

fragments. Where great effort and time was formerly consumed in getting a platy mineral particle to stand edge-wise by means of broken pieces of cover glass—only to have the index oil float the particle away—the mineral may now be crushed and sprinkled over the tacky mount. A quick examination of the slide will usually reveal the orientation desired.

The gelatin is neutral to all the immersion oils in current use. However, the mount cannot be used with crystals easily soluble in water or with an immersion media having water as a component. The thin coating allows a ready determination of the Becke line and oblique illumination tests. The mount may be used to good advantage with the universal stage.

REFERENCES

- FAIRBAIRN, H. W., 1943, Gelatin coated slides for refractive index immersion mounts: *Am. Mineral.*, **28**, 396–397.
- HERBERT, PAUL, JR., 1942, Petrographic microscope slides of detrital mineral grains: *Jour. Sed. Petrology*, **12**, 90–91.
- MARSHALL, C. E., AND JEFFRIES, C. D., 1945, Mineralogical methods in soil research: The correlation of soil types and parent materials, with supplementary information on weathering processes. C. Gelatin slide method for mounting soil separates for microscopic study: *Soil Science Soc. Amer. Proc.*, **10**, 397–405.
- VEDENEVA, N., AND MELANCHOLIN, N., 1932, The theodolite immersion method, etc.: *Trans. Sci. Invest. Inst. Industry, No. 503. Inst. App. Miner., Paper 54* (Russian and English).

THE AMERICAN MINERALOGIST, VOL. 45, SEPTEMBER–OCTOBER, 1960

DEHYDRATION STUDIES BY INFRARED SPECTROSCOPY

J. M. SERRATOSA, *Instituto de Edafologia, C.S.I.C., Madrid, Spain*.¹

While studying the orientation of OH bond axes in layer silicates (Serratosa & Bradley, 1958) by their absorption in the O-H stretching frequency range ($\approx 3700 \text{ cm.}^{-1}$) some concern was felt because no flexion frequency was certainly correlated with the stretching feature which was utilized. The proper allocation of a band to this expected mode is now arrived at by following the course of dehydration of a montmorillonite from Tidinit, Morocco, and a nontronite from Utah, each previously characterized by x-ray diffraction.

Samples of each were dispersed, and $<1 \mu$ fractions were permitted to evaporate on plastic slides. Dried films were then peeled off for examination.

¹ Experimental work conducted while on leave at the Illinois State Geological Survey, Urbana, Illinois.

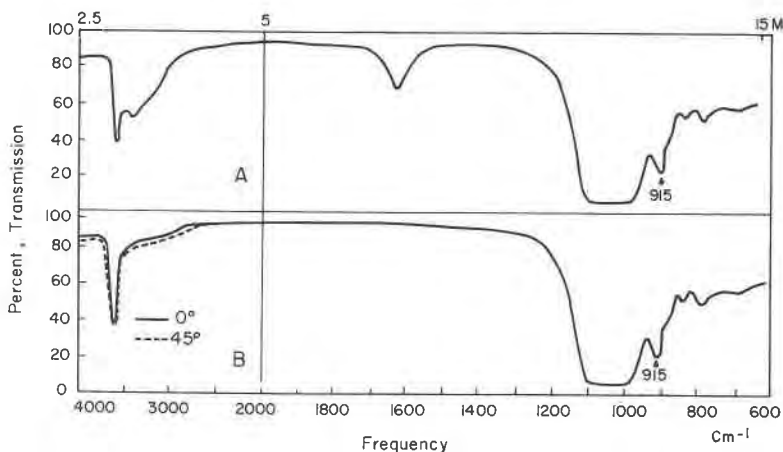


FIG. 1. Infrared spectra of Tidinit montmorillonites: (A) Air dried. (B) Dried at 200° C., and for two incidence angles. The bands from the Nujol have not been included.

Figures 1 and 2 present the absorption spectra of the samples, air dried, and after drying at 200° C. The heated films were covered with Nujol to minimize rehydration from the atmosphere. In each case heating dispelled the water absorption bands at 3400, 3240 and 1640 cm^{-1} , but the structural OH stretching frequency between 3600 and 3700 cm^{-1} persisted and was not markedly influenced by change of the angle of incidence of radiation to the clay film (characteristic of dioctahedral composition).

The greatest lower-frequency difference between the two minerals

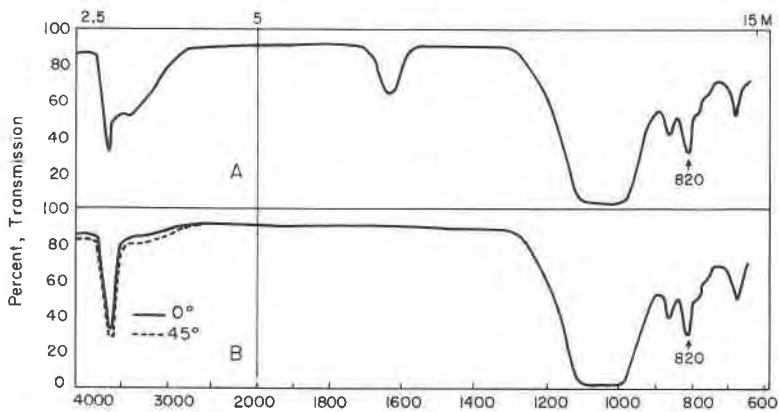


FIG. 2. Infrared spectra of nontronite: (A) Air dried. (B) Dried at 200° C. and for two incidence angles. The bands from the Nujol have not been included.

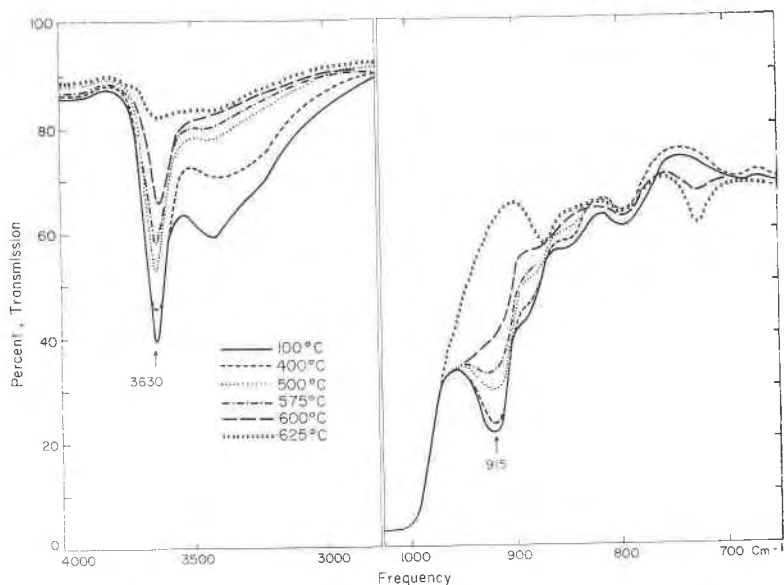


FIG. 3. Infrared spectra of Tidinit montmorillonite heated at increasing temperatures.

was an absorption feature appearing at 915 cm^{-1} for the Tidinit montmorillonite, but at 820 cm^{-1} for the nontronite.

In Figs. 3 and 4 are superposed absorption records following heating at successively increasing temperatures. The 915 cm^{-1} at 820 cm^{-1} bands decrease in intensity at rates concomitant with those of the stretching frequency in each case, even though practical dehydration of the montmorillonite requires heating to nearly 600° C ., but that of the nontronite only to about 425° C .

Although any lowering of the frequency of the stretching vibration by heavier octahedral ion neighbors was within the limits of precision of the frequency measurements in the previous study, the effect of heavier octahedral neighbors on the OH flexion frequency is pronounced.

In these layer structures dioctahedral compositions provide each structural OH with two octahedral ion neighbors, and in the case of the nontronite, it is even possible to speculate that the doubtful feature at about 860 cm^{-1} , which loses intensity less rapidly than the 820 cm^{-1} feature, represents hydroxyls which have one Fe and one Al or Mg neighbor.

This same allocation of bands at 910 and 938 cm^{-1} in kaolinite and at 820 cm^{-1} in nontronite to $\text{H-O} \cdots \text{Al}^{+3}$ and $\text{H-O} \cdots \text{Fe}^{+3}$ respectively has now been authenticated by Stubican and Roy (1960) on the basis

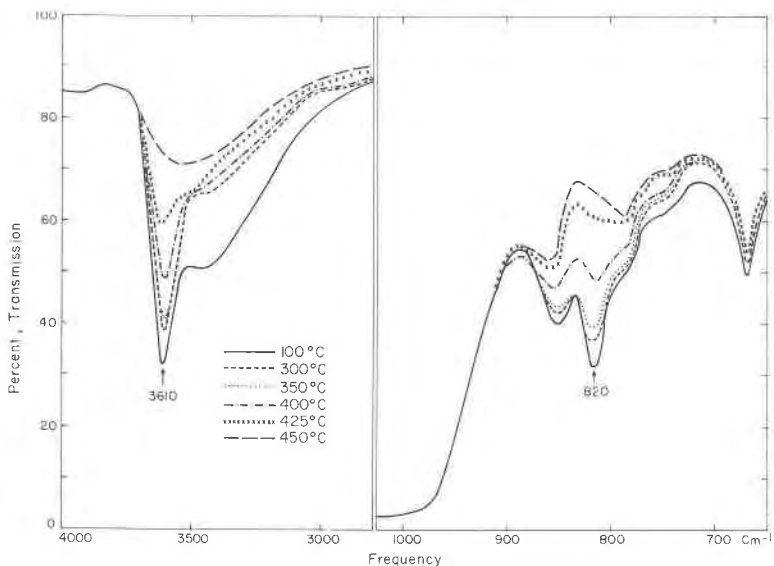


FIG. 4. Infrared spectra of nontronite heated at increasing temperatures.

of comparisons of spectra for these minerals with those of their deuterated synthetic analogues.

REFERENCES

- SERRATOSA, J. M. AND BRADLEY, W. F. (1958). Determination of the orientation of OH bond axes in layer silicates by infrared absorption. *J. Phys. Chem.* **62**, 1164-67.
- STUBICAN, V. AND ROY, R. (1960). Influence of controlled ionic substitution on the infrared absorption spectra of synthetic clay minerals. Abstracts of Cleveland Meeting of the American Chemical Society, April 1960.

THE AMERICAN MINERALOGIST, VOL. 45, SEPTEMBER-OCTOBER, 1960

X-RAY DIFFRACTION TECHNIQUE FOR SMALL SAMPLES

RONALD K. SOREM, *Department of Geology, Washington State University, Pullman, Washington.*

In work with the x-ray powder camera, little difficulty is found in preparing suitable powder mounts from samples weighing several milligrams, but when samples of a much smaller size are used, suitable mounts are not easy to prepare using ordinary techniques. Consider, for example, a few particles perhaps less than 10 microns in size scratched from a mineral grain. A sample of this kind may be required if a pure