INTRODUCTION

Chemical reactions and concentrations fluctuations over time can involve a color change. Such color variations can be readily detected by UV-Vis spectroscopy. These types of experiments are usually carried out using fixed-pathlength cuvettes. However, Demountable Liquid Cells, with their adjustable pathlengths, flow, and thermal control options are also well suited for this type of application.

This note explores the use of a Demountable Liquid Cell (Figure 1) to analyze the changes that occur as hydrochloric acid is added to a saturated aqueous CuSO₄ solution.

EXPERIMENTAL

The measurements were carried out using a PTFE Demountable Liquid Cell with Luer fittings, a pathlength of 950 nm, and 13mm diameter SiO₂ windows (Figure 1). This cell was used in conjunction with a commercial UV-Vis spectrometer. Spectra were collected from 200 nm to 1500 nm. Due to the small clear aperture of the cell, the spectrometer slit was set to reduced for these measurements.

A 50 mL saturated solution of CuSO₄ was prepared at room temperature. 12 M HCl was added to the solution in 0.2 mL increments until a total of 4 mL were added, at which point the acid was added 0.5 mL at a time. The samples were introduced into the cell using a Luer-Lok syringe which was rinsed thoroughly between samples.

RESULTS AND DISCUSSION

Color Change

When hydrochloric acid is added to the solution, the
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following substitution reaction occurs:

\[ [\text{Cu(H}_2\text{O)}_6]^{2+} + 4 \text{Cl}^- \rightarrow [\text{CuCl}_4]^{2-} + 6 \text{H}_2\text{O} \]

The resulting complex ion, tetrachlorocuprate II, appears yellow in its pure aqueous form, strongly absorbing wavelengths in the 900 nm region. Copper sulfate in solution takes on a blue color due to the formation of the coordination compound \([\text{Cu(H}_2\text{O)}_6]^{2+}\). This complex ion is highly absorbing in the red visible light region, making it appear blue to the human eye. The most weakly absorbing spectrum (shown in red) in Figure 2 illustrates the strong absorbance of the initial solution in the red portion of the visible spectral region around 800 nm. When not completely reacted with blue copper sulfate, the resulting mixture appears increasingly green (Figure 3 right). The spectra displayed in Figure 2 show the shift in peak maxima from 800 nm to 860 nm as hydrochloric acid is added to the solution. This is reflective of the accompanying color change from blue to light green. This shift occurs over the course of the 11 mL of 12 M HCl added. Although this color change is not noticeable to the naked eye until around 6.5 mL of HCl have been added, the spectrometer detects the reaction as gradually as it takes place.

**Peak Intensity Change**

Although the initial solution is chemically saturated with CuSO₄, the copper hydride species formed is not as strong of an absorber as \([\text{CuCl}_4]^{2-}\). As a result, the gradual formation of this species results in increased absorbance as well as the peak shift described previously. Due to the high absorption index of tetrachlorocuprate II, very little light reaches the detector near 900 nm. This makes the instrument noise more prominent in that region as more hydrochloric acid is added.

**Conclusion**

Demountable liquid cells coupled with UV-Vis spectroscopy can be used effectively to observe chemical changes in liquids such as aqueous copper sulfate. These cells offer two viable alternatives to the traditional cuvette. They are also available in a flow-through model to detect change nearly continuously.

**References**