used for the experiments did not show any perceptible change under the ore microscope.

In conclusion, therefore, it is recommended that extreme caution be exercised in interpreting the results of refractive index measurements made on sulphides and related minerals by the Brewster angle method.

ACKNOWLEDGMENTS

The authors are grateful to S. A. Forman and J. D. Keys of the Mineral Sciences Division of the Mines Branch for helpful suggestions in the inception and development of this work.

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THE AMERICAN MINERALOGIST, VOL. 52, JULY-AUGUST, 1967

A MICRO METHOD FOR DETERMINATION OF CATION EXCHANGE CAPACITY OF CLAYS BY INFRARED SPECTROSCOPY[1]

ALLAN F. BURNS, *Johns-Manville Research Center and Engineering, Manville, New Jersey*, AND JOE L. WHITE, *Department of Agronomy, Purdue University, Lafayette, Indiana.*

Most of the many methods devised for the determination of the cation exchange capacity (c.e.c.) of clays require substantial amounts of material and involve laborious chemical analyses of either the solutions or the clay material itself. For example, a common method for determining c.e.c. involves saturation of a material with NH_4^+, followed by a chemical analysis to determine the amount of NH_4^+ retained on the sample. Presented herewith is a technique which has application in c.e.c. determinations and which obviates or greatly decreases the necessity for chemical analyses and requires a very small amount of material.

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[1] Journal Paper No. 2843, Purdue University Agricultural Experiment Station, Lafayette, Indiana.

While working with an expanding-lattice mineral produced by alteration of muscovite, the authors found that when NH_4^+ was exchanged onto the mineral, the intensity of the NH_4^+ deformation band in the infrared spectra at about 1430 cm^{-1} was quite sensitive to the amount of NH_4^+ present (Burns, 1963). A series of NH_4^+-substituted samples were prepared in which the NH_4^+ contents were carefully determined by a micro-Kjeldahl procedure. One ml of a suspension containing 0.5 mg of clay per ml was pipetted onto an Irtran-2 window having an area of 4.5 cm^2, and air dried; this gave a film having a weight of 0.11 mg/cm^2. J. D. Russell has recently reported a similar technique using larger amounts of clay (1 to 4 mg/cm^2) in the form of unsupported films (Russell, 1965). The film thickness used in our study is too small to form a stable unsupported film. The KBr pellet technique permits the use of very small amounts of material but is unsatisfactory due to possible K-NH_4^+ exchange.

Infrared absorption spectra were obtained using a Perkin-Elmer Model 421 diffraction grating spectrophotometer. A scale expansion of 5× was used for recording the NH_4^+ deformation bands. Variations in the intensity of the deformation band of NH_4^+ due to slight variations in clay film thickness on the Irtran window were corrected by normalizing the absorbance (optical density) of the Si-O stretching frequency (about 1020 cm^{-1}) of the sample and that of a standard containing no NH_4^+. This correction factor was then applied to the absorbance value for the NH_4^+ deformation band of the sample. In the samples studied, the correction factor varied between 1 and 12 percent with an average value of 6 percent. A slight amount of absorption occurred at about 1430 cm^{-1} in the "zero" NH_4^+ sample;[1] the absorbance values were corrected for this and plotted versus NH_4^+ content in Figure 1. A satisfactory linear relation was obtained up to 124 meq/100 g; for NH_4^+ contents in excess of 95 meq/100 g, slight scatter is evident.

The possibility of using this technique for rapid c.e.c. determinations is evident. Once a graph similar to Figure 1 is established by each investigator, samples for analysis need only be saturated with NH_4^+ and the NH_4^+ deformation band intensity measured. The c.e.c. may then be read off the graph. The relative rapidity and ease of the method, coupled with the small samples required, should make it particularly attractive in applications where limited amounts of materials are available, *i.e.* mineral synthesis studies, weathering crusts, "clay skins," etc.

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[1] Expanding 2:1 minerals adsorb NH_3 very readily from the air. It is virtually impossible to prevent adsorption of small amounts of NH_3 by these minerals under ordinary laboratory conditions unless elaborate precautions are taken.

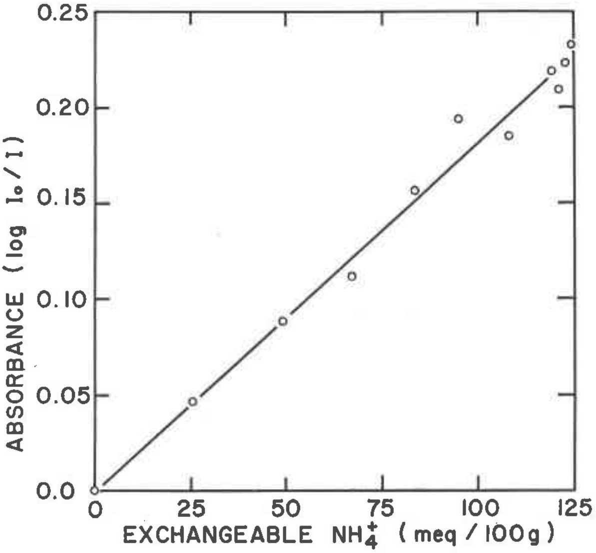

FIG. 1. Relationship between the amount of NH_4^+ exchanged onto altered muscovite and the intensity of the NH_4^+ deformation band (1430 cm^{-1}).

SUMMARY

A technique is herewith presented which has application in c.e.c. determinations and which obviates or greatly decreases the necessity for chemical analyses and requires a very small amount of material. The possibility of using this technique for rapid c.e.c. determinations is evident. The relative rapidity and ease of the method, coupled with the small samples necessary, should make it particularly attractive in applications where limited amounts of materials are available.

ACKNOWLEDGMENT

This research was supported by a grant from the National Science Foundation (NSF G6063) made through the Purdue Research Foundation (PRF 2007).

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