Low Temperature Analysis of Kaolin Clay Using Diffuse Reflectance

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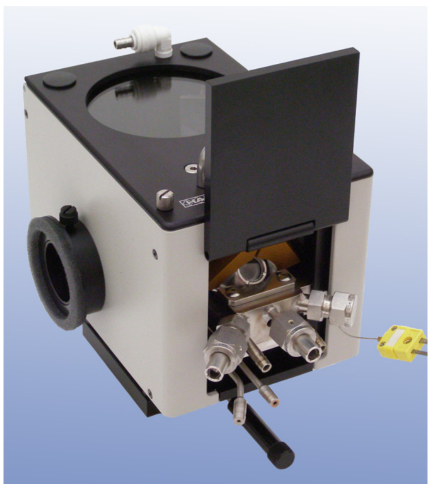

Figure 1. Praying Mantis™ with its High Temperature Reaction Chamber and Cooling Cartridge installed.

INTRODUCTION

Clays, like kaolinite and smectite, are hygroscopic and it is well known that the adsorbed water can be driven off at elevated temperatures. Changes in the O-H bond vibrational modes have been observed below 200K[1].

This note explores changes in water bands of kaolin at temperatures down to -23°C.

EXPERIMENTAL

The spectra were taken using a Praying Mantis™ diffuse reflectance accessory in a commercial FTIR spectrometer with a DTGS detector. The sample analyzed was kaolin clay supplied by Ward's Natural Science. The sample was cooled in a Praying Mantis™ High Temperature Reaction Chamber equipped with its cooling cartridge (Figure 1) using a Julabo F25-ED refrigerating-heating circulator and insulated tubing. The circulator was connected to the cooling cartridge using two 0.5 m

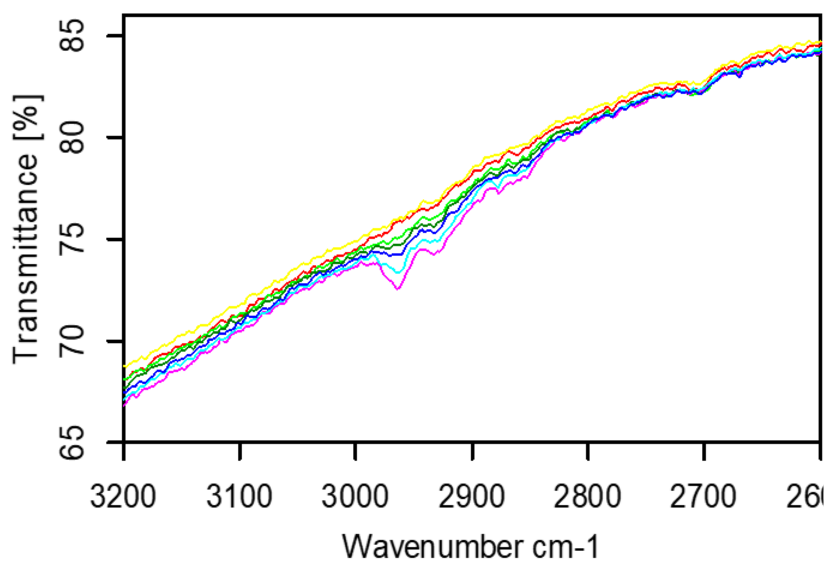

Figure 2. Spectra of Kaolin at temperatures ranging from 23°C (red) to -23°C (magenta).

lengths of 8-mm ID viton insulated tubing. The sample was chilled in 2.5°C increments, from 23°C down to -23°C. Each time the temperature was lowered, the sample was allowed to stabilize. Eight minutes after achieving equilibrium, the infrared spectra were collected over range of 4000 cm^{-1} to 600 cm^{-1}. Spectra were signal averaged over 32 scans at an 8 cm^{-1} resolution.

Temperature measurements were recorded using the thermocouple imbedded in the sample cup and directly from the circulator to obtain a measure of the heat losses between the reaction chamber Wand circulator.

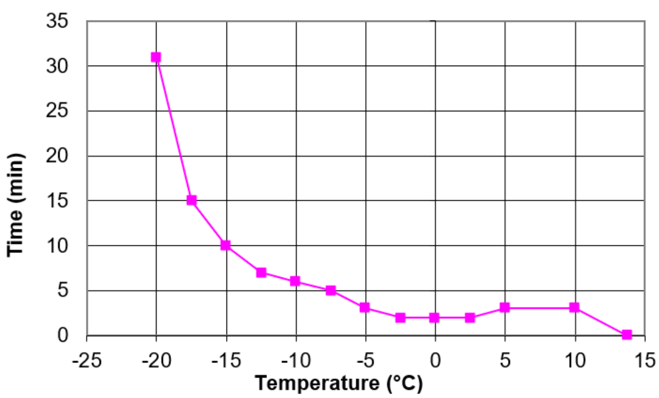

Figure 3. Rate of cooling.

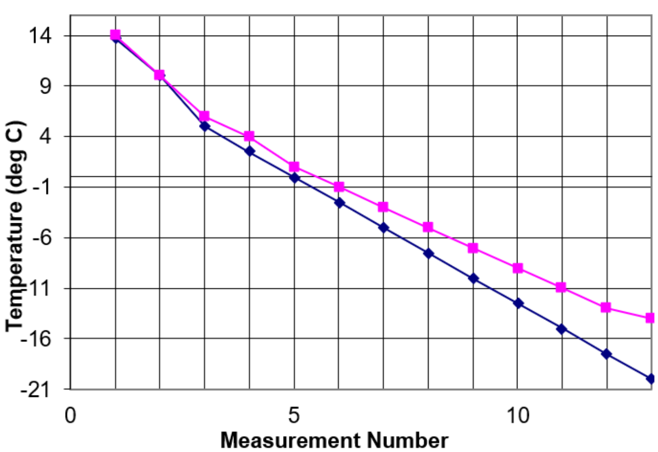

Figure 4. Deviation of the sample cup temperature (squares) from the circulator temperature (diamonds).

RESULTS AND DISCUSSION

Figure 2 shows the spectrum of kaolin in the C-H stretching region, recorded at several different temperatures. Cooling results in the emergence of two peaks at 2960 and 2850 cm^{-1}, in addition to a peak shift in the band at 3434 cm^{-1}. Band shifts and the emergence of bands are typically associated with changes in temperature. In this case, these peaks are likely due to unknown organic impurities.

The temperatures reported in Figure 3 were measured at the circulator. Figure 3 shows the discrepancy in temperature between the circulator and the sample cup. As expected the differential between the two increases at lower temperatures. Note that, as temperature decreased, the circulator also took increasingly longer to drop an additional 2.5°C, as shown in Figure 4. Note that low temperatures, down to -95°C, could be achieved by this method using an appropriate chiller and coolant.

CONCLUSION

The low temperatures achieved were sufficient to observe minor changes in the water bands which enhanced the spectra of the impurities in the kaolin but were not sufficient to induce previously reported O-H vibrational changes. In short, the Praying Mantis with its cooling cartridge and an appropriate chiller can be used effectively for moderately low temperature experiments.

REFERENCES

1. R. Prost, A. Dameme, E. Huard, J. Driard, and J. P. Leydecker, *Clay and Clay Minerals*, 37 (5), 464-468, (1989).
2. Etienne Balan, Simon DeLattre, Maxime Guillaumet, and Ekhard Salje, *American Mineralogist*, 95, 1257-1266, (2010).