Electronic Supplementary Information

Highly Efficient and Concise Synthesis of Both Antipodes of SB204900, Clausenamide, Neoclausenamide, Homoclausenamide and -Clausenamide, Implication of Biosynthetic Pathways of Clausena Alkaloids

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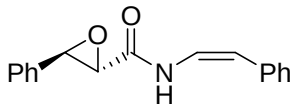
Table of Contents

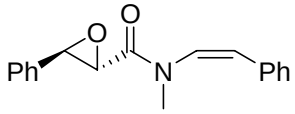
1, General information

^{1}H and ^{13}C NMR spectra were recorded on a Bruker Advance 300 spectrometer at ambient temperature. Chemical shifts are reported in ppm with either tetramethylsilane or the residual solvent resonance as an internal standard. Melting points are uncorrected. All yields reported were isolated yield and ee value were determined by HPLC using isopropanol and hexane (1:9) as eluent. All chemicals were dried or purified according to standard procedures prior to use. (+) and (-)-3-phenyloxirane-2-carboxamide **22**[1,2] were prepared following the literature methods.

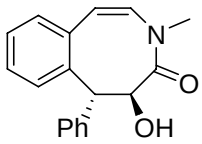
2, Synthetic procedures and characterization data of products



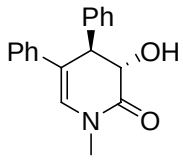
(+)-23: oil; $[\alpha]_D^{25}$ +284^o (*c* 1.0, $CHCl_3$); {lit.[3] (-)-**23**, $[\alpha]_D^{25}$ -282° (*c* 1.1, $CHCl_3$)}; IR (KBr) ν 3392, 1698, 1654 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.45 (d, *J* = 11.1 Hz, 1H), 7.24-7.41 (m, 10H), 6.94 (dd, *J* = 9.6, 11.1 Hz, 1H), 5.86 (d, *J* = 9.6 Hz, 1H), 3.92 (d, *J* = 1.9 Hz, 1H), 3.59 (d, *J* = 2.0 Hz, 1H).



(+)-SB204900 (+)-7: oil; $[\alpha]_D^{25}$ +14.2^o (*c* 3.3, $CHCl_3$); {lit.[3] (-)-**7**, $[\alpha]_D^{25}$ -16° (*c* 3.3, $CHCl_3$)}; IR (KBr) ν 1673, 1637 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.96-7.26 (m, 10H), 6.33 (d, *J* = 8.6 Hz 1H), 6.20 (d, *J* = 8.6 Hz, 1H), 3.79 (d, *J* = 1.7 Hz, 1H), 3.76 (d, *J* = 1.8 Hz, 1H), 3.12 (s, 3H).

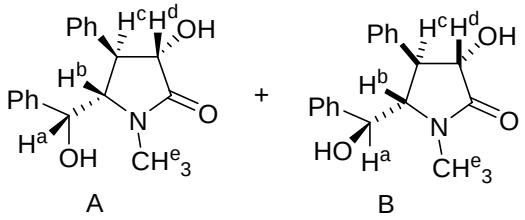


(-)-ξ-Clausenamide (-)-4: oil; $[\alpha]_D^{25}$ -140.3^o (*c* 0.91, $CHCl_3$); {lit.[3] (+)-**4**, $[\alpha]_D^{25}$ +147° (*c* 1.1, $CHCl_3$)}; IR (KBr) ν 3361, 1644 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.10-7.25 (m, 9H), 6.82 (d, *J* = 8.3 Hz 1H), 6.17 (d, *J* = 8.3 Hz, 1H), 5.09 (t, *J* = 9.2 Hz, 1H), 4.13 (d, *J* = 9.6 Hz, 1H), 3.54 (d, *J* = 8.9 Hz, 1H), 2.94 (s, 3H).



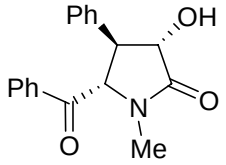
(-)-Homoclausenamide (-)-3: oil; $[\alpha]_D^{25}$ -265 ^{o} (*c* 2.50, CH_2Cl_2); IR (KBr) ν 3259, 1664, 1633 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 300K) δ7.04-7.25 (m, 10H), 6.42 (d, *J* = 1.8 Hz, 1H), 4.35 (dd, *J* = 2.6, 10.0 Hz, 1H), 4.20 (dd, *J* = 1.7, 10.0 Hz, 1H), 3.68 (d, *J* = 2.6 Hz, 1H), 3.24 (s, 3H); ^{13}CNMR (75 MHz, $CDCl_3$, 300K) δ169.2, 139.0, 137.7,

128.7, 128.6, 128.2, 127.1, 126.6, 126.1, 120.8, 73.3, 49.6, 34.3; MS (ESI): 279 $[M]^+$(59), 261(38), 250(100). Anal. Calcd. for $C_{18}H_{17}NO_2$: C, 77.40; H, 6.13; N, 5.01. Found: C, 77.11; H, 6.19; N, 5.10.

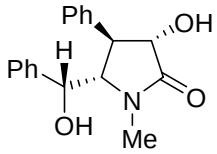


A B

2+2' (3 : 7): oil; IR (KBr) ν 3439, 3194, 1685 cm^{-1}; ^1H NMR (300 MHz, d_6-DMSO, 300K) δ6.63-7.26 (m, 10H), 5.77 (d, *J* = 4.6 Hz, 0.30H), 5.60 (d, *J* = 6.5 Hz, 0.30H), 5.48 (d, *J* = 4.0 Hz, 0.70H), 5.40 (d, *J* = 6.3 Hz, 0.70H), H_A^a, 5.01 (t, *J* = 3.1 Hz, 0.30H), H_B^a 4.64 (t, *J* = 2.9 Hz, 0.70H), H_B^b 4.29 (dd, *J* = 2.1, 8.4 Hz, 0.70H), H_A^b, H_A^d, H_B^d 3.78-3.92 (m, 1.30H), H_B^c 3.51 (dd, *J* = 8.6, 10.8 Hz, 0.70H), H_A^c, 3.06 (t, *J* = 7.2, 0.30H), H_B^e 3.01 (s, 2.10H), H_A^e 2.92 (s, 0.90H); ^{13}CNMR (75 MHz, $CDCl_3$, 300K) δ179.4, 178.1, 147.2, 145.1, 141.5, 133.9, 133.0, 132.9, 132.8, 132.7, 132.5, 132.2, 132.0, 132.8, 131.5, 131.3, 131.0, 82.3, 77.2, 73.8, 72.8, 70.3, 54.4, 51.7, 35.6, 33.1; MS (EI): 297$[M]^+$(3), 279(6), 190(100), 191(52), 174(28), 173(78).

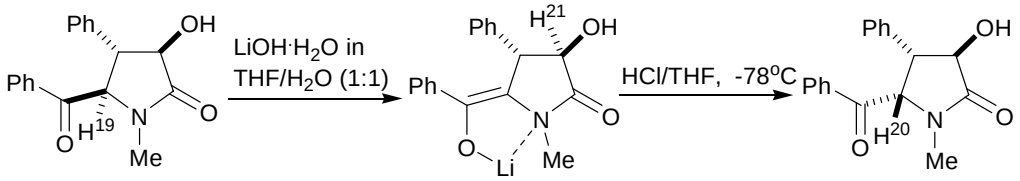


(+)-Neoclausenamidone (+)-19: mp 174-176℃, $[\alpha]_D^{25}$ +14.3° (*c* 0.56, $CHCl_3$); {lit.[2] (-)-neoclausenamidone, mp 165-169℃, $[\alpha]_D^{25}$ -14.55° (*c* 0.50, $CHCl_3$)}; IR (KBr) ν 3276，1684 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.17-7.67 (m, 10H), 5.09 (d, *J* = 6.5 Hz, 1H), 4.48 (dd, *J* = 3.8, 6.5 Hz, 1H), 3.29 (t, *J* = 6.5 Hz, 1H), 3.09 (d, *J* = 3.8 Hz, 1H), 2.95 (s, 3H).

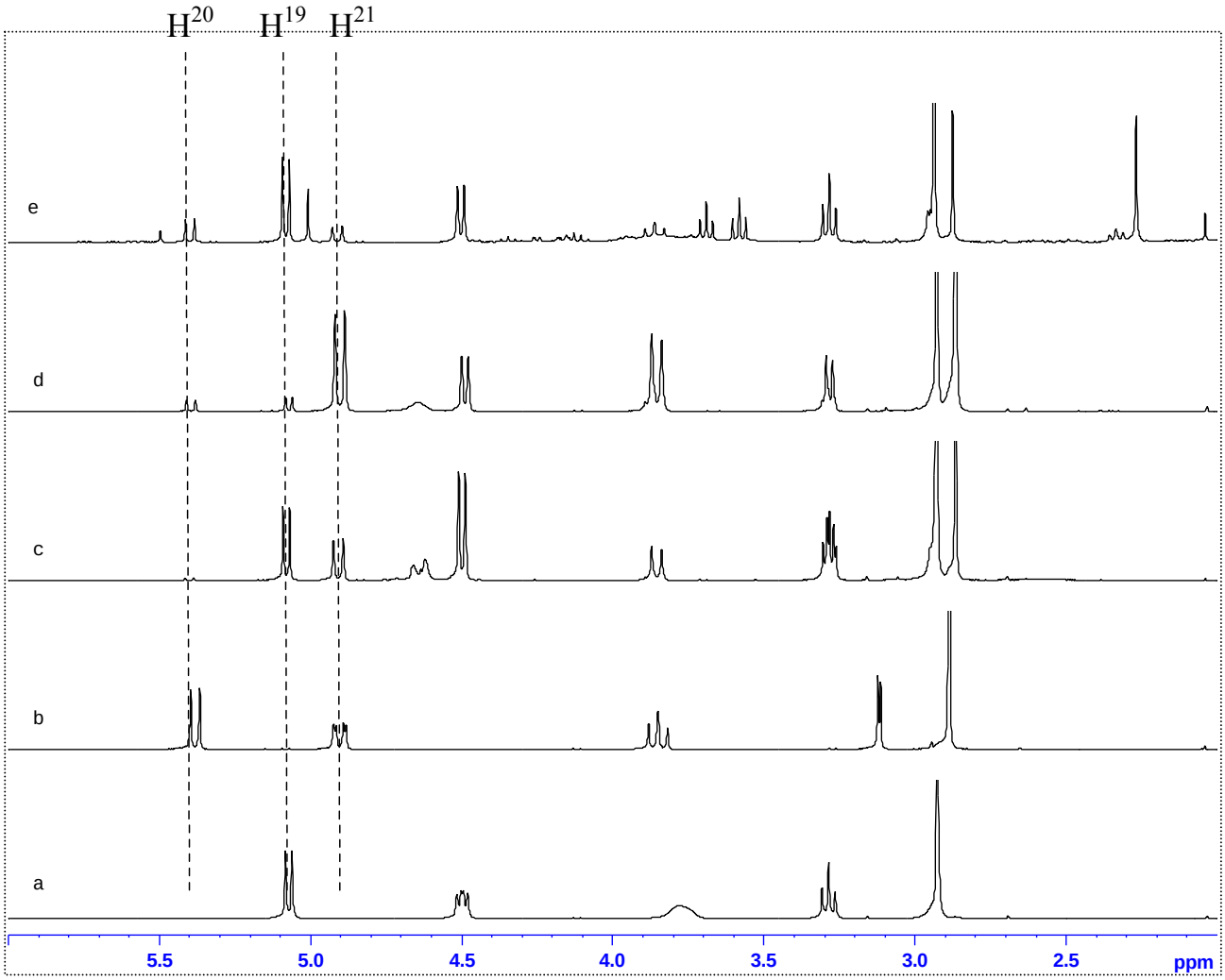


(-)-Neoclausenamide (-)-2: mp 187-188℃, $[\alpha]_D^{25}$ -87.8° (*c* 0.41, CH_3OH); {lit.[5] (+)-neoclausenamide, mp 179.6-181.4℃, $[\alpha]_D^{25}$ +87.7° (*c* 0.13, CH_3OH)}; IR (KBr) ν 3416, 1692 cm^{-1}; ^1H NMR (300 MHz, d_6-DMSO) δ 6.84-7.26 (m, 10H), 5.72 (d, *J* = 4.5 Hz, 1H), 5.57 (d, *J* = 5.7 Hz, 1H), 5.00 (t, *J* = 3.4 Hz, 1H), 3.85-3.92 (m, 2H), 3.06 (t, *J* = 7.1, Hz, 1H), 2.91 (s, 3H).

(±)-Clausenamidone (±)-20:



19 **21** **20**



a. ^{1}H NMR spectrum of racemic **19** (0.04 mmol, 12 mg) in $CDCl_3$ (0.5 mL) at room temperature.

b. ^{1}H NMR spectrum of racemic **20** (0.04 mmol, 12 mg) in $CDCl_3$ (0.5 mL) at room temperature.

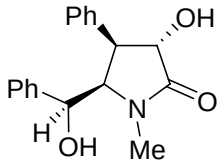
c. ^{1}H NMR spectrum of a mixture of racemic **19** (0.04 mmol, 12 mg) and LiOH • $2H_2O$（0.08 mmol, 3.4 mg）in $CDCl_3$ (0.45 mL) and D_2O (0.05 mL) at room temperature after 12 h.

d. ^{1}H NMR spectrum of a mixture of racemic **20** (0.04 mmol, 12 mg) and LiOH • $2H_2O$（0.08 mmol, 3.4 mg）in $CDCl_3$ (0.45 mL) and D_2O (0.05 mL) at room temperature after 12 h.

e. To a mixture of racemic (±)-**19** (0.5 mmol, 148 mg) in THF (5 mL) and water (5 mL) was added LiOH • $2H_2O$ (1 mmol, 42 mg). After stirring for 12 h at room temperature, the reaction flask was cooled down to -78 ^{o}C, and the mixture was kept

stirring for another 12 h. The precipitate was observed in the mixture. While keeping at -78 ^{o}C, a solution of aqueous hydrochloric acid in THF (2 N, 1 mL) [prepared from concentrate hydrochloric acid (12 N) and THF] was added through a syringe. The temperature of the reaction mixture was then allowed to rise to room temperature gradually for about 2 h. Water (50 mL) was added and the mixture was extracted with ethyl acetate (50 mL×3). Organic layer was combined and dried over with anhydrous Na_2SO_4. After filtration and concentration under vacuum, the crude sample (10 mg) was dissolved in $CDCl_3$ (0.5 mL) and the ^{1}H NMR was recorded. The ratio of 20 : **19** : **21** was roughly determined by measuring proton peaks of H^{19}, H^{20} and H^{21}. In spectrum c, **20** : **19** : **21** = 2 : 55 : 43. In spectrum d, **20** : **19** : **21** = 7 : 10 : 83. In spectrum e, **20** : **19** : **21** = 18 : 60 : 22.

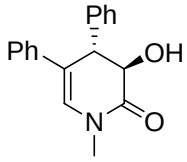
(-)-Clausenamidone (-)-20: mp 195-197℃, $[\alpha]_D^{25}$ -345° (*c* 0.29, CH_3OH) {lit.[2] (+)-clausenamidone, mp 203-206℃, $[\alpha]_D^{25}$ +333° (*c* 0.01, CH_3OH)}; IR (KBr) ν 3331, 1692 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.02-7.54 (m, 10H), 5.38 (d, *J* = 8.9 Hz, 1H), 4.90 (dd, *J* = 2.5, 9.9 Hz, 1H), 3.85 (t, *J* = 9.2 Hz, 1H), 3.12 (t, *J* = 2.6 Hz, 1H), 2.89 (s, 3H).



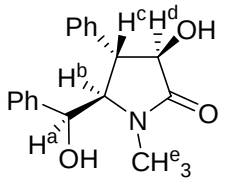
(-)-Clausenamide (-)-1: mp 176-178℃. $[\alpha]_D^{25}$ -123° (*c* 0.26, DMSO:H_2O = 9:1 (v/v); {lit.[6] (+)-clausenamide, mp 152-153℃, $[\alpha]_D^{25}$ +123.19° (*c* 0.46, DMSO : H_2O = 9:1 (v/v)}; IR (KBr) ν 3408，3207, 1688 cm^{-1}; ^{1}H NMR (300 MHz, d_6-DMSO) δ.04-7.26 (m, 8H); 6.63-6.66 (m, 2H); 5.45 (d, *J* = 4.0 Hz, 1H), 5.38 (d, *J* = 6.3 Hz, 1H), 4.64 (s, 1H), 4.29 (dd, *J* = 2.0, 8.3 Hz, 1H), 3.81 (dd, *J* = 6.4, 11.0 Hz, 1H), 3.50 (dd, *J* = 8.5, 10.8 Hz, 1H), 3.01 (s, 3H).

The procedures for the synthesis of (-)-7[3], (±)-7[3], (+)-3, (+)-4[3], (±)-13[4], (±)-15[4] and (±)-15'[4], (-)-19, (+)-2, (+)-20, and (+)-1 were the same as their enantiomers. The

characterization of **(±)-7**[3]**,** (+)-**4**[3], **(±)-14**[4], **(±)-15**[4] and **(±)-15'**[4] was reported in our preliminarily communications.



(+)-Homoclausenamide (+)-3: Yield, 62%; oil, $[\alpha]_D^{25}$ +265° (*c* 2.50, CH_2Cl_2); IR (KBr) ν 3259, 1664, 1633 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 300K) δ 7.05-7.25 (m, 10H), 6.42 (d, *J* = 1.7 Hz, 1H), 4.35 (dd, *J* = 2.5, 10.0 Hz, 1H), 4.20 (dd, *J* = 1.6, 10.0 Hz, 1H), 3.66 (d, *J* = 2.6 Hz, 1H), 3.23 (s, 3H).



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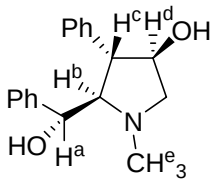

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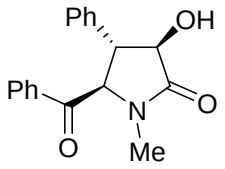
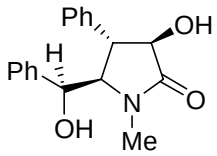
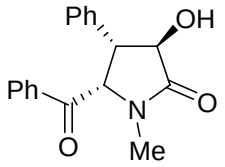
(+)-2 + 2' (3 : 7): Yield, 75%; oil; IR (KBr) ν 3440, 3207, 1686 cm^{-1}; ^1H NMR (300 MHz, d_6-DMSO, 300K) δ 6.63-7.25 (m, 10H), 5.75 (d, *J* = 4.7 Hz, 0.30H), 5.59 (d, *J* = 6.4 Hz, 0.30H), 5.47 (d, *J* = 4.0 Hz, 0.70H), 5.39 (d, *J* = 6.3 Hz, 0.70H), H_A^a, 5.00 (t, *J* = 4.2 Hz, 0.30H), H_B^a 4.64 (t, *J* = 2.5 Hz, 0.70H), H_B^b 4.29 (dd, *J* = 2.3, 8.3 Hz, 0.70H), H_A^b, H_A^d, H_B^d 3.79-3.90 (m, 1.30H), H_B^c 3.50 (dd, *J* = 8.4, 10.8 Hz, 0.70H), H_A^c, 3.06 (t, *J* = 7.2, 0.30H), H_B^e 3.01 (s, 2.10H), H_A^e 2.91 (s, 0.90H).



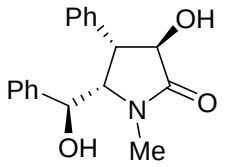
(-)-Neoclausenamidone (-)-19: Yield, 91%; mp 174-176℃, $[\alpha]_D^{25}$ -14.3° (*c* 0.56, $CHCl_3$); {lit.[2] (-)-neoclausenamidone, mp 165-169℃, $[\alpha]_D^{25}$ -14.55° (*c* 0.50, $CHCl_3$)}; IR (KBr) ν 3276，1684 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.17-7.67 (m, 10H), 5.07 (d, *J* = 6.5 Hz 1H), 4.50 (dd, *J* = 4.1, 6.4 Hz, 1H), 3.78 (b, 1H), 3.29 (t, *J* = 6.5 Hz, 1H), 2.93 (s, 3H).



(+)-Neoclausenamide (+)-2: mp 187-188℃, $[\alpha]_D^{25}$ +87.8° (*c* 0.41, CH_3OH); {lit.[5] (+)-neoclausenamide, mp 179.6-181.4℃, $[\alpha]_D^{25}$ +87.7° (*c* 0.13, CH_3OH)}; IR (KBr) ν 3417, 1692 cm^{-1}; ^1H NMR (300 MHz, d_6-DMSO) δ 6.85-7.25 (m, 10H), 5.73 (d, *J* = 4.6 Hz, 1H), 5.60 (d, *J* = 6.4 Hz, 1H), 5.00 (t, *J* = 3.4 Hz, 1H), 3.86-3.92 (m, 2H), 3.06 (t, *J* = 7.2, Hz, 1H), 2.91 (s, 3H).

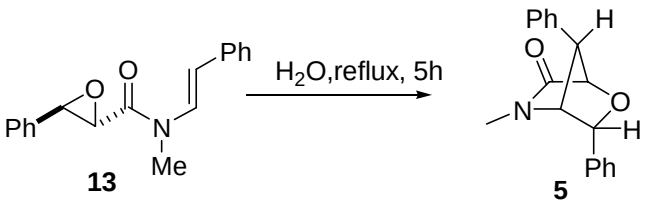


(+)-Clausenamidone (+)-20: Yield, 61%; mp 195-197℃, $[\alpha]_D^{25}$ +345° (*c* 0.29, CH_3OH); {lit.[2] (+)-clausenamidone, mp 203-206℃, $[\alpha]_D^{25}$ +333° (*c* 0.01, CH_3OH)}; IR (KBr) ν 3331, 1692 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.02-7.54 (m, 10H), 5.38 (d, *J* = 8.9 Hz, 1H), 4.90 (dd, *J* = 2.6, 9.8 Hz, 1H), 3.86 (t, *J* = 9.3 Hz, 1H), 3.30 (t, *J* = 3.7 Hz, 1H), 2.89 (s, 3H).



(+)-Clausenamide (+)-1: Yield, 90%; mp 176-178℃; $[\alpha]_D^{25}$ +123° (*c* 0.26, DMSO : H_2O = 9:1 (v/v)); {lit.[6] (+)-clausenamide, mp 152-153℃, $[\alpha]_D^{25}$ +123.19° (*c* 0.46, DMSO : H_2O = 9:1 (v/v)}; IR (KBr) ν 3408，3207, 1688 cm^{-1}; ^1H NMR (300 MHz, d_6-DMSO) δ 7.03-7.26 (m, 8H), 6.63-6.66 (m, 2H), 5.42 (s, 1H), 5.36 (d, *J* = 4.9 Hz, 1H), 4.64 (s, 1H), 4.29 (dd, *J* = 2.2, 8.3 Hz, 1H), 3.83 (dd, *J* = 6.4, 10.9 Hz, 1H), 3.50 (dd, *J* = 8.6, 10.8 Hz, 1H), 3.01 (s, 3H).

Synthesis of Cycloclausenamide (±)**-5.**

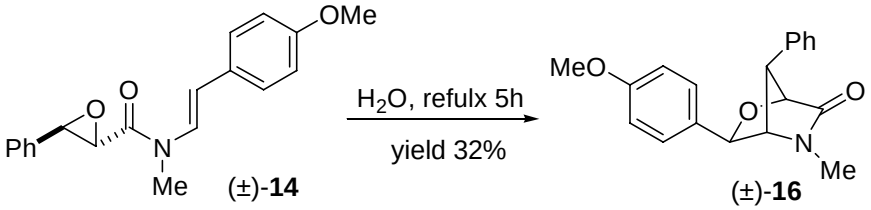


13 **5**

Our previous method for the reaction of racemic *N*-(*E*-styryl)-3-phenyloxirane-2-carboxamide (±)-**13** was adopted[4]. Refluxing a suspension of enamides (±)-**13** (5 mmol, 1.395 g) in pure water (150 mL) for 5 h under argon protection gave rise to a homogeneous solution. After addition of brine (50 mL), the mixture was extracted with ethyl acetate (3×50 mL). The organic layer was dried with anhydrous Na_2SO_4, filtrated and concentrated under vacuum. The chromatography using a silica gel (200-300 mesh) column eluting with a mixture of petroleum ether and ethyl acetate (1:1) gave cycloclausenamide (±)-**5** (55 mg, 4%) and **2+2'** (30:70, 1.351 g, 91%).

Cycloclausenamide (±)-**5:** mp 140-142℃ (Lit.[7] mp 164-166℃); IR (KBr) ν 3435, 1672, 1639 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 300K) δ 7.08-7.47 (m, 10H), 5.00 (s, 1H), 4.81 (dd, *J* = 1.2, 2.3 Hz, 1H), 4.09 (s, 1H), 3.60 (s, 1H), 2.95 (s, 3H).

Synthesis of Cycloclausenamide analog (±)-**16.**



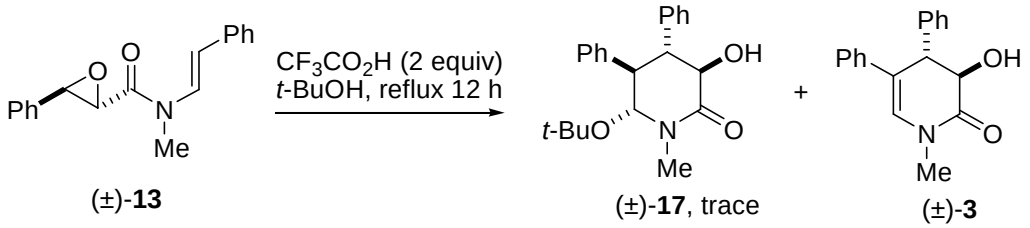
(±)-**14** (±)-**16**

Our previous method for the reaction of racemic *N*-(*E*-styryl)-3-phenyloxirane-2-carboxamide (±)-**14** was adopted[4]. Refluxing a suspension of enamides (±)-**14** (1 mmol, 309mg) in pure 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 Na_2SO_4, filtrated and concentrated under vacuum. The chromatography using a silica gel (200-300 mesh) column eluting with a mixture of petroleum ether and ethyl acetate (1:1) gave products (±)-**16** (99 mg, 32%) and **15+15'** (30:70, 222 mg, 68%).

Cycloclausenamide analog (±)-**16**: mp 158-160℃; IR (KBr) ν 1704 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$, 300K) δ 6.93-7.39 (m, 9H), 4.96 (s, 1H), 4.79 (s, 1H), 4.04 (s, 1H),

3.83 (s, 3H), 3.61 (s, 1H), 2.94 (s, 3H); ^{13}CNMR (75 MHz, $CDCl_3$, 300K) δ 172.4, 159.3, 122.8, 131.1, 128.7, 127.9, 127.4, 126.8, 114.1, 80.3, 80.2, 70.3, 55.4, 50.6, 27.4; MS (ESI) $[M+1]^+$ (100), 332 $[M+Na]^+$ (42). Anal. Calcd. for $C_{19}H_{19}NO_3$: C, 73.77; H, 6.19; N, 4.53. Found: C, 73.52; H, 6.17; N, 4.52.

Synthesis of Lansamide-3 analog (±)-17.



(±)-**13** (±)-**17**, trace (±)-**3**

Our previous method for the reaction of racemic *N*-(*E*-styryl)-3-phenyloxirane-2-carboxamide (±)-**13** was adopted[4]. A mixture of enamide (±)-**13** (1 mmol, 279 mg) in dry Bu^{t}OH (30 mL) under argon protection was heated to reflux, and then *p*-TFA (2 mmol, 224 mg) was added. After refluxing for another 12 h, the mixture was cooled and a saturated aqueous solution of $NaHCO_3$ (30 mL) was added. The resulting mixture was extracted with ethyl acetate (3×30 mL). The organic layer was dried with anhydrous Na_2SO_4, filtrated and concentrated under vacuum. Pure product (±)-**3** (234 mg, 84%) and Lansamide-4 analogs (±)-**17** was obtained after silica gel (200-300 mesh) column chromatography using a mixture of petroleum ether and ethyl acetate (2:1) as an eluant.

Lansamide-3 analogs (±)-**17**: mp 159-161℃; IR (KBr) ν 3324, 3227, 1684 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$, 300K) δ 7.16-7.31 (m, 8H), 6.77-6.81 (m, 2H), 4.73 (d, *J* = 6.2 Hz, 1H), 4.13 (t, *J* = 4.4 Hz, 1H), 4.16 (d, *J* = 4.8 Hz, 1H), 3.70 (dd, *J* = 3.9, 6.2 Hz, 1H), 3.03 (t, *J* = 3.9 Hz, 1H), 3.98 (s, 3H), 1.11 (s, 9H); ^{13}CNMR (75 MHz, $CDCl_3$, 300K) δ 175.0, 141.8, 141.5, 128.7, 128.2, 127.8, 127.7, 126.7, 75.6, 71.6, 49.2, 31.8, 28.8; MS (ESI) 354 $[M+1]^+$. Anal. Calcd. for $C_{22}H_{27}NO_3$: C, 74.76; H, 7.70; N, 3.96. Found: C, 74.50; H, 7.53; N, 4.06.

3, References

1, Aysmmtric Epoxidation see: (a) Katsuki, T., Sharpless, K. B., *J. Am. Chem. Soc.* **1980**, *102*, 5974-5978. Gao, Y., Hanson, R. M., Klunder, J. M., Ko, S. Y., Masamune, H., Sharpless, K. B., *J. Am. Chem. Soc.* **1987**, *109*, 5765. (b) Jean, N. D., Andrew, E. G., Serra, A. A., Luche, M. J., *J. Org. Chem.* **1986**, *51*, 46-50.

2, oxidation and esterification see: Huang, D. F., Huang, L. *Tetrahedron,* 1990, 46, 3135-3142.

3, Yang, L.; Deng, G.; Wang, D.-X.; Huang, Z.-T.; Zhu, J.; Wang, M.-X. *Org. Lett.* **2007**, *9*, 1387.

4, Yang, L.; Zheng, Q.-Y.; Wang, D.-X.; Huang, Z.-T.; Wang, M.-X. *Org. Lett.* **2008**, *10*, 2461.

5, Wang, J.-Q.; Tian, W.-S. *J. Chem. Soc., Perkin Trans 1,* **1996**, 209-211.

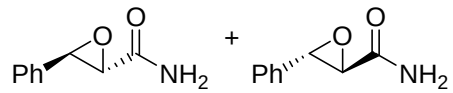
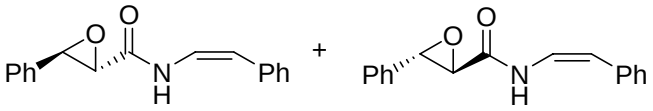
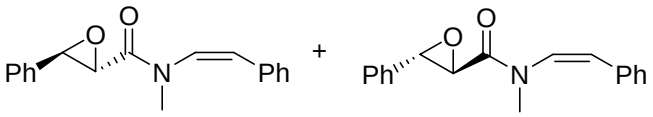
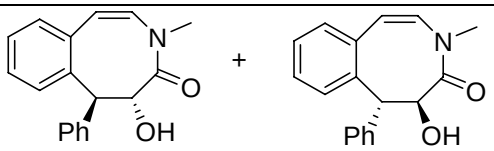
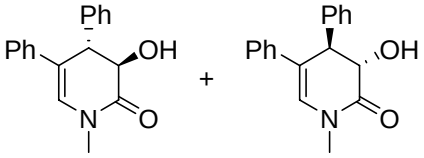
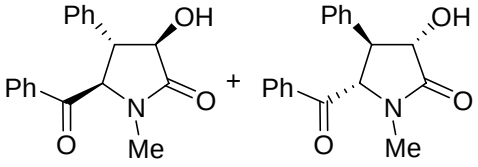
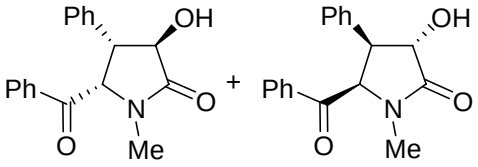
6, Hartwig, W.; Born, L. *J. Org. Chem.* **1987**, *52*, 4352.

7. Yang, M.-H.; Chen, Y.-Y.; Huang, L. *Phytochemistry* **1988**, *27*, 445.

4, HPLC analysis (+)22, (+)23, (+)7, (-)4, (-)3, (+)19, (-)20, (-)22, (-)23, (-)7, (+)4, (+)3, (-)19, (+)20

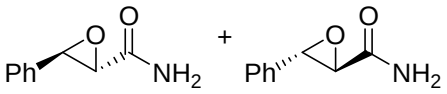
A Shimadzu LC-10AVP HPLC system was used to determine enantiomeric excess values of all products.

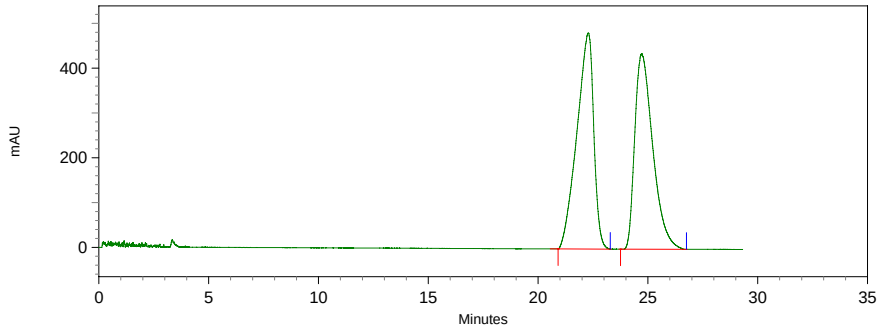
Chiral HPLC Analysis

entry	**Structure**	**retention time/min**	**column**
1		22.3/24.7	OD
2		12.7/15.7	OD
3		32.6/34.7	ADH
4		48.5/34.2	AD
5		32.2/21.3	AD
6		86.5/56.8	AD
7		57.8/88.4	AD

Chiral column were purchased form DAICEL Chemical Industries, LTD. Chiralcel ADH employed hexane : isopropanol = 9:1 as mobile phase, flow rate 0.5ml/min, 25℃; while Chiralcel AD or OD employed hexane : isopropanol = 9:1 as mobile phase, flow rate 0.8ml/min, 25℃.

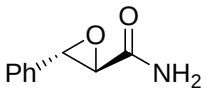
racemate 22

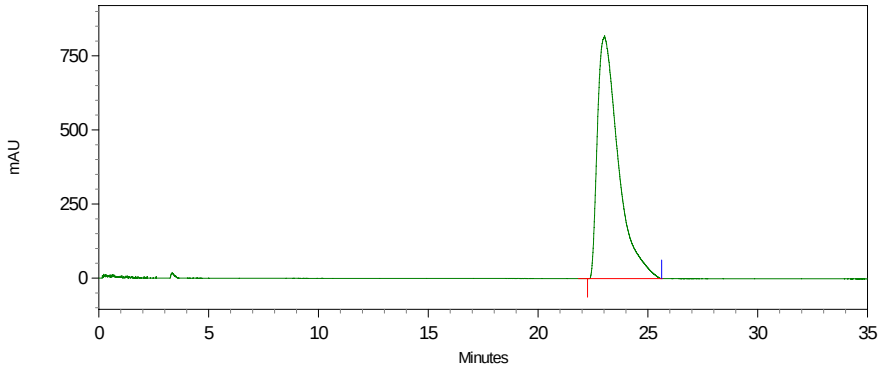




Pk #	Retention Time	Area	Area Percent
1	22.278	25716700	50.86
2	24.712	24843704	49.14
Totals		50560403	100.00

(-)-22





Pk #	Retention Time	Area	Area Percent
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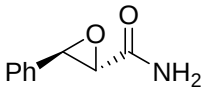
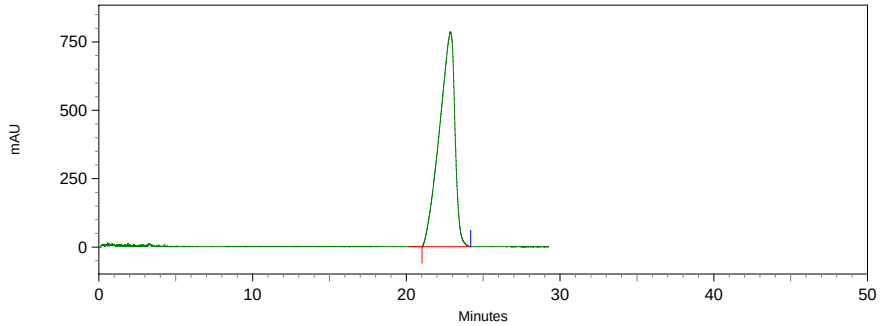
Totals		53780849	100.00

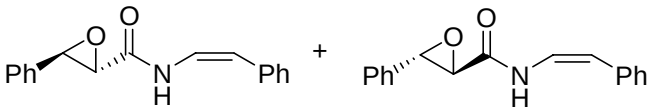
(+)-22

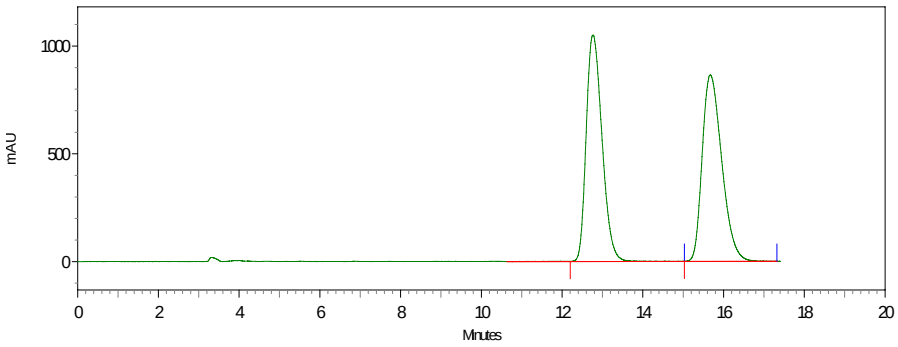




Pk #	Retention Time	Area	Area Percent
1	22.849	55135848	100.00
Totals		55135848	100.00

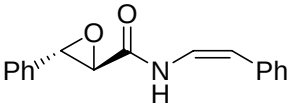
racemate 23

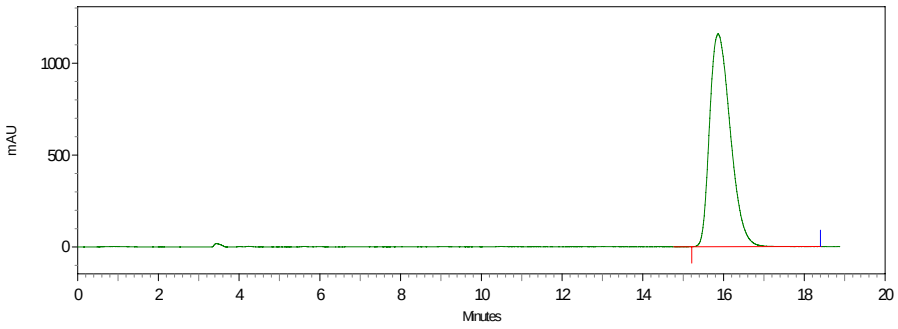




Pk #	Retention Time	Area	Area Percent
1	12.752	28582889	49.55
2	15.668	29102849	50.45
Totals		57685737	100.00

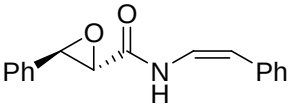
(-)-23

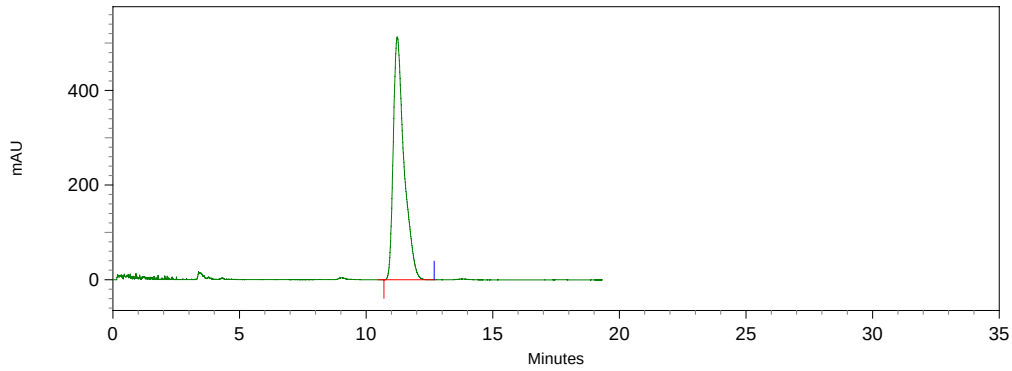




Pk #	Retention Time	Area	Area Percent
1	15.864	41129023	100.00
Totals		41129023	100.00

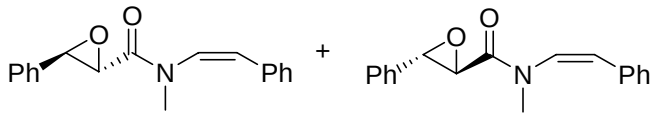
(+)-23

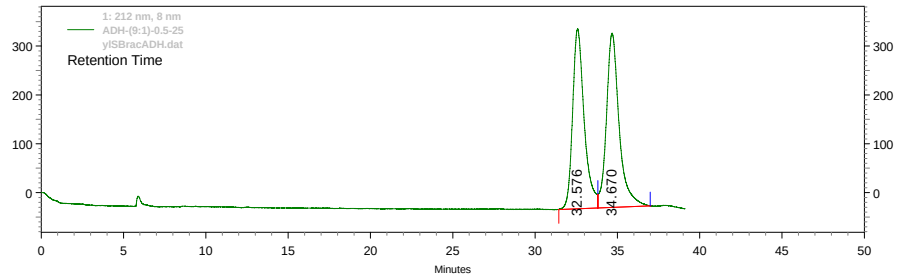




Pk #	Retention Time	Area	Area Percent
1	11.220	15079913	100.00
Totals		15079913	100.00

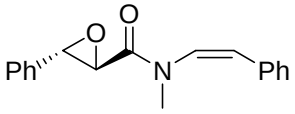
racemate 7

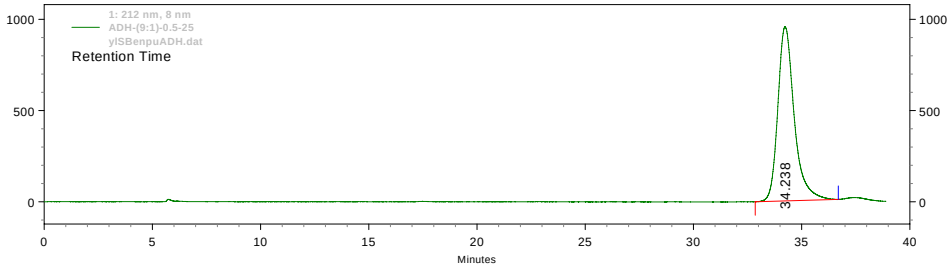




Pk #	Retention Time	Area	Area Percent
1	32.576	18068388	48.55
2	34.670	19145495	51.45
Totals		37213883	100.00

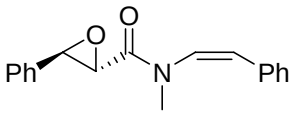
(-)-7

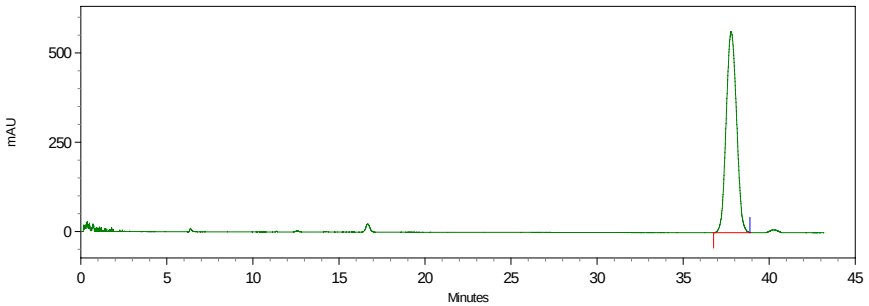




Pk #	Retention Time	Area	Area Percent
1	34.238	50273637	100.00
Totals		50273637	100.00

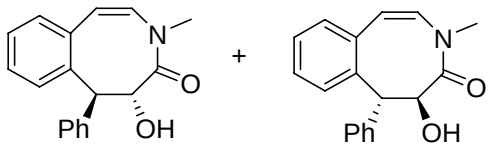
(+)-7

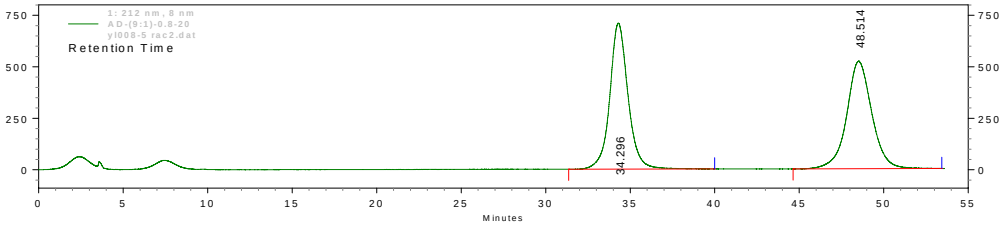




Pk #	Retention Time	Area	Area Percent
1	37.782	23525709	100.00
Totals		23525709	100.00

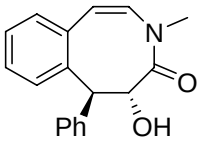
racemate 4

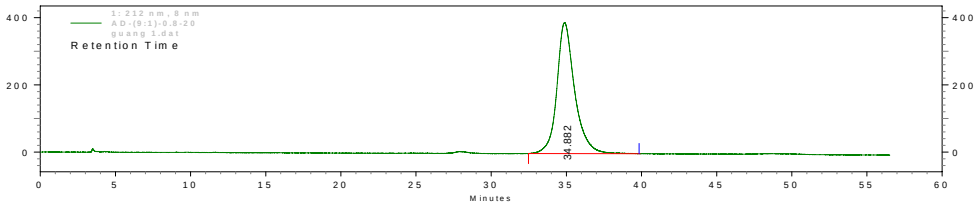




Pk #	Retention Time	Area	Area Percent
1	34.296	53248406	49.68
2	48.514	53941077	50.32
Totals		107189483	100.00

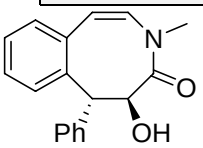
(+)-4

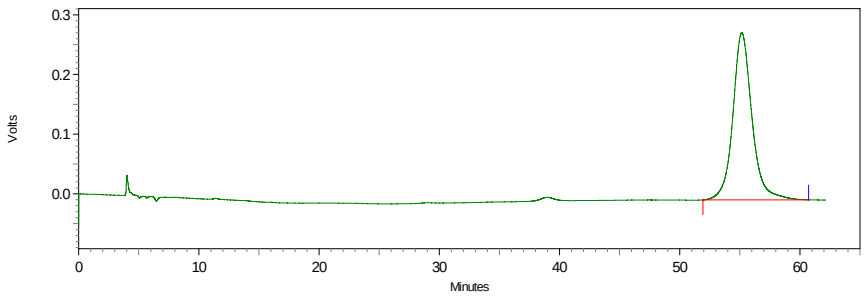




Pk #	Retention Time	Area	Area Percent
1	34.882	32069538	100.00
Totals		32069538	100.00

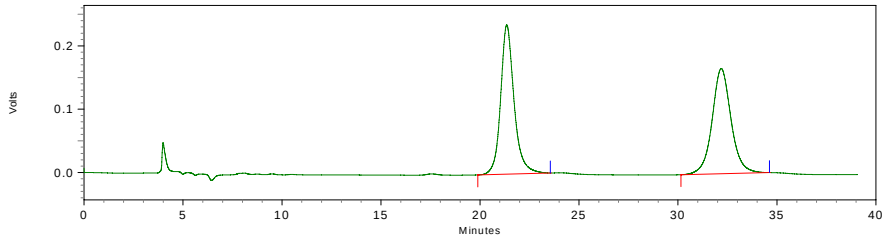
(-)-4





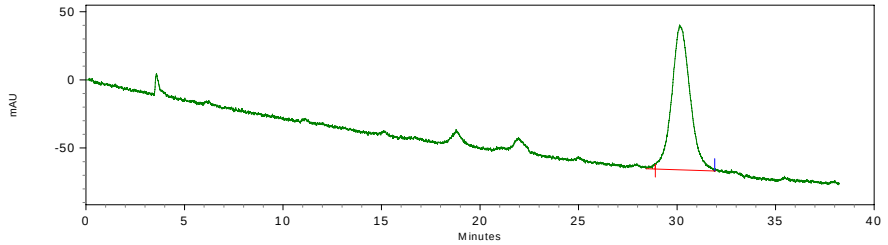
Pk #	Retention Time	Area	Area Percent
1	55.157	31056971	100.00
Totals		31056971	100.00

racemate 3



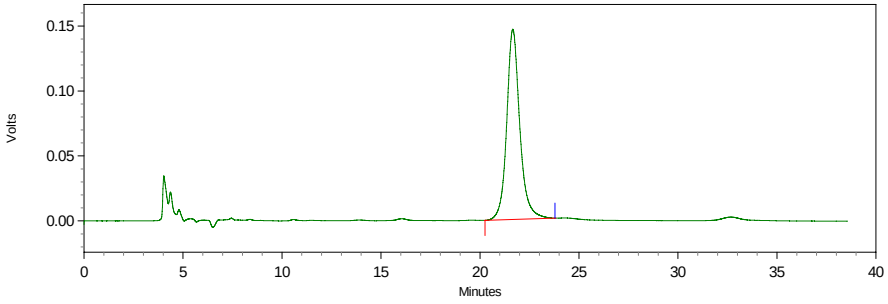
Pk #	Retention Time	Area	Area Percent
1	21.347	11107667	50.12
2	32.181	11053515	49.88
Totals		22161182	100.00

(+)-3



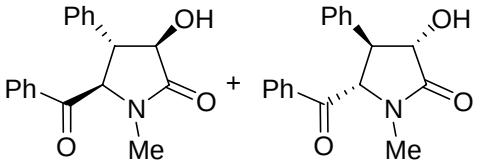
Pk #	Retention Time	Area	Area Percent
1	30.173	6809067	100.00
Totals		6809067	100.00

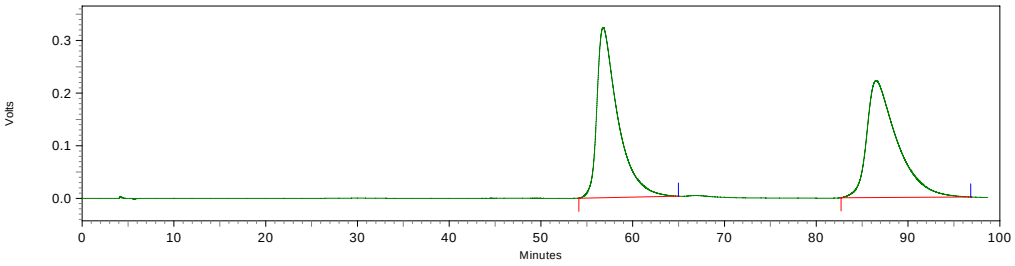
(-)-3



Pk #	Retention Time	Area	Area Percent
1	21.652	6713348	100.00
Totals		6713348	100.00

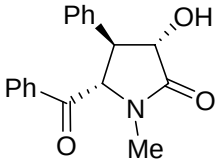
racemate 19

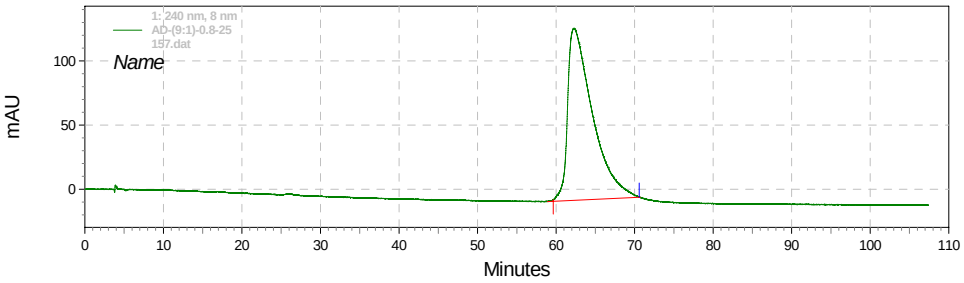




Pk #	Retention Time	Area	Area Percent
1	56.770	51421662	50.25
2	86.520	50912080	49.75
	Totals	102333742	100.00

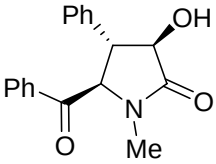
(+)-19

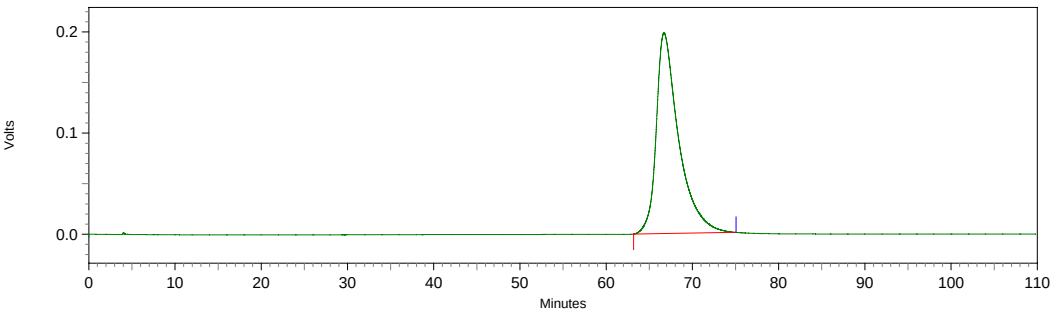




Pk #	Retention Time	Area	Area Percent
1	62.244	36180795	100.00
Totals		36180795	100.00

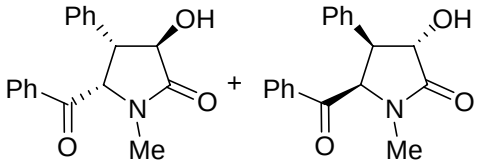
(-)-19

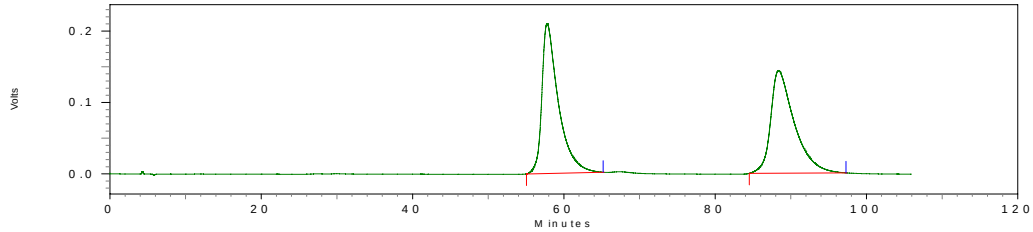




Retention Time	Area	Area Percent	Name
66.699	35908487	100.00	
Totals	35908487	100.00	

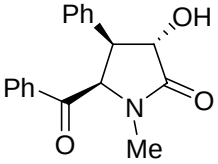
racemate 20

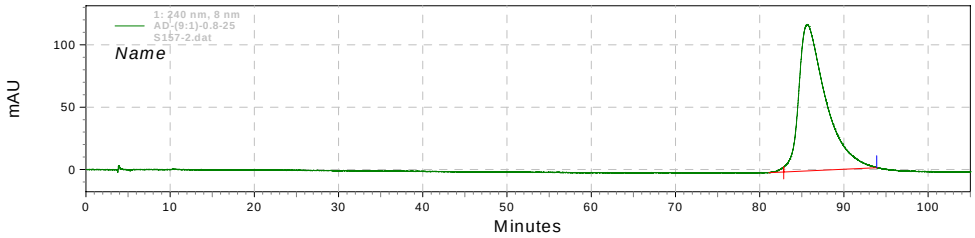




Retention Time	Area	Area Percent	Pk #
57.781	31890818	50.55	1
88.379	31197464	49.45	2
Totals	63088282	100.00	

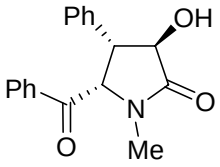
(-)-20

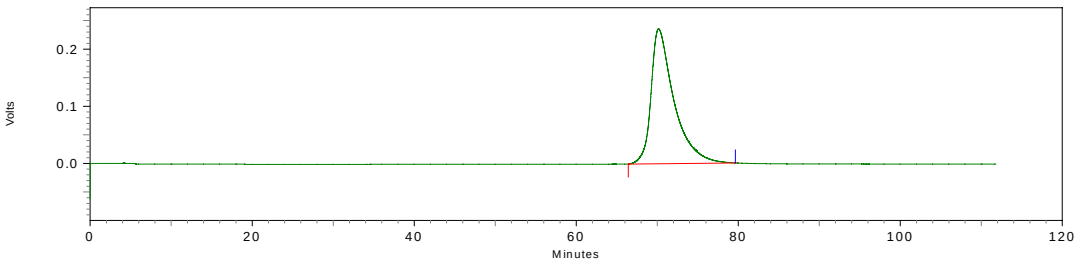




Pk #	Retention Time	Area	Area Percent
1	85.584	44389496	100.00
Totals		44389496	100.00

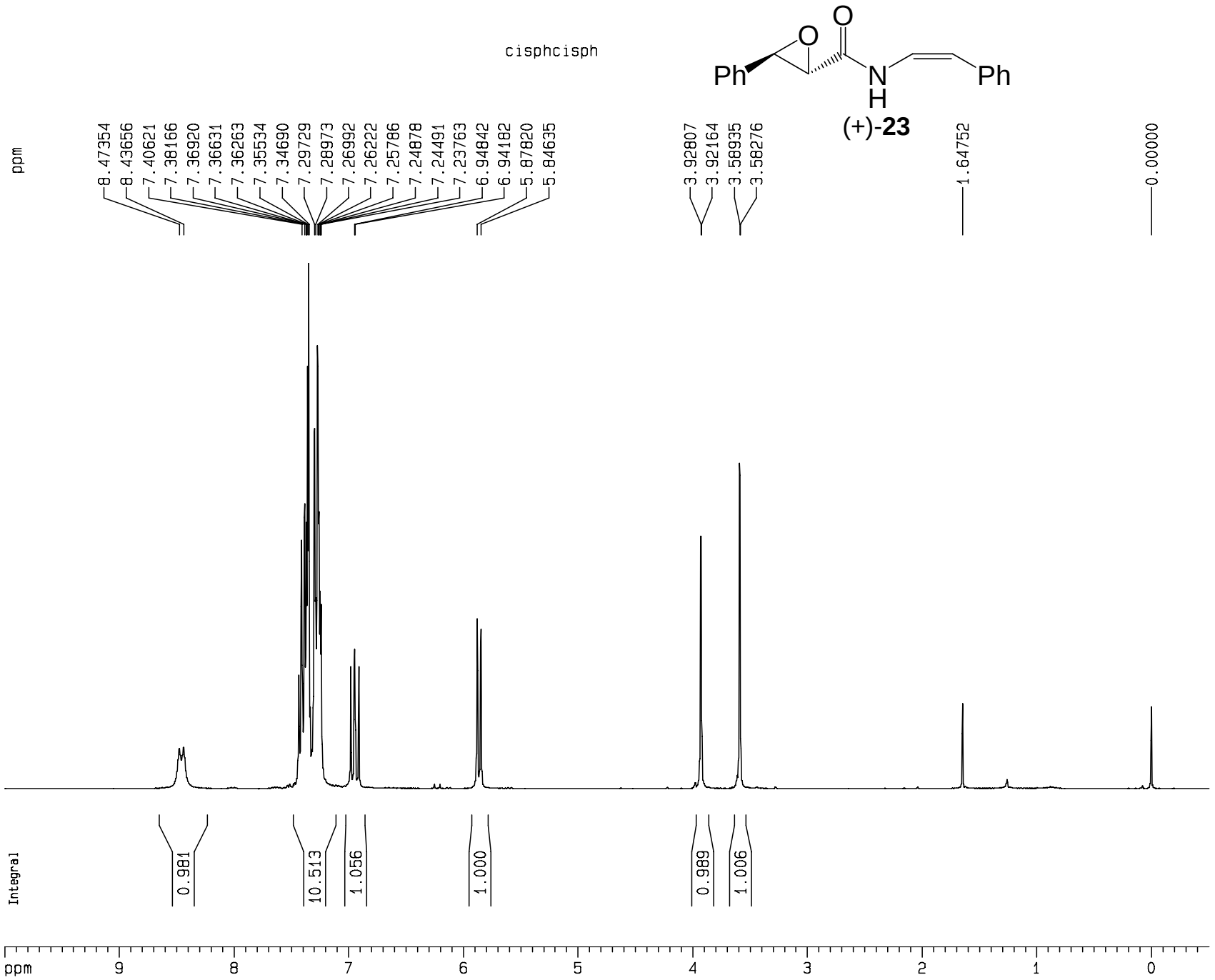
(+)-20





Pk #	Retention Time	Area	Area Percent
1	70.170	46375031	100.00
	Totals	46375031	100.00

5, Copies of ^{1}H and ^{13}C NMR spectra of compounds



Current Data Parameters

NAME	yl004
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20051120
Time	16.23
INSTRUM	av300
PROBHD	5 mm DUL 13C-1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	161.3
DW	81.000 usec
DE	6.00 usec
TE	295.8 K
D1	2.00000000 sec

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

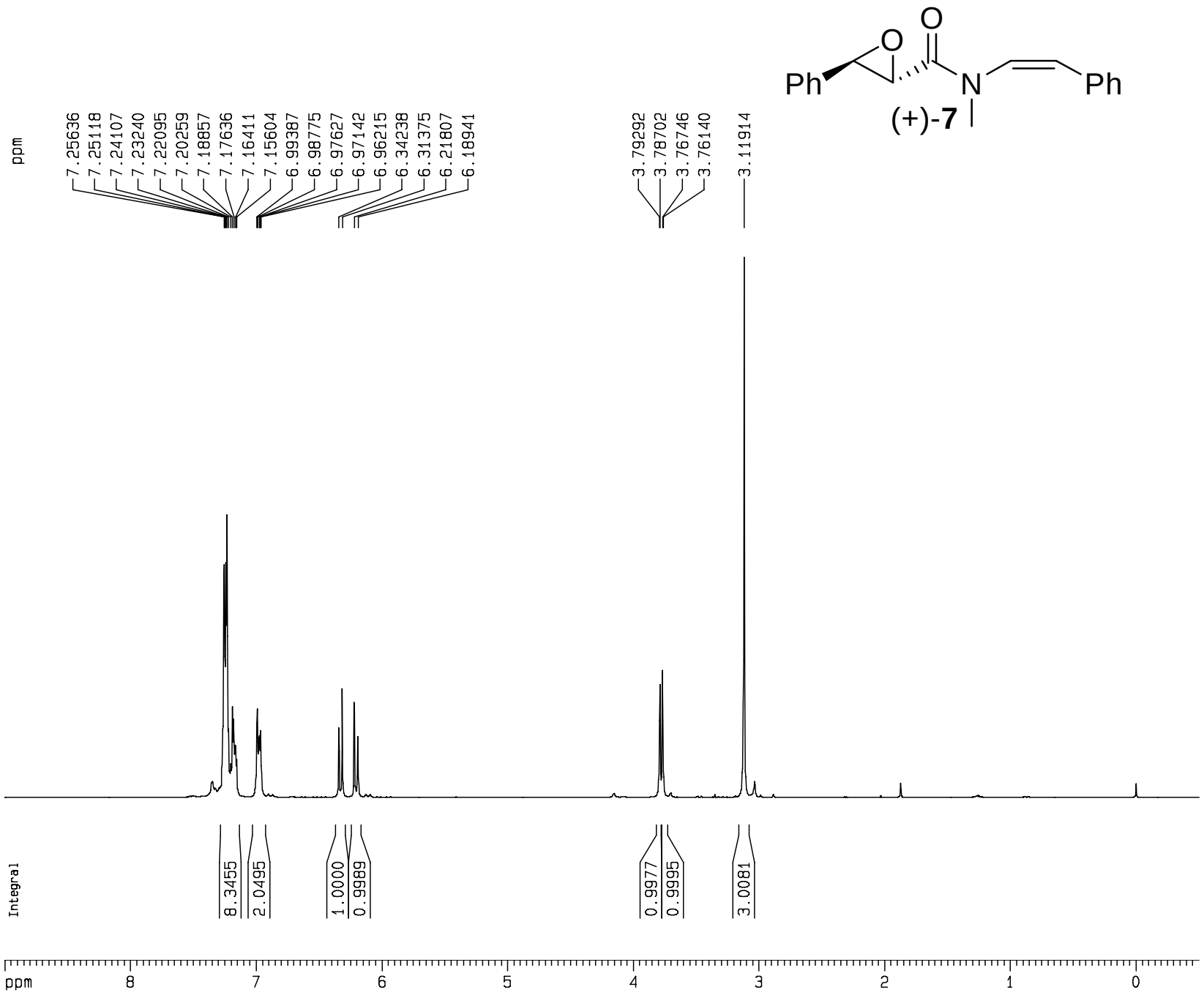
NUC1	1H
P1	9.30 usec
PL1	-1.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

SI	32768
SF	300.1300096 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

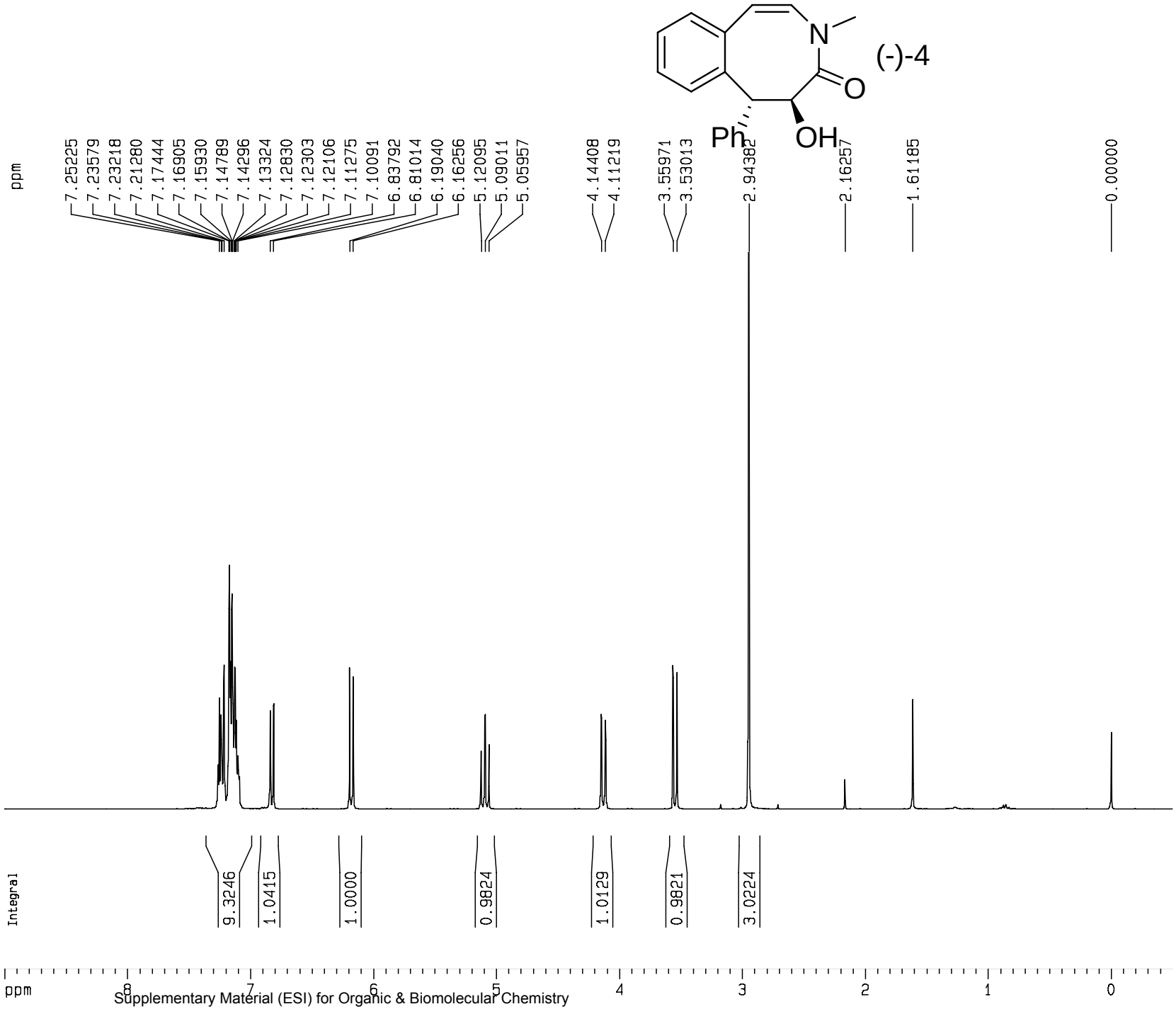
1D NMR plot parameters

CX	22.00 cm
CY	10.00 cm
F1P	10.000 ppm
F1	3001.30 Hz
F2P	-0.500 ppm
F2	-150.06 Hz
PPMCM	0.47727 ppm/cm
HZCM	143.24385 Hz/cm

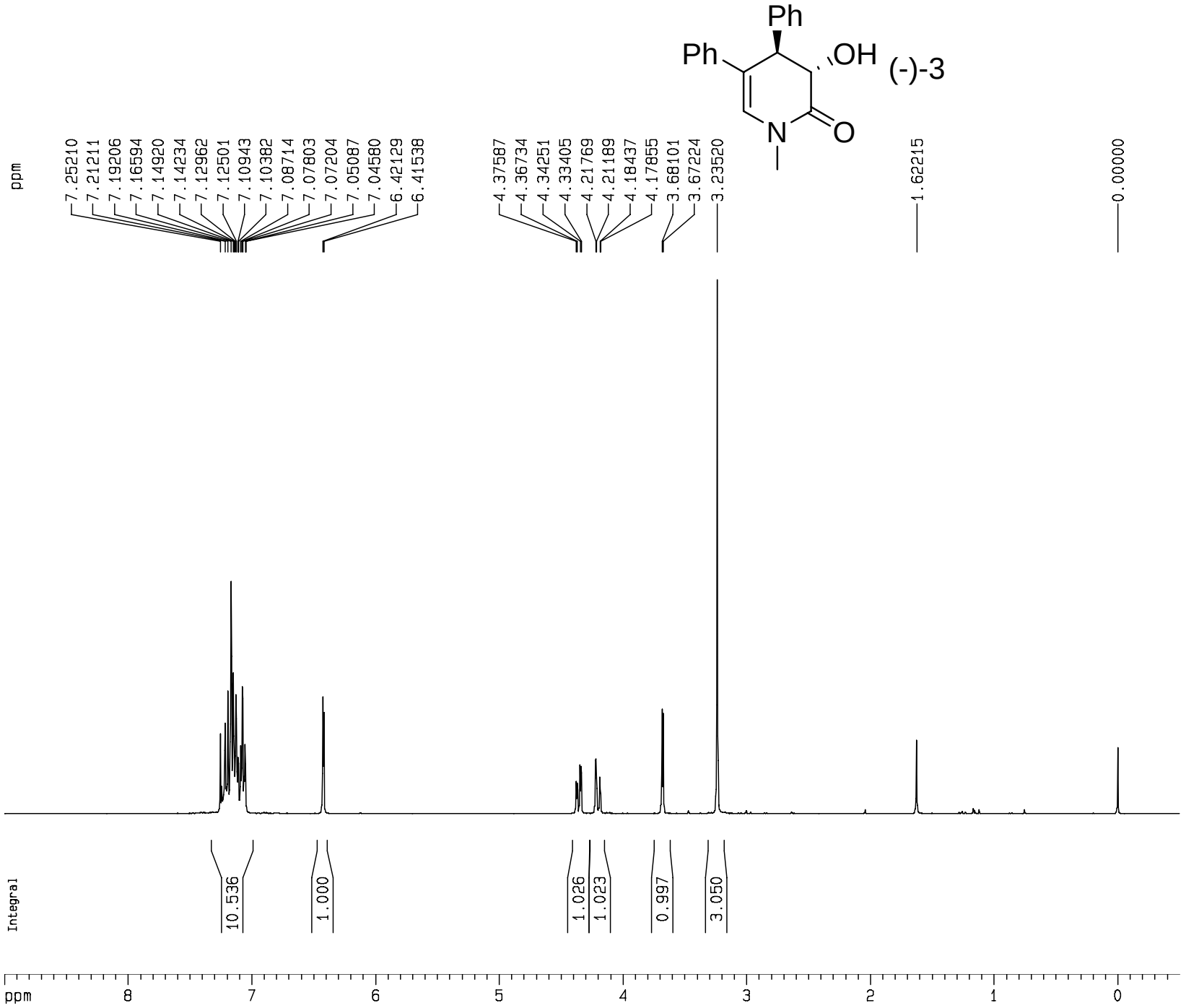


(+)-**7**

```
Current Data Parameters
NAME                  yl5SB
EXPNO                    10
PROCNO                    1

F2 - Acquisition Parameters
Date_              20070425
Time                 18.03
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                     90.5
DW                  55.600 usec
DE                   6.00 usec
TE                  300.5 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             300.1300070 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                 -0.500 ppm
F2                 -150.07 Hz
PPMCM              0.43182 ppm/cm
HZCM             129.60159 Hz/cm
```




```
Current Data Parameters
NAME                  yls008-5
EXPNO                       30
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20080606
Time                     11.14
INSTRUM                  spect
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                       161.3
DW                       55.600 usec
DE                        6.00 usec
TE                       673.2 K
D1                    1.00000000 sec
MCREST                0.00000000 sec
MCWRK                 0.01500000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                        7.00 usec
PL1                      -1.00 dB
SFO1                300.1324010 MHz

F2 - Processing parameters
SI                       32768
SF                  300.1300084 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       22.00 cm
CY                       15.00 cm
F1P                       9.000 ppm
F1                     2701.17 Hz
F2P                      -0.500 ppm
F2                     -150.07 Hz
PPMCM                 0.43182 ppm/cm
HZCM                129.60159 Hz/cm
```



Current Data Parameters

NAME	yl5055-1
EXPNO	40
PROCNO	1

F2 - Acquisition Parameters

Date_	20080606
Time	11.19
INSTRUM	spect
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	181
DW	55.600 usec
DE	6.00 usec
TE	673.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

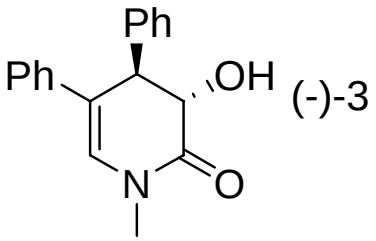
NUC1	1H
P1	7.00 usec
PL1	-1.00 dB
SFO1	300.1324010 MHz

F2 - Processing parameters

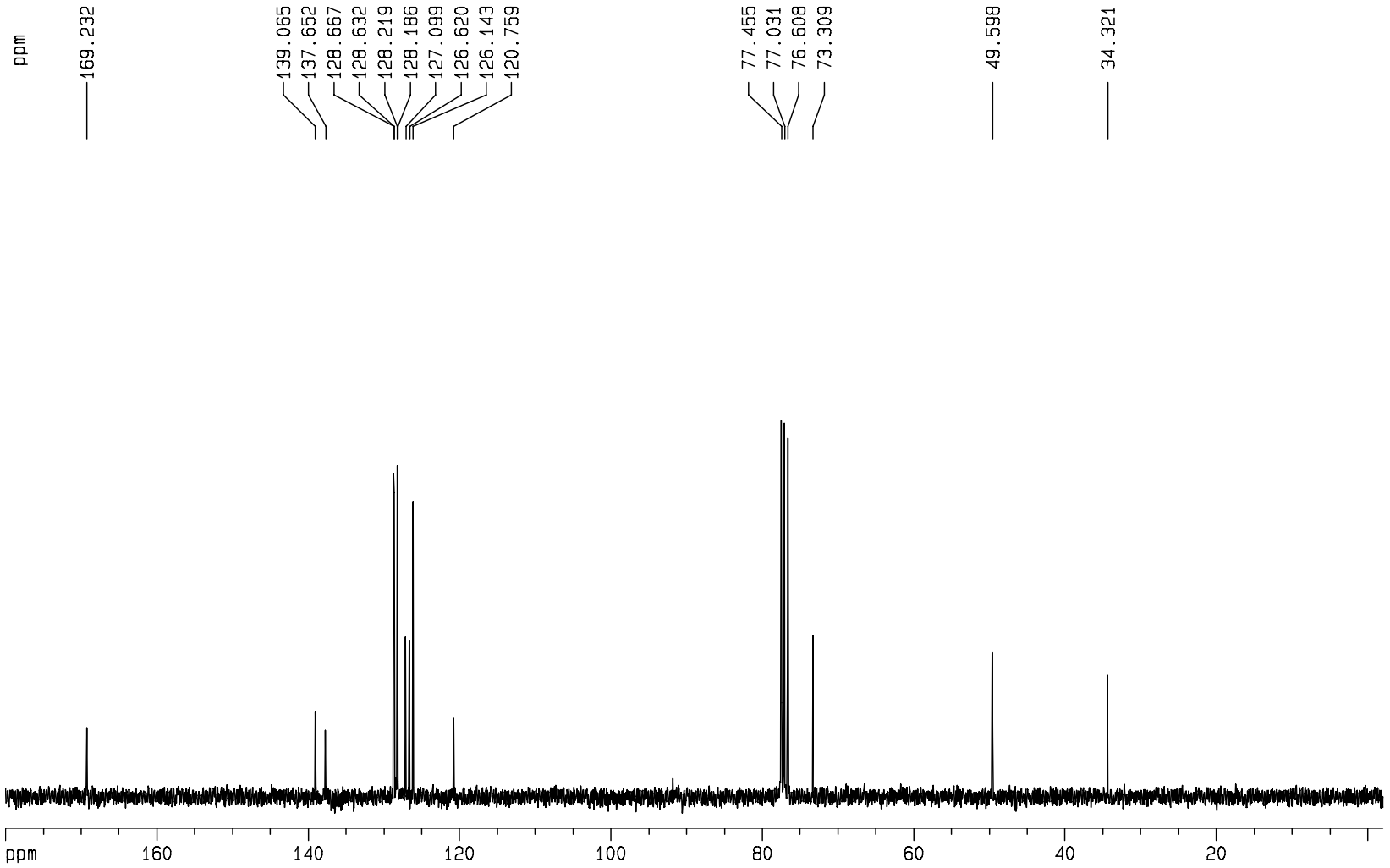
SI	32768
SF	300.1300084 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

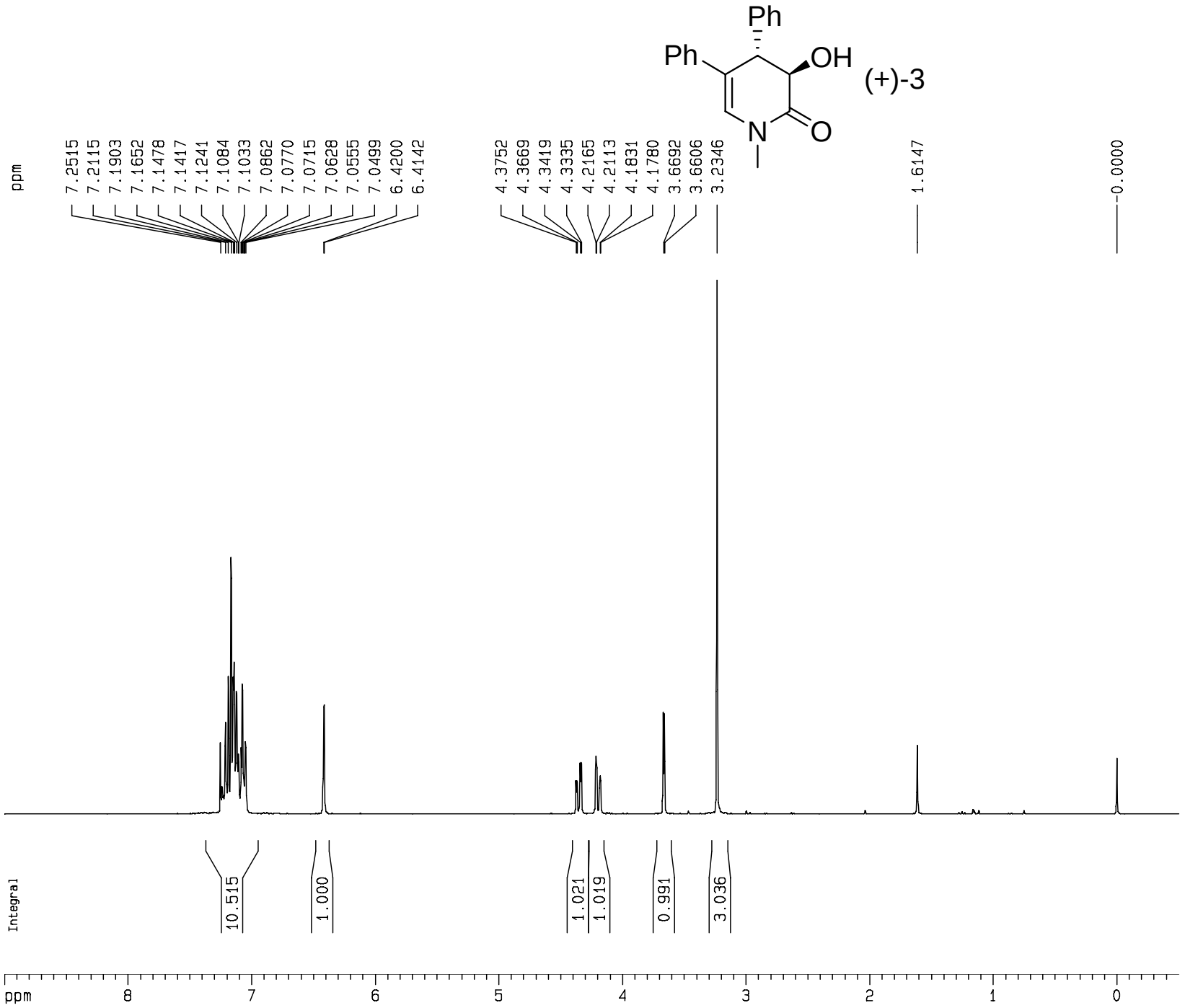
CX	22.00 cm
CY	10.00 cm
F1P	9.000 ppm
F1	2701.17 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.43182 ppm/cm
HZCM	129.60159 Hz/cm



(-)-3




```
Current Data Parameters
NAME            ylS055-1
EXPNO                 41
PROCNO                 1

F2 - Acquisition Parameters
Date_           20080606
Time                11.22
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   130
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                673.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -1.00 dB
PL12              20.16 dB
PL13              16.98 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                 6.00 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	ylR055-1
EXPNO	31
PROCNO	1

F2 - Acquisition Parameters

Date_	20080605
Time	20.34
INSTRUM	spect
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	256
DW	55.600 usec
DE	6.00 usec
TE	673.2 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

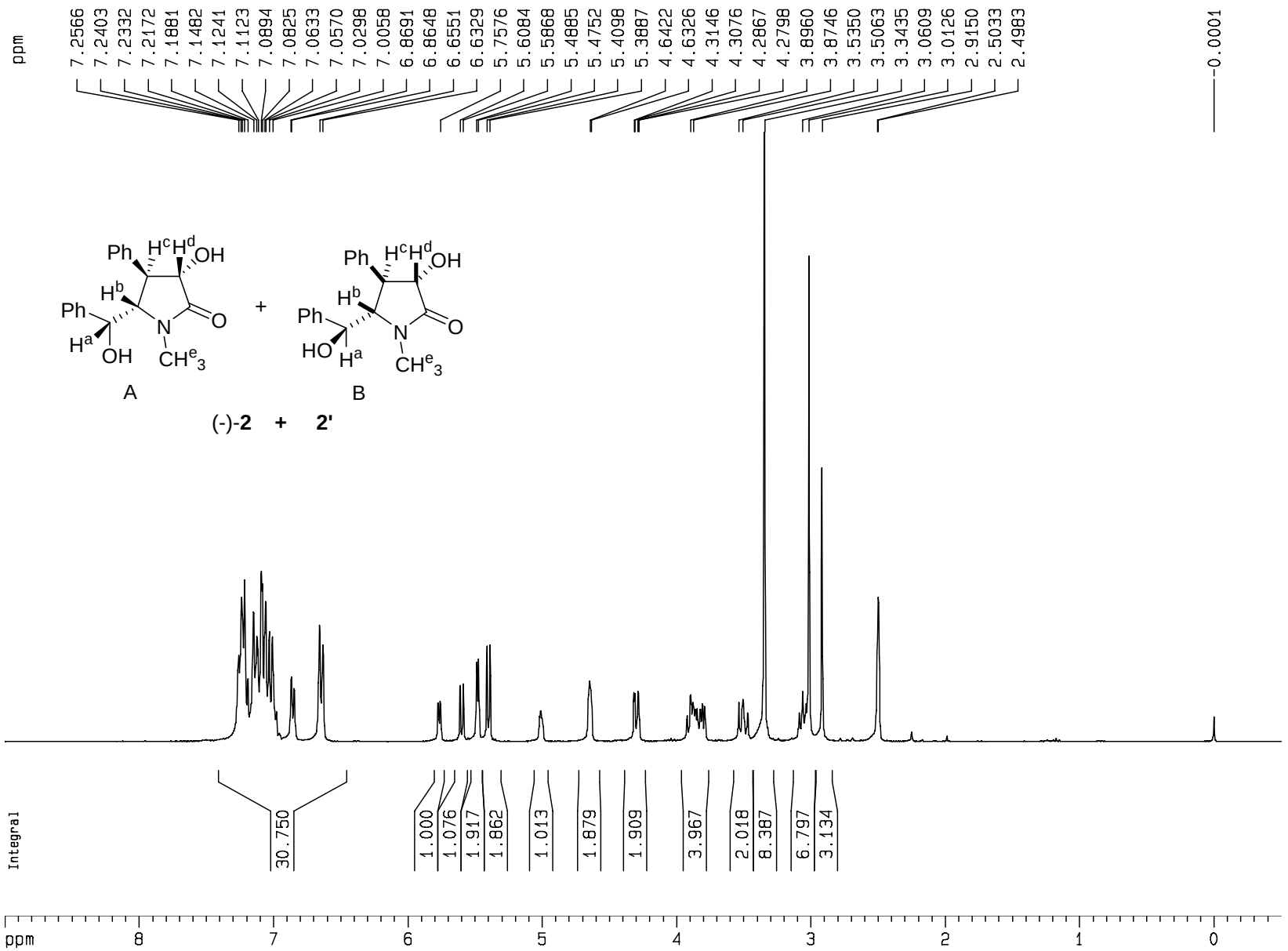
NUC1	1H
P1	7.00 usec
PL1	-1.00 dB
SFO1	300.1324010 MHz

F2 - Processing parameters

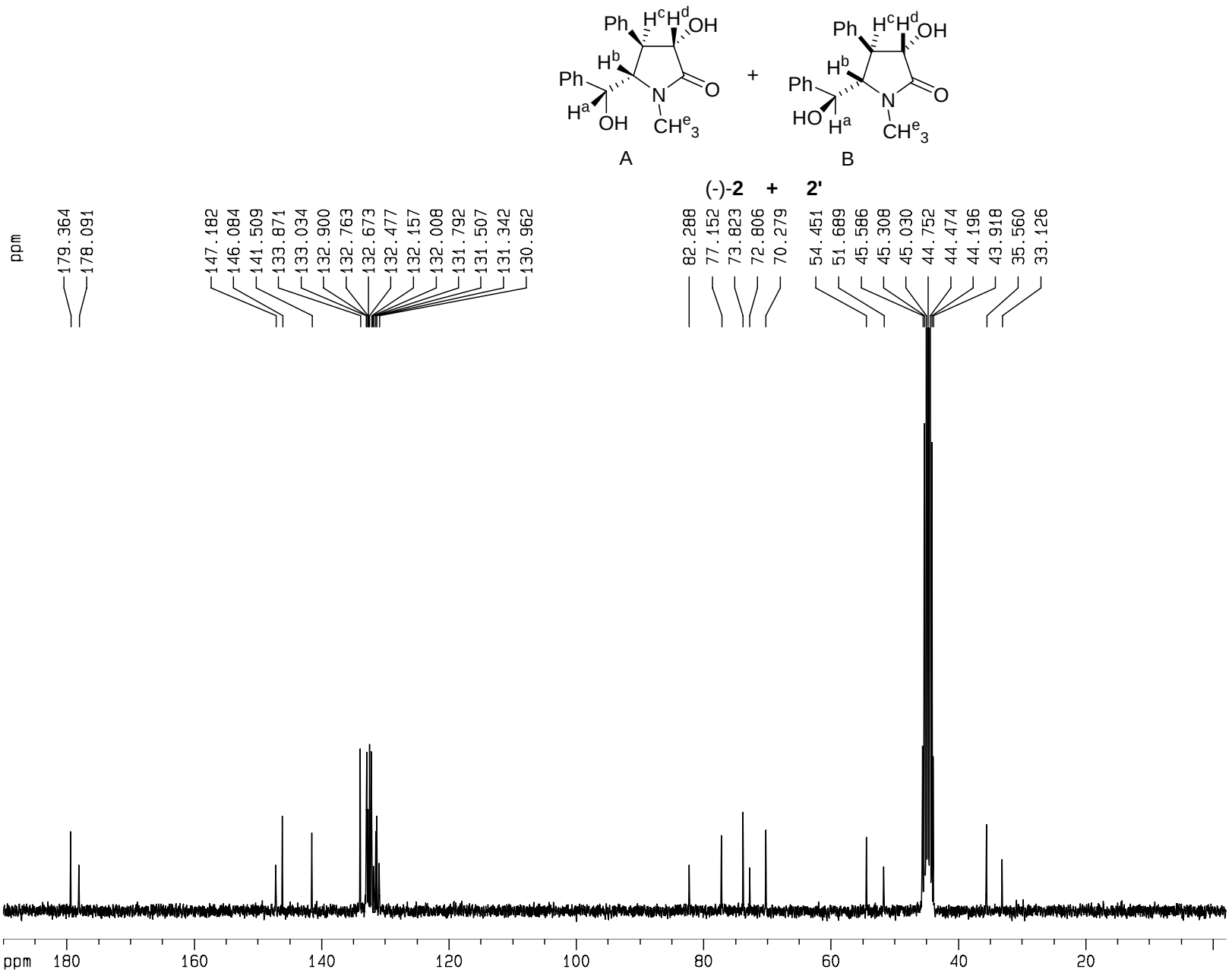
SI	32768
SF	300.1300086 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

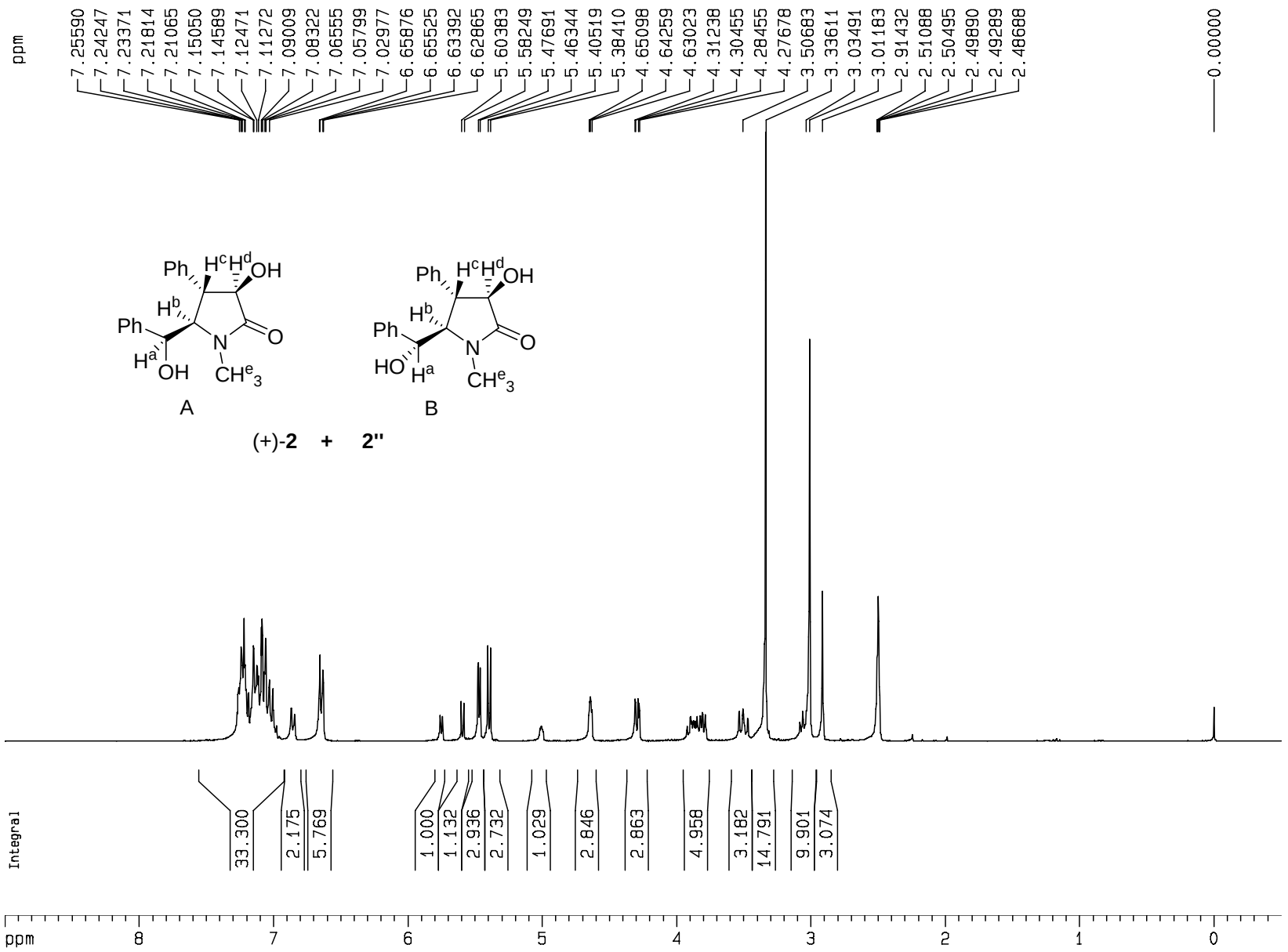
CX	22.00 cm
CY	10.00 cm
F1P	9.000 ppm
F1	2701.17 Hz
F2P	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.43182 ppm/cm
HZCM	129.60159 Hz/cm



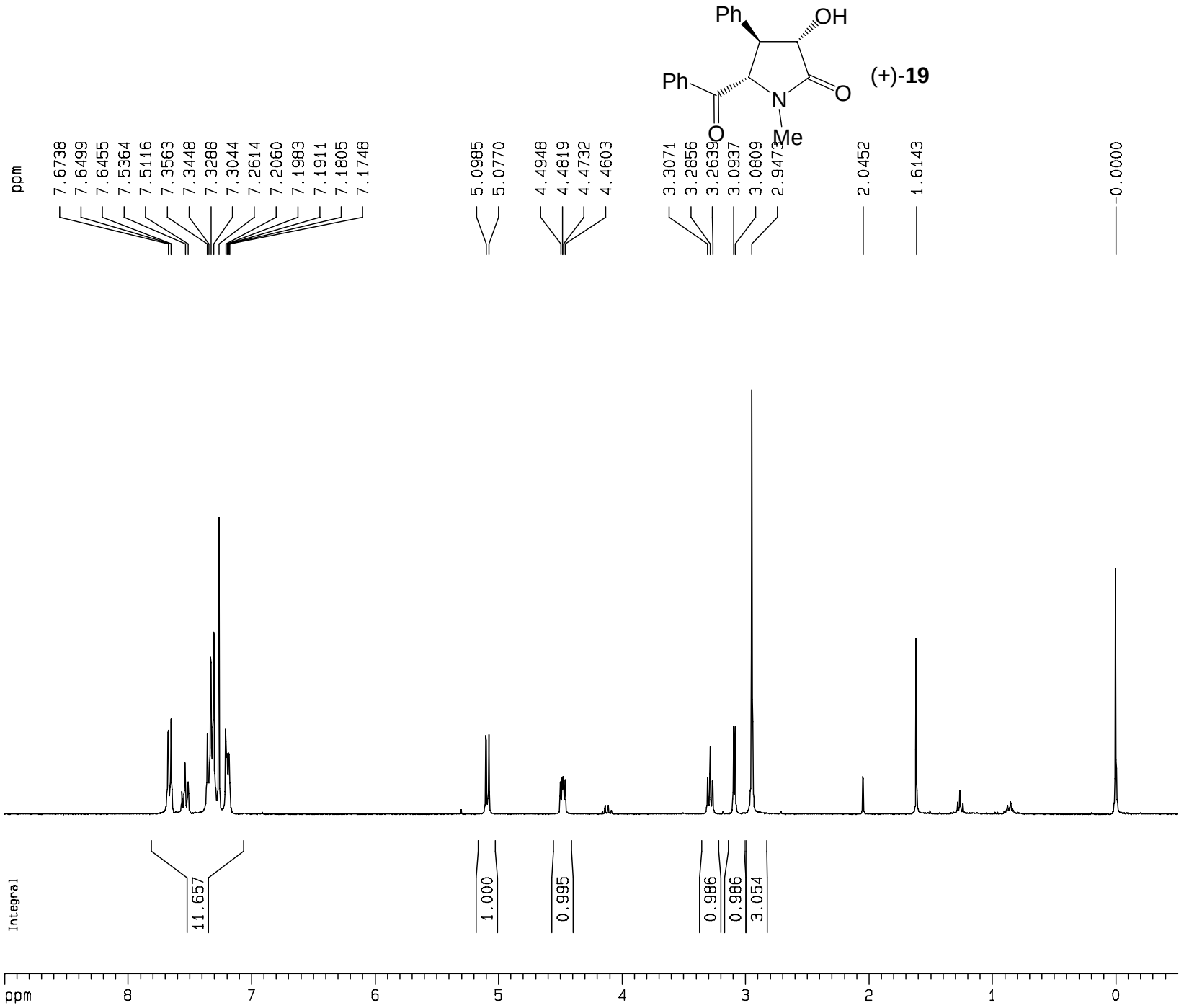

```
Current Data Parameters
NAME                  yl5052
EXPNO                     50
PROCNO                     1

F2 - Acquisition Parameters
Date_               20080612
Time                   11.20
INSTRUM                spect
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                     203.2
DW                    55.600 usec
DE                     6.00 usec
TE                    673.2 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     7.00 usec
PL1                   -1.00 dB
SFO1           300.1324010 MHz

F2 - Processing parameters
SI                     32768
SF             300.1314271 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     22.00 cm
CY                     16.00 cm
F1P                    9.000 ppm
F1                   2701.18 Hz
F2P                   -0.500 ppm
F2                   -150.07 Hz
PPMCM                0.43182 ppm/cm
HZCM               129.60222 Hz/cm
```




```
Current Data Parameters
NAME                  yls052
EXPNO                     51
PROCNO                     1

F2 - Acquisition Parameters
Date_              20080612
Time                  11.28
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     590
DS                       4
SWH              17985.611 Hz
FIDRES               0.274439 Hz
AQ                1.8219508 sec
RG                   5792.6
DW                   27.800 usec
DE                    6.00 usec
TE                   673.2 K
D1               2.00000000 sec
D11              0.03000000 sec
DELTA            1.89999998 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    12.50 usec
PL1                    2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                 80.00 usec
PL2                   -1.00 dB
PL12                  20.16 dB
PL13                  16.98 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
```




```
Current Data Parameters
NAME                  ylR052
EXPNO                     30
PROCNO                     1

F2 - Acquisition Parameters
Date_              20080611
Time                  20.36
INSTRUM               spect
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                       362
DW                    55.600 usec
DE                     6.00 usec
TE                    673.2 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     7.00 usec
PL1                   -1.00 dB
SFO1           300.1324010 MHz

F2 - Processing parameters
SI                     32768
SF             300.1300009 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                    -0.500 ppm
F2                   -150.07 Hz
PPMCM                0.43182 ppm/cm
HZCM               129.60159 Hz/cm
```



Current Data Parameters

NAME	yl5157-1
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20081120
Time	19.29
INSTRUM	spect
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	362
DW	55.600 usec
DE	6.00 usec
TE	297.5 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

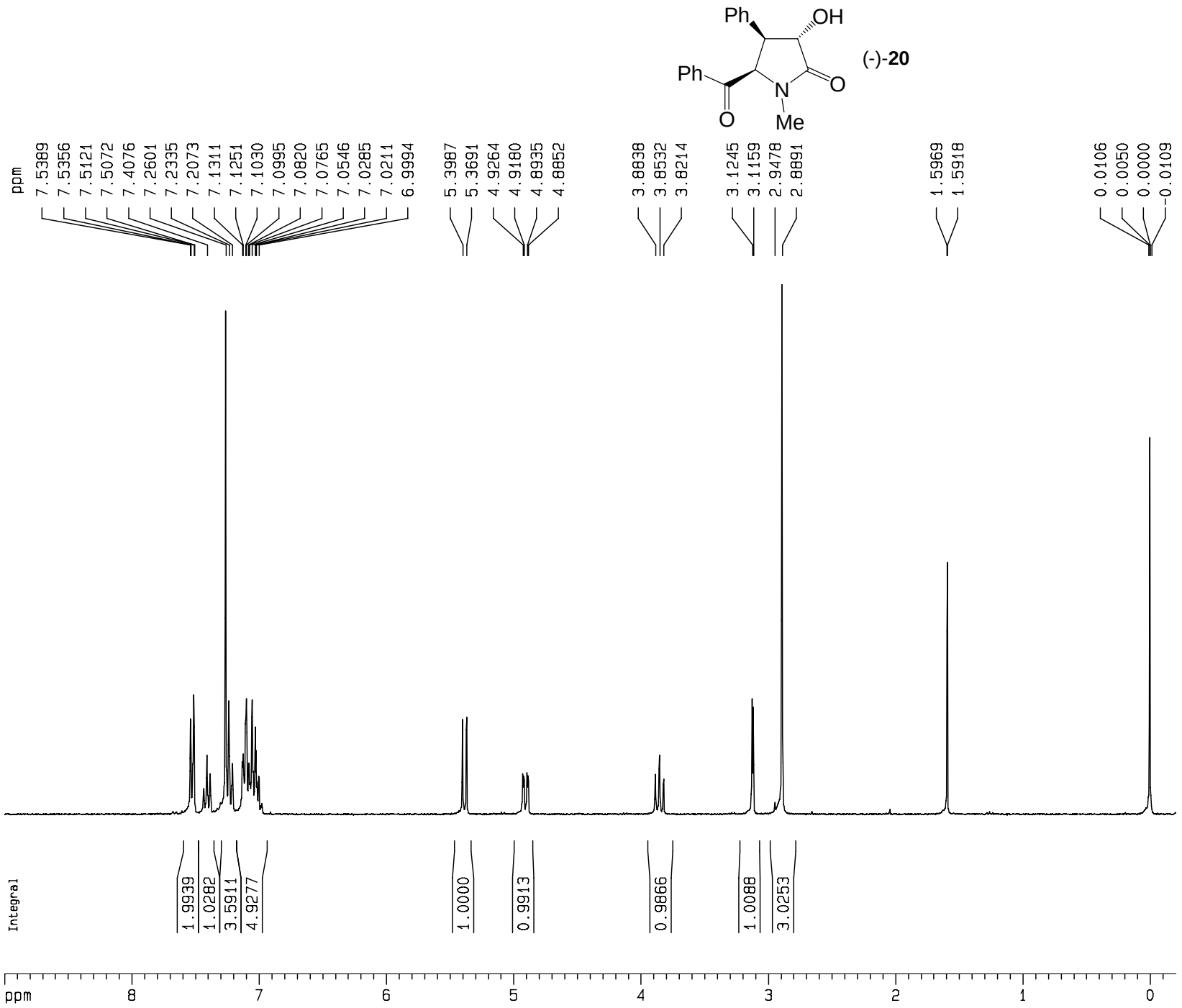
NUC1	1H
P1	7.00 usec
PL1	-1.00 dB
SFO1	300.1324010 MHz

F2 - Processing parameters

SI	32768
SF	300.1300055 MHz
WDW	EM
SSB	0
LB	0.30 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	-0.500 ppm
F2	-150.07 Hz
PPMCM	0.43182 ppm/cm
HZCM	129.60159 Hz/cm



Current Data Parameters

NAME	y1157-2
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20081016
Time	20.01
INSTRUM	spect
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	362
DW	55.600 usec
DE	6.00 usec
TE	300.4 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

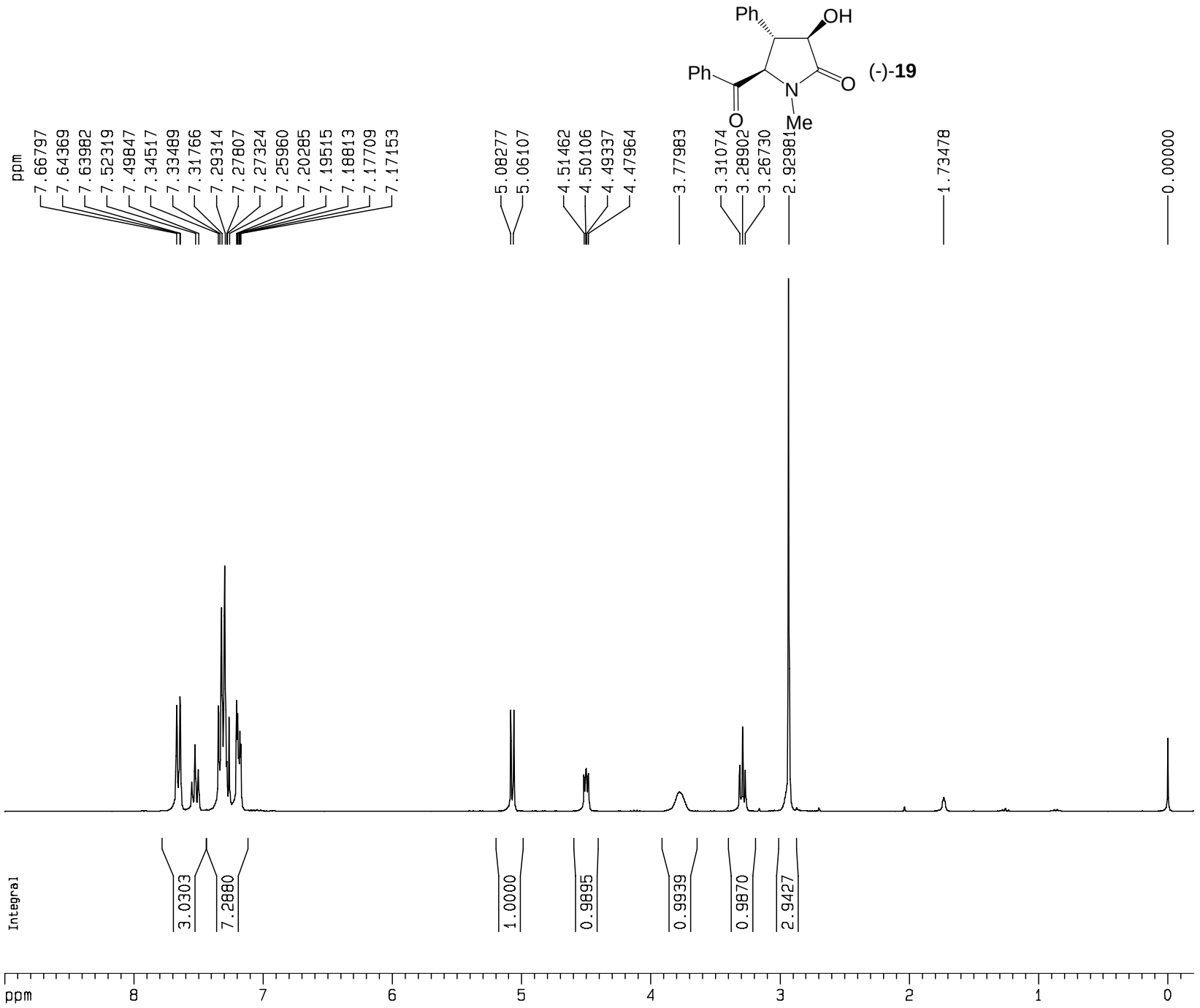
NUC1	1H
P1	7.00 usec
PL1	-1.00 dB
SFO1	300.1324010 MHz

F2 - Processing parameters

SI	32768
SF	300.1300058 MHz
WDW	EM
SSB	0
LB	0.30 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	y1157-1
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20081015
Time	20.04
INSTRUM	spect
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	362
DW	55.600 usec
DE	6.00 usec
TE	302.7 K
D1	1.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

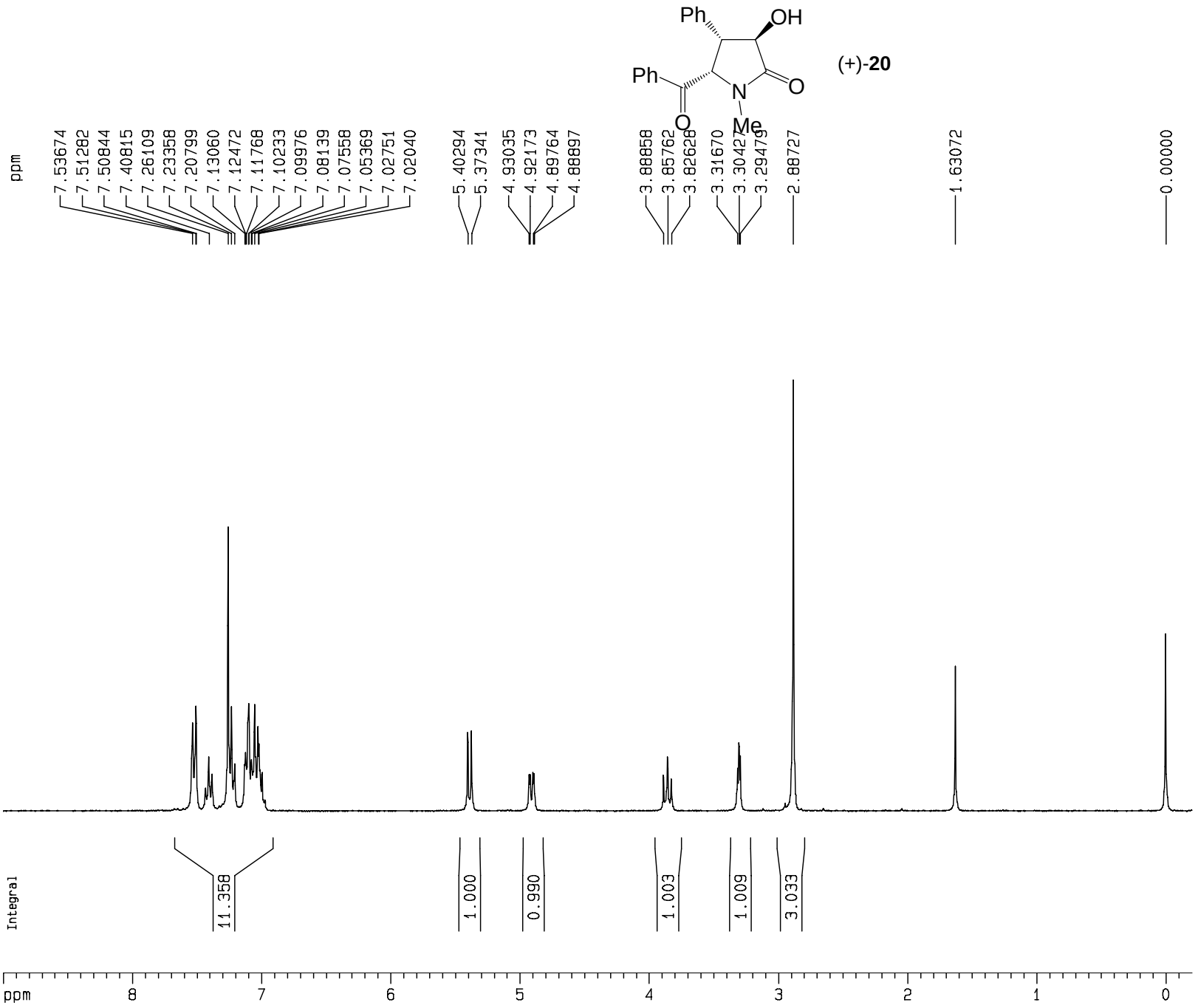
NUC1	1H
P1	7.00 usec
PL1	-1.00 dB
SFO1	300.1324010 MHz

F2 - Processing parameters

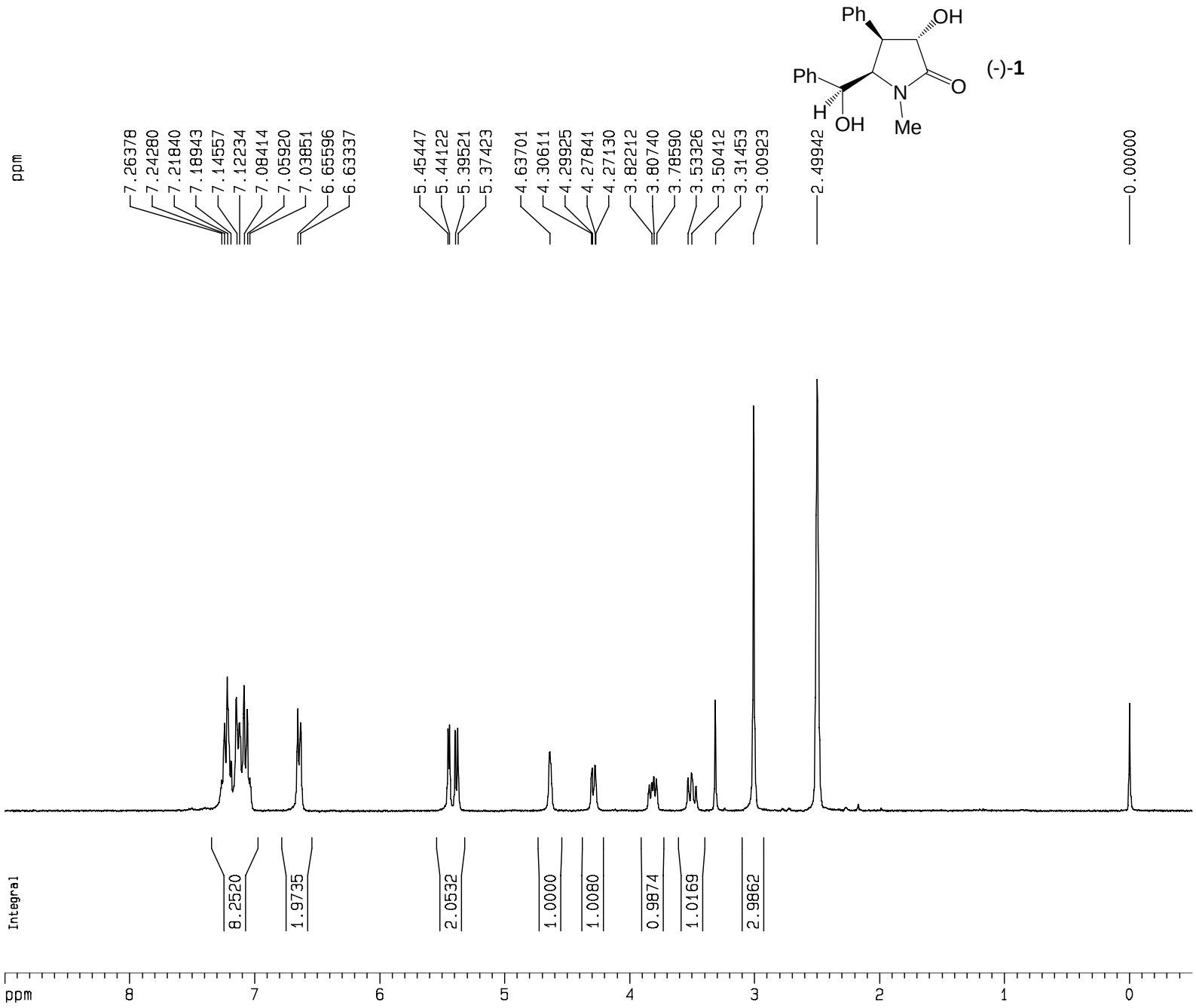
SI	32768
SF	300.1300061 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

1D NMR plot parameters

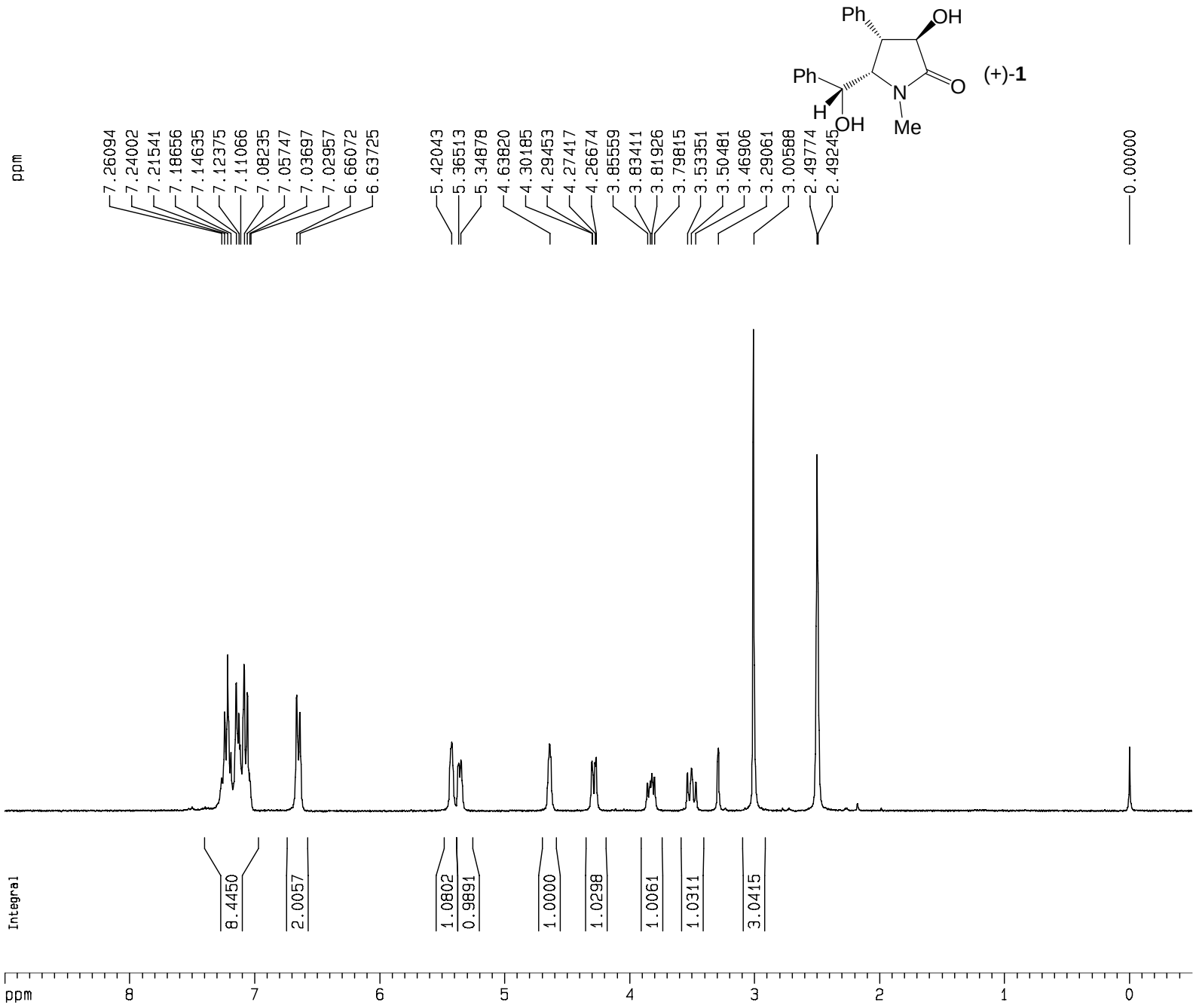
CX	22.00 cm
CY	10.00 cm
F1P	9.000 ppm
F1	2701.17 Hz
F2P	-0.200 ppm
F2	-60.03 Hz
PPMCM	0.41818 ppm/cm
HZCM	125.50892 Hz/cm



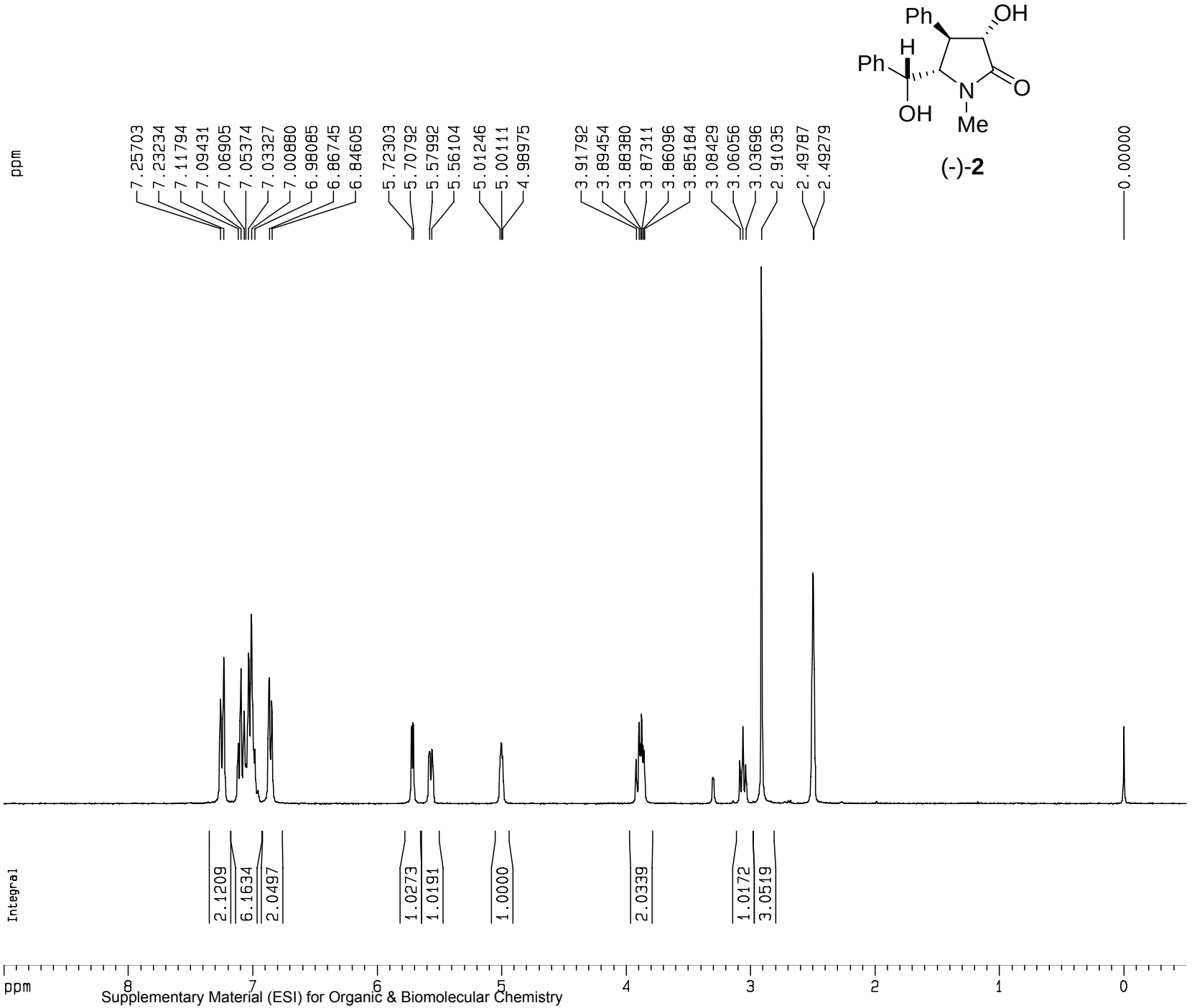

```
Current Data Parameters
NAME            ylR157-2
EXPNO                 10
PROCNO                 1

F2 - Acquisition Parameters
Date_           20081127
Time               19.26
INSTRUM            spect
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                   362
DW                55.600 usec
DE                 6.00 usec
TE                298.8 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.00 usec
PL1               -1.00 dB
SFO1         300.1324010 MHz

F2 - Processing parameters
SI                 32768
SF           300.1300056 MHz
WDW                   EM
SSB                    0
LB                  0.30 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                  y1S159
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_              20081127
Time                  19.31
INSTRUM               spect
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                       362
DW                   55.600 usec
DE                    6.00 usec
TE                   298.6 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    7.00 usec
PL1                  -1.00 dB
SFO1           300.1324010 MHz

F2 - Processing parameters
SI                     32768
SF             300.1300009 MHz
WDW                       EM
SSB                        0
LB                    0.30 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                  -0.500 ppm
F2                 -150.07 Hz
PPMCM              0.43182 ppm/cm
HZCM             129.60159 Hz/cm
```




```
Current Data Parameters
NAME                  y1R159
EXPNO                     10
PROCNO                     1

F2 - Acquisition Parameters
Date_               20081128
Time                   19.21
INSTRUM                spect
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                       362
DW                  55.600 usec
DE                   6.00 usec
TE                   299.6 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                   7.00 usec
PL1                  -1.00 dB
SFO1            300.1324010 MHz

F2 - Processing parameters
SI                       32768
SF                300.1300017 MHz
WDW                        EM
SSB                         0
LB                   0.30 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                   22.00 cm
CY                    9.00 cm
F1P                  9.000 ppm
F1                2701.17 Hz
F2P                 -0.500 ppm
F2                -150.07 Hz
PPMCM              0.43182 ppm/cm
HZCM             129.60159 Hz/cm
```



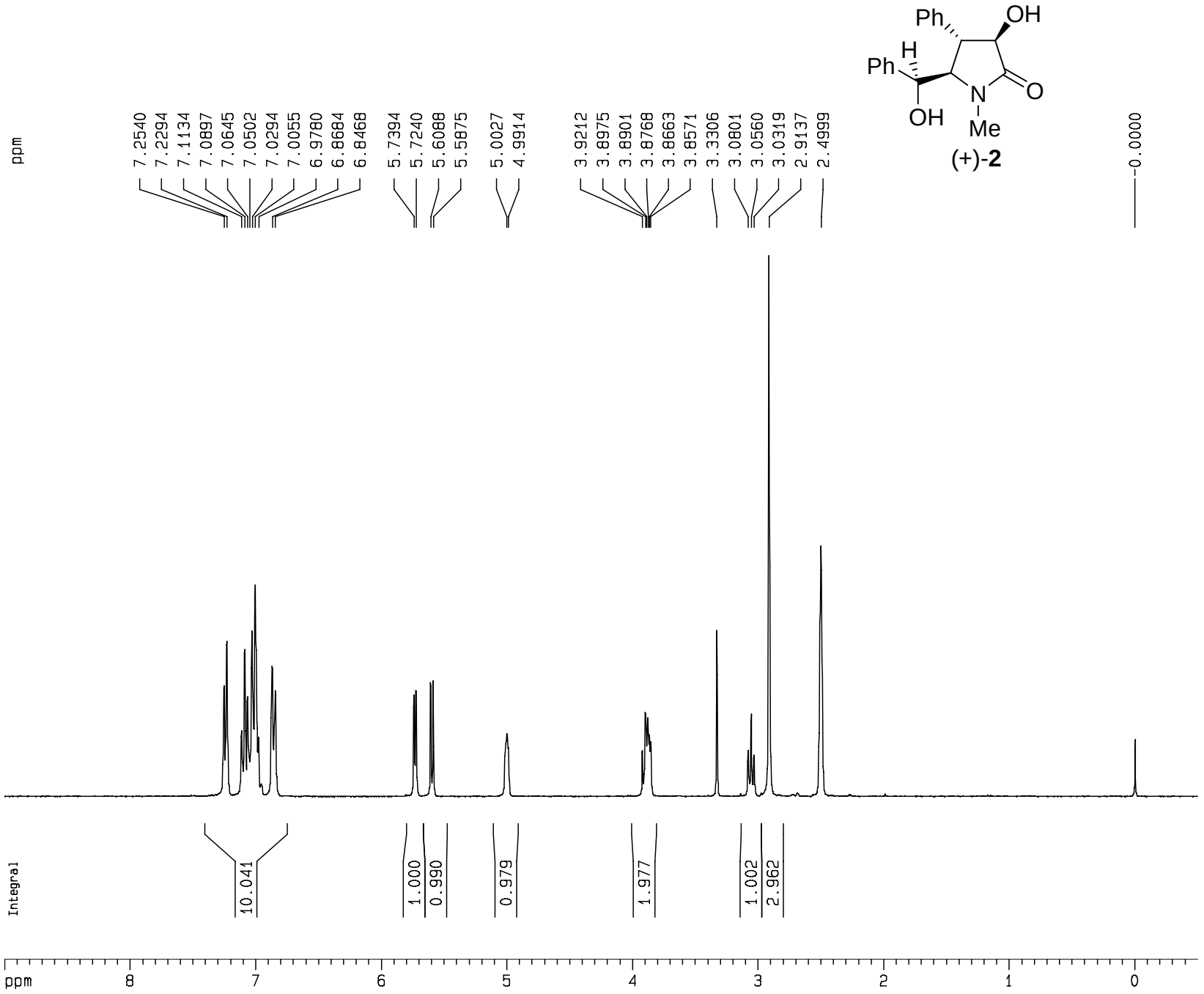
Current Data Parameters
NAME ylS158
EXPNO 20
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081223
Time 13.05
INSTRUM spect
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 362
DW 55.600 usec
DE 6.00 usec
TE 298.3 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.00 usec
PL1 -1.00 dB
SFO1 300.1324010 MHz

F2 - Processing parameters
SI 32768
SF 300.1300014 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

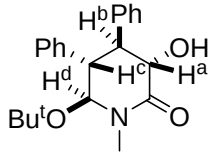
1D NMR plot parameters
CX 22.00 cm
CY 10.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.43182 ppm/cm
HZCM 129.60159 Hz/cm



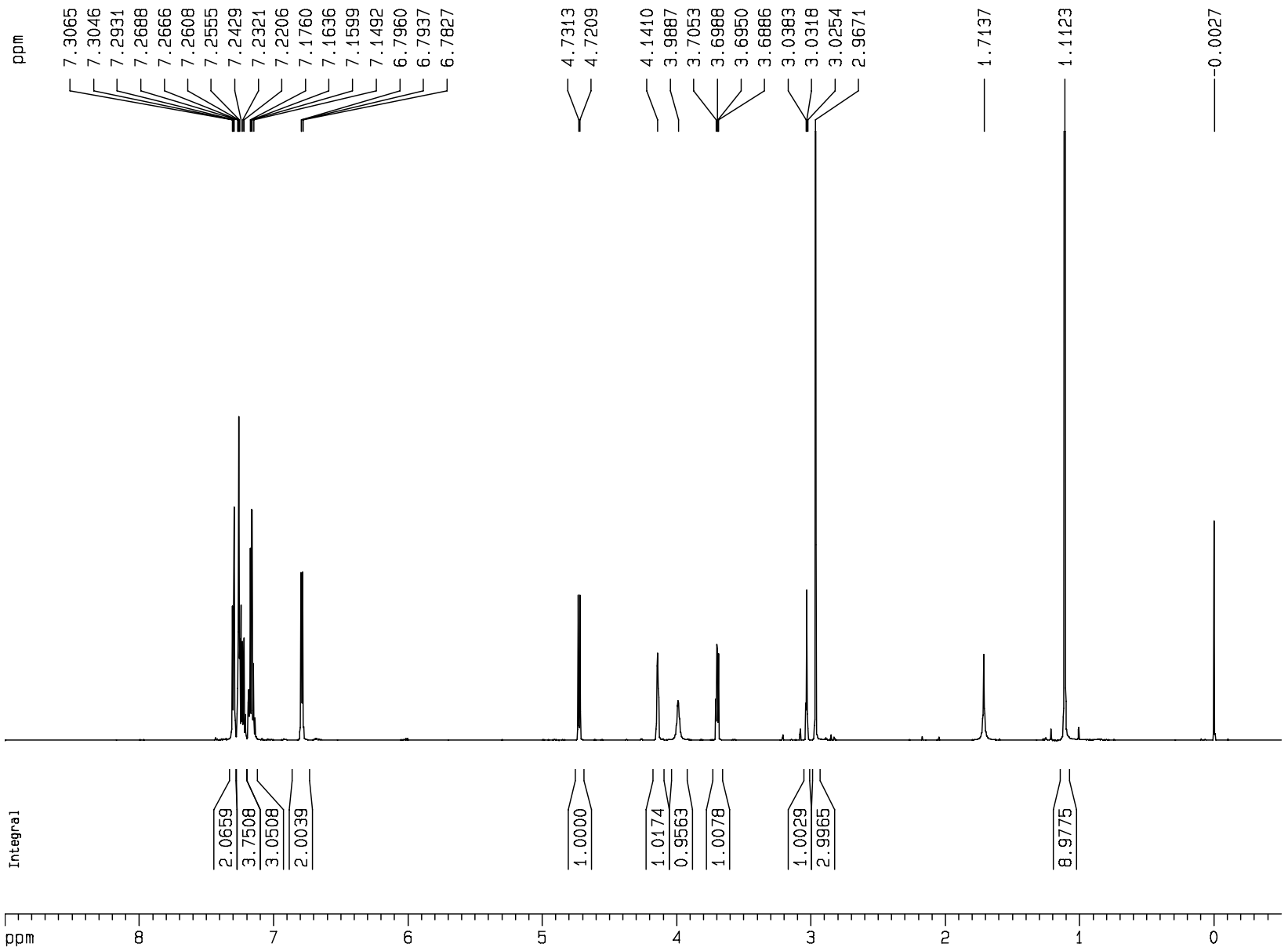
(+)-**2**

```
Current Data Parameters
NAME                  ylR158
EXPNO                     40
PROCNO                     1

F2 - Acquisition Parameters
Date_              20081223
Time                  20.18
INSTRUM               spect
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                       362
DW                  55.600 usec
DE                   6.00 usec
TE                   295.9 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    7.00 usec
PL1                  -1.00 dB
SFO1           300.1324010 MHz

F2 - Processing parameters
SI                       32768
SF             300.1300007 MHz
WDW                      EM
SSB                       0
LB                     0.30 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                  -0.500 ppm
F2                 -150.07 Hz
PPMCM              0.43182 ppm/cm
HZCM             129.60159 Hz/cm
```

YL-55-a



17



Current Data Parameters

NAME	noe
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20070816
Time	15.17
INSTRUM	av600
PROBHD	5 mm BBI 1H-BB
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	161.3
DW	27.800 usec
DE	6.00 usec
TE	291.9 K
D1	5.00000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

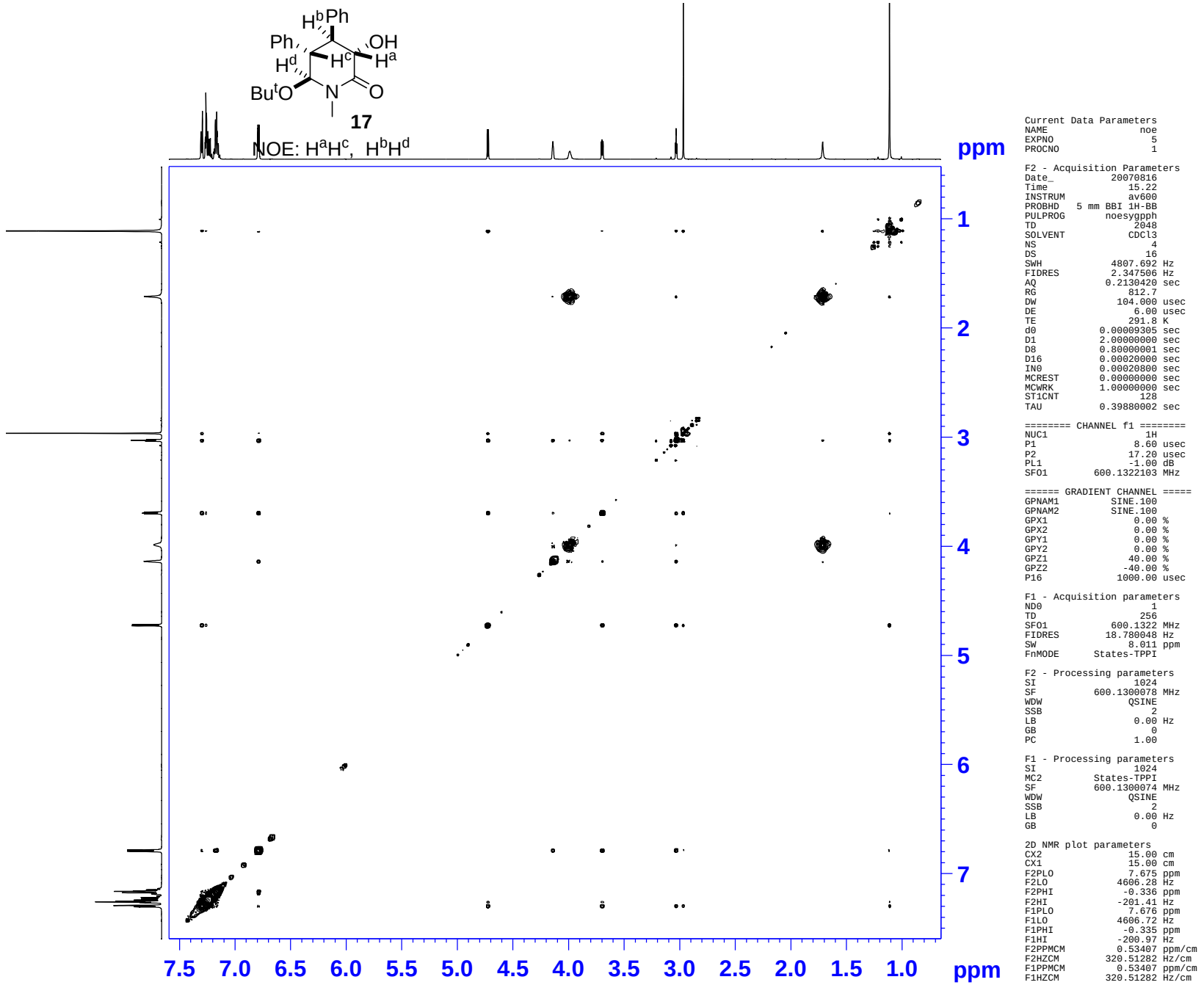
NUC1	1H
P1	8.60 usec
PL1	-1.00 dB
SFO1	600.1348010 MHz

F2 - Processing parameters

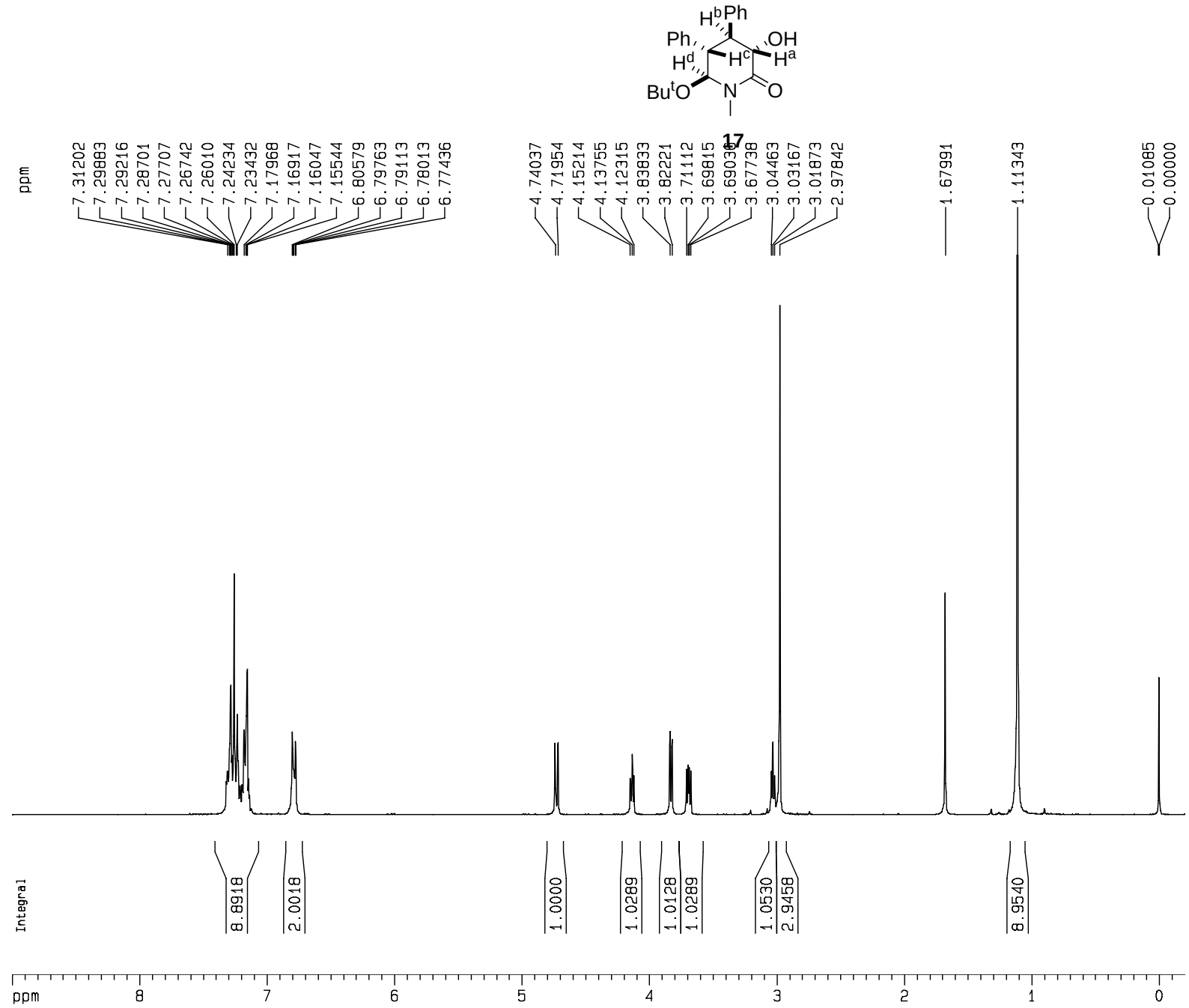
SI	32768
SF	600.1300088 MHz
WDW	EM
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	22.00 cm
CY	50.00 cm
F1P	9.000 ppm
F1	5401.17 Hz
F2P	-0.500 ppm
F2	-300.07 Hz
PPMCM	0.43182 ppm/cm
HZCM	259.14706 Hz/cm




```
Current Data Parameters
NAME                   noe
EXPNO                    5
PROCNO                   1

F2 - Acquisition Parameters
Date_             20070816
Time                 15.22
INSTRUM              av600
PROBHD   5 mm BBI 1H-BB
PULPROG         noesygpph
TD                   2048
SOLVENT              CDCl3
NS                       4
DS                      16
SWH              4807.692 Hz
FIDRES           2.347506 Hz
AQ               0.2130420 sec
RG                   812.7
DW                 104.000 usec
DE                   6.00 usec
TE                  291.8 K
d0               0.00009305 sec
D1               2.00000000 sec
D8               0.80000001 sec
D16              0.00020000 sec
IN0              0.00020800 sec
MCREST           0.00000000 sec
MCWRK            1.00000000 sec
ST1CNT                 128
TAU              0.39880002 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   8.60 usec
P2                  17.20 usec
PL1                 -1.00 dB
SFO1           600.1322103 MHz

====== GRADIENT CHANNEL =====
GPNAM1            SINE.100
GPNAM2            SINE.100
GPX1                 0.00 %
GPX2                 0.00 %
GPY1                 0.00 %
GPY2                 0.00 %
GPZ1                40.00 %
GPZ2               -40.00 %
P16               1000.00 usec

F1 - Acquisition parameters
ND0                      1
TD                     256
SFO1             600.1322 MHz
FIDRES          18.780048 Hz
SW                8.011 ppm
FnMODE       States-TPPI

F2 - Processing parameters
SI                    1024
SF             600.1300078 MHz
WDW                   QSINE
SSB                       2
LB                   0.00 Hz
GB                        0
PC                    1.00

F1 - Processing parameters
SI                    1024
MC2            States-TPPI
SF             600.1300074 MHz
WDW                   QSINE
SSB                       2
LB                   0.00 Hz
GB                        0

2D NMR plot parameters
CX2                  15.00 cm
CX1                  15.00 cm
F2PLO                7.675 ppm
F2LO               4606.28 Hz
F2PHI               -0.336 ppm
F2HI               -201.41 Hz
F1PLO                7.676 ppm
F1LO               4606.72 Hz
F1PHI               -0.335 ppm
F1HI               -200.97 Hz
F2PPMCM           0.53407 ppm/cm
F2HZCM          320.51282 Hz/cm
F1PPMCM           0.53407 ppm/cm
F1HZCM          320.51282 Hz/cm
```



Current Data Parameters

NAME	y1055-a
EXPNO	10
PROCNO	1

F2 - Acquisition Parameters

Date_	20070731
Time	8.27
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	203.2
DW	55.600 usec
DE	6.00 usec
TE	300.2 K
D1	5.00000000 sec

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

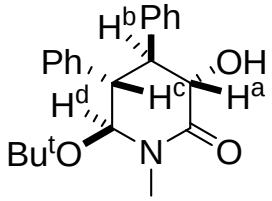
NUC1	1H
P1	10.60 usec
PL1	-1.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

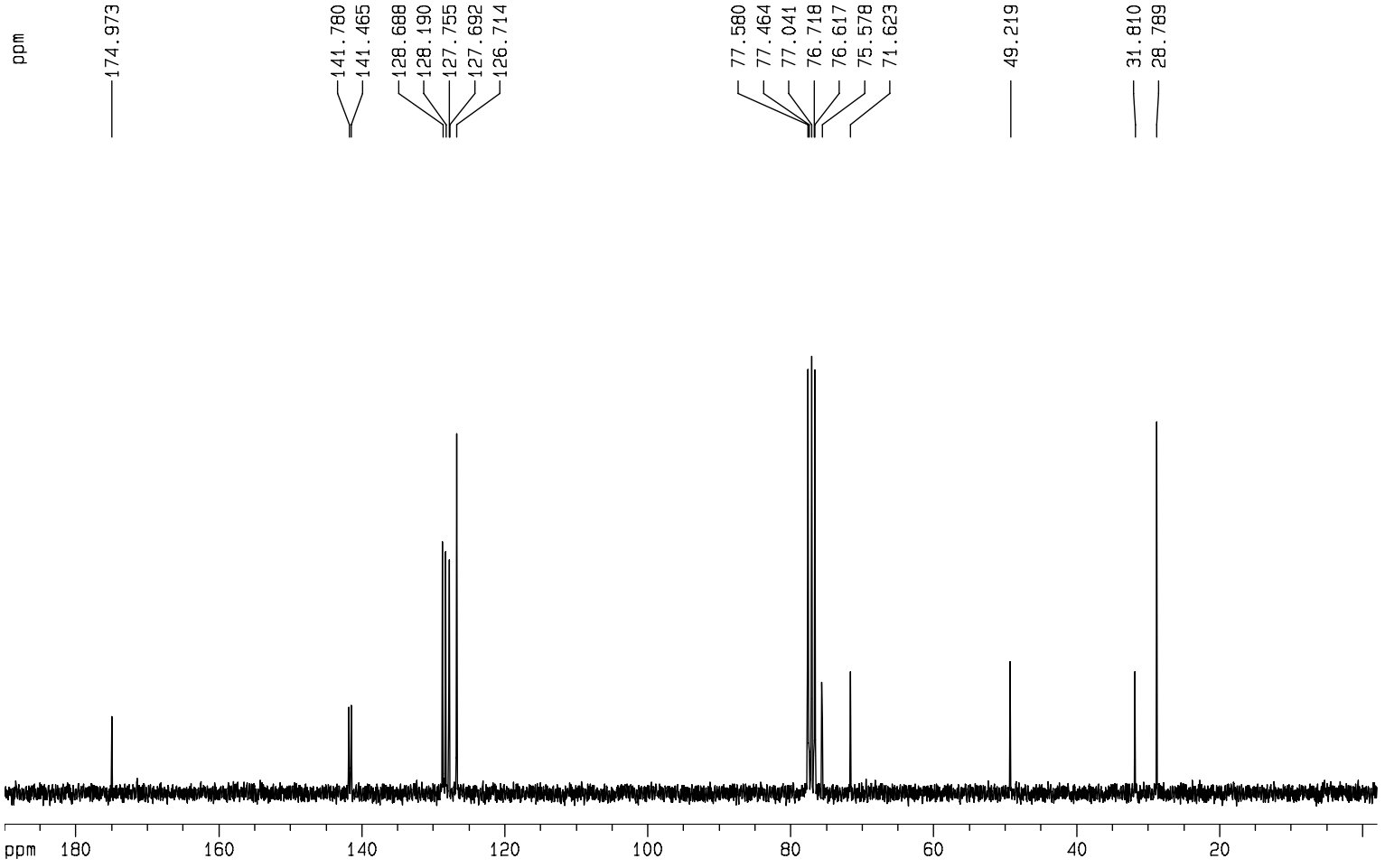
SI	32768
SF	300.1300062 MHz
WDW	EM
SSB	0
LB	0.35 Hz
GB	0
PC	1.00

1D NMR plot parameters

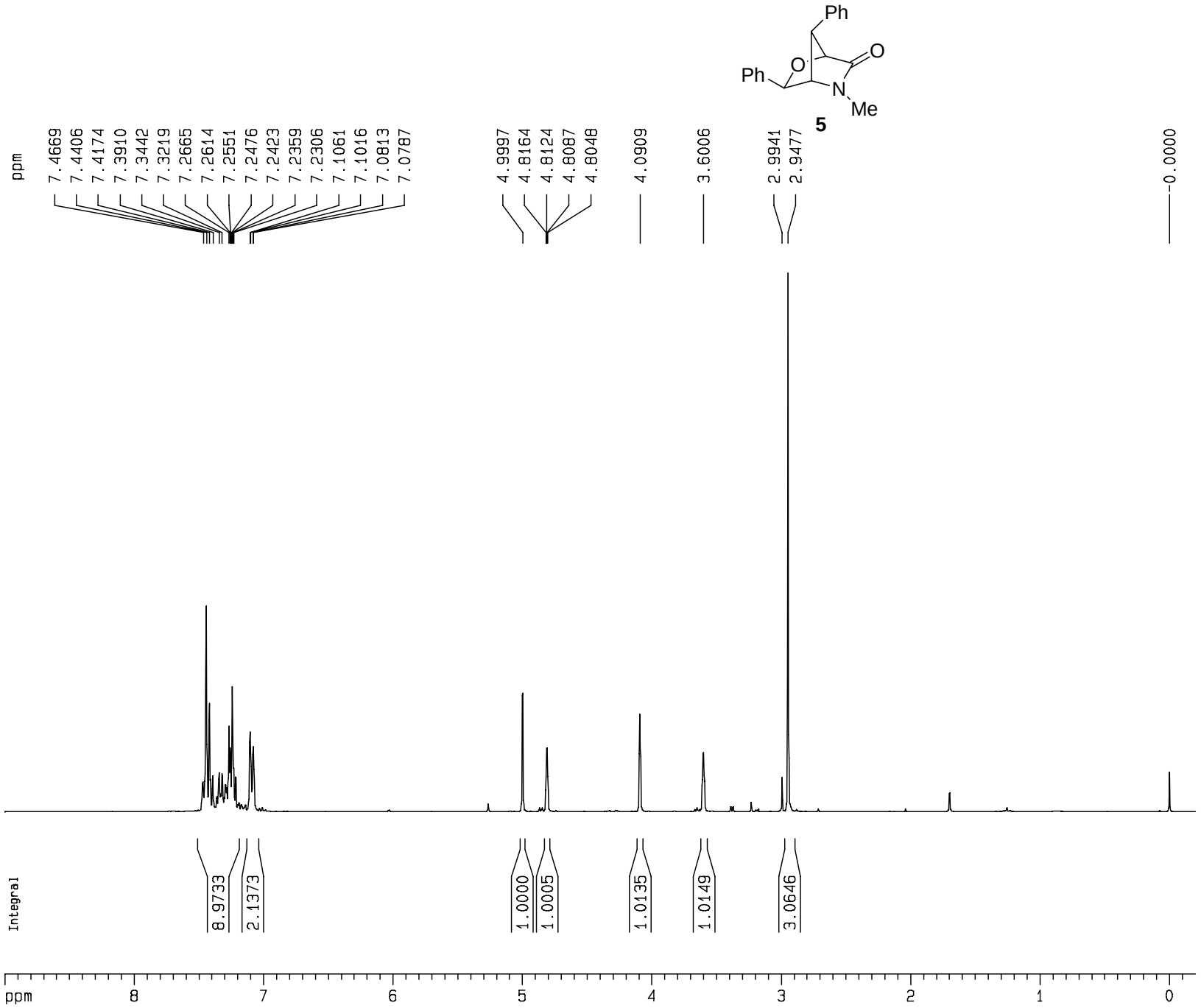
CX	22.00 cm
CY	30.00 cm
F1P	9.000 ppm
F1	2701.17 Hz
F2P	-0.200 ppm
F2	-60.03 Hz
PPMCM	0.41818 ppm/cm
HZCM	125.50892 Hz/cm



17




```
Current Data Parameters
NAME                  yl055-a
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_                20070511
Time                    11.22
INSTRUM                 av300
PROBHD   5 mm DUL 13C-1
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         157
DS                           4
SWH                 17985.611 Hz
FIDRES                0.274439 Hz
AQ                   1.8219508 sec
RG                        6502
DW                      27.800 usec
DE                       6.00 usec
TE                      298.9 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                        7.00 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
```



Current Data Parameters

NAME	y1055-0
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20070409
Time	11.48
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	228.1
DW	55.600 usec
DE	6.00 usec
TE	297.9 K
D1	5.00000000 sec

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

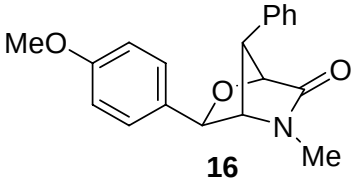
NUC1	1H
P1	9.30 usec
PL1	-1.00 dB
SFO1	300.1318534 MHz

F2 - Processing parameters

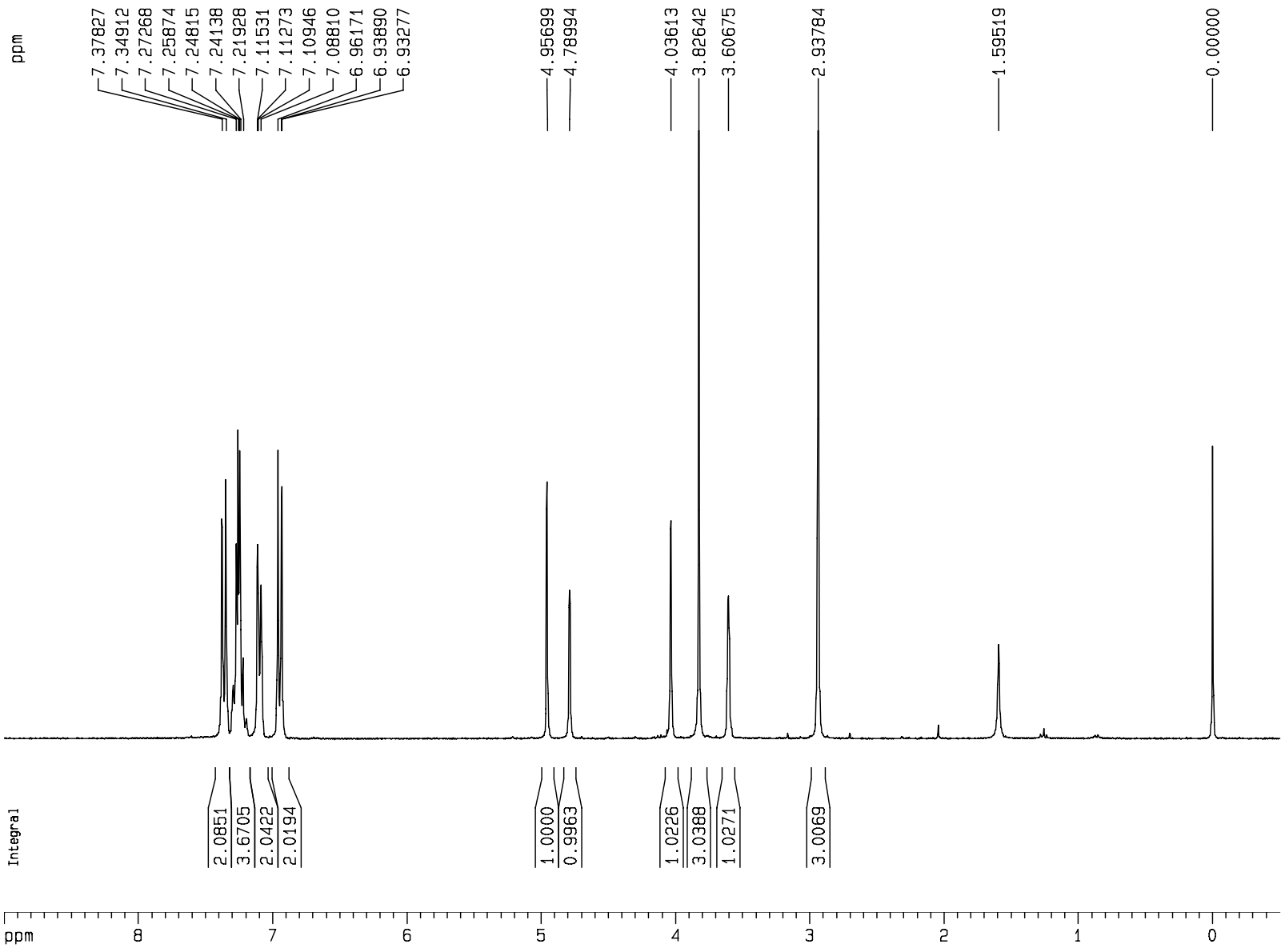
SI	32768
SF	300.1300077 MHz
WDW	EM
SSB	0
LB	0.35 Hz
GB	0
PC	1.00

1D NMR plot parameters

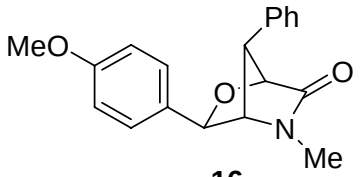
CX	22.00 cm
CY	10.00 cm
F1P	9.000 ppm
F1	2701.17 Hz
F2P	-0.200 ppm
F2	-60.03 Hz
PPMCM	0.41818 ppm/cm
HZCM	125.50892 Hz/cm



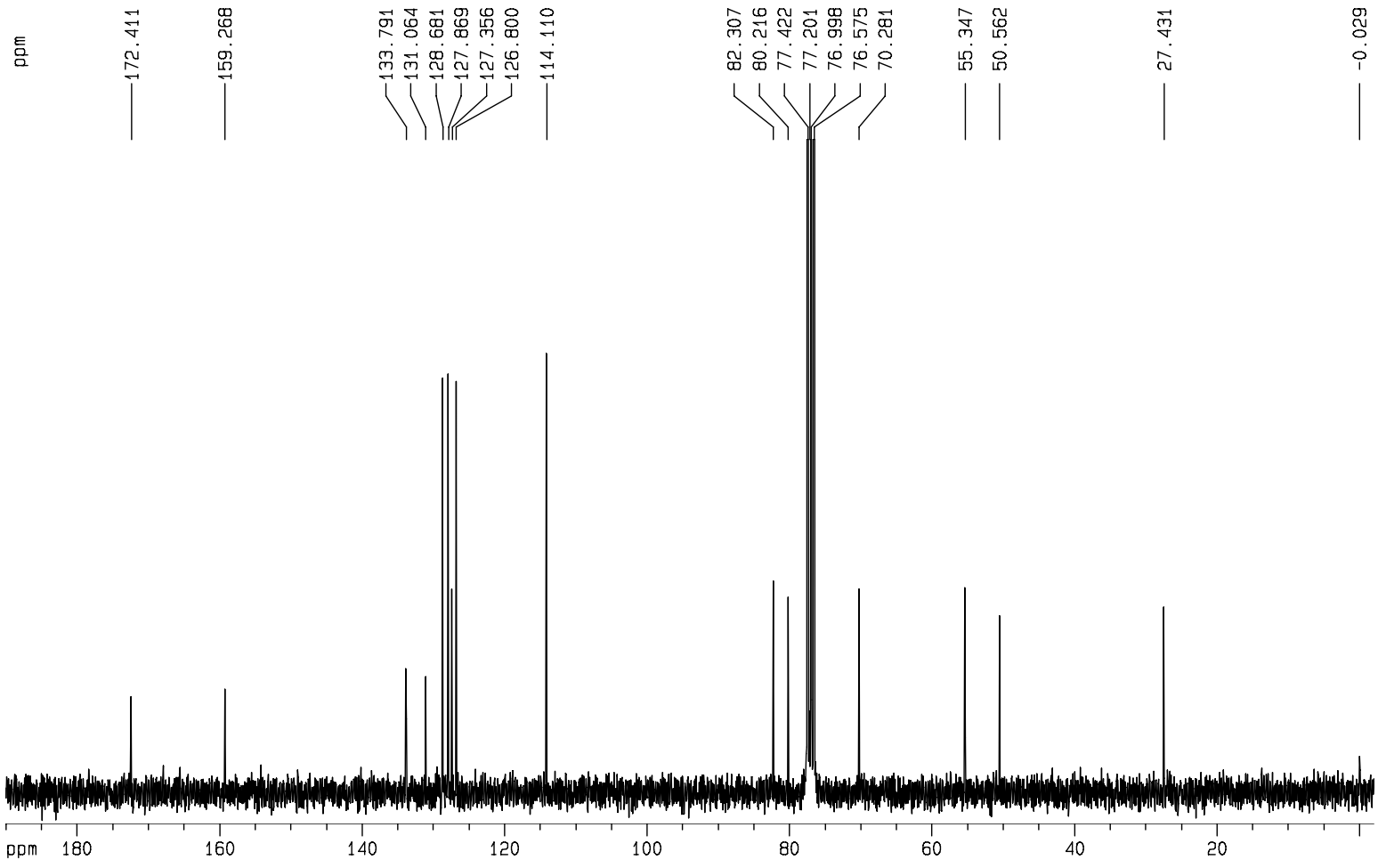
16



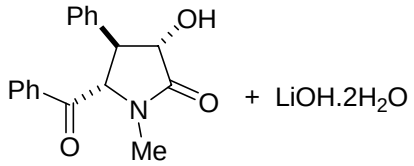

```
Current Data Parameters
NAME                  yl003-4-0
EXPNO                        10
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20070419
Time                      19.33
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                       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                  300.1300065 MHz
WDW                          EM
SSB                           0
LB                        0.35 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                      -0.500 ppm
F2                     -150.07 Hz
PPMCM                  0.43182 ppm/cm
HZCM                 129.60159 Hz/cm
```



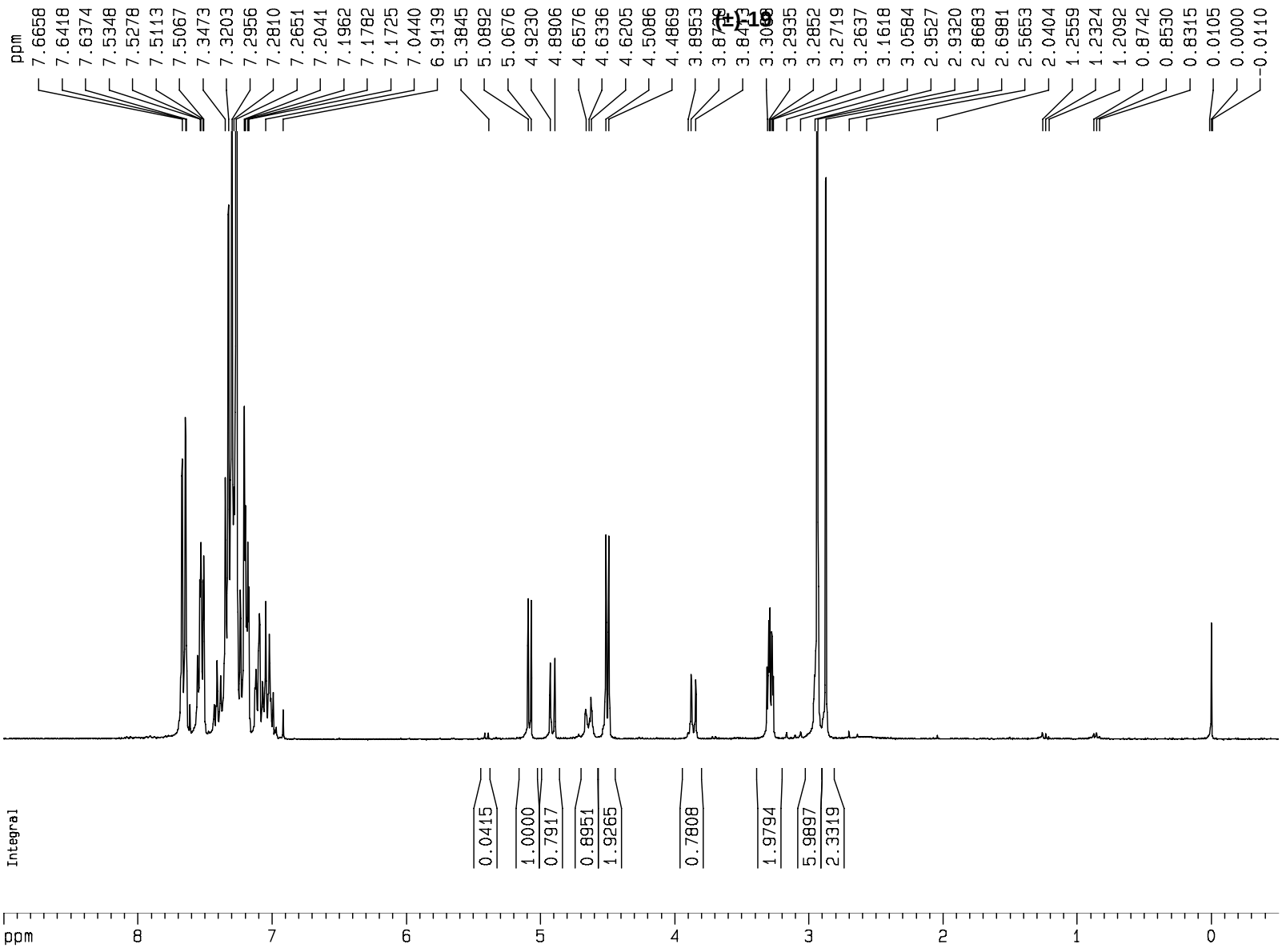
16



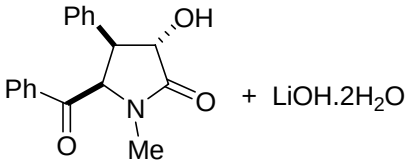

```
Current Data Parameters
NAME          yl003-4-0
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20070420
Time              11.33
INSTRUM           av300
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  717
DS                    4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG               1448.2
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                  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                30.00 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
```



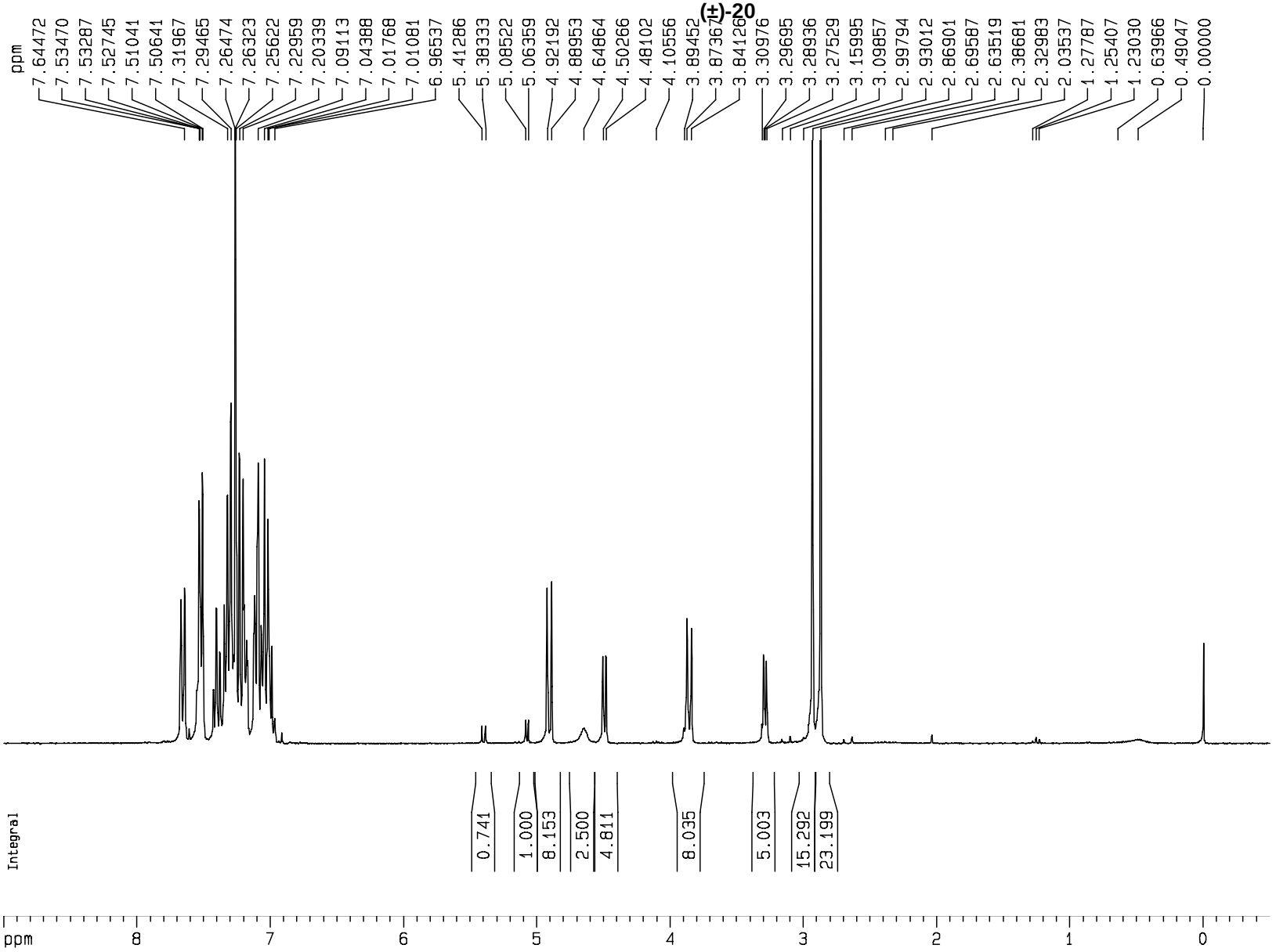
(±)-15




```
Current Data Parameters
NAME                  yl157-1
EXPNO                      30
PROCNO                      1

F2 - Acquisition Parameters
Date_               20081017
Time                    7.58
INSTRUM                 spect
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                     287.4
DW                    55.600 usec
DE                     6.00 usec
TE                     300.9 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     7.00 usec
PL1                   -1.00 dB
SFO1            300.1324010 MHz

F2 - Processing parameters
SI                     32768
SF              300.1300046 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     22.00 cm
CY                     90.00 cm
F1P                    9.000 ppm
F1                  2701.17 Hz
F2P                   -0.500 ppm
F2                  -150.07 Hz
PPMCM                0.43182 ppm/cm
HZCM               129.60159 Hz/cm
```

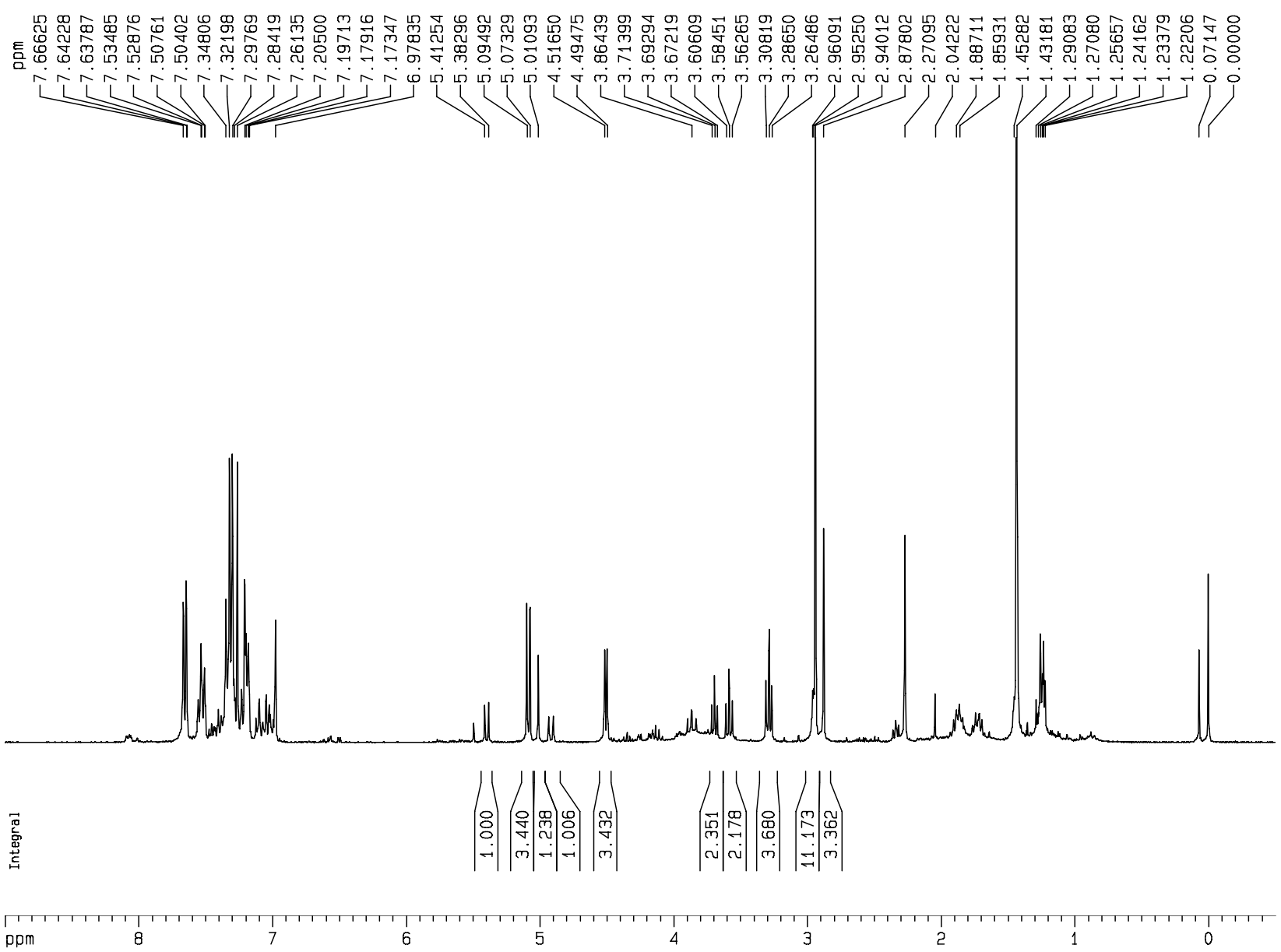


(±)-20




```
Current Data Parameters
NAME                  y1157-2
EXPNO                       9
PROCNO                      1

F2 - Acquisition Parameters
Date_              20081017
Time                   7.47
INSTRUM               spect
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                      362
DW                   55.600 usec
DE                    6.00 usec
TE                   302.9 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    7.00 usec
PL1                  -1.00 dB
SFO1            300.1324010 MHz

F2 - Processing parameters
SI                     32768
SF              300.1300047 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                  -0.500 ppm
F2                  -150.07 Hz
PPMCM               0.43182 ppm/cm
HZCM              129.60159 Hz/cm
```

After the conversion of (±)19 into (±)20, before chromatography.




```
Current Data Parameters
NAME                  yl157-1
EXPNO                       7
PROCNO                      1

F2 - Acquisition Parameters
Date_              20081026
Time                  10.32
INSTRUM               spect
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                     228.1
DW                    55.600 usec
DE                     6.00 usec
TE                     299.0 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      7.00 usec
PL1                    -1.00 dB
SFO1             300.1324010 MHz

F2 - Processing parameters
SI                      32768
SF               300.1300055 MHz
WDW                        EM
SSB                         0
LB                      0.30 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                   -0.500 ppm
F2                   -150.07 Hz
PPMCM                 0.43182 ppm/cm
HZCM                129.60159 Hz/cm
```