Measuring Properties of Inorganic Compounds in Lab

1. IR spectra
2. UV-Vis spectra
3. Conductivity
Measuring Properties of Inorganic Compounds in Lab

1. IR spectra

M(II)hexamine complexes

- N-H stretch
- N-H bend
- M-NH₃ bend
- M-NH₃ rock
Principle of IR spectroscopy

- Molecules are made up of atoms linked by chemical bonds. The movement of atoms and the chemical bonds like like spring and balls (vibration)

- This characteristic vibration are called Natural frequency of vibration.
**BASIC PRINCIPLE:**

- A chemical substance shows marked selective absorption in IR region.
- After absorption of IR radiation the molecules of chemical substance vibrate at many rates of vibration giving rise to closely packed absorption spectrum called as **IR absorption spectrum**.
- Various bands present in the IR spectrum corresponds to characteristic functional group and bonds present in the chemical substance.
- Thus IR spectrum of a chemical substance is fingerprint for its identification.
MULL TECHNIQUE:

- In this technique a small quantity of sample is thoroughly ground in a clean mortar until the powder is very fine.

- After grinding, the mulling agent (mineral oil or Nujol) is introduced in small quantities just sufficient to take up the powder (mixture approximates the consistency of a toothpaste).

- The mixture is then transferred to the mull plates & the plates are squeezed together to adjust the thickness of the sample between IR transmitting windows.

- This is then mounted in a path of IR beam and the spectrum is run.
**Demerit:**

- Although Nujol is transparent throughout IR region, yet it has a disadvantage that it has absorption maxima at 2915, 1462, 1376 & 719 cm\(^{-1}\).

- So when IR spectrum of solid sample is taken in Nujol mull, absorption bands of solid sample that happen to coincide with the absorption bands of the Nujol mull will be hidden (but others will be clearly seen in IR spectrum) and then interferes with the absorption of the sample.
- **Infrared spectroscopy (IR)** measures the bond vibration frequencies in a molecule and is used to determine the functional group.

- **The IR region** is divided into three regions:
  1. The near IR (12500-4000 cm\(^{-1}\)) (overtons region)
  2. The mid IR (4000-200 cm\(^{-1}\))
  3. The far IR (200-10 cm\(^{-1}\))

- **The mid IR region** is of greatest practical use to the organic compounds.
Cyclohexanol neat solution
<table>
<thead>
<tr>
<th>Type of bond</th>
<th>Wavenumber (cm$^{-1}$)</th>
<th>Intensity</th>
</tr>
</thead>
<tbody>
<tr>
<td>C≡N</td>
<td>2260–2220</td>
<td>medium</td>
</tr>
<tr>
<td>C≡C</td>
<td>2260–2100</td>
<td>medium to weak</td>
</tr>
<tr>
<td>C≡C</td>
<td>1680–1600</td>
<td>medium</td>
</tr>
<tr>
<td>C=N</td>
<td>1650–1550</td>
<td>medium</td>
</tr>
<tr>
<td></td>
<td>~1600 and ~1500–1430</td>
<td>strong to weak</td>
</tr>
<tr>
<td>C=O</td>
<td>1780–1650</td>
<td>strong</td>
</tr>
<tr>
<td>C−O</td>
<td>1250–1050</td>
<td>strong</td>
</tr>
<tr>
<td>C−N</td>
<td>1230–1020</td>
<td>medium</td>
</tr>
<tr>
<td>O−H (alcohol)</td>
<td>3650–3200</td>
<td>strong, broad</td>
</tr>
<tr>
<td>O−H (carboxylic acid)</td>
<td>3300–2500</td>
<td>strong, very broad</td>
</tr>
<tr>
<td>N−H</td>
<td>3500–3300</td>
<td>medium, broad</td>
</tr>
<tr>
<td>C−H</td>
<td>3300–2700</td>
<td>medium</td>
</tr>
</tbody>
</table>
MOLECULAR VIBRATIONS

There are 2 types of vibrations.

1) Stretching vibrations

2) Bending vibrations

• 1) Stretching vibrations: in this bond length is altered.
• They are of 2 types
• a) symmetrical stretching: 2 bonds increase or decrease in length.
b) Asymmetrical stretching: in this one bond length is increased and other is decreased.

2) Bending vibrations:
• These are also called as deformations.
• In this bond angle is altered.
• These are of 2 types
  • a) in plane bending → scissoring, rocking
  • b) out plane bending → wagging, twisting
Scissoring:

This is an in plane bending.

In this bond angles are decreased. 2 atoms approach each other.

Rocking:

- In this movement of atoms takes place in same direction.
Wagging:
It is an out of plane bending.
In this 2 atoms move to one side of the plane. They move up and down the plane.

Twisting:
- In this one atom moves above the plane and the other atom moves below the plane.
SYMMETRIC STRETCHING

ANTISYMMETRIC STRETCHING

ROCKING

WAGGING

TWISTING

SCISSORING
Here, color can distinguish the two
Also Far IR (500 – 300 cm$^{-1}$)
Trans: one peak
Cis: two peaks
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2. UV-Vis spectra

![UV-Vis spectra graph showing absorbance vs. wavelength for NH$_3$, Co$^{2+}$ ion, and [Co(NH$_3$)$_6$]$^{2+}$ complexes.](image)
Principles of Spectroscopy

• The principle is based on the measurement of spectrum of a sample containing atoms / molecules.

• Spectrum is a graph of intensity of absorbed or emitted radiation by sample verses frequency (ν) or wavelength (λ).

• Spectrometer is an instrument design to measure the spectrum of a compound.
The electronic spectrum of $[\text{Ni(H}_2\text{O)}_6]^{2+}$:

The complex looks green, because it absorbs only weakly at 500 nm, the wavelength of green light.
Sample vis spectrum
Measuring Properties of Inorganic Compounds in Lab

3. Conductivity
[Co(NH₃)₃Cl₃] – does not ionize.
[Co(NH₃)₄Cl₂]Cl = [Co(NH₃)₄Cl₂]⁺ + Cl⁻ (2 ions)
[Co(NH₃)₆]Cl₃ = [Co(NH₃)₆]³⁺ + 3Cl⁻ (4 ions)
[Co(NH₃)₅Cl]Cl₂ = [Co(NH₃)₅Cl]²⁺ + 2Cl⁻ (3 ions)

As the number of ions in solution increases, their conductivity also increases. Therefore, conductivity follows the order:
[Co(NH₃)₃Cl₃] < [Co(NH₃)₄Cl₂]Cl < [Co(NH₃)₅Cl]Cl₂ < [Co(NH₃)₆]Cl₃

<table>
<thead>
<tr>
<th>Number of ions</th>
<th>molar conductivity, Ω⁻¹ cm² mol⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>2 (1:1)</td>
<td>96-150</td>
</tr>
<tr>
<td>3 (1:2)</td>
<td>225-273</td>
</tr>
<tr>
<td>4 (1:3)</td>
<td>380-435</td>
</tr>
<tr>
<td>5 (1:4)</td>
<td>540-560</td>
</tr>
</tbody>
</table>

(For aqueous solutions)
OPERATING INSTRUCTIONS

1. Connect the conductivity cell or resistance to be measured between the two blue binding posts on the top panel.
2. Connect compensating capacitor, if any, between the end-blue and the black binding posts on the top panel.
3. Apply power, selecting either LINE or 1K Hertz bridge frequency.
4. Adjust SENSITIVITY control to obtain a dark "window" in the center of the null indicator.
5. Adjust SENSITIVITY control as necessary. Indication may be ambiguous between two adjacent ranges.
6. Rotate the RANGE/FUNCTION switch to obtain the longest "window" on the null indicator. Re-adjust the SENSITIVITY control as necessary. It may be necessary to switch to an adjacent range if it value is near one end of the dial scale.
7. Read the conductance (resistance) value off the dial scale and multiply by scale factor from the function switch to obtain the final reading.

(For further information see instruction manual.)
OPERATING INSTRUCTIONS

1. Connect the conductivity cell or resistance to be measured between the two blue binding posts on the top panel.
2. Connect compensating capacitor, if any, between the end-blue and the black binding posts on the top panel.
3. Apply power, selecting either LINE or 1K Hertz bridge frequency.
4. Adjust SENSITIVITY control to obtain a dark “window” in the center of the null-indicator.
5. Rotate the RANGE/FUNCTION switch to find the longest dark-window on the null indicator. Re-adjust the SENSITIVITY control as necessary. Indication may be ambiguous between two adjacent ranges.
6. Rotate the DRIVE control to obtain the longest window on the null-indicator. Re-adjust the SENSITIVITY control as necessary. It may be necessary to switch to an adjacent range if value is near one end of the dial-scale.
7. Read the conductance (resistance) value off of the dial scale and multiply by scale factor from the function switch to obtain the final reading.

(For further information see instruction manual.)