KJM 9250

AVI-600 ^{13}C NMR spectra

Version 7.3

Topspin 1.3 Windows XP AVI600

AVI-600 ^{13}C NMR spectra

1.0 Introduction

aw coded AVI-600 carbon parameter files typically have 64 K points across a 240 ppm window with D1 typically = 1.5 or 4 sec for quaternary 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 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.
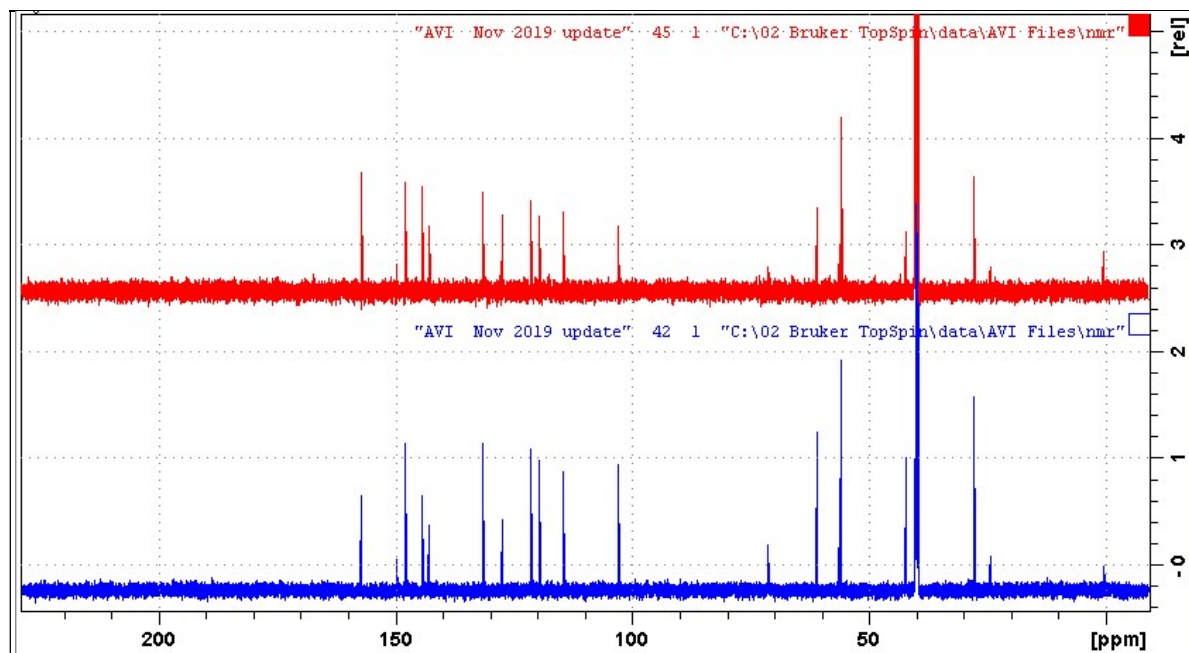**SW** = 240 ppm, **O1P** = 110 ppm.
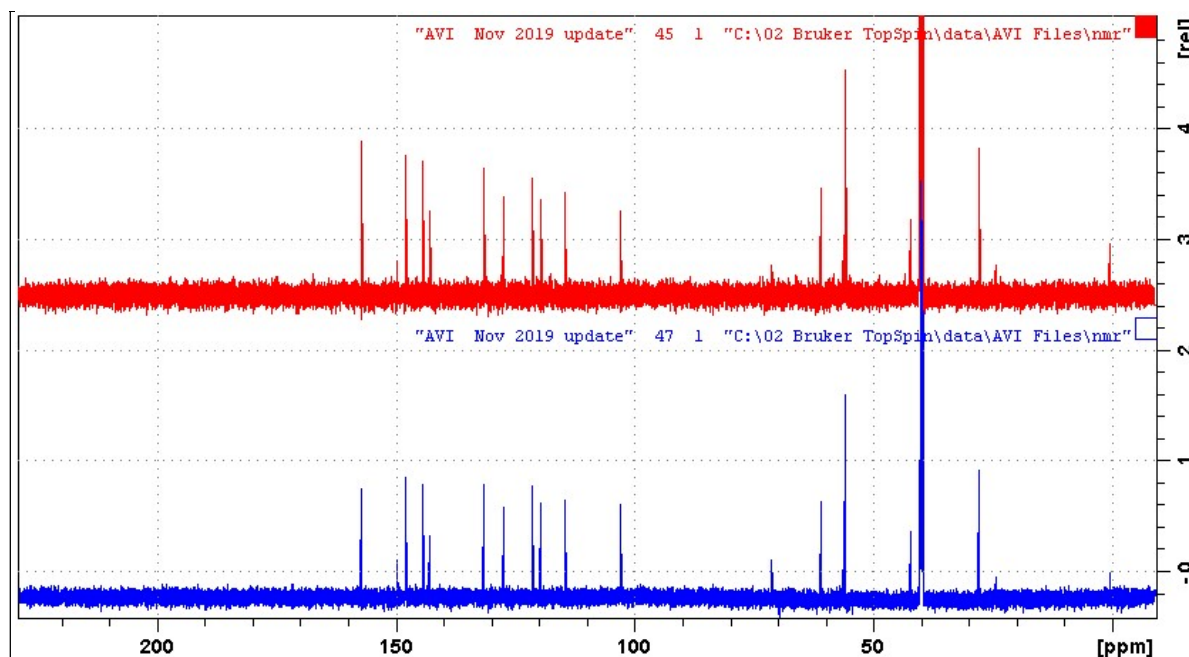**NS** = Any number, **DS** = 4, 8 or 16.

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 =
30-40.

Lower: ^{13}C spectrum of quinine in D$_6$-DMSO with a 70º pulse.
Upper: ^{13}C spectrum of quinine in D$_6$-DMSO with a 30º pulse.

2.2 Inverse gated ^{13}C NMR spectra

Parameter sets: **awcarbon30ig, awcarbon45ig, awcarbon70ig, awcarbon90ig (+ getprosol)**
Pulse programmes: **zgig30, awzgig45, awzgig70, zgig respectively**
   Spectra are ^{1}H decoupled <u>without</u> 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° 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°) will be required. After phasing baseline roll can be eliminated using the **multiabsn** macro with n = 30-50.
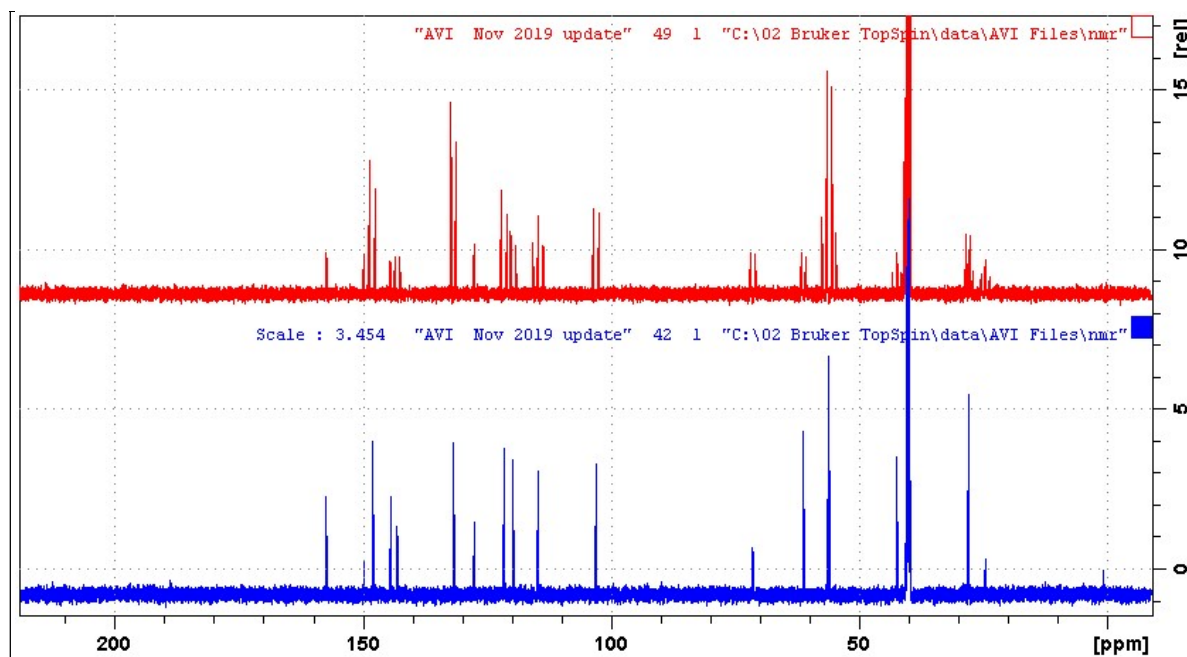
Lower: ^{13}C spectrum of quinine in D_6-DMSO with a 70° pulse.
Upper: ^{1}H coupled ^{13}C spectrum of quinine in D_6-DMSO with a 70° pulse.

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.
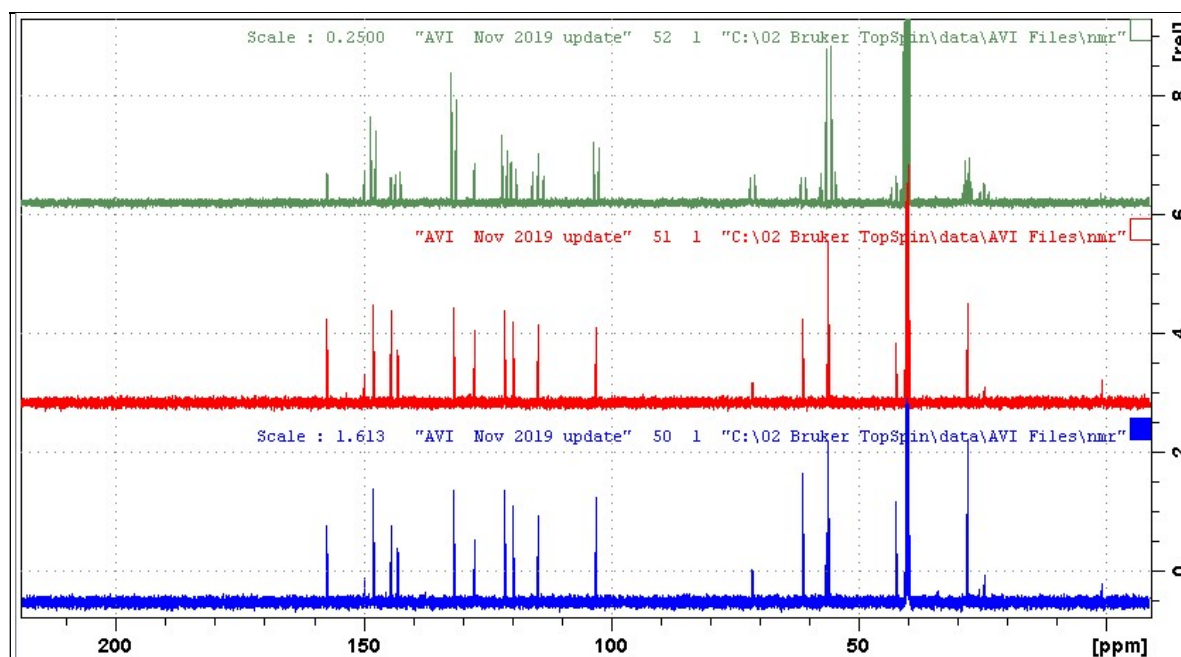**P0 = P1*CNST0/90.0**; **CNST0 = 70º** or other tip angle of your choice (30º-90º range).
D1 = **1.5 sec** (zrestse and zrestseig) or **4 sec** (zrestsend) or other time of your choice.

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 70º P0 pulse.
Lower: power gated **zrestse** spectrum. *Center:* inverse gated **zrestseig** spectrum.
Upper: ^1H 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.
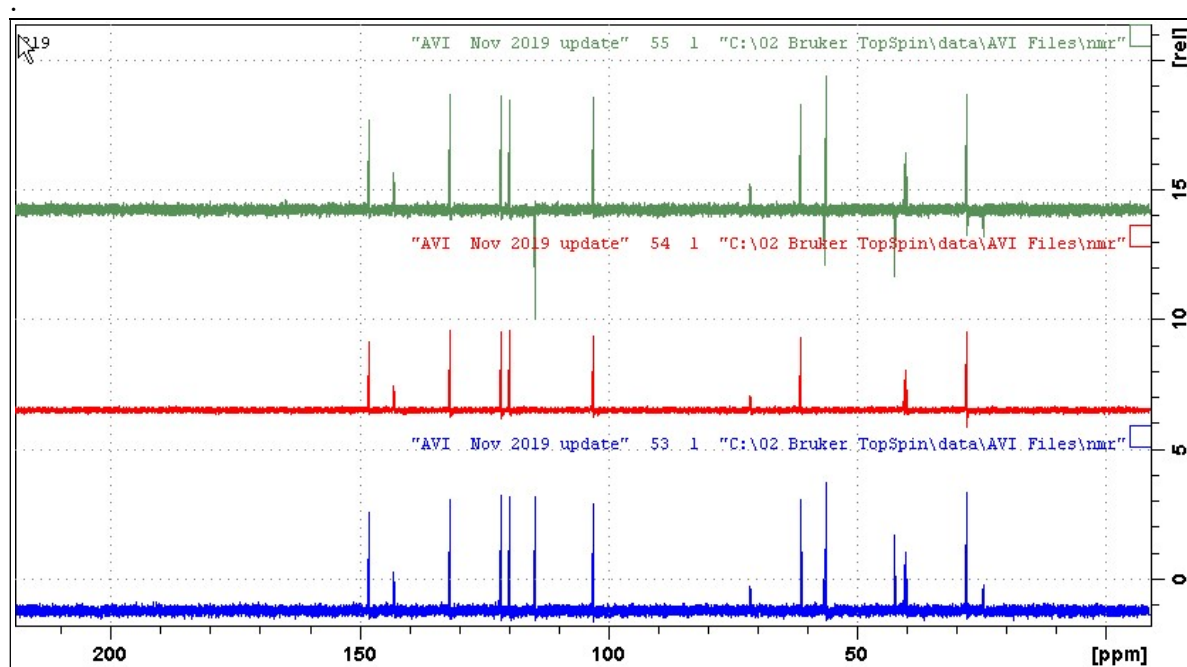**NS** = Any number, **DS** = 4, 8 or 16.

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

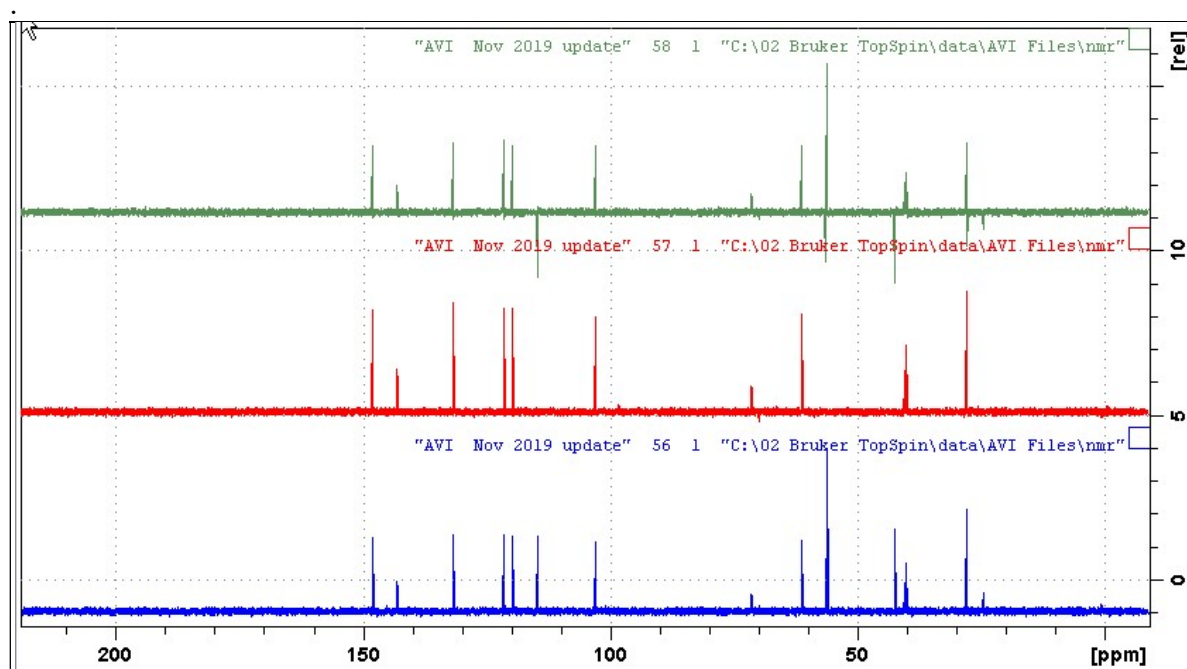**NS** = Any number, **DS** = 4, 8 or 16.
D1 = 1.5 sec or other time of your choice.
CNST2 = 145 Hz = ^1J (^{13}C-^1H) or other value of your choice.

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: **awdeptnd** with a **P0 = 45** degree pulse

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

NS = Any number, **DS** = 4, 8 or 16.
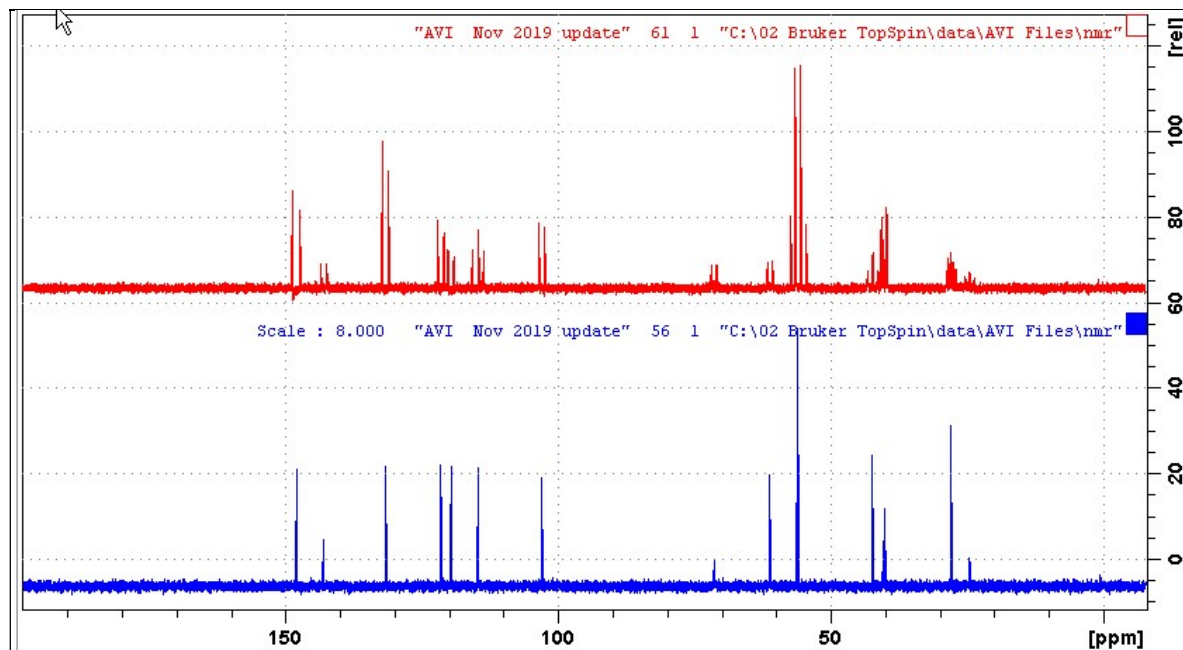**D1** = 1.5 sec or other time of your choice.
CNST2 = 145 Hz = ^1J (^{13}C-^{1}H) or other value of your choice.
P0 is auto-set up as **0.5*P3** to afford a dept45nd spectrum.

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: DEPTND 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.
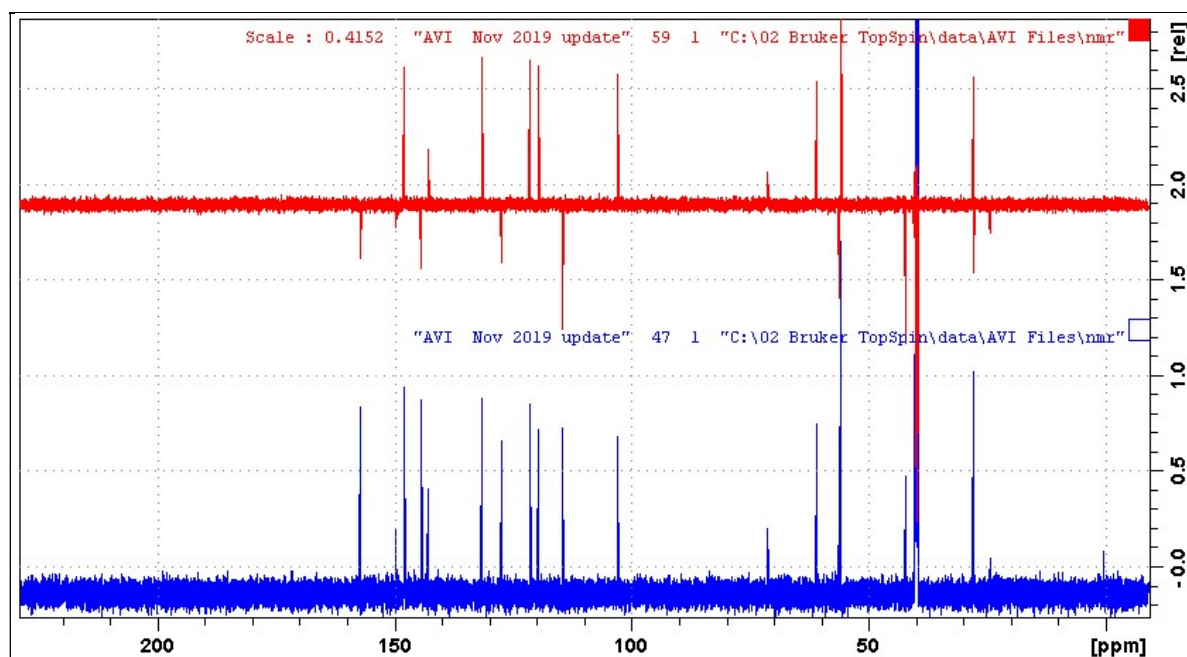**CNST2** = 145 Hz = $^1J\,(^{13}C\text{-}^1H)$ or other value of your choice.
CNST12 = 1.5 for DEPT135 spectrum with quaternary carbons.

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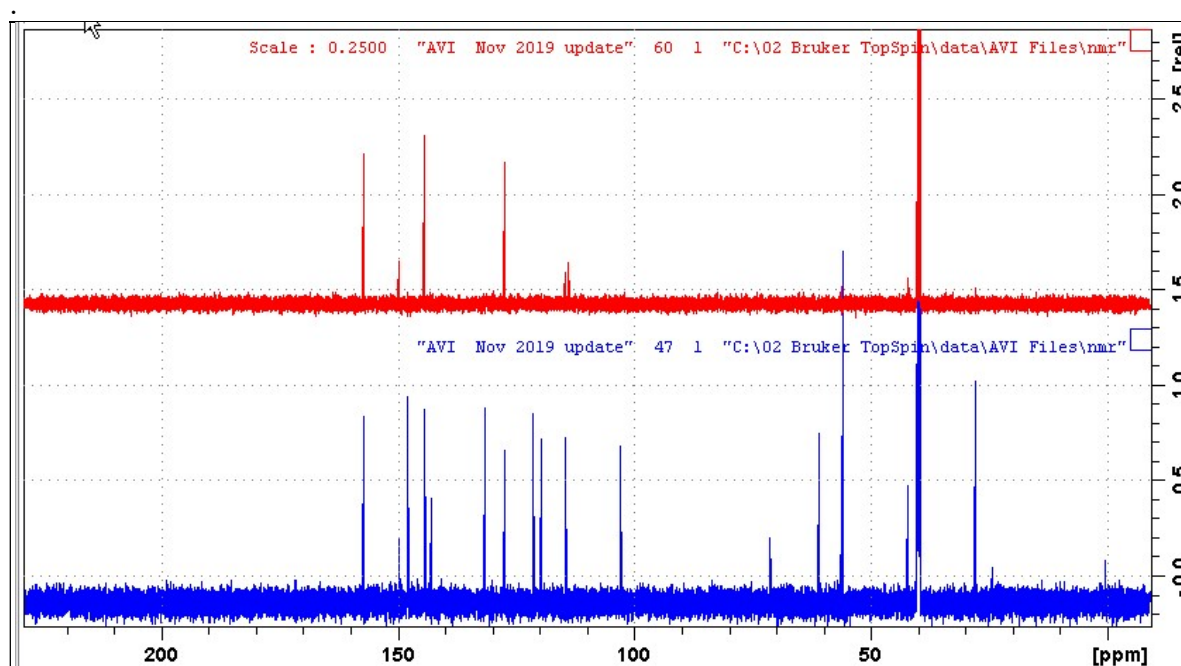**CNST2** = 145 Hz = ^1J (^{13}C-^1H) or other value of your choice.
CNST12 = 1.5 for alternating cancellation of DEPT135 carbon signals.

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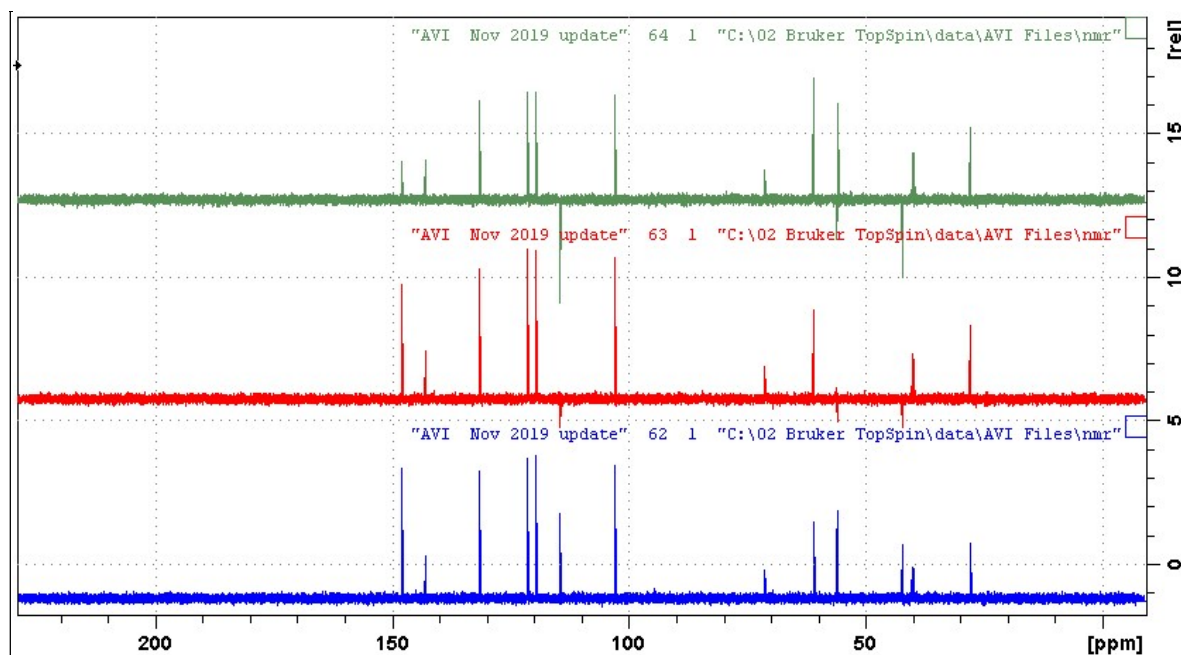**D1** = 1.5 sec or other time of your choice.
CNST2 = 145 Hz = ^1J (^{13}C-^{1}H) or other value of your choice.
CNST11 = **6** for INEPT45,
            = **4** for INEPTT90
            = **3** for INEPT135

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 = $^1J\,(^{13}C\text{-}^1H)$ or other value of your choice.
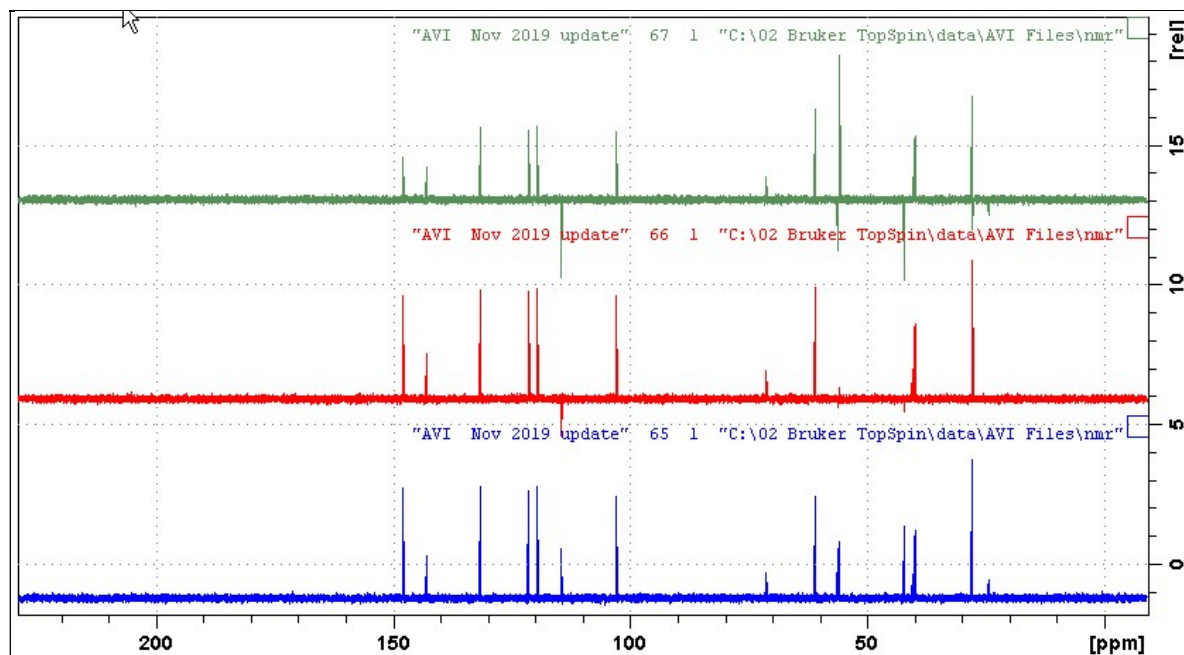**CNST11** = **6** for INEPT45SP,
            = **4** for INEPTT90SP
            = **3** for INEPT135SP

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.
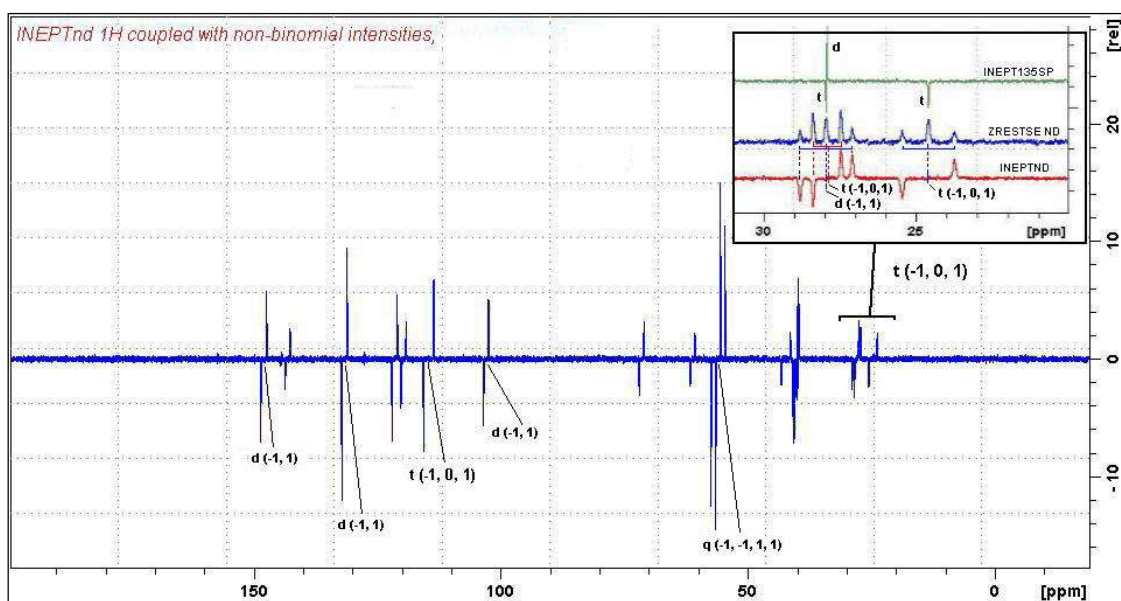**CNST2** = 145 Hz = ^1J (^{13}C-^1H) or other value of your choic.e

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.
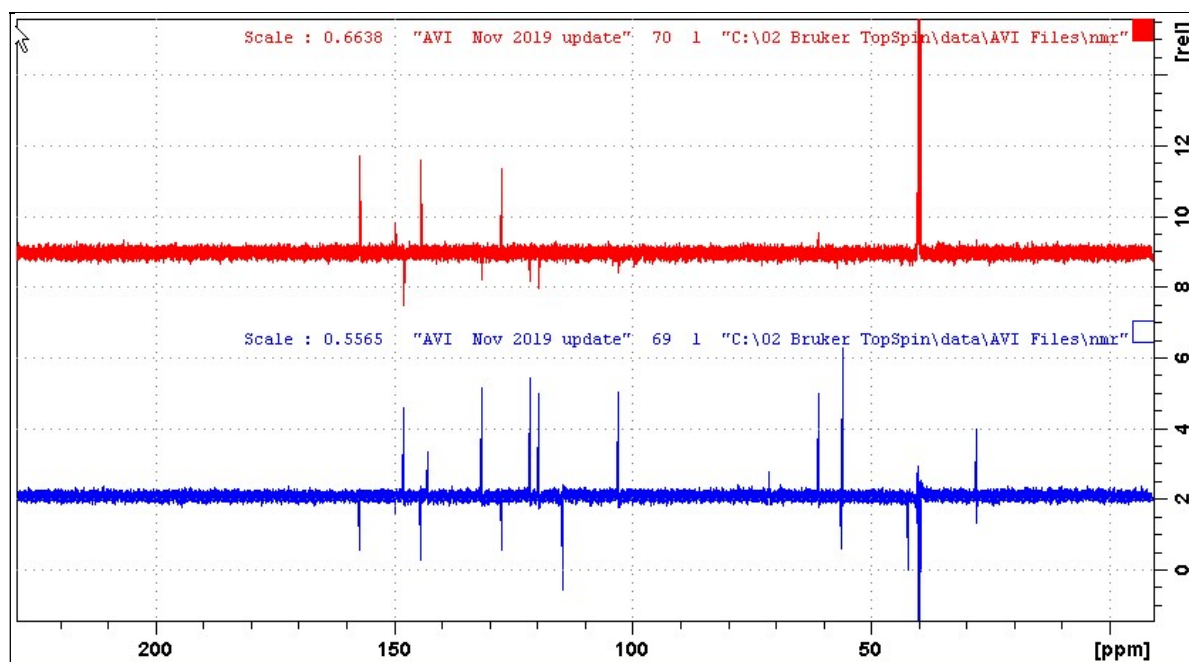**D1** = 1.5 sec (jmod) or 4 sec (jmodq) spectra or other time of your choice.
CNST2 = 145 Hz = ^1J (^{13}C-^1H) or other value of your choice.
CNST11 = 1 (CH, CH$_3$ positive, C, CH$_2$ negative), or
           = 2 for C (quaternary) only carbons.

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 ^1J 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.
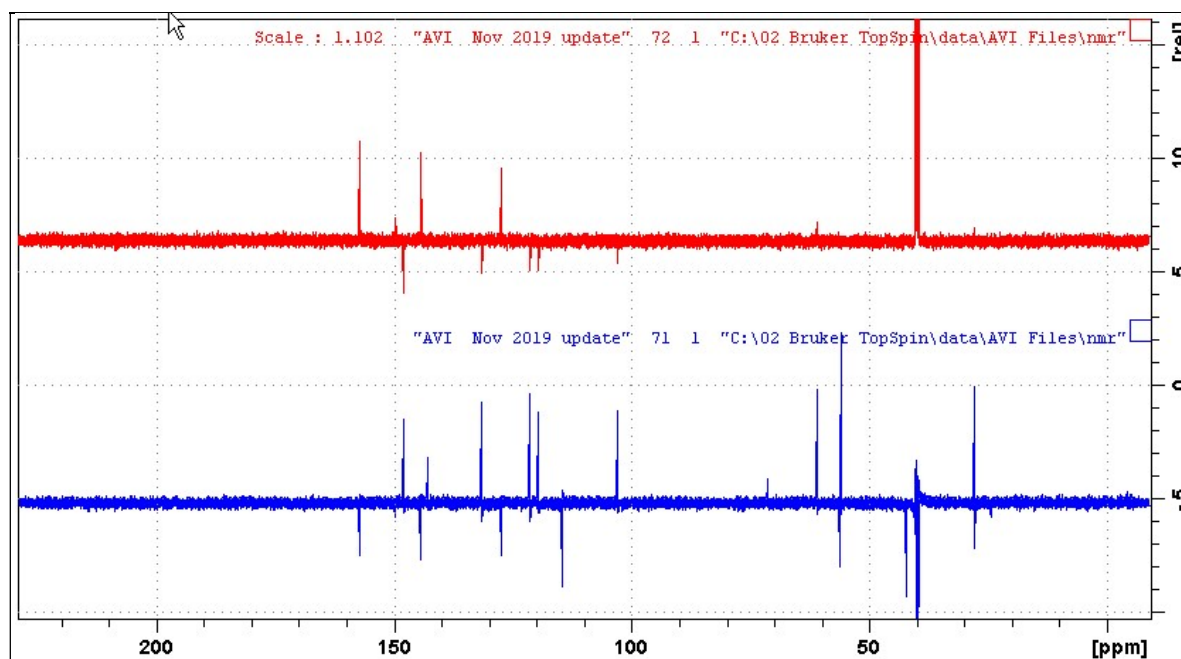**CNST2** = 145 Hz = ^1J (^{13}C-^1H) or other value of your choice.
CNST11 = 1 (CH, CH$_3$ positive, C, CH$_2$ negative), or
          = 2 for C (quaternary) only carbons

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 ^1J 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.
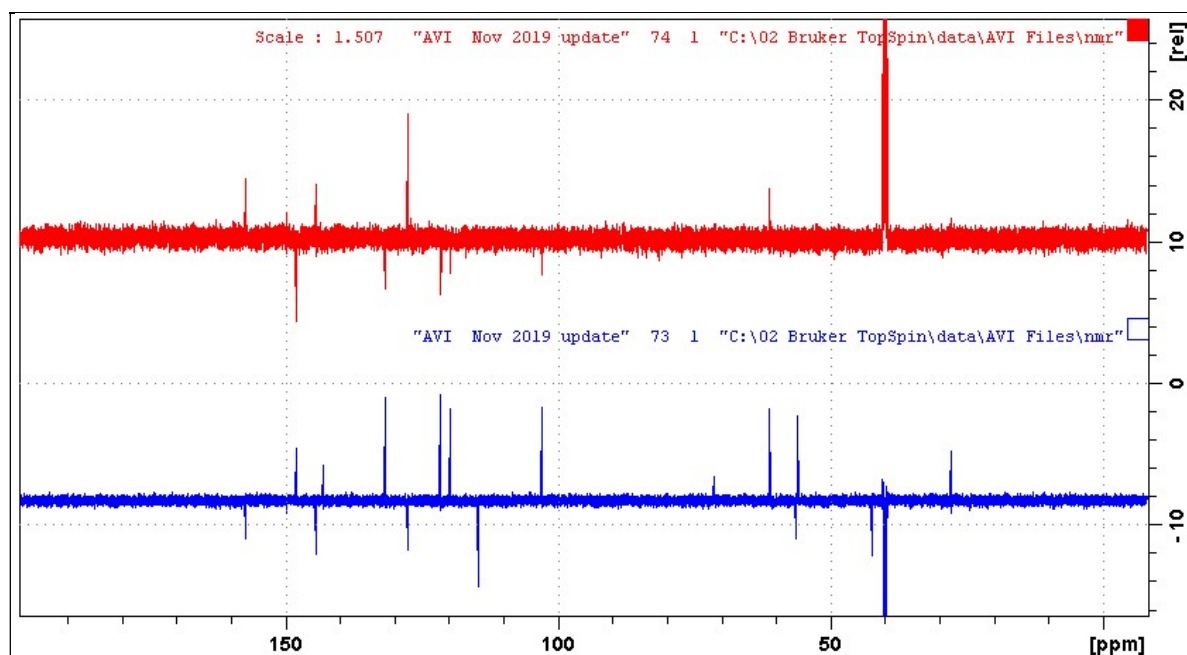**D1** = 1.5 sec (APT) or 3-4 sec (APTQ) spectra or other time of your choice.
CNST2 = 145 Hz = ^1J (^{13}C-^1H) or other value of your choice.
CNST11 = 1 (CH, CH$_3$ positive, C, CH$_2$ negative), or
$\qquad$ **= 2** for C (quaternary) only carbons.

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 ^1J 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°).
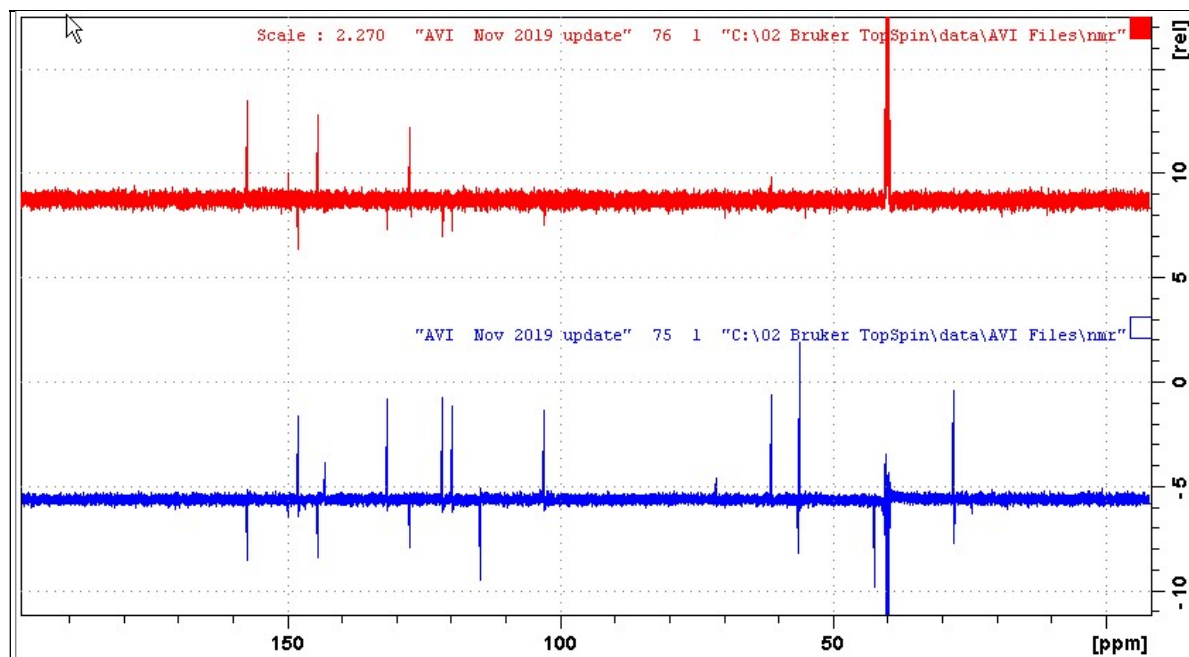**D1** = 1.5 sec (ATPSP) or 4 sec (APTQSP) spectra or other time of your choice.
CNST2 = 145 Hz = ^1J (^{13}C-^1H) or other value of your choice**.**
CNST11 = 1 (CH, CH$_3$ positive, C, CH$_2$ negative), or
                   = 2 for C (quaternary) only carbons.

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 ^1J coupling constants differ significantly from CNST2 = 145 Hz.