

THE AMERICAN MINERALOGIST, VOL. 49, JANUARY-FEBRUARY, 1964

MICROPROBE TECHNIQUE FOR THE ANALYSIS OF  
MULTIPHASE MICROCRYSTALLINE POWDERSE. W. WHITE, *Materials Research Laboratory, and Mineral  
Constitution Laboratory, The Pennsylvania State University.*

There is a need for a method of obtaining accurate chemical analyses on the individual particles in very fine-grained powders. For many samples it is impossible to separate mechanically the phases for conventional chemical analysis. Likewise the techniques of *x*-ray powder diffraction and light microscopy will not suffice in many cases. Important examples of such materials are natural clay or zeolite mixtures and synthetic oxide powders.

An electron microprobe technique has been developed which involves the minimum amount of sample preparation time and eliminates the possibility of accidentally analyzing more than just one grain of the powder at a time. Grains in the size range of 2 to 20 $\mu$  can be analyzed for all elements from magnesium to uranium. Samples are prepared by first making a dilute suspension of the powder in water or other non-reactive volatile liquid. To insure complete dispersion of the grains it is sometimes desirable to add a deflocculant and to treat the suspension in an ultrasonic cleaner. Droplets of the suspension are then placed on the surface of polished beryllium rods and allowed to evaporate. Special sample holders have been made for the Applied Research Laboratories (ARL) microprobe which accommodate five  $\frac{1}{4}$  inch diameter beryllium rods per holder. Each rod will usually accommodate two drops which gives 10 different samples per sample holder. The liquid evaporates leaving the individual grains scattered over the surface of the beryllium rod. No conductive overcoat is required. Beryllium was chosen as the support material because it:

1) is a good electrical conductor, 2) takes a high polish, 3) provides excellent back-scatter contrast even for silicate grains, and 4) has no interfering *x*-ray lines.

Grains under about 10 $\mu$  in size often do not absorb the entire electron beam depending upon excitation voltage, composition and thickness. Absolute intensity measurements have been shown to become meaningless for small grains and only intensity ratios are useful. Since the ARL probe is a multichannel instrument it is possible to collect simultaneously intensities for as many as three elements at one time. It has been modified to operate on a preset time base for this work. Figure 1 is a calibration curve for Ca/Si ratios constructed from a series of well known standards. Each point on the curve is the average intensity ratio for just five sepa-

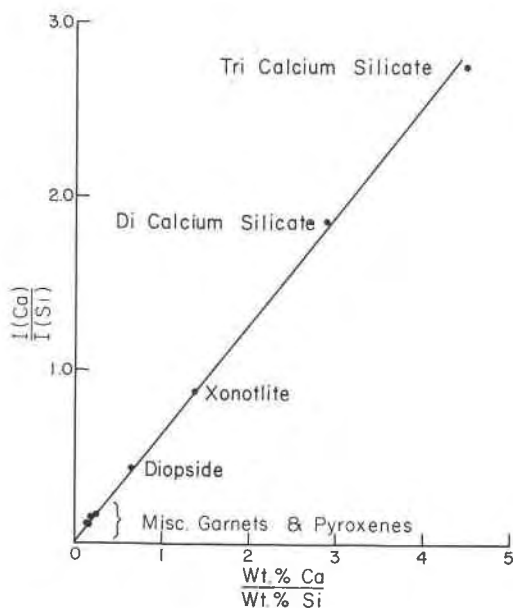


FIG. 1. Calcium/silicon ratio calibration curves for several silicates.

rate grains of each sample. The analysis is good to about  $\pm 2$  weight per cent calcium assuming no error in the silicon reading or in the analysis of the standards. Once the instrument has been set up one can analyze separate phases at the rate of about one phase per twenty minutes.

The technique is being used extensively in this laboratory to analyze samples from clay-zeolite cation partition function experiments, fine powders from phase equilibrium experiments where solid solubility limits are to be established, natural clay mixtures, and phase equilibrium products from systems such as technetium-oxygen.

This work has been supported by the American Petroleum Institute Project 55, under the direction of Professor Rustum Roy.