Getting started on av401

Purple *stands for* ***(optional)*** *commands that have to be typed into the command line followed by the enter key.*

Red *stands for windows / buttons that have to be clicked with left mouse button.*

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	position sample into spinner using depth gauge and wipe it with a tissue
(halt)	in case previous experiment is still running (as indicated in status bar at bottom)
ej	switch on eject air, wait for fizzling noise and place sample at magnet entrance
ij	switch off eject air, sample slowly enters magnet
(ro)	switch on/off sample spinning
	open **NMR data browser** and the data directory **/opt/topspin3.2/data/standard** click on desired experiment (e.g. **standard_4h** for ^{1}H) and drag into main window
new	under **name** enter your initials followed by sample number/code followed by _4exp (e.g. mj-01a_4h für ^{1}H), under **dir** select workgroup/user, under **title** enter title
lock	select solvent and wait for stable horizontal yellow-orange line in **lock display**
atma	automatic tuning and matching of probe for each nucleus of the current experiment
tg	open **topshim** (**shim**) window and click **start** for automatic shimming (1-2 min)

(ns)	adjust number of scans, **expt** displays experiment time
(sw, o1p)	adjust spectral window (from o1p+sw/2 to o1p-sw/2)
rga	optimize receiver gain for current experiment
zg	start measurement
(tr)	save FID onto disc while experiment is running
(halt)	interrupt running experiment, FID is saved onto disc
(go)	continue experiment, FID is added onto existing data

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efp	Fourier transformation 1D data
apk	automatic phase correction 1D data
abs n	automatic baseline correction 1D data
xfb	Fourier transformation 2D data
(projd)	select 1D traces for display along with 2D data
ej	switch on eject air to remove the sample
ij, (ro)	switch off eject and spinning air

Variable Temperature on av401

Create dataset, lock/shim the sample and tune/match the probe at 25 °C

edte open VT control program

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for T > 25 °C

Change Target Temp. (< 10 K below boiling point of solvent, see under **edlock** -> **properties**)

Set maximum power of heater (e.g. 2% for 25 °C, 7% for 50 °C, 12% for 75 °C, 17% for 100 °C)

do not change air flow (400 L/h)

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for T < 25 °C

change target temp. (> 10 K above melting point of solvent)

switch off heater (click **ON**)

fill dewar with LN_2 (ask staff) and place it on the right side of the magnet ~15 cm away from both walls

mount LN_2 evaporator (has to be free of moisture) onto dewar using O-ring and clamp

wait until pressure has dropped or release pressure (avoid LN_2 escaping from transfer line!)

remove black air hose from probe

mount transfer line on holder using **screw A** (it should be in line with the gas inlet of the probe)

gently push transfer line onto the probe and fasten **screw B**

connect evaporator power supply cable to the grey cable on rear side of the spectrometer console, the control program should now show “cooling OFF”

switch on cooling (click **OFF**) and **change** cooling power (5% for 0 °C, 10% for -25 °C, 20% for -50 ° and 30% for -75 °C)

switch on heater and **set maximum** power to 5-10%

cooling has to be stopped immediately if A) the transfer line is not tightly connected to the probe (fizzling noise), B) more than 1 cm of ice have formed on the transfer line, C) either the shim system (red) or the magnet bottom flange (black) show moisture

after measurement, switch off heater and cooling

gently remove transfer line from probe (do not force, ice may have to be removed first) and mount black air hose back on probe

change target temperature back to 25 °C and switch on heating (max 2%)

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general

the probe can be tuned / matched, the sample shimmed and short measurements can be done if the current temperature is within 1 °C of the target temperature

longer measurements (2D) require the current temperature to be stable within 0.1 °C of the target temperature, this usually takes > 10 min

book enough time to make sure the magnet is stable at 25°C (heating max. 2%), this may take ~30min

People that repeatedly fail to follow the above procedure will be excluded from access to the instrument!