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DETERMINATION OF (060) REFLECTIONS OF CLAY MINERALS BY MEANS OF COUNTER TYPE *X*-RAY DIFFRACTION INSTRUMENTS

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The position of the (060) reflection is useful in distinguishing between dioctahedral and trioctahedral clay minerals (1). A simple and rapid method for measuring this reflection with a counter type *x*-ray diffraction instrument has been tested for several standard clay minerals and those from soils. The basis of the method is the orientation of clay particles so as to emphasize scattering along the (0*k*0) reciprocal lattice line. The method orients the clay particles in only one dimension and there is random orientation in the other two dimensions, but the removal of one degree of randomness increases the intensity of the (060) reflection. Orientation is accomplished by sedimenting the clay on a thin aluminum foil, which is then mounted in a plane perpendicular to the *x*-ray beam when the *x*-ray detector is at zero degrees. Thus, diffracted *x*-rays are transmitted through the sample and aluminum foil.

The sample holder, designed for the General Electric XRD-3, consists of a frame made of sheet aluminum, $\frac{1}{8}$ inch thick, having outside dimensions of 1 by $1\frac{1}{2}$ inch and a centrally located "window" $\frac{5}{8}$ by 1 inch. Thin (0.0003 inch) aluminum foil, such as is used in light-proofing *x*-ray cameras, is placed on the frame and folded over the sides of the frame and fastened to the back side with scotch tape. The foil or tape should not cover the back side of the "window." One-half ml. of a suspension containing 15-20 mg. of well dispersed clay is transferred to the aluminum foil over the center of the window. After the suspension has dried, the sample is mounted 90° to the usual position so that the diffracted *x*-rays are transmitted through the aluminum foil to the *x*-ray detector. The sample is scanned between 55 and 65 degrees 2θ if copper radiation is used.

Heat treatment may be employed to distinguish further between some of the clay minerals. For instance, the (060) reflection of kaolinite is at 1.49 A and dioctahedral illite at 1.50 A. In mixtures there may be some doubt as to the mineral causing a reflection in this region. The (060) reflection of kaolinite is removed by heating the clay to 550° C. while that of illite remains. The assembly described may be heated to 550° C. and although the scotch tape is burned, the aluminum foil remains in place. There may be some loosening of the clay flake but this may be secured at the edges by a suitable cement. Thinner flakes are less likely to loosen but there is some decrease of intensity of the reflections by reducing the sample thickness.

The optimum concentration for the clay being studied was determined experimentally but the clay flake thickness probably is not uniform. The sample occupied an area of about 2 cm.^{2} and thus the average concentration of clay was 7.5 to 10 mg. per cm.^{2}. Nickel foil also was tried but this causes excessive background radiation.

Typical results for a soil clay show a peak height of 180 cps (counts per second) at 1.50 Å and a background of 60 cps. This was with copper radiation at 35 KV and 23 ma. The slit system was as follows: 0.2° detector slit, MR soller slit, and a 1° beam slit. Several reflections other than (060) may be obtained. Aluminum itself has few reflections and these do not interfere with most of those obtained with clays. Patterns obtained with "randomly" oriented specimens using the counter method gave much less distinct (060) reflections and it often was necessary to resort to the time-consuming powder camera technique to obtain satisfactory measurements. Many methods currently employed to give "random" orientation of clays may be open to question because of the parallel orientation of clay flakes on packing. This orientation probably often reduces the intensity for the (060) and other reflections. The proposed method takes advantage of the orienting habit of clay particles.

REFERENCE

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LARGE ANGULAR APERTURE AND USEFUL INTERFERENCE FIGURES

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Mineralogists and petrographers are, or should be, aware of the relation between angular aperture of the objective and the usefulness of the interference figure. The angular aperture determines the distance of various features such as melatopes, isogyres, and bisectrices, from the center of the interference figure, according to Mallard's law, $D = K \cdot n \cdot \sin r$, where D is measured distance from the center of the field, K is an instrumental constant depending upon the units of measurement of D and upon the numerical aperture of the objective, n is the appropriate refractive index of the crystal and r is the angle between the the microscope axis and the wave-normal-direction of the light traversing the crystal. If R is the radius of the field of view and A the numerical aperture of the objective, then $R = K \cdot A$, so that $K = R/A$, and Mallard's law becomes $D = (R/A) \cdot n \cdot \sin r$. Evidently increasing the A permits observation of interference effects at larger angles r.