PHASE IDENTIFICATION AND QUANTITATIVE ANALYSIS USING X-RAY DIFFRACTION

Introduction

One of the most common uses of an x-ray diffractometer is to identify the phases present in a specimen. If the approximate chemical composition of the specimen is known and if it does not contain too many different phases then one can usually identify all of the phases present and possibly even the concentration of each phase. One way to identify the phases present is to use the diffractometer to obtain a diffraction profile and then index the pattern to figure out what crystal structures are present and what their lattice parameters are. Another way is to simply compare your pattern with every pattern in a database containing just about every organic or inorganic substance. (There are over 100,000 diffraction patterns are in the ICDD database.) In this experiment you will have an opportunity to try the second method. Using the search-match capabilities of this system and the ICDD database you will attempt to identify an unknown substance. Once all of the phases present have been identified you will then attempt to estimate how much of each phase is present by comparing the intensities of selected diffraction peaks to those in standards.

Objective

This experiment has two straight-forward objectives: to identify the phases present in a powder sample and to estimate the relative proportions of each phase in this sample.

Materials

1. Approximately 10 grams of a powder sample.

2. Corundum powder which will be added to your sample.

Equipment

The equipment used during this experiment is:

1. Scintag XDS 2000 x-ray diffractometer

2. Electronic balance (0.01 g sensitivity) and weighing accessories

3. Sample holders for powder samples

Procedure

1. Perform a quick preliminary scan of the specimen over a wide range of 2θ so that you can see where the peaks are. Once you are more familiar with the specimen scan it again at a lower scan rate (1-5 degrees per minute) to obtain a higher quality profile. You should limit the range of 2θ to those regions where the most prominent peaks are located. Run the background stripping and peak finder programs using the default settings and then run the search-match program to try and identify the phases present. When identifying the phases present make sure that all peaks are accounted for.

2. Print out the PDF files for each of the phases identified and note the values of the RIR (I/I_{cor}) parameter. If any of the RIR values are not reported then you will have to generate your own. This will involve mixing up 50/50 samples of each phase present with corundum, scanning the prominent peaks of each, and then calculating the intensity ratios.

3. Rescan several of the stronger peaks of each phase in your sample and then run the RIR program on the raw data. (If the data from step 1 is of sufficient quality you can use that data instead.) Note the compositions reported. How do you know these results are correct?

4. Add about 10% by weight of alumina to your sample and mix it in carefully and thoroughly. Rescan this sample using the same conditions as in step 3 and then perform the RIR analysis. Does this program report the correct amount of alumina? Make the necessary corrections to see if this analysis also gives the same result as in step 3.

Results

1. Positive identification of all phases present in the sample.

2. An estimate of the relative proportions of the phases present.

3. Any additional information that convinces you that the results are indeed correct.

Additional

1. Careful preparation of the samples is essential.