NE 226 L

Characterization of Materials

Title Page

Name:	Rajesh Swaminathan
ID Number:	20194189
Partners' Names:	Anterjot Bains
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1. Introduction

Fourier Transform Infrared Spectroscopy (FTIR) is a useful way of non-destructively analyzing the kinds of chemical bonds present in a material. Different bonds respond differently to incoming radiation due to variety in their molecular vibrations of stretching and bending. This response may be characterized by observing the percent transmission of infrared radiation and comparing it with known standards to identify the type and nature of chemical bonds. FTIR is applicable not only to solid materials, but liquids and solids as well. One limitation of IR spectroscopy is that molecule being studied must have a permanent dipole to be IR active.

The objective of FTIR is to determine the type of bonds present in a material by obtaining the percent transmission as a function of incident radiation wavelength. The ratio of the intensities of the transmitted and incident light are plotted against the incident light's wavelength (or frequency/wavenumber) to obtain a fingerprint spectrum that is unique to that material. Interpretation of the spectrum with the help of known standards helps us identify the nature of the chemical bonds present in the material (qualitative). It also helps us determine, with the help of Beer-Lambert's law, the amount of material present in a sample by analyzing the relative size of the absorption peaks (quantitative). Further, an unknown sample can be identified by comparing its spectrum with spectrums of common materials available in computer databases. This application requires the use of modern software algorithms.

The major idea behind FTIR is that all molecules above absolute zero temperatures are in constant stretching and bending vibration. These molecules absorb incident radiation if its own vibrational frequency equals the frequency of the incident radiation. Most importantly, molecules can absorb frequencies only corresponding to discrete energy levels due to energy quantization.

2. Materials and Methods

The instrument used in this experiment is the Tensor 27 FT-IR manufactured by Bruker Optics GmbH. The experimental procedure used for this experiment was outlined in the lab manual of *Characterization of Materials* [1], under experiment number 1. The two samples studied were chromium (II) acetate ($Cr(CH_3COO)_2 \cdot H_20$) and ethanol (CH_3CH_2OH).

FTIR is an advancement over dispersive instruments like prisms and diffraction graters in that it allows us to examine all frequencies simultaneously. This helps us obtain spectrums much more quickly (in about one second per scan) and results in greater optical throughput. The FTIR is much more sensitive than its dispersive counterparts resulting in much lower noise levels. These combined advantages make FTIR measurements extremely accurate and reproducible.

The basic components of an FTIR system are: the radiation source, the interferometer and the DTGS detector (Figure 1). A background spectrum must be obtained each time the

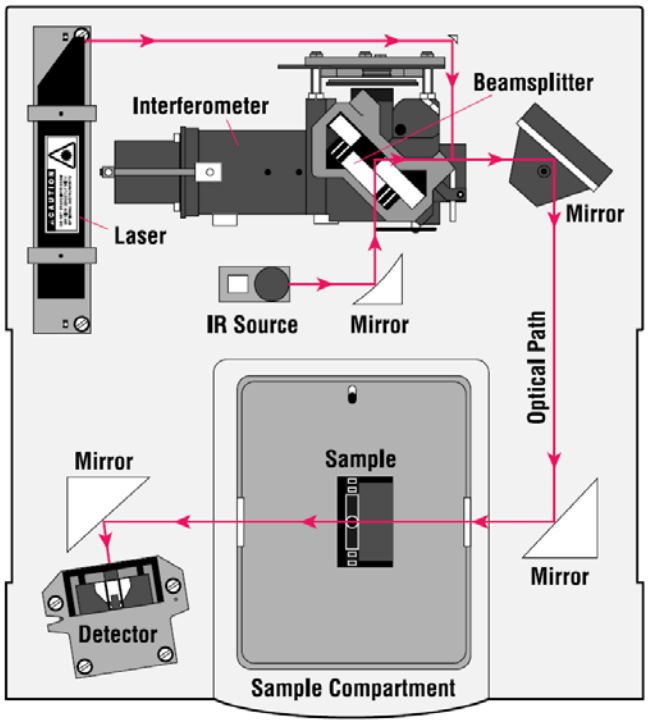

Figure 1 Basic Components of an FTIR System: The interferometer serves as the replacement counterpart for prisms (or diffraction gratings) used in traditional dispersion optics.

FTIR is used in order to remove all instrumental characteristics, thereby ensuring that the resulting spectrum is strictly a function of the sample only. The instrumentation process outlining the sequence of steps involved in using an FTIR is summarized in Figure 2.

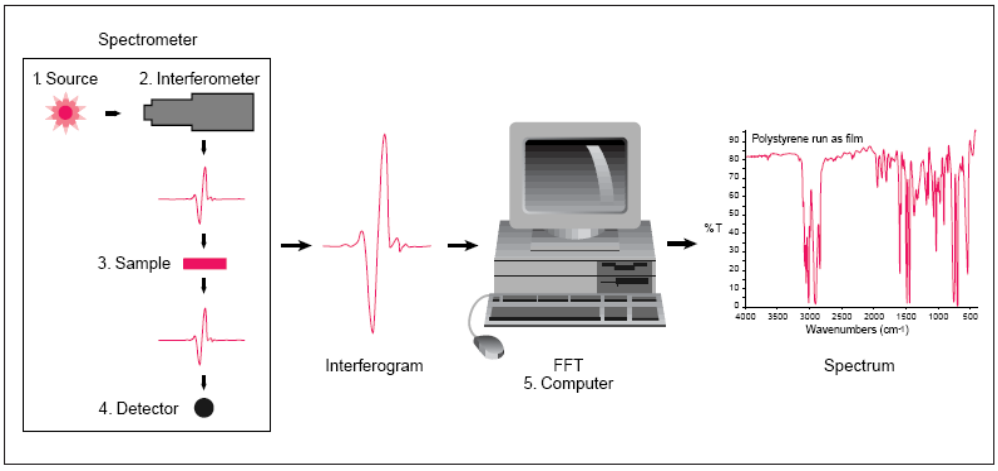

Figure 2 FTIR Instrumentation Process: The sequence of steps involved in obtaining an IR spectrum using an FTIR.

3. Results

The transmittance (%) vs. wavenumber (cm^{-1}) spectroscopy printouts for a) chromium acetate and b) ethanol are attached at the end of this report.

4. Discussion

The observed peaks for a) chromium acetate and b) ethanol and their corresponding functional groups and type of vibrations are summarized in Tables 1 and 2. The type of vibration was determined from IR literature values. [2]

Wavenumber (cm^{-1})	Functional Group	Type of Vibration
1544.99	Alkane	H-C-H stretch
1451.79	Alkane	H-C-H bend
1031.41	Ester	C-O stretch
676.83	Chromium (II) Acetate	Cr-O stretch

Table 1 Principle Absorption Peaks for Chromium Acetate

We observe strong absorption near a frequency of 1031.41 cm^{-1} that corresponds to one or more esters. We also identified alkane groups such as CH_3 and CH_2. The stretch vibration occurs at a higher frequency (higher energy) than the bend vibration.

Wavenumber (cm^{-1})	Functional Group	Type of Vibration
3341.07	Alcohol	Hydrogen bonded O-H stretch
2973.77	Alkane	H-C-H Asymmetric & Symmetric stretch
2258.30	Nitrile	C≡N Stretch
2135.52	Alkyne	C≡C Stretch

Table 2 Principle Absorption Peaks for Ethanol

We know that ethanol does not contain nitriles or alkynes. This might be due to some sample left over in the liquid cell from a previous run, impurities in the sample, or a defect with the FTIR equipment. There were a few other absorptions near the fingerprint region of the spectrum which are not shown in Table 2. The major functional groups of interest are the alcohol at 3341.07 cm^{-1} and the alkane at 2973.77 cm^{-1}. These results, together with the result of a mass spectrometer or an NMR will provide us enough information to immediately tell us whether this compound is methanol, ethanol, or propanol, etc.

5. Conclusions

The FTIR spectrums obtained, together with IR literature values tell us that ethanol contains alkane groups like CH_3 and CH_2, and an OH group that is characteristic of alcohols. The spectrum for chromium (II) acetate gives us information that the compound

contains H-C-H bonds. In addition, absorptions corresponding to C-O stretches tell us the molecule contains ester functional groups as well.

6. References

[1] Q. Xie, F. McCourt, *Nanotechnology Engineering NE 225 Lab Manual*, University of Waterloo, Waterloo, pp 16-27 (2007).

[2] Principal IR Absorptions for Certain Functional Groups, *IR Literature Values*, NE 225 FTIR Supplement.