Protocol for X-nuclei NMR at AM400 (AV III HD 400 NanoBay)

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Book time and log in computer as usual

Routine **X-nuclei** with H-1 decoupling or without decoupling
(X#: X - isotope symbol; # - isotope number)

Procedure:
Open Topspin 3.6.1

run H-1 NMR prior to X-nuclei NMR (Ah1): follow the basic procedure from your basic H1 training.
run X-nuclei NMR with H-1 decoupling or without decoupling

A. Under "Start",
1. create dataset(**new** or **edc**): name; exp #; pro #; solvent; directory (D:\group name\user name\yrmo)
2. (**rpar Ax#*)** (select Ax#cpd or Ax#: cpd – decoupling; no cpd – without decoupling)

B. Under "Acquire",
3. no eject sample/no insert sample
4. no lock
5. tune (**atma**)
6. no spinning
7. no shim
8. prosol (**getprosol**)
9. gain (**rga**)
10. (**ns**), (**ds**), (**d1**): d1 is relaxation time in seconds; ds is dummy scans for warming up the spin system; ns is number of scans. In general, X-nuclei needs large number of scans.
11. (**zg**): zeros (overwrites) current data set and starts acquisition
12. (**tr**): saves the data of the current number of scans while the experiment is still running
13. (**efp;apk**): process the data
14. If the data is enough, (**halt**) stops the acquisition run and saves the data; if the data is not enough, keep running until the ns finishes. If the data is still not enough until the ns finishes, type new (**ns**) and (**go**): restarts the acquisition and appends the new data to the current data set.

C. Under "Process",
15. adjust phase if necessary
16. calibrate spectrum
17. pick peaks
18. integrate
19. advanced if necessary

D. Under "Analyse"
20. eject sample (**ej**); take sample; (**ij**) to turn off air
21. close topspin
22. log off
23. **put cap back on magnet**

Remark:

" lock and shim well
" tune probe
" no spinning
" no pulsecal in automation
" optimize offset (**o1p**) and spectral width (**SW**) for advanced applications