

Figure 1. The Harrick Emission Accessory with the Harrick Heated Vacuum Chamber (HVC) being used as the sample holder.

Analysis of Water Content of Clay Using Infrared Emission Spectroscopy

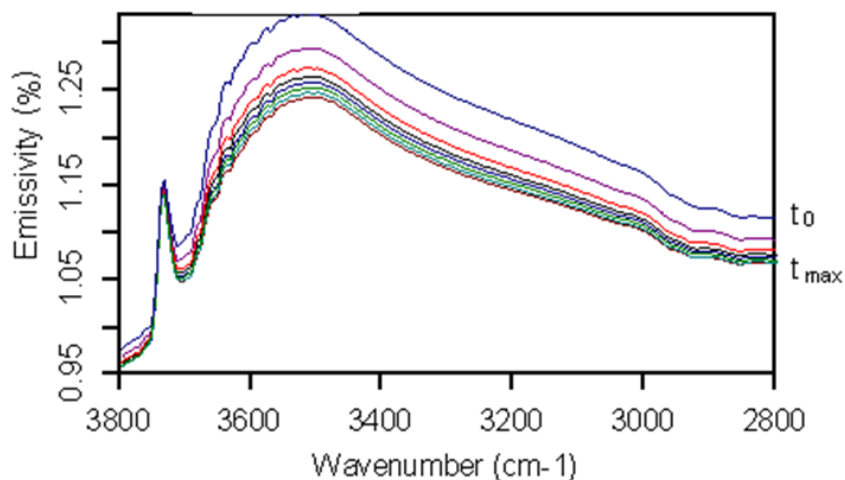
INTRODUCTION

Infrared emission spectroscopy is a powerful, if underutilized spectroscopic technique. One advantage of infrared emission is the ability to study *in situ* the effects of heating samples. The Harrick Emission Accessory with the Heated Vacuum Chamber (HVC) (see Figure 1) attaches to the emission port of the IR spectrometer, making the sample the IR source, and can heat the sample to temperatures up to 910 °C using the Harrick Temperature Controller (ATC). A potential application of IR emission is the observation of loss of water from a sample at high temperature¹. This applications note demonstrates the use of IR emission in the evaporation of water from kaolinite (clay) at 700 °C, based on the work of Frost and Vassallo¹.

EXPERIMENTAL

Emission spectra were collected on an FTIR spectrometer with an emission port with the Harrick emission accessory as the source. An MCT detector cooled to 77 K with liquid nitrogen was used. All spectra were a result of 64 averaged scans at a resolution of 4 cm⁻¹. The gain

Figure 2. Spectra of kaolinite taken at 700 °C in 10-minute intervals.



ANALYSIS OF WATER CONTENT OF CLAY USING INFRARED EMISSION SPECTROSCOPY NO. 21135

was set to 8, and the aperture was set to 100 (fully open), and spectra were collected in the range 4000-650 cm^{-1} . Kaolinite was obtained from Ward's Natural Science (Rochester, NY; Reference # 46E4330). A thin layer of clay was spread using a spatula across a 316 stainless steel pedestal that fit snugly into the HVC sample cup. The chamber was enclosed by its flat top window assembly, without a window installed, to stabilize the temperature. The sample was heated to 700 °C using the ATC. Single-beam spectra were taken every 10 minutes and ratioed (1:1) to a single-beam spectrum of a pinhole at 700 °C, intended to approximate a blackbody.

RESULTS AND DISCUSSION

The infrared emission spectra of clay over the range 3800-2800 cm^{-1} , measured over 1.5 hours, are shown in Figure 1. The broad peak centered around 3500 cm^{-1} is a result of -OH bands from residual water. The band is even broader than usually seen in IR spectra, and broader than in the work of Frost and Vassallo, most likely because their samples were dried under desiccation, while the samples used here were not. Thus both interstitial and adsorbed water is observed, broadening the band significantly. The intensity of the band at each subsequent time interval decreased, as water evaporated from the heated sample.

CONCLUSIONS

Infrared emission spectroscopy has a number of useful applications, one of which is observing the change in the amount of water contained in a solid sample such as clay. The -OH band in the spectra of clay at 700 °C clearly decreased in intensity as the sample remained at temperature for increasing amounts of time. The nature of IR emission and the Harrick emission accessory enabled the measurements to be made *in situ*, without needing to heat and then cool the sample before subjecting it to spectroscopic measurements. A more quantitative treatment could potentially enable the determination of the specific amount of water within a sample.

REFERENCES

1. Frost, R.L.; Vassallo, A.M. *Clay and Clay Minerals* 44 (1995) 635-651.