

## Introduction to the X-ray Diffractometer

C:\a-StudioClassroom\minex08.doc; July 6, 2005

We will X-ray the reagents we studied at the beginning of the semester (Exercise 1). You saved the experimental products, right?

We need X-ray data for the reagents out of the bottle and also after they were cooked to 1200°C. That means we will collect eight X-ray diffraction patterns:

aluminum hydroxide before and after  
silicic acid before and after  
magnesium oxide before and after  
calcium carbonate before and after

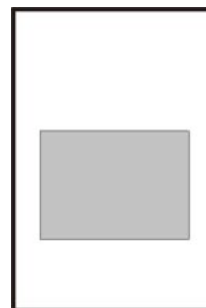
For this round of X-ray work, each group will prepare and X-ray one sample. The instructor and TA will do the others. Then we will share results.

After seeing the results, your job is to use the new information to further interpret the initial heating experiments we did.

### Using the X-ray Diffractometer:

Step 1: Take a small amount of sample and grind it very fine in a mortar. Always keep it wet with acetone while grinding. Work under a hood or have the windows to the room open when you do this so you don't get goofy from inhaling the acetone fumes. The more you grind, the better your X-ray will come out. You should grind until it is "floury" which means the pestle sort of "stubs its toe" as you grind. If you still hear the grinding - gritty noise, you have not ground enough. You need to go way beyond that.

Step 2: After grinding, use a spatula and put a small amount of powder on a glass slide. Then add a drop of acetone and spread it out evenly. A teasing needle or an unbent paper clip works well for this. The powder should completely cover the slide in the area that is going to be hit by X-rays - more or less shown in gray in this drawing. It need not be thick, but no glass can show.



Step 3: Follow TA instructions to get and analyze an X-ray pattern. For analysis we use a computer program. The program will give choices of answers and you have to think and reject things that are absurd. Don't, for example, tell us that you X-rayed iridium-phosphate.

You cannot all X-ray at once, so there will be a signup sheet. Also, you may not X-ray if the instructor or TA are not around to supervise.

Note that it takes about an hour to X-ray a sample.

### Your Report

You already interpreted the weight loss when the reagents were heated. Now you have X-ray data to check your interpretations. Write a report (2-3 pages of text, plus X-ray scans) that describes and explains your X-ray results. Be sure to discuss if they match your earlier interpretations.