UV-VIS Diffuse Reflection Spectroscopy of Thermochromic Materials

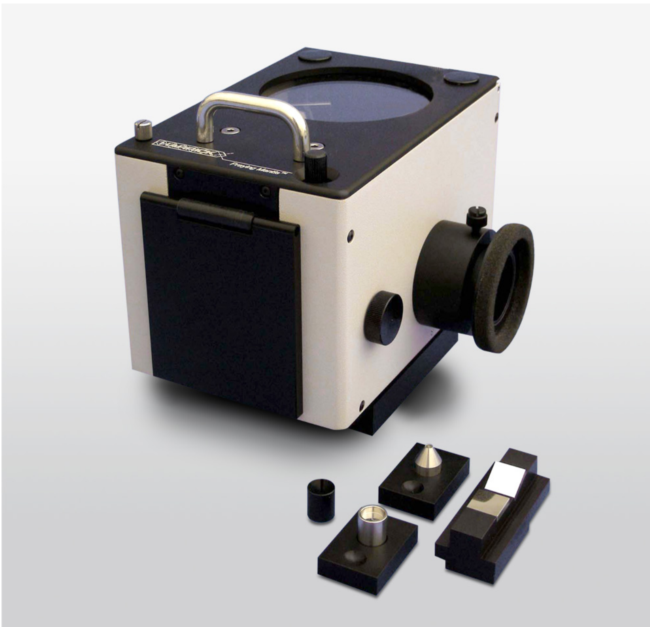

Figure 1. The Praying Mantis™ Diffuse Reflectance Accessory.

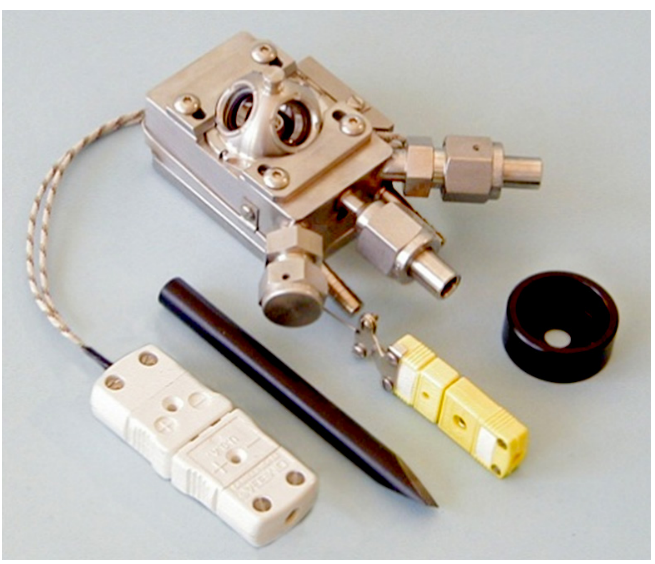

Figure 2. High Temperature Reaction Chamber for the Praying Mantis™.

INTRODUCTION

Some materials undergo electronic transitions upon heating. These transitions can readily be studied by UV-VIS spectroscopy. Most samples examined in this manner are liquids. Such liquids are simply poured into commercially available thermostated cuvettes that the liquid is simply poured for analysis by transmission spectroscopy. Analysis of powders and other solids is less straightforward. Many of these samples are simply dissolved in a liquid for easy analysis.

For samples that are not readily soluble or must be analyzed neat, the only feasible UV-VIS analytical methods are specular reflectance and diffuse reflectance. The former is suitable for highly reflective materials; the latter for powders and roughened solids. Both require a mirror assembly to direct the radiation to and from the sample, in addition to a thermostated sampling stage.

This application note explores the use of diffuse reflectance to probe the color changes of a rough-surfaced solid at various temperatures.

EXPERIMENTAL

The sample investigated herein was a Thermal Liquid Crystal Paint (Edmund Scientific, 3053489) with color changes in the 40-45°C temperature range. The paint was applied to one end of a sandblasted 316 stainless steel disk which fit into the sample cup of the reaction chamber. The paint was allowed to dry thoroughly prior to measure-ment. An unpainted disk was used as the reference.

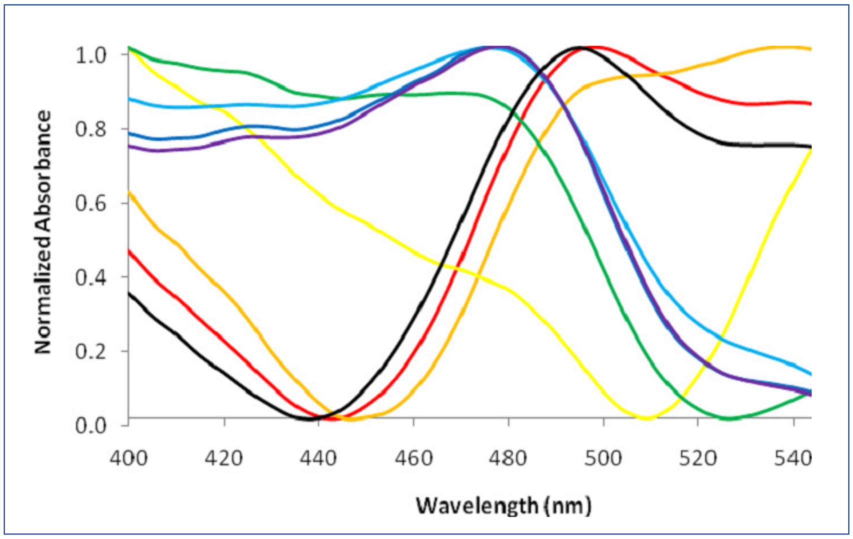

Figure 3. Diffuse Reflectance Spectra of Thermal Liquid Crystal Paint Measured at 26°C (red), 39°C (orange), 41°C (yellow), 42°C (green), 43°C (light blue), 44°C (dark blue), 45°C (purple) and 46°C (black).

The diffuse reflectance measurements were carried out using a commercial UV-VIS spectrometer with the Harrick Praying Mantis™ diffuse reflection accessory (Figure 1) along with its High Temperature Reaction Chamber (Figure 2) installed. Harrick's Temperature Controller was connected to the K-type thermocouple and the heater of the reaction chamber to regulate and monitor the temperature of the cell.

Prior to data collection, the reference disk was installed in the reaction chamber and the temperature controller was initially auto-tuned for operation at 40°C. The sample stage was then allowed to cool to ambient.

After the temperature stabilized, a reference spectrum was collected. This reference was used for all subsequent measurements.

The reference was then exchanged for the painted disk. Once the reference was in place, the temperature controller set point was selected to be 26°C. The controller was turned on and allowed to reach equilibrium at that temperature and then the sample spectrum was collected. Measurements were taken in this fashion at 39°C, 41°C, 42°C, 43°C, 44°C, 45°C and 46°C.

All the diffuse reflectance spectra were measured with a UV-VIS spectrometer in its double-beam mode with an open slit, a 2-nm SBW and a 5-nm data interval.

RESULTS AND DISCUSSION

Figure 3 shows the spectra of the sample recorded at eight different temperatures. At the lowest temperature, 26°C, the spectrum has a peak at 500nm, in the green region of the visible. As the sample is heated, the peak at 500nm decreases in intensity and another peak arises at 480nm, in the blue. Thus this particular paint changes color in the blue-green region of the visible spectrum as a function of temperature between 26° and 45°. Beyond the working range of the paint, at 46°C, the paint reverts to something akin to its original green wavelength.

CONCLUSION

From the above, it is clear that diffuse reflectance, using the Praying Mantis with its high temperature reaction chamber, is an effective way of measuring temperature-induced wavelength changes, *i.e.* thermochromism, and observing the underlying electronic transitions. The diffuse reflectance sampling technique, combined with a temperature-controlled chamber, is an effective analytical tool for studying temperature-induced changes in solid materials such as powders and rough-surfaced solids. Additional applications include analyzing gas-solid interactions including hetero-geneous catalysis.

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