A new technique of preparing fine mineral powders for infrared studies is described here. Fine powders can be prepared by self-grinding the crystals in a test tube containing acetone. Both the Christiansen effect and the effect of prolonged grinding with a mortar and pestle are eliminated from the spectra. The powder infrared spectrum of pyromorphite is used to illustrate the spectra obtained with powders ground by various procedures.

During the recent investigation of the vibrational spectra of several geological apatites (Levitt and Condrate, 1968), a marked Christiansen effect (Levitt, 1969; Price and Tetlow, 1948) was noted in the infrared spectra of the lead apatites (pyromorphite, mimetite and vanadinite). KBr pellets of apatite powders ground for short periods by pestle and mortar were used. Figure 1A illustrates the spectra observed for pyromorphite. Prolonged grinding by mortar and pestle produces spectra free from the Christiansen effect. However, significant changes occur in the intensities and positions of absorption bands (see Fig. 1B).

In this investigation, we have developed a technique to prepare powders of crystals possessing appropriate particle sizes without generating unsatisfactory side effects. A few small crystals or particle aggregates were placed in a 10 ml. pyrex test tube. The tube was then half filled with acetone, capped and shaken in a Wig-L-Bug vibrator. Particle-size reduction was accomplished by the impact of the particles with themselves and with the test tube walls. After vibration for about ten minutes, the tube was allowed to stand for a few seconds allowing “large” particles to settle to the bottom. The suspension of the fine particles was then removed with a syringe, placed in another test tube and the acetone was evaporated off. A sufficient amount of fine-particle powder remained in the test tube, from which KBr pellets could be prepared and analyzed by infrared spectroscopy (see Fig. 1C). The changes in spectra caused

1 Presented in Part at the 23rd Symposium on Molecular Structure and Spectroscopy, Columbus, Ohio, September, 1968.
2 Present address:
Ferro Corporation, Technical Center
4150 E. 56 Street
Cleveland, Ohio 44105
Fig. 1. The infrared transmission spectra of pyromorphite (Phoenixville, Pa.) showing Christiansen effect (A), effect of long grinding (B), and results of acetone method (C).
both by the Christiansen effect and by prolonged grinding with the pestle and mortar are eliminated. The vibration time, standing time and type of suspending liquid may be varied to optimize the results. One advantage of this method is the elimination of contaminating media, such as steel from grinding balls, which is a problem in many other particle-size reduction methods. It is expected that the acetone technique of powder preparation will be useful in the infrared absorption studies of many other minerals.

Acknowledgement

The work was supported by a research training grant (PHS Grant No. DE00146) from the National Institute of Dental Research, Public Health Service.

References


