MINERAL ID LAB: WET GEOCHEMICAL 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 WET GEOCHEMICAL 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 LABORATORY CHEMICAL ANALYSIS p.83

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_4^{3-}, 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	|	Isometric crystals are also isotropic Here light propagates at the same
words, transpositions	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,	|	The density of gold is greater then? the density of silver.
strings of nouns	|	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.

MINERAL ID LAB: WET GEOCHEMICAL ANALYSIS
SAMPLE DIGESTION

Name:	Course section ID

POPULATE AND COMPLETE THE FOLLOWING:

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

Instruction:

You will need a small sample fragment to be destroyed, about 2 g (2,000 mg) worth. If you have very little sample to work with, do the powder XRD analysis first. You can then use this powder prepared for XRD work to run the geochemical analysis.

Materials: Small sample fragment, mortar and pestle, analytical balance, weighing dish, mesh sieve
Depending on the digestive procedure used, you will also need acids, chemicals, beakers, etc.
Please follow the instructions in the Lab Manual precisely.

Chose digestive method:

☐ For suspected silicates use the lithium metaborate flux digestion. This is probably your safest bet!

☐ For metallic and semi-metallic ores, the aqua regia digestion may suffice.

Note: If sample is mailed to an analytical lab, ignore the digestive method.

PREP - Make sure your sample is clean / fresh with no impurities or oxidized surfaces.

1 - Powder a small fragment to 200 mesh (75 μ)

2 - Follow procedure as explicitly outlined in the lab manual. Record readings in the table below!
Make sure you weigh and record everything! See below

Check if CLIENT SAMPLE ☐Yes ☐No

Sample Weight (g):		Flux Weight (g):	
Wet Digest Weight (g):			

Note: If sample is mailed to an analytical lab, just record the sample weight of the material mailed to the lab.

LOSS-ON-IGNITION (LOI):

Note: If sample is mailed to an analytical lab, ignore LOI procedures. Use LOI data reported by the analytical Lab.

1. Get a heat resistant weighing boat. Weigh about 1,000 mg of a 200 mesh sample split with the analytical balance to at least 3 decimals exactly directly into the weighing boat.
2. Preheat the furnace to 1,000°C. Place the weighing boat with your sample into the hot furnace. Once 1,000°C is reached, start timer for 30 minutes.
3. After 30 minutes remove weighing boat and let cool.
4. Weigh sample again on the analytical balance to at least 3 decimals (just the sample, NOT the boat).
5. Calculate %LOI: (Sample weight - Heated weight) / Sample weight x 100

LOI	Dry Sample weight (exact to 3 decimals)	Weight after LOI (exact to 3 decimals)	% LOI
(Sample weight - Heated weight) / Sample weight x 100			
Calculation:			
Interpretation:			

NOTE: LOI is useful to identify hydrated and/or carbonate minerals, as well as those with volatile contents.

NOTE: If you have only small sample amounts you may use the LOI sample to do the sample digest. Otherwise use fresh sample for the wet geochemical analytical procedures.

IMPORTANT NOTE: Your digest should be analyzed ASAP, especially the analysis for Si in solution. Within 24 hours is preferred.

MINERAL ID LAB: WET GEOCHEMICAL ANALYSIS
ICP ANALYSIS & CALCULATIONS

Name:	Course section ID

IMPORTANT NOTE: ICP analysis should immediately follow the sample digestion within 1 hour. Plan your laboratory time accordingly. If a longer period exists between the digest and the ICP anlysis, the HACH method described below should be used for Si analysis. Other elements are usually fine.

POPULATE AND COMPLETE THE FOLLOWING:

Description of analytical METHOD (NOT procedure or instructions) - One paragraph; 3 - 5 sentences: (If sample is mailed to an analytical lab, summarize the method from that lab):

Instruction:
1 - Prepare a sample blank by using the exact same routine and chemicals you used with your wet digestion, but without any sample added. This will be your blank to be analyzed.

2 - Under the direction of the Lab Coordinator, analyze your sample blank and your sample using the ICP-MS instrument. Record any dilution applied!

NOTE: DIGESTS MUST BE FILTERED THROUGH **45µM FILTER** BEFORE ANY ANALYSIS. FOR ICP-MS WORK, SAMPLE DIGEST MUST **NOT EXCEED 1.5MS/CM (1,500µS/CM)** ELECTRIC CONDUCTIVITY (EC). OTHERWISE YOU MUST DILUTE! FOLLOW FILTERING AND DILUTION INSTRUCTIONS!	Dilution:

3 - Record ICP analytical results in the table below! WATCH YOUR CONCENTRATION UNITS! Leave all zeros or non-detected blank!

Sample Weight (g):		Flux Weight (g):	
Wet Digest Weight (g):			

Note: If sample is mailed to an analytical lab, just record the sample weight of the material mailed to the lab.

NOTE: Use Excel or similar to calculate and populate the following table. You may attach your Excel spreadsheet to this lab as long as lines and columns are preserved as indicated.

		Si	Al	Ca	Mg	K	Na	Fe	Mn
1	**Sample** ICP values **ppb**								
2	**Sample Blank** ICP values **ppb**								
3	**Sample - Sample Blank ppb (if negative record as 0)**								
4	Multiply by dilution factor **ppb**								
5	**Recalculate line 4** to dry sample concentrations **ppm** **(see examples in Lab Manual)**								
6	Oxide	SiO_2	Al_2O_3	CaO	MgO	K_2O	Na_2O	Fe_2O_3	MnO
7	**Recalculate line 5** to oxide concentrations **%** **(1% = 10,000 ppm)**								

		Ti	P	Cr	As*	B**	Ba	Be	C***
1	**Sample** ICP values **ppb**								
2	**Sample Blank** ICP values **ppb**								
3	**Sample - Sample Blank ppb (if negative record as 0)**								
4	Multiply by dilution factor **ppb**								
5	**Recalculate line 4** to dry sample concentrations **ppm** **(see examples in Lab Manual)**								
6	Oxide	TiO_2	P_3O_4	Cr_2O_3	* Volatile! Partial loss on digestion. ** Part of lithium borate flux used for digestion. Value inaccurate. *** Inaccurate! Graphite crucibles used for digestion				
7	**Recalculate line 5** to oxide concentrations **%** **(1% = 10,000 ppm)**								

Note: If sample is mailed to an analytical lab, steps 1 through 4 can be omitted. Record the results from the analytical lab as PPM in row 5 (WATCH THE REPORTED UNITS if PPB or PPM; convert if necessary).
Line 7 must still be calculated as indicated, unless the analytical lab already reports indicated element as oxide. In this case enter the reported oxide value from the analytical lab.

		Co	Cu	Li*	Mo	Ni	Pb	Rb	S
1	**Sample** ICP values **ppb**								
2	**Sample Blank** ICP values **ppb**								
3	**Sample - Sample Blank ppb (if negative record as 0)**								
4	Multiply by dilution factor **ppb**								
5	**Recalculate line 4** to dry sample concentrations **ppm (see examples in Lab Manual)**								

* Part of lithium borate flux used for digestion. Value inaccurate.

		Sb*	Se	Sn	Sr	Te*	V	W	Zn
1	**Sample** ICP values **ppb**								
2	**Sample Blank** ICP values **ppb**								
3	**Sample - Sample Blank ppb (if negative record as 0)**								
4	Multiply by dilution factor **ppb**								
5	**Recalculate line 4** to dry sample concentrations **ppm (see examples in Lab Manual)**								

* Volatile! Partial loss on digestion.

			Radioactive		Precious Metals			Rare Earth	
		Zr	Th	U	Ag	Au	Pt	Ce	Dy
1	**Sample** ICP values **ppb**								
2	**Sample Blank** ICP values **ppb**								
3	**Sample - Sample Blank ppb (if negative record as 0)**								
4	Multiply by dilution factor **ppb**								
5	**Recalculate line 4** to dry sample concentrations **ppm (see examples in Lab Manual)**								

		Rare Earth and others							
		Er	Eu	Ga	Gd	Ge	Hf	Ho	Ir
1	**Sample** ICP values **ppb**								
2	**Sample Blank** ICP values **ppb**								
3	**Sample - Sample Blank ppb (if negative record as 0)**								
4	Multiply by dilution factor **ppb**								
5	**Recalculate line 4** to dry sample concentrations **ppm (see examples in Lab Manual)**								

		Rare Earth and others							
		La	Lu	Nb	Nd	Pr	Sc	Sm	Ta
1	**Sample** ICP values **ppb**								
2	**Sample Blank** ICP values **ppb**								
3	**Sample - Sample Blank ppb (if negative record as 0)**								
4	Multiply by dilution factor **ppb**								
5	**Recalculate line 4** to dry sample concentrations **ppm (see examples in Lab Manual)**								

		Rare Earth and others			
		Tb	Tm	Y	Yb
1	**Sample** ICP values **ppb**				
2	**Sample Blank** ICP values **ppb**				
3	**Sample - Sample Blank ppb (if negative record as 0)**				
4	Multiply by dilution factor **ppb**				
5	**Recalculate line 4** to dry sample concentrations **ppm (see examples in Lab Manual)**				

MINERAL ID LAB: WET GEOCHEMICAL ANALYSIS
SILICA ANALYSIS Hach™ 10098 & CALCULATIONS
Ignore if Si is analyzed with ICP immediately after sample digestion

Name:	Course section ID

Note: If sample is mailed to an analytical lab that analyzes silica, populate the Analytical Methods field referencing the method used by the analytical lab.

POPULATE AND COMPLETE THE FOLLOWING:

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

Instruction:

VERY IMPORTANT: Use this HACH method if the ICP analysis can NOT be performed immediately after the sample digest. The HACH Si analysis should still be completed within 24 hours after sample digestion. Check with Lab Coordinator concerning the availability of the Hach™ method instrument and chemicals and your own availability to do this lab in order to meet the 24 hours deadline.

1 - Use the same sample digest and sample blank prepared for the ICP analysis.

2 - Use Method Hach™ 8185. Download directly from Hach™ https://www.hach.com/asset-get.download.jsa?id=7639983849 and print out!

3 - Analyze your sample digest to see if it falls below the 100mg/L upper detection limit. If it is out of range, dilute the sample until you are within range. Several tests might be necessary to get this right

If applicable, record the dilution used for final analysis	Dilution:

4 - Record analytical results in the table below! WATCH YOUR CONCENTRATION UNITS!

		Si
1	**Sample** Hach™ values **ppm** (1 mg/L = 1 ppm)	
2	**Sample Blank** Hach™ values **ppm** (1 mg/L = 1 ppm)	
3	**Sample (line 1) - Sample Blank (line 2) ppm (if negative record as 0)**	
4	If applicable, multiply by dilution factor **ppm**	
5	**Recalculate line 4** to dry sample concentrations **ppm (see examples in Lab Manual)**	
6	Oxide	SiO_2
7	**Recalculate line 5** to oxide concentrations **% (1% = 10,000 ppm)**	

Note: If sample is mailed to an analytical lab, just record the results from the analytical lab.

CITATIONS: if applicable! ALL citations should be processed through the ZOTERO citation database software, freely available at https://www.zotero.org/ At minimum enter appropriate citation for the Hach™ 10098 Method.

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.