

Supporting Information

Reversal of Nucleophilicity of Enamides in Water: Control of Cyclization Pathways by Reaction Media for the Orthogonal Synthesis of Dihydropyridinone and Pyrrolidinone *Clausena* Alkaloids

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1. Experimental Details.

General information: ¹H and ¹³C NMR spectra were recorded on a Bruker Avance 300 spectrometer at ambient temperature (otherwise mentioned specially). Chemical shifts are reported in ppm versus tetramethylsilane with either tetramethylsilane or the residual solvent resonance used as an internal standard. Melting points are uncorrected. Elemental analyses were performed at the Analytical Laboratory of the Institute.

General procedure for the *N*-vinylation of oxiranecarboxamides: Under argon protection, a mixture of oxiranecarboxamide (1 mmol), CuI (0.4 mmol, 76mg), *N,N*-dimethylglycine hydrochloride (0.8 mmol, 112mg), Cs₂CO₃ (2 mmol, 650mg) and trans-β-phenylethenebromide (all trans-β-phenylethenebromides were prepared

according to a known method, Jaya Prakash Das Sujit Roy*, *J. Org. Chem.* 2002, 67, 7861-7864) (3 mmol) dry 1,4-dioxane (34 ml) was refluxed for 3 hours. After cooling, ethyl acetate (100 ml) was added and the resulting mixture was filtrated through a short silica gel (100-200 mesh) pad. The filtrate was concentrated and the residue was subjected to a silica gel column (100-200 mesh) using a mixture of petroleum ether and ethyl acetate (5:1) to give *E*-enamides. All products had been obtained as the by-products and fully characterized, Luo Yang, Gang Deng, De-Xian Wang, Zhi-Tang Huang, Jie-Ping Zhu, and Mei-Xiang Wang*, *Organic Letters*, **2007**, 9, 1387-1390).

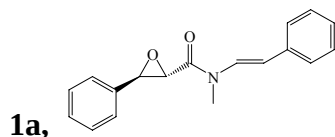
General procedure for the preparation of *N*-methyl-*N*-phenylvinyl

oxiranecarboxamides: Under argon protection, *N*-phenylvinyl oxiranecarboxamide (1 mmol) was dissolved in 10ml dry DMF at 0°C and purified NaH (2mmol, 48 mg) was added in one portion. After the mixture was stirred at room temperature for 1 h, CH₃I dissolved in 1ml dry DMF was added dropwise. Then the reaction mixture was stirred at room temperature for two hours more and water was added. The resulting mixture was extracted with ethyl acetate (3×15ml). The organic layer was washed with brine. The organic layer was dried with anhydrous Na₂SO₄, filtrated and concentrated under vacuum. Pure product **1a-f** was obtained after silica gel(200-300 mesh) column chromatography using a mixture of petroleum ether and ethyl acetate (4:1) as an eluant.

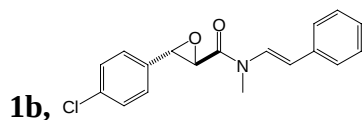
Table S1. Preparation of 1a-f

entry	Ar ¹	Ar ²	1' , yield (%)	1 , yield (%)
1	Ph	Ph	1a' , 73	1a , 100
2	4-Cl-C ₆ H ₄	Ph	1b' , 76	1b , 100
3	4-Me-C ₆ H ₄	Ph	1c' , 76	1c , 100
4	Ph	4-Cl-C ₆ H ₄	1d' , 64	1d , 75

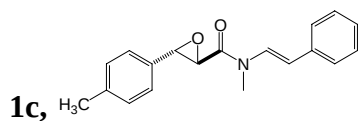
5	Ph	4-Me-C ₆ H ₄	1e' , 76	1e , 83
6	Ph	4-MeO-C ₆ H ₄	1f' , 86	1f , 87



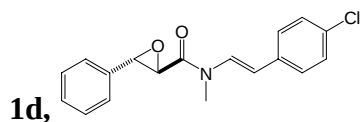
mp 100-101°C; IR (KBr) ν 1661, 1643 cm⁻¹; ¹H NMR (300 MHz, d⁶-DMSO, 360K) δ 7.69 (d, J =14.6 Hz, 1H); 7.12-7.40 (m, 10H); 6.20 (d, J =14.5 Hz, 1H); 4.21 (s, 1H); 4.13 (s, 1H); 3.24 (s, 3H); ¹³CNMR (75 MHz, d⁶-DMSO, 360K) δ 166.5, 137.4, 136.5, 129.5, 129.4, 129.3, 128.4, 127.3, 127.0, 126.5, 113.5, 58.3, 57.9, 31.8; MS (ESI): 280[M+1]⁺ (100), 312[M+Na]⁺ (30). Anal. Calcd. for C₁₈H₁₇NO₂: C, 77.40; H, 6.13; N, 5.01; Found: C, 77.33; H, 6.17; N, 5.20.



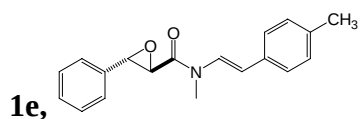
mp 136-137°C; IR (KBr) ν 1673, 1646 cm⁻¹; ¹H NMR (300 MHz, d⁶-DMSO, 360K) δ 7.70 (d, J =13.8 Hz, 1H); 7.17-7.44 (m, 9H); 6.24 (d, J =13.8 Hz, 1H); 4.23 (s, 1H); 4.18 (s, 1H); 3.25 (s, 3H); ¹³CNMR (75 MHz, d⁶-DMSO, 350K) δ 165.5, 137.5, 135.7, 134.6, 129.6, 129.2, 128.5, 127.5, 126.7, 113.7, 58.0, 57.8, 32.0; MS (EI): 313 [M]⁺ (7), 315(2), 295(9), 297(3), 207(100), 209(33), 178(60), 180(20), 99(30). Anal. Calcd. for C₁₈H₁₆NO₂Cl: C, 68.90; H, 5.14; N, 4.46; Found: C, 69.09; H, 5.14; N, 4.40.



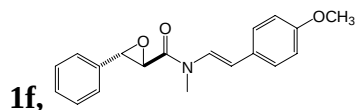
mp 117-118°C; IR (KBr) ν 1677, 1647 cm⁻¹; ¹H NMR (300 MHz, d⁶-DMSO, 350K) δ 7.71 (d, J =14.4 Hz, 1H); 7.13-7.34 (m, 9H); 6.22 (d, J =14.4 Hz, 1H); 4.21 (s, 1H); 4.09 (s, 1H); 3.24 (s, 3H); 2.97 (s, 3H); ¹³CNMR (75 MHz, d⁶-DMSO, 350K) δ 166.5, 139.0, 137.3, 133.3, 129.9, 129.3, 128.3, 127.2, 127.0, 126.4, 113.2, 58.1, 57.7, 31.8, 21.5; MS (EI): 294 [M]⁺ (15), 275(18), 234(60), 197(78), 187(100), 179(26), 158(49). Anal. Calcd. for C₁₉H₁₉NO₂: C, 77.79; H, 6.53; N, 4.77; Found: C, 77.68; H, 6.66; N, 4.95.



mp 138-139°C; IR (KBr) ν 1658, 1638 cm⁻¹; ¹H NMR (300 MHz, d⁶-DMSO, 350K) δ 7.75 (d, *J*=14.5 Hz, 1H); 7.27-7.41 (m, 9H); 6.21 (d, *J*=14.5 Hz, 1H); 4.26 (s, 1H); 4.15 (s, 1H); 3.24 (s, 3H); ¹³CNMR (75 MHz, d⁶-DMSO, 350K) δ 165.0, 134.8, 130.2, 128.0, 127.8, 127.5, 126.4, 125.5, 110.3, 56.7, 56.1, 30.2; MS (ESI): 314 [M+1]⁺(100), 316 (30), 336(50), 338(17); Anal. Calcd. for C₁₈H₁₆NO₂Cl: C, 68.90; H, 5.14; N, 4.46; Found: C, 68.98; H, 5.08; N, 4.52.



mp 118-119°C; IR (KBr) ν 1655, 1637 cm⁻¹; ¹H NMR (300 MHz, d⁶-DMSO, 350K) δ 7.68 (d, *J*=14.5 Hz, 1H); 7.39-7.42 (m, 5H); 7.23 (d, *J*=7.7 Hz, 2H); 7.09 (d, *J*=7.7 Hz, 2H); 6.21 (d, *J*=14.5 Hz, 1H); 4.23 (s, 1H); 4.16 (s, 1H); 3.25 (s, 3H); 2.28 (s, 3H); ¹³CNMR (75 MHz, d⁶-DMSO, 350K) δ 166.6, 136.8, 136.7, 134.7, 130.2, 129.7, 129.5, 127.8, 127.3, 126.6, 113.7, 58.4, 58.1, 32.0, 21.6; MS (EI): 293 [M]⁺(6), 275(9), 194(13), 173(100), 144(54); Anal. Calcd. for C₁₉H₁₉NO₂: C, 77.79; H, 6.53; N, 4.77; Found: C, 77.84; H, 6.59; N, 4.83.



mp 90-91°C; IR (KBr) ν 1657, 1640 cm⁻¹; ¹H NMR (300 MHz, d⁶-DMSO, 360K) δ 7.59 (d, *J*=14.5 Hz, 1H); 7.42 (s, 5H); 7.28 (d, *J*=8.0 Hz, 1H); 6.87 (d, *J*=8.0 Hz, 1H); 4.23 (s, 1H); 4.15 (s, 1H); 3.76 (s, 3H); 3.25 (s, 3H); ¹³CNMR (75 MHz, d⁶-DMSO, 350K) δ 166.4, 159.6, 136.7, 130.1, 129.7, 129.5, 127.9, 127.2, 127.0, 115.5, 113.8, 58.4, 58.1, 56.3, 32.1; MS (EI): 309 [M]⁺ (15), 291 (32), 210 (24), 173 (100), 144 (48); Anal. Calcd. for C₁₉H₁₉NO₃: C, 73.77; H, 6.19; N, 4.53; Found: C, 73.56; H, 6.27; N, 4.64.

General procedure for the synthesis of homoclausenamides 2. To a mixture of enamides **1** (1 mmol) and molecular sieve (4Å, 0.2g) in dry Bu^tOH (30 mL) under argon protection was added TFA (2 mmol). After the mixture was refluxed for 12 h, a saturated aqueous solution of NaHCO₃ (30 mL) was added, and the resulting mixture

was extracted with ethyl acetate (3×20 mL). The organic layer was dried with anhydrous Na₂SO₄, filtrated and concentrated under vacuum. Pure product **2** was obtained after silica gel (200-300 mesh) column chromatography using a mixture of petroleum ether and ethyl acetate (1:1) as an eluant.

General procedure for the synthesis of neoclausenamides **4 and their 6-epimers **4'**.** Refluxing a suspension of enamides **1** (1 mmol) in deionized water (30 mL) for 5 h under argon protection gave rise to a homogeneous solution. After addition of brine (30 mL), the mixture was extracted with ethyl acetate (3×20 mL). The organic layer was dried with anhydrous Na₂SO₄, filtrated and concentrated under vacuum. The chromatography using a silica gel (200-300 mesh) column eluting with a mixture of ethyl acetate and methanol (95:5) gave products **4** and **4'**.

2. X-ray structures of **2a, **4a**, **4a'****

Single crystal of **2a** was obtained from slow evaporation of the solvent from **2a** solution in a mixture of ethyl acetate and hexane (1:1). To obtain single crystals of **4a** and **4a'**, recrystallization of a mixture of reaction products **4a** and **4a'** (**4a**:**4a'** = 3:7) in ethyl acetate gave pure **4a'** as solid. Slow evaporation of the solvent from **4a'** solution in a mixture of ethyl acetate and THF (1:1) gave single crystal of **4a'**. The filtrate, which contains a mixture of **4a** and **4a'** (**4a**:**4a'** = 1:1) after recrystallization of **4a'**, was subjected to the preparative HPLC with a C-18 column to give a fraction in which the ratio of **4a** over **4a'** was 6:1. Solw evaporation of the solvent from its solution in a mixture of ethyl acetate and THF (1:1) gave single crystal of **4a**. CCDCs 673548-673550 contain the crystallographic data for compounds **2a**, **4a** and **4a'**.

These data can be obtained free of charge via www.ccdc.ac.uk/cont/retriving.html (or from the Cambridge Crystallographic Data Center, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (+44)1223-336-033; or deposit@ccdc.cam.ac.uk).

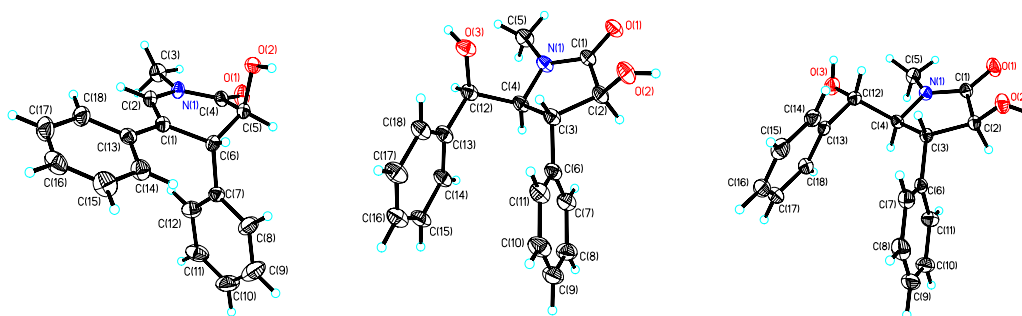
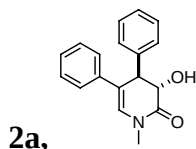
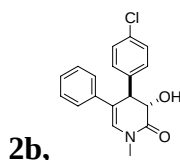


Figure S1. X-ray crystal structures of **2a** (left), **4a** (middle) and **4a'** (right)

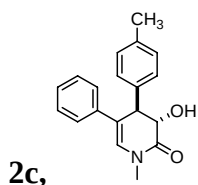
3. Spectroscopic data of products prepared.



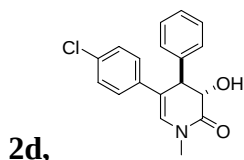
mp 193-194 °C; IR (KBr) ν 3259, 1664, 1633 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3 , 300K) δ 7.04-7.25 (m, 10H); 6.41 (d, $J=1.9$ Hz, 1H); 4.36 (dd, $J=2.4, 10.5$ Hz, 1H); 4.20 (dd, $J=1.8, 10.5$ Hz, 1H); 3.52 (d, $J=2.5$ Hz, 1H); 3.25 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3 , 300K) δ 169.2, 139.0, 137.7, 128.7, 128.6, 128.2, 127.1, 126.6, 126.1, 120.8, 73.3, 49.6; MS (ESI): 279 $[\text{M}]^+$ (48), 264 (67), 250 (100); Anal. Calcd. for $\text{C}_{18}\text{H}_{17}\text{NO}_2$: C, 77.40; H, 6.13; N, 5.01; Found: C, 77.09; H, 6.15; N, 5.13;



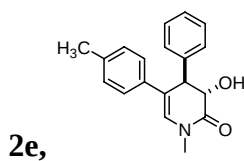
mp 154-156 °C; IR (KBr) ν 3363, 1663, 1647 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3 , 300K) δ 7.00-7.26 (m, 9H); 2.51 (d, $J=5.5$ Hz, 1H); 6.37 (d, $J=2.1$ Hz, 1H); 4.31 (dd, $J=2.1, 11.3$ Hz, 1H); 4.19 (dd, $J=2.1, 11.3$ Hz, 1H); 3.59 (d, $J=2.1$ Hz, 1H); 3.24 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3 , 300K) δ 169.2, 138.0, 137.3, 132.8, 130.1, 128.7, 128.6, 128.3, 126.8, 126.3, 120.5, 73.0, 19.0, 34.3; MS (EI): 313 $[\text{M}]^+$ (39), 315 (13), 295 (47), 297 (16), 284 (100), 286 (25); Anal. Calcd. for $\text{C}_{18}\text{H}_{16}\text{NO}_2\text{Cl}$: C, 68.90; H, 5.14; N, 4.46; Found: C, 68.61; H, 5.26; N, 4.42;



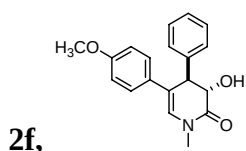
mp 180-182 °C; IR (KBr) ν 3285, 1662, 1648 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3 , 300K) δ 7.02-7.15 (m, 9H); 6.40 (d, $J=1.8$ Hz, 1H); 4.32 (dd, $J=2.6, 10.0$ Hz, 1H); 4.16 (dd, $J=1.8, 10.1$ Hz, 1H); 3.52 (d, $J=2.7$ Hz, 1H); 3.23 (s, 3H); 2.23 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3 , 300K) δ 169.3, 137.8, 136.6, 135.9, 129.4, 128.5, 126.6, 126.1, 120.9, 73.4, 49.2, 34.3, 21.0; MS (EI): 293 $[\text{M}]^+$ (55), 275 (80); 264 (100); Anal. Calcd. for $\text{C}_{19}\text{H}_{19}\text{NO}_2$: C, 77.79; H, 6.53; N, 4.77; Found: C, 77.65; H, 6.35; N, 4.86;



mp 195-197 °C; IR (KBr) ν 3296, 1661, 1646 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3 , 300K) δ 7.09-7.26 (m, 7H); 6.97 (dt, $J=2.4, 8.5$ Hz, 2H); 6.39 (d, $J=1.9$ Hz, 1H); 4.36 (dd, $J=2.2, 10.7$ Hz, 1H); 4.16 (dd, $J=2.2, 10.7$ Hz, 1H); 3.51 (d, $J=2.3$ Hz, 1H); 3.24 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3 , 300K) δ 169.2, 138.9, 136.2, 132.3, 128.7, 128.68, 128.6, 128.3, 127.4, 127.2, 119.8, 73.1, 49.5, 34.3; MS (ESI): 314 $[\text{M}+1]^+$ (23), 316 (8), 336 $[\text{M}+\text{Na}]^+$ (100), 336 (33); Anal. Calcd. for $\text{C}_{18}\text{H}_{16}\text{NO}_2\text{Cl}$: C, 68.90; H, 5.14; N, 4.46; Found: C, 68.62; H, 5.28; N, 4.38;

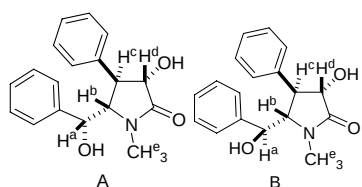


mp 169-170 °C; IR (KBr) ν 3318, 1660, 1646 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3 , 300K) δ 7.13-7.26 (m, 5H); 6.95 (s, 4H); 6.37 (d, $J=1.8$ Hz, 1H); 4.35 (dd, $J=2.4, 10.4$ Hz, 1H); 4.18 (dd, $J=1.7, 10.4$ Hz, 1H); 3.48 (d, $J=2.6$ Hz, 1H); 3.23 (s, 3H); 2.23 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3 , 300K) δ 169.2, 139.1, 136.4, 134.7, 128.9, 128.7, 128.6, 127.5, 127.1, 125.9, 120.6, 73.4, 49.6, 34.3, 21.0; MS (EI): 293 $[\text{M}]^+$ (70), 264 (100); Anal. Calcd. for $\text{C}_{19}\text{H}_{19}\text{NO}_2$: C, 77.79; H, 6.53; N, 4.77; Found: C, 77.79; H, 6.71; N, 4.50;

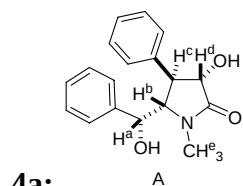


mp 161-163 °C; IR (KBr) ν 3287, 1659, 1648 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3 , 300K) δ 7.04-7.25 (m, 5H); 6.98 (dt, $J=2.1, 9.7$ Hz, 1H); 6.68 (dt, $J=2.1, 8.9$ Hz, 1H); 6.33 (d, $J=1.9$ Hz, 1H); 4.34 (dd, $J=2.5, 10.4$ Hz, 1H); 4.16 (dd, $J=1.8, 10.3$ Hz, 1H); 3.71 (s, 3H); 3.51 (d, $J=2.6$ Hz, 1H); 3.23 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3 , 300K) δ 169.1, 158.4, 139.2, 130.1, 128.7, 128.6, 127.3, 127.1, 127.0, 120.5, 113.6, 73.3, 55.2, 49.6, 34.3; MS (EI): 309 $[\text{M}]^+$ (95), 280 (100); Anal. Calcd. for $\text{C}_{19}\text{H}_{19}\text{NO}_3$: C, 73.77; H, 6.19; N, 4.53; Found: C, 73.66; H, 6.32; N, 4.19;

4a+4a': (30:70)



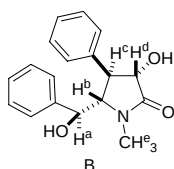
mp 197-199 °C; ^1H NMR (300 MHz, d^6 -DMSO, 300K) δ 6.61-7.24 (m, 10H); $\text{H}_\text{A}^\text{a}$, 5.01 (d, $J=2.5$ Hz, 0.30H); $\text{H}_\text{B}^\text{a}$ 4.62 (d, $J=2.2$ Hz, 0.70H); $\text{H}_\text{B}^\text{b}$ 4.28 (dd, $J=2.4, 10.7$ Hz, 0.70H); $\text{H}_\text{A}^\text{b}$, $\text{H}_\text{A}^\text{d}$, 3.86-3.90 (m, 0.60H); $\text{H}_\text{B}^\text{d}$ 3.82 (d, $J=11.1$ Hz, 0.70H); $\text{H}_\text{B}^\text{c}$ 3.50 (dd, $J=8.4, 11.0$ Hz, 0.70H); $\text{H}_\text{A}^\text{c}$, 3.05 (t, $J=7.0$, 0.30H); $\text{H}_\text{B}^\text{e}$ 3.00 (s, 2.10H); $\text{H}_\text{A}^\text{e}$ 2.91 (s, 0.90H); ^1H NMR (300 MHz, d^6 -DMSO+ D_2O , 300K) δ 6.6-7.25 (m, 10H); $\text{H}_\text{A}^\text{a}$, 5.02 (d, $J=2.5$ Hz, 0.30H); $\text{H}_\text{B}^\text{a}$ 4.63 (d, $J=2.1$ Hz, 0.70H); $\text{H}_\text{B}^\text{b}$ 4.29 (dd, $J=2.4, 8.4$ Hz, 0.70H); $\text{H}_\text{A}^\text{b}$, $\text{H}_\text{A}^\text{d}$, 3.86-3.90 (m, 0.60H); $\text{H}_\text{B}^\text{d}$ 3.83 (d, $J=11.0$ Hz, 0.70H); $\text{H}_\text{B}^\text{c}$ 3.51 (dd, $J=8.4, 10.9$ Hz, 0.70H); $\text{H}_\text{A}^\text{c}$, 3.06 (t, $J=7.0$, 0.30H); $\text{H}_\text{B}^\text{e}$ 3.01 (s, 2.10H); $\text{H}_\text{A}^\text{e}$ 2.91 (s, 0.90H);



4a:

mp 198-201 °C; IR (KBr) ν 3440, 3346, 1658 cm^{-1} ; ^1H NMR (300 MHz, d^6 -DMSO, 300K) δ 6.85-7.26 (m, 10H); 5.72 (d, $J=4.7$ Hz, 1H); 5.58 (d, $J=6.3$ Hz, 1H); $\text{H}_\text{A}^\text{a}$,

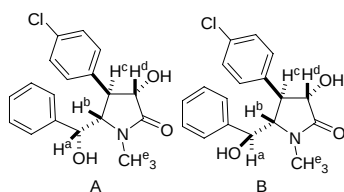
5.00 (t, $J=4.1$ Hz, 1H); H_A^d , 3.90 (t, $J=6.7$ Hz, 1H); H_A^b , 3.87 (dd, $J=2.8, 6.6$ Hz, 1H); H_A^c , 3.05 (dd, 7.1, 14.3 Hz, 1H); H_A^e 2.91 (s, 3H); ^{13}C NMR (75 MHz, d^6 -DMSO, 300K) δ 127.8, 141.9, 140.8, 127.8, 127.5, 127.4, 126.5, 126.1, 125.7, 77.0, 68.6, 67.5, 46.4, 27.9; MS (ESI): 298[M+1] $^+$ (100), 320[M+Na] $^+$ (48); Anal. Calcd. for $\text{C}_{18}\text{H}_{19}\text{NO}_3$: C, 72.71; H, 6.44; N, 4.71; Found: C, 72.54; H, 6.33; N, 4.84;



4a':

mp 203-205°C; IR (KBr) ν 3418, 3221, 1673 cm^{-1} ; ^1H NMR (300 MHz, d^6 -DMSO, 300K) δ 7.08-7.25 (m, 10H); H_B^a , 4.59 (d, $J=4.4$ Hz, 1H); H_B^b , 4.01 (d, $J=7.8$ Hz, 1H); H_B^d , 3.85 (dd, $J=4.5, 7.3$ Hz, 1H); H_B^c , 2.93 (t, $J=7.6$ Hz, 1H); H_B^e , 2.70 (s, 3H); ^{13}C NMR (75 MHz, d^6 -DMSO, 300K) δ 173.1, 142.6, 140.9, 128.9, 127.8, 126.9, 126.4, 126.3, 76.0, 72.9, 67.5, 50.1, 29.9; MS (ESI): 298[M+1] $^+$, Anal. Calcd. for $\text{C}_{18}\text{H}_{19}\text{NO}_3$: C, 72.71; H, 6.44; N, 4.71; Found: C, 72.72; H, 6.58; N, 4.87;

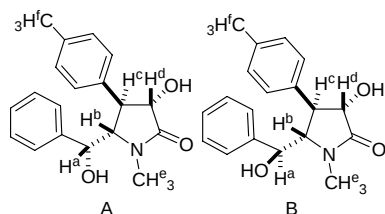
4b+4b': (10:90)



mp 213-215°C; IR (KBr) ν 3382, 1669 cm^{-1} ; ^1H NMR (300 MHz, d^6 -DMSO, 300K) δ 7.08-7.55 (m, 9H); 5.77 (d, $J=5.0$ Hz, 0.90H); 5.70 (d, $J=6.0$ Hz, 0.90H); 5.52 (d, $J=6.4$ Hz, 0.10H); 5.45 (d, $J=4.9$ Hz, 0.10H); H_A^a , 4.90 (dd, $J=6.5, 10.5$ Hz, 0.10H); H_B^a , 4.58 (t, $J=4.7$ Hz, 0.90H); H_A^b , 4.23 (d, $J=5.0$ Hz, 0.10H); H_A^d , 4.16 (d, $J=8.1$ Hz, 0.10H); H_B^d , 4.00 (dd, $J=6.2, 7.8$ Hz, 0.90H); H_B^b , 3.85 (dd, $J=4.6, 7.7$ Hz, 0.90H); H_A^c , 3.53 (dd, $J=8.0, 10.0$ Hz, 0.10H); H_B^c , 2.89 (t, $J=8.0$ Hz, 0.90H); H_B^e , 2.73 (s, 2.70H); H_A^e , 2.16 (s, 0.30H); ^{13}C NMR (75 MHz, d^6 -DMSO, 300K) δ 174.6, 172.9, 143.8, 142.3, 139.8, 136.6, 130.9, 130.4, 129.8, 128.2, 128.0, 127.89, 127.8, 127.0, 126.6, 126.4, 125.3, 46.0, 73.3, 69.8, 69.0, 67.0, 66.1, 51.2, 49.7, 30.5, 29.9;

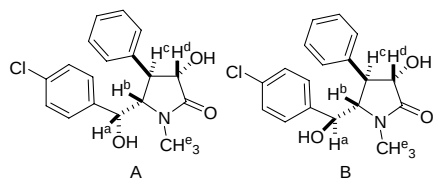
MS (ESI): $[M+1]^+$ (74), 334 (25), 354 $[M+Na]^+$ (100), 356 (33); Anal. Calcd. for $C_{18}H_{18}NO_3Cl$: C, 65.16; H, 5.47; N, 4.22; Found: C, 64.77; H, 5.48; N, 4.06;

4c+4c': (50:50)



mp 168-169°C; IR (KBr) ν 3411, 3336, 3375, 1642, 1627 cm^{-1} ; 1H NMR (300 MHz, $CDCl_3$, 300K) δ 6.97-7.27 (m, 9H); H_A^a , H_B^a , 4.95-4.99 (m, 1.00H); H_B^d , 4.42 (d, $J=10.2$ Hz, 0.50H); H_A^d , 4.13 (d, $J=10.5$ Hz, 0.50H); H_B^b , 3.80 (dd, $J=10.6, 12.7$ Hz, 0.50H); 3.71 (d, $J=1.0$ Hz, 0.50H); H_A^b , 3.67 (dd, $J=2.7, 12.6$ Hz, 0.50H); 3.57 (s, 0.50H); H_A^c , H_B^c , 3.18-3.28 (m, 1.00H); 3.10 (s, 1.50H); 3.09 (s, 1.50H); 2.51 (d, $J=5.5$ Hz, 0.50H); 2.21 (s, 1.50H); 2.20 (s, 1.50H); 2.16 (d, $J=3.6$ Hz, 0.50H); ^{13}C NMR (75 MHz, $CDCl_3$, 300K) δ 172.6, 139.1, 137.2, 136.3, 136.1, 129.7, 129.4, 129.2, 129.1, 128.7, 128.53, 128.5, 128.4, 128.2, 128.1, 127.8, 127.32, 127.3, 126.1, 87.1, 84.9, 74.2, 71.6, 54.2, 49.8, 48.9, 43.3, 33.4, 31.4, 21.0; MS (ESI): $[M+1]^+$ (100), 334 $[M+Na]^+$ (92); Anal. Calcd. for $C_{19}H_{21}NO_3$: C, 73.29; H, 6.80; N, 4.50; Found: C, 73.08; H, 6.86; N, 4.79;

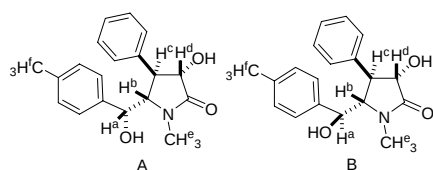
4d+4d': (44:56)



mp 185-186°C; IR (KBr) ν 3403, 3336, 1688, 1648 cm^{-1} ; 1H NMR (300 MHz, d^6 -DMSO, 300K) δ 6.67-7.49 (m, 9H); 5.78 (d, $J=4.8$ Hz, 0.45H); 5.60 (s, 0.55H); 5.58 (d, $J=4.4$ Hz, 0.45H); 5.42 (d, $J=6.3$ Hz, 0.55H); H_A^a , 5.04 (t, $J=2.9$ Hz, 0.45H); H_B^a , 4.68 (dd, $J=1.7, 3.8$ Hz, 0.55H); H_B^b , 4.27 (dd, $J=2.0, 8.3$ Hz, 0.55H); H_B^d , 4.08 (dd, $J=6.4, 10.9$ Hz, 0.55H); H_A^b , H_A^d , 3.86-3.94 (m, 0.90H); H_B^c , 3.49 (dd, $J=8.4,$

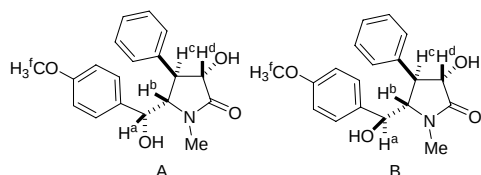
10.8 Hz, 0.55H); H_A^c , 3.04 (d, $J=7.8$ Hz, 0.45H); H_B^e , 2.99 (s, 1.65H); H_A^e , 2.92 (s, 1.35H); ^{13}C NMR (75 MHz, d^6 -DMSO, 300K) δ 174.1, 172.7, 141.6, 140.0, 139.8, 136.0, 131.0, 130.9, 128.7, 128.2, 127.8, 127.7, 127.6, 127.2, 127.1, 126.2, 125.6, 77.1, 70.8, 68.7, 67.6, 67.0, 65.0, 49.2, 46.3, 29.8, 27.7; MS (ESI): $[M+1]^+$ (47), 334 (19), 354 $[M+Na]^+$ (100), 356 (33); Anal. Calcd. for $\text{C}_{18}\text{H}_{18}\text{NO}_3\text{Cl}$: C, 65.16; H, 5.47; N, 4.22; Found: C, 64.19; H, 5.51; N, 3.93 ;

4e+4e': (45:55)



mp 199-201 °C; IR (KBr) ν 3405, 3336, 1688 cm^{-1} ; ^1H NMR (300 MHz, d^6 -DMSO, 300K) δ 6.49-7.32 (m, 9H); 5.68 (d, $J=4.6$ Hz, 0.08H); 5.57 (d, $J=6.4$ Hz, 0.08H); 5.37 (d, $J=6.2$ Hz, 0.92H); 5.36 (d, $J=3.8$ Hz, 0.92H); H_A^a , 4.92 (s, 0.08 H); H_B^a , 4.56 (t, $J=2.8$ Hz, 0.92H); H_B^b , 4.28 (dd, $J=2.3, 10.6$ Hz, 0.92H); H_A^d , 3.89 (t, $J=7.0$ Hz, 0.08H); H_A^b , 3.83 (dd, $J=2.9, 6.9$ Hz, 0.08H); H_B^d , 3.64 (dd, $J=6.2, 11.0$ Hz, 0.92H); H_B^c , 3.50 (t, $J=8.3$ Hz, 0.92H); H_B^e , 3.03 (s, 2.76H); H_A^e , 2.89 (s, 0.24H); H_B^f , 2.21 (s, 2.76H); H_A^f , 2.15 (s, 0.24H); ^{13}C NMR (75 MHz, d^6 -DMSO, 300K) δ 176.4, 174.7, 140.6, 139.0, 138.0, 137.5, 137.0, 136.2, 129.3, 129.5, 128.9, 128.8, 128.3, 127.4, 127.1, 127.0, 126.2, 125.0, 75.3, 70.9, 70.5, 70.3, 68.3, 52.3, 50.5, 31.6, 31.1, 21.0; MS (ESI): 312 $[M+1]^+$ (74), 334 $[M+Na]^+$ (100); 350 $[M+K]^+$ (55); Anal. Calcd. for $\text{C}_{19}\text{H}_{21}\text{NO}_3$: C, 73.29; H, 6.80; N, 4.50; Found: C, 73.22; H, 6.86; N, 4.57;

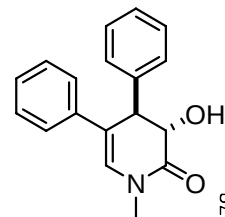
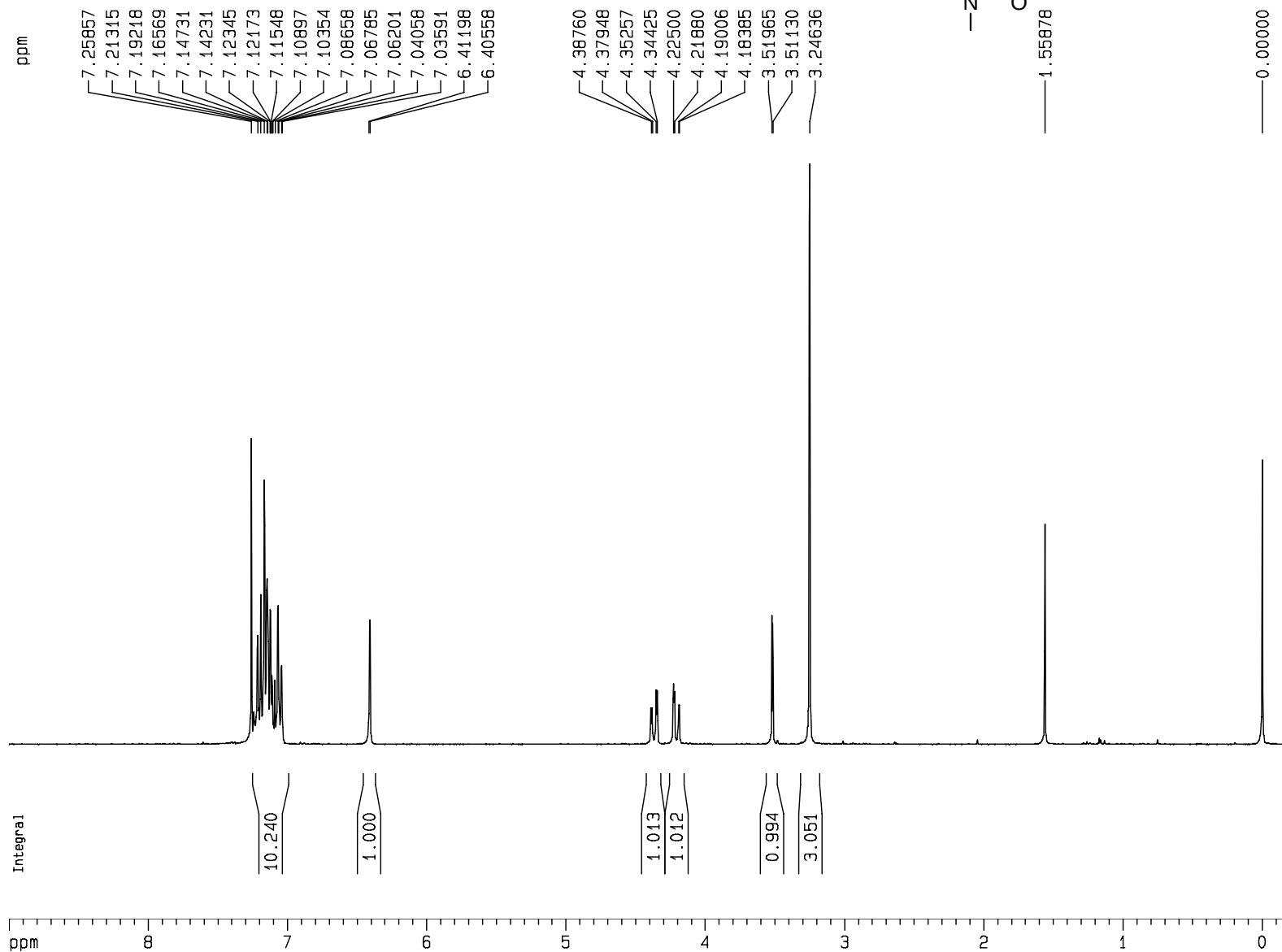
4f+4f': (61:39)



mp 146-148 °C; IR (KBr) ν 3393, 1684 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3 , 300K) δ 6.71-7.50 (m, 9H); H_A^a , 5.23 (dd, $J=2.2, 10.4$ Hz, 0.61H); H_B^a , 4.82 (t, $J=3.4$ Hz,

0.39H); H_A^b, 4.50 (t, *J*=5.4 Hz, 0.61H); H_B^b, 4.21 (t, *J*=4.5 Hz, 0.39H); H_A^d, 3.95 (d, *J*=8.2 Hz, 0.61H); H_B^b, H_B^d, 3.87-3.90 (m, 0.78H); H_A^f, 3.79 (s, 1.83H); H_B^f, 3.77 (s, 1.17H); H_A^c, 3.72 (dd, *J*=8.5, 10.3 Hz, 0.61H); 3.23 (d, *J*=2.5 Hz, 0.39H); H_B^c, 3.02 (t, *J*=5.3 Hz, 0.61H); H_B^e, 2.91 (s, 1.17H); 2.85 (d, *J*=3.3 Hz, 0.39H); H_A^e, 2.41 (s, 1.83H); 2.03 (t, *J*=5.4 Hz, 1H); ¹³CNMR (75 MHz, CDCl₃, 300K) δ 176.4, 174.6, 159.3, 158.9, 140.7, 136.2, 134.0, 133.1, 128.9, 128.8, 128.2, 127.6, 127.4, 127.0, 126.9, 126.2, 113.8, 113.78, 77.2, 75.6, 70.9, 70.5, 70.2, 68.3, 55.3, 55.2, 52.4, 50.5, 31.7, 31.1; MS (ESI): 328 [M+1]⁺ (100), 350[M+Na]⁺ (67); Anal. Calcd. for C₁₉H₂₁NO₄: C, 69.71; H, 6.47; N, 4.28; Found: C, 69.50; H, 6.57; N, 4.39;

4. ¹H and ¹³C NMR spectra of 2a-2f, 4a-4f and 4a'-4f'.



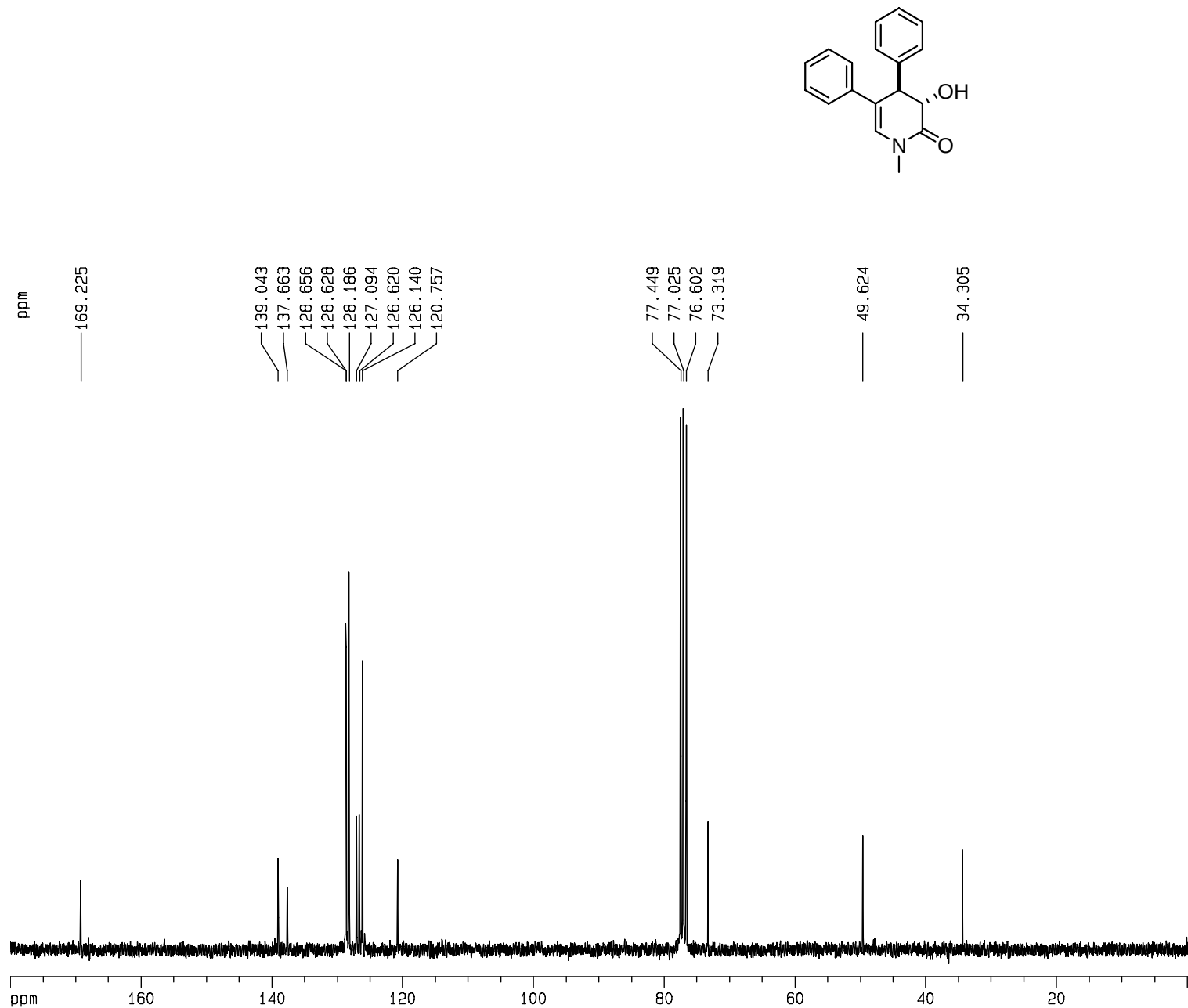
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PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 322.5
DW 55.600 usec
DE 6.00 usec
TE 297.7 K
D1 5.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300069 MHz
WDW EM
SSB 0
LB 0.35 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 10.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.41818 ppm/cm
HZCM 125.50892 Hz/cm



Current Data Parameters
 NAME y1055-1
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters

Date_ 20061028
 Time 16.23
 INSTRUM av300
 PROBHD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 404
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 456.1
 DW 27.800 usec
 DE 6.00 usec
 TE 300.1 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====

NUC1 13C
 P1 9.40 usec
 PL1 -1.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====

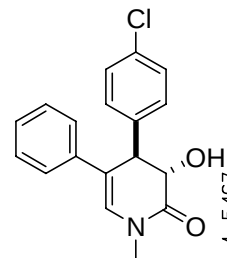
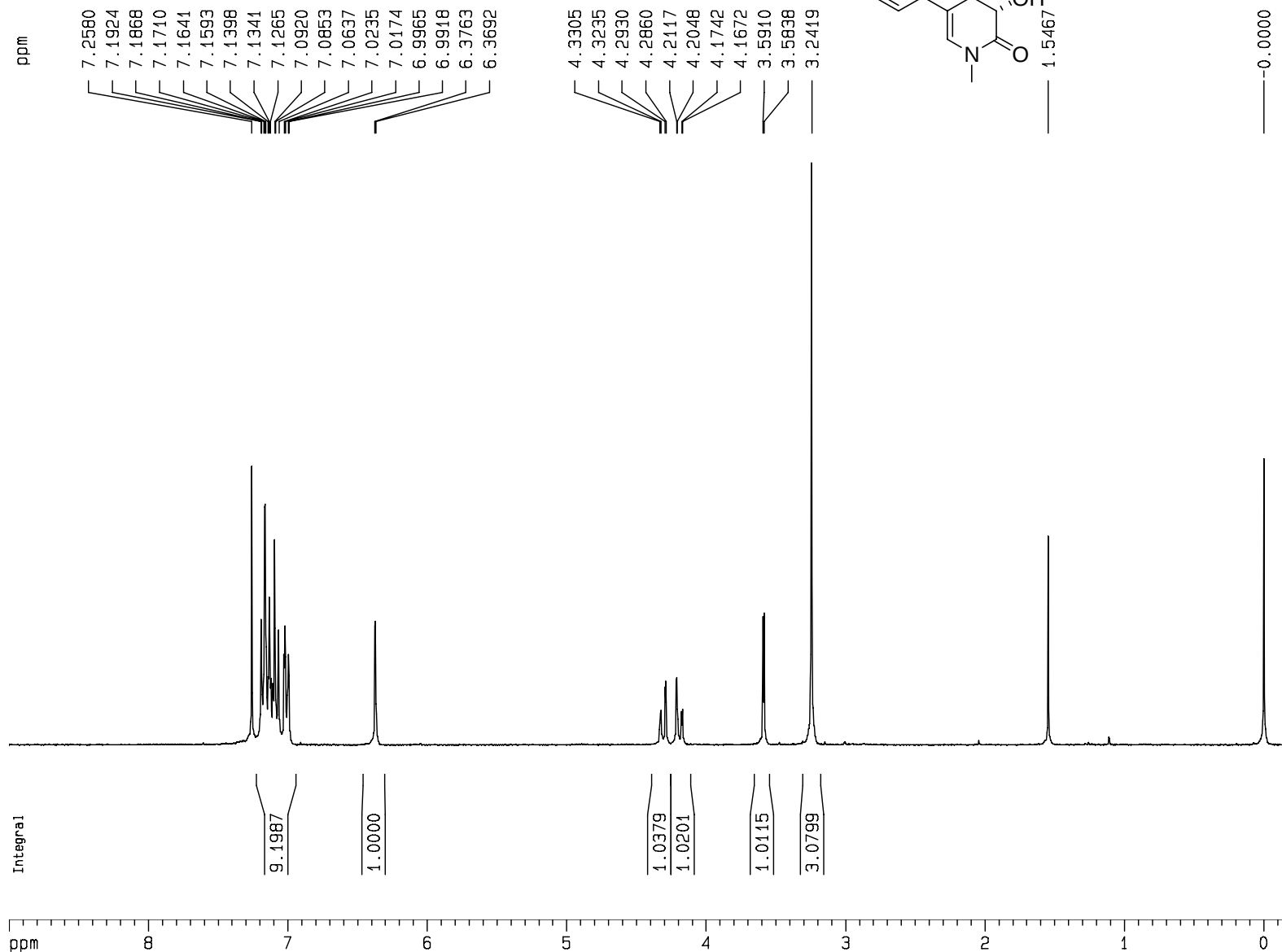
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 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 18.00 dB
 PL13 18.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters

SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters

CX 22.00 cm
 CY 10.00 cm
 F1P 180.000 ppm
 F1 13584.20 Hz
 F2P -2.000 ppm
 F2 -150.94 Hz
 PPMCM 8.27273 ppm/cm
 HZCM 624.32410 Hz/cm



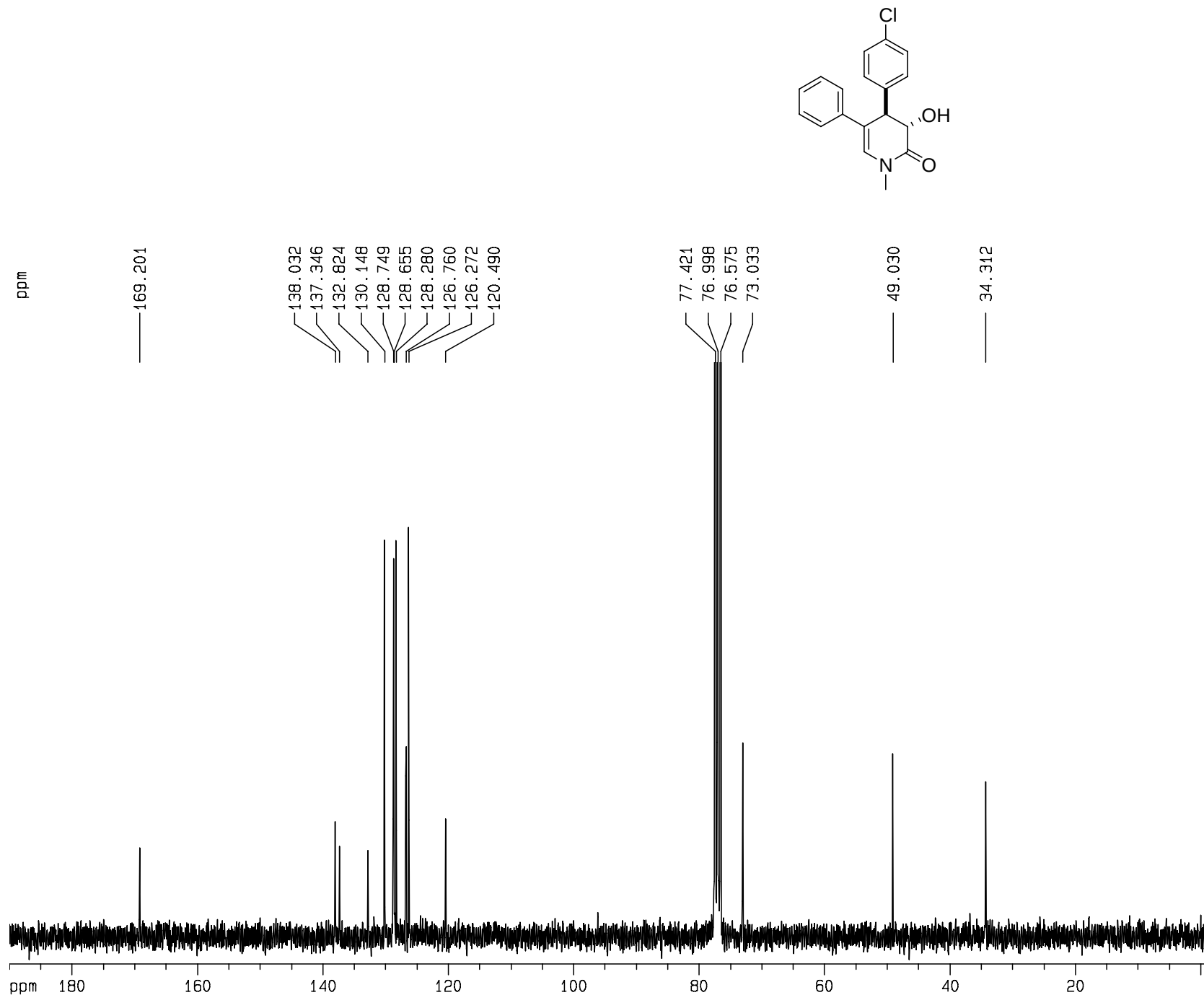
Current Data Parameters
NAME y1011-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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INSTRUM av300
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PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 574.7
DW 55.600 usec
DE 6.00 usec
TE 300.1 K
D1 5.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.30 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300069 MHz
WDW EM
SSB 0
LB 0.35 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 10.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.41818 ppm/cm
HZCM 125.50892 Hz/cm



Current Data Parameters
 NAME y1011-4
 EXPNO 50
 PROCNO 1

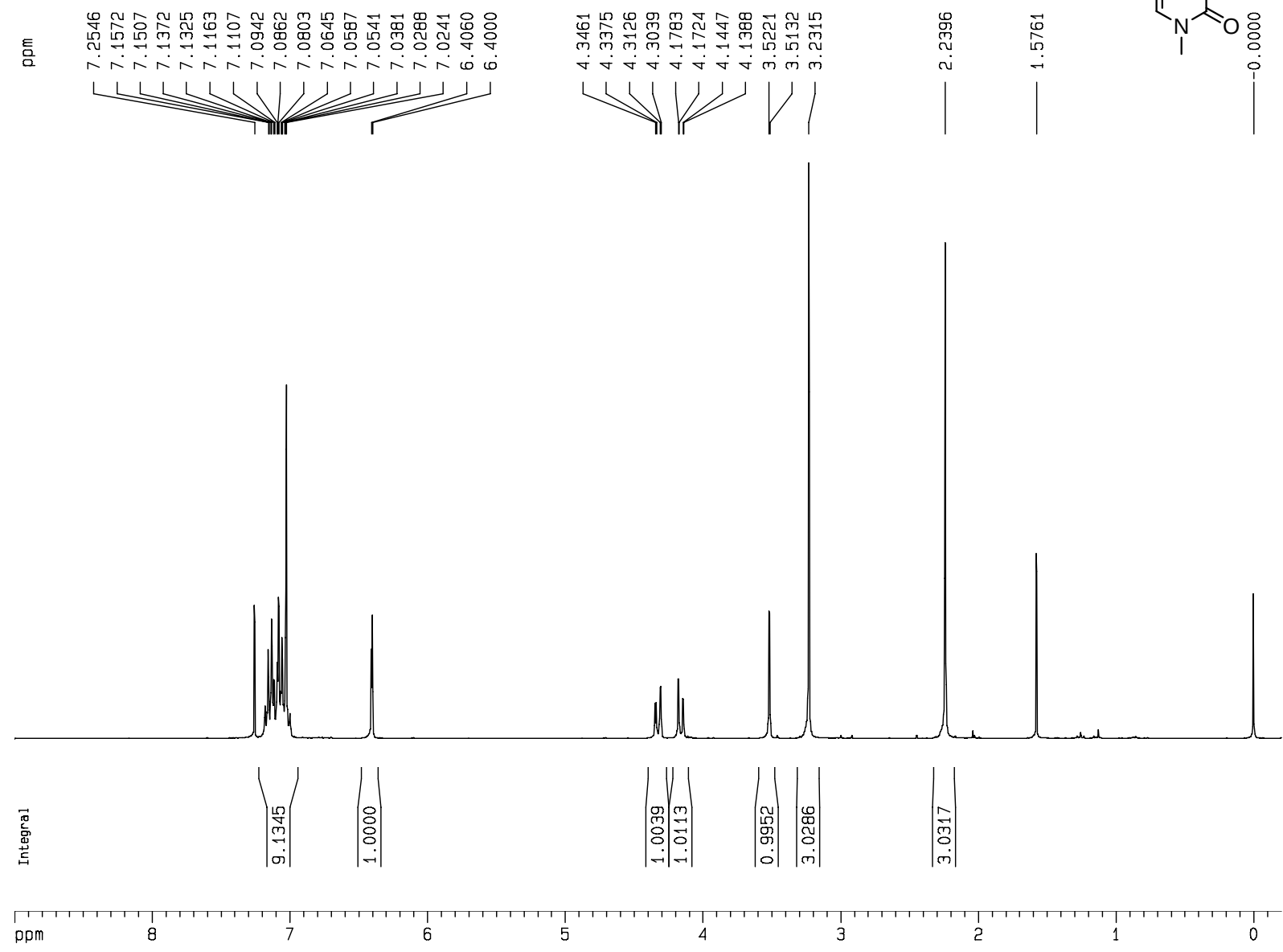
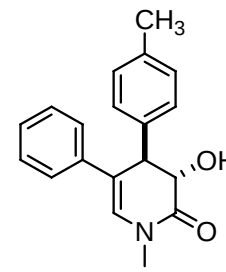
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 Time 19.39
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 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1230
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 2298.8
 DW 27.800 usec
 DE 6.00 usec
 TE 300.7 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.40 usec
 PL1 -1.00 dB
 SF01 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 18.00 dB
 PL13 18.00 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 22.00 cm
 CY 30.00 cm
 F1P 190.000 ppm
 F1 14338.87 Hz
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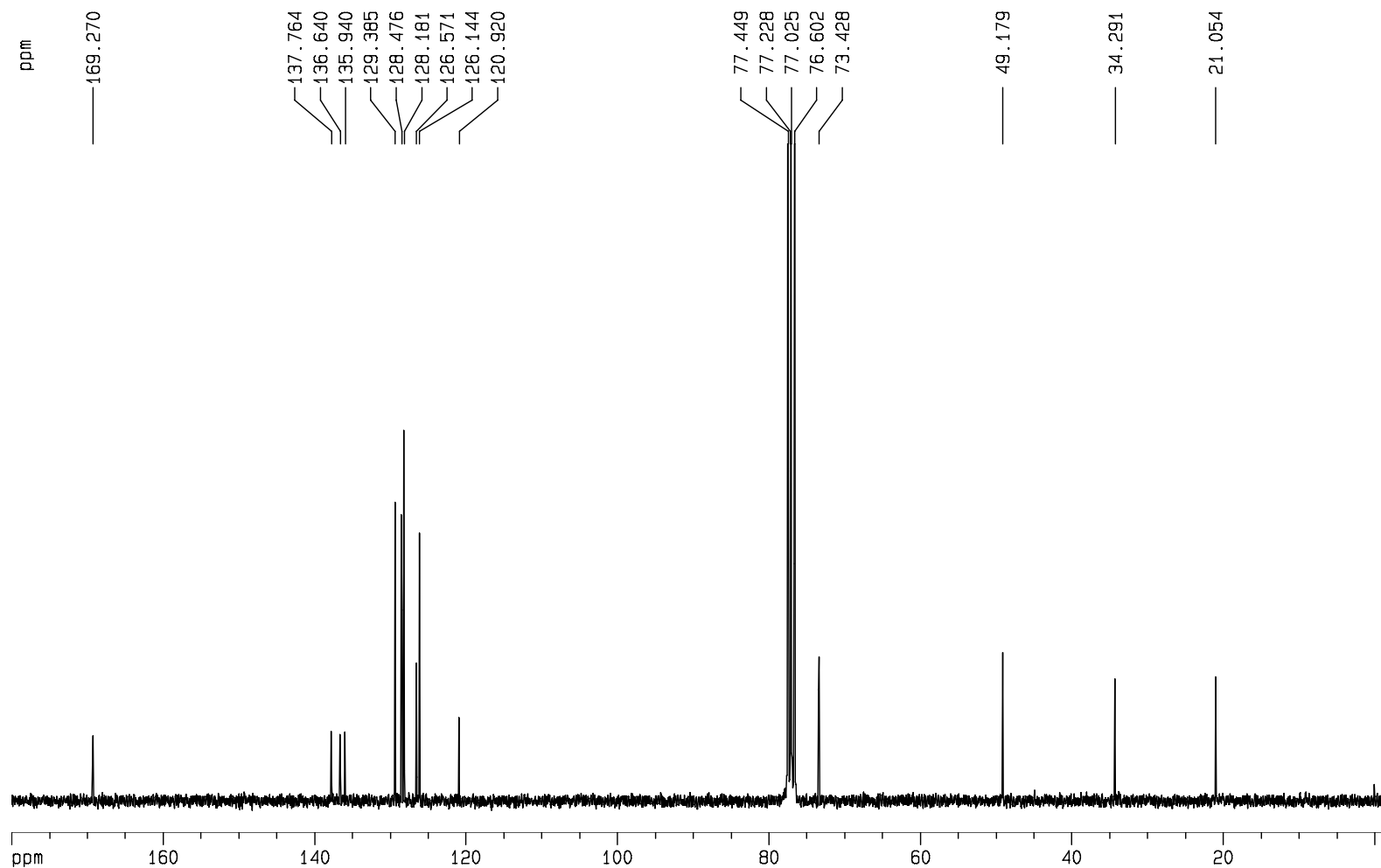
Current Data Parameters
NAME y1019-4
EXPNO 51
PROCNO 1

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PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 322.5
DW 55.600 usec
DE 6.00 usec
TE 300.7 K
D1 5.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300078 MHz
WDW EM
SSB 0
LB 0.35 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 10.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.41818 ppm/cm
HZCM 125.50892 Hz/cm



Current Data Parameters

NAME y1019-4
EXPNO 52
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070814
Time 12.14
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2043
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 2580.3
DW 27.800 usec
DE 6.00 usec
TE 300.7 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 14.20 usec
PL1 -5.00 dB
SF01 75.4752953 MHz

===== CHANNEL f2 =====

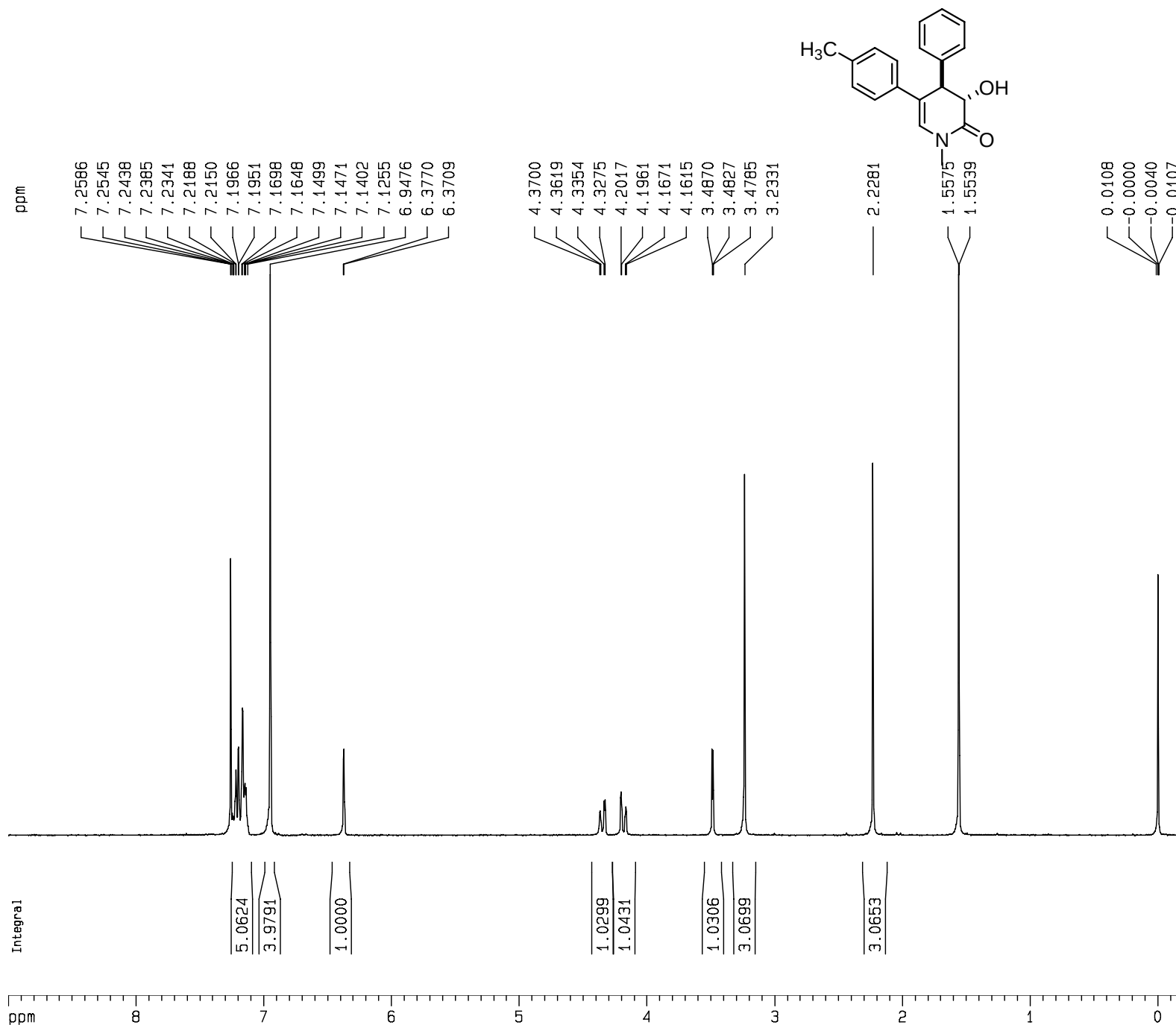
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 16.56 dB
PL13 18.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters

SI 32768
SF 75.4677484 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 22.00 cm
CY 15.00 cm
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F2P -2.000 ppm
F2 -150.94 Hz
PPMCM 8.27273 ppm/cm
HZCM 624.32410 Hz/cm



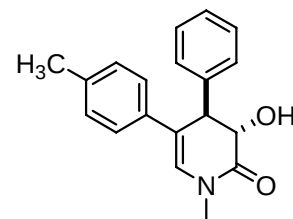
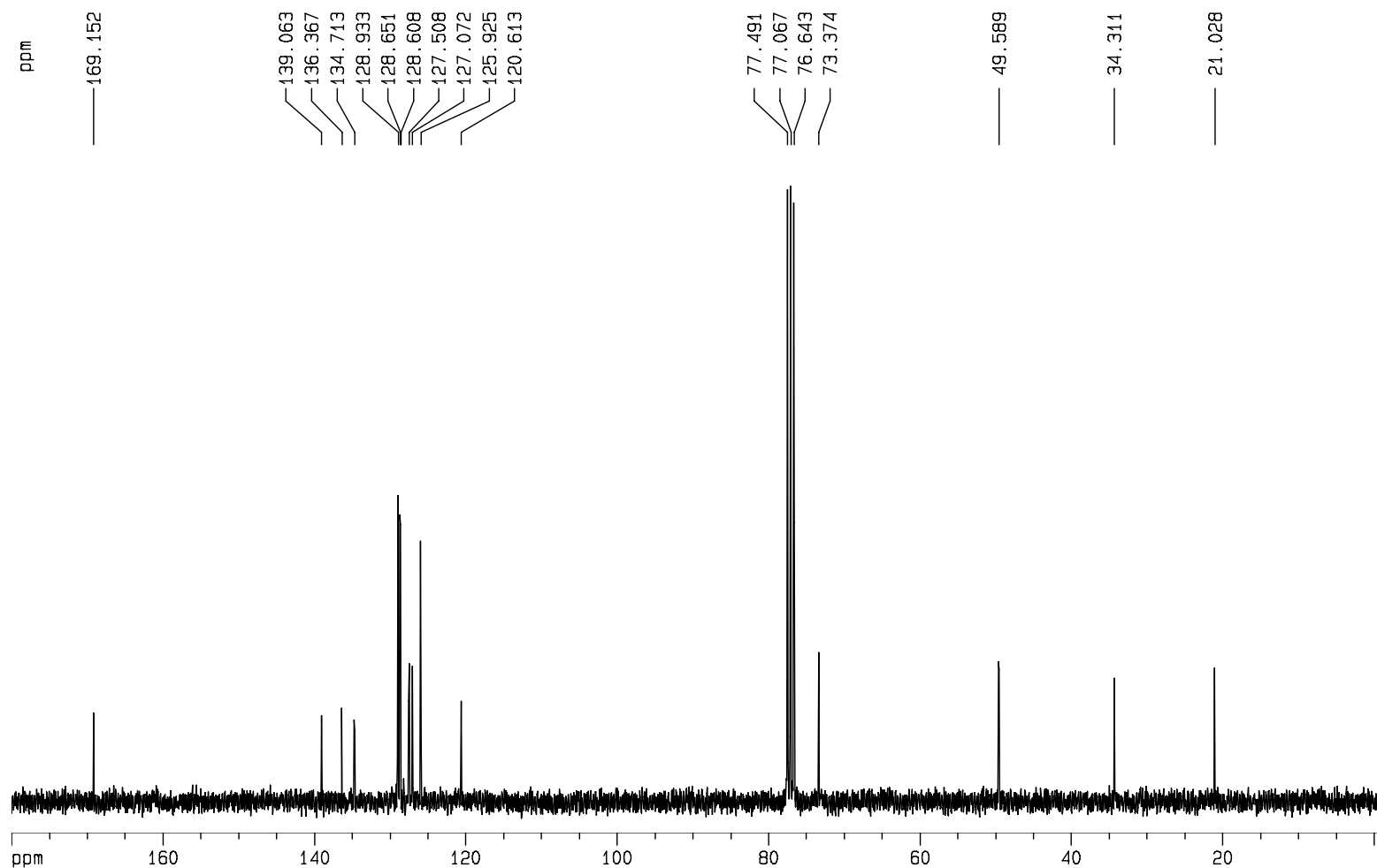
Current Data Parameters
NAME y1021-4-1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
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Time 18.24
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 574.7
DW 55.600 usec
DE 6.00 usec
TE 299.9 K
D1 5.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.60 usec
PL1 -1.00 dB
SFO1 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.130069 MHz
WDW EM
SSB 0
LB 0.35 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 12.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.41818 ppm/cm
HZCM 125.50892 Hz/cm



Current Data Parameters
 NAME y1021-4
 EXPNO 21
 PROCNO 1

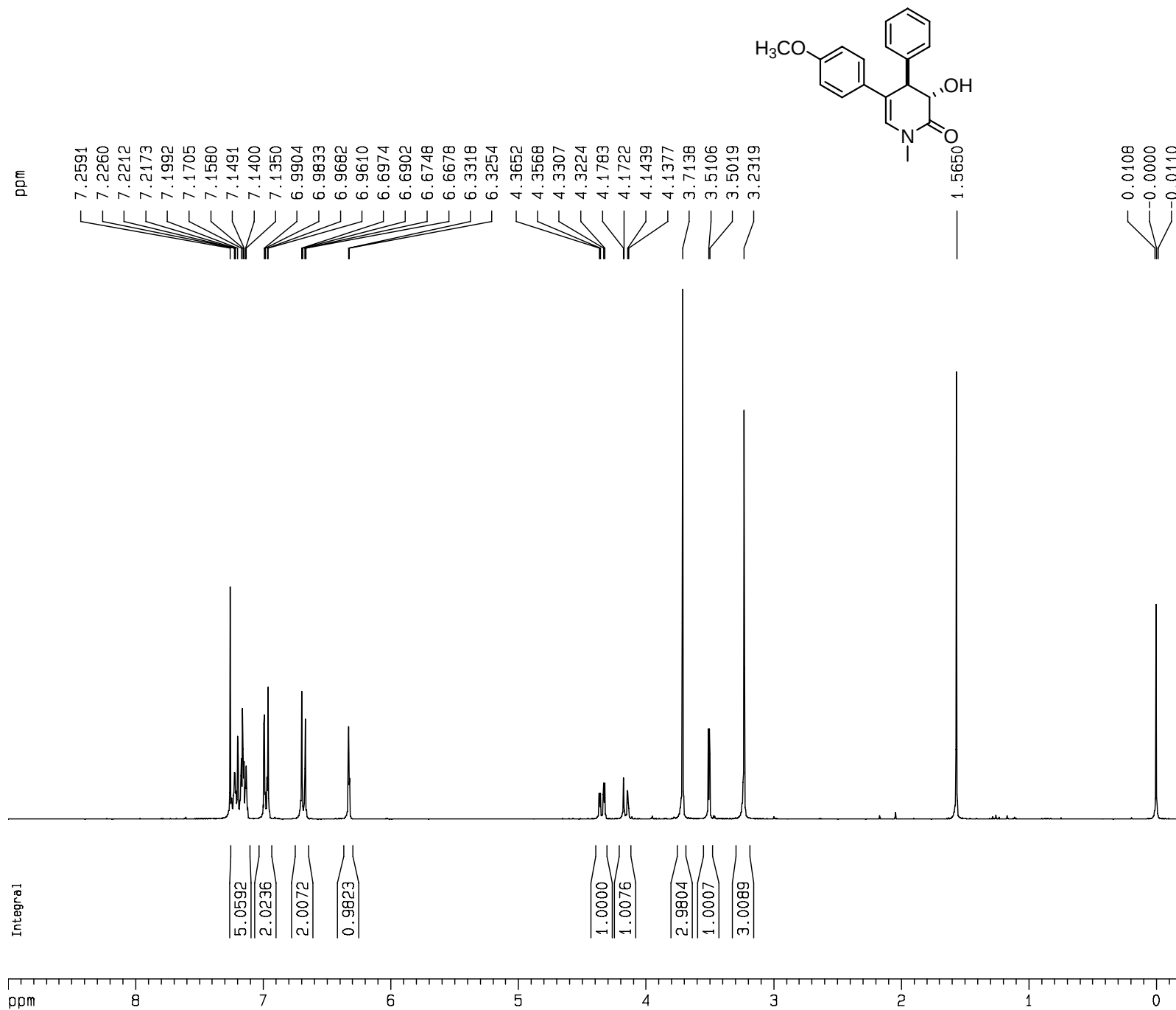
F2 - Acquisition Parameters
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 Time 11.35
 INSTRUM av300
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 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 400
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 8192
 DW 27.800 usec
 DE 6.00 usec
 TE 300.8 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 14.20 usec
 PL1 -5.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 16.56 dB
 PL13 18.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 22.00 cm
 CY 10.00 cm
 F1P 180.000 ppm
 F1 13584.20 Hz
 F2P -2.000 ppm
 F2 -150.94 Hz
 PPMCM 8.27273 ppm/cm
 HZCM 624.32410 Hz/cm



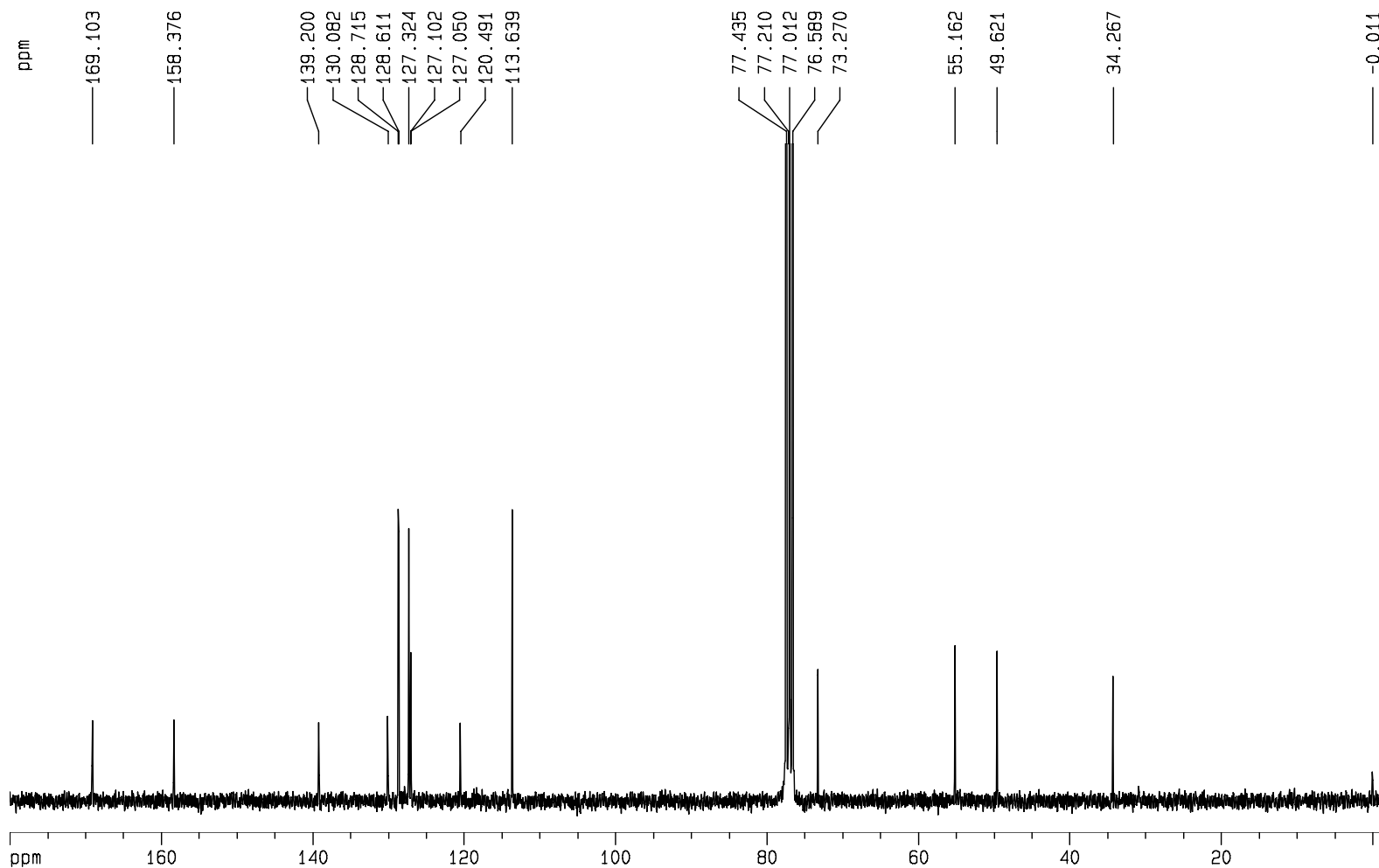
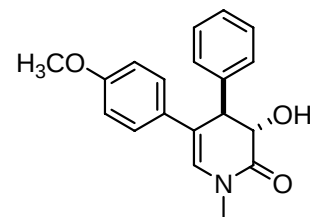
Current Data Parameters
 NAME y1003-4-1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070802
 Time 11.16
 INSTRUM av300
 PROBHD 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 8992.806 Hz
 FIDRES 0.137219 Hz
 AQ 3.6438515 sec
 RG 574.7
 DW 55.600 usec
 DE 6.00 usec
 TE 299.5 K
 D1 5.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.60 usec
 PL1 -1.00 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300066 MHz
 WDW EM
 SSB 0
 LB 0.35 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 10.00 cm
 F1P 9.000 ppm
 F1 2701.17 Hz
 F2P -0.200 ppm
 F2 -60.03 Hz
 PPMCM 0.41818 ppm/cm
 HZCM 125.50892 Hz/cm



Current Data Parameters

NAME y1003-4-1
EXPNO 70
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070809
Time 11.07
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3915
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 3251
DW 27.800 usec
DE 6.00 usec
TE 300.6 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 14.20 usec
PL1 -5.00 dB
SF01 75.4752953 MHz

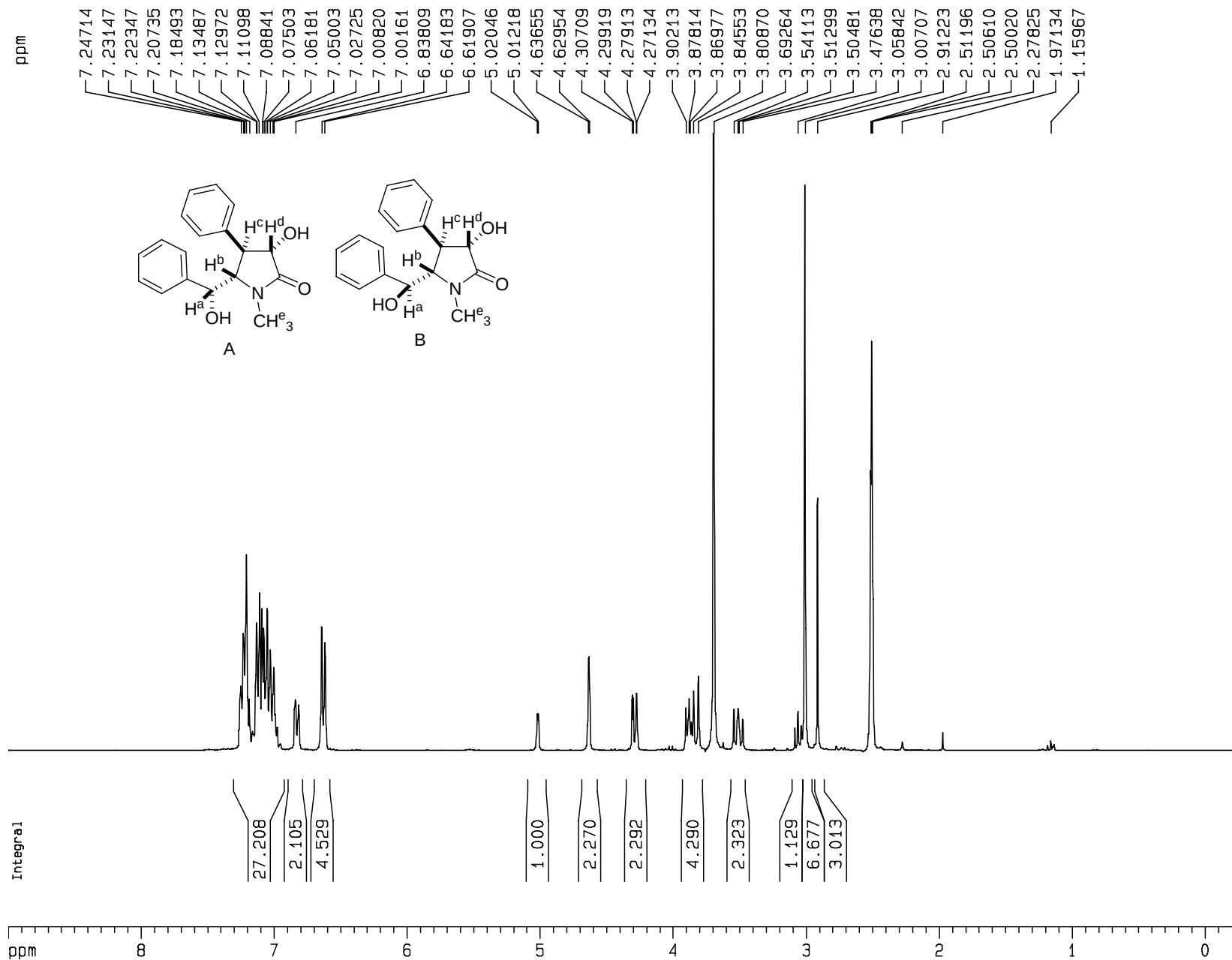
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 16.56 dB
PL13 18.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters

SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 22.00 cm
CY 30.00 cm
F1P 180.000 ppm
F1 13584.20 Hz
F2P -2.000 ppm
F2 -150.94 Hz
PPMCM 8.27273 ppm/cm
HZCM 624.32410 Hz/cm



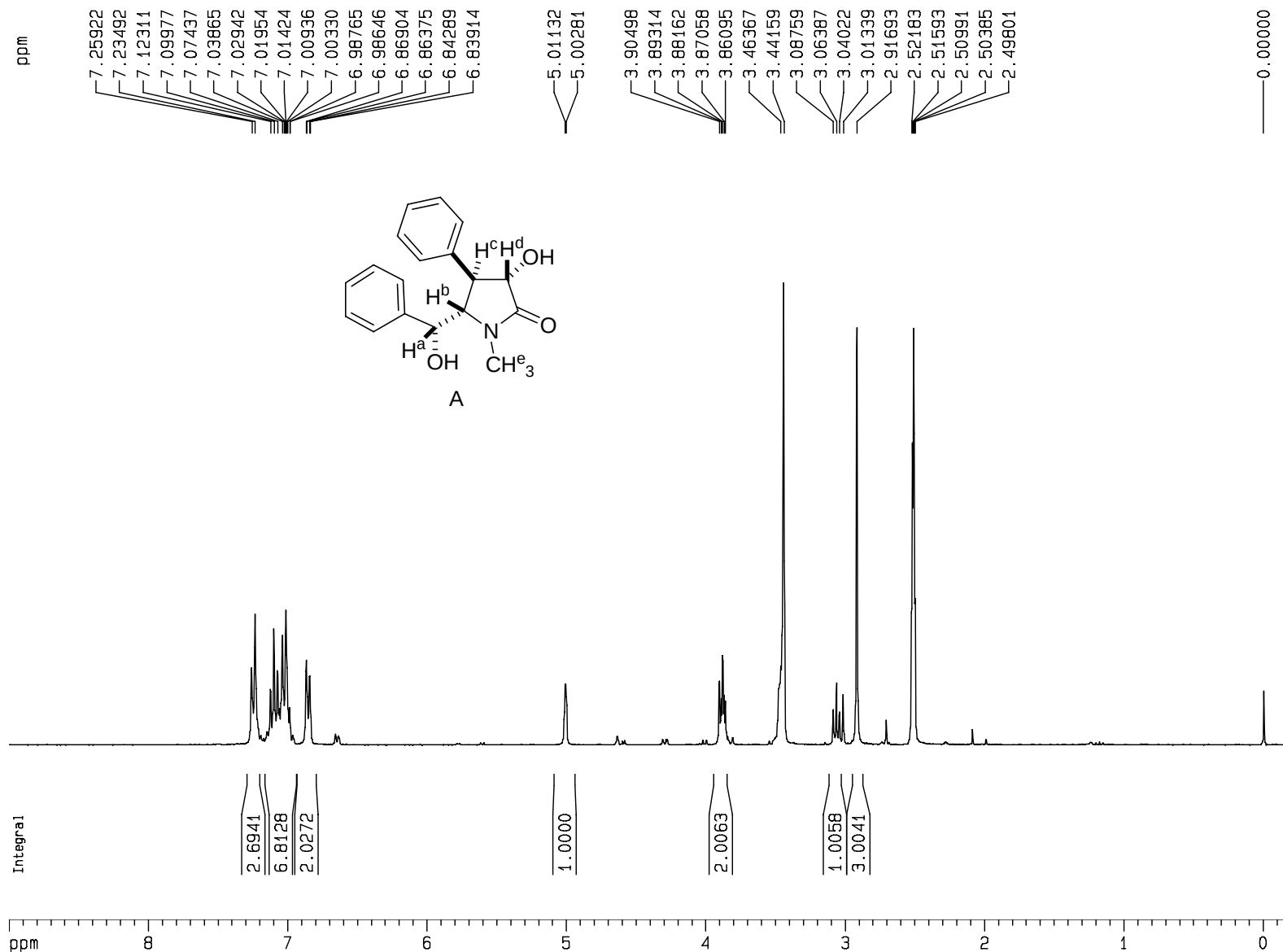
Current Data Parameters
 NAME yl052
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060824
 Time 8.37
 INSTRUM av300
 PROBHD 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 228.1
 DW 81.000 usec
 DE 6.00 usec
 TE 299.5 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.30 usec
 PL1 -1.00 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 20.00 cm
 F1P 9.000 ppm
 F1 2701.17 Hz
 F2P -0.200 ppm
 F2 -60.03 Hz
 PPMCM 0.41818 ppm/cm
 HZCM 125.50892 Hz/cm



Current Data Parameters
 NAME y1052-2
 EXPNO 2
 PROCNO 1

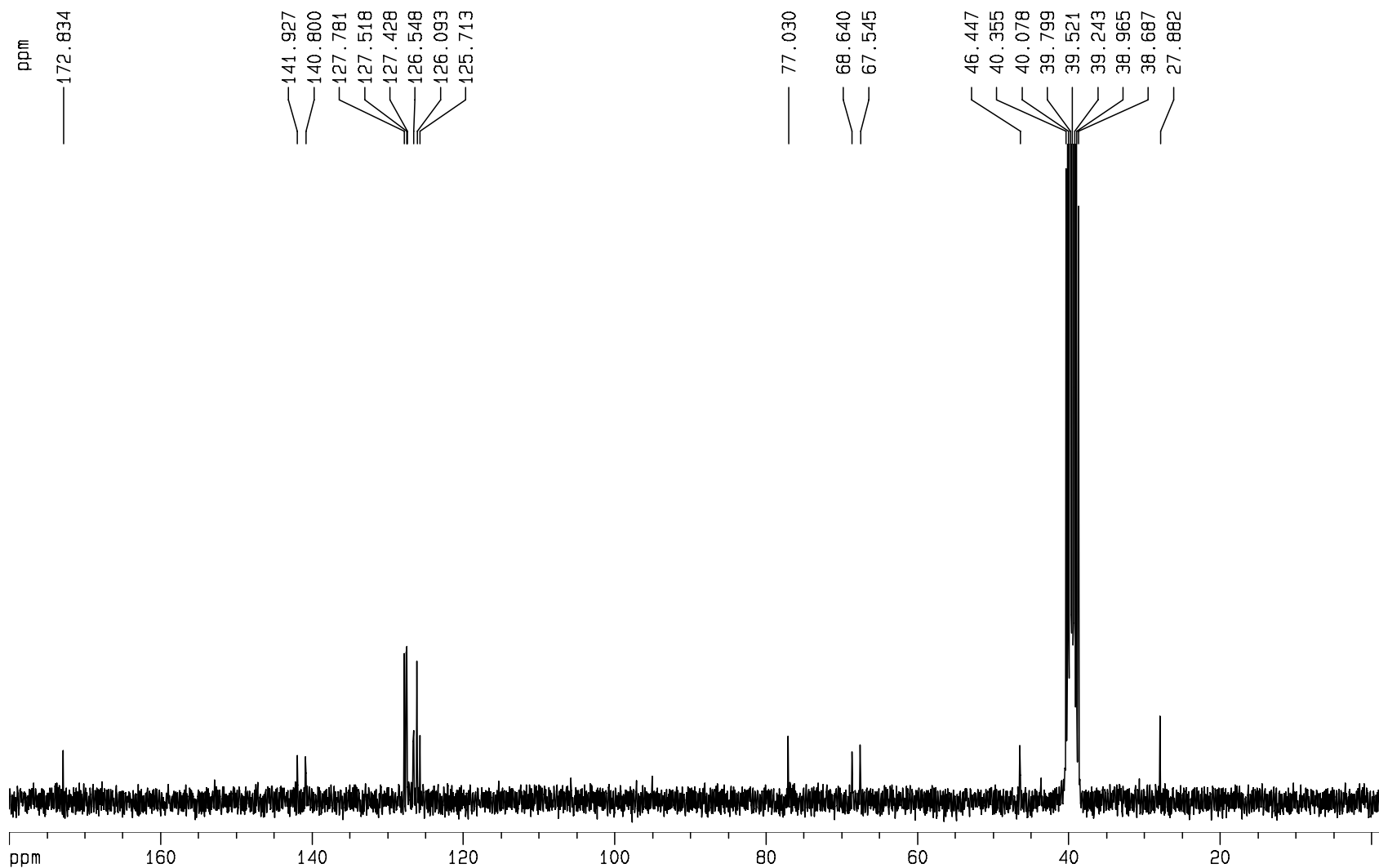
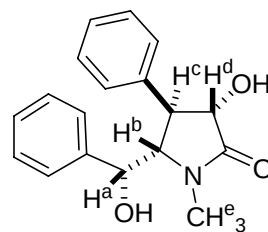
F2 - Acquisition Parameters
 Date_ 20070910
 Time 7.47
 INSTRUM av300
 PROBHD 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 32
 DS 0
 SWH 8992.806 Hz
 FIDRES 0.137219 Hz
 AQ 3.6438515 sec
 RG 574.7
 DW 55.600 usec
 DE 6.00 usec
 TE 300.8 K
 D1 5.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.60 usec
 PL1 -1.00 dB
 SFO1 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1299985 MHz
 WDW EM
 SSB 0
 LB 0.35 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 8.00 cm
 F1P 9.000 ppm
 F1 2701.17 Hz
 F2P -0.200 ppm
 F2 -60.03 Hz
 PPMCM 0.41818 ppm/cm
 HZCM 125.50892 Hz/cm

052-2 DMSO



Current Data Parameters
NAME y1052-2
EXPNO 50
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070515
Time 12.26
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1524
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1824.6
DW 27.800 usec
DE 6.00 usec
TE 301.2 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

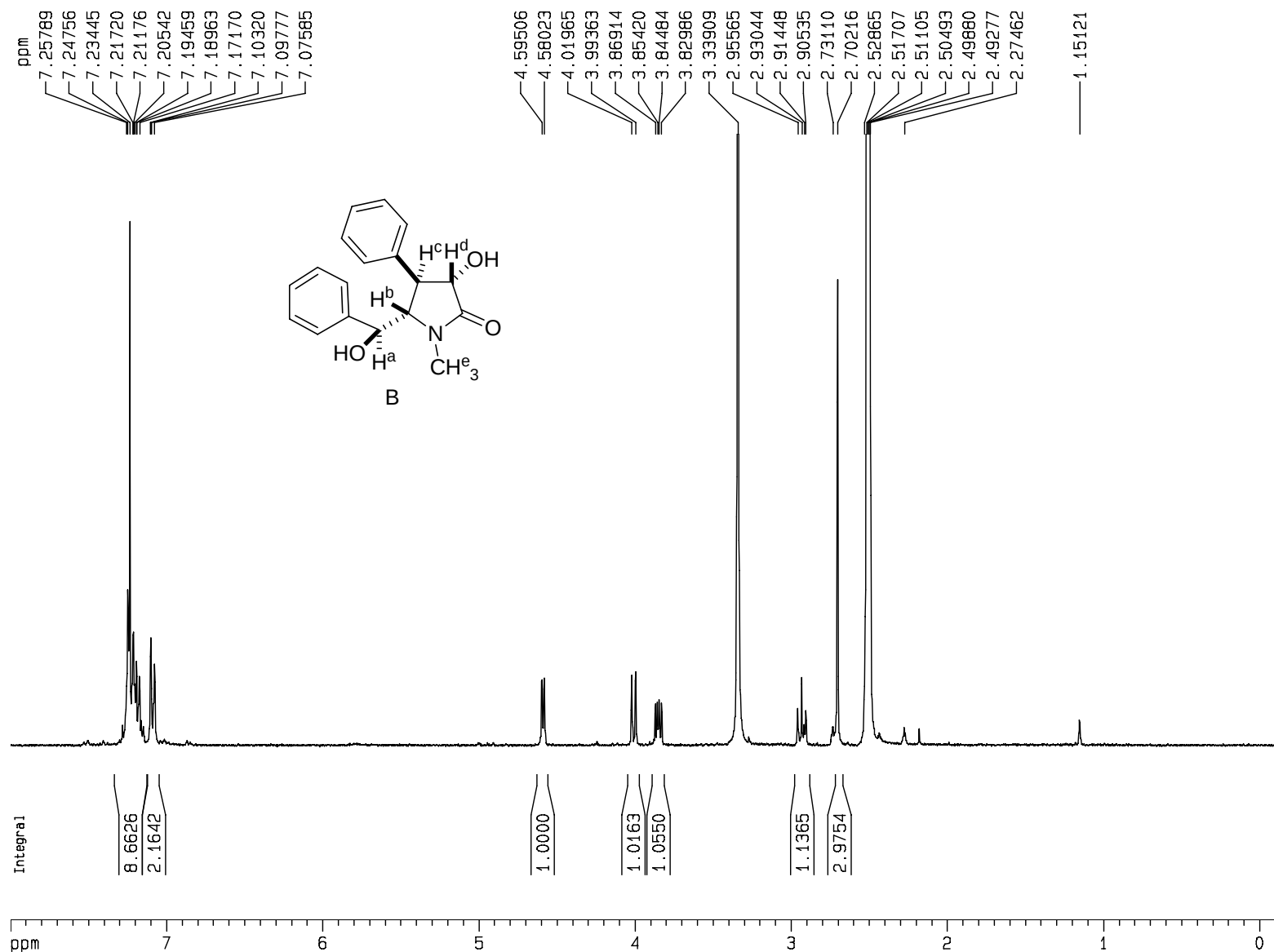
===== CHANNEL f1 =====
NUC1 13C
P1 14.20 usec
PL1 -5.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 16.56 dB
PL13 18.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 22.00 cm
CY 70.00 cm
F1P 180.000 ppm
F1 13584.20 Hz
F2P -2.000 ppm
F2 150.94 Hz
PPMCM 8.27273 ppm/cm
HZCM 624.32446 Hz/cm

052-3 DMSO



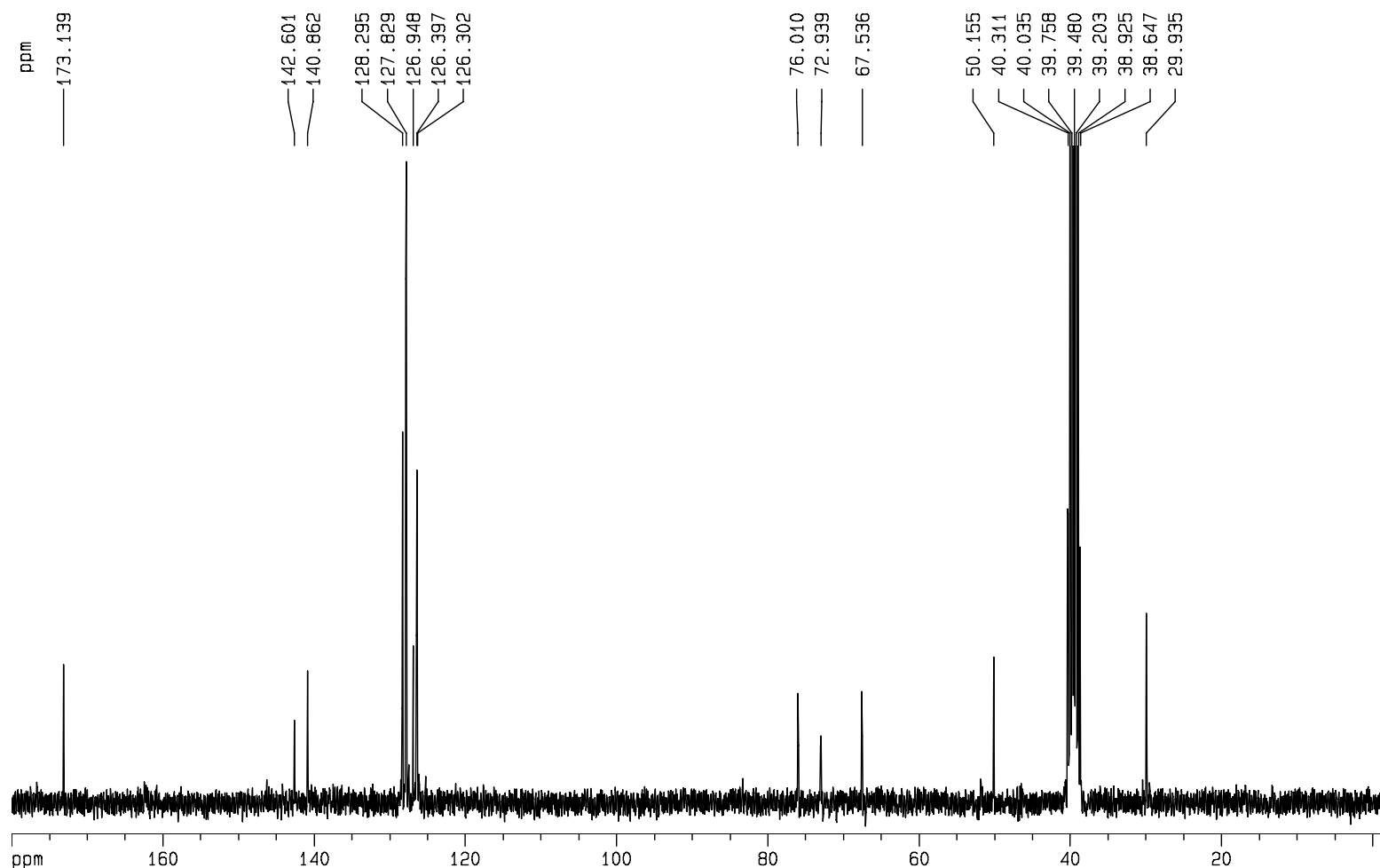
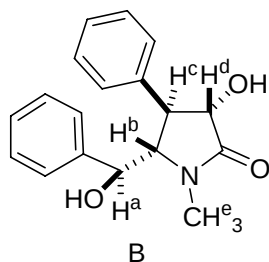
Current Data Parameters
 NAME y1052-3
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20060831
 Time 12.16
 INSTRUM av300
 PROBHD 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 6172.839 Hz
 FIDRES 0.094190 Hz
 AQ 5.3084660 sec
 RG 512
 DW 81.000 usec
 DE 6.00 usec
 TE 301.2 K
 D1 2.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 9.30 usec
 PL1 -1.00 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 50.00 cm
 F1P 8.000 ppm
 F1 2401.04 Hz
 F2P -0.200 ppm
 F2 -60.03 Hz
 PPMCM 0.37273 ppm/cm
 HZCM 111.86664 Hz/cm



Current Data Parameters
 NAME y1052-3
 EXPNO 20
 PROCNO 1

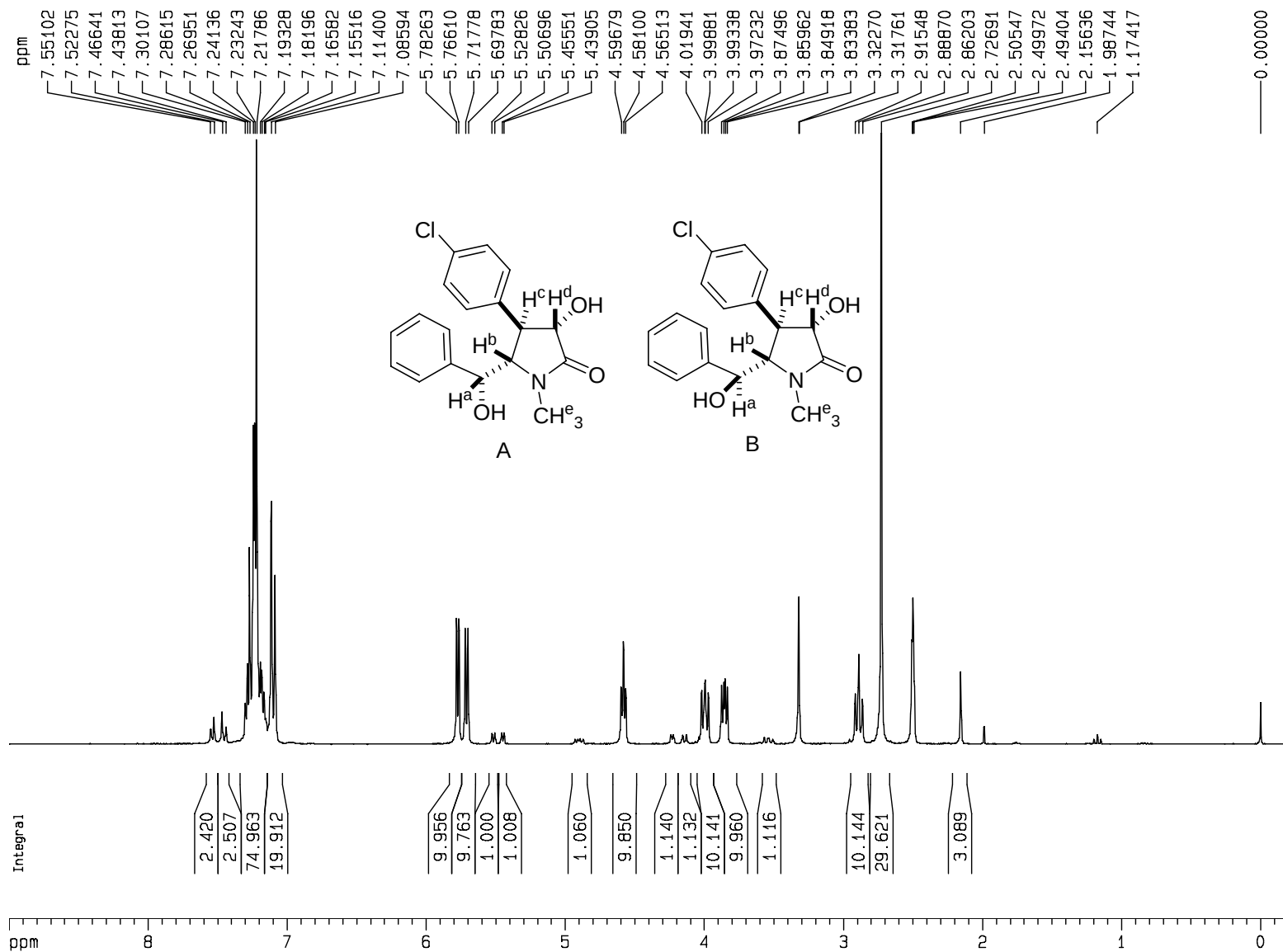
F2 - Acquisition Parameters
 Date_ 20070104
 Time 9.35
 INSTRUM av300
 PROBHD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 427
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 7298.2
 DW 27.800 usec
 DE 6.00 usec
 TE 294.7 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.40 usec
 PL1 -1.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 18.00 dB
 PL13 18.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677867 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 22.00 cm
 CY 30.00 cm
 F1P 180.000 ppm
 F1 13584.20 Hz
 F2P -2.000 ppm
 F2 -150.94 Hz
 PPMCM 8.27273 ppm/cm
 HZCM 624.32446 Hz/cm



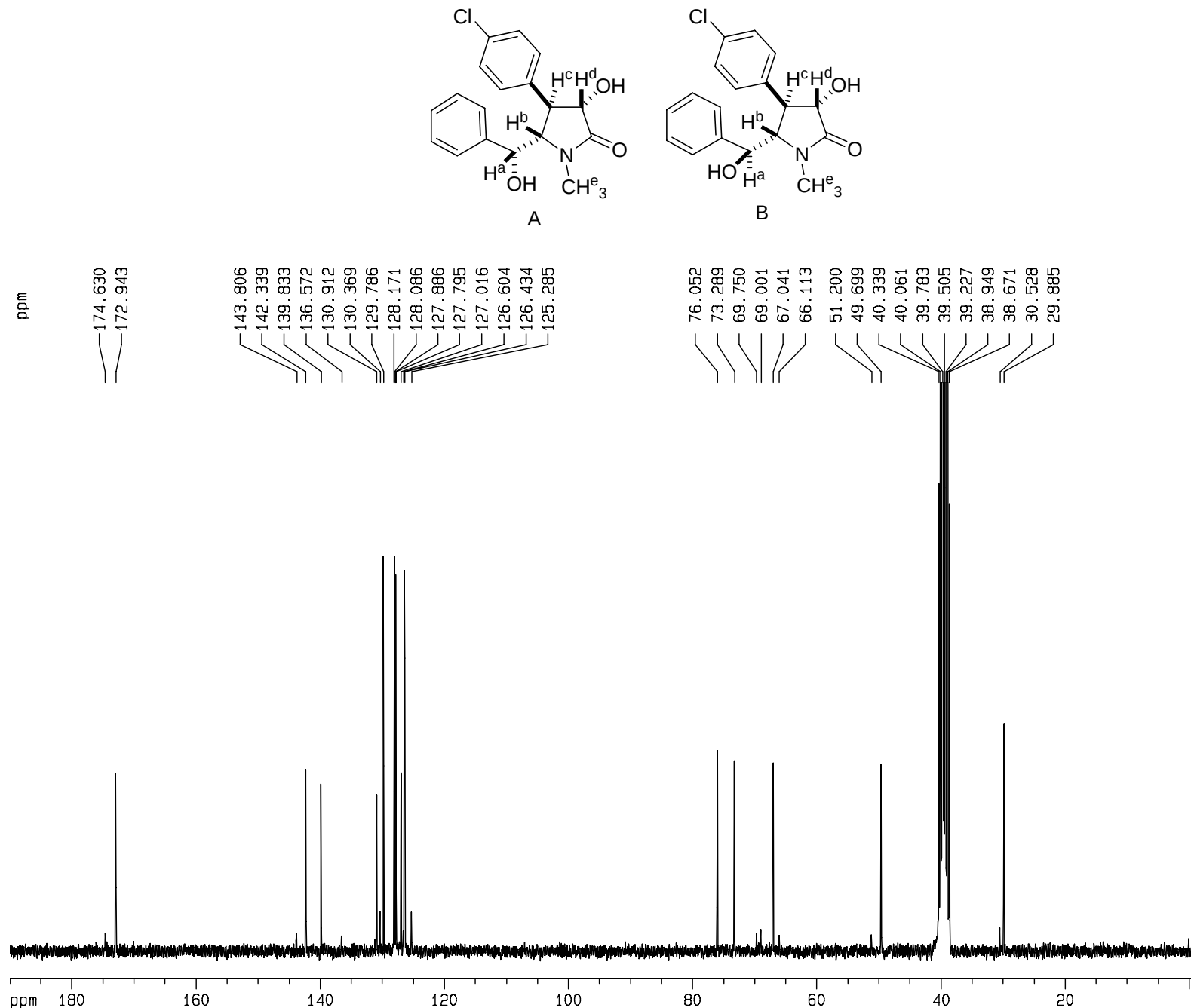
Current Data Parameters
 NAME y1011-3
 EXPNO 60
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070806
 Time 18.48
 INSTRUM av300
 PROBHD 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 0
 SWH 8992.806 Hz
 FIDRES 0.137219 Hz
 AQ 3.6438515 sec
 RG 256
 DW 55.600 usec
 DE 6.00 usec
 TE 299.9 K
 D1 5.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.60 usec
 PL1 -1.00 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300015 MHz
 WDW EM
 SSB 0
 LB 0.35 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 12.00 cm
 F1P 9.000 ppm
 F1 2701.17 Hz
 F2P -0.200 ppm
 F2 -60.03 Hz
 PPMCM 0.41818 ppm/cm
 HZCM 125.50892 Hz/cm



Current Data Parameters
 NAME y1011-3
 EXPNO 80
 PROCNO 1

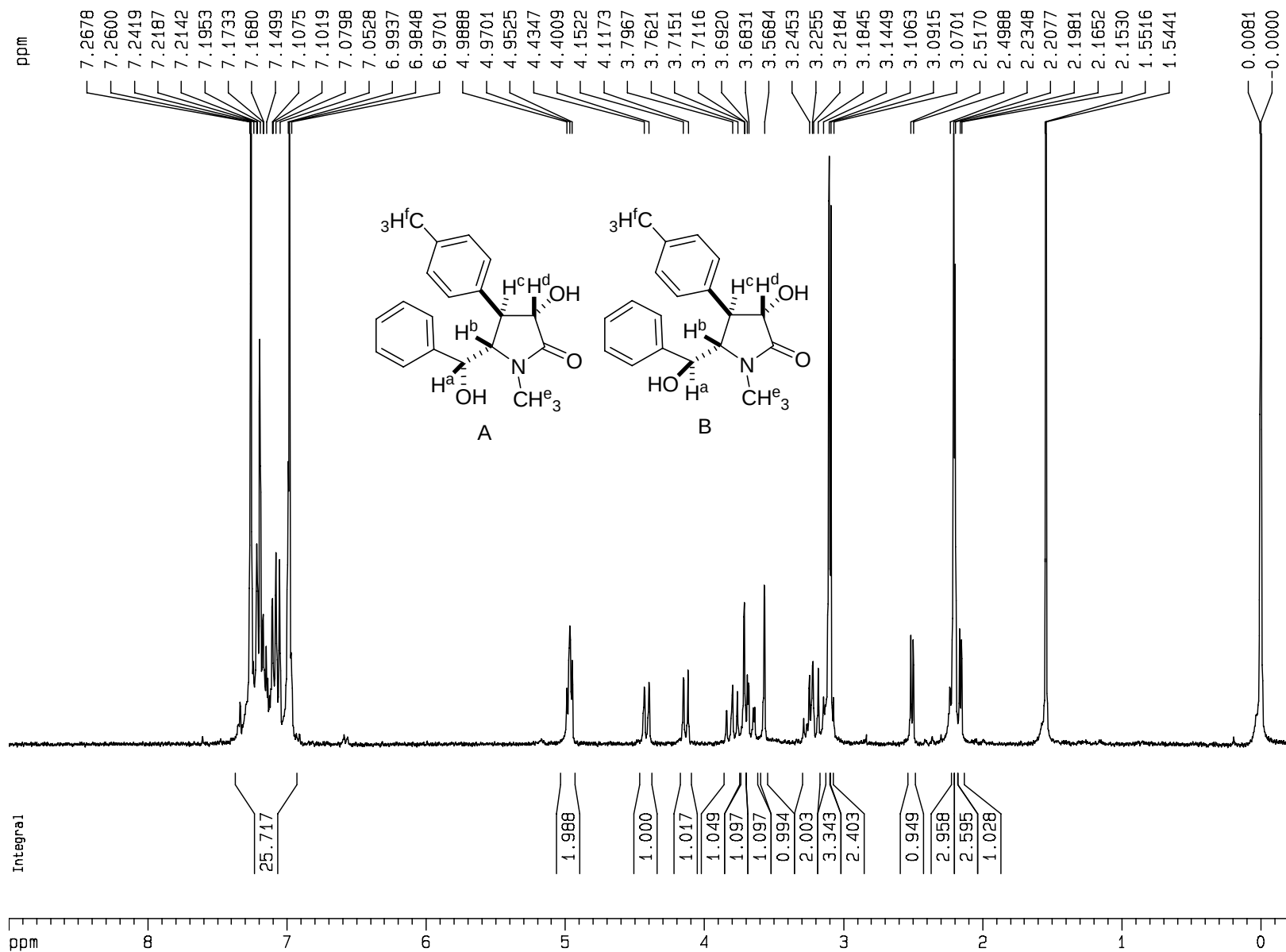
F2 - Acquisition Parameters
 Date_ 20070809
 Time 8.42
 INSTRUM av300
 PROBHD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 9287
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 912.3
 DW 27.800 usec
 DE 6.00 usec
 TE 300.3 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 14.20 usec
 PL1 -5.00 dB
 SFO1 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 16.56 dB
 PL13 18.00 dB
 SFO2 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677867 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 22.00 cm
 CY 60.00 cm
 F1P 190.000 ppm
 F1 14338.88 Hz
 F2P -2.000 ppm
 F2 -150.94 Hz
 PPMCM 8.72727 ppm/cm
 HZCM 658.62793 Hz/cm



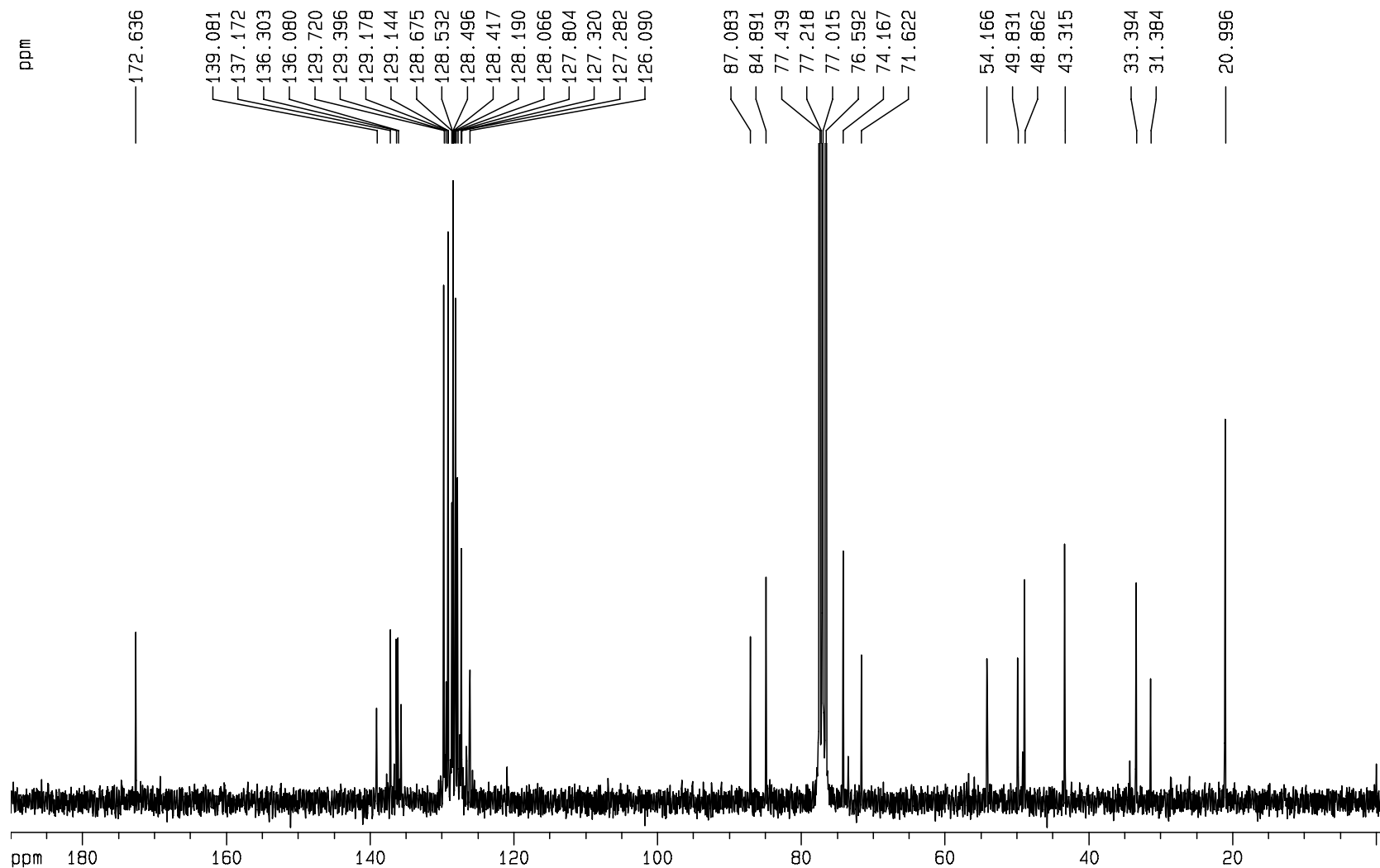
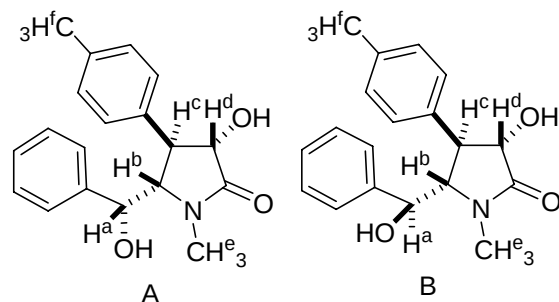
Current Data Parameters
NAME y1019-3
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070330
Time 12.12
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 30
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 574.7
DW 55.600 usec
DE 6.00 usec
TE 296.9 K
D1 5.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 9.30 usec
PL1 -1.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.35 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 30.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.41818 ppm/cm
HZCM 125.50892 Hz/cm



Current Data Parameters
NAME y1019-3
EXPNO 20
PROCNO 1

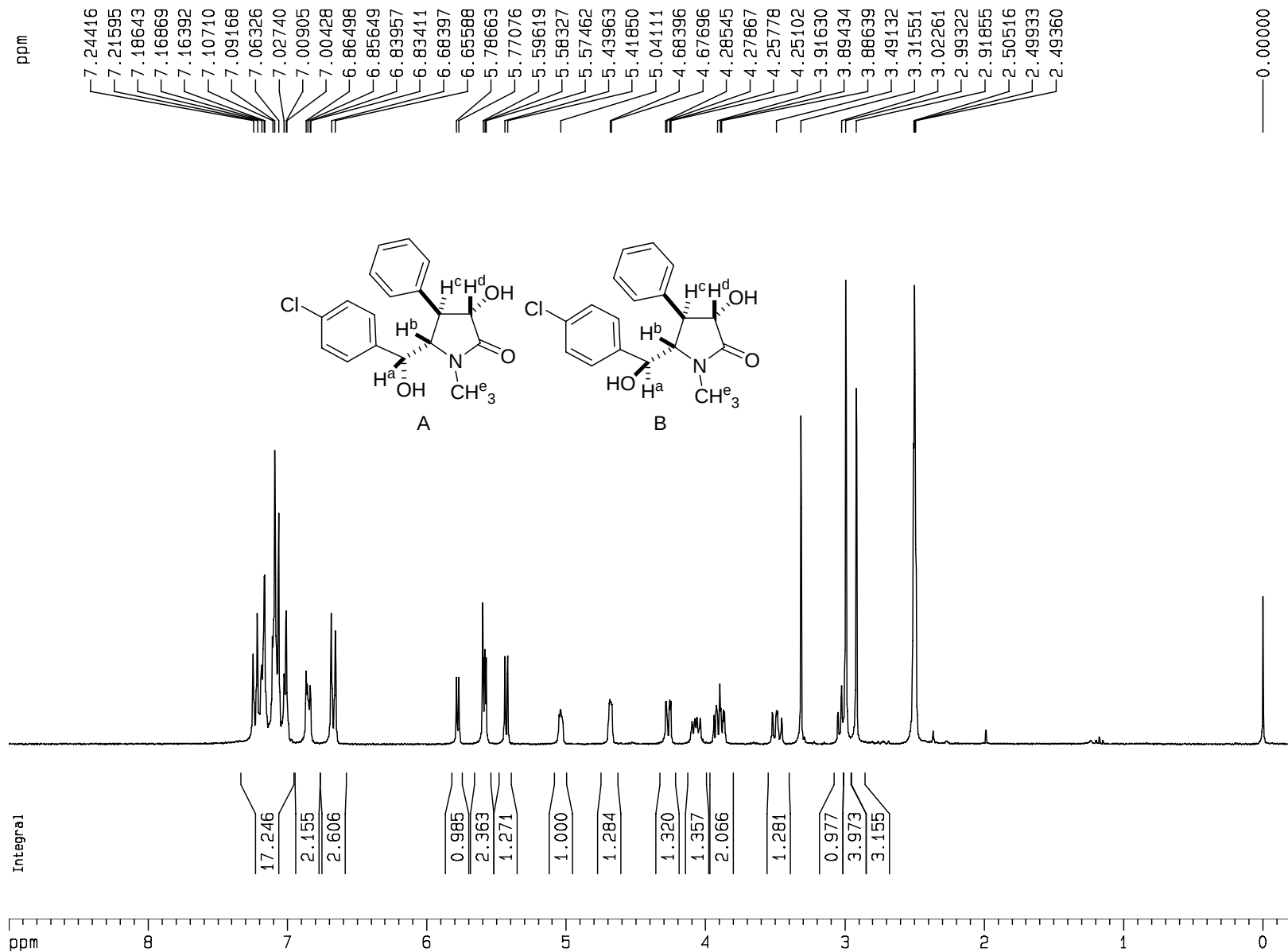
F2 - Acquisition Parameters
Date_ 20070407
Time 12.15
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 2607
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 724.1
DW 27.800 usec
DE 6.00 usec
TE 298.6 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.40 usec
PL1 -1.00 dB
SF01 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 22.00 cm
CY 50.00 cm
F1P 190.000 ppm
F1 14338.87 Hz
F2P -2.000 ppm
F2 3150.94 Hz
PPMCM 8.72727 ppm/cm
HZCM 658.62756 Hz/cm



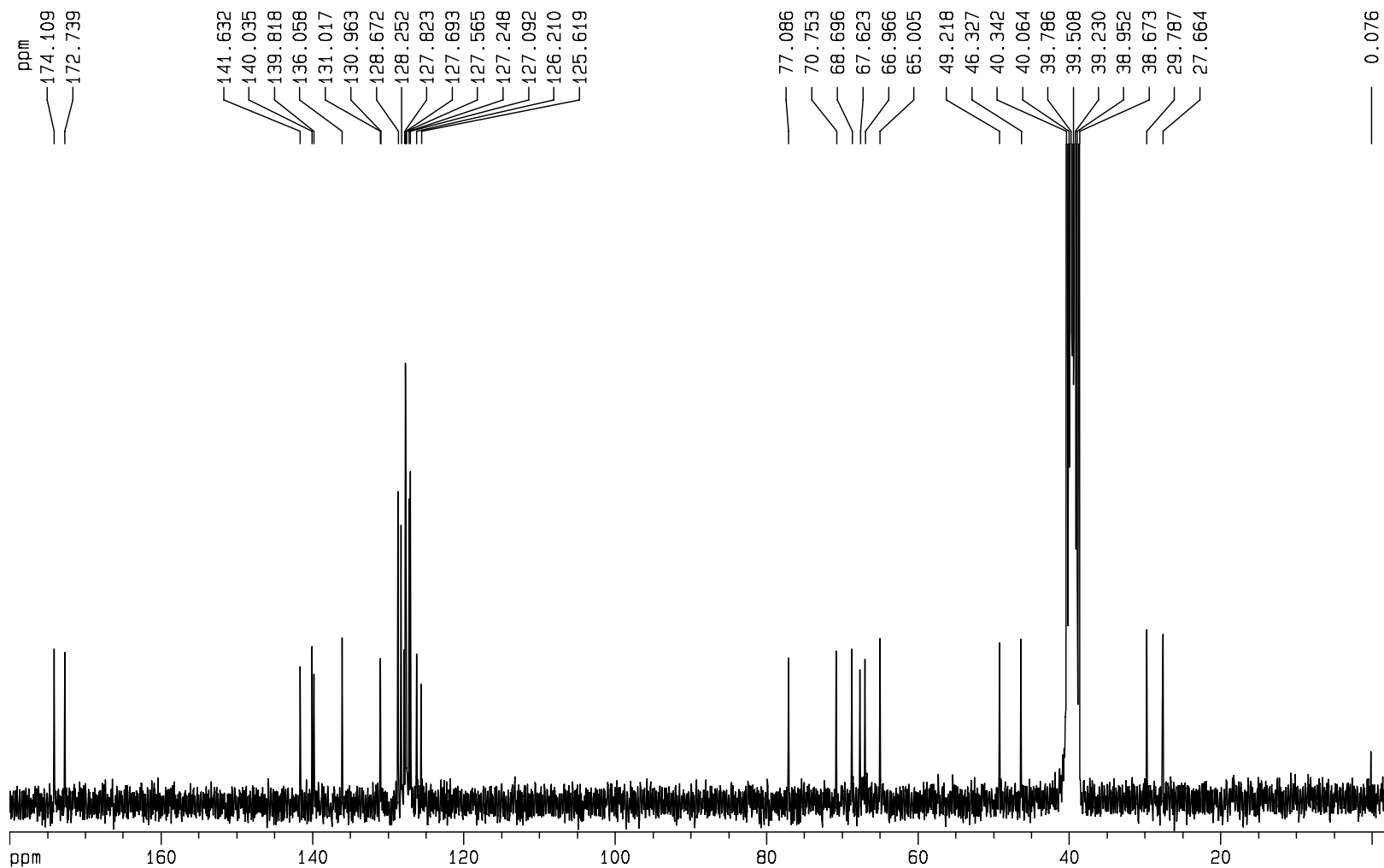
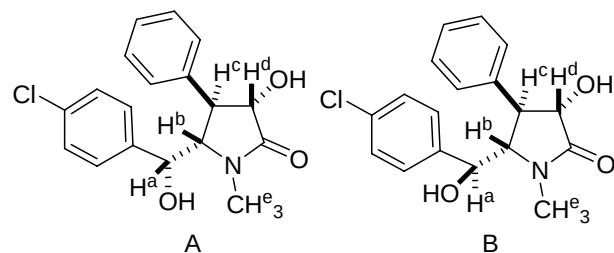
Current Data Parameters
NAME y1013-3
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070904
Time 23.55
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 574.7
DW 55.600 usec
DE 6.00 usec
TE 298.9 K
D1 5.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.60 usec
PL1 -1.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300014 MHz
WDW EM
SSB 0
LB 0.35 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 8.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.41818 ppm/cm
HZCM 125.50892 Hz/cm



Current Data Parameters

NAME y1013-3'
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters

Date_ 20070905
Time 7.55
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 7510
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 1024
DW 27.800 usec
DE 6.00 usec
TE 299.5 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====

NUC1 13C
P1 12.20 usec
PL1 -0.32 dB
SF01 75.4752953 MHz

===== CHANNEL f2 =====

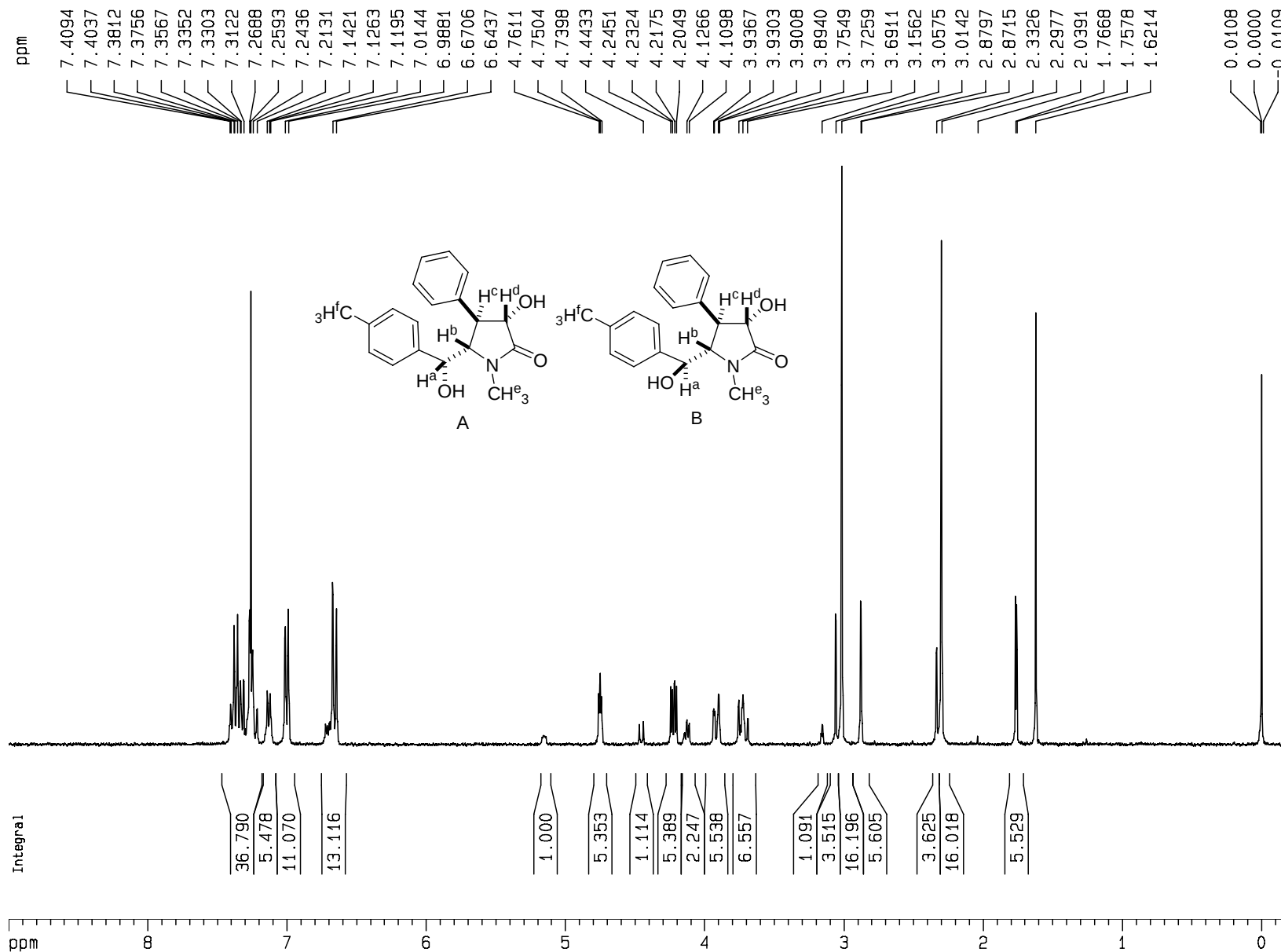
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 16.56 dB
PL13 18.00 dB
SF02 300.1312005 MHz

F2 - Processing parameters

SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 22.00 cm
CY 200.00 cm
F1P 180.000 ppm
F1 13584.20 Hz
F2P -2.000 ppm
F2 -150.94 Hz
PPMCM 8.27273 ppm/cm
HZCM 624.32446 Hz/cm



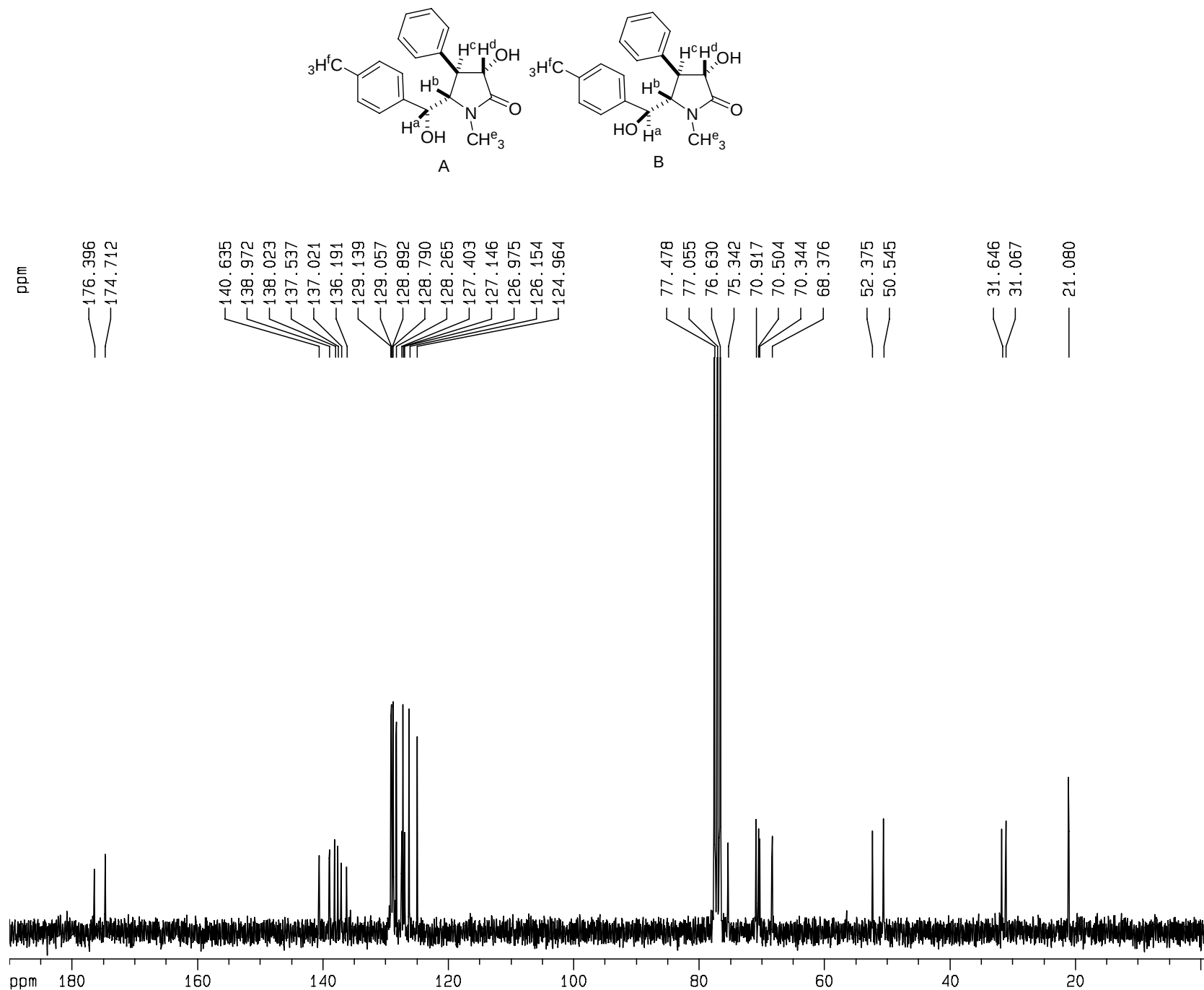
Current Data Parameters
 NAME y1021-3
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20070907
 Time 2.12
 INSTRUM av300
 PROBHD 5 mm DUL 13C-1
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 1
 DS 0
 SWH 8992.806 Hz
 FIDRES 0.137219 Hz
 AQ 3.6438515 sec
 RG 322.5
 DW 55.600 usec
 DE 6.00 usec
 TE 300.3 K
 D1 5.00000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.60 usec
 PL1 -1.00 dB
 SF01 300.1318534 MHz

F2 - Processing parameters
 SI 32768
 SF 300.1300065 MHz
 WDW EM
 SSB 0
 LB 0.35 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 22.00 cm
 CY 10.00 cm
 F1P 9.000 ppm
 F1 2701.17 Hz
 F2P -0.200 ppm
 F2 -60.03 Hz
 PPMCM 0.41818 ppm/cm
 HZCM 125.50892 Hz/cm



Current Data Parameters
 NAME y1021-3
 EXPNO 30
 PROCNO 1

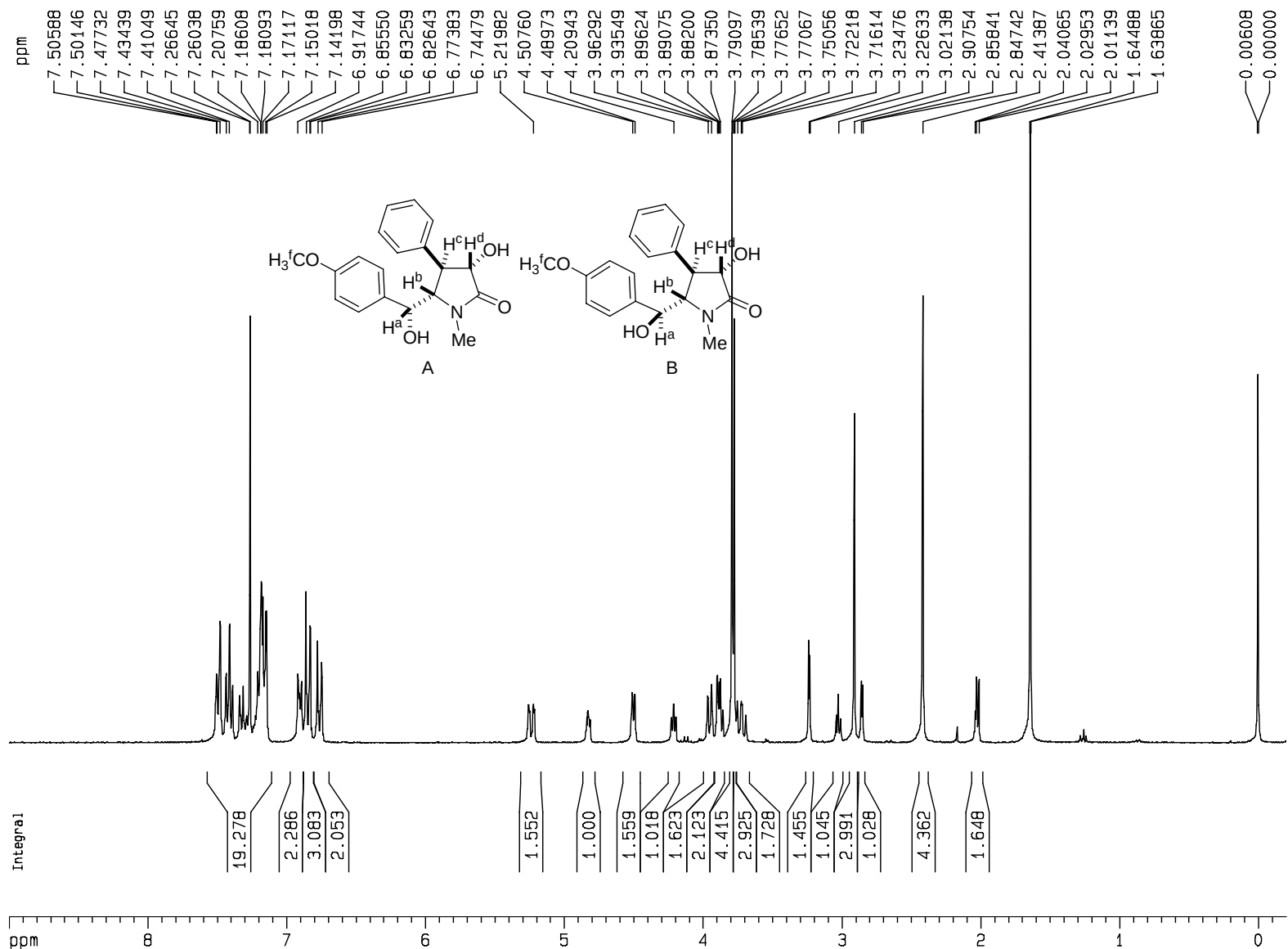
F2 - Acquisition Parameters
 Date_ 20070526
 Time 12.17
 INSTRUM av300
 PROBHD 5 mm DUL 13C-1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 611
 DS 4
 SWH 17985.611 Hz
 FIDRES 0.274439 Hz
 AQ 1.8219508 sec
 RG 16384
 DW 27.800 usec
 DE 6.00 usec
 TE 298.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 14.20 usec
 PL1 -5.00 dB
 SF01 75.4752953 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 16.56 dB
 PL13 18.00 dB
 SF02 300.1312005 MHz

F2 - Processing parameters
 SI 32768
 SF 75.4677490 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 22.00 cm
 CY 20.00 cm
 F1P 190.000 ppm
 F1 14338.87 Hz
 F2P -2.000 ppm
 F2 3190.94 Hz
 PPMCM 8.72727 ppm/cm
 HZCM 658.62756 Hz/cm



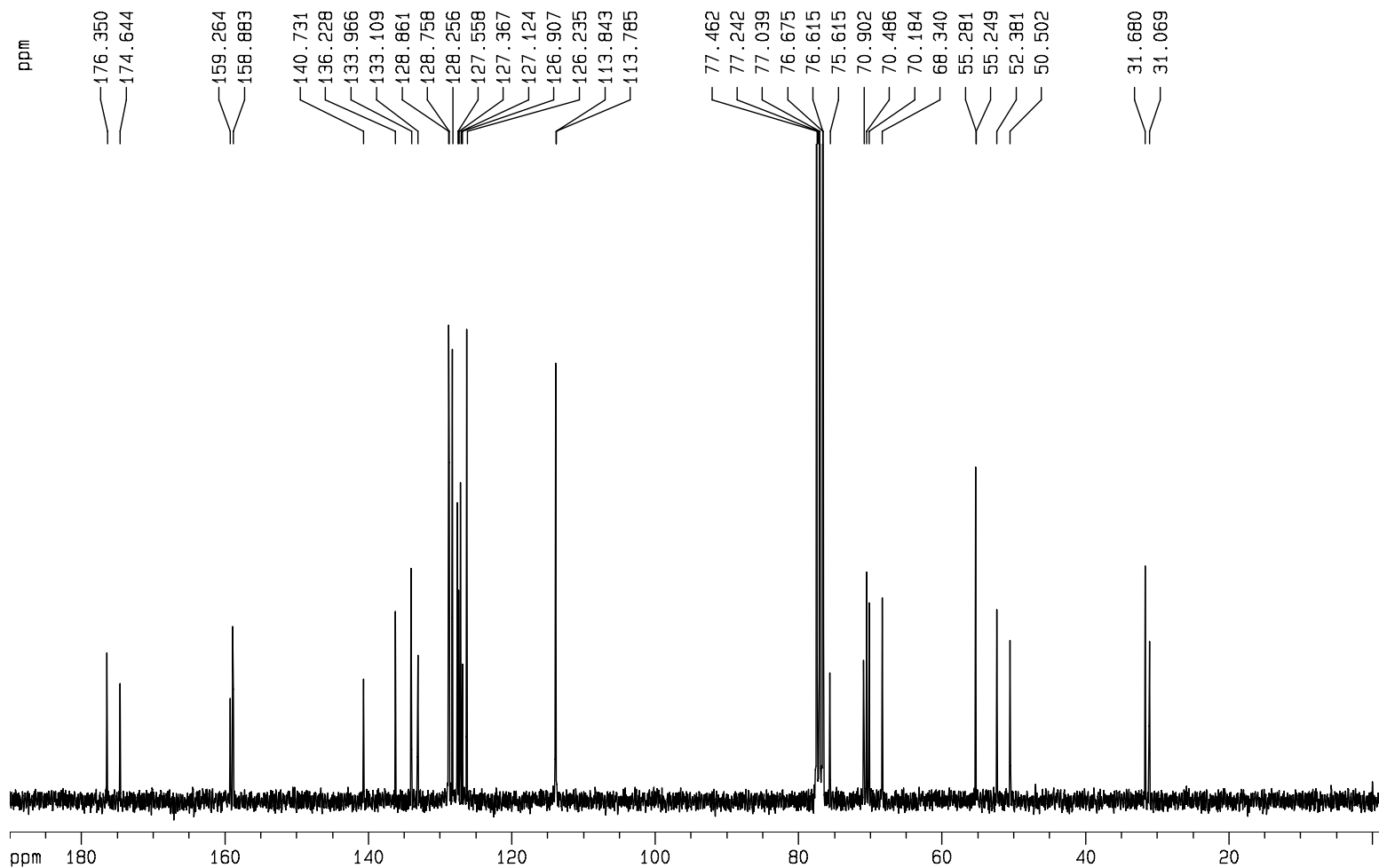
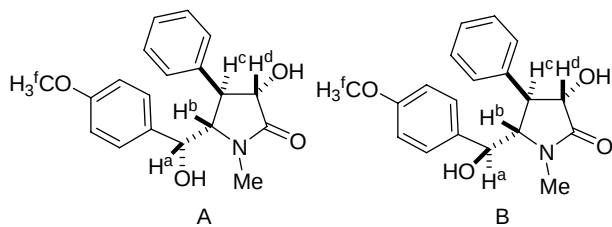
Current Data Parameters
NAME y1003-3
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070812
Time 22.05
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 48
DS 0
SWH 8992.806 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 362
DW 55.600 usec
DE 6.00 usec
TE 300.6 K
D1 5.00000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.60 usec
PL1 -1.00 dB
SF01 300.1318534 MHz

F2 - Processing parameters
SI 32768
SF 300.1300059 MHz
WDW EM
SSB 0
LB 0.35 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 18.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.200 ppm
F2 -60.03 Hz
PPMCM 0.41818 ppm/cm
HZCM 125.50892 Hz/cm



Current Data Parameters
NAME y1003-3
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070407
Time 11.24
INSTRUM av300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 658
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 724.1
DW 27.800 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 9.40 usec
PL1 -1.00 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -1.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 22.00 cm
CY 20.00 cm
F1P 190.000 ppm
F1 14338.87 Hz
F2P -2.000 ppm
F2 -150.94 Hz
PPMCM 8.72727 ppm/cm
HZCM 658.62756 Hz/cm

