

## Geology 221a – Mineralogy Mineral Synthesis Assignment

The nature of this assignment depends on your mineral, specifically whether it is hydrous or anhydrous. If your mineral is anhydrous, the assignment is to attempt to grow your mineral in the lab from other materials. If your mineral is hydrous, the assignment is to determine its high temperature stability: At what temperature does the mineral break down to other minerals and what are the breakdown products?

### Anhydrous Minerals

- (1) Examine the mineralogical literature to see if others have attempted to synthesize your mineral. You may find a good recipe to follow.
- (2) Pick a chemical composition to synthesize. You may attempt to make an end member composition or an intermediate composition. If your mineral contains  $\text{Fe}^{+2}$ , I recommend making the Mg end member because the iron will oxidize to  $\text{Fe}^{+3}$  in our oven.
- (3) Examine the chemical reagents available in SR-107. Calculate how much of each reagent is needed to make your mineral. I suggest trying to make about 5 grams of your mineral to reduce the effect of weighing errors and adsorbed water. In some cases, you may wish to dehydrate the reagent. If you need other reagents, please ask.
- (4) Weigh out the calculated amounts of starting materials. Mix your sample very well. I suggest using the McCrone Micronizing mill and grinding for 10 minutes. The defects introduced are likely to encourage new mineral growth.
- (5) X-ray your starting material, collecting a good ( $1^\circ 2\theta$  per minute or slower).
- (6) Place your sample in a ceramic crucible with lid. Place the crucible in one of the two ovens at a temperature of your choosing, **but not higher than 1100°C**. If you believe that a higher temperature is needed, please ask for help. **Polyester clothing is quite flammable.** Special caution must be exercised when working at higher temperatures.
- (7) Normally, you should leave the crucible in the oven for 24 hours. You may need to reserve the oven or to share it with one or more other students who can work at the same temperature. X-ray your sample, collecting a good diffraction pattern ( $1^\circ 2\theta$  per minute or slower). Compare the resulting powder diffraction pattern with the diffraction pattern of the starting material.
- (8) Identify the new crystalline solids that grew during your experiment.
- (9) If you succeeded in growing crystals of your mineral, you may be done with your experiments. (If you have only a few small peaks from your mineral, you may wish to grind your cooked sample again and heat it for another day.)
- (10) If you do not see any peaks of your mineral in the diffraction pattern, but do see new peaks, you should identify what crystals have grown, if possible. You may wish to heat your sample again at another temperature or try some different starting materials.
- (11) Write a report describing your experiment. The report should include all important experimental details (sample preparation, temperatures, times), including a list of starting materials and the quantity of each used (in a table). Include as figures x-ray diffraction patterns for the starting material and for any experiment products. Interpret your results and compare them with attempts by others to synthesize your mineral.

### Hydrous Minerals

- (1) Examine the mineralogical literature to see if others have attempted to dehydrate your mineral. You may find a good recipe to follow.
- (2) Grind your mineral to create a powder for the experiment. If you wish, you can use the same powder that you used for x-ray diffraction when you determined your unit cell.
- (3) Place the crucible in one of the two ovens at a temperature of your choosing, **but not higher than 1100°C**. If you believe that a higher temperature is needed, please ask for help. **Polyester clothing is quite flammable.** Special caution must be exercised when working at higher temperatures. Your goal is to determine the temperature of dehydration, so you should start with a temperature that will not lead to dehydration and then try higher temperatures in intervals.
- (4) Leave the crucible in the oven for 2 hours. You may need to reserve the oven or to share it with one or more other students who can work at the same temperature.
- (5) X-ray your starting material, collecting a good ( $1^\circ 2\theta$  per minute or slower). Compare the resulting powder diffraction pattern with the diffraction pattern of the starting material.
- (6) Identify the new crystalline solids that grew during your experiment, if any.
- (7) If your mineral has not changed, repeat steps (3) to (5).
- (8) Write a report describing your experiment. The report should include all important experimental details (sample preparation, temperatures, times). Include as figures x-ray diffraction patterns for the starting material and for any experiment products. Interpret your results and compare them with attempts by others to dehydrate your mineral.