PULSE PROGRAM CATALOGUE: I. 1D & 2D NMR EXPERIMENTS

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TOPSPIN v3.0
NMRGuide

UAB

15-04-2010
Part Number --------

VOLUME I: 1D & 2D NMR EXPERIMENTS

NMR Building Blocks

Basic Elements

Filters

Selective Excitation Elements

Solvent Suppression Elements

Evolution Periods

Homonuclear Mixing Blocks

1H-to-X Transfer

X-to-1H Transfer

Miscelaneous Elements

Triple-Resonance Elements

BRUKER
PULSE PROGRAM
CATALOGUE

NMRGuide

INTRODUCTION

BRUKER
Pulse Program
Catalogue

written by Teodor Parella

This catalogue presents the pulse sequence diagram for all standard pulse programs included by default in TOPSPIN v3.0. This information is part of NMRGuide, which is also available for BRUKER AVANCE spectrometers.

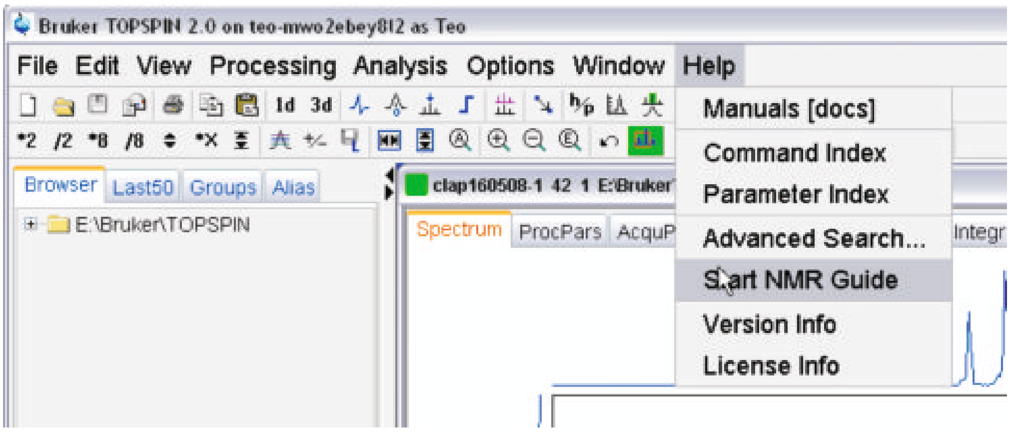
These pulse programs are located in the

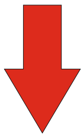
/TOPSPINHOME/exp/stan/nmr/lists/pp/

directory after conventional installation using **expinstall** and they can be visualized directly into the TOPSPIN program from the PulseProg section. Otherwhise, alternative pulse program sequence graphical display is also available using the **showpp** program.

For more details on pulse programs, parameter sets, tutorials, experiment descriptions, bibliographic references and other related information, please refer to the electronic version of NMRGuide.

Starting NMRGuide ...

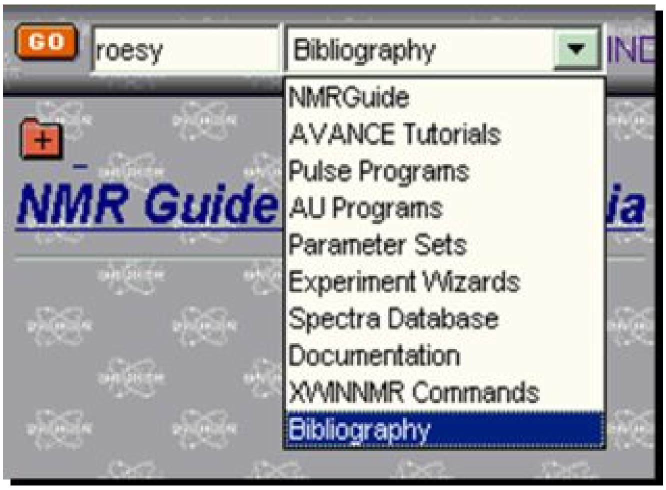




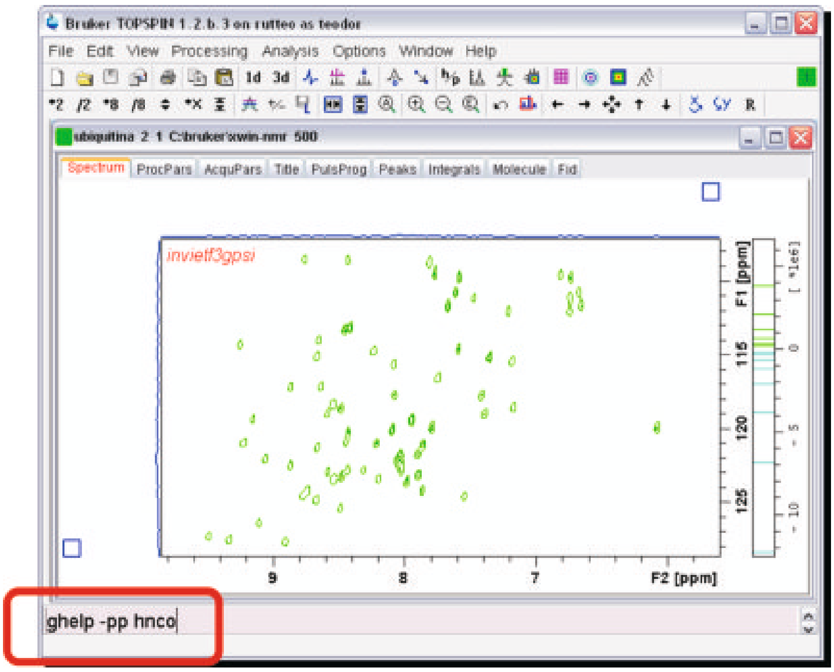
More Info using NMRGuide: The ghelp search tool

The **ghelp** program allows the direct user interaction with NMRGuide from the TOPSPIN command line. Otherwhise, select NMRGuide from the Help menu to open the Explorer browser.
The ghelp optionsb are:

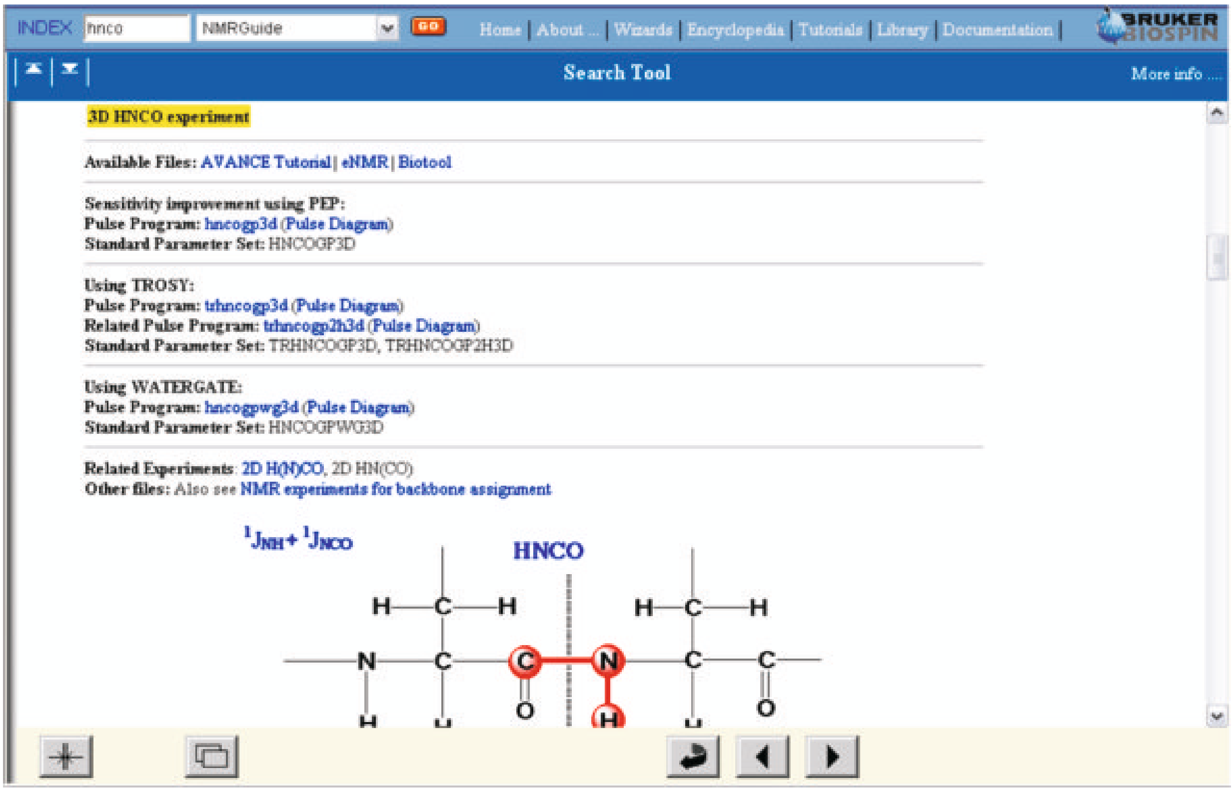
-tut: Search into AVANCE Tutorials
-pp: Search into pulse programs
-au: Search into AU programs
-par: Search into parameter sets
-exp: Search into Experiment Wizards
-spec: Search into Spectra Database
-doc: Search into Bruker Documentation
-bib: Search into Bibliography
-cmd: Search into TOPSPIN commands



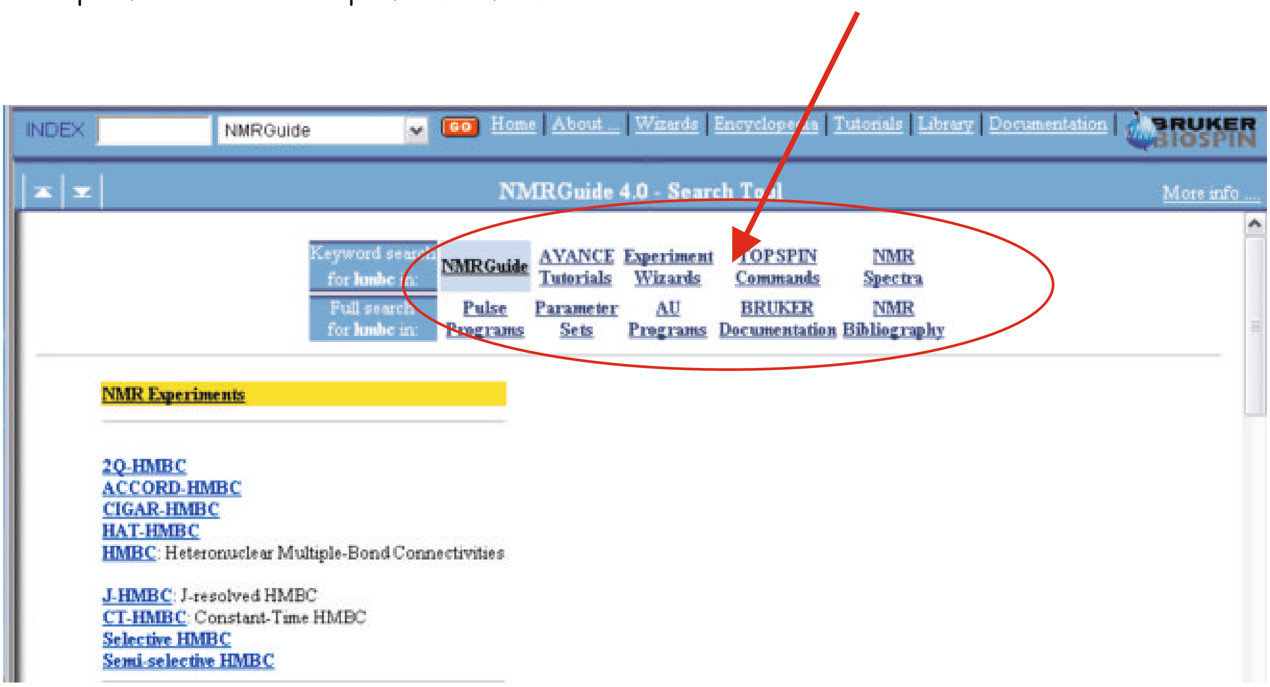
For instance, if you want information about pulse programs related to "HNCO" you can type "ghelp -pp hnco" into the command line as shown:



The output looks like this:



and a browser window will be automatically opened with the keywords found in the NMRGuide database. Then, further specific search can be performed from the buttons:



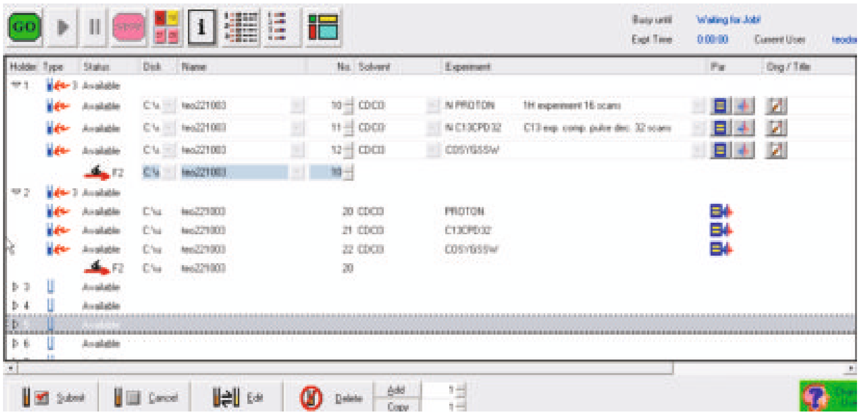
AUTOMATION INTO TOPSPIN

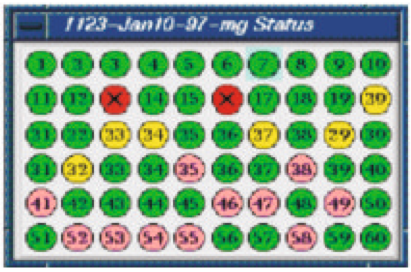
Several Options for automated Acquisition/Processing into TOPSPIN:

1. Using Standard Parameter Sets (**rpar** and **getprosol**)
2. Using macros (**edmac**)
3. Using Guides
4. Using ButtonNMR (**buttonnmr** & **butselnmr**)
5. Using ICONNMR (**iconnmr**)

Other Automation acquisition and processing protocols using
AU Programs. (**edau** & xau)

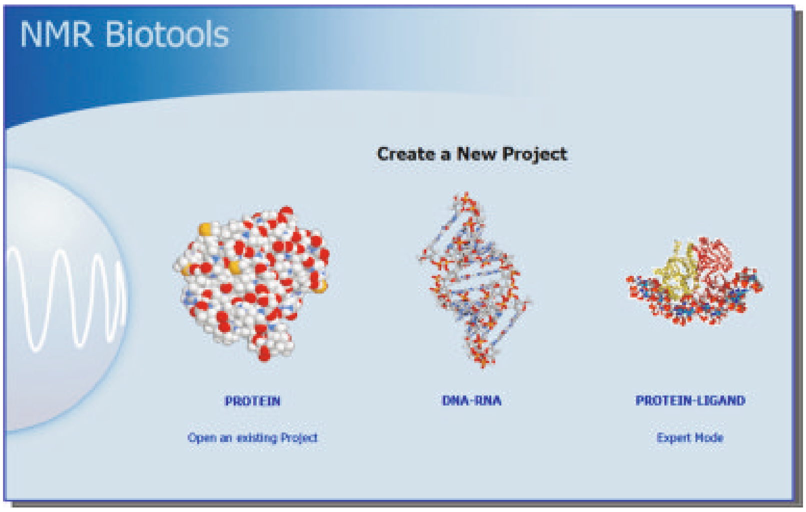
Multiple sample/Multiple Experiment definition





Real-time sample status also available via www

<u>Automation using NMR BIOTOOL:</u> Automated Biomolecular NMR using a guided, full-automated process



Automation using AU Programs:

Au programs is the best option to perform full automated processes.
See NMRGuide and "AU Reference manual" for more information.

Some Features:
1. More than 400 programs by default
2. Written in C and python.
3. TOPSPIN commands are fully compatible
4. Edited with **edau** and executed automatically with the corresponding name.
6. Included into automated Acquisition and Processing modules under AUNM. and AUNMP.

Examples:

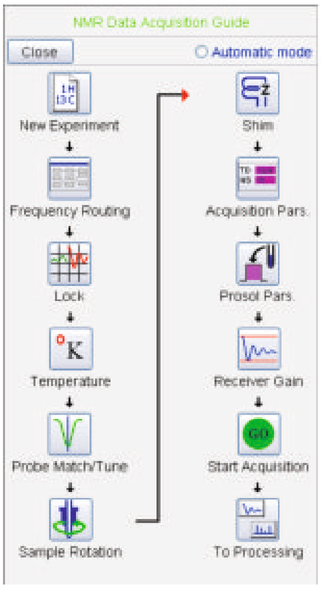
topshim: Automatic Shimming process
pulsecal: Performs automatic 90 proton pulse calibration
multizg: Performs multiple acquisition
multi_zgvd: Performs multiple acquisition at predefined times
multi_zgvt: Performs multiple acquisition at predefined temperatures
calcphhomo: Performs automatic phase calibration in 2D homonuclear experiments

Automation using MACROS:

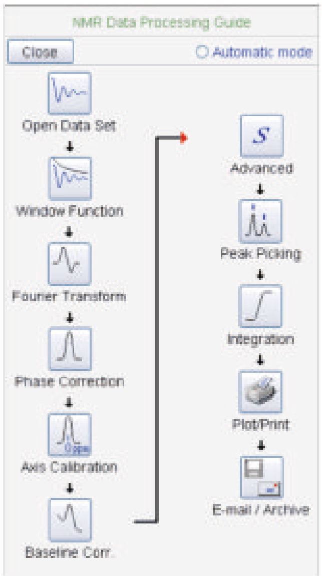
To avoid much and repetitive typing, the use of macros can be highly useful. Macros are invoked with the edmac command and are very simple to create. As an example, we create a macro called proton to acquire and process automatically a conventional 1D proton spectrum:

1. Type **edmac proton**
2. Write the following sequential commands
3.Execute the macro simply typing the name of the macro. In this case, type **proton** and the macro will ask the necessary inputs and then will execute acquisition and processing commands.

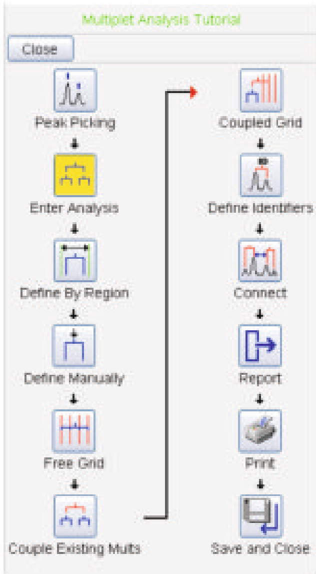
```
edc
rpar teo_proto all
ii
ns
rga
zg
ft
apk
abs
```

aqguide

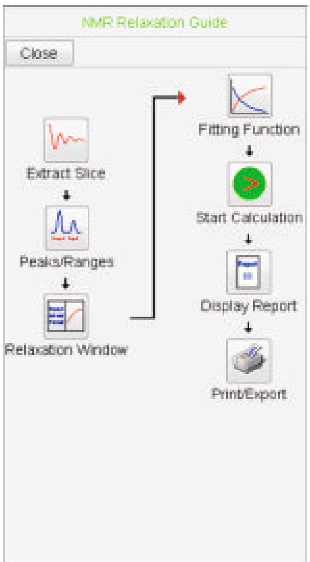


prguide

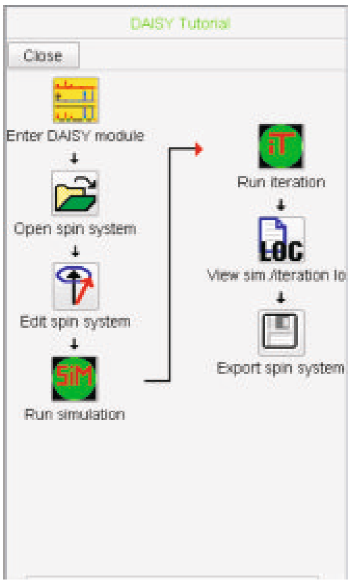


managuide

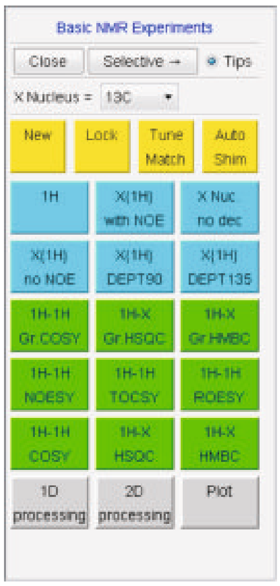
solaguide



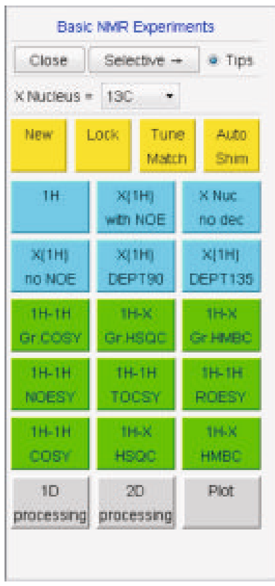
t1guide



daisyguide



buttonnmr



butselnmr

1. Sample Preparation

- Filter the sample.
- Use clean NMR tubs of optimum quality.
- Use the required volume (0.6 ml for 5mm NMR tubes).
- Sample concentration as a function of NMR experiment sensitivity.

2. NMR Preparation

2.1 Put the sample into the magnet.

2.2 Select solvent.

2.3 Spin the sample.

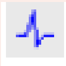
2.3 Shimming.

2.4 Tuning and Matching .

3. Data Acquisition.

edc	: Define the file
rpar "filename" all	: Read starting parameter set
getprosol	: Get pulses and power levels
ased (or eda)	: Check original parameters and change if required
rga	: Adjust detector
zg	: Start Acquisition

4. Data processing

	Subroutine	1D	2D
Fourier Transformation		ft	xfb (xf2+xf1)
Phase Correction		apk	xfbp (xfb+pk)
Baseline Correction		abs	abs2 + abs1
Automatic Referencing		sref	

5. Data Analysis

 Enter peak picking mode

 Enter integration mode

 Enter multiple display mode

 Enter distance measurement mode

1. NMR Strategy

Molecular Size
Natural Abundance vs Isotopic Labeling
Structural vs Dynamic studies

2. Which NMR Experiment?

Sample Concentration
Available Probehead
Magnetic Field
NMR Parameters →
Which Nucleus?

Chemical Shift
Coupling Constants: J/D
NOE/ROE
Diffusion
Relaxation
Chemical Exchange

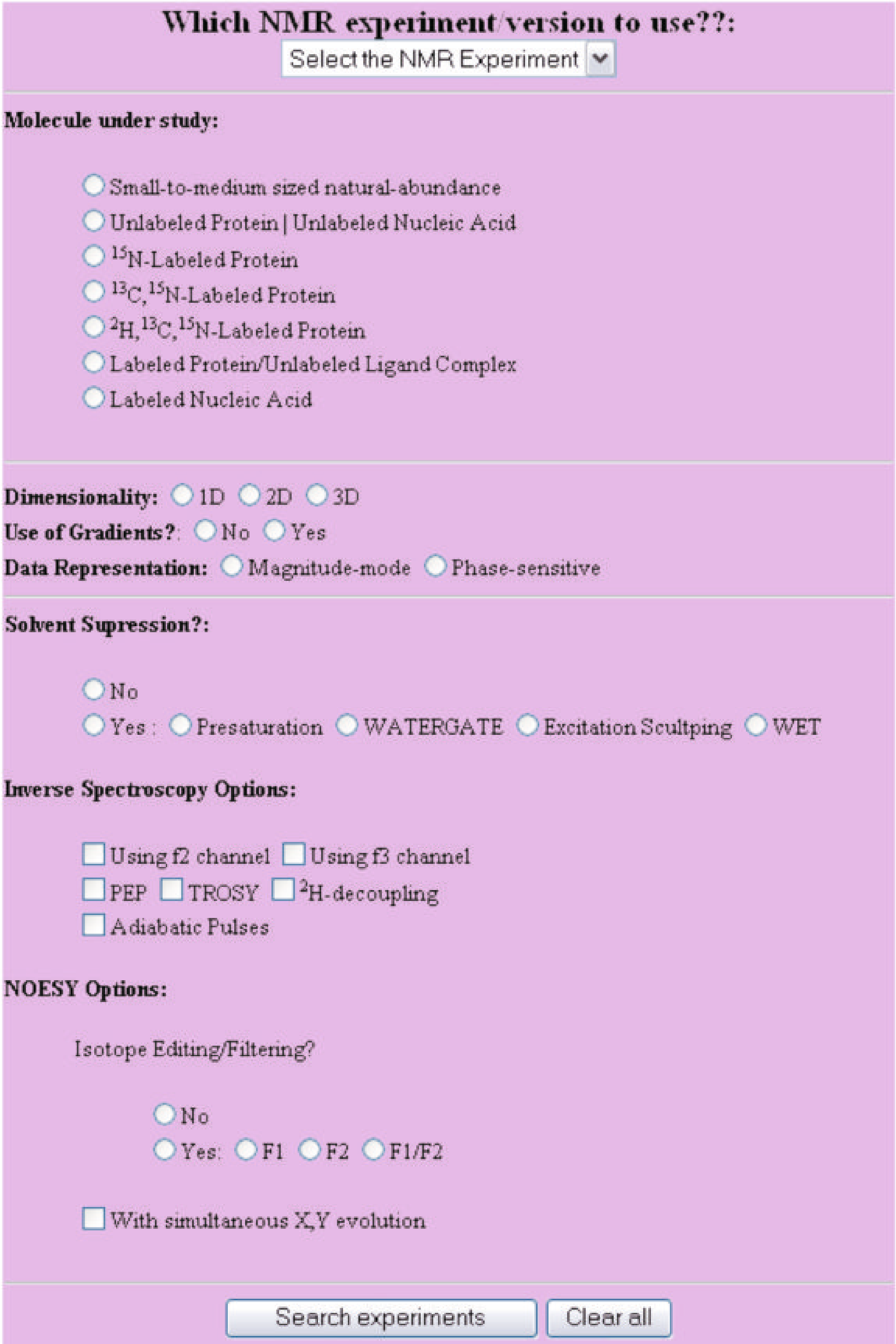
3. Which Version?

1D/2D/3D ???
Pulsed Field-gradients or phase-cycled
Qualitative or quantitative analysis?
Magnitude-mode or phase-sensitive
Sensitivity vs Resolution
Direct detection or Inverse Spectroscopy
Solvent Suppression?

4. Others

Spectrometer Set-up
Experiment Optimization: Pulse Calibration
Variable Temperature
Automation

Loocking for a pulse program or NMR experiment using the **NMR Experiment Selector** tool



Which NMR experiment/version to use??:

Select the NMR Experiment

Molecule under study:

- Small-to-medium sized natural-abundance
- Unlabeled Protein | Unlabeled Nucleic Acid
- ^{15}N-Labeled Protein
- ^{13}C,^{15}N-Labeled Protein
- ^{2}H,^{13}C,^{15}N-Labeled Protein
- Labeled Protein/Unlabeled Ligand Complex
- Labeled Nucleic Acid

Dimensionality: 1D 2D 3D
Use of Gradients?: No Yes
Data Representation: Magnitude-mode Phase-sensitive

Solvent Supression?:

- No
- Yes : Presaturation WATERGATE Excitation Scultping WET

Inverse Spectroscopy Options:

- Using f2 channel Using f3 channel
- PEP TROSY ^{2}H-decoupling
- Adiabatic Pulses

NOESY Options:

Isotope Editing/Filtering?

- No
- Yes: F1 F2 F1/F2

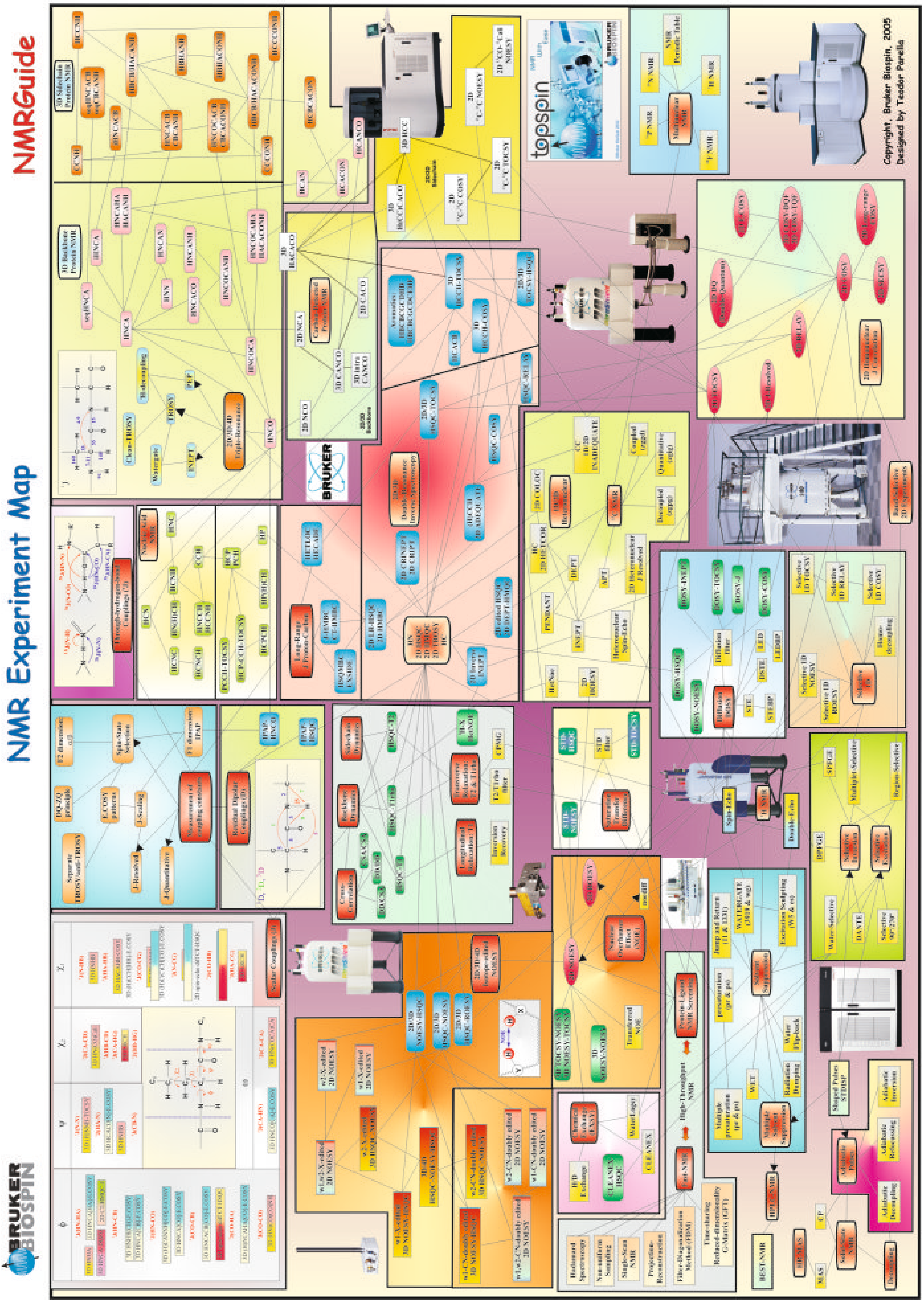
With simultaneous X,Y evolution

Search experiments | Clear all

AVANCE Tutorials

NMR Assistance

NMR Experiment	Parameter Set	Pulse Program	Modify ...
Conventional 1D Experiments			
1D ^{1}H	PROTON	zg30	d1, ns, o1p, sw
1D ^{13}C {^{1}H}	C13CPD	zgpg30	d1, ns, o1p, sw
1D DEPT-135	C13DEPT135	dept135	d1, ns, o1p, sw
1D DEPT-90	C13DEPT90	dept90	d1, ns, o1p, sw
Conventional 2D 1H-1H Experiments			
ge-2D ^{1}H-^{1}H COSY (magnitude)	COSYGPSW	cosygpqf	d1, ns, o1p, 1 sw, 2 sw
2D ^{1}H-^{1}H NOESY (phase sensitive)	NOESYPHSW	noesygpph	ns, o1p, 1 sw, 2 sw, d8
2D ^{1}H-^{1}H ROESY (phase sensitive)	ROESYPHSW	roesyph	ns, o1p, 1 sw, 2 sw, p15
2D ^{1}H-^{1}H TOCSY (phase sensitive)	MLEVPHSW	mlevph	ns, o1p, 1 sw, 2 sw, d9
2D Inverse 1H-13C Experiments			
ge-2D ^{1}H-^{13}C HMQC (magnitude)	HMQCGP	hmqcgp	ns, o1p, o2p, 1 sw, 2 sw
ge-2D ^{1}H-^{13}C HSQC (phase sensitive)	HSQCGP	hsqcetgpsi2	ns, o1p, o2p, 1 sw, 2 sw
ge-2D ^{1}H-^{13}C HSQC edited (phase sensitive)	HSQCEDETGP	hsqcedetgp	ns, o1p, o2p, 1 sw, 2 sw
ge-2D ^{1}H-^{13}C HMBC (magnitude)	HMBCGP	hmbcgplpndqf	d6, ns, o1p, o2p, 1 sw, 2 sw
Selective 1D Experiments			
Selective 1D NOESY	SELNOGP	selnogp	ns, spoffs2, p12, sp2, d8
Selective 1D ROESY	SELROGP	selrogp	ns, spoffs2, p12, sp2, p15
Selective 1D TOCSY	SELMLGP	selmlgp	ns, spoffs2, p12, sp2, d9
31P Experiments			
1H-decoupled 31P	P31CPD	zgpg30	ns, sw, o1p
1H-coupled 31P	P31	zg30	ns, sw, o1p
31P-decoupled 1H spectrum	PROP31DEC	zggd30	ns, o2p
2D 1H-31P HMBC	HMBCGP	hmbcgpndqf	ns, o2p, 1 sw, 1 td, d6


NMRGuide
NMR Experiment Map
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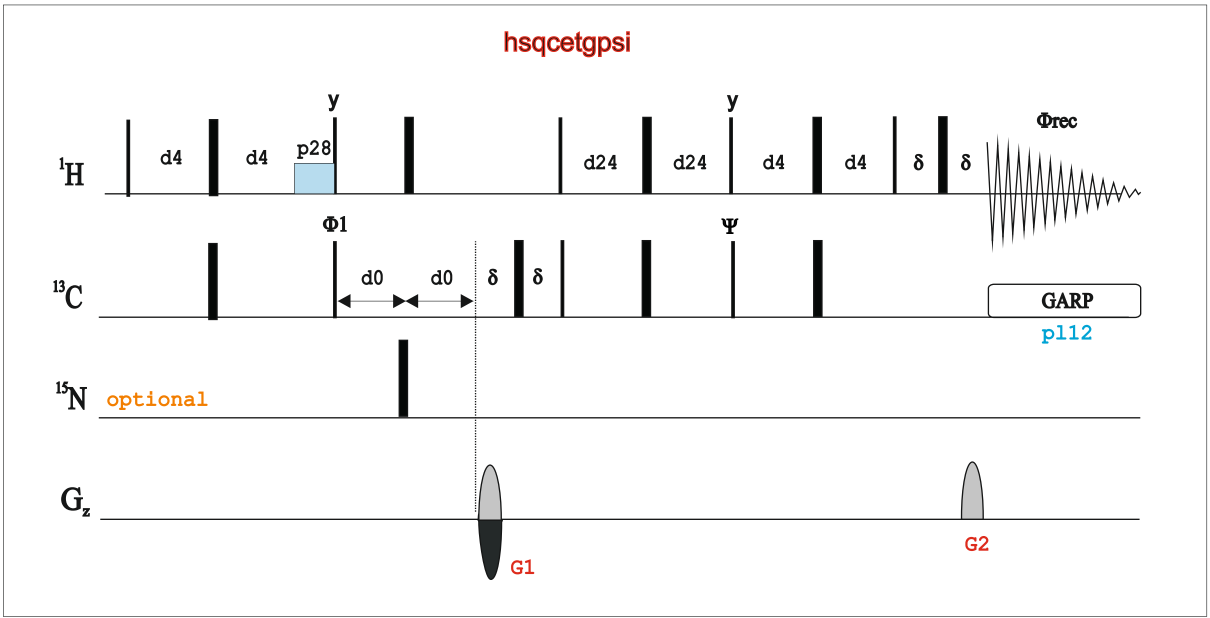
Designed by Teodor Parella

Pulse Sequence Diagram: A brief Outlook.

This is a classical NMR pulse sequence representation:



We can observe the following Pulse Sequence Elements:

1. Different Rf Channels: 1H, 13C, 15N, Gz ...
2. Radiofrequency (Rf) Pulses (90°, 180° ...) applied at specific power levels
3. Inter-pulse Fixed Delays (d1, d4, d24 ....) conveniently optimized to J, NOEs, relax ...
4. Variable delays that define dimensionality: d0 and d10
5. Multiple-pulse sequences: trim pulses, GARP decoupling, TOCSY transfers ...
6. Phase Cycling: Only the most important are usually described (see pulse program for a detailed description)
7. Gradients: Coherence Selection & Purgue Elements
8. Starting Point: Important for sensitivity and repetition rates aspects
9. Final Point (see the FID): Important for sensitivity

<u>Can I run a given NMR experiment on my spectrometer? This is a typical question for non-experienced users. It is needed to consider three independent aspects: Software, Hardware and Sample Requirements:</u>

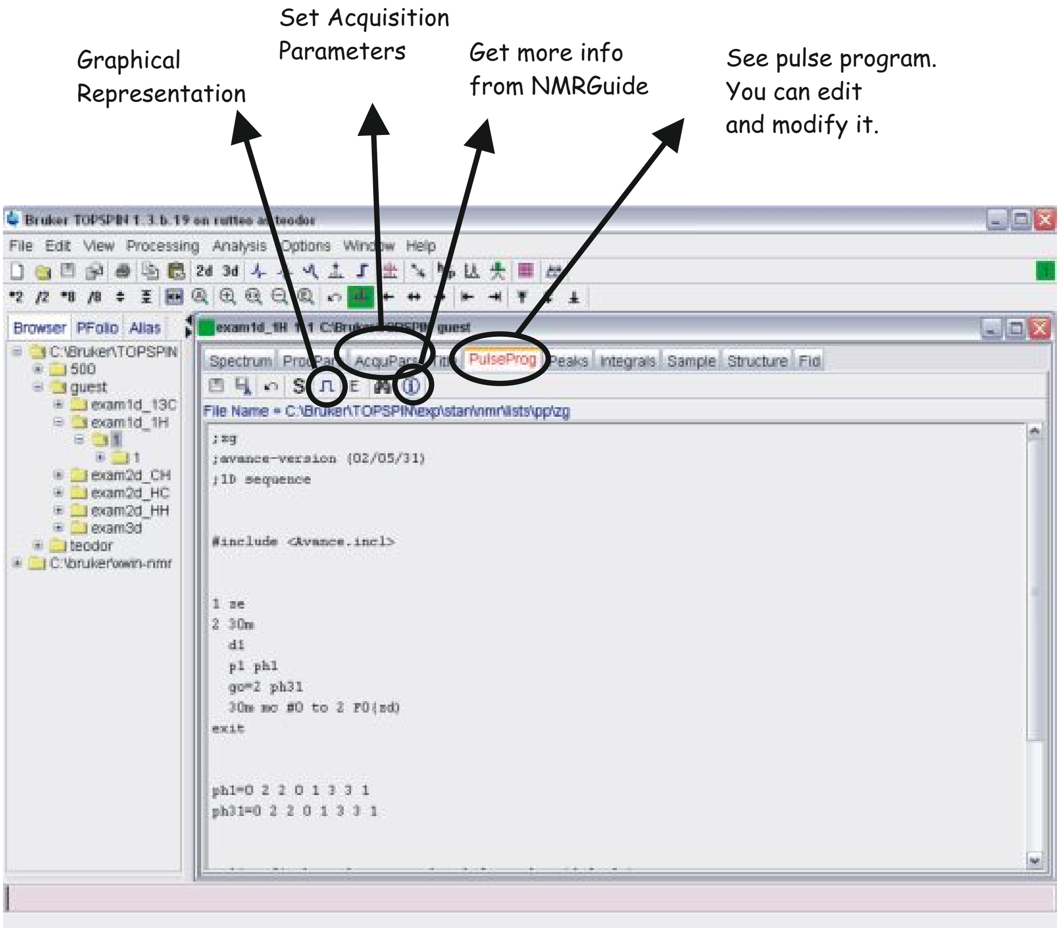
In principle, there is no limitations due to SOFTWARE requirements usingTOPSPIN.

The main limitations came from HARDWARE requirements:

1. The number of available Rf channels
2. Inverse Spectroscopy capabilities
2. Availability of Pulsed-Field Gradients
3. Check for the available Probeheads!!!!

Another important question: SAMPLE requirements???

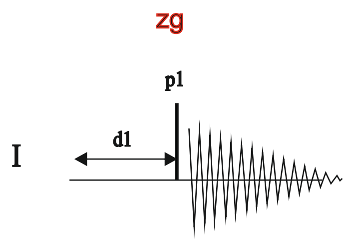
Many experiments are usually driven in **natural abundance** samples but in biomolecules are necessary some **isatopic labeling strategies**: 15N-labeled, doubly-15N,13C-labeled, partial or fully 2H combined with 15N,13C-labeled, or other selective labeling approaches



The NMR Pulse Sequence: Diagram vs Microprogram

Any NMR Experiment is closely related to a pulse sequence that can be analyzed in detail using TOPSPIN.

The pulse sequence diagram



The pulse program

```
;zg
;avance-version (02/05/31)
;1D sequence
```
1. Header: Experiment Description

```
#include <Avance.incl>
```
2. Additional required files

```
1 ze
2 30m
  d1
  p1 ph1
  go=2 ph31
  30m mc #0 to 2 F0(zd)
exit
```
3. Pulse-Delay timing execution

```
ph1=0 2 2 0 1 3 3 1
ph31=0 2 2 0 1 3 3 1
```
4. Phase Cycle

```
;pl1 : f1 channel - power level for pulse (default)
;p1 : f1 channel -  high power pulse
;d1 : relaxation delay; 1-5 * T1
;NS: 1 * n, total number of scans: NS * TD0
```
5. Parameter Description

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NMRGuide

STANDARD TESTS &
PULSE CALIBRATIONS

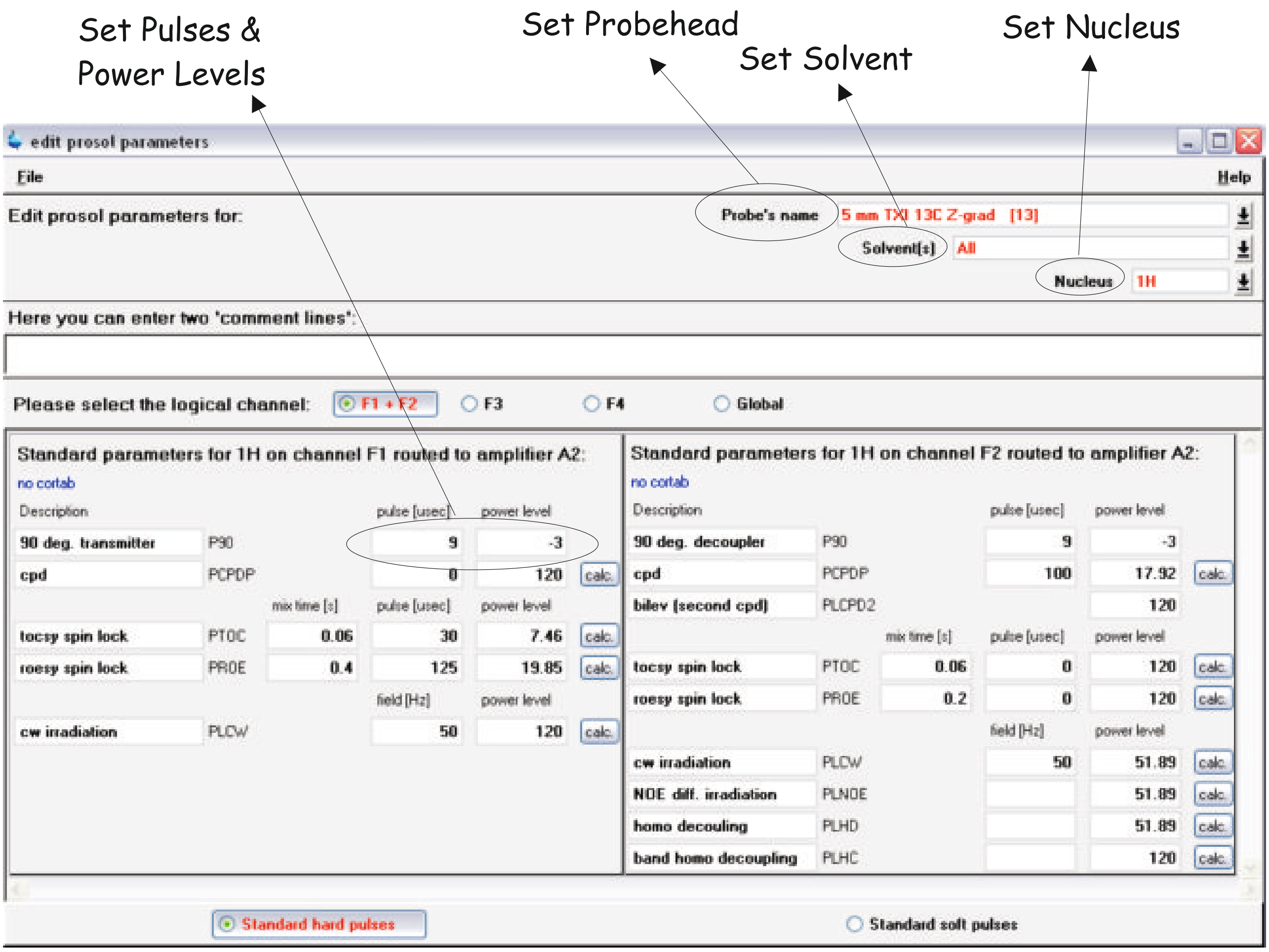
BASIC TEST ON AVANCE SPECTROMETERS:

- Lineshape test for ^{1}H without rotation (lsnh)
- Lineshape test for ^{1}H with rotation (lsrh)
- Resolution test for ^{1}H (rsh)
- Determination of 90 degree ^{1}H high power transmitter (tph)
- Sensitivity test for ^{1}H (snh)
- Water suppression test (ws)
- Lineshape test for ^{13}C (lsc)
- Resolution test for ^{13}C (rsc)
- Determination of 90 degree ^{13}C high power transmitter pulse with popt (tpc)
- Determination 90 degree 1H pulse for high-power decoupling (CPD pulses) (dph)
- Determination 90 degree 1H pulse for low-power decoupling (CPD pulses) (cph)
- Sensitivity test for 13C ASTM (without 1H decoupling) (sna)
- Sensitivity test for 13C EB (with 1H CPD decoupling) (snc)
- Determination 90 degree 13C high power decoupling (puc)
- Determination 90 degree 13C low power decoupling (for GARP) (gac)
- Inverse spin-echo difference test (inv)
- Determination 90 degree 31P high power
- Sensitivity test for 31P (snp)
- Sensitivity test for 15N (snn)
- Determination 90 degree 15N high power decoupling (pun)
- Determination 90 degree 15N low power decoupling (GARP) (gan)
- Gradient Recovery Test
- Determination of selective shaped 90 degree 1H pulse
- Selective Excitation test
- Selective Experiment test
- Homodecoupling test (hde)
- Sensitivity test for 19F (snf)
- GARP decoupling 13C (garc)
- Decoupling test 19F (with 1H CPD decoupling) (decf)
- Triple Resonance experiment (tri)
- Dataset for quad image adjustment (qad)
- Decoupling test 19F (with 19F GARP decoupling) (garf)
- B1 homogeneity test 1H from HWT (b1h)

Setting Pulses and Power Levels

Once pulse lengths and power levels are determined for a given probehead, they are defined into **edprosol**.

These values are automatically placed into a parameter set using the **getprosol** command

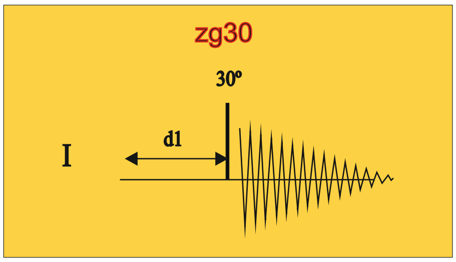


NMR Experiment: Lineshape test for ^{1}H without rotation (lsnh)
Basic Parameter Set: PROHUMP
Pulse Program: zg30

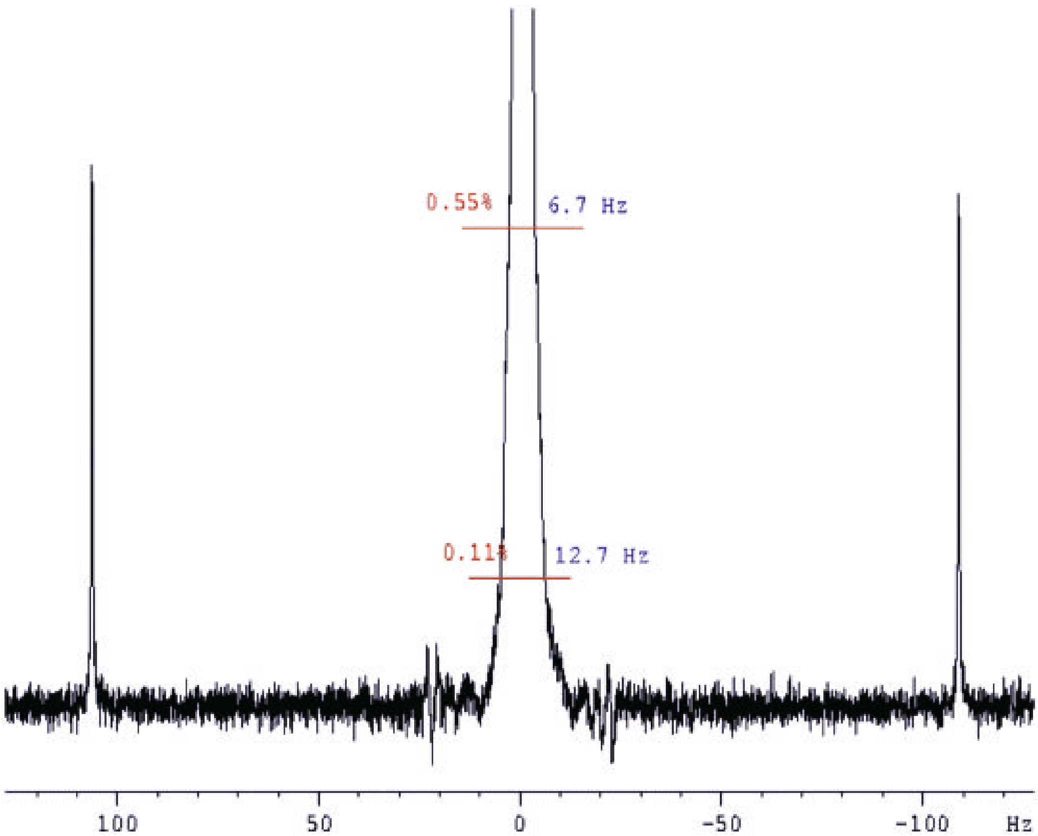
Sample: 1% CHCl3 in Acetone-d_6

Spin: off

Basic acquisition parameters:
d1 2s
sw=1000Hz
o1p on the CHCl3 resonance
ns=1 ds=0



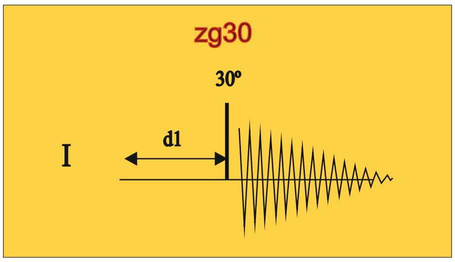
Analysis: The linewidth is automatically determined at 0.55% of the chloroform line and at one fifth there of (0.11%) by the **humpcal** program



NMR Experiment: Lineshape test for ^{1}H with rotation (lsrh)
Basic Parameter Set: PROHUMP
Pulse Program: zg30

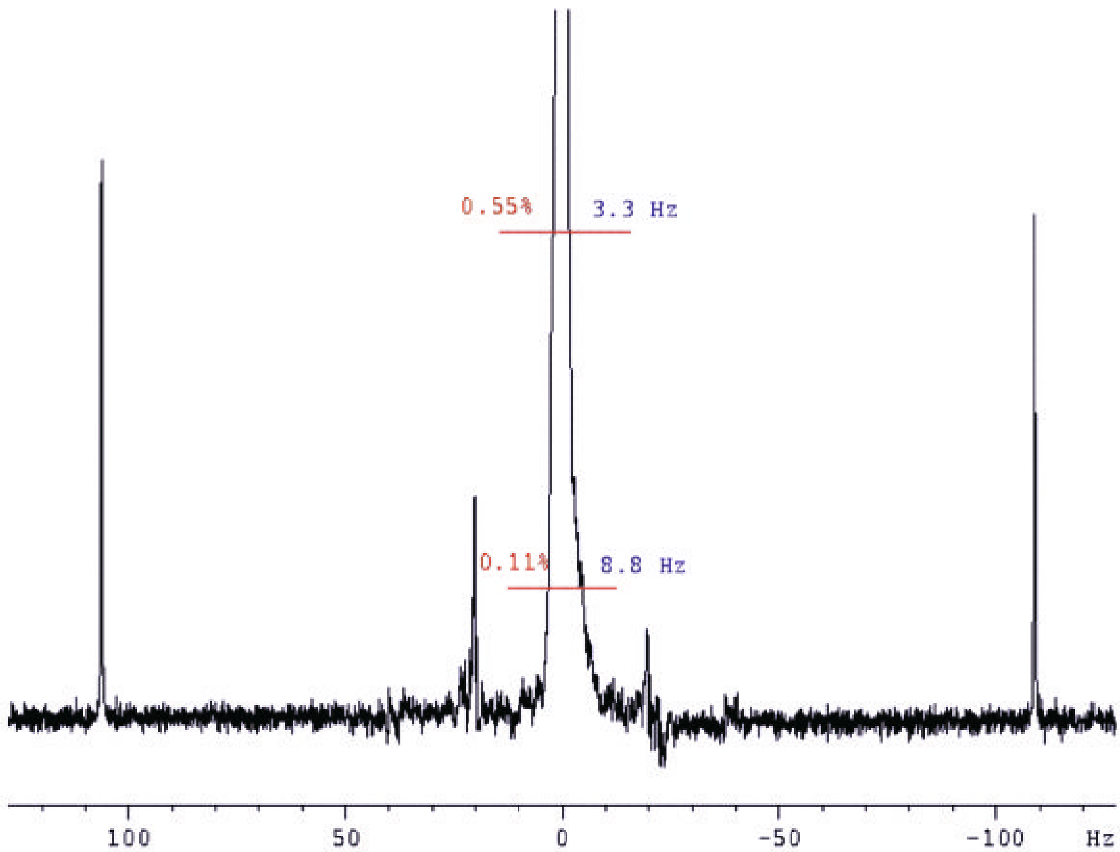
Sample: 1% CHCl3 in Acetone-d_6

Spin: on



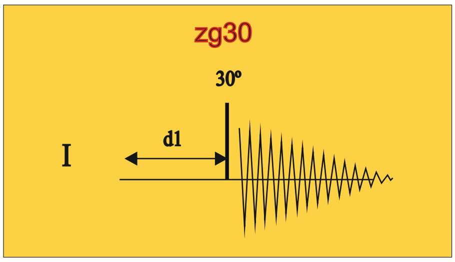
Basic acquisition parameters:
d1 2s
sw=1000Hz
o1p on the CHCl3 resonance
Ns=4 ds=0

Analysis: The linewidth is automatically determined at 0.55% of the chloroform line and at one fifth there of (0.11%) by the **humpcal** program



NMR Experiment: Resolution test for ^{1}H (rsh)
Basic Parameter Set: PRORESOL
Pulse Program: zg30

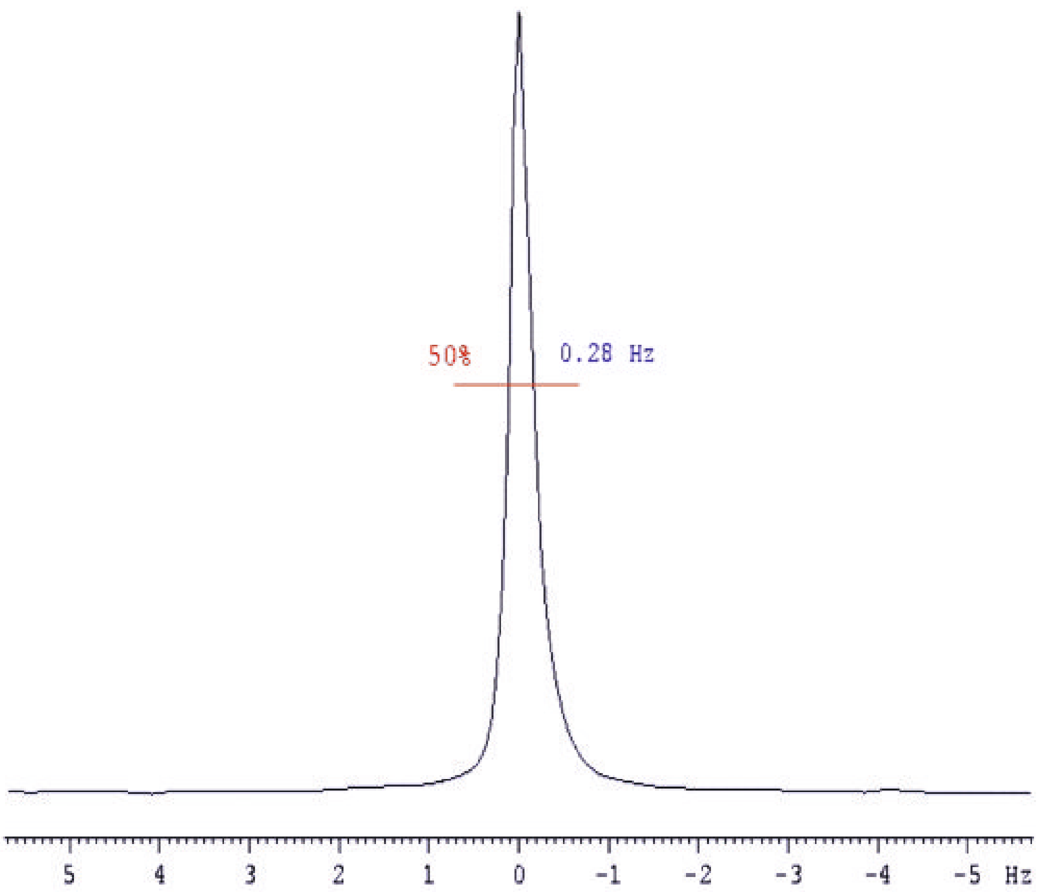
Sample: 1% CHCl3 in Acetone-d_6



Spin: on

Basic acquisition parameters:
d1 1s
sw=1000Hz
o1p on the CHCl3 resonance
ns=1 ds=0

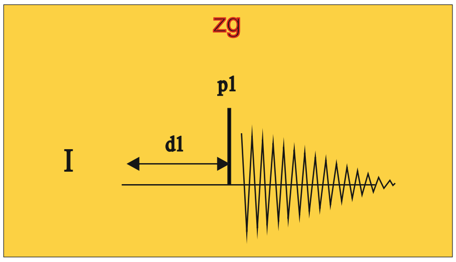
Analysis: The resolution is measured at half height of the chloroform line and calculated with **humpcal**



NMR Experiment: Determination of 90 degree ^{1}H high power transmitter pulse with popt (tph)
Basic Parameter Set: PROTON
Pulse Program: zg

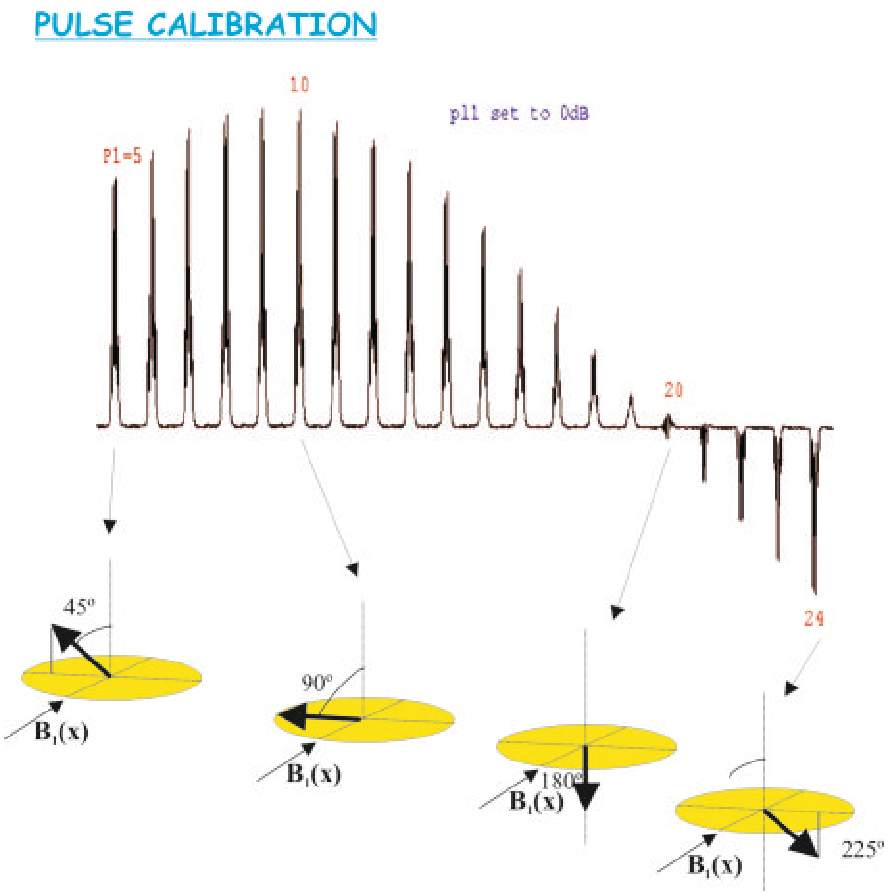
Sample: 0,1% Ethylbenzene in CDCl3

Spin: on



Basic acquisition parameters:
d1 120s
sw 3000Hz
o1p on the methylene resonance
ns=1 ds=0
Process with ef (lb=1)

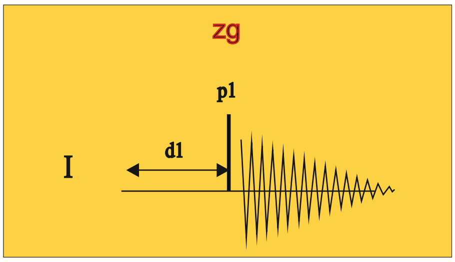
Analysis: The pulse length strongly depends on the transmitter power and probe design. Remember to tune and match before you start calibration with **popt**. The first maximum is the 90°, the first null point is 180° and the second 360°



NMR Experiment: Sensitivity test for ^{1}H (snh)
Basic Parameter Set: PROSENS
Pulse Program: zg

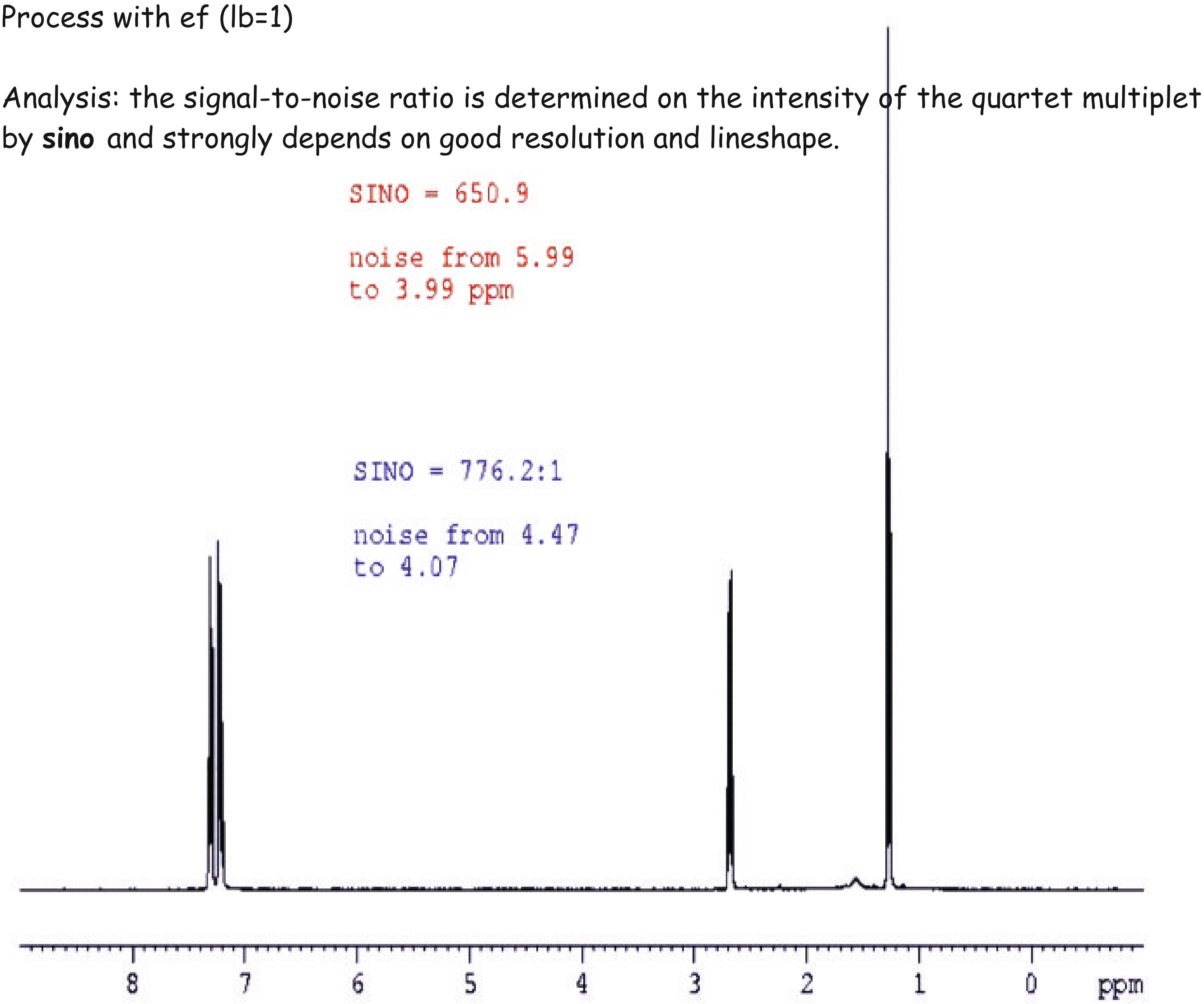
Sample: 0,1% Ethylbenzene in CDCl3

Spin: on



Basic acquisition parameters:
d1 60s
sw 3000Hz
ns=1 ds=0
Set p1 to the calibrated 90 degree value at pl1 power level
Process with ef (lb=1)

Analysis: the signal-to-noise ratio is determined on the intensity of the quartet multiplet by **sino** and strongly depends on good resolution and lineshape.

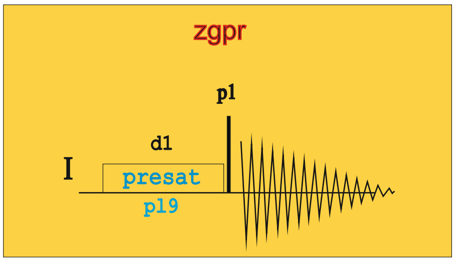


NMR Experiment: Water suppression test (ws)
Basic Parameter Set: WATERSUP
Pulse Program: zgpr

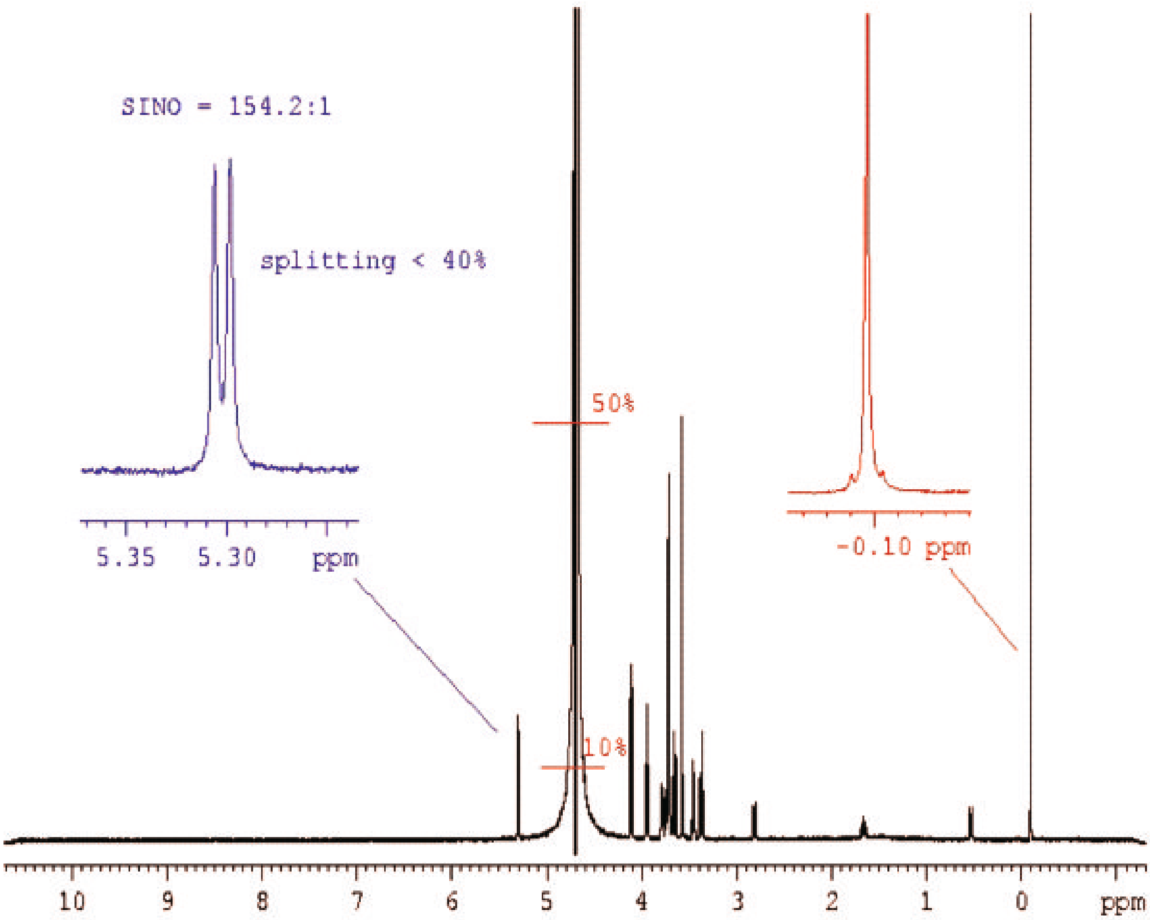
Sample: 2mM sucrose with 0.5mM DSS, 2mM NaN3 in 10% D2O and 90% H2O

Spin: off

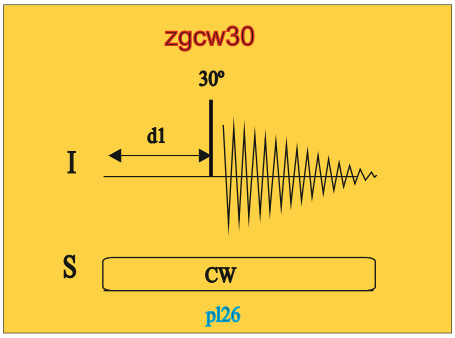
Basic acquisition parameters:
d1 5s
set aq=1s and sw=12ppm (td is automatically calculated)
Set exactly o1p on the water resonance (about 4.7ppm) by minimizing the FID in **gs** mode
ns=8 ds=4
Set pl9 between 55-65dB (50Hz rf field)
Process without window function



Analysis: The linewidth is measured at 50% height of the DSS signal and a 10% thereof. The S/N ratio is measured on the doublet left to the water signal using **sino**. On this doublet the splitting (resolution) is also measured.



NMR Experiment: Lineshape test for ^{13}C (lsc)
Parameter Set: C13HUMP
Pulse Program: zgcw30



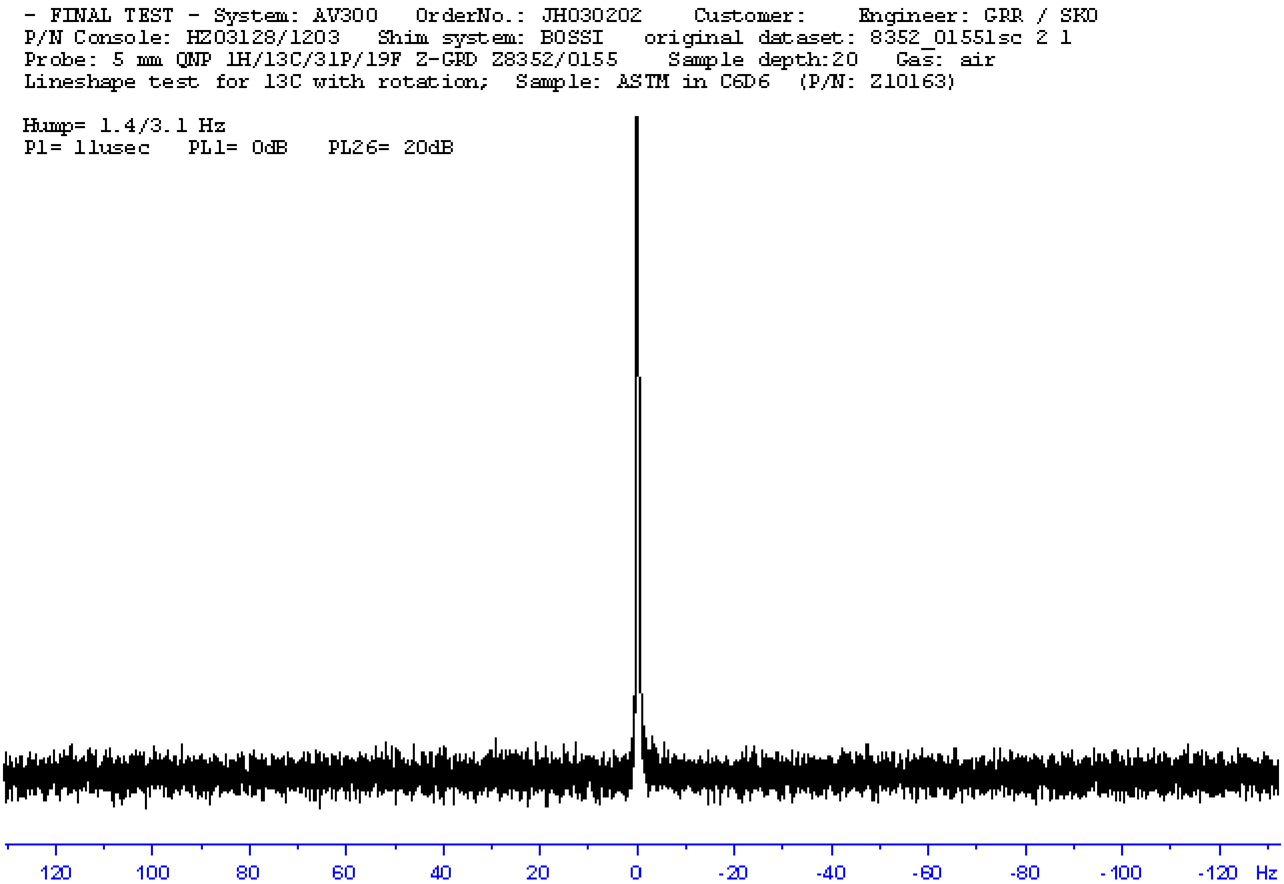
Sample: ASTM (60% C6D6 / 40% p-Dioxane)

Spin: on

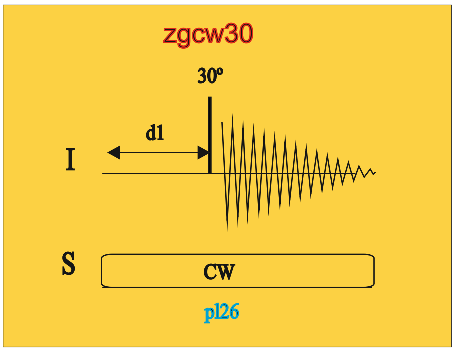
Basic acquisition parameters:
d1 10s
ns 4 and ds 0
o1p 66.5ppm
Optimize o2 (start with 3.39ppm) in gs mode by maximizing the FID

Process without window function

Analysis: The linewidth is measured automatically with humpcal at 0.55% and 0.11% levels.



NMR Experiment: Resolution test for ^{13}C (rsc)
Parameter Set: C13RESOL
Pulse Program: zgcw30



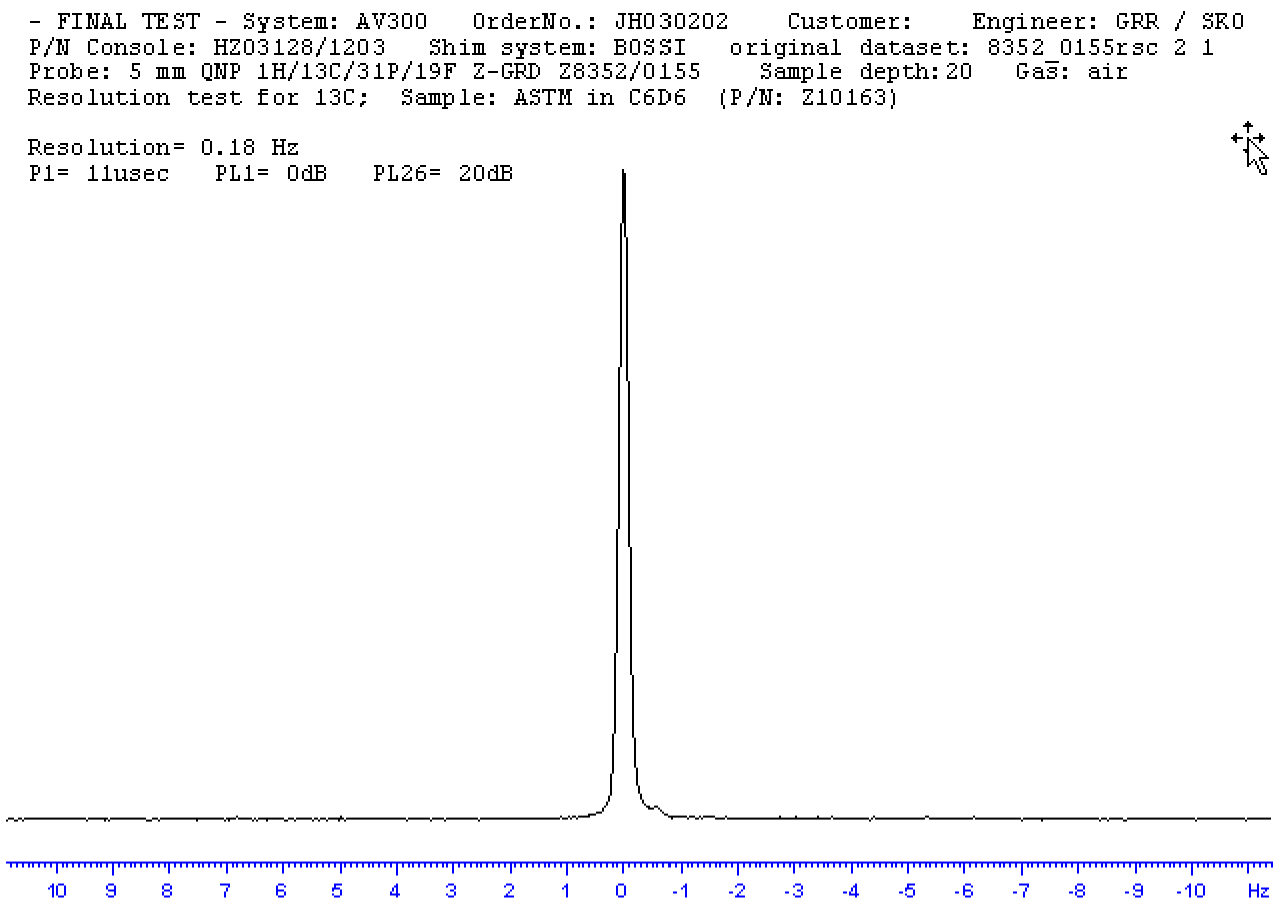
Sample: ASTM (60% C6D6 / 40% p-Dioxane)

Spin: on

Basic acquisition parameters:
d1 10s
ns 1 and ds 0
o1p 66.5ppm
Optimize o2 (start with 3.39ppm) in gs mode by maximizing the FID

Process without window function

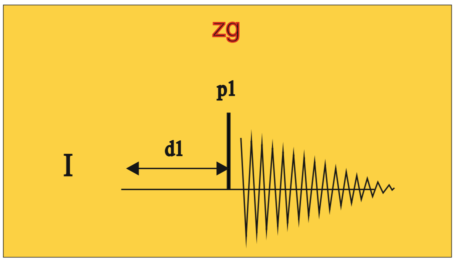
Analysis: The resolution is measured automatically with humpcal athalf height of the signal.



NMR Experiment: Determination of 90 degree ^{13}C high power transmitter pulse with popt (tpc)
Basic Parameter Set: C13CPD
Pulse Program: zg

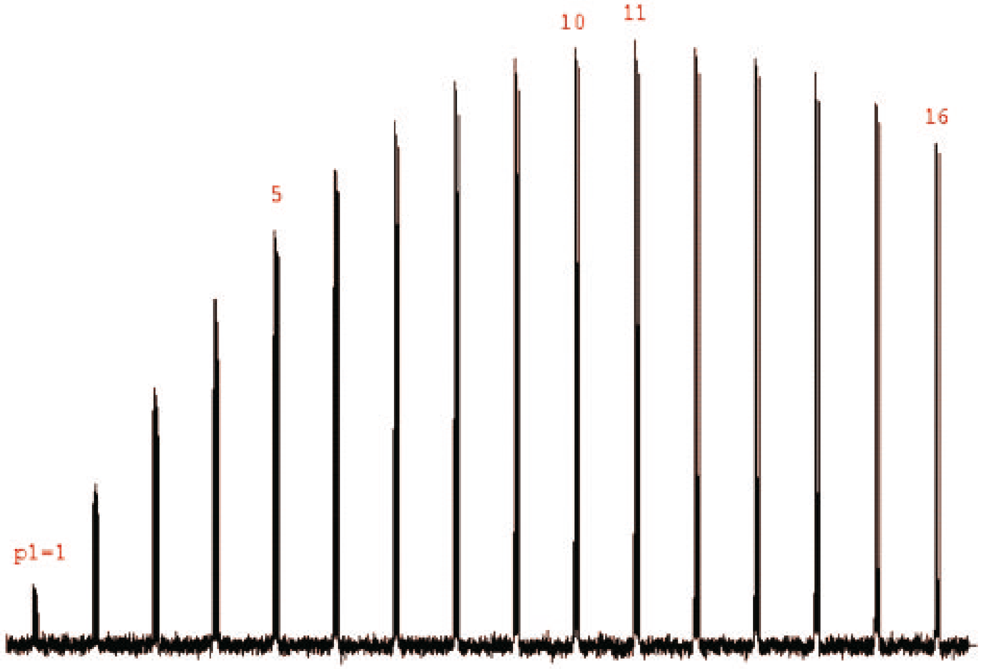
Sample: ASTM (60% C6D6 /40% p-Dioxane)

Spin: on



Basic acquisition parameters:
d1 80s (very important to use a lond delay!!)
sw 15000Hz
o1p on-resonance
ns=1 ds=0
Process with ef (lb=3.5)

Analysis: The pulse length strongly depends on the transmitter power and probe design. Remember to tune and match before you start calibration with **popt**. The first maximum is the 90°, the first null point is 180° and the second 360°

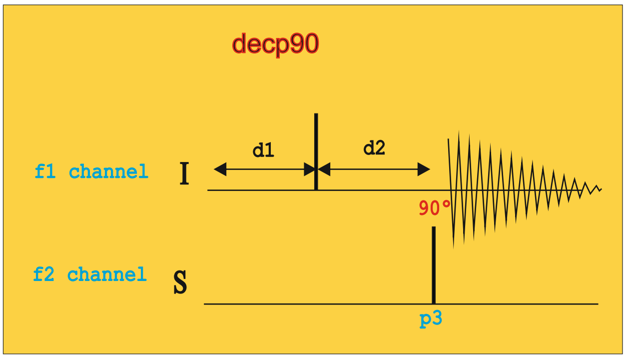


NMR Experiment: Determination 90 degree 1H pulse for high-power decoupling (CPD pulses) (dph)
Basic Parameter Set: C13CPD
Pulse Program: decp90

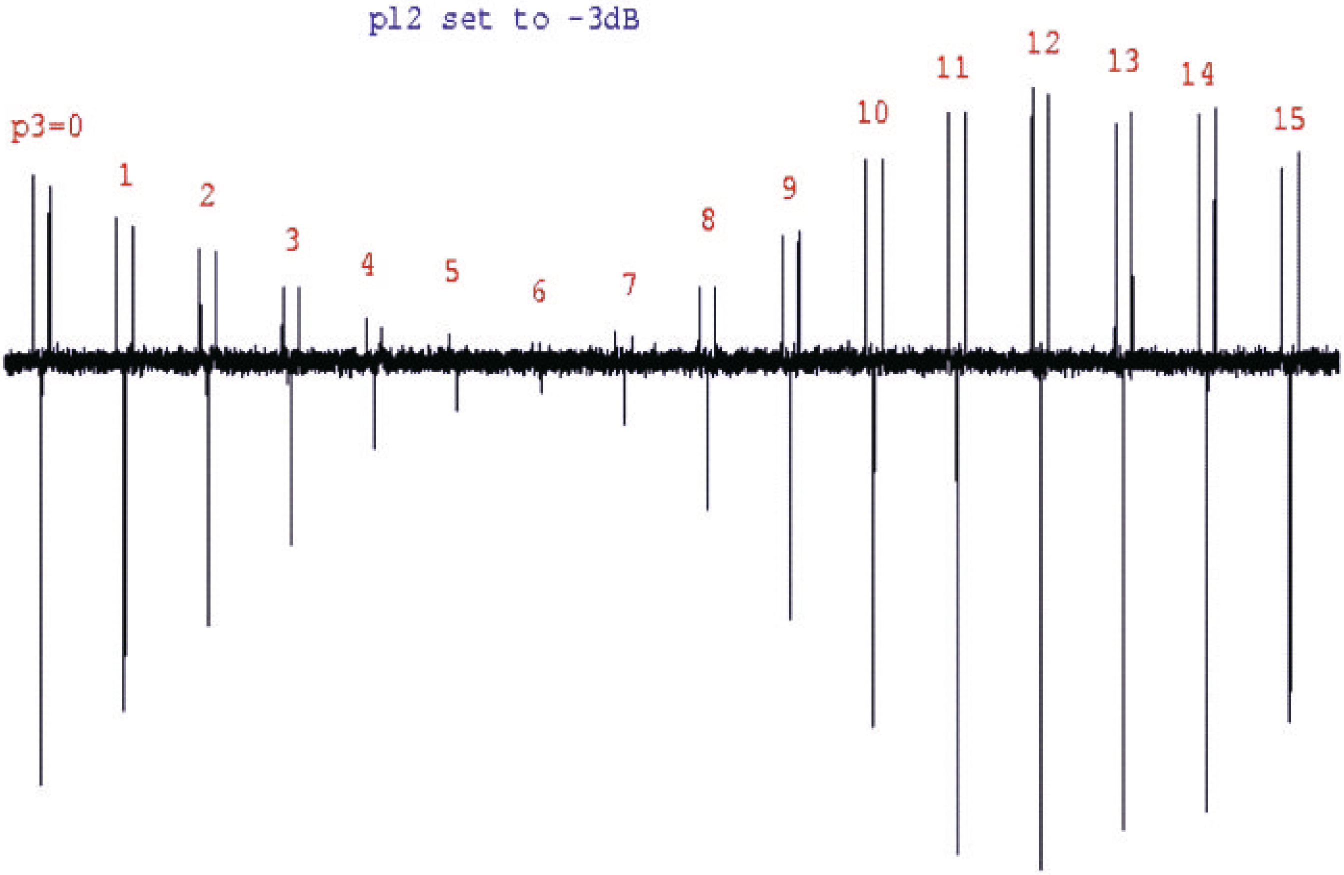
Sample: ASTM (60% C6D6 / 40% p-Dioxane)

Spin: on

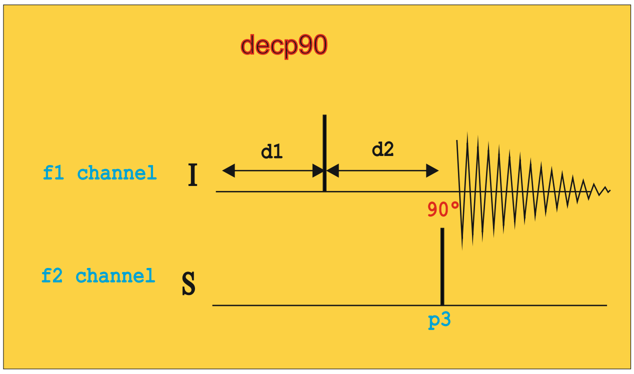
Basic acquisition parameters:
d1 45s
ns 1 and ds 0
o1p 66.5ppm; o2p=3.7ppm
CNST2=142Hz; d203.52ms
Process with ef (lb=3.5Hz)



Analysis: Without decoupling we observe an antiphase triplet, with the two outer lines in phase and the center line in opposite phase. Set pl2 (for instance, to -3dB) and varies p3 using **popt** until the triplet becomes zero.



NMR Experiment: Determination 90 degree 1H pulse for low-power decoupling (CPD pulses) (cph)
Basic Parameter Set: C13CPD
Pulse Program: decp90



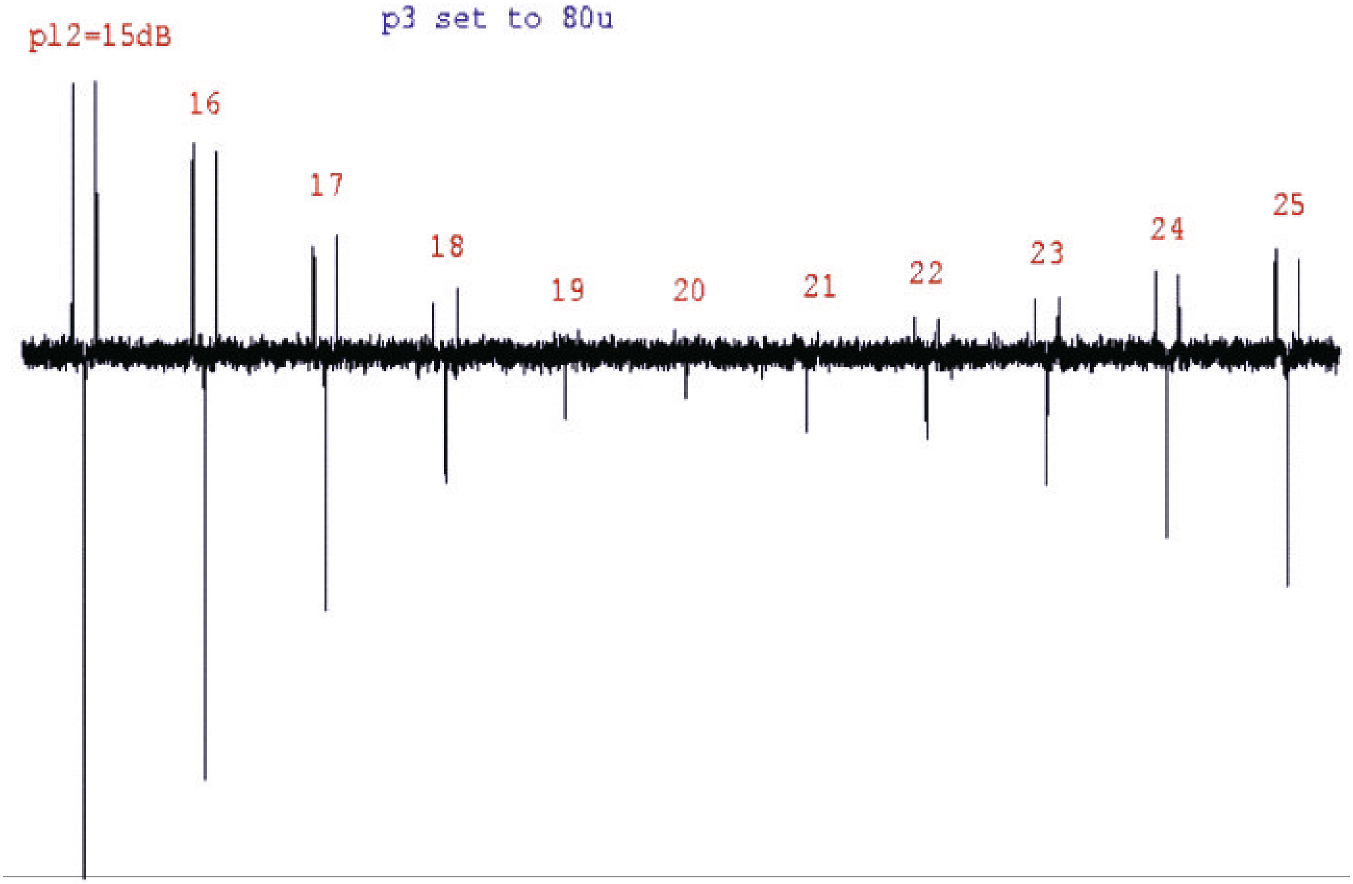
Sample: ASTM (60% C6D6 / 40% p-Dioxane)

Spin: on

Basic acquisition parameters:
d1 45s
ns 1 and ds 0
o1p 66.5ppm; o2p=3.7ppm
CNST2=142Hz; d2=3.52ms

Process with ef (lb=3.5Hz)

Analysis: Without decoupling we observe an antiphase triplet, with the two outer lines in phase and the center line in opposite phase. Set p3=80us and start with a low power pl2=30dB. Increase pl2 using **popt** until the triplet becomes zero. These values will be used for CPD 1H decoupling experiments.



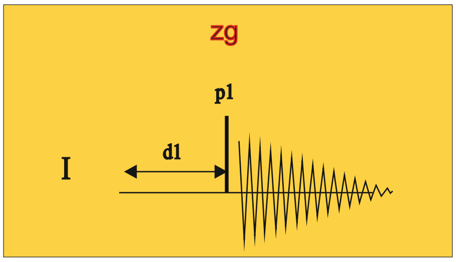
NMR Experiment: Sensitivity test for 13C ASTM (without 1H decoupling) (sna)
Basic Parameter Set: C13SENS
Pulse Program: zg

Sample: ASTM (60% C6D6 / 40% p-Dioxane)

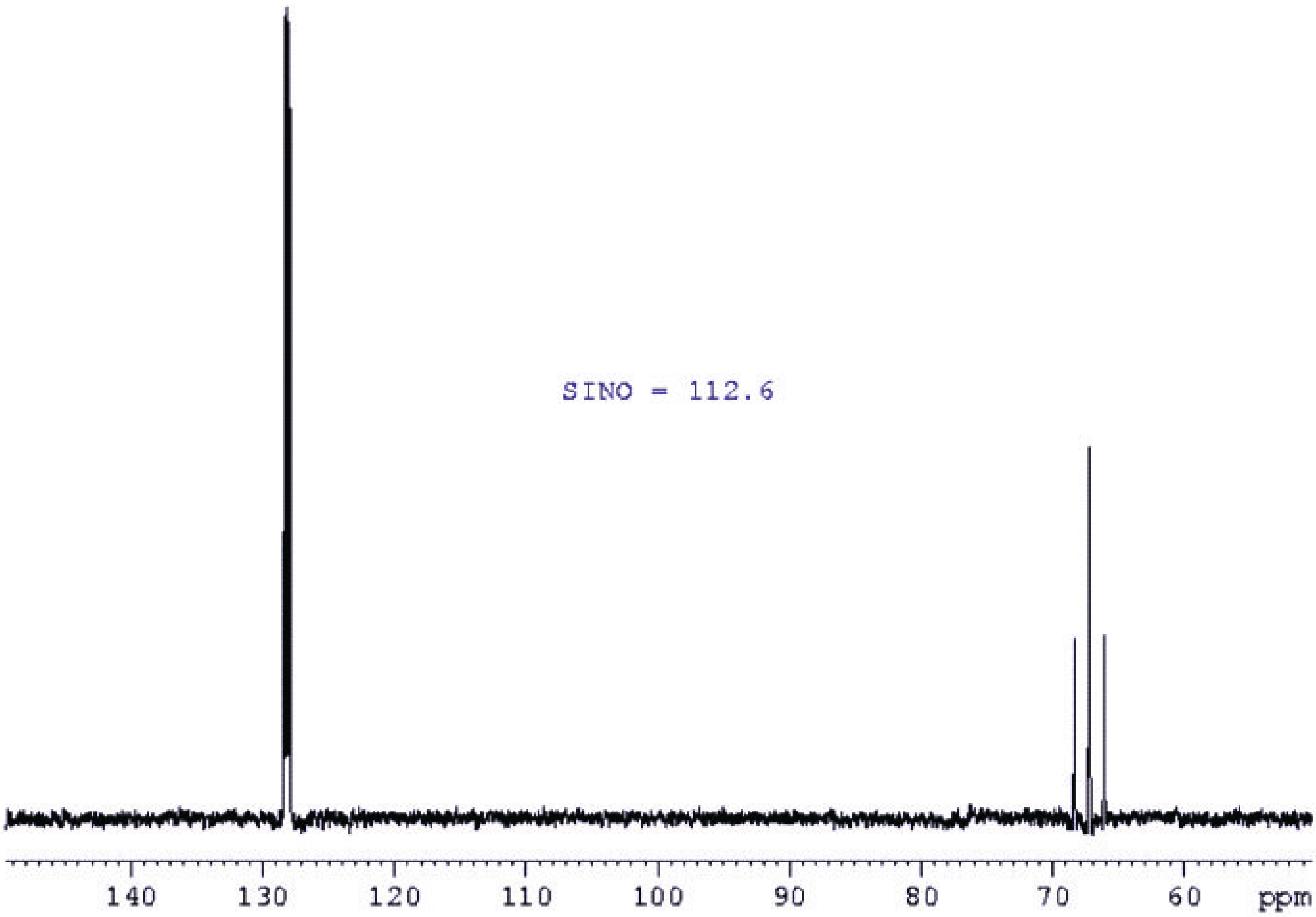
Spin: on

Basic acquisition parameters:
d1 300s
p1 must be the calibrated 90° pulse at pl1
ns 1 and ds 0

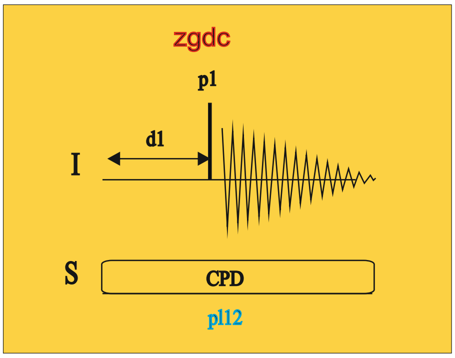
Process with ef (lb=3.5Hz)



Analysis: The S/N is determined on the triplet of the deuterated benzene at 128ppm using **sino**. The splitting of the 1:1:1 triplet should go lower than 9%.



NMR Experiment: Sensitivity test for 13C EB (with 1H CPD decoupling) (snc)
Basic Parameter Set: C13SENS
Pulse Program: zgdc

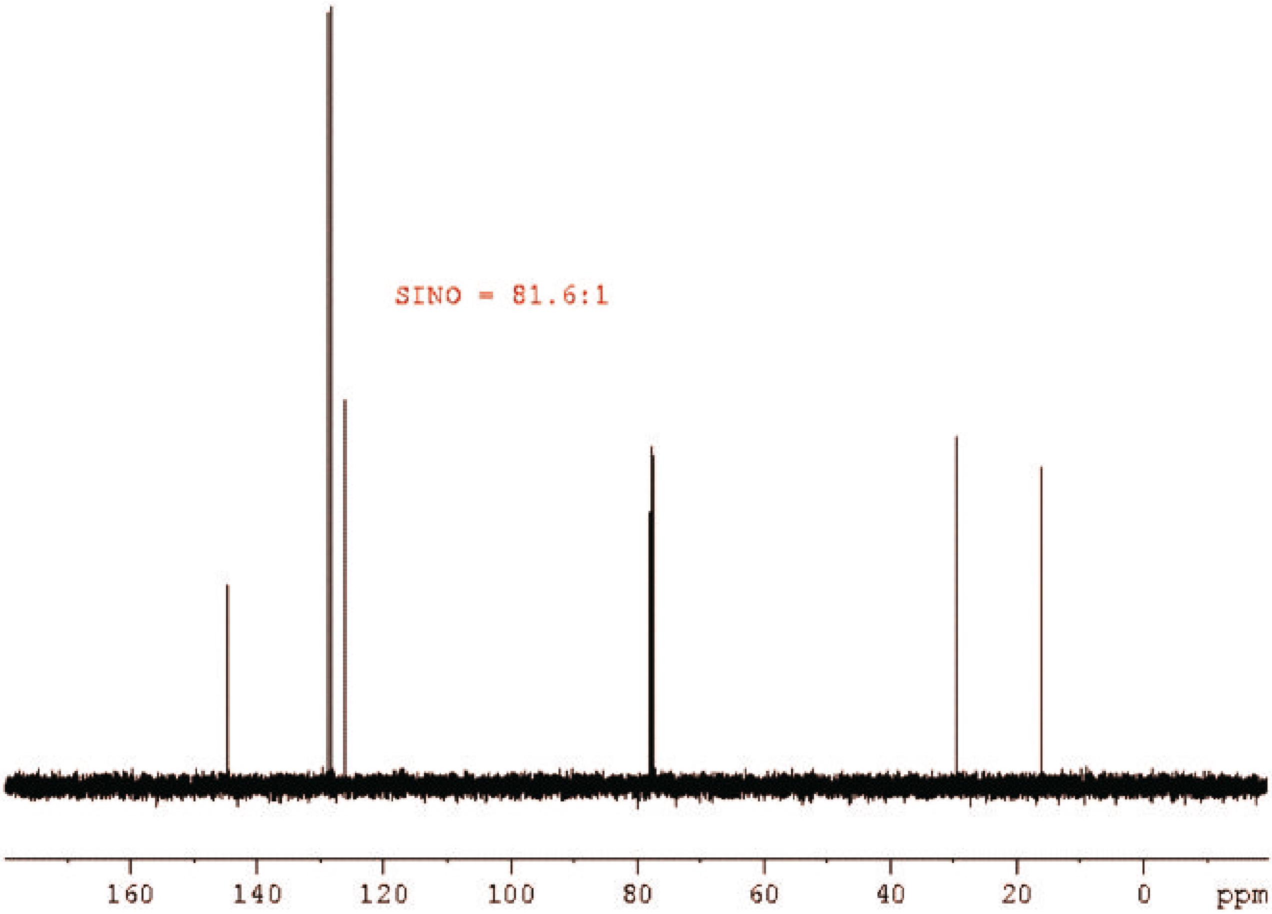


Sample: 10% Ethylbenzene in CDCl3

Spin: on

Basic acquisition parameters:
d1 120s
p1 must be the calibrated 90° pulse at pl1
ns 1 and ds 0
o1p=80ppm, o2p=5ppm, sw=200ppm and pl12 for CPD
Process with ef (lb=0.3Hz)

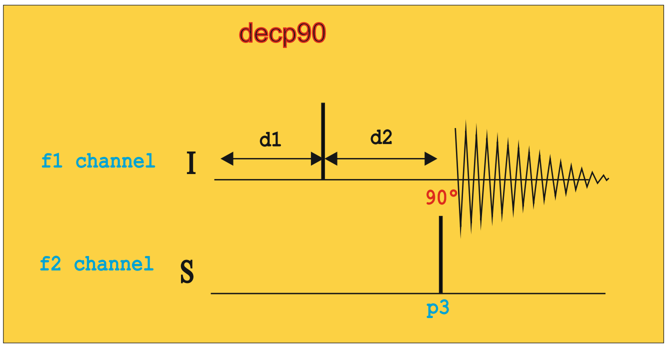
Analysis: The S/N is determined on the highest peak of the aromatic part. It is calculated using **sino** over a range of 40ppm between 30-125ppm.



NMR Experiment: Determination 90 degree 13C high power decoupling (puc)
Basic Parameter Set: HMQC1D
Pulse Program: decp90

Sample: 100mM Urea 15N, 100mM CH3OH 13C in DMSO-d6

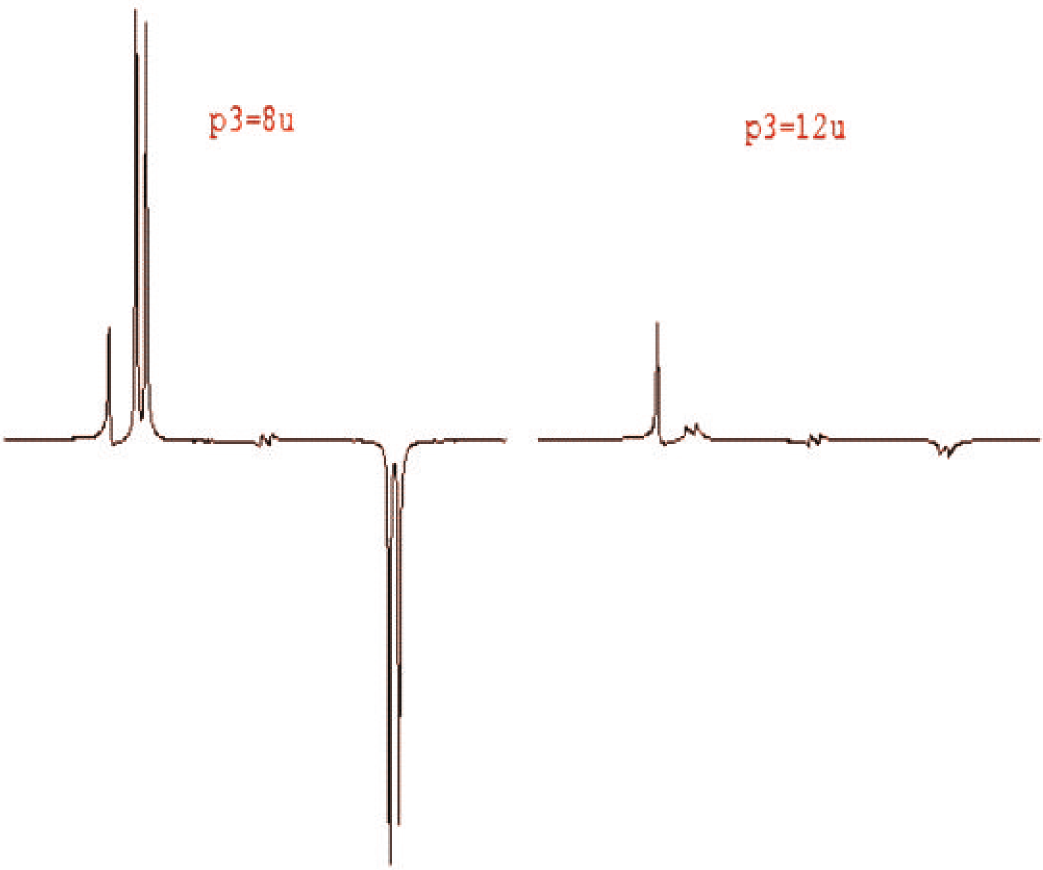
Spin: off



Basic acquisition parameters:
d1 30s
ns 1 and ds 0
o1p=4ppm o2p=49.5ppm
CNST2=139Hz (d2=3.59ms)

Process with ef (lb=0.3Hz)

Analysis: Without decoupling we observe antiphase signals at 3.2ppm. Increase p3 at power level pl2 until the signals become zero.



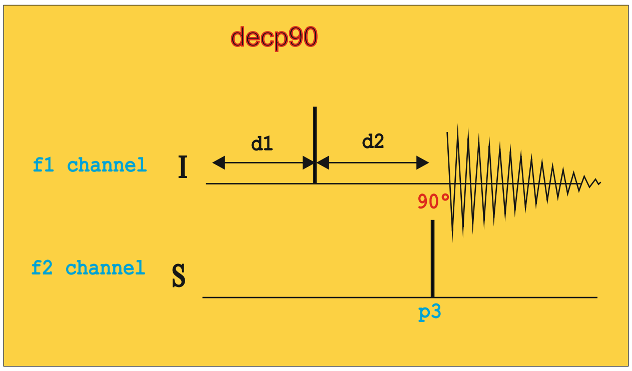
NMR Experiment: Determination 90 degree 13C low power decoupling (for GARP) (gac)
Basic Parameter Set: HMQC1D
Pulse Program: decp90

Sample: 100mM Urea 15N, 100mM CH3OH 13C in DMSO-d6

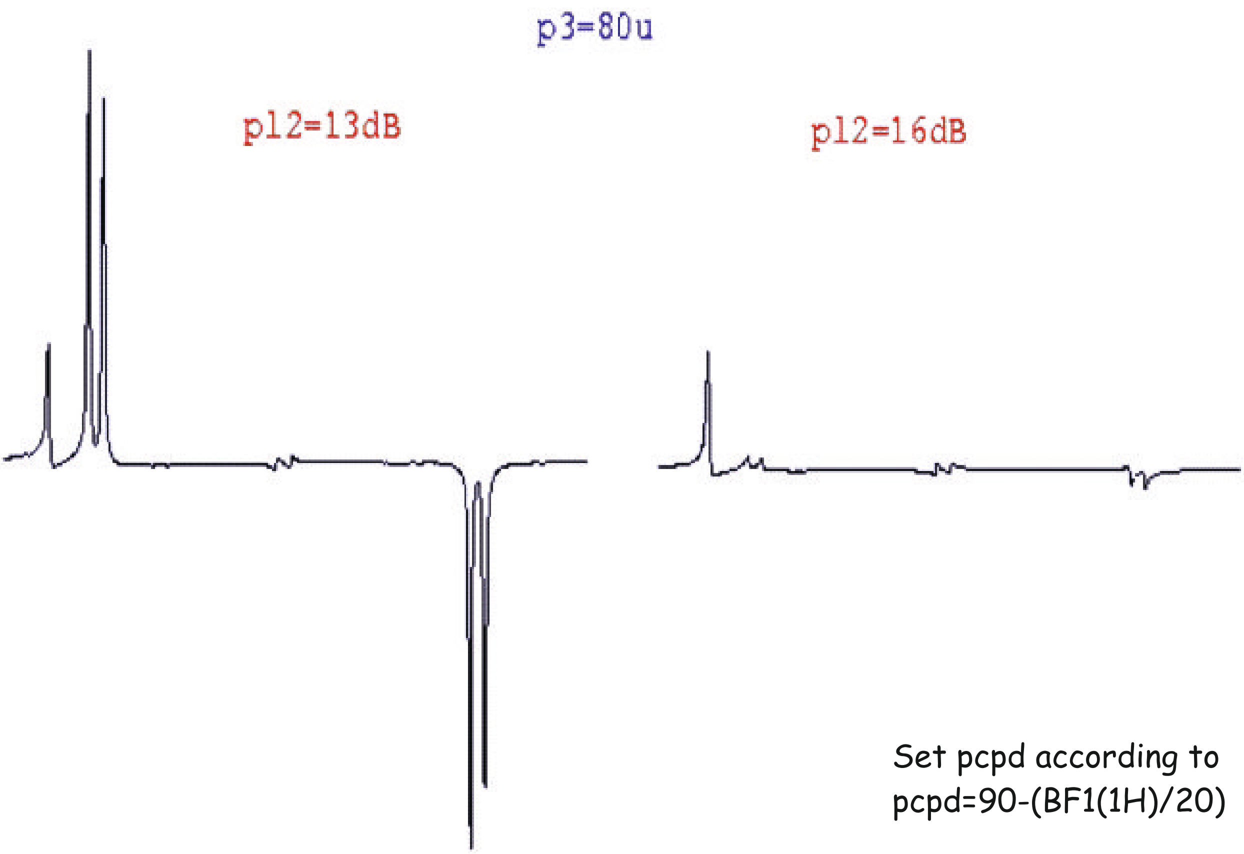
Spin: off

Basic acquisition parameters:
d1 2s
ns 1 and ds 0
o1p=4ppm o2p=49.5ppm
CNST2=139Hz (d2=3.59ms)

Process with ef (lb=0.3Hz)



Analysis: Without decoupling we observe antiphase signals at 3.2ppm. Set p3=80us and optimize the power level pl2 until the signals become zero.



Set pcpd according to
pcpd=90-(BF1(1H)/20)

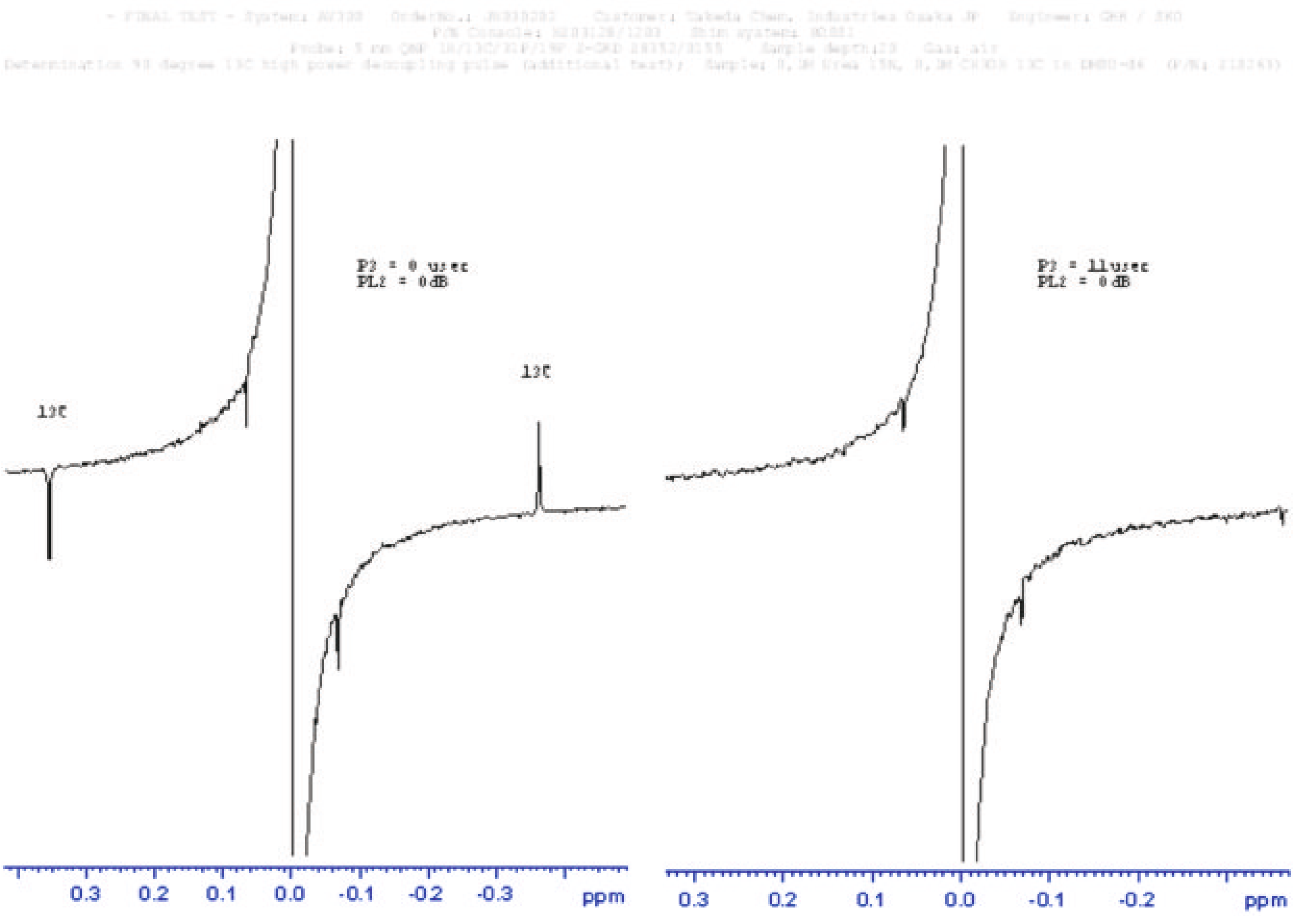
Sample:1% or 3% $CHCl_3$ in acetone-d6

Spin: off

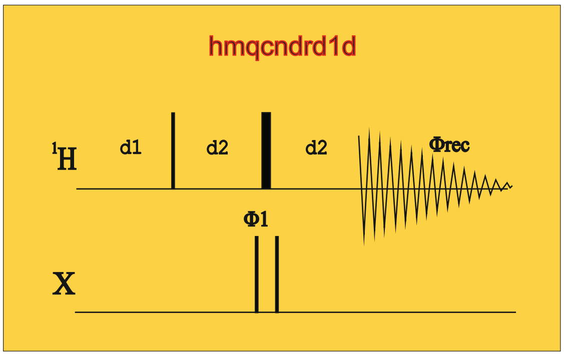
Basic acquisition parameters:
d1 2s
ns 1 and ds 0
o1p=4ppm sw=8ppm
o2p=77ppm
CNST2=214Hz (d2=2.336ms)

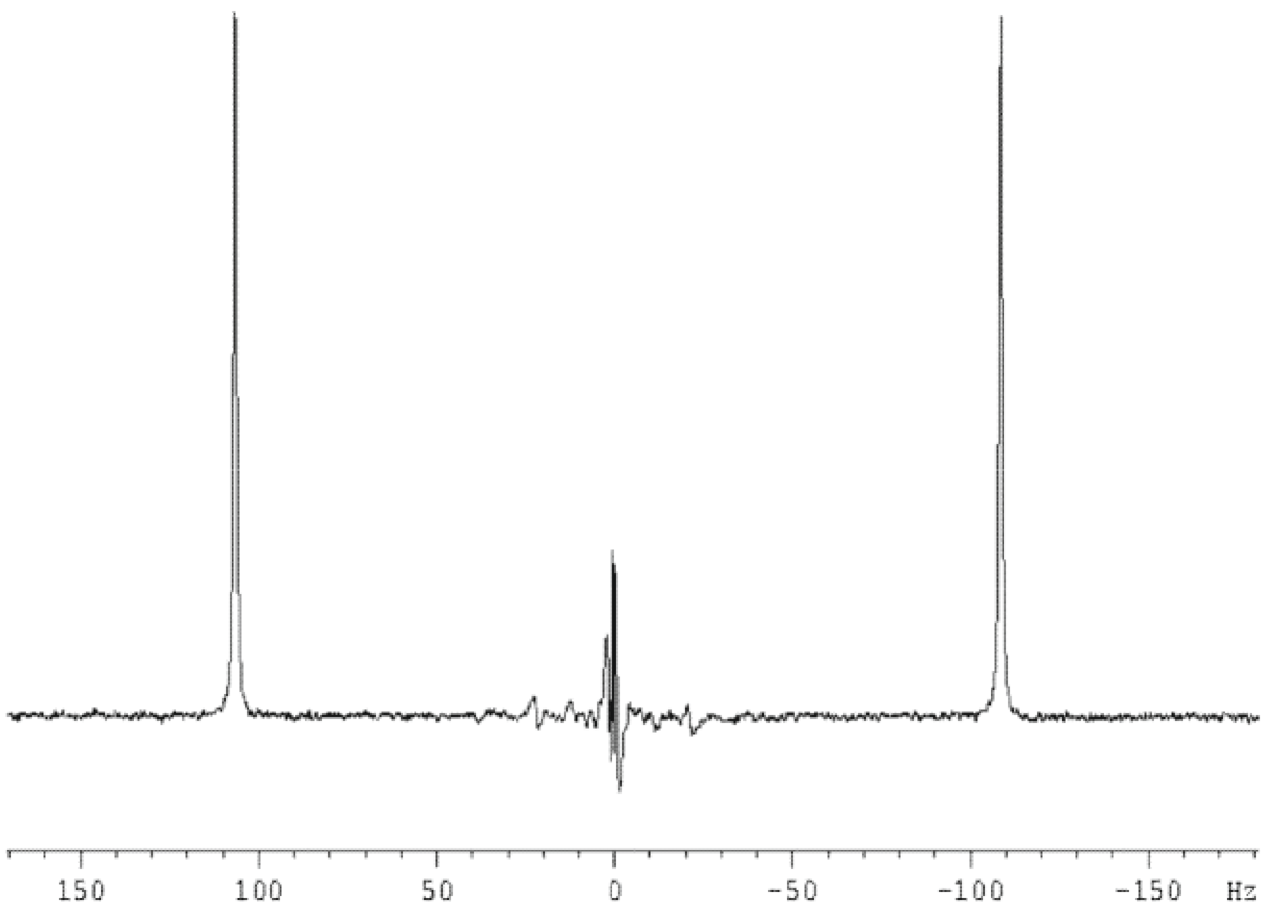
Process with ef (lb=0.3Hz)

Analysis: Without decoupling we observe antiphase satellites. Increase p3 until the 13C pulse satellites become zero. Use a suitable power level pl2 for 90 degree high power level pulse F2. Use all necessary filters.



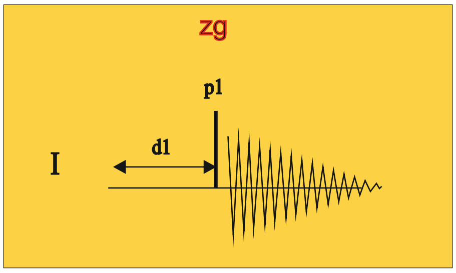
NMR Experiment: Inverse spin-echo differece test (inv)
Basic Parameter Set: HMQC1D
Pulse Program: hmqcndrd1d





NMR Experiment: Determination 90 degree 31P high power
Basic Parameter Set: P31
Pulse Program: zg

Sample: 0.0485M Triphenylphosphate (TPP) in acetone-d6

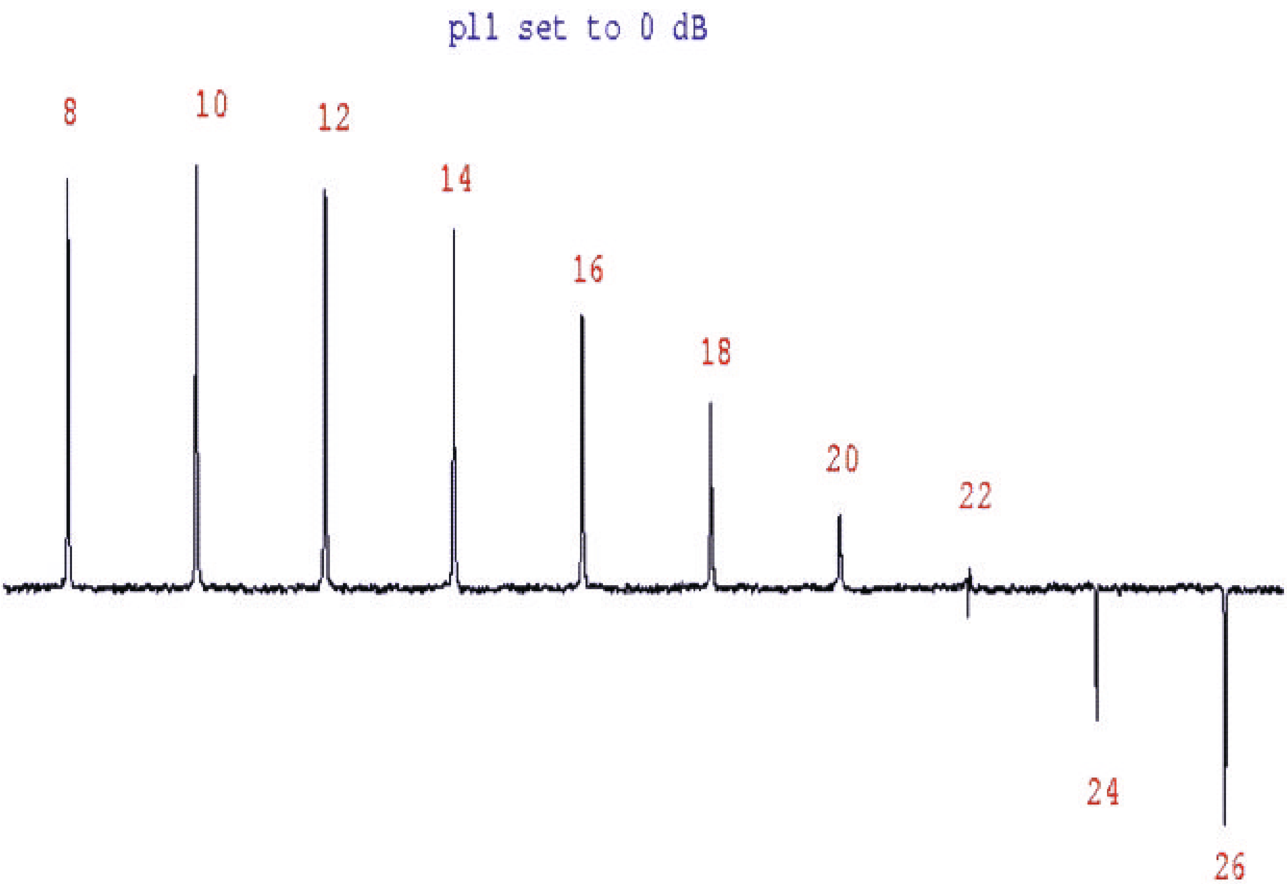


Spin: on

Basic acquisition parameters:
d1 60s
ns 1 and ds 0
o1p=-16ppm

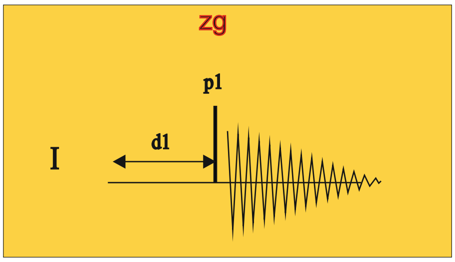
Process with ef (lb=5Hz)

Analysis: The pulse length strongly depends on the transmitter power and probe design. Remember to tune and match before you start calibration with **popt**. The first maximum is the 90°, the first null point is 180° and the second 360°



NMR Experiment: Sensitivity test for 31P (snp)
Basic Parameter Set: P31
Pulse Program: zg

Sample: 0.0485M Triphenylphosphate (TPP) in acetone-d6

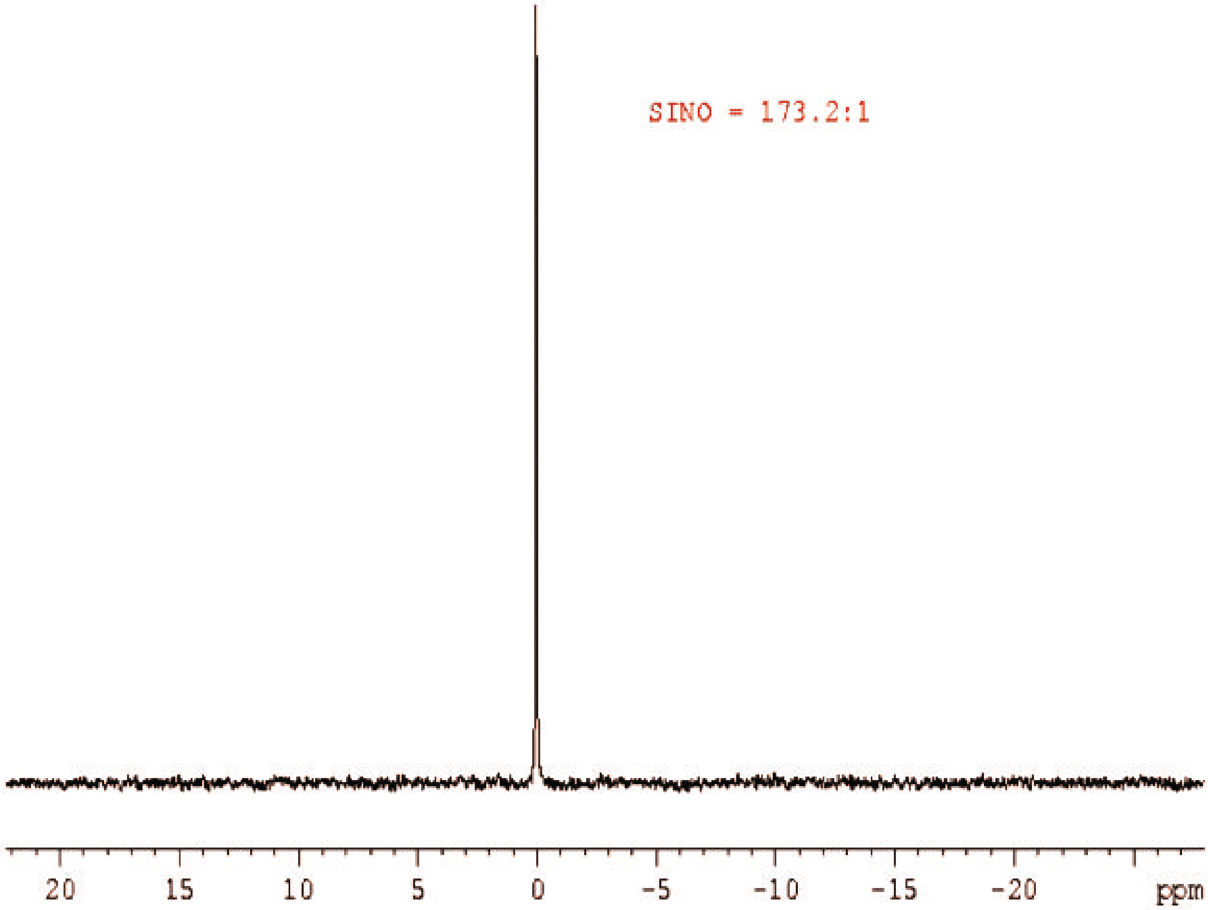


Spin: on

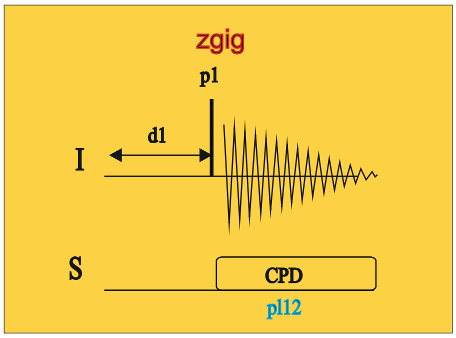
Basic acquisition parameters:
d1 60s
ns 1 and ds 0
o1p=-16ppm
Set p1 at the 90° degree pulse at power level pl1.

Process with ef (lb=5Hz)

Analysis: The 31P sensitivity is determined on the fully coupled resonance line of TPP. S/N is calculated using sino over a range of 5 ppm.



NMR Experiment: Sensitivity test for 15N (snn)
Basic Parameter Set: N15IG
Pulse Program: zgig



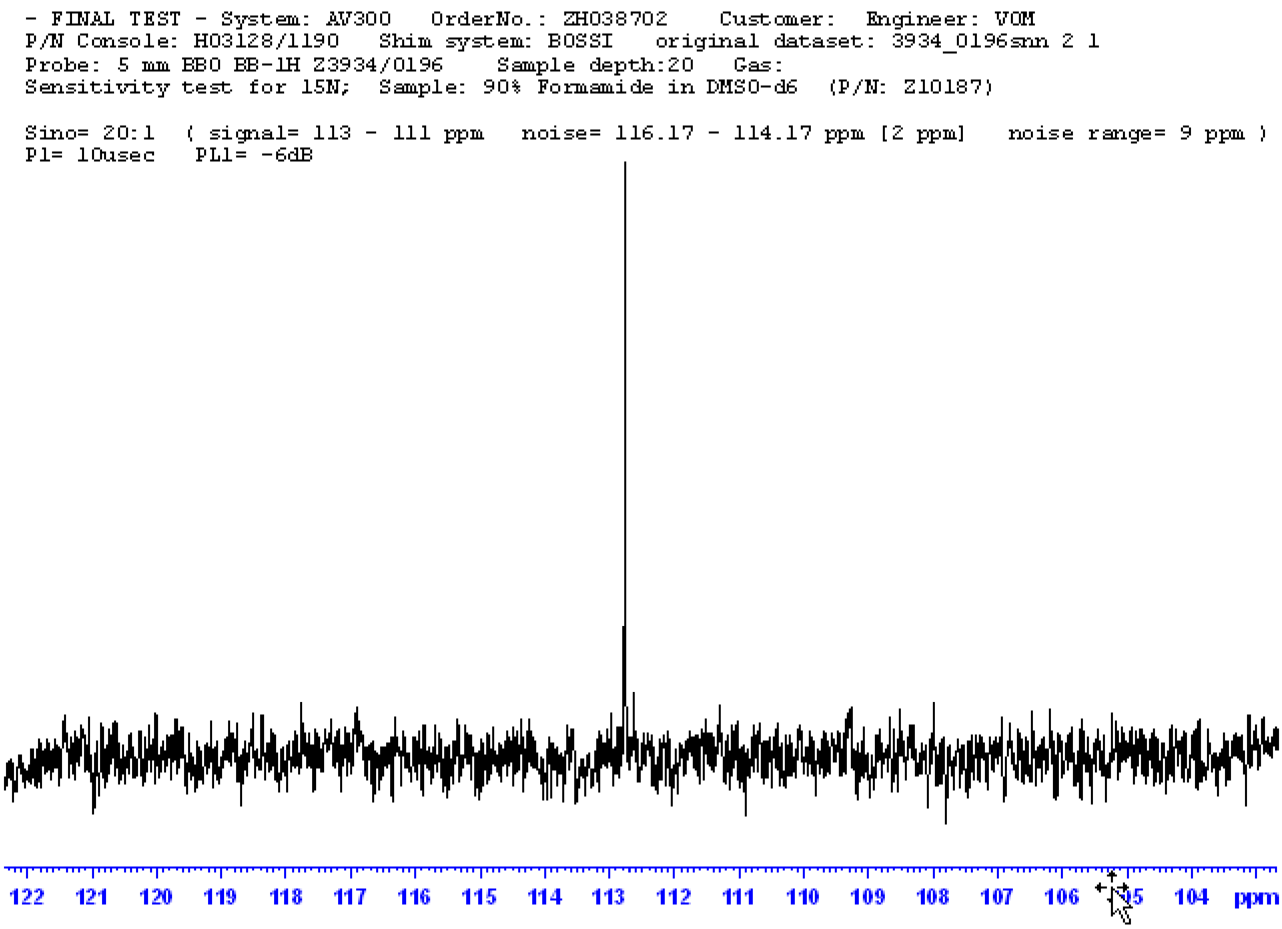
Sample: 90% Formamide in DMSO-d6

Spin: on

Basic acquisition parameters:
d1 120s
ns 1 and ds 0
o1p=112.5ppm o2p=7.3ppm sw=20ppm
Set pl12 as determined for CPD 1H decoupling.

Process with ef (lb=0.3Hz)

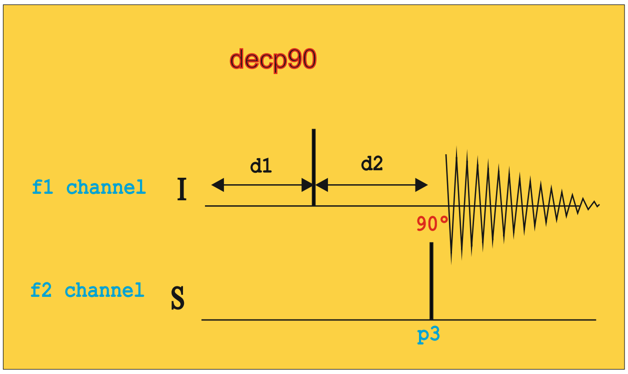
Analysis: The nitrogen sensitivity is measured on the formamide resonance using **sino** over a range of 2ppm. Before this, you must determine the 90 degree pulse 15N (p1) at power level pl1 using **popt**.



NMR Experiment: Determination 90 degree 15N high power decoupling (pun)
Basic Parameter Set:
Pulse Program: decp90

Sample: 100mM urea 15N, 100mM Ch3OH 13C in DMSO-d6

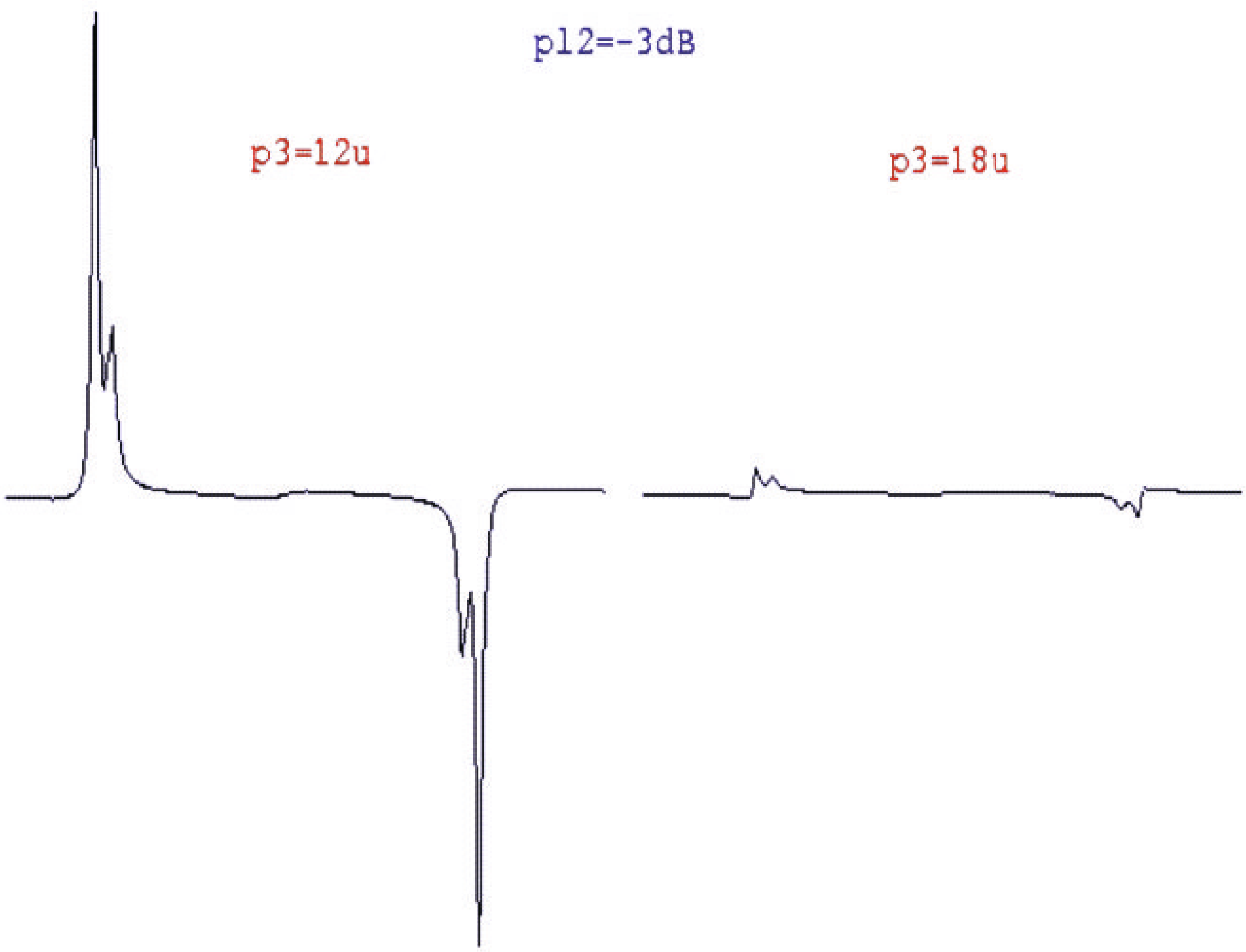
Spin: off



Basic acquisition parameters:
d1 24s
ns 1 and ds 0
o1p=4ppm o2p=76ppm sw=8ppm
CNST2=88.5Hz (d2=5.649ms)

Process with ef (lb=0.3Hz)

Analysis: Without decoupling we observe an antiphase doublet. Increase p3 (at power level pl2) until the doublet becomes zero. For three-channel system use pulprog=decp90f3.

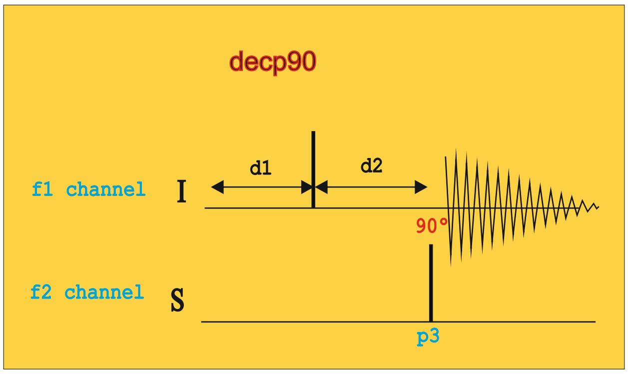


NMR Experiment: Determination 90 degree 15N low power decoupling (GARP) (pun)
Basic Parameter Set:
Pulse Program: decp90

Sample: 100mM urea 15N, 100mM Ch3OH 13C in DMSO-d6

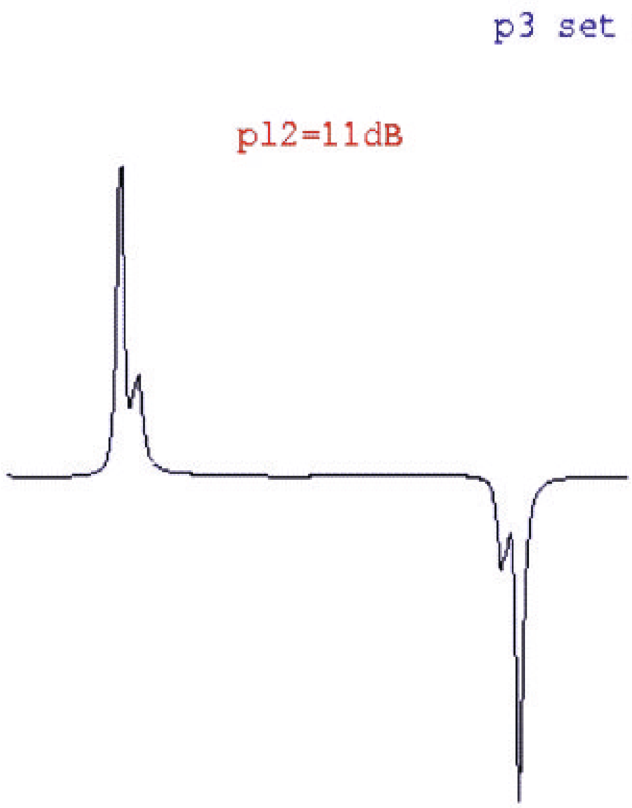
Spin: off

Basic acquisition parameters:
d1 2s
ns 1 and ds 0
o1p=4ppm o2p=76ppm sw=8ppm
CNST2=88.5Hz (d2=5.649ms)

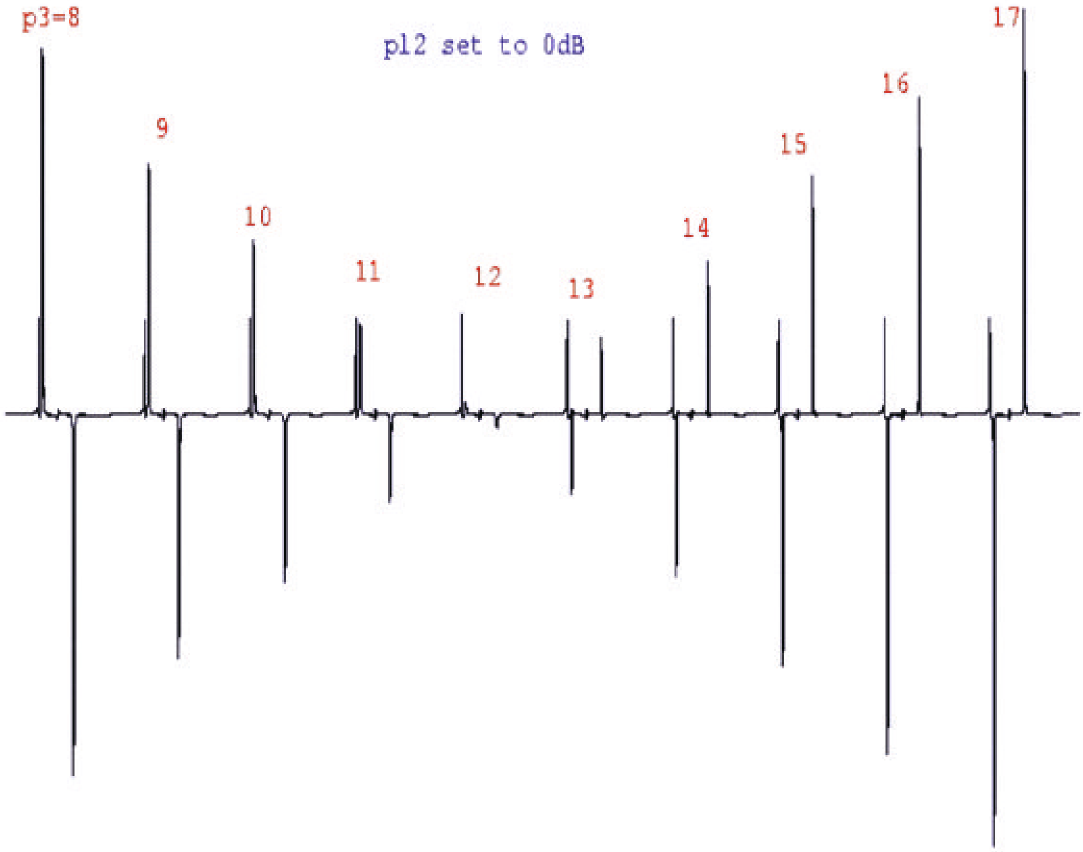


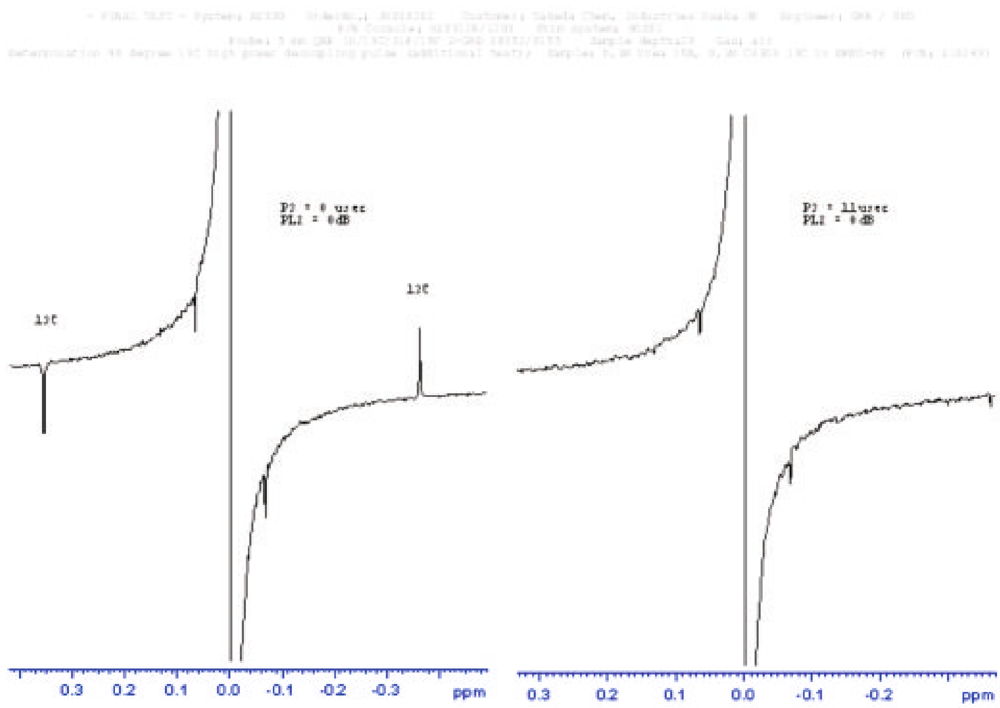
Process with ef (lb=0.3Hz)

Analysis: Without decoupling we observe an antiphase doublet. Set p3=180-200us and decrease power level pl2 staring with a pl2=30dB until the doublet becomes zero. For three-channel system use pulprog=decp90f3.

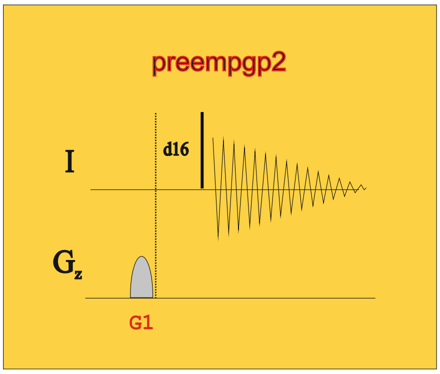


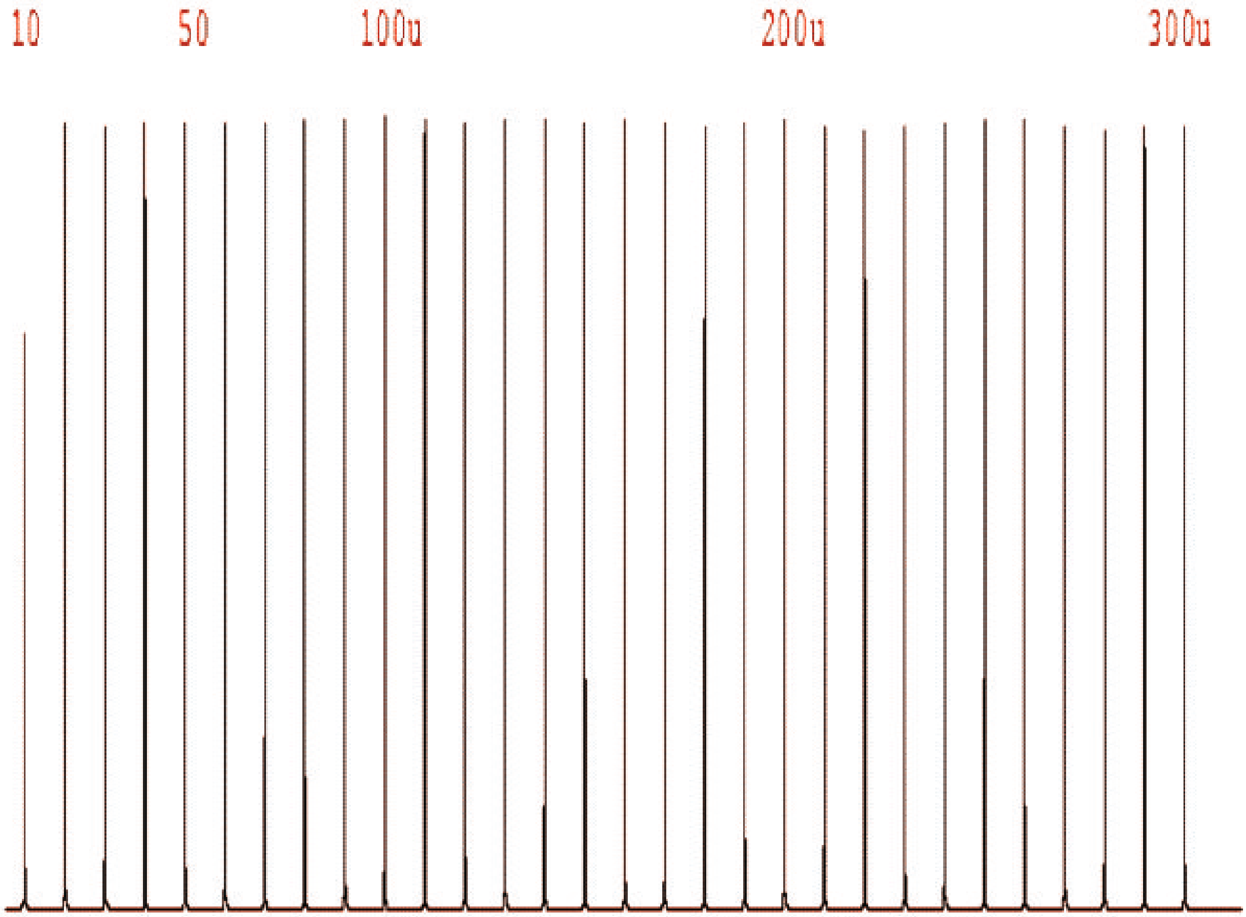
CPD 15N pulse according to:
pcpd= 300 - (BF1(1H)/5)


p3=8
pl2 set to 0dB
9
10
11
12
13
14
15
16
17

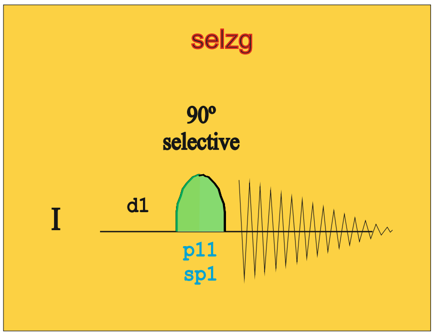

13C
13C
0.3
0.2
0.1
0.0
-0.1
-0.2
-0.3
ppm
0.3
0.2
0.1
0.0
-0.1
-0.2
ppm

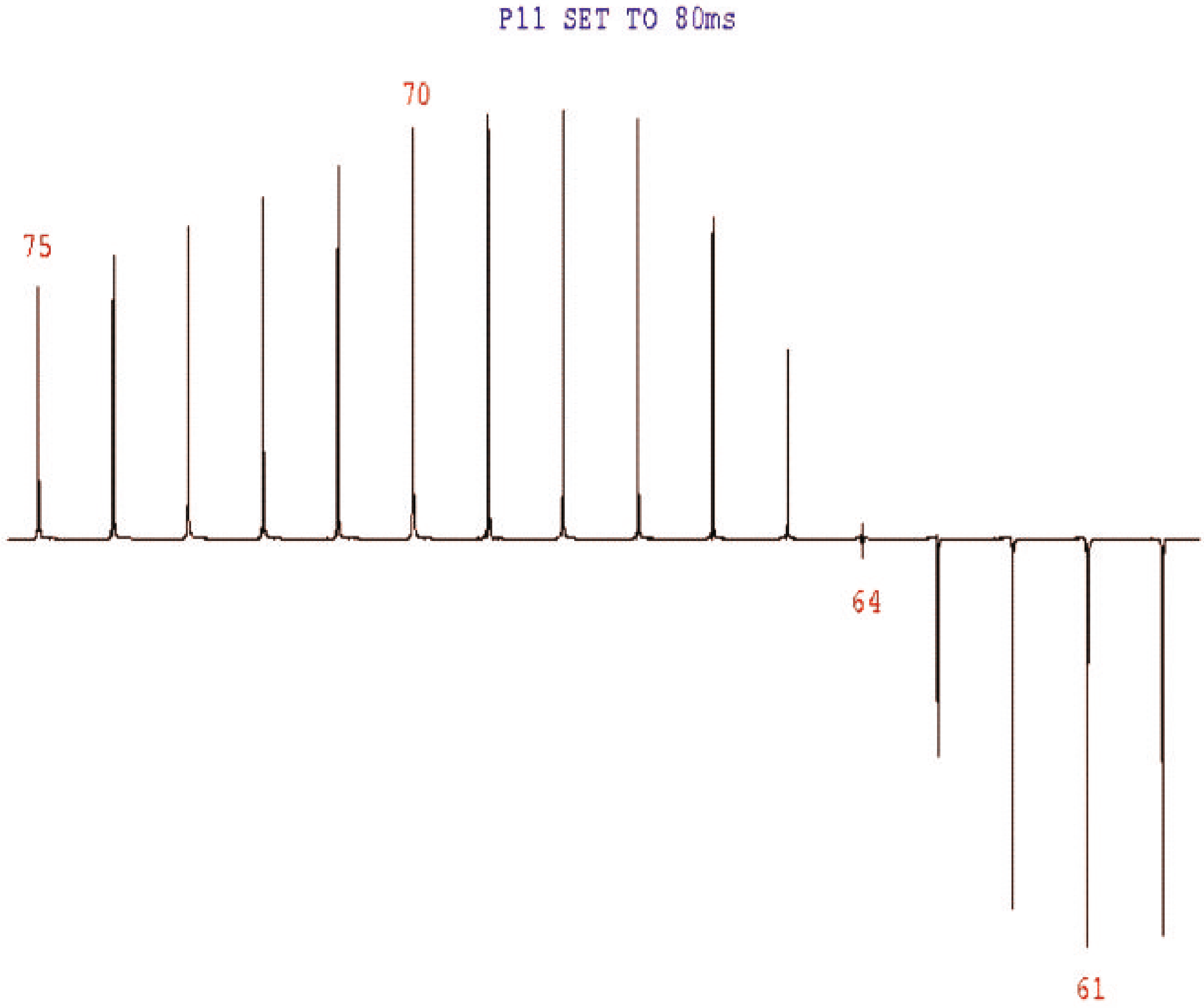
NMR Experiment: Gradient Recovery Test
Basic Parameter Set: PROTON
Pulse Program: preemgp2



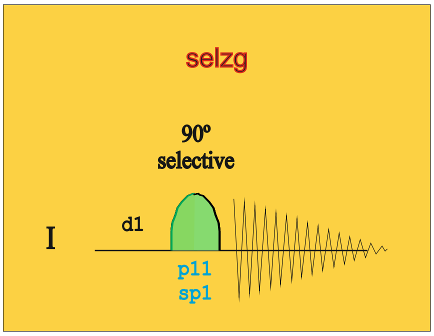


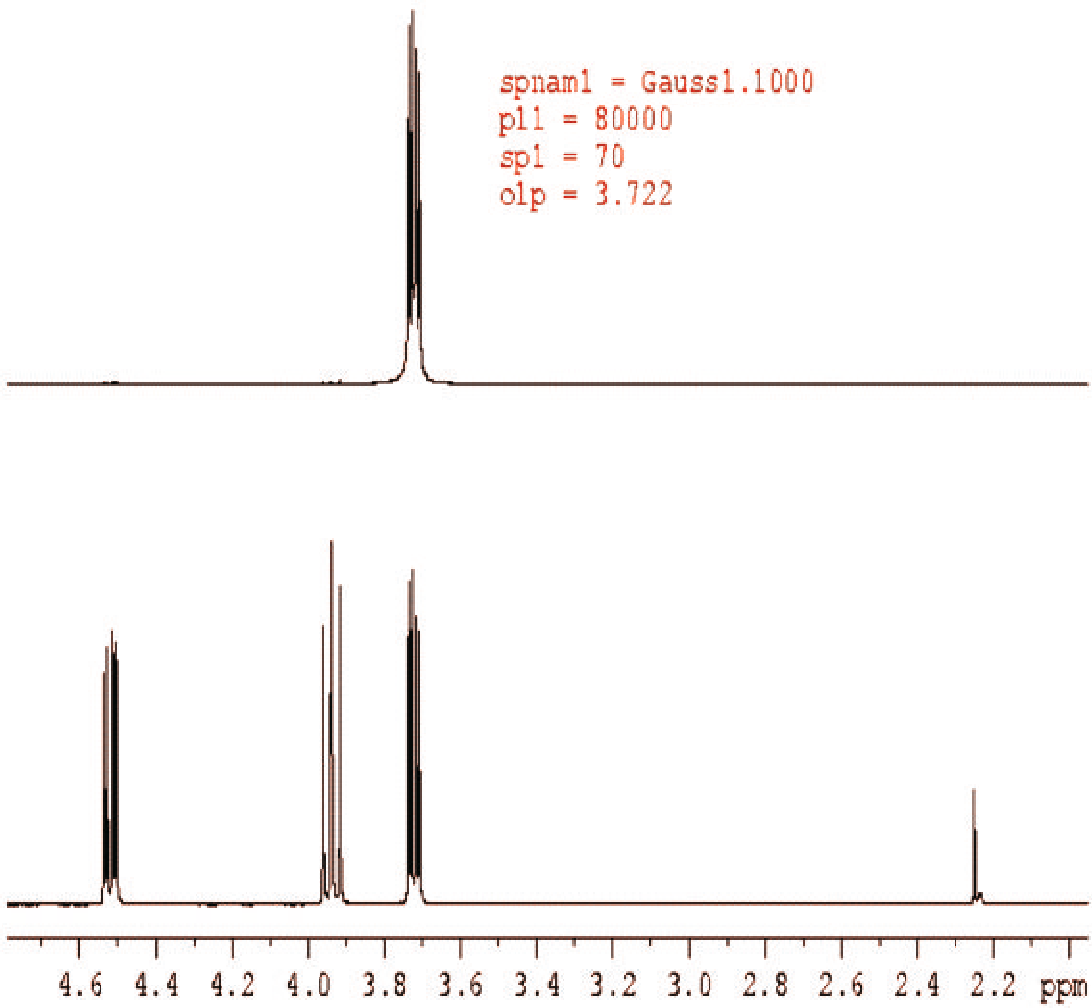
NMR Experiment: Determination of selective shaped 90 degree 1H pulse
Basic Parameter Set: SELZG
Pulse Program: selzg



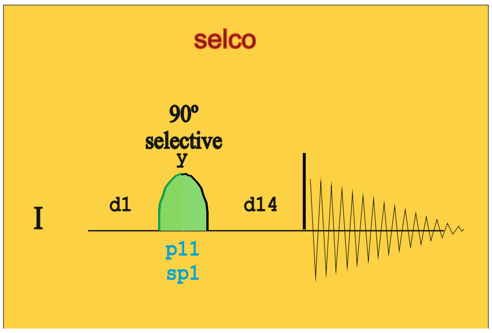


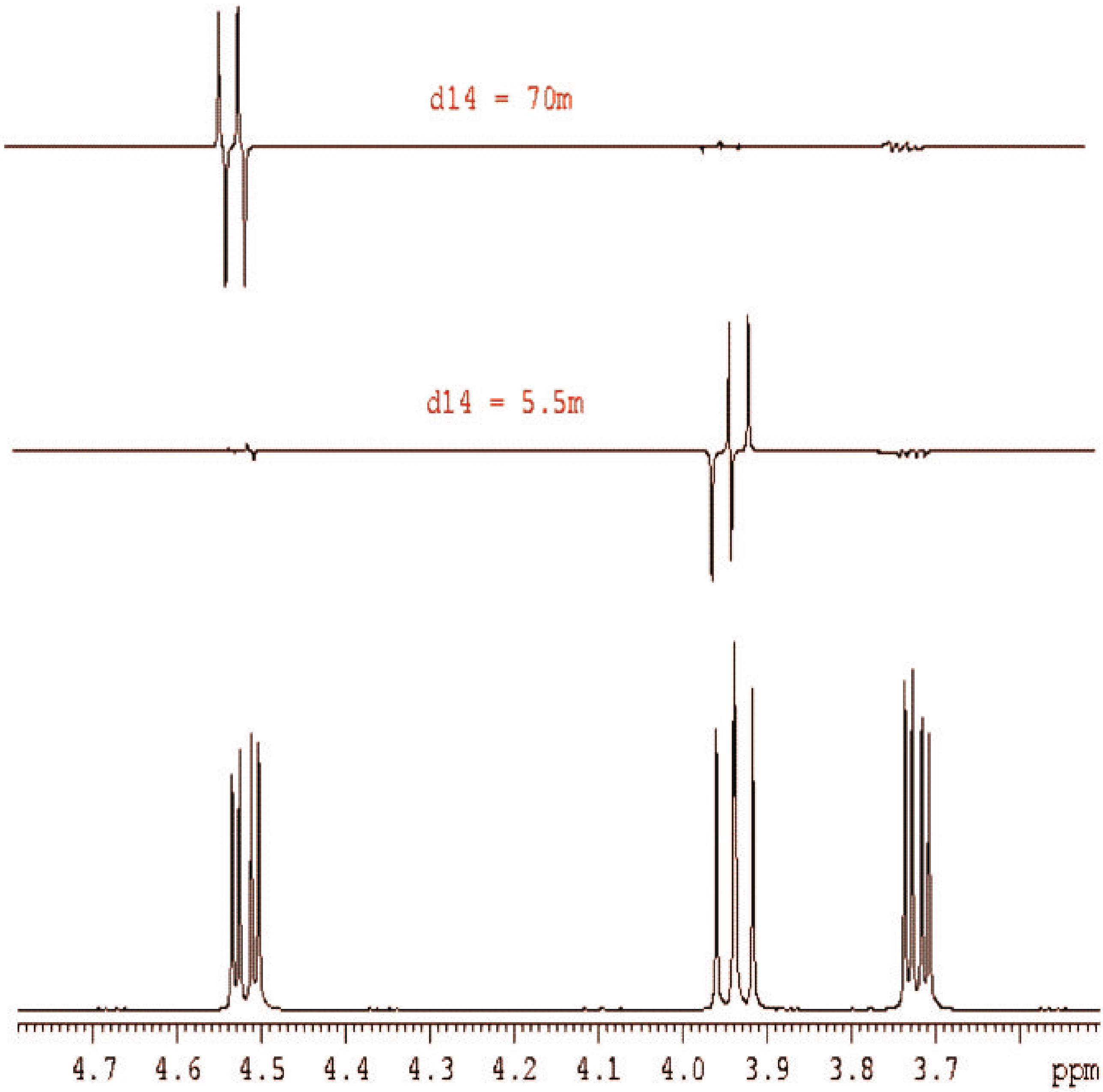
NMR Experiment: Selective Excitation test
Basic Parameter Set: SELZG
Pulse Program: selzg



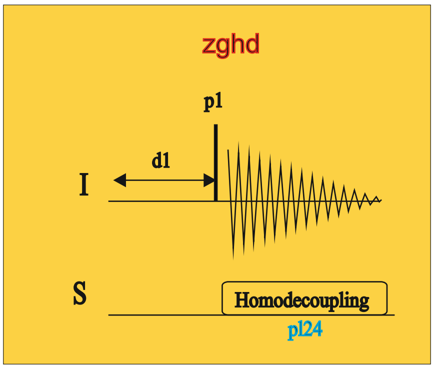


NMR Experiment: Selective Experiment test
Basic Parameter Set: SELCO
Pulse Program: selco





NMR Experiment: Homodecoupling test
Basic Parameter Set: PROHOMODEC
Pulse Program: zghd

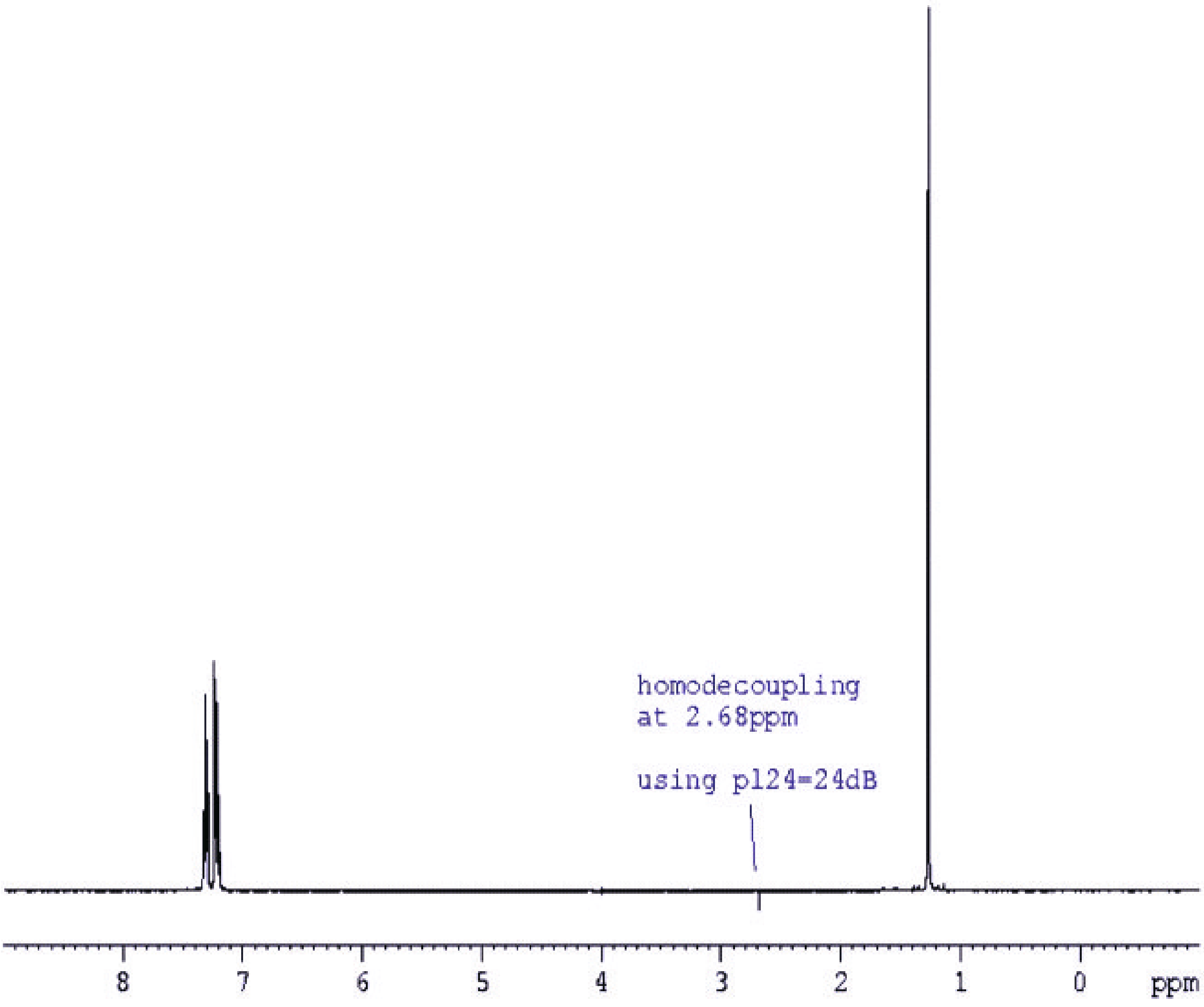


Sample:0.1% Ethylbenzene in CDCl3

Spin: off

Basic acquisition parameters:
d1 3
ns 8 and ds 2
Set pl24 for hd decoupling (45-50dB)

Process with ef (lb=1Hz)

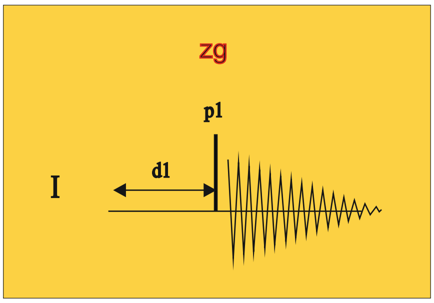


NMR Experiment: Sensitivity test for 19F (snf)
Basic Parameter Set: F19
Pulse Program: zg

Sample:0.05% Trifluorotoluene (TFT) in $CDCl_3$

Spin: on

Basic acquisition parameters:
d1 20s (T1 is about 3.5s)
ns 1 and ds 0
o1p=-63ppm sw=10ppm

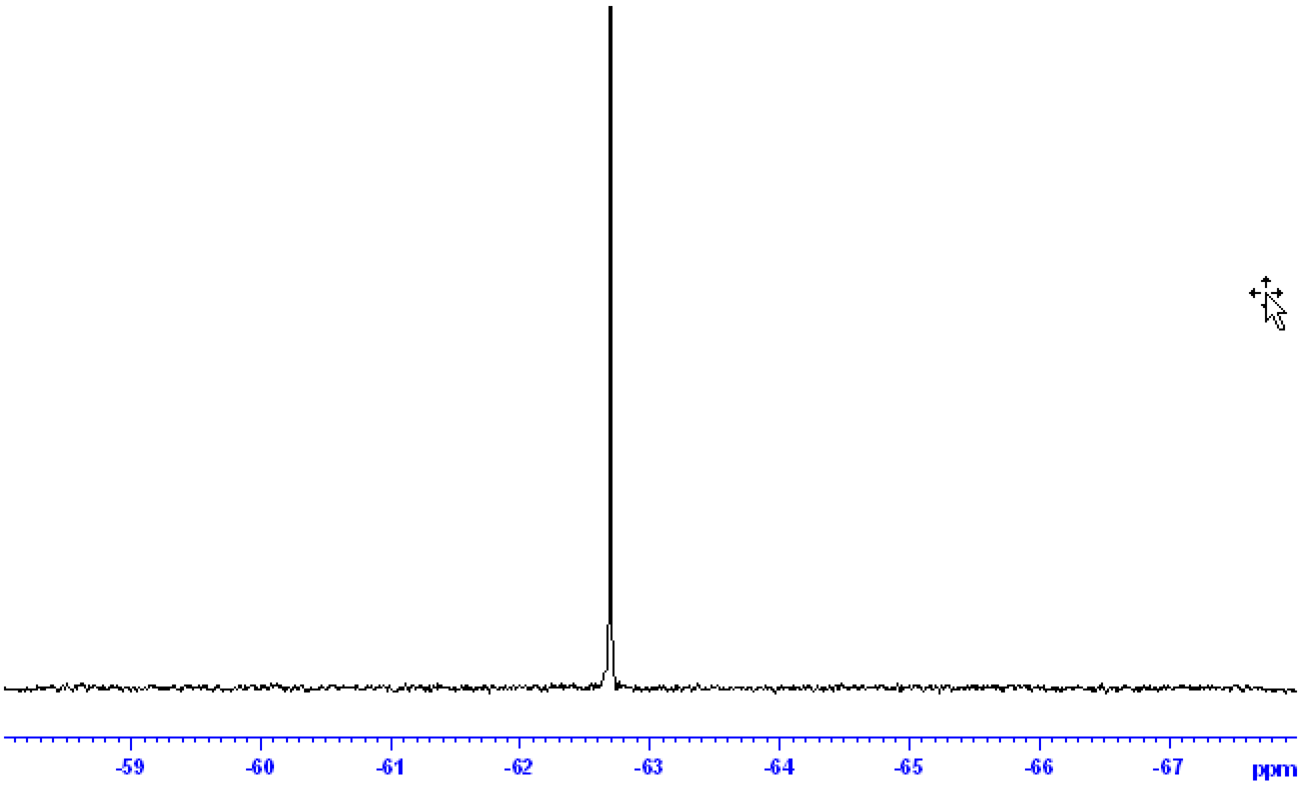


Process with ef (lb=2Hz)

Analysis: Determine the 90 degree pulse p1. The fluorine sensitivity is measured on the TFT resonance by using the **sino** program over a range of 1 ppm.

```
- FINAL TEST - System: AV300   OrderNo.: JH030202    Customer:    Engineer: GRR / SKO
P/N Console: HZ03128/1203   Shim system: BOSSI
Probe: 5 mm QNP 1H/13C/31P/19F Z-GRD Z8352/0155    Sample depth:20    Gas: air
Sensitivity test for 19F;  Sample: 0,05% TFT in CDCl3  (P/N: Z10234)

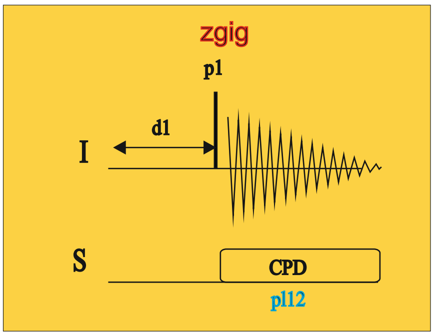
Sino= 229:1  ( signal= -61 - -65 ppm   noise= -61.42 - -62.42 ppm [1 ppm]   noise range= 4.5 ppm )
P1= 5.75usec   PL1= -5dB
```



NMR Experiment: GARP decoupling 13C
Basic Parameter Set:
Pulse Program: zgig

Sample: 100mM Urea 15N, 100mM CH3OH 13C in DMSO-d6

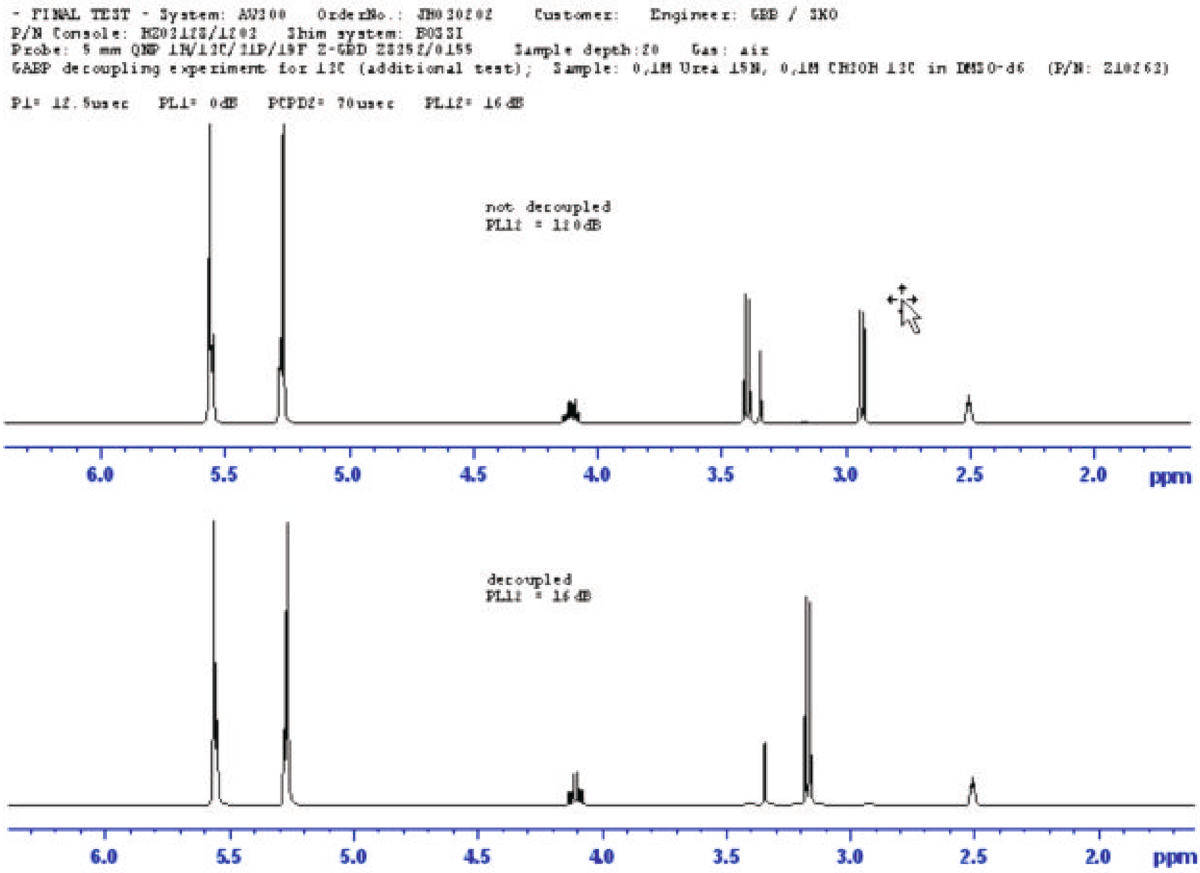
Spin: off



Basic acquisition parameters:
d1 20s
ns 4 and ds 2
o1p=4ppm sw=8ppm AQ=1s
o2p=49.5ppm (13C)
pl12 is the determined power level for garp 13C decoupling
cpdprg2=garp
pcpd2=65....80us

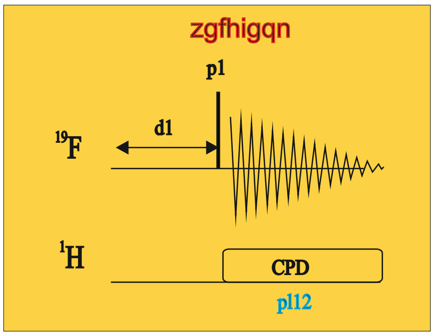
Process with ef (lb=0.3Hz)

Analysis: Use all necessary filters and also an extra 2H-stop filter in the 13C channel at the probehead input. Be aware that a GARP decoupling sequence produces always modulation spikes around large signals on a level of <0.5%.



NMR Experiment: Decoupling test 19F (with 1H CPD decoupling) (decf)
Basic Parameter Set: F19CPD
Pulse Program: zgfhigqn

Sample:0.05% Trifluorotoluene (TFT) in CDCl3



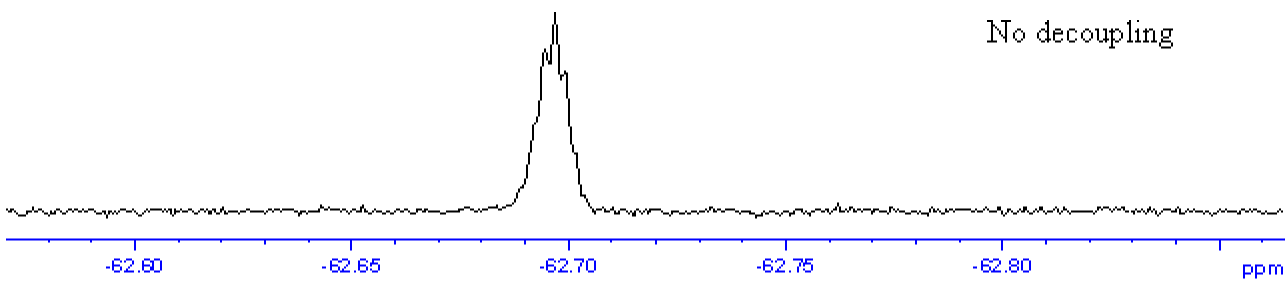
Spin: on

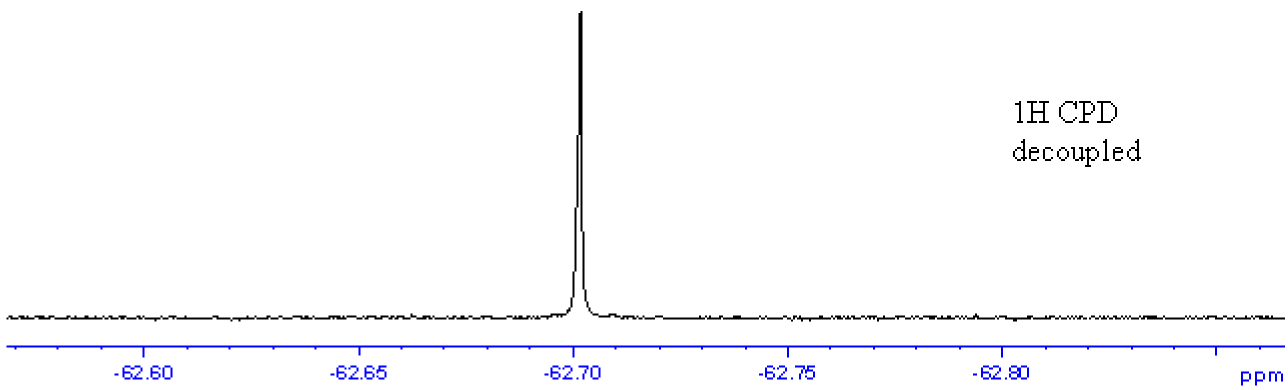
Basic acquisition parameters:
d1 1s
ns 1 and ds 0
o1p=-63ppm sw=10ppm
o2p=5ppm
pl12 is the determined power level for cpd decoupling

Process with ft

Analysis: Use all necessary filters

- FINAL TEST - System: AV300 OrderNo.: JH030202 Customer: Engineer: GRR / SKO
P/N Console: HZ03128/1203 Shim system: BOSSI
Probe: 5 mm QNP 1H/13C/31P/19F Z-GRD Z8352/0155 Sample depth:20 Gas: air
Decoupling experiment 19F observe, 1H CPD-decoupling; Sample: 0,05% TFT in CDCl3 (P/N: Z10234)

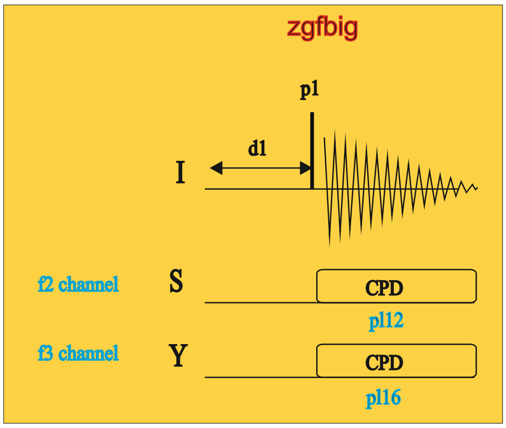




NMR Experiment:Triple Resonance experiment
Basic Parameter Set:
Pulse Program: zgfbig

Sample: 100mM Urea 15N, 100mM CH3OH 13C in DMSO-d6

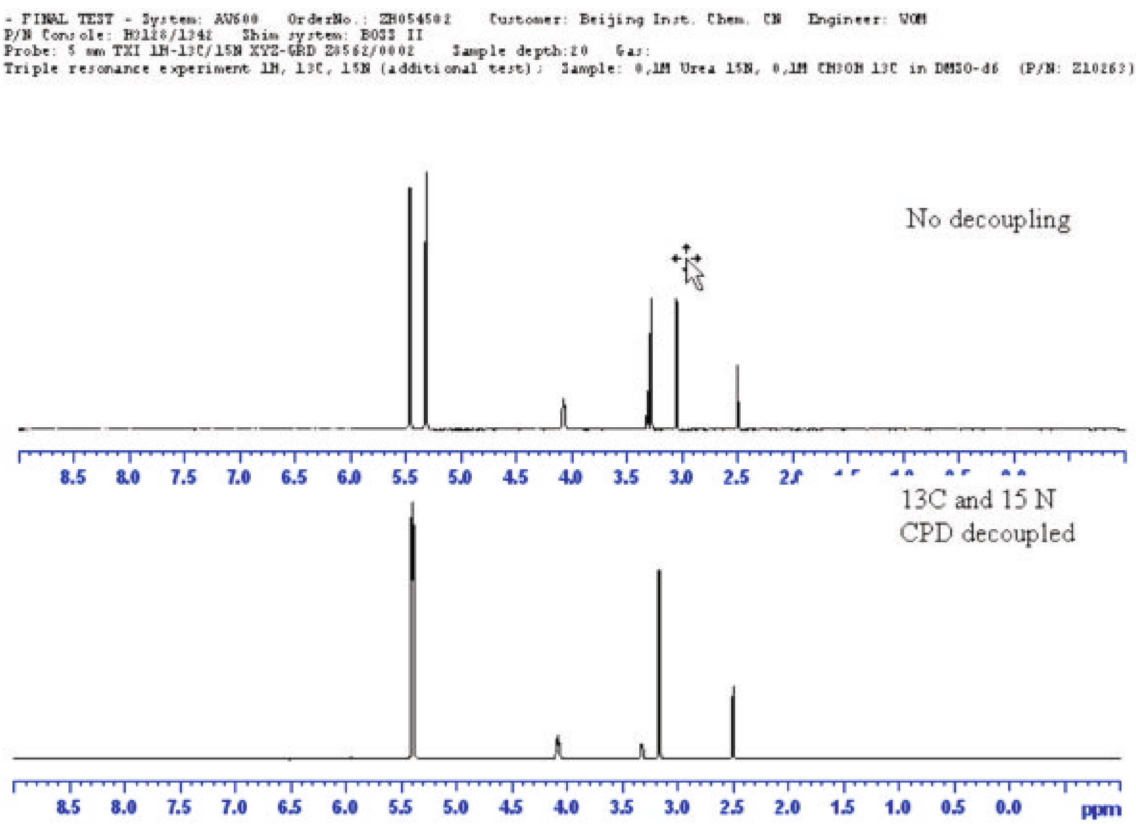
Spin: on



Basic acquisition parameters:
d1 5s
ns 4 and ds 2
o1p=4ppm sw=10ppm
o2p=49.5ppm (13C)
o3p=76ppm (15N)
cpdprg2=cpdprg3=garp
pcpd2=65....80us
pcpd3=180...200us

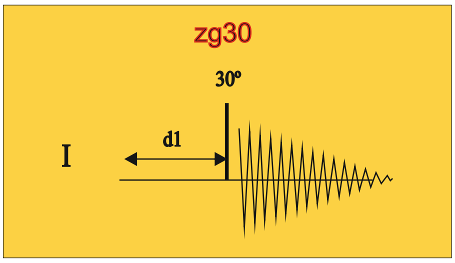
Process with ef (lb=0.3Hz)

Analysis: It is important to use enough delay in between two scans to keep the temperature stable during the experiment. Use all necessary filters (13C-pass 2H-stop and 15N-pass 2H stop)



NMR Experiment: Dataset for quad image adjustment
Basic Parameter Set:
Pulse Program: zg30

Sample: 0.1mg GdCl3/ml + 1% H2O

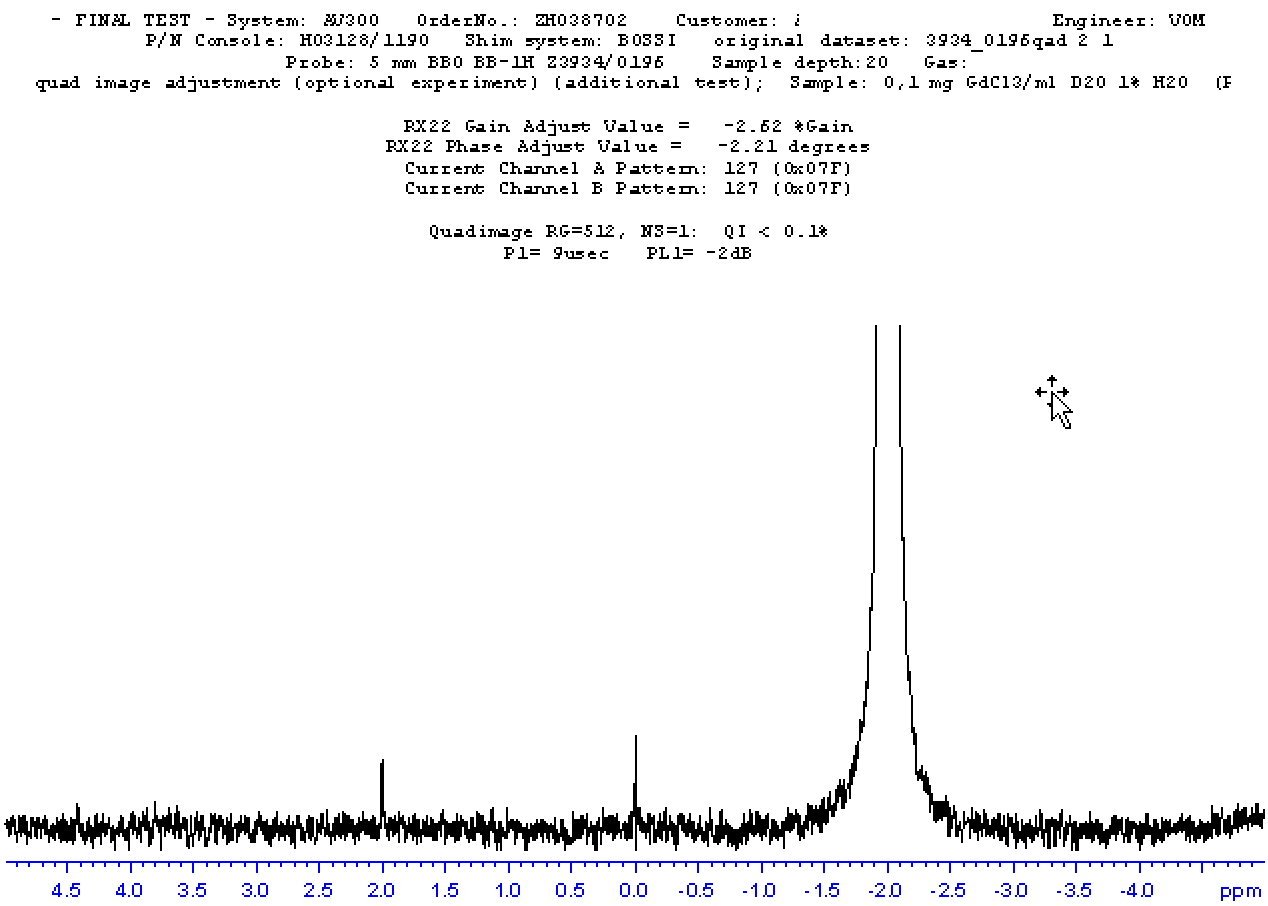


Spin: off

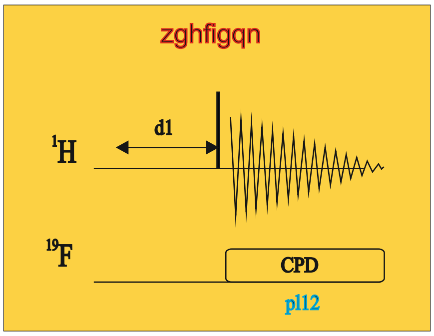
Basic acquisition parameters:
d1 1s
ns 1 and ds 0
o1p=7ppm sw=10ppm
AQMOD=QSIM

Process with ef (lb=1Hz)

Analysis: Start the Rx22 service tool from Unitool and adjust first DC offsets before phase and amplitude calibration. The residual quad image must be smaller than 1%.



NMR Experiment: Decoupling test 19F (with 19F GARP decoupling)
Basic Parameter Set: PROF19DEC
Pulse Program: zghfigqn



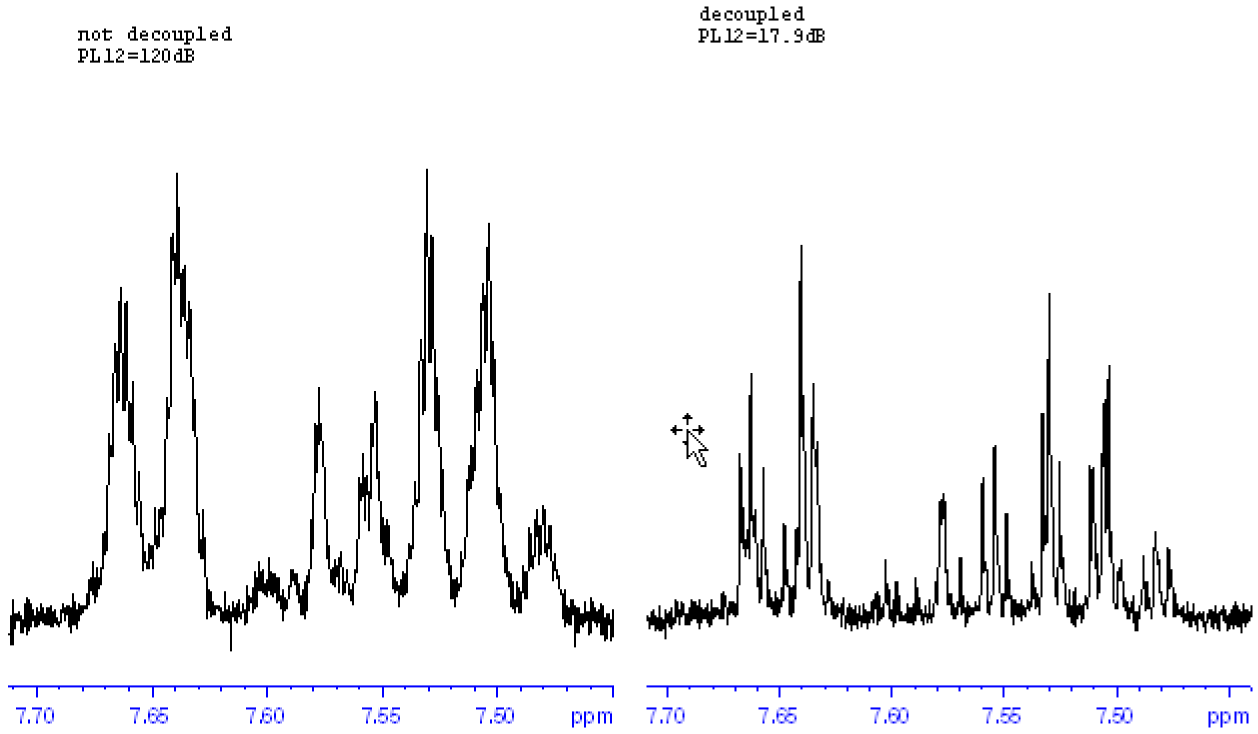
Sample:0.05% Trifluorotoluene (TFT) in CDCl3

Spin: on

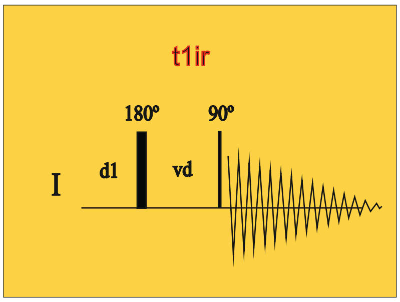
Basic acquisition parameters:
d1 5s
ns 8 and ds 0
o1p=5ppm sw=10ppm
o2p=-63ppm
pl12 is the determined power level for garp decoupling
pcpd2=95us

Process with ft

Analysis: Use all necessary filters. Pulse accroding to formula pcpd=110-(BF1_1H/20).
For instance, in a 300MHz: pcpd=110-(300/20)=95us



NMR Experiment: B1 homogeneity test 1H from HWT (b1h)
Basic Parameter Set:
Pulse Program:



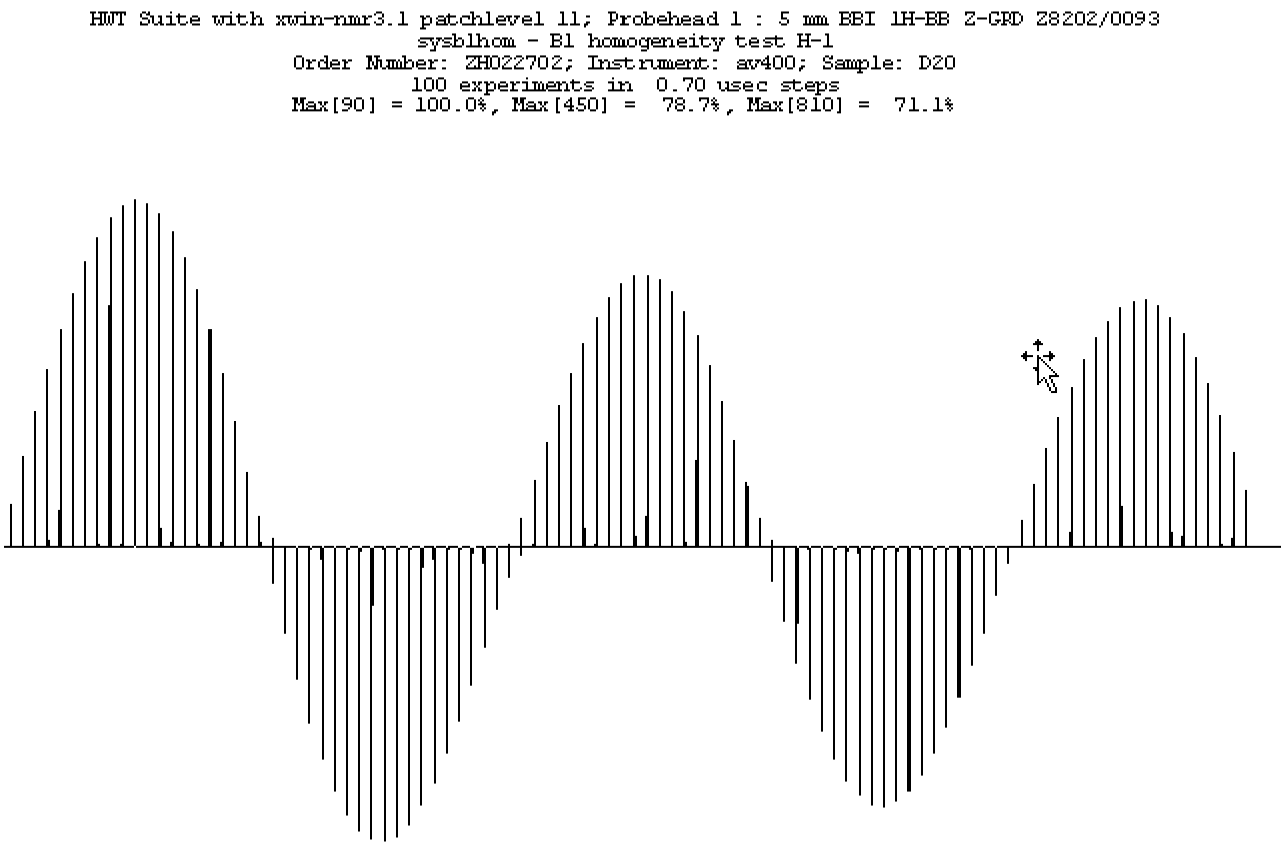
Sample: 0.1mg GdCl3/ml D2O +1% H2O, 0.05% Meth.

Spin: off

Basic acquisition parameters:
d1 10s
ns 1 and ds 0
vdlist=syst1list

Process with ef (lb=1Hz)

Analysis: HWT; select HWTgen; select B1 homgeneity test H1.
The amplitude is measured at 810 degree as a percent level of the 90 degree pulse width



BRUKER
PULSE PROGRAM
CATALOGUE

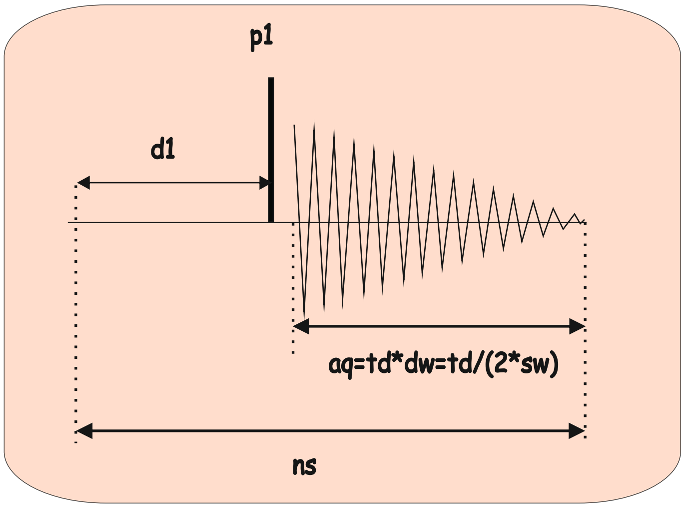
NMRGuide

BASIC 1D PULSE SEQUENCES

BASIC ACQUISITION PARAMETERS IN 1D

Once a parset has been loaded and getprosol optionally applied, the user can check and modify some acquisition parameters before start acquisition:

d1 is pre-scan delay
ns is the number of scans
o1p is the center of the spectrum
sw is the spectral width
td is the number of points to be acquired
aq is acquisition time
dw is dwell time
o2p is the decoupler frequency (optional)



BASIC PROCESSING PARAMETERS IN 1D

Processing parameters can be checked and modified fromt he ProcPars section:

si is the number of points to be processed (zero-filling)
wdw is the window function to be applied on the fid
ft: Fourier transformation
ef (em+ft): transform with a exponential using line broadening (**lb**)
gf (gm+ft): Transform with a Gaussian using **lb** and **gb**
apk is automatic phase correction
abs is automatic baseline correction
sr is the spectrum reference frequency

Basic 1D pulse sequences

- **Standard Experiments:**

Conventional ^{1}H spectrum (**zg30** / **zg** / **zg0** | PROTON)
Acquired as 2D (**zg2d**)

1D ^{1}H Homodecoupling (**zg0hd** / **zghd** / **zghd.2** | PROHOMODEC)
1D ^{1}H Band-selective homodecoupling (**zghc** / **zghc.2** / **zghc.3**)
NOEDIFF experiment:
Single irradiation (**zgf2pr**)
Using frequency list (**noediff** / **noediff.2** / **noedif.2** | NOEDIFF)
Irradiation multiplet frequencies within one multiplet (**noemul**)

^{13}C spectrum with selective ^{1}H decoupling using CW (**zgcw30** / **zgcw** / **zg0cw**)
^{1}H-decoupled ^{13}C spectrum (**zgdc30** / **zgdc** / **zg0dc** | C13CPD)
^{1}H-coupled ^{13}C spectrum (**zggd30** / **zggd** / **zg0gd** | C13GD)
^{1}H-decoupled ^{13}C spectrum without NOE (**zgig30** / **zgig** / **zg0ig** | C13IG)

^{1}H,^{31}P-decoupled ^{13}C spectrum without NOE (**zgfbig**)
^{1}H,^{31}P-decoupled ^{13}C spectrum without NOE in the channel f3 (**zgdcf2igf3**)
^{1}H,^{31}P-decoupled ^{13}C spectrum without NOE (**zgigf2igf3**)

Antiring sequence (**aring**, **aring2**)
1D sequence for suppression of background signals using composite pulse (**zgbs**)

UDEFT sequence with flip back pulse at the end of acquisition to decrease relaxation delay (**udeft**)

- **Multinuclear Applications:**

^{31}P-decoupled 1D ^{1}H spectrum (**zgig30** / **zgig** | PROP31DEC)
^{11}B-decoupled 1D ^{1}H spectrum (**zgig30** / **zgig** | PROB11DEC)
^{1}H-decoupled ^{15}N spectrum without NOE (**zgig** / **zgf3ig** | N15IG)
^{1}H-coupled ^{15}N spectrum without NOE (**zg** | N15)
^{1}H-decoupled ^{31}P spectrum (**zgpg30** | P31CPD)
^{1}H-coupled ^{31}P spectrum (**zg30** | P31)

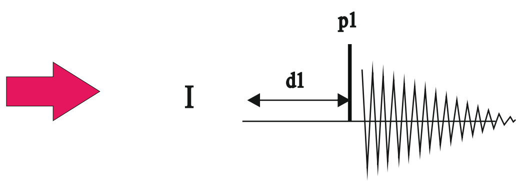
- **Standard BRUKER parameter sets available for other nuclei:**

1D ^{11}B spectrum (**zg** | B11ZG)
1D ^{17}O spectrum (**zg** | O17ZG)
1D ^{23}Na spectrum (**zg** | NA23ZG)
1D ^{27}Al spectrum (**zg** | AL27ND)
1D ^{1}H-decoupled ^{29}Si spectrum (**zgig** | SI29IG)
1D ^{35}Cl spectrum (**zg** | CL35ZG)
1D ^{37}Cl spectrum (**zg** | CL37ZG)
1D ^{71}Ga spectrum (**zg** | GA71ZG)
1D ^{71}Ga spectrum (**zg** | SE77ZG)
1D ^{103}Rh spectrum (**zg** | RH103ZG)
1D ^{111}Cd spectrum (**zg** | CD111ZG)
1D ^{113}Cd spectrum (**zg** | CD113ZG)
1D ^{1}H-decoupled ^{119}Sn spectrum (**zgig** | SN119IG)
1D ^{195}Pt spectrum (**zg** | PT195ZG)
1D ^{199}Hg spectrum (**zgpg** | HG199CPD)

Basic NMR Elements:
Pre-Scan Delay
Read Pulse
Acquisition Period
Phase Cycle

```
1 ze
2 30m
  d1
  p1 ph1
  go=2 ph31
  30m mc #0 to 2 F0(zd)
exit


ph1=0 2 2 0 1 3 3 1
ph31=0 2 2 0 1 3 3 1
```

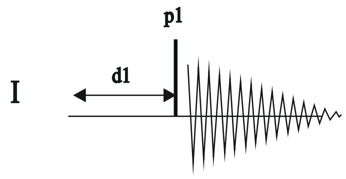


A) Pre-Scan Delay: Represented by **d1** (in seconds), is a period to allow relaxation to the original z position.
B) Read Pulse: Represented by **p1** (in microseconds and applied at pl1 power) is the time needed to create transverse magnetization from the original Iz magnetization.
C) Acquisition Period: represented by **go** (the duration is **aq** in seconds) is the time required for the receiver to monitor the free-induction decay in the transverse plane.
D) A phase Cycle is always included in all pulse programs to specify from which axis pulses and receivers are applied:

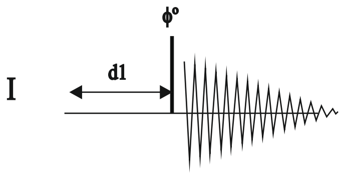
0 is x axis
1 is y axis
2 is -x axis
3 is -y axis

A Cyclops or Exorcycle loop is usually applied for better signal selection and artefact sippression

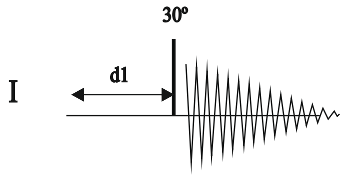
zg
zgadc
zg2d

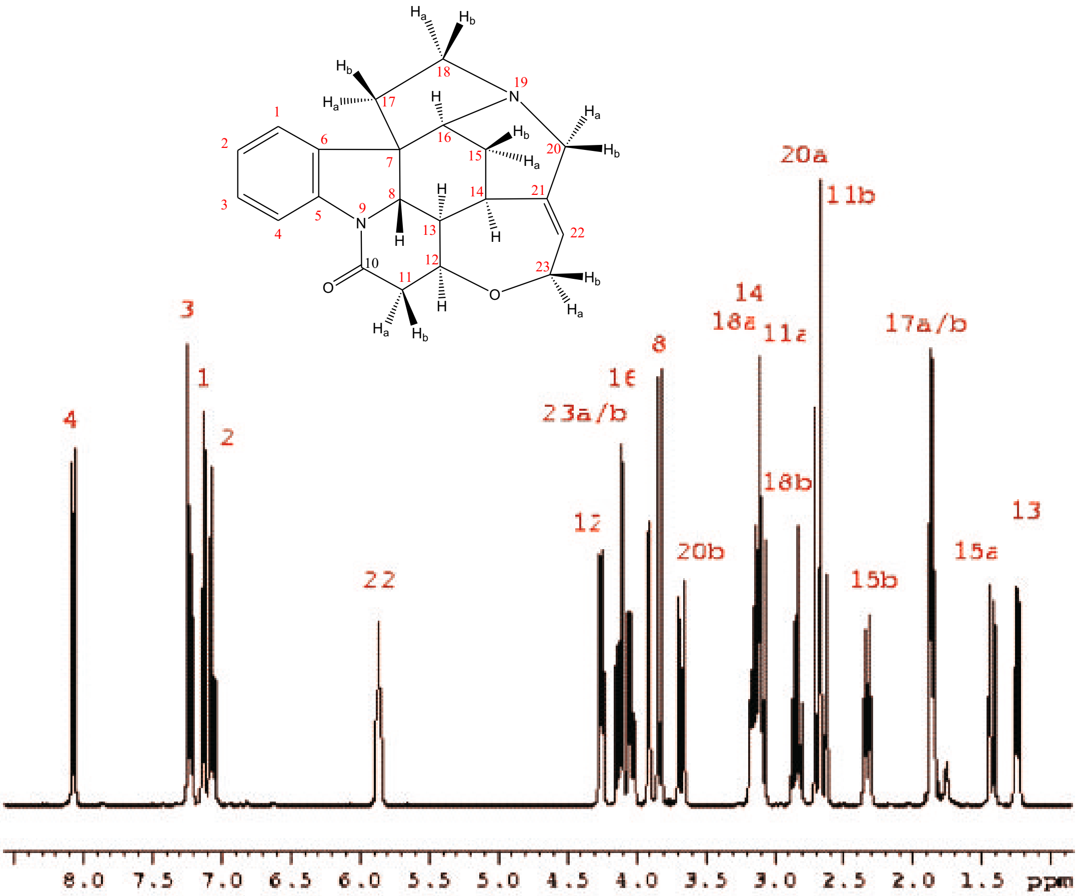


zg0



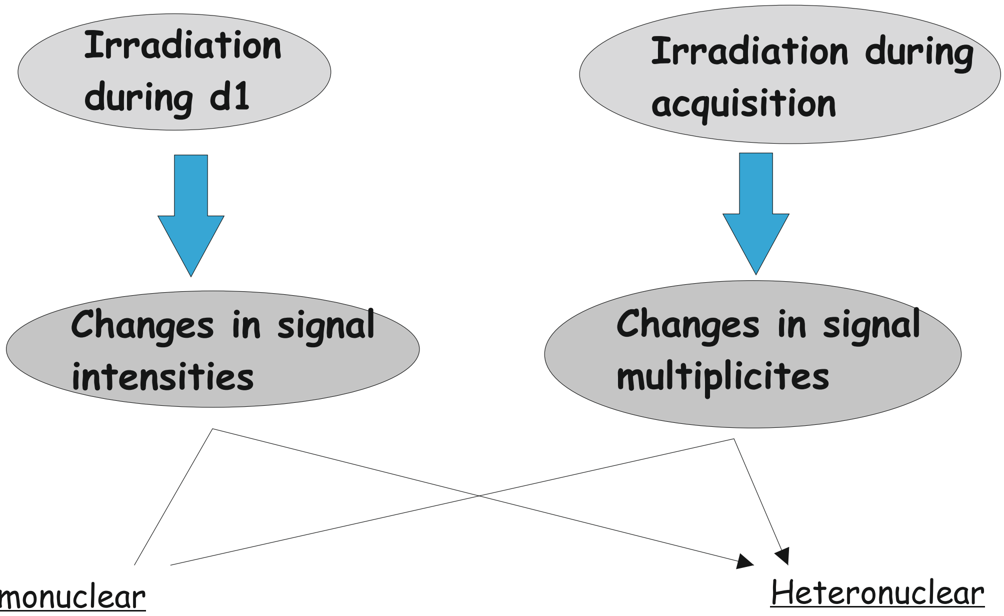
zg30



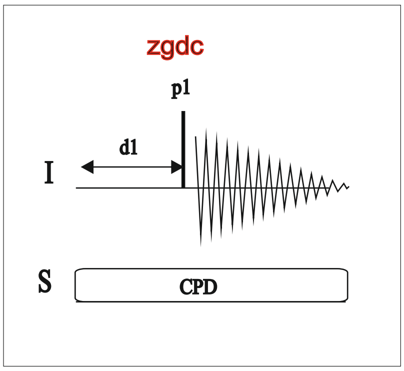


1H NMR spectrum Analysis from: Chemical shifts, Multiplicities, Coupling Constants, Linewidths and Integration

Double-Resonance Experiments

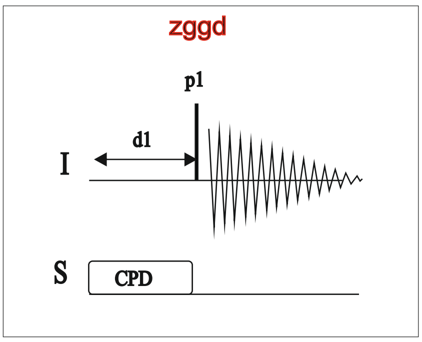


homodecoupling
(rpar PROHOMODEC all)

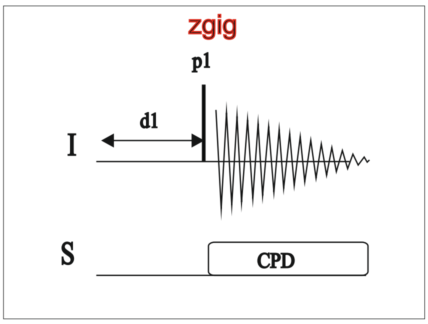


Conventional
^{13}C decoupled
(rpar C13CPD all)

Noediff
(rpar NOEDIF all)
presat
(rpar ZGPR all)



^{13}C coupled with NOE
(gated version)
(rpar C13GD all)

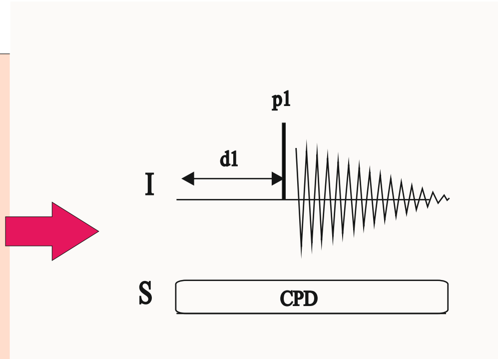


Decoupled ^{13}C without NOE effect
(for quantitation purposes uses a large d1 value!!!)
(rpar C13IG all)

Basic NMR Elements:
Broadband Decoupling

```
...
1 ze
  d11 pl12:f2
2 30m do:f2
  d11 cpd2:f2
  d1
  p1 ph1
  go=2 ph31
  30m do:f2 mc #0 to 2 F0(zd)
exit
....

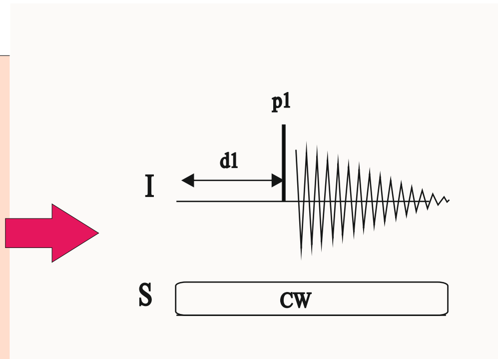
;pl12: f2 channel - power level for CPD/BB decoupling
;cpd2: decoupling according to sequence defined by cpdprg2
;pcpd2: f2 channel - 90 degree pulse for decoupling sequence
```

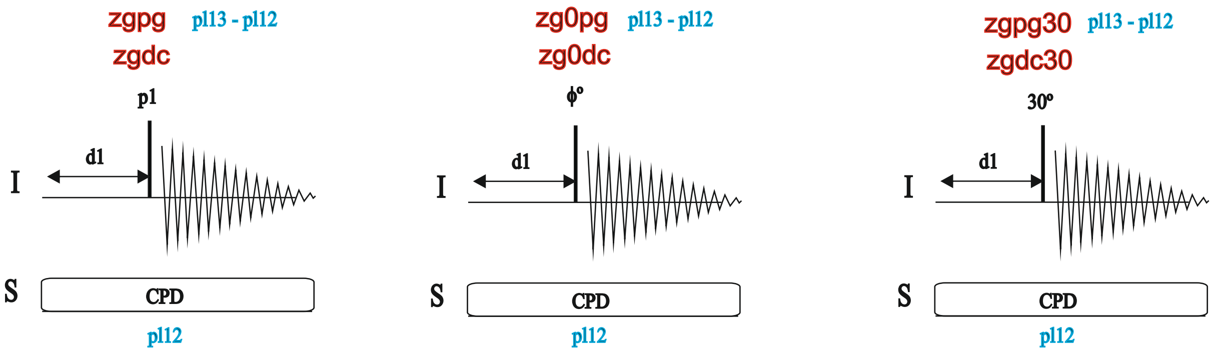


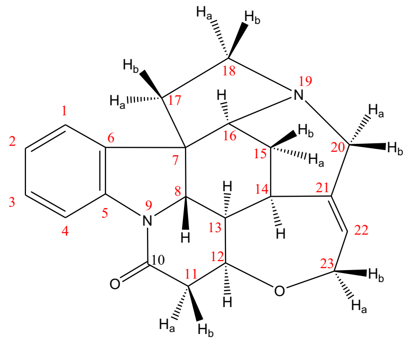
Selective Decoupling

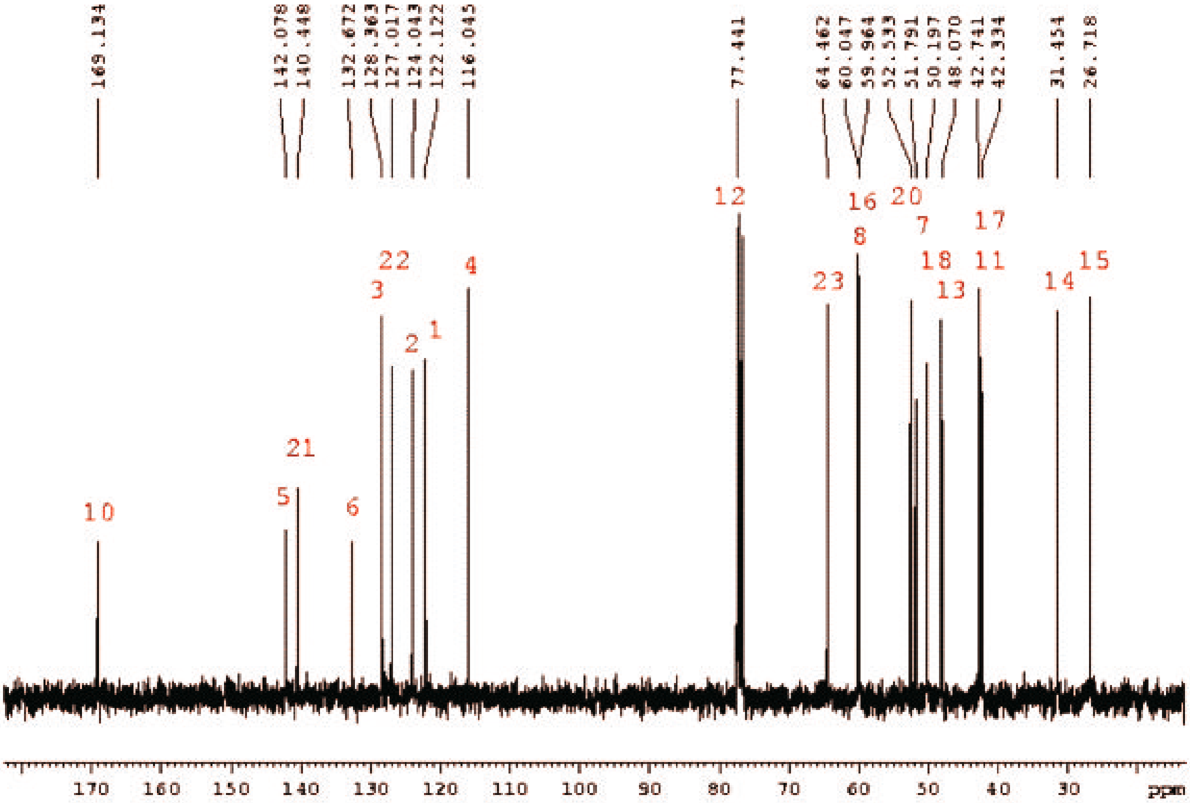
```
...
1 ze
  d11 pl26:f2
2 30m do:f2
  d11 cw:f2
  d1
  p1 ph1
  go=2 ph31
  30m do:f2 mc #0 to 2 F0(zd)
exit
....

;pl26: f2 channel - power level for cw decoupling
```



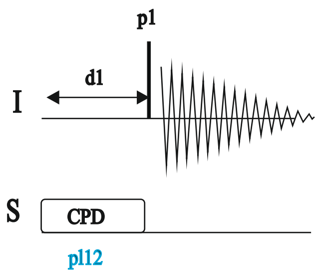




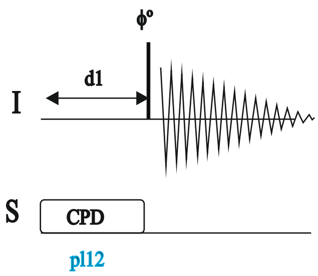


13C NMR spectrum Analysis from Chemical shifts.

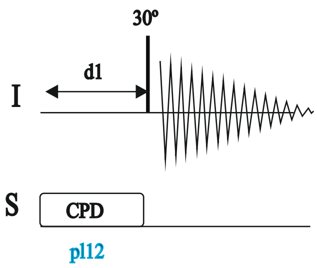
zggd



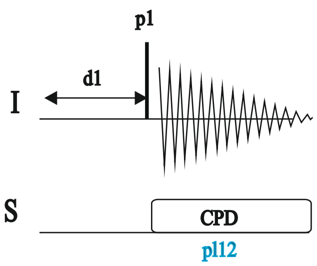
zg0gd



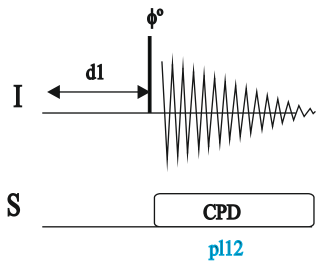
zggd30



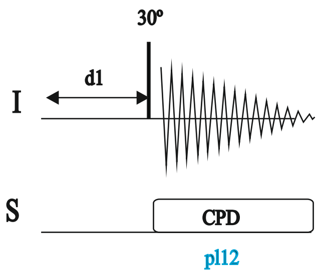
zgf3ig pl16
zgig



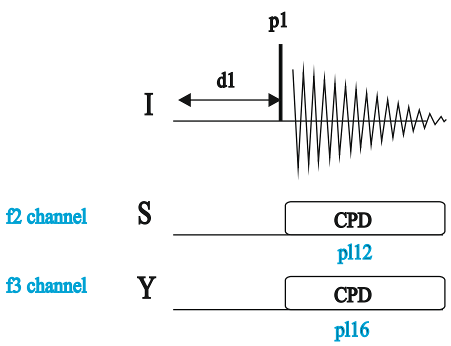
zg0ig



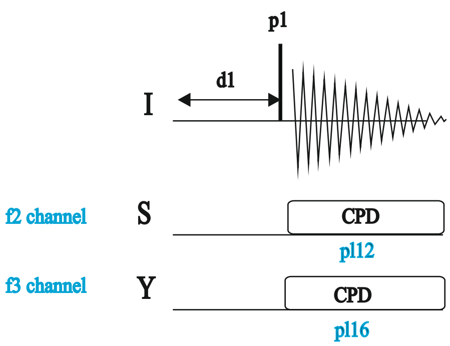
zgig30



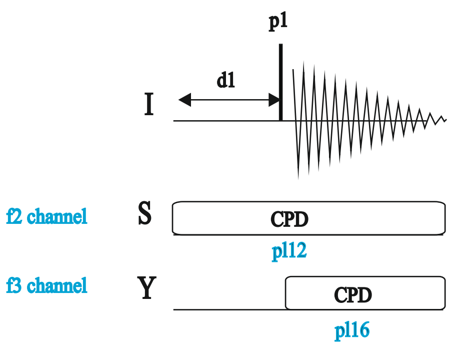
zgfbig



zgigf2igf3



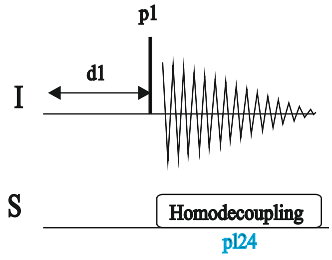
zgdcf2igf3



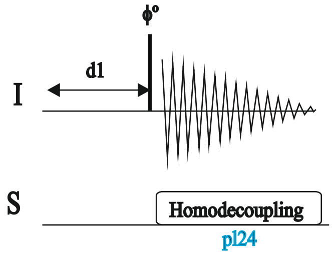
Selective irradiation with homodecoupling (see DIGMOD in eda)

zghd.2
zghd

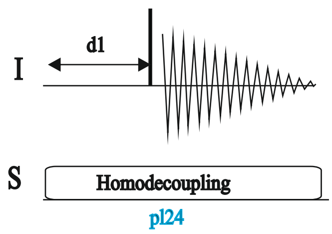
zghc
zghc.2
zghc.3

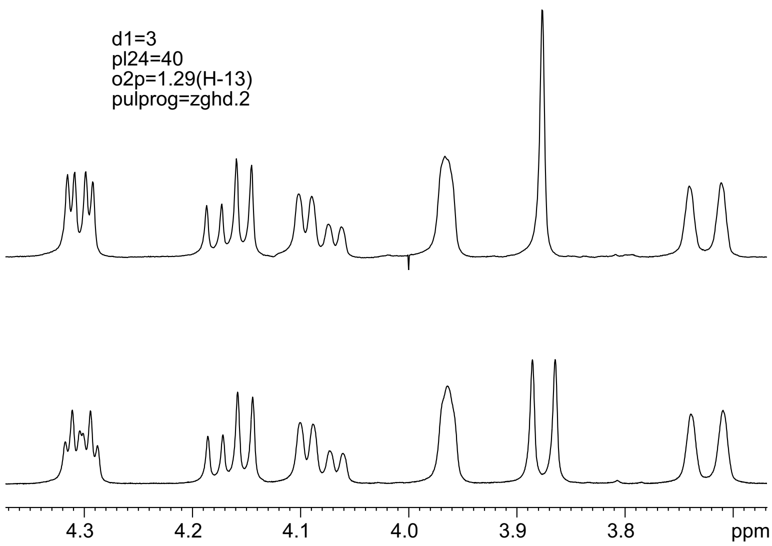


zg0hd



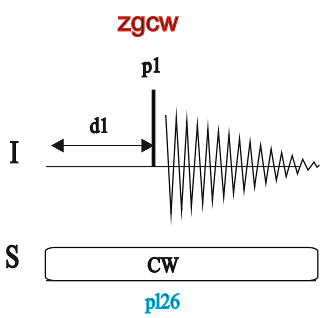
zgadchd

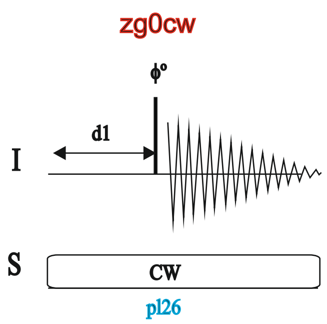


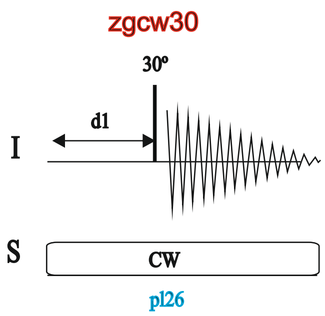


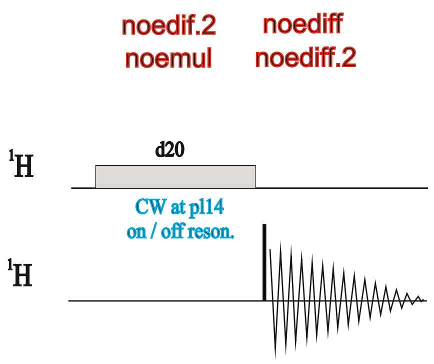
Expansion of the homodecoupling spectrum of strychnine after irradiate at 1.29ppm. Note that J-coupled protons appear simplified.

Selective irradiation with CW





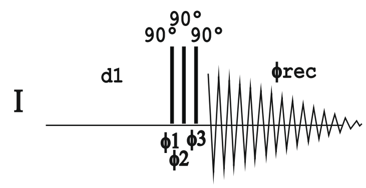




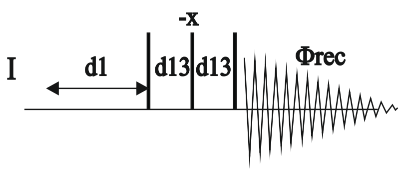
Also see: solvent suppression (zgf2pr)

Miscellaneous Experiments

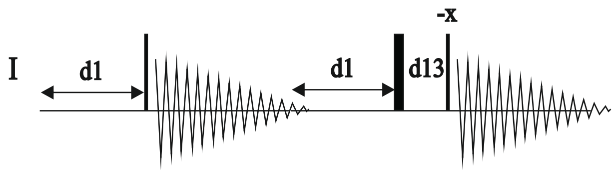
zgbs



aring



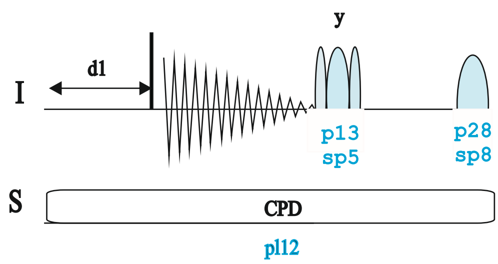
aring2



UDEFT:

M. Piotto, M. Bourdonneau, K. Elbayed, J.-M. Wieruszeski & G. Lippens, Magn. Reson. Chem. 44, 943-947 (2006)

udeft

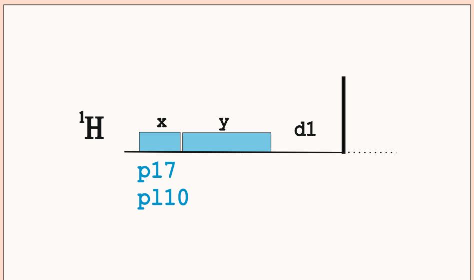


NMR Element: Purge Element Before d1

```
....

  d12 pl10:f1
  p17 ph3
  p17*2 ph4
  d1 pl1:f1

....
```




```
ph3=0
ph4=1

;pl10: f1 channel - power level for TOCSY-spinlock
;p0 : f1 channel -  20 to 90 degree high power pulse
;p1 : f1 channel -  90 degree high power pulse
;p17: f1 channel - trim pulse                               [2.5 msec]
```

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T1 & T2 RELAXATION

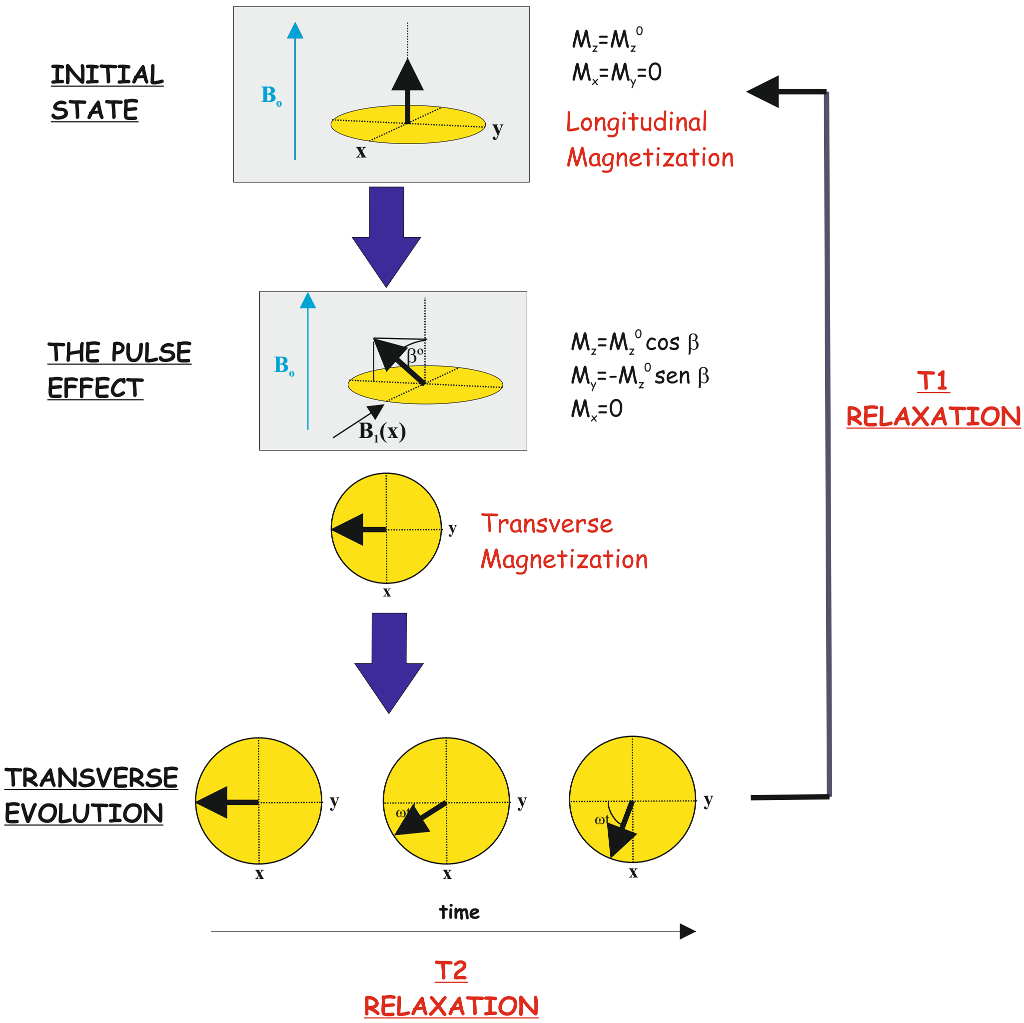
Experiment Description:

T1 and T2 relaxation measurements are usually monitored by means of a series of 1D proton spectra acquired using inversion-recovery or CPMG pulse schemes, respectively.

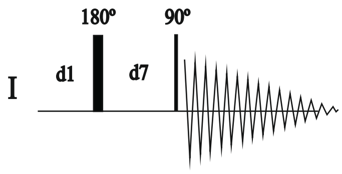
T1 & T2 Relaxation

- ^{1}H T_1 measurements:
  As 1D acquisition (**t1ir1d**)
  As 2D acquisition (**t1ir** || PROTONT1)
- T_1 ^{13}C measurements (**t1irpg**) ^{1}H T_2 measurements
  As 1D acquisition (**cpmg1d**)
  As 2D acquisition (**cpmg**)
  As 1D acquisition with presaturation (**cpmgpr1d**)
  As 1D acquisition with presaturation and purge element (**zgpurge**)

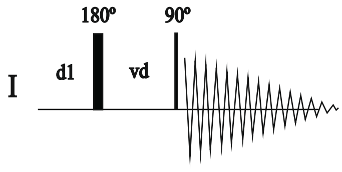
Also see:
2D HSQC and 3D HNCO experiments for Backbone Dynamics in Proteins



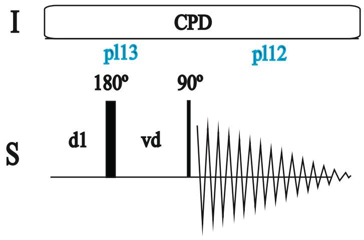
t1ir1d

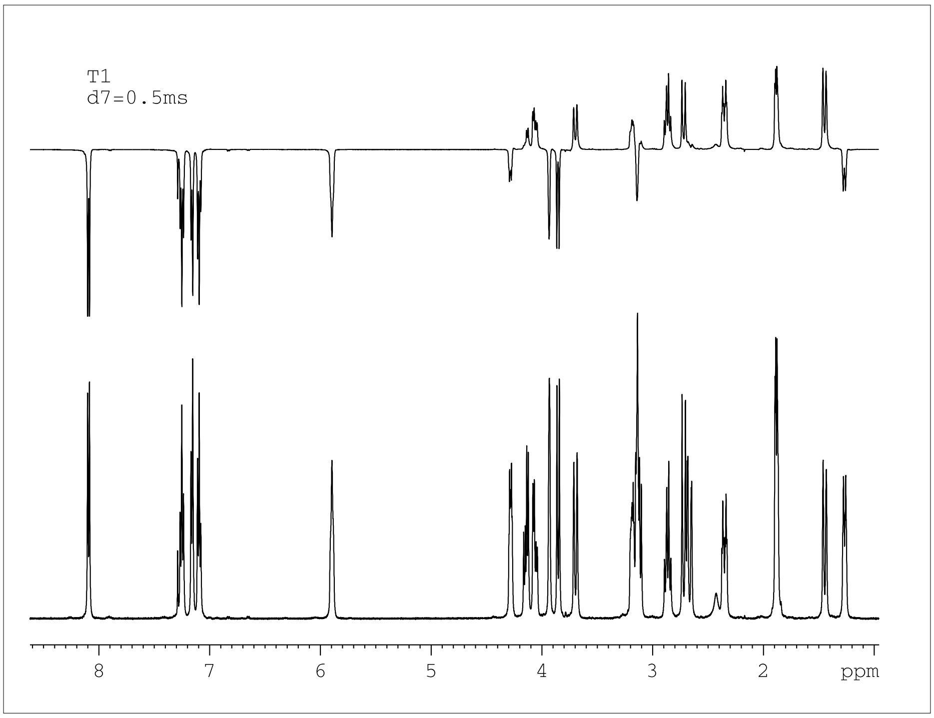


t1ir



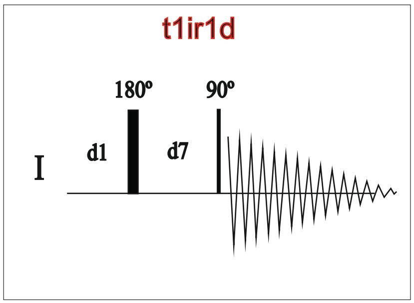
t1irpg





1D Inversion-Recovery spectrum (top) of strychnine acquired with pulprog=t1ir1d (d7=0.5ms) showing the faster relaxation of methylenic CH2 protons (resonances up) with respect to CH ones (resonances down)

T1 measurements:
The inversion-recovery experiment

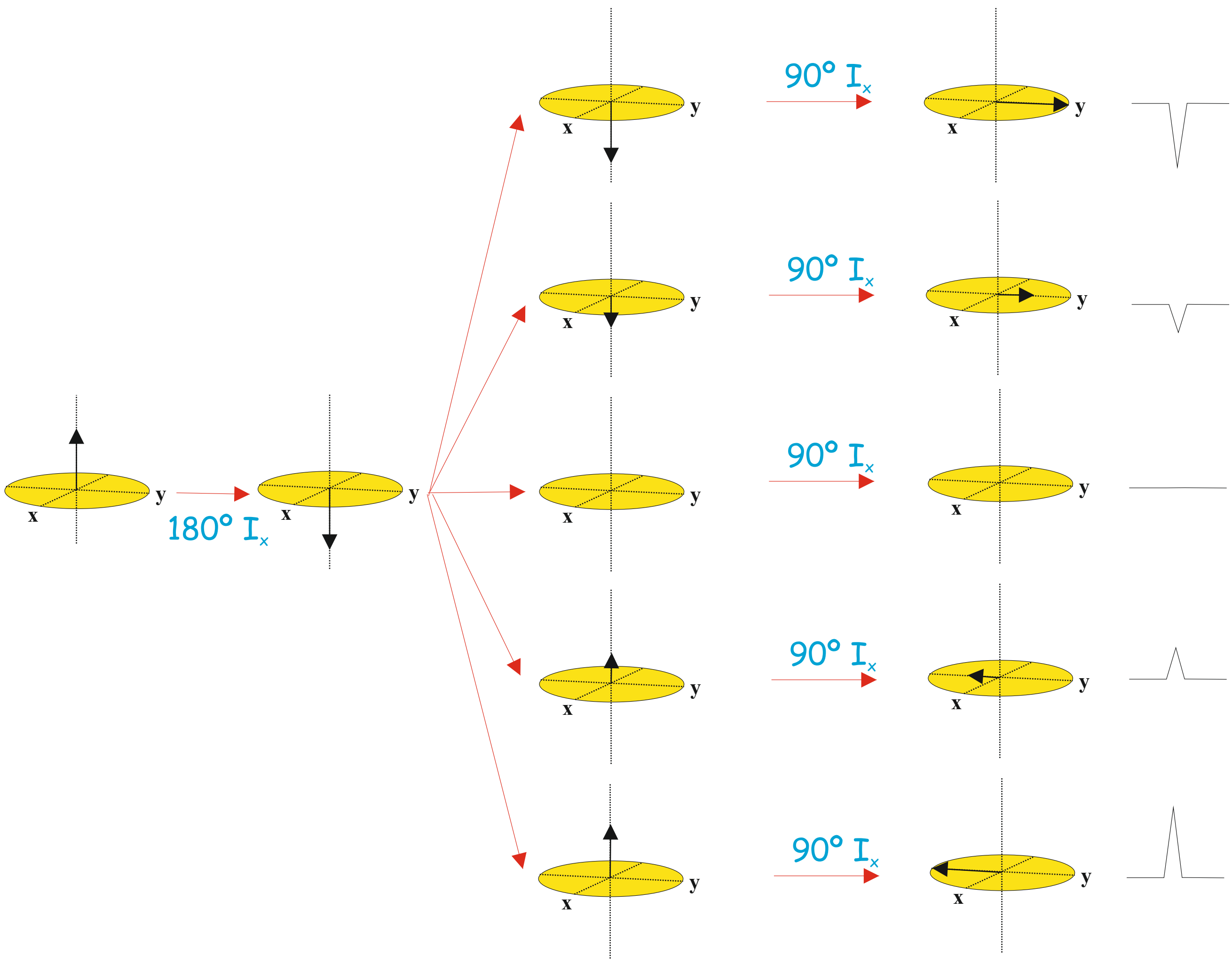


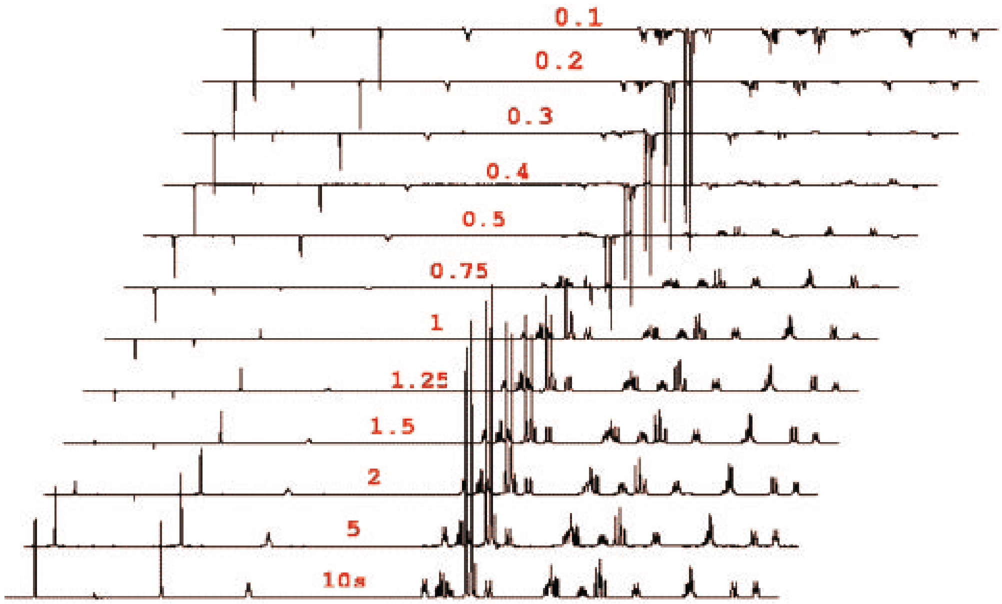
$$dM_z/dt = R_1(M_{eq} - M_z) = (M_{eq} - M_z)/T_1$$

$R_1 = 1/T_1$ Hz is the longitudinal relaxational rate constant

T_1 (in seconds) is the longitudinal relaxation time

$$I_z \xrightarrow{180°\ I_x} -I_z \xrightarrow{d7} -I_z \exp(-d7/T_1) \xrightarrow{90°\ I_x} I_y \exp(-d7/T_1)$$



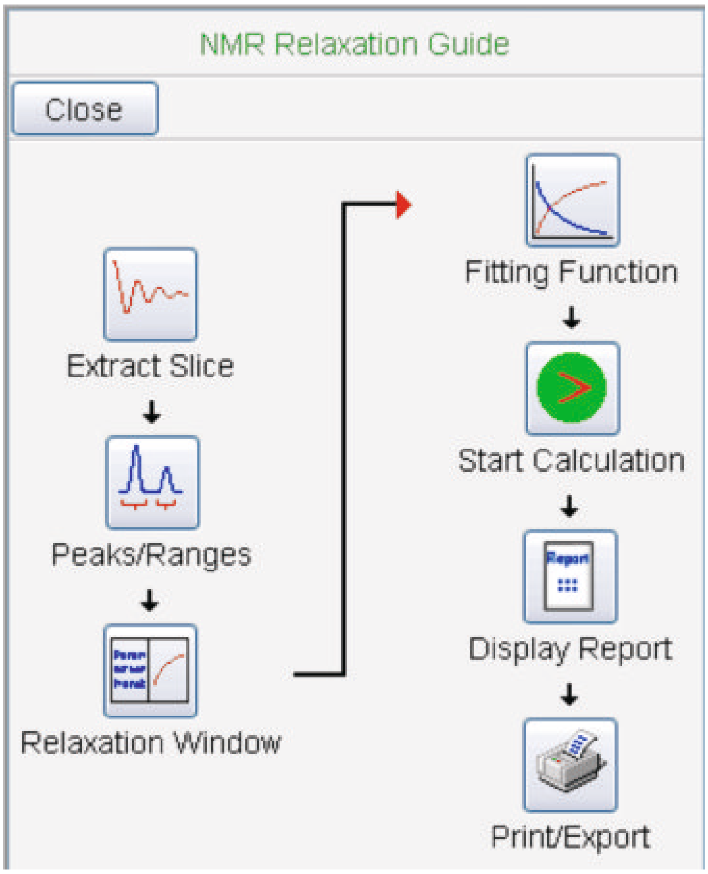


A series of 1D Inversion-Recovery spectra of brucine automatically acquired with pulprog=t1ir and with a predefined delay list. 1D spectra can be automatically processed and then analyzed with the relaxation T1/T2 tool (**t1guide**) included into TOPSPIN. Otherwhise, a simplified calculation can be done by detecting the so-calling null point.

$$I_z=I_0(1-2\exp(-d7/T_1)$$

$$\ln(I_0-I_z)=\ln(2I_0)-d7/T_1$$

$$t_{null}=T_1*\ln 2$$



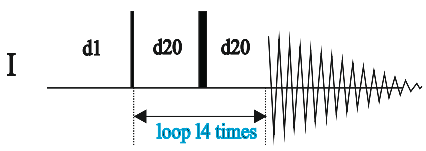
$$dM_{xy}/dt = -R_2M_{xy} = -M_{xy}/T_2$$

$R_2 = 1/T_2$ Hz is the transverse relaxation rate constant

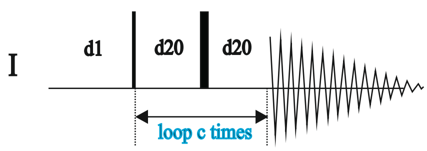
T_2 (in seconds) is the spin-spin trasnverse relaxation time

<u>**T2 filter using CPMG (Carr-Purcell-Meiboom-Gill) sequence**</u>

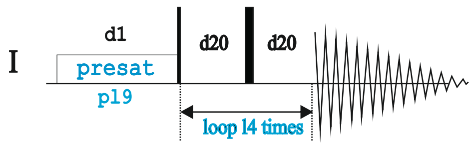
cpmg1d



cpmg



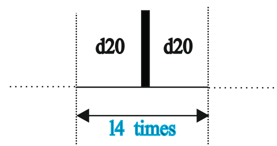
cpmgpr1d



NMR Building Block: A T2 Filter.

```
....
3 d20
  p2 ph1
  d20
  lo to 3 times l4
....

;d20: fixed echo time to allow elimination of J-mod. effects
;        d20 should be << 1/J ,but > (50 * P2)         [1-2 msec]
;l4: loop for T2 filter                                [4 - 20]
```

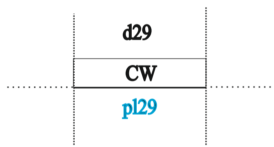


NMR Building Block: A T1(rho) Filter.

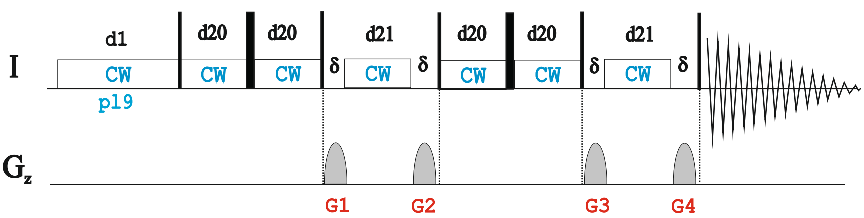
```
"p29=d29"

.........
4u pl29:f1
   (p29 ph1)
...........

;p29: f1 channel - trim pulse
;d29: spinlock time                          [10 - 50 msec]
```



zgpurge



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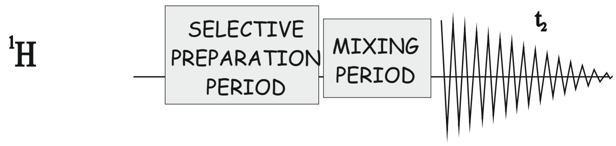
SELECTIVE 1D EXPERIMENTS

Experiment Description

For small molecules, it is usually only necessary to obtain specific information. For this reason, it is not necessary to record a time-consuming 2D dataset. A general approach is to convert the 2D experiment to a 1D counterpart by removing the variable t1 evolution period and introducing some selective excitation element.
Selective excitation can be achieved by a selective 90 pulse (selzg) or by a selective gradient-based echo block that uses a selective 180 pulse (selgpse).



Sample Requirements

Selective experiments can be recorded on any type of sample.The only requirement is a well isolated resonance.

Hardware Requirements

In principle, selective experiments can be recorded on any probehead and on any modern spectrometer. An optional pulsed-field gradient coil (highly recommended) is required for gradient-based versions.

Set-up

For classical selective phase-cycled experiments:
- **p11** is the duration of the selective 90 pulse (in microseconds)
- **sp1** is the power level
- **spnam1** is the pulse shape (for instance, Gauss1.001)
- **spoffs1** is the offset (in Hz) to irradiate with respect to the central o1 frequency.

For gradient-based selective experiments:
- **p12** is the duration of the selective 180 pulse (in microseconds)
- **sp2** is the power level
- **spnam2** is the pulse shape (for instance, Gauss1.001)
- **spoffs2** is the offset (in Hz) to irradiate with respect to the central o1 frequency.

The result is a 1D spectrum equivalent to the specific row of the 2D analog. The advantages are: i) Acquisition is faster; ii) The resulting 1D spectrum offer better high-resolution properties than 2D, and iii) The experiment can be individually optimized.

Selective Excitation & 1D Selective Homonuclear experiments

- **Phase-Cycled:**

  Using a shaped 90° pulse (**selzg** | SELZG1H)

  Selective 1D COSY experiment (**selco** | SELCO1H)
  Selective 1D RELAY experiment (**selcorl**)
  Selective 1D TOCSY experiment (**selmlzf** | SELMLZF1H)
  Selective 1D NOESY experiment
  Using d8 random variation (**selno** | SELNO1H)
  Using d8 and z-filter (**selnozf**)
  Selective 1D ROESY experiment (**selro** | SELRO1H)

  DANTE Excitation

  2-2-6-2-2 DANTE-z scheme (**dazzg**)
  3-6-3 DANTE-z scheme (**daz363zg**)
  1-1 DANTE-z scheme (**daz11zg**)

- **Gradient-based using SPFGE:**

  Using selective pulsed-field-gradient spin-echo or SPFGE (**selgpse** | SELGPSE)

  Selective ge-1D COSY experiment (**selcogp** | SELCPGO)
  Selective ge-1D TOCSY experiment:
  using MLEV (**selmlgp** | SELMLGP)
  using MLEV with ZQ suppression (**selmlgp.2**)
  using DIPSI-2 (**seldigp**)
  using DIPSI-2 with ZQ suppression (**seldigp.2**)
  Selective ge-1D NOESY experiment (**selnogp** | SELNOGP)
  with ZQ suppression (**selnogpzs**)
  Selective ge-1D ROESY experiment (**selrogp** | SELROGP)
  Selective ge-1D T-ROESY experiment (**selrogp.2**)

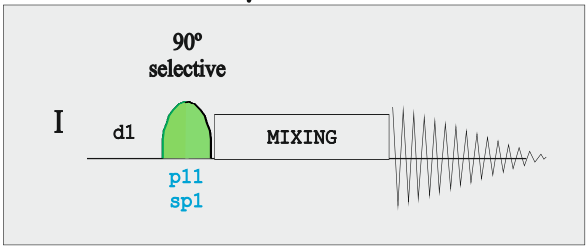
- **Gradient-based using Chemical Shift Selective Filter (CSSF):**

  Using selective echo (**selcssf**)
  Using selective echo and ZQ suppression (**selcssfzs**)
  Using double selective echo (**selcssf.2**)
  Using double selective echo and ZQ suppression (**selcssfzs.2**)

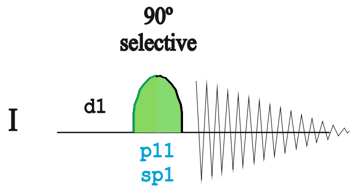
  Selective ge-1D TOCSY experiment:
  using DIPSI-2, single echo CSSF and ZQ suppression (**selcssfdizs**)
  using DIPSI-2, double echo CSSF and ZQ suppression (**selcssfdizs.2**)

  Selective ge-1D NOESY experiment
  using single echo CSSF and ZQ suppression (**selcssfnozs**)
  using double echo CSSF and ZQ suppression (**selcssfnozs.2**)

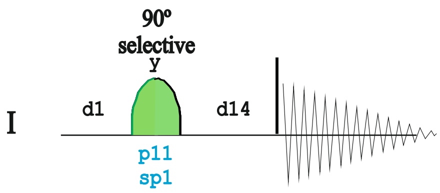
General Scheme
Phase-cycled Selective



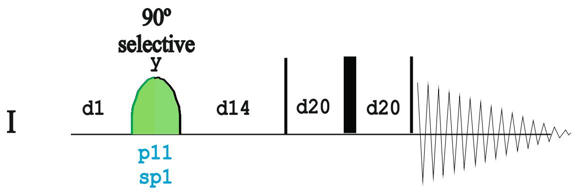
selzg



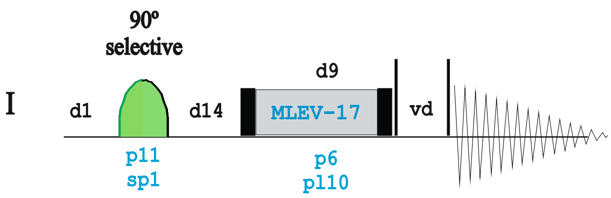
selco



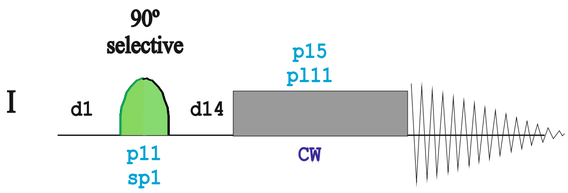
selcorl



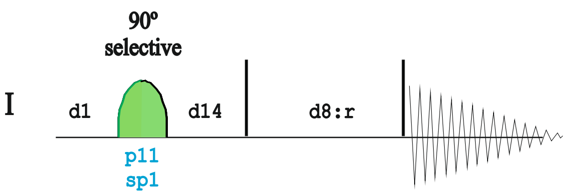
selmlzf



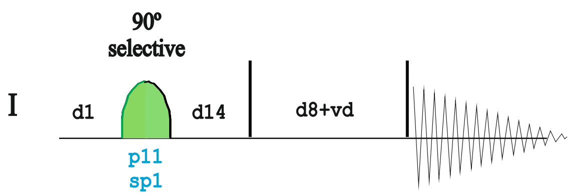
selro



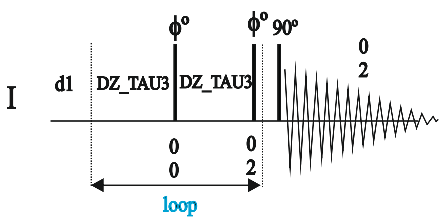
selno



selnozf

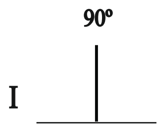


dazzg
daz11zg
daz363zg

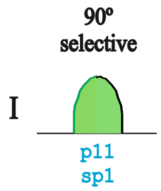


NMR Building Block:
Rectangular (Hard) vs Shaped (selective) Pulse

```
....
  4u pl1:f1
  p1 ph1
....
```



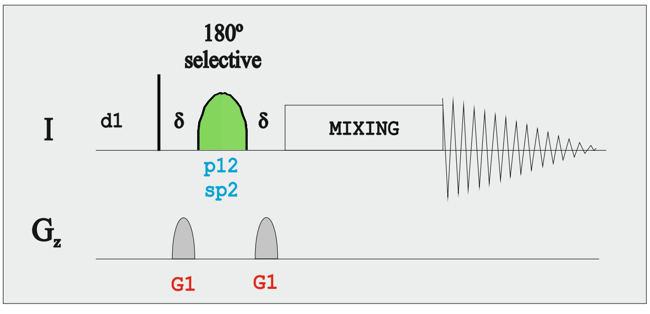

```
....
  4u pl0:f1
  p11:sp1:f1 ph1:r
....
```



NMR Building Block:
SPFGE=Gradient-180sel-Gradient

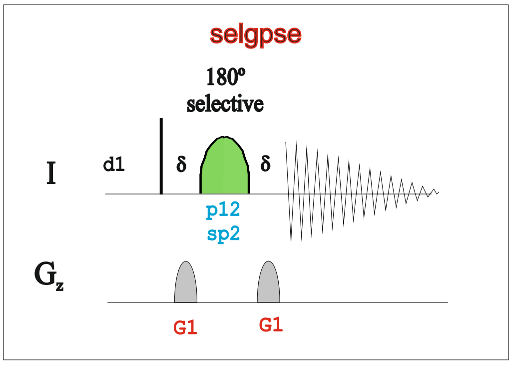
```
 50u UNBLKGRAD
....
 p16:gp1
 d16 pl0:f1
 p12:sp2:f1 ph2:r
 p16:gp1
 d16 BLKGRAD
...
```

General Scheme
Geadient-Based SPFGE

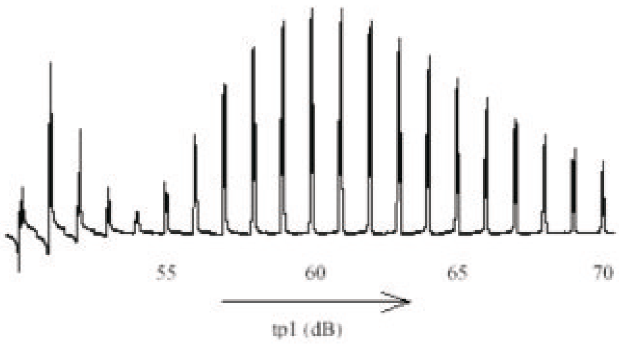


Common Parameters of Mixing Processes:
p15 is the mixing time in ROESY experiments
d8 is the mixing time in NOESY experiments
d9 is the mixing time in TOCSY experiments
d4 is the evolution time in COSY experiments

NMR Experiment: Calibration selective 180° shaped pulse
Parameter Set: SELGPSE
Pulse PRogram: selgpse



1. Record a 1H spectrum
2. Define "**pulprog selgpse**"
3. Set o1p on the selected resonance
4. Set p12=20m
5. Set spnam2=Gauss1.1000
6. Run **paropt** varying **sp2** starting from 70 dB and with an increment of -2dB
7. The first maximum is the selective 180° pulse of 20ms of duration

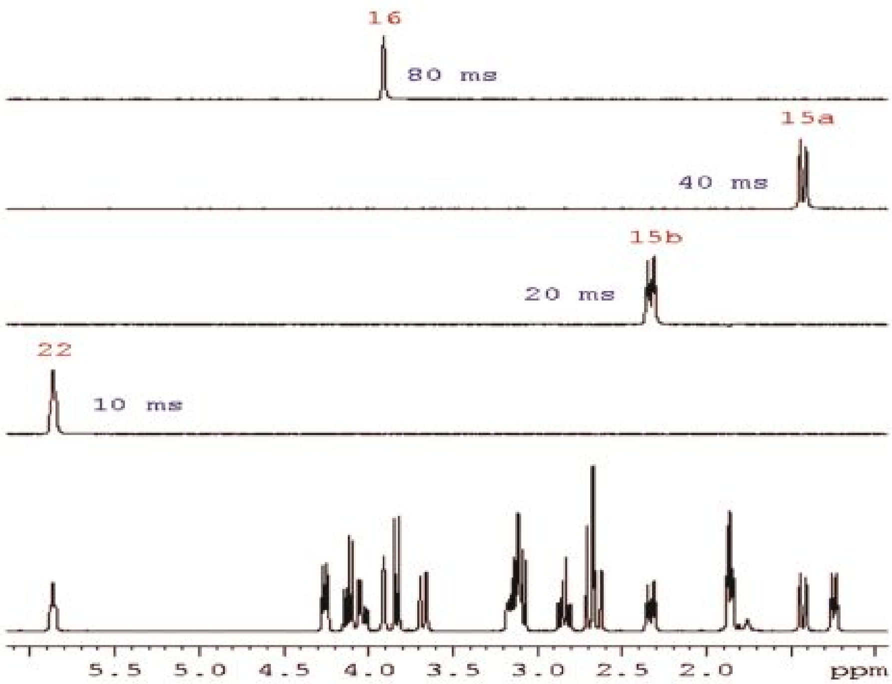


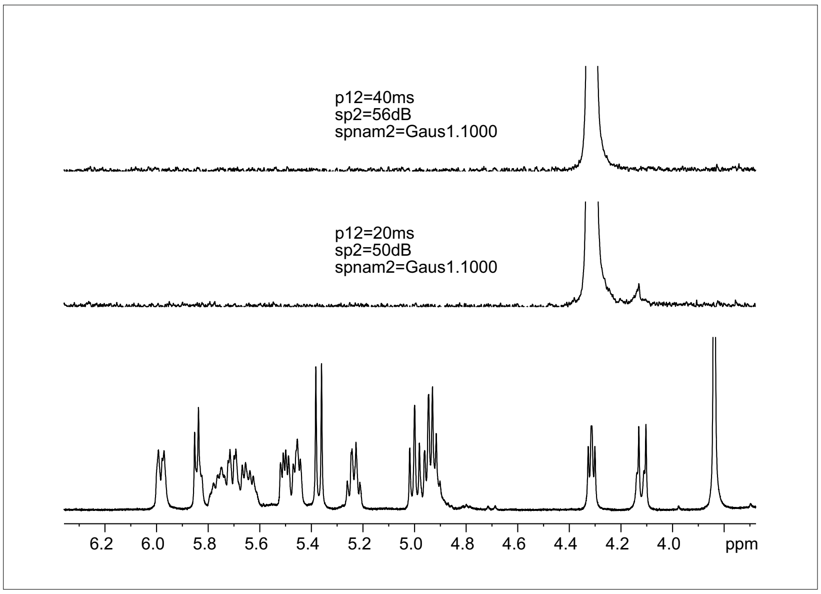
Remember Linear Amplifiers:
Selectivity: p12*2 and sp2+6dB

If the selective 180 pulse of 20 ms of duration needs 60 dB:
- The selective 90 pulse of 10 ms is 60 dB
- The selective 180 pulse of 10 ms is 54dB
- The selective 180 pulse of 40 ms is 66 dB

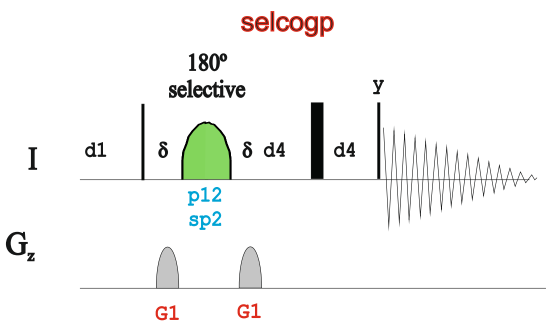
Selectivity in selective experiments:

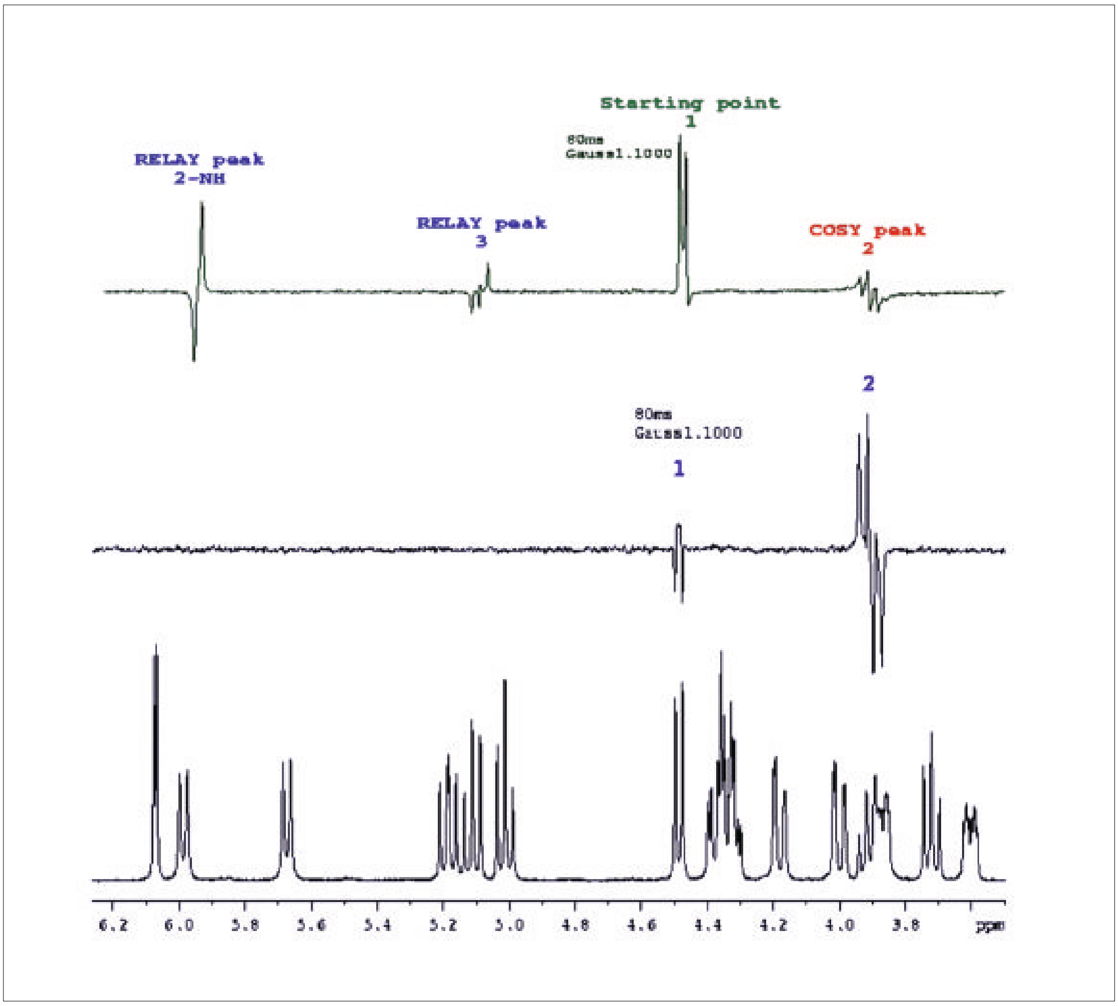
Depends how isolated is the selected resonance. As a good starting conditions. A selective 180 pulse (spnam2=Gauss1.1000) of 20000 microseconds (p12) affords an effective field around 60Hz. More selectivity is achieved by using longer pulses, at expense of the presence of unwanted relaxation effects.



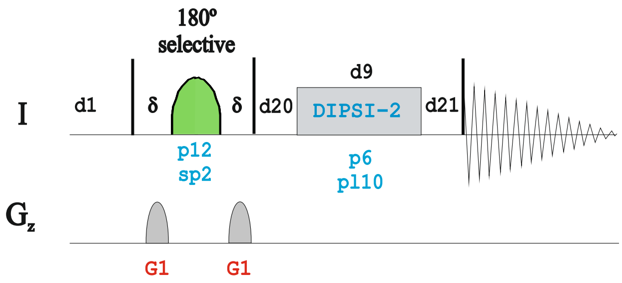


Use the shape tool (**stdisp**) to generate new shapes and to calculate a different number of parameters and features related to them.

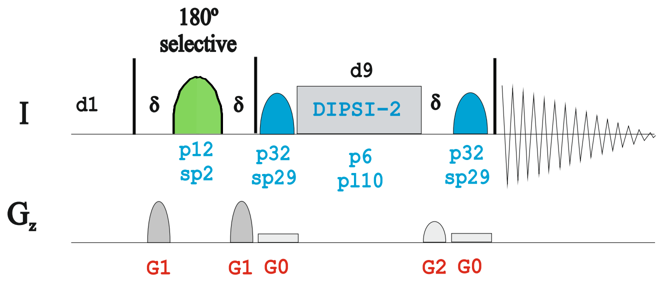

selcogp
180°
selective
y
I
d1
δ
δ
d4
d4
p12
sp2
G_z
G1
G1


RELAY peak
2-NH
RELAY peak
3
Starting point
1
80ms
Gauss1.1000
COSY peak
2
2
80ms
Gauss1.1000
1
6.2
6.0
5.8
5.6
5.4
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
ppm

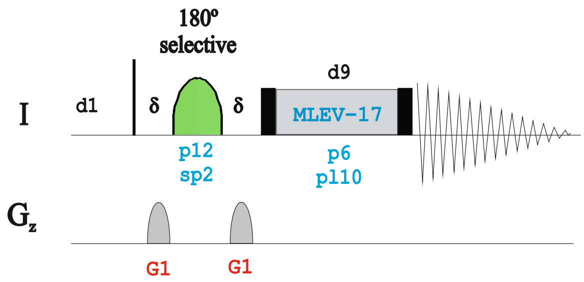
seldigp



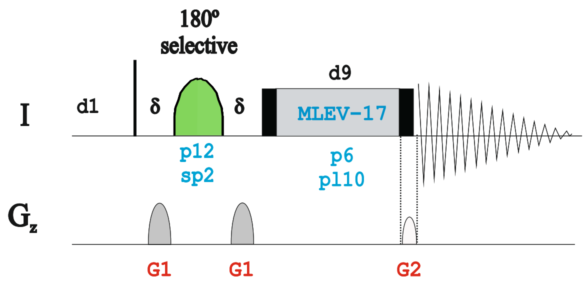
seldigpzs

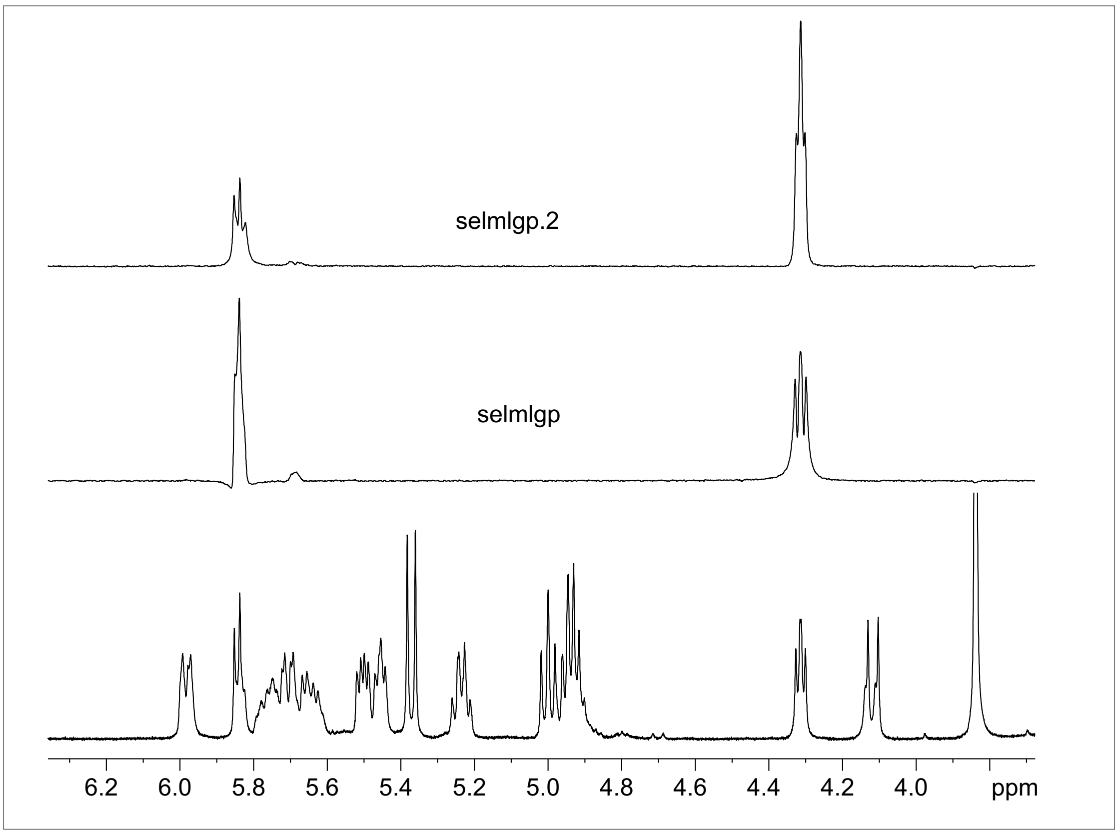


selmlgp



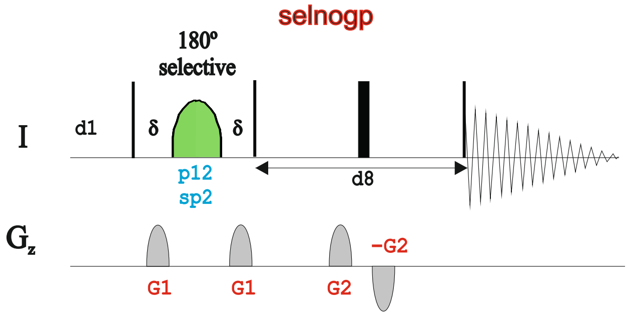
selmlgp.2



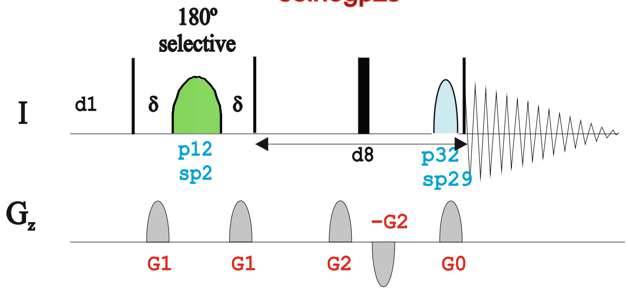


Suppression of ZQC in selective 1D TOCSY experiments:

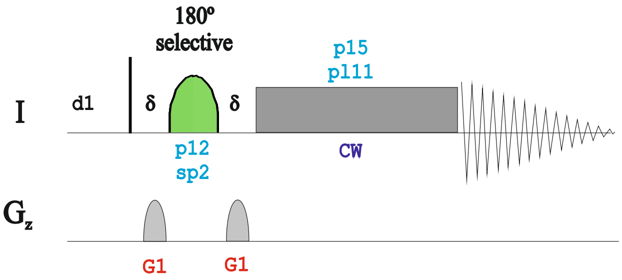
selnogp



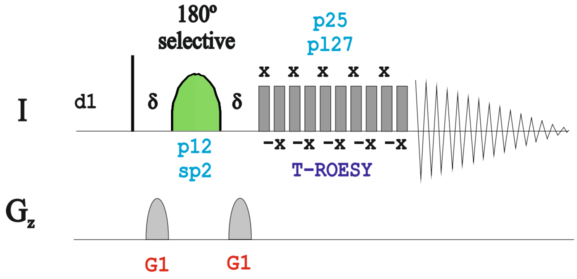
selnogpzs



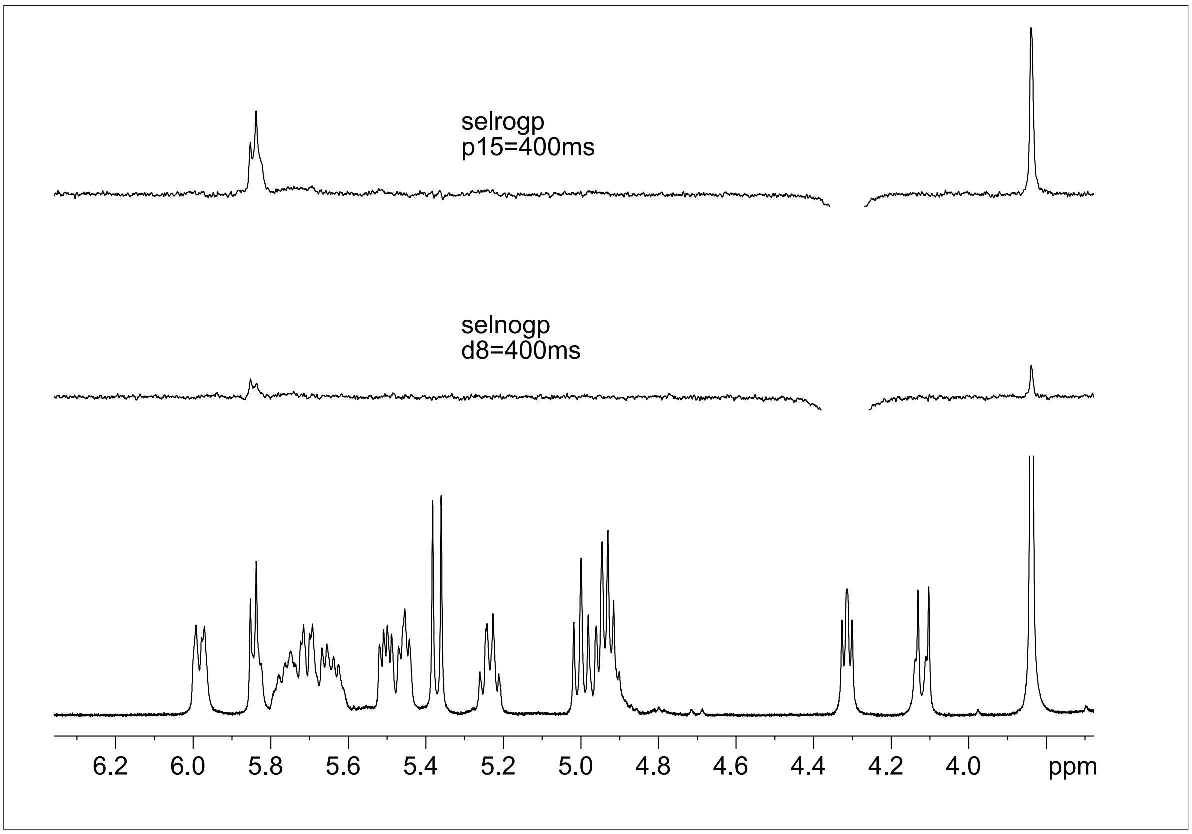
selrogp



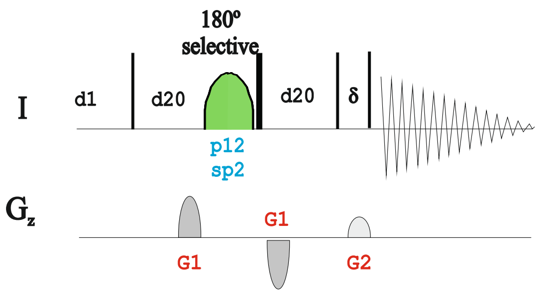
selrogp.2



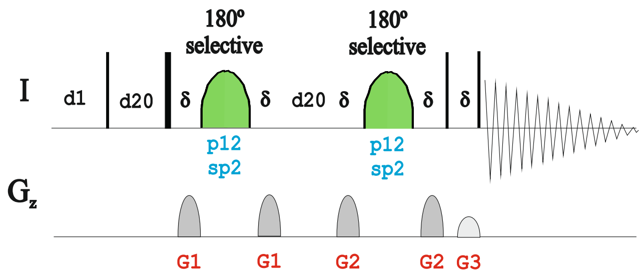
Selective ROESY vs NOESY in medium-sized molecules in a 500MHz



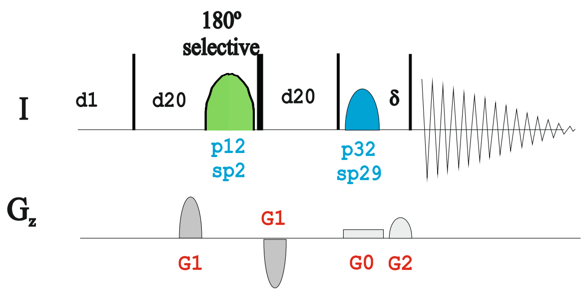
selcssf



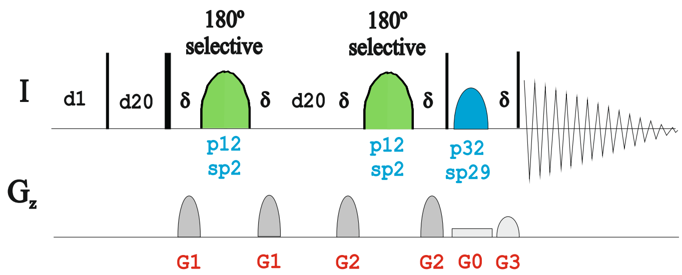
selcssf.2



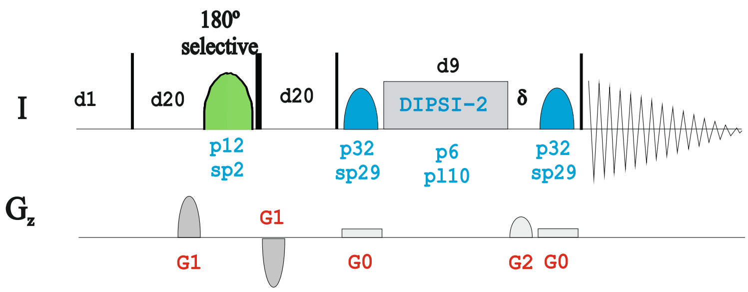
selcssfzs



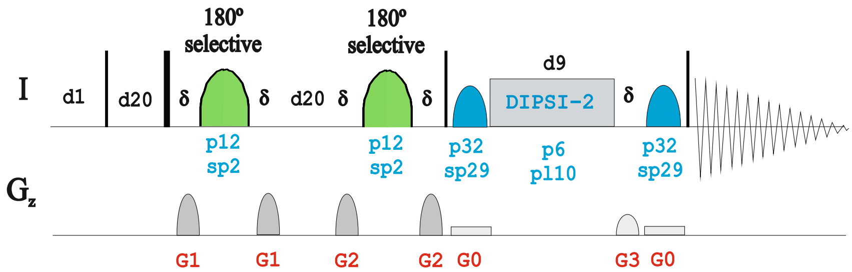
selcssfzs.2



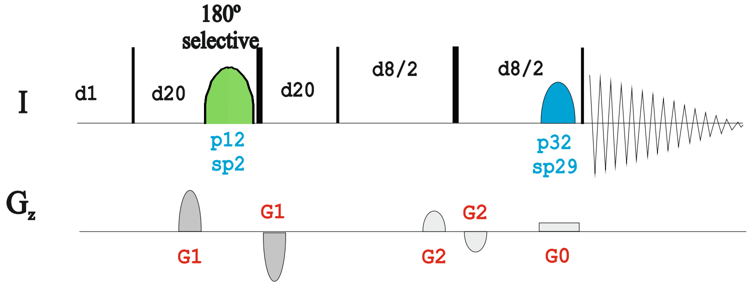
selcssfdizs



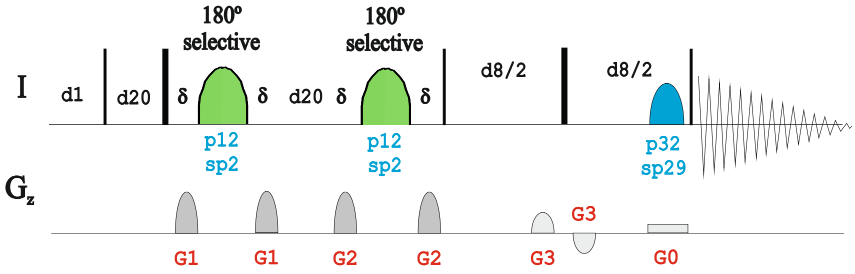
selcssfdizs.2



selcssfnozs



selcssfnozs.2



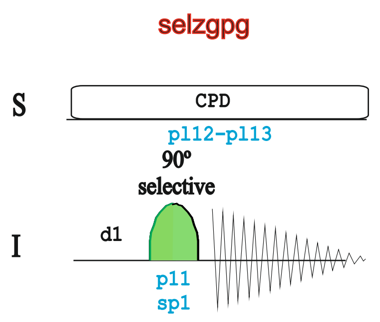
Selective Excitation & 1D Selective Heteronuclear experiments

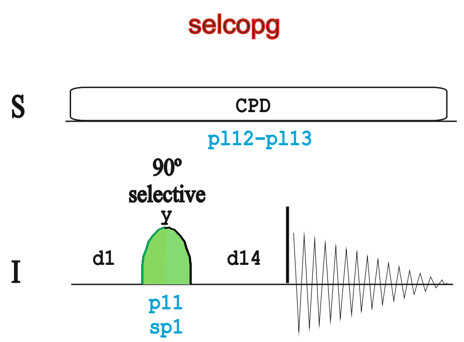
^{13}C Selective excitation using a shaped 90° pulse (**selzgpg**)

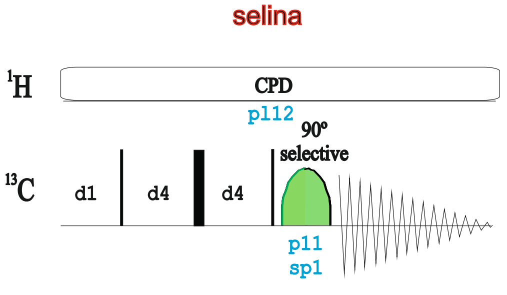
Selective 1D X-X COSY experiment (**selcopg**)
Selective 1D INADEQUATE experiment (**selina**)

Selective ge-1D HSQC experiment (**selhsqcgpsp**)
Selective ge-1D HSQC experiment with PEP (**selhsqcgpsisp**)
Selective ge-1D Long-range HSQC experiment (HSQMBC) (**selhsqcgplrndsp**)
Selective ge-1D HSQC-NOESY experiment (**selhsqcgpnosp**)
Selective ge-1D HSQC-NOESY experiment without decoupling (**selhsqcgpndnosp**)

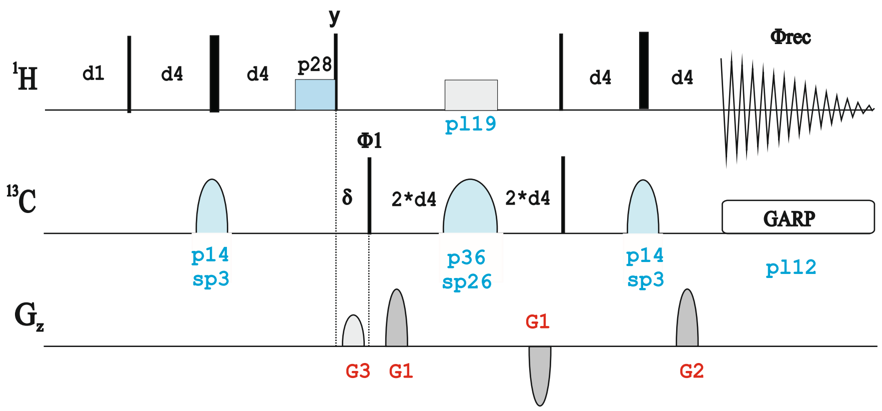
Selective 1D INEPT (INAPT) experiment (**selineptlrrdsp**)



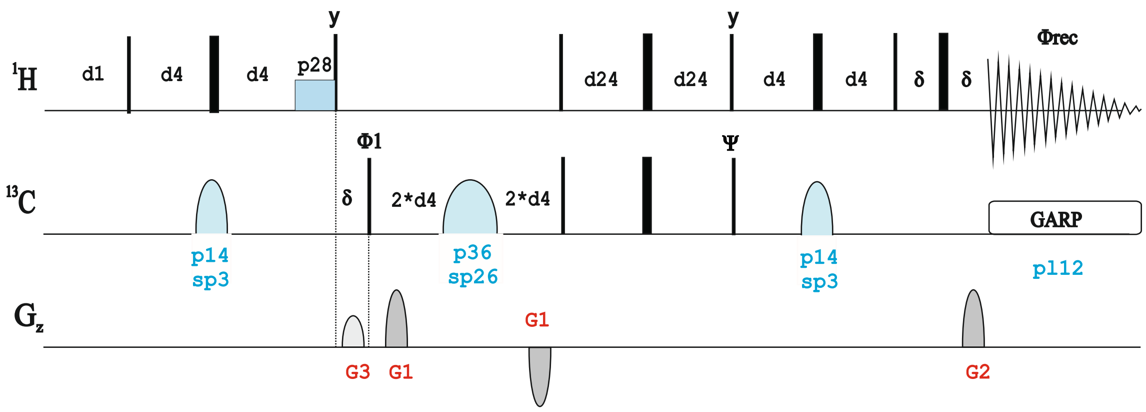




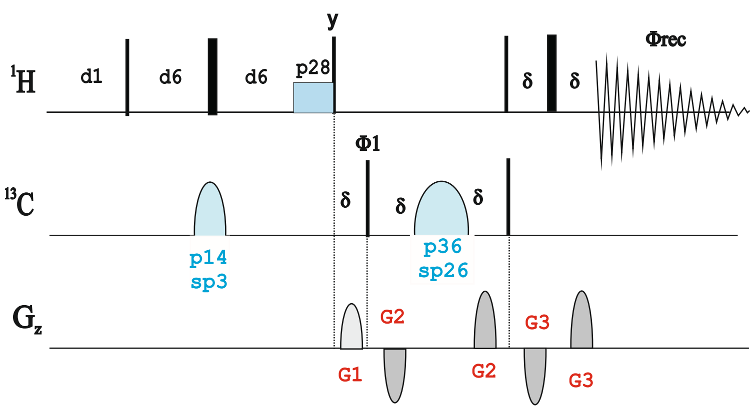
selhsqcgpsp

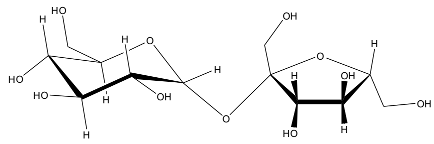


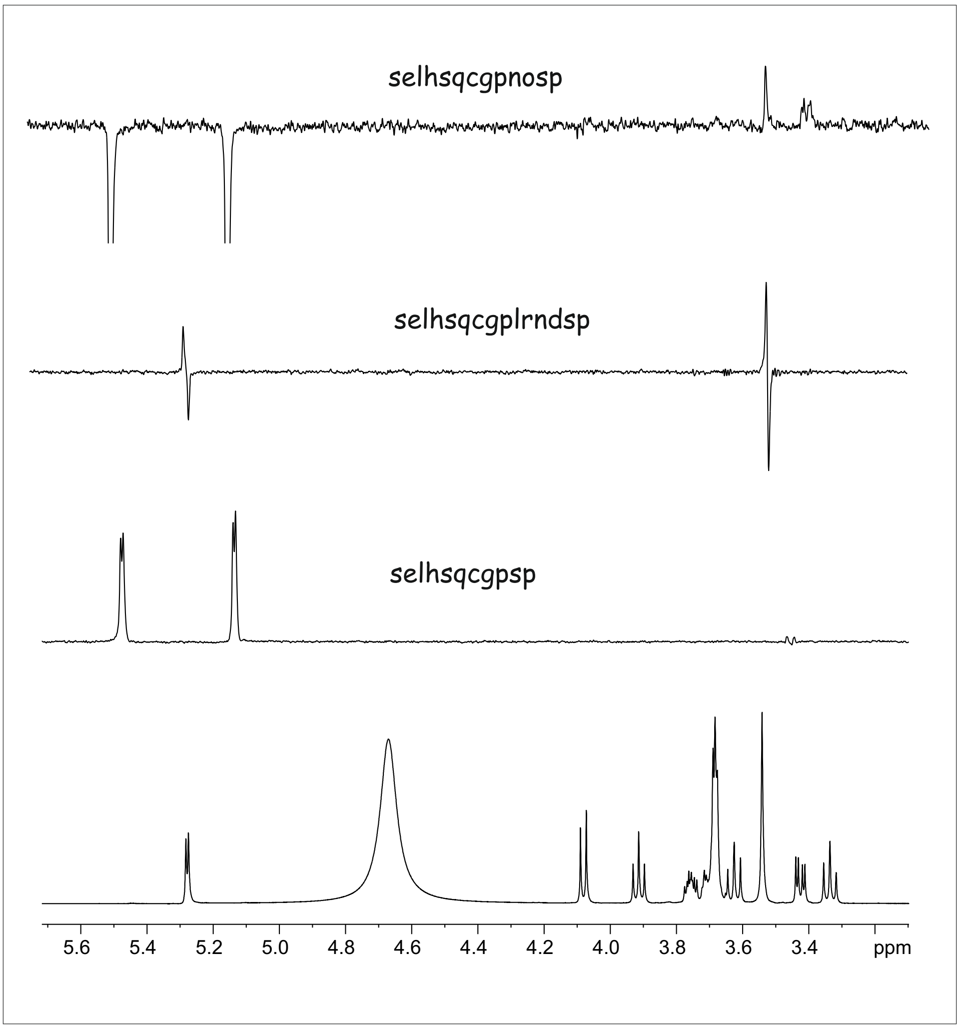
selhsqcgpsisp



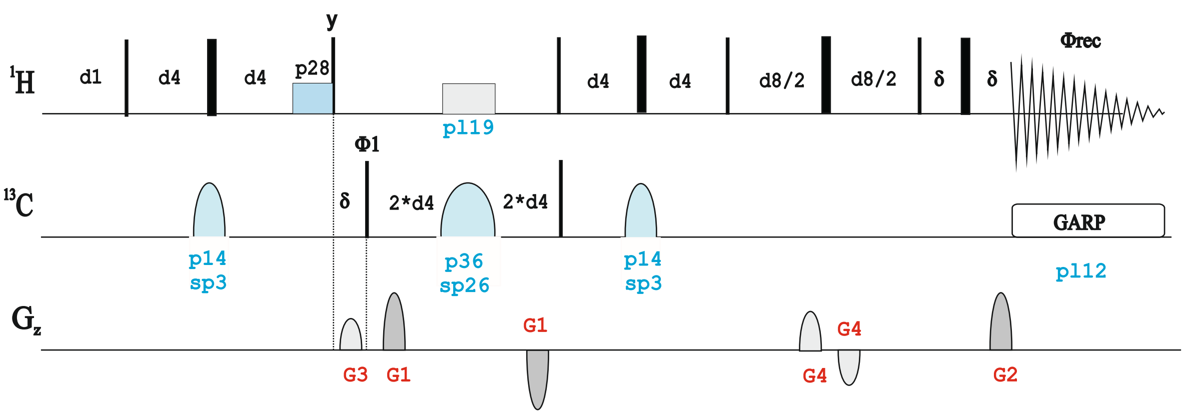
selhsqcgplrndsp



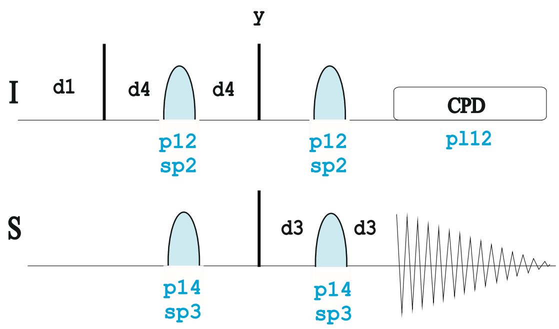


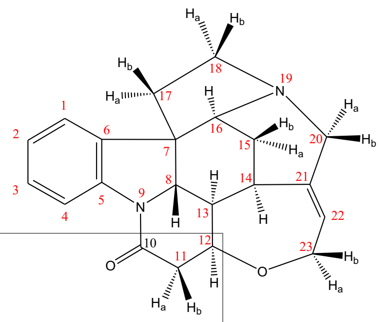


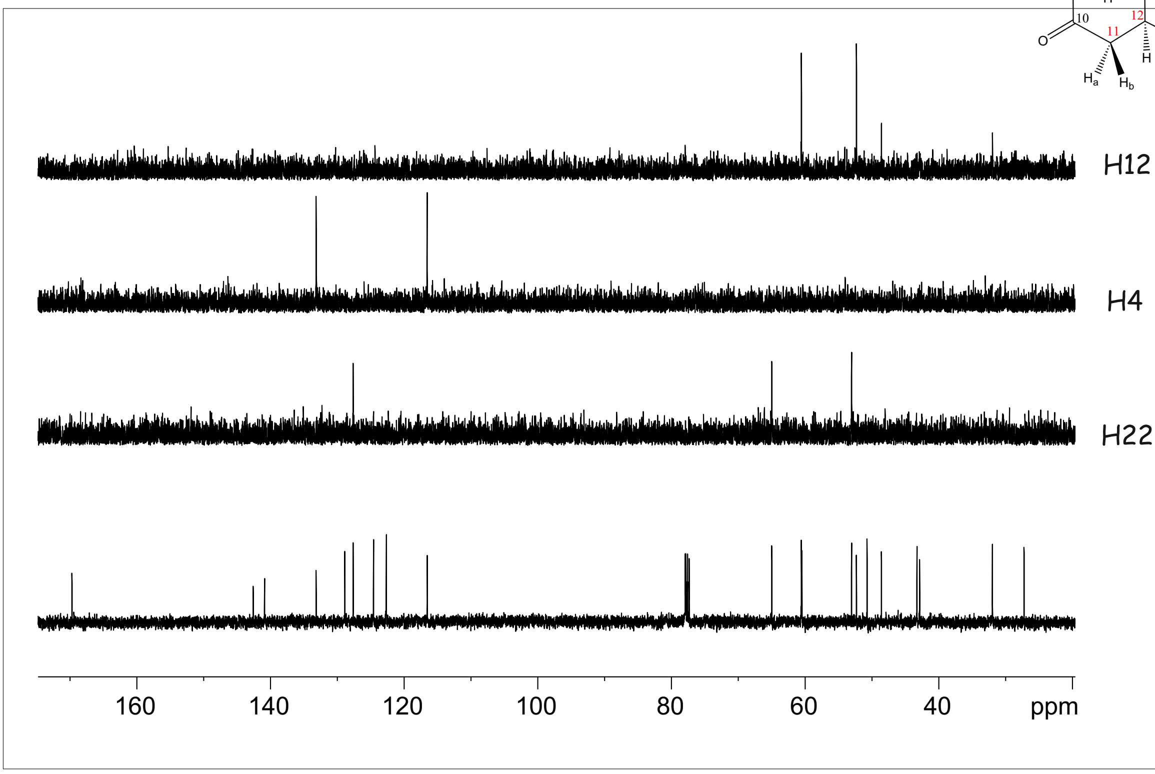
selhsqcgpnosp



selineptlrrdsp







A complete Description of creating, analyzing and Manipulating RF and Gradient Shapes in the **Shape Tool Manual**
(see **Help** menu in Topspin)

BRUKER
PULSE PROGRAM
CATALOGUE

NMRGuide

SOLVENT SUPPRESSION

1D Solvent suppression

- **Classical:**

1D solvent presaturation:

Conventional (**zgpr** / **zg0pr** | ZGPR)
Using composite pulses (**zgcppr** | ZGCPPR)
Using spoil gradient (**zggppr**)
Using composite pulse and spoil gradient (**zgcpgppr**)
From f2 channel (**zgf2pr** / **zg0f2pr**)
Using shaped pulse for off-resonance presaturation (**zgps**)

Jump and return:

1-1 scheme (**p11**)
1-3-3-1 scheme (**p1331**)

- **Gradient-based:**

1D WATERGATE:

Using 3-9-19 scheme (**p3919gp** | P3919GP)
Using 3-9-19 and flip back pulse (**p3919fpgp**)
Using 90° water-selective pulses (**zggpwg** | ZGGPWG)

1D Excitation Sculpting:

Using 180° water-selective pulses (**zgesgp** | ZGESGP)
Using 180° water-selective and flip back pulse (**zgesfpgp**)
Using W5 pulse train (**zggpw5**)

1D WET Scheme:

Conventional (**wet**)
With ^{13}C decoupling on f2 during WET and AQ (**wetdc** | LC1DWTDC)
With ^{13}C decoupling on f2 during WET (**wetdw**)
With shape pulse and C-13 decoupling on f2 during WET and acquisition for LC isocratic runs (**lc2wetdc**)
With shape pulse and C-13 decoupling on f2 during WET and AQ with intermediate preparation scan into second dataset for LC gradient runs with updated shapes (**lc2wetdcus** | LC2DWTUS) With shape pulse and C-13 decoupling on f2 during WET and acquisition with intermediate preparation scan into second dataset for LC gradient runs with updated shapes (**lc2grdonflow**)

Related Experiments:

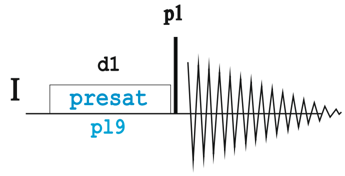
- All these 1D experiments can be incorporated in any multidimensional NMR experiment. Please refer to each chapter to check the different possibilities for 2D and 3D solvent-suppressed experiments
- LC-NMR Experiments

NMR Building Block: Solvent Presaturation

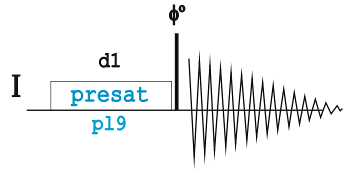
```
   ...
   d12 pl9:f1
   d1 cw:f1 ph29
   d13 do:f1
   d12 pl1:f1
   ...

ph29=0
.....
;pl9 : f1 channel - power
; level for presaturation
```

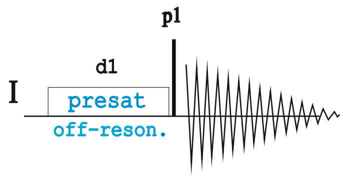
zgpr



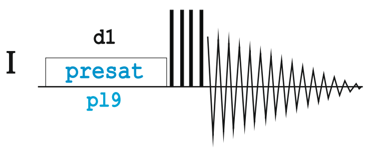
zg0pr



zgps

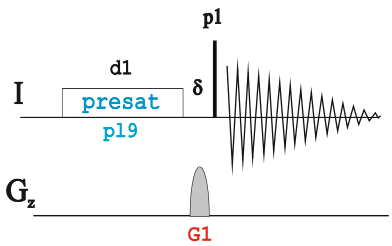


zgcppr

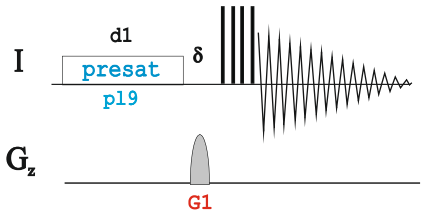


Also see: LC-NMR experiments

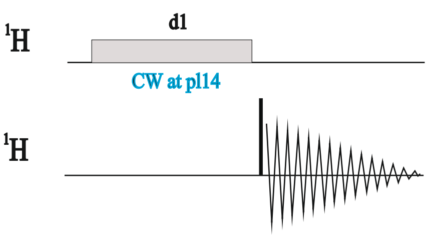
zggppr



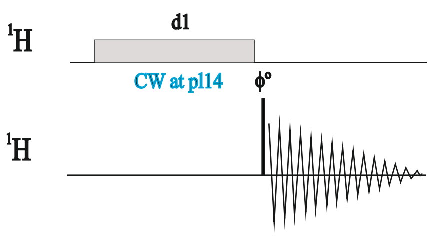
zgcpgppr



zgf2pr

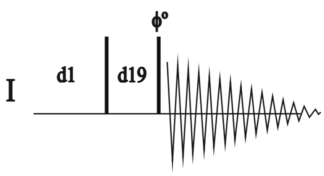


zg0f2pr

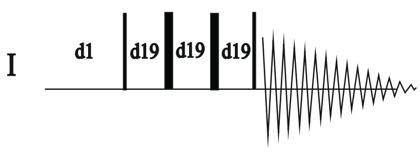


Also see: noediff

p11

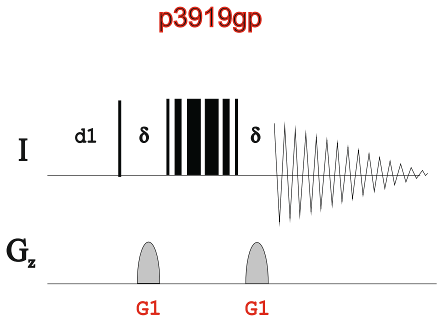


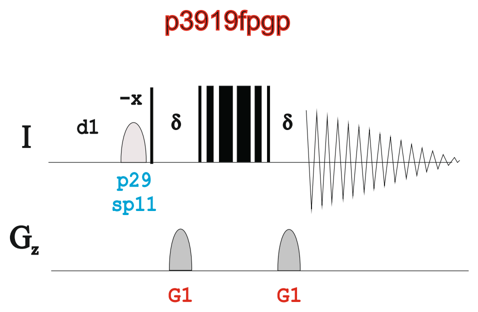
p1331



WATERGATE:

1. M. Piotto, V. Saudek & V. Sklenar, J. Biomol. NMR 2, 661 - 666 (1992)
2. V. Sklenar, M. Piotto, R. Leppik & V. Saudek, J. Magn. Reson., Series A 102, 241 -245 (1993)





NMR Element: WATERGATE (3-9-19)

```
...

  50u UNBLKGRAD
  p16:gp1
  d16 pl18:f1
  p27*0.231 ph4
  d19*2
  p27*0.692 ph4
  d19*2
  p27*1.462 ph4
  d19*2
  p27*1.462 ph5
  d19*2
  p27*0.692 ph5
  d19*2
  p0*0.231 ph5
  50u
  p16:gp1
  d16
  4u BLKGRAD

.....

ph4=0
ph5=2

.....

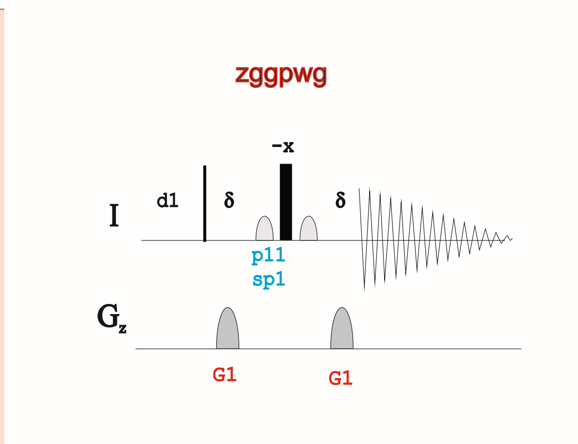
;pl1 : f1 channel - power level for pulse (default)
;pl18: f1 channel - power level for 3-9-19-pulse (watergate)
;p0 : f1 channel -  90 degree pulse at pl18
;                   use for fine adjustment
;p27: f1 channel -  90 degree pulse at pl18
;d16: delay for homospoil/gradient recovery
;d19: delay for binomial water suppression
;     d19 = (1/(2*d)), d = distance of next null (in Hz)
```

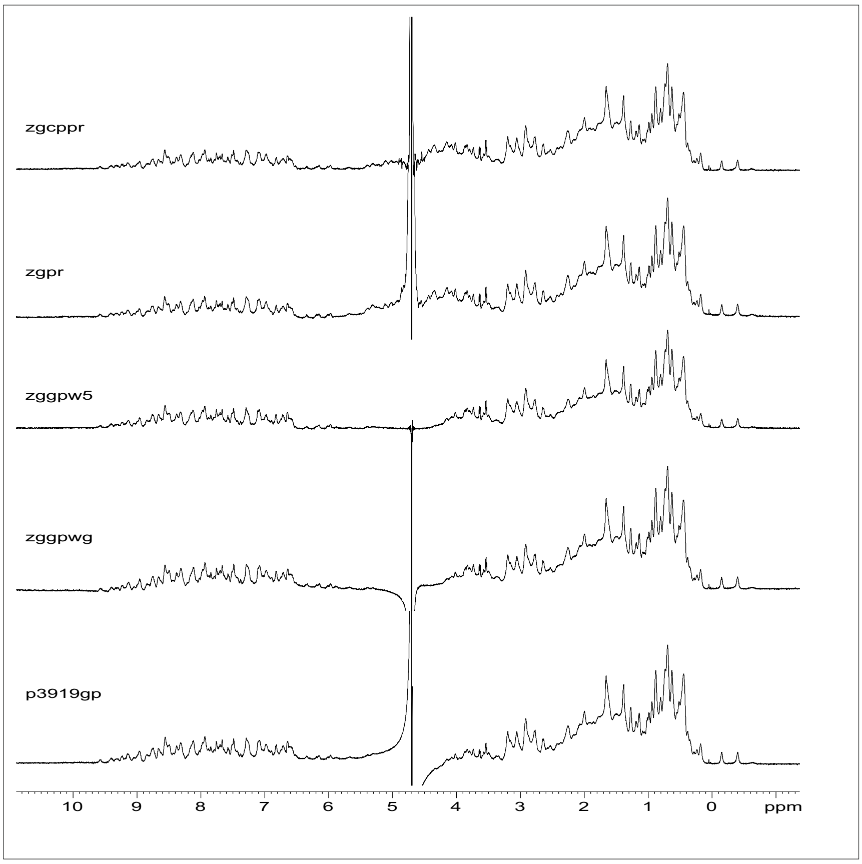
NMR Building Block:
WATERGATE (water-selective 90°)

```
  ...
  50u UNBLKGRAD
  p16:gp1
  d16 pl0:f1
  (p11:sp1 ph2:r):f1
  4u
  d12 pl1:f1
  (p2 ph3)
  4u
  d12 pl0:f1
  (p11:sp1 ph2:r):f1
  46u
  p16:gp1
  d16
  4u BLKGRAD
  ...

ph2=0
ph3=2

;pl0 : 120dB
;sp1 : f1 channel - shaped pulse  90 degree
;p2 : f1 channel - 180 degree high power pulse
;p11: f1 channel -  90 degree shaped pulse
;p16: homospoil/gradient pulse
;d16: delay for homospoil/gradient recovery
```

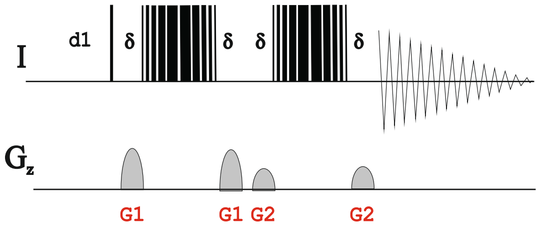




1D Solvent suppression methods in a doubly-labeled ubiquitin sample dissolved in 95%H2O/5%D2O

M. Liu, X. Mao, C. He, H. Huang, J.K. Nicholson & J.C. Lindon, J. Magn. Reson. 132, 125 - 129 (1998)

zggpw5



NMR Element: Excitation Sculpting (W5)

```
...
50u UNBLKGRAD
p16:gp1
d16 pl18:f1
p27*0.087 ph3
d19*2
p27*0.206 ph3
d19*2
p27*0.413 ph3
d19*2
p27*0.778 ph3
d19*2
p27*1.491 ph3
d19*2
p27*1.491 ph4
d19*2
p27*0.778 ph4
d19*2
p27*0.413 ph4
d19*2
p27*0.206 ph4
d19*2
p27*0.087 ph4
50u
p16:gp1
d16

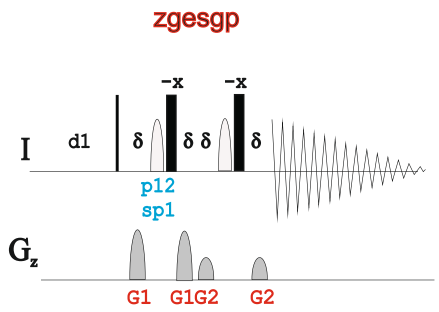
4u
p16:gp2
d16
p27*0.087 ph5
d19*2
p27*0.206 ph5
d19*2
p27*0.413 ph5
d19*2
p27*0.778 ph5
d19*2
p27*1.491 ph5
d19*2
p27*1.491 ph6
d19*2
p27*0.778 ph6
d19*2
p27*0.413 ph6
d19*2
p27*0.206 ph6
d19*2
p27*0.087 ph6
p16:gp2
d16

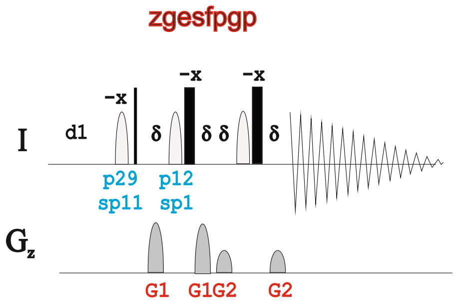
.......
ph3=0
ph4=2
ph5=0
ph6=2
```

```
;pl18: f1 channel - power level for 3-9-19-pulse (watergate)
;p16: homospoil/gradient pulse
;p27: f1 channel -  90 degree pulse at pl18
;d16: delay for homospoil/gradient recovery
;d19: delay for binomial water suppression
;     d19 = (1/(2*d)), d = distance of next null (in Hz)

;gpz1: 34%
;gpz2: 22%
```

T.-L. Hwang & A.J. Shaka, J. Magn. Reson., Series A 112 275-279 (1995)





NMR Element: Excitation Sculpting (water-selective 180°)

```
    ...
    50u UNBLKGRAD
    p16:gp1
    d16 pl0:f1
    (p12:sp1 ph2:r):f1
    4u
    d12 pl1:f1

    p2 ph3

    4u
    p16:gp1
    d16
    TAU
    p16:gp2
    d16 pl0:f1
    (p12:sp1 ph4:r):f1
    4u
    d12 pl1:f1

    p2 ph5

    4u
    p16:gp2
    d16

  ....
  ph2=0
  ph3=2
  ph4=0
  ph5=2
  .....

  ;pl0 : 120dB
  ;sp1 : f1 channel - shaped pulse 180 degree
  ;p2 : f1 channel - 180 degree high power pulse
  ;p12: f1 channel - 180 degree shaped pulse (Squa100.1000)    [2 msec]
  ;p16: homospoil/gradient pulse
  ;d16: delay for homospoil/gradient recovery

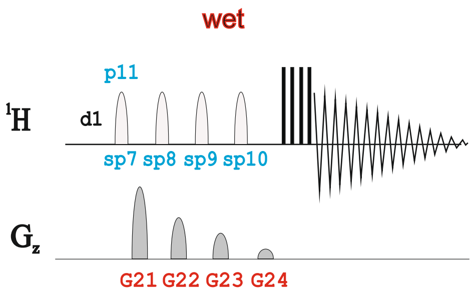
  ;gpz1: 31%
  ;gpz2: 11%
```

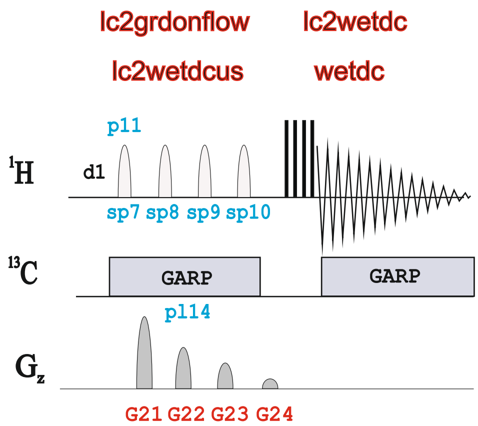
NMR Building Block: WET

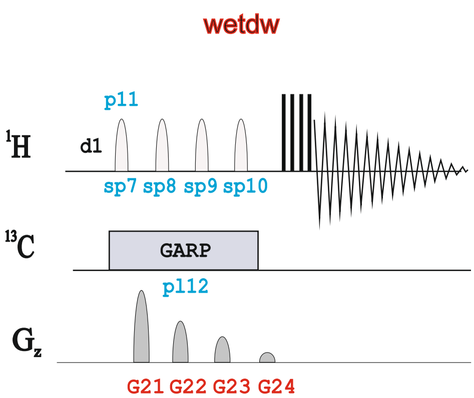
```
   ...
   50u UNBLKGRAD
   d12 pl0:f1
   d13
   (p11:sp7 ph5):f1
   4u
   p16:gp21
   d16 pl0:f1
   (p11:sp8 ph6):f1
   4u
   p16:gp22
   d16 pl0:f1
   (p11:sp9 ph6):f1
   4u
   p16:gp23
   d16 pl0:f1
   (p11:sp10 ph6):f1
   4u
   p16:gp24
   d16

.....
ph5=0
ph6=1
.....

;pl0 : 120 dB
;sp1: f1 channel - shaped pulse
;         powerlevel for 90 degree pulse
;sp7: f1 channel - shaped pulse
;sp8: f1 channel - shaped pulse
;sp9: f1 channel - shaped pulse
;sp10: f1 channel - shaped pulse
;p11: f1 channel -  90 degree shaped pulse
;p16: homospoil/gradient pulse                      [2msec]
;d16: delay for homospoil/gradient recovery
;gpz21: 80%
;gpz22: 40%
;gpz23: 20%
;gpz24: 10%
;use power levels              sp7        sp8        sp9        sp10
;                       sp1    +0.87      -1.04      +2.27      -5.05
```

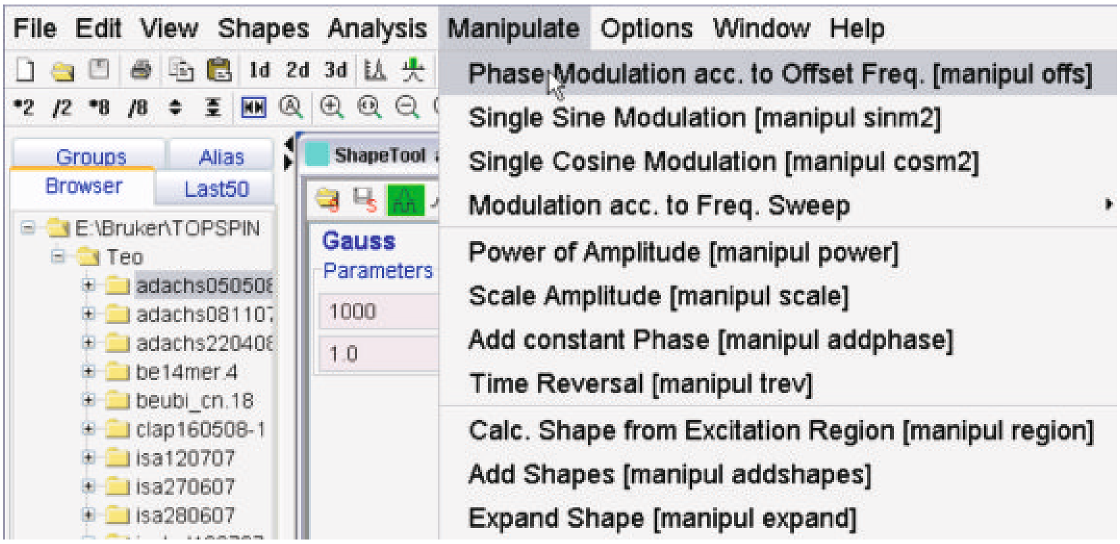




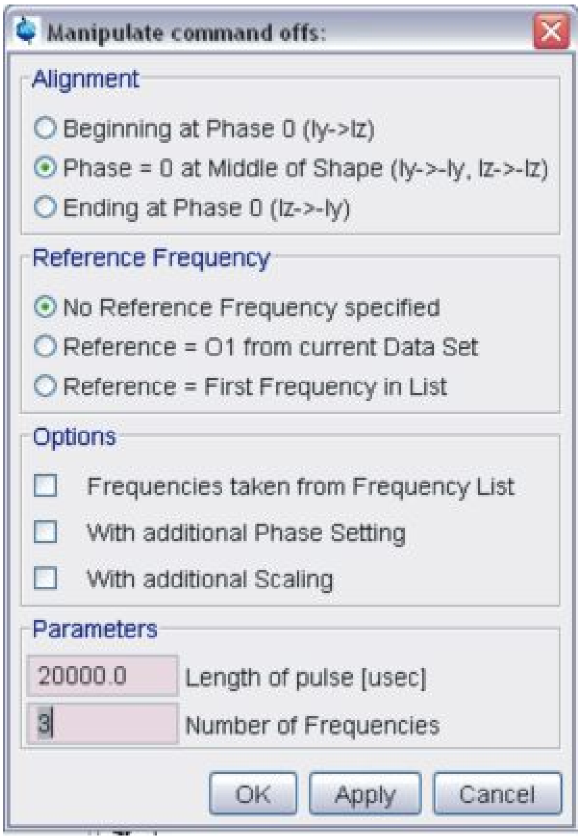


How to generate a multi-frequency shape.

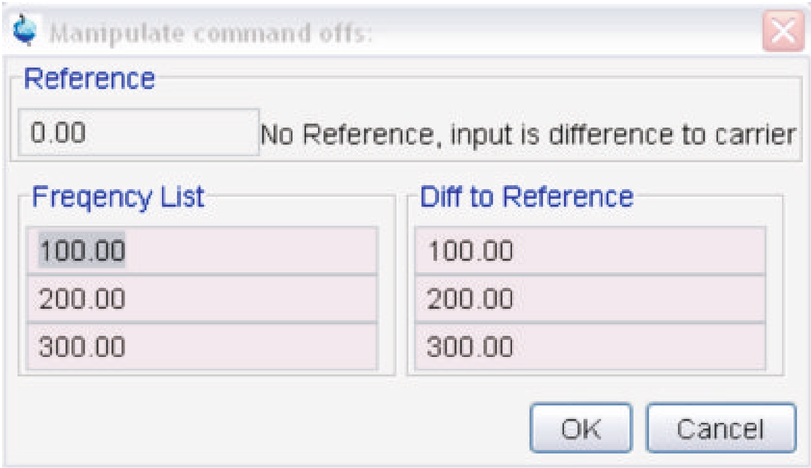
Step 1: Open the stdisp tool and select the appropiate shape (e.g: Gauss1.1000)
Step 2: Select Phase Modulation according offset in the Manipulate menu:



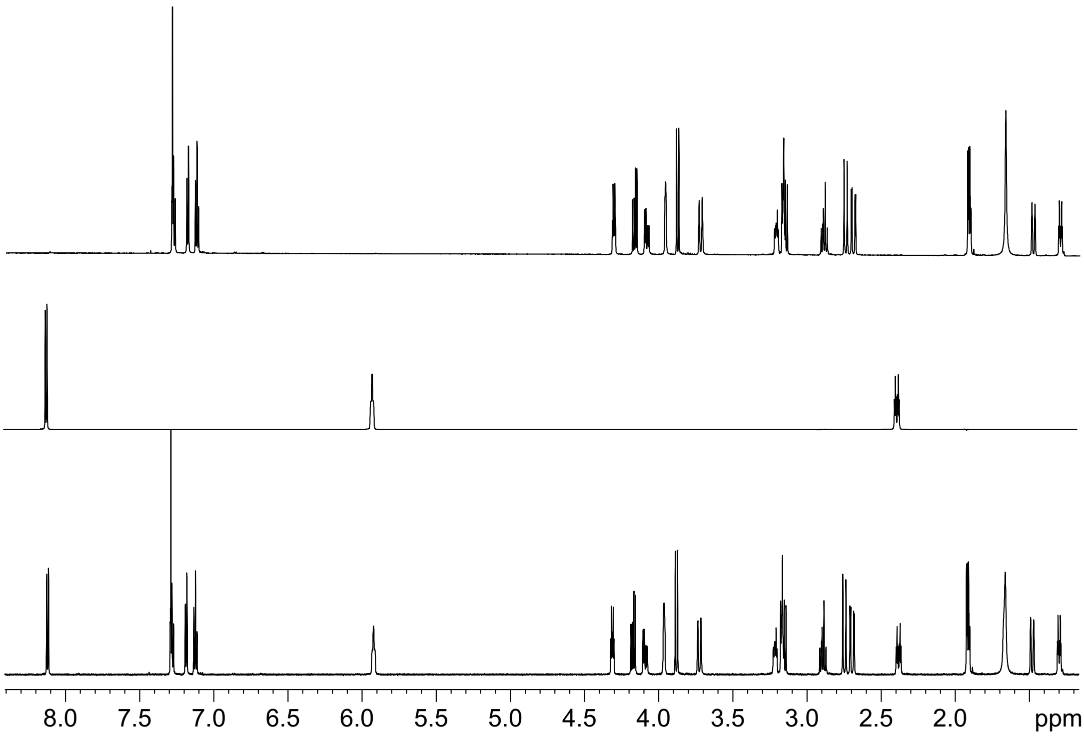
Step 3:Define type of manipulation and number of frequencies to be excited:



Step 4:Define frequencies:



Step 5:Store the resulting shape. It can be used in any selective-excitation or solvent-suppression pulse program using shaped (sp) pulses for multiexcitation (Hadamard spectroscopy) or multiple-solvent suppression (HPLC-NMR) purposes



A complete Description of creating, analyzing and Manipulating RF and Gradient Shapes in the **Shape Tool Manual** (see **Help** menu in Topspin)

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SINGLE/MULTIPLE SUPPRESION
LC-NMR EXPERIMENTS

Single-Multiple Presaturation
LC-NMR Experiments

1D ^{1}H spectrum

- 1D ^{1}H with double presaturation (**lc1prf2** | LC1D12)
- 1D ^{1}H with triple presaturation (**lc1prft**)
- 1D ^{1}H with presaturation using shaped pulses, composite pulses and CW decoupling on f2 during acquisition (**lclcpcwps**)

1D NOESY

- 1D NOESY with presaturation (**noesypr1d**)
- 1D NOESY with presaturation and CW decoupling on f2 (**lc1pncw**)
- 1D NOESY with double presaturation and CW decoupling on f2 (**lc1pncwfd**)
- 1D NOESY with presaturation using shaped pulse and CW decoupling on f2 (**lc1pncwps**)
- 1D NOESY with double presaturation (**lc1pnf2**)
- 1D NOESY with multiple presaturation (**lc1pnfr**)
- 1D NOESY with triple presaturation (**lc1pnft**)
- 1D NOESY with presaturation using shaped pulse (**lc1pnps**)

Pseudo-2D-sequence

- Pseudo-2D-sequence for lc-nmr on flow detection (**lc2**)
- Pseudo-2D-sequence for lc-nmr on flow detection with power-gated decoupling (**lc2pg**)
- Pseudo-2D-sequence for lc-nmr on flow detection with presaturation (**lc2pn**)
- Pseudo-2D-sequence for lc-nmr on flow detection with double presaturation (**lc2pnf2**)
- Pseudo-2D-sequence for lc-nmr on flow detection with solvent gradients (**lc2pnf2ul**)
- Pseudo-2D-sequence for lc-nmr on flow detection with solvent gradients (**lc2pnpl**)
- Pseudo-2D-sequence for lc-nmr on flow detection (**lc2pnps**)
- Pseudo-2D-sequence for lc-nmr on flow detection with solvent gradients (**lc2pnul**)
- Pseudo-2D-sequence for lc-nmr on flow detection with presaturation (**lc2pr**)
- Pseudo-2D-sequence for lc-nmr on flow detection with double presaturation (**lc2prf2**)
- Pseudo-2D-sequence for lc-nmr on flow detection with presaturation using shape pulse (**lc2ps**)

Also see in 1D solvent suppresion methods

1D ^{1}H spectrum using WET solvent suppression

- 1D ^{1}H with WET (wet)
- 1D ^{1}H with WET and CW decoupling on f2 during WET and acquisition (wetdc | LC1DWTDC)
- 1D ^{1}H with WET and CW decoupling on f2 during WET (wetdw)
- 1D ^{1}H WET solvent suppression with shape pulse and C-13 decoupling on f2 during WET and acquisition for LC isocratic runs (lc2wetdc)
- 1D ^{1}H WET solvent suppression with shape pulse and C-13 decoupling on f2 during WET and AQ with intermediate preparation scan into second dataset for LC gradient runs with updated shapes (lc2wetdcus | LC2DWTUS)
- 1D ^{1}H WET solvent suppression with shape pulse and C-13 decoupling on f2 during WET and acquisition with intermediate preparation scan into second dataset for LC gradient runs with updated shapes (lc2grdonflow)

Also see LC-NMR related pulse programs in:

2D homonuclear J-resolved

- 2D J-resolved with double presaturation and cw-decoupling on f2 (lcjrescwfdprqf)
- 2D J-resolved with presaturation and cw-decoupling on f2 (lcjrescwprqf)
- 2D J-resolved with presaturation using shape pulse and cw-decoupling on f2 (lcjrescwpsqf)
- 2D J-resolved with double presaturation (lcjresf2prqf)
- 2D J-resolved with presaturation (lcjresprqf)
- 2D J-resolved with presaturation using shape pulse (lcjrespsqf)

2D TOCSY

- 2D TOCSY with double presaturation and cw-decoupling on f2 (lcmlevcwfdpcph)
- 2D TOCSY with presaturation and cw-decoupling on f2 (lcmlevcwpcphps)
- 2D TOCSY with double presaturation using composite pulse (lcmlevf2pcph)
- 2D TOCSY with double presaturation (lcmlevf2phpr | LCML12)
- 2D TOCSY with presaturation using shape pulse and composite pulse (lcmlevpcphps)
- 2D TOCSY with presaturation using composite pulse (lcmlevpcph)

2D Experiments using WET

- 2D COSY using WET (cosydcphwt | COSYDCPHWT)
- 2D TOCSY using WET (mlevdcphwt | MLEVDCPHWT)
- 2D HSQC using WET (hsqcetgpsiwt | HSQCETGPSIWT)

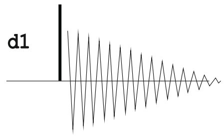
2D Experiments using single/multiple presaturation using shape pulse

- Phase-sensitive 2D COSY using using single/multiple presaturation (cosycwphps | COSYCWPHPS)
- Phase-sensitive 2D HSQC using using single/multiple presaturation (hsqcphps)
- 2D HMBC using using single/multiple presaturation (hmbcndpsqf)

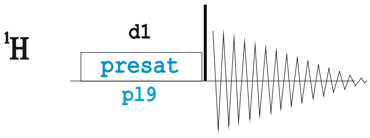
Related experiments:

- Also see 1D Solvent suppression

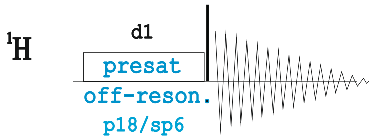
lc2



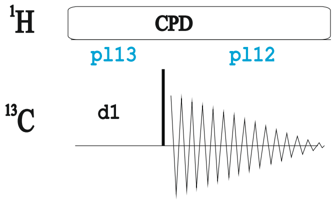
lc2pr



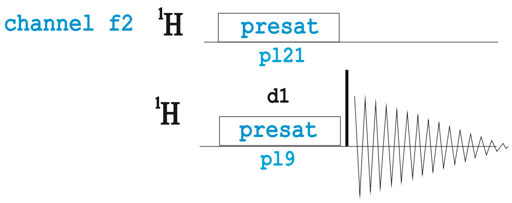
lc2ps



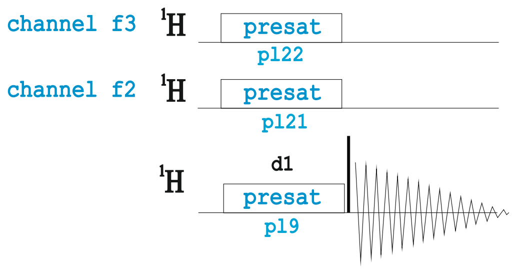
lc2pg



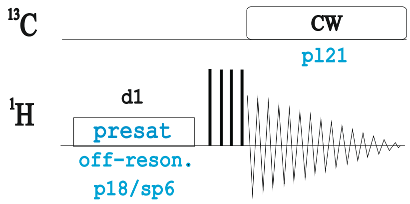
lc1prf2
lc2prf2



lc1prft

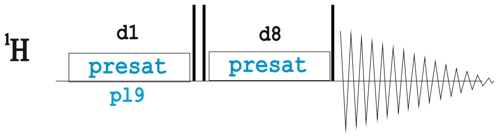


lc1cpcwps



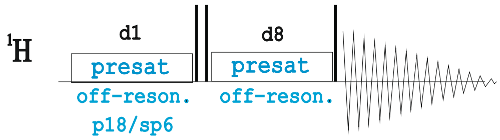
lc2pn

lc2pnul

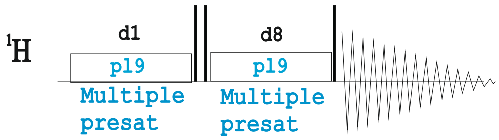


lc2pnps

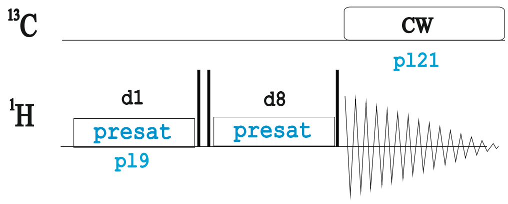
lc1pnps



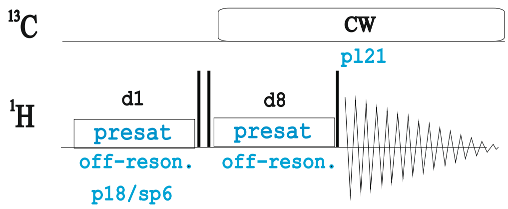
lc1pnfr



lc1pncw



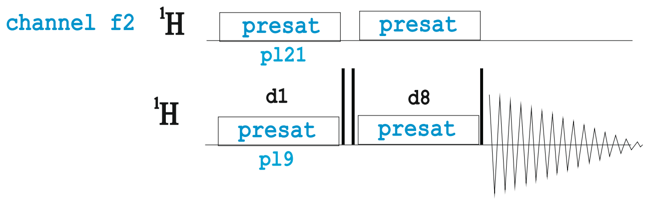
lc1pncwps



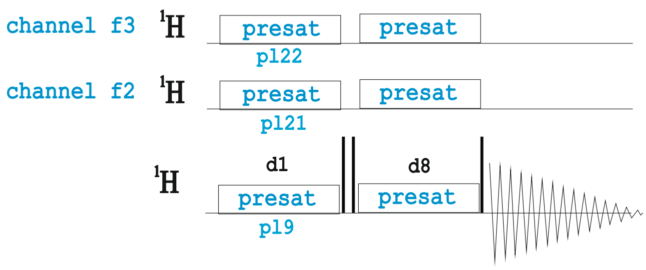
lc2pnf2

lc1pnf2

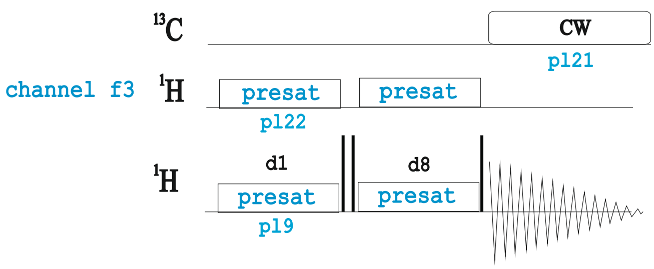
lc2pnf2ul



lc1pnft



lc1pncwfd



A Decription of setting-up and rnning LC-NMR Experiment can be found in the **LC-NMR Manual** (see **Help** menu in Topspin)

List of Automation Protocols to run in LC-NMR Applications:

1. expsetup: Automation Setup
2. getinfo: Get information into the title.
3. multicmd: Perform Multiple Commands
4. au_lc1d: 1D spectra with solvent suppression
5. au_lc2d: 2D spectra with solvent suppression
6. au_lconflow: Onflow spectra with solvent suppression
7. lcprep: Acquire preparation spectrum
8. lcsetup: Search for solvents
9. lcabsf: Baseline Correction
10. lcsino: Signal-to-noise Calculation
11. proc_onflow: 1D & 2D processing
12. lctshim: Shimming with TOPSHIM
13: lcgshim: Shimming with gradshimau
14. lczshim: Iterative Shimming of the lineshape

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^{19}F SPECIFIC EXPERIMENTS

^{19}F NMR Experiments

- **1D ^{19}F experiments:**

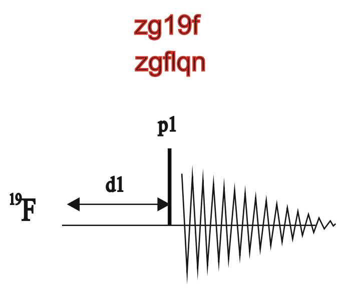
  ^{1}H-decoupled ^{19}F spectrum (**zgfhigqn** / **zgfhigqn.2** | F19CPD)
  ^{1}H-coupled ^{19}F spectrum (**zgflqn** | F19)
  Using 19F lockswitch unit (**zg19f**)
  ^{19}F-homodecoupled ^{19}F spectrum (**zhflhdqn**)

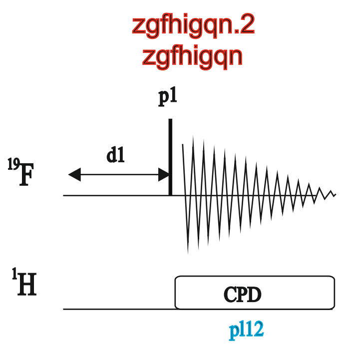
  ^{19}F-decoupled 1D ^{1}H spectrum (**zghfigqn** / **zghfigqn30** | PROF19DEC)

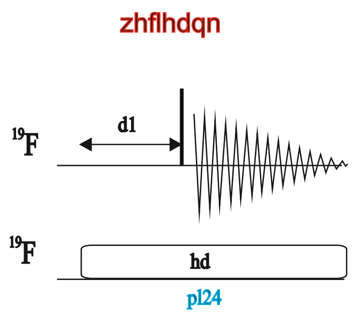
  ^{1}H spectrum with ^{19}F-presaturation (**zgf2hfpr**)

- **2D ^{19}F experiments:**

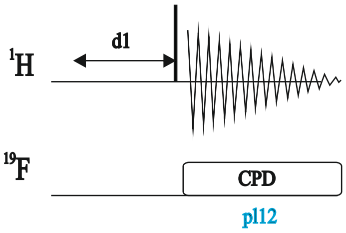
  2D ^{19}F-^{1}H HETCOR experiment (**hfcoqfqn**)
  2D ^{19}F-^{1}H HOESY experiment (**hoesyfhqfqnrv**)



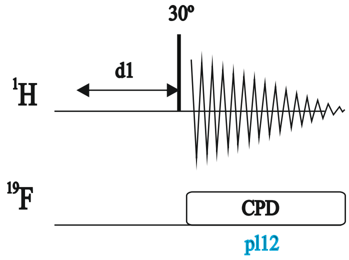


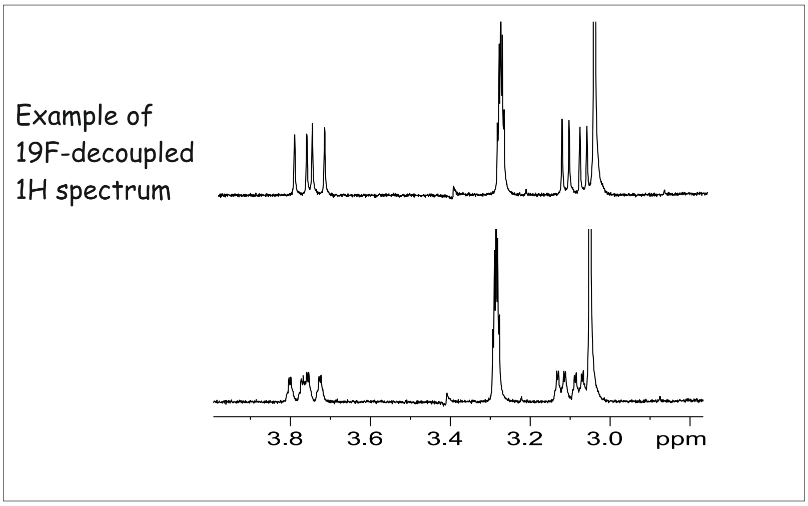


zghfigqn

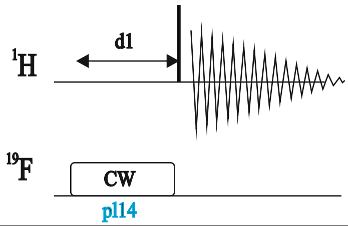


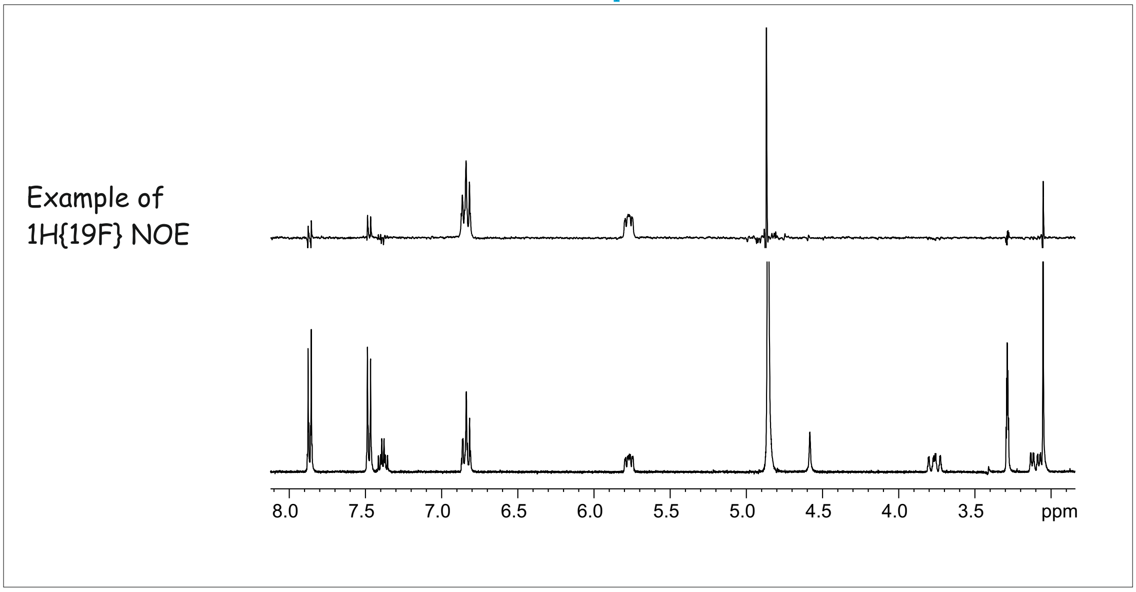
zghfigqn30



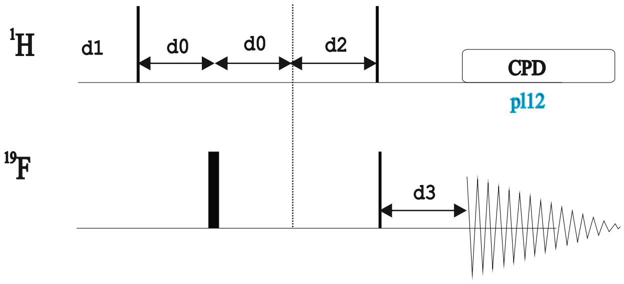


zgf2hfpr

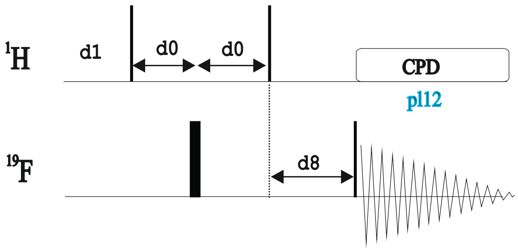


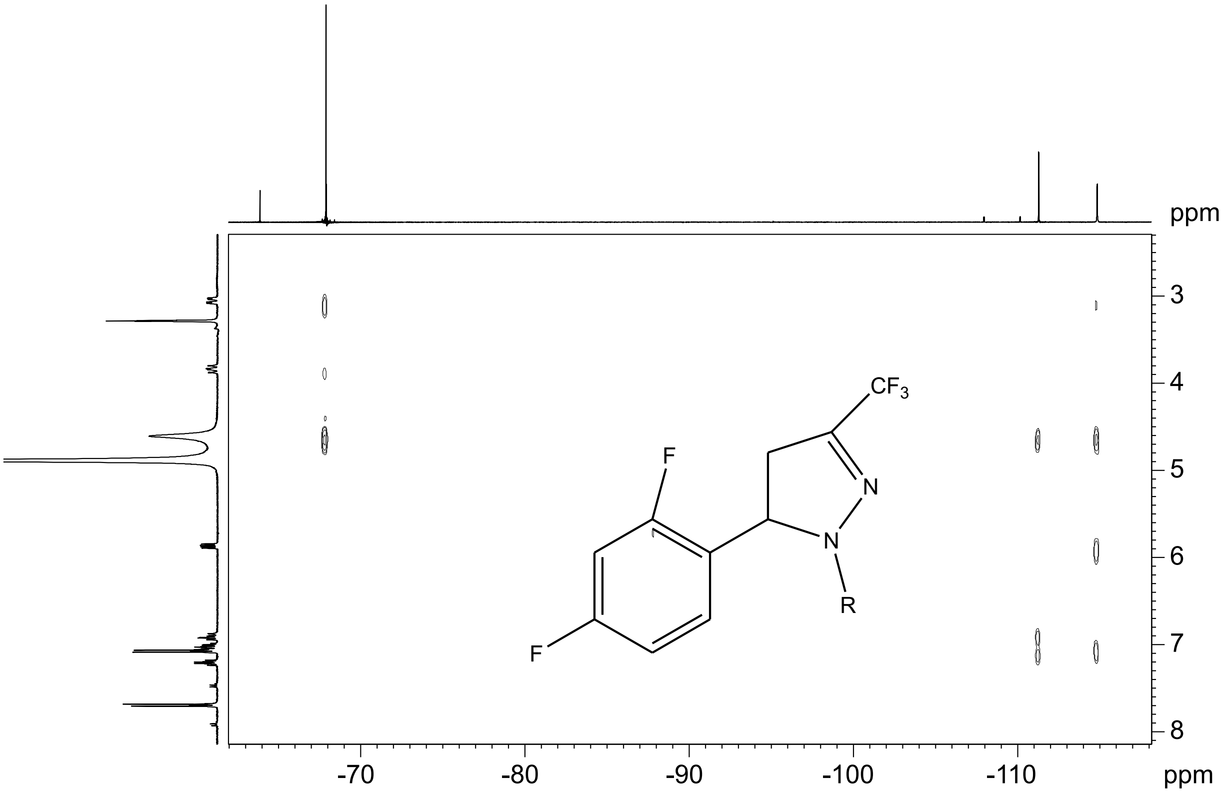


hfcoqfqn



hoesyfhqfqnrv





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^{2}H SPECIFIC EXPERIMENTS

^{2}H Experiments

- **1D spectra:**

  1D ^{2}H spectrum (**zg2h**)
  Using ^{2}H lockswitch unit (**zg2h.2**)
  1D X-decoupled ^{2}H spectrum (**zgig2h**, **zgig2hf4**)

- **2D spectra:**

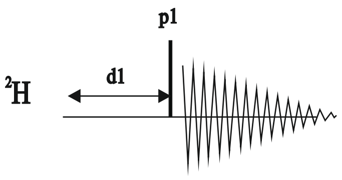
  Magnitude-mode 2D HETCOR with ^{2}H-decoupling (**hxcoqf2h**)

- **Miscellaneous:**

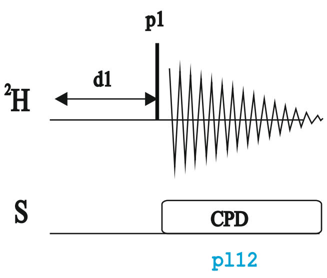
  High-power 90° ^{2}H decouple pulse calibration (**decp902h**, **decp902hf4**)

Also see:
2H-decoupled 3D triple-resonance experiments

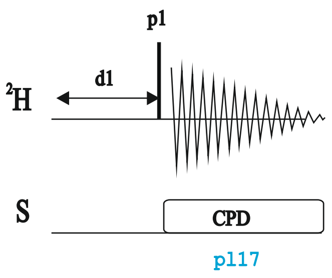
zg2h.2
zg2h



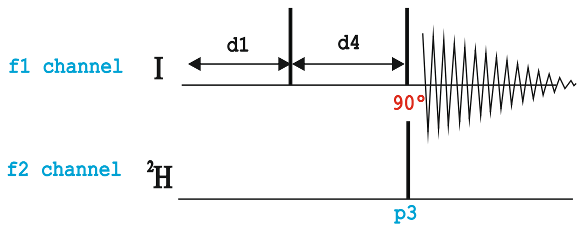
zgig2h



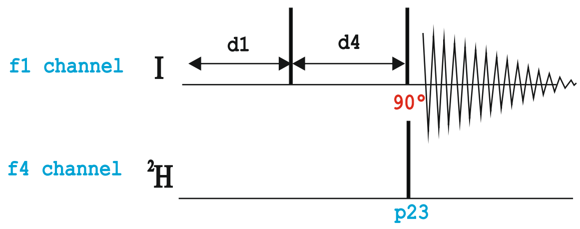
zgig2hf4



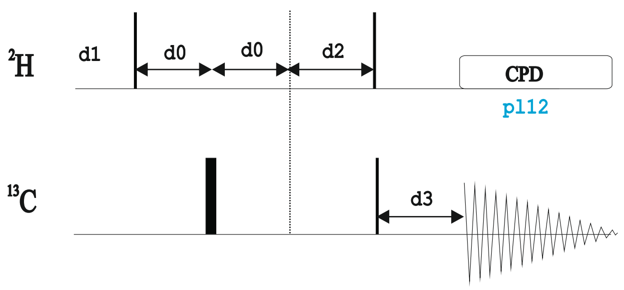
decp902h



decp902hf4



hxcoqf2h



A Decription of Gradient-Shimming interface and the User Manual for the Automatic Shimming can be found in the **Gradient Shimming Manual** and **Topshim Manual**, respectively
(see **Help** menu in Topspin)

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BASIC 1D GRADIENTS

Basic 1D Gradients

- **Standard:**

  Gradient-enhanced 1D Echo experiment (**zggegp**)
  Gradient-enhanced 1D Spin-Echo experiment (**zggpse**)

- **Gradient Calibration:**

  Gradient Strength Calibration (**calibgp**)
  Gradient Preemphasis Adjustament. Gradient Recovery Test (**preempgp2**)

- **Gradient shimming:**

  1D Gradient Echo for gradshim-procedure (**imgegp1d**)
  Using 2H lockswitch unit (**imgegp1d2h**)
  Using 19F lockswitch unit (**imgegp1d19f**)
  Using selective pulse (**imgegpsp1d**)

  1D convection-compensated Gradient Echo for gradshim-procedure (**imgegpcv1d**)
  Using 2H lockswitch unit (**imgegpcvsp1d2h**)

  2D Gradient Echo for gradshim-procedure (**imgegp2d**)

  3D Gradient Echo for gradshim-procedure (**imgegp3d**)
  Using BSMS RCB board(**imrcbgegp3d**)

- **Zero-Quantum Gradient Calibration:**

  ZQ setup calibration (**zs_setup**)

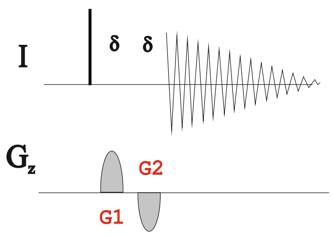
Basic Syntax for a Gradient:

```
#include <Grad.incl>

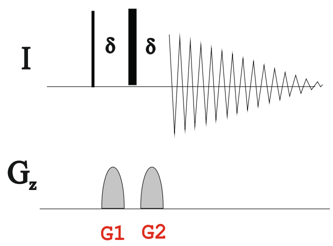
....
 50u UNBLKGRAD
  p16:gp1
  d16
  20u BLKGRAD
.....

;p16: homospoil/gradient pulse
;d16: delay for homospoil/gradient recovery
;gpz1: gradient strength in %
;gpnam1: SMSQ10.100
```

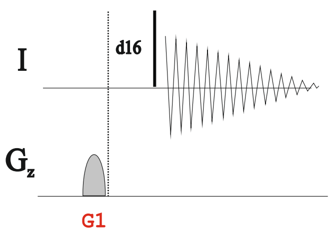
zggegp



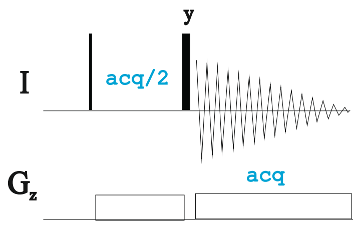
zggpse



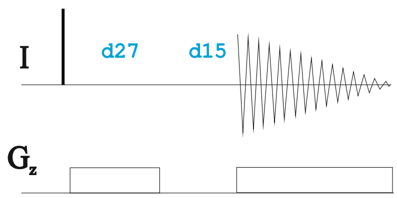
preempgp2



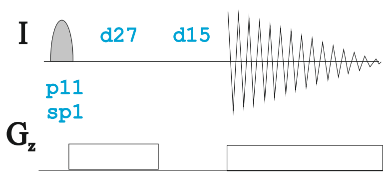
calibgp



imgegp2d
imgegp3d
imgegp1d
imgegp1d19f
imgegp1d2h



imgegpsp1d



imgegpspcv1d

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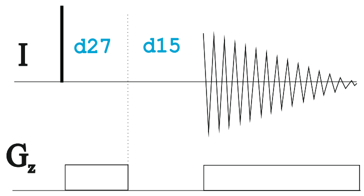
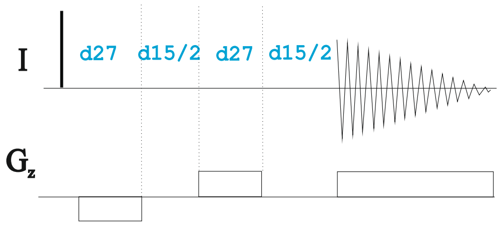
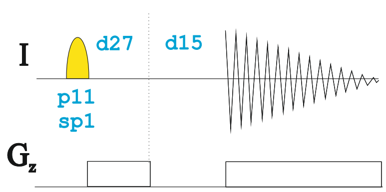


else



imgegpspcv1d2h

if ZGOPTNS= -DLABEL_NOCOMP



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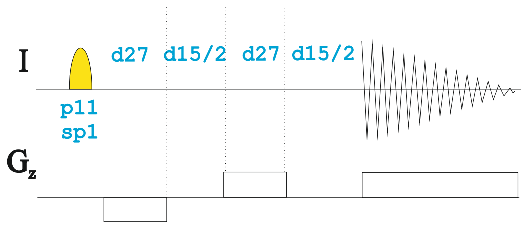
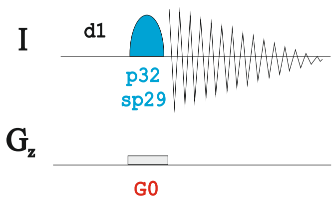


zs_setup



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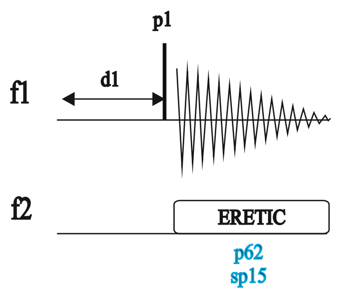
1D ERETIC SEQUENCES

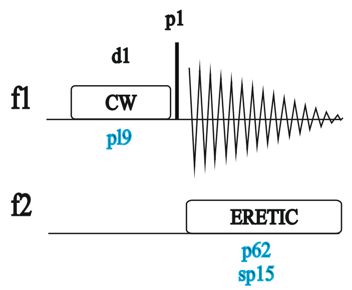
ERETIC 1D pulse sequences

- 1D ERETIC for quantitative measurements and using f2 channel for ERETIC signal
  - Conventional 1D (**eretic**)
  - Conventional 1D using read pulse of 30° (**eretic30**)
  - Conventional using presaturation (**ereticpr**)
  - Conventional using presaturation and read pulse of 30° (**ereticpr30**)
  - Using 1D NOESY and preaturation (**ereticgppn**)

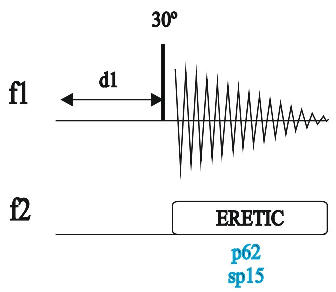
eretic



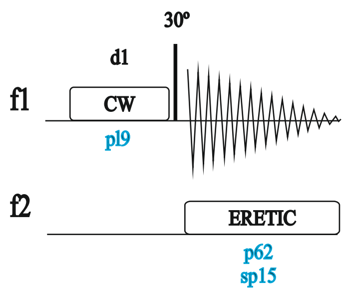
ereticpr



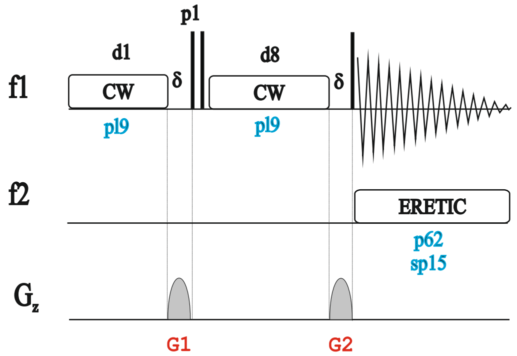
eretic30



ereticpr30



ereticgppn



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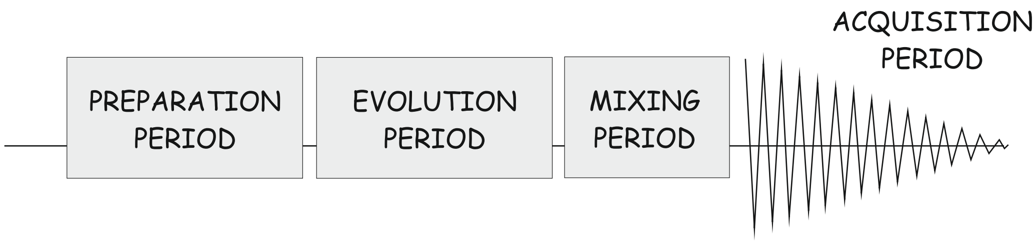
GENERAL SCHEMES FOR
2D/3D/4D EXPERIMENTS

NMR Pulse Sequence: Definition of Time Periods

A NMR pulse sequence can be splitted in several different and independent parts, namely, preparation, evolution, mixing and acquisition periods:



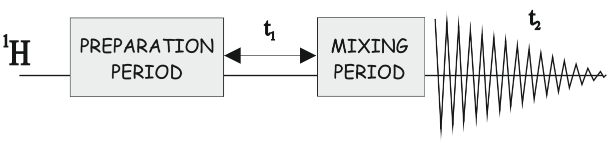
The relative sensitivity of a given NMR pulse scheme depends basically of two factors: i) the starting nucleus and ii) the detected nucleus as a function of the following relationship:

$$S/N \;\alpha\; \gamma_{excited}\gamma_{detected}^{3/2}$$

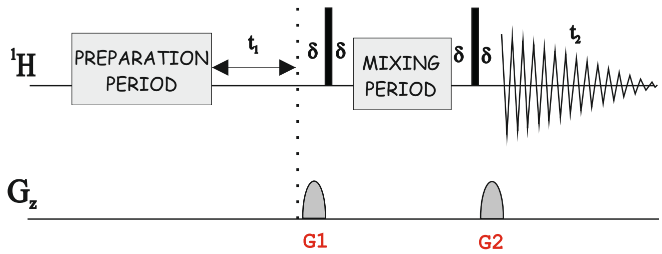
Thus, due to the highest γ value for 1H, it is generally preferable to start from 1H and to detect 1H when possible.

GENERAL 2D HOMONUCLEAR SCHEMES

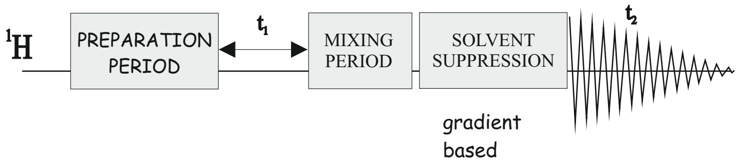
1. Phase-Cycled homonuclear



2. Gradient-Enhanced homonuclear



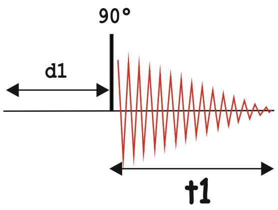
3. Solvent-Suppressed homonuclear



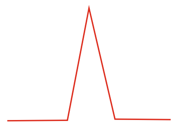
The preparation period can also includes a solvent-suppression element

HOW 2D NMR WORKS?

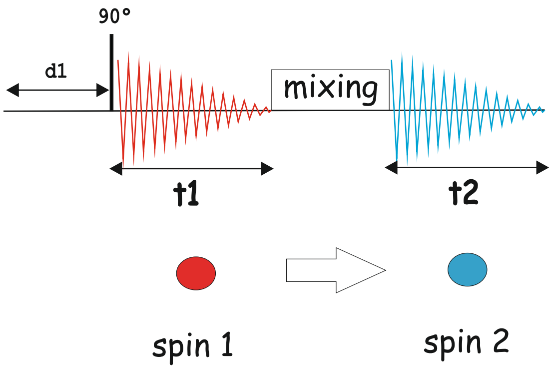
Consider the most basic 1D experiment:



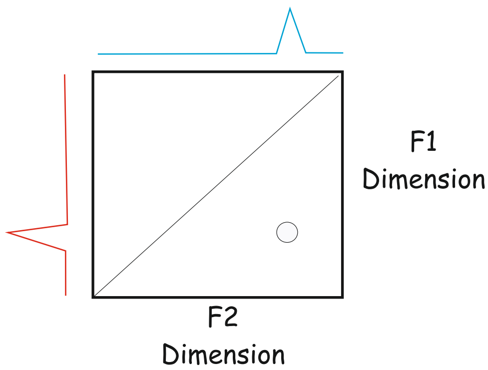
A target spin 1 evolves during t1:



Imagine an experiment in which the information of spin 1 is transferred to spin 2



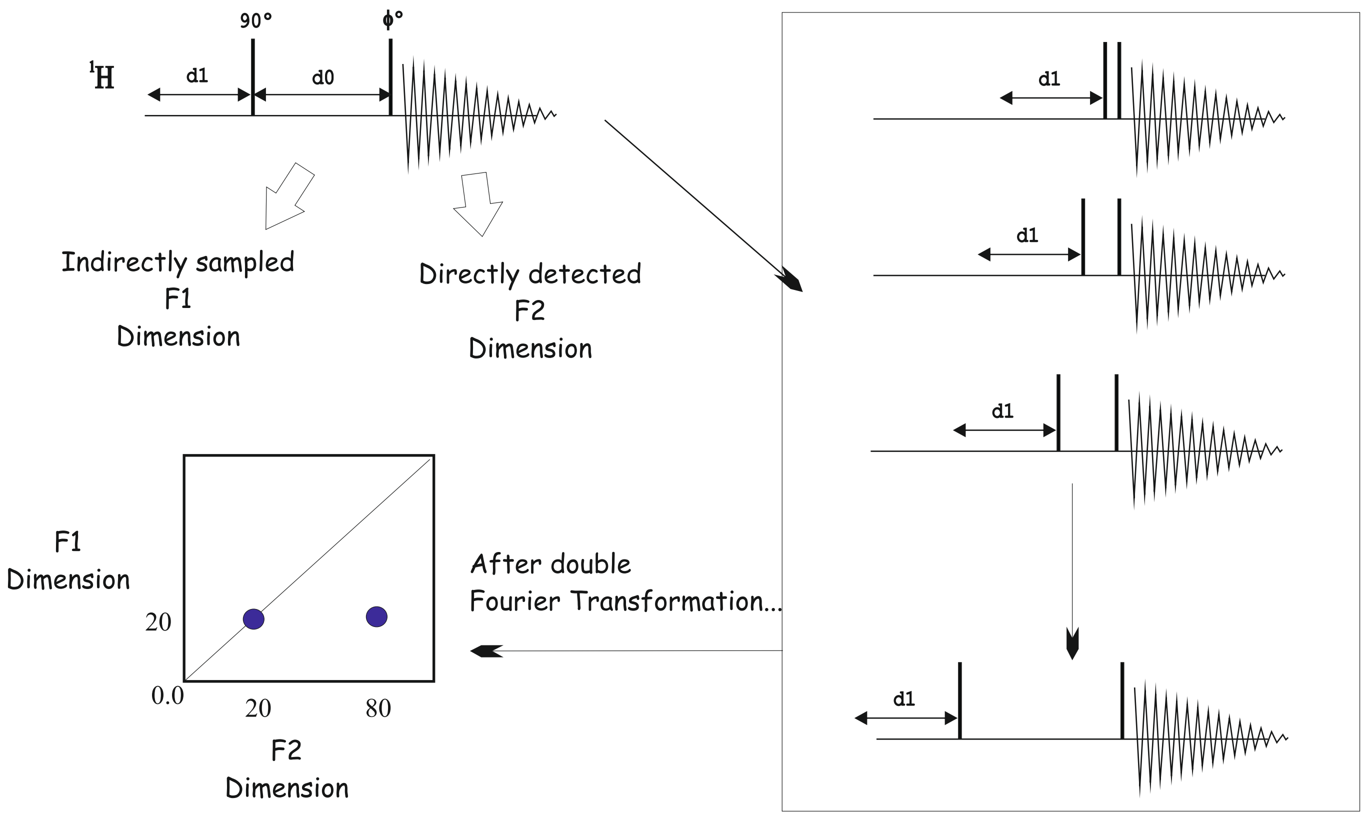
The resulting 2Dmap looks like this



HOW 2D NMR WORKS?: COSY

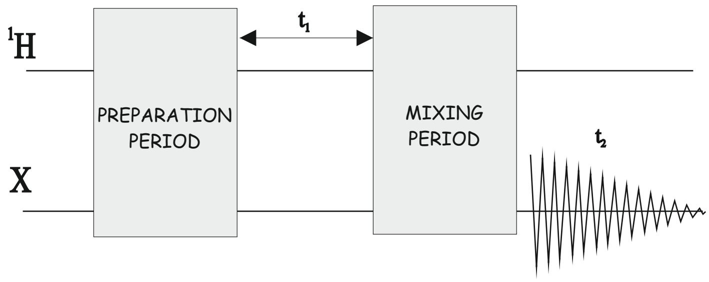
The experiment is repeated **td** times using a variable period with an initial **d0** value and an increment of **in0**, giving the maximum value of **d0*td**

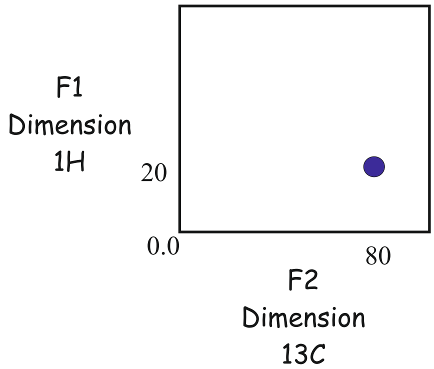
cosyqf



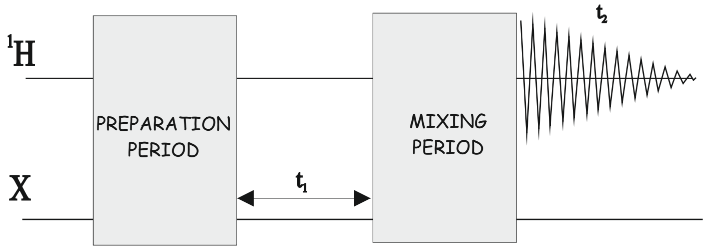
GENERAL 2D 1H-X HETERONUCLEAR

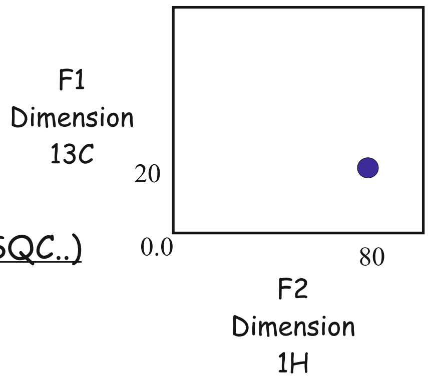
1. Old X-detected (HETCOR, COLOC ....)



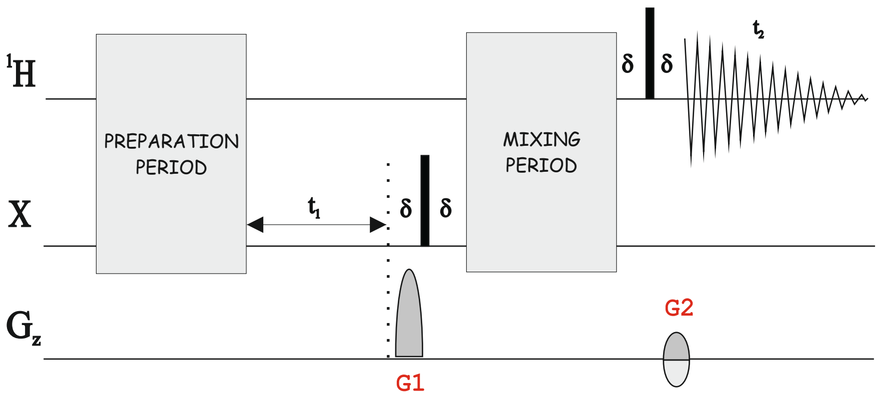


2. Phase-Cycled Inverse (old HMQC, old HSQC...)



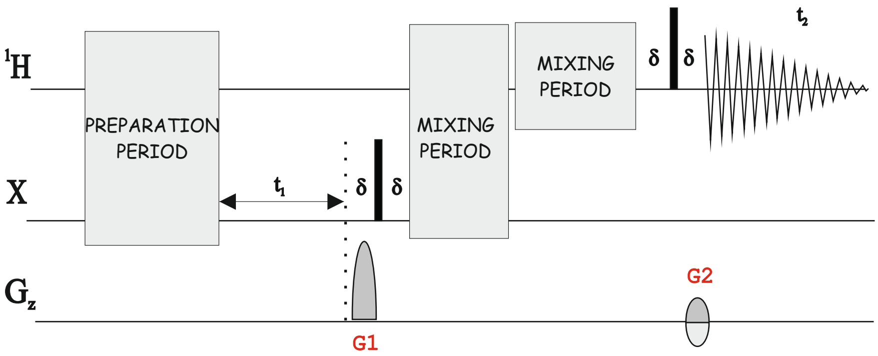


3. Gradient-Enhanced Inverse (Gradient-Enhanced HMQC, HSQC..)

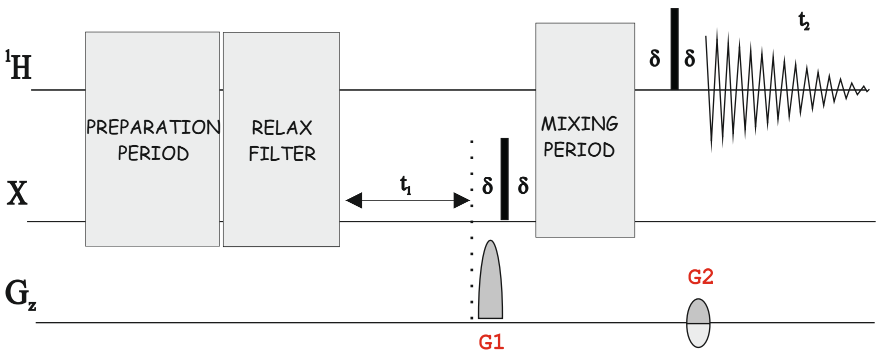


Solvent-suppression elements can be included in both preparation and mixing periods

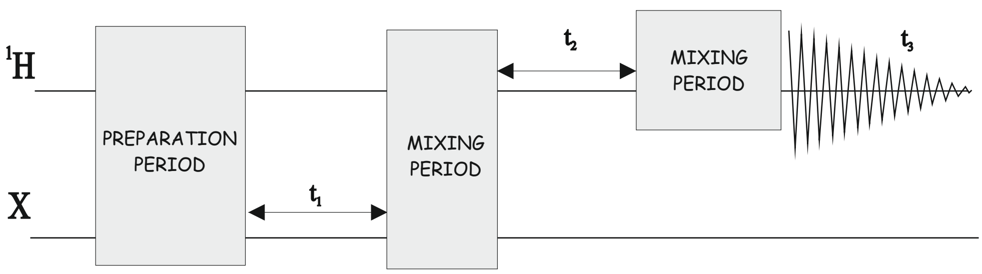
GENERAL 2D HYBRID INVERSE

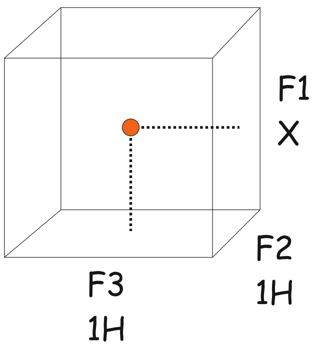


GENERAL 2D RELAXATION

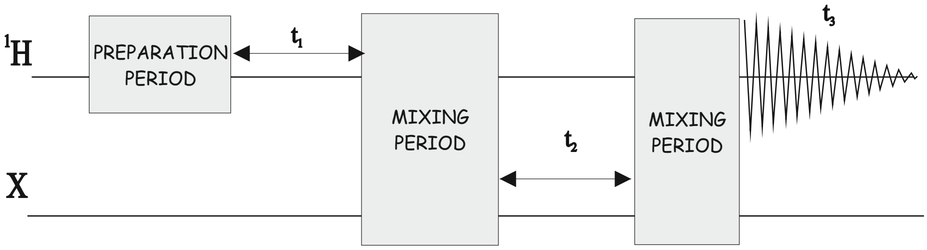


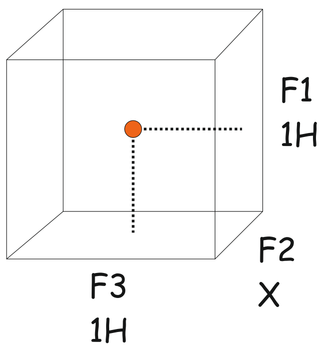
GENERAL 3D DOUBLE-RESONANCE





Example: 3D HSQC-TOCSY experiment

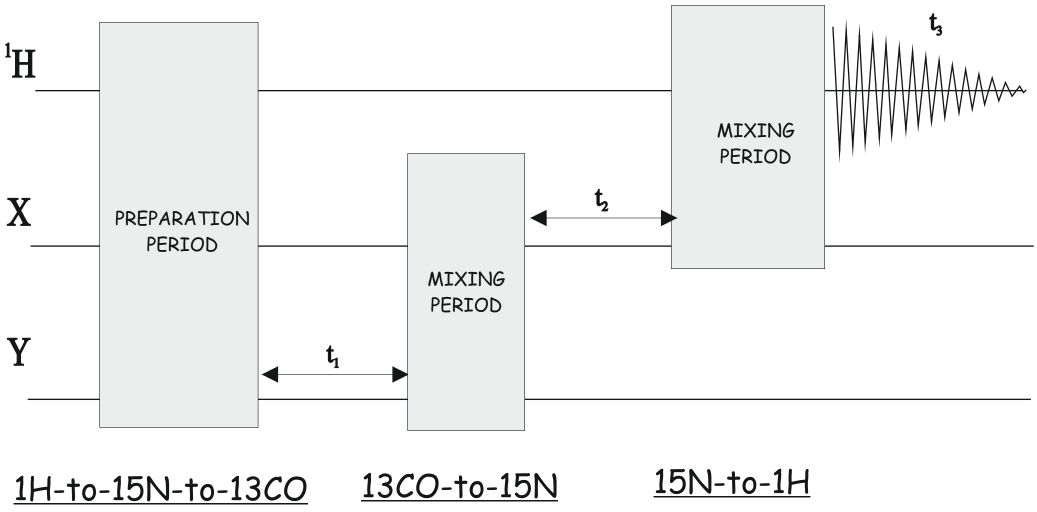




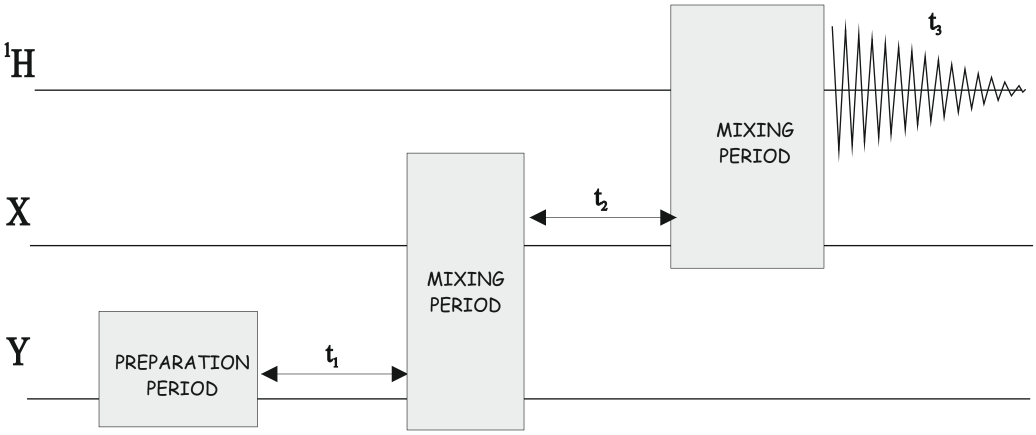
Example: 3D TOCSY-HSQC experiment

GENERAL 3D TRIPLE-RESONANCE

Out-and-back
(Example: 3D HNCO)

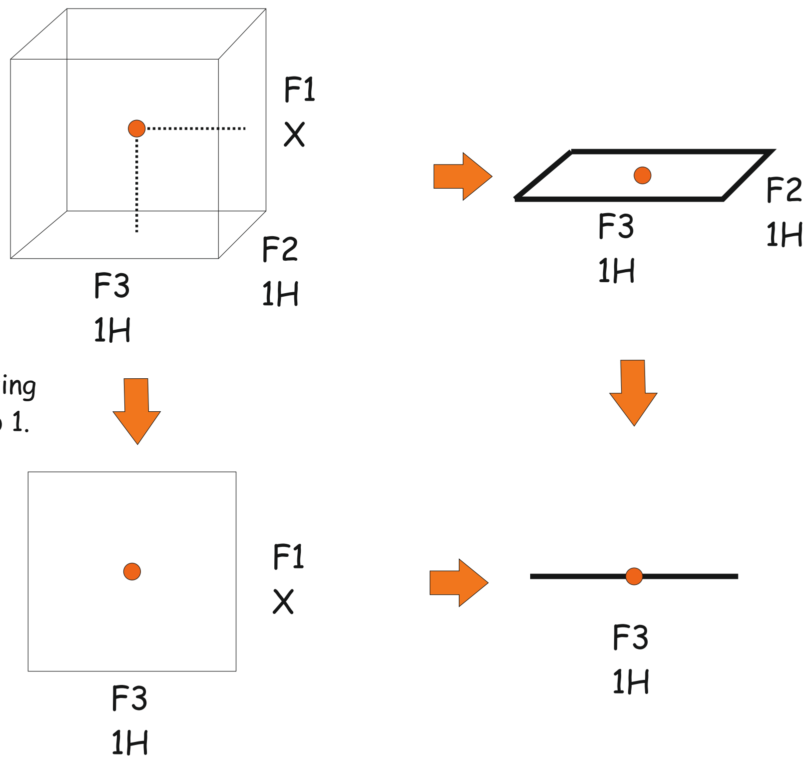


Out-and-stay



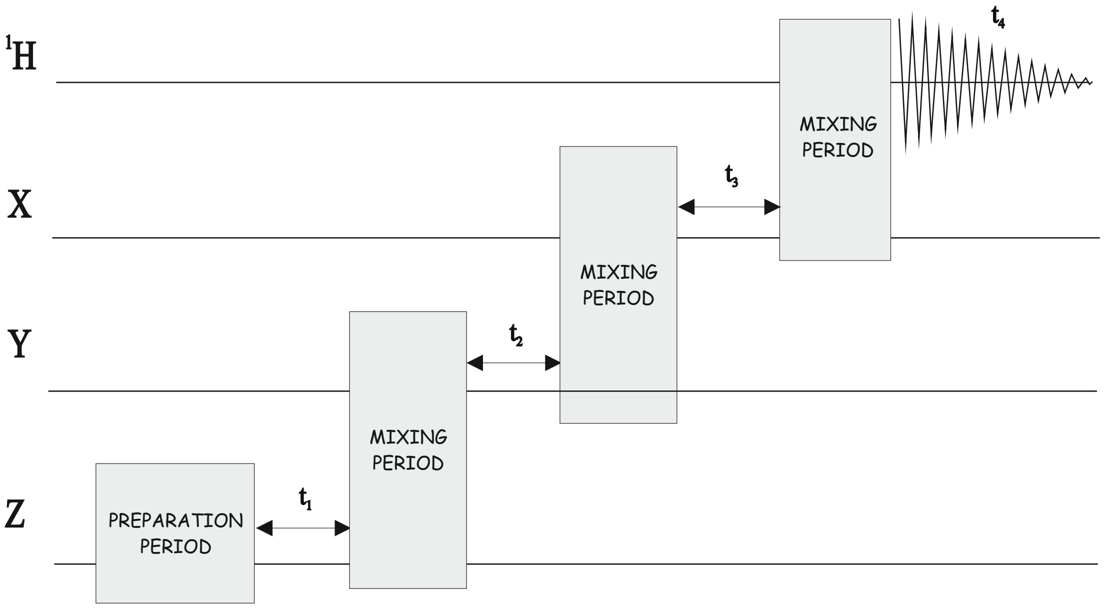
ANALYZING 3D SPECTRA

The same as 2D NMR but with two independent variable evolution periods. The first planes can be recorded from the same dataset by setting the number of TD in each indirect dimension to 1.



GENERAL 4D

4 different frequencies can be sampled during the same experiment



In principle, nD spectroscopy is possible by introducing (n-1) variable evolution periods.
Major advantage: Excellent dispersion and maximum information from a single spectrum.
Major inconvenients are:
i) the sequence becomes very long and sensitivity losses due to fast transverse relaxation is usually a great problem;
ii) The overall experimental acquisition time is increased.

Some solutions:
1. Combination of several 3D experiments
2. Use of time-sharing evolution approach: Reduced dimensionality ...

Classification of NMR Experiments. The 3 key points:

Initial Nucleus: Determine d1
Indirect Evolving Nucleus: Determine F1 dimension
Detected Nucleus: Determine F2 dimension

H1 → H2

J_{HH}	NOE
COSY	NOESY
RELAY	ROESY
TOCSY	(EXSY)

H1 → C1

$^{1}J_{CH}$	NOE
DEPT/INEPT	
HETCOR	HOESY

H1 → C2

$^{n}J_{CH}$

COLOC

H1 → C1 → H1

$^{1}J_{CH}$

HMQC
HSQC
Edited HSQC
TROSY

H1 → C2 → H1

$^{n}J_{CH}$

HMBC

H1 → C1 → H1 → H2

$^{1}J_{CH}$ + J_{HH}

HSQC-COSY
HSQC-TOCSY

$^{1}J_{CH}$ + NOE

HSQC-NOESY
HSQC-ROESY

H1 → H2 → C2 → H1

J_{HH} + $^{1}J_{CH}$

TOCSY-HSQC

NOE + $^{1}J_{CH}$

NOESY-HSQC

H1 → C1 → C2

C1 → C2

$^{1}J_{CC}$

INADEQUATE

H1 → C1 → C2 → C1 → H1

$^{1}J_{CH}$ + $^{1}J_{CC}$

ADEQUATE

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2D COSY EXPERIMENTS

Experiment Description

A COSY (COrrelation SpectroscopY) experiment is a high-sensitive tool that provides through-bond proton-proton connectivities by means of scalar J coupling constants.

Sample Requirements

COSY experiments can be recorded on any type of sample.
Solvent-supressed versions are required for samples dissolved in H2O.

Hardware Requirements

In principle, COSY experiments can be recorded on any probehead.
An optional pulsed-field gradient coil (highly recommended) is required for gradient-based versions.

NMR Spectrum

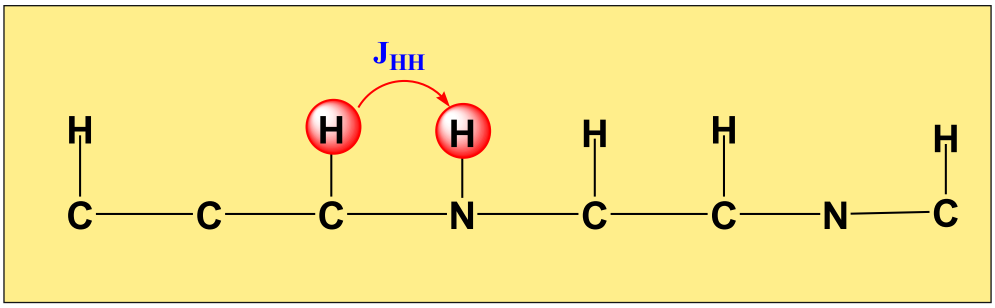
The experiment yields a 2D proton-proton correlation map with two different types of signals: i) autocorrelation diagonal peaks, and ii) Off-diagonal cross-peaks correlating J-coupled spins. COSY spectra are usually represented in magnitude-mode, and a COSY-DQF experiment is recommended when phase-sensitive data is required,

References:

W.P. Aue, E. Bartholdi, R.R. Ernst, J. Chem. Phys. 64, 2229 (1976)
K. Nagayama et al., J. Magn. Reson. 40, 321 (1980)

Related Experiments

Selective 1D COSY
2D COSY-DQF version
2D Relayed and TOCSY experiments



2D COSY Experiments

- **Phase-cycled:**

Magnitude-mode 2D COSY (**cosyqf** | COSY45SW / COSY90SW)
Magnitude-mode 2D COSY using a 45 pulse (**cosyqf45** | COSY45SW)
Magnitude-mode 2D COSY using a 90 pulse (**cosyqf90** | COSY90SW)
Magnitude-mode 2D COSY using purge pulses before d1 (**cosyppqf**)
Phase-sensitive 2D COSY (**cosyph**)

Magnitude-mode Long-Range optimized 2D COSY (**cosylrqf**)

Constant-Time 2D COSY (**cosyjdqf**)

- **Phase-cycled and solvent suppression:**

Magnitude-mode 2D COSY with presaturation (**cosyprqf**)
Phase-sensitive 2D COSY with presaturation (**cosyphpr** | COSYPHPR)

- **Gradient-based:**

Magnitude-mode ge-2D COSY (**cosygpqf** | COSYGPSW)
Magnitude-mode ge-2D COSY using presaturation (**cosygpprqf**)
Magnitude-mode ge-2D COSY using purge pulses before d1 (**cosygpppqf**)
Phase-sensitive ge-2D COSY using echo-antiecho (**cosyetgp**)
Phase-sensitive 2D z-COSY using ZQ suppression (**cosygpphzfzs**)

- **Gradient-based and solvent suppression:**

Magnitude-mode ge-2D COSY using presaturation (**cosygpprqf**)
2D COSY using WET (**cosydcphwt** | COSYDCPHWT)

- **Miscellaneous:**

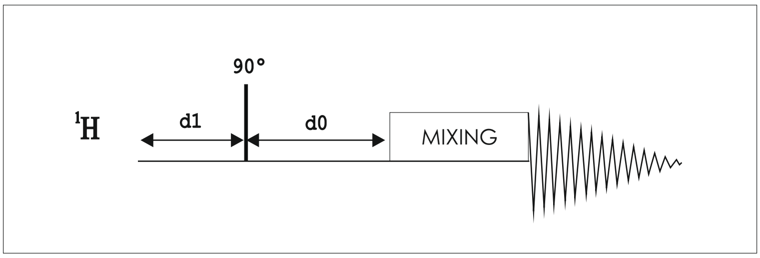
Phase-sensitive w_1-region-selective 2D COSY (**scosyph**)
Phase-sensitive w_1-region-selective 2D COSY with refocusing (**scosyphrd)**

Phase-sensitive 2D COSY with off-resonance single or multiple presaturation (**cosycwphps** | COSYCWPHPS)
Magnitude-mode ge-2D COSY with off-resonance single or multiple presaturation (**cosycwgppsqf**)

Magnitude-mode 2D ^{13}C-^{13}C COSY (**cosydcqf**)
Magnitude-mode long-range optimized 2D ^{13}C-^{13}C COSY (**cosydclrqf**)
Phase-sensitive 2D ^{13}C-^{13}C COSY **(cosydcph)**

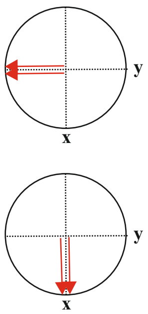
NMR Building Block: A Variable Evolution Period.

```
"in0=inf1"
"d0=3u"
...
  d0
...
  d1 mc #0 to 2 F1QF(caldel(d0, +in0))
...
;d0 : incremented delay (2D)       [3 usec]
;inf1: 1/SW = 2 * DW
;in0: 1/(1 * SW) = 2 * DW
;nd0: 1
```

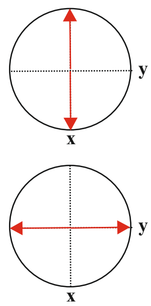


$$I_{1z} \xrightarrow{90^\circ I_x} -I_{1y} \xrightarrow{2\pi\omega t_1 I_z} -I_{1y}\cos\omega_1 t_1 + I_{1x}\,\mathrm{sen}\,\omega_1 t_1$$

$$\xrightarrow{2\pi J_{12} t_1 I_{1z} I_{2z}}$$



$$-I_{1y}\cos\omega_1 t_1 \cos\pi J_{12} t_1 + 2I_{1x}I_{2z}\cos\omega_1 t_1\,\mathrm{sen}\,\pi J_{12} t_1$$
$$+ I_{1x}\,\mathrm{sen}\,\omega_1 t_1 \cos\pi J_{12} t_1 + 2I_{1y}I_{2z}\,\mathrm{sen}\,\omega_1 t_1\,\mathrm{sen}\,\pi J_{12} t_1$$

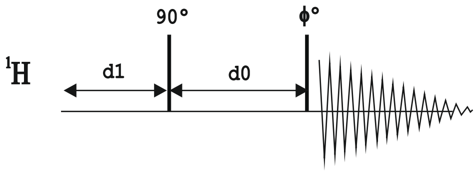


$$\downarrow 90^\circ I_x$$

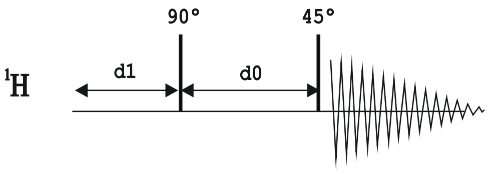
$-I_{1z}\cos\omega_1 t_1 \cos\pi J_{12} t_1$	see NOESY
$-2I_{1x}I_{2y}\cos\omega_1 t_1\,\mathrm{sen}\,\pi J_{12} t_1$	see Double-Quantum Filtered COSY
$+I_{1x}\,\mathrm{sen}\,\omega_1 t_1\cos\pi J_{12} t_1$	Autocorrelated (Diagonal) COSY
$-2I_{1z}I_{2y}\,\mathrm{sen}\,\omega_1 t_1\,\mathrm{sen}\,\pi J_{12} t_1$	COSY cross-peak

Phase-Cycled COSY

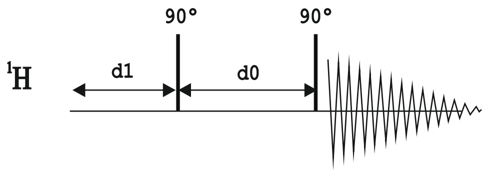
cosyph
cosyqf



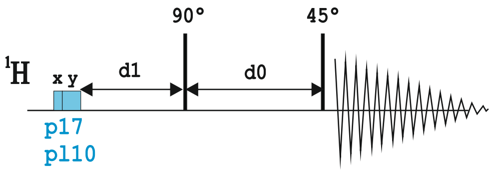
cosyqf45



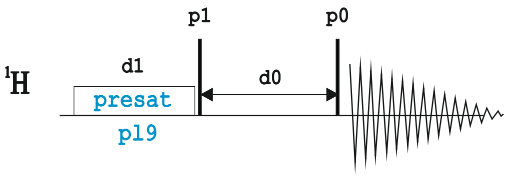
cosyqf90



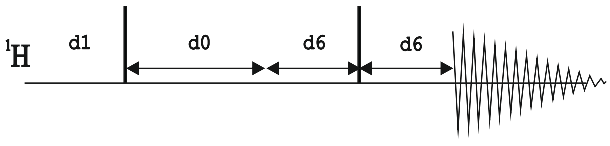
cosyppqf



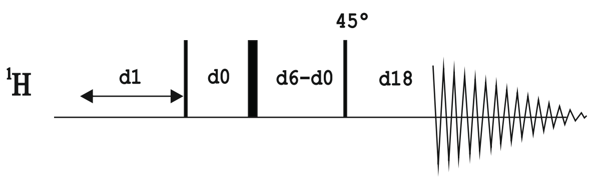
cosyphpr
cosyprqf



cosylrqf

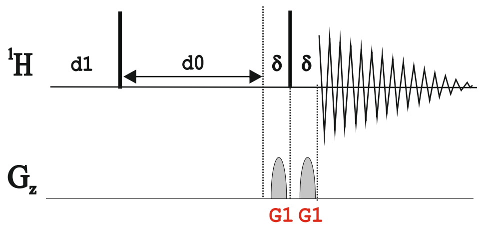


cosyjdqf

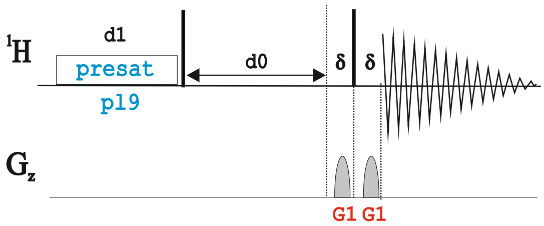


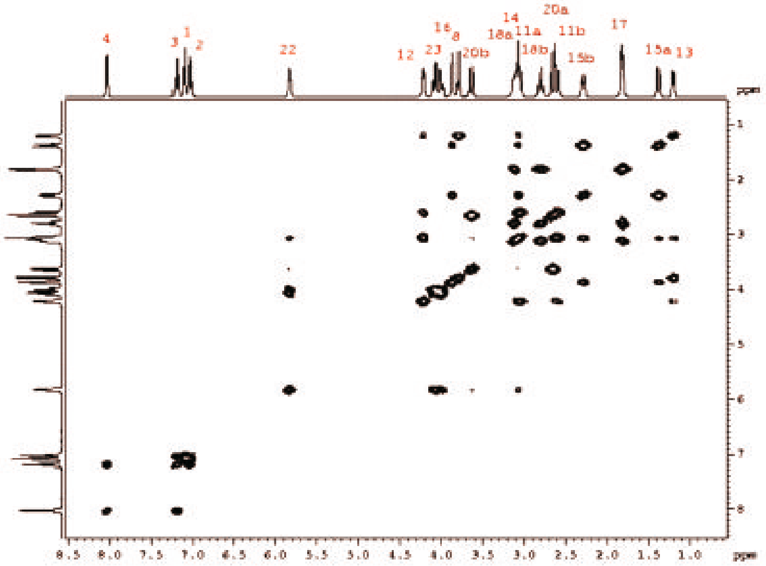
COSY with Gradient Selection

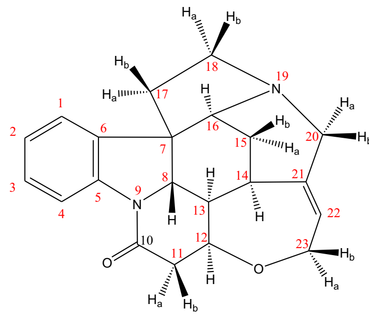
cosygpqf



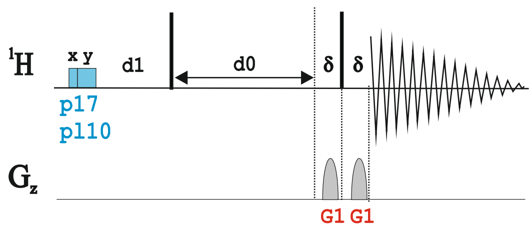
cosygpprqf



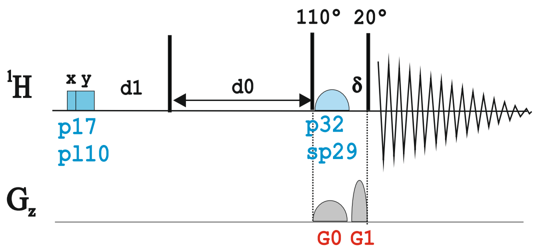




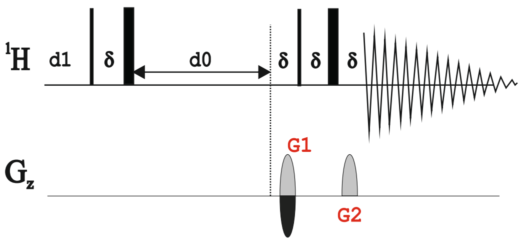
cosygpppqf



cosygpphzfzs

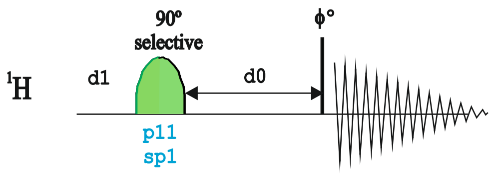


cosyetgp

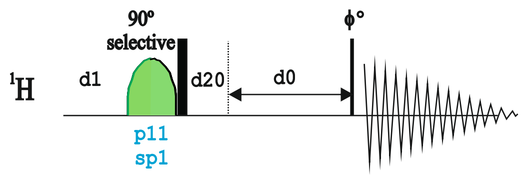


Region-Selective COSY

scosyph

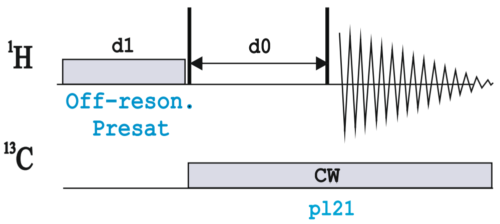


scosyphrd

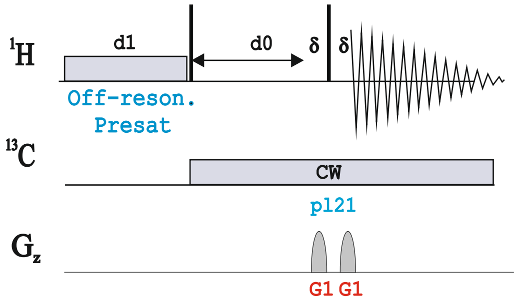


X-Decoupled COSY

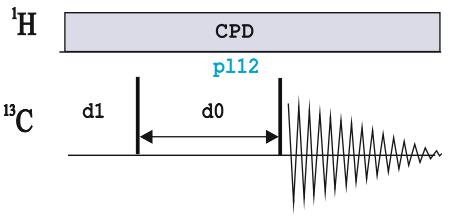
cosycwphps



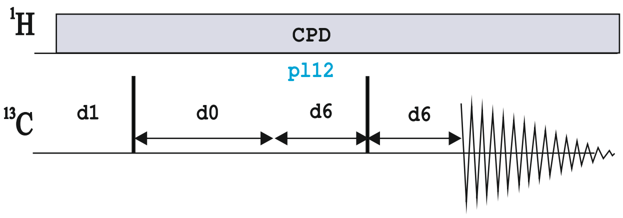
cosycwgppsqf



cosydcqf
cosydcph

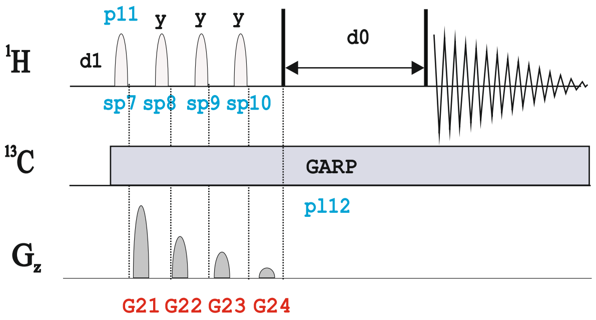


cosydclrqf

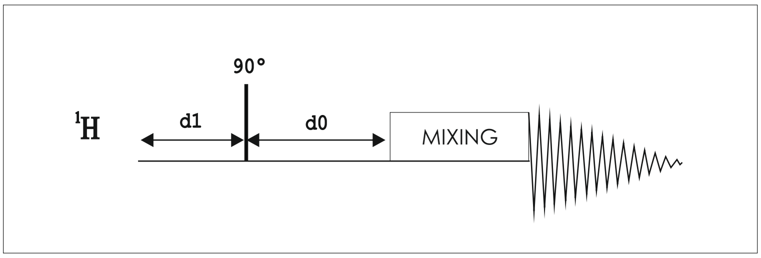


COSY with WET Solvent Suppression

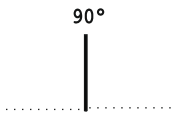
cosydcphwt



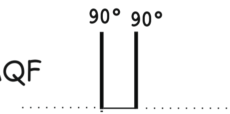
Homonuclear Mixing Times



COSY

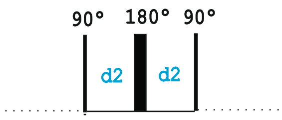


COSY-MQF



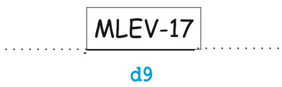
Also with Gradients

RELAY



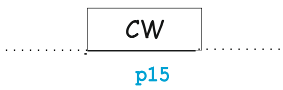
Also with Multiple Relay Steps

TOCSY



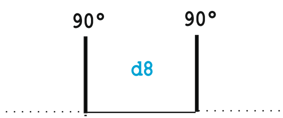
Also with DIPSI-2 ...

ROESY



Also with T-ROESY ..

NOESY



Also with Purge Gradients ....

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2D COSY-DQF EXPERIMENTS

Experiment Description

A COSY-MQF experiment is a modified COSY pulse scheme that incorporates a multiple-quantum filter just prior acquisition.
It provides through-bond proton-proton connectivities by means of scalar J coupling constants

Sample Requirements

COSY-DQF experiments can be recorded on any type of sample.
Solvent-supressed versions are required for samples dissolved in H2O.

Hardware Requirements

In principle, COSY-DQF experiments can be recorded on any probehead.
An optional pulsed-field gradient coil is required for gradient-based versions.

NMR Spectrum

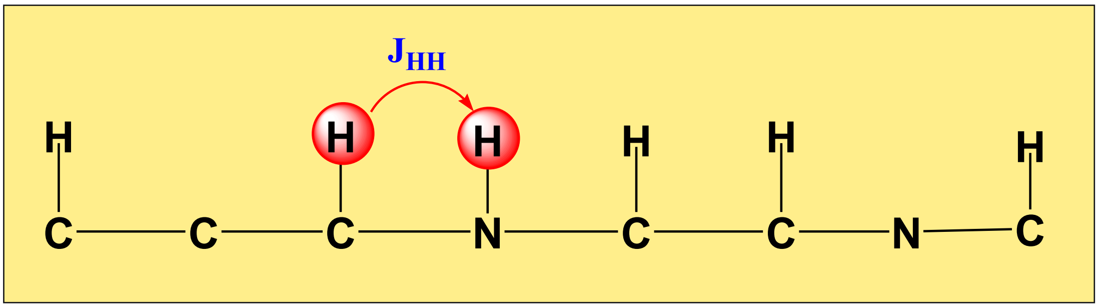
The experiment yields a 2D proton-proton correlation map with two different types of signals: i) autocorrelation diagonal peaks, and ii) Off-diagonal cross-peaks correlating J-coupled spins. Although COSY-DQF can be represented in magnitude-mode, phase-sensitive data is most suitable for analyzing the anti-phase character of cross-peaks.

Related Experiments

Selective 1D COSY
2D COSY experiment
2D Relayed and TOCSY experiments

References:

1. A.A. Shaw, C. Salaun, J.-F. Dauphin & B. Ancian, J. Magn. Reson. A 120, 110-115 (1996)
2. B. Ancian, I. Bourgeois, J.-F. Dauphin & A.A. Shaw, J. Magn. Reson. A 125, 348- 354 (1997)



2D COSY-MQF Experiments

- **Phase-cycled:**

  Magnitude-mode 2D COSY with DQF (**cosydfqf**)
  Magnitude-mode 2D COSY with TQF (**cosyqftf**)
  Phase-sensitive 2D COSY with DQF (**cosydfph** | COSYDQFPHSW)
  Phase-sensitive 2D COSY with DQF and purge pulse (**cosydfphpp**)
  Phase-sensitive 2D COSY with TQF (**cosyphtf**)

  Phase sensitive 2D E.COSY -KcMAX=3 (**ecos3nph**)
  Complementary Phase sensitive 2D E.COSY - KcMAX=3 (**ecos3cph**)

- **Phase-cycled and solvent suppression:**

  Phase-sensitive 2D COSY with DQF & presaturation (**cosydfphpr**)

- **Gradient-based:**

  Magnitude-mode ge-2D COSY with multiple-quantum filter (**cosygpmfqf** | COSYGPMFSW)
  Magnitude-mode ge-2D COSY with multiple-quantum filter and purge pulse (**cosygpmfppqf**)
  Phase-sensitive ge-2D COSY with multiple-quantum filter (**cosygpmfph** | COSYGPDFPHSW) Phase-sensitive ge-2D COSY with multiple-quantum filter and purge pulse (**cosygpmfphpp**)
  Phase-sensitive ge-2D COSY with DQF using echo-antiecho (**cosydfetgp.1**)
  Phase-sensitive ge-2D COSY with gradient-based DQF using echo-antiecho (**cosydfetgp.2**)
  Phase-sensitive ge-2D COSY with gradient-based DQF using echo-antiecho and purge pulse (**cosydfetgppp.2**)

  Gradient E.COSY (**ecosygpph**)

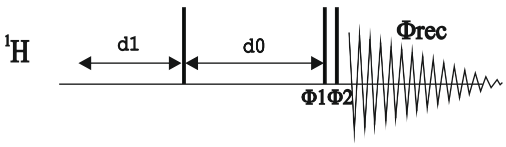
- **Gradient-based and solvent supression:**

  Phase-sensitive 2D COSY-DQF with WATERGATE using 3-9-19 (**cosydfgpph19**)
  Phase-sensitive 2D COSY-DQF with Excitation Sculpting using 180 water-selective pulse (ES element) (**cosydfesgpph**)
  Using purge pulses before d1 (**cosydfesgpphpp**)

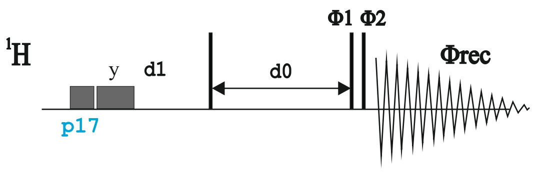
Also See:
2D COSY Experiments

Phase-Cycled COSY-QMF

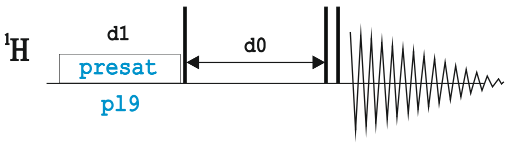
cosydfph cosyphtf ecos3cph
cosydfqf cosyqftf ecos3nph

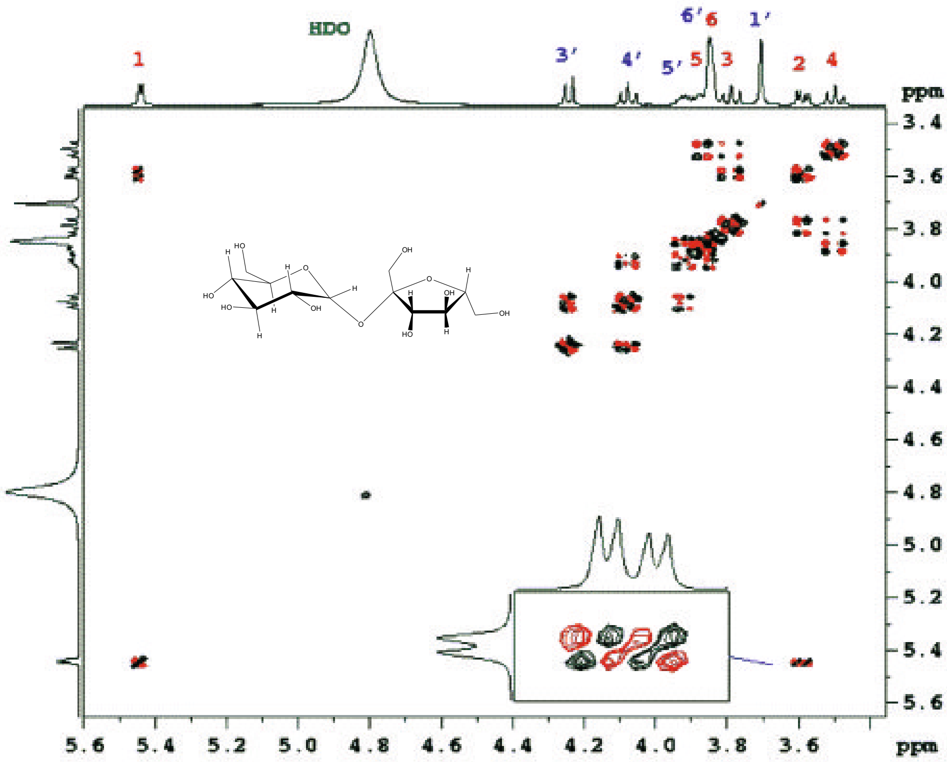


cosydfphpp



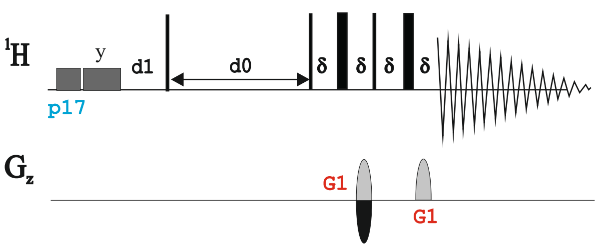
cosydfphpr



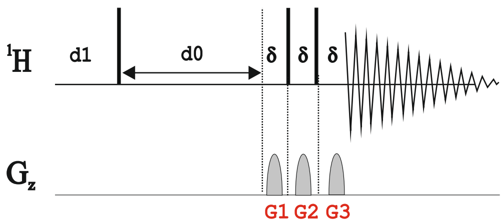


Gradient-Enhanced COSY-QMF

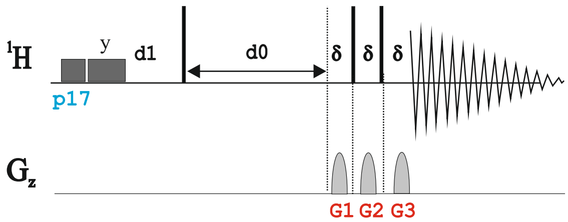
ecosygpph



cosygpmfqf

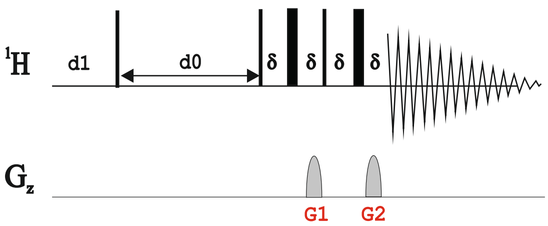


cosygpmfppqf

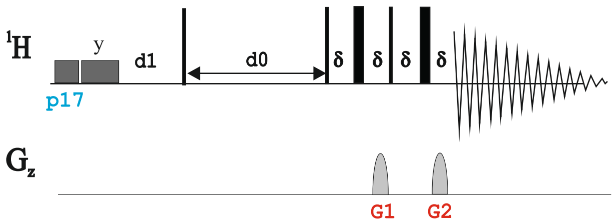


16:12:40 for DQ filter
4:12:40 for TQ filter

cosygpmfph

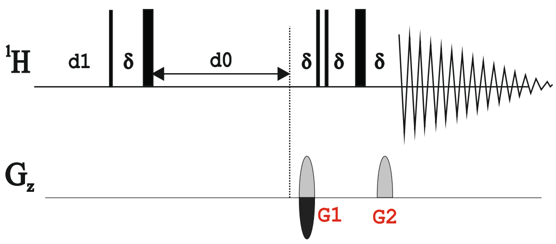


cosygpmfphpp

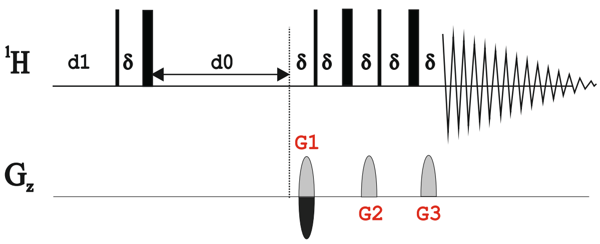


10:20 for DQ filter
10:30 for TQ filter

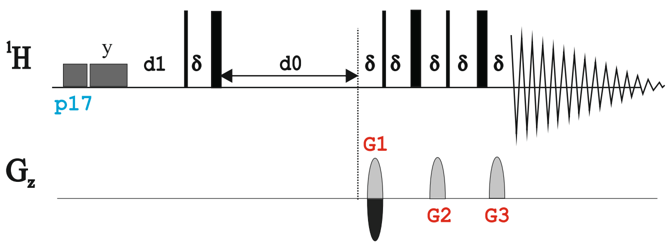
cosydfetgp.1



cosydfetgp.2

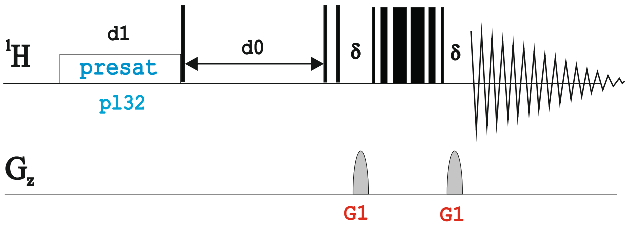


cosydfetgppp.2

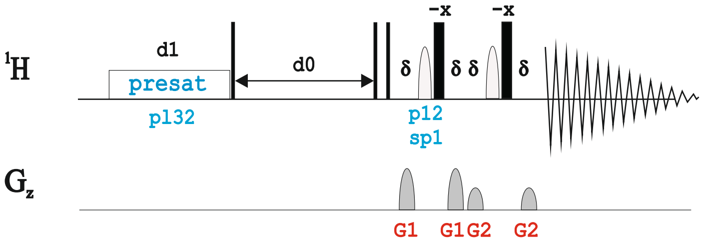


COSY-QMF with Gradient Solvent Suppression

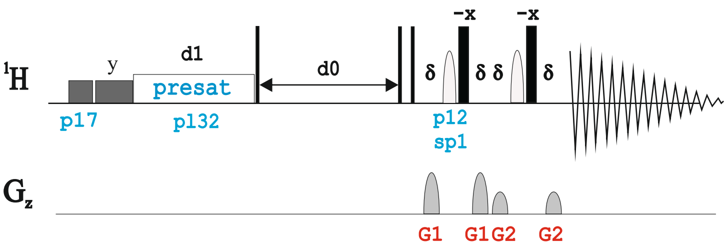
cosydfgpph19



cosydfesgpph



cosydfesgpphpp



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2D SECSY EXPERIMENTS

Experiment Description

A SECSY (Spin-Echo Correlated SpectroscopY) experiment is actually an obsolete tool that provides through-bond proton-proton connectivities by means of scalar J coupling constants in the same way as described for COSY experiment.

Sample Requirements

SECSY experiments can be recorded on any type of sample.

Hardware Requirements

SECSY experiments can be recorded on any probehead.

NMR Spectrum

The experiment yields a 2D correlation map in which the conventional diagonal axis is rotated to F1=0. J Cross-peaks are correlated about an axis of 135° relative to F1=0

Related Experiments

See 2D COSY experiments

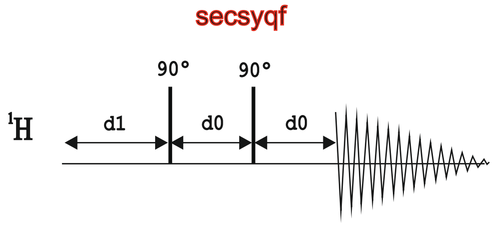
References:

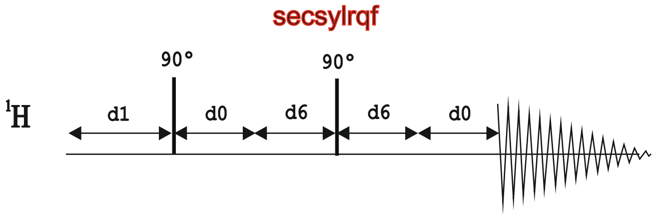
K. Nagayama et al., J. Magn. Reson. 40, 321 (1980)

2D SECSY Experiments

Magnitude-mode 2D SECSY (**secsyqf**)
Magnitude-mode long-range optimized 2D SECSY (**secsylrqf**)

Also See:
2D COSY and 2D COSY-DQF Experiments





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2D RELAY EXPERIMENTS

Experiment Description

RELAY experiments are extensions of the conventional COSY experiment and provide through-bond proton-proton connectivities by means of scalar J coupling constant in a step-by-step way. Thus, one-step, two-step, or three-step RELAY versions are available for the analysis of individual subspin systems into a molecular framework.

Sample Requirements

RELAY experiments can be recorded on any type of sample.

Hardware Requirements

RELAY experiments can be recorded on any probehead.

NMR Spectrum

The experiment yields a 2D proton-proton correlation map with different types of signals: i) autocorrelation diagonal peaks, ii) Off-diagonal cross-peaks correlating directly J-coupled spins (COSY peaks), and iii) relayed off-diagonal cross-peaks correlating spins belonging to the same spin system but not J-coupled.

Related Experiments

Selective 1D RELAY
2D COSY and 2D TOCSY experiment

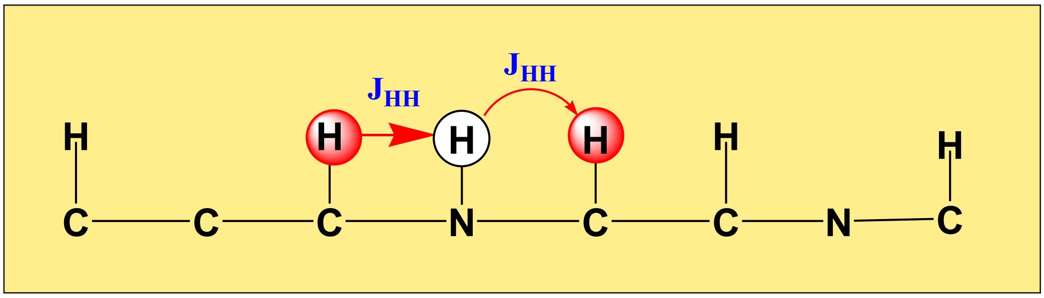
References:

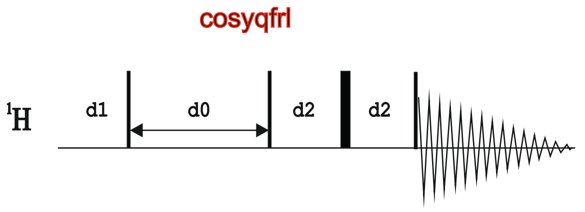
1. G. Wagner, J. Magn. Reson. 55, 151 (1983)
2. A. Bax & G. Drobny, J. Magn. Reson. 61, 306 (1985)

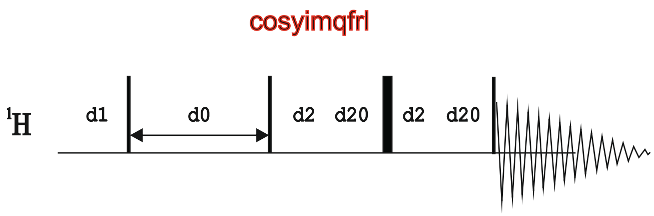
2D RELAY Experiments

Magnitude-mode one-step 2D RELAY (**cosyqfr1**)
Magnitude-mode one-step 2D RELAY with incremented mixing times (**cosyimqfr1**)
Magnitude-mode two-step 2D RELAY (**cosyqfr2**)
Magnitude-mode two-step 2D RELAY with incremented mixing times (**cosyimqfr2**) Magnitude-mode three-step 2D RELAY (**cosyqfr3**)

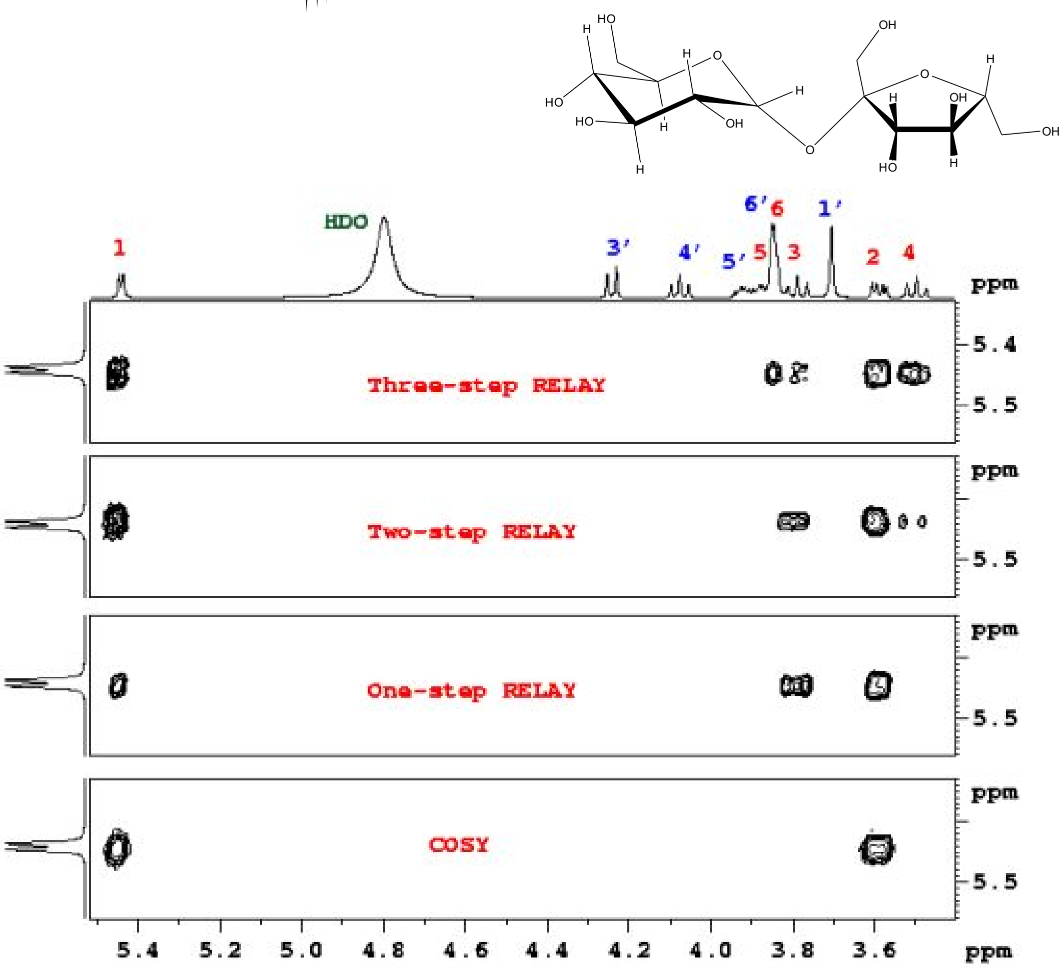
Also See:
2D COSY and 2D COSY-DQF Experiments
2D TOCSY Experiment

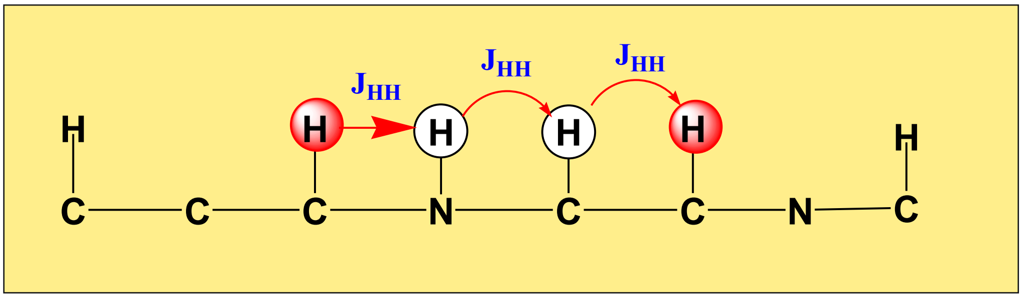




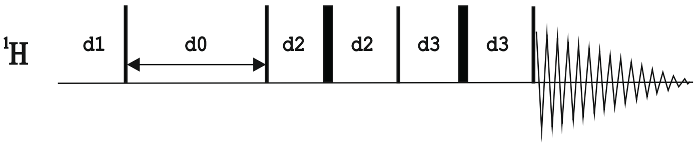


The delays d2, d3, and d4 define the one-step, two-step, and three-step RELAY periods. They are optimized to 1/4J(HH)

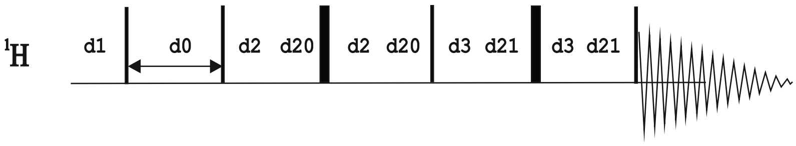


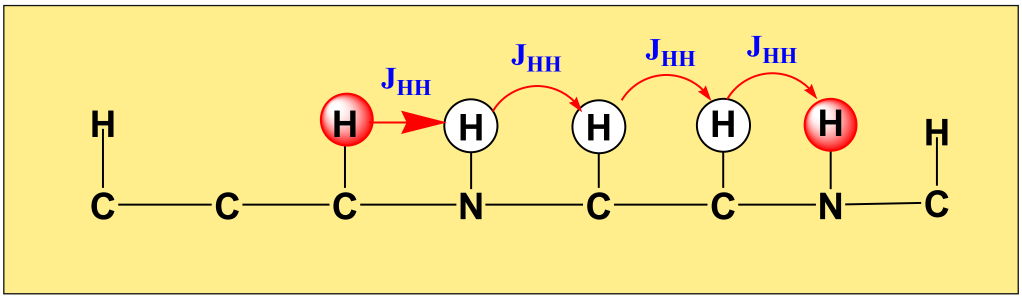


cosyqfr2

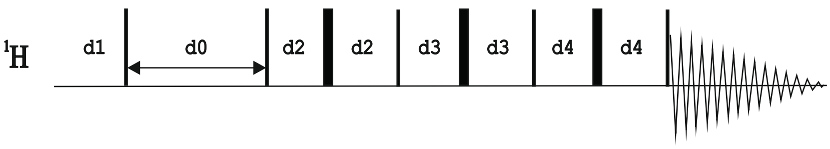


cosyimqfr2





cosyqfr3



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2D TOCSY EXPERIMENTS

Experiment Description

A TOCSY (TOtal Crrelation SpectroscopY) experiment is a high-sensitive tool that allows the analysis of individual J-coupled spin systems.

Sample Requirements

TOCSY experiments can be recorded on any type of sample.
Solvent-supressed versions are required for samples dissolved in H2O.

Hardware Requirements

TOCSY experiments can be recorded on any probehead.
Optionally, a pulsed-field gradient coil (highly recommended) is required for gradient-based versions.

NMR Spectrum

The experiment yields a 2D proton-proton correlation map with two different types of signals: i) autocorrelation diagonal peaks, and ii) Off-diagonal cross-peaks correlating spins belonging to the same spin system. TOCSY spectra are usually represented in phase-sensitiv mode.

Related Experiments

Selective 1D TOCSY
2D COSY experiment
2D Relayed experiments
2D HMQC-TOCSY and HSQC-TOCSY experiments

References:

1. A. Bax & D.G. Davis, J. Magn. Reson. 65, 355-360 (1985)
2. J. Cavanagh & M. Rance, J. Magn. Reson. 88, 72-85 (1990)

The delay d9 defines the mixing period in all TOCSY experiments, independently if DIPSI-2 or MLEV are used. Typical values range from 0 to 120 ms and transfer efficiency depends on spin system topologies.

2D TOCSY Experiments

- **Phase-cycled**

Phase-sensitive 2D TOCSY using MLEV (**mlevph** | MLEVPHSW)
Phase-sensitive 2D TOCSY using MLEV with purge pulses before d1 (**mlevphpp**)
Phase-sensitive 2D TOCSY using DIPSI-2 (**dipsi2ph**)

- **Phase-cycled and solvent suppression**

Phase-sensitive 2D TOCSY with presaturation using MLEV (**mlevphpr** | MLEVPHPR)
Phase-sensitive 2D TOCSY with presaturation using MLEV only using first trim pulse (**mlevphpr.2** | H2OSUPMLEV)
Phase-sensitive 2D TOCSY with presaturation using MLEV and spoil gradient (**mlevgpphprzs**)
Phase-sensitive 2D TOCSY with presaturation using DIPSI-2 (**dipsi2phpr**)
Phase-sensitive 2D TOCSY with presaturation using DIPSI-2 and spoil gradient (**dipsi2gpphpr**)
Phase-sensitive 2D Clean-TOCSY with presaturation using MLEV (**clmlevphpr**)

- **Gradient-based**

Phase-sensitive ge-2D TOCSY with MLEV using echo-antiecho (**mlevetgp**)
Phase-sensitive ge-2D TOCSY with DIPSI-2 using echo-antiecho (**dipsi2etgp**)
Phase-sensitive ge-2D TOCSY with DIPSI-2 using PEP (**dipsi2etgpsi**)
Phase-sensitive ge-2D TOCSY with DIPSI-2 and Zero-Quantum suppression (**dipsi2gpphzs**)

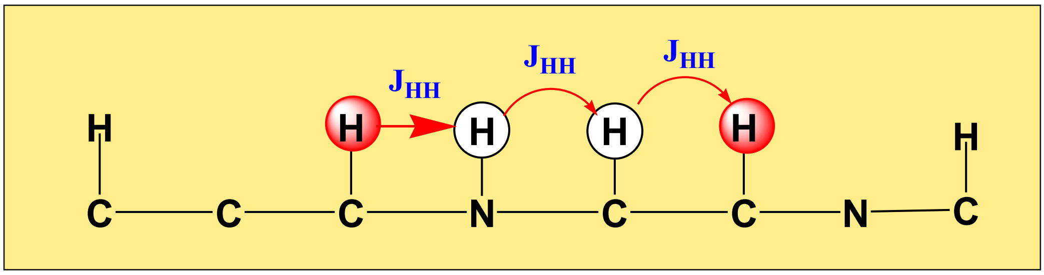
- **Gradient-based and solvent suppression**

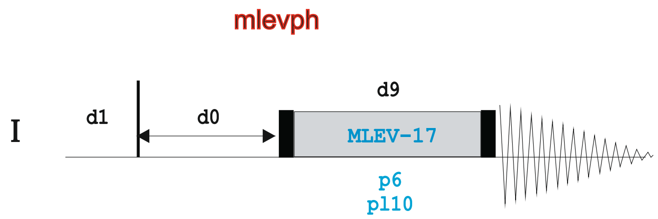
Phase-sensitive 2D TOCSY with WATERGATE (3-9-19) using MLEV (**mlevgpph19** | MLEVGPPH19SW)
Phase-sensitive 2D TOCSY with WATERGATE (3-9-19) using DIPSI-2 (**dipsi2gpph19**)
Phase-sensitive sensitivity-improved 2D TOCSY with WATERGATE (3-9-19) and using DIPSI-2 (**dipsi2etgpsi19**)
Phase-sensitive 2D Adiabatic TOCSY with WATERGATE (3-9-19) using X_M16 sequence (**atocsygpph19**)

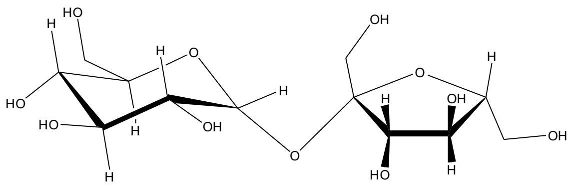
Phase-sensitive 2D TOCSY with excitation sculpting (W5) using MLEV (**mlevgpphw5**)
Phase-sensitive 2D TOCSY with excitation sculpting (180 water-selective pulse-ES element) using MLEV (**mlevesgpph**)
Phase-sensitive 2D TOCSY with excitation sculpting (180 water-selective pulse-ES element) using DIPSI-2 (**dipsi2esgpph**)
Phase-sensitive 2D TOCSY with excitation sculpting (180 water-selective pulse-ES element) using DIPSI-2 and optional 13C,15N-decoupling (**dipsi2esfbgpph**)

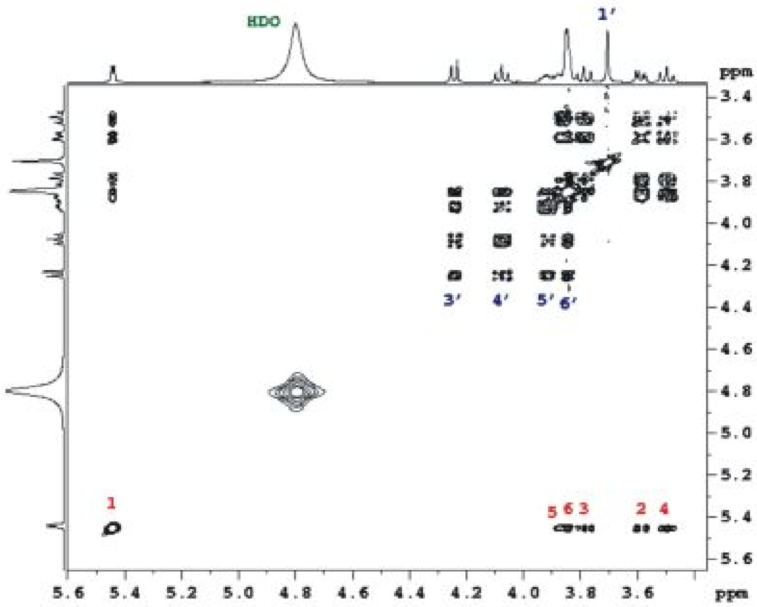
- **Single/Multiple Solvent Suppression (LC-NMR):**

2D TOCSY with double presaturation and cw-decoupling on f2 (**lcmlevcwfdpcph**)
2D TOCSY with presaturation and cw-decoupling on f2 (**lcmlevcwpcphps**)
2D TOCSY with double presaturation using composite pulse (**lcmlevf2pcph**)
2D TOCSY with double presaturation (**lcmlevf2phpr** | LCML12)
2D TOCSY with presaturation using shape pulse and composite pulse (**lcmlevpcphps**)
2D TOCSY with presaturation using composite pulse (**lcmlevpcph**)
2D TOCSY using WET (**mlevdcphwt** | MLEVDCPHWT)





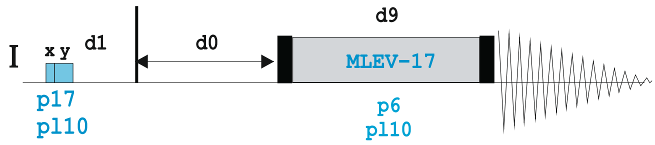




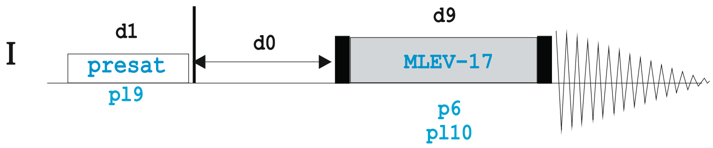
2D TOCSY spectrum of sucrose (mixing time d9 of 90ms). Note that the glucose (red) and fructose (blue) subresidues are clearly identified.

Phase-Cycled TOCSY

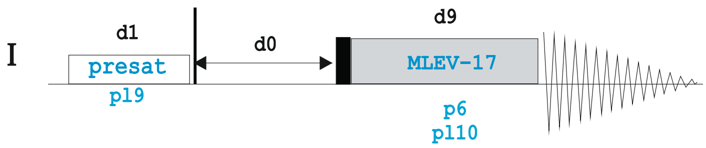
mlevphpp



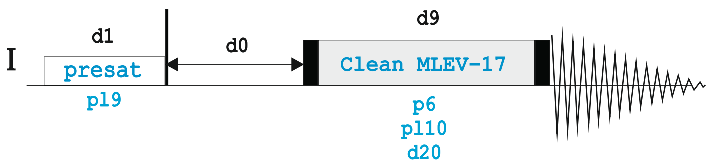
mlevphpr



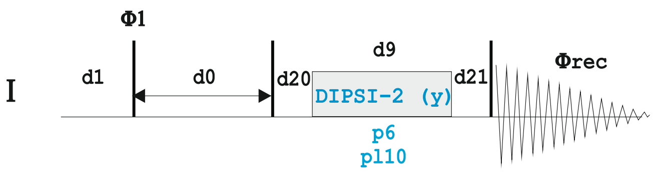
mlevphpr.2



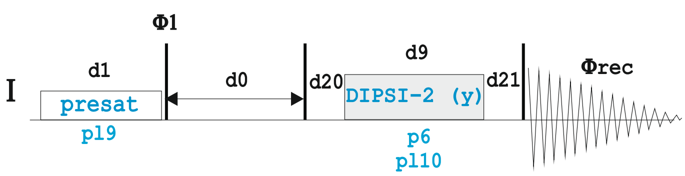
clmlevphpr



dipsi2ph



dipsi2phpr



NMR BUILDING BLOCK: MLEV-17

```
"p5=p6*.667"
"p7=p6*2"

"SCALEF=p7*2/p5"
"FACTOR1=((d9-p17*2)/(p6*64+p5))/SCALEF"
"l1=FACTOR1*SCALEF"
.............

4u pl10:f1
  (p17 ph26)
;begin MLEV17
4 (p6 ph22 p7 ph23 p6 ph22)
  (p6 ph24 p7 ph25 p6 ph24)
  (p6 ph24 p7 ph25 p6 ph24)
  (p6 ph22 p7 ph23 p6 ph22)
  (p6 ph24 p7 ph25 p6 ph24)
  (p6 ph24 p7 ph25 p6 ph24)
  (p6 ph22 p7 ph23 p6 ph22)
  (p6 ph22 p7 ph23 p6 ph22)
  (p6 ph24 p7 ph25 p6 ph24)
  (p6 ph22 p7 ph23 p6 ph22)
  (p6 ph22 p7 ph23 p6 ph22)
  (p6 ph24 p7 ph25 p6 ph24)
  (p6 ph22 p7 ph23 p6 ph22)
  (p6 ph22 p7 ph23 p6 ph22)
  (p6 ph24 p7 ph25 p6 ph24)
  (p6 ph24 p7 ph25 p6 ph24)
  (p5 ph23)
  lo to 4 times l1
;end MLEV17
  (p17 ph26)

......
ph22=3
ph23=0
ph24=1
ph25=2
ph26=0

;pl10: f1 channel - power level for TOCSY-spinlock
;p5 : f1 channel -  60 degree low power pulse
;p6 : f1 channel -  90 degree low power pulse
;p7 : f1 channel - 180 degree low power pulse
;p17: f1 channel - trim pulse                        [2.5 msec]
;d1 : relaxation delay; 1-5 * T1
;d9 : TOCSY mixing time
;l1: loop for MLEV cycle: (((p6*64) + p5) * l1) + (p17*2) = mixing time
```

The Basic Elementof the MLEV (acronym of **M**alcom **Lev**itt´s CPD sequence)
is the composite R=90(x)-180(y)-90(x) cluster.
The 16 elements in the MLEV-16 supercycle are cycled as RR**RR** **R**RR**R** **RR**RR R**RR**R
where **R** is the inverse of R.

Reference:

MH Levitt, R. Freeman, T. Frenkiel, J. Magn. Reson. , 47, 328-330 (1982).

NMR BUILDING BLOCK: z-filtered DIPSI-2

```
"FACTOR1=(d9/(p6*115.112))/2"
"l1=FACTOR1*2"

p1 ph2
  d20 pl10:f1

;begin DIPSI2
4 p6*3.556 ph23
  p6*4.556 ph25
  p6*3.222 ph23
  p6*3.167 ph25
  p6*0.333 ph23
  p6*2.722 ph25
  p6*4.167 ph23
  p6*2.944 ph25
  p6*4.111 ph23

  p6*3.556 ph25
  p6*4.556 ph23
  p6*3.222 ph25
  p6*3.167 ph23
  p6*0.333 ph25
  p6*2.722 ph23
  p6*4.167 ph25
  p6*2.944 ph23
  p6*4.111 ph25

  p6*3.556 ph25
  p6*4.556 ph23
  p6*3.222 ph25
  p6*3.167 ph23
  p6*0.333 ph25
  p6*2.722 ph23
  p6*4.167 ph25
  p6*2.944 ph23
  p6*4.111 ph25

  p6*3.556 ph23       ;p6 : f1 channel -  90 degree low power pulse
  p6*4.556 ph25       ;d0 : incremented delay (2D)
  p6*3.222 ph23       ;d1 : relaxation delay; 1-5 * T1
  p6*3.167 ph25       ;d9 : TOCSY mixing time
  p6*0.333 ph23       ;d12: delay for power switching                [20 usec]
  p6*2.722 ph25       ;d20: first z-filter delay                     [2 msec]
  p6*4.167 ph23       ;d21: second z-filter delay                    [3 msec]
  p6*2.944 ph25       ;l1: loop for DIPSI cycle: ((p6*115.112) * l1) = mixing time
  p6*4.111 ph23
  lo to 4 times l1
;end DIPSI2

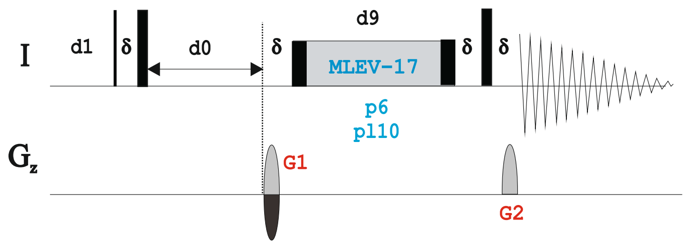
  d21 pl1:f1
  p1 ph3

 ph23=3
 ph25=1
```

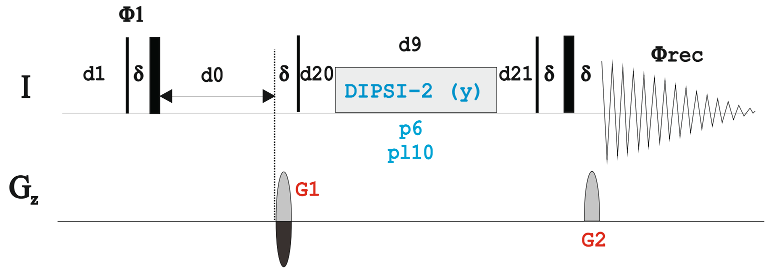
DIPSI = Decoupling In the Presence of Scalar Interactions
R Element= 320(x), 410(-x), 290(x), 285(-x), 30(x), 245(-x), 375(x), 265(-x), 370(x)
Ref: AJ Shaka, CJ Lee, A. Pines, J. Magn. Reson. 77, 274-293 (1988).

Gradient-based TOCSY using echo/antiecho

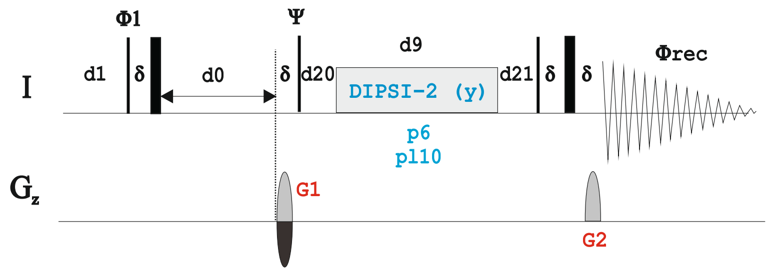
mlevetgp



dipsi2etgp

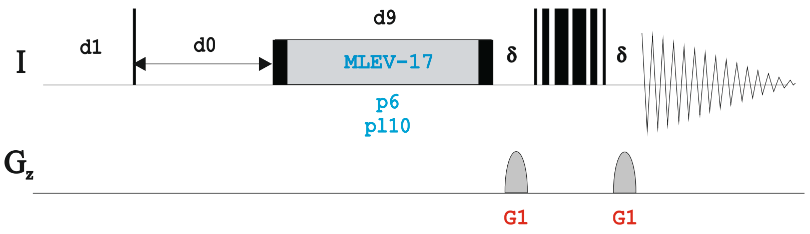


dipsi2etgpsi

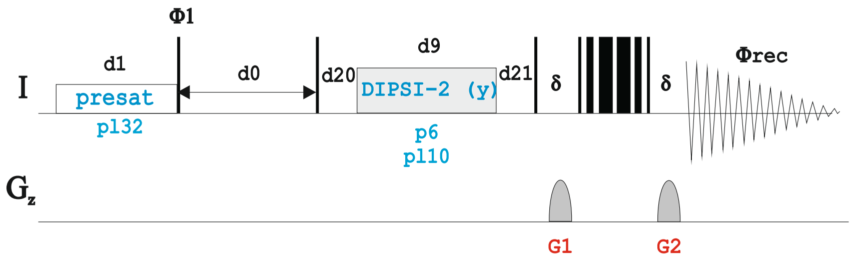


TOCSY using WATERGATE

mlevgpph19



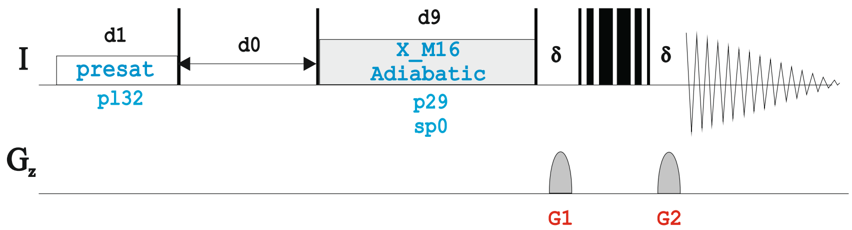
dipsi2gpph19



Adiabatic TOCSY:

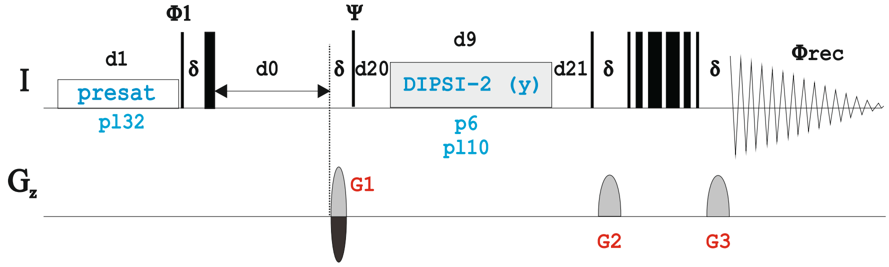
1. ;E. Kupce, P. Schmidt, M. Rance & G. Wagner, J. Magn. Reson. 135, 361-367 (1998)
2. W.Peti, C. Griesinger & W. Bermel, J. Biomol. NMR 18, 199 - 205 (2000)

atocsygpph19



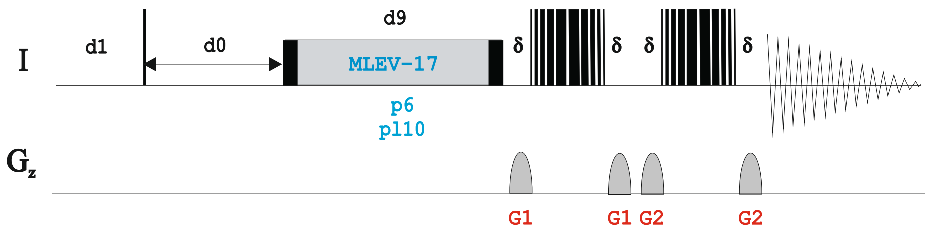
TOCSY using WATERGATE and echo-antiecho

dipsi2etgpsi19

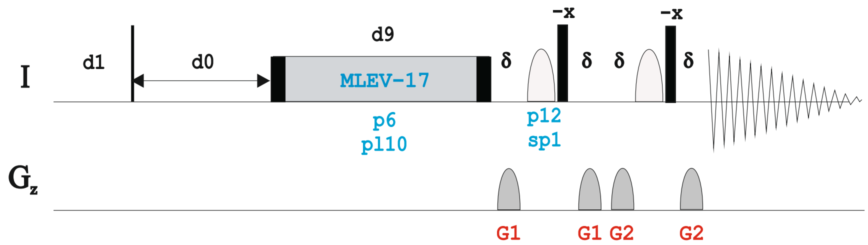


TOCSY using excitation scultping solvent suppresion

mlevgpphw5



mlevesgpph



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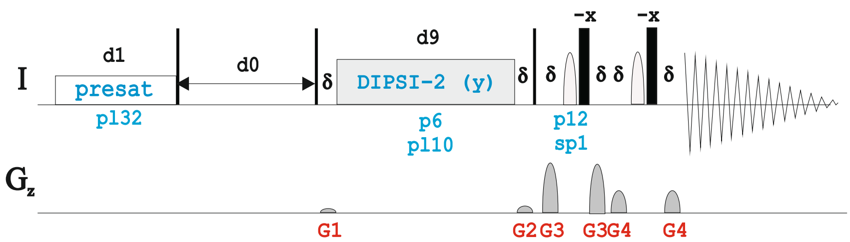
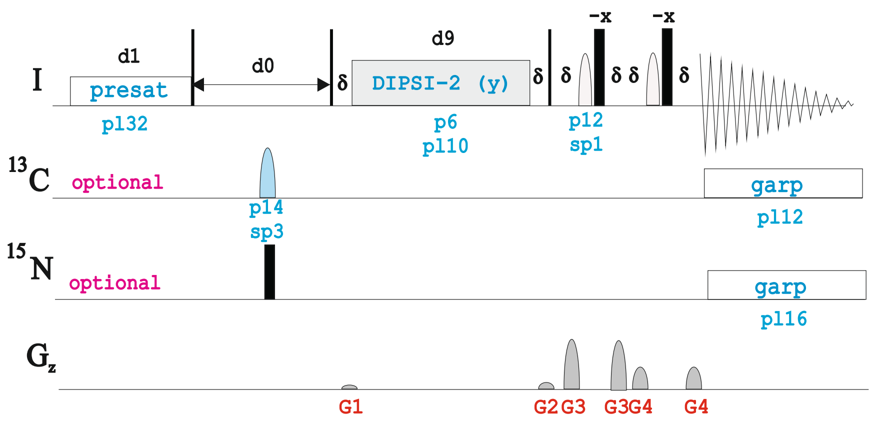
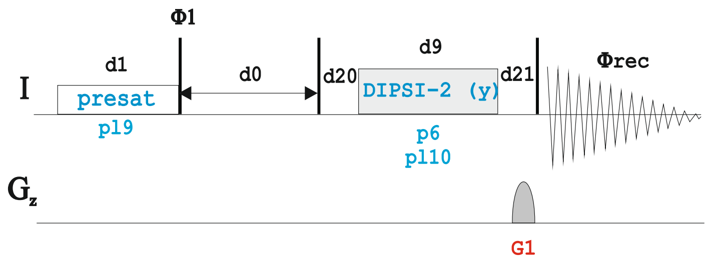


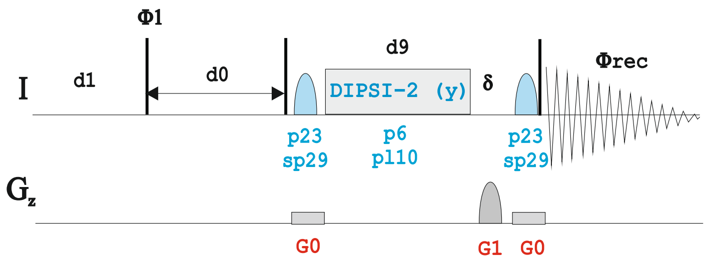
dipsi2esfbgpph



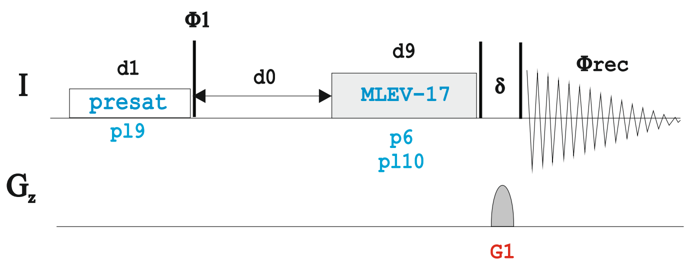
dipsi2gpphpr



dipsi2gpphzs

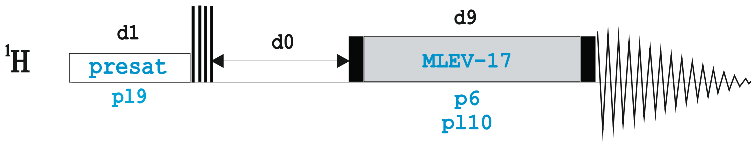


mlevgpphprzs

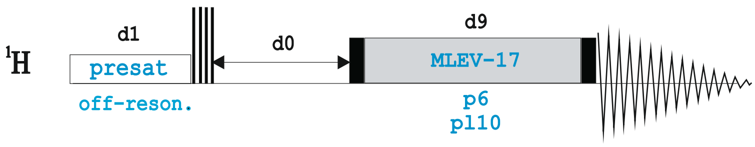


TOCSY using singlemultiple solvent suppression (LC-NMR applications)

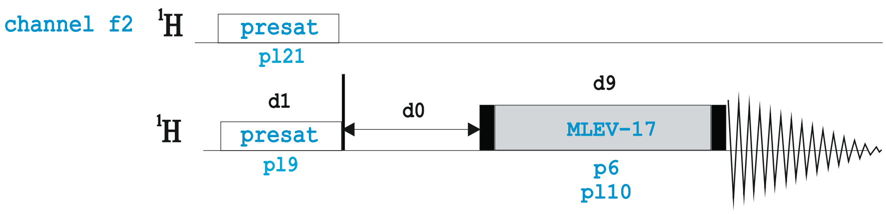
lcmlevpcph



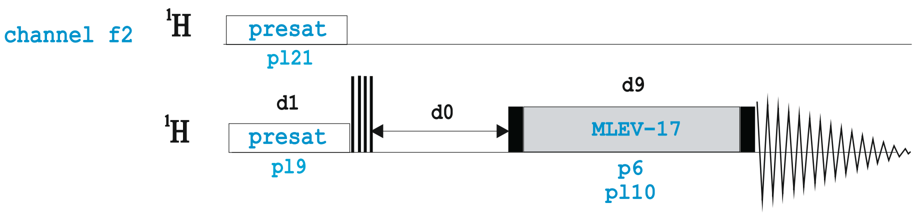
lcmlevpcphps



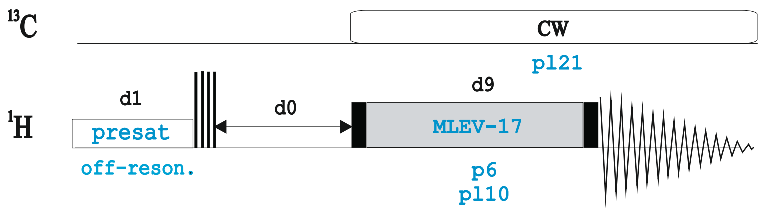
lcmlevf2phpr



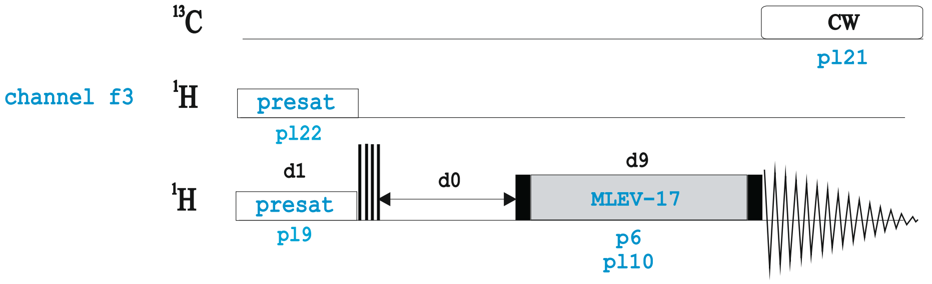
lcmlevf2pcph



lcmlevcwpcphps

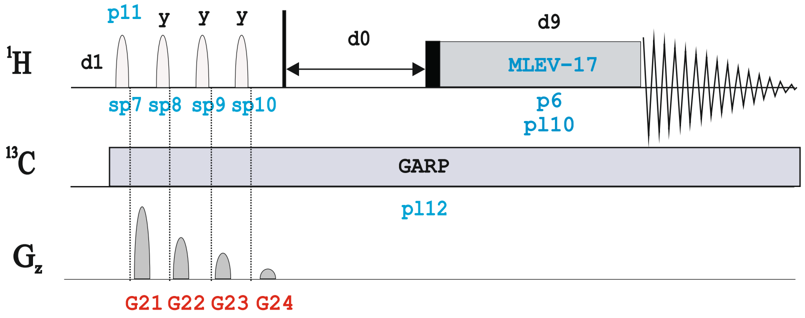


lcmlevcwfdpcph

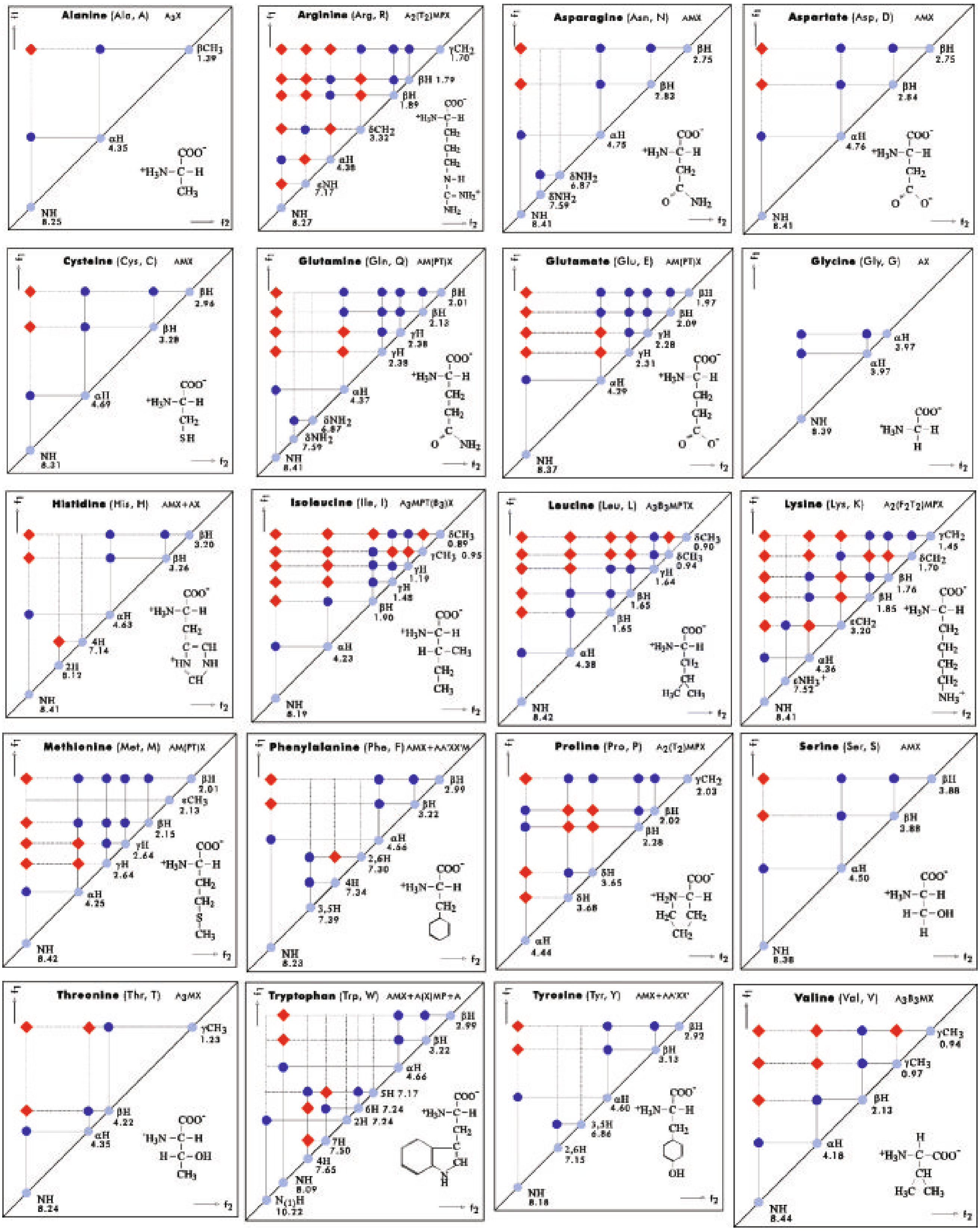


TOCSY using WET solvent suppression

mlevdcphwt



20 AMINOACIDS: 20 DIFFERENT TOCSY PATTERNS



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NMRGuide

2D NOESY EXPERIMENTS

Experiment Description

A NOESY (Nuclear Overhauser SpectroscopY) experiment provides through-space proton-proton connectivities.

EXSY (Exchange SpectroscopY) experiments use the same pulse sequences.

Sample Requirements

NOESY experiments can be recorded on any type of sample. Solvent-supressed versions are required for samples dissolved in H2O.

Hardware Requirements

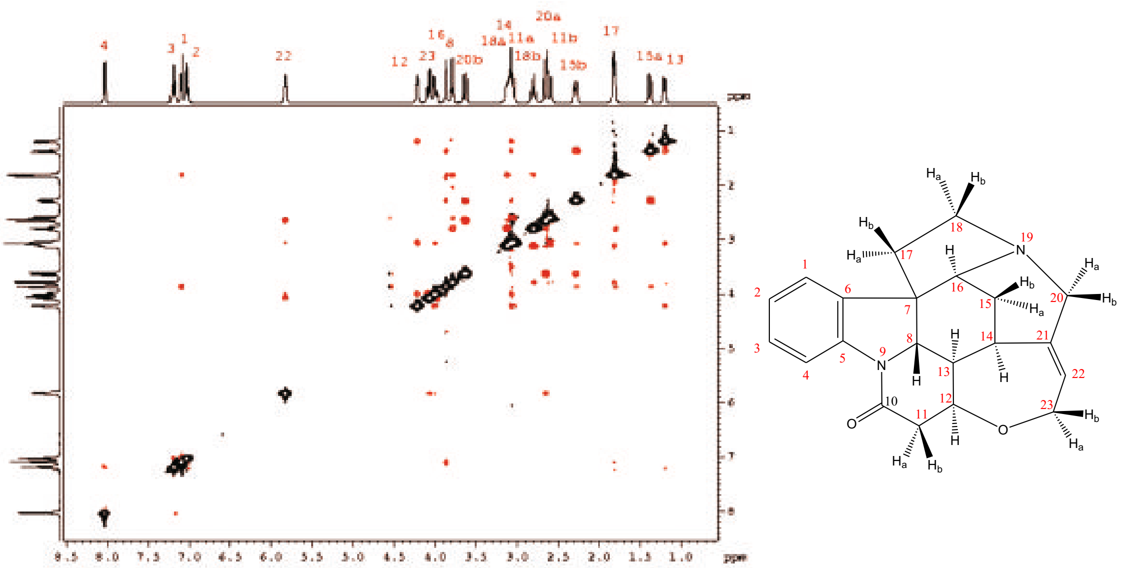
NOESY experiments can be recorded on any probehead. A pulsed-field gradient coil (highly recommended) is required for gradient-based versions.

NMR Spectrum

The NOESY experiment yields a 2D proton-proton correlation map with two different types of signals: i) autocorrelation diagonal peaks, and ii) Off-diagonal cross-peaks correlating spins close each other. NOESY spectra are represented in phase-sensitive mode in order to distinguish exchange cross-peaks and unwanted anti-phase COSY contributions.

Related Experiments

Selective 1D NOESY
2D ROESY experiment
2D HMQC-NOESY and HSQC-NOESY experiments



2D NOESY spectrum of strychnine. In small molecules, NOE cross-peaks have opposite phase with respect to diagonal autocorrelation peaks.

2D NOESY Experiments

- **1D Version:**

  1D NOESY **(noesy1d)**

  1D NOESY with presaturation during d1 and mixing time **(noesypr1d)**
  1D NOESY with presaturation during d1 and mixing time and spoil gradients **(noesygppr1d)**
  1D NOESY with presaturation during d1 and spoil gradients **(noesygppr1d.2)**

  1D NOESY with X-decoupling during acquisition **(noesyig1d)**

  Also see in "LC-NMR Experiments":
  1D NOESY experiments with single/multiple presaturation

- **Phase-cycled:**

  Phase-sensitive 2D NOESY (**noesyph** | NOESYPHSW)
  Phase-sensitive 2D NOESY using purgue pulses before d1 (**noesyphpp**)
  Phase-sensitive 2D NOESY using random mixing time (**noesyphrv**)

- **Phase-cycled and solvent suppression:**

  Phase-sensitive 2D NOESY with presaturation (**noesyphpr** | H2OSUPNOESY)
  Phase-sensitive 2D NOESY with presaturation using random mixing time (**noesyphprrv**)
  Phase-sensitive 2D NOESY with 1-1 solvent suppression (**noesyph11**)
  Phase-sensitive ge-2D NOESY with presaturation using optional ^{13}C,^{15}N-decoupling (**noesyfbphpr**)
  Phase-sensitive 2D NOESY using jump-and-return and optional ^{13}C,^{15}N decoupling (**noesygpphjrrs**)

- **Gradient-based:**

  Phase-sensitive ge-2D NOESY (**noesygpph**)
  Phase-sensitive ge-2D NOESY using purgue pulses before d1 (**noesygpphpp**)
  Phase-sensitive ge-2D NOESY with z-spoil (**noesygpphzs**)
  Phase-sensitive ge-2D NOESY using echo-antiecho (**noesyetgp**)

  Phase-sensitive ge-2D NOESY using presaturation (**noesygpphpr**)

- **Gradient-based and solvent suppression:**

  Phase-sensitive 2D NOESY with WATERGATE:
  - Using 3-9-19 (**noesygpph19** | NOESYGPPH19SW)
  - Using water flip-back and 3-9-19 (**noesyfpgpph19**)
  - Using water flip-back and water-selective 90 pulses (**noesyfpgpphwg**)
  - Using water flip-back, 3-9-19 and PFG in t_1 (**noesyfpgpphrs19**)
  - Using water flip-back, water-selective 90 pulses and PFG in t_1 (**noesyfpgpphrswg**)

Phase-sensitive 2D NOESY with excitation sculpting:

Using W5 (**noesygpphw5**)

Using 180 water-selective pulse (ES element) (**noesyesgpph**)

Using 180 water-selective pulse (ES element) and ZQ suppression (**noesyesgpphzs**)

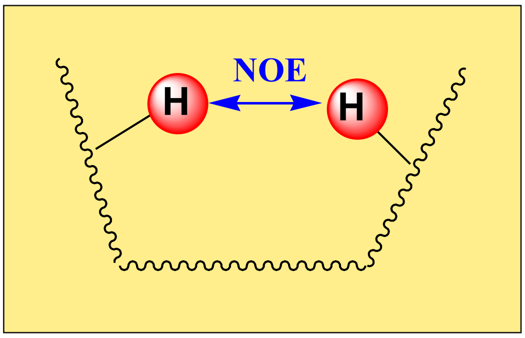
Using 180 water-selective pulse (ES element) and optional ^{13}C,^{15}N-decoupling (**noesyesfbgpph**)

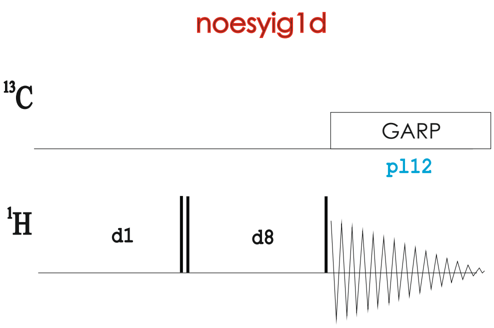
Using 180 water-selective pulse (ES element), water flip-back and optional ^{13}C,^{15}N-decoupling (**noesyesfpgpphrs**)

- **Related Experiment:**

Phase-sensitive 2D NOESY with RELAY and DQF (NOESY-RELAY experiment) (**noesydfphrl**)

Also see:
Selective 1D NOESY, 2D ROESY, 2D HSQC-NOESY & 2D HMQC-NOESY, 3D NOESY-HSQC & 3D HSQC-NOESY-HSQC, 2D & 3D X-filtered NOESY.





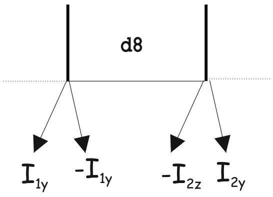
The delay d8 defines the mixing period in all NOESY experiments,

NMR Element: NOESY Block

```
...
p1 ph2
d8
p1 ph3
....

ph2=0
ph3=0

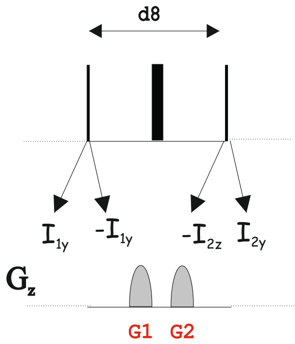
;d8 : mixing time
```




```
"TAU=d8*0.5-p16-d16-50u"

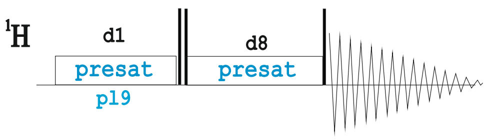

...
  p1 ph2
  TAU
  50u UNBLKGRAD
  p16:gp1
  d16
  3u
  (p2 ph4):f1
  3u
  p16:gp1*-1
  d16
  50u BLKGRAD
  TAU
  p1 ph3
...

ph2=0
ph3=0
```

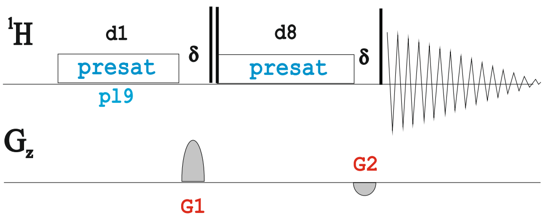


1D NOESY with presaturation (also see related pulse programs in lc-nmr experiments)

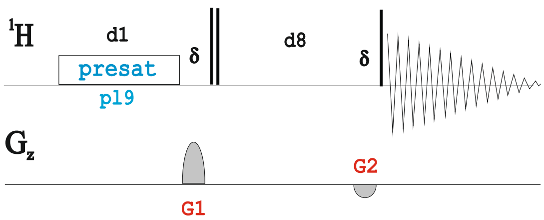
noesypr1d



noesygppr1d

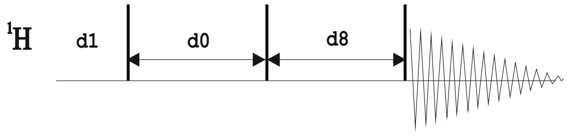


noesygppr1d.2

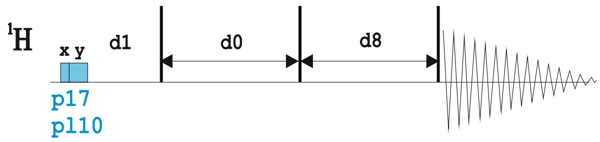


Classical 2D NOESY with presaturation

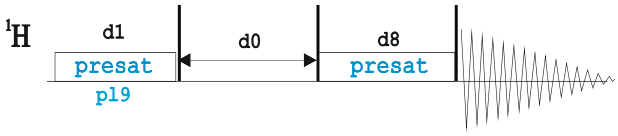
noesyph
noesyphrv



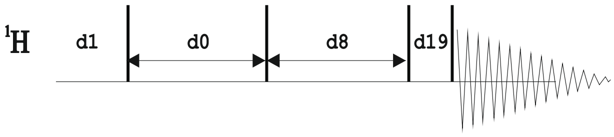
noesyphpp



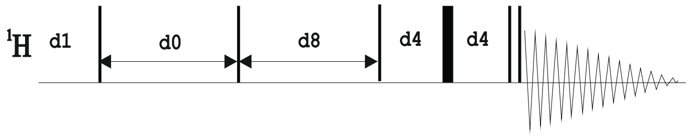
noesyphpr
noesyphprrv



noesyph11

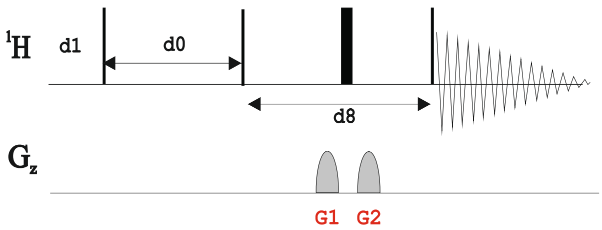


noesydfphrl

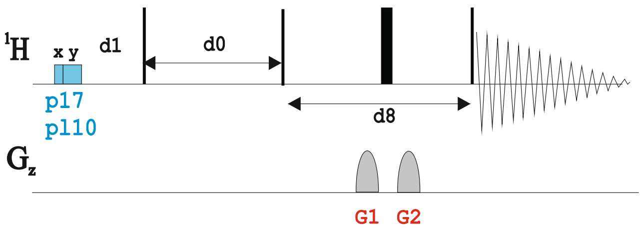


2D NOESY using Gradients

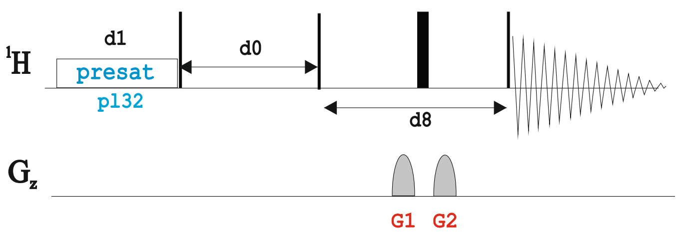
noesygpph



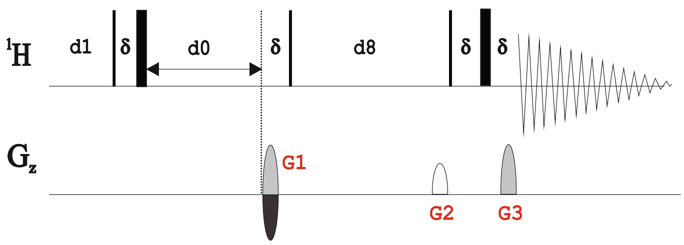
noesygpphpp



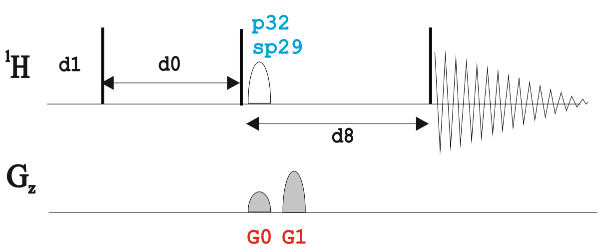
noesygpphpr



noesyetgp

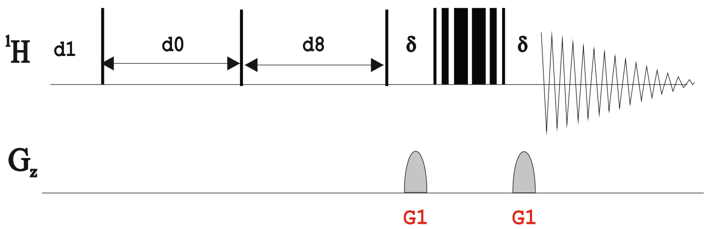


noesygpphzs

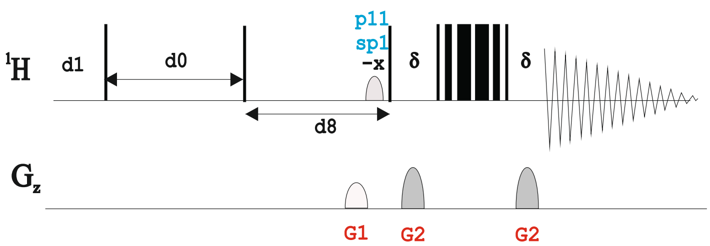


2D NOESY using WATERGATE

noesygpph19



noesyfpgpph19



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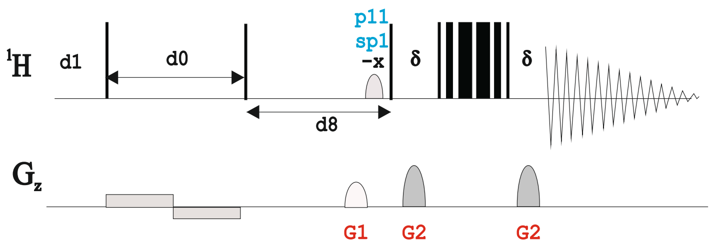

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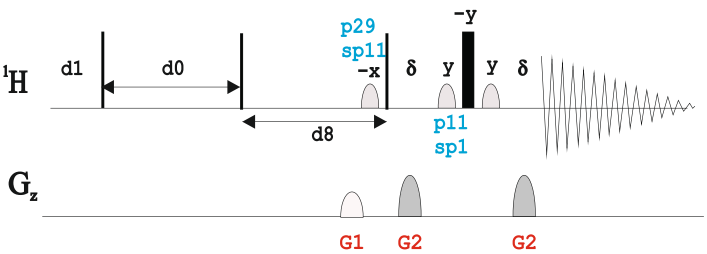

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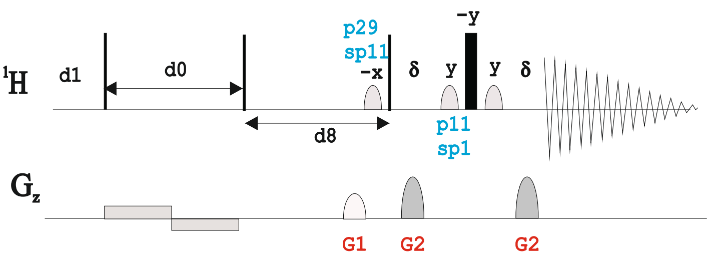
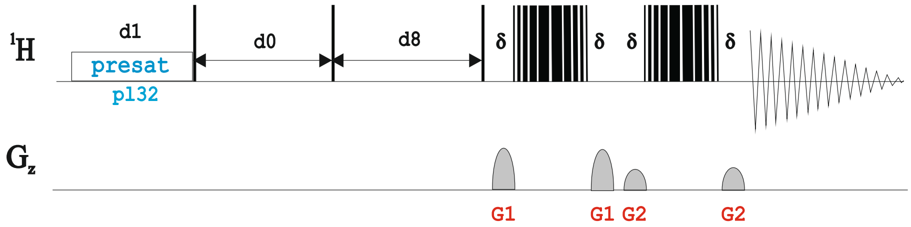
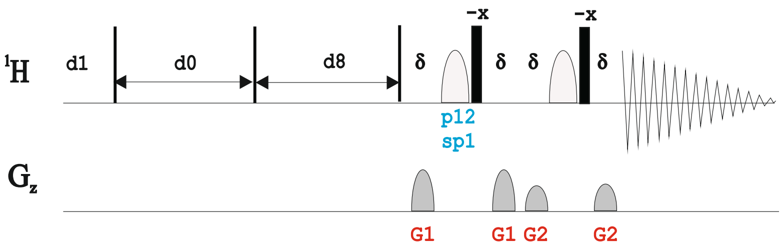


Classical 2D NOESY using excitation sculpting

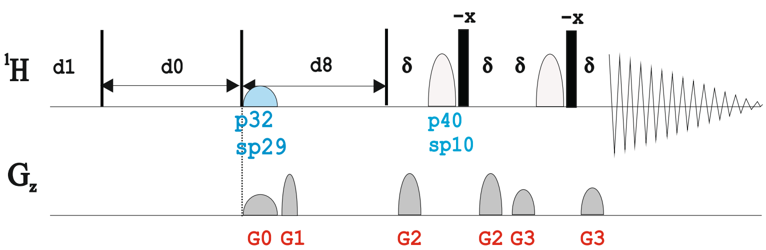
noesygpphw5



noesyesgpph



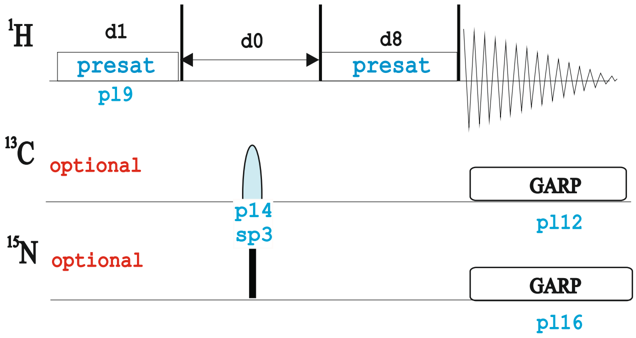
noesyesgpphzs



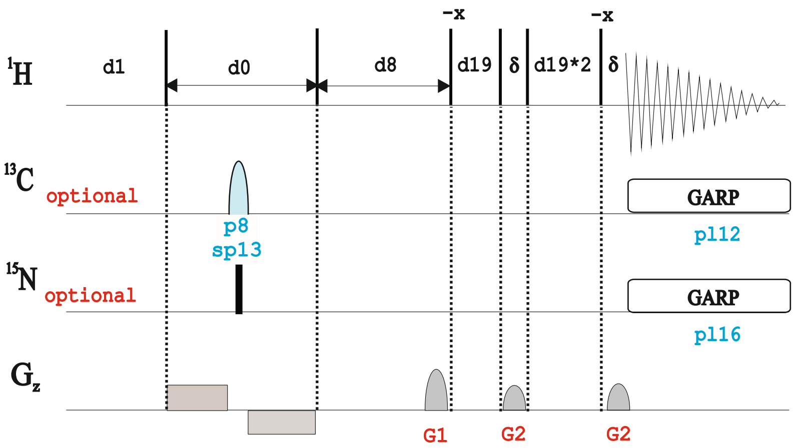
10:40:31:11

2D NOESY with Heteronuclear Decoupling (for labelled compounds)

noesyfbphpr



noesygpphjrrs



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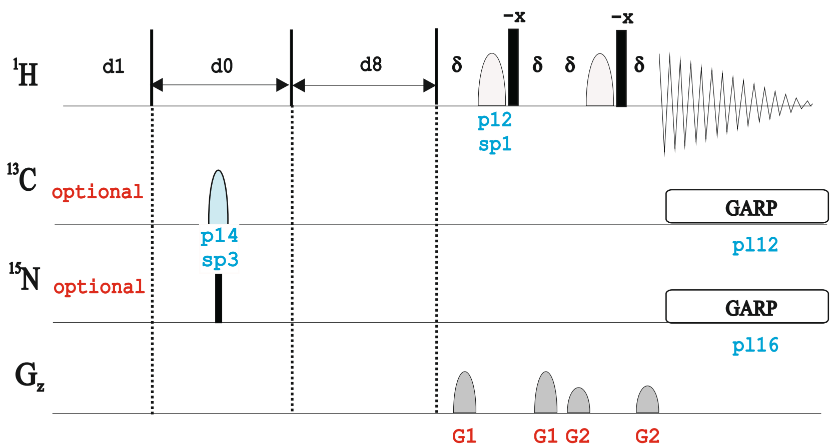
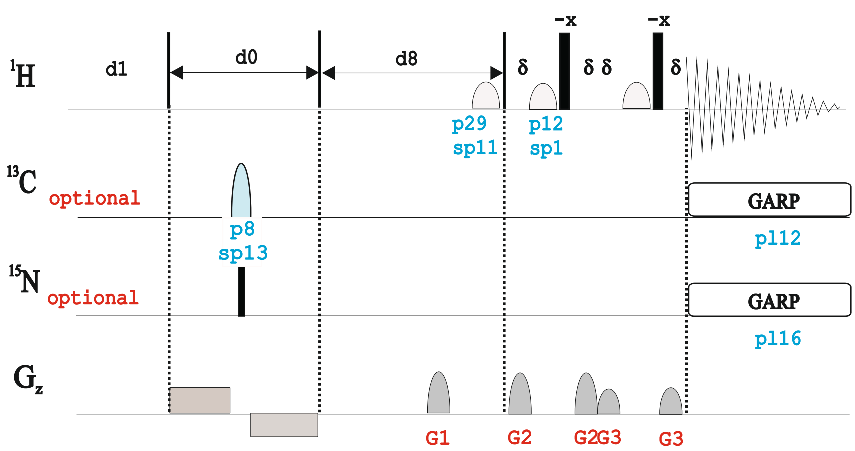


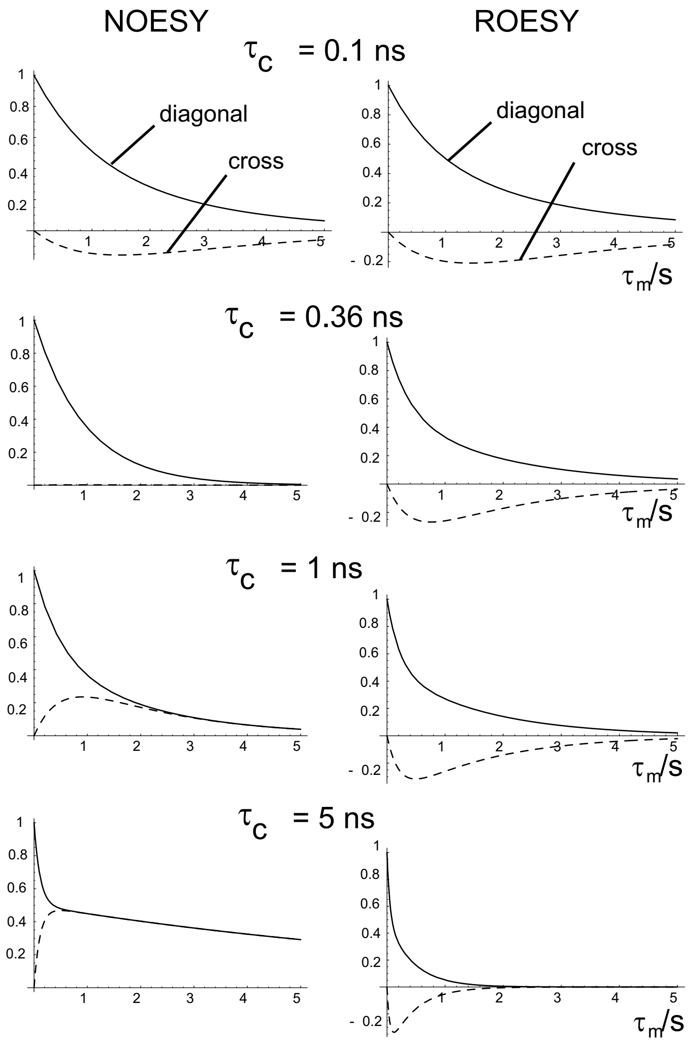
noesyesfpgpphrs



NOE in small molecules is negative
NOE in large molecules is positive
There is a MW range near to NOE null point: **Need for ROESY Experiment!!!**

In NOESY possibility of exchange cross-peaks (positive) that can originate false NOEs (negative), three-spin effects (positive) and COSY cross-peaks (antiphase-J)

In ROESY experiments, the ROE cross-peaks are always negative: There is also possibility of exchange cross-peaks (positive) that can originates false ROEs (negative), three-spin effects (positive) and TOCSY cross-peaks (inphase-J)



NOE transfer strongly depends onTemperature and Solvent (viscosity), Magnetic field, molecular weight and mixing time

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2D ROESY EXPERIMENTS

Experiment Description

A ROESY (Rotating-Frame Overhauser SpectroscopY) experiment provides through-space proton-proton connectivities in an alternative way to NOESY experiment in null-NOE experimental conditions.

Sample Requirements

ROESY experiments can be recorded on any type of sample.
Solvent-supressed versions are required for samples dissolved in H2O.

Hardware Requirements

ROESY experiments can be recorded on any probehead.
A pulsed-field gradient coil (highly recommended) is required for gradient-based versions.

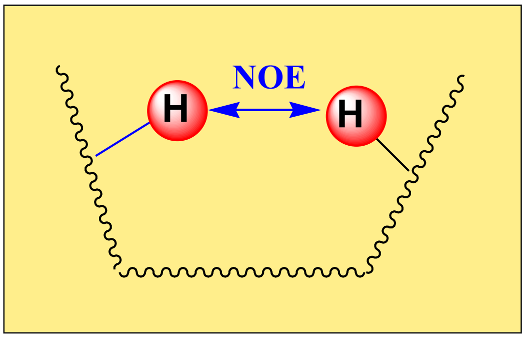
NMR Spectrum

The ROESY experiment yields a 2D proton-proton correlation map with two different types of signals: i) autocorrelation diagonal peaks, and ii) Off-diagonal cross-peaks correlating spins close each other. ROESY spectra are represented in phase-sensitive mode in order to distinguish exchange cross-peaks and unwanted TOCSY contributions.

Related Experiments

Selective 1D ROESY
2D NOESY experiment
2D HMQC-ROESY and HSQC-ROESY experiments

The pulse p15 (in microseconds), applied at power level pl11, defines the mixing period in all ROESY experiments.



2D ROESY Experiments

- **Phase-cycled:**

  Phase-sensitive 2D ROESY (**roesyph** | ROESYPHSW)
  Phase-sensitive 2D ROESY using purgue pulses before d1 (**roesyphpp**)
  Phase-sensitive 2D T-ROESY (**roesyph.2**)
  Phase-sensitive 2D T-ROESY using purgue pulses before d1 (**roesyphpp.2**)
  Phase-sensitive 2D ROESY with compensation (**croesyph**)
  Phase-sensitive off-resonance 2D ROESY (**troesyph**)

- **Phase-cycled and solvent suppression:**

  Phase-sensitive 2D ROESY with presaturation (**roesyphpr** | ROESYPHPR)
  Phase-sensitive 2D T-ROESY with presaturation (**roesyphpr.2**)
  Phase-sensitive 2D ROESY with compensation and presaturation (**croesyphpr**)
  Phase-sensitive off-resonance 2D ROESY with presaturation (**troesyphpr**)

- **Gradient-based:**

  Phase-sensitive ge-2D ROESY using echo-antiecho (**roesyetgp**)
  Phase-sensitive ge-2D ROESY with T-ROESY using echo-antiecho (**roesyetgp.2**)
  EASY-ROESY: Phase-sensitive ge-2D ROESY using adiabatic spin-locks and presaturation (**roesyadjsphpr**)

- **Gradient-based and solvent suppression:**

  Phase-sensitive 2D ROESY with WATERGATE using 3-9-19 (**roesygpph19**)
  Phase-sensitive 2D T-ROESY with WATERGATE using 3-9-19 (**roesygpph19.2**)
  Phase-sensitive 2D ROESY with excitation sculpting using 180 water-selective pulse (ES element) (**roesyesgpph**)

Also see:
Selective 1D ROESY Experiments
2D NOESY Experiments
2D HSQC-ROESY Experiments

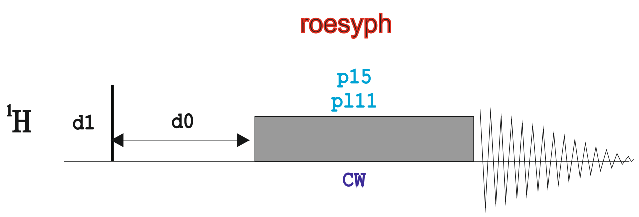
References:
1. A. Bax & D.G. Davis, J. Magn. Reson 63, 207-213 (1985)
2. T.-L. Hwang & A.J. Shaka, J. Am. Chem. Soc. 114, 3157-3159 (1992)
3. J. Schleucher, J. Quant, S. Glaser & C. Griesinger,J. Magn. Reson A 112, 144-151 (1995)

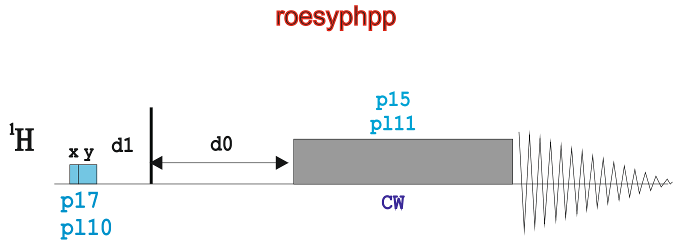
NMR Element: ROESY Element

```
...
  4u pl11:f1
  p15 ph2
....

ph2=0

;pl11: f1 channel - power level for ROESY-spinlock
;p15: f1 channel - pulse for ROESY spinlock
```





NMR Element: T-ROESY Element

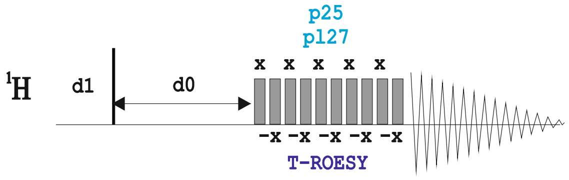
```
"l4=p15/(p25*2)"


....
  4u pl27:f1
4 p25 ph2
  p25 ph3
  lo to 4 times l4
  ....

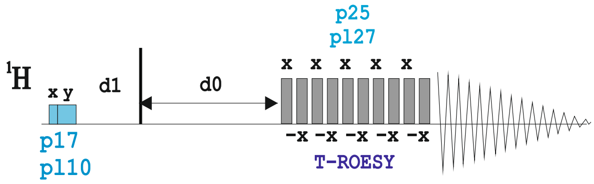

ph2=0 2 0 2 1 3 1 3
ph3=2 0 2 0 3 1 3 1
ph31=0 2 2 0 1 3 3 1


;pl27: f1 channel - power level for pulsed ROESY-spinlock
;p15: f1 channel - pulse for ROESY spinlock
;p25: f1 channel - 180 degree pulse at pl27
;l4: loop for spinlock = p15 / p25*2
```

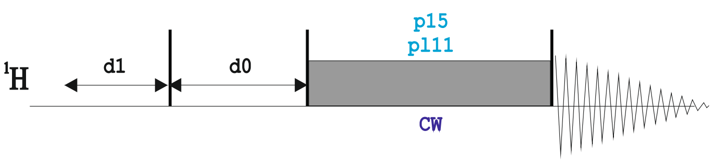
roesyph.2



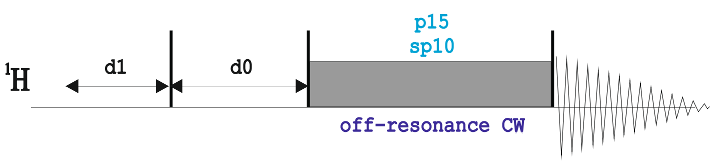
roesyphpp.2



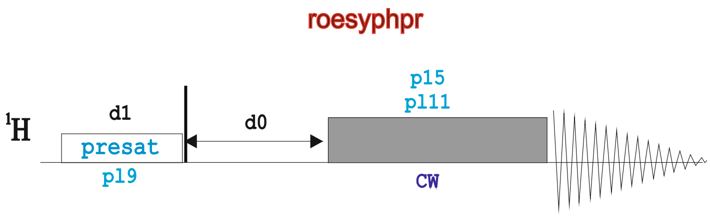
croesyph

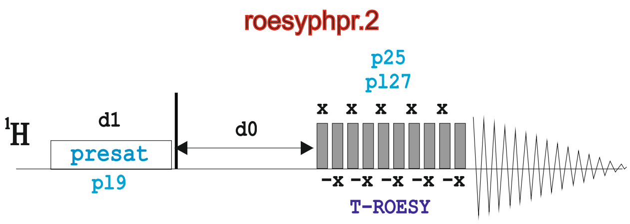


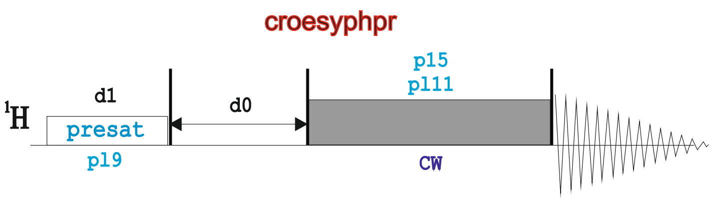
troesyph

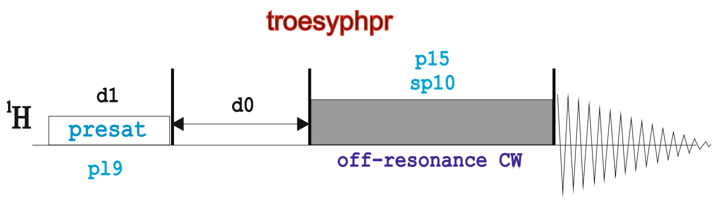


2D ROESY with presaturation



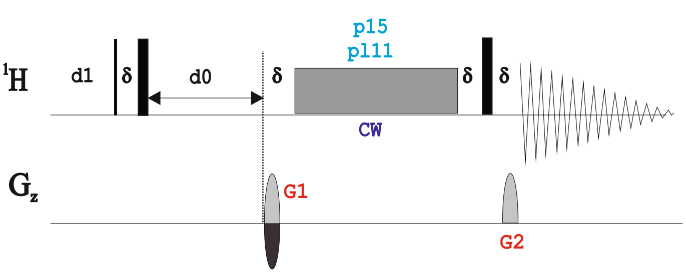




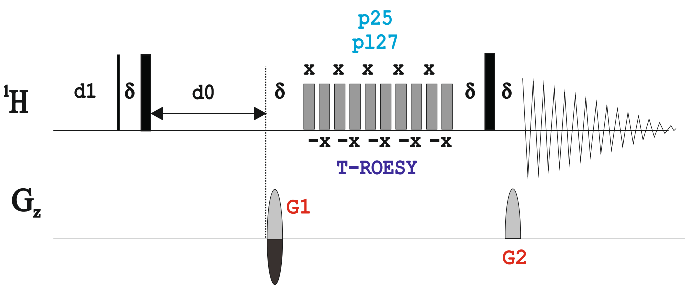


2D ROESY with echo/antiecho

roesyetgp

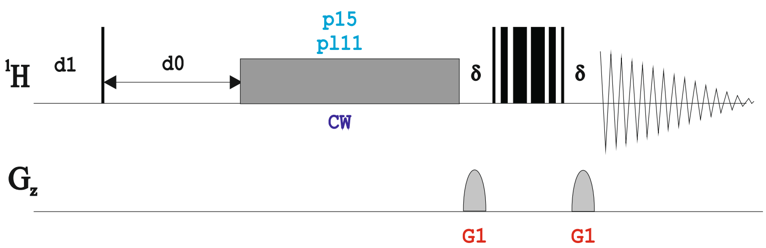


roesyetgp.2

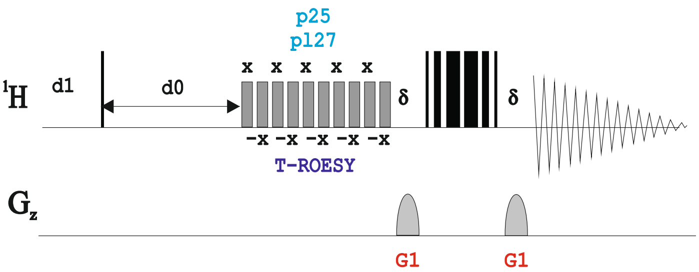


2D ROESY using WATERGATE or excitation scultping

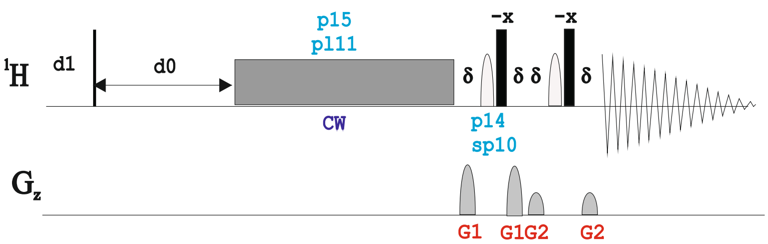
roesygpph19



roesygpph19.2

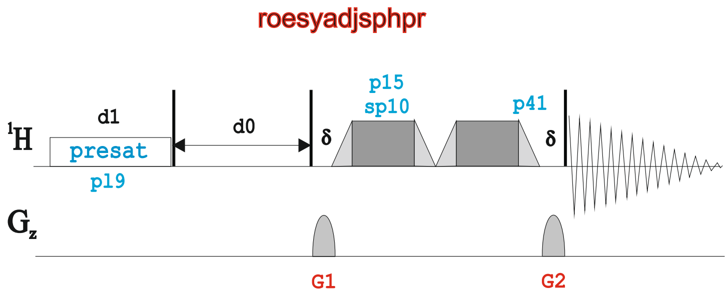


roesyesgpph



2D ROESY using adiabatic ramps

C.M. Thiele, K. Petzold & J. Schleucher, Chem. Eur. J. 15, 585-588 (2009)



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1D & 2D MULTIPLE-QUANTUM EXPERIMENTS

Experiment Description

Multiple-Quantum experiments permit to study J-coupled homonuclear spin systems by the generation of MQ coherences. The creation of particular double-quantum (DQ), triple-quantum (TQ)... coherences can be achieved by specific phase cycling or appropriate gradient ratios.

Sample Requirements

MQ experiments can be recorded on any type of sample.
Solvent-supressed versions are required for samples dissolved in H2O.

Hardware Requirements

MQ experiments can be recorded on any probehead.
A pulsed-field gradient coil is required for gradient-based versions.

NMR Spectrum

The MQ experiment yields a 2D correlation map correlating MQ frequencies with conventional chemical shifts. For instance, in a DQ experiment, non-coupled resonances (singlets) are filtered out and they do not appear in the spectrum.

Related Experiments

1D & 2D INADEQUATE

References:

1. U. Piantini et al., J. Am. Chem. Soc. 104, 6800 (1982)
2. T.H. Mareci & R. Freeman, J. Magn. Reson. 51, 531 (1983)
2D DQ:
3. C. Dalvit and J-M. Boehlen, J. Magn. Reson. B111, 76 (1996)
4. C. Dalvit and J-M. Boehlen, J. Magn. Reson. B113, 195 (1996)

The delay d4 defines the initial echo period that allows to generate the corresponding anti-phase state that is suitable to afford MQ coherences after a second 90 pulse.

1D & 2D DQ Experiments

- **1D Double-Quantum (DQ)**

  1D Double-Quantum experiments (**dqs1d**)
  1D Multiple Quantum Filter (**mqsgp1d** | **mqsgp1d2**)

- **2D Phase-cycled Double-Quantum (DQ)**

  Magnitude-mode 2D Double-Quantum (DQ) (**dqsqf**)
  Phase-sensitive Double-Quantum (DQ)(**dqsph**)
  Phase-sensitive 2D Double-Quantum (DQ) with presaturation (**dqsphpr**)

- **2D Gradient-based Double-Quantum (DQ)**

  Phase-sensitive ge-2D Double-quantum using echo-antiecho, 45/135 degree conversion pulse for better sensitivity and remote peak minimisation (**dqseagp135**)
  Phase-sensitive ge-2D Double-quantum using echo-antiecho (**dqseagp90**)

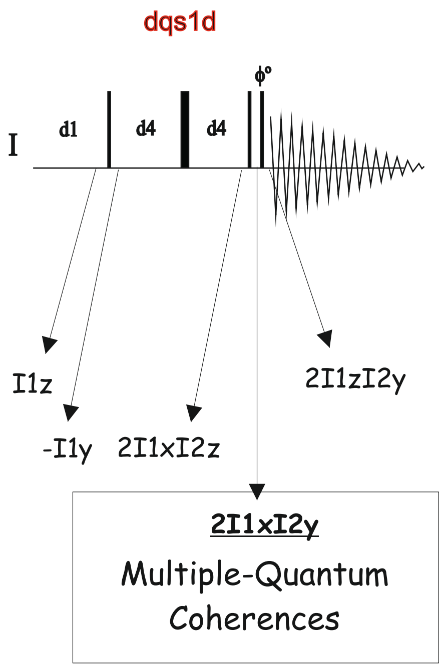
Also see:
1D & 2D INADEQUATE

NMR Building Block: Generation of Homonuclear Multiple-Quantum Coherences

```
...
d1
  p1 ph1
  d4
  p2 ph2
  d4
  p1 ph1
  d13
  p0 ph3
  go=2 ph31
  30m mc #0 to 2 F0(zd)
exit

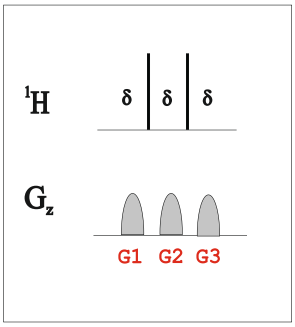

ph1=0 2
ph2=0 2 0 2 0 2 0 2 2 0 2 0 2 0 2 0
ph3=0 0 2 2 3 3 1 1
ph31=0 0 2 2 1 1 3 3

....
;d4 : 1/(4J)
```

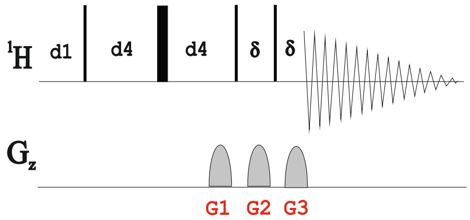


NMR Building Block: Gradient-Based MQ Filter

```
...
  50u UNBLKGRAD
  p16:gp1
  d16
  p1 ph2
  d13
  p16:gp2
  d16
  p1 ph3
  50u
  p16:gp3
  d16
  4u BLKGRAD
.....
```



mqsgp1d2



p1G1+p2G2+p3G3=0

By Definition: p1=1 and p3=-1

G1+p2G2-G3=0

Selection of DQC
p2=+2

G1+2G2-G3=0

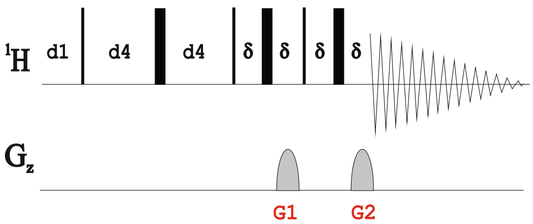
10:10:30 for dq filter

Selection of TQC
p2=+3

G1+3G2-G3=0

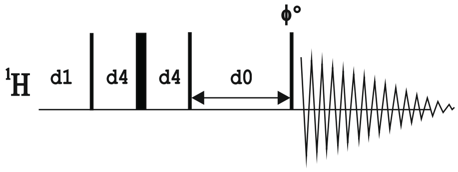
10:10:40 for tq filter

mqsgp1d

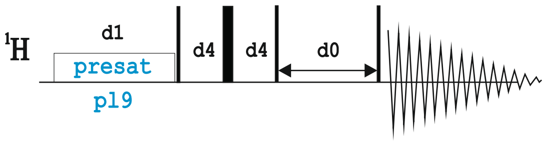


10:20 for double quantum filter
10:30 for triple quantum filter

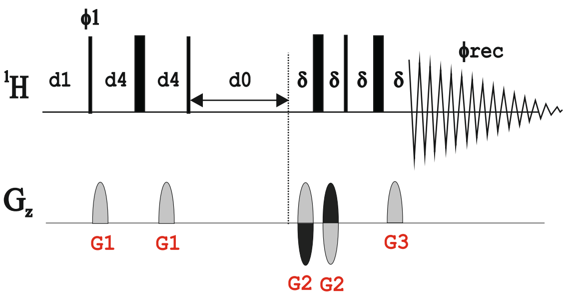
dqsph
dqsqf



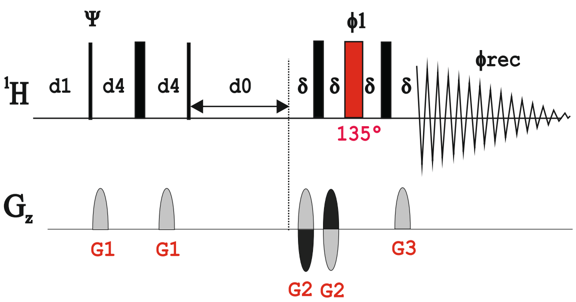
dqsphpr



dqseagp90



dqseagp135



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2D J-RESOLVED EXPERIMENTS

Experiment Description

A 2D J-Resolved experiment is a simple variable spin-echo period that allows to decode all J-coupling information from chemical shift information

Sample Requirements

J-Resolved experiments can be recorded on any type of sample.
Solvent-supressed versions are required for samples dissolved in H2O. In dilute samples, X-decoupling is needed to efficiently remove satellites from the strong solvent resonances

Hardware Requirements

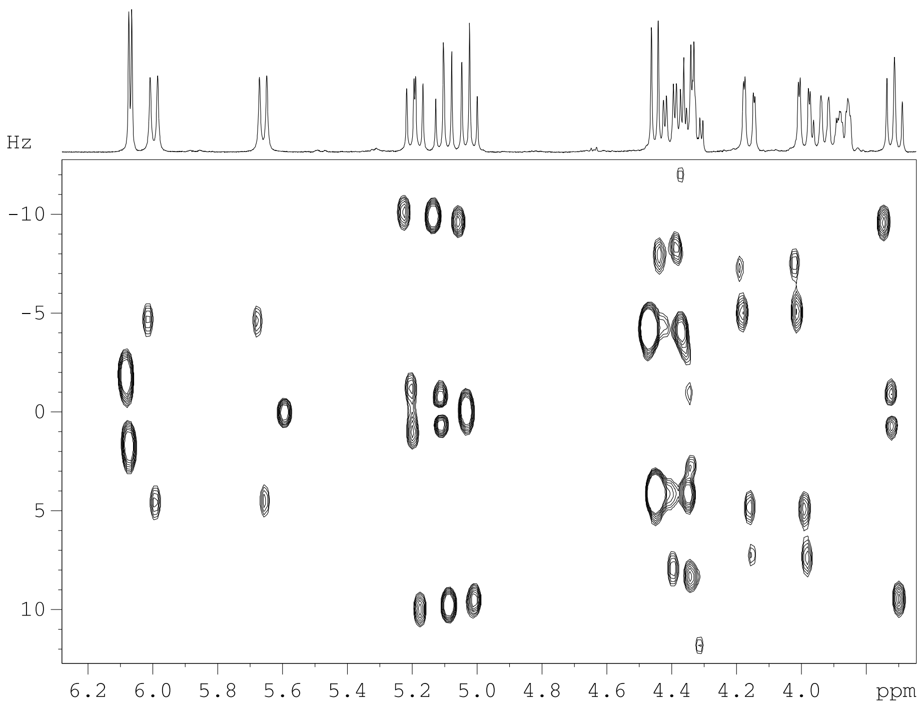
J-Resolved experiments can be recorded on any probehead.

NMR Spectrum

The J-Resolved experiment yields a 2D correlation map correlating J-coupling with conventional chemical shifts.

Related Experiments

2D Heteronuclear J-Resolved



2D J-Resolved Experiments

- **Classical:**

  Magnitude-mode 2D J-Resolved (**jresqf**)
  Magnitude-mode 2D J-Resolved with f2 decoupling (**jresdcqf**)

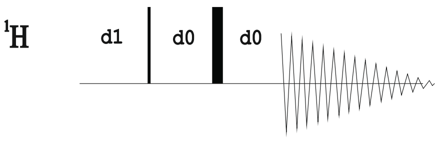
- **Gradients and presaturation:**

  ge-2D J-Resolved with presaturation and gradients (**jresgpprqf**)

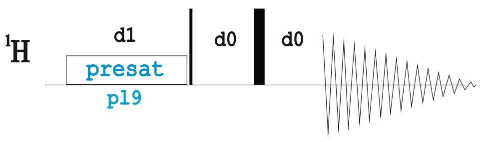
- **With single/multiple solvent suppression (LC-NMR):**

  2D J-Resolved with presaturation (**lcjresprqf**)
  2D J-Resolved with presaturation using shape pulse (**lcjrespsqf**)
  2D J-resolved with double presaturation and cw-decoupling on f2 (**lcjrescwfdprqf**)
  2D J-resolved with presaturation and cw-decoupling on f2 (**lcjrescwprqf**)
  2D J-resolved with presaturation using shape pulse and cw-decoupling on f2 (**lcjrescwpsqf**)
  2D J-resolved with double presaturation (**lcjresf2prqf**)

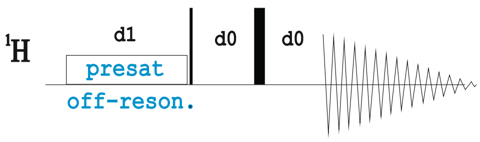
jresqf



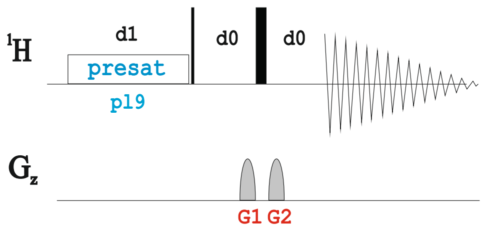
lcjresprqf



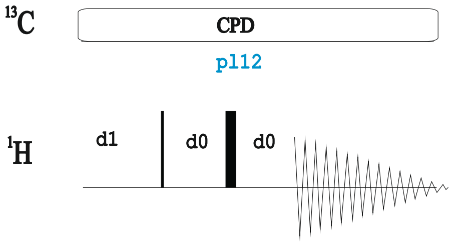
lcjrespsqf



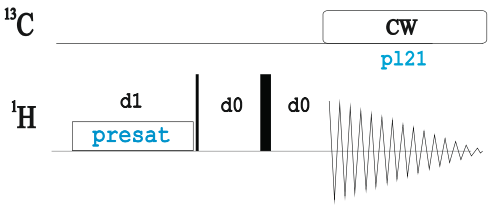
jresgpprqf



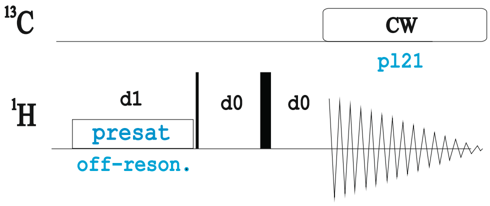
jresdcqf



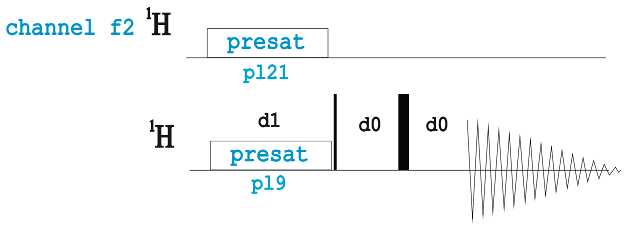
lcjrescwprqf



lcjrescwpsqf



lcjresf2prqf



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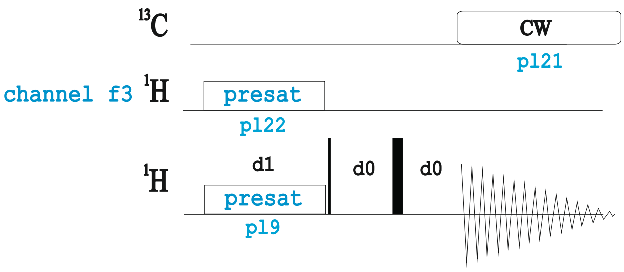

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1D INEPT EXPERIMENTS

Experiment Description

The INEPT experiment is based on a heteronuclear pulse sequence in which signal intensities are strongly enhanced by polarization transfer.

Sample Requirements

INEPT experiments can be recorded on any type of sample.

Hardware Requirements

INEPT experiments can be recorded on any probehead.

NMR Spectrum

The non-refocused INEPT version affords anti-phase multiplets with enhanced sensitivity. On the other hand, refocused INEPT versions afford in-phase multiplets that can be decoupled.

Related Experiments

1D DEPT experiments and Other 1D heteronuclear editing methods
2D HETCOR and 2D HSQC corrleation experiments

1D INEPT Experiments

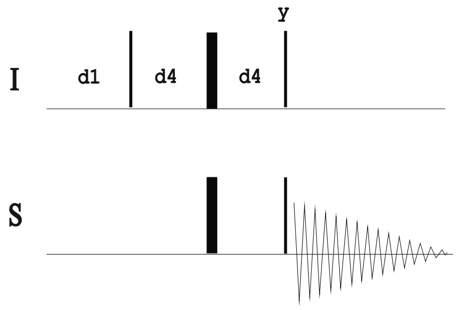
INEPT without refocusing (**ineptnd**)
Refocused INEPT with decoupling (**ineptrd**)
Refocused INEPT with decoupling using adiabatic pulses (**ineptrdsp**)
INEPT+ without decoupling (**ineptpnd**)

Non-refocused ^{1}H-coupled ^{15}N spectrum using INEPT (**ineptnd**)
^{1}H-decoupled ^{15}N spectrum using INEPT (**ineptrd** | N15INEPT)
Refocused ^{1}H-coupled ^{15}N spectrum using INEPT+ (**ineptpnd**)

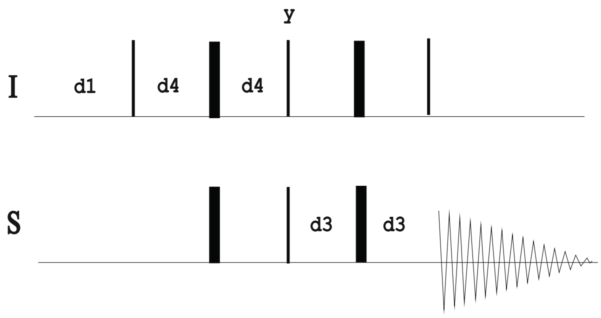
1D X-relayed H,X-COSY (**ineptrl1** / **ineptrl2**)

Also see:
DEPT Experiments

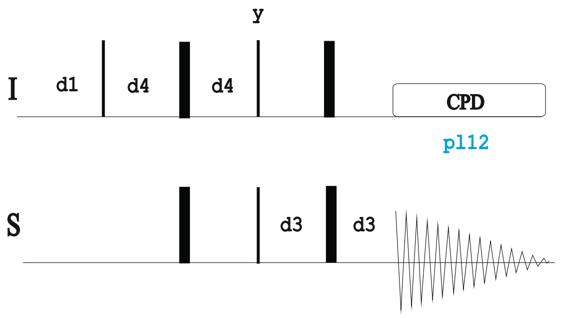
ineptnd



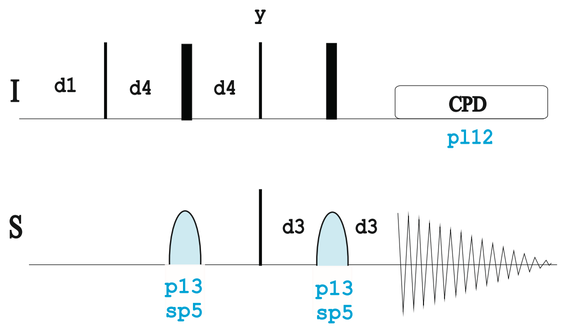
ineptpnd



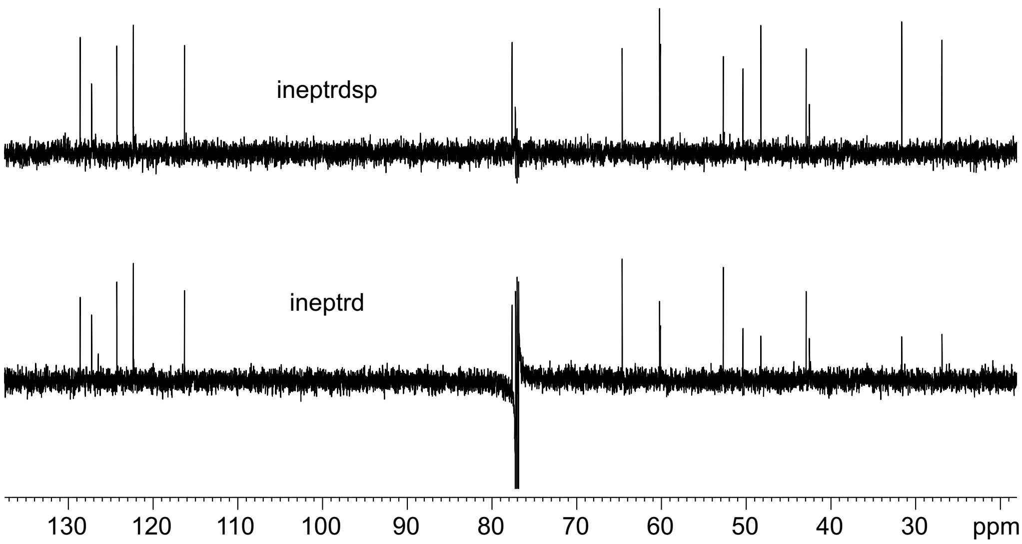
ineptrd



ineptrdsp



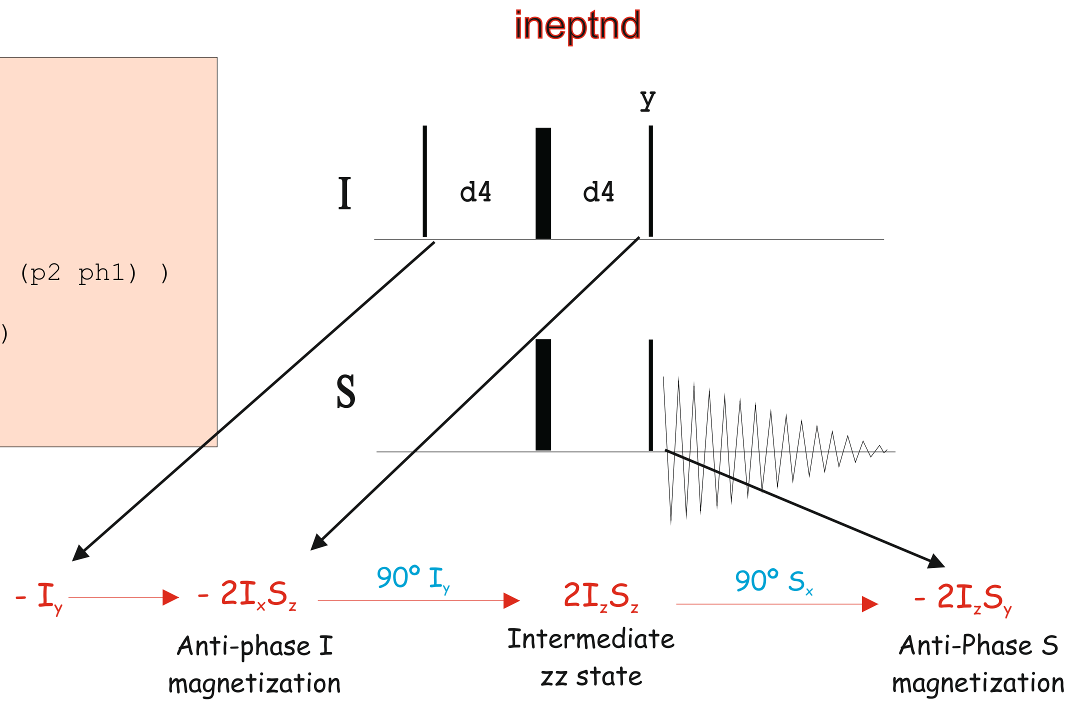
Adiabatic Pulses in Refocused INEPT



NMR BUILDING BLOCK:
POLARIZATION TRANSFER VIA INEPT

```
"d4=1s/(cnst2*4)"
....

  d1
  (p3 ph1):f2
  d4
  (center (p4 ph1):f2 (p2 ph1) )
  d4
  (p3 ph3):f2 (p1 ph1)
.....
ph1=0
ph2=1
```

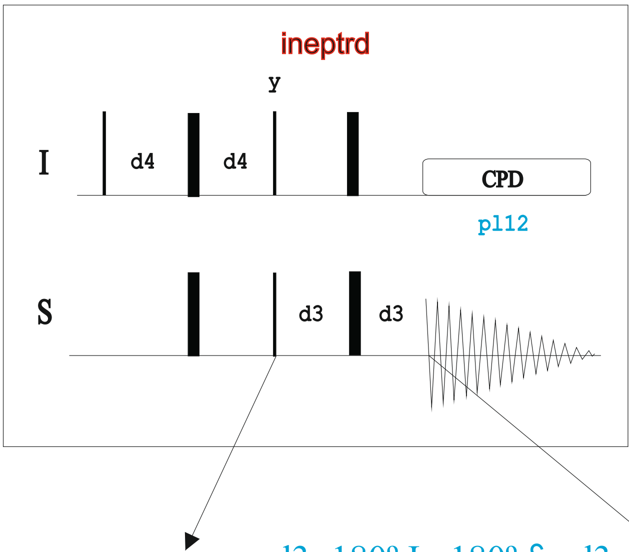


The delay d4 is optimized to 1/4J(XH).

NOE	$1+ \gamma_I/2*\gamma_S$
INEPT	γ_I/γ_S

X	^{11}B	^{13}C	^{15}N	^{29}Si	^{57}Fe	^{103}Rh	^{109}Ag	^{119}Sn	^{183}W
NOE	2.56	2.99	-3.94	-1.52	16.48	-16.89	-9.75	-0.41	13.02
INEPT	3.12	3.98	9.87	5.03	30.95	31.77	21.50	2.81	24.04

REFOCUSED INEPT



Delay d3 :
1/(6J(XH)) XH, XH2, XH3 positive
1/(4J(XH)) XH only
1/(3J(XH)) XH, XH3 positive, XH2 negative

$-2I_zS_y$ $\xrightarrow{d3-180^\circ I_x-180^\circ S_x-d3}$ S_x

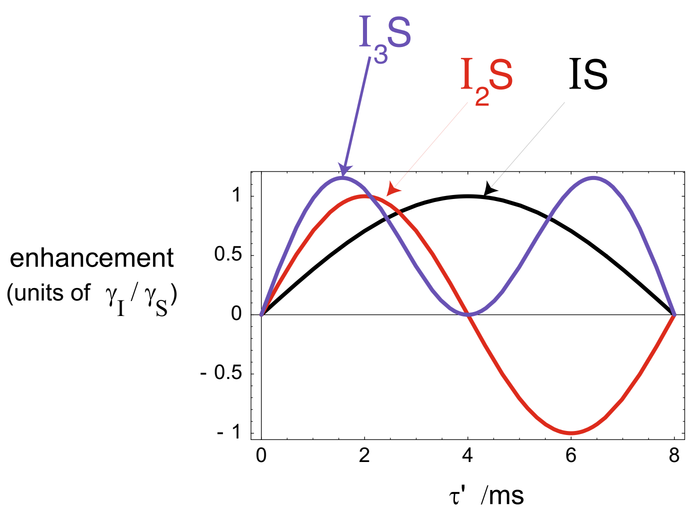
Antiphase S Magnetization → In-Phase S Magnetization

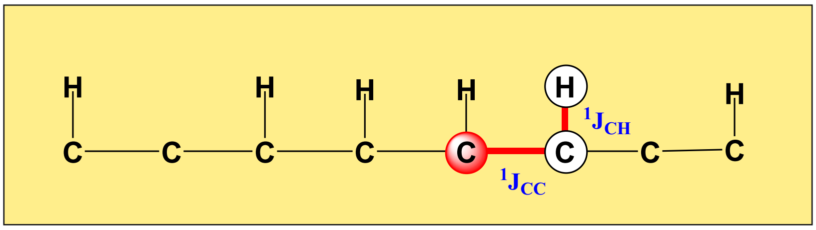
CH spin systems: $-2I_{1z}S_y\cos + \mathbf{S_x\sin}$

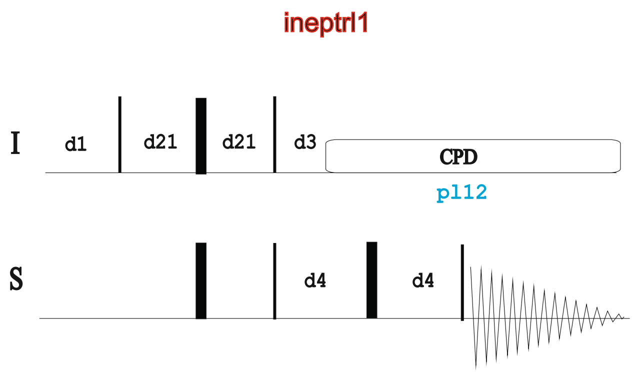
CH_2 spin systems:
$-2I_{1z}S_y\cos\cos + \mathbf{S_x\sin\cos}$
$-2I_{1z}I_{2z}S_y\cos\sin + 2I_{2z}S_y\sin\sin$

Ch_3 spin systems:
$-2I_{1z}S_y\cos\cos\cos + \mathbf{S_x\sin\cos\cos}$
$-4I_{1z}I_{2z}S_y\cos\sin\cos + 2I_{2z}S_y\sin\sin\cos$
$+4I_{1z}I_{3z}S_x\cos\cos\sin + 2I_{3z}S_y\sin\cos\sin$
$+8I_{1z}I_{2z}I_{3z}S_x\cos\sin\sin - 4I_{2z}I_{3z}S_x\sin\sin\sin$

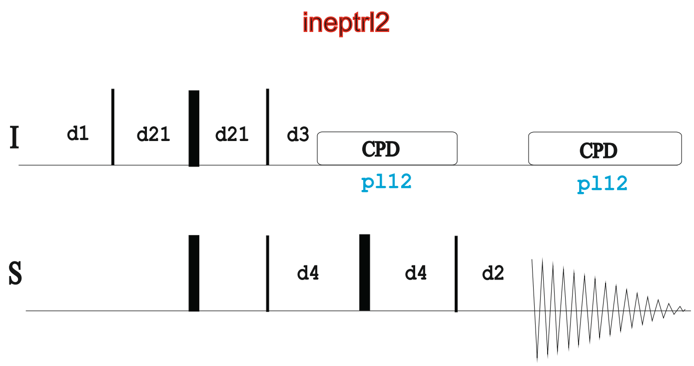
Signal intensity of a I_nS peak depends of $\sin(\pi J_{IS}\tau)\cos^{n-1}(\pi J_{IS}\tau)$







Also see inepin



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1D DEPT EXPERIMENTS

Experiment Description

The DEPT (Dirtosntionless Enhancement Polarization Transfer) experiment is based on a heteronuclear pulse sequence in which signal intensities are enhanced by polarization transfer and their phases depend of a proton flip angle.

Sample Requirements

DEPT experiments can be recorded on any type of sample.

Hardware Requirements

DEPT experiments can be recorded on any probehead.

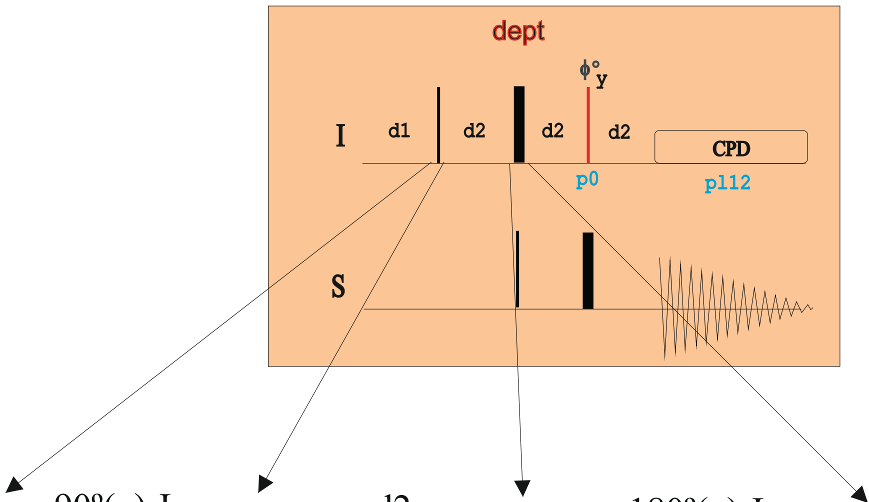
NMR Spectrum

A DEPT experiment affords a 13C spectrum in which the phase of each signal is edited as a function of carbon multiplicity. For instance, a conventional DEPT135 spectrum gives CH and CH3 with a positive phase and CH2 with a negative phase.

Related Experiments

1D INEPT experiments and Other 1D heteronuclear editing methods
2D HETCOR and 2D DEPT-HMQC corrleation experiments

Key: Evolution of Multiple-Quantum Coherences
Result: Multiplicity Information



$$I_z \xrightarrow{90°(x)\text{-}I} -I_y \xrightarrow{d2} 2I_xS_z \xrightarrow[90°(x)\text{-}S]{180°(x)\text{-}I} -2I_xS_y$$

<u>IS Spin Systems:</u>

$$\xrightarrow{d2} -2I_xS_y \xrightarrow[180°(x)\text{-}S]{\phi°(y)\text{-}I} 2I_xS_y\cos(\phi) - \mathbf{2I_zS_y\sin(\phi)} \xrightarrow{d2} \mathbf{S_x\sin(\phi)} + ...$$

<u>I_1I_2S Spin Systems:</u>

$$\xrightarrow{d2} -4I_{1x}I_{2z}S_x \xrightarrow[180°(x)\text{-}S]{\phi°(y)\text{-}I} -4I_{1x}I_{2z}S_x\cos(\phi)\cos(\phi) + \mathbf{4I_{1z}I_{2z}S_x\sin(\phi)\cos(\phi)}$$
$$-4I_{1x}I_{2x}S_x\cos(\phi)\sin(\phi) + 4I_{1z}I_{2x}S_x\sin(\phi)\sin(\phi)$$

$$\xrightarrow{d2} \mathbf{S_x\sin(\phi)\cos(\phi)} + ...$$

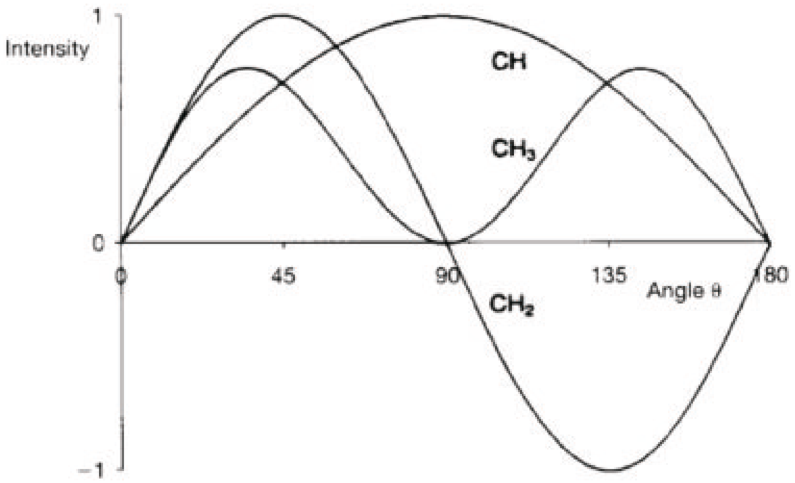
<u>$I_1I_2I_3S$ Spin Systems:</u>

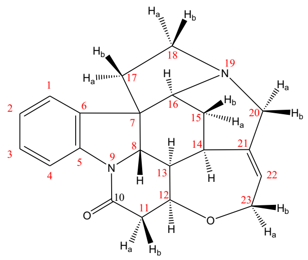
$$\xrightarrow{d2} -8I_{1x}I_{2z}I_{3z}S_y \xrightarrow[180°(x)\text{-}S]{\phi°(y)\text{-}I}$$

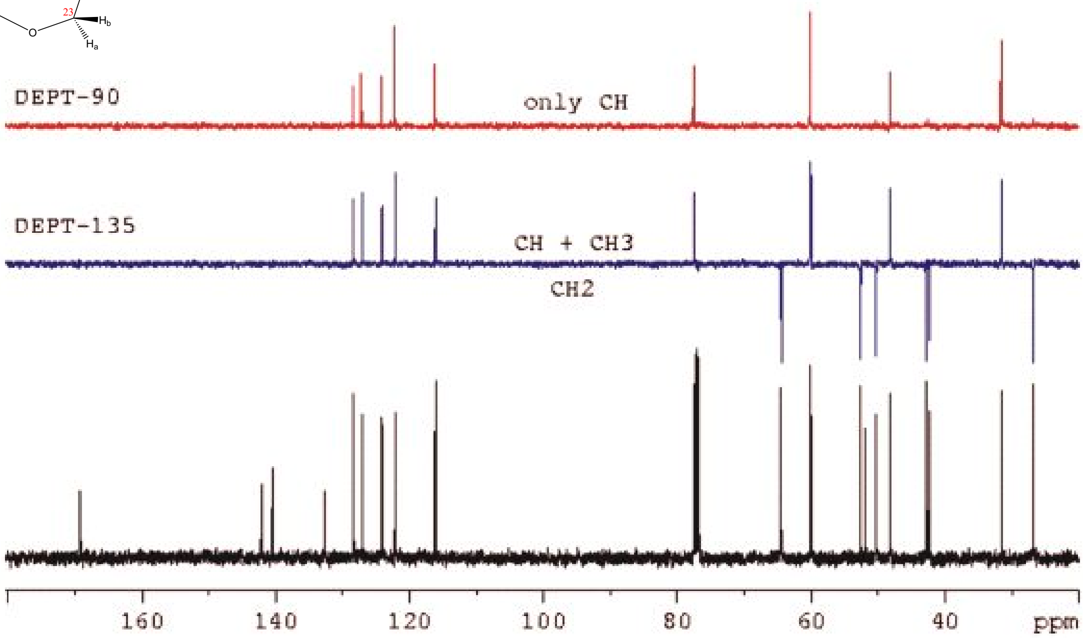
$$8I_{1x}I_{2z}I_{3z}S_y\cos(\phi)\cos(\phi)\cos(\phi) - \mathbf{8I_{1z}I_{2z}I_{3z}S_y\sin(\phi)\cos(\phi)\cos(\phi)}$$
$$8I_{1x}I_{2x}I_{3z}S_y\cos(\phi)\sin(\phi)\cos(\phi) - 8I_{1z}I_{2x}I_{3z}S_y\sin(\phi)\sin(\phi)\cos(\phi)$$
$$8I_{1x}I_{2z}I_{3y}S_y\cos(\phi)\cos(\phi)\sin(\phi) - 8I_{1z}I_{2z}I_{3y}S_y\sin(\phi)\cos(\phi)\sin(\phi)$$
$$8I_{1x}I_{2x}I_{3y}S_y\cos(\phi)\sin(\phi)\sin(\phi) - 8I_{1z}I_{2x}I_{3y}S_y\sin(\phi)\sin(\phi)\sin(\phi)$$

$$\xrightarrow{d2} \mathbf{S_x\sin(\phi)\cos(\phi)\cos(\phi)} + ...$$

Signal intensity of a I_nS peak in the DEPT pulse scheme depends of $\sin(\phi)\cos^{n-1}(2\phi)$







The delay d2 is optimized to 1/2J(XH).

1D DEPT Experiments

Classical

- DEPT (**dept**)
- DEPT-45 (**dept45** | C13DEPT45)
- DEPT-90 (**dept90** | C13DEPT90)
- DEPT-135 (**dept135** | C13DEPT135)

Using Adiabatic pulses

- DEPT-45 with adiabatic pulses (**deptsp45** / **deptsp**)
- DEPT-90 with adiabatic pulses (**deptsp90**)
- DEPT-135 with adiabatic pulses (**deptsp135**)

Using Composite pulses

- DEPT with composite pulses (**deptcp**)
- DEPT-45 with composite pulses (**deptcp45**)
- DEPT-90 with composite pulses (**deptcp90**)
- DEPT-135 with composite pulses (**deptcp135**)

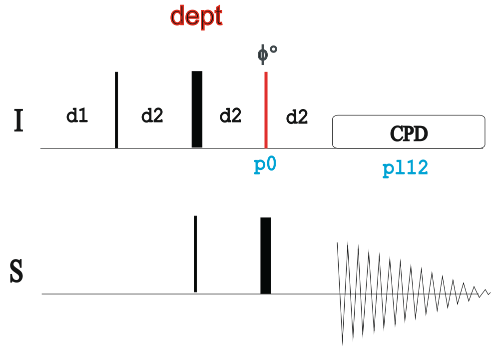
Without Decoupling

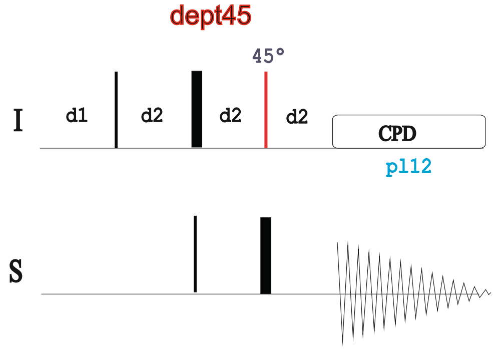
- DEPT without ^{1}H-decoupling (**deptnd**)
- DEPT++ without ^{1}H-decoupling (**deptppnd**)

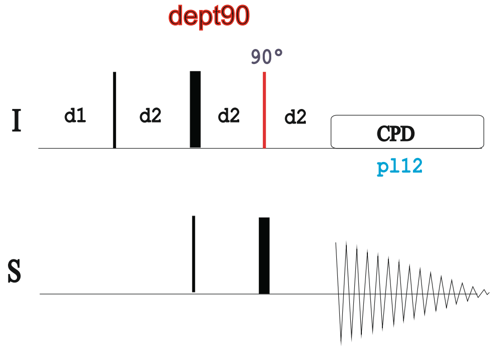
DEPTQ for quaternary carbons

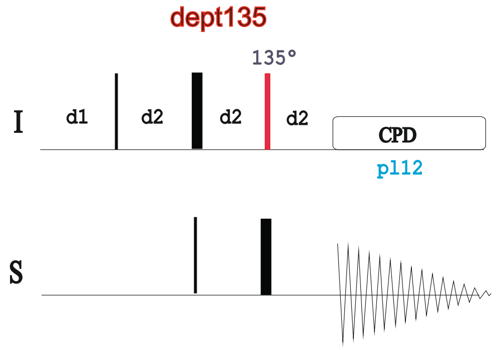
- Using adiabatic pulses (**deptqsp**)
- Using gradients and adiabatic pulses (**deptqgpsp**)
- Using gradients, adiabatic pulses and NOE (**deptqgpsp.2**)

Also see:
INEPT Experiments

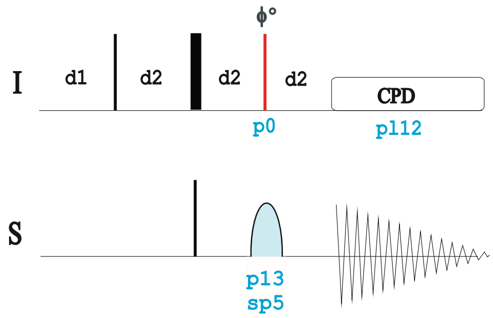




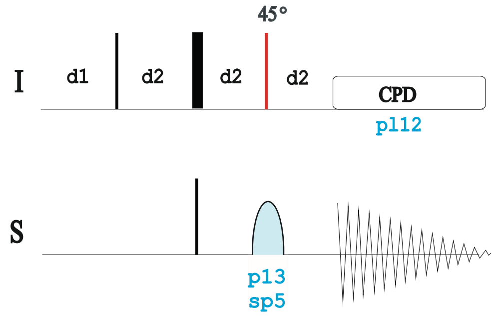




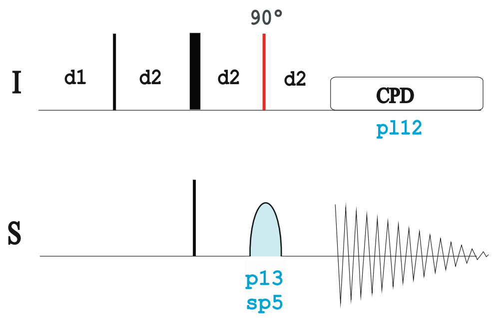
deptsp



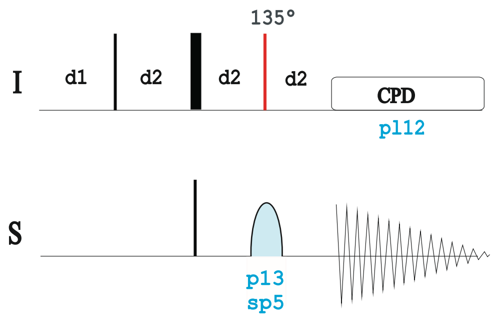
deptsp45



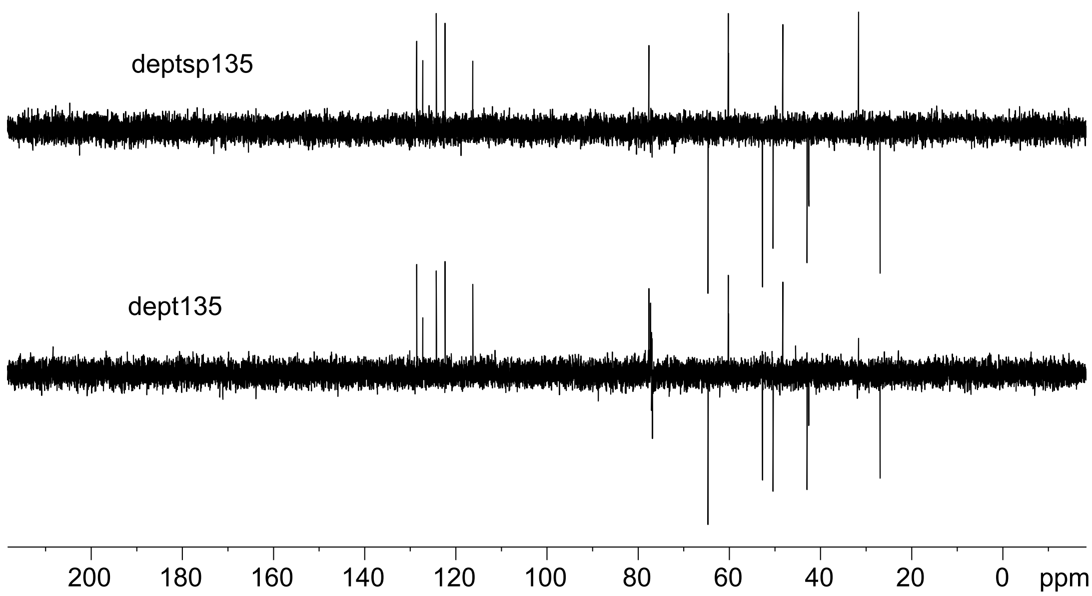
deptsp90



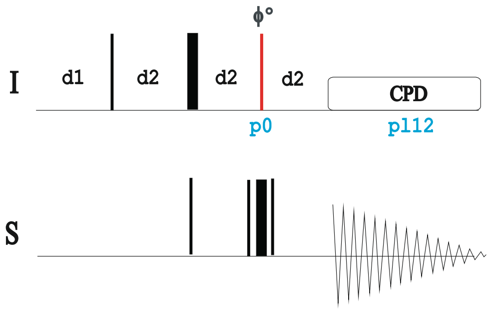
deptsp135



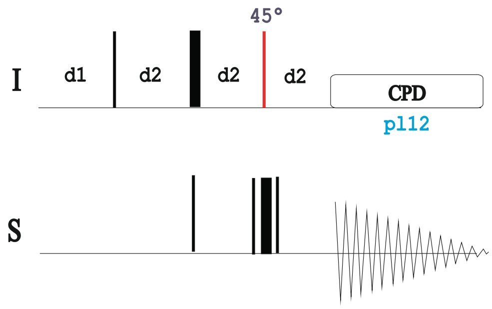
Adiabatic pulse in DEPT:



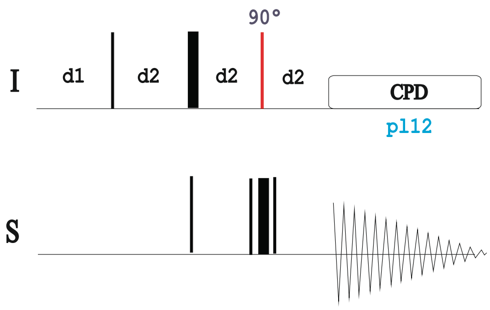
deptcp



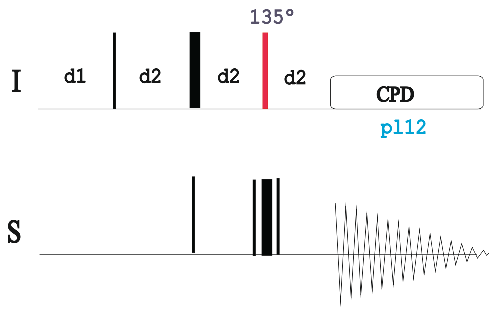
deptcp45



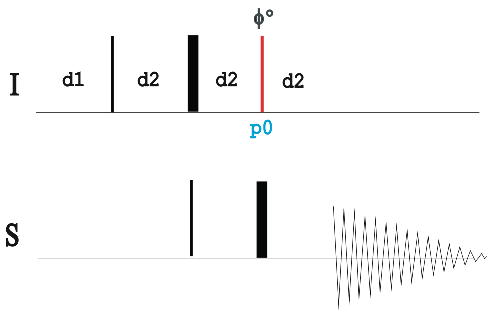
deptcp90



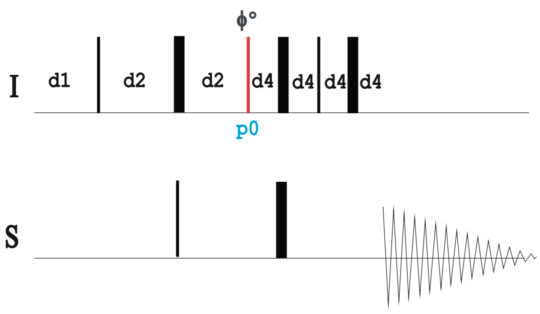
deptcp135



deptnd



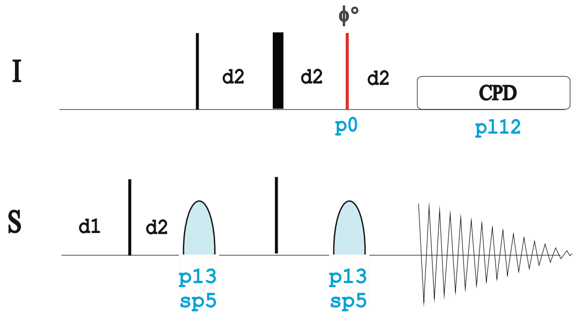
deptppnd



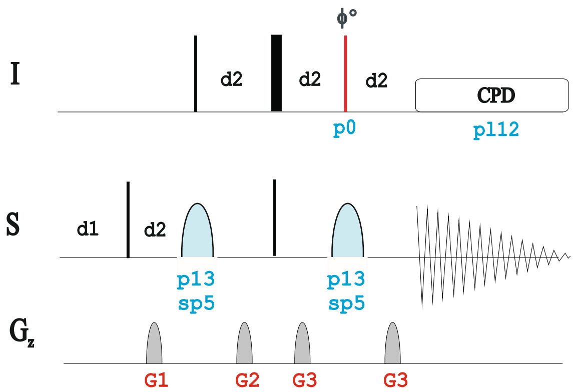
DEPTQ

R. Burger & P. Bigler, J. Magn. Reson. 135, 529-534 (1998)

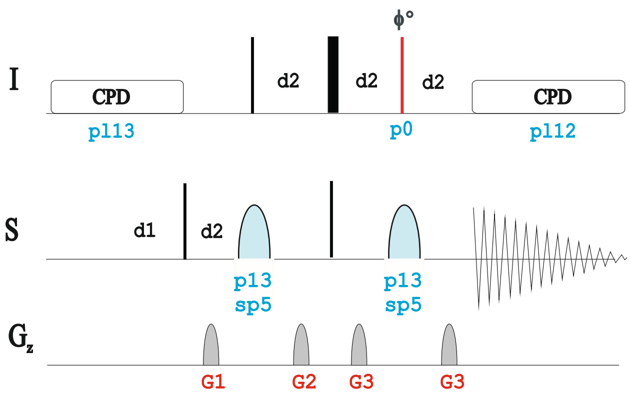
deptqsp



deptqgpsp

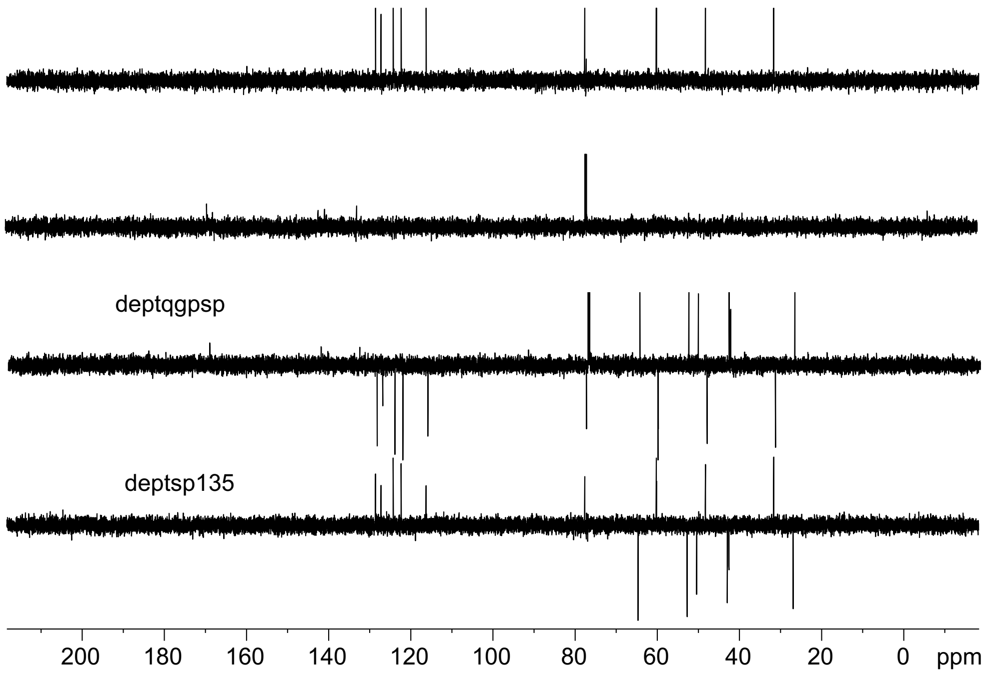


deptqgpsp.2



Gradients in DEPTQ

gp1:gp2:gp3
31:31:31 for all C
0:31:31 for CHn only
31:31:11 for Cquat only



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OTHER 13C EDITING EXPERIMENTS

Other 1D ^{13}C-editing Experiments

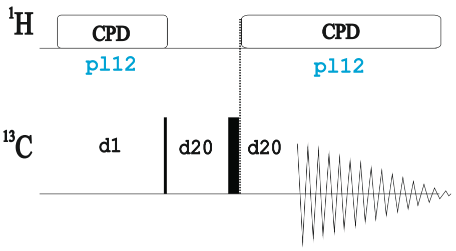
Spin-Echo or SEFT (**jmod**)
Conventional APT (**apt** | C13APT)
APT with J-compensation (**aptjc**)

Quaternary-carbons with decoupling (**quatd**)
Quaternary-carbons without decoupling (**quat**)

Also see:
1D INEPT and 1D DEPT experiments

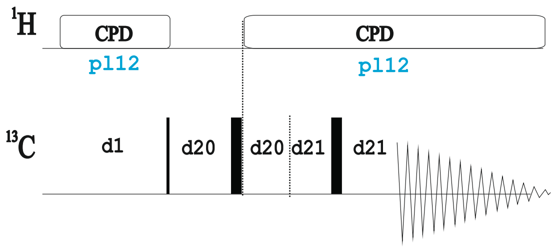
APT: S.L. Patt & J.N. Shoolery, J. Magn. Reson. 46, 535-539 (1982)
ATJ-J Compensated: A.M. Torres, T.T. Nakashima & R.E.D. McClung, J. Magn. Reson. A101, 285-294 (1993)
QUAT: M.R. Bendall & D.T. Pegg, J. Magn. Reson. 53, 272 (1983)

jmod

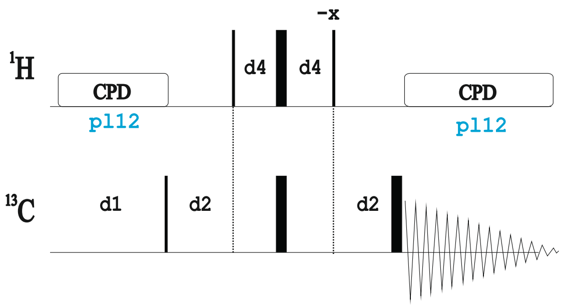


d20: 1/(J(XH)) X, XH2 positive, XH, XH3 negative
1/(2J(XH)) X only

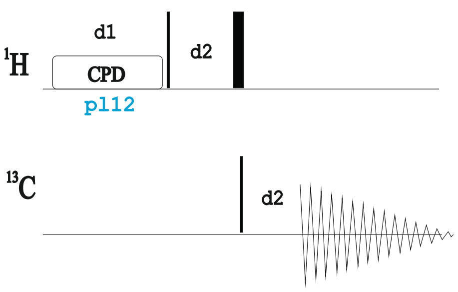
apt



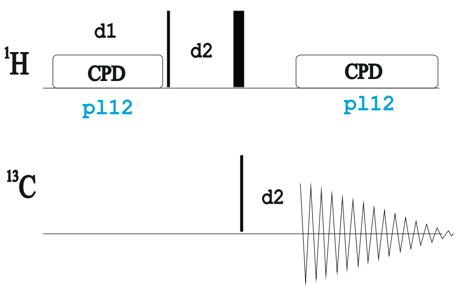
aptjc



quat



quatd



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2D X-DETECTED HETCOR EXPERIMENTS

Experiment Description

The HETCOR (Heteronuclear CORrelation) experiment is a 2D version of the 1D INEPT or DEPT pulse schemes. Therefore, it is a X-detected experiment providing heteronuclear correlation between 1H and X heteronuclei via the scalar coupling constant, J(XH)

Sample Requirements

HETCOR experiments can be recorded on any type of sample but sensitivity suffers of the low receptivity o the detected X nucleus

Hardware Requirements

X-1H HETCOR experiments can be recorded on any probehead that can be tuned to X but a direct probe is recommended.

NMR Spectrum

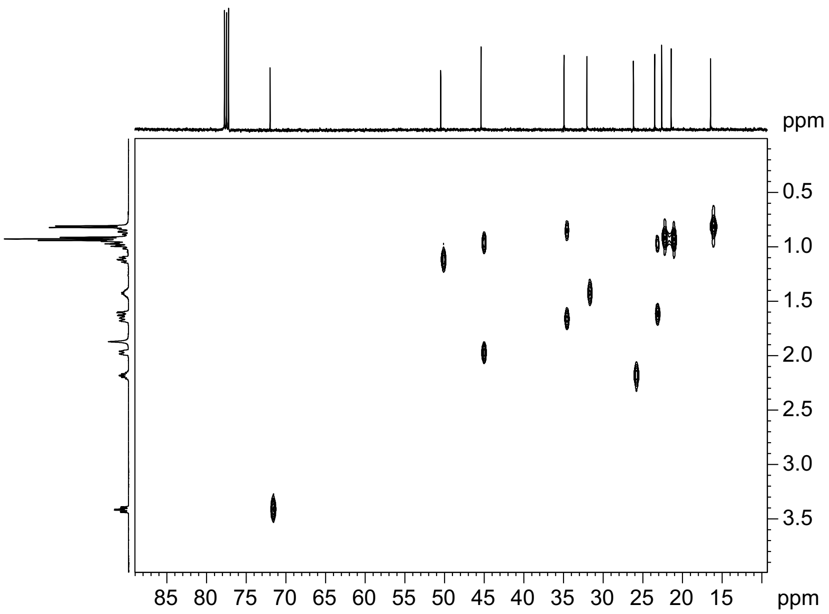
A 2D HETCOR map correlates 1H and X chemical shifts via 1J(XH).

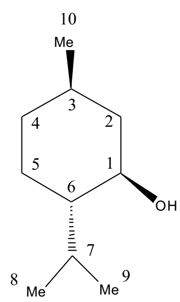
Related Experiments

1D INEPT and DEPT experiments
2D HMQC and HSQC correlation experiments
2D Inverse-INEPT experiment
2D COLOC experiment

References:

A. Bax & G.A. Morris, J. Magn. Reson. 42, 501 (1981)
A. Bax, J. Magn. Reson. 53, 517 (1983)
V. Rutar, J. Magn. Reson. 58, 306 (1984)
J.A. Wilde & P.H. Bolton, J. Magn. Reson. 59, 343 (1984)





2D HETCOR Experiments

- **INEPT-Based HETCOR**
  - Magnitude-mode 2D HETCOR (**hxcoqf** | HCCOSW)
  - Magnitude-mode 2D HETCOR with 2H-decoupling (**hxcoqf2h**)
  - Magnitude-mode 2D HETCOR using composite pulses (**hxcocpqf**)
  - Magnitude-mode 2D HETCOR with ^{1}H-^{1}H decoupling in F1 using BIRD (**hxcobiqf**)
  - Magnitude-mode 2D HETCOR with ^{1}H-^{1}H decoupling in F1 using BIRD and composite pulses (**hxcobicpqf**)
  - Magnitude-mode 2D HETCOR with refocusing of chemical shifts (**hxinepqf**)
  - Phase-sensitive 2D HETCOR with refocusing of chemical shifts (**hxinepph**)

- **DEPT-based HETCOR**
  - Magnitude-mode DEPT-based 2D HETCOR (**hxdeptqf**)
  - Phase-sensitive DEPT-based 2D HETCOR (**hxdeptph**)
  - Magnitude-mode DEPT-based 2D HETCOR with ^{1}H-^{1}H decoupling in F1 using BIRD (**hxdeptbiqf**)
  - Phase-sensitive DEPT-based 2D HETCOR with ^{1}H-^{1}H decoupling in F1 using BIRD (**hxdeptbiph**)
  - Phase-sensitive DEPT-based TOCSY-HETCOR experiment (**hxdeptmlph**)

- **2D H-relayed HETCOR experiment**
  - Magnitude-mode 2D H-relayed HETCOR (**hhxcoqf** / **hhxcoqf.2**)

- **2D X-relayed HETCOR experiment**
  - Magnitude-mode 2D X-relayed HETCOR (**hxxcoqf**)
  - Also see in 2H-specific Experiments:
    Magnitude-mode 2D HETCOR with ^{2}H-decoupling (**hxcoqf2h**)

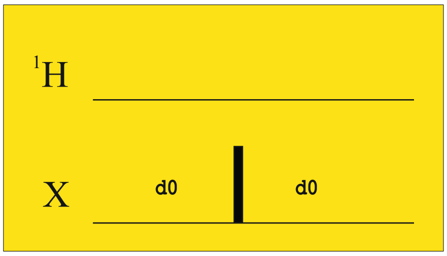
- **2D COLOC experiment**
  - Magnitude-mode 2D COLOC (**colocqf** | HCCOLOCSW)

Also see in 19F-specific Experiments:
2D ^{19}F-^{1}H HETCOR experiment (hfcoqfqn)

Also see in 2D Inverse-INEPT experiments:
Phase-sensitive ge-2D Inverse INEPT using echo-antiecho (xhcoetgp)

NMR BUILDING BLOCK:
1H EVOLUTION and X-DECOUPLING

All HETCOR-type experiments start from 1H and finish with X-detection. The first step is the 1H chemical evolution during the variable d0 period:



Variable period

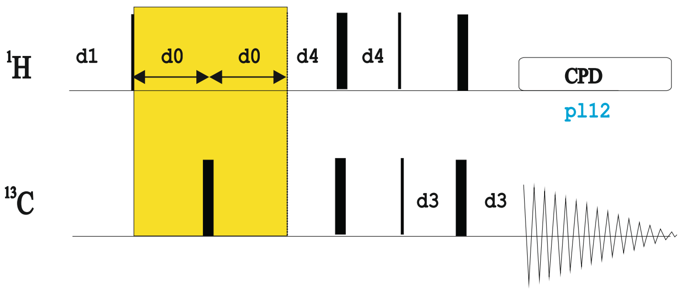
Overall Duration: 2d0

^{1}H Chemical shift: d0 + d0 = 2d0

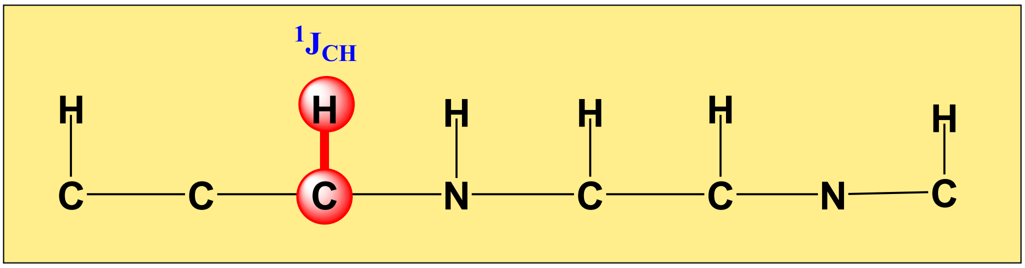
X Chemical shift: d0 - d0 = 0

J(XH) Coupling Constant: d0 - d0 = 0

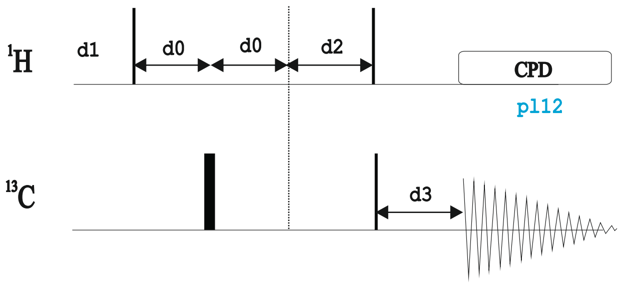
J(HH) and J(XX) Coupling Constant: d0 + d0 = 2d0



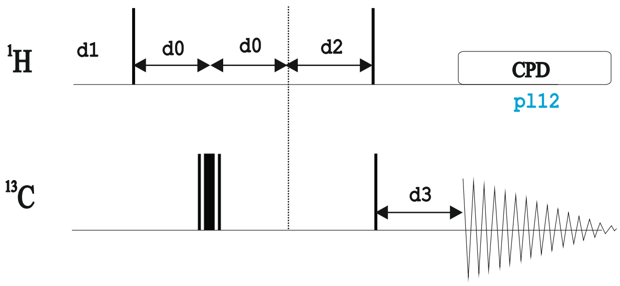

```
 ...
 (p3 ph1):f2
 d0
 p2 ph4
 d0
 d4
 (center (p4 ph2):f2 (p2 ph4) )
 d4
 (p3 ph3):f2 (p1 ph5)
 d3
 (center (p4 ph2):f2 (p2 ph6) )
 d3 pl12:f2
 go=2 ph31 cpd2:f2
 d11 mc #0 to 2 F1QF(caldel(d0, +in0))
....
```



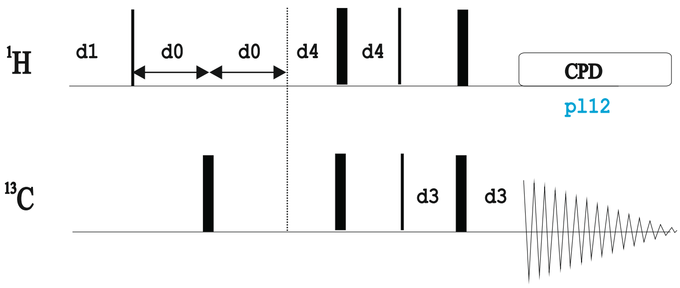
hxcoqf



hxcocpqf



hxinepqf
hxinepph



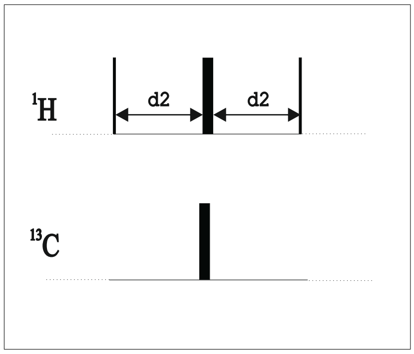
d3:
1/(6J(XH)) XH,XH2,XH3 positive
1/(4J(XH)) XH only
1/(3J(XH)) XH,XH3 positive,XH2 negative

NMR Element:BIRD(x) Element

```
....
  (p3 ph1):f2
  d2
  (center (p2 ph1) (p4 ph1):f2 )
  d2
  (p3 ph1):f2
  d0
 ....

ph1=0

;d2 : 1/(2J(XH))
```

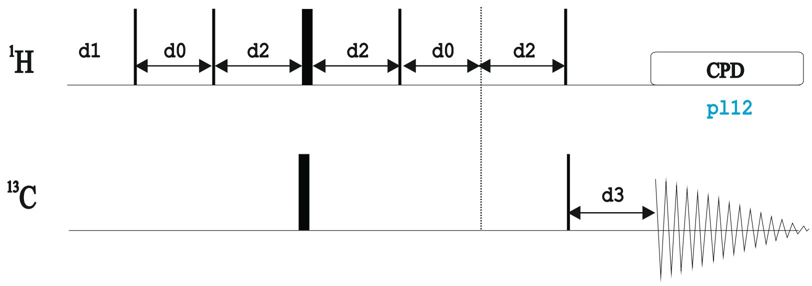


BIRD(x) inverts 1H-13C whereas 1H-12C is not affected:

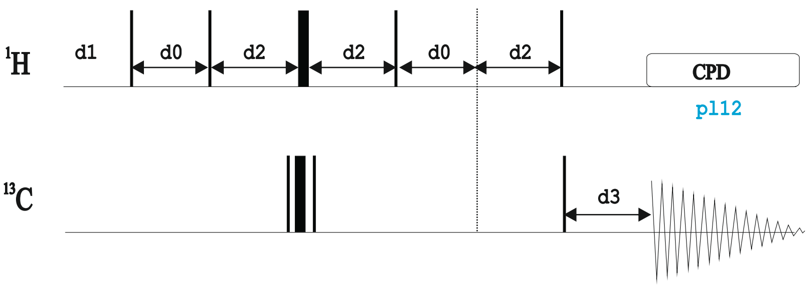
1H-12C: Iz —BIRD(x)→ Iz 1H-13C: Iz —BIRD(x)→ -Iz	or	1H-12C: Ix —BIRD(x)→ Ix 1H-13C: Ix —BIRD(x)→ -Ix

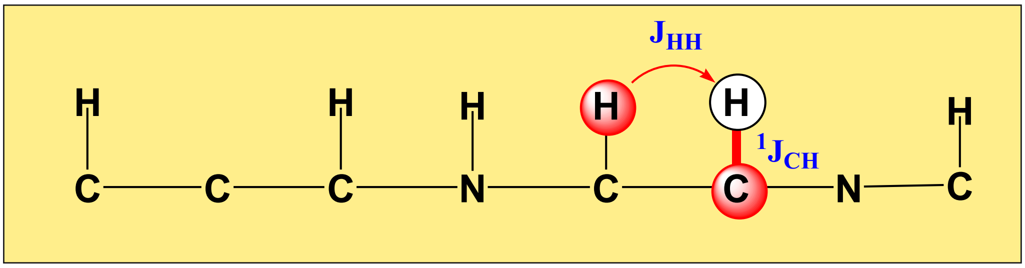
BIRD:
A. Bax and S. Subramanian, J. Magn. Reson. 67, 565-569 (1986)
BIRD-HETCOR: To minimize J(HH) evolution during the d0 period

hxcobiqf



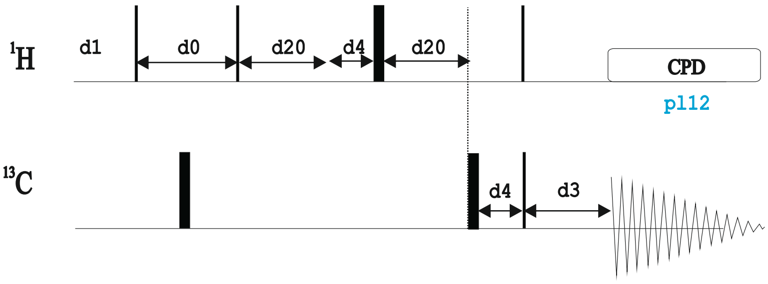
hxcobicpqf



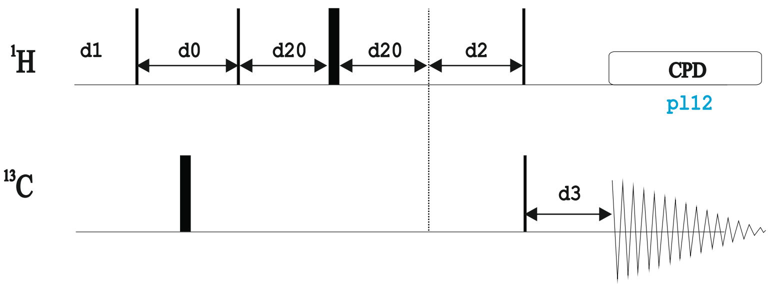


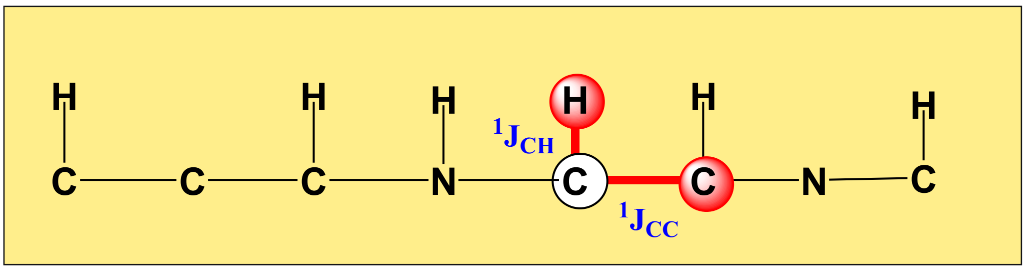
P.H. Bolton, J. Magn. Reson. 48, 336 (1982)
A. Bax, J. Magn. Reson. 53, 149 (1983)
H. Kessler et al., J. Am. Chem. Soc. 105, 6944 (1983)

hhxcoqf



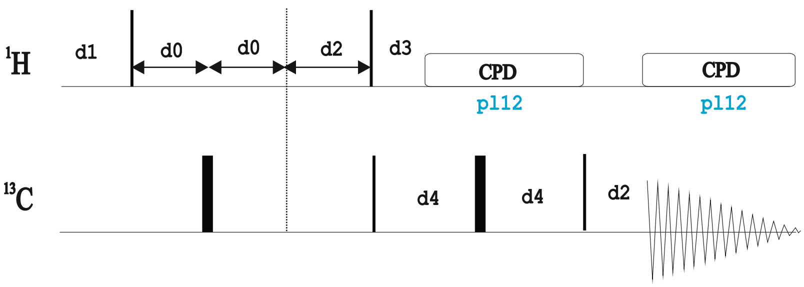
hhxcoqf.2

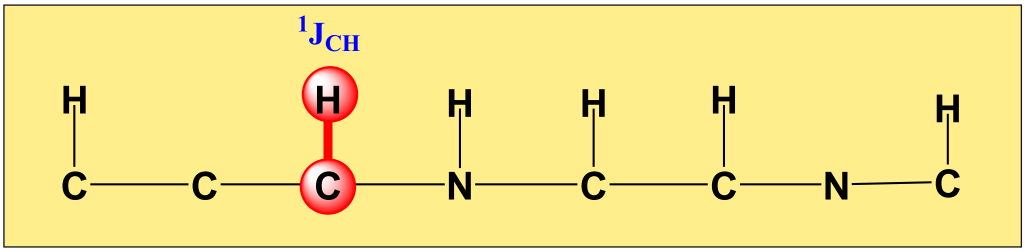




H. Kessler, W. Bermel & C. Griesinger, J. Magn. Reson. 62, 573 (1985)

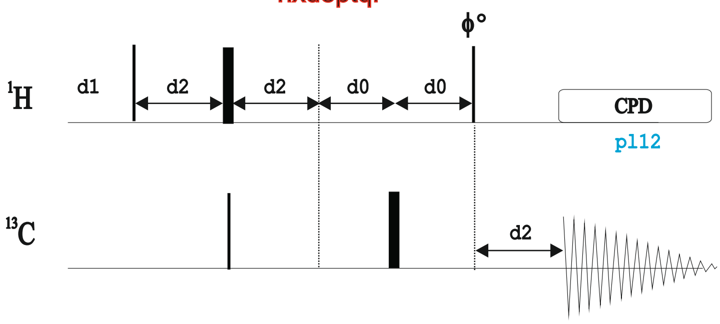
hxxcoqf





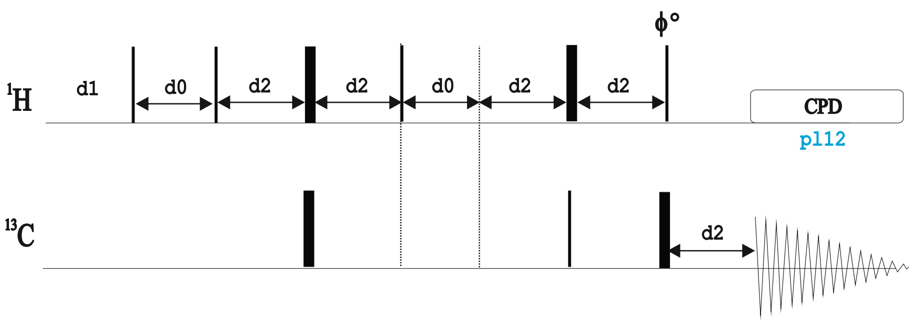
M.R. Bendall & D.T. Pegg, J. Magn. Reson. 53, 144 (1983)
T.T. Nakashima et al., J. Magn. Reson. 59, 124 (1984)

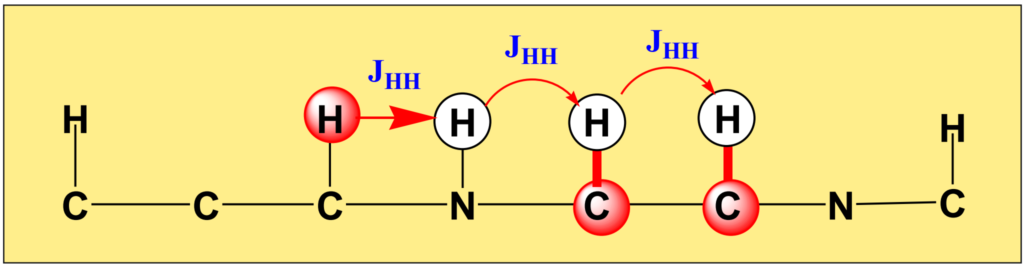
hxdeptph
hxdeptqf



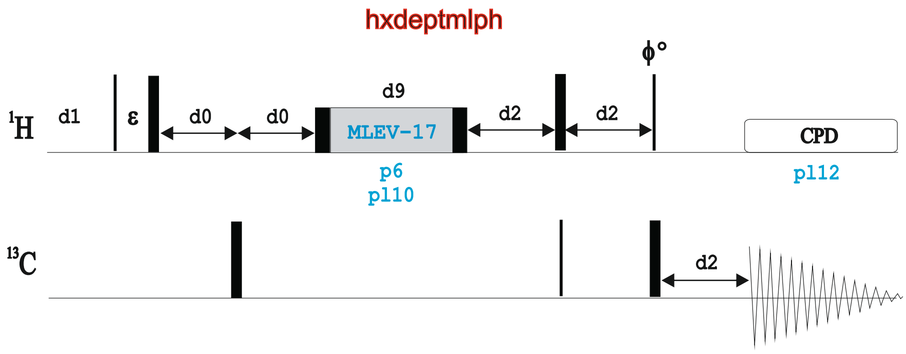
p0
45 degree - all positive
90 degree - XH only
135 degree - XH, XH3 positive, XH2 negative

hxdeptbiph
hxdeptbiqf





H. Kessler, G. Gemmecker, M. Koeck, R. Osowski & P. Schmieder, Magn. Reson. Chem. 28, 62 - 67 (1990)

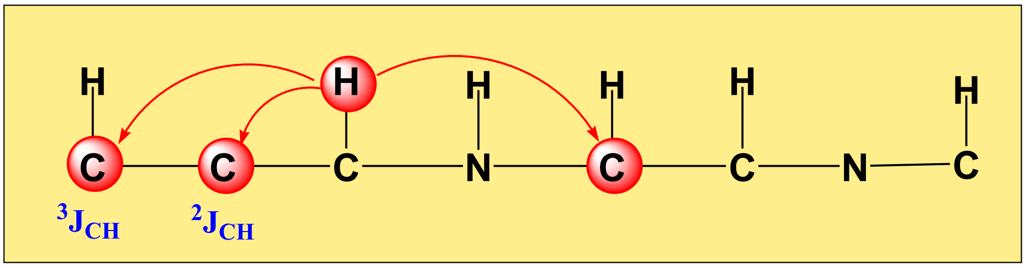


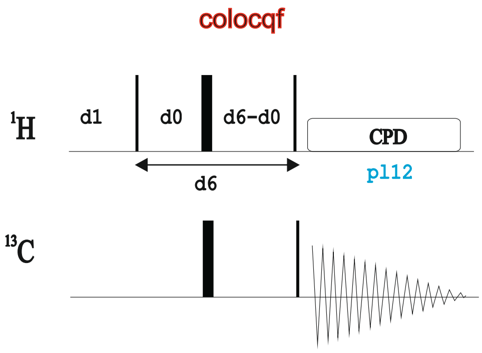
Experiment Description

The COLOC experiment is a X-detected experiment providing long-range heteronuclear correlations between 1H and X heteronuclei via the scalar coupling constant, J(XH). It can be understood as a long-range optimized constant-time version of the HETCOR experiment.

References

H. Kessler et al., J. Magn. Reson. 57, 331 (1984)





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2D HETERONUCLEAR
J-RESOLVED EXPERIMENT

Experiment Description

The HETJRES (Heteronuclear J-Resolved) experiment is the heteronuclear version of the J-Resolved experiment. Basically, it consists of a variable 13C spin-echo period in which J(CH) evolves whereas 13C chemical shift is refocused.

Sample Requirements

Heteronuclear J-Resolved experiments can be recorded on any type of sample but its sensitivity is related to 13C detection.

Hardware Requirements

Heteronuclear J-Resolved experiments can be recorded on any probehead but better sensitivity will be achieved in direct X probes.

NMR Spectrum

A basic Heteronuclear J-Resolved experiment yields a 2D map in which 13C chemical shift is displayed in the F2 dimension whereas J(CH) is displayed in the F1 dimension. Thus, methine carbons will display a doublet, a methylene carbon a triplet, and a methyl carbon a typical quartet.

The proton-selective version allows the measurement of small, long-range proton-carbon coupling constants. The only requisite is a well-isolated proton resonance.

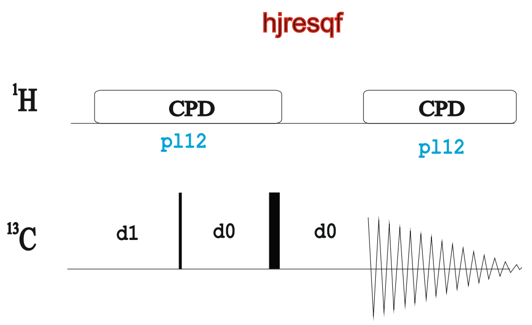
Related Experiments

Homonuclear 2D J-Resolved experiment

2D Heteronuclear J-Resolved Experiments

Magnitude-mode 2D Heteronuclear J-Resolved (**hjresqf**)

Also see chapter: Measurement of long-range proton-carbon coupling constant.
Magnitude-mode proton-selective 2D Heteronuclear J-Resolved (seljresqfsp)



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2D HOESY EXPERIMENTS

Experiment Description

The HOESY (Heteronuclear nOESY) experiment is the heteronuclear version of the conventional NOESY experiment.

Sample Requirements

HOESY experiments can be recorded on any type of sample but its sensitivity is very low.

Hardware Requirements

HOESY experiments can be recorded on any probehead but better sensitivity will be achieved in direct X probes. However, HOESY pulse scheme can be also applied with proton detection in inverse probes.

NMR Spectrum

A HOESY experiment provides a 2D heteronuclear correlation map. Directly-attached CH systems will give a strong peak and the important long-range NOE peaks are usually very weak.

Related Experiments

2D NOESY experiment

References

C. Yu & G. Levy, J. Am. Chem. Soc. 106, 6533 (1984)

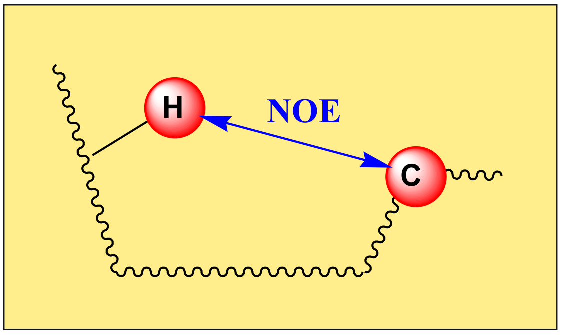
The delay d8 defines the mixing time in HOESY experiments

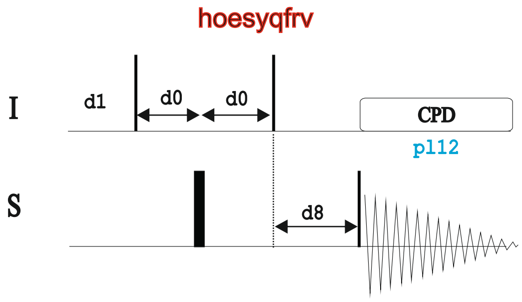
2D HOESY Experiments

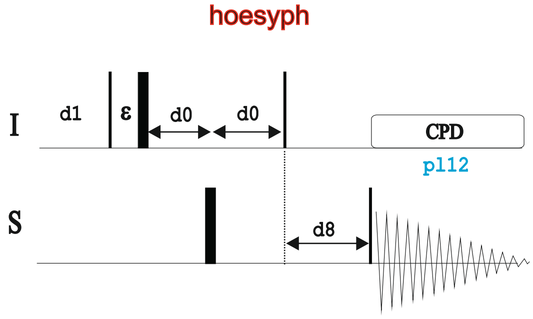
Magnitude-mode 2D ^{1}H-^{13}C HOESY(**hoesyqfrv**)
Phase-sensitive 2D ^{1}H-X HOESY (**hoesyph**)

Also see in Chapter 19F experiments:
2D ^{19}F-^{1}H HOESY experiment (hoesyfhqfqnrv)

Also see:
2D NOESY Experiment







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1D & 2D INADEQUATE
EXPERIMENTS

Experiment Description

The INADEQUATE experiment is the 13C version of the Double-quantum experiment.

Sample Requirements

The INADEQUATE experiment is a very insensitive experiment because two adjacent 13C nuclei are needed. Therefore, a highly concentrated sample is needed for molecules at natural abundance. Otherwhise, 13C-labeled material is highly recommended.

Hardware Requirements

The INADEQUATE experiment can be recorded on any probehead but better sensitivity will be achieved in direct X probes.

NMR Spectrum

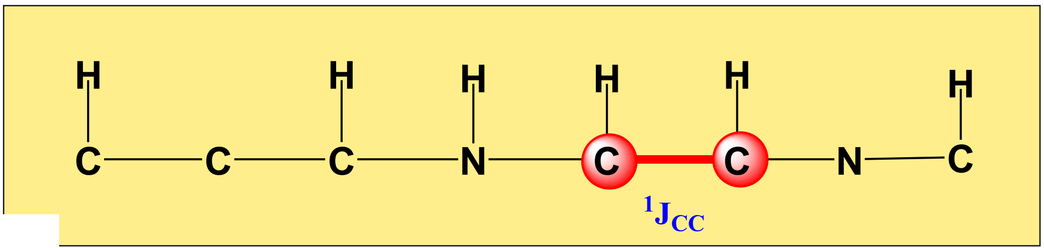
As similar to the DQ experiment, the INADEQUATE affords a 2D map correlating 13C chemical shift in F2 and double-quantum frequencies (13Ca+13Cb) in the indirect F1 dimension.

Related experiments

1D and 2D Multiple-quantum experiments

References

D.L. Turner, J. Magn. Reson. 49, 175 (1982)
M. Bourdonneau & B. Ancian, J. Magn. Reson. 132, 316-327 (1998)



The delay d4 is optimized to 1/4J(CC).

2D INADEQUATE Experiments

- <u>1D INADEQUATE</u>

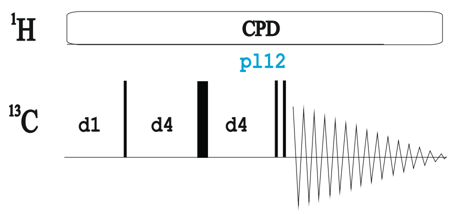
  1D INADEQUATE without refocusing (**inad1d**)
  1D INADEQUATE using composite pulses (**inadcp1d**)
  1D INADEQUATE with refocusing (**inadrd1d**)
  1D INADEQUATE using initial INEPT (**inepin**)

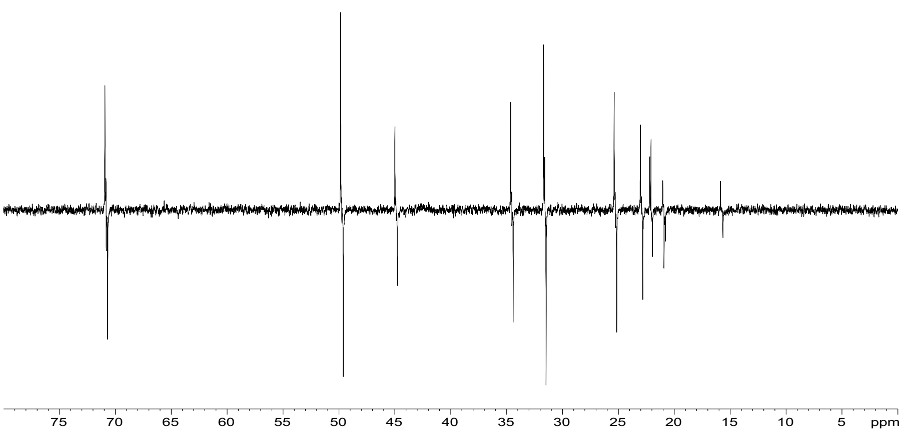
- <u>2D INADEQUATE</u>

  Magnitude-mode 2D INADEQUATE (**inadqf**/ **inadqf.2** | INAD)
  Phase sensitive 2D INADEQUATE(**inadph**)
  Phase sensitive 2D INADEQUATE using adiabatic pulse (**inadphsp**)
  Magnitude-mode symmetric 2D INADEQUATE(**inadqfsy**)
  Magnitude-mode 2D INADEQUATE using adiabatic pulse and gradients (**inadgpqfsp**)

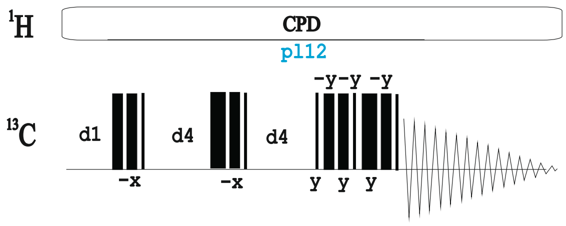
Also see: 1D & 2D Multiple-Quantum Experiments

inad1d

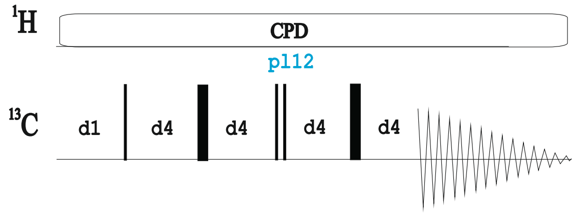


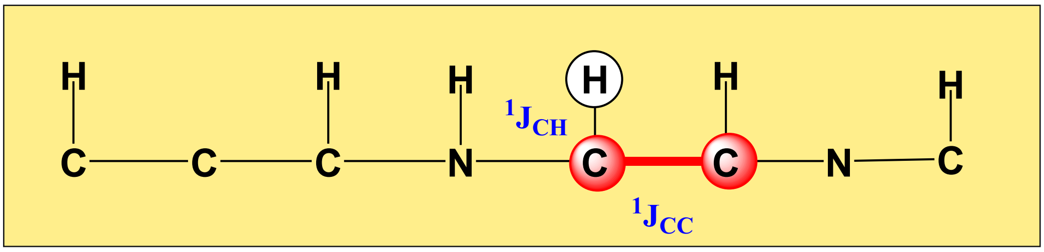


inadcp1d



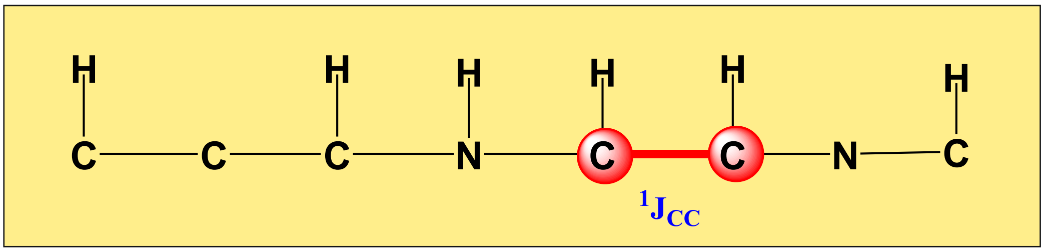
inadrd1d



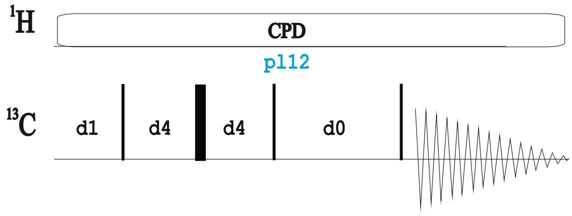


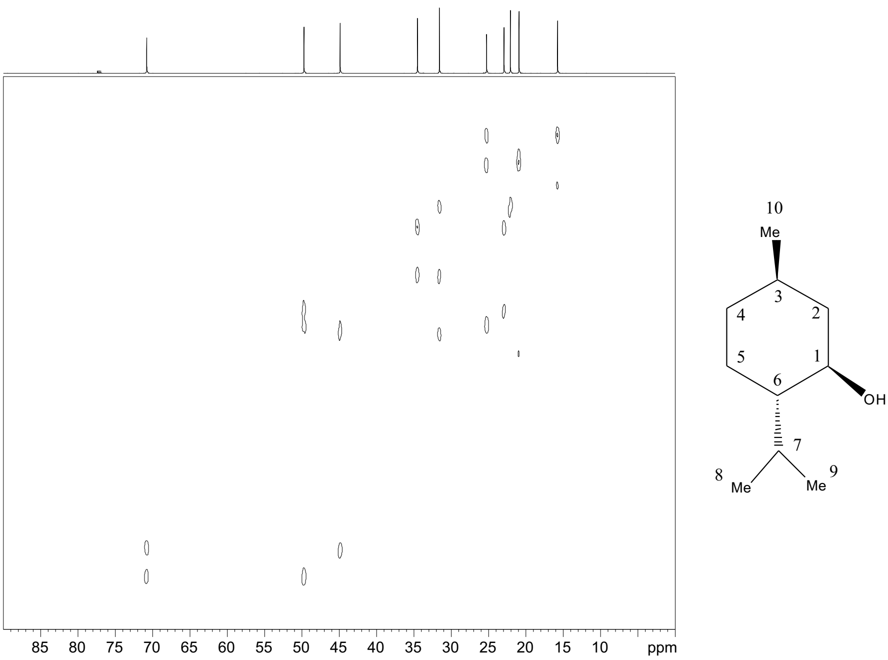
inepin

Also see ineptrl1

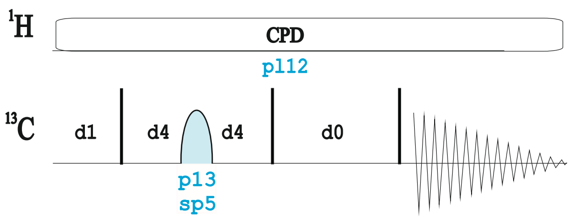


inadqf.2
inadph
inadqf

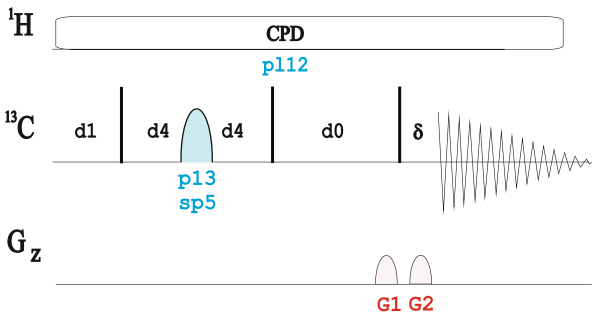




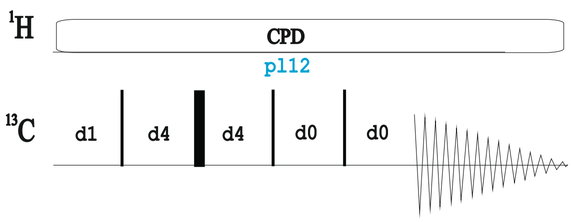
inadphsp



inadphsp



inadqfsy



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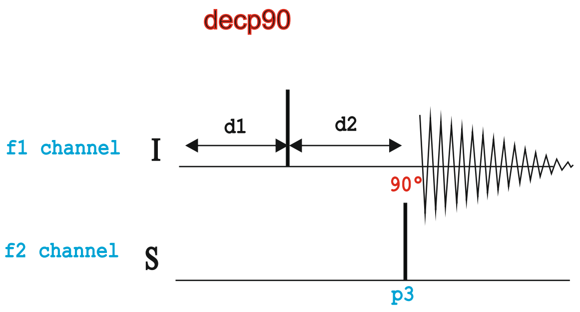
DECOUPLER PULSE CALIBRATION

Experiment Description:

In heteronuclear experiments is necessary to calibrate the duration of the hard 90/180 pulses applied from the decoupler (the non-observed nucleus) as well as the required power level for effective CPD decoupling. A series of simple pulse sequences are used for these purposes. Variants of these experiments also allows to calibrate duration and power levels for shaped pulses as, for instance, adiabatic pulses.

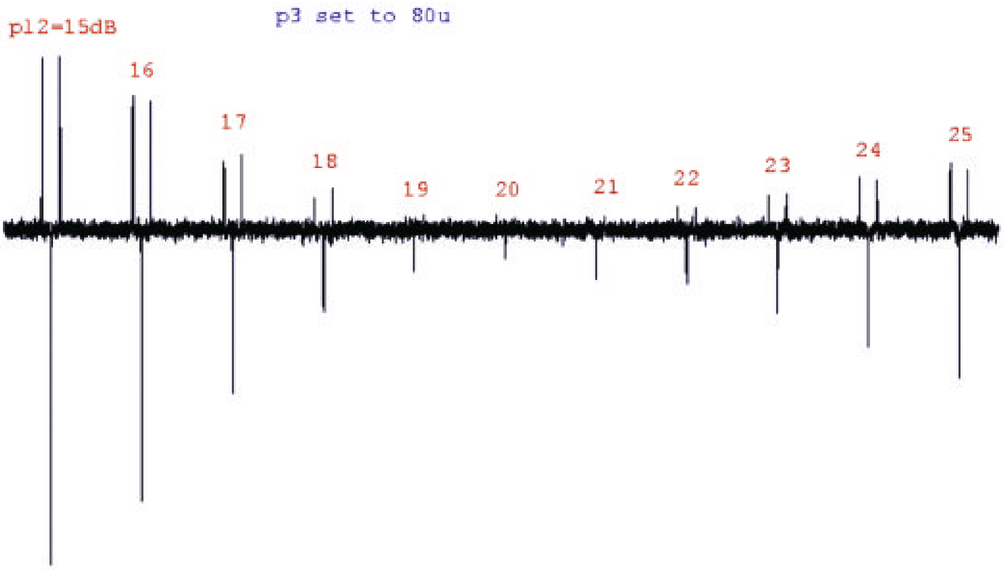


Example: Determination 90 decoupler pulse needed for 1H decoupling (using WALTZ16) whereas observing 13C:

Basic acquisition parameters:
Sample: ASTM (60% C6D6 / 40% p-Dioxane)
Spin: on, d1 45s, ns 1 and ds 0, o1p 66.5ppm; o2p=3.7ppm, CNST2=142Hz; d2=3.52ms

Process with ef (lb=3.5Hz)

Analysis: Without decoupling we observe an antiphase triplet, with the two outer lines in phase and the center line in opposite phase. Set p3=80us and start with a low power pl2=30dB. Increase pl2 using **popt** until the triplet becomes zero. These values will be used for CPD 1H decoupling experiments.



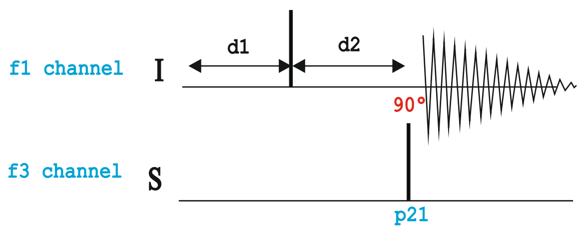
Decoupler Pulse Calibration

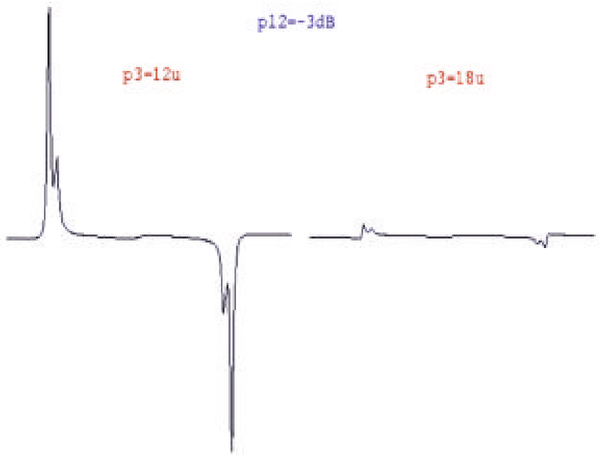
Calibration of the 90 decoupler pulse (**decp90**, **decp90f3**)
Calibration of the 90 decoupler shaped pulse (**decp90sp**)

Calibration of the 180 decoupler pulse (**dec180**)
Calibration of the shaped 180 decoupler pulse (**dec180sp**)
Calibration of the 180 decoupler pulse using presaturation (**dec180pr**, **dec180f3pr**)

Also See:
Pulse Calibration and Tests

decp90f3





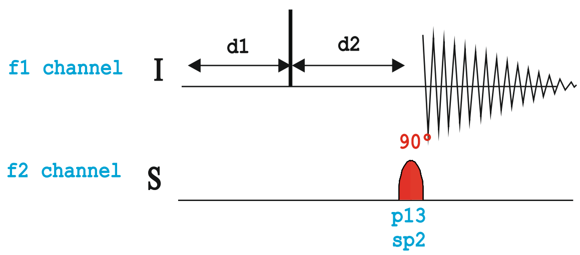
<u>Example: Determination 90 decoupler pulse needed for 15N whereas observing 1H:</u>

Basic acquisition parameters:
Sample: 100mM urea 15N, 100mM Ch3OH 13C in DMSO-d6
Spin: off, d1 24s, ns 1 and ds 0, o1p=4ppm o2p=76ppm sw=8ppm,CNST2=88.5Hz (d2=5.649ms)

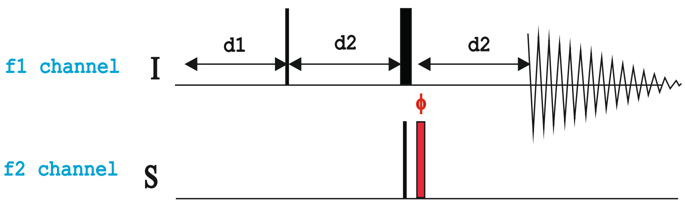
Process with ef (lb=0.3Hz)

Analysis: Without decoupling we observe an antiphase doublet. Increase p3 (at power level pl2) until the doublet becomes zero. For three-channel system use pulprog=decp90f3.

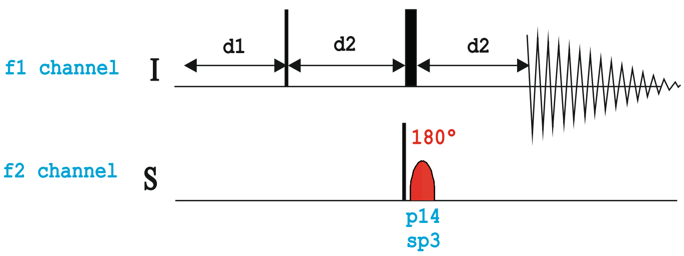
decp90sp



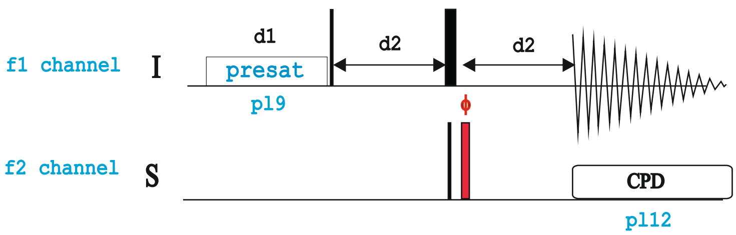
dec180



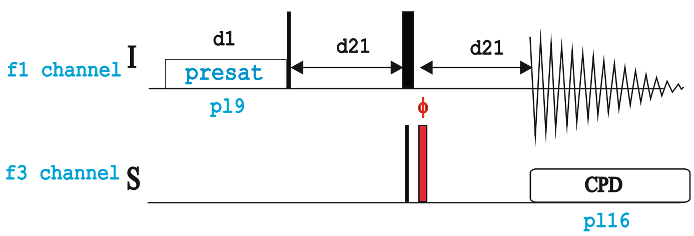
dec180sp



dec180pr



dec180f3pr



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2D HMQC EXPERIMENT

Experiment Description

The HMQC (Heteronuclear Multiple-Quantum Correlation) experiment is a proton-detected experiment designed to obtain heteronuclear correlation between 1H and X heteronuclei via the scalar coupling constant, 1J(XH)

Sample and Hardware Requirements

HMQC experiments can be recorded on any type of sample. It can be recorded on any probehead but an inverse probe equipped with gradients is strongly recommended.

NMR Spectrum

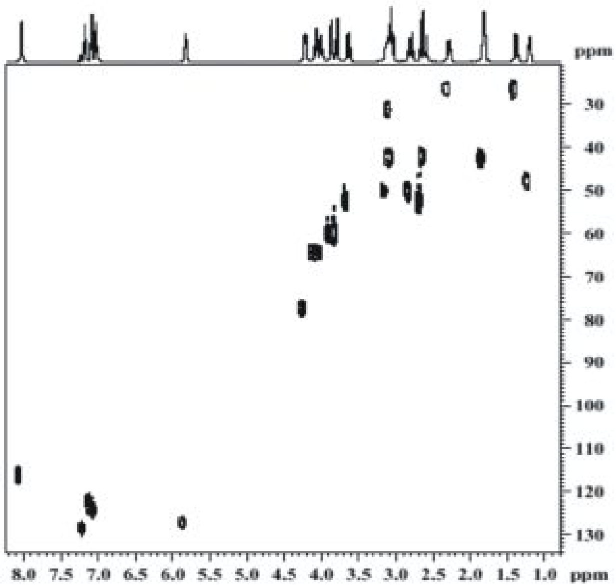
A 2D HMQC map correlates 1H and X chemical shifts via 1J(XH).

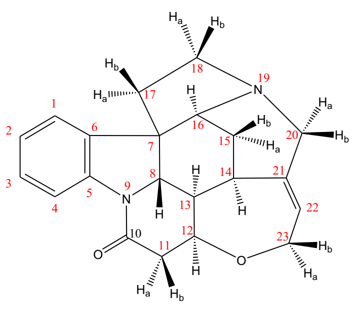
Related Experiments

1D Inverse, 2D HSQC and 2D HMBC experiments
Also see other 2D HMQC-related experiments: HMQC-TOCSY, HMQC-NOESY ...

References

1. A. Bax, R.H. Griffey & B.L. Hawkins, J. Magn. Reson. 55, 301 (1983)
2. A. Bax and S. Subramanian, J. Magn. Reson. 67, 565-569 (1986)
3. V. Sklenar & A. Bax, J. Magn. Reson. 74, 469 (1987)
4. A.G. Palmer III, J. Cavanagh, P.E. Wright & M. Rance,J. Magn. Reson. 93, 151-170 (1991)





2D HMQC Experiments

- **Phase-cycled:**

Magnitude-mode 2D HMQC (**hmqcqf** | HMQC)
Magnitude-mode 2D HMQC without decoupling (**hmqcndqf**)
Magnitude-mode 2D HMQC using BIRD (**hmqcbiqf** | HMQCBI)
Magnitude-mode 2D HMQC using BIRD without decoupling (**hmqcbindqf**)
Phase-sensitive 2D HMQC (**hmqcph** | HMQCPH)
Phase-sensitive 2D HMQC without decoupling (**hmqcndph**)
Phase-sensitive 2D HMQC using BIRD (**hmqcbiph** | HMQCBIPH)
Phase-sensitive 2D HMQC using BIRD without decoupling (**hmqcbindph**)

- **Phase-cycled and solvent suppression**

From f2 channel:
Phase-sensitive 2D HMQC with presaturation (**hmqcphpr** | HMQCPHPR)
Phase-sensitive 2D HMQC using BIRD and presaturation (**hmqcbiphpr**) / **hmqcbiphpr2**)
Phase-sensitive 2D HMQC with 1-1 water suppression (**hmqcph11**)
From f3 channel:
Phase-sensitive 2D ^{1}H-^{15}N HMQC (**hmqcf3ph**)
Phase-sensitive 2D ^{1}H-^{15}N HMQC using presaturation (**hmqcf3phpr**)
Phase-sensitive 2D ^{1}H-^{15}N HMQC using BIRD (**hmqcbif3ph**)
Phase-sensitive 2D ^{1}H-^{15}N HMQC using decoupling in a third f2 channel (**hmqcfbph**)

- **Gradient-based:**

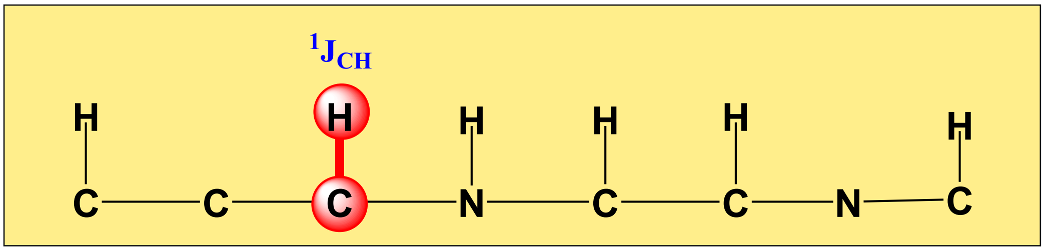
From f2 channel:
Magnitude-mode ge-2D HMQC (**hmqcgpqf** | HMQCGP)
Phase-sensitive ge-2D HMQC using echo-antiecho (**hmqcetgp**)
Phase-sensitive ge-2D HMQC using echo-antiecho with adiabatic refocusing (**hmqcetgp.2**)
Phase-sensitive ge-2D HMQC using PEP (**hmqcetgpsi**)
Phase-sensitive ge-2D HMQC using PEP and shorter overall timing (**hmqcetgpsi.2**)
From f3 channel:
Phase-sensitive ge-2D ^{1}H-^{15}N HMQC using echo-antiecho (**hmqcetf3gp**)
Phase-sensitive ge-2D ^{1}H-^{15}N HMQC using PEP (**hmqcetf3gpsi**)
Phase-sensitive ge-2D ^{1}H-^{15}N HMQC using PEP and shorter overall timing(**hmqcetf3gpsi.2**)

- **Gradient-based and solvent suppression**

Phase-sensitive ge-2D ^{1}H-^{15}N HMQC using WATERGATE (3-9-19) (**hmqcf3gpph19**)
Phase-sensitive ge-2D ^{1}H-^{15}N HMQC using WATERGATE (3-9-19) in the middle of t1 (**hmqcf3gpph19.2**)
Phase-sensitive ge-2D ^{1}H-^{15}N HMQC using WATERGATE (selective 90 pulses) in the middle of t1 (**hmqcf3gpphwg**)

Also see in chapter "SOFAST/BEST experiments":
NH-selective 2D Sofast-HMQC experiment for rapid pulsing (sfhmqcf3gpph | SFHMQCF3GPPH)
NH-selective 2D Sofast-HMQC with inversion of water/aliphatic protons (hetsfhmqcf3gpph)
NH-selective 2D Sofast-HMQC with sensitivity improved (sfhmqcf3gpphiasi)

Also see other related correlation experiments: 2D HMQC-DEPT, 2D HSQC , 2D CT-HMQC...

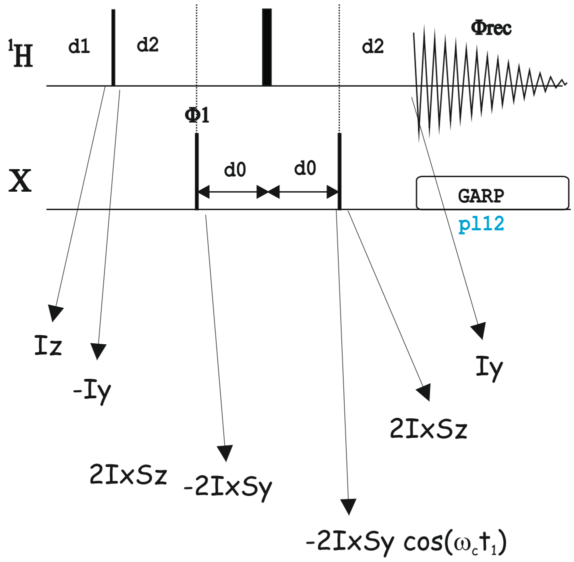


NMR Building Block: Generation of Heteronuclear Multiple-Quantum Coherences

```
...
2 d1
  d12 pl1:f1 pl2:f2
3 p1 ph1
  d2 pl2:f2
  p3:f2 ph3
....
ph1=0
ph3=0

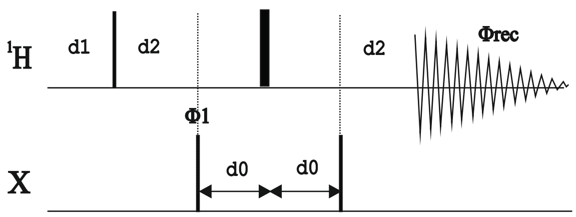
;d2 : 1/(2J)XH
```

hmqcqf
hmqcph

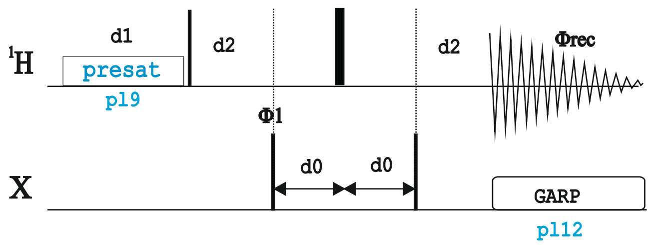


Evolution of **Multiple-Quantum** Coherences!!!

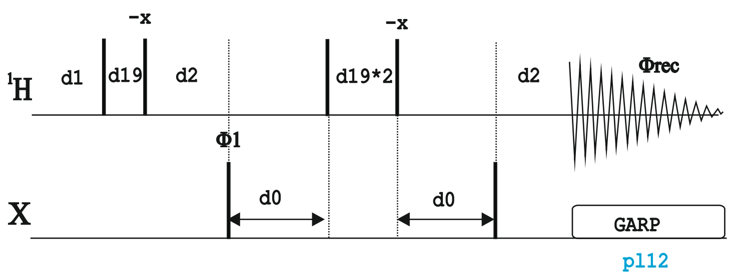
hmqcndph
hmqcndqf



hmqcphpr



hmqcph11

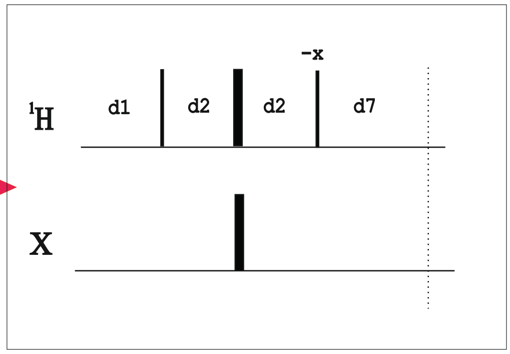


NMR Element: BIRD(-x)-Recovery Delay Element

```
 ....
 p1 ph1
 d2
 (center (p2 ph1) (p4 ph1):f2 )
 d2
 p1 ph2
 d7
 ....


ph1=0
ph2=2

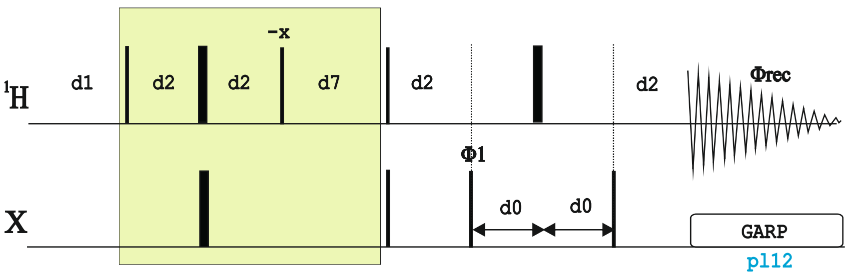
;d2 : 1/(2J)XH
;d7 : delay for inversion recovery
;     optimize to give null for protons bound to C-12
```



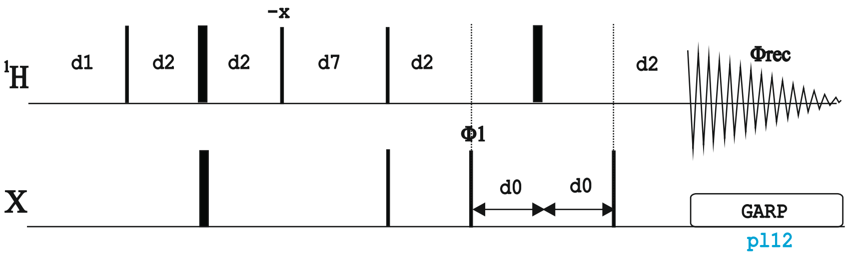
BIRD(-x) inverts 1H-12C whereas 1H-13C is not affected:

1H-12C: Iz	BIRD(-x) →	-Iz	d7=null point →	Iz=0	90° →	0
1H-13C: Iz	BIRD(-x) →	Iz	d7=null point →	Iz	90° →	-Iy

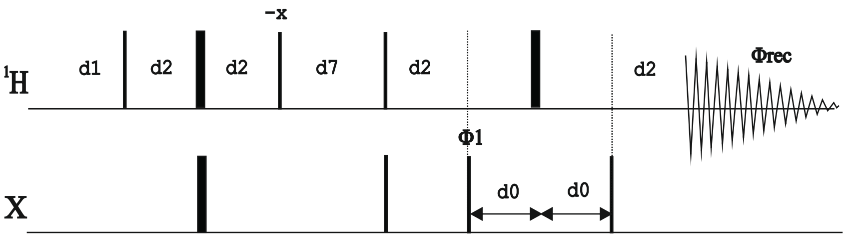
BIRD:
A. Bax and S. Subramanian, J. Magn. Reson. 67, 565-569 (1986)



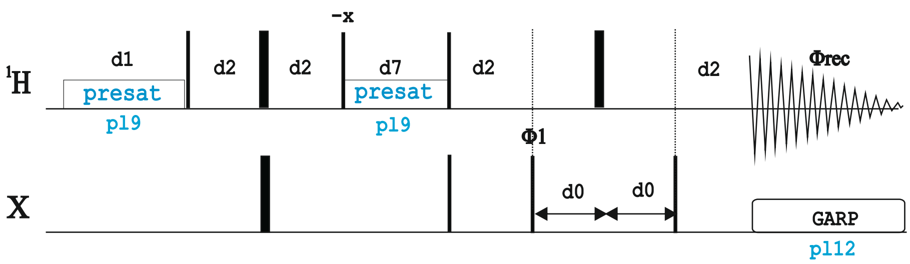
hmqcbiph
hmqcbiqf



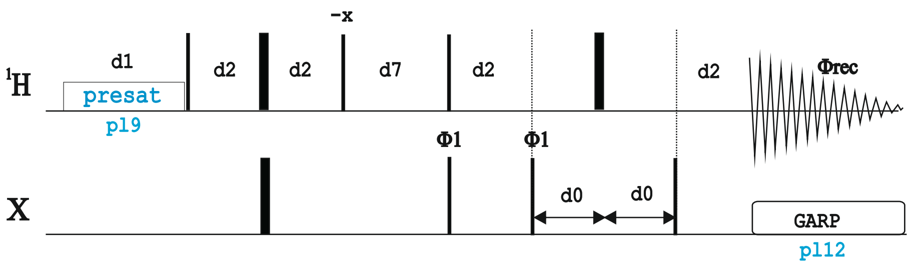
hmqcbindph
hmqcbindqf

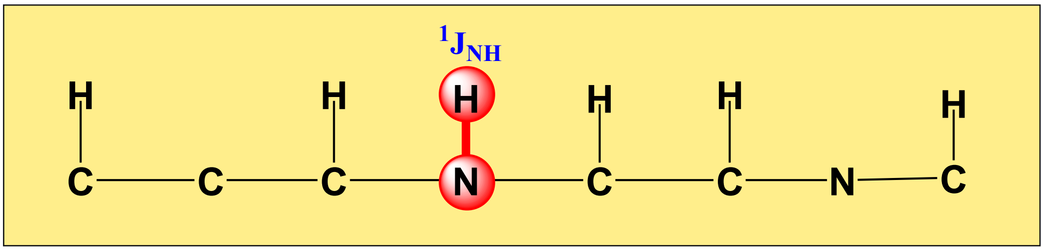


hmqcbiphpr

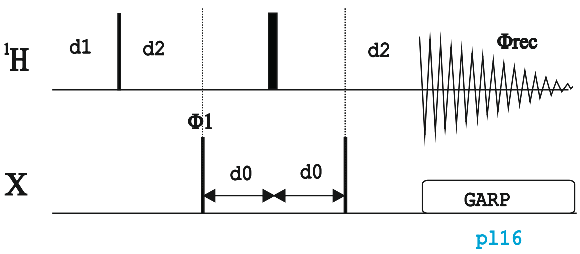


hmqcbiphpr2

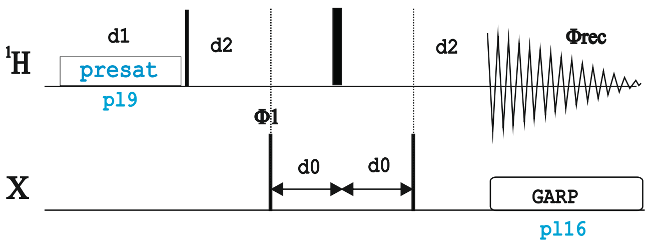




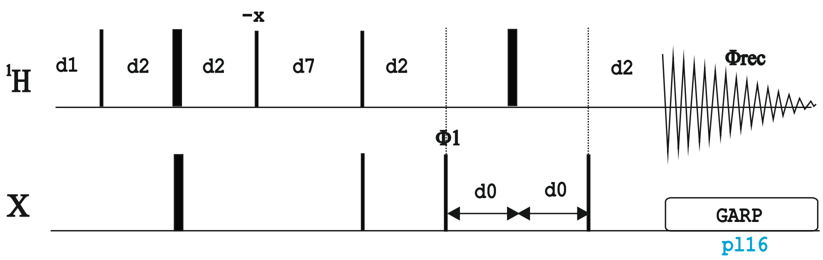
hmqcf3ph



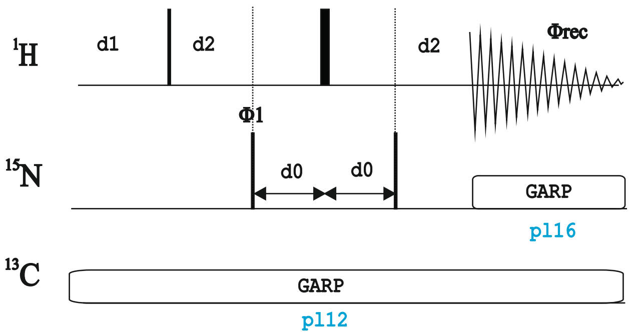
hmqcf3phpr



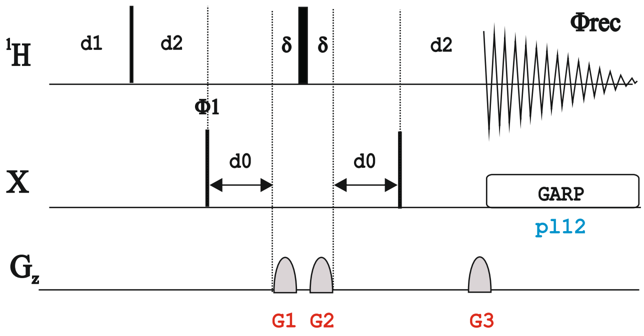
hmqcbif3ph



hmqcfbph

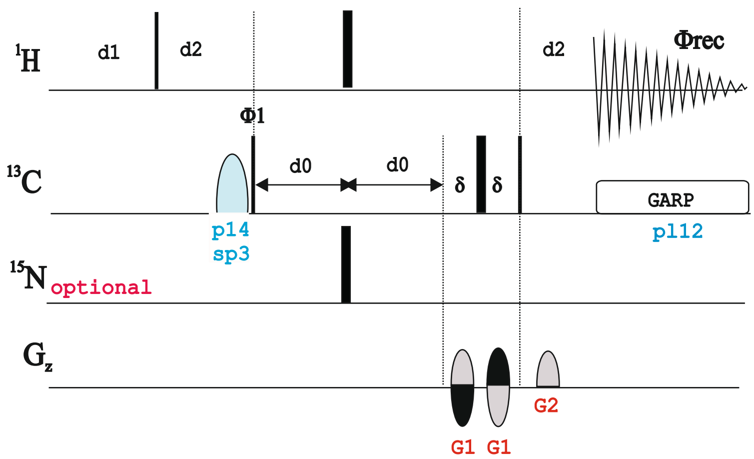


hmqcgpqf

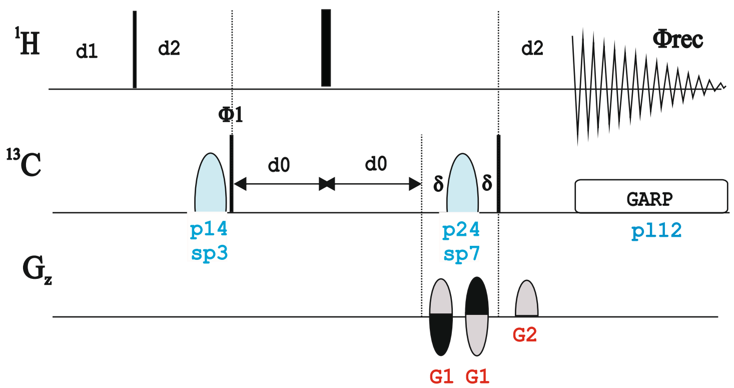


gp 1 : gp 2 : gp 3		
50 :	30 : 40.1	for C-13
70 :	30 : 50.1	for N-15

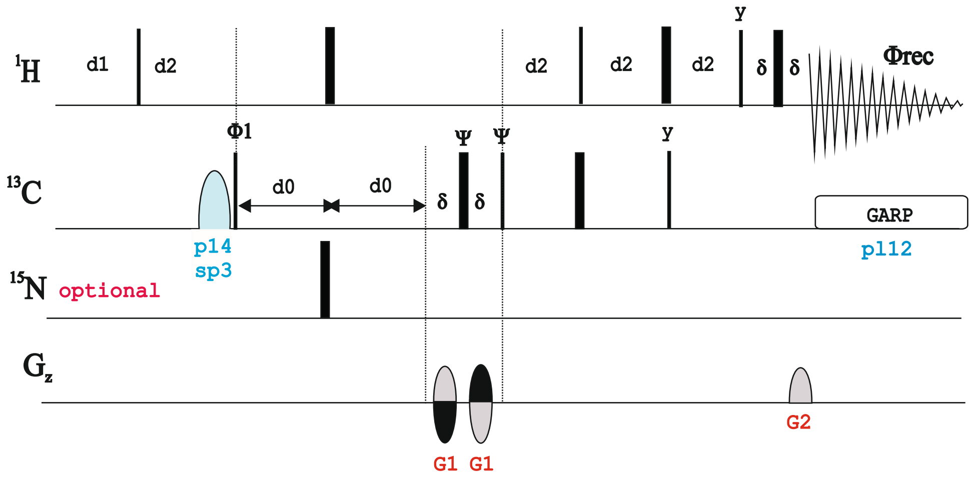
hmqcetgp



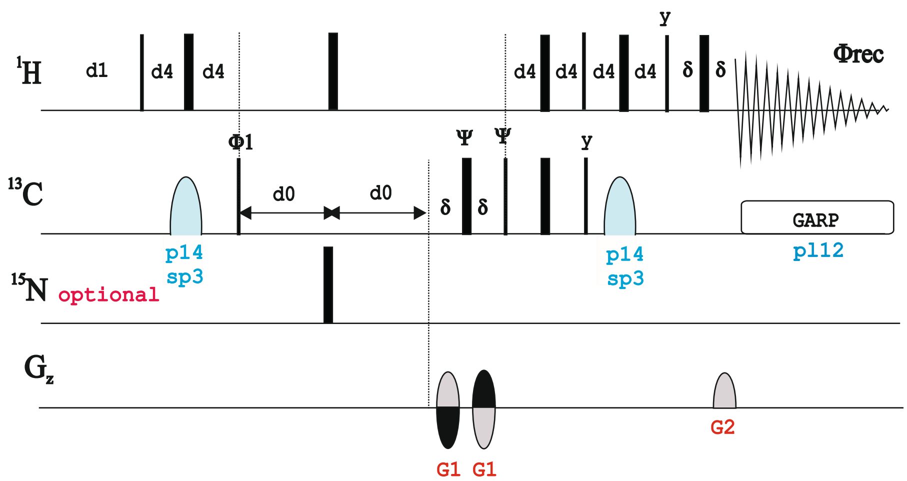
hmqcetgp.2



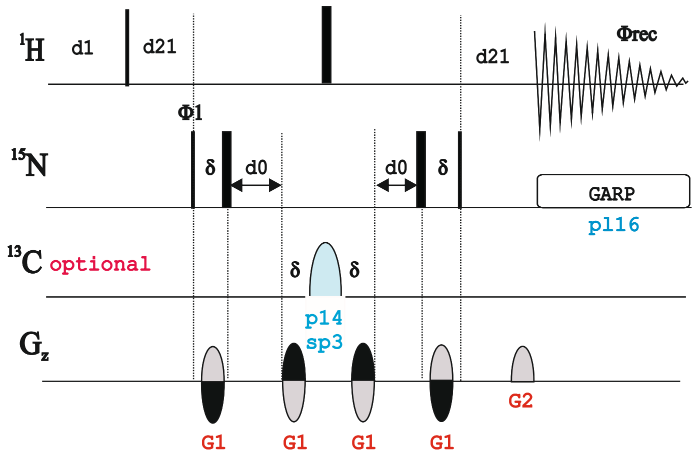
hmqcetgpsi



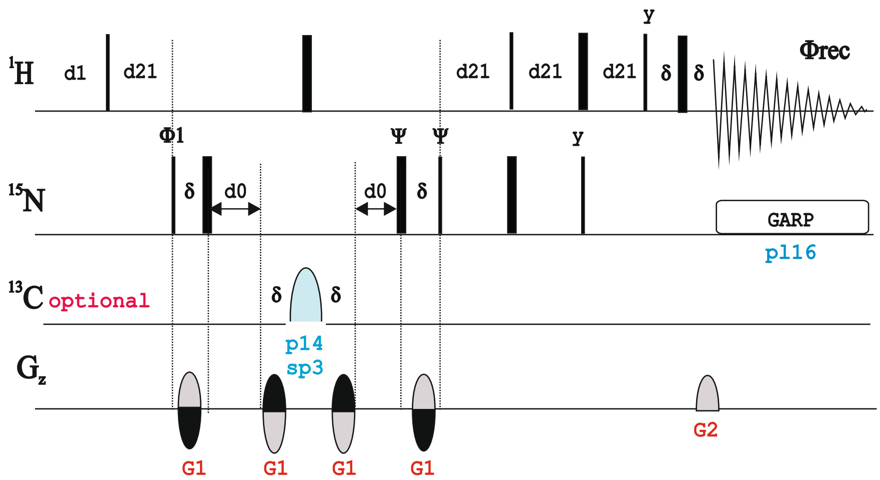
hmqcetgpsi.2



hmqcetf3gp



hmqcetf3gpsi



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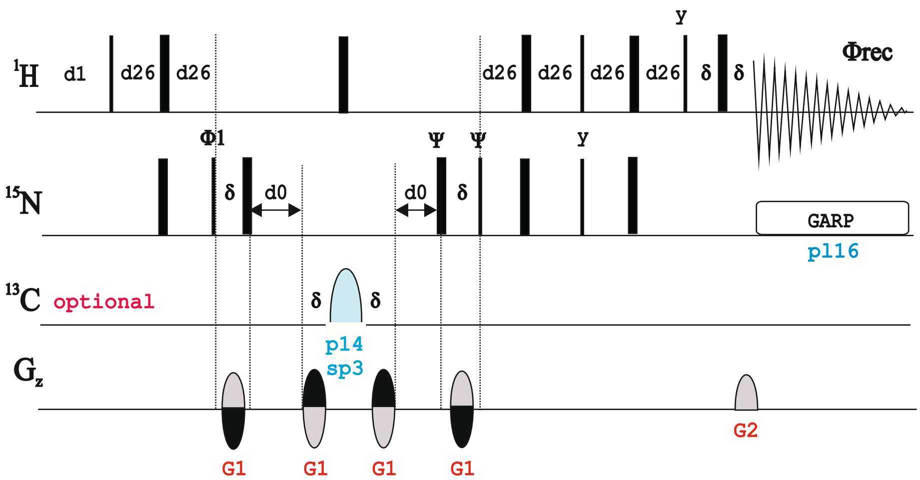
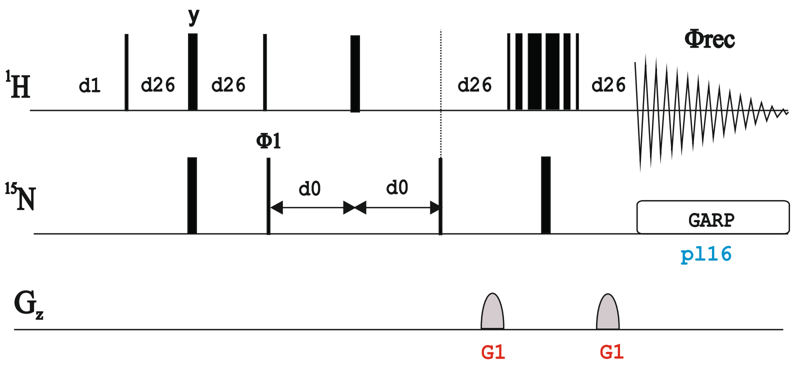
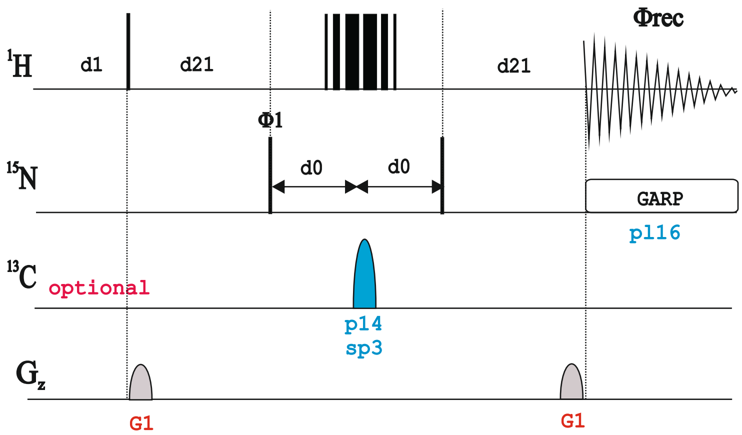


HMQC with WATERGATE

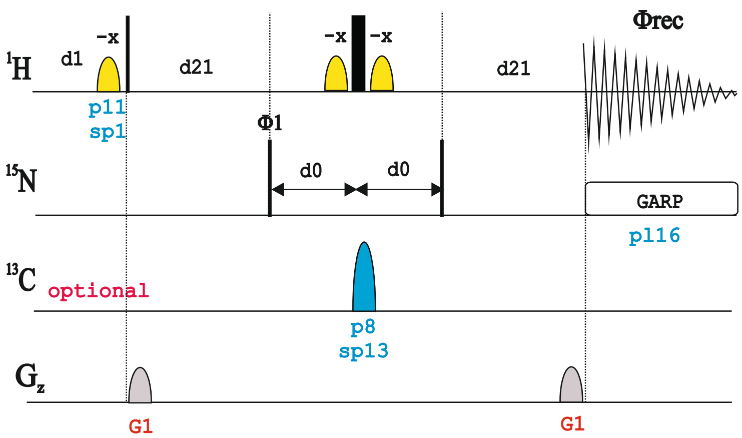
hmqcf3gpph19



hmqcf3gpph19.2



hmqcf3gpphwg



1D HMQC

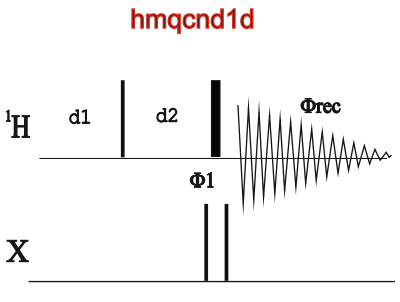
- **Phase-Cycled:**

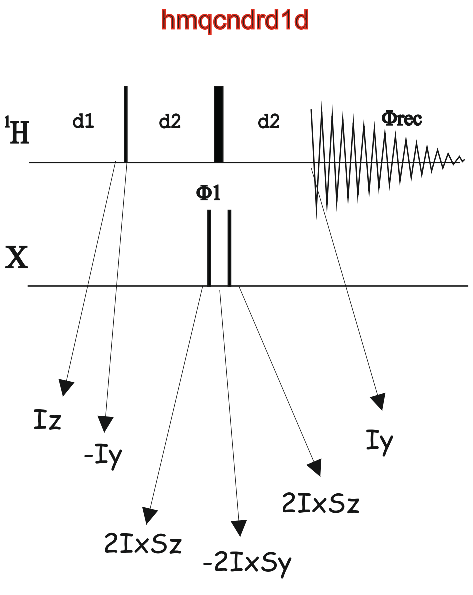
  1D HMQC with refocusing but not decoupling (**hmqcndrd1d** | HMQC1D)
  1D HMQC without refocusing and without decoupling (**inv3nd1d/ hmqcnd1d**)
  1D HMQC with refocusing and decoupling (**hmqcrd1d**)
  1D HMQC using BIRD without refocusing and without decoupling (**hmqcbind1d**)
  1D HMQC using BIRD with refocusing and without decoupling (**hmqcbindrd1d**)
  1D HMQC using BIRD with refocusing and decoupling (**hmqcbird1d**)

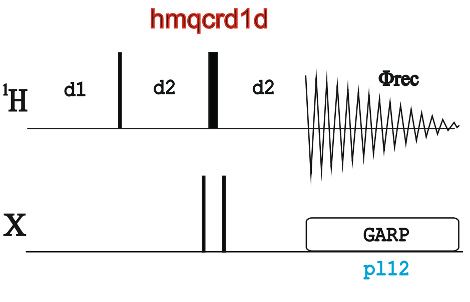
- **Gradient-based:**

  ge-1D HMQC with refocusing but not decoupling (**hmqcgpnd1d**)

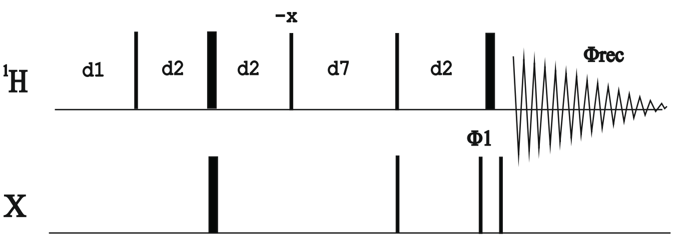
Inverse 1D HMQC
A. Bax, R.H. Griffey & B.L. Hawkins, J. Magn. Reson. 55, 301 (1983)



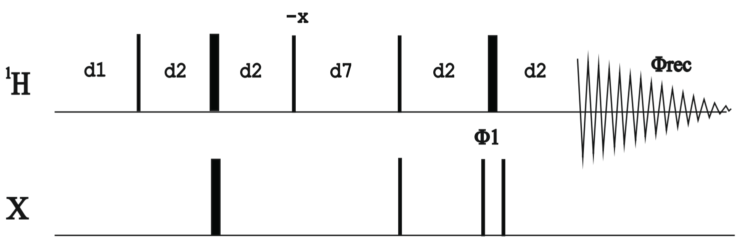




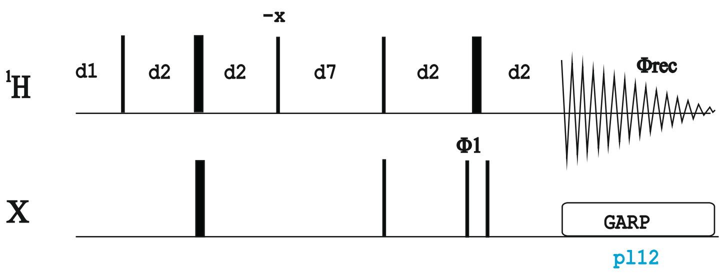
hmqcbind1d



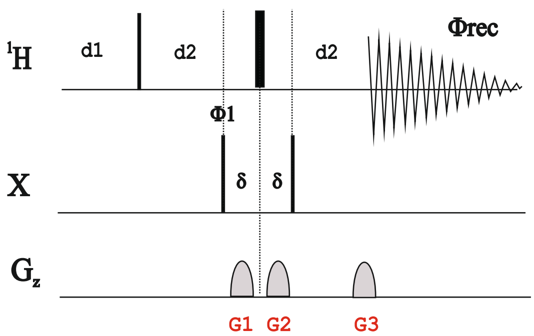
hmqcbindrd1d



hmqcbird1d



hmqcgpnd1d



gp 1 : gp 2 : gp 3
50 : 30 : 40.1 for C-13
70 : 30 : 50.1 for N-15

BRUKER PULSE PROGRAM CATALOGUE

NMRGuide

2D HSQC EXPERIMENT

Experiment Description

The HSQC (Heteronuclear Sinlge-Quantum Correlation) experiment is a proton-detected experiment designed to obtain heteronuclear correlation between 1H and X heteronuclei via the scalar coupling constant, 1J(XH)

Sample Requirements

HSQC experiments can be recorded on any type of sample.

Hardware Requirements

HSQC experiments can be recorded on any probehead but an inverse probe equipped with gradients is strongly recommended.

NMR Spectrum

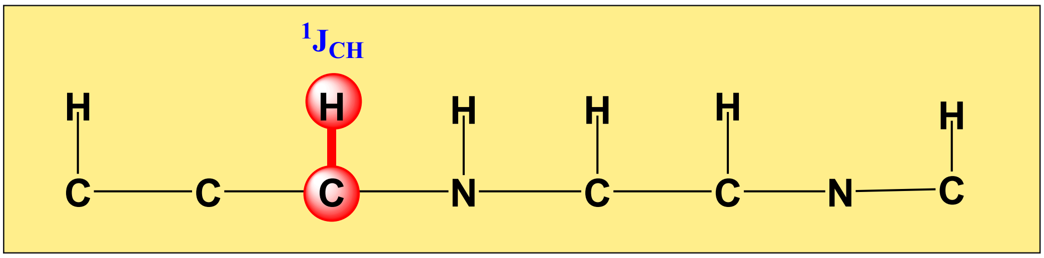
A 2D HSQC map correlates 1H and X chemical shifts via 1J(XH).

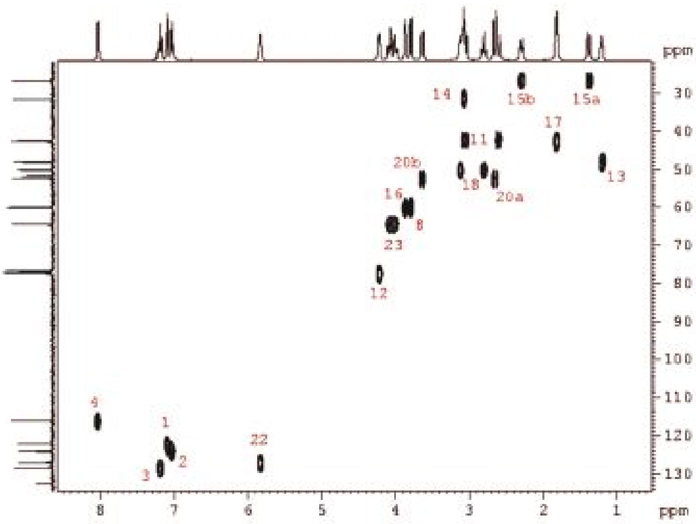
Related Experiments

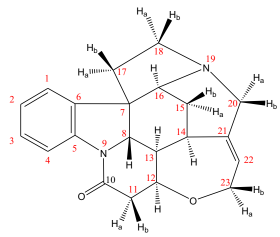
1D Inverse, 2D HMQC and 2D HSQC-edited experiments
Also see other 2D HSQC-related experiments: HSQC-TOCSY, HSQC-NOESY ...

References

G. Bodenhausen & D.J. Ruben, Chem. Phys. Lett. 69, 185 (1980)







2D HSQC Experiments (f2 channel)

- **Phase-cycled:**

Phase-sensitive 2D HSQC (**hsqcph**)

- **Phase-cycled and solvent suppression:**

Phase-sensitive 2D HSQC with presaturation (**hsqcphpr**)
Phase-sensitive 2D HSQC with off-resonance presaturation (**hsqcphps**)

- **Gradient-based:**

ge-1D HSQC with refocusing and no decoupling (**hsqcgpnd1d**)

Phase-sensitive ge-2D HSQC using z-filter and selection before t1 (**hsqcgpph** | HSQCGP)
Phase-sensitive ge-2D HSQC using z-filter and selection after t1 (**hsqcgpph2**)

Phase-sensitive ge-2D HSQC using echo-antiecho (**hsqcetgp** | HSQCETGP)
Phase-sensitive ge-2D HSQC using echo-antiecho and adiabatic pulses for inversion (**hsqcetgpsp**)
Phase-sensitive ge-2D HSQC using echo-antiecho and adiabatic pulses for inversion and refocusing(**hsqcetgpsp.2**)
Phase-sensitive ge-2D HSQC using echo-antiecho and adiabatic pulses for inversion and refocusing and BS effects(**hsqcetgpsp.3**)

Phase-sensitive ge-2D HSQC using PEP (**hsqcetgpsi**)
Phase-sensitive ge-2D HSQC using PEP with gradients in back-inept (**hsqcetgpsi2**)
Phase-sensitive ge-2D HSQC using PEP with gradients in back-inept and water flip-back (**hsqcetfpgpsi2**)
Phase-sensitive ge-2D HSQC using PEP and adiabatic pulses for inversion (**hsqcetgpsisp** | HSQCETGPSISP)
Phase-sensitive ge-2D HSQC using PEP and adiabatic pulses for inversion with gradients in back-inept (**hsqcetgpsisp2**)
Phase-sensitive ge-2D HSQC using PEP and adiabatic pulses for inversion and refocusing (**hsqcetgpsisp.2** | HSQCETGPSISP.2)
Phase-sensitive ge-2D HSQC using PEP and adiabatic pulses for inversion and refocusing with gradients in back-inept (**hsqcetgpsisp2.2**)
Phase-sensitive ge-2D HSQC using PEP and adiabatic pulses for inversion and refocusing with gradients in back-inept and presaturation (**hsqcetgpprsisp2.2**)
Phase-sensitive ge-2D HSQC using PEP and adiabatic pulses for inversion and refocusing with gradients in back-inept with suppression of HSQC-COSY peaks (**hsqcetgpsisp23**)

ge-2D ^{1}H-X HSQC experiment with X-Y-decoupling during acquisition and with selective Cb/C=O decoupling. (**hsqcdhetgpsp**)

Phase-sensitive ge-2D HSQC with WET solvent suppression (**hsqcetgpsiwt** | HSQCETGPSIWT)

2D HSQC Experiments (f3 channel)

- **Phase-cycled:**

  Phase-sensitive 2D ^{1}H-^{15}N HSQC (**hsqcf3ph**)

- **Phase-cycled and solvent suppression:**

  Phase-sensitive 2D ^{1}H-^{15}N HSQC using presaturation (**hsqcf3phpr**)

- **Gradient-based:**

  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC using echo-antiecho (**hsqcetf3gp** | HSQCETF3GP)
  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC using PEP (**hsqcetf3gpsi** | HSQCETF3GPSI)
  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC using PEP with gradients in back-inept (**hsqcetf3gpsi2**)

  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC using XY16-CPMG(**hsqcetf3gpxy, hsqcetf3gpxy.2**)

- **Gradient-based and solvent suppression**

  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC using water flip-back and echo-antiecho (**hsqcetfpf3gp** | HSQCETFPF3GP)
  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC using water flip-back and PEP (**hsqcetfpf3gpsi** | HSQCETFPF3GPSI)
  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC using water flip-back and PEP with gradients in back-inept (**hsqcetfpf3gpsi2**)

  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC using WATERGATE (3-9-19) (**hsqcf3gpph19**)
  Fast-HSQC, Phase-sensitive ge-2D ^{1}H-^{15}N HSQC using WATERGATE (3-9-19) (**fhsqcf3gpph** | FHSQCF3GPPH)
  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC using water flip-back and WATERGATE (selective pulse) (**hsqcfpf3gpphwg** | HSQCFPF3GPPHWG)

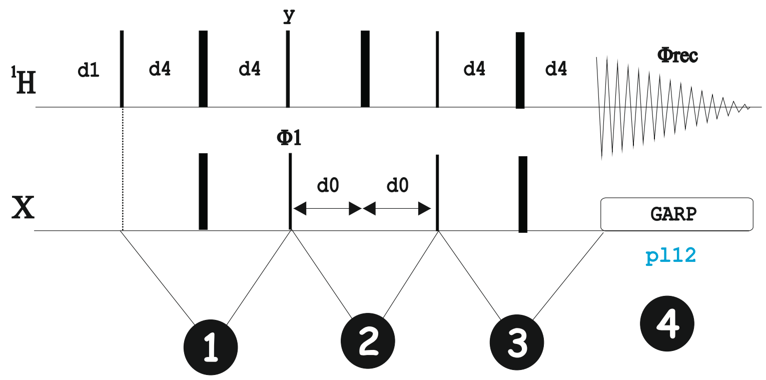
- **Gradient-based simultaneous CN-HSQC**

  Phase-sensitive ge-2D simultaneous ^{1}H-^{15}N/^{1}H-^{13}C HSQC using echo-antiecho (**hsqcetgpsismsp**)

Also see in chapter SOFAST/BEST experiments:
NH-selective SOFAST/BEST-HMQC experiment for rapid pulsing (b_hsqcetf3gpsi)

Also see other related correlation experiments (HSQC, TROSY ...), hybrid experiments (HSQC-TOCSY, HSQC-NOESY...) or related experiments for relaxation or coupling constants measurements

HSQC: Sequential Four-Step Experiment



1. **1H-to-X INEPT transfer** optimized to 1/4J(XH)
2. **δ(X) evolution and J(CH) refocusing** during variable d0 period
3. **X-to-1H retro-INEPT transfer** optimized to 1/4J(XH)
4. **1H detection with optional X-decoupling**

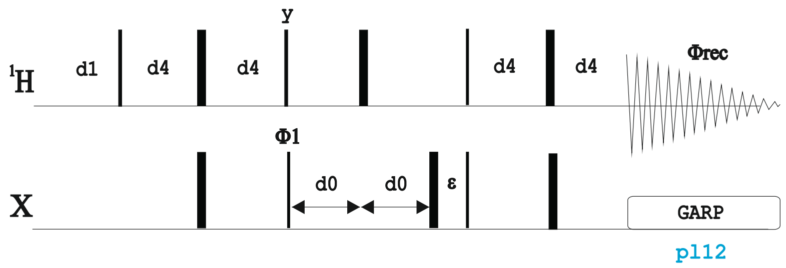
Options for HSQC included in this chapter:

Use of Trim Pulses in step 1
Use of Gradients for Coherence Selection (step 2 and 3)
Use of Gradients for purge (in steps 1 and/or 3)
Water-flip back pulses (in zz-filters and step 3)
Sensitivity Improved (PEP) version (step 3)
Use of WATERGATE solvent suppression (in step 3)
Use of Adiabatic 180° Pulses instead of hard 180 pulses in steps 1,2 and 3.

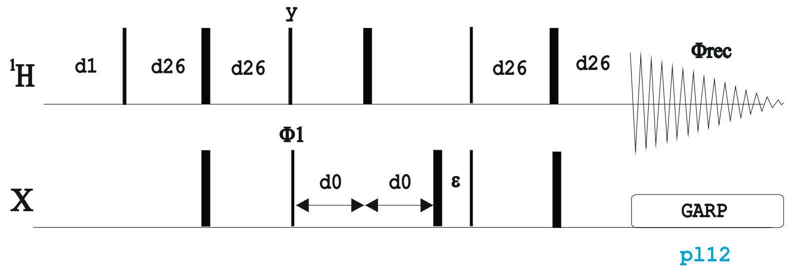
Options for HSQC included in other chapters:

Multiplicity-editing incorporated in step 2
Constant-Time periods incorporated in step 2
Spin-state selection incorporated in steps 2 and/or 3
Relaxation blocks in steps 2 or 3
Additional mixing processes between steps 3 and 4

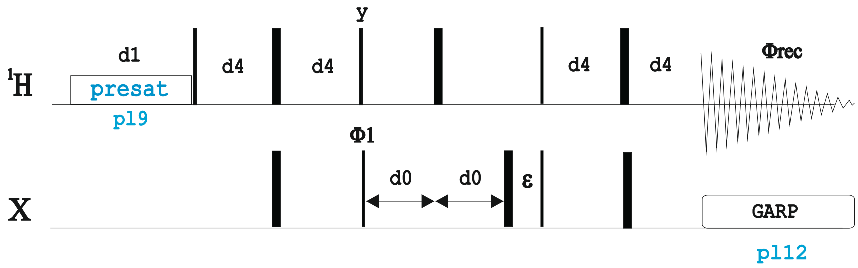
hsqcph



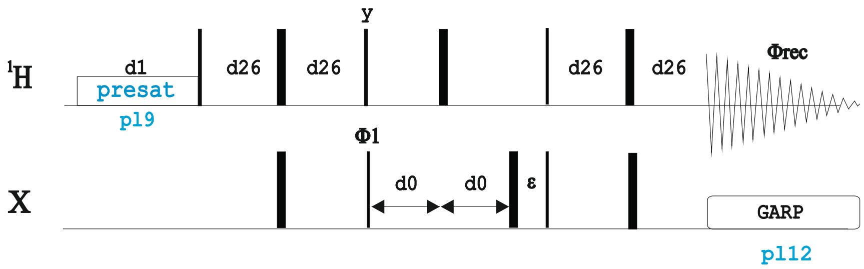
hsqcf3ph



hsqcphpr



hsqcf3phpr



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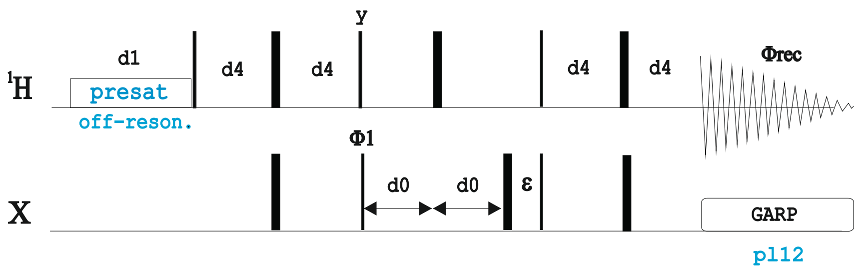

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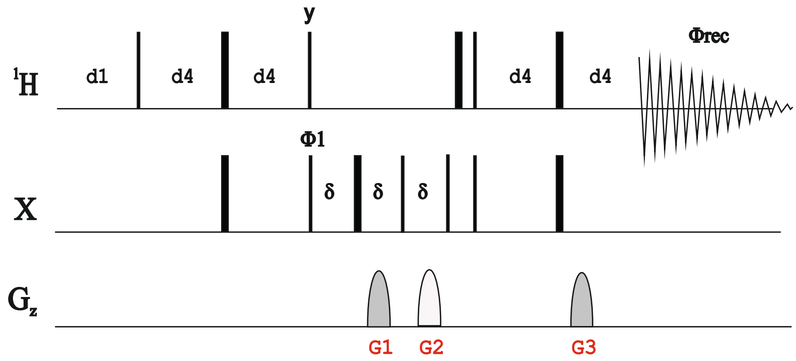
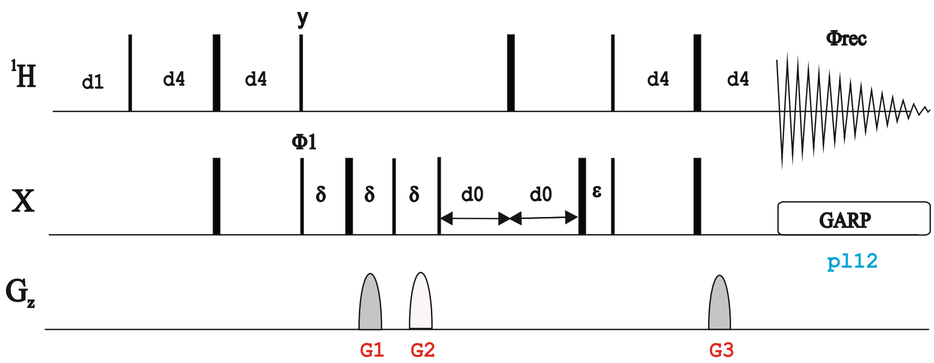
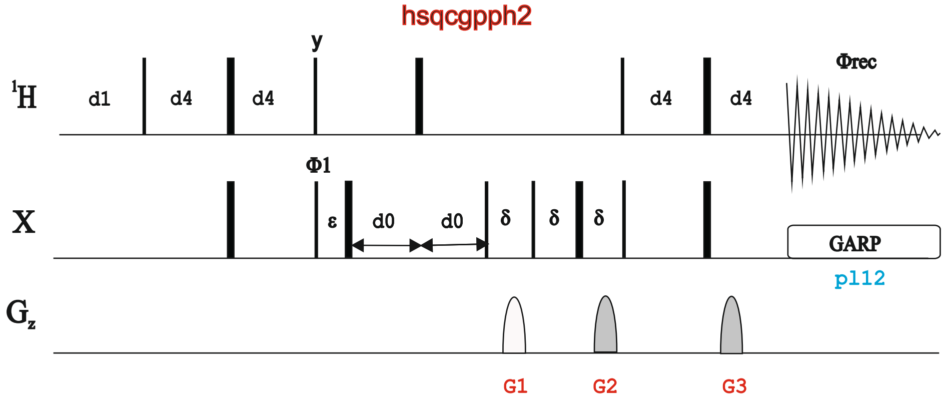


gp 1 : gp 2 : gp 3
80 : 30 : 20.1 for C-13
80 : 30 : 8.1 for N-15

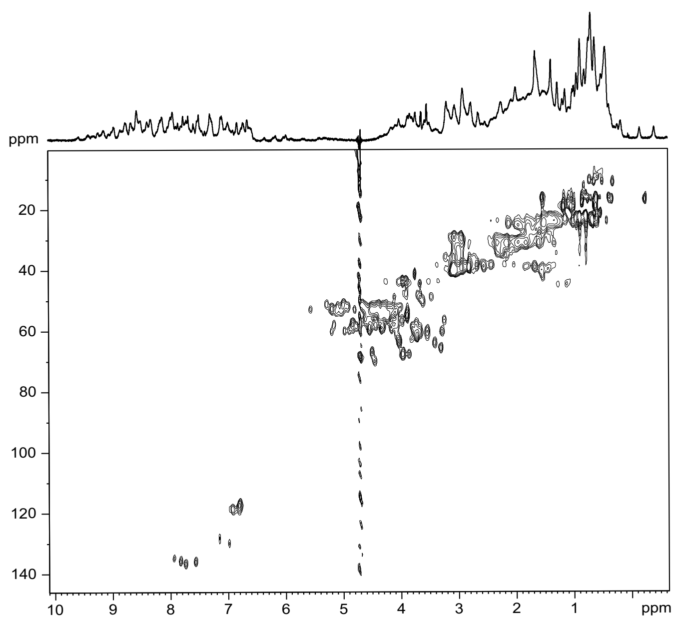
hsqcgpph



hsqcgpph2

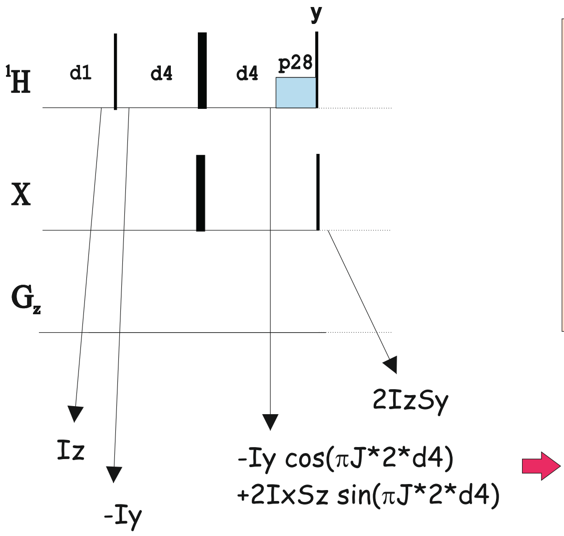


gp 1 : gp 2 : gp 3
30:80:20.1 for C-13
30:80:8.1 for N-15



NMR Building Block:
Modified INEPT Transfers in HSQC-Type Experiments

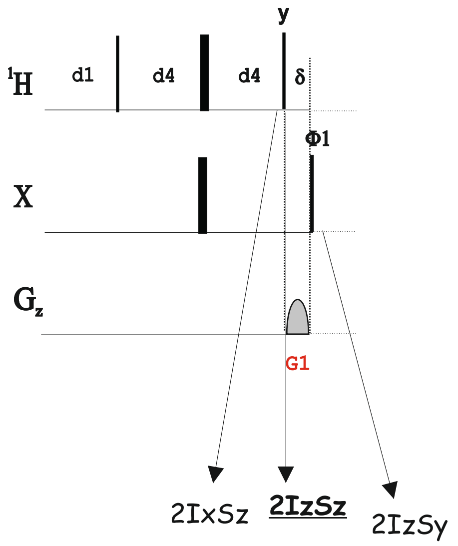
A. INEPT with a trim pulse




```
....
  d12 pl1:f1 pl2:f2
3 (p1 ph1)
  d4
  (center (p2 ph1) (p4 ph1):f2 )
  d4
  p28 ph1
  4u
  (p1 ph2) (p3 ph1):f2
....
```

An optional short trim pulse of duration p28=1msec applied at high power from the x axis removes any residual Iy contribution.

B. INEPT with zz-purge Gradient:

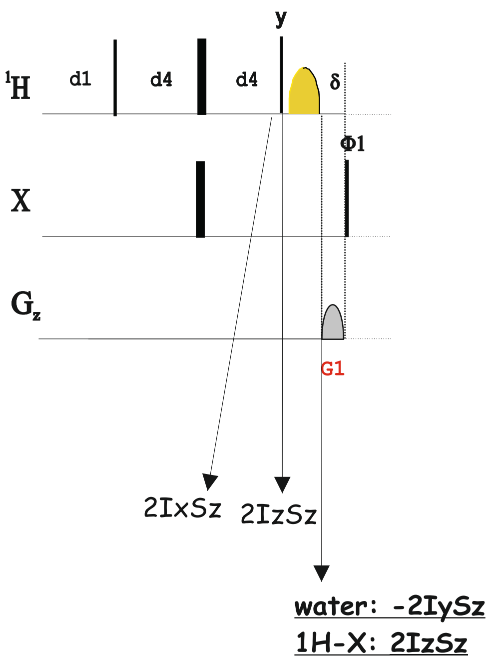



```
....
  d12 pl1:f1 pl2:f2
3 (p1 ph1)
  d4
  (center (p2 ph1) (p4 ph1):f2 )
  d4 UNBLKGRAD
  (p1 ph2)
  3u
  p16:gp1
  d16
  (p3 ph1):f2
....
```

A gradient G1 applied just after the 90°y(1H) removes any residual transverse magnetization. IzSz coherences are not dephased by this purge gradient.

The trim pulse and the purge gradient are independents and they can be applied simultaneously

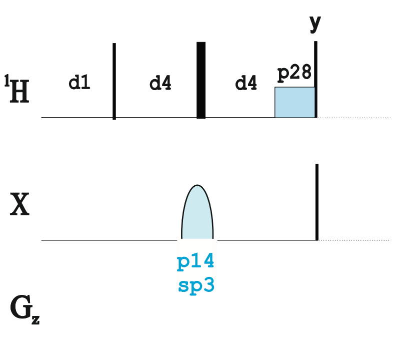
C. INEPT with Water Flip-Back and zz-purge Gradient:



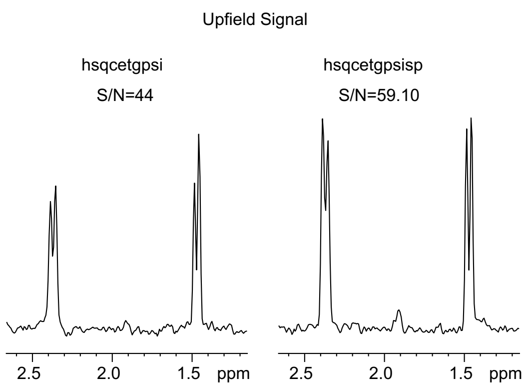

```
....
  d12 pl1:f1 pl2:f2
3 (p1 ph1)
  d4
  (center (p2 ph1) (p4 ph1):f2 )
  d4 UNBLKGRAD
  (p1 ph2)
  4u pl0:f1
  (p11:sp1 ph1:r):f1
  4u pl1:f1
  p16:gp1
  d16
  (p3 ph1):f2
....
```

A water-selective 90° pulse creates transverse magnetization for the water whereas other signals are unaffected. The gradient G1 dephases water signal

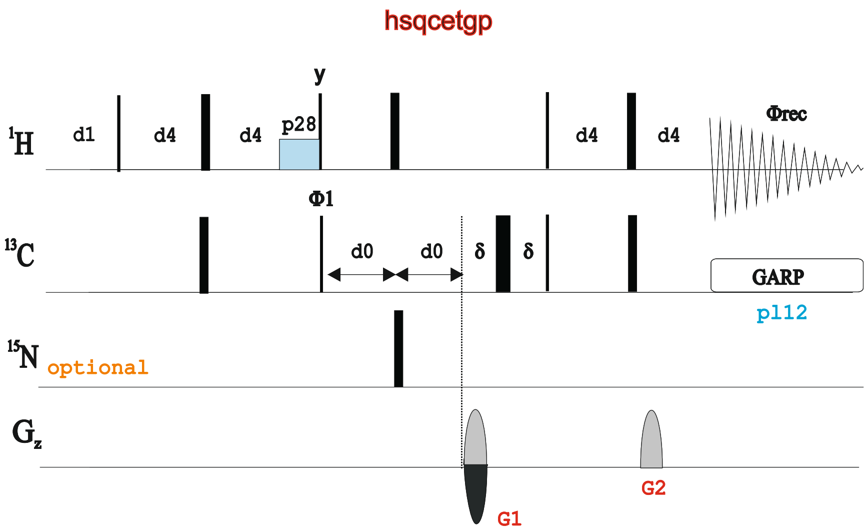
D. INEPT with Adiabatic 180° pulse:



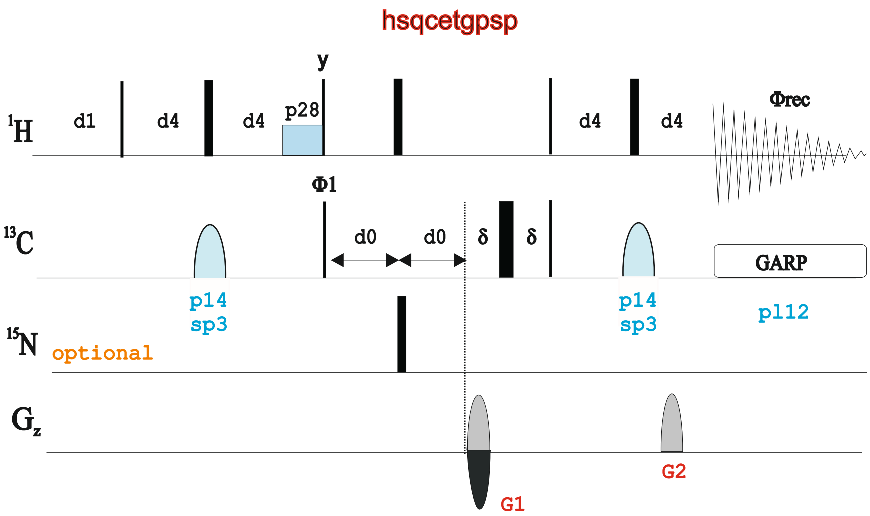
Hard 180° 13C pulses suffer of unwanted off-resonance effects. In 500MHz spectrometers (and higher) it is recommended to use a 500ms long adiabatic pulse as a inversion 13C element to improve sensitivity.



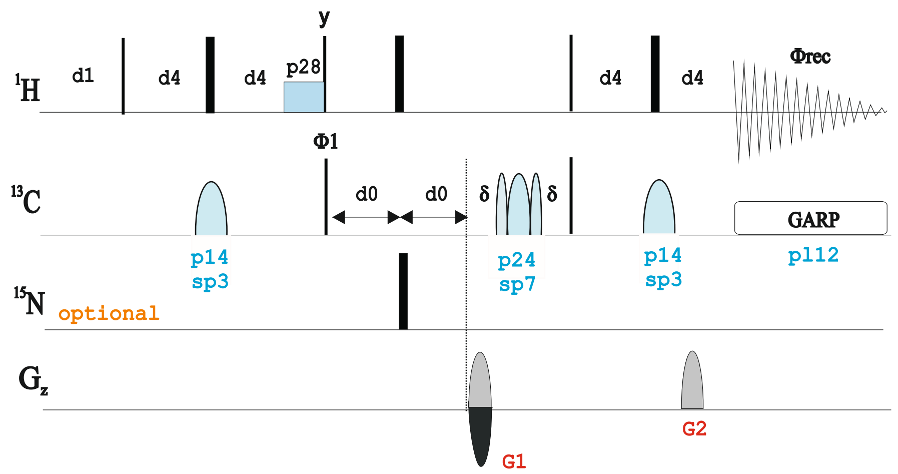
HSQC with Gradient Selection



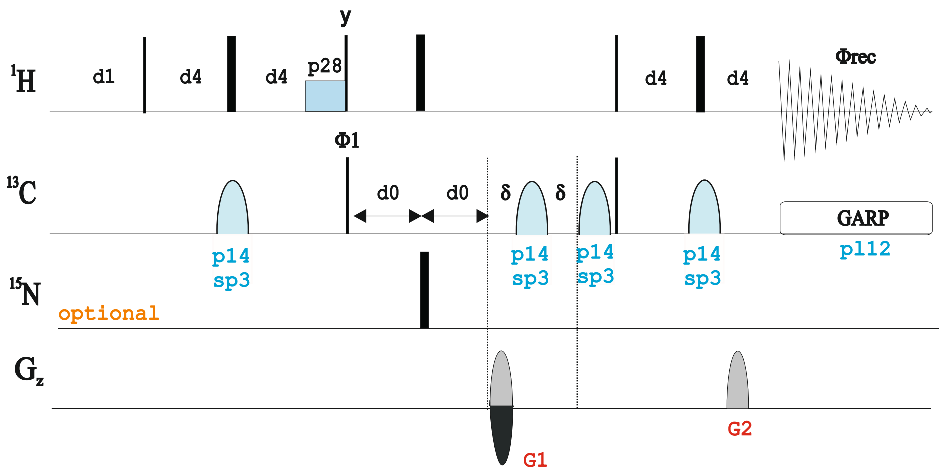
gp 1 : gp 2
80:20.1 for C-13
80:8.1 for N-15



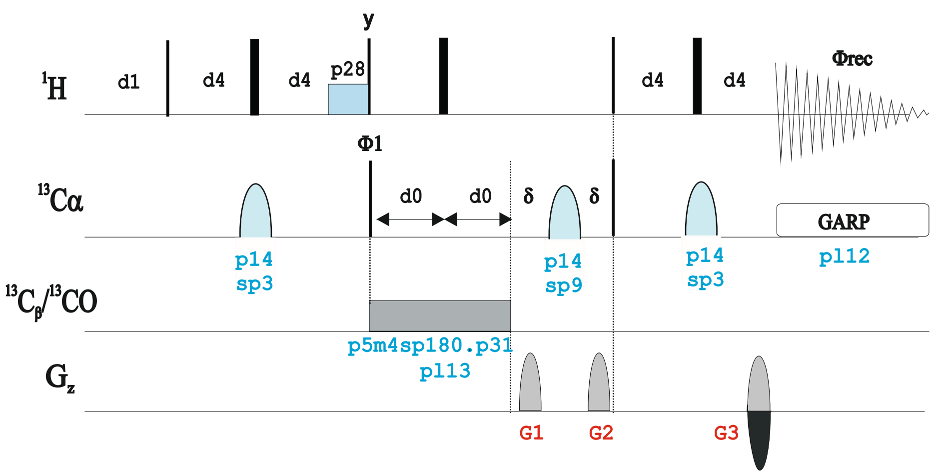
hsqcetgpsp.2



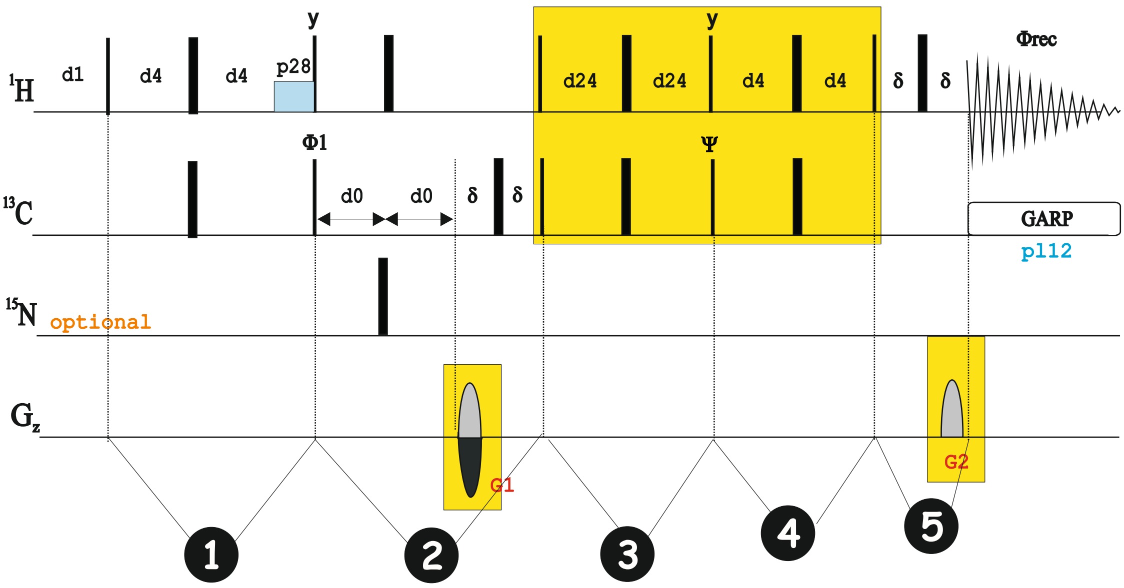
hsqcetgpsp.3



hsqcdhetgpsp



HSQC: Sensitivity Improved Version



1. J(CH) evolution using a 1H-to-X INEPT transfer
2. δ(C) evolution and J(CH)/J(NH) refocusing
   Refocused-Echo to accomodate gradient G1 encoding
3. J(CH) evolution using a X-to-1H retro-INEPT transfer
4. **J(CH) evolution using a X-to-1H retro-INEPT transfer (PEP methodology)**
5. **Echo to accomodate gradient G2 decoding**
6. 1H detection with optional X-decoupling

References SI:

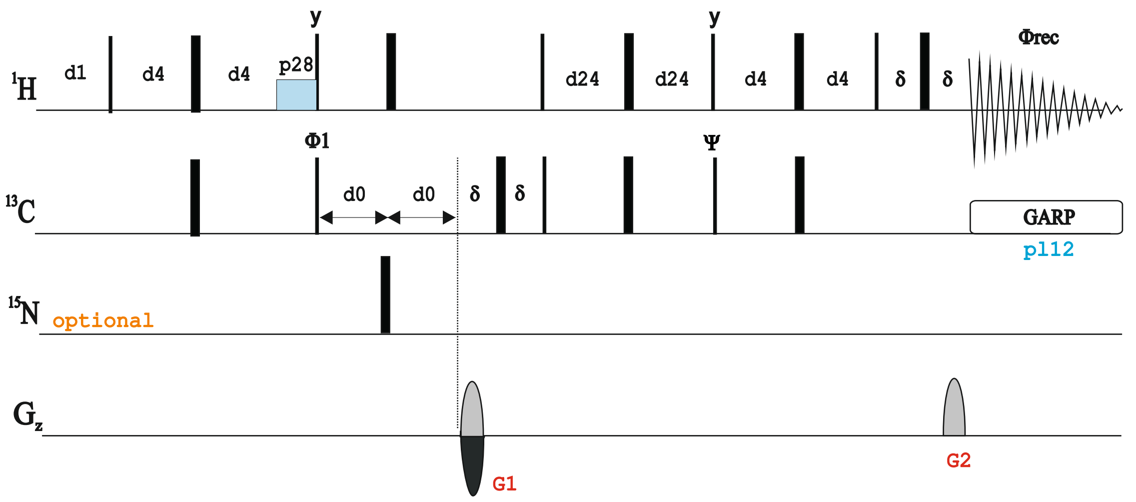
1. A.G. Palmer III, J. Cavanagh, P.E. Wright & M. Rance, J. Magn.Reson. 93, 151-170 (1991)
2. L.E. Kay, P. Keifer & T. Saarinen, J. Am. Chem. Soc. 114, 10663-5 (1992)
3. J. Schleucher, M. Schwendinger, M. Sattler, P. Schmidt, O. Schedletzky, S.J. Glaser, O.W. Sorensen & C. Griesinger, J. Biomol. NMR 4, 301-306 (1994)

SI version affords a theoretical sensitivity gain of S/N=100% in IS spin systems

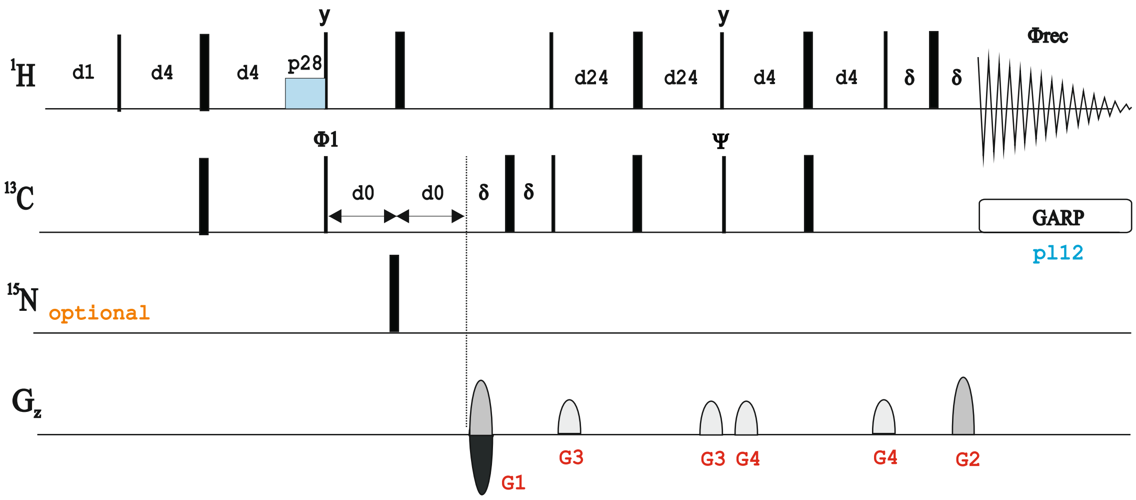
HSQC with PEP

gp 1 : gp 2
80:20.1 for C-13
80:8.1 for N-15

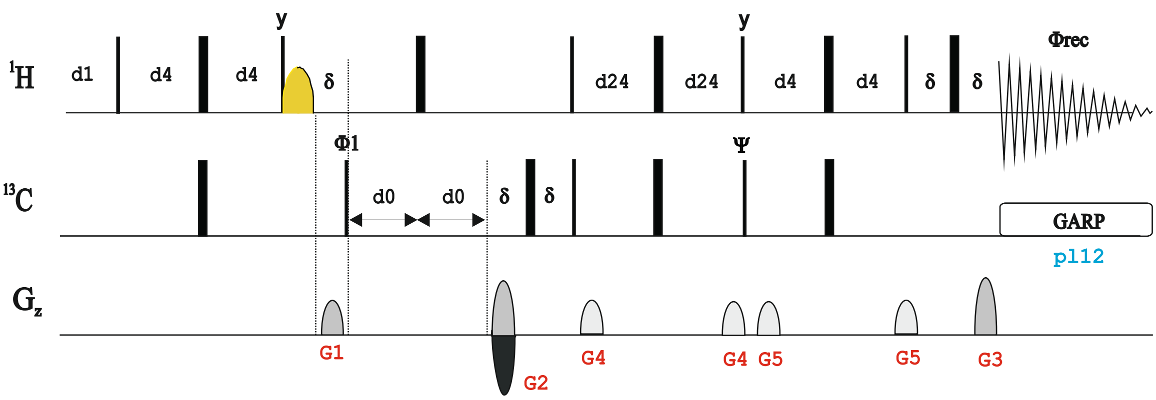
hsqcetgpsi



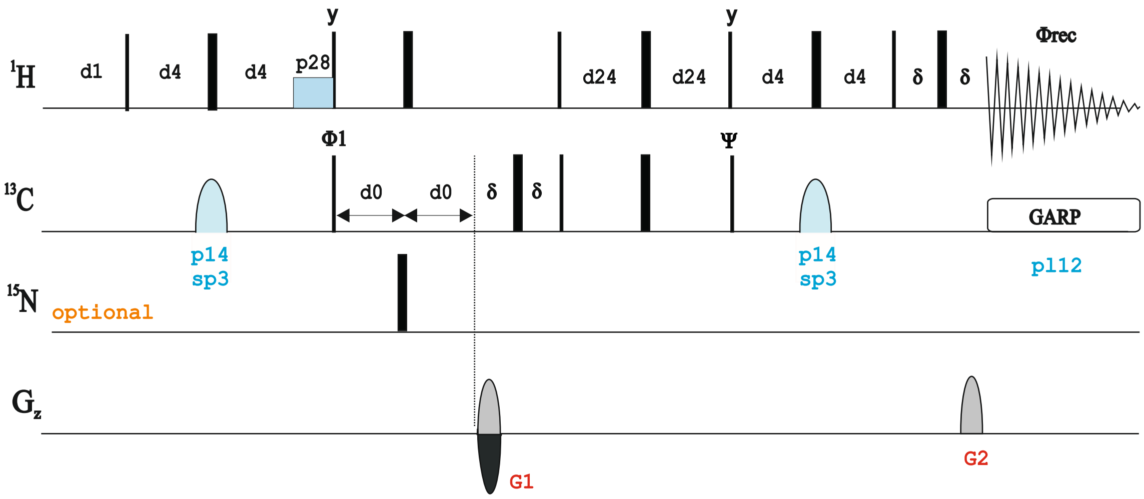
hsqcetgpsi2



hsqcetfpgpsi2



hsqcetgpsisp



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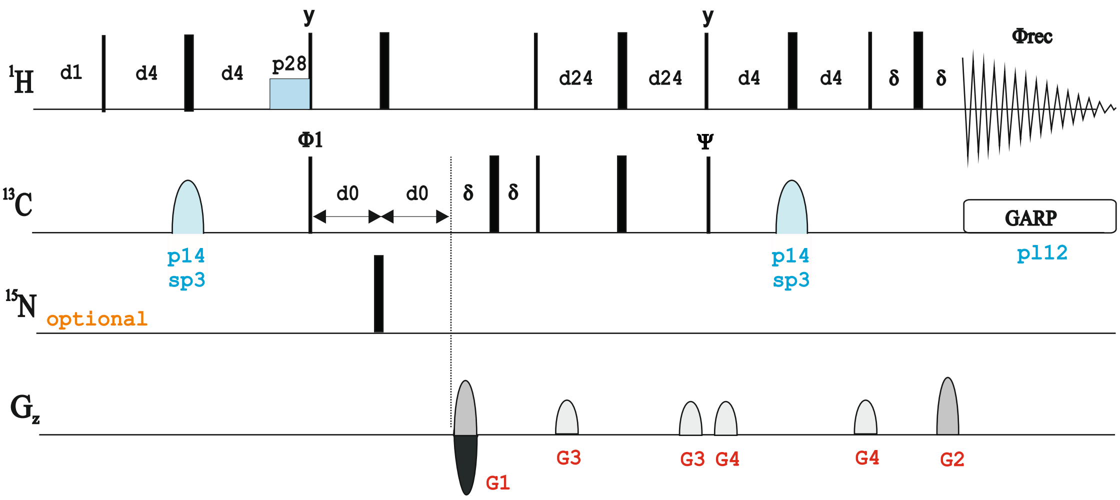
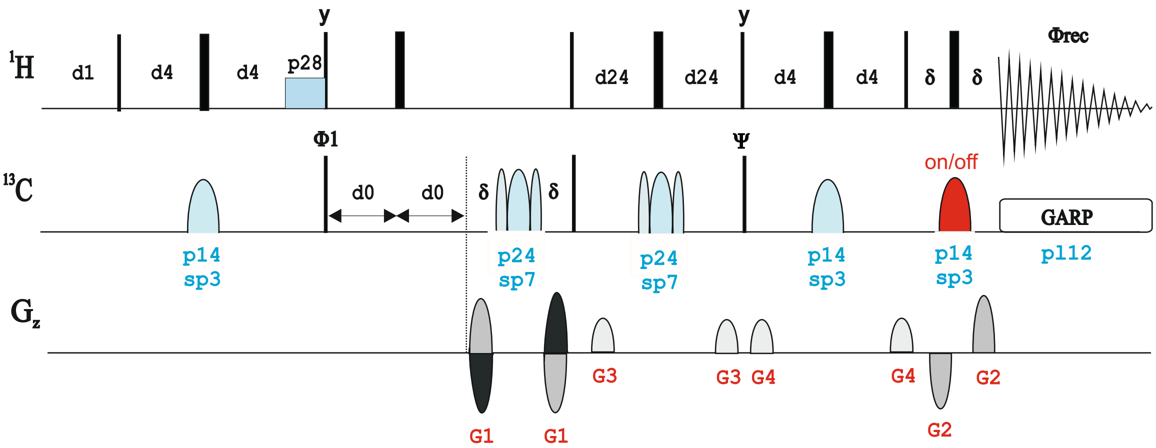
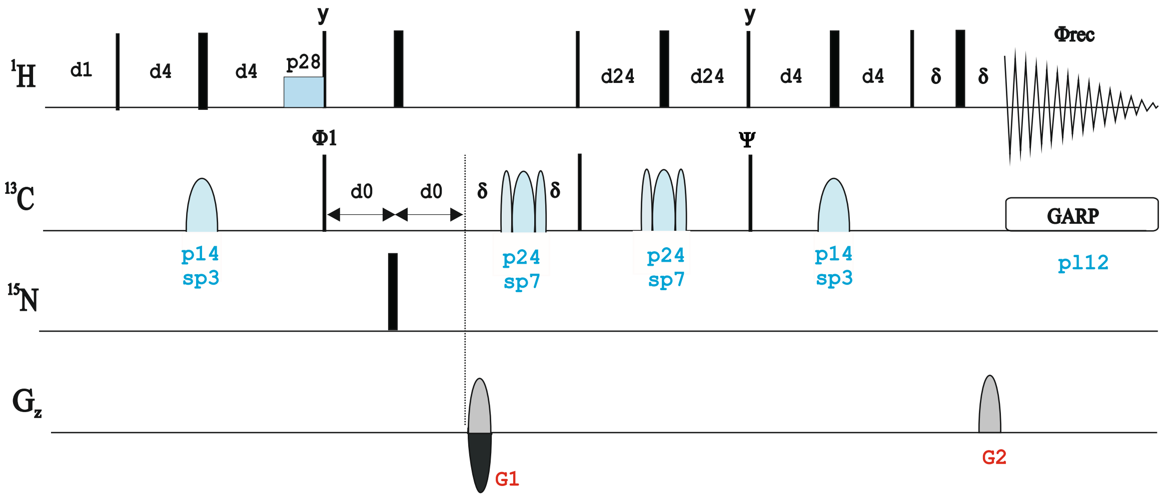


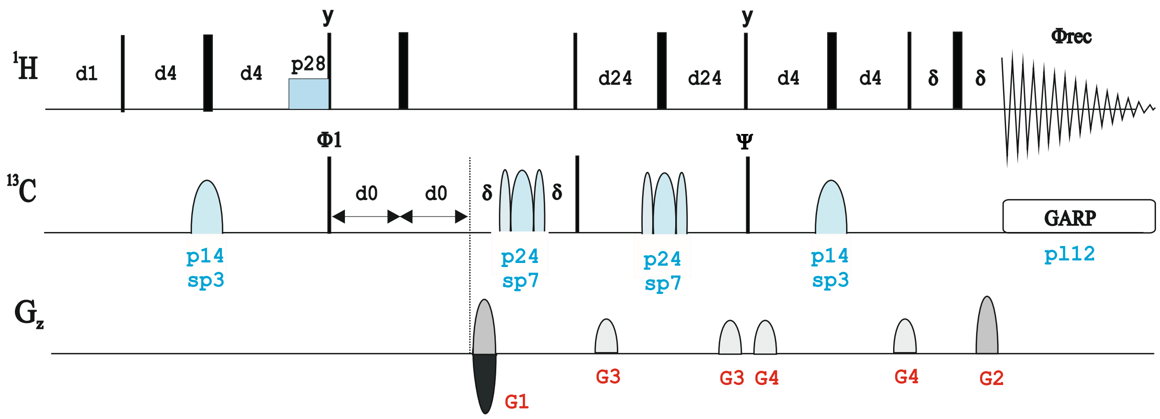
hsqcetgpsisp2.3



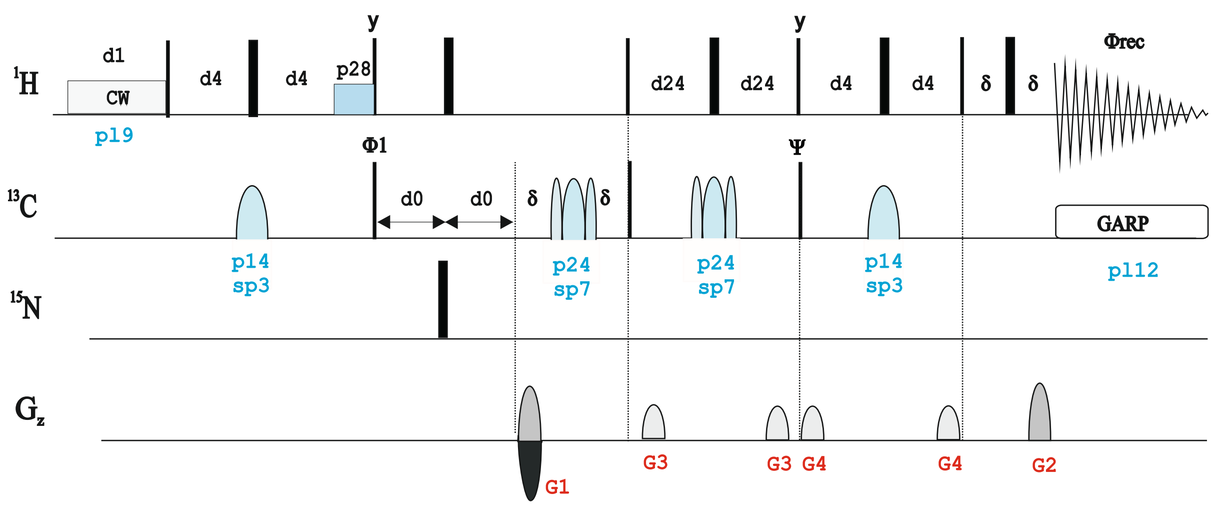
hsqcetgpsisp.2



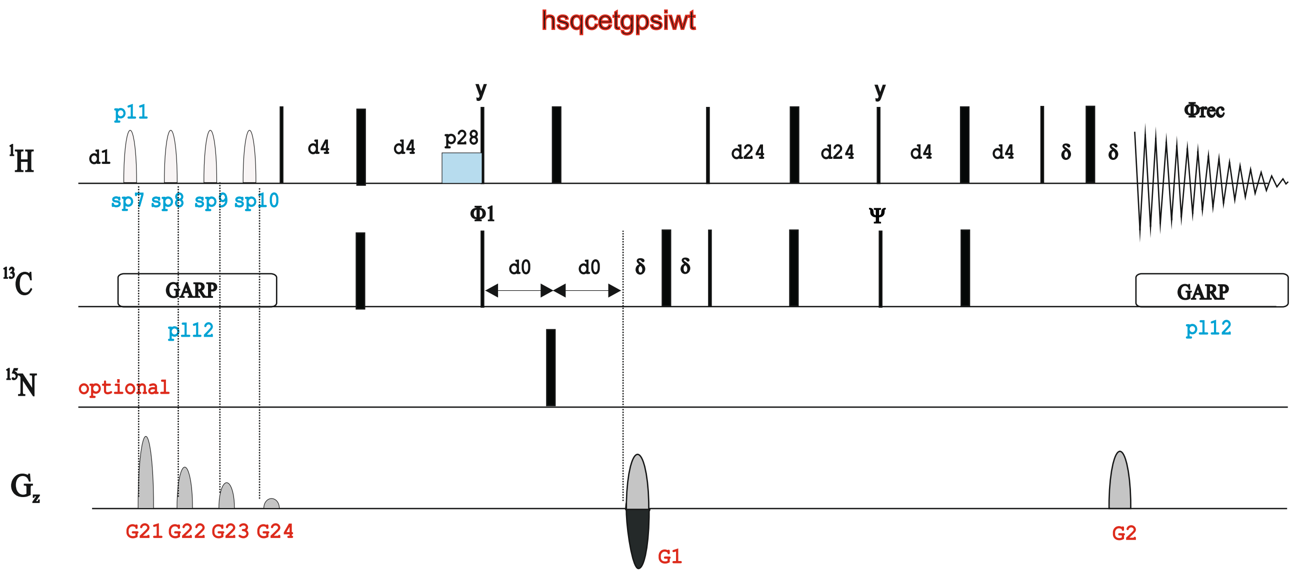
hsqcetgpsisp2.2



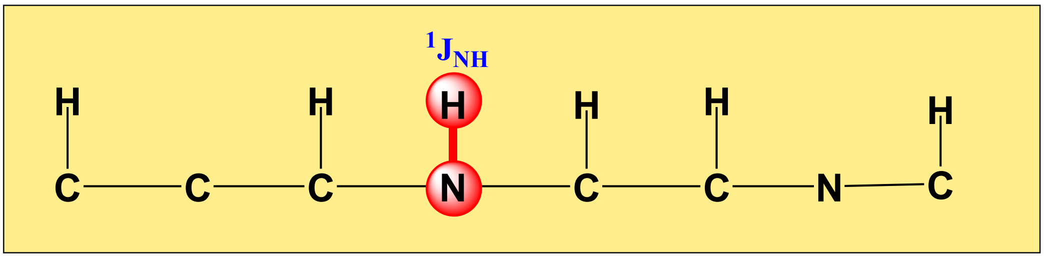
hsqcetgpprsisp2.2



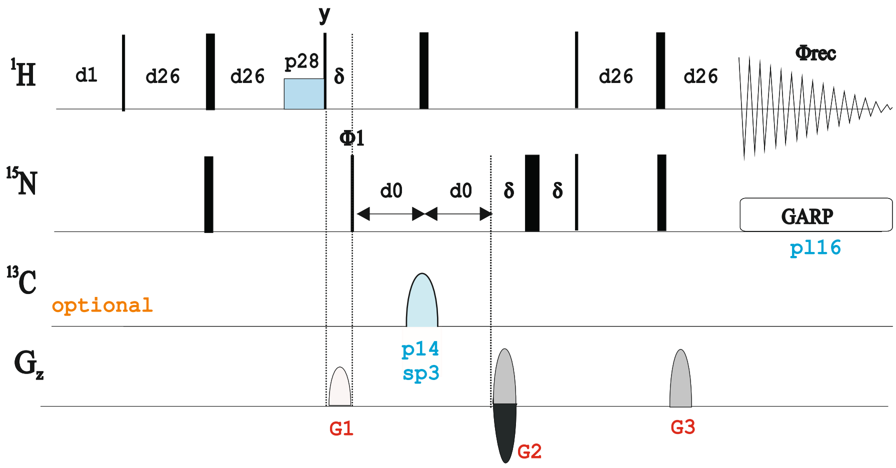
HSQC with WET solvent Suppression

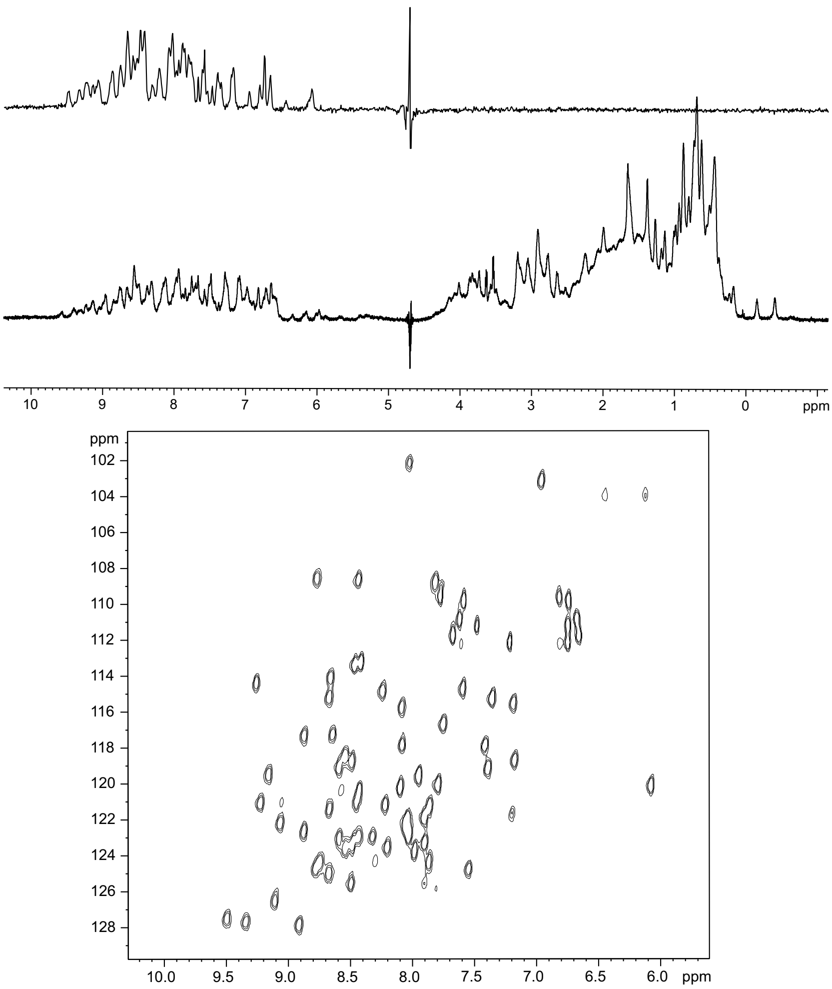


HSQC from f3 channel

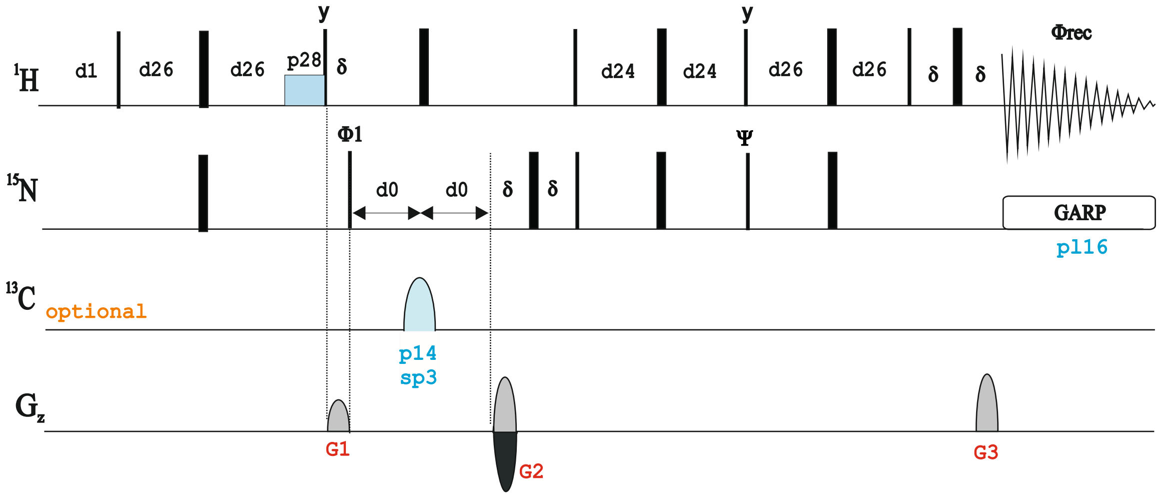


hsqcetf3gp





hsqcetf3gpsi



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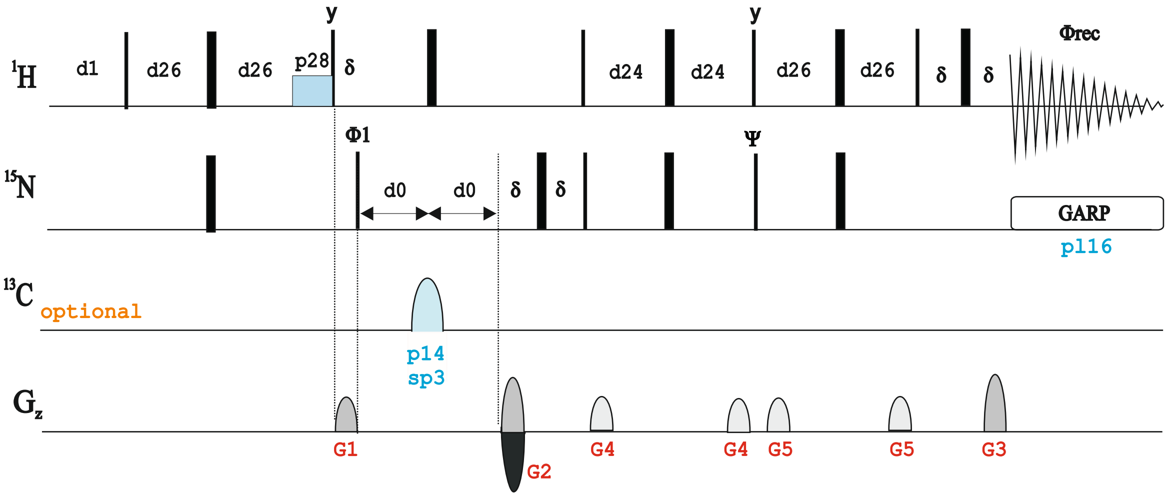
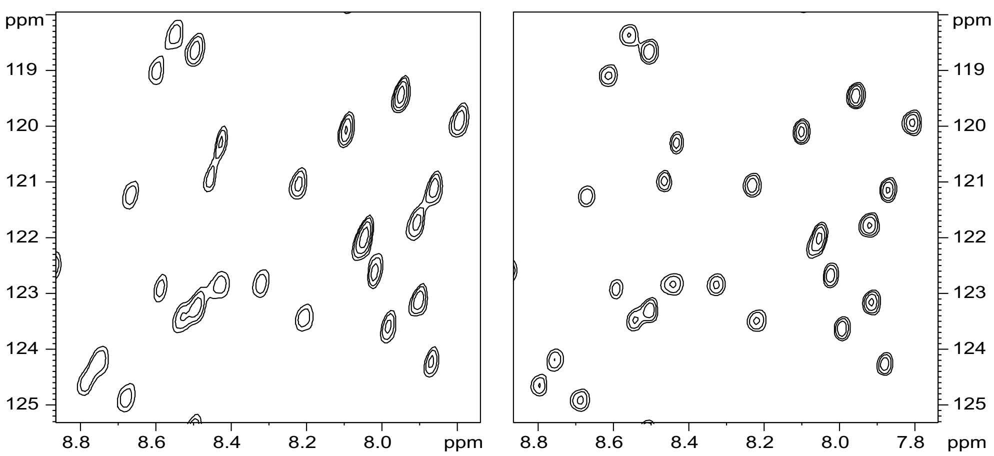


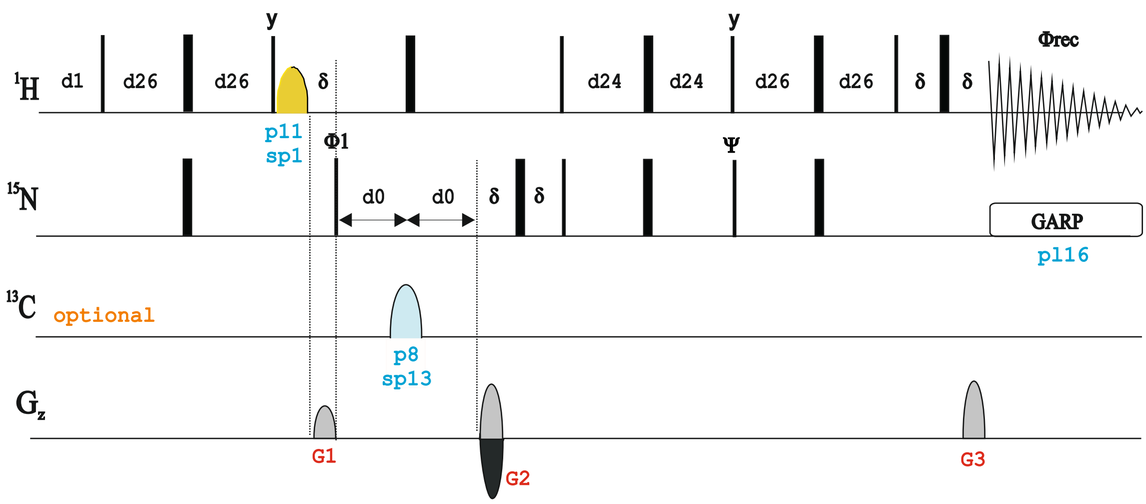
1H-15N HSQC Ubiquitina 1mM 500 MHz

Optional 13C decoupling during t1
in doubly-labeled proteins

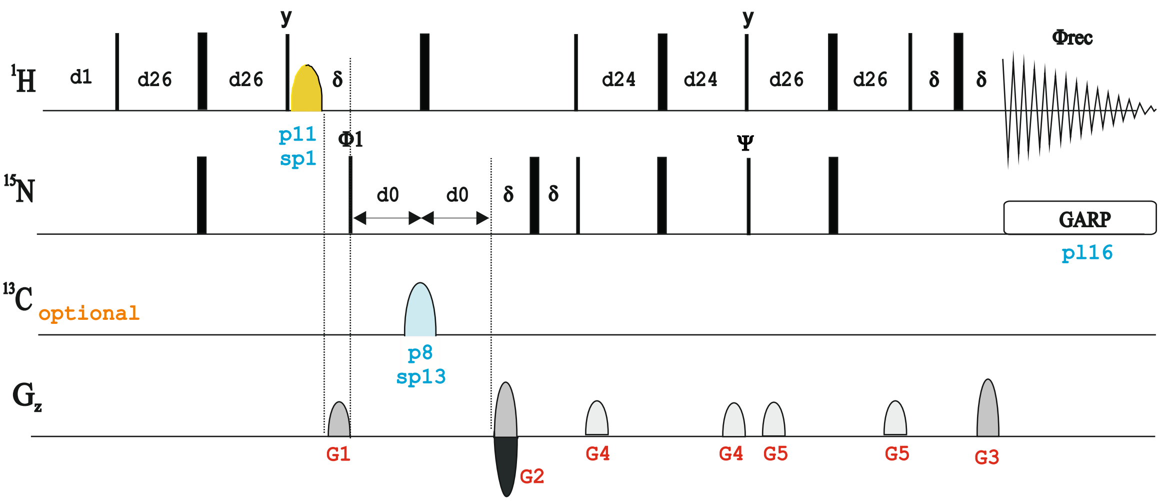
zgoptns -DLABEL_CN in eda



hsqcetfpf3gpsi



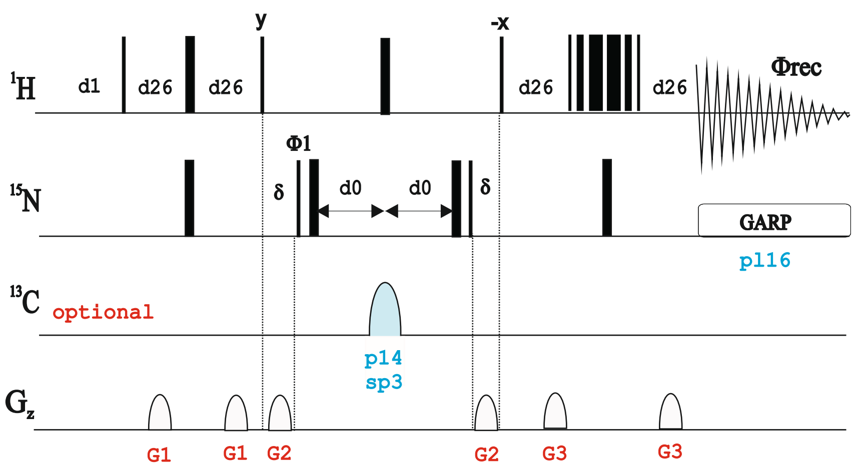
hsqcetfpf3gpsi2



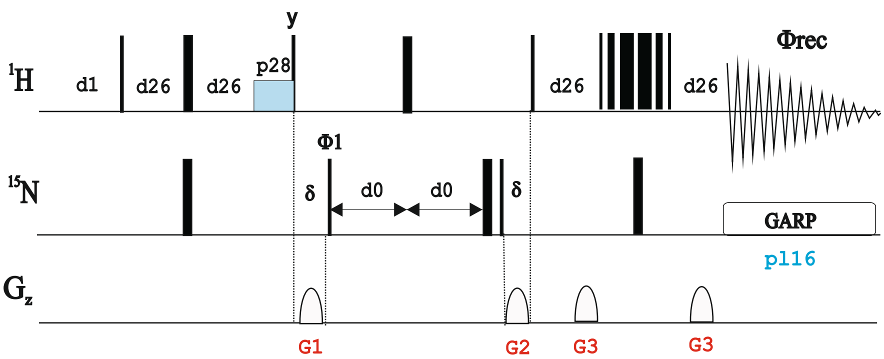
HSQC using WATERGATE
FAST-HMQC:

S. Mori, C. Abeygunawardana, M. O'Neil-Johnson & P.C.M. van Zijl, J. Magn. Reson. B 108, 94-98 (1995)

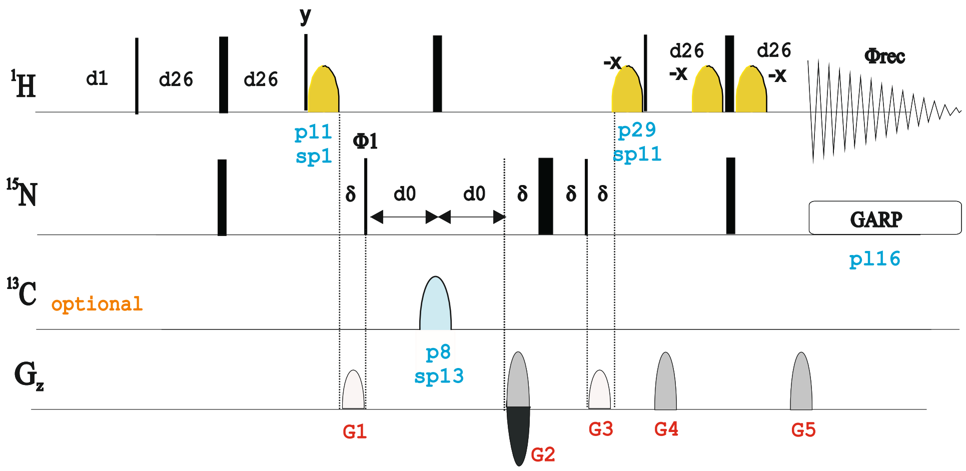
fhsqcf3gpph



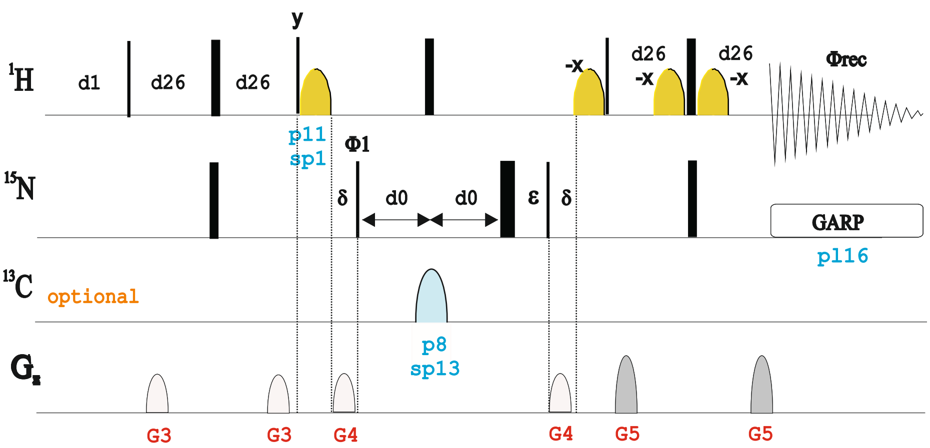
hsqcf3gpph19



hsqcetfpf3gp



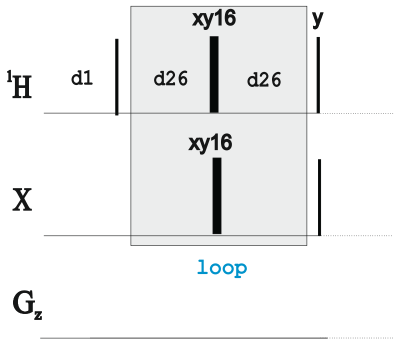
hsqcfpf3gpphwg



CPMG-HSQC:

F.A.A. Mulder, C.A.E.M. Spronk, M. Slijper, R. Kaptein & R. Boelens, J. Biomol. NMR 8, 223-228 (1996)

NMR Building Block:
A CPMG-INEPT Block

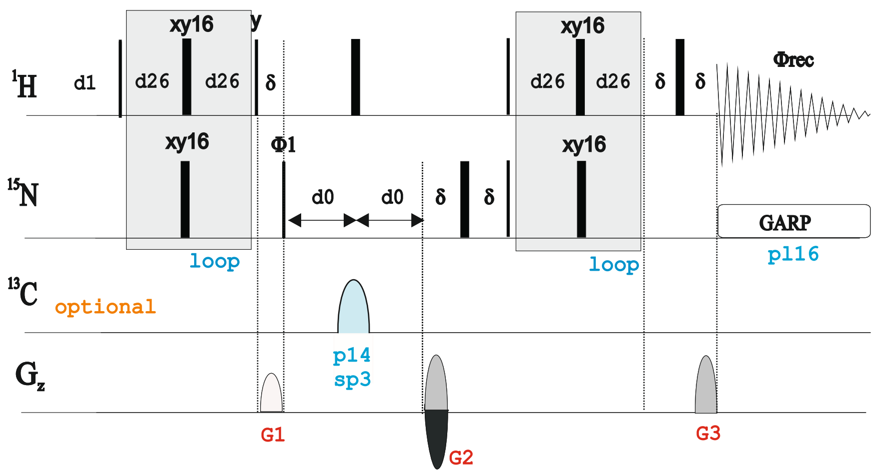



```
"DELTA1=(d26*2/16-p22)/2"

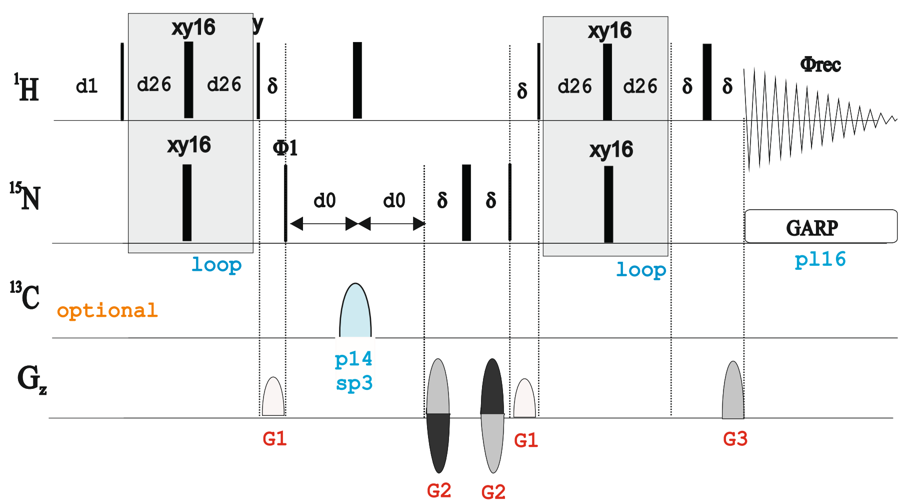
....
4 DELTA1
   (center (p2 ph6) (p22 ph16):f3 )
   DELTA1 ipp6 ipp16
   lo to 4 times 16
....

ph6=0
ph16=0
```

hsqcetf3gpxy



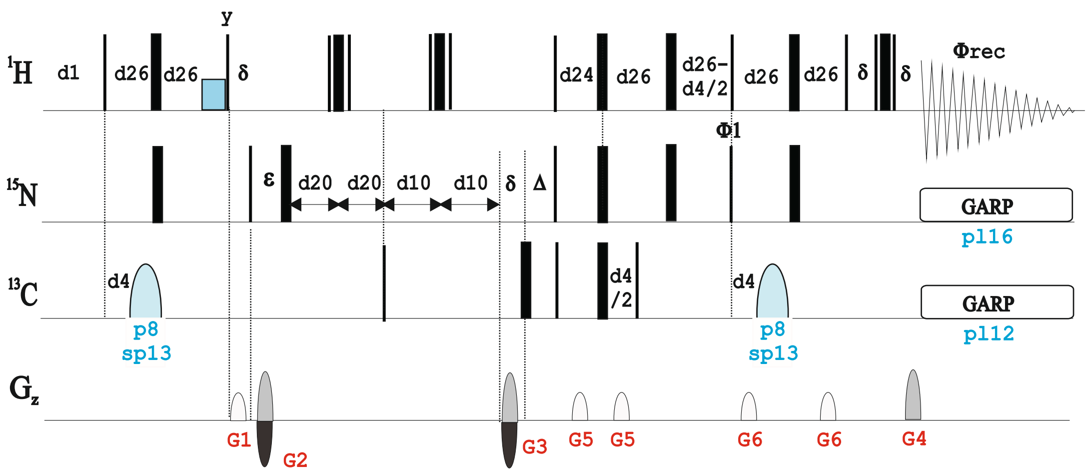
hsqcetf3gpxy.2

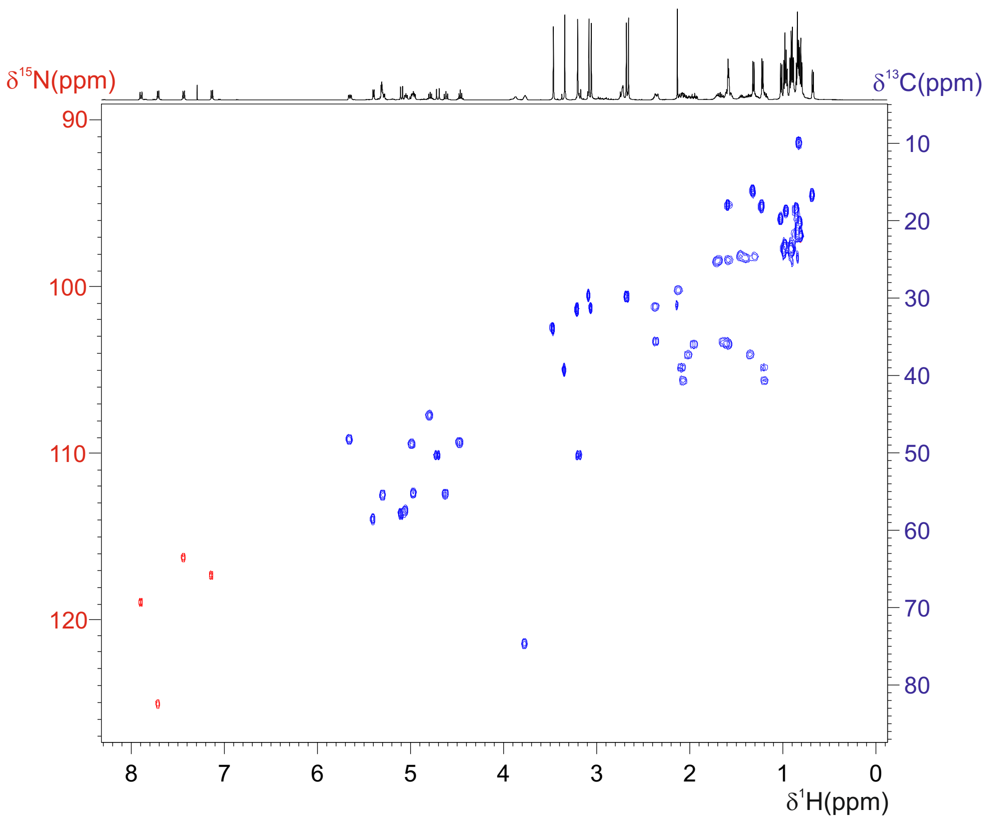


Simultaneous C,N-HSQC:

M. Sattler, M. Maurer, J. Schleucher & C. Griesinger, J. Biomol. NMR 5, 97-102 (1995))

hsqcetgpsismsp





BRUKER
PULSE PROGRAM
CATALOGUE

NMRGuide

2D CONSTANT-TIME HSQC AND HMQC EXPERIMENTS

Experiment Description

The Constant-time version of the HSQC experiment(CT-HSQC) is designed to remove the J(CC) evolution during the variable d0 period.

Sample Requirements

CT-HSQC experiments are recorded in 13C-labeled compounds. Pulse programs also includes suitable 15N decoupling and therefore they are ready to be applied on 15N-labeled compounds.

Hardware Requirements

CT-HSQC experiments can be recorded on any probehead but triple-resonance probes equipped with gradients is strongly recommended.

NMR Spectrum

A 2D CT-HSQC map correlates 1H and X chemical shifts via 1J(XH) and J(CC) is removed from the F1 dimension.

Related Experiments

2D HSQC experiments
Also see other 2D CT-HSQC to measure coupling constants in labeled proteins.

References

G.W. Vuister & A. Bax, J. Magn. Reson. 98, 428-435 (1992)

2D Constant-time Heteronuclear Correlations

Phase-sensitive Constant-time ge-2D HSQC (CT-HSQC)

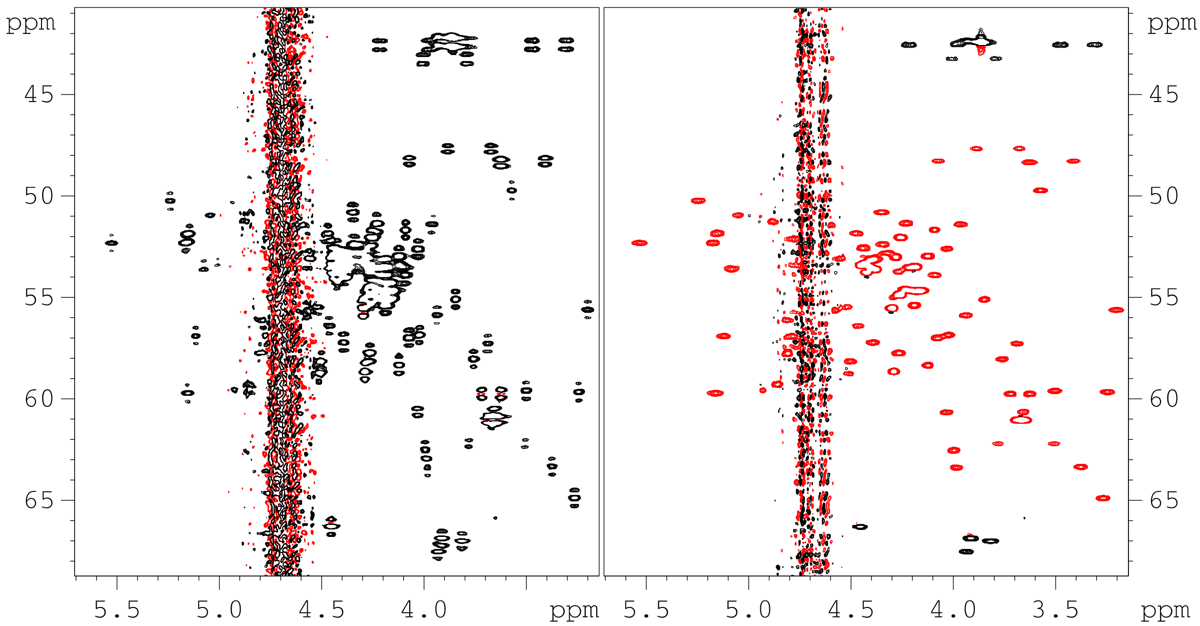
- Using adiabatic pulses (**hsqcctetgpsp**)
- Using adiabatic pulses without CO refocusing (**hsqcctetgpsp.2**)
- Using adiabatic pulses and PEP (**hsqcctetgpsisp**)
- 2D H-1/C-13 CT-HSQC for measuring 1J(CH) via J-modulation (**hsqcctetgpjc**)

Phase-sensitive Constant-time ge-2D HMQC (CT-HMQC)

- Using adiabatic pulses (**hmqcctetgp**)
- For correlating CH_2 groups (**hmqcctetgp.2**)

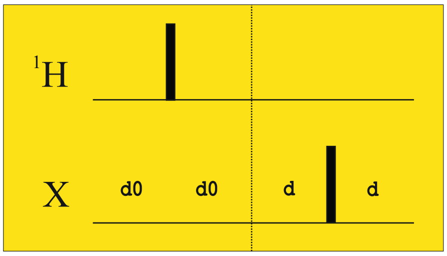
Also see in Coupling Constants in Proteins:
2D spin-echo difference CT-HSQC (hsqcctetgpjclr | HSQCCTETGPJCLR) - 3J[CO-CB] via spin-echo difference

Also see: 2D HMQC and 2D HSQC experiments



HSQC **CT-HSQC**

Evolution Period in Conventional HSQC

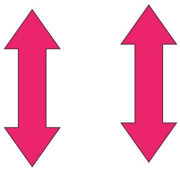


Overall Duration: 2d+2d0

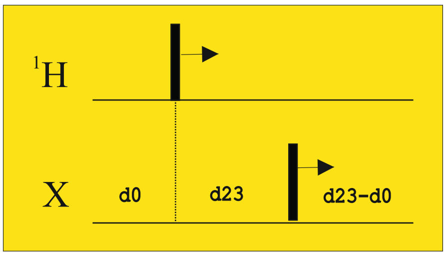
X Chemical shift: d0 + d0 + d - d = 2d0

J(XH) Coupling Constant: d0 - d0 - d + d = 0

J(HH) and J(XX) Coupling Constant: d0 + d0 + d + d = 2d0 + 2d



Evolution Period in Constant-Time HSQC



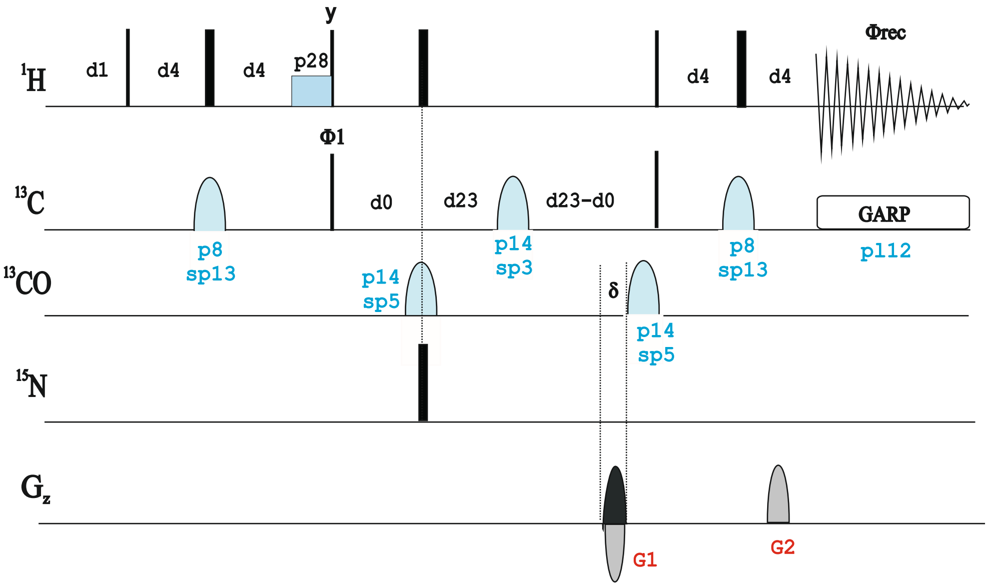
Variable Constant-time period
Overall Duration: 2d23

X Chemical shift: d0 + d23 - (d23-d0) = 2d0

J(XH) Coupling Constant: d0 - d23 + (d23-d0) = 0

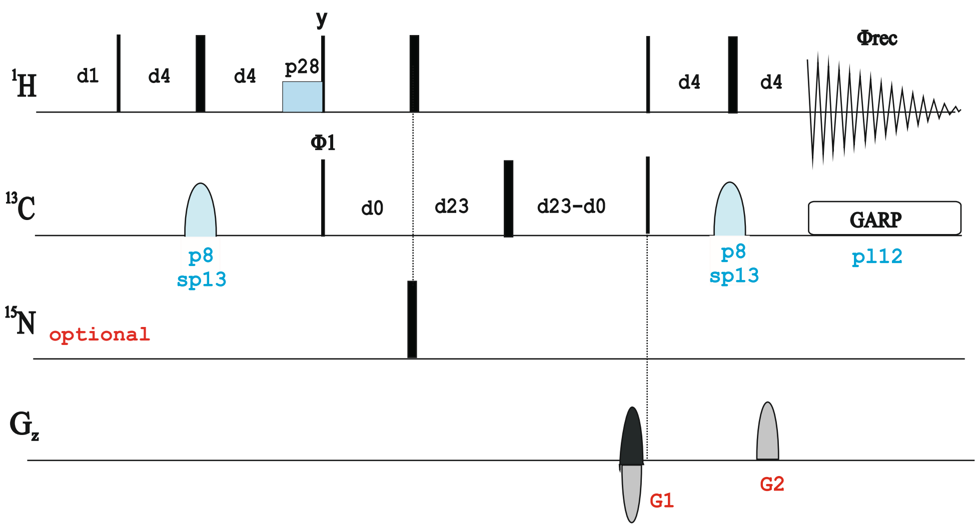
J(HH) and J(XX) Coupling Constant: d0 + d23 + (d23-d0) = 2d23

hsqcctetgpsp

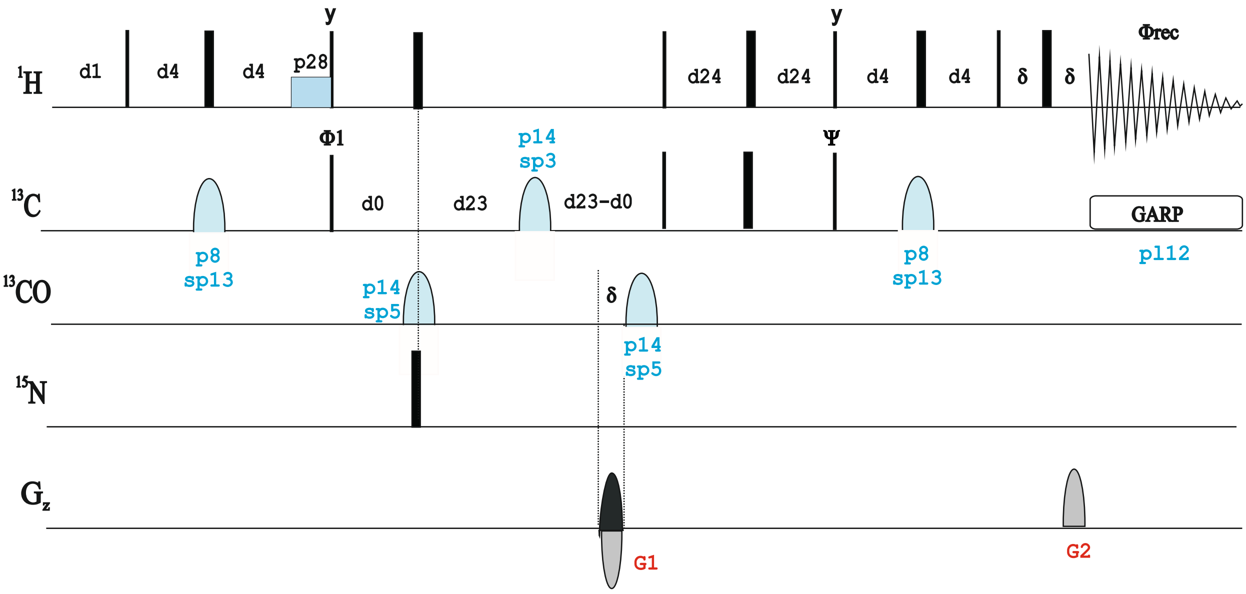


d23: d23 = T : 13.3 or 26.6 msec
2T (constant time period) = n/J(CC)

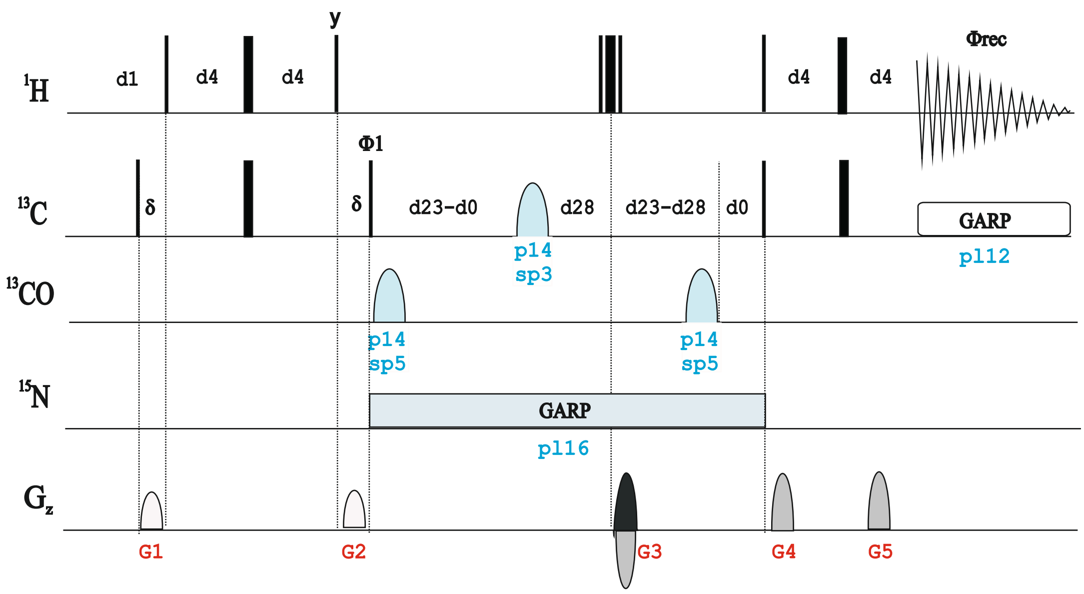
hsqcctetgpsp.2



hsqcctetgpsisp



hsqcctetgpjc



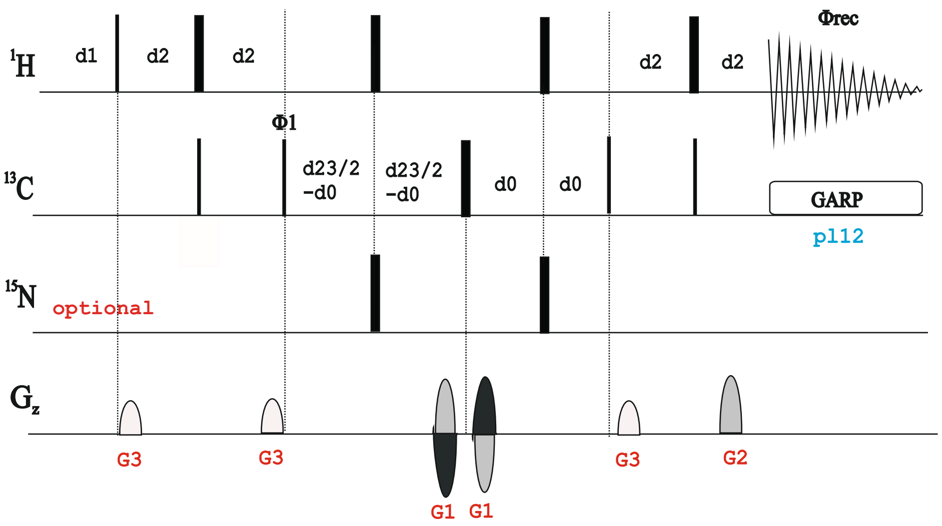
N. Tjandra & A. Bax, J. Magn. Reson. 124, 512-515 (1997)

CT-HMQC:

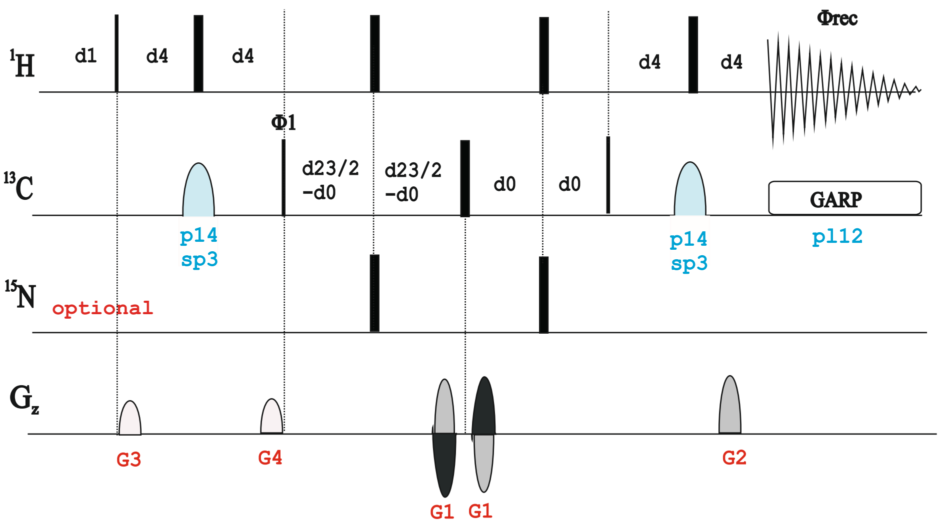
J.P. Marino, J.L. Diener, P.B. Moore & C. Griesinger, J. Am. Chem. Soc. 119, 7361-7366 (1997)

d23 = T ,2T (constant time period) = n/J(CC) [8.8 msec]

hmqcctetgp.2



hmqcctetgp



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2D INVERSE HETCOR EXPERIMENT

Experiment Description:

The 2D Inverse HETCOR experiment can be understood as the proton-detected version of the HETCOR experiment with the use of gradients for coherence selection. Experimentally, theinverse INEPT experiment is quite similar to an HSQC expèriment with the major difference that the pulse scheme starts from carbon magnetization instead of proton magnetization.

Sample and hardware requirements are the same as described for the HSQC experiment.

NMR Experiment Sensitivity

$$S/N \propto n\gamma_e \sqrt{\gamma_d^3 B_o^3 t}$$

	START NUCLEUS	DETECTED NUCLEUS
HETCOR	1H	13C
HMQC/HSQC	1H	1H
INVERSE INEPT	13C	1H

IMPORTANT: The pre-scan delay d1 must optimized as a function of the T1 values for the starting nucleus

Inverse HETCOR Experiments

- **1D Inverse INEPT/DEPT:**

1D inverse DEPT with refocusing and no decoupling (**ideptnd**)

1D inverse INEPT without refocusing and without decoupling (**iineptnd**)
1D inverse INEPT with refocusing and decoupling (**iineptrd**)

- **2D Inverse INEPT:**

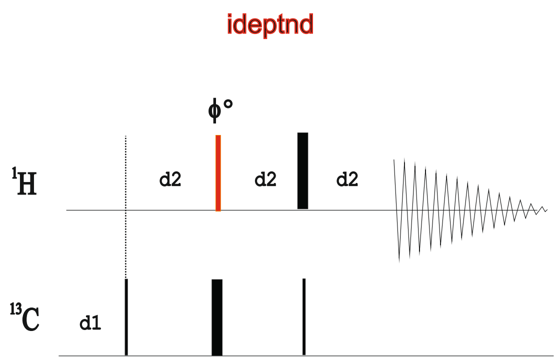
Phase-sensitive ge-2D Inverse INEPT using echo-antiecho (**xhcoetgp**)

Also see:
2D Inverse ^{1}H-^{31}P INEPT experiments for Nucleic Acids
Using echo-antiecho (na_xhcoetf3gp)
Constant-time using echo-antiecho (na_xhcoctetf3gp)

2D HETCOR, 2D HMQC and 2D HSQC experiments

Inverse DEPT:
M.R. Bendall, D.T. Pegg, D.M. Doddrell & J. Field, J. Mgn. Reson. 51, 520 - 526 (1983)

p0:
45 degree - all positive
90 degree - XH only
135 degree - XH, XH3 positive, XH2 negative

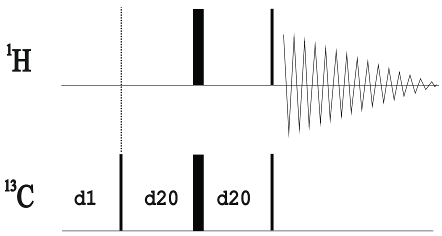


Inverse INEPT:

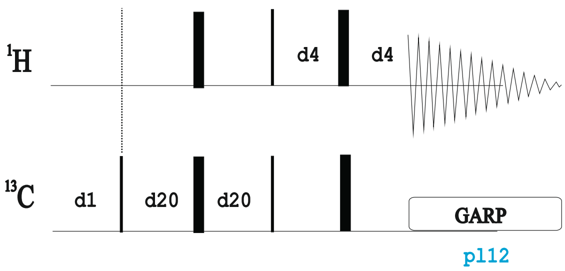
M.R. Bendall, D.T. Pegg, D.M. Doddrell & J. Field, J. Mgn. Reson. 51, 520 - 526 (1983)

d20: 1/(6J(XH)) for all multiplicities
d4 : 1/(4J(XH))

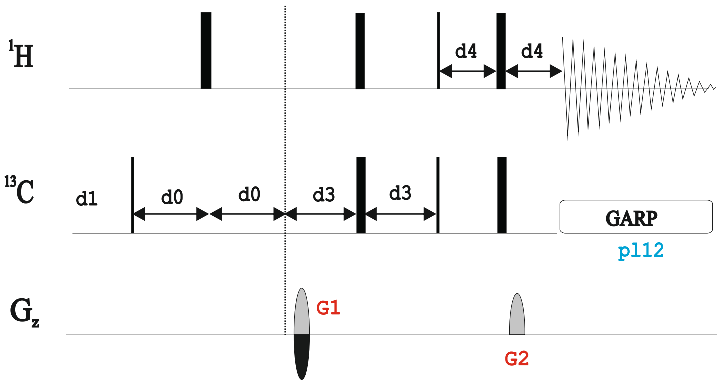
iineptnd



iineptrd



xhcoetgp



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2D MULTIPLICITY-EDITED HSQC EXPERIMENT

Experiment Description

The multiplicity edited HSQC experiment is a simple variant of the conventional HSQC pulse scheme designed to obtain simultaneouslythe heteronuclear correlation between 1H and X heteronuclei via the scalar coupling constant, 1J(XH), and the multiplicity of each cross-peak

Sample Requirements

Multiplicity-edited HSQC experiments can be recorded on any type of sample.

Hardware Requirements

Multiplicity-edited HSQC experiments can be recorded on any probehead but an inverse probe equipped with gradients is strongly recommended.

NMR Spectrum

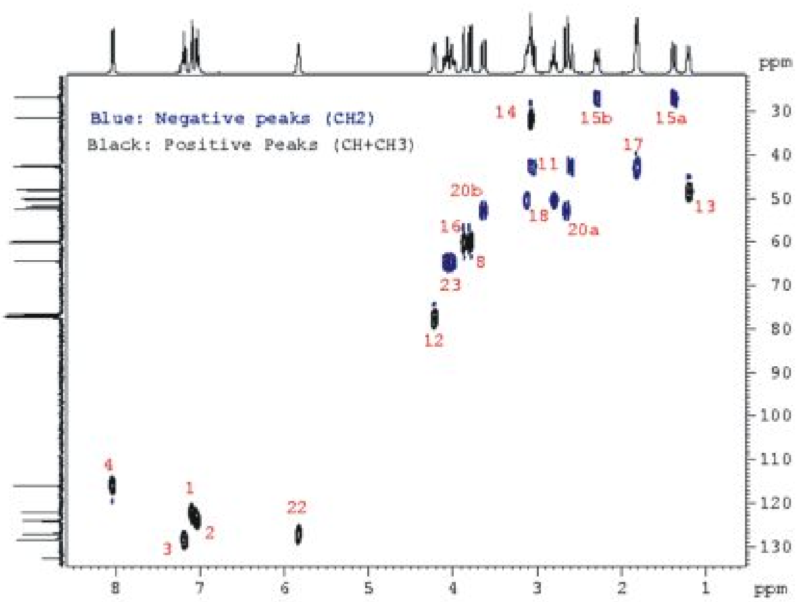
A 2D Multiplicity-edited HSQC map correlates 1H and X chemical shifts via 1J(XH). CH and CH3 cross peaks present opposite phase with respect to CH2 cross peaks.

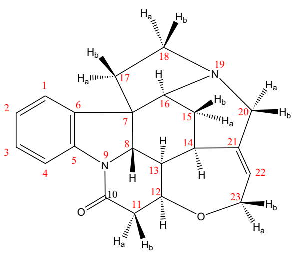
Related Experiments

1D DEPT and other carbon edited menthods
2D HSQC experiments
2D HMQC-DEPT

References:

W. Willker, D. Leibfritz, R. Kerssebaum & W. Bermel, Magn. Reson. Chem. 31, 287-292 (1993)





2D Multiplicity-edited HSQC Experiments

- **<u>Gradient-enhanced form f2 channel</u>**

  Phase-sensitive ge-2D multiplicity-edited HSQC using z-filter (**hsqcedgpph** | HSQCEDGPPH)

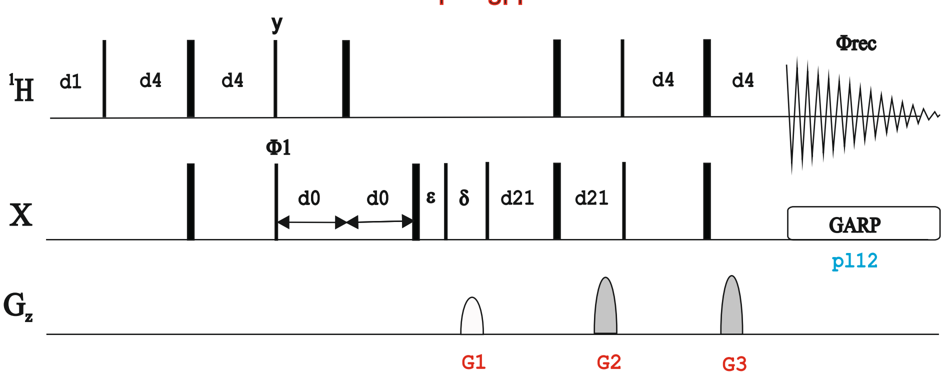
  Phase-sensitive ge-2D multiplicity-edited HSQC using echo-antiecho (**hsqcedetgp** | HSQCEDETGP)
  Phase-sensitive ge-2D multiplicity-edited HSQC using echo-antiecho and adiabatic pulses (**hsqcedetgpsp**)
  Phase-sensitive ge-2D multiplicity-edited HSQC using echo-antiecho and inversion and matched sweep adiabatic pulses (**hsqcedetgpsp.3**)

  Phase-sensitive ge-2D multiplicity-edited HSQC using PEP and adiabatic inversion pulses (**hsqcedetgpsisp**)
  Phase-sensitive ge-2D multiplicity-edited HSQC using PEP and adiabatic inversion and refocusing pulses (**hsqcedetgpsisp.2**)
  Phase-sensitive ge-2D multiplicity-edited HSQC using PEP and adiabatic inversion pulses with gradients in back-inept (**hsqcedetgpsisp2**)
  Phase-sensitive ge-2D multiplicity-edited HSQC using PEP and adiabatic inversion and refocusing pulses with gradients in back-inept (**hsqcedetgpsisp2.2**)
  Phase-sensitive ge-2D multiplicity-edited HSQC using PEP and inversion, refocusing and matched sweep adiabatic pulses with gradients in back-inept (**hsqcedetgpsisp2.3**)
  Phase-sensitive ge-2D multiplicity-edited HSQC using PEP and inversion, refocusing and matched sweep adiabatic pulses with gradients in back-inept with HSQC-COSY peak suppression (**hsqcedetgpsisp2.4**)

- **<u>Gradient-enhanced form f3 channel</u>**

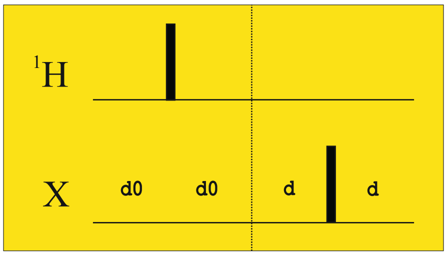
  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC-edited using PEP (**hsqcedetf3gpsi**)
  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC-edited using PEP with gradients in back-inept (**hsqcedetf3gpsi2**)

hsqcedgpph



gp 1 : gp 2 : gp 3
30:80:20.1 for C-13
30:80:8.1 for N-15

Evolution Period in Conventional HSQC



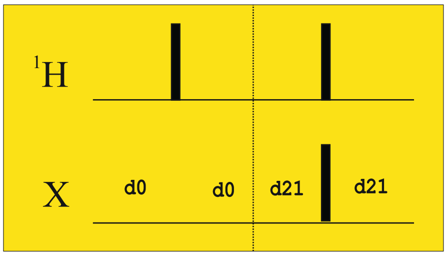
Overall Duration: 2d+2d0

X Chemical shift: d0 + d0 + d - d = 2d0

J(XH) Coupling Constant: d0 - d0 - d + d = 0

J(HH) and J(XX) Coupling Constant: d0 + d0 + d + d = 2d0 + 2d

Evolution Period in Multiplicity-edited HSQC



Overall Duration: 2d21+2d0

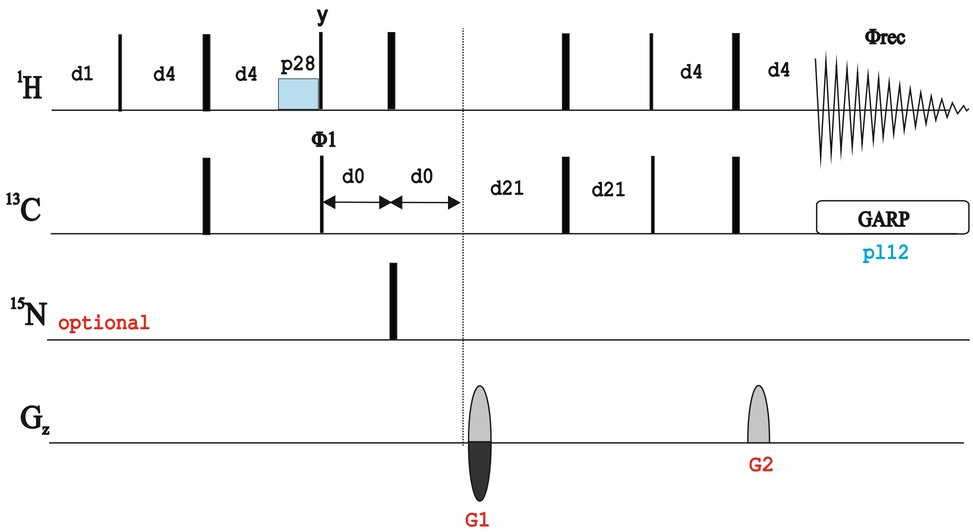
X Chemical shift: (d21+d0) + d0 - d21 = 2d0

J(XH) Coupling Constant: (d21+d0) - d0 + d21 = 2d21

J(HH) and J(XX) Coupling Constant: (d21+d0) +d0 + d21 = 2d21 +2d0

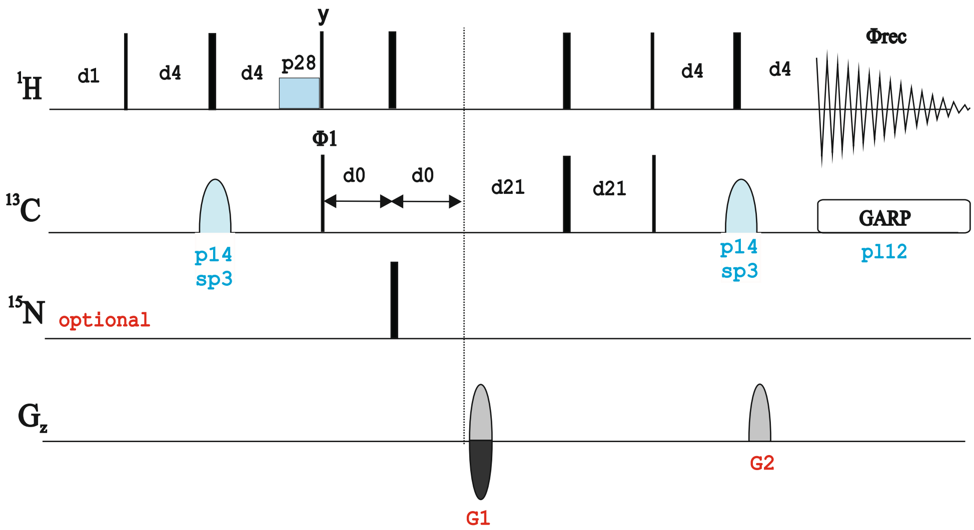
d21: set d21 according to multiplicity selection
1/(2J(XH)) XH, XH3 positive, XH2 negative

hsqcedetgp

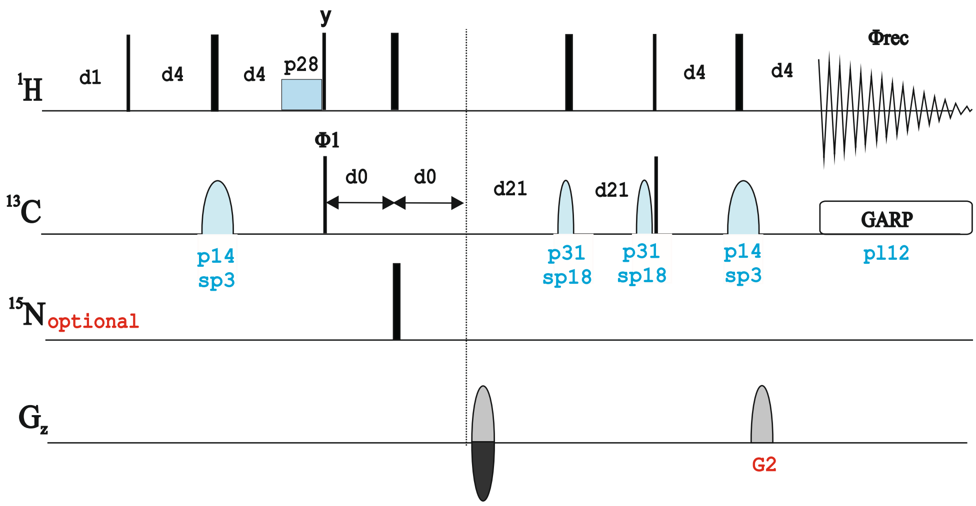


gp 1 : gp 2
80:20.1 for C-13
80:8.1 for N-15

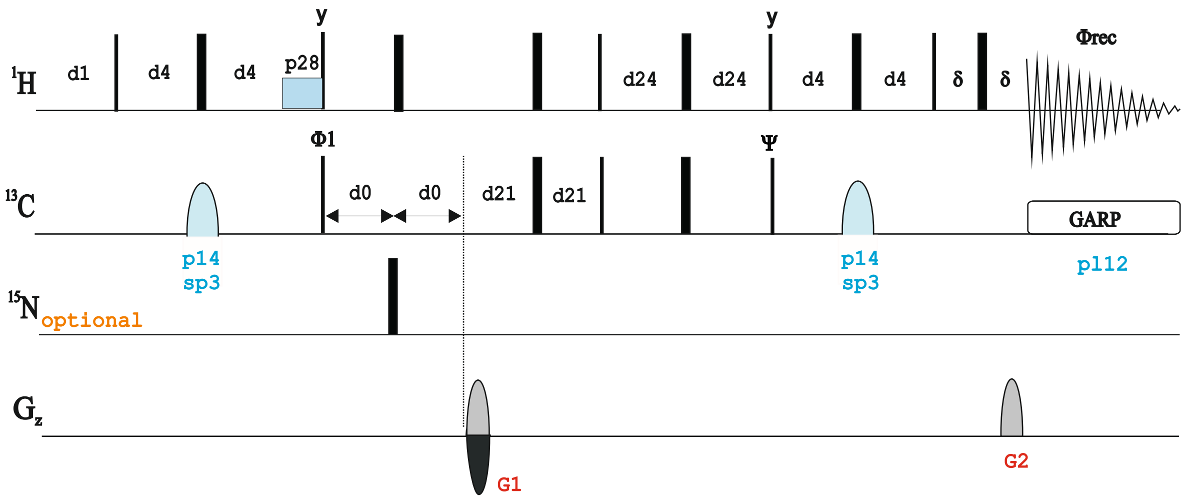
hsqcedetgpsp



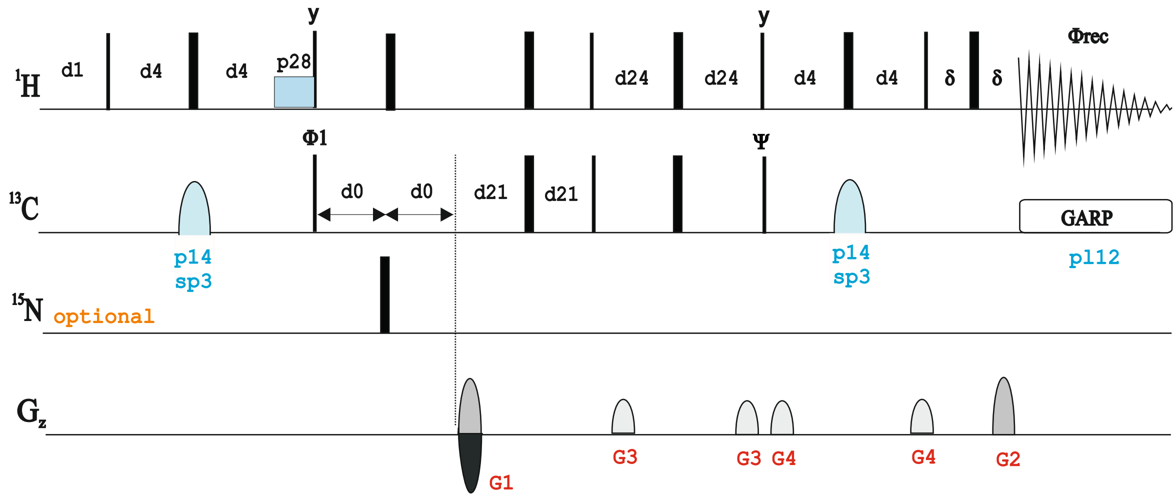
hsqcedetgpsp.3

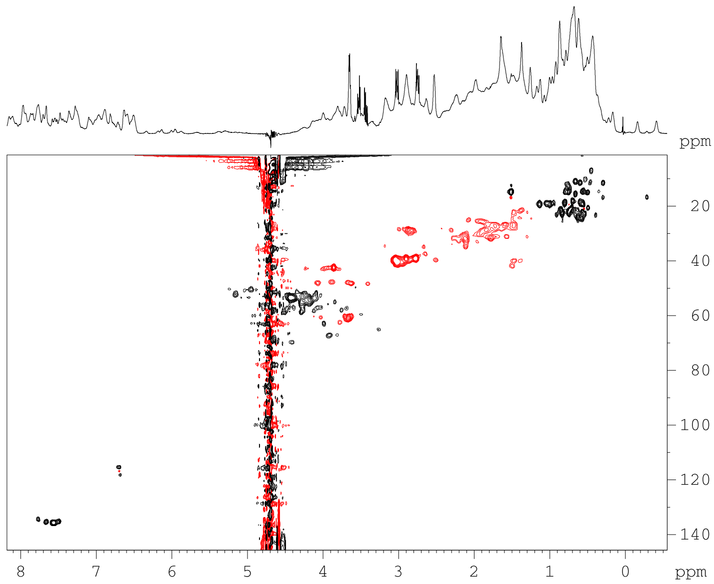


hsqcedetgpsisp

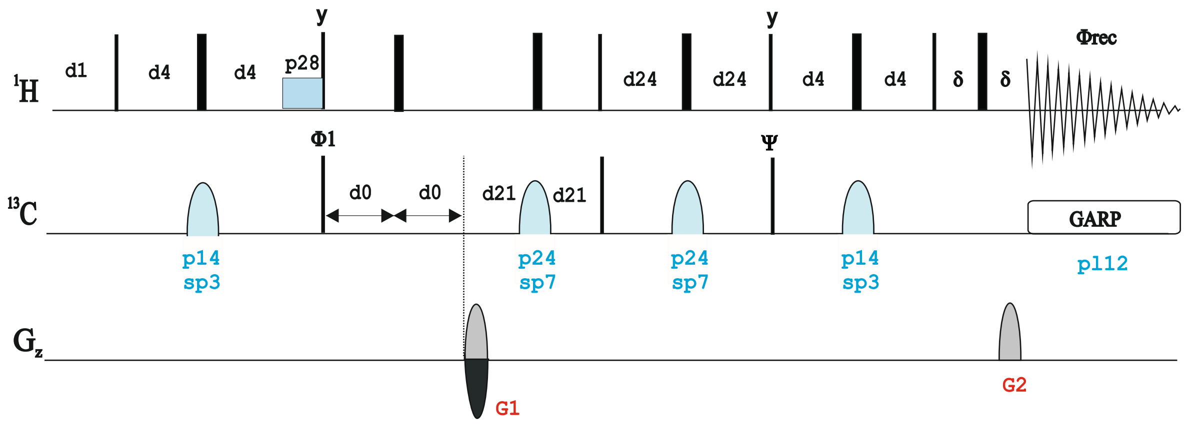


hsqcedetgpsisp2

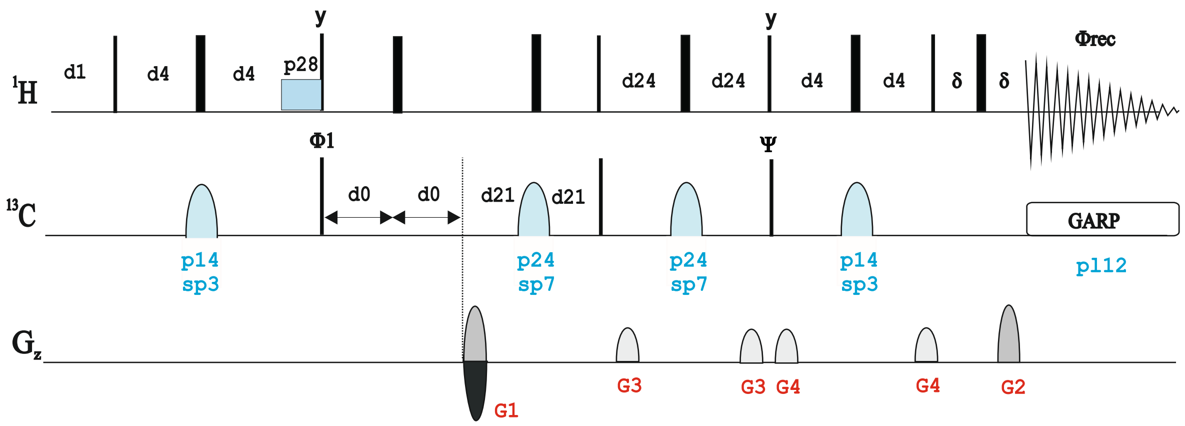




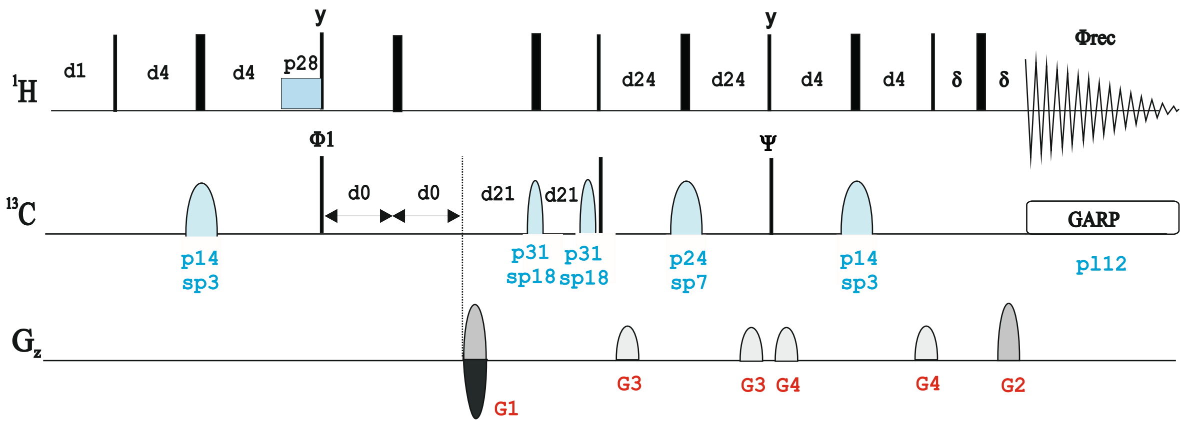
hsqcedetgpsisp.2



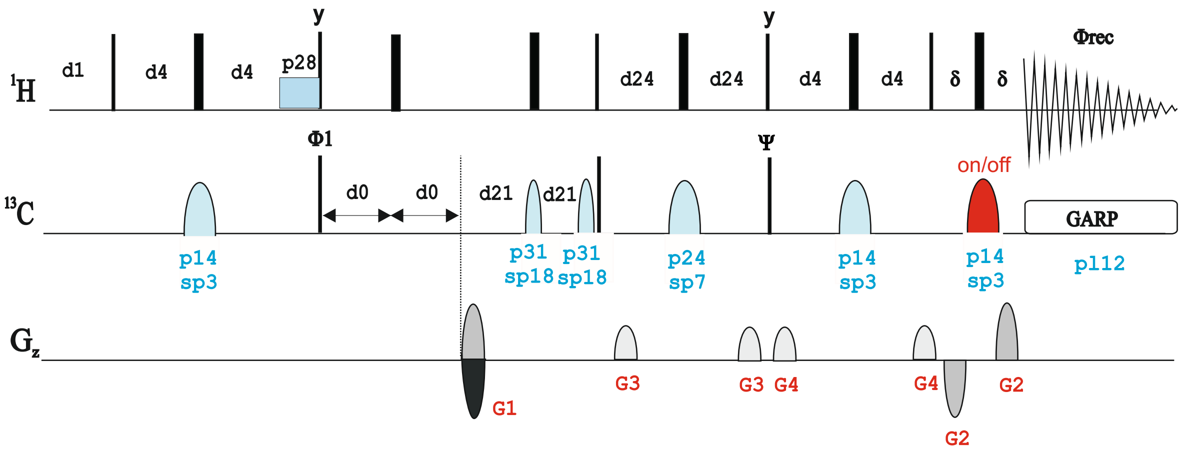
hsqcedetgpsisp2.2



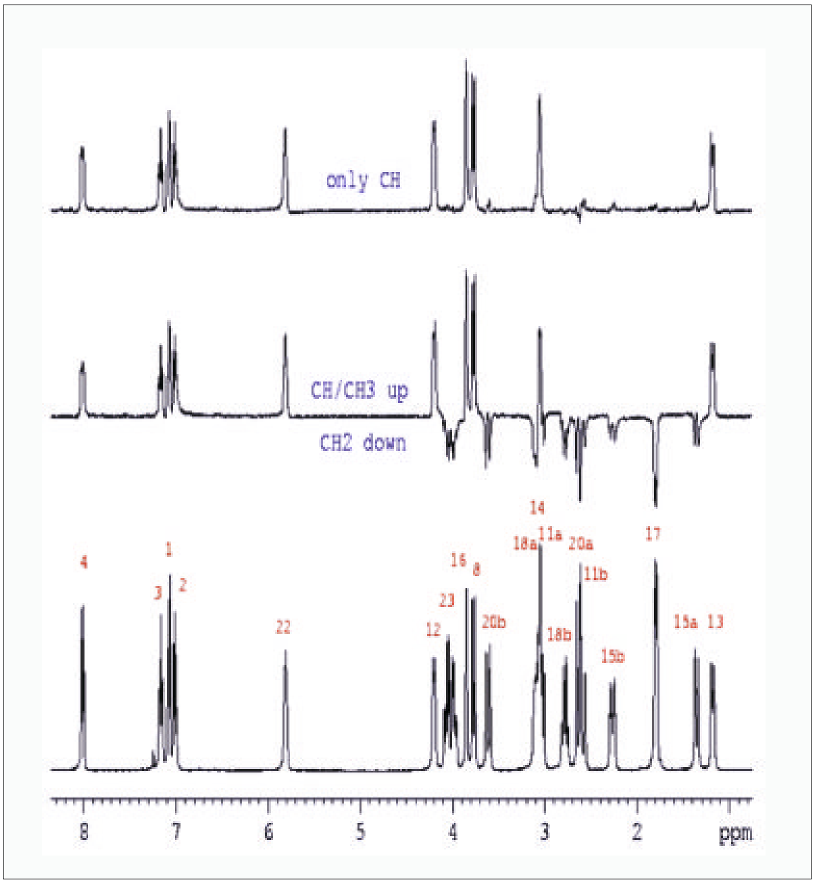
hsqcedetgpsisp2.3

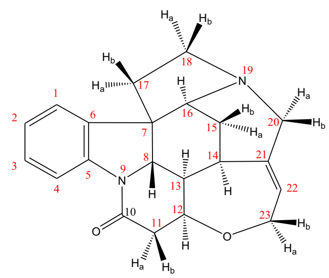


hsqcedetgpsisp2.4

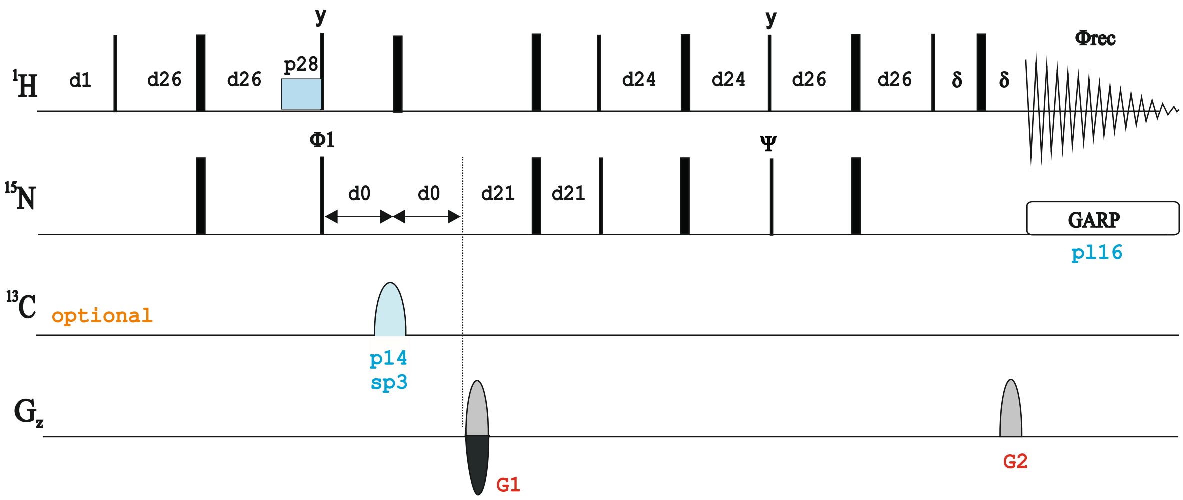


Edited-HSQC in 1D mode:

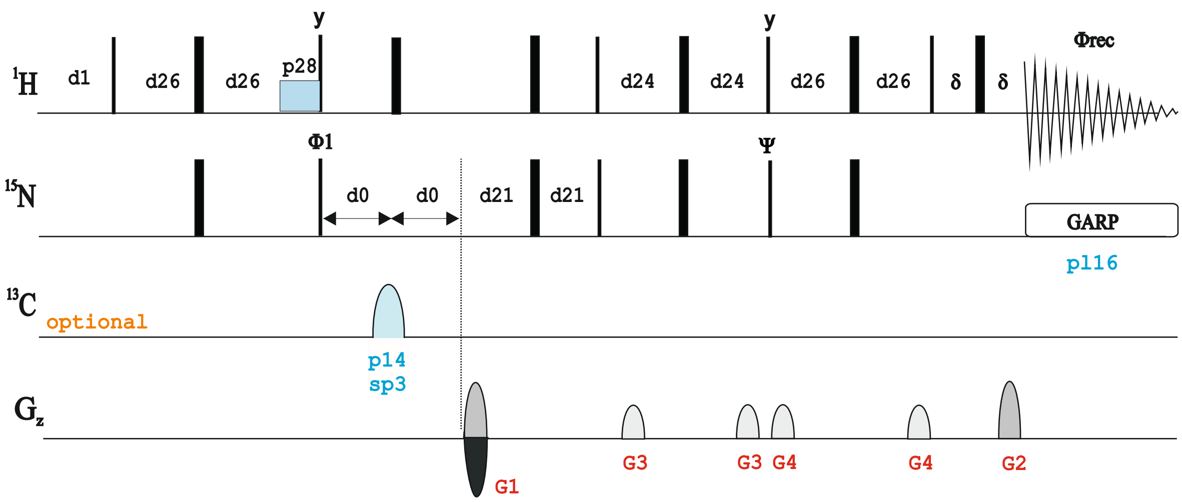


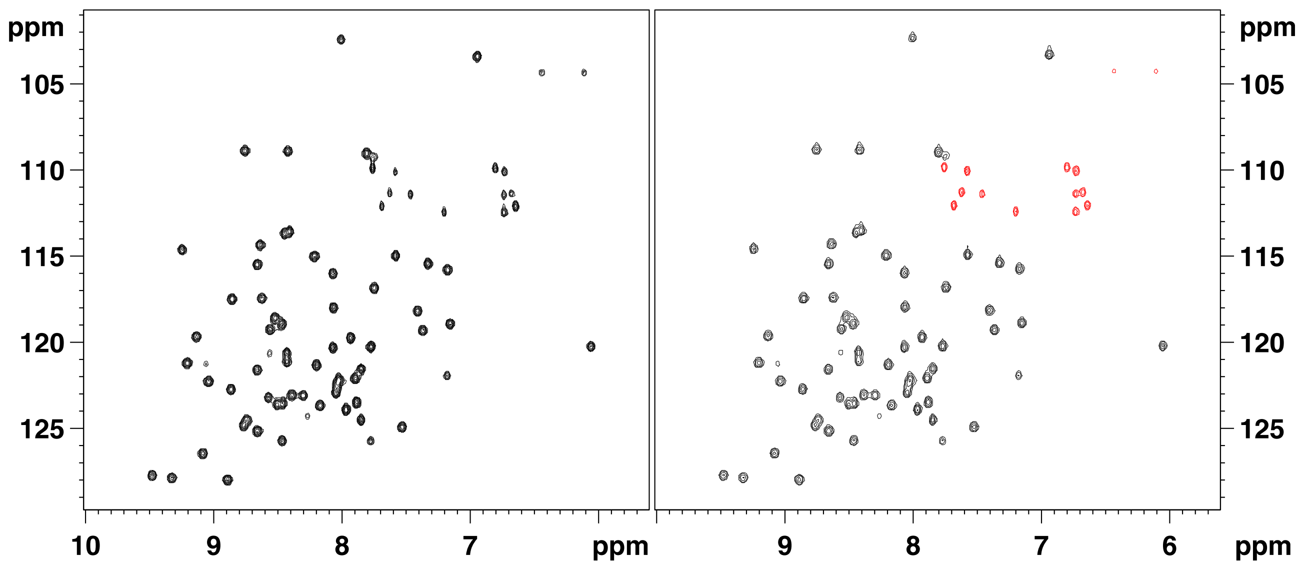


hsqcedetf3gpsi



hsqcedetf3gpsi2





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2D DEPT-HMQC EXPERIMENT

Experiment Description:
The 2D DEPT-HMQC experiment is a variant of the HMQC experiment in which a DEPT pulse train has been incorporated as an initial preparation block to obtain carbon multiplicity information.
Also see multiplicity-edited HSQC experiment for similar purposes.

References:
H. Kessler, P. Schmieder & M. Kurz, J. Magn. Reson 85, 400-405 (1989)

HMQC-DEPT Experiments

1D DEPT-HMQC

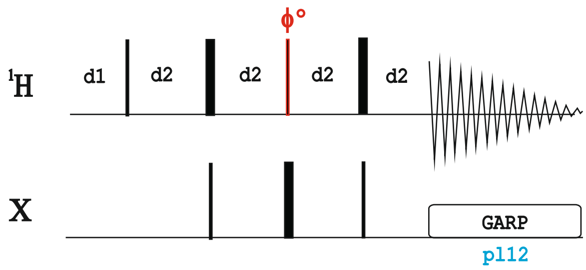
1D DEPT-HMQC with refocusing and decoupling (**indecord1d**)
1D DEPT-HMQC using BIRD with refocusing and decoupling (**indecobird1d**)

2D DEPT-HMQC

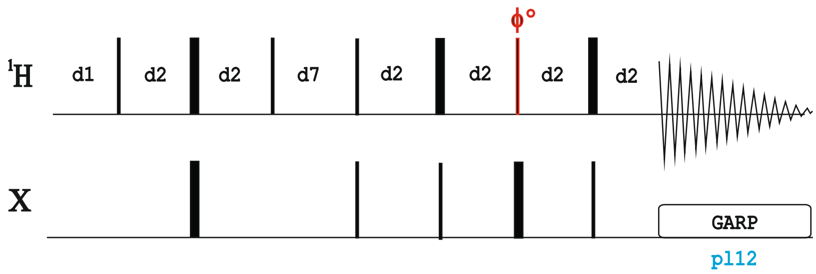
Phase-sensitive 2D DEPT-HMQC (**indecoph**)
Phase-sensitive 2D DEPT-HMQC using BIRD (**indecobiph**)
Phase-sensitive 2D DEPT-HMQC-TOCSY using BIRD (**indecobimlph**)

Also see:
2D HMQC Experiments

indecord1d

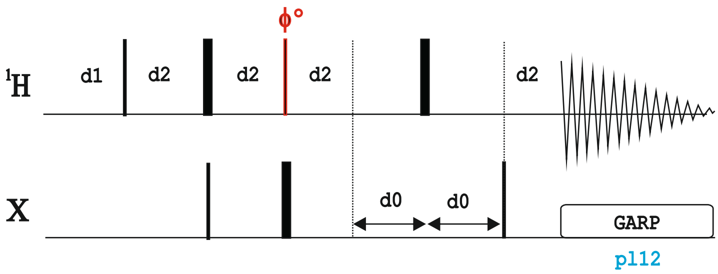


indecobird1d

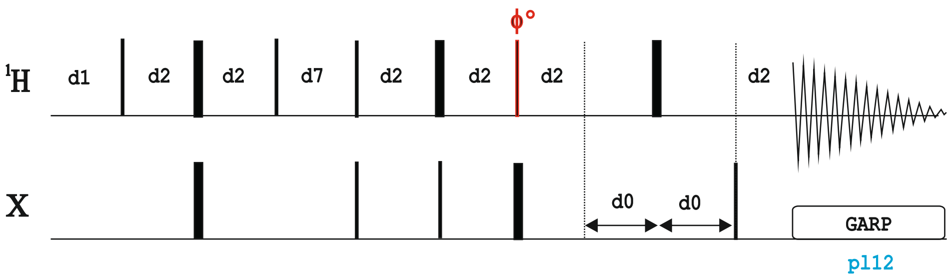


p0 in 1D DEPT-HMQC:
60 degree - XH positive, XH2, XH3 negative
90 degree - XH2 only
120 degree - XH3 positive, XH, XH2 negative
180 degree - XH2 positive, XH, XH3 negative

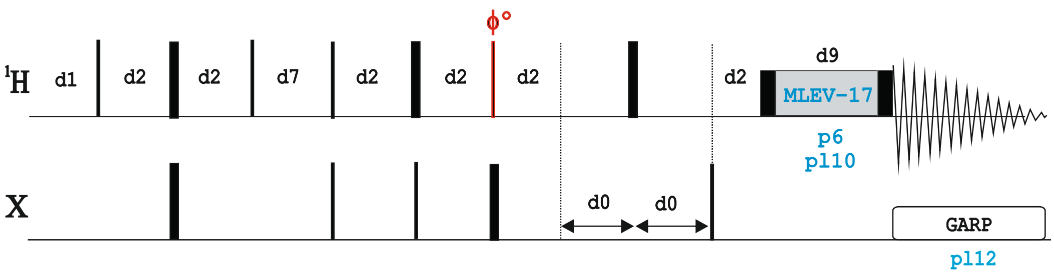
indecoph



indecobiph



indecobimlph



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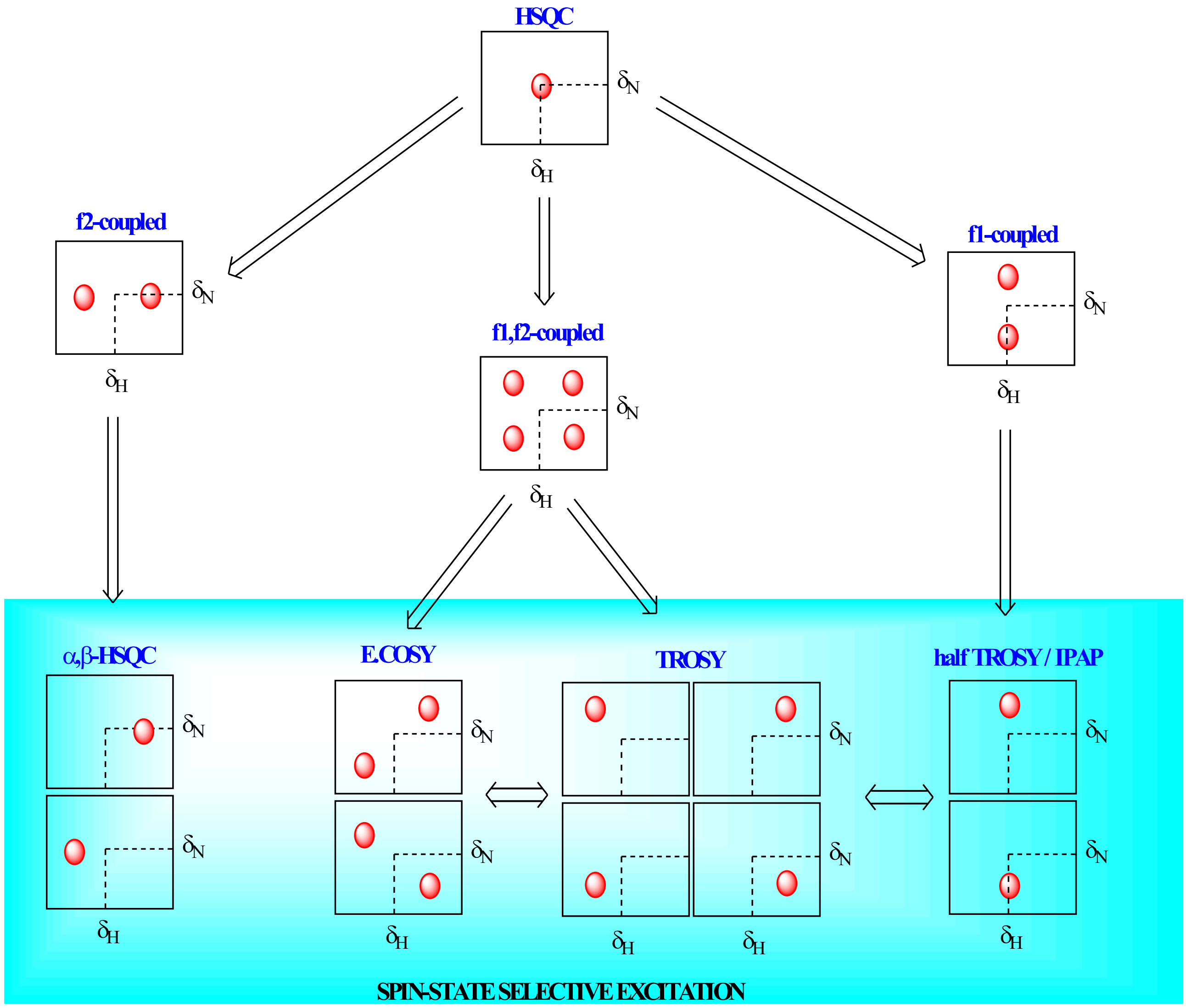
2D SPIN-STATE-EDITED HSQC EXPERIMENTS

The HSQC pulse scheme can be modified in a great number of ways in order to afford different versions that can be used for many different purposes.
Of particular interest are the spin-state edited versions that allows to simplify the analysis and interpretation of conventional coupled spectra resulting of recording HSQC experiments without decoupling during acquisition (F2-coupled), during d0 (F1-coupled) or during both periods (F1,F2 coupled).



2D Spin-state Edited HSQC Experiments

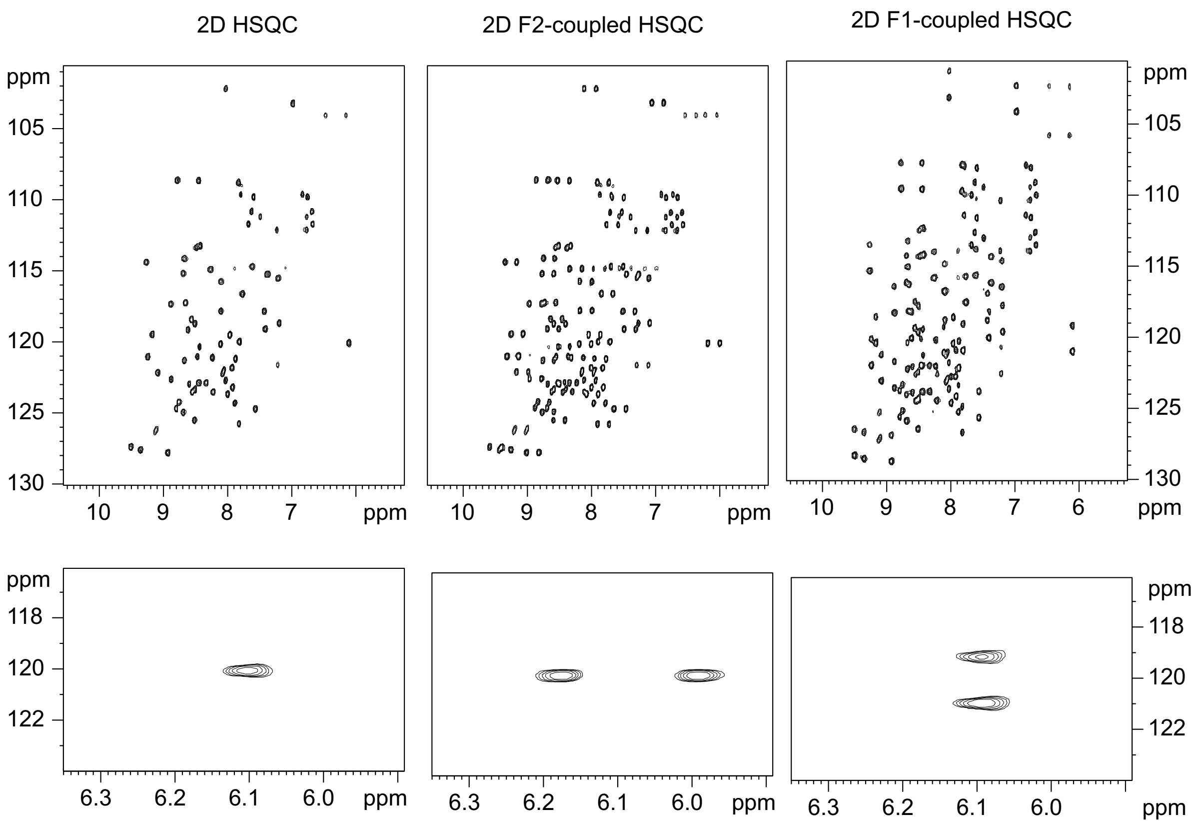
From f2 channel

- ge-2D ^{1}H-^{13}C HSQC-IPAP with adiabatic inversion pulses and IPAP editing in the indirect F1 dimension (**hsqcetgpiasp)**

From f3 channel

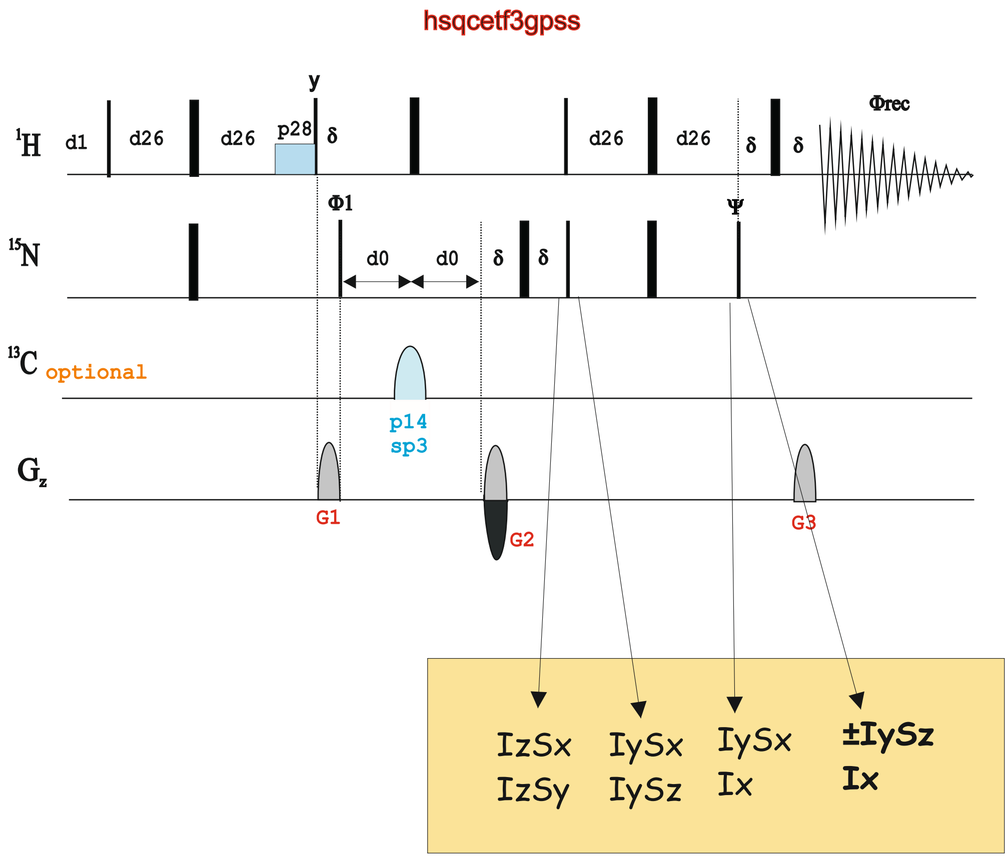
- ge-2D ^{1}H-^{15}N α,β-HSQC with IPAP editing in the F2 detected dimension (**hsqcetf3gpss)**
- ge-2D ^{1}H-^{15}N HSQC-IPAP using watergate with IPAP editing in the indirect F1 dimension (**hsqcf3gpiaphwg**)
  ge-2D ^{1}H-^{15}N HSQC-IPAP using watergate with similar IP and AP delays and IPAP editing in the indirect F1 dimension (**hsqcf3gpiaphwg.2**)
- ge-2D ^{1}H-^{15}N HSQC-IPAP using watergate, water flip-back and sensitivity improvement and with IPAP editing in the indirect F1 dimension (**hsqcf3gpiaphsiwg**)

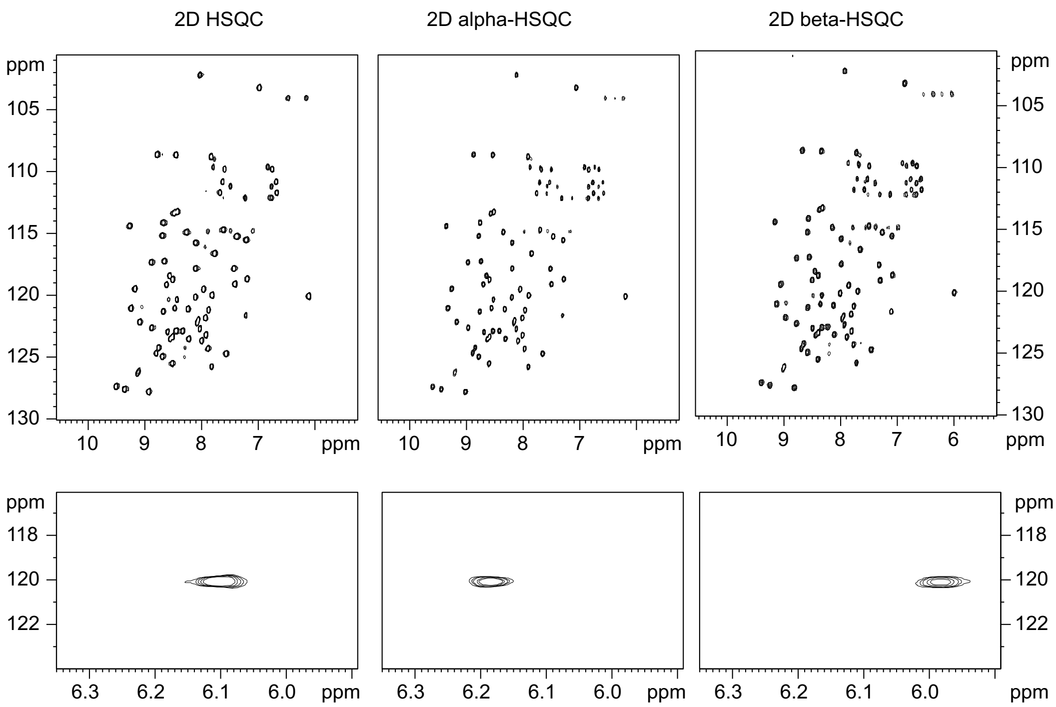
Also see all experiments in "2D TROSY experiments". TROSY is a special case of spin-state selection.



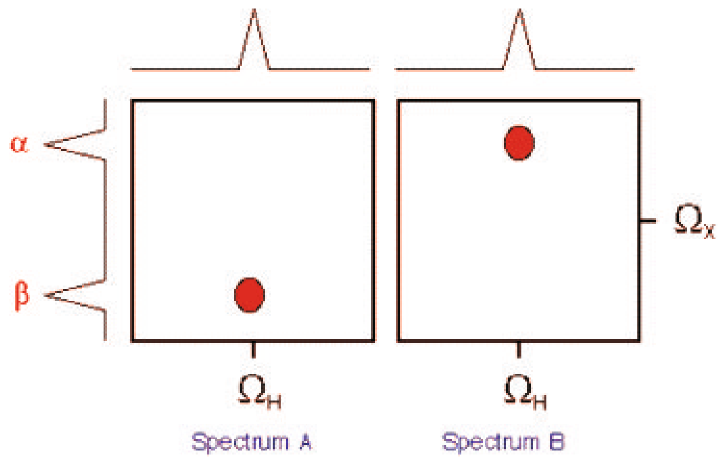
Comparison of 1H-15N HSQC spectra of ubiquitin: decoupled (left), F2-coupled (middle) and F1-coupled (right).

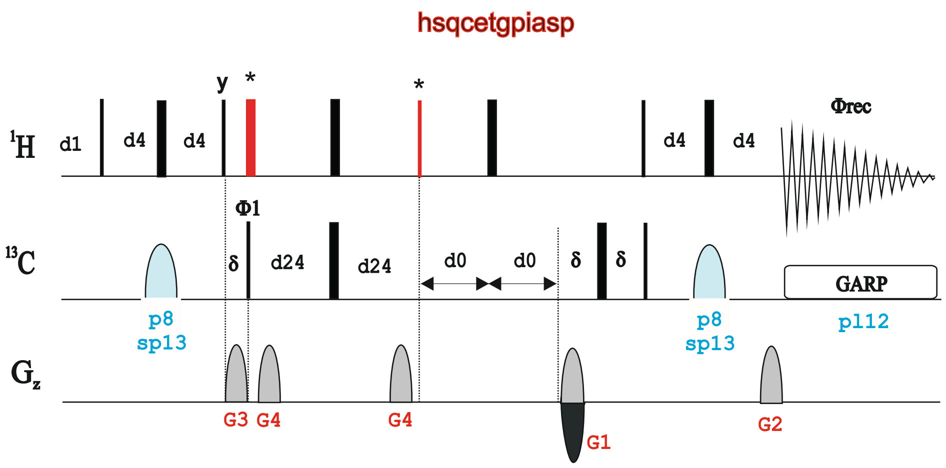
Spin-State Selection in Reverse INEPT





Comparison of 1H-15N HSQC spectra of ubiquitin: decoupled (left), alpha-spin-edited (middle) and beta-spin-eited (right).

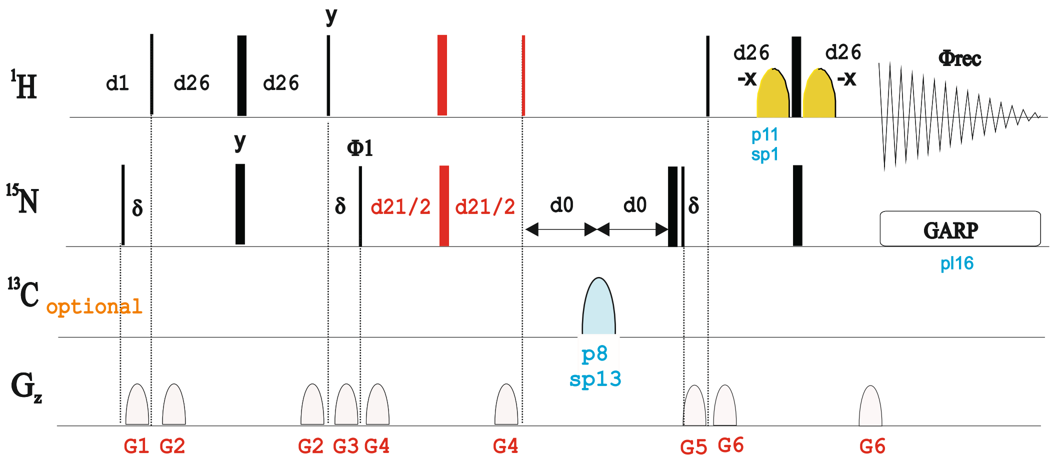

α
β
Ω
Ω
Ω
Spectrum A
Spectrum B


hsqcetgpiasp
y
*
*
d1
d4
d4
d4
d4
Φrec
Φ1
δ
d24
d24
d0
d0
δ
δ
GARP
p8
sp13
p8
sp13
p112
G3
G4
G4
G1
G2

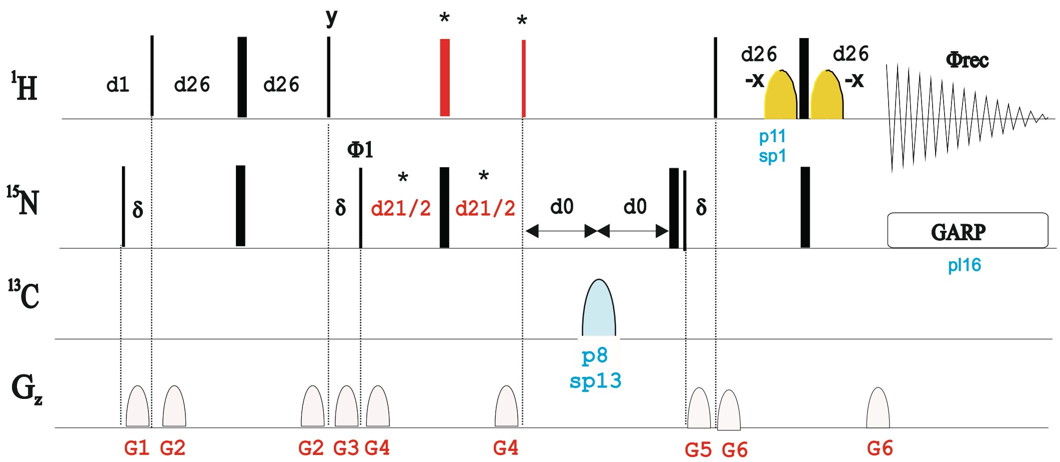
IPAP-HSQC

1. M.Ottiger, F. Delaglio & A. Bax, J. Magn. Reson. 131, 373-378 (1998)
2. F. Cordier, A.J. Dingley & S. Grzesiek, J. Biomol. NMR 13, 175-180 (1999)

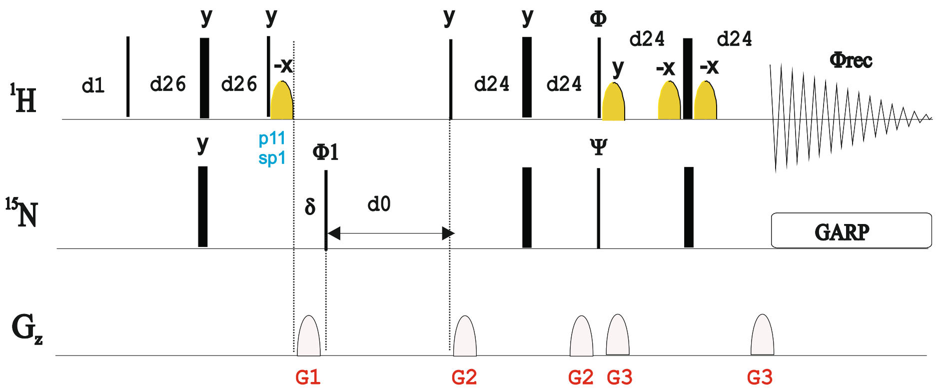
hsqcf3gpiaphwg



hsqcf3gpiaphwg.2



hsqcf3gpiaphsiwg



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2D TROSY EXPERIMENTS

Experiment Description

The TROSY (Transverse-Relaxation Optimized SpectroscopY) pulse scheme can be understood as a spin-state-edited HSQC experiment in which only one of the four components of a fully-F1,F2-coupled multiplet is observed.

Sample Requirements

TROSY experiments are usually applied in large biomolecules labeled with 15N.

Hardware Requirements

TROSY experiments are recorded in triple-resonance inverse probeheads equipped with gradients.

NMR Spectrum

The TROSY experiment afford a conventional 1H-Xchemical shift correlation map

Related Experiments

2D HSQC
Also see 3D TROSY-like triple-resonance NMR experiments applied on labeled proteins.

References:

1. M. Czisch & R. Boelens, J. Magn. Reson. 134, 158-160 (1998)
2. K. Pervushin, G. Wider & K. Wuethrich, J. Biomol. NMR 12, 345-348 (1998)
3. A. Meissner, T. Schulte-Herbrueggen, J. Briand & O.W. Sorensen, Mol. Phys. 96, 1137-1142 (1998)
4. J. Weigelt, J. Am. Chem. Soc. 120, 10778-10779 (1998)
5. M. Rance, J.P. Loria & A.G. Palmer III, J. Magn. Reson. 136, 91-101 (1999)
6. G. Zhu, X.M. Kong & K.H. Sze, J. Biomol. NMR 13, 77-81 (1999)

2D TROSY Experiments

From f2 channel:

Phase-sensitive ge-2D TROSY with presaturation (**trosygpphpr**)
ge-2D TROSY for aromatic residues with WATERGATE (**trosyargpphwg**)

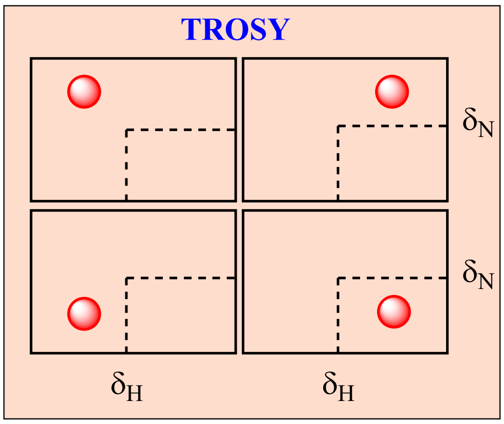
From f3 channel:

Phase-sensitive ge-2D ^{1}H-^{15}N TROSY

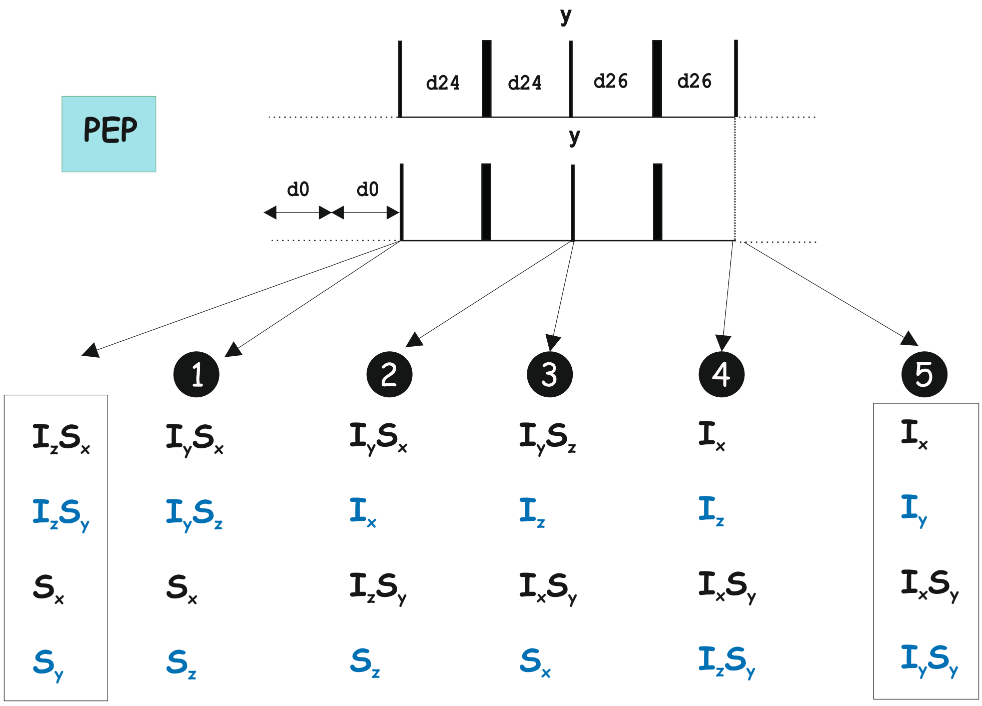
- Using echo-antiecho (**trosyetf3gpsi** | TROSYETF3GPSI)
- Using echo-antiecho (**trosyetf3gpsi.2**)
- Using echo-antiecho and water flip-back (**trosyetfpf3gpsi**)
- Using echo-antiecho and different phase cycling (**trosyetf3gpsi2**)
- Using echo-antiecho and different phase cycling to give IPAP TROSY (**trosyetf3gpiasi**)
- Using echo-antiecho and different phase cycling to give IPAP TROSY (**trosyetf3gpiasi.2**)
- Using WATERGATE (3-9-19) (**trosyf3gpph19** | TROSYF3GPPH19)
- Using WATERGATE and improved sensitivity (**trosyf3gpphsi19** | TROSYF3GPPHSI19)

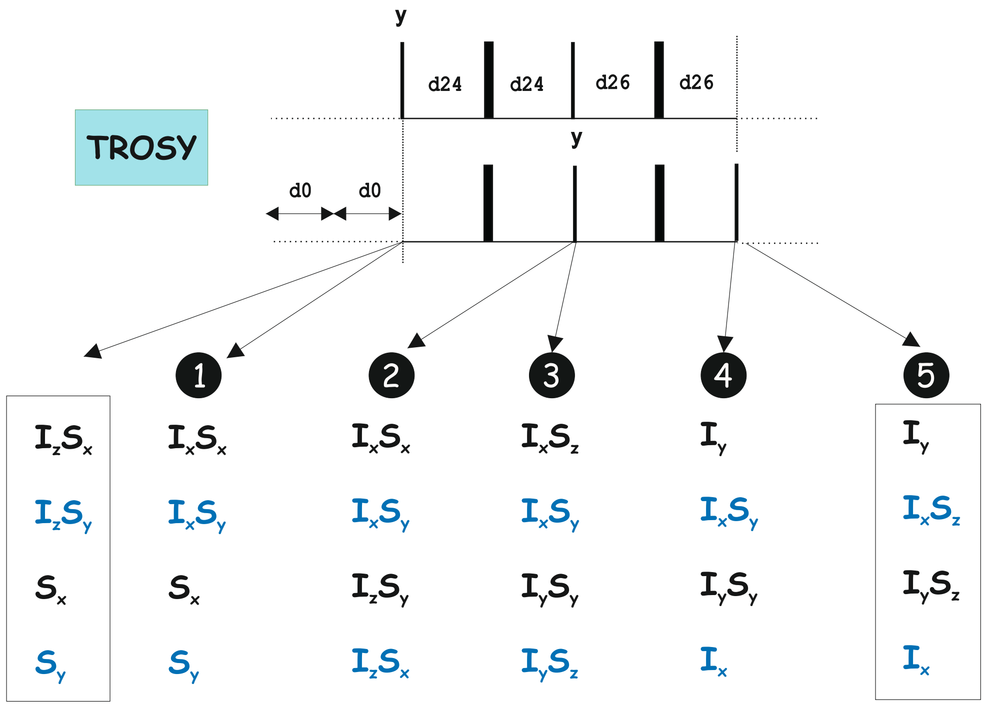
Phase-sensitive ge-2D ^{1}H-^{15}N ZQ-TROSY using WATERGATE (**trosyzqgpphwg**)

Also see:
Spin-State Edited HSQC-IPAP experiments
CRINEPT Experiments

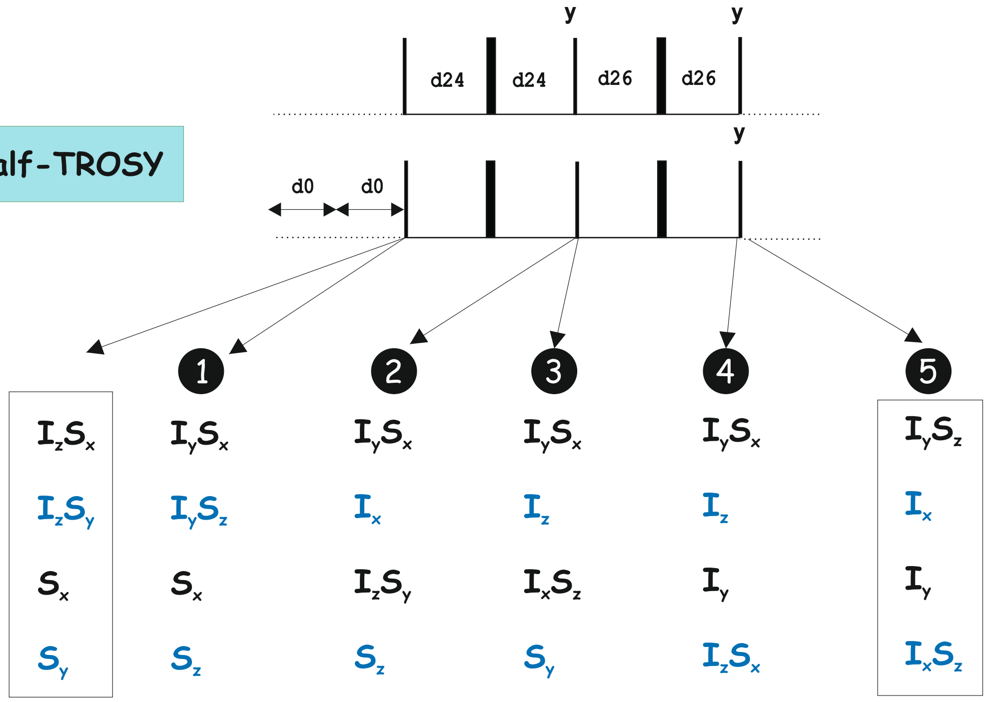


PEP vs TROSY and half-TROSY

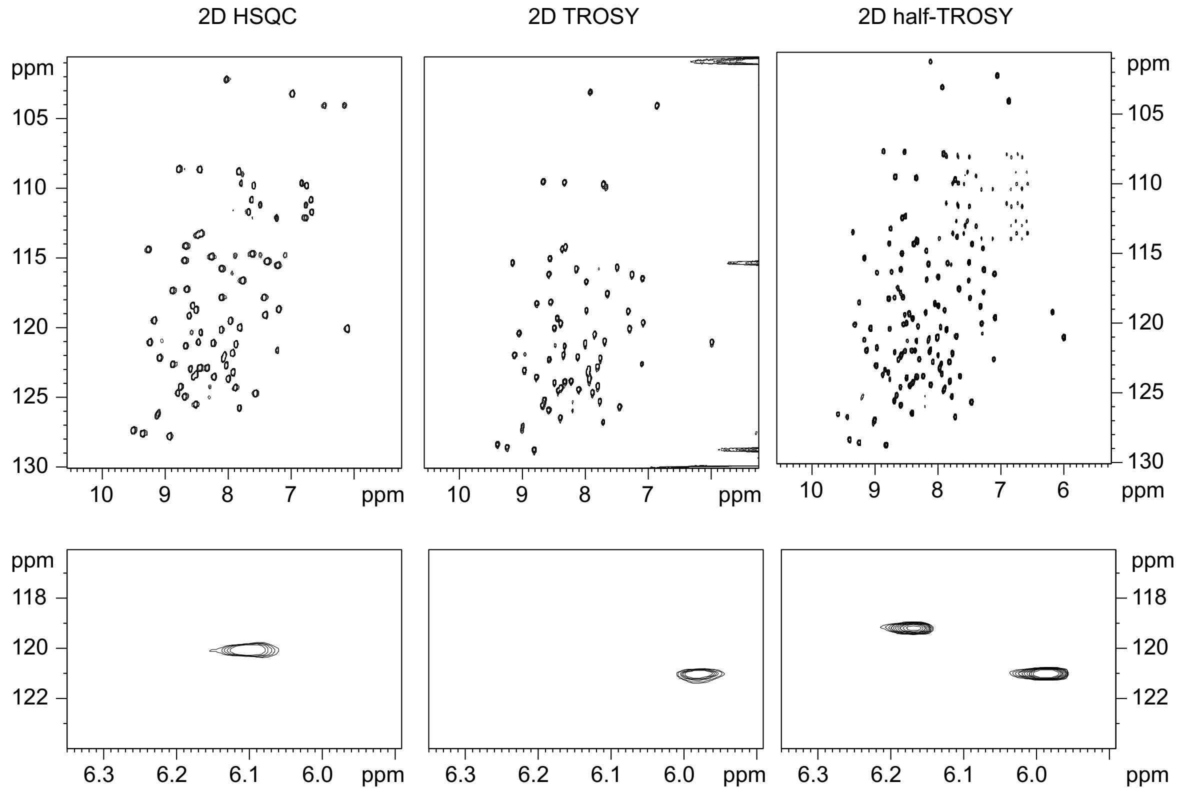




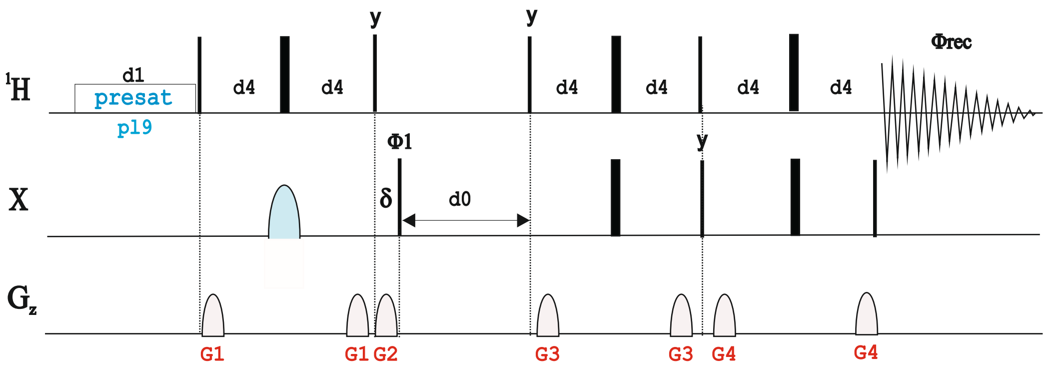
Half-TROSY



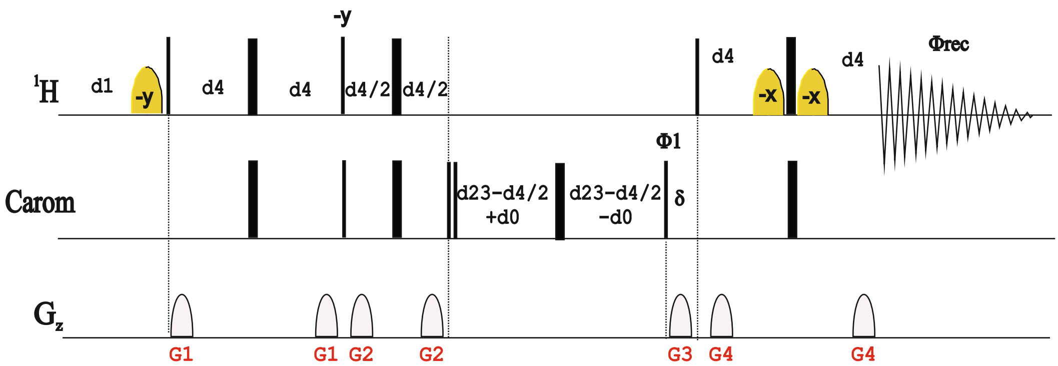
1. D. Nietlispach, J. Biomol. NMR 31, 161-166 (2005)
2. D.W. Yang & L.E. Kay, J. Biomol. NMR 13, 3-10 (1999)



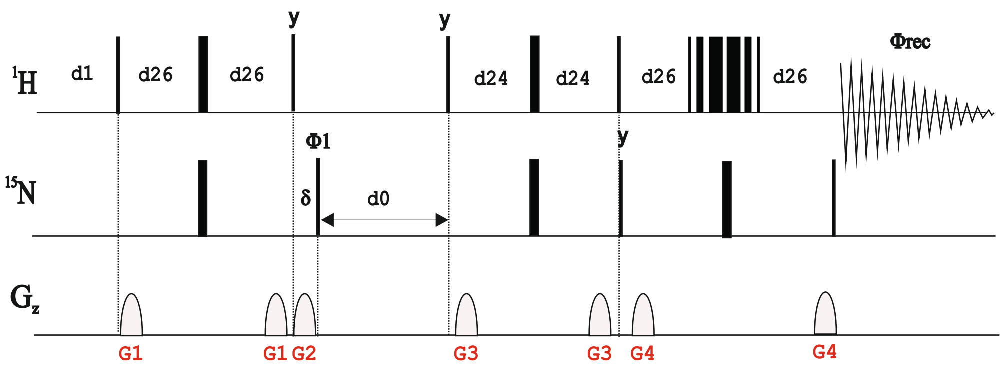
trosygpphpr



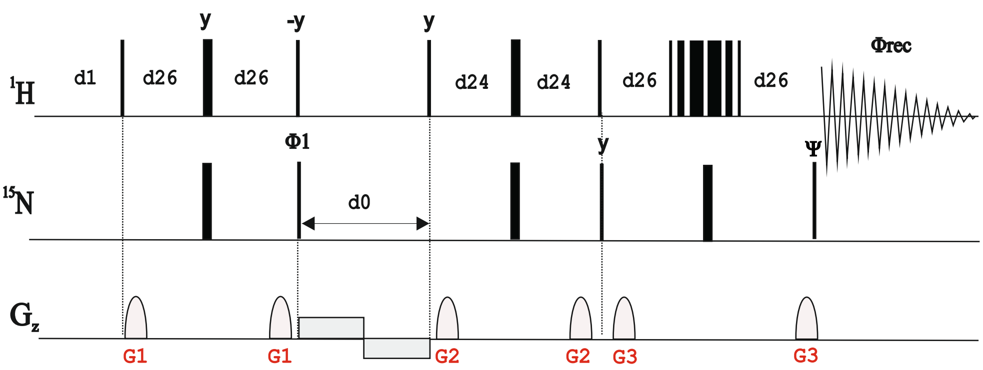
trosyargpphwg



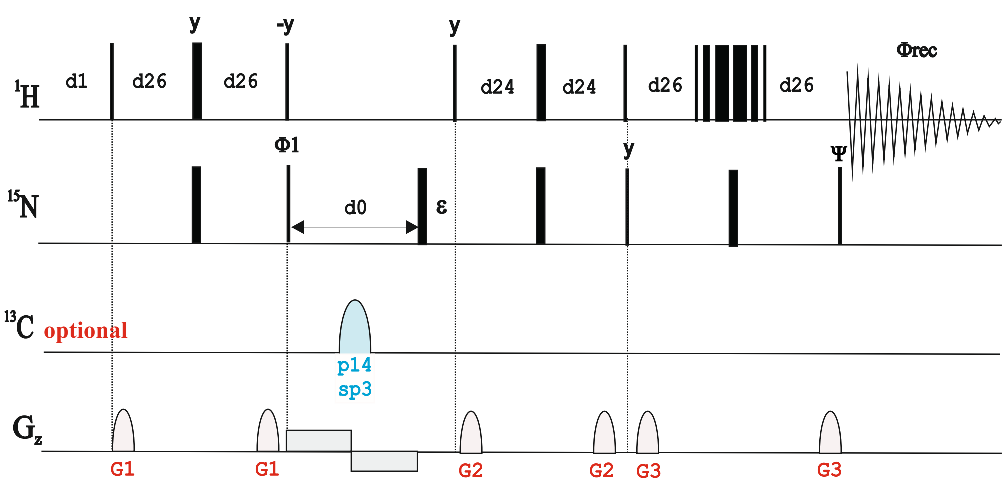
trosyf3gpph19



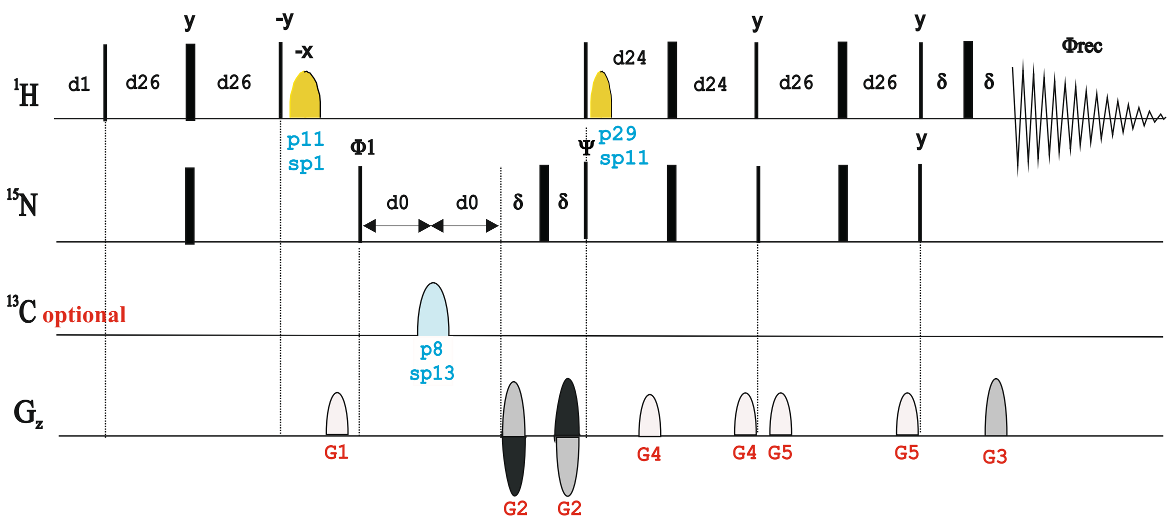
trosyf3gpphsi19



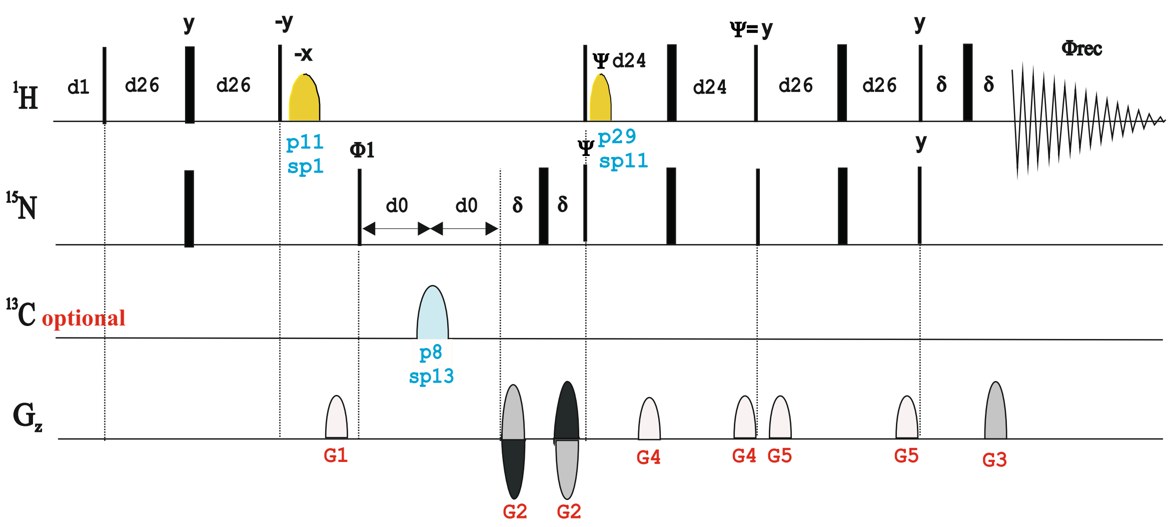
trosyf3gpphsi19.2



trosyetf3gpiasi



trosyetf3gpiasi.2



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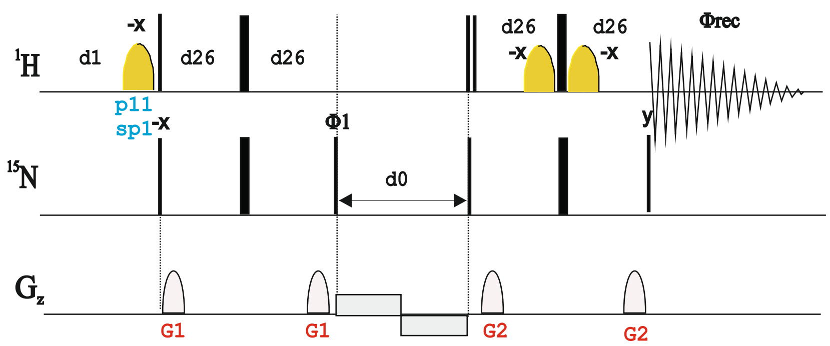
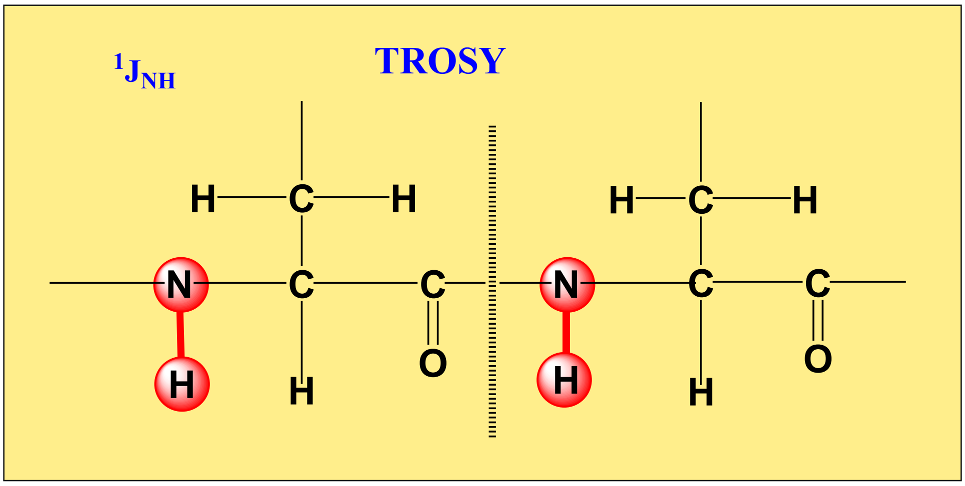
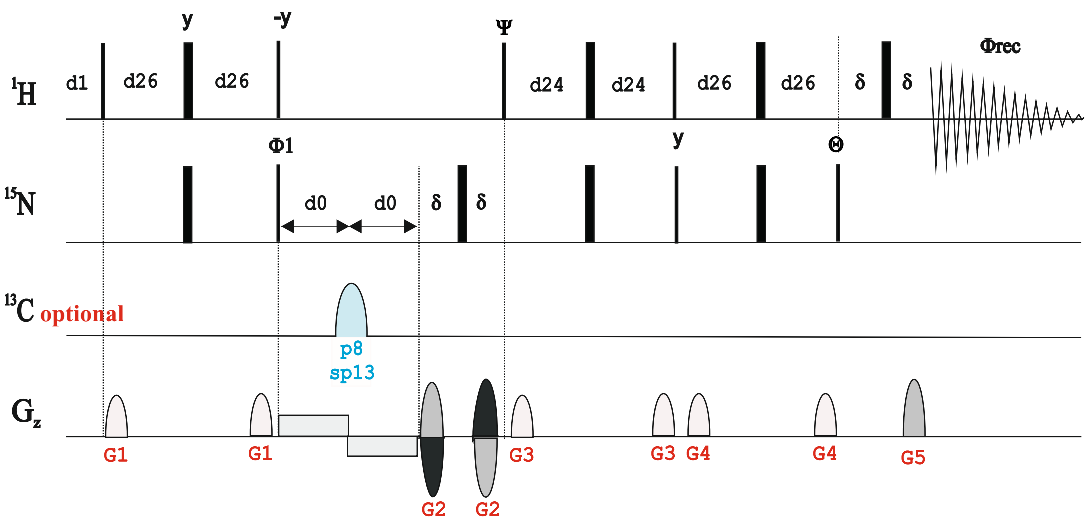


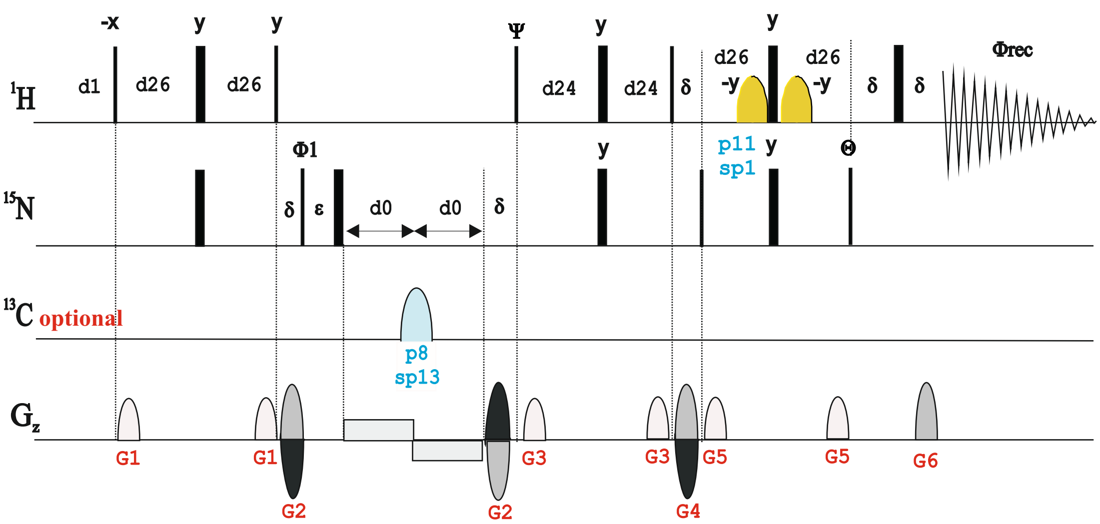
;K.V. Pervushin, G. Wider, R. Riek & K. Wuethrich, PNAS, 96, 9607-9612 (1999)



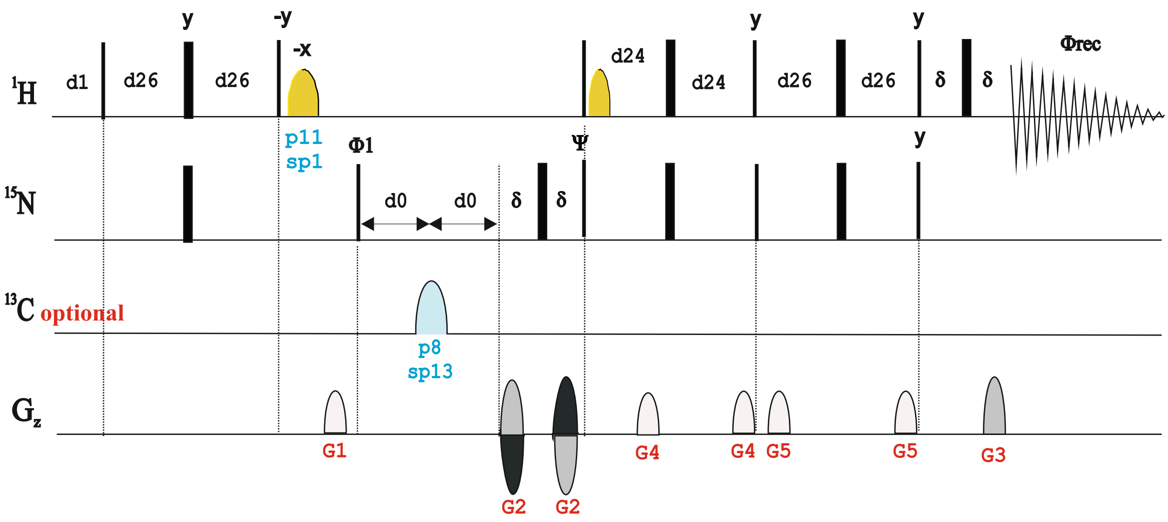
trosyetf3gpsi



trosyetf3gpsi.2



T. Schulte-Herbrueggen & O.W. Sorensen, J. Magn. Reson. 144, 123 - 128 (2000) trosyetf3gpsi2



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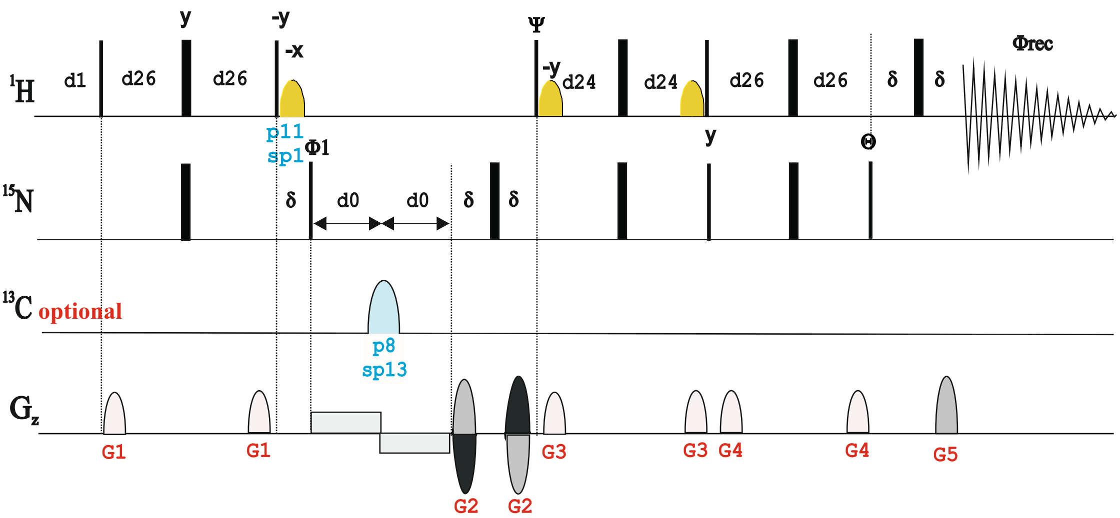

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2D CRINEPT EXPERIMENT

Experiment Description:

The CRINEPT (Cross-Correlated Relaxation Enhanced INEPT) experiment has been specifically designed to observe NH correlation peaks in very large 15N-labeled proteins.

References:

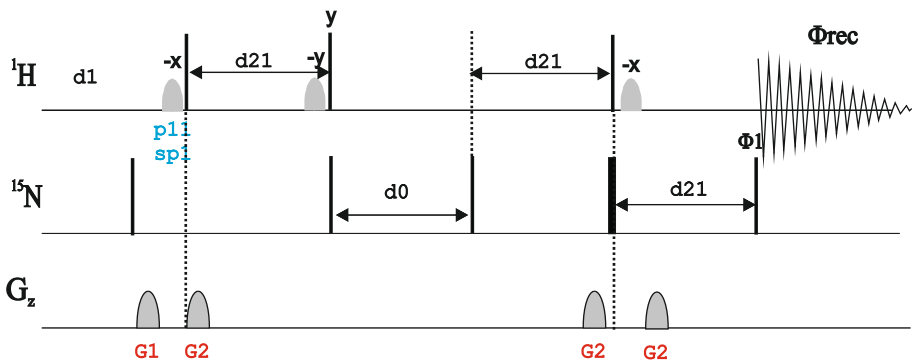
1. R. Riek, G. Wider, K. Pervushin & K. Wuethrich, Proc. Natl. Acad. Sci. USA 96, 4918-4923 (1999)

2D CRINEPT Experiments

ge-2D ^{1}H-^{15}N CRINEPT using flip-back (**crineptgpph**)

Also see: TROSY experiments

crineptgpph



use AU-program **splitcrinept** to process data

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2D IDIS-HSQC EXPERIMENT

IDIS-HSQC: Isotopically Discriminated HSQC experiment

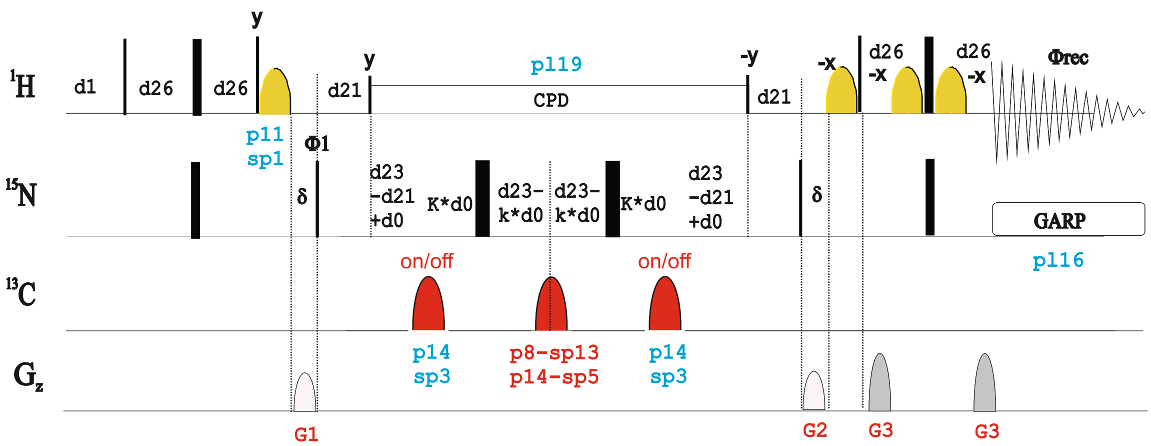
References:

1. A.P. Golovanov, R.T. Blankley, J.M. Avis & W. Bermel, J. Am. Chem. Soc. 129, 6528-6535 (2007)
2. W. Bermel, E.N. Tkach, A.G. Sobol & A.P. Golovanov, J. Am. Chem. Soc. 131, 8564-8570 (2009)

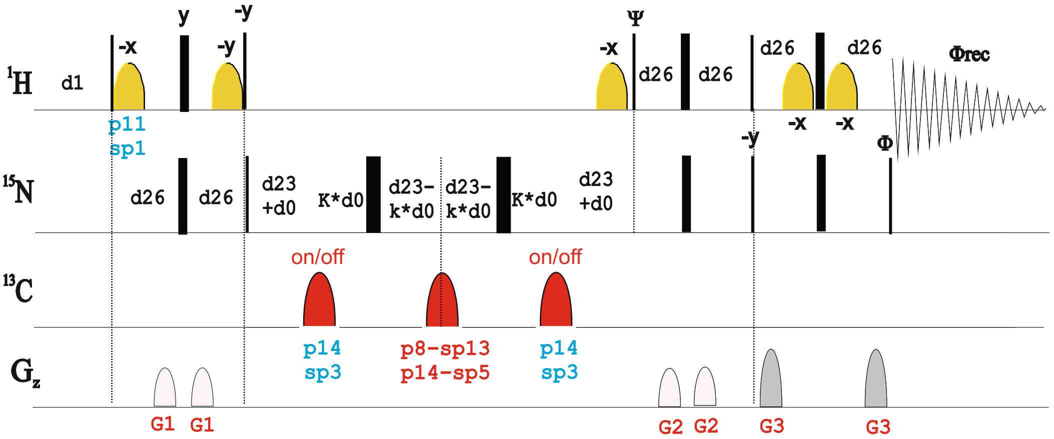
2D IDIS-HSQC Experiments

- Phase-sensitive 2D ^1H-^{15}N IDIS-HSQC for isotopical discrimination between $^{15}N(^{13}CO)$ and $15N(^{12}CO)$ using WATERGATE (**hsqcf3gpidphwg**)
- Phase-sensitive 2D ^1H-^{15}N IDIS-TROSY for isotopical discrimination between $^{15}N(^{13}CO)$ and $15N(^{12}CO)$ using WATERGATE (**trosyf3gpidphwg**)
- Phase-sensitive 2D ^1H-^{15}N IDIS-TROSY-IPAP for isotopical discrimination between $^{15}N(^{13}CO)$ and $15N(^{12}CO)$ using WATERGATE (**trosyf3gpiaidphwg**)

hsqcf3gpidphwg



trosyf3gpidphwg



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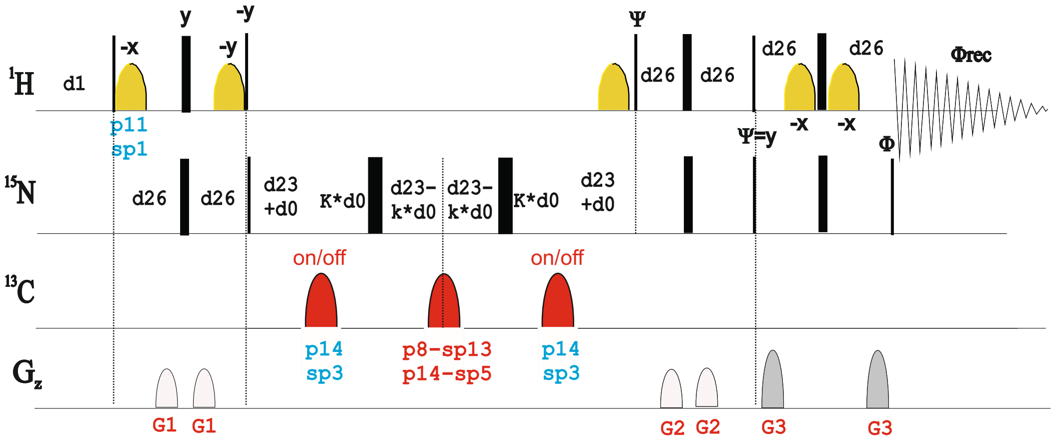

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2D HMQC-COSY EXPERIMENTS

2D HMQC-COSY Experiments

- **Phase cycled:**

  Magnitude-mode 2D HMQC-COSY using BIRD (**hmqcbindqfrl**)
  Phase-sensitive 2D HMQC-COSY using BIRD with decoupling (**hmqcbiphrl**)
  Phase-sensitive 2D HMQC-COSY using BIRD without decoupling (**hmqcbindphrl**)
  Phase-sensitive 2D HMQC-COSY-DQF using BIRD (**hmqcbidfphrl**)

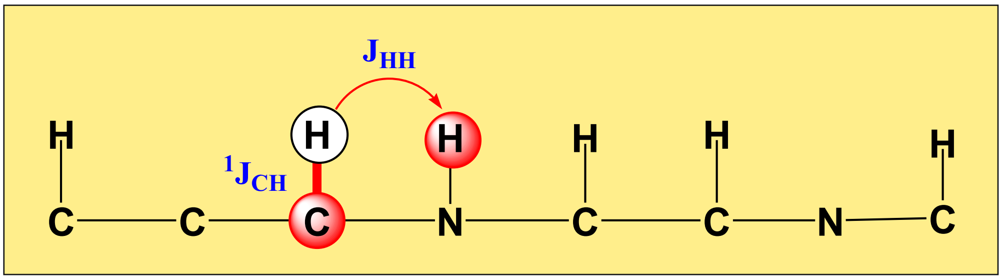
- **Gradient-enhanced:**

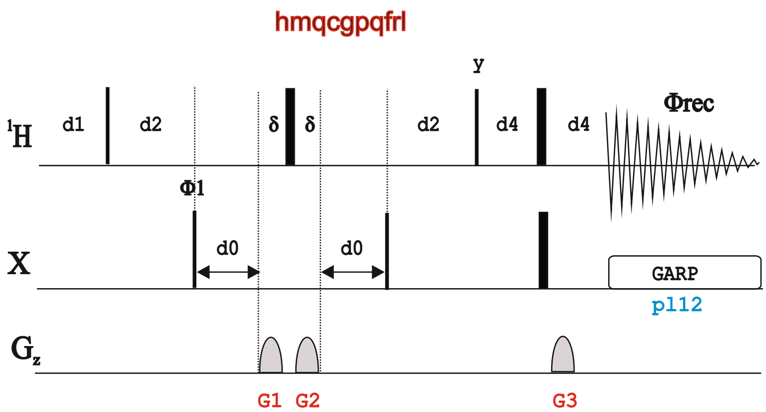
  Magnitude-mode ge-2D HMQC-COSY (**hmqcgpqfrl**)
  H2BC experiment with a three-low-pass filter (**h2bcetgpl3**)

- **H2BC experiment:**

  H2BC experiment with a three-low-pass filter (**h2bcetgpl3**)
  Multiplicity-edited H2BC experiment with a three-low-pass filter (**h2bcedetgpl3**)
  HAT-HMBC (H2BC + edited HMBC) experiment with a three-low-pass filter (**hathmbcetgpl3**)

Also see HMQC and HMQC-TOCSY experiments

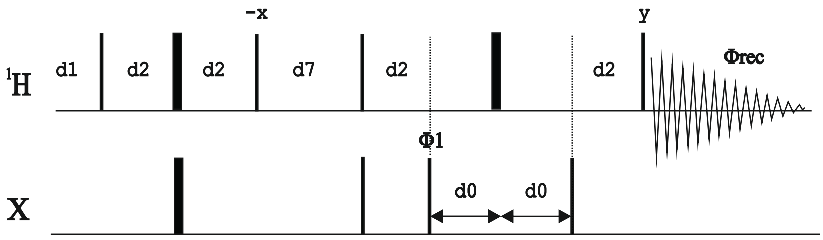




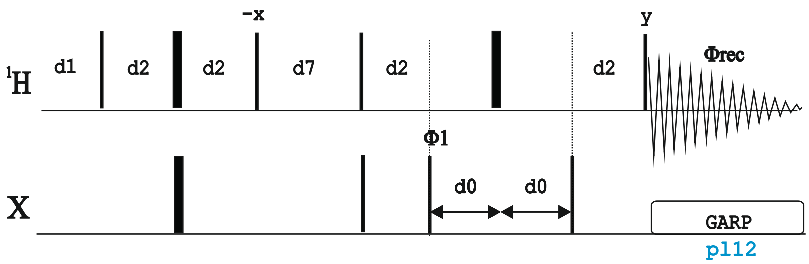
1. L. Lerner & A. Bax, J. Magn. Reson. 69, 375-380 (1986)
2. W. Willker, D. Leibfritz, R. Kerssebaum & W. Bermel, Magn. Reson. Chem. 31, 287-292 (1993)

hmqcbindqfrl

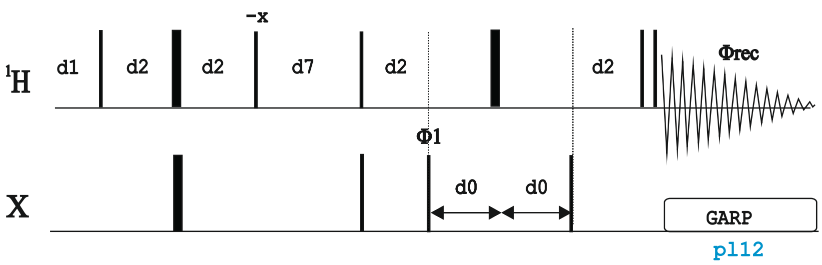
hmqcbindphrl



hmqcbiphrl



hmqcbidfphrl



H2BC:

1. N.T. Nyberg, J.O. Duus & O.W. Soerensen, J. Am. Chem. Soc. 127, 6154-6155 (2005)

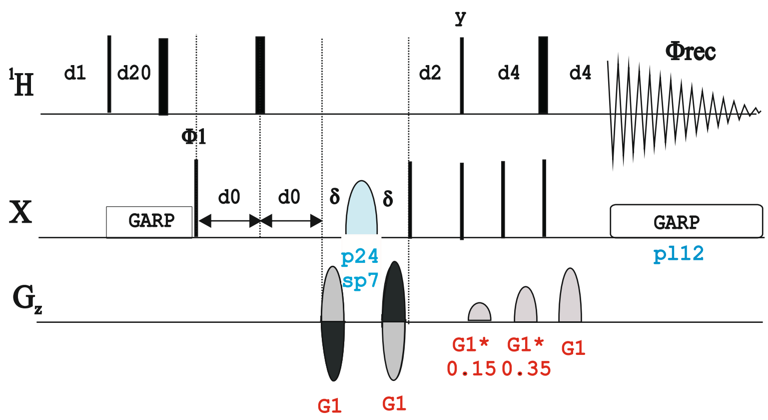
Edited-H2BC:

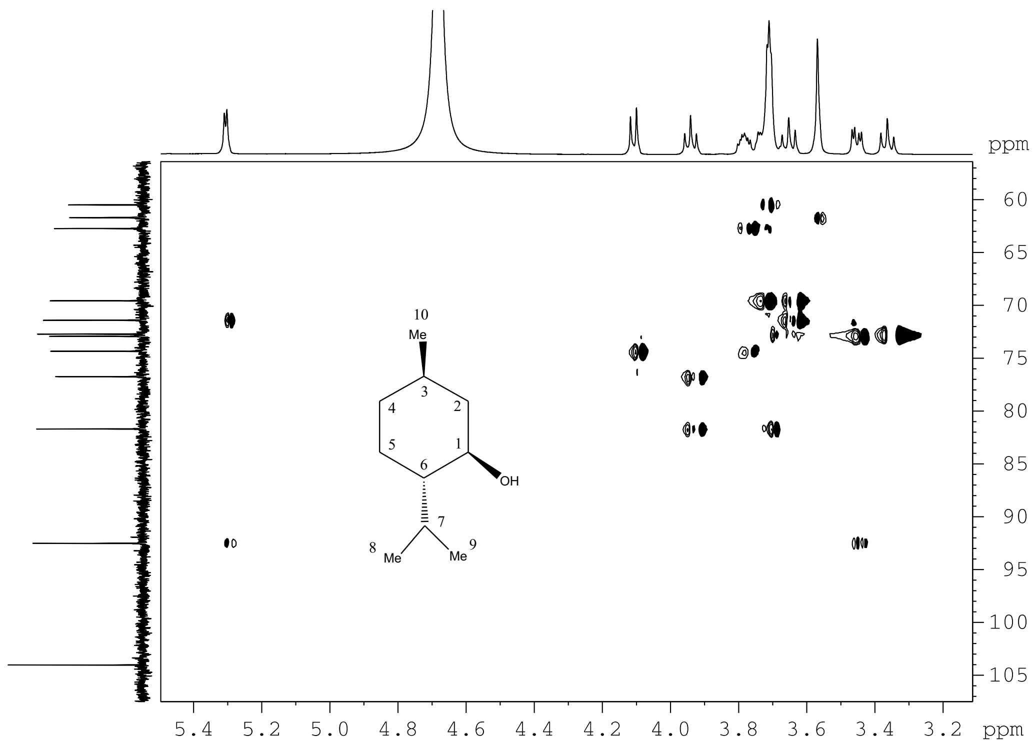
2. N.T. Nyberg, J.O. Duus & O.W. Soerensen, Magn. Reson. Chem. 43, 971-974 (2005)

HAT-HMBC:

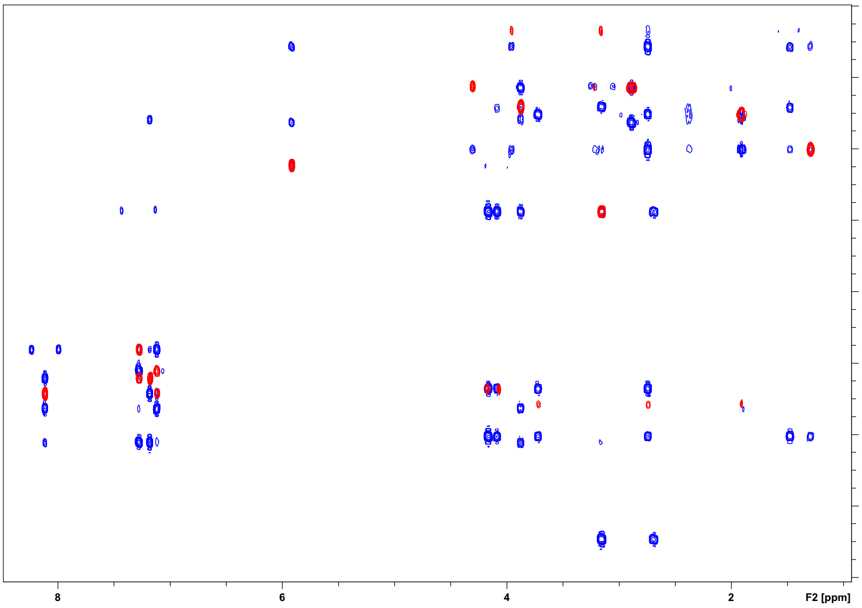
3. A.J. Benie & O.W. Soerensen, J. Magn. Reson. 184, 315-321 (2007)

h2bcetgpl3

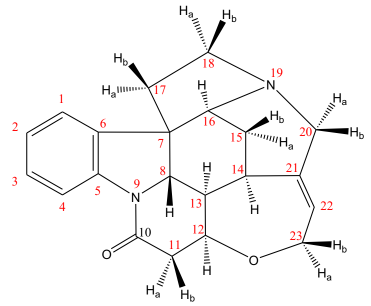




H2BC:
Two-bond correlations but no signals from quaternary carbons



Comparison between HMBC(blue) and H2BC (red) allows differentiation2-bond vs 3-bond connectivities



h2bcedetgpl3

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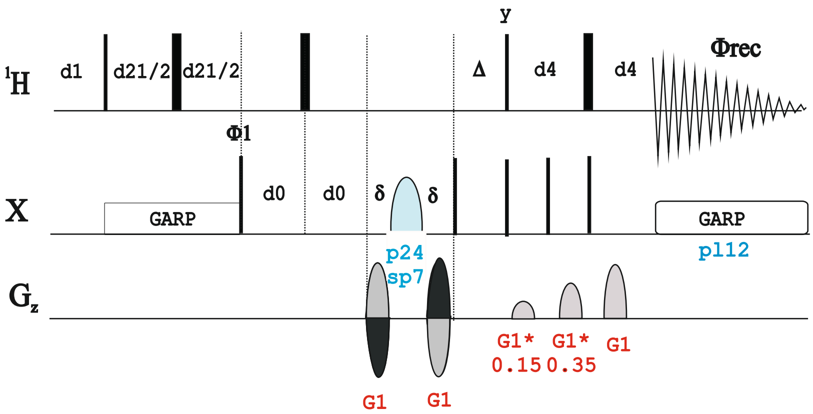
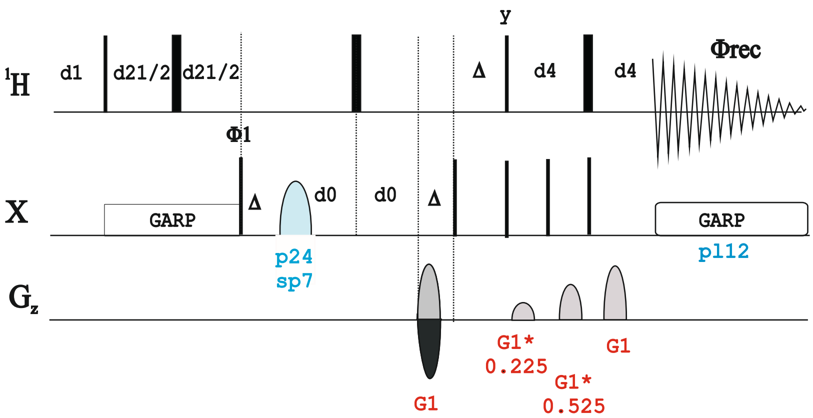


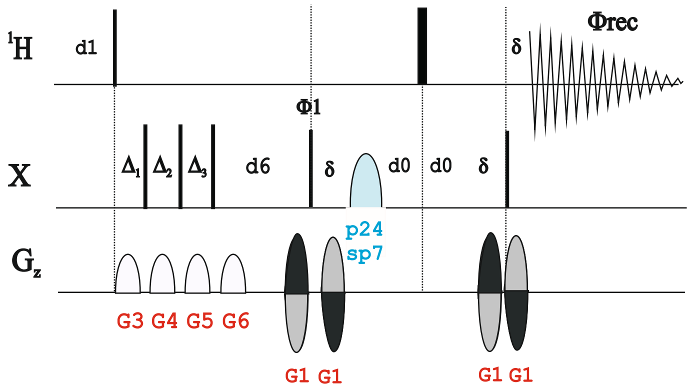
DATA B



Processing:
use AU-program **split [ipap 2]** to create separate datasets

hathmbcetgpl3

DATA A



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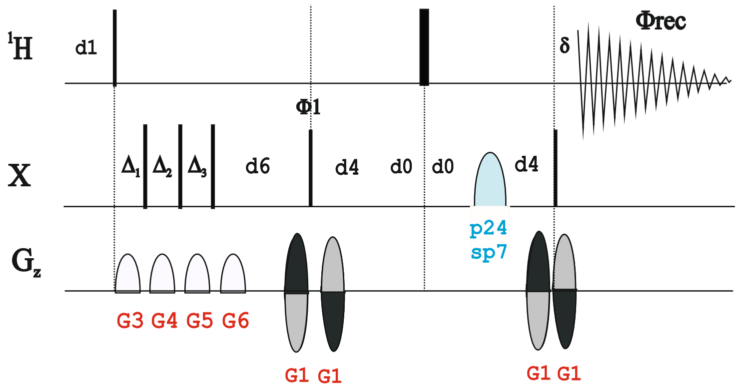

Processing:
use AU-program **split [ipap 2]** to create separate datasets

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2D HMQC-TOCSY EXPERIMENTS

Experiment Description:

The 2D HMQC-TOCSY experiment is an hybrid experiment consisting of a first refocused HMQC pulse train (optimized to d2=1/2J(XH)) followed by a mixing TOCSY process defined by d9. Other experimental set-up as usually made in HMQC experiments.
Two type of peaks are obtained in a conventional 2D correlation map : i) Direct X-H correlations and ii) Relayed correlations connecting each protonated X nucleus with all 1H belonging to the same spin system.

See 2D TOCSY and 2D HSQC-TOCSY for related experiments.

2D HMQC-TOCSY Experiments

- **Phase-cycled:**

  Phase-sensitive 2D HMQC-TOCSY (**hmqcmlph**)
  Phase-sensitive 2D HMQC-TOCSY without decoupling (**hmqcmlndph**)
  Magnitude-mode 2D HMQC-TOCSY using BIRD (**hmqcbimlqf**)
  Magnitude-mode 2D HMQC-TOCSY using BIRD without decoupling (**hmqcbimlndqf**)

  Phase-sensitive 2D HMQC-TOCSY using BIRD (**hmqcbimlph**)
  Phase-sensitive 2D HMQC-TOCSY using BIRD without decoupling (**hmqcbimlndph**)

- **Phase-cycled and solvent suppression:**

  Phase-sensitive 2D HMQC-TOCSY with presaturation (**hmqcmlphpr**)
  Phase-sensitive2D HMQC-TOCSY with presaturation and without decoupling (**hmqcmlndphpr**)

- **Gradient-enhanced from f2 channel:**

  Magnitude-mode ge-2D HMQC-TOCSY with MLEV (**hmqcgpmlqf** | HMQCGPML)
  Phase-sensitive ge-2D HMQC-TOCSY with DIPSI-2 using echo-antiecho (**hmqcdietgp**)
  Phase-sensitive ge-2D HMQC-TOCSY with DIPSI-2 using PEP (**hmqcdietgpsi**)
  Phase-sensitive ge-2D HMQC-TOCSY with DIPSI-2 using PEP using shorter overall timing (**hmqcdietgpsi.2**)

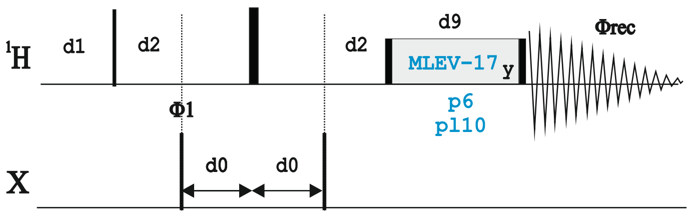
- **Gradient-enhanced from f3 channel:**

  Phase sensitive ge-2D ^{1}H-^{15}N HMQC-TOCSY with DIPSI-2 using echo-antiecho (**hmqcdietf3gp**)
  Phase sensitive ge-2D ^{1}H-^{15}N HMQC-TOCSY with DIPSI-2 using PEP (**hmqcdietf3gpsi**)
  Phase sensitive ge-2D ^{1}H-^{15}N HMQC-TOCSY with DIPSI-2 using PEP and shorther overall timing(**hmqcdietf3gpsi.2**)

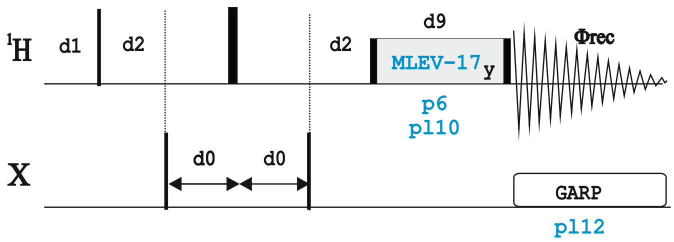
Also see in 3D HMQC-TOCSY experiments
3D ^{1}H-^{13}C HMQC-TOCSY experiment using BIRD (hmqcmlevbi3d)

Also see:
2D HMQC and 2D TOCSY experiments
2D HSQC-TOCSY experiments

hmqcmlndph



hmqcmlph



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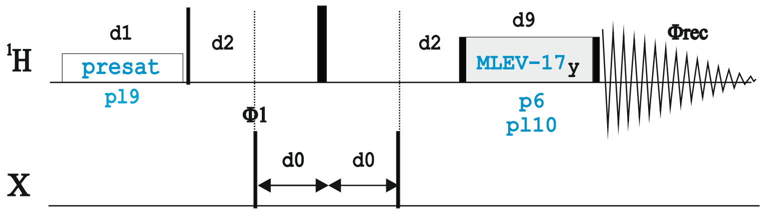
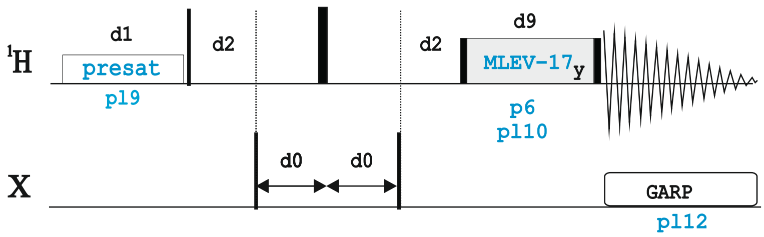
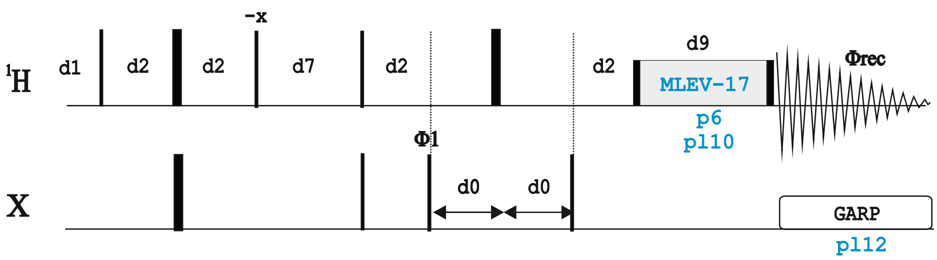


hmqcmlphpr



hmqcbimlqf
hmqcbimlph



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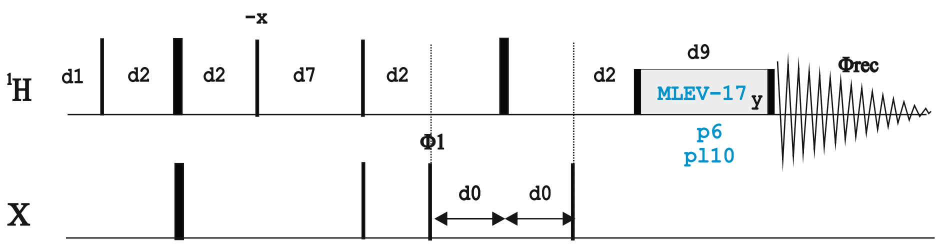
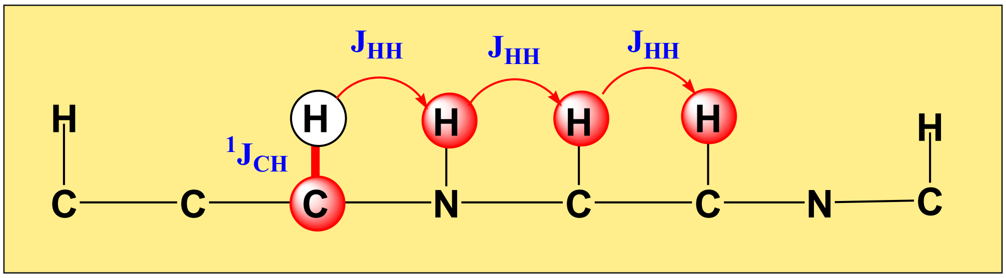
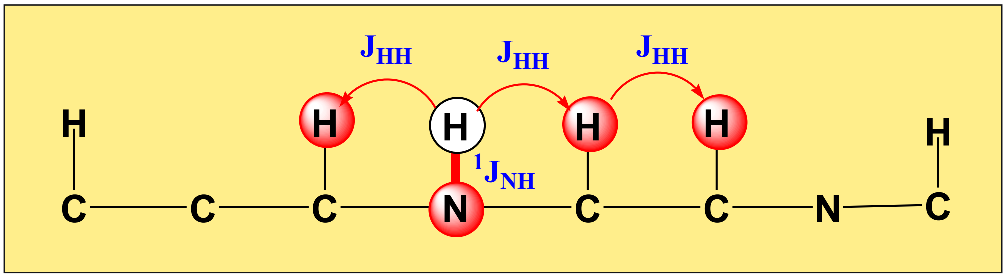
hmqcbimlndph

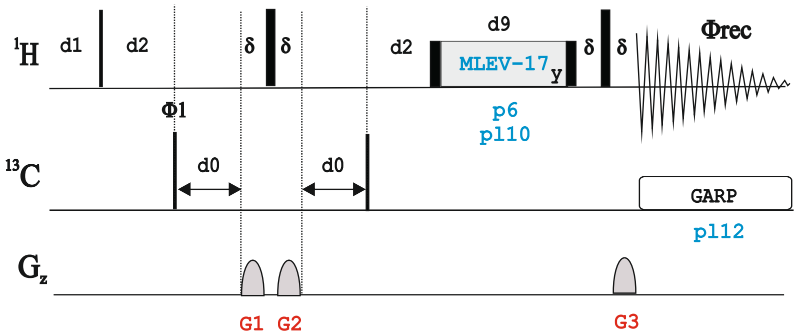


The delay d9 defines the TOCSY mixing period, independently if DIPSI-2 or MLEV are used. Typical values range from 0 to 120 ms and transfer efficiency depends on spin system topologies.



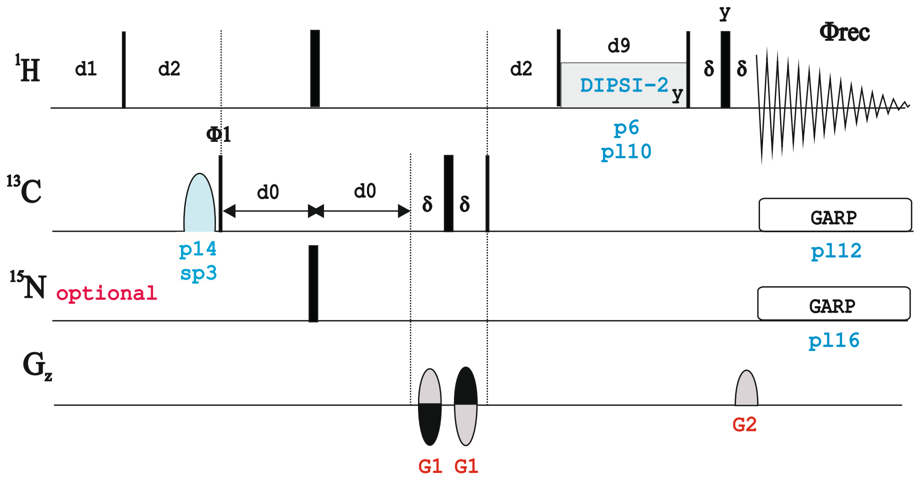


hmqcgpmlqf

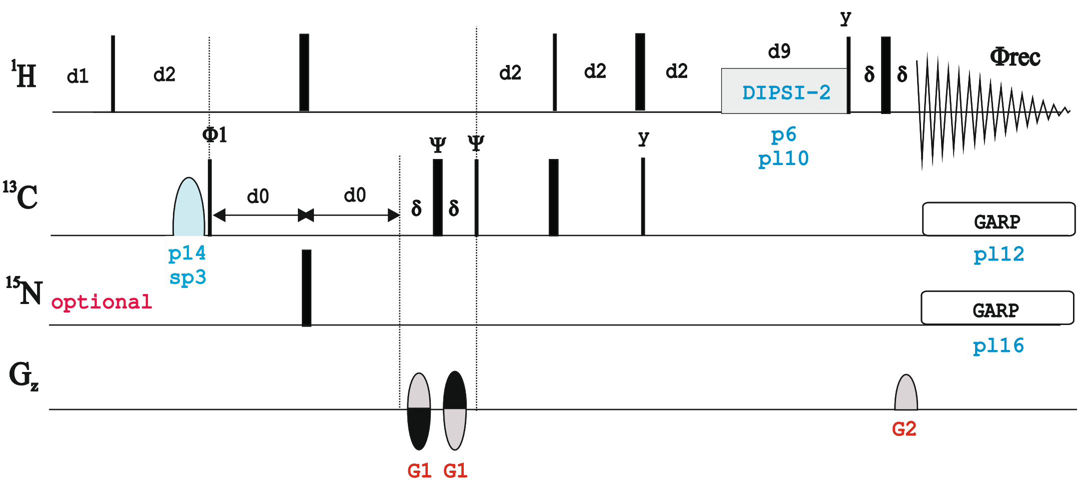


1. L. Lerner & A.Bax, J. Magn. Reson. 69, 375-380 (1986
2. A.G. Palmer III, J. Cavanagh, P.E. Wright & M. Rance, J. Magn. Reson. 93, 151-170 (1991)

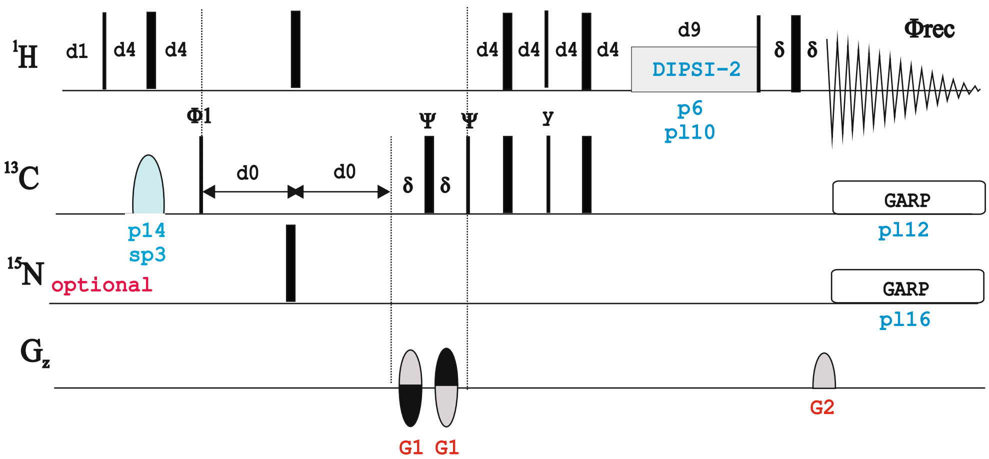
hmqcdietgp



hmqcdietgpsi



hmqcdietgpsi.2



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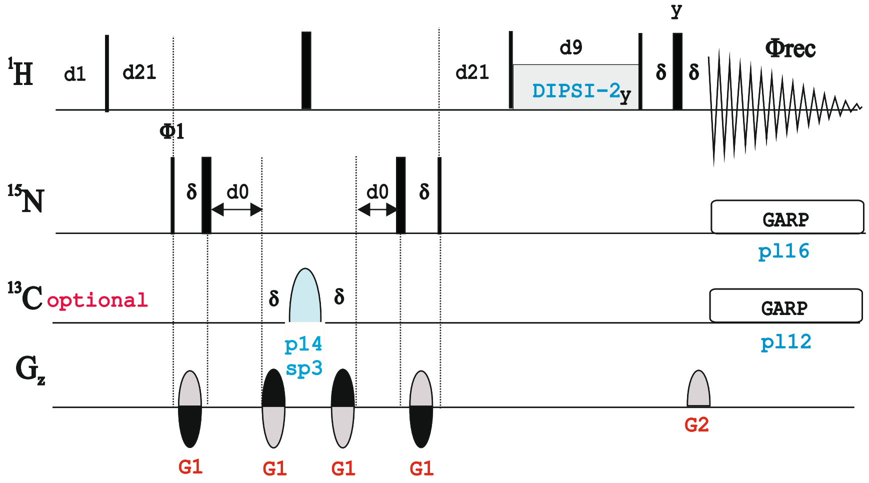

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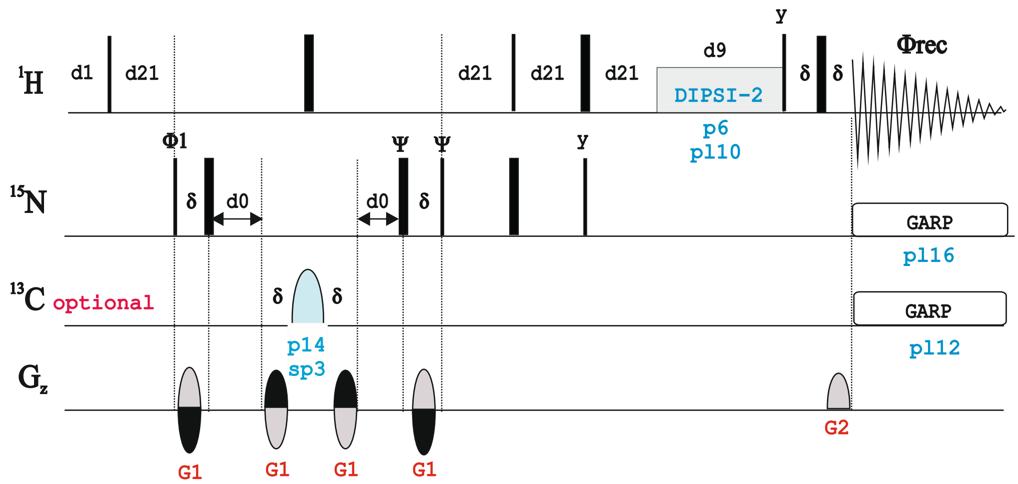
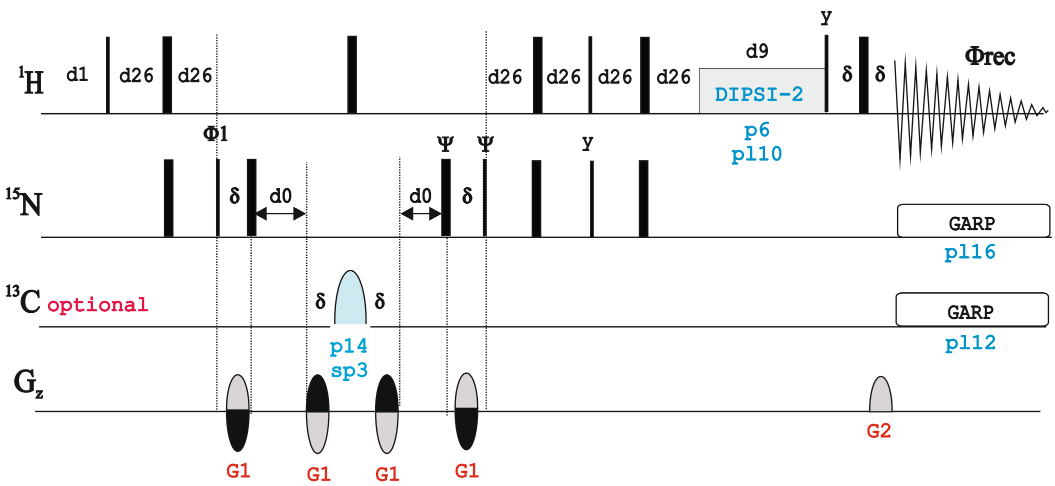


hmqcdietf3gpsi.2



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2D HMQC-ROESY EXPERIMENTS

Experiment Description:

The 2D HMQC-ROESY experiment is an hybrid experiment consisting of a first refocused HMQC pulse train (optimized to d2=1/2J(XH)) followed by a mixing ROESY process defined by p15. Other experimental set-up as usually made in HMQC experiments.
Two type of peaks are obtained in a conventional 2D correlation map : i) Direct X-H correlations and ii) ROE correlations connecting a protonated X nucleus with a 1H resonance which is close to the directly-attached 1H-X proton.

The experiment can be recorded without X-decoupling during 1H acquisition (pl12 or pl16 set to 120dB). This is useful to observe NOE between degenerate protons (for instance, symmetrical molecules).

See 2D ROESY and 2D HSQC-ROESY for related experiments.

References:

A. Bax & D.G. Davis, J. Magn. Reson 63, 207-213 (1985).

2D HMQC-ROESY Experiments

- **Gradient-enhanced from the f2 channel**

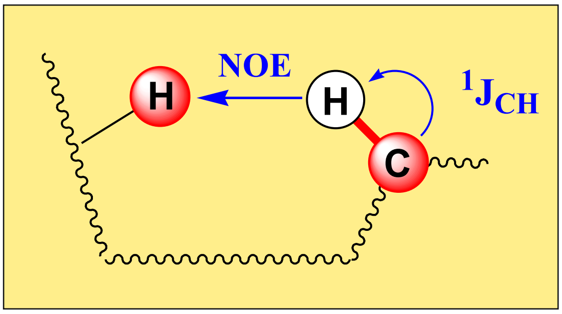
  Phase-sensitive ge-2D HMQC-ROESY using echo-antiecho (**hmqcetgpro**)
  Phase-sensitive ge-2D HMQC-ROESY with T-ROESY using echo-antiecho (**hmqcetgpro.2**)

- **Gradient-enhanced from the f3 channel**

  Phase-sensitive ge-2D ^{1}H-^{15}N HMQC-ROESY using echo-antiecho (**hmqcetf3gpro**)
  Phase-sensitive ge-2D ^{1}H-^{15}N HMQC-ROESY with T-ROESY using echo-antiecho (**hmqcetf3gpro.2**)

Also see:
2D HMQC and 2D ROESY Experiments
2D HMQC-NOESY experiments

The pulse p15 (in microseconds), applied at power level pl11, defines the mixing period in all ROESY experiments.



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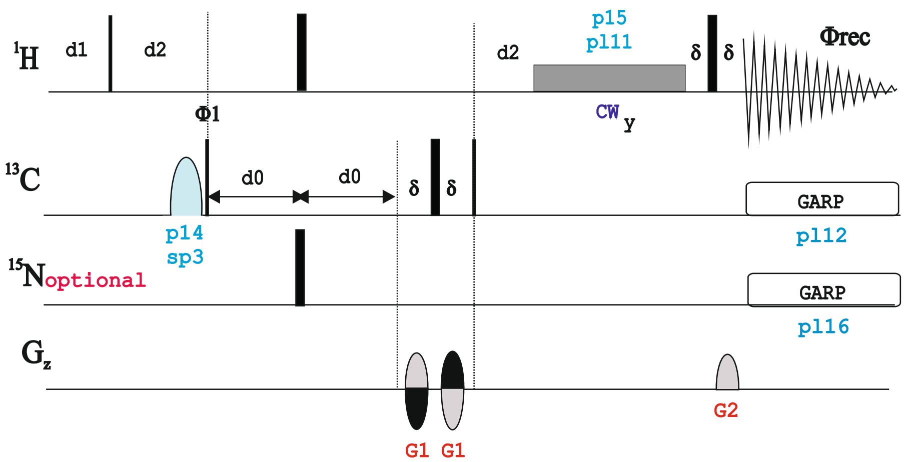

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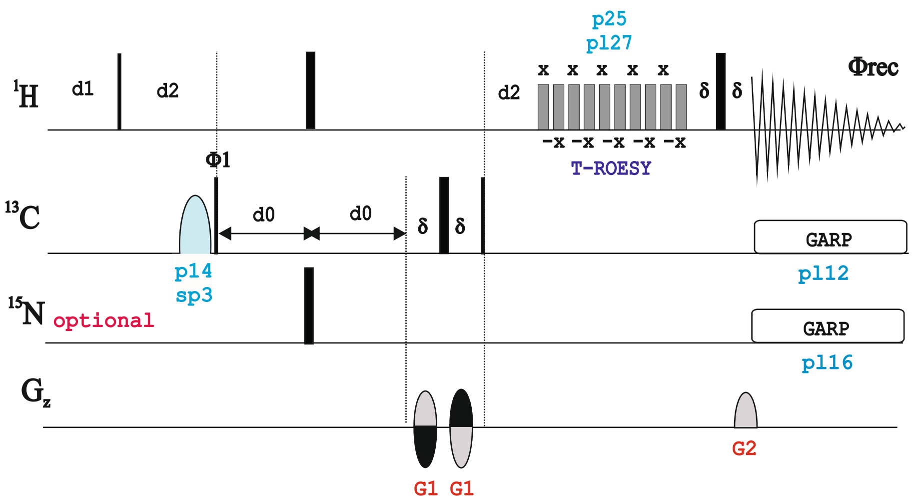

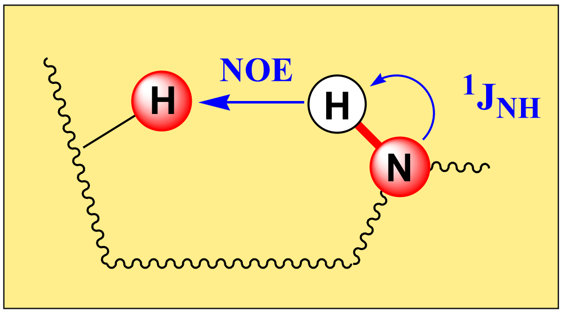

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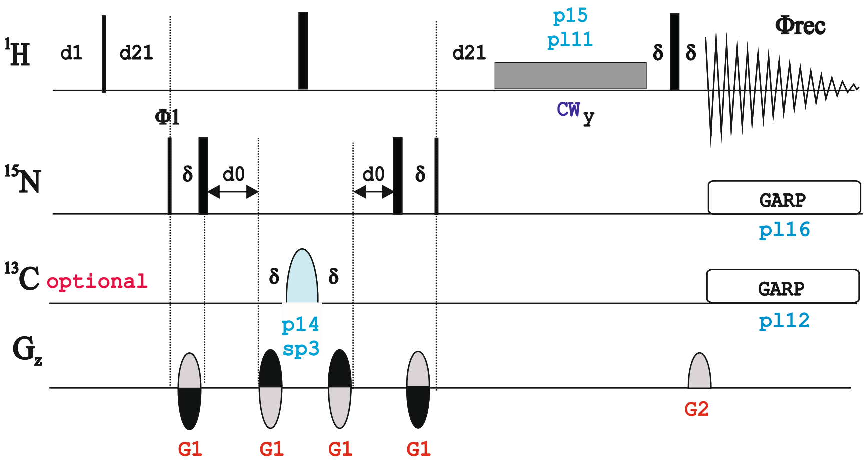

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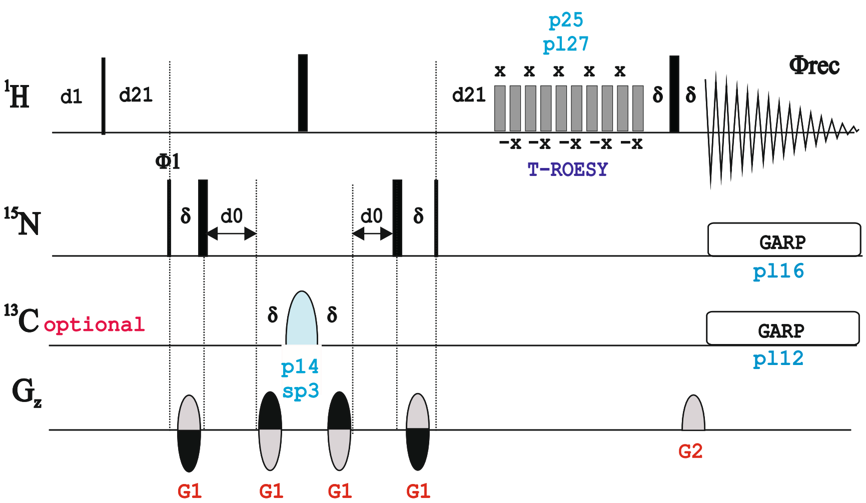

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2D HMQC-NOESY EXPERIMENTS

Experiment Description:

The 2D HMQC-NOESY experiment is an hybrid experiment consisting of a first refocused HMQC pulse train (optimized to d2=1/2J(XH)) followed by a mixing NOESY building block defined by d8. Other experimental set-up as usually made in HMQC experiments.
Two type of peaks are obtained in a conventional 2D correlation map : i) Direct X-H correlations and ii) ROE correlations connecting a protonated X nucleus with a 1H resonance which is close to the directly-attached 1H-X proton.

The experiment can be recorded without X-decoupling during 1H acquisition (pl12 or pl16 set to 120dB). This is useful to observe NOE between degenerate protons (for instance, symmetrical molecules).

See 2D NOESY, 2D HOESY and 2D HSQC-NOESY for related experiments.

2D HMQC-NOESY Experiments

- **Phase cycled:**

  Phase-sensitive 2D HMQC-NOESY with presaturation (**hmqcnophpr**)
  Phase-sensitive 2D HMQC-NOESY using BIRD (**hmqcbinoph**)

- **Gradient-enhanced from the f2 channel:**

  Phase-sensitive ge-2D HMQC-NOESY using echo-antiecho (**hmqcetgpno**)

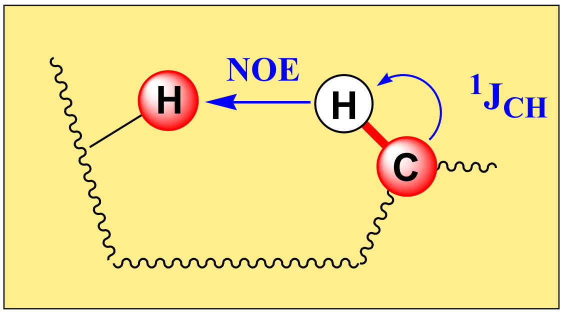
- **Gradient-enhanced from the f3 channel:**

  Phase-sensitive ge-2D ^{1}H-^{15}N HMQC-NOESY using echo-antiecho (**hmqcetf3gpno**)

Also see in “3D HMQC-NOESY experiments”:
3D ^{1}H-^{13}C HMQC-NOESY experiment using BIRD (hmqcnoesybi3d)

Also see:
2D HMQC and 2D NOESY Experiments
2D HMQC-ROESY experiments

The delay d8 defines the mixing period in all NOESY experiments,



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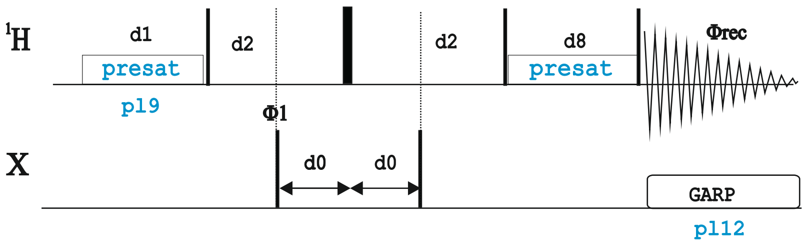

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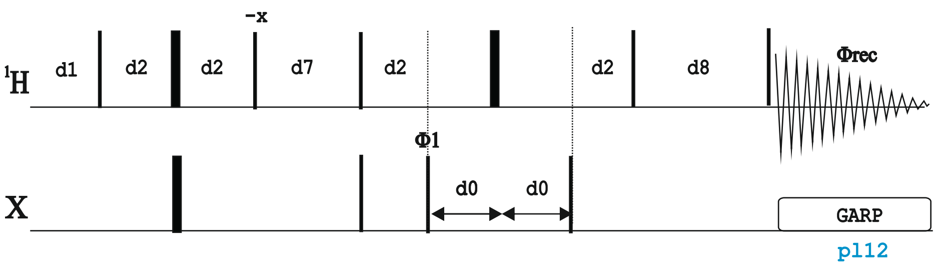
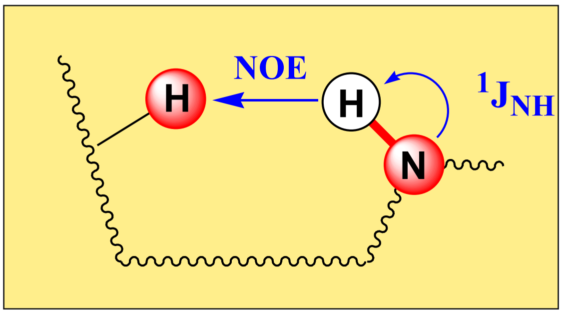
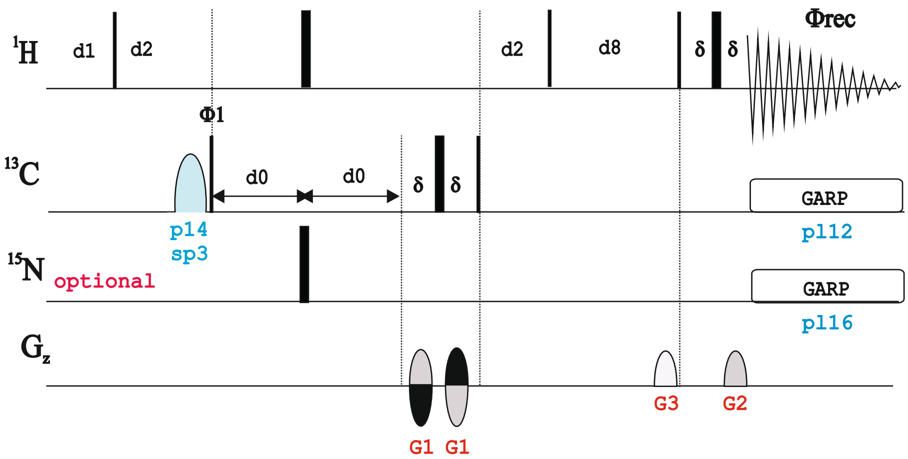




hmqcetgpno



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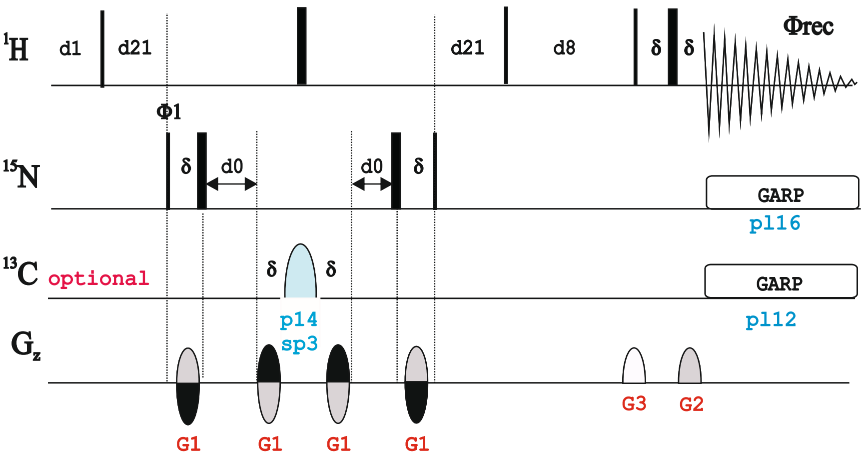

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2D HSQC-TOCSY EXPERIMENTS

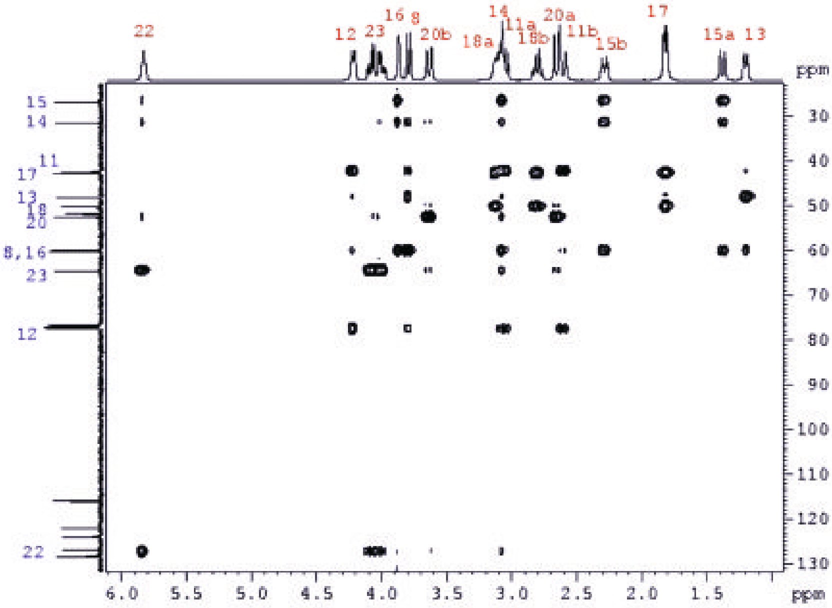
Experiment Description:

The 2D HSQC-TOCSY experiment is an hybrid experiment consisting of a first refocused HSQC pulse train (optimized to d4=1/4J(XH)) followed by a mixing TOCSY process defined by d9. Other experimental set-up as usually made in regular HSQC experiments.
Two type of peaks are obtained in a conventional 2D correlation map : i) Direct X-H correlations and ii) Relayed correlations connecting each protonated X nucleus with all 1H belonging to the same spin system.

Versions of the basic experiment include:

i) Use of MLEV or DIPSI as a TOCSY mixing process.
ii) Use of gradients for coherence selection. OPtional combination with sensitivity-improved building block (PEP).
iii) Use of adiabatic 13C 180 pulses.
iv) Use of editing of direct responses in order to differentiate direct and relayed correlations .
v) Use of multiplicity editing to know the multiplicity of the carbon resonance.
vi) Optional use of the f2 and f3 channel version, for triple resonance probeheads.

Also see 2D TOCSY, 2D HMQC-TOCSY and 3D HSQC-TOCSY for related experiments.



2D 1H-13C HSQC-TOCSY spectrum of strychnine

2D HSQC-TOCSY Experiments

- **Gradient-enhanced from the f2 channel**

  Phase sensitive ge-2D HSQC-TOCSY with MLEV using z-filter (**hsqcgpmlph** | HSQCGPMLPH)
  Phase-sensitive ge-2D HSQC-TOCSY with MLEV using echo-antiecho (**hsqcetgpml** | HSQCETGPML)
  Phase-sensitive ge-2D HSQC-TOCSY with DIPSI-2 using PEP (**hsqcdietgpsi**)
  Phase-sensitive ge-2D HSQC-TOCSY with DIPSI-2 using PEP and adiabatic inversion pulses (**hsqcdietgpsisp** | HSQCDIETGPSISP)
  Phase-sensitive ge-2D HSQC-TOCSY with DIPSI-2 using PEP and adiabatic inversion and refocusing pulses (**hsqcdietgpsisp.2**)

- **Gradient-enhanced with editing from the f2 channel**

  Phase sensitive ge-2D HSQC-TOCSY using PEP with editing of multiplicity (**hsqcdiedetgpsisp.1**)
  Phase sensitive ge-2D HSQC-TOCSY using PEP with editing of direct responses (**hsqcdiedetgpsisp.2**)
  Phase sensitive ge-2D HSQC-TOCSY using PEP with editing of multiplicity and direct responses (**hsqcdiedetgpsisp.3**)

- **Gradient-enhanced from the f3 channel**

  Phase sensitive ge-2D ^{1}H-^{15}N HSQC-TOCSY with MLEV using echo-antiecho (**hsqcetf3gpml**)
  Phase sensitive ge-2D ^{1}H-^{15}N HSQC-TOCSY with DIPSI-2 using PEP (**hsqcdietf3gpsi** | HSQCDIETF3GPSI)

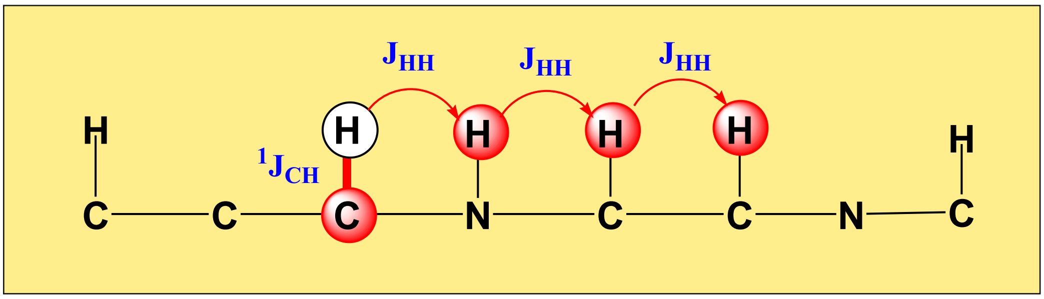
Also see:

HSQC-TOCSY type experiments (HETLOC, HECADE, spin-edited HSQC-TOCSY) for $^{n}J_{CH}$ measurements

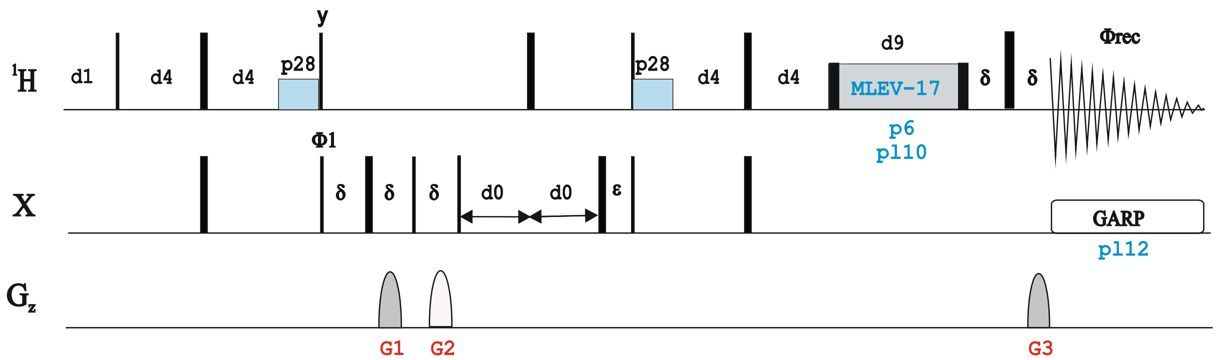
3D HSQC-TOCSY experiments:
Phase-sensitive ge-2D HSQC-TOCSY with MLEV using echo-antiecho (hsqcetgpml3d.2)
Phase-sensitive ge-2D HSQC-TOCSY with DIPSI-2 using PEP and adiabatic inversion and refocusing pulses (hsqcdietgpsisp3d.2)

Also see:
2D HSQC and 2D TOCSY experiments
2D HMQC-TOCSY Experiments

The delay d9 defines the TOCSY mixing period, independently if DIPSI-2 or MLEV are used. Typical values range from 0 to 120 ms and transfer efficiency depends on spin system topologies.



hsqcgpmlph



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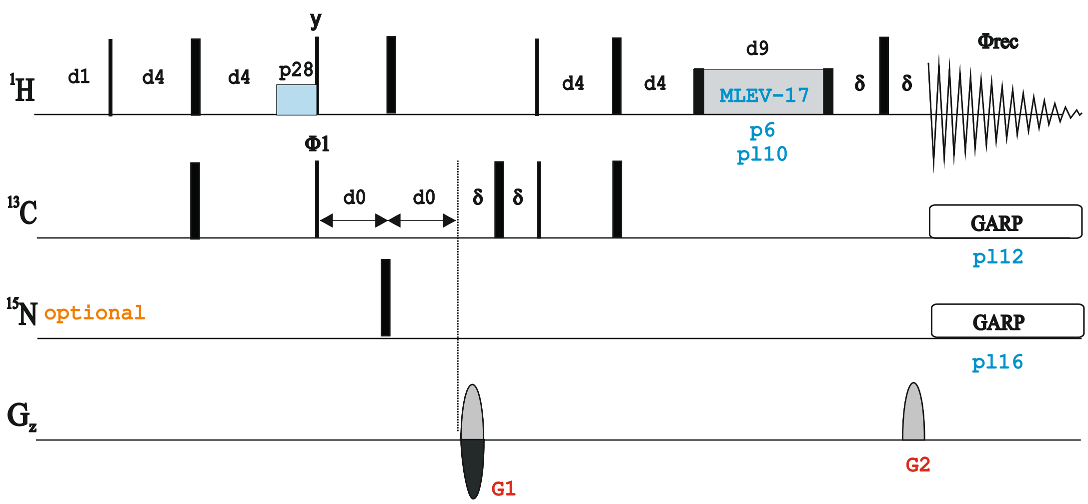
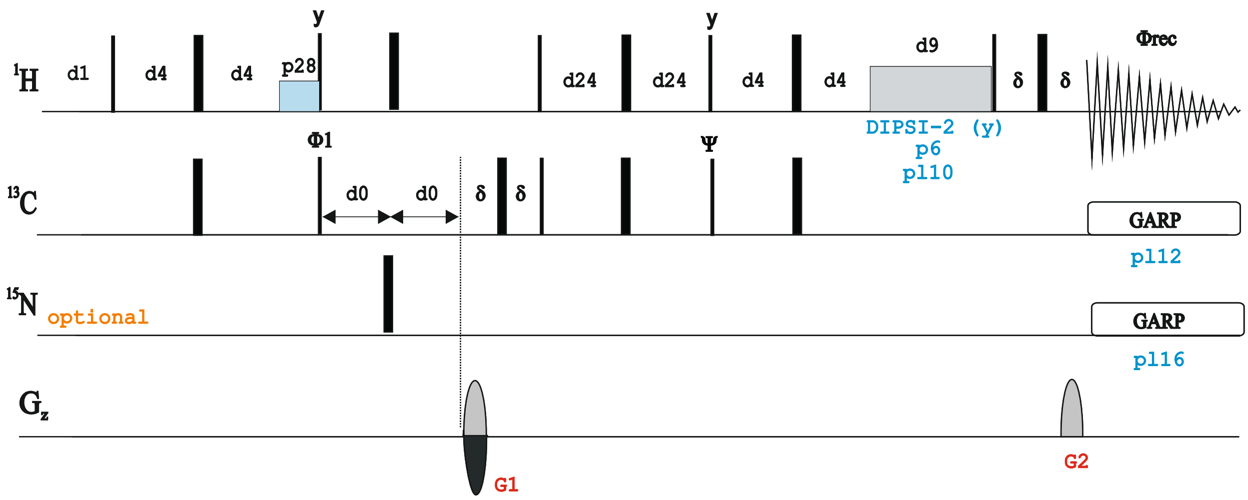
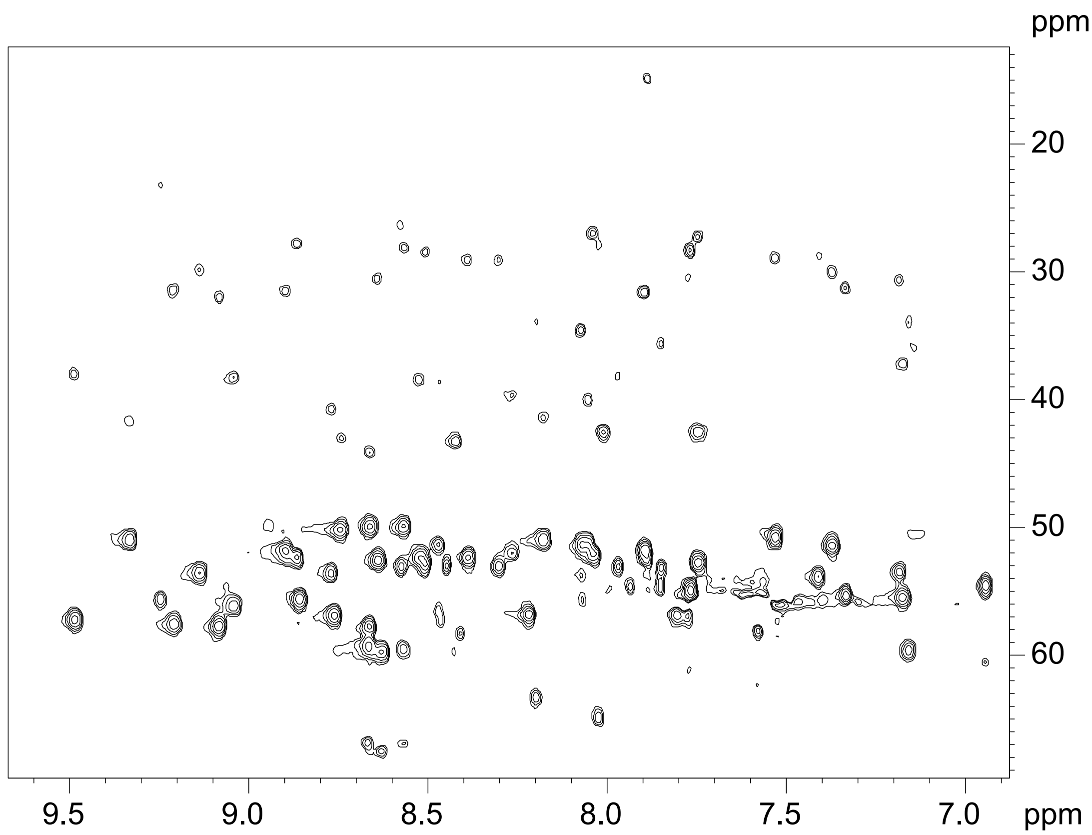


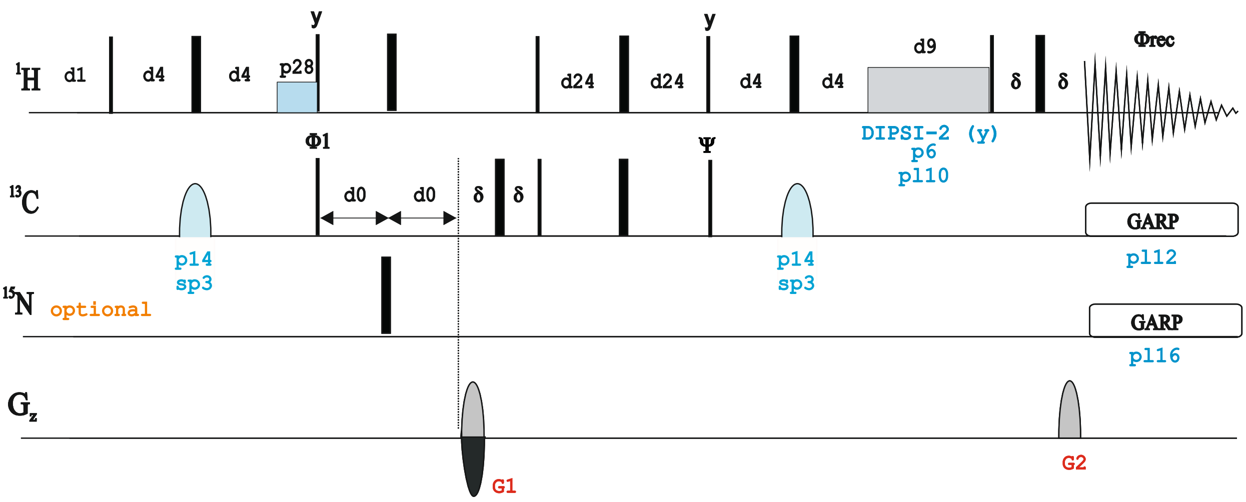
hsqcdietgpsi





Expansion region of the 2D 1H-13C HSQC-TOCSY spectrum of ubiquitine

hsqcdietgpsisp



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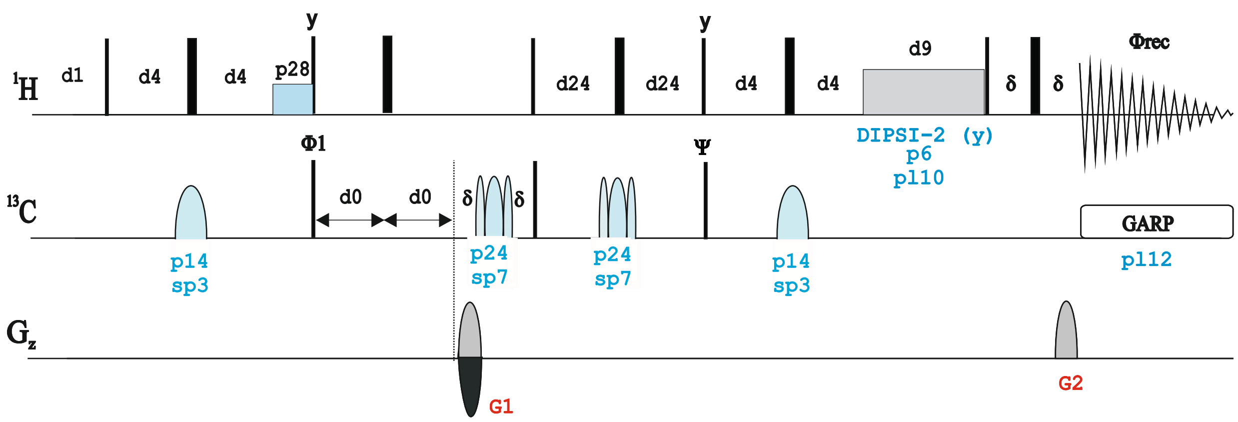

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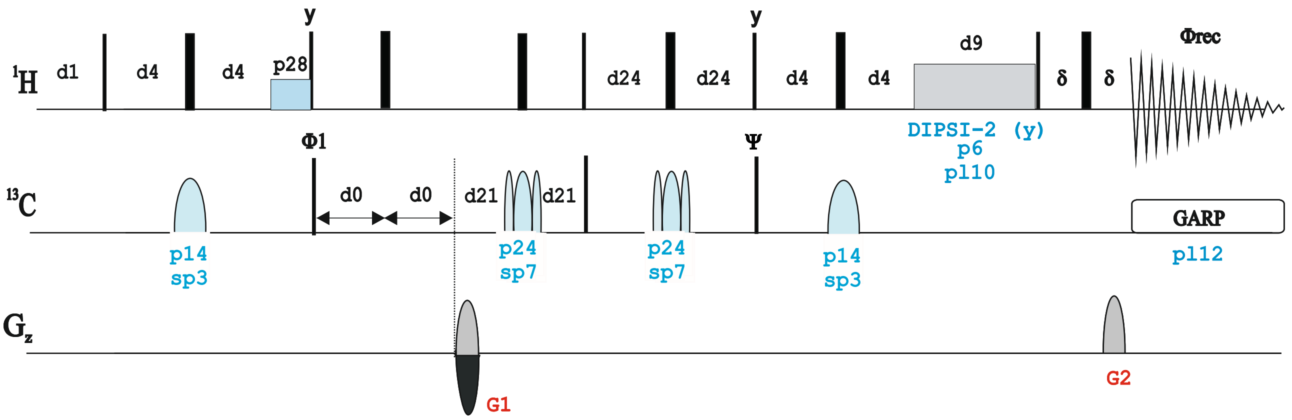

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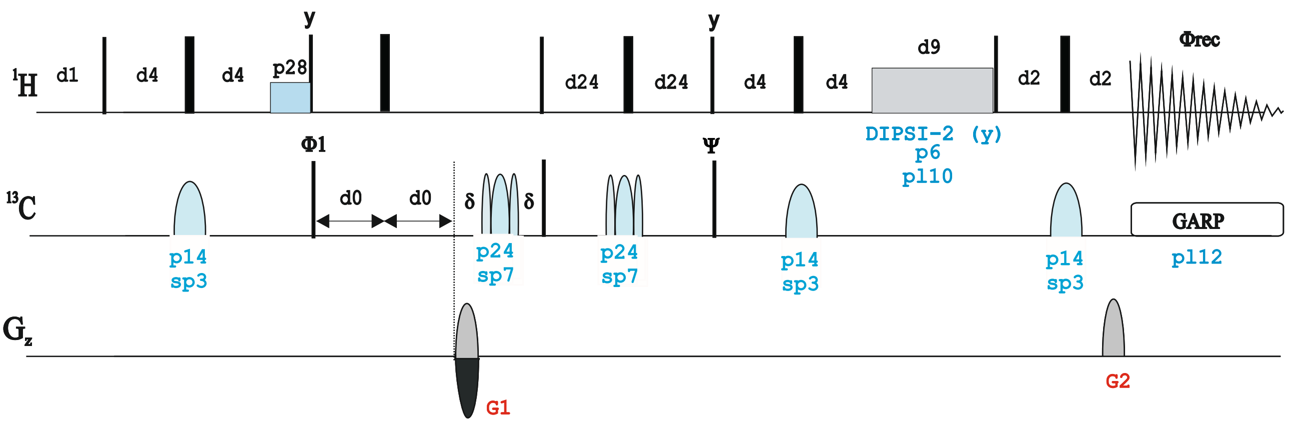
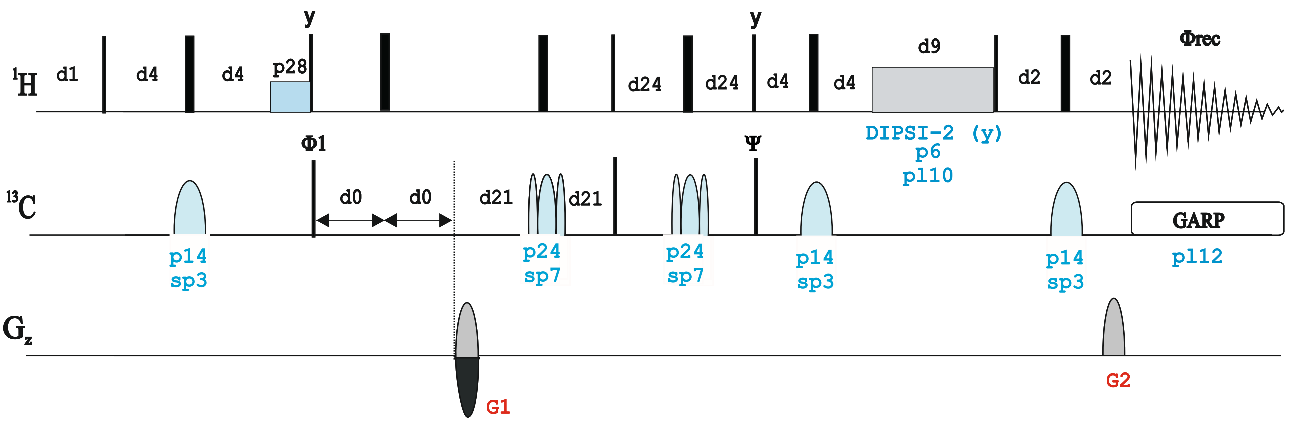
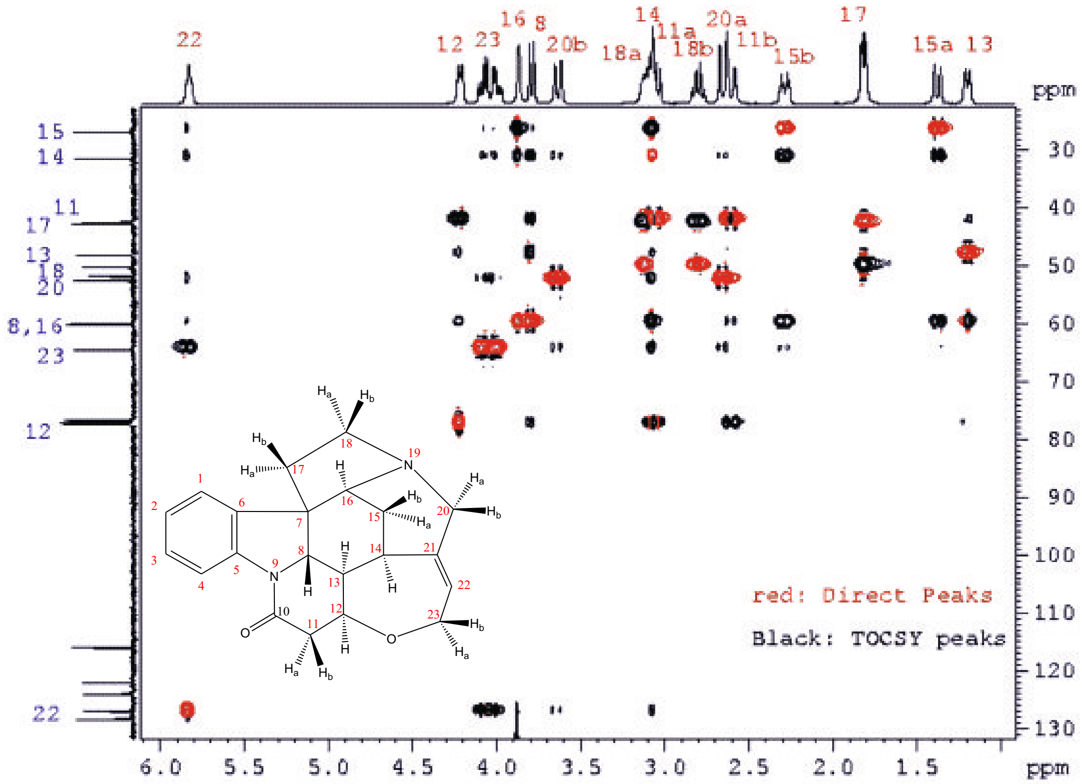
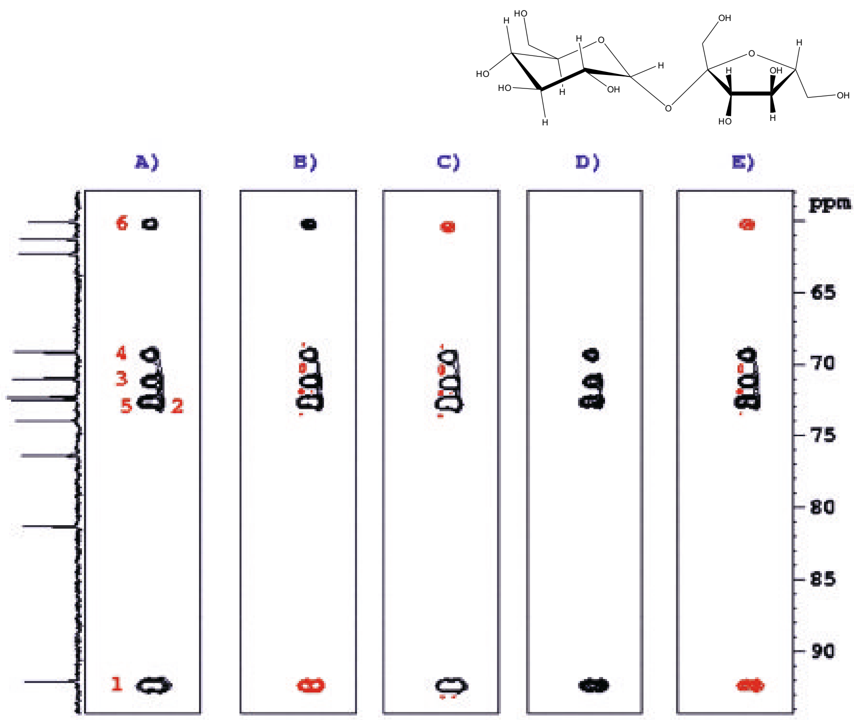


hsqcdiedetgpsisp.3

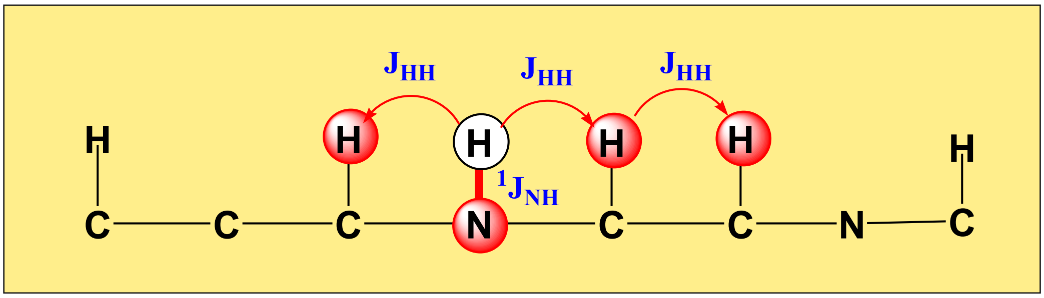




2D 1H-13C HSQC-TOCSY spectrum of strychnine with editing of direct responses



Column expansions of the 2D 1H-13C HSQC-TOCSY spectrum of sucrose with several combinations of editing of direct responses and/or editing of carbon multiplicity



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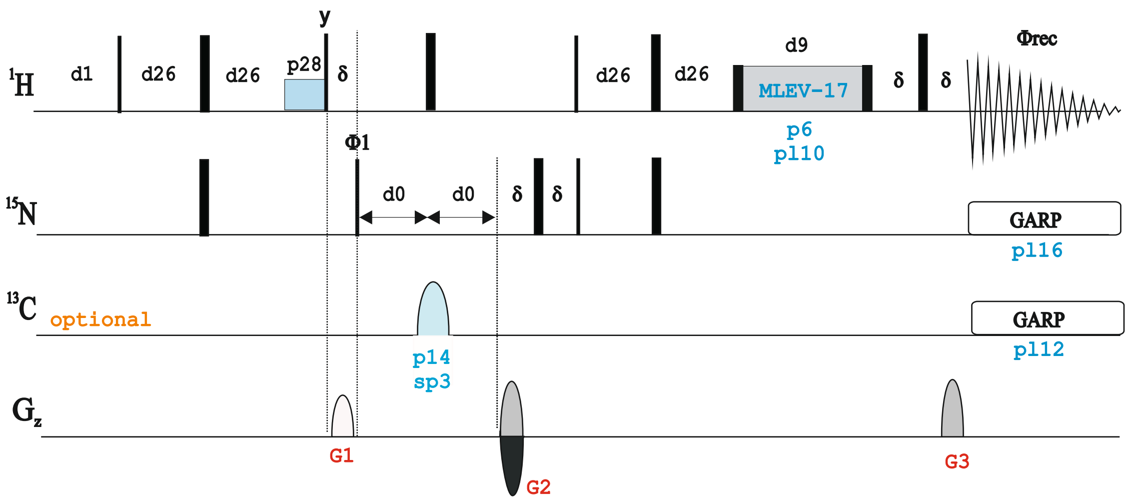

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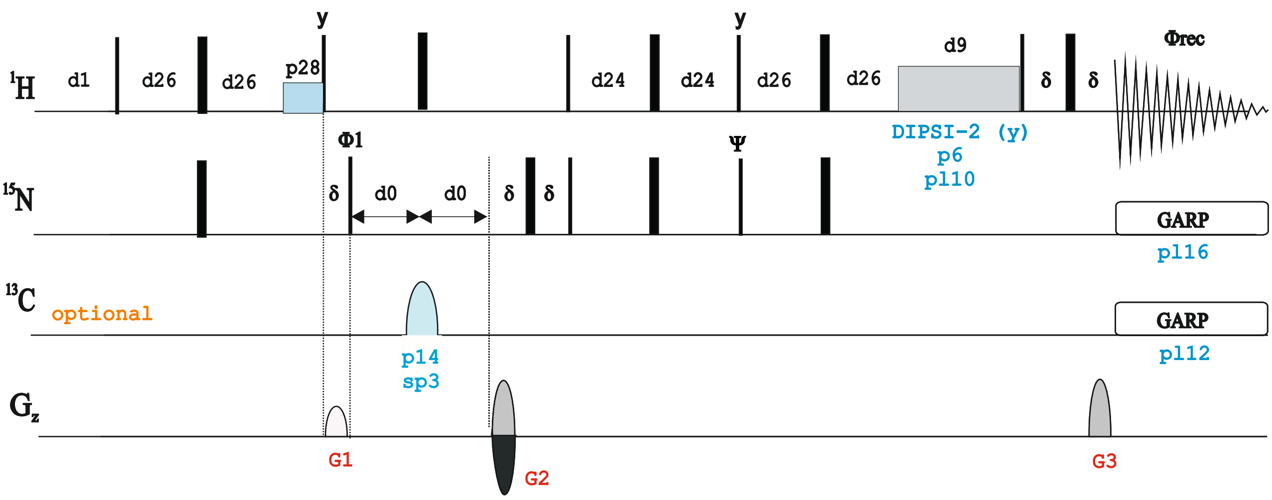

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2D HSQC-ROESY EXPERIMENT

Experiment Description:

The 2D HSQC-ROESY experiment is an hybrid experiment consisting of a first refocused HSQC pulse train (optimized to d4=1/4J(XH)) followed by a mixing ROESY process defined by p15. Other experimental set-up as usually made in HMQC experiments.
Two type of peaks are obtained in a conventional 2D correlation map : i) Direct X-H correlations and ii) ROE correlations connecting a protonated X nucleus with a 1H resonance which is close to the directly-attached 1H-X proton.

The experiment can be recorded without X-decoupling during 1H acquisition (pl12 or pl16 set to 120dB). This is useful to observe NOE between degenerate protons (for instance, symmetrical molecules).

See 2D ROESY and 2D HMQC-ROESY for related experiments.

References

A. Bax & D.G. Davis, J. Magn. Reson 63, 207-213 (1985)

2D HSQC-ROESY Experiments

- **Gradient-enhanced from the f2 channel**

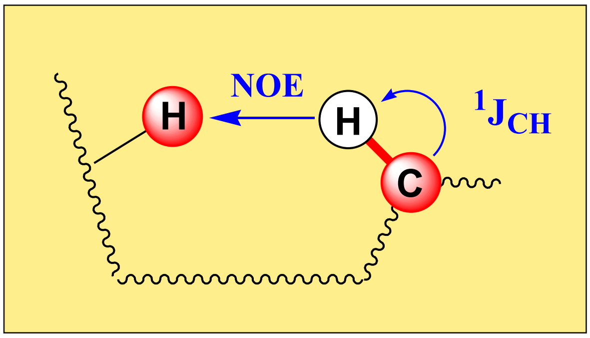
  Phase-sensitive ge-2D HSQC-ROESY using echo-antiecho and adiabatic pulses (**hsqcetgprosp** | HSQCETGPROSP)
  Phase-sensitive ge-2D HSQC-ROESY using echo-antiecho and adiabatic pulses with T-ROESY(**hsqcetgprosp.2**)

- **Gradient-enhanced from the f3 channel**

  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC-ROESY using echo-antiecho (**hsqcetf3gpro**)
  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC-ROESY with T-ROESY using echo-antiecho (**hsqcetf3gpro.2**)

Also see:
2D HSQC and 2D ROESY
2D HMQC-ROESY experiments

The pulse p15 (in microseconds), applied at power level pl11, defines the mixing period in all ROESY experiments.



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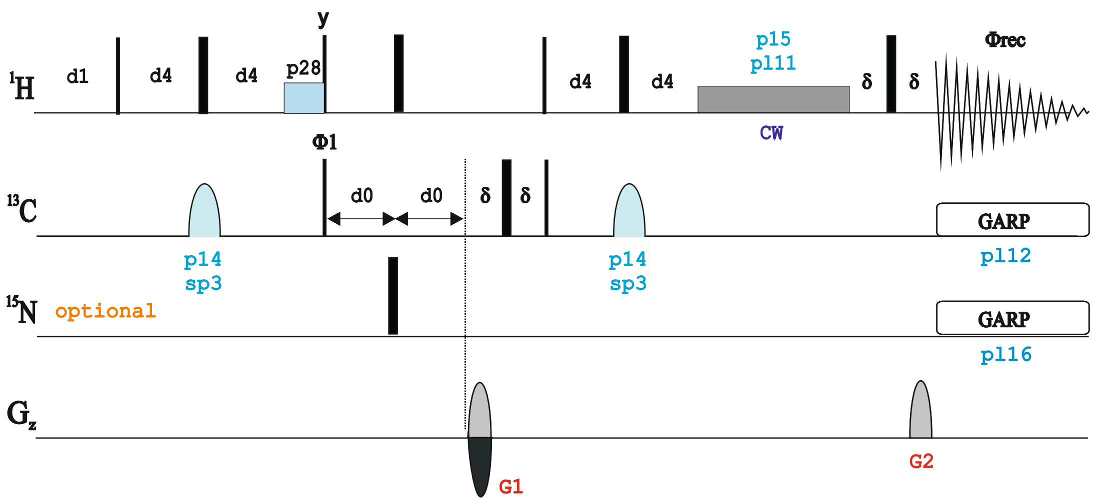

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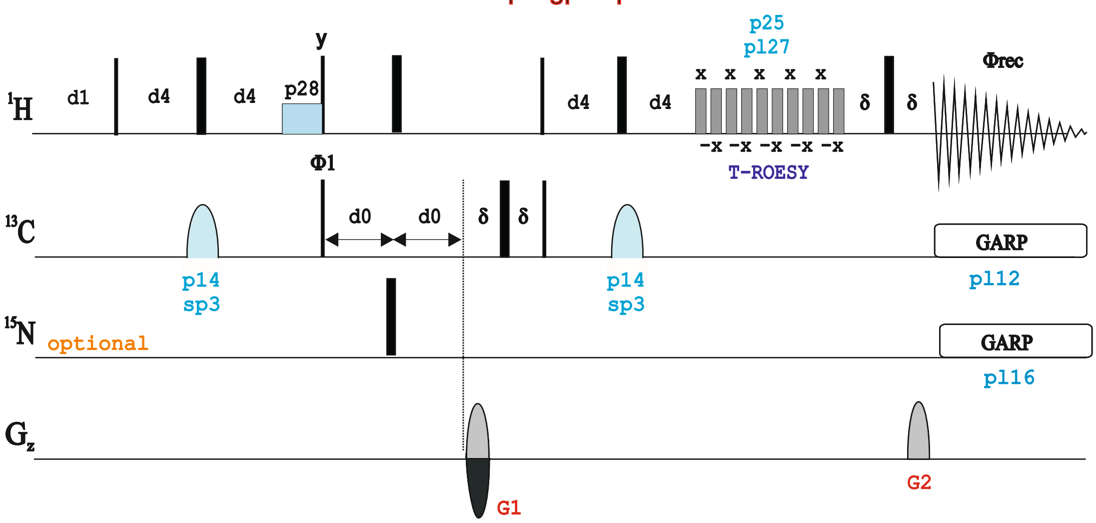

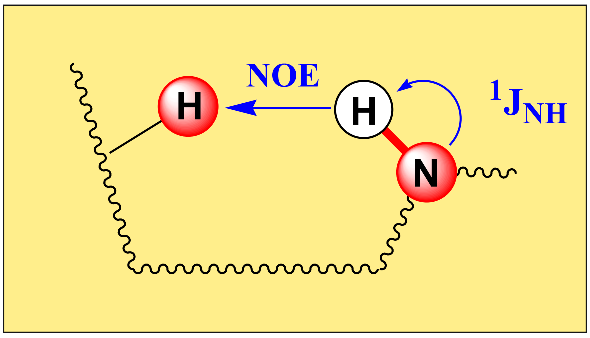
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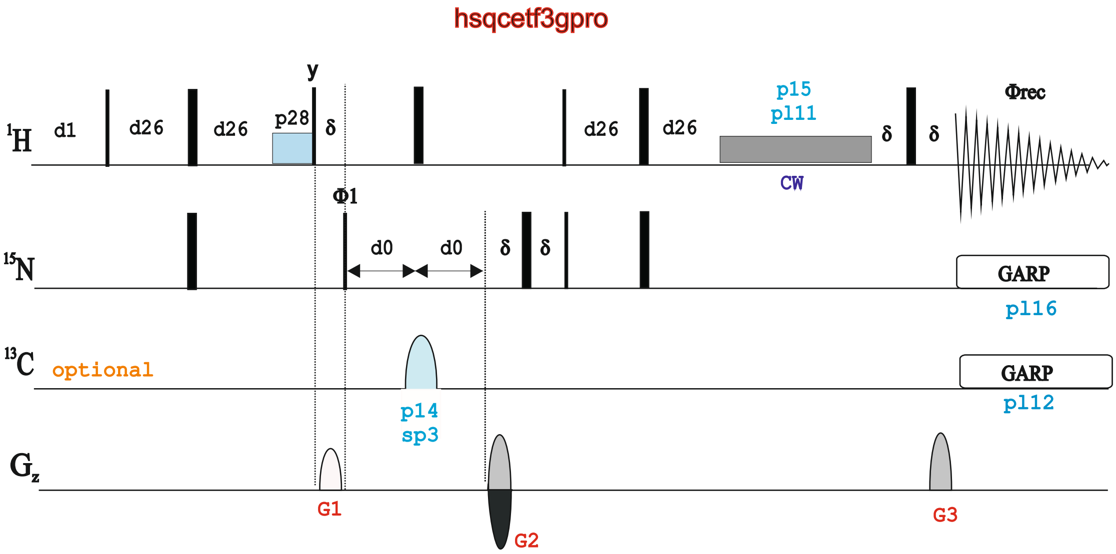
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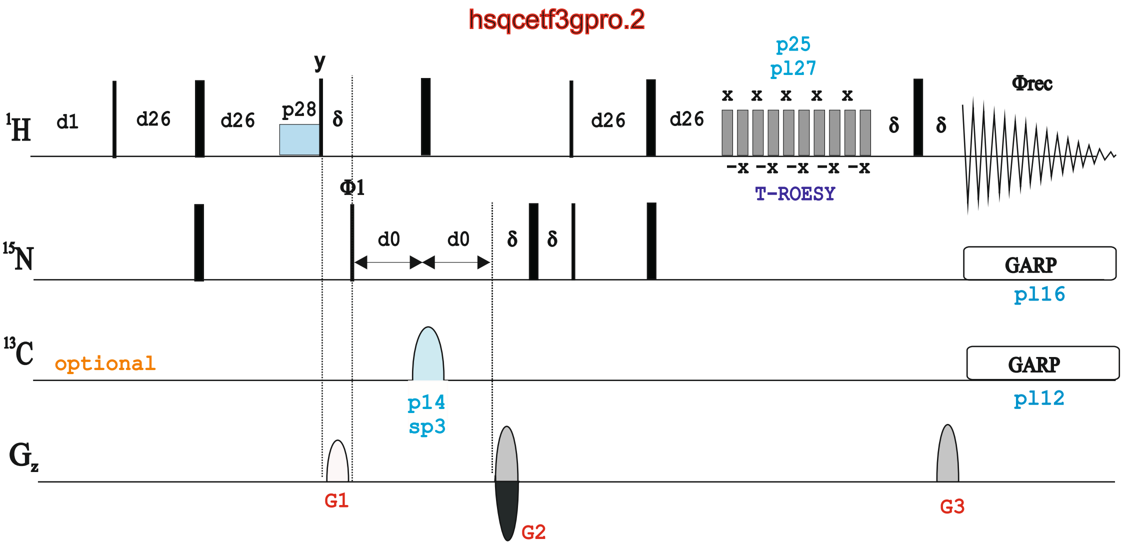
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13C
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G1
G2
G3

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2D HSQC-NOESY EXPERIMENT

Experiment Description:

The 2D HSQC-NOESY experiment is an hybrid experiment consisting of a first refocused HSQC pulse train (optimized to d4=1/4J(XH)) followed by a mixing NOESY building block defined by d8. Other experimental set-up as usually made in HSQC experiments.
Two type of peaks are obtained in a conventional 2D correlation map : i) Direct X-H correlations and ii) ROE correlations connecting a protonated X nucleus with a 1H resonance which is close to the directly-attached 1H-X proton.

The experiment can be recorded without X-decoupling during 1H acquisition (pl12 or pl16 set to 120dB). This is useful to observe NOE between degenerate protons (for instance, symmetrical molecules).

See 2D NOESY and 2D HMQC-NOESY for related experiments.

2D HSQC-NOESY Experiments

- **Gradient-enhanced from the f2 channel**

  Phase-sensitive ge-2D HSQC-NOESY using echo-antiecho and adiabatic pulses (**hsqcetgpnosp** | HSQCETGPNOSP)

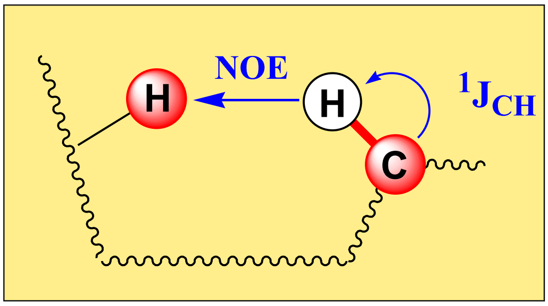
- **Gradient-enhanced from the f3 channel**

  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC-NOESY using echo-antiecho (**hsqcetf3gpno** | HSQCETF3GPNO)
  Phase-sensitive ge-2D ^{1}H-^{15}N HSQC-NOESY using XY16 and WATERGATE (**hsqcf3gpnowgxy**)

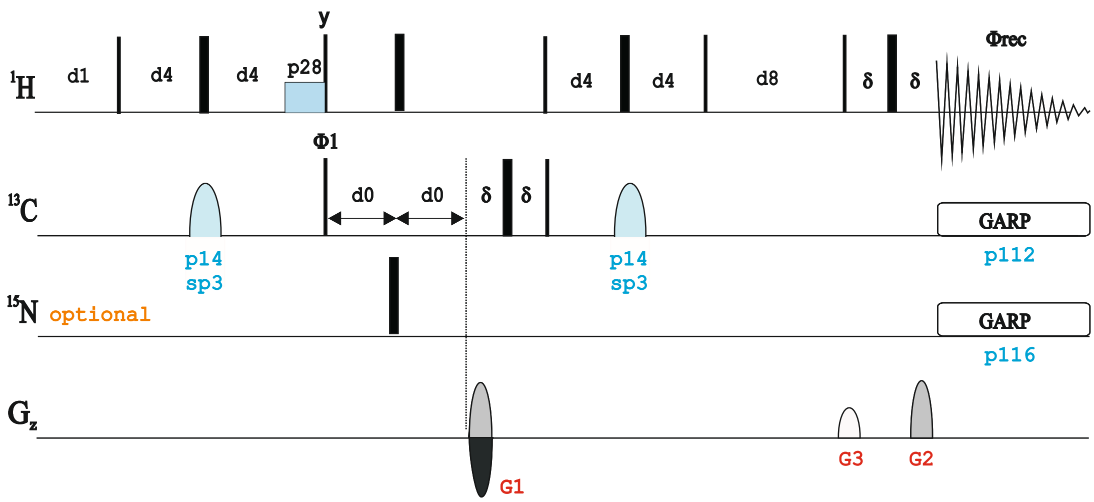
Also see in "Selective 1D Experiments":
Selective ge-1D HSQC-NOESY experiment (selhsqcgpnosp)
Selective ge-1D HSQC-NOESY experiment without decoupling (selhsqcgpndnosp)

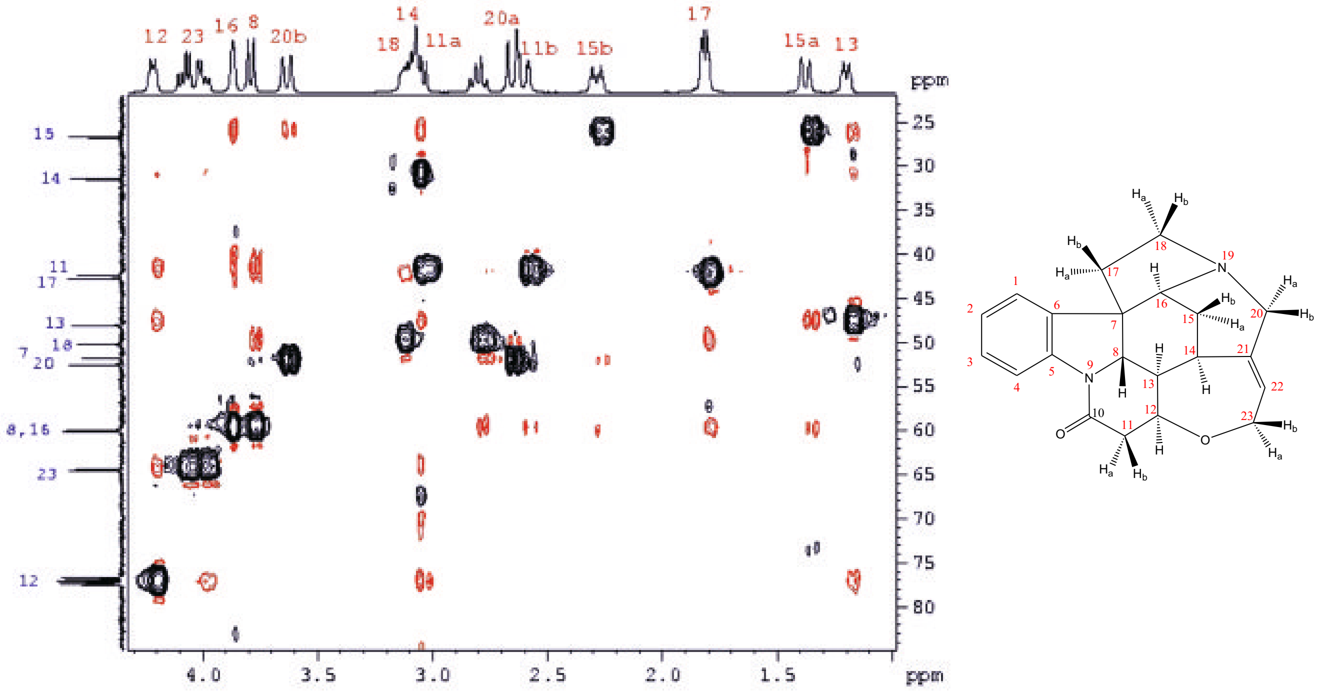
Also see:
2D HSQC and 2D NOESY
2D HSQC-ROESY Experiments

The delay d8 defines the mixing period in all NOESY experiments,

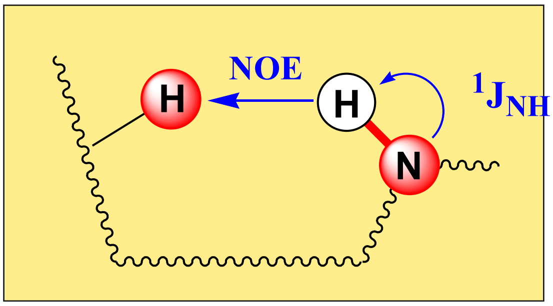


hsqcetgpnosp





2D 1H-13C HSQC-NOESY spectrum of strychnine



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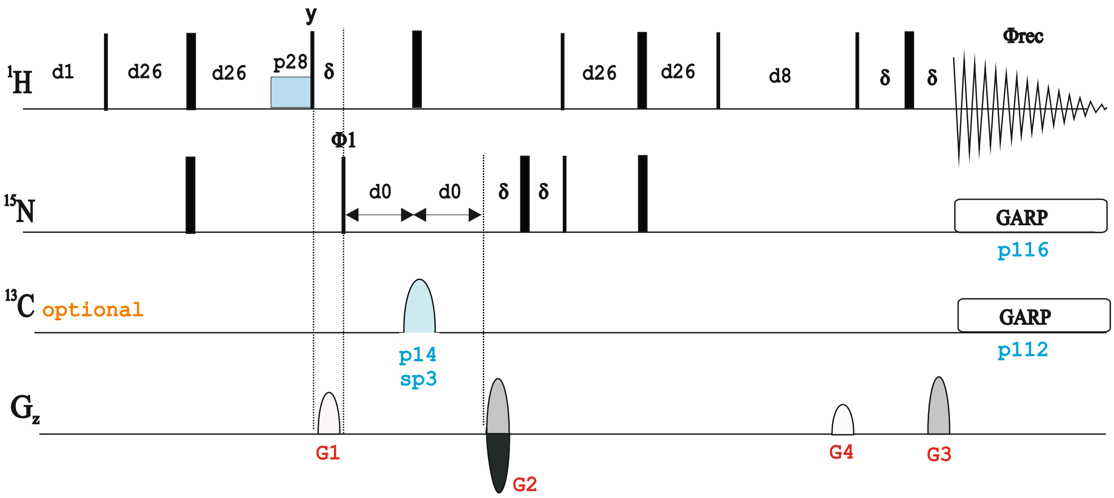

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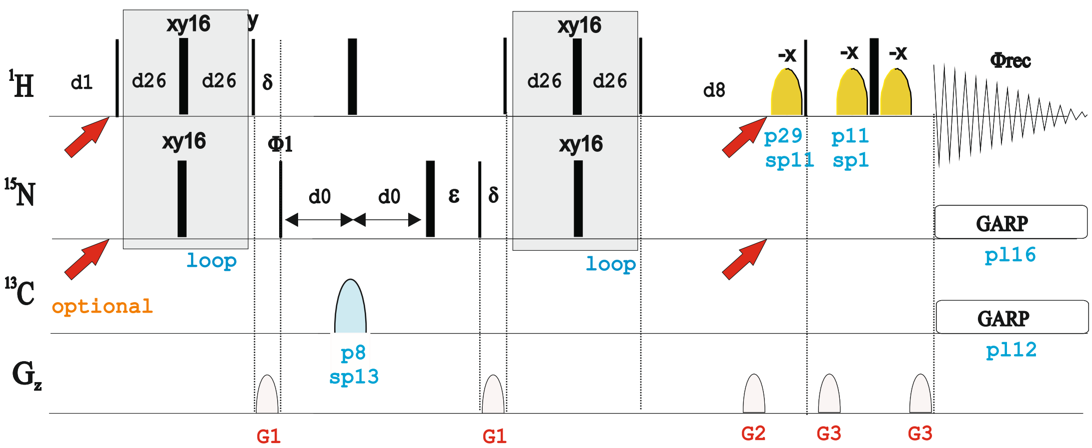

1. L. Mueller, P. Legault & A. Pardi, J. Am. Chem. Soc. 117, 11043-11048 (1995)
2. F.A.A. Mulder, C.A.E.M. Spronk, M. Slijper, R. Kaptein & R. Boelens, J. Biomol. NMR 8, 223-228 (1996)

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2D HMBC EXPERIMENTS

Experiment Description

The HMBC (Heteronuclear Multiple-Bond Correlation) experiment is a proton-detected experiment designed to obtain long-range (usually two-bond and three-bond) heteronuclear correlations between 1H and X heteronuclei via the scalar coupling constant (2J(XH), 3J(XH ...).

The experiment can be applied on any heteronucleus, typically 13C, 15N, 29Si, 31P ...

Sample Requirements

HMBC experiments can be recorded on any type of sample.

Hardware Requirements

HMBC experiments can be recorded on any probehead but an inverse probe equipped with gradients is strongly recommended.

NMR Spectrum

A 2D HMBC experiment yields a typical 1H-X map that correlates 1H and X chemical shifts via nJ(XH) (n>1). However, residual one-bond connectivities can be also present as a large doublets (1J around 130-160 Hz). Versions that use low-pass filters can be used to minimize them.

The most important parameter to optimize is the **d6 delay** that allows the evolution to nJ(CH) in order to achieve antiphase magnetization. Usually, a d6 value of 60-65 ms is a good starting point for routine applications.

Related Experiments

See modified 2D HMBC and long-range HSQC (HSQMBC) pulse sequences to measure quantitatively the value of long-range proton-carbon coupling constants, nJ(XH).

Also see other experiments that provide long-range heteronuclear correlations such as 2D COLOC, 2D ADEQUATE ...

References:

1. A. Bax & M.F. Summers, J. Am. Chem. Soc. 108, 2093 - 2094 (1986)

2D HMBC Experiments

- **Phase cycled:**

  Magnitude-mode 2D HMBC using low-pass J-filter (**hmbclpndqf** | HMBCLPND)
  Magnitude-mode 2D HMBC with presaturation (**hmbcndprqf**)
  Magnitude-mode 2D HMBC with off-resonance presaturation (**hmbcndpsqf**)

- **Gradient-based from f2 channel:**

  Magnitude-mode ge-2D HMBC (**hmbcgpndqf** | HMBCGPND)
  Magnitude-mode ge-2D HMBC using low-pass J-filter (**hmbcgplpndqf** | HMBCGPLPND)
  Magnitude-mode ge-2D HMBC using double low-pass J-filter (**hmbcgpl2ndqf**)

  Phase-sensitive ge-2D HMBC using echo-antiecho (**hmbcetgpnd**)
  Phase-sensitive ge-2D HMBC using a two-fold low-pass J-filter (**hmbcetgpl2nd**)
  Phase-sensitive ge-2D HMBC using a three-fold low-pass J-filter (**hmbcetgpl3nd**)
  Phase-sensitive ge-2D Multiplicity-edited HMBC using a three-fold low-pass J-filter (**hmbcedetgpl3nd**)

  ge-2D Constant-time HMBC (CT-HMBC) using echo-antiecho (**hmbcctetgpnd**)
  ge-2D Constant-time HMBC (CT-HMBC) using echo-antiecho and a two-fold low-pass J-filter (**hmbcctetgpl2nd**)

  Magnitude-mode band-selective ge-2D HMBC without decoupling (**shmbcgpndqf**)

  Magnitude-mode CIGAR-HMBC without decoupling (**hmbcacgplpndqf**)
  Magnitude-mode CIGAR-HMBC with decoupling (**hmbcacgplpqf**)

  ge-2D 2J,3J HMBC, STAR-HMBC (**hmbcacbigpl2ndqf**)
  ge-2D HSMC (**hmscetgpnd**)

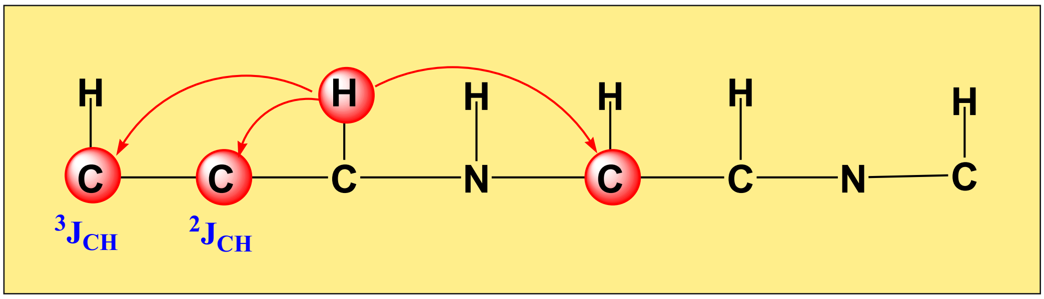
- **Gradient-based form f3 channel:**

  Magnitude-mode ge-2D HMBC (**hmbcf3gpndqf**)
  Magnitude-mode ge-2D HMBC using low-pass J-filter (**hmbcf3gplpndqf**)
  Phase-sensitive ge-2D HMBC using echo-antiecho (**hmbcetf3gpnd**)
  Phase-sensitive ge-2D HMBC using a three-fold low-pass J-filter (**hmbcetf3gpl3nd**)

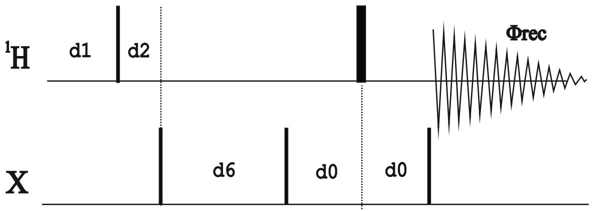
- **Gradient-based simultaneous CN-HMBC**

  Magnitude-mode ge-2D simultaneous ^{1}H-^{15}N/^{1}H-^{13}C HMBC (**hmbcetgpl2ndsm**)

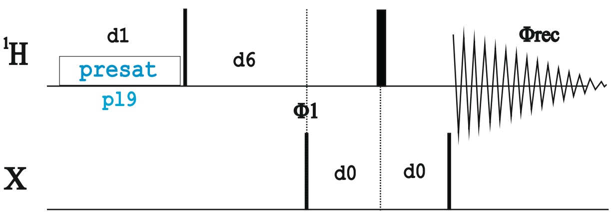
Also see in "Measurement of long-range proton-carbon coupling constants":
  ge-2D J-HMBC using a two-fold low-pass J-filter (hmbcetgpjcl2nd)
  Long-Range HSQC Experiments (HSQMBC)



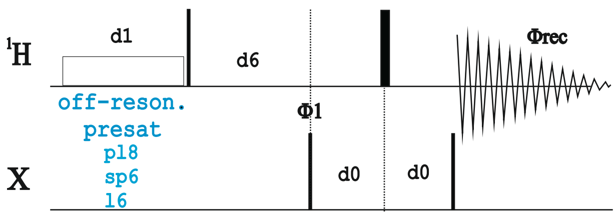
hmbclpndqf

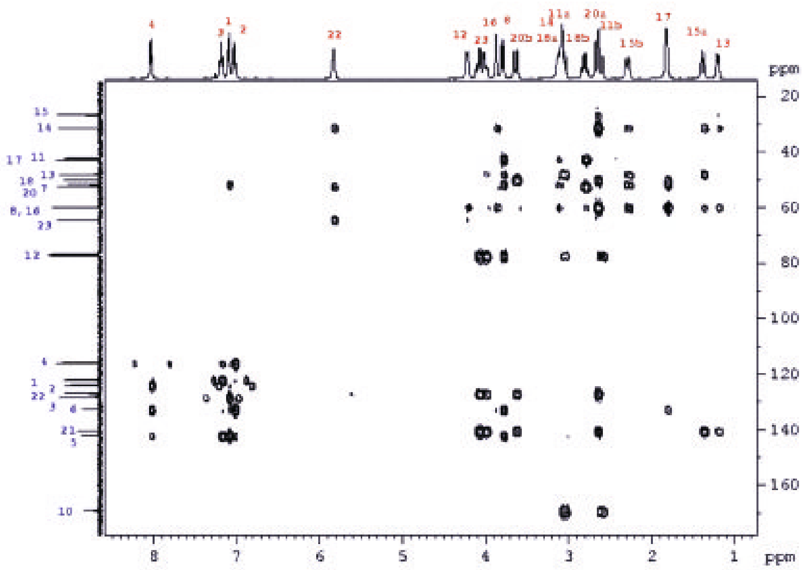


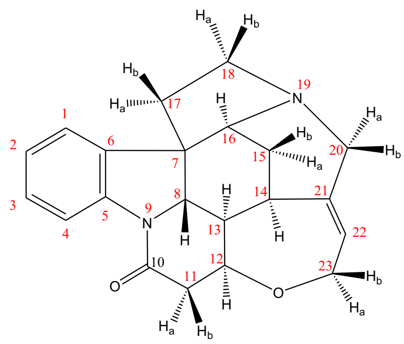
hmbcndprqf



hmbcndpsqf





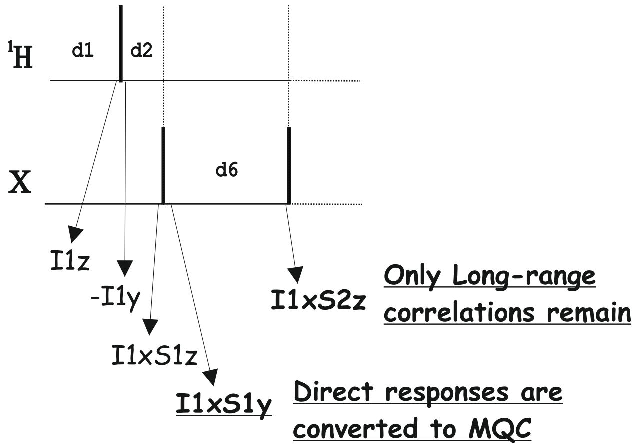


2D 1H-13C HMBC spectrum of strychnine

NMR Building Block:
A Low-Pass Filter

```
...
1 ze
2 d1
3 p1 ph1
  d2
  p3:f2 ph3
  d6
  p3:f2 ph4
.....

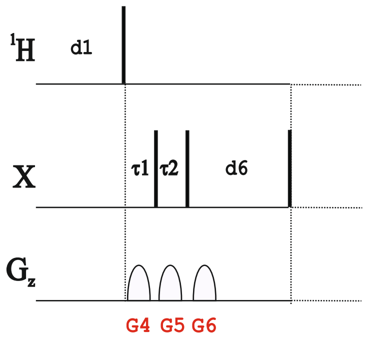
;d2 : 1/(2J)XH
;d6 : delay for evolution
;of long range couplings
```



NMR Building Block:
A Two-Fold Low-Pass Filter

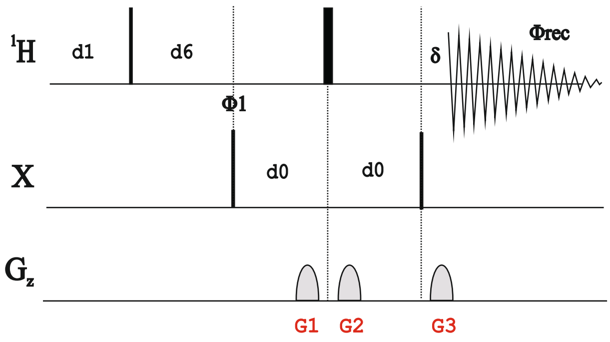
```
"DELTA1=1s/(2 * cnst6)-p16-d16"
"DELTA2=1s/(2 * cnst7)-p16-d16"
"DELTA3=d6-p16-d16-4u"

...
3 p1 ph1
  DELTA1 UNBLKGRAD
  p16:gp4
  d16
  p3:f2 ph3
  DELTA2
  p16:gp5
  d16
  p3:f2 ph3
  4u
  p16:gp6
  d16
  DELTA3
  p3:f2 ph4
....
;cnst6: = 1J(XH)min
;cnst7: = 1J(XH)max
;cnst13: = J(XH) long range
;gpz4: 15%
;gpz5: -10%
;gpz6: -5%
```



hmbcgpndqf

hmbcf3gpndqf

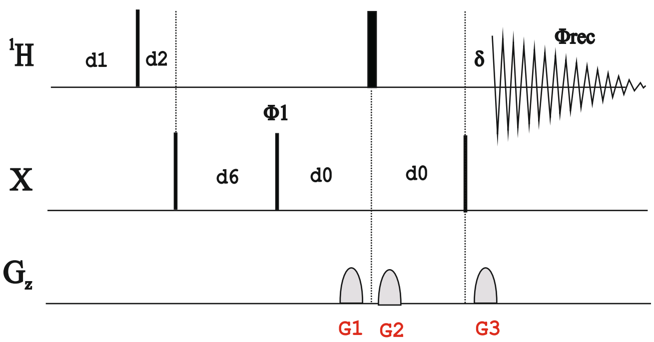


gp 1 : gp 2 : gp 3
50 : 30 : 40.1 for C-13
70 : 30 : 50.1 for N-15

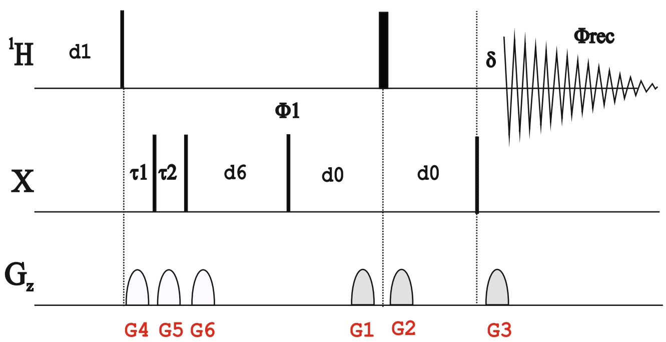
Use the AU program **gradratio** for calculation_of gradient ratios for common inverse gradient pulse programs.

hmbcgplpndqf

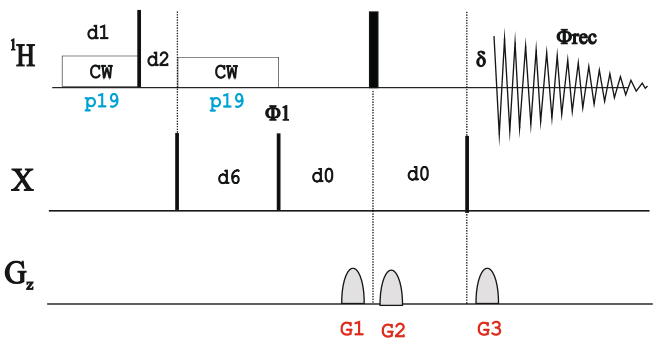
hmbcf3gplpndqf

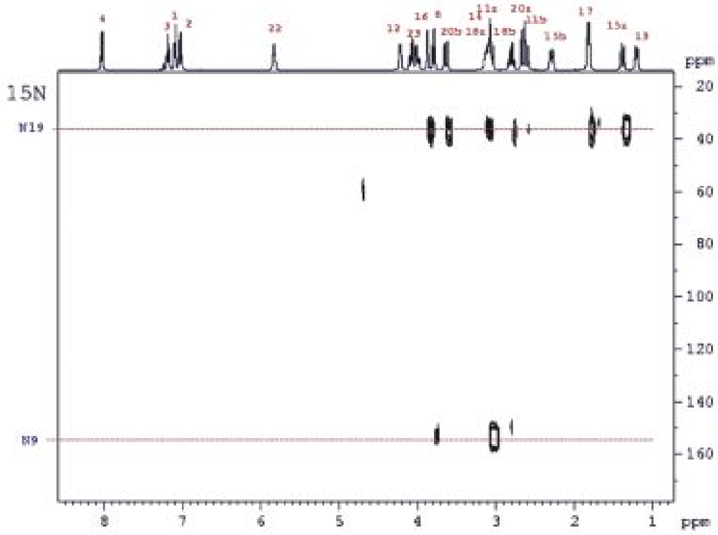


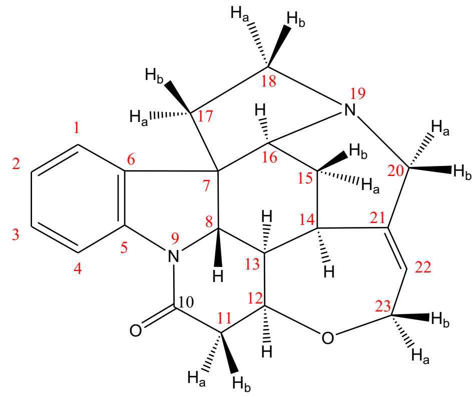
hmbcgpl2ndqf



hmbcgplpndprqf

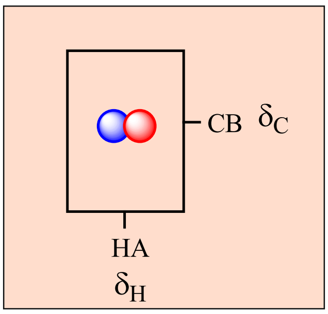




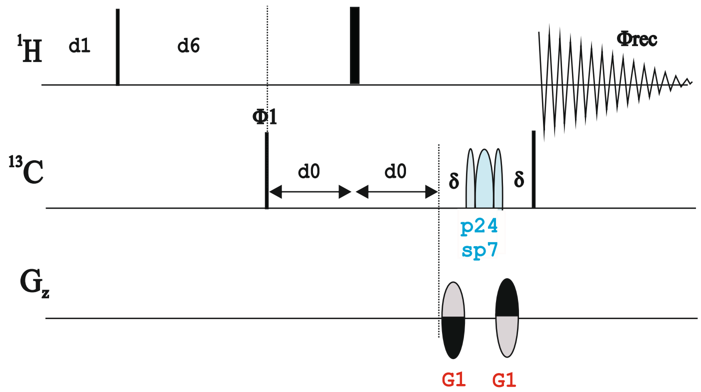


2D 1H-15N HMBC spectrum of strychnine

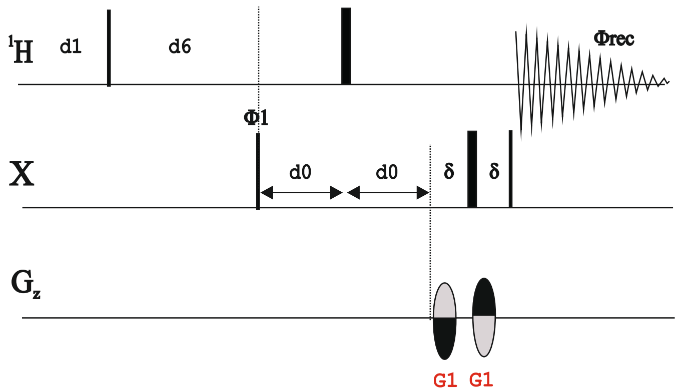
Phase-Sensitive HMBC:
For Measurement of nJCH.
D.O. Cicero, G. Barbato & R. Bazzo, J. Magn. Reson. 148, 209-213 (2001)



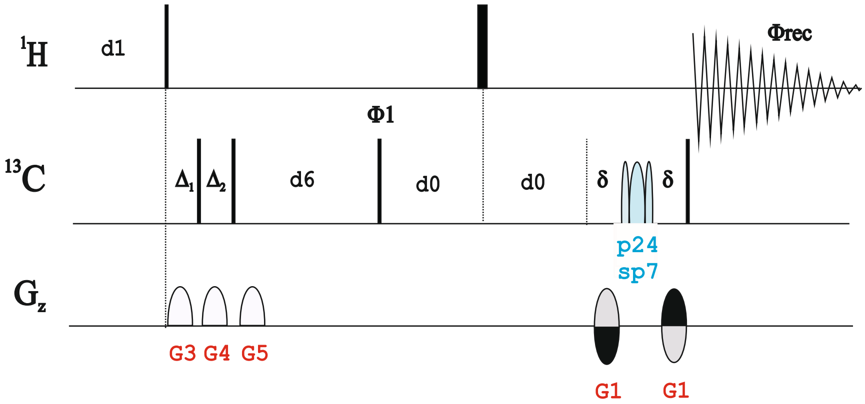
hmbcetgpnd



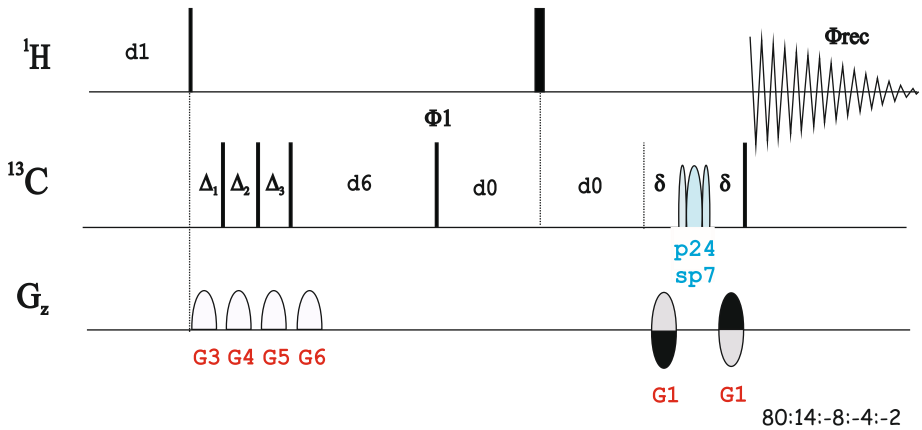
hmbcetf3gpnd



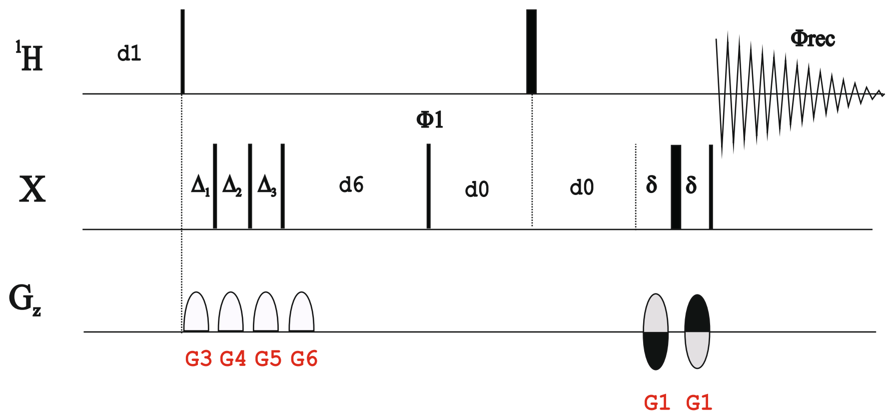
hmbcetgpl2nd



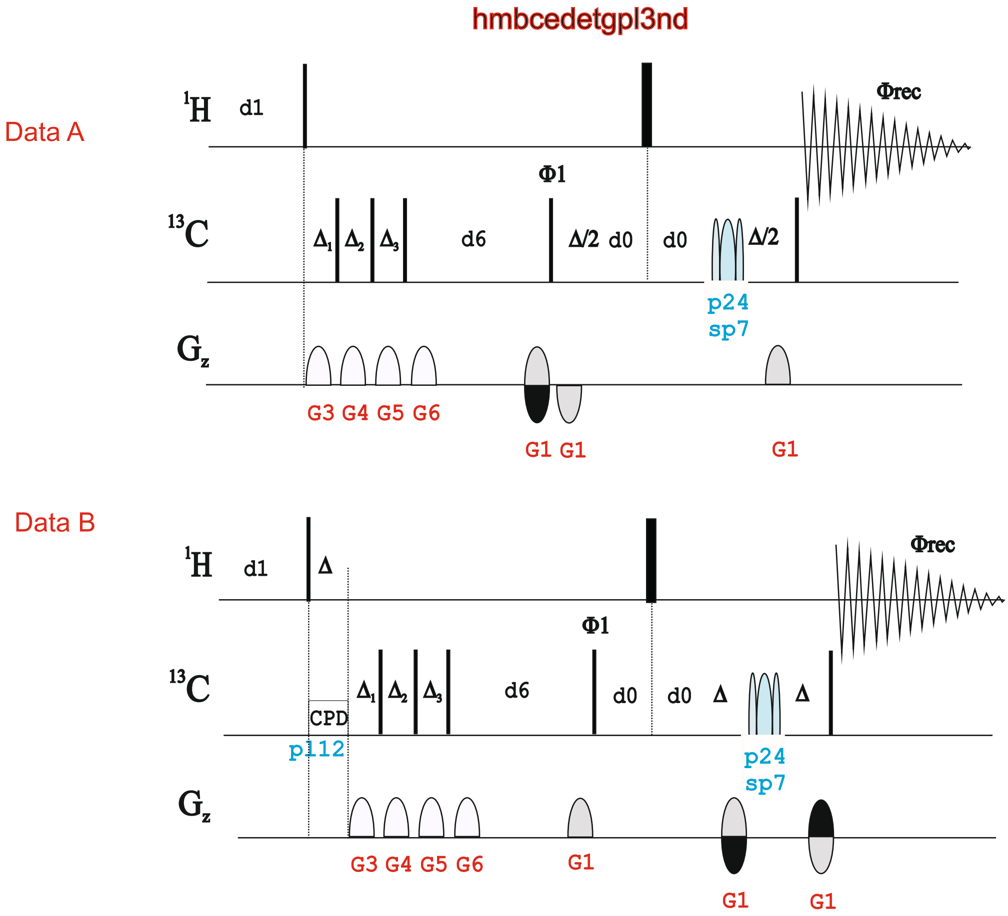
hmbcetgpl3nd



hmbcetf3gpl3nd



Edited-HMBC:
N.T. Nyberg & O.W. Soerensen, Magn. Reson. Chem. 44, 451-454 (2006)

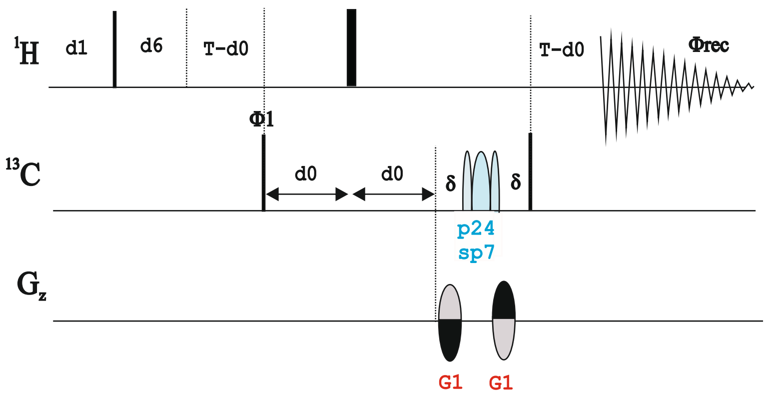


Processing:
use AU-program split [ipap 2] to split data

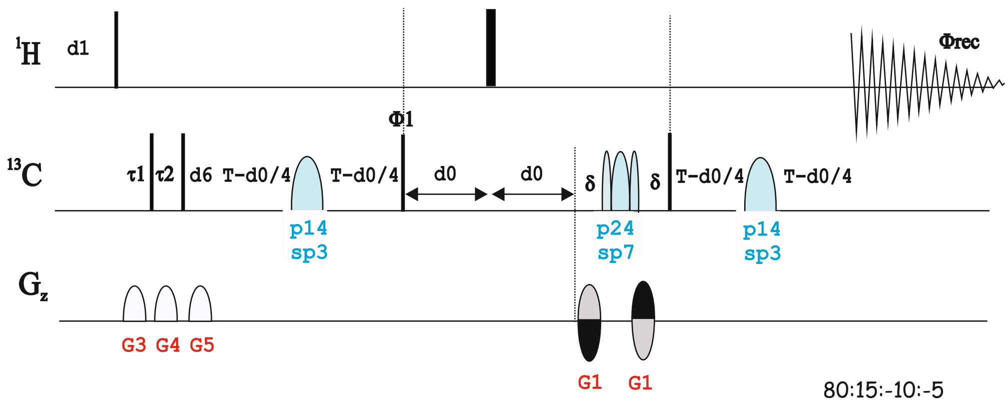
CT-HMBC:

T.D.W. Claridge & I.Perez-Victoria, Org. Biomol. Chem. 1, 3632-3534 (2003)

hmbcctetgpnd

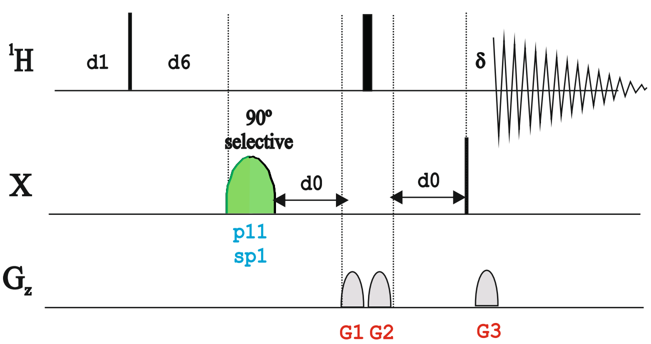


hmbcctetgpl2nd

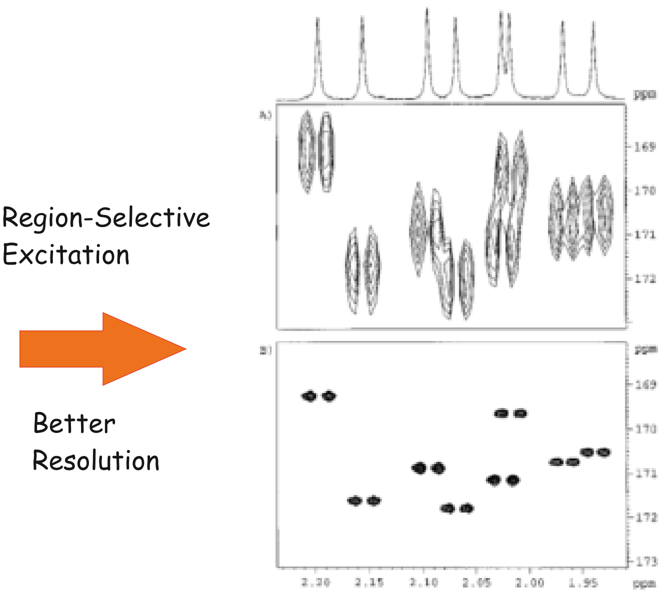


Band-Selective HMBC

shmbcgpndqf



Region-Selective
Excitation

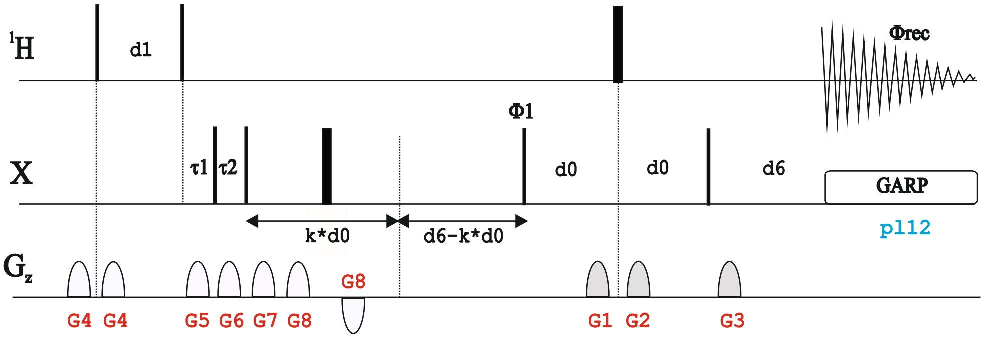


Better
Resolution

CIGAR-HMBC:

1. C.E. Hadden, G.E. Martin & V.V. Krishnamurthy, Magn. Reson. Chem. 38, 143-147 (2000)
2. C.E. Hadden, G.E. Martin & V.V. Krishnamurthy, J. Magn. Reson. 140, 274-280 (1999)
3. R. Wagner & S. Berger, Magn. Reson. Chem. 36, S44-S46 (1998).

hmbcacgplpqf



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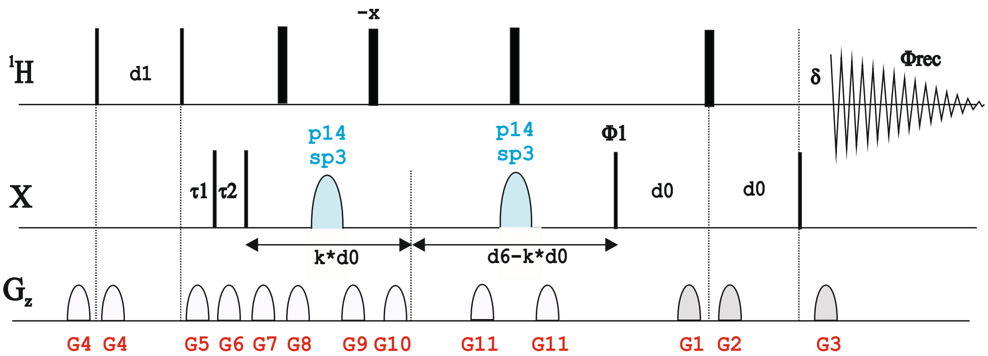

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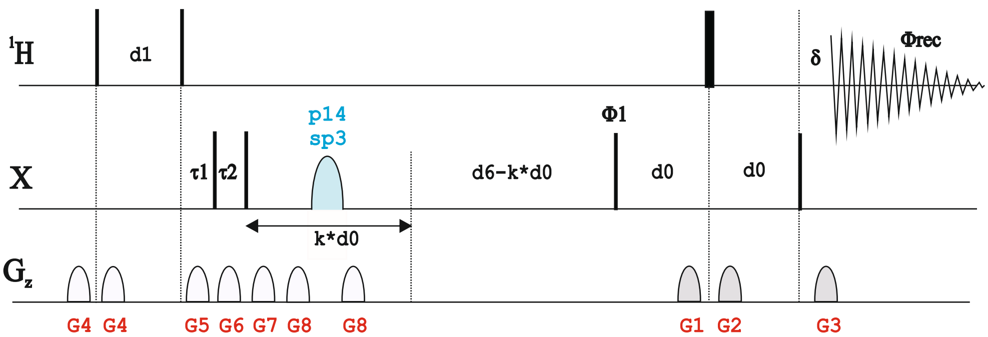
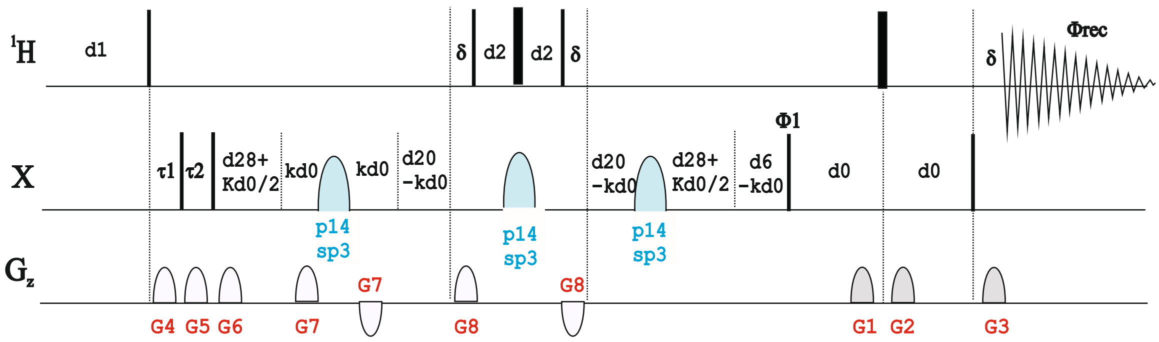


50:30:40.1:5:60:-40:-20:17

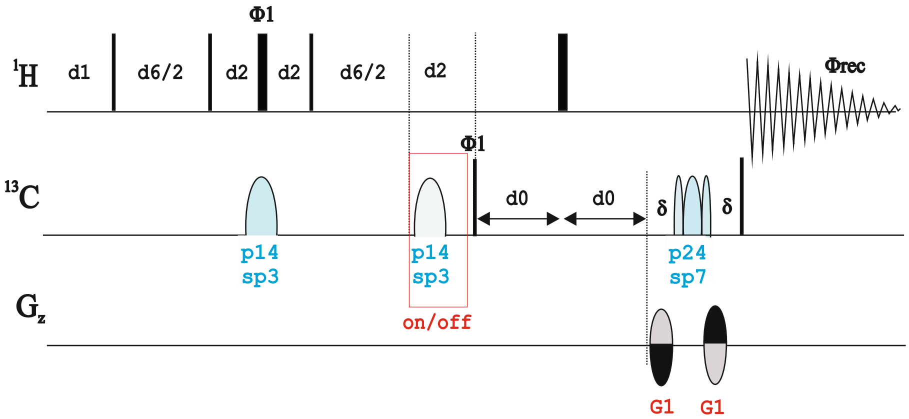
2J,3J-HMBC or STAR-HMBC:
V.V. Krishnamurthy, D.J. Russel, C.E. Hadden & G.E. Martin,
J. Magn. Reson. 146, 232-239 (2000)

hmbcacbigpl2ndqf



HMSC:
R. Burger, C. Schorn & P.Bigler, J. Magn. Reson. 148, 88-94 (2001)

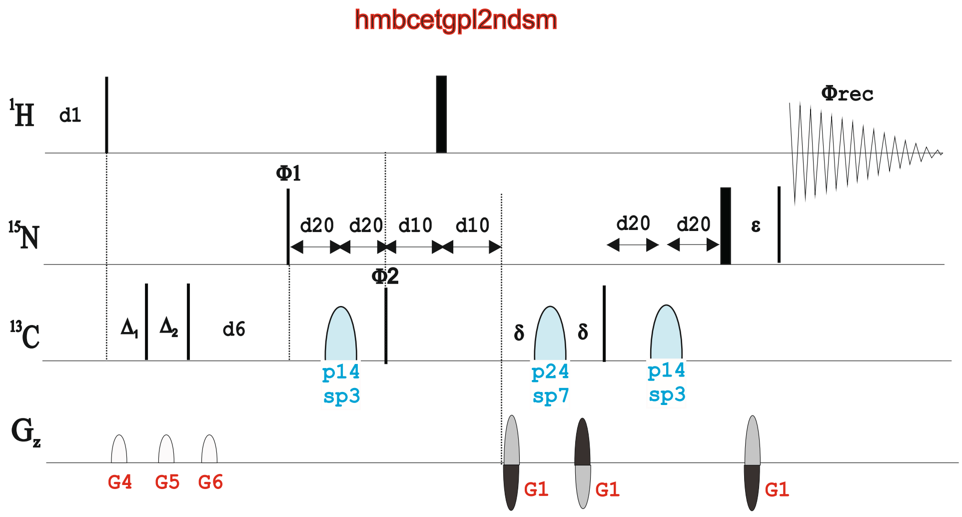
hmscetgpnd

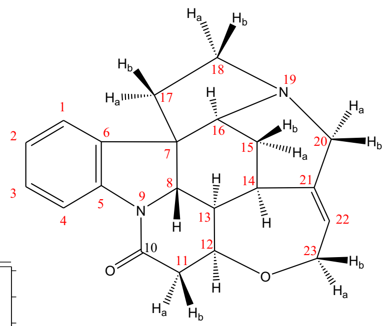


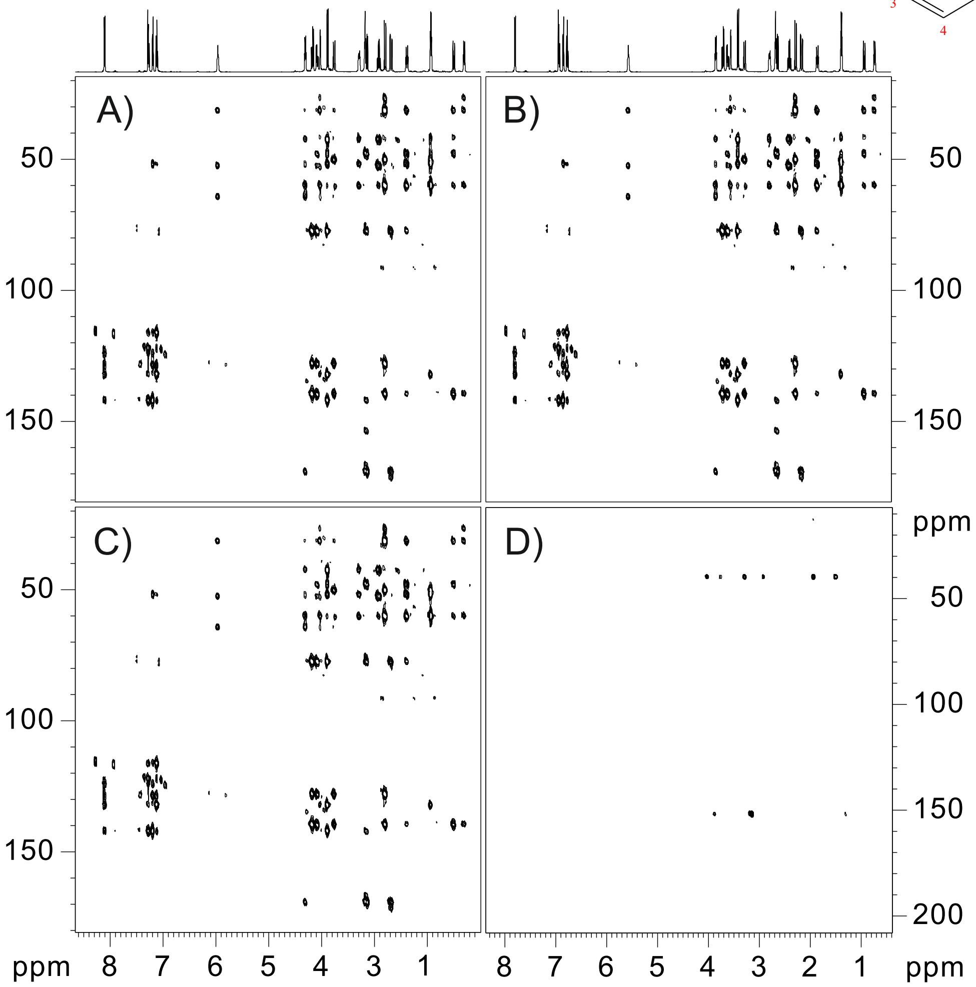
Processing:
use AU-program **split [ipap 2]** to split and recombine data

Simultaneous CN-HMBC:

M. Perez-Trujillo, P. Nolis, W. Bermel & T. Parella, Magn. Reson. Chem. 45, 325-9 (2007)







BRUKER
PULSE PROGRAM
CATALOGUE

NMRGuide

2D EXPERIMENTS TO MEASURE LONG-RANGE PROTON-CARBON COUPLING CONSTANTS

2D Experiments to measure $^{n}J_{CH}$ measurement

- **J- HMBC experiments**

  ge-2D J-HMBC using a two-fold low-pass J-filter (**hmbcetgpjcl2nd**)

- **Long-range optimized ge-2D HSQC**

  Phase-sensitive ge-2D long-range optimized HSQC (HSQMBC) (**hsqcetgplrsp**)
  Phase-sensitive ge-2D long-range optimized HSQC using G-BIRD (GBIRD-HSQMBC) (**hsqcetgpjclrnd**)
  Phase-sensitive ge-2D long-range optimized HSQC using CPMG-XY16 (GBIRD-HSQMBC) (**hsqcetgpjclrndxy**)
  ge-2D long-range optimized J-HSQC (EXSIDE) (**hsqcetgplrjcsp**)

- **ge-2D HSQC-TOCSY type experiments**

  ge-2D w1-filtered TOCSY using DIPSI-2 (HETLOC) (**dipsi2etgpjcsix1**)
  Phase-sensitive ge-2D HSQC-HECADE (**hsqcdietgpjcndsisp**)
  Phase-sensitive ge-2D F2-spin-edited HSQC-TOCSY using DIPSI-2 and sensitivity improvement (**hsqcdietgpiasisp**)

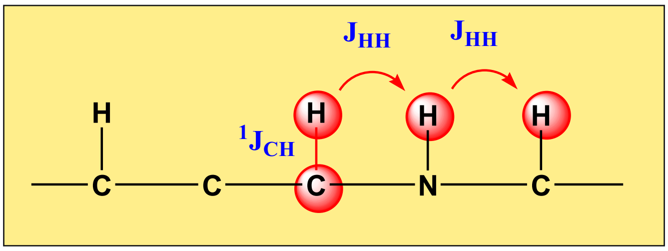
- **2D Selective J-Resolved experiments**

  Magnitude-mode proton-selective 2D Heteronuclear J-Resolved (**seljresqfsp**)

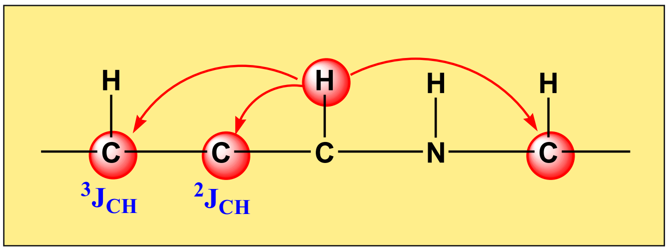
Also see in 2D HMBC Experiments:
Phase-sensitive ge-2D HMBC using echo-antiecho (hmbcetgpnd)
Phase-sensitive ge-2D HMBC using a two-fold low-pass J-filter (hmbcetgpl2nd)
Phase-sensitive ge-2D HMBC using a three-fold low-pass J-filter (hmbcetgpl3nd)
Phase-sensitive ge-2D HMBC using echo-antiecho from f3 channel (hmbcetf3gpnd)
Phase-sensitive ge-2D HMBC using a three-fold low-pass J-filter from f3 channel (hmbcetf3gpl3nd)
Phase-sensitive ge-2D Multiplicity-edited HMBC using a three-fold low-pass J-filter (hmbcedetgpl3nd)
ge-2D Constant-time HMBC (CT-HMBC) using echo-antiecho (hmbcctetgpnd)
ge-2D Constant-time HMBC (CT-HMBC) using echo-antiecho and a two-fold low-pass J-filter (hmbcctetgpl2nd)

Direct measurement on Multiplets in F2-detected Dimension

HSQC-TOCSY

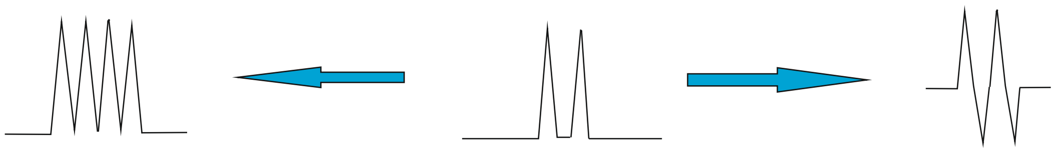


HMBC



Others: HMQC-TOCSY, HECADE, HETLOC
Two steps: ^{1}JCH + JHH
Only for protonated carbons
In-phase Magnetization

Others: HSQMBC, EXSIDE, J-HMBC
A single step: ^{n}JCH
For all carbons
Anti-phase Magnetization



Measurement from spin-state selective patterns:
alpha vs beta in F2
TROSY/antiTROSY pattern
ECOSY pattern

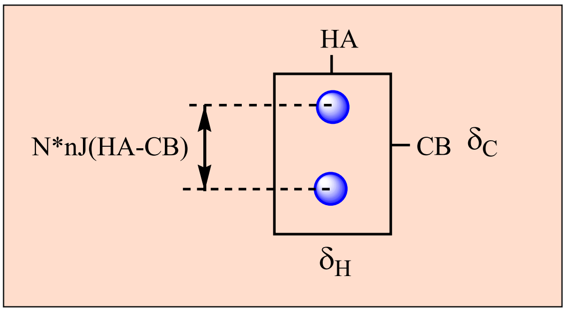
Direct measurement on Multiplets in F1-indirect Dimension
J-Resolved Experiments
J-HMBC Experiments: Amplification factor

J-HMBC:

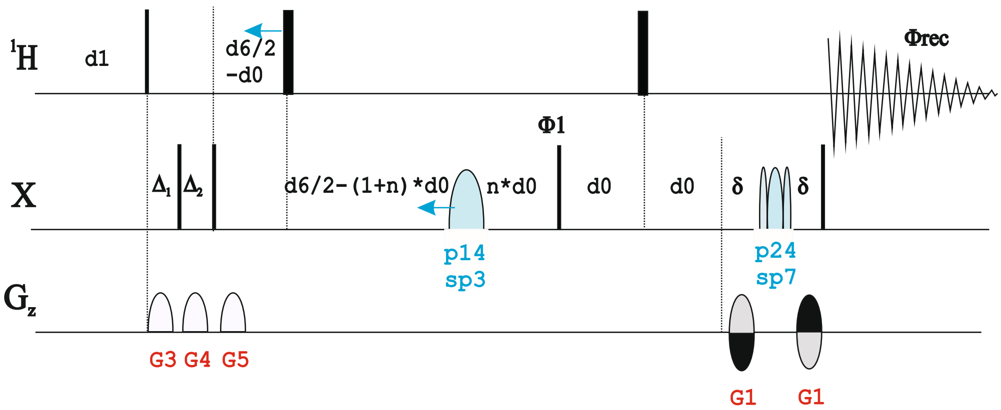
A. Meissner & O.W. Soerensen, Magn. Reson. Chem. 39, 49-52 (2001)

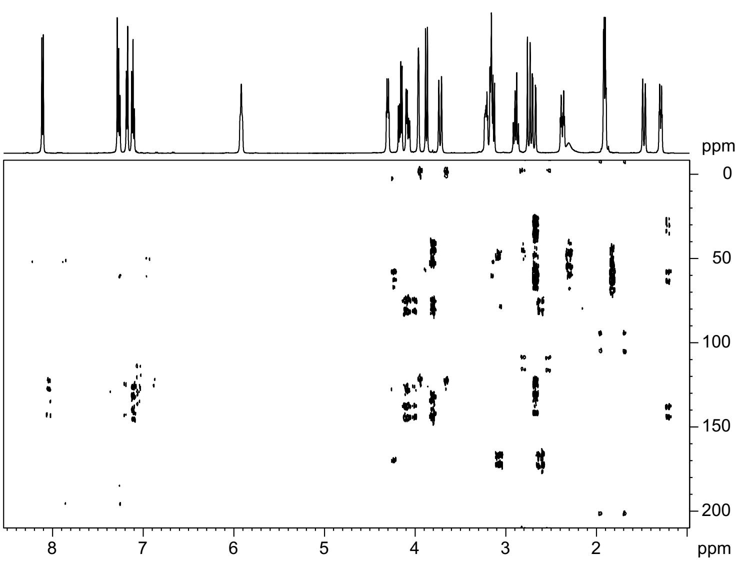
J-scaling factor of N

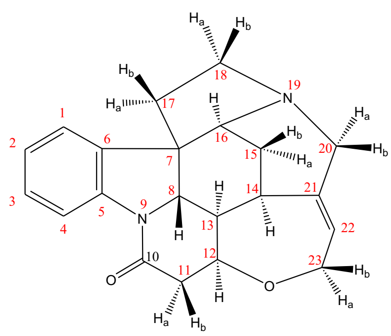
N depends of sw and td1



hmbcetgpjcl2nd

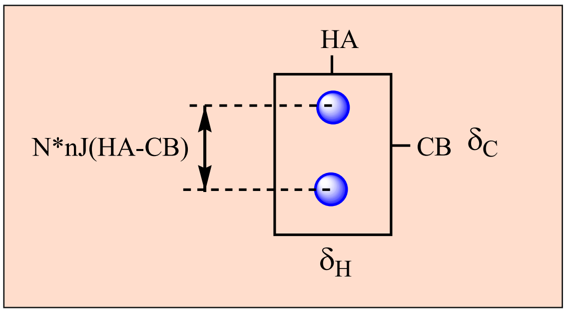




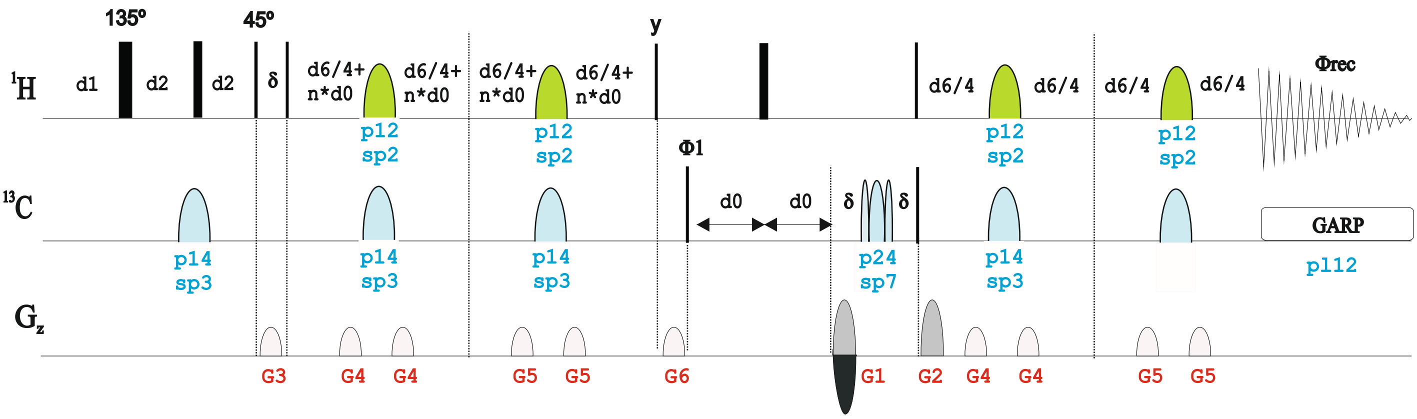


EXSIDE:
V.V. Krishnamurthy, J. Magn. Reson., Series A 121, 33-41 (1996)

J-scaling factor of N



hsqcetgplrjcsp

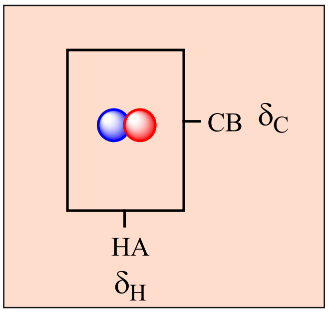


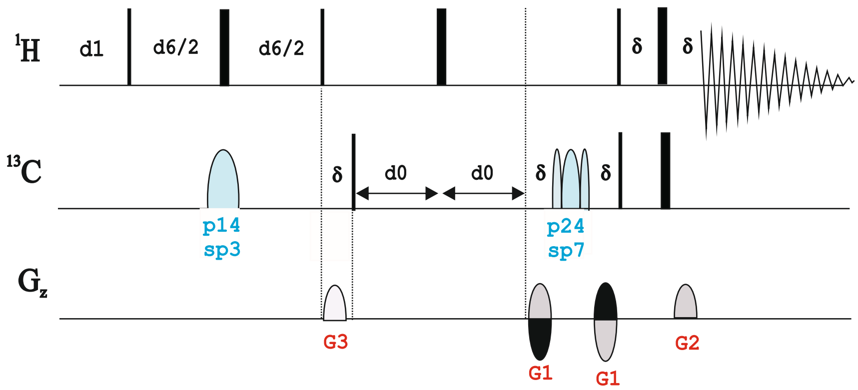
cnst16: = J(scale) factor
p12: f1 channel - 180 degree shaped pulse (selective)

HSQMBC:

1. B.L. Marquez, W.H. Gerwick & R.T. Williamson, Magn. Reson. Chem. 39, 499-530 (2001)
2. R.T. Williamson, B.L. Marquez, W.H. Gerwick & K.E. Kover,
Magn. Reson. Chem. 38, 265-273 (2000)

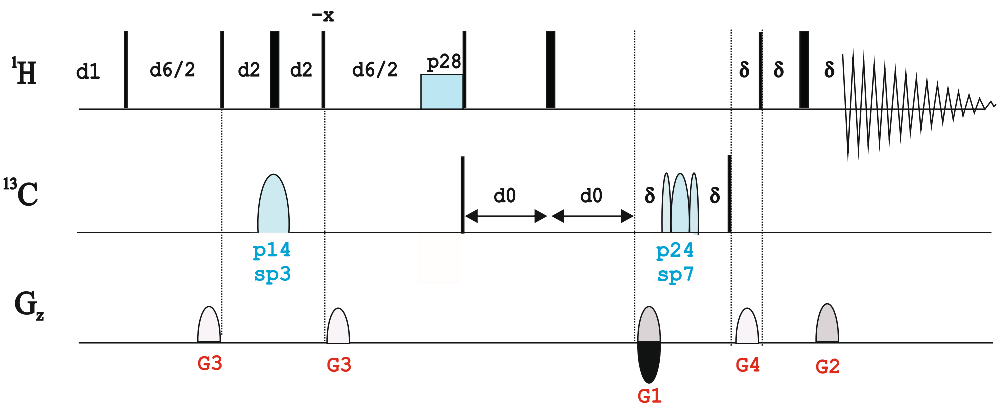
hsqcetgplrsp





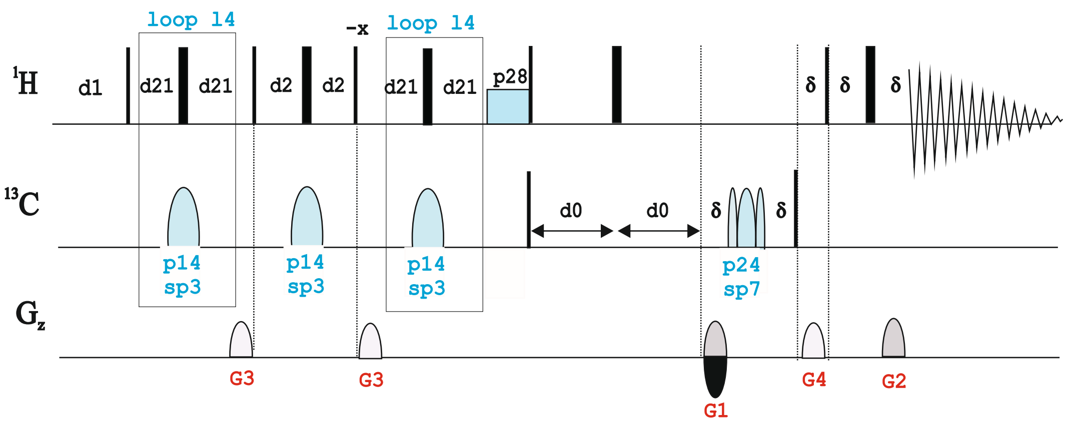
HSQMBC-BIRD

hsqcetgpjclrnd



HSQMBC-BIRD-CPMG

hsqcetgpjclrndxy

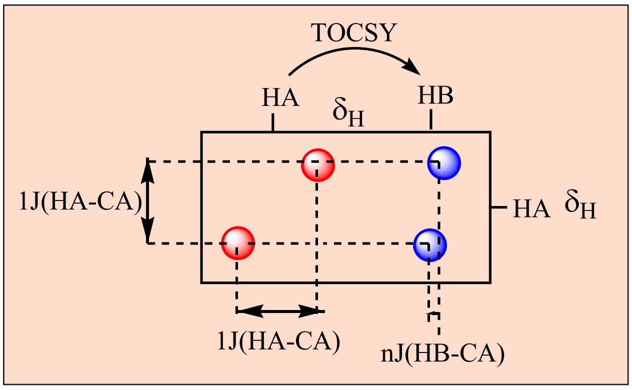


80:20.1:17:11

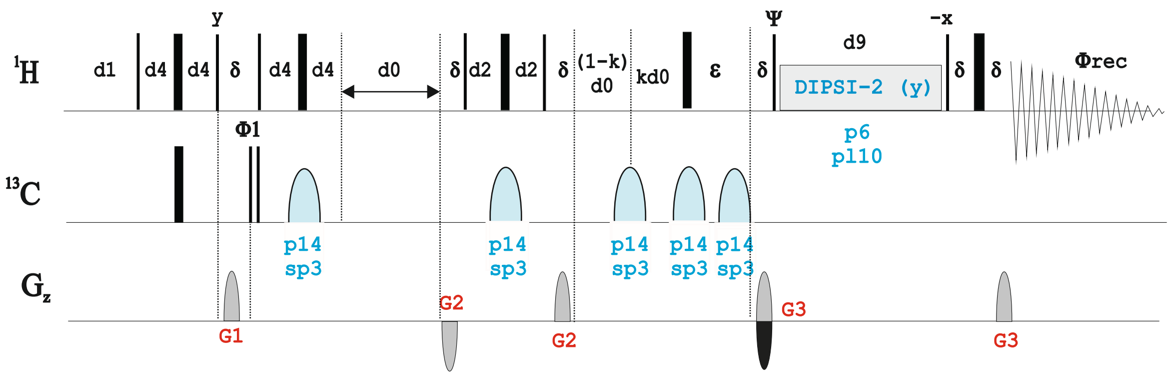
HETLOC:

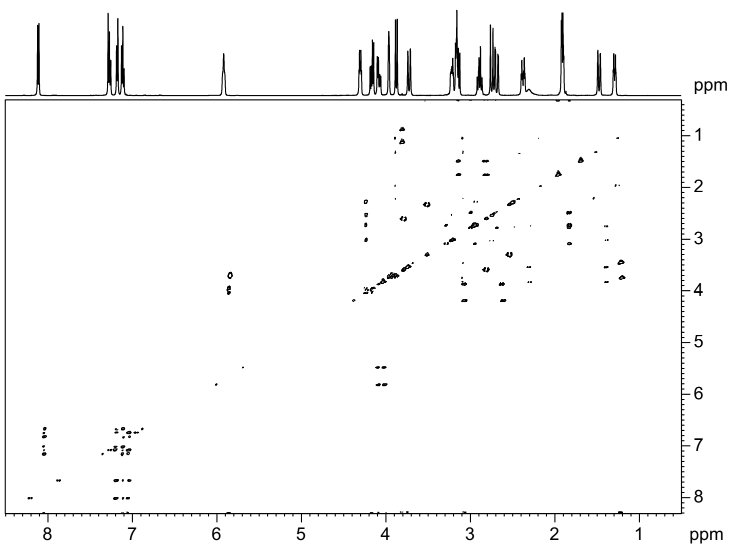
D. Uhrin, G. Batta, V.J. Hruby, P.N. Barlow & K.E. Kover, J. Magn. Reson. 130, 155-161 (1998)

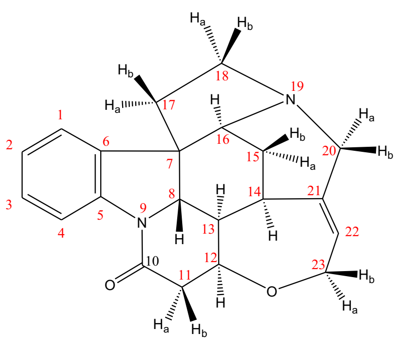
J-scaling factor of N



dipsi2etgpjcsix1



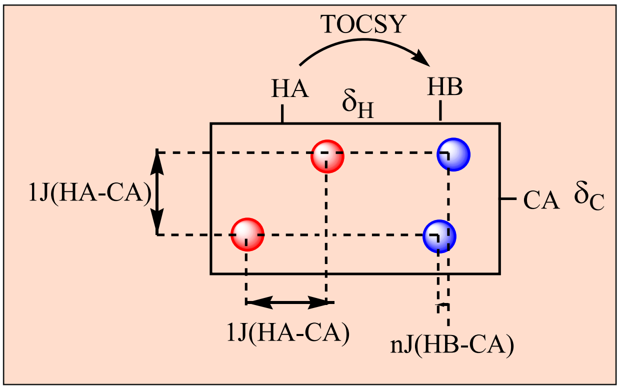




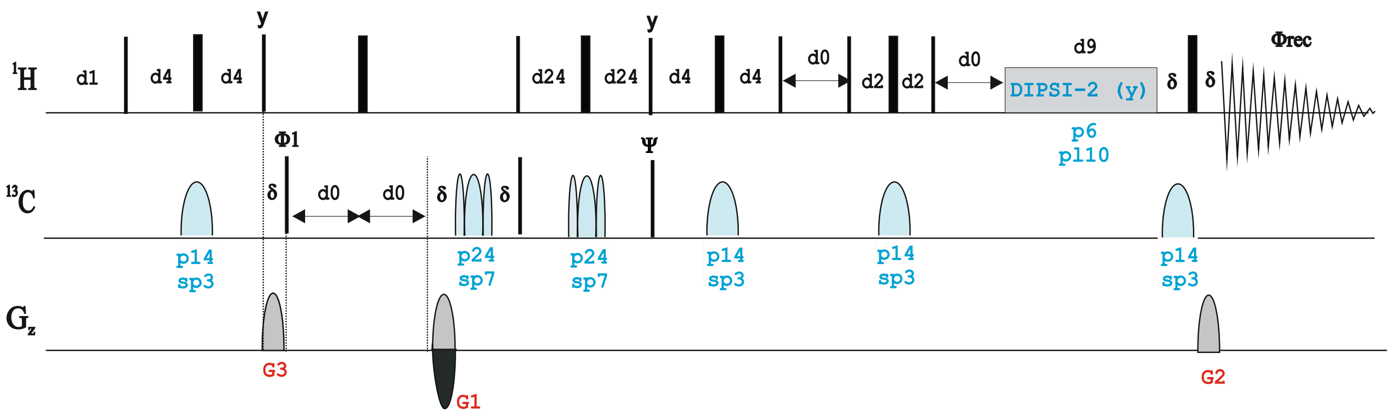
HSQC-HECADE:

W. Kozminski & D. Nanz, J. Magn. Reson. 142, 294-299 (2000)

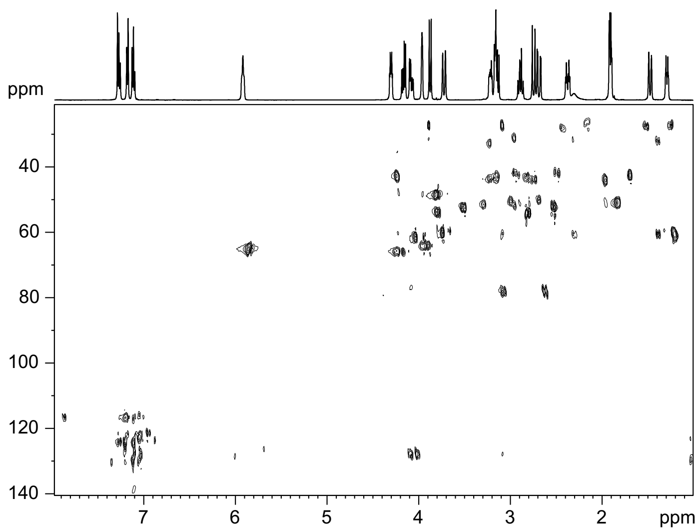
J-scaling factor of N

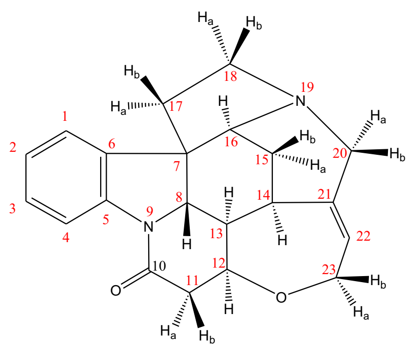


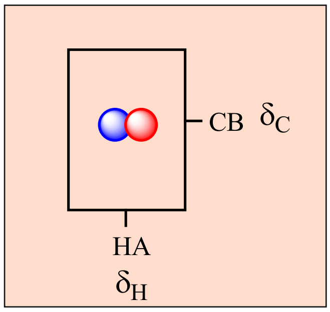
hsqcdietgpjcndsisp



cnst16: = J(scale) factor

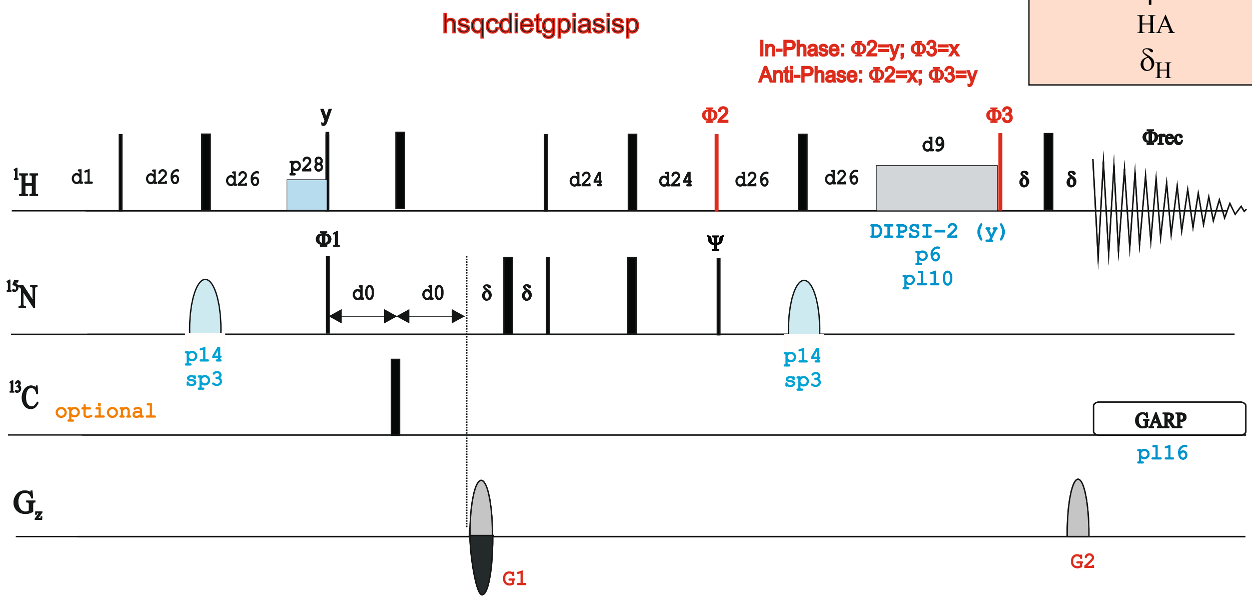






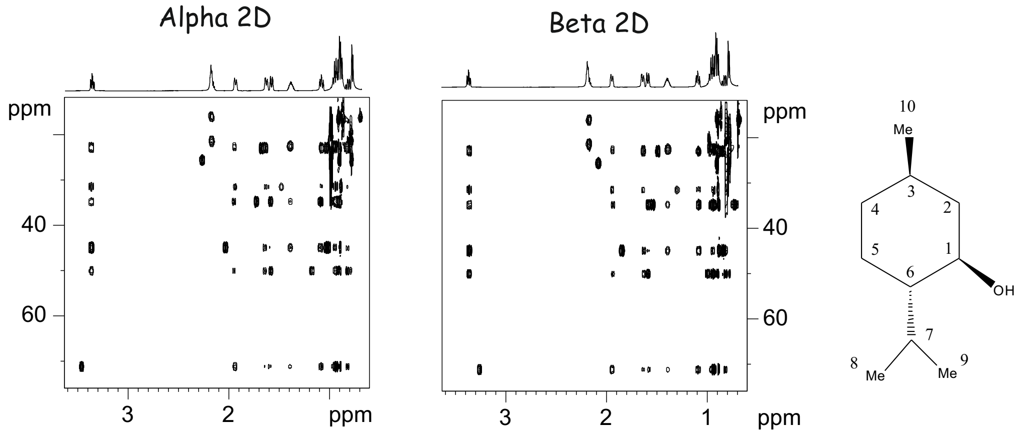
Spin-state Selective HSQC-TOCSY:

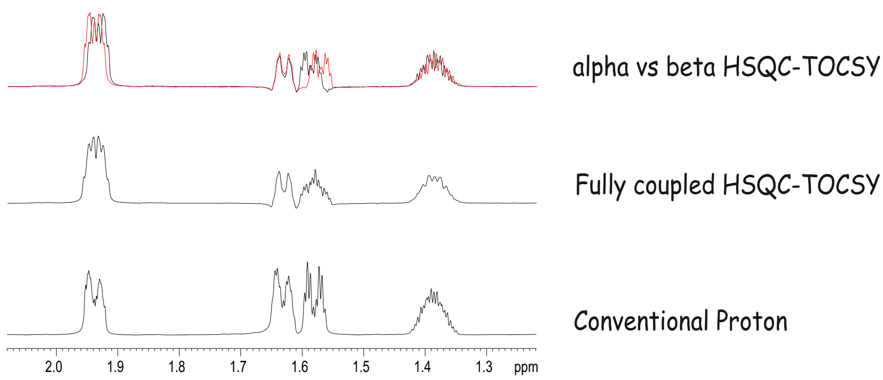
P. Nolis & T. Parella, J. Magn. Reson., 176, 15-26 (2005)



Processing:

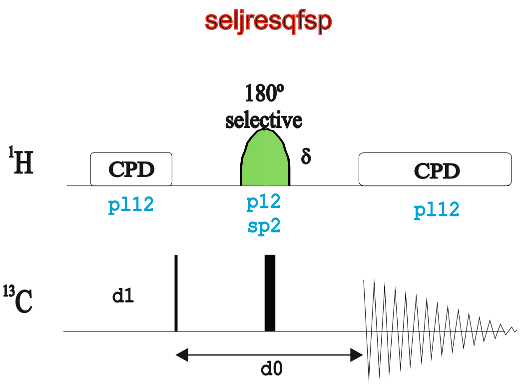
use AU-program **split [ipap 2]** to process data

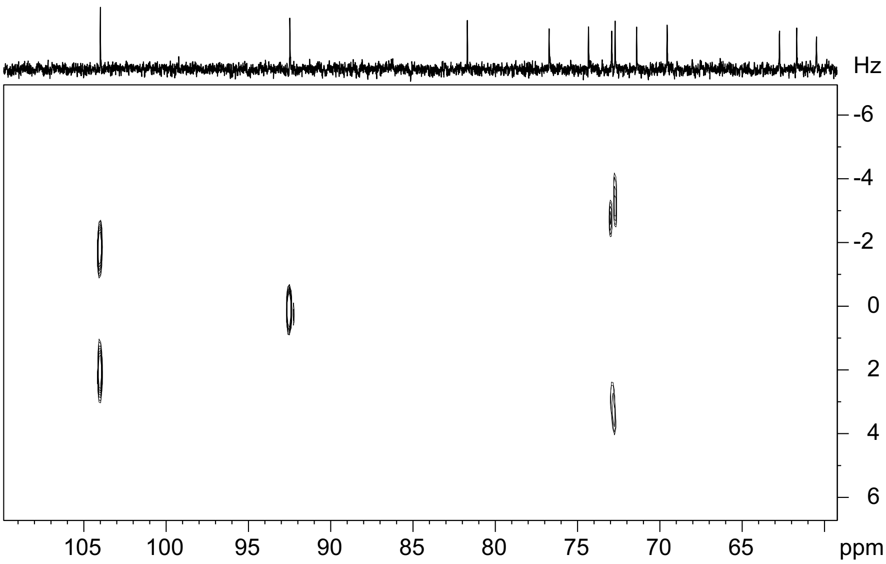




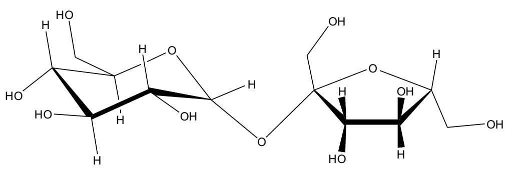
Selective J-Resolved

R. Freeman and A. Bax, JACS, 1082 (1982)





Selective J-Resolved spectrum after selective inversion of the H1 proton of sucrose



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2D ADEQUATE EXPERIMENTS

Experiment Description:

The ADEQUATE experiment allows to connect 1H and 13C separated by two- or more bonds. The basic pulse sequence consists of an INADEQUATE pulse scheme incorporated into an HSQC experiment. Several versions are available as a function how the experiment/delays are optimized. Thus, an 1,1-ADEQUATE experiment is equivalent to an HCC experiment in which the delays are optimized to 1J(CH) and 1J(CC). On the other hand, an 1,n-ADEQUATE experiment is optimized to 1J(CH) and nJ(CC).

The experiment is based on the selection of double-quantum 13C-13C coherences and therefore it can be considered of very low sensitivity.

Other related experiments: 2D INADEQUATE, 2D HMBC and 2D HSQC experiments

2D ADEQUATE Experiments

- **1,1-ADEQUATE:**

  Phase-sensitive 1,1 ADEQUATE using adiabatic pulses (**adeq11etgpsp**)
  Phase-sensitive w1-refocused 1,1 ADEQUATE using adiabatic pulse (**adeq11etgprdsp**)
  Phase-sensitive w1-refocused 1,1 ADEQUATE using adiabatic pulses with evolution of J(CC) (**adeq11etgprdsp.2**)

  Phase-sensitive w1-refocused 1,1 J-ADEQUATE using adiabatic pulses for measuring J(CC) (**adeq11etgpjcrdsp.2**)

- **1,n-ADEQUATE:**

  Phase-sensitive 1,n ADEQUATE (**adeq1netgp**)

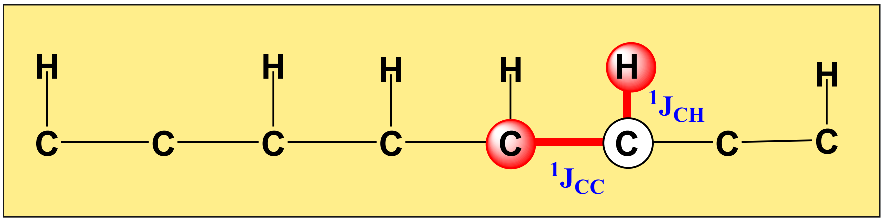
- **n,1-ADEQUATE:**

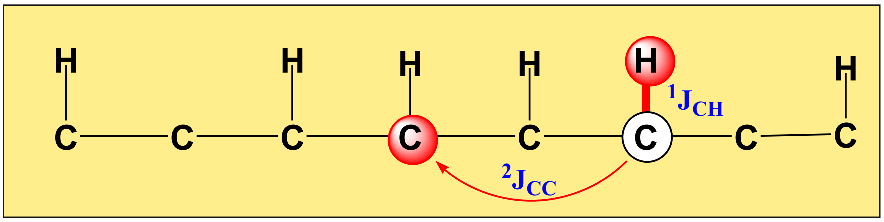
  Phase-sensitive n,1 ADEQUATE (**adeqn1etgp**)

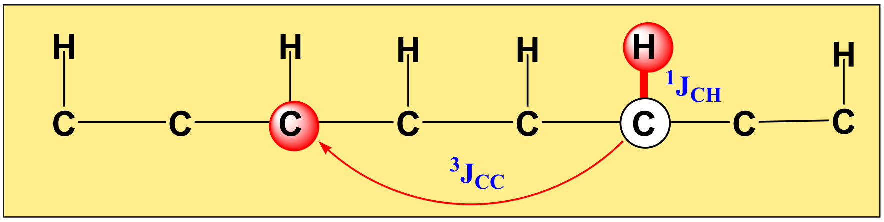
- **n,n-ADEQUATE:**

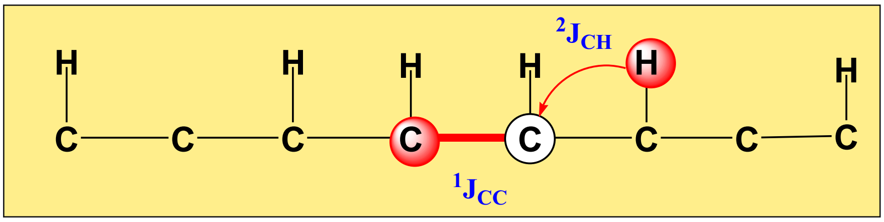
  Phase-sensitive n,n ADEQUATE (**adeqnnetgp**)

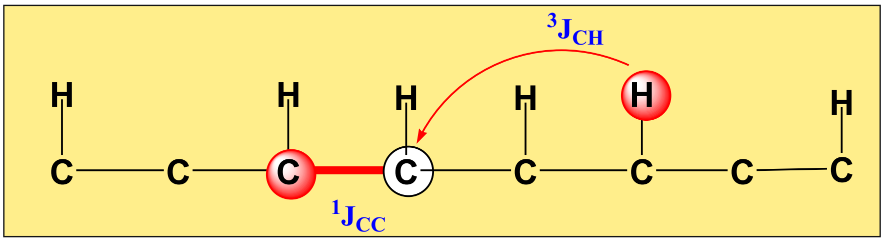
Also see:
2D INADEQUATE

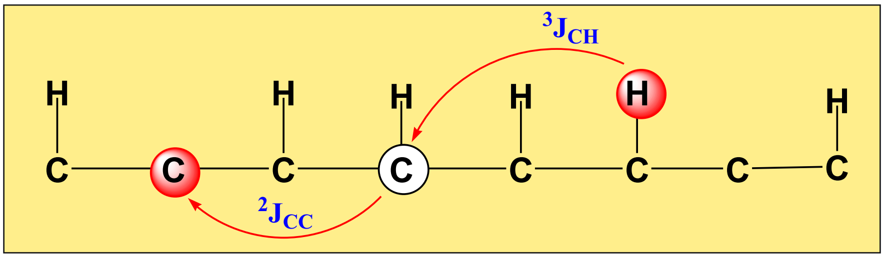

$^{1}J_{CH}$
$^{1}J_{CC}$

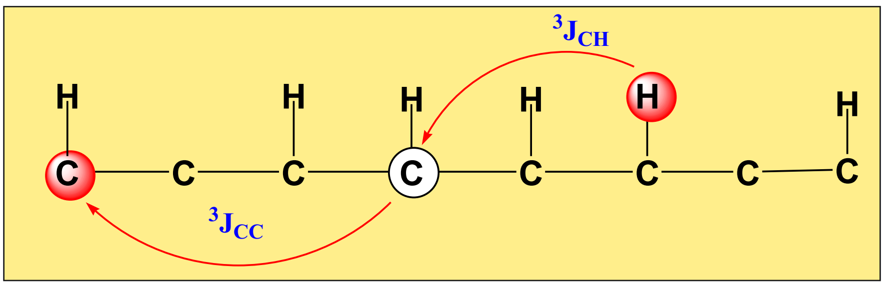

$^{1}J_{CH}$
$^{2}J_{CC}$


$^{1}J_{CH}$
$^{3}J_{CC}$

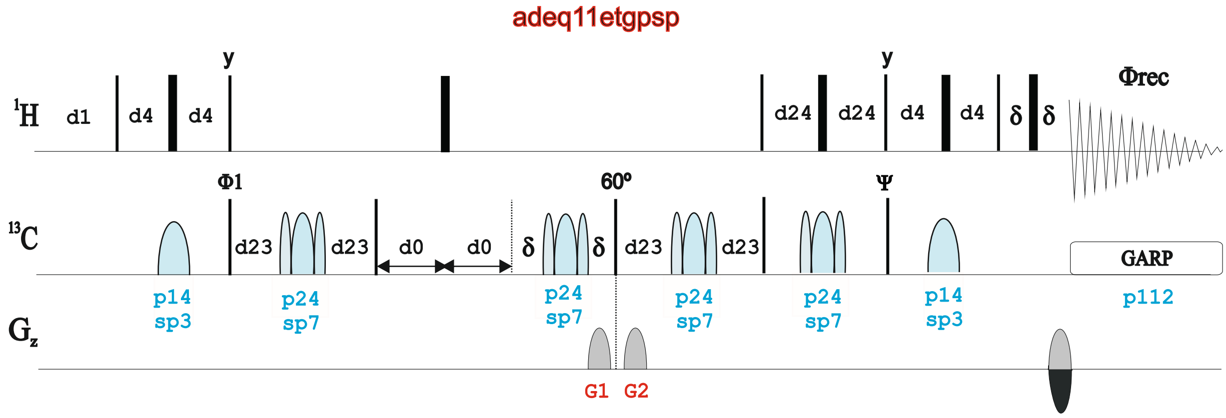

$^{2}J_{CH}$
$^{1}J_{CC}$

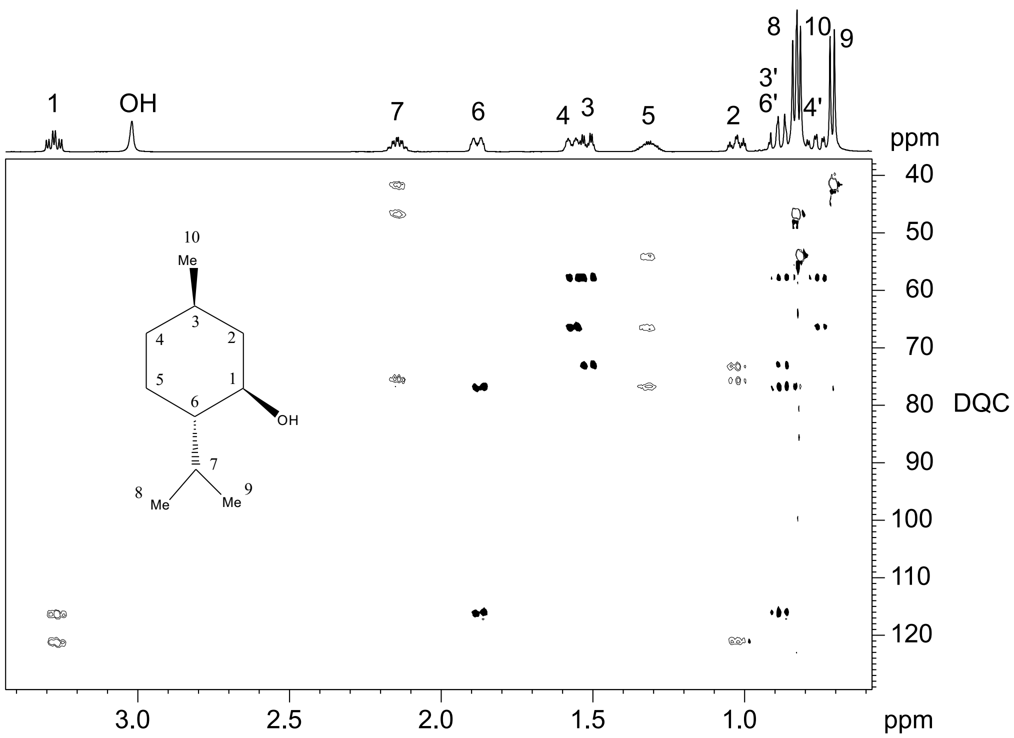

$^{3}J_{CH}$
$^{1}J_{CC}$


$^{3}J_{CH}$
$^{2}J_{CC}$


$^{3}J_{CH}$
$^{3}J_{CC}$

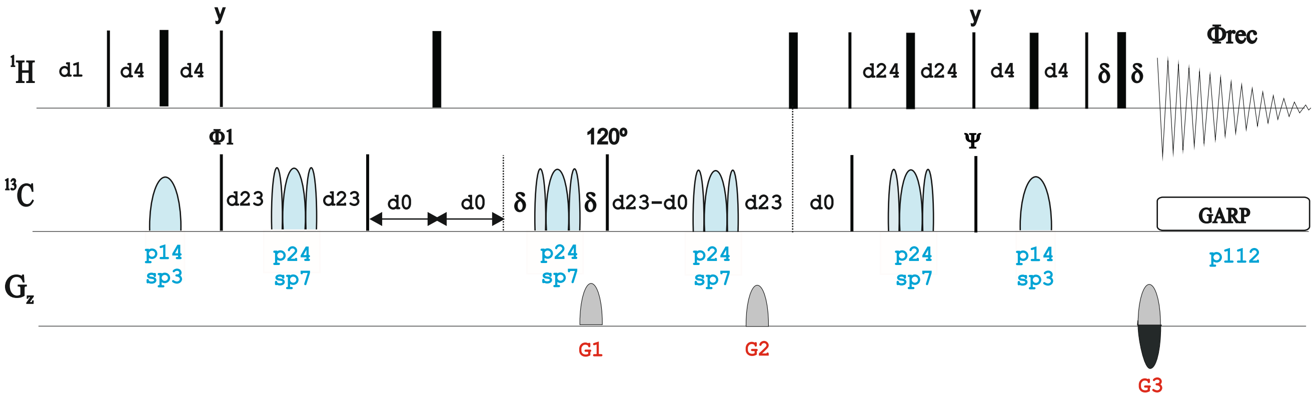
1. B. Reif, M. Koeck, R. Kerssebaum, H. Kang, W. Fenical & C. Griesinger J. Magn. Reson. A118, 282-285 (1996).
2. B. Reif, M. Koeck, R. Kerssebaum, J. Schleucher & C. Griesinger
J. Magn. Reson. B112, 295-301 (1996)
3. M. Koeck, R. Kerssebaum & W. Bermel, Magn. Reson. Chem. 41, 65-69 (2003)

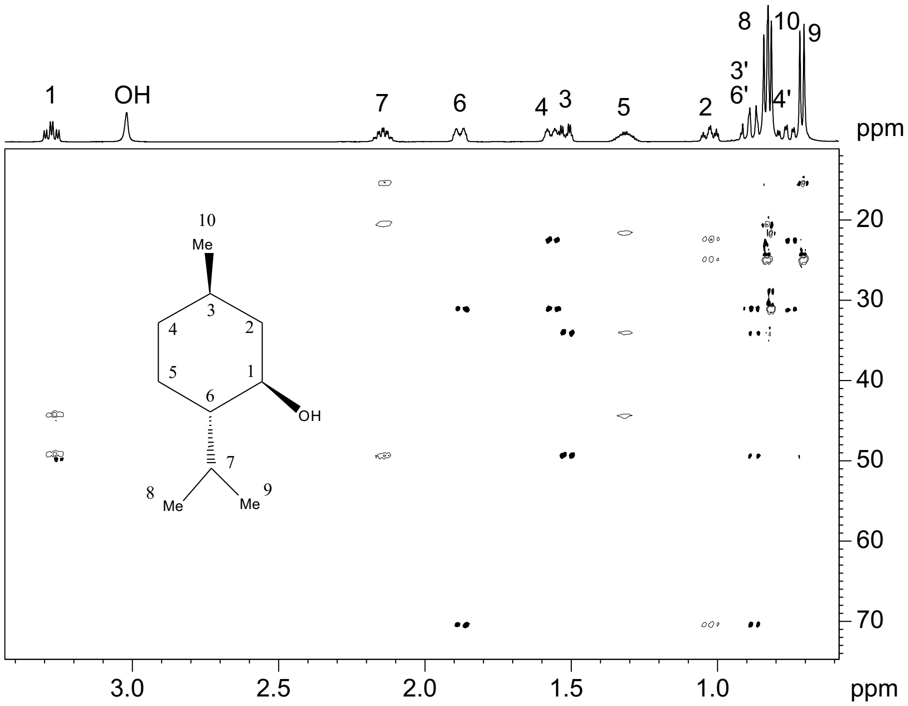




2D 1,1-ADEQUATE spectrum of menthol

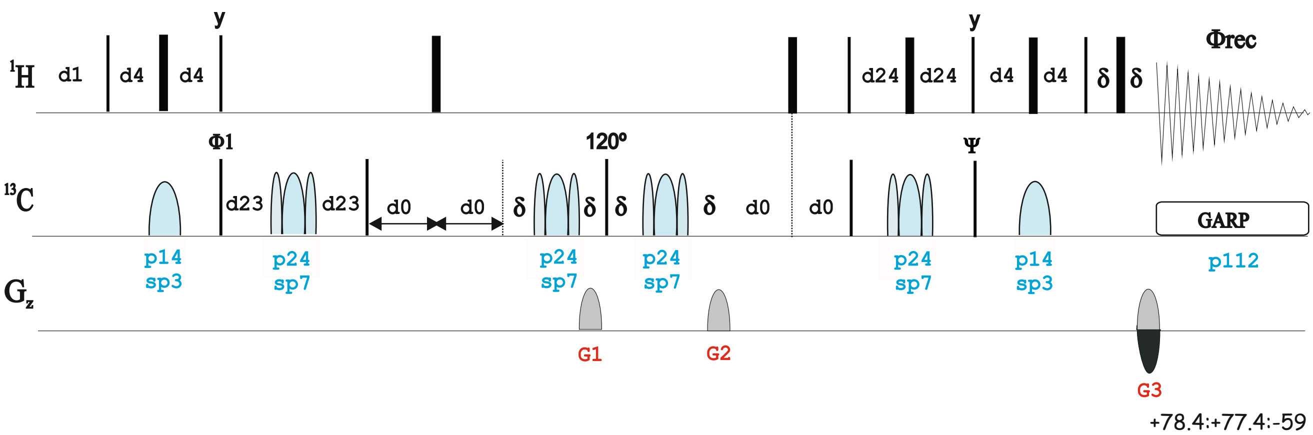
adeq11etgprdsp





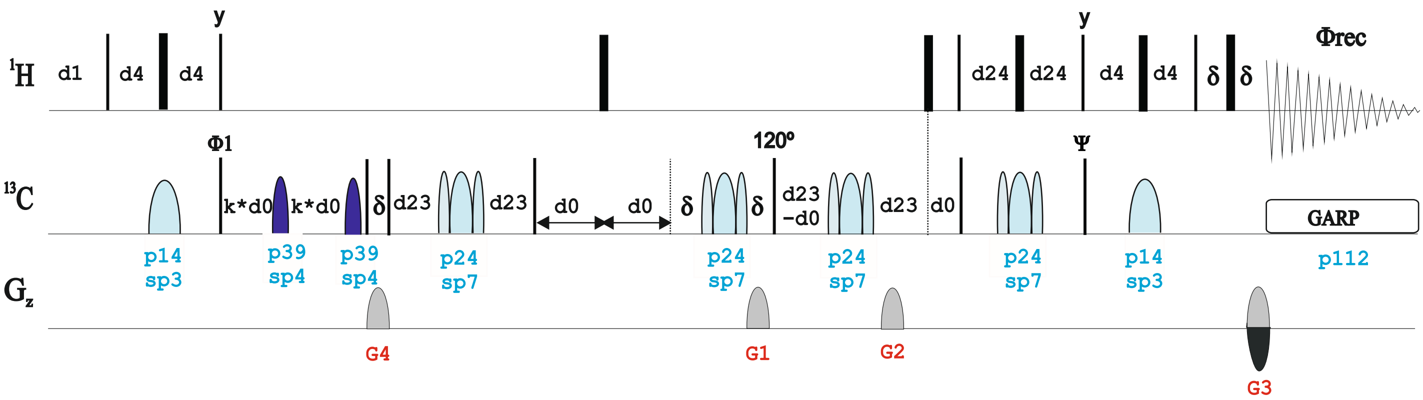
2D Refocused 1,1-ADEQUATE spectrum of menthol

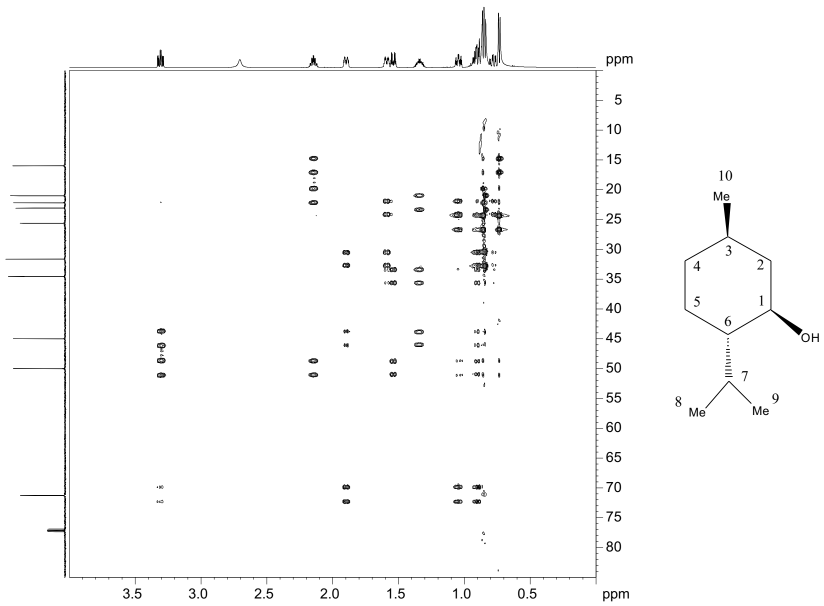
adeq11etgprdsp.2



J-Adequate:
C.M. Thiele & W. Bermel, Magn. Reson. Chem. 45, 889-894 (2007)
K.E. Kover, P. Forgo, J. Magn. Reson. 166, 47-52 (2004)

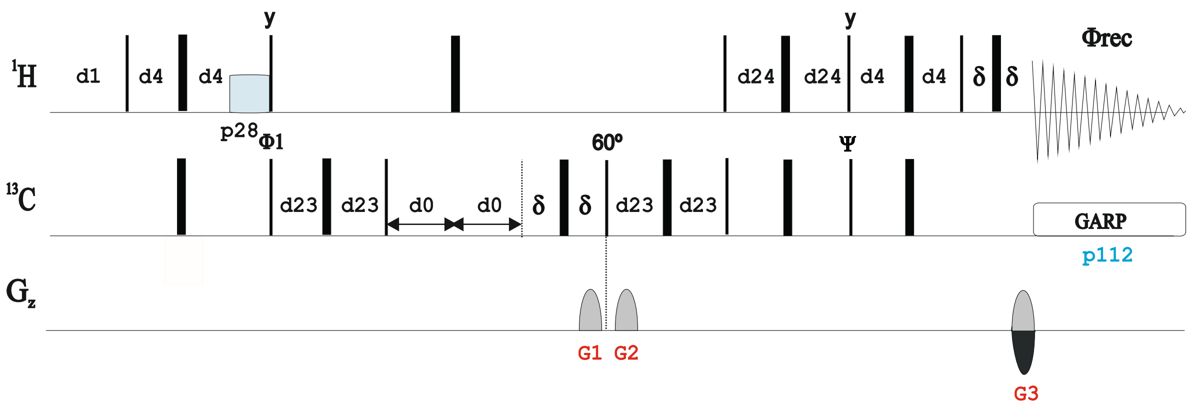
adeq11etgpjcrdsp



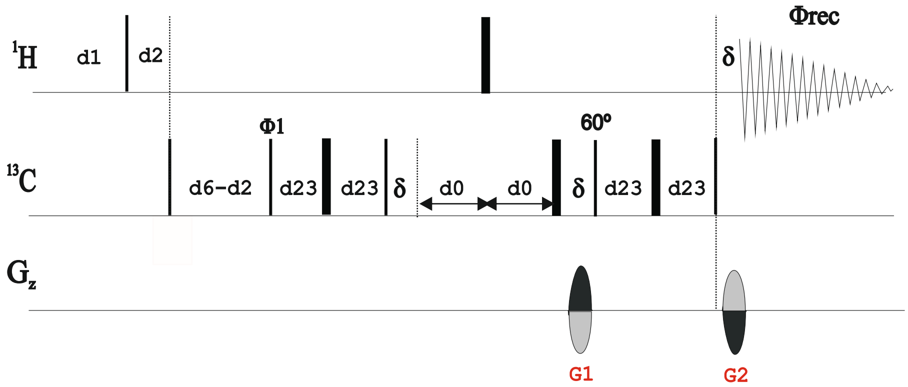


2D 1,1-J-ADEQUATE spectrum of menthol

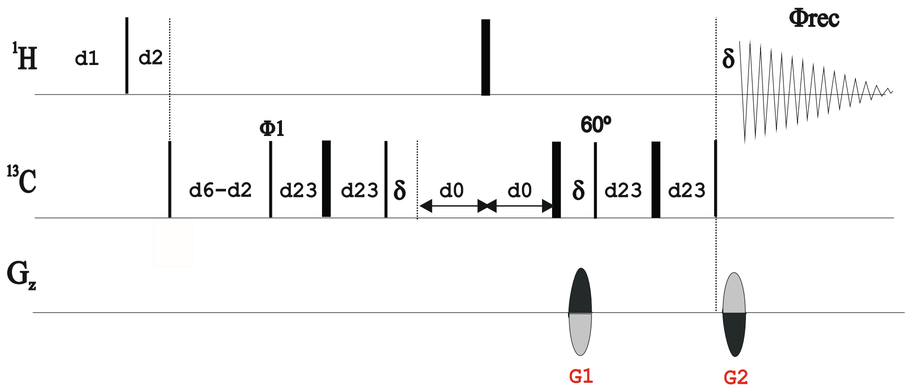
adeq1netgp



adeqn1etgp



adeqnnetgp



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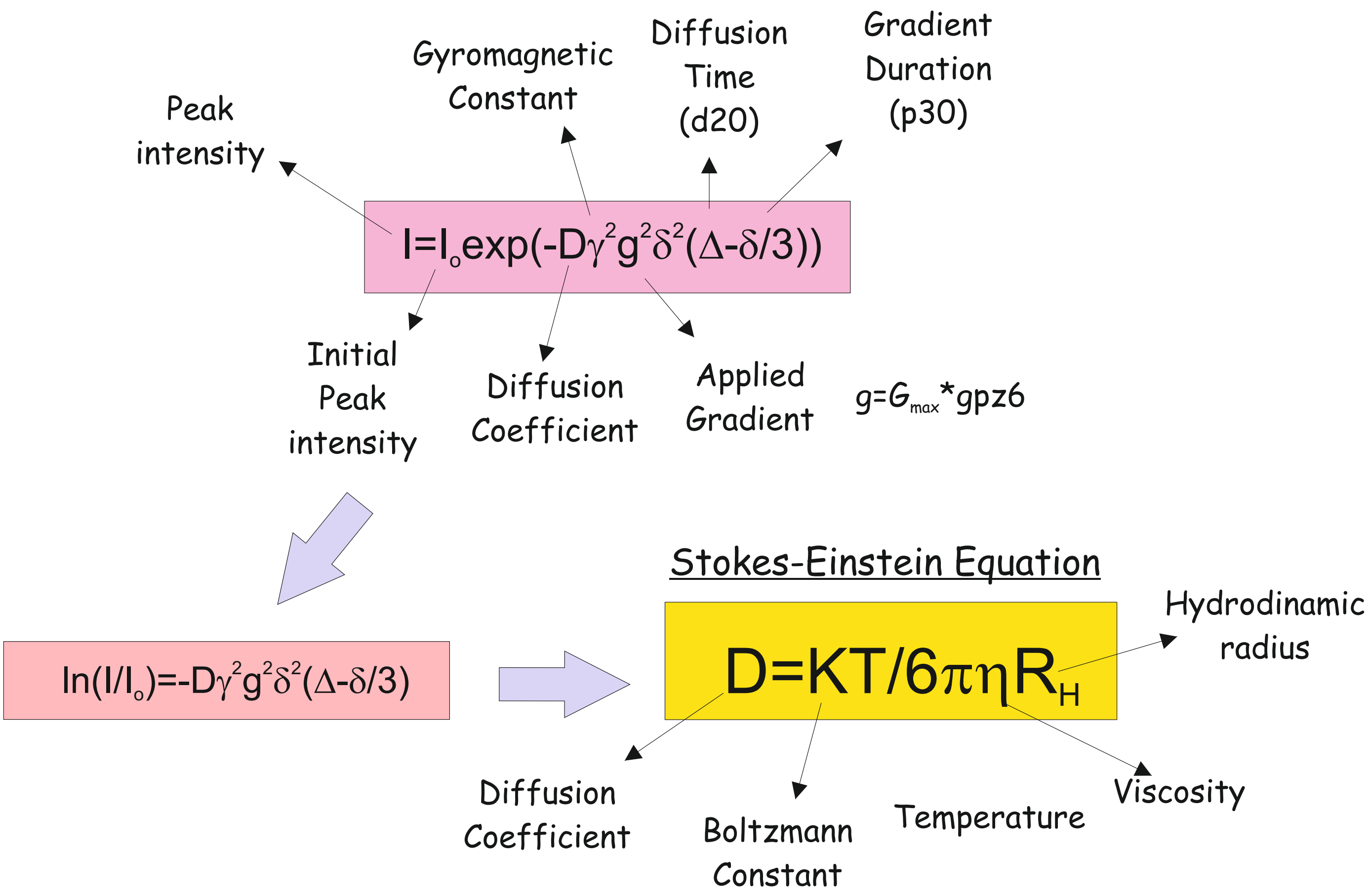
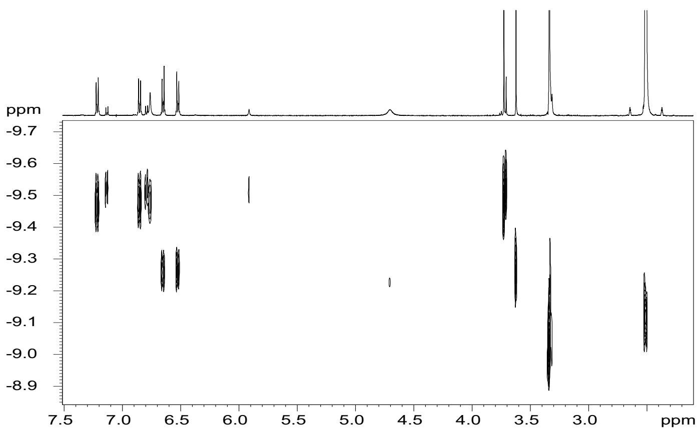
DIFFUSION/DOSY
EXPERIMENTS



DOSY Spectrum:
Chemical Shift vs Diffusion Coefficient



A Decription of setting up and running Diffusion/DOSY experiments can be found in the **DOSY Manual** (see **Help** menu in Topspin)

DOSY/Diffusion

- **Conventional 1D:**

1D Stimulated Echo experiment (STE) (**stegp1s1d**)
1D Stimulated Echo experiment (STE) using bipolar gradients (**stebpgp1s1d**)
1D LED experiment (**ledgp2s1d**)
1D LED experiment using bipolar gradients (**ledbpgp2s1d**)
1D LED experiment using bipolar gradients and presaturation (**ledbpgppr2s1d**)
1D Double-Stimulated Echo Experiment (DSTE) (**dstegp3s1d**)
1D Double-Stimulated Echo Experiment (DSTE) using bipolar gradients (**dstebpgp3s1d**)

1D Stimulated Echo experiment using bipolar gradients and WATERGATE (**stebpgp1s191d**)

1D STE-INEPT experiment (**stebpgpin1s1d**)

- **2D DOSY maps:**

2D Stimulated Echo experiment (STE) (**stegp1s**)
2D Stimulated Echo experiment using bipolar gradients (**stebpgp1s**)

2D Double-Stimulated Echo Experiment (DSTE) (**dstegp3s**)
2D Double-Stimulated Echo Experiment (DSTE) using bipolar gradients (**dstebpgp3s**)
2D LED experiment (**ledgp2s**)
2D LED experiment using bipolar gradients (**ledbpgp2s**)
2D LED experiment using bipolar gradients and presaturation (**ledbpgppr2s**)

2D Stimulated Echo experiment using bipolar gradients and WATERGATE (**stebpgp1s19**)

2D STE-INEPT experiment (**stebpgpin1s**)

- **2D & 3D DOSY related experiments:**

3D DOSY-COSY using LED with bipolar gradients (**ledbpgpco2s3d**)

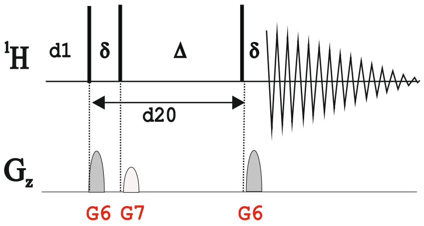
2D DOSY-TOCSY with LED using bipolar gradients (**ledbpgpml2s2d**)
2D DOSY-TOCSY with LED using bipolar gradients and WATERGATE (**ledbpgpml2s192d**)
3D DOSY-TOCSY using LED with bipolar gradients (**ledbpgpml2s3d**)

3D DOSY-NOESY using LED with bipolar gradients (**ledbpgpno2s3d**)

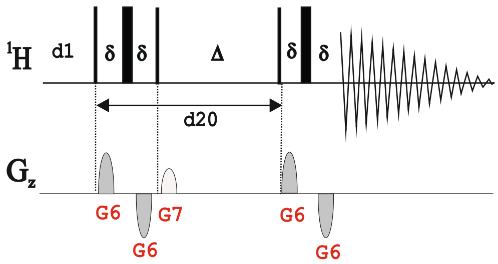
Automated DOSY Data Acquisition:
Run DOSY experiment with "**dosy**" and answer the questions.

Automated DOSY Data Processing:
Transform the n 1D spectra with **xf2**
Phase the first ser and apply the phase correction with **xf2p**
Baseline correction with **abs2**
Set diffussion parameters with **setdiffparm**
Transform fiddusion dimension with **dosy2d**

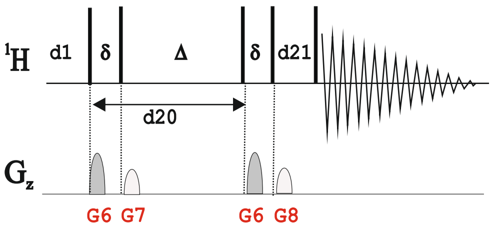
stegp1s
stegp1s1d



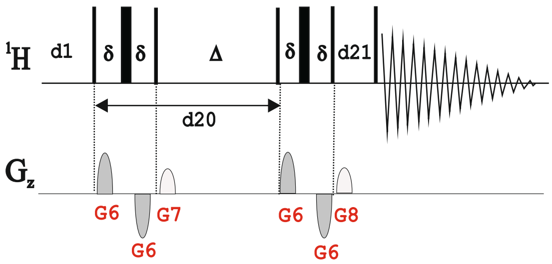
stebpgp1s
stebpgp1s1d



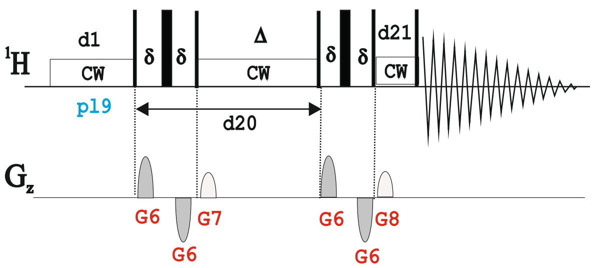
ledgp2s
ledgp2s1d



ledbpgp2s
ledbpgp2s1d

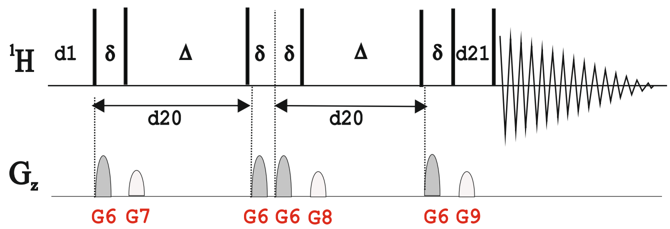


ledbpgppr2s
ledbpgppr2s1d



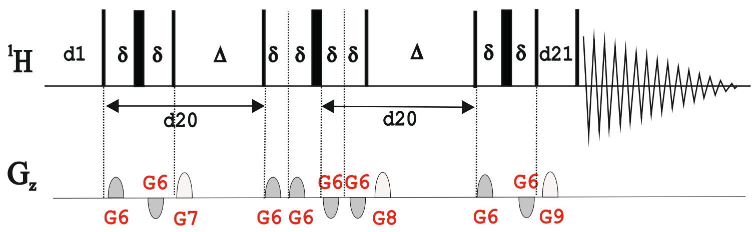
dstegp3s

dstegp3s1d



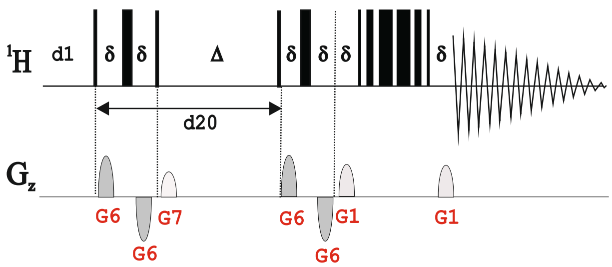
dstebpgp3s1d

dstebpgp3s



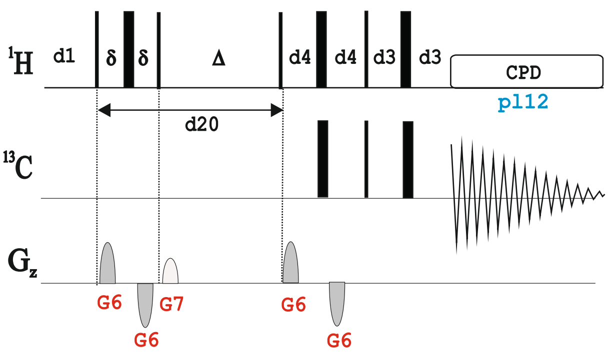
stebpgp1s19

stebpgp1s191d



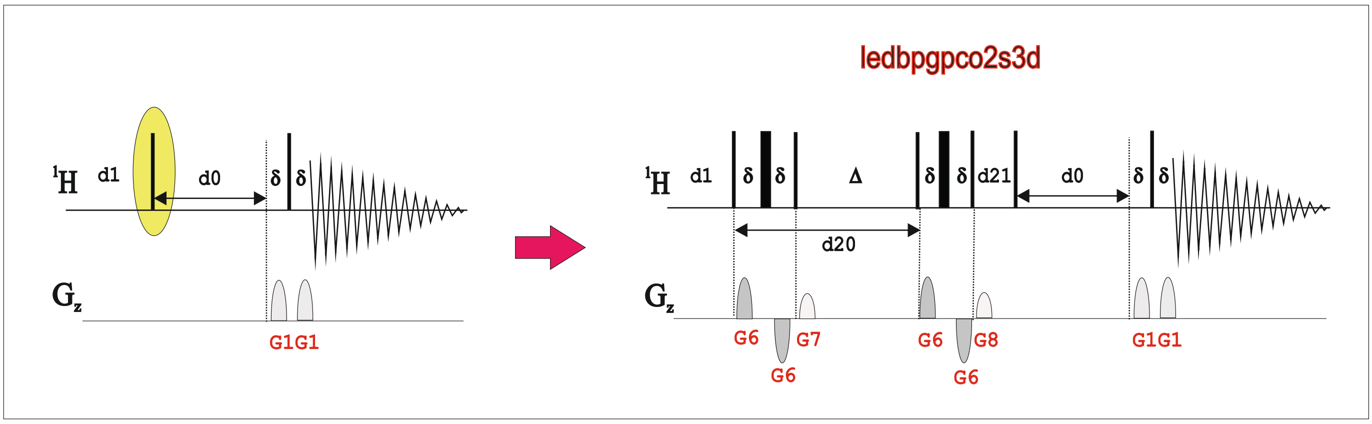
stebpgpin1s1d

stebpgpin1s

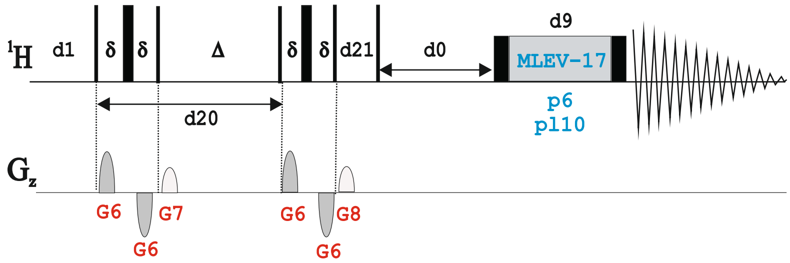


NMR Building Block: A Diffusion Filter.

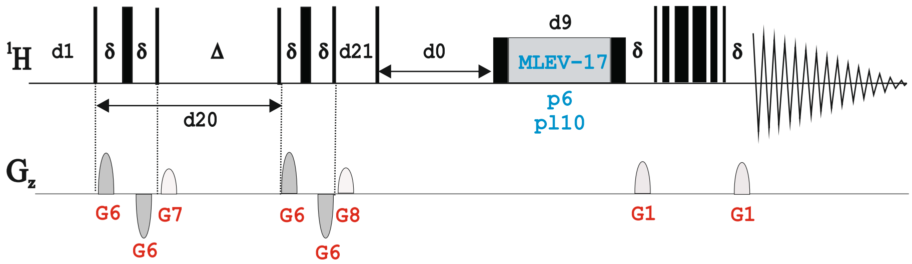
Any 1D Diffusion Experiment (STE,DSTE, LED or LEDBP) can be used as a diffusion filter in nD Experiments.
In the example, the initial read 90 pulse in a COSY experiment is replaced by a LEDBP building block



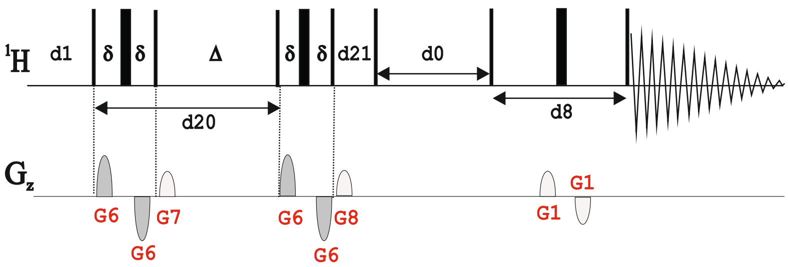
ledbpgpml2s3d
ledbpgpml2s2d



ledbpgpml2s192d



ledbpgpno3s3d



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1D & 2D SATURATION TRANSFER DIFFERENCE (STD) EXPERIMENTS

Saturation Transfer Difference (STD) Experiments

- **1D STD:**

1D STD **(stddiff)**
1D STD with spoil **(stddiff.2)**
1D STD with spoil and T2 filter **(stddiff.3)**

- **1D STD with solvent suppression:**

1D STD using 3-9-19 WATERGATE **(stddiffgp19)**
1D STD with spoil using 3-9-19 WATERGATE **(stddiffgp19.2)**
1D STD with spoil and T2 filter using 3-9-19 WATERGATE **(stddiffgp19.3)**

1D STD using excitation scultping **(stddiffesgp)**
1D STD with spoil using excitation scultping **(stddiffesgp.2)**
1D STD with spoil and T2 filter using excitation scultping **(stddiffesgp.3)**

- **2D STD-TOCSY:**

2D STD-TOCSY **(stdmlevph)**
2D STD-TOCSY using 3-9-19 WATERGATE **(stdmlevgpph19)**
2D STD-TOCSY using excitation sculpting **(stdmlevesgpph)**

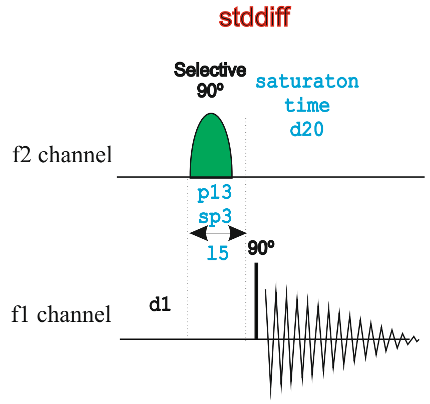
- **2D STD-NOESY:**

2D STD-NOESY with T2 filter in F2 **(stdnoesygpph)**
2D STD-NOESY with T2 filter in F1 and F2 **(stdnoesygpph.2)**
2D STD-NOESY using 3-9-19 WATERGATE with T2 filter in F2 **(stdnoesygpph19)**
2D STD-NOESY using 3-9-19 WATERGATE with T2 filter in F1 and F2 **(stdnoesygpph19.2)**
2D STD-NOESY using excitation sculpting with T2 filter in F2 **(stdnoesyesgpph)**
2D STD-NOESY using excitation sculpting with T2 filter in F1 and F2 **(stdnoesyesgpph.2)**

- **2D STD-HSQC:**

2D STD-HSQC using echo-antiecho **(stdhsqcetgpsp)**
2D STD-HSQC with sensitivity-improvement **(stdhsqcetgpsisp)**

References:
1. M. Mayer & B. Meyer, Angew. Chem. Int. Ed. 38, 1784-1788 (1999)
2. M. Mayer & B. Meyer, Angew. Chem. 111, 1902-1906 (1999)



NMR Element: Basic Saturation Loop for STD

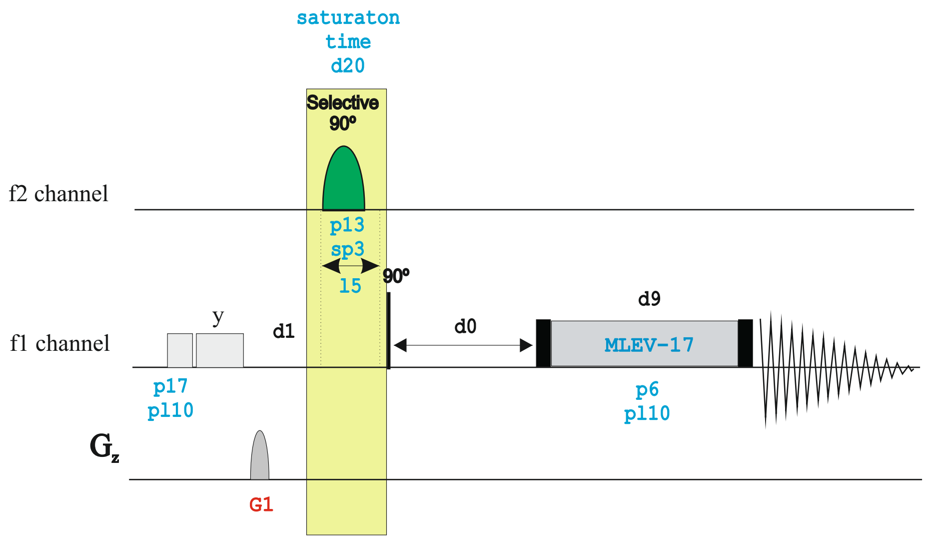
```
   ...
   6 (p13:sp13 ph1):f2
     4u
     lo to 6 times l5
   ....

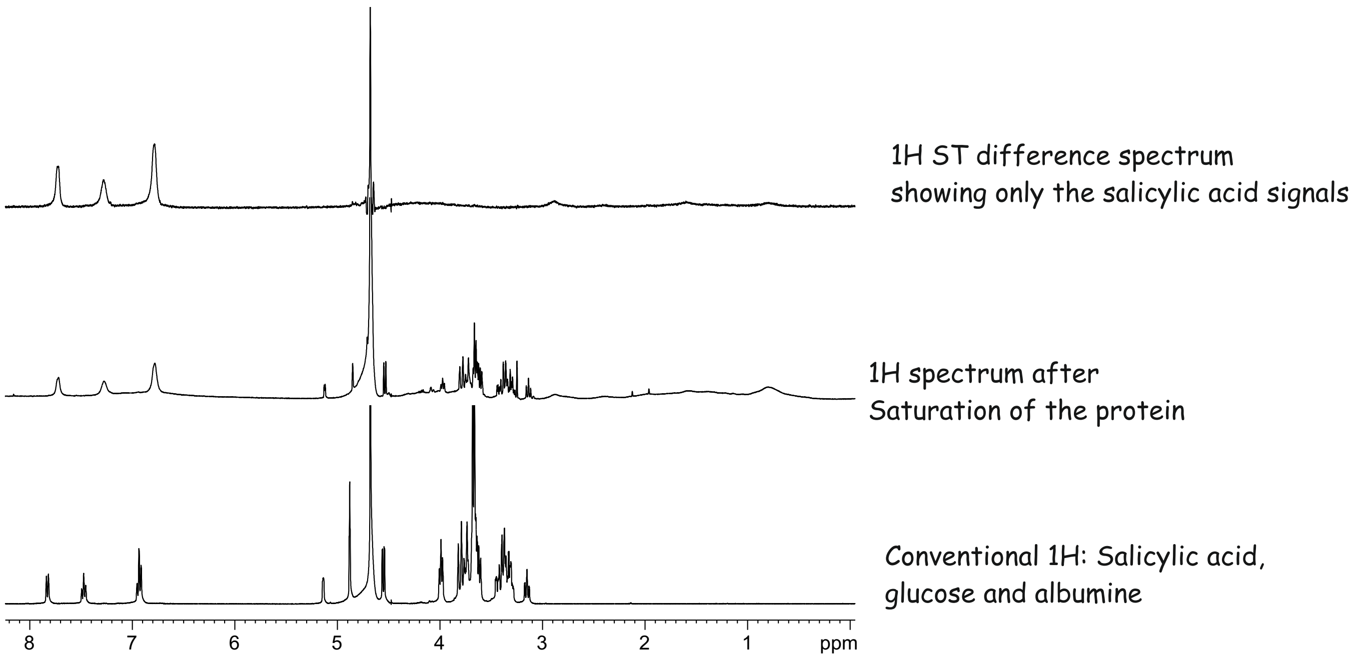


;sp13: f2 channel - shaped pulse  for saturation          [40 - 60 dB]
;p13: f2 channel -  shaped pulse for saturation           [50 msec]
;d20: saturation time
;l4: l4 = number of averages = (total number of scans) / NS
;l5: loop for saturation: p13 * l5 = saturation time
;td1: number of experiments
;NBL: NBL = number of irradiation frequencies

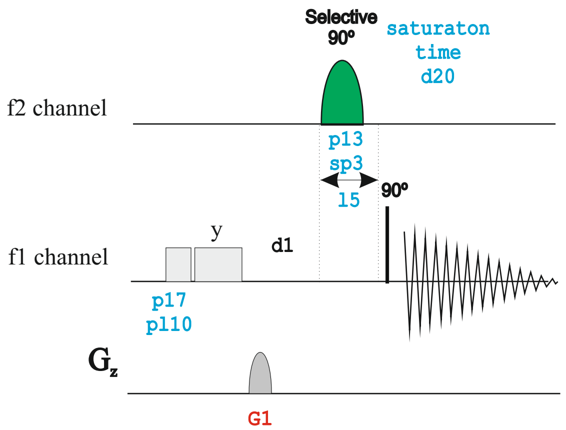
;define FQ2LIST (irradiation frequencies)
;                (list has to be stored in "/u/exp/stan/nmr/lists/f1")
```

A STD Element can beincorporated just prior the read 90 pulse in any nD Experiments.
In the example, the STD element is incorporated in a TOCSY experiment.

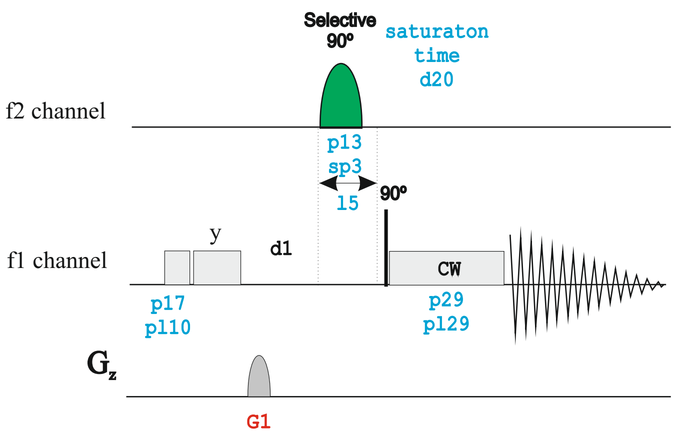




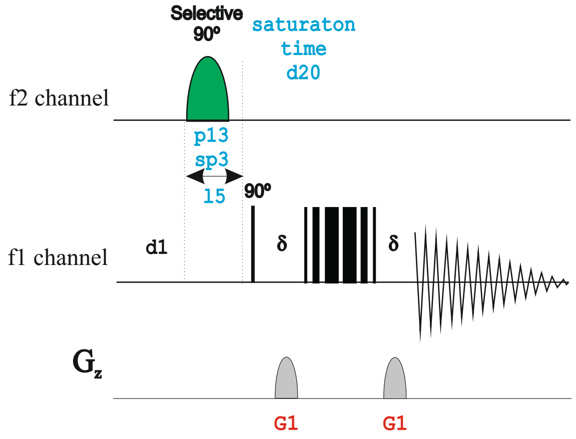
stddiff.2



stddiff.3



stddiffgp19



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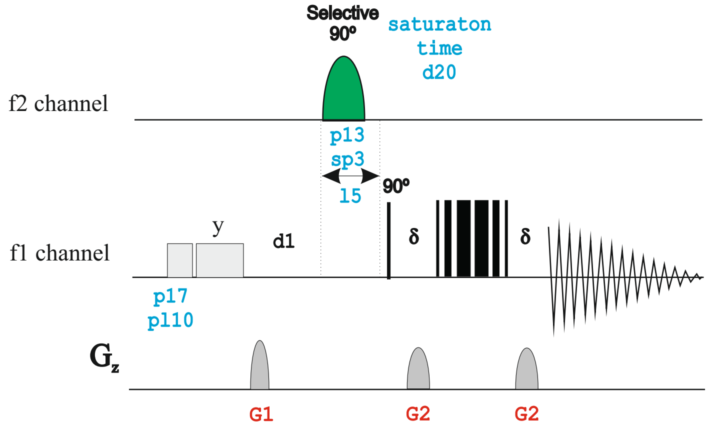

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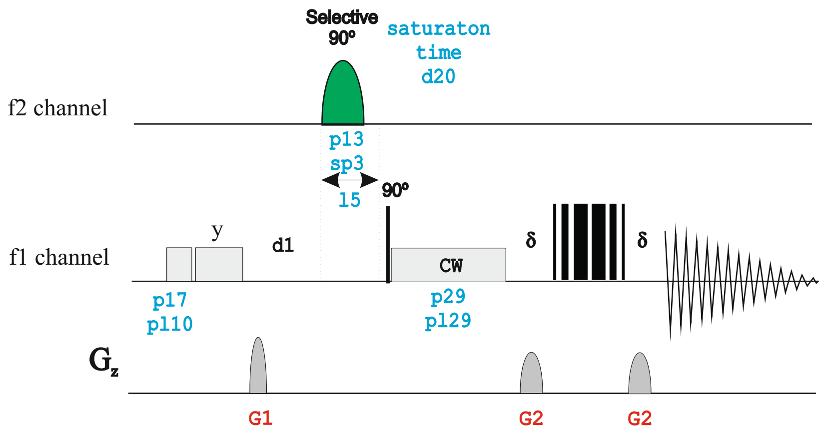
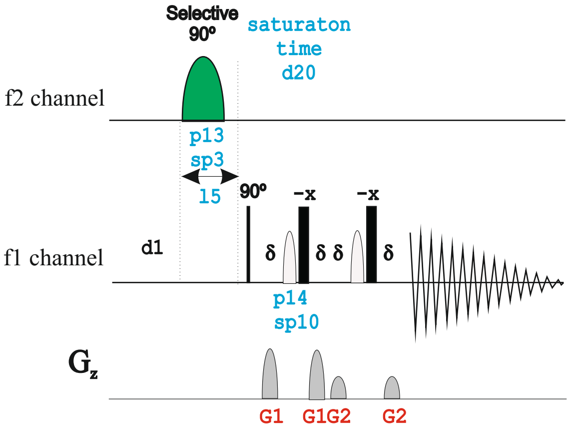


stddiffesgp



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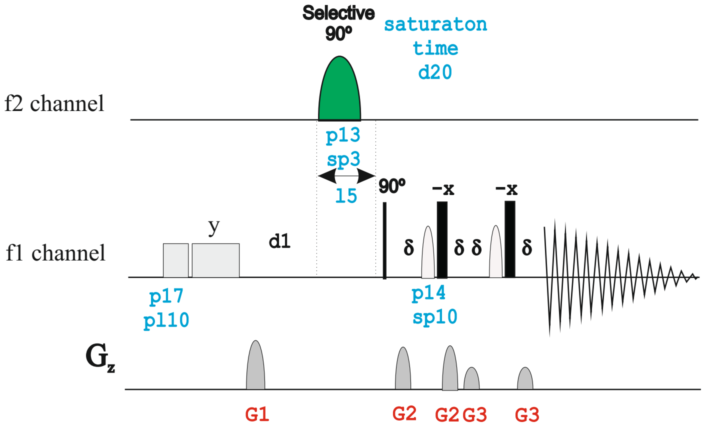
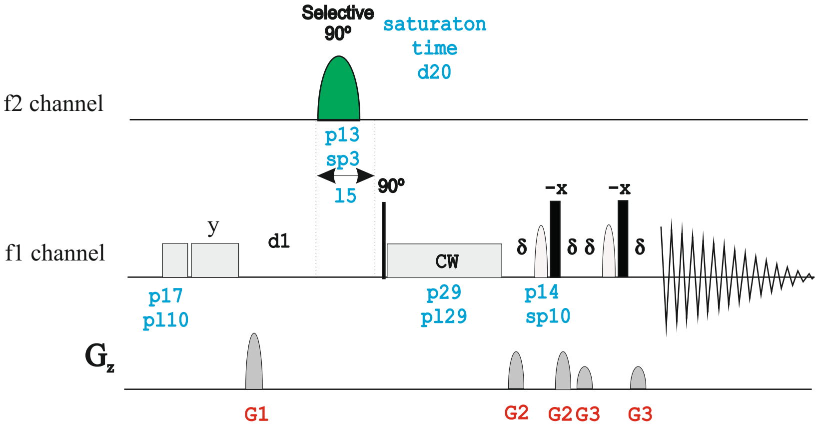
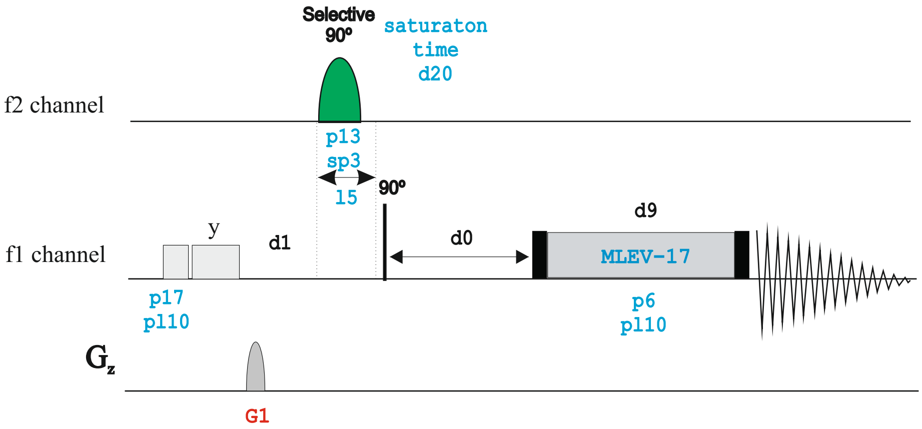


stddiffesgp.3



stdmlevph



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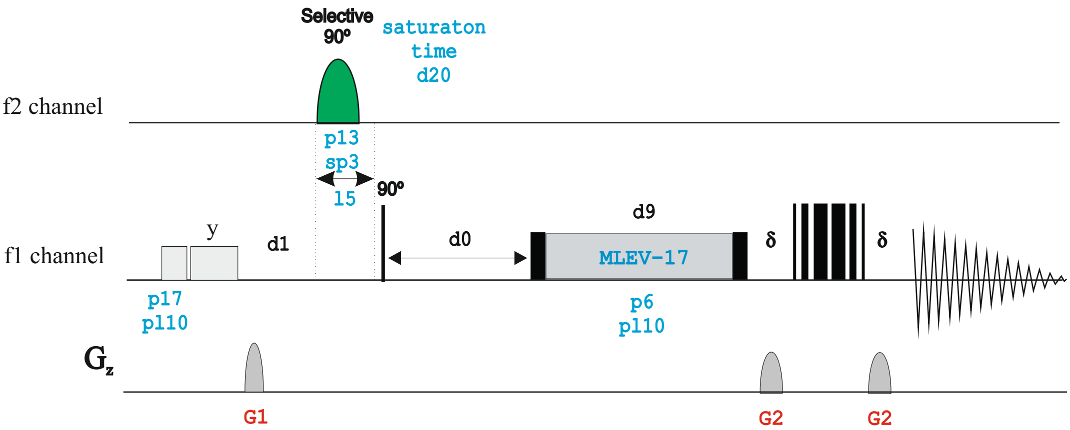

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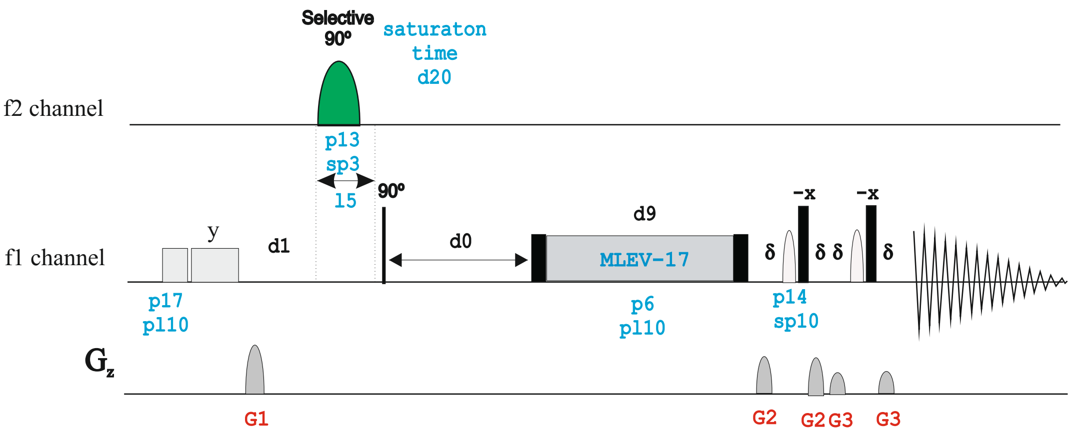
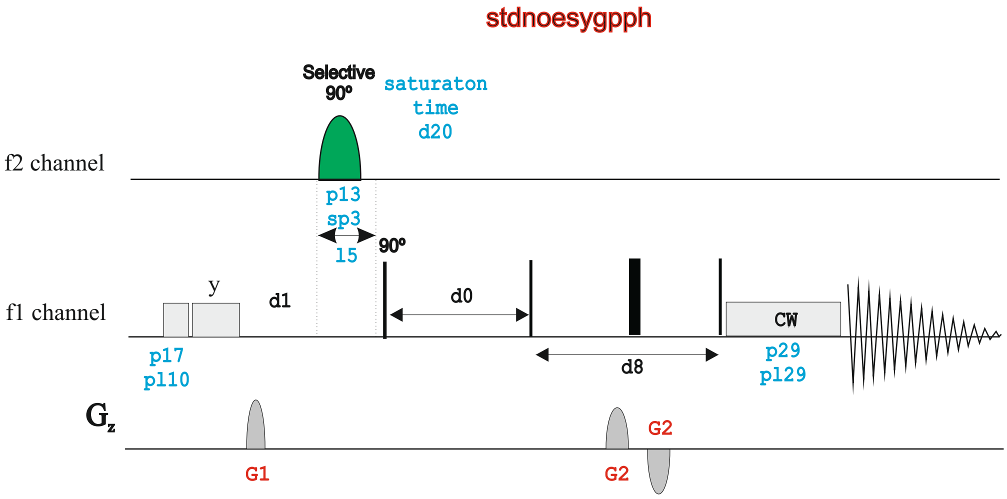
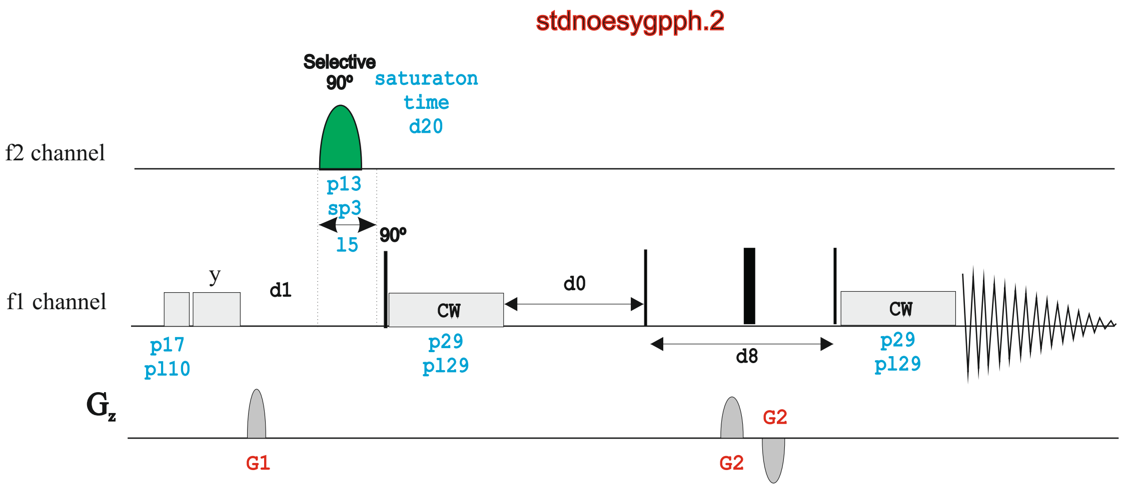



stdnoesygpph
Selective
90º
saturaton
time
d20
f2 channel
p13
sp3
15
90º
y
d1
d0
f1 channel
CW
p17
p110
d8
p29
p129
Gz
G1
G2
G2


stdnoesygpph.2
Selective
90º
saturaton
time
d20
f2 channel
p13
sp3
15
90º
y
d1
d0
f1 channel
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Gz
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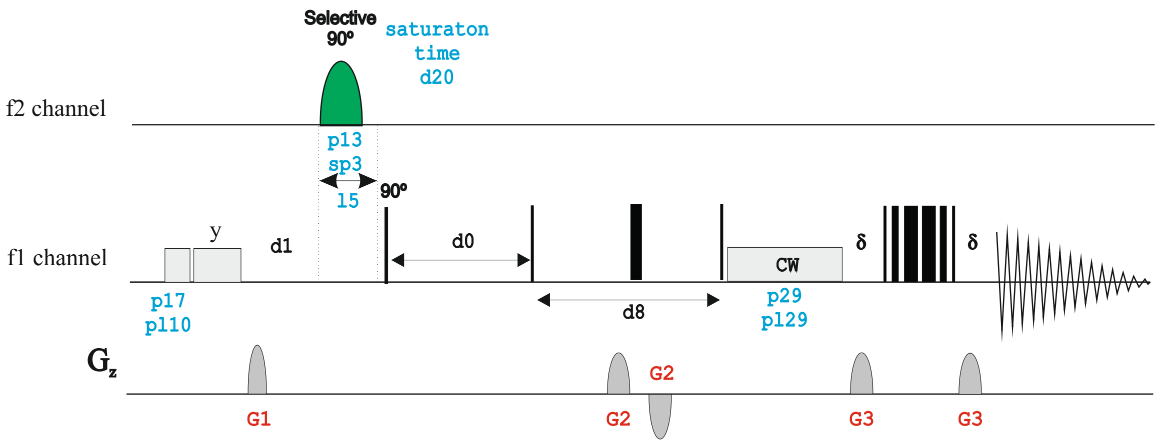
stdnoesygpph19



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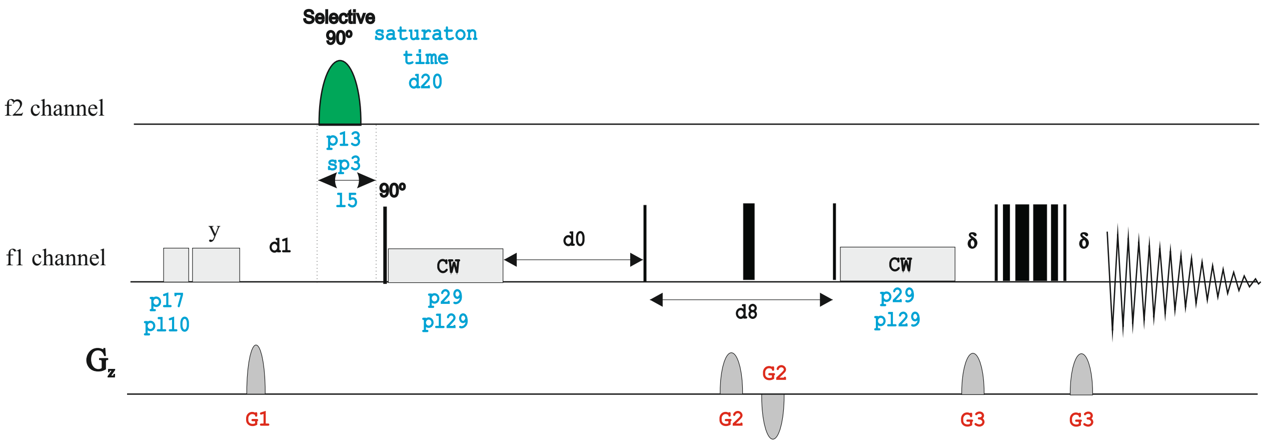

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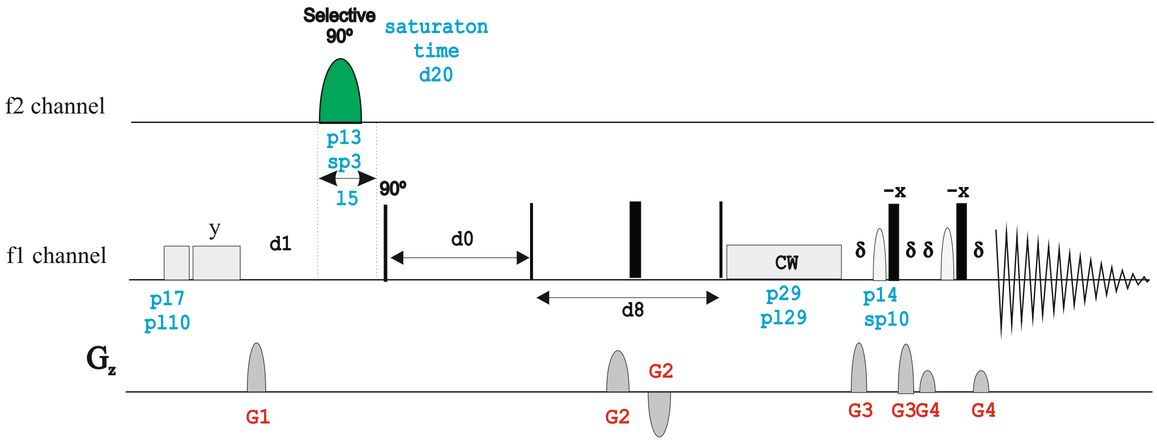
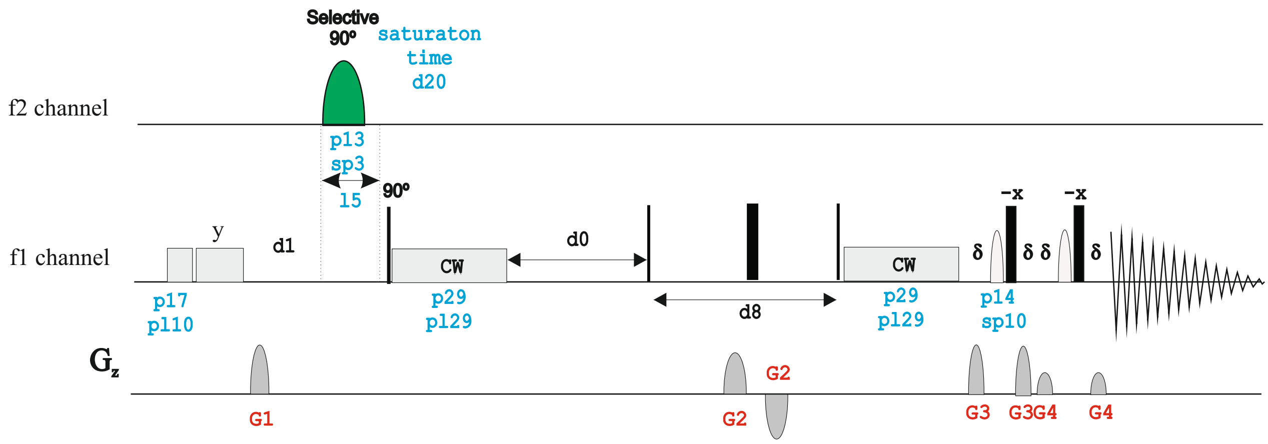
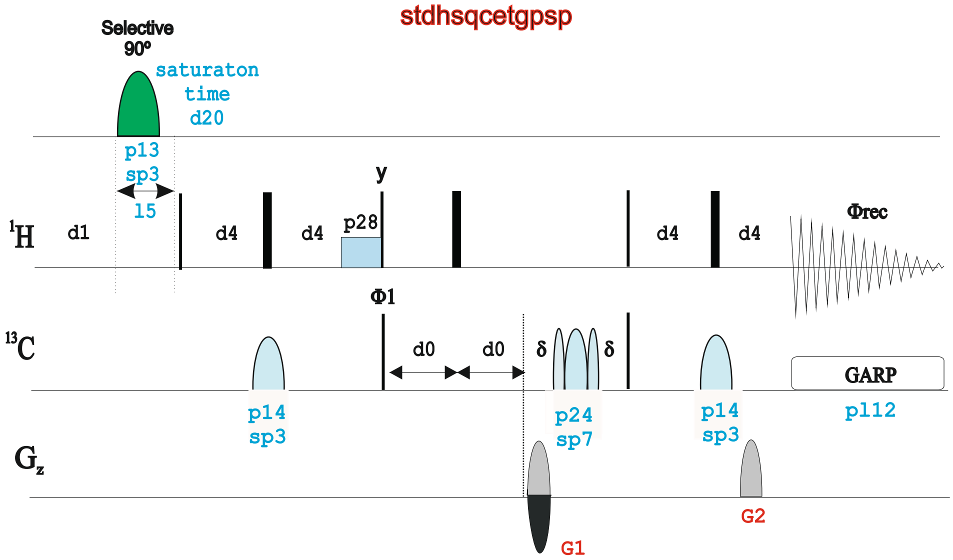


stdnoesyesgpph.2



stdhsqcetgpsp



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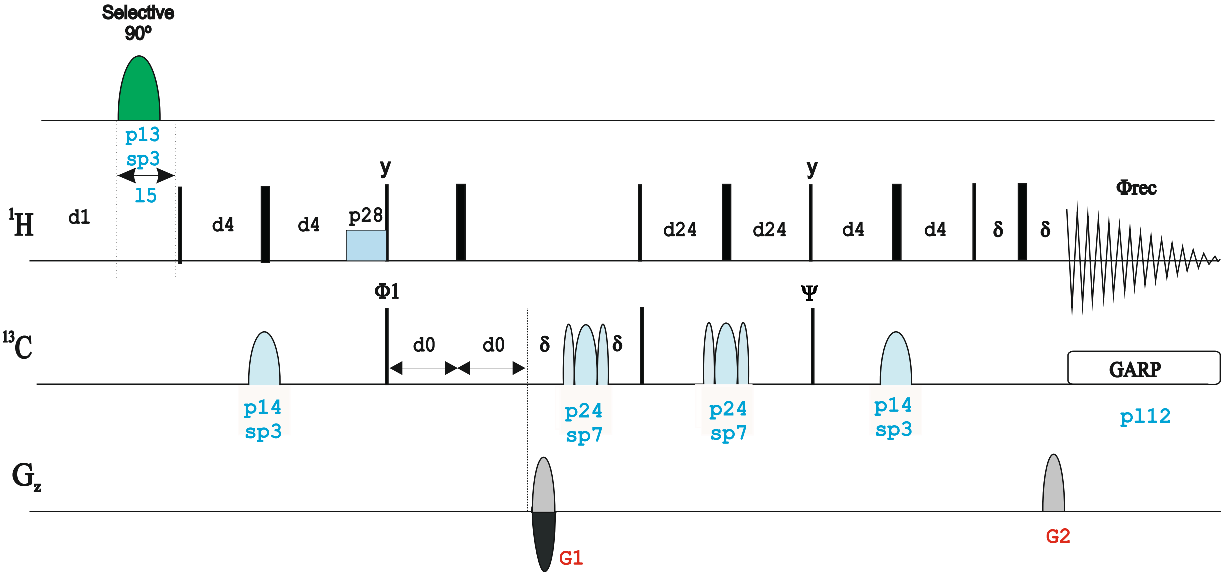

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CLEANEX EXPERIMENTS

CLEANEX: Clean Exchange Spectroscopy.
Water-selective experiments to study exchange processes

References:

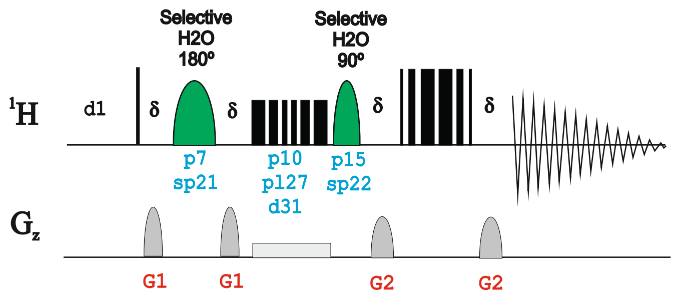
1. T.L. Hwang, S. Mori, A.J. Shaka & P.C.M. van Zijl, J.Am. Chem. Soc. 119, 6203-6204 (1997)
2. T.-L. Hwang & A.J. Shaka, J. Magn. Reson., Series A 112 275-279 (1995)

CLEANEX Experiments

- 1D CLEANEX using 3-9-19 WATERGATE **(zgcxgp19)**
- 1D CLEANEX using excitation sculpting **(zgcxesgp)**

- 2D CLEANEX-Fast HSQC using 3-9-19 WATERGATE **(fhsqccxf3gpph)**
- 2D CLEANEX-TROSY using 3-9-19 WATERGATE **(trosycxf3gpphsi19)**

zgcxgp19



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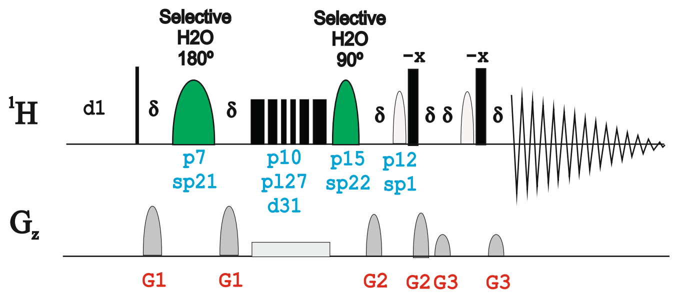
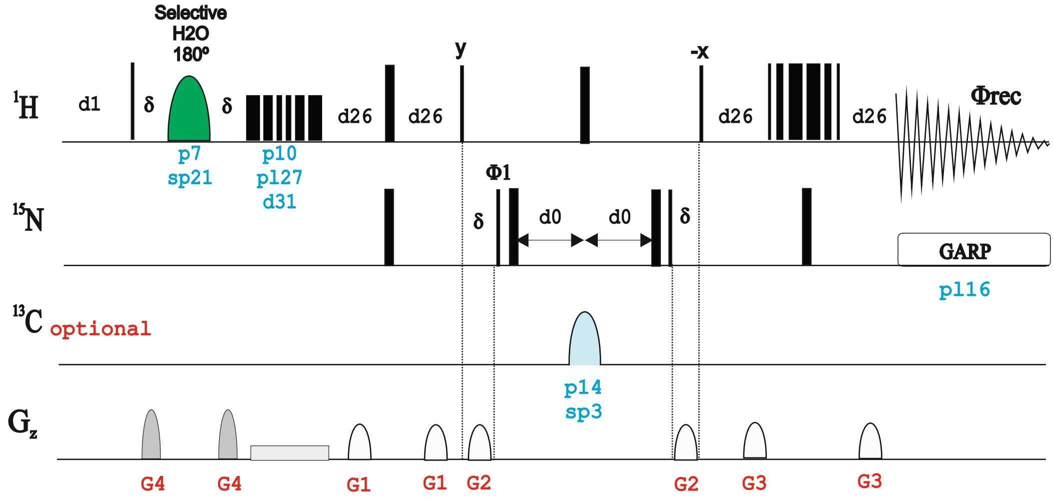
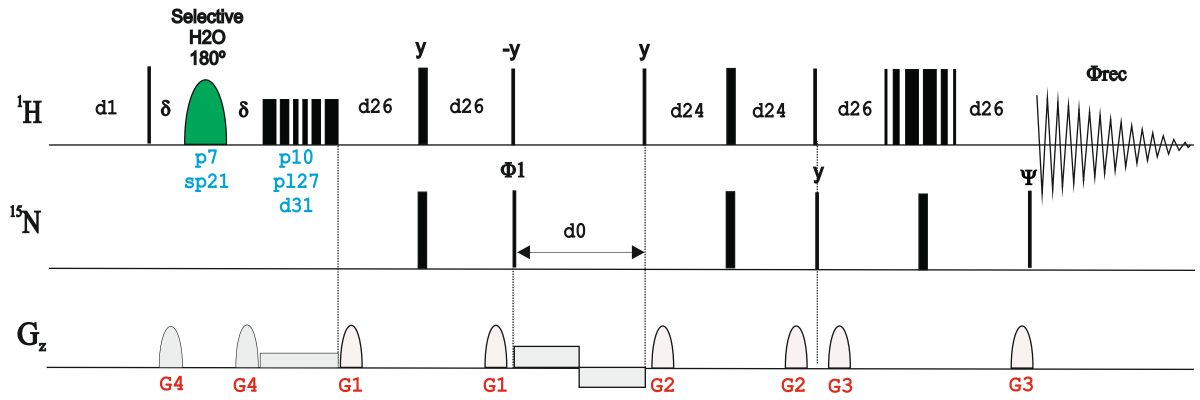


T.L. Hwang, P.C.M. van Zijl & S. Mori, J. Biomol. NMR 11, 221-226 (1998)

fhsqccxf3gpph



trosycxf3gpphsi19



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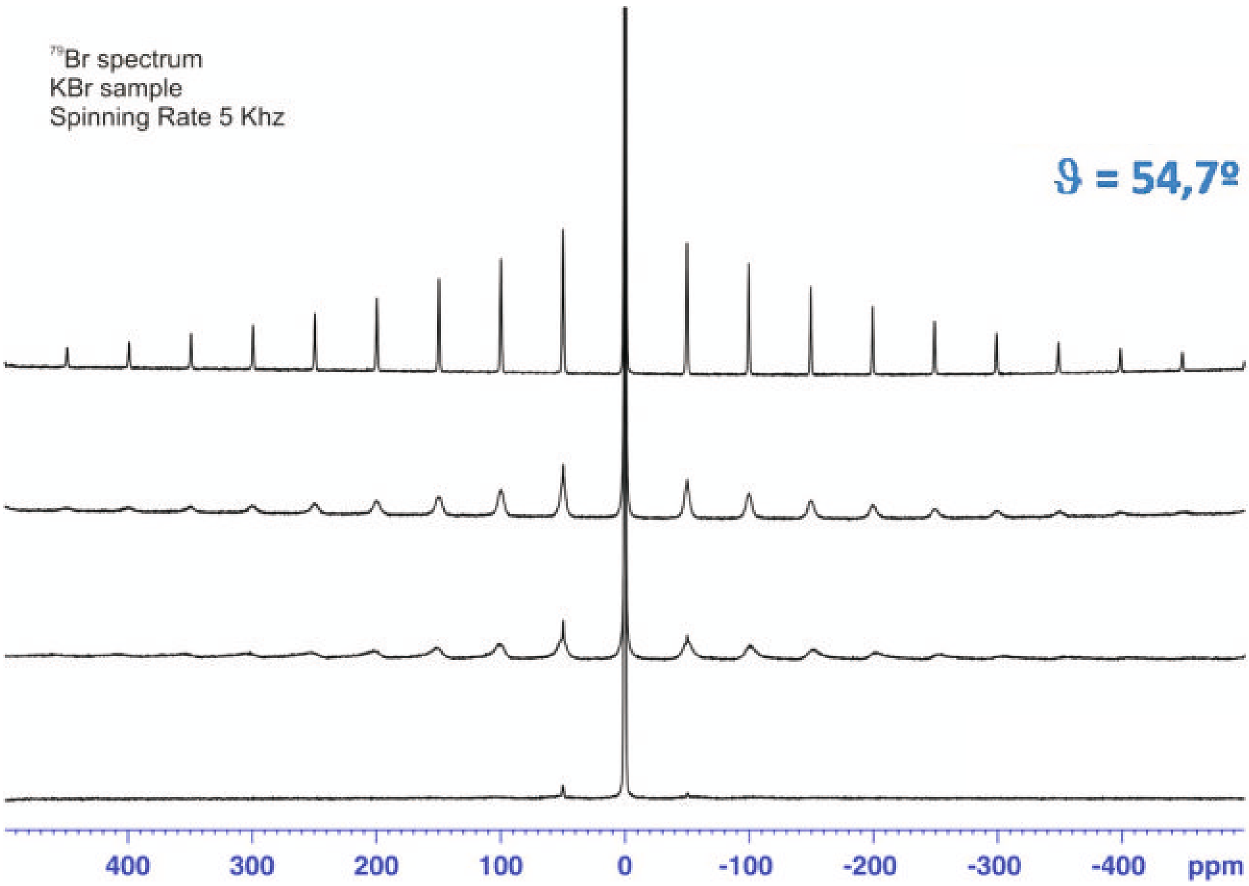
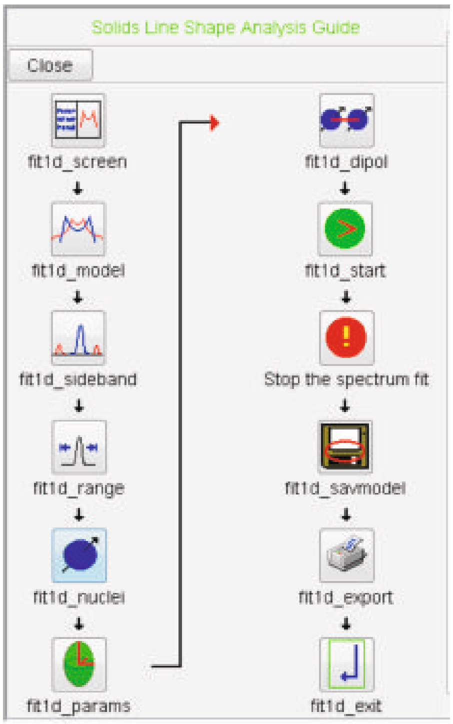
SOLID-STATE
NMR EXPERIMENTS

$$H_D^{I,S} = -\frac{\mu_0 \mu_N^2 g_I g_S}{4\pi\hbar^2}\frac{1}{r_{IS}^3}\frac{1}{2}\left(3\cos^2\theta - 1\right)2I_z S_z$$



Magic Angle Calibration with Kbr

Enter to Solids Line Shape Analysis Guide
by typing **solaguide**:



A Complete Decription of Solid-State NMR Experiments can be found in the **AVANCE Solids User Manual** (see **Help** menu in Topspin)

Solid-State NMR Experiments

Basic ssNMR experiments:

- Hahn-Echo experiment to be used in MAS experiments (**hahnecho.av**)
- 1D acquisition on X with High-power proton decoupling (**hpdec.av**)
- 2D J-resolved experiment for MAS experiments(**jres.av**)
- CPMG experiment for quadrupolar nuclei(**qcpmg.av** | qcpmgall.av | qcpmgint.av)
- Saturation recovery T1 experiment using echo prior to detection (**satrecechot1.av**)
- Saturation recovery T1 experiment (**satrect1.av**)
- 90-90 solid echo sequence for wide line observation (**solidecho.av**)
- 1D solid-state APT experiment (**sostapt.av**)
- 1D solid-state APT experiment using shaped pulse for phase modulated Lee-Goldburg decoupling for homonuclear dipolar (**sostapt_pmlg.av**)

1D Cross-Polarization Experiments:

- Basic CP experiment (**cp.av**)
- Basic CP experiment with flipback 90 (**cp90.av**)
- Basic CP experiment with explicit programming of acquisition (**cpadc.av**)
- Basic CP experiment with composite 180 pulse, for pulse determination (**cpc180.av**)
- Basic CP experiment with Hahn echo for stationary CP (**cphahn.av**)
- T1 inverion recovery with CP detection (**cpht1.av**)
- Proton T1rho experiment, X detected via cross polarisation (**cpht1rho.av**)
- Basic CP experiment with NQS, refocussed(**cpnqs.av**)
- CP editing experiment by phase inversion (**cppi.av**)
- CP editing experiment by phase inversion (**cppircp.av**)
- CP editing experiment by phase inversion (**cppispi.av**)
- Basic CP experiment with SELTICS SSB-suppression (**cpseltics.av**)
- Basic CP experiment with recoupling of CSA (**cpsuper.av**)
- Basic CP experiment with TOtal Suppreson of Sidebands (**cptoss.av**)
- Basic CP experiment with TOtal Suppreson of Sidebands and Non Quaternary Suppression (**cptoss_nqs.av**)
- Basic CP experiment with variable contact time (**cpvc.av**)
- Basic CP experiment with X T1 inverion recovery (**cpxt1.av**)
- Basic CP experiment for T1rho measurement of X-nucleus (**cpxt1rho.av**)
- Frequency shifted Lee-Goldburg decoupling after cp to see X-H J couplings (**fslg.av**)
- Basic cp experiment, contains calculations for various decoupling schemes: cw, tppm, xix, and pidec (**vacp.av**)

Double-CP experiments:

- Basic double CP experiment (**doubcp2d.av**)
- Basic double CP experiment (**doubcp.av**)

Multiple-Quantum Spectroscopy:

Using BABA

- 2D SQ-DQ correlation experiment for 1 rotor period recoupling using BABA. Standard experiment for 1H DQ spectra (baba1.av)
- 2D SQ-DQ correlation experiment for 2 rotor periods recoupling using BABA for weaker dipole dipole interactions, e.g. 19F, compensated for pulse imperfections (baba2rot.av)
- 2D SQ-DQ correlation experiment for 4 rotor periods recoupling using BABA. DQ experiment for weaker interaction hamiltonians, full compensated for pulse imperfections and offset effects (baba4rot.av)
- 2D SQ-TQ correlation experiment for 1 rotor period recoupling using BABA. Standard experiment for 1H DQ spectra (baba_1rp3qc.av)
- 2D SQ-DQ correlation experiment for 2 rotor period recoupling using BABA with cross polarization for weak dipole dipole couplings, compensated for pulse imperfections (babacp2.av)
- 2D SQ-DQ correlation experiment for 4 rotor period recoupling using BABA with cross polarization for weak dipole dipole couplings, full compensated for pulse imperfections and offset effects (babacp4.av)

Using POST-C7

- SQ-TQ experiment using POST_C7 sequence (pc7_1dtqf.av)
- 1D DQ excitation sequence POST_C7 with cross polarization (pc7cp1d.av)
- 2D SQ-DQ correlation experiment with POST_C7 sequence and cross polarization (pc7cp2d.av)
- 2D SQ-DQ correlation experiment with POST_C7 and cross polarization for large sweep width(pc7cp2dlsw.av)
- CS-CS correlation through dipolar couplings NOESY type sequence using POST-C7 for broader DQ excitation range (pc7cp2dnoe.av)
- 1D DQ excitation sequence POST_C7 for setup of the 2D INADEQUATE type experiments (pc71d.av)
- 2D SQ-DQ experiment using POST_C7 sequence (pc72d.av)
- 2D SQ-DQ experiment using POST_C7 sequence (pc72dlsw.av)
- 2D SQ-TQ experiment using POST_C7 sequence (pc72dlswtq.av)

Using R14_2^6

- 1D DQ excitation sequence R14_2^6 supercycled (r14_2_1d.av)
- 2D SQ-DQ correlation experiment with R14_2^6 supercycled use r14_2_1d.av for setup (r14_2_2d.av)
- 1D DQ excitation sequence R14_2^6 supercycled (r14_2_cp1d.av)
- 2D SQ-DQ correlation through dipole dipole interaction R14_2^6 supercycled (r14_2_cp2d.av)

Using SC14

- SC14 1D sequence to set up SC14 power level pl11 (sc14cp1d.av)
- DQ correlation - inadequate type sequence using SC14 (sc14cp2d.av)

Using SPC5

- 1D SQ-DQ correlation experiment with SPC5 sequence and cross polarization (spc5cp1d.av)
- 2D SQ-DQ correlation experiment with SPC5 sequence and cross polarization (spc5cp2d.av)
- 2D SQ-DQ correlation experiment with SPC5 sequence and cross polarization (spc5cp2dlsw.av)

Homonuclear Correlation / Exchange Experiments:

- 2D exchange NMR in rotating solids (cpnoesy.av)
- 2D INADEQUATE using cross-polarizarion (cpinadequate.av)
- 2D exchange NMR in rotating solids (cprfdr.av)
- 2D exchange NMR in rotating solids (rfdrps.av)

Heteronuclear Correlation Experiments:

- 2D correlation experiment using heteronuclear multiple quantum coherences (hmqc.av)
- Heteronuclear correlation between protons and X nuclei with or without homonuclear decoupling during t1 possible decoupling schemes are FSLG, PMLG, and DUMBO (hxhetcor.av)
- 2D HETCOR with FLSG during t1(lghetfq.av)
- 2D HETCOR with FLSG during t1(lghetfqpi.av)
- 2D HETCOR with FLSG during t1(lghetloop.av)
- 2D FSLG-HETCOR with shaped pulse during t1 (lghetshape.av)
- 2D MAS-J-HMQC experiment with FSLG decoupling (masjhmqc.av)

CA-CSA Heteronuclear Correlation Experiments:

- PASS CS-CSA experiment (cppass.av)

Recoupling Experiments:

- 2D REDOR experiment (cpredor.av)
- 2D REDOR experiment. Interleaved acquisition of S and S0 signal (cpredori.av)
- 1D Rotational echo double resonance experiment (cpredxy8.av)
- 2D Rotational echo double resonance experiment (cpredxy82d.av)

- REDOR experiment (redor.av)
- REDOR experiment (redori.av)
- 1D rotational echo double resonance experiment (selredor1d.av)
- 2D rotational echo double resonance experiment (selredor2d.av)

MQMAS Experiments for quadrupolar nuclei:

- 3Q MAS pulse program for half integer spin nuclei (mp3q.av)
- 3Q MAS pulse program for odd half integer spin nuclei (mp3qdfs.av)
- 3Q MAS pulse program for odd half integer spin nuclei. 4-pulse experiment with z-filter(mp3qdfsz.av)
- 3Q MAS pulse program for 3/2 nuclei (mp3qfam.av)
- 3Q MAS pulse program for 3/2 nuclei. 4-pulse experiment with z-filter (mp3qfamz.av)
- 3Q MAS pulse program for half integer spin nuclei. 4-pulse experiment with z-filter (mp3qzfil.av)
- 3Q MAS pulse program for half integer spin nuclei and with zero quantum filter (mp3qzqf.av)

Heteronuclear Experiments for quadrupoalr nuclei:

- 3Q HETCOR experiment for spin-5/2, spin-7/2, and spin-9/2 nuclei. 3Q in the indirect dimension for the quadrupole nucleus and MAS in the direct dimension for the spin-1/2 nucleus (mqhetcor.av)
- MQ-INEPT based 2D heteronuclear correlation experiment between spin 1/2 and half integer nucleus (mqjhetcor.av)

PISEMA Experiments:

- 2D PISEMA experiment to correlate X chemical shifts with 1H-X dipolar couplings, frequency switched Lee-Goldburg during 1H-15N dipolar evolution (pisema.av)
- 2D PISEMA experiment 2D pulse sequence to correlate X chemical shifts with 1H-X dipolar couplings and using ramped contact during FSLG period (pisemaramp.av)
- PISEMA experiment based on the Magic-Sandwich Decoupling in the indirect dimension (sammy.av)

STMAS Experiments:

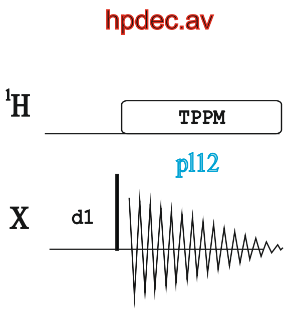
- (satellite transition) STMAS experiment on half integer nuclei (stmas.av)
- (satellite transition) STMAS experiment on half integer nuclei using double quantum filter for CT/CT signal suppression and z-filter (stmasdqfe.av)
- (satellite transition) STMAS experiment on half integer nuclei using double quantum filter for CT/CT signal suppression and z-filter (stmasdqfz.av)

CRAMPS Experiments:

- 1H CRAMPS, BR24 experiment (br24.av)
- 1H CRAMPS, MREV-8 experiment (mrev8.av)

Miscellaneous Experiments:

- Double frequency sweep followed by a selective excitation pulse(dfs90sel.av)
- Pseudo-2d pulse program for acquiring quad echo data with goniometer automation.(echogon.av)
- LGCR(lgcr1.av)
- Scalar Coupling Driven DQF chemical shift correlation (uc2qfcosyph.av)
- Pseudo-2d pulse program for acquiring quad echo data with goniometer automation(zggon.av)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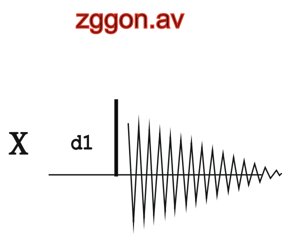
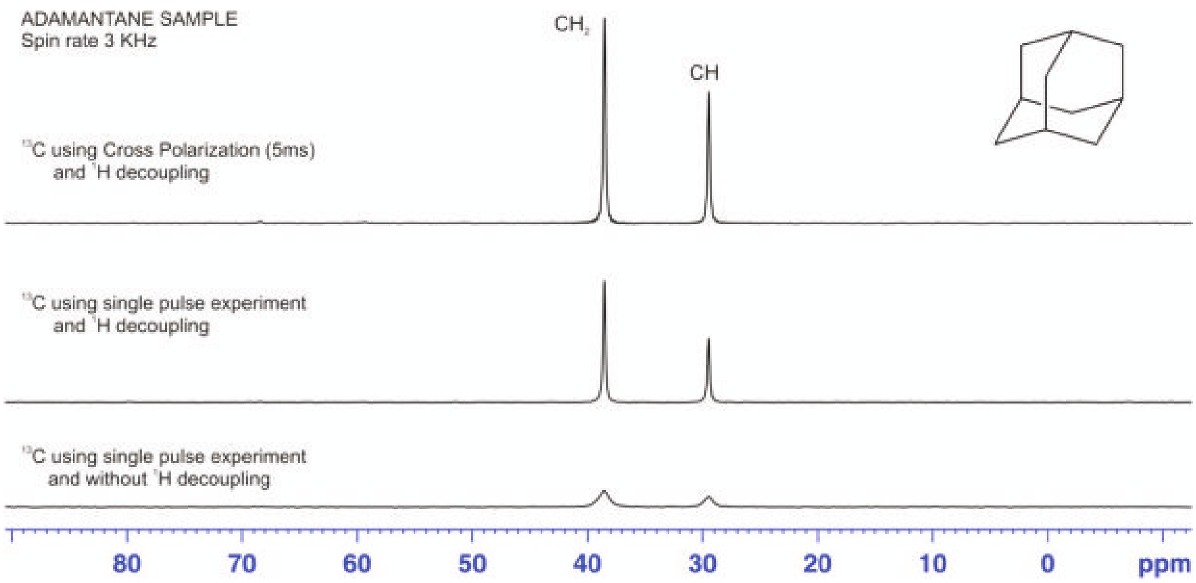
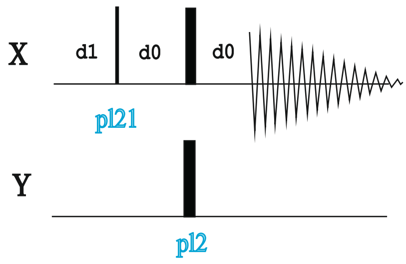
1H
TPPM
pl12
X
d1


zggon.av
X
d1


ADAMANTANE SAMPLE
Spin rate 3 KHz
CH2
CH
13C using Cross Polarization (5ms)
and 1H decoupling
13C using single pulse experiment
and 1H decoupling
13C using single pulse experiment
and without 1H decoupling
80
70
60
50
40
30
20
10
0
ppm

jres.av



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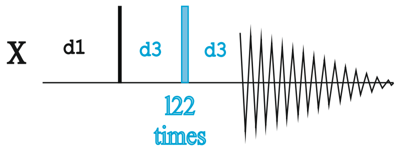
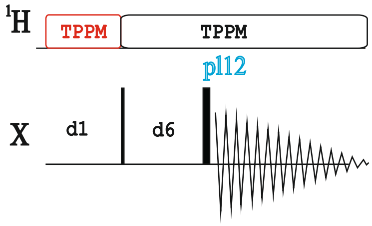
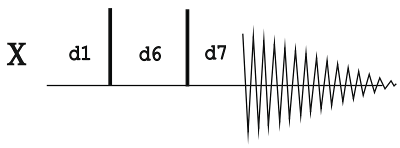


hahnecho.av

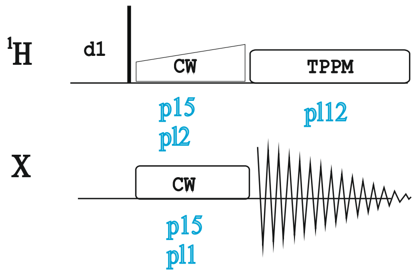


solidecho.av

echogon.av



cp.av
cpadc.av



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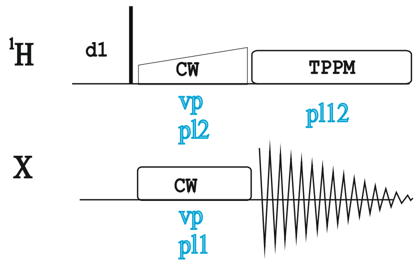
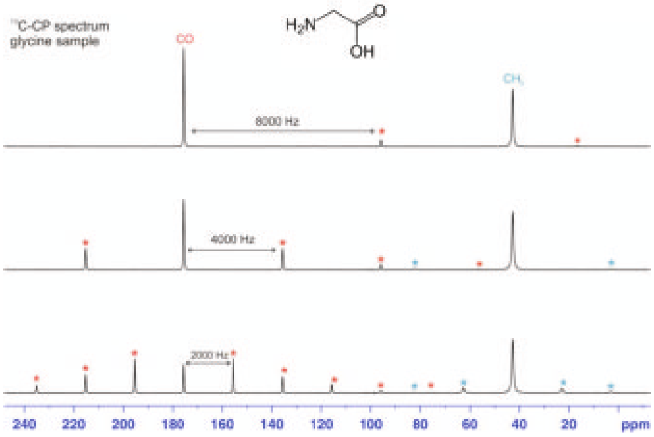
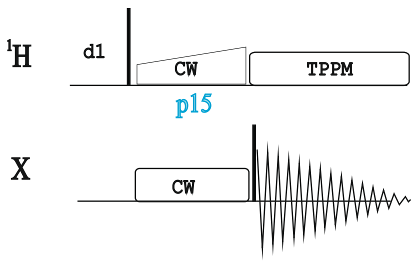




cp90.av
vacp.av



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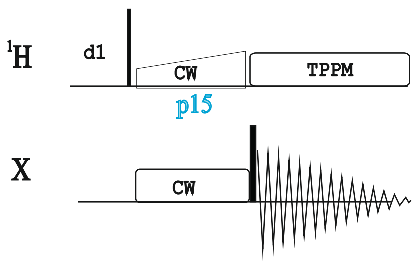
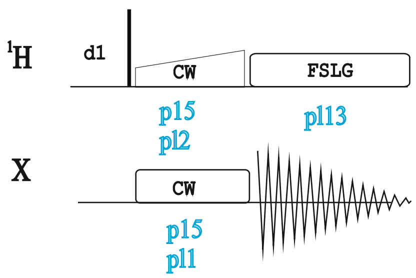


fqlg.av



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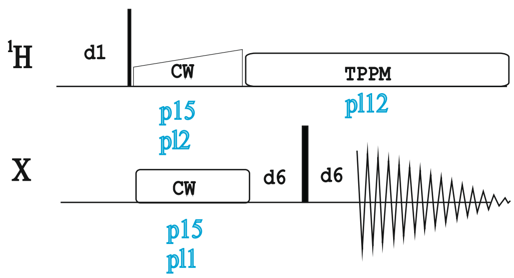

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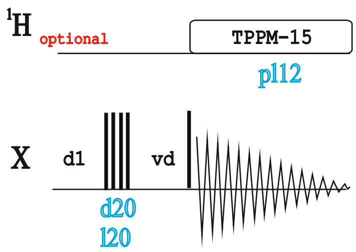

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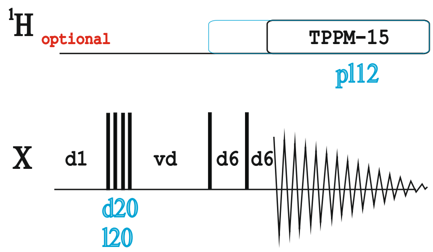

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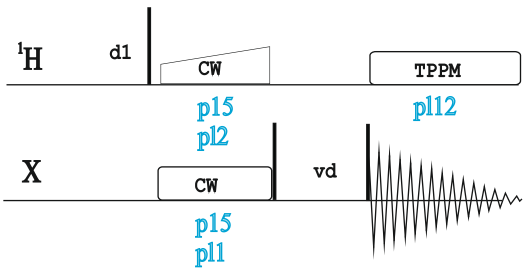

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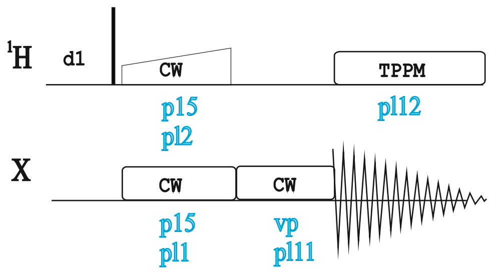

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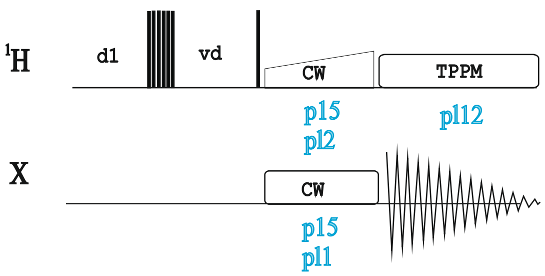

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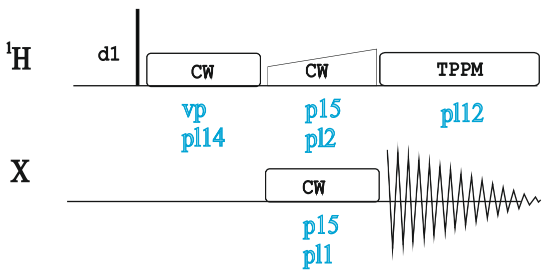

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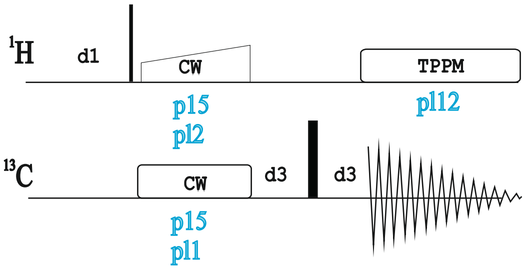

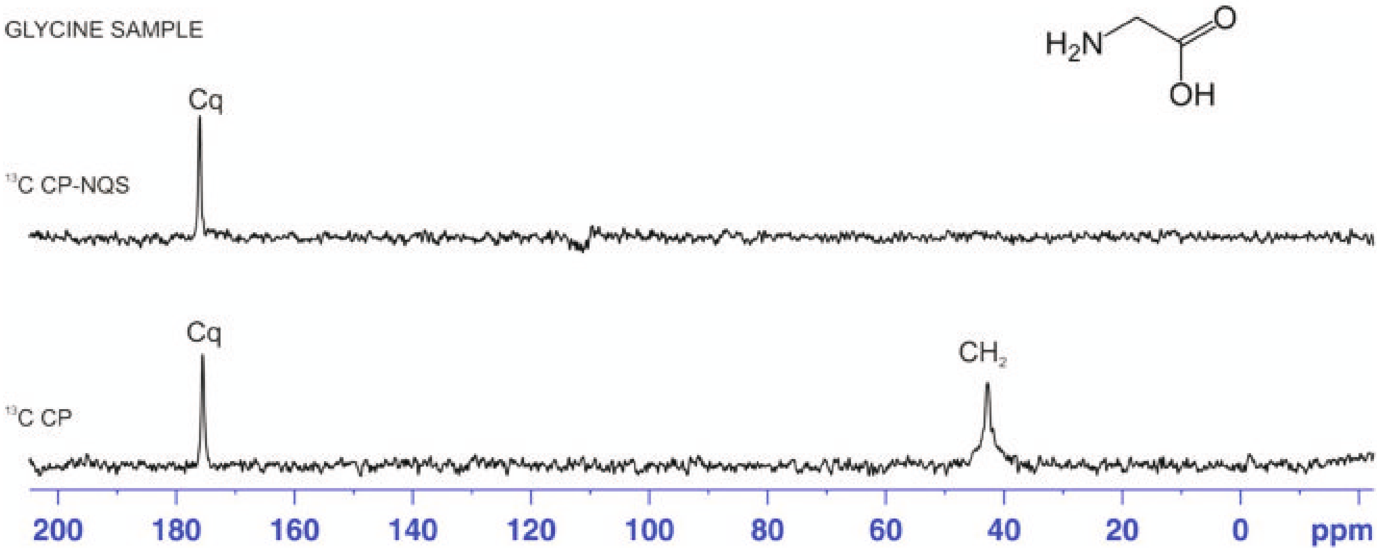

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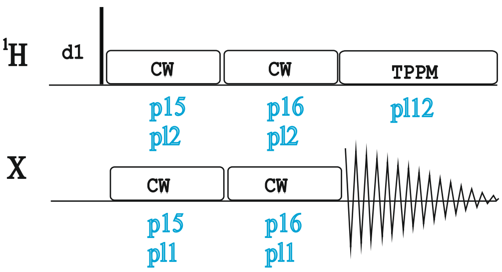

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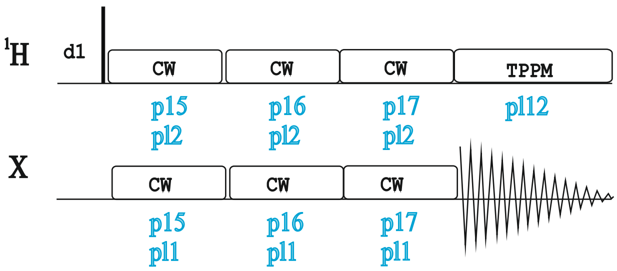

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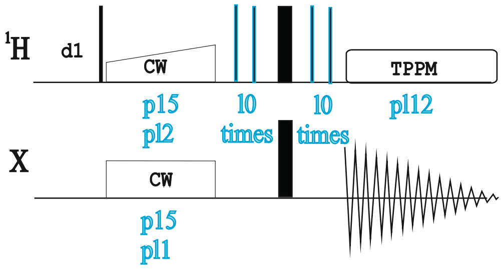

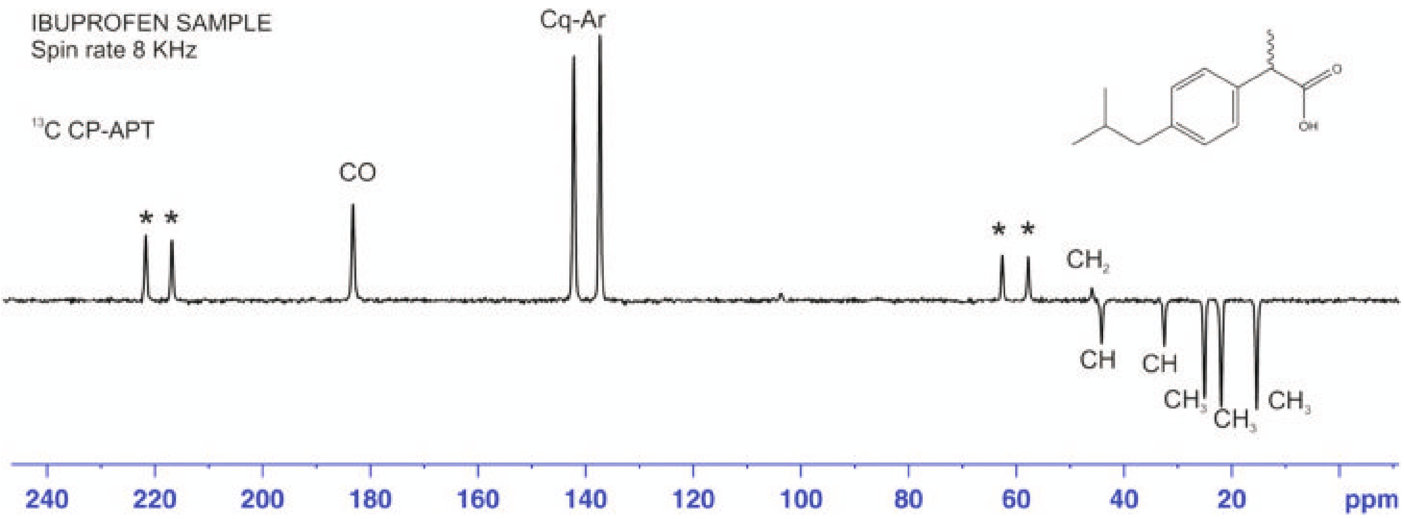

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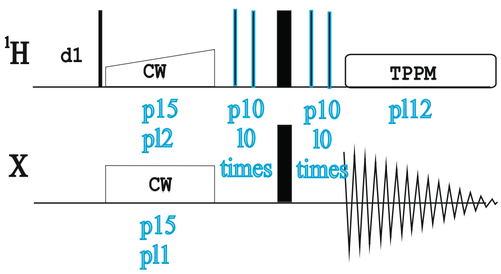

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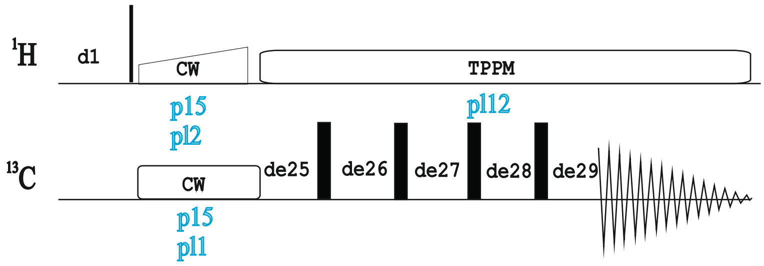

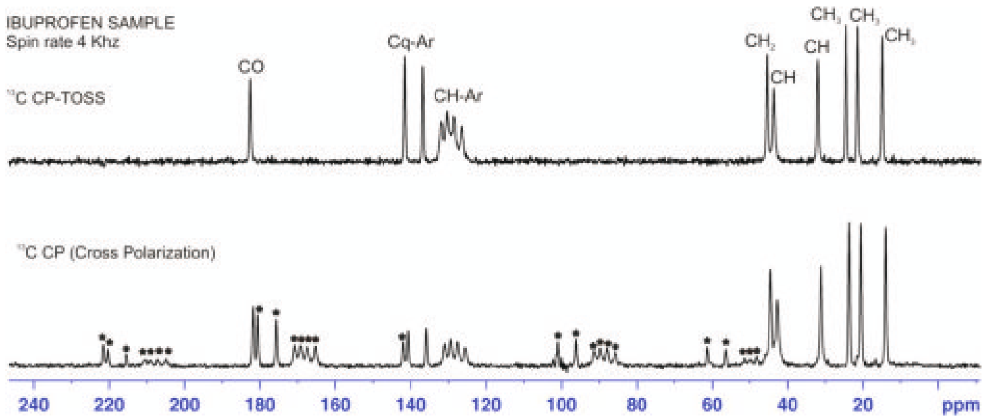

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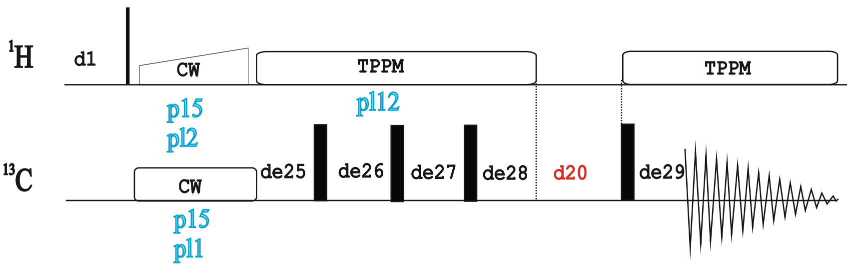

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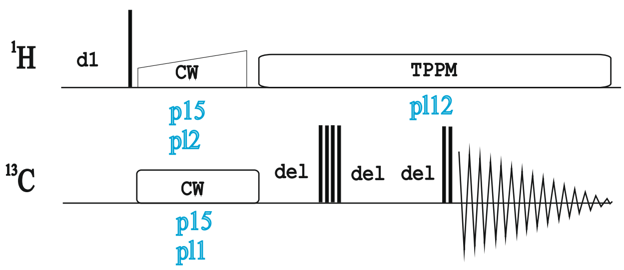

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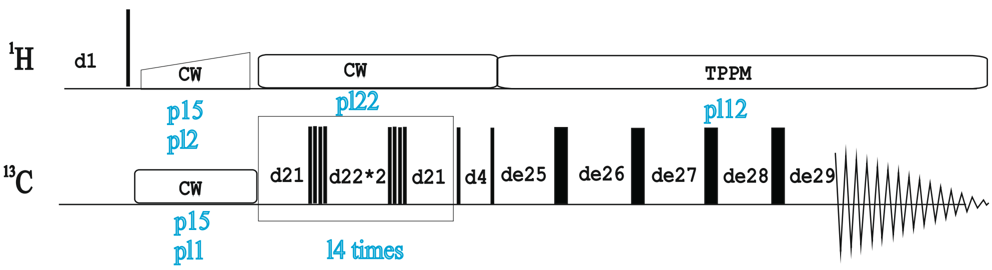

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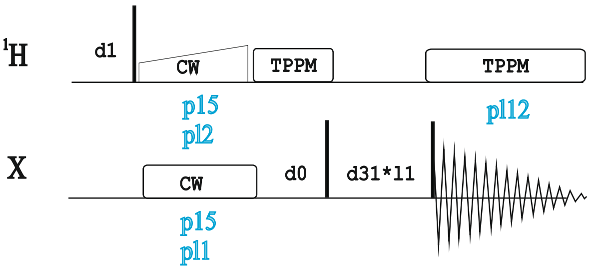

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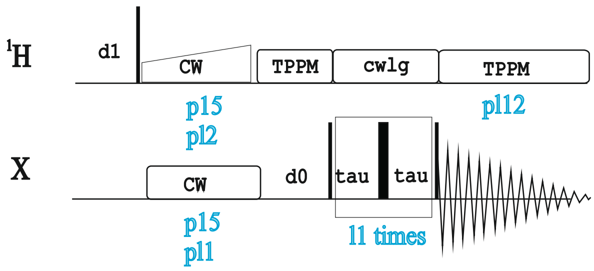

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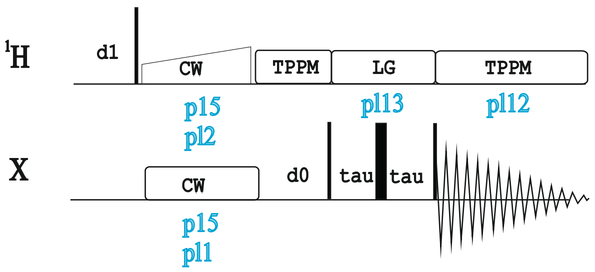

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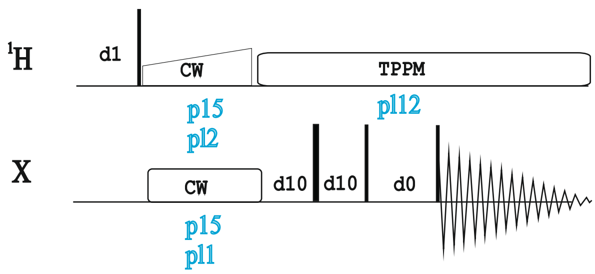

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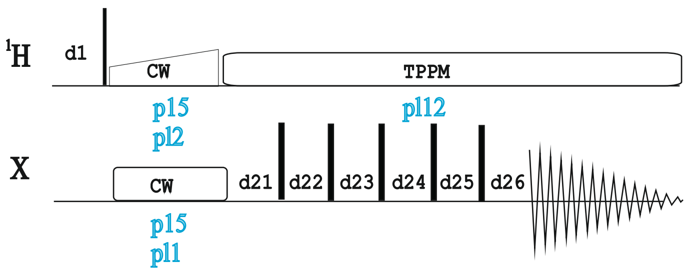

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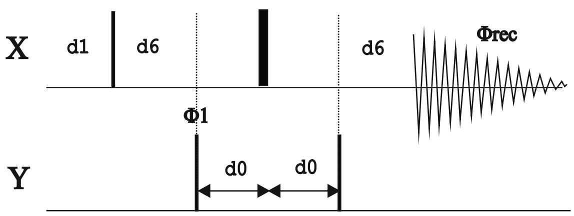

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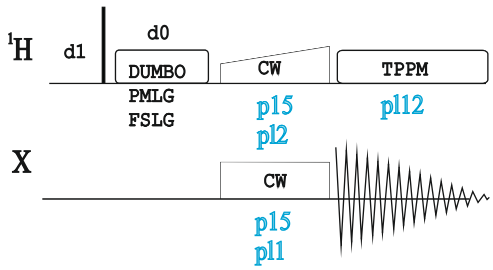

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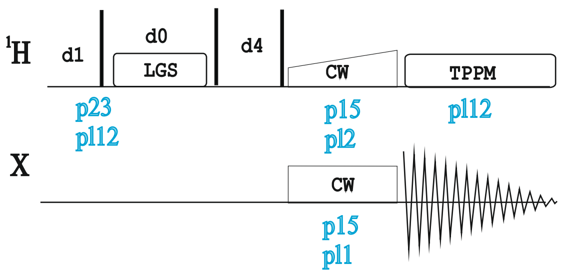

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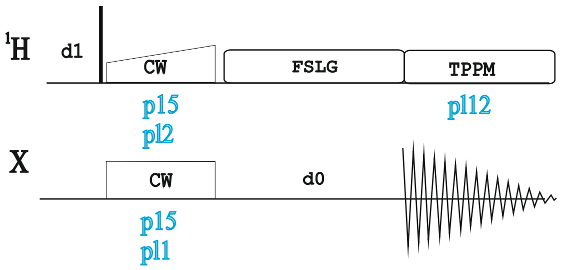

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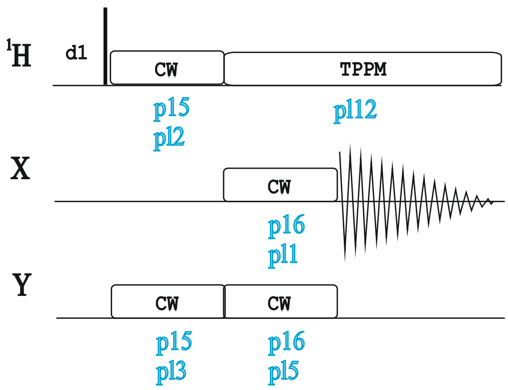

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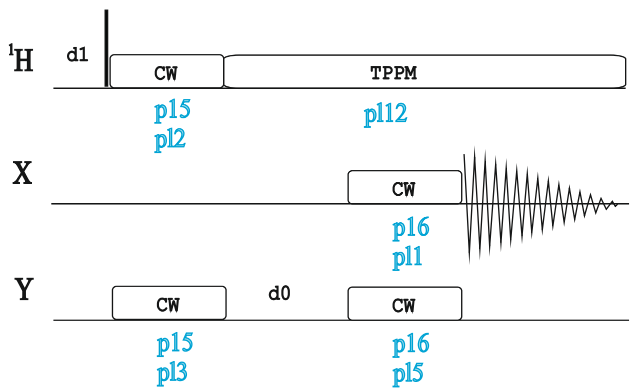

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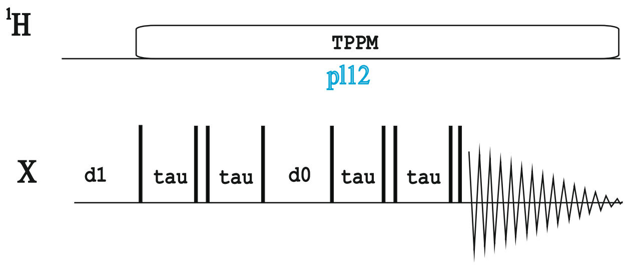

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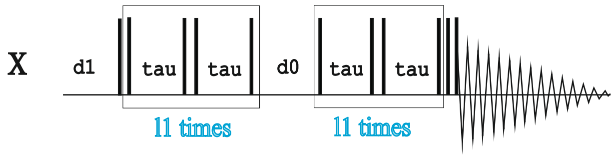

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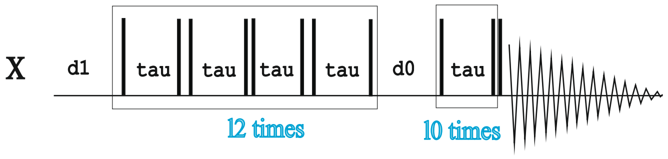

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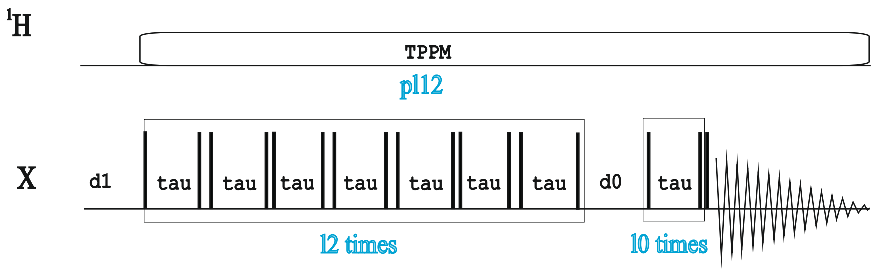

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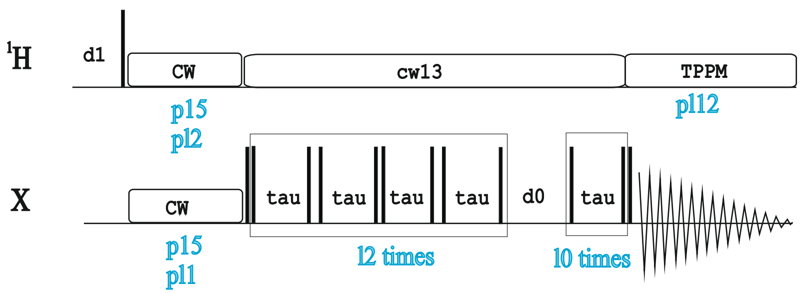

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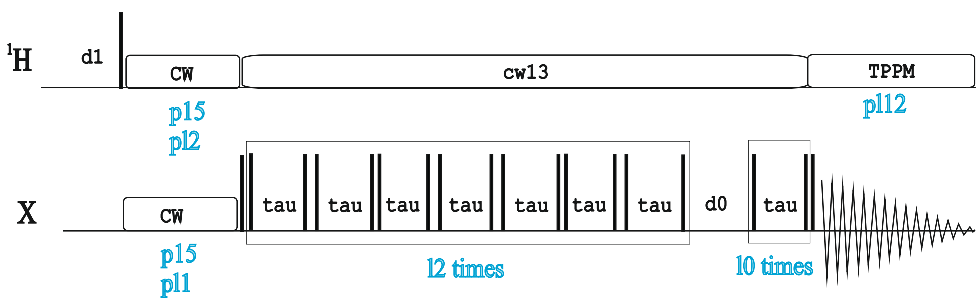

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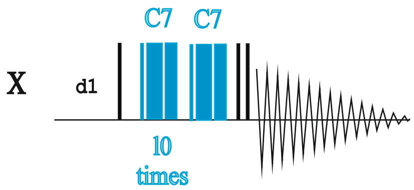

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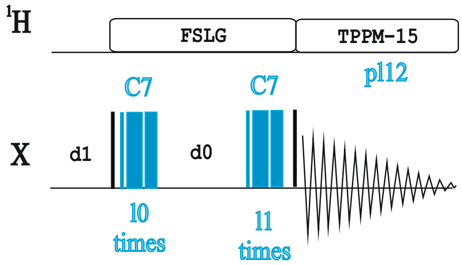

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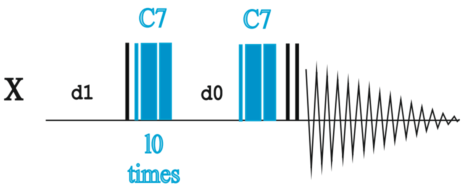

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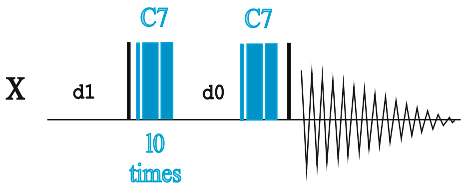

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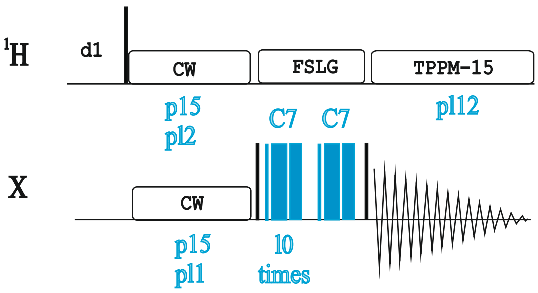

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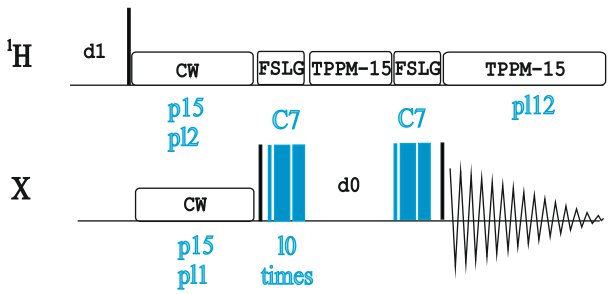

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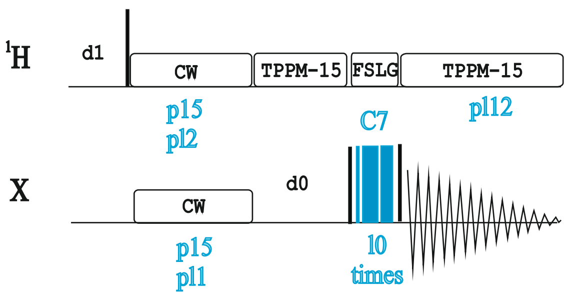

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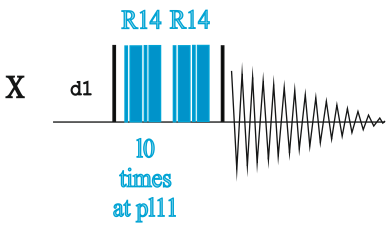

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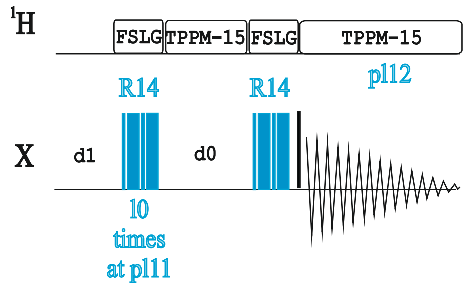

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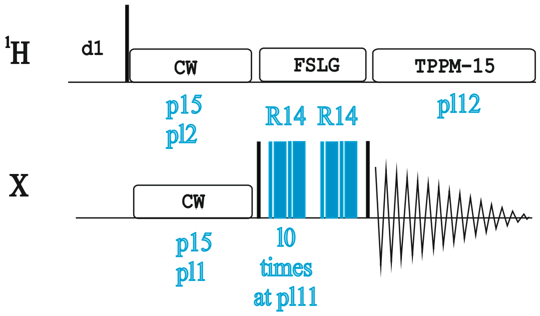

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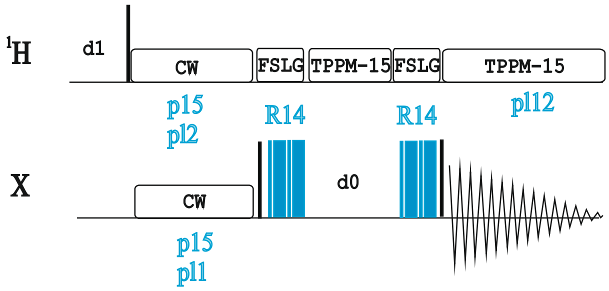

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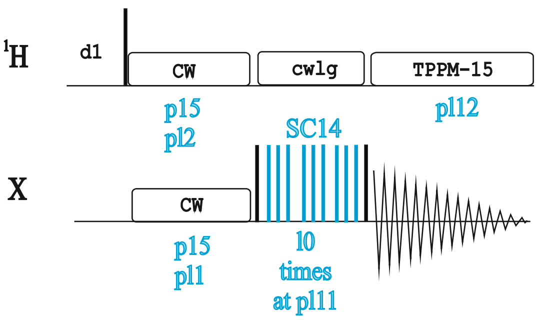

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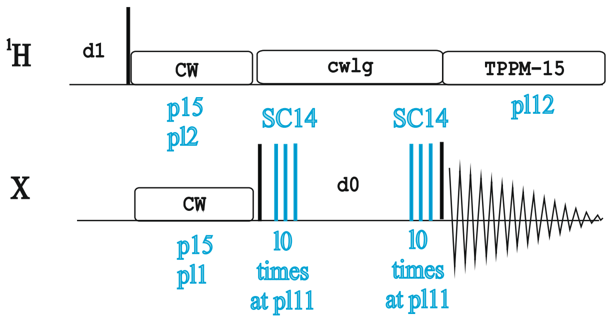

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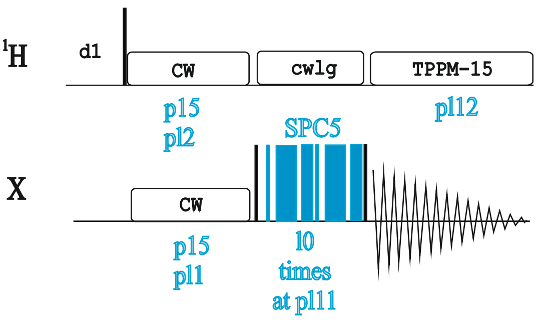

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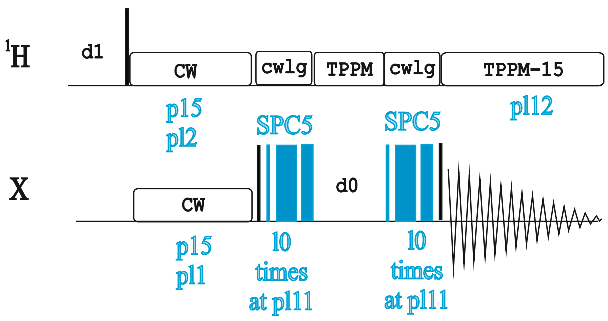

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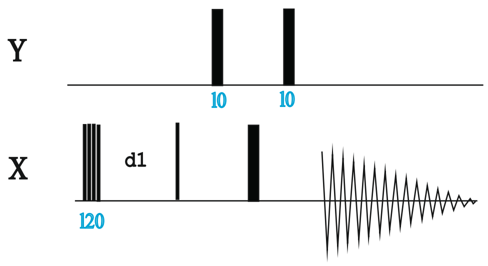

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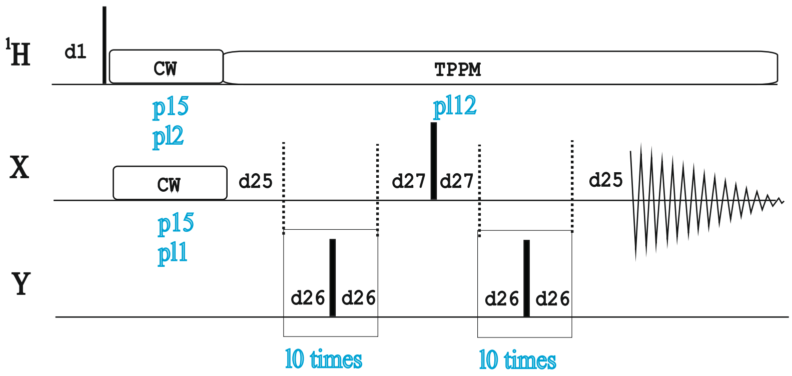

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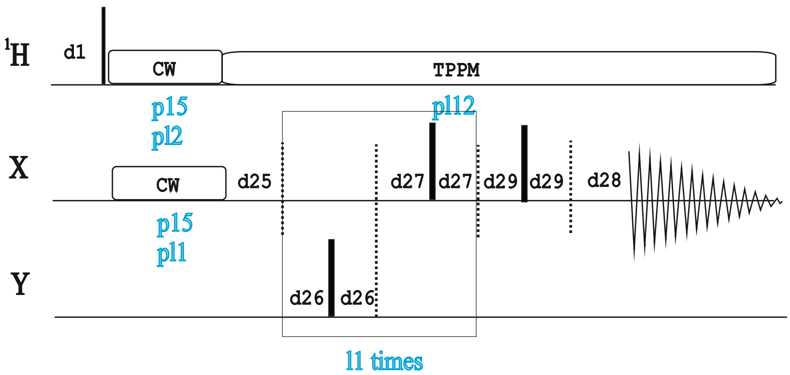

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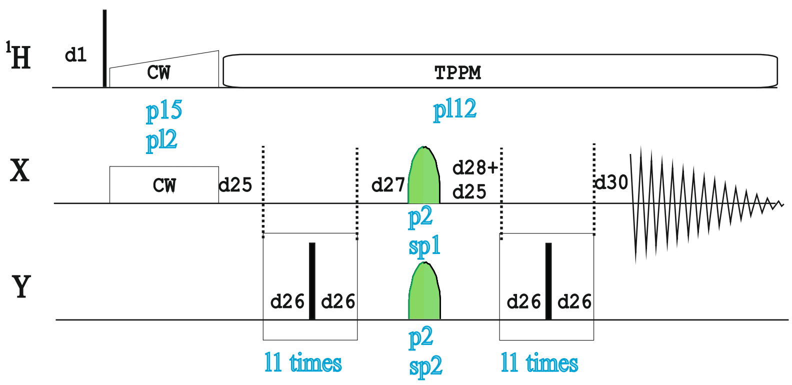

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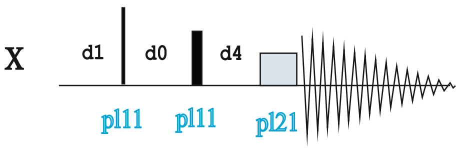

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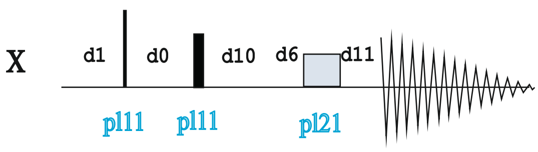

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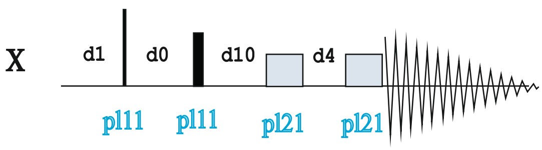

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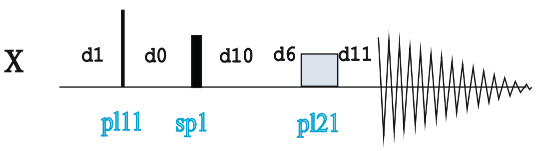

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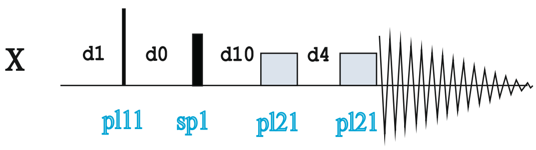

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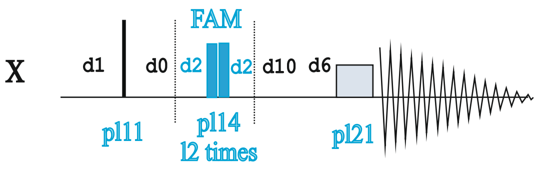

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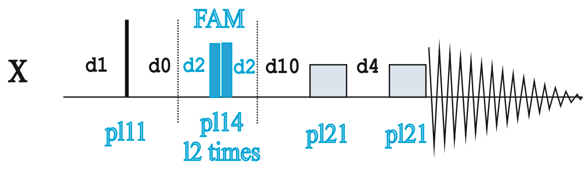

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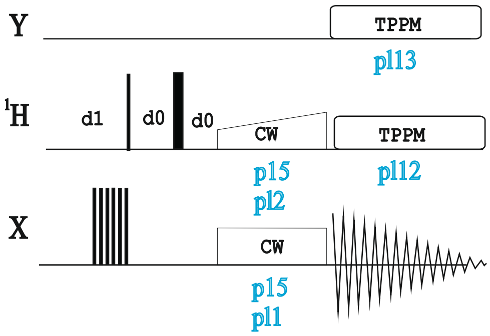

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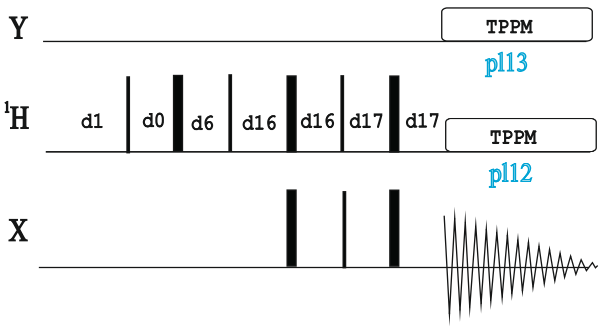

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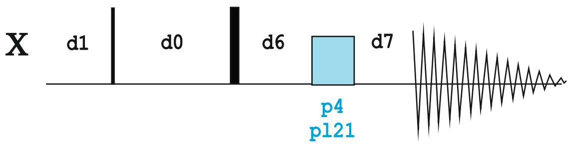

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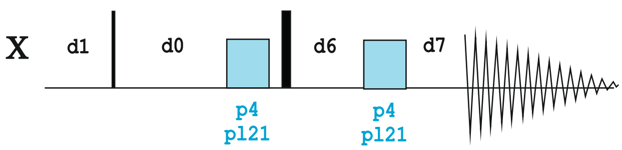

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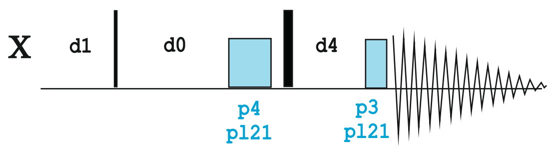

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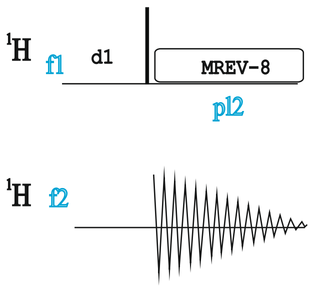

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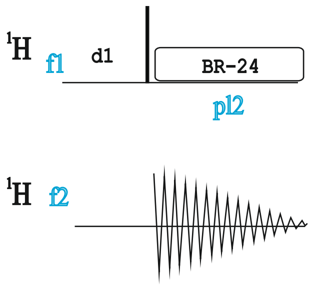

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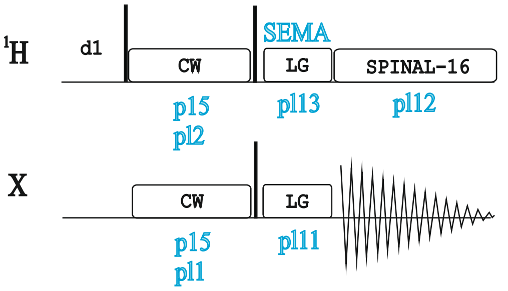

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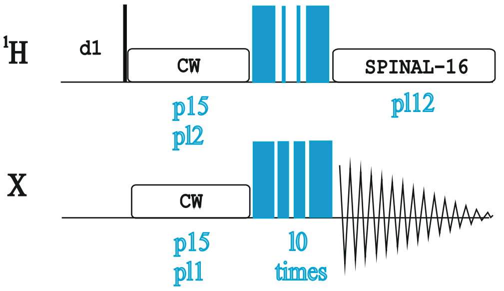

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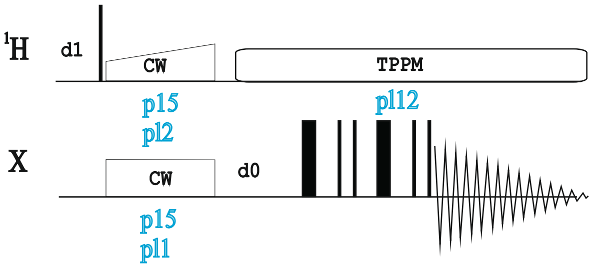

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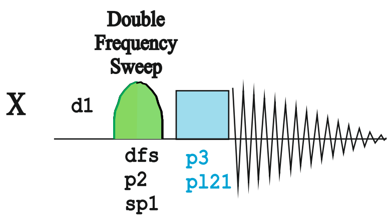

BRUKER PULSE PROGRAM CATALOGUE

NMRGuide

APPENDIX

;Pulprog.info
;avance-version (10/01/28)
;
;$CLASS=HighRes Info
;$COMMENT=

;For a pulseprogram the first characters (usually up to 6, but
;sometimes more) specify the type of experiment, e.g. DEPT, COSY,
;NOESY etc.. Further properties of the pulseprogram are
;indicated by a two-character code, which is added to the name
;in alphabetical order. For 2D experiments the mode (absolute value,
;phase sensitive or echo-antischo) is always indicated. H- or X-
;decoupling is assumed to be default for heteronuclear experiments,
;but not for homonuclear ones (except inad).
;In case of redundant information some two-character codes may be
;ommitted.
;
;The two-character codes used are the following:

ac accordion type experiment
ad using adiabatic spinlock
ar experiment for aromatic residues
at adiabatic TOCSY
bi with bird pulse for homonuclear J-decoupling
bp using bipolar gradients
cc cross correlation experiment
cn C13 and N15 dependent information in different indirect dimensions
co with COSY transfer
cp with composite pulse
ct constant time
cv convection compensated
cw decoupling using cw command
cx using CLEANEX_PM
dc decoupling using cpd command
df double quantum filter
di with DIPSI mixing sequence
dh homonuclear decoupling in indirect dimension
dw decoupling using cpd command only during wet sequence
dq double quantum coherence
ea phase sensitive using Echo/Antiecho method
ec with E.COSY transfer
ed with multiplicity editing
es excitation sculpting
et phase sensitive using Echo/Antiecho-TPPI method
fb using f2 - and f3 - channel
fd using f1 - and f3 - channel (for presaturation)
fr with presaturation using a frequency list
ft using f1 -, f2 - and f3 - channel (for presaturation)
fh F-19 observe with H-1 decoupling
fp using a flip-back pulse
fl for F-19 ecoupler
fw forward directed type experiment
f2 using f2 - channel (for presaturation)
f3 using f3 - instead of f2 - channel

f4 using f4 - instead of f2 - channel
gd gated decoupling using cpd command
ge gradient echo experiment
gp using gradients with ":gp" syntax
gr using gradients
gs using shaped gradients
hb hydrogen bond experiment
hc homodecoupling of a region using a cpd-sequence
hd homodecoupling
hf H-1 observe with F-19 decoupling
hs with homospoil pulse
ia InPhase-AntiPhase (IPAP) experiment
id IDIS - isotopically discriminated spectroscopy
ig inverse gated
ii using inverse (invi/HSQC) sequence
im with incremented mixing time
in with INEPT transfer
i4 using inverse (inv4/HMQC) sequence
jc for determination of J coupling constant
jd homonuclear J-decoupled
jr with jump-return pulse
js jump symmetrized (roesy)
lp with low-pass J-filter
lq with Q-switching (low Q)
lr for long-range couplings
l2 with two-fold low-pass J-filter
l3 with three-fold low-pass J-filter
mf multiple quantum filter
ml with MLEV mixing sequence
mq using multiple quantum
nc N15 and C13 dependent information in different indirect dimensions
nd no decoupling
no with NOESY mixing sequence
pc with presaturation and composite pulse
pg power-gated
ph phase sensitive using States-TPPI, TPPI, States or QSEQ
pl preparing a frequency list
pn with presaturation using a 1D NOESY sequence
pp using purge pulses
pr with presaturation
ps with presaturation using a shaped pulse
qf absolute value mode
qn for QNP-operation
qs phase sensitive using qseq-mode
rc for determination of residual dipolar couplings (RDC)/ J couplings
rd refocussed
rl with relay transfer
ro with ROESY mixing sequence
rs with radiation damping suppression using gradients
rt real time
ru using radiation damping compensation unit
rv with random variation
r2 with 2 step relay transfer
r3 with 3 step relay transfer

se spin echo experiment
sh phase sensitive using States et al. method
si sensitivity improved
sm simultaneous evolution of X and Y chemical shift
sp using a shaped pulse
sq using single quantum
ss spin-state selective experiment
st phase sensitive using States-TPPI method
sy symmetric sequence
s3 S3E experiment
tf triple quantum filter
tp phase sensitive using TPPI
tr using TROSY sequence
tz zeroquantum (ZQ) TROSY
ul using a frequency list
us updating shapes
wg watergate using a soft-hard-soft sequence
wt with WET watersuppression
w5 watergate using W5 pulse
xf x-filter experiments
xy with XY CPMG sequence
x1 x-filter in F1
x2 x-filter in F2
x3 x-filter in F3
zf with z-filter
zq zero quantum coherence
zs using a gradient/rf spoil pulse
1d 1D version
1s using 1 spoil gradients
11 using 1-1 pulse
19 using 3-9-19 pulse
19f for F19
2h using 2H lockswitch unit
2s using 2 spoil gradients
3d 3D sequence
3n for E.COSY (3 spins, negative correlation)
3p for E.COSY (3 spins, positive correlation)
3s using 3 spoil gradients
30 using a 30 degree flip angle
45 using a 45 degree flip angle
90 using a 90 degree flip angle
135 using a 135 degree flip angle
180 using a 180 degree pulse

;Typical experiment names would be:
; cosy, dept, dipsi2, hmbc, hmqc, hoesy, hsqc, inad, inept,
; mlev, noesy, roesy or trosy.

;Inverse correlations are denoted as hmbc, hmqc or hsqc.
; Experiments with a BIRD sequence in the beginning
; also contain a bi in the name.

```
;1D experiments, which are analogues of 2D experiments by virtue of
;   a selective pulse, start with sel.
;Semiselective 2D experiments have the same name as the unselective
;   version but with an s at the beginning:
;
;   scosyph <-> cosyph.


;A phase-sensitive (States-TPPI, TPPI etc.) NOESY experiment with
;   presaturation would then be:
;
;           noesy + ph + pr  =    noesyphpr.


;In the other direction the pulseprogram hmbcgplpndqf would be
;
;       hmbc + gp + lp + nd + qf
;
;           and therefor an:
;
;           inverse correlation for long-range couplings (HMBC) with
;          coherence selection using gradients with ":gp" syntax,
;          low-pass J-filter,
;          no decoupling
;          in absolute value mode.

;The nomenclature of parameters is described in Pulprog.info.



;Comments like:
;
;           ;avance-version
;           ;begin ____
;           ;end ____
;
;           with (____ = MLEV17, DIPSI2, ...)
;
;are evaluated by NMRSIM for the pulseprogram display and should
;therefor not be removed. The syntax for begin/end statements allows
;characters, numbers and '_'. Arithmetic operators must not be used.
;
;
;The comments:
;                                    ;preprocessor-flags-start
;                        ;preprocessor-flags-end
;
;are also evaluated to identify flags used in the pulseprogram and
;must also not be removed.

;$Id: $
```

```
;Param.info
;avance-version (10/02/01)
;
;The following convention is used for power levels, pulses, delays
;and loop counters throughout the microprograms:
;
;$CLASS=HighRes Info
;$COMMENT=


;pl0 :
;pl1 : f1 channel - power level for pulse (default)                {all, PL90(F1)}
;pl2 : f2 channel - power level for pulse (default)                {all, PL90(F2)}
;pl3 : f3 channel - power level for pulse (default)                {all, PL90(F3)}
;pl4 : f4 channel - power level for pulse (default)                {all, PL90(F4)}
;pl5 : f5 channel - power level for pulse (default)                {}
;pl6 : f6 channel - power level for pulse (default)                {}
;pl7 : f7 channel - power level for pulse (default)                {}
;pl8 : f8 channel - power level for pulse (default)                {}
;pl9 : f1 channel - power level for presaturation                  {default+lcnmr+triple+triple2+triple_na,
SQPL[3](F1)}
;pl10: f1 channel - power level for TOCSY-spinlock                 {all, SQPL[1](F1)}
;pl11: f1 channel - power level for ROESY-spinlock                 {all, SQPL[2](F1)}
;pl12: f2 channel - power level for CPD/BB decoupling              {all, SQPL[0](F2)}
;pl13: f2 channel - power level for second CPD/BB decoupling       {default+lcnmr+triple_c, SQPL[4](F2)}
; or   f2 channel - power level for Cbeta/CO decoupling            {triple+triple2, SHPL[22](F2)}
;pl14: f2 channel - power level for cw saturation                  {default, SQPL[13](F2)}
; or   f2 channel - power level for low power decoupling           {lcnmr+triple+triple2, SQPL[5](F2)}
;pl15: f2 channel - power level for TOCSY-spinlock                 {all, SQPL[1](F2)}
;pl16: f3 channel - power level for CPD/BB decoupling              {all, SQPL[0](F3)}
;pl17: f4 channel - power level for CPD/BB decoupling              {all, SQPL[0](F4)}
;pl18: f1 channel - power level for 3-9-19-pulse (watergate)       {default+lcnmr+triple+triple2+triple_na,
PL90(F1)}
;pl19: f1 channel - power level for CPD/BB decoupling              {default+lcnmr+triple+triple2+triple_na,
SQPL[0](F1)}
;pl20: f1 channel - power level for Dante-z pulse                  {}
; or   f2 channel - power level for TOCSY-spinlock (high sel.)     {triple, SQPL[8](F2)}
; or   f2 channel - power level for TOCSY-spinlock (med. sel.)     {triple_na, SQPL[7](F2)}
;pl21: f1 channel - power level for TOCSY-spinlock (med. sel.)     {triple, SQPL[7](F1)}
; or   f2 channel - power level for presaturation                  {default+lcnmr, SQPL[3](F2)}
;pl22: f2 channel - power level for TOCSY-spinlock (med. sel.)     {triple, SQPL[7](F2)}
; or   f3 channel - power level for presaturation                  {lcnmr, SQPL[3](F3)}
; or   f3 channel - power level for TOCSY-spinlock (med. sel.)     {triple_na, SQPL[9](F3)}
;pl23: f3 channel - power level for TOCSY-spinlock                 {default+lcnmr+triple_c, SQPL[1](F3)}
; or   f3 channel - power level for Rexchange/T2 spinlock          {triple+triple2, SQPL[16]{(F3)}
; or   f3 channel - power level for TOCSY-spinlock                 {triple_na, SQPL[8](F3)}
;pl24: f2 channel - power level for hd/hc decoupling               {all, SQPL[14](F2)}
;pl25: f1 channel - power level for TOCSY spinlock (higher sel.)   {triple_na, SQPL[8](F1)}
; or   f2 channel - power level for Eretic                         {default+lcnmr, SHPL[20](F2)}
; or   f3 channel - power level for T1rho spinlock                 {triple+triple2, SQPL[15](F3)}
;pl26: f2 channel - power level for cw decoupling                  {default+lcnmr, SQPL[12](F2)}
; or   f2 channel - power level for TOCSY spinlock (higher sel. II) {triple_na, SQPL[8](F2)}
; or   f3 channel - power level for low power decoupling           {triple, SQPL[5](F3)}
;pl27: f1 channel - power level for pulsed ROESY-spinlock          {default+lcnmr, SQPL[11](F1)}
```

```
; or   f1 channel - power level for cleanex spinlock                 {triple2, SQPL[10](F1)}
; or   f2 channel - power level for TOCSY spinlock (higher sel. III)  {triple_na, SQPL[9](F2)}
; or   f3 channel - power level for TOCSY-spinlock                  {triple, SQPL[1](F3)}
;pl28: f2 channel - power level for selective Ca or CO decoupling    {triple+triple2, SHPL[20](F2)}
; or   f2 channel - power level for selective decoupling             {triple_na, SHPL[28](F2)}
;pl29: f1 channel - power level for trim pulse (T1rho filter) in STD  {default, SHPL[1](F1)}
; or   f2 channel - power level for simultaneous Ca and CO decoupling {triple2, SHPL[21](F2)}
;pl30: f2 channel - power level for bilev decoupling               {default+triple+triple2+triple_na, SQPL[0](F2)}
;pl31: f2 channel - power level for bilev decoupling               {default+triple+triple2+triple_na, SQPL[6](F2)}
;pl32: f1 channel - power level for low power presaturation          {default+lcnmr+triple+triple2+triple_na,
SQPL[12](F1)}
;
;
;
;
;sp0 : f1 channel - shaped pulse 180 degree  (adiabatic TOCSY)          {}
; or   f2 channel - shaped pulse 180 degree  (two-fold modulated)        {triple_na, SH[29](F2)}
;sp1 : f1 channel - shaped pulse for selective excitation              {default, SH[0](F1)}
; or   f1 channel - shaped pulse for wet                          {lcnmr, SH[7](F1)}
; or   f1 channel - shaped pulse for water flipback                  {triple+triple2+triple_na, SH[5](F1)}
;sp2 : f1 channel - shaped pulse 180 degree                        {default, SH[1](F1)}
; or   f1 channel - shaped pulse for wet                          {lcnmr, SH[7](F1)}
; or   f2 channel - shaped pulse  90 degree  (on resonance)            {triple+triple2, SH[6](F2)}
; or   f2 channel - shaped pulse  90 degree  (on resonance)            {triple_na, SH[23](F2)}
;sp3 : f2 channel - shaped pulse 180 degree  (adiabatic)               {default+lcnmr, SH[4](F2)}
; or   f2 channel - shaped pulse 180 degree  (on resonance)            {triple+triple2, SH[8](F2)}
; or   f2 channel - shaped pulse 180 degree  (on resonance)            {triple_na, SH[25](F2)}
;sp4 : f2 channel - shaped pulse  90 degree  (off resonance)            {triple+triple2, SH[6](F2)}
; or   f2 channel - shaped pulse 180 degree  (short, broadband)          {default+lcnmr, SH[14](F2)}
;sp5 : f1 channel - shaped pulse 180 degree  (adiabatic)               {default, SH[5](F1)}
; or   f1 channel - shaped pulse 180 degree  (off resonance)           {triple+triple2, SH[8](F2)}
; or   f2 channel - shaped pulse 180 degree  (off resonance)           {triple_na, SH[25](F2)}
;sp6 : f1 channel - shaped pulse for presaturation                {default+lcnmr+triple2+triple_na, SH[4](F1)}
; or   f2 channel - shaped pulse  90 degree  (off res., time reversed)    {triple, SH[7](F2)}
;sp7 : f1 channel - shaped pulse for wet                          {lcnmr, SH[7]*0.817(F1)}
; or   f1 channel - shaped pulse 180 degree  (adiabatic)               {triple_c, SH[5](F1)}
; or   f2 channel - shaped pulse 180 degree  (adiabatic)               {default, SH[5](F2)}
; or   f2 channel - shaped pulse 180 degree  (off resonance2)           {triple+triple2, SH[8](F2)}
; or   f2 channel - shaped pulse 180 degree  (off resonance2)           {triple_na, SH[25](F2)}
;sp8 : f1 channel - shaped pulse for wet                          {lcnmr, SHPL[7]*1.270(F1)}
; or   f1 channel - shaped pulse 180 degree  (adiabatic)               {default, SH[4](F1)}
; or   f2 channel - shaped pulse  90 degree  (on res., time reversed)     {triple+triple2, SH[7](F2)}
; or   f2 channel - shaped pulse  90 degree  (on res., time reversed)     {triple_na, SH[24](F2)}
;sp9 : f1 channel - shaped pulse for wet                          {lcnmr, SHPL[7]*0.593(F1)}
; or   f2 channel - shaped pulse 180 degree  (higher selectivity)        {triple+triple2, SH[11](F2)}
; or   f3 channel - shaped pulse 180 degree  (on resonance)            {triple_na, SH[9](F3)}
;sp10: f1 channel - shaped pulse for tilted ROESY                     {}
; or   f1 channel - shaped pulse for wet                          {lcnmr, SHPL[7]*3.198(F1)}
; or   f1 channel - shaped pulse 180 degree  (excitation sculpting)      {default, SH[5](F1)}
; or   f2 channel - shaped pulse  90 degree  (higher selectivity)        {triple+triple2, SH[9](F2)}
; or   f2 channel - shaped pulse  90 degree  (higher selectivity)        {triple_na, SH[26](F2)}
;sp11: f1 channel - shaped pulse for water flipback                 {default+lcnmr, SH[5](F1)}
; or   f1 channel - shaped pulse for water flipback2                {triple+triple2+triple_na, SH[6](F1)}
; or   f2 channel - shaped pulse for water flipback                 {triple_c, SH[5](F2)}
```

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;sp12: f1 channel - shaped pulse for wet2                        {}
; or   f2 channel - shaped pulse  90 degree  (higher sel., time rev.)     {triple+triple2, SH[10](F2)}
; or   f2 channel - shaped pulse  90 degree  (higher sel., time rev.)     {triple_na, SH[26](F2)}
;sp13: f1 channel - shaped pulse for wet2                        {}
; or   f1 channel - shaped pulse 180 degree  (adiabatic)              {triple_c, SH[4](F1)}
; or   f2 channel - shaped pulse 180 degree  (adiabatic)              {triple+triple2+triple_na, SH[4](F2)}
;sp14: f1 channel - shaped pulse for wet2                        {}
; or   f2 channel - shaped pulse 180 degree  (adiabatic bilev decoupling) {default+triple+triple2, SH[13](F2)}
; or   f3 channel - shaped pulse 180 degree  (adiabatic)              {triple_na, SH[8](F3)}
;sp15: f2 channel - shaped pulse 180 degree for decoupling (Ca or CO)     {triple, SH[20](F2)}
; or   f2 channel - shaped pulse 180 degree for decoupling (Cbeta)        {triple2, SH[22](F2)}
; or   f2 channel - shaped pulse 180 degree for decoupling (C')           {triple_na, SH[28](F2)}
;sp16: f2 channel - shaped pulse 180 degree  (higher sel., off res.)      {triple+triple2, SH[11](F2)}
;sp17: f2 channel - shaped pulse 180 degree  (higher sel., off res.)      {triple+triple2, SH[11](F2)}
;sp18: f2 channel - shaped pulse 180 degree  (adiabatic matched sweep)    {default+triple, SH[19](F2)}
;sp19: f1 channel - shaped pulse for wet                         {default, SH[7]*0.817(F1)}
; or   f2 channel - shaped pulse 180 degree  (inversion (sharp))          {triple, SH[31](F2)}
; or   f2 channel - shaped pulse  90 degree  (NH)                     {triple_c, SH[10](F2)}
; or   f3 channel - shaped pulse  90 degree  (T1rho, adiabatic ramp up)   {triple2, SH[6](F3)}
;sp20: f1 channel - shaped pulse for wet                         {default, SH[7]*1.270(F1)}
; or   f2 channel - shaped pulse 180 degree  (off resonance3)             {triple, SH[8](F2)}
; or   f2 channel - shaped pulse  90 degree  (NH, time reversed)          {triple_c, SH[11](F2)}
; or   f3 channel - shaped pulse  90 degree  (T1rho, adiabatic ramp down) {triple2, SH[7](F3)}
;sp21: f1 channel - shaped pulse for wet                         {default, SH[7]*0.593(F1)}
; or   f1 channel - shaped pulse 180 degree  (cleanex, H2O)               {triple2, SH[17](F1)}
; or   f1 channel - shaped pulse 180 degree  (med. selectivity)           {triple_c, SH[15](F1)}
; or   f2 channel - shaped pulse 180 degree  (refocussing (sharp))        {triple, SH[32](F2)}
;sp22: f1 channel - shaped pulse for wet                         {default, SH[7]*3.198(F1)}
; or   f1 channel - shaped pulse  90 degree  (cleanex, H2O)               {triple2, SH[16](F1)}
; or   f1 channel - shaped pulse 180 degree  (off resonance)              {triple_c, SH[8](F1)}
;sp23: f1 channel - shaped pulse  90 degree  (on resonance)               {triple_c, SH[6](F1)}
; or   f1 channel - shaped pulse 120 degree  (NH, best- )                 {triple, SH[8](F1)}
; or   f1 channel - shaped pulse 180 degree  (off resonance)              {triple_na, SH[19](F1)}
; or   f2 channel - shaped pulse 180 degree  (med. selectivity)           {triple2, SH[15](F2)}
;sp24: f1 channel - shaped pulse 180 degree  (on resonance)               {triple_c, SH[8](F1)}
; or   f1 channel - shaped pulse 180 degree  (NH, best-, I)               {triple, SH[9](F1)}
; or   f1 channel - shaped pulse 180 degree  (off resonance2)             {triple_na, SH[19](F1)}
; or   f2 channel - shaped pulse 180 degree  (high selectivity)           {triple2, SH[16](F2)}
;sp25: f1 channel - shaped pulse  90 degree  (on res., time reversed)     {triple_c, SH[7](F1)}
; or   f1 channel - shaped pulse  90 degree  (NH, best-, I)               {triple, SH[10](F1)}
; or   f2 channel - shaped pulse 180 degree  (higher selectivity)         {triple_na, SH[28](F2)}
; or   f2 channel - shaped pulse  90 degree  (high selectivity)           {triple2, SH[17](F2)}
;sp26: f1 channel - shaped pulse 180 degree  (off resonance)              {triple_c, SH[8](F1)}
; or   f1 channel - shaped pulse 180 degree  (NH, best-, II)              {triple, SH[12](F1)}
; or   f1 channel - shaped pulse 180 degree  (C, selective)               {default, SH[1](F1)}
; or   f2 channel - shaped pulse  90 degree  (high selectivity, tr)       {triple2, SH[18](F2)}
;sp27: f1 channel - shaped pulse 180 degree  (off resonance)              {triple_c, SH[8](F1)}
; or   f1 channel - shaped pulse  90 degree  (NH, best-, I tr)            {triple, SH[11](F1)}
; or   f2 channel - shaped pulse  90 degree  (high selectivity)           {triple2, SH[17](F2)}
;sp28: f1 channel - shaped pulse 180 degree  (higher selectivity)         {triple_c, SH[11](F1)}
; or   f1 channel - shaped pulse  90 degree  (NH, best-, II)              {triple, SH[13](F1)}
; or   f2 channel - shaped pulse 180 degree  (higher selectivity)         {triple2, SH[11](F2)}
;sp29: f1 channel - shaped pulse 180 degree  (off resonance)              {triple_c, SH[8](F1)}
; or   f1 channel - shaped pulse 180 degree  (adiabatic sweep: z-spoil)   {default, SH[18](F1)}
```

```
; or   f1 channel - shaped pulse  90 degree  (NH, best-, II tr)          {triple, SH[14](F1)}
; or   f2 channel - shaped pulse 180 degree  (high selectivity)          {triple2, SH[16](F2)}
;sp30: f1 channel - shaped pulse 180 degree  (sim. Ca + CO)              {triple_c, SH[21](F1)}
; or   f1 channel - shaped pulse 180 degree  (broadband, best-)          {triple, SH[15](F1)}
; or   f2 channel - shaped pulse 180 degree for decoupling (sim. Ca + CO)  {triple2, SH[21](F2)}
;sp31: f2 channel - shaped pulse 180 degree  (adiabatic bilev decoupling)  {default+triple+triple2+triple_na,
SH[12](F2)}
;
;
;
;
;p0 :                                              {all, PW90(F1)}
;p1 : f1 channel -  90 degree high power pulse                      {all, PW90(F1)}
;p2 : f1 channel - 180 degree high power pulse                      {all, PW90*2(F1)}
;p3 : f2 channel -  90 degree high power pulse                      {all, PW90(F2)}
;p4 : f2 channel - 180 degree high power pulse                      {all, PW90*2(F2)}
;p5 : f1 channel -  60 degree low power pulse                       {all, SQPW[1]*0.66(F1)}
;p6 : f1 channel -  90 degree low power pulse                       {all, SQPW[1](F1)}
;p7 : f1 channel - 180 degree low power pulse                       {default+lcnmr+triple+triple_c, SQPW[1]*2(F1)}
; or  f1 channel - 180 degree shaped pulse (cleanex sel. H2O)         {triple2, SHPW[17](F1)}
; or  f2 channel -  90 degree pulse at pl20 (TOCSY, higher sel.)      {triple_na, SQPW[7](F2)}
;p8 : f2 channel -  60 degree low power pulse                       {}
; or  f1 channel -  90 degree shaped pulse (wet)                      {default, SHPW[7](F1)}
; or  f1 channel - 180 degree shaped pulse (adiabatic)                {triple_c, SHPW[4](F1)}
; or  f2 channel - 180 degree shaped pulse (adiabatic)                {triple+triple2+triple_na, SHPW[4](F2)}
;p9 : f2 channel -  90 degree low power pulse (TOCSY)                 {all, SQPW[1](F2)}
;p10: f1 channel -  90 degree low power pulse (cleanex spinlock)      {triple2, SQPW[10](F1)}
; or  f2 channel - 180 degree low power pulse                       {default+lcnmr+triple+triple_c, SQPW[1]*2(F2)}
; or  f2 channel - 180 degree shaped pulse (higher selectivity)       {triple_na, SHPW[28](F2)}
;p11: f1 channel -  90 degree shaped pulse (selective excitation)     {default, SHPW[0](F1)}
; or  f1 channel -  90 degree shaped pulse (selective excitation)     {triple_c, SHPW[6](F1)}
; or  f1 channel -  90 degree shaped pulse (wet)                      {lcnmr, SHPW[7](F1)}
; or  f1 channel -  90 degree shaped pulse (water flipback/watergate)  {triple+triple2+triple_na, SHPW[5](F1)}
;p12: f1 channel - 180 degree shaped pulse (H, selective)             {default+lcnmr, SHPW[1](F1)}
; or  f1 channel - 180 degree shaped pulse (C, selective)             {triple_c, SHPW[8](F1)}
; or  f1 channel - 180 degree shaped pulse (excitation sculpting)     {triple+triple2, SHPW[5]*2(F1)}
; or  f1 channel - 180 degree shaped pulse (H, selective)             {triple_na, SHPW[19](F1)}
;p13: f1 channel - 180 degree shaped pulse (C, adiabatic)             {default+lcnmr, SHPW[5](F1)}
; or  f2 channel -  90 degree shaped pulse                          {triple+triple2, SHPW[6](F2)}
; or  f2 channel -  90 degree shaped pulse                          {triple_na, SHPW[23](F2)}
; or  f2 channel -  90 degree shaped pulse (H, selective)             {triple_c, SHPW[10](F2)}
;p14: f2 channel - 180 degree shaped pulse (adiabatic)                {default+lcnmr, SHPW[4](F2)}
; or  f2 channel - 180 degree shaped pulse (selective)                {triple+triple2, SHPW[8](F2)}
; or  f2 channel - 180 degree shaped pulse (selective)                {triple_na, SHPW[25](F2)}
;p15: f1 channel - pulse for ROESY spinlock                         {default+lcnmr, SQPW[2](F1)}
; or  f1 channel -  90 degree shaped pulse (cleanex sel. H2O)         {triple2, SHPW[16](F1)}
; or  f2 channel - 180 degree shaped pulse (adiabatic matched sweep)   {triple, SHPW[19](F2)}
; or  f2 channel -  90 degree shaped pulse (higher selectivity)       {triple_na, SHPW[26](F2)}
;p16: homospoil/gradient pulse                                    {all, P_grad1}
;p17: f1 channel - trim pulse at pl10 or pl15                        {all, P_mlev(F1)}
;p18: f1 channel - shaped pulse (off resonance presaturation)         {default+lcnmr+triple+triple2+triple_na,
SHPW[4](F1)}
;p19: homospoil/gradient pulse 2                                  {all, P_grad2}
;p20: f2 channel - trim pulse                                     {all, P_mlev(F2)}
```

;p21: f3 channel - 90 degree high power pulse {all, PW90(F3)}
;p22: f3 channel - 180 degree high power pulse {all, PW90*2(F3)}
;p23: f1 channel - 180 degree shaped pulse (med. selectivity) {triple_c, SHPW[15](F1)}
; or f2 channel - 90 degree shaped pulse (higher selectivity) {triple+triple2, SHPW[9](F2)}
; or f2 channel - 90 degree shaped pulse (twofold modulated) {triple_na, SHPW[29](F2)}
; or f4 channel - 90 degree high power pulse {default, PW90(F4)}
;p24: f1 channel - 180 degree shaped pulse (adiabatic) {triple_c, SHPW[5](F1)}
; or f2 channel - 180 degree shaped pulse (adiabatic) {default, SHPW[5](F2)}
; or f2 channel - 180 degree shaped pulse (higher selectivity) {triple+triple2, SHPW[11](F2)}
; or f3 channel - 90 degree pulse at pl22 (TOCSY, higher sel.) {triple_na, SQPW[9](F3)}
; or f4 channel - 180 degree high power pulse {}
;p25: f1 channel - 90 degree pulse at pl27 (pulsed ROESY) {default+lcnmr, SQPW[11]*2(F1)}
; or f1 channel - 90 degree shaped pulse (higher selectivity) {triple_c, SHPW[11](F1)}
; or f3 channel - pulse for t1rho experiment {pp}
; or f3 channel - pulse for TOCSY-spinlock experiment {triple, SQPW[1]}
; or f3 channel - 90 degree pulse at pl23 (TOCSY) {triple_na, SQPW[8](F3)}
; or f3 channel - 180 degree low power pulse (Rexchange) {triple2, SQPW[16]*2(F3)}
;p26: f1 channel - 90 degree pulse at pl19 {triple*, SQPW[0](F1)}
; or f1 channel - 180 degree shaped pulse (adiabatic) {default, SHPW[4](F1)}
;p27: f1 channel - 90 degree pulse at pl18 (3-9-19 watergate) {default+lcnmr+triple+triple2+triple_na, PW90(F1)}
;p28: f1 channel - trim pulse at pl1 {all, P_hsqc(F1)}
;p29: f1 channel - 90 degree shaped pulse (water flipback) {default, SHPW[5](F1)}
; or f1 channel - 90 degree shaped pulse (water flipback2) {triple+triple_na, SHPW[6](F1)}
; or f2 channel - 90 degree shaped pulse (water flipback) {triple_c, SHPW[5](F2)}
; or f3 channel - 90 degree shaped pulse (T1rho adiabatic ramp) {triple2, SHPW[6](F3)}
; or homospoil/gradient pulse 3 {pp}
;p30: f1 channel - 180 degree shaped pulse (sim. Ca + CO) {triple_c, SHPW[21](F1)}
; or f2 channel - 180 degree shaped pulse (sim. Ca + CO decoupling) {triple2, SHPW[21](F2)}
; or f3 channel - 180 degree pulse at pl23 (T2) {triple, SQPW[16]*2(F3)}
; or f3 channel - 180 degree shaped pulse {triple_na, SHPW[5](F3)}
; or homospoil/gradient pulse 4 {pp}
; or gradient pulse for diffusion (dosy) {}
;p31: f2 channel - 180 degree shaped pulse (adiabatic matched sweep) {default, SHPW[19](F2)}
; or f2 channel - 180 degree shaped pulse (sel. Ca or CO decoupling) {triple, SHPW[20](F2)}
; or f2 channel - 180 degree shaped pulse (Cbeta decoupling) {triple2, SHPW[22](F2)}
; or f2 channel - 180 degree shaped pulse (sel. C decoupling) {triple_na, SHPW[28](F2)}
; or f2 channel - 90 degree pulse (low power decoupling) {lcnmr, SQPW[5](F2)}
; or homospoil/gradient pulse 5 {pp}
;p32: f1 channel - 180 degree shaped pulse (adiabatic sweep: z-spoil) {default, SHPW[18](F1)}
; or f3 channel - 180 degree shaped pulse (adiabatic) {triple_na, SHPW[8](F3)}
;p33: f2 channel - 180 degree shaped pulse (med. selectivity) {triple2, SHPW[15](F2)}
; or f3 channel - trim pulse {triple_na, P_mlev(F3)}
;p34: f2 channel - 180 degree shaped pulse (high selectivity) {triple2, SHPW[16](F2)}
;p35: f2 channel - 90 degree shaped pulse (high selectivity) {triple2, SHPW[17](F2)}
;p36: f2 channel - 180 degree shaped pulse (C, selective) {default, SHPW[1](F2)}
;
;p39: f1 channel - 120 degree shaped pulse for excitation (best-) {triple, SHPW[8][F1}
; or f2 channel - 180 degree shaped pulse (short, broadband) {default, SHPW[14](F2)}
;p40: f1 channel - 180 degree shaped pulse for refocussing (best-) {triple, SHPW[9][F1}
; or f1 channel - 180 degree shaped pulse (excitation sculpting) {default, SHPW[5]*2(F1)}
;p41: f1 channel - 90 degree shaped pulse for refocussing (best-) {triple, SHPW[10](F1)}
;p42: f1 channel - 180 degree shaped pulse for refocussing (best-) {triple, SHPW[12](F1)}
;p43: f1 channel - 90 degree shaped pulse for refocussing (best-) {triple, SHPW[13](F1)}

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;p44: f1 channel - 180 degree shaped pulse for refocussing (best-)     {triple, SHPW[15](F1)}
;p45: f2 channel -  90 degree pulse at pl20 ((hetero) TOCSY high sel.) {triple, SQPW[8](F2)}
;p46: f2 channel -  90 degree pulse at pl20 ((hetero) TOCSY med. sel.) {triple, SQPW[7](F2)}
;p47: f2 channel - 180 degree shaped pulse for inversion (sharp)       {triple, SHPW[31](F2)}
;p48: f2 channel - 180 degree shaped pulse for refocussing (sharp)     {triple, SHPW[32](F2)}
;p49: f1 channel - 180 degree shaped pulse (H2O)                       {triple, SHPW[21](F1)}
;
;p61: f2 channel -  90 degree pulse (low power decoupling)           {triple+triple2, SQPW[5](F2)}
;p62: f3 channel -  90 degree pulse (low power decoupling)           {triple+triple2, SQPW[5](F3)}
;p63: f2 channel - 180 degree shaped pulse (adiabatic bilev sweep)     {default+triple+triple2+triple_na,
SHPW[12](F2)}
;
;
;
;
;d0 : incremented delay (2D or 3D)                 [3 usec]
;d1 : relaxation delay; 1-5 * T1
;d2 : 1/(2J)
;d3 : 1/(3J) or 1/(6J)
;d4 : 1/(4J)
;d5 : DE/2
;d6 : delay for evolution of long range couplings
;d7 : delay for inversion recovery
;d8 : NOESY mixing time
;d9 : TOCSY mixing time                                {all, TTOC(F1)}
;d10: incremented delay (3D)
;d11: delay for disk I/O                      [30 msec]
;d12: delay for power switching                   [20 usec]
;d13: short delay                           [4 usec]
;d14: delay for evolution after shaped pulse
;d15: TOCSY mixing time (CC)                           {triple*, TTOC(F2)}
;d16: delay for homospoil/gradient recovery                   {all, D_grad}
;d17: delay for DANTE pulse-train
;d18: delay for evolution of long range couplings
;d19: delay for binomial water suppression
;d20: for different applications
;d21: for different applications
;d22: 1/(2J(XY))
;d23: 1/(4J(XY)) or 1/(2J(XY))
;d24: for different applications
;d25: 1/(6J(YH)) or 1/(8J(XY))
;d26: 1/(4J(YH))
;d27: for different applications
;d28: for different applications
;d29: for different applications
;d30: for different applications
;d31: incremented delay (> 3D, t1)
;d32: incremented delay (> 3D, t2)
;d33: incremented delay (> 3D, t3)
;d34: incremented delay (> 3D, t4)
;d35: incremented delay (> 3D, t5)
;d41: decremented delay (> 3D, t1)
;d42: decremented delay (> 3D, t2)
;d43: decremented delay (> 3D, t3)
```



```
;d44: decremented delay (> 3D, t4)
;d45: decremented delay (> 3D, t5)
;d51: incremented delay (> 3D, t1)
;d52: incremented delay (> 3D, t2)
;d53: incremented delay (> 3D, t3)
;d54: incremented delay (> 3D, t4)
;d55: incremented delay (> 3D, t5)
;
;
;
;
;cnst0 : for protein experiments - N chemical shift (offset, in ppm)
; or     for na experiments - calculated chemical shift (offset, in ppm)
; or     for na experiments - N(aro) chemical shift (offset, in ppm)        [195 ppm]
;cnst1 : J (HH)
;cnst2 : J (XH)
;cnst3 : J (XX)
;cnst4 : J (YH)
;cnst5 : J (XY)
;cnst6 : J (XH)min
;cnst7 : J (XH)max
;cnst8 : bandwidth of excitation for Dante-z pulse
;cnst9 : for different applications as J
;cnst10: for different applications as J
;cnst11: for multiplicity selection
;cnst12: for multiplicity selection
;cnst13: J (XH) long range
;cnst14: J (XH) long range (min)
;cnst15: J (XH) long range (max)
;cnst16: J-scale factor
; or     for na experiments - H6/8 and/or H1' chemical shift (offset, in ppm)
;cnst17: factor to compensate for coupling evolution during a pulse
; or     for na experiments - H1' chemical shift (offset, in ppm)
;cnst18: for protein experiments - H2O chemical shift (offset, in ppm)
; or     for na experiments - H2O chemical shift (offset, in ppm)
;cnst19: for protein experiments - H(N) chemical shift (offset, in ppm)
; or    : for na experiments - H(N) chemical shift (offset, in ppm)
;cnst20: for protein experiments - Haliphatic chemical shift (offset, in ppm)
;cnst21: for na experiments - C1' chemical shift (offset, in ppm)          [90 ppm]
; or     for protein experiments - CO chemical shift (offset, in ppm)
;cnst22: for protein experiments - Calpha chemical shift (offset, in ppm)
; or     for na experiments - C6/8 chemical shift (offset, in ppm)         [137 ppm]
;cnst23: for protein experiments - Caliphatic chemical shift (offset, in ppm)
; or     for na experiments - C2' chemical shift (offset, in ppm)          [72 ppm]
;cnst24: for protein experiments - Caromatic chemical shift (offset, in ppm)
; or     for na experiments - C4 (C/U) chemical shift (offset, in ppm)      [169 ppm]
;cnst25: for protein experiments - flag for cross peak / reference experiments
; or     for na experiments - C6 (A) chemical shift (offset, in ppm)        [160 ppm]
;cnst26: for protein experiments - Call chemical shift (offset, in ppm)
; or     for na experiments - C5 (G) chemical shift (offset, in ppm)        [119 ppm]
;cnst27: for protein experiments - ( Cgamma chemical shift (offset, in ppm) )
; or     for na experiments - C2/4 chemical shift (offset, in ppm)          [152 ppm]
;cnst28: for protein experiments - Haromatic chemical shift (offset, in ppm)
; or     for na experiments - C5 (C/U) chemical shift (offset, in ppm)      [105 ppm]
```

```
;cnst29: for protein experiments - N(H) chemical shift (offset, in ppm)
; or     for na experiments - C(aro) chemical shift (offset, in ppm)        [145 ppm]
;cnst30: for protein experiments - Cbeta chemical shift (offset, in ppm)
; or     for na experiments - N(H) chemical shift (offset, in ppm)          [151 ppm]
;cnst31: scaling factor
; or     for na experiments - N(H2) chemical shift (offset, in ppm)         [81 ppm]
;cnst34: for music experiments - Cab(Leu) chemical shift (offset, in ppm)    [48.0 ppm]
;cnst35: for music experiments - Cbgd(Leu) chemical shift (offset, in ppm)   [35.0 ppm]
;cnst36: for music experiments - Cgd(Leu) chemical shift (offset, in ppm)    [19.0 ppm]
;cnst37: for music experiments - Cb(Val)/Cg(Ile) chem. shift (offset,in ppm) [27.0 ppm]
;cnst38: for music experiments - Cab(Asp)Cd(Arg) chem. shift (offset,in ppm) [42.0 ppm]
;cnst39: for music experiments - Cg(Arg)/Cd(Lys) chem. shift (offset,in ppm) [29 ppm]
;cnst40: compensation of chemical shift evolution during p42 (best-)
; or     for music experiments - Ce(Lys) chemical shift (offset, in ppm)     [44.5 ppm]
;cnst41: compensation of chemical shift evolution during p41 (best-)
; or     for music experiments - Cg(Phe,Tyr,His) chem. shift (offset, in ppm)[136.0 ppm]
;cnst42: compensation of chemical shift evolution during p42 (best-)
; or     for music experiments - Cg(Trp) chemical shift (offset, in ppm)     [104.0 ppm]
;cnst43: compensation of chemical shift evolution during p43 (best-)
; or     for music experiments - Cb(Thr) chemical shift (offset, in ppm)     [68.5 ppm]
;cnst44: for music experiments - Cab(Ser) chemical shift (offset, in ppm)    [60.5 ppm]
;cnst45: for music experiments - Cd(Ile) chemical shift (offset, in ppm)     [11.0 ppm]
;cnst46: for music experiments - Cbg(Ile) chemical shift (offset, in ppm)    [33.0 ppm]
;cnst47: for music experiments - N(H) chemical shift (offset, in ppm)        [117.0 ppm]
;cnst48: for music experiments - N(Pro) chemical shift (offset, in ppm)      [134.0 ppm]
;cnst49: for protein experiments - H(methyl) chemical shift (offset, in ppm) [0.5 ppm]
;
;
;
;
;l1 : loop for spinlock cycle
;l2 : loop for GARP cycle: l2 * 31.75 * 4 * p9 => AQ
;l3 : loop for phase sensitive 2D or 3D using
;      States et al. or States-TPPI method: l3 = td1/2
;l4 : for different applications
;l5 : for different applications
;l6 : loop for shaped pulse presaturation during relaxation delay
;l7 : loop for shaped pulse presaturation during mixing time
;l8 : number of frequencies for multiple presaturation
;l11: loop for spinlock cycle 2
;
;
;
;
;vc : variable loop counter, taken from vc-list
;vd : variable delay, taken from vd-list


;$Id: $
```



```
;Relations.info
;avance-version (10/02/01)
;
;$CLASS=HighRes Info
;$COMMENT=
;The following convention is used for power levels, pulses, delays
;and loop counters in the different relation files for prosol:
;
;all = default + lcnmr + triple + triple2 + triple_c + triple_na
;triple* = triple + triple2 + triple_c + triple_na
;!__ = except
;
;prosol par.    rel. file           pulseprogram parameter
;
;DE             all                 de
;D_grad         all                 d16: delay for homospoil/gradient recovery
;
;PW90(F1)       all                 p0 :
;PW90(F1)       all                 p1 : f1 channel -  90 degree high power pulse
;PW90(F1)       all(!triple_c)      p27: f1 channel -  90 degree pulse at pl18 (3-9-19 watergate)
;PW90*2(F1)     all                 p2 : f1 channel - 180 degree high power pulse
;PW90(F2)       all                 p3 : f2 channel -  90 degree high power pulse
;PW90*2(F2)     all                 p4 : f2 channel - 180 degree high power pulse
;PW90(F3)       all                 p21: f3 channel -  90 degree high power pulse
;PW90*2(F3)     all                 p22: f3 channel - 180 degree high power pulse
;PW90(F4)       default             p23: f4 channel -  90 degree high power pulse
;
;PL90(F1)       all                 pl1 : f1 channel - power level for pulse (default)
;PL90(F1)       all(!triple_c)      pl18: f1 channel - power level for 3-9-19-pulse (watergate)
;PL90(F2)       all                 pl2 : f2 channel - power level for pulse (default)
;PL90(F3)       all                 pl3 : f3 channel - power level for pulse (default)
;PL90(F4)       all                 pl4 : f4 channel - power level for pulse (default)
;
;SH[0](F1)      default             sp1 : f1 channel - shaped pulse for selective excitation
;SH[1](F1)      default             sp2 : f1 channel - shaped pulse 180 degree
;SH[1](F2)      default             sp26: f2 channel - shaped pulse 180 degree
;SH[4](F1)      all(!triple+triple_c)  sp6 : f1 channel - shaped pulse for presaturation
;SH[4](F1)      default             sp8 : f1 channel - shaped pulse 180 degree  (adiabatic)
;SH[4](F1)      triple_c            sp13: f1 channel - shaped pulse 180 degree  (adiabatic)
;SH[4](F2)      default+lcnmr       sp3 : f2 channel - shaped pulse 180 degree  (adiabatic)
;SH[4](F2)      triple+triple2+triple_na sp13: f2 channel - shaped pulse 180 degree  (adiabatic)
;SH[5](F1)      triple+triple2+triple_na sp1 : f1 channel - shaped pulse for water flipback
;SH[5](F1)      default             sp5 : f1 channel - shaped pulse 180 degree  (adiabatic)
;SH[5](F1)      triple_c            sp7 : f1 channel - shaped pulse 180 degree  (adiabatic)
;SH[5](F1)      default             sp10: f1 channel - shaped pulse for water flipback
;SH[5](F1)      default+lcnmr       sp11: f1 channel - shaped pulse for water flipback
;SH[5](F2)      default             sp7 : f2 channel - shaped pulse 180 degree  (adiabatic)
;SH[5](F2)      triple_c            sp11: f2 channel - shaped pulse  90 degree (water flipback)
;SH[6](F1)      triple+triple2+triple_na sp11: f1 channel - shaped pulse for water flipback2
;SH[6](F1)      triple_c            sp23: f1 channel - shaped pulse  90 degree  (on resonance)
;SH[6](F2)      triple+triple2      sp2 : f2 channel - shaped pulse  90 degree  (on resonance)
;SH[6](F2)      triple+triple2      sp4 : f2 channel - shaped pulse  90 degree  (off resonance)
;SH[6](F3)      triple2             sp19: f3 channel - shaped pulse  90 degree (T1rho, adiab. ramp up)
```

```
;SH[7](F1)        lcnmr              sp1 : f1 channel - shaped pulse for wet
;SH[7](F1)        lcnmr              sp2 : f1 channel - shaped pulse for wet
;SH[7]*0.817(F1)  lcnmr              sp7 : f1 channel - shaped pulse for wet
;SH[7]*0.817(F1)  default            sp19 : f1 channel - shaped pulse for wet
;SH[7]*1.270(F1)  default            sp20: f1 channel - shaped pulse for wet
;SH[7]*0.593(F1)  default            sp21: f1 channel - shaped pulse for wet
;SH[7]*3.198(F1)  default            sp22: f1 channel - shaped pulse for wet
;SH[7](F1)        triple_c           sp25: f1 channel - shaped pulse  90 degree  (on resonance)
;SH[7](F2)        triple             sp6 : f2 channel - shaped pulse  90 degree  (off res., time reversed)
;SH[7](F2)        triple+triple2     sp8 : f2 channel - shaped pulse  90 degree  (on res., time reversed)
;SH[7](F3)        triple2            sp20: f3 channel - shaped pulse  90 degree (T1rho, adiab. ramp down)
;SH[8](F1)        triple_c           sp22: f1 channel - shaped pulse 180 degree  (off resonance)
;SH[8](F1)        triple             sp23: f1 channel - shaped pulse 120 degree  (NH, best- )
;SH[8](F1)        triple_c           sp24: f1 channel - shaped pulse 180 degree  (on resonance)
;SH[8](F1)        triple_c           sp26: f1 channel - shaped pulse 180 degree  (off resonance)
;SH[8](F1)        triple_c           sp27: f1 channel - shaped pulse 180 degree  (off resonance)
;SH[8](F1)        triple_c           sp29: f1 channel - shaped pulse 180 degree  (off resonance)
;SH[8](F2)        triple+triple2     sp3 : f2 channel - shaped pulse 180 degree  (on resonance)
;SH[8](F2)        triple+triple2     sp5 : f2 channel - shaped pulse 180 degree  (off resonance)
;SH[8](F2)        triple+triple2     sp7 : f2 channel - shaped pulse 180 degree  (off resonance2)
;SH[8](F2)        triple             sp20: f2 channel - shaped pulse 180 degree  (off resonance3)
;SH[8](F3)        triple_na          sp14: f3 channel - shaped pulse 180 degree (_NA: N, adiabatic)
;SH[9](F1)        triple             sp24: f1 channel - shaped pulse 180 degree  (NH, best-, I)
;SH[9](F2)        triple+triple2     sp10: f2 channel - shaped pulse  90 degree  (higher selectivity)
;SH[9](F3)        triple_na          sp9 : f3 channel - shaped pulse 180 degree (_NA: N)
;SH[10](F2)       triple+triple2     sp12: f2 channel - shaped pulse  90 degree  (higher sel., time rev.)
;SH[10](F2)       triple_c           sp19: f2 channel - shaped pulse  90 degree (HN)
;SH[10](F1)       triple             sp25: f1 channel - shaped pulse  90 degree  (NH, best-, I)
;SH[11](F1)       triple             sp27: f1 channel - shaped pulse  90 degree  (NH, best-, I tr)
;SH[11](F1)       triple_c           sp28: f1 channel - shaped pulse 180 degree  (higher selectivity)
;SH[11](F2)       triple+triple2     sp9 : f2 channel - shaped pulse 180 degree  (higher selectivity)
;SH[11](F2)       triple+triple2     sp16: f2 channel - shaped pulse 180 degree  (higher selectivity)
;SH[11](F2)       triple+triple2     sp17: f2 channel - shaped pulse 180 degree  (higher selectivity)
;SH[11](F2)       triple_c           sp20: f2 channel - shaped pulse  90 degree (HN tr)
;SH[11](F2)       triple2            sp28: f2 channel - shaped pulse 180 degree  (higher selectivity)
;SH[12](F1)       triple             sp26: f1 channel - shaped pulse 180 degree  (NH, best-, II)
;SH[12](F2)       default+triple+triple2   sp31: f2 channel - shaped pulse 180 degree (adiabatic decoupling)
;                 +triple_na
;SH[13](F1)       triple             sp28: f1 channel - shaped pulse  90 degree  (NH, best-, II)
;SH[13](F2)       default+triple+triple2   sp14: f2 channel - shaped pulse 180 degree (adiabatic bilev decoupling)
;SH[14](F1)       triple             sp29: f1 channel - shaped pulse  90 degree  (NH, best-, II tr)
;SH[14](F2)       default+lcnmr      sp4 : f2 channel - shaped pulse 180 degree  (short, broadband)
;SH[15](F1)       triple             sp30: f1 channel - shaped pulse 180 degree  (broadband, best-)
;SH[15](F1)       triple_c           sp21: f1 channel - shaped pulse 180 degree  (med. selectivity)
;SH[15](F2)       triple2            sp23: f2 channel - shaped pulse 180 degree  (med. selectivity)
;SH[16](F1)       triple2            sp22: f1 channel - shaped pulse  90 degree (H2O on resonance)
;SH[16](F2)       triple2            sp24: f2 channel - shaped pulse 180 degree  (high selectivity)
;SH[16](F2)       triple2            sp29: f2 channel - shaped pulse 180 degree  (high selectivity)
;SH[17](F1)       triple2            sp21: f1 channel - shaped pulse 180 degree (H2O on resonance)
;SH[17](F2)       triple2            sp25: f2 channel - shaped pulse  90 degree  (high selectivity)
;SH[17](F2)       triple2            sp27: f2 channel - shaped pulse  90 degree  (high selectivity)
;SH[18](F1)       default            sp29: f1 channel - shaped pulse 180 degree  (adiabatic: z-spoil)
;SH[18](F2)       triple2            sp26: f2 channel - shaped pulse  90 degree  (high selectivity, tr)
;SH[19](F1)       triple_na          sp23: f1 channel - shaped pulse 180 degree (_NA: H)
;SH[19](F1)       triple_na          sp24: f1 channel - shaped pulse 180 degree (_NA: H)
```



```
;SH[19](F2)     default+triple          sp18: f2 channel - shaped pulse 180 degree  (adiabatic matched sweep)
;SH[20](F2)     triple                  sp15: f2 channel - shaped pulse 180 degree for decoupling (Ca or CO)
;SH[21](F1)     triple_c                sp30: f1 channel - shaped pulse 180 degree  (sim. Ca + CO decoupling)
;SH[21](F1)     triple                  sp32: f1 channel - shaped pulse 180 degree  (H2O)
;SH[21](F2)     triple2                 sp30: f2 channel - power level for simultaneous Ca and CO decoupling
;SH[22](F2)     triple2                 sp15: f2 channel - shaped pulse 180 degree for decoupling (Cbeta)
;SH[23](F2)     triple_na               sp2 : f2 channel - shaped pulse  90 degree (_NA: C)
;SH[24](F2)     triple_na               sp8 : f2 channel - shaped pulse  90 degree (_NA: C, tr)
;SH[25](F2)     triple_na               sp3 : f2 channel - shaped pulse 180 degree (_NA: C)
;SH[25](F2)     triple_na               sp5 : f2 channel - shaped pulse 180 degree (_NA: C)
;SH[25](F2)     triple_na               sp7 : f2 channel - shaped pulse 180 degree (_NA: C)
;SH[26](F2)     triple_na               sp10: f2 channel - shaped pulse  90 degree (_NA: C, higher sel.)
;SH[27](F2)     triple_na               sp12: f2 channel - shaped pulse  90 degree (_NA: C, higher sel., tr)
;SH[28](F2)     triple_na               sp15: f2 channel - shaped pulse 180 degree (_NA: C, decoupling)
;SH[28](F2)     triple_na               sp25: f2 channel - shaped pulse 180 degree (_NA: C, higher sel.)
;SH[29](F2)     triple_na               sp0 : f2 channel - shaped pulse 180 degree (_NA: C, twofold mod)
;SH[31](F2)     triple                  sp19: f2 channel - shaped pulse 180 degree (inversion (sharp))
;SH[32](F2)     triple                  sp21: f2 channel - shaped pulse 180 degree (refocussing (sharp))
;
;SHPL[7]*1.270(F1) lcnmr                sp8 : f1 channel - shaped pulse for wet
;SHPL[7]*0.593(F1) lcnmr                sp9 : f1 channel - shaped pulse for wet
;SHPL[7]*3.198(F1) lcnmr                sp10: f1 channel - shaped pulse for wet
;SHPL[20](F2)   default+lcnmr           pl25: f2 channel - shaped pulse (Eretic)
;SHPL[20](F2)   triple+triple2          pl28: f2 channel - power level for selective Ca or CO decoupling
;SHPL[21](F2)   triple2                 pl29: f2 channel - power level for simultaneous Ca and CO decoupling
;SHPL[22](F2)   triple+triple2          pl13: f2 channel - power level for Cbeta/CO decoupling
;SHPL[28](F2)   triple_na               pl28: f2 channel - shaped pulse 180 degree (_NA: C, decoupling)
;
;SHPW[0](F1)    default                 p11: f1 channel -  90 degree shaped pulse (selective excitation)
;SHPW[1](F1)    default+lcnmr           p12: f1 channel - 180 degree shaped pulse (H, selective)
;SHPW[1](F2)    default                 p36: f2 channel - 180 degree shaped pulse (C, selective)
;SHPW[4](F1)    triple_c                p8 : f1 channel - 180 degree shaped pulse (adiabatic)
;SHPW[4](F1)    all(!triple_c)          p18: f1 channel - shaped pulse (off resonance presaturation)
;SHPW[4](F2)    triple+triple2+triple_na   p8 : f2 channel - 180 degree shaped pulse (adiabatic)
;SHPW[4](F2)    default+lcnmr           p14: f2 channel - 180 degree shaped pulse (adiabatic)
;SHPW[4](F1)    default                 p26: f1 channel - 180 degree shaped pulse (adiabatic)
;SHPW[5](F1)    triple+triple2+triple_na   p11: f1 channel -  90 degree shaped pulse (water flipback/watergate)
;SHPW[5]*2(F1)  triple+triple2          p12: f1 channel - 180 degree shaped pulse (excitation sculpting)
;SHPW[5](F1)    default+lcnmr           p13: f1 channel - 180 degree shaped pulse (C, adiabatic)
;SHPW[5](F1)    triple_c                p24: f1 channel - 180 degree shaped pulse (adiabatic)
;SHPW[5](F1)    default                 p29: f1 channel -  90 degree shaped pulse (water flipback)
;SHPW[5]*2(F1)  default                 p40: f1 channel - 180 degree shaped pulse (excitation sculpting)
;SHPW[5](F2)    default+lcnmr           p24: f2 channel - 180 degree shaped pulse (adiabatic)
;SHPW[5](F2)    triple_c                p29: f2 channel -  90 degree shaped pulse for inversion (water flipback)
;SHPW[5](F3)    triple_na               p30: f3 channel - 180 degree shaped pulse (_NA: N)
;SHPW[6](F1)    triple_c                p11: f1 channel -  90 degree shaped pulse
;SHPW[6](F1)    triple+triple_na        p29: f1 channel -  90 degree shaped pulse (water flipback2)
;SHPW[6](F2)    triple+triple2          p13: f2 channel -  90 degree shaped pulse
;SHPW[6](F3)    triple2                 p29: f3 channel - shaped pulse for adiabatic ramping
;SHPW[7](F1)    default                 p8 : f1 channel -  90 degree shaped pulse (wet)
;SHPW[7](F1)    lcnmr                   p11: f1 channel -  90 degree shaped pulse (wet)
;SHPW[8](F1)    triple_c                p12: f1 channel - 180 degree shaped pulse (selective)
;SHPW[8](F1)    triple                  p39: f1 channel - 120 degree shaped pulse (NH, best-)
;SHPW[8](F2)    triple+triple2          p14: f2 channel - 180 degree shaped pulse (selective)
;SHPW[8](F3)    triple_na               p32: f3 channel - 180 degree shaped pulse (_NA: N, adiabatic)
```

;SHPW[9](F1) triple p40: f1 channel - 180 degree shaped pulse (NH, best-, I)
;SHPW[9](F2) triple+triple2 p23: f2 channel - 90 degree shaped pulse (higher selectivity)
;SHPW[10](F1) triple p41: f1 channel - 90 degree shaped pulse (NH, best-, I)
;SHPW[10](F2) triple_c p13: f2 channel - 90 degree shaped pulse (H, selective)
;SHPW[11](F1) triple_c p25: f1 channel - 180 degree shaped pulse (higher selectivity)
;SHPW[11](F2) triple+triple2 p24: f2 channel - 180 degree shaped pulse (higher selectivity)
;SHPW[12](F1) triple p42: f1 channel - 180 degree shaped pulse (NH, best-, II)
;SHPW[12](F2) default+triple+triple2 p63 : f2 channel - 180 degree shaped pulse (adiabatic decoupling)
;SHPW[13](F1) triple p43: f1 channel - 90 degree shaped pulse (NH, best-, II)
;SHPW[14](F2) default p39: f2 channel - 180 degree shaped pulse (short, broadband)
;SHPW[15](F1) triple_c p23: f1 channel - 180 degree shaped pulse (med. selectivity)
;SHPW[15](F1) triple p44: f1 channel - 180 degree shaped pulse (broadband, best-)
;SHPW[15](F2) triple2 p33: f2 channel - 180 degree shaped pulse (med. selectivity)
;SHPW[16](F1) triple2 p15: f1 channel - 90 degree shaped pulse (H2O on resonance)
;SHPW[16](F2) triple2 p34: f2 channel - 180 degree shaped pulse (high selectivity)
;SHPW[17](F1) triple2 p7 : f1 channel - 180 degree shaped pulse (H2O on resonance, cleanex))
;SHPW[17](F2) triple2 p35: f2 channel - 90 degree shaped pulse (high selectivity)
;SHPW[18](F1) default p32: f1 channel - 180 degree shaped pulse for inversion (adiabatic: z-spoil)
;SHPW[19](F1) triple_na p12: f1 channel - 180 degree shaped pulse (_NA: H)
;SHPW[19](F1) default p31: f1 channel - 180 degree shaped pulse for inversion (adiabatic matched sweep)
;SHPW[19](F2) triple p15: f2 channel - 180 degree shaped pulse for inversion (adiabatic matched sweep)
;SHPW[20](F2) triple p31: f2 channel - 180 degree shaped pulse (sel. Ca or CO decoupling)
; +triple_na
;SHPW[21](F1) triple_c p30: f1 channel - 180 degree shaped pulse (sim. Ca + CO decoupling)
;SHPW[21](F1) triple p49: f1 channel - 180 degree shaped pulse (H2O)
;SHPW[21](F2) triple2 p30: f2 channel - 180 degree shaped pulse (sim. Ca + CO decoupling)
;SHPW[22](F2) triple2 p31: f2 channel - 180 degree shaped pulse (Cbeta decoupling)
;SHPW[22](F2) triple* pcpd8: f2 channel - 180 degree shaped pulse (Cbeta decoupling)
;SHPW[23](F2) triple_na p13: f2 channel - 90 degree shaped pulse (_NA: C)
;SHPW[25](F2) triple_na p14: f2 channel - 180 degree shaped pulse (_NA: C)
;SHPW[26](F2) triple_na p15: f2 channel - 90 degree shaped pulse (_NA: C, higher sel.)
;SHPW[28](F2) triple_na p10: f2 channel - 180 degree shaped pulse (_NA: C, higher sel.)
;SHPW[28](F2) triple_na p31: f2 channel - 180 degree shaped pulse (_NA: C, decoupling)
;SHPW[29](F2) triple_na p23: f2 channel - 180 degree shaped pulse (_NA: C, twofold mod)
;SHPW[31](F2) triple p47: f2 channel - shaped pulse 180 degree (inversion (sharp))
;SHPW[32](F2) triple p48: f2 channel - shaped pulse 180 degree (refocussing (sharp))
;
;SQPL[0](F1) all(!triple_c) pl19: f1 channel - power level for CPD/BB decoupling
;SQPL[0](F2) all pl12: f2 channel - power level for CPD/BB decoupling
;SQPL[0](F2) default+triple+triple2 pl30: f2 channel - power level for CPD/BB decoupling
; +triple_na
;SQPL[0](F3) all pl16: f3 channel - power level for CPD/BB decoupling
;SQPL[0](F4) all pl17: f4 channel - power level for CPD/BB decoupling
;SQPL[1](F1) all pl10: f1 channel - power level for TOCSY-spinlock
;SQPL[1](F1) default pl29: f1 channel - power level for TOCSY-spinlock (STD SL filter)
;SQPL[1](F2) all pl15: f2 channel - power level for TOCSY-spinlock
;SQPL[1](F3) default+lcnmr+triple_c pl23: f3 channel - power level for TOCSY-spinlock
;SQPL[1](F3) triple pl27: f3 channel - power level for TOCSY-spinlock
;SQPL[2](F1) all pl11: f1 channel - power level for ROESY-spinlock
;SQPL[3](F1) all(!triple_c) pl9 : f1 channel - power level for presaturation
;SQPL[3](F2) default+lcnmr pl21: f2 channel - power level for presaturation
;SQPL[3](F3) lcnmr pl22: f3 channel - power level for presaturation
;SQPL[4](F2) default+lcnmr+triple_c pl13: f2 channel - power level for second CPD/BB decoupling

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;SQPL[5](F2)      lcnmr+triple+triple2     pl14: f2 channel - power level for low power decoupling
;SQPL[5](F3)      triple                   pl26: f3 channel - power level for low power decoupling
;SQPL[6](F2)      default+triple+triple2   pl31: f2 channel - power level for bilev dec. (cw part)
;                 +triple_na
;SQPL[7](F1)      triple                   pl21: f1 channel - power level for TOCSY med. sel.
;SQPL[7](F2)      triple_na                pl20: f2 channel - power level for TOCSY med. sel.
;SQPL[7](F2)      triple                   pl22: f2 channel - power level for TOCSY med. sel.
;SQPL[8](F1)      triple_na                pl25: f1 channel - power level for hetero TOCSY
;SQPL[8](F2)      triple                   pl20: f2 channel - power level for hetero TOCSY
;SQPL[8](F2)      triple_na                pl26: f2 channel - power level for hetero TOCSY
;SQPL[8](F3)      triple_na                pl23: f3 channel - power level for hetero TOCSY
;SQPL[9](F2)      triple_na                pl27: f2 channel - power level for hetero TOCSY higher sel.
;SQPL[9](F3)      triple_na                pl22: f3 channel - power level for hetero TOCSY higher sel.
;SQPL[10](F1)     triple2                  pl27: f1 channel - power level for CLEANEX spinlock
;SQPL[11](F1)     default                  pl27: f1 channel - power level for pulsed ROESY-spinlock
;SQPL[12](F1)     all(!triple_c)           pl32: f1 channel - power level for low power presaturation
;SQPL[12](F2)     lcnmr                    pl26: f2 channel - power level for cw decoupling
;SQPL[13](F2)     default                  pl14: f2 channel - power level for cw saturation
;SQPL[14](F2)     all                      pl24: f2 channel - power level for hd/hc decoupling
;SQPL[15](F3)     triple*                  pl25: f3 channel - power level for T1rho spinlock
;SQPL[16](F3)     triple+triple2           pl23: f3 channel - power level for Rexchange/T2
;
;SQPW[0](F1)      triple+triple2+triple_na p26: f1 channel -  90 degree pulse at pl19
;SQPW[0](F1)      all                      pcpd1: f1 channel -  90 degree pulse for CPD decoupling
;SQPW[0](F2)      all                      pcpd2: f2 channel -  90 degree pulse for CPD decoupling
;SQPW[0](F3)      all                      pcpd3: f3 channel -  90 degree pulse for CPD decoupling
;SQPW[0](F4)      triple*                  pcpd4: f4 channel -  90 degree pulse for CPD decoupling
;SQPW[1](F1)      all                      p6 : f1 channel -  90 degree low power pulse
;SQPW[1]*0.66(F1)  all(!triple_na)         p5 : f1 channel -  60 degree low power pulse
;SQPW[1]*2(F1)    all(!triple2+triple_na)  p7 : f1 channel - 180 degree low power pulse
;SQPW[1](F2)      all                      p9 : f2 channel -  90 degree low power pulse
;SQPW[1]*2(F2)    all(!triple2+triple_na)  p10: f2 channel - 180 degree low power pulse
;SQPW[1](F3)      triple                   p25: f3 channel -  90 degree pulse at pl23
;SQPW[2](F1)      default+lcnmr            p15: f1 channel - pulse for ROESY spinlock
;SQPW[5](F2)      lcnmr                    p31: f2 channel -  90 degree low power pulse (decoupling)
;SQPW[5](F2)      triple+triple2           p61: f2 channel -  90 degree low power pulse (decoupling)
;SQPW[5](F3)      triple+triple2           p62: f3 channel -  90 degree low power pulse (decoupling)
;SQPW[7](F2)      triple_na                p7 : f2 channel -  90 degree low power pulse (TOCSY med. sel.)
;SQPW[7](F2)      triple                   p46: f2 channel -  90 degree low power pulse (TOCSY med. sel.)
;SQPW[8](F2)      triple                   p45: f2 channel -  90 degree low power pulse ((hetero) TOCSY high sel.)
;SQPW[8](F3)      triple_na                p25: f3 channel -  90 degree low power pulse ((hetero) TOCSY high sel.)
;SQPW[9](F3)      triple_na                p24: f3 channel -  90 degree low power pulse ((hetero) TOCSY very high
sel.)
;SQPW[10](F1)     triple2                  p10: f1 channel - 180 degree low power pulse (CLEANEX spinlock)
;SQPW[11](F1)*2   default+lcnmr            p25: f1 channel -  90 degree pulse at pl27 (pulsed ROESY)
;SQPW[16]*2(F3)   triple                   p30: f3 channel - 180 degree pulse at pl23 (T2)
;SQPW[16](F3)*2   triple2                  p25: f3 channel - 180 degree low power pulse (Rexchange)
;
;P_grad1          all                      p16: homospoil/gradient pulse
;P_grad2          all                      p19: homospoil/gradient pulse 2
;P_hsqc           all                      p28: f1 channel -  trim pulse at pl1
;P_mlev           all                      p17: f1 channel - trim pulse at pl10
;P_mlev           all                      p20: f2 channel - trim pulse at pl15
;P_mlev           triple_na                p33: f3 channel - trim pulse at pl23
```

Complete list of pulse programs/files included into the $HOME/exp/stan/nmr/lists/pp directory (Topspin v3.0)

Avance.incl
Daz.incl
De.incl
Delay.incl
Grad.incl
Param.info
Pulprog.info
README
Relations.info
Sysconf.incl
Update.info
adeq11etgpjcrdsp
adeq11etgprdsp.2
adeq11etgprdsp
adeq11etgpsp
adeq1netgp
adeqn1etgp
adeqnnetgp
aptjc
apt
aring2
aring
atocsygpph19
b_hncacbgp3d
b_hncacbigp3d
b_hncacogp3d
b_hncacogp4d
b_hncagp3d
b_hncaigp3d
b_hncocacbgp3d
b_hncocacbgp4d
b_hncocagp3d
b_hncocagp4d
b_hncogp3d
b_hncoigp3d
b_hsqcetf3gpsi
b_trhncacbgp3d
b_trhncacbigp3d
b_trhncacogp3d
b_trhncagp3d
b_trhncaigp3d
b_trhncocacbgp3d
b_trhncocagp3d
b_trhncogp3d
b_trhncoigp3d
b_trosyetf3gpsi
b_trosyf3gpph
c_caco_ia
c_caco_s3
c_caco
c_can_iasq
c_can_mq.2
c_can_mq
c_canco_ia3d.2
c_canco_ia3d
c_cancoi_ia3d
c_cbcaco_ia3d
c_cbcaco_s33d
c_cbcacon_ia3d
c_cbcanco_ia3d
c_ccco_ia3d
c_ccco_s33d
c_cccon_ia3d
c_ccflopsy16_ctia
c_ccflopsy16_ct
c_ccflopsy16_ia
c_ccflopsy16
c_ccnoesy2
c_ccnoesy_ct
c_ccnoesy
c_coca_ia
c_coca_mq.2
c_coca_mq
c_coca
c_con_iasq
c_con_mqia
c_con_mq
c_con_sq
c_cosy2_ct
c_cosy_ct
c_cosy
c_hacaco_3d
c_hcaco_ia3d
c_hcaco_s33d
c_hcan_ia3d
c_hcanco_ia3d
c_hcancoi_ia3d
c_hcbca_ia3d
c_hcbcaco_ia3d
c_hcbcaco_s33d
c_hcbcan_ia3d
c_hccflopsy16_3d
c_hnca_ia3d
c_hncaco_ia3d
c_hncaco_s33d
c_hnco_ia3d
c_hncoca2_ia3d
c_hncoca_ia3d
calibgp
cbcaconhgp3d
cbcaconhgpwg3d.2
cbcaconhgpwg3d
cbcaconhgpwg4d
cbcanhgp3d
cbcanhgpwg3d
ccaconhgp2h3d
ccaconhgp3d.2
ccaconhgp3d
ccanhgp2h3d
ccanhgp3d.2
ccanhgp3d
ccconhgp2h3d
ccconhgp3d
clmlevphpr
colocqf
cosycwgppsqf
cosycwphps
cosydclrqf
cosydcphwt
cosydcph
cosydcqf
cosydfesgpphpp
cosydfesgpph
cosydfetgp.1
cosydfetgp.2
cosydfetgppp.2
cosydfgpph19
cosydfphpp
cosydfphpr
cosydfph
cosydfqf
cosyetgp
cosygpmfphpp
cosygpmfph
cosygpmfppqf
cosygpmfqf
cosygpphzfzs
cosygpppqf
cosygpprqf
cosygpqf
cosyjdqf

cosylrqf
cosyphpr
cosyphtf
cosyph
cosyppqf
cosyprqf
cosyqf45
cosyqf90
cosyqfr2
cosyqfr3
cosyqfrl
cosyqftf
cosyqf
cpmg1d
cpmgpr1d
cpmg
crineptgpph
croesyphpr
croesyph
dazzg
dec180f3pr
dec180pr
dec180sp
dec180
decp902hf4
decp902h
decp90f3
decp90sp
decp90
dept135
dept45
dept90
deptnd
deptppnd
deptqgpsp.2
deptqgpsp
deptqsp
deptsp135
deptsp45
deptsp90
deptsp
dept
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dipsi2esgpph
dipsi2etgpjcsix1
dipsi2etgpsi19
dipsi2etgpsi
dipsi2etgp
dipsi2gpph19
dipsi2gpphpr
dipsi2gpphwgx1
dipsi2gpphwgx2
dipsi2gpphwgxf
dipsi2gpphzs
dipsi2phpr
dipsi2ph
dipsihsqcf3gpsi3d
dipsitretf3gp3d
dqs1d
dqseagp135
dqseagp90
dqsphpr
dqsph
dqsqf
dstebpgp3s1d
dstebpgp3s
dstegp3s1d
dstegp3s
ecos3cph
ecos3nph
ecosygpph
eretic30
ereticgppn
ereticpr30
ereticpr
eretic
fhsqccxf3gpph
fhsqcf3gpph
h2bcedetgpl3
h2bcetgpl3
hacahbcosygp3d
haconhgpwg3d
hanhgpwg3d
hathmbcetgpl3
hbcbcgcchargp
hbcbcgcdcehegp
hbcbcgcdhdgp
hbhacbcaconhgpwg5d
hbhacbcanhgpwg4d
hbhaconhgp3d
hbhaconhgpwg3d
hbhaconhgpwg4d
hbhanhgp3d
hbhanhgpwg3d
hcacogp3d
hcacogpjc3d
hcacongp2h
hcacongp3d
hcaconhgp3d
hcaconhgpwg3d
hcangp2h
hcangp3d
hcannhgp3d
hcbcacongp3d
hccccosygp3d
hcccgcbcaconhgp3d
hcccgcbcaconhgpwg3d
hccconhgp3d1
hccconhgp3d2
hccconhgp3d3
hccconhgpwg3d2
hccconhgpwg3d3
hccconhgpwg4d
hcchatgp3d2
hcchatgp3d
hcchcogp3d2
hcchcogp3d
hcchcogp4d
hcchcosygp3d
hcchdigp3d2
hcchdigp3d
hcchdigp4d
hcchecosgp3d
hcchetgplr
hetsfhmqcf3gpph
hfcoqfqn
hhxcoqf.2
hhxcoqf
hjresqf
hmbcacbigpl2ndqf
hmbcacgplpndqf.2
hmbcacgplpndqf
hmbcacgplpqf
hmbcctetgpl2nd
hmbcctetgpnd
hmbcedetgpl3nd
hmbcetf3gpl3nd
hmbcetf3gpnd
hmbcetgpjcl2nd
hmbcetgpl2ndsm
hmbcetgpl2nd
hmbcetgpl3nd
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hmbcgplpndprqf

hmbcgplpndqf
hmbcgpndqf
hmbclpndqf
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hmcmcbcagpwg3d
hmcmcbcanhgpwg3d2
hmcmcbcanhgpwg3d
hmqcbidfphrl
hmqcbif3ph
hmqcbimlndph
hmqcbimlndqf
hmqcbimlph
hmqcbimlqf
hmqcbind1d
hmqcbindphrl
hmqcbindph
hmqcbindqfrl
hmqcbindqf
hmqcbindrd1d
hmqcbinoph
hmqcbiphpr2
hmqcbiphpr
hmqcbiphrl
hmqcbiph
hmqcbiqf
hmqcbird1d
hmqcctetgp.2
hmqcctetgp
hmqcdietf3gpsi.2
hmqcdietf3gpsi
hmqcdietf3gp
hmqcdietgpsi.2
hmqcdietgpsi
hmqcdietgp
hmqcetf3gpno
hmqcetf3gpro.2
hmqcetf3gpro
hmqcetf3gpsi.2
hmqcetf3gpsi
hmqcetf3gp
hmqcetgp.2
hmqcetgpno
hmqcetgpro.2
hmqcetgpro
hmqcetgpsi.2
hmqcetgpsi
hmqcetgp
hmqcf3gpph19.2
hmqcf3gpph19
hmqcf3gpphwg
hmqcf3phpr
hmqcf3ph
hmqcfbph
hmqcgpmlqf
hmqcgpnd1d
hmqcgpqfrl
hmqcgpqf
hmqcjgp
hmqcmlevbi3d
hmqcmlndphpr
hmqcmlndph
hmqcmlphpr
hmqcmlph
hmqcnd1d
hmqcndph
hmqcndqf
hmqcndrd1d
hmqcnoesybi3d
hmqcnoesyhmqcnngp4d
hmqcnoesyhsqcncgp4d
hmqcnophpr
hmqcph11
hmqcphpr
hmqcph
hmqcqf
hmqcrd1d
hmscetgpnd
hncacbgp2h3d
hncacbgp3d
hncacbgpjc3d
hncacbgpwg2h3d
hncacbgpwg3d
hncacbigp2h3d
hncacbigp3d
hncacbigpwg2h3d
hncacbigpwg3d
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hncacogp4d
hncacogpwg2h3d
hncacogpwg2h4d
hncacogpwg3d
hncacogpwg4d
hncacosygp3d
hncadhgp3d
hncadqzqgp3d
hncaecosgp3d2
hncaecosgp3d
hncagp2h3d
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