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A Simplified Technique for the Collection of an HCl/DCl Gas Mixture

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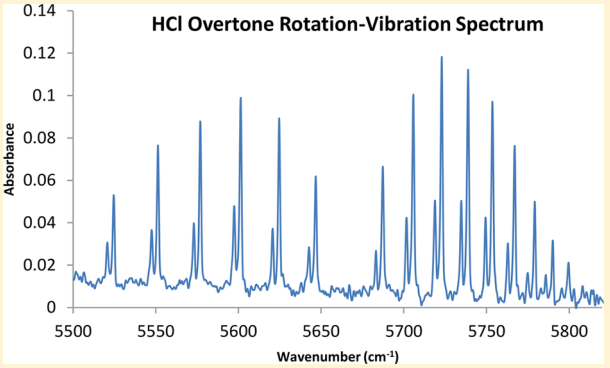
Supporting Information

ABSTRACT: A simple and safe procedure is proposed which allows for the collection of HCl and DCl gas produced via slow heating of an aqueous mixture of each component.



KEYWORDS: *Physical Chemistry, Upper-Division Undergraduate, Gases, IR Spectroscopy, Laboratory Instruction*

The collection of HCl/DCl gas, and the subsequent analysis via rotational−vibrational spectroscopy, is a nearly ubiquitous component of physical chemistry laboratory curricula. Many approaches have been proposed which allow students to carry out the procedure in a safe and controlled manner.[1−11] However, significant safety and logistical concerns associated with such processes still exist. In this letter, we present an alternative method that allows for the simultaneous collection of HCl and DCl gas via simple heating of an aqueous mixture containing each component. This method avoids the use of concentrated H_2SO_4 (a highly caustic chemical), avoids reactions involving acyl halides (suspected carcinogens), avoids the use of lecture bottles (which are expensive to dispose of), does not require liquid nitrogen, and is carried out using common glassware found in nearly all undergraduate teaching institutions.

The experimental procedure calls for the simple apparatus shown in Figure 1. Aqueous HCl (36.5−38.0%, J. T. Baker) and DCl (35% wt in D_2O, Sigma-Aldrich), in 5 mL aliquots, are placed in a 50 mL round-bottom flask, which is then attached to a 10 cm air condenser that has been filled with glass wool and a drying agent (such as calcium chloride). These items are suspended in a hot water bath maintained at 80 °C. By use of Tygon tubing, the air condenser is attached to a typical infrared gas cell which was previously purged with gaseous nitrogen. More Tygon tubing allows the HCl/DCl vapor to travel to a bromocresol green indicator solution in order to determine the point at which the gas cell is effectively filled with HCl/DCl gas. This procedure should be performed in a fume hood and takes about 20 min to complete (see the Supporting Information). The contents of the gas cell are subsequently used to produce a rotational−vibrational spectrum containing peaks for each component of the mixture,

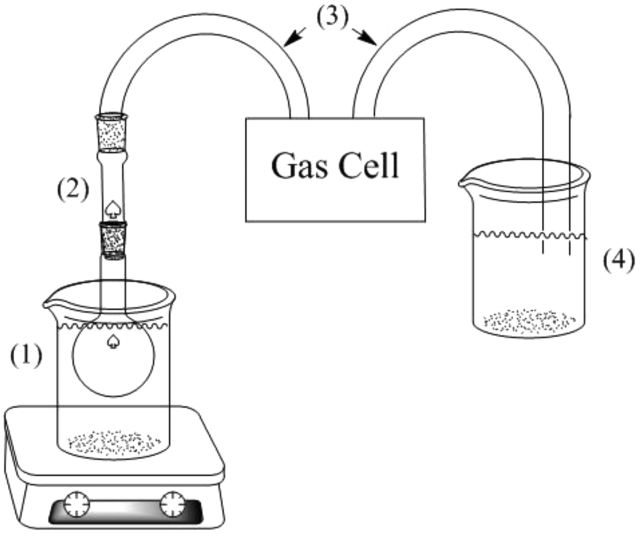


Figure 1. Experimental apparatus. (1) Water bath heating a round-bottom flask containing a mixture of HCl (aq) and DCl (aq). (2) Condenser containing a drying agent. (3) Tygon tubing leading to and from the gas cell. (4) Beaker containing indicator solution.

allowing for data analysis to determine spectroscopic constants and experimental bond lengths.[10−15]

Example spectra acquired via the simple heating technique can be found in Figure 2. The spectra were acquired using a Jasco 460Plus FTIR set at 1 cm^{-1} resolution, and the gaseous mixture was contained in a Pike Technologies gas cell fit with

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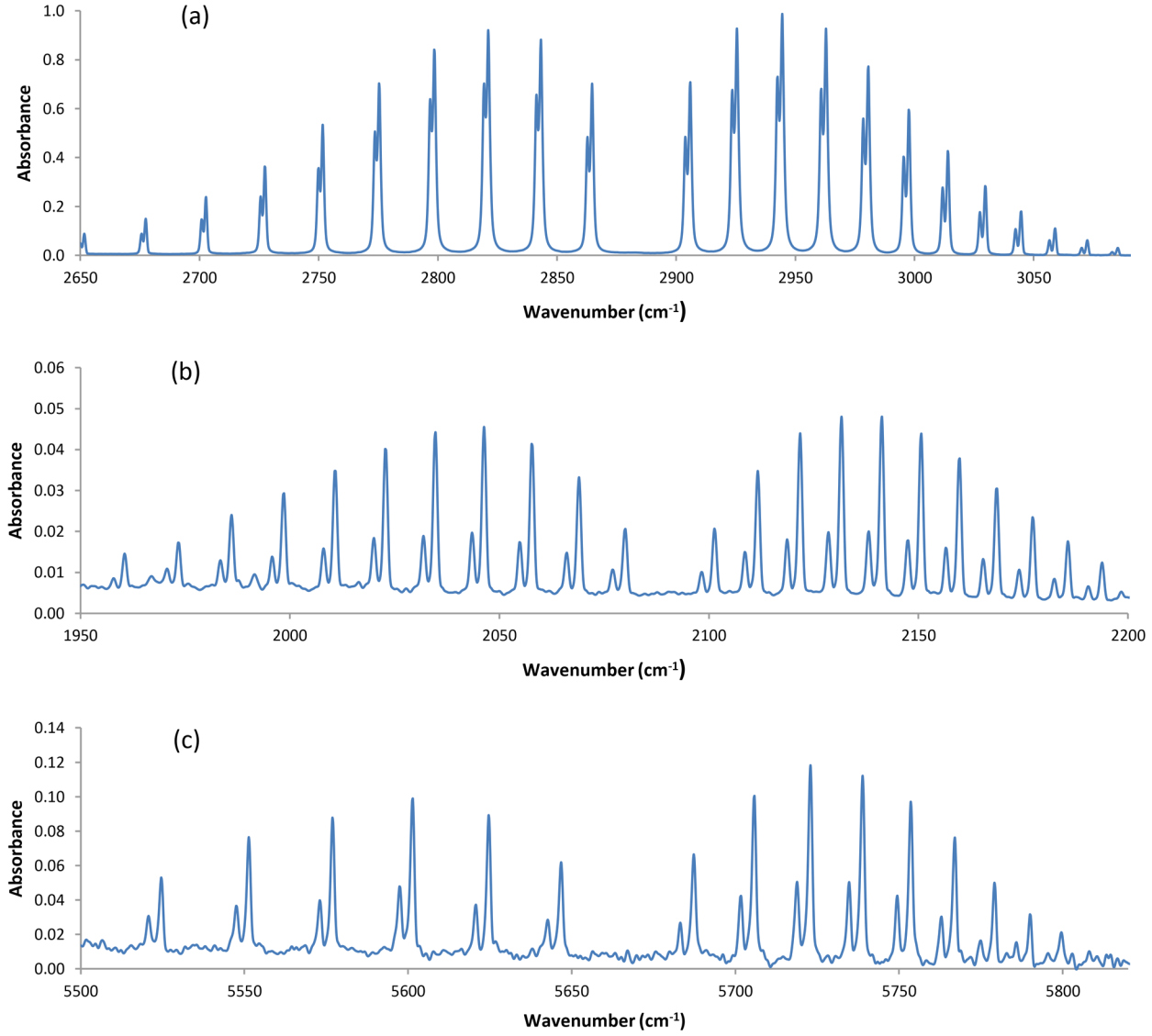

Figure 2. High resolution spectra acquired via the simple heating method for (a) the fundamental rotation−vibration transitions for gaseous HCl, (b) the fundamental rotation−vibration transitions for gaseous DCl, and (c) the rotation−vibration transition for the first overtone (0 → 2) transition of gaseous HCl.

inert sapphire windows. The $^{37}Cl-^{35}Cl$ isotope effect is evident for both HCl and DCl, and the rovibrational bands of the HCl overtone transition are also shown. Acquiring such information adds to the possibilities of the subsequent analysis students can undertake.

Our institution had run this lab for years as a "dry-lab" (where spectra were simply provided to the students) due to the many aforementioned difficulties arising from the collection of the necessary gases. Adopting this simple procedure has greatly enhanced the student experiences associated with the lab. By performing the gas collection steps, students gain a better understanding of the complexities associated with working with gaseous systems and they are exposed to use of important instrumentation (the FTIR) and have greater ownership of the data they analyze due to their hands-on experience of acquiring it themselves.

HAZARDS AND SAFETY PRECAUTIONS

Hydrogen chloride and deuterium chloride are corrosive in both aqueous and gaseous forms. Care must be taken to verify that all vapor is kept inside of the fume hood and that the gas cell is not opened when full. Gloves should be worn throughout the entirety of the experiment. All glassware must be properly cleaned and dried in order to avoid unsafe reactions inside of the gas cell.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.8b00702.

Student handout with instructions (PDF, DOCX)

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Notes

The authors declare no competing financial interest.

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