Agilent Cary 630 FTIR Spectrometer Supports Undergraduate Teaching Laboratories

Rotational spectrum of HCl

Application Note

Academic

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Introduction

IR spectroscopy is a fundamental analytical technique that undergraduate physical, analytical, and organic chemistry students must understand and have the opportunity to use. The demands of undergraduate labs require that FTIR spectrometers possess certain design attributes associated with the multiuser environment. The spectrometer must be easy to use, flexible, rugged, reliable, compact, and cost attractive. The Agilent Cary 630 FTIR meets these requirements effectively.

The Cary 630 FTIR spectrometer provides spectral measurements for a wide range of materials and chemicals. Designed with a compact footprint, with its full complement of accessories, the Cary 630 can measure liquids, solids, gases, and provide analyses in virtually all traditional application spaces.

With minimal training, students can use the Cary 630 for qualitative analysis associated with organic synthesis, as well as quantitative analysis in an analytical or instrumental laboratory.

This application note shows the Cary 630 used for a typical undergraduate analytical and physical chemistry experiment to measure the properties of hydrogen chloride or a similar diatomic gas.

Agilent Cary 630 FTIR Spectrometer

Advantages for the academic environment, applications in teaching and routine research support

Overall system design

- Most compact, superior performing FTIR available. Ultracompact design preserves valuable bench top space
- Lightweight, allows system to be relocated as needed
- Completely sealed optics and compact size enables use in standard fume hoods
    - Broad range of sampling interfaces available, handling liquids, solids, and gases
    - Overall performance and ease-of-use; advantages translate into virtually all analyses requiring less than one minute to execute

Optomechanical components

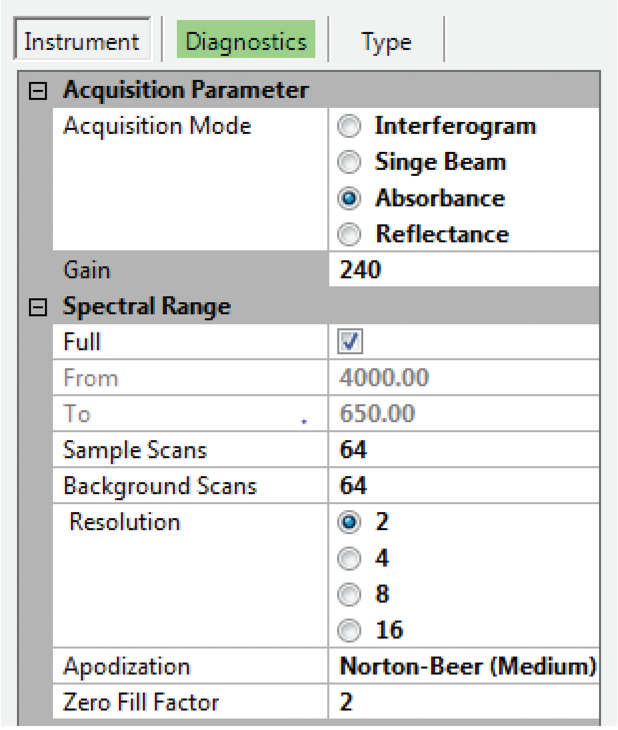
- The permanently aligned interferometer and optical system results in an extremely reliable, everyday workhorse system.
- Interferometer and optics are exceedingly rugged. The Cary 630 uses the same optomechanical components as those used in Agilent portable and handheld industrial systems, which require the highest level of robustness.
- Large aperture optics and short internal optical paths provide class-leading performance.

Method and Measurements

A Cary 630 FTIR spectrometer with a transmission accessory (G8043 #300) and an Agilent 50-mm gas cell (G8043 #306) were used to make these measurements. Agilent MicroLab Expert software (G4097AA) was used to control the instrument as well as measure and analyze the data.

The measurement of HCL gas was carried out as follows: The gas cell was installed into the transmission module positioning the inlet and outlet to allow the sample to be added and removed from the cell. With the cell empty of the sample gas, the spectrometer gain was set to an intensity level between 18,000 and 25,000. This provided sufficient signal level for measuring the sample with an optical resolution of 2 cm^{-1}. The data collection parameters were set and a background spectrum was obtained before adding the sample and collecting the sample spectrum.

Figure 1 shows a screenshot of the parameters used for data collection.



Acquisition Parameter	
Acquisition Mode	○ Interferogram ○ Singe Beam ● Absorbance ○ Reflectance
Gain	240
Spectral Range	
Full	☑
From	4000.00
To	650.00
Sample Scans	64
Background Scans	64
Resolution	● 2 ○ 4 ○ 8 ○ 16
Apodization	Norton-Beer (Medium)
Zero Fill Factor	2

Figure 1. Screenshot of data collection parameters.

Sampling interfaces

- No-alignment, interchangeable sampling accessories allow students to understand and readily practice different experimental methods, such as transmission and ATR.
- Sampling interfaces available include: diamond ATR, diffuse reflectance, solid, liquid, and gas transmission, as well as the Agilent innovative, exclusive DialPath technology for liquids analysis.
- The diamond ATR sampling interface is ideal for the analysis of reaction starting materials, reagents, and product. It is impervious to scratching, and highly chemically resistant (pH 1–14).
- The ATR sampling interface enables grab sample, neat reaction mixture analysis. Sample dilution is not necessary.
- A powder press, which ensures good optical contact of solids with ATR crystal, cannot be overtightened, that is, the diamond window cannot be damaged by overpressure.

Software and user interface

- Highly visual, intuitive software allows data acquisition with essentially no training required.
- Real-time spectrum display works with an ATR powder press to ensure adequate contact between solid samples and a diamond ATR crystal.
- Experiment results can be stored under an individual student's name or other identifier.
- Students can verify the identity of starting materials, isolated intermediate compounds, and final products by comparison with spectra in the on-board IR spectral library.
- For alternative data analysis or display options, data files are formatted to readily transfer to standard commercially available data processing, analysis, and display packages.
- On-board analytics ensure peak operating efficiency.

MicroLab Expert software provides the ability to visualize and manipulate spectroscopic data especially valuable in the academic setting, for multilevel users in areas of nonroutine analysis such as synthesis and R&D lab work. Microlab Expert software is a full featured package, capable of:

- Spectral display and manipulation
- Mathematical functions
- Functional group analysis
- Library searching
- Options for univariate and multivariate quantitative analysis

Microlab Expert software also is seamlessly interfaced to the method guided tools in MicroLab PC, retaining all the benefits provided by this software for the introductory lab setting.

A few comments on parameter selection

- **Number of scans:** Can be reduced to 32 or even 16, if speed is a requirement. The use of 64 scans still keeps the scan time in the range of ~ 1 minute.
- **Apodization:** The use of boxcar apodization is commonly considered to minimize the band or line width. In this case, by changing to a Norton-Beer medium apodization, no significant contribution to the line width occurs. The lines remain well separated and the baseline ringing is reduced, improving the appearance of the spectrum.
- **Zero Fill Factor:** By increasing the ZFF to 2, the calculation inserts one data point between each existing point from a corresponding transform using no zero fill. The result is an improved peak shape, and no increase in the line width. Drawbacks are that the file will be twice as large and unless apodization is used, the baseline ripple would be larger.

The instructor can allow the students to learn how to set the parameters for an experiment, and direct them to quickly change parameters to see how results are affected. For example, if the $H^{35}Cl/H^{37}Cl$ isotope ratio is desired, then box car apodization and additional zero-filling will improve the displayed spectrum for making those measurements. Alternately, the instructor can set up experimental parameters, and install an experimental method that operates in pushbutton mode.

Discussion - Measuring the Spectrum of Hydrogen Chloride Gas

This experiment typically examines the fine structure of the vibrational fundamental line for $H^{35}Cl$ to determine several spectroscopic parameters such as rotational constants. Assuming that the HCl molecule behaves approximately as two masses connected by a stiff spring of constant *k*, rotating with internuclear distance *r*, these molecular parameters can be extracted from the appropriate spectroscopic constants.

Spectral measurement of diatomic gases must be made at a resolution sufficient to adequately separate the rotational lines of the molecule, and allow accurate line position measurements to be made. The Cary 630, when operated at a resolution of 2 cm^{-1}, delivers the expected spectral results.

The collected HCl spectral results shown in Figure 2 provide the position of 10 lines from each of the P and R branches. This data can then be used to calculate physical constants and spectral values, as specified by the instructor's goals for the experiment.

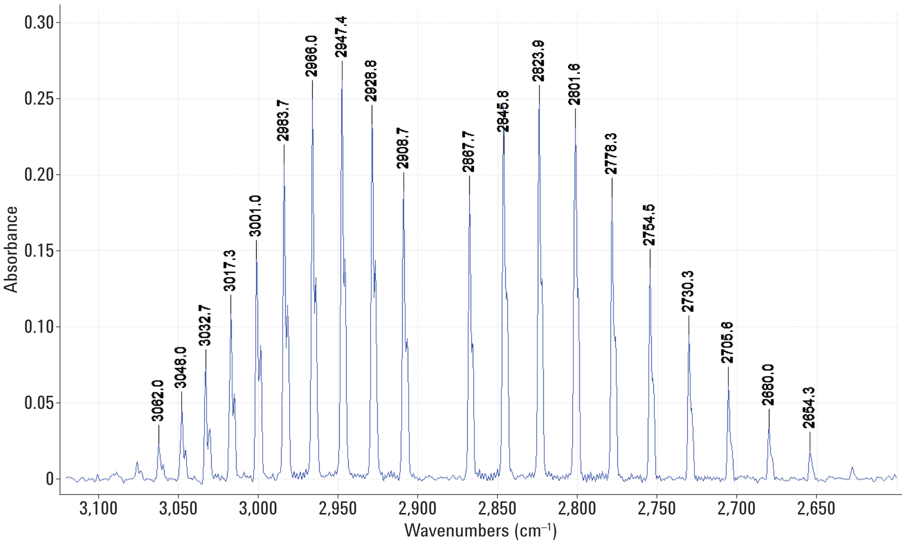


Figure 2. The absorbance spectrum of HCL is shown here. P-branch lines shown to the right of center and the R-branch lines shown to the left of center.

Conclusion

The Agilent Cary 630 FTIR has a unique combination of versatile sampling, ease-of-use, and robust system components, combined with attractive pricing and low cost of long-term operation. Thus, the spectrometer effectively meets and exceeds the needs and requirements of the multiuser academic environment. Students in analytical instrumentation laboratories, physical chemistry, and organic chemistry labs will find the spectrometer perfect for their work, and instructors will find that the system provides hassle-free operation.

This application note shows the Cary 630 FTIR measuring the rotational-vibrational spectrum of hydrogen chloride in a common physical chemistry laboratory. The acquired spectrum provides the student with values to calculate the physical properties of the molecule, including average bond length, rotational constants, and fundamental line positions.

References

1. Numerous laboratory experiments can be found from simple internet searches. These describe sample handling, precautions, theory, and calculations needed in the write-up from collected spectral data.
2. Garland, Nibler, Shoemaker. Experiments in Physical Chemistry, Ed 8 (2008).

For More Information

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