**EXAM #1: Chemical Instrumentation 6614 Spring 2017 \_\_\_/106**

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

* 1. **The Solution is… (16 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

1. You must prepare a 5.0 L aqueous solution of 0.002347 M Cu2+  from CuCl2\*2H2O (MW=170.44 g/mol) How many grams (to the nearest 0.001 g) of CuCl2\*2H2O must be weighed out to create this solution ? (5 pts)

CV=mol Cu2+=mol CuCl2\*2H2O = 0.002347\*5=0.011735 mol CuCl2\*2H2O

0.011735 mol CuCl2\*2H2O\*170.44g/mol =2.000

grams CuCl2\*2H2O=\_\_2.000\_\_\_\_

1. 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\_

d) 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**

\_\_\_/17 includes name

* 1. **Fitting Rewards (17 points) 2/6**

a) The following absorbances (A) are recorded for known concentrations of Cu2+ at 800 nm with a UV-VIS spectrophotometer. An unknown sample of Cu2+ yields an absorbance at 800 nm of 0.138.

a) What is the best fit (linear regression) line of Afit vs. [Cu2+] at 800 nm ? (4 pts)

**Afit = 8.8886[Cu2+] + 0.0019**

b) To the nearest 0.001 M, what is the unknown Cu2+ concentration ? (2 pts) 0.138=9.3171x-0.001428

(0.138+0.001428)/9.3171=0.0150

**Unknown Cu2+ concentration = \_\_\_0.015(3)\_\_\_\_\_\_\_\_M**

|  |  |
| --- | --- |
| [Cu2+], M | A(at 800 nm) |
| 0.000 | 0.000 |
| 0.020 | 0.180 |
| 0.040 | 0.368 |
| 0.060 | 0.560 |
| 0.080 | 0.640 |
| 0.100 | 0.930 |

b) 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)

0.88=8x+0.4\*0.2=> 0.8=8x=>x=0.1 1=5y=> 1/5=y=0.2

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

**\_\_\_/17**

* 1. **What Every Atomic and UV-VIS Spectroscopy Geek should know…( 39 pts) 3/6**

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)
6. ICP stands for… \_\_\_Inductively Coupled Plasma\_\_\_\_\_\_\_
7. UV-VIS cell material that provides the best (most expensive) window into the UV\_quartz\_\_\_\_
8. What is the `cell’ containing the sample analyzed in an AAS ? \_\_\_flame\_\_\_\_\_\_\_\_\_\_\_\_\_
9. Name for the traditional `circular’configuration of an ICP ? \_\_Rowland circle\_\_\_\_\_\_\_\_\_

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

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

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

13) Name two advantages an AAS has over a UV-VIS for element analysis:

1. \_\_\_\_\_\_no N2 problem\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
2. \_\_\_\_\_\_\_higher sensitivity\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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

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

16) 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) Item in an AAS that atomizes the sample so it can be burned: \_\_nebulizer\_\_\_\_\_\_\_\_\_

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

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

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

20) Correct order for Electrothermal analysis:

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

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

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

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

23) Source and cell in an ICP:\_\_\_\_\_\_plasma torch\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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

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

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**1.3. What Every Atomic and UV-VIS Spectroscopy Geek should know…(continued) 4/6**

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

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

27) What is the ~ temperature in an AAS flame ? 200 C 1200 C 2200 C 10,000 C

28) What is the ~ temperature in an ICP flame 200 C 1200 C 2200 C 10,000 C

29) What is the gas excited in both ICP and HCL ? \_argon\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

30) What part of the uv-vis instrument helps us reduce noise by phase locking to it?chopper

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

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

\_\_\_\_\_tungsten-halogen lamp\_\_\_\_\_\_\_\_\_\_\_\_\_\_

33) The following plot is obtained for a calibration of an AAS standard addition of Ni with an

unknown. What’s wrong and what error is likely to have occurred during the solution

preparations ?

What’s wrong here ?

No unknown standard add ! A=0 at N=0 (solution prep error)

A

A= 0

0 1 2 3 4 =N N= # std additions of 10 ppm Ni

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

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

35) For a mixture of 5 unknowns how many calibration plots are necessary to solve for the

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

* 1. **IR Basics (31 pts/1 pts each, except where noted)**

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

2.)The IR effect requires either a \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ or a \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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)

5) Does CO2 have an active IR band ? Yes No 1

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**IR Basics (continued) 5/6**

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 abbreviation vs br stands for: \_\_very strong, broad\_\_\_\_\_\_\_\_\_\_\_\_\_

10.) 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

11.) A strong, broad band near 3450 cm-1 is what molecular motion ? \_\_O-H stretch\_

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

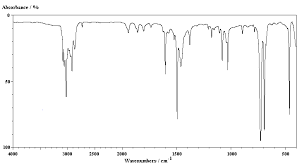
13.) Key frequency indicative of a C=O (carbonyl) stretch: \_\_~1700\_\_\_\_\_\_\_\_\_\_\_\_\_ cm-1

14.) 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:

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

15) 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

16.) 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

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

18.) What physically scans in an FTIR?

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

19.) 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

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

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

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

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

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

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

24) What is being protected by putting a dessicant inside the housing of our FTIR ?

\_\_\_\_KBr Beam splitter\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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

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