

University of Kansas Solid-State NMR Facility

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Facility Instrumentation

- Bruker Avance III three-channel spectrometer
 - 1 kilowatt amp on both primary channels
 - ½ kilowatt amp on tertiary channel
- 9.4 Tesla magnet (400 MHz ^1H)
- Variable temperature capable

Facility Instrumentation

- Revolution NMR Multiple Sample Solids Probe
 - Four independent CP/MAS circuits:
 - Two 7 mm, two-channel (HX)
 - One 2.5 mm, two-channel (HX)
 - One 4 mm, three channel (HXY)
- Bruker 4 mm CP/MAS two-channel probe

Facility Director Experience

- Pharmaceuticals
 - Physical form identification
 - Crystalline Polymorphs, Solvates, Amorphous
 - Quantitation of physical forms in formulations
 - Characterization of stability samples
- Crystalline systems
 - Molecular Conformation in Unknown Crystal Structures
 - Complement X-ray structures
 - Identify Z'
 - Complement Rietveld Refinement
 - Molecular Dynamics
- Catalysis
 - Catalyst characterization
 - In situ reactivity

Advantages of Solid-State NMR

- Non-destructive
 - Sample can be returned
- Non-invasive
 - Samples can be studied without being perturbed (e.g., tablets)
- Selective
 - Nucleus specific (^{13}C , ^{15}N , ^{31}P , ^1H , ^{19}F)
 - Sample content
 - Mixture components
- Quantitative
 - Can measure relative concentration of physical forms without calibration curves

Research Contributions

- Characterization of materials in solid state
 - Crystal vs. amorphous
 - Influence of particle size, crystal quality, bulk environment
 - Lot-to-lot variation
 - Can isolate components of mixtures (e.g., drug and excipient)
- Molecular Structure
 - Conformation, arrangement, degree of disorder
- Dynamics
 - Lattice mobility, glass transition (T_g)

Previous Facility Contributions

- Pharmaceuticals
- Soil Science
- Biomass
- Catalysis
- Organometallic
- Biological
- Inorganic

What Can You Learn ?

- Chemical shifts:
 - Functional groups, fingerprint of sample components
- Peak analysis
 - Amount of components present
- Evaluate differences between sample
 - Impact of different exposures
 - Lot-to-lot variations