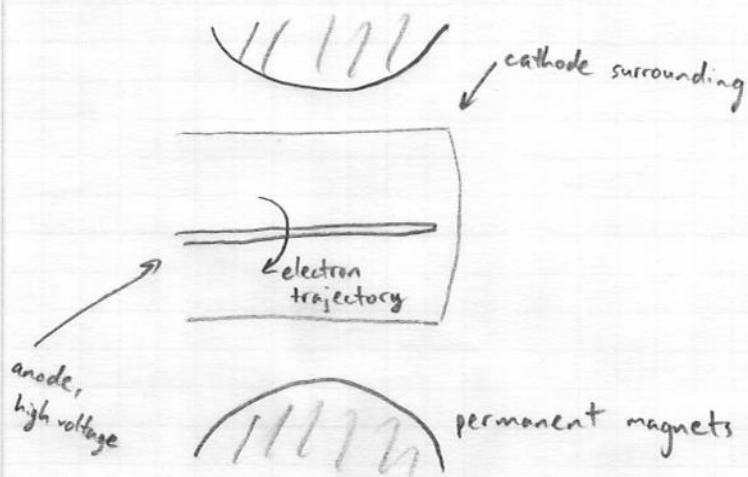
Collector current ends up being directly proportional to pressure over entire gauge range.

Don't turn on above  $\sim 10^{-3}$  Torr; filament can oxidize and burn out

Pros: Accurate, stable, reproducible  
Easy startup.

Cons: Filament burns out  
Gunk accumulates; regular degassing (cooking, basically) recommended

## Cold cathode ion gauge



Electron plasma trapped in interior by crossed electric + magnetic fields. Amplitudes + directions chosen to trap these light particles, substantially increasing likelihood of interaction with ambient molecules. These molecules ionize + are scooped up by cathode. Ionized electrons contribute to plasma.

Tricky part: Initializing plasma. Can wait until molecule gets close to pointy bit on cathode/anode + ionizes directly, at which time cascade begins. Recall pointy bit  $\Rightarrow$  strong local E-field. Startup time can be seconds at  $10^{-4}$ , minutes at  $10^{-5}$ , etc. Some gauges use radioactive sources to hasten ionization. Conversely, gauge floods at above  $10^{-3}$ . Suggest turning on at  $10^{-4}$ .

Pros: Lower currents, less power draw/heat  
No hot filament to burn out

Cons: Highly nonlinear pressure/current relationship.  
Accuracy + ease/robustness of calibration suffer  
Less user configurable (fairly fixed operating voltages/currents)