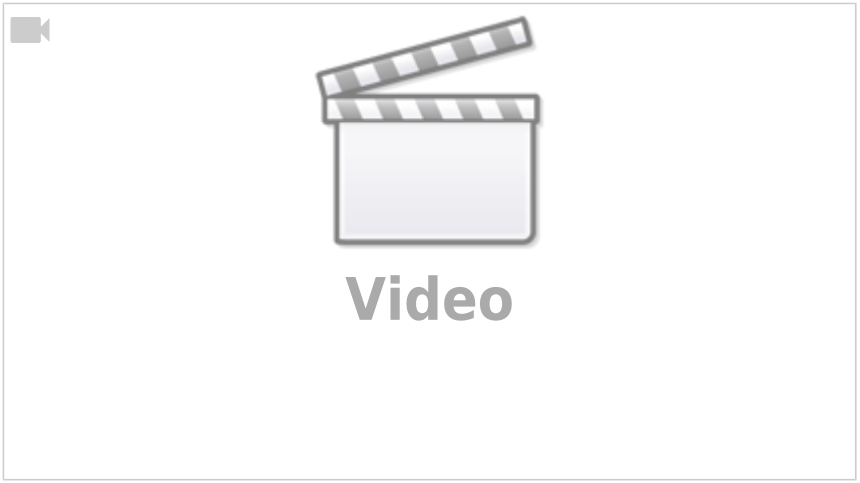
Notre Dame NMR Operation and Concepts video series

This is a series of video tutorials created for training of NMR users at the University of Notre Dame. The series is focused on using Bruker NMR spectrometers with broadband probes. It is required that all tutorials are viewed in sequential order because the later tutorials refer to the material discussed in earlier ones without repeated explanations.

Episode 1. Introduction to Notre Dame NMR Operation and Concepts series

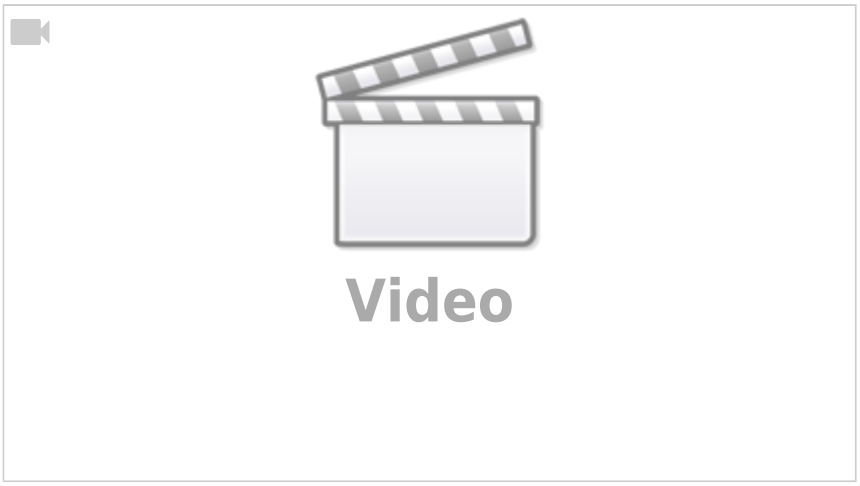


Summary

- http://nmr.nd.edu - main source of your information and training materials
- Recommended books:
  - **High-Resolution NMR Techniques in Organic Chemistry** by Timothy Claridge
  - **Fundamentals of Protein NMR Spectroscopy** by Gordon Rule and Kevin Hitchens
  - **Protein NMR Spectroscopy: Principles and Practice** by John Cavanaugh, Wayne Fairbrother, Arthur Palmer III, Mark Rance, Nicholas Skelton

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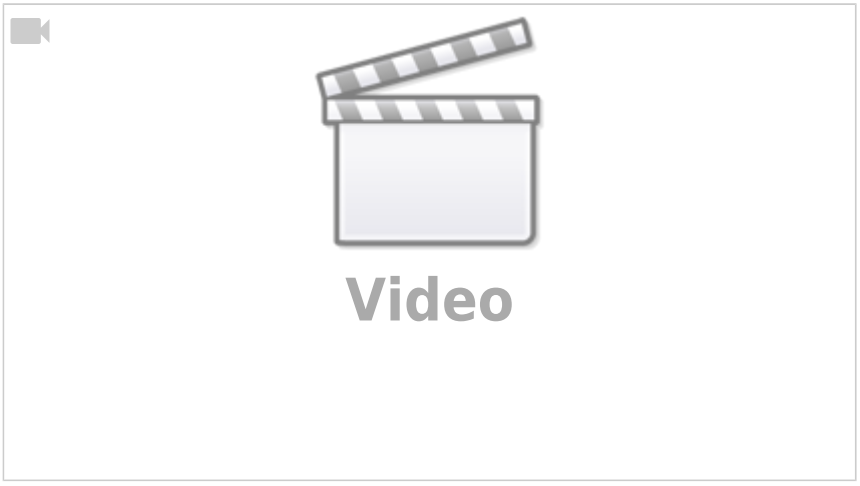
Episode 2. NMR Probe Tuning



To tune the NMR probe:

- Decide which heteronucleus you will need
- Display experiment where it is shown in Nucleus section (along with proton)
- Submit 'ro off', 'atma', and 'wobb' to Spooler: qu ro off; qu atma; qu wobb
- Wait till atma quits and wobb displays a wobble curve
- Inspect the wobble curve for both channels
- Proceed to locking, shimming and NMR acquisition
- If you need to work with another heteronucleus next - you must tune the broadband channel again!
- Please, tune the broadband channel back to carbon after you finished your work.

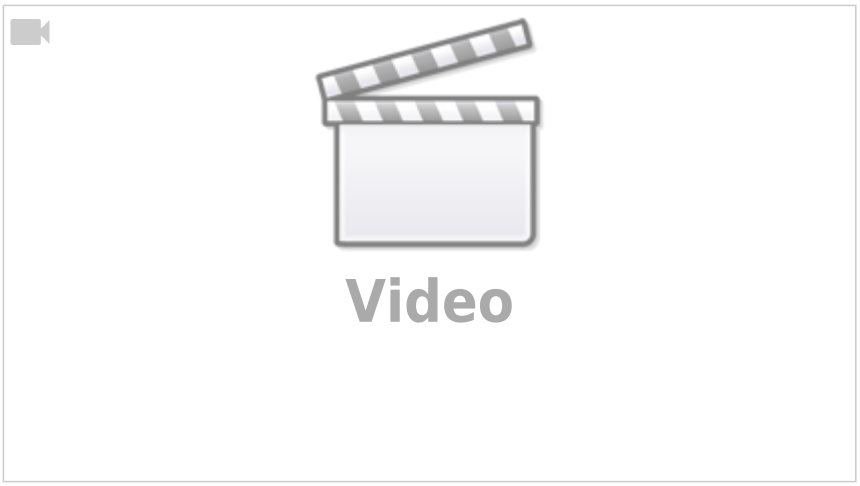
Episode 3. NMR Spectrometer Locking



To lock the NMR spectrometer:

- Confirm that your sample contains >10% of deuterated solvent
- Load a standard shimset ('bbfo-latest' on McCourtney 500 and Stepan 400)
- Click [Acquire: Lock] or type 'lock' on a command line
- Select your solvent from the lock table
- Wait for the lock system to finish (watch for a steady oscillating trace and a message 'lockn: finished' in the status bar)

Episode 4. Shim System

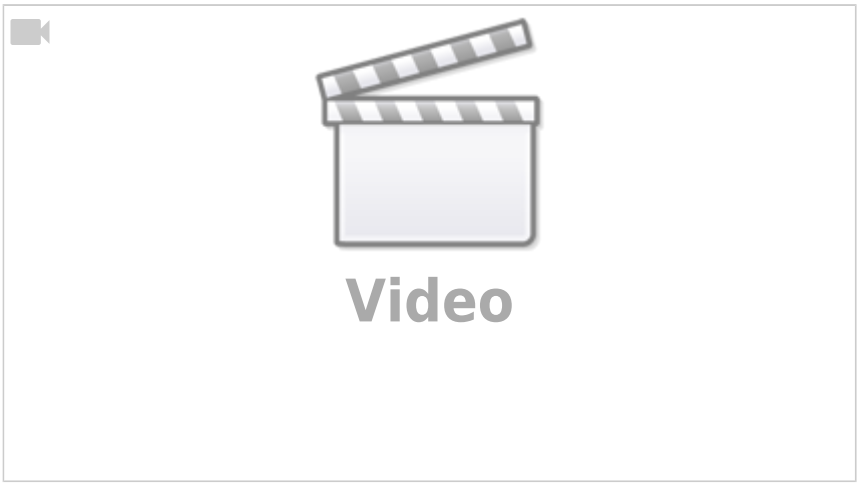


Summary:

- NMR magnet needs shimming to achieve high resolution
- Majority of the magnetic field distortion is taken care of by loading a standard shimset
- Each and every sample must be shimmed to make the magnetic field perfect before the NMR acquisition
- NMR samples must be at least 40 mm long to allow for shimming

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Episode 5. Shimming Workflow

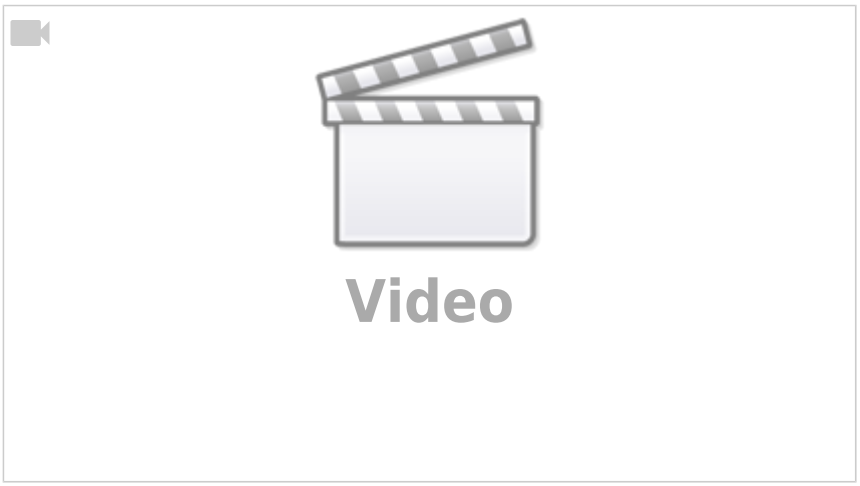


In summary, to shim the sample:

1. Tune the probe and load a standard shimset
2. Lock on your solvent
3. Start sample rotation
4. Launch 'topshim gui' and verify the nucleus “To optimize for”
5. Start shimming (click Start and open Report tab)

- If the “Final B0 stddev” > 1 Hz, repeat shimming (click Start again)
- “Final B0 stddev” < 1 Hz means that Topshim found optimal shims
- Record a proton 1D to assess real spectral resolution (discussed in next episodes)

Episode 6. Shimming problems caused by the samples



For success of your shimming, please, follow these simple rules

- Buy NMR tubes rated for your field or higher

- Make sure your samples are soluble, homogeneous, and don't have dissolved gases
- Load enough to make 40mm-tall solvent column
- Use depth gauge to set the tube in the spinner to a correct depth
- Test the standard sample if your sample does not shim

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