Exp. No. 7	Experiment/Subject CHEM438 (XRF)	Date 10/25/2024	
Name Abdul Fayeed	Lab Partner Justin M	Locker/ Desk No.	Course & Section No. 021L

PRE LAB Questions.

1. Electrons from the atoms get ionized from the absorption of X-rays of sufficient energy. Removing the e^- from inner shell of atom will cause the e^- from the outer shell to fall and fill the vacancy. e^- excess energy emission is the X-ray. Every element has different electron energy levels, so the signature is different for each element.

2. $6.4\ \text{keV}\left(\frac{1000\ \text{eV}}{1\ \text{keV}}\right)\left(\frac{1.602\times10^{-19}\ \text{J}}{1\ \text{eV}}\right)\left(\frac{1}{6.63\times10^{-34}\ \text{J·s}}\right) = 1.546\times10^{18}\ \text{s}^{-1}$

$$\lambda = \frac{c}{\nu} = \frac{2.998\times10^{8}\ \text{m/s}}{1.546\times10^{18}\ \text{/s}} = 1.937\times10^{-10}\ \text{m}\left(\frac{1\text{nm}}{10^{-9}\text{m}}\right) = 0.194\ \text{nm}$$

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Objective

The purpose of this lab is to determine the trace of Zn contamination on lab benches via dispersive x-ray fluorescence measurement.

Introduction

A wipe test is performed to collect the sample of zinc on the lab benches. As opposed to the acid digestion method, x-ray fluorescence will be done instead to identify the contaminants and quantitate the zinc.

Procedure

1. Get trained on the use of x-ray spectrometer.
2. Assemble the XRF cells. (Be careful not to overstretch and tear the mylar film)
3. Make 6 sample cells. Don't contaminate the film.
4. Set up the experiment in the software.
5. Analyze the wipe sample through qualitative and quantitative measurement.

Qualitative

1. Use high tube voltage and do survey scan
2. Identify and label the peaks.
3. Adjust voltage, filters, and current to produce a clear spectrum. Label appropriately.

Calibration micropipette.

Number	mass (g)
0	empty. (of the apt bottle)
1	↓ add 100µL consecutively
2	
3	
4	
5	
6	
7	
8	
9	
10	

Zn Kα line : 8.639 keV

-2x : -17.278 keV

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Quantitative

1. Make standard solution of Zn from 1000 ppm stock (~100mL of Zn stock adds an appropriate mass of Zn to towel).
2. Make 6 towel rounds. Cut out circles of lab w/ scissors.
3. Use one of the 3-ply rounds, lightly wet w/ 100 µL distilled water, and wipe the entire surface of lab bench. Expose the surface evenly.
4. Dry the towel in oven for 5 minutes. Cool down then measure the spectrum.
5. Repeat the wipe sample by using a second 3-ply towel round.
6. ~~The other~~ → Use the other 4 3-ply rounds to make the calibration curve by adding aliquots of Zn with the micropipette.
7. Aim to ~~be~~ span the range 100mg above and below the estimated wipe value.
8. Dry the circles in oven in labeled beaker for 5 minutes. Cool down then measure the spectrum.
9. Record the cps (counts/~~min~~ sec)
10. Measure the blanks: unexposed towel round and mylar films.
11. Calibrate micropipette by weighing empty bottle and cap (±0.1mg) and add 100µL of water, then ~~weigh and re~~ recap and reweigh. Repeat 10 times.

Sample	Conc. Zn (ppm)	$K\alpha_1$ (ev)	$K\alpha_1$ (ev)	$K\alpha_1$ (e
Blank				
Unk. 1				
Unk. 2				
Stand 1				
Stand. 2				
Stand. 3				
Stand. 4				

Conclusions:

X-ray fluorescence can be used to detect the trace of Zn and both quantitate and qualitatively measure the trace.

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