TEMPERATURE CONTROLLED GAS CELL **NO. 21167**

Temperature Controlled Gas Cell and Its Application

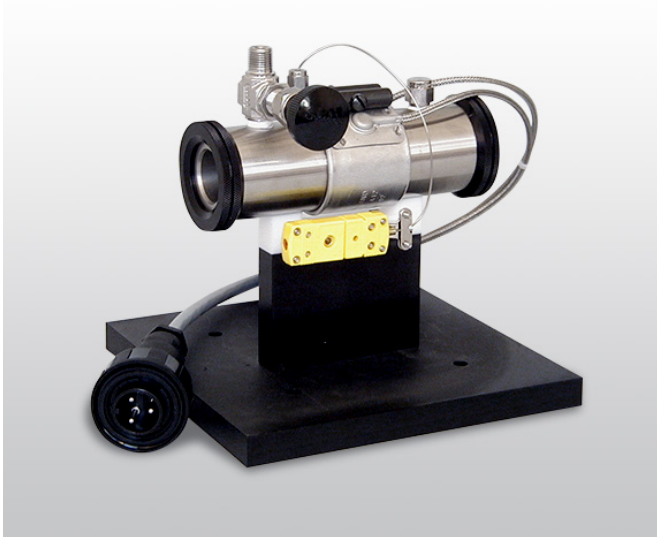

Figure 1. Temperature Controlled Gas Cell.

INTRODUCTION

When studying the vibrational spectroscopy of organic molecules in the condensed phase, the functional groups interaction with other species and neighboring molecules (solvent) can result in significant broadening of the rotational transitions resulting in a featureless spectrum. However, in the gas-phase, the molecules will be able to rotate and vibrate freely. Depending on the resolution of the spectrometer used, they will produce prominent peaks of the rotational lines for the various isotopic species present.

EXPERIMENTAL

Synthesis of HCl (DCl) from acetyl chloride and H_2O (D_2O) was carried out after capturing an acetyl chloride background using a vacuum system operating at 50 torr with a reaction flask attached to the Harrick temperature-controlled gas cell (see Figure 1). Acetyl chloride is introduced to the reaction vessel via septum injection and is allowed to undergo phase transition into its vaporous state, filling both the reaction vessel and the Harrick gas cell. The gas cell can then be isolated from the system and placed into a commercial FTIR (2.0 cm^{-1} resolution, 32 scan average) to obtain a background. The cell can then be reintroduced into the vacuum system and the synthesis of HCl (DCl) is initiated. H_2O (D_2O) is injected into the reaction vessel and allowed to react with the acetyl chloride producing HCl vapor. The HCl/acetyl chloride vapor will fill the gas cell and can then be isolated and removed from the system for analysis using the FTIR spectrometer.

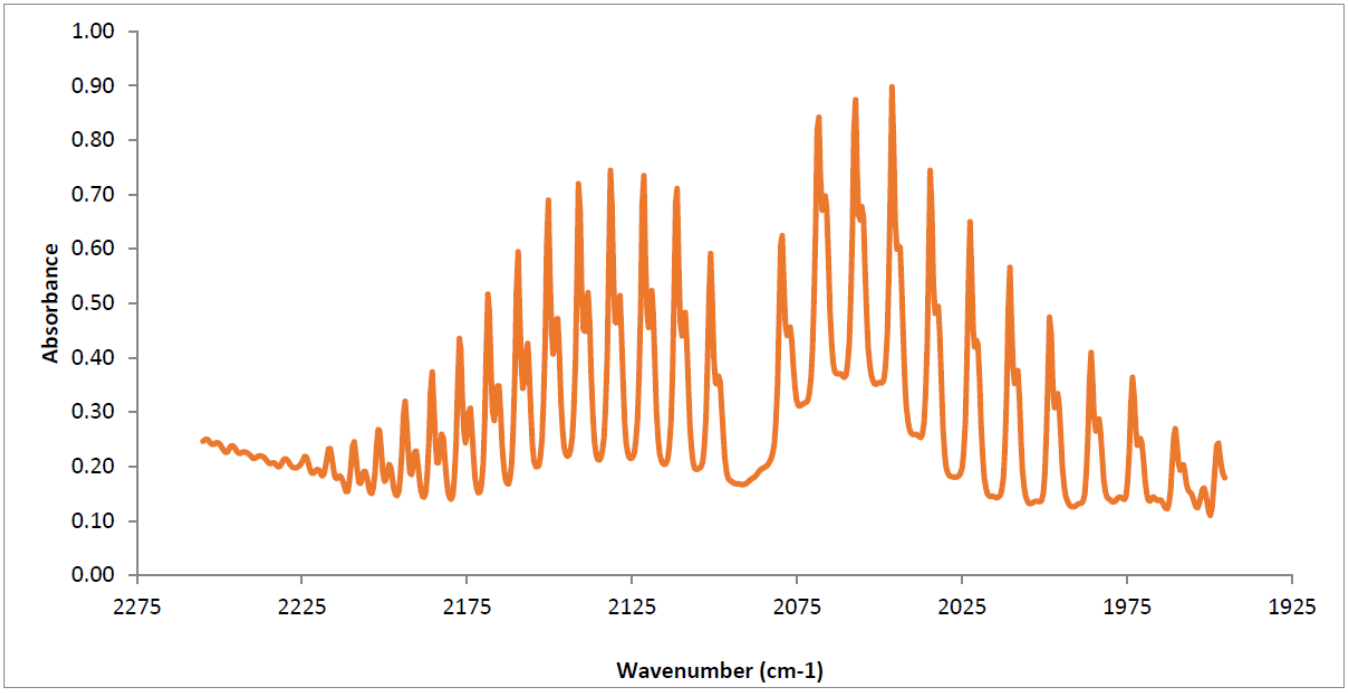

Figure 2. Transmission Spectrum of DCL.

RESULTS AND DISCUSSION

The IR spectra generated from the reaction of Acetyl chloride and D_2O using the Harrick temperature-controlled gas cell shown in Figure 2 can be identified as the vibrational spectrum for the transition between the ground state and the first excited state showing all of the rotational peaks in the two allowed branches (P and R).

CONCLUSION

A crucial advantage of using the Harrick temperature-controlled gas cell is the clean contained environment that it provides. After each synthesis, the Harrick cell can be connected to the vacuum system and pumped out while heating the cell using an 80°C liquid circulator to remove all traces of acetyl chloride from the cell. Failure to do this leads to significant degradation of a prior cell over the course of a few weeks.

When using reactants that produce non-ecofriendly product exposure limits can be elevated. The gas cell provides a contained environment for the reaction to take place and also allows transport to the IR instrument for analysis limiting exposure to the technician or student. The Harrick gas cell provides an alternative for chemical work up for IR analysis and final product identification.

REFERENCES

Atkinson, Dean, revised 1/17/2020. Physical Chemistry Lab: RoVibrational Spectra of HCl and DCl. {adapted from "Vibrational-Rotational Spectra of HCl and DCl" in Experiments in Physical Chemistry by C. W. Garland, J. W. Nibler, and D.P. Shoemaker 8th Ed. McGraw-Hill, (2009) ISBN 978-0-07-282842-9.}