Massachusetts
Institute of
Technology

CHEMISTRY

5.35

Sample Preparation Considerations and Issues

Choice of Sampling Technique

- State of analyte
- Material compatibility
- IR absorbance of cell materials
- Path length

Warning Signs for Poor Samples

- Big/wide water signal
- No features or only Nujol/PE/etc
- Broad and non-distinct signals
- Sloping base line
- Misaligned positioning

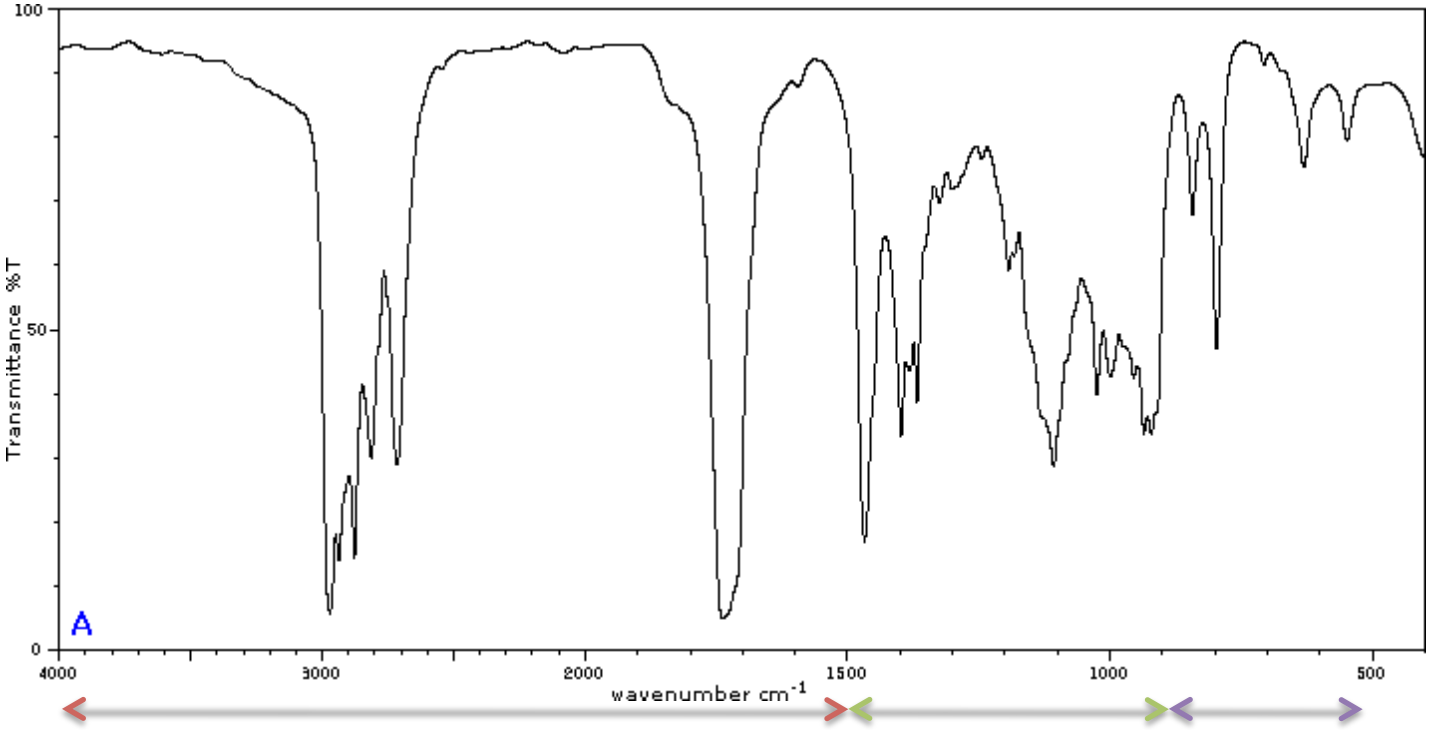
Sample Preparation Caveats

- IR signal scatter – dirt, scratches, cracks
- Too concentrated/dilute
- Opaque pellets
- Too thick
- Too thin (fragile)
- Wet KBr
- Wet sample
- Reproducible preparation
- Sample is not what you think it is

Warning Signs of Instrument Failure

- Unresponsive (hardware/software)
- Error messages
- No instrument/computer/printer comm
- Laser weak or dead
- CCD or photodetector is dead
- Bad settings from previous user(s)

IR Spectroscopy – Spectra Interpretation



IR Spectroscopy – Spectra Interpretation

Challenges

- Ambiguous peaks
- Misinterpreting overtone, combination vibrations
- Misinterpreting impurities
- Poor reproducibility of sample preparation – artifacts

Important Tools / Strategies

- knowledge of chemistry, reactants, solvents
- systematic peak assignment (e.g. Mohrig)
- complementary characterization (NMR, UV-Vis, EA)
- peak shape/intensity, shoulders
- computerized databases

5.35 / 5.35U Introduction to Experimental Chemistry

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