

Supporting Information

Novel synthesis of fused spiro piperidone-cyclopropanes from cyclopropyl amides and electron-deficient alkene

Xiao-Dan Han¹, Gao-Liang Peng², Hui-Bin Wang¹, Lei Wu¹, Jian-ping Fu¹, Zhong-Sheng Tang¹,
Ju-Wu Hu^{1*}, Wei Xiong^{1*}

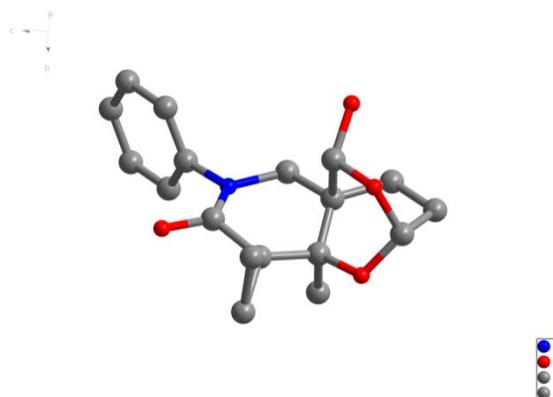
1) Department of Applied Chemistry, Jiang Xi Academic of Sciences, Nanchang, 330096, China

2) Jiangxi university of Applied Science, Nanchang, 330100, China

email: hjw19771985@163.com or xiongwei19821985@sina.com

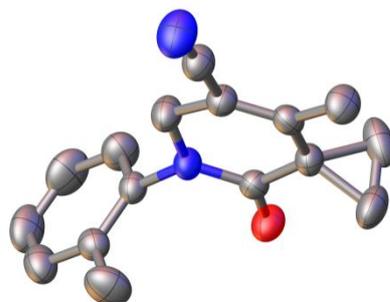
I. Crystal data and ORTEP drawing of compound 3a and 5d

Crystal data for **3a**: C₁₈H₂₁NO₄, colorless crystal, *M* = 315.36, orthorhombic, Pnma, *a* = 15.2387(4) Å, *b* = 10.3818(4) Å, *c* = 9.6545(3) Å, $\alpha = 90.0^\circ$, $\beta = 90.0^\circ$, $\gamma = 90.0^\circ$, *V* = 1527.39(9) Å³, *Z* = 4, *T* = 150K, *F*₀₀₀ = 672, *R*₁ = 0.0692(1315), *wR*₂ = 0.1658(1426). CCDC1948958.



Compound 3a

Crystal data for **5d**: C₁₆H₁₆N₂O, colorless crystal, *M* = 250.29, monoclinic, P21/c, *a* = 11.4303(7) Å, *b* = 23.1780(16) Å, *c* = 10.3616(8) Å, $\alpha = 90.0^\circ$, $\beta = 91.780(6)^\circ$, $\gamma = 90.0^\circ$, *V* = 2743.8(3) Å³, *Z* = 8, *T* = 293, K, *F*₀₀₀ = 1056, *R*₁ = 0.0728(3987), *wR*₂ = 0.2095(4833). CCDC 1948938.



Compound 5d

II. Copies of ¹H NMR and ¹³C NMR spectra of compounds 3, 4 and 5

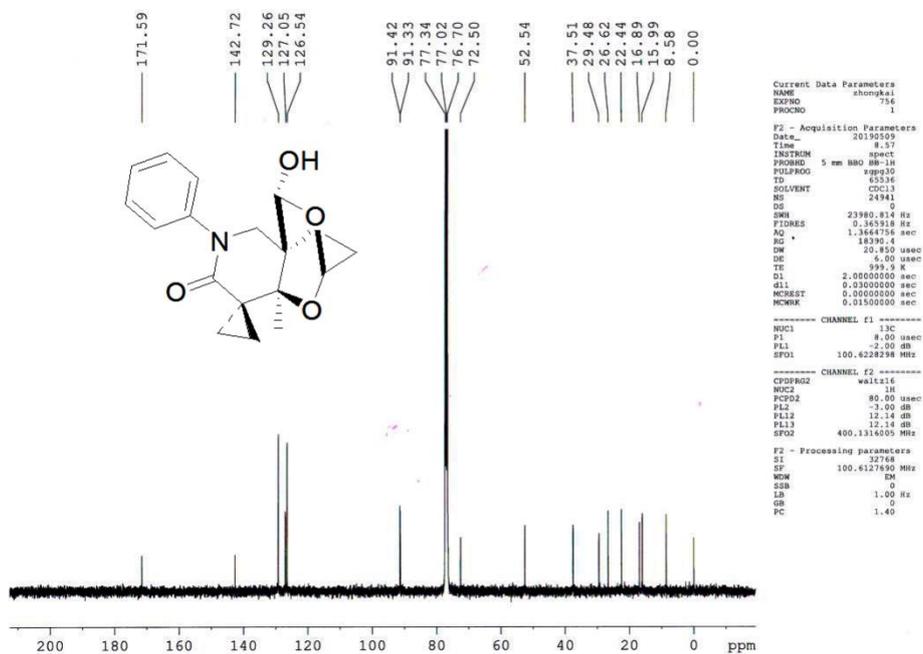
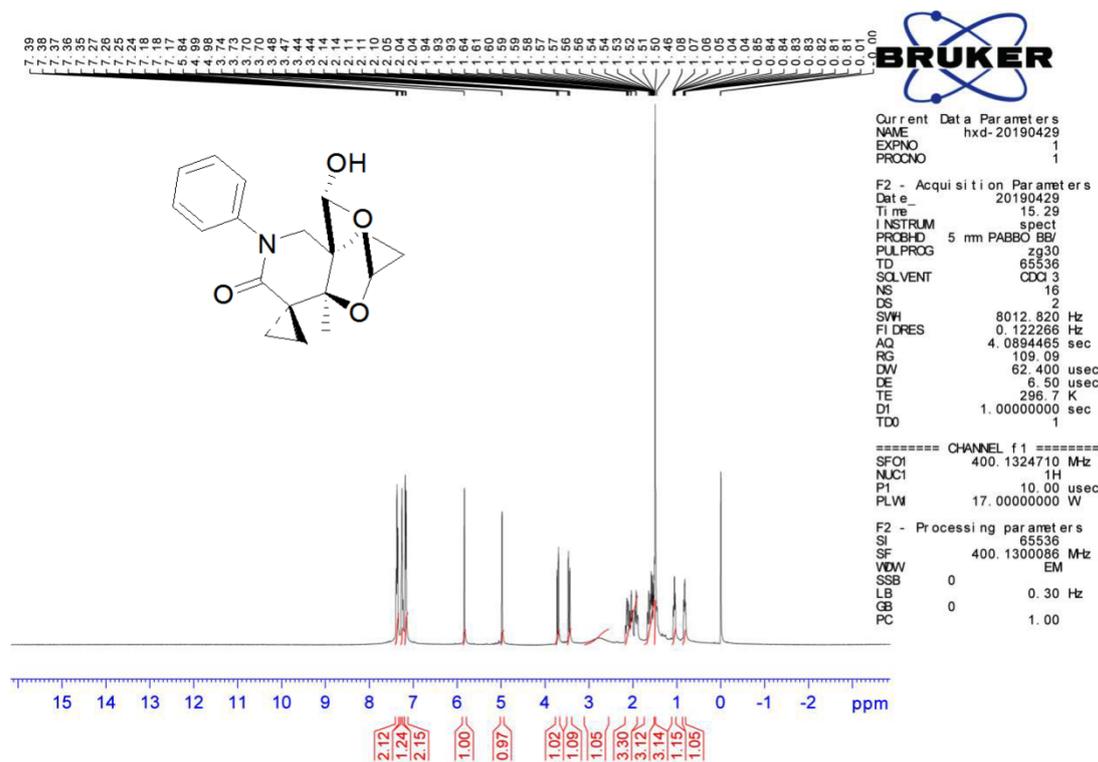


Figure 1. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 3a.

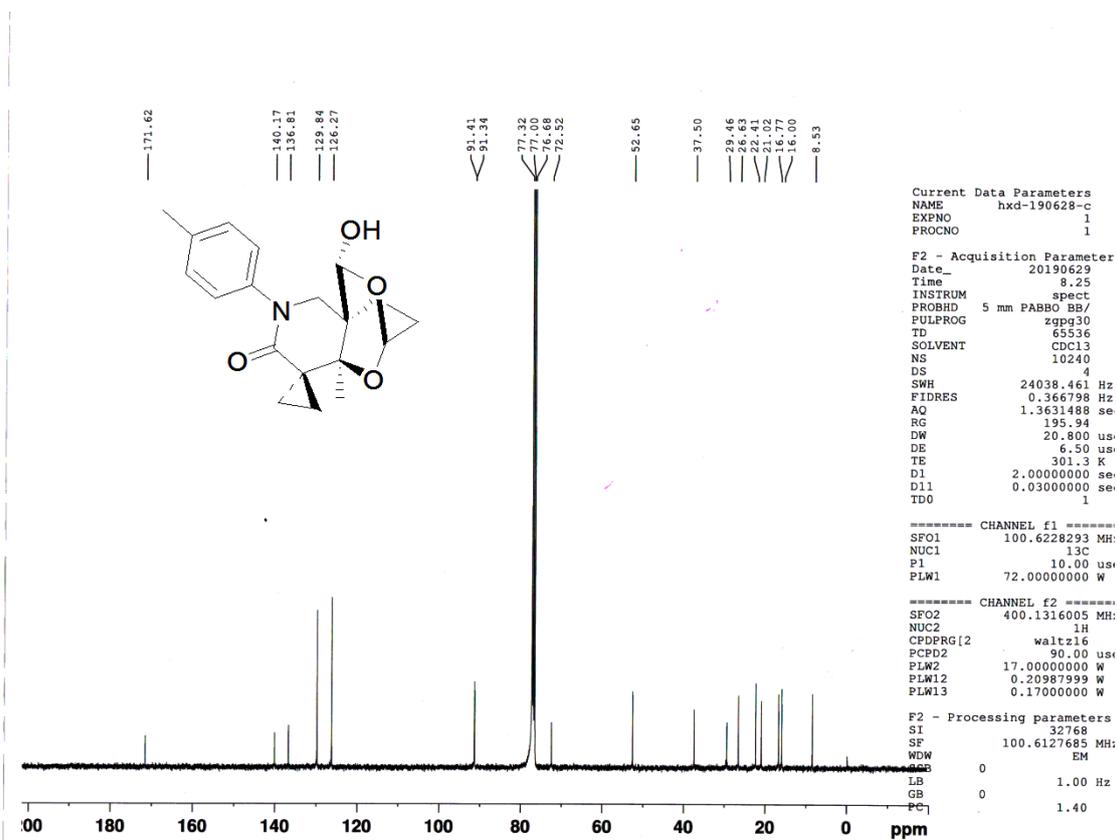
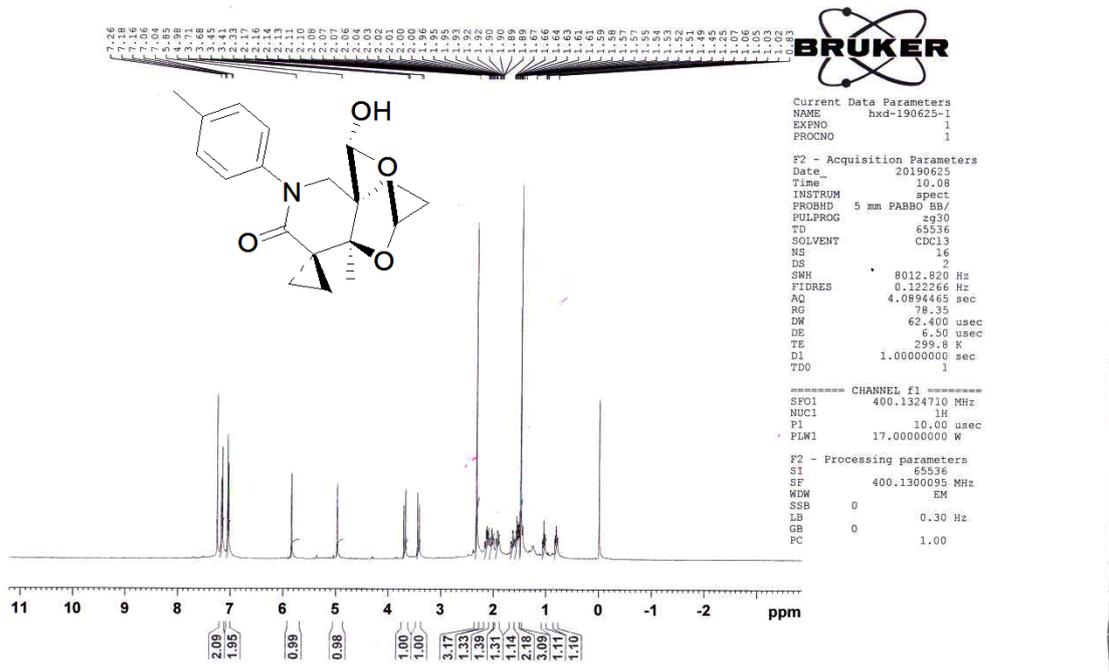


Figure 2. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 3b.

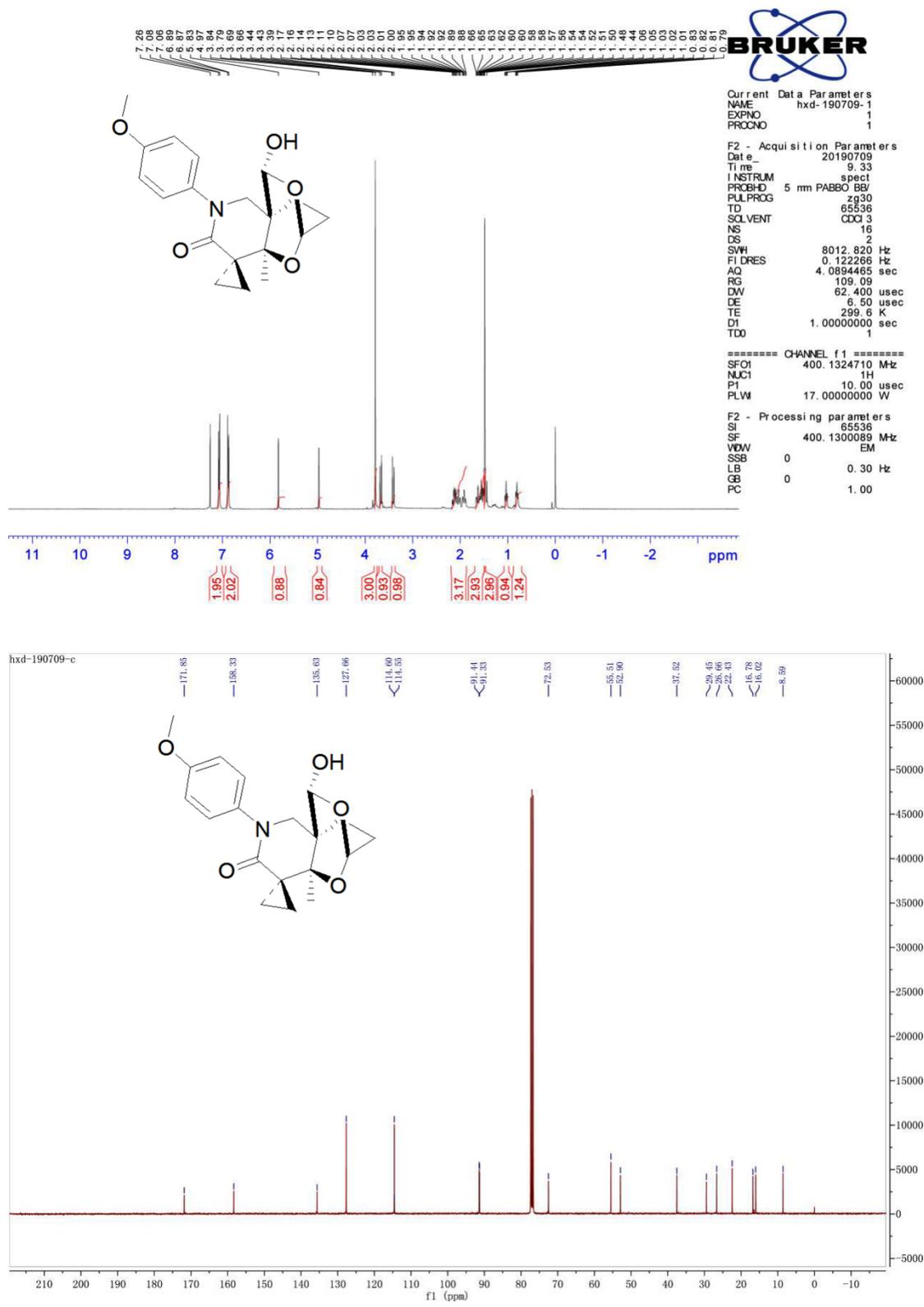


Figure 3. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 3c.

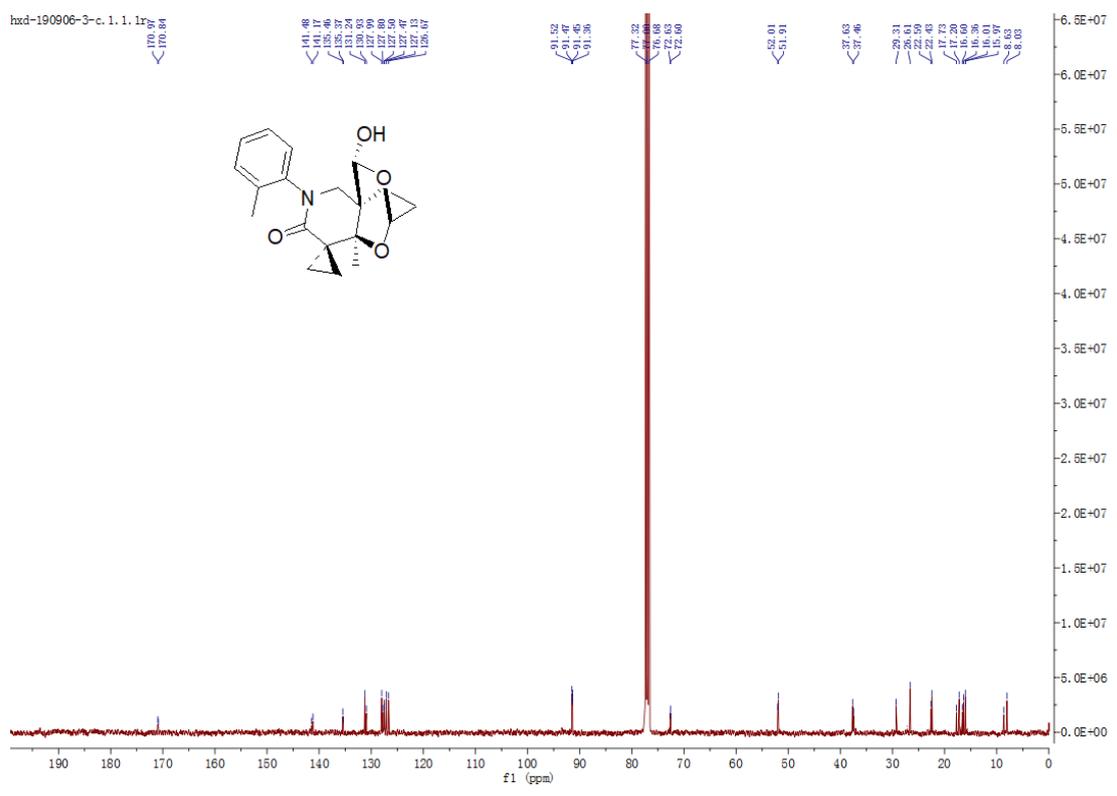
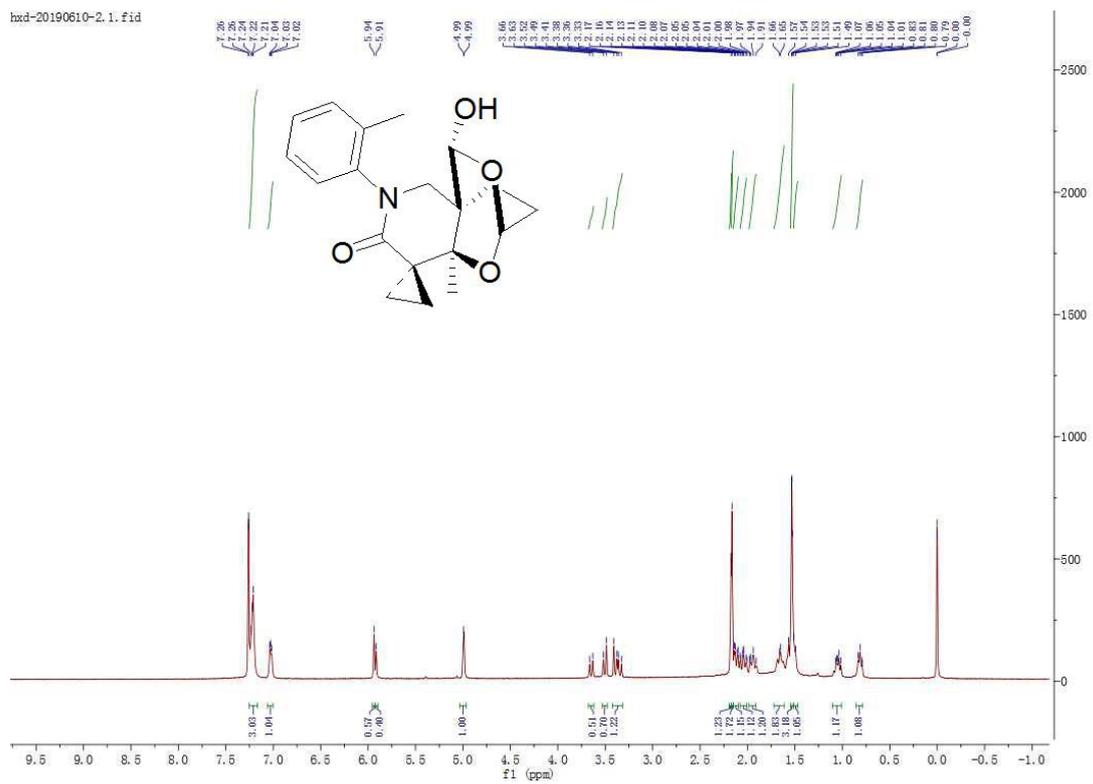


Figure 4. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **3d**.

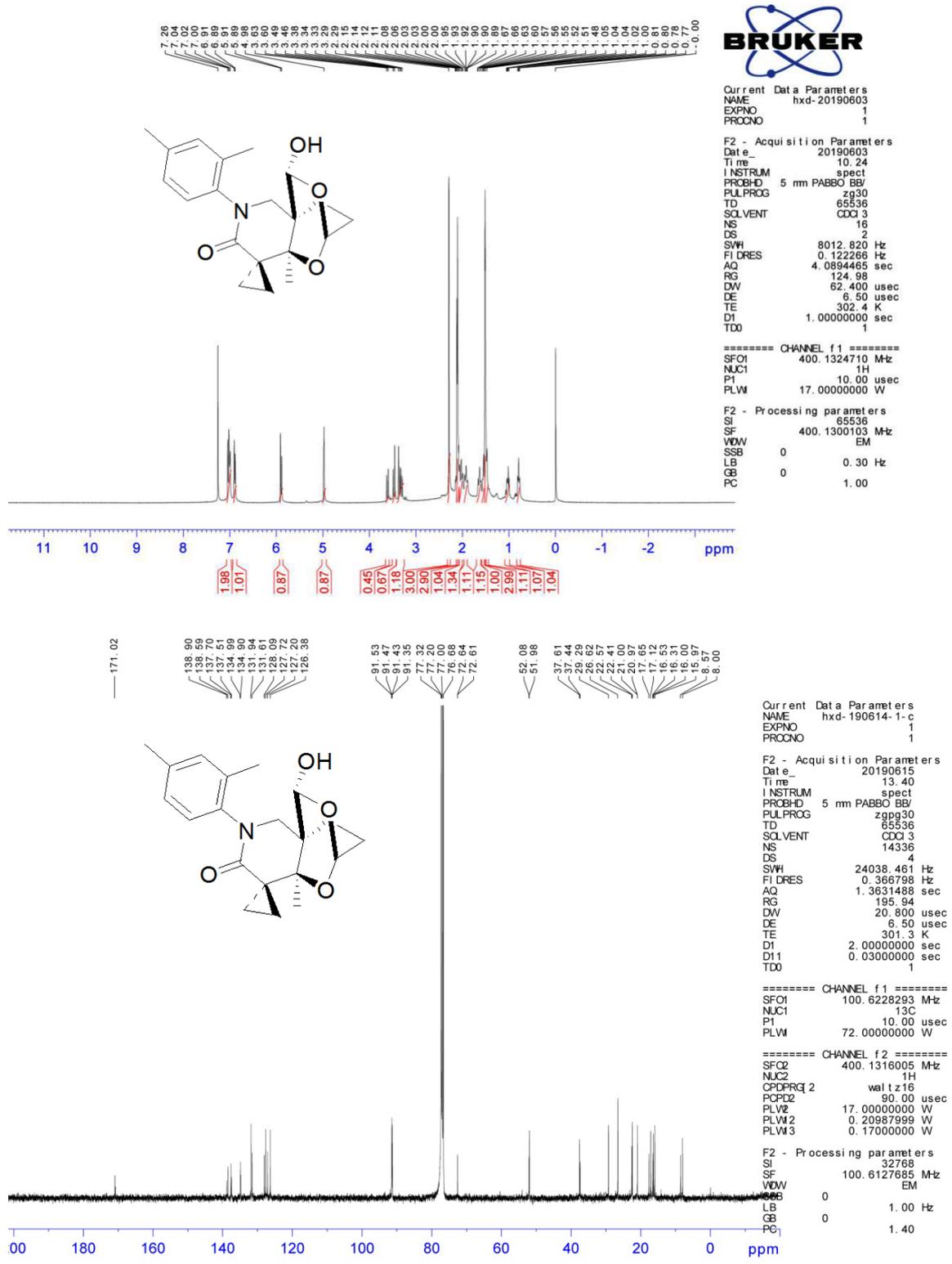


Figure 5. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 3e.

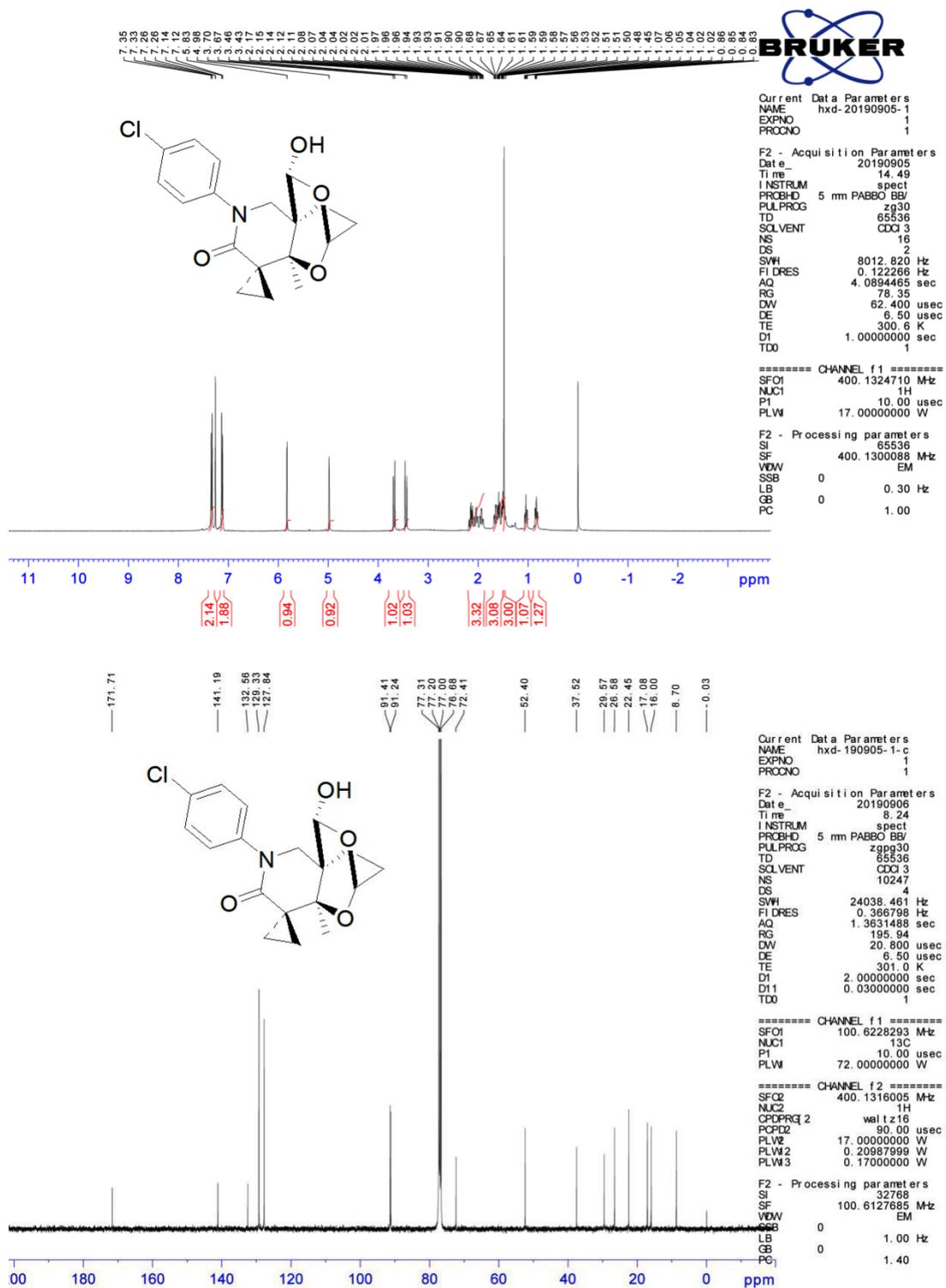


Figure 6. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 3f.

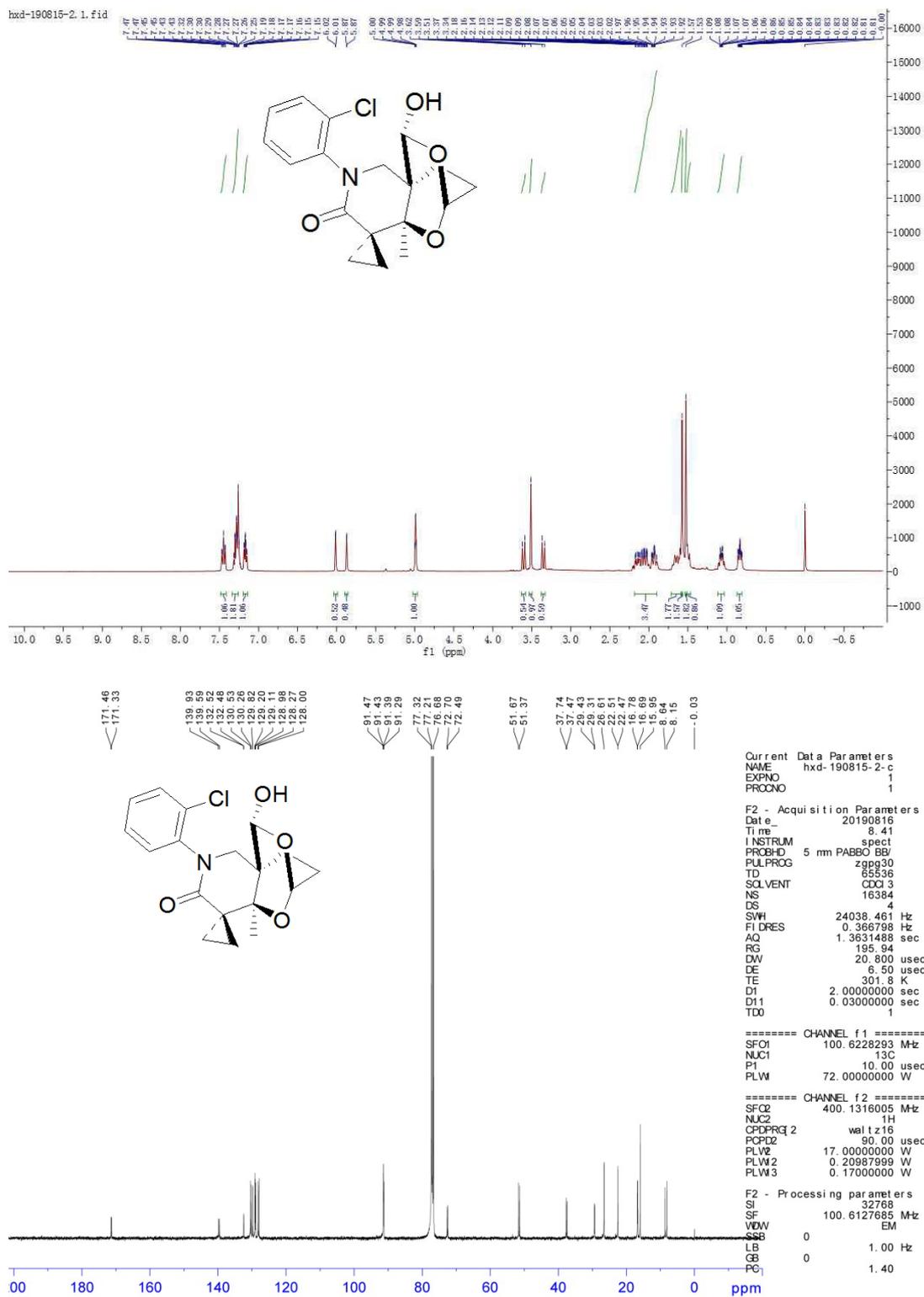


Figure 7. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound **3g**.

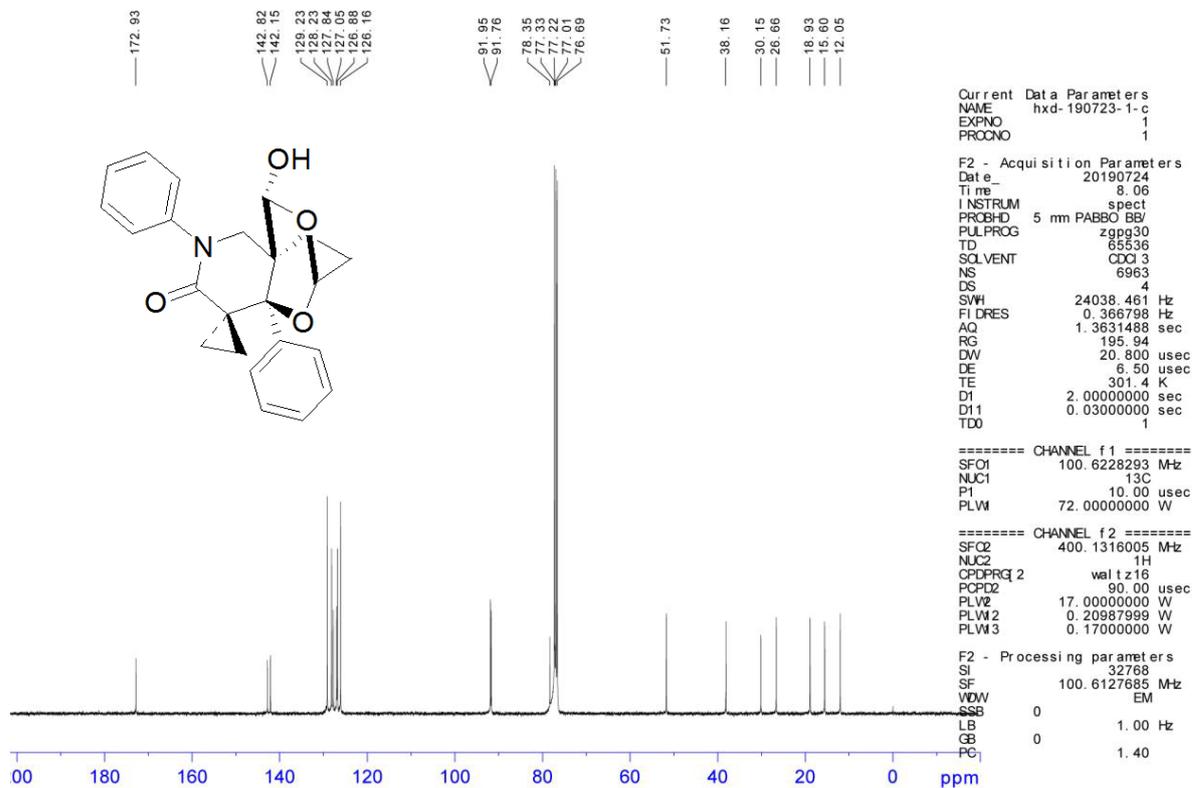
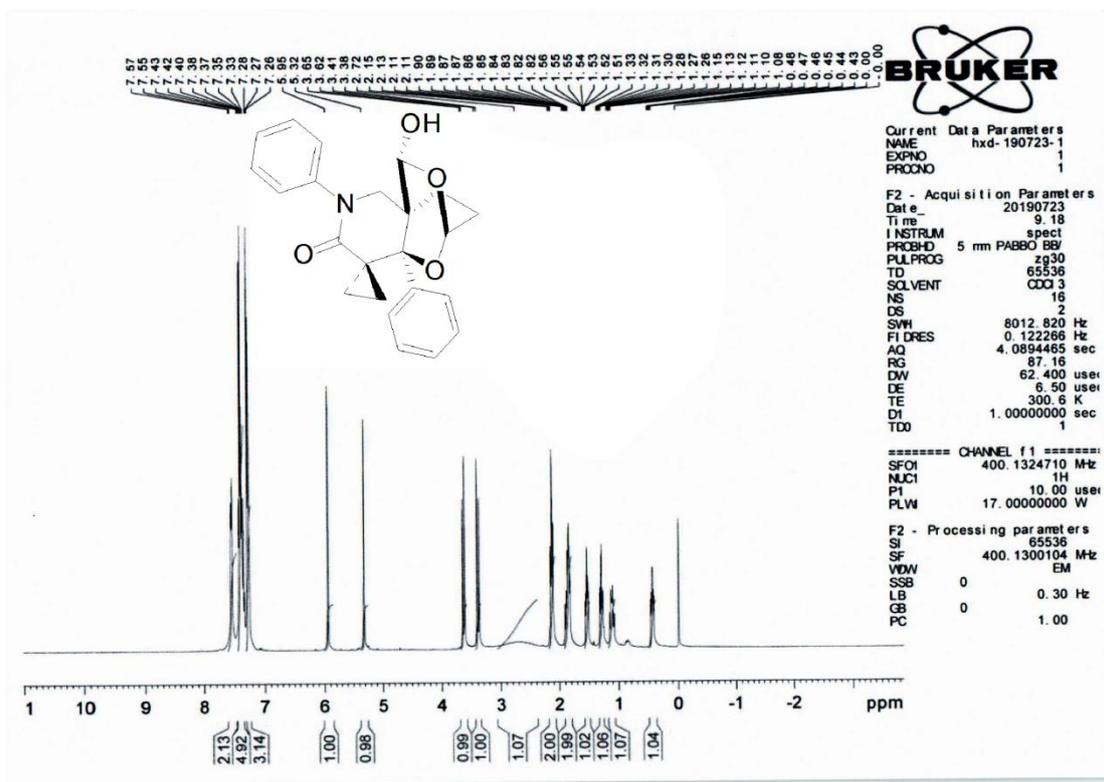


Figure 8. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 3h.

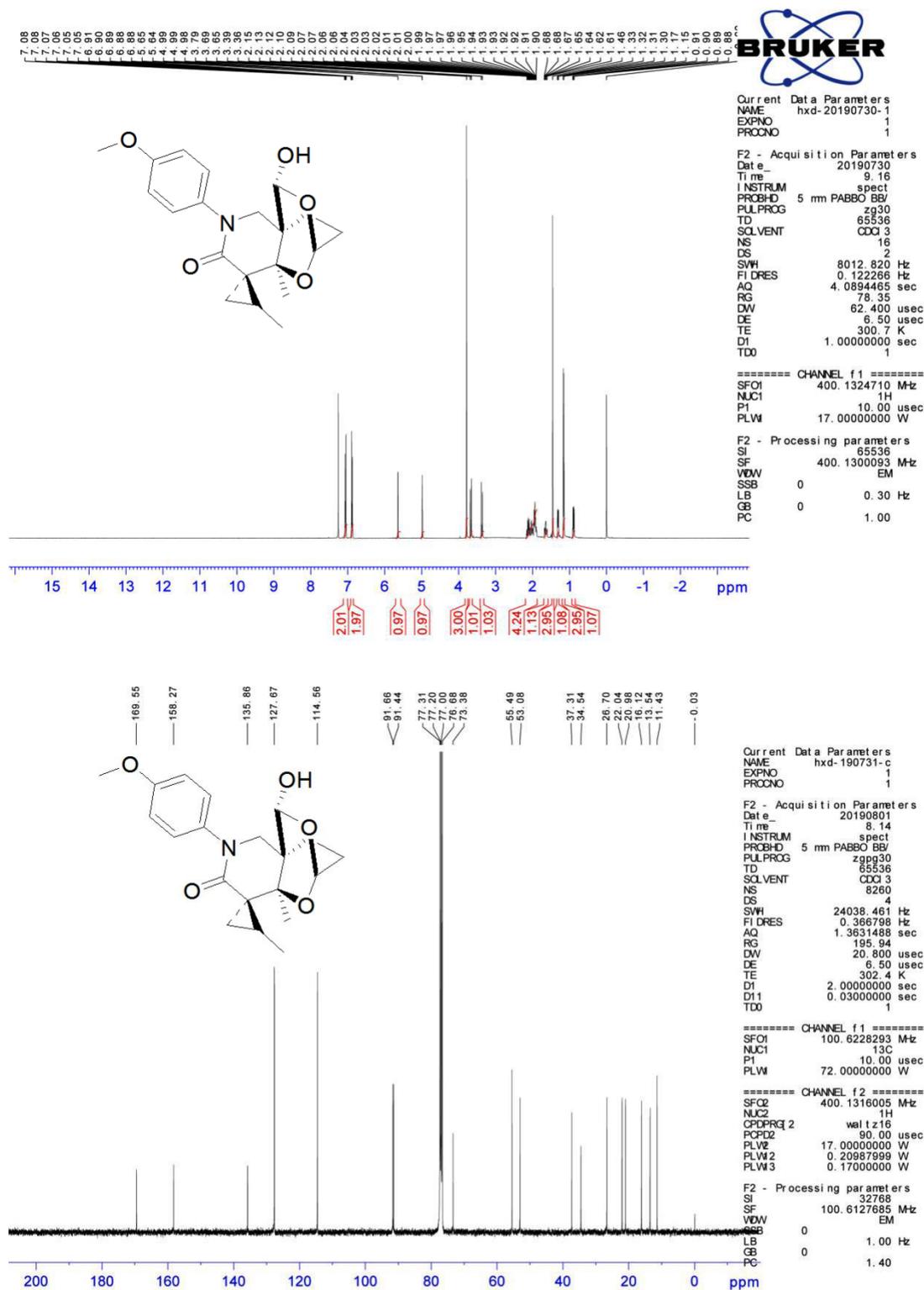


Figure 9. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound **3j**.

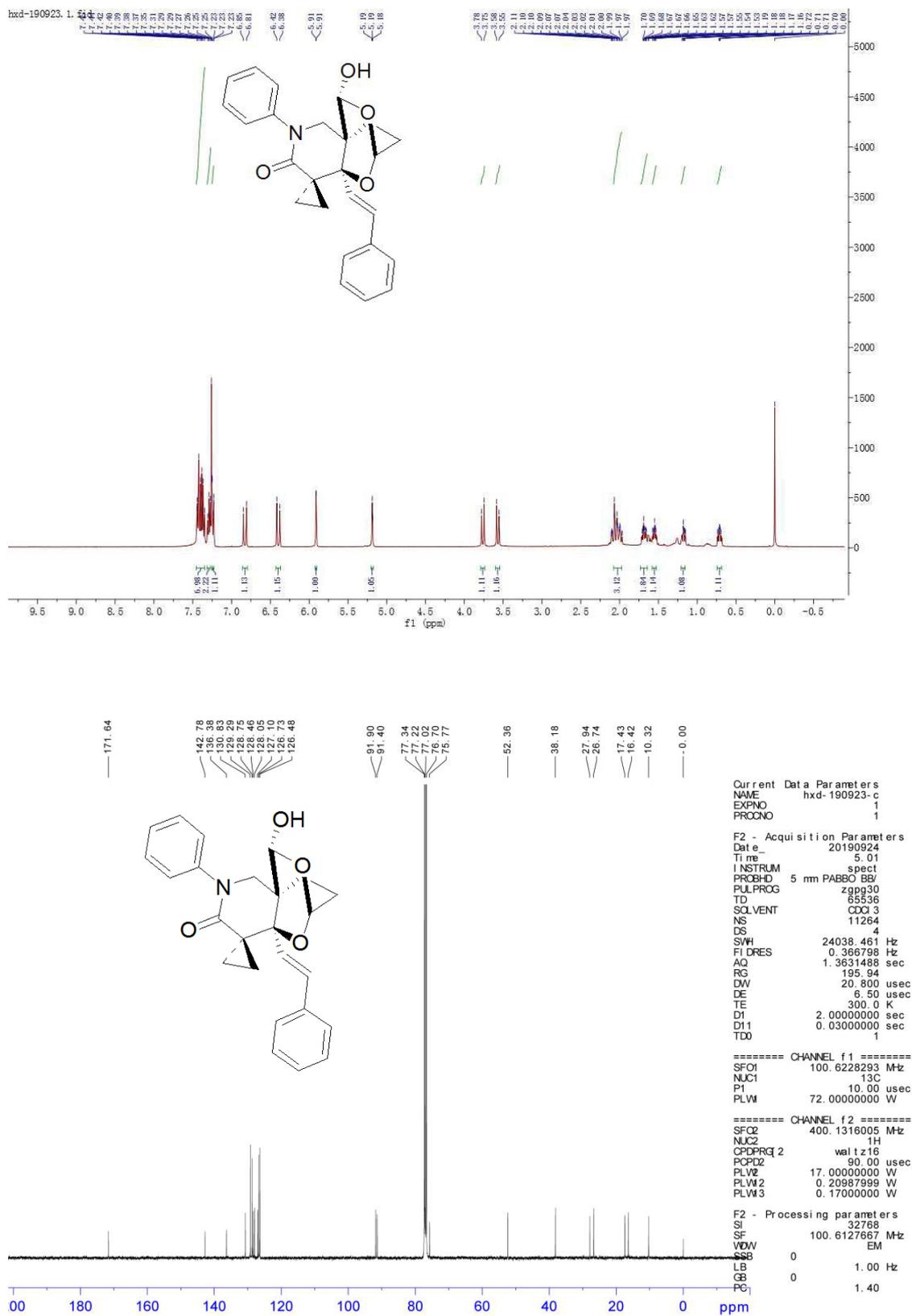


Figure 10. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound **3k**.

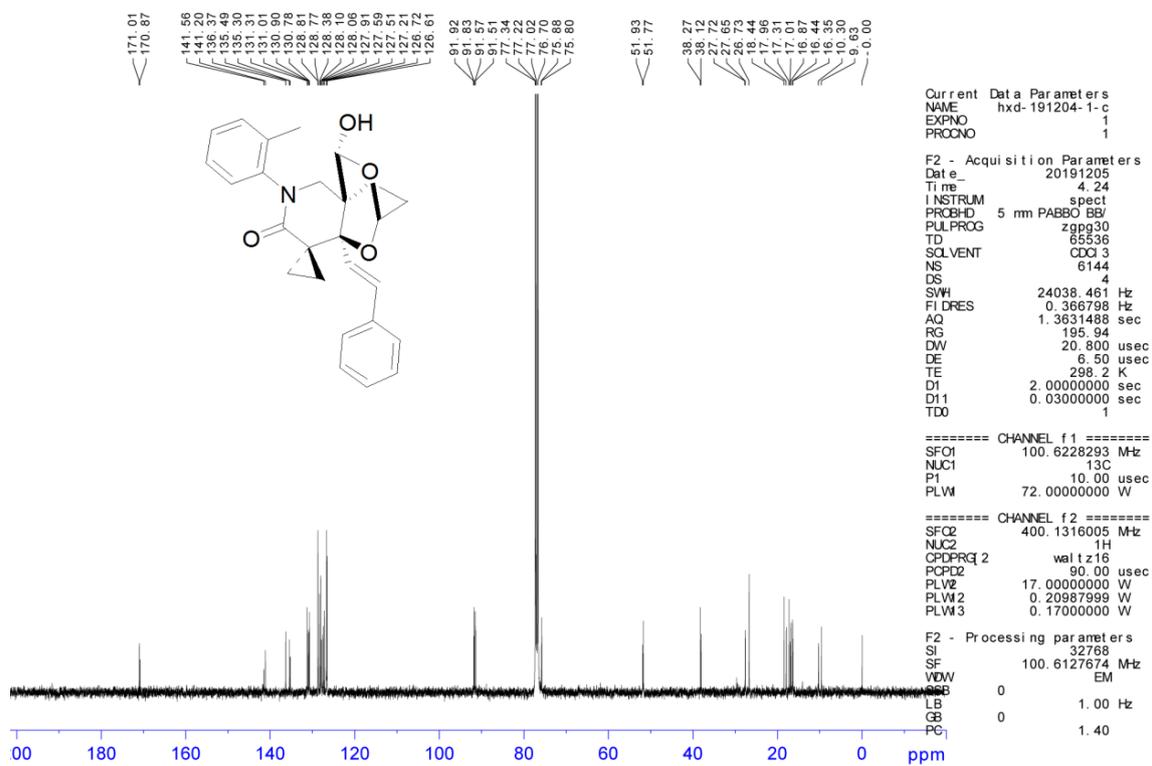
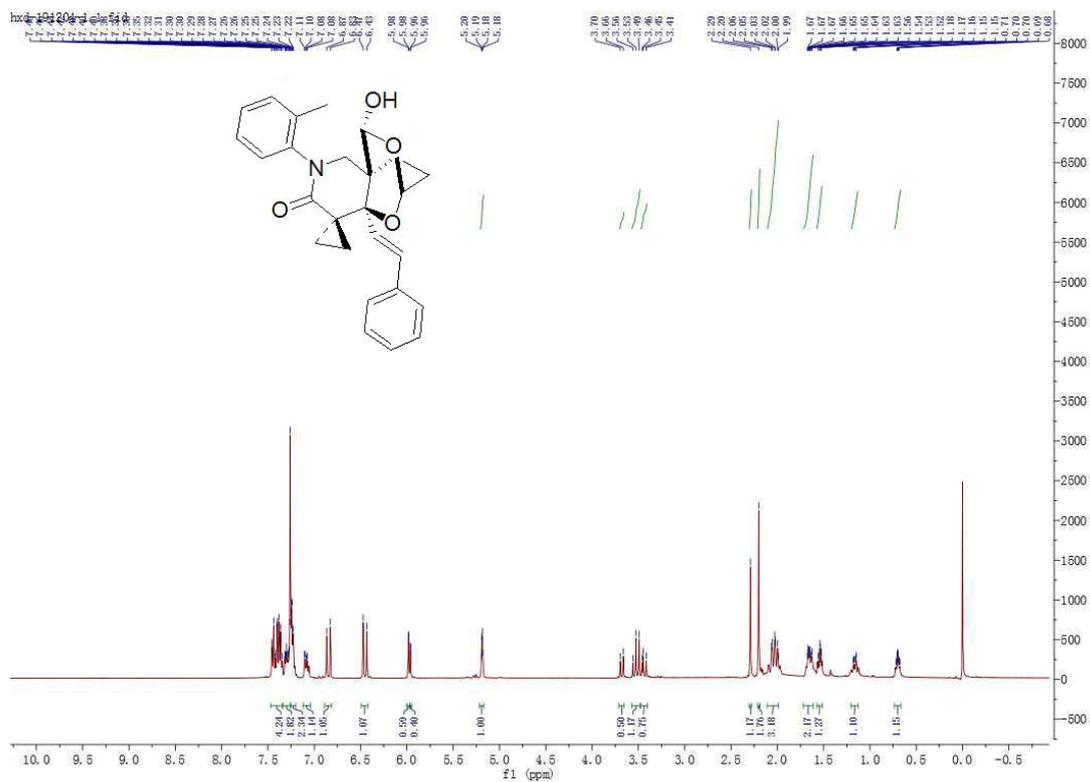


Figure 11. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 31.

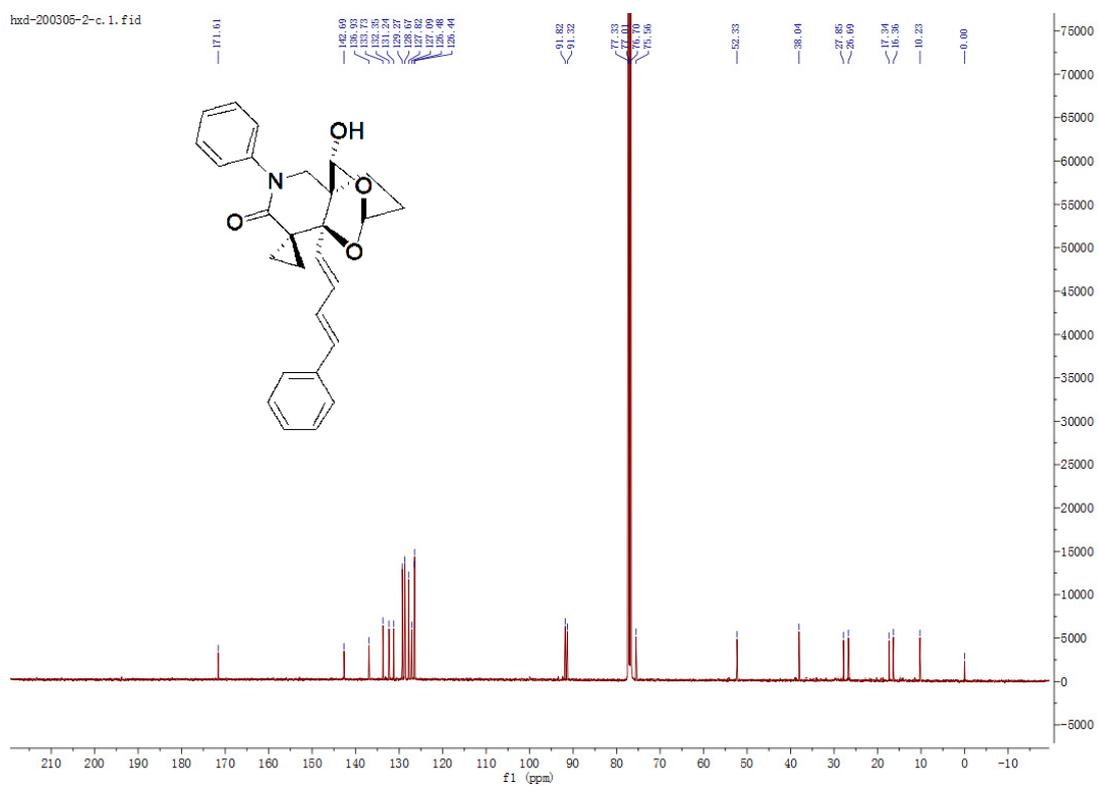
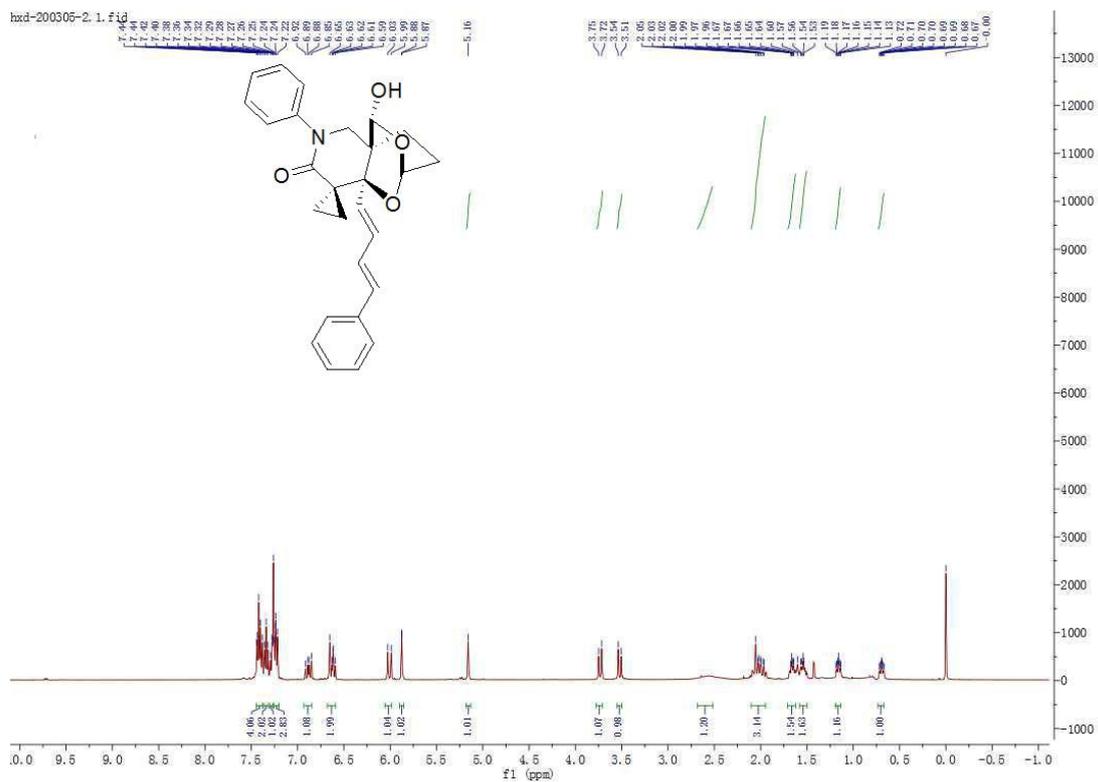


Figure 12. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **3m**.

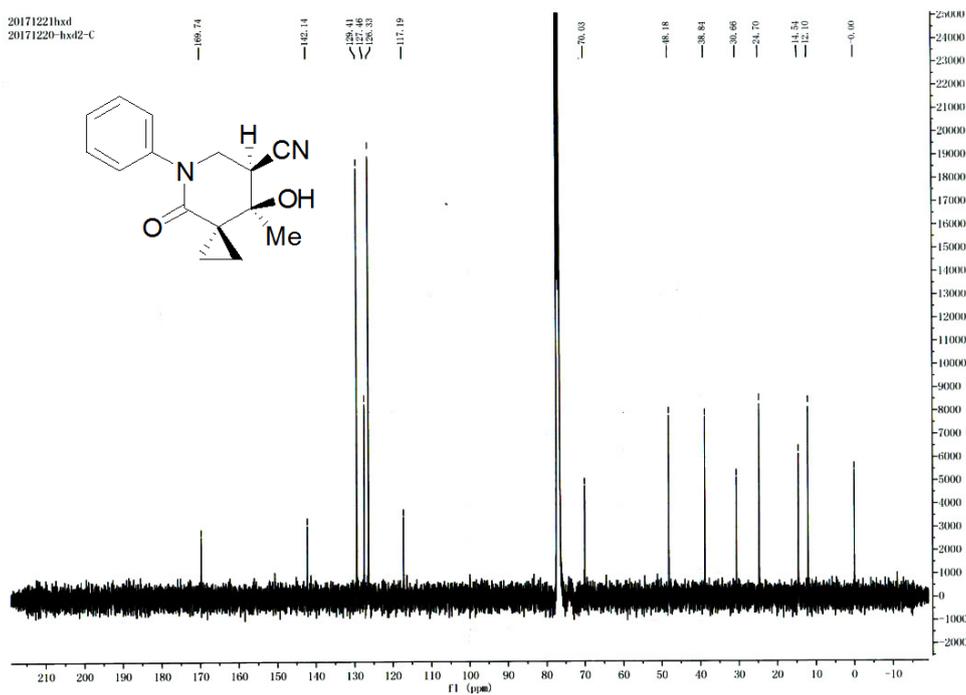
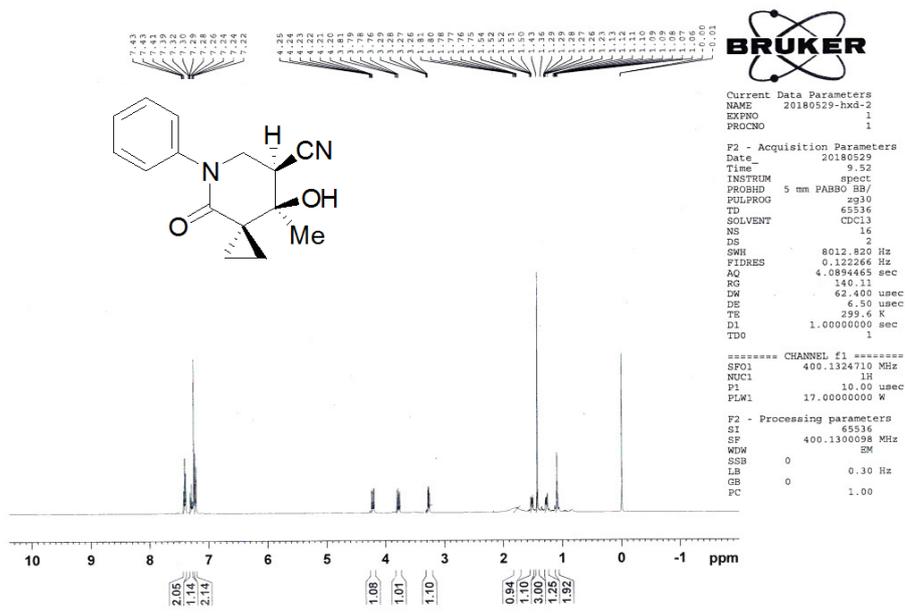
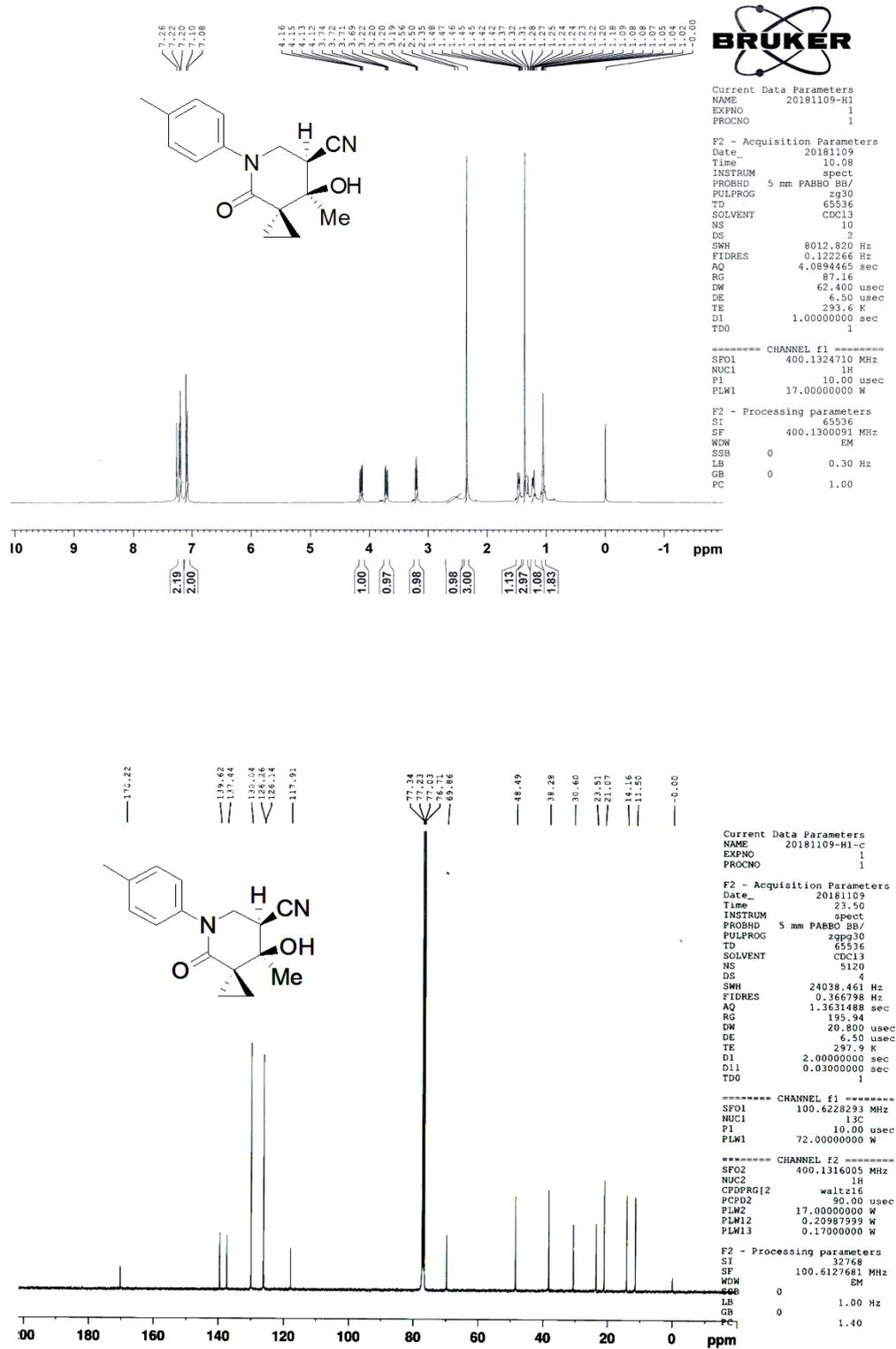


Figure 14. ^1H - (upper) and ^{13}C -NMR (lower) spectra of compound **4a**.



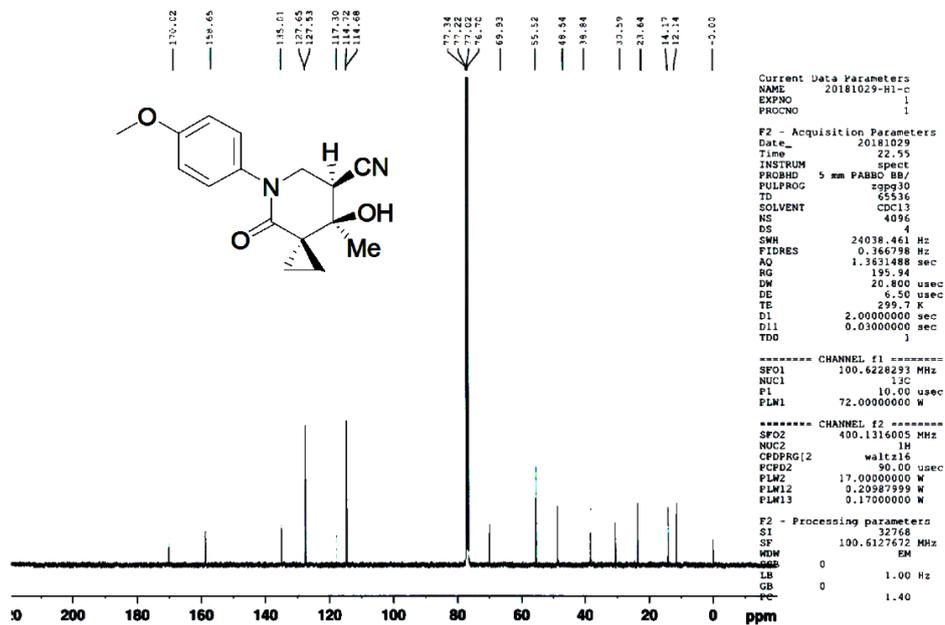
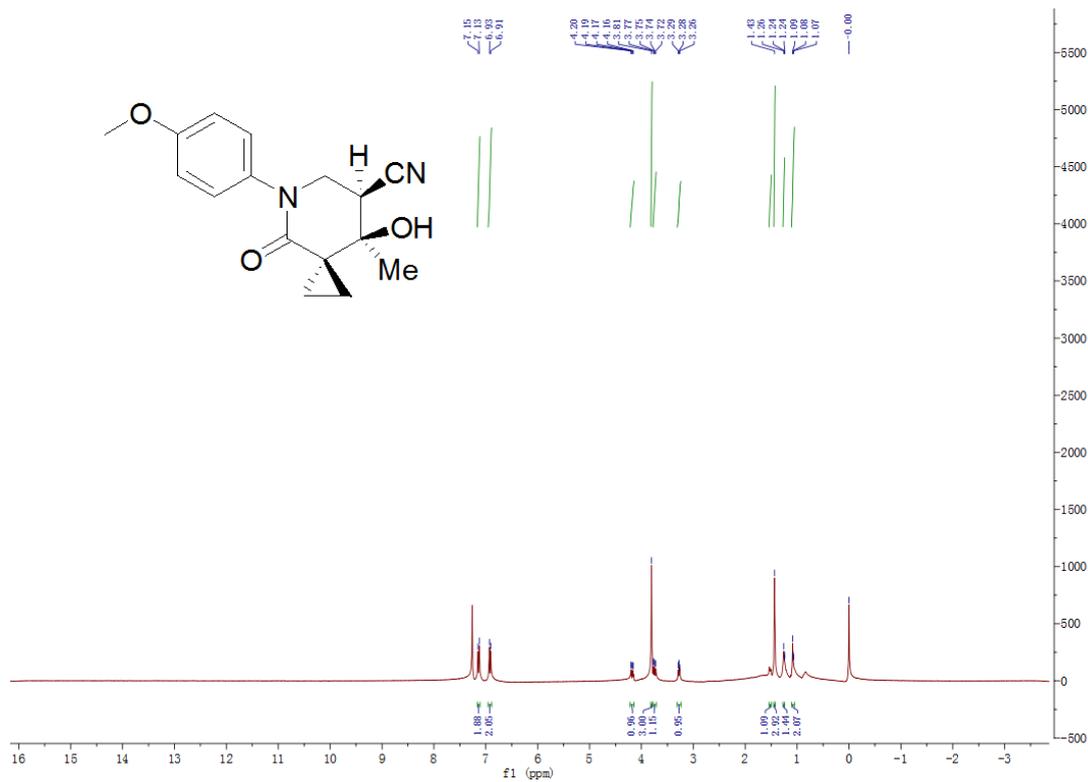


Figure 16. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 4c.

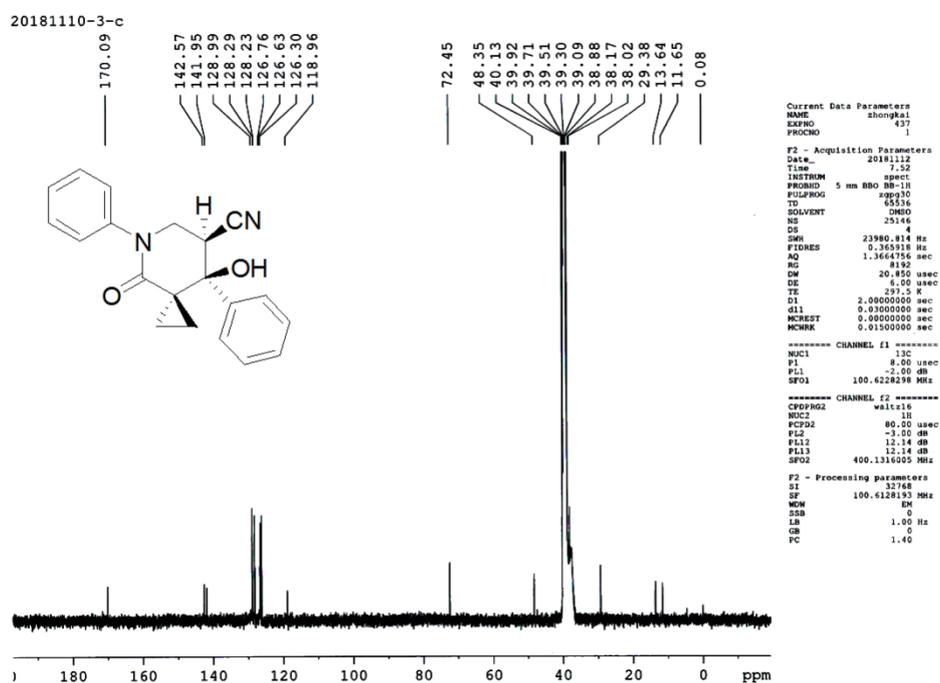
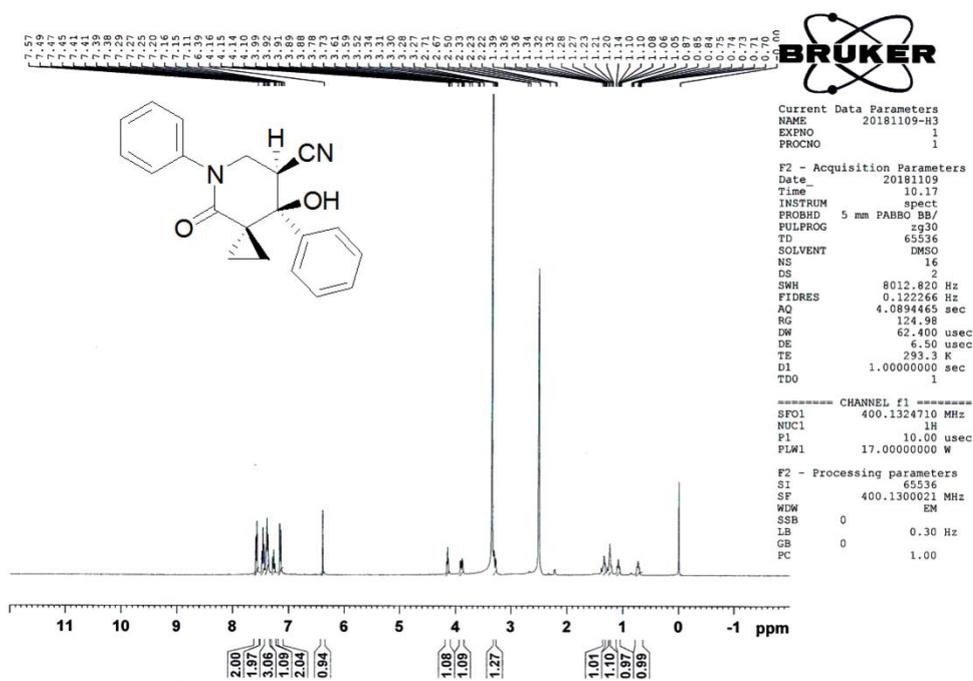


Figure 17. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 4d.

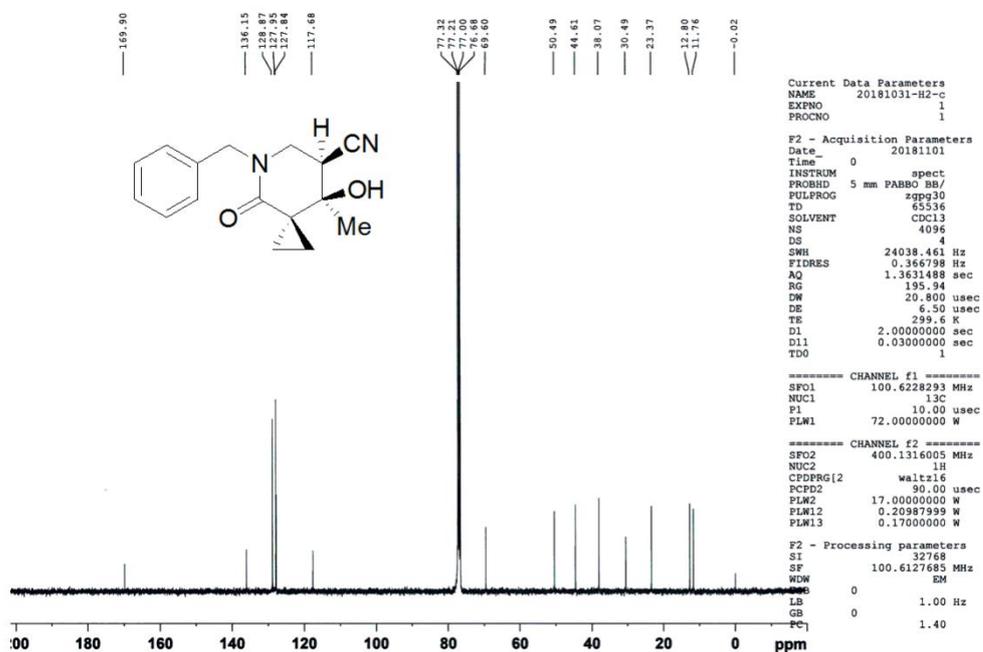
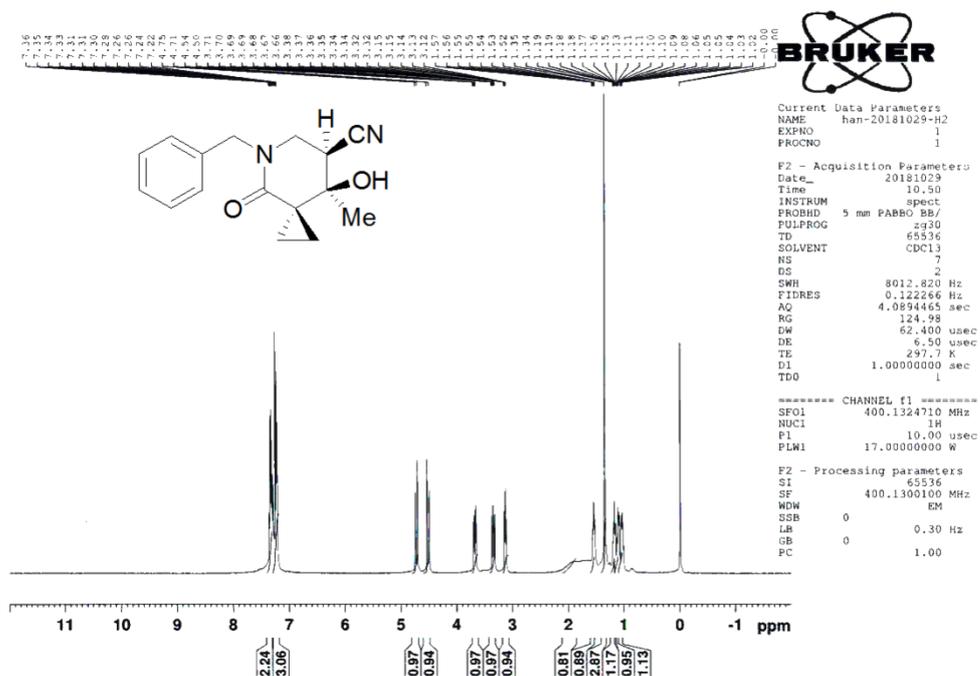


Figure 18. ^1H -NMR (upper) and ^{13}C -NMR (lower) spectra of compound **4e**.

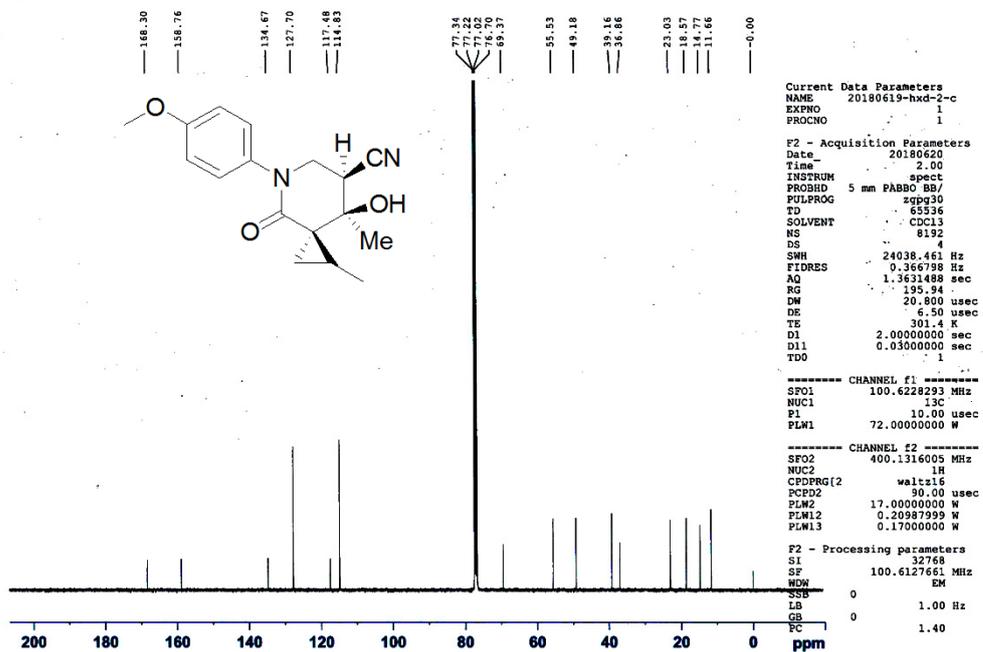
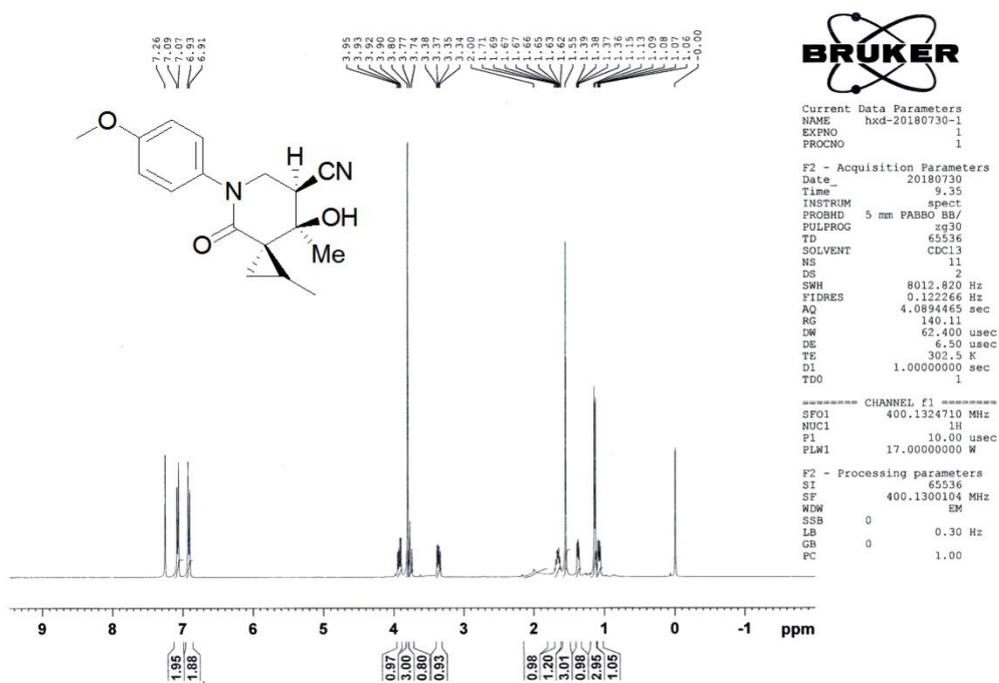


Figure 19. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 4f.

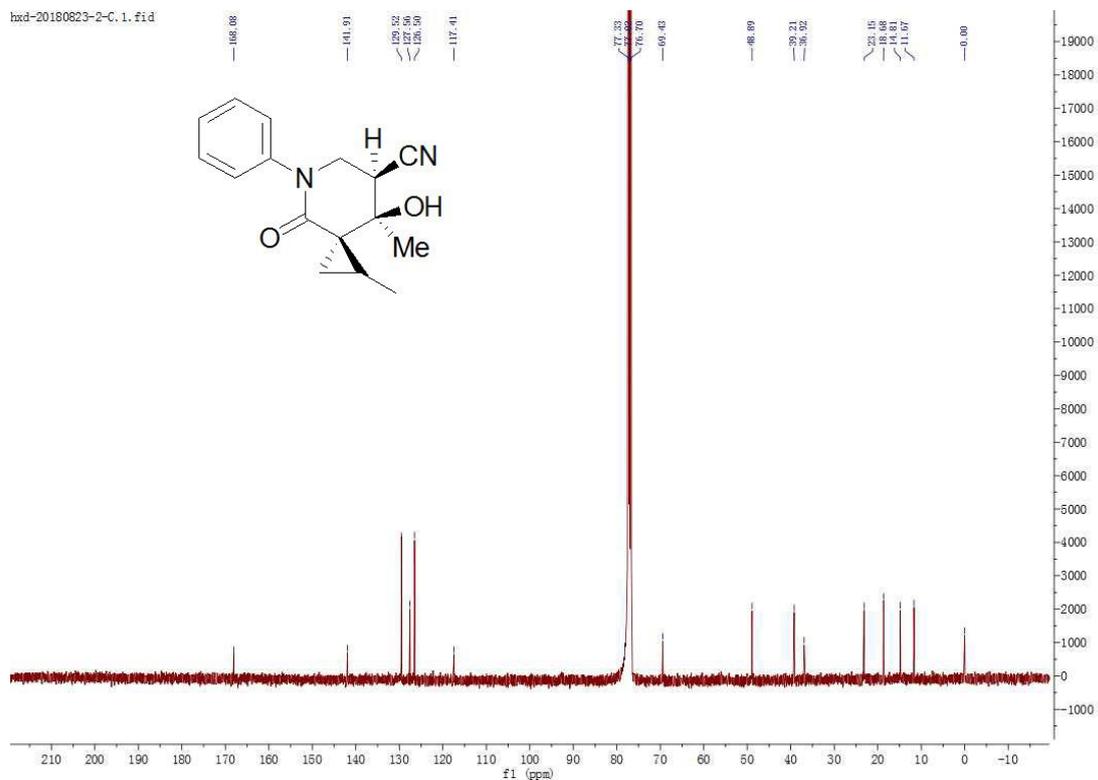
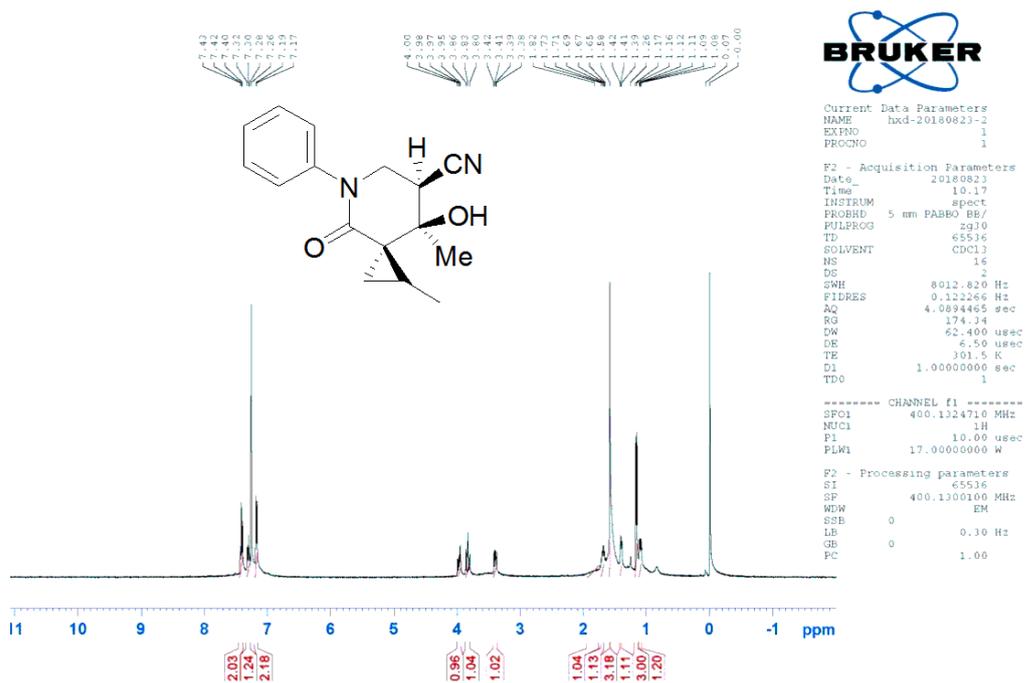


Figure 20. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 4g.

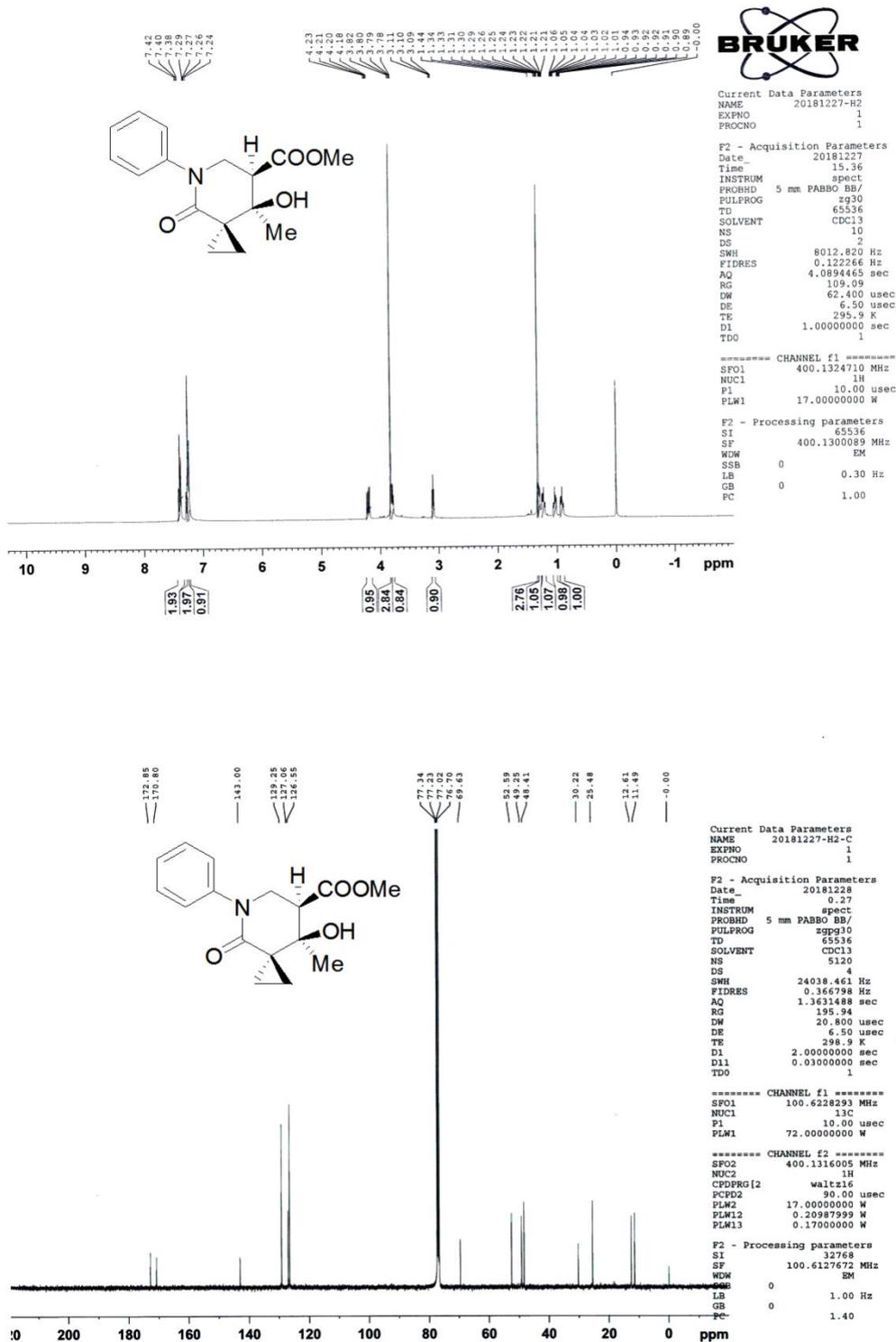


Figure 21. ^1H -NMR (upper) and ^{13}C -NMR (lower) spectra of compound 4h.

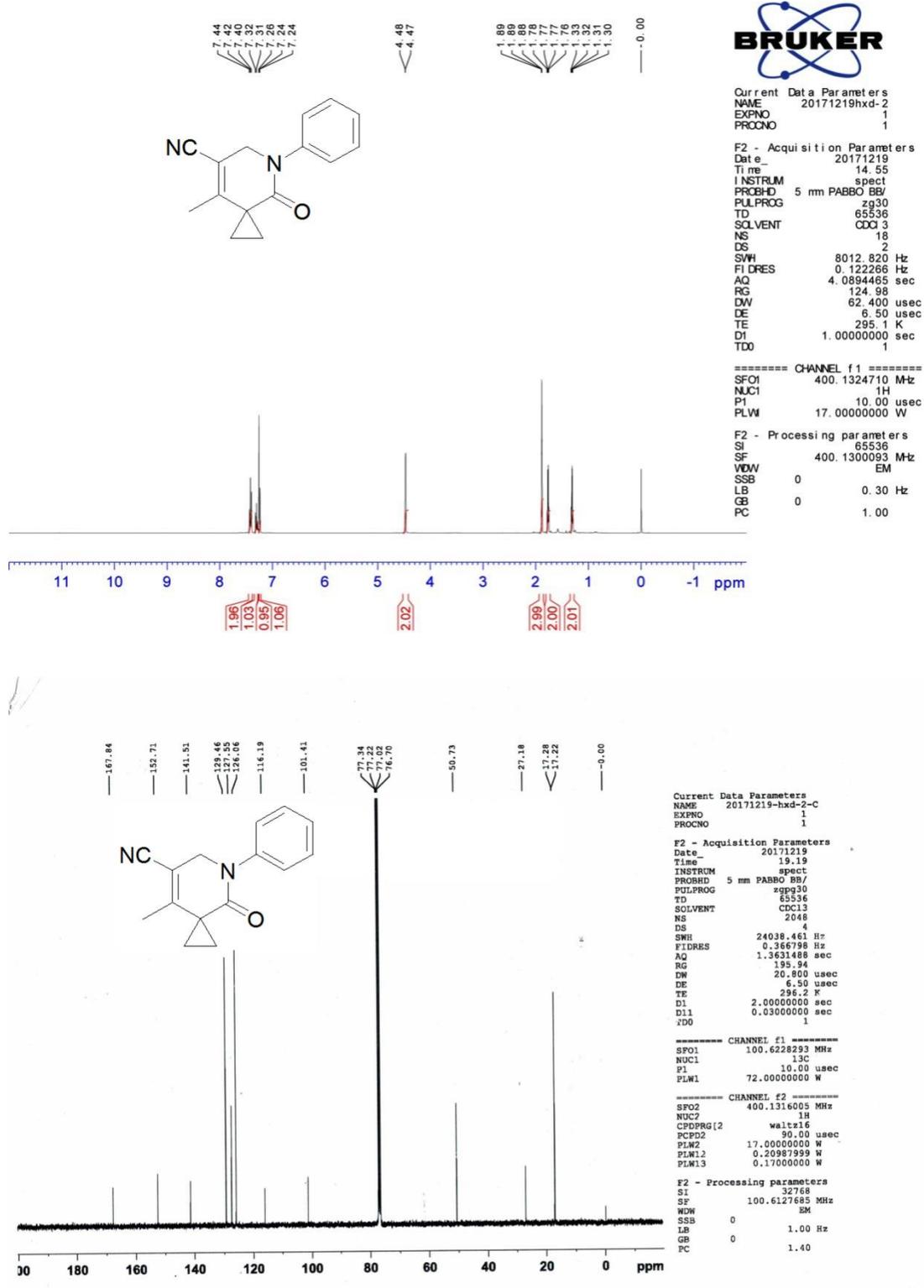


Figure 22. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 5a.



Figure 23. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 5b.

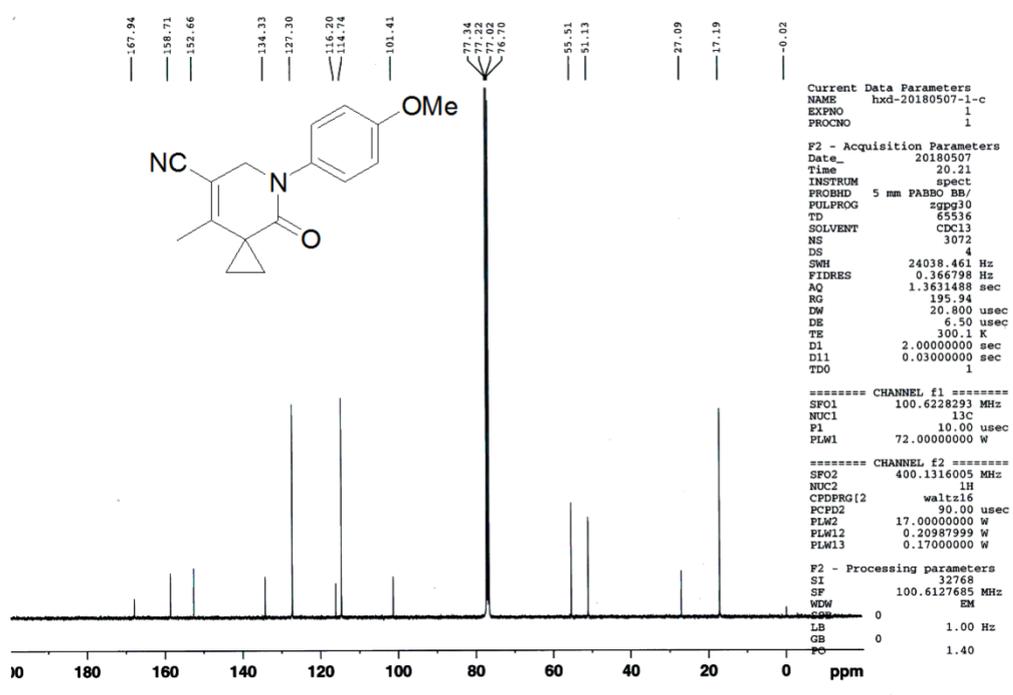
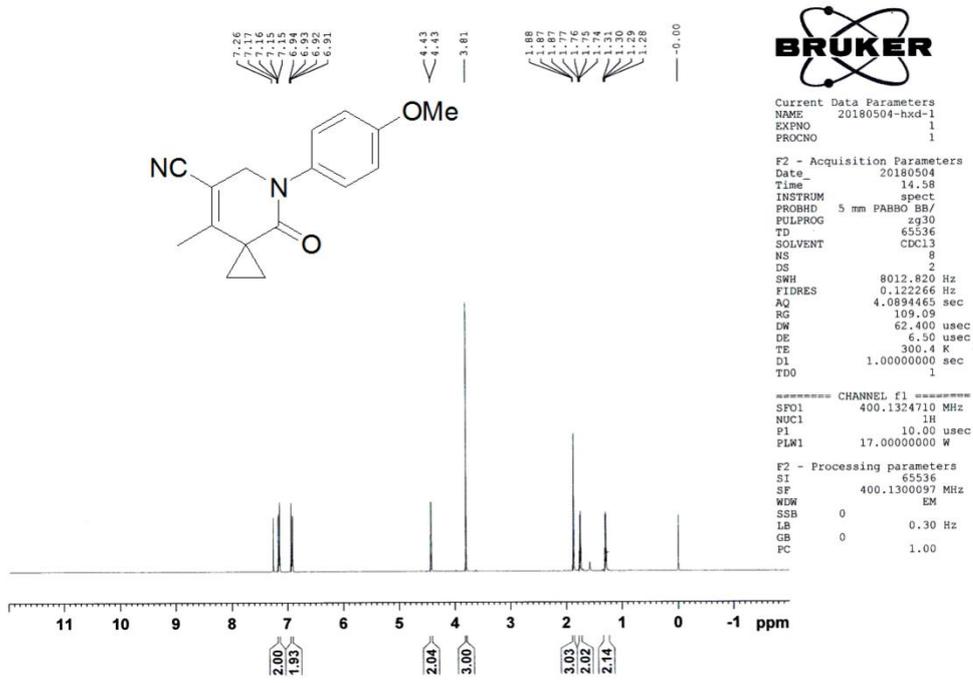
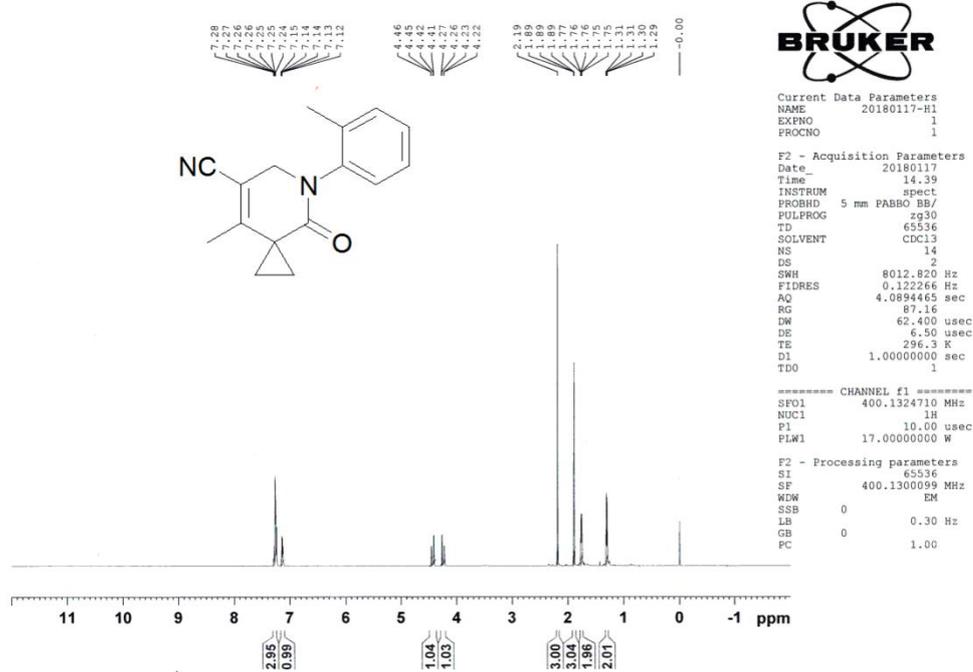


Figure 24. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 5c.

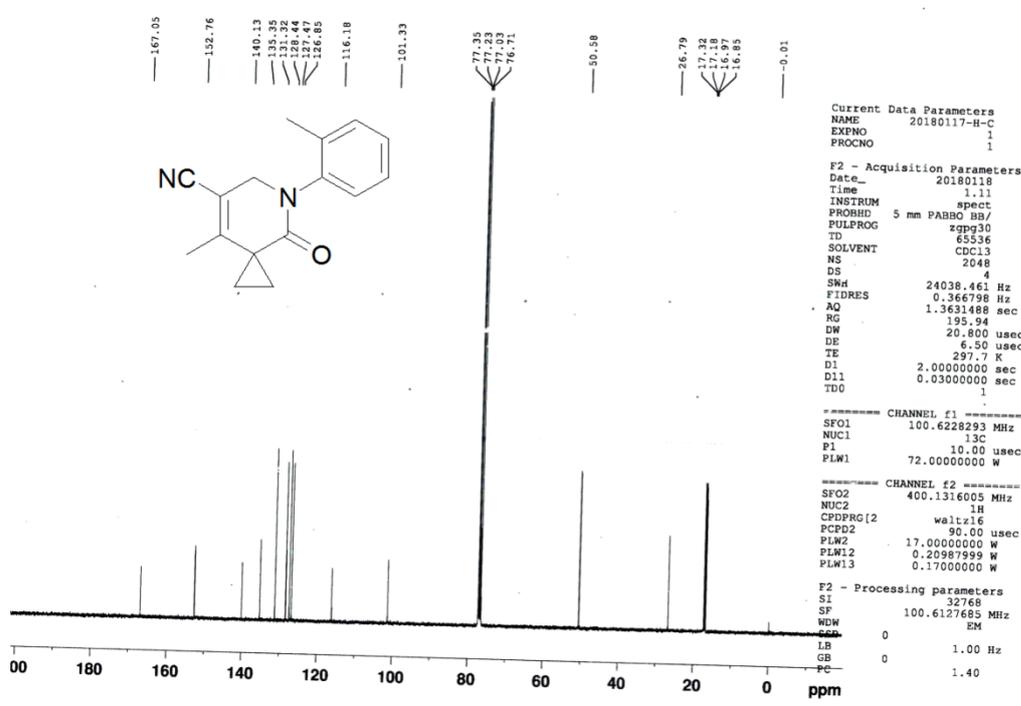


Current Data Parameters
 NAME 20180117-H1
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180117
 Time 14.39
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 14
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.089465 sec
 RG 87.16
 DW 62.400 usec
 DE 6.50 usec
 TE 296.3 K
 D1 1.0000000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 400.1324710 MHz
 NUC1 1H
 P1 10.00 usec
 PLW1 17.0000000 W

F2 - Processing parameters
 SI 65536
 SF 400.1300099 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



Current Data Parameters
 NAME 20180117-H-C
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20180118
 Time 1.11
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2048
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631488 sec
 RG 195.94
 DW 20.800 usec
 DE 6.50 usec
 TE 297.7 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TDO 1

===== CHANNEL f1 =====
 SFO1 100.6228293 MHz
 NUC1 13C
 P1 10.00 usec
 PLW1 72.0000000 W

===== CHANNEL f2 =====
 SFO2 400.1316005 MHz
 NUC2 1H
 CPDPRG2 waltz16
 PCPD2 90.00 usec
 PLW2 17.0000000 W
 PLW12 0.20987999 W
 PLW13 0.17000000 W

F2 - Processing parameters
 SI 32768
 SF 100.6127685 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

Figure 25. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 5d.

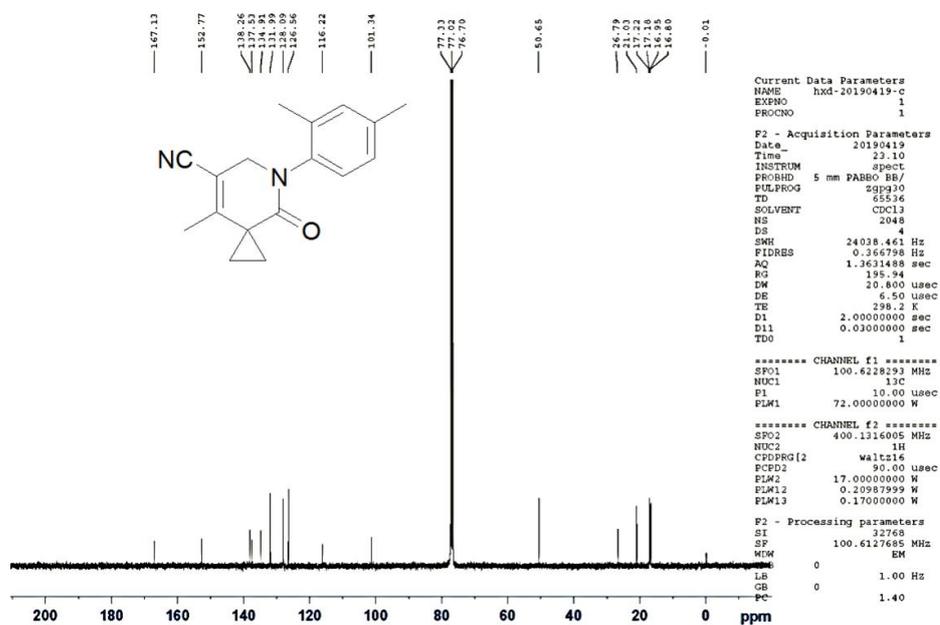
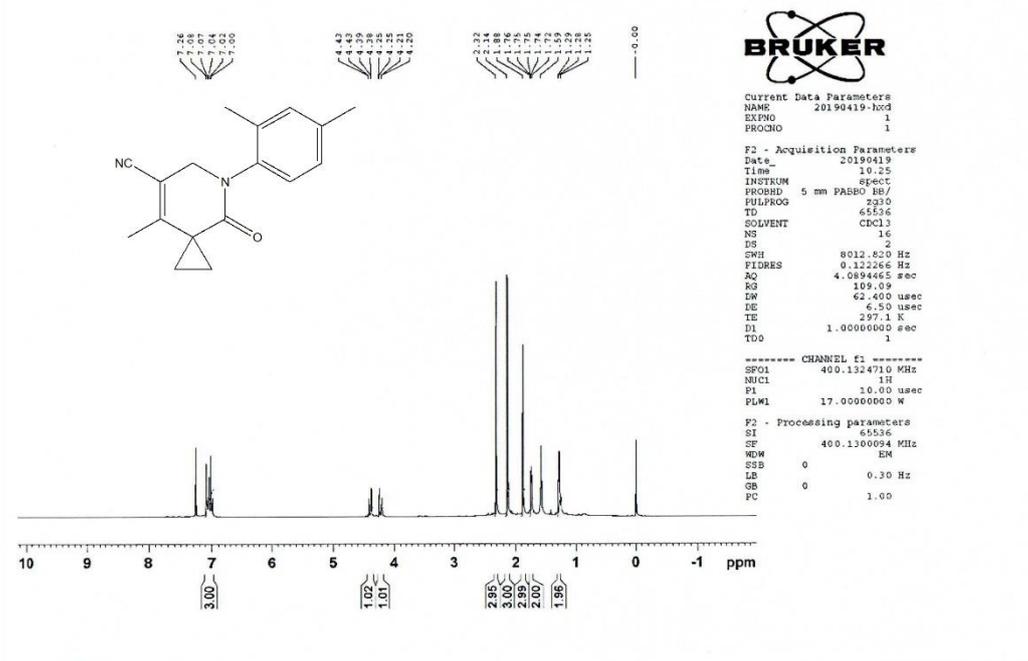


Figure 26. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 5e.

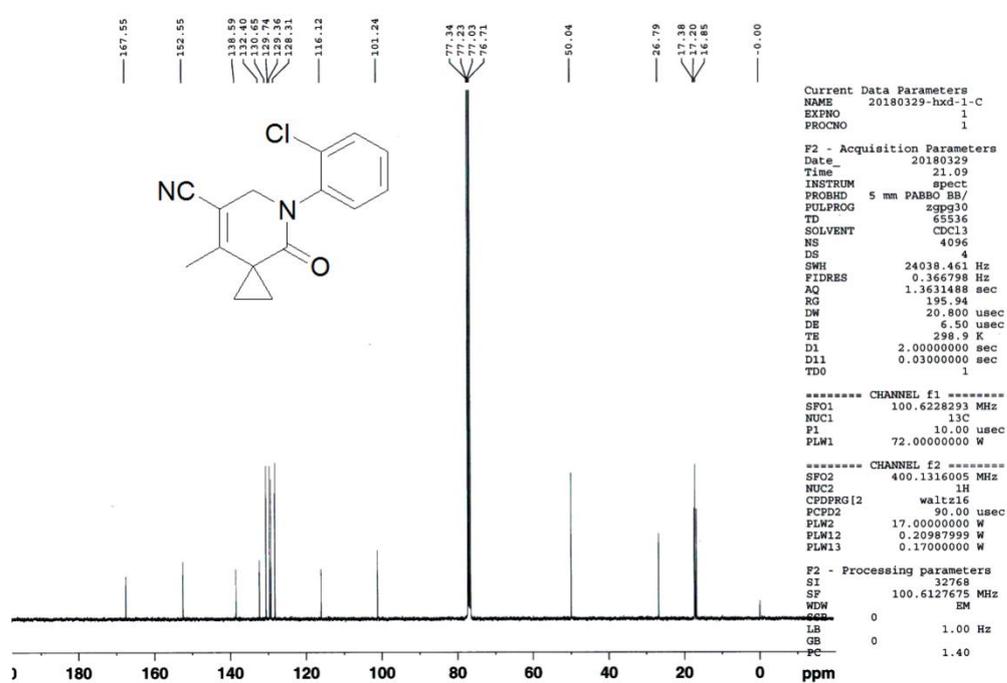
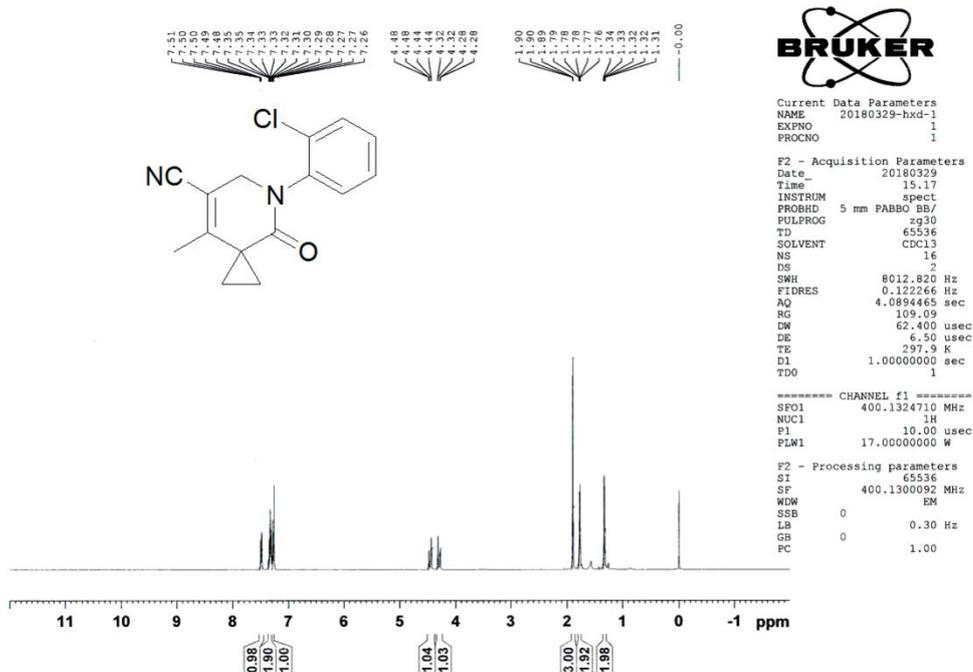


Figure 27. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound 5f.

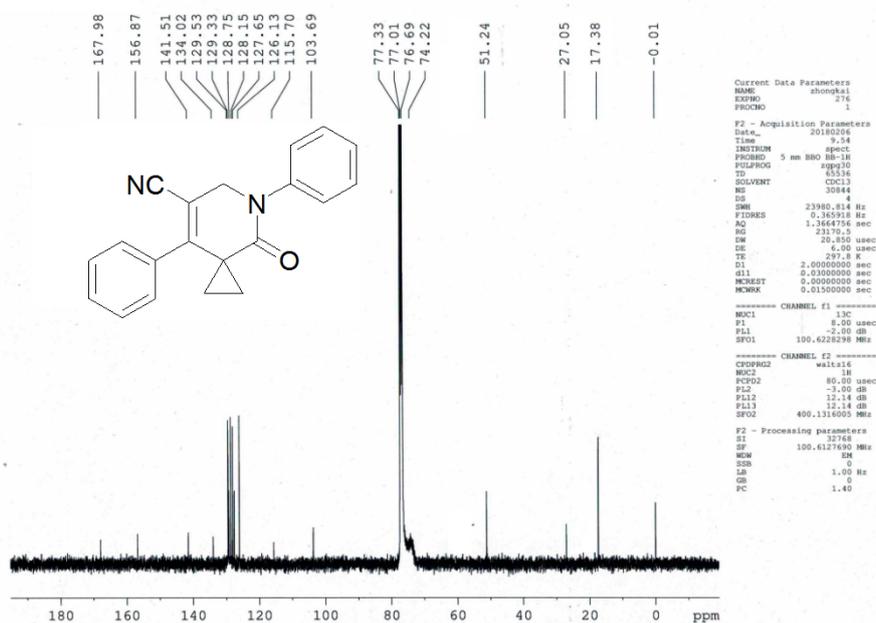
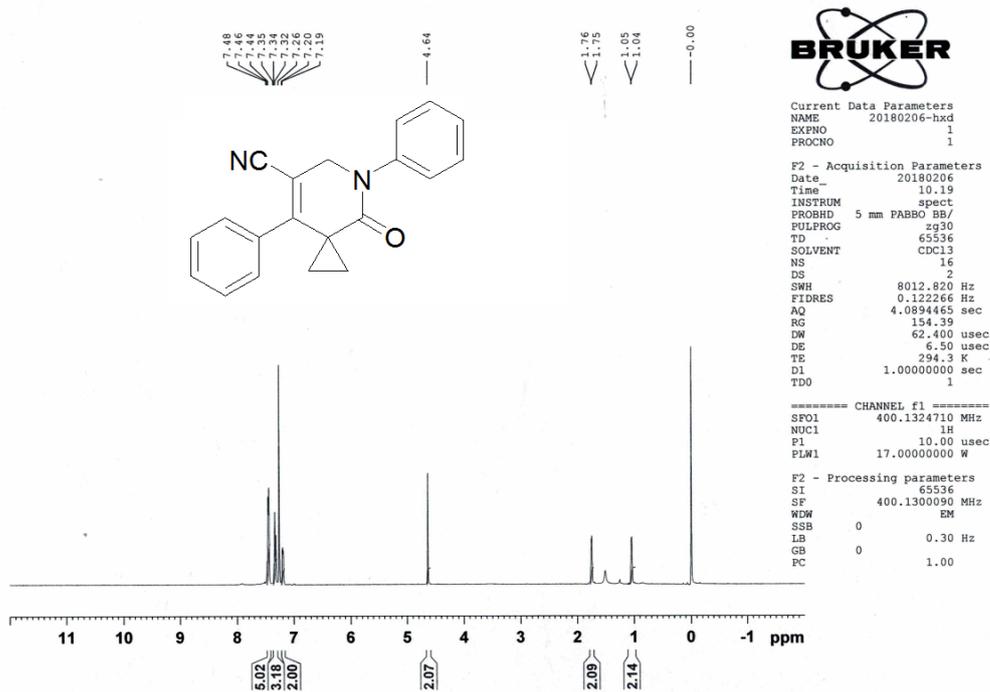


Figure 28. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound **5g**.

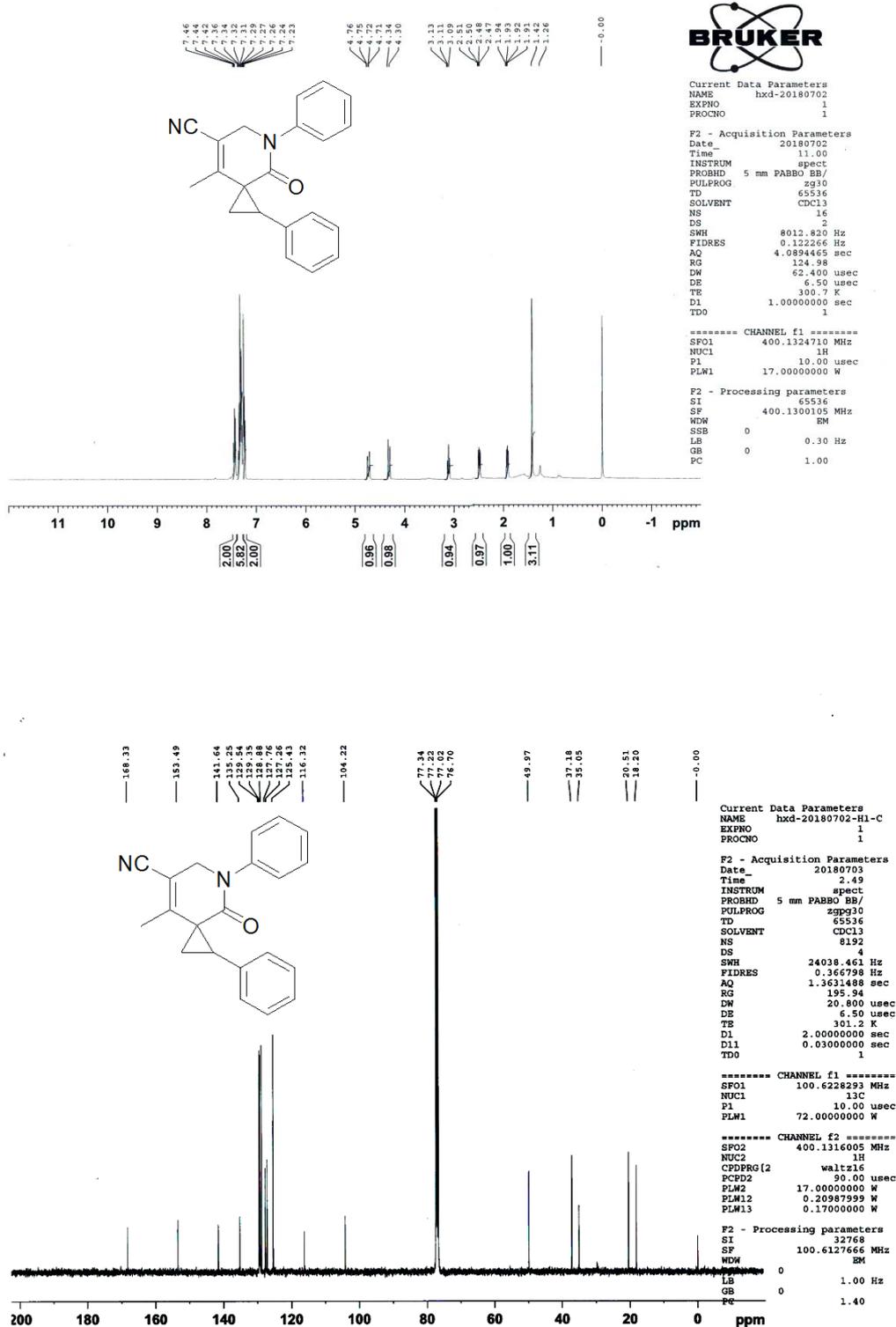


Figure 30. ¹H-(upper) and ¹³C-NMR (lower) spectra of compound **5i**.

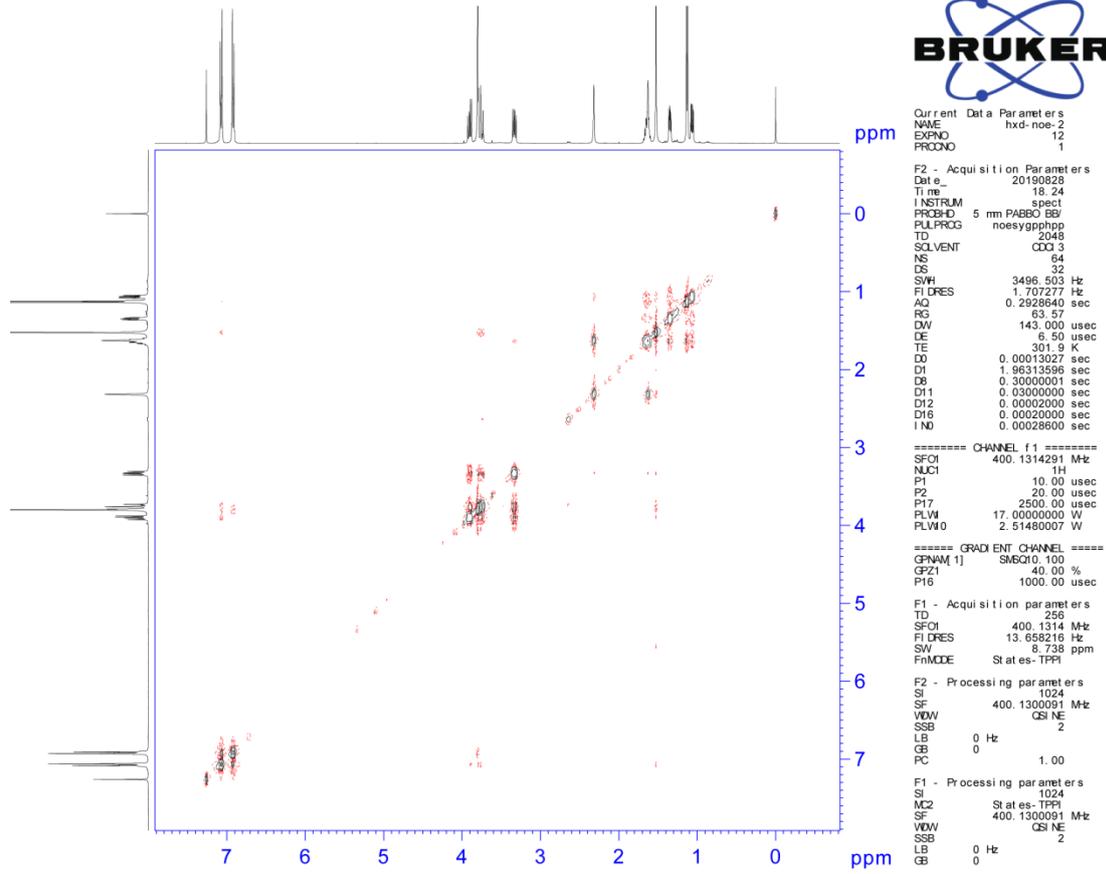


Figure 31. Noesy spectra of compound 4f