Exam 1 Name:_____

Be sure to show your work, write CLEARLY, and put your answers in the boxes on each problem.

1. Ascorbic acid ($C_6H_8O_6$; FM= 176.1238 g mol^{-1}) reacts with I_3^- to form dehydroascorbic acid ($C_6H_8O_7$) according to the following equation. Starch is used as an indicator of the reaction. The end point is marked by the appearance of a deep blue starch-iodine complex when the first fraction of a drop of unreacted I_3^- remains in solution.

$$C_6H_8O_6 + I_3^- + H_2O \rightarrow C_6H_8O_7 + 3I^- + 2H^+$$

An I_3^- solution was standardized against 0.1970 $\pm$ 0.0003 g of pure ascorbic acid. If 29.41 $\pm$ 0.03 ml of I_3^- solution was required to react with the ascorbic acid, what is the molarity of the I_3^-solution? Be sure to report the error associated with the molarity. (10 pts)

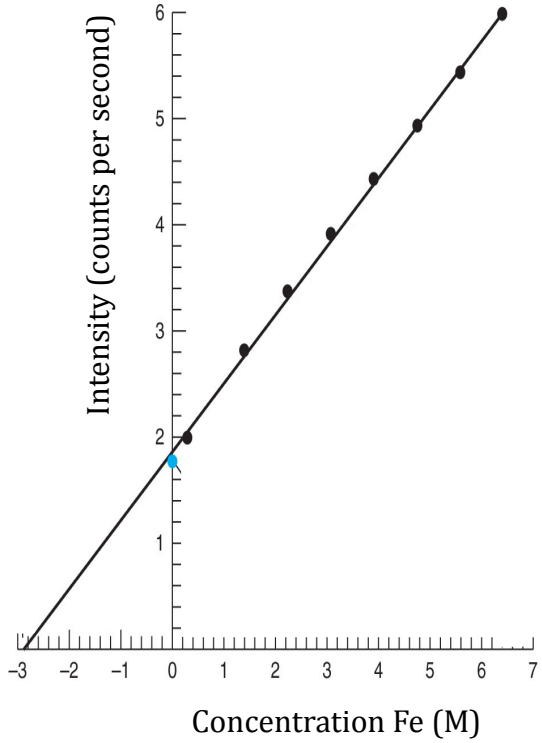
$[I_3^-]$ (M)	

2. Calculate the concentration of Fe from the graph if the line of best fit can be described with the following equation:

$y = 0.57 x + 1.8$

2a. Indicate on the graph where the concentration of Fe in the original sample can be read. (2 pts)

2b. Solve for the concentration of Fe in solution using the equation of the line of best fit. Be sure to show your work. (8 pts)



Fe (M)	

3. You measure 5 standards in order to generate a calibration curve (data shown below). You calculate the line of best-fit y = 1.53x + 0.003 with an R^2 = 0.99579. You perform triplicate measurements of an unknown with an average absorbance reading of 0.678. Calculate the concentration of Ni (M) and the error associated with the measurement (s_x). (10 pts)

x_i	y_i	x_iy_i	x_i^2	d_i	d_i^2
0.5	0.78	0.39	0.25	0.012	0.000144
0.4	0.59	0.236	0.16	-0.025	0.000625
0.3	0.47	0.141	0.09	0.008	6.4E-05
0.2	0.32	0.064	0.04	0.011	0.000121
0.1	0.15	0.015	0.01	-0.006	3.6E-05
Σx_i=1.5	Σy_i=2.31	Σx_iy_i= 0.846	Σx_i^2= 0.55		Σd_i^2= 0.00099
x= 0.3	y= 0.462		$\Sigma(x_i-x)^2$= 0.09		

Ni (M)	

4. Quantify and describe the random and systematic sources or error in the procedures described below. (2 pts each)
Define Random error

Define Systematic error

a. A 10 ml pipet consistently delivers 10.05± 0.08 mL

Error type	Magnitude	Explanation
Random		
Systematic		

b. Four consecutive 20 μl injections of a standard into a Capillary Electrophoresis yielded peak values of 50.4, 52.7, 49.8, 52.3.

c. Calibration checks of your 750 μS cm^{-1} standard for a field electrical conductivity meter yields consecutive readings, taken at 2 hr intervals during the day, of 753, 749, 735, 727, 711 μS cm^{-1}.

5. Match the following terms with the best statement. Note: there are more statements than terms. **Select the single best statement for each term.** (1 pt ea)

Terms

____Accuracy

____Random error

____Spike recovery

____Systematic error

____Gaussian distribution

____Confidence Interval

____Grubbs test

____Internal Standard

____Reagent Blank

____Method Blank

Statements

1. A method of comparing sample set means
2. Measuring the concentration of analyte in the acid used to dilute your sample
3. A blank exposed to the field site.
4. Errors with the same magnitude on all measurements
5. Unavoidable in all measurements and measured by the standard deviation of replicate measurements
6. A test used to compare standard deviations
7. A way to extrapolate from a small sample set to a population
8. A range about the measured mean in which the true mean is likely to lie
9. A blank that has undergone the same preparation as the samples.
10. Used in chromatography to account for small fluctuation in flow through the instrument
11. A QA measure where a matrix-matched sample with analyte added is measured as an unknown
12. A test used to identify outliers
13. Can be assessed using certified reference material

6. The procedure outlined below is real data quantifying metal impurities from an industrial process. Define each of the following QA/QC terms and identify the location(s) in the procedure where each terms is evident. (3 pts each)

_____Spike recovery

_____Analysis of reference materials

_____Quality control samples

Impurities 2010 Form

	No.	Sample	ppm Te
1	7	Western Utah Copper	20
	8	Sierrita	49
	9	Mission North	56
2	10	CCu-1b	117
		CCu-1b Known	70
		CCu-1b Recovery	167
	11	Bed Rock	49
	12	ACR Anode Casting Fines	77
	13	ACR Foul Cathode (no metallics)	186
	14	ACR Refined Casting Fines	21
	15	ACR Sharp Slag	3,992
	16	ACR PM Brick	5,879
	17	ACR Residue	119
3	18	Lab QC	237
		Lab QC Known	180
		Lab QC Recovery	132
	25	Ray Tails	12
	26	Hayden Tails	14
	27	Ray Feed	14
	28	Hayden Feed	14
	29	Slag	74
4	30	Slag Duplicate Spike	66
	31	Slag Duplicate Spike	65
		Slag Duplicate Spike Recovery	-9.15
		Slag Duplicate Spike Precision	102
	32	Trench Slag Clean	67
	33	Trench Slag Medium	66
	34	SCO Furnace Flux	<10
	35	SCO Converter Flux	<10
	36	Filter Rock Bedding Rock	47
5	37	CCu-1c	73
		CCu-1c Known	23
		CCu-1c Recovery	317

7. Determine if the two sets of replicate analyses of a certified reference material shown in the table below are statistically different from the certified value of this standard (48 ppb) at the 95% CI.

Be sure to show your work!
Draw a figure indicating the relative sizes of a 50% CI and a 95%CI. (2 pts)

First Run Fire Assay-AA 30 g Aliquot		Second Run Fire Assay-AA 30 g Aliquot	
14	9	38	40
11	13	26	39
4	10	37	42
10	11	39	34
16	6	40	42
21	11	45	40
13	25	30	41
11	19	23	43
12	21	35	36
8	22	41	37
12	15		43
16	25		
12	18		
11	14		
15	12		
14	24		
Average = 14 +/– 5		Average = 38 +/– 6	

Average fire assay 14 ppb (32 aliquots) range: 4 to 25
Average fire assay 38 ppb (21 aliquots) range: 26 to 45

Calculate the 95% Confidence intervals for both data sets shown in the table. (8 pts)

	t_{tab}	CI	Range	Statistical difference?
Run 1				
Run 2				