150

Basic-

NMR-

Experiments

MMO January 2000 Version 2.0

CONTENTS

Introduction

Here you will find some information about the Bruker pulse programs and parameters, which are needed to repeat the experiments shown in the book: “150 and More Basic NMR Experiments” written by S. Braun, H.-O. Kalinowski, S. Berger, VCH Weinheim, Germany.

First you will find the experiment number, followed by the Bruker pulse program, the settings of the different channels and a list of the acquisition and processing parameters.

The number of the chapters are identically with the number of the chapters in the book.

The book contains a lot of very interesting experiments. If you want to repeat such experiments with a BRUKER Avance instrument you need the pulse program and the parameters belonging to the pulse program. The needed parameters are sometimes different or more then mentioned in the book. BRUKER has its own nomenclature for the parameters, which is different from the book. For example the 90° transmitter pulse is always P1, D2 is a delay depending on the coupling constant (1/2 J) and so on.

It is possible that the needed pulse program isn’t yet in your library, in that case send me an e-mail: Monika.Moertter@bruker.de.

Chapter 2
- Determination of the Pulse-Duration

Summary

Experiment	Pulse program	Description
2.1	zg0	Determination of the 90° ^{1}H Transmitter Pulse-Duration
2.2	zg0dc	Determination of the 90° ^{13}C Transmitter Pulse-Duration
2.3	decp90	Determination of the 90° ^{1}H Decoupler Pulse-Duration
2.4	zg0	The 90° ^{1}H Pulse with Inverse Spectrometer Configuration
2.5	decp90	The 90° ^{13}C Decoupler Pulse with Inverse Configuration
2.6	exp2_6a.mo and exp2_6b.mo	Composite Pulses
2.7	zg0	Radiation Damping
2.8	zg	Pulse and Receiver Phases
2.9	zg	Determination of Radiofrequency Power

Experiment 2.1
- Determination of the 90° 1H Transmitter Pulse Duration

pulse program: zg0
1D-sequence, using p0 for any flip angle. Result is a routine proton NMR spectrum.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 : F1 channel - high power level for ^{1}H transmitter pulse, here 3dB was used

D1 : 30 sec - relaxation delay

SW : 500 Hz

NS : 1

P0 : F1 channel - ^{1}H transmitter pulse, to be varied, 1 usec as initial value and increase by 2 usec

TD : 4 K

O1 : on resonance of $CHCl_3$ signal

RG : receiver gain for correct ADC input

Processing parameters

SI :2 K

WDW :EM

FT :fourier transformation

BC_mod :quad

LB :1 Hz

phase correction :adjust the phase of the first spectrum to pure absorption and for all other experiments use the same values for the phase correction (PK)

baseline correction :ABS

plot :use XWINPLOT

Experiment 2.2

- Determination of the 90° ^{13}C Transmitter Pulse Duration

pulse program: zg0dc
1D-sequence with F2 decoupling, using p0 for any flip angle. Result is a standard 13C NMR spectrum with proton broad-band decoupling.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :F1 channel - high power level for ^{13}C transmitter pulse, here 3 dB was used

P0 :F1 channel - ^{13}C transmitter pulse, 7 usec for experiment a and 14 usec for experiment b

PL12 :F2 channel - power level for CPD decoupling

PCPD2 :F2 channel – 90° pulse for decoupling sequence

CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2

D1 :60 sec - relaxation delay

D11 :30 msec - delay for disk I/O

TD :4 K

SW :500 Hz

O1 :on resonance of ^{13}C signal

O2 :middle of ^{1}H NMR spectrum

NS :1

RG :receiver gain for correct ADC input

Processing parameters

SI :2 K

BC_mod :quad

WDW :EM

LB :1 Hz

FT :fourier transformation

phase correction :adjust the phase of the first spectrum to pure absorption and for all other experiments use the same values for the phase correction (PK)

baseline correction : ABS

plot : use XWINPLOT

Experiment 2.3

- Determination of the 90° ^{1}H Decoupler Pulse Duration

pulse program: decp90
1D-sequence to determine the 90° decoupler pulse-duration

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :F1 channel - high power level for ^{13}C transmitter pulse

P1 :F1 channel - 90° ^{13}C transmitter pulse

PL2 :F2 channel - high power level for ^{1}H decoupler pulse, here 0 dB was used

P3 :F2 channel - ^{1}H decoupler pulse, use 1 usec as starting value, to be varied

D1 :60 sec - relaxation delay

D2 :1/[2J(C,H)]= 2.36 msec, calculated from ^{1}J(C,H)=212 Hz

TD :4 K

SW :500 Hz

O1 :on resonance of ^{13}C signal

O2 :on resonance of ^{1}H NMR signal

NS :1

RG :receiver gain for correct ADC input

In a second set of experiments use high decoupler attenuation (PL2=22 dB) and vary it so that P3 is in the region of 100 usec (for WALTZ).

Processing parameters

SI :2 K
WDW :EM
FT :fourier transformation
baseline correction :ABS
BC_mod :quad
LB :2 Hz
phase correction :adjust the doublet in antiphase and use the same values for the other phase corrections (PK).
plot :use XWINPLOT

Experiment 2.4

- The 90° ^{1}H Pulse with Inverse Spectrometer Configuration

pulse program: zg0
compare with Experiment 2.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :F1 channel - high power level for ^{1}H transmitter pulse
D1 :5 sec - relaxation delay
TD :4 K
O1 :100 Hz towards higher frequency of $CHCl_3$ signal
NS :8
P0 :F1 channel - ^{1}H transmitter pulse, near 360° as starting value, to be varied
SW :500 Hz
RG : receiver gain for correct ADC input

Processing parameters

No signal processing is required, since the FID is directly observed.

Experiment 2.5

- The 90° ^{13}C Decoupler Pulse with Inverse Configuration

pulse program: decp90
compare with Experiment 2.3

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :F1 channel - high power level for ^{1}H transmitter pulse
PL2 :F2 channel - high power level for ^{13}C decoupler pulse, here 0 dB was used
D1 :20 sec - relaxation delay
TD :4 K
O1 :on resonance of ^{1}H signal
P1 :F1 channel - 90° ^{1}H transmitter pulse
P3 :F2 channel - ^{13}C decoupler pulse, 1 usec as starting value, to be varied.
D2 :1/[2J(C,H)]= 2.33 msec, calculated from ^{1}J(C,H)=215 Hz
SW :500 Hz
O2 :on resonance of ^{13}C NMR signal

NS :1 **RG** :receiver gain for correct ADC input

In a second set of experiments use high decoupler attenuation (PL2) and vary it so that P3 becomes in the range of 70 usec (for GARP).

Processing parameters

SI :2 K **BC_mod** :quad

WDW :EM **LB** : 1 Hz

FT :fourier transformation **phase correction** :adjust the phase of the big signal descended from the protons bound to ^{12}C in dispersion: look for a clean anti phase pattern of the ^{13}C satellites and use the same values for the next phase correction (PK).

baseline correction :ABS **plot** :use XWINPLOT

Experiment 2.6

- Composite Pulses

a)pulse program: exp2_6a.mo
Sequence with a normal 180° pulse to compensate pulse imperfections.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

Perform two experiments, one with the pulse program exp2_6.mo and one with exp2_2b.mo. Use the same parameters for both experiments.

PL1 :F1 channel - high power level for ^{1}H transmitter pulse, 3dB was used here **P1** :F1 channel – 90° ^{1}H transmitter pulse

P2 :F1 channel – 180° ^{1}H transmitter pulse

D1 :30 sec - relaxation delay **D15** :10 msec - fixed delay

TD :64 K **SW** :80 ppm

O1 :10 kHz towards higher frequencies from the resonance of the $CHCl_3$ signal

NS :8 **RG** : receiver gain for correct ADC input

Processing parameters

Use the same processing parameters for both experiments

SI :32 K **BC_mod** :quad

WDW :EM **LB** :1 Hz

FT :fourier transformation **phase correction** :adjust the phase of the $CHCl_3$ signal to be negative

baseline correction :ABS **plot** :use XWINPLOT

b)pulse program: exp2_6b.mo
A sequence with a 180° composite pulse to compensate pulse imperfections.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :F1 channel - high power level for ^{1}H transmitter pulse, 3dB was used here
P1 :F1 channel – 90° ^{1}H transmitter pulse
P2 :F1 channel – 180° ^{1}H transmitter pulse
D1 :30 sec - relaxation delay
D15 :10 msec - fixed delay
TD :64 K
SW :80 ppm
O1 :10 kHz towards higher frequencies from the resonance of the $CHCl_3$ signal
NS :8
RG : receiver gain for correct ADC input

Processing parameters

Use the same processing parameters for both experiments
SI :32 K
BC_mod :quad
WDW :EM
LB :1 Hz
FT :fourier transformation
phase correction :adjust the phase of the $CHCl_3$ signal to be negative
baseline correction :ABS
plot :use XWINPLOT

Experiment 2.7

- Radiation Damping

pulse program: zg0
compare with Experiment 2.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

Perform two experiments with different pulses.
PL1 :F1 channel - high power level for ^{1}H transmitter pulse (3 dB)
P0 :F1 channel - ^{1}H transmitter pulse, a) 360° and b) 180°
D1 :2 sec - relaxation delay
TD :4 K
SW :500 Hz
O1 :on resonance of H_2O signal
NS :1
RG : receiver gain for correct ADC input

Processing parameters

process the two FIDs with the same parameters
SI :2 K
BC_mod :quad
WDW :EM
LB :0.3 Hz
FT :fourier transformation
phase correction :adjust the phase to pure absorption
baseline correction :ABS
plot :use XWINPLOT, both traces should be plotted on the same vertical scale

Experiment 2.8

- Pulse and Receiver Phases

pulse program: zg
1D-sequence, using a 90° pulse. Result is a routine proton NMR spectrum

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

Display both quadrature channels of the receiver. Record an FID with the offset on resonance and change the transmitter phase in the pulse program so that only the left quadrature channel receives a signal. Then set the offset 50 Hz off resonance and repeat the experiment. Now change the transmitter phase in 90° steps and observe the changes on both FID channels and on the spectrum.

PL1 :F1 channel - high power level for ^{1}H transmitter pulse (3 dB)
P1 :F1 channel – 90° ^{1}H transmitter pulse
D1 :1 sec - relaxation delay
TD :4 K
SW :500 Hz
O1 :50 Hz off resonance of $CHCl_3$ signal
NS :1
RG : receiver gain for correct ADC input

Processing parameters

SI :2 K
BC_mod :quad
WDW :EM
LB :1 Hz
FT :fourier transformation
phase correction :adjust the phase of the first spectrum for pure absorption and use the same values for the other phase corrections (PK).
baseline correction :ABS
plot :use XWINPLOT

Experiment 2.9

- Determination of Radiofrequency Power

pulse program: zg
compare with Experiment 2.8

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :F1 channel - high power level for ^{1}H transmitter pulse, 0 dB initial value, to be increased in 3 dB steps.
P1 :F1 channel - 90° ^{1}H transmitter pulse, to be determinred for each attenuation level.
D1 :60 sec - relaxation delay
TD :4 K
SW :500 Hz
O1 :on resonance of ^{1}H signal
NS :1
RG :receiver gain for correct ADC input

Processing parameters

SI :2 K
BC_mod :quad
WDW : EM
LB :1 Hz
FT :fourier transformation
phase correction :adjust the phase to pure absorption and use the same values for the next phase correction (PK)..
baseline correction :ABS

Chapter 3
- Routine NMR Spectroscopy and Standard Tests

Summary

Experiment	Pulse program	Description
3.1	zg30	The Standard ^{1}H NMR Experiment
3.2	zgdc30	The Standard ^{13}C NMR Experiment
3.3	zg	The Application of Window Functions
3.4	zg	Computer-aided Spectral Analysis
3.5	zg	Line-Shape Test for ^{1}H NMR Spectroscopy
3.6	zg	Resolution Test for ^{1}H NMR Spectroscopy
3.7	zg	Sensitivity Test for ^{1}H NMR Spectroscopy
3.8	zgcw	Line-Shape Test for ^{13}C NMR Spectroscopy
3.9	zg	ASTM Sensitivity Test for ^{13}C NMR Spectroscopy
3.10	zgdc	Sensitivity Test for ^{13}C NMR Spectroscopy
3.11	zg	Quadrature Image Test
3.12	zg	Dynamic Range Test for Signal Amplitudes
3.13	zgphase.mo	13° Phase Stability Test

Experiment 3.1
- The Standard ^{1}H NMR Experiment

pulse program: zg30
1D-sequence, using a 30° flip angle. Result is a routine proton NMR spectrum.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :F1 channel - high power level for ^{1}H transmitter pulse
P1 :F1 channel - 90° ^{1}H transmitter pulse
D1 : 0.1 sec - relaxation delay
TD : 32 K
SW : 20 ppm
O1 : middle of the ^{1}H NMR spectrum
NS :8
RG : receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :0.1 Hz

FT :fourier transformation

integration :is done with ABS or can be done manual

baseline correction :ABS

plot :use XWINPLOT

phase correction :adjust the phase to pure absorption.

referencing : set the TMS signal to 0 ppm.

peak picking :choose the desired level

Experiment 3.2

- The Standard ^{13}C NMR Experiment

pulse program : zgdc30
1D-sequence with decoupling, using a 30° flip angle. Result is a standard ^{13}C NMR spectrum with proton broad-band decoupling.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :F1 channel - high power level for ^{13}C transmitter pulse

PL12 :F2 channel - power level for CPD decoupling

CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2

D1 :0.4 sec - relaxation delay

TD :32 K

O1 :middle of the ^{13}C NMR spectrum

NS :128

RG :receiver gain for correct ADC input

P1 :F1 channel - 90° ^{13}C transmitter pulse

PCPD2 :F2 channel – 90° pulse for decoupling sequence

D11 :30 msec - delay for disk I/O

SW :250 ppm

O2 :middle of ^{1}H NMR spectrum

DS :2

Processing parameters

SI :16 K

WDW :EM

FT :fourier transformation

referencing :set the TMS signal to 0 ppm.

peak picking :choose the desired level

BC_mod :quad

LB :2 Hz

phase correction :adjust the phases to pure absorption.

baseline correction :ABS

plot :use XWINPLOT

Experiment 3.3

- The Application of Window Functions

pulse program: zg
compare with Experiment 2.8

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :F1 channel - high power level for ^{1}H transmitter pulse

D1 : 1 sec - relaxation delay

TD : 32 K

P1 :F1 channel - 90° ^{1}H transmitter pulse

SW : 1 ppm

O1 : center of ODCB multiplet
NS :1
RG : receiver gain for correct ADC input

Processing parameters
a) transform FID without any weighting function
b) transform FID with EM
c) transform FID with GM
SI :16 K
BC_mod :quad
WDW :EM
LB :0.07 Hz
WDW :GM
LB :-0.06 Hz
GB :0.25 Hz
FT :fourier transformation
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT
referencing : set the TMS signal to 0 ppm.

Experiment 3.4
- Computer-aided Spectral Analysis

pulse program: zg
compare with Experiment 2.8

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters
PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D1 :1 sec - relaxation delay
TD :32 K
SW :1 ppm
O1 :center of ODCB multiplet
spinning rate :20 Hz
NS :1
RG :receiver gain for correct ADC input

Processing parameters
SI :32 K
BC_mod :quad
WDW :no
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT
Transfer the spectrum to a PC. The procedure shown in the book was performed with the CALM software, obtainable on the Internet.

Experiment 3.5
- Line-Shape Test for ^{1}H NMR Spectroscopy

pulse program: zg
compare with Experiment 2.8

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D1 :60 sec - relaxation delay
TD :32 K
SW :500 Hz
O1 :on resonance of ^{1}H signal
spinning rate :20 Hz
NS :1
RG :receiver gain for correct ADC input

Processing parameters

SI :32 K
BC_mod :quad
WDW :no
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
referencing :set the TMS signal to 0 ppm
CY :1000 and check, whether the satellites have a height of 5.5.
plot :use XWINPLOT

Experiment 3.6

- Resolution Test for ^{1}H NMR Spectroscopy

pulse program: zg
compare with Experiment 2.8

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D1 :1 sec - relaxation delay
TD :32 K
SW :1 ppm
O1 :center of ODCB multiplet
spinning rate :20 Hz
NS :1
RG :receiver gain for correct ADC input

Processing parameters

SI :32 K
BC_mod :quad
WDW :no
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 3.7

- Sensitivity Test for ^{1}H NMR Spectroscopy

pulse program: zg
compare with Experiment 2.8

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H
P1 :f1 channel - 90° ^{1}H transmitter pulse

transmitter pulse
D1 :60 sec - relaxation delay
TD :32 K
O1 :middle of the ^{1}H NMR spectrum
NS :1
SW :10 ppm
RG :receiver gain for correct ADC input

Processing parameters
SI 32 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :1 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT: the full spectrum should be plotted and the noise between 3 ppm and 5 ppm enlarged to allow a correct peak to peak noise measurement.

Experiment 3.8
- Line-Shape Test for ^{13}C NMR Spectroscopy

pulse program: zgcw
1D-sequence with CW decoupling, using 90° flip angle. Depending where O2 is set, different results are possible like an ^{1}H off-resonance decoupled ^{13}C NMR spectrum (O2 on resonance of ^{1}H TMS signal) or an ^{1}H single frequency decoupled ^{13}C NMR pectrum (O2 on resonance on a special ^{1}H group).

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters
PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL14 :f2 channel - low power level for ^{1}H decoupler pulse
D1 :1 sec - relaxation delay
TD :16 K
O1 :on resonance of ^{13}C signal
NS :1
spinning rate :20 Hz
P1 :f1 channel - 90° ^{13}C transmitter pulse
decoupler attenuation for continous wave decoupling
D11 :30 msec - delay for disk I/O
SW :200 Hz
O2 :on resonance of ^{1}H signal
RG :receiver gain for correct ADC input

Processing parameters
SI :16 K
WDW :no
FT :Fourier transformation
baseline correction :ABS
CY :set the intensity of the main signal to 1000 and check the line-width at heights 500, 5.5 and 1.1.
BC_mod :quad
phase correction :adjust the phase to pure absorption.
referencing :set the TMS signal to 0 ppm
plot :use XWINPLOT

Experiment 3.9
- ASTM Sensitivity Test for ^{13}C NMR Spectroscopy

pulse program: zg
compare with Experiment 2.8

Setting of the needed channels: F1: ^{13}C
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
D1 :300 sec - relaxation delay
TD :32 K
SW :200 ppm
O1 :middle of ^{13}C NMR spectrum
decoupler :off
NS :1
RG :receiver gain for correct ADC input

Processing parameters

SI :64 K
BC_mod :quad
WDW :EM
LB :3.5 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT: the full spectrum should be plotted and the noise between 120 ppm and 80 ppm enlarged to allow a correct peak to peak noise measurement.

Experiment 3.10
- Sensitivity Test for ^{13}C NMR Spectroscopy

pulse program: zgdc
1D-sequence with F2 decoupling, using 90° flip angle. Result is a standard ^{13}C NMR spectrum with proton broad-band decoupling.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel – 90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :300 sec – relaxation delay
D11 :30 msec - delay for disk I/O
TD :64 K
SW :200 ppm
O1 :middle of ^{13}C NMR spectrum
O2 :middle of ^{1}H NMR spectrum
NS :1
RG :receiver gain for correct ADC input

Processing parameters

SI :64 K
BC_mod :quad

WDW :EM
FT :Fourier transformation

referencing :set the TMS signal to 0 ppm
baseline correction :ABS

LB :0.3 Hz
phase correction :adjust the phase to pure absorption.

plot :use XWINPLOT: the full spectrum should be plotted and the noise between 120 ppm and 80 ppm enlarged to allow a correct peak to peak noise measurement.

Experiment 3.11
- Quadrature Image Test

pulse program: zg
compare with Experiment 2.8

Setting of the needed channels: F1: 1H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
D1 :1 sec – relaxation delay
TD :8 K
O1 :250 Hz towards high frequency of $CHCl_3$ signal
NS :1

P1 :f1 channel - 90° ^{1}H transmitter pulse
SW :1000 Hz
RG :receiver gain for correct ADC input

Processing parameters

SI :4 K
WDW :EM
FT :Fourier transformation

CY :set the intensity of the CHCl3 signal to 1000 and enlarge the quadrature image signal, which is found 250 Hz towards higher frequencies from the offset position**.**
baseline correction :ABS

BC_mod :quad
LB :1 Hz
phase correction :adjust the phase to pure absorption.

plot :use XWINPLOT

Experiment 3.12
- Dynamic Range Test for Signal Amplitudes

pulse program: zg
compare with Experiment 2.8

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

P1 :f1 channel - 90° ^{1}H transmitter pulse

D1 :5 sec – relaxation delay
TD :32 K
SW :10 ppm
O1 :middle of ^{1}H NMR spectrum
NS :1
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :0.2 Hz
FT :Fourier transformation
phase correction :carefully correct the phase of the water signal, try to detect the very small signal of t-butanol at 1.28 ppm, and adjust the phase of this signal as well.
integration :integrate the four relevant signals and check the integrals for consistency with the molar ratios of the four compounds in the sample.
baseline correction :ABS
plot :use XWINPLOT

Experiment 3.13
– 13° Phase Stability Test

pulse program: zgphase.mo
The 13° phase stability test shown here transforms phase stability into signal amplitudes and measures the phase stability between two r.f. pulses.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

Use an automation routine which performs this experiment 64 times in sequence
PL1 :f1 channel - high power level for ^{1}H transmitter pulse, 3dB was used
P1 :f1 channel - 90° ^{1}H transmitter pulse
D1 :20 sec – relaxation delay
D20 :1 msec - fixed delay
TD :4 K
SW :500 Hz
O1 :37 Hz to higher frequencies from $CHCl_3$ signal
NS :1
RG :receiver gain for correct ADC input

Processing parameters

SI :2 K
BC_mod :quad
WDW :EM
LB :1 Hz
FT :Fourier transformation
phase correction :Adjust the phse of the first spectrum roughly for dispersion and always use the same digital phase correction (PK)
baseline correction :ABS
plot :use XWINPLOT

Chapter 4
- Decoupling Techniques

Summary

Experiment	Pulse program	Description
4.1	zg0hd	Decoupler Calibration for Homonuclear Decoupling
4.2	zg0cw	Decoupler Calibration for Heteronuclear Decoupling
4.3	zg0cw	Low Power Calibration for Heteronuclear Decoupling
4.4	zg0hd	Homonuclear Decoupling
4.5		Homonuclear Decoupling at Two Frequencies
4.6	zgspt.mo	The Homonuclear SPT Experiment
4.7	zgndspt.mo	The Heteronuclear SPT Experiment
4.8	zgf2pr.mo	1D Nuclear Overhauser Difference Spectroscopy
4.9	noemul	1D NOE Spectroscopy with Multiple Selective Irradiation
4.10	zg0cw	^{1}H Off-Resonance Decoupled ^{13}C NMR Spectra
4.11	zg0gd	The Gated ^{1}H-Decoupling Technique
4.12	zg0ig	The Inverse Gated ^{1}H-Decoupling Technique
4.13	zg0cw	^{1}H Single Frequency Decoupling of ^{13}C NMR Spectra
4.14	zg0cw2.mo	^{1}H Low Power Decoupling of ^{13}C NMR Spectra
4.15	hetnoe.mo	Measurement of the Heteronuclear Overhauser Effect

Experiment 4.1
- Decoupler Calibration for Homonuclear Decoupling

pulse program: zg0hd
1D-sequence with homodecoupling, using p0 for any flip angle.By this technique residual multiplets are obtained in which the spin coupling to the irradiated proton is missing.

Setting of the needed channels: F1: ^{1}H
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

P0 :f1 channel - 45° ^{1}H transmitter pulse

PL24 :f2 channel - power level for hd/hc

decoupling, to be varied

D1 :2 sec – relaxation delay
D12 :20 usec - delay for power switching
TD :4 K
SW :500 Hz
O1 :on resonance of ^{1}H signal
O2 :50 Hz towards lower frequency from O1
digmod :homodecoupling-digital
NS :1
RG :receiver gain for correct ADC input

Processing parameters

SI :4 K or more, use zero-filling to ensure enough data points for the relatively small Bloch-Siegert shifts
BC_mod :quad
WDW :EM
LB :0.1 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS

Experiment 4.2

- Decoupler Calibration for Heteronuclear Decoupling

pulse program: zg0cw
1D-sequence with CW decoupling, using p0 for any flip angle. Depending where O2 is set, different results are possible like an ^{1}H off-resonance decoupled ^{13}C NMR spectrum (O2 on resonance of ^{1}H TMS signal) or an ^{1}H single frequency decoupled ^{13}C NMR pectrum (O2 on resonance on a special ^{1}H group).

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P0 :f1 channel - 45° ^{13}C transmitter pulse
PL14 :f2 channel - power level for cw/hd decoupling, to be varied
D1 :2 sec – relaxation delay
D11 :30 msec - delay for disk I/O
TD :4 K
SW :500 Hz
O1 :on resonance of ^{13}C signal
O2 :50 Hz offset from ^{1}H signal
NS :1
RG :receiver gain for correct ADC input

Processing parameters

SI :4 K or more, use zero-filling to ensure enough data points to obtain accurate values for the residual splittings
BC_mod :quad
WDW :EM
LB :2 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
referencing :set the TMS signal to 0 ppm

Experiment 4.3

- Low Power Calibration for Heteronuclear Decoupling

pulse program: zg0cw
compare with Experiment 4.2

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL14 :f2 channel - power level for cw/hd decoupling, to be varied
D1 :2 sec – relaxation delay
TD :2 K
O1 :on resonance for carboxyl ^{13}C nucleus of acetic acid
NS :1
P0 :f1 channel - 45° ^{13}C transmitter pulse
D11 :30 msec - delay for disk I/O
SW :100 Hz
O2 :25 Hz offset from the ^{1}H resonance of the CH_3 group of acetic acid
RG :receiver gain for correct ADC input

Processing parameters

SI :4 K or more, use zero-filling to ensure enough data points for the reduced splittings
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :0.3 Hz
phase correction :adjust the phase to pure absorption.

Experiment 4.4

- Homonuclear Decoupling

pulse program: zg0hd
compare with Experiment 4.1

Setting of the needed channels: F1: ^{1}H
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel – high power level for ^{1}H transmitter pulse
PL14 :f2 channel - power level for cw/hd decoupling
D1 :1 sec – relaxation delay
TD :32 K
O1 :middle of the ^{1}H NMR spectrum
NS :8
RG :receiver gain for correct ADC input
P0 :f1 channel - 45° ^{1}H transmitter pulse
D12 :20 usec - delay for power switching
SW :10 ppm
O2 :on resonance of irradiated proton
digmod :homodecoupling-digital

Processing parameters

SI :16 K
WDW :EM
BC_mod :quad
LB :0.3 Hz

FT :Fourier transformation

phase correction :adjust the phase to pure absorption.

baseline correction :ABS

plot :use XWINPLOT

Experiment 4.5

- Homonuclear Decoupling at Two Frequencies

pulse program:

Setting of the needed channels: F1: ^{1}H
F2: ^{1}H

Acquisition parameters

Processing parameters

Experiment 4.6

- The Homonuclear SPT Experiment

pulse program: zgspt.mo
1D-sequence, using selective population transfer to provides the relative sign information of spin coupling constants.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse, 3 dB was used here

P0 :f1 channel - 30° ^{1}H transmitter pulse

PL21 :f1 channel - low power level, here 90 dB was used (see Exp. 2.6)

P28 :f1 channel - 180° ^{1}H transmitter low power pulse, here 0.8 sec was used

D1 :5 sec – relaxation delay

D12 :20 usec - delay for power switching

TD :8 K

SW :2.5 ppm

O1 :on resonance of a chosen multiplet line of the sample

NS :1

RG :receiver gain for correct ADC input

Processing parameters

SI :4 K

BC_mod :quad

WDW :EM

LB :0.1 Hz

FT :Fourier transformation

phase correction :adjust the phase to pure absorption.

baseline correction :ABS

plot :use XWINPLOT

Experiment 4.7

- The Heteronuclear SPT Experiment

pulse program: zgndspt.mo
1D-sequence with no decoupling. The heteronuclear selective population transfer experiment is especially able for determining the relative sign of long-range spin coupling constants.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL14 :f2 channel - power level for decoupling, γB_2=1 Hz, 90 dB was used here
D1 :2 sec – relaxation delay
D12 :20 usec - delay for power switching
TD :64 K
O1 :middle of the ^{13}C NMR spectrum
NS :8

P0 :f1 channel - 45° ^{13}C transmitter pulse
P10 :f2 channel - 180° ^{1}H decoupler pulse, here 0.4 sec was used
D11 :30 msec - delay for disk I/O
SW :200 ppm
O2 :exact transition frequency of a ^{13}C satellite: adjust O2 to a frequency 2Hz above that of the left-most line of the proton doublet at 7.5 ppm.
RG :receiver gain for correct ADC input

Processing parameters

SI :32 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS

BC_mod :quad
LB :0.2 Hz
phase correction :use the same values as for the coupled ^{13}C spectra (PK)
plot :use XWINPLOT

Experiment 4.8

- 1D Nuclear Overhauser Difference Spectroscopy

pulse program: zgf2pr.mo
1D-sequence with presaturation in F2. The presaturation of different signals and later building of differences (substraction of spectra: one with presaturation outside and one with presaturation of a signal of interest) gives information about the NOE.

Setting of the needed channels: F1: ^{1}H
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel – high power level for ^{1}H transmitter pulse
PL14 :f2 channel – low power level for cw/hd decoupling, here 70 dB was used
D1 :0.1 sec – relaxation delay
D20 :6 sec - fixed delay

P1 :f1 channel - 90° ^{1}H transmitter pulse
D13 :3 usec - short delay

TD :32 K
SW :10 ppm
O1 :middle of the ^{1}H NMR spectrum
O2 :on resonance for the methyl protons in the first experiment and on resonance of the residual ^{1}H signal of $CHCl_3$ in the reference spectrum
NS :16
DS :4
RG :receiver gain for correct ADC input

Processing parameters

NOE difference spectra can be processed in different ways. Since one wants to observe signal intensity changes of 2% to 10%, one should use an exponential window function with LB=2 Hz to minimize artefacts of substraction. One can either transform the two spectra separatly using a digitally indentical phase correction and substract the two spectra, or, more conveniently, substract the two FIDs directly from each other. In the difference spectrum, adjust the phase of the methyl group signal to be negative and the phase of the reference signal ($CHCl_3$) to be positive. Evaluate only signals which have correct phase and have therefore not been affected by inadequate spectrometer stability.

Experiment 4.9

- 1D NOE Spectroscopy with Multiple Selective Irradiation

pulse program: noemul

This experiment is a technical variant of the NOE difference experiment. Instead of irradiating the center of a broad multiplet, in the experiment each line of the multiplet is irradiated for a short time with a bandwidth of ca. 1-2 Hz and the irradiating frequency is cycled repeatedly in a stepwise manner through the entire multiplet during the pre-irradiation time.

Setting of the needed channels: F1: ^{1}H
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel – high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
PL14 :f2 channel – power level for presaturation (85 dB)
D1 :0.1 sec – relaxation delay
D11 :30 msec - delay for disk I/O
D12 :20 usec – delay for power switching
D20 :irradiation time per frequency, here 400 msec was used
L4 :overall irradiation time: D20*L4, here 3
TD :32 K
SW :10 ppm
O1 :middle of the ^{1}H NMR spectrum
O2 :lists of frequencies within the multiplets to be irradiated
NS :8
DS :4
au-program :noemult
for each signal, which should be irradiated an own list has to be created (noedif.1, noedif.2 ...)
FQ2LIST :noedif.1
average cycles :ns*number of average cycles, here 4 was used
RG :receiver gain for correct ADC input

Processing parameters

NOE difference spectra can be processed in different ways. Since one wants to

observe signal intensity changes of 2% to 10%, one should use an exponential window function with LB=2 Hz to minimize artefacts of substraction. One can either transform the two spectra separatly using a digitally indentical phase correction and substract the two spectra, or, more conveniently, substract the two FIDs directly from each other.

Experiment 4.10

- ^{1}H Off-Resonance Decoupled ^{13}C NMR Spectra

pulse program: zg0cw
compare with Experiment 4.3

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL14 :f2 channel – power level for cw/hd decoupling, γB_2=3500 Hz (see Exp. 2.6 and 4.2)
D1 :0.5 sec – relaxation delay
TD :64 K
O1 :middle of the ^{13}C NMR spectrum
NS :512
P0 :f1 channel - 45° ^{13}C transmitter pulse
D11 :30 msec - delay for disk I/O
SW :200 ppm
O2 :on resonance of ^{1}H TMS signal
RG :receiver gain for correct ADC input

Processing parameters

SI :32 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :1 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 4.11

- The Gated ^{1}H-Decoupling Technique

pulse program: zg0gd
1D-sequence with gated decoupling, using p0 for any flip angle. This experiment is used for determining C,H spin-spin coupling constants without loosing nuclear Overhauser enhancement.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL12 :f2 channel - power level for CPD decoupling
PL13 :f2 channel - power level for second CPD decoupling
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
P0 :f1 channel - 45° ^{13}C transmitter pulse
PCPD2 :f2 channel – 90° pulse for decoupling sequence

D1 :2 sec – relaxation delay
TD :64 K
O1 :middle of the ^{13}C NMR spectrum
NS :512
D11 :30 msec - delay for disk I/O
SW :200 ppm
O2 :middle of ^{1}H NMR spectrum
RG :receiver gain for correct ADC input

Processing parameters

SI :32 K
WDW :EM (or GM is also possible)
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :0.3 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 4.12

- The Inverse Gated ^{1}H-Decoupling Technique

pulse program: zg0ig
1D-sequence with gated decoupling, using p0 for any flip angle. This experiment yields ^{1}H-decoupled NMR spectra of X-nuclei without signal enhancement by the nuclear Overhauser effect.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL12 :f2 channel - power level for CPD decoupling
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
P0 :f1 channel - 45° ^{13}C transmitter pulse
PCPD2 :f2 channel – 90° pulse for decoupling sequence
D1 :10 sec – relaxation delay
TD :32 K
O1 :middle of the ^{13}C NMR spectrum
NS :512
D11 :30 msec - delay for disk I/O
SW :200 ppm
O2 :middle of ^{1}H NMR spectrum
RG :receiver gain for correct ADC input

Processing parameters

SI :64 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :1 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 4.13

- ^{1}H Single Frequency Decoupling of ^{13}C NMR Spectra

pulse program: zg0cw
compare with Experiment 4.2

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse

P0 :f1 channel - 45° ^{13}C transmitter pulse

PL14 :f2 channel - power level for cw/hd decoupling, γB_2=150 Hz (45 dB was used here)

D1 :1 sec – relaxation delay

D11 :30 msec - delay for disk I/O

TD :64 K

SW :200 ppm

O1 :middle of the ^{13}C NMR spectrum

O2 :center of methyl group ^{1}H resonance at 1.6 ppm

NS :8

RG :receiver gain for correct ADC input

Processing parameters

SI :32 K

BC_mod :quad

WDW :EM

LB :2 Hz

FT :Fourier transformation

phase correction :adjust the phase to pure absorption.

baseline correction :ABS

plot :use XWINPLOT

Experiment 4.14

- ^{1}H Low Power Decoupling of ^{13}C NMR Spectra

pulse program: zg0cw2.mo

1D-sequence with CW decoupling, using p0 for any flip angle. This technique correlates an ^{1}H signal with ^{13}C signals which are separated by two, three or more bonds.

Setting of the needed channels: F1: ^{13}C

F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse

P0 :f1 channel - 45° ^{13}C transmitter pulse

PL12 :f2 channel - power level for CPD decoupling

PCPD2 :f2 channel – 90° pulse for decoupling sequence

PL14 :f2 channel - power level for decoupler attenuation during acquisition, γB_2=15 Hz (70 dB was used here)

CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2

D1 :1 sec – relaxation delay

D11 :30 msec - delay for disk I/O

D12 :20 usec – delay for power switching

TD :64 K

SW :200 ppm

O1 :middle of the ^{13}C NMR spectrum

O2 :center of ^{1}H signal of the upfield olefinic proton

NS :8

RG :receiver gain for correct ADC input

Processing parameters

SI :32 K

BC_mod :quad

WDW :EM

LB :2 Hz

FT :Fourier transformation

phase correction :adjust the phase to pure absorption.

baseline correction :ABS

plot :use XWINPLOT

Experiment 4.15

- Measurement of the Heteronuclear Overhauser Effect

pulse program: hetnoe.mo
Experiment to measure the heteronuclear Overhauser effect.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse

P1 :f1 channel - 90° ^{13}C transmitter pulse

PL14 :f2 channel -- power level for cw/hd decoupling

D1 :200 sec – relaxation delay

D11 :30 msec - delay for disk I/O

D12 :20 usec – delay for power switching

TD :1 K

SW :500 Hz

O1 :on resonance of ^{13}C signal of cyclohexane

O2 :list of frequencies

Parmod :2D

FQ2LIST :freqlist, contains two different values: first value 200 kHz off resonance, second value on resonance of ^{1}H signal of cyclohexane

NS :8

RG :receiver gain for correct ADC input

Processing parameters

au-program :splitser, to get 1D files

SI :1 K

BC_mod :quad

WDW :EM

LB :3 Hz

FT :Fourier transformation

phase correction :adjust the phase of to pure absorption and use the same values for the second experiment.

baseline correction :ABS

integration :is done manual using wmisc and rmisc, measure the two integrals and divide one by the other to obtain n+1.

plot :use XWINPLOT

Chapter 5
- Dynamic NMR Spectroscopy

Summary

Experiment	Pulse program	Description
5.1	zg0	Low Temperature Calibration with Methanol
5.2	zg0	High Temperature Calibration with 1,2-Ethandiol
5.3	zg0hd	Dynamic ^{1}H NMR Spectroscopy on Dimethylformamid
5.4	zgdclo.mo	The Saturation Transfer Experiment
5.5		Measurement of the Rotating Frame Relaxation Time $T_{1\rho}$

Experiment 5.1
- Low Temperature Calibration with Methanol

pulse program: zg0
compare with Experiment 2.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
D1 :300 sec – relaxation delay
TD :32 K
O1 :middle of the ^{1}H NMR spectrum
NS :1
P0 :f1 channel - 45° ^{1}H transmitter pulse
SW :8 ppm
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :0.1 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 5.2
- High Temperature Calibration with 1,2-Ethandiol

pulse program: zg0
compare with Experiment 2.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P0 :f1 channel - 45° ^{1}H transmitter pulse
D1 :300 sec – relaxation delay
TD :32 K
SW :8 ppm
O1 :middle of the ^{1}H NMR spectrum
NS :1
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :0.1 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 5.3

- Dynamic ^{1}H NMR Spectroscopy on Dimethylformamid

pulse program: zg0hd
compare with Experiment 4.1

Setting of the needed channels: F1: ^{1}H
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P0 :f1 channel - 45° ^{1}H transmitter pulse
PL14 :f2 channel - power level for decoupler attenuation corresponding to γB_2=10 Hz
D1 :300 sec - to equilibrate temperature
TD :32 K
SW :12 ppm
O1 :middle of the ^{1}H NMR spectrum
O2 :on resonance of the aldehyde proton
digmod :homodecoupling-digital
stable gas flow for temperature regulation
NS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :0.1 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT
for each temperature run an expanded plot of the signals of the methyl group

Experiment 5.4

- The Saturation Transfer Experiment

pulse program: zgdclo.mo
1D-sequence with a low power pulse and Waltz-decoupling. One signal is irradiated with a low power pulse and a change is observed in the intensity of another signal which is connected with the irradiated one by chemical exchange.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL9 :f1 channel - power level to saturate only the signal on resonance (70 dB)
PL12 :f2 channel - power level for CPD decoupling
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :0.1 sec – relaxation delay
D12 :20 usec – delay for power switching
TD :8 K
O1 :on resonance of low frequency methyl group signal
NS :8

RG :receiver gain for correct ADC input

P1 :f1 channel - 90° ^{13}C transmitter pulse
P6 :f1 channel - 25 sec pre-irradiation pulse at power level PL9
PCPD2 :f2 channel – 90° pulse for decoupling sequence
D11 :30 msec - delay for disk I/O
SW :25 ppm
O2 :middle of ^{1}H NMR spectrum

temperature is changed from 300 K up to 350 K

Processing parameters

SI :4 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :2 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 5.5

- Measurement of the Rotating Frame Relaxation Time $T_{1\rho}$

pulse program:

The $T_{1\rho}$ experiment measures the relaxation time in the rotating frame and provides a means to determine the rate constants k and the chemical shift difference $\Delta\nu$ in cases where the low temperature regime cannot be reached. The $T_{1\rho}$ relaxation time becomes an important parameter in 2D experiments which use a spin-lock, such as TOCSY or ROESY.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

Processing parameters

Chapter 6
- 1D Multipulse Sequences

Summary

Experiment	Pulse program	Description
6.1	t1irdc.mo	Measurement of the Spin-Lattice Relaxation Time T_1
6.2	cpmg	Measurement of the Spin-Spin Relaxation Time T_2
6.3	jmod	^{13}C NMR Spectra with SEFT
6.4	apt	^{13}C NMR Spectra with APT
6.5	ineptnd	The Basic INEPT Technique
6.6	ineptpnd	INEPT+
6.7	ineptrd	Refocused INEPT
6.8	iineptnd	Reverse INEPT
6.9	dept	DEPT-135
6.10	dept	Editing ^{13}C NMR Spectra with DEPT
6.11	pendant.mo	Multiplicity Determination with PENDANT
6.12	inad1d	1D-INADEQUATE
6.13	invbnd1d	The BIRD Filter
6.14	tango.mo	TANGO
6.15	inv4nd1d	The Heteronuclear Double Quantum Filter
6.16	exp6_16.mo	Purging with a Spin-Lock Pulse
6.17	zgpr	Water Suppression by Presaturation
6.18	p11	Water Suppression by the Jump and Return Method

Experiment 6.1
- Measurement of the Spin-Lattice Relaxation Time T_1

pulse program: t1irdc.mo
This is an inversion recovery experiment to measure the spin-lattice relaxation time T1.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse

PL12 :f2 channel - power level for CPD decoupling

CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2

D1 :60 sec – relaxation delay

D12 :20 usec – delay for power switching

P1 :f1 channel - 90° ^{13}C transmitter pulse

P2 :f1 channel - 180° ^{13}C transmitter pulse

PCPD2 :f2 channel – 90° pulse for decoupling sequence

D11 :30 msec - delay for disk I/O

TD :32 K
O1 :middle of the ^{13}C NMR spectrum
NS :8
VD :variable delay, taken from VD-LIST (0.5;1;3;6;10;16;24;50 [s])
TD1 :8 - number of experiments
define VD-LIST

SW :200 ppm
O2 :middle of ^{1}H NMR spectrum
DS :4
L4 :number of experiments=number of delays in VD-LIST, here 8
Parmod :2D
RG :receiver gain for correct ADC input

Processing parameters

au-program :splitser
WDW :EM
XF2 :transformation is only performed in the F2 direction

SI :16 K
LB :2 Hz
phase correction :to adjust phase, read spectrum number 8, in which all signals have positive phase, and transfer this phase correction to all other spectra (PH_mod: pk)

plot :use XWINPLOT

Experiment 6.2

- Measurement of the Spin-Spin Relaxation Time T_2

pulse program: cpmg
1D-sequence to measure the relaxation time T2 using the Carr-Purcell-Meiboom-Gill sequence.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

P1 :f1 channel - 90° ^{1}H transmitter pulse

P2 :f1 channel - 180° ^{1}H transmitter pulse

D1 :150 sec – relaxation delay
D20 :10 msec – fixed echo time to allow elimination of diffusion and J-mod. effects

D11 :30 msec - delay for disk I/O

TD :1 K
O1 :on ^{1}H resonance
NS :8
VC :variable loop counter, taken from vc-list (2; 20; 50; 100; 200; 300; 400; 500; 750; 1000)
Parmod :2D
TD1 :10 – number of experiments
DE :as short as possible

SW :500 Hz

DS :16
L4 :number of experiments = number of values in vc-list (10)

define VCLIST
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
LB(F2) :2 Hz
XF2P :phase correction only for the rows

WDW(F2) :EM
XF2 :transformation in F2 direction
plot :use XWINPLOT

Experiment 6.3

- ^{13}C NMR Spectra with SEFT

pulse program: jmod
This experiment uses the SEFT (Spin-Echo Fourier Transform) technique. It is the simplest method of encoding the multiplicity of a ^{13}C signal into the phase of a fully decoupled ^{13}C NMR spectrum.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse

P1 :f1 channel - 90° ^{13}C transmitter pulse

P2 :f1 channel - 180° ^{13}C transmitter pulse

PL12 :f2 channel - power level for CPD decoupling

PCPD2 :f2 channel – 90° pulse for decoupling sequence

CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2

D1 :4 sec – relaxation delay

D13 :3 usec - short delay

D20 :1/[J(C,H)]= 7 msec, calculated from $^{1}J(C,H)$=140 Hz

TD :64 K

SW :200 ppm

O1 :middle of the ^{13}C NMR spectrum

O2 :middle of ^{1}H NMR spectrum

NS :16

DS :4

DE :as short as possible

RG :receiver gain for correct ADC input

Processing parameters

SI :32 K

BC_mod :quad

WDW :EM

LB :2 Hz

FT :Fourier transformation

phase correction :adjust the phase for the signals of the methyl groups to be positive and for the carboxyl nucleus negative

baseline correction :ABS

plot :use XWINPLOT

Experiment 6.4

- ^{13}C NMR Spectra with APT

pulse program: apt
The ATP (Attached Proton Test) technique differentiates between C, CH, CH_2 and CH_3 groups.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse

P0 :f1 channel - 45° ^{13}C transmitter pulse

P2 :f1 channel - 180° ^{13}C transmitter pulse

PL12 :f2 channel – power level for CPD

PCPD2 :f2 channel – 90° pulse for

decoupling

CPD2 :WALTZ16 – CPD decoupling sequence, defined by cpdprg2

decoupling sequence

D1 :2 sec – relaxation delay
D11 :30 msec - delay for disk I/O
D20 :1/[J(C,H)]= 7 msec, calculated from ^{1}J(C,H)=140 Hz
D12 :20 usec - delay for power switching
D21 :set equal to preacquisition delay DE
TD :64 K
SW :200 ppm
O1 :middle of the ^{13}C NMR spectrum
O2 :middle of ^{1}H NMR spectrum
NS :512
DS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :32 K
BC_mod :quad
WDW :EM
LB :2 Hz
FT :Fourier transformation
phase correction :adjust the phase for the TMS signal positive and for the carboxyl nucleus negative
referencing :set the TMS signal to 0 ppm
baseline correction :ABS
plot :use XWINPLOT

Experiment 6.5

- The Basic INEPT Technique

pulse program: ineptnd

The INEPT (Insensitive Nuclei Enhanced by Polarization Transfer) experiment increase the sensitivity of hetero nuclei by a polarization transfer from protons via X, H spin coupling. The result is a coupled X-nucleus NMR spectrum.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse
PL2 :f2 channel - high power level for ^{1}H decoupler pulse
P3 :f2 channel - 90° ^{1}H decoupler pulse
P4 :f2 channel - 180° ^{1}H decoupler pulse
D1 :10 sec – relaxation delay
D4 :1/[4J(C,H)]= 1.18 msec, calculated from ^{1}J(C,H)=212 Hz
TD :4 K
SW :500 Hz
O1 :on resonance of ^{13}C NMR signal
O2 :on resonance of ^{1}H NMR signal
NS :1 for the first and 4 for the second experiment
DS :16
RG :receiver gain for correct ADC input

Processing parameters

SI :2 K
BC_mod :quad
WDW :EM
LB :2 Hz
FT :Fourier transformation
phase correction :adjust the phase for the signals positive

and negative

baseline correction :ABS **plot** :use XWINPLOT

Experiment 6.6
- INEPT+

pulse program: ineptpnd
This INEPT version yields coupled polarization-enhanced NMR spectra of X-nuclei with correct intensities within the multiplets.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL2 :f2 channel - high power level for ^{1}H decoupler pulse
D1 :2 sec – relaxation delay
D4 :1/[4J(C,H)]= 1.78 msec, calculated from $^{1}J(C,H)$=140 Hz
TD :64 K
O1 :middle of the ^{13}C NMR spectrum
NS :128
RG :receiver gain for correct ADC input

P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse
P3 :f2 channel - 90° ^{1}H decoupler pulse
P4 :f2 channel - 180° ^{1}H decoupler pulse
D3 :0.375/[J(C,H)]= 2.68 msec, calculated from $^{1}J(C,H)$=140 Hz
SW :200 ppm
O2 :middle of ^{1}H NMR spectrum
DS :16

Processing parameters

SI :32 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS

BC_mod :quad
LB :2 Hz
phase correction :adjust the phase for the signals positive and negative
plot :use XWINPLOT

Experiment 6.7
- Refocused INEPT

pulse program: ineptrd
This variant of INEPT spectroscopy yields proton-decoupled and polarization-enhanced NMR spectra of X-nuclei.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL2 :f2 channel - high power level for ^{1}H

P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse
P3 :f2 channel - 90° ^{1}H decoupler pulse

decoupler pulse

P4 :f2 channel - 180° ^{1}H decoupler pulse

PL12 :f2 channel - power level for CPD decoupling

PCPD2 :f2 channel – 90° pulse for decoupling sequence

CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2

D1 :2 sec – relaxation delay

D3 :1/[3J(C,H)]= 2.38 msec, calculated from ^{1}J(C,H)=140 Hz

D4 :1/[4J(C,H)]= 1.78 msec, calculated from ^{1}J(C,H)=140 Hz

D12 :20 usec - delay for power switching

TD :64 K

SW :200 ppm

O1 :middle of the ^{13}C NMR spectrum

O2 :middle of ^{1}H NMR spectrum

NS :128

DS :16

RG :receiver gain for correct ADC input

Processing parameters

SI :32 K

BC_mod :quad

WDW :EM

LB :2 Hz

FT :Fourier transformation

phase correction :adjust the phase for the signals positive and negative

baseline correction :ABS

plot :use XWINPLOT

Experiment 6.8

- Reverse INEPT

pulse program: iineptnd

This experiment is an inverse INEPT sequence without decoupling. Starting from X-nucleus magnetization, the X, H spin coupling is observed by proton detection.

Setting of the needed channels: F1: ^{1}H

F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

P1 :f1 channel - 90° ^{1}H transmitter pulse

P2 :f1 channel - 180° ^{1}H transmitter pulse

PL2 :f2 channel - high power level for ^{13}C decoupler pulse

P3 :f2 channel - 90° ^{13}C decoupler pulse

P4 :f2 channel - 180° ^{13}C decoupler pulse

D1 :30 sec – relaxation delay

D4 :1/[4J(C,H)]= 1.19 msec, calculated from ^{1}J(C,H)=214 Hz

TD :4 K

SW :500 Hz

O1 :on resonance of ^{1}H NMR signal

O2 :on resonance of ^{13}C NMR signal

NS :8

DS :16

RG :receiver gain for correct ADC input

Processing parameters

SI :2 K

BC_mod :quad

WDW :EM

LB :0.5 Hz

FT :Fourier transformation

phase correction :adjust the phase for the satellites positive and negative

baseline correction :ABS

plot :use XWINPLOT

Experiment 6.9

- DEPT-135

pulse program: dept
The DEPT (Distortionless Enhancement by Poarization Transfer) experiment uses a polarization transfer from protons to an X-nucleus to increase the signal strength.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL2 :f2 channel - high power level for ^{1}H decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :2 sec – relaxation delay
D12 :20 usec – delay for power switching
TD :64 K
O1 :middle of the ^{13}C NMR spectrum
NS :512
RG :receiver gain for correct ADC input

P0 :f1 channel - 135° ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse
P3 :f2 channel - 90° ^{1}H decoupler pulse
P4 :f2 channel - 180° ^{1}H decoupler pulse
PCPD2 :f2 channel – 90° pulse for decoupling sequence
D2 :1/[2J(C,H)]= 3.5 msec, calculated from $^{1}J(C,H)$=140 Hz
SW :200 ppm
O2 :middle of ^{1}H NMR spectrum
DS :8

Processing parameters

SI :32 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :2 Hz
phase correction :adjust the phase for the TMS signal positive
plot :use XWINPLOT

Experiment 6.10

- Editing ^{13}C NMR Spectra with DEPT

pulse program: dept
compare with Experiment 6.9

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL2 :f2 channel - high power level for ^{1}H decoupler pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse
P3 :f2 channel - 90° ^{1}H decoupler pulse

P4 :f2 channel - 180° ^{1}H decoupler pulse

P0 :f1 channel – use 45°, 90° and 135° ^{1}H decoupler pulse for three successive spectra a, b and c. Spectrum a will give the signals of CH, CH_2 and CH_3 groups positive; b gives only the signals of CH groups, and c gives the signals of CH and CH_3 groups positive and the signals of CH_2 groups negative. The second spectrum b gives a clear indication whether the decoupler pulse is determined correctly.

PL12 :f2 channel - power level for CPD decoupling

PCPD2 :f2 channel – 90° pulse for decoupling sequence

CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2

D1 :2 sec – relaxation delay

D2 :1/[2J(C,H)]= 3.5 msec, calculated from ^{1}J(C,H)=140 Hz

D12 :20 usec – delay for power switching

TD :64 K

SW :200 ppm

O1 :middle of the ^{13}C NMR spectrum

O2 :middle of ^{1}H NMR spectrum

NS :512

DS :8

RG :receiver gain for correct ADC input

Processing parameters

SI :32 K

BC_mod :quad

WDW :EM

LB :2 Hz

FT :Fourier transformation

phase correction :adjust the phase for spectrum a to pure absorption, for spectrum b also and for spectrum c adjust the phase of the CH and the CH_3 groups positive and the CH_2 groups negative. For editing purposes the three spectra have to be further manipulated. This is done in the dual mode. Substraction of b from a yields spectrum d, where the signals of CH_2 and CH_3 groups both remain positive. Substraction of b from c yields spectrum e, where the signals of CH_2 are negative and those of the CH_3 groups remain positive. Substraction of e from d yields f with only signals of CH_2 groups, whereas addition of e to d yields spectrum g with only signals of the CH_3 groups.

baseline correction :ABS

plot :use XWINPLOT

Experiment 6.11

- Multiplicity Determination with PENDANT

pulse program: pendant.mo

The PENDANT (Polarization Enhancement During Attached Nucleus Testing) method is a method for distinguishing CH, CH_2, CH_3 and quaternary carbons with the same sensitivity like DEPT.

Setting of the needed channels: F1: ^{13}C

F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse

P1 :f1 channel - 90° ^{13}C transmitter pulse

P2 :f1 channel - 180° ^{13}C transmitter pulse

PL2 :f2 channel - high power level for ^{1}H decoupler pulse

P3 :f2 channel - 90° ^{1}H decoupler pulse

P4 :f2 channel - 180° ^{1}H decoupler pulse

PL12 :f2 channel - power level for CPD decoupling

PCPD2 :f2 channel – 90° pulse for decoupling sequence

CPD2 :WALTZ16 - CPD decoupling

sequence, defined by cpdprg2

D1 :2 sec – relaxation delay

D4 :1/[4J(C,H)]= 1.72 msec, calculated from ^{1}J(C,H)=145 Hz

D12 :20 usec – delay for power switching

D15 :5/[8J(C,H)]= 4.31 msec, calculated from ^{1}J(C,H)=145 Hz

TD :64 K

SW :250 ppm

O1 :middle of ^{13}C NMR spectrum

O2 :middle of ^{1}H NMR spectrum

NS :16

DS :8

RG :receiver gain for correct ADC input

DE :as short as possible

Processing parameters

SI :32 K

BC_mod :quad

WDW :EM

LB :2 Hz

FT :Fourier transformation

phase correction :adjust the phase for the signals of the methyl groups to be positive and for the carcoxyl nucleus negative.

baseline correction :ABS

plot :use XWINPLOT

Experiment 6.12

- 1D-INADEQUATE

pulse program: inad1d
This is a 1D-INADEQUATE sequence.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse

P1 :f1 channel - 90° ^{13}C transmitter pulse

P2 :f1 channel - 180° ^{13}C transmitter pulse

PL12 :f2 channel - power level for CPD decoupling

PCPD2 :f2 channel – 90° pulse for decoupling sequence

CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2

D1 :3 sec – relaxation delay

D4 :1/[4J(C,C)]= 7.6 msec, calculated from ^{1}J(C,C)=33 Hz

D11 :30 msec – delay for disk I/O

D13 :3 usec - short delay

TD :32 K

SW :60 ppm (spectral range for $C_6H_{13}OH$)

O1 :40 ppm downfield from TMS (middle of that range)

O2 :middle of ^{1}H NMR spectrum

NS :512

DS :16

RG :receiver gain for correct ADC input

Processing parameters

SI :64 K

BC_mod :quad

WDW :EM

LB :0.5 Hz

FT :Fourier transformation

phase correction :adjust the signals positive and negative

baseline correction :ABS

plot :use XWINPLOT

Experiment 6.13
- The BIRD Filter

pulse program: invbnd1d
With this experiment a suppression for signals from protons bond to ^{12}C is performed with a BIRD (Bilinear Rotation Decoupling) sandwich. It rotates the magnetization of the protons attached to ^{12}C into the -z direction of the rotating frame, but leaves the magnetization of the ^{13}C- bond protons unchanged. If one waits a suitable time after the BIRD sandwich, the signals of the protons bond to ^{12}C are at the null point and therefore not excited during the following pulse sequence. The sequence is without decoupling.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
D1 :60 sec – relaxation delay
D7 :20 sec - to be varied
TD :4 K
O1 :on ^{1}H resonance
NS :4
RG :receiver gain for correct ADC input

P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P4 :f2 channel - 180° ^{13}C decoupler pulse
D2 :1/[2J(C,H)]= 2.38 msec, calculated from $^{1}J(C,H)$=214 Hz
D13 :3 usec - short delay
SW :500 Hz
O2 :on ^{13}C resonance
DS :4

Processing parameters

SI :2 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :1 Hz
phase correction :correct the satellites positive
plot :use XWINPLOT

Experiment 6.14
- TANGO

pulse program: tango.mo
The TANGO (Testing for Adjacent Nuclei with a Gyration Operator) sequence introduces a 90° phase angle between the protons bond to ^{13}C and the protons bond to ^{12}C.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse

P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P4 :f2 channel - 180° ^{13}C decoupler pulse

D1 :20 sec – relaxation delay

D2 :1/[2J(C,H)]= 2.38 msec, calculated from ^{1}J(C,H)=214 Hz

D12 :20 usec – delay for power switching
TD :4 K
SW :500 Hz
O1 :on ^{1}H resonance
O2 :on ^{13}C resonance
NS :8
DS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :2 K
BC_mod :quad
WDW :EM
LB :1 Hz
FT :Fourier transformation
phase correction :correct the satellites positive.
baseline correction :ABS
plot :use XWINPLOT

Experiment 6.15

- The Heteronuclear Double Quantum Filter

pulse program : inv4nd1d
This experiment suppress the main signal of protons attached to ^{12}C or ^{14}N using a double quantum filter, where single quantum magnetization is filtered out by the phase cycle.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
D1 :200 sec – relaxation delay
D2 :1/[2J(C,H)]= 2.38 msec, calculated from ^{1}J(C,H)=214 Hz
D12 :20 usec – delay for power switching
D13 :3 usec
TD :4 K
SW :500 Hz
O1 :on ^{1}H resonance
O2 :on ^{13}C resonance
NS :8
DS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :2 K
BC_mod :quad
WDW :EM
LB :0.1 Hz
FT :Fourier transformation
phase correction :correct the satellites positive
baseline correction :ABS
plot :use XWINPLOT

Experiment 6.16

- Purging with a Spin-Lock Pulse

pulse program : exp6_16.mo
This experiment uses a spin-lock pulse to select protons attached to ^{13}C.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse, 3 dB was used here

P1 :f1 channel - 90° ^{1}H transmitter pulse

P2 :f1 channel - 180° ^{1}H transmitter pulse

P28 :f1 channel - spin-lock pulse, 2 msec, be sure, that your power is not to high! The transmitter pulse with power level pl1 should be not shorter than 6 usec.

PL2 :f2 channel - high power level for ^{13}C decoupler pulse

P4 :f2 channel - 180° ^{13}C decoupler pulse

D1 :10 sec – relaxation delay

D4 :1/[4J(C,H)]= 1.16 msec, calculated from $^{1}J(C,H)$=215 Hz

TD :4 K

SW :500 Hz

O1 :on ^{1}H resonance

O2 :on ^{13}C resonance

NS :1

DS :4

RG :receiver gain for correct ADC input

Processing parameters

SI :2 K

BC_mod :quad

WDW :EM

LB :0.1 Hz

FT :Fourier transformation

phase correction :correct the satellites up and down

baseline correction :ABS

plot :use XWINPLOT

Experiment 6.17

- Water Suppression by Presaturation

pulse program: zgpr
1D-sequence with F1 presaturation. Sometimes, there is a need to suppress the huge solvent signal.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

P1 :f1 channel - 90° ^{1}H transmitter pulse

PL9 :f1 channel - power level for presaturation

D1 :2 sec – relaxation delay

D12 :20 usec - delay for power switching

D13 :3 usec – short delay

TD :32 K

SW :10 ppm

O1 :on resonance of water signal

DS :2

NS :8

RG :receiver gain for correct ADC input

for inverse probeheads: spinner off

Processing parameters

SI :16 K

BC_mod :quad

WDW :no
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
FT :Fourier transformation
referencing :set the TMS signal to 0 ppm
plot :use XWINPLOT

Experiment 6.18

- Water Suppression by the Jump and Return Method

pulse program: p11
Water suppression with the jump and return method which does not affect exchangeable protons.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
D1 :2 sec – relaxation delay
TD :32 K
O1 :on resonance of water signal
NS :8
P0 :f1 channel - 90° ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D19 :125 usec - delay for binominal water suppression (D19=(1/2*d), d=distance of next null (in Hz))
SW :10 ppm
DS :2
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
plot :use XWINPLOT
BC_mod :quad
LB :0.1 Hz
phase correction :adjust the phase of the small signals to be positive, the water signal is in dispersion
referencing :set the TMS signal to 0 ppm

Chapter 7
- NMR Spectroscopy with Selective Pulses

Summary

Experiment	Pulse program	Description
7.1	selzg	Determination of a Shaped 90° ^{1}H Transmitter Pulse
7.2	decp90sp.mo	Determination of a Shaped 90° ^{1}H Decoupler Pulse
7.3	decp90sp.mo	Determination of a Shaped 90° ^{13}C Decoupler Pulse
7.4	dante.mo	Selective Excitation with DANTE
7.5	selco	SELCOSY
7.6	selincor.mo	SELINCOR: Selective Inverse H,C Correlation via ^{1}J (C,H)
7.7	selina	SELINQUATE
7.8	selmlzf	Selective TOCSY
7.9	selinapt.mo	INAPT
7.10	sellr.mo	Determination of Long-Range C,H Coupling Constants
7.11	selreso.mo	SELRESOLV
7.12	serf.mo	SERF

Experiment 7.1
- Determination of a Shaped 90° ^{1}H Transmitter Pulse

pulse program: selzg
An experiment to determine the 90° shaped pulse. It must be determined by varying the attenuation of the transmitter and not the pulse duration.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

SP1 :f1 channel - power level for shaped pulse, to be varied in steps of 2 dB, initial value 90 dB

D1 :20 sec – relaxation delay

TD :4 K

O1 :on ^{1}H resonance

PHCOR :difference in phases between power level SP1 and PL1

RG :receiver gain for correct ADC input

P1 :f1 channel - 90° ^{1}H transmitter pulse

P2 :f1 channel - 180° ^{1}H transmitter pulse

P11 :f1 channel – 90° (or 270°) shaped pulse, 50 msec was used here

SW :500 Hz

NS :1

Gaussian shape with 1024 data points was used

Processing parameters

SI :2 K

WDW :EM

BC_mod :quad

LB :0.1 Hz

FT :Fourier transformation

phase correction :adjust the phase to pure absorption.

baseline correction :ABS

Experiment 7.2

- Determination of a Shaped 90° ^{1}H Decoupler Pulse

pulse program: decp90sp.mo
This experiment is used to calibrate the shaped 90° decoupler pulse duration.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse

PL2 :f2 channel - high power level for ^{1}H decoupler pulse

PL21 :f1 channel - low power level, 15 dB was used here

SP2 :2 channel - power level for shaped pulse, initial value 80 dB, to be varied

D1 :10 sec – relaxation delay

TD :4 K

O1 :on ^{13}C resonance

NS :1

RG :receiver gain for correct ADC input

P1 :f1 channel - 90° ^{13}C transmitter pulse

P3 :f2 channel - leave at 0 for the determination of the correct attenuation of the shaped pulse. For phase determination set to hard 90° ^{1}H decoupler pulse.

P28 :f1 channel - spin-lock pulse, length=P13

P13 :f2 channel – 90° shaped pulse, 30 msec

D2 :1/[2J(C,H)]= 2.36 msec, calculated from ^{1}J(C,H)=212 Hz

SW :500 Hz

O2 :on ^{1}H resonance

Gaussian shape with 1024 data points was used

Processing parameters

SI :2 K

WDW :EM

FT :Fourier transformation

baseline correction :ABS

BC_mod :quad

LB :2 Hz

phase correction :adjust the satellites up and down

plot :use XWINPLOT

Experiment 7.3

- Determination of a Shaped 90° ^{13}C Decoupler Pulse

pulse program: decp90sp.mo
compare with Experiment 7.2

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

P1 :f1 channel - 90° ^{1}H transmitter pulse

PL2 :f2 channel - high power level for ^{13}C decoupler pulse

PL21 :f1 channel - low power level, typical attenuation 12 dB

SP2 :f2 channel - power level for shaped pulse, to be varied, initial value 80 dB

D1 :10 sec – relaxation delay

TD :4 K

O1 :on ^{1}H resonance

NS :1

RG :receiver gain for correct ADC input

P2 :f1 channel - 180° ^{1}H transmitter pulse

P3 :f2 channel - leave at 0 for the determination of the correct attenuation of the shaped pulse. For phase determination set to hard 90° ^{13}C decoupler pulse.

P28 :f1 channel - spin-lock pulse, length=P13

P13 :f2 channel – 90° shaped pulse, 10 msec

D2 :1/[2J(C,H)]= 2.33 msec, calculated from $^{1}J(C,H)$=215 Hz

SW :500 Hz

O2 :on ^{13}C resonance

Gaussian shape with 1024 data points was used

Processing parameters

SI :2 K

WDW :EM

FT :Fourier transformation

baseline correction :ABS

BC_mod :quad

LB :0.1 Hz

phase correction :adjust the satellites up and down

plot :use XWINPLOT

Experiment 7.4

- Selective Excitation with DANTE

pulse program: dante.mo
1D-sequence to excite a single resonance selectively, also with older instruments possible.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL9 :f1 channel - low power level for ^{13}C transmitter pulse

PL12 :f2 channel - power level for CPD decoupling

CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2

D1 :2 sec – relaxation delay

D15 :0.5 msec –yielding a total length of DANTE excitation of 25 msec

TD :4 K

O1 :on ^{13}C resonance of the selected methyl group

DS :4

RG :receiver gain for correct ADC input

P0 :f1 channel - 1° ^{13}C transmitter pulse

PCPD2 :f2 channel – 90° pulse for decoupling sequence

D12 :20 usec - delay for power switching

L4 :number of pulse P0, here 50 was used

SW :10 ppm

O2 :middle of ^{1}H NMR spectrum

NS :128

Processing parameters

SI :2 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :0.5 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 7.5

- SELCOSY

pulse program: selco
This is a 1D variant of the 2D COSY. The selective COSY method yields the same connectivity information as the homonuclear decoupling technique.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
SP1 :f1 channel - power level for shaped pulse
D1 :2 sec – relaxation delay
D14 :~1/[2J(H,H)], typically 37 msec, calculated from $^{1}J(H,H)$=8 Hz
TD :32 K
O1 :on resonance of selected signal or use SPOFFS
NS :16
RG :receiver gain for correct ADC input
P1 :f1 channel - 90° ^{1}H transmitter pulse
P11 :f1 channel – 90° shaped pulse, 50 msec was used here
D13 :3 usec - short delay
SW :10 ppm
Gaussian shape with 1024 data points was used
DS :4

Processing parameters

SI :16 K
WDW :EM
FT :Fourier transformation
plot :use XWINPLOT
BC_mod :quad
LB :0.1 Hz
phase correction :note that the selective COSY produces antiphase signals of the active coupling partners

Experiment 7.6

- SELINCOR: Selective Inverse H,C Correlation via ^{1}J (C,H)

pulse program: selincor.mo
This experiment is the selective 1D version of the 2D inverse H,C correlation. The experiment correlates a selected carbon atom with the attached proton via one bond C,H coupling, using proton sensitivity for observation.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL0 :f2 channel - 120 dB fixed power level

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

P1 :f1 channel - 90° ^{1}H transmitter pulse

P2 :f1 channel - 180° ^{1}H transmitter pulse

PL2 :f2 channel - high power level for ^{13}C decoupler pulse

P3 :f2 channel - 90° ^{13}C decoupler pulse

P4 :f2 channel - 90° ^{13}C decoupler pulse

SP2 :f2 channel - power level for shaped pulse

P13 :f2 channel - 90° or 270° shaped pulse, 5 msec

D1 :1 sec – relaxation delay

D2 :1/[2J(C,H)]= 3.57 msec, calculated from $^{1}J(C,H)$=140 Hz

D7 :2.5 sec – BIRD delay, to be optimized for minimum FID; observe in the set-up mode the incoming FID and adjust D7 for minimum intensity

D20 :same length as selective pulse P13, 5 msec was used here

TD :32 K

SW :10 ppm

O1 :middle of ^{1}H NMR signal

O2 :on resonance of selected ^{13}C nucleus

NS :32

DS :4

Gaussian shape with 1024 data points was used

RG :receiver gain for correct ADC input

Processing parameters

SI :16 K

BC_mod :quad

WDW :EM or MC (if the phase of the satellites are not very pure)

LB :0.1 Hz

FT :Fourier transformation

phase correction :adjust the phase to pure absorption (WDW=MC no phase correction is necessary)

plot :use XWINPLOT

Experiment 7.7

- SELINQUATE

pulse program: selina

This is the selective version of the INADEQUATE (Incredible Natural Abundance Double QUAtum Transfer). It is possible to measure specific ^{13}C, ^{13}C coupling constants over one or more bonds selectively with the high digital resolution of an 1D method. The experiment yields connectivity information for the irradiated carbon nucleus and ^{13}C, ^{13}C spin coupling constants with high accuracy.

Setting of the needed channels: F1: ^{13}C

F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse

P1 :f1 channel - 90° ^{13}C transmitter pulse

P2 :f1 channel - 180° ^{13}C transmitter pulse

SP1 :f1 channel - power level for shaped pulse

P11 :f1 channel - 270° shaped pulse, 10 msec was used here

PL12 :f2 channel - power level for CPD decoupling

PCPD2 :f2 channel - 90°pulse for decoupling sequence

CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :4 sec – relaxation delay
D4 :1/[4J(C,C)]= 7.6 msec, calculated from ^{1}J(C,C)=33 Hz, for long range couplings J(C,C)=4 Hz= 62.5 msec
TD :16 K
O1 : on resonance of selected signal or use SPOFFS
NS :256
PHCOR3 :difference of phases between power level SP1 and PL1
RG :receiver gain for correct ADC input

D11 :30 msec - delay for disk I/O
D13 :3 usec - short delay
SW :23 ppm
O2 :middle of ^{1}H NMR spectrum
DS :4
Gaussian shape with 1024 data points was used

Processing parameters

SI :8 K
WDW :EM
FT :Fourier transformation

BC_mod :quad
LB :2 Hz
phase correction :.note that the experiment yields 13C satellites in antiphase. The residual signal of the molecule containing only one 13C atom should not be used for phasing.

plot :use XWINPLOT

Experiment 7.8
- Selective TOCSY

pulse program: selmlzf.mo
This experiment is the selective 1D version of the 2D TOCSY (TOtal Correlation SpectroscopY) method. One proton is excited by a shaped pulse and this produces a response from all protons that are connected by spin coupling within a chain.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL0 :f1 channel - 120 dB, fixed power level
PL1 :f1 channel - high power level for ^{1}H transmitter pulse
SP1 :f1 channel - power level for shaped pulse
PL10 :f1 channel - power level for TOCSY-spinlock, 12 dB was used here

P1 :f1 channel - 90° ^{1}H transmitter pulse
P11 :f1 channel - 270° shaped pulse, 50 msec was used here
P5 :f1 channel - 60° low power pulse
P6 :f1 channel - 90° low power pulse [40 usec]
P7 :f1 channel - 180° low power pulse
P17 :f1 channel - trim pulse [2.5 msec]

D1 :2 sec – relaxation delay
D9 :200 msec – TOCSY mixing time
D13 :3 usec – short delay

D11 :30 msec - delay for disk I/O
D14 :delay for evolution after shaped pulse: for self-refocussing pulse 20 usec

VD :variable delay, taken from vd-list to be used for z-filter:
Example for z-filter list:
0.004s;0.016s;0.010s;0.006s;0.004s;0.010s;0.017s;0.011s;0.018s;0.012s
L4 :number of delays in VD-list (10)
TD :32 K
SW :10 ppm
O1 :on resonance of selected signal or use SPOFFS
define VDLIST
PHCOR1 :difference in phases between power level SP1 and PL10
Gaussian shape with 1000 data points was used
NS :8
DS :4
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :0.1 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 7.9

- INAPT

pulse program: selinapt.mo
This experiment is the selective version of INEPT. Here only a particular proton is excited and used for polarization transfer, in order to identify ^{13}C nuclei that are connected to this proton via spin-spin coupling. The experiment is mainly used for detecting long-range interactions and provides a good method for assigning quaternary carbon nuclei.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL0 :f2 channel - 120 dB, fixed power level
PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse
SP2 :f2 channel - power level for shaped pulse, here 67 dB was used
P13 :f2 channel - 90°° rectangular shaped ^{1}H decoupler pulse, here 20 msec
P14 :f2 channel - 180° rectangular shaped ^{1}H decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :3 sec – relaxation delay
D20 :10 msec - fixed delay
D21 :20 msec – fixed delay
TD :32 K
SW :55 ppm
O1 :center of aromatic region of the ^{13}C NMR spectrum
O2 :on resonance of selected ^{1}H NMR signal
Rectangular shaped pulse with 1000 data points was used
NS :64
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :2 Hz
phase correction :note that the sign of the signals may be positive or negative
plot :use XWINPLOT

Experiment 7.10

- Determination of Long-Range C,H Coupling Constants

pulse program: sellr.mo

The experiment presented here demonstrates a 2D method related to 2D J-resolved spectroscopy and employing a selective pulse. It yields directly the desired spin coupling constant of a chosen C,H pair free of other passive spin couplings. Unlike the original method the pulse sequence given here uses a shaped RE-BURP pulse.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse
SP2 :f2 channel - power level for shaped RE-BURP pulse, 46 dB was used
P14 :f2 channel - 180°shaped pulse, 40 msec was used here
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D0 :3 usec – incremented delay
D1 :2 sec - relaxation delay
D11 :30 msec – delay for disk I/O
D12 :20 usec - delay for power switching
Parmod :2D
ND0 :2
TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :200 ppm
SW1 :50 Hz
O1 :middle of the ^{13}C NMR spectrum
O2 :on resonance of the methyl group at 1.8 ppm
NS :8
DS :16
RE-BURP shape with 256 points was used
IN0 :1/[2*SW1]
DE :as small as possible
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :SINE
SSB(F2) :2
PH-mod(F2) :no
MC2 :QF
SI(F1) :256 W
WDW(F1) :SINE
SSB(F1) :2
PH-mod(F1) :mc
XFB :fourier transformation in both directions
phase correction :not necessary
plot :use XWINPLOT

Experiment 7.11
- SELRESOLV

pulse program: selreso.mo
The experiment presented here demonstrates a 2D J-resolved spectroscopy and employing a selective pulse. It yields directly the desired spin coupling constant of a chosen C,H pair independent of other passive spin couplings. In contrast to Experiment 7.10, however, the SELRESOLV method is a proton detected experiment and hence more sensitivity.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
PL19 :f1 channel - power level for CPD decoupling, 28 dB
SP2 :f2 channel - power level for shaped pulse, here 66 dB was used
CPD1 :WALTZ16 - CPD decoupling sequence, defined by cpdprg1
D0 :3 usec – incremented delay
D1 :6 sec – relaxation delay
D6 :1/[2J(C,H)]= 50 msec, calculated from $^{n}J(C,H)$=10 Hz
Parmod :2D
TD2 :2 K data points in F2
SW2 :1 ppm
O1 :center of methyl group region of ^{1}H NMR spectrum
NS :16
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
PCPD1 :f1 channel - 90° pulse for decoupling sequence, 100 usec
P13 :f2 channel - 90° shaped pulse, 10 msec was used here
half Gaussian shape
D11 :30 msec - delay for disk I/O
D12 :20 usec - delay for power switching
ND0 :2
TD1 :32 data points in F1
SW1 :45 Hz
O2 :on resonance of the olefinic carbon atom C-2 at 123.6 ppm
DS :16
DE :as small as possible

Processing parameters

SI(F2) :2 K
WDW(F2) :SINE
SSB(F2) :0
PH-mod(F2) :no
MC2 :QF

phase correction :not necessary

SI(F1) :128 W
WDW(F1) :SINE
SSB(F1) :0
PH-mod(F1) :mc
XFB :fourier transformation in both directions
plot :use XWINPLOT

Experiment 7.12
- SERF

pulse program: serf.mo
The SERF (Selective ReFocussing) experiment is a 2D method. It directly yields the desired coupling constant of a chosen spin pair without other passive spin couplings.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
SP1 :f1 channel - power level for E-BURP2, here 55 dB was used
SP5 :f1 channel - power level for RE-BURP, here 45 dB was used
P28 :f1 channel - 5 msec purging pulse
P11 :f1 channel - 90° shaped pulse, E-BURP2 pulse, 50 msec length
P12 :f1 channel - 180° shaped pulse, double selective RE-BURP pulse, 50 msec length
D0 :3 usec – incremented delay
D1 :2 sec – relaxation delay
D12 :20 usec - delay for power switching
Parmod :2D
ND0 :2
TD2 :1 K data points in F2
TD1 :64 data points in F1
SW2 :8 ppm
SW1 :50 Hz
O1 :middle of ^{1}H NMR spectrum
NS :4
DS :16
IN0 :1/[2*SW1]
DE :as small as possible
RG :receiver gain for correct ADC input
calibrate the double selective RE-BURP shape, so that this pulse acts simultaneously on the olefinic proton at 6.9 ppm and the methyl group at 1.8 ppm
the E-BURP2 shape acts on the olefinic signal at 6.9 ppm

Processing parameters

SI(F2) :512 W
SI(F1) :128 W
WDW(F2) :SINE
WDW(F1) :SINE
SSB(F2) :0
SSB(F1) :0
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
phase correction :not necessary
plot :use XWINPLOT

Chapter 8
- Auxiliary Reagents, Quantitative Determinations and Reaction Mechanism

Summary

Experiment	Pulse program	Description
8.1	zg30	Signal Separation Using a Lanthanide Shift Reagent
8.2	zg30	Signal Separation of Enantiomers Using a Chiral Shift Reagent
8.3	zg30	Signal Separation of Enantiomers Using a Chiral Solvating Agent
8.4	zg30	Determination of Enantiomeric Purity with Pirkle's Reagent
8.5	zg0dc	Determination of Enantiomeric Purity by ^{31}P NMR
8.6	zg30	Determination of Absolute Configuration by the Advanced Mosher Method
8.7	zg30	Aromatic Solvent-Induced Shift (ASIS)
8.8	zg30	NMR Spectroscopy of OH-Protons and H/D Exchange
8.9	zgdc30	Isotope Effects on Chemical Shielding
8.10	zgdc30	pK_a Determination with ^{13}C NMR
8.11	zg0dc	The Relaxation Reagent $Cr(acac)_3$
8.12	zg30	Determination of Paramagnetic Susceptibility by NMR
8.13	zg0 and zg0dc	^{1}H and ^{13}C NMR of Paramagnetic Compounds
8.14	zgdc30	The CIDNP Effect
8.15	zg0	Quantitative ^{1}H NMR Spectroscopy: Determination of the Alcohol Content of Polish Vodka
8.16	zgig	Quantitative ^{13}C NMR Spectroscopy with Inverse Gated ^{1}H-Decoupling
8.17	zg30	NMR Using Liquid-Crystal Solvents

Experiment 8.1
- Signal Separation Using a Lanthanide Shift Reagent

pulse program: zg30
compare with Experiment 3.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D1 :0.1 sec – relaxation delay
TD :32 K
SW :15 ppm
O1 :middle of the ^{1}H NMR spectrum
NS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :0.1 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 8.2

- Signal Separation of Enantiomers Using a Chiral Shift Reagent

pulse program: zg30
compare with Experiment 3.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D1 :0.1 sec – relaxation delay
TD :32 K
SW :15 ppm
O1 :middle of the ^{1}H NMR spectrum (also signals at -3 ppm)
NS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :0.1 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 8.3

- Signal Separation of Enantiomers Using a Chiral Solvating Agent

pulse program: zg30
compare with Experiment 3.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D1 :0.1 sec – relaxation delay
TD :32 K
SW :15 ppm
O1 :middle of the ^{1}H NMR spectrum
NS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :0.1 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 8.4

- Determination of Enantiomeric Purity with Pirkle's Reagent

pulse program: zg30
compare with Experiment 3.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D1 :0.1 sec – relaxation delay
TD :32 K
SW :15 ppm
O1 :middle of the ^{1}H NMR spectrum
NS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :GM
GB :0.2
LB :-0.5 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 8.5

- Determination of Enantiomeric Purity by ^{31}P NMR

pulse program: zg0dc
compare with Experiment 2.2

Setting of the needed channels: F1: ^{31}P
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C
P0 :f1 channel - 30° ^{13}C transmitter pulse

transmitter pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :2 sec – relaxation delay
D11 :30 msec - delay for disk I/O
TD :16 K
SW :20 ppm
O1 :middle of the ^{31}P NMR spectrum
O2 :middle of ^{1}H NMR spectrum
NS :32
RG :receiver gain for correct ADC input

Processing parameters
SI :8 K
BC_mod :quad
WDW :EM
LB :3 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
integration :is done with ABS or can be done manual
referencing :reference against external 85% H_3PO_4 with δp=0
baseline correction :ABS
plot :use XWINPLOT

Experiment 8.6
- Determination of Absolute Configuration by the Advanced Mosher Method

pulse program: zg30
compare with Experiment 3.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters
measure both solutions with the same parameters
PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
D1 :0.1 sec – relaxation delay
TD :32 K
SW :20 ppm
O1 : middle of ^{1}H NMR spectrum
NS :8
RG :receiver gain for correct ADC input

Processing parameters
process both FIDs with the same parameters
SI :16 K
BC_mod :quad
WDW :EM
LB :0.1 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT
use the dual display mode to extract the chemical shift differences of the two spectra

Experiment 8.7
- Aromatic Solvent-Induced Shift (ASIS)

pulse program: zg30
compare with Experiment 3.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

record the spectra of the compound dissolved in both solutions

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
D1 :0.1 sec – relaxation delay
TD :32 K
SW :20 ppm
O1 : middle of ^{1}H NMR spectrum
NS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :0.1 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
referencing :reference both spectra to δH=0 and inspect the aromatic region
plot :use XWINPLOT

Experiment 8.8

- NMR Spectroscopy of OH-Protons and H/D Exchange

pulse program: zg30
compare with Experiment 3.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

record a spectrum, remove the tube from the magnet, add a drop of D_2O, and shake the NMR tube thoroughly and record again a spectrum

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
D1 :0.1 sec – relaxation delay
TD :32 K
SW :20 ppm
O1 : middle of ^{1}H NMR spectrum
NS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :0.1 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 8.9
- Isotope Effects on Chemical Shielding

pulse program: zgdc30
compare with Experiment 3.2

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

first measure mixture of the deuterated solvents alone, then add the mixture of the undeuterated solvents and repeat the measurement

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel –90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :1 sec – relaxation delay
D11 :30 msec - delay for disk I/O
TD :64 K
SW :200 ppm
O1 :middle of the ^{13}C NMR spectrum
O2 :middle of ^{1}H NMR spectrum
NS :256
RG :receiver gain for correct ADC input

Processing parameters

SI :64 K
BC_mod :quad
WDW :EM
LB :0.3 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 8.10
- pK_a Determination with ^{13}C NMR

pulse program: zgdc30
compare with Experiment 3.2

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel –90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :2 sec – relaxation delay
D11 :30 msec - delay for disk I/O
TD :64 K
SW :200 ppm
O1 :middle of the ^{13}C NMR spectrum
O2 :middle of ^{1}H NMR spectrum
NS :32
RG :receiver gain for correct ADC input

Processing parameters

SI :32 K
BC_mod :quad
WDW :EM
LB :2 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 8.11

- The Relaxation Reagent $Cr(acac)_3$

pulse program: zg0dc
compare with Experiment 2.2

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P0 :f1 channel - 45° ^{13}C transmitter pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel –90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :0.5 sec – relaxation delay
D11 :30 msec - delay for disk I/O
TD :64 K
SW :250 ppm
O1 :middle of the ^{13}C NMR spectrum
O2 :middle of ^{1}H NMR spectrum
NS :64
RG :receiver gain for correct ADC input

Processing parameters

SI :32 K
BC_mod :quad
WDW :EM
LB :2 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 8.12

- Determination of Paramagnetic Susceptibility by NMR

pulse program: zg30
compare with Experiment 3.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D1 :0.1 sec – relaxation delay
TD :32 K
SW :15 ppm
O1 :middle of the ^{1}H NMR spectrum
NS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :0.1 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 8.13

- ^{1}H and ^{13}C NMR of Paramagnetic Compounds

a) pulse program: zg0
compare with Experiment 2.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse, here 3 dB was used
D1 :0.1 sec – relaxation delay
TD :64 K
O1 :25 ppm to lower frequencies from TMS signal
NS :8
P0 :f1 channel - 45° ^{1}H transmitter pulse
SW :110pm
RG :receiver gain for correct ADC input

Processing parameters

SI :32 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :5 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

b) pulse program: zg0dc
compare with Experiment 2.2

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL12 :f2 channel - power level for CPD decoupling
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :0.1 sec – relaxation delay
TD :64 K
O1 :400 ppm to higher frequencies from TMS signal
NS :2048
P0 :f1 channel - 45° ^{13}C transmitter pulse
PCPD2 :f2 channel –90° pulse for decoupling sequence
SW :990 ppm
O2 :on resonance of the previously determined ^{1}H NMR frequency of the cobaltocene signal
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :100 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 8.14

- The CIDNP Effect

pulse program: zgdc30
compare with Experiment 3.2

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL12 :f2 channel – power level for CPD decoupling
CPD2 :WALTZ16 – CPD decoupling sequence, defined by cpdprg2
D1 :1 sec – relaxation delay
TD :32 K
O1 :middle of the ^{13}C NMR spectrum
NS :16
prepare with edc 20 data sets and use the au-program multizg to measure the 20 spectra
P1 :f1 channel - 90° ^{13}C transmitter pulse
PCPD2 :f2 channel –90° pulse for decoupling sequence
D11 :30 msec - delay for disk I/O
SW :250 ppm
O2 :middle of ^{1}H NMR spectrum
RG :receiver gain for correct ADC input
set the temperature to 120°C and start immediately the automatic program

Processing parameters

SI :16 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :2 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 8.15

- Quantitative ^{1}H NMR Spectroscopy: Determination of the Alcohol Content of Polish Vodka

pulse program: zg0
compare with Experiment 2.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
D1 :5 sec – relaxation delay
TD :32 K or more (use a large data set to
P0 :f1 channel - 45° ^{1}H transmitter pulse
SW :10 ppm

get 10 points/Hz digital resolution)
O1 :middle of the ^{1}H NMR spectrum
NS :16 (obtain a good S/N, at least 35:1) **RG** :receiver gain for correct ADC input

Processing parameters
SI :64 K **BC_mod** :quad
WDW :EM **LB** :0.1 Hz
FT :Fourier transformation **phase correction :**adjust the phase to pure absorption.
integration :manual **baseline correction** :ABS
plot :use XWINPLOT

Experiment 8.16
- Quantitative ^{13}C NMR Spectroscopy with Inverse Gated ^{1}H-Decoupling

pulse program: zgig
1D-sequence with inverse gated decoupling, using a 90° pulse. This experiment yields ^{1}H-decoupled NMR spectra of X-nuclei without signal enhancement by the nuclear Overhauser effect.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters
PL1 :f1 channel - high power level for ^{13}C transmitter pulse **P1** :f1 channel - 90° ^{13}C transmitter pulse
PL12 :f2 channel - power level for CPD decoupling **PCPD2** :f2 channel - 90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :10 sec – relaxation delay **D11** :30 msec - delay for disk I/O
TD :2 K (short aq to avoid NOE build-up during acquisition) **SW** :20 ppm
O1 :middle of aromatic region of the ^{13}C NMR spectrum **O2** :middle of aromatic region of ^{1}H NMR spectrum
NS :160 **RG** :receiver gain for correct ADC input

Processing parameters
SI :8 K **BC_mod** :quad
WDW :EM **LB** :2 Hz
FT :Fourier transformation **phase correction :**adjust the phase to pure absorption.
integration :manual **baseline correction** :ABS
plot :use XWINPLOT

Experiment 8.17
- NMR Using Liquid-Crystal Solvents

pulse program: zg30
compare with Experiment 3.1

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D1 :1 sec – relaxation delay
D11 :30 msec - delay for disk I/O
TD :64 K
SW :22 ppm
O1 :on resonance of the 1H benzene signal in isotropic phase
DE :100 usec, to avoid break through of the matrix signal
NS :8
RG :receiver gain for correct ADC input

set the temperature to 330 K and measure the liquid crystal alone, go back to 300 K and measure again. Add the benzene and measure at 300 K.

Processing parameters

SI :32 K
BC_mod :quad
WDW :EM
LB :0.3 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Chapter 9
- Heteronuclear NMR Specroscopy

Summary

Experiment	Pulse program	Description
9.1	dept	^{1}H-Decoupled ^{15}N NMR Spectra with DEPT
9.2	deptnd	^{1}H-Coupled ^{15}N NMR Spectra with DEPT
9.3	zg30	^{19}F NMR Spectroscopy
9.4	dept	^{29}Si NMR Spectroscopy with DEPT
9.5	exp9_5.mo	^{29}Si NMR Spectroscopy with Spin-Lock Polarization
9.6	zgdc30	^{119}Sn NMR Spectroscopy
9.7	zgdc	^{2}H NMR Spectroscopy
9.8	zgdc	^{11}B NMR Spectroscopy
9.9	aring2.mo	^{17}O NMR Spectroscopy with RIDE
9.10	zg aring	$^{47/49}$Ti NMR Spectroscopy with ARING

Experiment 9.1
- ^{1}H-Decoupled ^{15}N NMR Spectra with DEPT

pulse program: dept
compare with Experiment 6.9

Setting of the needed channels: F1: ^{15}N
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{15}N transmitter pulse

PL2 :f2 channel - high power level for ^{1}H decoupler pulse

PL12 :f2 channel - power level for CPD decoupling

CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2

D1 :2 sec – relaxation delay

D2 :1/[2J(N,H)]= 5.6 msec, calculated from ^{1}J(N,H)=90 Hz

TD :32 K

O1 :220 ppm upfield from CH_3NO_2 (middle of NH region)

NS :4

P1 :f1 channel - 90° ^{15}N transmitter pulse

P2 :f1 channel - 180° ^{15}N transmitter pulse

P0 :f2 channel - 45° ^{1}H decoupler pulse (optimum for NH_2)

P3 :f2 channel - 90° ^{1}H decoupler pulse

P4 :f2 channel - 180° ^{1}H decoupler pulse

PCPD2 :f2 channel - 90° pulse for decoupling sequence

D12 :20 usec - delay for power switching

SW :350 ppm (chemical shift range of NH-groups)

O2 :middle of ^{1}H NMR spectrum

DS :8

RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :2 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 9.2

- ^{1}H-Coupled ^{15}N NMR Spectra with DEPT

pulse program: deptnd
DEPT experiment without decoupling.

Setting of the needed channels: F1: ^{15}N
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{15}N transmitter pulse
PL2 :f2 channel - high power level for ^{1}H decoupler pulse
D1 :2 sec – relaxation delay
TD :32 K
O1 :220 ppm upfield from CH_3NO_2
NS :32
RG :receiver gain for correct ADC input
P1 :f1 channel - 90° ^{15}N transmitter pulse
P2 :f1 channel - 180° ^{15}N transmitter pulse
P0 :f1 channel - 45° ^{1}H decoupler pulse
P3 :f2 channel - 90° ^{1}H decoupler pulse
P4 :f2 channel - 180° ^{1}H decoupler pulse
D2 :1/[2J(N,H)]= 5.6 msec, calculated from ^{1}J(N,H)=90 Hz
SW :350 ppm
O2 :middle of ^{1}H NMR spectrum
DS :8

Processing parameters

SI :16 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :1 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 9.3

- ^{19}F NMR Spectroscopy

pulse program: zg30
compare with Experiment 3.1

Setting of the needed channels: F1: ^{19}F
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{19}F
P1 :f1 channel - 90° ^{19}F transmitter pulse

transmitter pulse
D1 :1 sec – relaxation delay
TD :64 K
SW :300 ppm (typical range for fluorine bound to carbon)
O1 :about 100 ppm upfield from CCl_3F (center of that range)
NS :1
RG :receiver gain for correct ADC input

Processing parameters
SI :32 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :0.1 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 9.4
- ^{29}Si NMR Spectroscopy with DEPT

pulse program: dept
compare with Experiment 6.9

Setting of the needed channels: F1: ^{29}Si
F2: ^{1}H

Acquisition parameters
PL1 :f1 channel - high power level for ^{29}Si transmitter pulse
P1 :f1 channel - 90° ^{29}Si transmitter pulse
P2 :f1 channel - 180° ^{29}Si transmitter pulse
PL2 :f2 channel - high power level for ^{1}H decoupler pulse
P0 :f2 channel - 16.8° ^{1}H decoupler pulse corresponding to the 12 equivalent protons of the sample
P3 :f2 channel - 90° ^{1}H decoupler pulse
P4 :f2 channel - 180° ^{1}H decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :1 sec – relaxation delay
D2 :1/[2J(Si,H)]= 0.07 sec, calculated from ^{2}J(Si,H)=7 Hz
TD :64 K
SW :250 ppm
O1 :70 ppm upfield from ^{29}Si signal of TMS
O2 :middle of ^{1}H NMR spectrum
NS :32
DS :64
RG :receiver gain for correct ADC input

Processing parameters
SI :32 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :3 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 9.5

- ^{29}Si NMR Spectroscopy with Spin-Lock Polarization

pulse program: exp9_5.mo
With the spin-lock technique a superior polarization can be achieved, which is here demonstated for the liquid state. This type of polarization transfer in liquids works well for nuclei with no directly attached hydrogen atom.

Setting of the needed channels: F1: ^{29}Si
F2: ^{1}H

Acquisition parameters

PL2 :f2 channel - high power level for ^{1}H decoupler pulse, here 3 dB was used
PL10 :f1 channel - low power level
PL15 :f2 channel - low power level
PL12 :f2 channel - power level for CPD decoupling
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :4 sec – relaxation delay
D12 :20 usec – delay for power switching
TD :4 K
O1 :on resonance of ^{29}Si signal of TMS
L4 :32, the whole time should be 152 msec corresponding to ^{2}J(Si,H)= 7 Hz
NS :1
P3 :f2 channel - 90° ^{1}H decoupler pulse
P6 :f1 channel - 90° ^{29}Si transmitter pulse, 50 usec was used
P9 :f2 channel - 90° ^{1}H decoupler pulse, 50 usec was used
PCPD2 :f2 channel - 90° pulse for decoupling sequence, here 100 usec was used
D9 :152 msec – delay for spin-lock
D11 :30 msec – delay for disk I/O
SW :500 Hz
O2 :on resonance of ^{1}H NMR signal of TMS
RG :receiver gain for correct ADC input

Processing parameters

SI :2 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :1 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 9.6

- ^{119}Sn NMR Spectroscopy

pulse program: zgdc30
compare with Experiment 3.2

Setting of the needed channels: F1: ^{119}Sn
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{119}Sn transmitter pulse
PL12 :f2 channel – power level for CPD decoupling
P1 :f1 channel - 90° ^{119}Sn transmitter pulse
PCPD2 :f2 channel - 90° pulse for decoupling sequence

CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :1 sec – relaxation delay
D11 :30 msec - delay for disk I/O
TD :32 K
SW :600 ppm (Sn chemical shift range typical for $R_{4-n}SnX_n$)
O1 :100 ppm upfield from $Sn(CH_3)_4$ (center of that chemical shift range)
O2 :middle of ^1H NMR spectrum
NS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :3 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 9.7

- ^2H NMR Spectroscopy

pulse program: zgdc
compare with Experiment 3.10

Setting of the needed channels: F1: ^2H
F2: ^1H

Acquisition parameters

PL1 :f1 channel - high power level for ^2H transmitter pulse
P1 :f1 channel - 90° ^2H transmitter pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :100 msec – relaxation delay
D11 :30 msec - delay for disk I/O
TD :8 K
SW :8 ppm
O1 :middle of the ^2H NMR spectrum
O2 :middle of ^1H NMR spectrum
NS :256
RG :receiver gain for correct ADC input

Processing parameters

SI :4 K
BC_mod :quad
WDW :EM
LB :2 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
integration :is done with ABS or can be done manual
plot :use XWINPLOT

Experiment 9.8

- ^{11}B NMR Spectroscopy

pulse program: zgdc
compare with Experiment 3.10

Setting of the needed channels: F1: ^{11}B
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{11}B transmitter pulse
P1 :f1 channel - 90° ^{11}B transmitter pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :100 msec – relaxation delay
D11 :30 msec - delay for disk I/O
D2 :1/[2J(C,H)]= 2.36 msec, calculated from $^{1}J(C,H)$=212 Hz
D12 :20 usec - delay for power switching
TD :4 K
SW :36 ppm
O1 :middle of the ^{11}B NMR spectrum
O2 :middle of ^{1}H NMR spectrum
NS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :2 K
BC_mod :quad
WDW :EM
LB :2 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
referencing :use the external reference of the standard; be sure not to change the magnetic field between the two measurements
baseline correction :ABS
plot :use XWINPLOT

Experiment 9.9

- ^{17}O NMR Spectroscopy with RIDE

pulse program: aring2.mo
The RIDE (Ring Down Elimination) pulse sequence, is used to eliminate probe-head ringing, which occurs for quadrupolar nuclei with a relatively low y-value like 170.

Setting of the needed channels: F1: ^{17}O
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{17}O transmitter pulse
P1 :f1 channel - 90° ^{17}O transmitter pulse
P2 :f1 channel - 180° ^{17}O transmitter pulse
D1 :10 msec – relaxation delay
D13 :3 usec - short delay
TD :4 K
SW :500 ppm
O1 :200 ppm downfield from ^{17}O water signal
O2 :middle of ^{1}H ^{13}C NMR spectrum
NS :4*128
DE :15 usec
RG :receiver gain for correct ADC input

Processing parameters

SI :2 K
BC_mod :quad

WDW :EM
FT :Fourier transformation
baseline correction :ABS
LB :200 Hz
phase correction :adjust the phase to pure absorption.
plot :use XWINPLOT

Experiment 9.10

- $^{47/49}$Ti NMR Spectroscopy with ARING

a) pulse program: zg
compare with Experiment 2.8

Setting of the needed channels: F1: $^{47/49}$Ti
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for $^{47/49}$Ti transmitter pulse
P1 :f1 channel - 90° $^{47/49}$Ti transmitter pulse
D1 :10 msec – relaxation delay
DE :10 usec
TD :8 K
SW :600 ppm
O1 :middle of the titanium NMR spectrum
NS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :2 K
BC_mod :quad
WDW :EM
LB :15 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

b) pulse program: aring
A 1D sequence to suppress probe-head ringing.

Setting of the needed channels: F1: $^{47/49}$Ti
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{17}O transmitter pulse
P1 :f1 channel - 90° ^{17}O transmitter pulse
P2 :f1 channel - 180° ^{17}O transmitter pulse
D1 :10 msec – relaxation delay
D13 :3 usec - short delay
TD :8 K
SW :600 ppm
O1 : middle of the titanium NMR spectrum
DE :10 usec
NS :8
RG :receiver gain for correct ADC input

Processing parameters

SI :2 K
BC_mod :quad
WDW :EM
LB :15 Hz
FT :Fourier transformation
phase correction :adjust the phase to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Chapter 10
- The Second Dimension

Summary

Experiment	Pulse program	Description
10.1	jres	2D J-Resolved ^{1}H NMR Spectroscopy
10.2	hjres	2D J-Resolved ^{13}C NMR Spectroscopy
10.3	cosy90	The Basic H,H-COSY-Experiment
10.4	cosylr	Long-Range COSY
10.5	cosytp	Phase-Sensitive COSY
10.6	cosytp	Phase-Sensitive COSY-45
10.7	ecos3ntp	E.COSY
10.8	cosydfprtp	Double Quantum Filtered COSY with Presaturation
10.9	hxcondtp.mo	Fully Coupled C,H Correlation (FUCOUP)
10.10	hxco	C,H Correlation by Polarization Transfer (HETCOR)
10.11	hxco	Long-Range C,H Correlation by Polarization Transfer
10.12	coloc	C,H Correlation via Long-Range Couplings (COLOC)
10.13	inv4nd	The Basic HMQC Experiment
10.14	invbtp	Phase-Sensitive HMQC with BIRD Filter and GARP Decoupling
10.15	exp10_15.mo	Poor Man's Gradient HMQC
10.16	invblrndtp.mo	Phase-Sensitive HMBC with BIRD Filter
10.17	invindtp.mo	The Basic HSQC Experiment
10.18	mlevtp	The HOHAHA or TOCSY Experiment
10.19	noesytp	The NOESY Experiment
10.20	roesytp.2	The CAMELSPIN or ROESY Experiment
10.21	hoesy	The HOESY Experiment
10.22	inad	2D-INADEQUATE
10.23	noesytp	The EXSY Experiment
10.24	coxyf3.mo/ inv4xyf3.mo	X, Y Correlation

Experiment 10.1
- 2D J-Resolved ^{1}H NMR Spectroscopy

pulse program: jres:
In the 2D J-resolved experiment chemical shift and spin-spin coupling informations are separated and displayed on different axes of the 2D matrix.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
Parmod :2D
ND0 :2
TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :8 ppm
SW1 :40 Hz
O1 :middle of ^{1}H NMR spectrum
NS :4
DS :16
DE :as small as possible
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :256 W
WDW(F2) :SINE
WDW(F1) :SINE
SSB(F2) :0
SSB(F1) :0
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
TILT
SYMJ
phase correction :not necessary
plot :use XWINPLOT

Experiment 10.2

- 2D J-Resolved ^{13}C NMR Spectroscopy

pulse program: hjres
In the 2D J-resolved experiment chemical shift and spin-spin coupling informations of a ^{1}H coupled ^{13}C NMR spectrum are separated and displayed on different axes of the 2D matrix.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :WALTZ16- CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D1 :2 sec – relaxation delay
D12 :20 usec - delay for power switching
Parmod :2D
ND0 :2
TD2 :1 K data points in F2
TD1 :64 data points in F1
SW2 :175 ppm
SW1 :250 Hz
O1 : middle of ^{13}C NMR spectrum
O2 :middle of ^{1}H NMR spectrum
NS :32
DS :16
DE :as short as possible
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :QSINE
SSB(F2) :2
PH-mod(F2) :no
MC2 :QF

SYMJ
phase correction :not necessary

SI(F1) :256 W
WDW(F1) :QSINE
SSB(F1) :2
PH-mod(F1) :mc
XFB :fourier transformation in both directions

plot :use XWINPLOT

Experiment 10.3

- The Basic H,H-COSY-Experiment

pulse program: cosy90

The COSY (Correlation SpectroscopY) pulse sequence generates a 2D NMR spectrum in which the signals of a normal ^{1}H NMR spectrum are correlated with each other. Cross-peaks appear if the spin coupling is present.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
D0 :3 usec - incremented delay
Parmod :2D
TD2 :1 K data points in F2
SW2 :8 ppm
O1 : middle of ^{1}H NMR spectrum
NS :4
IN0 :1/[1*SW1]

P1 :f1 channel - 90° ^{1}H transmitter pulse
D1 :2 sec - relaxation delay
ND0 :1
TD1 :128 data points in F1
SW1 :8 ppm
DS :16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :SINE
SSB(F2) :0
PH-mod(F2) :no
MC2 :QF

SYM :may be performed
plot :use XWINPLOT

SI(F1) :512 W
WDW(F1) :SINE
SSB(F1) :0
PH-mod(F1) :mc
XFB :fourier transformation in both directions
phase correction :not necessary

Experiment 10.4

- Long-Range COSY

pulse program: cosylr

With the long-range variant of the standard COSY pulse sequence it is possible to observe cross-signals between protons which are connected by a very small coupling constant.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D0 :3 usec - incremented delay
D1 :2 sec – relaxation delay
D6 :200 msec
Parmod :2D
ND0 :1
TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :8 ppm
SW1 :8 ppm
O1 : middle of ^{1}H NMR spectrum
NS :4
DS :16
IN0 :1/[SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :512 W
WDW(F2) :SINE
WDW(F1) :SINE
SSB(F2) :0
SSB(F1) :0
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
SYM :may be performed
phase correction : not necessary
plot :use XWINPLOT

Experiment 10.5

- Phase-Sensitive COSY

pulse program: cosytp
Additional to the COSY90 the information of the spin coupling constants can be taken from the phase-sensitive COSY.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P0 :f1 channel - 90° ^{1}H transmitter pulse
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
Parmod :2D
ND0 :2
TD2 :2 K data points in F2
TD1 :256 data points in F1
SW2 :1.5 ppm
SW1 :1.5 ppm
O1 : middle of ^{1}H NMR spectrum
NS :4
DS :16
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :1 K
SI(F1) :1 K
WDW(F2) :GM
WDW(F1) :GM
LB(F2) : depending on the
LB(F1) : depending on the
GB(F2) : resolution
GB(F1) : resolution
PH-mod(F2) :pk
PH-mod(F1) :pk
PHC0(F2) :should be 0 before first transformation
PHC0(F1) :should be 0 before first transformation
PHC1(F2) :should be 0 before first transformation
PHC1(F1) :should be 0 before first transformation

MC2 :TPPI

XFB :fourier transformation in both directions

phase correction : use the 2D-phase correction routine, adjust strong diagonal peaks at the left and right of the spectrum in dispersion

XF2P :will be executed after correction of the rows

XF1P :will be executed after correction of the columns

plot :use XWINPLOT

Experiment 10.6

- Phase-Sensitive COSY-45

pulse program: cosytp

Additional to the COSY90 the information of the spin coupling constants can be taken from the phase-sensitive COSY. The difference to Experiment 10.5 is a smaller angle for the second pulse. The intensities of the autocorrelation signals, which are the cross-signals within the multiplets, become smaller; the diagonal will be narrower and cross signals near the diagonal can be observed more easily.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

P1 :f1 channel - 90° ^{1}H transmitter pulse

P0 :f1 channel - 45° ^{1}H transmitter pulse

D0 :3 usec - incremented delay

D1 :2 sec - relaxation delay

Parmod :2D

ND0 :2

TD2 :2 K data points in F2

TD1 :256 data points in F1

SW2 :1.5 ppm

SW1 :1.5 ppm

O1 : middle of ^{1}H NMR spectrum

NS :4

DS :16

IN0 :1/[2*SW1]

RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :1 K

SI(F1) :1 K

WDW(F2) :GM

WDW(F1) :GM

LB(F2) : depending on the

LB(F1) : depending on the

GB(F2) : resolution

GB(F1) : resolution

PH-mod(F2) :pk

PH-mod(F1) :pk

PHC0(F2) :should be 0 before first transformation

PHC0(F1) :should be 0 before first transformation

PHC1(F2) :should be 0 before first transformation

PHC1(F1) :should be 0 before first transformation

MC2 :TPPI

XFB :fourier transformation in both directions

phase correction :use the 2D-phase correction routine, adjust strong diagonal peaks at the left and right of the spectrum in dispersion

XF2P :will be executed after correction of the rows

XF1P :will be executed after correction of the columns

plot :use XWINPLOT

Experiment 10.7
- E.COSY

pulse program: ecos3ntp
The extraction of correct spin coupling constants may be hindered due to mutual cancellation of nearby positive and negative signals. E.COSY (Exclusive Correlation SpectroscopY) provides a solution of this problem, since cross-peak patterns are simplified, displaying only signals of transitions which are directly connected in the energy level diagram, so that signals of the passive spin in a coupling network disappear.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D11 :30 msec - delay for disk I/O
D13 :3 usec - short delay
Parmod :2D
ND0 :2
TD2 :2 K data points in F2
TD1 :256 data points in F1
SW2 :1.5 ppm
SW1 :1.5 ppm
O1 : middle of ^{1}H NMR spectrum
NS :12
DS :16
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :1 K
SI(F1) :1 K
WDW(F2) :EM
WDW(F1) :EM
LB(F2) : depending on the
LB(F1) : depending on the
PH-mod(F2) :pk
PH-mod(F1) :pk
PHC0(F2) :should be 0 before first transformation
PHC0(F1) :should be 0 before first transformation
PHC1(F2) :should be 0 before first transformation
PHC1(F1) :should be 0 before first transformation
MC2 :TPPI
XFB :fourier transformation in both directions
phase correction :use the 2D-phase correction routine, adjust strong diagonal peaks at the left and right of the spectrum in dispersion
XF2P :will be executed after correction of the rows
XF1P :phase correction in F1 is usually not necessary
plot :use XWINPLOT

Experiment 10.8
- Double Quantum Filtered COSY with Presaturation

pulse program: cosydfprtp
This COSY pulse sequence includes a water suppression technique: the presaturation and, the COSY variant with the double quantum filter.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
PL9 :f1 channel - power level for presaturation, typically in the range of 65 dB
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D11 : 30 msec – delay for disk I/O
D12 :20 usec - delay for power switching
D13 :3 usec – short delay
Parmod :2D
ND0 :2
TD2 :2 K data points in F2
TD1 :256 data points in F1
SW2 :10 ppm
SW1 :10 ppm
O1 :on resonance of water signal
NS :64
DS :16
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :1 K
SI(F1) :1 K
WDW(F2) :GM
WDW(F1) :GM
LB(F2) : depending on the
GB(F2) : resolution
LB(F1) : depending on the
GB(F1) : resolution
PH-mod(F2) :pk
PH-mod(F1) :pk
PHC0(F2) :should be 0 before first transformation
PHC0(F1) :should be 0 before first transformation
PHC1(F2) :should be 0 before first transformation
PHC1(F1) :should be 0 before first transformation
MC2 :TPPI
XFB :fourier transformation in both directions
phase correction :use the 2D-phase correction routine, adjust strong diagonal peaks at the left and right of the spectrum in dispersion
XF2P :will be executed after correction of the rows
XF1P :will be executed after correction of the columns
plot :use XWINPLOT

Experiment 10.9

- Fully Coupled C,H Correlation (FUCOUP)

pulse program: hxcondtp.mo
This sequence describes the simplest C,H correlation method, consisting only of three r.f. pulses. It leads to a 2D spectrum where the C,H spin coupling remains to be seen in both dimensions; therefore it has been called FUCOUP (Fully COUPled).

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
PL2 :f2 channel - high power level for ^{1}H decoupler pulse
P3 :f2 channel - 90° ^{1}H decoupler pulse
D0 :3 usec - incremented delay
D1 :10 sec - relaxation delay
D11 : 30 msec – delay for disk I/O
D12 :20 usec - delay for power switching
Parmod :2D
ND0 :2
TD2 :512 data points in F2
TD1 :64 data points in F1

SW2 :500 Hz
O1 :on resonance of ^{13}C NMR signal of $CHCl_3$
NS :2
IN0 :1/[2*SW1]

SW1 :500 Hz
O2 :on resonance of ^{1}H NMR signal of $CHCl_3$
DS :16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :E_M
LB(F2) : depending on the resolution
PH-mod(F2) :pk
PHC0(F2) :should be 0 before first transformation
PHC1(F2) :should be 0 before first transformation
MC2 :TPPI

SI(F1) :128 W
WDW(F1) :EM
LB(F1) : depending on the resolution
PH-mod(F1) :pk
PHC0(F1) :90
PHC1(F2) :should be 0 before first transformation
XFB :fourier transformation in both directions

phase correction :use the 2D-phase correction routine, adjust the phase in F2 to give antiphase signals and 90° phase correction in F1

XF2P :will be executed after correction of the rows
plot :use XWINPLOT

Experiment 10.10

- C,H Correlation by Polarization Transfer (HETCOR**)**

pulse program: hxco

This 2D-method leads to a C,H correlation by polarization transfer. Cross signals for all protons and ^{13}C nuclei which are connected by a ^{13}C, ^{1}H coupling over one bond are detected.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL2 :f2 channel - high power level for ^{1}H decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
CPD2 :WALTZ16 - decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D2 :1/[2J(C,H)]= 3.45 msec, calculated from $^{1}J(C,H)$=145 Hz
D11 : 30 msec – delay for disk I/O
Parmod :2D
TD2 :1 K data points in F2
SW2 :175 ppm
O1 : middle of ^{13}C NMR spectrum
NS :32
IN0 :1/[2*SW1]

P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse
P3 :f2 channel - 90° ^{1}H decoupler pulse
PCPD2 :f2 channel - 90° pulse for decoupling sequence
D1 :2 sec - relaxation delay
D3:1/[3JC,H)]= 2.29ec, calculated from $^{1}J(C,H)$=145 Hz
D12 :20 usec - delay for power switching
ND0 :2
TD1 :128 data points in F1
SW1 :8 ppm
O2 :middle of ^{1}H NMR spectrum
DS :16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :QSINE
SSB(F2) :2
PH-mod(F2) :no
MC2 :QF

phase correction :not necessary

SI(F1) :256 W
WDW(F1) :QSINE
SSB(F1) :2
PH-mod(F1) :mc
XFB :fourier transformation in both directions
plot :use XWINPLOT

Experiment 10.11

- Long-Range C,H Correlation by Polarization Transfer

pulse program: hxco

In this case it is possible to observe cross-signals for C,H spin pairs connected by two- or three-bond couplings $^{2}J(C,H)$ or $^{3}J(C,H)$. This can be achieved with the same pulse sequence as used in the Experiment 10.8 by adjusting the appropriate delays.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
PL2 :f2 channel - high power level for ^{1}H decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D2 :1/[2J(C,H)]= 50 msec, calculated from $^{n}J(C,H)$=10 Hz
D11 : 30 msec – delay for disk I/O
Parmod :2D
TD2 :1 K data points in F2
SW2 :175 ppm
O1 : middle of ^{13}C NMR spectrum
NS :64
IN0 :1/[2*SW1]

P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse
P3 :f2 channel - 90° ^{1}H decoupler pulse
PCPD2 :f2 channel - 90° pulse for decoupling sequence
D1 :2 sec - relaxation delay
D3 :1/[3J(C,H)]= 33 msec, calculated from $^{n}J(C,H)$=10 Hz
D12 :20 usec - delay for power switching
ND0 :2
TD1 :128 data points in F1
SW1 :8 ppm
O2 :middle of ^{1}H NMR spectrum
DS :16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :QSINE
SSB(F2) :2
PH-mod(F2) :no
MC2 :QF

phase correction :not necessary

SI(F1) :256 W
WDW(F1) :QSINE
SSB(F1) :2
PH-mod(F1) :mc
XFB :fourier transformation in both directions
plot :use XWINPLOT

Experiment 10.12
- C,H Correlation via Long-Range Couplings (COLOC)

pulse program: coloc
The COLOC (Correlation spectroscopy via Long range Couplings) is a 2D-method, to get cross-signals for protons and ^{13}C nuclei connected by two- or three-bond couplings.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel – high power level for ^{13}C transmitter pulse
PL2 :f2 channel – high power level for ^{1}H decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D6 :25 msec – note that D6 must be larger than TD1 times 1/[2*SW1]
D12 :20 usec – delay for power switching

P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse
P3 :f2 channel - 90° ^{1}H decoupler pulse
P4 :f2 channel - 180° ^{1}H decoupler pulse
PCPD2 :f2 channel - 90° pulse for decoupling sequence
D1 :2 sec - relaxation delay
D11 : 30 msec - delay for disk I/O
D18 :1/[3J(C,H)]= 33 msec, calculated from $^{n}J(C,H)$=10 Hz

Parmod :2D
TD2 :1 K data points in F2
SW2 :175 ppm
O1 : middle of ^{13}C NMR spectrum
NS :128
IN0 :1/[2*SW1]

ND0 :2
TD1 :64 data points in F1
SW1 :8 ppm
O2 :middle of ^{1}H NMR spectrum
DS :16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :QSINE
SSB(F2) :2
PH-mod(F2) :no
MC2 :QF

phase correction :not necessary

SI(F1) :256 W
WDW(F1) :QSINE
SSB(F1) :2
PH-mod(F1) :mc
XFB :fourier transformation in both directions
plot :use XWINPLOT

Experiment 10.13
- The Basic HMQC Experiment

pulse program: inv4nd
This is the basic HMQC (Heteronuclear Multiple Quantum Coherence) method. It is the simplest form of an inverse H,X correlation technique. The suppression of the unwanted signals is performed only by the phase cycle. This experiment is without decoupling.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
D0 :3 usec - incremented delay
D1 :2 sec – relaxation delay
D2 :1/[2J(C,H)]= 3.5 msec, calculated from ^{1}J(C,H)=145 Hz
Parmod :2D
ND0 :2
TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :8 ppm
SW1 :175 ppm
O1 : middle of ^{1}H NMR spectrum
O2 : middle of ^{13}C NMR spectrum
NS :8
DS :16
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :256 W
WDW(F2) :QSINE
WDW(F1) :QSINE
SSB(F2) :2
SSB(F1) :2
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
phase correction :not necessary
plot :use XWINPLOT

Experiment 10.14

- Phase-Sensitive HMQC with BIRD Filter and GARP Decoupling

pulse program: invbtp
This experiment gives an inverse H,C correlation. The suppression of the H-^{12}C signals is performed with a BIRD sandwich and the decoupling is done with GARP (Globally optimized Alternating-phase Rectangular Pulses).

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
P4 :f2 channel - 180° ^{13}C decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :GARP- CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D1 :1 sec - relaxation delay
D2 :1/[2J(C,H)]= 3.5 msec, calculated from ^{1}J(C,H)=145 Hz
D7 :ca. 1 sec - BIRD delay to be optimized for minimum FID; observe in the set-up mode the incoming FID and adjust D7 for minimum intensity
Parmod :2D
ND0 :4
TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :8 ppm
SW1 :175 ppm

O1 :middle of ^{1}H NMR spectrum
O2 :middle of ^{13}C NMR spectrum
NS :8
DS :16
IN0 :1/[4*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :256 W
WDW(F2) :GM
WDW(F1) :GM
LB(F2) : depending on the
GB(F2) : resolution
LB(F1) : depending on the
GB(F1) : resolution
PH-mod(F2) :pk
PH-mod(F1) :pk
PHC0(F2) :should be 0 before first transformation
PHC0(F1) :can be set by au-program calcphinv
PHC1(F2) :should be 0 before first transformation
PHC1(F1) :can be set by au-program calcphinv
MC2 :TPPI
XFB :fourier transformation in both directions
phase correction :use the 2D-phase correction routine, correct the signals positive
au-program :calcphinv (to calculate phase for F1)
XF2P :will be executed after correction of the rows
plot :use XWINPLOT

Experiment 10.15

- Poor Man's Gradient HMQC

pulse program: exp10_15.mo
A spin-lock pulse in connection with the BIRD sequence, reduces unwanted signals nearly to the level known from pulsed field gradients and allows the use of a higher receiver gain.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P28 :f1 channel - spin-lock pulse, **2 msec! Not more!**
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
P4 :f2 channel - 180° ^{13}C decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :GARP- CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D1 :1 sec - relaxation delay
D2 :1/[2J(C,H)]= 3.5 msec, calculated from ^{1}J(C,H)=145 Hz
D7 :ca. 1 sec - BIRD delay to be optimized for minimum FID; observe in the set-up mode the incoming FID and adjust D7 for minimum intensity
D4 :1/[4J(C,H)]= 1.75 msec, calculated from ^{1}J(C,H)=145 Hz
Parmod :2D
ND0 :4

TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :8 ppm
SW1 :175 ppm
O1 :middle of ^{1}H NMR spectrum
O2 :middle of ^{13}C NMR spectrum
NS :2
DS :16
IN0 :1/[4*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :256 W
WDW(F2) :GM
WDW(F1) :GM
LB(F2) : depending on the
GB(F2) : resolution
LB(F1) : depending on the
GB(F1) : resolution
PH-mod(F2) :pk
PH-mod(F1) :pk
PHC0(F2) :should be 0 before first transformation
PHC0(F1) :can be set by au-program calcphinv
PHC1(F2) :should be 0 before first transformation
PHC1(F1) :can be set by au-program calcphinv
MC2 :TPPI
XFB :fourier transformation in both directions
phase correction :use the 2D-phase correction routine, correct the signals positive
au-program :calcphinv (to calculate phase for F1)
XF2P :will be executed after correction of the rows
plot :use XWINPLOT

Experiment 10.16

- Phase-Sensitive HMBC with BIRD Filter

pulse program: invblrndtp.mo
To obtain long-range H,C correlations a special sequence called HMBC (Heteronuclear Multiple Bond Correlation) was developed. The purpose of this method is to suppress correlations via ^{1}J(C,H). This is a phase-sensitive version without decoupling.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
P4 :f2 channel - 180° ^{13}C decoupler pulse
D0 :3 usec - incremented delay
D1 :1 sec - relaxation delay
D2 :1/[2J(C,H)]= 3.5 msec, calculated from ^{1}J(C,H)=145 Hz
D6 :1/[2J(C,H)]= 50 msec, calculated from ^{n}J(C,H)=10 Hz
D7 :ca. 1 sec – BIRD delay to be optimized for minimum FID; observe in the set-up mode the incoming FID and adjust D7 for minimum intensity
D15 :46.5 msec - D6-D2
Parmod :2D
ND0 :4
TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :8 ppm
SW1 :175 ppm
O1 :middle of ^{1}H NMR spectrum
O2 :middle of ^{13}C NMR spectrum

NS :128
IN0 :1/[4*SW1]
DS :16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :GM
LB(F2) : depending on the
GB(F2) : resolution
PH-mod(F2) :pk
PHC0(F2) :should be 0 before first transformation
PHC1(F2) :should be 0 before first transformation
MC2 :TPPI

phase correction :use the 2D-phase correction routine, correct the signals positive
XF2P :will be executed after correction of the rows

SI(F1) :256 W
WDW(F1) :GM
LB(F1) : depending on the
GB(F1) : resolution
PH-mod(F1) :pk
PHC0(F1) :can be set by au-program calcphinv
PHC1(F1) :can be set by au-program calcphinv
XFB :fourier transformation in both directions
au-program :calcphinv (to calculate phase for F1)

plot :use XWINPLOT

Experiment 10.17

- The Basic HSQC Experiment

pulse program: invindtp.mo

The HSQC (Heteronuclear Single Quantum Coherence) method performs the H,C correlation via the ^{13}C chemical shift evolution of a single quantum coherence. In this sequence the signals are not broadened by homonuclear H,H couplings in F1. This experiment is without decoupling.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
D0 :3 usec - incremented delay
D4 :1/[4J(C,H)]= 1.72 msec, calculated from $^{1}J(C,H)$=145 Hz
Parmod :2D
TD2 :1 K data points in F2
SW2 :8 ppm
O1 :middle of ^{1}H NMR spectrum
NS :8
IN0 :1/[4*SW1]

P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
P4 :f2 channel - 180° ^{13}C decoupler pulse
D1 :2 sec - relaxation delay

ND0 :4
TD1 :128 data points in F1
SW1 :175 ppm
O2 :middle of ^{13}C NMR spectrum
DS :16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :EM

SI(F1) :256 W
WDW(F1) :EM

LB(F2) : depending on the resolution
PH-mod(F2) :pk
PHC0(F2) :should be 0 before first transformation
PHC1(F2) :should be 0 before first transformation
MC2 :TPPI

LB(F1) : depending on the resolution
PH-mod(F1) :pk
PHC0(F1) :can be set by au-program calcphinv
PHC1(F1) : can be set by au-program calcphinv
XFB :fourier transformation in both directions

phase correction :use the 2D-phase correction routine, correct the signals positive
XF2P :will be executed after correction of the rows

au-program :calcphinv (to calculate phase for F1)

plot :use XWINPLOT

Experiment 10.18

- The HOHAHA or TOCSY Experiment

pulse program: mlevtp
The TOCSY (Total Correlation SpectroscopY) method can give a total correlation of all protons of a chain with each other. This is the phase-sensitive variant.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL10 :f1 channel - power level for TOCSY-spinlock

P1 :f1 channel - 90° ^{1}H transmitter pulse
P5 :f1 channel - 60° ^{1}H transmitter low power pulse
P6 :f1 channel - 90° ^{1}H transmitter low power pulse
P7 :f1 channel - 180° ^{1}H transmitter low power pulse
P17 :f1 channel - 2.5 msec trim pulse

D0 :3 usec - incremented delay
D9 :200 msec – TOCSY mixing time
Parmod :2D
TD2 :1 K data points in F2
SW2 :10 ppm
O1 : middle of ^{1}H NMR spectrum
NS :4
IN0 :1/[2*SW1]

D1 :2 sec - relaxation delay
D12 :20 usec - delay for power switching
ND0 :2
TD1 :128 data points in F1
SW1 :10 ppm

DS :16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :EM
LB(F2) : depending on the resolution
PH-mod(F2) :pk
PHC0(F2) :should be 0 before first transformation
PHC1(F2) :should be 0 before first transformation
MC2 :TPPI

SI(F1) :512 W
WDW(F1) :EM
LB(F1) : depending on the resolution
PH-mod(F1) :pk
PHC0(F1) :should be 0 before first transformation
PHC1(F1) :should be 0 before first transformation
XFB :fourier transformation in both directions

phase correction :use the 2D-phase correction routine, correct the signals positive.

XF2P :will be executed after correction of the rows

XF1P :will be executed after correction of the columns

plot :use XWINPLOT

Experiment 10.19

- The NOESY Experiment

pulse program: noesytp

The NOESY (Nuclear Overhauser Enhancement SpectroscopY) experiment is the 2D equivalent of the NOE difference experiment and yields correlation signals which are caused by dipolar cross-relaxation between nuclei in a close spatial relationship.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

P1 :f1 channel - 90° ^{1}H transmitter pulse

D0 :3 usec - incremented delay

D1 :2 sec - relaxation delay

D8 :2 sec – mixing time

Parmod :2D

ND0 :2

TD2 :1 K data points in F2

TD1 :256 data points in F1

SW2 :10 ppm

SW1 :10 ppm

O1 :middle of ^{1}H NMR spectrum

NS :16

DS :16

IN0 :1/[2*SW1]

RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W

SI(F1) :512 W

WDW(F2) :EM

WDW(F1) :EM

LB(F2) : depending on the resolution

LB(F1) : depending on the resolution

PH-mod(F2) :pk

PH-mod(F1) :pk

PHC0(F2) :should be 0 before first transformation

PHC0(F1) :should be 0 before first transformation

PHC1(F2) :should be 0 before first transformation

PHC1(F1) :should be 0 before first transformation

MC2 :TPPI

XFB :fourier transformation in both directions

phase correction :use the 2D-phase correction routine, adjust the phase of the diagonal signals so that they are negative.

XF2P :will be executed after correction of the rows

XF1P :will be executed after correction of the columns

plot :use XWINPLOT

Experiment 10.20

- The CAMELSPIN or ROESY Experiment

pulse program: roesytp.2
This is a 2D version of the ROESY (Rotating frame Overhauser Enhancement SpectroscopY) experiment. It is an experiment to measure NOE, but under spin-lock conditions. It is used for molecules with a molar mass in the order of 1000 to 3000, because the cross-signals measured with the NOESY (Nuclear Overhauser Enhancement SpectroscopY) may disappear.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL11 :f1 channel - power level for ROESY-spinlock, 23 dB was used here
P1 :f1 channel - 90° ^{1}H transmitter pulse
P25 :f1 channel - 180° pulse at transmitter attenuation of spin-lock, here 180 usec
P15 :f1 channel – pulse for ROESY spinlock, here 300 msec
D0 :3 usec - incremented delay
D1 :2 sec – relaxation delay
D12 :20 usec - delay for power switching
L4 :832 for 300 msec spin-lock. The loop parameter must be an even number**.**
Parmod :2D
ND0 :2
TD2 :1 K data points in F2
TD1 :256 data points in F1
SW2 :10 ppm
SW1 :10 ppm
O1 : middle of ^{1}H NMR spectrum
NS :16
DS :16
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :512 W
WDW(F2) :EM
WDW(F1) :QSINE
LB(F2) :depending on the resolution
SSB(F1) :2
PH-mod(F2) :pk
PH-mod(F1) :pk
PHC0(F2) :should be 0 before first transformation
PHC0(F1) :should be 0 before first transformation
PHC1(F2) :should be 0 before first transformation
PHC1(F1) :should be 0 before first transformation
MC2 :TPPI
XFB :fourier transformation in both directions
phase correction : use the 2D-phase correction routine, adjust the phase of the diagonal signals negative, so that the ROESY correlation signals are positive.

XF2P :will be executed after correction of the rows
XF1P :will be executed after correction of the columns
plot :use XWINPLOT

Experiment 10.21
- The HOESY Experiment

pulse program: hoesy
The HOESY (Heteronuclear Overhauser Enhancement SpectroscopY) is a 2D experiment to measure the heteronuclear Overhauser effect.

Setting of the needed channels: F1: ^{6}Li
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{6}Li transmitter pulse
PL2 :f2 channel - high power level for ^{1}H decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D9 :1.7 sec – mixing time
Parmod :2D
TD2 :512 data points in F2
SW2 :4 ppm
O1 : middle of ^{6}Li NMR spectrum
NS :32
IN0 :1/[2*SW1]

P1 :f1 channel - 90° ^{6}Li transmitter pulse
P2 :f1 channel - 180° ^{6}Li transmitter pulse
P3 :f2 channel - 90° ^{1}H decoupler pulse
PCPD2 :f2 channel - 90° pulse for decoupling sequence
D1 :6 sec - relaxation delay
D12 :20 usec - delay for power switching
ND0 :2
TD1 :128 data points in F1
SW1 :9 ppm
O2 :middle of ^{1}H NMR spectrum
DS :16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :256 W
WDW(F2) :EM
LB(F2) :depending on the resolution
PH-mod(F2) :no
MC2 :QF
phase correction :not necessary

SI(F1) :256 W
WDW(F1) :EM
LB(F1) : depending on the resolution
PH-mod(F1) :mc
XFB :fourier transformation in both directions
plot :use XWINPLOT

Experiment 10.22
- 2D-INADEQUATE

pulse program: inad
The INADEQUTE (Incredible Natural Abundance DoublE QUAntum Transfer Experiment) is a 2D experiment. It observe ^{13}C,^{13}C couplings over two bonds and suppress the strong ^{12}C signals.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse

P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse

PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D1 :3 sec - relaxation delay
D4 :1/[4J(C,C)]= 7.6 msec, calculated from ^{1}J(C,C)=33 Hz
D11 : 30 msec - delay for disk I/O
Parmod :2D
ND0 :1
TD2 :1 K data points in F2
TD1 :128 data points in F1
O1 : middle of ^{13}C NMR spectrum
SW2 :60 ppm
SW1 :120 ppm (double quantum frequency)
NS :128
DS :16
IN0 :1/[2*SW2]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :256 W
WDW(F2) :SINE
WDW(F1) :SINE
SSB(F2) :2
SSB(F1) :2
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
phase correction :not necessary
plot :use XWINPLOT

Experiment 10.23

- The EXSY Experiment

pulse program: noesytp

The 2D EXSY (Exchange SpectroscopY) method can indicate chemical exchange before line-broadening occurs. the pulse sequence is exactly the same as that used for phase-sensitive NOESY.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D8 :1 sec – mixing time
Parmod :2D
ND0 :2
TD2 :512 data points in F2
TD1 :32 data points in F1
SW2 :0.7 ppm
SW1 :0.7 ppm
O1 : middle of methyl group region
NS :4
DS :16
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :256 W
SI(F1) :256 W
WDW(F2) :EM
WDW(F1) :EM
LB(F2) :depending on the resolution
LB(F1) : depending on the resolution
PH-mod(F2) :pk
PH-mod(F1) :pk
PHC0(F2) :should be 0 before first transformation
PHC0(F1) :should be 0 before first transformation

PHC1(F2) :should be 0 before first transformation
PHC1(F1) :should be 0 before first transformation
MC2 :TPPI
XFB :fourier transformation in both directions
phase correction : use the 2D-phase correction routine, phase correction is usually only necessary in F2, the cross-signals have the same phase as the diagonal signals
XF2P :will be executed after correction of the rows
XF1P :will be executed after correction of the columns
plot :use XWINPLOT

Experiment 10.24
- X, Y Correlation

a) pulse program: coxyf3.mo
This experiment correlates two hetero-atoms X and Y with each other under complete proton decoupling. For this experiment a tripleresonance probe-head and a three-channel spectrometer are required.

Setting of the needed channels:
F1: ^{m}Y (^{13}C)
F2: ^{1}H
F3: ^{n}X (^{31}P)

Acquisition parameters

PL1 :f1 channel - high power level for ^{13}C transmitter pulse
P1 :f1 channel - 90° ^{13}C transmitter pulse
P2 :f1 channel - 180° ^{13}C transmitter pulse
PL3 :f3 channel - high power level for ^{31}P decoupler pulse
P21 :f3 channel - 90° ^{31}P decoupler pulse
PL12 :f2 channel - power level for CPD/BB decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D11 :30 msec – delay for disk I/O
D22 :1/[2J(X,Y)]= 25 msec, calculated from ^{n}J(X,Y)=20 Hz
Parmod :2D
ND0 :2
TD2 :1 K data points in F2
TD1 :64 data points in F1
SW2 :12 ppm
SW1 :1 ppm
O1 :middle of ^{13}C NMR spectrum
O2 :middle of ^{1}H NMR spectrum
O3 :middle of ^{31}P NMR spectrum
NS :8
DS :16
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :256 W
WDW(F2) :SINE
WDW(F1) :SINE
SSB(F2) : 0
SSB(F1) : 0
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
phase correction :not necessary
plot :use XWINPLOT

b) pulse program: inv4xyf3.mo
compare with Experiment 10.24 a)

Setting of the needed channels:
F1: ^{n}X (^{31}P)
F2: ^{1}H
F3: ^{m}Y (^{13}C)

Acquisition parameters

PL1 :f1 channel - high power level for ^{31}P transmitter pulse
P1 :f1 channel - 90° ^{31}P transmitter pulse
P2 :f1 channel - 180° ^{31}P transmitter pulse
PL3 :f3 channel - high power level for ^{13}C decoupler pulse
P21 :f3 channel - 90° ^{13}C decoupler pulse
PL12 :f2 channel - power level for CPD/BB decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D11 :30 msec – delay for disk I/O
D22 :1/[2J(X,Y)]= 25 msec, calculated from $^{n}J(X,Y)$=20 Hz
Parmod :2D
ND0 :2
TD2 :256 data points in F2
TD1 :128 data points in F1
SW2 :1 ppm
SW1 :12 ppm
O1 :middle of ^{31}P NMR spectrum
O2 :middle of ^{1}H NMR spectrum
O3 :middle of ^{13}C NMR spectrum
NS :8
DS :16
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :256 W
WDW(F2) :SINE
WDW(F1) :SINE
SSB(F2) : 0
SSB(F1) : 0
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
phase correction :not necessary
plot :use XWINPLOT

Chapter 11
- NMR Spectroscopy with Pulsed Field Gradients

Summary

Experiment	Pulse program	Description
11.1	calibgp	Calibration of Pulsed Field Gradients
11.2	preempgp2.mo	Gradient Preemphasis
11.3	calibam.mo	Gradient Amplifier Test
11.4	zggp30.mo	Determination of Pulsed Field Gradient Ring-Down Delays
11.5	zggpse	The Pulsed Gradient Spin-Echo Experiment
11.6		Excitation Pattern of Selective Pulses
11.7	exp11_7.mo	The Gradient zz-Filter
11.8	selcogp.mo	gs-SELCOSY
11.9	selgpml.mo	gs-SELTOCSY
11.10	selnogp.3	DPFGSE-NOE
11.11	selincorgp.mo	gs-SELINCOR
11.12		GRECCO
11.13	p3919gp	WATERGATE
11.14	dpfgse.mo	Water Suppression by Excitation Sculpting

Experiment 11.1
- Calibration of Pulsed Field Gradients

pulse program: calibgp
Calibration of gradient strength

This experiment needs a special sample, preparation as described in the book.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P16 : 10 msec - homospoil/gradient pulse
D1 :1 sec - relaxation delay
D11 :delay for disk I/O
D12 :delay for power switching
D16 :100 usec - delay for homospoil/gradient recovery
D28 :equal to aq
TD :2 K
SW :100 KHz
O1 :on resonance of water signal
gpz 1 :1%
NS :2
RG :receiver gain for correct ADC input

Processing parameters

SI :1 K
BC_mod :quad
WDW :EM
LB :20 Hz
FT :Fourier transformation
phase correction :mc

Measure the width of the dip (Hz), and calculate the gradient strength G_z according to the Equation.

Experiment 11.2

- Gradient Preemphasis

pulse program: preempgp2.mo
In this experiment it is described how to adjust the preemphasis using a sample of chloroform.

Setting of the needed channels: F1: ^1H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^1H transmitter pulse
P0 :f1 channel - 10° ^1H transmitter power pulse
P16 : 1 msec - homospoil/gradient pulse
D1 :0.1 sec - relaxation delay
D16 :300 msec-50 usec - delay for homospoil/gradient recovery, will be varied
TD :4 K
SW :5000 Hz
O1 :1000 Hz off resonance from $CHCl_3$ signal
NS :1
GPNAM1 : rectangular.1
gpz 1 :75%
RG :receiver gain for correct ADC input

Processing parameters

No processing required

Experiment 11.3

- Gradient Amplifier Test

pulse program: calibam.mo
The simple test checks whether positive and negative gradient pulses have the same effect and thus detects any imbalance of the configuration.

Setting of the needed channels: F1: ^1H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^1H transmitter pulse
P1 :f1 channel - 90° ^1H transmitter power pulse
P16 : 1 msec - homospoil/gradient pulse
D1 :5 sec - relaxation delay
D16 :100 usec - delay for homospoil/gradient recovery
TD :4 K
SW :500 Hz
O1 :on resonance of $CHCl_3$ signal
NS :16
gpnam1 :SINE.100
gpz 1 :50%

gpnam2 :SINE.100
DS :4
gpz 2 :-50%
RG :receiver gain for correct ADC input

Processing parameters

SI :2 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
plot :use XWINPLOT
BC_mod :quad
LB :2 Hz
phase correction :adjust to pure absorption.
referencing :set the TMS signal to 0 ppm

Experiment 11.4

- Determination of Pulsed Field Gradient Ring-Down Delays

pulse program: zggp30.mo
The experiment described here demonstrates a calibration routine to define a suitable ringdown delay.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
D1 :5 sec - relaxation delay
D11 :30 msec - delay for disk I/O
TD :4 K
O1 :on resonance of $CHCl_3$ signal
gpnam1 :rectangular.1
RG :receiver gain for correct ADC input
P1 :f1 channel - 90° ^{1}H transmitter power pulse
P16 : 1msec - homospoil/gradient pulse
D16 :1sec - 1 usec - to be varied
SW :500 Hz
NS :1
gpz 1 :80%

Processing parameters

SI :2 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :2 Hz
phase correction : adjust the signals to pure absorption.
plot :use XWINPLOT

Experiment 11.5

- The Pulsed Gradient Spin-Echo Experiment

pulse program: zggpse
This spin-echo experiment can be used to determine the strength of field gradients, if the diffusion constant of the sample is accurately known by other means.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H
P1 :f1 channel - 90° ^{1}H transmitter pulse

transmitter pulse

P2 :f1 channel - 180° ^{1}H transmitter pulse
P16 : 4 msec - homospoil/gradient pulse, variable strength from 0 to 0.2 T/m in 10 steps

D1 :2 sec - relaxation delay
D16 :500 usec - delay for homospoil/gradient recovery

TD :1 K
SW :1000 Hz

O1 :on resonance of water signal

gpnam1 :SINE.100
gpz 1 :1%

gpnam2 :SINE.100
gpz 2 :1%

NS :2
RG :receiver gain for correct ADC input

Processing parameters

Process all 10 spectra identically.

SI :512 W
BC_mod :quad

WDW :EM
LB :5 Hz

FT :Fourier transformation
phase correction :adjust the phase to pure absorption.

baseline correction :ABS

integration :integrate the water signal in all spectra and refer all integrals to the integral value of the starting spectrum with gradient stength of 0. Compile a table of integral ratios I_g/I_0 vs. gradient strength G used, where the gradient stength is determined as described in Experiment 11.1.

Experiment 11.6

- Excitation Pattern of Selective Pulses

a) pulse program:

The width of the excitation of a selective pulse corresponds only very roughly to the inverse of ist duration. This method produces an image of the excitation pattern in one scan and provide determinations of the excitation pattern of a a) 90° and a b) 180° selective pulse.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

Processing parameters

b) pulse program:

compare with Exp. 11.6 a)

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

Processing parameters

Experiment 11.7
- The Gradient zz-Filter

pulse program: exp11_7.mo
In many exoeriments one wants to selectively observe protons that are attached to 13C or 15N. The strong signals of protons attached to 12C or 14N need to be suppressed in order to be able to adjust the receiver gain for the desired signals only. One technique to achieve this goal is to dephase unwanted signals with pulsed field gradients after storing the desired magnetization as z-magnetization.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

PL2 :f2 channel - high power level for ^{13}C decoupler pulse

D1 :10 sec - relaxation delay

D16 :100 usec - delay for homospoil/gradient recovery

TD :4 K

O1 :on ^{1}H resonance

NS :1

gpnam1 :SINE.100

RG :receiver gain for correct ADC input

P1 :f1 channel - 90° ^{1}H transmitter pulse

P2 :f1 channel - 180° ^{1}H transmitter pulse

P4 :f2 channel - 180° ^{13}C decoupler pulse

P16 : 1.5 msec - homospoil/gradient pulse

D4 :1/[4J(C,H)]= 1.17 msec, calculated from $^{1}J(C,H)$=214 Hz

SW :500 Hz

O2 :on ^{13}C resonance

DS :16

gpz 1 :5%

Processing parameters

SI :2 K

WDW :EM

FT :Fourier transformation

baseline correction :ABS

BC_mod :quad

LB :0.1 Hz

phase correction :adjust the phase of the satellites up and down

plot :use XWINPLOT

Experiment 11.8
- gs-SELCOSY

pulse program: selcogp.mo
This is the advanced 1D variant of the most common 2D experiment. Instead of recording the full 2D matrix, one can simply measure one ”row” by replacing the first 90° pulse of the COSY experiment with a soft pulse, thus looking only for spin couplings that affect the particular proton excited.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

P1 :f1 channel - 90° ^{1}H transmitter power pulse

P2 :f1 channel - 180° ^{1}H transmitter power pulse

SP1 :f1 channel - power level for shaped pulse, here 64 dB was used

P12 :f1 channel - 180° shaped pulse, 50 msec was used here

P16 : 2 msec - homospoil/gradient pulse

D1 :2 sec - relaxation delay

D16 :500 usec - delay for homospoil/gradient recovery

D2 : 30-60 msec, adjusted to ≈1/[2J(H,H)]

TD :32 K

SW :10 ppm

O1 :on resonance of selected signal or use spoffs

PHCOR2 :difference of phases between power level SP1 and PL1

gpnam1 :SINE.100

gpz 1 :40%

gpnam2 :SINE.100

gpz 2 :40%

gpnam3 :SINE.100

gpz 3 :7%

gpnam4 :SINE.100

gpz 4 :7%

gpnam5 :SINE.100

gpz 5 :20%

gpnam6 :SINE.100

gpz 6 :-20%

NS :1

DS :4

Gaussian shape with 1024 data points was used

RG :receiver gain for correct ADC input

Processing parameters

SI :16 K

BC_mod :quad

WDW :EM

LB :0.5 Hz

FT :Fourier transformation

phase correction :note that the signals of the coupling partners show the active coupling in antiphase

baseline correction :ABS

plot :use XWINPLOT

Experiment 11.9

- gs-SELTOCSY

pulse program: selgpml.mo

This is the 1D variant of the gs-TOCSY experiment. Compared with the selective TOCSY method, the gradient-selected method gives clean results without the need of phase cycling, using only one scan.

Setting of the needed channels: F1: ^{1}H

F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

P1 :f1 channel - 90° ^{1}H transmitter power pulse

P2 :f1 channel - 180° ^{1}H transmitter power pulse

PL10 :f1 channel - power level for TOCSY-spinlock

P5 :f1 channel - 60° low power pulse

P6 :f1 channel - 90° low power pulse

P7 :f1 channel - 180° low power pulse

SP1 :f1 channel - power level for shaped pulse, here 62 dB was used

P12 :f1 channel - 180° shaped pulse, 50 msec was used here

P16 : 1 msec - homospoil/gradient pulse
D1 :2 sec - relaxation delay
D16 :500 usec - delay for homospoil/gradient recovery
D9 :250 msec; 76 msec; 215 msec – mixing time (3 different experiments)
D20 :1msec - equal to the effective length of the gradient pulse
TD :32 K
SW :10 ppm
O1 : on resonance of selected signal or use spoffs
PHCOR2 :difference of phases between power level SP1 and PL1
gpnam1 :SINE.100
gpz 1 : 7%
gpnam2 :SINE.100
gpz 2 : -3%
gpnam3 :SINE.100
gpz 3 :-10%
NS :1
DS :2
Gaussian shape with 1024 data points was used
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :0.1 Hz
FT :Fourier transformation
phase correction :adjust the signals to pure absorption.
baseline correction :ABS
plot :use XWINPLOT

Experiment 11.10

- DPFGSE-NOE

pulse program: selnogp.3
Using pulsed field gradients, unwanted signals can be better suppressed and, with a selective excitation pulse tailored to the multiplet under consideration, the desired NOE effects can be recorded without interference from other signals.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL0 :120 dB
PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter power pulse
P2 :f1 channel - 180° ^{1}H transmitter power pulse
SP1 :f1 channel - power level for shaped pulse, here 62 dB was used
P12 :f1 channel - 180° shaped pulse, 50 msec was used here, Gaussian shape
P16 : 1 msec - homospoil/gradient pulse
D1 :2 sec - relaxation delay
D8 :0.7 sec - mixing time
D16 :500 usec - delay for homospoil/gradient recovery
D20 :d8*0.5-p16-d16
TD :32 K
SW :10 ppm
O1 :middle of ^{1}H NMR spectrum
NS :32
gpnam1 :SINE.100
gpz 1 : 11%
gpnam2 :SINE.100
gpz 2 : 17%
gpnam3 :SINE.100
gpz 3 : 40%
gpnam4 :SINE.100
gpz 4 :-40 %
DS :4
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
WDW :EM
FT :Fourier transformation
baseline correction :ABS
BC_mod :quad
LB :0.3 Hz
phase correction :adjust a negative phase for the irradiated multiplet
plot :use XWINPLOT

Experiment 11.11

- gs-SELINCOR

pulse program: selincorgp.mo
This experiment yields 1D proton spectra in which the desired proton signal is selected via a selective pulse on the directly bonded ^{13}C nucleus using the $^{1}J(C,H)$ spin coupling. The HSQC (Heteronuclear Single Quantum Coherence) method is used and the elimination of protons bond to ^{12}C is achieved by pulsed field gradients.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
PL21 :f1 channel - power level for spin-lock pulse
SP2 :f2 channel - power level for shaped pulse, here 66 dB was used
CPD2 :WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1 :2 sec - relaxation delay
D11 :30 msec - delay for disk I/O
DELTA :d4-p16-d16
TD :32 K
O1 :middle of ^{1}H spectrum
NS :128
gpnam1 :SINE.100
gpnam2 :SINE.100
gpnam3 :SINE.100
gpnam4 :SINE.100
gpnam5 :SINE.100
RG :receiver gain for correct ADC input
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
P4 :f2 channel - 180° ^{13}C decoupler pulse
PCPD2 :f2 channel - 90° pulse for decoupling sequence
P28 :f1 channel - spin-lock pulse, same length as p13 (40 msec)
P14 :f2 channel - 180° shaped pulse, 40 msec was used here
P16 : 1.5 msec - homospoil/gradient pulse
D4 :1/[4J(C,H)]= 1.8 msec, calculated from $^{1}J(C,H)$=140 Hz
D16 :100 usec - delay for homospoil/gradient recovery
DELTA2 :d4-p16-d16-4u
SW :10 ppm
O2 :on resonance of chosen ^{13}C NMR signal
DS :16
gpz 1 :5%
gpz 1 :5%
gpz 1 :-40%
gpz 1 :40%
gpz 1 :-20%

Processing parameters

SI :16 K
BC_mod :quad

WDW :EM
FT :Fourier transformation
baseline correction :ABS
LB :0.5 Hz
phase correction :adjust the signals to pure absorption.
plot :use XWINPLOT

Experiment 11.12
- GRECCO

pulse program:

The GRECCO (Gradient Enhanced Carbon Coupling) experiment selectively detects $^{2}J(C,C)$ and $^{3}J(C,C)$ carbon couplings, which are useful for a conformational analysis.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

Processing parameters

Experiment 11.13
- WATERGATE

pulse program: p3919gp
This is a pulsed field gradient method to suppress the water signal.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL18 :f1 channel - low power level for 3-9-19 pulse (watergate)
P1 :f1 channel - 90° ^{1}H transmitter power pulse
P0 :f1 channel -- 90° pulse at PL18
P16 : 2 msec - homospoil/gradient pulse
P28 : f1 channel - 90° pulse at PL18
D1 :1 sec - relaxation delay
D16 :500 usec - delay for homospoil/gradient recovery
D19 :150 usec - delay for binomial water suppression, D19=(1/(2*d)), d=distance of next null (in Hz), usually 2*DW
TD :32 K
SW :10 ppm
O1 :on water resonance
NS :16
gpnam1 :SINE.100
gpz 1 : 20%
gpnam2 :SINE.100
gpz 2 : 20%
DS :4
RG :receiver gain for correct ADC input

Processing parameters

SI :16 K
BC_mod :quad
WDW :EM
LB :0.5 Hz

FT :Fourier transformation

phase correction :ignore the phase of the water signal and adjust the others to pure absorption.

baseline correction :ABS

referencing :set the TMS signal to 0 ppm

plot :use XWINPLOT

Experiment 11.14

- Water Suppression by Excitation Sculpting

pulse program: dpfgse.mo

The WATERGATE technique has problems with baseline roll and signal phasing. A new technique, termed DPFGSE (Double Pulsed Field Gradient Spin Echo), also being called Excitation Sculpting solves this problem by applying the WATERGATE sequence twice.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

PL18 :f1 channel - low power level for 3-9-19 pulse (watergate)

D1 :1 sec - relaxation delay

D19 :250 usec

TD :32 K

O1 :on water resonance

gpnam1 :SINE.100

gpnam2 :SINE.100

gpnam3 :SINE.100

gpnam4 :SINE.100

DS :4

P1 :f1 channel - 90° ^{1}H transmitter power pulse

P0 :f1 channel -- 90° pulse at PL18

P16 : 1 msec - homospoil/gradient pulse

P28 : f1 channel - 90° pulse at PL18

D16 :500 usec - delay for homospoil/gradient recovery

SW :10 ppm

NS :16

gpz 1 : 40%

gpz 2 : 40%

gpz 3 : 7%

gpz 4 : 7%

RG :receiver gain for correct ADC input

Processing parameters

SI :16 K

WDW :EM

FT :Fourier transformation

baseline correction :ABS

BC_mod :quad

LB :0.5 Hz

phase correction :ignore the phase of the water signal and adjust the others to pure absorption.

plot :use XWINPLOT

Chapter 12
- 2D NMR Spectroscopy with Field Gradients

Summary

Experiment	Pulse program	Description
12.1	cosygp	gs-COSY
12.2	cosydfgptp.mo	Phase-Sensitive gs-DQF-COSY
12.3	inv4gp	gs-HMQC
12.4	inv4gplplrnd	gs-HMBC
12.5	inv4acgplplr.mo	ACCORD-HMBC
12.6	invietgpsi	Phase-Sensitive gs-HSQC with Sensitivity Enhancement
12.7	mlevgp.mo	gs-TOCSY
12.8	inv4gpml	gs-HMQC-TOCSY
12.9		2Q-HMBC
12.10	ineptinadgp.mo	Gradient-Selected ^{1}H-Detected 2D INEPT-INADEQUATE
12.11	noesygpst	gs-NOESY
12.12	invietgpno.mo	gs-HSQC-NOESY
12.13		gs-HOESY
12.14	inv4gpnd.mo	^{1}H, ^{15}N Correlation with gs-HMQC

Experiment 12.1
- gs-COSY

pulse program: cosygp
This COSY pulse sequence can be achieved with only one scan per T1 increment

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P0 :f1 channel - 90° ^{1}H transmitter pulse
P16 :2 msec - homospoil/gradient pulse
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D13 :3 usec - short delay
D16 :500 usec - delay for homospoil/gradient recovery
Parmod :2D
ND0 :1
TD2 :1 K data points in F2
TD1 :256 data points in F1
SW2 :10 ppm
SW1 :10 ppm
O1 : middle of ^{1}H NMR spectrum
NS :1
DS :16
gpnam1 :SINE.100
gpz 1 : 10%
gpnam2 :SINE.100
gpz 2 : 10%
IN0 :1/[SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :SINE
SSB(F2) :0
PH-mod(F2) :no
MC2 :QF

SYM (may be performed)
phase correction :not necessary

SI(F1) :512 W
WDW(F1) :SINE
SSB(F1) :0
PH-mod(F1) :mc
XFB :fourier transformation in both directions

plot :use XWINPLOT

Experiment 12.2

- Phase-Sensitive gs-DQF-COSY

pulse program: cosydfgptp.mo
2D homonuclear shift correlation, using gradients as double quantum filter, phase sensitive using TPPI.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
D0 :3 usec - incremented delay
D13 :3 usec – short delay
D20 :2 msec – same length as gradient pulse
Parmod :2D
TD2 :2 K data points in F2
SW2 :10 ppm
O1 : middle of ^{1}H NMR spectrum
NS :4
gpnam1 :SINE.100
gpnam2 :SINE.100
IN0 :1/[2*SW1]

P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P16 :2 msec - homospoil/gradient pulse
D1 :2 sec - relaxation delay
D16 :500 usec - delay for homospoil/gradient recovery
ND0 :2
TD1 :512 data points in F1
SW1 :10 ppm
DS :16
gpz 1 : 10%
gpz 2 : 20%
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :1 K
WDW(F2) :EM or GM
LB(F2) : depending on the
GB(F2) : resolution
PH-mod(F2) :pk
PHC0(F2) :should be 0 before first transformation
PHC1(F2) :should be 0 before first transformation
MC2 :TPPI

phase correction :use the 2D-phase correction routine
plot :use XWINPLOT

SI(F1) :1 K
WDW(F1) :EM or GM
LB(F1) : depending on the
GB(F1) : resolution
PH-mod(F1) :pk
PHC0(F1) :90

PHC1(F1) :should be 0 before first transformation
XFB :fourier transformation in both directions
XF2P :will be executed after correction of the rows

Experiment 12.3
- gs-HMQC

pulse program: inv4gp
This is a HMQC experiment with pulsed field gradients and a BIRD filter to suppress the signals of protons bond to ^{12}C.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
CPD2 :GARP - CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D2 :1/[2J(C,H)]= 3.57 msec, calculated from $^{1}J(C,H)$=140 Hz
D16 :500 usec - delay for homospoil/gradient recovery
Parmod :2D
TD2 :1 K data points in F2
SW2 :10 ppm
O1 : middle of ^{1}H NMR spectrum
NS :1
gpnam1 :SINE.100
gpnam2 :SINE.100
gpnam3 :SINE.100
IN0 :1/[2*SW1]

P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
P16 :2 msec - homospoil/gradient pulse
PCPD2 :f2 channel - 90° pulse for decoupling sequence
D12 :20 usec - delay for power switching
D13 :3 usec - short delay
D20 :D2-P16-D13-D12, but ≥D16
ND0 :2
TD1 :256 data points in F1
SW1 :165 ppm
O2 :middle of ^{13}C NMR spectrum
DS :16
gpz 1 : 50%
gpz 2 : 30%
gpz 3 : 40.1
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :EM
LB(F2) :5 Hz
PH-mod(F2) :no
MC2 :QF
phase correction :not necessary

SI(F1) :512 W
WDW(F1) :QSINE
SSB(F1) :3
PH-mod(F1) :mc
XFB :fourier transformation in both directions
plot :use XWINPLOT

Experiment 12.4
- gs-HMBC

pulse program: inv4gplplrnd
This is a HMBC (Heteronuclear Multiple Bond Correlation) pulse sequence to obtain a H,C correlation via $^{2}J(C,H)$ and $^{3}J(C,H)$. It is a gradient-selected version without decoupling.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
D0 :3 usec - incremented delay
D2 :1/[2J(C,H)]= 3.57 msec, calculated from $^{1}J(C,H)$=140 Hz
D13 :3 usec - short delay
Parmod :2D
TD2 :1 K data points in F2
SW2 :10 ppm
O1 : middle of ^{1}H NMR spectrum
NS :2
gpnam1 :SINE.100
gpnam2 :SINE.100
gpnam3 :SINE.100
IN0 :1/[2*SW1]

P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
P16 :2 msec - homospoil/gradient pulse
D1 :2 sec - relaxation delay
D6 :1/[2J(C,H)]= 60 msec, calculated from $^{n}J(C,H)$=8 Hz
D16 :500 usec - delay for homospoil/gradient recovery
ND0 :2
TD1 :256 data points in F1
SW1 :165 ppm
O2 :middle of ^{13}C NMR spectrum
DS :16
gpz 1 : 50%
gpz 2 : 30%
gpz 3 : 40.1
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :EM
LB(F2) :5 Hz
PH-mod(F2) :no
MC2 :QF
phase correction :not necessary

SI(F1) :512 W
WDW(F1) :QSINE
SSB(F1) :3
PH-mod(F1) :mc
XFB :fourier transformation in both directions
plot :use XWINPLOT

Experiment 12.5

- ACCORD-HMBC

pulse program: inv4acgplplr.mo
This is a HMBC (Heteronuclear Multiple Bond Correlation) pulse sequence to obtain a H,C correlation via $^{2}J(C,H)$ and $^{3}J(C,H)$. It is a gradient-selected version without decoupling.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
PL12 :f2 channel - power level for CPD decoupling, 12 dB

P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
PCPD2 :f2 channel - 90° pulse for decoupling sequence, 70 usec
P16 :1 msec - homospoil/gradient pulse

CPD2 :GARP - CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D2 :1/[2J(C,H)]= 3.57 msec, calculated from ^{1}J(C,H)=140 Hz
D6 :1/[2J(C,H)]= 200 msec, calculated from $^{2/3}$J(C,H)=2.5 Hz
D16 :50 usec - delay for homospoil/gradient recovery
D20 :1s/(2*(cnst6)+0.146*(cnst7-cnst6))-p16-d16=2.7 msec
D21 :1s/(2*(cnst7)-0.146*(cnst7-cnst6))-p16-d16=2.1 msec
cnst6 :J(XH)min, here 128 Hz
cnst7 :J(XH)max, here 163 Hz
Parmod :2D
ND0 :2
TD2 :2 K data points in F2
TD1 :256 data points in F1
SW2 :10 ppm
SW1 :165 ppm
O1 : middle of ^{1}H NMR spectrum
O2 :middle of ^{13}C NMR spectrum
NS :2
DS :16
gpnam1 :SINE.100
gpz 1 : 15%
gpnam2 :SINE.100
gpz 2 : -10%
gpnam3 :SINE.100
gpz 3 : -5%
gpnam4 :SINE.100
gpz 4 : 50%
gpnam5 :SINE.100
gpz 5 : 30%
gpnam6 :SINE.100
gpz 6 : 40%
gpnam7 :SINE.100
gpz 7 : -5%
gpnam8 :SINE.100
gpz 8 : 5%
IN0 :1/[2*SW1]
IN6 :(200 msec-20 msec)/td1=0.7 msec, corresponding 2.5 Hz to 25Hz

RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :1 K
SI(F1) :512 W
WDW(F2) :EM
WDW(F1) :QSINE
LB(F2) :5 Hz
SSB(F1) :3
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
phase correction :not necessary
plot :use XWINPLOT

Experiment 12.6

- Phase-Sensitive gs-HSQC with Sensitivity Enhancement

pulse program: invietgpsi
The HSQC (Heteronuclear Single Quantum Coherence) method performs the H,C correlation via the ^{13}C chemical shift evolution of a single quantum coherence. In this case it is a gradient-selected correlation using echo/antiecho selection method.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
P4 :f2 channel - 180° ^{13}C decoupler pulse
P16 :1.6 msec - homospoil/gradient pulse

P28 :2 msec - trim pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :GARP - CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D4 :1/[4J(C,H)]= 1.8 msec, calculated from ^{1}J(C,H)=140 Hz
D11 : 30 msec - delay for disk I/O
D13 :3 usec - short delay
D16 :100 usec - delay for homospoil/gradient recovery
D24 :1/(8 J(XH) for all multiplicities
L3 :loop for phase sensitive 2D using E/A method : L3=TD1/2=64
Parmod :2D
ND0 :2
TD2 :1 K data points in F2
TD1 :2 times 64 data points in F1
SW2 :10 ppm
SW1 :165 ppm
O1 : :middle of ^{1}H NMR spectrum
O2 :middle of ^{13}C NMR spectrum
NS :1
DS : =16
gpnam1 :SINE.100
gpz 1 : 80%
gpnam2 :SINE.100
gpz 2 : 20.1%
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :512 W
WDW(F2) :EM
WDW(F1) :GM
LB(F2) :3 Hz
PH-mod(F2) :pk
PH-mod(F1) :no
PHC0(F2) :should be 0 before first transformation
PHC1(F2) :should be 0 before first transformation
MC2 :echo-antiecho
XFB :fourier transformation in both directions
phase correction :use the 2D-phase correction routine, phase correction is usually only necessary in F2
XF2P :will be executed after correction of the rows
plot :use XWINPLOT

Experiment 12.7

- gs-TOCSY

pulse program: mlevgp.mo
This experiment is the gradient-selected version of the TOCSY (Total Correlation SpectroscopY) method, which can be done with one scan.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
PL10 :f1 channel - power level for TOCSY-spinlock
P5 :f1 channel - 60° ^{1}H transmitter low power pulse
P6 :f1 channel - 90° ^{1}H transmitter low power pulse
P7 :f1 channel - 180° ^{1}H transmitter low

power pulse
P16 :2 msec - homospoil/gradient pulse
P17 :f1 channel - 2.5 msec - trim pulse

D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D9 : 100 msec- mixing time
D12 :20 usec - delay for power switching
D16 :500 usec - delay for homospoil/gradient recovery

Parmod :2D
ND0 :1
TD2 :1 K data points in F2
TD1 :256 data points in F1
SW2 :9 ppm
SW1 :9 ppm
O1 : middle of ^{1}H NMR spectrum
NS :1
DS :16
gpnam1 :SINE.100
gpz 1 : 10%
gpnam2 :SINE.100
gpz 2 : 10%
IN0 :1/[SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :512 W
WDW(F2) :SINE
WDW(F1) :SINE
SSB(F2) :0
SSB(F1) :0
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
phase correction :not necessary
plot :use XWINPLOT

Experiment 12.8

- gs-HMQC-TOCSY

pulse program: inv4gpml
This is a combination of the HMQC (Heteronuclear Multiple Quantum Coherence) method with the TOCSY (Total Correlation SpectroscopY) sequence. Starting from each HMQC cross-signal one finds in F1 additional signals which are caused by a TOCSY transfer. This variant is a gradient-selected method, which does not need a BIRD filter.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}Htransmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
PL10 :f1 channel - power level for TOCSY-spinlock
P5 :f1 channel - 60° ^{1}H transmitter low power pulse
P6 :f1 channel - 90° ^{1}H transmitter low power pulse
P7 :f1 channel - 180° ^{1}H transmitter low power pulse
P16 :2 msec - homospoil/gradient pulse
P17 :f1 channel - 2.5 msec - trim pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
CPD2 :GARP - CPD decoupling sequence,

defined by cpdprg2

D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D2 :1/[2J(C,H)]= 3.57 msec, calculated from ^{1}J(C,H)=140 Hz
D9 : 81.8 msec - mixing time
D12 :20 usec - delay for power switching
D13 :3 usec - short delay
D16 :500 usec - delay for homospoil/gradient recovery
D21 :P16+D16+D12
Parmod :2D
ND0 :2
TD2 :1 K data points in F2
TD1 :256 data points in F1
SW2 :10 ppm
SW1 :165 ppm
O1 : middle of ^{1}H NMR spectrum
O2 :middle of ^{13}C NMR spectrum
NS :4
DS :16
gpnam1 :SINE.100
gpz 1 : 50%
gpnam2 :SINE.100
gpz 2 : 30%
gpnam3 :SINE.100
gpz 3 : 40.1%
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :512 W
WDW(F2) :SINE
WDW(F1) :SINE
SSB(F2) :0
SSB(F1) :0
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
phase correction :not necessary
plot :use XWINPLOT

Experiment 12.9

- 2Q-HMBC

pulse program:

This experiments detects long-range carbon-carbon connectivities. The transfer from protons to ^{13}C uses a ^{3}J(C,H) or a ^{2}J(C,H) instead of a ^{1}J(C,H) coupling and that it is not dependent on the C,C coupling constants. Therefore carbon-carbon relationships can also be detected, where the C,C spin coupling constant is close to zero.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
PL2 :f2 channel – high power level for ^{13}C decoupler pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
P4 :f2 channel - 180° ^{13}C decoupler pulse
P16 :1 msec - homospoil/gradient pulse
P28 :f1 channel - 2 msec - trim pulse
D0 :3 usec - incremented delay
D1 :4 sec - relaxation delay
D11 :30 msec – delay for disk I/O
D16 :100 usec - delay for homospoil/gradient recovery
cnst0 :ds=ns*2*cnst0
cnst6 :159 Hz
cnst7 :179 Hz
cnst8 :8.5 Hz
Parmod :2D
ND0 :2
TD2 :1 K data points in F2
TD1 :128 data points in F1

SW2 :5.2 ppm

SW1 :206 ppm (C,C double quantum frequency)

O1 :center of ^{1}H NMR spectrum
O2 :center of ^{13}C NMR spectrum
NS :32
L3 :TD1/2
gpnam1 :SINE.100
gpz 1 : 30%
gpnam2 :SINE.100
gpz 2 : -20%
gpnam3 :SINE.100
gpz 3 : -10%
gpnam4 :SINE.100
gpz 4 : 30%
gpnam5 :SINE.100
gpz 5 : -10%
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :1024 W
SI(F1) :256 W
WDW(F2) :SINE
WDW(F1) :QSINE
SSB(F2) :4
SSB(F1) :2
PH-mod(F2) :pk
PH-mod(F1) :no
PHC0(F2) :should be 0 before first transformation
MC2 :echo-antiecho
XFB :fourier transformation in both directions
phase correction :use the 2D-phase correction routine, phase correction is usually only necessary in F2
XF2P :will be executed after correction of the rows

Experiment 12.10

- Gradient-Selected ^{1}H-Detected 2D INEPT-INADEQUATE

pulse program: ineptinadgp.mo (ineptingp_mo)
This experiment detects carbon-carbon connectivities, but starts from ^{1}H magnetization and detects ^{1}H magnetization. The suppressing of protonsbond to ^{12}C is achieved by the use of pulsed field gradients. Connectivities between two quaternary carbon atoms cannot be detected.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
PL2 :f2 channel – high power level for ^{13}C decoupler pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
P4 :f2 channel - 180° ^{13}C decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
PCPD2 :f2 channel - 90° pulse for decoupling sequence
P16 :1 msec - homospoil/gradient pulse
P28 :f1 channel - 2 msec - trim pulse
CPD2 :GARP - CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D1 :1.5 sec - relaxation delay
D4 :1/[4J(C,H)]= 1.8 msec, calculated from ^{1}J(C,H)=140 Hz
D11 :30 msec – delay for disk I/O
D16 :200 usec - delay for
D20 :D4-P16-D16-4u

homospoil/gradient recovery

D21 :D16+P2+D0*2

D23 :1/[4J(C,C)]= 5 msec, calculated from ^{1}J(C,C)=50 Hz

cnst0 :ds=ns*2*cnst0
L3 :TD1/2
Parmod :2D
ND0 :2
TD2 :1 K data points in F2
TD1 :2 times 512 data points in F1
SW2 :3.7 ppm
SW1 :80 ppm (C,C double quantum frequency)
O1 :center of ^{1}H NMR spectrum
O2 :center of ^{13}C NMR spectrum
NS :12
DS :16
gpnam1 :SINE.100
gpz 1 : 39.7%
gpnam2 :SINE.100
gpz 2 : 39.7%
gpnam3 :SINE.100
gpz 3 : 40%
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :1024 W
SI(F1) :1024 W
WDW(F2) :EM
WDW(F1) :QSINE
LB(F2) :6.5
SSB(F1) :2
PH-mod(F2) :pk
PH-mod(F1) :no
PHC0(F2) :should be 0 before first transformation
MC2 :echo-antiecho
XFB :fourier transformation in both directions
phase correction :use the 2D-phase correction routine, phase correction is usually only necessary in F2
XF2P :will be executed after correction of the rows

Experiment 12.11

- gs-NOESY

pulse program: noesygpst
This gs-NOESY method replaces the phase cycling procedure by one pulsed field gradient during the entire mixing time. In practice, only two transients for each t_1 increment are needed.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}Htransmitter pulse
P16 :1 msec - homospoil/gradient pulse
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D8 :250 msec - mixing time
D11 :30 msec - delay for disk I/O
D16 :100 usec - delay for homospoil/gradient recovery
D20 :D8*0.5 – p16 – d16
Parmod :2D
ND0 :1
TD2 :2 K data points in F2
TD1 :256 data points in F1
SW2 :10 ppm
SW1 :10 ppm
O1 : middle of ^{1}H NMR spectrum
L3 :l3=td1/2 – loop for States-TPPI
NS :2
DS :16

gpnam1 :SINE.100
gpnam2 :SINE.100
IN0 :1/[1*SW1]
gpz 1 : 40%
gpz 2 : -40%
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :EM
LB(F2) :2
PH-mod(F2) :pk
PHC1(F2) :should be 0 before first transformation
MC2 :States-TPPI
SI(F1) :512 W
WDW(F1) :QSINE
SSB(F1) :2
PH-mod(F1) :pk
PHC1(F1) :should be 0 before first transformation
XFB :fourier transformation in both directions

phase correction :use the 2D-phase correction routine, correct the signals positive

XF2P :will be executed after correction of the rows
XF1P :will be executed after correction of the columns

Experiment 12.12

- gs-HSQC-NOESY

pulse program: invietgpno.mo

It is very difficult to observe and evaluate NOESY cross peaks if the corresponding diagonal signals are very close together or overlap. A remedy to these problems can be achieved by editing the NOESY spectra by the carbon chemical shift. The acquisition of the data is performed without ^{13}C decoupling, which allows one to observe an NOE effect between a proton bound to ^{13}C and a proton in the same molecule with the identical chemical shift but bound to ^{12}C.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL2 :f2 channel – high power level for ^{13}C decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
CPD2 :GARP - CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay
D4 :1/[4J(C,H)]= 1.56 msec, calculated from $^{1}J(C,H)$=160 Hz
D11 :30 msec – delay for disk I/O
D16 :200 usec - delay for homospoil/gradient recovery
Parmod :2D
TD2 :1 K data points in F2

P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
P4 :f2 channel - 180° ^{13}C decoupler pulse
PCPD2 :f2 channel - 90° pulse for decoupling sequence
P16 :1 msec - homospoil/gradient pulse
P28 :f1 channel - 1 msec - trim pulse
D1 :1 sec - relaxation delay
D8 :2 sec – mixing time
D12 :20 usec – delay for power switching
ND0 :2
TD1 :64 data points in F1

SW2 :2.0 ppm
O1 :center of ^{1}H NMR spectrum
NS :12

gpnam1 :SINE.100
gpnam2 :SINE.100
gpnam3 :SINE.100
gpnam4 :SINE.100
IN0 :1/[2*SW1]

SW1 :12 ppm
O2 :center of ^{13}C NMR spectrum
DS :>=16
L3 :TD1/2
gpz 1 : 50%
gpz 2 : 80%
gpz 3 : 30%
gpz 4 : 20.1%
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :EM
LB(F2) :10
PH-mod(F2) :pk
PHC0(F2) :should be 0 before first transformation
XFB :fourier transformation in both directions

SI(F1) :128 W
WDW(F1) :EM
SSB(F1) :3
PH-mod(F1) :no
MC2 :echo-antiecho

phase correction :use the 2D-phase correction routine, phase correction is usually only necessary in F2. The NOE signals have the opposite phase.

XF2P :will be executed after correction of the rows

Experiment 12.13

- gs-HOESY

pulse program:

This gs-HOEY experiment yields information on the spatial relationship between spins in the heteronuclear case. It will be of main value in cases where information from spin-spin couplings is unhelpful or unavailable.

Setting of the needed channels: F1: ^{1}H
F2: ^{7}Li

Acquisition parameters

Processing parameters

Experiment 12.14

- ^{1}H, ^{15}N Correlation with gs-HMQC

pulse program: inv4gpnd.mo

Due to the low receptivity of ^{15}N it is very tedious to obtain ^{15}N NMR spectra of organic compounds if they are available only in miligrams. Inverse detection is therefore the method of choice, particularily if the unwanted signals can be efectively suppressed with pulsed field gradients.

Setting of the needed channels: F1: ^{1}H
F2: ^{15}N

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse

PL2 :f2 channel - high power level for ^{15}N decoupler pulse

D0 :3 usec - incremented delay

D2 :1/[2J(N,H)]= 50 msec, calculated from $^{2,3}J(C,H)$=10 Hz

D16 :100 usec - delay for homospoil/gradient recovery

D21 :D16+P2+D0*2

Parmod :2D

TD2 :1 K data points in F2

SW2 :10 ppm

O1 :middle of ^{1}H NMR spectrum

NS :4

gpnam1 :SINE.100

gpnam2 :SINE.100

gpnam3 : SINE.100

IN0 :1/[2*SW1]

P1 :f1 channel - 90° ^{1}H transmitter pulse

P2 :f1 channel - 180° ^{1}H transmitter pulse

P3 :f2 channel - 90° ^{15}N decoupler pulse

P16 :2 msec - homospoil/gradient pulse

D1 :2 sec - relaxation delay

D11 : 30 msec - delay for disk I/O

D20 :D4-P16-D16-4usec

D23 : 1/[4J(C,C)]= 5 msec, calculated from $^{1}J(C,C)$=50 Hz

ND0 :2

TD1 :128 data points in F1

SW1 :400 ppm

O2 :middle of ^{15}N NMR spectrum

DS : 16

gpz 1 : 55%

gpz 2 : 45%

gpz 3 : 20.14%

RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W

WDW(F2) :SINE

LB(F2) :2

PH-mod(F2) :no

MC2 :QF

phase correction :phase correction is not necessary

SI(F1) :512 W

WDW(F1) :SINE

SSB(F1) :2

PH-mod(F1) :mc

XFB :fourier transformation in both directions

plot :use XWINPLOT

Chapter 13
- The Third Dimension

Summary

Experiment	Pulse program	Description
13.1	invbcosytp3d.mo	3D HMQC-COSY
13.2	invigpml3d.mo	3D gs-HSQC-TOCSY
13.3	h_c_p3d.mo	3D H,C,P-Correlation
13.4		3D HMBC

Experiment 13.1
- 3D HMQC-COSY

pulse program: invbcotp3d.mo
This is a 3D experiment, in which the COSY spectra are ”edited” via C,H correlation.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{13}C decoupler pulse
PL12 :f2 channel - power level for CPD decoupling
CPD2 :GARP - CPD decoupling sequence, defined by cpdprg2
D0 :3 usec - incremented delay (F1 in 3D)
D1 :2 sec - relaxation delay
D2 :1/[2J(C,H)]= 3.5 msec, calculated from $^{1}J(C,H)$= 145 Hz
Parmod :3D
TD3 :256 data points in F3 (^{1}H)
TD1 :128 data points in F1 (^{13}C)
SW3 :3.3 ppm
SW1 :42 ppm
O1 : middle of selected ^{1}H NMR region
O3 :middle of selected ^{13}C NMR region
NS :4
IN0 :1/[4*SW1]
RG :receiver gain for correct ADC input

P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P3 :f2 channel - 90° ^{13}C decoupler pulse
P4 :f2 channel - 180° ^{13}C decoupler pulse
PCPD2 :f2 channel - 90° pulse for decoupling sequence
D7 :ca. 0.4 sec - BIRD delay to be optimized for minimum FID
D10 :3 usec - incremented delay (F2 in 3D)
D11 : 30 msec - delay for disk I/O
ND0 :4
TD2 :64 data points in F2 (^{1}H)
ND10 :2
SW2 :3.3 ppm
O2 :middle of selected ^{1}H NMR region
DS :32
IN10 :1/[2*SW2]

Processing parameters

SI(F3) :256 W
SI(F2) :128 W

SI(F1) :128 W
WDW(F3) :EM
WDW(F2) :QSINE
WDW(F1) :QSINE
LB(F3) :5 Hz
SSB(F2) :2
SSB(F1) :2
MC2(F2) :TPPI
MC2(F1) :TPPI
PH-mod(F3) :pk
PH-mod(F2) :pk
PH-mod(F1) :pk
PHC0(F3) :should be 0 before first transformation
PHC1(F3) :should be 0 before first transformation
PHC0(F2) :should be 0 before first transformation
PHC1(F2) :should be 0 before first transformation
PHC0(F1) :should be 0 before first transformation
PHC1(F1) :should be 0 before first transformation
AQORDER :3 - 1 - 2
TF3, TF2, TF1 :fourier transformation in all dimensions
phase correction :should be performed after the FT of each dimension
plot :use XWINPLOT

Experiment 13.2
- 3D gs-HSQC-TOCSY

pulse program:

This 3D experiment is a combination of a HSQC (Heteronuclear Single Quantum Coherence) and a TOCSY (Total Correlation SpectroscopY) sequence. It is a gradient-selected experiment.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

Processing parameters

Experiment 13.3
- 3D H,C,P-Correlation

pulse program:

3D experiment to measure the correlation between three different nuclei.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C
F3: ^{31}P

Acquisition parameters

Processing parameters

Experiment 13.4
- 3D HMBC

pulse program:

Instead of measuring several HMBC spectra with different delays, a 3D version was recently proposed in which the corresponding delay is incremented; thus the whole range of C,H long-range coupling constants are actually used for double quantum excitation.

Setting of the needed channels: F1: ^{1}H
F2: ^{13}C

Acquisition parameters

Processing parameters

Chapter 14
- Solid-State NMR Spectroscopy

Summary

Experiment	Pulse program	Description
14.1		Shimming Solid-State Probe-Heads
14.2		Adjusting the Magic Angle
14.3		Hartmann-Hahn Matching
14.4		The Basic CP/MAS Experiment
14.5		TOSS
14.6		SELTICS
14.7		Multiplicity Determination in the Solid-State

Experiment 14.1
- Shimming Solid-State Probe-Heads

a)pulse program:

In solid-state NMR there is usually no lock channel and because of that a resonable basic shim is necessary.

Setting of the needed channels: F1: ^{1}H
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D2 :1/[2J(N,H)]= 50 msec, calculated from ^{2,3}J(C,H)=10 Hz
D11 : 30 msec - delay for disk I/O
D16 :100 usec - delay for homospoil/gradient recovery
D20 :D4-P16-D16-4usec
D21 :D16+P2+D0*2
D23 : 1/[4J(C,C)]= 5 msec, calculated from ^{1}J(C,C)=50 Hz
TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :10 ppm
SW1 :400 ppm
O1 :middle of ^{1}H NMR spectrum
O2 :middle of ^{15}N NMR spectrum
NS :4
DS : 16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :512 W
WDW(F2) :SINE
WDW(F1) :SINE
LB(F2) :2
SSB(F1) :2
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
phase correction :phase correction is not
plot :use XWINPLOT

necessary

b)pulse program:

In solid-state NMR there is usually no lock channel and because of that a resonable basic shim is necessary.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel – high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
PL2 :f2 channel – high power level for ^{15}N decoupler pulse
P3 :f2 channel - 90° ^{15}N decoupler pulse
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D2 :1/[2J(N,H)]= 50 msec, calculated from $^{2,3}J(C,H)$=10 Hz
D11 : 30 msec - delay for disk I/O
D16 :100 usec - delay for homospoil/gradient recovery
D20 :D4-P16-D16-4usec
D21 :D16+P2+D0*2
D23 : 1/[4J(C,C)]= 5 msec, calculated from $^{1}J(C,C)$=50 Hz
TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :10 ppm
SW1 :400 ppm
O1 :middle of ^{1}H NMR spectrum
O2 :middle of ^{15}N NMR spectrum
NS :4
DS : 16
IN0 :1/[2*SW1]
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :512 W
WDW(F2) :SINE
WDW(F1) :SINE
LB(F2) :2
SSB(F1) :2
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
phase correction :phase correction is not necessary
plot :use XWINPLOT

Experiment 14.2

– Adjusting the Magic Angle

pulse program:

Setting of the needed channels: F1: ^{79}Br
F2: off

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D2 :1/[2J(N,H)]= 50 msec, calculated from $^{2,3}J(C,H)$=10 Hz
D11 : 30 msec - delay for disk I/O

D16 :100 usec - delay for homospoil/gradient recovery
D20 :D4-P16-D16-4usec
D21 :D16+P2+D0*2
D23 : 1/[4J(C,C)]= 5 msec, calculated from ^{1}J(C,C)=50 Hz
TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :10 ppm
SW1 :400 ppm
O1 :middle of ^{1}H NMR spectrum
O2 :middle of ^{15}N NMR spectrum
NS :4
DS : 16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :512 W
WDW(F2) :SINE
WDW(F1) :SINE
LB(F2) :2
SSB(F1) :2
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
phase correction :phase correction is not necessary
plot :use XWINPLOT

Experiment 14.3

- Hartmann-Hahn Matching

pulse program:

Standard CP/MAS spectra are acquired with Cross Polarization from protons to carbon.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{15}N decoupler pulse
P3 :f2 channel - 90° ^{15}N decoupler pulse
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D2 :1/[2J(N,H)]= 50 msec, calculated from ^{2,3}J(C,H)=10 Hz
D11 : 30 msec - delay for disk I/O
D16 :100 usec - delay for homospoil/gradient recovery
D20 :D4-P16-D16-4usec
D21 :D16+P2+D0*2
D23 : 1/[4J(C,C)]= 5 msec, calculated from ^{1}J(C,C)=50 Hz
TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :10 ppm
SW1 :400 ppm
O1 :middle of ^{1}H NMR spectrum
O2 :middle of ^{15}N NMR spectrum
NS :4
DS : 16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :512 W
WDW(F2) :SINE
WDW(F1) :SINE
LB(F2) :2
SSB(F1) :2

PH-mod(F2) :no
MC2 :QF

phase correction :phase correction is not necessary

PH-mod(F1) :mc
XFB :fourier transformation in both directions
plot :use XWINPLOT

Experiment 14.4
– The Basic CP/MAS Experiment

pulse program:

The CP/MAS method provides high resolution NMR spectra in the solid state and is mostly performed on ^{13}C with cross polarization from ^{1}H.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{15}N decoupler pulse
D0 :3 usec - incremented delay
D2 :1/[2J(N,H)]= 50 msec, calculated from $^{2,3}J(C,H)$=10 Hz
D16 :100 usec - delay for homospoil/gradient recovery
D21 :D16+P2+D0*2

TD2 :1 K data points in F2
SW2 :10 ppm
O1 :middle of ^{1}H NMR spectrum
NS :4

P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P3 :f2 channel - 90° ^{15}N decoupler pulse
D1 :2 sec - relaxation delay
D11 : 30 msec - delay for disk I/O
D20 :D4-P16-D16-4usec
D23 : 1/[4J(C,C)]= 5 msec, calculated from $^{1}J(C,C)$=50 Hz
TD1 :128 data points in F1
SW1 :400 ppm
O2 :middle of ^{15}N NMR spectrum
DS : 16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :SINE
LB(F2) :2
PH-mod(F2) :no
MC2 :QF

phase correction :phase correction is not necessary

SI(F1) :512 W
WDW(F1) :SINE
SSB(F1) :2
PH-mod(F1) :mc
XFB :fourier transformation in both directions
plot :use XWINPLOT

Experiment 14.5
- TOSS

pulse program:

TOSS is a technique to suppress the spinning side-bands.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{15}N decoupler pulse
P3 :f2 channel - 90° ^{15}N decoupler pulse
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D2 :1/[2J(N,H)]= 50 msec, calculated from ^{2,3}J(C,H)=10 Hz
D11 : 30 msec - delay for disk I/O
D16 :100 usec - delay for homospoil/gradient recovery
D20 :D4-P16-D16-4usec
D21 :D16+P2+D0*2
D23 : 1/[4J(C,C)]= 5 msec, calculated from ^{1}J(C,C)=50 Hz
TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :10 ppm
SW1 :400 ppm
O1 :middle of ^{1}H NMR spectrum
O2 :middle of ^{15}N NMR spectrum
NS :4
DS : 16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
SI(F1) :512 W
WDW(F2) :SINE
WDW(F1) :SINE
LB(F2) :2
SSB(F1) :2
PH-mod(F2) :no
PH-mod(F1) :mc
MC2 :QF
XFB :fourier transformation in both directions
phase correction :phase correction is not necessary
plot :use XWINPLOT

Experiment 14.6

- SELTICS

pulse program:

SELTICS (Sideband ELimination by Temporary Interruption of the Chemical Shift) is another method to suppress the spinning side-bands.

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{15}N decoupler pulse
P3 :f2 channel - 90° ^{15}N decoupler pulse
D0 :3 usec - incremented delay
D1 :2 sec - relaxation delay
D2 :1/[2J(N,H)]= 50 msec, calculated from ^{2,3}J(C,H)=10 Hz
D11 : 30 msec - delay for disk I/O
D16 :100 usec - delay for homospoil/gradient recovery
D20 :D4-P16-D16-4usec
D21 :D16+P2+D0*2
D23 : 1/[4J(C,C)]= 5 msec, calculated from ^{1}J(C,C)=50 Hz
TD2 :1 K data points in F2
TD1 :128 data points in F1
SW2 :10 ppm
SW1 :400 ppm
O1 :middle of ^{1}H NMR spectrum
O2 :middle of ^{15}N NMR spectrum

NS :4
DS : 16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :SINE
LB(F2) :2
PH-mod(F2) :no
MC2 :QF
phase correction :phase correction is not necessary
SI(F1) :512 W
WDW(F1) :SINE
SSB(F1) :2
PH-mod(F1) :mc
XFB :fourier transformation in both directions
plot :use XWINPLOT

Experiment 14.7

- Multiplicity Determination in the Solid-State

pulse program:

The first editing method was baptized NQS (Non Quaternary Suppression).

Setting of the needed channels: F1: ^{13}C
F2: ^{1}H

Acquisition parameters

PL1 :f1 channel - high power level for ^{1}H transmitter pulse
PL2 :f2 channel - high power level for ^{15}N decoupler pulse
D0 :3 usec - incremented delay
D2 :1/[2J(N,H)]= 50 msec, calculated from $^{2,3}J(C,H)$=10 Hz
D16 :100 usec - delay for homospoil/gradient recovery
D21 :D16+P2+D0*2
TD2 :1 K data points in F2
SW2 :10 ppm
O1 :middle of ^{1}H NMR spectrum
NS :4
P1 :f1 channel - 90° ^{1}H transmitter pulse
P2 :f1 channel - 180° ^{1}H transmitter pulse
P3 :f2 channel - 90° ^{15}N decoupler pulse
D1 :2 sec - relaxation delay
D11 : 30 msec - delay for disk I/O
D20 :D4-P16-D16-4usec
D23 : 1/[4J(C,C)]= 5 msec, calculated from $^{1}J(C,C)$=50 Hz
TD1 :128 data points in F1
SW1 :400 ppm
O2 :middle of ^{15}N NMR spectrum
DS : 16
RG :receiver gain for correct ADC input

Processing parameters

SI(F2) :512 W
WDW(F2) :SINE
LB(F2) :2
PH-mod(F2) :no
MC2 :QF
phase correction :phase correction is not necessary
SI(F1) :512 W
WDW(F1) :SINE
SSB(F1) :2
PH-mod(F1) :mc
XFB :fourier transformation in both directions
plot :use XWINPLOT