KJM 9250

AVI-600 and AVII-600 ^{13}C NMR spectra

Version 5.0

Topspin 3.5 Windows 7
Topspin 1.3 Windows XP

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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.

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 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.**

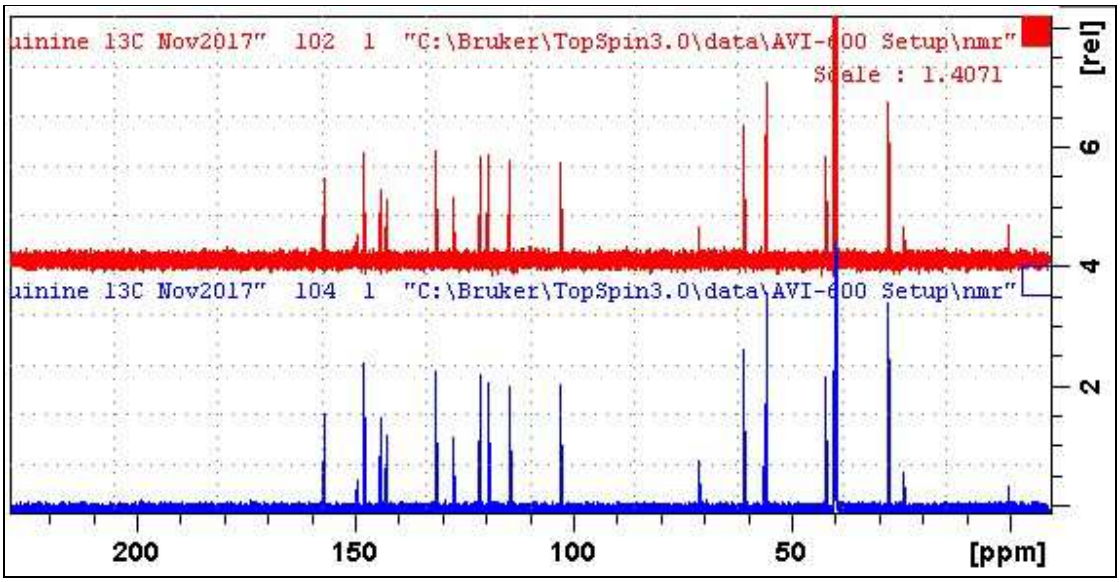
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.
NS = Any number, **DS** = 4, 8 or 16.

D1 = 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 -650º) 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 70º pulse (NS = 8).
Upper: ^{13}C spectrum of quinine in D_6-DMSO with a 30º pulse (NS = 8).

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

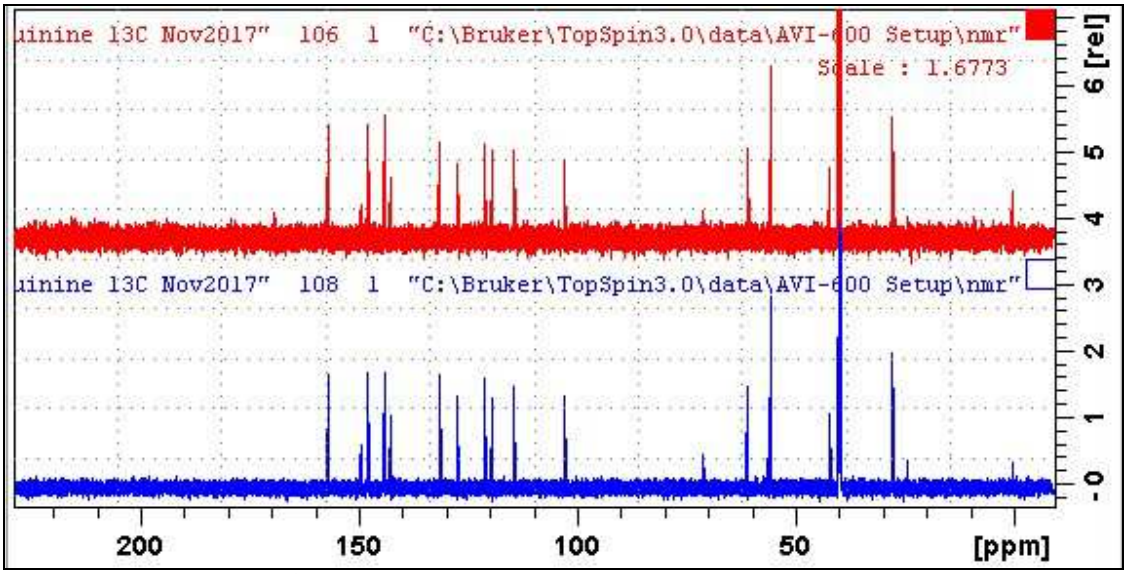
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

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 (NS = 16).
Upper: Inverse gated ^{13}C spectrum of quinine in D_6-DMSO with a 30° pulse (NS = 16).

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

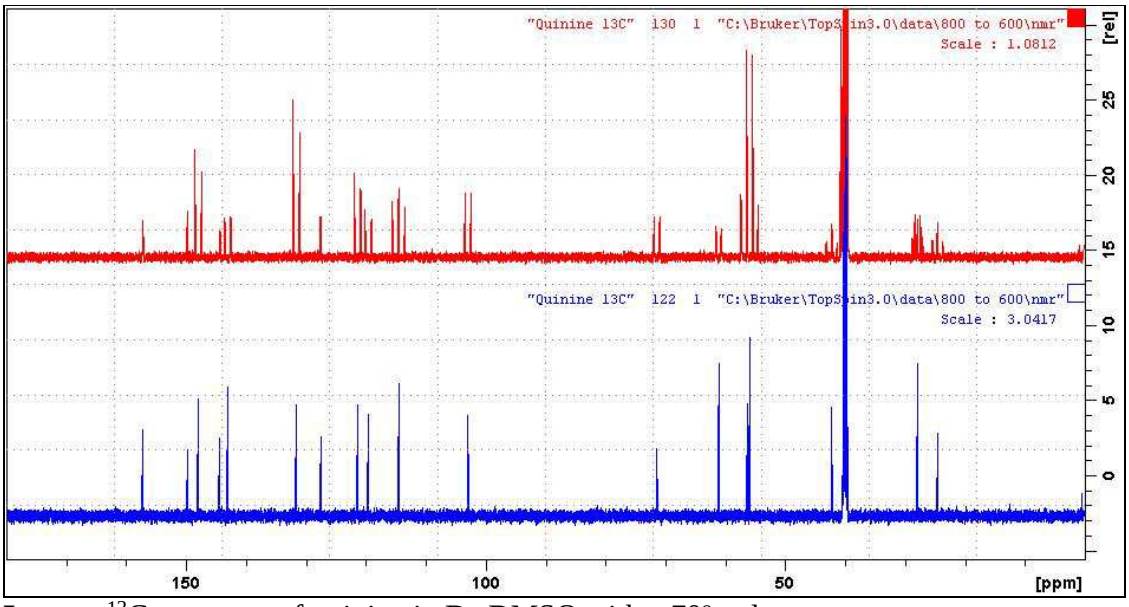
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

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.

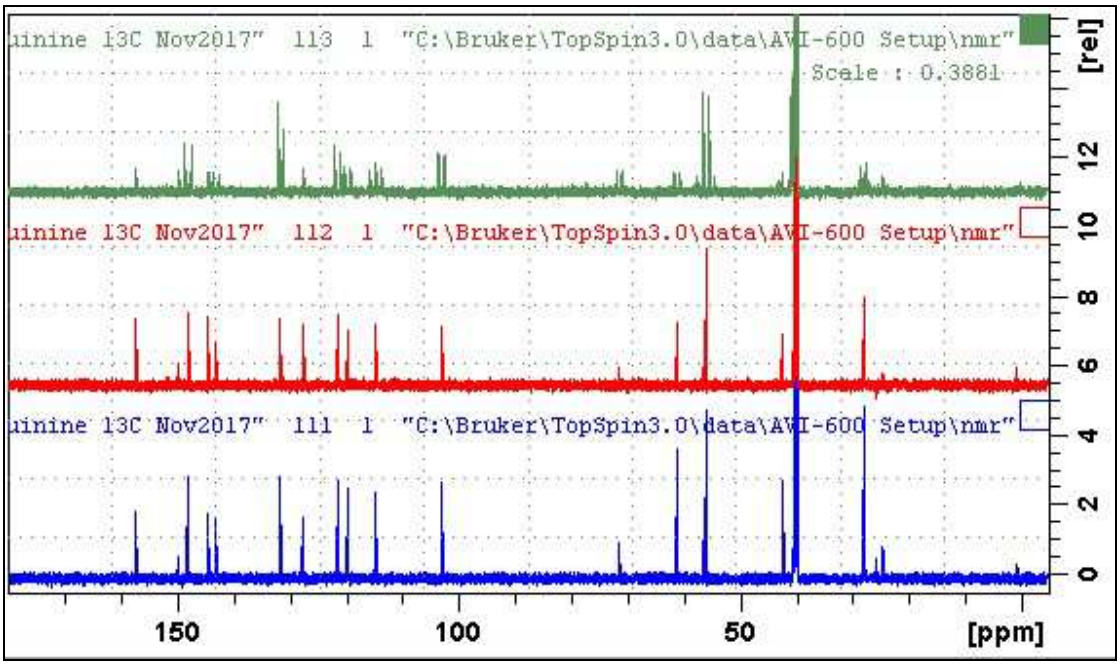
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

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).

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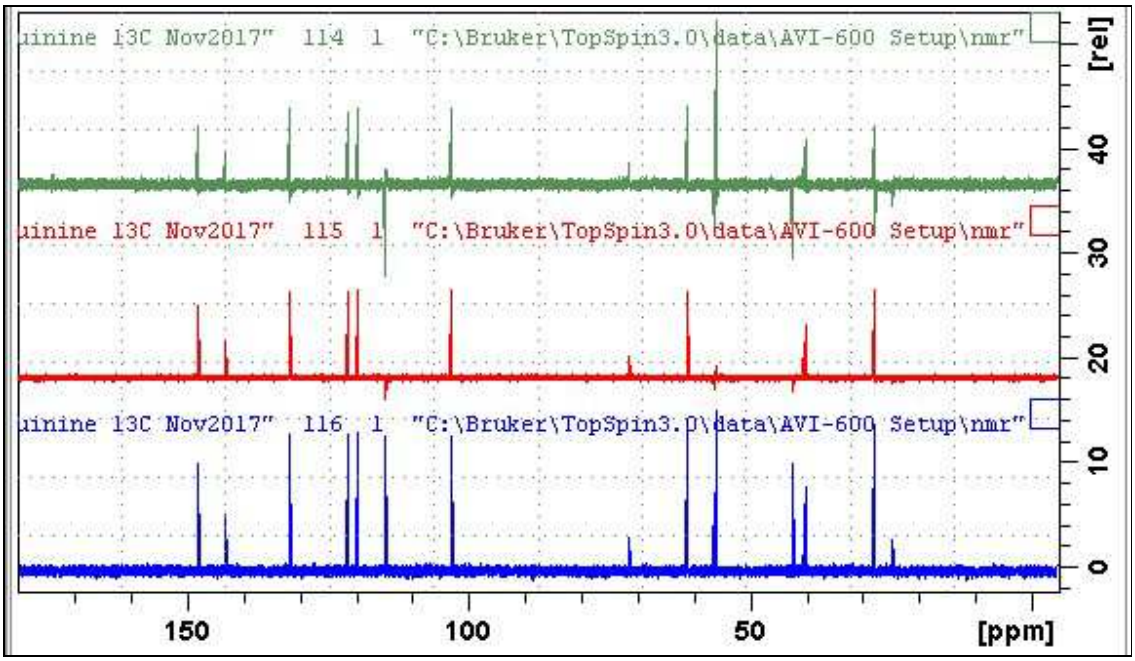

DEPT spectra of quinine in D_6-DMSO. **Lower: DEPT45** spectrum.
Center: DEPT90 spectrum. **Upper: DEPT135** spectrum.

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).
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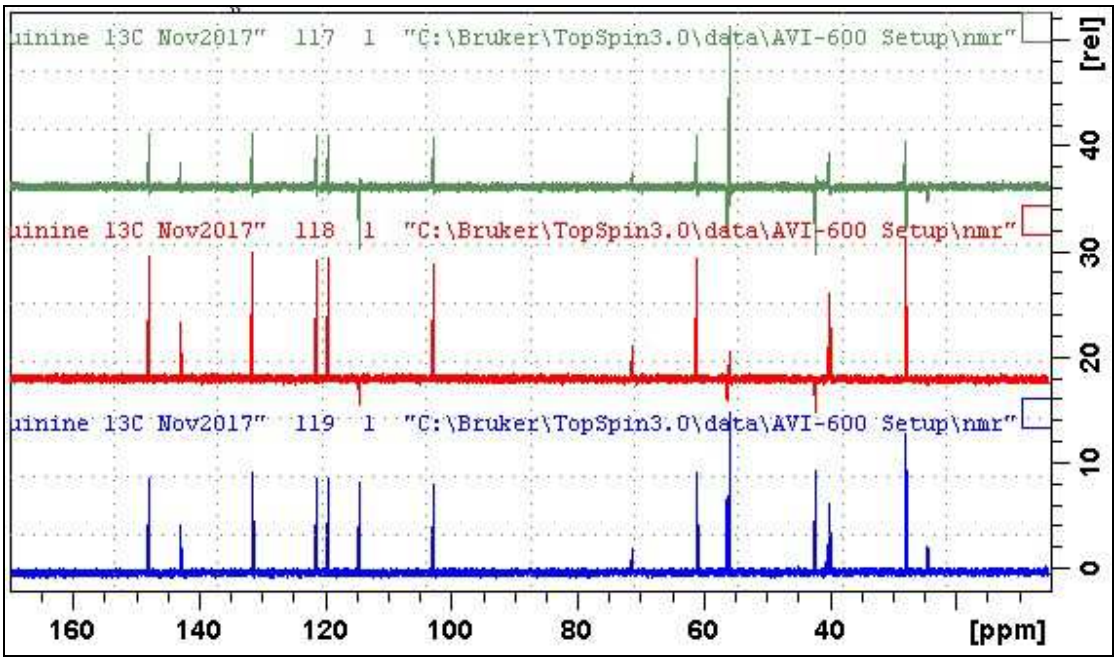


DEPTSP spectra of quinine in D_6-DMSO. **Lower: DEPT45SP** spectrum.
Center: DEPT90SP spectrum. **Upper: DEPT135SP** spectrum.

2.5.3 ^{1}H Coupled DEPTND Spectra

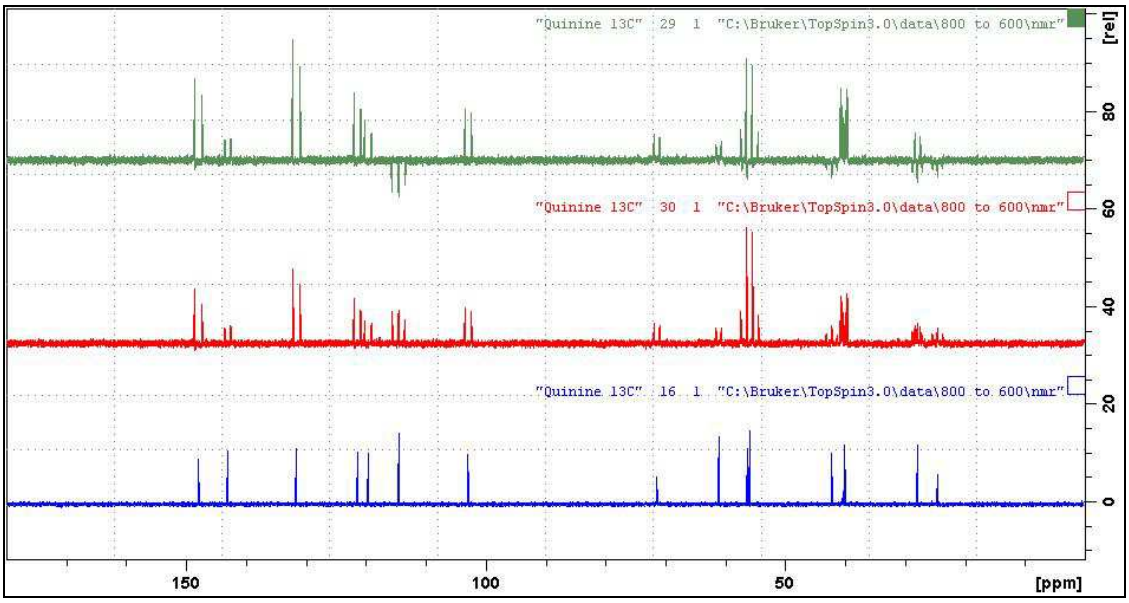
Parameter set: **awdeptnd (+ getprosol)**
Pulse programme: **deptnd** with **P0 =** 45, 90 or 135 degree pulse

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.
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.
Center: DEPT45ND spectrum. **Upper: DEPT135ND** spectrum.

2.6.1 DEPT135Q spectrum with quaternary carbons

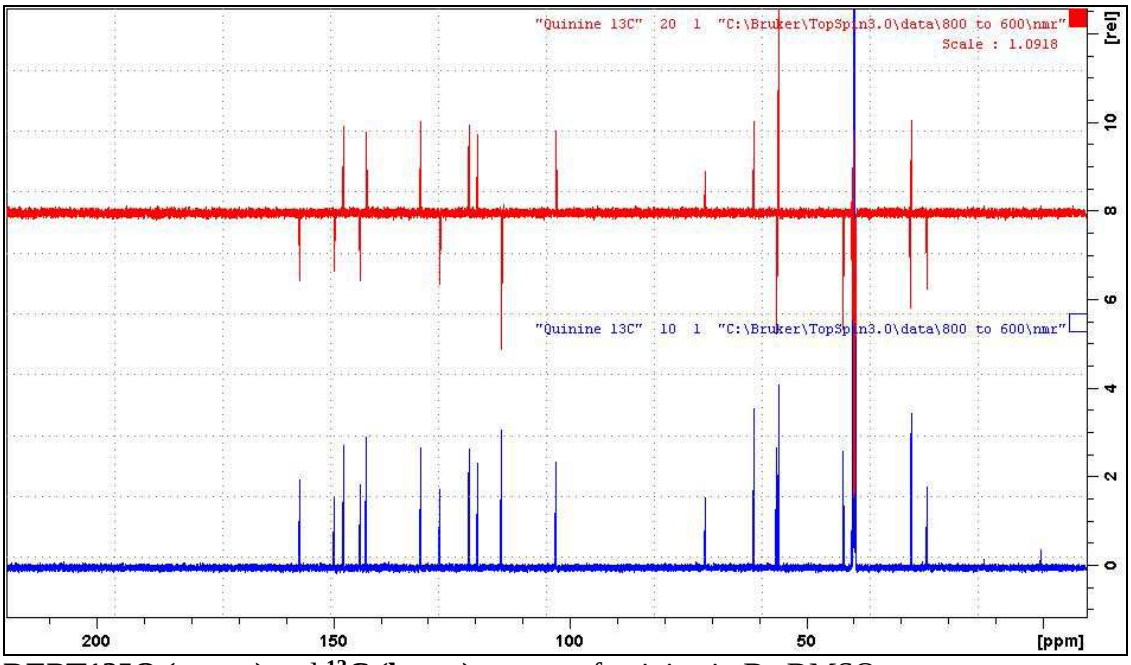
Parameter set: **awdept135q (+ getprosol)**
Pulse programme: **deptqgpsp**

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 = 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).



DEPT135Q (upper) and **^{13}C (lower)** spectra of quinine in D_6-DMSO**.**
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).

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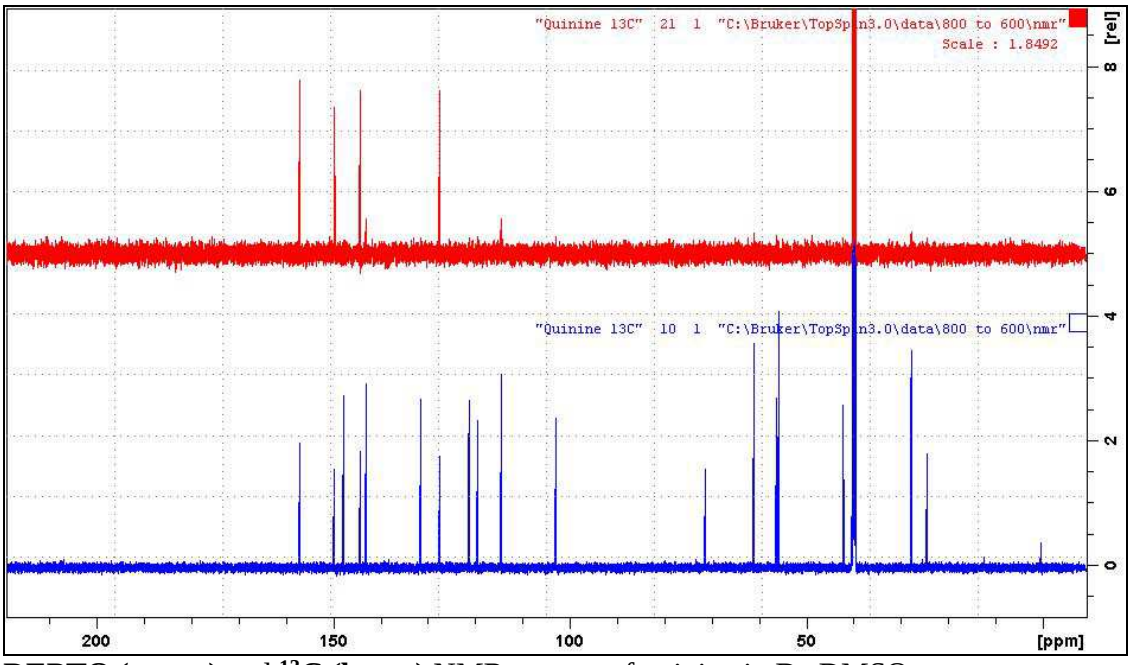


DEPTQ (upper) and **^{13}C (lower)** NMR spectra of quinine in D_6-DMSO.

2.7.1 INEPT45, INEPT90 or INEPT135 spectra

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

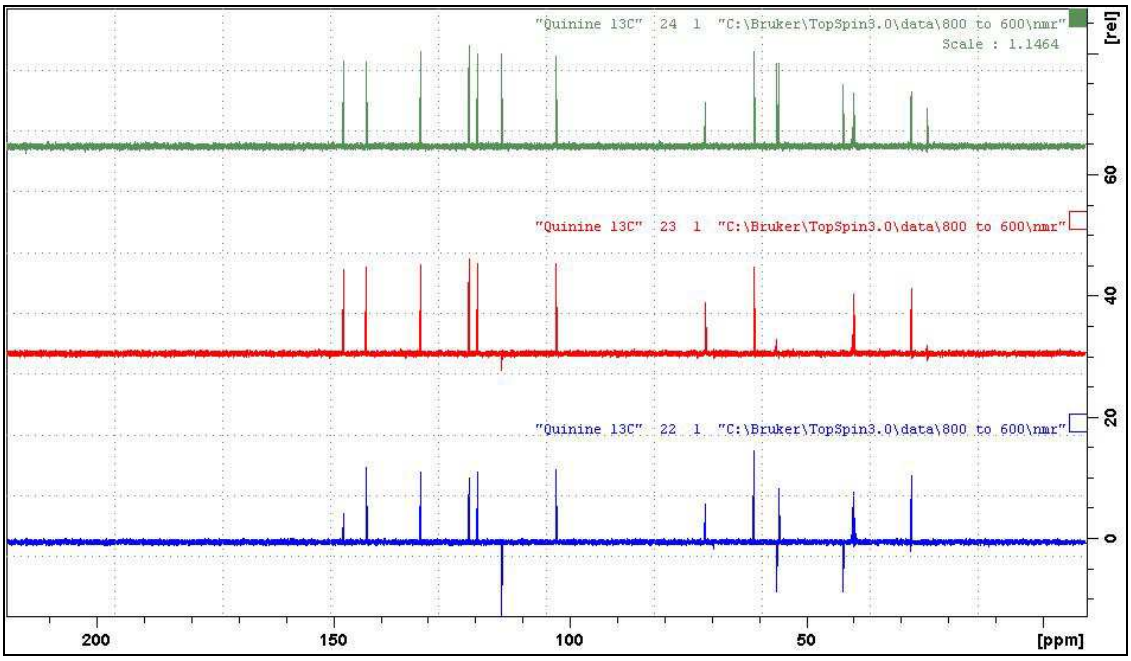
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

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 = **3** for INEPT45,
= **4** for INEPTT90
= **6** 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:*** INEPT135 spectrum.
Center: INEPT90 spectrum ***Upper:*** INEPT45 spectrum.

2.7.2 INEPT45SP, INEPT90SP or INEPT135SP spectra

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

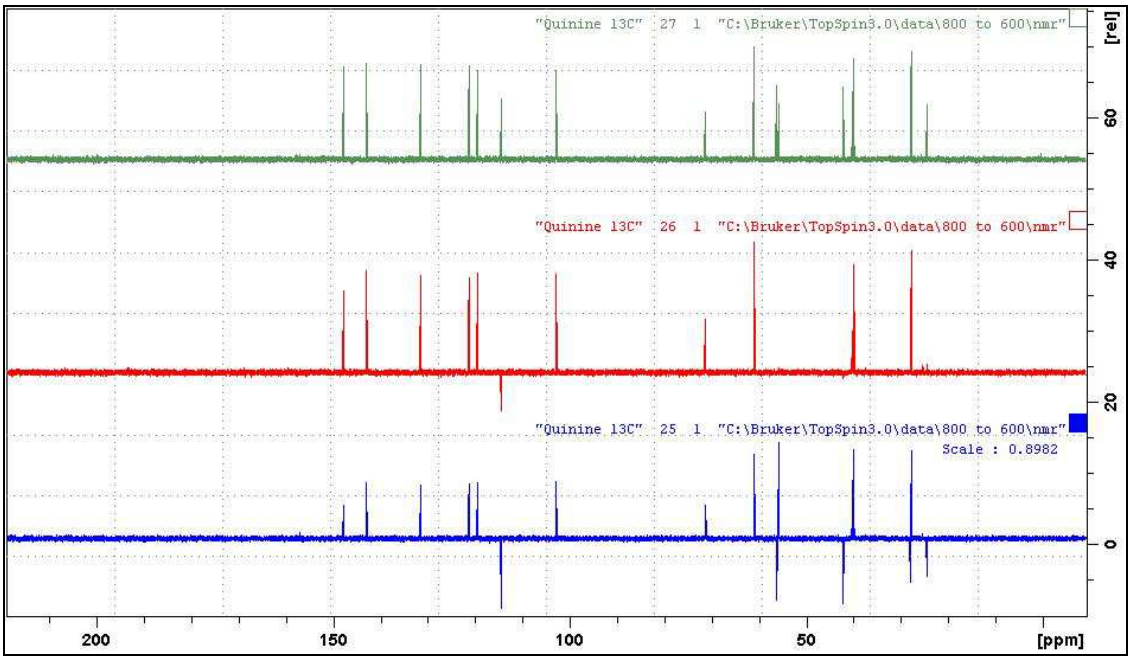
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

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 = **3** for INEPT45SP,
= **4** for INEPTT90SP
= **6** 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:*** INEPT135SP spectrum.
Center: INEPT90SP spectrum ***Upper:*** INEPT45SP spectrum.

2.7.3 INEPTND spectrum

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

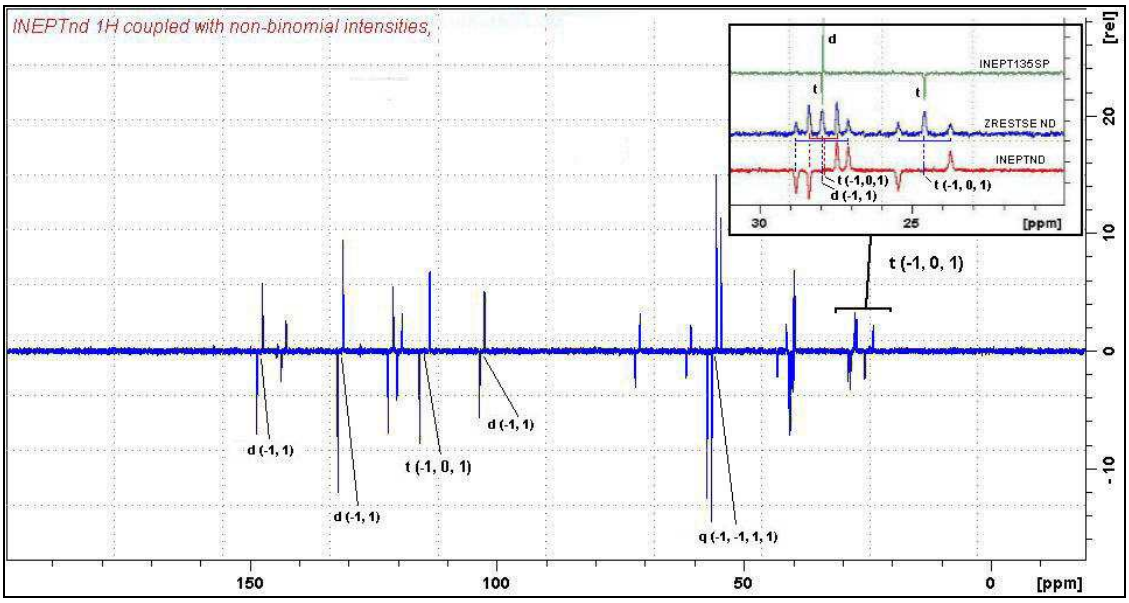
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

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 muitpilicity 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**

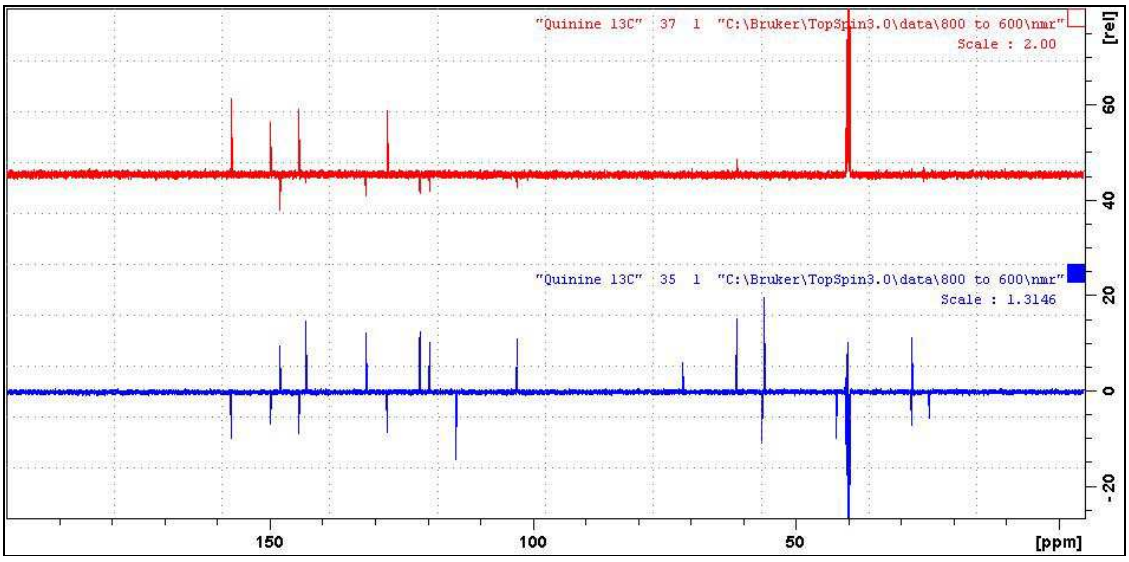
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

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

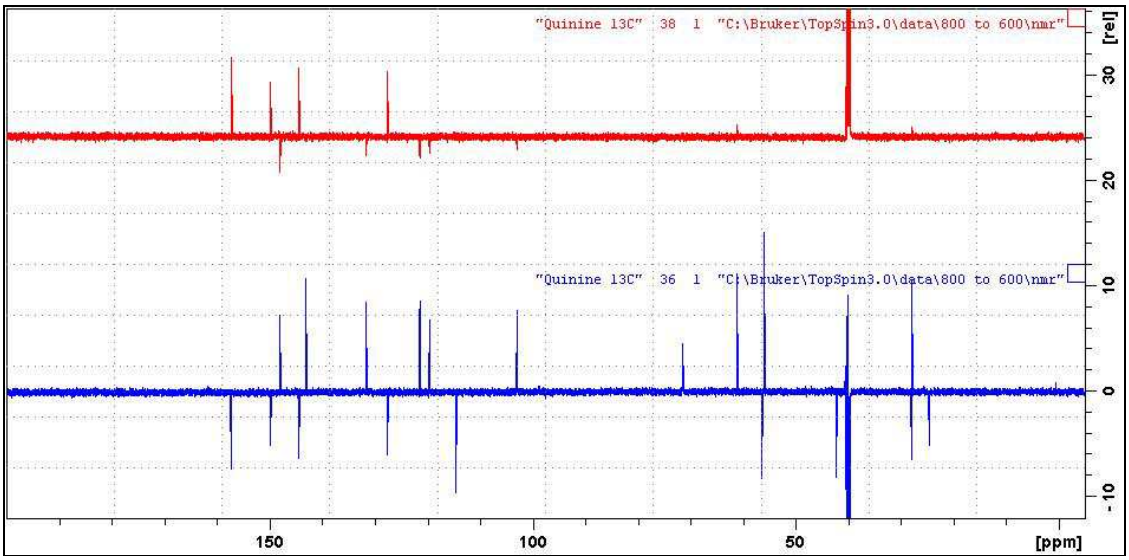
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

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

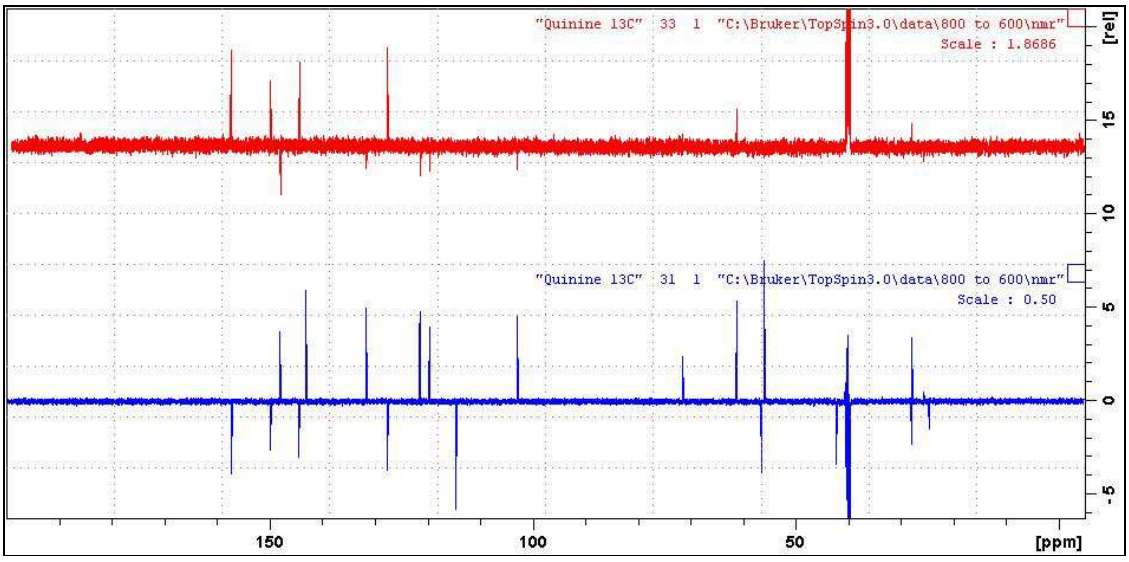
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

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

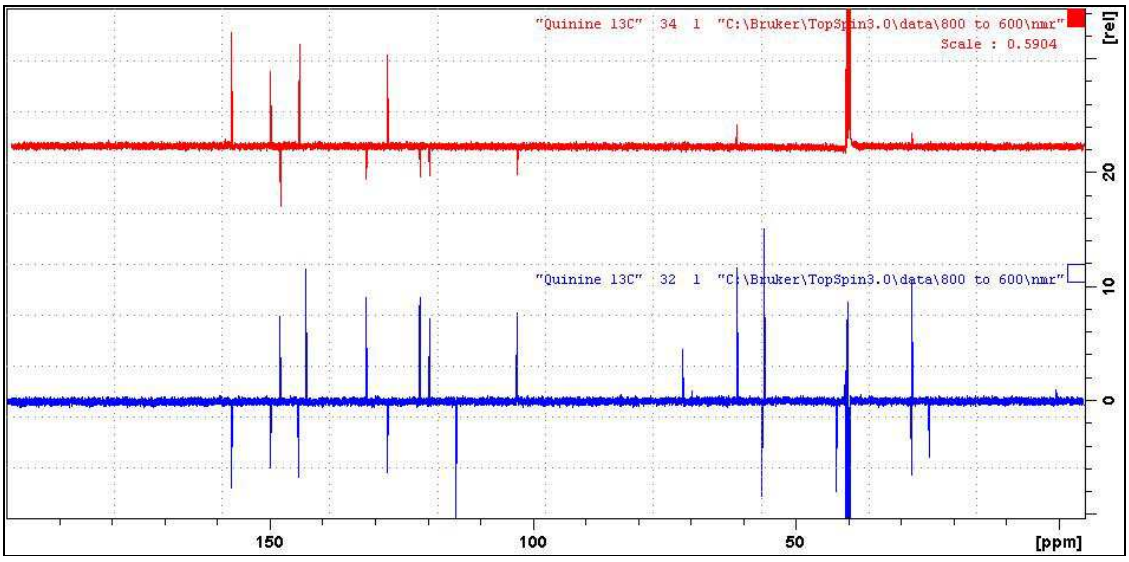
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.

NS = multiple of 4 or 8, DS = 4, 8 or 16.
P0 = 90º or other tip angle of your choice (30º - 90º).
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.