KJM 5250 and KJM 9250

^{13}C NMR spectra on the AVneo400 spectrometer.

Version 3.1

Topspin 4.3

© Professor Emeritus Alistair Lawrence Wilkins,
University of Waikato, New Zealand.
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© Professor Frode Rise, University of Oslo, Norway.
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AVneo400 ^{13}C NMR spectra. Topspin 4.3

1.0 Introduction

A series of aw coded **^{1}H NMR** parameter sets have been created on the **NEO400** spectrometer. **NEO400** spectra can be processed using **Topspin 3.x or TS4**.

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

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 spectrum (no NOE)**
- **2.3 DEPT45, DEPT90 and DEPT135 spectra**
- **2.4 Coupled DEPT spectra**
- **2.5 DEPT135SP spectrum**
- **2.6 DEPT135Q spectrum with quaternary carbons**
- **2.7 DEPTQ quaternary (singlet) carbons only spectrum**
- **2.8 JMOD spectra**
- **2.9 APT spectra**

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

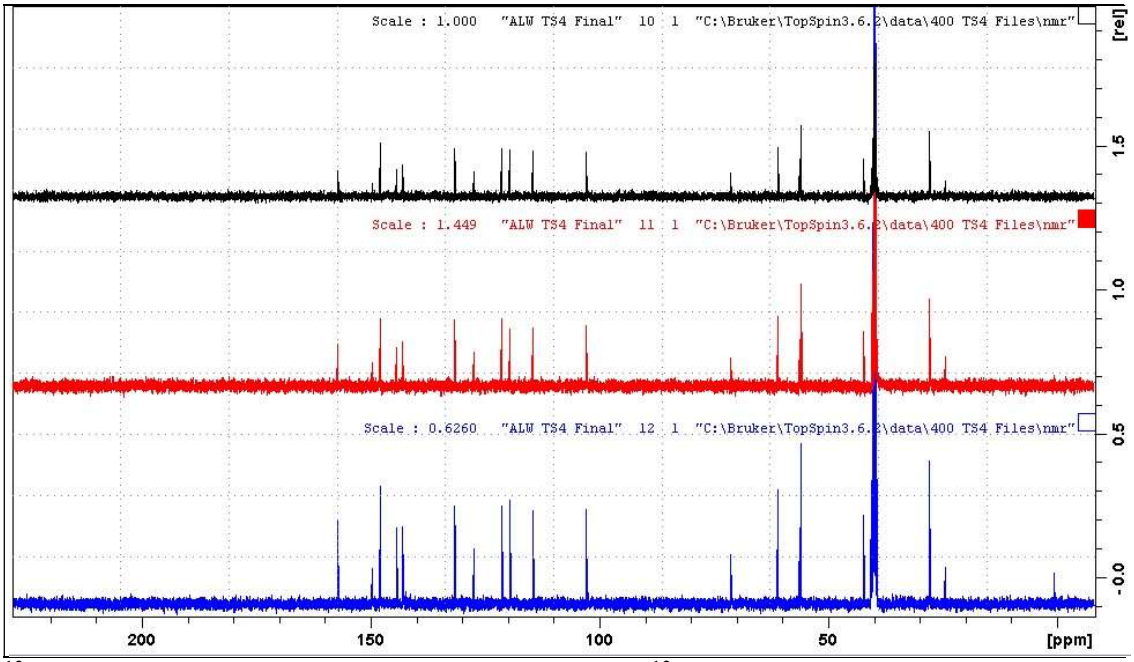
Parameter sets: **awcarbon45, awcarbon70** or **awcarbon90 (+ getprosol)**
Pulse programmes: **awzgpg45, awzgpg70** or **zgpg** respectively.
Spectra are ^{1}H decoupled with NOE.

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 .

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

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

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



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

2.2 Inverse gated ^{13}C spectrum

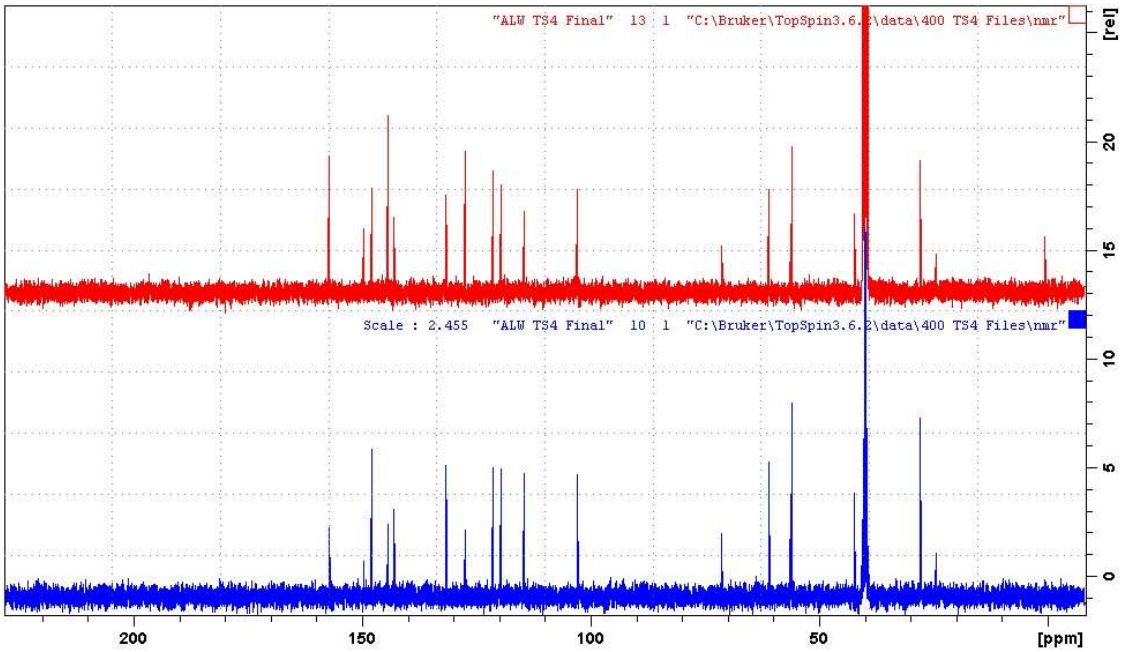
Parameter sets: **awcarbon90ig (+ getprosol)**
Pulse programmes: **zgig**

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.

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

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

Process with **EF** or **EFP** (applies **LB = 30-50**.



Lower: ^{13}C NMR spectrum of quinine in D_6-DMS. ***Upper:*** Inverse gated ^{13}C spectrum with a 90° pulse.

2.3 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!).*

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

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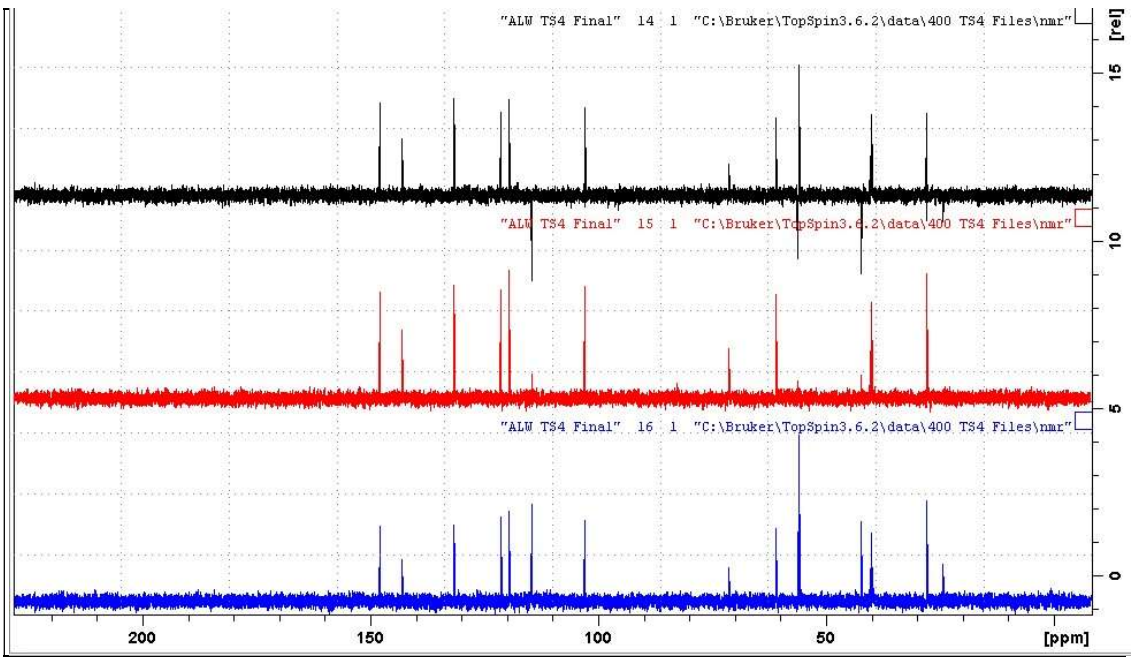


DEPT NMR spectra of quinine in D_6-DMSO. ***Lower:*** DEPT45 spectrum. ***Center:*** DEPT90 spectrum. ***Upper:*** DEPT1355 spectrum.

2.4 ^{1}H Coupled DEPT spectra

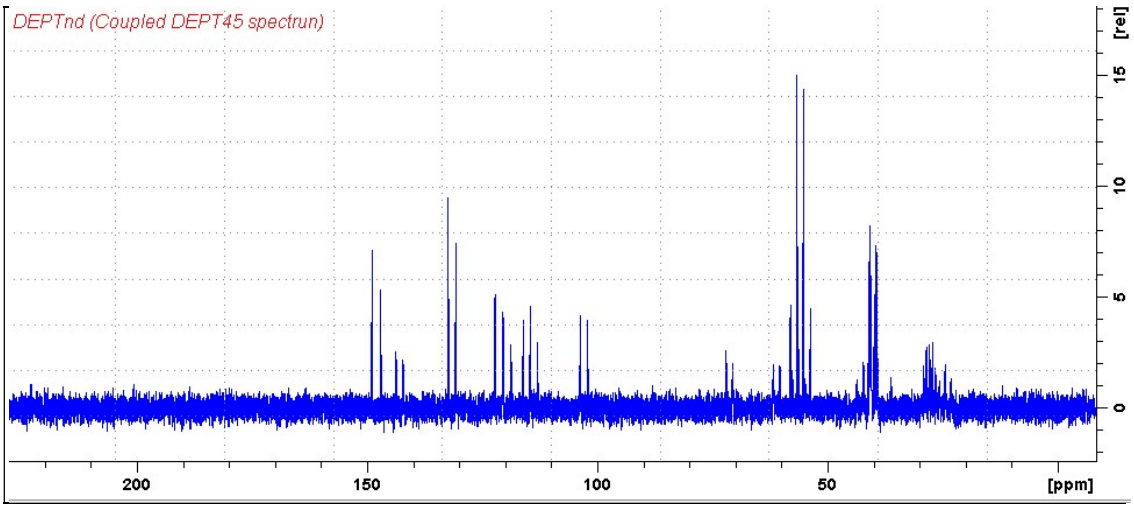
Parameter set: **awdeptnd (+ getprosol)**
Pulse programme: **deptnd** with **CNST12 = 0.5, 1.0 or 1.5**

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!).*
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.

2.5 DEPT135SP spectrum

Parameter set: **awdept135sp (+ getprosol)**
Pulse programmes: **deptsp135**

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!).*

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

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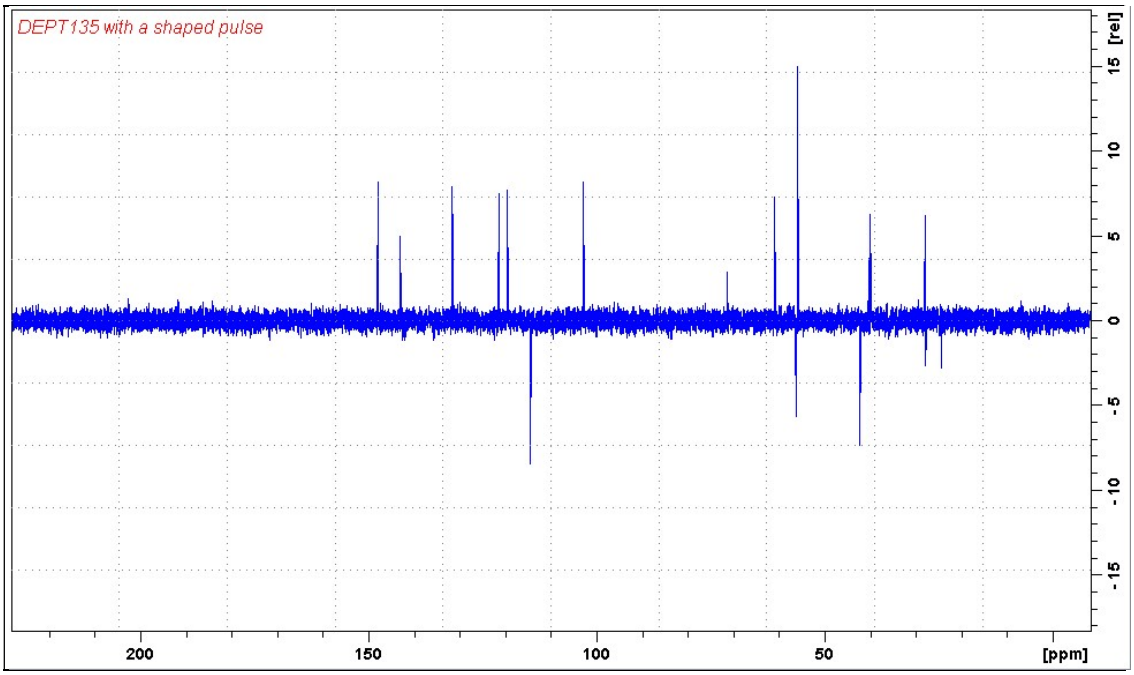


DEPT135SP NMR spectrum of quinine in D_6-DMSO.

DEPTSP spectra with a shaped multiplicity (CH, CH_2, CH_3) selection pulse have wider linear response windows (in Hz) than standard DEPT spectra. While this is not an issue on the at 400 MHz it can be/is an issue at higher fields (eg at 800 MHz). Versions of the DEPT135SP pulse program are available (see below) which afford DEPT135SP spectra with singlet (quaternary) carbons, or quaternary carbons only.

2.6 DEPT135Q spectrum with quaternary carbons

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

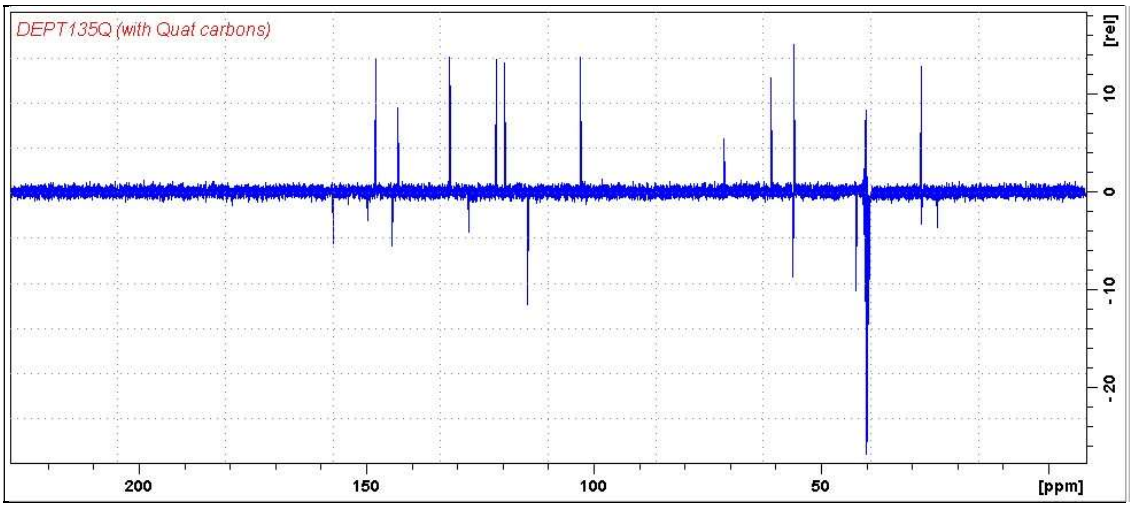
TD = 64 K, **SI** = 64 K.
SW = 240 ppm, **O1P** = 110 ppm.
NS = Any number, **DS** = 4, 8 or 16.

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

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



DEPT135Q NMR spectrum of quinine in D_6-DMSO.

2.7 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!).*

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

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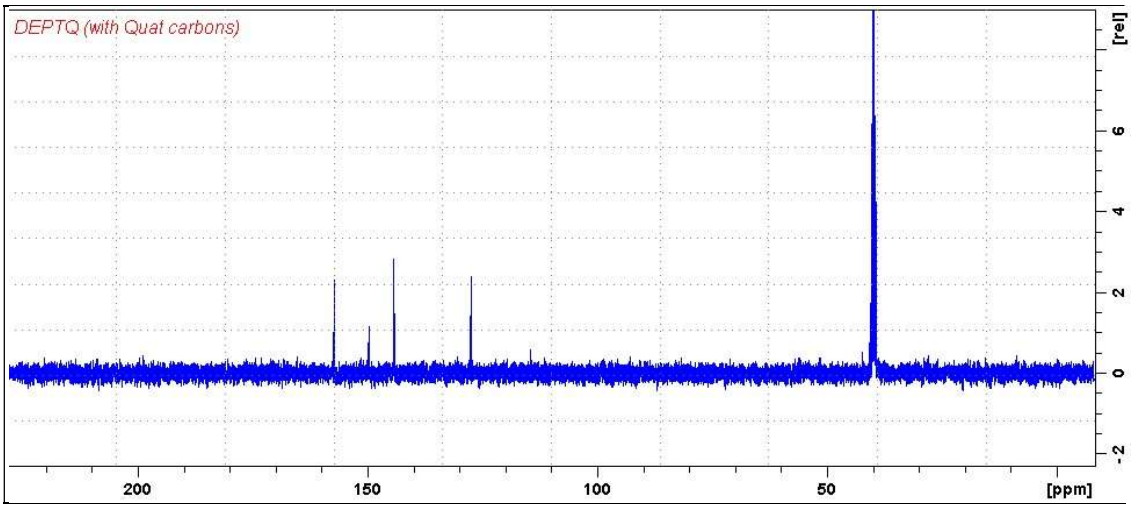


DEPTQ spectrum of quinine in D_6-DMSO.

2.8 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.

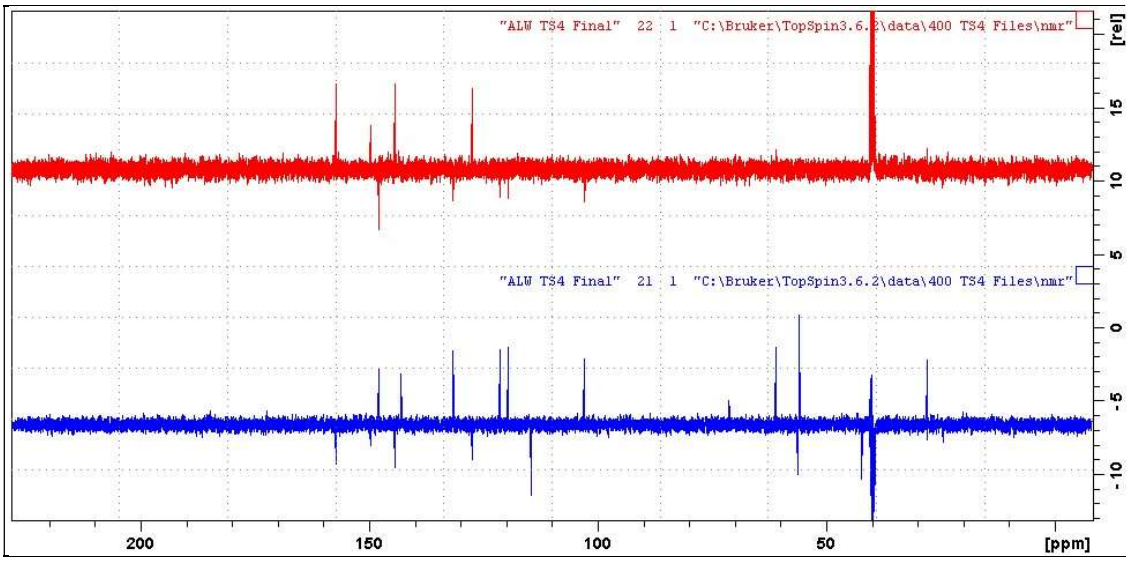
NS = Any number, **DS** = 4, 8 or 16.
D1 = 1.5 sec (awjmodsp) or 4 sec (awjmodspq) spectra or other time of your choice**.**

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).



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

2.9 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°).

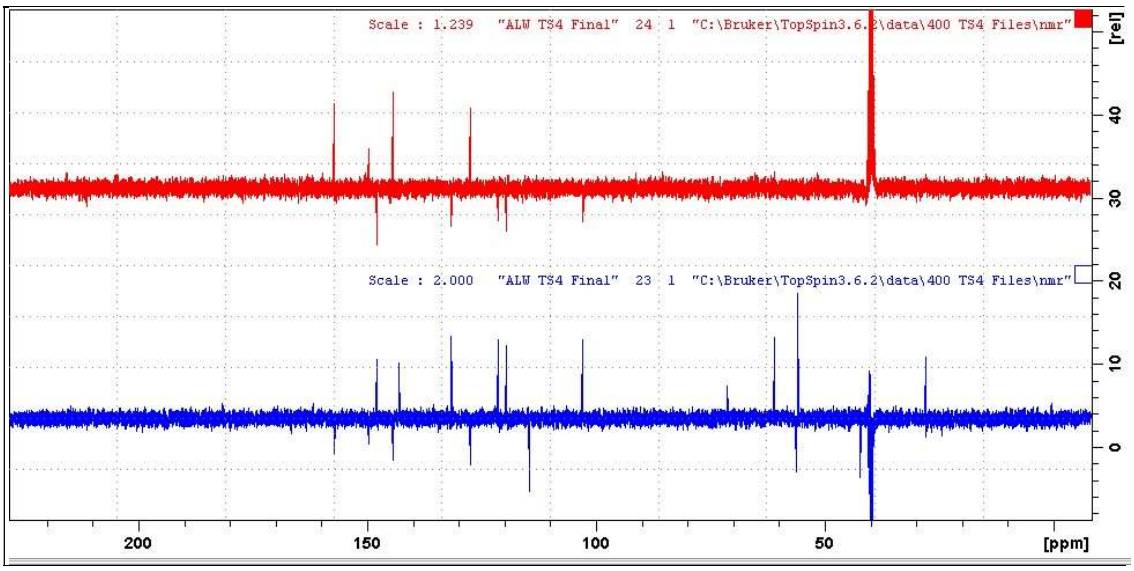
D1 = 1.5 sec (**awapt**) or 4 sec (**awaptq**) spectra or other time of your choice.
D21 = 1 msec or other time of your choice (0.5–2 msec).

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).



Lower: ATP spectrum of quinine in D_6-DMSO.
Upper: ATPQ (quaternary carbon only) 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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