# Petrology Laboratory Exercise: Crystallization of Magmas

In this exercise you will use three methods to explore the crystallization behavior of a rock of known composition at 1 atmosphere pressure:

* **Experimental method**: Melt your rock at 1300°C and then cool it to 1000°C using a programmable glass melting furnace. Make grain mounts of your run products and identify the phases. We may also use the SEM to image and analyze your run products.
* **Phase diagram method**: Calculate the normative composition of your rock and plot it on the Fo-An-Si diagram; predict the crystallization sequence.
* **Numerical method**: Use the computer program MELTS to predict the crystallization sequence. Also explore differences in crystallized assemblages and evolution of liquid compositions between batch and fractional crystallization.

You will also use the chemical composition of the rock to classify it and to make up REE, tectonic discrimination and spider diagrams and interpret them.

## Procedures

Four powdered rocks are available, so four groups will be assigned a rock to study. Here are the compositions:

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| **SAMPLE** | **AGV-2** | **BCR-2** | **GSP-2** | **QLO-1** |
| SiO2 | 59.3 | 54.1 | 66.6 | 65.6 |
| TiO2 | 1.05 | 2.26 | 0.66 | 0.62 |
| Al2O3 | 16.91 | 13.5 | 14.9 | 16.2 |
| Fe2O3 | 6.69 | 13.8 | 4.9 | 4.35 |
| MgO | 1.79 | 3.59 | 0.96 | 1 |
| CaO | 5.2 | 7.12 | 2.1 | 3.17 |
| Na2O | 4.19 | 3.16 | 2.78 | 4.2 |
| K2O | 2.88 | 1.79 | 5.38 | 3.6 |
| P2O5 | 0.48 | 0.35 | 0.29 | 0.25 |
| Ba | 1140 | 683 | 1340 | 1370 |
| Be | 2.3 |  | 1.5 |  |
| Ce | 68 | 53 | 410 | 54 |
| Co | 16 | 37 | 7.3 | 7.2 |
| Cr | 17 | 18 | 20 | 3.2 |
| Cs | 1.16 | 1.1 | 1.2 | 1.8 |
| Cu | 53 | 19 | 43 | 29 |
| Dy | 3.6 |  | 6.1 | 3.8 |
| Er | 1.79 |  | 2.2 | 2.3 |
| Eu | 1.54 | 2 | 2.3 | 1.43 |
|  F | 440 | 440 | 3000 | 280 |
| Ga | 20 | 23 | 22 |  |
| **SAMPLE** | **AGV-2** | **BCR-2** | **GSP-2** | **QLO-1** |
| Gd | 4.69 | 6.8 | 12 |  |
| Hf | 5.08 | 4.8 | 14 |  |
| Ho | 0.71 | 1.33 | 1 |  |
| La | 38 | 25 | 180 | 27 |
| Li | 11 | 9 | 36 | 25 |
|  Lu | 0.25 | 0.51 | 0.23 | 0.37 |
|  Mn | 770 | 1520 |  |  |
| Nb | 15 |  | 27 | 10 |
| Nd | 30 | 28 | 200 | 26 |
| Ni | 19 |  | 17 |  |
| Pb | 13 | 11 | 42 | 20 |
| Pr | 8.3 | 6.8 | 51 |  |
| Rb | 68.6 | 48 | 245 | 74 |
|  Sb | 0.6 |  |  |  |
| Sc | 13 | 33 | 6.3 |  |
| Sm | 5.7 | 6.7 | 27 | 4.9 |
|  Sn | 2.3 |  |  | 2.3 |
| Sr | 658 | 346 | 240 | 340 |
| Ta | 0.89 |  |  | 0.82 |
| Tb | 0.64 | 1.07 |  | 0.71 |
| Th | 6.1 | 6.2 | 105 | 4.5 |
|  Tl | 0.27 |  | 1.1 |  |
| Tm | 0.26 | 0.54 | 0.29 | 0.37 |
|  U | 1.88 | 1.69 | 2.4 | 1.9 |
|  V | 120 | 416 | 52 | 54 |
|  Y | 20 | 37 | 28 | 24 |
| Yb | 1.6 | 3.5 | 1.6 | 2.3 |
| Zn | 86 | 127 | 120 | 61 |
| Zr | 230 | 188 | 550 | 185 |

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### Making Diagrams

After creating each diagram copy it to the clipboard and paste it into PowerPoint. Save all of your diagrams in one PowerPoint file that you will later present to the class.

1. What rock did your group work on? \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
2. Use the Excel template [NormCalc.xlt](file:///%5C%5CEES2%5CAnonymous%5CEES226%5CNormCalc.xlt) to calculate the normative mineralogy of your rock. What are the normative minerals in your rock, and their percentages?
3. Enter major element concentrations into cells C2:E2 on sheet “My Data” in the Excel template [Alkalinity.xlt](file:///%5C%5CEES2%5CAnonymous%5CEES226%5CAlkalinity.xlt). Is your rock alkaline or subalkaline? What rock type is it classified as in the TAS (Total Alkalis vs. Silica) diagram?
4. For samples QLO-1 and GSP-2 only: Enter the normative composition of your rock into cells B17:D17 on sheet “Ternplot” in the Excel template [TTGternaryplot.xlt](file:///%5C%5CEES2%5CAnonymous%5CEES226%5CTTGternaryplot.xlt). What is the classification of your rock?
5. Enter trace element concentrations into cells B3:B10 on sheet “My Data” in the Excel template [Discrimination.xlt](file:///%5C%5CEES2%5CAnonymous%5CEES226%5CDiscrimination.xlt). Examine the various plots that are automatically produced on the other sheets. What conclusions can you draw about the tectonic environment that your rock formed in?
6. Replace the values in cells B2:B16 in the sheet “Normed REE data” in the template [REEPlot.xlt](file:///%5C%5CEES2%5CAnonymous%5CEES226%5CREEPlot.xlt) with the REE concentrations in your rock in ppm. Describe the shape of the REE pattern. Is your rock enriched or depleted in the LREE? Explain why. Is there a Ce or Eu anomaly? Why or why not?
7. Replace the values in cells B2:B29 in the sheet “Normalized T.E. data” in the template [SpiderPlot.xlt](file:///%5C%5CEES2%5CAnonymous%5CEES226%5CSpiderPlot.xlt) with the trace element concentrations in your rock in ppm. What are the characteristic features? What conclusions can you draw about the environment that your rock formed in?

### Experimental Method

First read about Experimental Petrology at: <http://serc.carleton.edu/research_education/equilibria/experimentalpetrology.html>

In this exercise you will grow your own volcanic rock! First take a standard rock powder of known composition. Make a grain mount using immersion oil with an index of fraction of 1.54 (\*note: make sure you clean both the glass slide and the cover slip using alcohol or acetone before making the mount). Use the optical microscope to identify the minerals in your rock.

1) List the minerals and their approximate percentages.

2) Are the minerals you identified consistent with the rock type?

 Next put ~1g of powder into a Pt crucible, place the crucible in the programmable glass-melting furnace in SC5712, and program the controller to heat to 1300°C at 1000°C/hr and then cool to 1000°C at 200°C/hr. Let dwell for 30 minutes, then remove the crucible from the furnace and drop it into an ice water bath to quickly quench your sample. Remove the glass slug; break off a piece, powder it in a mortar and pestle, and make a grain mount and identify the minerals.

Questions:

1. Describe the textures observed in the grain mount.
2. What minerals are present in your sample? Can you infer a crystallization sequence based on crystal sizes and textural relationships (e.g., inclusion of one phase within another)?

### Phase Diagram Method

Use the normative Fo (or En), An and Si contents to plot your rock composition on the Fo-An-Si diagram.

Questions:

1. List the minerals in order of crystallization, and estimate the temperature at which they start to crystallize. Estimate the proportions of each phase in the rock when it solidifies.



### Numerical Method

First read about MELTS at: <http://serc.carleton.edu/research_education/equilibria/melts.html>

Use the following procedures to run the MELTS program on a Macintosh computer in SC 6703:

1. Have the TA log you in as Administrator
2. Open XDarwin by clicking the “X” icon on the bar at the bottom of the screen
3. At the command prompt in the upper-left of the screen type “cd desktop/Melts”
4. Type “./Melts-public”
5. Type “n”
6. Click on the upper-left side of the screen to place your Melts window.
7. Type in your rock composition.
8. Choose “Intensive Variables: f O2 constraint” and select “Hm-Mt” for the Hematite-Magnetite buffer. This is the most oxidizing buffer, closest to the ambient fO2 in which you will perform your experiment.
9. Choose “Intensive Variables: T, P” and set “Start” and “Stop” conditions as 1200°C and 1 bar pressure.
10. Choose “Composition: Computer redox state” to calculate the Fe2O3/FeO ratio corresponding to the Hm-MT buffer at the specified conditions.
11. Choose “Composition: Normalize” to recalculate your concentrations so that they sum to 100%.

Now you will use Melts to identify the equilibrium phases and their compositions. Melts uses a thermodynamic database and a numerical procedure to find the most stable assemblage at the specified conditions; it adjusts the list of phases and the compositions of the phases until it finds the set with the minimum Gibbs Free Energy. This is a tricky calculation, and sometimes Melts doesn’t find the correct solution. In those cases you have to adjust the conditions until it finds the correct solution, and then gradually change the conditions until it finds the solution at the desired conditions. Specifically, Melts has trouble converging on a solution near the liquidus or solidus; it can do so more effectively if you slowly approach the liquidus from lower T because it "remembers" the stable phase assemblage from the prior calculation.

1. Go to “Intensive Variables: T, P”. Set the start and end temperatures equal; leave the T increment at 50 or blank (it doesn't like it if you set it to zero). Choose a temperature significantly below the liquidus; I recommend T = 1150°C (note that the temperatures produced by the "Find Liquidus" option are meaningless).
2. Choose “Commands: Execute” to find the equilibrium assemblage.
3. Repeat steps 12-13 while gradually increasing the T until % liquid is approaching 100; I recommend that you end at T = 1200°C.
4. Now use Melts to cool your melt by setting the “Start” T to 1200°C and the “End” T to 1000°C; set the T increment to 25°C. Execute.
5. Close Melts, and then double-click on the “Melts” folder on the desktop. Double-click on the “Melts.out” file. Read off the proportions of each phase at each temperature on your cooling path and record them in a spreadsheet; make a single plot showing how the wt% of each phase (including liquid) changes with temperature.
6. Open the file “melts-liquid.tbl” in Excel. Make two charts; one plots liquid viscosity (log10 poise) vs. T, and the other wt.% oxides vs. wt% SiO2. The oxides you should plot are wt% Al2O3, wt% FeO + Fe2O3, wt% MgO, wt% CaO, wt% Na2O and wt% K2O.

Questions:

1. How do the proportions of each phase compare with those observed in your run products? Try to explain any discrepancies.
2. Explain the change in liquid viscosity with temperature (I believe this is just for liquid and not liquid + crystals).
3. Does the Harker diagram you made look like ones in your text that we discussed in class (e.g., Crater Lake) for series of cogenetic rocks? Explain.

Summary Questions:

1. Compare the crystallization sequences predicted by the Numerical and Phase Diagram methods and observed using the Experimental Method. Try to explain any discrepancies.
2. How do you know the crystals in your run product grew during the experiment and were not retained from the starting material? How could you test this experimentally?
3. Which method is the most generally useful? Explain.
4. Which method is likely to be most accurate? Explain.