Lab #8 – Assay and Analysis

Name: _____ Grade: _____ Feedback:

Group Name: _____ Day:

Pledge: "On my honor as a Virginia Tech student, I have neither given nor received unauthorized assistance on this assignment." Initial_____

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If you answer yes to any questions in the Hokie Health survey (questions can be posted in the syllabus), you must not attend class in person. Notify me by email and contact Schiffert Health Center for testing and quarantine protocol.

Introduction

In mineral processing operations, metal assay values are crucial for determine feed grades, product quality, and even solid mass flows around plant unit operations (via the two-product formula). Depending on the product type (base metals vs. industrial minerals vs. construction materials) several different measures of quality may be significant, and as such, numerous methods of characterization may be applicable. For metallic ores, including base sulfides, precious metals, and some industrial minerals, the percent metal is the most important assay, which must be closely monitored during production.

For high-precision metal assays, samples are often fully-digested by using a prescribed mixture of strong acids at aggressive temperature and pressure conditions. Once digested, the elemental content of the of the liquid aliquot can be determined by several methods including: atomic absorption spectroscopy (AAS), inductively coupled plasma optical emission spectrometry (ICP-OES), or inductively coupled plasma mass spectrometry (ICP-MS). Each technique is characterized by different elemental ranges, different detection limits, and subsequently, different costs.

Figure 1. Hand-Held XRF.

While these digestion-based methods are ultimately needed for final analysis and reporting, one alternative method engineers and metallurgists can use to get quick solutions is via portable X-ray florescence (XRF) analyzers. This technique provides elemental assays for dry, particulate samples, and thus does not require sample digestion prior to analysis. As shown in Figure 1, these instruments are often designed for rugged, field environments and provide rapid results often less than 60 seconds. This rapid analysis is crucial for both exploration and plant personnel who need to make quick decisions based on production data.

Since XRF is a dry analytical method, sample homogeneity is critical for reproducible analysis. For particle samples, such as those found in the processing plants, this homogeneity must be achieved by careful sample preparation. If high-precision is required, the samples are often ground extremely fine by hand and then pressed into a pellet prior to analysis. If even higher precision is required, samples may be melted and fused into a glass using a special fluxing instrument.

In this laboratory exercise, you will use a hand-held XRF unit to analyze an unknown sample and determine which valuable elements may be present in the feed. You will then determine which elements were separated by analyzing feed, concentrate, and tailings streams that have been produced by an unknown separation device. You then use data from repeat analyses to determine the statistical confidence intervals as well as possible deviation from independent assays. Lastly, you will investigate the precision the instrument by a using several methods of sample preparation.

Helpful Equations

Two Product Formula	$Y = 100\,\frac{(f-t)}{(c-t)}$
Recovery	$R = 100\,\frac{Cc}{Ff} = (100\,Y)\,\frac{c}{f}$
Rejection	$J = 100\,\frac{Tt}{Ff} = (100 - Y)\,\frac{t}{f}$
Nomenclature:	C, T, and F: Mass or Mass Flow Rates of Concentration, Tailings, and Feed. c, t, and f: assays of concentrate, tailings and feed

Confidence Interval	$CI = \bar{x} \pm t\,\frac{s}{\sqrt{n}}$
Nomenclature:	$\bar{x}$: sample mean s: sample standard deviation n: sample number t: t-statistic determined from a tabular listing of the Student's t distribution Recall: degrees of freedom (df) = n – 1; and a two-sided distribution is needed for the CI calculation. If only a single-sided distribution is available, lookup α/1 α = 100 – P; i.e. if a 95% confidence interval is required, α = 0.05 **Alternatively, the confidence interval ($t\,s/\sqrt{n}$ can be can be calculated directly using Excel function =*confidence.t(alpha, standard deviation, size)*.**
Hypothesis Testing (two samples, unpaired)	Null Hypothesis: $H_0 : \mu_1 = \mu_2$ (i.e. the mean values are equal) Alternative Hypothesis: $H_a : \mu_2 \neq \mu_1$ (i.e. there is a difference between the means) To test the validity of the null hypothesis, three steps are usually required: 1. Calculate the mean and sample standard deviation of both sample sets. 2. Calculate s and t-statistics using the equations below (only valid for two-sample, unpaired tests with the hypotheses given above). 3. Look up the observed t-stat in a tabular listing of the Student's t-distribution to determine the significance level, p (or use *t.dist.rt* or *t.dist.2t* in excel) 4. Reject the null hypothesis (i.e. conclude that the samples are different) if and only if p is less than a hurdle rate, often α = 0.05. t-statistic: $t = \frac{\overline{x_1} - \overline{x_2}}{s_p\sqrt{1/N_1 + 1/N_2}}$ Pooled St. Dev: $s_p = \sqrt{\frac{(N_1 - 1)s_1^2 + (N_2 - 1)s_2^2}{N_1 + N_2 - 2}}$ **Alternatively, the p value can be can be calculated directly using Excel function =*t.test(array1, array2, tails, type), where array corresponds to the sample data, tails indicates the 1 or 2-tailed, and type confirms whether the test is paired or unpaired.***

Procedure

Unknown Sample Analysis

1. Do all appropriate safety and personal protective equipment.

2. The instructor will provide you three sample cups that have been prepared for XRF analysis.

3. Use the XRF unit to analyze these three samples. The XRF procedures are given in an Appendix at the end of this document.

4. Determine which major elements have been concentrated and rejected in the various process streams. You may need to browse the sample history on the on-board memory. You should not need to re-analyze the sample.

5. Use the two-product formula and other process equations to determine the yield, recovery, and rejection of each element.

Copper Sample Analysis

1. The instructor will provide you a sample of copper feed that has been screened at 30 mesh as well as 5 clean sample cups.

2. Fill the 5 sample scups with powdered sample. First, prepare the samples by grinding small batches by hand using a mortar and pestle. Fill the cups after grinding. Label this Sample Set A. Analyze all cups using the procedure given in the Appendix.

3. Use a laboratory press to generate 5 small pellets. Label this Sample Set B. Analyze each pellet using the procedure given in the Appendix.

4. Perform the calculations and statistical analysis described below.

Data Records & Calculations

Unknown Sample Analysis

Element Name	Feed Assay (%)	Concentrate Assay (%)	Tailings Assay (%)	Yield (%)	Recovery (%)	Rejection (%)
Ti						
Zr						
Si						
Fe						

Copper Feed Sample Analysis

	Sample Set A	Sample Set B
Sample Preparation Method		
Sample 1 Cu Content (%)		
Sample 2 Cu Content (%)		
Sample 3 Cu Content (%)		
Sample 4 Cu Content (%)		
Sample 5 Cu Content (%)		
Mean Content (%)		
Standard Deviation		

Discussion Questions

Unknown Sample Analysis

1. In the first exercise, which elements were concentrated and which were rejected? Based on your recovery and rejection values, is this process efficient? Explain.

2. Are your yield values in agreement? Why or why not? What steps do you recommend to resolve any deviations?

Copper Sample Analysis

1. Determine the 95% confidence intervals for the two sample means. List those values (mean +/- CI) below. Use t = 2.571.

2. Use a two-tailed, unpaired, equal variance t-test to determine if the two sample preparation method are producing different results. Assume a hurdle rate $\alpha/2 < 0.025$ is needed to reject the null hypothesis and conclude that the means are different, and therefore rejection requires a t-statistic greater than 2.306. Show your work and report the t-statistic below.

3. The mining group of your company has conducted an independent set of assays, which has produced the following results: 0.58%, 0.67%, 0.51%, 0.59%, and 0.62%. Use a two-tailed, unpaired equal variance t-test to determine if their assays are in agreement with yours (use your most consistent data set from the two sample preparation methods). Assume a hurdle rate $\alpha/2 < 0.025$ is needed to reject the null hypothesis and conclude that the means are different. Report the t statistic. What does this result indicate, and what is the implication for your processing plant?

Conclusions

1. What was the objective of this laboratory exercise?

2. What were your major findings?

3. What important fundamental concepts did you learn from the exercise?

Appendix: XRF Operational Procedure

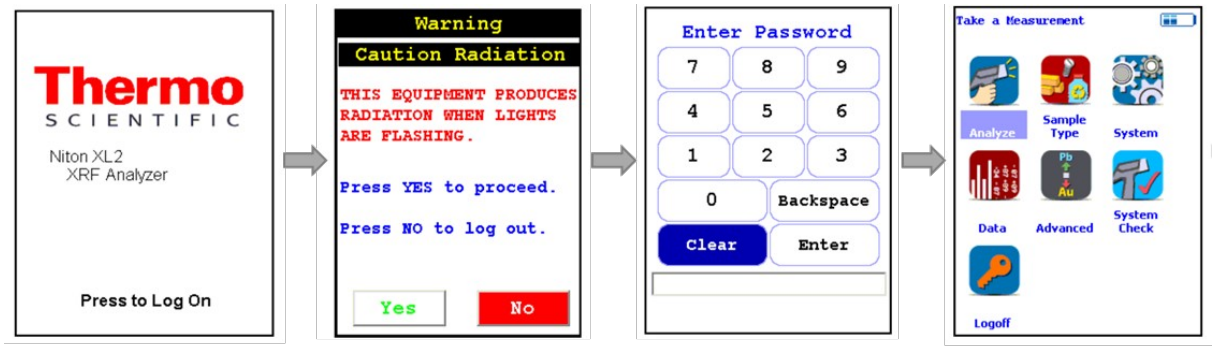
1. Startup Procedure

Turn on the analyzer by pressing the on/off button on the control panel until the *Touch Screen* comes on. When the startup is complete, the screen will be replaced by the *Logon Screen*. Tap anywhere on this screen to continue.

The *Logon Screen* will be replaced by a *Warning Screen*, advising you that this analyzer produces radiation when the lights are flashing. You must acknowledge this warning by selecting the *Yes button* before logging on. Selecting the *No button* will return you to the *Logon Screen*.

After selecting the *Yes button*, the *Virtual Numeric Keypad* becomes available for you to log onto the analyzer.

Select the 4-digit security code (1-2-3-4), followed by the *Enter button*. After you have completed the log on procedure, the word "USER" will appear on the bottom of the screen, then the *Main Menu* will appear. Wait five minutes before using the analyzer, allowing the instrument electronics to stabilize.

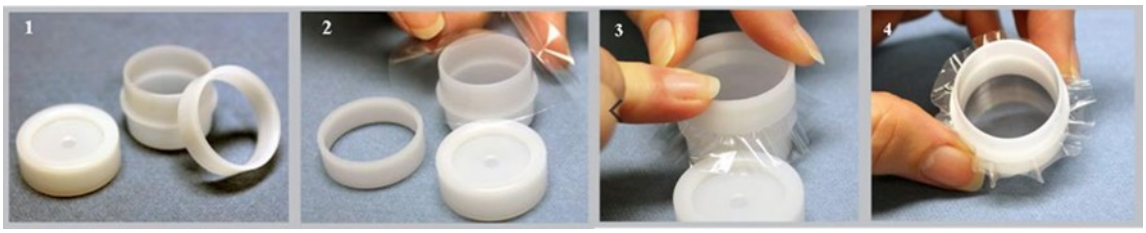


Logon Screen | Warning Screen | Virtual Numeric Keypad | Main Menu

2. Sample preparation

Sample Set A

Sample cups are configured and packaged with a cup cylinder, a collar and a cap. They are assembled by placing a film on top of the sample cup collar. Then the sample cup cylinder is pressed into the collar so that the film is pulled tightly between the collar and cylinder, creating an enclosure. Next, the sample cup is turned over with the film down so that sample material can be placed into the cup.



Load the sample by placing a few scoops of material in the sample cup until the material reached the half height of the sample cup. Place the batting on the top of the sample so that when the sample is inverted, the material is held firmly against the film. Last, place the sample cap on top of the batting and, if desired, affix a label to the cap.

Sample Set B

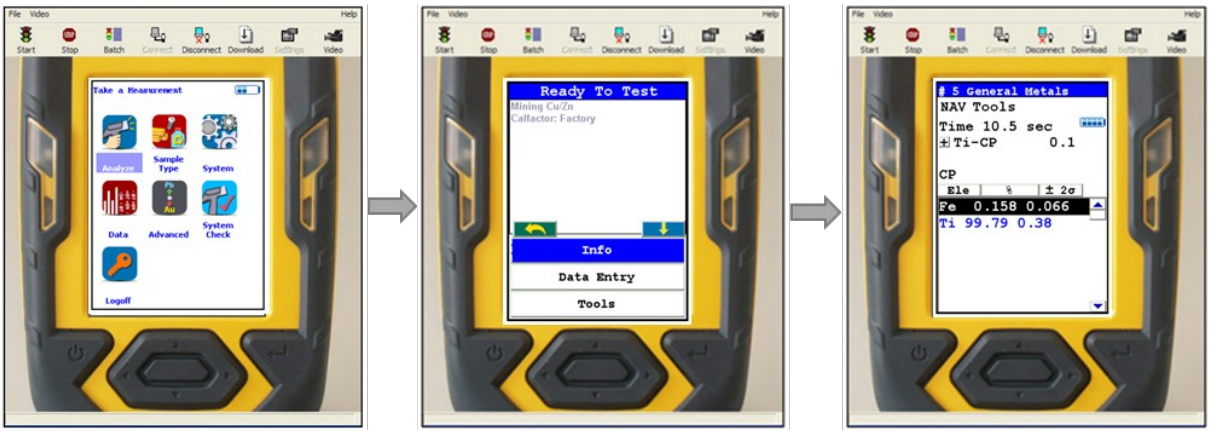
To create a pellet for testing will require using a hydraulic press and the Carver Press Test Cylinder. First take the cylinder and insert the ¾" plunger into the end with the 2" outer diameter. Place the combined plunger and cylinder on the scale and zero the scale. Using a small scoop, place approximately 4 grams of sample inside the cylinder. Next take the cylinder to the hydraulic press and insert the 3 ¼" plunger into the top of the cylinder. Make sure to support the small plunger in the bottom of the cylinder when it is picked up from the scale. Give the cylinder assembly a few light taps to ensure the sample covers the entire top of the bottom plunger.

The hydraulic press consists of a bottle jack, a support frame, and two press plates. To operate the press, you need to open the small valve at the bottom of the jack by turning the knob counter clockwise. To turn the knob, use the slotted end of the jack handle by placing the slots over the fingers on the knob and twisting. This releases pressure from the jack and will allow you to place the cylinder assembly between the press plates. Once the assembly is centered between the press plates, turn the knob clockwise until it stops. Remove the jack handle and insert it into the jack actuator and pump up the pressure to compress the sample. Ideally, the pressure should be increased until the gauge's black needle reaches the red needle on the gauge's face plate. If you are unable to apply that much pressure, be sure to apply as much as possible to ensure a stable compressed pellet.

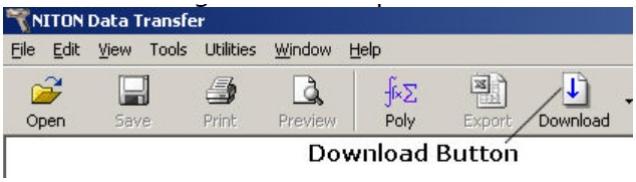
Once the sample has been compressed, use the jack handle to turn the know counter clockwise to release the jack's pressure. Place a piece of cloth or padding on the counter's surface and place the cylinder ejector on top of the padding. Remove the bottom plunger and set the cylinder and top plunger on top of the extractor. Using a rubber mallet, tap the top of the plunger to drive it down forcing the pellet out of the bottom of the cylinder. Once the pellet is out of the cylinder, it is ready for analysis. To prepare for the next sample, use water and a soft brush to scrub off any material remaining inside the cylinder and on the plungers.

3. Analysis

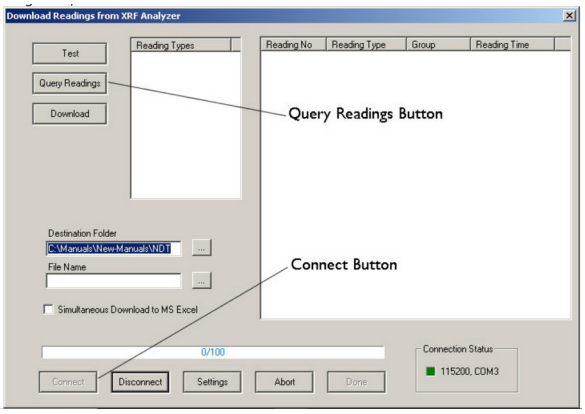
Place the sample cup in the mobile test stand. Open the software ***NDTr*** on the computer. The virtual interface operates exactly as the analyzer would. Select the ***Analyze icon*** by mouse. You will now be at the ***Ready To Test Menu***. Click on the ***Start bottom*** in the toolbar then the test starts. Write down the number in the blue bar which is the sample number in the XRF analyzer. Do not open the cap when the lights are flashing.



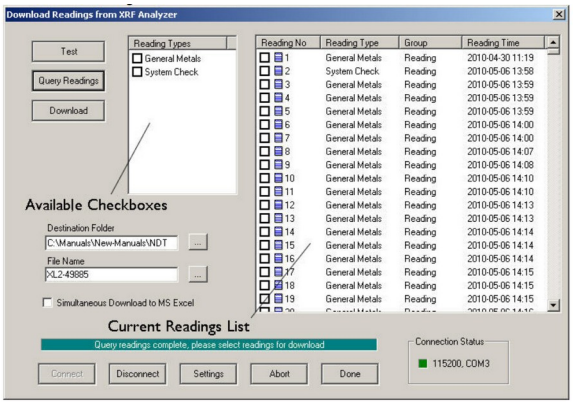
After all the tests are finished, Close the *NDTr* software. Open the *NDT* software. Click the *Download button*.



The *Download dialog box* will open.



Click the *Query Readings button*. This will return a list of all current readings on your analyzer. The list appears in the large white box in the *Download dialog box*. Select the readings that you want to download. Enter a name for the file in the *File Name field*. Click the *Download button*. When the progress bar shows that all the readings are downloaded, click the *Done button*.



You should now see the readings you selected for download displayed, one reading per horizontal line. The data has been saved to the folder and filename you indicated prior to downloading. You can also automatically save reports in .csv format for importing into Excel by click ***Export button*** in the toolbar.