S7 INFRARED SPECTROSCOPY

AIM
1. To become familiar with the operation of an infra-red spectrometer
2. To run spectra using different sample methods
3. To examine absorption peaks characteristic of some organic functional groups
4. To determine the identity of an unknown organic liquid by comparing its infrared spectrum to standard spectra

INTRODUCTION
Absorption of light in the infrared region of the electromagnetic spectrum is due to the energy interacting with the atoms in the molecules, resulting in an increase in the vibrational and rotational modes of the bonded atoms.

In organic compounds, the spectrum of a compound is in most cases characteristic of the compound and spectra can thus be used to help in the identification of unknown compounds.

The infrared spectrophotometers available in the laboratory are of two types:
- similar construction to UV-vis spectrophotometers except with a different radiation source and detector
- Fourier Transform Infra Red (FTIR) instrument which has an entirely different optical system

The sample preparation for both instruments is similar.

When using infrared spectrophotometers it is rare to use a blank although a reference beam is used to compensate for the strong absorption of infrared radiation by the CO₂ and H₂O present in the air.

The selection of the materials of construction for sample containers and the selection of solvents for use in infrared spectrophotometry is restricted because many common materials absorb infrared radiation.

In IR it is usual to express the wavelength in micrometers (microns), 1 micro = 10⁻⁶ m, or in wavenumbers, cm⁻¹.

Wavenumber = number of wavelengths of the radiation that would fit into 1 cm.
PROCEDURE
The teacher will demonstrate the use of the spectrophotometer. Examine the main parts of scanning infrared spectrophotometer, noting the sample compartment and the types of sample holders available.

You are to record the following spectra:

I. Polystyrene Thin Film
1. A pre-mounted sample of polystyrene film is available. Place it in the sample beam (no reference) and record its spectrum using

The scan is useful as a calibration tool. Compare the observed wavenumber of the calibration bands with the listed values for polystyrene. Present the results as a table that shows the error found at different parts of the IR spectrum.

II. Liquid Film Spectrum
Characteristic Functional Group Peaks
2. Record by the liquid film technique (placing a couple of drops of the liquid on NaCl plates), the spectra of the following:
   * cyclohexane
   * cyclohexanol
   * methylbenzene (toluene)
   * phenylethanone (acetophenone)
   * cyclohexene
   * cyclohexanone
   * phenylmethanol (benzyl alcohol)
   * phenylmethanal (benzaldehyde)

III Nujol mull spectrum
3. Place 1-2 drops of Nujol oil on a NaCl plate and cover with another NaCl plate. Mount the two in the appropriate holder, place in the sample beam and record the spectrum.
   If you have made the sample too thick, and some of the peaks are bottoming out and being truncated, remove one plate, rub it clean on a tissue, place it back on the other plate. If the sample is now too thin add more paste and repeat.
4. Place 2-3 drops of Nujol in the agate mortar and add a small amount of benzoic acid. Grind well for about 5 minutes until an even mixture is obtained.
5. Scrape the mull onto a NaCl plate and record the spectrum as for a liquid film spectrum.
IV. KBr Disk

6. The sample compound in a KBr disc should be at a concentration of 1-2% w/w. It is not necessary to accurately measure the weights of benzoic acid and KBr. Observe the approximate amount used in the demonstration. 5-10 crystals of benzoic acid and 2-3 ‘microspatula-full’s’ of KBr should be sufficient.

7. Make sure that you grind the mixture well in a clean agate mortar and pestle before attempting to press the disc. The mixture should have the consistency of cornstarch.

8. The die comes in four pieces:
   (i) cylindrical exterior body 
   (ii) inset base 
   (iii) plunger 
   (iv) small cylindrical metal disc

9. Place the small disc (iv) on the centre of the base (ii) and place the exterior body over the top, so that the base and body seal properly.

10. Sprinkle sufficient of the KBr mixture into the press to just cover the metal surface. Insert the plunger and with hand pressure, rotate to spread the material evenly.

11. Carefully remove the plunger, and check that no material has stuck to it. If there is, it may mean that the mixture is moist and should be discarded.

12. If there are any thin spots in the powder, repeat step 8, and then press at 10 tonnes, under vacuum, for 30 seconds.

13. Release the pressure slowly, invert the die and remove the base.

14. Gently tap the plunger on the bench until the friction seal is broken. The small disc will emerge above the base. Carefully remove it, and using a spatula or razor-blade, place the KBr disc into its holder. Inspect for major cracks or holes.

15. Place the disc holder in the sample beam and record the spectrum.
RESULTS

I Polystyrene film

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<th>Expected cm$^{-1}$</th>
<th>Found cm$^{-1}$</th>
<th>Expected cm$^{-1}$</th>
<th>Found cm$^{-1}$</th>
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<td>1028</td>
<td>2851</td>
</tr>
<tr>
<td>1181</td>
<td>3027</td>
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II Characteristic Functional Group Peaks

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<tr>
<th>Compound</th>
<th>Structure</th>
<th>Characteristic Peak(s) (shape, cm$^{-1}$)</th>
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<tr>
<td>phenylmethanal</td>
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III Nujol Mull
- Window for use with Nujol
- Appearance of peaks in the Nujol window

IV KBr Disk
Describe the appearance of the spectrum
DISCUSSION

- Determine the characteristic peaks for the functional groups in Part II Liquid Film Spectroscopy
- in terms of convenience and appearance of spectrum, compare the spectrum of benzoic acid from:
  i) the KBr disc
  iv) Nujol mull
- explain why NaCl and KBr are used as cell materials in IR spectroscopy.

QUESTIONS
1. Why is infrared spectroscopy
   a) excellent for qualitative analysis
   b) poor for quantitative analysis?
2. Why is agate used as the material for the mortar and pestle, rather than the normal ceramic material used in other laboratories?
3. Why is care taken when handling NaCl plates not to touch their faces with bare fingers?
4. Which of the following compounds would be a suitable cell material for IR:
   * sodium bromide  * sodium hydroxide  * silver chloride
   * potassium sulfate  * caesium iodide  * quartz
Explain your reasons.

Have you?

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<td>Completed the standard register</td>
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Teachers signature