KJM 9250

AVII-600 ^{13}C NMR spectra.

Version 7.3

Topspin 3.2 Windows 7 AVII600

AVII-600 ^{13}C NMR spectra

1.0 Introduction

All 600 MHz **aw coded** carbon parameter files have 64 K points across a 240 ppm window with D1 typically = 1.5 or 4 sec for quaternay carbon only spectra.

Quaternary carbons *may have moderate to long T1'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. 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 spectra with power gated ^{1}H decoupling and NOE

2.2 Inverse gated ^{13}C 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
^{1}H Coupled DEPTND spectra

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

2.7 INEPT spectra
INEPTSP spectra
INEPTND spectrum

2.8 JMOD spectra
JMODSP spectra

2.9 APT spectra
APTSP spectra

2.1 ^{13}C NMR spectra using a 30°, 45°, 70° or 90° pulse

Parameter sets: **awcarbon30, awcarbon45, awcarbon70** or **awcarbon90 (+ getprosol)**
Pulse programmes: **zgpg30/awzgp30, awzgpg45, awzgpg70** or **zgpg/awzgpg90** respectively.
Spectra are **^{1}H decoupled with NOE.**

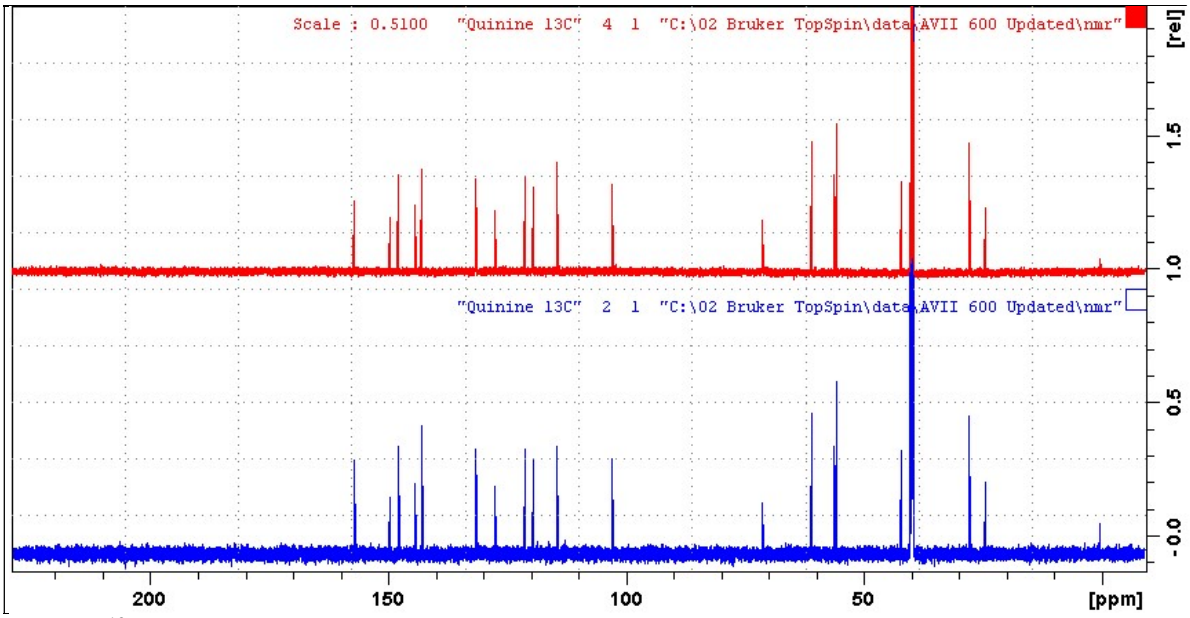
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.
NS = Any number, **DS** = 4, 8 or 16.

D1 = 1.5 sec or other time of your choice.
DE = 50 or 60 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!).*

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 = 30-40.



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

2.2 Inverse gated ^{13}C NMR spectra

Parameter sets: **awcarbon30ig, awcarbon45ig, awcarbon70ig, awcarbon90ig (+ getprosol)**
Pulse programmes: **awzgig30, awzgig45, awzgig70, awzgig respectively**
Spectra are ^{1}H decoupled without NOE

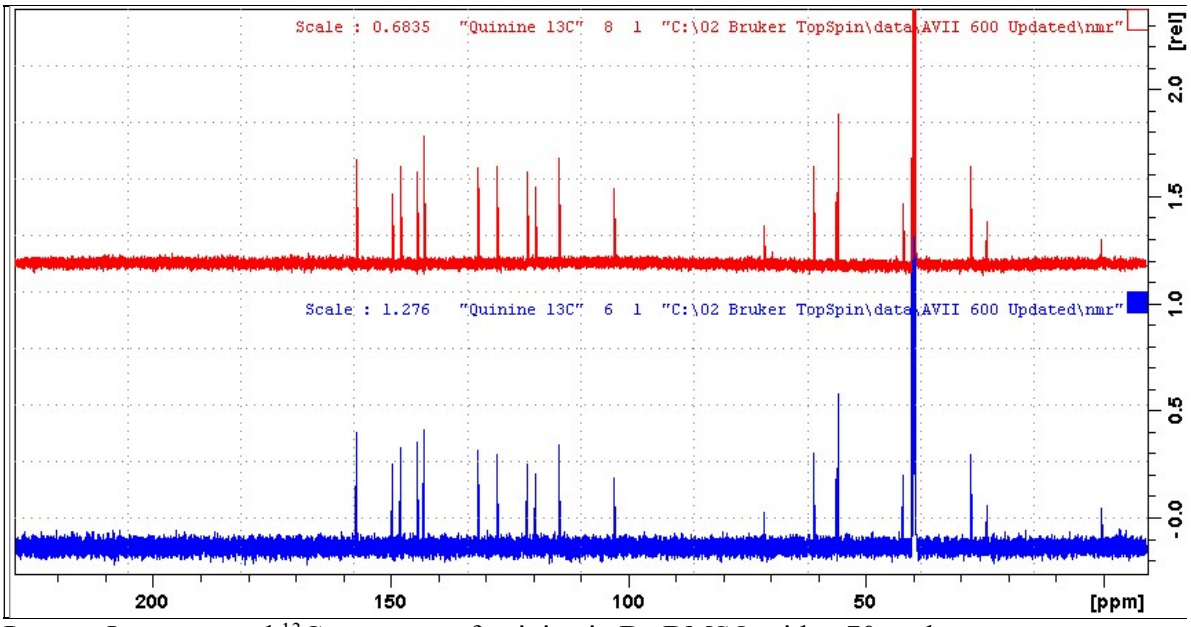
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

NS = Any number, **DS** = 4, 8 or 16.
D1 = 1.5 sec or other time of your choice.
DE = 50 or 60 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!).*

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

2.3 ^{1}H coupled ^{13}C NMR spectrum with NOE

Parameter set: **awcarbon70nd (+ getprosol)**
Pulse programme: **awzg70nd**
^{1}H decoupled ^{13}C spectrum with NOE using a 70^{o} pulse

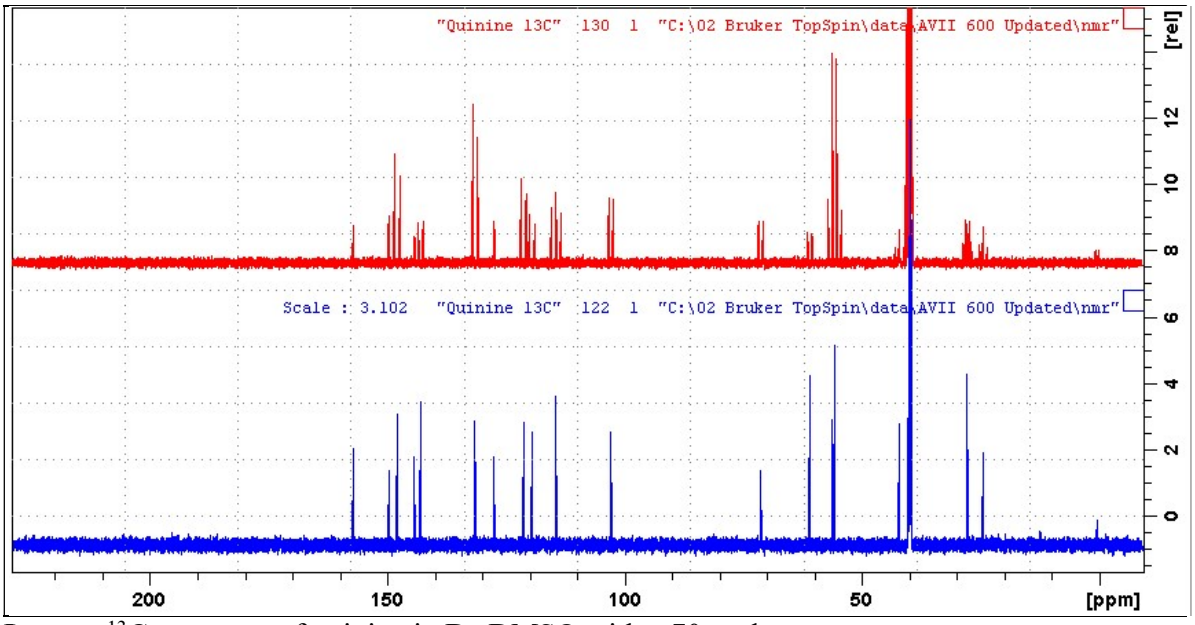
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

NS = Any number, **DS** = 4, 8 or 16.
D1 = 1.5 sec or other time of your choice.
DE = 50 or 60 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!).*

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



Lower: ^{13}C spectrum of quinine in D_6-DMSO with a 70^{o} pulse.
Upper: ^{1}H coupled ^{13}C spectrum of quinine in D_6-DMSO with a 70^{o} pulse.

2.4 ZRESTSE ^{13}C spectra

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

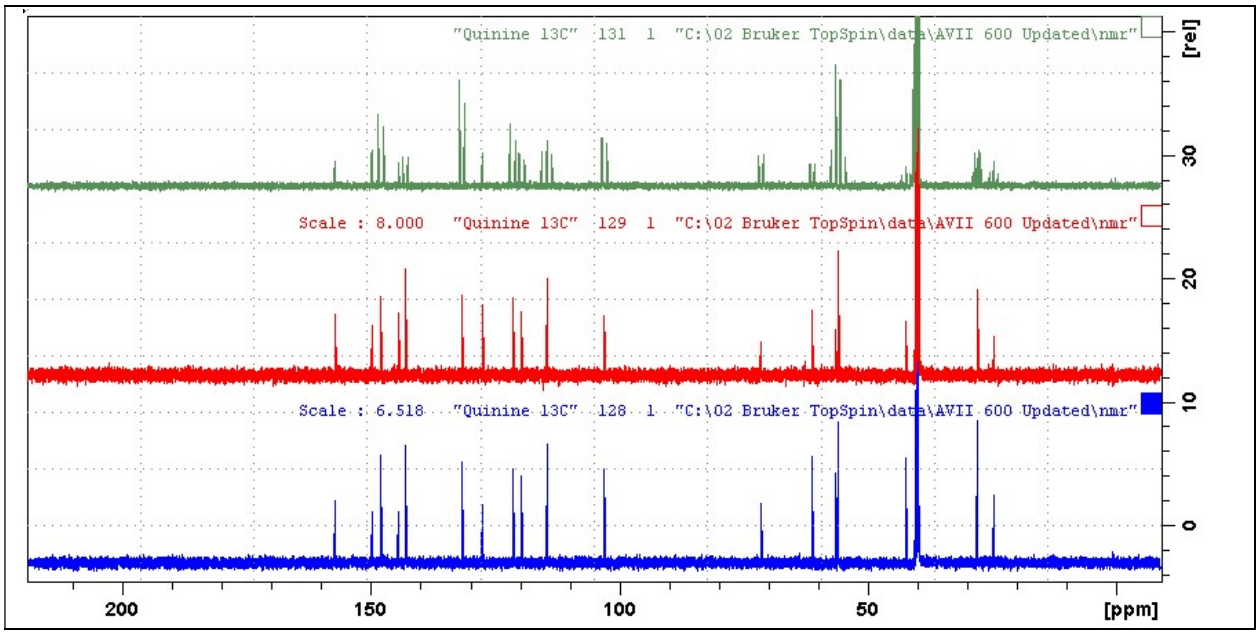
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

NS = Any number, **DS** = 4, 8 or 16.
P0 = P1*CNST0/90.0; **CNST0 = 70°** or other tip angle of your choice (30°-90° range).
D1 = **1.5 sec** (zrestse and zrestseig) or **4 sec** (zrestsend) or other time of your choice.

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

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

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



ZRESTSE spectra of quinine in D_6-DMSO determined with a 70° P0 pulse.
Lower: power gated **zrestse** spectrum. ***Center:*** inverse gated **zrestseig** spectrum.
Upper: ^{1}H coupled **zrestsend** spectrum.

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.

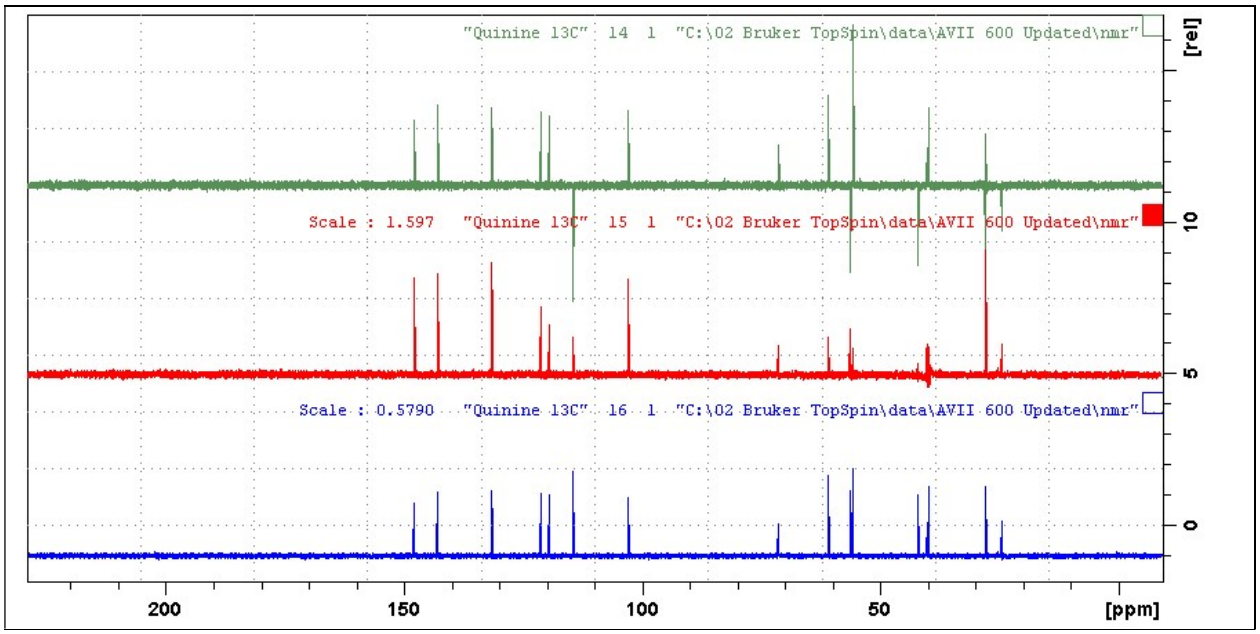
D1 = 1.5 sec or other time of your choice.
CNST2 = 145 Hz = ^{1}J (^{13}C-^{1}H) or other value of your choice.

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

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

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

.



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

2.5.2 DEPTSP45, DEPTSP90 and DEPTSP135 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.

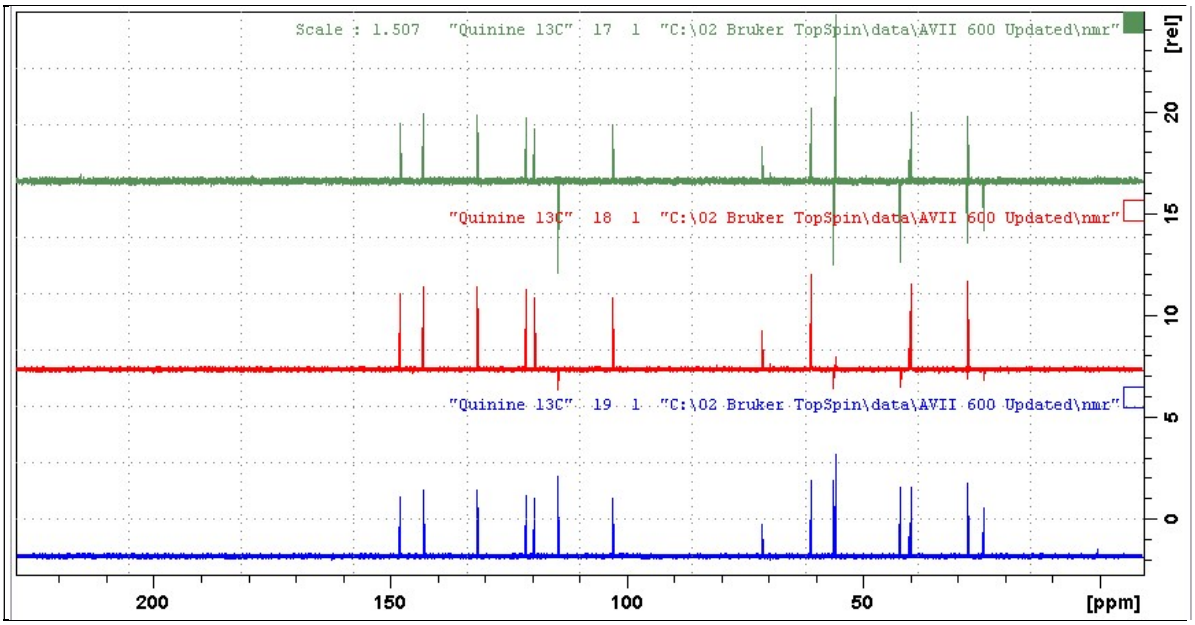
NS = Any number, **DS** = 4, 8 or 16.
D1 = 1.5 sec or other time of your choice.
CNST2 = 145 Hz = ^{1}J (^{13}C-^{1}H) or other value of your choice.

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

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

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

.



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

2.5.3 ^{1}H Coupled DEPTND Spectra

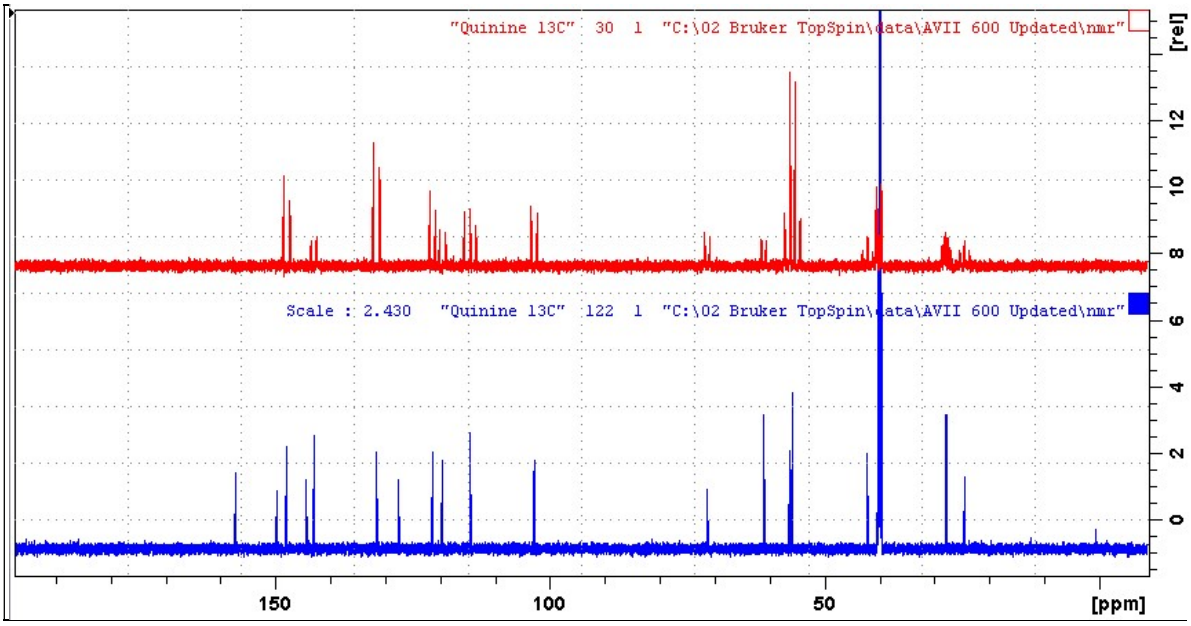
Parameter set: **awdeptnd (+ getprosol)**
Pulse programme: **deptnd**

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 = 145 Hz = ^{1}J (^{13}C-^{1}H) or other value of your choice.
CNST12 = 0.5. 1.0 or 1.5 for a 45, 90 or 135 degree pulse

Type **ased** (enter) and review parameters used in the job.
Set **receiver gain** using **RGA** *(important!).*

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



Lower: DEPT45 spectrum of quinine in D_6-DMSO.
Upper: DEPT45ND spectrum.

2.6.1 DEPT135Q spectrum with quaternary carbons

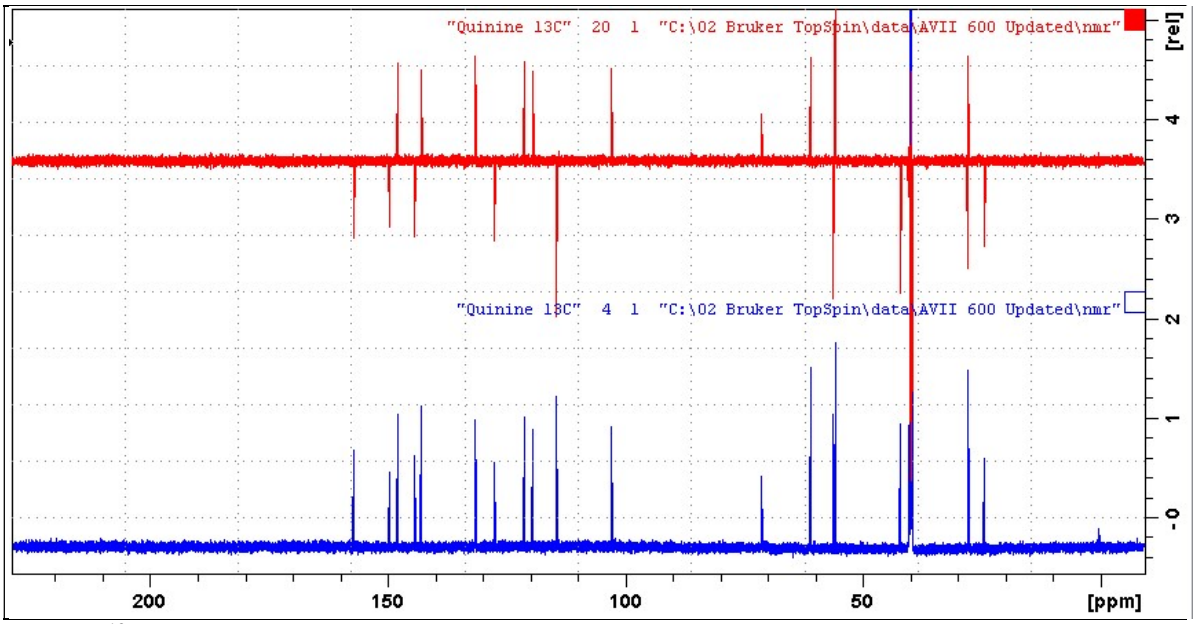
Parameter set: **awdept135q (+ getprosol)**
Pulse programme: **deptqgpsp**

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 = 145 Hz = ^{1}J (^{13}C-^{1}H) 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!).*

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



Lower: ^{13}C NMR spectra of quinine in D_6-DMSO**. Upper: Dept135** 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**

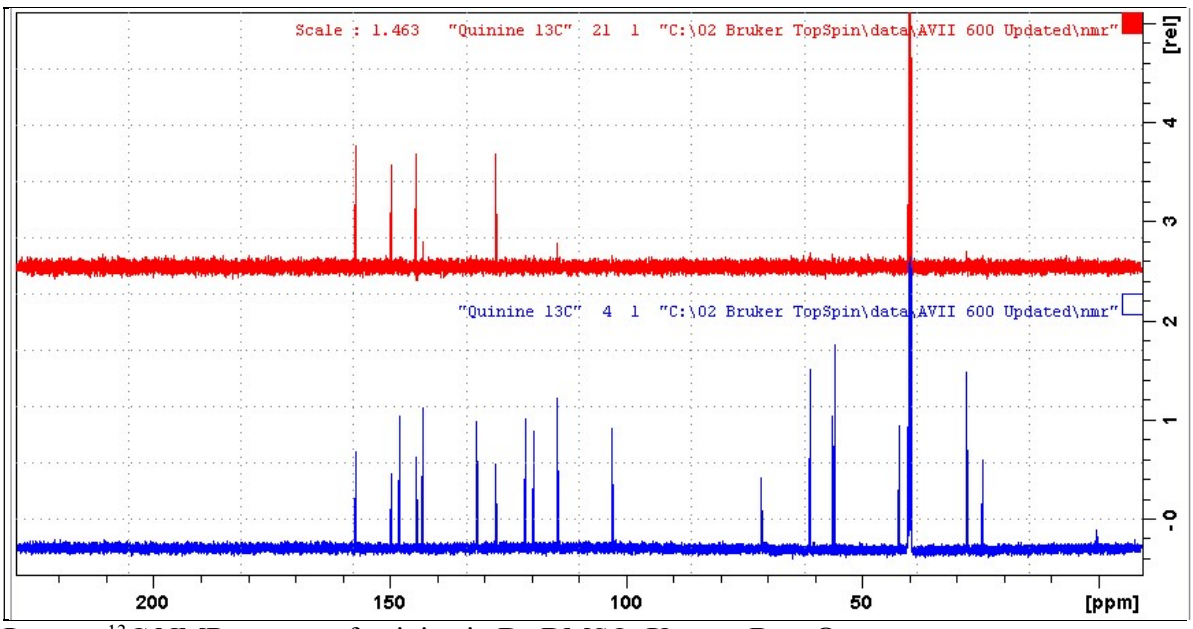
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, O1P = 110 ppm.

NS = multiple of 4 or 8, **DS** = 8 or 16.
D1 = 1.5 sec or other time of your choice.
CNST2 = 145 Hz = ^{1}J (^{13}C-^{1}H) or other value of your choice.
CNST12 = 1.5 for alternating cancellation of DEPT135 carbon signals.

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!).*

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

.



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: **ineptrd with CNST11 = 6, 4 or 3** respectively

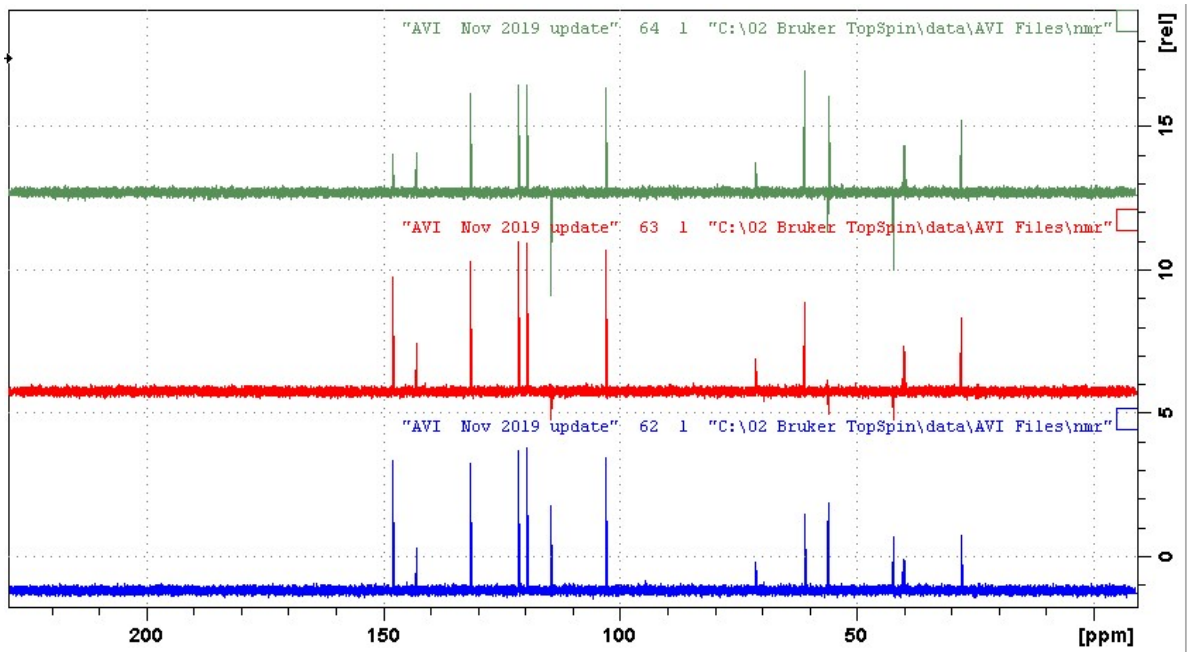
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

NS = Any number, **DS** = 4, 8 or 16.
D1 = 1.5 sec or other time of your choice.
CNST2 = 145 Hz = ^{1}J (^{13}C-^{1}H) or other value of your choice.
CNST11 = **6** for INEPT45,
= **4** for INEPTT90
= **3** for INEPT135

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

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

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



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

2.7.2 INEPT45SP, INEPT90SP or INEPT135SP spectra

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

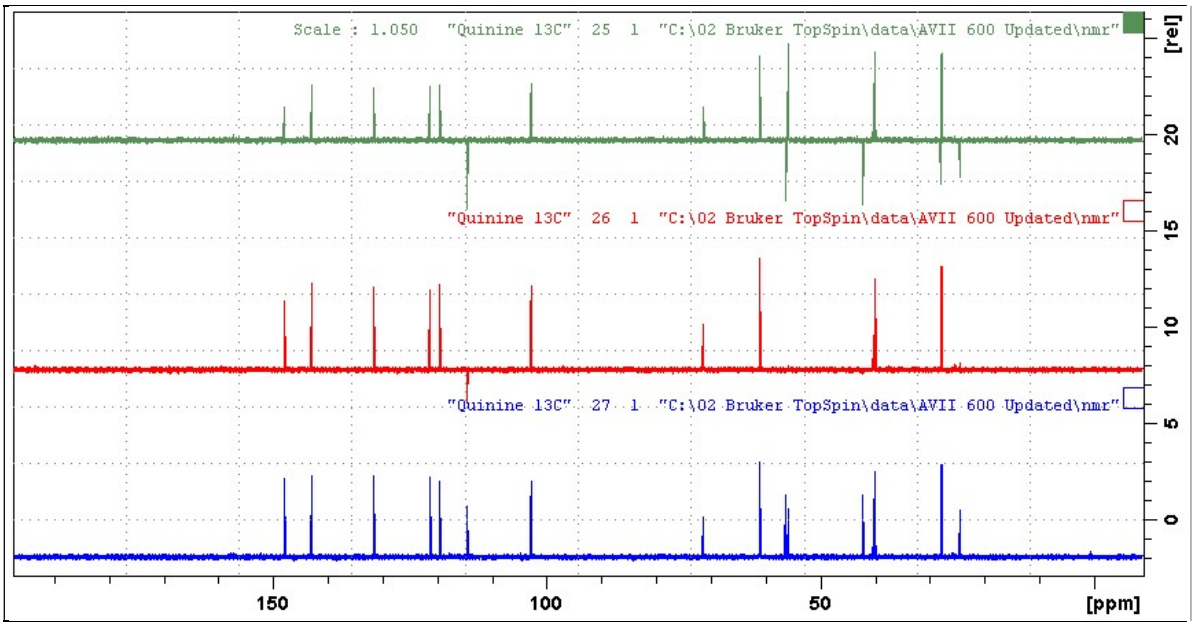
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

NS = Any number, **DS** = 4, 8 or 16.
D1 = 1.5 sec or other time of your choice.
CNST2 = 145 Hz = ^{1}J (^{13}C-^{1}H) or other value of your choice.
CNST11 = **6** for INEPT45SP,
= **4** for INEPTT90SP
= **3** for INEPT135SP

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

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

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



INEPTSP spectra of quinine in D_6-DMSO. ***Lower:*** INEPT145SP spectrum.
Center: INEPT90SP spectrum ***Upper:*** INEPT135SP spectrum.

2.7.3 INEPTND spectrum

Parameter set: **awineptnd (+ getprosol)**
Pulse programme**: ineptnd**

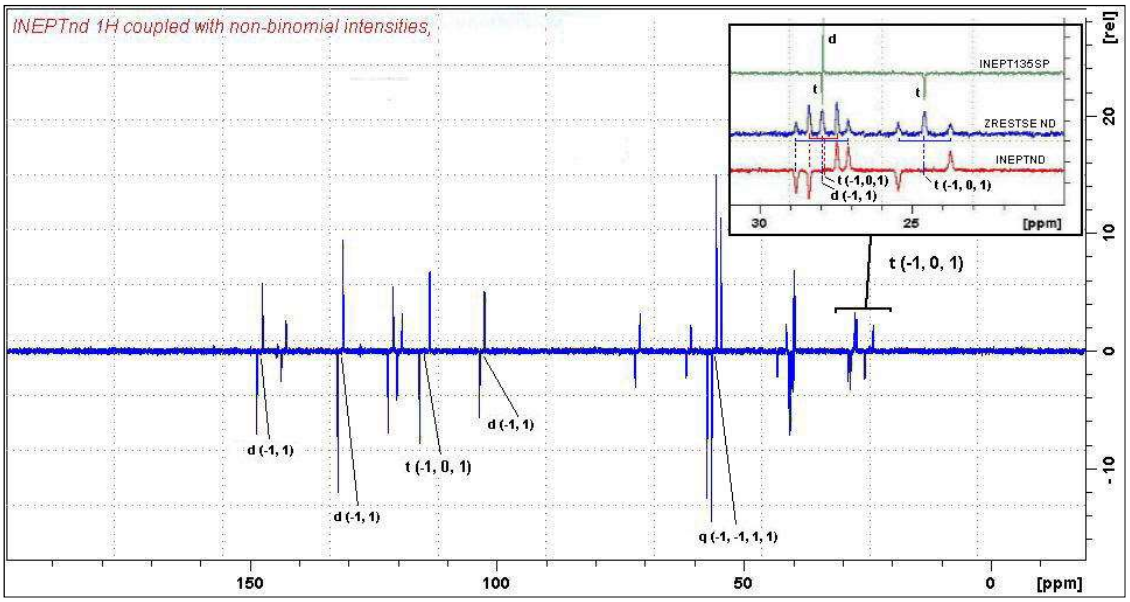
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

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

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

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

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)



INEPTND spectrum of quinine in D_6-DMSO. The multiplicity of some signals is shown.

Insert: Expansions of the 20-30 ppm regions of **ineptnd**, **zrestsend** and **inept135sp** spectra with signal annotations.

2.8.1 JMOD (J-modulated) spectra

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

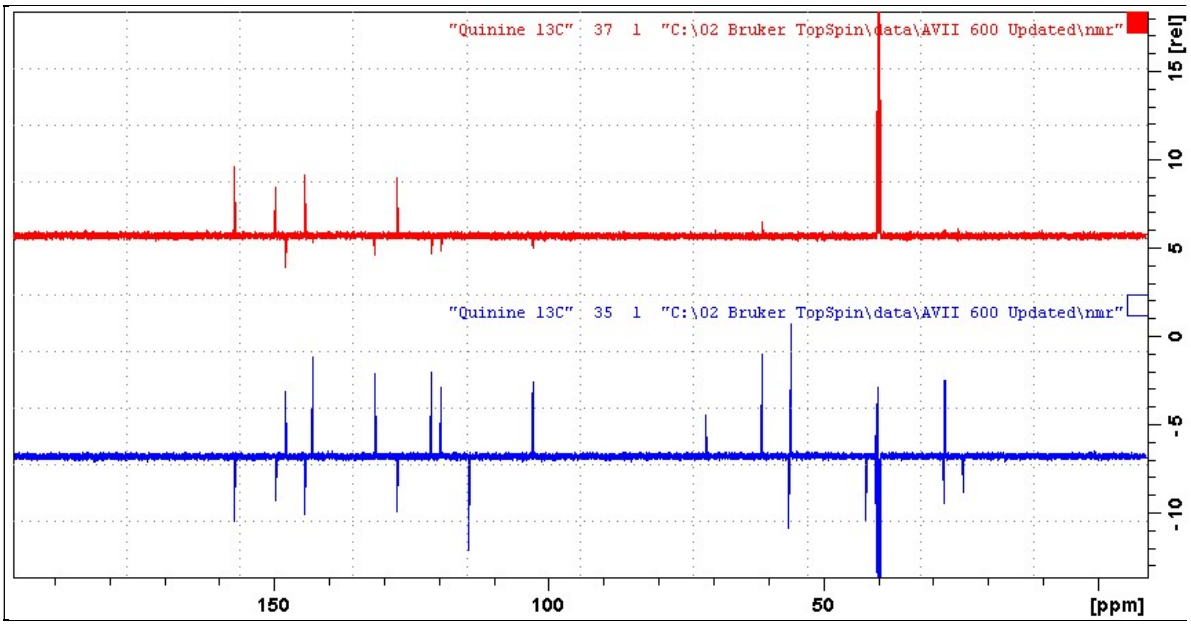
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

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

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



JMOD (lower) and **JMODQ (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.

2.8.2 JMODSP spectra with shaped refocusing pulses

Parameter set: awjmodsp or awjmodqsp (+ getprosol)
Pulse programme**: awjmodsp** with **cnst 11 = 1 or 2** respectively

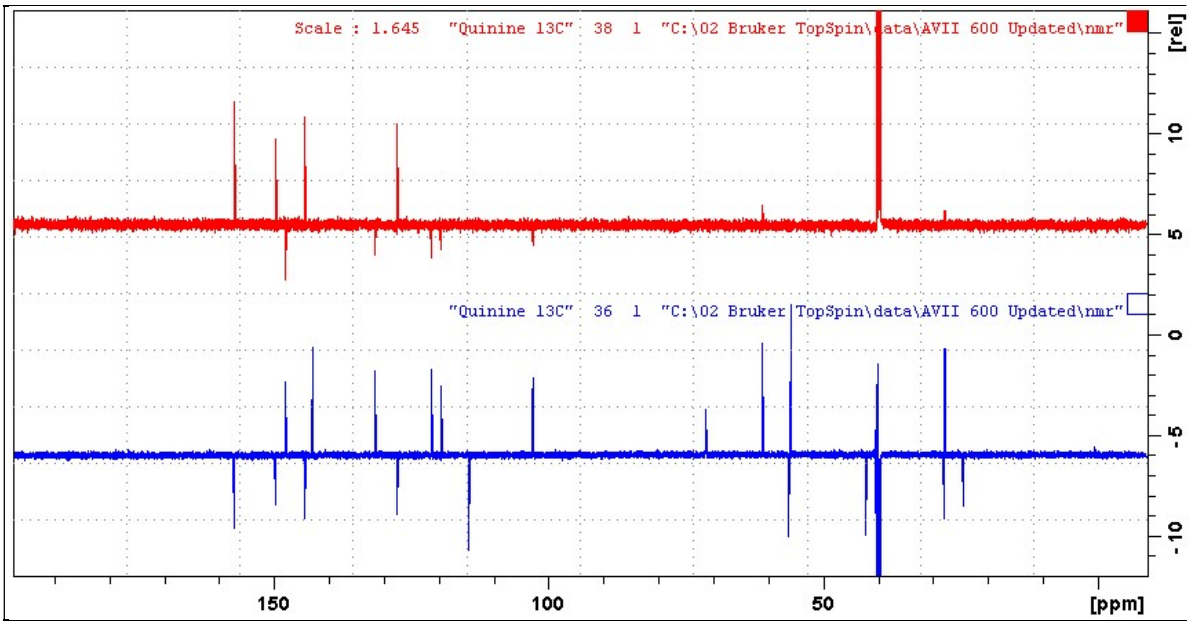
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

NS = multiple of 4 or 8, **DS** = 4, 8 or 16.
D1 = 1.5 sec (jmodsp) or 3-4 sec (jmodqsp) spectra or other time of your choice.
CNST2 = 145 Hz = ^{1}J (^{13}C-^{1}H) 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!).*

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



JMODSP (lower) and **JMODSPQ (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.

2.9.1 APT (Attached Proton Test) spectra

Parameter sets: **awapt or awaptq (+ getprosol)**
Pulse programme**: apt** with **cnst 11 = 1 or 2** respectively

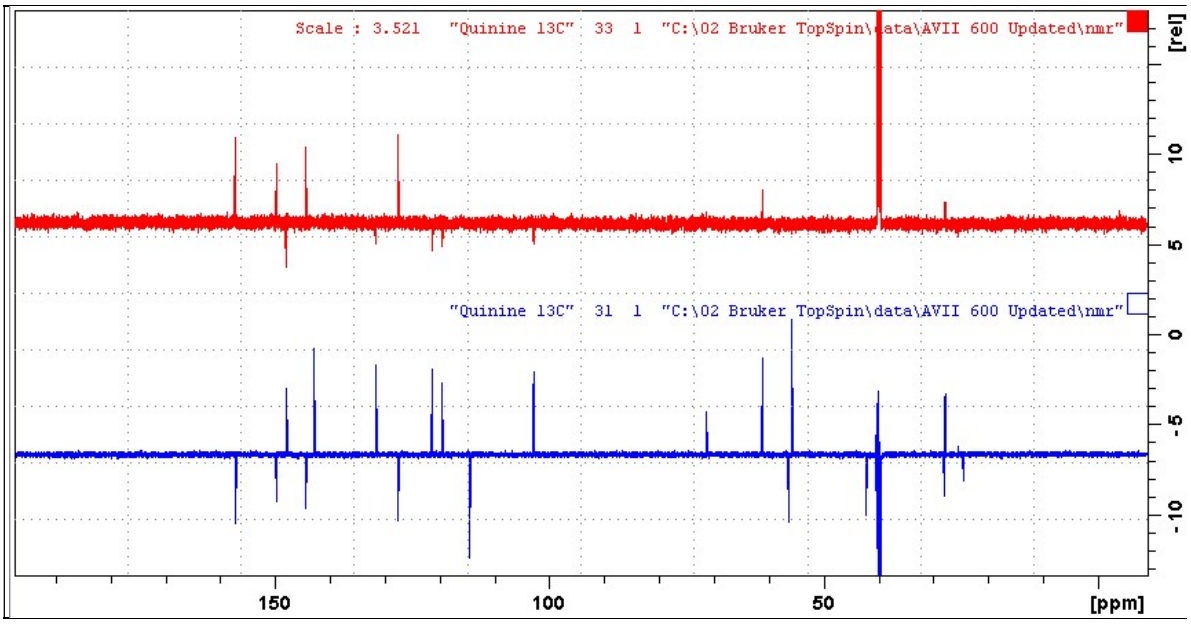
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

NS = Any number, **DS** = 4, 8 or 16.
D1 = 1.5 sec (APT) or 3-4 sec (APTQ) spectra or other time of your choice.
CNST2 = 145 Hz = ^{1}J (^{13}C-^{1}H) 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).*

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

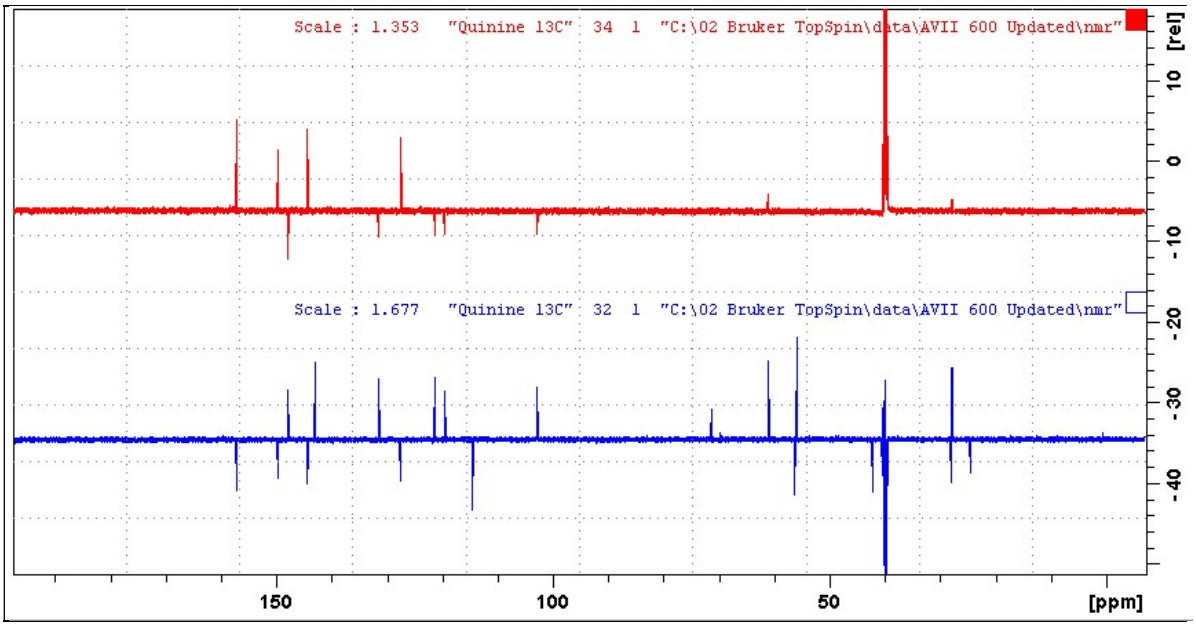
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

NS = multiple of 4 or 8, DS = 4, 8 or 16.
P0 = 90° or other tip angle of your choice (30° - 90°).
D1 = 1.5 sec (ATPSP) or 4 sec (APTQSP) spectra or other time of your choice.
CNST2 = 145 Hz = ^{1}J (^{13}C-^{1}H) 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!).*

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.