**EXAM #1: Chemical Instrumentation 6614 Spring 2018 \_\_\_/100**

Your name:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ 1 pt

* 1. **The Solution is… (10 pts total) show work please !**

a) 1 ppm is defined as: (circle one answer) (1 pt)

 i) 1 g/1 L ii) 1 mg/100 mL iii) 1 mg/1 L iv) 1 μg/ 1 L

a) You have a 1.0 L solution of 246.93 ppm Ni2+ (w/v) How many grams ( to the nearest 0.001 g)

of NiCl2\*6H2O (MW=237.69 g/mol) were dissolved in the 1.0 L solution to reach this concentration ? The atomic mass of Ni =58.693 g/mol. ( 5 pts)

**246.93 ppm Ni2+ =0.24693 g Ni2+/L => 0.24693/58.693 mol Ni/L = mol NiCl2\*6H2O/L=0.004207 mol**

 **Mass NiCl2\*6H2O= 0.004207 mol\*237.69 g/mol=1.000 g**

grams of NiCl2\*6H2O =\_\_\_**1.000\_**

 b) A 5 mL volume HCl originally 2.0 **M** in concentration is diluted to 1 L with distilled water to make a stock HCl solution. A 2 mL volume of this prepared stock is then delivered to a 500 mL volumetric flask to make an intermediate stock. Finally, 2.5 mL of the intermediate stock are rediluted to a total of 25 mL in a 25 mL volumetric flask to make the final `standard’ HCl .

What is the concentration of the final standard HCl in μ**M**  (micro moles/L). ( 5 pts)

**Dilution factors: 2.0 M \*(5/1000) \* (2/500)\*(2.5/25)=4\*10-6 M =4 μM**

 Final standard HCl concentration = \_\_\_\_4\_\_\_\_\_\_μ**M**

\_\_\_/12 includes name

* 1. **Fitting Rewards (10 points)**

a) A calibration of Cu standard additions (**N**) vs absorbance **A** at 352 nm using the AAS yields the fit below. Given that the standard addition volume is 5 mL and that one ( (1) standard addition volume of the unknown Cu is in each of the 25 mL volumes containing the calibration standards, what is the concentration of the unknown Cu solution ? **Assume the reference concentration of the intermediated standard Cu is 20 ppm**

 **0=0.125Nu +0.0625=> 0.0625/0.125=Nu =0.5**

 **Nu\*20 ppm =Cu =20\*0.5=10**

**A = 0.125\*N + 0.0625**

 **Unknown Cu2+** concentration = \_\_\_\_\_\_\_10\_\_\_\_\_\_ppm (3 pts)

 c) The following fits are found for Cu and Ni standards using the Lambda 25 UV-VIS spectrophotometer.

*Copper coefficients* *Nickel coefficents*

(m=slope b=intercept)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Wavelength | mCu | bCu | mNi | bNi |
| λ1=400 nm | 0 | 0 | 5.00 | 0.0 |
| λ2=800 nm |  8.00 | 0.0 | 0.40 | 0.0 |

A mixture of Cu and Ni in an unknown solution produce the observed absorbances, A1 and A2 below: (where above coefficients represent fits of general form: A= m[Cu or Ni] + b)

The observed unknown A1 (at 400 nm) = 1.00

The observed unknown A2 (at 800 nm) = 0.88

Set up the two equation, two unknown system you would have to solve given that **x**= [Cu2+] M and **y** = [Ni2+] M using the coefficients above and the given unknown absorbances A1 and A2.

 **EQUATIONS HERE (3 pts)**

**A1 (400 nm) = 1.00 = 5y**

**A2(800 nm) = 0.88 = 8x +0.4y**

Use the above equations to solve for the concentrations of Cu2+ =x and Ni2+ = y (4 pts)

 **x=[Cu2+] = \_\_\_\_0.1\_\_\_\_\_\_M y=[Ni2+]=\_\_\_0.2\_\_\_\_\_\_\_ M**

(hint: One of the equations lets you solve directly for Ni2+. Find it first then substitute into the other equation to solve for Cu2+)

**1=5y=> 1/5=y=0.2**

 **0.88=8x+0.4\*0.2=> 0.8=8x=>x=0.1**

* 1. **What Every Atomic and UV-VIS Spectroscopy Geek should know…( 32 pts)**
1. Acronym for the source of an AAS \_\_\_\_HCL\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
2. New school transducer that converts light to current in Lambda 25 UV-VIS\_photodiodes\_\_
3. Acronym for older transducer that converts light to current in Lambda 4B UV-VIS \_PMT\_\_\_
4. UV source of a UV-VIS spectrophotometer\_\_deuterium lamp\_\_\_\_\_\_\_\_
5. What `gadget’ divides up the incoming source beam in a double beam UV-VIS into a reference and a source signal \_\_\_\_chopper\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ (hint: it looks like a propeller)

\_\_\_\_\_/15

* 1. **What Every Atomic and UV-VIS Spectroscopy Geek should know…(continued)**
1. ICP stands for… \_\_\_Inductively Coupled Plasma\_\_\_\_\_\_\_
2. UV-VIS cell material that provides the best (most expensive) window into the UV\_quartz\_\_\_\_
3. What is the `cell’ containing the sample analyzed in an AAS ? \_\_\_flame\_\_\_\_\_\_\_\_\_\_\_\_\_

9) Hyphenated name for UV-VIS beam path configuration: \_\_Czerny\_\_\_\_\_-\_\_\_Turner\_\_\_\_\_\_

10) Grating design in modern ICP \_\_\_echelle\_\_\_\_\_\_\_\_\_ (rhymes with gazelle)

11) Achilles heel of AAS\_\_\_\_slotted burner\_\_\_\_\_\_\_\_\_\_\_\_\_

 12) Main problem with using uv-vis for samples containing many elements: N2 problem\_\_\_\_\_\_\_

 13) Atomic spectroscopy method of choice for small sample sizes: \_\_HGA graphite furnace\_

 14) New-fangled grating used in Lambda 25 that simplifies optical path: \_\_holographic grating\_

 15) Name two advantages of ICP over AAS for element analysis:

1. \_\_\_\_\_\_\_\_simultaneous analysis of all elements\_\_\_\_\_\_\_\_\_\_\_\_\_\_
2. \_\_\_\_\_\_\_\_emission based (no need for sources)\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Also: more sensitive, no chemical problems

17) Which are the double beam instruments below ? (circle all that apply, (2 pts)

Lambda 4B UV-VIS Spec 20 PE 200 AAS ICP

18) Russian sounding graphite sample holder for HGA: \_\_\_Lvov\_\_\_\_\_\_\_\_\_ platform

19) Correct order for Electrothermal analysis:

* 1. flash- ash- dry b)dry-flash- ash **c)dry-ash-flash**  d)flash-dry-ash

20) For higher scan rate in a uv-vis:

a)increase slit width b) decrease slit width c) shorten wavelength range d) increase PMT gain

 21) Which technique is based on emission lines ? \_\_\_\_\_ICP\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

 22) Source and cell in an ICP:\_\_\_\_\_\_\_Plasma torch\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

 23) AAS and uv-vis both involve excitation of: (circle one)

a)vibrations b)rotations c)spin flips d)electrons

24) Name for device that selects wavelength\_\_\_\_\_\_monochromator\_\_\_\_\_\_\_\_\_\_\_\_

25) The AAS requires that the sample elements become \_\_\_\_\_atomized\_\_\_\_\_in the flame.

26)What is the gas excited in both ICP and HCL ? \_\_\_\_\_\_Ar (argon)\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

27) What part of the uv-vis instrument helps us reduce noise by phase locking to it? Chopper\_\_\_

28) Which is more sensitive: ICP or UV-VIS ? \_\_\_\_ICP\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

29) What is the source for the visible part of the light in a UV-VIS spectrophotometer?

 \_\_\_\_\_\_\_\_tungsten lamp\_\_\_\_\_\_\_\_\_\_\_

30) For a mixture of 6 unknowns how many calibration plots are necessary to solve for the

 unknown concentrations using a UV-VIS analysis ? \_\_\_\_\_\_36\_\_\_\_\_\_\_\_\_\_\_\_\_

31) For a mixture of 6 unknowns how many calibration plots are necessary to solve for the

 unknown concentrations using AAS analysis ? \_\_\_\_\_\_\_\_6\_\_\_\_\_\_\_

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* 1. **IR Basics (25 pts/1 pts each, except where noted)**

1.) The diagnostic region measures what basic vibrational motion ?\_\_\_stretches\_\_\_

2.)The IR effect requires either a(n) \_permanent dipole\_\_\_ or a(n) \_induced dipole\_\_\_\_

3.) The fingerprint region measures what basic vibrational motion ?\_bends\_\_\_\_\_\_\_\_

4.) If you have a 10 atom, non-linear molecule, how many vibrations are possible?

\_\_\_\_3\*10-6=24\_\_\_\_\_\_\_\_\_\_\_ (a number)

6.) What is the `heart’ of an FT IR ?

 \_\_\_\_\_\_\_\_\_\_\_\_Michelson-Morley interferometer\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

7.) Name the two fundamental (not technological) advantages of FTIR vs. dispersive IR

i) higher light thru put (Jacquinot advantage)\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ 1 pt

ii)\_\_\_can average over many (n) scans (S/N)~n1/2\_\_\_Fellgett’s advantage\_\_\_ 1 pt

8.)The basic IR effect involves \_\_vibrational\_\_\_\_\_\_\_\_\_ transition in the ground

 electronic state (1 pt each)

9.) The correct order of analysis typical for a dispersive IR spectrophotometer is:

a) source🡪 sample and reference🡪 slit🡪 chopper🡪 monochromator🡪 detector

b) source🡪 slit🡪 chopper🡪 sample or reference🡪 monochromator🡪 detector

c) chopper🡪 source🡪 sample or reference🡪 monochromator🡪 detector

d) monochromator🡪 source🡪 chopper🡪 sample or reference🡪 detector

10.) strong sharp doublets just below 3000 cm-1 is what molecular motion ?C-C-H stretch

11.) Key frequency indicative of a C=O (carbonyl) stretch: \_\_~1700\_\_\_\_\_\_\_\_\_\_\_\_\_ cm-1

12.) The IR below is best assigned to what molecule listed below the IR ? (circle choice)

1600

3000

1500



>2 overtones=> substituted aromatic ring

I am:

\_\_\_/12

**IR Basics (continued) 6/6**

13) what device in a dispersive IR is `moved’ to balance the reference and sample sides

 of the instrument ?

a) monochromator b) chopper c) beam splitter d) reference attenuator e)mirror

14.) What device in a dispersive IR is responsible for moving the device in question 15 ?

a) beam splitter` b) chopper c) attenuator d) synchronous motor e) tuning rectifier

15.) What sampling technique allows us to avoid use of KBr pellets for solid samples ? (can be an acronym) \_\_\_\_\_ATR head\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

16.) What physically scans in an FTIR?

a) beam splitter b) moving mirror c) monochromator d) laser interferogram

17.) A weak FTIR signal exhibits a signal to noise ratio, S/N of 2 after 50 scans. How many scans are necessary if you want to improve the S/N to 40? \_\_20,000\_\_\_\_\_\_\_\_\_\_\_\_\_scans

 (3 pts)

(x/50 )1/2 = 40/2=20

x/50 =202=400

x=50\*400=20,000

18) Acronym for the detector in ASC’s FTIR\_\_\_DTGS\_\_\_\_\_\_\_\_\_

19) The signal physically recorded by our FTIR is a measurement of the \_power\_\_\_

 spectrum which occurs in the \_\_time\_\_\_\_\_\_ domain.

20) FT in FTIR stands for \_\_Fourier Transform\_\_\_\_\_\_\_\_\_\_\_\_\_

21) Old school thingie you make to look at solid samples that involves a press and stainless

 steel die \_\_\_\_KBr disk \_\_\_\_\_\_\_\_\_\_\_\_

* 1. **Gas Attack: GC Trivial Pursuit (22 pts/1 pt each, except where noted)**

1) Chromatograms should exhibit SSS: = Sharp, \_\_**symmetric**\_\_\_\_\_, Separated

2) The mobile phase in GC (here in the US) is :\_\_\_\_\_\_**\_Helium (He)**\_\_\_\_\_\_\_

3) Name for plumbing `fitment’ used to directly connect plumbing to GC ports

 \_\_\_\_\_\_\_Swagelok\_\_\_\_\_\_\_\_\_\_\_\_\_\_

4) The two main kinds of column configurations common in GC are:

a)\_\_\_\_\_\_\_\_\_packed\_\_\_\_\_\_\_columns b) \_\_capillary (open)\_\_\_\_\_\_\_columns

5) Kind of detector we used in lab during the GC experiment (not the acronym-the full name:)

\_\_\_\_\_\_\_\_\_**\_\_flame ionization detector**\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

6) The one GC detector that can detect any gas is the\_\_\_\_thermocouple (TCD)\_\_detector.

7) The \_\_\_stationary\_\_\_\_\_\_\_\_\_\_\_phase in the GC column is responsible for creating separation.

8) The GC oven is used to produce a(n) \_\_\_thermal ramp\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

9) In order to reduce the sample size going we used a 20:1 \_\_\_\_split ratio\_\_\_\_\_\_\_

\_\_\_\_\_\_/20

**1.6. Gas Attack: GC Trivial Pursuit (cont.)**

10) The old school, pre-computer GC detector required balancing a\_Wheatstone\_

 Bridge.

11) GC detector requiring an Atomic Energy Commission license to own:

 \_\_ECD (electron capture detector)\_\_\_\_ (acronym or name)

12) Controls flow and routes gas to the GC injector port \_\_\_\_mass\_\_\_\_ \_\_\_flow\_\_\_

 \_controller\_\_\_\_\_\_ (3 words)

13) A PID creates ions out of samples using \_\_\_\_\_uv light\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

14) Diatomaceous earth is also referred to as the \_\_\_packing\_\_\_\_\_\_\_\_\_\_\_

15) One advantage of the PID is that it is essentially blind to :\_\_aliphatic and alcohol\_\_\_(non-aromatic)\_\_\_\_\_\_\_\_\_\_\_compounds.

16) What would be the most likely order of appearance for the chemicals below:

 --🡪 increasing tr

a) 1- butanol, ethanol, hexanol

b) ethanol, hexanol, butanol

c) hexanol, butanol, ethanol

d) none of the above

17) The ASC detectors burn what dangerously flammable fuel ? \_\_\_H2 (Hydrogen)\_\_\_\_

18) The typical inlet temperature of a GC is normally set around: \_\_200-230\_\_\_\_\_\_oC

19) What marks a tank nut as being left hand threaded ? \_\_\_\_notches \_\_\_\_\_

20) The GC detector is located:

a) next to the inlet port and before the column

b) just after the secondary tank valve

c) after the capillary column and inside the oven

d) after the capillary column and above the oven

21) Any day doing chemical instrumentation is a \_\_\_\_great\_\_\_\_\_\_\_\_\_\_\_\_\_\_day.

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\_\_\_\_\_/12