

NMR Thermometer

Variable Temperature Control Using the 2H Lock System of AVANCE III HD Spectrometers

User Manual Version 002

Innovation with Integrity

NMR

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1 Introduction

This manual is planned as a user manual with limited technical detail. The main focus is on usability, whereas a few easy examples are provided showing how to get started with this new tool. Through use of this manual the user should get an idea for what the NMR Thermometer[™] might be used for.

The comparability of data (chemical shifts) and results derived from NMR data (diffusion data, relaxation measurements), as well as the quality of the NMR spectra, depend on an accurate and precise temperature measurement. That is the reason why the temperature measurement should ideally take place inside the NMR tube and not outside of it. In general the temperature sensor of the probe is not reflecting the real situation inside the NMR tube.

The NMR Thermometer not only monitors the temperature, but also compensates for different heating effects (e.g. RF heating) that occur during an NMR experiment.

2 Principle of NMR Thermometer

The NMR Thermometer measures the temperature inside the sample by observing the chemical shifts of two ²H signals using the lock channel (LTRX board) of the NMR system. The distance between the two signals is transferred into a temperature and directly used by the temperature control unit (BVT) for regulation. Thus, the NMR Thermometer acts as a temperature sensor (see figure below).

To obtain the second lock signal, a thermometer substance needs to be added. One of the signals should also be temperature dependent.





Figure 2.1: Principle of the NMR Thermometer.

3 Requirements

Hardware: Avance III HD. A hardware upgrade is required for Avance III, whereas an update to SmartVT[™] and Digilock 2G is required.

Software: TopSpin version 3.2 or higher.

Firmware: Versions for Avance III HD.

4 Getting Started

This chapter provides a short and straightforward introduction on how the NMR Thermometer works and guides you through the first steps using the NMR Thermometer. As an overview, the steps include:

- · Preparation of the edlock table if not yet done.
- Locking on the corresponding solvent used for the NMR Thermometer.
- · Optimizing the shim and lock phase.
- Activating the NMR Thermometer in the variable temperature control.
- Performing self-tuning of the variable temperature unit. This will optimize the regulation parameters for both the VT control with the sensor and the NMR Thermometer.
- Calibration of the spectrometer for measurements of real samples after the setup of the NMR Thermometer is finished.

4.1 The edlock Menu

First we will provide a short introduction to the software implementation (edlock, edte).

Start edlock by typing edlock on the TopSpin command line. The edlock window opens up.

The new *edlock* (starting for TopSpin 3.0 and higher) contains both the solvent list (formerly *edsolv* in the Edit Solvent Parameters figure below) and the lock parameters for every solvent (see the The Submenu Lock figure below).

Edlock		
<u>S</u> olvents <u>E</u> dit I	3SMS <u>H</u> elp	
Auto Phase	Auto phase algorithm	Urrent probe
🗌 Auto phase duri	ng lock Auto phase algorithm Enhanced Lock Level 💌	5 mm CPTCI 1H-13C/15N/D Z-GRD Z75812/0055
Solvents Lock	Spectrum Reference Properties	
△ Solvent		Description
Acetic	acetic acid-d4	·
Acetone	acetone-d6	
C2CI4D2	tetrachloroethane	
C6D6	benzene-d6	
C6D6+Dioxane	ASTM Sample	
C6D6_CDCI3		
CD2CI2	methylenechloride-d2	
CD3CN	acetonitrile-d3	
CD3CN_SPE	LC-SPE Solvent (Acetonitrile)	
CD3OD_SPE	LC-SPE Solvent (Methanol-d4)	
CDCI3	chloroform-d	
CH3CN+D2O	HPLC Solvent (Acetonitril/D2O)	
CH3OH+D2O	HPLC Solvent (Methanol/D2O)	
D2O	deuteriumoxide	
D2O_MeOD		
DEE	diethylether-d10	
Dioxane	dioxane-d8	
DME	dimethylether-d6	
DMF	dimethylformamide-d7	
DMF2	dimethylformamide-d7	
DMF3	dimethylformamide-d7	
DMSO	dimethylsulfoxide-d6	
EtOD	ethanol-d6	
Gly20		
Glycol	Ethylenglycol	×
		Glose

Figure 4.1: The Submenu Bar: Solvent.

The Edit Solvent Parameters window allows you to enter several parameters for the solvent, the melting point setting, and the boiling point of the solvent:

🍯 Edit solvent parar	ieters	□ X
Edit solvent "Acetic". Solvent parameters Solvent name:	Acetic	
Solvent description:	acetic acid-d4	
Lock Nucleus: Lock Solvent: Hidden:	2H 2	
Auto Phase:		
Melting Point [K]:	288.7	
Boiling Point [K]:	388.7	
		<u>QK</u> <u>C</u> ancel

Figure 4.2: Edit Solvent Parameters.

In the submenu Lock window the solvents are listed, along with the probe for which the definition is valid (generic or specific probe), as well as other lock parameters (lock power, lock regulation triplet etc.).

Eulock										
<u>Solvents</u> Edit <u>B</u> S	MS <u>H</u> elp									
Lock Nucleus Auto P	hase	Auto phase	algorithm C	Current probe						
● 2H O 19F □ Auto	o phase during lock	Lock Level (Default) 🔍 🤇	Current probe:	5 mm CPTCI 1H	-13C/15N/D Z-	GRD Z75812/005	5		
Solvents Lock S	pectrum Reference	Properties								
△ Solvent	Probe .	Lock PowerLoc	k Power Instep	Loop Gain	Loop Time	Loop Filter	Shift (ppm) R	el Type		
P Acetic	Generic	-38	10	-10	0.1	100	2.03			
∽ Acetone	Generic	-38	10	-2	0.1	200	2.04			
C2CI4D2	Generic	-20	10.1	-10	0.1	100	6			
~ C6D6	Generic	-26	10	-0	0.2	300	7.16			
CD2CI2	Generic	-30	10.1	-7	0.25	100	5.32			
CD3CN	Generic	-38	10	-2	0.1	200	1.93			
► CD3CN_SPE	Generic	-20	10	-10	0.1	100	1.93			
CD3OD_SPE	Generic	-25	10	-5	0.1	100	3.3			
► CDCI3	Generic	-30	10	-12	0.4	100	7.24			
∽ CH3CN+D2O	Generic	-18	10	-10	0.3	100	4.7			
CH3OH+D2O	Generic	-18	10	-10	0.3	100	4.7			
	Generic	-18	10.1	5	0.25	500	7.24			
← D2O	Generic	-18	10	5	0.25	500	4.7			
↔ DEE	Generic	-30	10.1	-15	0.2	100	1.07			
🕈 Dioxane	Generic	-30	10.1	-15	0.2	100	3.53			
○ DME	Generic	-35	10.1	-10	0.1	100	3.3			
► DMF	Generic	-20	10	5	0.25	500	2.91			
∽ DMSO	Generic	-20	10	5	0.25	500	2.49			
► EtOD	Generic	-30	10	-15	0.2	100	1.11			
↔ H2O+D2O	Generic	-18	10	-5	0.35	100	4.7			
HDMSO	Generic	-25	10	-9.4	0.464	50	2.49			
∽ MeOD	Generic	-35	10	-5	0.1	100	3.3			
- MeOD_T	5 mm C	-35	10	-5	0.1	100	3.3			į
- Signal 1							3.3	3 Lock		
Signal 2							4.8	1 Temperature		
∽ Pyr	Generic	-25	10	-15	0.1	100	8.71			
► TFE	Generic	-30	10	-12	0.4	100	3.88			
THF	Generic	-25	10	-10	0.1	100	1.73			
∾ Tol	Generic	-38	10	-10	0.1	100	2.09			
										Close
										2000

Figure 4.3: The Submenu Lock.

By executing a right mouse click on a solvent entry, a pull-down menu opens with the option for editing the lock parameters:

Getting Started

Edlock				• • ×							
Solvents	Edit BSMS Help										
Lock Nucle	eus Auto Phase /	Auto phase algorithm	Current probe								
© 2H ⊘ 1	9F Auto phase during lock	Auto phase algorithm	Lock Level (Default) Current probe: 5 mm PABBO BB-1H/D Z-GRD Z116098/0004								
Solvents	Lock Spectrum Reference Proper	ties									
△ Solv	ent Probe	Lock Power	Lock Power Instep Loop Gain Loop Time Loop Filter Lock Phase S	Shift [ppm]							
Acetic	Generic	-38	10 -10 0.1 100 310	2.03							
. Aci	Expand	-38	10 -2 0.1 200 252	2.04							
. C2	Expand all	-38	10 -2 0.1 200 310	5.32							
@ C6	Add new solvent	-26	Edit lock parameters	7.16							
. CD	Edit solvent	-38	Edit lock parameters for solvent "Acetic"	5.32							
. CD	Delete solvent	-38	Lock parameters	1.93							
B CD	Copy and paste solvent	-20	-20 Probe name: 5 mm CPTCl 1H-13C/15N/D Z-GRD Z75812/0055 -25 Probe description:								
. CD	Hide current solvent	-25									
. CD	Edit lock parameters	-30	lock power: -38	7.24							
. CH	Delete probe	-18	Loop gain: -10	4.7							
. CH	Delete probe from all solvents	-18	Loop time: 0.1	4.7							
@ D2	Copy probe to all solvents	-18	Loop filter: 100	4.7							
Dic	Edit parameters of "Generic" probe	-38	Lock Phase 225	3.53							
. DN	Copy Lock Phase to all solvents	-38	Lock Phase. 223	2.91							
. DN	Сору	-20	Lock power instep: 10	2.49							
. EtC	Export	-30	Temperature lock power: (-38	1.11							
H2	Import		Signals	4.7							
. HD	Print	-25	Signal Shift [ppm] Relative intensity Type Description Delete	2.49							
Jui-	Print preview		1 2.03 3 Lock 💌	4.7							
. Me	Table properties	-35	2 11.65 1 Signal 💌	3.3							
Plasma	a Generic	-18	Add Signal Delete Signal	4.7							
Pyr	Generic	-38		8.71							
TFE	Generic	-30	Add temperature/shift values	3.88							
0-THF	Generic	-38		1.73							
I Tol	Generic	-38	<u>QK</u> <u>C</u> ancel	2.09							

Figure 4.4: Edit Lock Parameters.

Every solvent entry contains information about every ²H signal for that compound.

It is possible to define all the signals for solvents with more than one ²H signal either as Signal, as Lock (signal used for field lock) or as Temperature (signal used for the NMR Thermometer).

For any NMR Thermometer substance, temperature and shift values can be added or imported (see below). The NMR Thermometer has its own lock power (figure above). For methanol (NMR Thermometer, standard sample: 99.8% deuterated methanol) a default solvent with corresponding lock parameters and temperature-shift value has already been defined.

4.2 Setting up the NMR System for the NMR Thermometer

Since the NMR Thermometer is observing a ²H signal, the system has to be properly set up (lock, shimming, optimal lock parameters for the field lock and NMR Thermometer), similar to any other NMR measurement. This means that you should perform an automatic tuning and matching (*atma*), lock-in, and shimming. The lock-in procedure works as usual (type **lock** on the command line and select the solvent). If you lock directly on a solvent dedicated for the NMR Thermometer (methanol or another solvent), a temperature value is immediately shown, either in the monitoring mode or regulation mode (figure below). After that you can perform *topshim*. A decent line shape (no unusual line splitting) is mandatory for an exact temperature measurement inside the sample.

Getting Started



Figure 4.5: Edte Window: NMR Thermometer monitoring mode (disabled, top), regulation mode (enabled, middle) and the selection of both modes in the Configuration menu of the edte window (bottom).

Another important parameter is the *lockphase*, which can be optimized automatically by starting *autophase* (BSMS display). The procedure used for *autophase* is selected in the edlock window (Lock Level Default, Spectrum, and Enhanced Lock Level in the figure below).

i Edlock			
<u>S</u> olvents <u>E</u> di	t <u>B</u> SMS	<u>H</u> elp	
Auto Phase		Auto phase algorithm	Current probe
🗌 Auto phase d	luring lock	Auto phase algorithm Enhanced Lock Level 💌	5 mm CPTCI 1H-13C/15N/D Z-GRD Z75812/0055
Solvents Loc	k Spectri	um Reference Properties	

Figure 4.6: Selection of the Auto Phase Algorithm.

To prevent saturation, the *Lockpower* and *lockgain* for both lock channels should be optimized as well.

Since the NMR Thermometer contains both the lock and the temperature regulation components, the PID values for the temperature regulation need to be adjusted for each by using *selftune*. You will be notified by the system if a *selftune* is recommended:

🍯 PID s	settings	
и Р Т	Warning: 'our "Tune settings" might not be appropriate. Executing "Self tune" is highly recommended. larget temperature does not match – Current = 350.8K, whereas range of Loaded = 249.5K to 34	9.5K.
		ose

Figure 4.7: Selftune warning about PID parameters misfit.

Temperature Monit	oring Record Corr	ection Self tune Config	uration Log He	lp			
Self tune							
Execute self tune to im You can self tune each To save the self tune pa	prove the regulation cap channel independently trameters for a defined	oabilities of the VTU. (select self tune for the appi temperature, gas flow, prob	opriate channel) or e and sensor press	self tune all availab the "Get" button of t	le channels simu the desired chan	Itaneously (select self tune all channels), nel and enter a name for the settings.	
Chan	nel	Sensor	Start self tune	Stop self tune	Get self tune pa	rameters View self tune parameters	
AI	L. C.		Start	Stop			
1 5 mm CPTCI 1H-13	C/15N/D Z-GRD	adapter connection 1	Start	Stop	Get	View	
						PID settings	
Available self tune set	angs	Prohe	Temperat	ture (K1) Elow (lpb	1 Sensor		
BBFO	5 mm PABBO BB-1H/D 2	-GRD Z114607/0007	299.5	535.000	1	PID settings for NMR thermomete	er:
BBFO_1	5 mm PABBO BB-1H/D 7	-GRD Z114607/0007	198.8	800.000	1	Pagulator KP: 0.525 W/K	
BBFO_2	5 mm PABBO BB-1H/D 2	-GRD Z114607/0007	369.9	535.000	1	Bagulator TD: 0.000 a	
BBFO_SP	5 mm PABBO BB-1H/D I	-GRD Z114607/0007	298.0	535.000	1	Regulator TD: 0.000 S	
CP_TCI	5 mm CPTCI 1H-13C/1	5N/D Z-GRD Z75B12/0055	298.0	535.000	1	Regulator II: 19.2323	
CP_TCL_STOK	5 mm CPTCLTH-13C/19	5N/U 2-GRU 275812/0055	312.5	535.000	1		
	3 IIII PATA 11/0-13C	1381-460 2833801/00/7	236.0	330.000	1	Qlose	e

Figure 4.8: Starting the selftune procedure from the edte window.

The selftune should be carried out on both temperature channels (All).

4.3 Examples

An easy example to begin with is to use the methanol sample (standard sample: 99.8% deuterated).

Since this sample is the reference for the NMR Thermometer, the solvent entry in *edlock* containing the temperature and shift values is already predefined.

As mentioned, the NMR Thermometer is running in two different modes: monitoring and regulation mode (see above).

Assume that the lock parameters are already optimized.

4.3.1 Monitoring Mode

The following steps need to be performed:

- Insert sample.
- Tune and match by using the *atma* command.
- Lock-in temperature appears.
- · Topshim.
- · Selftune.

Temperature Monitoring Record	Correction Self tune Con	figuration Log Help			
📀 Configure					
Temperature display properties Pow	er display properties	Gas flow display pro	General display pr	operties	
Show overview 🗌 Auto scale 🗹 Sho	ow overview 🗌 🛛 Auto scale 🗹	Show overview 🔲 🧳	Auto scale 🗹 🛛 Update interval [s]: 1	
Channel	Current Temperature	Target Temperature	Current Power Maximum P	ower Current Gas Flow	Target Gas Flow
1 5 mm CPTCI 1H-13C/15N/D Z-GRD					
*2*8/2/8 \$ 毫주\$↓₩&@					
Temperature (Channel 1, 5 mm CF	тсі 1H-13C/15N/D Z-C	RD Z75812/0055)			Ξ
······					- 00
		<u>~-</u>			- 88 -
		ſ			29 Derati
	······				7-1- 296
	/				4
					292
100	200	300	400	500	[s]
*2*8/2/8 ≑ 至 ₸ ‡ ¥ 🥰					
Power (Channel 1, 5 mm CPTCI 1H	-13C/15N/D Z-GRD Z7	5812/0055)			[W]
]]				F
	\				Ē
					-9
$ \neg \neg \rangle^{\chi} $		$\langle $			ower
		V			
					-
					-0
100	200	300	400	500	[5]

VTU: On 🥑 Sample Temperature: Corr. 297,9 K Probe Regulation: Transient 😳 Tune: OK 🕐 Recording: Off Probe: 5 mm CPTCl 1H–13C/15N/D Z–GRD Z75812/0055

Figure 4.9: Selftune in Progess.

- Edte disable NMR Thermometer (if not already done).
- Change target temperature (*edte* for instance, a 10K temperature jump).

As an exercise we will perform a temperature jump (e.g. 10K) and follow the different temperature and other BSMS values (flow/heater) in the monitoring window (*edte* - Monitoring, as well as, activate NMR Sensor Temperature, NMR Thermometer, Target Temperature, Current Power etc.).



Figure 4.10: Monitoring several BSMS values during a temperature jump.

4.3.2 Regulation Mode

The following steps need to be performed:

- · Insert sample.
- Tune and match *atma*.
- Lock-in temperature value appears.
- · Topshim.
- Selftune (maybe already done in example 1).
- Edte enable NMR Thermometer.
- Perform an experiment with internal RF heating (e.g. TOCSY).

After setting up the system (lock, shim) and enabling the NMR Thermometer (*edte*) the sample temperature is used for temperature regulation, which is visible on the status bar:



Figure 4.11: TopSpin status bar with NMR Thermometer enabled.

To test the performance of the NMR Thermometer start a TOCSY experiment and follow the temperature values (sensor, NMR Thermometer) in the monitoring window of the VTU display (*edte*). One can nicely see that the temperature inside the sample increases over a certain

period of time and that the system immediately reacts to that by reducing the heater power (figure below - lower part), and hence the sensor temperature (figure below - upper part, white line).



Figure 4.12: Monitoring of the sensor and the sample temperature in edte during a TOCSY experiment

In the figure above, the upper part shows the temperature of the sensor and the NMR Thermometers; the lower part shows the heater power.

5 Advanced Operation

5.1 Define a New Solvent

To work with your own NMR Thermometer substances, you first have to define a new solvent in the *edlock* table.

Select a solvent which is similar to your mixture (similar lock parameters) and click the right mouse button. In the resulting pull-down menu you can add the new solvent:

olvents Edit	BSMS Help)					
ock Nucleus	Nuto Phase	Auto	phase algorithm	Current probe			
2H © 19F	Auto phase of	during lock	k Level (Defauit) 🔹	Current probe:	5 mm PABBO BB-1H		
olvents Lock	Spectrum Refe	rence Properties					
△ Solvent	Probe	Lock Power	Lock Power instep	Loop Gain	Loop Time		
CD3CN	Generic	-38	10	-2	0.1		
CD3CN_SPE	Generic	-20	10	-10	0.1		
CD3OD_SPE	Generic	-25	10	-5	0.1		
CDCI3	5 mm PA	-30	10	-12	0.4		
CH3CN+D2O	Generic	-18	10	-10	0.3		
CH3OH+D2O	5 mm PA	-18	10	-10	0.3		
D20 EVD	bod		10	5	0.25		
DIO	and of		10	-2	0.1		
DM			10	-2	0.1		
DM	new sorvent		10	5	0.25		
EtO Dole	solveni to solvent		10	-15	0.2		
H2C Con	and pasto col	work	10	-5	0.35	Create solvent	
HDI Hide	current solven	t	10	-9.4	0.464		
Juic	lock parameter		10	-5	0.35	Ureate a new solven	Ľ.
Met Dok	to probo	3	10	-5	0.1	Solvent parameters	
Plat Dele	te probe from :	all solvents	10	-5	0.35	Solvent name:	
Pyr Con	in probe to all si	oluente	10	-2	0.1	Column de conistion	
TFE Edit	parameters of	"Generic" probe	10	-12	0.4	Solvent description:	
THE CON	Lock Dhase to	all envente	10	-2	0.1	Lock Nucleus:	2
Tol	Y LOCK PIIdSE I	o di opivento	10	-10	0.1	Lock Solvent:	
Urir Evo	y at		10	-5	0.35	Hidden:	m
Imp	art.					Auto Phase:	
Deire	11					Meiting Point [K]:	Г
Print						Poiling Point B/I	
Print	preview					boiling Point [K].	

Figure 5.1: Adding New Solvents.

In the edit lock parameters window the signals can be defined (Shift) and assigned (Type) as Lock, Signal or Temperature:

Cancel

lvents Edi	t BSMS Help					
Lock Nucleus Auto Phase Auto phase algorithm			Current probe			
2H O THE	Auto shate duting lock	Lock Loval (Default)	Current probe	5 mm CPTCL1H	H-120/158/0 7-090 775912/0055
all and	Course suiters annual mos	COLK CEVEL (veraono [-	content prope.	a min or ror at	and a second a second second
iolvents Loo	k Spectrum Reference	Properties				
a Solv	ent Probe L	ock PowerLoc	k Power Instep	Loop Gain	Loop Time	Loop Filter Shift (spm) - Rel Type
Acetic	Generic	-38	10	-10	0.1	100 2.03
Acetone	Generic	-38	10	-2	0.1	200 2.04
C2C14D2	Generic	-20	10.1	-10	0.1	100 6
- C6D6	Ceneric	-26	10	-0	0.2	300 7.16
CD2CI2	Generic	-30	10.1	-7	0.25	C Edit lock parameters
CD3CN	Generic	-38	10	-2	0.1	Edit lock parameters for solvent MaOD T
CD3CN_SPE	Generic	-20	10	-10	0.1	
CD3OD_SPE	Generic	-25	10	-5	0.1	Lock parameters
CDCI3	Generic	-30	10	-12	0.4	Probe name: 5 mm CPTCI 1H-13C/15N/D 2+CRD 275812/0055
- CH3CN+D2	O Generic	-18	10	-10	0.3	Probe description:
CH3OH+D2	O Generic	-18	10	-10	0.3	Lock power: -35
- CHCI3	Generic	+18	10.1	5	0.25	Loop gain -5
D20	Generic	-18	10	5	0.25	
DEE	Generic	-30	10.1	-15	0.2	Loop time: 0.1
Dioxane	Generic	-30	10.1	-15	0.2	Loop filter: (100
DME	Generic	-35	10.1	-10	0.1	Lock Phase: 220
DMF	Generic	-20	10	5	0.25	Lock power instep: 10
> DMSO	Generic	-20	10	5	0.25	Darah
- EtOD	Generic	-30	10	-15	0.2	agnas
H20+D20	Generic	-18	10	-5	0.35	Signal Shift [ppm] Relative intensity Type Description Delete
HOMSO	Generic	+25	10	-9.4	0.464	1 3.3 3 Lock 💌
MeOD	Ceneric	+35	10	-5	0.1	2 4.8 1 Temperature *
MeOD T	5 mm C	-35	10	-5	0.1	
Signal 1						Add Signal Delete Signal
Signal 2						Temperature values
Pyr	Ceneric	+25	10	-15	0.1	A STATUTE MARKE FRANCE
TEE	Ceneric	-30	10	-12	0.4	Value Shift [ppm] Temperature [K] Delete
THE	Canacic	-25	10	-10	0.1	
Tol	Canaric	-38	10	-10	0.1	And Value Distance Value
	Generic		10	-10	0.1	LUND VALUE LIFECE VALUE
						[Description of the second seco
						QK Cancel

Figure 5.2: Assign Signals to Type of Signal (Signal, Lock, Temperature).

In the next step you should import the shift-temperature values (.csv or .xml format) or fill in the values manually. You can also create your own .xml (see below).



Figure 5.3: Dialog for importing temperature shift value files.

The .xml file (figure below) contains the name of the solvent (identical with the solvent name) and two shift values, one for the field shift and one for the temperature shift. The values have to be identical with the values defined for the particular solvent in *edlock*.

	•
<field shift="4.7"></field>	
<temperature shift="1.8"></temperature>	•
<point <="" delta-shift="2.4766" th=""><th>temperature="346.1600" /></th></point>	temperature="346.1600" />
<point <="" delta-shift="2.5198" th=""><th>temperature="341.3700" /></th></point>	temperature="341.3700" />
<point <="" delta-shift="2.5634" th=""><th>temperature="336.6200" /></th></point>	temperature="336.6200" />
<point <="" delta-shift="2.6080" th=""><th>temperature="331.8000" /></th></point>	temperature="331.8000" />
<point <="" delta-shift="2.6532" th=""><th>temperature="327.0000" /></th></point>	temperature="327.0000" />
<point <="" delta-shift="2.6990" th=""><th>temperature="322.2400" /></th></point>	temperature="322.2400" />
<point <="" delta-shift="2.7458" th=""><th>temperature="317.4400" /></th></point>	temperature="317.4400" />
<point <="" delta-shift="2.7933" th=""><th>temperature="312.6200" /></th></point>	temperature="312.6200" />
<point <="" delta-shift="2.8422" th=""><th>temperature="307.8100" /></th></point>	temperature="307.8100" />
<point <="" delta-shift="2.8915" th=""><th>temperature="302.9800" /></th></point>	temperature="302.9800" />
<point <="" delta-shift="2.9422" th=""><th>temperature="298.1800" /></th></point>	temperature="298.1800" />
<point <="" delta-shift="2.9937" th=""><th>temperature="293.3600" /></th></point>	temperature="293.3600" />
<point <="" delta-shift="3.0464" th=""><th>temperature="288.5500" /></th></point>	temperature="288.5500" />
<point <="" delta-shift="3.1004" th=""><th>temperature="283.7200" /></th></point>	temperature="283.7200" />
<point <="" delta-shift="3.1566" th=""><th>temperature="278.9200" /></th></point>	temperature="278.9200" />

Figure 5.4: Example of a shift-temperature file in .xml format.

After the import the shift and temperature value are filled in the *edlock* table for the selected solvent and used afterwards for the lock-in process Create a new XML file:

Edit lock	parameters for solv	ent "MeOD_T".			
Lock pa	rameters				
	Probe name	: 5 mm CPTCI 1H-	-13C/15N/D Z-C	RD Z75812/0055	
	Probe description	:			
	Lock power	: -35			
	Loop gain	: -5			
	Loop time	: 0.1			
	Loop filter	: 100			
	Lock Phase	: 220			
	Lock power instep	: 10			
Tem	perature lock power	: -35			
Signals					
Signal	Shift [ppm] Relat	ive intensity	Туре	Description	Delete
1	3.3	3 Lock	-		
2	4.8	1 Tem	perature 💌		
		Add Signal	Delete Signal		
Temper	ature shift values				
	1 2440				
19	1.3449 31	6.07			
20	1.3546 31	5.1			
21	1.3644 31	4.16			
22	1.3742 31	3.19			
23	1.3839 31	2.23			-
		Add Value	Delete Value		
				OK	Cancel

Figure 5.5: Temperature shift values filled in.

For many NMR applications (for instance, Bio-NMR in aqueous solutions) an estimate of the slope (be aware of the fact that the slope can have negative or positive sign!) of the temperature dependency and the knowledge of the offset (absolute temperature correction) are sufficient (the necessary temperature range might be rather small) to create an .xml file. It is off course also possible to determine the correct slope automatically.

5.2 Selection of NMR Thermometer Compounds

The simplest compound for the NMR Thermometer is fully deuterated methanol used as a NMR solvent. While the deuterium signal of the methyl group is used for the field lock, the deuterium signal of the hydroxyl group is used for the NMR Thermometer. The deuterium chemical shifts of commonly used organic NMR solvents like $CDCI_3$, acetone-d₆ and DMSO-d₆ are virtually independent on the sample temperature. In that case, deuterated methanol could be placed in a capillary or mixed with the solvent.

The chemical shift of water strongly depends on the temperature. For samples in aqueous solutions, a deuterated organic small molecule can be added as a thermometer compound. Details will be described in the following section.

Name	CAS number	Availability	Number of D	Chemical shift	Minimum Concentration
				[ppm]	[CryoProbe TCI 5mm 600MHz]
Methanol-d4	811-98-3	yes	3	3.3 (CD3)	
				4.8 (OD)	
Acetic acid-d4	1186-52-3	yes	3	2.03	20-50mM
Sodium acetate-d3	39230-37-0	yes	3	1.8	20-50mM
DMSO-d6	2206-27-1	yes	6	2.49	20mM
Acetone-d6	666-52-4	yes	6	2.04	20mM
Acetonitril-d3	2206-26-0	yes	3	1.93	20-50mM
Tetramethylammoniumchlorid-d12	23789-03-9	yes	12	3.2	10mM
TRIS-d11	n.a.	yes	6	3.5	20mM
TSP-d14	n.a.	on request	9	0	n.a.
DSS-d16	n.a.	on request	9	0	n.a.
Pivalic acid	42983-07-3	yes	9	1	10-20mM

The compounds listed in the table below have been tested and can be considered.

Figure 5.6: Suggested compounds for the NMR Thermometer and samples in aqueous solution (D2O 5 – 100%), their properties and estimated concentration. Fully deuterated DSS and TSP are currently not available but will be synthesized on Bruker's request at small amounts only for internal tests.

For the selection of a suitable compound the following points should be considered:

- Large number of deuterons: The larger the number of chemically equivalent deuterons, the lower the required concentration. Tetramethylammonium chloride-d₁₂ is in this respect, the preferred compound, whereas sodium acetate is less favorable.
- Possibly one additional ²H signal.
- Moderate or no salt effect: Salts and acids in aqueous solution will increase the conductivity of the sample, which might cause a loss of sensitivity and an increase of the pulse length.
- The compound should not interact with the sample or change essential structural properties of the sample.
- Acceptance. Difference compounds are commonly added to protein solutions. These can be buffers like TRIS or stabilizers like EDTA. These compounds can be used if deuterated and available at a sufficiently high concentration.
- Price and availability.

5.2.1 Predefined Solvents for the NMR Thermometer

The lock table contains solvents which are setup for usage with the NMR Thermometer. For these solvents a temperature calibration of the chemical shift difference of the two ²H signals has been performed and is included. These are the following solvents:

T_MeOD: methanol-d₄

- T_H2O+D2O+NaAc: sodiumacetate-d₃ in 90% H₂O, 10% D₂O
- T_H2O+D2O+Me4NCI: tetramethylammoniumchlorid-d₁₂ in 90%, H₂O, 10% D₂O
- T_H2O+D2O+Pivalate: pivalic acid-d9 sodium salt in 90%, H₂O, 10% D₂O

Depending on the probe, the lock power may need to be adjusted.

5.2.2 Technical Considerations

When adding a thermometer substance to the sample, there are a few technical points to consider.

Depending on your NMR system (room temperature or cryogenically cooled probe, spectrometer frequency etc.) you need to add a sufficient amount of the compound in order to obtain a decent lock signal. Another concern is the distance from the ²H signal which is used for the field lock (figure below). In addition, the dynamic range, which is the intensity ratio of the ²H signal used for frequency lock and the NMR Thermometer, plays an important role when a highly or fully deuterated solvent (e.g. D_2O) is used as a field lock solvent.

The requirements for obtaining reliable lock-in and lock regulation performance include:

- S/N (²H) > about 200:1 (signal > ~3 ppm distant from the main signal)
- Intensity ratio < 500:1 (field lock signal: frequency lock signal; △ ~3 ppm)

The smaller the distance to the second signal, the higher the concentration of the compound used for the NMR Thermometer should be. As a rule of thumb: reducing the distance of the two lock signals by a factor of 2 increases the necessary concentration (decreases the possible intensity ratio of the two signals) of the NMR Thermometer compound by the same factor. Examples:

- The D of the NMR Thermometer signal to the field lock signals is about 3 ppm. The required signal to noise ratio of the NMR Thermometer compound has to be at least 200:1 (I-ratio <500:1).
- The D of the NMR Thermometer signal to the field lock signals is about 1.5 ppm. The required signal to noise ratio of the NMR Thermometer compound has to be at least 400:1(I-ratio <250:1).
- If the field lock signal in very large (for example using pure D₂O) the concentration of the NMR Thermometer compound has to be adjusted accordingly (see figure below).

Thermometer substances with a signal closer than 1 ppm to the field lock signal are critical and should not be selected as NMR Thermometer signals.



Figure 5.7: Approximate dynamic range of field lock (blue) vs. the NMR Thermometer signal (red) if the S/N of the frequency lock signal is sufficient (> 200:1).

The best performance (with low concentrations of the thermometer substance) can be expected using cryogenically cooled probes at high magnetic field strengths (600 MHz and higher), because the ²H sensitivity will be the highest. Therefore, check the ²H sensitivity of your system beforehand. Nevertheless, the NMR Thermometer also works at lower fields (e.g. 400 MHz) with room temperature probes.

The ²H sensitivity of a room temperature probe, such as an inverse or broad band observe probe, is about a factor 5-10 times lower compared to a cryogenically cooled probe. The concentration of the NMR Thermometer compound therefore needs to be higher, as listed in the following table:

NMR Thermometer compound	Solvent	Chemical shift	Minimum Concentration	Minimum Concentration
		[ppm]	[CryoProbe TCI 5mm 600MHz]	[BBFO 5mm 500MHz]
Methopol d4	mothanal d4	2.2 (CD2)		
Methanol-04	methanoi-04	4.8 (OD)		
Acetic acid-d4	D2O, 5-100% in H2O	2.03	20-50mM	200-500mM
Sodium acetate-d3	D2O, 5-100% in H2O	1.8	20-50mM	200-500mM
DMSO-d6	D2O, 5-100% in H2O	2.49	20mM	200mM
Acetone-d6	D2O, 5-100% in H2O	2.04	20mM	200mM
Acetonitril-d3	D2O, 5-100% in H2O	1.93	20-50mM	200-500m M
Tetramethylammoniumchlorid-d12	D2O, 5-100% in H2O	3.2	10mM	100mM
TRIS-d11	D2O, 5-100% in H2O	3.5	20mM	200mM
TSP-d14	D2O, 5-100% in H2O	0	n.a.	n.a.
DSS-d16	D2O, 5-100% in H2O	0	n.a.	n.a.
Pivalic acid	D2O, 5-100% in H2O	1	10-20mM	100-200mM

Figure 5.8: Typical concentrations required for the NMR Thermometer compound. If the NMR Thermometer compound is used with pure D2O as solvent, the higher concentration of the thermometer compound is required due to the high dynamic range of the intensity of both lock compounds.

5.2.3 Considerations for Shimming with Topshim

Depending on the solvent, topshim will use ¹H or ²H as a shim nucleus. For aqueous solutions, e.g. 5% D_2O in H_2O , ¹H is the shim nucleus, therefore the shim routine for Topshim does not need to be changed. For deuterated organic solvents ²H is used as the shim nucleus. If the solvent contains more than one ²H signal, like methanol-d₄ and pyridine-d₅, Topshim will use a selective ²H pulse in order not to excite additional signals.

In the case where the thermometer compound is used together with an organic solvent, either in a mixture with the solvent or as an external capillary, the shim routine has to be adapted. In the following the procedure for the setup of the shimming routine for a new solvent is described.

Two steps should be performed:

- Define a new solvent with *edlock*. The procedure is described in the section <u>Define a New</u> <u>Solvent [> 19]</u> of this manual.
- Define the shimming routine for Topshim.

The command for defining the Topshim shim parameters is:

topshim solvcal solvent= <new solvent name as in edlock> where <new solvent name as in edlock> is exactly the name of the solvent as defined in the lock/solvent table.

The following example will show the setup for a solvent called New.

1. Enter the command **topshim solvcal solvent=New**. A window pops up allowing the selection of a solvent. Here it is of no importance which solvent is selected:

♥ select file			_ O X
Look <u>I</u> n: 🗖 solve	ents	• A	
user Acetic C6D6 CD2Cl2 CD3CN CD3CN_SPE CDCl3 CH3CN+D20 CH3OH+D20 D20 DEE DefaultSolvent	DMF DMS0 EtOD H20+D20 MeOD oC6D4Cl2 pC6D4Br2 Pyr TFA THF Tol		
D DME			
File <u>N</u> ame: H2C Files of <u>T</u> ype: Al)+D2O I Files		~
	[select file	Cancel

2. Select the solvent of your choice and press the **select file** button. A new window will open, select the **OK** button to modify the shim parameters:

♥ tops	shim	o x
2	Do you want to modify the parameters loaded for New? Press - OK to continue - CANCEL to save the parameters without modifications and e - ABORT to quit without saving	×it
i.	<u>Q</u> K <u>C</u> ancel <u>A</u> bor	t

3. A new window will open and allow the selection of the shim nucleus. Enter **2** for 2 H as the shim nucleus:

🥌 topshim	
Nucleus used for shimming $(1 = 1H, 2 = lock)$:	
2	
<u>O</u> K	<u>C</u> ancel

4. Now the frequency of the signal has to be defined. As default, the frequency of the field lock solvent, which is the so-called *lockshift*, will be selected. Enter **1**:

♥ topshim	• ×
Mode for setting excitation frequency o1p (1 = lock shift, :	2 = manual):
1	
QK	<u>C</u> ancel

5. Enter 2 to activate the selective excitation:

Cancel
+

6. The definition of the selectivity for selective excitation is entered next. This depends on the difference of the chemical shift of the field lock solvent to the next closest signal of the thermometer compound. As an example, a selectivity of 0.5 ppm is sufficient for chemical shift difference of 1ppm. For larger shift differences a selectivity of 1 ppm shall be used:

• × •
- 10.00
<u>O</u> K <u>C</u> ancel

7. The T₁ relaxation time of the shim nucleus is used to define the repetition time for the shim procedure. Typical values for the T₁ relaxation time are about 1 sec. for D₂O, 3 sec for C₆D₆ and 5 sec. for acetone-d₆.

♥ topshim		×
Approximate T1 relaxation time [s]: 0.01 - 1000.00		
3.00		
		7
	ei	

8. In a final step the optimization parameter has to be defined. There are three options:

- **ss**: Shims will be optimized for solvent suppression
- Is: Shims will be optimized for the line shape
- Ishump: Shims will be optimized for narrow hump.

In the example shown here, solvents suppression has been selected:

♥ topshim			X
Specific optimisation parameter file :			
ss			
	<u>O</u> K <u>C</u> ar	ncel	

Further details of the setup of a new solvent for shimming can be found in the Topshim reference manual which can be accessed with the command **help topshim** in TopSpin.

6 Applications

6.1 Identical Chemical Shifts at Different Spectrometers

One of the most important applications of the NMR Thermometer is to use it as internal temperature reference and correct the temperature offset between sample and the temperature sensor of the probe. This is in general comparable to the conventional temperature correction (described elsewhere) using the methanol sample (or other temperature calibration samples) derived from two ¹H spectra measured at two different temperatures.

In the case of the NMR Thermometer you just insert the methanol sample (99.8% deuterated) and after setting up the system (tuning/matching/shimming) the sample temperature is displayed immediately. After enabling the NMR Thermometer the sample temperature can be used as target temperature (edte). If the temperature is stable the sample is replaced by, for example, 2 mM sucrose sample in 9:1 H_2O/D_2O and a ¹H spectrum is acquired. Repeating the same procedure at a second spectrometer leads to a very small shift difference corresponding to a temperature difference of about 40 mK:



Figure 6.1: Overlay of a 1H spectrum of 2 mM sucrose (standard sample) measured at 600 MHz (TXI probe) and 800 MHz (TCI CryoProbe).

Changing the sample to a 0.5 mM ubiquitin sample in 9:1 H_2O/D_2O (figure below) nicely shows the precision of the temperature correction obtained by the NMR Thermometer using the methanol sample (99.8% deuterated) as temperature reference.



Figure 6.2: Overlay of 15N HSQC spectra (overview: left part) of 0.5 mM ubiquitin 9:1 H2O/D2O.

6.2 Identical Chemical Shifts for Experiments with Different Heating due to Experimental Conditions (Temperature Compensation)

The superior feature of the NMR Thermometer is the ability to compensate for sample heating inside the sample due to different sources (RF heating, spinning speed, HR MAS).

6.2.1 RF Heating

RF heating inside the sample is caused by, for instance, a spinlock sequence as used in TOCSY-type of pulse sequences, or decoupling as well as CPMG sequences. Such experiments are widely used in biomolecular NMR. As an example we show a 0.5 mM ¹³C- and ¹⁵N-labeled ubiquitin in H₂O/D₂O with sodium acetate-d₃ added as NMR Thermometer substance. In the example the 2D HSQC-planes of 3D NOESY-HSQC, TOCSY-HSQC and CPMG-HSQC experiments are compared. Reference planes were measured without temperature compensation (figure below, left part) and the others with NMR Thermometer in the regulation mode (figure below, right part).



Figure 6.3: Overlay of NOESY-HSQC (blue), TOCSY-HSQC (orange) and CPMG-HSQC (green) spectra of 0.5mM ubiquitin in 95:5% H2O/D2O measured at 800MHz TCI CP.

In the figure above the left side shows the NMR Thermometer disabled (monitoring mode) and the right side the NMR Thermometer enabled (regulation mode).

6.2.2 Heating caused by Spinning Speed (HR-MAS)



Figure 6.4: 1H NMR spectra on a liver sample with sodium acetate added.

In the figure above, different spinning speeds were used (1, 2, 4, 6 and 8 kHz). Left part: NMR Thermometer disabled (monitoring mode); Right part: NMR Thermometer enabled (regulation mode).

An interesting application of the NMR Thermometer is high resolution MAS on biological material. Depending on the spinning speed used in HR-MAS (1 to 8 kHz) the frictional heating in the rotor is different and hence the temperature varies. To compensate for the heating is not only of interest for the comparability of the spectra, but it is also important in order to preserve sensitive sample material (like tissue material). The temperature difference between sample and probe sensor can be as much as 5K in the case of 8 kHz spinning speed.

Applying a spin lock sequence (TOCSY) on top of that could increase the temperature even further. Both heating effects can be compensated for by the NMR Thermometer:



Figure 6.5: Comparison of TOCSY experiments of a liver sample with sodium acetate added measured with HR-MAS at 4 kHz spinning speed.

In the figure above is a comparison of TOCSY experiments of a liver sample with sodium acetate added measured with HR-MAS at 4 kHz spinning speed. The reference 1H spectrum is plotted as projection. Left part: NMR Thermometer disabled (monitoring mode); Right part: NMR Thermometer enabled (regulation mode).

7 Frequently Asked Questions (FAQ)

The lock procedure works well with the solvent, but not with the frequency lock.

- 1. The wrong solvent has been selected and thus the frequency of the compound used for the NMR Thermometer is out of range.
- 2. The concentration of the compound used for the NMR Thermometer is too low. Please check table 1 and 2 which give typical concentrations required.
- 3. The lock power for the thermometer compound is too high or too low. Change the value "Temperature Lock power" in the edlock table and repeat the lock procedure. The value of the temperature lock power typically is a few dB lower than for the field lock power.
- 4. The chemical shift of the compound used for the NMR Thermometer is too closed to the chemical shift of the solvent. Here it can happen that the lock procedure for both, the field and the frequency lock, is working fine on a 700 MHz spectrometer, while it fails on a lower field spectrometer due to the reduced shift difference (in Hz) at a lower field. In that case a different thermometer compound has to be used.

The lock procedure worked well for field and frequency lock, but the temperature regulation of the NMR Thermometer is instable.

• The lock power for the thermometer compound is too high. Reduce the value "Temperature Lock power" in the edlock table and repeat the lock procedure. The value for the temperature power typically is a few dB lower than for the field lock power.

Does the NMR Thermometer work when experiments with pulsed field gradients are performed?

• Yes, like the field lock the NMR Thermometer can be used together with pulsed field gradients. Both, the field and the frequency lock regulation are triggered with lock hold commands of the pulse program. Lock hold commands are standard in all standard pulse programs using pulsed field gradients.

After enabling the NMR Thermometer the temperature starts to increase/decrease or during an experiment with RF heating (see above) temperature is decreasing?

• The chemical shift – temperature values in the edlock table for the particular solvent are probably wrong for instance after creating an .xml file and using a positive sign for the slope instead of a negative one.

8 Contact

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WEEE DE43181702

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