IMPORTANT

Experiment Guides

Experiment Guides are brief, step-by-step procedures for operating the Eft spectrometer. The Guides serve both as learning tools for the new user and as quick reference tools for the experienced user.

Sample Preparation Guide

The quality of results on the Eft NMR spectrometer is critically dependent on using appropriate sample preparation methods. For all samples:

1. Use a total sample solution volume of 0.7ml (~ **1 inch** liquid height).
2. If the sample contains particulate matter, ***filter*** the solution through glass wool or cotton that is loosely packed in a Pasteur pipette.

^{1}H Samples

1. Sample concentration of about 5% or about 0.3M is generally appropriate.
2. TMS or HMDS at 0.5% is sufficient to reference a 0.3M solution.

^{13}C Samples

1. Neat (meaning 100% concentration) liquids will give good spectra in 1 minute and 1M samples will allow good spectra to be obtained in 5 minutes.
2. TMS at 2% should be adequate to reference a 1M solution.

Sample Spinners

All Eft Spectrometers (60 and 90MHz) with ^{13}C or broadband observe capabilities use an Aii probe and the Aii sample spinner.

THE POSITION OF THE SPINNER IS VERY IMPORTANT AND MUST BE SET TO THE APPRORIATE POSITION WITH THE DEPTH GAUGE BEFORE SAMPLE IS INSERTED IN THE PROBE.

1. Write
   a) your name
   b) type of experiment (^{1}H or ^{13}C or HETCOR or COSY)
   c) date
   d) time in the log book.
2. Eject the reference sample using the EJECT button inside the lid of the probe cover (left side).
3. Take the spinner off the reference.
4. Place the spinner on your NMR tube.
5. Position the spinner to the appropriate depth using the depth gauge.
6. Wipe the NMR tube with clean tissue (kimwipe).
7. Place the NMR tube with your sample in it into the probe.
8. Turn the spinner off (using the toggle switch) and back on.

Instrument Setup Guide

Unless otherwise instructed, this section will be completed by the instructor!

Step	Function or Dialog Box	<Keystroke>/[Select]/<Data	Comment
A	**PREPARE**		Purpose: adjust field and optimize shims. Run if instrument idle > week otherwise skip to B.
1	Sample		Tap water, spinning. (Do not use the sealed Homo Adj. sample supplied with original CW
2	Enter PNMR.	<Alt+Tab>	(If necessary.)
3	Acquire data.	**Hl**>prep<Enter>	Spectrometer enters GS mode.
4	Gain Adjusted		The spectrometer adjusts the receiver gain.
5	Spectrum acquisition.		Prep takes a spectrum
6	Enter NUTS and process data.	<Alt+Tab> >a2	Trim phase as required. Use cursor to determine peak position in ppm, including sign.
7	Enter PNMR and set field for shimming.	<Alt+Tab> value<Enter>	Change to PNMR and enter the peak position in the User Input box.
8	Adjust spinning speed.	Adjust spin air flow <Ctrl+Q>	Adjust spinning speed so the crest-to-crest spacing in the FID is about 5 horizontal divisions.
9	Minimize side bands.		Y, X, Z, etc shims optimized.
10	Adjust spinning speed.	Adjust spin air flow <Ctrl+Q>	Adjust spinning speed so the crest-to-crest spacing in the FID is about 2 horizontal divisions.
11	Optimize resolution.		Spinning shims set, spectrum acquired.
12	Enter NUTS and process data.	<Alt+Tab> >a2	(Trim phase as required.) Use cursor to determine peak position in ppm, including sign.
13	Enter PNMR and set field.	<Alt+Tab> vaiue<Enter>	Change to PNMR and enter the peak position in the User Input box.
B	SHIM THE MAGNET		Purpose: Optimize resolution.
1	Sample		5% ethylbenzene or 5% ethylmethacrylate
2	Enter PNMR.	<Alt+Tab>	(If necessary.)
3	Enter shim routine.	Hl>shim<Enter> or C13>shim<Enter>	The spectrometer automatically adjusts the gain before shimming.
4	Enter RD value	value<Enter>	Dilute sample RD=5; Cone, sample RD=2
5	Shim		Allow time to shim.
C	**TMS REFERENCE**		**Purpose: Correctly position the spectrum.**
1	Sample		5% ethylbenzene or 5% ethylmethacrylate
2	Enter PNMR.	<Alt+Tab>	(If necessary.)
3	Verify parameters.		Verify that the parameters make sense. Otherwise Hl>re hi . ini<Enter>
4	Acquire data.	Hl>zg<Enter> <Enter> to use the default file name	If FID display is red, reduce RG, and repeat zg. Proceed to next step when prompted.
5	Enter NUTS and process data	<Alt+Tab> >a2	(Trim phase as required.) Use cursor to determine TMS peak position in ppm, including sign.
6	Enter PNMR and enter TMS peak position.	<Alt+Tab> Hl>fo<Enter> va_Zue<Enter> 0<Enter>	Switch back to PNMR, execute the fo command, enter TMS position determined above for current position, and 0 (zero) for desired position. Repeat to confirm.
D	PHASE CHECK ^{1}H		If Step 5 above does not give spectrum with correct phase, see "Update Phase Correction Parameters" in the Appendix.

^{1}H Survey Spectra Guide

Step	Function or Dialog Box	<Keystroke>/[Select]/<Data	Comment
1	Sample		See Sample Preparation Guide. Position sample spinner using the depth gauge, place in probe
2	Enter PNMR program.	<Alt +Tab>	(If necessary)
3	**Select ^{1}H observe.**	C13>nu H1<Enter>	Required only if the prompt is not "H1".
4	Verify parameters.		Verify that parameters make sense.
5	**Acquire data.**	Hl>zg<Enter> then <Enter> for default or [file name]<Enter>	Enter file name if desired but it is usually 'better to use the default (pnmr fid) unless intending to save the data long term.
6	Enter NUTS.	<Alt+Tab>	
7	**Process data.**	<a2>	Processes FID with FT.
8	Enter zoom routine.	>zo	Set up for phasing.
9	Select two regions of interest, (see comment)	<1> then <2> <Enter> to exit "zo"	Drag cursor over a strong peak on left. Press <1> to assign as region 1 . Drag cursor over a strong peak on the right and press <2> to assign as region 2.
10	Trim phase.	>pe <Enter> to exit "pe"	Phase left side peak by pressing and holding left MB while dragging mouse side to side. Repeat using the right MB to adjust the right peak.
11	Fit baseline.	>fb <1> <Enter>	Enter fb subroutine, remove stripes on or too close to peaks, press the letter "l" for Least Squares fit, save result and exit fb with <Enter>.
12	Enter integral display.	>id	
13	Integrate data.	two clicks of left MB, then one left click < Enter > to exit "id"	For each broken integral, click left MB twice on left side of peak(s) then once on right side. To assign a relative integral value place cursor on integral, click left MB, press <v> and enter number. <Ctrl+I> toggles integrals on/off.
14	Pick Peaks a. Automatic or b. Manual	>PP (or use the icon on the menu) or >dp <c> <Enter> to exit "dp"	a. Automatic peak pick - Vertical red lines indicate selected peaks. Use MH and RM to change peaks selected, b. Manual peak pick -The cursor becomes a crosshair with a DP label. <a> automatically picks peak; <c> clears all peak picks; <k> removes a single peak pick nearest the cursor. Add peak by clicking the left MB near any peak. <t> writes peak list to the table. <Ctr 1+B> toggles peak pick table on/off. <Ctrl+P> toggles the peak labels on/off.
15	Expand selected region.	>zo <Enter> to exit "zo"	Select expansion region with mouse or <f > to enter fixed offsets with information dialog box. <Ctrl+E> gives the expanded region <Ctrl+F> gives the full spectrum.
16	Plot Data.	>pl (or use the icon on the menu)	

^{13}C Survey Spectra Guide

Step	Function or Dialog	<Keystroke>/[Select]/<Data	Comment
1	Sample		See Sample Preparation Guide. Position sample spinner using the depth gauge, place in probe.
2	Enter PNMR program.	<Alt+Tab>	(If necessary.)
3	Select ^{13}C observe.	Hl>nu C13<Enter>	Required only if the prompt is not C13>.
	Optional: Shim sample.	C13>shim<Enter>	Follow on-screen directions. Shims are optimized.
4	**Acquire ^{1}H spectrum.**	C13>zgh<Enter>	Obtain 'H spectrum using default conditions
5	Enter NUTS and process data.	<Alt+Tab> >a2	Trim phase as required. Use cursor to determine TMS peak position in ppm, including sign.
6	Return to PNMR and enter TMS peak position.	<Alt+Tab> C13>fo<Enter> value<Enter> 0<Enter>	Enter the current position (in ppm) of the TMS peak to the first dialog box and 0 (zero) to the second dialog box. Repeat to confirm.
	Optional: Confirm field offset and save 'H spectrum for border of HETCOR	C13>zgh<Enter> <Alt+Tab>, a2 > filename	Acquire proton spectrum. Switch to NUTS and process with a2 link. Enter filename for 'H spectrum, for example *border_hl* . *<Alt+Tab>* to return to PNMR
7	Verify parameters.		Verify that parameters make sense: for neat samples ns-12, for 1M samples ns=60, for concentration <1M use the BAPR program.
8	**Acquire data.**	C13>zg<Enter> then data \filename<Enter> or <Enter> for default	Enter file name if desired but it is usually better to use the default (pnmr.fid) unless intending to save the data long term.
9	Enter NUTS.	<Alt+Tab>	
10	**Process data**.	<Ctrl+F3> then [filename] [Open] to select a file or [Open] for default	Process using aii_C13.mac that references TMS and sets display range from 220 to -10 ppm. Does an automatic peak pick.
11	Enter line broadening.	value<Enter>	LB=0.5 Hz is a typical value.
	Optional: Pick peaks manually.	>dp <Enter> (or use the pick peak icon on menu)	The cursor becomes a crosshair with a Dp label. <c> clears all peak picks; <k> removes a single peak pick at the cursor location. Add peaks by aligning cursor on a peak and clicking the left MB. <t> to write peak list to the table.
12	Plot Data.	>pl (or use the print icon on menu)	
	Optional: Save ^{13}C spectrum for border of HETCOR plot.	<Ctrl+B> <Ctrl+P> >sa *filename*	Remove peak labels and table displays. Enter filename for ^{13}C spectrum, for example *border_ cl 3* .
	Optional: Add scans	C13>Go<Enter>	In case the signal to noise is too low, the acquisition can be extended.
	Number of added scans	scans<Enter>	For neat samples ns-12, for 1M samples ns=60, for concentration <1M use the BAPR program.
		data \filename<Enter>	The program will ask for a filename when finished. Use the same name as step 8.

NOTE: For weak samples use Block Averaging with Peak Registration (BAPR)