X-RAY DIFFRACTION PROCEDURE FOR POSITIVE DIFFERENTIATION OF MONTMORILLONITE FROM HYDROUS MICA

M. L. JACKSON AND N. N. HELLMAN

IN X-ray diffraction analysis of clays, the (001) spacing or line, being the only one that is decidedly characteristic or different, is frequently used to distinguish minerals of the montmorillonite group from those of the hydrous mica group. This is true especially in the analysis of mineral mixtures in which the relatively strong diffraction lines corresponding to the interplanar spacings (001) are recorded, while other distinctive structures, represented by weaker lines, do not show in the pattern (6, 13, 20, 22). Hence, any conditions which might cause the crystal lattice plates of montmorillonite to come close together so as to result in a shift of the (001) X-ray diffraction line to a position similar to that of a hydrous mica would lead to a mistaken identification of it as hydrous mica. Furthermore, the (001) lines of diffraction patterns of some soil clays have been reported to be very weak or absent (6, 13), leading to uncertainty of identification of the minerals present.

The purpose of the present investigation was to study the influence of character of base saturation and conditions of drying of clay minerals on the intensity and the relative position of the (001) line in the X-ray diffraction pattern and thus to make possible a positive identification and differentiation of the montmorillonite and hydrous mica groups in soil clays and in certain naturally occurring clay deposits.

REVIEW OF LITERATURE

On the basis of X-ray analysis, Alexander, et al. (1), Hendricks and Alexander (13), Kelley, Dore, and Page (20), and Nagelschmidt (22) report the presence of hydrous mica and montmorillonite in soil clays. In the analyses, it was assumed that under the conditions of sample preparation used, a 14 to 16 A. spacing and a 10 A. spacing indicated the presence of montmorillonite and hydrous mica respectively since these spacings were given by pure minerals used as controls. Various investigators have reported that several factors influence the (001) spacing of montmorillonite. Variability of the (001) spacing of the crystal plates of montmorillonite from 9.6 to 19.6 A. with varying water content was demonstrated by Hofmann and co-workers (17, 18, 19). A progressive decrease of this (001) spacing with water loss accompanying increasing temperature was measured by Nagelschmidt (21). Entrances of water into the interplanar spaces in an ordered, stepwise manner, at a time, was indicated by measurements of the (001) spacings at various humidities and water contents by Hendricks, Nelson, and Hendricks (15) attribute the stepwise entrance of water first to the formation of the exchangeable bases, and then to completion of one, two, and three layers of water molecules between the plates.

The specific effects of different exchangeable base hydration properties of a given soil clay were studied by Baver (2) in his hygroscopic coefficient studies. The difference in hydration between Ca-montmorillonite and K-montmorillonite resulted in (001) spacings of 15 and 12 A., respectively, at equilibrium under 50% R. H. temperature in Nagelschmidt’s experiments (22), and hydration properties of the crystal plates. Hendricks, Nelson, and Alexander (8), using large organic bases, demonstrated the size and nature of the exchangeable bases upon the hydration properties of the crystal plates. Hendricks (12) showed that the large organic bases not only entered into the position of exchangeable bases but were sorbed on the surfaces of the crystal plates by van der Waals forces and that this further affected the interplanar spacing. Gruner (11) found that water was lost from between the layers of vermiculite crystals at 100°C without change in the interplanar spacing, while further water lost at 750°C brought the layers as close together as those in talc and mica.

The tendency of clays to dry from water with their plates oriented in parallel and thus to form broad sheets utilized in X-ray work (5, 14, 22); however, the tendency of clays to dry as soft powders when dried from a liquid such as carbon tetrachloride (23) has not been extensively studied by X-ray diffraction. Imperfect arrangement and orientation of the crystal plates of some soil clays and consequent obscurity of their (001) diffraction lines were described by Clark, Riecken, and Reynolds (6), and also suggested by Hendricks and Alexander (13). The condition was ascribed to a process of weathering of the clay plates (6).

From these researches it appears that variability of the (001) montmorillonite spacing is dependent upon following three or more distinct factors, all of...