SNS COLLEGE OF PHARMACY AND HEALTH SCIENCES

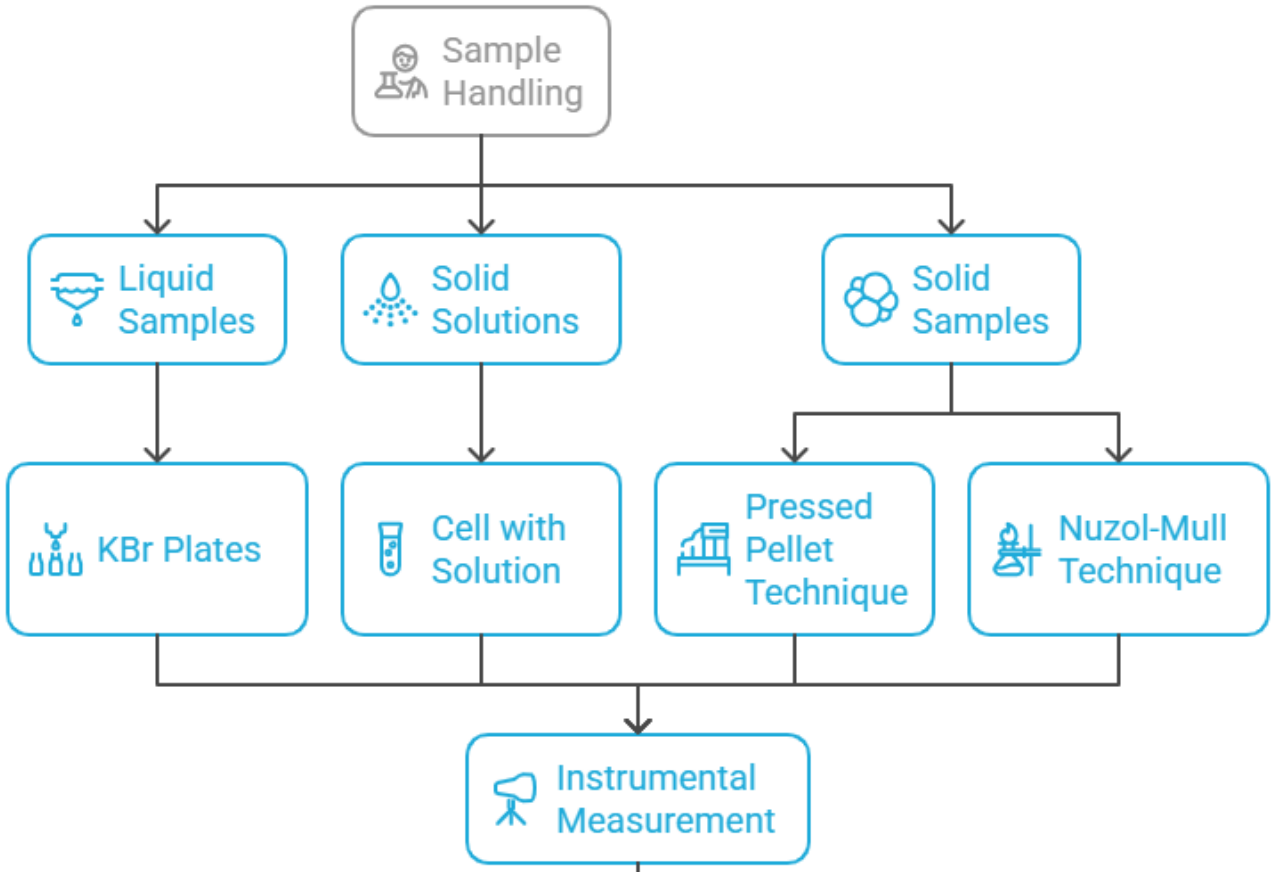
Affiliated To The Tamil Nadu Dr. MGR Medical University, Chennai
Approved by Pharmacy Council of India, New Delhi.
Coimbatore -641035

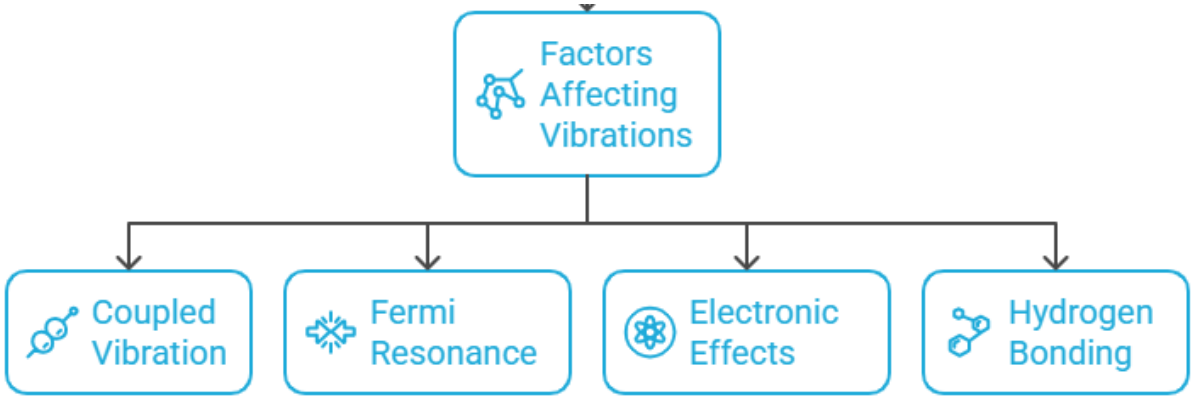
COURSE NAME: INSTRUMENTAL METHODS OF ANALYSIS (BP 701 T)

VII SEM/ IV YEAR

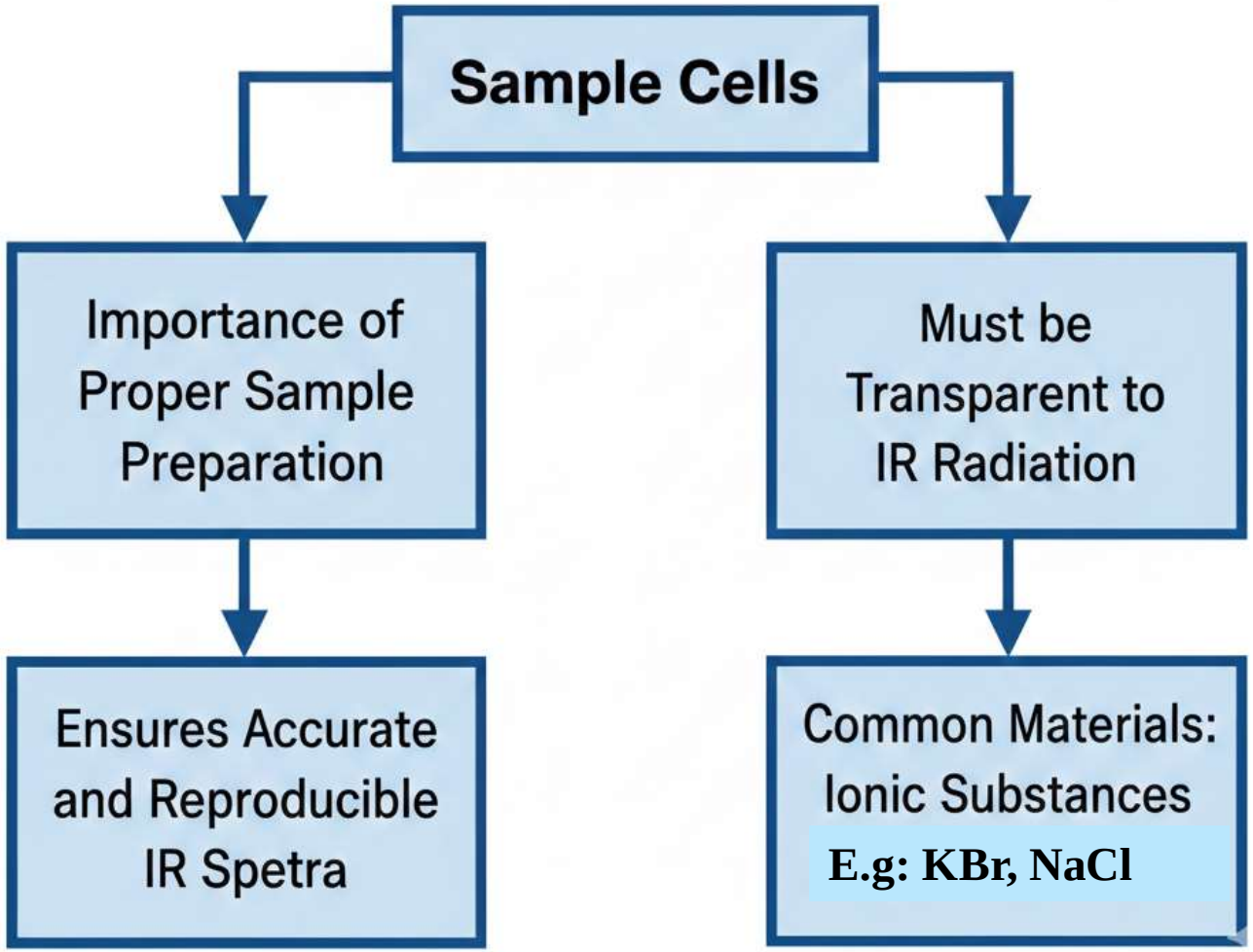
TOPIC 12: SAMPLE HANDLING AND FACTORS AFFECTING VIBRATIONS

MINDMAP:

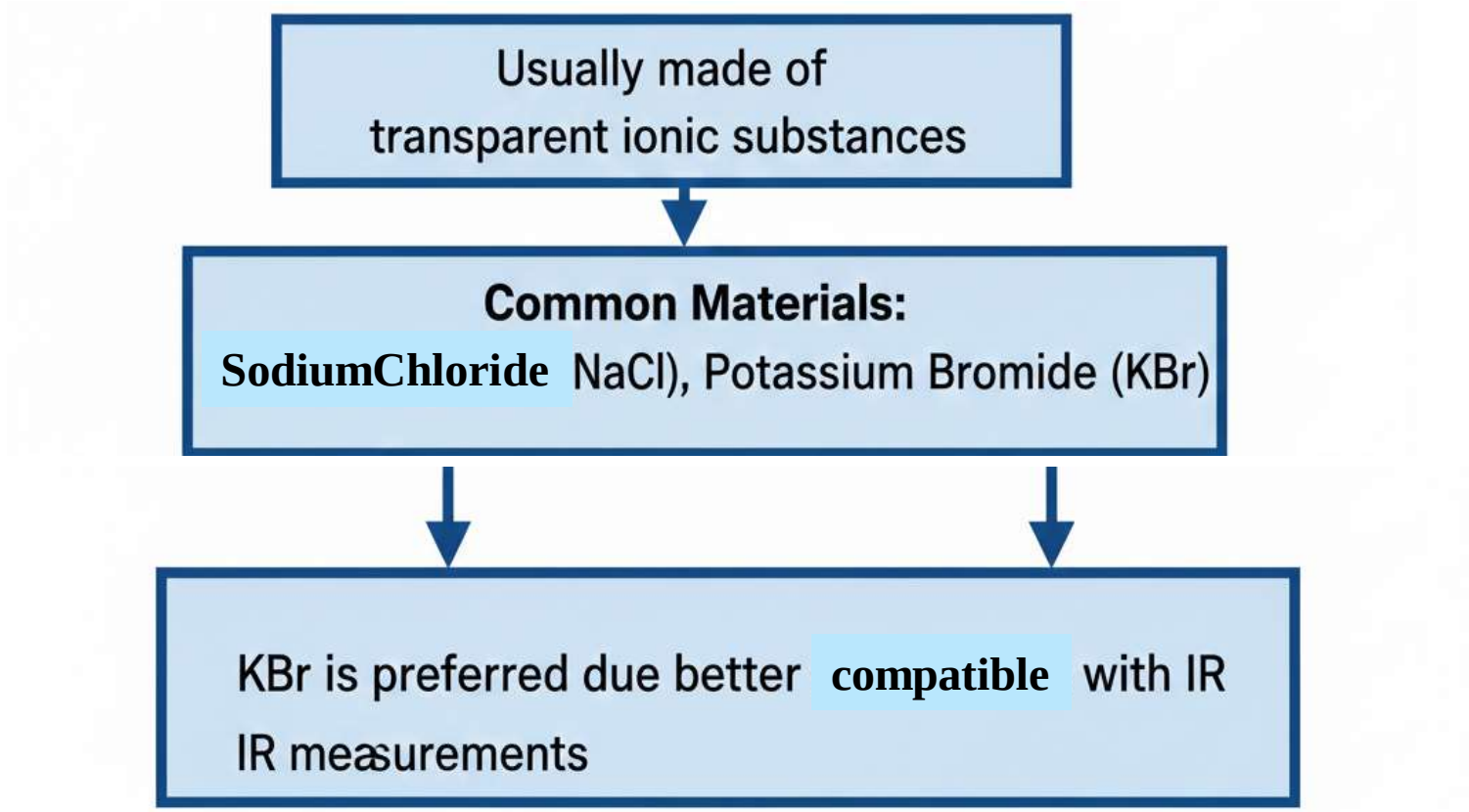

Sample Handling
Liquid Samples
Solid Solutions
Solid Samples
KBr Plates
Cell with Solution
Pressed Pellet Technique
Nuzol-Mull Technique
Instrumental Measurement


Factors Affecting Vibrations
Coupled Vibration
Fermi Resonance
Electronic Effects
Hydrogen Bonding

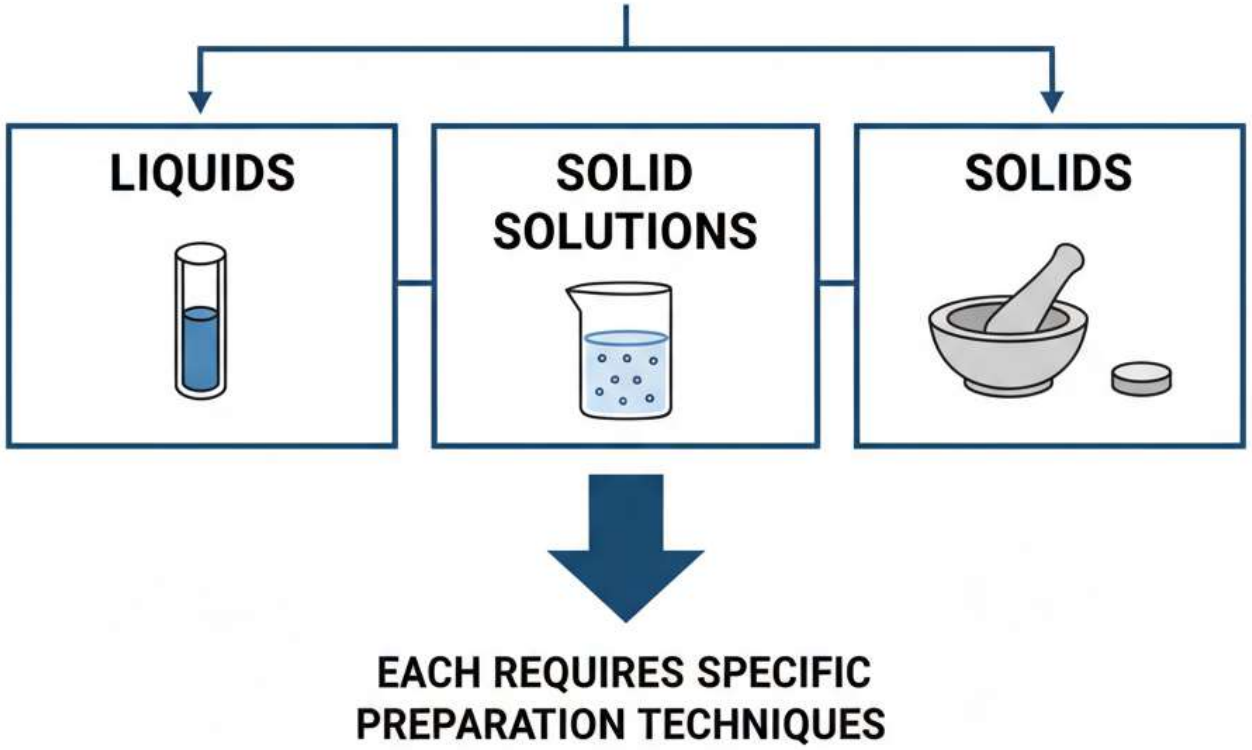
Introduction to Sample Handling in IR Spectroscopy



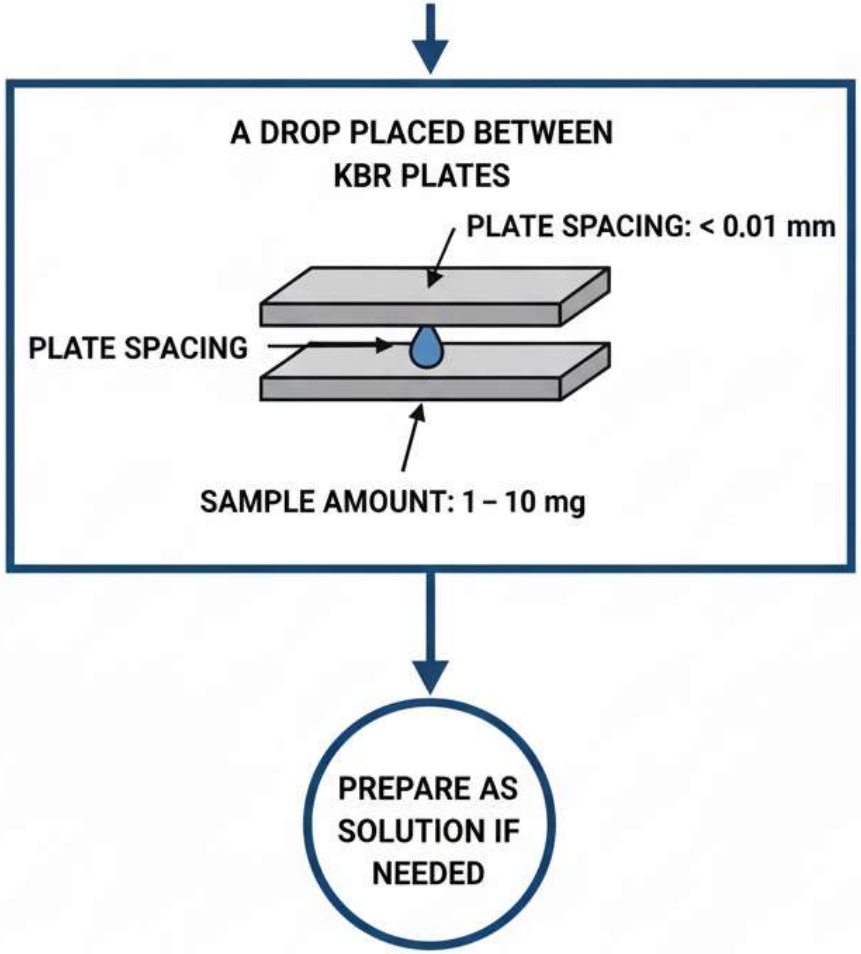
Materials for Sample Cells



Forms of Infrared Samples

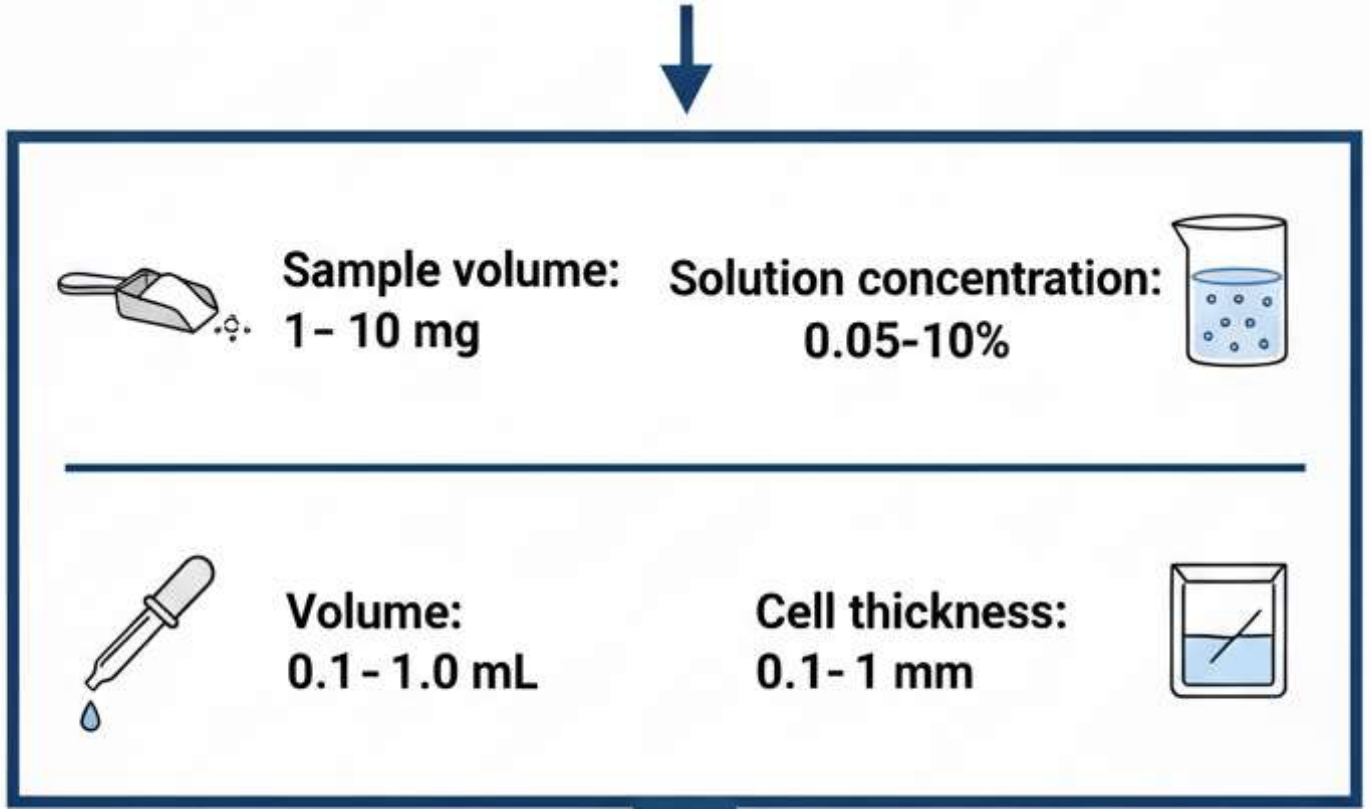


Handling Liquid Samples

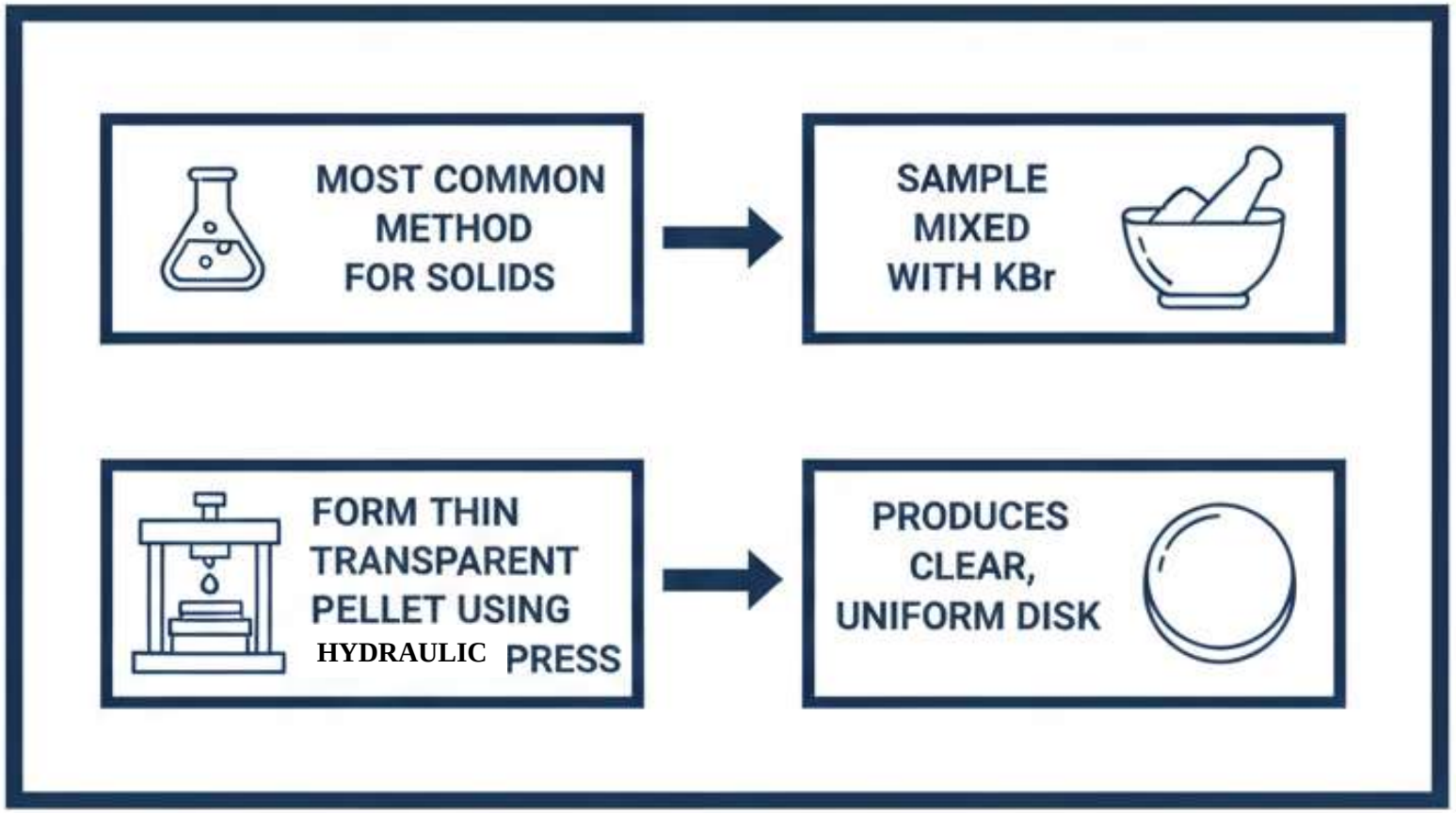


Considerations for Liquid Samples

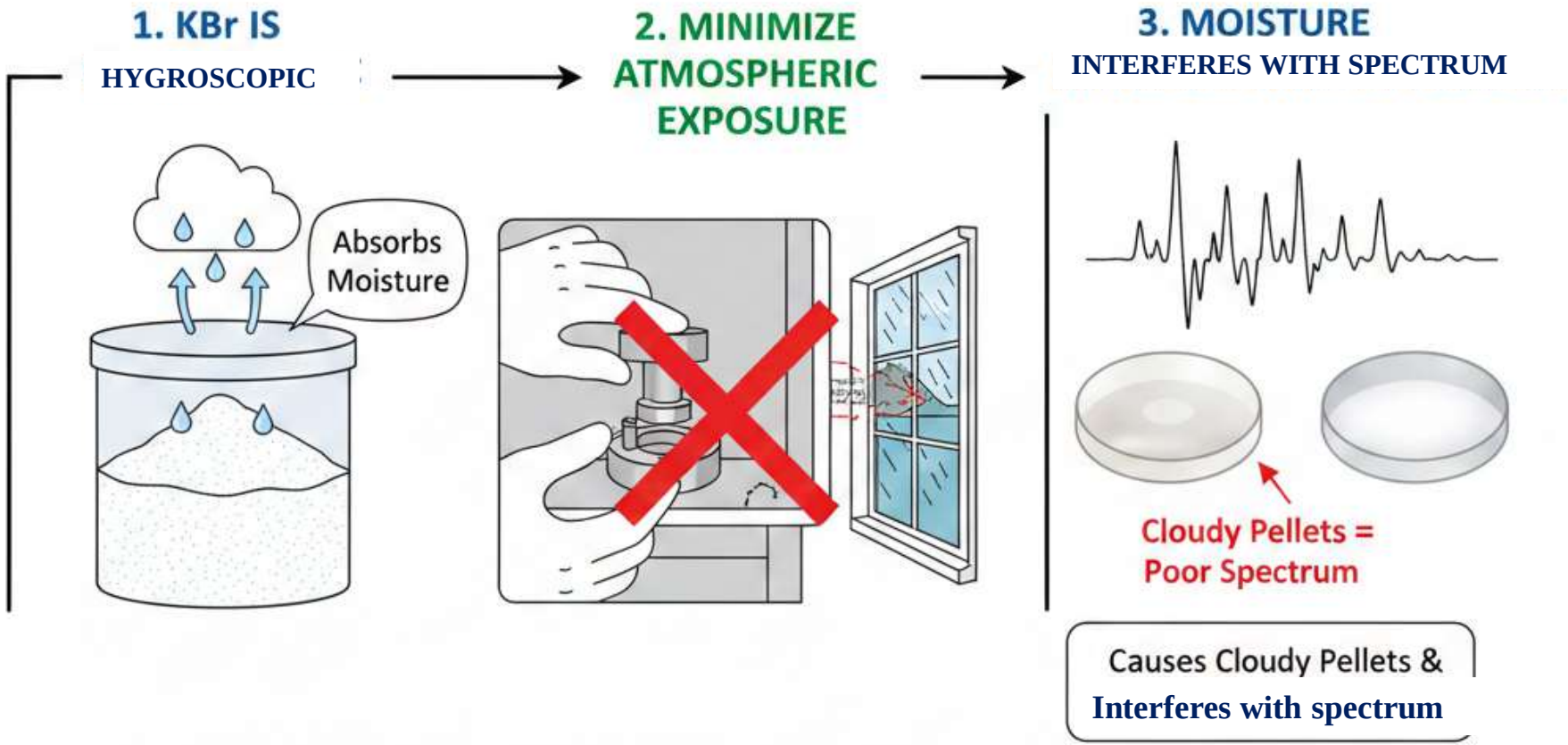
Solid Solutions



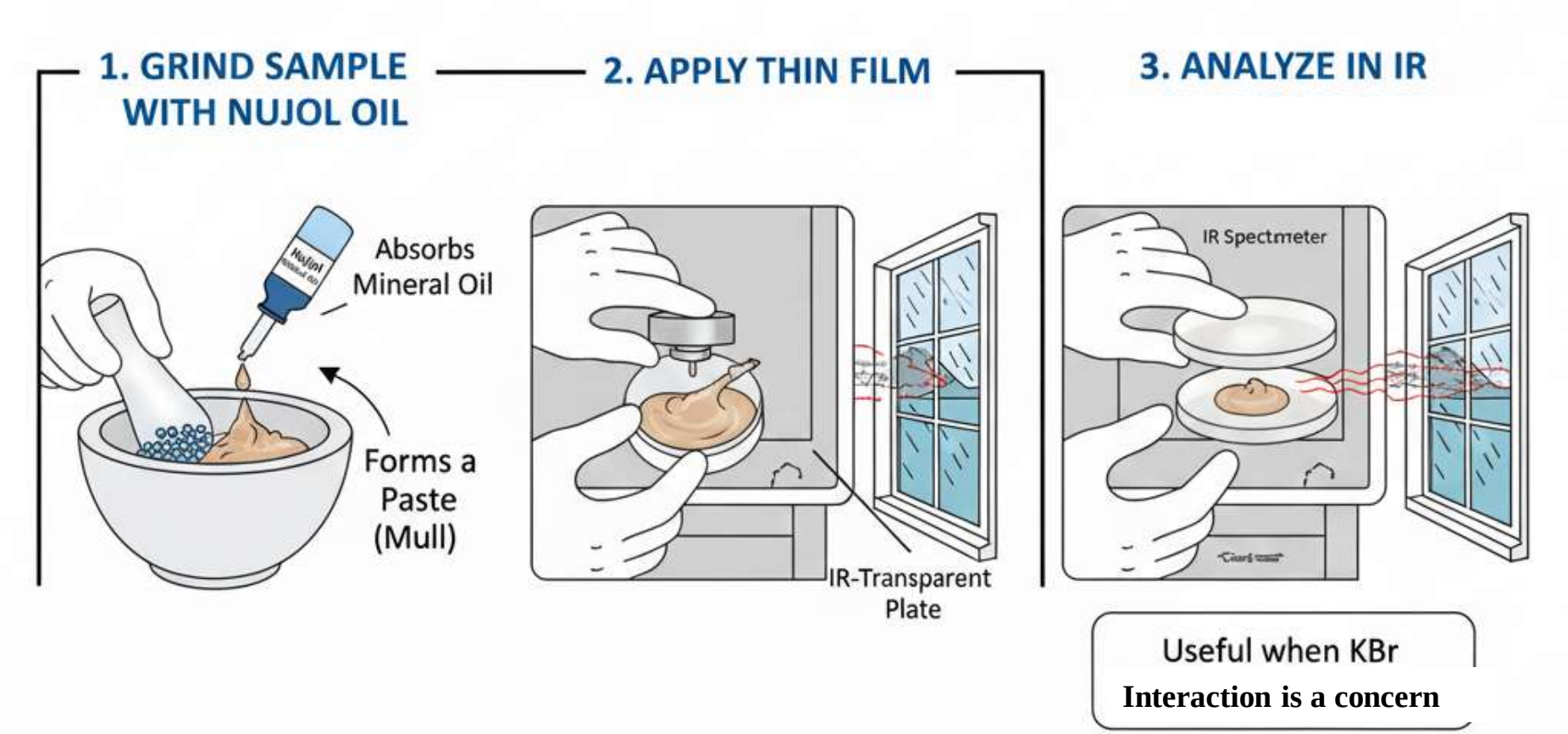
Handling Solid Samples – Pressed Pellet Technique



Precautions with KBr Pellets



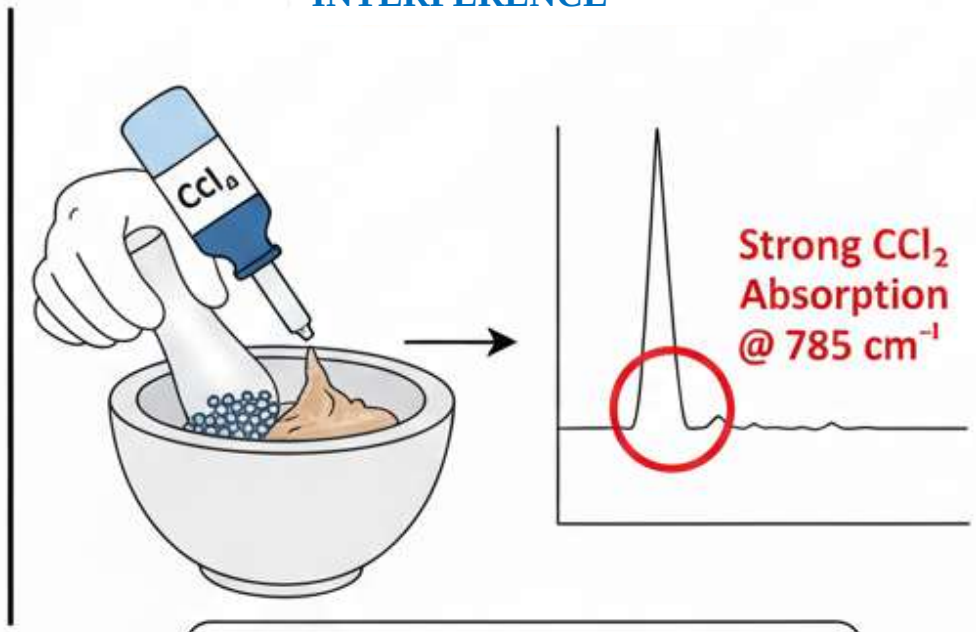
Nujol Mull (Mull Technique)



Caution with Solvents in Mull Technique

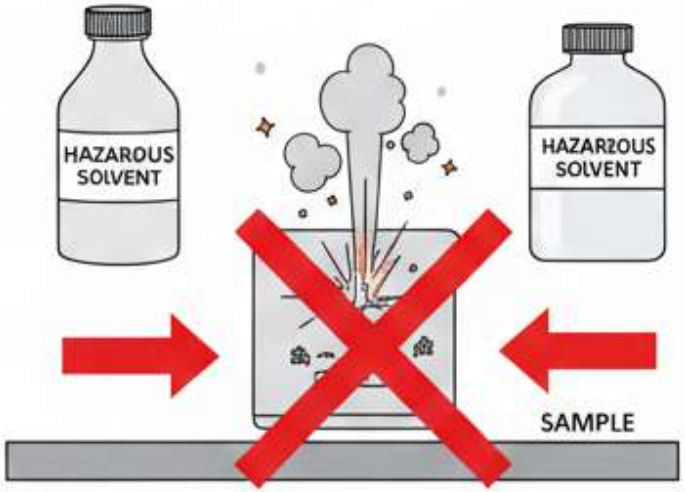
1. CCl_2 ABSORSPTION

INTERFERENCE



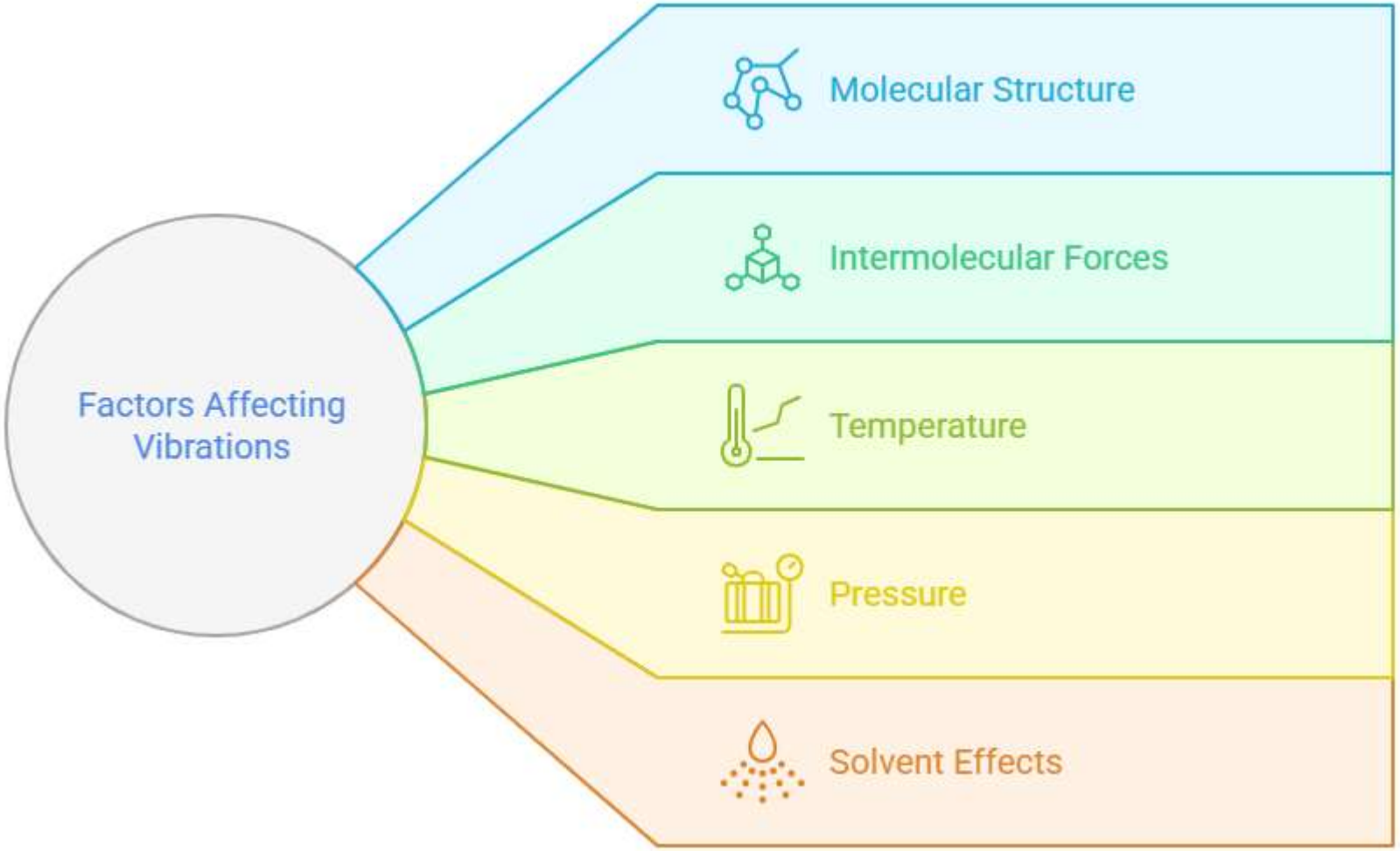
CCl_2 absorbs strongly, **Interfering** with spectrum analysis.

2. AVOID INSTANT REACTIONS



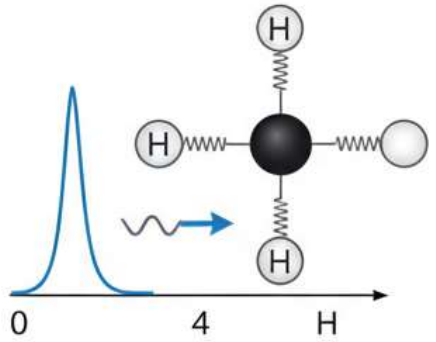
Avoid solvent-sample combinations that react **instantly**

Factors Affecting Vibrations



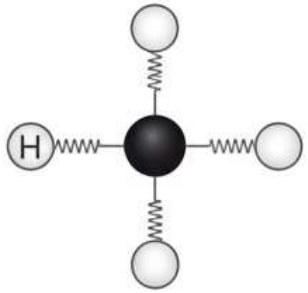
Coupled Vibrations

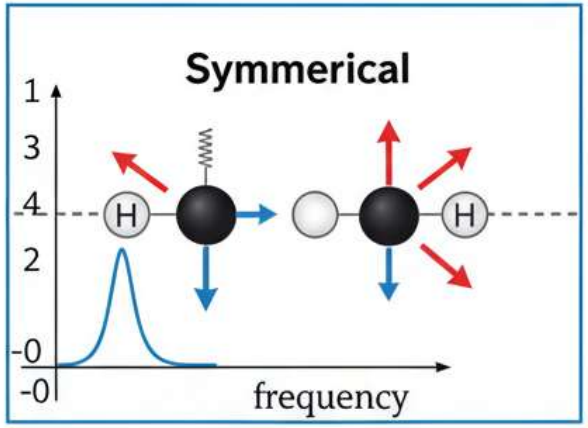
Isolated C–H:

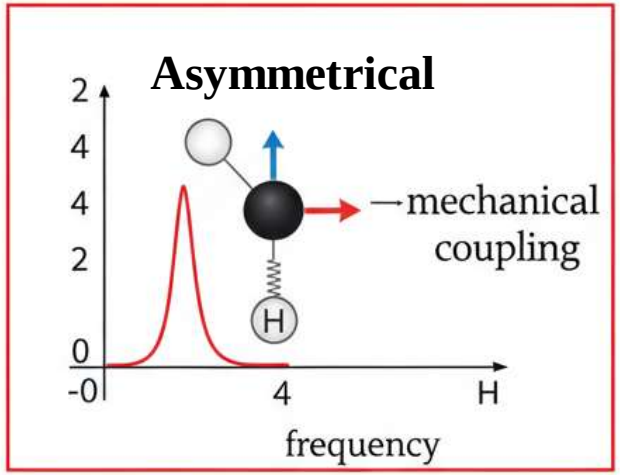


Single strecthing frequency

Methylene group (CH_2-):





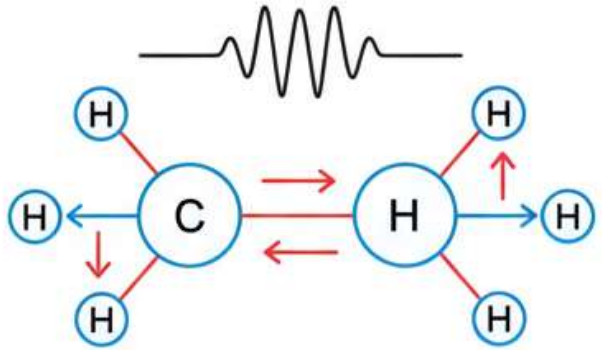


Two streching modes

Due to mechanical coupling

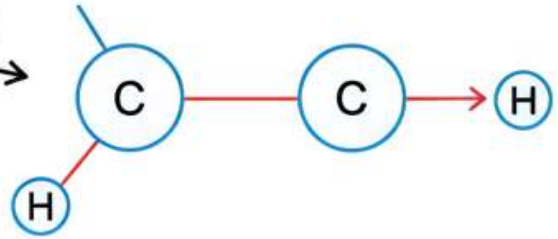
Coupled Vibrations

Asymmetrical Stretching: Higher Frequency



Symmetrical Stretching: Lower Frequency

Isolated C–H Stretch



Occur at different frequencies than isolated C–H

Fermi Resonance

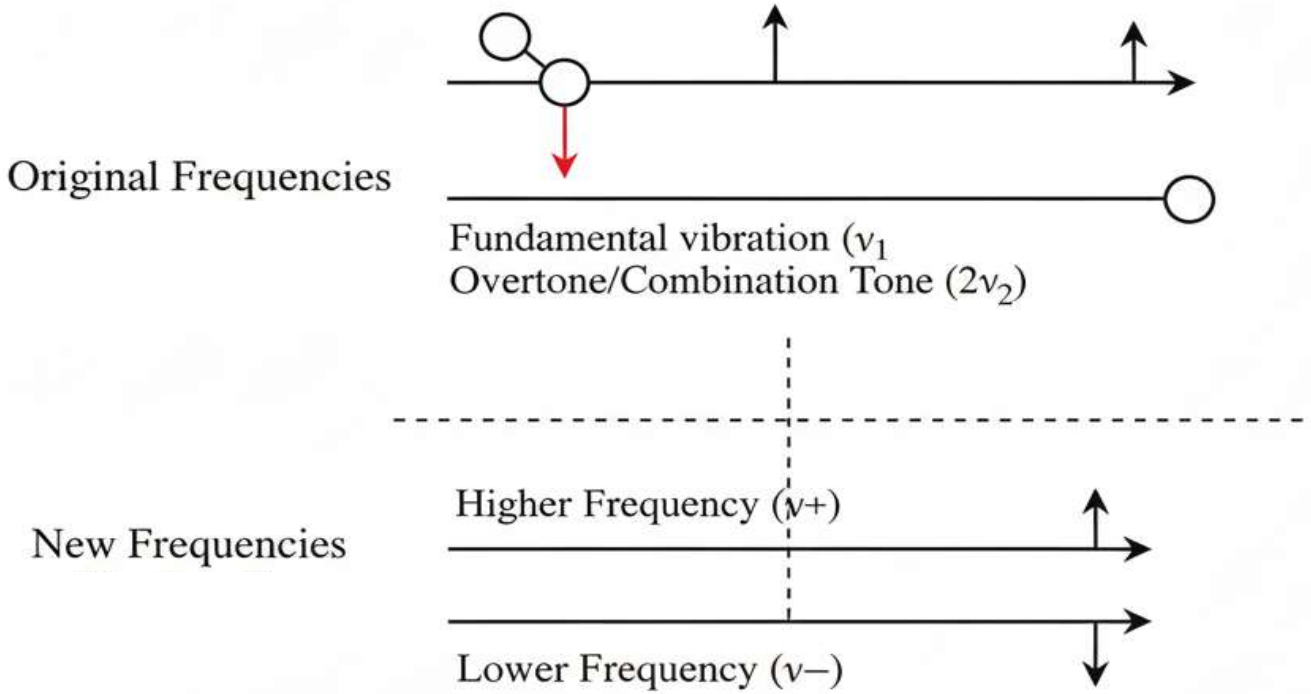
Interaction between:

Fundamental vibration
Overtone or combination tone

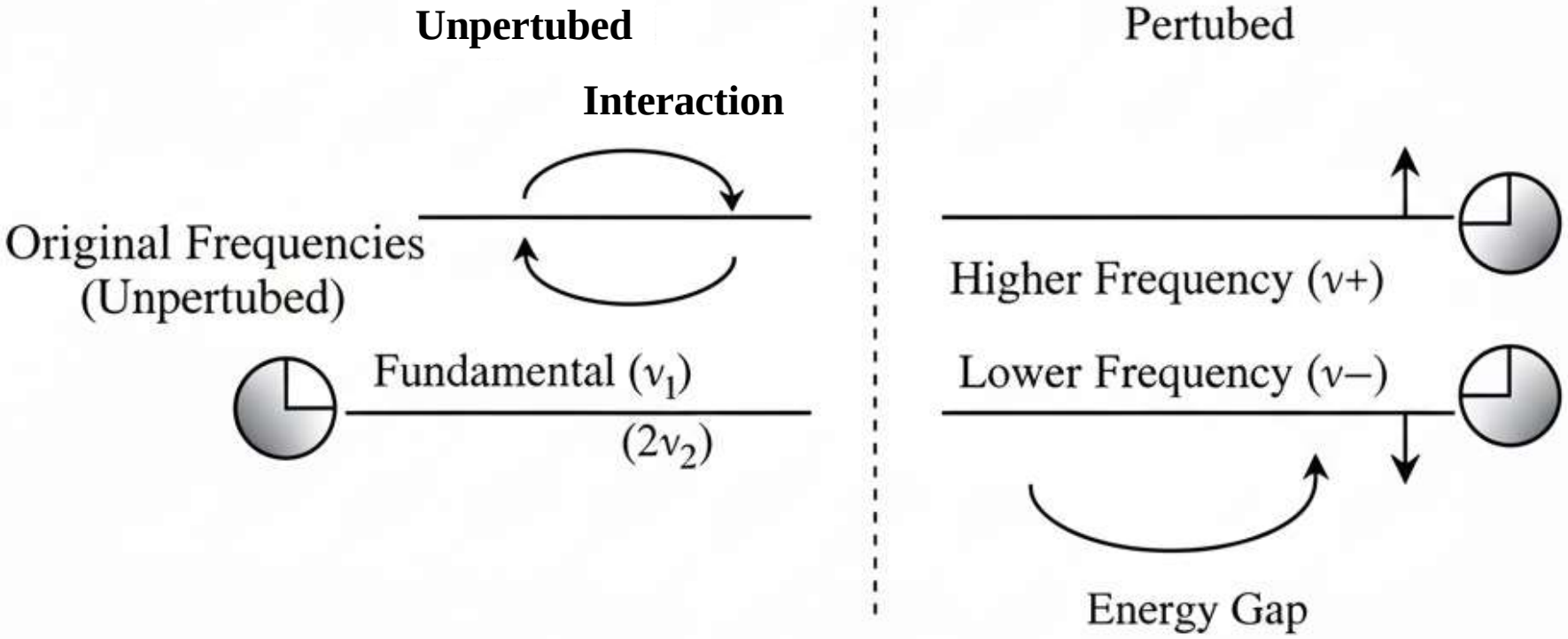
Produces two new vibrational modes

One higher, one lower than original frequency

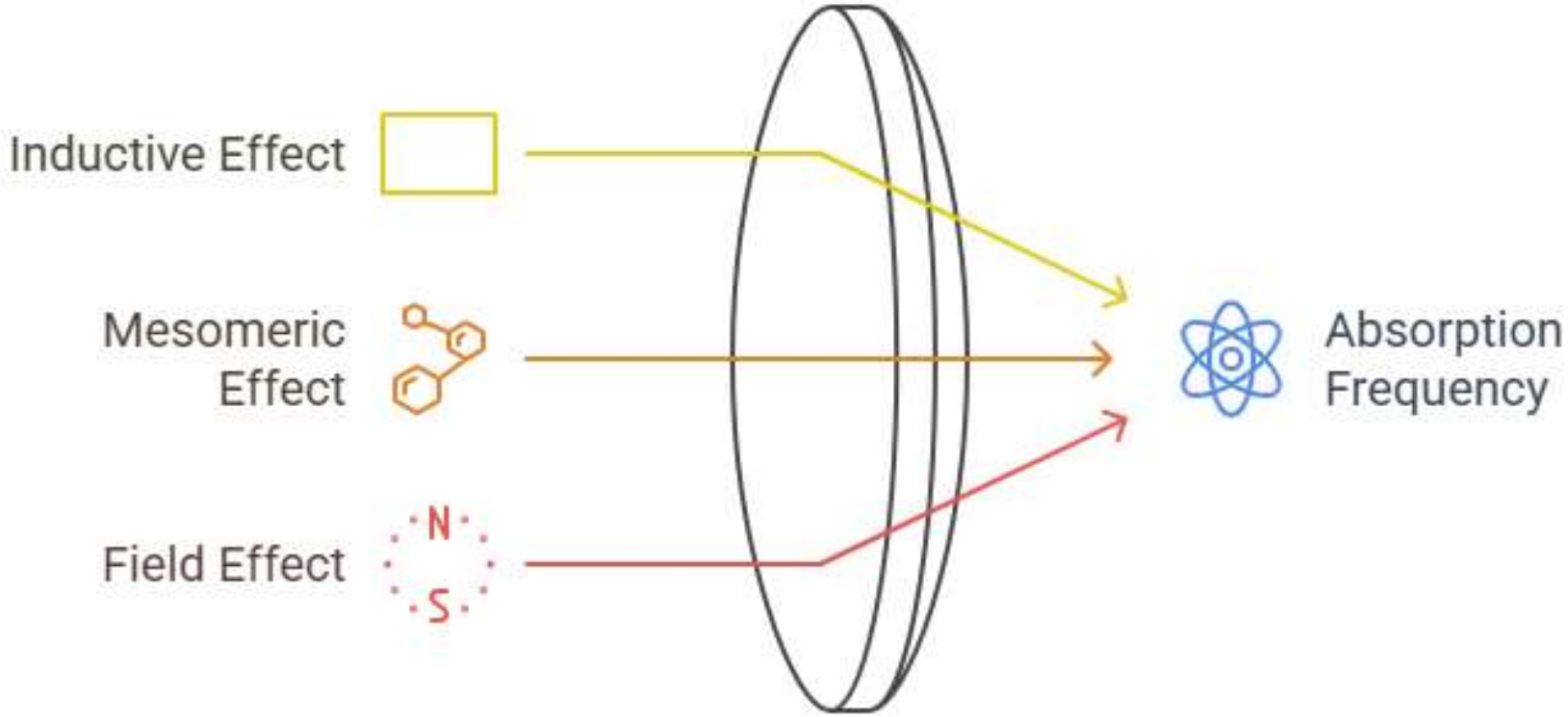
Interaction



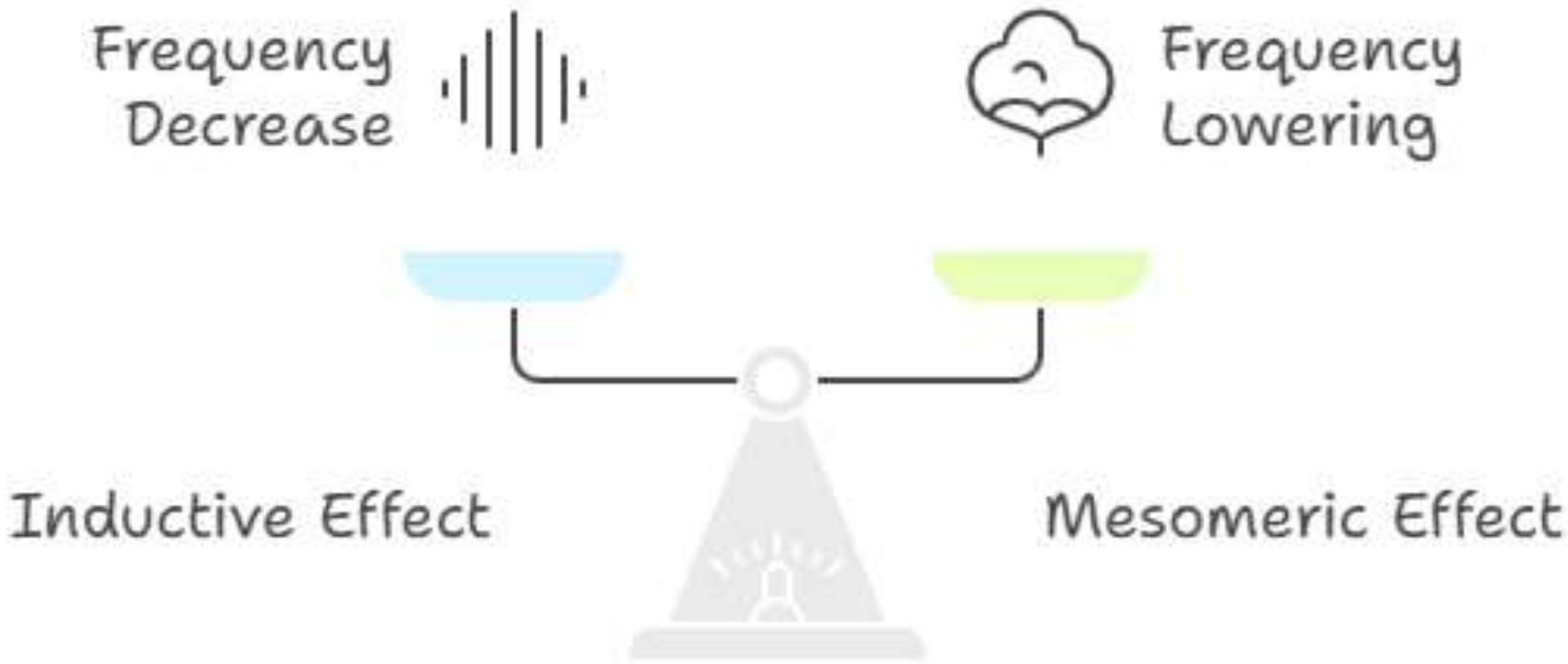
Fermi Resonance



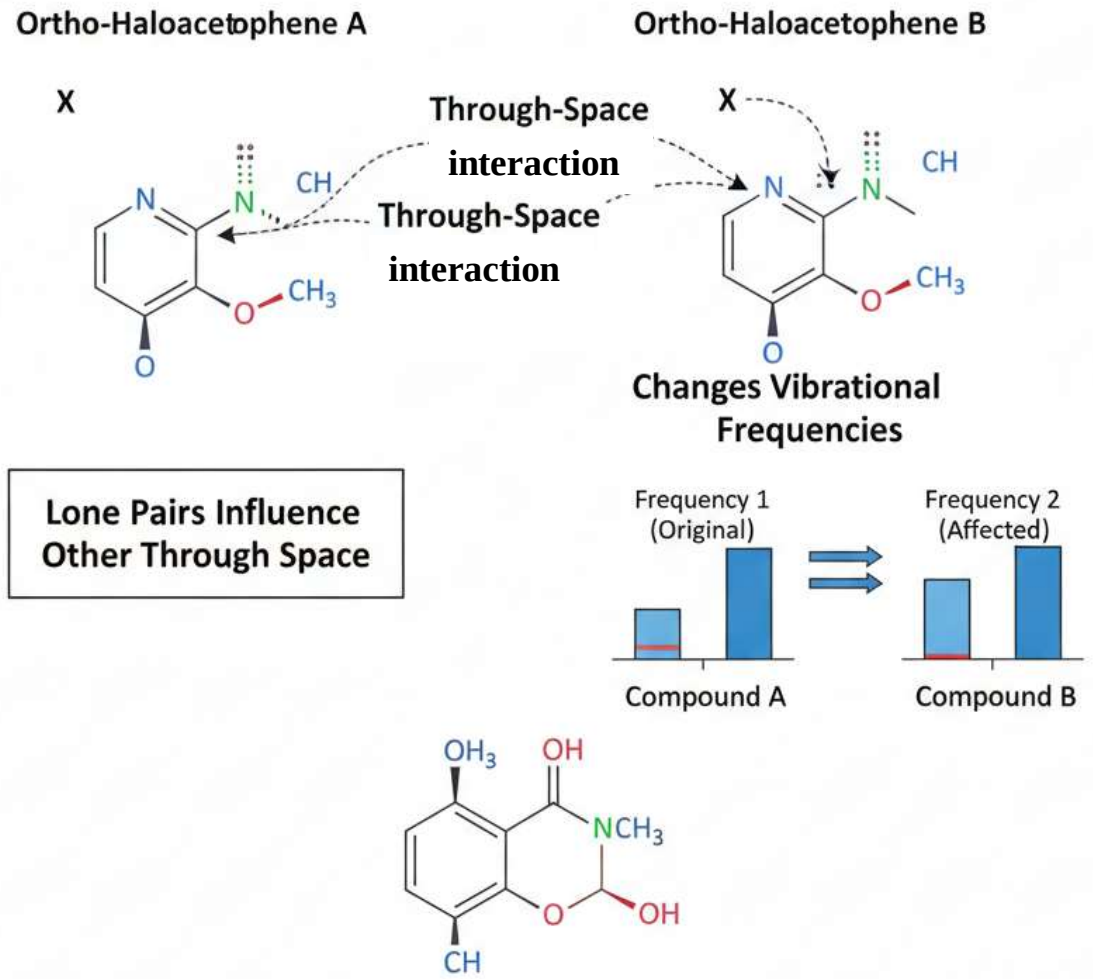
Electronic Effects



Inductive & Mesomeric effect



Field effect

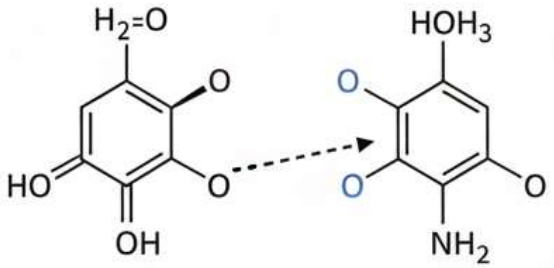


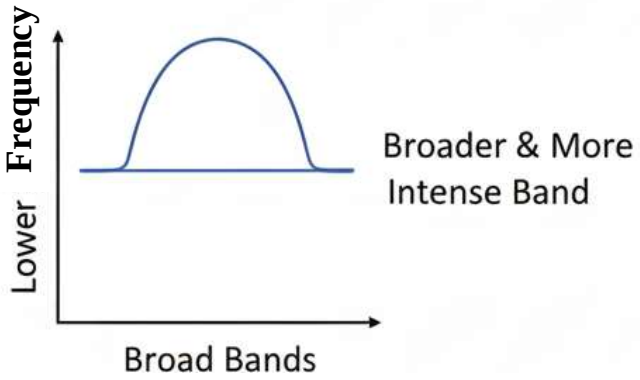
Hydrogen Bonding

Occurs Between Proton Donor and Acceptor

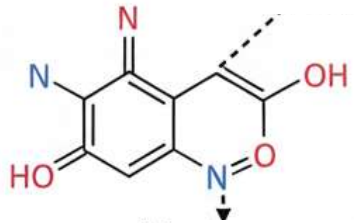
Stronger H-bond → Longer O–H bond

Intermolecular

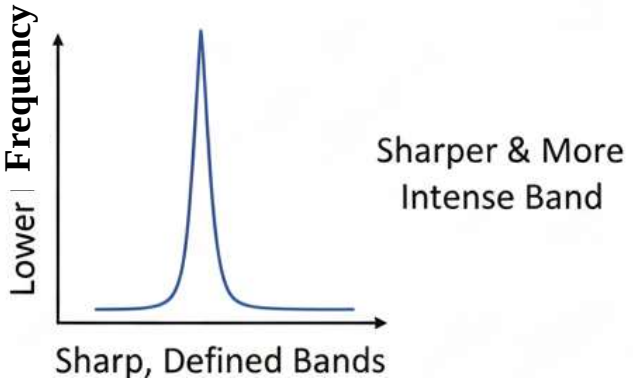




Intramolecular Hydrogen Bonding



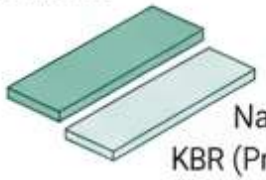
stable



Summary:

SAMPLE HANDLING

- **CELLS**



NaCl
KBR (Prefered)

SAMPLE FORMS

Transparent ionic substances

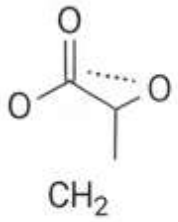
- **Liquids:**
  - KBR plates (<0.01mm)
  - Anhdyrous Organic Solvents (e.g,, CHLC)
  - Avoid H_2O

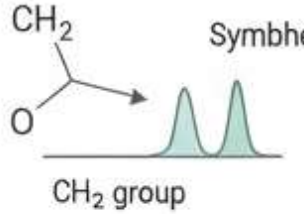
- **Solid Solutions:**
  - 1-10mg solid + CCl_4 (0.1-1ml)
  - 0.05-10% solution

0.05-10% solution
Cell thickness 0.1-1mm

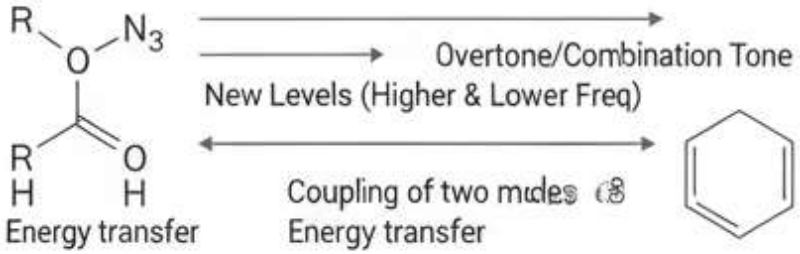
FACTORS AFFECTING VIBRATIONS

- **Coupled Vibration**





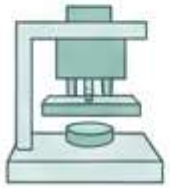
- **Fermi Reseance**



Summary:

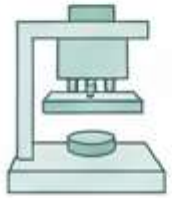
SAMPLE HANDLING

- **Solids**



Pressed Pellet (KBR + Sample)
Thin transparent
Thin film on cell
KBR is hyigoscopic

- **Solids**

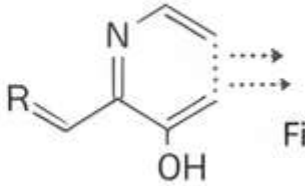


Mull Technique
Thin film on cell)

Mull Technique
(Nujol + Sample)

FACTORS AFFECTING VIBRATIONS

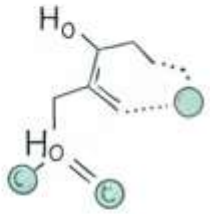
- **Electronic Effects**



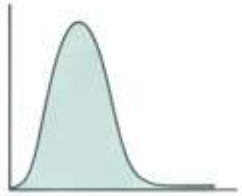
Mesomeric effect = Lower Higher Frq
(Bond Weakning)

Field Effects Effect = Lower Frq H

- **Hydrogen Bonding**



Intermolecular
(Broad Band)
(Sharp Band)



Salicylic acid
Proton Donor + Acceptor
Stronger H-bond = Lower Frey,
Broader, More Intense

Assessment

1. Which solvent should be used for analyzing liquid samples?

Assessment

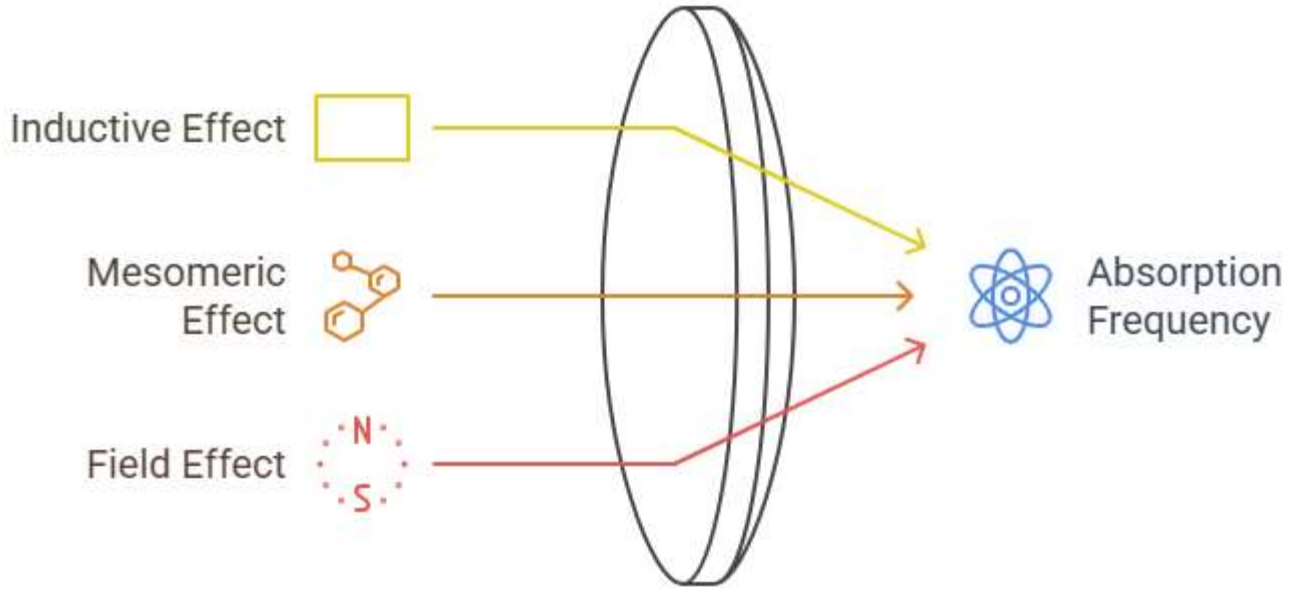
1. Which solvent should be used for analyzing liquid samples?

Assessment

2. In which type of electronic effect the lone pairs influence each other through space?

Assessment

2. In which type of electronic effect the lone pairs influence each other through space?

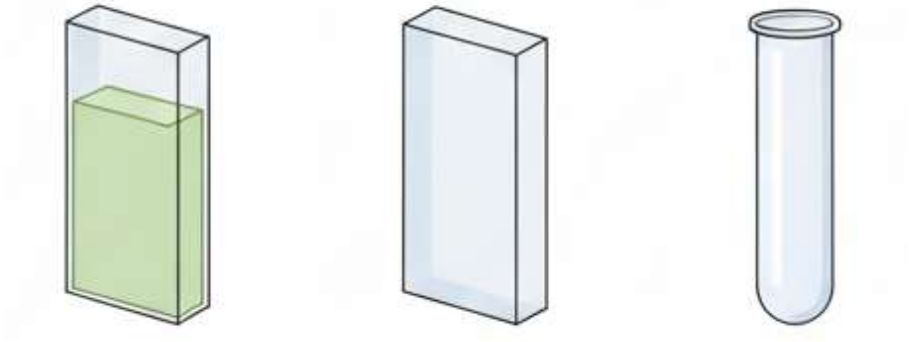


Assessment

3. Which material is preferred for Sample Cells?

Assessment

3. Which material is preferred for Sample Cells?



NaCl **Quartz** **Plastic**

References

1. Pavia DL, Lampman GM, Kriz GS, Vyvyan JR. Introduction to spectroscopy. 5th ed. Boston (MA): Cengage Learning; 2015.
2. Silverstein RM, Webster FX, Kiemle DJ, Bryce DL. Spectrometric identification of organic compounds. 8th ed. Hoboken (NJ): John Wiley & Sons; 2015.
3. Nakanishi K, Solomon PH. Infrared absorption spectroscopy. 2nd ed. San Francisco (CA): Holden-Day; 1977.
4. Colthup NB, Daly LH, Wiberley SE. Introduction to infrared and Raman spectroscopy. 3rd ed. San Diego (CA): Academic Press; 1990.
5. Banwell CN, McCash EM. Fundamentals of molecular spectroscopy. 4th ed. London: McGraw-Hill; 1994.


Thank you!