

SOP Nicolet IR

Computer:

Username: iruser

Password: iruser

OMNIC: organic

Procedure:

1. Acquire a background spectrum of the closed absorption cell.
2. Take the HCl absorption spectrum. Leave the valves on the absorption cell closed so that you don't release the gases into the room.
 - a. Install the absorption cell containing HCl in the FTIR. Slide the black mounting plate down into the slot in the sample holder within the spectrometer. Close the sample chamber.
 - b. Start the OMNIC spectroscopic program on the computer. We have recorded a background with an empty cell. Select this background by going into the collect>collect setup dialog box and clicking on the lowest button for background handling. This button selects "read background from file."
 - c. Then click the browse button and select the file "C:\omnic\spectra\434f06\hcl_bg.spa" as your background file.
 - d. Set the number of scans to 8 and set the resolution to 0.25 cm^{-1} and make sure that the final format is set to "absorbance".
 - e. Record the spectrum (under the menu collect, select collect sample). A dialog pops up asking you to prepare for the spectrum. Select OK.
 - f. When the spectrum is done, add it to window one and examine it. Save the spectrum under a reasonable name and record the name in your lab book.
3. The HCl peaks are at around 2900 cm^{-1} .
 - a. Zoom in on the HCl region. Be sure to include HCl small peaks on the edges of the spectrum.
 - b. Note that all peaks are doubled, with an intensity ratio of approximately 3:1. The smaller peak is from H^{37}Cl and the larger from H^{35}Cl . The intensity ratio arises from the fact that the natural abundance of chlorine isotopes is $3\text{ }^{35}\text{Cl} : 1\text{ }^{37}\text{Cl}$.
 - c. You are going to analyze the H^{35}Cl peaks, so you need to determine the wavenumbers of the absorption peaks for this isotopomer.
4. Open a log (under the file menu). This starts a log file which will contain the spectral peak information for later analysis.

5. Find the peaks (under the analysis menu). Adjust the threshold level (the horizontal line across the spectrum) downward until all the visible peaks are picked (probably you will pick a few noise peaks also).
6. Click on the “add” button at the top right of the peak picking window.
 - a. Enter a title for the new window which will be created. This adds the peaks to the spectrum and writes the data to the log.
7. Zoom in on the spectrum so it looks good and print it.
8. Close the log file.
9. Copy the log file to a floppy disk. This file is a RTF file, which can be read by MS word or other word processing programs.
10. Acquire the HCl overtone absorption spectrum.
 - a. FTIR spectrometer has been reset to a higher frequency range ($5000\text{-}6000\text{ cm}^{-1}$)
 - b. Sample cell has been filled to 1 atmosphere of HCl.
 - c. Record at a resolution of 0.25cm^{-1} , 24 scans, and save the acquired spectrum is called "hclover.spa".
 - d. Zoom in on the HCl region, including the HCl small peaks on the edges of the spectrum.