beam intensity due to electrical power variations, and, second, that since the entire diffraction pattern is recorded simultaneously, both quartz and internal standard lines are affected equally by any instrumental variations.

A relatively new instrument, the Norelco Geiger-counter spectrometer, described by Friedman,[13] shows promise of solving many of the difficulties encountered in the analysis of crystalline dusts. Klug, Alexander and Kummer [14] have presented an internal standard technic using recorded counts of the x-ray quanta entering the Geiger tube as a measure of line intensity and have briefly mentioned a faster, simpler, direct reading method. The use of an internal standard technic with the Geiger counter spectrometer does not appear too sound a procedure, since the intensity of the quartz and internal standard lines are not measured simultaneously. Unless a number of measurements are taken and averaged, a serious error may arise from power supply fluctuations. The direct reading method mentioned by Klug and associates [14] allows fuller realization of the capabilities of the Geiger counter spectrometer, reduces the variables to be controlled to a minimum and increases the speed with which analyses may be performed. It is readily adapted to routine use and does not require the accurate weighings and mixings that are necessary in the internal standard technic. The method has limitations, owing to the possibility that the diffracted rays will be absorbed by compounds in the sample, and to the possibility of line superposition. The superior resolution of the Geiger counter spectrometer minimizes the problem of line superposition, whereas the accuracy with which the background and the line shapes may be determined corrects to a large degree for absorption and scattering. The method described below has been developed as a routine to be used for determining quartz in industrial dusts and other materials.

EQUIPMENT

The Norelco spectrometer consists essentially of an x-ray source, a Geiger tube for reception of diffracted rays, a scaling circuit with mechanical count recorder for measurement of diffracted peak intensities by quantum counting procedures, and a high speed recording strip chart potentiometer for automatic recording of diffraction patterns. Slits for limiting the height and the width of x-ray beams are provided at the exit window of the x-ray tube and the entrance window of the Geiger tube. The specimen post is geared to rotate ½ the angular velocity of the Geiger tube. Synchronous motors are used to drive the Geiger tube and the strip chart of the potentiometer. For a more complete description of the instrument, the reader is referred to the paper by Friedman.[13]

METHOD

The sample is first ground to pass a 200 mesh sieve. Approximately 1 Gm. of this powder is then ground with ethyl alcohol in a power-driven motor and pestle for two hours. Sufficient alcohol is used so that at the end of the two hours a paste is formed, owing to the evaporation of the alcohol. The paste is then dried at 105 C. This procedure gives a powder with an average particle size of 1 to 3 microns, with a maximum of about 5. The particle size of 5 microns or less has been found necessary to prevent preferred orientation of the crystal fragments. Five-tenths gram of this dried paste is then mixed with 0.5 Gm. of Dow-Corning silicone stopcock grease. The resulting mixture is placed in a specially prepared slide. This slide is made of a 1/16 in. (1.5 mm.) thick aluminum sheet, as shown

13. Friedman, H.: Geiger Counter Spectrometer for Industrial Research, Electronics **18**:132, 1945.

14. Klug, H. P.; Alexander, L., and Kummer, E.: X-Ray Diffraction Analysis of Crystalline Dusts, J. Indust. Hyg. & Toxicol. **30**:166, 1948.