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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.

Introduction
Chapter 2
- Determination of the Pulse-Duration

Summary

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<td>Determination of the 90° $^1$H Transmitter Pulse-Duration</td>
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<tr>
<td>2.2</td>
<td>zg0dc</td>
<td>Determination of the 90° $^{13}$C Transmitter Pulse-Duration</td>
</tr>
<tr>
<td>2.3</td>
<td>decp90</td>
<td>Determination of the 90° $^1$H Decoupler Pulse-Duration</td>
</tr>
<tr>
<td>2.4</td>
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<td>The 90° $^1$H Pulse with Inverse Spectrometer Configuration</td>
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<td>2.5</td>
<td>decp90</td>
<td>The 90° $^{13}$C Decoupler Pulse with Inverse Configuration</td>
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<tr>
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<td>zg</td>
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</tr>
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</table>

Experiment 2.1
- Determination of the 90° $^1$H 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
<table>
<thead>
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<th>Experiment 2.2</th>
<th>- Determination of the 90° $^{13}$C Transmitter Pulse Duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>pulse program:</td>
<td>zg0dc 1D-sequence with F2 decoupling, using p0 for any flip angle. Result is a standard $^{13}$C NMR spectrum with proton broad-band decoupling.</td>
</tr>
</tbody>
</table>
| 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 |

<table>
<thead>
<tr>
<th>Experiment 2.3</th>
<th>- Determination of the 90° $^1$H Decoupler Pulse Duration</th>
</tr>
</thead>
<tbody>
<tr>
<td>pulse program:</td>
<td>decp90 1D-sequence to determine the 90° decoupler pulse-duration</td>
</tr>
</tbody>
</table>
| 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/[2$^1$J(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
- **BC_mod**: quad
- **LB**: 2 Hz
- **phase correction**: adjust the doublet in antiphase and use the same values for the other phase corrections (PK).
- **baseline correction**: ABS
- **plot**: use XWINPLOT

---

**Experiment 2.4**

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

**Pulse program**: zg0

**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

**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
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**

<table>
<thead>
<tr>
<th>SI</th>
<th>2 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>WDW</td>
<td>EM</td>
</tr>
<tr>
<td>FT</td>
<td>fourier transformation</td>
</tr>
<tr>
<td>BC_mod</td>
<td>quad</td>
</tr>
<tr>
<td>LB</td>
<td>1 Hz</td>
</tr>
<tr>
<td>phase correction</td>
<td>adjust the phase of the big signal descended from the protons bound to $^{13}$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).</td>
</tr>
<tr>
<td>baseline correction</td>
<td>ABS</td>
</tr>
<tr>
<td>plot</td>
<td>use XWINPLOT</td>
</tr>
</tbody>
</table>

**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:

<table>
<thead>
<tr>
<th>F1</th>
<th>$^1$H</th>
</tr>
</thead>
<tbody>
<tr>
<td>F2</td>
<td>off</td>
</tr>
</tbody>
</table>

**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 |
| D1 | 30 sec - relaxation delay |
| TD | 64 K |
| O1 | $^{10}$ kHz towards higher frequencies from the resonance of the CHCl$_3$ signal |
| NS | 8 |

**Processing parameters**

Use the same processing parameters for both experiments

| SI | 32 K |
| WDW | EM |
| FT | fourier transformation |
| BC_mod | quad |
| LB | 1 Hz |
| 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:

<table>
<thead>
<tr>
<th>F1</th>
<th>$^1$H</th>
</tr>
</thead>
<tbody>
<tr>
<td>F2</td>
<td>off</td>
</tr>
</tbody>
</table>
Acquisition parameters

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

**D1**: 30 sec - relaxation delay

**TD**: 64 K

**O1**: 10 kHz towards higher frequencies from the resonance of the CHCl$_3$ signal

**NS**: 8

**P1**: F1 channel – 90° $^1$H transmitter pulse

**P2**: F1 channel – 180° $^1$H transmitter pulse

**D15**: 10 msec - fixed delay

**SW**: 80 ppm

**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

**plot**: use XWINPLOT

baseline correction :ABS

---

**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)

**D1**: 2 sec - relaxation delay

**TD**: 4 K

**O1**: on resonance of H$_2$O signal

**NS**: 1

**P0**: F1 channel - $^1$H transmitter pulse, a) 360° and b) 180°

**SW**: 500 Hz

**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

**phase correction**: adjust the phase to pure absorption

**plot**: use XWINPLOT, both traces should be plotted on the same vertical scale

baseline correction :ABS

---

**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  
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 determined for each attenuation level.  
D1: 60 sec - relaxation delay  
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  
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

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<td>Resolution Test for $^1$H NMR Spectroscopy</td>
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<td>Sensitivity Test for $^1$H NMR Spectroscopy</td>
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<td>3.13</td>
<td>zgphase.mo</td>
<td>$^{13}$ Phase Stability Test</td>
</tr>
</tbody>
</table>

### 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  
- **O1**: middle of the $^1$H NMR spectrum  
- **NS**: 8  
- **SW**: 20 ppm  
- **RG**: receiver gain for correct ADC input

**Processing parameters**

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

<table>
<thead>
<tr>
<th>Channel</th>
<th>Symbol</th>
<th>Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{13}$C</td>
<td></td>
</tr>
<tr>
<td>F2</td>
<td>$^1$H</td>
<td></td>
</tr>
</tbody>
</table>

**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

**Processing parameters**

- **SI**: 16 K
- **WDW**: EM
- **FT**: fourier transformation
- **BC_mod**: quad
- **LB**: 2 Hz
- **phase correction**: adjust the phases to pure absorption.
- **baseline correction**: ABS
- **plot**: use XWINPLOT
- **referencing**: set the TMS signal to 0 ppm.
- **peak picking**: choose the desired level

**Experiment 3.3**

- The Application of Window Functions

Pulse program: zg

Compare with Experiment 2.8

**Setting of the needed channels:**

<table>
<thead>
<tr>
<th>Channel</th>
<th>Symbol</th>
<th>Parameter</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^1$H</td>
<td></td>
</tr>
<tr>
<td>F2</td>
<td>off</td>
<td></td>
</tr>
</tbody>
</table>

**Acquisition parameters**

- **PL1**: F1 channel - high power level for $^1$H transmitter pulse
- **P1**: F1 channel - 90° $^1$H transmitter pulse
- **PLP**: F2 channel - power level for CPD decoupling
- **PCPD**: F2 channel – 90° pulse for decoupling sequence
- **D1**: 1 sec - relaxation delay
- **TD**: 32 K
- **O1**: middle of the $^{1}$H NMR spectrum
- **O2**: middle of $^{13}$C NMR spectrum
- **SW**: 1 ppm
- **DS**: 2
- **RG**: receiver gain for correct ADC input
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  
WDW :=EM  
WDW :=GM  
FT :fourier transformation  
plot :use XWINPLOT

Experiment 3.4  
- Computer-aided Spectral Analysis

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

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

Experiment 3.5  
- Line-Shape Test for 1H NMR Spectroscopy

pulse program: zg  
compare with Experiment 2.8

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

Transfer the spectrum to a PC. The procedure shown in the book was performed with the CALM software, obtainable on the Internet.
Acquisition parameters

**PL1**: f1 channel - high power level for $^1$H transmitter pulse
**P1**: f1 channel - $90^\circ$ $^1$H transmitter pulse
**D1**: 60 sec - relaxation delay
**TD**: 32 K
**O1**: on resonance of $^1$H signal
**NS**: 1

**SW**: 500 Hz
**spinning rate**: 20 Hz
**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.
**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:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^1$H</td>
</tr>
<tr>
<td>F2</td>
<td>off</td>
</tr>
</tbody>
</table>

### Experiment 3.7
- Sensitivity Test for $^1$H NMR Spectroscopy

pULSE program: zg
compare with Experiment 2.8

Setting of the needed channels:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^1$H</td>
</tr>
<tr>
<td>F2</td>
<td>off</td>
</tr>
</tbody>
</table>
transmitter pulse
D1 : 60 sec - relaxation delay
TD : 32 K
O1 : middle of the 1H 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.

cycle correction : 10 ppm

Experiment 3.8 - Line-Shape Test for 13C 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 1H off-resonance decoupled 13C NMR spectrum (O2 on resonance of 1H TMS signal) or an 1H single frequency decoupled 13C NMR spectrum (O2 on resonance on a special 1H group).

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

Acquisition parameters
PL1 : f1 channel - high power level for 13C transmitter pulse
PL14 : f2 channel - low power level for 1H decoupler pulse
D1 : 1 sec - relaxation delay
TD : 16 K
O1 : on resonance of 13C signal
NS : 1
spinning rate : 20 Hz

P1 : f1 channel - 90° 13C transmitter pulse
decoupler attenuation for continuous wave decoupling
D11 : 30 msec - delay for disk I/O
SW : 200 Hz
O2 : on resonance of 1H 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 true 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:**

<table>
<thead>
<tr>
<th>Channel</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{13}$C</td>
</tr>
<tr>
<td>F2</td>
<td>off</td>
</tr>
</tbody>
</table>

**Acquisition parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel - high power level for $^{13}$C transmitter pulse</td>
</tr>
<tr>
<td>P1</td>
<td>f1 channel - 90° $^{13}$C transmitter pulse</td>
</tr>
<tr>
<td>D1</td>
<td>300 sec - relaxation delay</td>
</tr>
<tr>
<td>TD</td>
<td>32 K</td>
</tr>
<tr>
<td>O1</td>
<td>middle of $^{13}$C NMR spectrum</td>
</tr>
<tr>
<td>NS</td>
<td>1</td>
</tr>
<tr>
<td>SW</td>
<td>200 ppm</td>
</tr>
<tr>
<td>decoupler</td>
<td>off</td>
</tr>
<tr>
<td>RG</td>
<td>receiver gain for correct ADC input</td>
</tr>
</tbody>
</table>

**Processing parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI</td>
<td>64 K</td>
</tr>
<tr>
<td>BC_mod</td>
<td>quad</td>
</tr>
<tr>
<td>WDW</td>
<td>EM</td>
</tr>
<tr>
<td>FT</td>
<td>Fourier transformation</td>
</tr>
<tr>
<td>baseline correction</td>
<td>ABS</td>
</tr>
<tr>
<td>phase correction</td>
<td>adjust the phase to pure absorption.</td>
</tr>
<tr>
<td>plot</td>
<td>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.</td>
</tr>
</tbody>
</table>

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:**

<table>
<thead>
<tr>
<th>Channel</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{13}$C</td>
</tr>
<tr>
<td>F2</td>
<td>$^1$H</td>
</tr>
</tbody>
</table>

**Acquisition parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel - high power level for $^{13}$C transmitter pulse</td>
</tr>
<tr>
<td>PL12</td>
<td>f2 channel - power level for CPD decoupling</td>
</tr>
<tr>
<td>CPD2</td>
<td>WALTZ16 - CPD decoupling sequence, defined by cpdprg2</td>
</tr>
<tr>
<td>D1</td>
<td>300 sec - relaxation delay</td>
</tr>
<tr>
<td>TD</td>
<td>64 K</td>
</tr>
<tr>
<td>O1</td>
<td>middle of $^{13}$C NMR spectrum</td>
</tr>
<tr>
<td>NS</td>
<td>1</td>
</tr>
<tr>
<td>P1</td>
<td>f1 channel - 90° $^{13}$C transmitter pulse</td>
</tr>
<tr>
<td>PCDP2</td>
<td>f2 channel – 90° pulse for decoupling sequence</td>
</tr>
<tr>
<td>D11</td>
<td>30 msec - delay for disk I/O</td>
</tr>
<tr>
<td>SW</td>
<td>200 ppm</td>
</tr>
<tr>
<td>O2</td>
<td>middle of $^1$H NMR spectrum</td>
</tr>
<tr>
<td>RG</td>
<td>receiver gain for correct ADC input</td>
</tr>
</tbody>
</table>

**Processing parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI</td>
<td>64 K</td>
</tr>
<tr>
<td>BC_mod</td>
<td>quad</td>
</tr>
<tr>
<td>WDW</td>
<td>EM</td>
</tr>
<tr>
<td>FT</td>
<td>Fourier transformation</td>
</tr>
<tr>
<td>baseline correction</td>
<td>ABS</td>
</tr>
<tr>
<td>phase correction</td>
<td>adjust the phase to pure absorption.</td>
</tr>
<tr>
<td>plot</td>
<td>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.</td>
</tr>
</tbody>
</table>
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

Processing parameters
- SI: 4 K
- WDW: EM
- FT: Fourier transformation

CY: set the intensity of the CHCl$_3$ signal to 1000 and enlarge the quadrature image signal, which is found 250 Hz towards higher frequencies from the offset position.

baseline correction: ABS

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

Processing parameters
- SI: 4 K
- WDW: EM
- FT: Fourier transformation

CY: set the intensity of the CHCl$_3$ signal to 1000 and enlarge the quadrature image signal, which is found 250 Hz towards higher frequencies from the offset position.

baseline correction: ABS

plot: use XWINPLOT
D1 : 5 sec – relaxation delay
TD : 32 K
O1 : middle of 1H NMR spectrum
NS : 1

SW : 10 ppm
RG : receiver gain for correct ADC input

Processing parameters
SI : 16 K
WDW : EM
FT : Fourier transformation

BC_mod : quad
LB : 0.2 Hz
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: 1H
F2: off

Acquisition parameters
Use an automation routine which performs this experiment 64 times in sequence
PL1 : f1 channel - high power level for 1H transmitter pulse, 3dB was used
P1 : f1 channel - 90° 1H transmitter pulse
D1 : 20 sec – relaxation delay
TD : 4 K
O1 : 37 Hz to higher frequencies from CHCl₃ signal

NS : 1
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 roughly for dispersion and always use the same digital phase correction (PK)

baseline correction : ABS
plot : use XWINPLOT
Chapter 4
- Decoupling Techniques

Summary

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<th>Pulse program</th>
<th>Description</th>
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<td>Decoupler Calibration for Homonuclear Decoupling</td>
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<td>4.3</td>
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<td>4.7</td>
<td>zgndspt.mo</td>
<td>The Heteronuclear SPT Experiment</td>
</tr>
<tr>
<td>4.8</td>
<td>zgfl2pr.mo</td>
<td>1D Nuclear Overhauser Difference Spectroscopy</td>
</tr>
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<td>4.9</td>
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</tr>
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<td>4.10</td>
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<td>4.11</td>
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<td>The Gated $^1$H-Decoupling Technique</td>
</tr>
<tr>
<td>4.12</td>
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<td>The Inverse Gated $^1$H-Decoupling Technique</td>
</tr>
<tr>
<td>4.13</td>
<td>zg0cw</td>
<td>$^1$H Single Frequency Decoupling of $^{13}$C NMR Spectra</td>
</tr>
<tr>
<td>4.14</td>
<td>zg0cw2.mo</td>
<td>$^1$H Low Power Decoupling of $^{13}$C NMR Spectra</td>
</tr>
<tr>
<td>4.15</td>
<td>hetnoe.mo</td>
<td>Measurement of the Heteronuclear Overhauser Effect</td>
</tr>
</tbody>
</table>

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

\[ D_1 = 2 \text{ sec} \] - relaxation delay

\[ TD = 4 \text{ K} \]

\[ O_1 = \text{on resonance of } ^1\text{H signal} \]

\[ \text{digmod} = \text{homodecoupling-digital} \]

\[ NS = 1 \]

\[ D_{12} = 20 \text{ usec} \] - delay for power switching

\[ SW = 500 \text{ Hz} \]

\[ O_2 = 50 \text{ Hz towards lower frequency from } O_1 \]

\[ TD = 4 \text{ K} \]

\[ SW = 500 \text{ Hz} \]

\[ O_1 = \text{on resonance of } ^1\text{H signal} \]

\[ \text{NS} = 1 \]

Processing parameters

\[ SI = 4 \text{ K or more, use zero-filling to ensure enough data points for the relatively small Bloch-Siegert shifts} \]

\[ BC_{\text{mod}} = \text{quad} \]

\[ WDW = \text{EM} \]

\[ FT = \text{Fourier transformation} \]

\[ \text{baseline correction} = \text{ABS} \]

**Experiment 4.2**

- Decoupler Calibration for Heteronuclear Decoupling

Pulse program: \( \text{zg0cw} \)

1D-sequence with CW decoupling, using \( p_0 \) for any flip angle. Depending where \( O_2 \) is set, different results are possible like an \(^1\text{H} \) off-resonance decoupled \(^{13}\text{C} \) NMR spectrum (\( O_2 \) on resonance of \(^1\text{H} \) TMS signal) or an \(^1\text{H} \) single frequency decoupled \(^{13}\text{C} \) NMR pectrum (\( O_2 \) on resonance on a special \(^1\text{H} \) group).

Setting of the needed channels:

\[ F_1: \quad ^{13}\text{C} \]

\[ F_2: \quad ^1\text{H} \]

Acquisition parameters

\[ \text{PL}_1: f_1 \text{ channel} - \text{high power level for } ^{13}\text{C} \]

\[ \text{transmitter pulse} \]

\[ \text{PL}_14: f_2 \text{ channel} - \text{power level for cw/hd decoupling, to be varied} \]

\[ D_1 = 2 \text{ sec} \] - relaxation delay

\[ TD = 4 \text{ K} \]

\[ O_1 = \text{on resonance of } ^{13}\text{C signal} \]

\[ NS = 1 \]

\[ \text{D}_{11} = 30 \text{ msec} \] - delay for disk I/O

\[ SW = 500 \text{ Hz} \]

\[ O_2 = 50 \text{ Hz offset from } ^1\text{H signal} \]

\[ \text{RG} = \text{receiver gain for correct ADC input} \]

Processing parameters

\[ SI = 4 \text{ K or more, use zero-filling to ensure enough data points to obtain accurate values for the residual splittings} \]

\[ BC_{\text{mod}} = \text{quad} \]

\[ WDW = \text{EM} \]

\[ FT = \text{Fourier transformation} \]

\[ \text{baseline correction} = \text{ABS} \]

Baseline correction: \[ \text{ABS} \]

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**: $f_1$ channel - high power level for $^{13}$C transmitter pulse
- **PL14**: $f_2$ 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**: $f_1$ 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 $\text{CH}_3$ 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
- **BC_mod**: quad
- **WDW**: EM
- **FT**: Fourier transformation
- **phase correction**: adjust the phase to pure absorption.
- **LB**: 0.3 Hz
- **baseline correction**: ABS

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**: $f_1$ channel – high power level for $^1$H transmitter pulse
- **PL14**: $f_2$ 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**: $f_1$ 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
- **BC_mod**: quad
- **WDW**: EM
- **LB**: 0.3 Hz
Experiment 4.5
- Homonuclear Decoupling at Two Frequencies

pulse program:

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

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: 1H  
F2: off

Acquisition parameters  
PL1 :f1 channel - high power level for 1H transmitter pulse, 3 dB was used here  
PL21 :f1 channel - low power level, here 90 dB was used (see Exp. 2.6)  
D1 :5 sec – relaxation delay  
TD :8 K  
O1 :on resonance of a chosen multiplet line of the sample  
NS :1  

Processing parameters  
SI :4 K  
WDW :EM  
FT :Fourier transformation  
baseline correction :ABS

Baseline correction:  
FT :Fourier transformation  
phase correction :adjust the phase to pure absorption.  
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, $\gamma B_2 = 1$ Hz, 90 dB was used here  
- D1 : 2 sec – relaxation delay  
- D12 : 20 usec - delay for power switching  
- TD : 0.64 K  
- O1 : middle of the $^{13}$C NMR spectrum

Acquisition parameters
- 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 2 Hz above that of the left-most line of the proton doublet at 7.5 ppm.

Processing parameters
- 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 (subtraction 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  
- D13 : 3 usec - short delay
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 subtraction. One can either transform the two spectra separately using a digitally identical phase correction and subtract the two spectra, or, more conveniently, subtract 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 (CHCl₃) 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:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Signal</th>
<th>Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>¹H</td>
<td></td>
</tr>
<tr>
<td>F2</td>
<td>¹H</td>
<td></td>
</tr>
</tbody>
</table>

Acquisition parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel – high power level for ¹H transmitter pulse</td>
</tr>
<tr>
<td>PL14</td>
<td>f2 channel – power level for presaturation (85 dB)</td>
</tr>
<tr>
<td>D1</td>
<td>0.1 sec – relaxation delay</td>
</tr>
<tr>
<td>D12</td>
<td>20 usec – delay for power switching</td>
</tr>
<tr>
<td>L4</td>
<td>Overall irradiation time: D20*L4, here 3</td>
</tr>
<tr>
<td>TD</td>
<td>:32 K</td>
</tr>
<tr>
<td>O1</td>
<td>:middle of the ¹H NMR spectrum</td>
</tr>
<tr>
<td>NS</td>
<td>:8</td>
</tr>
<tr>
<td>au-program</td>
<td>:noemult</td>
</tr>
<tr>
<td>FQ2LIST</td>
<td>:noedef.1</td>
</tr>
<tr>
<td>RG</td>
<td>:receiver gain for correct ADC input</td>
</tr>
</tbody>
</table>

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 subtraction. One can either transform the two spectra separately using a digitally identical phase correction and subtract the two spectra, or, more conveniently, subtract 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 (CHCl₃) to be positive. Evaluate only signals which have correct phase and have therefore not been affected by inadequate spectrometer stability.
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 separately using a digitally identical phase correction and subtract the two spectra, or, more conveniently, subtract the two FIDs directly from each other.

**Experiment 4.10**

- **^1H Off-Resonance Decoupled ^13C NMR Spectra**

  puls program: zg0cw
  
  compare with Experiment 4.3

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

  **Acquisition parameters**
  - PL1: f1 channel - high power level for ^13C transmitter pulse
  - PL14: f2 channel – power level for cw/hd decoupling, \( \gamma B_2 = 3500 \) Hz (see Exp. 2.6 and 4.2)
  - D1: 0.5 sec – relaxation delay
  - TD: 64 K
  - O1: middle of the ^13C NMR spectrum
  - NS: 512
  - P0: f1 channel - 45° ^13C transmitter pulse
  - D11: 30 msec - delay for disk I/O
  - SW: 200 ppm

  **Processing parameters**
  - SI: 32 K
  - WDW: EM
  - FT: Fourier transformation
  - BC_mod: quad
  - phase correction: adjust the phase to pure absorption.
  - plot: use XWINPLOT

**Experiment 4.11**

- **The Gated ^1H-Decoupling Technique**

  pulsed 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: ^13C, F2: ^1H

  **Acquisition parameters**
  - PL1: f1 channel - high power level for ^13C 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° ^13C transmitter pulse
  - PCPD2: f2 channel – 90° pulse for decoupling sequence
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:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>(^{13})C</td>
</tr>
<tr>
<td>F2</td>
<td>(^1)H</td>
</tr>
</tbody>
</table>

Acquisition parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel - high power level for (^{13})C transmitter pulse</td>
</tr>
<tr>
<td>PL12</td>
<td>f2 channel - power level for CPD decoupling</td>
</tr>
<tr>
<td>CPD2</td>
<td>WALTZ16 - CPD decoupling sequence, defined by cpdprg2</td>
</tr>
<tr>
<td>D1</td>
<td>10 sec - relaxation delay</td>
</tr>
<tr>
<td>TD</td>
<td>32 K</td>
</tr>
<tr>
<td>O1</td>
<td>middle of the (^{13})C NMR spectrum</td>
</tr>
<tr>
<td>NS</td>
<td>512</td>
</tr>
</tbody>
</table>

Processing parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI</td>
<td>32 K</td>
</tr>
<tr>
<td>WDW</td>
<td>EM (or GM is also possible)</td>
</tr>
<tr>
<td>FT</td>
<td>Fourier transformation</td>
</tr>
<tr>
<td>baseline correction</td>
<td>ABS</td>
</tr>
</tbody>
</table>

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:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>(^{13})C</td>
</tr>
<tr>
<td>F2</td>
<td>(^1)H</td>
</tr>
</tbody>
</table>
Acquisition parameters

- **PL1**: \( f1 \) channel - high power level for \(^{13}\text{C} \) transmitter pulse
- **PL14**: \( f2 \) channel - power level for \( \text{cw/hd} \) decoupling, \( \gamma B_2 = 150 \text{ Hz} \) (45 dB was used here)
- **D1**: 1 sec – relaxation delay
- **TD**: 64 K
- **O1**: middle of the \(^{13}\text{C} \) NMR spectrum
- **NS**: 8

- **P0**: \( f1 \) channel - 45° \(^{13}\text{C} \) transmitter pulse
- **D11**: 30 msec - delay for disk I/O
- **SW**: 200 ppm
- **O2**: center of methyl group \(^1\text{H} \) resonance at 1.6 ppm
- **RG**: receiver gain for correct ADC input

Processing parameters

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

Experiment 4.14

**1H Low Power Decoupling of \(^{13}\text{C} \) NMR Spectra**

- **pulse program**: zg0cw2.mo
  - 1D-sequence with CW decoupling, using \( p0 \) for any flip angle. This technique correlates an \(^1\text{H} \) signal with \(^{13}\text{C} \) signals which are separated by two, three or more bonds.

- **Setting of the needed channels**: F1: \(^{13}\text{C} \), F2: \(^1\text{H} \)

Acquisition parameters

- **PL1**: \( f1 \) channel - high power level for \(^{13}\text{C} \) transmitter pulse
- **PL12**: \( f2 \) channel - power level for CPD decoupling
- **PL14**: \( f2 \) channel - power level for decoupler attenuation during acquisition, \( \gamma B_2 = 15 \text{ Hz} \) (70 dB was used here)
- **CPD2**: WALTZ16 - CPD decoupling sequence, defined by cpdprg2
- **D1**: 1 sec – relaxation delay
- **D12**: 20 usec – delay for power switching
- **TD**: 64 K
- **O1**: middle of the \(^{13}\text{C} \) NMR spectrum
- **NS**: 8

- **P0**: \( f1 \) channel - 45° \(^{13}\text{C} \) transmitter pulse
- **PCPD2**: \( f2 \) channel – 90° pulse for decoupling sequence
- **D11**: 30 msec - delay for disk I/O
- **SW**: 200 ppm
- **O2**: center of \(^1\text{H} \) signal of the upfield olefinic proton
- **RG**: receiver gain for correct ADC input

Processing parameters

- **SI**: 32 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 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

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel - high power level for $^{13}$C transmitter pulse</td>
</tr>
<tr>
<td>PL14</td>
<td>f2 channel -- power level for cw/hd decoupling</td>
</tr>
<tr>
<td>D1</td>
<td>200 sec – relaxation delay</td>
</tr>
<tr>
<td>D12</td>
<td>20 usec – delay for power switching</td>
</tr>
<tr>
<td>TD</td>
<td>1 K</td>
</tr>
<tr>
<td>O1</td>
<td>on resonance of $^{13}$C signal of cyclohexane</td>
</tr>
<tr>
<td>Parmod</td>
<td>:2D</td>
</tr>
<tr>
<td>NS</td>
<td>8</td>
</tr>
</tbody>
</table>

Processing parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>au-program</td>
<td>:splitser, to get 1D files</td>
</tr>
<tr>
<td>SI</td>
<td>:1 K</td>
</tr>
<tr>
<td>WDW</td>
<td>:EM</td>
</tr>
<tr>
<td>FT</td>
<td>:Fourier transformation</td>
</tr>
<tr>
<td>BC_mod</td>
<td>:quad</td>
</tr>
<tr>
<td>LB</td>
<td>:3 Hz</td>
</tr>
<tr>
<td>phase correction</td>
<td>:adjust the phase of to pure absorption and use the same values for the second experiment.</td>
</tr>
<tr>
<td>integration</td>
<td>:is done manual using wmisc and rmisc, measure the two integrals and divide one by the other to obtain n+1.</td>
</tr>
<tr>
<td>plot</td>
<td>:use XWINPLOT</td>
</tr>
</tbody>
</table>
Chapter 5
- Dynamic NMR Spectroscopy

Summary

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<td>Low Temperature Calibration with Methanol</td>
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<td>5.2</td>
<td>zg0</td>
<td>High Temperature Calibration with 1,2-Ethandiol</td>
</tr>
<tr>
<td>5.3</td>
<td>zg0hd</td>
<td>Dynamic (^1)H NMR Spectroscopy on Dimethylformamid</td>
</tr>
<tr>
<td>5.4</td>
<td>zgdclo.mo</td>
<td>The Saturation Transfer Experiment</td>
</tr>
<tr>
<td>5.5</td>
<td></td>
<td>Measurement of the Rotating Frame Relaxation Time (T_1)</td>
</tr>
</tbody>
</table>

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

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
- **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 5.3
- 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
- **PL14**: f2 channel - power level for decoupler attenuation corresponding to $\gamma_B = 10$ Hz
- **D1**: 300 sec - to equilibrate temperature
- **TD**: 32 K
- **O1**: middle of the $^1$H NMR spectrum
- **digmod**: homodecoupling-digital
- **NS**: 8

- **O2**: on resonance of the aldehyde proton
- **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

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 $T_{1p}$ 

pulse program: 
The $T_{1p}$ 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 $T_{1p}$ 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

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

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 $T_1$.

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
Experiment 6.2
- Measurement of the Spin-Spin Relaxation Time $T_2$

pulse program: cpmg
1D-sequence to measure the relaxation time $T_2$ 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
D1 : 150 sec – relaxation delay
P2 : f1 channel - 180° $^1$H transmitter pulse
D11 : 30 msec - delay for disk I/O
D20 : 10 msec – fixed echo time to allow elimination of diffusion and J-mod. effects
TD : 1 K
SW : 500 Hz
O1 : on $^1$H resonance
DS : 16
NS : 8
L4 : number of experiments = number of values in vc-list (10)
Parmod : 2D
define VCLIST
TD1 : 10 – number of experiments
DE : as short as possible
RG : receiver gain for correct ADC input

Processing parameters

SI(F2) : 512 W
WDW(F2) : EM
LB(F2) : 2 Hz
XF2 : transformation in F2 direction
plot : use XWINPLOT

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 6.3**
- $^{13}$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
- PL12: f2 channel - power level for CPD decoupling
- CPD2: WALTZ16 - CPD decoupling sequence, defined by cpdprg2
- D1: 4 sec – relaxation delay
- D20: $1/|J(C,H)| = 7$ msec, calculated from $J(C,H)=140$ Hz
- TD: 64 K
- O1: middle of the $^{13}$C NMR spectrum
- NS: 16
- DE: as short as possible

**Processing parameters**
- SI: 32 K
- WDW: EM
- FT: Fourier transformation
- BC_mod: quad
- LB: 2 Hz
- 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
- PL12: f2 channel – power level for CPD
- P0: f1 channel - 45° $^{13}$C transmitter pulse
- P2: f1 channel - 180° $^{13}$C transmitter pulse
- PCPD2: f2 channel – 90° pulse for decoupling sequence
### Experiment 6.5
- The Basic INEPT Technique

**Pulse program:** ineptnd

The INEPT (Insensitive Nuclei Enhanced by Polarization Transfer) experiment increases 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:** channel - high power level for $^{13}$C transmitter pulse
- **PL2:** channel - high power level for $^1$H decoupler pulse
- **D1:** 10 sec – relaxation delay
- **TD:** 4 K
- **O1:** on resonance of $^{13}$C NMR signal
- **NS:** 1 for the first and 4 for the second experiment
- **RG:** receiver gain for correct ADC input

**Processing parameters**
- **SI:** 2 K
- **WDW:** EM
- **FT:** Fourier transformation

---

### Decoupling Sequence

<table>
<thead>
<tr>
<th>Decoupling sequence</th>
<th>Decoupling sequence</th>
</tr>
</thead>
<tbody>
<tr>
<td>D11: 30 msec - delay for disk I/O</td>
<td>D1 : 2 sec - relaxation delay</td>
</tr>
<tr>
<td>D12: 20 usec - delay for power switching</td>
<td>D20: 1/[J(C,H)] = 7 msec, calculated from $^1$J(C,H) = 140 Hz</td>
</tr>
<tr>
<td>D12: 20 usec - delay for power switching</td>
<td>D21: set equal to preacquisition delay DE</td>
</tr>
<tr>
<td>TD: 64 K</td>
<td>D21: 1/[J(C,H)] = 7 msec, calculated from $^1$J(C,H) = 212 Hz</td>
</tr>
<tr>
<td>O1: middle of the $^{13}$C NMR spectrum</td>
<td>D21: set equal to preacquisition delay DE</td>
</tr>
<tr>
<td>NS: 512</td>
<td>TD: 64 K</td>
</tr>
<tr>
<td>RG: receiver gain for correct ADC input</td>
<td>SW: 200 ppm</td>
</tr>
<tr>
<td></td>
<td>O2: middle of $^1$H NMR spectrum</td>
</tr>
<tr>
<td></td>
<td>OS: 8</td>
</tr>
<tr>
<td></td>
<td>referencing: set the TMS signal to 0 ppm</td>
</tr>
<tr>
<td></td>
<td>baseline correction: ABS</td>
</tr>
<tr>
<td></td>
<td>plot: use XWINPLOT</td>
</tr>
</tbody>
</table>

**Processing parameters**
- **SI:** 32 K
- **WDW:** EM
- **FT:** Fourier transformation
- **BC_mod:** quad
- **LB:** 2 Hz
- **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

---

**Processing parameters**
- **SI:** 32 K
- **WDW:** EM
- **FT:** Fourier transformation
- **BC_mod:** quad
- **LB:** 2 Hz
- **phase correction:** adjust the phase for the signals positive
**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:**

<table>
<thead>
<tr>
<th>Channel</th>
<th>Nucleus</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{13}\text{C}$</td>
</tr>
<tr>
<td>F2</td>
<td>$^1\text{H}$</td>
</tr>
</tbody>
</table>

**Acquisition parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>$f_1$ channel - high power level for $^{13}\text{C}$ transmitter pulse</td>
</tr>
<tr>
<td>P1</td>
<td>$f_1$ channel - 90° $^{13}\text{C}$ transmitter pulse</td>
</tr>
<tr>
<td>P2</td>
<td>$f_1$ channel - 180° $^{13}\text{C}$ transmitter pulse</td>
</tr>
<tr>
<td>P3</td>
<td>$f_2$ channel - 90° $^1\text{H}$ decoupler pulse</td>
</tr>
<tr>
<td>P4</td>
<td>$f_2$ channel - 180° $^1\text{H}$ decoupler pulse</td>
</tr>
<tr>
<td>PL2</td>
<td>$f_2$ channel - high power level for $^1\text{H}$ decoupler pulse</td>
</tr>
<tr>
<td>D1</td>
<td>2 sec – relaxation delay</td>
</tr>
<tr>
<td>D2</td>
<td>Calculated from $^1J(\text{C,H})=140$ Hz</td>
</tr>
<tr>
<td>D3</td>
<td>$0.375/[J(\text{C,H})]=2.68$ msec, calculated from $^1J(\text{C,H})=140$ Hz</td>
</tr>
<tr>
<td>D4</td>
<td>$1/[4J(\text{C,H})]=1.78$ msec, calculated from $^1J(\text{C,H})=140$ Hz</td>
</tr>
<tr>
<td>TD</td>
<td>64 K</td>
</tr>
<tr>
<td>O1</td>
<td>Middle of the $^{13}\text{C}$ NMR spectrum</td>
</tr>
<tr>
<td>O2</td>
<td>Middle of $^1\text{H}$ NMR spectrum</td>
</tr>
<tr>
<td>NS</td>
<td>128</td>
</tr>
<tr>
<td>RG</td>
<td>Receiver gain for correct ADC input</td>
</tr>
<tr>
<td>SW</td>
<td>200 ppm</td>
</tr>
<tr>
<td>SI</td>
<td>32 K</td>
</tr>
<tr>
<td>FT</td>
<td>Fourier transformation</td>
</tr>
<tr>
<td>BC_mod</td>
<td>Quad</td>
</tr>
<tr>
<td>LB</td>
<td>2 Hz</td>
</tr>
<tr>
<td>phase correction</td>
<td>Adjust the phase for the signals positive and negative</td>
</tr>
</tbody>
</table>

**Processing parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>BC_mod</td>
<td>Quad</td>
</tr>
<tr>
<td>LB</td>
<td>2 Hz</td>
</tr>
<tr>
<td>phase correction</td>
<td>Adjust the phase for the signals positive and negative</td>
</tr>
</tbody>
</table>

**Baseline correction:** ABS

**Plot:** use XWINPLOT

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**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:**

<table>
<thead>
<tr>
<th>Channel</th>
<th>Nucleus</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{13}\text{C}$</td>
</tr>
<tr>
<td>F2</td>
<td>$^1\text{H}$</td>
</tr>
</tbody>
</table>

**Acquisition parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>$f_1$ channel - high power level for $^{13}\text{C}$ transmitter pulse</td>
</tr>
<tr>
<td>P1</td>
<td>$f_1$ channel - 90° $^{13}\text{C}$ transmitter pulse</td>
</tr>
<tr>
<td>P2</td>
<td>$f_1$ channel - 180° $^{13}\text{C}$ transmitter pulse</td>
</tr>
<tr>
<td>P3</td>
<td>$f_2$ channel - 90° $^1\text{H}$ decoupler pulse</td>
</tr>
<tr>
<td>P4</td>
<td>$f_2$ channel - 180° $^1\text{H}$ decoupler pulse</td>
</tr>
<tr>
<td>PL2</td>
<td>$f_2$ channel - high power level for $^1\text{H}$ decoupler pulse</td>
</tr>
</tbody>
</table>

**Processing parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>BC_mod</td>
<td>Quad</td>
</tr>
<tr>
<td>LB</td>
<td>2 Hz</td>
</tr>
<tr>
<td>phase correction</td>
<td>Adjust the phase for the signals positive and negative</td>
</tr>
</tbody>
</table>

**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  
PL2 : f2 channel - high power level for ^13^C decoupler pulse  
D1 : 30 sec – relaxation delay  
TD : 4 K  
O1 : on resonance of ^1^H NMR signal  
NS : 8  
RG : receiver gain for correct ADC input  

Processing parameters  
SI : 2 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  

P4 : f2 channel - 180° ^1^H decoupler pulse  
PCPD2 : f2 channel – 90° pulse for decoupling sequence  
D3 : 1/[3J(C,H)]= 2.38 msec, calculated from J(C,H)=140 Hz  
D12 : 20 usec - delay for power switching  
SW : 200 ppm  
O2 : middle of ^1^H NMR spectrum  
DS : 16  

D1 : 30 sec – relaxation delay  
D4 : 1/[4J(C,H)]= 1.78 msec, calculated from 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  

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  

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 for the satellites positive and negative  
plot : use XWINPLOT
Experiment 6.9
- DEPT-135

pulse program: dept

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

Setting of the needed channels:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Nucleus</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{13}$C</td>
</tr>
<tr>
<td>F2</td>
<td>$^1$H</td>
</tr>
</tbody>
</table>

**Acquisition parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel - high power level for $^{13}$C transmitter pulse</td>
</tr>
<tr>
<td>PL2</td>
<td>f2 channel - high power level for $^1$H decoupler pulse</td>
</tr>
<tr>
<td>PL12</td>
<td>f2 channel - power level for CPD decoupling</td>
</tr>
<tr>
<td>CPD2</td>
<td>WALTZ16 - CPD decoupling sequence, defined by cpdprg2</td>
</tr>
<tr>
<td>D1</td>
<td>2 sec – relaxation delay</td>
</tr>
<tr>
<td>D12</td>
<td>20 usec – delay for power switching</td>
</tr>
<tr>
<td>TD</td>
<td>64 K</td>
</tr>
<tr>
<td>O1</td>
<td>middle of the $^{13}$C NMR spectrum</td>
</tr>
<tr>
<td>NS</td>
<td>512</td>
</tr>
<tr>
<td>RG</td>
<td>receiver gain for correct ADC input</td>
</tr>
<tr>
<td>P0</td>
<td>f1 channel - 135° $^{13}$C transmitter pulse</td>
</tr>
<tr>
<td>P1</td>
<td>f1 channel - 90° $^{13}$C transmitter pulse</td>
</tr>
<tr>
<td>P2</td>
<td>f1 channel - 180° $^{13}$C transmitter pulse</td>
</tr>
<tr>
<td>P3</td>
<td>f2 channel - 90° $^1$H decoupler pulse</td>
</tr>
<tr>
<td>P4</td>
<td>f2 channel - 180° $^1$H decoupler pulse</td>
</tr>
<tr>
<td>PCD2</td>
<td>f2 channel – 90° pulse for decoupling sequence</td>
</tr>
<tr>
<td>D2</td>
<td>1/[2J(C,H)] = 3.5 msec, calculated from $^1$J(C,H) = 140 Hz</td>
</tr>
<tr>
<td>SW</td>
<td>200 ppm</td>
</tr>
<tr>
<td>O2</td>
<td>middle of $^1$H NMR spectrum</td>
</tr>
<tr>
<td>DS</td>
<td>8</td>
</tr>
</tbody>
</table>

**Processing parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI</td>
<td>32 K</td>
</tr>
<tr>
<td>WDW</td>
<td>EM</td>
</tr>
<tr>
<td>FT</td>
<td>Fourier transformation</td>
</tr>
<tr>
<td>BC_mod</td>
<td>quad</td>
</tr>
<tr>
<td>LB</td>
<td>2 Hz</td>
</tr>
<tr>
<td>phase correction</td>
<td>adjust the phase for the TMS signal positive</td>
</tr>
<tr>
<td>plot</td>
<td>use XWINPLOT</td>
</tr>
</tbody>
</table>

Experiment 6.10
- Editing $^{13}$C NMR Spectra with DEPT

pulse program: dept

compare with Experiment 6.9

Setting of the needed channels:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Nucleus</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{13}$C</td>
</tr>
<tr>
<td>F2</td>
<td>$^1$H</td>
</tr>
</tbody>
</table>

**Acquisition parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel - high power level for $^{13}$C transmitter pulse</td>
</tr>
<tr>
<td>PL2</td>
<td>f2 channel - high power level for $^1$H decoupler pulse</td>
</tr>
<tr>
<td>P1</td>
<td>f1 channel - 90° $^{13}$C transmitter pulse</td>
</tr>
<tr>
<td>P2</td>
<td>f1 channel - 180° $^{13}$C transmitter pulse</td>
</tr>
<tr>
<td>P3</td>
<td>f2 channel - 90° $^1$H decoupler pulse</td>
</tr>
<tr>
<td>P4</td>
<td>f2 channel - 180° $^1$H decoupler pulse</td>
</tr>
</tbody>
</table>
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, \( \text{CH}_2 \), \( \text{CH}_3 \) and quaternary carbons with the same sensitivity like DEPT.

Setting of the needed channels:

- **F1**: \( ^{13}\text{C} \)
- **F2**: \( ^{1}\text{H} \)

**Acquisition parameters**

- **PL1**: \( f1 \) channel - high power level for \( ^{13}\text{C} \) transmitter pulse
- **PL2**: \( f2 \) channel - high power level for \( ^{1}\text{H} \) decoupler pulse
- **PL12**: \( f2 \) channel - power level for CPD decoupling
- **CPD2**: WALTZ16 - CPD decoupling sequence, defined by cpdprg2

Processing parameters

- **PL1**: \( f1 \) channel - high power level for \( ^{13}\text{C} \) transmitter pulse
- **PL2**: \( f2 \) channel - high power level for \( ^{1}\text{H} \) decoupler pulse
- **PL12**: \( f2 \) channel - power level for CPD decoupling
- **PCPD2**: \( f2 \) channel – 90° pulse for decoupling sequence

- **P0**: \( f1 \) channel – use 45°, 90° and 135° \( ^{1}\text{H} \) decoupler pulse for three successive spectra a, b and c. Spectrum a will give the signals of \( \text{CH} \), \( \text{CH}_2 \) and \( \text{CH}_3 \) groups positive; b gives only the signals of \( \text{CH} \) groups, and c gives the signals of \( \text{CH} \) and \( \text{CH}_3 \) groups positive and the signals of \( \text{CH}_2 \) 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
- **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 \( ^{13}\text{C} \) NMR spectrum
- **NS**: 512
- **RG**: receiver gain for correct ADC input
- **O2**: middle of \( ^{1}\text{H} \) NMR spectrum
- **SW**: 200 ppm
- **DS**: 8

Baseline correction: ABS

**Plot**=
use XWINPLOT

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 \( \text{CH}_3 \) groups positive and the \( \text{CH}_2 \) groups negative. For editing purposes the three spectra have to be further manipulated. This is done in the dual mode. Subtraction of b from a yields spectrum d, where the signals of \( \text{CH} \) and \( \text{CH}_3 \) groups both remain positive. Subtraction of b from c yields spectrum e, where the signals of \( \text{CH}_3 \) groups are negative and those of the \( \text{CH}_2 \) groups remain positive. Subtraction of e from d yields f with only signals of \( \text{CH}_2 \) groups, whereas addition of e to d yields spectrum g with only signals of the \( \text{CH}_3 \) groups.
sequence, defined by cpdprg2

**D1**: 2 sec – relaxation delay

**D12**: 20 usec – delay for power switching

**TD**: 64 K

**O1**: middle of $^{13}$C NMR spectrum

**NS**: 16

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

**D4**: $1/(4J(C,H)) = 1.72$ msec, calculated from $1J(C,H) = 145$ Hz

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

**SW**: 250 ppm

**O2**: middle of $^1$H NMR spectrum

**DS**: 8

**DE**: as short as possible

**Processing parameters**

**SI**: 32 K

**WDW**: EM

**FT**: Fourier transformation

**BC_mod**: quad

**LB**: 2 Hz

**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

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**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**: $f_1$ channel - high power level for $^{13}$C transmitter pulse

**PL12**: $f_2$ channel - power level for CPD decoupling

**CPD2**: WALTZ16 - CPD decoupling sequence, defined by cpdprg2

**D1**: 3 sec – relaxation delay

**D11**: 30 msec – delay for disk I/O

**TD**: 32 K

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

**NS**: 512

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

**P1**: $f_1$ channel - 90° $^{13}$C transmitter pulse

**P2**: $f_1$ channel - 180° $^{13}$C transmitter pulse

**PCPD2**: $f_2$ channel – 90° pulse for decoupling sequence

**D4**: $1/([4J(C,C)]= 7.6$ msec, calculated from $1J(C,C)=33$ Hz

**D13**: 3 usec - short delay

**SW**: 60 ppm (spectral range for C$_6$H$_5$OH)

**O2**: middle of $^1$H NMR spectrum

**DS**: 16

**Processing parameters**

**SI**: 64 K

**WDW**: EM

**FT**: Fourier transformation

**baseline correction**: ABS

**BC_mod**: quad

**LB**: 0.5 Hz

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

**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 $^{13}$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
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 : 60 sec – relaxation delay
D2 : 1/[2J(C,H)] = 2.38 msec, calculated from $^1$J(C,H)=214 Hz
D7 : 20 sec – to be varied
D13 : 3 usec - short delay
TD : 4 K
O1 : on $^1$H resonance
O2 : on $^{13}$C resonance
NS : 4
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 : correct the satellites positive
plot : use XWINPLOT
baseline correction : ABS

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 $^{12}$C and the protons bond 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
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
Experiment 6.15
- The Heteronuclear Double Quantum Filter

pulse program: inv4nd1d
This experiment suppress the main signal of protons attached to $^{13}$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
PL2: f2 channel - high power level for $^{13}$C decoupler pulse
D1: 200 sec – relaxation delay
D12: 20 usec – delay for power switching
D2: $1/[2J(C,H)]=2.38$ msec, calculated from $^1$J(C,H)=214 Hz
D13: 3 usec
TD: 4 K
O1: on $^1$H resonance
O2: on $^{13}$C resonance
NS: 8
RG: receiver gain for correct ADC input

Processing parameters
SI: 2 K
WDW: EM
FT: Fourier transformation
baseline correction: ABS

bc: 2 K
BC_mod: quad
LB: 1 Hz
phase correction: correct the satellites positive
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**

<table>
<thead>
<tr>
<th>PL1</th>
<th>f1 channel - high power level for (^1)H transmitter pulse, 3 dB was used here</th>
</tr>
</thead>
<tbody>
<tr>
<td>P1</td>
<td>f1 channel - 90° (^1)H transmitter pulse</td>
</tr>
<tr>
<td>P2</td>
<td>f1 channel - 180° (^1)H transmitter pulse</td>
</tr>
<tr>
<td>P28</td>
<td>f1 channel - spin-lock pulse, 2 msec, be sure, that your power is not too high! The transmitter pulse with power level pl1 should be not shorter than 6 usec.</td>
</tr>
<tr>
<td>P4</td>
<td>f2 channel - 180° (^{13})C decoupler pulse</td>
</tr>
<tr>
<td>PL2</td>
<td>f2 channel - high power level for (^{13})C decoupler pulse</td>
</tr>
<tr>
<td>D1</td>
<td>10 sec – relaxation delay</td>
</tr>
<tr>
<td>TD</td>
<td>0.4 K</td>
</tr>
<tr>
<td>O1</td>
<td>on (^1)H resonance</td>
</tr>
<tr>
<td>NS</td>
<td>1</td>
</tr>
<tr>
<td>RG</td>
<td>receiver gain for correct ADC input</td>
</tr>
</tbody>
</table>

**Processing parameters**

<table>
<thead>
<tr>
<th>SI</th>
<th>2 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>WDW</td>
<td>EM</td>
</tr>
<tr>
<td>FT</td>
<td>Fourier transformation</td>
</tr>
<tr>
<td>SI</td>
<td>16 K</td>
</tr>
<tr>
<td>BC_mod</td>
<td>quad</td>
</tr>
<tr>
<td>LB</td>
<td>0.1 Hz</td>
</tr>
<tr>
<td>phase correction</td>
<td>correct the satellites up and down</td>
</tr>
<tr>
<td>plot</td>
<td>use XWINPLOT</td>
</tr>
</tbody>
</table>

**Experiment 6.17**

- Water Suppression by Presaturation

puls 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**

<table>
<thead>
<tr>
<th>PL1</th>
<th>f1 channel - high power level for (^1)H transmitter pulse</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL9</td>
<td>f1 channel - power level for presaturation</td>
</tr>
<tr>
<td>D1</td>
<td>2 sec – relaxation delay</td>
</tr>
<tr>
<td>D13</td>
<td>3 usec – short delay</td>
</tr>
<tr>
<td>TD</td>
<td>32 K</td>
</tr>
<tr>
<td>O1</td>
<td>on resonance of water signal</td>
</tr>
<tr>
<td>NS</td>
<td>8</td>
</tr>
<tr>
<td>for inverse probeheads: spinner off</td>
<td></td>
</tr>
</tbody>
</table>

| P1   | f1 channel - 90° \(^1\)H transmitter pulse |
| P12  | 20 usec - delay for power switching |
| SW   | 0 ppm |
| DS   | 2 |
| RG   | receiver gain for correct ADC input |

**Processing parameters**

<table>
<thead>
<tr>
<th>SI</th>
<th>16 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>BC_mod</td>
<td>quad</td>
</tr>
</tbody>
</table>
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

Processing parameters
SI : 16 K
WDW : EM
FT : Fourier transformation

baseline correction : ABS
plot : use XWINPLOT

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

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

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<th>Description</th>
</tr>
</thead>
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<td>Determination of a Shaped 90° (^1)H Transmitter Pulse</td>
</tr>
<tr>
<td>7.2</td>
<td>decp90sp.mo</td>
<td>Determination of a Shaped 90° (^1)H Decoupler Pulse</td>
</tr>
<tr>
<td>7.3</td>
<td>decp90sp.mo</td>
<td>Determination of a Shaped 90° (^13)C Decoupler Pulse</td>
</tr>
<tr>
<td>7.4</td>
<td>dante.mo</td>
<td>Selective Excitation with DANTE</td>
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<td>selco</td>
<td>SELCOSY</td>
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<td>7.6</td>
<td>selincor.mo</td>
<td>SELINCOR: Selective Inverse H,C Correlation via (^1)J (C,H)</td>
</tr>
<tr>
<td>7.7</td>
<td>selina</td>
<td>SELINQUATE</td>
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<td>Selective TOCSY</td>
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<tr>
<td>7.9</td>
<td>selinapt.mo</td>
<td>INAPT</td>
</tr>
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<td>7.10</td>
<td>sellr.mo</td>
<td>Determination of Long-Range C,H Coupling Constants</td>
</tr>
<tr>
<td>7.11</td>
<td>selreso.mo</td>
<td>SELRESOLV</td>
</tr>
<tr>
<td>7.12</td>
<td>serf.mo</td>
<td>SERF</td>
</tr>
</tbody>
</table>

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
- **G**: 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
**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° $^1$H decoupler pulse duration.

Setting of the needed channels:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{13}$C</td>
</tr>
<tr>
<td>F2</td>
<td>$^1$H</td>
</tr>
</tbody>
</table>

**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**: f2 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
- **P2**: f1 channel - 90° $^1$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° $^1$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 J(C,H)=212 Hz
- **SW**: 500 Hz
- **O2**: on $^1$H resonance
- **NS**: Gaussian shape with 1024 data points was used

**Processing parameters**

- **SI**: 2 K
- **WDW**: EM
- **FT**: Fourier transformation

- **BC_mod**: quad
- **LB**: 2 Hz

**baseline correction**: ABS

**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:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^1$H</td>
</tr>
<tr>
<td>F2</td>
<td>$^{13}$C</td>
</tr>
</tbody>
</table>

**Acquisition parameters**

- **PL1**: f1 channel - high power level for $^1$H transmitter pulse
- **P1**: f1 channel - 90° $^1$H transmitter pulse
**Experiment 7.4**

- **Selective Excitation with DANTE**

  **puls 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
  - **phase correction:** adjust the satellites up and down
  - **baseline correction:** ABS
  - **BC.mod:** quad
  - **LB:** 0.1 Hz
  - **plot:** use XWINPLOT

  **D1:** 10 sec – relaxation delay
  **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

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

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

  **L4:** number of pulse P0, here 50 was used

  **SW:** 10 ppm

  **O2:** middle of $^1$H NMR spectrum

  **NS:** 128
**Processing parameters**

<table>
<thead>
<tr>
<th>S1</th>
<th>:2 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>WDW</td>
<td>:EM</td>
</tr>
<tr>
<td>FT</td>
<td>:Fourier transformation</td>
</tr>
<tr>
<td>baseline correction</td>
<td>:ABS</td>
</tr>
<tr>
<td>BC_mod</td>
<td>:quad</td>
</tr>
<tr>
<td>LB</td>
<td>:0.5 Hz</td>
</tr>
</tbody>
</table>

**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:

<table>
<thead>
<tr>
<th>F1</th>
<th>1H</th>
</tr>
</thead>
<tbody>
<tr>
<td>F2</td>
<td>off</td>
</tr>
</tbody>
</table>

**Acquisition parameters**

<table>
<thead>
<tr>
<th>PL1</th>
<th>:f1 channel - high power level for 1H transmitter pulse</th>
</tr>
</thead>
<tbody>
<tr>
<td>SP1</td>
<td>:f1 channel - power level for shaped pulse</td>
</tr>
<tr>
<td>D1</td>
<td>:2 sec - relaxation delay</td>
</tr>
<tr>
<td>D14</td>
<td>:3 usec - short delay</td>
</tr>
<tr>
<td>TD</td>
<td>:32 K</td>
</tr>
<tr>
<td>O1</td>
<td>:on resonance of selected signal or use SPOFFS</td>
</tr>
<tr>
<td>NS</td>
<td>:16</td>
</tr>
<tr>
<td>RG</td>
<td>:receiver gain for correct ADC input</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>P1</th>
<th>:f1 channel - 90° 1H transmitter pulse</th>
</tr>
</thead>
<tbody>
<tr>
<td>P11</td>
<td>:f1 channel – 90° shaped pulse, 50 msec was used here</td>
</tr>
<tr>
<td>D13</td>
<td>:3 usec - short delay</td>
</tr>
<tr>
<td>SW</td>
<td>:10 ppm</td>
</tr>
<tr>
<td>DS</td>
<td>:4</td>
</tr>
</tbody>
</table>

**Processing parameters**

<table>
<thead>
<tr>
<th>S1</th>
<th>:16 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>WDW</td>
<td>:EM</td>
</tr>
<tr>
<td>FT</td>
<td>:Fourier transformation</td>
</tr>
<tr>
<td>BC_mod</td>
<td>:quad</td>
</tr>
<tr>
<td>LB</td>
<td>:0.1 Hz</td>
</tr>
</tbody>
</table>

**phase correction** : note that the selective COSY produces antiphase signals of the active coupling partners

**plot** : use XWINPLOT

---

**Experiment 7.6**

- SELINCOR: Selective Inverse H,C Correlation via 1J (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:

<table>
<thead>
<tr>
<th>F1</th>
<th>1H</th>
</tr>
</thead>
<tbody>
<tr>
<td>F2</td>
<td>13C</td>
</tr>
</tbody>
</table>
Acquisition parameters

**PL0** : f2 channel - 120 dB fixed power level

**PL1** : f1 channel - high power level for \(^1\)H transmitter pulse

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

**SP2** : f2 channel - power level for shaped pulse

**D1** : 1 sec – relaxation delay

**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

**TD** : 32 K

**O1** : middle of \(^1\)H NMR signal

**O2** : on resonance of selected \(^{13}\)C nucleus

**NS** : 32

Gaussian shape with 1024 data points was used

**PL12** : f2 channel - power level for CPD decoupling

**PCPD2** : f2 channel - 90° pulse for decoupling sequence

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

**SP1** : f1 channel - power level for shaped pulse

**PL12** : f2 channel - power level for CPD decoupling

**P1** : f1 channel - 90° \(^{13}\)C transmitter pulse

**P2** : f1 channel - 180° \(^{13}\)C transmitter pulse

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

**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 1J(C,C)=33 Hz, for long range couplings J(C,C)=4 Hz= 62.5 msec

D11: 30 msec - delay for disk I/O
D13: 3 usec - short delay

D1: 4 sec – relaxation delay
D4: 1/[4J(C,C)]= 7.6 msec, calculated from 1J(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
PHCORA: difference of phases between power level SP1 and PL1
RG: receiver gain for correct ADC input

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: 1H
F2: off

Acquisition parameters
PL0: f1 channel - 120 dB, fixed power level
PL1: f1 channel - high power level for 1H 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° 1H 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]
D11: 30 msec - delay for disk I/O
D14: delay for evolution after shaped pulse: for self-refocussing pulse 20 usec

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

O1: on resonance of selected signal or use SPOFFS

PHCOR1: difference in phases between power level SP1 and PL10

Processing parameters

<table>
<thead>
<tr>
<th>SI</th>
<th>16 K</th>
</tr>
</thead>
<tbody>
<tr>
<td>WDW</td>
<td>EM</td>
</tr>
<tr>
<td>FT</td>
<td>Fourier transformation</td>
</tr>
<tr>
<td>baseline correction</td>
<td>ABS</td>
</tr>
<tr>
<td>BC_mod</td>
<td>quad</td>
</tr>
<tr>
<td>LB</td>
<td>0.1 Hz</td>
</tr>
<tr>
<td>phase correction</td>
<td>adjust the phase to pure absorption.</td>
</tr>
<tr>
<td>plot</td>
<td>use XWINPLOT</td>
</tr>
</tbody>
</table>

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 |
| SP2 | f2 channel - power level for shaped pulse, here 67 dB was used |
| PL12 | f2 channel - power level for CPD decoupling |
| CPD2 | WALTZ16 - CPD decoupling sequence, defined by cpdp2rg |
| D1 | 3 sec - relaxation delay |
| D21 | 20 msec – fixed delay |
| TD | 32 K |
| O1 | center of aromatic region of the $^{13}$C NMR spectrum |

| P1 | f1 channel - 90° $^{13}$C transmitter pulse |
| P2 | f1 channel - 180° $^{13}$C transmitter pulse |
| P13 | f2 channel - 90° rectangular shaped $^1$H decoupler pulse, here 20 msec |
| P14 | f2 channel - 180° rectangular shaped $^1$H decoupler pulse |
| PCPD2 | f2 channel - 90° pulse for decoupling sequence |
| D20 | 10 msec - fixed delay |
| SW | 55 ppm |
| 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 |
### 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
- **SP2**: f2 channel - power level for shaped RE-BURP pulse, 46 dB was used
- **PL12**: f2 channel - power level for CPD decoupling
- **CPD2**: WALTZ16 - CPD decoupling sequence, defined by cpdprg2
- **D0**: 3 usec – incremented delay
- **D11**: 30 msec – delay for disk I/O
- **Parmod**: 2D
- **TD2**: 1 K data points in F2
- **SW2**: 200 ppm
- **O1**: middle of the $^{13}$C NMR spectrum
- **NS**: 8
- **IN0**: $1/[2*SW1]
- **RG**: receiver gain for correct ADC input
- **P1**: f1 channel - 90° $^{13}$C transmitter pulse
- **P2**: f1 channel - 180° $^{13}$C transmitter pulse
- **P14**: f2 channel - 180° shaped pulse, 40 msec was used here
- **PCPD2**: f2 channel - 90° pulse for decoupling sequence
- **D1**: 2 sec - relaxation delay
- **D12**: 20 usec - delay for power switching
- **ND0**: 2
- **TD1**: 128 data points in F1
- **SW1**: 50 Hz
- **O2**: on resonance of the methyl group at 1.8 ppm
- **DS**: 16
- **DE**: as small as possible

**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
- **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 $^J(C,H)=10$ 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  
- **INO**: $1/[2^SW1]$  
- **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  
- **plot**: use XWINPLOT

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: ¹H  
F2: off

Acquisition parameters

- **PL1**: f1 channel - high power level for ¹H 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
- **D0**: 3 usec – incremented delay
- **D12**: 20 usec - delay for power switching
- **Parmod**: 2D
- **TD2**: 1 K data points in F2
- **SW2**: 8 ppm
- **O1**: middle of ¹H NMR spectrum
- **NS**: 4
- **INO**: 1/(2*SW1)
- **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*

- **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
- **D1**: 2 sec – relaxation delay
- **ND0**: 2
- **TD1**: 64 data points in F1
- **SW1**: 50 Hz
- **DS**: 16
- **DE**: as small as possible

  *the E-BURP2 shape acts on the olefinic signal at 6.9 ppm*

Processing parameters

- **SI(F2)**: 512 W
- **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
Chapter 8
- Auxiliary Reagents, Quantitative Determinations and Reaction Mechanism

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Experiment 8.1
- Signal Separation Using a Lanthanide Shift Reagent

pulse program: zg30
compare with Experiment 3.1
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  
O1: middle of the $^1$H NMR spectrum  
NS :8  
SW :15 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 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  
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  
  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
**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
- **O1**: middle of the $^1$H NMR spectrum
- **NS**: 8
- **SW**: 15 ppm
- **RG**: receiver gain for correct ADC input

**Processing parameters**

- **SI**: 16 K
- **WDW**: EM
- **FT**: Fourier transformation
- **BC_mod**: quad
- **LB**: 0.1 Hz
- **phase correction**: adjust the phase to pure absorption.
- **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
- **O1**: middle of the $^1$H NMR spectrum
- **NS**: 8
- **SW**: 15 ppm
- **RG**: receiver gain for correct ADC input

**Processing parameters**

- **SI**: 16 K
- **WDW**: GM
- **LB**: -0.5 Hz
- **FT**: Fourier transformation
- **BC_mod**: quad
- **GB**: 0.2
- **phase correction**: adjust the phase to pure absorption.
- **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 transmitter pulse
- **PO**: f1 channel - 30° $^{13}$C transmitter pulse
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:  "H  
F2: off

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

Processing parameters  
process both FIDs with the same parameters
SI: 16 K  
WDW: EM  
FT: Fourier transformation  
baseline correction: ABS
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
- **O1**: middle of $^1$H NMR spectrum
- **NS**: 8
- **SW**: 20 ppm
- **RG**: receiver gain for correct ADC input

**Processing parameters**
- **SI**: 16 K
- **WDW**: EM
- **FT**: Fourier transformation
- **BC_mod**: quad
- **LB**: 0.1 Hz
- **phase correction**: adjust the phase to pure absorption.
- **referencing**: reference both spectra to $\delta H=0$ 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$_2$O, 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
- **O1**: middle of $^1$H NMR spectrum
- **NS**: 8
- **SW**: 20 ppm
- **RG**: receiver gain for correct ADC input

**Processing parameters**
- **SI**: 16 K
- **WDW**: EM
- **FT**: Fourier transformation
- **BC_mod**: quad
- **LB**: 0.1 Hz
- **phase correction**: adjust the phase to pure absorption.
- **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  
deuterated 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  
O1 : middle of the $^{13}$C NMR spectrum  
O2 : middle of $^1$H NMR spectrum

D11 : 30 msec - delay for disk I/O  
SW : 200 ppm

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  
first measure mixture of the deuterated solvents alone, then add the mixture of the  
deuterated 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 : 2 sec – relaxation delay  
D11 : 30 msec - delay for disk I/O

TD : 64 K  
O1 : middle of the $^{13}$C NMR spectrum  
O2 : middle of $^1$H NMR spectrum

D11 : 30 msec - delay for disk I/O  
SW : 200 ppm

NS : 32  
RG : receiver gain for correct ADC input
**Processing parameters**

SI: 32 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 8.11**

- The Relaxation Reagent Cr(acac)₃

**Pulse program:** zg0dc

Compare with Experiment 2.2

**Setting of the needed channels:**

F1: ¹³C
F2: ¹H

---

**Acquisition parameters**

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

P0: f1 channel - 45º ¹³C transmitter pulse

PL12: f2 channel - power level for CPD decoupling

PCPD2: f2 channel –90º 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

O1: middle of the ¹³C NMR spectrum

O2: middle of ¹H NMR spectrum

NS: 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: 2 Hz
Phase correction: adjust the phase to pure absorption.
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: ¹H
F2: off

---

**Acquisition parameters**

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

P1: f1 channel - 90º ¹H transmitter pulse

D1: 0.1 sec – relaxation delay

TD: 32 K

O1: middle of the ¹H NMR spectrum

NS: 8

SW: 15 ppm

RG: receiver gain for correct ADC input
### Processing parameters

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<th>Parameter</th>
<th>Value</th>
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<td>SI</td>
<td>16 K</td>
</tr>
<tr>
<td>WDW</td>
<td>EM</td>
</tr>
<tr>
<td>FT</td>
<td>Fourier transformation</td>
</tr>
<tr>
<td>baseline correction</td>
<td>ABS</td>
</tr>
</tbody>
</table>

### BC_mod

- quad

### LB

- 0.1 Hz

### phase correction

- adjust the phase to pure absorption.

### FT

- Fourier transformation

### plot

- use XWINPLOT

### Experiment 8.13

- 1H and 13C NMR of Paramagnetic Compounds

#### a) pulse program: zg0

- compare with Experiment 2.1

##### Setting of the needed channels:

- F1: 1H
- F2: off

##### Acquisition parameters

<table>
<thead>
<tr>
<th>PL1</th>
<th>channel - high power level for 1H transmitter pulse, here 3 dB was used</th>
</tr>
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<tr>
<td>D1</td>
<td>0.1 sec – relaxation delay</td>
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<tr>
<td>TD</td>
<td>64 K</td>
</tr>
<tr>
<td>O1</td>
<td>25 ppm to lower frequencies from TMS signal</td>
</tr>
<tr>
<td>NS</td>
<td>8</td>
</tr>
</tbody>
</table>

| P0 | channel - 45° 1H transmitter pulse |
| SW | 110 pm |

##### 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: 13C
- F2: 1H

##### Acquisition parameters

<table>
<thead>
<tr>
<th>PL1</th>
<th>channel - high power level for 13C transmitter pulse</th>
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<td>CPD2</td>
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<tr>
<td>D1</td>
<td>0.1 sec – relaxation delay</td>
</tr>
<tr>
<td>TD</td>
<td>64 K</td>
</tr>
<tr>
<td>O1</td>
<td>400 ppm to higher frequencies from TMS signal</td>
</tr>
<tr>
<td>NS</td>
<td>2048</td>
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</table>

| P0 | channel - 45° 13C transmitter pulse |
| PCPD2 | channel –90° pulse for decoupling sequence |
| SW | 990 ppm |
| O2 | on resonance of the previously determined 1H NMR frequency of the cobaltocene signal |
| RG | receiver gain for correct ADC input |
Processing parameters

SI :16 K  
WWD :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 8.14
- 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  
P1 :f1 channel - 90° $^{13}$C transmitter pulse  
PCPD2 :f2 channel –90° pulse for decoupling sequence

PL12 :f2 channel – power level for CPD decoupling  
CPD2 :WALTZ16 – CPD decoupling sequence, defined by cpdprg2  
D1 :1 sec – relaxation delay  
D11 :30 msec - delay for disk I/O

TD :32 K  
O1 :middle of the $^{13}$C NMR spectrum
O2 :middle of $^1$H NMR spectrum
NS :16  
prepare with edc 20 data sets and use the au-program multizg to measure the 20 spectra

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  
WWD :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  
P0 :f1 channel - 45° $^1$H transmitter pulse

D1 :5 sec – relaxation delay  
TD :32 K or more (use a large data set to

SW :10 ppm
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  
PL12 : f2 channel - power level for CPD decoupling  
CPD2 : WALTZ16 - CPD decoupling sequence, defined by cpdprg2  
D1 : 10 sec - relaxation delay  
TD : 2 K (short aq to avoid NOE build-up during acquisition)  
O1 : middle of aromatic region of the $^{13}$C NMR spectrum  
NS : 160

Processing parameters
SI : 64 K  
WDW : EM  
FT : Fourier transformation  
integration : manual  
plot : use XWINPLOT

Experiment 8.17
- NMR Using Liquid-Crystal Solvents

pulse program: zg30
compare with Experiment 3.1

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  
WDW : EM  
FT : Fourier transformation  
integration : manual  
plot : use XWINPLOT

BC_mod : quad  
LB : 0.1 Hz  
phase correction : adjust the phase to pure absorption.

baseline correction : ABS
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**: 64 K
- **O1**: on resonance of the $^1$H benzene signal in isotropic phase
- **NS**: 8

- **P1**: f1 channel - 90° $^1$H transmitter pulse
- **D11**: 30 msec - delay for disk I/O
- **SW**: 0.2 ppm
- **DE**: 100 usec, to avoid break through of the matrix signal

- **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
- **WDW**: EM
- **FT**: Fourier transformation
- **baseline correction**: ABS

- **BC_mod**: quad
- **LB**: 0.3 Hz
- **phase correction**: adjust the phase to pure absorption

- **plot**: use XWINPLOT
Chapter 9
- Heteronuclear NMR Spectroscopy

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<td>9.7</td>
<td>zgdc</td>
<td>$^1$H NMR Spectroscopy</td>
</tr>
<tr>
<td>9.8</td>
<td>zgdc</td>
<td>$^11$B NMR Spectroscopy</td>
</tr>
<tr>
<td>9.9</td>
<td>aring2.mo</td>
<td>$^{17}$O NMR Spectroscopy with RIDE</td>
</tr>
<tr>
<td>9.10</td>
<td>zg</td>
<td>$^{47/49}$Ti NMR Spectroscopy with ARING</td>
</tr>
</tbody>
</table>

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$_3$NO$_2$ (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
**Processing parameters**

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

**baseline correction**: ABS

**Experiment 9.2**

- H-Coupled 15N NMR Spectra with DEPT

*Pulse program:* deptnd

DEPT experiment without decoupling.

Setting of the needed channels:

- F1: 15N
- F2: 1H

**Acquisition parameters**

- **PL1**: f1 channel - high power level for 15N transmitter pulse
- **PL2**: f2 channel - high power level for 1H decoupler pulse
- **P1**: f1 channel - 90° 15N transmitter pulse
- **P2**: f1 channel - 180° 15N transmitter pulse
- **P0**: f1 channel - 45° 1H decoupler pulse
- **P3**: f2 channel - 90° 1H decoupler pulse
- **P4**: f2 channel - 180° 1H decoupler pulse
- **D1**: 2 sec – relaxation delay
- **D2**: 1/[2J(N,H)] = 5.6 msec, calculated from 1J(N,H) = 90 Hz
- **TD**: 32 K
- **O1**: 220 ppm upfield from CH3NO2
- **NS**: 32
- **RG**: receiver gain for correct ADC input
- **SW**: 350 ppm
- **O2**: middle of 1H NMR spectrum
- **DS**: 8

**Processing parameters**

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

**baseline correction**: ABS

**Experiment 9.3**

- 19F NMR Spectroscopy

*Pulse program:* zg30

compare with Experiment 3.1

Setting of the needed channels:

- F1: 19F
- F2: off

**Acquisition parameters**

- **PL1**: f1 channel - high power level for 19F transmitter pulse
- **P1**: f1 channel - 90° 19F transmitter pulse
- **P0**: f1 channel - 45° 1H decoupler pulse
- **P3**: f2 channel - 90° 1H decoupler pulse
- **P4**: f2 channel - 180° 1H decoupler pulse
- **D2**: 1/[2J(N,H)] = 5.6 msec, calculated from 1J(N,H) = 90 Hz
- **TD**: 32 K
- **O1**: 220 ppm upfield from CH3NO2
- **NS**: 32
- **RG**: receiver gain for correct ADC input
- **SW**: 350 ppm
- **O2**: middle of 1H NMR spectrum
- **DS**: 8

**Processing parameters**

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

**baseline correction**: ABS
transmitter pulse
D1 : 1 sec – relaxation delay
TD : 64 K

O1 : about 100 ppm upfield from CCl₃F
(center of that range)
NS : 1

SW : 300 ppm (typical range for fluorine
bound to carbon)

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
- ²⁹Si NMR Spectroscopy with DEPT

pulse program: dept
compare with Experiment 6.9

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

Acquisition parameters
PL1 : f1 channel - high power level for ²⁹Si
transmitter pulse

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

PL12 : f2 channel - power level for CPD
decoupling

CPD2 : WALTZ16 - CPD decoupling
sequence, defined by cpdprg2

D1 : 1 sec – relaxation delay
TD : 64 K

O1 : 70 ppm upfield from ²⁹Si signal of TMS
NS : 32

P1 : f1 channel - 90° ²⁹Si transmitter pulse
P2 : f1 channel - 180° ²⁹Si transmitter pulse
P0 : f2 channel - 16.8° ¹H decoupler pulse
corresponding to the 12 equivalent
protons of the sample
P3 : f2 channel - 90° ¹H decoupler pulse
P4 : f2 channel - 180° ¹H decoupler pulse
PCPD2 : f2 channel - 90° pulse for
decoupling sequence

D2 : 1/[(2J(Si,H))]= 0.07 sec, calculated from
²J(Si,H)=7 Hz

SW : 250 ppm
O2 : middle of ¹H NMR spectrum
DS : 64

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 demonstrated 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:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Substance</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{29}$Si</td>
</tr>
<tr>
<td>F2</td>
<td>$^1$H</td>
</tr>
</tbody>
</table>

Acquisition parameters

| PL2     | $f_2$ channel - high power level for $^1$H decoupler pulse, here 3 dB was used |
| PL10    | $f_1$ channel - low power level                                        |
| PL15    | $f_2$ channel - low power level                                        |
| PL12    | $f_2$ 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 $^2J(Si,H)= 7$ Hz |
| NS      | 1                                                                      |
| P3      | $f_2$ channel - 90° $^1$H decoupler pulse                               |
| P6      | $f_1$ channel - 90° $^{29}$Si transmitter pulse, 50 usec was used        |
| P9      | $f_2$ channel - 90° $^1$H decoupler pulse, 50 usec was used             |
| PCD2    | $f_2$ channel - 90° pulse for decoupling sequence, here 100 usec was used |

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                                                            |
| RG      | receiver gain for correct ADC input                                    |

Experiment 9.6

$^{119}$Sn NMR Spectroscopy

pulse program: zgdc30

compare with Experiment 3.2

Setting of the needed channels:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Substance</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{119}$Sn</td>
</tr>
<tr>
<td>F2</td>
<td>$^1$H</td>
</tr>
</tbody>
</table>

Acquisition parameters

| PL1     | $f_1$ channel - high power level for $^{119}$Sn transmitter pulse      |
| PL12    | $f_2$ channel – power level for CPD decoupling                          |
| P1      | $f_1$ channel - 90° $^{119}$Sn transmitter pulse                        |
| PCD2    | $f_2$ channel - 90° pulse for decoupling sequence                       |
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
PL12: f2 channel - power level for CPD decoupling
CPD2: WALTZ16 - CPD decoupling sequence, defined by cpdprg2
D1: 100 msec – relaxation delay
TD: 8 K
O1: middle of the ²H NMR spectrum
NS: 256

Processing parameters
SI: 4 K
WDW: EM
FT: Fourier transformation
baseline correction: ABS
plot: use XWINPLOT

Experiment 9.8
- ¹¹B NMR Spectroscopy

pulse program: zgdc
compare with Experiment 3.10
Setting of the needed channels: 
F1: $^{11}$B  
F2: $^1$H

Acquisition parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel - high power level for $^{11}$B transmitter pulse</td>
</tr>
<tr>
<td>PL12</td>
<td>f2 channel - power level for CPD decoupling</td>
</tr>
<tr>
<td>CPD2</td>
<td>WALTZ16 - CPD decoupling sequence, defined by cpdprg2</td>
</tr>
<tr>
<td>D1</td>
<td>100 msec – relaxation delay</td>
</tr>
<tr>
<td>D2</td>
<td>5.86 msec, calculated from $^{1}$J(C,H)=212 Hz</td>
</tr>
<tr>
<td>TD</td>
<td>:4 K</td>
</tr>
<tr>
<td>O1</td>
<td>:middle of the $^{11}$B NMR spectrum</td>
</tr>
<tr>
<td>NS</td>
<td>:8</td>
</tr>
<tr>
<td>P1</td>
<td>f1 channel - 90° $^{11}$B transmitter pulse</td>
</tr>
<tr>
<td>PCDP2</td>
<td>f2 channel - 90° pulse for decoupling sequence</td>
</tr>
<tr>
<td>D11</td>
<td>:30 msec - delay for disk I/O</td>
</tr>
<tr>
<td>D12</td>
<td>:20 usec - delay for power switching</td>
</tr>
<tr>
<td>SW</td>
<td>:36 ppm</td>
</tr>
<tr>
<td>O2</td>
<td>:middle of $^1$H NMR spectrum</td>
</tr>
<tr>
<td>RG</td>
<td>:receiver gain for correct ADC input</td>
</tr>
</tbody>
</table>

Processing parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI</td>
<td>:2 K</td>
</tr>
<tr>
<td>WDW</td>
<td>:EM</td>
</tr>
<tr>
<td>FT</td>
<td>:Fourier transformation</td>
</tr>
<tr>
<td>referencing</td>
<td>:use the external reference of the standard; be sure not to change the magnetic field between the two measurements</td>
</tr>
<tr>
<td>baseline correction</td>
<td>:ABS</td>
</tr>
<tr>
<td>plot</td>
<td>:use XWINPLOT</td>
</tr>
<tr>
<td>BC_mod</td>
<td>:quad</td>
</tr>
<tr>
<td>LB</td>
<td>:2 Hz</td>
</tr>
<tr>
<td>phase correction</td>
<td>:adjust the phase to pure absorption.</td>
</tr>
</tbody>
</table>

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

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

Processing parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI</td>
<td>:2 K</td>
</tr>
<tr>
<td>BC_mod</td>
<td>:quad</td>
</tr>
</tbody>
</table>
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  

Processing parameters  
SI : 2 K  
WDW : EM  
BC_mod : quad  
LB : 15 Hz  
phase correction : adjust the phase to pure absorption.  
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  
D13 : 3 usec - short delay  
DE : 10 usec  
TD : 8 K  
SW : 600 ppm  
O1 : middle of the titanium NMR spectrum  
NS : 8  

Processing parameters  
SI : 2 K  
WDW : EM  
BC_mod : quad  
LB : 15 Hz  
phase correction : adjust the phase to pure absorption.  
plot : use XWINPLOT
# Chapter 10

- The Second Dimension

## Summary

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<th>Description</th>
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<td>2D J-Resolved $^{13}$C NMR Spectroscopy</td>
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<td>Phase-Sensitive COSY</td>
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<td>cosydfprtp</td>
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<td>hxcondtp.mo</td>
<td>Fully Coupled C,H Correlation (FUCOUP)</td>
</tr>
<tr>
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<td>hxco</td>
<td>C,H Correlation by Polarization Transfer (HETCOR)</td>
</tr>
<tr>
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<td>hxco</td>
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<td>10.12</td>
<td>coloc</td>
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</tr>
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<td>invbtp</td>
<td>The Basic HMOC Experiment</td>
</tr>
<tr>
<td>10.14</td>
<td>invbtp</td>
<td>Phase-Sensitive HMOC with BIRD Filter and GARP Decoupling</td>
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<tr>
<td>10.15</td>
<td>exp10_15.mo</td>
<td>Poor Man's Gradient HMOC</td>
</tr>
<tr>
<td>10.16</td>
<td>invblndtp.mo</td>
<td>Phase-Sensitive HMBC with BIRD Filter</td>
</tr>
<tr>
<td>10.17</td>
<td>invindtp.mo</td>
<td>The HOHAHA or TOCSY Experiment</td>
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<td>10.19</td>
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<td>The NOESY Experiment</td>
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<td>10.20</td>
<td>roesytp.2</td>
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</tr>
<tr>
<td>10.21</td>
<td>hoesy</td>
<td>The HOESY Experiment</td>
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<td>10.22</td>
<td>inad</td>
<td>2D-INADEQUATE</td>
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<td>10.23</td>
<td>noesytp</td>
<td>The EXSY Experiment</td>
</tr>
<tr>
<td>10.24</td>
<td>coxyf3.mo/inv4xyf3.mo</td>
<td>X, Y Correlation</td>
</tr>
</tbody>
</table>

### 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: 'H  
F2: off

**Acquisition parameters**

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

**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**: not necessary  
**SYMJ**: use XWINPLOT

**Experiment 10.2**

- 2D J-Resolved 'C NMR Spectroscopy

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

Setting of the needed channels:  
F1: 'C  
F2: 'H

**Acquisition parameters**

**PL1**: f1 channel - high power level for 'C transmitter pulse  
**P1**: f1 channel - 90° 'C transmitter pulse  
**PL12**: f2 channel - power level for CPD decoupling  
**P2**: f1 channel - 180° 'C transmitter pulse  
**PCPD2**: f2 channel - 90° pulse for decoupling sequence  
**D0**: 3 usec - incremented delay  
**D1**: 2 sec - relaxation delay  
**Parmod**: 2D  
**D12**: 20 usec - delay for power switching  
**TD2**: 1K data points in F2  
**ND0**: 2  
**TD1**: 64 data points in F1  
**SW2**: 175 ppm  
**SW1**: 250 Hz  
**O1**: middle of 'C NMR spectrum  
**O2**: middle of 'H NMR spectrum  
**NS**: 32  
**DS**: 16  
**IN0**: 1/2*SW1
Experiment 10.3
- The Basic H,H-COSY-Experiment

pulse program:  
  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  
  INO : 1/[1*SW1]

Processing parameters  
  SI(F2) : 512 W  
  WDW(F2) : QSINE  
  SSB(F2) : 2  
  PH-mod(F2) : no  
  MC2 : QF  
  SYM : may be performed  
  plot : use XWINPLOT

Experiment 10.4
- Long-Range COSY

pulse program:  
  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
**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:**

<table>
<thead>
<tr>
<th>Channel</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>'H</td>
</tr>
<tr>
<td>F2</td>
<td>off</td>
</tr>
</tbody>
</table>

**Acquisition parameters**

**PL1** : f1 channel - high power level for \(^1\text{H}\) transmitter pulse

- \(D0\) : 3 usec - incremented delay
- \(D1\) : 2 sec – relaxation delay
- \(\text{Parmod}\) : 2D
- \(\text{TD2}\) : 1 K data points in F2
- \(\text{SW2}\) : 8 ppm
- \(\text{O1}\) : middle of \(^1\text{H}\) NMR spectrum
- \(\text{NS}\) : 4
- \(\text{IN0}\) : 1/[2\*\(\text{SW1}\)]

**P1** : f1 channel - 90° \(^1\text{H}\) transmitter pulse

- \(\text{D6}\) : 200 msec
- \(\text{ND0}\) : 1
- \(\text{TD1}\) : 128 data points in F1
- \(\text{SW1}\) : 8 ppm
- \(\text{DS}\) : 16
- \(\text{RG}\) : 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 10.6
- 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:  'H  
F2:  off

Acquisition parameters  
PL1 :f1 channel - high power level for 'H transmitter pulse  
P1 :f1 channel - 90° 'H transmitter pulse  
D0 :3 usec - incremented delay  
P0 :f1 channel - 45° 'H transmitter pulse  
Parmod :2D  
TD2 :2 K data points in F2  
D1 :2 sec - relaxation delay  
TD1 :256 data points in F1  
SW2 :1.5 ppm  
SW1 :1.5 ppm  
O1 : middle of 'H NMR spectrum  
NS :4  
IN0 :1/[2*SW1]  
DS :16  
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 resolution  
LB(F1) : depending on the resolution  
GB(F2) :  
GB(F1) :  
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:  
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.

Experiment 10.8
- Double Quantum Filtered COSY with Presaturation

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

| PL1 | f1 channel - high power level for $^{13}\text{C}$ transmitter pulse |
| PL9 | f1 channel - power level for presaturation, typically in the range of 65 dB |
| PL1 | f1 channel - 90° $^{13}\text{C}$ transmitter pulse |
| D0 | 3 usec - incremented delay |
| D1 | 30 msec – delay for disk I/O |
| D13 | 3 usec – short delay |
| Parmod | 2D |
| TD2 | 2 K data points in F2 |
| SW2 | 10 ppm |
| O1 | on resonance of water signal |
| NS | 64 |
| IN0 | $1/[2\times SW1]$ |

### Processing parameters

| SI(F2) | 1 K |
| WDW(F2) | :GM |
| LB(F2) | : depending on the resolution |
| GB(F2) | :phk |
| PHC0(F2) | : should be 0 before first transformation |
| PHC1(F2) | : should be 0 before first transformation |
| MC2 | :TPPI |

### Experiment 10.9
- Fully Coupled C,H Correlation (FUCOUP)

#### pulse program: hxxcondtp.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 | $^{13}\text{C}$ |
| F2 | $^1\text{H}$ |

#### Acquisition parameters

| PL1 | f1 channel - high power level for $^{13}\text{C}$ transmitter pulse |
| PL2 | f2 channel - high power level for $^1\text{H}$ decoupler pulse |
| D0 | 3 usec - incremented delay |
| D11 | 30 msec – delay for disk I/O |
| Parmod | 2D |
| TD2 | 512 data points in F2 |

| P1 | f1 channel - 90° $^{13}\text{C}$ transmitter pulse |
| P3 | f2 channel - 90° $^1\text{H}$ decoupler pulse |
| D1 | 10 sec - relaxation delay |
| D12 | 20 usec - delay for power switching |
| ND0 | 2 |
| TD1 | 64 data points in F1 |
Experiment 10.10
- C,H Correlation by Polarization Transfer (HETCOR)

<table>
<thead>
<tr>
<th>Pulse program:</th>
<th>hxco</th>
</tr>
</thead>
<tbody>
<tr>
<td>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.</td>
<td></td>
</tr>
</tbody>
</table>

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 |
| PCD2 | f2 channel - 90° pulse for decoupling sequence |
| D1 | :2 sec - relaxation delay |
| D3 | :1/[3J(C,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 |
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 $^2J(C,H)$ or $^3J(C,H)$. 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: $^{13}\text{C}$
- F2: $^1\text{H}$

Acquisition parameters

| PL1 | :f1 channel - high power level for $^{13}\text{C}$ transmitter pulse |
| PL2 | :f2 channel - high power level for $^1\text{H}$ decoupler pulse |
| PL12| :f2 channel - power level for CPD decoupling |
| CPD2| :WALTZ16 - CPD decoupling sequence, defined by cpdprg2 |
| P1  | :f1 channel - 90° $^{13}\text{C}$ transmitter pulse |
| P2  | :f1 channel - 180° $^{13}\text{C}$ transmitter pulse |
| P3  | :f2 channel - 90° $^1\text{H}$ decoupler pulse |
| PCPD2| :f2 channel - 90° pulse for decoupling sequence |
| D0  | :3 usec - incremented delay |
| D2  | :[(2J(C,H))= 50 msec, calculated from $^2J(C,H)=10$ Hz |
| D11 | :30 msec – delay for disk I/O |
| Parmod| :2D |
| TD2 | :1 K data points in F2 |
| SW2 | :175 ppm |
| O1  | :middle of $^{13}\text{C}$ NMR spectrum |
| NS  | :64 |
| IN0 | :1/2*SW1 |
| D1  | :2 sec - relaxation delay |
| D3  | :1/[3J(C,H)]= 33 msec, calculated from $^3J(C,H)=10$ Hz |
| D11 | :20 usec - delay for power switching |
| ND0 | :2 |
| TD1 | :128 data points in F1 |
| SW1 | :8 ppm |
| O2  | :middle of $^1\text{H}$ 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 $^{1}J(C,H)=10$ Hz
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
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]

Processing parameters
SI(F2):512 W
WDW(F2):QSINE
SSB(F2):2
PH-mod(F2):no
MC2:QF
phase correction:not necessary
S1(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
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-\(^13\)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

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

D0 : 3 usec - incremented delay

D1 : 2 sec – relaxation delay

**Parmod** : 2D

**TD2** : 1 K data points in F2

**SW2** : 8 ppm

O1 : middle of \(^1\)H NMR spectrum

NS : 8

IN0 : 1/[2*SW1]

**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

D2 : 1/[2J(C,H)] = 3.5 msec, calculated from \(J(C,H)=145\) Hz

**Parmod** : 2D

**TD2** : 1 K data points in F2

**SW2** : 8 ppm

**D0** : 3 usec - incremented delay

**D1** : 2 sec – relaxation delay

**D2** : 1/[2J(C,H)] = 3.5 msec, calculated from \(J(C,H)=145\) Hz

**Parmod** : 2D

**TD2** : 1 K data points in F2

**SW2** : 8 ppm

**PL1** : f1 channel - 90° \(^1\)H transmitter pulse

**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. 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

**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

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

Processing parameters

**SI(F1)** : 256 W

**SI(F2)** : 512 W

**WDW(F2)** : QSINE

**WDW(F1)** : QSINE

**SSB(F2)** : 2

**SSB(F1)** : 2

**PH-mod(F2)** : no

**PH-mod(F1)** : mc

**MC2** : QF

**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-\(^{13}\)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

**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

D2 : 1/[2J(C,H)] = 3.5 msec, calculated from \(J(C,H)=145\) Hz

**Parmod** : 2D

**TD2** : 1 K data points in F2

**SW2** : 8 ppm

**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. 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

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

---

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-\(^{13}\)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

**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

D2 : 1/[2J(C,H)] = 3.5 msec, calculated from \(J(C,H)=145\) Hz

**Parmod** : 2D

**TD2** : 1 K data points in F2

**SW2** : 8 ppm

**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. 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

**RG** : receiver gain for correct ADC input
O1: middle of $^1$H NMR spectrum
NS: 8
IN0: 1/[4*SW1]

O2: middle of $^{13}$C NMR spectrum
DS: 16
RG: receiver gain for correct ADC input

Processing parameters
SI(F2): 512 W
WDW(F2): GM
LB(F2): depending on the resolution
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 resolution
GB(F1): resolution
PH-mod(F1): pk
PHC0(F1): can be set by au-program calcpinhv
PHC1(F1): can be set by au-program calcpinhv
XFB: 4: fourier transformation in both directions
au-program: calcpinhv (to calculate phase for F1)

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: $f_1$ channel - high power level for $^1$H transmitter pulse

PL2: $f_2$ channel - high power level for $^{13}$C decoupler pulse

PL12: $f_2$ channel - power level for CPD decoupling

CPD2: GARP- CPD decoupling sequence, defined by cpdprg2

D0: 3 usec - incremented delay
D2: 1/[2J(C,H)]= 3.5 msec, calculated from $^1$J(C,H)=145 Hz
D4: 1/[4J(C,H)]= 1.75 msec, calculated from $^1$J(C,H)=145 Hz
Parmod: 2D

P1: $f_1$ channel - 90° $^1$H transmitter pulse
P2: $f_1$ channel - 180° $^1$H transmitter pulse
P28: $f_1$ channel - spin-lock pulse, 2 msec! Not more!

P3: $f_2$ channel - 90° $^{13}$C decoupler pulse
P4: $f_2$ channel - 180° $^{13}$C decoupler pulse
PCPD2: $f_2$ channel - 90° pulse for decoupling sequence

D1: 1 sec - relaxation delay
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
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 $^1J(C,H)$. This is a phase-sensitive version without decoupling.

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

Acquisition parameters

PL1 :f1 channel - high power level for $^1H$ transmitter pulse  
PL2 :f2 channel - high power level for $^{13}C$ decoupler pulse  
D0 :3 usec - incremented delay  
D2 :1/[2J(C,H)]= 3.5 msec, calculated from $^1J(C,H)=145$ 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  
TD2 :1 K data points in F2  
SW2 :8 ppm  
O1 :middle of $^1H$ NMR spectrum

P1 :f1 channel - 90° $^1H$ transmitter pulse  
P2 :f1 channel - 180° $^1H$ transmitter pulse  
P3 :f2 channel - 90° $^{13}C$ decoupler pulse  
P4 :f2 channel - 180° $^{13}C$ decoupler pulse  
P5 :f2 channel - 180° $^{13}C$ decoupler pulse  
D1 :1 sec - relaxation delay  
D6 :1/[2J(C,H)]= 50 msec, calculated from $^1J(C,H)=10$ Hz  
D15 :46.5 msec - D6-D2

ND0 :4  
TD1 :128 data points in F1  
SW1 :175 ppm  
O2 :middle of $^{13}C$ NMR spectrum
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 $^{13}\text{C}$ 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: $^1\text{H}$
F2: $^{13}\text{C}$

Acquisition parameters
PL1 : $f_1$ channel - high power level for $^1\text{H}$ transmitter pulse
PL2 : $f_2$ channel - high power level for $^{13}\text{C}$ decoupler pulse
D0 : 3 usec - incremented delay
D4 : $1/[4J(C,H)]=1.72$ msec, calculated from $J(C,H)=145$ Hz
Parmod : 2D
TD2 : 1 K data points in F2
SW2 : 8 ppm
O1 : middle of $^1\text{H}$ NMR spectrum
NS : 8
INO : $1/[4*SW1]$

Processing parameters
SI(F2) : 512 W
WDW(F2) : EM
SI(F1) : 256 W
WDW(F1) : EM

O1 : middle of $^1\text{H}$ NMR spectrum
O2 : middle of $^{13}\text{C}$ NMR spectrum

ND0 : 4
TD1 : 128 data points in F1
SW1 : 175 ppm
DS : 16
RG : receiver gain for correct ADC input

plot : use XWINPLOT
**Experiment 10.18**

- The HOHAHA or TOCSY Experiment

**puls 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:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Settings</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1:</td>
<td>1H</td>
</tr>
<tr>
<td>F2:</td>
<td>off</td>
</tr>
</tbody>
</table>

**Acquisition parameters**

- **PL1**: f1 channel - high power level for 1H transmitter pulse
- **PL10**: f1 channel - power level for TOCSY spinlock
- **P1**: f1 channel - 90° 1H transmitter pulse
- **P5**: f1 channel - 60° 1H transmitter low power pulse
- **P6**: f1 channel - 90° 1H transmitter low power pulse
- **P7**: f1 channel - 180° 1H 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 1H NMR spectrum
- **NS**: 4
- **IN0**: 1/[2*SW1]

**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
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

- \(\text{PL1} : f_1 \) channel - high power level for \(^1\)H transmitter pulse
- \(\text{D0} : 3\) usec - incremented delay
- \(\text{D8} : 2\) sec - mixing time
- \(\text{Parmod} : 2\)D
- \(\text{TD2} : 1\) K data points in F2
- \(\text{SW2} : 10\) ppm
- \(\text{O1} : \) middle of \(^1\)H NMR spectrum
- \(\text{NS} : 16\)
- \(\text{IN0} : 1/(2\times\text{SW1})\)

Processing parameters

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

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

\textbf{XF2P}: will be executed after correction of the rows
\textbf{XF1P}: will be executed after correction of the columns
\textbf{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: ¹H
F2: off

Acquisition parameters
PL1 : f1 channel - high power level for ¹H transmitter pulse
PL11 : f1 channel - power level for ROESY-spinlock, 23 dB was used here
P1 : f1 channel - 90° ¹H 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
L4 : 320 for 300 msec spin-lock. The loop parameter must be an even number.

Parmod : 2D
TD2 : 1 K data points in F2
SW2 : 10 ppm
O1 : middle of ¹H NMR spectrum
NS : 16
IN0 : 1/[2*SW1]

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

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
plot : use XWINPLOT

XF1P : will be executed after correction of the columns
**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
- **P1**: f1 channel - 90° $^6$Li transmitter pulse
- **P2**: f1 channel - 180° $^6$Li transmitter pulse
- **P3**: f2 channel - 90° $^1$H decoupler pulse
- **PL12**: f2 channel - high power level for CPD decoupling
- **PCPD2**: f2 channel - 90° pulse for decoupling sequence
- **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
- **O2**: middle of $^1$H NMR spectrum
- **NS**: 32
- **IN0**: $1/[2 \times SW1]$
- **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

- **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
- **P3**: f2 channel - 90° $^1$H decoupler pulse
- **D0**: 3 usec - incremented delay
- **D9**: 1.7 sec - mixing time
- **Parmod**: 2D
- **TD2**: 512 data points in F2
- **SW2**: 4 ppm
- **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
- **phase correction**: not necessary

**Processing parameters**

- **SI(F2)**: 256 W
- **WDW(F2)**: EM
- **LB(F2)**: depending on the resolution
- **PH-mod(F2)**: no
- **MC2**: QF

- **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 10.23**

- **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:** $f_1$ channel - high power level for $^1$H transmitter pulse
  - **D0:** 3 usec - incremented delay
  - **D8:** 1 sec - mixing time
  - **Parmod:** 2D
  - **TD2:** 512 data points in F2
  - **SW2:** 0.7 ppm
  - **O1:** middle of methyl group region
  - **NS:** 4
  - **IN0:** $1/[2*SW1]$

  - **P1:** $f_1$ channel - 90° $^1$H transmitter pulse
  - **D1:** 2 sec - relaxation delay
  - **ND0:** 2
  - **TD1:** 32 data points in F1
  - **SW1:** 0.7 ppm
  - **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):** pk
  - **PHC0(F2):** should be 0 before first transformation

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

  **plot:** use XWINPLOT
Experiment 10.24
- X, Y Correlation

a) pulse program: coxy3.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:

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<th>Symbol</th>
<th>Assignment</th>
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<td>&quot;Y (13C)</td>
<td></td>
</tr>
<tr>
<td>F2</td>
<td>1H</td>
<td></td>
</tr>
<tr>
<td>F3</td>
<td>&quot;X (31P)</td>
<td></td>
</tr>
</tbody>
</table>

Acquisition parameters

- **PL1**: f1 channel - high power level for 13C transmitter pulse
- **PL3**: f3 channel - high power level for 31P decoupler pulse
- **PL12**: f2 channel - power level for CPD/BB decoupling
- **D0**: 3 usec - incremented delay
- **D11**: 30 msec – delay for disk I/O
- **Parmod**: 2D
- **TD2**: 1 K data points in F2
- **SW2**: 12 ppm
- **O1**: middle of 13C NMR spectrum
- **O3**: middle of 31P NMR spectrum
- **NS**: 8
- **IN0**: 1/[2*SW1]

Processing parameters

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

- **Si(F1)**: 256 W
- **WDW(F1)**: SINE
- **SSB(F1)**: 0
- **PH-mod(F1)**: mc
- **XFB**: Fourier transformation in both directions
- **plot**: use XWINPLOT

b) pulse program: inv4xy3.mo

compare with Experiment 10.24 a)
Setting of the needed channels:
F1: \(^1\)X (\(^{31}\)P) 
F2: \(^1\)H 
F3: \(^1\)Y (\(^{13}\)C)

Acquisition parameters

**PL1**: f1 channel - high power level for \(^{31}\)P transmitter pulse

**PL3**: f3 channel - high power level for \(^{13}\)C decoupler pulse

**PL12**: f2 channel - power level for CPD/BB decoupling

**CPD2**: WALTZ16 - CPD decoupling sequence, defined by cpdprg2

**D0**: 3 usec - incremented delay
**D11**: 30 msec – delay for disk I/O

**Parmod**: 2D
**TD2**: 256 data points in F2
**SW2**: 1 ppm
**O1**: middle of \(^{31}\)P NMR spectrum
**O3**: middle of \(^{13}\)C NMR spectrum
**NS**: 8
**IN0**: 1/[2*SW1]

**P1**: f1 channel - 90° \(^{31}\)P transmitter pulse

**P2**: f1 channel - 180° \(^{31}\)P transmitter pulse

**P21**: f3 channel - 90° \(^{13}\)C decoupler pulse

**PCPD2**: f2 channel - 90° pulse for decoupling sequence

**D1**: 2 sec - relaxation delay
**D22**: 1/[2J(X,Y)]= 25 msec, calculated from \(^{3}\)J(X,Y)=20 Hz

**ND0**: 2
**TD1**: 128 data points in F1
**SW1**: 12 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)**: SINE 
**SSB(F2)**: 0 
**PH-mod(F2)**: no 
**MC2**: QF 
**phase correction**: not necessary

**SI(F1)**: 256 W 
**WDW(F1)**: SINE 
**SSB(F1)**: 0 
**PH-mod(F1)**: mc 
**XFB**: fourier transformation in both directions 
**plot**: use XWINPLOT
Chapter 11
- NMR Spectroscopy with Pulsed Field Gradients

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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: "H F2: off

Acquisition parameters
PL1 : 1 channel - high power level for "H transmitter pulse
P1 : f1 channel - 90° "H transmitter pulse
P2 : f1 channel - 180° "H transmitter pulse
P16 : 10 msec - homospoil/gradient pulse
D11 : delay for disk I/O
D28 : equal to aq
D1 : 1 sec - relaxation delay
P16 : 10 msec - homospoil/gradient pulse
D16 : 100 usec - delay for homospoil/gradient recovery
TD : 2 K
O1 : on resonance of water signal
SW : 100 KHz
GPZ : 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:  

\[ F1: \ ^1H \]
\[ F2: \text{off} \]

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  
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  
RG :receiver gain for correct ADC input  
gpz 1 :75%

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:  

\[ F1: \ ^1H \]
\[ F2: \text{off} \]

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  
D16 :100 usec - delay for homospoil/gradient recovery  
TD :4 K  
SW :500 Hz  
O1 : on resonance of CHCl\(_3\) signal  
gpnam1 : SINE.100  
NS :16  
gpz 1 :50%
Experiment 11.4
- Determination of Pulsed Field Gradient Ring-Down Delays

pulsel program: zgpp30.mo
The experiment described here demonstrates a calibration routine to define a suitable ringdown delay.

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

Acquisition parameters
PL1 : f1 channel - high power level for 'H transmitter pulse
P1 : f1 channel - 90° 'H transmitter power pulse
D1 : 5 sec - relaxation delay
D11 : 30 msec - delay for disk I/O
TD : 4 K
O1 : on resonance of CHCl3 signal
gpnam1 : rectangular.1
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.
baseline correction : ABS
plot : use XWINPLOT

Experiment 11.5
- The Pulsed Gradient Spin-Echo Experiment

pulsel 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: 'H
F2: off

Acquisition parameters
PL1 : f1 channel - high power level for 'H transmitter pulse
P1 : f1 channel - 90° 'H transmitter power pulse
transmitter pulse

P2 : f1 channel - 180° ¹H transmitter pulse
P16 : 4 msec - homospoil/gradient pulse, variable strength from 0 to 0.2 T/m in 10 steps
D16 : 500 usec - delay for homospoil/gradient recovery
SW : 1000 Hz

D1 : 2 sec - relaxation delay
TD : 1 K
O1 : on resonance of water signal
gpnam1 : SINE.100
gpnam2 : SINE.100
NS : 2

gpz 1 : 1%
gpz 2 : 1%
RG : receiver gain for correct ADC input

Processing parameters
Process all 10 spectra identically.
SI : 512 W
WDW : EM
FT : Fourier transformation
BC mod : quad
LB : 5 Hz
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 strength of 0. Compile a table of integral ratios I_g/I_0 vs. gradient strength G used, where the gradient strength 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 its duration. This method produces an image of the excitation pattern in one scan and provide determinations of the excitation pattern of a) 90° and a b) 180° selective pulse.

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

Acquisition parameters

Processing parameters

b) pulse program:
compare with Exp. 11.6 a)

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

Processing parameters

Experiment 11.7
- The Gradient zz-Filter

pulse program: exp11_7.mo
In many experiments 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: 1H F2: 13C

Acquisition parameters
PL1 : f1 channel - high power level for 1H transmitter pulse
P1 : f1 channel - 90° 1H transmitter pulse
PL2 : f2 channel - high power level for 13C decoupler pulse
P2 : f1 channel - 180° 1H transmitter pulse
P4 : f2 channel - 180° 13C decoupler pulse
D1 : 10 sec - relaxation delay
D4 : 1/[4J(C,H)] = 1.17 msec, calculated from J(C,H) = 214 Hz
D16 : 1.5 msec - homospoil/gradient pulse
D16 : 100 usec - delay for homospoil/gradient recovery
TD : 4 K
O1 : on 1H resonance
NS : 1
GPnam1 : SINE.100
RG : receiver gain for correct ADC input

Processing parameters
SI : 2 K
WDW : EM
FT : Fourier transformation
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: 1H F2: off
Acquisition parameters

- **PL1**: f1 channel - high power level for $^1\text{H}$ transmitter pulse
- **P1**: f1 channel - 90° $^1\text{H}$ transmitter power pulse
- **P2**: f1 channel - 180° $^1\text{H}$ transmitter power pulse
- **P10**: 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
- **P12**: f1 channel - 180° shaped pulse, 50 msec was used here

- **P12**: f1 channel - 180° shaped pulse, 50 msec was used here
- **P16**: 2 msec - homospoil/gradient pulse
- **D16**: 500 usec - delay for homospoil/gradient recovery

- **O1**: on resonance of selected signal or use spoiffs
- **gpnam1**: SINE.100
- **gpnam2**: SINE.100
- **gpnam3**: SINE.100
- **gpnam4**: SINE.100
- **gpnam5**: SINE.100
- **gpnam6**: SINE.100
- **gpz 1**: 40%
- **gpz 2**: 40%
- **gpz 3**: 7%
- **gpz 4**: 7%
- **gpz 5**: 20%
- **gpz 6**: -20%

- **NS**: 1
  - 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
- **plot**: use XWINPLOT

Baseline correction: **ABS**

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**: $^1\text{H}$
- **F2**: off

Acquisition parameters

- **PL1**: f1 channel - high power level for $^1\text{H}$ transmitter pulse
- **P1**: f1 channel - 90° $^1\text{H}$ transmitter power pulse
- **P2**: f1 channel - 180° $^1\text{H}$ 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
- **P12**: f1 channel - 180° shaped pulse, 50 msec was used here

- **SP1**: f1 channel - power level for shaped pulse, here 64 dB was used
- **D1**: 2 sec - relaxation delay
- **D2**: 30-60 msec, adjusted to $1/[2\varepsilon(H,H)]$
- **TD**: 32 K
- **P12**: f1 channel - 180° shaped pulse, 50 msec was used here
- **D16**: 500 usec - delay for homospoil/gradient recovery

- **D2**: 30-60 msec, adjusted to $1/[2\varepsilon(H,H)]$
- **TD**: 32 K
- **O1**: on resonance of selected signal or use spoiffs
- **gpnam1**: SINE.100
- **gpnam2**: SINE.100
- **gpnam3**: SINE.100
- **gpnam4**: SINE.100
- **gpnam5**: SINE.100
- **gpnam6**: SINE.100
- **gpz 1**: 40%
- **gpz 2**: 40%
- **gpz 3**: 7%
- **gpz 4**: 7%
- **gpz 5**: 20%
- **gpz 6**: -20%

- **NS**: 1
  - Gaussian shape with 1024 data points was used

- **RG**: receiver gain for correct ADC input

Baseline correction: **ABS**

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
- **plot**: use XWINPLOT

Baseline correction: **ABS**
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: \textsuperscript{1}H
F2: off

Acquisition parameters
PL0 : 120 dB
PL1 : f1 channel - high power level for \textsuperscript{1}H transmitter pulse
SP1 : f1 channel - power level for shaped pulse, here 62 dB was used
D1 : 2 sec - relaxation delay
D16 : 500 usec - delay for homospoil/gradient recovery
TD : 32 K
O1 : middle of \textsuperscript{1}H NMR spectrum
gpnam1 : SINE.100
gpnam2 : SINE.100
gpnam3 : SINE.100
gpnam4 : SINE.100
NS : 1
Gaussian shape with 1024 data points was used

D9 : 250 msec; 76 msec; 215 msec – mixing time (3 different experiments)
D16 : 500 usec - delay for homospoil/gradient recovery
D20 : 1 msec - equal to the effective length of the gradient pulse

Processing parameters
SI : 16 K
WDW : EM
FT : Fourier transformation
baseline correction : ABS

BC_mod : quad
LB : 0.1 Hz
phase correction : adjust the signals to pure absorption.
plot : use XWINPLOT

RG : receiver gain for correct ADC input

PL0 : 120 dB
PL1 : f1 channel - high power level for \textsuperscript{1}H transmitter pulse
SP1 : f1 channel - power level for shaped pulse, here 62 dB was used
D1 : 2 sec - relaxation delay
D16 : 500 usec - delay for homospoil/gradient recovery
TD : 32 K
O1 : middle of \textsuperscript{1}H NMR spectrum
gpnam1 : SINE.100
gpnam2 : SINE.100
gpnam3 : SINE.100
gpnam4 : SINE.100
NS : 1
Gaussian shape with 1024 data points was used

D9 : 250 msec; 76 msec; 215 msec – mixing time (3 different experiments)
D16 : 500 usec - delay for homospoil/gradient recovery
D20 : 1 msec - equal to the effective length of the gradient pulse

Processing parameters
SI : 16 K
WDW : EM
FT : Fourier transformation
baseline correction : ABS

BC_mod : quad
LB : 0.1 Hz
phase correction : adjust the signals to pure absorption.
plot : use XWINPLOT

RG : receiver gain for correct ADC input

PL0 : 120 dB
PL1 : f1 channel - high power level for \textsuperscript{1}H transmitter pulse
SP1 : f1 channel - power level for shaped pulse, here 62 dB was used
D1 : 2 sec - relaxation delay
D16 : 500 usec - delay for homospoil/gradient recovery
TD : 32 K
O1 : middle of \textsuperscript{1}H NMR spectrum
gpnam1 : SINE.100
gpnam2 : SINE.100
gpnam3 : SINE.100
gpnam4 : SINE.100
NS : 1
Gaussian shape with 1024 data points was used

D9 : 250 msec; 76 msec; 215 msec – mixing time (3 different experiments)
D16 : 500 usec - delay for homospoil/gradient recovery
D20 : 1 msec - equal to the effective length of the gradient pulse

Processing parameters
SI : 16 K
WDW : EM
FT : Fourier transformation
baseline correction : ABS

BC_mod : quad
LB : 0.1 Hz
phase correction : adjust the signals to pure absorption.
plot : use XWINPLOT

RG : receiver gain for correct ADC input
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 $^{13}$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
- PCDP2: 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
Experiment 11.12
- GRECCO

pulse program:

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

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

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: $^1H$  
F2: off

Acquisition parameters

Processing parameters

WDW :EM  
FT :Fourier transformation  
LB :0.5 Hz  
phase correction : adjust the signals to pure absorption.  
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:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1:</td>
<td>$^1$H</td>
</tr>
<tr>
<td>F2:</td>
<td>off</td>
</tr>
</tbody>
</table>

Acquisition parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>$^1$H transmitter pulse</td>
</tr>
<tr>
<td>PL18</td>
<td>low power level for 3-9-19 pulse (watergate)</td>
</tr>
<tr>
<td>D1</td>
<td>1 sec - relaxation delay</td>
</tr>
<tr>
<td>D19</td>
<td>250 usec</td>
</tr>
<tr>
<td>TD</td>
<td>32 K</td>
</tr>
<tr>
<td>O1</td>
<td>on water resonance</td>
</tr>
<tr>
<td>gpnam1</td>
<td>SINE.100</td>
</tr>
<tr>
<td>gpnam2</td>
<td>SINE.100</td>
</tr>
<tr>
<td>gpnam3</td>
<td>SINE.100</td>
</tr>
<tr>
<td>gpnam4</td>
<td>SINE.100</td>
</tr>
<tr>
<td>DS</td>
<td>4</td>
</tr>
<tr>
<td>P1</td>
<td>90° $^1$H transmitter power pulse</td>
</tr>
<tr>
<td>P0</td>
<td>90° pulse at PL18</td>
</tr>
<tr>
<td>P16</td>
<td>1 msec - homospoil/gradient pulse</td>
</tr>
<tr>
<td>P28</td>
<td>90° pulse at PL18</td>
</tr>
<tr>
<td>D16</td>
<td>500 usec - delay for homospoil/gradient recovery</td>
</tr>
<tr>
<td>SW</td>
<td>10 ppm</td>
</tr>
<tr>
<td>NS</td>
<td>16</td>
</tr>
<tr>
<td>gpz 1</td>
<td>40%</td>
</tr>
<tr>
<td>gpz 2</td>
<td>40%</td>
</tr>
<tr>
<td>gpz 3</td>
<td>7%</td>
</tr>
<tr>
<td>gpz 4</td>
<td>7%</td>
</tr>
<tr>
<td>RG</td>
<td>receiver gain for correct ADC input</td>
</tr>
</tbody>
</table>

Processing parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI</td>
<td>16 K</td>
</tr>
<tr>
<td>WDW</td>
<td>EM</td>
</tr>
<tr>
<td>FT</td>
<td>Fourier transformation</td>
</tr>
<tr>
<td>BC_mod</td>
<td>quad</td>
</tr>
<tr>
<td>LB</td>
<td>0.5 Hz</td>
</tr>
<tr>
<td>phase correction</td>
<td>ignore the phase of the water signal and adjust the others to pure absorption.</td>
</tr>
<tr>
<td>baseline correction</td>
<td>ABS</td>
</tr>
<tr>
<td>plot</td>
<td>use XWINPLOT</td>
</tr>
</tbody>
</table>

baseline correction: ABS
plot: use XWINPLOT
# Chapter 12
- 2D NMR Spectroscopy with Field Gradients

## Summary

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Pulse program</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>12.1</td>
<td>cosygp</td>
<td>gs-COSY</td>
</tr>
<tr>
<td>12.2</td>
<td>cosydfgпт.pmo</td>
<td>Phase-Sensitive gs-DQF-COSY</td>
</tr>
<tr>
<td>12.3</td>
<td>inv4gp</td>
<td>gs-HMOC</td>
</tr>
<tr>
<td>12.4</td>
<td>inv4gplпnd</td>
<td>gs-HMBC</td>
</tr>
<tr>
<td>12.5</td>
<td>inv4agplпlr.pmo</td>
<td>ACCORD-HMBC</td>
</tr>
<tr>
<td>12.6</td>
<td>invrietгpsi</td>
<td>Phase-Sensitive gs-HSQC with Sensitivity Enhancement</td>
</tr>
<tr>
<td>12.7</td>
<td>mlevgp.pmo</td>
<td>gs-TOCSY</td>
</tr>
<tr>
<td>12.8</td>
<td>inv4gpml</td>
<td>gs-HMOC-TOCSY</td>
</tr>
<tr>
<td>12.9</td>
<td></td>
<td>2O-HMBC</td>
</tr>
<tr>
<td>12.10</td>
<td>ineptинadgp.pmo</td>
<td>Gradient-Selected $^1$H-Detected 2D INEPT-INADEQUATE</td>
</tr>
<tr>
<td>12.11</td>
<td>noesygпst</td>
<td>gs-NOESY</td>
</tr>
<tr>
<td>12.12</td>
<td>invrietгpnпo.pmo</td>
<td>gs-HSQC-NOESY</td>
</tr>
<tr>
<td>12.13</td>
<td></td>
<td>gs-HOESY</td>
</tr>
<tr>
<td>12.14</td>
<td>inv4gpnd.pmo</td>
<td>$^1$H, $^{15}$N Correlation with gs-HMOC</td>
</tr>
</tbody>
</table>

## 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**: $^1$ channel - high power level for $^1$H transmitter pulse  
- **P1**: $^1$ channel - 90° $^1$H transmitter pulse  
- **P0**: $^1$ channel - 90° $^1$H transmitter pulse  
- **P16**: 2 msec - homospoil/gradient pulse  
- **D1**: 2 sec - relaxation delay  
- **D16**: 500 usec - delay for homospoil/gradient recovery  
- **ND0**: 1  
- **TD1**: 256 data points in F1  
- **SW1**: 10 ppm  
- **DS**: 16  
- **gpz 1**: 10%  
- **gpz 2**: 10%  
- **RG**: receiver gain for correct ADC input
### 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**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel - high power level for $^1$H transmitter pulse</td>
</tr>
<tr>
<td>P1</td>
<td>f1 channel - 90° $^1$H transmitter pulse</td>
</tr>
<tr>
<td>P2</td>
<td>f1 channel - 180° $^1$H transmitter pulse</td>
</tr>
<tr>
<td>P16</td>
<td>2 msec - homospoil/gradient pulse</td>
</tr>
<tr>
<td>D0</td>
<td>3 usec - incremented delay</td>
</tr>
<tr>
<td>D1</td>
<td>2 sec - relaxation delay</td>
</tr>
<tr>
<td>D13</td>
<td>3 usec - short delay</td>
</tr>
<tr>
<td>D16</td>
<td>500 usec - delay for homospoil/gradient recovery</td>
</tr>
<tr>
<td>D20</td>
<td>2 msec - same length as gradient pulse</td>
</tr>
<tr>
<td>Parmod</td>
<td>2D</td>
</tr>
<tr>
<td>ND0</td>
<td>2</td>
</tr>
<tr>
<td>TD2</td>
<td>2 K data points in F2</td>
</tr>
<tr>
<td>TD1</td>
<td>512 data points in F1</td>
</tr>
<tr>
<td>SW2</td>
<td>10 ppm</td>
</tr>
<tr>
<td>SW1</td>
<td>10 ppm</td>
</tr>
<tr>
<td>O1</td>
<td>middle of $^1$H NMR spectrum</td>
</tr>
<tr>
<td>NS</td>
<td>4</td>
</tr>
<tr>
<td>gpnam1</td>
<td>SINE.100</td>
</tr>
<tr>
<td>gpnam2</td>
<td>SINE.100</td>
</tr>
<tr>
<td>IN0</td>
<td>1/[2*SW1]</td>
</tr>
<tr>
<td>RG</td>
<td>receiver gain for correct ADC input</td>
</tr>
<tr>
<td>gpz1</td>
<td>10%</td>
</tr>
<tr>
<td>gpz2</td>
<td>20%</td>
</tr>
<tr>
<td>DS</td>
<td>16</td>
</tr>
</tbody>
</table>

**Processing parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si(F2)</td>
<td>512 W</td>
</tr>
<tr>
<td>WDW(F2)</td>
<td>SINE</td>
</tr>
<tr>
<td>SSB(F2)</td>
<td>0</td>
</tr>
<tr>
<td>PH-mod(F2)</td>
<td>no</td>
</tr>
<tr>
<td>MC2</td>
<td>TPPI</td>
</tr>
<tr>
<td>PHC0(F2)</td>
<td>should be 0 before first transformation</td>
</tr>
<tr>
<td>PHC1(F2)</td>
<td>should be 0 before first transformation</td>
</tr>
<tr>
<td>PHC0(F1)</td>
<td>90</td>
</tr>
<tr>
<td>PHC1(F1)</td>
<td>should be 0 before first transformation</td>
</tr>
</tbody>
</table>

**Plot:** use XWINPLOT
**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 $^{13}$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
  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 $^J(C,H) = 140$ 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]

  **Processing parameters**
  
  SI(F2) : 512 W
  WDW(F2) : EM
  LB(F2) : 5 Hz
  PH-mod(F2) : no
  MC2 : QF

  phase correction: not necessary

  plot : use XWINPLOT

**Experiment 12.4**

- **gs-HMBC**

  pulse program: inv4gpplrnd
  
  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
Experiment 12.5
- ACCORD-HMBC

pulse program: inv4acgplkr.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**

<table>
<thead>
<tr>
<th>Channel</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel - high power level for $^{1}H$ transmitter pulse</td>
</tr>
<tr>
<td>PL2</td>
<td>f2 channel - high power level for $^{13}C$ decoupler pulse</td>
</tr>
<tr>
<td>D0</td>
<td>3 usec - incremented delay</td>
</tr>
<tr>
<td>D2</td>
<td>$1/[2J(C,H)] = 3.57$ msec, calculated from $^{1}J(C,H)$=140 Hz</td>
</tr>
<tr>
<td>D13</td>
<td>3 usec - short delay</td>
</tr>
</tbody>
</table>

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  
D16: 100 usec - delay for homospoil/gradient recovery

<table>
<thead>
<tr>
<th>Parmod</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>ND0</td>
<td>2</td>
</tr>
<tr>
<td>TD1</td>
<td>256 data points in F1</td>
</tr>
<tr>
<td>SW1</td>
<td>165 ppm</td>
</tr>
<tr>
<td>O2</td>
<td>middle of $^{13}C$ NMR spectrum</td>
</tr>
<tr>
<td>DS</td>
<td>16</td>
</tr>
<tr>
<td>gpz 1</td>
<td>50%</td>
</tr>
<tr>
<td>gpz 2</td>
<td>30%</td>
</tr>
<tr>
<td>gpz 3</td>
<td>40.1</td>
</tr>
<tr>
<td>RG</td>
<td>receiver gain for correct ADC input</td>
</tr>
</tbody>
</table>

**Processing parameters**

<table>
<thead>
<tr>
<th>Channel</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI(F2)</td>
<td>512 W</td>
</tr>
<tr>
<td>WDW(F2)</td>
<td>EM</td>
</tr>
<tr>
<td>LB(F2)</td>
<td>5 Hz</td>
</tr>
<tr>
<td>PH-mod(F2)</td>
<td>no</td>
</tr>
<tr>
<td>MC2</td>
<td>QF</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Parmod</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI(F1)</td>
<td>512 W</td>
</tr>
<tr>
<td>WDW(F1)</td>
<td>QSINE</td>
</tr>
<tr>
<td>SSB(F1)</td>
<td>3</td>
</tr>
<tr>
<td>PH-mod(F1)</td>
<td>mc</td>
</tr>
<tr>
<td>XFB</td>
<td>Fourier transformation in both directions</td>
</tr>
</tbody>
</table>

phase correction: not necessary  
plot: use XWINPLOT

Setting of the needed channels: 
F1: $^{1}H$  
F2: $^{13}C$
CPD2: GARP - CPD decoupling sequence, defined by cpdprg2

D0: 3 usec - incremented delay
D2: 1/[2J(C,H)] = 3.57 msec, calculated from 1J(C,H) = 140 Hz
D16: 50 usec - delay for homospoil/gradient recovery
D21: 1s/2*(cnst7)-0.146*(cnst7-cnst6)-p16-d16 = 2.1 msec
cnst6: J(XH)min, here 128 Hz

Parmod: >2
TD2: 2 K data points in F2
SW2: 10 ppm
O1: middle of 1H NMR spectrum
NS: 2
gpnam1: SINE.100
gpnam2: SINE.100
gpnam3: SINE.100
gpnam4: SINE.100
gpnam5: SINE.100
gpnam6: SINE.100
gpnam7: SINE.100
gpnam8: SINE.100
IN0: 1/[2*SW1]

RG: receiver gain for correct ADC input

Processing parameters
S1(F2): 1 K
WDW(F2): EM
LB(F2): 5 Hz
PH-mod(F2): no
MC2: QF
phase correction: not necessary

gpzm: 15%
gpz 2: -10%
gpz 3: -5%
gpz 4: 50%
gpz 5: 30%
gpz 6: 40%
gpz 7: -5%
gpz 8: 5%
IN6: (200 msec-20 msec)/td1 = 0.7 msec, corresponding 2.5 Hz to 25Hz

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 13C chemical shift evolution of a single quantum coherence. In this case it is a gradient-selected correlation using echo/anteecho selection method.

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

Acquisition parameters
PL1: f1 channel - high power level for 1H transmitter pulse
PL2: f2 channel - high power level for 13C decoupler pulse

P1: f1 channel - 90° 1H transmitter pulse
P2: f1 channel - 180° 1H transmitter pulse
P3: f2 channel - 90° 13C decoupler pulse
P4: f2 channel - 180° 13C decoupler pulse
P16: 1.6 msec - homospoil/gradient pulse
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:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>(^1\text{H})</td>
</tr>
<tr>
<td>F2</td>
<td>off</td>
</tr>
</tbody>
</table>

Acquisition parameters

<table>
<thead>
<tr>
<th>PL1</th>
<th>:f1 channel - high power level for (^1\text{H}) transmitter pulse</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL10</td>
<td>:f1 channel - power level for TOCSY-spinlock</td>
</tr>
</tbody>
</table>

Processing parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI(F2)</td>
<td>512 W</td>
</tr>
<tr>
<td>WDW(F2)</td>
<td>EM</td>
</tr>
<tr>
<td>LB(F2)</td>
<td>3 Hz</td>
</tr>
<tr>
<td>PH-mod(F2)</td>
<td>pk</td>
</tr>
<tr>
<td>PHC0(F2)</td>
<td>should be 0 before first transformation</td>
</tr>
<tr>
<td>MC2</td>
<td>echo-antiecho</td>
</tr>
</tbody>
</table>

phase correction: use the 2D-phase correction routine, phase correction is usually only necessary in F2

plot: use XWINPLOT
power pulse
P16: 2 msec - homospoil/gradient pulse
P17: f1 channel - 2.5 msec - trim pulse

D9: 100 msec - mixing time
D16: 500 usec - delay for homospoil/gradient recovery

ND0: 1
TD1: 256 data points in F1
SW1: 9 ppm

DS: 16
gpz 1: 10%
gpz 2: 10%
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

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: ¹H
F2: ¹³C

Acquisition parameters
PL1: f1 channel - high power level for ¹H transmitter pulse
PL2: f2 channel - high power level for ¹³C decoupler pulse
PL10: f1 channel - power level for TOCSY-spinlock

PL12: f2 channel - power level for CPD decoupling
CPD2: GARP - CPD decoupling sequence,

P1: f1 channel - 90° ¹H transmitter pulse
P2: f1 channel - 180° ¹H transmitter pulse
P3: f2 channel - 90° ¹³C decoupler pulse
P5: f1 channel - 60° ¹H transmitter low power pulse
P6: f1 channel - 90° ¹H transmitter low power pulse
P7: f1 channel - 180° ¹H transmitter low power pulse
P16: 2 msec - homospoil/gradient pulse
P17: f1 channel - 2.5 msec - trim pulse
PCPD2: f2 channel - 90° pulse for decoupling sequence
Experiment 12.9
- 2Q-HMBC

Pulse program:

This experiment detects long-range carbon-carbon connectivities. The transfer from protons to $^{13}\text{C}$ uses a $3\text{J(C,H)}$ or a $2\text{J(C,H)}$ instead of a $1\text{J(C,H)}$ 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: $^1\text{H}$
F2: $^{13}\text{C}$

Acquisition parameters

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

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

$\text{D0}: 3$ usec - incremented delay
$\text{D11}: 30$ msec - delay for disk I/O

$\text{cnst0}: \text{ds}=\text{ns}^2\text{cnst0}$
$\text{cnst7}: 179$ Hz
$\text{Parmod}: 2\text{D}$
$\text{TD2}: 1$ K data points in F2

$\text{P1}: f1$ channel - 90° $^1\text{H}$ transmitter pulse
$\text{P2}: f1$ channel - 180° $^1\text{H}$ transmitter pulse
$\text{P3}: f2$ channel - 90° $^{13}\text{C}$ decoupler pulse
$\text{P4}: f2$ channel - 180° $^{13}\text{C}$ decoupler pulse
$\text{P16}: 1$ msec - homospoil/gradient pulse
$\text{P28}: f1$ channel - 2 msec - trim pulse
$\text{D1}: 4$ sec - relaxation delay
$\text{D16}: 100$ usec - delay for homospoil/gradient recovery

$\text{cnst6}: 159$ Hz
$\text{cnst8}: 8.5$ Hz
$\text{ND0}: 2$
$\text{TD1}: 128$ data points in F1

Processing parameters

$\text{SI(F1)}: 512$ W
$\text{SI(F2)}: 512$ W
$\text{WDW(F1)}: \text{SINE}$
$\text{WDW(F2)}: \text{SINE}$
$\text{SSB(F1)}: 0$
$\text{SSB(F2)}: 0$
$\text{PH-mod(F1)}: \text{mc}$
$\text{PH-mod(F2)}: \text{no}$
$\text{MC2}: \text{QF}$

phase correction: not necessary
plot: use XWINPLOT
SW2 : 5.2 ppm
O1 : center of $^1$H NMR spectrum
NS : 32
gpnam1 : SINE.100
gpnam2 : SINE.100
gpnam3 : SINE.100
gpnam4 : SINE.100
gpnam5 : SINE.100
IN0 : $1/[2^*SW1]$

SW1 : 206 ppm (C,C double quantum frequency)
O2 : center of $^{13}$C NMR spectrum
L3 : TD1/2
gpz 1 : 30%
gpz 2 : -20%
gpz 3 : -10%
gpz 4 : 30%
gpz 5 : -10%
RG : receiver gain for correct ADC input

Processing parameters
SI(F2) : 1024 W
WDW(F2) : SINE
SSB(F2) : 4
PH-mod(F2) : pk
PHC0(F2) : should be 0 before first transformation
XFB : fourier transformation in both directions

XP2P : 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 protons bond to $^{13}$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
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 : $1/[4J(C,H)]=1.8$ msec, calculated from $J(C,H)=140$ Hz
D16 : 200 usec - delay for

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 - 2 msec - trim pulse

D0 : 3 usec - incremented delay
D4 : $1/[4J(C,H)]=1.8$ msec, calculated from $J(C,H)=140$ Hz
D16 : 200 usec - delay for
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 t1 increment are needed.

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 pulse
P2 :f1 channel - 180° 1H transmitter pulse
P16 :1 msec - homospoil/gradient pulse
D1 :2 sec - relaxation delay
D11 :30 msec - delay for disk I/O
D20 :D8*0.5 – p16 – d16

Parmod :2D
TD2 :2 K data points in F2
SW2 :10 ppm
O1 : middle of 1H NMR spectrum
NS :2

Homospoil/gradient recovery
D21 :D16+P2+D0*2

\[ \frac{1}{2J(C,C)} = 5 \text{ msec}, \text{ calculated from} \]
\[ J(C,C) = 50 \text{ Hz} \]

D23 :1/[4J(C,C)] = 5 msec, calculated from \[ J(C,C) = 50 \text{ Hz} \]

L3 :TD1/2

ND0 :2

TD1 :2 times 512 data points in F1
SW1 :80 ppm (C.C double quantum frequency)

O2 : center of 13C NMR spectrum
DS :16
gpz 1 : 39.7%
gpz 2 : 39.7%
gpz 3 : 40%
RG : receiver gain for correct ADC input

Processing parameters
SI(F2) :1024 W
WDW(F2) :EM
LB(F2) :6.5
PH-mod(F2) : pk
PHC0(F2) : should be 0 before first transformation
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

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 pulse
P2 :f1 channel - 180° 1H transmitter pulse
P16 :1 msec - homospoil/gradient pulse
D1 :2 sec - relaxation delay
D11 :30 msec - delay for disk I/O
D20 :D8*0.5 – p16 – d16

Parmod :2D
TD2 :2 K data points in F2
SW2 :10 ppm
O1 : middle of 1H NMR spectrum
NS :2
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

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

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

ND0 : 2
TD1 : 64 data points in F1
Experiment 12.13
- gs-HOESY

pulse program:
This gs-HOESY 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: \(^1\text{H}\)
F2: \(^7\text{Li}\)

Acquisition parameters

Processing parameters

Experiment 12.14
- \(^1\text{H}, ^{15}\text{N}\) Correlation with gs-HMQC

pulse program: inv4gpnd.mo
Due to the low receptivity of \(^{15}\text{N}\) it is very tedious to obtain \(^{15}\text{N}\) NMR spectra of organic compounds if they are available only in miligrams. Inverse detection is therefore the method of choice, particularly if the unwanted signals can be effectively 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 $2J(C,H) = 10$ 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**: $1/[2*SW1]$

**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

**plot**: use XWINPLOT
Chapter 13
- The Third Dimension

Summary

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<th>Description</th>
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<td>3D HMQC-COSY</td>
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<td>h_c_p3d.mo</td>
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<td>13.4</td>
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<td>3D HMBC</td>
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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 cpdpgr2  
- **D0**: 3 usec - incremented delay (F1 in 3D)  
- **D1**: 2 sec - relaxation delay  
- **D2**: \(1/(2J(C,H))\) = 3.5 msec, calculated from \(J(C,H) = 145\) 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

Processing parameters

- **SI(F3)**: 256 W  
- **SI(F2)**: 128 W
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

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<th>Description</th>
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<td>TOSS</td>
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<td>14.6</td>
<td></td>
<td>SELTICS</td>
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<td>14.7</td>
<td></td>
<td>Multiplicity Determination in the Solid-State</td>
</tr>
</tbody>
</table>

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
- **D0**: 3 usec - incremented delay
- **D2**: 1/[2J(N,H)]= 50 msec, calculated from \(^2\)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
- **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
- **SI(F1)**: 512 W  
- **WDW(F1)**: SINE  
- **SSB(F1)**: 2  
- **PH-mod(F1)**: mc  
- **XFB**: fourier transformation in both directions

**phase correction**: phase correction is not  
**plot**: use XWINPLOT
b) pulse program:

In solid-state NMR there is usually no lock channel and because of that a reasonable basic shim is necessary.

Setting of the needed channels:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Spin</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{13}$C</td>
</tr>
<tr>
<td>F2</td>
<td>$^1$H</td>
</tr>
</tbody>
</table>

**Acquisition parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel – high power level for $^1$H transmitter pulse</td>
</tr>
<tr>
<td>P1</td>
<td>f1 channel - 90° $^1$H transmitter pulse</td>
</tr>
<tr>
<td>PL2</td>
<td>f2 channel – high power level for $^{15}$N decoupler pulse</td>
</tr>
<tr>
<td>P2</td>
<td>f1 channel - 180° $^1$H transmitter pulse</td>
</tr>
<tr>
<td>P3</td>
<td>f2 channel - 90° $^{15}$N decoupler pulse</td>
</tr>
<tr>
<td>D0</td>
<td>3 usec - incremented delay</td>
</tr>
<tr>
<td>D1</td>
<td>2 sec - relaxation delay</td>
</tr>
<tr>
<td>D11</td>
<td>30 msec - delay for disk I/O</td>
</tr>
<tr>
<td>D16</td>
<td>100 usec - delay for homospoil/gradient recovery</td>
</tr>
<tr>
<td>D20</td>
<td>D4-P16-D16-4usec</td>
</tr>
<tr>
<td>D21</td>
<td>D16+P2+D0*2</td>
</tr>
<tr>
<td>TD2</td>
<td>1 K data points in F2</td>
</tr>
<tr>
<td>SW2</td>
<td>10 ppm</td>
</tr>
<tr>
<td>O1</td>
<td>middle of $^1$H NMR spectrum</td>
</tr>
<tr>
<td>NS</td>
<td>4</td>
</tr>
<tr>
<td>IN0</td>
<td>1/[2*SW1]</td>
</tr>
</tbody>
</table>

**Processing parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI(F2)</td>
<td>512 W</td>
</tr>
<tr>
<td>WDW(F2)</td>
<td>SINE</td>
</tr>
<tr>
<td>LB(F2)</td>
<td>2</td>
</tr>
<tr>
<td>PH-mod(F2)</td>
<td>no</td>
</tr>
<tr>
<td>MC2</td>
<td>QF</td>
</tr>
<tr>
<td>phase correction</td>
<td>phase correction is not necessary</td>
</tr>
<tr>
<td>plot</td>
<td>use XWINPLOT</td>
</tr>
</tbody>
</table>

---

**Experiment 14.2**

– Adjusting the Magic Angle

Setting of the needed channels:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Spin</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{79}$Br</td>
</tr>
<tr>
<td>F2</td>
<td>off</td>
</tr>
</tbody>
</table>

**Acquisition parameters**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel - high power level for $^1$H transmitter pulse</td>
</tr>
<tr>
<td>P1</td>
<td>f1 channel - 90° $^1$H transmitter pulse</td>
</tr>
<tr>
<td>D0</td>
<td>3 usec - incremented delay</td>
</tr>
<tr>
<td>D1</td>
<td>2 sec - relaxation delay</td>
</tr>
<tr>
<td>D11</td>
<td>30 msec - delay for disk I/O</td>
</tr>
</tbody>
</table>
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:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Element</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{13}\text{C}$</td>
</tr>
<tr>
<td>F2</td>
<td>$^{1}\text{H}$</td>
</tr>
</tbody>
</table>

Acquisition parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL1</td>
<td>f1 channel - high power level for $^{1}\text{H}$ transmitter pulse</td>
</tr>
<tr>
<td>PL2</td>
<td>f2 channel - high power level for $^{15}\text{N}$ decoupler pulse</td>
</tr>
<tr>
<td>D0</td>
<td>3 usec - incremented delay</td>
</tr>
<tr>
<td>D2</td>
<td>$1/(2J(N,H))=50$ msec, calculated from $2J(C,H)=10$ Hz</td>
</tr>
<tr>
<td>D16</td>
<td>100 usec - delay for homospoil/gradient recovery</td>
</tr>
<tr>
<td>D21</td>
<td>D16+P2+D0*2</td>
</tr>
<tr>
<td>TD2</td>
<td>1 K data points in F2</td>
</tr>
<tr>
<td>SW2</td>
<td>10 ppm</td>
</tr>
<tr>
<td>O1</td>
<td>middle of $^{1}\text{H}$ NMR spectrum</td>
</tr>
<tr>
<td>NS</td>
<td>4</td>
</tr>
</tbody>
</table>

Processing parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>SI(F2)</td>
<td>512 W</td>
</tr>
<tr>
<td>WDW(F2)</td>
<td>SINE</td>
</tr>
<tr>
<td>LB(F2)</td>
<td>2</td>
</tr>
<tr>
<td>PH-mod(F2)</td>
<td>no</td>
</tr>
<tr>
<td>MC2</td>
<td>QF</td>
</tr>
<tr>
<td>D0</td>
<td>3 usec</td>
</tr>
<tr>
<td>D1</td>
<td>2 sec</td>
</tr>
<tr>
<td>D2</td>
<td>$1/(4J(C,C))=5$ msec, calculated from $J(C,C)=50$ Hz</td>
</tr>
<tr>
<td>TD2</td>
<td>1 K data points in F2</td>
</tr>
<tr>
<td>TD1</td>
<td>128 data points in F1</td>
</tr>
<tr>
<td>SW1</td>
<td>400 ppm</td>
</tr>
<tr>
<td>O2</td>
<td>middle of $^{15}\text{N}$ NMR spectrum</td>
</tr>
<tr>
<td>DS</td>
<td>16</td>
</tr>
<tr>
<td>RG</td>
<td>receiver gain for correct ADC input</td>
</tr>
</tbody>
</table>

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 $^{12}$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

Processing parameters

- **SI(F2)**: 512 W
- **WDW(F2)**: SINE
- **LB(F2)**: 2
- **PH-mod(F2)**: no
- **MC2**: QF

- **SI(F1)**: 512 W
- **WDW(F1)**: SINE
- **SSB(F1)**: 2
- **PH-mod(F1)**: mc
- **XFB**: fourier transformation in both directions

- **plot**: use XWINPLOT

phase correction: phase correction is not necessary

---

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

**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 $^{2J}(C,H)=10$ 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

**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

**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:

<table>
<thead>
<tr>
<th>Channel</th>
<th>Setting</th>
</tr>
</thead>
<tbody>
<tr>
<td>F1</td>
<td>$^{13}$C</td>
</tr>
<tr>
<td>F2</td>
<td>$^1$H</td>
</tr>
</tbody>
</table>

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 $^{2J}(C,H)=10$ 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

**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 $^{1J}(C,C)=50$ 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
**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:  $^1$C  
F2:  $^1$H

**Acquisition parameters**

PL1 : $f_1$ channel - high power level for $^1$H transmitter pulse  
PL2 : $f_2$ channel - high power level for $^{15}$N decoupler pulse  
D0 : 3 usec - incremented delay  
D2 : $1/[2J(N,H)]=50$ msec, calculated from $^2J(C,H)=10$ 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  

**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

plot : use XWINPLOT