CHEM 212
Exam 4
Fall 2013

Name Key

1. What does ICP stand for?

2 Inductively coupled plasma

2. Diagram the ICP portion of the instrument and label the pieces

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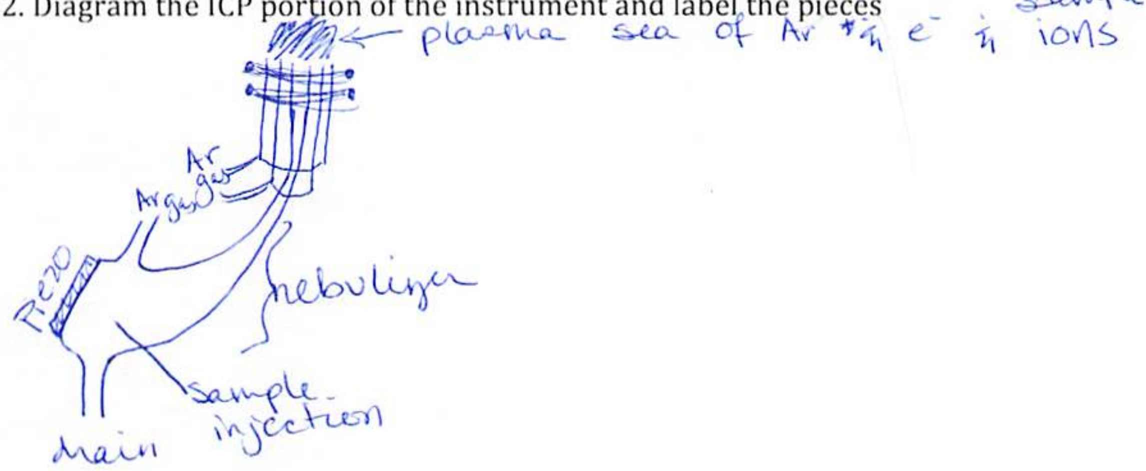

6 3. Describe the function of the pieces of the instrument listed below.

Piezo crystal
vibrates to assist sample aerosilization & delivery to carrier gas

Carrier gas
carries aerosolized sample to plasma

Radio Frequency coil
generates Ar plasma

nebulizer
aerosolizes sample

sample injection
aqueous sample injected ~~as aqueous~~ into nebulizer

plasma 10,000 K
excites ~~all~~ sample ions to excited electronic state
ionizes samples.

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5 4. Describe what happens to the sample from the point of injection until it is in the plasma

1. sample aerosolized by piezo crystal
2. sample carried by carrier gas to plasma
3. sample ~~el~~ ions electronically excited & ionized by plasma

2 5. What are the TWO types of detectors are generally used with an ICP source?

MS -

OES

MS ions swept into mass spec portion of the instrument. Then separated from photons through bending. Separated based on $\frac{m}{z}$ & using a quadropole & detected.

OES emission spectrum (~~generated~~ photons generated by atoms transitioning from excited to ground state) are measured after wavelength is selected using a monochromator.

7

3 6. Diagram and describe the function of each part of a GENERALIZED mass spectrometer.

① ion generation, ion acceleration → ② selection or separation of ions ($\frac{m}{z}$) → ③ ion detection

1. ions are generated from sample molecules & accelerated
2. ions are separated on the basis of $\frac{m}{z}$
3. ions are detected & electron multiplication is used to generate a detectable signal

7. What does a mass spectrometer measure?

2 $\frac{mass}{charge}$ of ions (or) $\frac{m}{z}$ (or) mass to charge ratio

8. What is the pressure inside a mass spectrometer (roughly)? Why is pressure controlled?

10^{-6} torr → 10^{-9} torr

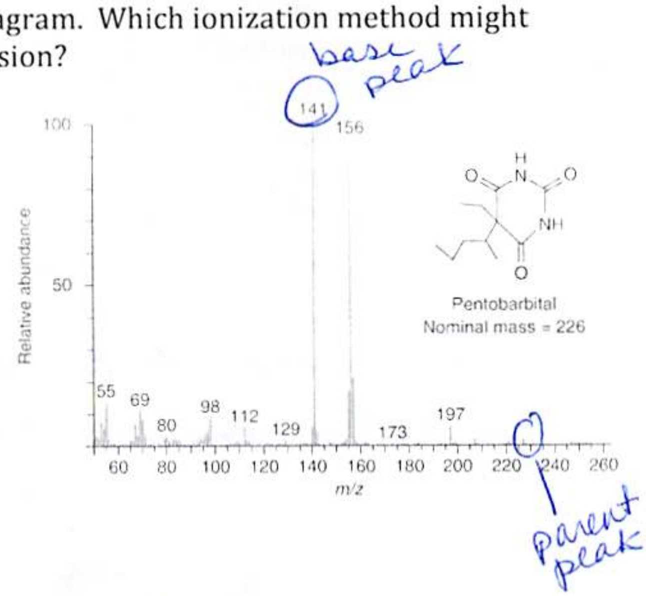
2 magnetic sector — TOF

to prevent air molecules colliding w/ sample molecules

9. Define and label the parent peak and base peak in the diagram. Which ionization method might
4 have been used and what evidence leads you to this conclusion?

Parent peak

$M^{+\cdot}$ • mass of molecule being measured



Base peak

• mass of largest peak in spectra

electron ionization b/c there is a very small parent peak

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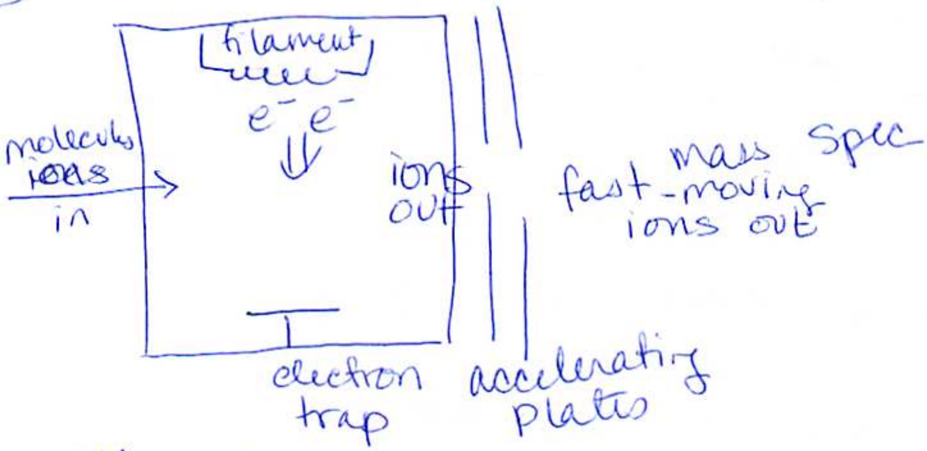
5 10. Draw an electron ionization method. And label the pieces.



all under vacuum

5 11. Describe how this piece of the instrument generates ions.

1. molecules are flowed into vacuum where they interact w/ electrons generated at the filament & accelerated to ion trap.
2. collisions b/w sample molecules & electrons generates enough energy to liberate a valence electron & sometimes fragment the molecule further
3. ions are then accelerated into the mass spec

3 12. Electron ionization is a relatively aggressive ionization technique. What does this mean in terms of the relative abundance of peaks.

low abundance of parent peak.

higher abundance of fragmentation peaks

3 13. What is the difference between electron ionization (EI) and chemical ionization (CI)?

CI has the same setup but has a reagent gas present at the pressure of ~1mbar to bear the brunt of interactions w/ e^-

3 14. What changes in the fragmentation pattern would you expect from a CI vs an EI spectrum for the same compound.

CI has a larger $MH^{\cdot +}$ peak

EI has little or no parent peak

EI has a higher abundance of fragmentation peaks

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15. Draw a magnetic sector mass spectrometer and describe how it works.

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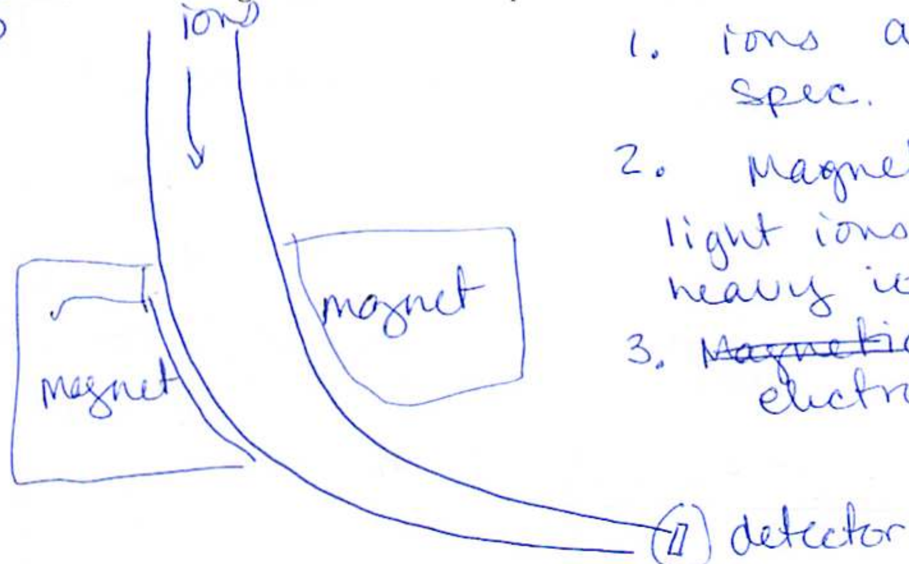


1. ions accelerated into Mass spec.
2. Magnetic field bends ions. light ions bent more than heavy ions
3. ~~Magnetic~~ ions detected w/ electron multiplication

4. magnetic field varied in time to detect different $\frac{m}{z}$ ratios.

16. How can a magnetic sector mass spectrometer be used to detect multiple ions.

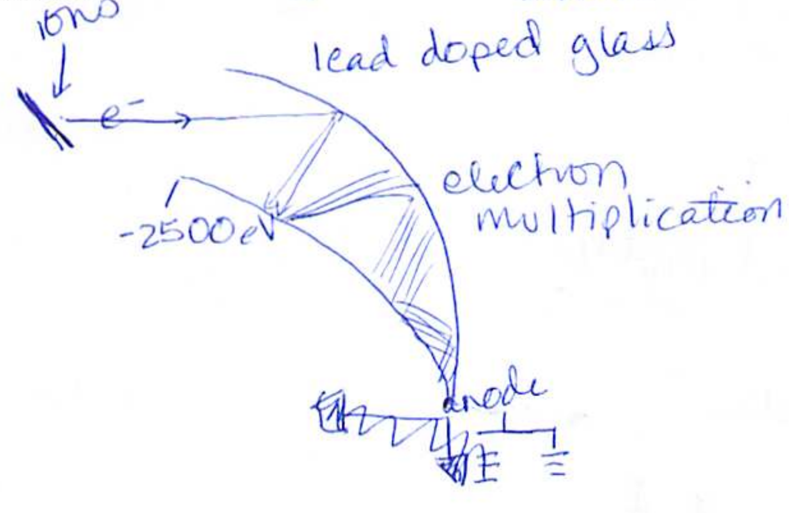
3

magnetic field varied in time to detect ions of different $\frac{m}{z}$ ratios.

17. Diagram an electron multiplier or channeltron. Describe how this piece of instrumentation works.

5

See pg 7 — shown here



1. ions ~~being~~ converted to electrons
2. electron impact lead doped glass & progressively multiplied in cornucopia
3. current at anode converted to a voltage & measured.

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4

18. Define what chromatography is used for.

chromatography is used to separate a mixture into its component species

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19. If solute A and B have retention times of 16.7 and 17.8 minutes, respectively and a $w_{1/2} = 0.3$ min for each peak. Calculate the resolution of these peaks. Is it adequate for a quantitative separation?

$$\text{Resolution} = \frac{0.589 \cdot (17.8 - 16.7)}{0.3} = 2.16$$

yes Resolution > 1.5 is considered quantitative

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20. Calculate the number of plates in this column relative to solute A.

$$N = \frac{5.55 \ (16.7)^2}{(0.3)^2} = \frac{1548}{0.09} = 17{,}200 \text{ plates}$$

9

State the mathematical relationships between each pair of variables shown below. Also indicate the direction of change to the second variable as the first is increased.

21. Column length and Resolution

3

column length $\propto$ resolution

↑ ↑

3

22. elution time and band width

elution time $\propto$ band width

↑ ↑

3

23. particle size and resolution

particle size $\propto \frac{1}{\text{resolution}}$

↑ ↓

29

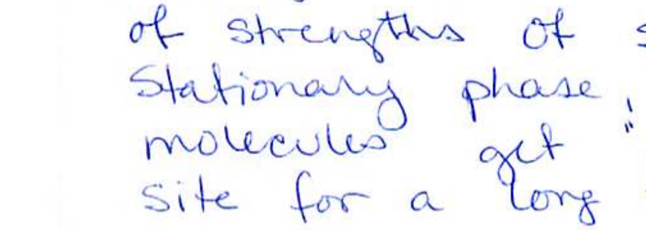
4 24. Describe the causes of "fronting"

Fronting results from overloading the column so some solute molecules dont get as much time in the stationary phase

4 25. Describe the causes of "tailing"

Tailing arises when there are a variety of strengths of sorption sites on the stationary phase, such that some solute molecules get "stuck" in a strong sorption site for a long time & are retained longer in the column.

17| Electron multiplier



1. ions impact the cathode & are converted to e⁻
2. electrons multiplied by dynodes held at progressively more positive potentials.
3. electrons captured as a current at the anode.
4. current converted to a voltage for measurement

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