MINERAL ID LAB: DRY XRF & XRD ANALYSIS

Note: *Anything marked in gray is filled in by the instructor. All other fields, checks, and write-ups to be completed by you!*

Name:	Course section ID
Date received: Due Date:	m/d/yy
⚠ The following **Due Date Penalty** applies: -10% / day	☐ 100% or _____%
Overall DRY XRF & XRD ANALYSIS LAB Grade:	%
	/50

Lab Access Badge #: Badge ID

Lab safety training completed on: m/d/yy

Maintenance Infraction(s):
☐ Warning Only! ☐ -5% ☐ -10%
☐ -15% and Lab Revocation

Assigned Lab Equipment BIN number:

Assigned PLM number:

Refer to Manual of Rapid Mineral Identification - Volume I: Mineral ID Tests and Determinations

12.1.2 Semiquantitative Handheld XRF (X-ray Fluorescence)	p.91
12.2 XRD POWDER DIFFRACTION ANALYSIS	p.94

ASSIGNMENTS: I am using ☐ the client sample ☐ alternate sample

/10 COMPOSITION & LAYOUT - 2 point deduction per infraction

The appearance is neat and orderly. Generally, the template is followed. The lab is typed, and graphics and data are electronically prepared and analyzed. Subscripts and superscripts are appropriately used, and equations are explained. The lab is complete with all fields populated. Graphics and data are placed in a coherent form. Proper formatted citations are included.

/10 WRITING & GRAMMAR - one point deduction per infraction

Spelling and grammar are correct. Word repetition and use of first person language is avoided. Statements are factually correct. Appropriate and complete language becoming to a professional report is used.

/20 METHOD, EXECUTION, DATA COLLECTION - one or multiple point deduction per infraction

The METHOD (NOT Procedure or Instructions!) is adequately described and explanation for using the METHOD is given. Appropriate methodology of the lab is evident from the writing and data. Make / Model of instrumentations used is indicated. Any software or App used is cited. For numerical data, significant figures are watched and applied and precision is calculated. When appropriate, one or multiple controls are used and described to identify the integrity of the data. Any data inconsistencies are explained.

/10 GRAPHICS, ILLUSTRATIONS, TABLES - one or multiple point deduction per infraction

At minimum a graphical representation of the lab set-up and/or execution is required. All illustrations must be electronically prepared. Pictures should be clear, of high quality, and with neutral background. Graphs should have a title with appropriate axis and unit labels. Graphics need to meet minimum resolution (300 DPI) requirements. All graphics & tables have properly formatted captions. Citation of source must be included in the captions.

ALL ANSWERS MUST BE TYPED USING A WORD PROCESSOR! This includes chemical formulas, equations, tables and special characters. Become intimately familiar with these functions in your preferred word processor. Where graphics are indicated insert the proper graphic or picture. Be familiar with placing and sizing visuals into a written document. Attach your completed document(s) to this sheet!

LAB PROCEDURE: You may work with a partner for collecting data and running the lab experiments. However, this is **NOT** a group project. Each individual in the group is responsible for his/her own lab write-up, which includes OWN pictures, data tables, graphics, etc.! Do **NOT** copy and share except for RAW data!!!

For ALL assignments use a citation's database:

ZOTERO citations database	ZOTERO is a citations database that incorporates itself into Word and your Browser.	Free open source software available at https://www.zotero.org/

Note: You may use a different citations database, if desired. But you MUST use a citations database!

NOTE: After downloading and installing ZOTERO, take an hour or two to become familiar with the operation of the software. One of your first tasks will be to set the citation style to the United States Geologics Survey (USGS) format in ZOTERO. The USGS citation system will be required for this course and your final product.

Loading the USGS citation format into ZOTERO: Once Zotero Standalone is installed, click on "Preferences" under the Edit tab. Once there click on "Cite" and go to the "Styles" tab. Click on the "Get additional styles..." below the Styles Manger Box. When the Zotero Styles Repository Window opens, click on the "geology" button within the "Fields" section. A list of geology journals should now appear. Scroll down to the U.S.Geological Survey and click on it. The U.S.G.S style will now be part of your Zotero system and you can set your citations to be formatted accordingly in Zotero.

General Writing Instruction Summary:

- Use professional language, which means AVOID first person expressions such as "I", "we", "our". Use normal prose, active voice and third party language. Do NOT use informal wording, contractions, jargon, slang terms, or superlatives. Exclude similes/metaphors (and humor!)
- Use present tense to report well accepted facts, e.g. 'Pyrite is a sulfide mineral'. Use past tense to describe specific results, e.g. 'When acid was applied, the specimen effervesced'
- Be quantitative wherever relevant (stats, numbers etc.).

Subscript & Superscript: Use appropriate subscript and superscript, especially when it comes to chemical formulas and mathematical units..
Acceptable examples: 2.9 g/cm^3, H_2O, PO_{43-}, a_g=9.8m/s^2
Unacceptable examples: 2.9 g/cm3, H2O, PO4 3-, ag=9.8m/s^2

- Use precise concrete language, no ambiguity e.g, 'correlated' ≠ 'related'. Use simple language – no unnecessary "frills" (distractions). Pay attention to sentence structure and grammar

COMPILING TABLES, FIGURES and EQUATIONS

GRAPHICS are the heart of any report. Nothing is more true than in science that a picture is worth a thousand words. Always compile graphics first and then write your text focusing on your graphic. In order to create good graphics and photographs, follow the instructions below.

Cameras and Photos:

Cell phones with a 7.2MP camera are usually ok. When taking pictures, lighting and background is key. This means dark objects should be photographed with light or white backgrounds (sheet of paper) and visa versa for light objects. Having adequate lighting will also help to get sharp, crisp, in focus pictures. Blurry and out of focus pictures are not acceptable.

Graphics:

You should be able to modify, enhance, annotate or overlay graphics. Minimum resolution is 300dpi. Make sure graphics are crisp, clear and any label is easily readable. All graphics should have a caption stating the author and/or citation. Preferably use lossless graphic formats, such as .tif or .bmp. Unfortunately .jpg is NOT lossless and will degrade a little every time you open and save it again in order to manipulate picture contents.

Graphics Editor Software: You will need a graphics editor and learn how to use it. Windows comes with its default Windows Paint editor. It is found in the Windows Accessories Folder. For a more advanced option with many more professional features you may try GIMP: https://www.gimp.org/ This is a FREE, open source image editor working across all computing platforms (Windows, Apple, Linux, etc.). The software comes with ample documentation and examples on how to manipulate any picture or graphic.

Screen Capture Software: A screen capture or snipping software is advantageous in order to only grab the part of a graphic from the screen that is important or necessary. Windows 10 comes with a default snipping tools, such as "Snip & Sketch" found in its own folder or "Snipping Tool" found in the Windows Accessaries Folder.
Note: When snapping a picture, make sure it is large enough on the screen to capture enough pixels to have adequate resolution for the final product.

NOTE: Compiling acceptable and good looking graphics and photographs is very involved and can not be rushed. These are often the heart of any report and should be compiled BEFORE writing. Last minute thrown together graphics will without doubt lower your grade on the client report significantly.

COMPOSITION, LAYOUT, WRITING & GRAMMAR SUMMARY

Language

The following list is an example of common faults in language usage and attribution.

Errors / Mistakes / Faults	Examples with margin *Fault Counts & Codes*	
Spelling:	| |	The mineral florite has a mohs hardness of four.
incl. capitalization errors & spacing	|	Nicolas Steno__was trained in the classical texts on science.
Grammar:		
incl. punctuation, superfluous words, transpositions	|	Isometric crystals are also isotropic Here light propagates at the same speed.
	| |	Rocks are composed of many ~~many~~ minerals mixed.
Style:		
incl. paragraph, repetitive expressions / words	|*Para.*	... in the geologic sciences.¶Near the end of the 19th a new theory ...
erroneous expression / words,	|||*rep*	... is a light colored mineral. These light colored minerals are often light...
sub- or superscription,	|	Stalactites hang from the sealing? of a limestone cave.
unprofessional style,	|*sup*	The density of quartz is 2.65 g/cm3.
word insertion	| |	I was investigating the outcrop with my group.
	|	Sodium sulfate forms a chalky, ~~incoherent~~ precipitate. *amorphous?*
Sentence:		
incl. grammar, run-on, strings of nouns	|	The density of gold is greater then? the density of silver.
	|	Pyrite has a symmetrical crystal structure, it is cubic.
	| |	Skarn mineral zonation? is apparent in the sample.

Content

Errors in content are spelled out. Severe infractions may count for multiple errors.

Errors / Mistakes / Faults	Examples with margin *Fault Counter & Codes*	
Unclear / erroneous statements	*|unclear, units?*	Mohs hardness of the mineral in question is 16.5.
False / nonsense	*||Nonsense*	~~Glaciation cause severe metamorphism of the region~~

TABLES, FIGURES & EQUATIONS SUMMARY

Tables

Each table must be sufficiently complete that it could stand on its own, separate from text.

Only HORIZONTAL lines are allowed in tables. Do NOT use any VERTICAL lines.

DO consecutively number and caption tables and refer to them accordingly within your text. Captions go ABOVE the table.
DO provide a short description of your table within the caption.
DO place your tables appropriately, closest to their mention in the text.
DO make sure tables are legible and reproduce well. Print can be smaller than text, with an 8pt size minimum.
DO appropriately label columns. Do NOT forget units for numeric values.

AVOID splitting tables across pages.

Figures

Each figure must be sufficiently complete that it could stand on its own, separate from text.

DO number and caption figures consecutively and refer to them accordingly within your text. Captions go BELOW the figure.
DO provide a short description of your figure within the caption.
DO place your figures appropriately, closest to their mention in the text.
DO use appropriate citations for figures that are NOT your own. If you use a figure that has been modified by you, the phrase "modified after...." is most appropriate. Photos should show the name of the photographer.
DO make sure figures are legible and reproduce well. Print can be smaller than text, with an 8pt size minimum. Use a minimum resolution of 300dpi. This is a common publication standard.

AVOID cluttering of figures with too much detail. Simplify if necessary.
AVOID moire patterns in photos, a nuisance in copied or scanned pictures. Most scanners come with a moire pattern removal tool.

GRAPHS
Understand graphs: Bar graphs and/or line graphs are used when plotting nominal vs. ratio or interval data. Scatter plot graphs are used when plotting numeric vs. numeric data.
DO make sure that the graph axis are appropriately labeled and scaled. Axis should have titles as well as scalar units.
DO use electronic means to generated graphs. Hand drawn graphs are no longer acceptable.

Equations & Computations

- ☐ Equations should contain explanation of symbols used.
- ☐ A reader should be able to follow where your values or numbers come from. Indicate accordingly.
- ☐ WRITE DOWN UNITS!!!!
- ☐ Show equations used before indicating any computation

Acceptable Example: $F = m \times a = 0.034kg \times 9.8 {}^{m}/_{s^2} = 0.33 {}^{kgm}/_{s^2}$

where m is mass of the object in kg as determined with a triple beam balance and a is the gravitational acceleration. F indicates force expressed in kgm/s^2 or N (Newtons).

Unacceptable example: The answer is 0.33. This is obtained by multiplying gravity by 0.034.

Preamble

Creating and Manipulating Graphics for Reports

GRAPHICS EDITOR SOFTWARE: You will need a graphics editor and learn how to use it. Windows comes with its default Windows Paint editor. It is found in the Windows Accessories Folder. For a more advanced option with many more professional features you may try GIMP: https://www.gimp.org/ This is a FREE, open source image editor working across all computing platforms (Windows, Apple, Linux, etc.). The software comes with ample documentation and examples on how to manipulate any picture or graphic.

SCREEN CAPTURE SOFTWARE: A screen capture or snipping software is advantageous in order to only grab the part of a graphic from the screen that is important or necessary. Windows 10 comes with a default snipping tools, such as "Snip & Sketch" found in its own folder or "Snipping Tool" found in the Windows Accessaries Folder.

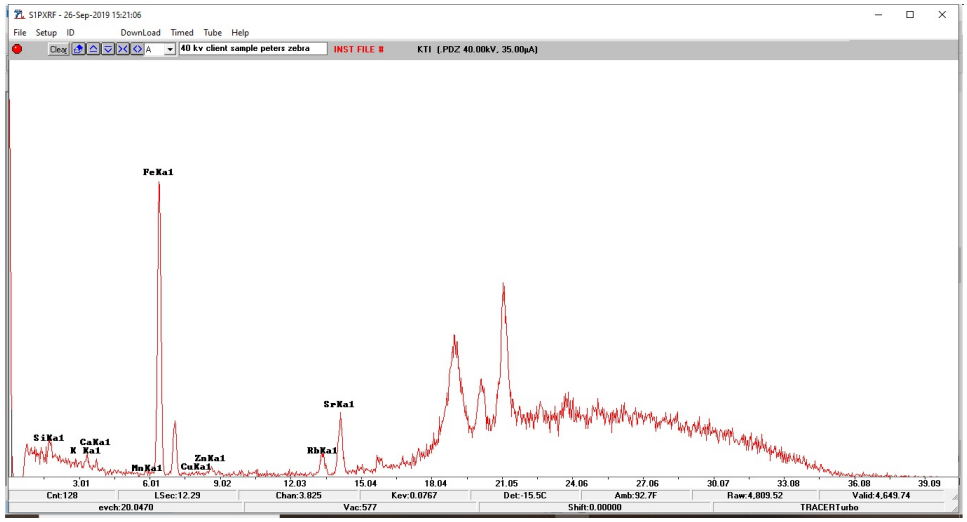
Note: When snapping a picture, make sure it is large enough on the screen to capture enough pixels to have adequate resolution for the final product.

Example

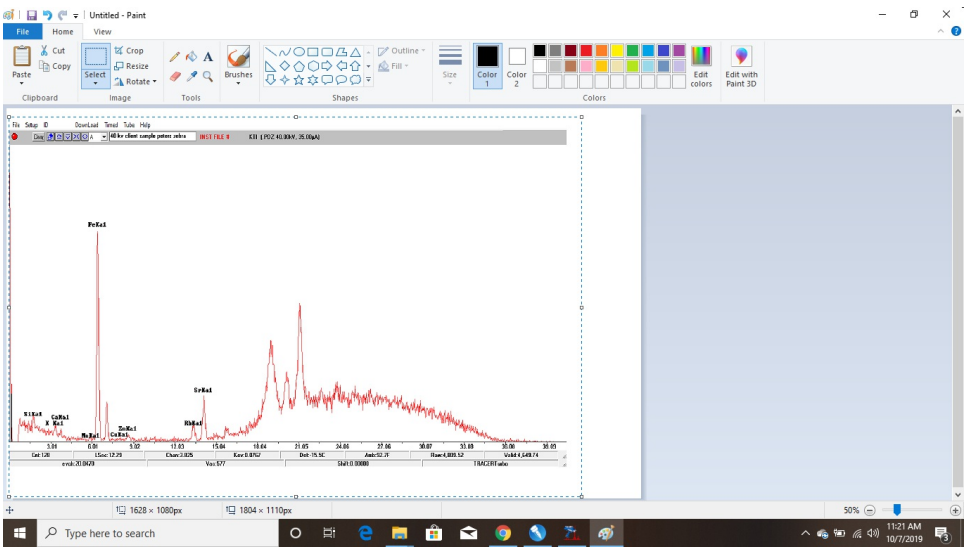
Overview of graphic manipulation for XRF data processed through the S1PXRF software:

1. Obtain the complete data and label the energy peaks.

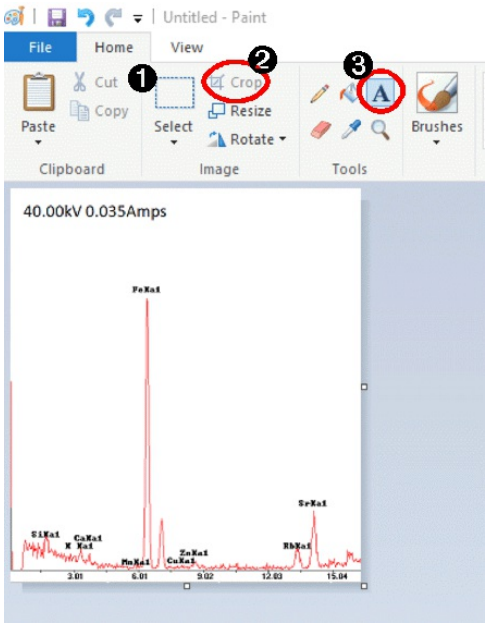
Notice: The labels are too small for report presentation and much information is redundant. Therefore your graphic needs to be manipulated.



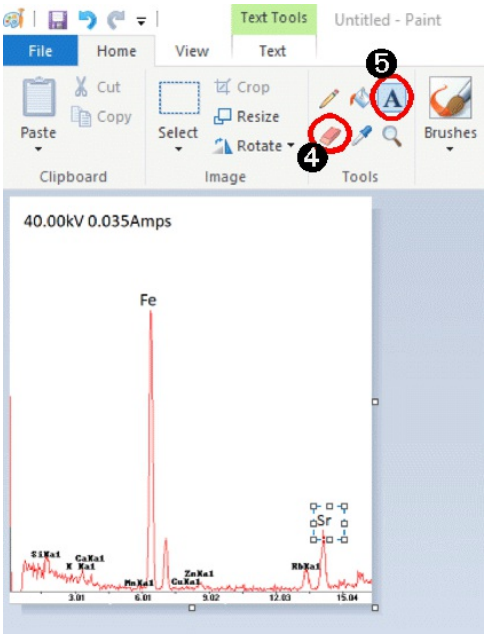
2. Open your graphic in a graphics editor, such as Windows Paint as in this example:



3. Use the Crop Function to eliminate any superfluous part in the graphic by first selecting the area to be maintained (1) and then clicking the crop symbol (2).



4. Add any relevant information, that was cut during cropping such as voltages for the obtained spectra by using the Text Function (3). Make sure that the font is large enough to be crisp on your final graphic.



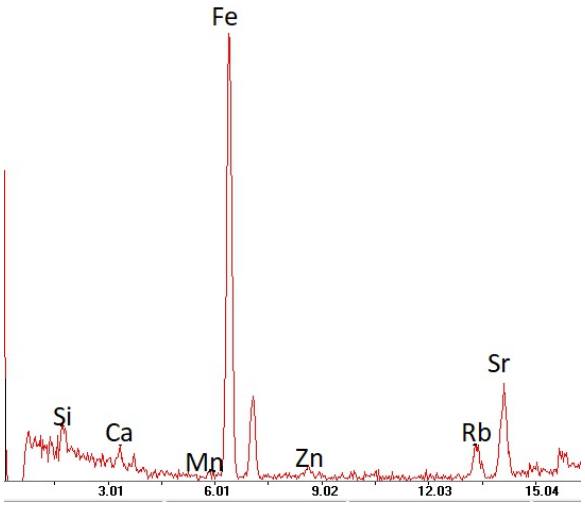
5. Replace any inadequate labeling (too small, too confusing) by either

(a) erasing the original label through the Erase Function (4) and then adding new labels through the Text Function (5) [shown on the left for Fe and Sr]

or (b) by saving / exporting the original XRF graph without the energy peak labels, cropping it and then adding the labels later using the Text Function in the graphics editor (5).

6. Once completed, save your graphic as a .bmp or .tiff file. These are considered "loss-less" graphic formats. Try to avoid .jpg, because the compression algorithms will lose a little resolution every time you edit and re-save a jpg file. Your final product should be of a good enough resolution (min. 300dpi) to look crisp when printed.

40.00kV 0.035Amps



Example of a finished graphic.

Note: The x-axis labels should have been edited as well to show at least a 10pt font size when printed.

Comparison: 12pt 10pt 8pt 6pt

MINERAL ID LAB: DRY XRF & XRD ANALYSIS

XRF ANALYSIS

Name:	Course section ID

POPULATE AND COMPLETE THE FOLLOWING:

Description of METHOD (NOT procedure or instructions) - One paragraph; 3 - 5 sentences:

Instruction:
Using our Brucker Handheld XRF, run the sample analysis according to instruction. No special sample preparation is necessary. Make sure you test a FRESH sample surface only, especially in major element mode.

CAUTION: X-rays. Follow safety instructions! **Note:** Collected data and S1PXRF software will be emailed to you!

1 - Run a trace element scan: 40keV - No filters. Collect /record data.

2 - Run a major element scan: 15keV - No filters. Collect /record data.

3 - Open you pdz data files in S1PXRF. Manipulate the graph until an applicable overview of the sample analysis is portrayed. Label identified major peaks with the appropriate corresponding chemical element. You may have to manipulate

4 - Identify chemical elements as those unequivocally present and those occurring as trace amounts!

Check if CLIENT SAMPLE ☐Yes ☐No

[Insert Low Energy XRF Spectra - 15.00kV. Erase this text when inserting!]	Elements present: Probable Element Traces: Comments:
[Insert High Energy XRF Spectra - 40.00kV. Erase this text when inserting!]	Elements present: Probable Element Traces: Comments:

Note: Under "Comments" you may allude to restrictions of analysis, e.g. sample depth, etc, especially when no spectra from a fresh surface can be obtained. This is most critical for Low Energy spectra! Clients looking for elusive precious metals or similar should be reassured that the element(s) may be present but not detected because of penetration depth and/or small analytical area of the whole sample.

MINERAL ID LAB: DRY XRF & XRD ANALYSIS

XRD SAMPLE PREP & RUN

Name:	Course section ID

POPULATE AND COMPLETE THE FOLLOWING:

Description of METHOD (NOT procedure or instructions) - One paragraph; 3 - 5 sentences:

Instruction:
Prepare a finely powdered sample according to the Lab Manual and mount in sample holder as instructed!

CAUTION: X-rays. Follow safety instructions! **Note:** Bring USB drive for data download. Our XRD unit uses an older computer with NO internet connection.

1 - Mount sample holder with sample in the XRD tray.

2 - Run XRD pattern scan. .

3 - Collect data, usually as a tab delimited ASCII file (.txt), as well as the XRD pattern graphic (bmp).

4 - Import the .txt file into Excel by opening the .txt file in a text editor (MS Notepad) and remove ALL header down to the column data. Rename and save as a new .txt file.

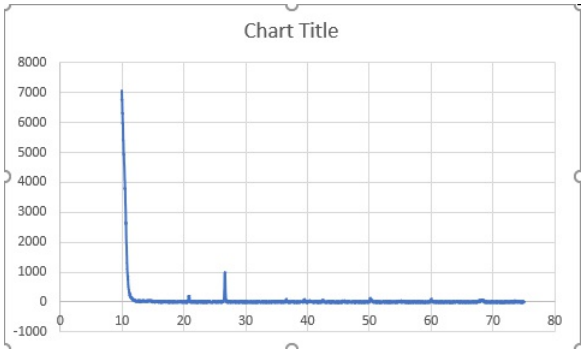
5 - Now open this NEW .txt in Excel. You should get three columns A, B, C populated with numbers. Usually C is not needed and can be erased. Column “A” shows the 2theta angles and column “B” the X-ray intensities. Further processing of the file explained in the next segment.

Check if CLIENT SAMPLE ☐Yes ☐No

MINERAL ID LAB: DRY XRF & XRD ANALYSIS
XRD DATA INTERPRETATION & REPORTING

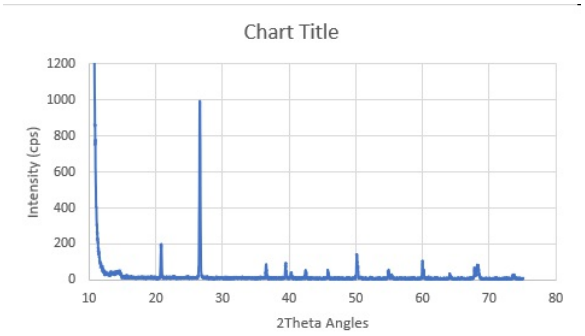
Name:	Course section ID

XRD Data Plot and Analysis:



6 - Highlight Columns A and B and plot as **XY (Scatter) Graph** with A on the x-axis and B on the y-axis.

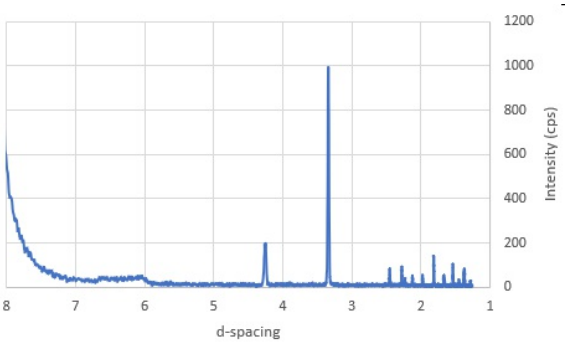
You will notice that the graph has very high counts around 10° 2Theta interfering with the interpretation of actual peaks and that the x-axis has a big empty spot before the 10° mark as shown in this example.



7 - Fix the graph by manipulating the x and y axis:
7a - Set x axis starting value to 10; Label x axis
7b - Set y axis maximum value a little bit over highest peak, e.g.1200 and set lowest value to 0; Label y axis

8 - Remove chart title, since it is not needed. Save your graph in a graphics file format for later use.

9 - **IMPORTANT:** When you present your results using this graph with 2Theta angles plotted on the x-axis it is imperative that you MUST show the data for the X-Ray tube used, e.g. Type of tube and Kα and Kβ wavelengths. There are multiple x-ray tubes in use and each tube will have peak patterns at different locations for the same mineral.



10 (optional) - In order to avoid the problem presented under #9 and for easy comparison with available mineral X-ray data patterns, it is advantageous to convert your x-axis data from 2Theta angles to d-spacings using Braggs equation. Manipulate your graph accordingly. However, by doing so, x-axis peaks will show in reverse order. To fix this, adjust the x-axis format by checking the "plot in reverse order" box.

Hint: Braggs Equation processing in Excel when using a Cu X-ray tube. Place the following equation in Field C2, then copy to all other fields in column C to end of data: =(1*1.5406)/(2*SIN(RADIANS(A2/2)))

XRD Data Plot and Analysis cont'd:

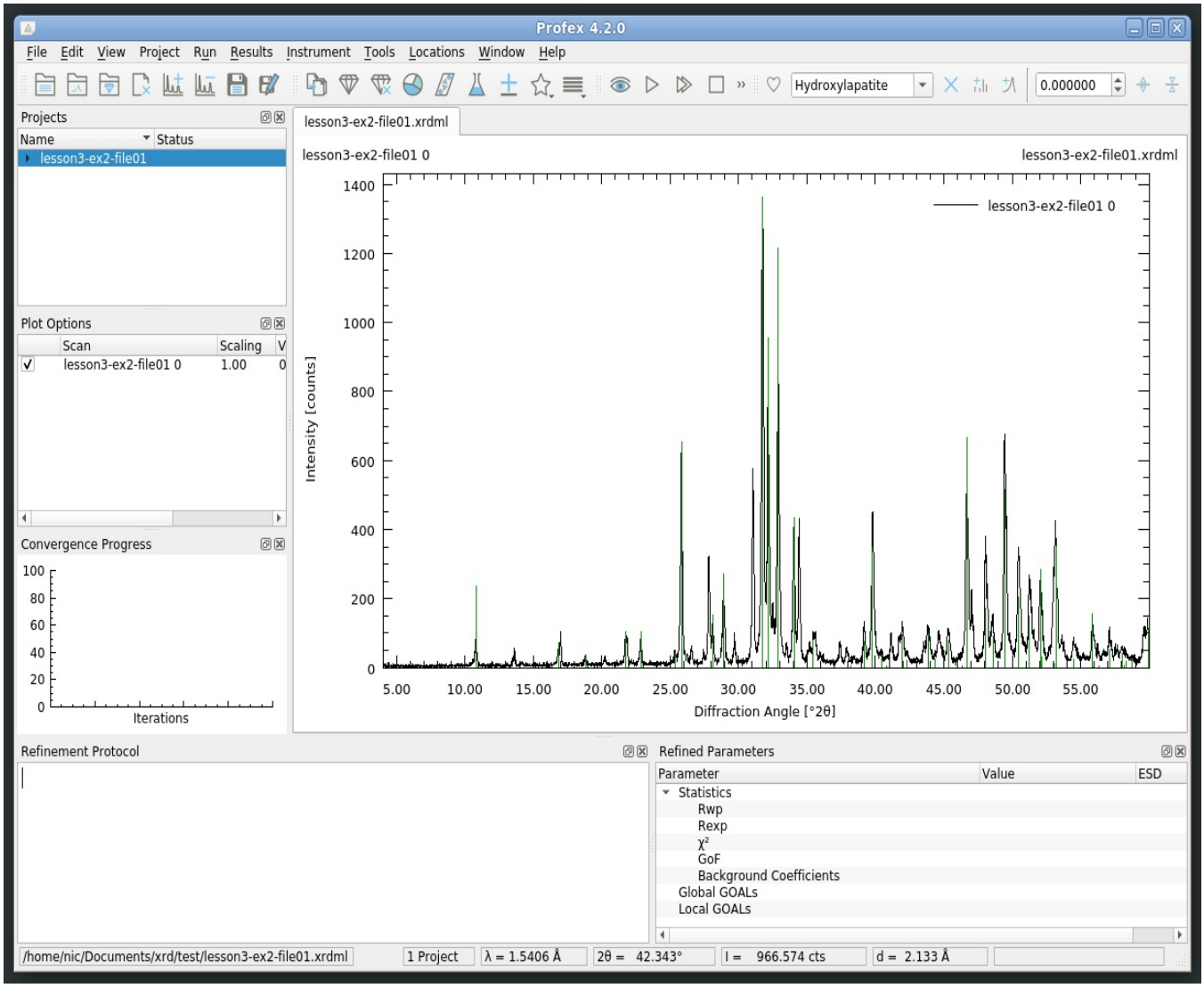
11 - GENERAL GRAPH INTERPRETATIONS:
11a - If your graph has NO peaks than you have an amorphous material with NO crystal structure, such as glass, amber or opal.
11b. - If you just have a few peaks with very high intensity counts you will probably have just a single pure mineral (as in the example given above under #6 through #9).
11c. - Smear slides (double sticky tape with mineral powder sprinkles for reconnaissance assessments or small sample quantities) will usually yield lower intensities from the get go, which may be harder to interpret..
11d. - If you get a good peak very early, around "2Theta" 10° to 13°, then you will most likely have a clay mineral.
11e. - If you have many peaks with lower intensity counts you will probably have a mixture of minerals called multiple phases. In this case you will need to separate the phases to positively identify an unknown mineral.

12 - SEPARATING PHASES:
12a - There is some software that will try to identify and separate multiple XRD phases automatically and can even provide estimated percentage concentrations for each phase. Most of these are comercial and rather expensive. One FREE software is called Xpowder and can be found at http://www.xpowder.com/. The use is limited to 300 days for free. Be aware that the learning curve for some of these software solutions can be steep.
12b - You can separate phases manually using suspected mineral XRD data overlays. To do this go to the extensive and free RRUFF.org database at http://rruff.info/index.htm. Search for the suspected mineral and download its XRD data either as a graphic (2Theta vs Intensity Plot) and/or as RAW or ASCII files. The later you must process using the procedures explained under #6 through #9 above.
IMPORTANT: The X-axis of both graphs, the unknown and the suspected known minerals MUST match exactly in size / scale for this procedure to work. Therefore, create a graph or stretch or shrink the downloaded graphic to match the X-axis of your sample plot. Then see if you get an exact peak match. If you do, label these peaks with the identified mineral name and process remaining peaks for unknown mineral identification.

13 - Complete the analysis by picking the three highest peaks, setting the highest one to 100% and list their exact corresponding d-spacing as discussed during lecture and described in the manual. Those with indicated multiple phases will have to go through a process of peak elimination to do single phase (mineral) identification.
Use the webmineral XRD site http://www.webmineral.com/help/XRayDiffraction.shtml and enter your data as discussed and shown in the manual.

PROFEX - OPEN SOURCE XRD SOFTWARE

XRD data interpretation software packages are usually expensive. There is an Open Source software out of Switzerland that is really good and free of charge called PROFEX. https://www.profex-xrd.org/



This software allows identification of possible mineral phases, which is really powerful.

The author has an own YouTube channel with instructional videos on how to use the software. Please go to https://www.youtube.com/channel/UCNxka_-vfLfqGdRO-cIHA4A to watch the tutorials.

There are also written lecture handouts available covering a variety of topics on how to use PROFEX. https://www.profex-xrd.org/?page_id=68

It is highly recommended to download and use PROFEX for analyzing your XRD results. It is a learning curve, though.

Note: Remove ALL text in square brackets [ ]!

XRD Analysis:

[(Paste your MEASURED XRD pattern graphic here)] [Label peaks especially for multiple phases]

X-ray Pattern

Major Peaks and d-spacing: [Sorted according to your measurement]				
	2 θ			
	d Å			
	I/I_1	100		

[leave the wavelength and Rad data presented here unless different x-ray tube is used] λ 1.5405 Rad: $CuK\alpha_1$

XRD pattern interpretation / comments:

CITATIONS: if applicable! ALL citations should be processed through the ZOTERO citation database software, freely available at https://www.zotero.org/

As minimum citation, any software used and the RRUFF data base should be referenced!

Citations should follow the USGS citation standard!

GENERAL NOTES:

- If you are using the CLIENT SAMPLE, then everything in the **green framed fields** needs to be transferred to your final client report as indicated in the report outline and template.
- Pay particular attention to the grading and comments / feedback associated with the **green framed fields**. Your grade on the final report depends in part on you incorporating these corrections.