

Identification of Unknown Minerals by X-Ray Diffraction

In this lab, you will use x-ray diffraction as a tool for identifying unknown minerals and mixtures of minerals. The x-ray diffraction pattern acts as a fingerprint of the mineral because each mineral has a unique crystal structure and composition, which results in a unique diffraction pattern.

A database of x-ray diffraction patterns has been compiled by the Joint Commission on Powder Diffraction Standards (JCPDS; their new name is the International Center for Diffraction Data, ICDD) and is continually updated by them. The unknown pattern must be compared with the patterns in the database to find a match. There are thousands of patterns in the database, and so a systematic procedure has been devised to find a match.

Actually there are several procedures, but each is a slight variation of the Hanawalt method. In this method, you make a list of the unknown peaks, that is, their values of d , in decreasing order of intensity. Then you look in the search manual in the section that contains the most intense peak. Within each section the minerals are listed in the order of decreasing d of the second most intense peak.

This procedure is imprecise because the relative intensity of the peaks can change upon changes in the procedure of preparing the sample for measurement or upon changes in composition or structure in complex minerals. Another complicating factor is that the peak positions of complex minerals that have a range in composition shift. Just think about the differences between the patterns for halite and sylvite from last week's lab.

In finding a match, you may have to switch the order of the intensities of the peaks and to be flexible about the values of d . The values of d will not range greatly, though. Also note that once you think you have found a match, every peak must match (the intensities may be different, though).

1. You are provided with the pattern of a single unknown mineral. The data are in a file (Unknown Min) in the Lab 11 folder on the file server. Make a plot of the data using Kaleidegraph and identify the peaks and their intensities. Use the Hanawalt procedure described above to identify it from a search manual. Once you have a match, look at the index card from the powder-diffraction file (in a book of card data for minerals) to see all the peaks, their intensities, and other mineral data.
2. The unknown mineral data are available on the pc in the x-ray lab. Use the program Jade, with an automatic search-match routine, to find a match for your unknown mineral. Is the result the same as your determination from 1?
3. Mixtures of minerals are much more difficult to identify because all the peaks for all minerals are present. Here you have to have some guesses about what could be present and then see whether they are. The data are in the file Mixture on the file server and in the file mixture on the pc in the x-ray lab. This mixture of minerals is from a sample of ground limestone. The identification of minerals in rocks by x-ray diffraction becomes an art. You have to develop a sense of what you may find. Cross out the peaks from a successfully identified mineral to see what's left. Then make another guess, and so on.