Instrument Design for Low-Temperature Infrared Spectroscopy of Materials

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ABSTRACT

Solid material analysis has come a long way in its ability to accurately characterize the structure of bulk materials progressing from magnifying glasses to today's atomic resolution instruments. Present common techniques include the use of various IR spectroscopies to determine the structural properties of these materials. Typically, these methods are low-resolution and are used to pinpoint only a few vibrational modes in the material's structure. In addition, these studies are either done near room temperature or at one temperature setting. This poster presents a custom-built cryogenic FTIR instrument that is capable of characterizing a given material from room temperature down to 30K with < 2 cm-1 resolution. Shown to the right and below is the design, creation and specific components of the cryogenic instrument, future plans include incorporating gas-species dosing to allow for gas adsorption experiments. The Dodson laboratory is one of few labs capable of cryogenic IR analysis of various metal and covalent-organic frameworks using these necessary techniques to fully characterize the changes in structure and vibrational modes of these materials as they approach extremely low temperatures.

CRYOGENIC-FTIR INSTRUMENT

Figure 1: A fully assembled apparatus in an open FTIR spectrometer

A helium cryo-stat is mounted above a Fourier Transform IR spectrometer that penetrates the sample at the bottom of the coldfinger. The expansion of compressed helium enables the cryo-stat to cool the sample down to 30K. Monitored by a temperature gauge, this design allows for the collection of multiple spectra over a range of temperatures with < 2 cm -1 resolution.

CURRENT APPLICATIONS

This design has aided in the cryogenic analysis of various COF and MOF compounds over a range of sub-ambient temperatures. Observing both peak shifts as a function of temperature, shown in the figure below can help map the progress of the compound's structure as it loses energy.

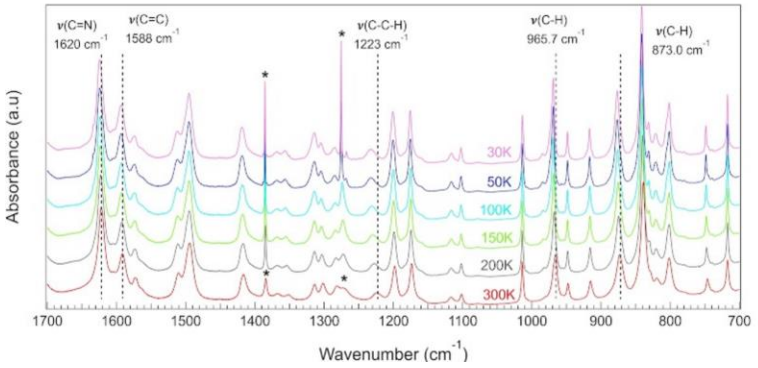

Figure 5: IR spectra of COF-300 over a range of temperatures

ACKNOWLEDGMENTS

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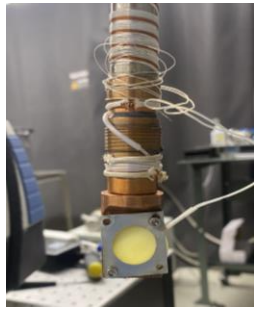
DESIGN

Figure 2: The custom material mount was manually carved out of copper and cut to screw into the coldfinger. An aluminum plate is used to press the sample into the copper mount for optimal thermal contact between the sample and the mount.



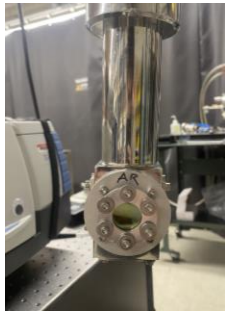


Figure 3: The mount is screwed into the coldfinger and a temperature diode is placed in contact with the sample. The customized mount optimizes the heat transfer between the coldfinger and the sample, allowing for accurate cryogenic analysis.

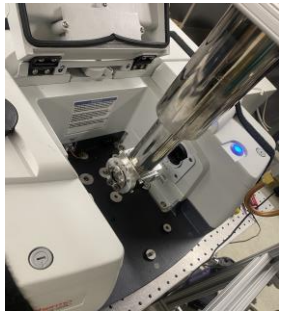


Figure 4: The sample and coldfinger are encapsulated in a steel shroud and placed under high vacuum. The optic lens at the bottom of the shroud allows for light to penetrate the sample without interference.