Which Experiment Should I Choose?

So Many to Choose From....
So Many to Choose From...
99 parameter sets, and 144 pulse sequences with "HSQC"

<table>
<thead>
<tr>
<th>Parameter Set</th>
<th>Description</th>
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<td>ea</td>
<td>phase sensitive using Echo/Antiecho method</td>
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</tbody>
</table>

The "Secret Decoder Ring"
<TopSpinHome>/exp/stand/nmr/lists/pp

"Pulprog.info"
New in TopSpin 3.0
“Show Recommended”

• “Recommended” parameter sets for some of the most commonly used Small Molecule Experiments

Not Rules Written in Stone ......
Just Things to Think About
**1H Observe**

**PROTON**
- zg30
- 1H acquire with 30° pulse
  - \( \cos(\theta) = e^{-(d1+aq)/T1} \)
  - 30° pulse is a nice compromise of signal and time for most T1 values
  - The zg pulse sequence uses a 90° pulse
- Not many options outside of D1, NS and SW/O1P
  - Keep in mind that DW=1/sw
    - Number of points stay constant, so changing sw affects the acquisition time.

**WATERSUPP**
- noesypppr1d
  - Presaturation applied during D1, and d8
    - Narrower residual water peak

**1H Observe**

Additional Parameter Sets for Automation

**CMCQ_PROTON**
- zg30
  - For quantitation purposes, so longer D1
  - AU program (cmcq_acquQuant) that does a pulse calibration on each sample

**WATER**
- zgcppr
  - Presaturation using composite 90° pulse
  - AU program (au_watersc) that does a scout scan to find the most intense signal and sets O1 there

**LC1DWTDNC**
- wetdc
  - WET with \(^{13}\text{C}\) decoupling during WET and AQ
  - AU program to automatically find solvent peaks and create the wet shape
    - Number of peaks to suppress defined by L30
**13C Observe**

- **C13CPD**
  -  zgpg30
    - 13C acquire with 30° pulse, and power gated decoupling during D1, and AQ
    - Not many options outside of D1, NS and SW/O1P
      - Keep in mind that DW=1/sw
        - Number of points stay constant, so changing sw affects the acquisition time.
- **C13DEPT135**
  - deptsp135
    - Most common DEPT experiment showing all protonated carbons
      - Uses an adiabatic 180° pulse

---

**13C Observe**

**Adiabatic Pulses**

![Adiabatic Pulses Diagram](image)

Gibberellic Acid in Acetone
13C Observe
Adiabatic Pulses

- **dept135**
- **deptsp135**

- **C13CPD**
  - **zgpg30**
    - 13C acquire with 30° pulse, and power gated decoupling during D1, and AQ
    - Not many options outside of D1, NS and SW/O1P
      - Keep in mind that DW=1/sw
        - Number of points stay constant, so changing sw affects the acquisition time.
  - **C13DEPT135**
    - **deptsp135**
      - Most common DEPT experiment showing all protonated carbons
        - Uses an adiabatic 180° pulse
- **Other Sequences**
  - **zgig30**
    - Sequence with inverse gated decoupling, so only during acquisition
  - **dept45sp**
  - **dept90sp**
**1H-1H Homonuclear 2D Experiments**

**Through Bond**

\[
\begin{array}{cccccc}
& H & & H & & H \\
C & C & C & N & C & N \\
\end{array}
\]

**COSY**

- **COSYGPSW**
  - \texttt{cosygpmpqf} -- Magnitude mode COSY (qf) with gradients (gp) and purge pulses (pp)
  - Gradient selected, so ns \( \geq 1 \)
  - Purge pulse to reduce artifacts from not waiting long enough for D1
    - D1=0.1 sec, AQ=0.8

---

**1H-1H Homonuclear 2D Experiments**

**COSY**

- **COSYGPSW**
  - \texttt{cosygpmpqf} -- Magnitude mode COSY (qf) with gradients (gp) and purge pulses (pp)
  - Gradient selected, so ns \( \geq 1 \)
  - Purge pulse to reduce artifacts from not waiting long enough for D1
    - D1=0.1 sec, AQ=0.8

---

**Caryophyllene Oxide in DMSO**

- No Purge Pulses
- With Purge Pulses
**1H-1H Homonuclear 2D Experiments**

**COSY**

- **COSYGPDFPHSW**
  - *cosygpmfpphpp* -- COSY with gradient pulses (gp), multiple quantum filter (mf), phase sensitive (ph), and purge pulses (pp)

  - Double quantum filter simplifies the diagonal
  - Phase sensitive information (active/passive coupling)

  - Difficult for a beginner to phase

---

**1H-1H Homonuclear 2D Experiments**

**Another COSY Option**

- **cosygpmfppqf** -- Magnitude mode (qf) COSY, with gradients (gp), multiple quantum filter (mf), and purge pulses (pp)

  - Double quantum filter to simplify the diagonal
  - Still magnitude mode so no phase necessary

---

Cholesterol Acetate in CDCl₃

Caryophyllene Oxide in DMSO
$^1$H-$^1$H Homonuclear 2D Experiments

Another COSY Option

+ Double quantum filter to simplify the diagonal – Especially if the window function is adjusted to bring out more signal (ssb = 4)

- CMCse_COSY
  - cosyapmfppgf

  » Because the parameter set was designed for CMCse, there is more resolution (512 increments) than other parameter sets
    - Longer experiment
    - Brings out peaks that are weakly coupled

$^1$H--$^1$H Homonuclear 2D Experiments

Through Bond

COSY

TOCSY
**1H-1H Homonuclear 2D Experiments**

**TOCSY**

- **MLEVPHSW**
  - `mlevphpp` -- Homonuclear Hartman-Hahn using MLEV17 sequence, phase sensitive (ph), and purge pulses (pp)

- **MLEVPHPR**
  - `mlevphpr.2` -- Homonuclear Hartman-Hahn using MLEV17 sequence, phase sensitive (ph), and presat (pr),

  TOCSY Mixing Time is defined by d9
  - Default is 0.08 seconds

![Strychnine in CDCl₃](image)

**1H-1H Homonuclear 2D Experiments**

**Through Bond**

- **COSY**

**Through Space**

- **NOESY**
- **ROESY**
**1H-1H Homonuclear 2D Experiments**  
**NOESY/ROESY**

- **NOESYPHSSW**
  - `noesyppphpp` -- NOESY with gradient pulses during mixing time, phase sensitive (ph), and purge pulses (pp)
    - Mixing time is defined by `d8`
    - Default is 0.3 seconds

- **ROESYPHSSW**
  - `roesypphp.2` -- ROESY sequence, phase sensitive (ph), and purge pulses (pp), using 180x-180x pulses for spin lock to suppress TOCSY artifacts (.2)
    - Mixing time is defined by `p15`
    - Default is 200 milliseconds

**Zero Crossing Depends on:**
- Magnetic Field
- Size of Molecule
- Temperature
- Viscosity

Around 1,000 – 2,000 Daltons

---

**Pamoic Acid**  
MW=388  
DMSO at 292 K

**NOESY**  
400 MHz  
**ROESY**  
400 MHz

**NOESY**  
500 MHz

**Small Molecule**  
- **NOESY**  
- **ROESY**

**Large Molecule**  
+ **NOESY**  
+ **ROESY**

**Exchange Peak**  
+ **NOESY**  
+ **ROESY**

---

High-Resolution NMR Techniques in Organic Chemistry  
Timothy D.W. Claridge 1999
1H-13C Heteronuclear 2D Experiments

**Single Bond**

\[ ^1H \quad ^{13}C \]

**HSQC**

Multiplex

Edited

Matched Sweep

Adiabatic Pulses

Shaped Pulses for Inversion (sp)

Sensitivity Improved

COSY peak Suppression

(2.3/4)

Gradients in Back INEPT (2)

Shaped Pulses for Inversion and Refocusing (.2)

Shaped Pulses for Inversion (sp)

"Bare Bones"
$^{1}$H-$^{13}$C Heteronuclear 2D Experiments

**HSQC**

- **Adiabatic Pulses**
- **Sensitivity Improved**
- **Multiplicity Edited**

$_{^{1}H-^{13}C}$ HSQC – Things to Consider

**HSQCEDETGPSISP_ADIA and HSQCETGPSISP_ADIA**

- **Bare Bones**
  - hsqcph
  - hsqgph

- **Adiabatic Pulses**
  - Shaped Pulses for Inversion (sp)
  - Shaped Pulses for Inversion and Refocusing (sp2)

- **Sensitivity Improved**
  - COSY peak Suppression (sp)
  - Gradient in Back INEPT (sp)

- **Multiplicity Edited**
  - Shaped Pulses for Inversion (sp)
  - Multinet Spectral Editing Pulse (sp1)
$^1$H-$^{13}$C HSQC – Things to Consider

Multiplicity Edited or Not?

- HSQCETGP
  - hsvcetgp
    - Simple Gradient HSQC – non Edited
- HSQCEDETGP
  - hsvcdetgp
    - Simple Multiplicity Edited Gradient HSQC

$^1$H-$^{13}$C Heteronuclear 2D Experiments

HSQC

Adiabatic Pulses

Sensitivity Improved

Multiplicity Edited
"Matched Sweep" Adiabatic Pulses
Removing the J Dependence

\[ \text{d21} = \frac{1}{2J_{zh}} \]
If \( J = 180 \text{ hz} \) \( \rightarrow \) 2.7ms
If \( J = 100 \text{ hz} \) \( \rightarrow \) 5 ms

The Matched Sweep Adiabatic Pulse
Sweeps through the \(^{13}\text{C}\) frequency range so that it inverts signals closer to when the time matches the \( \frac{1}{2J} \) condition

\(^1\text{H}-^{13}\text{C}\) HSQC – Things to Consider
Multiplicity Edited or Not?

- \text{hsqcetgp}
- \text{hsqcedetgp}
- \text{hsqcedetgpsp.3}

Menthyl Anthranilate in DMSO
\(^{1}\text{H}-^{13}\text{C} \) HSQC – Things to Consider

Multiplicity Edited or Not?

- **HSQCEDETGPSISP\_ADIA**
  - `hsqcedetgpsisp2.3` w/ `bi_p5m4sp_4sp.2` decoupling
    - Multiplicity Edited (ed)
      + You get the DEPT type information in addition to the \(^{1}\text{H}-^{13}\text{C}\) connectivity
    - Adiabatic Pulses (sp) – Including a Matched Sweep Adiabatic (.3)
      + No significant loss in sensitivity
    - Sensitivity Improved (si)

- **HSQCETGPSISP\_ADIA**
  - `hsqcetgpsisp2.2` w/ `bi_p5m4sp_4sp.2` decoupling
    - Not Multiplicity Edited
      + Simple, all peaks are Positive
    - Adiabatic Pulses (sp) – for both Inversion and Recovery (.2)
    - Sensitivity Improved (si)

\[d_{21} = \frac{1}{2J_{\text{HH}}} = 3.6 \text{ ms}\]

\[\delta = \text{gradient recovery delay} = 0.2\text{ms}\]

\(~ 7 \text{ ms longer of a sequence~}\)

Depending on the \(T_2\) relaxation rates of the molecule the non-edited version might be more sensitive:

But is it worth sacrificing the multiplicity information?
1H-13C HSQC – Things to Consider
Multiplicity Edited or Not?

Multiplicity Editing: 
~ 7 ms longer of a sequence

1 mg/ml Quinidine
1st fid from an HSQC

1 mg/ml Quinidine, 1 hour 20 Min each HSQC w/ 9 hour DEPT as projection
1H-13C HSQC – Things to Consider

Multiplicity Edited or Not?

0.1 mg/ml Quinidine, 10 hour each HSQC spectra w/ no DEPT

1H-13C HSQC – Things to Consider

Matched Sweep Adiabatic Pulse?
1H-13C HSQC – Things to Consider
Benefit of Matched Sweep

**Quinidine in DMSO**

\[d_{21} = \frac{1}{2J_{\text{ch}}},\]

- If \( J = 180 \text{ hz} \rightarrow 2.7 \text{ ms} \)
- If \( J = 100 \text{ hz} \rightarrow 5 \text{ ms} \)

**The Matched Sweep Adiabatic Pulse**

Sweeps through the 13C frequency range so that it inverts signals closer to when the time matches the 1/2J condition.
**1H-13C HSQC – Things to Consider**

**Matched Sweep Adiabatic Pulse?**

- **hsqcedetgpsisp2.3**
  - Multiplicity Edited
  - Matched Sweep Adiabatic Pulse
    - + Works well when J scales with Chemical Shift
    - − Problematic when J differs

- **hsqcedetgpsisp2.2**
  - Multiplicity Edited
  - Regular Adiabatic Pulses
    - + Less Sensitive to deviations in J
    - − No benefit from the matched sweep for “normal” resonances

\[ J_{hc} = 158 \text{ Hz} \]

α-Thujone in DMSO
**1H-13C HSQC – Things to Consider**

**Sensitivity Improved or Not?**

- **HSQCEDETGPSISP_ADIA**
  - hsqcedetgpsisp2.3
- **HSQCETGPSISP_ADIA**
  - hsqcetgpsisp2.2
  - Sensitivity Improved Element
    - Possible sensitivity improvement of \( \sim \sqrt{2} \)
- **HSQCEDETGPSP.3_ADIA**
  - hsqcedetgpssp.3
  - Multiplicity edited with Matched Sweep Adiabatic
- **HSQCETGPSP.2_ADIA**
  - hsqcetgpssp.2
  - Non Multiplicity Edited
  - No Sensitivity Improved Element
    - In general, less sensitive than the SI version
Strychnine in CDCl₃

$^{1}H-^{13}C$ HSQC – Things to Consider
Sensitivity Improved or Not?

$\text{d24} = \frac{1}{8J_{\text{HH}}} = 0.89m$

~2ms longer of a sequence

Depending on the $T_2$ relaxation rates of the molecule of interest, the non-si version might be actually be more sensitive.
$^{1}H - ^{13}C$ HSQC – Things to Consider
Sensitivity Improved or not?

d$24 = 1/8J_{xh}$
d$21 = 1/2J_{xh}$

Matched Sweep Adiabatic Pulses can be use (p31,sp10) to compensate for $J_{xh}$ in d21.

But no compensation available for $J_{xh}$ in d24

Strychnine in CDCl$_3$
1H-13C HSQC – Things to Consider
Sensitivity Improved or Not?

- **HSQCEDETGPSISP_ADIA**
  - hsqcdetgpsisp2.3
    - Multiplicity Edited
    - "Sensitivity Improved" INEPT element
    - Matched Sweep Adiabatic Pulses
      - + More Sensitive
      - - Non quantitative

- **HSQCEDETGPSP.3_ADIA** or CMCse_HSQC
  - hsqcedtgssp.3
    - Multiplicity Edited
    - Without “Sensitivity Improved” INEPT element
    - Matched Sweep Adiabatic Pulses
      - - Less Sensitive
      - + Quantitative integrals
        - Used in CMCse

1H-13C HSQC – Things to Consider
COSY Peak Suppression

- Bare Bones
- hsqph
- hsqppph
- Adiabatic Pulses
- Sensitivity Improved
- Multiplicity Edited
- Shaped Pulses for Inversion (2)
- Shaped Pulses for Inversion and Refocusing (1,2)
- Gradient in Block INEPT (2,4)
- COSY peak Suppression (2,4)
- Shaped Pulses for Inversion (4)
- Matched Sweep Adiabatic Pulses (4)
**$^1$H-$^{13}$C HSQC – Things to Consider**

**COSY Peak Suppression**

- **hsqcedetgpsisp2.4**
  - Sensitivity Improved
  - Multiplicity Edited
  - Matched Sweep
  - COSY Suppression

- **hsqctgpsisp2.3**
  - Sensitivity Improved
  - Non Multiplicity Edited
  - COSY Suppression

+ Removes the COSY artifacts that arise when using the “si” versions
- Less sensitive than regular “si” versions

---

**Menthyl Anthranilate in DMSO**

---

**Menthyl Anthranilate in DMSO**
1H-13C HSQC – Things to Consider

Long Refocusing Pulse

Adiabatic Pulses:
- Inversion (p14) = 0.5 ms
- Refocusing (p24) = 2 ms

Hard 180 Pulse:
- 16 us

Sensitivity Improved

Multiplicity Edited

13C Labeled Sucrose
**1H-13C HSQC – Things to Consider**

**When is Simple Better?**

- Adiabatic Pulses
- Sensitivity Improved
- Multiplicity Edited

- Shape then Pulse for Inversion (sp)
- Shape then Pulse for Refocusing (2,2)
- Gradient in Black (INEPT 2,2)
- Shape then Pulse for Inversion (sp)
- Matched Sequence Adiabatic Pulses (1,2)

---

**d21 = 1/2Jxh = 3.6 ms**

**d24 = 1/8Jxh = 0.89 ms**

**Adiabatic pulses:**

- Inversion = .5 ms
- Refocusing = 2 ms

**Hard 180 Pulse:**

- 16 us
HSQC – Things to Consider
When Is Simple Better?

$^1\text{H} - ^{11}\text{B}$ Spectra

$^1\text{H} - ^{13}\text{C}$ Heteronuclear 2D Experiments

Single Bond
HSQC/HMQC

Multiple Bond
HMBC
**1H-13C Heteronuclear 2D Experiments**

**HMBC**

- **HMBCGP**
  - hmbcgplpndqf
    - Gradients for coherence selection (gp)
    - Low pass filter (lp)
    - No decoupling during acquisition (nd)
    - Magnitude Mode (qf)
      - Simple
      - No 180° pulses

- **HMBCETGPL3ND**
  - hmbcetapl3nd
    - Echo Anti Echo (et)
    - Gradients for coherence selection (gp)
    - 3rd order Low Pass filter (l3)
      - Better suppression of 1J correlation peaks
      - More sensitive because of Echo Anti Echo Detection
      - More difficult to process (xfb + xf2m)

**1H-13C Heteronuclear 2D Experiments**

**HMBC - Sensitivity**

Quinidine in DMSO 1 mg/ ml

32 Scans, 256 Increments = 6 hours each
**1H-13C Heteronuclear 2D Experiments**

**HMBC – Low Pass Filter**

- \( d_2 = \frac{1}{2J_{xh}} \)

**hmbcgplpndqf**

- \( \Delta_1 = \frac{1}{2(J_{xh-min} + 0.07(J_{xh-max} - J_{xh-min}))} \)
- \( \Delta_2 = \frac{1}{J_{xh-min} + J_{xh-max}} \)
- \( \Delta_3 = \frac{1}{2(J_{xh-max} - 0.07(J_{xh-max} - J_{xh-min}))} \)

**hmbcetgp3nld**

**1H-13C Heteronuclear 2D Experiments**

**HMBC – Suppression of \( ^1J \) correlations**

- \( ^1J_{xh} = 145 \text{ Hz} \)
- \( ^1J_{xh \text{ (max)}} = 170 \text{ Hz} \)
- \( ^1J_{xh \text{ (min)}} = 120 \text{ Hz} \)
- \( ^1J_{xh} = 145 \text{ Hz} \)

**Gibberellic Acid in Acetone**

**Long Range \( J_{xh} \)**

- \( 8 \text{ Hz} \)
**$^{1}H-^{13}C$ Heteronuclear 2D Experiments**

Another HMBC option

- **hmbcetgpnd**
  - Gradients for coherence selection
  - Echo Anti Echo
  - Similar sensitivity to hmbcetgpl3nd
  - No Low Pass filter
  - $^{1}J$ correlations are often useful when interpreting the data instead of the HSQC

![Quinidine in DMSO](image)

Quinidine in DMSO
$1 \text{ mg/ ml}$

**Heteronuclear 2D Experiments**

Not Just $^{13}C$ – $^{1}H/^{15}N$ also

- **HMBCGP\_15N**
  - **hmbcgpndaf**
    - $^{15}N$ is routed through f2
    - Gradients for coherence selection
  - Ratio set to select for $^{1}H/^{15}N$ instead of $^{1}H/^{13}C$
  - Other nuclei are possible with the AU program "gradratio"

- **HSQCETGP\_15N**
  - **hsqcetpsi2**
    - $^{15}N$ is routed through f2
    - Echo-anti echo
    - Sensitivity improved
    - Gradients in the back inept
    - Gradients for coherence selection
  - Ratio set to select for $^{1}H/^{15}N$
**1H-13C Heteronuclear 2D Experiments**

**HSQC_TOCSY_ADIA**

- **HSQC_TOCSY_ADIA**
  - hsqcdietgpsisp.2
  - DIPS12 for Hartman-Hahn Mixing
  - Using adiabatic pulses
  - Sensitivity Improved
  - All Peaks Positive

- **Other Options**
  - hsqcdietgpsisp.3
  - Inversion of directly coupled protons
    - "HSQC" are +
    - "TOCSY" are -
  - Fully Edited
    - "HSQC" → CH/CH₃ + & CH₂ -
    - "TOCSY" → CH/CH₃ - & CH₂ +

---

**1H-13C Heteronuclear 2D Experiments**

**HSQC_TOCSY_ADIA**

Menthyl Anthranilate in DMSO

TOCSY

HSQC
1H-13C Heteronuclear 2D Experiments
HSQC_TOCSY_ADIA

New in TopSpin 3.0
“Show Recommended”

But There’s More If These Don’t Answer Your Question
**1H-13C Heteronuclear 2D Experiments**

**HMBC**

Strychnine in CDCl₃

**HSQC**

**HMBC**

How Do I Know $^2J$ vs $^3J$?

**H2BC (AKA HMQC-COSY)**

Heteronuclear 2 Bond Correlation

Strychnine in CDCl₃

**HSQC**

**h2bcetgpl3**
**Experimental Details**

### Advantages of the H2BC:

- It helps solve the problem of distinguishing two- and three-bond correlations in HMBC or HSQC-TOCSY
- Is independent of occasionally vanishing $^2J_{CH}$ coupling constants, which alleviates the problem of missing two-bond correlations in HMBC spectra

### Disadvantages of the H2BC:

- Only protonated carbons are observed (no $^4\beta$)
- Relies on $^3J_{HH}$ to get “2 Bond” correlations
  - $^4J_{HH}$ Couplings are not uncommon, and if large enough (>1Hz) will also be observed
- No Parameter Set in TopSpin
  - Contact the Applab, we do have one
- Pulse Sequence → h2bcetgpl3
- Processing → xfb + xf2m

---

**INADEQUATE**

### Advantages:

- Information rich!

### Disadvantages:

- Insensitive
- Relies on $^{13}$C next to another $^{13}$C
- 100 mg/ml Strychnine on a RT 400 MHz BBFO Smart Probe → 2.5 DAYS
- Single Scan 1D-$^{13}$C S:N of 100:1
**Experimental Details**

- **Pulse Sequence:**
  - inadphsp

- **Experimental Details:**
  - SW in F2 = $^{13}$C Spectrum
  - SW in F1 = 2 x $^{13}$C SW in F2

- **Referencing:**
  - Center of spectrum in F1 = 2x O1p

---

**How to Interpret**

Chemical Shift in Indirect Dimension = $C_a + C_b$

15→14→13→8
15→16→7→8
13→12→11
17→18
INADEQUATE

Benefit of Phase Sensitive

Chemical Shift in Indirect Dimension = C_a + C_b

Correlation at 51.8 ppm and 95 ppm
C7 = 51.9 ppm + C17 = 42.9 ppm –> 94.8

INADEQUATE

Experimental Details - Folding

- Pulse Sequence:
  - inadphsp
- Experimental Details:
  - SW in F2 & F1 = 13C Spectrum
- Referencing:
  - Center of spectrum in F1 = 2x O1p
- Position of Folded Peaks = SW + C_a + C_b
INADEQUATE
Benefit of Folding

Chemical Shift in Indirect Dimension = SW + C_a + C_b

Correlation at 51.9 ppm and 255 ppm
C7 = 51.9 ppm + C17 = 42.9 ppm + SW = 160 ppm → 254.8

ADEQUATE
Proton Detected $^{13}$C-$^{13}$C Correlations
Adequate Proton Detected $^{13}$C-$^{13}$C Correlations

1,1-ADQUATE

adeq11etgpsp

50 mg/ml Strychnine in CDCl$_3$

Room Temp 400 MHz BBFO

Smart Probe $\rightarrow$ 16 hours

Correlation at $H_a$ / $C_a$ + $C_b$

From HSQC $\rightarrow$ $H_4 = 8.1$ ppm $C_4 = 116.17$ ppm

Adequate Peaks at 244.5 ppm and 258.3 ppm

$C_4$ Next to Carbons at 127.8 (C3) and 142.13 (C5)

Adequate Proton Detected $^{13}$C-$^{13}$C Correlations

1,n-ADQUATE

adeq1netgpsp

50 mg/ml Strychnine in CDCl$_3$

500 MHz Prodigy $\rightarrow$ 4 days 4 Hours

Correlation at $H_a$ / $C_a$ + $C_b$

From HSQC $\rightarrow$ $H_4 = 8.1$ ppm $C_4 = 116.17$ ppm

Adequate Peaks at 238.4 ppm and 244.7 ppm

$C_4$ Next to Carbons at 124.0 (C1) and 132.67 (C3)
ADEQUATE
Proton Detected \(^{13}\text{C}-^{13}\text{C}\) Correlations

Refocused 1,1-ADQUATE
adeq11etgprdsp.2

50 mg/ml Strychnine in CDCl3
Room Temp 400 MHz BBFO
Smart Probe → 16 hours

Correlation at \(H_3 / C_8\)
Can Interpret like an HMBC/H2BC
Know it is Neighboring \(^{13}\text{C} (J_{CC})\)
Unlike H2BC – correlations to 4 Carbons are possible