JHU NMR Experiment Quick Guide

1-Dimensional Experiments

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Proton	proton	zg	**R400/N400**: 01m 50s **R300**: 02m 21s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
16	2	1	1	65536

Experiment Setup

- Use getprosol to read in proper pulse parameters

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Proton-solv_supp	--	zgcppr	**R400/N400**: 10m 49s **R300**: 13m 39s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
128	2	1	1	65536

Experiment Setup

- Use getprosol to read in proper pulse parameters
- Move o1p to be on-resonance with the solvent peak

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Deuterium	deuterium	zg2h	**R400/N400**: 04m 54s **R300**: 04m 02s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
64	4	2	1	16384

Experiment Setup

- This setup is through the lock channel, sensitivity will be lower compared to detection through the broad band channel.
- Open a *Proton* experiment and lock, tune, shim on the sample
- Once shimmed, read in the *Deuterium* parameters and perform rga (**DO NOT reshim or tune**)

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Boron11	boron11	zgpg30	**R400/N400**: 06m 06s **R300**: 07m 56s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
256	4	0.1	1	65536

Experiment Setup

- Use getprosol to read in proper pulse parameters for decoupling

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Carbon13-apt	c13apt	jmod	**R400/N400**: 14m 45s **R300**: 16m 42s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
64	4	2	4	65536

Experiment Setup

- Set cnst2 = J(XH), *J*-coupling between X and H.
  - If primarily aliphatic protons: cnst2=125.
  - If primarily aromatic protons: cnst2 = 150.
  - Default value: cnst2 = 140.
- Set cnst11=1

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Carbon13-dec30	c13cpd	zgpg30	**R400/N400**: 14m 49s **R300**: 16m 46s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
64	4	2	4	65536

Experiment Setup

- Use getprosol to read in proper pulse parameters for decoupling

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Carbon13-dept90	-	Udeft_13C	**R400/N400**: 14m 57s **R300**: ----------

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
64	8	2	4	65536

Experiment Setup

- Set cnst2 = J(XH), *J*-coupling between C and H.
  - Default value: cnst2 = 140.

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Carbon13-dept135	-	Udeft_13C	**R400/N400**: 14m 57s **R300**: ----------

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
64	8	2	4	65536

Experiment Setup

- Set cnst2 = J(XH), *J*-coupling between C and H.
  - Default value: cnst2 = 140.

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Carbon13-udeft	-	Udeft_13C	**R400/N400**: 31m 02s **R300**: 35m 22s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
64	0	3.5	4	65536

Experiment Setup

- Do not change d1
- d20 is an initial delay of the sequence which will only apply once. Should be set to be $5\times T_1$. Default value: d20 = 240s

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Nitrogen15	N15zg	zg	**R400/N400**: 06m 30s **R300**: 06m 39s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
32	4	10	1	32768

Experiment Setup

- Use getprosol to read in proper pulse parameters

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Nitrogen15-dec	N15cpd	zgig	**R400/N400**: 06m 30s **R300**: 06m 40s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
32	4	10	1	32768

Experiment Setup

- Use getprosol to read in proper pulse parameters for decoupling

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Nitrogen15-inept	N15inept	ineptrd	**R400/N400**: 08m 40s **R300**: 08m 53s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
32	16	10	1	32768

Experiment Setup

- Set cnst2 = J(XH), *J*-coupling between N and H.
  - Default value: cnst2 = 90.
- Set cnst11=6

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Silicon29-dept	si29dept	dept45	**R400/N400**: 51m 02s **R300**: 53m 01s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
128	4	5	4	65536

Experiment Setup

- Set cnst2 = J(XH), *J*-coupling between Si and H.
  - Default value: cnst2 = 7

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Silicon29-udeft	-	Udeft_29Si	**R400/N400**: 49m 52s **R300**: ----------

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
128	0	3.5	4	65536

Experiment Setup

- Do not change d1
- d20 is an initial delay of the sequence which will only apply once. Should be set to be $5\times T_1$. Default value: d20 = 90s

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Phosphorus31-dec30	p31cpd	zgpg30	**R400/N400**: 05m 29s **R300**: 05m 31s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
128	4	2	1	65536

Experiment Setup

- Use getprosol to read in proper pulse parameters for decoupling

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Selenium77	s77_1D	zg	**R400/N400**: 05m 56s **R300**: ----------

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
1024	4	0.1	1	16384

Experiment Setup

- Use getprosol to read in proper pulse parameters

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Selenium77-dec	s77cpd	zgpg	**R400/N400**: 06m 27s **R300**: ----------

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
1024	4	0.1	1	16384

Experiment Setup

- Use getprosol to read in proper pulse parameters for decoupling

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Fluorine19	F19zg	zgflqn	**R400/N400**: ---------- **R300**: 04m 11s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
64	4	1	1	131072

Experiment Setup

- Use getprosol to read in proper pulse parameters

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
Fluorine19-dec	F19cpd	zgfhigqn	**R400/N400**: ---------- **R300**: 04m 11s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
64	4	1	1	131072

Experiment Setup

- Use getprosol to read in proper pulse parameters and decoupling parameters

1-Dimensional Correlation Experiments

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
NOESY-sel_1D	selNOE_1D/1D_gradNOE	selnogp	**R400/N400**: 10m 58s **R300**: 13m 46s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
32	4	2	4	32768

Experiment Setup

- d8 = mixing time.
  - Small molecules: d8 ≈ T_1
  - Large molecules: d8 ≈ 0.5T_1
  - Default d8 = 800ms
- spoffs2 = offset frequency for selective shaped pulse
  - set to the frequency difference between the center (**o1**) and the location of the desired peak to be analyzed.

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
NOESY-pr_1D	solvent_suppression	noesypr1d	**R400/N400**: 10m 45s **R300**: 13m 45s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
32	4	2	4	32768

Experiment Setup

- d8 = mixing time.
  - Small molecules: d8 ≈ T_1
  - Large molecules: d8 ≈ 0.5T_1
  - Default d8 = 300ms

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
TOCSY-sel_1D	selTOCSY_1D	selmlgp	**R400/N400**: 02m 51s **R300**: 03m 18s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	1D Loop (td0)	FID Size (td, F2)
32	4	2	1	32768

Experiment Setup

- d9 = mixing time.
  - Default: d9 = 60 ms
- spoffs2 = offset frequency for selective shaped pulse
  - set to the frequency difference between the center (**o1**) and the location of the desired peak to be analyzed.

2-Dimensional Experiments

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
COSY-dqf	-	cosydfph	R400/N400: 07m 36s R300: 08m 05s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
1	16	1.5	2048	256

Experiment Setup

- Use getprosol to read in proper pulse parameters

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
COSY-grad	cosygpsw	cosygpqf	R400/N400: 07m 39s R300: 08m 10s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
1	16	1.5	2048	256

Experiment Setup

- Use getprosol to read in proper pulse parameters

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
COSY-HF	HFcorrelation	hfcoqfqn	R400/N400: ---------- R300: 33m 25s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
16	16	1.0	2048	256

Experiment Setup

- Use getprosol to read in proper pulse parameters

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
NOESY	NOESY	noesyph	R400/N400: 1h 25m 05s R300: 1h28m 50s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
8	16	2	2048	256

Experiment Setup

- d8 = mixing time.
  - Small molecules: d8 ≈ T_1
  - Large molecules: d8 ≈ $0.5T_1$
  - Default d8 = 300ms

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
NOESY-HF	HFnoesy	hoesyfqph.cdm	R400/N400: ---------- R300: 33m 25s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
16	16	1.0	4096	256

Experiment Setup

- d8 = mixing time.
  - Default d8 = 400ms

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
NOESY-pr	noesypr	noesyphpr	**R400/N400**: 1h 26m 05s **R300**: 1h 29m 50s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
8	16	2	2048	256

Experiment Setup

- d8 = mixing time.
  - Small molecules: d8 ≈ T_1
  - Large molecules: d8 ≈ 0.5T_1
  - Default d8 = 300ms

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
NOESY-grad	-	noesygpph	**R400/N400**: 1h 06m 54s **R300**: 1h 28m 16s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
16	16	0.5	2048	256

Experiment Setup

- d8 = mixing time.
  - Small molecules: d8 ≈ T_1
  - Large molecules: d8 ≈ 0.5T_1
  - Default d8 = 300ms

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
TOCSY	TOCSY	mlevph	**R400/N400**: 1h 16m 42s **R300**: 1h 20m 36s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
8	16	2	2048	256

Experiment Setup

- d9 = mixing time, longer the delay further the coupling distance
  - Default: d9 = 60 ms
- spoffs2 = offset frequency for selective shaped pulse
  - Set to the frequency difference between the center (**o1**) and the location of the desired peak to be analyzed.

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
HSQC-ed	edited-hsqc	hsqcedetgp	**R400/N400**: 15m 05s **R300**: 15m 42s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
2	16	1.5	2048	256

Experiment Setup

- cnst2 = J(XH)
  - Default: cnst2 = 145 Hz
- d21 = multiplicity selection by setting to 1/(2J(XH))

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
HMBC-grad	gradientHMBC	hmbcgplpndqf	**R400/N400**: 1h 07m 18s **R300**: 1h 11m 51s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
16	16	1.5	4096	128

Experiment Setup

- Set cnst2 and cnst13 to the short and long range X-H J-couplings
- cnst2 = J(XH)
  - Default: cnst2 = 145 Hz
- cnst13 = J(XH) long range couplings
  - Default: cnst13 = 10 Hz

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
HMBC-j_ed	j-HMBC	hmbcetgpl3nd	**R400/N400**: 1h 25m 11s **R300**: 1h 29m 17s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
16	16	2	4096	128

Experiment Setup

- Set cnst6 and cnst7 to the minimum and maximum X-H 1-bond J-couplings
- cnst6/cnst7 = 1J(XH)
  - Default: cnst6 = 110 Hz
    Cnst7 = 170 Hz
- cnst13 = J(XH) long range couplings
  - Default: cnst13 = 10 Hz

Current *rpar* Name	Previous *rpar* Name	Pulse Sequence	Experimental Time
H1-Kinetics	-	zg2dcppr	**R400/N400**: 10h 40m 03s **R300**: 10h 40m 04s

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
8	0	10	65536	128

Experiment Setup

- d20 = delay between experiments
  - Default : d20 = 300s

Previous *rpar* Name	Current *rpar* Name	Pulse Sequence	Experimental Time
DOSY-ledbpgp2s	-	ledbpgp2s	**R400**: varies **R300**: ---------

Scans (ns)	Dummy Scans (ds)	Recycle Delay (d1)	FID Size (td, F2)	2D Slices (td, F1)
8	0	**	16384	32

Experiment Setup

- Setup and acquire a standard proton experiment.
  - Set the sw and center your data in the spectrum so that there is approximately 10% of clear baseline (no signal) on both sides signal region.
- Measure the T_1 of your sample (see T_1 measurement notes) and set the d1 = 5×T_1 with respect to the longest relaxation measurement.
- Read in the DOSY-ledbpgp2s experiment
- Spin your sample at 20 Hz
- In the command line enter the following to start the experiment: **xau dosy 5 80 32 l y y**