



KJM 9250

^{13}C Detected XHDEPT, HETCOR and COLOC
Experiments on the AVIIIHD-800 Spectrometer

Version 5.0

Topspin 3.5

Windows 7



© Professor Emeritus Alistair Lawrence Wilkins,
University of Waikato, New Zealand.
January 2018

¹³C Detected XHDEPT, HETCOR and COLOC Experiments on the AVIIIHD-800 Spectrometer

1.0 Introduction

¹³C detected aw coded **XHDEPT, HETCOR and COLOC** parameter sets are set up with 1K or 2K acquired ¹³C points and 128 to 256 ¹H increments. ¹H and ¹³C spectral windows and their midpoints should be determined before setting up these experiments.

1.1 Processing

HXDEPT45, HXDEPT90 and HXDEPT135 experiments are phase sensitive experiments Which should be phased **before** using the **abs1** and **abs2** commands.

The **HXDEPTQF, HETCOR and COLOC** experiments are absolute value experiments. Phasing is not required.

2.0 Experiments and Parameter Sets

The following ¹H detected **HXDEPT, HETCOR and COLOC** experiments and parameter sets have been set up on the AVIIIHD-800 MHz spectrometer.

- 2.1 HXDEPT45, HXDEPT90 and HXDEPT135** spectra
- 2.2 HXDEPTQF** spectrum
- 2.3 HETCOR (= HXCOQF)** spectrum
- 2.4 HETCORLR** spectrum
- 2.5 COLOCQF** spectrum with manual or auto calculation of **d6** and **d18**.

2.1 HXDEPT45, HXDEPT90 and HXDEPT135 spectra

Parameter sets: **awhxdept45**, **awhxdept90** or **awhxdept135** (+ **getprosol**)

Pulse programme: **hxdept**

Type **eda** (enter) and enter **SW (¹H)** and **SW (¹³C)** in ppm.

Enter **O1P** = ¹³C spectral window midpoint in ppm.

Enter **O2P** = ¹H spectral window midpoint in ppm.

TD(F2) = 1K or 2K, **TD(F1)** = 128-256 (your choice).

NS = 8, 16 (multiple of 4 or 8 recommended), **DS** = 8 or 16.

D1 = repetition delay = **1.5 sec** or other time of your choice.

CNST12 = **0.5**, **1.0** or **1.5** for **P0** = **45°**, **90°** or **135°** as saved in parameter sets.

CNST2 = ¹J coupling constant = **145 Hz** or other value of your choice (eg 125-160 Hz).

Type **ased** (enter) and review parameters used in the job.

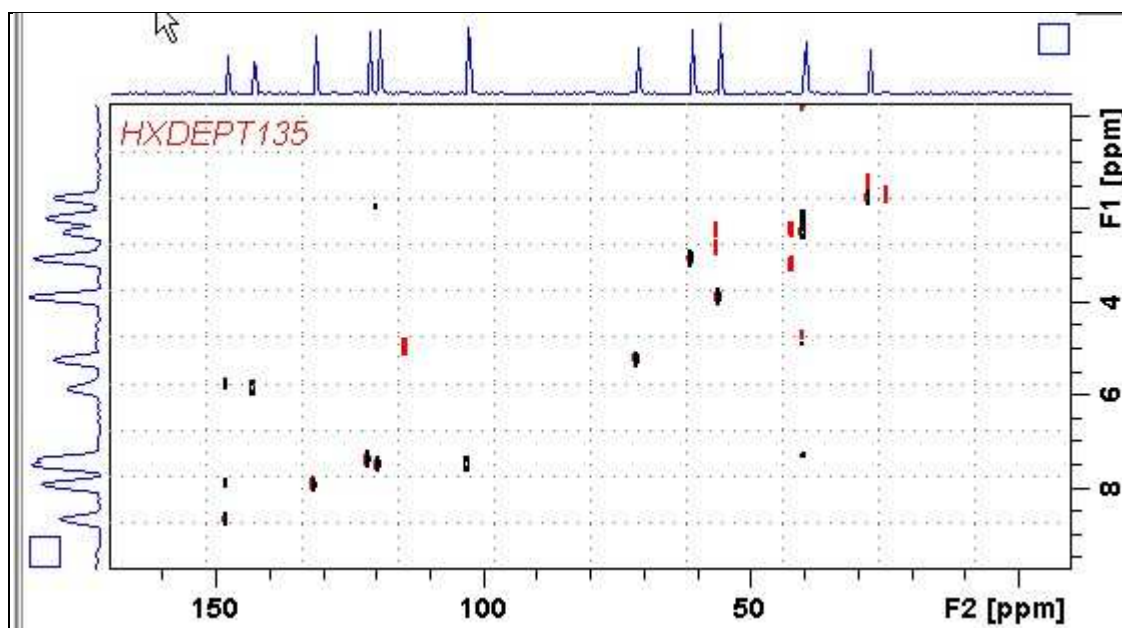
Set **receiver gain** using **RGA** (*Important!*).

Process with: **SI(F2)** = **SI(F1)** = **1K** or **2K**

WDW(F1) = **WDW(F2)** = **QSINE**

SSB(F2) = **SSB(F1)** = **2**

xfb, **abs1** and **abs2**



800 MHz **HXDEPT135** spectrum of quinine in D₆-DMSO.

Some low level artifact peaks of unknown origin may be observed in HXDEPT experiments.

2.2 HXDEPTQF spectrum

Parameter sets: **awhxdeptqf** (+ **getprosol**)

Pulse programme: **hxdeptqf**

Type **eda** (enter) and enter **SW (¹H)** and **SW (¹³C)** in ppm.

Enter **O1P** = ¹³C spectral window midpoint in ppm.

Enter **O2P** = ¹H spectral window midpoint in ppm.

TD(F2) = 1K or 2K, **TD(F1)** = 128-256 (your choice).

NS = 8, 16 (multiple of 4 or 8 recommended), **DS** = 8 or 16.

D1 = repetition delay = **1.5 sec** or other time of your choice.

CNST2 = ¹J coupling constant = **145 Hz** or other value of your choice (eg 125-160 Hz).

CNST12 = **0.5** for DEPT45-like outcome.

Type **ased** (enter) and review parameters used in the job.

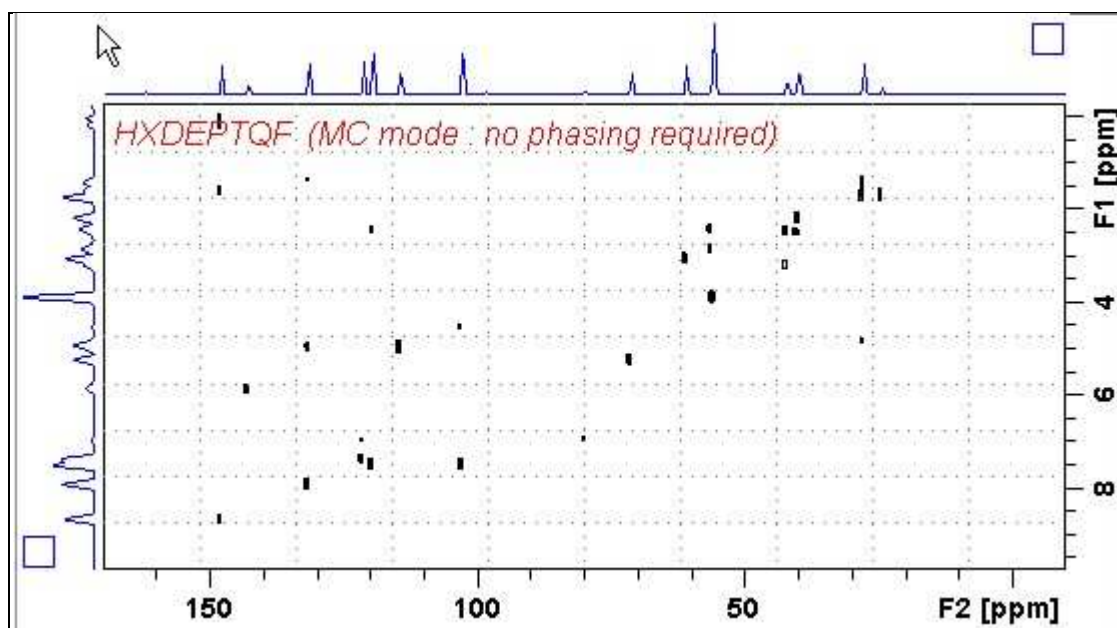
Set **receiver gain** using **RGA** (*Important!*).

Process with: **SI(F2)** = **SI(F1)** = **1K** or **2K**

WDW(F1) = **WDW(F2)** = **SINE**

SSB(F2) = **SSB(F1)** = **0**

xfb, abs1 and abs2



800 MHz **HXDEPTQF** spectrum of quinine in D₆-DMSO.

Some low level artifact peaks of unknown origin may be observed in HXDEPT experiments.

2.3 HETCOR spectrum

Parameter set: **awhetcor (+ getprosol)**

Pulse programme: **hxcoqf**

Type **eda** (enter) and enter **SW (¹H)** and **SW (¹³C)** in ppm.

Enter **O1P** = ¹³C spectral window midpoint in ppm.

Enter **O2P** = ¹H spectral window midpoint in ppm.

TD(F2) = 1K or 2K, **TD(F1)** = 128-256 (your choice).

NS = 8, 16 (multiple of 4 or 8 recommended), **DS** = 8 or 16.

D1 = repetition delay = **1.5 sec** or other time of your choice.

CNST2 = ¹J coupling constant = **145 Hz** or other value of your choice (eg: 125-160Hz).

CNST11 = **3** (used to auto calculate **D3**).

Type **ased** (enter) and review parameters used in the job.

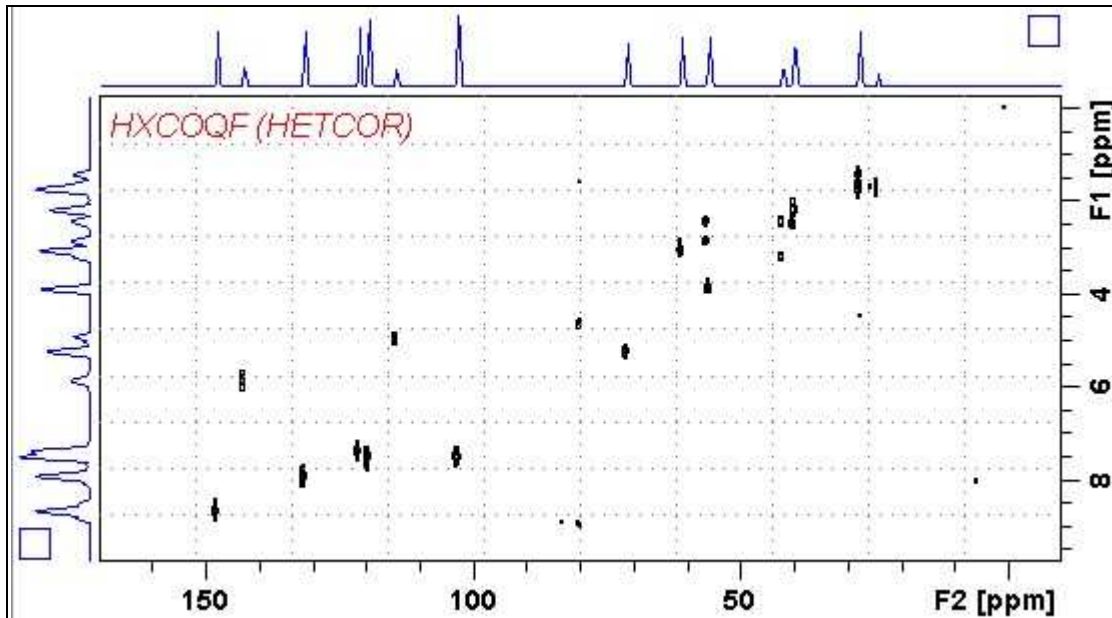
Set **receiver gain** using **RGA** (*Important!*).

Process with: **SI(F2) = SI(F1) = 1K or 2K**

WDW(F1) = WDW(F2) = SINE

SSB(F2) = SSB(F1) = 0

xfb, abs1 and abs2



800 MHz **HETCOR** spectrum of quinine in D₆-DMSO.

2.4 HETCORLR spectrum

Parameter sets: **awhetcorlr** (+ **getprosol**)

Pulse programme: **hxcoqf**

Type **eda** (enter) and enter **SW (¹H)** and **SW (¹³C)** in ppm.

Enter **O1P** = ¹³C spectral window midpoint in ppm.

Enter **O2P** = ¹H spectral window midpoint in ppm.

TD(F2) = 1K or 2K, **TD(F1)** = 128-256 (your choice).

NS = 8, 16 (multiple of 4 or 8 recommended), **DS** = 8 or 16.

D1 = repetition delay = **1.5 sec** or other time of your choice.

CNST2 = ⁿJ coupling constant = **10 Hz** or other value of your choice (eg: 6-14 Hz).

CNST11 = **3** (used to auto calculate **D3**).

Type **ased** (enter) and review parameters used in the job.

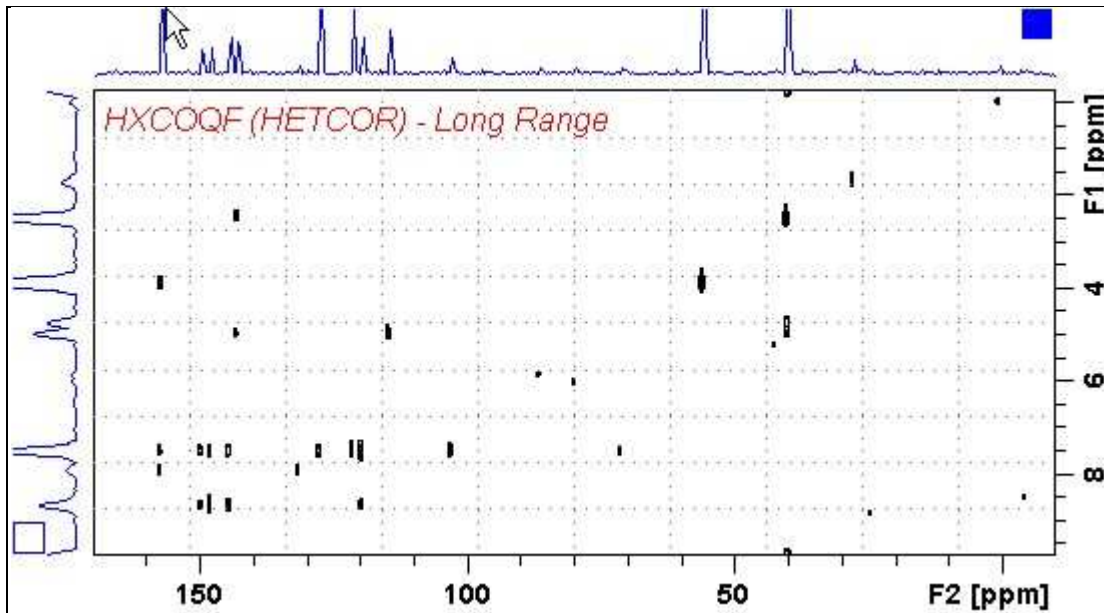
Set **receiver gain** using **RGA** (*Important!*).

Process with: **SI(F2)** = **SI(F1)** = **1K** or **2K**

WDW(F1) = **WDW(F2)** = **SINE**

SSB(F2) = **SSB(F1)** = **0**

xfb, **abs1** and **abs2**



800 MHz **HETCORLR** spectrum of quinine in D₆-DMSO optimized for ⁿJ = 10 Hz (**D2** = 50 msec, **D3** = 33 msec).

2.5.1 COLOC spectrum

Parameter sets: **awcoloc** (+ **getprosol**)

Pulse programme: **colocqf**

Type **eda** (enter) and enter **SW (¹H)** and **SW (¹³C)** in ppm.

Enter **O1P** = ¹³C spectral window midpoint in ppm.

Enter **O2P** = ¹H spectral window midpoint in ppm.

TD(F2) = 1K or 2K, **TD(F1)** = 128-256 (your choice).

NS = 8, 16 (multiple of 4 or 8 recommended), **DS** = 8 or 16.

D1 = repetition delay = **1.5 sec** or other time of your choice.

D6 = 50 msec or other delay for ⁿJ evolution [D6= 1000/(2* ⁿJ) msec].

D18 = 30 msec or other delay for ⁿJ evolution [D18= 1000/(3* ⁿJ) msec].

Parameter set **D6** and **D18** delay values are those for ⁿJ = ~10 Hz.

Type **ased** (enter) and review parameters used in the job.

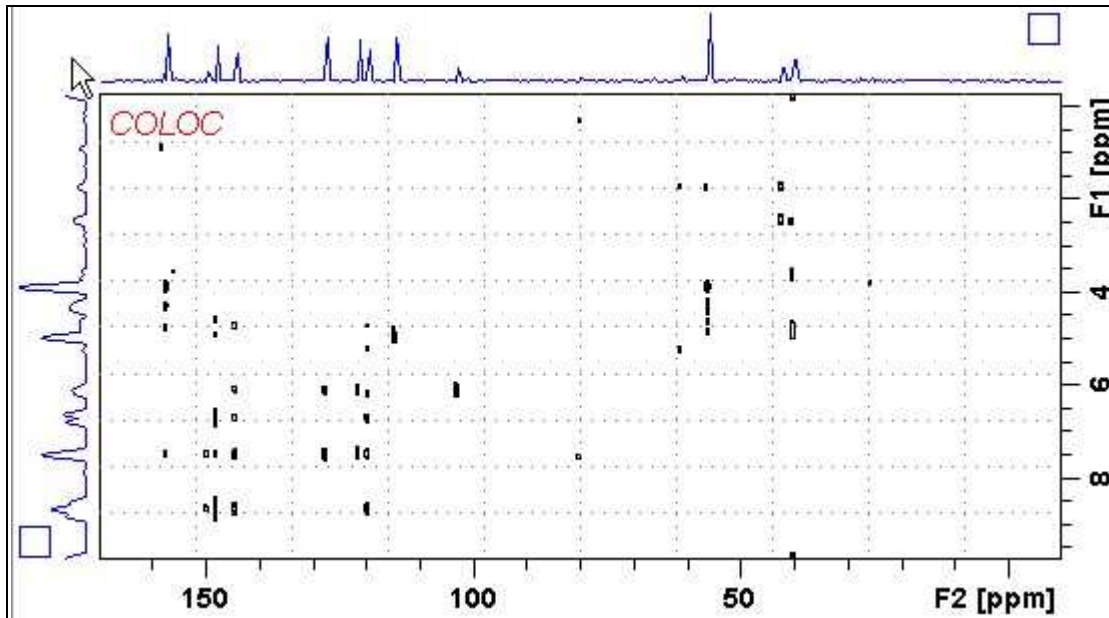
Set **receiver gain** using **RGA** (*Important!*).

Process with: **SI(F2) = SI(F1) = 1K** or **2K**

WDW(F1) = WDW(F2) = SINE

SSB(F2) = SSB(F1) = 0

xfb, abs1 and abs2



800 MHz **COLOC** spectrum of quinine in D₆-DMSO optimized for ⁿJ = ~10 Hz (**D6** = 50 msec, **D18** = 30 msec).

2.5.2 AWCOLORCQF spectrum

Parameter set: **awcoloclr** (+ **getprosol**)

Pulse programme: **awcolocqf**

d6 and **d18** are auto calculated from J_{LR} entered as **cnst21**

Type **eda** (enter) and enter **SW** (^1H) and **SW** (^{13}C) in ppm.

Enter **O1P** = ^{13}C spectral window midpoint in ppm.

Enter **O2P** = ^1H spectral window midpoint in ppm.

TD(F2) = 1K or 2K, **TD(F1)** = 128-256 (your choice).

NS = 8, 16 (multiple of 4 or 8 recommended), **DS** = 8 or 16.

D1 = repetition delay = **1.5 sec** or other time of your choice.

CNST21 = J_{LR} = **10 Hz** or other value of your choice.

D6 = $1000/(2 \cdot J)$ msec and **D18** = $1000/(3 \cdot J)$ msec are auto calculated from **CNST21**

Type **ased** (enter) and review parameters used in the job.

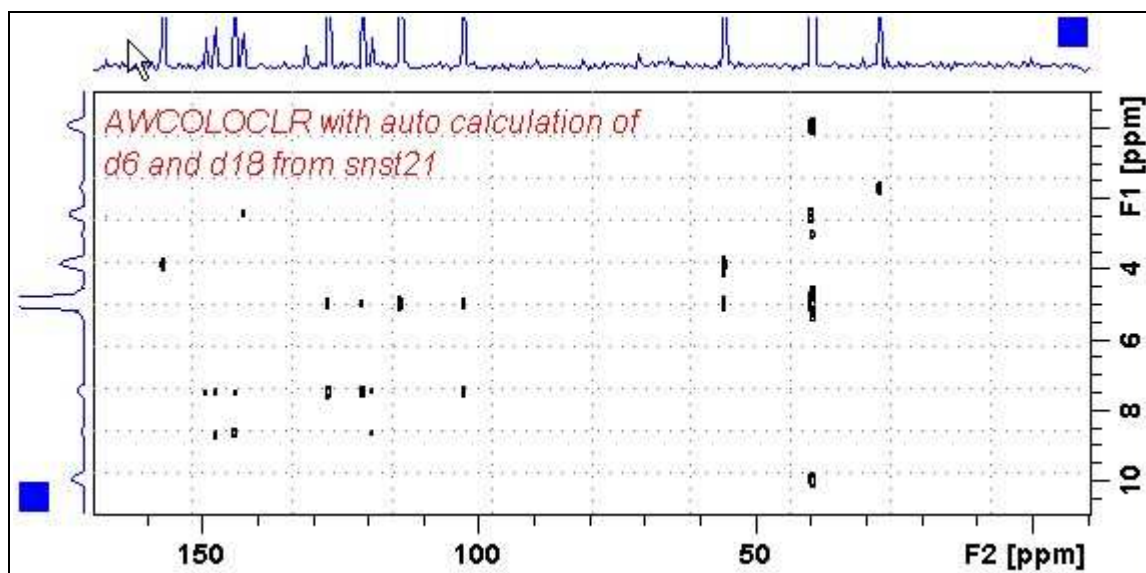
Set **receiver gain** using **RGA** (*Important!*).

Process with: **SI(F2)** = **SI(F1)** = **1K** or **2K**

WDW(F1) = **WDW(F2)** = **SINE**

SSB(F2) = **SSB(F1)** = **0**

xfb, **abs1** and **abs2**



800 MHz AWCOLORCQF spectrum of quinine in D_6 -DMSO with **D6** and **D18** auto calculated from **CNST21** = J_{LR} 10 Hz. (**D6** = 50 msec, **D18** = 33 msec).