Supporting Information

Highly Enantioselective Synthesis of β-Amino Alcohols : A Catalytic Version

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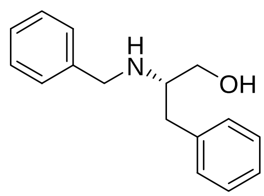
General

TLC was performed on pre-coated silica gel plates $60F_{254}$ and visualized either with a UV lamp (254 nm), or by using a solution of $KMnO_4/K_2CO_3/NaOH$ in water followed by heating. Flash chromatography was performed with Si60 silica gel (40–63 μm). Infrared (IR) spectra were recorded on a IRFT spectrometer and wave-numbers are indicated in cm^{-1}. ^1H NMR spectra were obtained using a 400 MHz spectrometer and data are reported as follows: chemical shift in ppm from tetramethylsilane as an internal standard, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or overlap of non equivalent resonances), integration. ^{13}C NMR spectra were obtained using a 100 MHz spectrometer and data are reported as follows: chemical shift in ppm with the solvent as an internal indicator ($CDCl_3$ δ 77.0 ppm), multiplicity with respect to proton (deduced from DEPT experiments, s = quaternary C, d = CH, t = CH_2, q = CH_3). Mass spectra were obtained by GC/MS with electron impact ionization at 70 eV. Microwave irradiation experiments were performed using a single-mode Initiator™ EXP (0–300W, 2.45 GHz) from Biotage.[1]

Synthesis of *N*,*N*-Dialkylamino alcohols

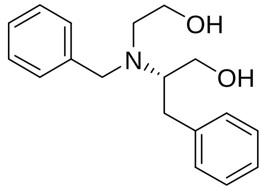


1a'

(*S*)-2-Benzylamino-3-phenylpropan-1-ol (1a').[2] To a solution of (*S*)-2-amino-3-phenylpropan-1-ol **1a** (500 mg, 3.3 mmol, 1.0 equiv) in CH_2Cl_2 (7 mL) was added 4Å molecular sieves (650 mg) and benzaldehyde (336 μL, 3.3 mmol, 1.0 equiv). After 3 h at rt, the suspension was filtered and concentrated under reduced pressure. The residue was dissolved in EtOH (7 mL) and $NaBH_4$ (164 mg, 4.4 mmol, 1.3 equiv) was added portionwise. After 18 h at rt, the reaction was hydrolyzed by addition of a saturated NH_4Cl solution and concentrated under reduced pressure. Treatment of the aqueous residue with NaOH (1N) was followed by extraction with CH_2Cl_2. The combined organic extracts were dried over $MgSO_4$, filtered and concentrated *in vacuo*. Purification of the crude residue by flash chromatography (silica gel, AcOEt/Et_3N : 99.5/0.5) afforded **1a'** (643 mg, 81%) as a white solid. $C_{16}H_{19}NO$; M = 241.15 g.mol^{-1}; M.p. 63–64 °C [lit.[2] M.p. 63–65 °C]; $[\alpha]^{20}_D$ = –11.7 (c 1.2, $CHCl_3$) [lit.[2] $[\alpha]^{20}_D$ = –9.0 (c 1.2, $CHCl_3$)]; IR (neat) 3700–2500, 1603, 1494, 1453, 1347, 1114, 1030, 741, 698 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.32–7.14 (10H), 3.77 (s, 2H), 3.65 (dd, *J* = 10.8, 3.9 Hz, 1H), 3.34 (dd, *J* = 10.8, 5.4 Hz, 1H), 2.96 (m, 1H), 2.85–2.73 (2H), 2.00 (bs, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.0 (s), 138.4 (s), 129.2 (d), 128.6 (d), 128.5 (d), 128.0 (d), 127.1 (d), 126.5 (d), 62.4 (t), 59.3 (d), 51.1 (t), 38.1 (t).

[1] For more information see: www.biotage.com

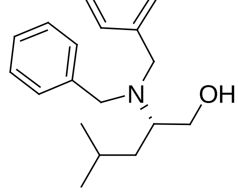
[2] McKay, C.; Wilson, R. J.; Rayner, C. M. *Chem. Commun.* **2004**, 1080–1081.



2a

(*S*)-2-[Benzyl-(2-hydroxyethyl)-amino]-3-phenylpropan-1-ol (2a).[3] To a solution of **1a'** (735 mg, 3.0 mmol, 1.0 equiv) and K_2CO_3 (1.26 g, 9.1 mmol, 3.0 equiv) in THF (25 mL) was added methylbromoacetate (577 µL, 6.1 mmol, 2.0 equiv). After 48 h at rt, water (25 mL) was added and the reaction mixture was extracted three times with EtOAc. The combined organic extracts were dried over $MgSO_4$, filtered and concentrated *in vacuo*. The residue was dissolved in THF (25 mL) and $LiAlH_4$ (146 mg, 3.8 mmol, 1.3 equiv) was added portionwise at 0 °C. After 2 h at rt, the reaction was hydrolyzed at 0 °C by dropwise addition of water (175 µL), NaOH (3.75 N; 175 µL) and water (415 µL). After 18 h at rt under stirring, the mixture was filtered over celite and concentrated *in vacuo*. Purification of the crude residue by flash chromatography (silica gel, CH_2Cl_2/MeOH : 95/5) afforded compound **2a** (538 mg, 62%) as a waxy solid. $C_{18}H_{23}NO_2$; M = 285.38 g.mol^{-1}; $[\alpha]^{20}_D$ = + 13.1 (*c* 1.2, MeOH) [lit.[3] $[\alpha]^{20}_D$ = –13.2 (*c* 1, MeOH) for *S* enantiomer]; IR (neat) 3600–3100, 2925, 2853, 1665, 1494, 1454, 1386, 1065, 1028, 1015, 747, 728, 741, 699 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30–7.15 (8H), 7.11–7.08 (2H), 3.84 (d, *J* = 13.7 Hz, 1H), 3,61 (d, *J* = 13.7 Hz, 1H), 3.57–3.37 (4H), 3.07 (m, 3H), 2.95–2.84 (2H), 2.63 (dt, *J* = 13.6, 3.8 Hz, 1H), 2.41 (dd, *J* = 13.5, 9.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.6 (s), 139.4 (s), 129.1 (d), 128.8 (d), 128.6 (d), 128.5 (d), 127.3 (d), 126.3 (d), 62.9 (d), 61.3 (t), 60.1 (t), 55.1 (t), 50.9 (t), 32.7 (t).

Dibenzylation : General procedure: A mixture of the amino alcohol (1 mmol, 1.0 equiv), benzyl bromide (262 µL, 2.2 mmol, 2.2 equiv), K_2CO_3 (415 mg, 3.0 mmol, 3.0 equiv), *n*-Bu_4NI (111 mg, 0.3 mmol, 0.3 equiv) in acetonitrile (10 mL), was stirred at reflux until the reaction is finished (TLC). The reaction media was concentrated under reduced pressure and the residue was dissolved in water (15 mL) and EtOAc (15 mL). Aqueous phase was extracted three times with EtOAc and the combined organic extracts were dried over $MgSO_4$, filtered and concentrated *in vacuo*. Purification of the crude residue by flash chromatography (silica gel, *n*-pentane/AcOEt) afforded the corresponding *N,N*-dibenzylamino alcohols.

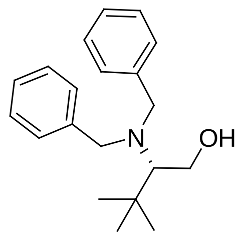


2d

(S)-2-*N,N*-Dibenzylamino-4-methylpentan-1-ol (2d).[4] (*n*-pentane/AcOEt : 90/10); 89% yield; yellow oil; $C_{20}H_{27}NO$; M = 297.43 g.mol^{-1}; $[\alpha]^{20}_D$ = + 86.4 (*c* 1, $CHCl_3$) [lit.[4] $[\alpha]_D$ = + 85.1 (*c* 1, $CHCl_3$)]; IR (neat) 3400–3200, 3027, 3951, 2866, 1494, 1454, 1364, 1028, 745, 728, 697 cm^{-1};

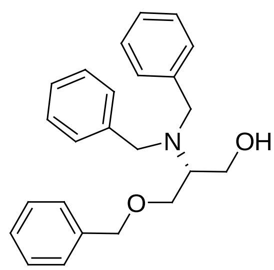
[3] Turgut, Y.; Sahin, E.; Togrul, M.; Hosgoren, H. *Tetrahedron: Asymmetry* **2004**, *15*, 1583–1588.
[4] Schwerdtfeger, J.; Kolczewski, S.; Weber, B.; Fröhlich, R.; Hoppe, D. *Synthesis* **1999**, *9*, 1573–1592.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33–7.21 (10H), 3.80 (d, J = 13.2 Hz, 2H), 3.48 (dd, J = 10.6, 5.1 Hz, 1H), 3.42 (d, J = 10.3 Hz, 1H), 3.37 (d, J = 13.2 Hz, 2H), 3.21 (bs, 1H), 2.84 (dddd, J = 10.0, 10.0, 5.0, 2.6 Hz, 1H), 1.56–1.46 (2H), 1.15 (dd, J = 9.9, 9.1 Hz, 1H), 0.92 (d, J = 6.3 Hz, 3H), 0.85 (d, J = 6.3 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.4 (s), 129.1 (d), 128.5 (d), 127.2 (d), 61.1 (t), 56.9 (d), 53.1 (t), 34.0 (t), 25.5 (d), 24.1 (q), 22.1 (q); MS-EI *m/z* (%): 297 ($M^{+\bullet}$, 0.1), 266 ($M^{+\bullet}$–$CH_2OH^\bullet$, 91), 250 (32), 238 (16), 210 (7), 181 (9), 91 (100).



2e

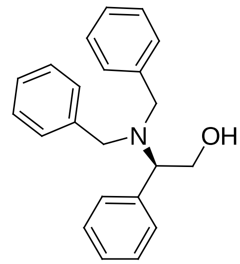
(*S*)-2-*N*,*N*-Dibenzylamino-3,3-dimethylbutan-1-ol (2e).[5] (hexane/AcOEt : 90/10); 75% yield; yellow oil; $C_{20}H_{27}NO$; M = 297.43 g.mol^{-1}; $[\alpha]_D^{20}$ = – 38.0 (*c* 0.5, $CHCl_3$); IR (neat) 3405, 3027, 2951, 1494, 1478, 1453, 1359, 1127, 1073, 1026, 998, 745, 697 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32–7.21 (10H), 3.97 (d, J = 13.3 Hz, 2H), 3.87 (d, J = 13.3 Hz, 2H), 3.71 (m, 2H), 2.65 (dd, J = 9.7, 4.2 Hz, 1H), 2.25 (bs, 1H), 1.03 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 140.1 (s), 129.3 (d), 128.4 (d), 127.1 (d), 66.9 (d), 59.2 (t), 55.2 (t), 36.6 (s), 29.2 (q); MS-EI *m/z* (%): 266 ($M^{+\bullet}$–$CH_2OH^\bullet$, 7), 240 ($M^{+\bullet}$–*t*$Bu^\bullet$, 53), 210 (4), 181 (8), 91 (100); Anal Calcd for $C_{20}H_{27}NO$: C, 80.76 ; H, 9.15 ; N, 4.71. Found : C, 80.72 ; H, 9.24 ; N, 4.63.



2g

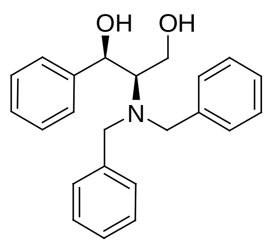
(*R*)-2-*N*,*N*-Dibenzylamino-3-*O*-benzyl-1,3-propanediol (2g).[6,7] (*n*-pentane/AcOEt : 90/10); 76% yield; colorless oil; $C_{24}H_{27}NO_2$; M = 361.48 g.mol^{-1}; $[\alpha]_D^{20}$ = + 90.3 (*c* 1.5, $CHCl_3$) [lit.[7] $[\alpha]^{20}_D$ = + 65.4 (*c* 1, EtOAc)]; IR (neat) 3430, 3027, 2854, 1494, 1452, 1361, 1071, 1026, 731, 696 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41–7.21 (15H), 4.55 (d, J = 12.1 Hz, 1H), 4.51 (d, J = 12.1 Hz, 1H), 3.87 (d, J = 13.3 Hz, 2H), 3.74 (dd, J = 9.9, 6.3 Hz, 1H), 3.60 (d, J = 13.3 Hz, 2H), 3.57–3.50 (3H), 3.15 (m, 1H), 2.87 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.5 (s), 138.1 (s), 129.0 (d), 128.5 (d), 128.4 (d), 127.7 (d), 127.5 (d), 127.2 (d), 73.4 (t), 67.9 (t), 59.7 (t), 58.2 (d), 54.1 (t); MS-EI *m/z* (%): 330 ($M^{+\bullet}$–$CH_2OH^\bullet$, 11), 240 (26), 181 (6), 91 (100).

[5] Brunin, T.; Cabou, J.; Bastin, S.; Brocard, J.; Pélinski, L. *Tetrahedron: Asymmetry* **2002**, *13*, 1241–1243.
[6] Weber, K.; Kuklinski, S.; Gmeiner, P. *Org. Lett.* **2000**, *2*, 647–649.
[7] Dix, D.; Imming, P. *Arch. Pharm. (Weinheim)* **1995**, *328*, 203–205.



2h

(*R*)-2-*N*,*N*-Dibenzylamino-2-phenylethanol (2h).[4,8] (hexane/AcOEt : 95/5); 79% yield; colorless oil; $C_{22}H_{23}NO$; M = 317.42 $g.mol^{-1}$; $[\alpha]_D^{20}$ = – 149.4 (*c* 2.5, $CHCl_3$) [lit.[4] $[\alpha]_D$ = + 148.6 (*c* 1, $CHCl_3$) for *S* isomer]; IR (neat) 3439, 3027, 2837, 1493, 1453, 1072, 1026, 745, 730, 696 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.45–7.24 (15H), 4.14 (dd, *J* = 10.6, 10.6 Hz, 1H), 3.96–3.90 (3H), 3.61 (dd, *J* = 10.6, 5.2 Hz, 1H), 3.15 (d, *J* = 13.4 Hz, 2H), 3.02 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.1 (s), 135.1 (s), 129.3 (d), 129.0 (d), 128.6 (d), 128.4 (d), 128.0 (d), 127.3 (d), 63.0 (d), 60.5 (t), 53.5 (t); MS-EI *m/z* (%): 286 ($M^{+\bullet}$–$CH_2OH^{\bullet}$, 45), 238 (8), 194 (16), 106 (16), 91 (100).



2i

(1*R*,2*R*)-2-*N*,*N*-Dibenzylamino-1-phenyl-1,3-propanediol (2i). (hexane/AcOEt : 75/25); 96% yield; white solid; $C_{23}H_{25}NO_2$; M = 347.45 $g.mol^{-1}$; M.p. 103–104 °C; [lit.[9] M.p. 102.5–104 °C]; $[\alpha]_D^{20}$ = – 89.8 (*c* 1, acetone) [lit.[9] $[\alpha]_D$ = + 92.3 (*c* 1, acetone) for (1*S*,2*S*) isomer]; IR (neat) 3362, 3028, 2923, 2855, 2360, 1494, 1452, 1229, 1198, 1117, 1063, 1052, 983, 749, 731, 698 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.38–7.15 (15H), 4.60 (d, *J* = 9.7 Hz, 1H), 4.12 (bs, 1H), 4.08 (d, *J* = 13.0 Hz, 2H), 3.79 (d, *J* = 13.0 Hz, 2H), 3.66 (m, 1H), 3.48 (m, 1H), 2.95 (m, 1H), 2.36 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.0 (s), 139.0 (s), 129.4 (d), 129.1 (d), 128.6 (d), 128.2 (d), 128.1 (d), 127.4 (d), 127.0 (d), 125.3 (d), 71.9 (d), 65.2 (d), 59.2 (t), 54.5 (t).

Synthesis of Mandelic esters

General procedure: The amino alcohol **3f** or **3g** (0.2 mmol, 1.0 equiv) was treated by DCC (50 mg, 0.24 mmol, 1.2 equiv), DMAP (5 mg, 0.04 mmol, 0.2 equiv), and (*R*)- or (*S*)-mandelic acid (33 mg, 0.2 mmol, 1.0 equiv) in CH_2Cl_2 at rt until the reaction is finished (TLC). The reaction media was filtered and water was added to the residue. Aqueous phase was extracted three times with ethyl acetate and the combined organic extracts were dried over $MgSO_4$, filtered and concentrated *in vacuo*. Purification of the crude residue by flash chromatography (silica gel, hexanes/AcOEt) afforded the corresponding mandelic esters **11a**, **11b**, or **12a**, **12b**.

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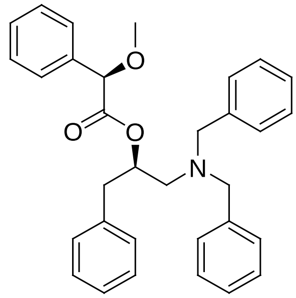
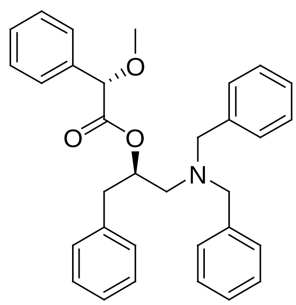
[8] Shibata, T.; Takahashi, T.; Konishi, T.; Soai, K. *Angew. Chem. Int. Ed. Engl.* **1997**, *36*, 2458–2460.
[9] Rozwadowska, M. D. *Tetrahedron* **1997**, *53*, 10615–10622.



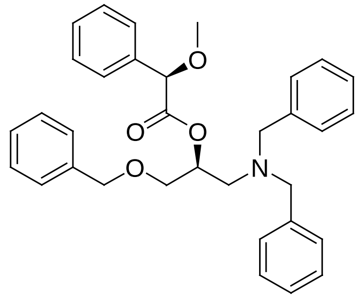
11a

(*R*)-Mandelic acid (*R*)-1-benzyl-2-*N*,*N*-dibenzylaminoethyl ester (11a). (hexane/AcOEt : 98/2); 80% yield; $C_{32}H_{33}NO_3$; 479.61 g.mol^{-1} ; $[\alpha]^{20}_{D}$ = – 24.6 (*c* 0.6, $CHCl_3$); IR (neat) 3027, 2925, 2825, 1744, 1494, 1453, 1172, 1104, 735, 696 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31–7.16 (18H), 7.05–7.03 (2H), 5.33 (m, 1H), 4.61 (s, 1H), 3.46 (d, *J* = 13.7 Hz, 2H), 3.38 (d, *J* = 13.7 Hz, 2H), 3.22 (s, 3H), 2.90 (dd, *J* = 14.1, 5.0 Hz, 1H), 2.71 (dd, *J* = 14.1, 8.2 Hz, 1H), 2.57 (dd, *J* = 13.5, 6.5 Hz, 1H), 2.48 (dd, *J* = 13.5, 5.1 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2 (s), 139.0 (s), 137.5 (s), 136.1 (s), 129.3 (d), 128.9 (d), 128.7 (d), 128.6 (d), 128.3 (d), 128.2 (d), 127.4 (d), 126.9 (d), 126.4 (d), 82.8 (d), 73.9 (d), 58.6 (t), 57.3 (q), 56.1 (t), 38.8 (t); MS-EI *m/z* (%): 479 ($M^{+\bullet}$, 0.2), 388 ($M^{+\bullet}$–$PhCH_2^{\bullet}$, 3), 313 (8), 210 (100), 121 (11), 91 (68); Anal Calcd for $C_{32}H_{33}NO_3$: C, 80.14 ; H, 6.94 ; N, 2.92. Found : C, 80.07 ; H, 7.01 ; N, 2.88.



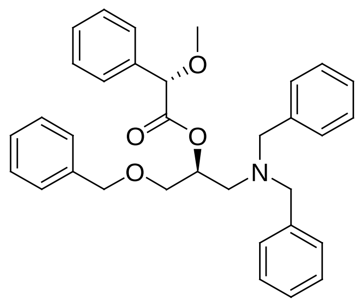
11b

(*S*)-Mandelic acid (*R*)-1-benzyl-2-*N*,*N*-dibenzylaminoethyl ester (11b). (hexane/AcOEt : 95/5); 89% yield; $C_{32}H_{33}NO_3$; M = 479.61 g.mol^{-1}; $[\alpha]^{20}_{D}$ = + 34.6 (*c* 0.5, $CHCl_3$); IR (neat) 3028, 2925, 2825, 1745, 1494, 1453, 1173, 1109, 736, 696 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28–7.00 (18H), 6.76–6.74 (2H), 5.31 (m, 1H), 4.61 (s, 1H), 3.61 (d, *J* = 13.6 Hz, 2H), 3.54 (d, *J* = 13.6 Hz, 2H), 3.35 (s, 3H), 2.78 (dd, *J* = 14.2, 4.8 Hz, 1H), 2.62 (dd, *J* = 13.4, 6.7 Hz, 1H), 2.57–2.49 (2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2 (s), 139.1 (s), 137.1 (s), 136.2 (s), 129.2 (d), 129.1 (d), 128.6 (d), 128.3 (d), 128.2 (d), 127.3 (d), 127.1 (d), 126.2 (d), 82.7 (d), 73.7 (d), 59.0 (t), 57.4 (q), 56.4 (t), 38.5 (t) ; MS-EI *m/z* (%): 479 ($M^{+\bullet}$, 0.2), 388 (2), 313 (7), 210 (100), 121 (12), 91 (68); Anal Calcd for $C_{32}H_{33}NO_3$: C, 80.14 ; H, 6.94 ; N, 2.92. Found : C, 79.91 ; H, 6.95 ; N, 2.79.



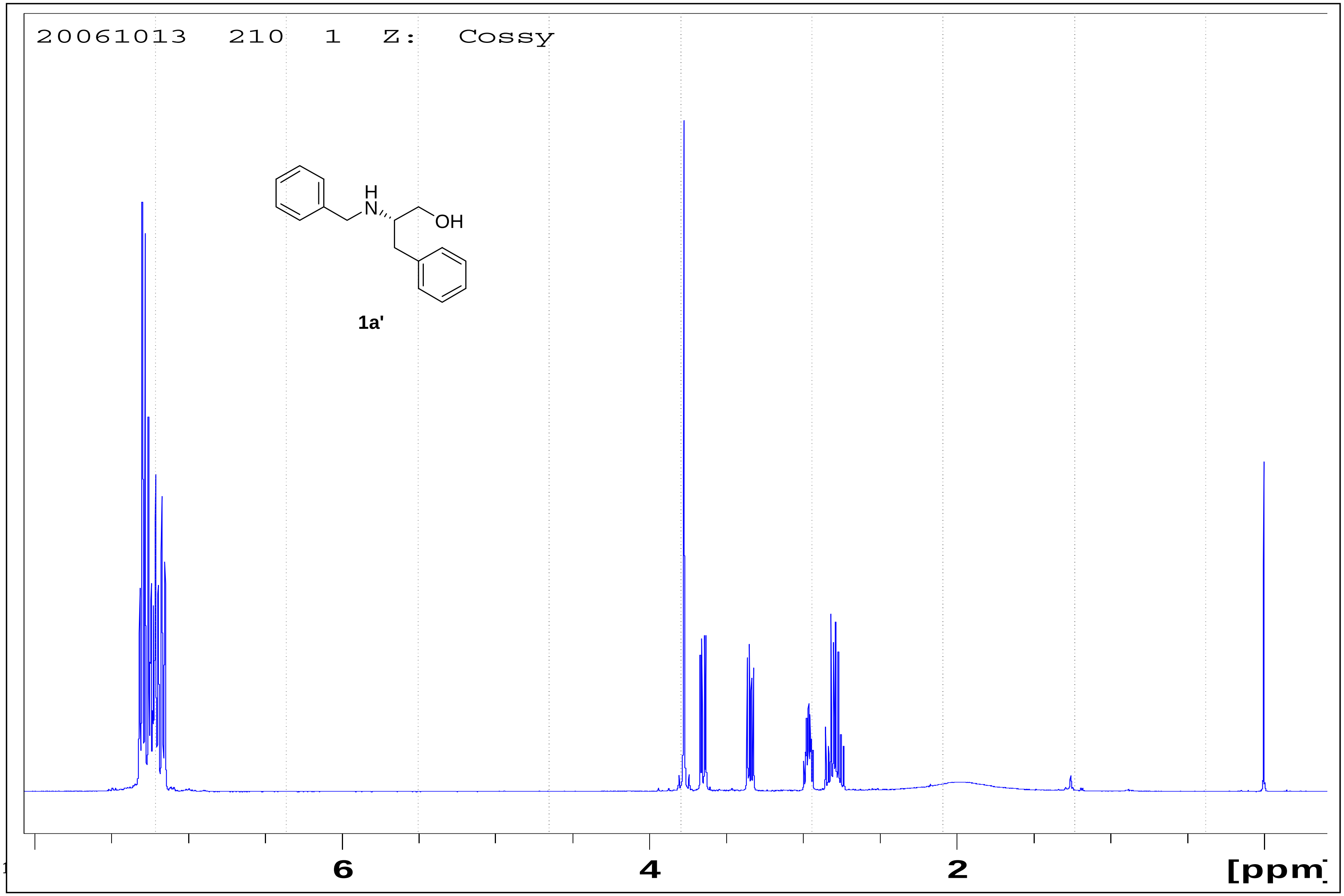
12a

(*R*)-Mandelic acid (*S*)-1-benzyloxymethyl-2-*N*,*N*-dibenzylaminoethyl ester (12a). (hexane/AcOEt : 95/5); 90% yield; $C_{33}H_{35}NO_4$; M = 509.63 g.mol^{-1}; $[\alpha]^{20}_{D}$ = – 22.2 (*c* 1, $CHCl_3$); IR (neat) 3028, 2926, 1746, 1494, 1453, 1174, 1103, 734, 696 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46–7.43 (2H), 7.35–7.13 (18H), 5.28 (m, 1H), 4.75 (s, 1H), 4.42 (d, *J* = 12.0 Hz, 1H), 4.38 (d, *J* = 12.0 Hz, 1H), 3.57–3.35 (9H), 2.54 (d, *J* = 6.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3 (s), 139.0 (s), 138.0 (s), 136.1 (s), 128.9 (d), 128.8 (d), 128.6 (d), 128.3 (d), 128.1 (d), 127.7 (d), 127.6 (d), 127.5 (d), 126.9 (d), 82.7 (d), 73.0 (t), 72.0 (d), 70.0 (t), 58.6 (t), 57.3 (q), 53.5 (t); MS-EI *m/z* (%): 418 ($M^{+\bullet}$–$PhCH_2^{\bullet}$, 1), 388 (3), 344 (3), 312 (5), 210 (82), 121 (25), 91 (100), 77 (19); HRMS (IC) Calcd for $C_{33}H_{36}NO_4$ [$M+H^+$] : 510.2644, Found : 510.2643.



12b

(*S*)-Mandelic acid (*S*)-1-benzyloxymethyl-2-*N*,*N*-dibenzylaminoethyl ester (12b). (hexane/AcOEt : 95/5); 87% yield; $C_{33}H_{35}NO_4$; M = 509.63 g.mol^{-1}; $[\alpha]^{20}_{D}$ = – 31.6 (*c* 1, $CHCl_3$); IR (neat) 3028, 2925, 2827, 1747, 1494, 1453, 1174, 1105, 734, 696 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43–7.39 (2H), 7.32–7.18 (16H), 7.08–7.04 (2H), 5.29 (m, 1H), 4.73 (s, 1H), 4.17 (s, 2H), 3.60 (d, *J* = 13.4 Hz, 2H), 3.54 (d, *J* = 13.4 Hz, 2H), 3.42 (s, 3H), 3.40 (m, 1H), 3.34 (m, 1H), 2.66 (d, *J* = 6.7 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4 (s), 139.1 (s), 138.1 (s), 136.3 (s), 129.0 (d), 128.6 (d), 128.5 (d), 128.3 (d), 128.2 (d), 127.5 (d), 127.3 (d), 127.1 (d), 82.7 (d), 73.0 (t), 72.0 (d), 69.9 (t), 58.9 (t), 57.5 (q), 53.7 (t); MS-EI *m/z* (%) : 418 ($M^{+\bullet}$–$PhCH_2^{\bullet}$, 1), 388 (2), 344 (3), 312 (5), 210 (93), 121 (19), 91 (100), 77 (18); HRMS (ESI) Calcd for $C_{33}H_{36}NO_4$ ($M+H^+$): 510.2639, Found : 510.2637.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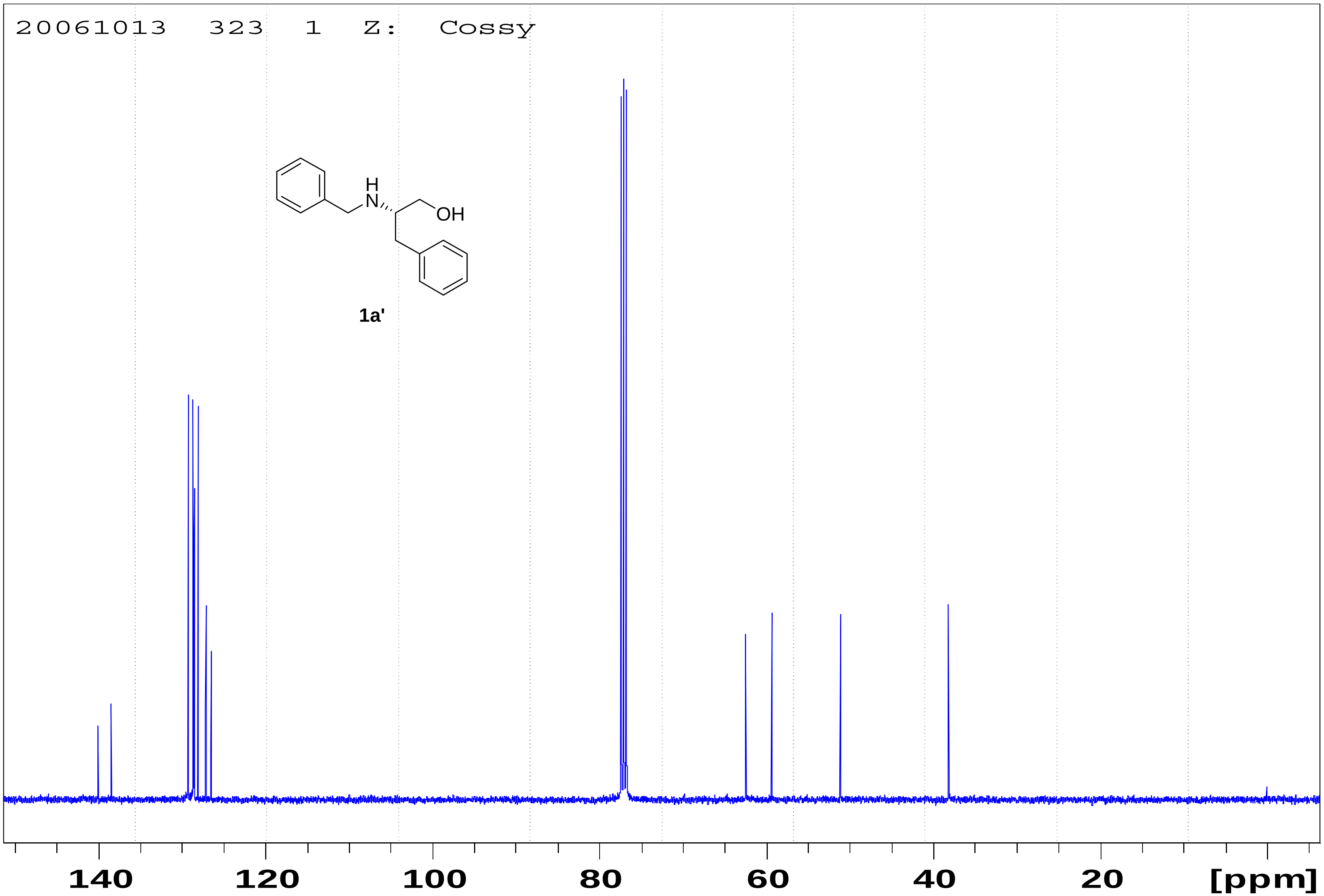
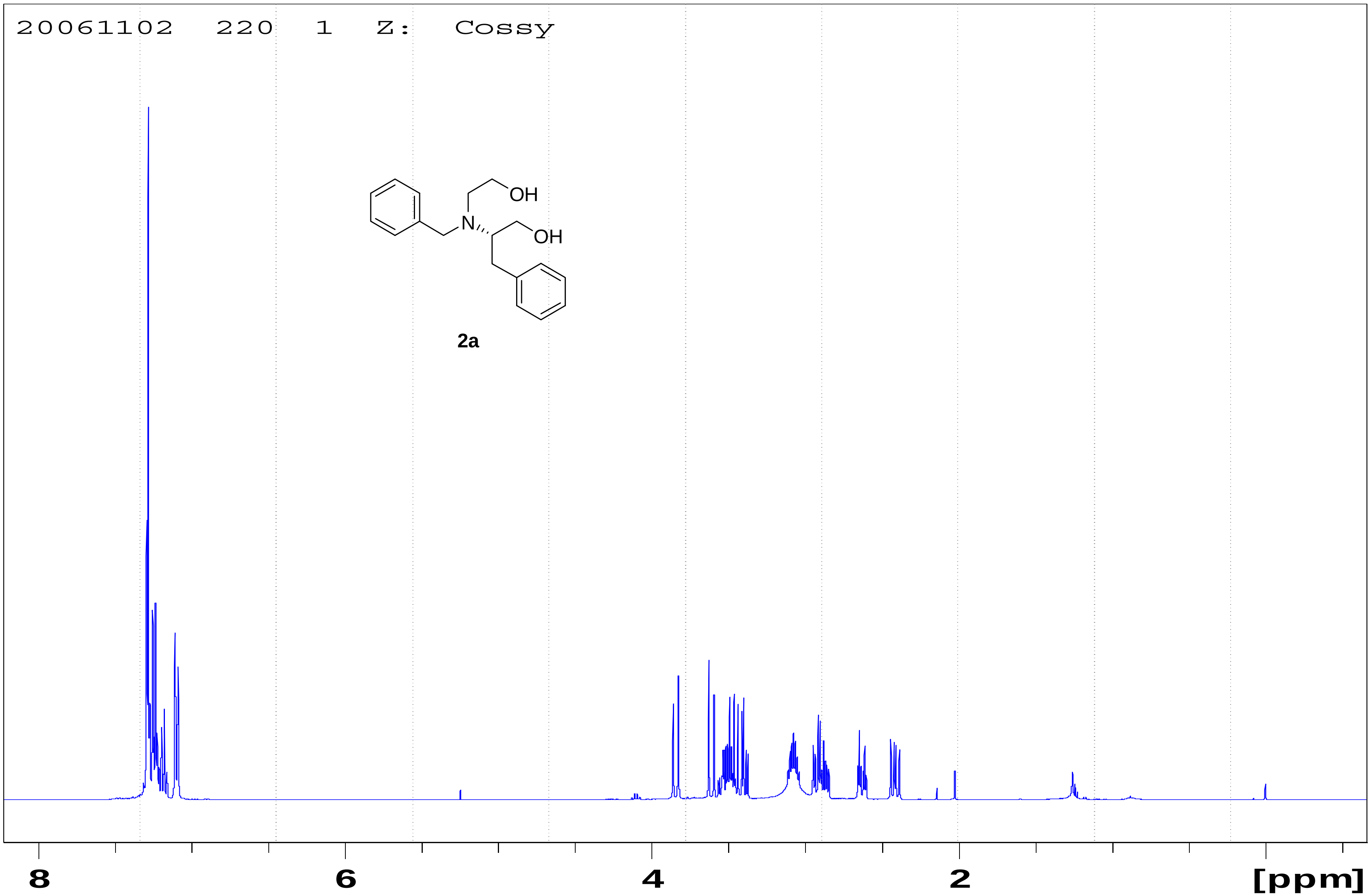
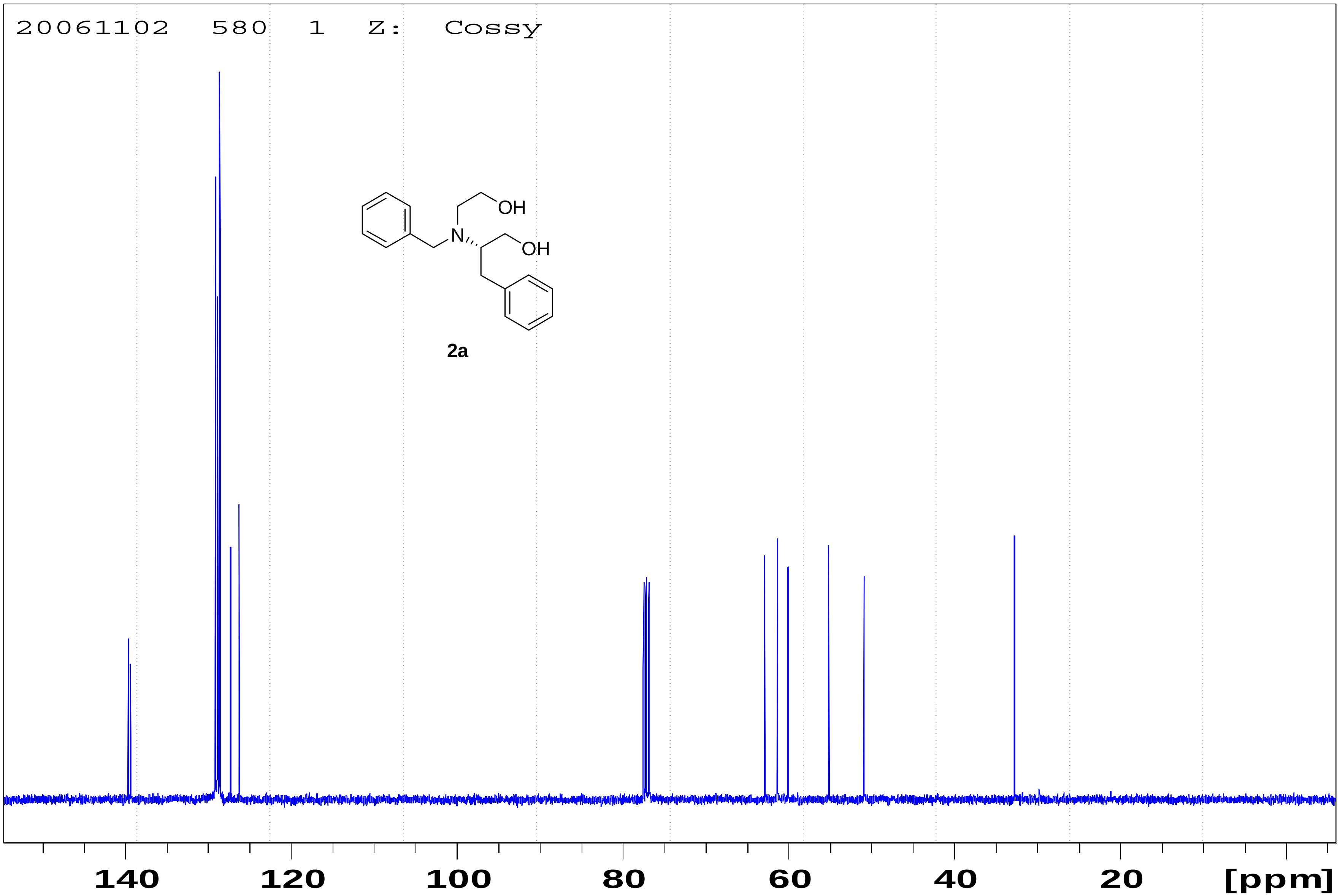
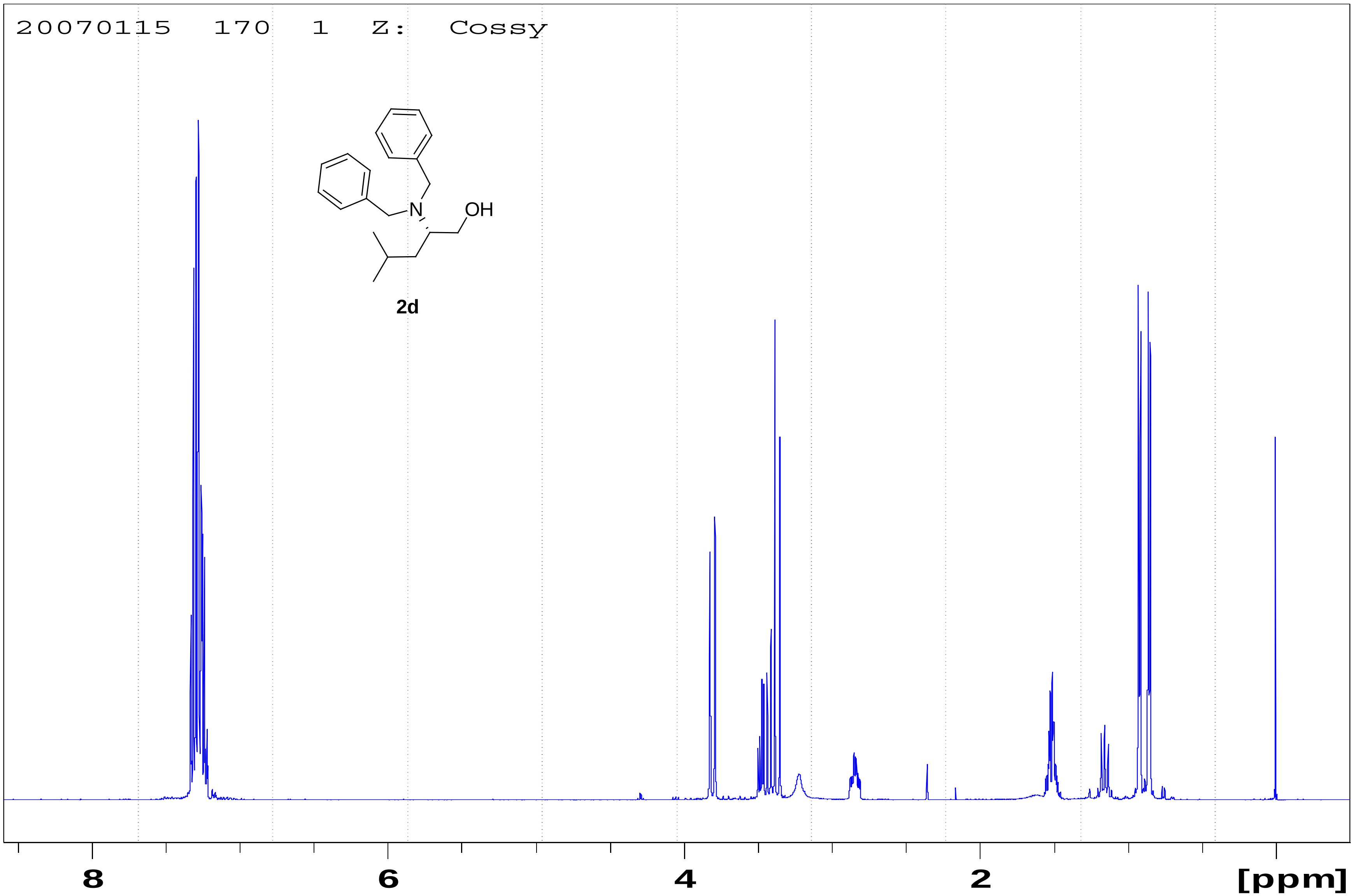
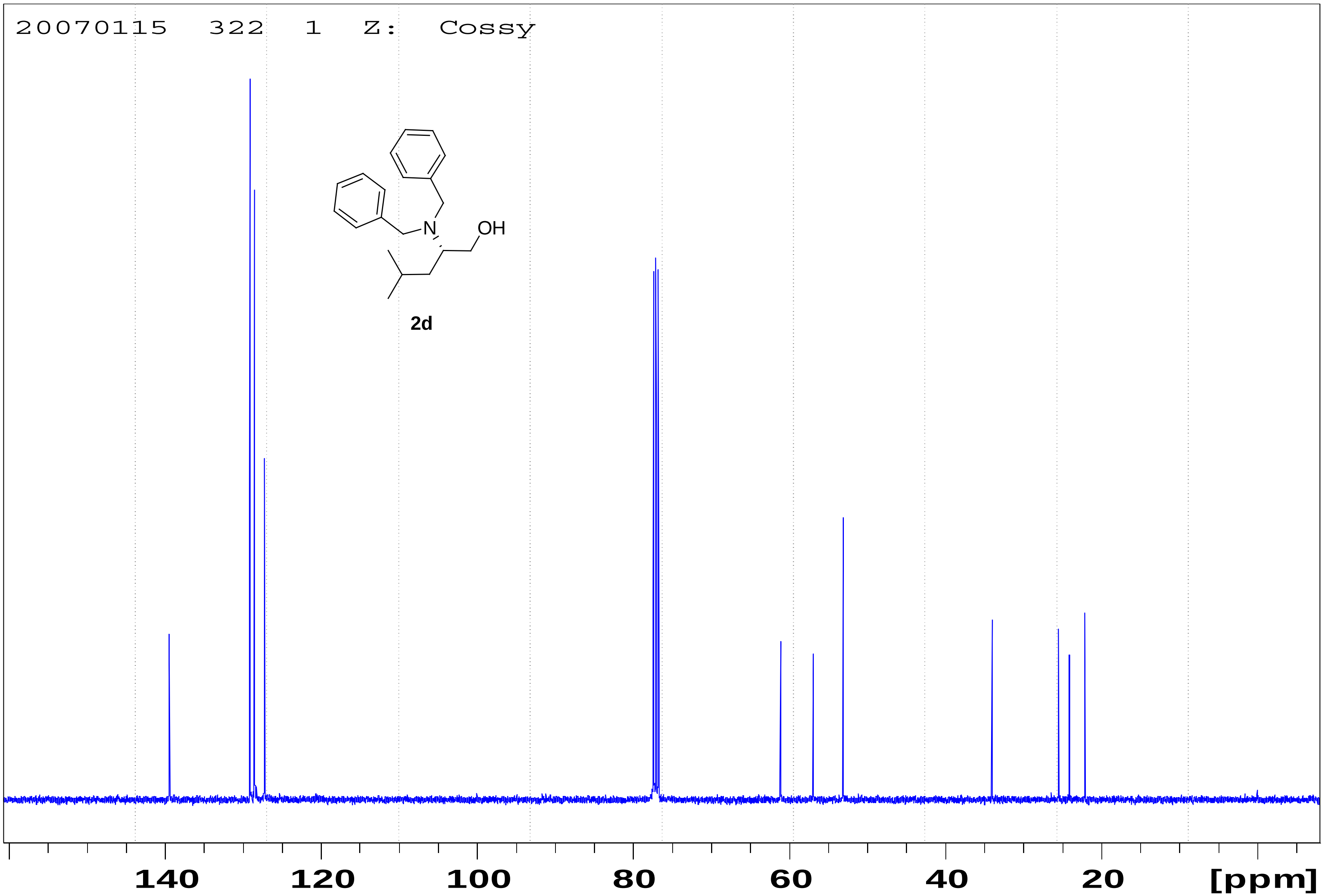
2
[ppm]

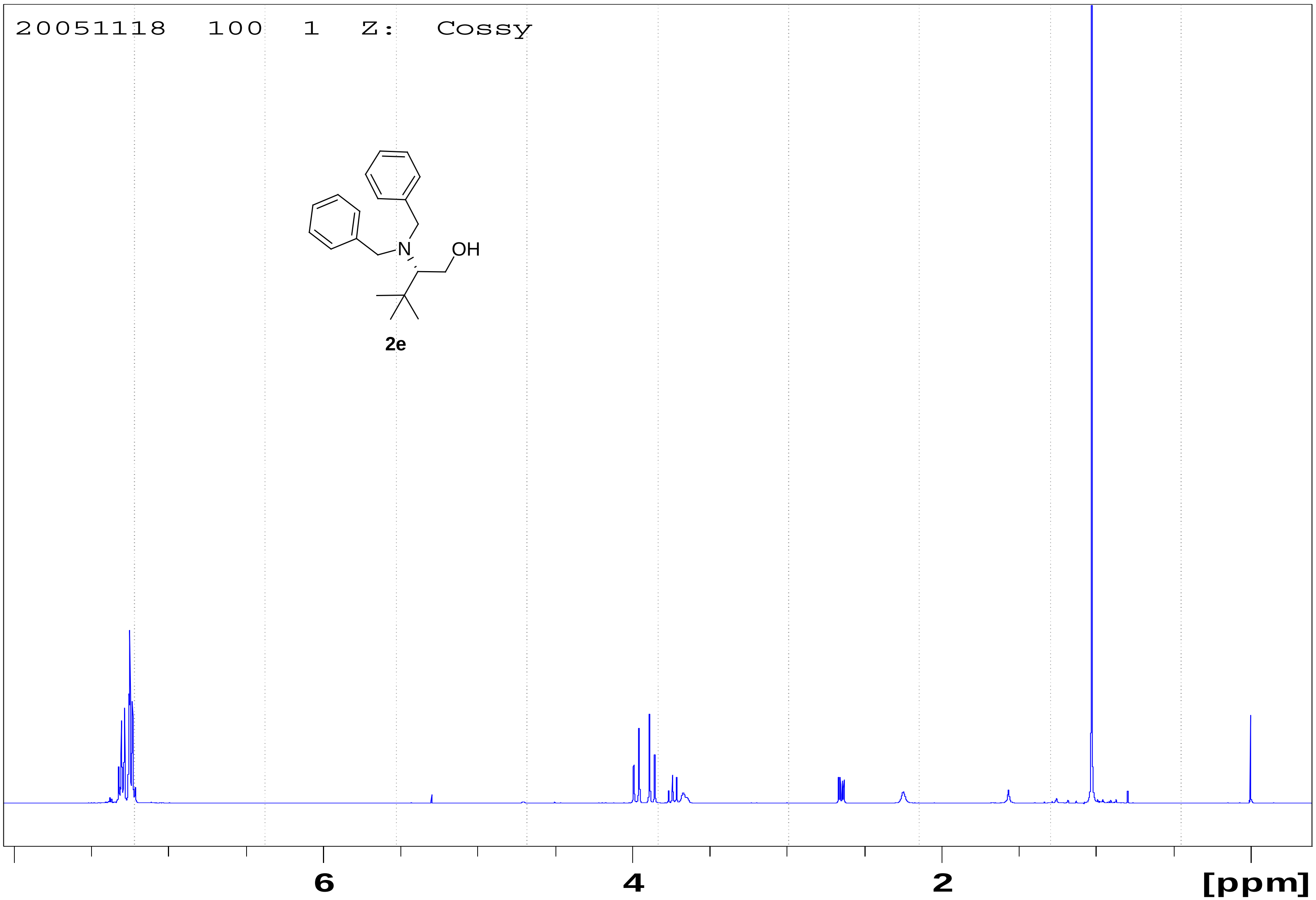

20061013 323 1 Z: Cossy
H
N
OH
1a'
140
120
100
80
60
40
20
[ppm]

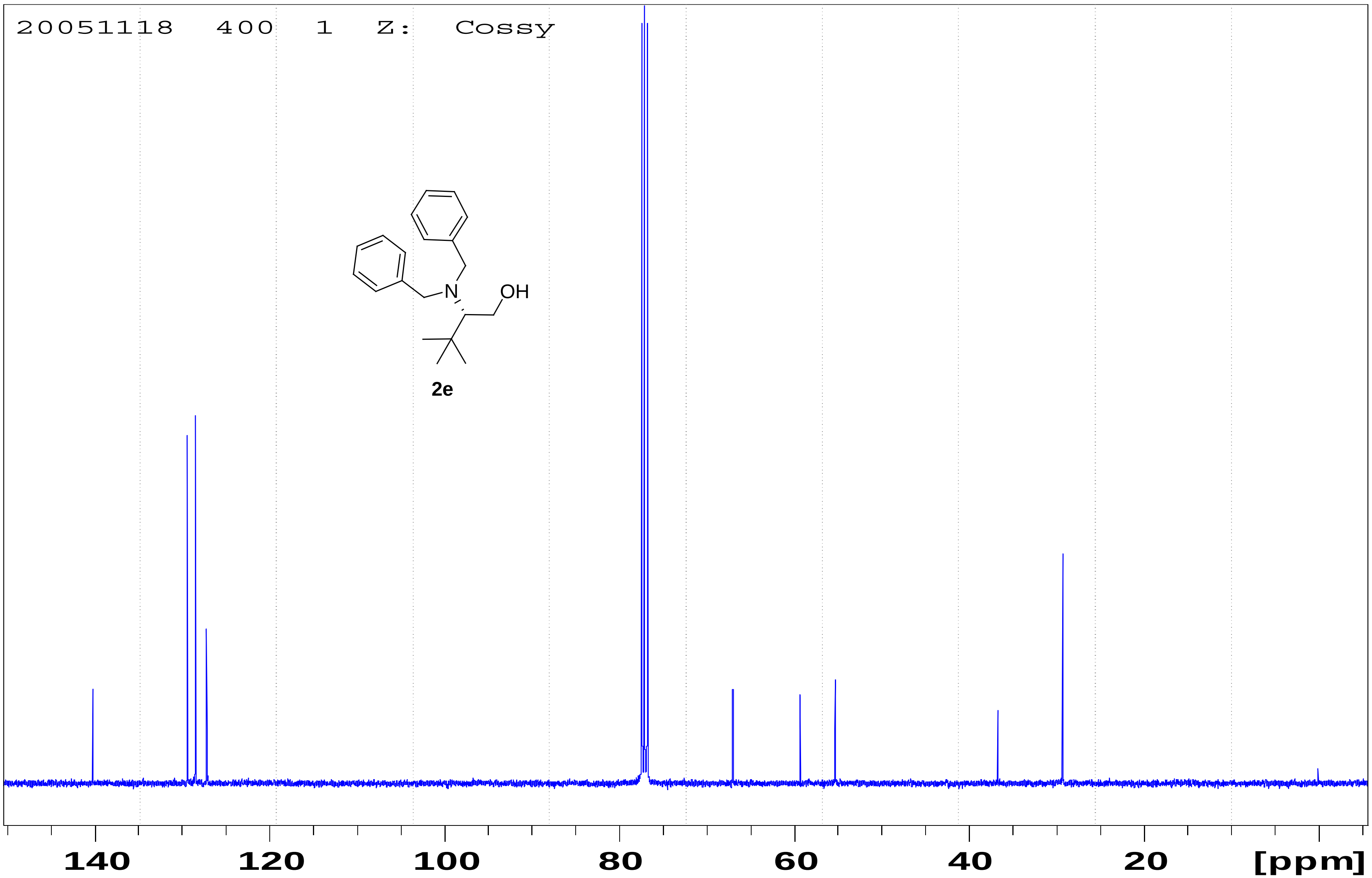

20061102 220 1 Z: Cossy
OH
N
OH
2a
8
6
4
2
[ppm]


20061102 580 1 Z: Cossy
OH
N
OH
2a
140
120
100
80
60
40
20
[ppm]

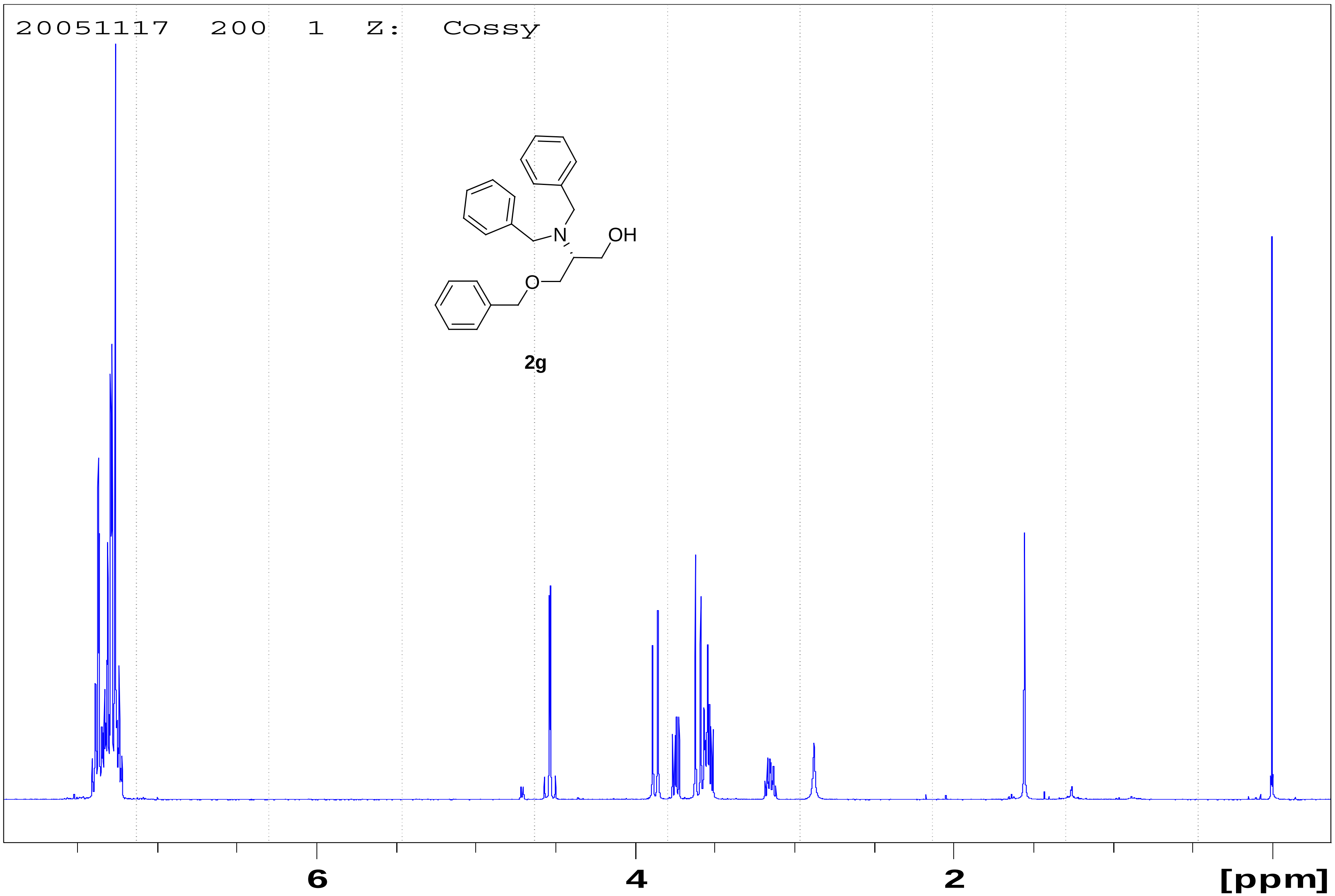

20070115 170 1 Z: Cossy
N
OH
2d
8
6
4
2
[ppm]


20070115 322 1 Z: Cossy
N
OH
2d
140
120
100
80
60
40
20
[ppm]

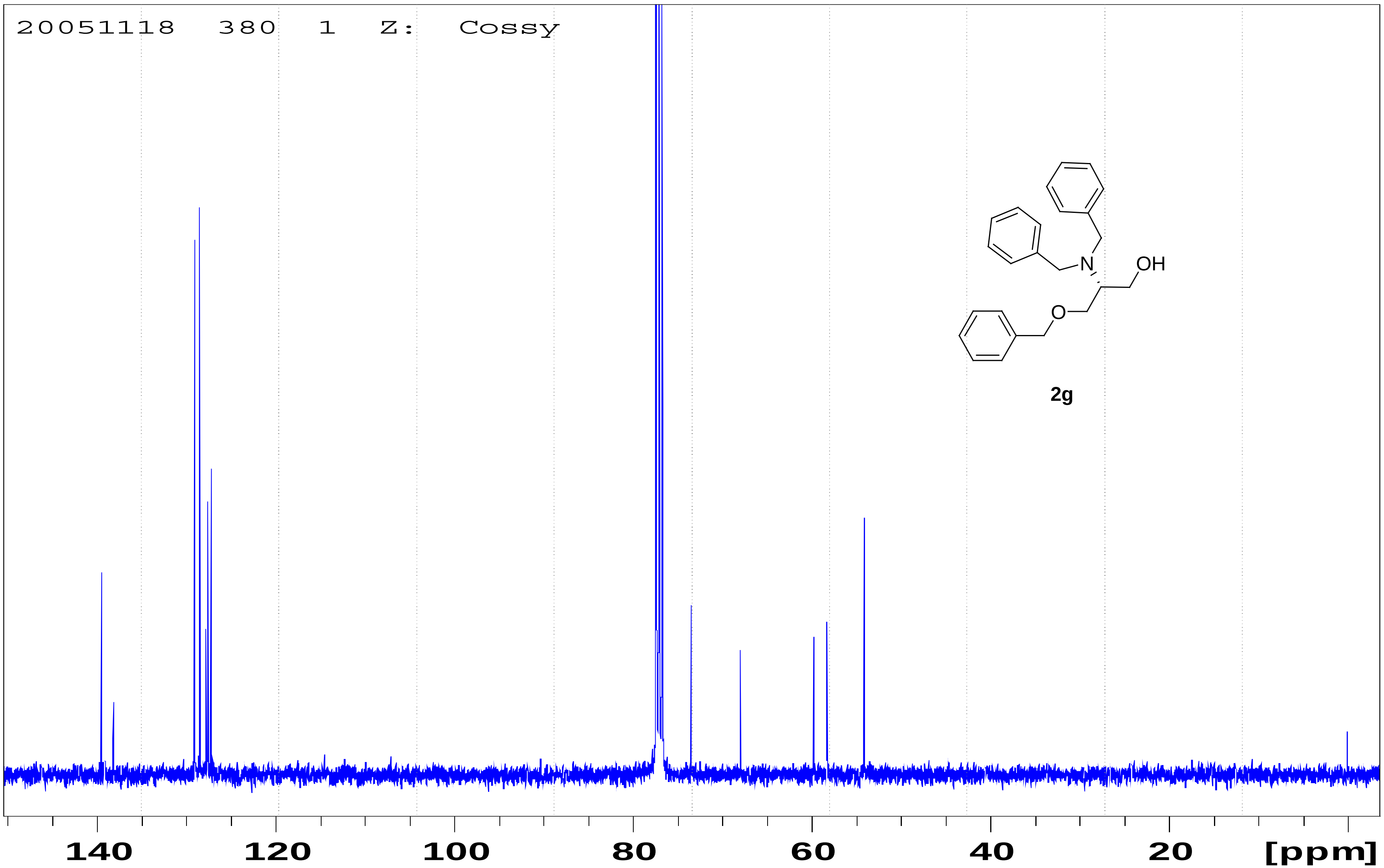

20051118 100 1 Z: Cossy
OH
N
2e
6
4
2
[ppm]



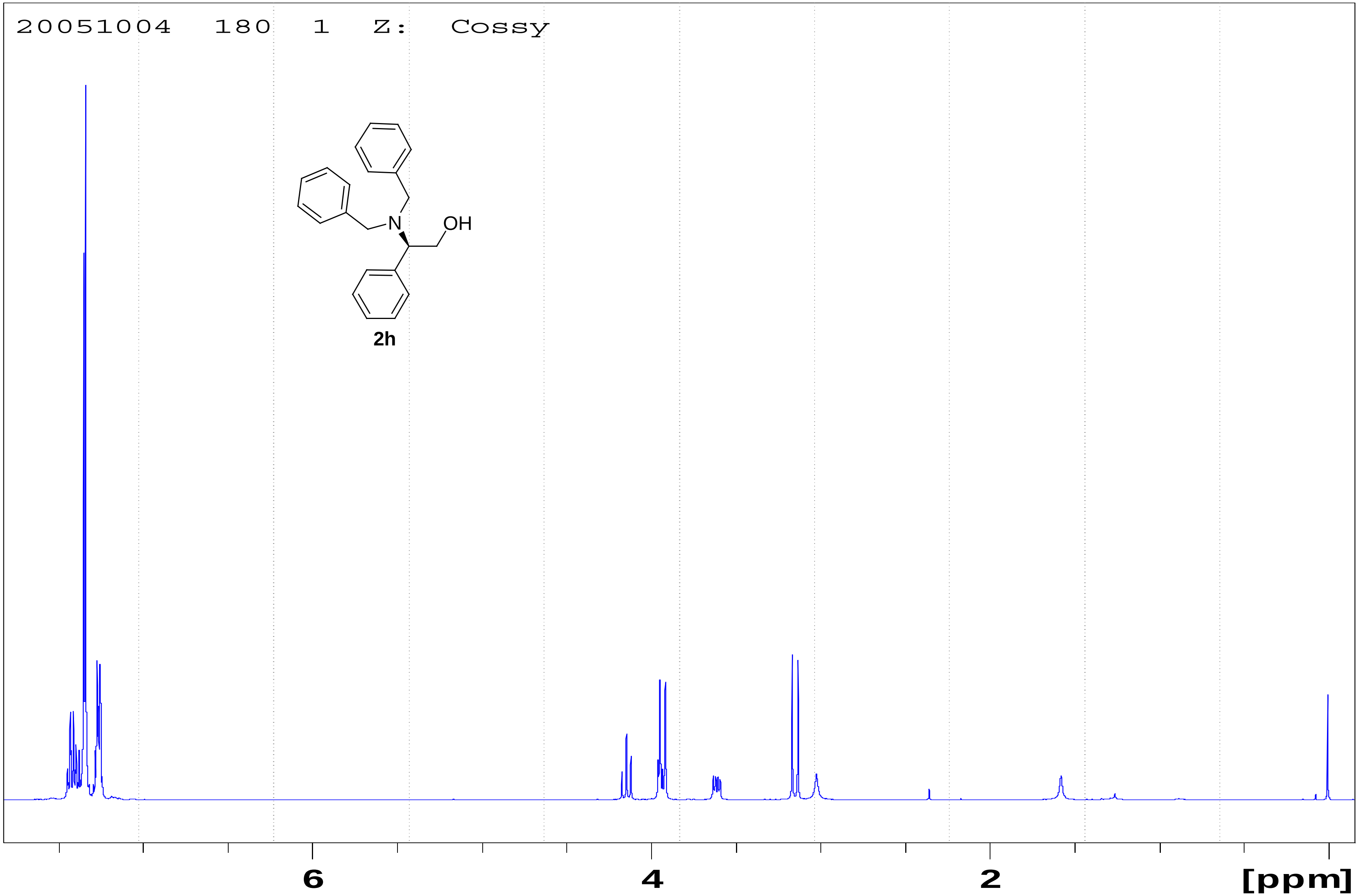
20051118 400 1 Z: Cossy
OH
N
2e
140
120
100
80
60
40
20
[ppm]

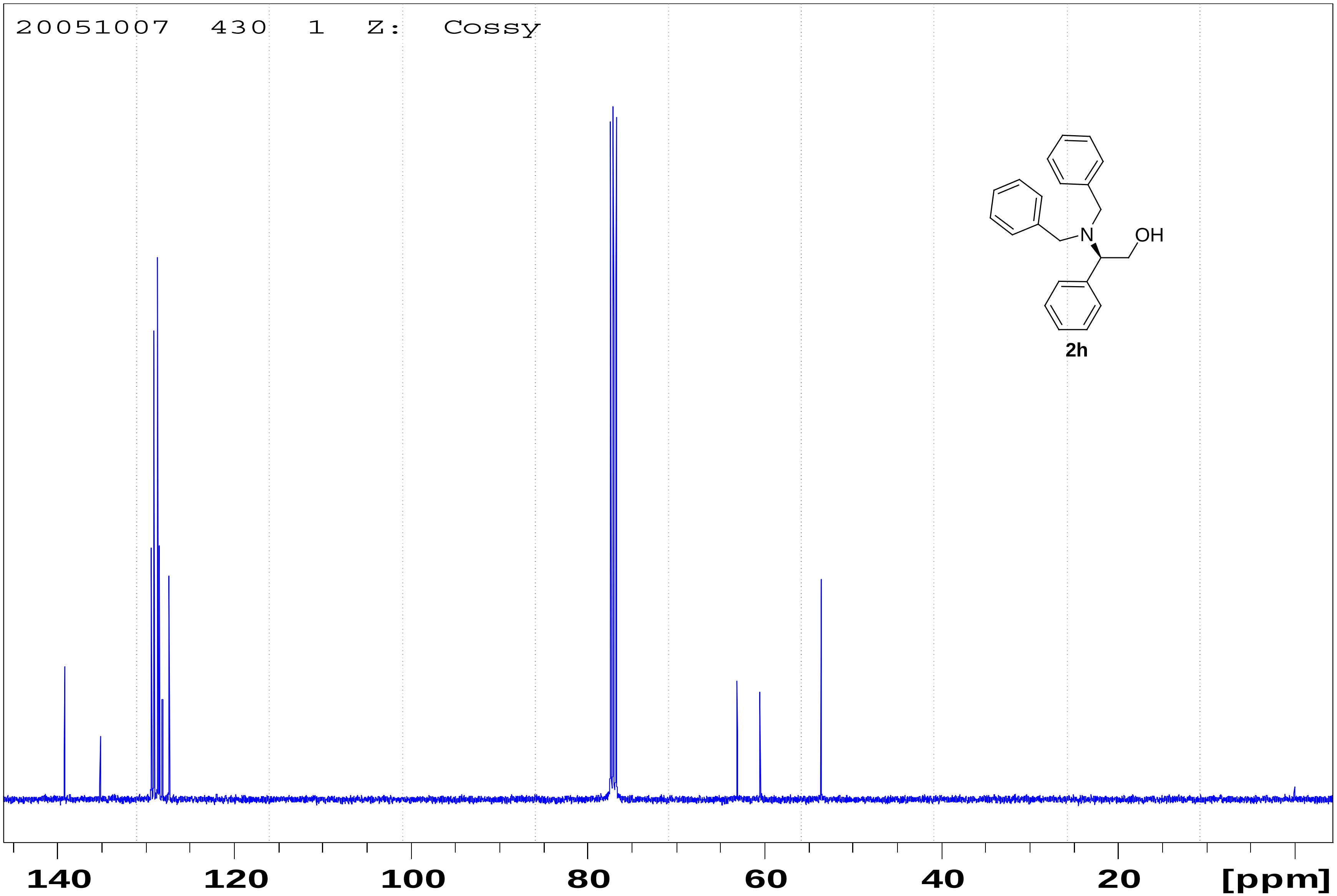


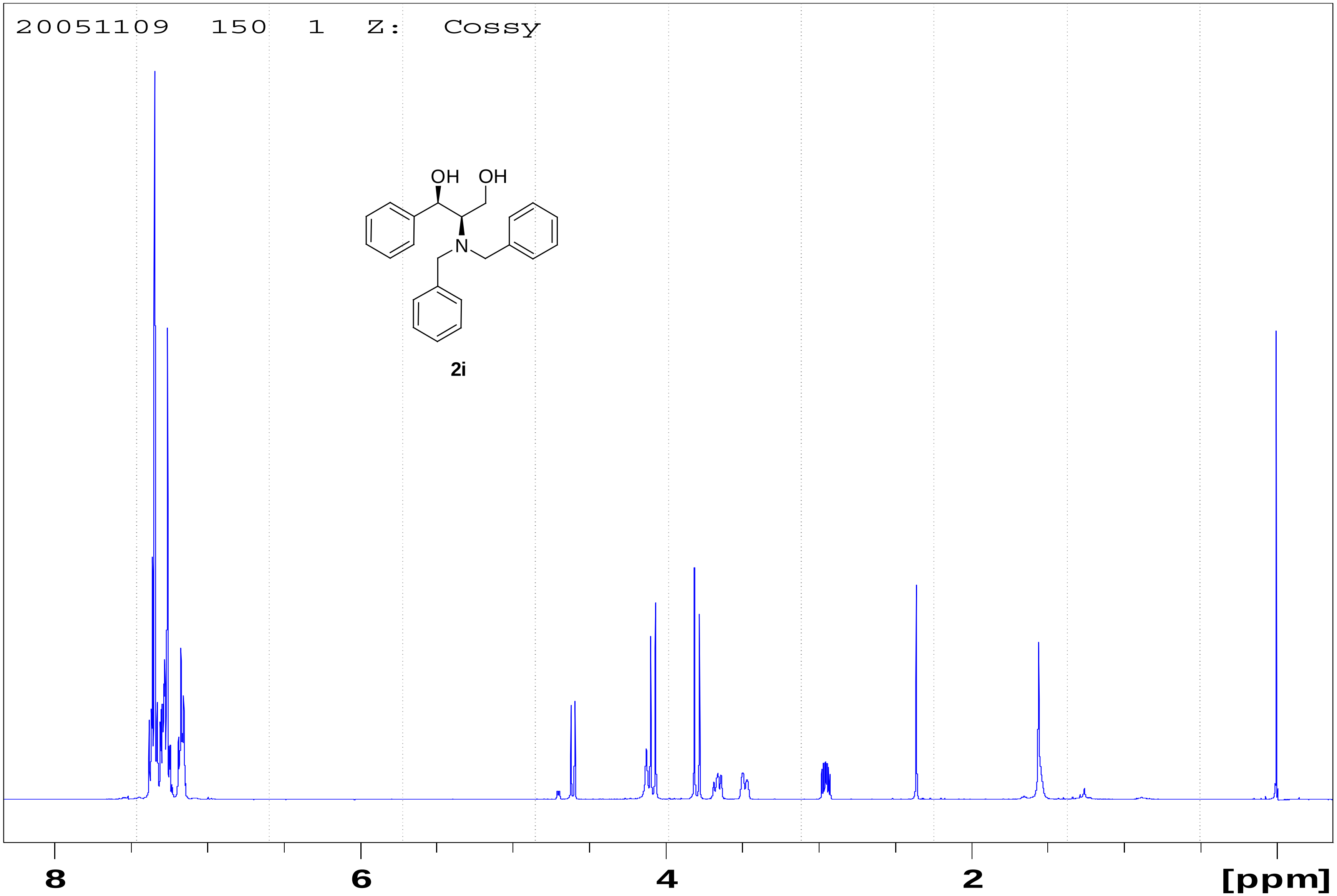
20051117 200 1 Z: Cossy
OH
N
O
2g
6
4
2
[ppm]

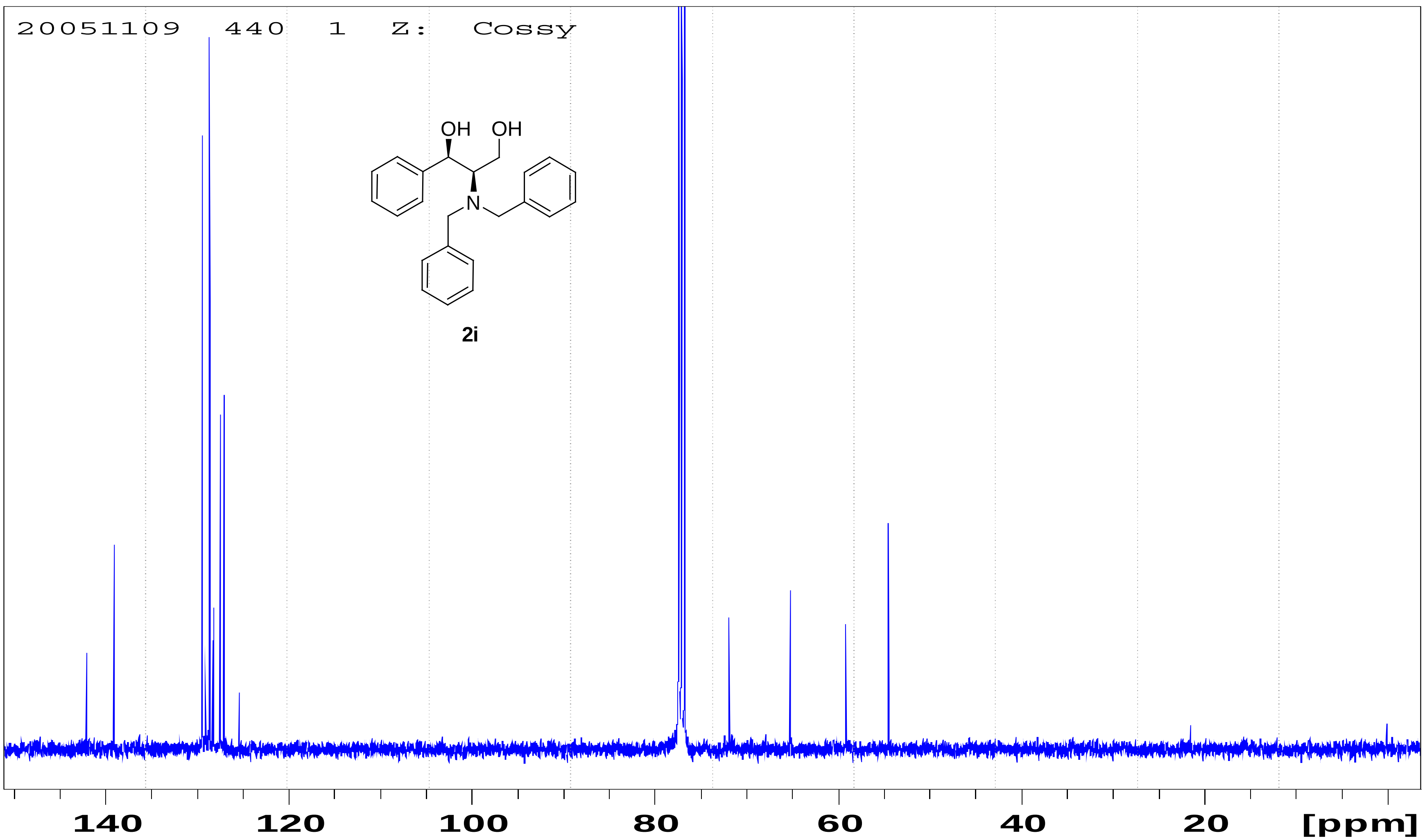


20051118 380 1 Z: Cossy
OH
N
O
2g
140
120
100
80
60
40
20
[ppm]

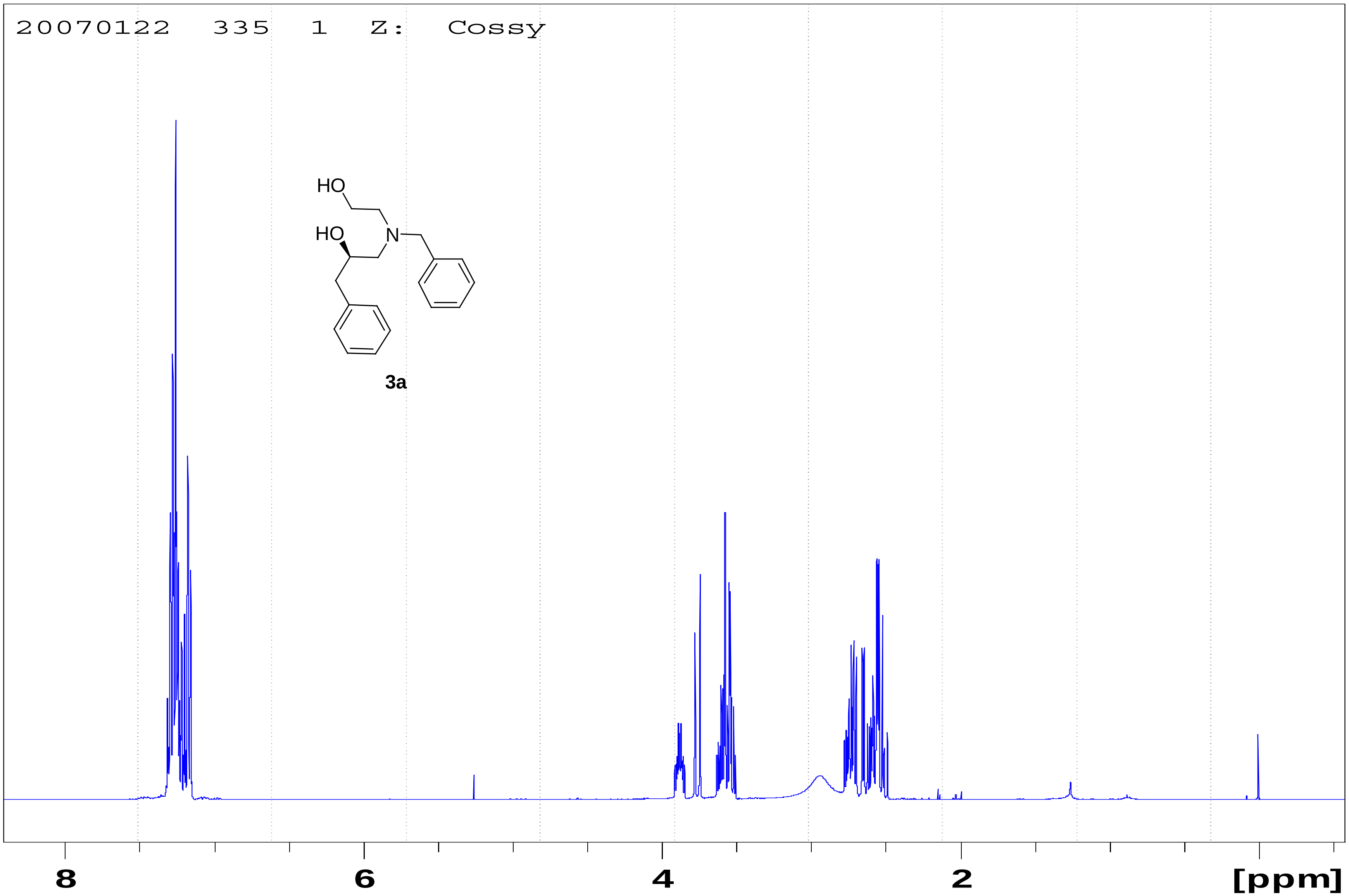

20051004 180 1 Z: Cossy
N
OH
2h
6
4
2
[ppm]

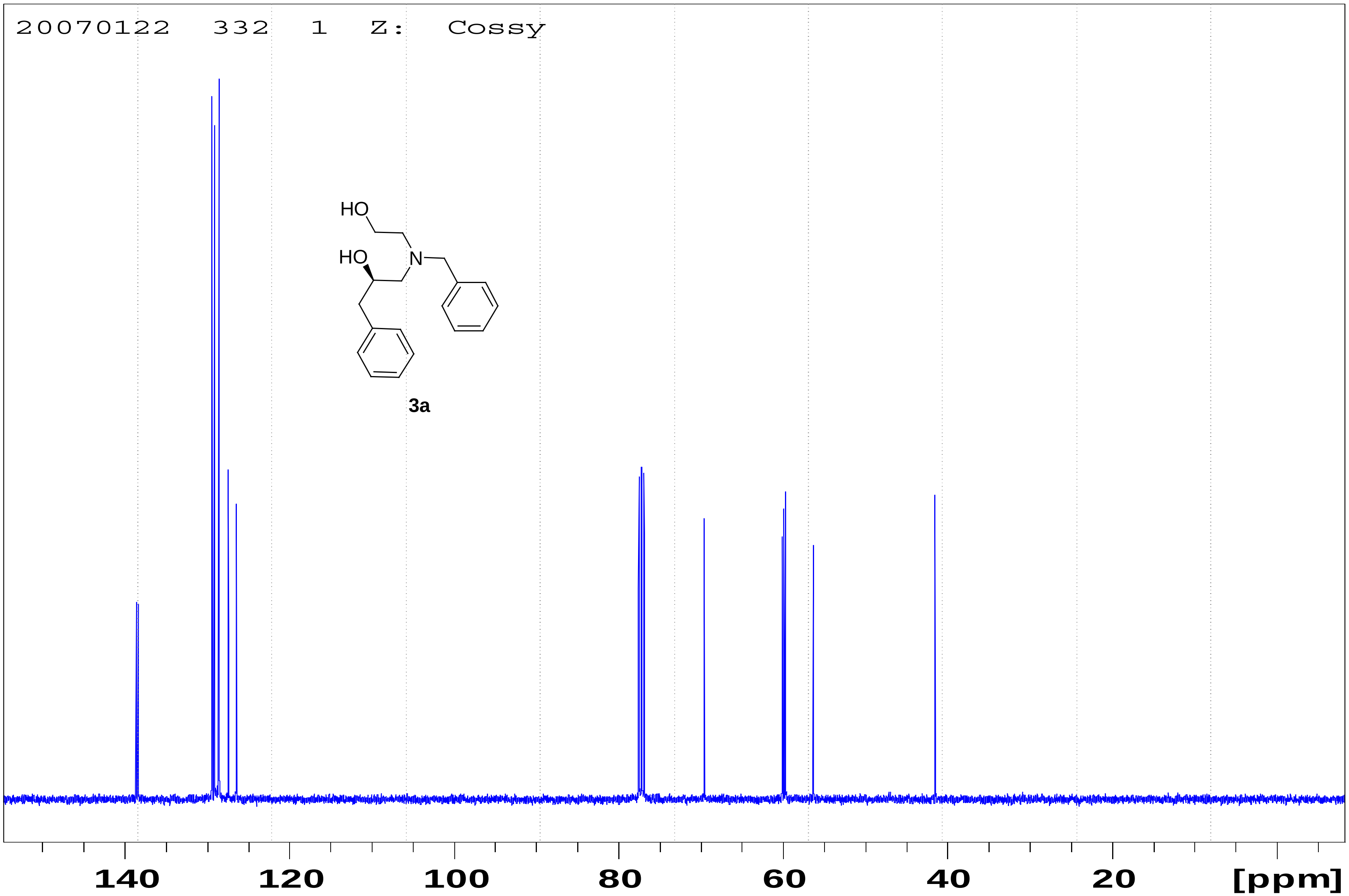

20051007 430 1 Z: Cossy
N
OH
2h
140
120
100
80
60
40
20
[ppm]


20051109 150 1 Z: Cossy
OH
OH
N
2i
8
6
4
2
[ppm]

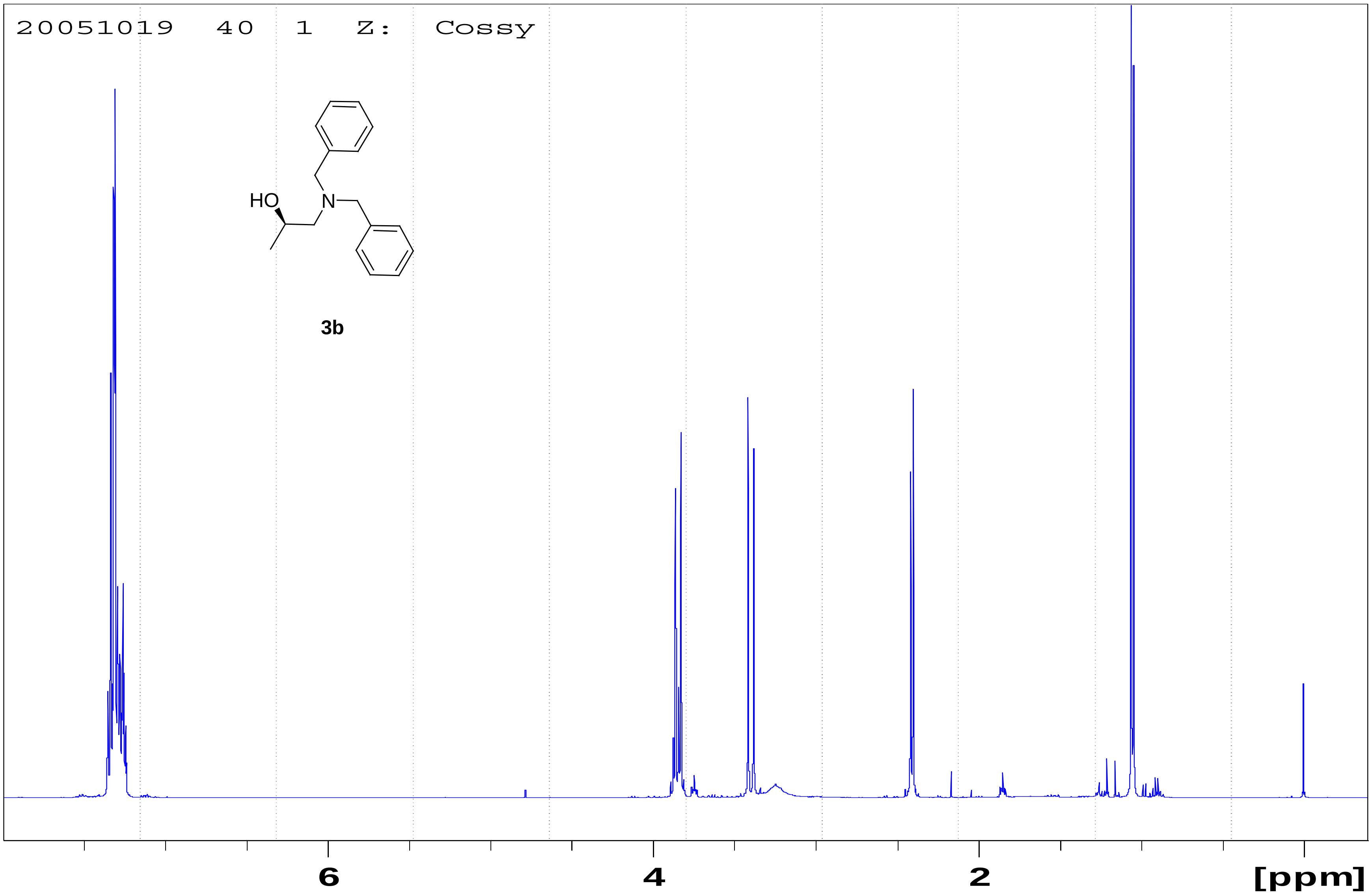


20051109 440 1 Z: Cossy
OH
OH
N
2i
140
120
100
80
60
40
20
[ppm]

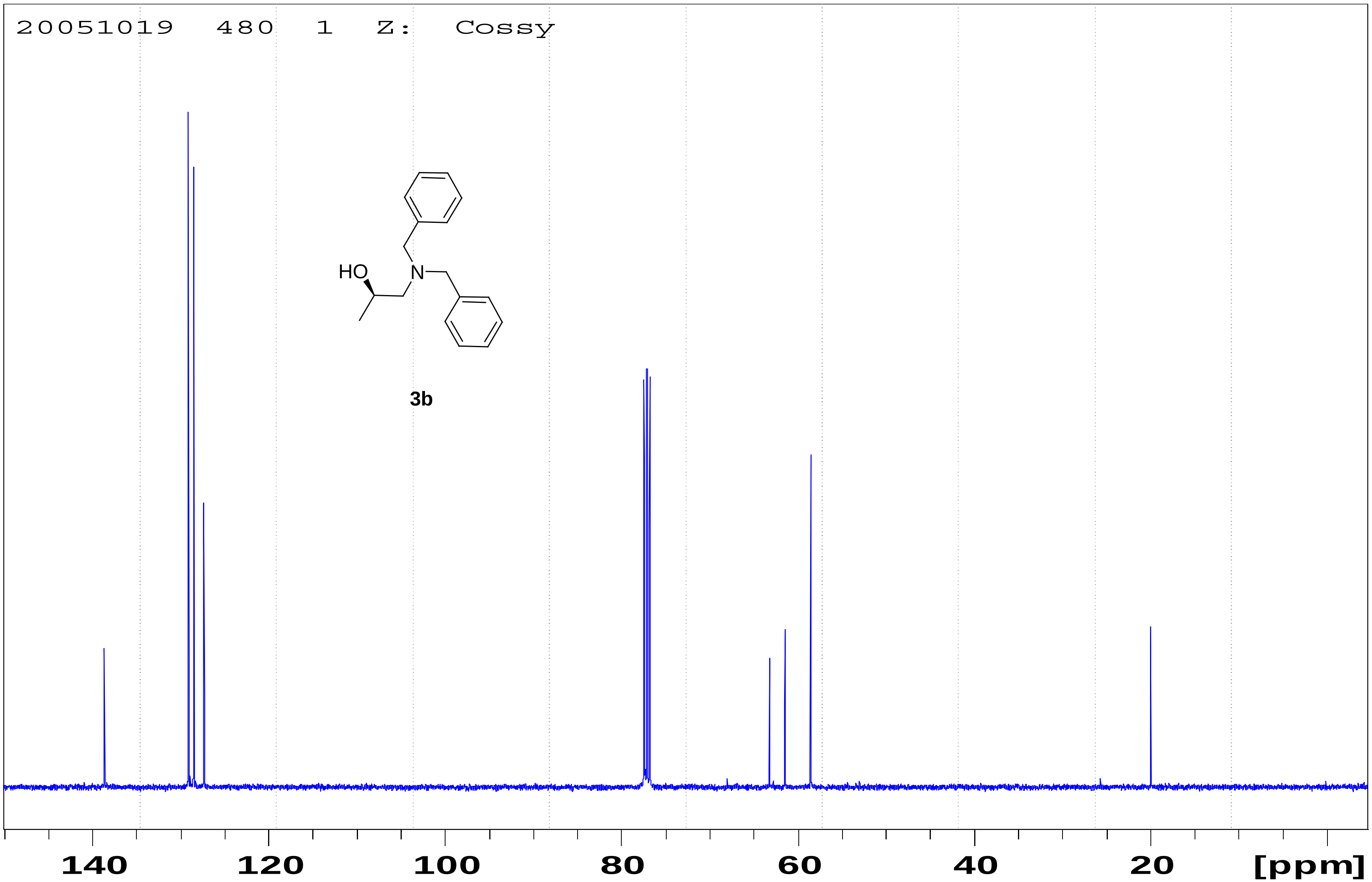

20070122 335 1 Z: Cossy
HO
HO
N
3a
8
6
4
2
[ppm]

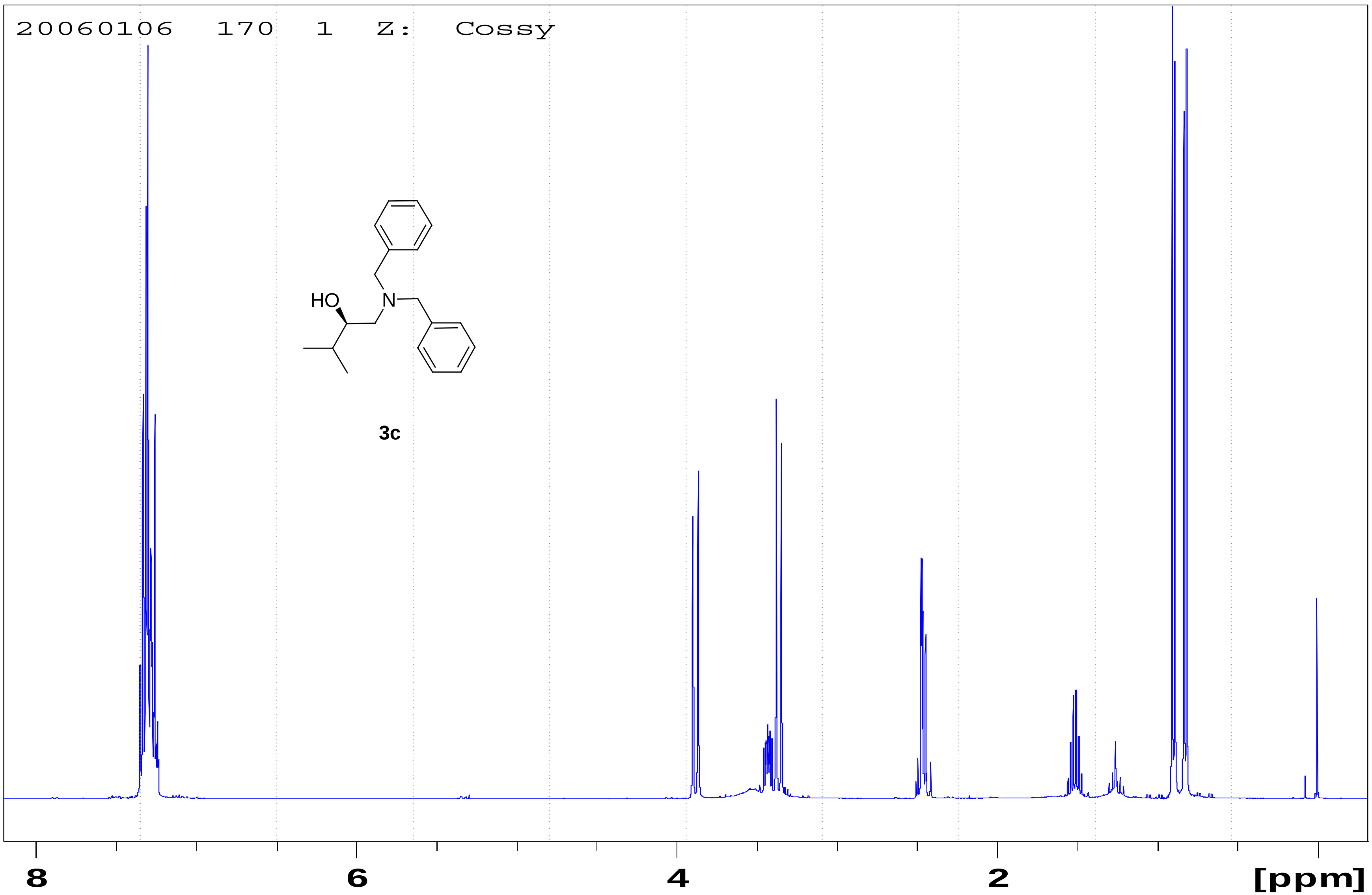


20070122 332 1 Z: Cossy
HO
HO
N
3a
140
120
100
80
60
40
20
[ppm]

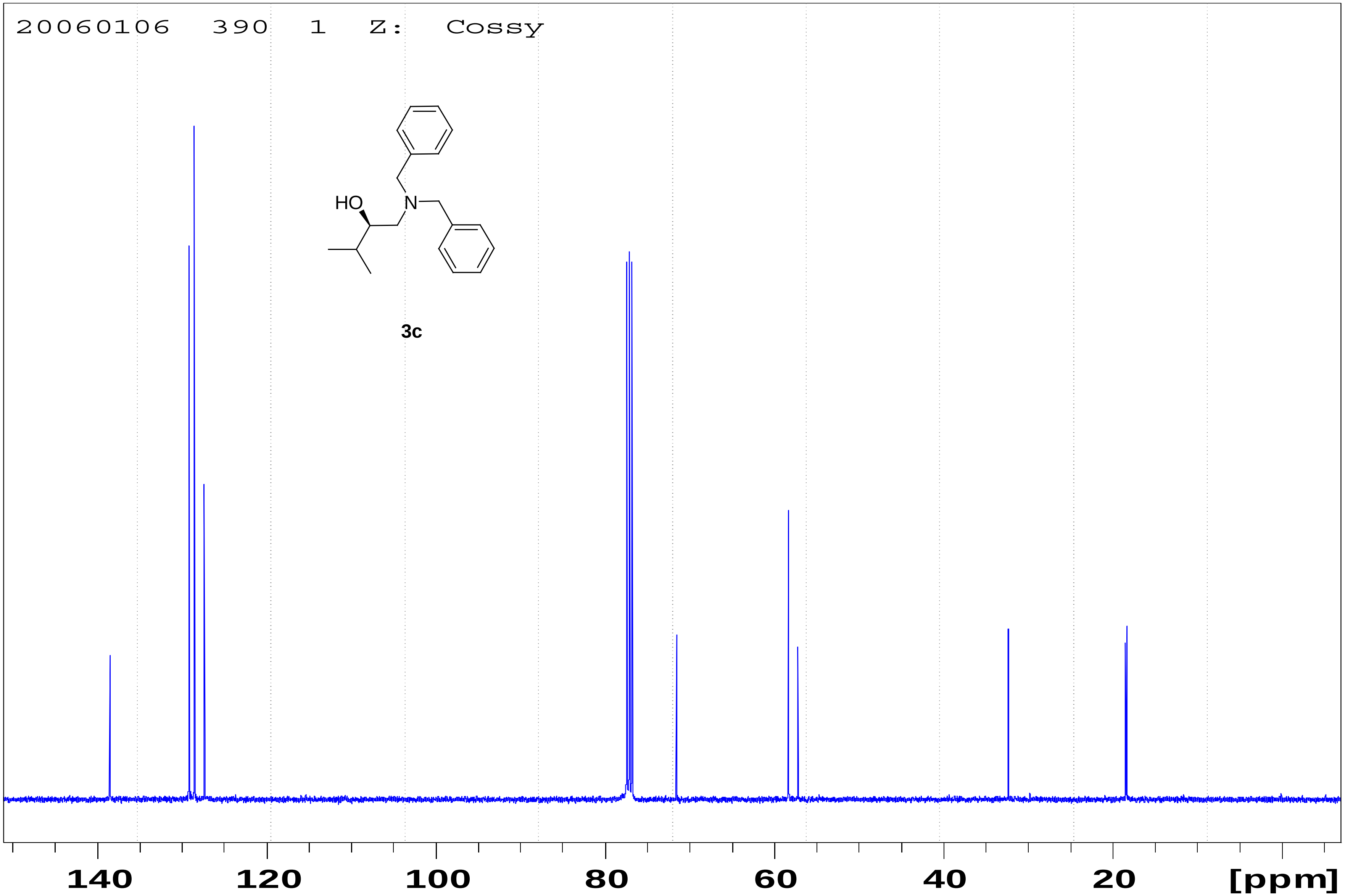


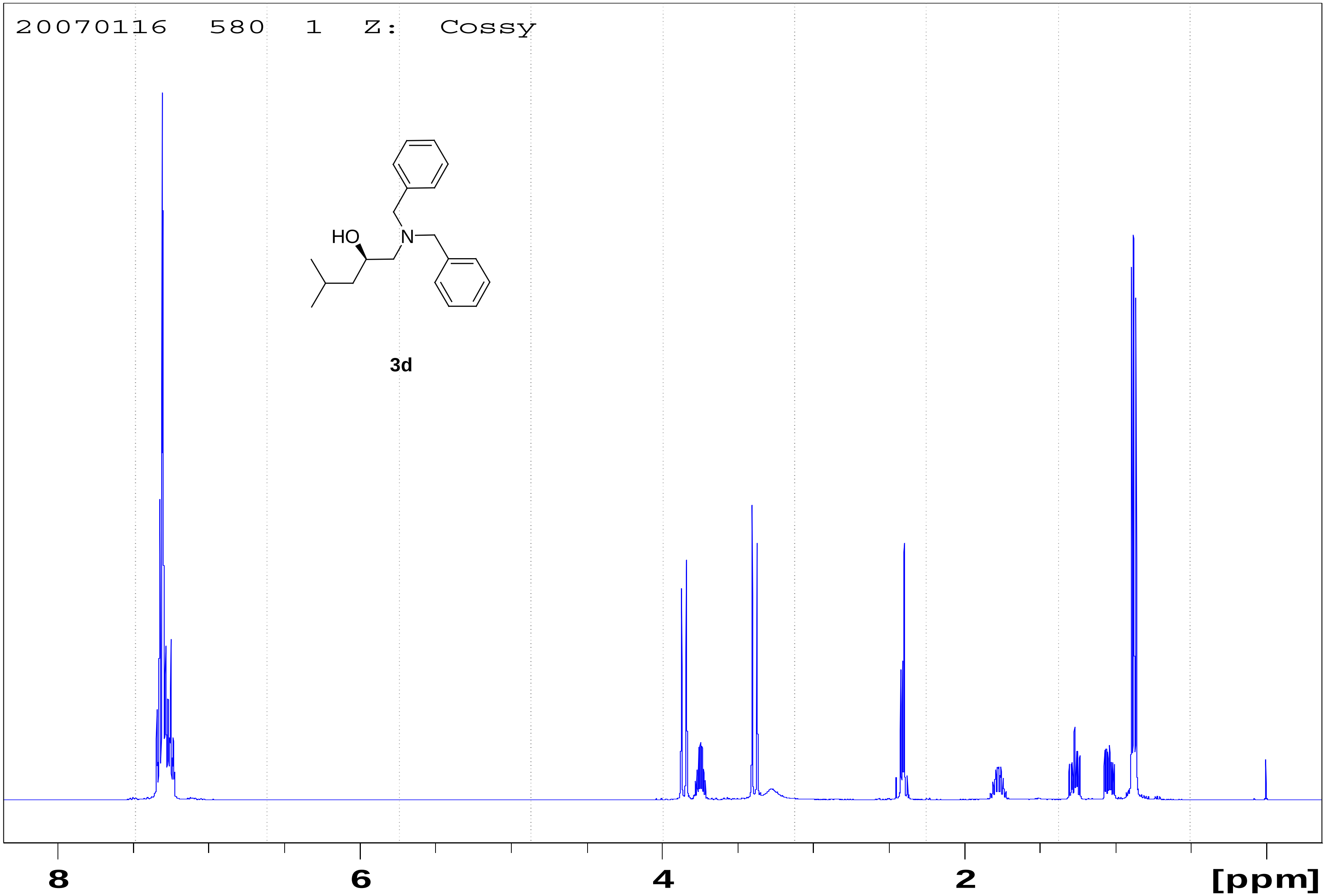
3b

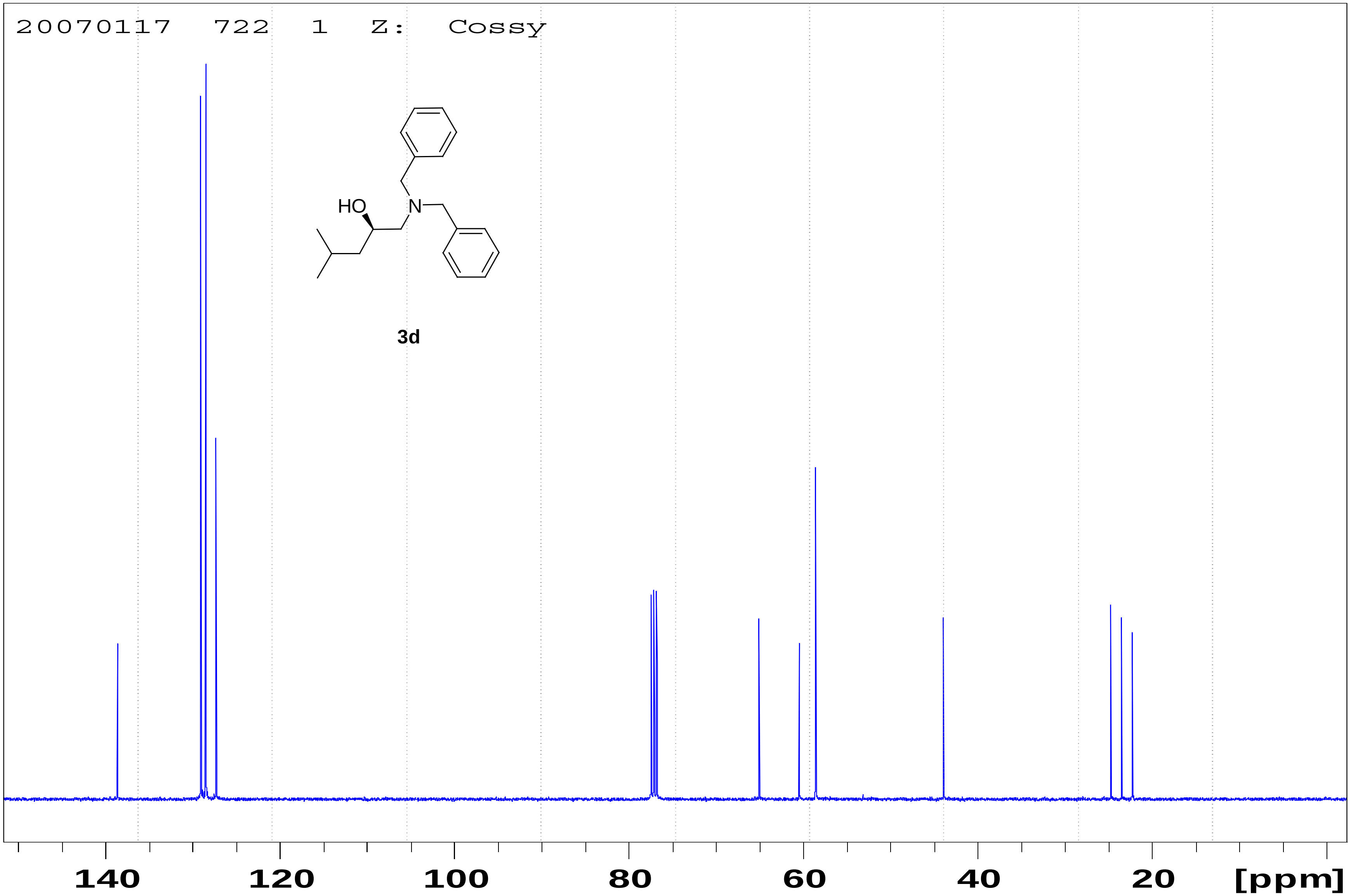

20051019 480 1 Z: Cossy
HO
N
3b
140
120
100
80
60
40
20
[ppm]

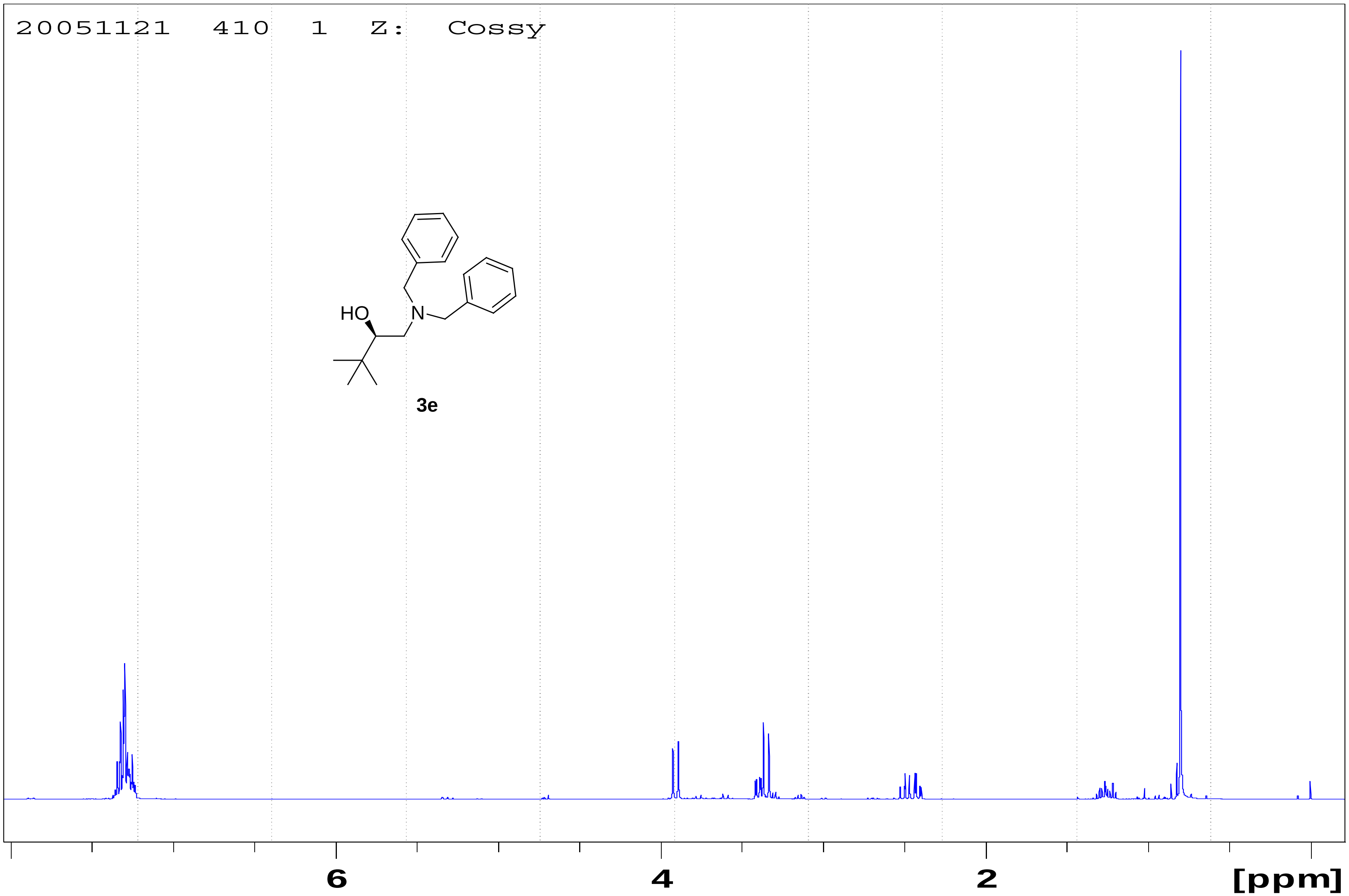


20060106 170 1 Z: Cossy
HO
N
3c
8
6
4
2
[ppm]

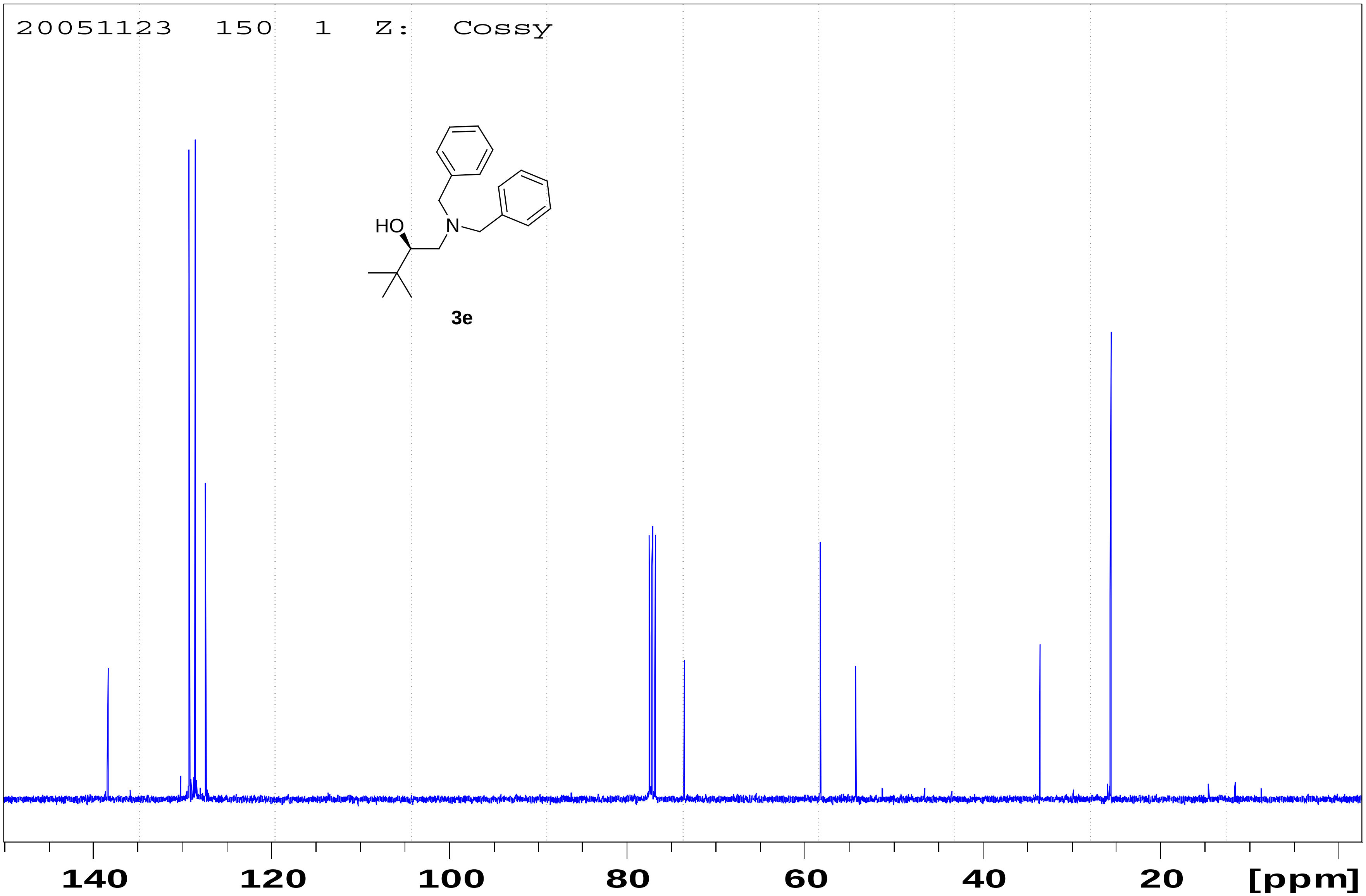

20060106 390 1 Z: Cossy
HO
N
3c
140
120
100
80
60
40
20
[ppm]

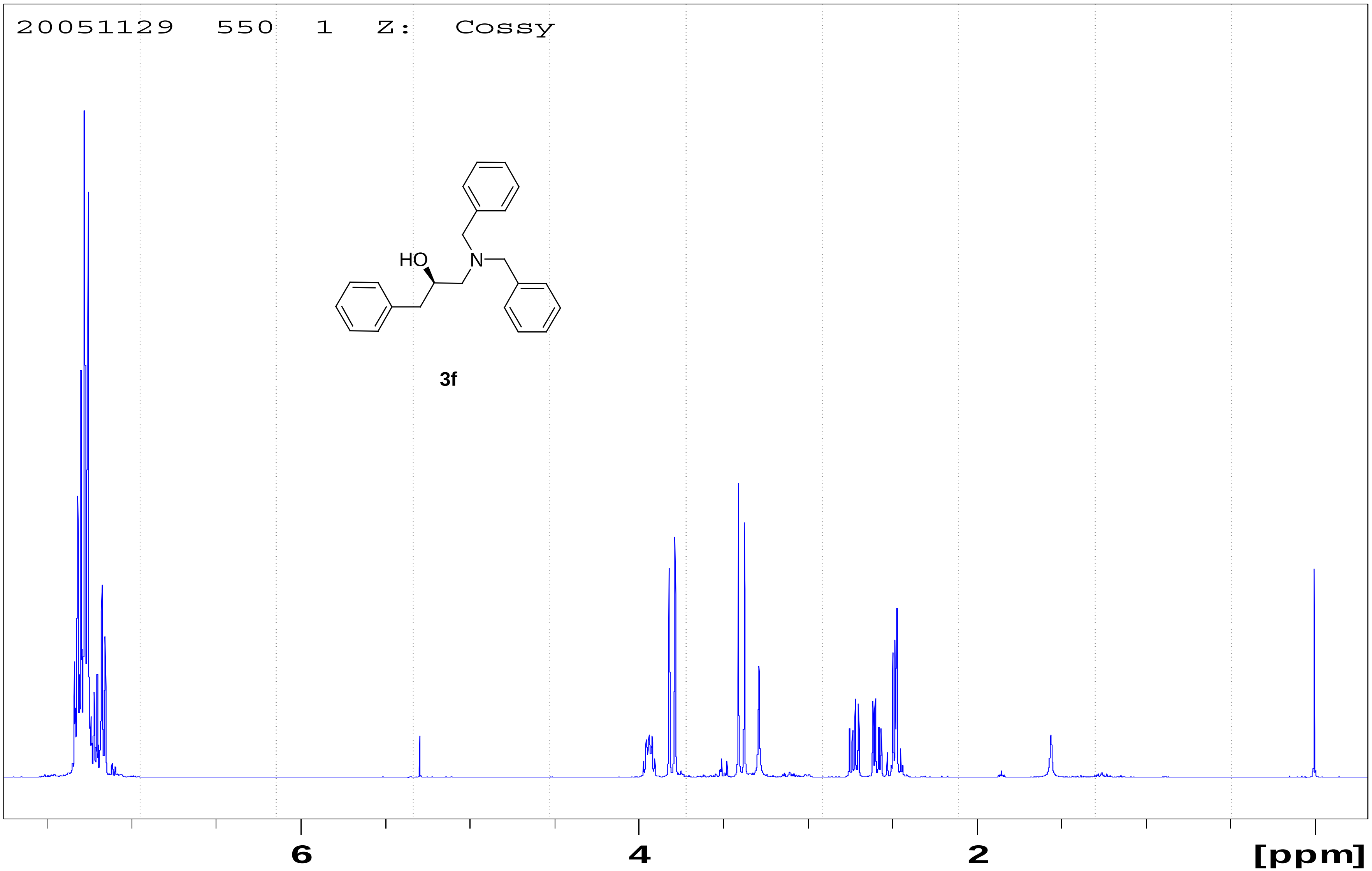

20070116 580 1 Z: Cossy
HO
N
3d
8
6
4
2
[ppm]

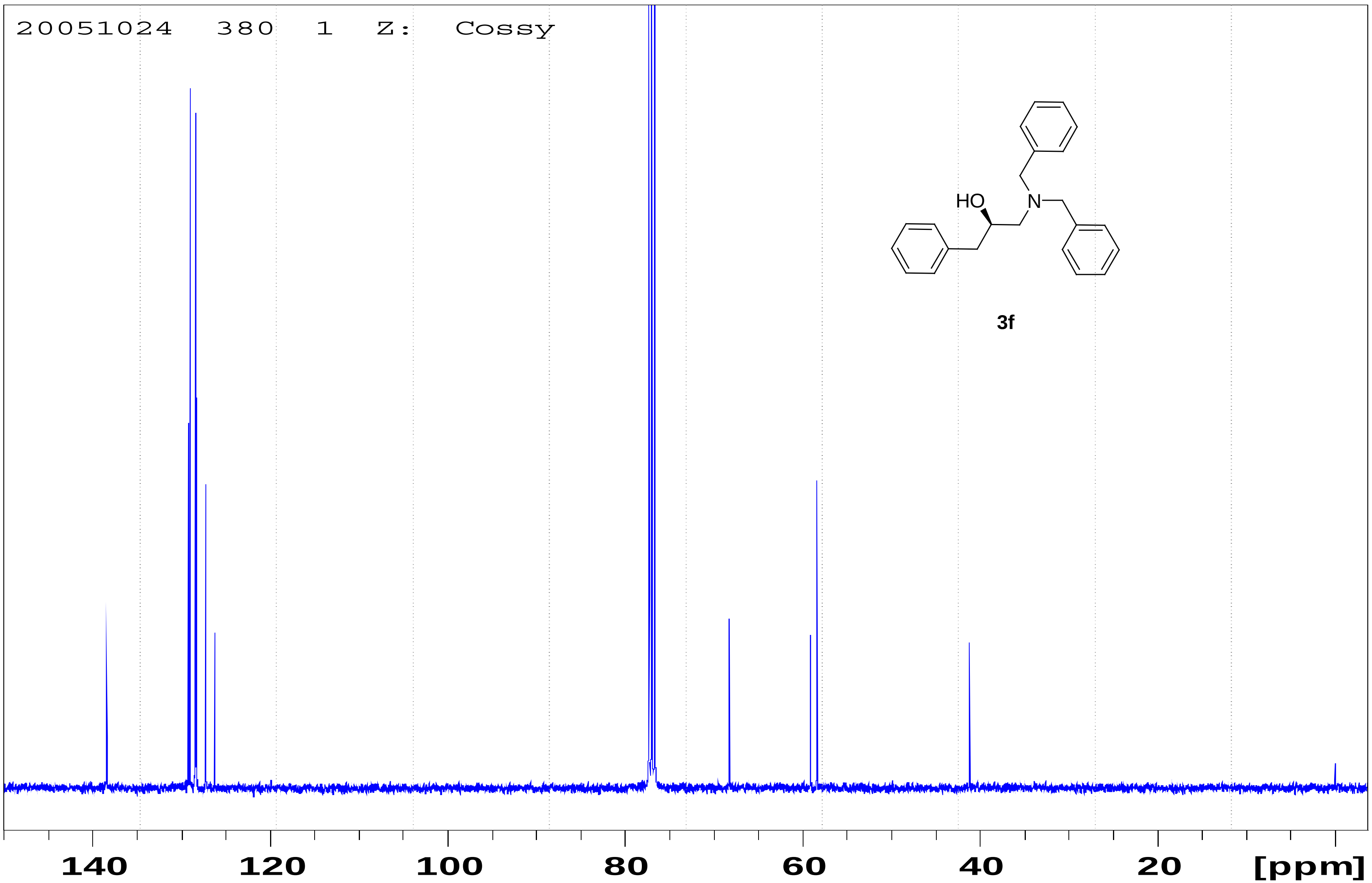

20070117 722 1 Z: Cossy
HO
N
3d
140
120
100
80
60
40
20
[ppm]

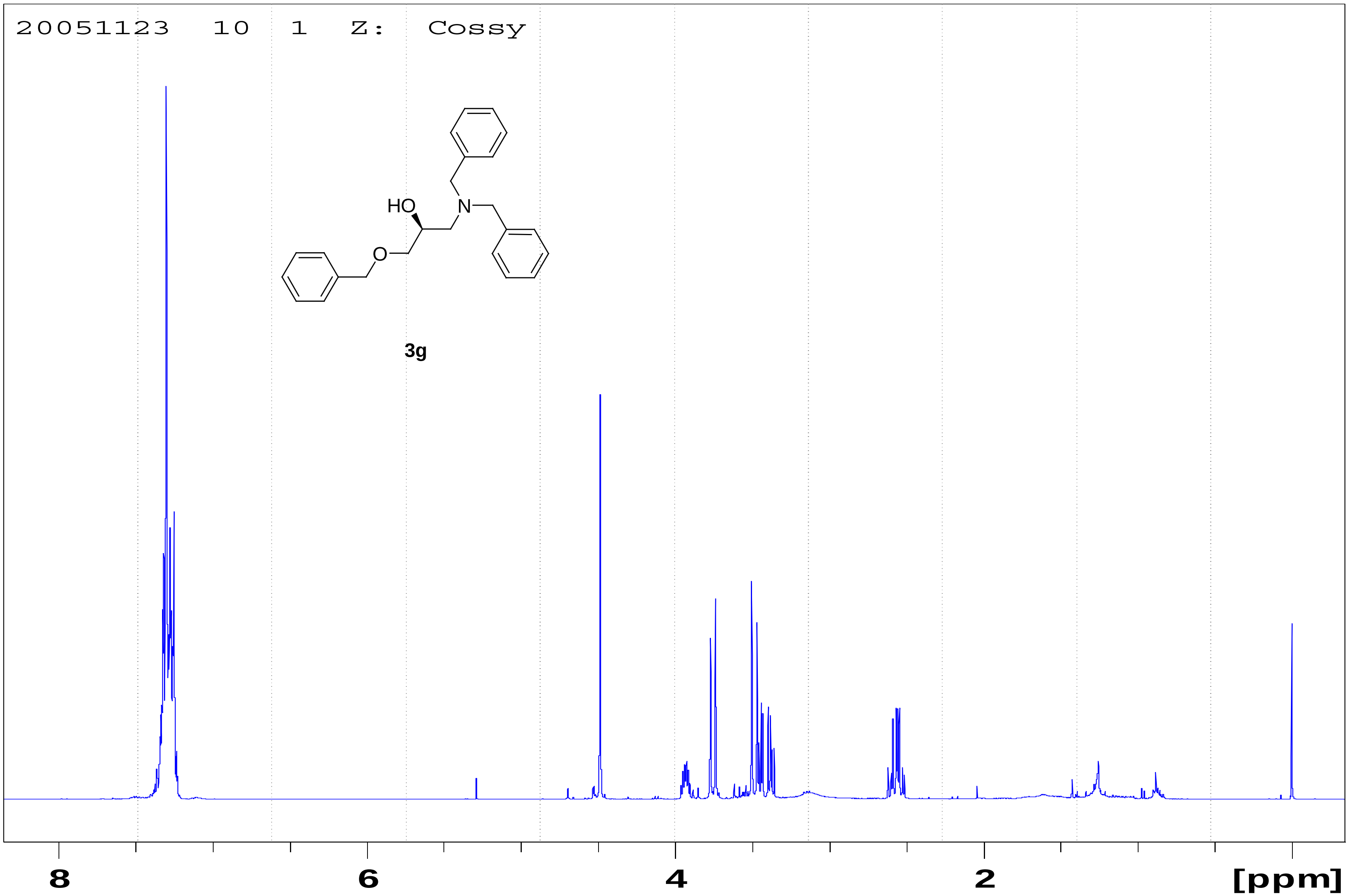


3e

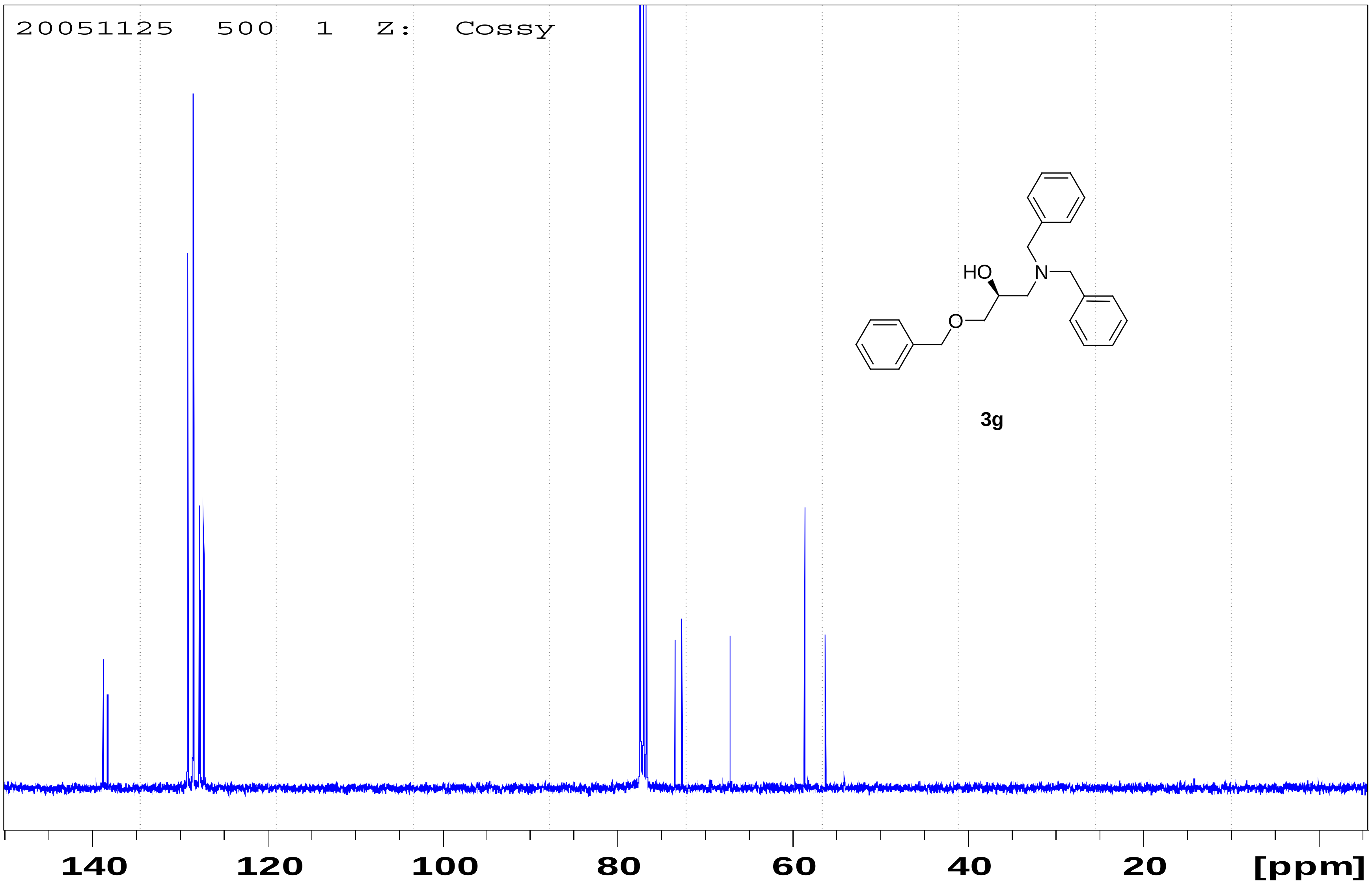

20051123 150 1 Z: Cossy
HO
N
3e
140
120
100
80
60
40
20
[ppm]

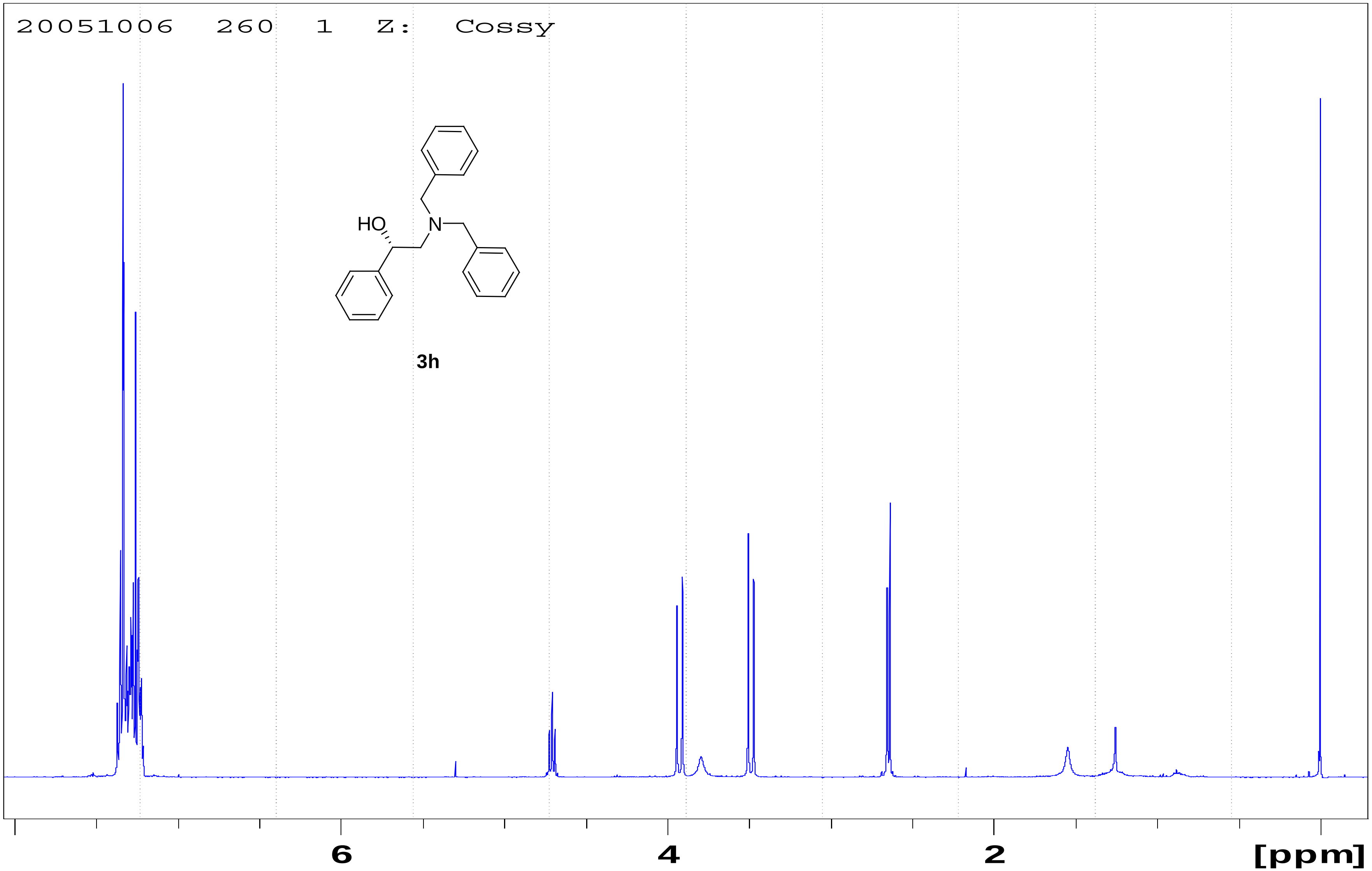

20051129 550 1 Z: Cossy
HO
N
3f
6
4
2
[ppm]

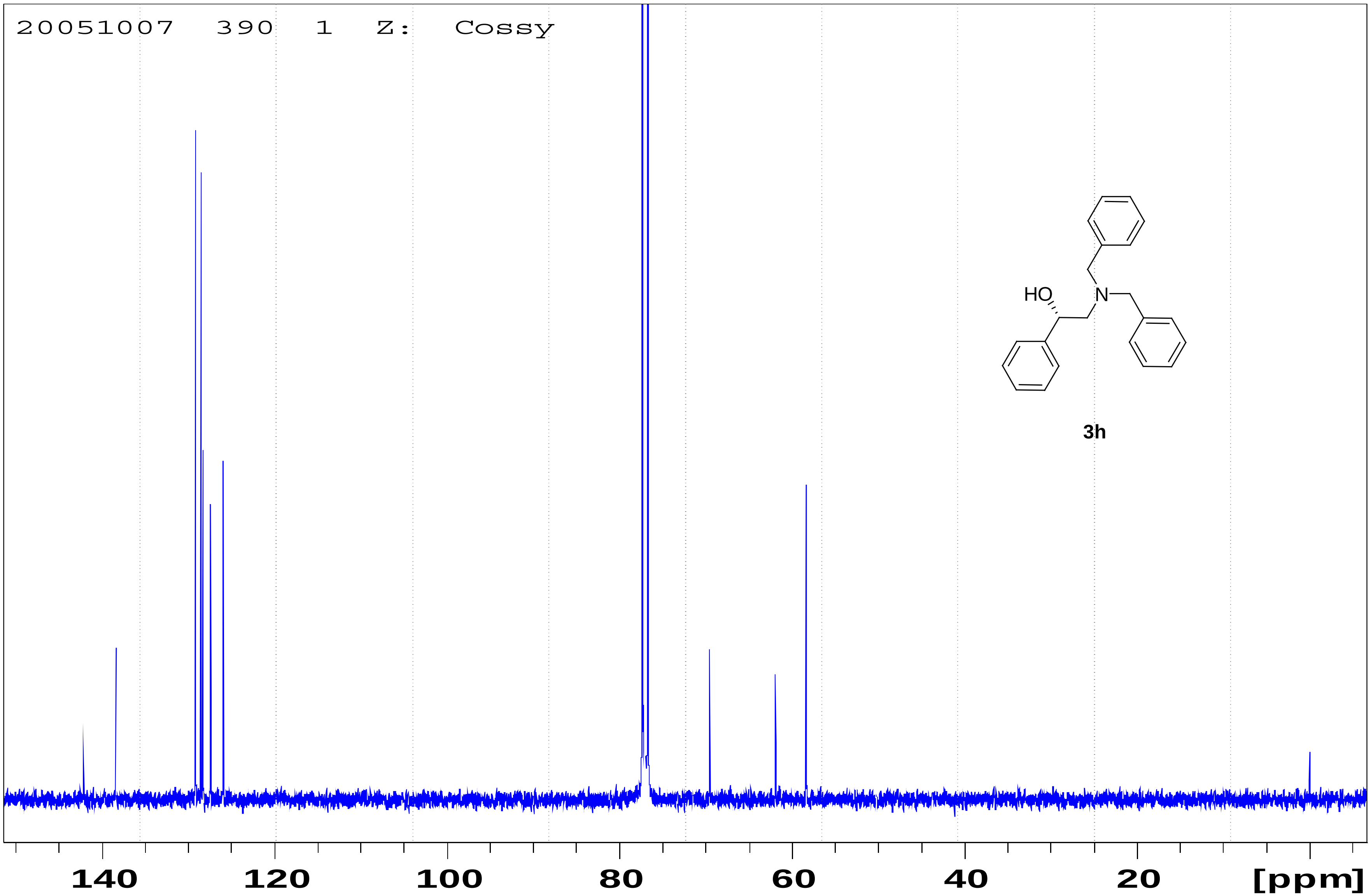

20051024 380 1 Z: Cossy
HO
N
3f
140
120
100
80
60
40
20
[ppm]

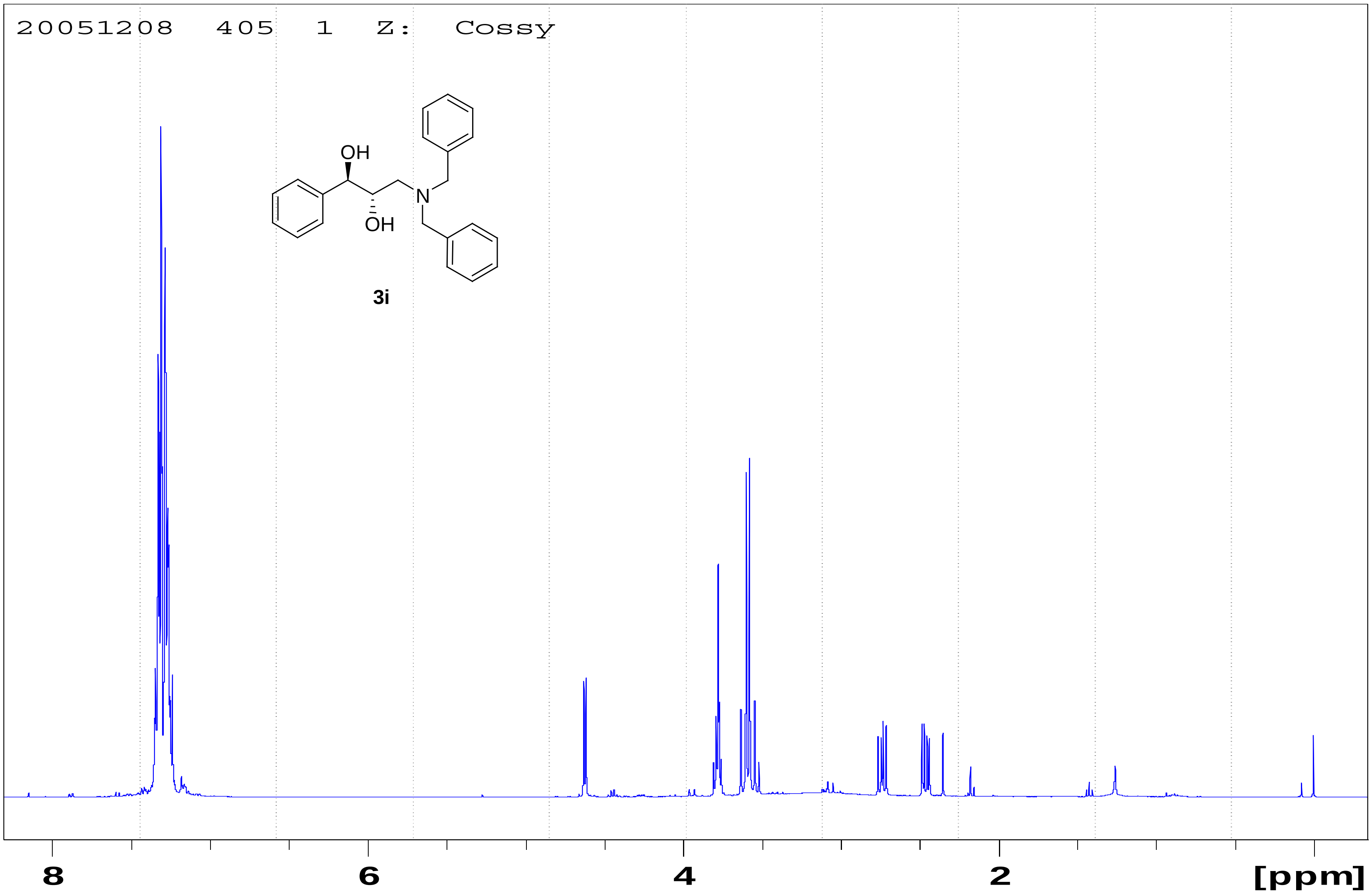


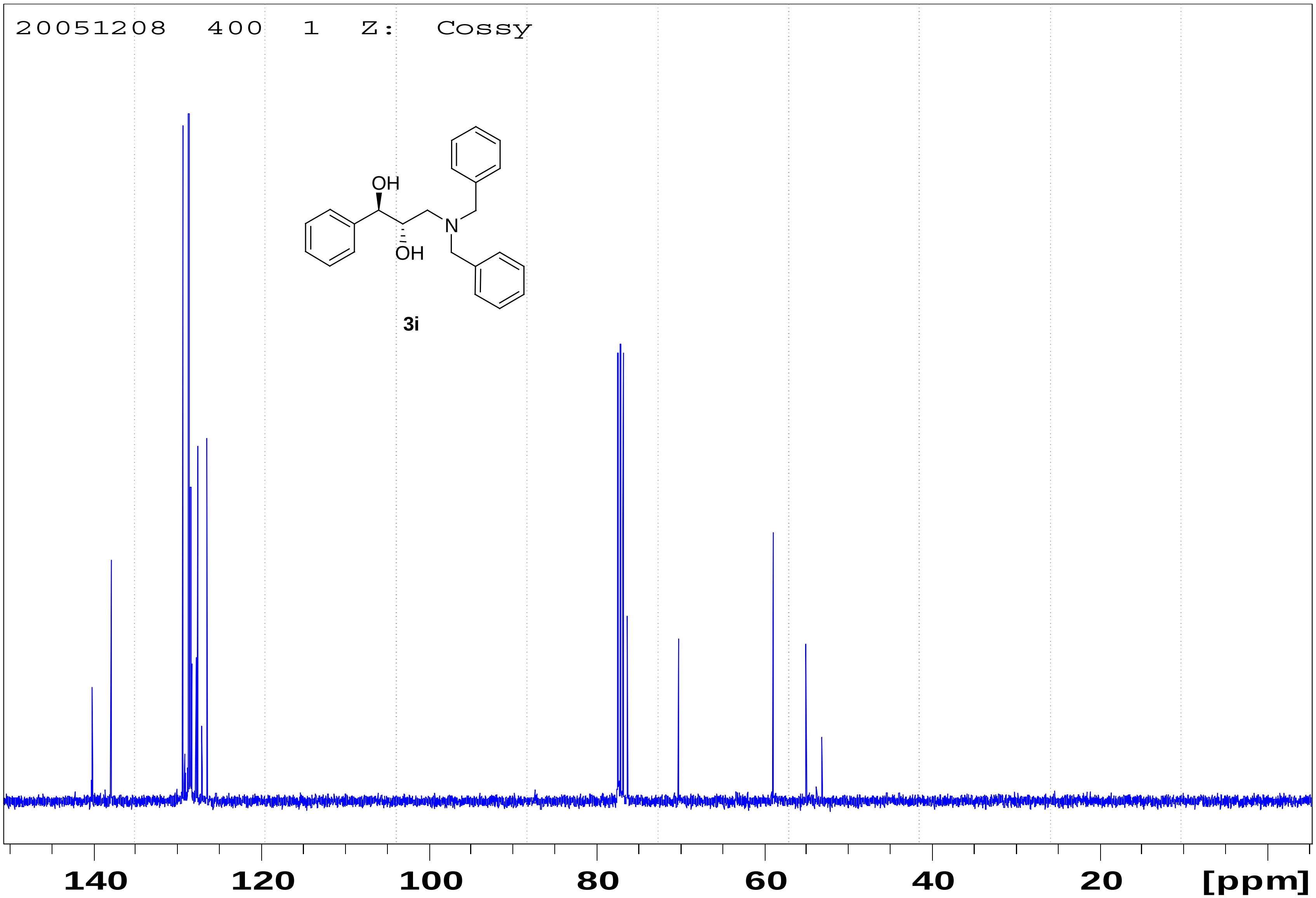
20051123 10 1 Z: Cossy
HO
O
N
3g
8
6
4
2
[ppm]

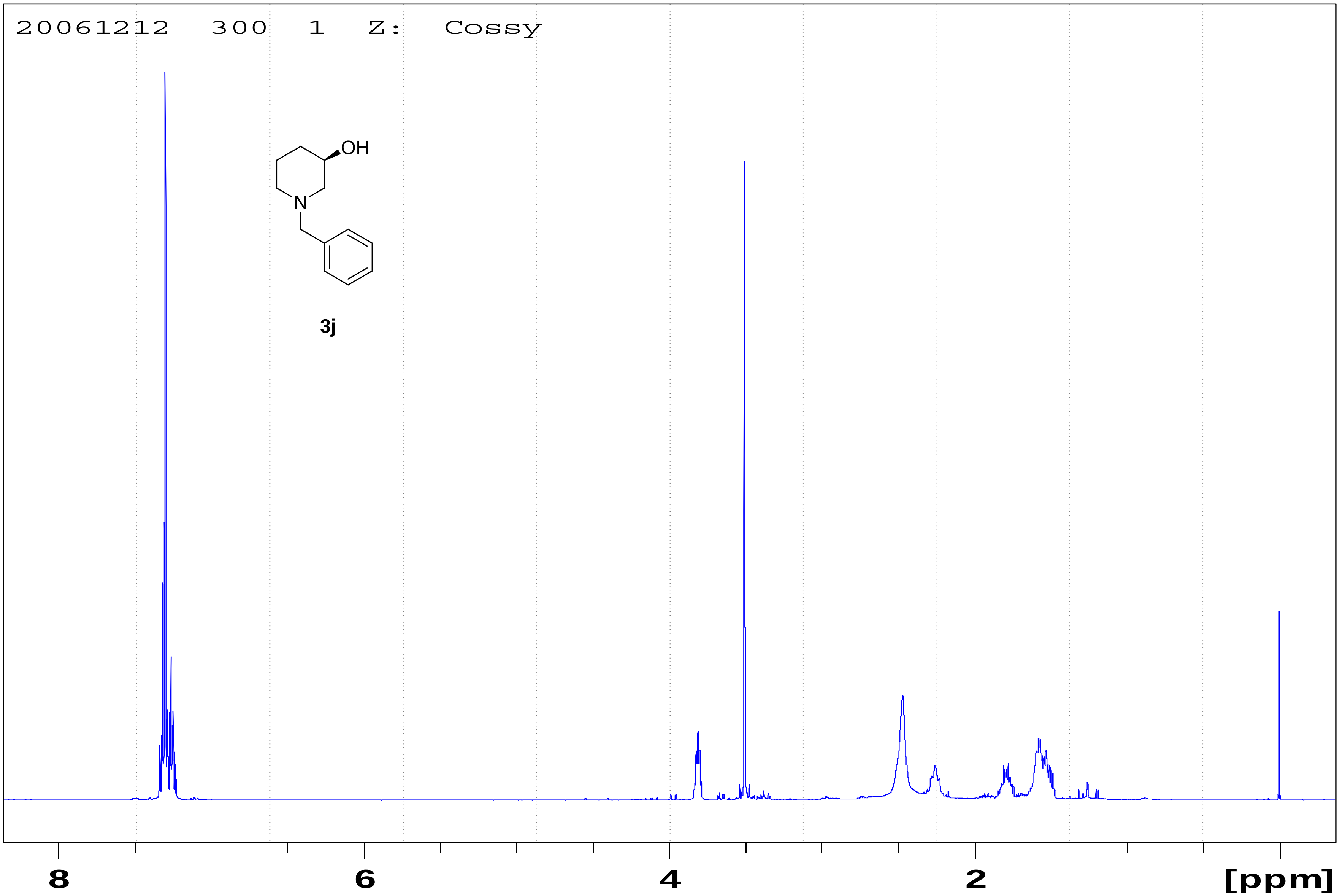

20051125 500 1 Z: Cossy
HO
N
O
3g
140
120
100
80
60
40
20
[ppm]

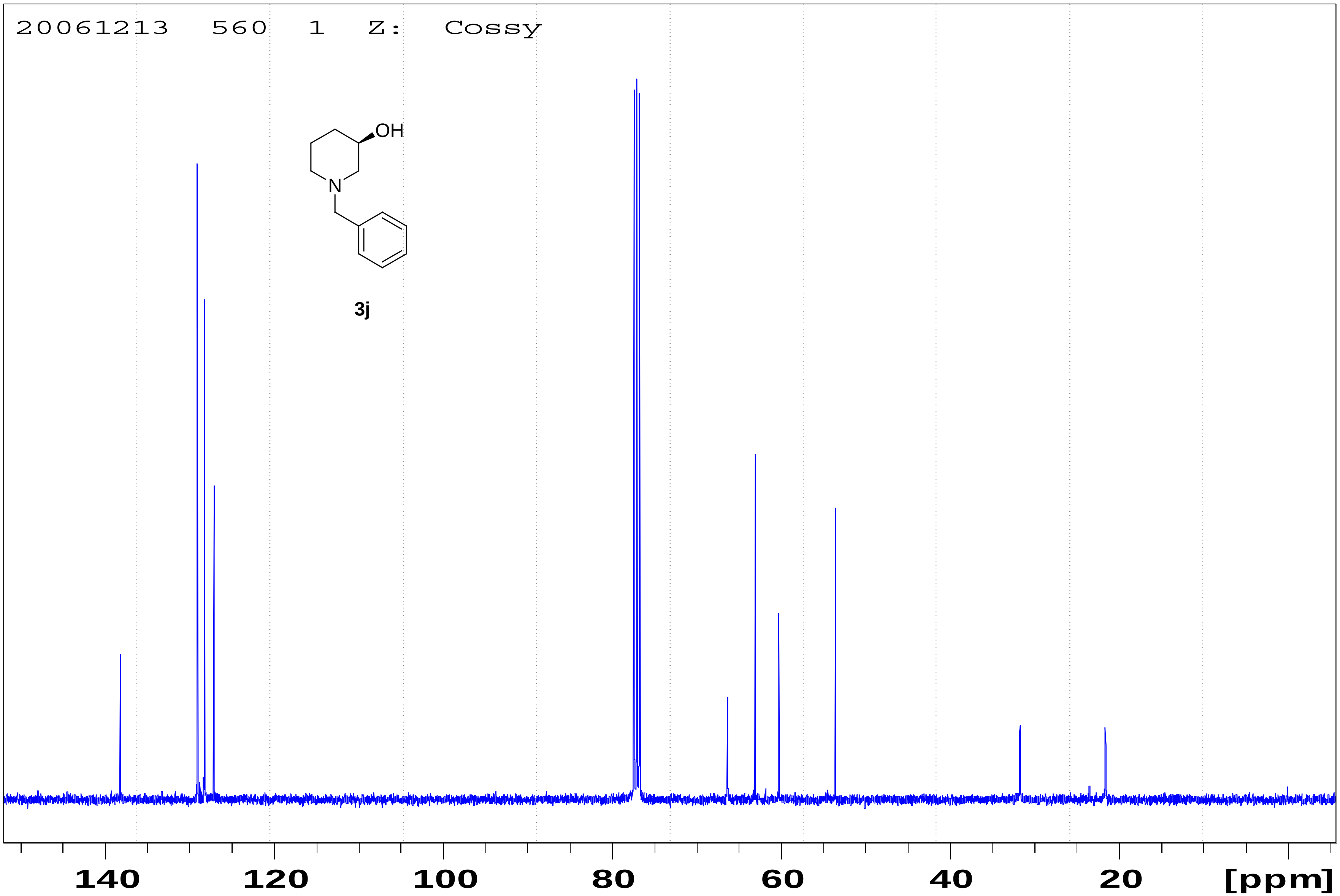

20051006 260 1 Z: Cossy
HO
N
3h
6
4
2
[ppm]


20051007 390 1 Z: Cossy
HO
N
3h
140
120
100
80
60
40
20
[ppm]

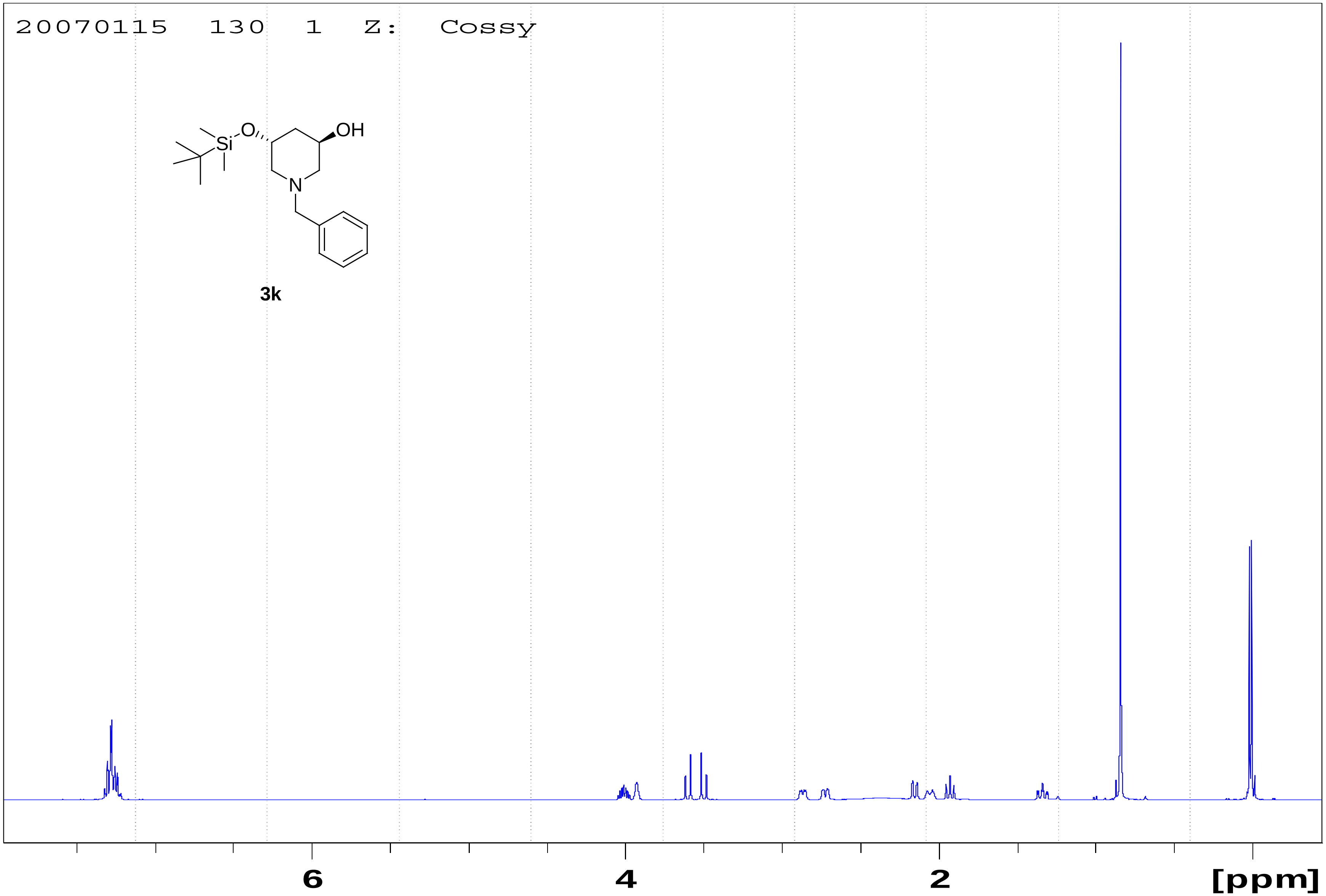

20051208 405 1 Z: Cossy
OH
OH
N
3i
8
6
4
2
[ppm]


20051208 400 1 Z: Cossy
OH
OH
N
3i
140
120
100
80
60
40
20
[ppm]

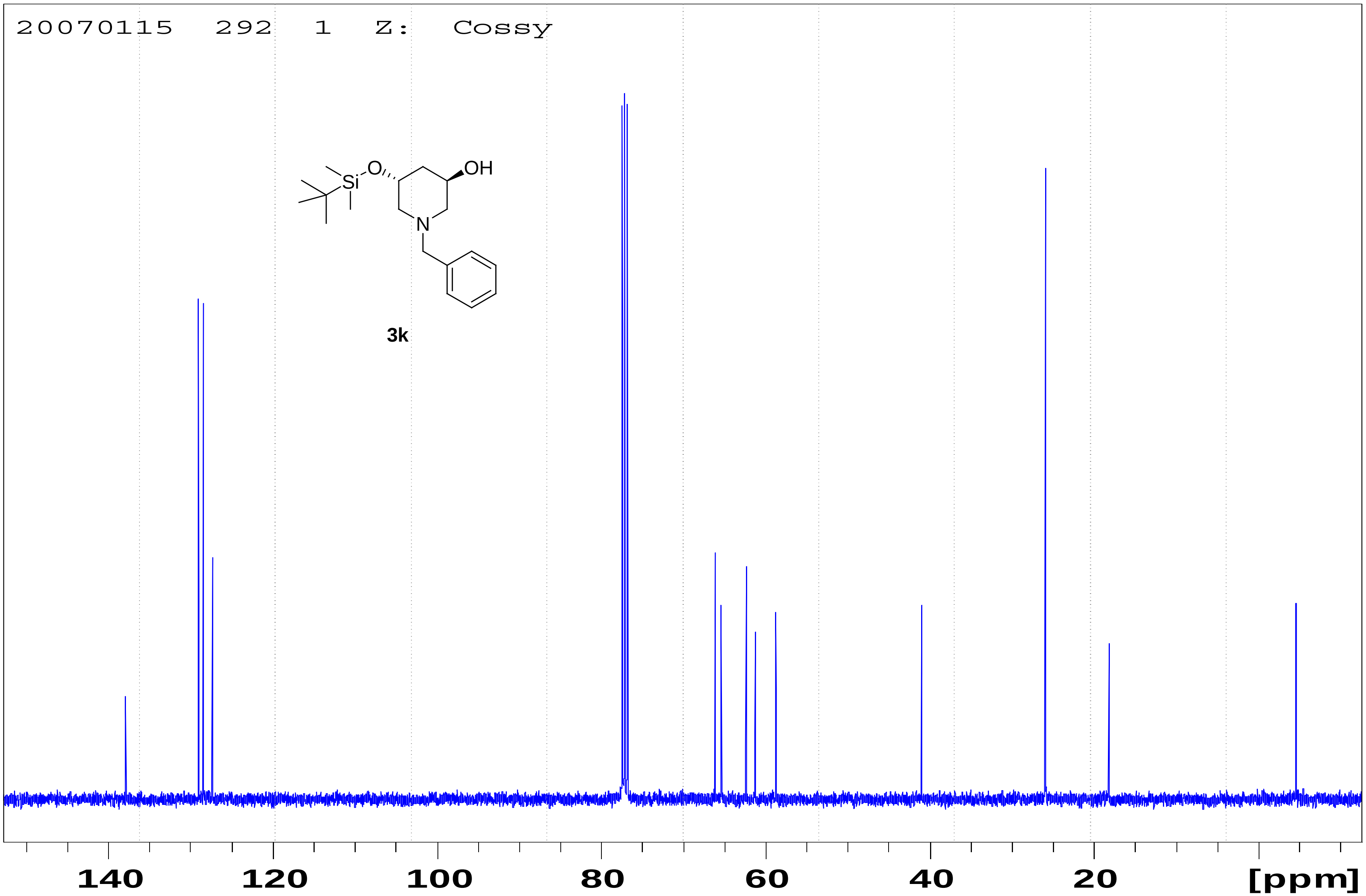

20061212 300 1 Z: Cossy
OH
N
3j
8
6
4
2
[ppm]

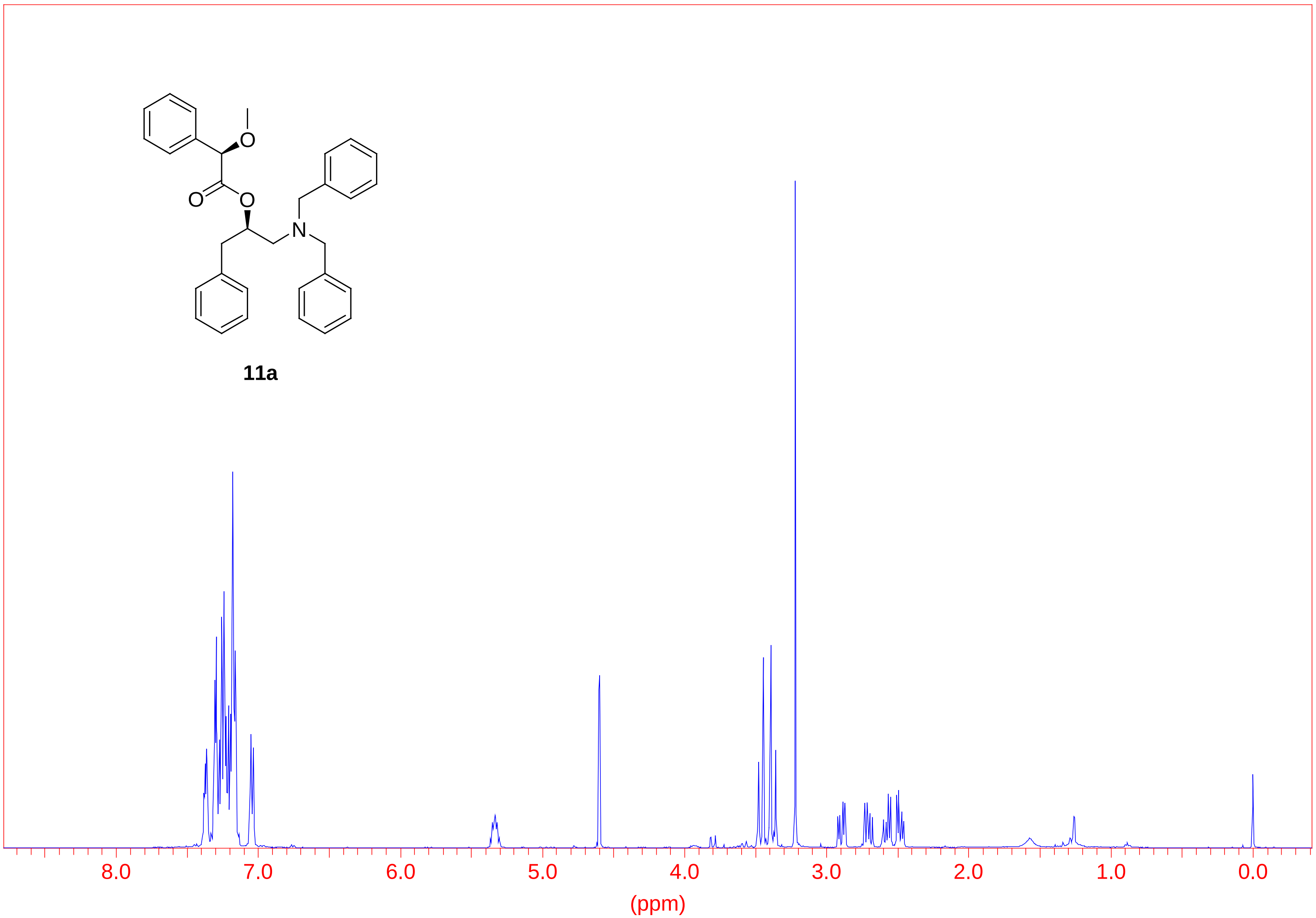


20061213 560 1 Z: Cossy
OH
N
3j
140
120
100
80
60
40
20
[ppm]

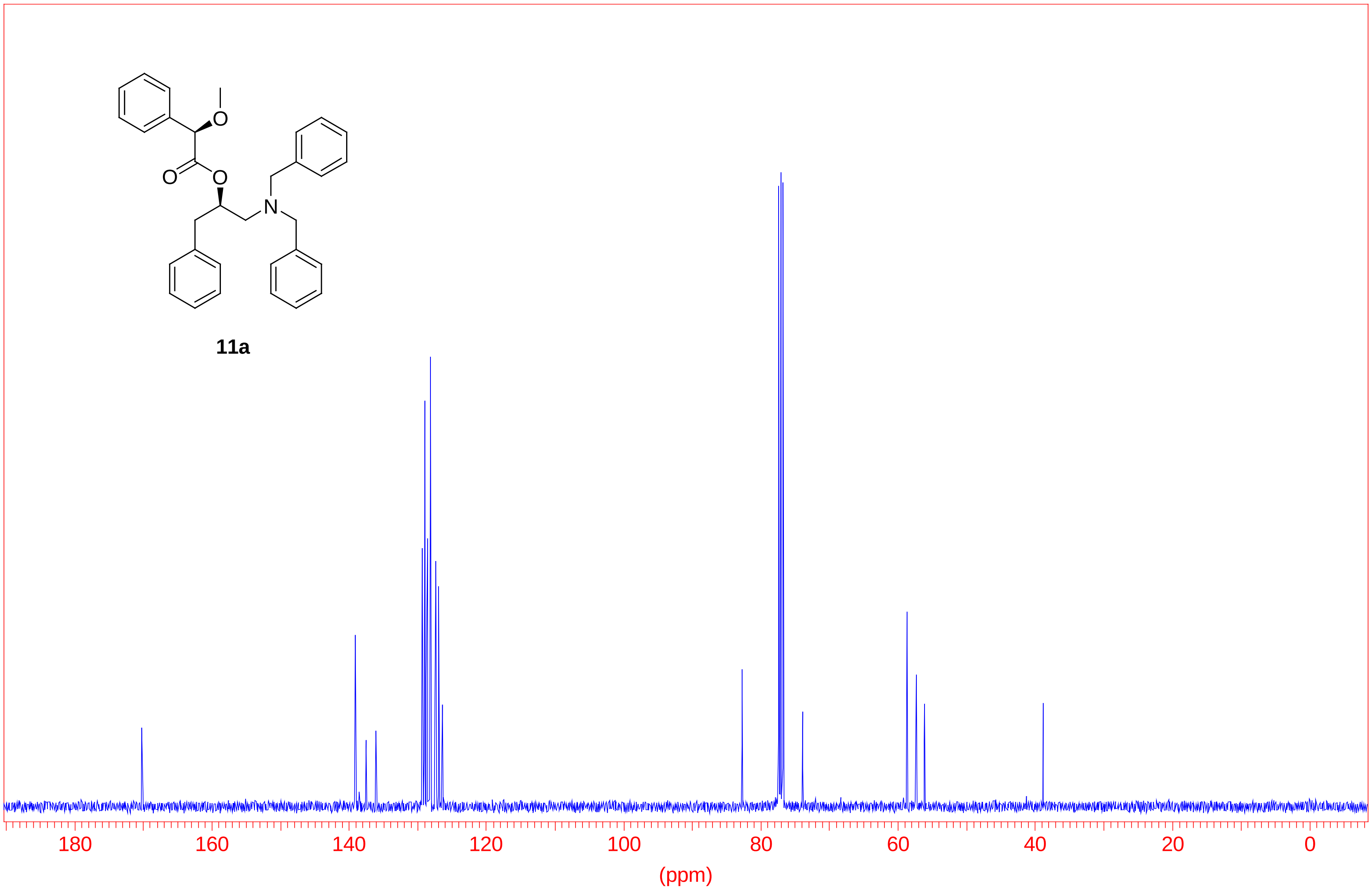


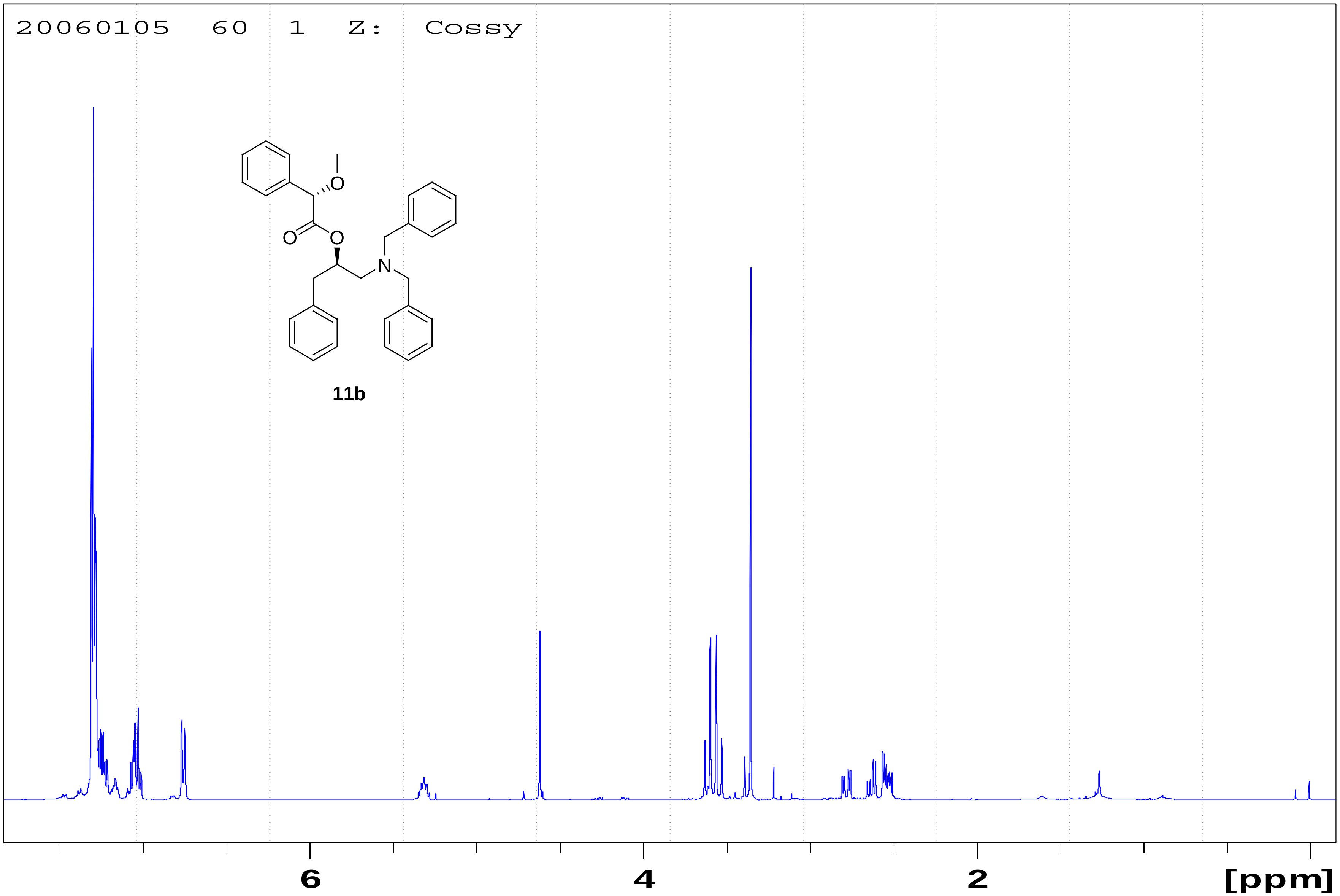
3k


20070115 292 1 Z: Cossy
OH
O
Si
N
3k
140
120
100
80
60
40
20
[ppm]

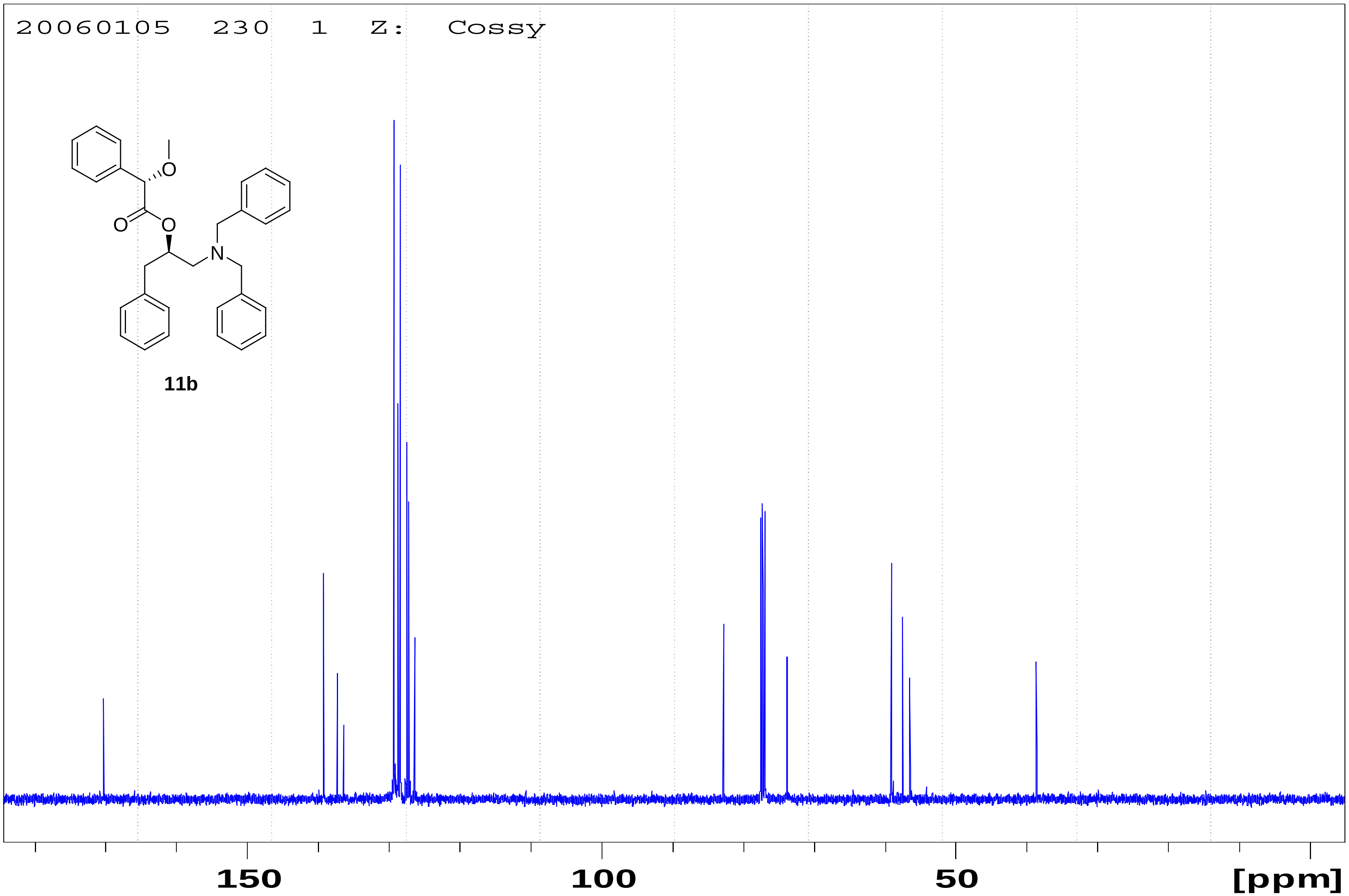


11a
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
(ppm)

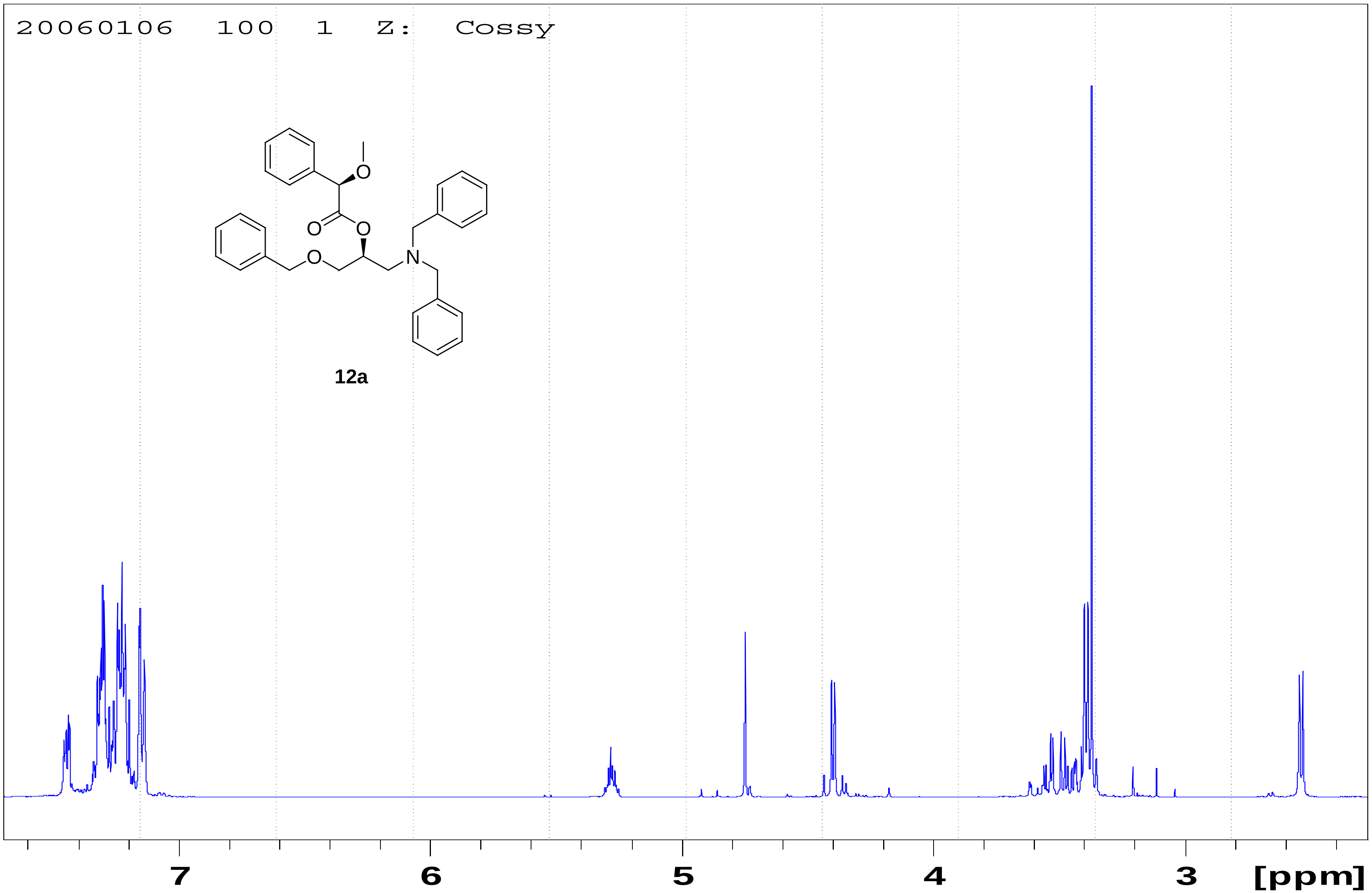

11a
180
160
140
120
100
80
60
40
20
0
(ppm)

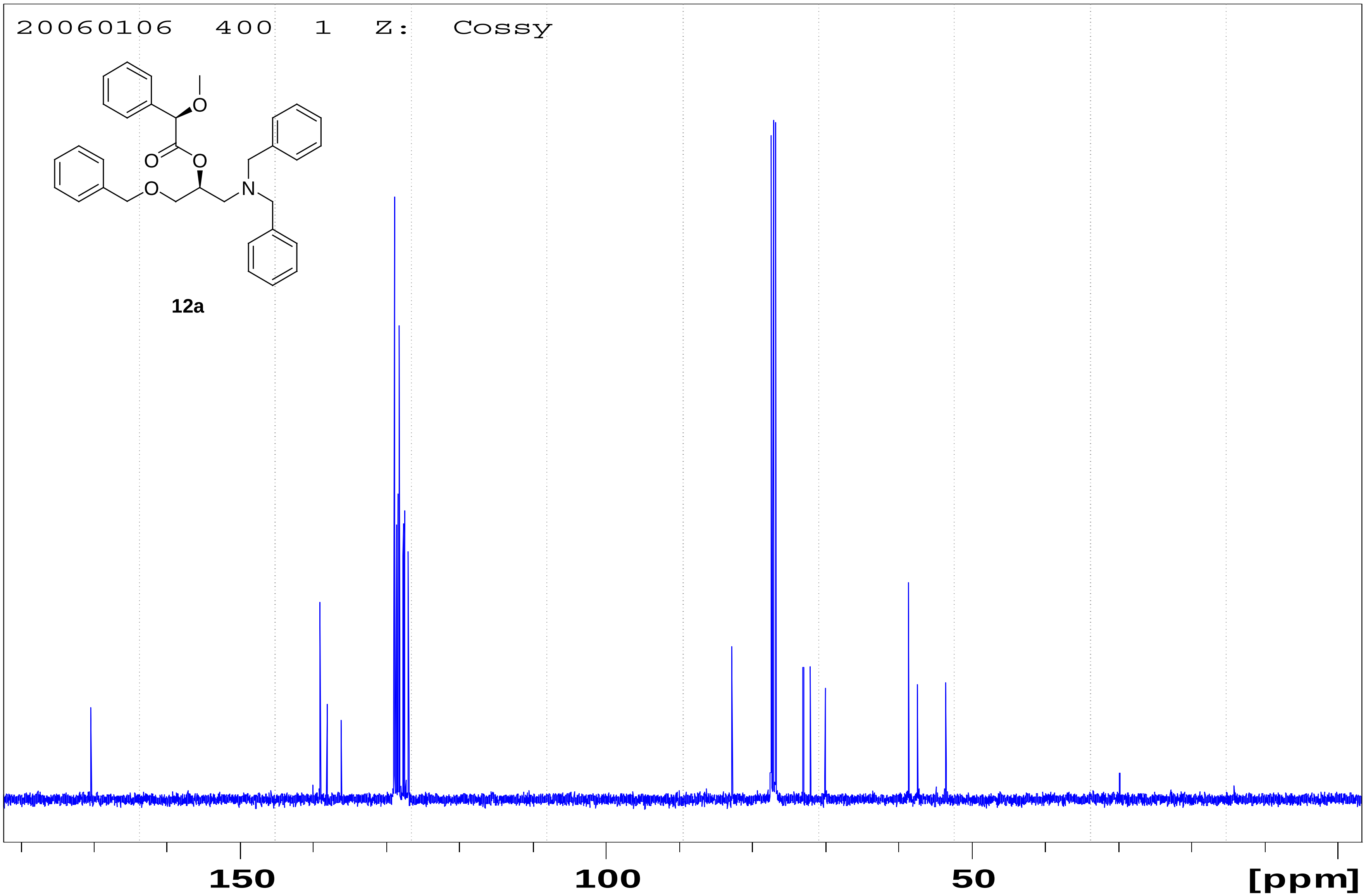


20060105 60 1 Z: Cossy
11b
6
4
2
[ppm]

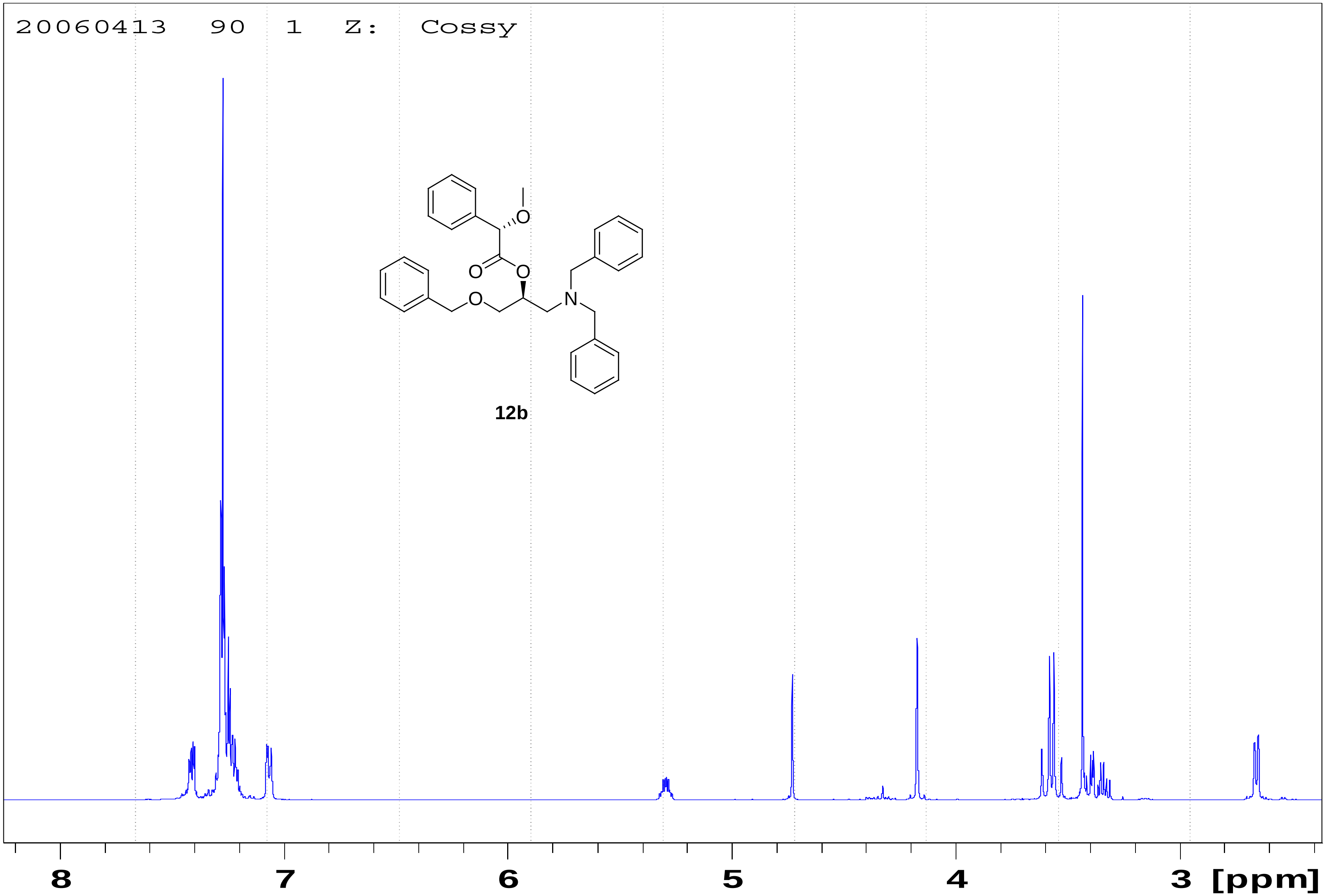


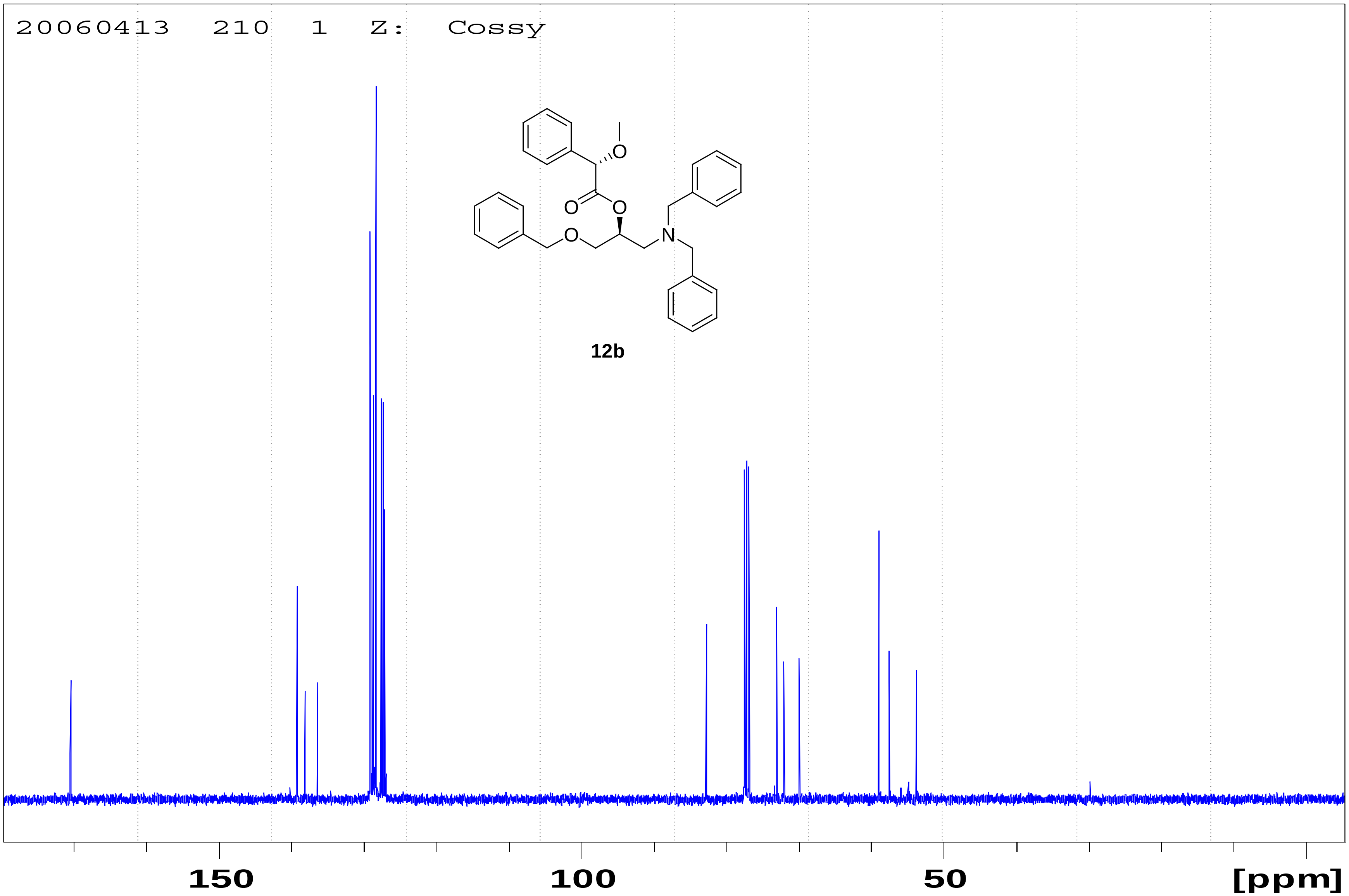
20060105 230 1 Z: Cossy
11b
150
100
50
[ppm]


20060106 100 1 Z: Cossy
12a
7
6
5
4
3
[ppm]



20060106 400 1 Z: Cossy
O
O
O
O
N
12a
150
100
50
[ppm]


20060413 90 1 Z: Cossy
12b
8
7
6
5
4
3 [ppm]



20060413 210 1 Z: Cossy
12b
150
100
50
[ppm]