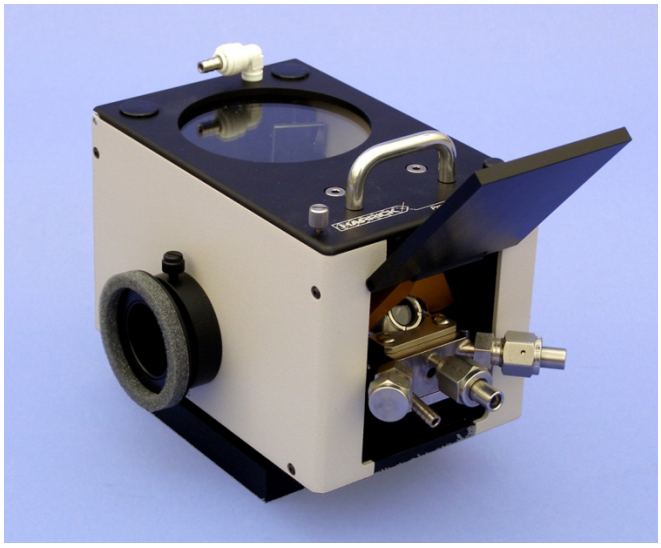

Figure 1. Praying Mantis™ shown with its High Temperature Reaction Chamber.

Figure 2. Temperature Controller.

UV-Vis Diffuse Reflectance Study of the Dehydration of Hexahydrate Nickel Sulfate

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INTRODUCTION

When certain inorganic salts crystallize, they retain some water molecules. These hydrous salts can undergo color changes when the bound water is driven off.

This note uses UV-Vis diffuse reflectance spectroscopy to explore the color change of powdered hexahydrate nickel sulfate, $NiSO_4(H_2O)_6$, upon heating.

EXPERIMENTAL

The measurements were taken using a Praying Mantis™ diffuse reflectance accessory with its High Temperature Reaction Chamber (see Figure 1). The heater and K-type thermocouple from the reaction chamber were connected to Harrick's Temperature Controller (see Figure 2). The controller was interfaced to a computer by an RS-485 to USB adapter and was controlled by the Watlow EZ-Configurator software. The temperature controller was auto-tuned to 600°C prior to use.

The diffuse reflectance accessory was installed in a commercial UV-Vis spectro-photometer. The data interval was set to 0.1 nm, the aperture was reduced,

and the spectra were measured over the range of 650 nm to 200 nm.

The sample was initially heated in 5° increments starting from ambient. To make sure that the sample had reached equilibrium at each temperature, one spectrum was taken right after the desired temperature was attained, and every 4 min. thereafter until no difference was observed from the previous spectrum. Up to four spectra were required in some cases. After the sample cup achieved 70°C, the powder was heated in 10° increments and, after 120°C, increments of 20° were used. The reflectance data were then transformed using the spectrometer's Kubelka-Munk function was plotted.

RESULTS AND DISCUSSION

Hydrous salts such as $NiSO_4$ are bright vivid colors in an air environment. This is due to the availability of water vapor in the air which allows nickel sulfate to form a coordination compound with water, $NiSO_4(H_2O)_6$. This complex is teal in appearance at room temperature. However as the temperature of the powder increases, the water evaporates and the complex slowly loses its color. Pure anhydrous NiSO4 is yellow in color, but during the heating process the color is extremely pale if at all visible to the naked eye.

This change is evident in UV-Vis spectra shown in Figure 3, where the sample becomes increasingly yellow as it is heated (Figure 3). This is corroborated by the peak maximum shifting from 400 nm, which corresponds roughly to blue, towards higher wavelengths. Note that the peak never reaches yellow (570 nm).

CONCLUSION

The combination of the Praying Mantis™ diffuse reflectance instrument and the high temperature reaction chamber is ideal for detecting changes in powders with temperature. Even the change caused by a temperature difference of 10° can be detected using this attachment.

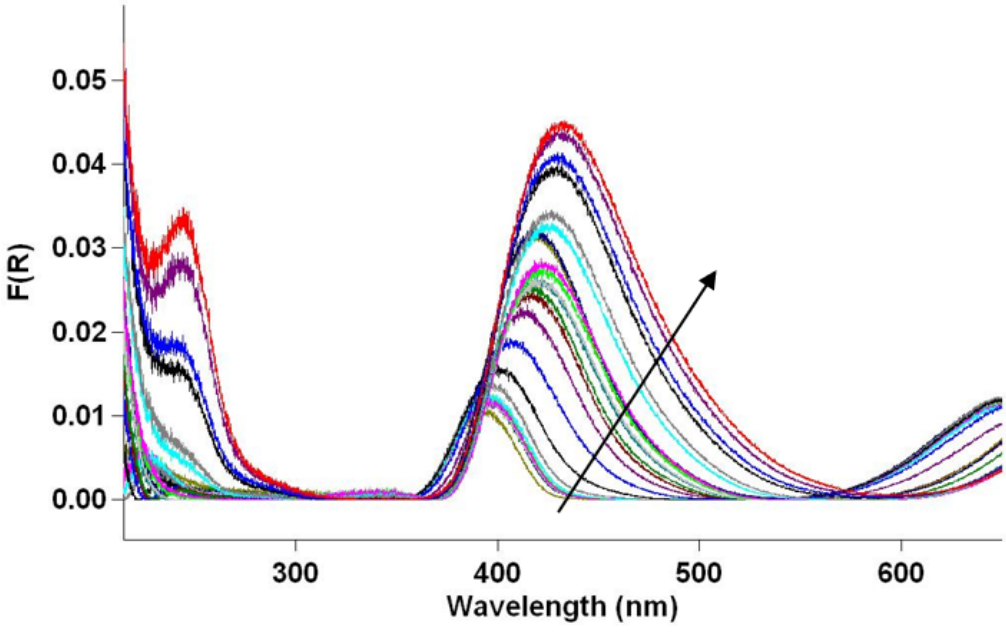

Figure 3. Diffuse Reflectance Spectrum of $NiSO_4$ at temperature from 20°C to 180°C, with the arrows showing increased temperature trends.

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