KJM 9250

AVIII HD 800 ^{13}C NMR spectra

Version 5.0

Topspin 3.5 Windows 7 AVIII HD 800

AVIIIHD-800 ^{13}C NMR spectra

1.0 Introduction

aw coded 800 MHz ^{13}C parameter files generally have 64 K points across a 240 ppm window.

^{13}C spectra acquired in **DIGITAL** mode require a large negative **PHC1** value.
^{13}C spectra acquired in **BASEOPT** mode have normal **PHC0** and **PHC1** constants.
An experiment's acquisition mode can be viewed via **eda.**

Quaternary carbons *may have moderate to long T_1's. If saturation is suspected* ***D1*** *should be increased from its default time.*

Shaped pulse DEPT, INEPT, JMOD and APT spectra have wider flat topped spectra windows that standard (non-shaped pulse) versions. This is most noticeable for signals within 0-20 ppm ppm of the high and low field sides of 240 ppm window ^{13}C spectra, especially so in INEPT,JMOD and APT spectra.

When processed with **EF** or **EFP** a default **LB** of **1 or 2 Hz** will be applied.

2.0 ^{13}C NMR Spectra

2.1 ^{13}C NMR spectra with power gated ^{1}H decoupling and NOE

2.2 Inverse gated ^{13}C NMR spectra (no NOE)

2.3 Coupled ^{13}C NMR spectrum with NOE

2.4 ZRESTSE, ZRESTSEIG and ZRESTSEND spectra

2.5 DEPT45, DEPT90 and DEPT135 spectra
DEPT45SP, DEPT90SP and DEPT135SP spectra
Coupled DEPT spectra

2.6 DEPTQ135 spectrum with quaternary carbons
DEPTQ quaternary (singlet) carbons only spectrum

2.7 INEPT spectra
INEPTSP spectra
Coupled INEPT spectra

2.8 JMOD spectra
JMODSP spectra

2.9 APT spectra
APTSP spectra

2.10 ^{13}C NMR Spectra acquired in BASEOPT mode

2.1 ^{13}C Spectra using a 30º, 45º, 70º or 90º pulse

Parameter sets: **awcarbon30, awcarbon45, awcarbon70** or **awcarbon90 (+ getprosol)**
Pulse programmes: **zgpg30, awzgpg45, awzgpg70** or **zgpg** respectively.
Spectra are ^{1}H decoupled with NOE.

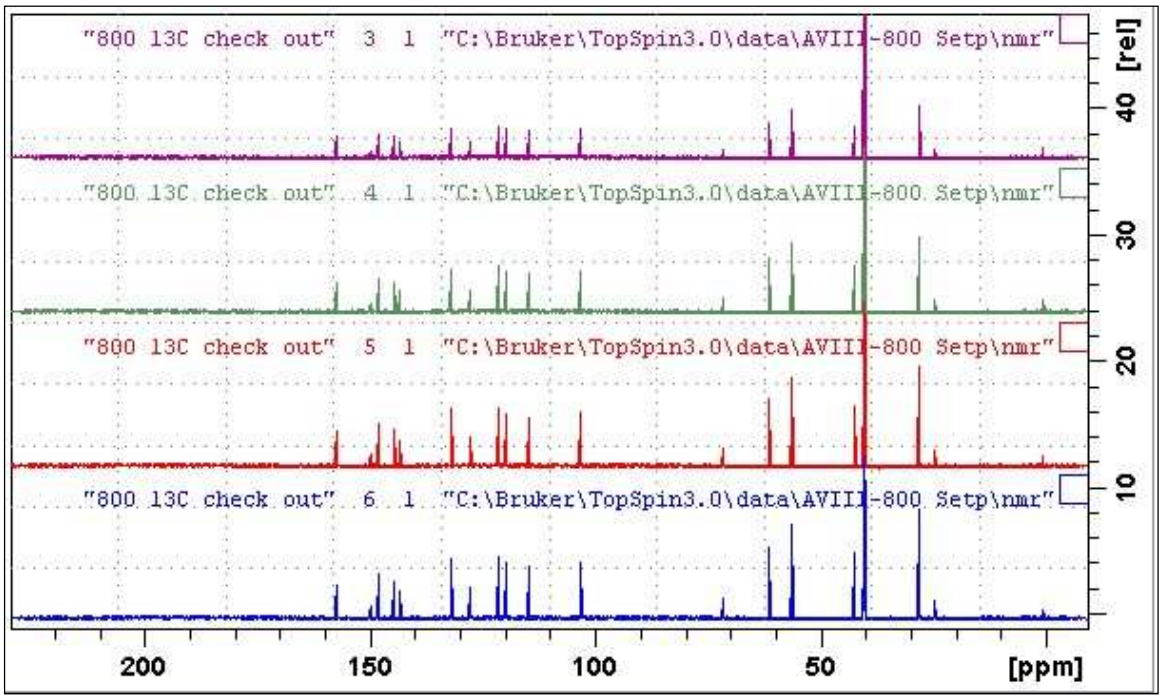
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.
NS = Any number, **DS** = 4, 8 or 16.

D1 = 2 sec or other time of your choice .
DE = 50 usec will reduce but not eliminate baseline roll in cyroprobe ^{13}C spectra.

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

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

Processing
Process with **EF** or **EFP** (applies **LB**). A large **PHC1** phase correction (eg = - 900º) will be required. Residual baseline roll can be eliminated using the **multiabsn** macro with **n = 30-50**.



^{13}C NMR spectra of quinine in D_6-DMSO. ***Lower:*** ^{13}C spectrum with a 90º pulse. ***2nd spectrum*****:** ^{13}C spectrum with a 70º pulse. ***3rd spectrum:*** ^{13}C spectrum with a 45º pulse. ***Upper:*** ^{13}C spectrum with a 30º pulse

2.2 Inverse gated ^{13}C spectra

Parameter sets: **awcarbon30ig** or **awcarbon70ig (+ getprosol)**
Pulse programmes: **zgig30** or **awzgig70** respective;y
Spectra are ^{1}H decoupled *without* NOE

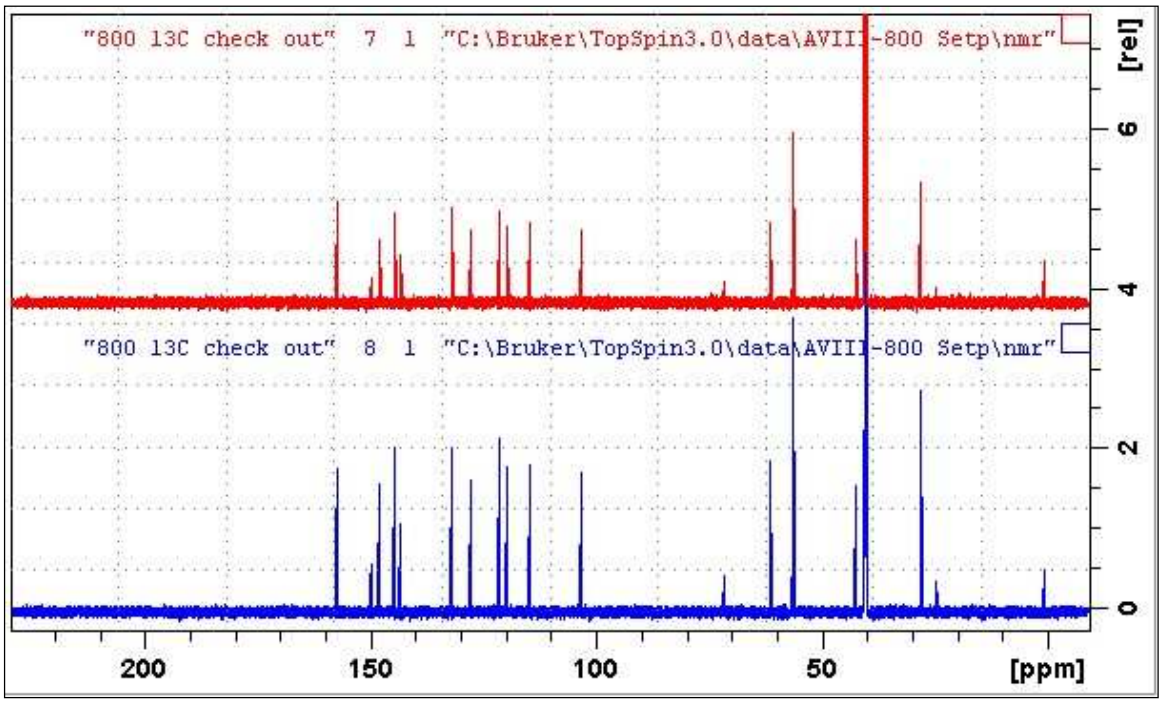
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.
NS = Any number, **DS** = 4, 8 or 16.

D1 = 2 sec or other time of your choice.
DE = 50 usec will reduce but not eliminate baseline roll in cyroprobe ^{13}C spectra.

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

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

Processing
Process with **EF** or **EFP** (applies **LB**). A large **PHC1** phase correction (eg = - 900°) will be required. Residual baseline roll can be eliminated using the **multiabsn** macro with **n = 30-50**.



Inverse gated ^{13}C NMR spectra of quinine in D_6-DMSO. ***Lower:*** Inverse gated ^{13}C spectrum with a 70° pulse ***Upper:*** Inverse gated ^{13}C spectrum with a 30° pulse.

2.3 ^{1}H coupled ^{13}C spectrum with NOE using a 70^{o} pulse

Parameter set: **awcarbon70nd (+ getprosol)**
Pulse programme: **awzg70nd**

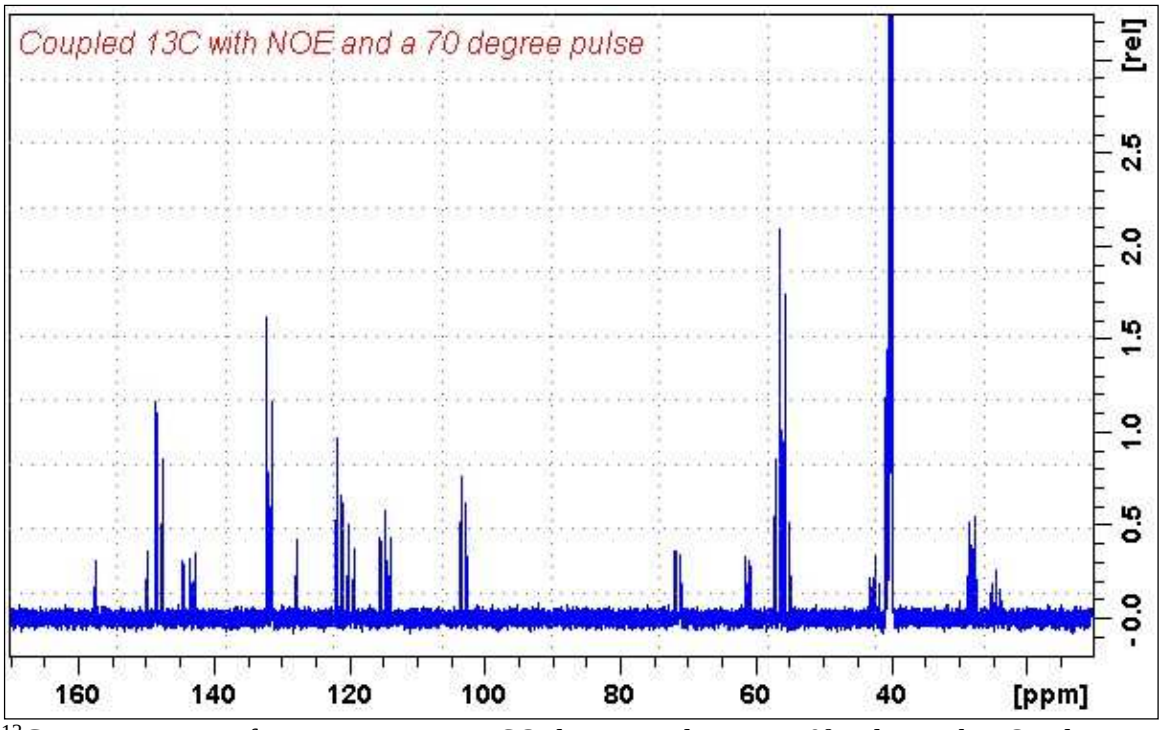
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.
NS = Any number, **DS** = 4, 8 or 16.

D1 = 2 sec or other time of your choice.
DE = 50 usec will reduce but not eliminate baseline roll in cyroprobe ^{13}C spectra.

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

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

Processing
Process with **EF** or **EFP** (applies **LB**). A large **PHC1** phase correction (eg = - 900^{o}) will be required. Residual baseline roll can be eliminated using the **multiabsn** macro with **n = 30-50**.



^{13}C NMR spectra of quinine in D_6-DMSO determined using a70^{o} pulse with NOE during D1.

2.4 ZRESTSE ^{13}C spectra

Parameter sets: **awzrestse, awrestseig** or **awzrestsend (+ getprosol)**
Pulse programmes: **zrestse.dp.jcm800, awzrestseig** or **awzrestsend** respectively
Spectra are free from baseline roll.

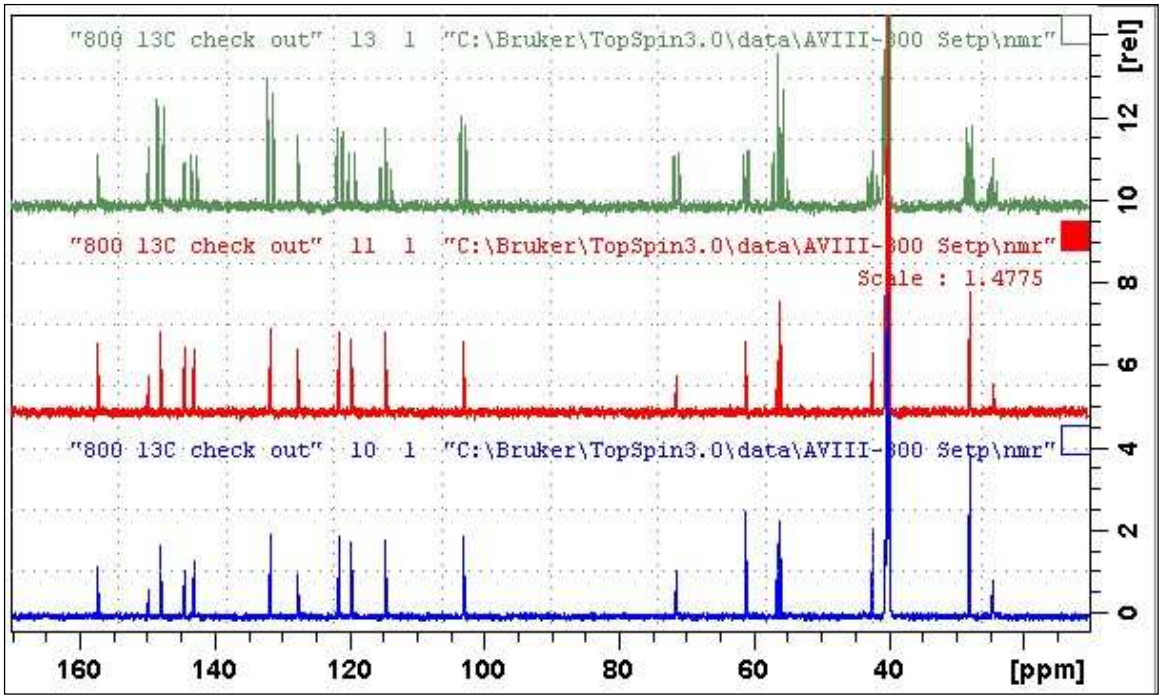
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.
NS = Any number, **DS** = 4, 8 or 16.

P0 = P1*CNST0/90.0; **CNST0** = 60º or other tip angle (30 to 90º) of your choice.
D1 = 2 sec or other time of your choice.

Type **ased** (enter) and review parameters used in the job.
Check **gradients** are OK

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

Processing
Process with **EF** or **EFP** (applies **LB)**



ZRESTSE ^{13}C NMR spectra of quinine in D6-DMSO. ***Lower:*** ZRESTSE spectrum with NOE and ^{1}H decoupling**.** ***Center:*** ZRESTSEIG spectrum with ^{1}H decoupling and no NOE (inverse gated mode)**.** ***Upper:*** ^{1}H coupled ZRESTSEND spectrum with NOE.

2.5.1 DEPT45, DEPT90 and DEPT135 spectra

Parameter sets: **awdept45, awdept90** or **awdept135 (+ getprosol)**
Pulse programmes: **dept45, dept90** or **dept135** respectively

TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.
NS = Any number, **DS** = 4, 8 or 16.

CNST2 = 145 Hz or other ^{1}J coupling constant (typically 125- 160 Hz) of your choice.
D1 = 2 sec or other time of your choice.

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

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

Processing
Process with **EF** or **EFP** (applies **LB**).
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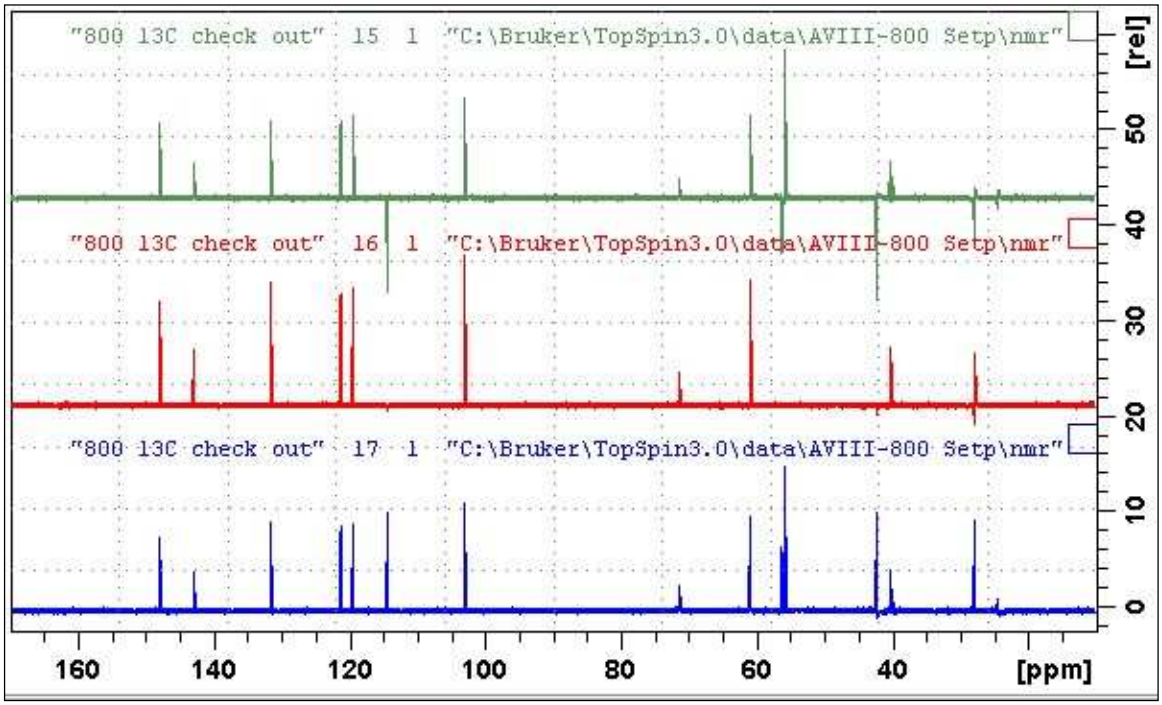

DEPT NMR spectra of quinine in D_6-DMSO. ***Lower:*** DEPT135 spectrum.
Center: DEPT90 spectrum. ***Upper:*** DEPT45 spectrum.

2.5.2 DEPT45SP, DEPT90SP and DEPT135SP spectra

Parameter sets: **awdept45sp, awdept90sp** or **awdept135sp (+ getprosol)**
Pulse programmes: **deptsp45, deptsp90** or **deptsp135** respectively

TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.
NS = Any number, **DS** = 4, 8 or 16.

CNST2 = 145 Hz or other ^{1}J coupling constant (typically 125- 160 Hz) of your choice.
D1 = 2 sec or other time of your choice.

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

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

Processing
Process with **EF** or **EFP** (applies **LB)**

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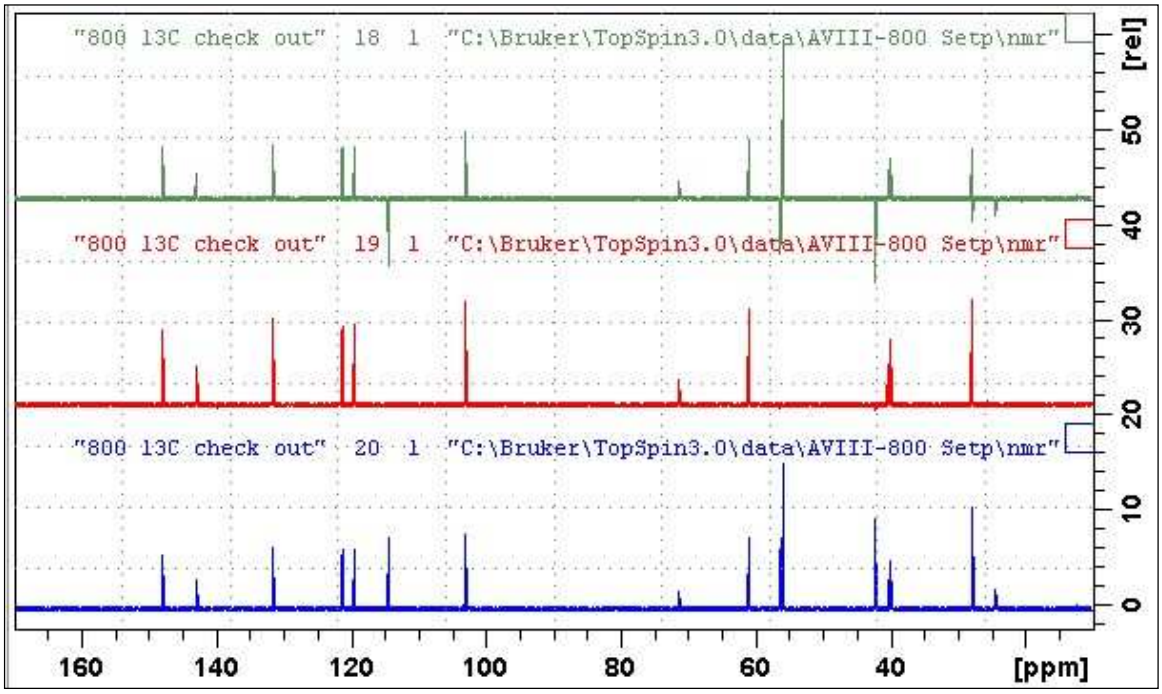


DEPTSP NMR spectra of quinine in D_6-DMSO. ***Lower:*** DEPT135SP spectrum. ***Center:*** DEPT90SP spectrum. ***Upper:*** DEPT45SP spectrum.

2.5.3 ^{1}H Coupled DEPT spectra

Parameter set: **awdeptnd (+ getprosol)**
Pulse programme: **deptnd** with **CNST12 = 0.5, 1.0 or 1.5**

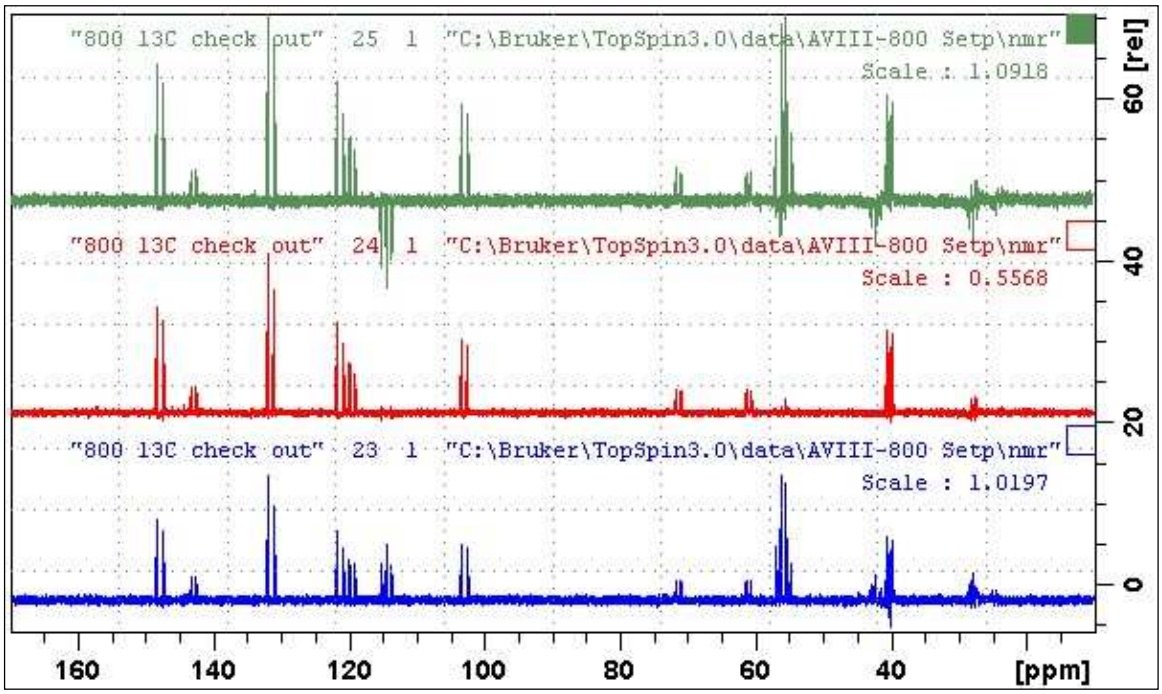
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.
NS = Any number, **DS** = 4, 8 or 16

CNST2 = **145 Hz** or other ^{1}J coupling constant (typically 125- 160 Hz) of your choice.
CNST12 = 0.5, 1.0 or **1.5** (coupled **DEPT45nd, DEPT90nd** or **DEPT135nd** spectra).
D1 = 2 sec or other time of your choice.

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

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

Processing
Process with **EF** or **EFP** (applies **LB)**



Coupled DEPTnd NMR spectra of quinine in D_6-DMSO. ***Lower:*** DEPT45nd spectrum. ***Center:*** DEPT90nd spectrum. ***Upper:*** DEPT135nd spectrum.

2.6.1 DEPT135Q spectrum with quaternary carbons

Parameter set: **awdept135q (+ getprosol)**
Pulse programme: **deptqgpsp**

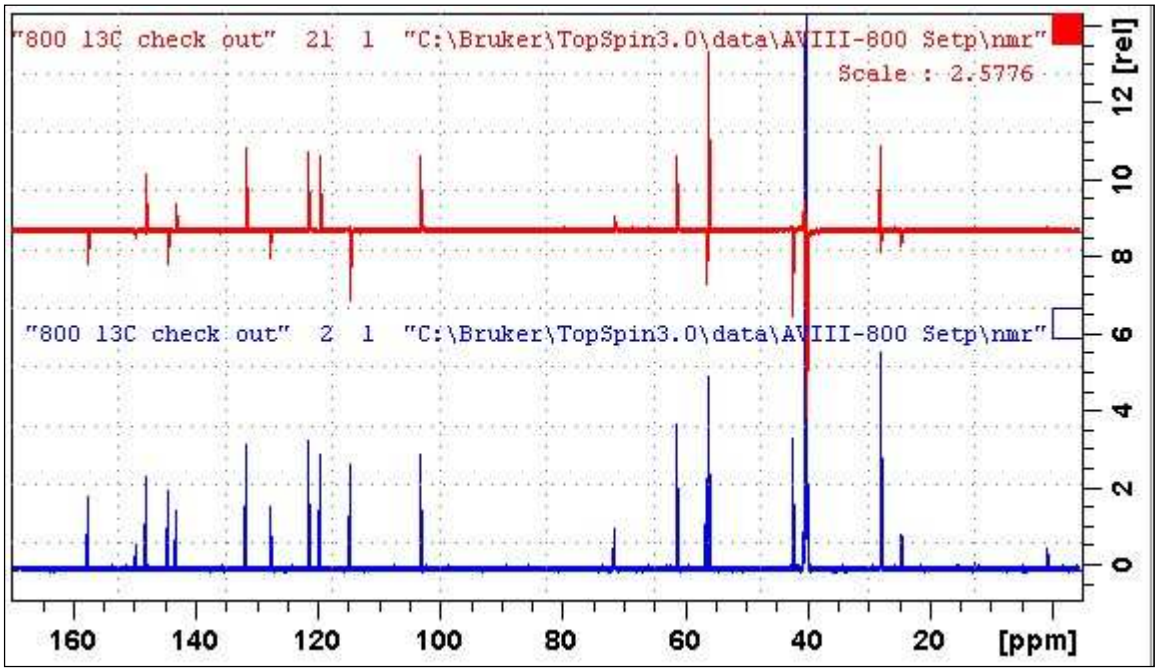
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.
NS = Any number, **DS** = 4, 8 or 16.

D1 = 1.5 sec or other time of your choice.
CNST2 = ^{1}J (^{13}C-^{1}H) **= 145 Hz** or other value of your choice.
CNST12 = 1.5 for DEPT135 spectrum with quaternary carbons

Type **ased** (enter) and review parameters used in the job.
Check **GPZ1, GPZ2** and **GPZ3** gradients = **31%.**

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

Processing
Process with **EF** or **EFP** (applies **LB**). A large negative **PHC1** phase correction (eg -650°) will be required. After phasing baseline roll can be eliminated using the **multiabsn** macro with n = 30-50.



Lower: ^{13}C NMR spectra of quinine in D_6-DMSO. **Upper:** DEPT135Q spectrum; CH and CH_3 carbons positive; C (quaternary) and CH_2 carbons negative.

2.6.2 DEPT Quaternary carbon only spectrum

Parameter set: **awdeptq (+ getprosol)**
Pulse programme: **deptqgpsp**

TD = 64 K, **SI** = 64 K.
NS = multiple of 4 or 8, **DS** = 8 or 16.

CNST2 = ^{1}J (^{13}C-^{1}H) **= 145 Hz** or other value of your choice.
CNST12 = 1.5 for alternating cancellation of DEPT135 carbon signals.
D1 = **1.5 sec** or other time of your choice.

Type **ased** (enter) and review parameters used in the job.
Check **GPZ1, GPZ2** and **GPZ3** gradients = **31%, 31%** and **11%** respectively.

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

Processing
Process with **EF** or **EFP** (applies LB)

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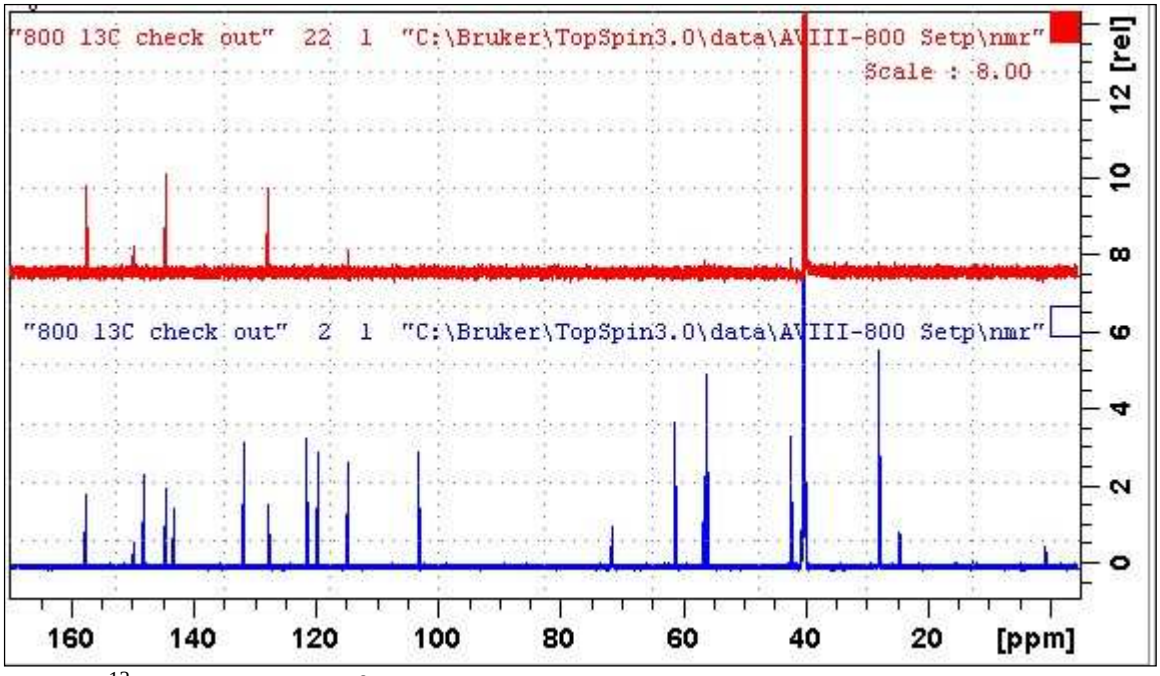


Lower: ^{13}C NMR spectra of quinine in D_6-DMSO. **Upper:** DEPTQ spectrum.

2.7.1 INEPT45, INEPT90 or INEPT135 spectra

Parameter sets: **awinept45, awinept90, awinept135 (+ getprosol)**
Pulse programme: **awineptrd with CNST11 = 6, 4 or 3** respectively

TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

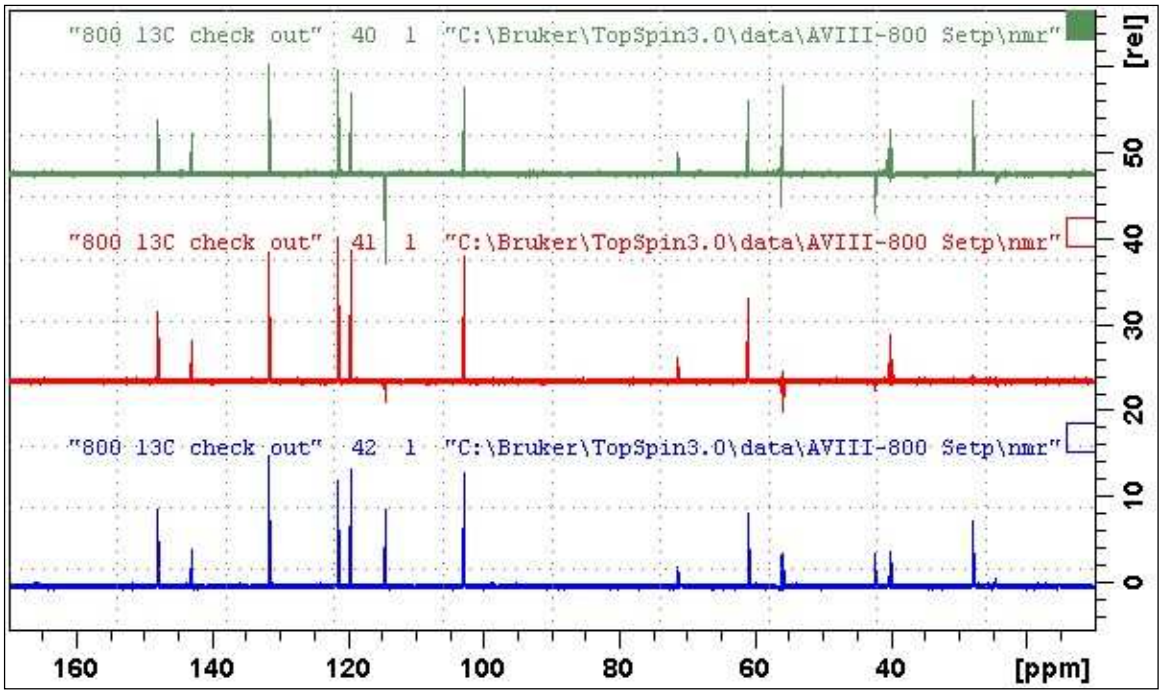
NS = Any number, **DS** = 4, 8 or 16.
D1 = 2 sec or other time of your choice.

CNST2 = ^{1}J (^{13}C-^{1}H) **= 145 Hz** or other value of your choice.
CNST11 = 6, 4 or 3 for I**NEPT45, INEPTT90** or **INEPT135** spectra respectively.

Type **ased** (enter) and review parameters used in the job.
Check your choice of **CNST11.**

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

Processing
Process with **EF** or **EFP** (applies LB)



INEPT spectra of quinine in D_6-DMSO. ***Lower:*** INEPT135 spectrum.
Center: INEPT90 spectrum ***Upper:*** INEPT45 spectrum.

2.7.2 INEPT45SP, INEPT90SP or INEPT135SP spectra

Parameter sets: **awinept45sp, awinept90sp or awinept135sp (+ getprosol)**
Pulse programme: **awineptrdsp with CNST11 = 6, 4 or 3** respectively

TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

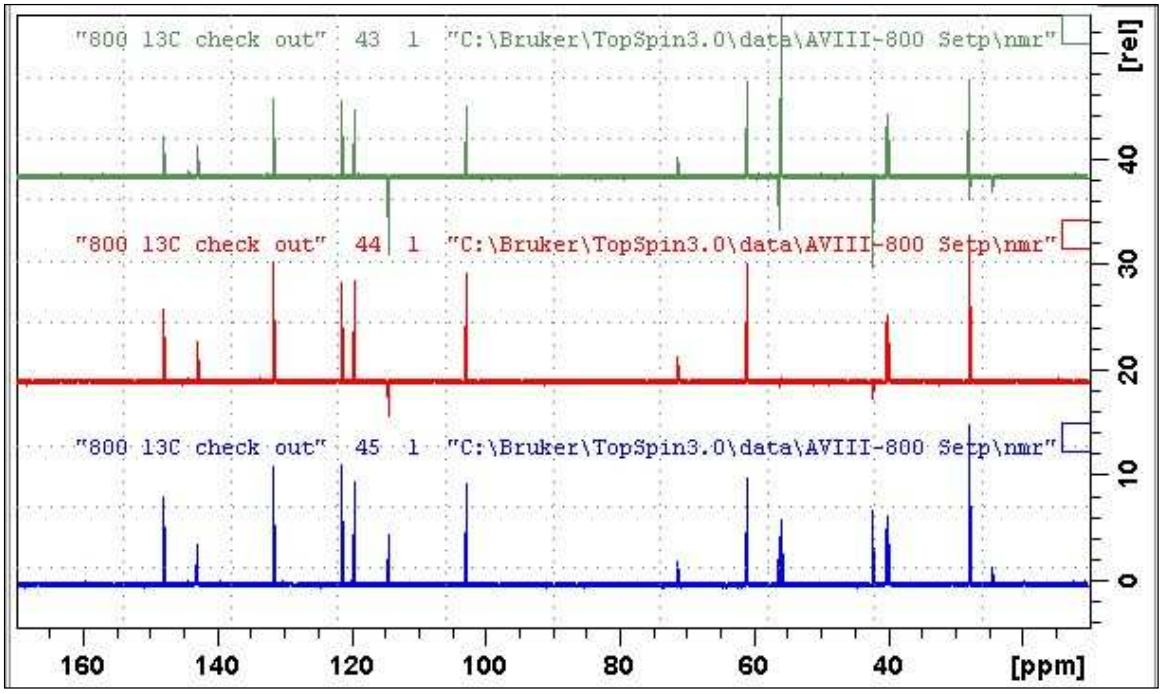
NS = Any number, **DS** = 4, 8 or 16.
D1 = 2 sec or other time of your choice.

CNST2 = ^{1}J (^{13}C-^{1}H) **= 145 Hz** or other value of your choice
CNST11 = 6, 4 or 3 for **INEPT145SP, INEPT90SP** or **INEPT135SP**spectra respectively

Type **ased** (enter) and review parameters used in the job. Check your choice of **CNST11.**

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

Processing
Process with **EF** or **EFP** (applies LB)



INEPTSP spectra of quinine in D_6-DMSO. **Lower:** INEPT135SP spectrum. **Center:** INEPT90SP spectrum. **Upper:** INEPT45SP spectrum.

2.7.3 INEPTND and INEPTNDSP spectra

Parameter set: **awineptnd or awineptndsp (+ getprosol)**
Pulse programme**: awineptnd or awineptndsp**

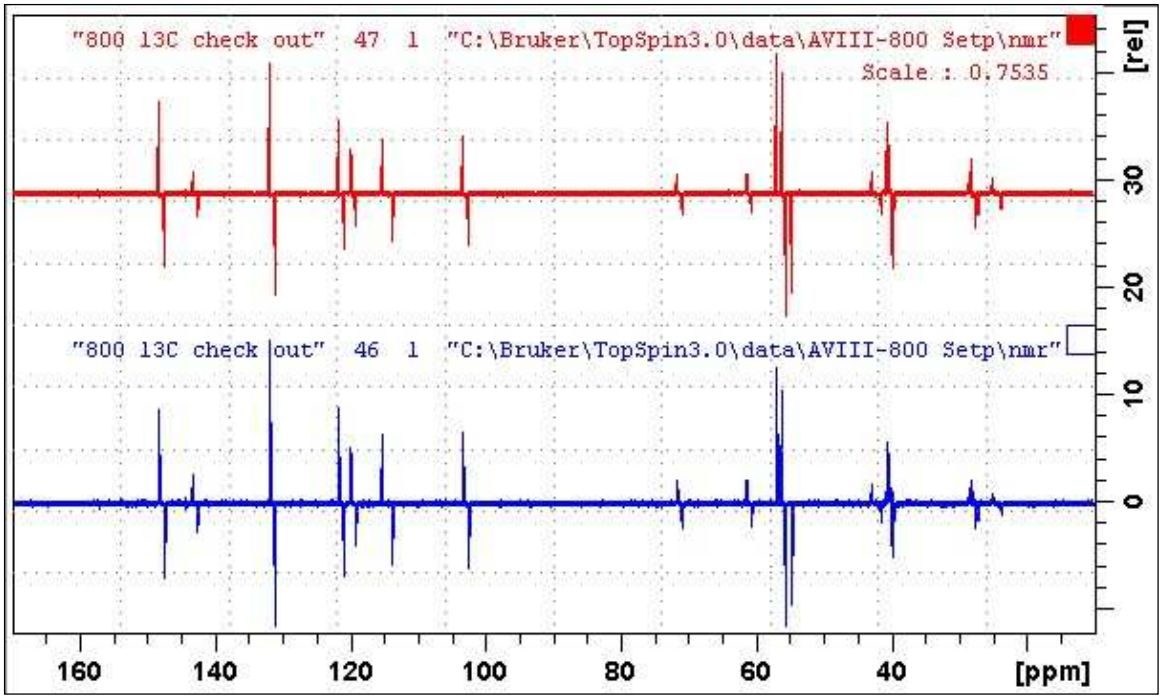
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

NS = Any number, **DS** = 4, 8 or 16.
D1 = 1.5 sec or other time of your choice.
CNST2 = ^{1}J (^{13}C-^{1}H) **= 145 Hz** or other value of your choice.

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

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

Processing
Process with **EF** or **EFP** (applies LB).
Signals have non-binomial intensities: CH (d) = (1, -1); CH_2 (t) = (1, 0, -1);
CH_3 (q) = (1, 1, -1, -1).



Coupled INEPTND spectra of quinine in D_6-DMSO. **Lower:** INEDPTND spectrum**.**
Upper: INEDPTNDSP spectrum.

2.8.1 JMOD spectra

Parameter sets: **awjmod or awjmodq (+ getprosol)**
Pulse programme: **awjmod** with **cnst11** = **1** or **2** respectiuvely

Standard settings
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

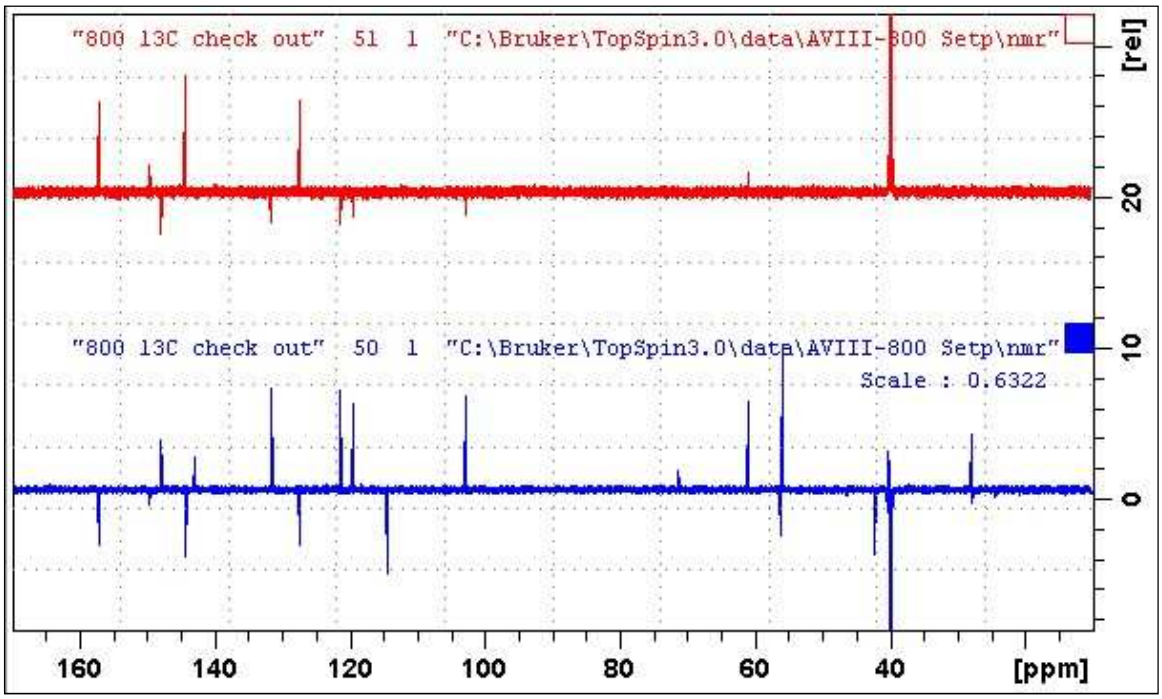
NS = Any number, **DS** = 4, 8 or 16.
D1 = 1.5 sec (awjmodsp) or 4 sec (awjmodspq) spectra or other time of your choice**.**

CNST2 = ^{1}J (^{13}C-^{1}H) **= 145 Hz** or other value of your choice.
CNST11 = 1 (CH, CH_3 positive, C, CH_2 negative) for *J*-modulated spectrum.
= 2 for C (quaternary) only carbons for quaternary carbons only spectrum.

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

Set **receiver gain** using **RGA** *(important!)*

Processing
Process with **EF** or **EFP** (applies LB).



J-modulated spectra of quinine in D_6-DMSO**. Lower:** JMOD spectrum.
Upper: JMODQ spectrum. Residual positive or negatively phased signals are seen ex protonated carbons whose ^{1}J coupling constants differ significantly from CNST2 = 145 Hz.

2.8.2 JMODSP spectra with shaped refocusing pulses

Parameter sets: **awjmodsp or awjmodqsp (+ getprosol)**
Pulse programme: **awjmodsp** with **cnst11** = **1** or **2** respectiuvely

TD = 64 K, **SI** = 64 K.
SW = 240 ppm, O1P = 110 ppm.

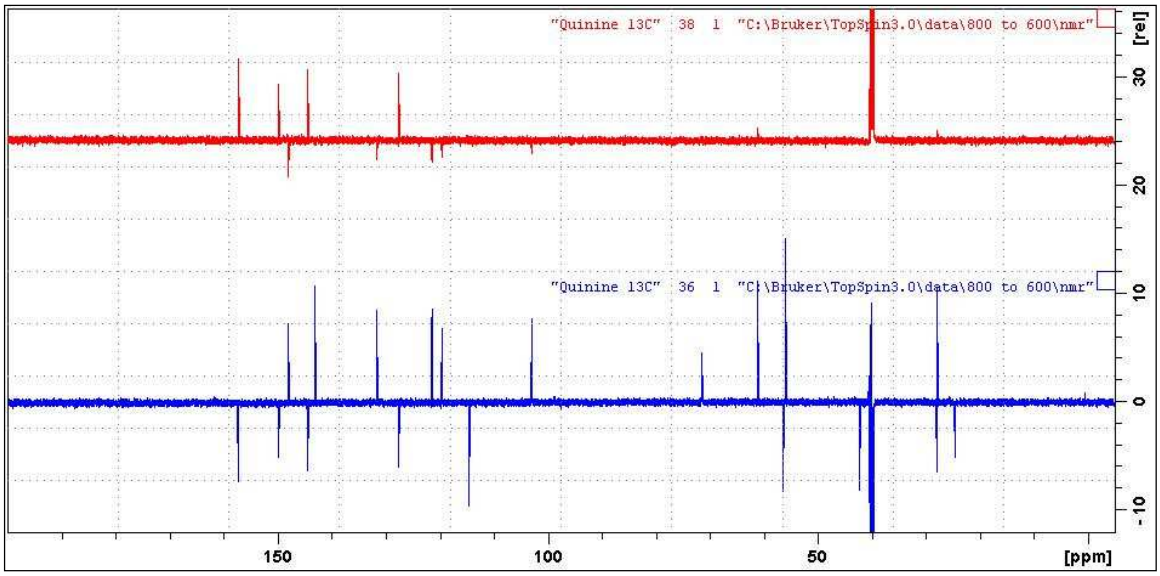
NS = multiple of 4 or 8, **DS** = 4, 8 or 16.
D1 = 1.5 sec (awjmodsp) or 4 sec (awjmodspq**)** spectra or other time of your choice.

CNST2 = ^{1}J (^{13}C-^{1}H) **= 145 Hz** or other value of your choice.
CNST11 = 1 (CH, CH_3 positive, C, CH_2 negative), or
= 2 for C (quaternary) only carbons.

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

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

Processing
Process with **EF** or **EFP** (applies LB).



J-modulated spectra of quinine in D_6-DMSO**. Lower:** JMODSP spectrum.
Upper: JMODQSP spectrum. Residual positive or negatively phased signals are seen ex protonated carbons whose ^{1}J coupling constants differ significantly from CNST2 = 145 Hz.

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2.9.1 APT (Attached Proton Test) spectra

Parameter sets: **awapt or awaptq (+ getprosol)**
Pulse programme: **awjmod** with **cnst11** = **1** or **2** respectiuvely

TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

NS = Any number, **DS** = 4, 8 or 16.
P0 = 90 degrees or other tip angle of your choice (30-90^{o}).

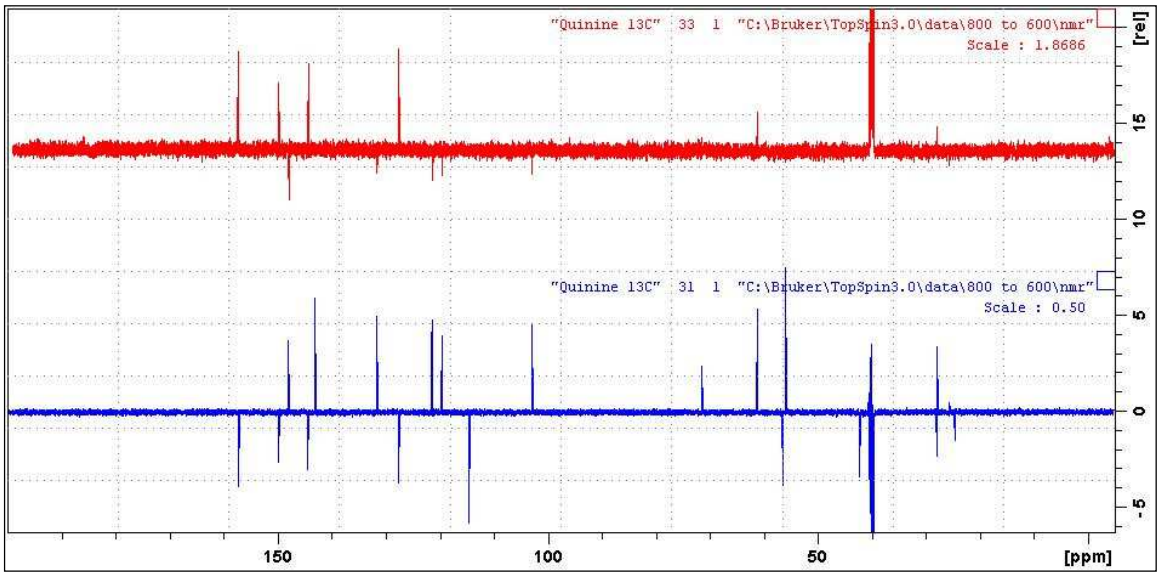
D1 = 1.5 sec (**awapt**) or 4 sec (**awaptq**) spectra or other time of your choice.
D21 = 2 msec or other time of your choice (0.5–2 msec).

CNST2 = ^{1}J (^{13}C-^{1}H) = 145 Hz or other value of your choice.
CNST11 = 1 (CH, CH_3 positive, C, CH_2 negative) for *J*-modulated APT spectrum.
= 2 for C (quaternary) only carbons for quaternary carbons only spectrum.

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

Set **receiver gain** using **RGA** *(important).*

Processing
Process with **EF** or **EFP** (applies LB).



APT (**lower**) and APTQ (**upper**) spectra of quinine in D_6-DMSO. Some residual positive or negatively phased signals are seen ex protonated carbons whose ^{1}J coupling constants differ significantly from CNST2 = 145 Hz

2.9.2 APTSP spectra with shaped refocusing pulses

Parameter sets: **awaptsp (+ getprosol)**
Pulse programme**:** **awaptspt0** with **cnst11 = 1 or 2** respectively

TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

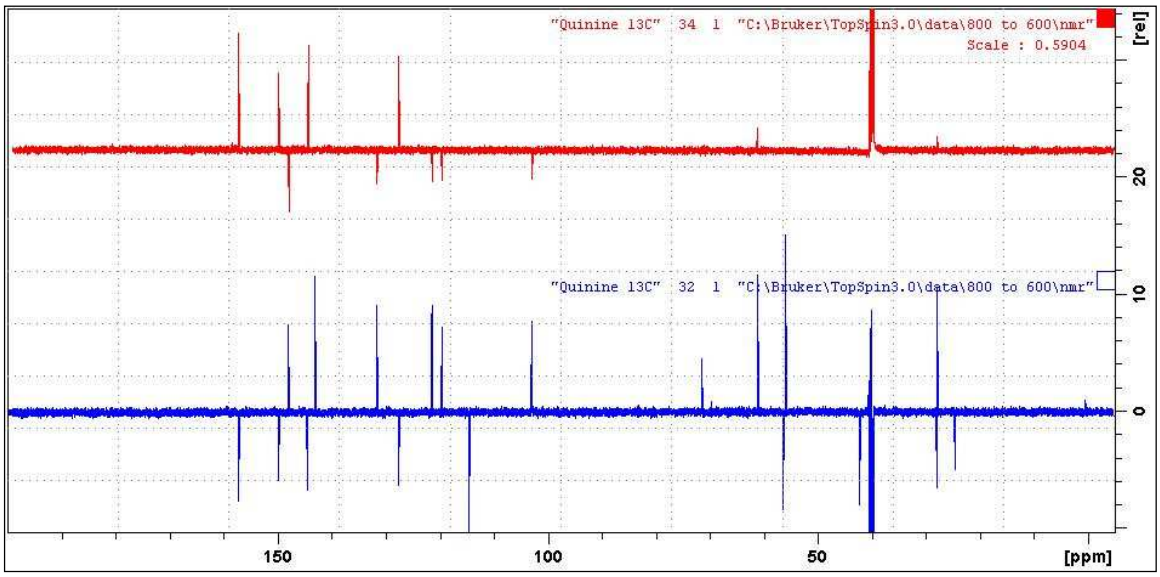
NS = multiple of 4 or 8, **DS** = 4, 8 or 16.
P0 = 90º or other tip angle of your choice (30- 90º).
D1 = 1.5 sec (aptsp) or 4 sec (aptqsp) spectra or other time of your choice.

CNST2 = ^{1}J (^{13}C-^{1}H) **= 145 Hz** or other value of your choice
CNST11 = 1 (CH, CH_3 positive, C, CH_2 negative), or
= 2 for C (quaternary) only carbons.

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

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

Processing
Process with **EF** or **EFP** (applies LB).



APTSP (**lower**) and APTSPQ (**upper**) spectra of quinine in D_6-DMSO. Some residual positive or negatively phased signals are seen ex protonated carbons whose ^{1}J coupling constants differ significantly from CNST2 = 145 Hz.

2.10 ^{13}C NMR Spectra using a p0 pulse in BASEOPT mode

Parameter sets: **awcarbonp0, awcarbonp0ig,** or **awcarbonp0nd (+ getprosol)**
Pulse programmes: **awzgp0, awzgp0ig** or **awzgpp0nd** respectively.

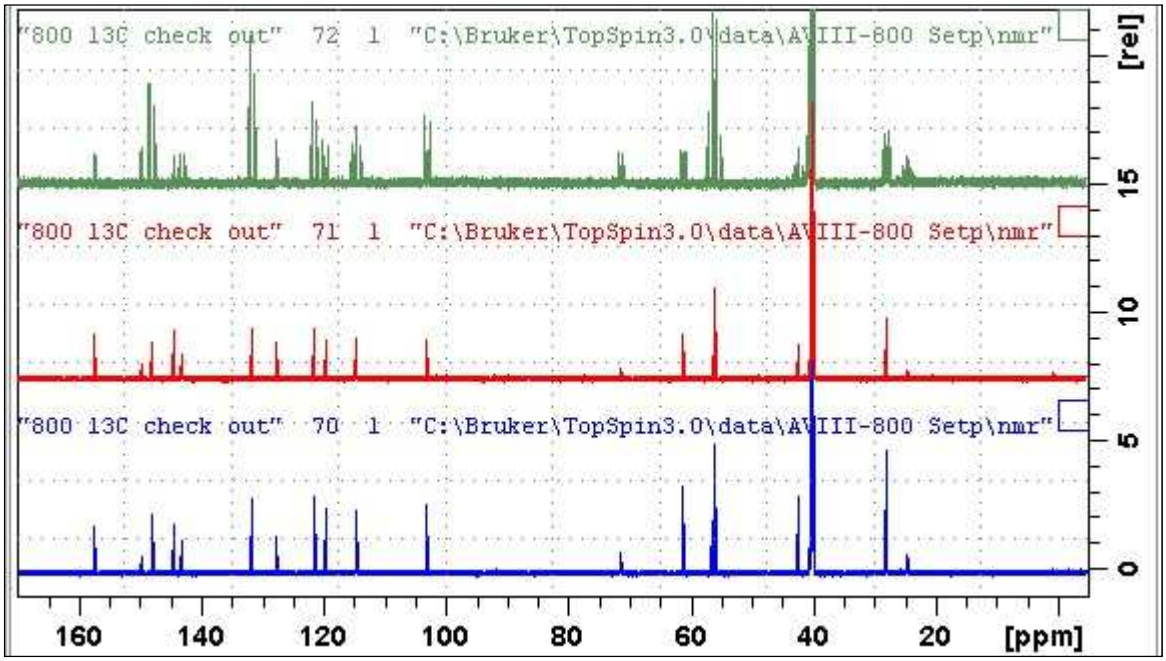
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

NS = Any number, **DS** = 4, 8 or 16.
P0 = P1*CNST0/90.0; **CNST0** = 70^{o} or other tip angle (30 to 90^{o}) of your choice.
degrees or other tip angle of your choice (30-90^{o}).
D1 = 2 sec or other time of your choice.
DE = 50 usec will reduce but not eliminate baseline roll in cyroprobe ^{13}C spectra.

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

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

Processing
Process with **EF** or **EFP** (applies **LB)**. Spectra will have normal PHC0 and PHC1 constants Residual baseline roll can be eliminated using the **multiabsn** macro with **n = 30-50**.



^{13}C NMR spectra of quinine in D_6-DMSO. **Lower:** Pulse gated ^{13}C spectrum with a p0 = 70^{o} pulse. **Center:** Inverse gated ^{13}C spectrum with a p0 = 70^{o} pulse. **Upper:** ^{1}H coupled ^{13}C spectrum with a p0 = 70^{o} pulse.