Topspin Setup Acquisition: Bio-NMR

Detlef Moskau

Setup the Acquisition: Flowchart

TopSpin Acquisition setup

Data set:

where to define parameters and store spectra

Sample:

temperature, insert, eject, lock, rotate, shim

Standard Parameter sets:

probe independent parameters

Probe dependent parameters:

pulses and power levels

Customize parameters:

spectral windows, number of scans,…

Optimize parameters:

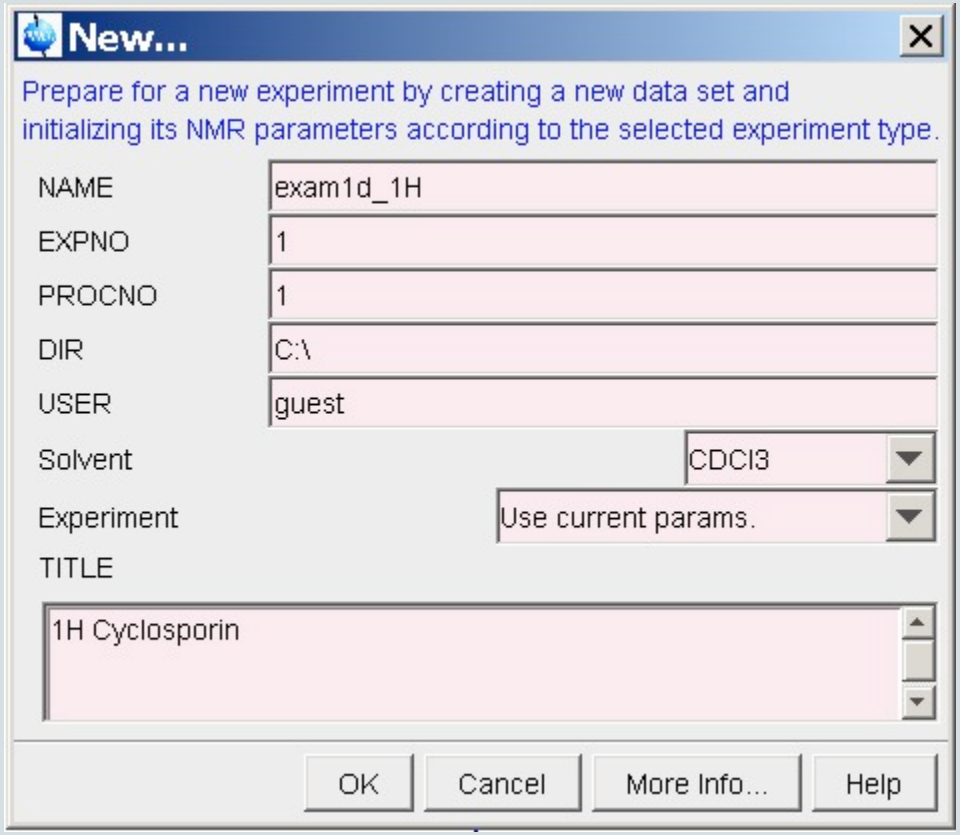
offsets, power levels, receiver gain

Acquisition:

start and stop

TopSpin Data set commands

Data set: where to define parameters and store spectra
new, edc

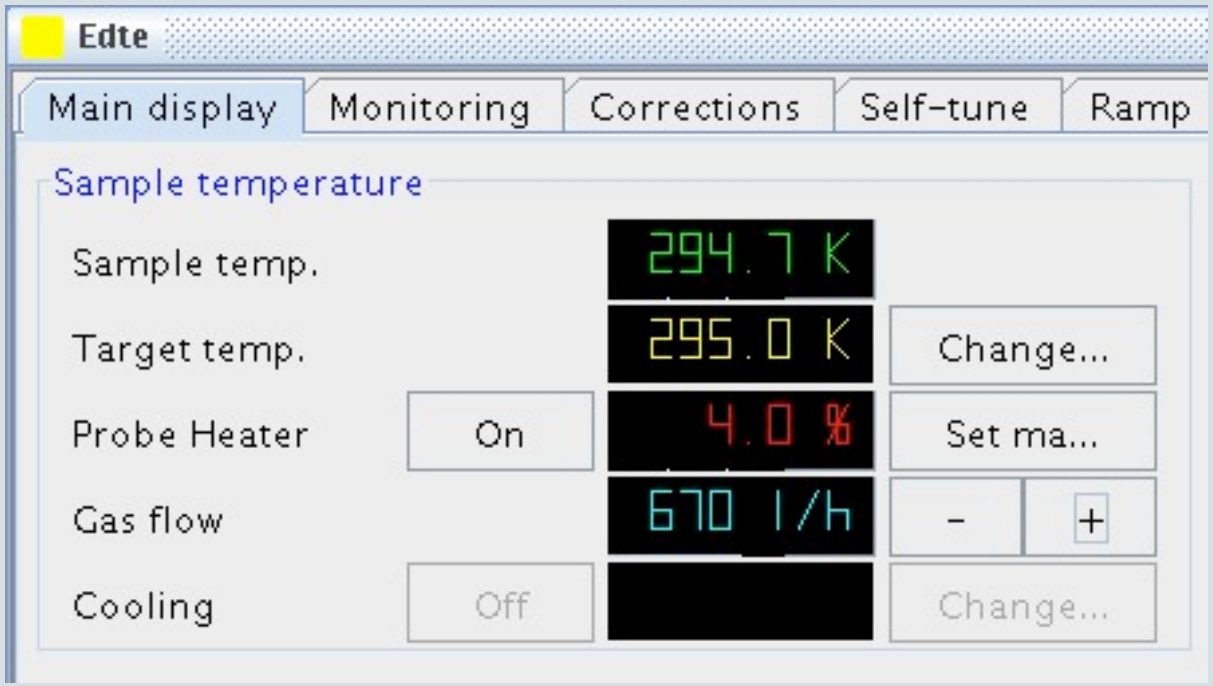


Raw data (FID) is stored under:
C:\data\guest\nmr\exam1d_1H\1
↓ ↓ ↓
USER NAME/PROJECT EXPNO

Processed data stored under:
C:\data\..\exam1d_1H\1\pdata\1
↓
PROCNO

TopSpin Sample

Sample: temperature, insert, eject, lock, rotate, shim
edte (te and teset)



Note for CryoProbe:

- Temperature range: 0° – 50° C (temperature has to be calibrated!)
- Gas flow: 670 l/h

TopSpin Sample

Sample:	temperature,	insert, eject, lock, rotate, shim
	bsmsdisp:	for all functions
	ej, ij:	eject, insert sample
	ro:	start, stop rotation
	lock:	lock
	lock –noauto:	lock on solvent with multiple solvent signals (MeOD)

TopSpin Sample

Sample: temperature, insert, eject, lock, rotate, shim

lock: for all functions

TopSpin Sample

Sample: temperature, insert, eject, lock, rotate,

shim

topshim: latest gradient shimming tool

gradshim: alternative gradient shimming tool

TopSpin Experiment definitions

Standard Parameter sets: probe independent parameters

Probe dependent parameters: pulses and power levels

TopSpin Standard Parameter files

Standard Parameter sets: probe independent parameters

rpar *(example: rpar HNCOGP3D all)*

Standard parameter files contain:

Default parameters for an experiment: pulse program, number of scans, spectra windows, time domain data points, window functions for processing etc.

Standard parameter files do ***NOT*** contain:

Parameters for pulses and power levels

TopSpin Standard Parameter files

How do I interprete the meaning of the parameter file name?:
example: rpar HNCOGP3D all

edpul Pulprog.info

```
;Pulprog.info
;The two-character codes used are the following:


ar     experiment for aromatic residues
at     adiabatic TOCSY
bi     with bird pulse for homonuclear J-decoupling
bp     using bipolar gradients
cc     cross correlation experiment
cp     with composite pulse
ct     constant time
cw     decoupling using cw command
cx     using CLEANEX_PM
dc     decoupling using cpd
……..
```

TopSpin Probe dependent parameters

Probe dependent parameters: pulses and power levels
getprosol

getprosol: load default pulses and power levels according to the current probe and solvent

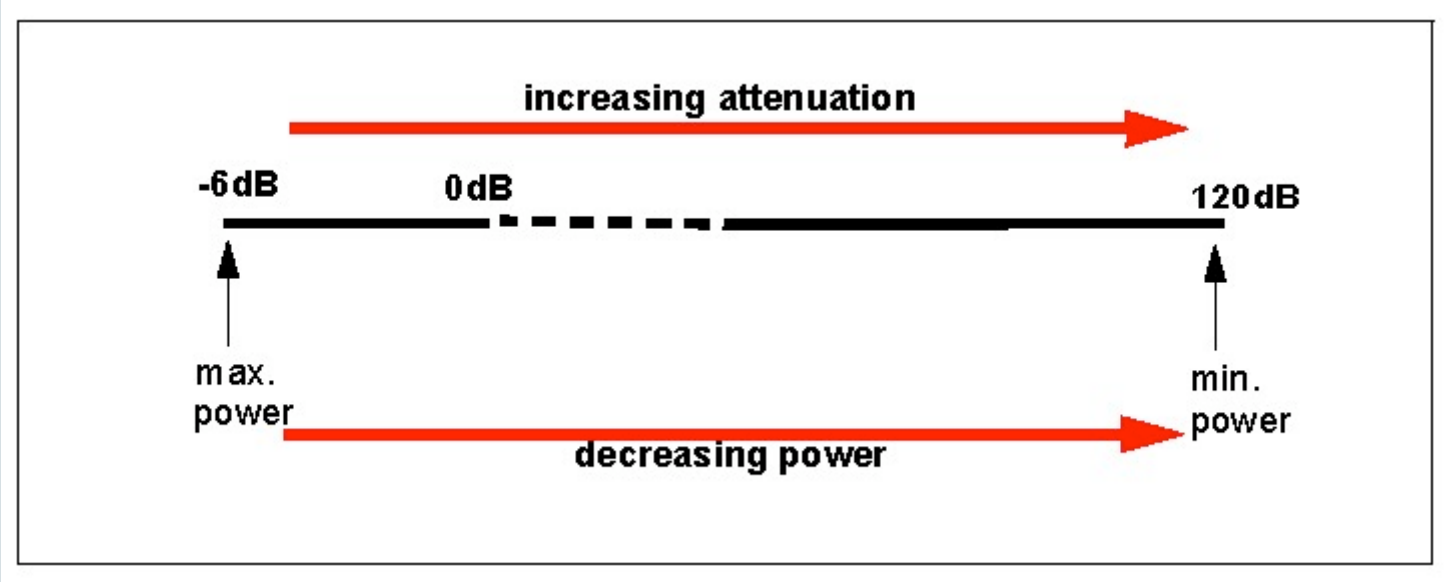
When a pulse does not correspond to the default value, all power levels for pulses of that nucleus can be recalculated for the current data set:

EXAMPLE!: getprosol 1H 10.5us -2.3db

for a 1H pulse of 10.5 usec at -2.3 dB

TopSpin Power level definitions

Power levels are defined as attenuation values in dB:



TopSpin Customizing parameters

Customize parameters: spectral windows, number of scans,..

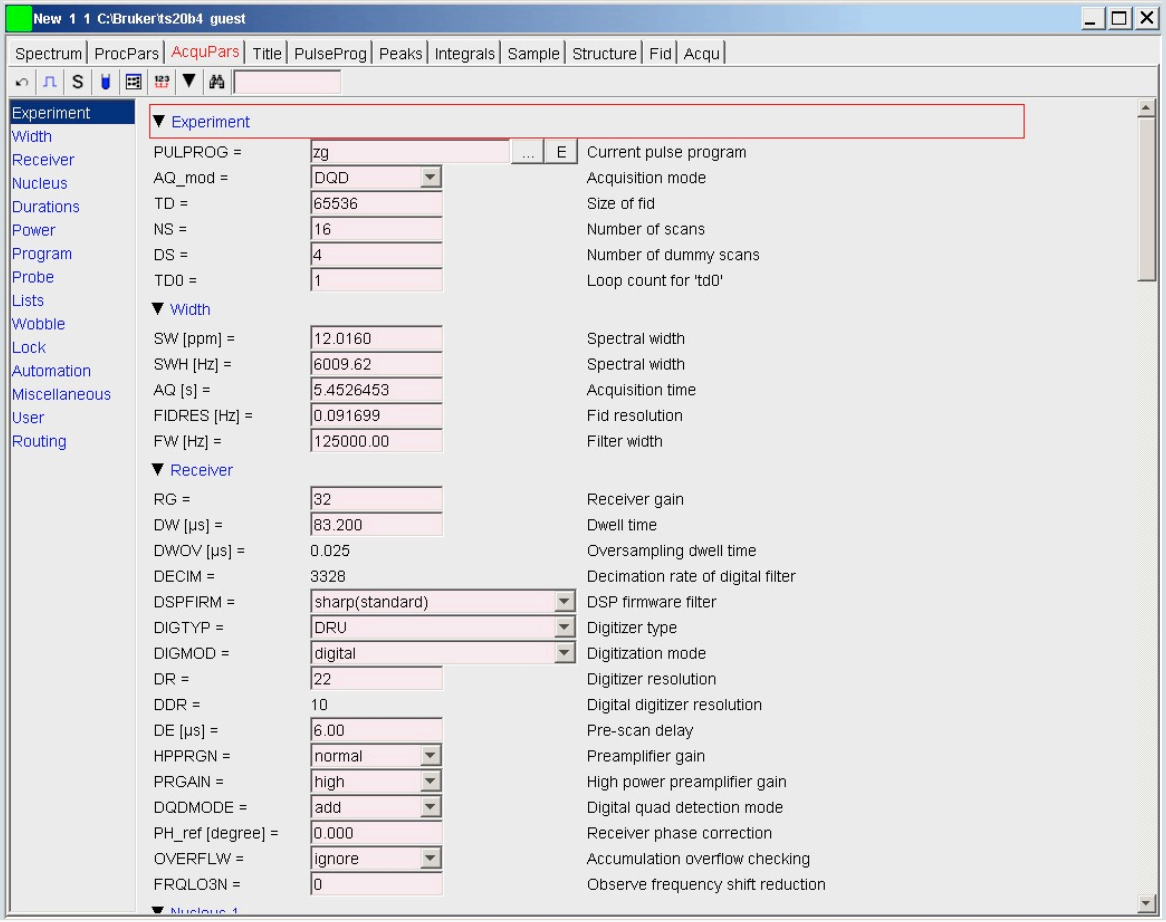
Acquisition:

eda: show all parameters

ased: show parameters related to current pulse program

TopSpin Customizing parameters

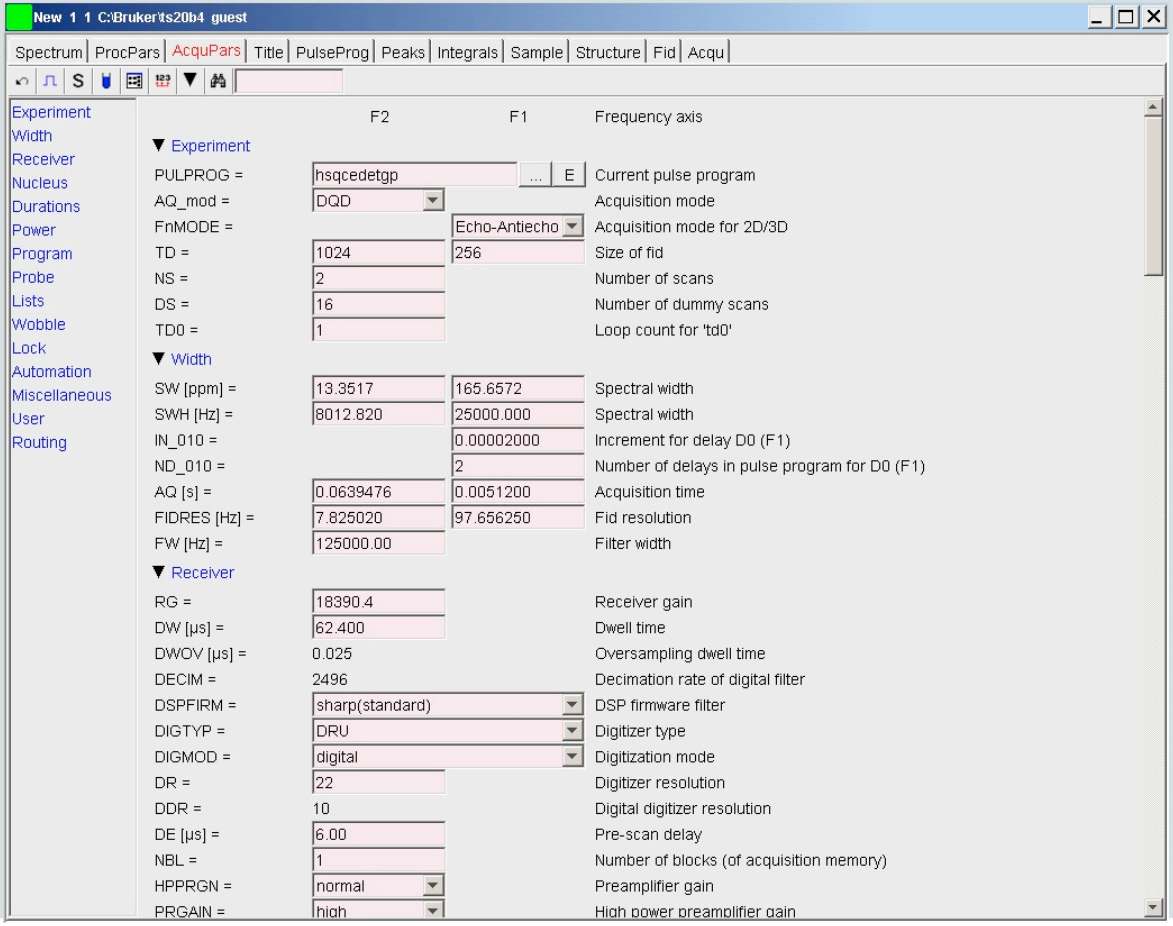
eda: show all parameters



1D-experiments

TopSpin Customizing parameters

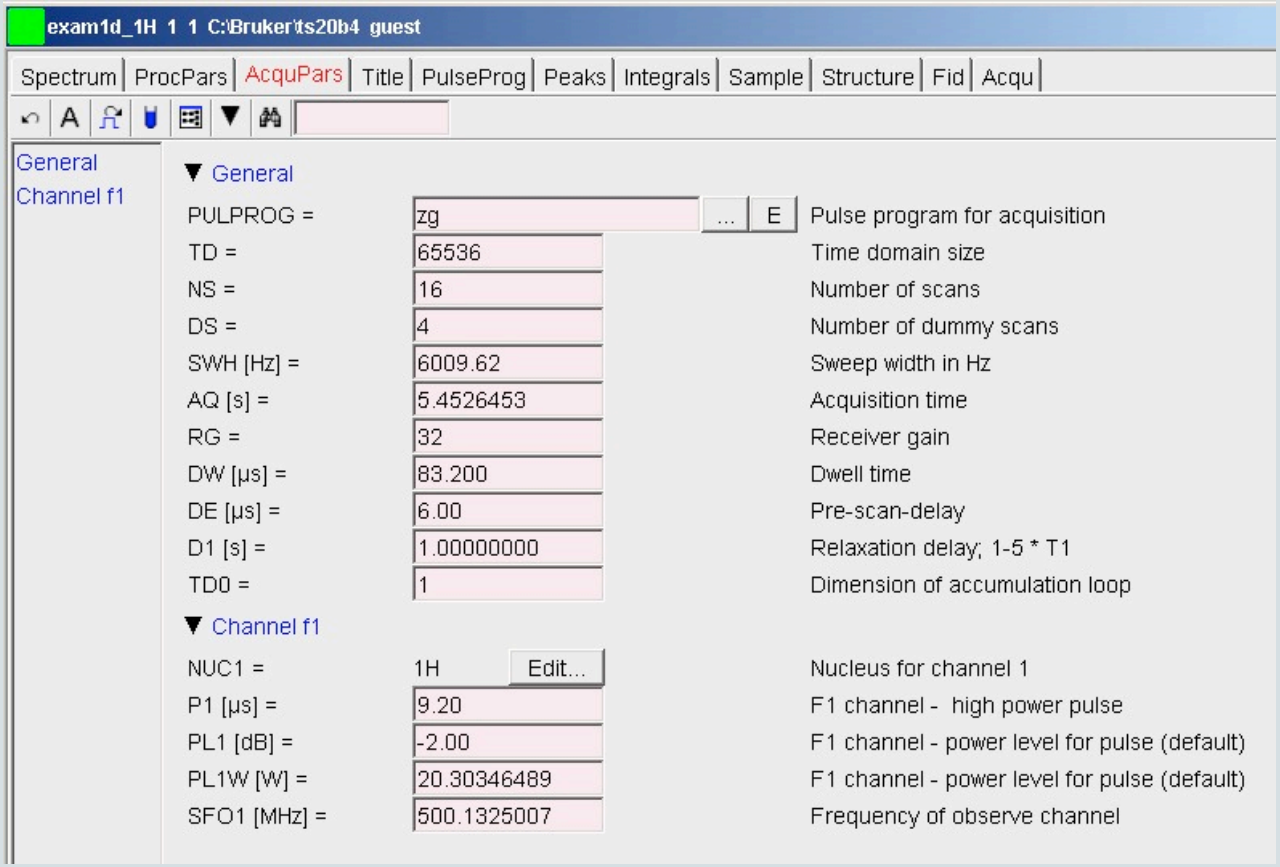
eda: show all parameters



2D-experiments

TopSpin Customizing parameters

ased: show parameters related to current pulse program



TopSpin Customizing parameters

Tuning and Matching of the probe:

wobb: manual mode, no Automatic Tuning and Matching accessory
(VU: w1, w2, w3 or wobb f1, wobb f2, …)

atma: fully automated tuning and matching, all active channels

atmm: manual mode of atma

TopSpin Customizing parameters

Typical parameters which are customized:

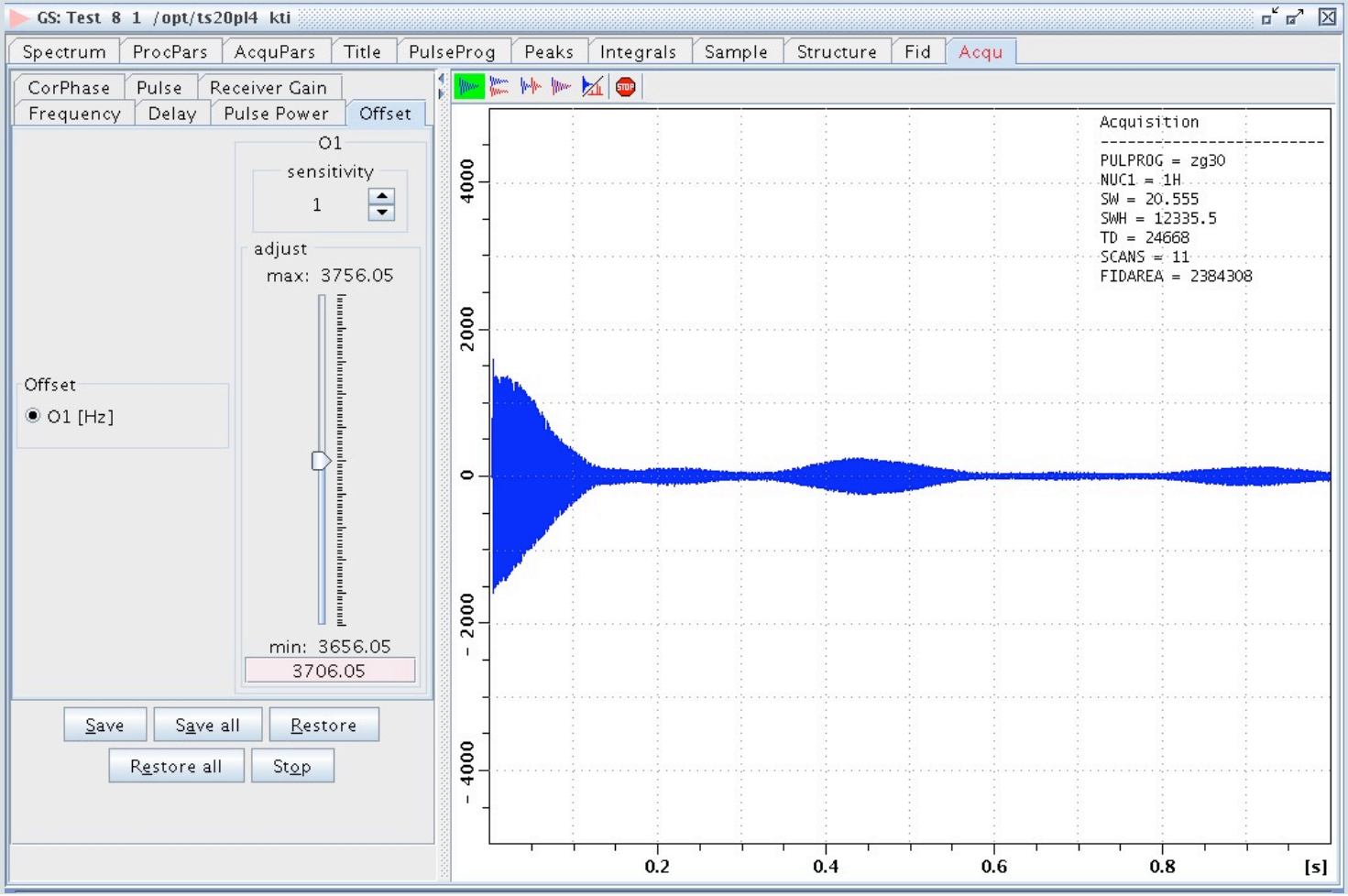
ns:	number of scans
ds:	number of dummy scans
td:	time domain data points
sw:	spectral window [ppm]
swh:	spectral window [Hz]
o1p:	transmitter offset [ppm]
o1:	transmitter offset [Hz]
o2p:	decoupler offset [ppm]
rg:	receiver gain

TopSpin Optimize parameters

Parameters, which need fine tuning (transmitter offset for presaturation, power level for shaped flip-back pulses) can be optimized in the go-setup mode gs.

TopSpin Optimize parameters

go-setup mode gs



TopSpin Acquisition Start and Stop

Acquisition: start and stop

Preparing start of acquisition:

expt	calculate experiment time
rga	automatic receiveer gain adjustment

Start acquisition:

zg	start and overwrite existing FID
go	start and add to current FID (for 1D)

Stop acuisition:

stop	stop immediately. For 1D, FID is lost!
halt	stop after current scan, FID will be saved

Transfer 1D-FID to disk:

tr	required for 1D only

TopSpin Flowchart commands for acquisition

Data set:	new, edc
Sample:	edte, lock, bsmsdisp, topshim, gradshim
Standard Parameter sets:	rpar
Probe dependent parameters:	getprosol
Customize parameters:	ased, eda
Optimize parameters:	wobb, atma, rg, rga, gs
Acquisition:	expt, zg, stop, halt, tr

TopSpin Flowchart commands for acquisition

Data set:	new, edc
Sample:	edte, lock, bsmsdisp, topshim, gradshim
Standard Parameter sets:	rpar
Probe dependent parameters:	getprosol
Customize parameters:	eda, ased
Optimize parameters:	wobb, atma, rg, rga, gs
Acquisition:	expt, zg, stop, halt, tr

Selected list of parameter sets

CBCACONHGPWG3D
CBCACONHGP3D
CCANHGP3D
CCACONHGP3D.2
CCACONHGP3D
CBCANHGPWG3D
CBCANHGP3D
CCCONHGP3D3
CCCONHGP3D2
CCCONHGP3D1
CCCONHGP3D
CCANHGP3D.2
CCCONHGPWG3D3
CCCONHGPWG3D2
DIPSIHSQCF3GPSI3D
HCACOGPJC3D
HCACOGP3D
HBHANHGPWG3D
HBHANHGP3D
HBHACONHGPWG3D
HBHACONHGP3D
HACAHBCOSYGP3D
HCCCONHGPWG3D3
HCCCONHGPWG3D2
HCCCONHGP3D3
HCCCONHGP3D2
HCCCONHGP3D1
HCANNHGP3D
HCAHBCOSYGP3D
HCCHECOSGP3D
HCCHDIGP3D2

HCCHDIGP3D *HCCH-TOCSY*
HCCHCOSYGP3D
HCCHCOGP3D
HNCACBGPJC3D
HNCACBGP3D
HNCACOSYGP3D
HNCACOGPWG3D
HNCACOGP3D
HNCACBGPWG3D
HNCAH3D
HNCAGPWG3D *watergate*
HNCAGP3D
HNCAECOSGP3D2
HNCAECOSGP3D
HNCADQZQGP3D
HNCOCACGP3D
HNCOCACBGPWG3D
HNCOCACBGP3D
HNCOCACAGP3D
HNCAJCGP3D
HNCAHAGP3D
HNCOECOSGP3D
HNCOCOGP3D
HNCOCGP3D
HNCOCAGPWG3D
HNCOCAGP3D
HNHBECOSGP3D
HNHAGP3D
HNCOGPWG3D
HNCOGPHB3D2
HNCOGPHB3D

HNCOGP3D
HNNHDIGP3D
HNHBGP3D
MLEVHSQCETGP3D
MLEVHSQCETF3GP3D

NA = Nucleic Acid
NA_HCCHFWDIECGP3D
NA_HCCHECGP3D
NA_GHCCHFWDIGP3D
NA_HCPDIETGPSI3D
NA_HCNETGPSISP3D
NA_HCNETGPSI3D
NA_HCCNHDIGPWG3D
NA_HCCHFWDIGP3D
NA_HCPETGPSI3D

NOESYHSQCF3GP193D
NOESYHSQCETGP3D
NOESYHSQCETF3GP3D
NOESYHSQCGPSM3D
NOESYHSQCGPSISM3D
NOESYHSQCFPF3GPSI3D
NOESYHSQCF3GPSI3D

TR = TROSY
TRHNCACOGP3D
TRHNCACBGP3D
TRCBCANHGP3D
TRCBCACONHGP3D
TRHNCAGP3D2.2
TRHNCAGP3D.2
TRHNCAGP3D2 *latest version*
TRHNCAGP3D
TRHNCOGP3D
TRHNCOCAGP3D

CCACONHGP2H3D
CCCONHGP2H3D
CCANHGP2H3D
HNCACOGP2H3D
HNCACBGP2H3D
HNCACOGP2H3D.2
HNCAGP2H3D ^{2}H-decoupled
HNCOCAGP2H3D
HNCOCACBGP2H3D
HNCOGP2H3D
TRHNCACBGP2H3D
TRHNCAGP2H3D2
TRHNCAGP2H3D
TRHNCACOGP2H3D
TRHNCOGP2H3D
TRHNCOCAGP2H3D
TRHNCOCACBGP3D
TRHNCOCACBGP2H3D

TopSpin Standard Parameter files

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edpul Pulprog.info

```
;Pulprog.info
;The two-character codes used are the following:


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ct     constant time
cw     decoupling using cw command
cx     using CLEANEX_PM
dc     decoupling using cpd
……..
```

Example: rpar HNCA*

HNCAGP3D gradient selected

HNCAGPWG3D watergate version

HNCAGP2H3D ^{2}H-decoupled / ^{1}H-suppression by 180°-pulses

TRHNCAGP3D2 TROSY / ^{1}H-suppression by 180°-pulses

TRHNCAETGP3D TROSY / echo-antiecho / ^{1}H-suppression by 180°-pulses

(JMR 144 (2000) 123)

TRHNCAGP2H3D TROSY / ^{2}H-decoupled / for 100% deuterated proteins

TRHNCAGP2H3D2 TROSY / ^{2}H-decoupled / for partially deuterated proteins / ^{1}H-suppression by 180°-pulses

Other triple resonance parameter sets

HNCOCAGP3D
HNCOCAGPWGD
HNCOCAGP2H3D
TRHNCOCAGP3D
TRHNCOCAGP2H3D

HNCOCACBGP3D „transfer“
HNCOCACBGPWG3D
HNCOCACBGP2H3D
TRHNCOCACBGP3D
TRHNCOCACBETGP3D echo-antiecho
TRHNCOCACBGP2H3D ^{2}H-decoupled

HNCACBGP3D „out&back“/ for larger proteins
HNCACBGPWG3D
HNCACBIGPWG3D intra-residue only
HNCACBGP2H3D
TRHNCACBGP3D
TRHNCACBIGP3D intra-residue only
TRHNCACBGP2H3D

HAHBCONHGP3D
HAHBCONHGPWG3D
TRHAHBCONHGP3D

HAHBNHGP3D
HAHBNHGPWG3D
TRHAHBNHGP3D

HNCOGP3D
HNCOGPWG3D watergate
TRHNCOGP3D TROSY

HNCACOGP3D
HNCACOGPWGD
HNCACOGP2H3D ^{2}H-decoupled
TRHNCACOGP3D
TRHNCACOGP2H3D

CBCACONHGP3D „transfer“ / for smaller proteins
CBCACONHGPWG3D
TRBCACONHGP3D

CBCANHGP3D „transfer“ / for smaller proteins
CBCANHGPWG3D
TRBCANHGP3D
TRBCANHETGP3D
SEQTRBCANHGP3D sequential TROSY

HCCCONHGP3D2 H(CCCO)NH
HCCCONHGPWG3D2

HCCCONHGP3D3 (H)CC(CO)NH
HCCCONHGPWG3D3
CCCONHGP2H3D 13C-start

Other useful parameter sets

^{13}C-correlated experiments

HCCHDIGP3D	HCCH-TOCSY
HCCHDIGP3D2	(H)CCH-TOCSY
HCCHCOGP3D	HCCH-COSY
NOESYHSQCETGP3D	3D-NOESY-HSQC

^{15}N-correlated experiments

NOESYHSQCF3GPSI3D	15N-NOESY-HSQC sensitivity enhanced
NOESYHSQCF3GP193D	15N-NOESY-HSQC with 3919 watergate
NOESYTZGP3D	15N-NOESY-ZQ-TROSY for large proteins

Other useful parameter sets

2D Homonuclear-correlated experiments

NOESYPHPR	**NOESY, phase sensitive, presat**
NOESYGPPH19SW	**NOESY, phase sensitive, 3919 WATERGATE**
MLEVPHPR	**TOCSY, phase sensitive, presat**
MLEVGPPH19SW	**TOCSY, phase sensitive, 3919 WATERGATE**
DIPSI2GPPH19	**TOCSY, phase sensitive, 3919 WATERGATE, DIPSY2 spin lock**

Where is the parameter file for a NOESY with Excitation Sculpting?

Not all versions of an experiment are covered by parameter files

Start from a related experiment and:

- **change the pulse program: pulprog noesyesgpph instead noesyphpr**
- **check corresponding parameters with ased**

Other useful parameter sets

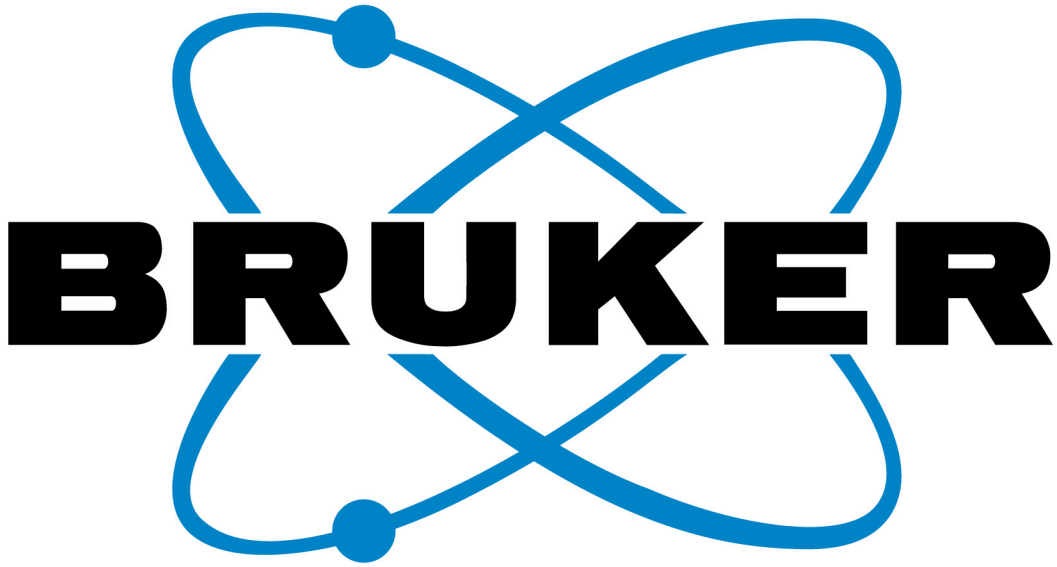
2D Heteronuclear-correlated experiments

13C:	HSQCETGP	HSQC, echo-antiecho
	HSQCETGPSISP	HSQC, echo-antiecho, sensitivity improved, adiabatic ^{13}C inversion
	HSQCETGPSISP.2	HSQC, echo-antiecho, sensitivity improved, adiabatic ^{13}C inversion and refocussing
15N;	HSQCETF3GP	HSQC, echo-antiecho
	HSQCETF3GPSI	HSQC, echo-antiecho, sensitivity improved

Quick Guide

Let‘s run a 3D CBCACONH with WATERGATE

edc	new data set
rpar CBCACONHGPWG3D all	read parameter file
lock	lock on solvent
atma f1	tune and match 1H channel automatically
topshim	gradient shimming
pulsecal	determine 1H pulse automatically
getprosol 1H x us y db	set all pulses, recalculate all 1H pulses
ased	all pulse parameters and delay correct?
eda	check NS, spectral windows and number of increments
rg	typically = 200
expt	check total experiment time
zg	start the acquisition



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