Supporting Information

Synthesis and Biological Evaluation of a New Calcium Channel Agonist

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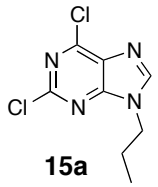
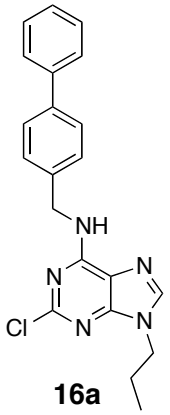
Contents

General information. All moisture and air-sensitive reactions were performed using syringe-septum cap techniques under an inert atmosphere (N_2 or Ar) in glassware that was dried in an oven at 140 °C for at least 2 h prior to use. Reactions carried out at a temperature below 0 °C employed a CO_2/acetone bath. All reagents and solvents were used as received unless otherwise specified. Triethylamine, *N,N*-dimethylaniline and pyridine were distilled over CaH_2. THF and Et_2O were distilled over sodium/benzophenone ketyl. Dichloromethane and toluene were purified using an alumina column filtration system. Anhydrous MeOH and Et_2O were purchased from Acros Organics and Fisher Scientific, respectively. Anhydrous DMF was purchased from Acros Organics or distilled and stored over 4 Å molecular sieves. Analytical thin-layer chromatography (TLC) was performed on pre-coated SiO_2 60 F254 plates (250 µm layer thickness) available from Merck. Visualization was accomplished by UV irradiation at 254 nm and/or by staining with Vaughn's reagent (4.8 g $(NH_4)_6Mo_7O_{24}$•4 H_2O and 0.2 g $Ce(SO_4)_2$•4 H_2O in 100 mL of a 3.5 N H_2SO_4 solution), a $KMnO_4$ solution (1.5 g $KMnO_4$ and 1.5 g K_2CO_3 in 100 mL of a 0.1% NaOH solution), a ninhydrin solution (2 g ninhydrin in 100 mL of EtOH), a PMA solution (5 g of phosphomolybdic acid in 100 mL of EtOH), or a *p*-anisaldehyde solution (2.5 mL of *p*-anisaldehyde, 2 mL of AcOH and 3.5 mL of conc. aq. H_2SO4 in 100 mL of EtOH). Preparative thin-layer chromatography was performed on pre-coated SiO_2 GF (UV_{254}) 1000 microns (20 x 20 cm) plates available from Analtech. Flash column chromatography was performed using SiO_2 60 (particle size 0.040–0.055 mm, 230–400 mesh, or Silicycle SiliaFlash® P60, 40-63 µm). Melting points were determined on a Meltemp capillary melting point apparatus fitted with a Fluke 51 II digital thermometer. Infrared spectra were recorded on a Smiths IdentifyIR ATR spectrometer or a Perkin Elmer Spectrum 100 FT-IR spectrometer using the Universal ATR Sampling Accessory for both oil and solid compounds. ^{1}H NMR and ^{13}C NMR spectra were obtained on Bruker Avance 300, 400 or 600 instruments at 300/75 MHz, 400/100 MHz or 600/150 MHz, respectively. Chemical shifts were reported in parts per million (ppm) as referenced to residual solvent. ^{1}H NMR spectra are tabulated as follows: chemical shift, multiplicity (app = apparent, b = broad, s = singlet, d = doublet, t = triplet, q = quartet, quint = quintuplet, sext = sextuplet, sept = septuplet, m = multiplet), number of protons, coupling constant(s). ^{13}C NMR were obtained using a proton-decoupled pulse sequence. Mass spectra were obtained on a Waters Autospec double focusing mass spectrometer (EI) or a Waters Q-Tof mass spectrometer (ESI).



15a

2,6-Dichloro-9-propyl-9*H*-purine (15a).[1] To a solution of 2,6-dichloro-9*H*-purine **14** (0.490 mg, 2.59 mmol) in anhydrous DMSO (3.0 mL) was added K_2CO_3 (1.10 g, 7.96 mmol) followed by 1-bromopropane (1.62 g, 13.1 mmol) at 16-18 °C (*i*-PrOH bath in a Dewar flask covered with aluminum foil). The reaction mixture was stirred at 16-18 °C for 17 h, quenched with water and extracted with EtOAc. The combined organic layers were washed with brine, dried ($MgSO_4$), concentrated, and purified by chromatography on SiO_2 (hexanes, 100%, to hexanes/EtOAc, 1:1) to yield **15a** (0.465 g, 2.01 mmol, 78% yield) as an off-white solid: IR (ATR, neat) 3677, 3078, 2974, 2939, 2880, 1596, 1553, 1496, 1466, 1442, 1408, 1383, 1370, 1347, 1312, 1270, 1229, 1196, 1180, 1141, 1084, 957, 901, 875, 860, 812, 785, 774, 681 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.10 (s, 1 H), 4.21 (t, 2 H, *J* = 7.2 Hz), 1.93 (sext, 2 H, *J* = 7.4 Hz), 0.94 (t, 3 H, *J* = 7.5 Hz); ^{13}C NMR (151 MHz, $CDCl_3$) δ 153.2, 152.7, 151.5, 146.0, 46.2, 23.1, 11.1.



16a

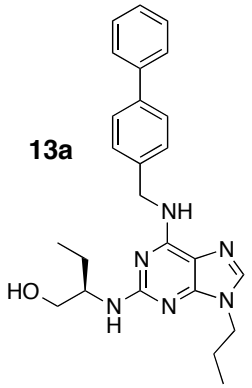
***N*-(Biphenyl-4-ylmethyl)-2-chloro-9-propyl-9*H*-purin-6-amine (16a).**[2] To a solution of **15a** (58.0 mg, 0.251 mmol) in n-BuOH (1.0 mL) were added 4-phenylbenzylamine (52.0

[1] Lambertucci, C.; Cristalli, G.; Dal Ben, D.; Kachare, D. D.; Bolcato, C.; Klotz, K.-N.; Spalluto, G.; Volpini, R., "New 2,6,9-trisubstituted adenines as adenosine receptor antagonists: A preliminary SAR profile." *Purinergic Signalling* **2007**, *3*, 339-346.

[2] Trova, M. P.; Barnes, K. D.; Barford, C.; Benanti, T.; Bielaska, M.; Burry, L.; Lehman, J. M.; Murphy, C.; O'grady, H.; Peace, D.; Salamone, S.; Smith, J.; Snider, P.; Toporowski, J.; Tregay, S.; Wilson, A.; Wyle, M.; Zheng, X.; Friedrich, T. D. Biaryl purine derivatives as potent antiproliferative agents: Inhibitors of cyclin dependent kinases. Part I. *Bioorg. Med. Chem. Lett.* **2009**, *19*, 6608-6612.

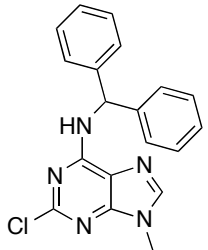
mg, 0.267 mmol) and triethylamine (40.6 mg, 0.402 mmol) under an N_2 atmosphere at room temperature. The reaction mixture was heated in a microwave at 120 °C for 20 min. The solvent was evaporated, and the crude residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined organic extracts were dried ($MgSO_4$) and concentrated to yield a colorless solid, which was resuspended (hexanes/Et_2O, 3:1), filtered, triturated (hexanes/Et_2O, 3:1) and dried under high-vacuum to yield **16a** (86.2 mg, 0.228 mmol, 91% yield) as a colorless solid: IR (ATR, neat) 3145, 2964, 1619, 1577, 1304, 1254 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.56 (d, 2 H, *J* = 8.4 Hz), 7.56 (d, 2 H, *J* = 7.8 Hz), 7.47-7.40 (m, 5 H), 7.35 (t, 1 H, *J* = 7.5 Hz), 6.88 (bm, 1 H), 4.87 (bs, 2 H), 4.05 (t, 2 H, *J* = 6.6 Hz), 1.85 (sext, 2 H, *J* = 7.4 Hz), 0.91 (t, 3 H, *J* = 6.9 Hz); ^{13}C NMR (151 MHz, $CDCl_3$) δ 155.3, 154.7, 150.4, 140.7, 140.4, 137.2, 128.9, 128.6, 127.5 (2 C), 127.2, 118.8, 45.6, 44.5, 23.4, 11.2.

13a



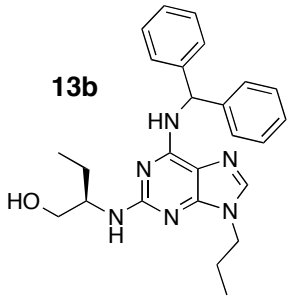
(*R*)-2-(6-(Biphenyl-4-ylmethylamino)-9-propyl-9*H*-purin-2-ylamino)butan-1-ol (13a). A mixture of **16a** (50.0 mg, 0.128 mmol) and (*R*)-(-)-2-amino-1-butanol (60.9 mg, 0.642 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 8 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (4x). The combined organic layers were washed with warmed water (50-55 °C, 2x), dried ($MgSO_4$), concentrated, and dried under high vacuum at 50 °C (oil bath) for 2 h to yield a yellow solid. Upon the addition of Et_2O to the yellow solid, an off-white solid precipitated. The solid was further washed with Et_2O (3x) and dried under high-vacuum at 40 °C overnight to yield **13a** (32.6 mg, 0.0757 mmol, 59%) as an off-white solid: Mp 130-131 °C; IR (ATR, neat) 3270, 2962, 2931, 1599, 1488, 1349 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.57 (app d, 2 H, *J* = 7.8 Hz), 7.54 (app d, 2 H, *J* = 7.8 Hz), 7.47-7.38 (m,

4 H), 7.37-7.28 (m, 2 H), 6.46 (bs, 1 H), 5.26-5.08 (bm, 1 H), 4.97 (s, 1 H), 4.91-4.66 (bm, 2 H), 3.92 (t, 2 H, *J* = 6.9 Hz), 3.90-3.85 (bm, 1 H), 3.81 (bd, 1 H, *J* = 12.0 Hz), 3.63 (app t, 1 H, *J* = 9.0 Hz), 1.82 (sext, 2 H, *J* = 7.2 Hz), 1.67-1.49 (m, 2 H), 1.00 (t, 3 H, *J* = 7.2 Hz), 0.91 (t, 3 H, *J* = 7.2 Hz); ^{13}C NMR (151 MHz, $CDCl_3$) δ 160.2, 154.9, 150.7, 140.7, 140.2, 137.9, 137.1, 128.7, 128.1, 127.2, 127.0, 114.3, 68.2, 56.2, 45.0, 44.0, 25.0, 23.1, 11.2, 10.9; HRMS (ES$^+$) *m/z* calcd for $C_{25}H_{31}N_6O$ $[M+H]^+$ 431.2559, found 431.2532.



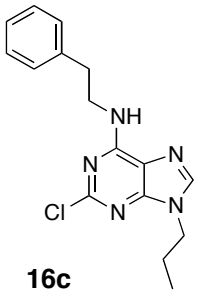
16b

2-Chloro-*N*-(2,2-diphenylethyl)-9-propyl-9*H*-purin-6-amine (16b). To a solution of **15a** (71.0 mg, 0.307 mmol) in *n*-BuOH (1.0 mL) was added aminodiphenylmethane (61.5 mg, 0.326 mmol) and triethylamine (50.1 mg, 0.495 mmol) under an N_2 atmosphere. The reaction mixture was heated in a microwave reactor at 120 °C for 20 min. The solvent was evaporated, and the residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined organic extracts were dried ($MgSO_4$) and concentrated to yield a colorless solid, which was resuspended (hexanes/Et_2O, 3:1), filtered, and triturated (hexanes/Et_2O, 3:1) to obtain an off-white solid. The filtrate was concentrated, resuspended (hexanes/Et_2O, 3:1), and filtered to obtain additional product. After drying on high-vacuum, **16b** (74.4 mg, 0.197 mmol, 65%) was obtained as an off-white solid: IR (ATR, neat) 3250, 2964, 1612, 1574, 1304, 1218 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.54 (bs, 1 H), 7.35-7.20 (m, 9 H), 7.17 (s, 1 H), 6.78 (d, 1 H, *J* = 6.6 Hz), 4.09-3.90 (bm, 2 H), 1.95-1.69 (bm, 2 H), 0.90 (t, 3 H, *J* = 6.9 Hz); ^{13}C NMR (151 MHz, $CDCl_3$) δ 154.4, 154.2, 150.3, 141.3, 140.4, 127.7, 127.5, 127.3, 118.5, 57.1, 45.4, 23.2, 11.0; LCMS (ESI) *m/z* calcd for $C_{21}H_{21}N_5Cl$ $[M+H]^+$ 378.1, found 378.1.



13b

(*R*)-2-(6-(2,2-Diphenylethylamino)-9-propyl-9*H*-purin-2-ylamino)butan-1-ol (13b). A mixture of **16b** (50.0 mg, 0.128 mmol) and (*R*)-(-)-2-amino-1-butanol (60.9 mg, 0.642 mmol) were heated in a vial immersed in an oil bath at 170 °C for 8 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (4x). The combined organic phases were washed with warm water (50-55 °C, 2x), dried ($MgSO_4$), concentrated, and dried under high-vacuum at 50 °C (oil bath) for 2 h to yield a solid-yellow oil. Upon addition of Et_2O, an off-white solid precipitated. The solid was rinsed with Et_2O (3x) and dried under high-vacuum at 40 °C to yield **13b** (36.9 mg, 0.0857 mmol, 67%) as an off-white solid: Mp 160-163 °C; IR (ATR, neat) 3269, 2960, 1606, 1556, 1439 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.40-7.26 (m, 8 H), 7.25-7.16 (m, 2 H), 6.64-6.29 (m, 2 H), 4.80 (d, 1 H, J = 4.8 Hz), 3.89 (t, 2 H, J = 6.9 Hz), 3.79-3.69 (bm, 1 H), 3.69-3.61 (bm, 1 H), 3.55-3.41 (bm, 1 H), 1.79 (sext, 2 H, J = 6.6 Hz), 1.61-1.37 (m, 2 H), 1.05-0.90 (m, 6 H); ^{13}C NMR (151 MHz, $CDCl_3$) δ 160.0, 153.9, 142.2, 137.2, 128.5 (2 C), 127.6, 127.2 (2 C), 114.3, 67.7, 57.9, 56.0, 45.0, 24.8, 23.1, 11.2, 10.8; HRMS (ES^+) *m/z* calcd for $C_{25}H_{31}N_6O$ $[M+H]^+$ 431.2559, found 431.2596.

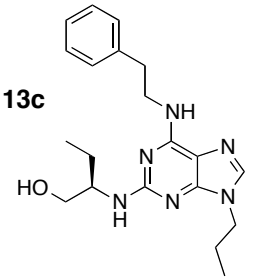


16c

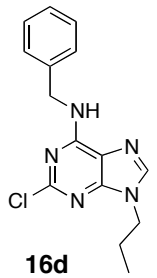
2-Chloro-*N*-phenethyl-9-propyl-9*H*-purin-6-amine (16c). To a solution of **15a** (58.0 mg, 0.251 mmol) in *n*-BuOH (1.0 mL) was added phenethylamine (32.8 mg, 0.269 mmol) and triethylamine (40.6 mg, 0.402 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 30 min. *n*-BuOH was evaporated and the residue was dissolved in EtOAc and washed with water. The aqueous phase

was further extracted with EtOAc and the combined organic layers were dried ($MgSO_4$) and concentrated to yield a colorless solid, which was resuspended (hexanes/Et_2O, 3:1, filtered, triturated (hexanes/Et_2O, 3:1) and dried under high-vacuum to yield **16c** (67.0 mg, 0.212 mmol, 85%) as an amorphous off-white solid: IR (ATR, neat) 3218, 2960, 1620, 1576, 1355, 1307, 1232 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.47-7.30 (m, 1 H), 7.25-7.19 (m, 2 H), 7.19-7.09 (m, 3 H), 6.91-6.63 (m, 1 H), 4.15-3.98 (bm, 2 H), 3.95-3.67 (bm, 2 H), 3.00-2.87 (bm, 2 H), 1.98-1.78 (bm, 2 H), 0.92 (bt, 3 H, J = 6.3 Hz); ^{13}C NMR (151 MHz, $CDCl_3$) δ 155.1, 154.4, 149.9, 139.8, 138.8, 128.7, 128.4, 126.2, 118.5, 45.3, 41.8, 35.4, 23.2, 11.0.

13c

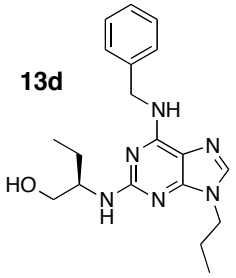


(***R***)**-2-(6-(Phenethylamino)-9-propyl-9*H*-purin-2-ylamino)butan-1-ol (13c**). A mixture of **16c** (50.0 mg, 0.158 mmol) and (*R*)-(-)-2-amino-1-butanol (76.0 mg, 0.801 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 7 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (3x). The combined organic phases were washed with warm water (50-55 °C, 2x), dried ($MgSO_4$), concentrated, and dried under high-vacuum at 50 °C (oil bath) for 2 h to yield an oily yellow residue. Upon the addition of Et_2O and a few drops of hexanes, an off-colorless solid precipitated. The solid was rinsed (Et_2O, 3x) by pipetting out the supernatant, and the solid was dried under high-vacuum at 40 °C overnight to yield **13c** (18.7 mg, 0.0508 mmol, 32%) as an off-white solid: Mp 105-107 °C; IR (ATR, neat) 3276, 2956, 2929, 1603, 1520 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.43 (bs, 1 H), 7.40-7.32 (m, 2 H), 7.32-7.22 (m, 3 H), 5.80 (bs, 1 H), 5.40 (bs, 1 H), 5.04-4.84 (bm, 1 H), 3.99 (t, 2 H, J = 6.6 Hz), 3.97-3.91 (bm, 1 H), 3.91-3.75 (bm, 3 H), 3.69 (t, 1 H, J = 9.0 Hz), 2.99 (t, 2 H, J = 6.3 Hz), 1.89 (sext, 2 H, J = 6.6 Hz), 1.75-1.55 (m, 2 H), 1.08 (t, 3 H, J = 7.2 Hz), 0.98 (t, 3 H, J = 7.2 Hz); ^{13}C NMR (151 MHz, $CDCl_3$) δ 160.2, 154.9, 139.0, 137.0, 128.8, 128.6, 126.4, 114.4, 68.6, 56.4, 45.0, 41.7, 35.9, 25.0, 23.2, 11.2, 10.9; HRMS (ES^+) *m/z* calcd for $C_{20}H_{29}N_6O$ $[M+H]^+$ 369.2403, found 369.2422.



16d

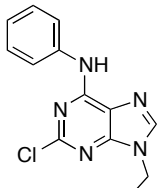
***N*-Benzyl-2-chloro-9-propyl-9*H*-purin-6-amine (16d).** To a solution of **15a** (63.0 mg, 0.273 mmol) in *n*-BuOH (1.0 mL) was added benzylamine (31.4 mg, 0.287 mmol) and triethylamine (43.6 mg, 0.430 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 30 min. *n*-BuOH was evaporated, and the residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc and the combined organic extracts were dried ($MgSO_4$) and concentrated to yield a colorless solid, which was resuspended (hexanes/Et_2O, 3:1), filtered, triturated (hexanes/Et_2O, 3:1), and dried under high-vacuum to yield **16d** (66.3 mg, 0.220 mmol, 81%) as a colorless solid: IR (ATR, neat) 3189, 2966, 1623, 1304, 1253 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.44-7.27 (m, 6 H), 7.10-6.75 (bm, 1 H), 4.82 (bs, 2 H), 4.20-4.00 (bm, 2 H), 1.95-1.80 (bm, 2 H), 0.93 (t, 3 H, J = 6.6 Hz); ^{13}C NMR (151 MHz, $CDCl_3$) δ 155.1, 154.5, 150.2, 140.2, 138.0, 128.6, 127.9, 127.5, 118.6, 45.4, 44.6, 23.3, 11.1.



13d

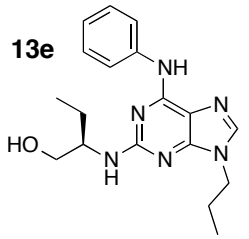
(*R*)-2-(6-(Benzylamino)-9-propyl-9*H*-purin-2-ylamino)butan-1-ol (13d). A mixture of **16d** (50.0 mg, 0.166 mmol) and (*R*)-(-)-2-amino-1-butanol (78.9 mg, 0.831 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 7 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (3x). The combined organic phases were washed with warm water (50-55 °C, 2x) dried ($MgSO_4$), concentrated, and dried under high-vacuum at 50 °C (oil bath) for 2 h to yield a

yellow solid. Upon the addition of Et_2O to the yellow solid, an off-white solid precipitated. The solid was rinsed (Et_2O, 3x) by pipetting out the supernatant and dried under high-vacuum at 40 °C overnight to yield **13d** (38.6 mg, 0.109 mmol, 66%) as an off-white solid: Mp 153-155 °C; IR (ATR, neat) 3262, 3201, 2961, 1624, 1603, 1513 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.23-7.40 (m, 6 H), 6.22 (bs, 1 H), 5.19 (bs, 1 H), 4.88-4.96 (m, 1 H), 4.75 (bs, 2 H), 3.92-3.97 (m, 2 H), 3.85-3.92 (m, 1 H), 3.81 (d, 1 H, *J* = 10.8 Hz), 3.62 (t, 1 H, *J* = 8.7 Hz), 1.90-1.80 (m, 2 H), 1.70-1.50 (m, 2 H), 1.02 (t, 3 H, *J* = 7.2 Hz), 0.94 (t, 3 H, *J* = 6.6 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.2, 154.8, 150.6, 138.7, 137.1, 128.5, 127.7, 127.3, 114.3, 68.4, 56.3, 45.0, 44.4, 25.0, 23.2, 11.2, 10.9; HRMS (ES) *m/z* calcd for $C_{19}H_{26}N_6O$ [M+H] 355.2246, found 355.2241.



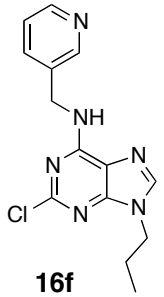
16e

2-Chloro-*N*-phenyl-9-propyl-9*H*-purin-6-amine (16e). To a solution of **15a** (72.0 mg, 0.312 mmol) in *n*-BuOH (1.0 mL) were added aniline (30.2 mg, 0.324 mmol) and triethylamine (50.1 mg, 0.495 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 30 min. *n*-BuOH was evaporated, and the residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined organic extracts were dried ($MgSO_4$) and concentrated to yield a colorless solid. The solid was resuspended (hexanes/Et_2O, 3:1), filtered, triturated (hexanes/Et_2O, 3:1), and dried under high-vacuum to yield **16e** (52.7 mg, 0.183 mmol, 59%) as an off-white amorphous solid: IR (ATR, neat) 3179, 2967, 1611, 1572, 1346, 1301 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 8.31 (bs, 1 H), 7.67-7.81 (m, 3 H), 7.36 (t, 2 H, *J* = 7.8 Hz), 7.12 (t, 1 H, *J* = 7.2 Hz), 4.11 (t, 2 H, *J* = 7.2 Hz), 1.89 (sext, 2 H, *J* = 7.2 Hz), 0.94 (t, 3 H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 153.9, 152.3, 150.7, 141.0, 138.0, 128.9, 123.9, 120.3, 119.1, 45.5, 23.2, 11.0.



13e

(*R*)-2-(6-(Phenylamino)-9-propyl-9*H*-purin-2-ylamino)butan-1-ol (13e). A mixture of **16e** (41.0 mg, 0.142 mmol) and (*R*)-(-)-2-amino-1-butanol (68.4 mg, 0.721 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 12 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (3x). The combined organic phases were washed with warm water (50-55 °C, 2x), dried ($MgSO_4$), concentrated, and dried under high-vacuum to yield a light-green solid. The crude solid was adsorbed onto SiO_2 and purified by chromatography on SiO_2 (hexanes/EtOAc, 1:1 to EtOAc, 100% with 1% Et_3N to 10% MeOH in EtOAc with 1% Et_3N) to yield **13e** (23.6 mg, 0.0693 mmol, 49%) as an off-white solid: Mp 190-194 °C; IR (ATR, neat) 3338, 2970, 1644, 1583, 1498, 1474, 1442 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.75 (d, 2 H, J = 7.8 Hz), 7.65 (bs, 1 H), 7.51 (s, 1 H), 7.35 (t, 2 H, J = 7.8 Hz), 7.08 (t, 1 H, J = 7.8 Hz), 5.03 (d, 1 H, J = 6.6 Hz), 3.93-4.02 (m, 3 H), 3.87 (dd, 1 H, J = 10.8, 1.8 Hz), 3.68 (dd, 1 H, J = 10.8, 7.2 Hz), 1.87 (sext, 2 H, J = 7.2 Hz), 1.57-1.73 (m, 2 H), 1.05 (t, 3 H, J = 7.2 Hz), 0.96 (t, 3 H, J = 7.2 Hz); ^{13}C NMR δ ($CDCl_3$, 150 MHz) δ 159.9, 152.4, 150.9, 139.0, 137.6, 128.9, 123.0, 120.0, 114.8, 56.1, 45.1, 29.7, 25.0, 23.2, 11.2, 10.9; HRMS (EI) *m/z* calcd for $C_{18}H_{24}N_6O$ 340.2012 found 340.2009.

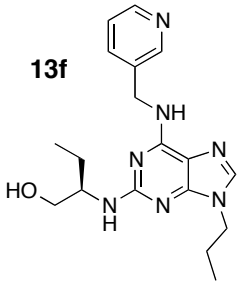


16f

2-Chloro-9-propyl-*N*-(pyridin-3-ylmethyl)-9*H*-purin-6-amine (16f). To a solution of **15a** (58.0 mg, 0.251 mmol) in *n*-BuOH (1.0 mL) were added 3-(aminomethyl)pyridine (28.6 mg, 0.265 mmol) and triethylamine (40.6 mg, 0.402 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 30 min. *n*-BuOH was evaporated, and the residue was dissolved in EtOAc and washed

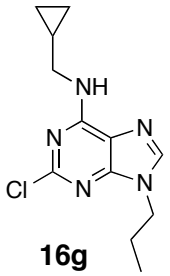
with water. The aqueous phase was further extracted with EtOAc, and the combined organic extracts were dried ($MgSO_4$) and concentrated to yield a colorless solid. The solid was resuspended (hexanes/Et_2O, 3:1), filtered, triturated (hexanes/Et_2O, 3:1), and dried under high-vacuum to yield **16f** (53.9 mg, 0.178 mmol, 71%) as a yellow amorphous solid: IR (ATR, neat) 3155, 2964, 1626, 1572, 1308, 1232 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 8.57 (s, 1 H), 8.52-8.44 (m, 1 H), 7.64 (d, 1 H, J = 7.8 Hz), 7.50 (bs, 1 H), 7.33 (s, 1 H), 7.18 (dd, 1 H, J = 7.2, 4.8 Hz), 4.79 (bs, 2 H), 4.05 (t, 2 H, J = 7.2 Hz), 1.83 (sext, 2 H, J = 7.2 Hz), 0.89 (t, 3 H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 154.9, 154.3, 151.0, 149.2, 148.8, 140.2, 135.5, 133.7, 123.4, 118.5, 45.4, 41.8, 23.2, 11.0.

13f



(*R*)-2-(9-Propyl-6-(pyridin-3-ylmethylamino)-9*H*-purin-2-ylamino)butan-1-ol (13f). A mixture of **16f** (35.0 mg, 0.116 mmol) and (*R*)-(-)-2-amino-1-butanol (55.1 mg, 0.581 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 12 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (3x). The combined organic phases were washed with warm water (50-55 °C, 2x), dried ($MgSO_4$), concentrated, and dried under high-vacuum to yield an oily yellow residue. Upon the addition of Et_2O and hexanes (drops), a sticky, yellow solid precipitated. The solid was carefully crushed with a glass rod to yield an off-white solid, which was rinsed with Et_2O (3x) by pipetting out the supernatant and dried under high-vacuum overnight to yield **13f** (27.0 mg, 0.0760 mmol, 66%) as an off-white solid: Mp 129-132 °C; IR (ATR, neat) 3257, 3209, 2964, 1601, 1530, 1477 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 8.68 (s, 1 H), 8.54 (d, 1 H, J = 4.8 Hz), 7.73 (d, 1 H, J = 7.8 Hz), 7.46 (s, 1 H), 7.30-7.28 (m, 1 H), 5.99 (bs, 1 H), 4.89-4.86 (m, 1 H), 4.81 (bs, 2 H), 4.00 (t, 2 H, J = 7.2 Hz), 3.92-3.86 (m, 1 H), 3.81 (d, 1 H, J = 10.2 Hz), 3.63 (dd, 1 H, J = 10.2, 7.8 Hz), 1.89 (sext, 2 H, J = 7.2 Hz), 1.73-1.63 (m, 2 H), 1.63-1.53 (m, 1 H), 1.04 (t, 3 H, J = 7.2 Hz), 0.97 (t, 3 H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.1, 154.7, 150.9, 149.3,

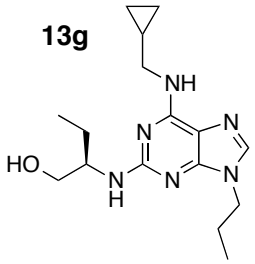
148.6, 137.3, 135.3, 134.5, 123.4, 114.3, 67.9, 56.1, 45.1, 41.8, 24.9, 23.1, 11.2, 10.9; HRMS (EI) *m/z* calcd for $C_{18}H_{25}N_7O$ 355.2121, found 355.2124.



16g

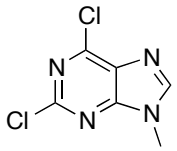
2-Chloro-*N*-(cyclopropylmethyl)-9-propyl-9*H*-purin-6-amine (16g). To a solution of **15a** (80.0 mg, 0.346 mmol) in *n*-BuOH (1 mL) were added aminomethylcyclopropane (28.7 mg, 0.391 mmol) and triethylamine (56.6 mg, 0.560 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 30 min. *n*-BuOH was evaporated, and the residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined organic extracts were dried ($MgSO_4$), filtered, concentrated, and dried under high-vacuum overnight to yield **16g** (87.1 mg, 0.328 mmol, 95%) as an amorphous colorless solid: IR (ATR, neat) 3260, 2970, 1621, 1578, 1302, 1253 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.67 (s, 1 H), 6.26 (bs, 1 H), 4.06 (t, 2 H, J = 7.2 Hz), 3.45-3.35 (m, 2 H), 1.84 (sext, 2 H, J = 7.2 Hz), 1.10-1.00 (m, 1 H), 0.89 (t, 2 H, J = 7.2 Hz), 0.48 (dd, 2 H, J = 13.8, 4.8 Hz), 0.23 (dd, 2 H, J = 9.6, 4.8 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 155.0, 154.3, 149.9, 139.8, 118.4, 60.2, 45.6, 45.3, 23.2, 10.9, 10.5, 3.4.

13g



(*R*)-2-(6-(Cyclopropylmethylamino)-9-propyl-9*H*-purin-2-ylamino)butan-1-ol (13g). A mixture of **15a** (50.0 mg, 0.188 mmol) and (*R*)-(-)-2-amino-1-butanol (89.2 mg, 0.941 mmol) was heated in a pressure tube immersed in an oil bath at 170 °C for 10 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (3x). The combined organic phases were washed with warm water (50-55 °C,

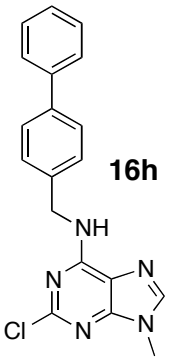
2x), dried ($MgSO_4$), concentrated, and dried under high-vacuum to yield a yellow solid. Upon the addition of Et_2O to the solid, an off-white solid was precipitated. The solid was rinsed (Et_2O, 3x) by pipetting out the supernatant and dried under high-vacuum to yield **13g** (37.4 mg, 0.117 mmol, 62%) as an off-white solid: Mp 146-149 °C; IR (ATR, neat) 3319, 2966, 2847, 1611, 1489 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.42 (s, 1 H), 5.81 (bs, 1 H), 5.51 (bs, 1 H), 4.96-4.88 (m, 1 H), 3.94 (t, 2 H, *J* = 7.2 Hz), 3.91-3.85 (m, 1 H), 3.82 (d, 1 H, *J* = 10.2 Hz), 3.63 (dd, 1 H, *J* = 10.2, 8.4 Hz), 3.38 (bs, 2 H), 1.84 (sext, 2 H, *J* = 7.2 Hz), 1.68-1.49 (m, 2 H), 1.15-1.05 (m, 1 H), 1.01 (t, 3 H, *J* = 7.2 Hz), 0.92 (t, 3 H, *J* = 7.2 Hz), 0.55-0.50 (m, 2 H), 0.28-0.23 (m, 2 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.2, 154.8, 150.5, 136.9, 114.2, 68.4, 56.3, 45.4, 45.0, 25.0, 23.1, 11.1, 10.9, 10.8, 3.4; HRMS (EI) *m/z* calcd for $C_{16}H_{26}N_6O$ 318.2168, found 318.2168.



15b

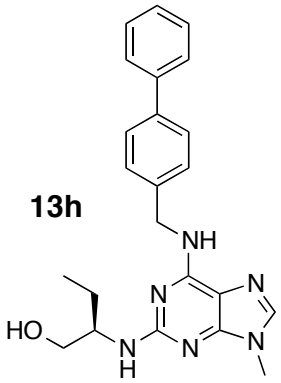
2,6-Dichloro-9-methyl-9*H*-purine (15b).[3] To a solution of 2,6-dichloro-9*H*-purine (0.120 g, 0.635 mmol) in anhydrous DMF (1.0 mL) was added K_2CO_3 (0.270 g, 1.95 mmol) followed by iodomethane (0.20 mL, 3.21 mmol) at 0 °C. The reaction mixture was stirred for 5 h at 0 °C, quenched with water and extracted with EtOAc. The organic layers were combined, washed with brine, dried ($MgSO_4$), concentrated, and purified by chromatography on SiO_2 (hexanes, 100%, to EtOAc, 100%) to yield **15b** (83.0 mg, 0.409 mmol, 64% yield) as a colorless solid: IR (ATR, neat) 3067, 1554, 1360, 1333, 1223, 1147 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 8.09 (s, 1 H), 3.89 (s, 3 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 153.3, 152.7, 151.3, 146.4, 130.4, 30.4.

[3] Beaman, A. G.; Robins, R. K., "Direct conversion of chloropurines to fluoropurines." *J. Org. Chem.* **1963**, *28*, 2310-2313.



16h

***N*-(Biphenyl-4-ylmethyl)-2-chloro-9-methyl-9*H*-purin-6-amine (16h).**[2] To a solution of **15b** (60.0 mg, 0.296 mmol) in *n*-BuOH (1.0 mL) were added 4-phenylbenzylamine (58.6 mg, 0.310 mmol) and triethylamine (48.6 mg, 0.476 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 30 min. *n*-BuOH was evaporated, and the residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined organic layers were dried ($MgSO_4$) and concentrated to yield a colorless solid. The solid was resuspended (hexanes/Et_2O, 2:1), filtered, triturated (hexanes/Et_2O, 2:1), and dried under high-vacuum to yield **16h** (89.0 mg, 0.254 mmol, 86%) as an off-white solid: IR (ATR, neat) 3214, 2387, 1620, 1602, 1482, 1308, 1233 cm^{-1}; ^1H NMR ($CDCl_3/CD_3OD$, 9/1, 600 MHz) δ 7.64 (bs, 1 H), 7.49 (d, 4 H, J = 7.8 Hz), 7.38 (d, 2 H, J = 7.8 Hz), 7.34 (t, 2 H, J = 7.8 Hz), 7.25 (t, 1 H, J = 7.8 Hz), 4.74 (bs, 2 H), 3.77 (s, 3 H); ^{13}C NMR ($CDCl_3/CD_3OD$, 9/1, 150 MHz) δ 154.7, 154.5, 150.1, 140.5, 140.4, 140.3, 136.7, 128.6, 128.2, 127.1, 126.8, 117.8, 44.1, 29.8.

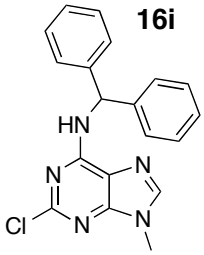


13h

(*R*)-2-(6-(Biphenyl-4-ylmethylamino)-9-methyl-9*H*-purin-2-ylamino)butan-1-ol (13h). A mixture of **16h** (50.0 mg, 0.143 mmol) and (*R*)-(-)-2-amino-1-butanol (68.4 mg, 0.721 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 15 h. The

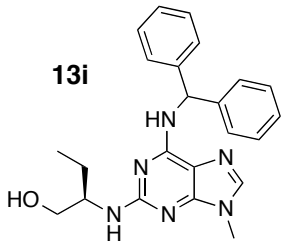
reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (3x). The combined organic phases were washed with warm water (50-55 °C, 2x) dried ($MgSO_4$), concentrated, and dried under high-vacuum to yield an oily, yellow-green residue. Addition of Et_2O and hexanes resulted in the precipitation of a light green solid. The solid was rinsed (Et_2O, 3x) by pipetting out the supernatant and dried under high-vacuum at 40 °C to yield impure product (90% purity). The solid and filtrate were combined, preadsorbed onto SiO_2 and purified by chromatography on SiO_2 (hexanes/EtOAc, 1:1, to EtOAc/Et_3N, 99:1, to EtOAc/MeOH/Et_3N, 98:10:1) to yield **13h** (35.7 mg, 0.0887 mmol, 62%) as a light green solid: Mp 133-136 °C; IR (ATR, neat) 3290, 2927, 1600, 1487 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.59 (d, 2 H, *J* = 7.2 Hz), 7.56 (d, 2 H, *J* = 7.8 Hz), 7.47-7.43 (m, 4 H), 7.41 (s, 1 H), 7.35 (t, 1 H, *J* = 7.2 Hz), 6.00 (bs, 1 H), 4.97-4.87 (m, 1 H), 4.83 (bs, 2 H), 4.00- 3.90 (m, 1 H), 3.83 (dd, 1 H, *J* = 10.8, 2.4 Hz), 3.66 (s, 3 H), 3.64 (dd, 1 H, *J* = 10.8, 7.8 Hz), 1.68-1.52 (m, 2 H), 1.03 (t, 3 H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.3, 154.8, 151.0, 140.8, 140.2, 137.8, 137.6, 128.7, 128.1, 127.3, 127.0, 114.2, 68.2, 56.2, 44.1, 29.4, 25.0, 10.9; HRMS (EI) *m/z* calcd for $C_{23}H_{26}N_6O$ 402.2168, found 402.2178.

16i

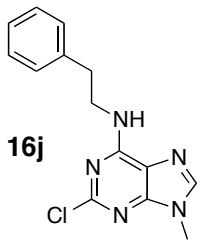


2-Chloro-*N*-(2,2-diphenylethyl)-9-methyl-9*H*-purin-6-amine (16i). To a solution of **15b** (68.0 mg, 0.335 mmol) in *n*-BuOH (1.0 mL) was added aminodiphenylmethane (66.2 mg, 0.350 mmol) and triethylamine (55.1 mg, 0.539 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 30 min. *n*-BuOH was evaporated, and the residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined organic extracts were dried ($MgSO_4$) and concentrated to yield a colorless solid. The solid was resuspended (hexanes/Et_2O, 2:1), filtered, triturated (hexanes/Et_2O, 2:1), and dried under high-vacuum to yield **16i** (83.0 mg, 0.237 mmol, 71%) as an off-white, crude solid that was used without further purification: Characteristic signals: ^1H NMR ($CDCl_3$, 600

MHz) δ 7.40-7.20 (m, 12 H), 6.75 (bs, 1 H), 3.69 (s, 3 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 154.5, 154.2, 150.7, 141.3, 140.9, 128.5, 127.6, 127.4, 118.4, 57.3, 29.9.

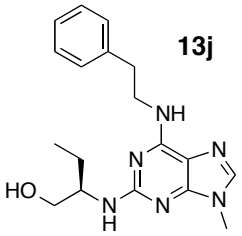


(*R*)-2-(6-(2,2-Diphenylethylamino)-9-methyl-9*H*-purin-2-ylamino)butan-1-ol (13i). A mixture of **16i** (50.0 mg, 0.143 mmol) and (*R*)-(-)-2-amino-1-butanol (68.4 mg, 0.721 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 15 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (3x). The combined organic phases were washed with warm water (50-55 °C, 2x) dried ($MgSO_4$), concentrated, and dried under high-vacuum at 50 °C (oil bath) for 2 h to yield an oily green residue. The crude residue was adsorbed onto SiO_2 and purified by chromatography on SiO_2 (hexanes/EtOAc, 1:1, to EtOAc/Et_2N, 99:1, to EtOAc/MeOH/Et_3N, 94:5:1) to yield **13i** (36.5 mg, 0.0907 mmol, 63%) as an off-white solid: Mp 130-134 °C; IR (ATR, neat) 3301 (br), 2932, 1597, 1474 cm^{-1}; ^{1}H NMR ($CDCl_3$, 600 MHz) δ 7.40-7.20 (m, 10 H), 6.60-6.40 (m, 2 H), 4.83 (d, 1 H, *J* = 6.0 Hz), 3.80-3.75 (m, 1 H), 3.70-3.65 (m, 1 H), 3.59 (s, 3 H), 3.60-3.50 (m, 1 H), 1.60-1.40 (m, 2 H), 0.94 (t, 3 H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.1, 153.9, 151.2, 142.1, 137.6, 128.51, 128.48, 127.57, 127.55, 127.3, 127.2, 114.1, 67.5, 58.0, 55.8, 29.3, 24.8, 10.8; HRMS (EI) *m/z* calcd for $C_{23}H_{26}N_6O$ 402.2168, found 402.2170.

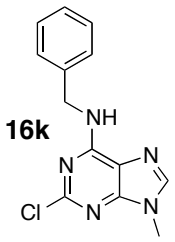


2-Chloro-9-methyl-*N*-phenethyl-9*H*-purin-6-amine (16j). To a solution of **15b** (60.0 mg, 0.296 mmol) in *n*-BuOH (1.0 mL) was added phenethylamine (38.0 mg, 0.312 mmol) and triethylamine (47.9 mg, 0.474 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 30 min. *n*-BuOH was evaporated,

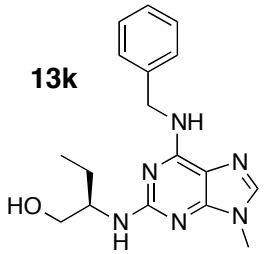
and the residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined organic extracts were dried ($MgSO_4$) and concentrated to yield a colorless solid. The solid was resuspended (hexanes/Et_2O, 3:1), filtered, triturated (hexanes/Et_2O, 3:1), and dried under high-vacuum to yield **16j** (71.0 mg, 0.247 mmol, 83%) as an off-white amorphous solid: IR (ATR, neat) 3233, 1615, 1578, 1299, 1231 cm^{-1}; ^1H NMR ($CDCl_3$ 600 MHz) δ 7.49 (bs, 1 H), 7.27 (d, 2 H, J = 7.2 Hz), 7.24-7.18 (m, 3 H), 6.41 (bs, 1 H), 3.93-3.83 (m, 2 H), 3.77 (s, 3 H), 2.97 (t, 2 H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 155.1, 154.6, 150.3, 140.5, 138.7, 128.8, 128.5, 126.4, 118.5, 41.9, 35.5, 29.9.



(*R*)-2-(9-Methyl-6-(phenethylamino)-9*H*-purin-2-ylamino)butan-1-ol (13j). A mixture of **16j** (50.0 mg, 0.174 mmol) and (*R*)-(-)-2-amino-1-butanol (83.6 mg, 0.882 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 15 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (3x). The combined organic phases were washed with warm water (50-55 °C, 2x), dried ($MgSO_4$), concentrated, and dried under high-vacuum to yield an oily yellow residue. Addition of Et_2O and a few drops of hexanes to the solid resulted in the precipitation of an off-white solid. The solid was rinsed (Et_2O, 3x) by pipetting out the supernatant and dried under high-vacuum at 40 °C. The solid and the filtrate were combined, adsorbed onto SiO_2, and purified by chromatography on SiO_2 (hexanes/EtOAc, 1:1, to EtOAc/Et_3N, 99:1, to EtOAc/MeOH/Et_3N, 94:5:1) to yield **13j** (37.8 mg, 0.111 mmol, 64%) as a light green solid: Mp 108-110 °C; IR (ATR, neat) 3922 (br), 2931, 1598, 1488 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.39 (s, 1 H), 7.32-7.24 (m, 4 H), 7.22 (t, 1 H, J = 7.2 Hz), 5.65 (bs, 1 H), 5.13 (bs, 1 H), 4.93-4.85 (m, 1 H), 3.97-3.90 (m, 1 H), 3.90-3.75 (m, 3 H), 3.67-3.63 (m, 1 H), 3.63 (s, 3 H), 2.96 (t, 2 H, J = 7.2 Hz), 1.70-1.55 (m, 2 H), 1.04 (t, 3 H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.3, 154.9, 150.9, 139.0, 137.5, 128.8, 128.6, 126.4, 114.2, 68.3, 56.2, 41.7, 35.9, 29.3, 25.0, 10.9; HRMS (EI) *m/z* calcd for $C_{18}H_{24}N_6O$ 340.2012, found 340.2014.



***N*-Benzyl-2-chloro-9-methyl-9*H*-purin-6-amine (16k).**[4] To a solution of **15b** (71.0 mg, 0.350 mmol) in *n*-BuOH (1.0 mL) were added benzylamine (40.50 mg, 0.370 mmol) and triethylamine (56.6 mmol, 0.560 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 30 min. *n*-BuOH was evaporated, and the residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined organic extracts were dried ($MgSO_4$) and concentrated to yield a colorless solid. The solid was resuspended (hexanes/Et_2O, 3:1), filtered, triturated (hexanes/Et_2O, 3:1), and dried under high-vacuum to yield **16k** (65.5 mg, 0.239 mmol, 68%) as a colorless solid: IR (ATR, neat) 2385, 1596, 1572, 1325, 1232 cm^{-1}; ^1H NMR ($CDCl_3/CD_3OD$, 9/1, 600 MHz) δ 7.72 (bs, 1 H), 7.40 (d, 2 H, J = 7.2 Hz), 7.35 (t, 2 H, J = 7.2 Hz), 7.29 (t, 1 H, J = 7.2 Hz), 4.79 (bs, 2 H), 3.79 (s, 3 H); ^{13}C NMR ($CDCl_3/CD_3OD$, 9/1, 150 MHz) δ 154.7, 154.5, 150.1, 140.5, 137.6, 128.4, 127.7, 127.4, 117.8, 44.4, 29.8.

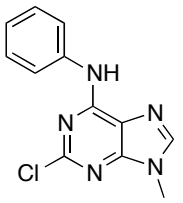


(*R*)-2-(6-(Benzylamino)-9-methyl-9*H*-purin-2-ylamino)butan-1-ol (13k).[5] A mixture of **16k** (49.0 mg, 0.179 mmol) and (*R*)-(-)-2-amino-1-butanol (85.5 mg, 0.902 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 11 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc

[4] Havlicek, L.; Hanus, J.; Vesely, J.; Leclerc, S.; Meijer, L.; Shaw, G.; Strnad, M., "Cytokinin-derived cyclin-dependent kinase inhibitors: Synthesis and cdc2 inhibitory activity of olomoucine and related compounds." *J. Med. Chem.* **1997**, *40*, 408-412.

[5] Otyepka, M.; Krystof, V.; Havlicek, L.; Siglerova, V.; Strnad, M.; Koca, J., "Docking-based development of purine-like inhibitors of cyclin-dependent kinase-2." *J. Med. Chem.* **2000**, *43*, 2506-2513.

(3x). The combined organic phases were washed with warm water (50-55 °C, 2x), dried ($MgSO_4$), concentrated, and dried under high-vacuum at 50 °C (oil bath) for 2 h to yield a yellow solid. The addition of Et_2O to the solid resulted in the precipitation of an off-white solid. The solid was rinsed (Et_2O, 3x) by pipetting out the supernatant and dried under high-vacuum overnight at 40 °C to yield an off-white solid. The crude mixture was purified by chromatography on SiO_2 (hexanes/EtOAc, 1:1, to EtOAc/MeOH/Et_3N, 94:5:1, to EtOAc/MeOH/Et_3N, 85:14:1) to yield **13k** (35.5 mg, 0.109 mmol, 61%) as a light yellow solid: Mp 118-120 °C; IR (ATR, neat) 3261 (br), 2958, 1610, 1493 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.40-7.25 (m, 6 H), 6.20 (bs, 1 H), 5.00-4.92 (m, 1 H), 4.74 (bs, 2 H), 3.95-3.90 (m, 1 H), 3.81 (dd, 1 H, *J* = 10.8, 2.4 Hz), 3.62 (s, 3 H), 3.67-3.58 (m, 1 H), 1.67-1.52 (m, 2 H), 1.01 (t, 3 H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.3, 154.8, 151.0, 138.7, 137.5, 128.5, 127.6, 127.2, 114.1, 67.9, 56.0, 44.3, 29.3, 24.9, 10.9; HRMS (EI) *m/z* calcd for $C_{17}H_{22}N_6O$ 326.1855, found 326.1843.

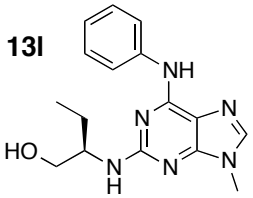


16l

2-Chloro-9-methyl-*N*-phenyl-9H-purin-6-amine (16l).[6] To a solution of **15b** (72.0 mg, 0.355 mmol) in *n*-BuOH (1.0 mL) were added aniline (34.0 mg, 0.365 mmol) and triethylamine (57.4 mg, 0.567 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120° C for 30 min. *n*-BuOH was evaporated, and the residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined organic extracts were dried ($MgSO_4$) and concentrated to yield a colorless solid. The solid was resuspended (hexanes/Et_2O, 3:1), filtered, triturated (hexanes/Et_2O, 3:1), and dried under high-vacuum to yield **16l** (59.7 mg, 0.230 mmol, 65%) as a colorless amorphous solid: IR (ATR, neat) 3286, 1620, 1574, 1437, 1308, 1236 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.85 (bs, 1 H), 7.80-7.70 (m, 3 H), 7.45-7.35 (m, 2 H), 7.20-7.10 (m, 1 H), 3.84 (s, 3 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 154.2, 152.3, 151.0, 141.4, 137.9, 129.1, 124.0, 120.2, 119.1, 30.1.

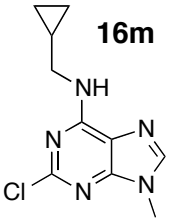
[6] Thompson, R. D.; Secunda, S.; Daly, J. W.; Olsson, R. A., "N^6,9-disubstituted adenines: Potent, selective antagonists at the A1 adenosine receptor." *J. Med. Chem.* **1991**, *34*, 2877-2882.

13l



(*R*)-2-(9-Methyl-6-(phenylamino)-9*H*-purin-2-ylamino)butan-1-ol (13l). A mixture of **16l** (46.0 mg, 0.177 mmol) and (*R*)-(-)-2-amino-1-butanol (85.5 mg, 0.902 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 11 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (3x). The combined organic phases were washed with warm water (50-55 °C, 2x) dried ($MgSO_4$), concentrated, and dried under high-vacuum to yield a light green solid. The solid was preadsorbed on SiO_2 and purified by chromatography on SiO_2 (hexanes/EtOAc, 1:1, to EtOAc/Et_3N, 99:1, to EtOAc/MeOH/Et_3N, 90:9:1) to yield **13l** (29.8 mg, 0.0954 mmol, 54%) as an off-white, slightly light green solid: Mp 202-206 °C; IR (ATR, neat) 3222, 3133 (br), 2930, 1579, 1498, 1442 cm^{-1}; ^1H NMR ($CDCl_3/CD_3OD$, 9/1, 600 MHz) δ 7.66 (dd, 2 H, J = 7.2, 1.2 Hz), 7.43 (s, 1 H), 7.25 (t, 2 H, J = 7.2 Hz), 6.97 (td, 1 H, J = 7.2, 1.2 Hz), 3.90 (s, 3 H), 3.92-3.85 (m, 1 H), 3.67 (dd, 1 H, J = 10.8, 3.6 Hz), 3.57-3.53 (m, 1 H), 1.65-1.50 (m, 2 H), 0.92 (t, 3 H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3/CD_3OD$, 9/1, 150 MHz) δ 159.6, 152.1, 151.1, 138.9, 137.79, 137.78, 128.6, 122.9, 119.9, 113.5, 65.2, 55.0, 29.1, 24.3, 10.4; HRMS (EI) *m/z* calcd for $C_{16}H_{20}N_6O$ 312.1699, found 312.1693.

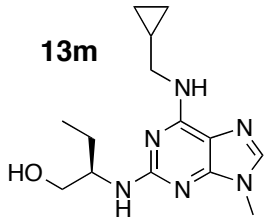
16m



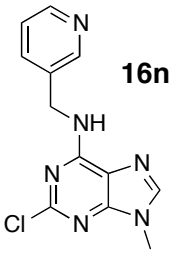
2-Chloro-*N*-(cyclopropylmethyl)-9-methyl-9*H*-purin-6-amine (16m). To a solution of **15b** (70.0 mg, 0.345 mmol) in *n*-BuOH (1.0 mL) were added aminomethylcyclopropane (30.0 mg, 0.4092 mmol) and triethylamine (55.9 mg, 0.552 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 30 min. *n*-BuOH was evaporated, and the residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined

organic extracts were dried ($MgSO_4$), concentrated, and dried under high-vacuum overnight to yield **16m** (77.9 mg, 0.328 mmol, 95% yield) as a colorless solid: IR (ATR, neat) 3260, 1618, 1581, 1301, 1234 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.68 (s, 1 H), 6.07 (bs, 1 H), 3.78 (s, 3 H), 3.50-3.40 (m, 2 H), 1.13-1.07 (m, 1 H), 0.55 (dd, 2 H; J = 12.6, 4.8 Hz), 0.30 (dd, 2 H, J = 9.6, 4.8 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 155.1, 154.6, 150.3, 140.5, 118.5, 45.8, 30.0, 10.5, 3.5.

13m

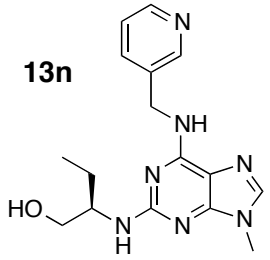


(*R*)-2-(6-(Cyclopropylmethylamino)-9-methyl-9*H*-purin-2-ylamino)butan-1-ol (13m). A mixture of **16m** (50.0 mg, 0.210 mmol) and (*R*)-(-)-2-amino-1-butanol (95.0 mg, 1.00 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 11 h. The reaction was cooled to room temperature, diluted with water, and extracted with EtOAc (3x). The combined organic phases were washed with warm water (50-55 °C, 2x), dried ($MgSO_4$), concentrated, and dried under high-vacuum to yield a green solid. Addition of Et_2O to the solid resulted in the precipitation of an off-white solid. The solid was rinsed (Et_2O, 3x) by pipetting out the supernatant and dried under high-vacuum to yield a light green solid. The crude residue was purified by chromatography on SiO_2 (hexanes/EtOAc, 1:1, to EtOAc/MeOH/Et_3N, 94:5:1, to EtOAc/MeOH/Et_3N, 84:15:1) to yield **13m** (35.8 mg, 0.123 mmol, 59%) as a crystalline green solid: Mp 147-150 °C; IR (ATR, neat) 3328, 3078, 2849, 1610, 1490 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.40 (s, 1 H), 5.83 (bs, 1 H), 5.37 (bs, 1 H), 5.00-4.87 (m, 1 H), 3.95-3.88 (m, 1 H), 3.82 (d, 1 H, J = 10.8 Hz), 3.67-3.58 (m, 1 H), 3.62 (s, 3 H), 3.39 (bs, 2 H), 1.68-1.50 (m, 2 H), 1.13-1.03 (m, 1 H), 1.02 (t, 3 H, J = 7.2 Hz), 0.58-0.48 (m, 2 H), 0.31-0.21 (m, 2 H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.3, 154.8, 150.8, 137.3, 114.0, 68.1, 56.1, 45.4, 29.3, 25.0, 10.9, 10.8, 3.4; HRMS (EI) *m/z* calcd for $C_{14}H_{22}N_6O$ 290.1855, found 290.1850.



16n

2-Chloro-9-methyl-*N*-(pyridin-3-ylmethyl)-9*H*-purin-6-amine (16n).[7] To a solution of **15b** (72.0 mg, 0.355 mmol) in *n*-BuOH (1.0 mL) was added 3-(aminomethyl)pyridine (39.7 mg, 0.367 mmol) and triethylamine (57.5 mg, 0.568 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 30 min. *n*-BuOH was evaporated, and the residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined organic extracts were dried ($MgSO_4$) and concentrated to yield a colorless solid. The solid was resuspended (hexanes/Et_2O, 1:1), filtered, triturated (hexanes/Et_2O, 1:1), and dried under high-vacuum to yield **16n** (76.0 mg, 0.277 mmol, 78%) contaminated with a small amount (~10%) of EtOAc and Et_2O as a fine yellow amorphous solid that was used for the next reaction without further purification: IR (ATR, neat) 3076, 2401, 1603, 1579, 1313, 1232 cm^{-1}; ^1H NMR ($CDCl_3/CD_3OD$, 9/1, 600 MHz) δ 8.50 (bs, 1 H), 8.35 (bs, 1 H), 7.71 (d, 1 H, J = 7.8 Hz), 7.65 (s, 1 H), 7.25-7.20 (m, 1 H), 4.70 (bs, 2 H), 3.68 (s, 3 H); ^{13}C NMR ($CDCl_3/CD_3OD$, 9/1, 150 MHz) δ 154.6, 154.3, 148.6, 147.9, 140.7, 136.2, 134.1, 123.6, 117.8, 41.6, 29.8.

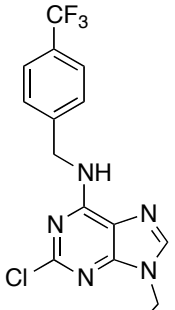


13n

(*R*)-2-(9-Methyl-6-(pyridin-3-ylmethylamino)-9*H*-purin-2-ylamino)butan-1-ol (13n). A mixture of **16n** (70.0 mg, 0.255 mmol) and (*R*)-(-)-2-amino-1-butanol (124 mg, 1.30 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 8.5 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with

[7] Schow, S. R.; Mackman, R. L.; Blum, C. L.; Brooks, E.; Horsma, A. G.; Joly, A.; Kerwar, S. S.; Lee, G.; Shiffman, D.; Nelson, M. G.; Wang, X.; Wick, M. M.; Zhang, X.; Lum, R. T., "Synthesis and activity of 2,6,9-trisubstituted purines." *Bioorg. Med. Chem. Lett.* **1997**, *7*, 2697-2702.

EtOAc (3x). The combined organic phases were washed with warmed water (50-55 °C, 2x) dried ($MgSO_4$), concentrated, and dried under high-vacuum to yield an oily, green solid. Addition of Et_2O and a few drops of hexanes to the solid resulted in the precipitation of a dark green solid. The solid was carefully crushed with a glass rod, rinsed (Et_2O, 3x) by pipetting out the supernatant, and dried under high-vacuum overnight to yield a light green solid. The crude residue was purified by chromatography on SiO_2 (hexanes/EtOAc, 1:1, to EtOAc/MeOH/Et_3N, 78:20:2) to yield **16n** (32.6 mg, 0.0936 mmol, 37%) as a green-gray solid: Mp 136-139 °C; IR (ATR, neat) 3256 (br), 2930, 1603, 1551, 1477 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 8.59 (d, 1 H, *J* = 1.8 Hz), 8.47 (dd, 1 H, *J* = 4.8, 1.8 Hz), 7.65 (d, 1 H, *J* = 7.8 Hz), 7.36 (s, 1 H), 7.20 (dd, 1 H, *J* = 7.8, 4.8 Hz), 6.58 (bs, 1 H), 5.08-5.00 (m, 1 H), 4.72 (bs, 2 H), 3.94-3.88 (m, 1 H), 3.76 (dd, 1 H, *J* = 10.8, 2,4 Hz), 3.61 (s, 3 H), 3.59 (dd, 1 H, *J* = 10.8, 7.2 Hz), 1.66-1.59 (m, 1 H), 1.55-1.49 (m, 1 H), 0.98 (t, 3 H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.0, 154.5, 151.1, 149.2, 148.5, 137.7, 135.3, 134.5, 123.4, 113.9, 67.3, 55.9. 41.8, 29.4, 24.9, 10.8; IR (ATR, neat) 3256 (br), 2930, 1603, 1551, 1477 cm^{-1}; HRMS (EI) *m/z* calcd for $C_{16}H_{21}N_7O$ 327.1808, found 327.1806.



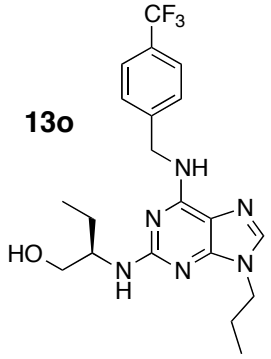
16o

2-Chloro-9-propyl-*N*-(4-(trifluoromethyl)benzyl)-9*H*-purin-6-amine (16o). To a solution of **15a**[8] (80.0 mg, 0.346 mmol) in *n*-BuOH (1.0 mL) were added 4-(trifluoromethyl)benzylamine (63.9 mg, 0.358 mmol) and triethylamine (55.9 mg, 0.552 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 30 min. *n*-BuOH was evaporated, and the residue was dissolved

[8] Dhainaut, A.; Regnier, G.; Tizot, A.; Pierre, A.; Leonce, S.; Guilbaud, N.; Kraus-Berthier, L.; Atassi, G., "New purines and purine analogs as modulators of multidrug resistance." *J. Med. Chem.* **1996**, *39*, 4099-4108.

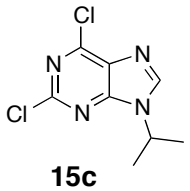
in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined organic extracts were dried ($MgSO_4$), and concentrated to yield a colorless solid. The solid was resuspended (hexanes/Et_2O, 3:1), filtered, triturated (hexanes/Et_2O, 3:1), and dried under high-vacuum to yield **16o** (78.0 mg, 0.211 mmol, 61%) as a colorless amorphous solid: IR (ATR, neat) 3261, 1630, 1580, 1325, 1308, 1253 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.58 (d, 2 H, J = 7.8 Hz), 7.52-7.45 (m, 3 H), 6.96 (bs, 1 H), 4.90 (bs, 2 H), 4.09 (t, 2 H, J = 7.2 Hz), 1.87 (sext, 2 H, J = 7.2 Hz), 0.94 (t, 3 H, J = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 155.0, 154.4, 150.4, 142.3, 140.3, 129.8 (q, J = 33 Hz), 128.0, 125.5 (q, J = 3 Hz), 124.0 (q, J = 270 Hz), 118.6, 45.5, 43.9, 23.3, 11.0.

13o



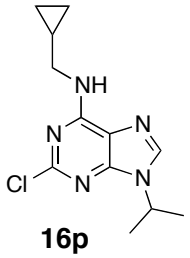
(*R*)-2-(9-Propyl-6-(4-(trifluoromethyl)benzylamino)-9*H*-purin-2-ylamino)butan-1-ol (13o). A mixture of **16o** (50.0 mg, 0.135 mmol) and (*R*)-(-)-2-amino-1-butanol (71.3 mg, 0.751 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 12 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (3x). The combined organic phases were washed with warm water (50-55 °C, 2x), dried ($MgSO_4$), concentrated, and dried under high-vacuum to yield a yellow solid. Addition of Et_2O/hexanes (1:1) to the solid resulted in the precipitation of a light green solid. The solid was rinsed (Et_2O/hexanes, 1:1) by pipetting out the supernatant and dried under high-vacuum overnight at 40 °C to yield **13o** (38.3 mg, 0.0907 mmol, 67%) as a light green crystalline solid: Mp 124-127 °C; IR (ATR, neat) 3266, 2962, 1600, 1545, 1326, 1104 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.56 (d, 2 H, J = 8.4 Hz), 7.46 (d, 2 H, J = 7.8 Hz), 7.38 (s, 1 H), 6.40 (bs, 1 H), 4.91 (d, 1 H, J = 6.0 Hz), 4.95-4.75 (m, 2 H), 3.95 (t, 2 H, J = 7.2 Hz), 3.89-3.82 (m, 1 H), 3.80 (dd, 1 H, J = 10.8, 1.8 Hz), 3.61 (dd, 1 H, J = 10.8, 7.8 Hz), 1.85 (sextet, 2 H, J = 7.2 Hz), 1.64-1.49 (m, 2 H), 0.99 (t, 3 H, J =

7.2 Hz), 0.94 (t, 3 H, *J* = 7.2 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.1, 154.7, 150.8, 143.2, 137.3, 129.4 (q, *J* = 33 Hz), 127.7, 125.4 (q, *J* = 3 Hz), 124.1 (q, *J* = 270 Hz), 114.3, 68.2, 56.2, 45.1, 43.8, 24.9, 23.2, 11.2, 10.9; HRMS (EI) *m/z* calcd for $C_{20}H_{25}F_3N_6O$ 422.2042, found 422.2038.



15c

2,6-Dichloro-9-isopropyl-9*H*-purine (15c).[9] To a solution of 2,6-dichloro-9*H*-purine (0.500 g, 2.65 mmol) in anhydrous DMSO (3.0 mL) cooled to 15 °C was added K_2CO_3 (1.10 g, 7.96 mmol) followed by 2-iodopropane (1.35 mL, 13.4 mmol). The mixture was stirred overnight at room temperature, quenched with water and extracted with EtOAc. The organic layers were combined, washed with brine, dried ($MgSO_4$), concentrated, and purified by chromatography on SiO_2 (hexanes, 100%, to hexanes/EtOAc, 1:1) to yield **15c** (0.415 g, 1.80 mmol, 68%) as a colorless solid: Mp 149-151 °C; IR (ATR, neat) 1587, 1554, 1356, 1214 cm^{-1}; ^{1}H NMR ($CDCl_3$, 600 MHz) δ 8.18 (s, 1 H), 4.92 (hept, 1 H, *J* = 6.6 Hz), 1.65 (d, 6 H, *J* = 6.6 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 152.7, 152.6, 151.6, 143.5, 131.0, 48.3, 22.5; HRMS (EI) *m/z* calcd for $C_8H_8Cl_2N_4$ 230.0126, found 230.0120.



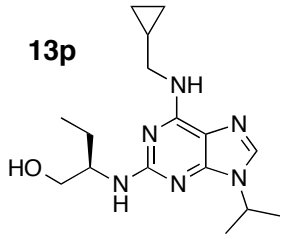
16p

2-Chloro-*N*-(cyclopropylmethyl)-9-isopropyl-9*H*-purin-6-amine (16p). To a solution of **15c** (100 mg, 0.433 mmol) in *n*-BuOH (1.5 mL) were added cyclopropylmethanamine (36.9 mg, 0.519 mmol) and Et_3N (70.9 mg, 0.692 mmol). The reaction was heated under microwave irradiation at 120 °C for 20 min. *n*-BuOH was evaporated *in vacuo*. The

[9] Oumata, N.; Bettayeb, K.; Ferandin, Y.; Demange, L.; Lopez-Giral, A.; Goddard, M.-L.; Myrianthopoulos, V.; Mikros, E.; Flajolet, M.; Greengard, P.; Meijer, L.; Galons, H., "Roscovitine-derived, dual-specificity inhibitors of cyclin-dependent kinases and casein kinases 1." *J. Med. Chem.* **2008**, *51*, 5229-5242.

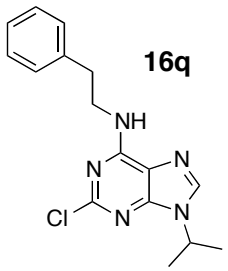
residue was diluted with water (5.0 mL), and the mixture was extracted with EtOAc (3 x 7.0 mL). The combined organic extracts were dried ($MgSO_4$), filtered, and concentrated to yield a pale yellow solid. The residue was resuspended (hexanes/Et_2O, 2:1), filtered, and washed (hexanes/Et_2O, 3:1). The solid was filtered and dried under high-vacuum to yield **16p (**60.3 mg, 0.227 mmol, 52%) as a pale yellow solid: Mp 70.2-72.7 °C; IR (ATR) 3286, 3086, 3068, 3055, 3038, 3030, 1647, 1627, 1592, 1575, 1560, 1446, 1314, 1273, 1204, 1174, 1159, 1150, 1075, 1027, 997, 943, 936, 917, 865, 813, 764, 719, 701, 691 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 7.80 (s, 1 H), 6.03 (bs, 1 H), 4.88-4.79 (hept, 1 H, *J* = 6.9 Hz), 3.49 (bs, 2 H), 1.58 (d, 6 H, *J* = 6.9 Hz), 1.20-1.10 (m, 1 H), 0.59 (q, 2 H, *J* = 5.7 Hz), 0.32 (q, 2 H, *J* = 4.5 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 155.2, 137.4, 118.9, 46.9, 45.9, 29.7, 22.8, 10.7, 3.6; EIMS *m/z* 265 (M^+, 71), 238 (63), 236 (91), 230 (86), 194 (72), 182 (57), 86 (94), 84 (100); HRMS (EI) *m/z* calcd for $C_{12}H_{16}ClN_5$ 265.1094, found 265.1096.

13p



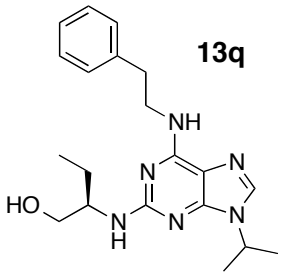
(*R*)-2-(6-(Cyclopropylmethylamino)-9-isopropyl-9*H*-purin-2-ylamino)butan-1-ol (13p). A mixture of **16p** (50.0 mg, 0.188 mmol) and (*R*)-(-)-2-amino-1-butanol (124.0 mg, 1.40 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 15 h. The reaction mixture was cooled to room temperature, diluted with water (7.0 mL), and extracted with EtOAc (4 x 10.0 mL). The combined organic phases were washed with warm water (50-55 °C, 2 x 5.0 mL), dried ($MgSO_4$), concentrated, and dried under high-vacuum overnight to yield a yellow oil. The yellow oil was dissolved in EtOAc and suspended in Et_2O. Dropwise addition of hexanes (minimal solvent added to achieve a homogeneous supernatant) precipitated an off-white solid. The solid was rinsed (Et_2O/hexanes, 2:1) by pipetting out the supernatant and dried to obtain crude **13p** (51.0 mg, 0.160 mmol, 85%) as an oil that was used without further purification: IR (ATR) 3286, 3086, 3068, 3055, 3038, 3030, 1647, 1627, 1592, 1575, 1560, 1446, 1314, 1273, 1204, 1174, 1159, 1150, 1075, 1027, 997, 943, 936, 917, 865, 813, 764, 719, 700, 691 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 7.52 (s, 1 H), 5.85 (bs, 1 H), 4.91 (bs, 1 H), 4.58

(hept, 1 H, *J* = 6.3 Hz), 3.92-3.80 (m, 2 H), 3.72-3.60 (m, 1 H), 3.60-3.30 (m, 2 H), 1.53 (d, 6 H, *J* = 5.7 Hz), 1.40-0.90 (m, 4 H), 1.03 (t, 3 H, *J* = 6.3 Hz), 0.57-0.52 (m, 2 H), 0.28 (bs, 2 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 160.1, 154.9, 134.4, 119.0, 114.6, 68.7, 56.4, 46.4, 29.7, 25.1, 22.6, 11.0, 3.5; EIMS *m/z* 318 (M^+, 46), 288 (54), 287 (100), 265 (36), 236 (82), 230 (71), 194 (41), 134 (46), 119 (32); HRMS (EI) *m/z* calcd for $C_{16}H_{26}N_6O$ 318.2168, found 318.2164.

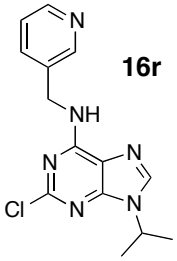


16q

2-Chloro-9-isopropyl-*N*-phenethyl-9*H*-purin-6-amine (16q). To a solution of **15c** (100 mg, 0.433 mmol) in *n*-BuOH (1.5 mL) were added 2-phenylethylamine (62.9 mg, 0.519 mmol) and Et_3N (70.8 mg, 0.692 mmol). The reaction mixture was heated under microwave irradiation at 120 °C for 60 min. *n*-BuOH was evaporated *in vacuo*, the residue was diluted with water, and extracted with EtOAc (3 x 7.0 mL). The combined organic extracts were dried ($MgSO_4$) and concentrated to yield a pale yellow solid. The solid was resuspended (hexanes/Et_2O, 2:1), filtered, and subsequently rinsed (hexanes/Et_2O, 3:1). The filtered solid was dried under high-vacuum to yield **16q** (112 mg, 0.353 mmol, 82%) as a pale yellow solid: Mp 146.7-148.7°C; IR (ATR) 3252, 3217, 3211, 3205, 3123, 2974, 1616, 1580, 1569, 1457, 1444, 1347, 1308, 1292, 1221, 1198, 1059, 745 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.75 (bs, 1 H), 7.33-7.21 (m, 5 H), 5.99 (bs, 1 H), 4.83 (hept, 1 H, *J* = 6.9 Hz), 3.91 (bs, 2 H), 3.00 (t, 2 H, *J* = 7.2 Hz), 1.58 (d, 6 H, *J* = 6.6 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 155.3, 149.7, 138.8, 137.5, 128.9, 128.6, 126.5, 118.9, 46.8, 42.0, 35.6, 22.9; EIMS *m/z* 315 (M^+, 82), 337 (23), 226 (93), 213 (83), 169 (84), 146 (93), 119 (100), 104 (83), 77 (87), 65 (81); HRMS (EI) *m/z* calcd for $C_{16}H_{18}ClN_5$ 315.1251, found 315.1244.



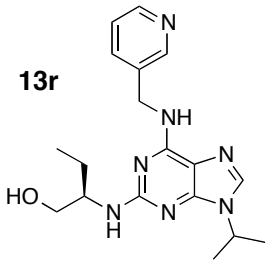
(*R*)-2-(9-Isopropyl-6-(phenethylamino)-9*H*-purin-2-ylamino)butan-1-ol (13q). A mixture of **16q** (51.0 mg, 0.161 mmol) and (*R*)-(-)-2-amino-1-butanol (72.0 mg, 0.792 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 15 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (4 x 10.0 mL). The combined organic layers were washed with warm water (50-55 °C, 2 x 5 mL), dried ($MgSO_4$), filtered, concentrated, and dried under high-vacuum at 70 °C (oil bath) for 2 h to yield an oily, yellow residue. The crude residue was purified by chromatography on SiO_2 (hexanes/EtOAc, 1:1, to EtOAc/MeOH/Et_3N, 84:5:1) to yield **13q** (20.4 mg, 0.0554 mmol, 34%) as a light yellow oil: IR (ATR) 3252, 3217, 3211, 3205, 3123, 2974, 1616, 1580, 1569, 1457, 1444, 1347, 1308, 1292, 1220, 1198, 1059, 745, 727 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 7.49 (s, 1 H), 7.34-7.20 (m, 5 H), 5.76 (bs, 1 H), 4.90 (d, 1 H, *J* = 5.7 Hz), 4.63 (hept, 1 H, *J* = 6.9 Hz), 3.95-3.80 (m, 4 H), 3.66 (dd, 2 H, *J* = 7.8, 10.5 Hz), 2.97 (t, 2 H, *J* = 7.2 Hz), 1.70-1.50 (m, 2 H), 1.53 (d, 6 H, *J* = 6.6 Hz), 1.05 (t, 3 H, *J* = 7.5 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 160.4, 155.2, 139.3, 134.7, 129.1, 128.9, 126.7, 115.0, 69.0, 56.7, 46.6, 42.1, 36.3, 25.3, 22.9, 11.3; EIMS *m/z* 368 (M^+, 84), 338 (77), 277 (43), 205 (77), 163 (77), 105 (85), 91 (100); HRMS (EI) *m/z* calcd for $C_{20}H_{28}N_6O$ 368.2325, found 368.2308.



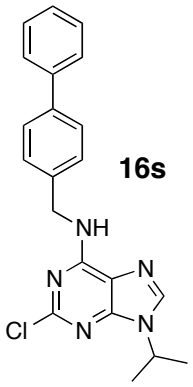
2-Chloro-9-isopropyl-*N*-(pyridin-3-ylmethyl)-9*H*-purin-6-amine (16r).[9] To a solution of **15c** (99.0 mg, 0.423 mmol) in *n*-BuOH (1.5 mL) were added 3-pyridinemethanamine (55.6 mg, 0.514 mmol) and Et_3N (70.1 mg, 0.685 mmol). The reaction mixture was heated under microwave irradiation at 120 °C for 20 min. *n*-BuOH was evaporated *in*

vacuo. The residue was diluted with water (5.0 mL) and extracted with EtOAc (3 x 7.0 mL). The combined organic extracts were dried ($MgSO_4$), filtered, and concentrated to yield a pale yellow solid. The solid residue was resuspended (hexanes/Et_2O, 2:1), filtered, and the solid was rinsed (hexanes/Et_2O, 3:1). The filtered solid was dried under high-vacuum to yield **16r** (112.0 mg, 0.389 mmol, 86%) as a pale yellow solid: ^1H NMR ($CDCl_3$, 300 MHz) δ 8.65 (s, 1 H), 8.54 (dd, 2 H, J = 1.2, 4.5 Hz), 7.73-7.70 (m, 1 H), 7.28-7.23 (m, 1 H), 6.62 (bs, 1 H), 5.00-4.75 (m, 2 H), 4.83 (hept, 1 H, J = 6.9 Hz), 1.57 (d, 6 H, J = 6.6 Hz).

13r

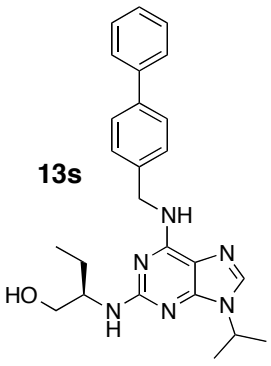


(*R*)-2-(9-Isopropyl-6-(pyridin-3-ylmethylamino)-9*H*-purin-2-ylamino)butan-1-ol (13r).[9] A mixture of **16r** (50.0 mg, 0.165 mmol) and (*R*)-(-)-2-amino-1-butanol (73.6 mg, 0.826 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 15 h. The reaction mixture was cooled to room temperature, diluted with water (5.0 mL), and extracted with EtOAc (4 x 10 mL). The combined organic phases were washed with warm water (50-55° C, 2 x 5 mL), dried ($MgSO_4$), filtered, concentrated, and dried under high-vacuum overnight to yield a yellow oil. The oil was dissolved in EtOAc and Et_2O, and upon drop-wise addition of hexanes an off-white solid precipitated. The solid was rinsed (Et_2O/hexanes, 2:1, 3x) by pipetting out the supernatant. After drying the solid under high-vacuum, **13r** (35.9 mg, 0.101 mmol, 61%) was obtained as a colorless amorphous solid: IR (ATR) 3252, 3217, 3211, 3205, 3123, 2974, 1616, 1580, 1569, 1457, 1444, 1347, 1308, 1292, 1221, 1198, 1059, 745, 727 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 8.60 (d, 1 H, J = 1.5 Hz), 8.48 (dd, 1 H, J = 1.2, 4.5 Hz), 7.70-7.65 (m, 1 H), 7.50-7.44 (m, 1 H), 7.20 (dd, 1 H, J = 4.8 Hz, 7.8 Hz), 6.57 (bs, 1 H), 4.98-4.95 (m, 1 H), 4.80-4.70 (m, 2 H), 4.58 (hept, 1 H, J = 6.9 Hz), 3.91-3.86 (m, 1 H), 3.77 (dd, 1 H, J = 3.0, 7.8 Hz), 3.61 (dd, 2 H, J = 7.2, 10.8 Hz), 1.75-1.40 (m, 2 H), 1.50 (d, 6 H, J = 6.6 Hz), 0.97 (t, 3 H, J = 7.5 Hz).



16s

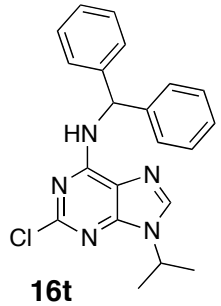
***N*-(Biphenyl-4-ylmethyl)-2-chloro-9-isopropyl-9*H*-purin-6-amine (16s)**.[2,9] To a solution of **15c** (150.0 mg, 0.649 mmol) in *n*-BuOH (1.5 mL) were added 4-phenylbenzylamine (0.125 g, 0.682 mmol) and triethylamine (108.0 mg, 1.06 mmol) under an N_2 atmosphere. The reaction mixture was heated under microwave irradiation at 120 °C for 20 min. *n*-BuOH was evaporated, and the residue was dissolved in EtOAc and washed with water. The aqueous phase was further extracted with EtOAc, and the combined organic extracts were dried ($MgSO_4$) and concentrated to yield a colorless solid. The solid was resuspended (hexanes/Et_2O, 3:1), filtered, and rinsed (hexanes/Et_2O, 3:1). The solid was dried under high-vacuum to yield **16s** (187.0 mg, 0.495 mmol, 76%) as an off-white solid: Mp 98-100 °C; IR (ATR, neat) 1615, 1571, 1350, 1308 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.68 (bs, 1 H), 7.61-7.57 (m, 4 H), 7.48-7.42 (m, 4 H), 7.36 (t, 1 H, *J* = 7.8 Hz), 6.55 (bs, 1 H), 4.88 (bs, 2 H), 4.82 (hept, 1 H, *J* = 6.6 Hz), 1.56 (d, 6 H, *J* = 6.6 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 155.1, 154.3, 149.8, 140.6, 137.7, 137.0, 128.8, 128.4, 127.4, 127.3, 127.1, 118.9, 46.9, 44.3, 22.8; HRMS (ES) *m/z* calcd for $C_{21}H_{20}ClN_5$ $[M+Na]^+$ 400.1305, found 400.1308.



13s

(*R*)-2-(6-(Biphenyl-4-ylmethylamino)-9-isopropyl-9*H*-purin-2-ylamino)butan-1-ol (13s).[2,9] A mixture of **16s** (32.0 mg, 0.0821 mmol), potassium fluoride (1.50 mg, 0.0258

mmol), and (*R*)-(-)-2-amino-1-butanol (61.8 mg, 0.651 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 12 h. The reaction mixture was cooled to room temperature, diluted with water, and extracted with EtOAc (4x). The combined organic phases were washed with warm water (50-55 °C, 2x), dried ($MgSO_4$), filtered, concentrated, and dried under high-vacuum at 70 °C (oil bath) for 2 h to yield an amorphous yellow semi-solid. After addition of Et_2O, the product was precipitated from the solution by drop-wise addition of hexanes (added in a minimal amount to achieve a homogeneous mixture). The solid was rinsed (Et_2O/hexanes, 2:1) by pipetting out the supernatant. The solid was dried under high-vacuum at 40 °C overnight (to eliminate a volatile impurity, ~0.9 ppm) to obtain **13s** (25 mg, 0.0581 mmol, 71%) as a light yellow solid: Mp 116-119 °C; IR (ATR, neat) 3265, 1600, 1542, 1485 cm^{-1}; ^1H NMR ($CDCl_3$, 600 MHz) δ 7.60-7.52 (m, 4 H), 7.46-7.39 (m, 5 H), 7.34 (t, 1 H, *J* = 7.8 Hz), 6.52 (bs, 1 H), 4.97 (s, 1 H), 4.80 (bs, 2 H), 4.59 (hept, 1 H, *J* = 6.6 Hz), 3.96-3.88 (m, 1 H), 3.83 (dd, 1 H, *J* = 10.8, 2.4 Hz), 3.51 (dd, 1 H, *J* = 10.8, 7.8 Hz), 1.68-1.50 (m, 2 H), 1.51 (d, 6 H, *J* = 6.6 Hz), 1.02 (t, 3 H, *J* = 7.8 Hz); ^{13}C NMR ($CDCl_3$, 150 MHz) δ 160.0, 154.8, 150.1, 140.8, 140.2, 138.0, 134.5, 128.7, 128.1, 127.3, 127.2, 127.0, 114.6, 68.3, 56.2, 46.4, 44.0, 25.0, 22.5, 22.4, 10.9; HRMS (ES) *m/z* calcd for $C_{25}H_{30}N_6O$ $[M+H]^+$ 431.2559, found 431.2538.

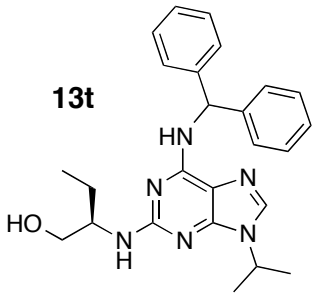


16t

***N*-Benzhydryl-2-chloro-9-isopropyl-9*H*-purin-6-amine (16t).** To a solution of **15c** (100.0 mg, 0.433 mmol) in *n*-BuOH (1.5 mL) were added diphenylamine (95.2 mg, 0.519 mmol) and Et_3N (70.8 mg, 0.692 mmol). The reaction mixture was heated under microwave irradiation at 120 °C for 20 min. *n*-BuOH was evaporated *in vacuo*, and the residue was diluted with water (5.0 mL), and extracted with EtOAc (3 x 7.0 mL). The combined organic extracts were dried ($MgSO_4$), filtered, and concentrated to yield a pale yellow solid that was resuspended (hexanes/Et_2O, 2:1), filtered, and washed

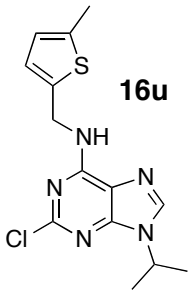
(hexanes/Et_2O, 3:1). The filtrate was dried under high-vacuum to obtain **16t** (117 mg, 0.310 mmol, 72%) as a pale yellow solid: Mp 191.1-193.2 °C; IR (ATR) 3252, 3217, 3211, 3205, 3123, 2974, 1616, 1580, 1569, 1457, 1444, 1347, 1308, 1292, 1221, 1198, 1059, 745, 727 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 7.63 (bs, 1H), 7.32-7.26 (m, 10 H), 6.76 (bs, 1 H), 4.80 (hept, 1 H, *J* = 6.6 Hz), 1.55 (d, 6 H, *J* = 6.9 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 154.4, 150.0, 141.5, 137.9, 128.8, 127.7, 127.5, 127.2, 118.8, 57.3, 46.9, 22.8; EIMS *m/z* 377 (M^+, 98), 379 (35), 334 (25), 182 (44), 167 (100), 165 (61); HRMS (EI) *m/z* calcd for $C_{21}H_{20}ClN_5$ 377.1407, found 377.1400.

13t



(*R*)-2-(6-(Benzhydrylamino)-9-isopropyl-9*H*-purin-2-ylamino)butan-1-ol (13t). A mixture of **16t** (50.0 mg, 0.132 mmol) and (*R*)-(-)-2-amino-1-butanol (87.5 mg, 0.981 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 15 h. The reaction mixture was cooled to room temperature, diluted with water (5.0 mL), and extracted with EtOAc (4 x 10.0 mL). The combined organic layers were washed with warm water (50-55 °C, 2 x 5.0 mL), dried ($MgSO_4$), filtered, concentrated, and dried under high-vacuum overnight to obtain a yellow oil. The oil was dissolved in EtOAc, resuspended in Et_2O, and hexanes was added drop-wise to achieve a homogeneous supernatant. An off-white solid precipitated from the solution, and the solid was rinsed (Et_2O/hexanes, 2:1) by pipetting out the supernatant and dried under high-vacuum to obtain **13t** (36.4 mg, 0.0845 mmol, 64%) as an off-white solid: Mp 72.2-75.0 °C; IR (ATR) 3286, 3086, 3068, 3055, 3038, 3030, 1647, 1627, 1592, 1575, 1560, 1446, 1314, 1273, 1204, 1174, 1159, 1150, 1075, 1027, 997, 943, 936, 917, 865, 813, 764, 719, 701, 691 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz): δ 7.46 (s, 1 H), 7.34-7.26 (m, 10 H), 6.53 (bs, 1 H), 6.41 (bs, 1 H), 4.81 (d, 1 H, *J* = 5.1 Hz), 4.59 (hept, 1 H, *J* = 6.6 Hz), 3.78-3.70 (m, 2 H), 3.57-3.51 (dd, 2 H, *J* = 7.5, 9.9 Hz), 1.6-1.3 (m, 2 H), 1.51 (d, 6 H, *J* = 6.6 Hz), 0.96 (t, 3 H, *J* = 7.5 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 159.9, 153.9, 142.2, 134.6, 128.5, 127.6,

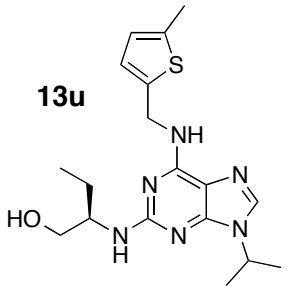
127.3, 114.6, 67.9, 57.9, 56.1, 46.4, 24.9, 22.6, 10.9; EIMS *m/z* 430 (M^+, 89), 400 (78), 399 (100), 358 (36), 168 (63), 165 (91), 152 (59); HRMS (EI) *m/z* calcd for $C_{25}H_{30}N_6O$ 430.2481, found 430.2486.



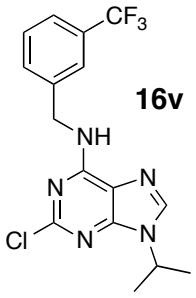
16u

2-Chloro-9-isopropyl-*N*-((5-methylthiophen-2-yl)methyl)-9*H*-purin-6-amine (16u). To a solution of **15c** (74.4 mg, 0.322 mmol) in *n*-BuOH (1.25 mL) was added (5-methylthien-2-yl)methylamine•HCl (55.7 mg, 0.340 mmol) and freshly distilled Et_3N (98.0 mg, 0.969 mmol) under an N_2 atmosphere. The reaction mixture was subjected to microwave irradiation at 120 °C for 30 min. White crystals were observed upon completion of the heating. *n*-BuOH was evaporated *in vacuo*, and the residue was dissolved in EtOAc (20.0 mL) and deionized water (10.0 mL). The aqueous phase was further extracted with EtOAc (3 x 10.0 mL), and the combined organic layers were dried ($MgSO_4$), filtered, and concentrated to yield a light yellow solid. The solid was resuspended (hexanes/Et_2O, 3:1), and the precipitated solid was filtered through a fritted funnel and dried under high-vacuum overnight to obtain **16u** (96.9 mg, 0.301 mmol, 94%) as light yellow amorphous solid: IR (ATR, neat) 3340, 3256, 3213, 3184, 3137, 3120, 3064, 2977, 2967, 2921, 1705, 1676, 1620, 1569, 1538, 1463, 1351, 1310, 1290, 1256, 1224, 1200, 1159, 1098, 1070, 1036, 1010, 969, 956, 798, 787, 761, 736, 725, 695, 678, 658 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.77 (bs, 1 H), 6.85 (d, 1 H, *J* = 3.3 Hz), 6.60 (d, 1 H, *J* = 3.3 Hz), 6.25 (bs, 1 H), 4.89 (bs, 2 H), 4.83 (sept, 1 H, *J* = 6.7 Hz), 2.45 (s, 3 H), 1.58 (d, 6 H, *J* = 6.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.8, 154.2, 149.9, 139.9, 138.5, 137.8, 126.0, 124.7, 118.8, 46.9, 39.6, 22.8, 15.4; HRMS (ES) *m/z* calcd for $C_{14}H_{16}N_5SCl$ 321.0850, found 321.0813.

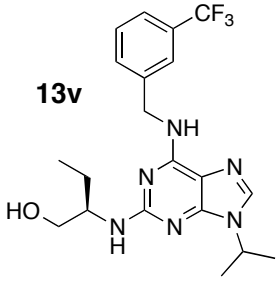
13u



(*R*)-2-(9-Isopropyl-6-((5-methylthiophen-2-yl)methylamino)-9*H*-purin-2-ylamino)butan-1-ol (13u). A mixture of **16u** (65.3 mg, 0.203 mmol) and (*R*)-2-aminobutan-1-ol (96.2 mg, 101 uL, 1.01 mmol, 5 equiv) were heated in a microwave vial immersed in an oil bath at 170 °C for 8 h. The reaction mixture was cooled to room temperature, diluted with water (15 mL), and extracted with EtOAc (3 x 20.0 mL). The combined organic phases were washed with warm water (2 x 10.0 mL, 50-55 °C), dried ($MgSO_4$), filtered, concentrated, and dried under high-vacuum at 50 °C (oil bath) for 2 h to yield a yellow solid. The crude residue was purified by chromatography on SiO_2 (hexanes/EtOAc, 9:1) to yield **13u** (61.8 mg, 0.165 mmol, 81%) as a light yellow foam: IR (ATR, neat) 3341, 3272, 3121, 2964, 2925, 2052, 2185, 1681, 1605, 1544, 1512, 1493, 1456, 1380, 1311, 1253, 1202, 1161, 1102, 1042, 1025, 971, 904, 861, 800, 755, 727, 723, 694, 675 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.47 (s, 1 H), 6.79 (d, 1 H, J = 3.6 Hz), 6.58-6.56 (m, 1 H), 5.98 (bs, 1 H), 5.25-5.00 (b, 1 H), 4.90 (app d, 1 H, J = 5.6 Hz), 4.82 (bs, 2 H), 4.59 (sept, 1 H, J = 6.8 Hz), 3.96-3.88 (m, 1 H), 3.84 (dd, 1 H, J = 10.4, 2.0 Hz), 3.65 (dd, 1 H, J = 10.8, 8.0 Hz), 2.42 (s, 3 H), 1.70-1.50 (m, 2 H), 1.52 (d, 6 H, J = 6.8 Hz), 1.04 (t, 3 H, J = 7.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.0, 154.4, 150.4, 139.7, 139.0, 134.6, 125.8, 124.7, 114.7, 68.6, 56.4, 46.5, 39.7, 29.7, 25.0, 22.6, 15.4, 11.0; HRMS (ESI) *m/z* calcd for $C_{18}H_{27}N_6OS$ $[M+Na]^+$ 397.1767, found 397.1787.



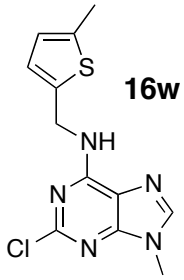
2-Chloro-9-isopropyl-*N*-(3-(trifluoromethyl)benzyl)-9*H*-purin-6-amine (16v).[10] To a solution of **15c** (65.8 mg, 0.273 mmol) in *n*-BuOH (1.0 mL) were added 3-(trifluoromethyl)benzylamine (52.4 mg, 0.300 mmol) and Et_3N (46.1 mg, 0.456 mmol) under a nitrogen atmosphere. The reaction mixture was heated with microwave irradiation at 120 °C for 30 min. The *n*-BuOH was evaporated, and the residue was dissolved in EtOAc (20.0 mL) and washed with water (10.0 mL). The aqueous phase was further extracted with EtOAc (2 x 10.0 mL), and the combined organic extracts were dried ($MgSO_4$), filtered, and concentrated to yield a colorless solid. The solid was resuspended (hexanes/Et_2O, 3:1), filtered, washed (hexanes/Et_2O, 3:1), and dried under high-vacuum to yield **16v** (63.0 mg, 0.170 mmol, 60%) as a colorless amorphous solid: IR (ATR, neat) 3250, 3150, 2990, 2925, 1625, 1446, 1313, 1230, 1159, 1140, 1099, 980, 930, 700 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.77 (s, 1 H), 7.63 (s, 1 H), 7.59 (d, 1 H, J = 7.5 Hz), 7.56 (d, 1 H, J = 7.8 Hz), 7.47 (t, 1 H, J = 7.5 Hz), 6.33 (bs, 1 H), 4.90 (bs, 2 H), 4.83 (sept, 1 H, J = 6.8 Hz), 1.58 (d, 6 H, J = 6.9 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.1, 154.2, 150.0, 139.4, 137.8, 131.2, 130.9 (q, J = 32.2 Hz), 129.3, 124.4, 124.3, 124.0 (q, J = 270.1 Hz), 118.8, 47.0, 43.9, 22.7; HRMS [EI] *m/z* calcd for [$C_{16}H_{15}ClF_3N_5$] 369.0968, found 369.9640.



(*R*)-2-(9-Isopropyl-6-(3-(trifluoromethyl)benzylamino)-9*H*-purin-2-ylamino)butan-1-ol (13v). A mixture of **16v** (56.2 mg, 0.152 mmol) and (*R*)-(-)-2-aminobutan-1-ol (95.0

[10] Dvorak, L.; Popa, I.; Starha, P.; Travnicek, Z., "In vitro cytotoxic-active platinum(II) complexes derived from carboplatin and involving purine derivatives." *Eur. J. Inorg. Chem.* **2010**, 3441-3448.

mg, 1.07 mmol) was heated in a microwave vial immersed in an oil bath at 170 °C for 8 h. The reaction mixture was cooled to room temperature, diluted with water (10.0 mL), and extracted with EtOAc (2 x 15.0 mL). The combined organic phases were washed with warm water (2 x 5.0 mL, 50-55 °C), dried ($MgSO_4$), filtered, concentrated, and dried under high-vacuum at 50 °C (oil bath temperature) for 2 h to yield a yellow solid. After addition of Et_2O, an off-white solid precipitated. The solid was rinsed (Et_2O, 3x) by pipetting out the supernatant and dried under high-vacuum overnight at 40 °C to yield **13v** (16.1 mg, 0.0381 mmol, 25%) as a colorless amorphous solid: Mp 149.9-153.6 °C; IR (ATR, neat) 3254, 3059, 2934, 1621, 1599, 1535, 1323, 1260, 1161, 1118, 1062, 797, 701 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.77 (s, 1 H), 7.58-7.51 (m, 3 H), 7.44 (t, 1 H, *J* = 7.5 Hz), 6.22 (bs, 1 H), 4.89 (d, 1 H, *J* = 5.7 Hz), 4.83 (bs, 2 H), 4.62 (sept, 1 H, *J* = 6.8 Hz), 3.95-3.80 (m, 1 H), 3.82 (dd, 1 H, *J* = 2.7, 10.5 Hz), 3.63 (dd, 1 H, *J* = 7.5, 10.5 Hz), 1.70-1.40 (m, 2 H), 1.54 (d, 6 H, *J* = 6.9 Hz), 1.01 (t, 3 H, *J* = 7.5 Hz); ^{13}C NMR (175 MHz, $CDCl_3$) δ 159.9, 154.6, 150.3, 140.0, 134.7, 130.9, 130.8 (q, *J* = 31.5 Hz), 129.0, 124.4 (q, *J* = 3.5 Hz), 124.1 (q, *J* = 3.5 Hz), 124.1 (q, *J* = 271.3 Hz), 114.5, 68.2, 56.2, 46.5, 43.8, 24.9, 22.5 (2 C), 10.8; HRMS (ES) *m/z* calcd for $C_{20}H_{26}F_3N_6O$ $[M+H]^+$ 423.2120, found 423.2103.

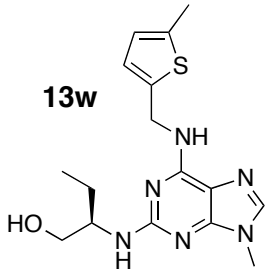


16w

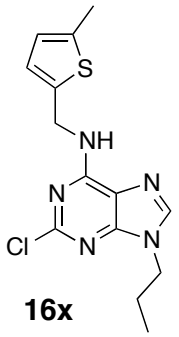
2-Chloro-9-methyl-*N*-[(5-methylthiophen-2-yl)methyl]-9H-purin-6-amine (16w). To a solution of **15b** (30.0 mg, 0.148 mmol, 1 eq) in dry *n*-BuOH (0.6 mL, 0.25 M) were added (5-methylthien-2-yl)methylamine•HCl (25.4 mg, 0.155 mmol, 1.05 eq) and freshly distilled triethylamine (44.9 mg, 0.443 mmol, 3 eq, 0.06 mL) under nitrogen. The reaction mixture was subjected to microwave irradiation at 120 °C for 35 min. The *n*-BuOH was evaporated, and the residue was dissolved in EtOAc (20 mL) and deionized water (10 mL). The layers were separated and the aqueous layer was further extracted with EtOAc (3 x 10 mL). The combined organic layers were dried ($MgSO_4$) and concentrated under reduced pressure to yield a yellow solid. The solid was washed with Et_2O and dried

under high-vacuum to yield **16w** (37.5 mg, 0.128 mmol, 86%) as a light yellow solid: Mp 217.5-219.6 ºC; IR (ATR, neat) 3058, 1607, 1575, 1340, 1303, 1232, 1094, 917, 796, 693 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 8.76 (bs, 1 H), 8.11 (s, 1 H), 6.79 (d, 1 H, J = 3.3 Hz), 6.60 (app d, 1 H, J = 2.2 Hz), 4.68 (d, 2 H, J = 5.6 Hz), 3.69 (s, 3 H), 2.34 (s, 3 H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 154.4, 152.9, 150.4, 142.1, 139.6, 138.4, 125.7, 124.6, 118.1, 38.5, 29.6, 14.9; HRMS (ES) *m/z* calcd for $C_{12}H_{11}N_5SCl$ $[M-H]^+$ 292.0424, found 292.0428.

13w

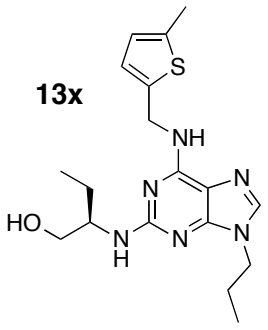


(2*R*)-2-[(9-Methyl-6-{[(5-methylthiophen-2-yl)methyl]amino}-9H-purin-2-yl)amino]butan-1-ol (13w). A mixture of **16w** (18.2 mg, 0.0620 mmol, 1 eq) and (*R*)-(-)-2-amino-1-butanol (27.6 mg, 0.029 mL, 0.276 mmol, 5 eq) was heated in a sealed vial in an oil bath at 170 ºC for 15 h. The reaction mixture was cooled at room temperature, treated with water (15 mL) and then extracted with EtOAc (3 x 20 mL). The combined organic layers were washed with warm water (2 x 10 mL, 50-55 ºC), dried ($MgSO_4$), filtered and concentrated. Purification by chromatography on SiO_2 (EtOAc/MeOH/Et_3N, 94:5:1) gave a yellow oil which was dried under high-vacuum at 50 ºC for 2 h to yield **13w** (20.0 mg, 0.0577 mmol, 93%) as a yellow oil which solidified to a dark yellow solid: Mp 56.4-58.3 ºC; IR (ATR, neat) 2934, 1601, 1545, 1512, 1415, 1215, 785 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.35 (s, 1 H), 6.77 (d, 1 H, J = 3.2 Hz), 6.55 (app d, 1 H, J = 2.3 Hz), 6.20 (bs, 1 H), 4.97 (d, 1 H, J = 6.1 Hz), 4.80 (bs, 2 H), 3.95 (app pent, 1 H, J = 5.8 Hz), 3.82 (dd, 1 H, J = 10.7, 2.6 Hz), 3.63 (dd, 1 H, J = 10.7, 7.5 Hz), 3.61 (s, 3 H), 2.41 (s, 3 H), 1.66-1.52 (m, 2 H), 1.02 (t, 3 H, J = 7.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.2, 154.3, 151.1, 139.5, 139.1, 137.6, 125.7, 124.6, 114.1, 67.9, 56.0, 39.5, 29.3, 25.0, 15.3, 10.9; HRMS (ES) *m/z* calcd for $C_{16}H_{23}N_6OS$ $[M+H]^+$ 347.1654, found 347.1659.



16x

2-Chloro-*N*-[(5-methylthiophen-2-yl)methyl]-9-propyl-9*H*-purin-6-amine (16x). To a solution of **15a** (33.9 mg, 0.147 mmol, 1 eq.) in *n*-BuOH (0.6 mL, 0.25 M) were added (5-methylthien-2-yl)methylamine·HCl (25.2 mg, 0.153 mmol, 1.05 eq.) and freshly distilled triethylamine (44.5 mg, 0.440 mmol, 3.00 equiv). The reaction mixture was subjected to microwave irradiation at 120 °C for 30 min. The residue was dissolved in EtOAc (20 mL) and deionized water (10 mL). The aqueous phase was further extracted with EtOAc (3 x 10 mL), and the combined organic layers were dried ($MgSO_4$), filtered and concentrated under reduced pressure. Purification on SiO_2 (EtOAc/hexanes, 1:1) gave **16x** (42.1 mg, 0.131 mmol, 89%) as a colorless solid: Mp 150.3-151.4 ºC; IR (ATR, neat) 3256, 3208, 2994, 2872, 1616, 1573, 1538, 1472, 1303, 1251, 1219, 1085, 811 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.57 (bs, 1 H), 6.80 (d, 1 H, *J* = 3.3 Hz), 6.75 (bs, 1 H), 6.57 (app dd, 1 H, *J* = 3.3, 1.0 Hz), 4.87 (bs, 2 H), 4.09 (t, 2 H, *J* = 7.2 Hz), 2.42 (s, 3 H), 1.88 (sext, 2 H, *J* = 7.4 Hz), 0.94 (t, 3 H, *J* = 7.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.8, 154.5, 150.5, 140.4, 140.2, 138.1, 126.4, 124.9, 118.7, 45.6, 39.8, 23.5, 15.5, 11.2; HRMS (ES) *m/z* calcd for $C_{14}H_{17}N_5SCl$ $[M+H]^+$ 322.0893, found 322.0890.



13x

(2*R*)-2-[(6-{[(5-Methylthiophen-2-yl)methyl]amino}-9-propyl-9*H*-purin-2-yl)amino]butan-1-ol (13x). A mixture of **16x** (20.0 mg, 0.0621 mmol, 1 equiv) and (*R*)-(-)-2-amino-1-butanol (27.7 mg, 0.311 mmol, 5 equiv) was heated in a sealed vial in an oil

bath at 170 ºC for 15 h. The reaction mixture was cooled to room temperature and water was added (15 mL). The mixture was extracted with EtOAc (3 x 20 mL), dried ($MgSO_4$), filtered and concentrated. Purification by chromatography on SiO_2 (EtOAc/MeOH, 98:2) provided **13x** (21.2 mg, 0.0566 mmol, 91%) as a colorless solid: Mp 124.8-129.6 ºC; IR (ATR, neat) 3418, 3374, 3260, 2958, 1605, 1512, 1402, 1333, 1215, 798, 796, 783 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.40 (s, 1 H), 6.79 (d, 1 H, J = 3.4 Hz), 6.57-6.56 (m, 1 H), 6.04 (bs, 1 H), 4.96 (app d, 1 H, J = 5.5 Hz), 4.82 (bs, 2 H), 3.97-3.88 (m, 3 H), 3.83 (dd, 1 H, J = 10.7, 2.5 Hz), 3.64 (dd, 1 H, J = 10.7, 7.7 Hz), 2.42 (s, 3 H), 1.84 (sext, 2 H, J = 7.2 Hz), 1.70-1.51 (m, 2 H), 1.03 (t, 3 H, J = 7.4 Hz), 0.93 (t, 3 H, J = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.1, 154.4, 151.0, 139.8, 139.1, 137.4, 126.0, 124.8, 114.5, 68.6, 56.5, 45.2, 39.7, 25.2, 23.3, 15.5, 11.3, 11.1; HRMS (ES) *m/z* calcd for $C_{18}H_{27}N_6OS$ $[M+H]^+$ 375.1967, found 375.1968.

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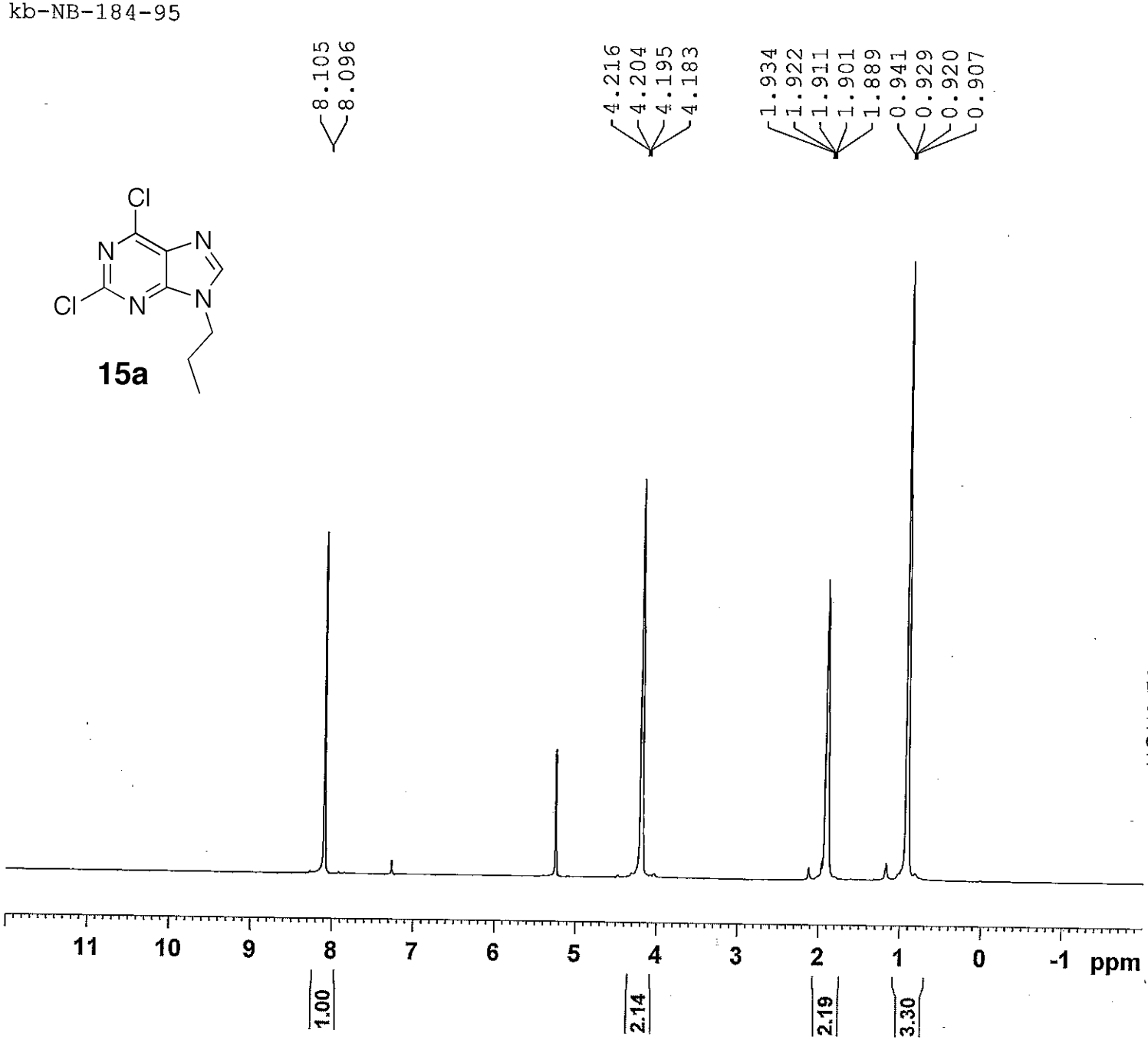

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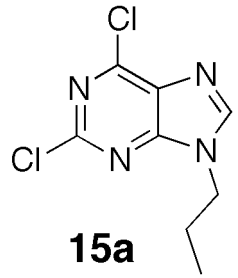
kb-NB-184-95



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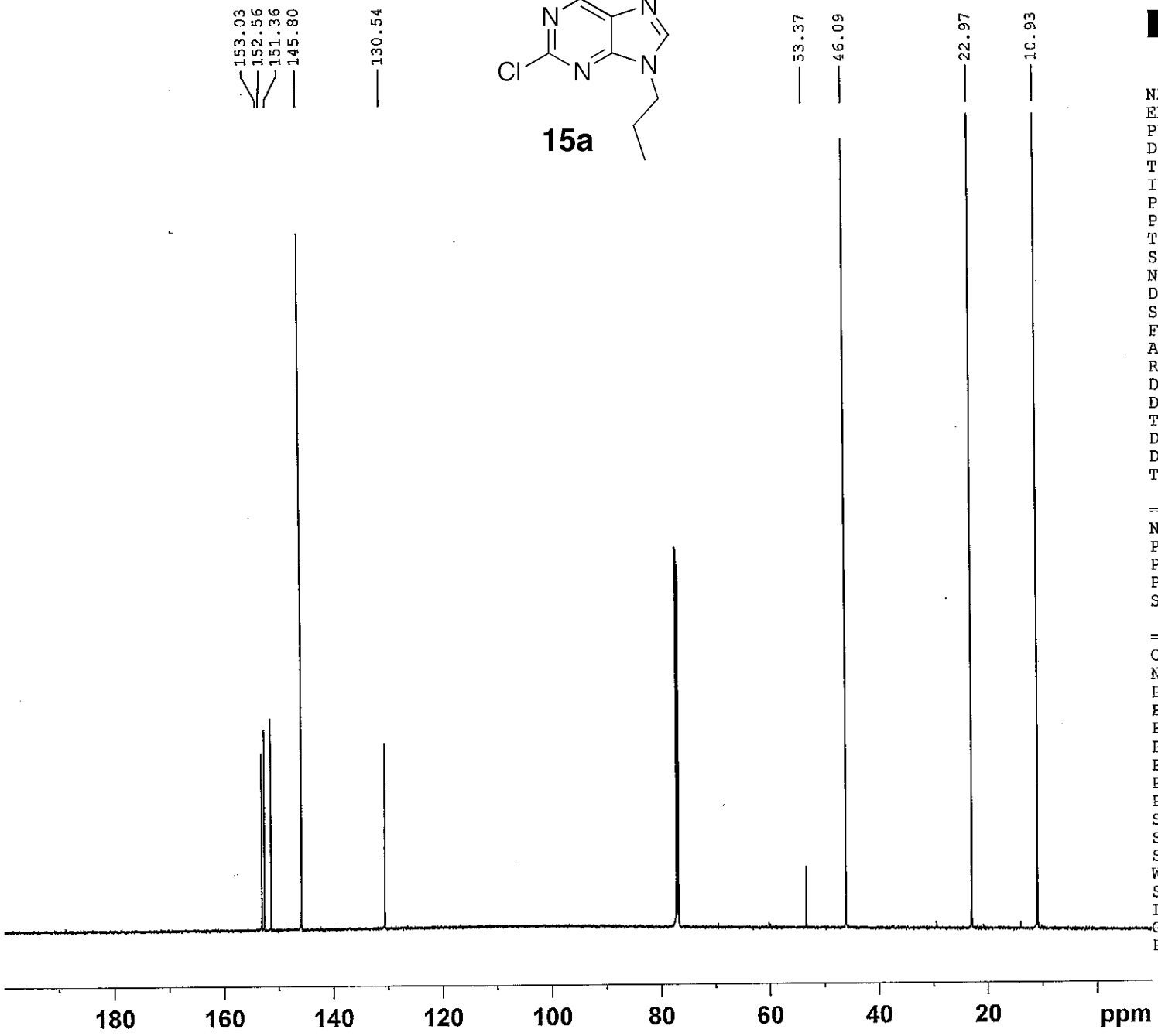

BRUKER

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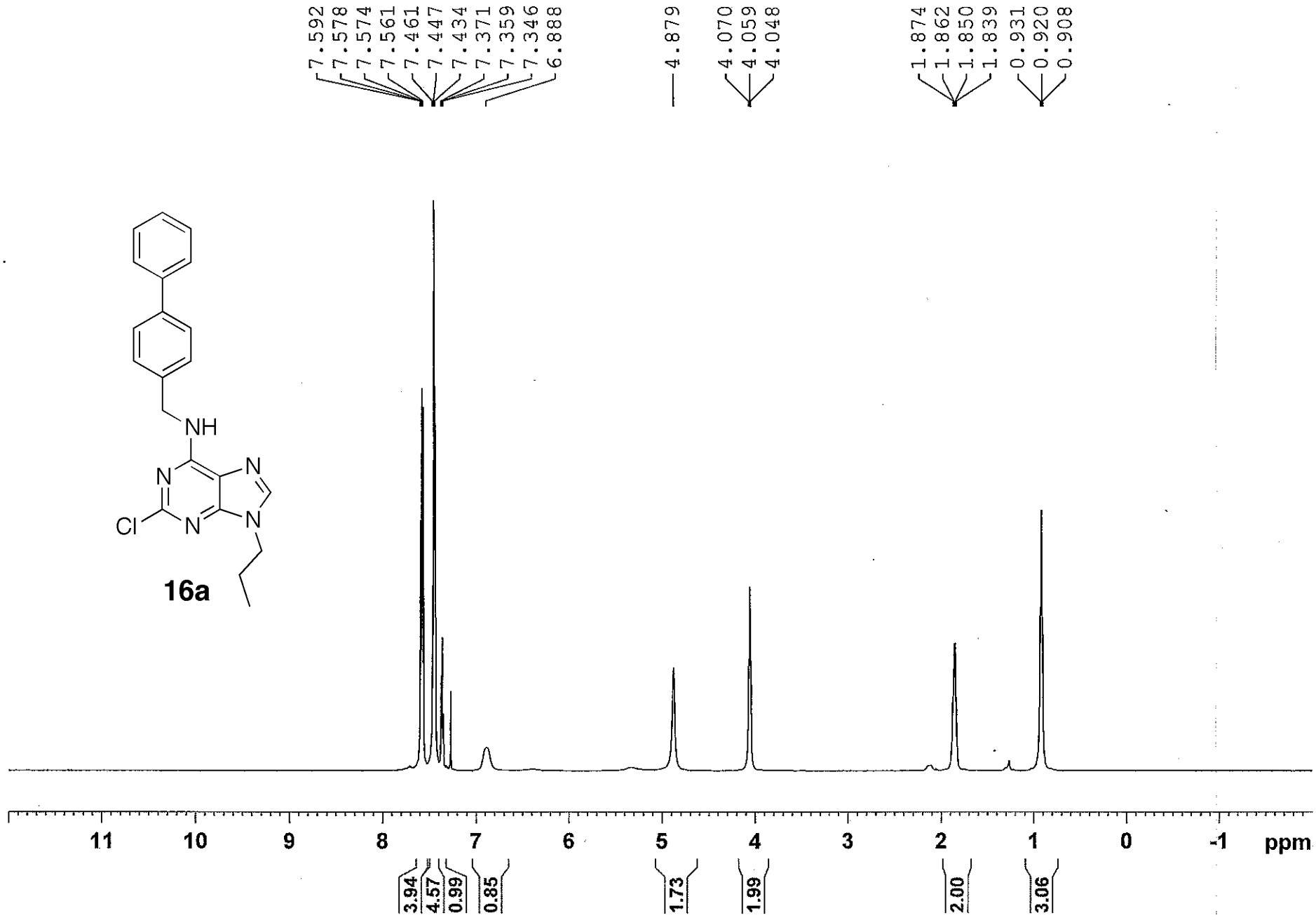
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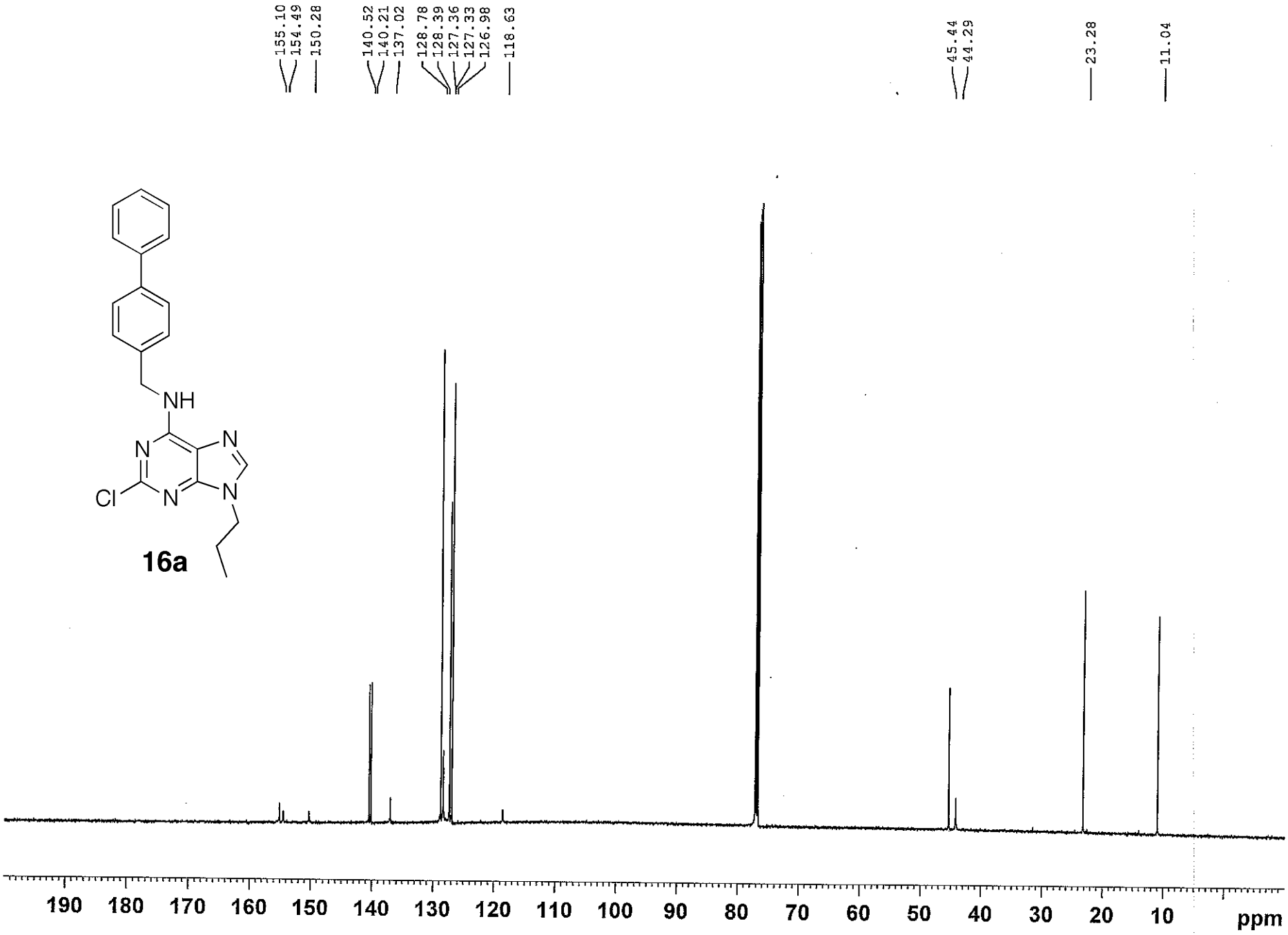
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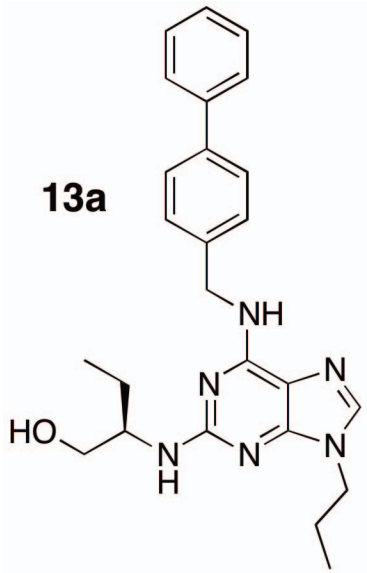
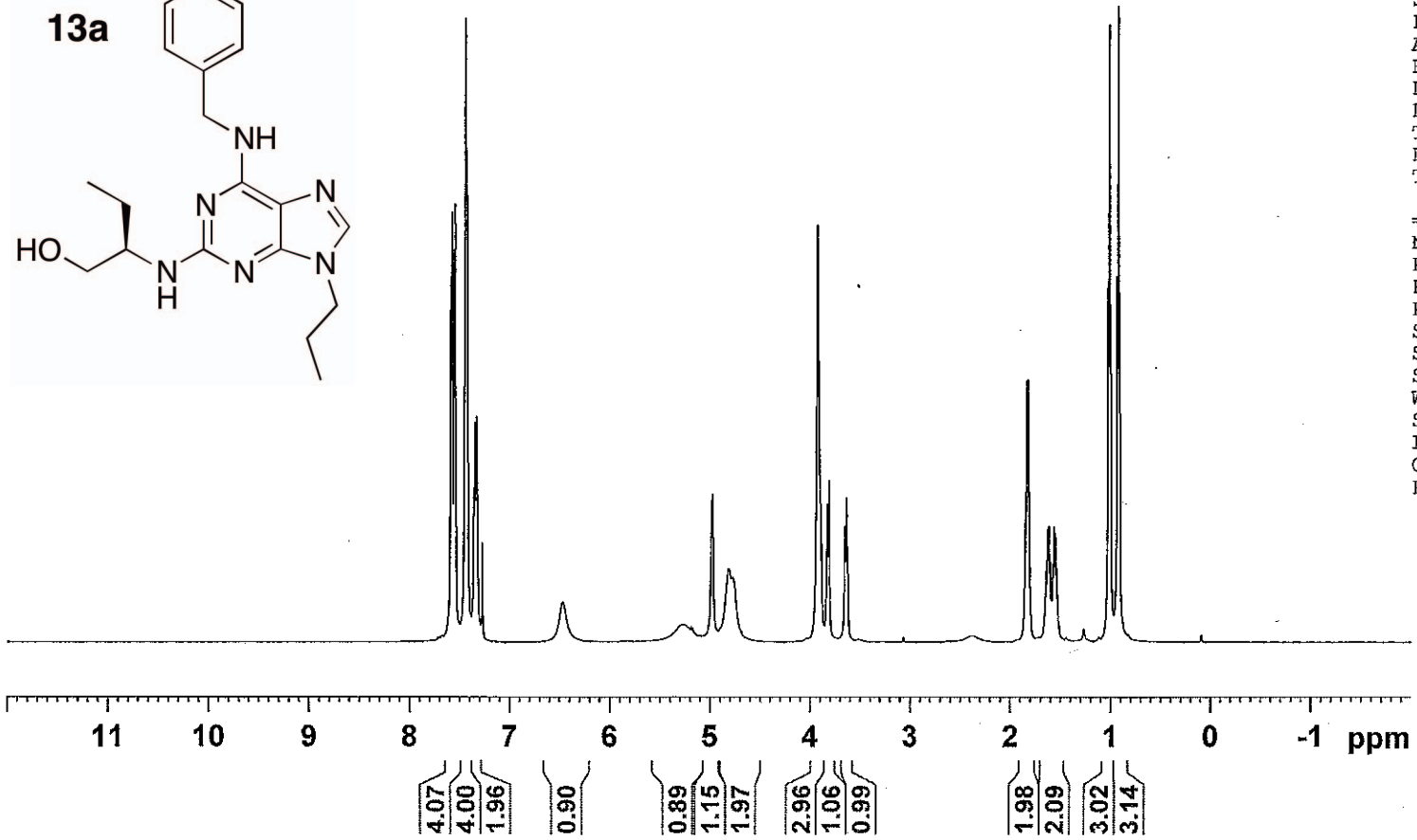
kb-NB-213-02

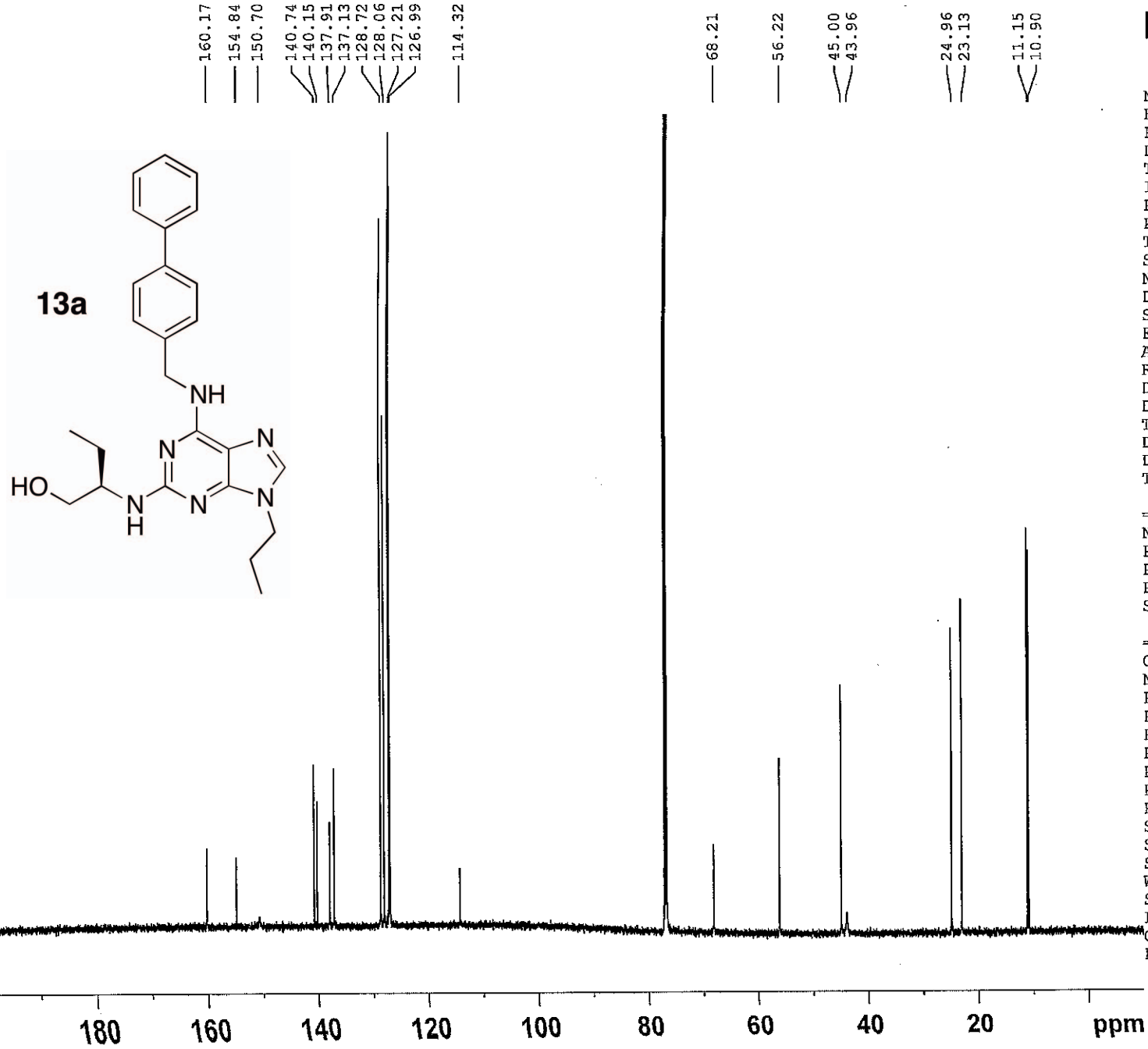
7.584 7.571 7.551 7.538 7.448 7.432 7.420 7.356 7.344 7.327 7.270 6.465 5.267 5.186 4.978 4.812 4.774 3.939 3.927 3.916 3.830 3.813 3.652 3.639 3.623 1.846 1.834 1.822 1.810 1.637 1.625 1.614 1.603 1.575 1.563 1.551 1.540 1.025 1.012 1.000 0.934 0.922 0.910





NAME	kb-NB-213-02
EXPNO	1
PROCNO	1
Date_	20090723
Time	10.21
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	32
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300044 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

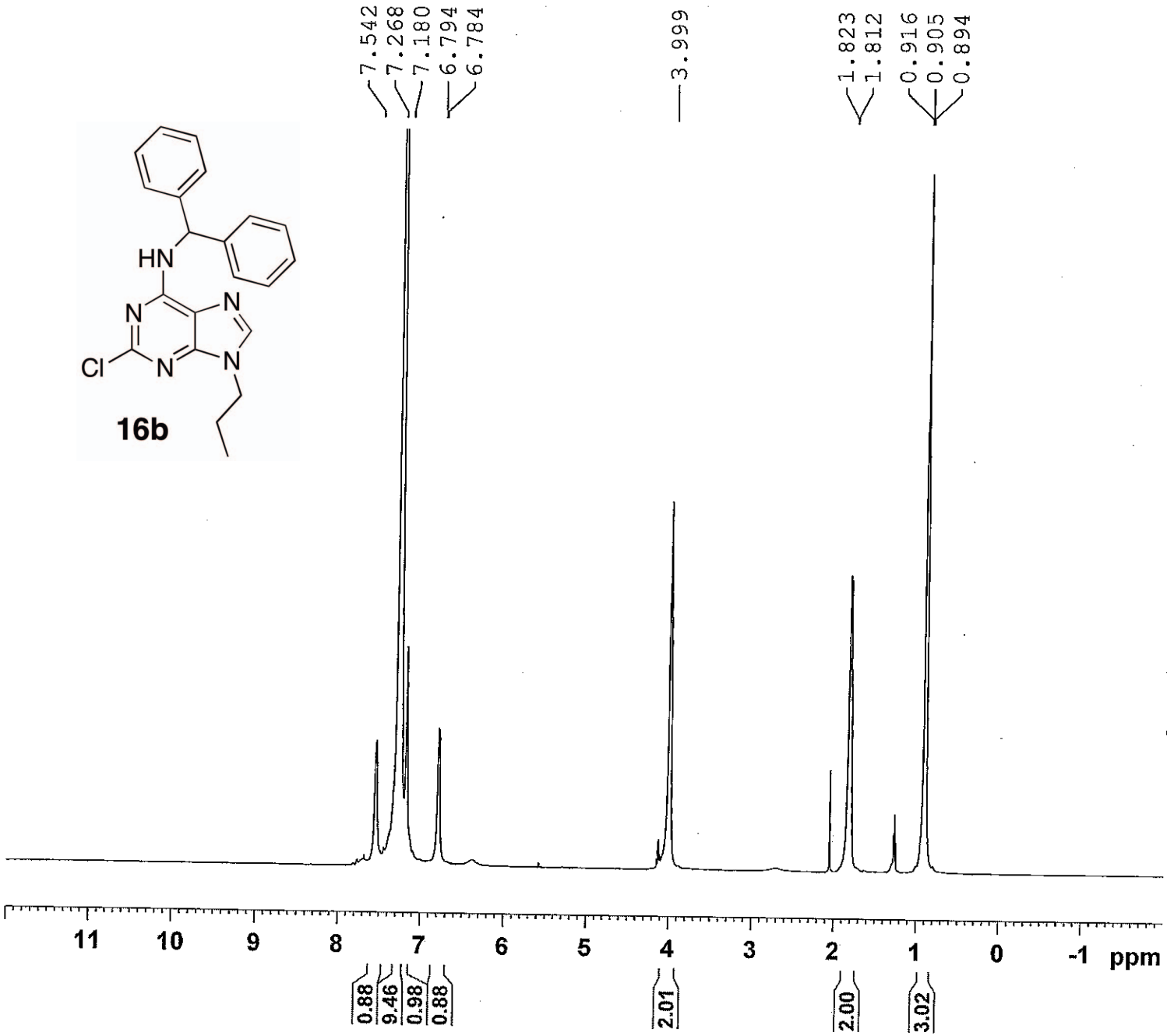
kb-NB-213-02



NAME	kb-NB-213-02
EXPNO	2
PROCNO	1
Date_	20090723
Time	10.37
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	16384
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028186 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

kb-NB-213-01

16b



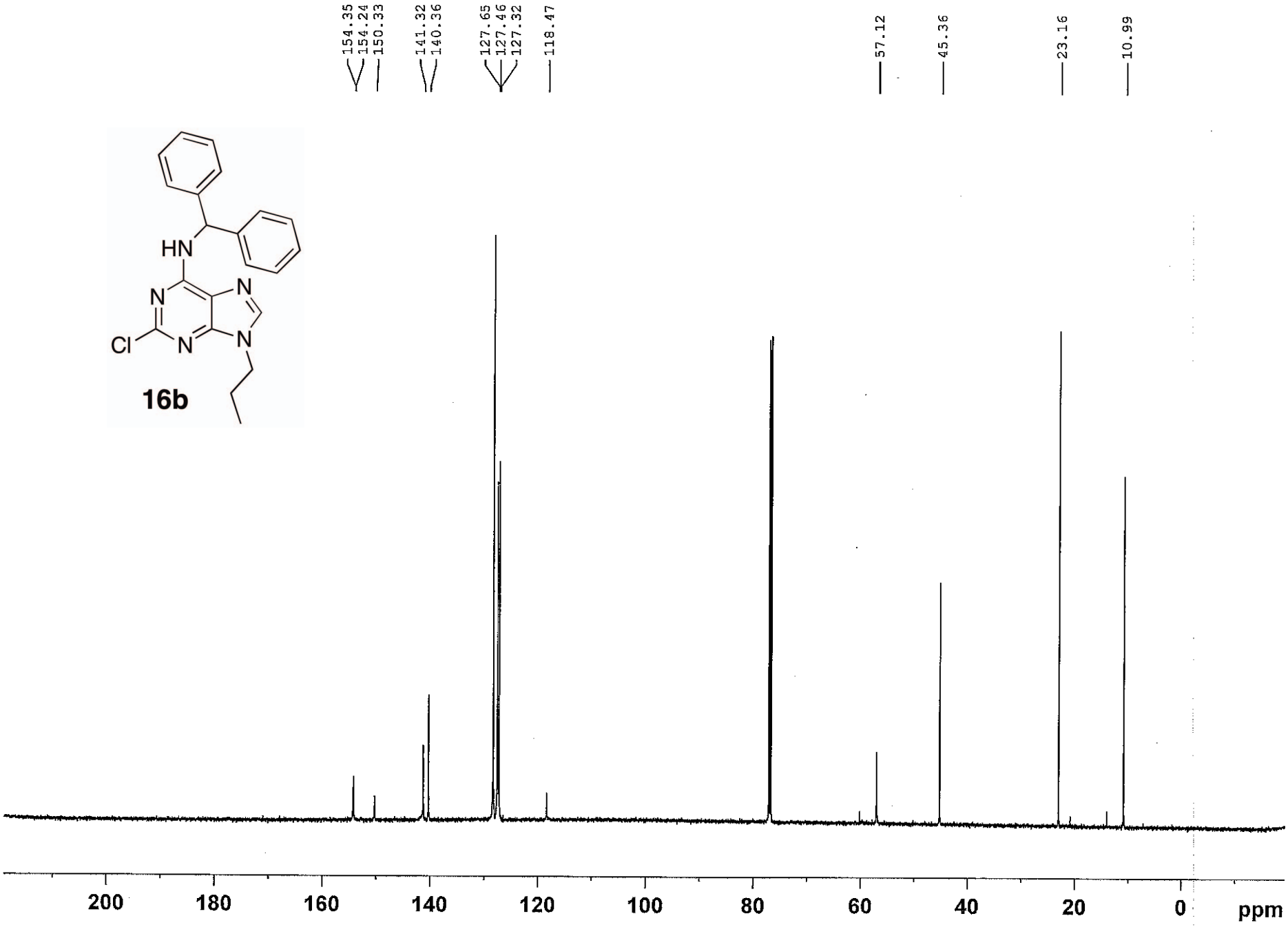
BRUKER

NAME	kb-NB-213-01
EXPNO	1
PROCNO	1
Date_	20090721
Time	10.28
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	14.3
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1

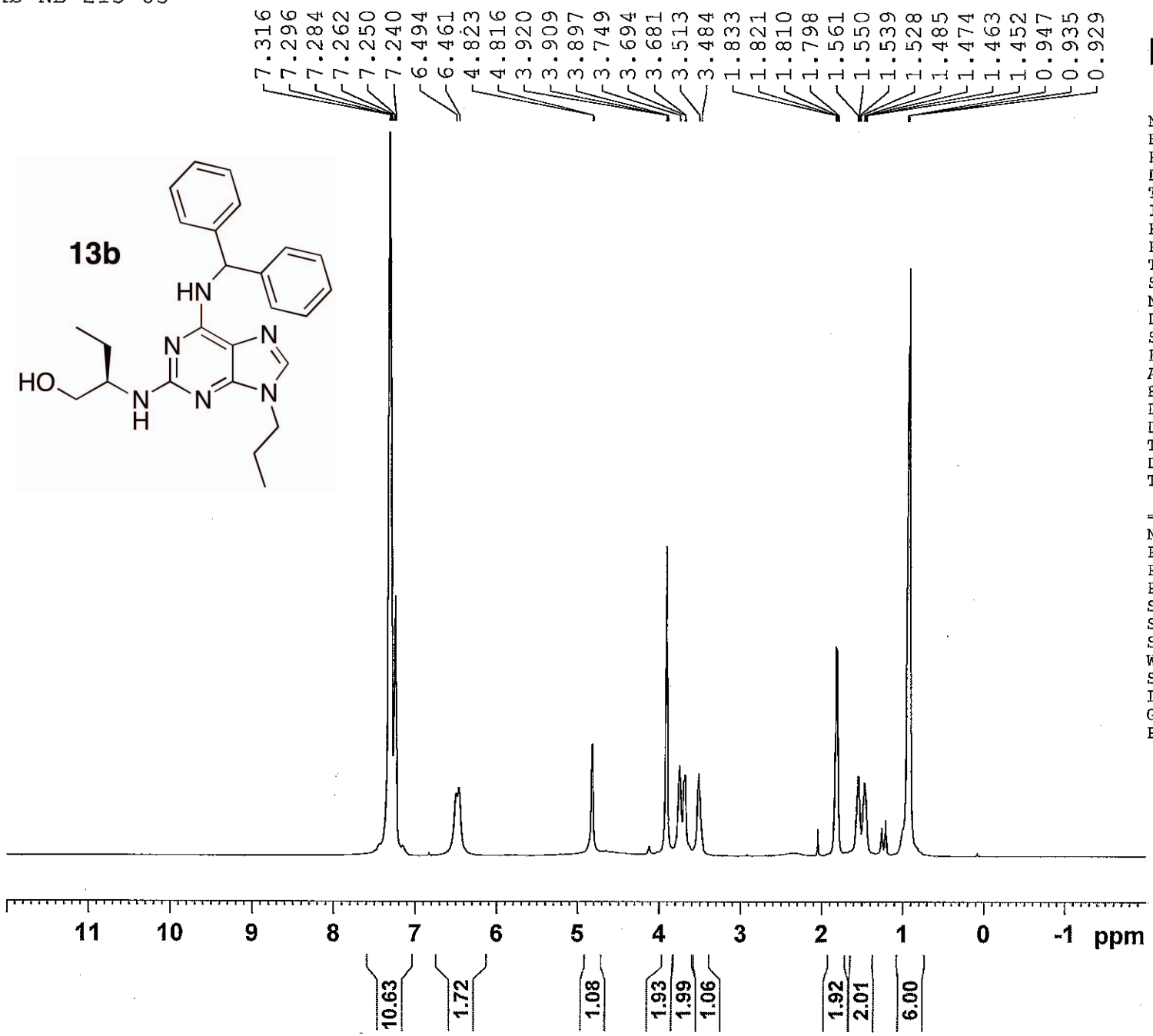
======== CHANNEL f1 ========

NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300040 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

kb-NB-213-01


154.35
154.24
150.33
141.32
140.36
127.65
127.46
127.32
118.47
57.12
45.36
23.16
10.99
HN
N
N
Cl
N
N
16b
200
180
160
140
120
100
80
60
40
20
0
ppm

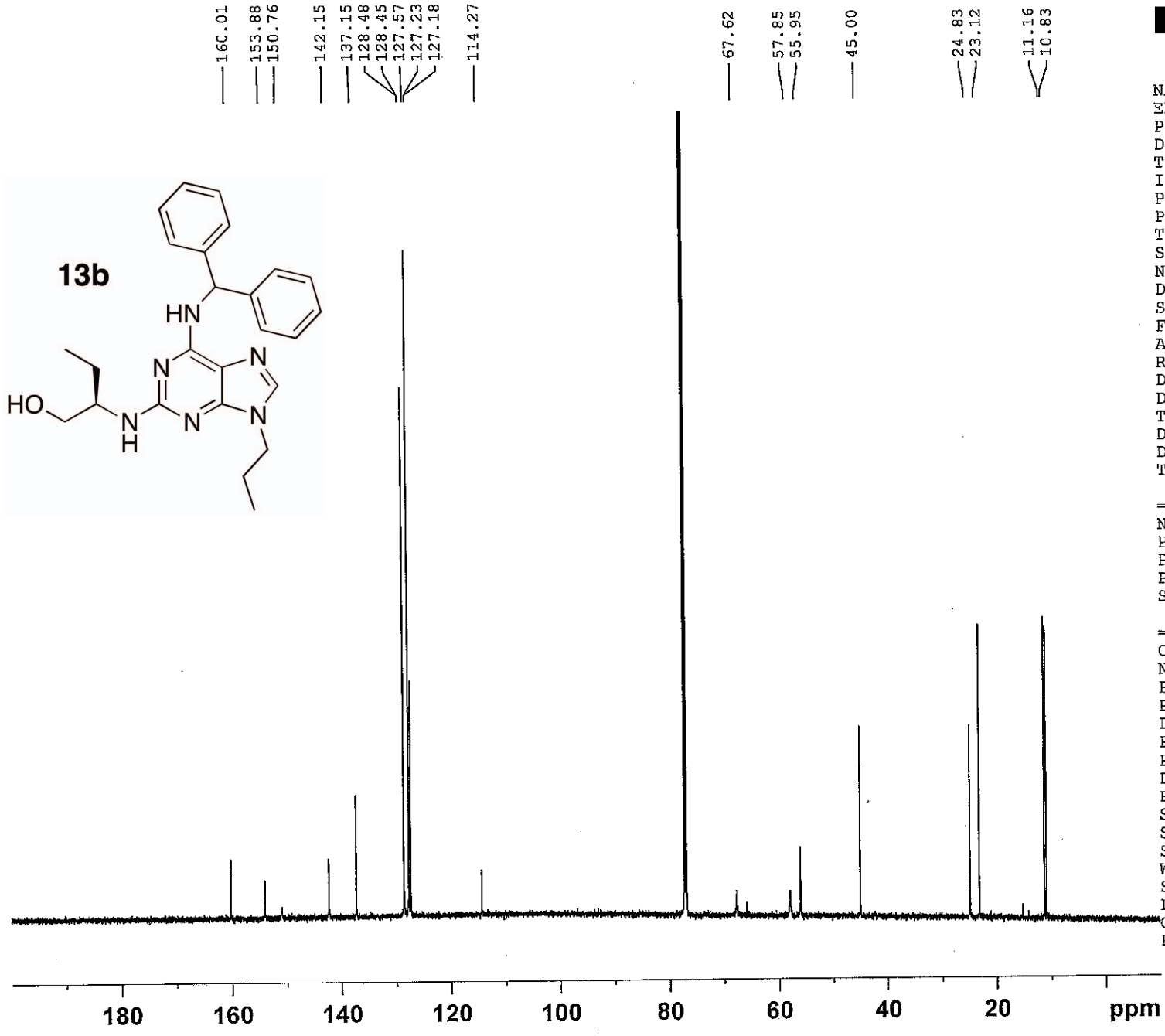
kb-NB-213-03



BRUKER

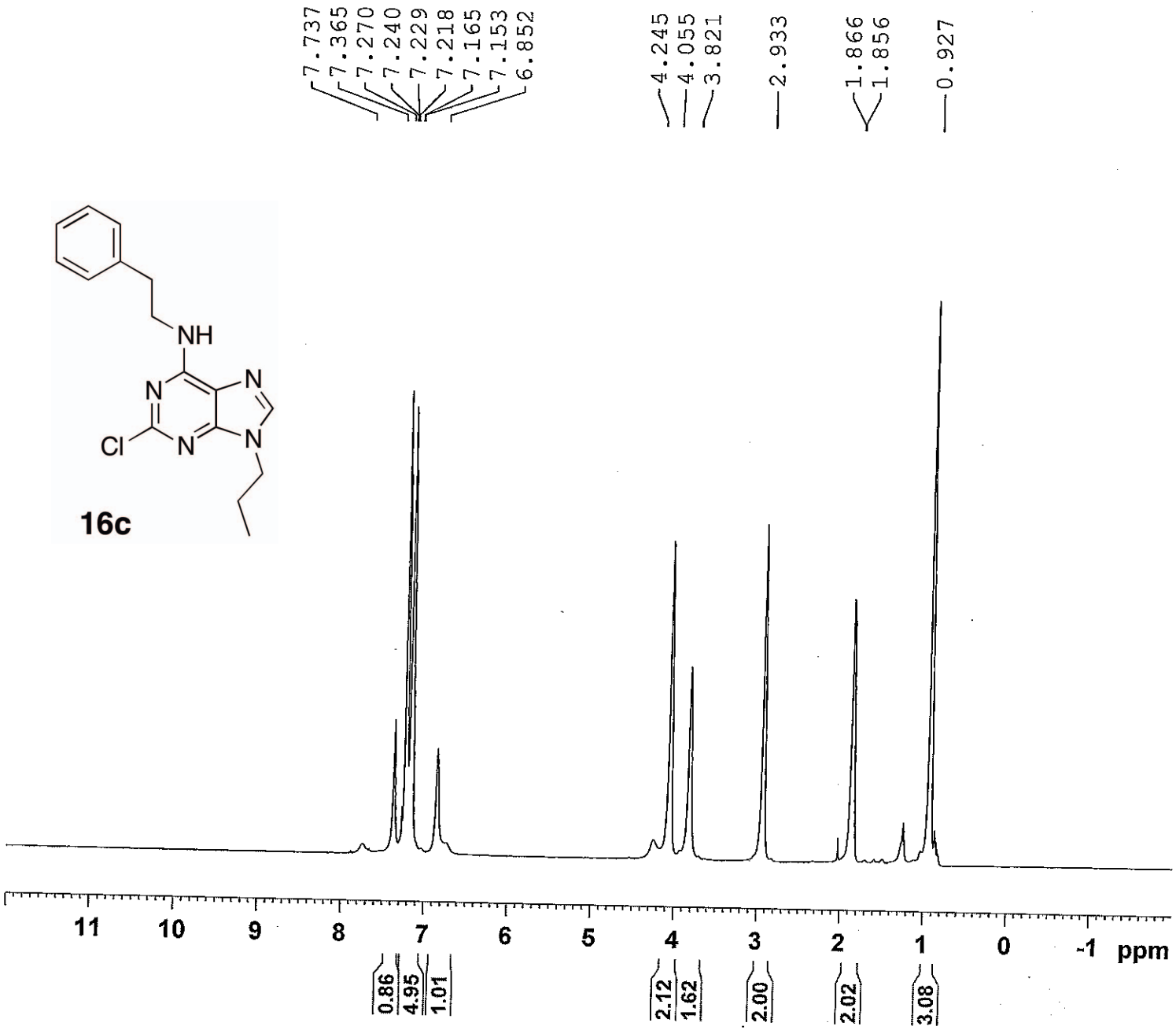
NAME	kb-NB-213-03
EXPNO	1
PROCNO	1
Date_	20090723
Time	10.44
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	28.5
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300089 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

kb-NB-213-03



NAME	kb-NB-213-03
EXPNO	2
PROCNO	1
Date_	20090723
Time	10.59
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	11585.2
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028175 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

kb-NB-213-04



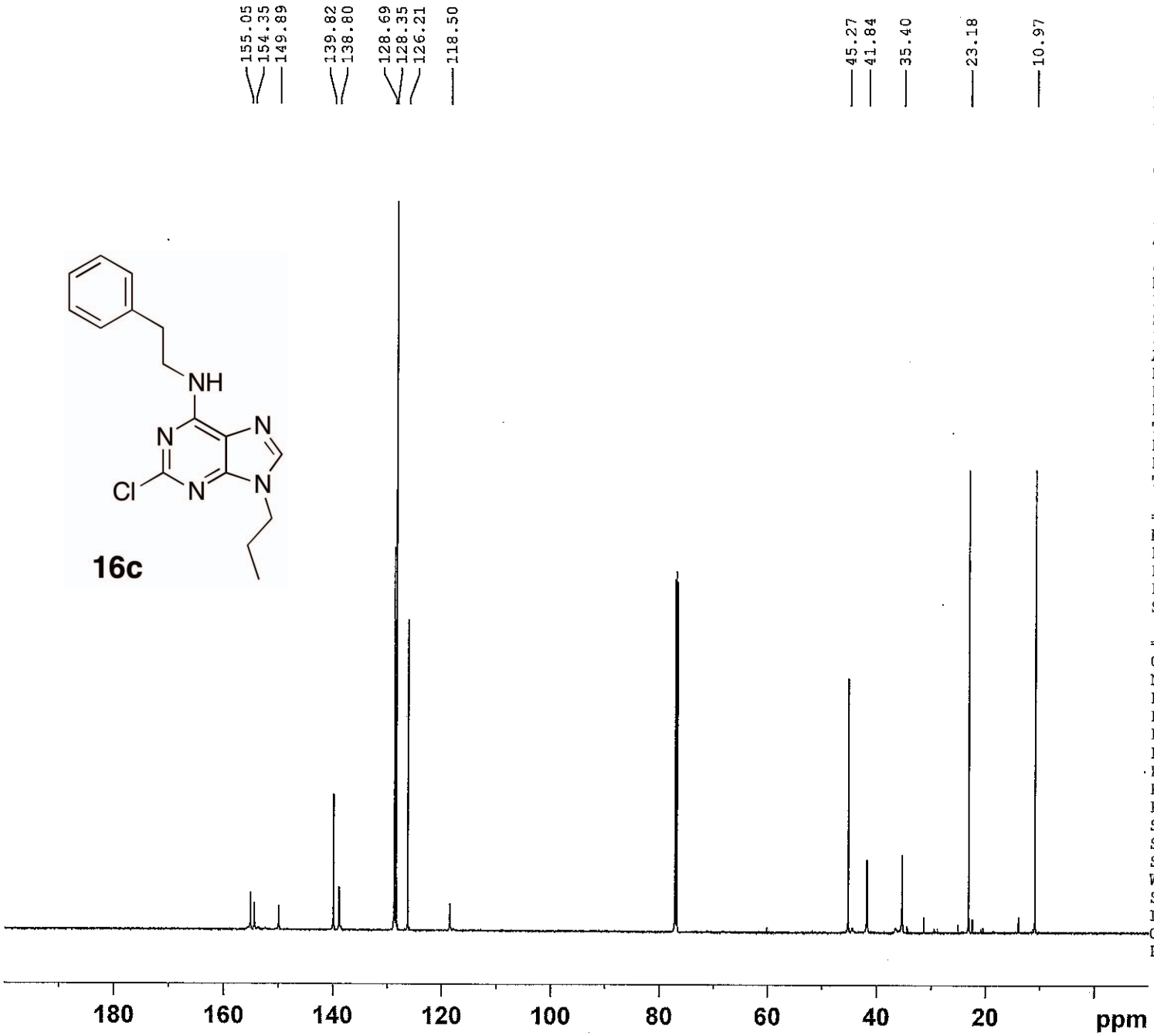
BRUKER

NAME	kb-NB-213-04
EXPNO	2
PROCNO	1
Date_	20090722
Time	9.19
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	10.1
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1

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

NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300044 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

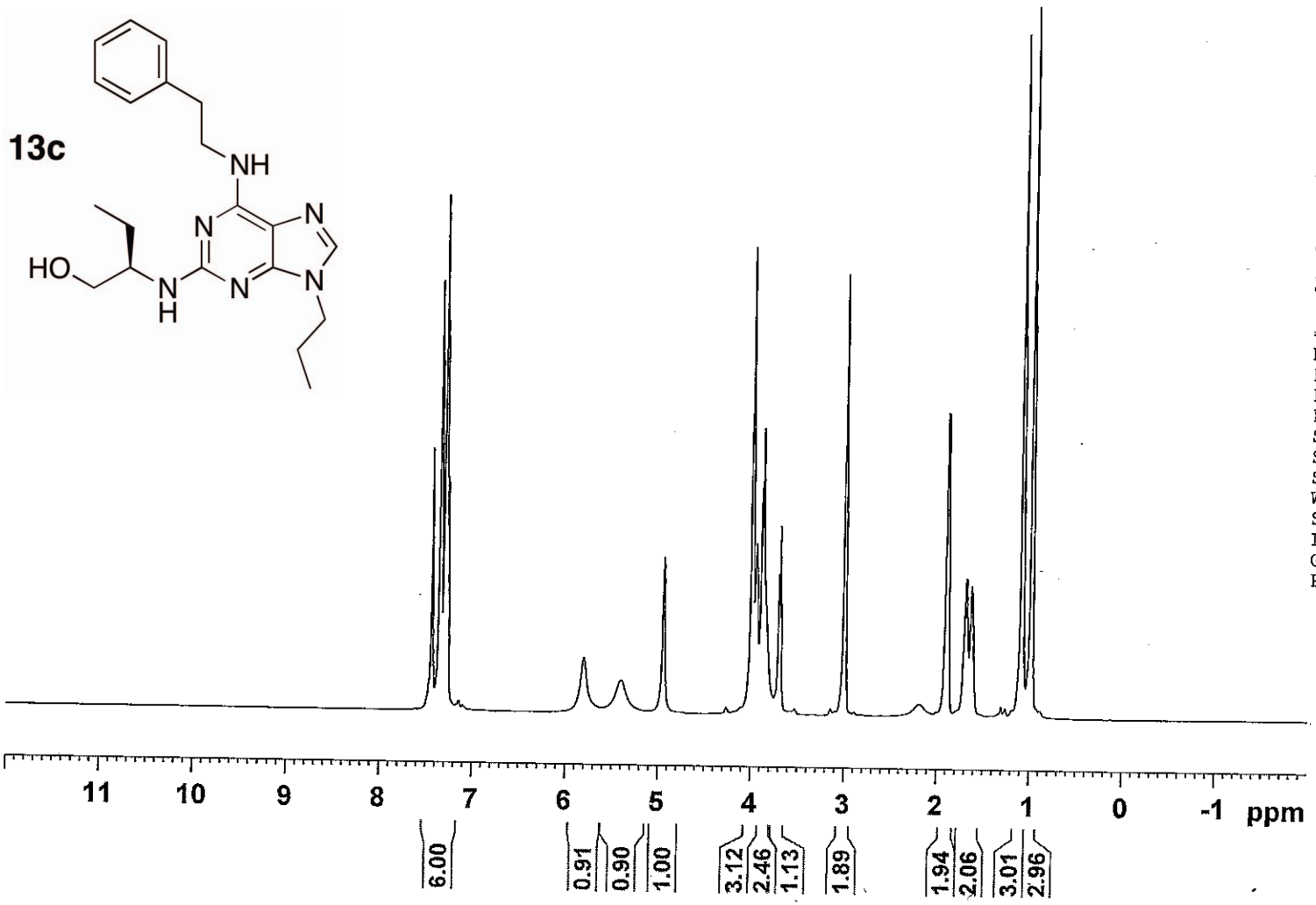
kb-NB-213-04



NAME	kb-NB-213-04
EXPNO	3
PROCNO	1
Date_	20090722
Time	9.35
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	13004
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028351 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

kb-NB-213-07

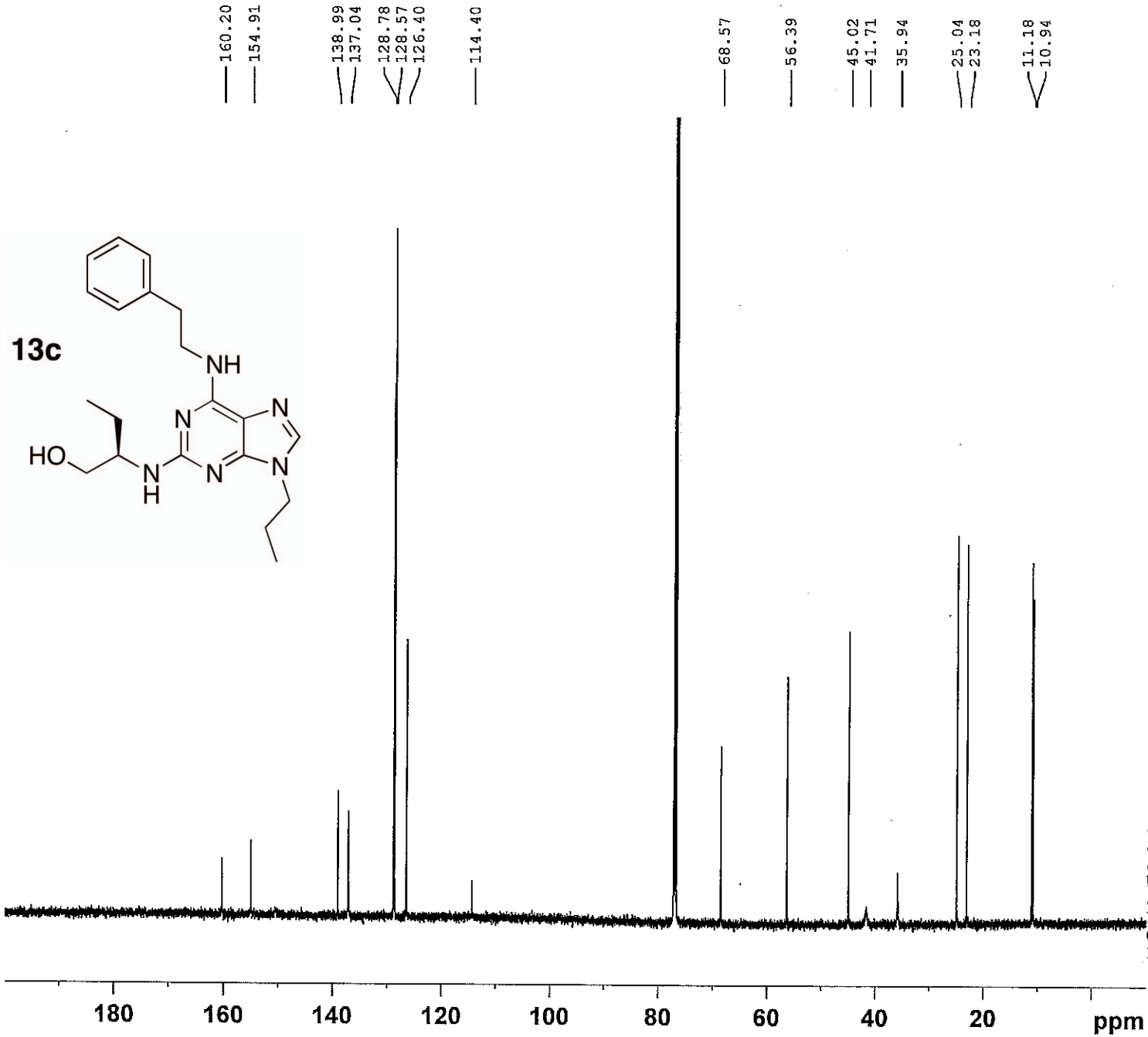
7.436 7.362 7.351 7.340 7.313 7.301 7.290 7.268 5.805 5.401 4.946 4.009 3.998 3.987 3.953 3.899 3.882 3.710 3.696 3.680 3.010 2.999 1.912 1.900 1.888 1.877 1.706 1.695 1.684 1.673 1.645 1.633 1.622 1.611 1.098 1.087 1.079



BRUKER

NAME	kb-NB-213-07
EXPNO	1
PROCNO	1
Date_	20090724
Time	15.29
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	57
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1299778 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

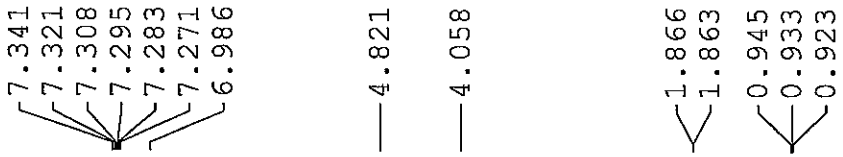
kb-NB-213-07

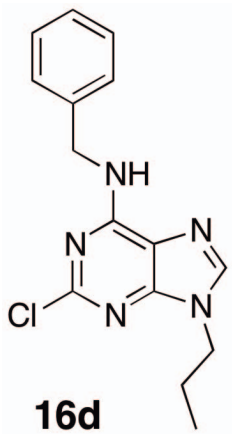


BRUKER

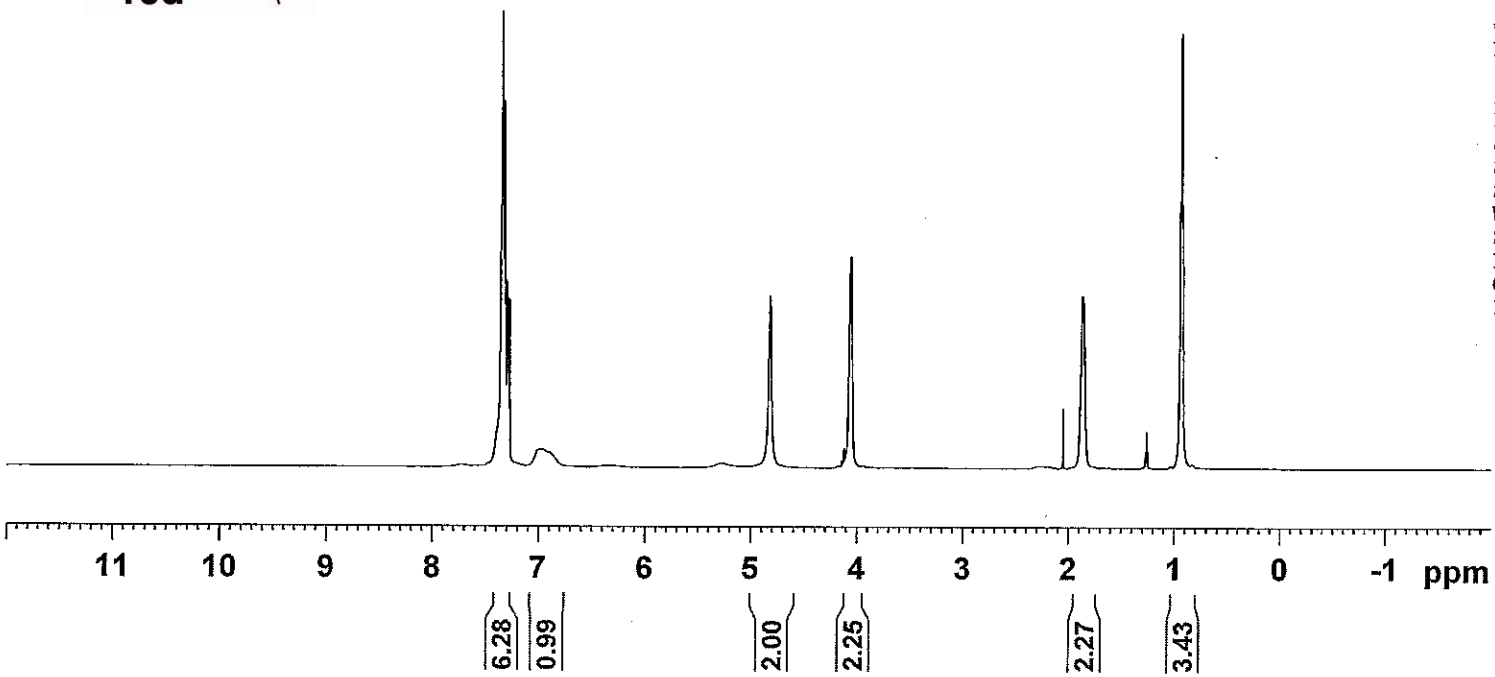
NAME	kb-NB-213-07
EXPNO	2
PROCNO	1
Date_	20090724
Time	15.48
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	320
DS	2
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	13004
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028151 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

kb-NB-213-05



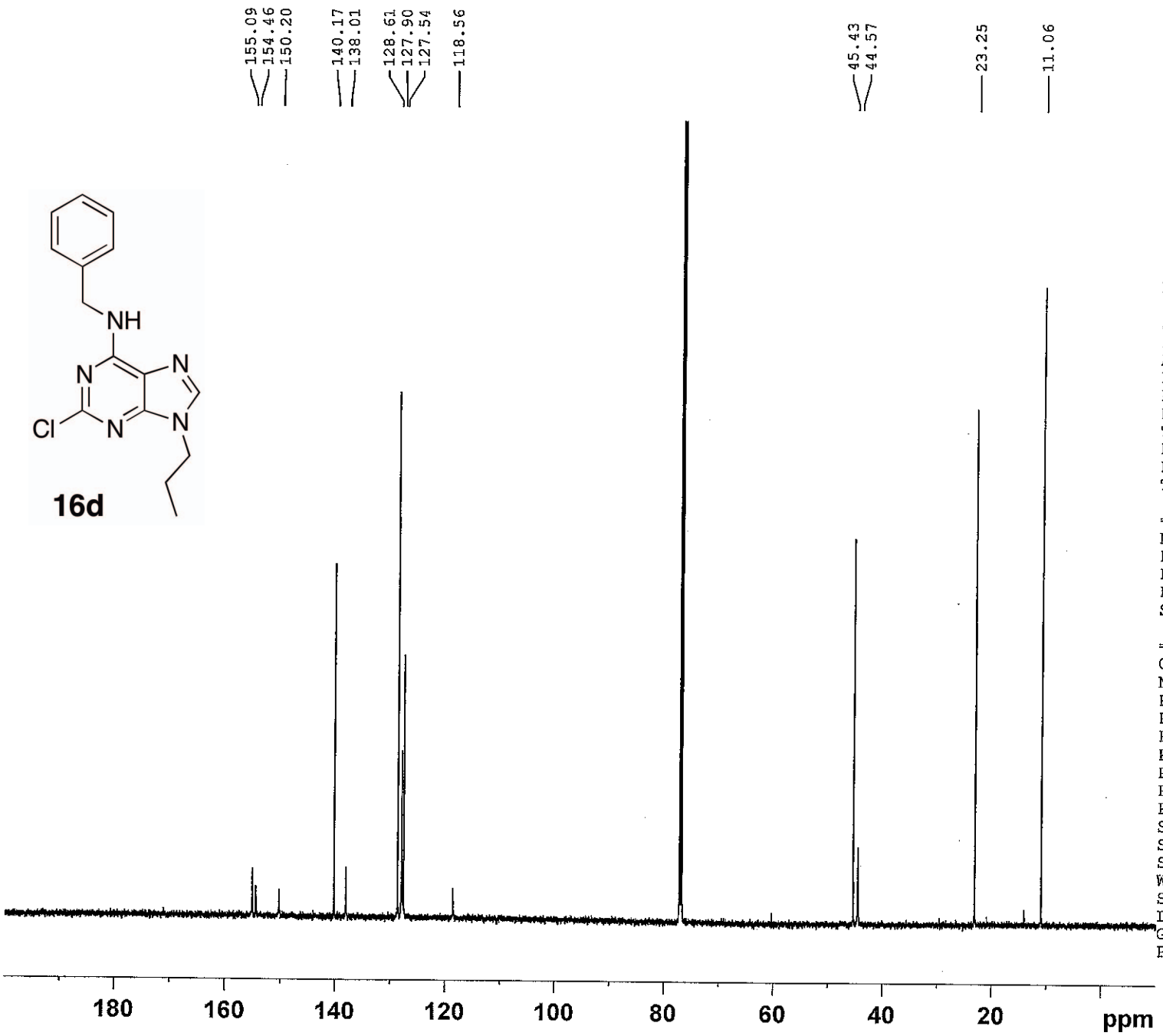


16d



NAME	kb-NB-213-05
EXPNO	1
PROCNO	1
Date_	20090722
Time	9.42
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	35.9
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300038 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

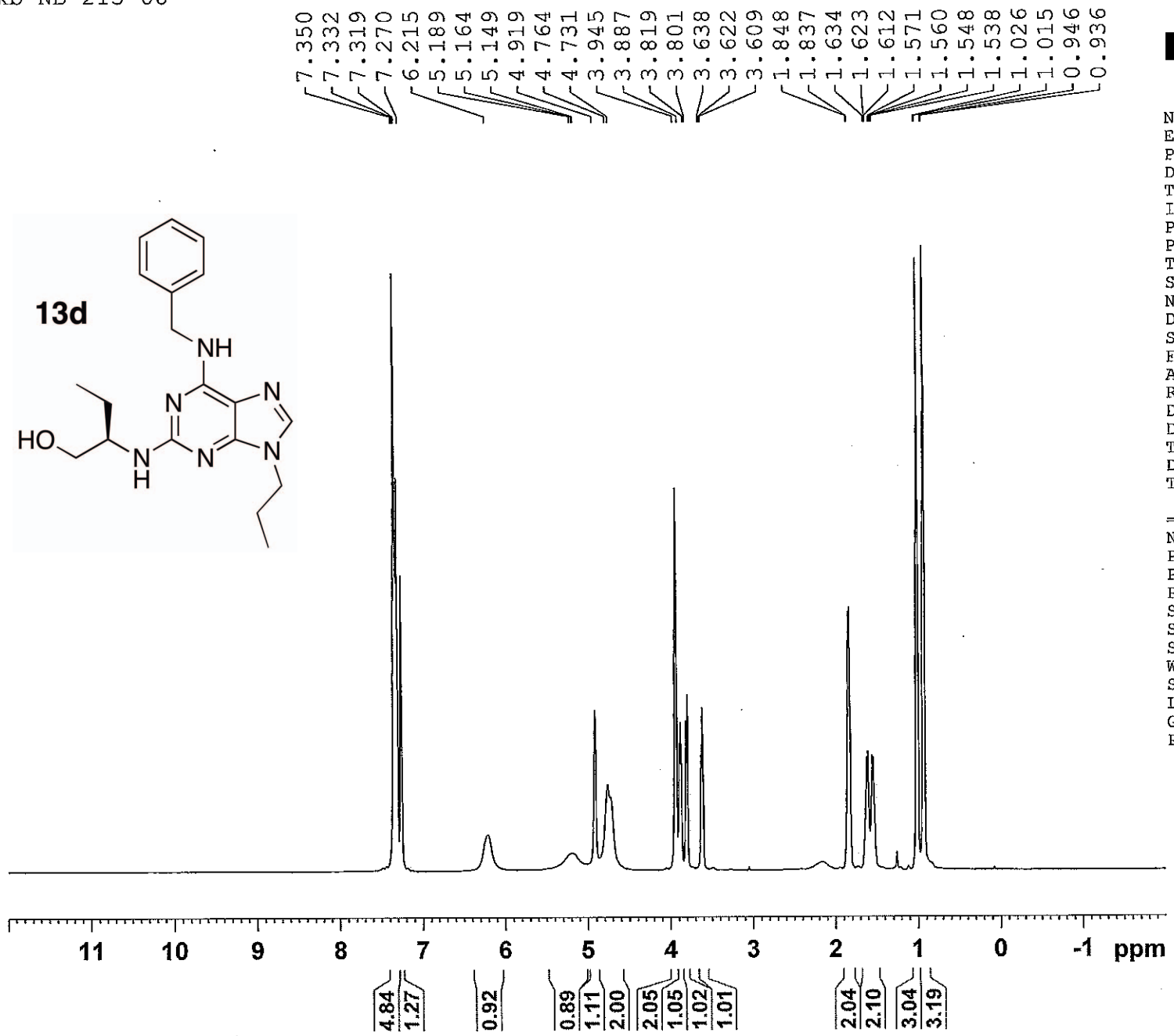
kb-NB-213-05



BRUKER

NAME	kb-NB-213-05
EXPNO	2
PROCNO	1
Date_	20090722
Time	9.58
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	16384
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028182 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

kb-NB-213-08



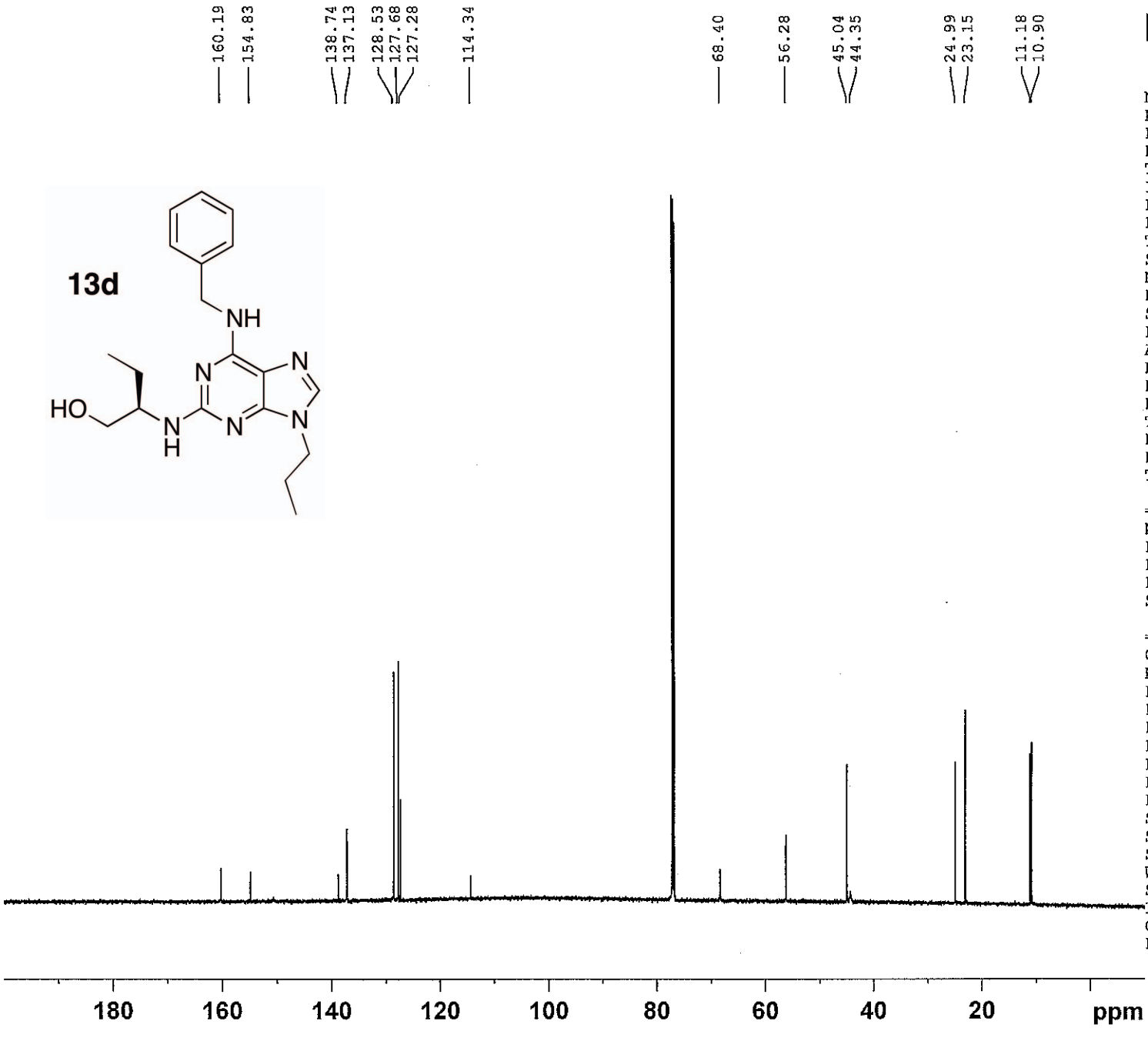
BRUKER

NAME	kb-NB-213-08
EXPNO	1
PROCNO	1
Date_	20090724
Time_	10.32
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	45.3
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1

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

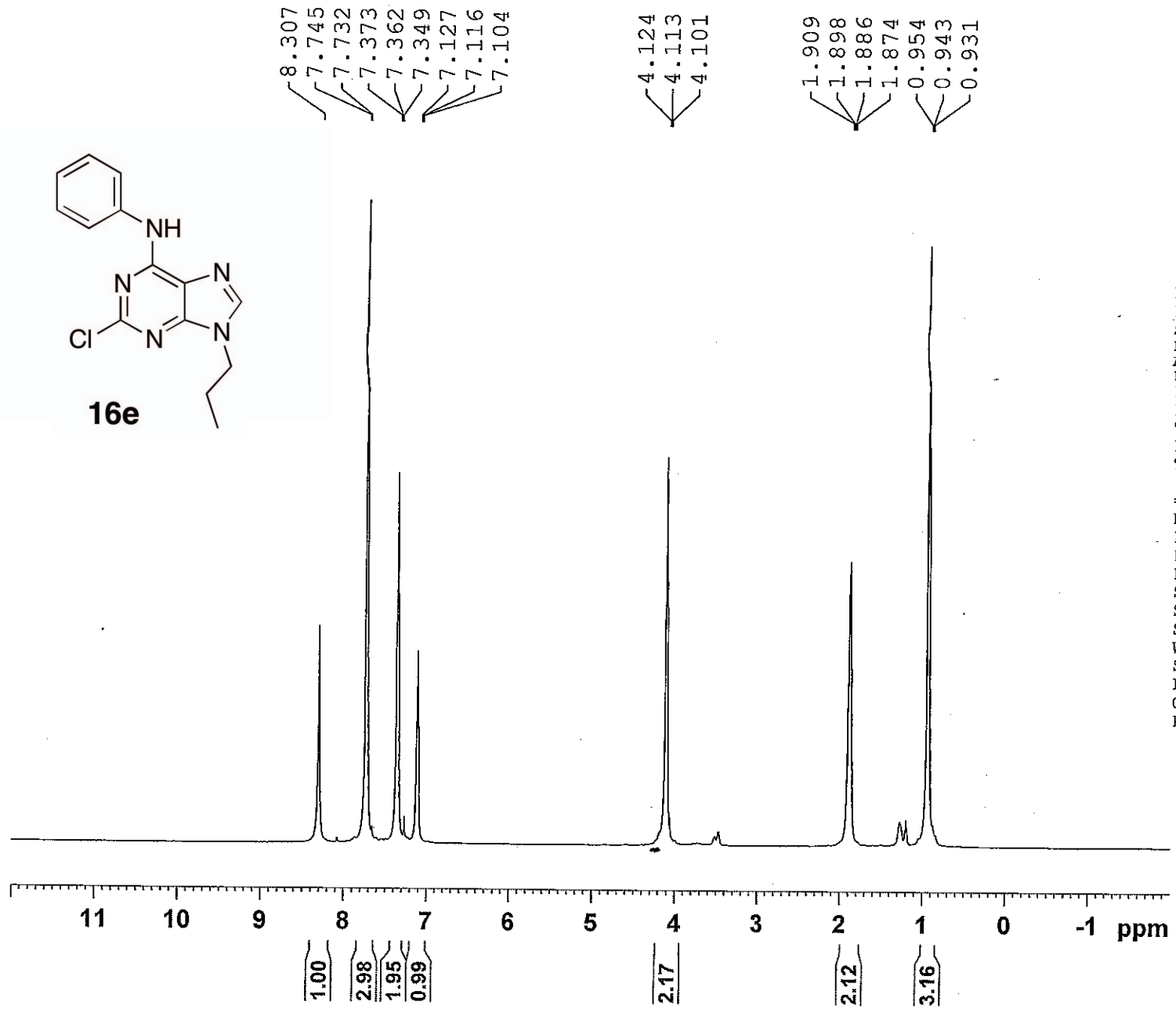
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300049 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

kb-NB-213-08



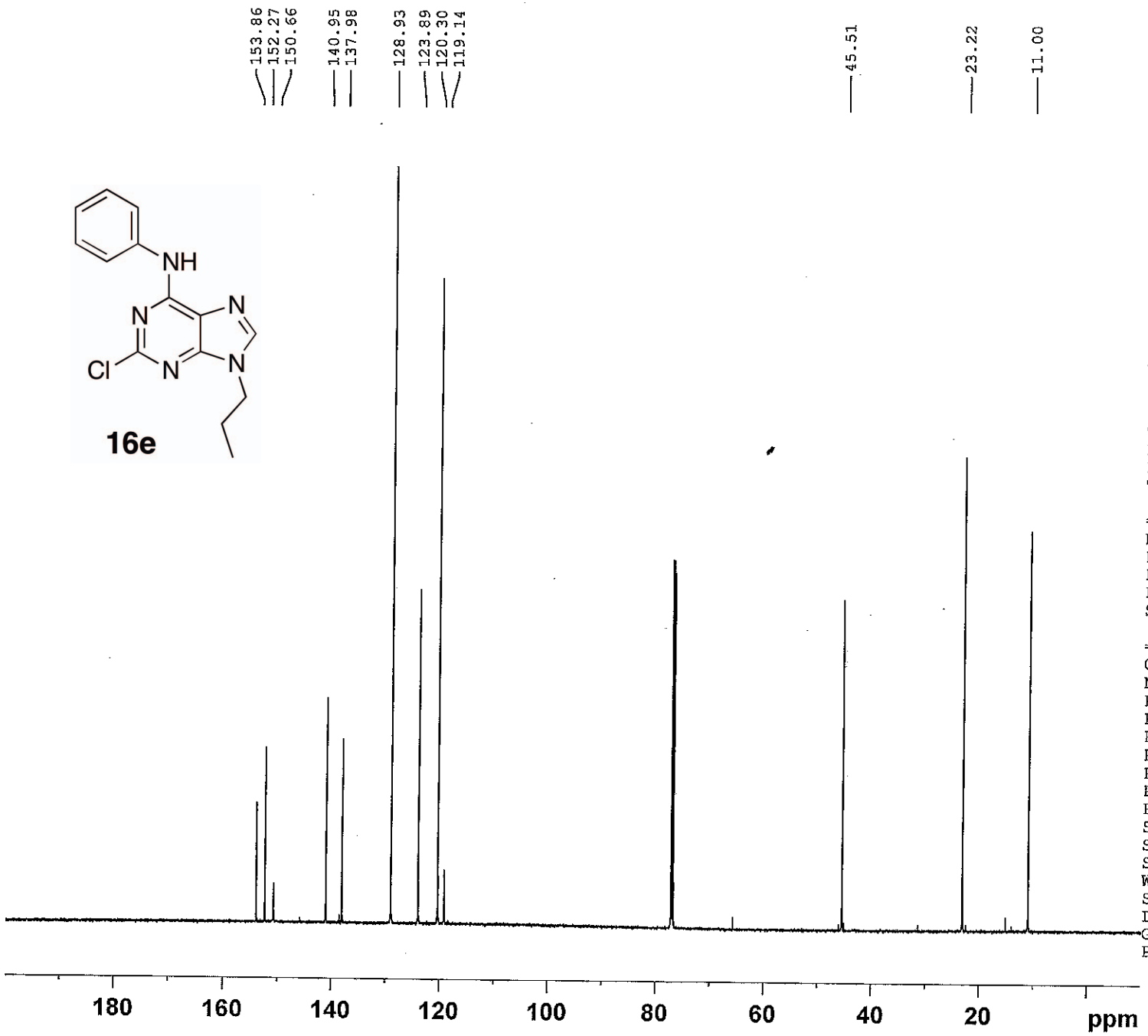
NAME	kb-NB-213-08
EXPNO	2
PROCNO	1
Date_	20090724
Time	10.48
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	2
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	11585.2
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028153 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

kb-NB-213-11



NAME	kb-NB-213-11
EXPNO	1
PROCNO	1
Date_	20090728
Time	13.51
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	18
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300045 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

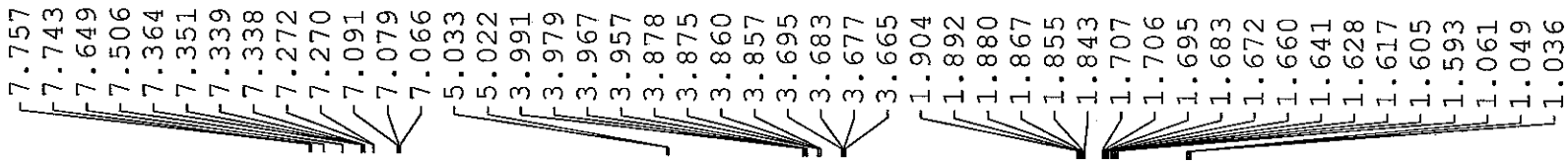
kb-NB-213-11

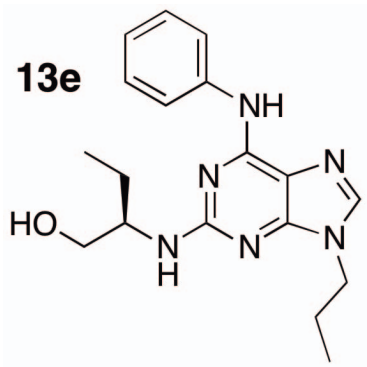


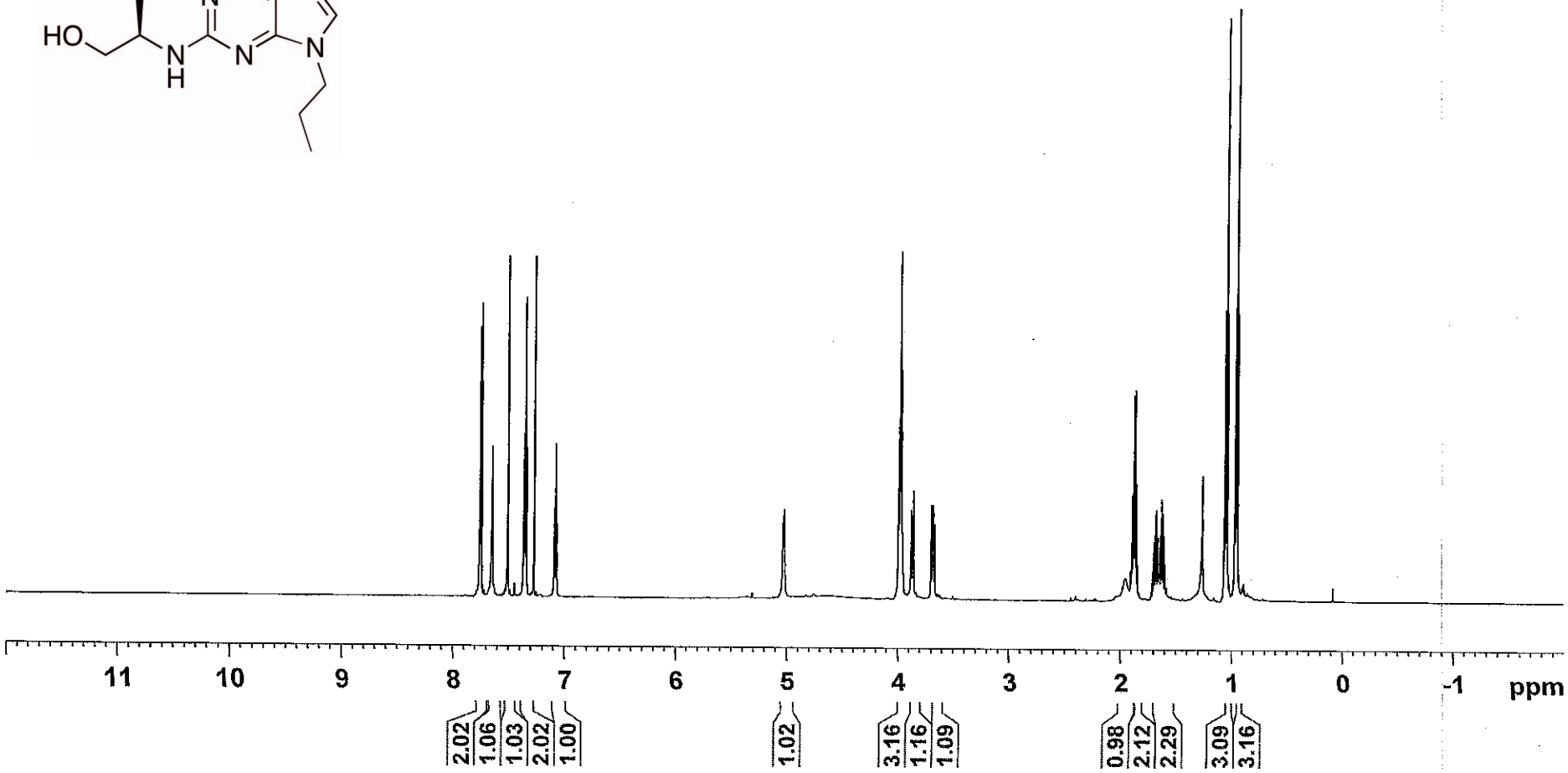
BRUKER

NAME	kb-NB-213-11
EXPNO	2
PROCNO	1
Date_	20090728
Time	14.04
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	192
DS	4
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	16384
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028285 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

kb-NB-213-13

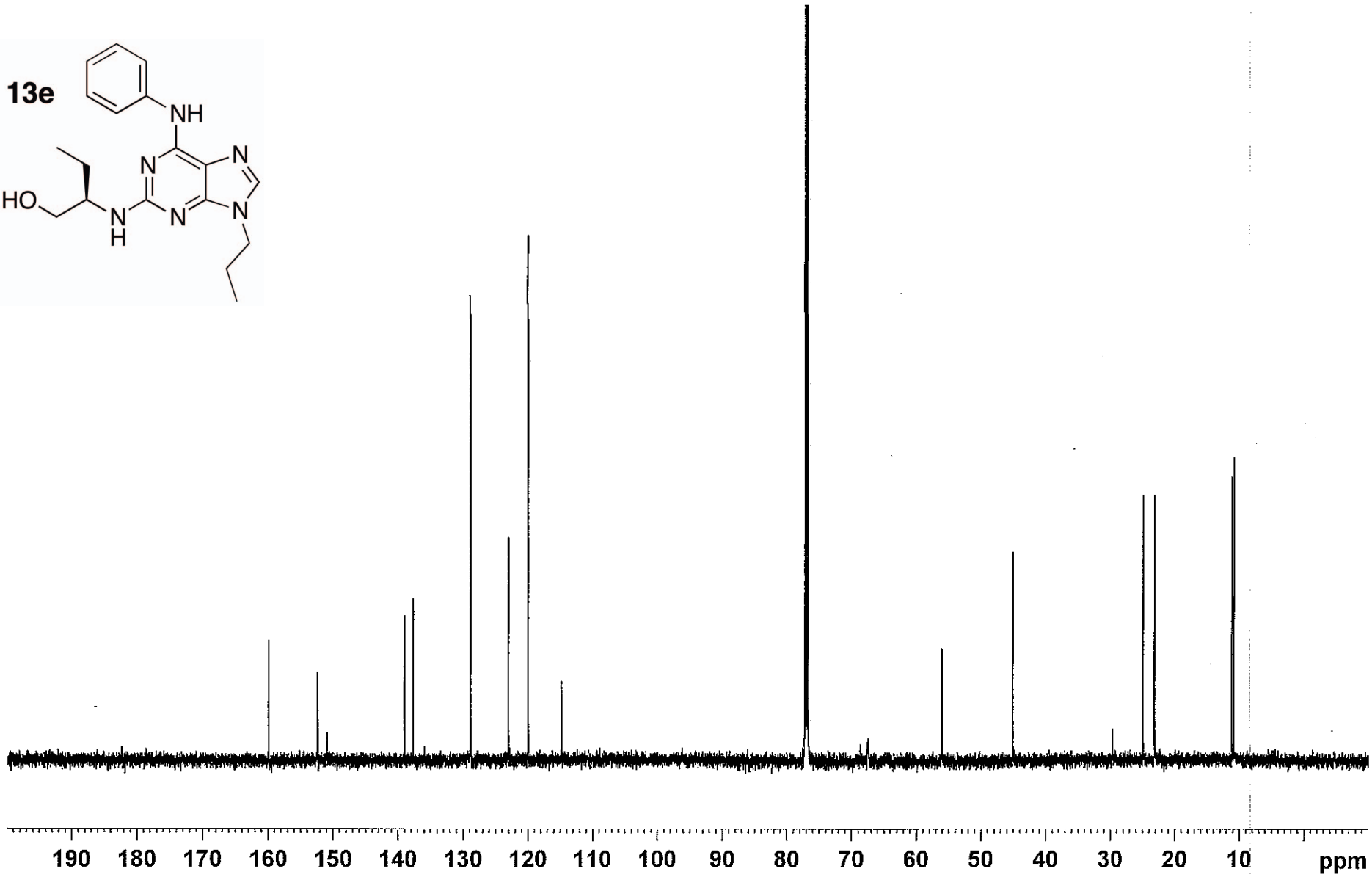

7.757
7.743
7.649
7.506
7.364
7.351
7.339
7.338
7.272
7.270
7.091
7.079
7.066
5.033
5.022
3.991
3.979
3.967
3.957
3.878
3.875
3.860
3.857
3.695
3.683
3.677
3.665
1.904
1.892
1.880
1.867
1.855
1.843
1.707
1.706
1.695
1.683
1.672
1.660
1.641
1.628
1.617
1.605
1.593
1.061
1.049
1.036


13e
NH
N
N
N
HO
N
H
N
N

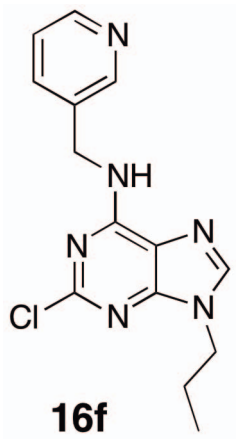

11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.02
1.06
1.03
2.02
1.00
1.02
3.16
1.16
1.09
0.98
2.12
2.29
3.09
3.16

kb-NB-213-13

159.85
152.36
150.93
138.97
137.64
128.85
123.04
120.01
114.80
56.14
45.13
29.68
24.97
23.18
11.21
10.91

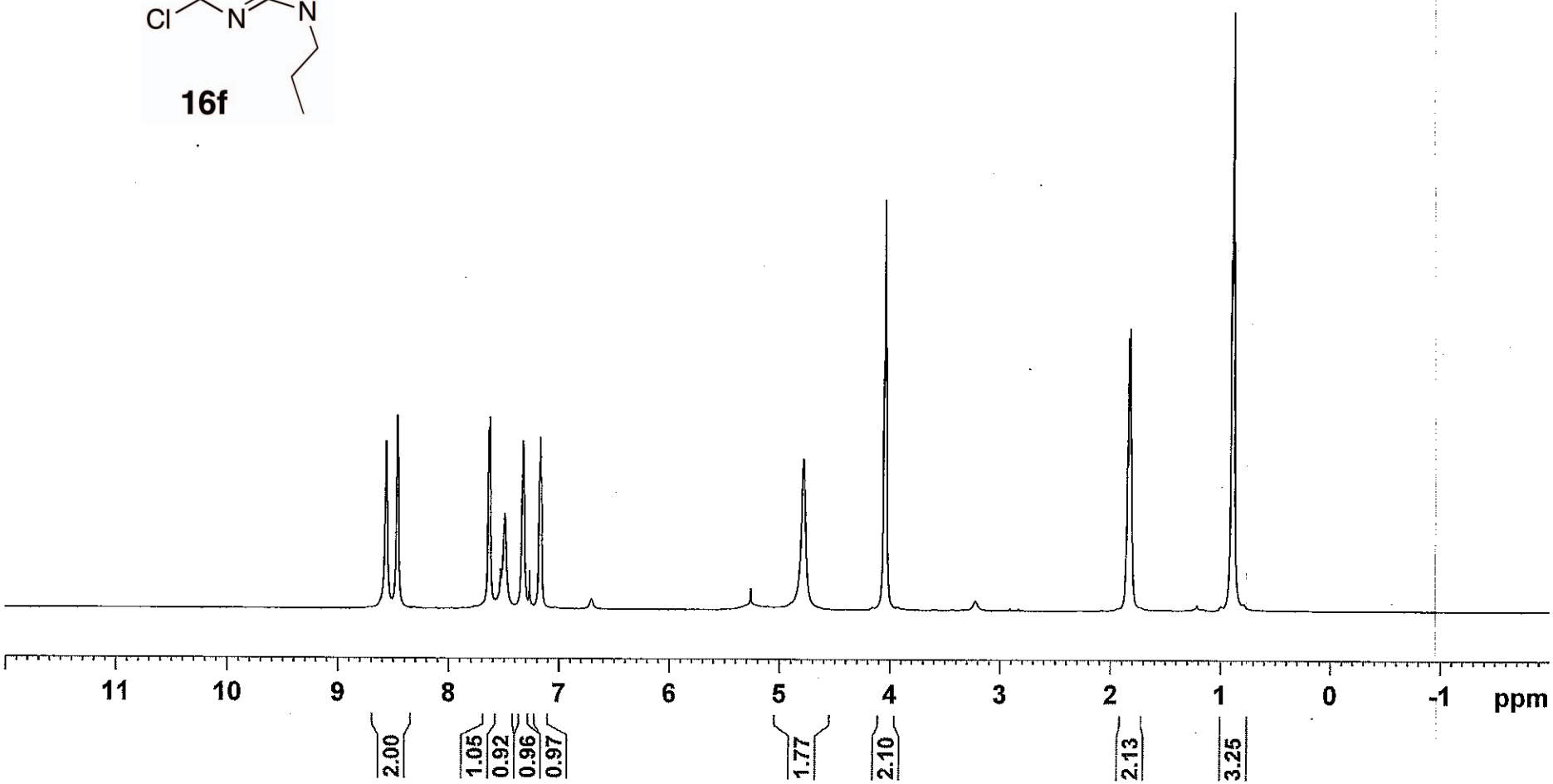


kb-NB-213-12

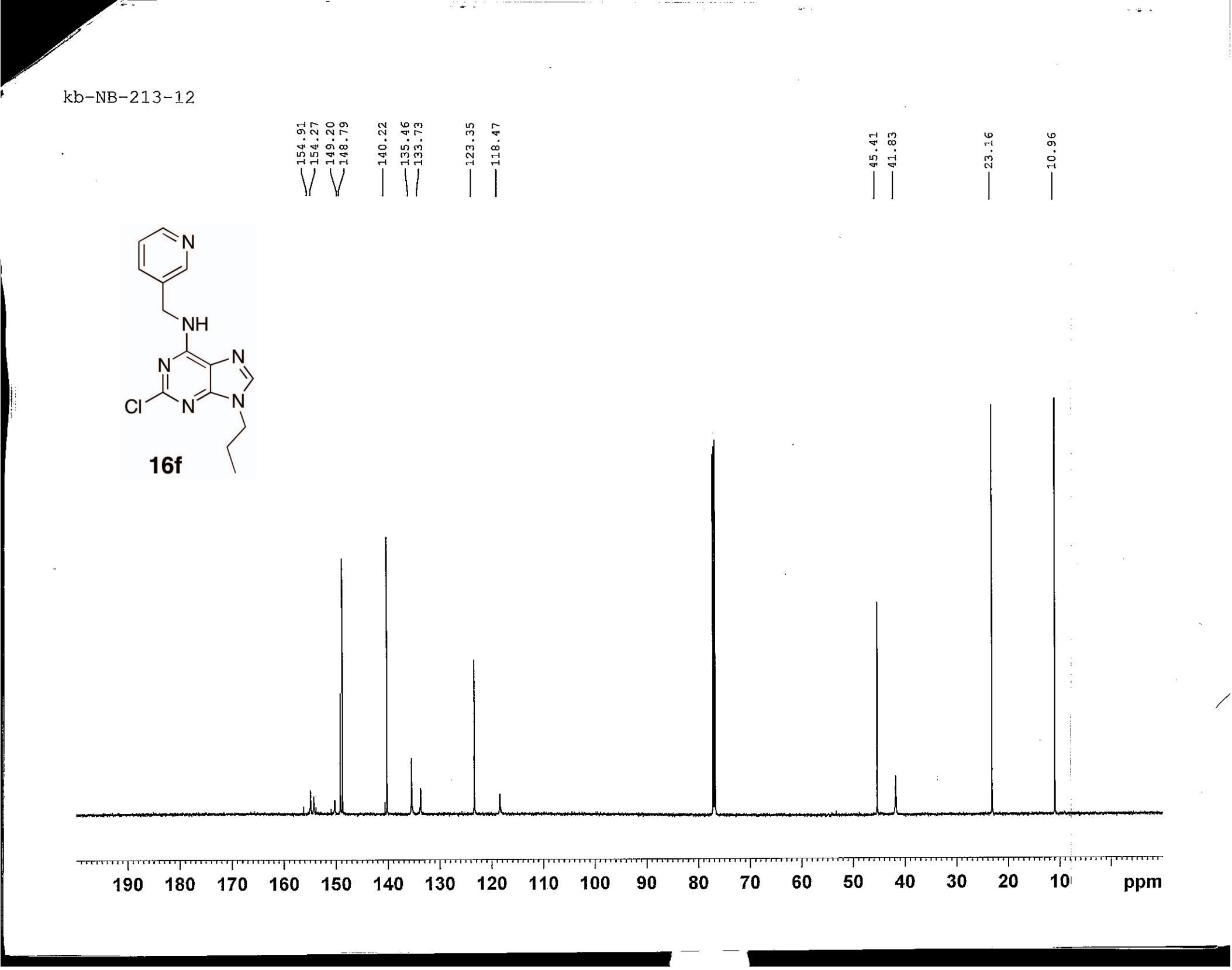


16f

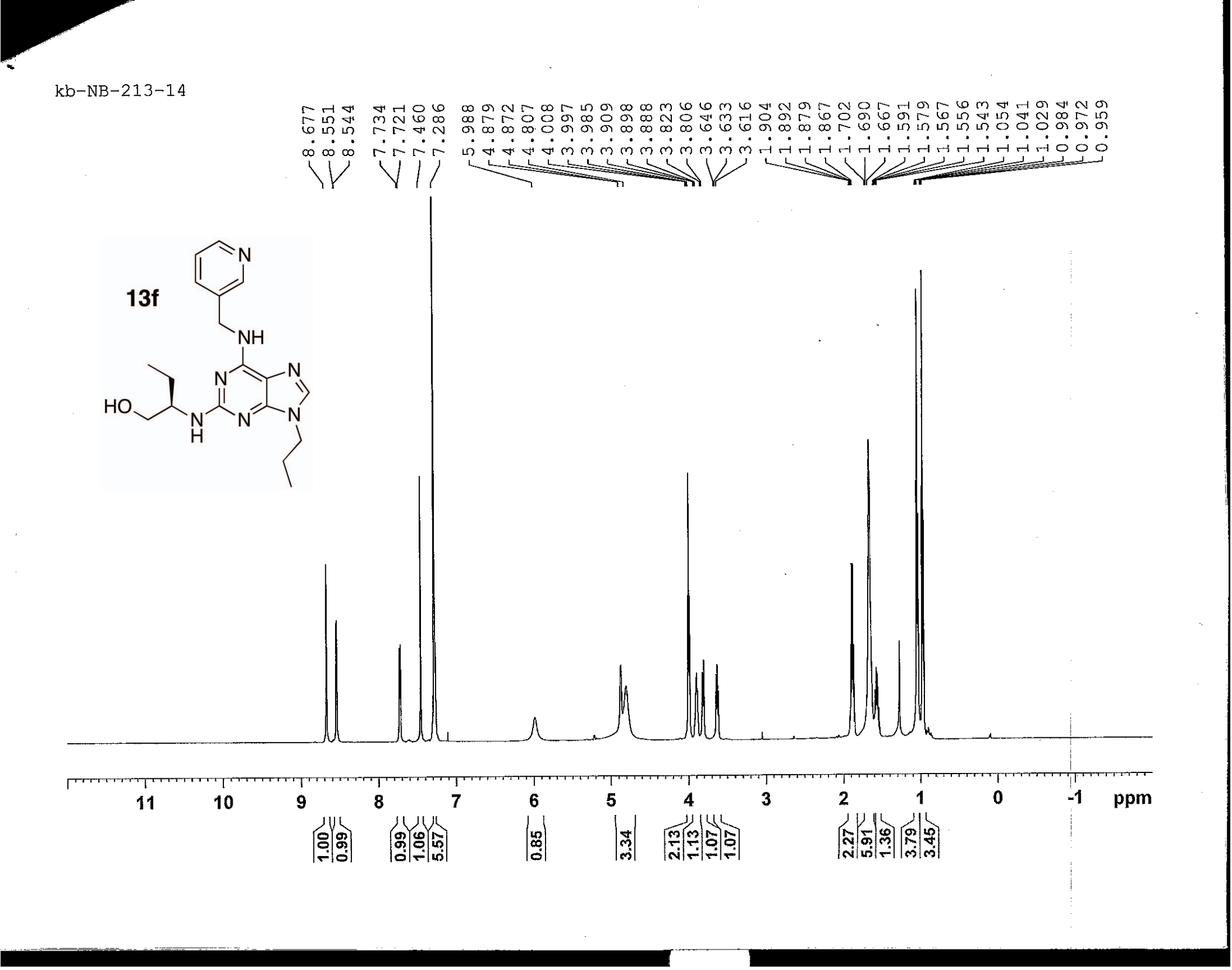
8.569
8.469
7.644
7.631
7.497
7.330
7.184
7.176
7.165
4.787
4.059
4.048
4.036
1.850
1.839
1.827
1.815
0.903
0.891
0.879



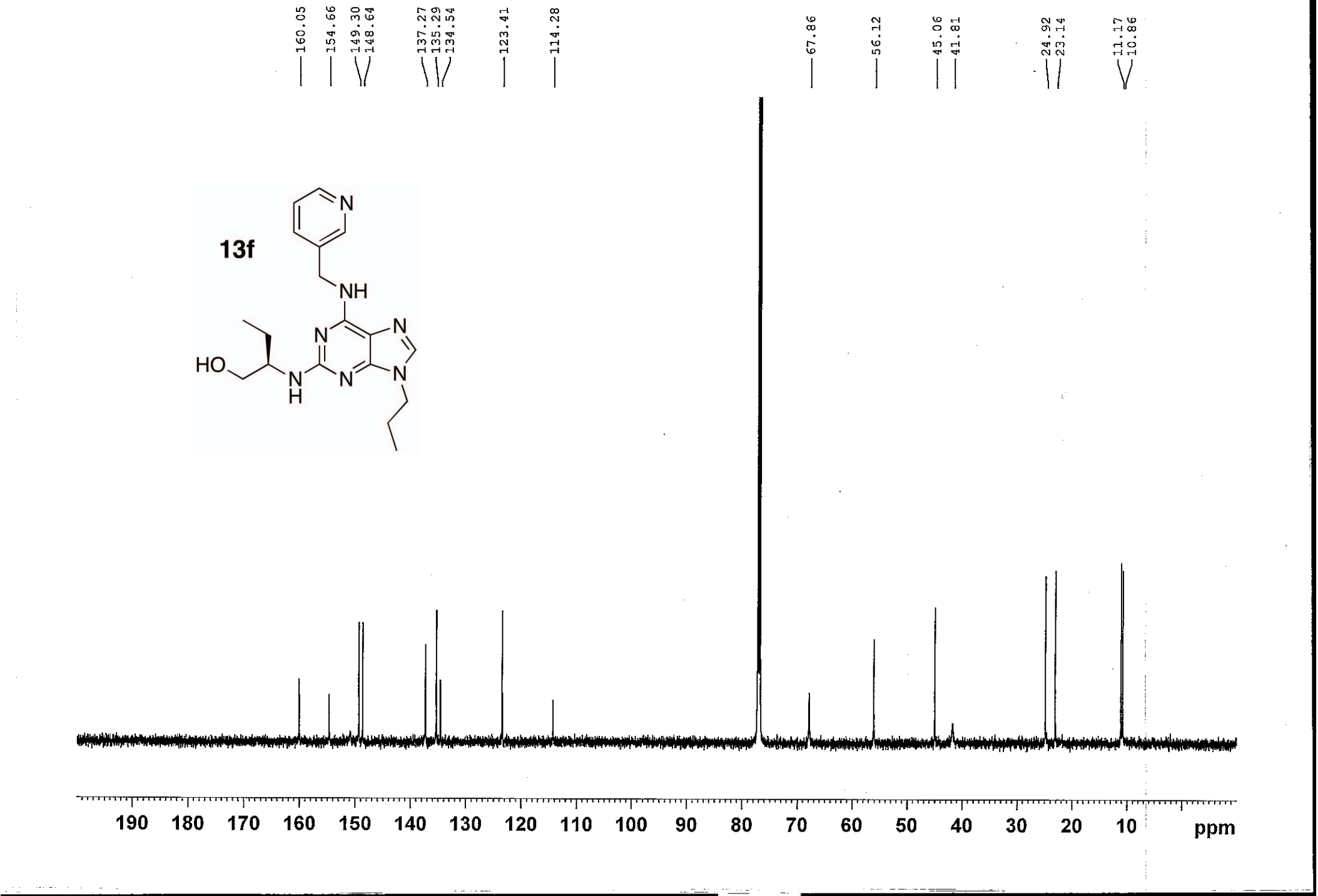
kb-NB-213-12


154.91
154.27
149.20
148.79
140.22
135.46
133.73
123.35
118.47
45.41
41.83
23.16
10.96
N
NH
N
N
N
Cl
N
N
16f
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

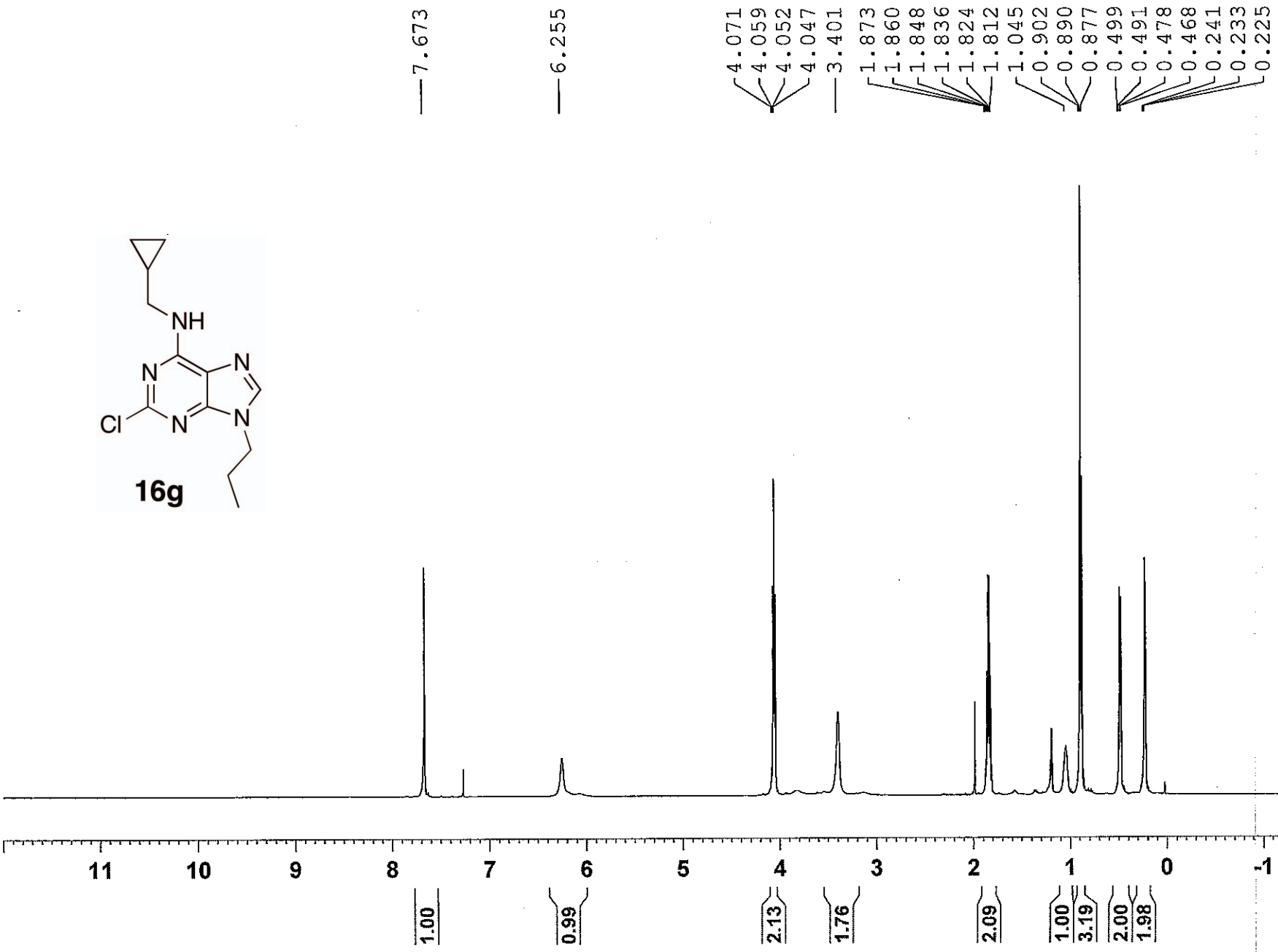
kb-NB-213-14


8.677
8.551
8.544
7.734
7.721
7.460
7.286
5.988
4.879
4.872
4.807
4.008
3.997
3.985
3.909
3.898
3.888
3.823
3.806
3.646
3.633
3.616
1.904
1.892
1.879
1.867
1.702
1.690
1.667
1.591
1.579
1.567
1.556
1.543
1.054
1.041
1.029
0.984
0.972
0.959
13f
HO
NH
N
N
N
N
N
N
H
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.00
0.99
0.99
1.06
5.57
0.85
3.34
2.13
1.13
1.07
1.07
2.27
5.91
1.36
3.79
3.45

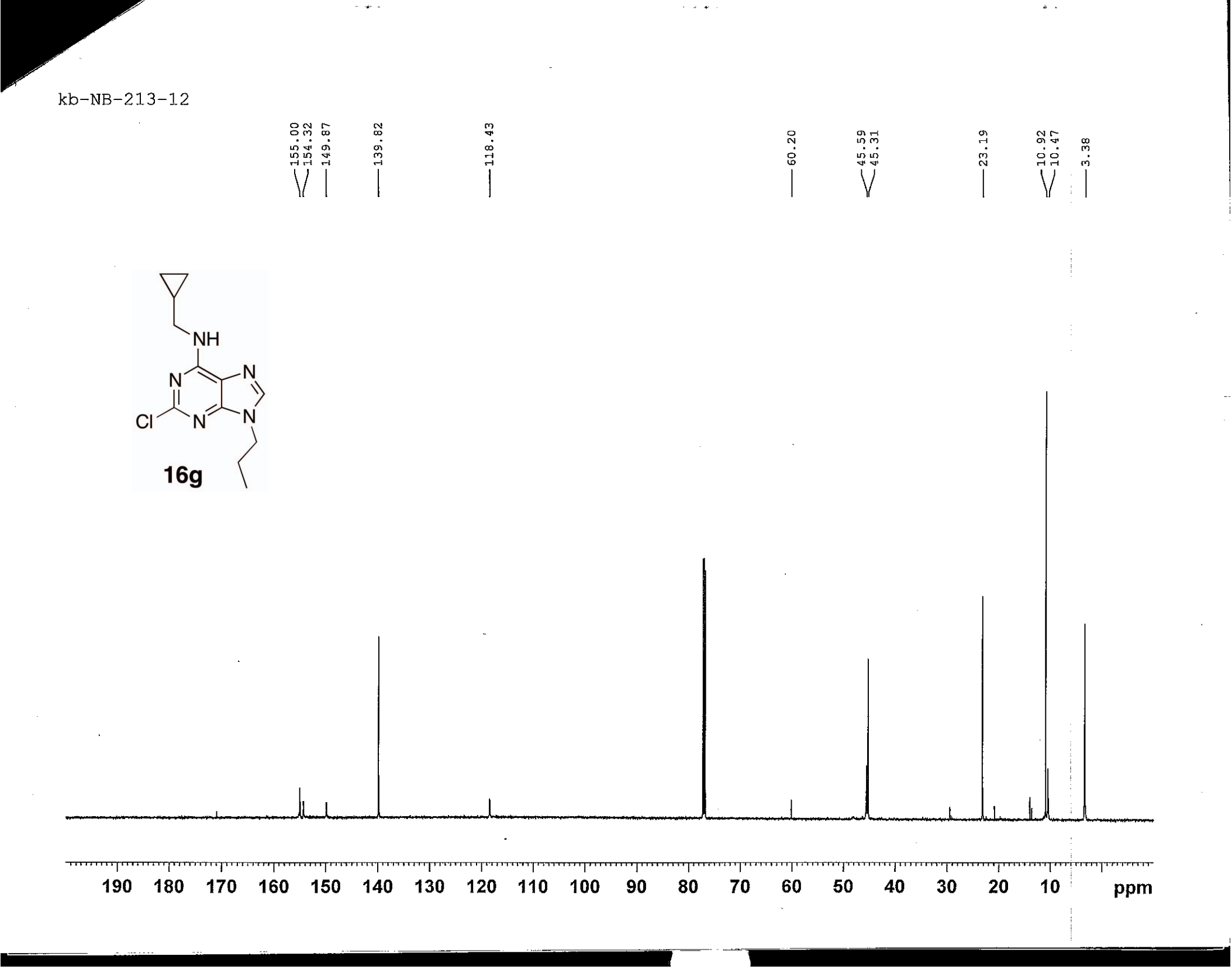
kb-NB-213-14


160.05
154.66
149.30
148.64
137.27
135.29
134.54
123.41
114.28
67.86
56.12
45.06
41.81
24.92
23.14
11.17
10.86
13f
N
NH
N
N
N
HO
N
H
N
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

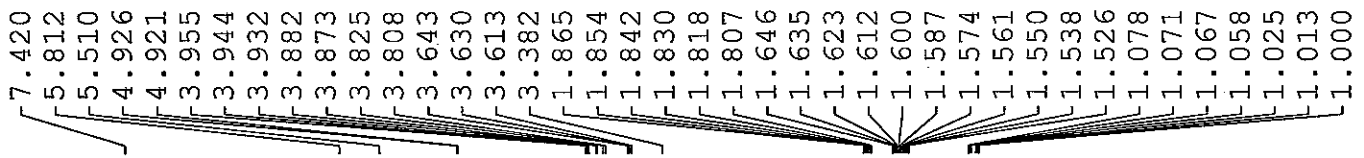
kb-NB-213-12

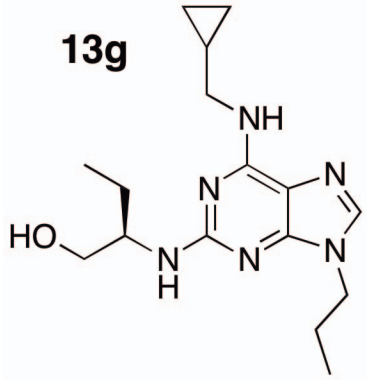

7.673
6.255
4.071
4.059
4.052
4.047
3.401
1.873
1.860
1.848
1.836
1.824
1.812
1.045
0.902
0.890
0.877
0.499
0.491
0.478
0.468
0.241
0.233
0.225
NH
N
N
Cl
N
N
16g
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.00
0.99
2.13
1.76
2.09
1.00
3.19
2.00
1.98

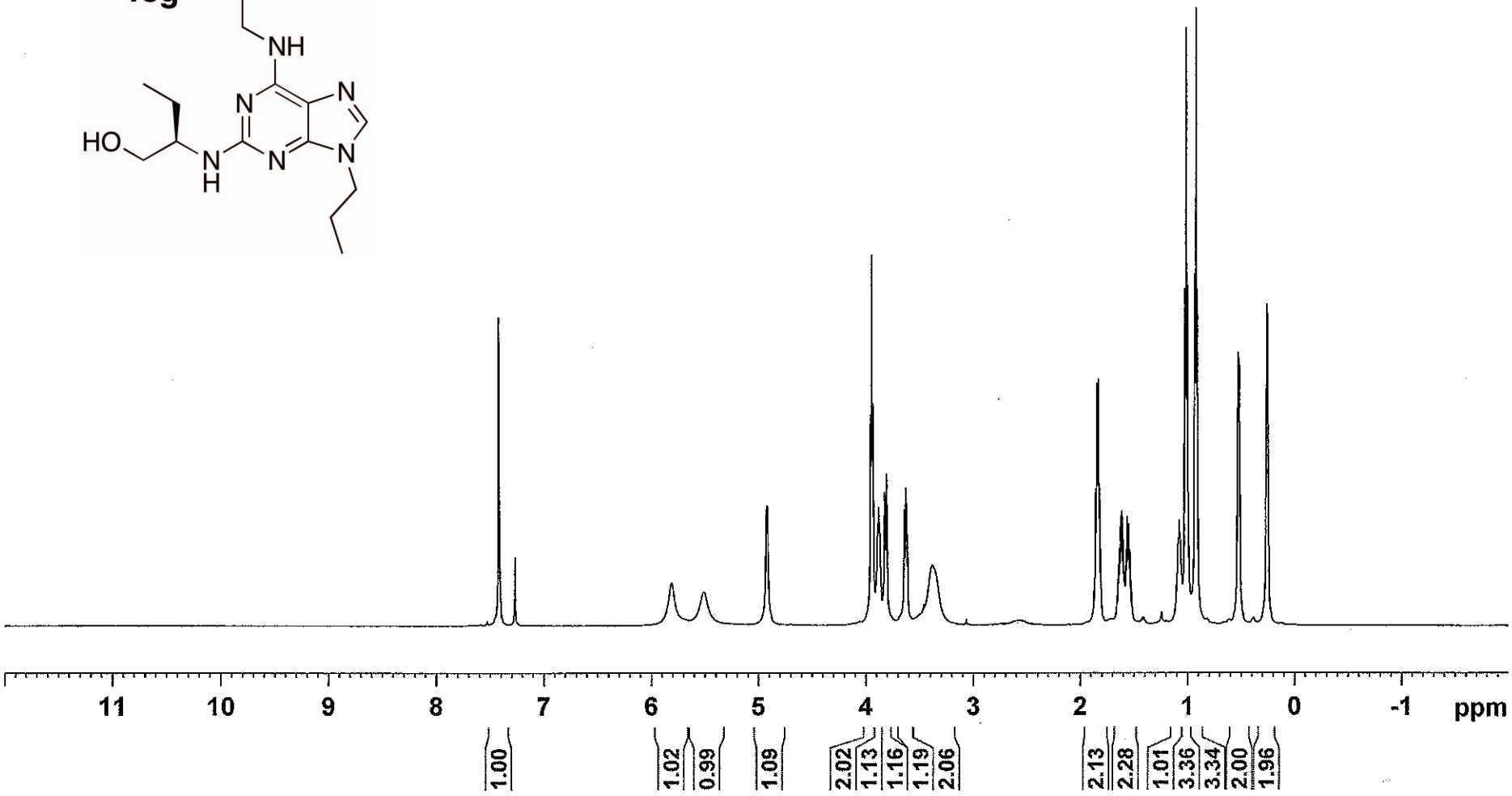
kb-NB-213-12


155.00
154.32
149.87
139.82
118.43
60.20
45.59
45.31
23.19
10.92
10.47
3.38
NH
N
N
N
N
Cl
16g
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

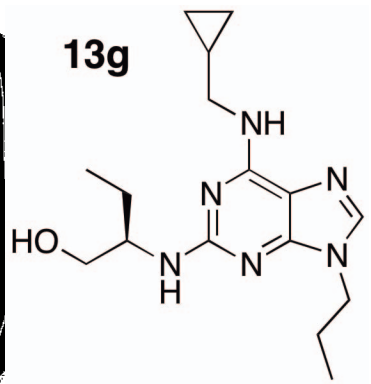
kb-NB-213-16


7.420
5.812
5.510
4.926
4.921
3.955
3.944
3.932
3.882
3.873
3.825
3.808
3.643
3.630
3.613
3.382
1.865
1.854
1.842
1.830
1.818
1.807
1.646
1.635
1.623
1.612
1.600
1.587
1.574
1.561
1.550
1.538
1.526
1.078
1.071
1.067
1.058
1.025
1.013
1.000


13g
NH
N
N
N
N
HO
N
H


11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.00
1.02
0.99
1.09
2.02
1.13
1.16
1.19
2.06
2.13
2.28
1.01
3.36
3.34
2.00
1.96

kb-NB-213-16

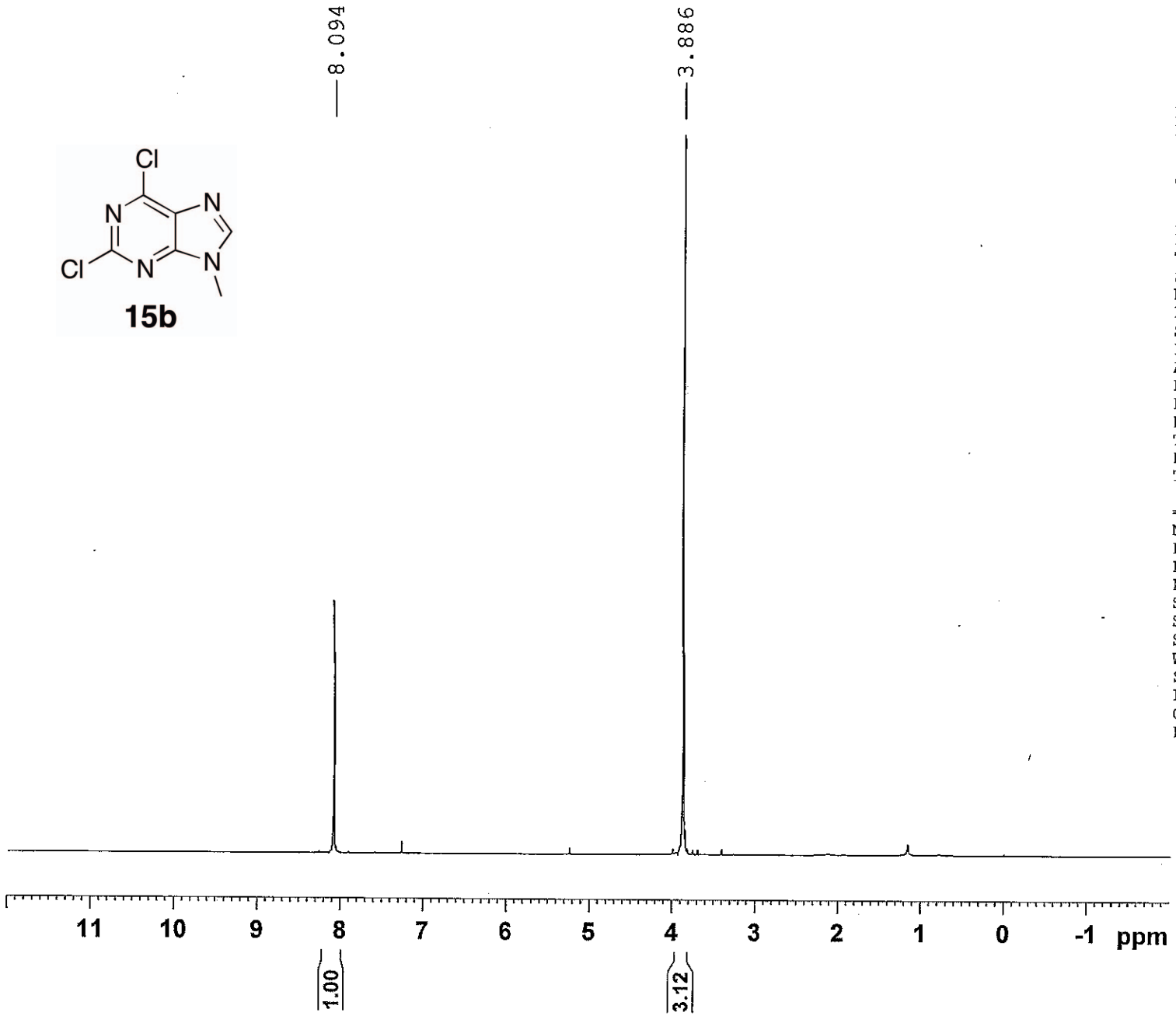


160.17
154.82
150.54
136.92
114.22
68.39
56.28
45.39
44.98
24.99
23.14
11.13
10.89
10.76
3.43

190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

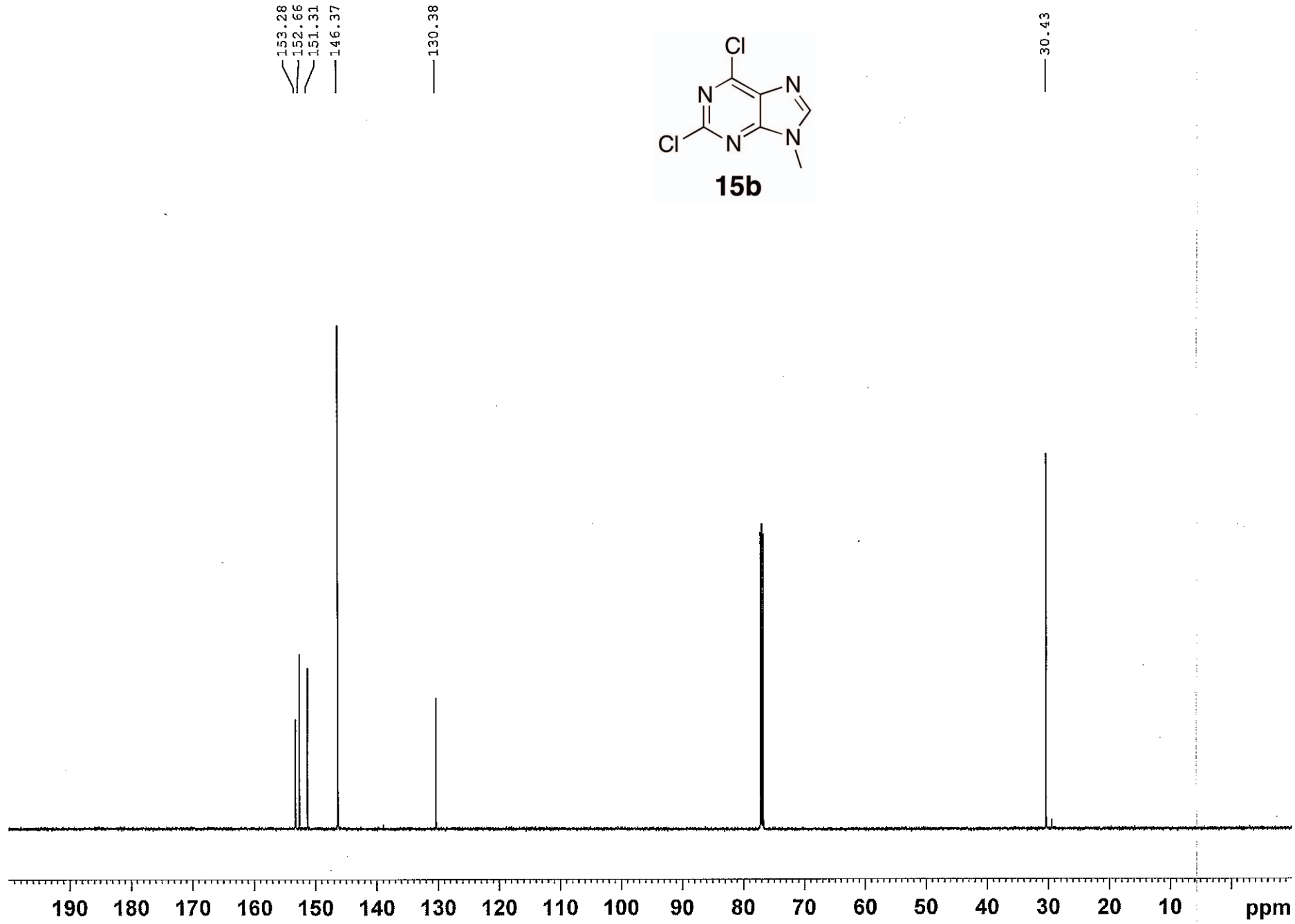
kb-NB-213-09-1

15b

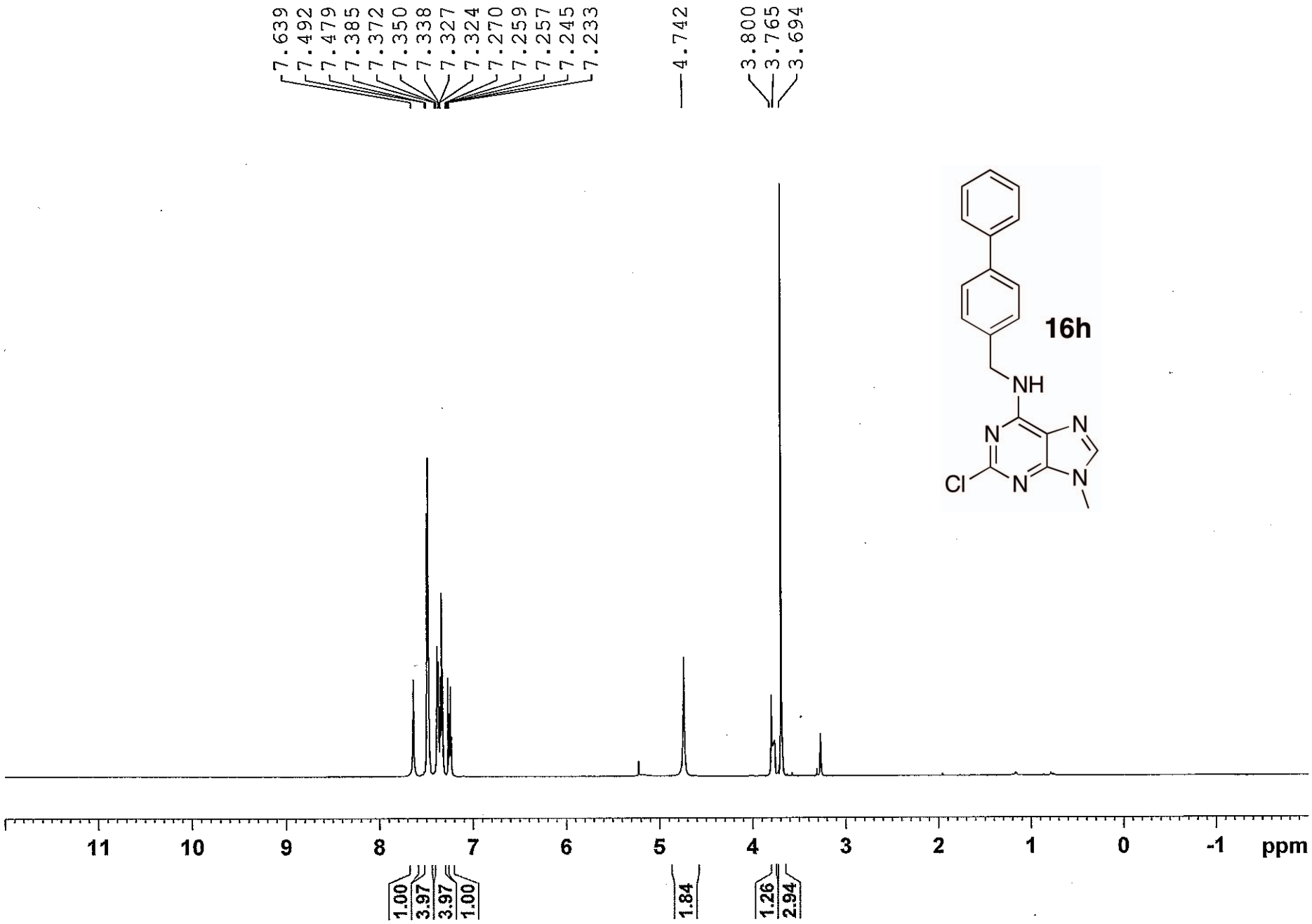


NAME	kb-NB-213-09-1	
EXPNO	1	
PROCNO	1	
Date_	20090724	
Time	20.15	
INSTRUM	spect	
PROBHD	5 mm CPTCI 1H-	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	12376.237	Hz
FIDRES	0.188846	Hz
AQ	2.6477449	sec
RG	28.5	
DW	40.400	usec
DE	6.50	usec
TE	298.0	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	8.00	usec
PL1	4.00	dB
PL1W	7.00000000	W
SFO1	600.1337060	MHz
SI	32768	
SF	600.1300042	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

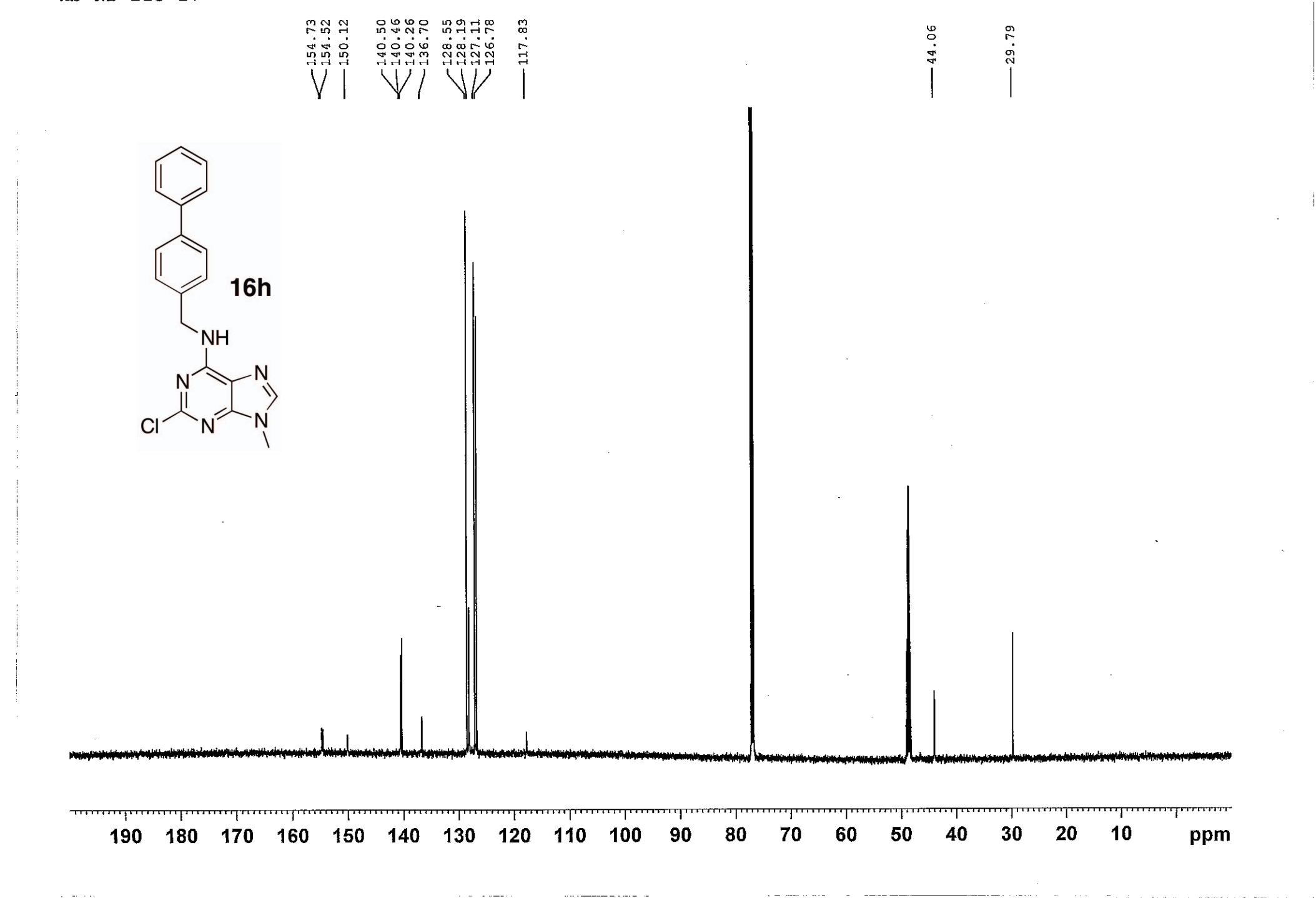
kb-NB-213-09-1


153.28
152.66
151.31
146.37
130.38
30.43
Cl
N
N
Cl
N
N
15b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

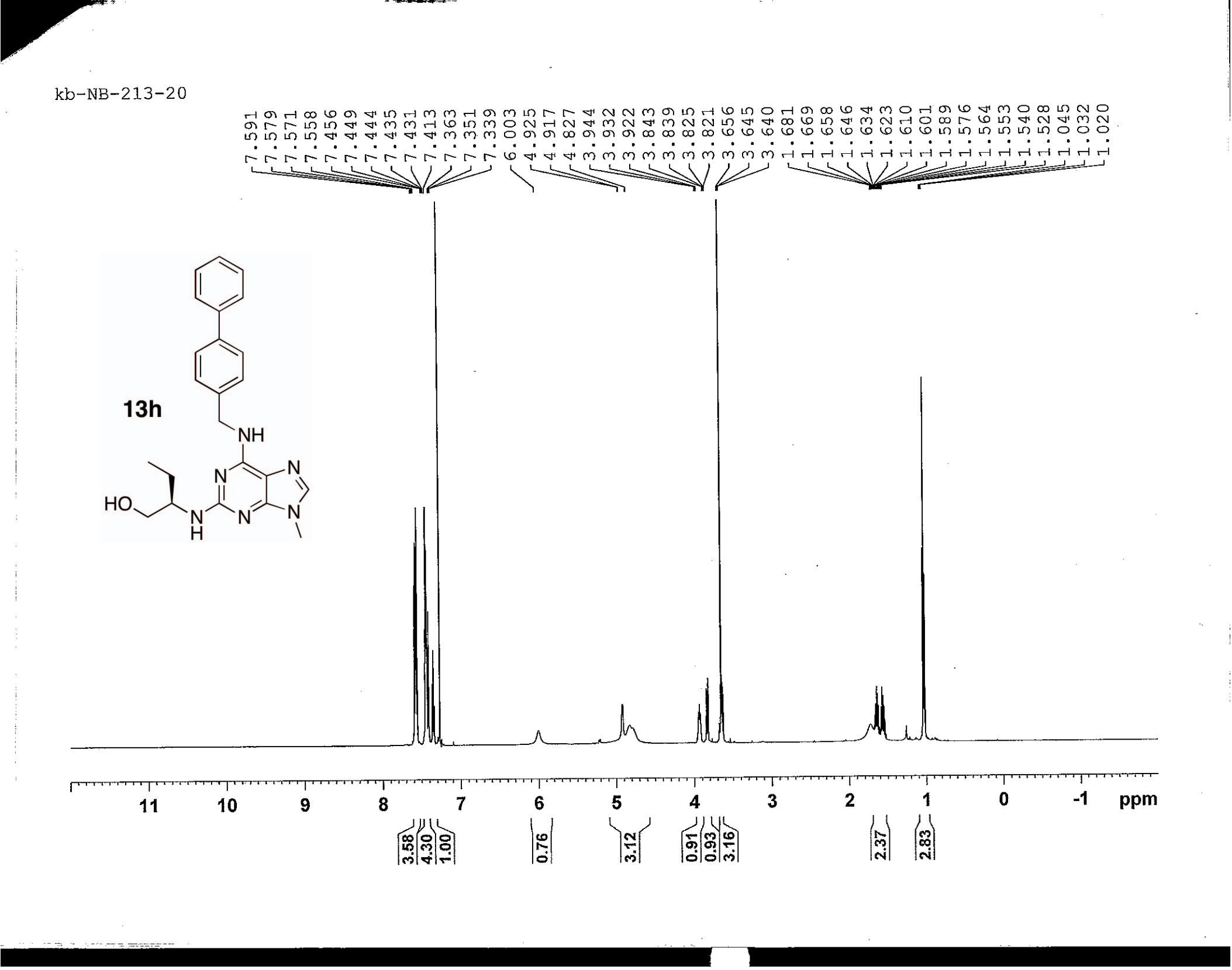
kb-NB-213-17


7.639
7.492
7.479
7.385
7.372
7.350
7.338
7.327
7.324
7.270
7.259
7.257
7.245
7.233
4.742
3.800
3.765
3.694
16h
NH
N
N
N
N
Cl
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.00
3.97
3.97
1.00
1.84
1.26
2.94

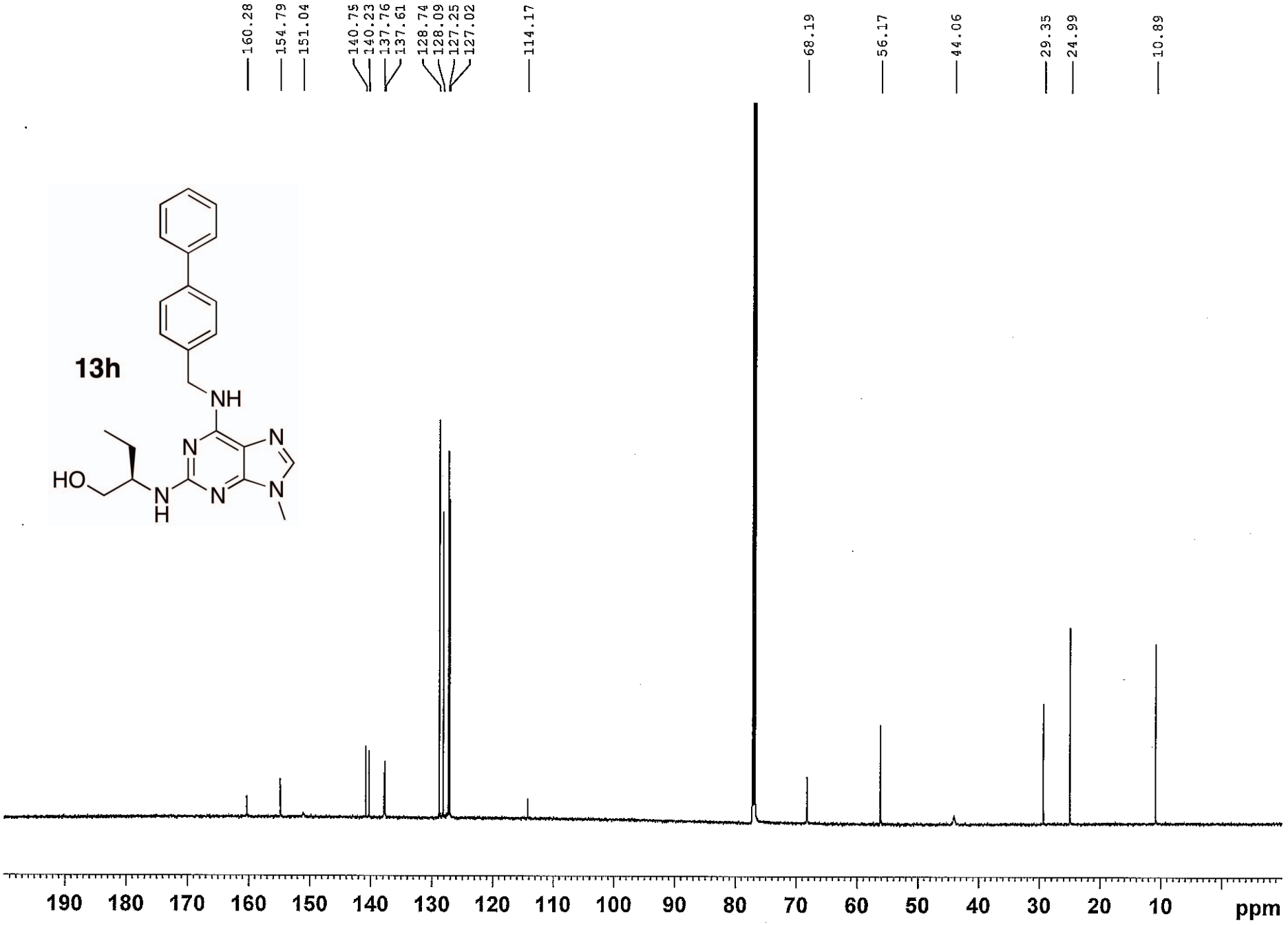
kb-NB-213-17


154.73
154.52
150.12
140.50
140.46
140.26
136.70
128.55
128.19
127.11
126.78
117.83
44.06
29.79
16h
NH
N
N
N
N
Cl
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

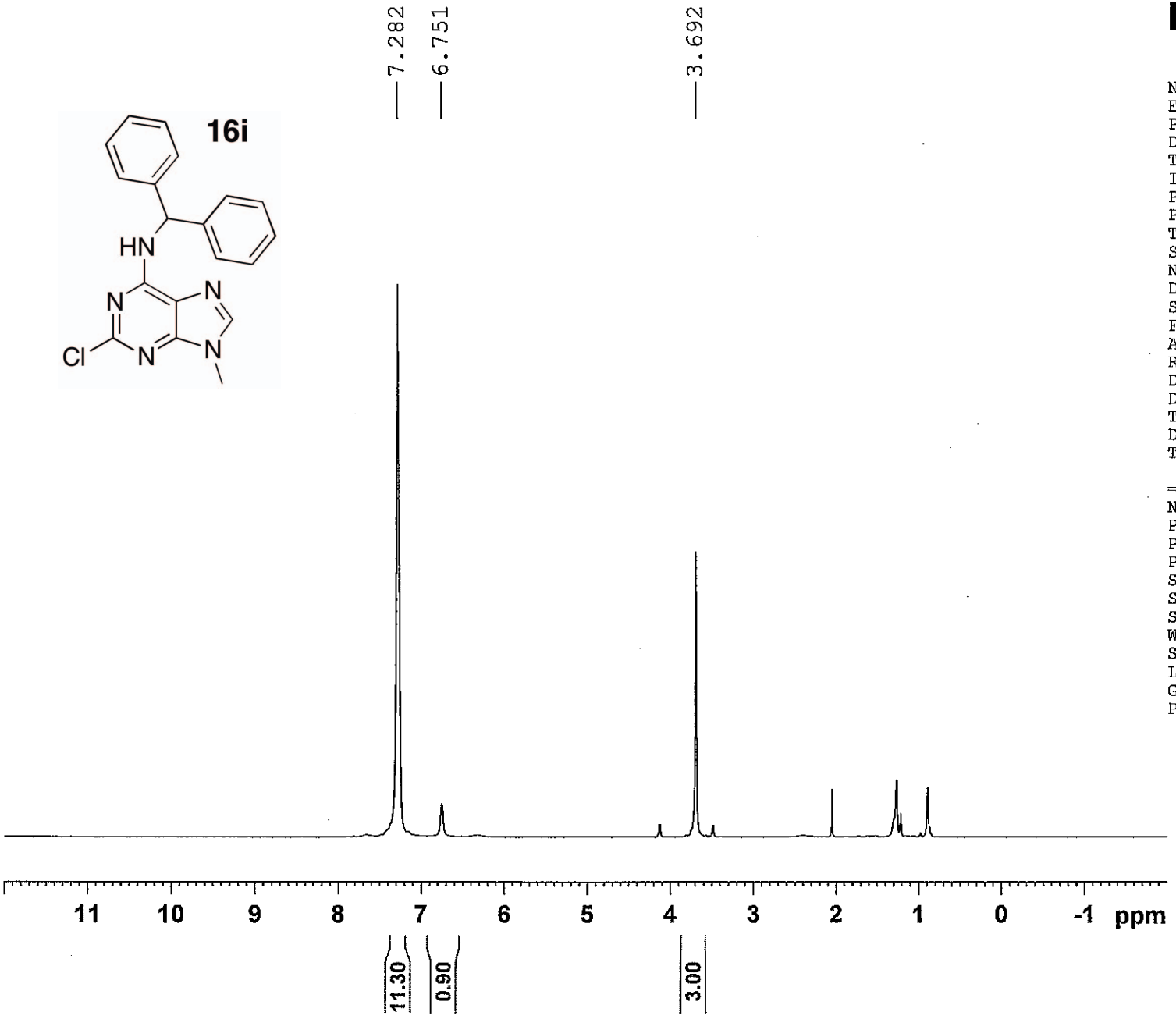
kb-NB-213-20


7.591
7.579
7.571
7.558
7.456
7.449
7.444
7.435
7.431
7.413
7.363
7.351
7.339
6.003
4.925
4.917
4.827
3.944
3.932
3.922
3.843
3.839
3.825
3.821
3.656
3.645
3.640
1.681
1.669
1.658
1.646
1.634
1.623
1.610
1.601
1.589
1.576
1.564
1.553
1.540
1.528
1.045
1.032
1.020
13h
NH
N
N
N
HO
N
H
N
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
3.58
4.30
1.00
0.76
3.12
0.91
0.93
3.16
2.37
2.83

kb-NB-213-20


160.28
154.79
151.04
140.75
140.23
137.76
137.61
128.74
128.09
127.25
127.02
114.17
68.19
56.17
44.06
29.35
24.99
10.89
13h
NH
N
N
N
N
HO
N
H
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

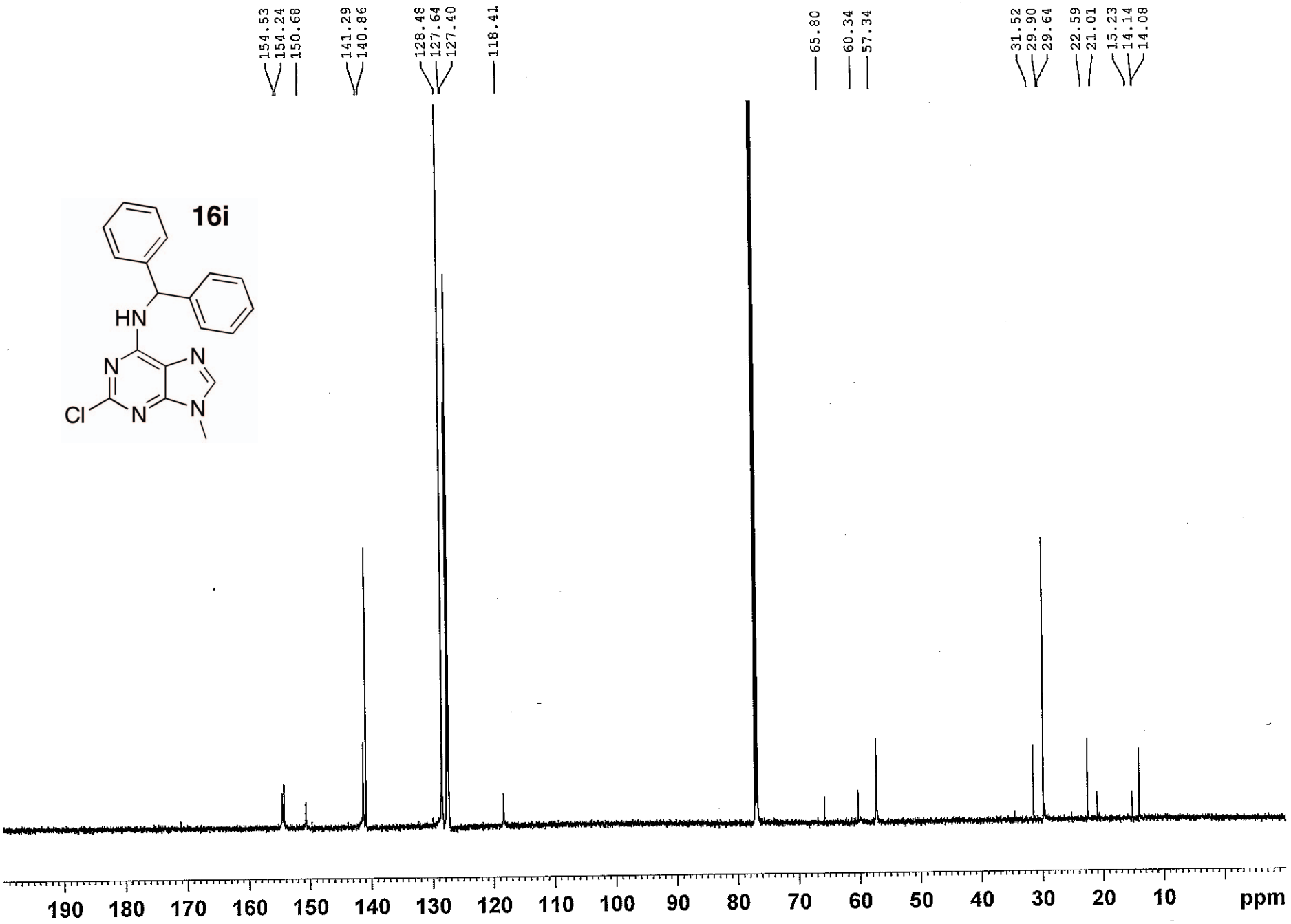
kb-NB-213-18 (calibrated)



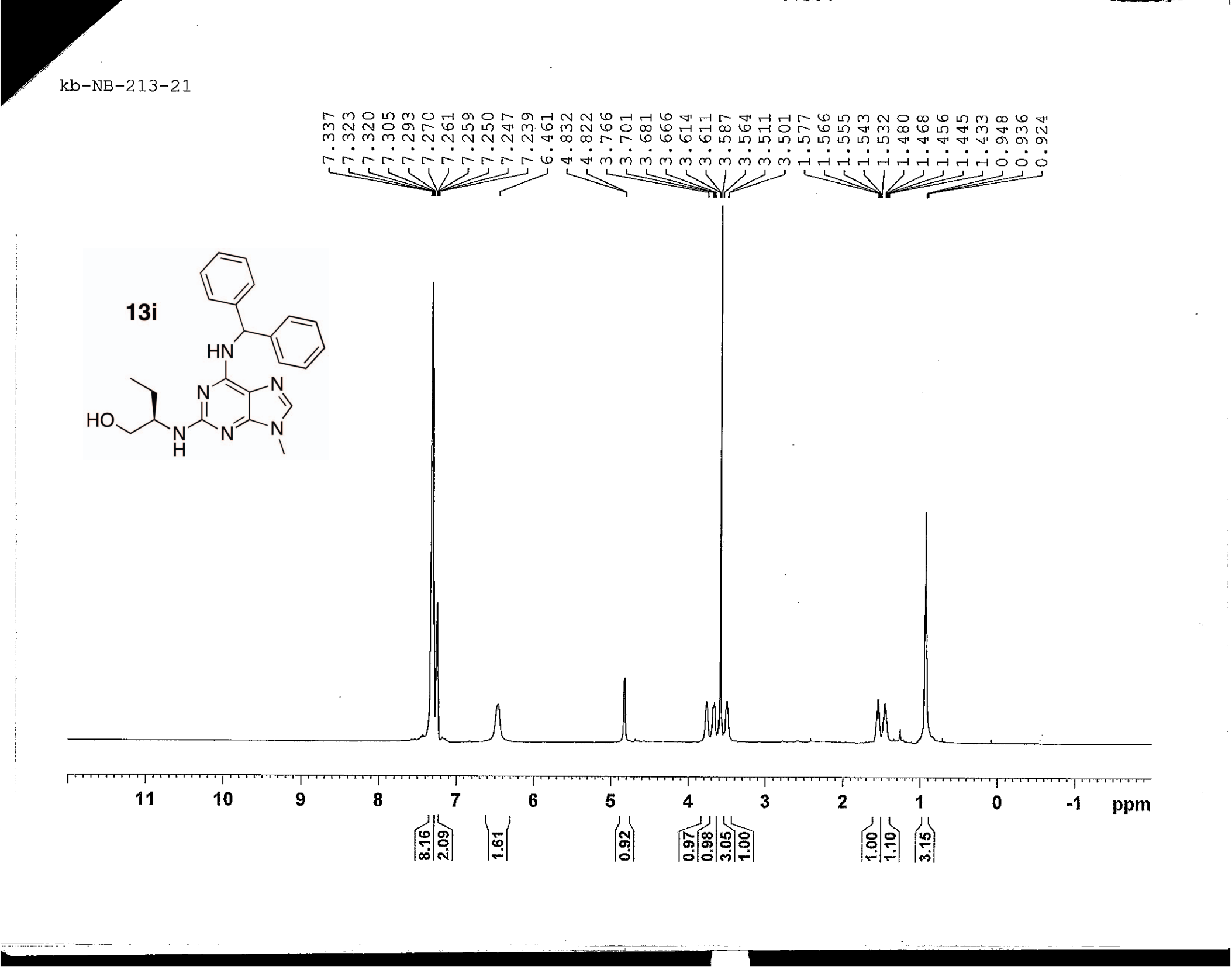
BRUKER

NAME	kb-NB-213-18
EXPNO	1
PROCNO	1
Date_	20090731
Time	14.00
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	25.4
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300042 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

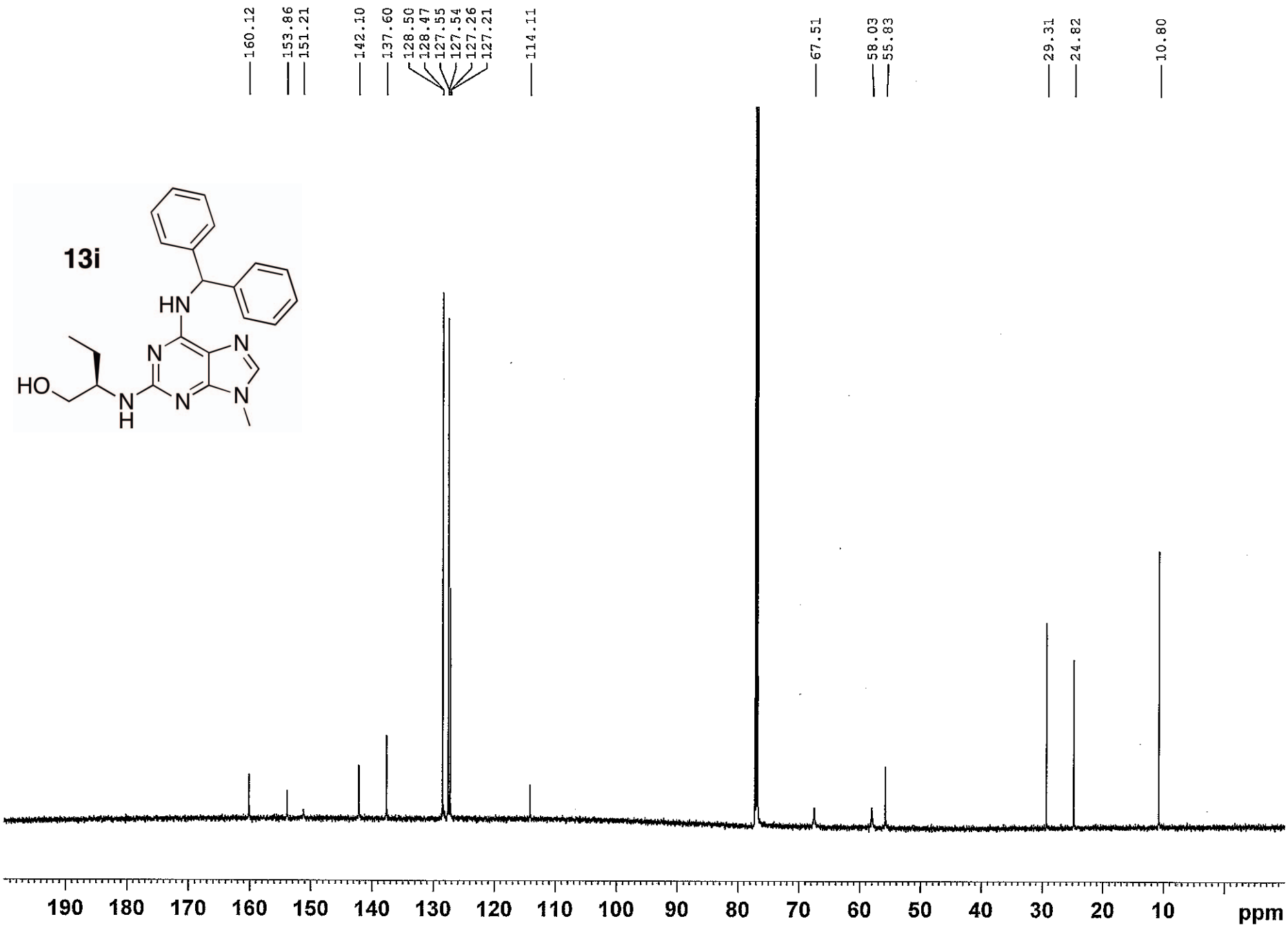
kb-NB-213-18


154.53
154.24
150.68
141.29
140.86
128.48
127.64
127.40
118.41
65.80
60.34
57.34
31.52
29.90
29.64
22.59
21.01
15.23
14.14
14.08
16i
HN
N
N
N
N
Cl
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

kb-NB-213-21


7.337
7.323
7.320
7.305
7.293
7.270
7.261
7.259
7.250
7.247
7.239
6.461
4.832
4.822
3.766
3.701
3.681
3.666
3.614
3.611
3.587
3.564
3.511
3.501
1.577
1.566
1.555
1.543
1.532
1.480
1.468
1.456
1.445
1.433
0.948
0.936
0.924
13i
HN
N
N
HO
N
H
N
N
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
8.16
2.09
1.61
0.92
0.97
0.98
3.05
1.00
1.00
1.10
3.15

kb-NB-213-21

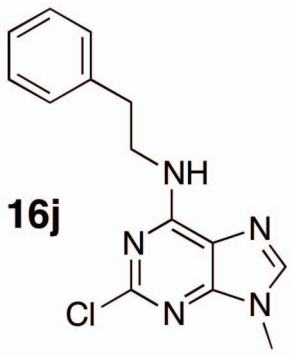

160.12
153.86
151.21
142.10
137.60
128.50
128.47
127.55
127.54
127.26
127.21
114.11
67.51
58.03
55.83
29.31
24.82
10.80
13i
HN
N
N
HO
N
H
N
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

kb-NB-213-19

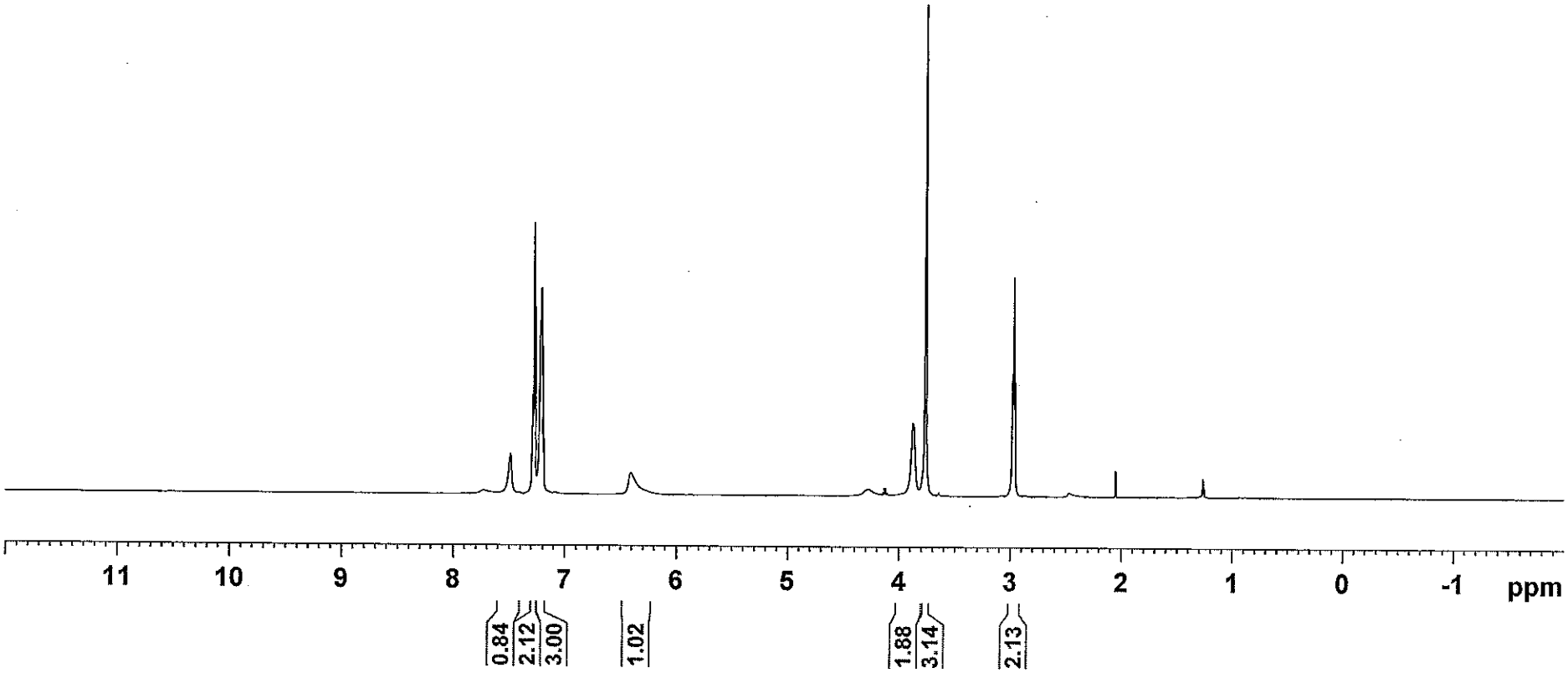
7.489
7.292
7.279
7.267
7.224
7.214
7.202
7.190
6.407

3.876
3.765

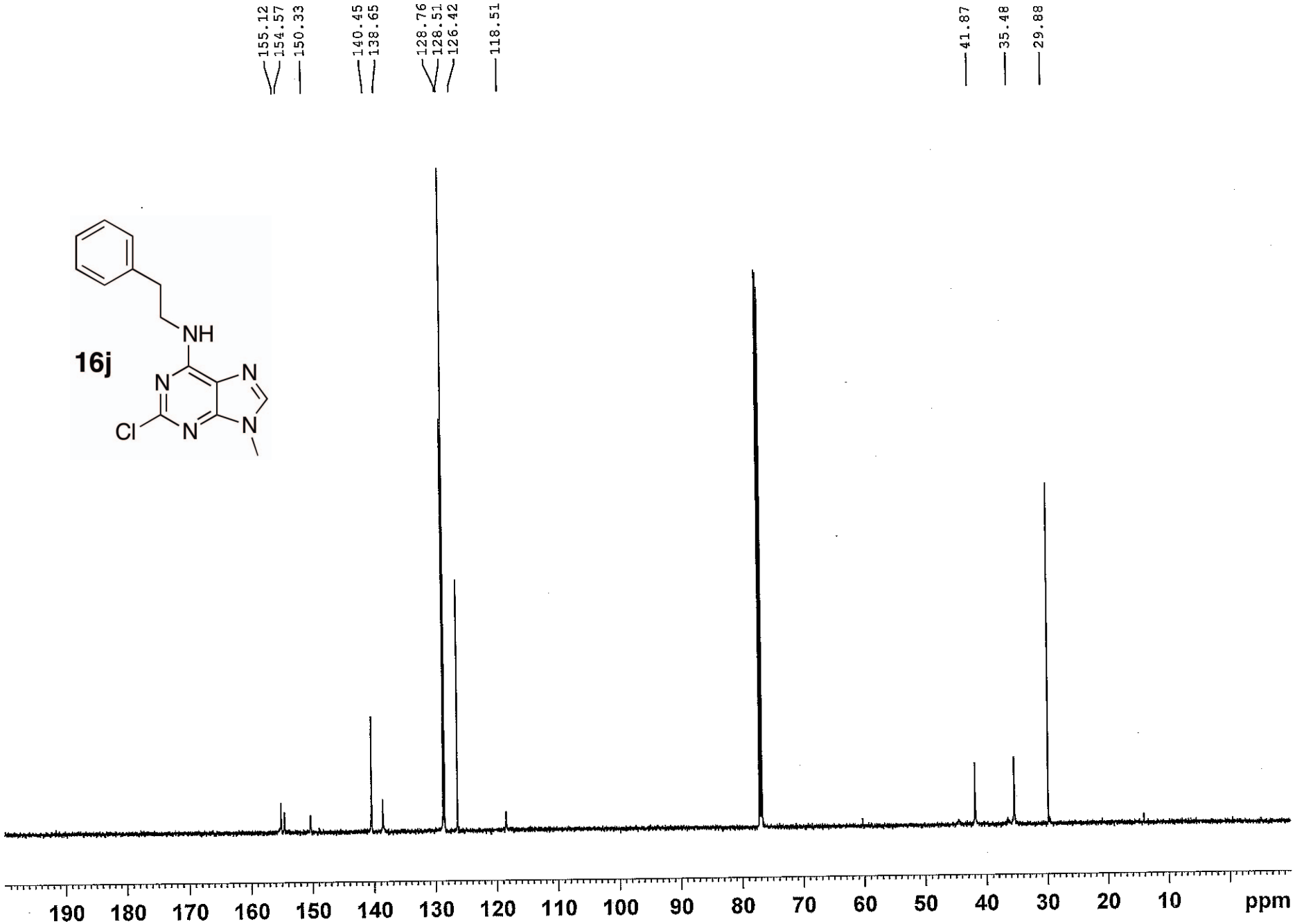
2.983
2.971
2.960



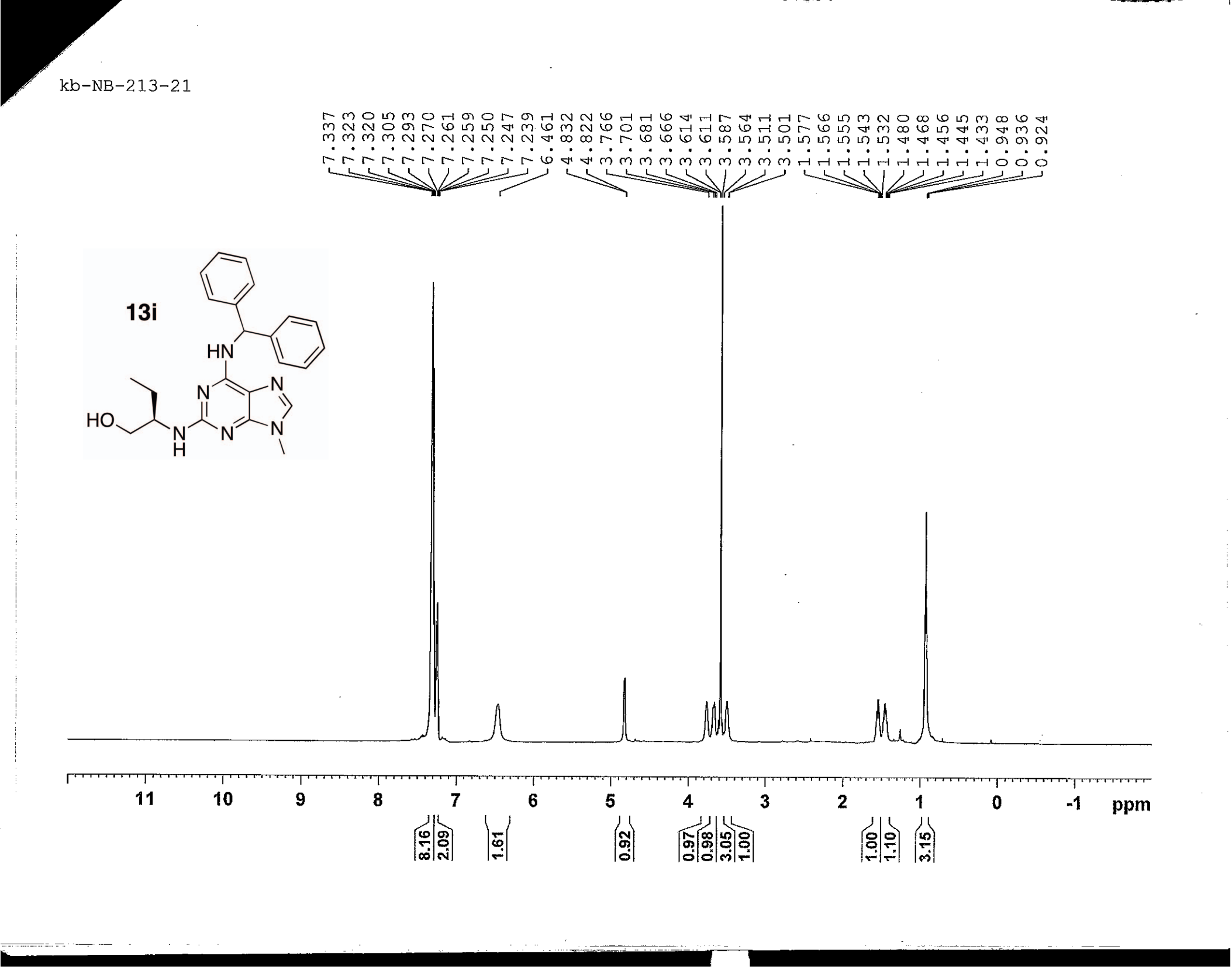
16j



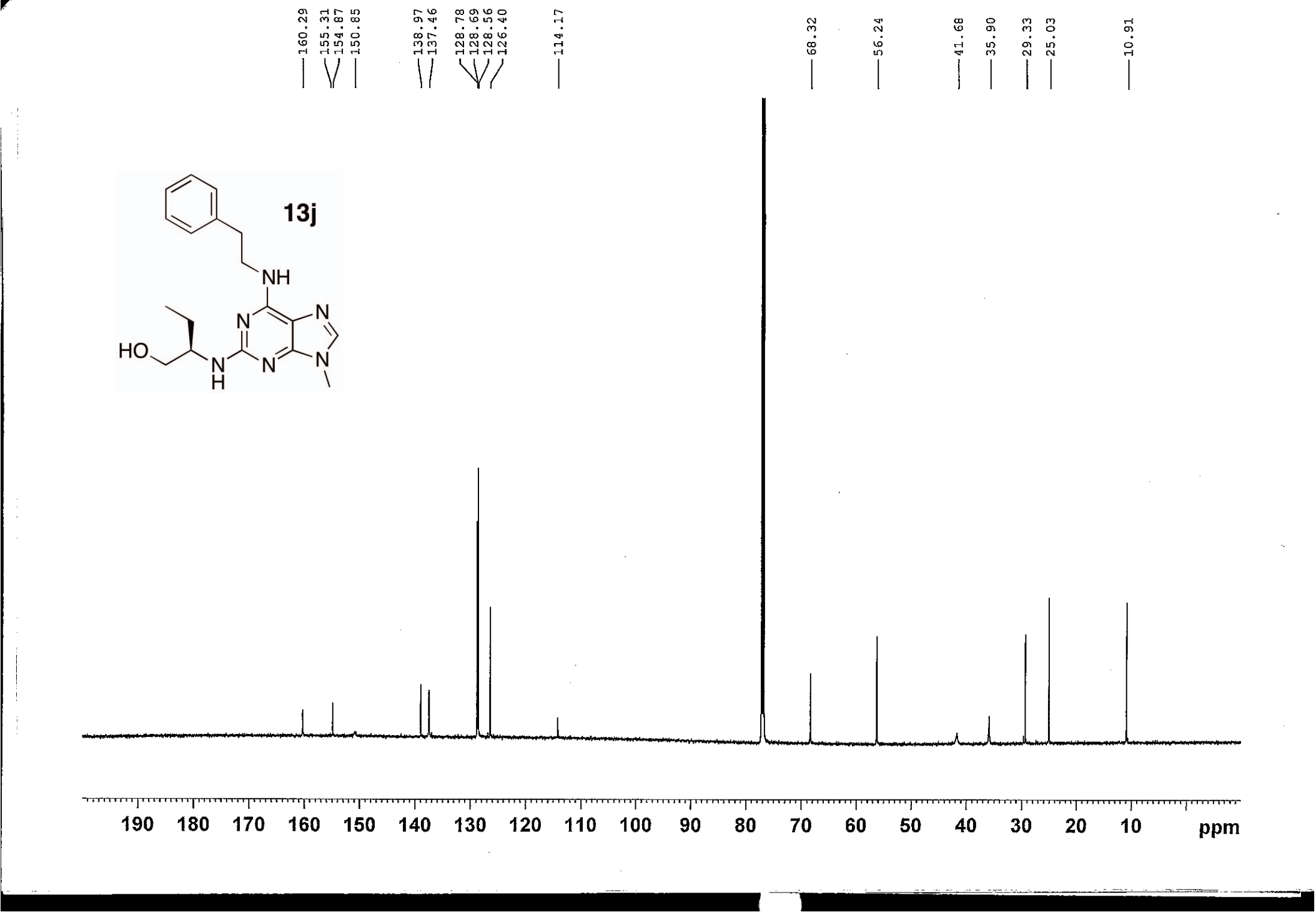
kb-NB-213-19


155.12
154.57
150.33
140.45
138.65
128.76
128.51
126.42
118.51
41.87
35.48
29.88
16j
NH
N
N
N
N
Cl
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

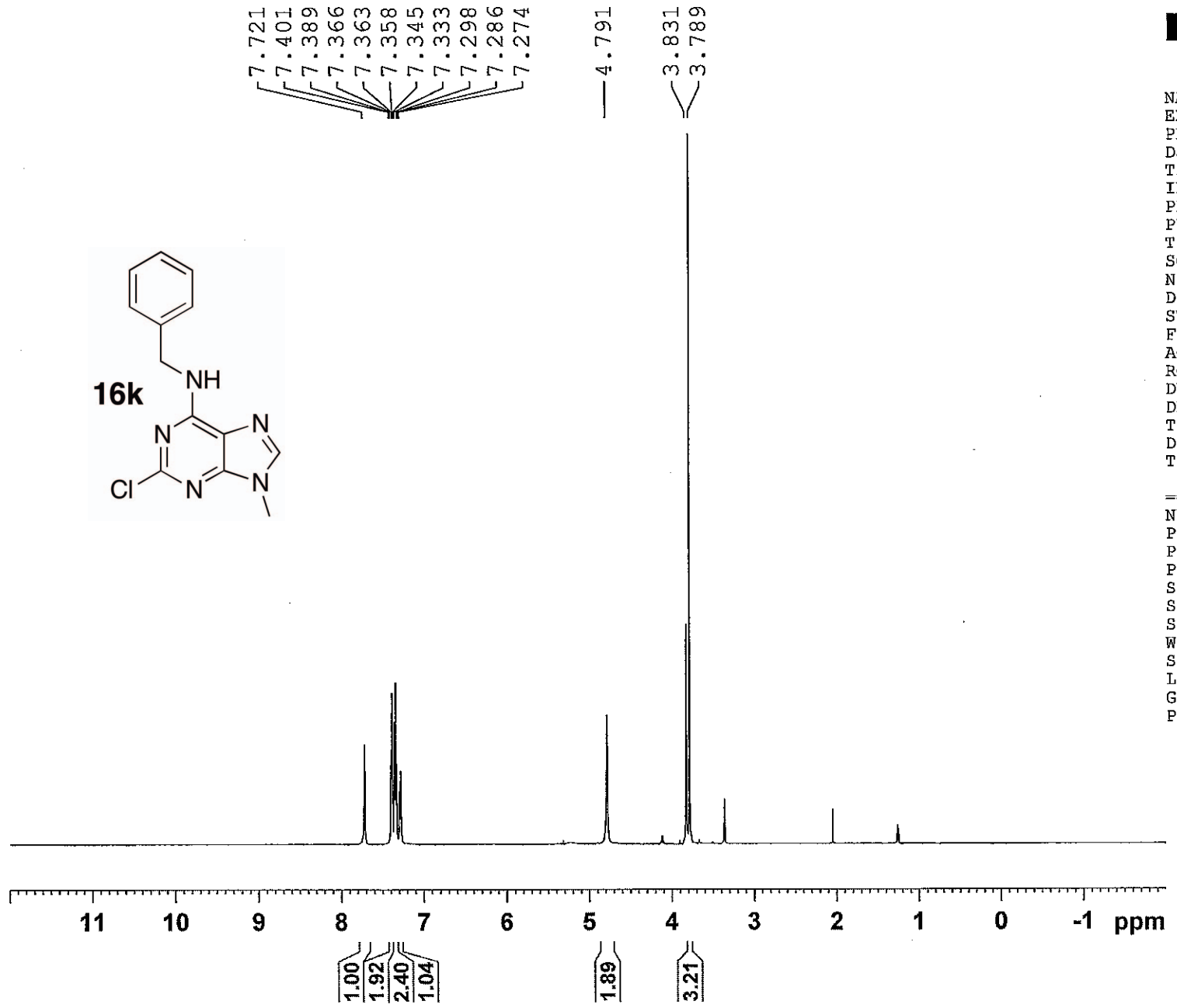
kb-NB-213-21


7.337
7.323
7.320
7.305
7.293
7.270
7.261
7.259
7.250
7.247
7.239
6.461
4.832
4.822
3.766
3.701
3.681
3.666
3.614
3.611
3.587
3.564
3.511
3.501
1.577
1.566
1.555
1.543
1.532
1.480
1.468
1.456
1.445
1.433
0.948
0.936
0.924
13i
HN
N
N
HO
N
H
N
N
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
8.16
2.09
1.61
0.92
0.97
0.98
3.05
1.00
1.00
1.10
3.15

kb-NB-213-22

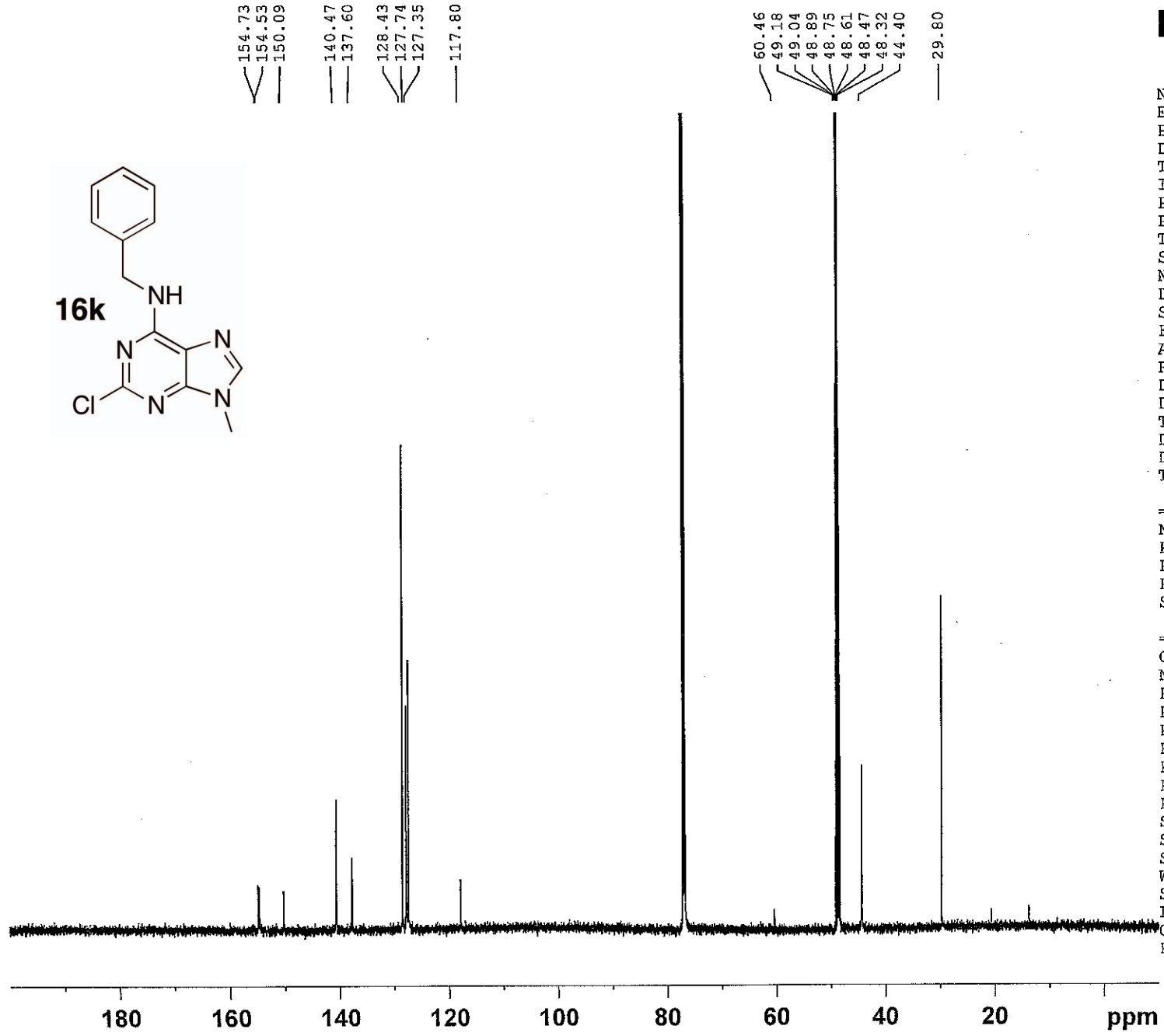

160.29
155.31
154.87
150.85
138.97
137.46
128.78
128.69
128.56
126.40
114.17
68.32
56.24
41.68
35.90
29.33
25.03
10.91
13j
NH
N
N
N
N
HO
N
H
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

kb-NB-213-23



NAME	kb-NB-213-23
EXPNO	3
PROCNO	1
Date_	20090805
Time	21.12
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	90.5
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1320466 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

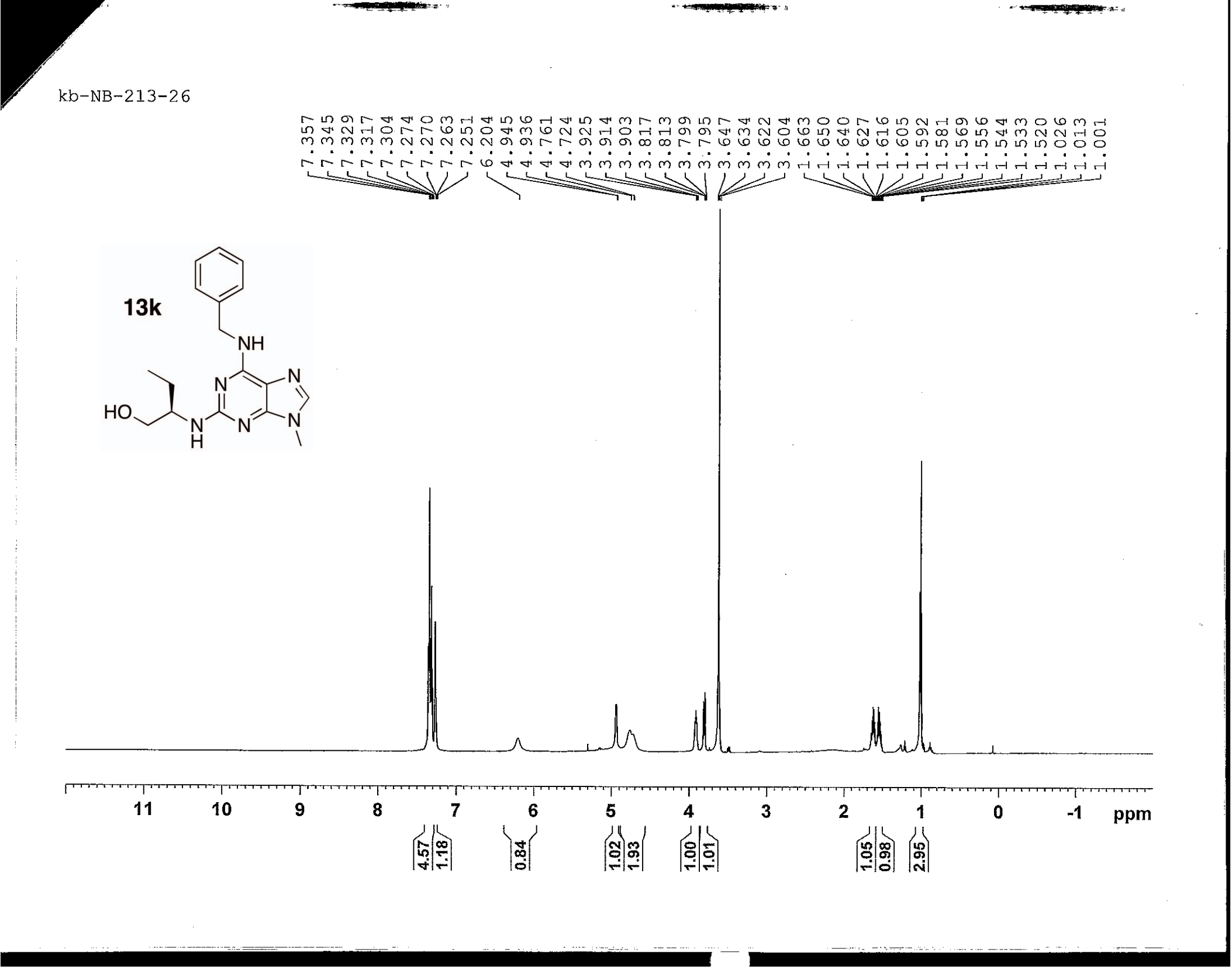
kb-NB-213-23



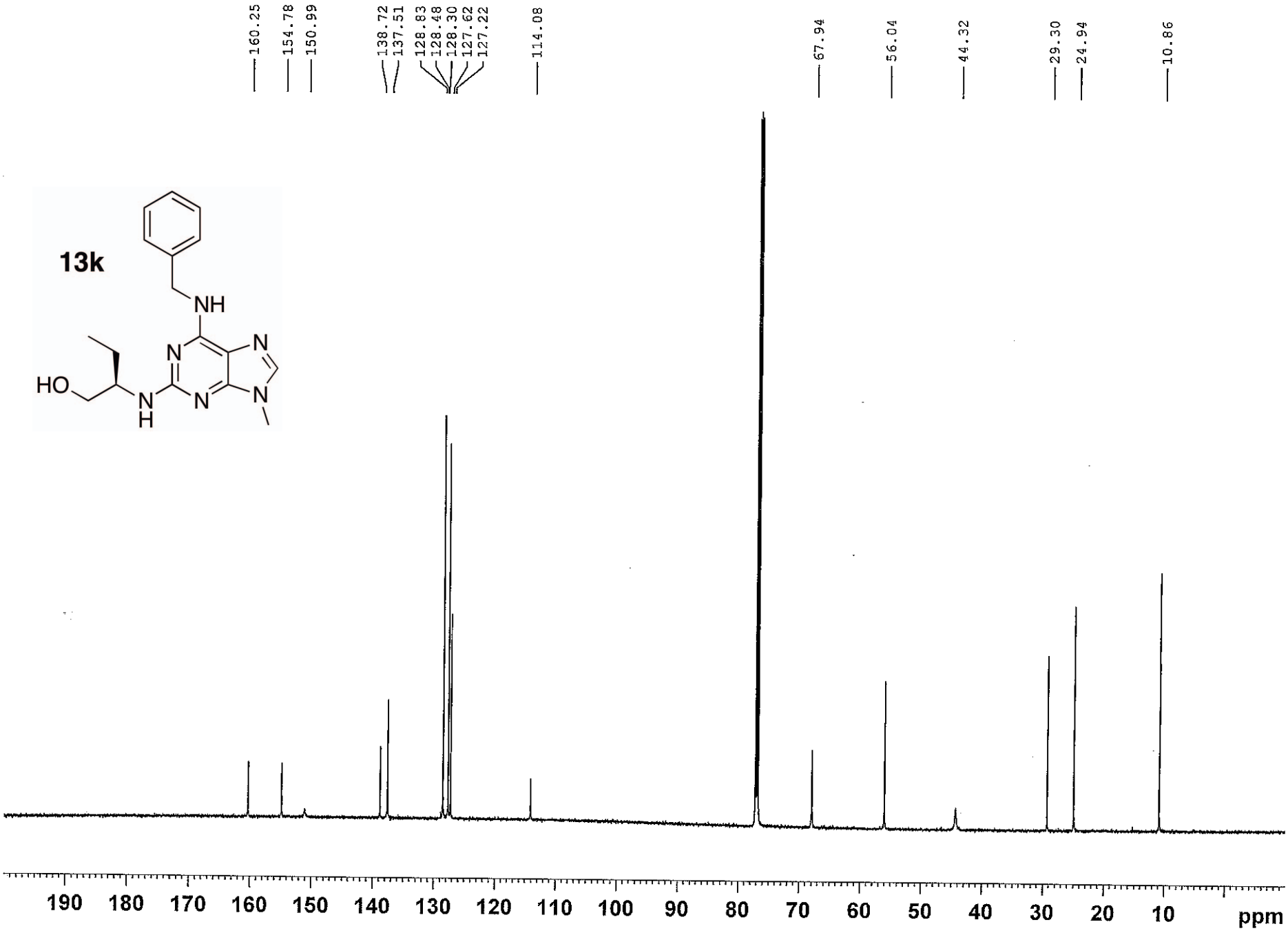
BRUKER

NAME	kb-NB-213-23
EXPNO	5
PROCNO	1
Date_	20090805
Time	22.12
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	630
DS	4
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	13004
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9033543 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

kb-NB-213-26

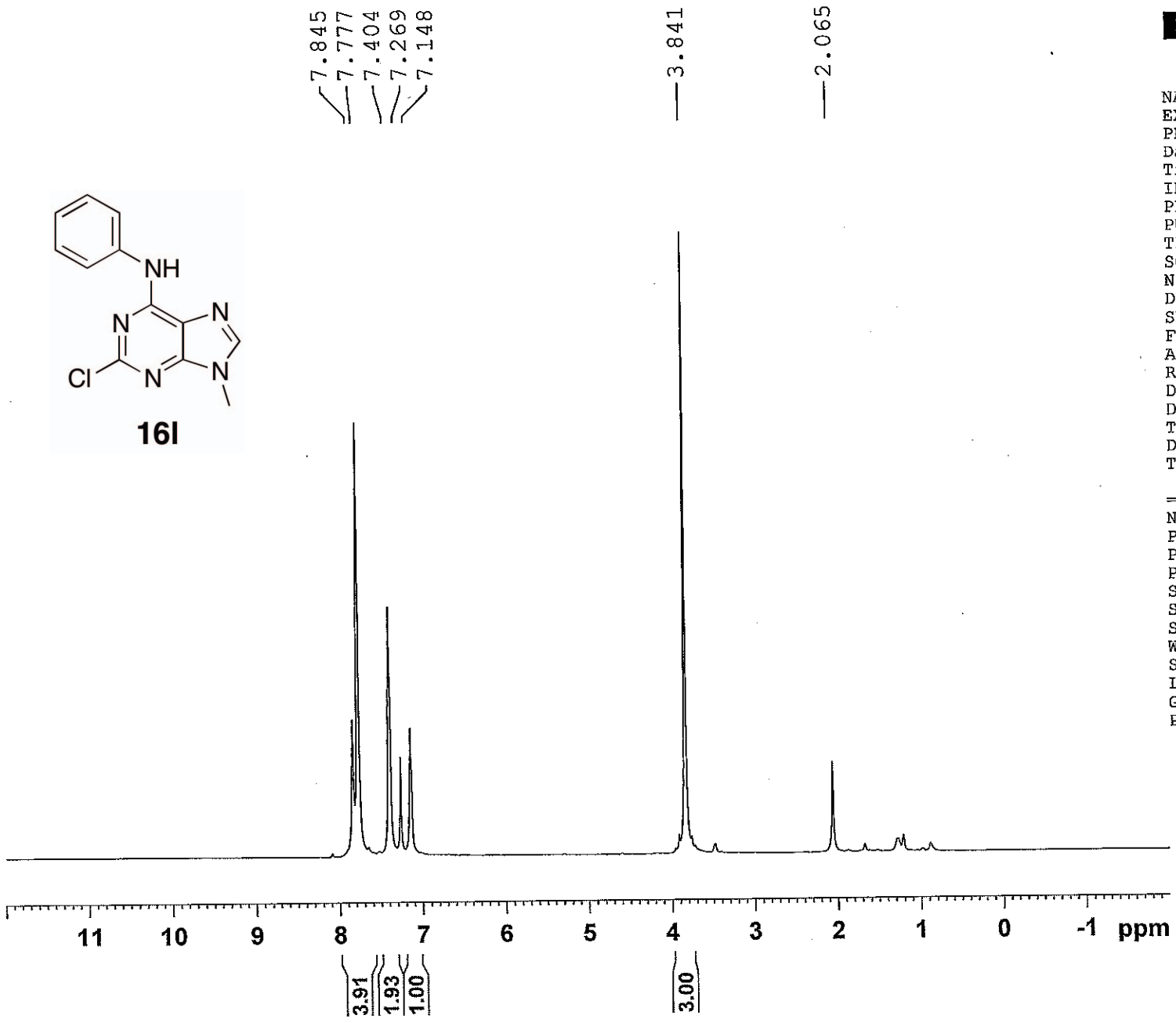

7.357
7.345
7.329
7.317
7.304
7.274
7.270
7.263
7.251
6.204
4.945
4.936
4.761
4.724
3.925
3.914
3.903
3.817
3.813
3.799
3.795
3.647
3.634
3.622
3.604
1.663
1.650
1.640
1.627
1.616
1.605
1.592
1.581
1.569
1.556
1.544
1.533
1.520
1.026
1.013
1.001
13k
NH
N
N
N
HO
N
H
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
4.57
1.18
0.84
1.02
1.93
1.00
1.01
1.05
0.98
2.95

kb-NB-213-26


160.25
154.78
150.99
138.72
137.51
128.83
128.48
128.30
127.62
127.22
114.08
67.94
56.04
44.32
29.30
24.94
10.86
13k
NH
N
N
N
N
HO
N
H
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

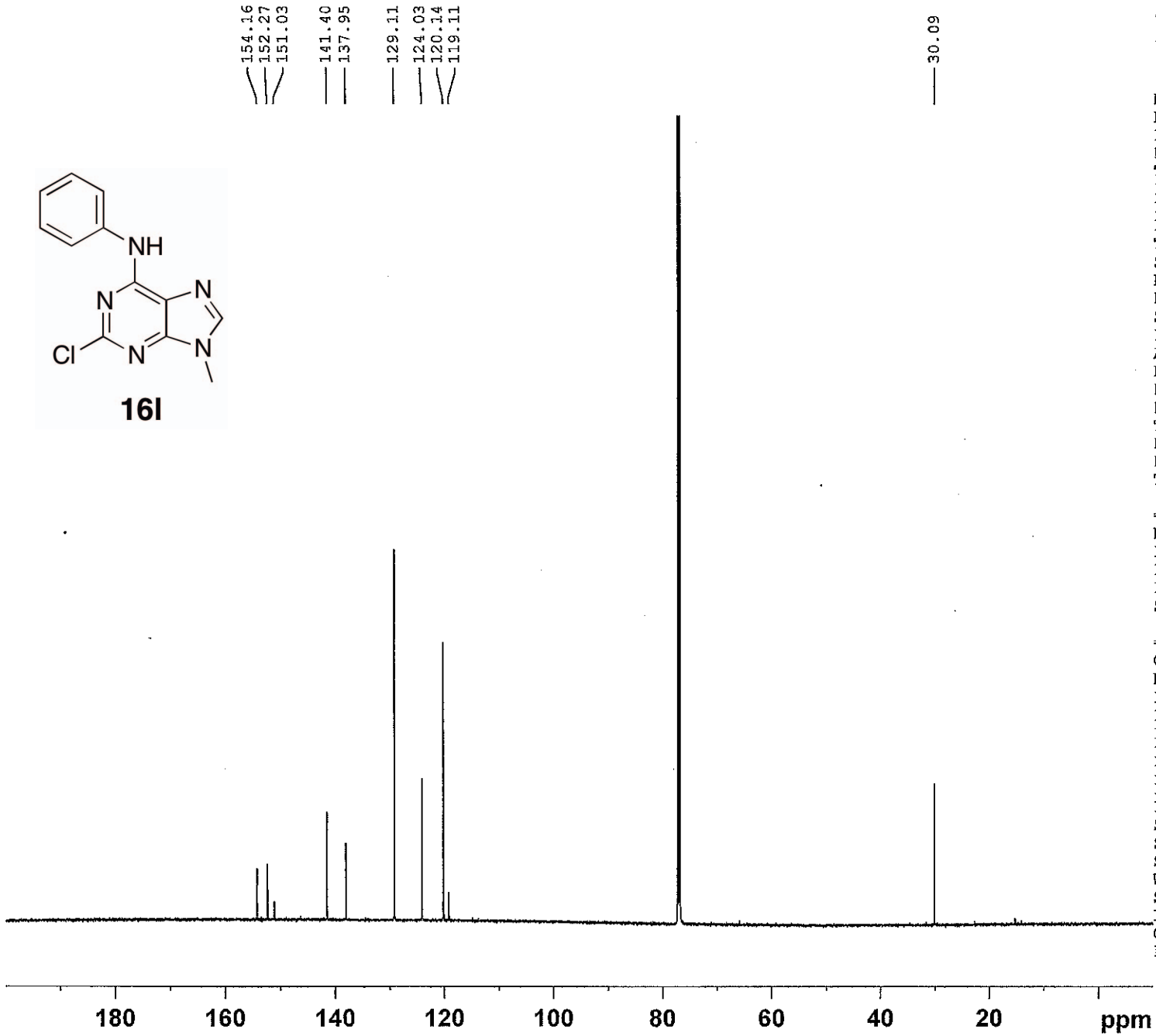
kb-NB-213-24

16l



NAME	kb-NB-213-24
EXPNO	1
PROCNO	1
Date_	20090805
Time	19.43
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	128
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300051 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

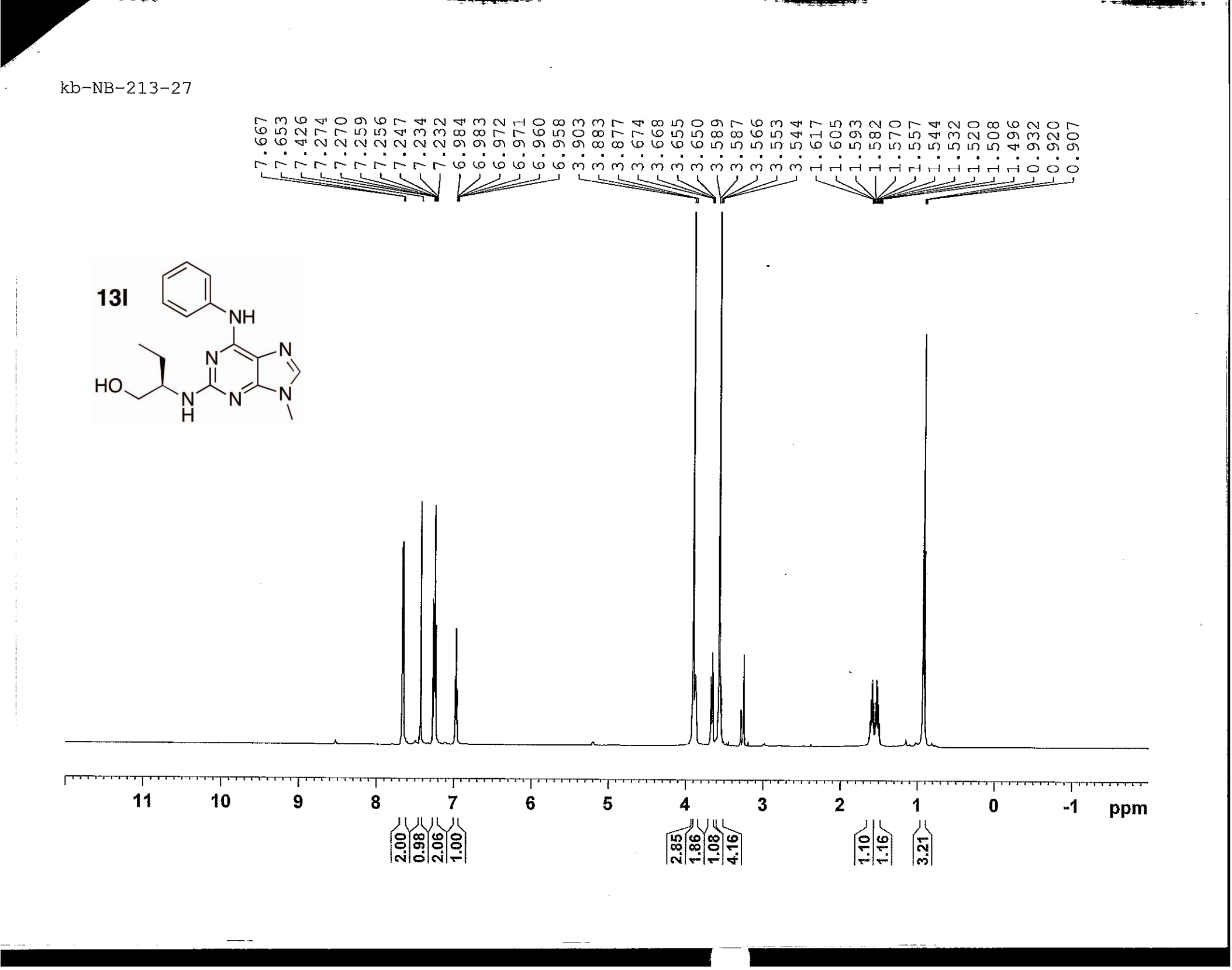
kb-NB-213-24



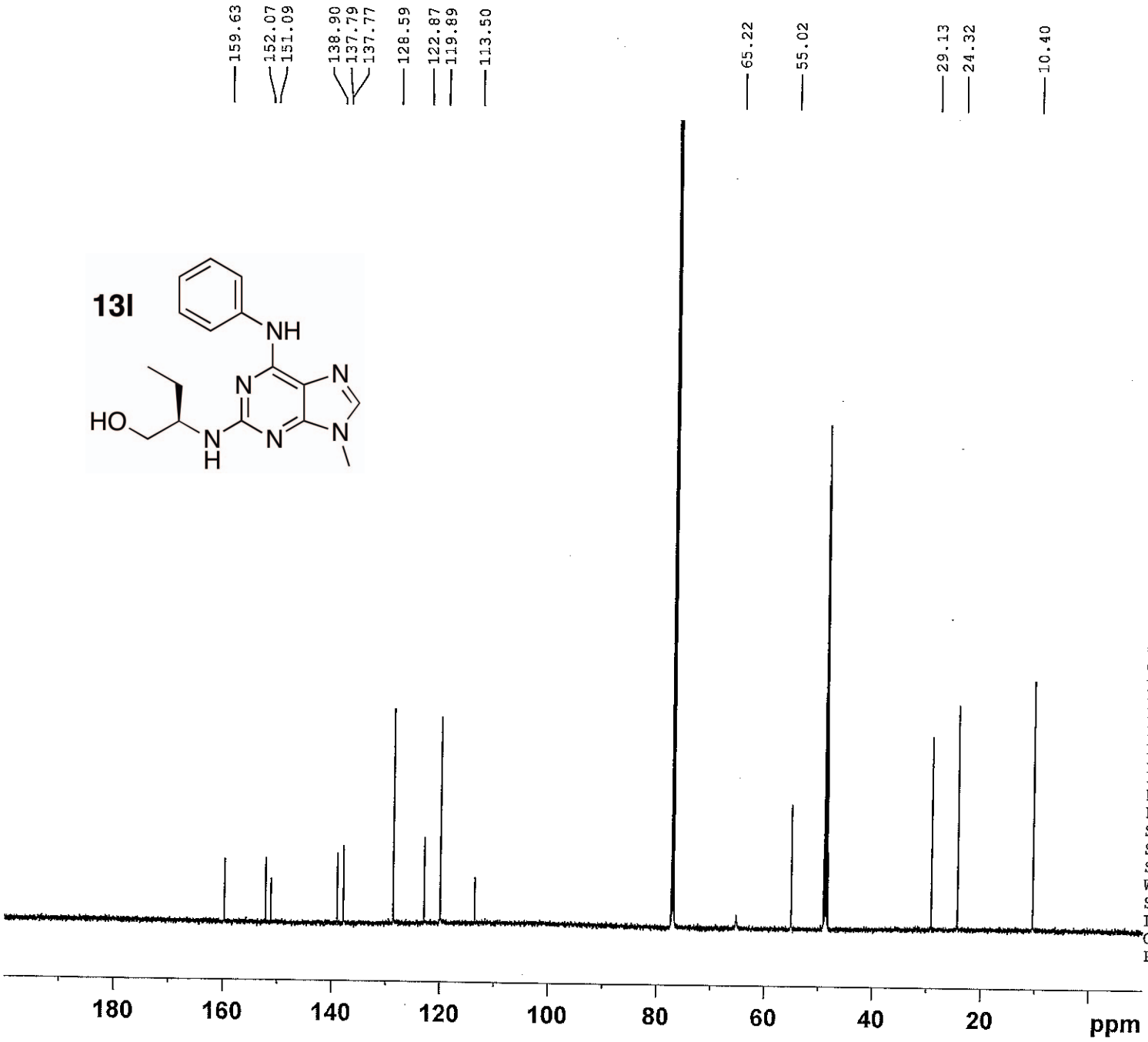
BRUKER

NAME	kb-NB-213-24
EXPNO	7
PROCNO	1
Date_	20090806
Time	0.09
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	16384
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028143 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

kb-NB-213-27


7.667
7.653
7.426
7.274
7.270
7.259
7.256
7.247
7.234
7.232
6.984
6.983
6.972
6.971
6.960
6.958
3.903
3.883
3.877
3.674
3.668
3.655
3.650
3.589
3.587
3.566
3.553
3.544
1.617
1.605
1.593
1.582
1.570
1.557
1.544
1.532
1.520
1.508
1.496
0.932
0.920
0.907
13I
NH
N
N
N
N
HO
H
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
0.98
2.06
1.00
2.85
1.86
1.08
4.16
1.10
1.16
3.21

kb-NB-213-27



BRUKER

NAME	kb-NB-213-27
EXPNO	2
PROCNO	1
Date_	20090811
Time	10.20
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	4
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	18390.4
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

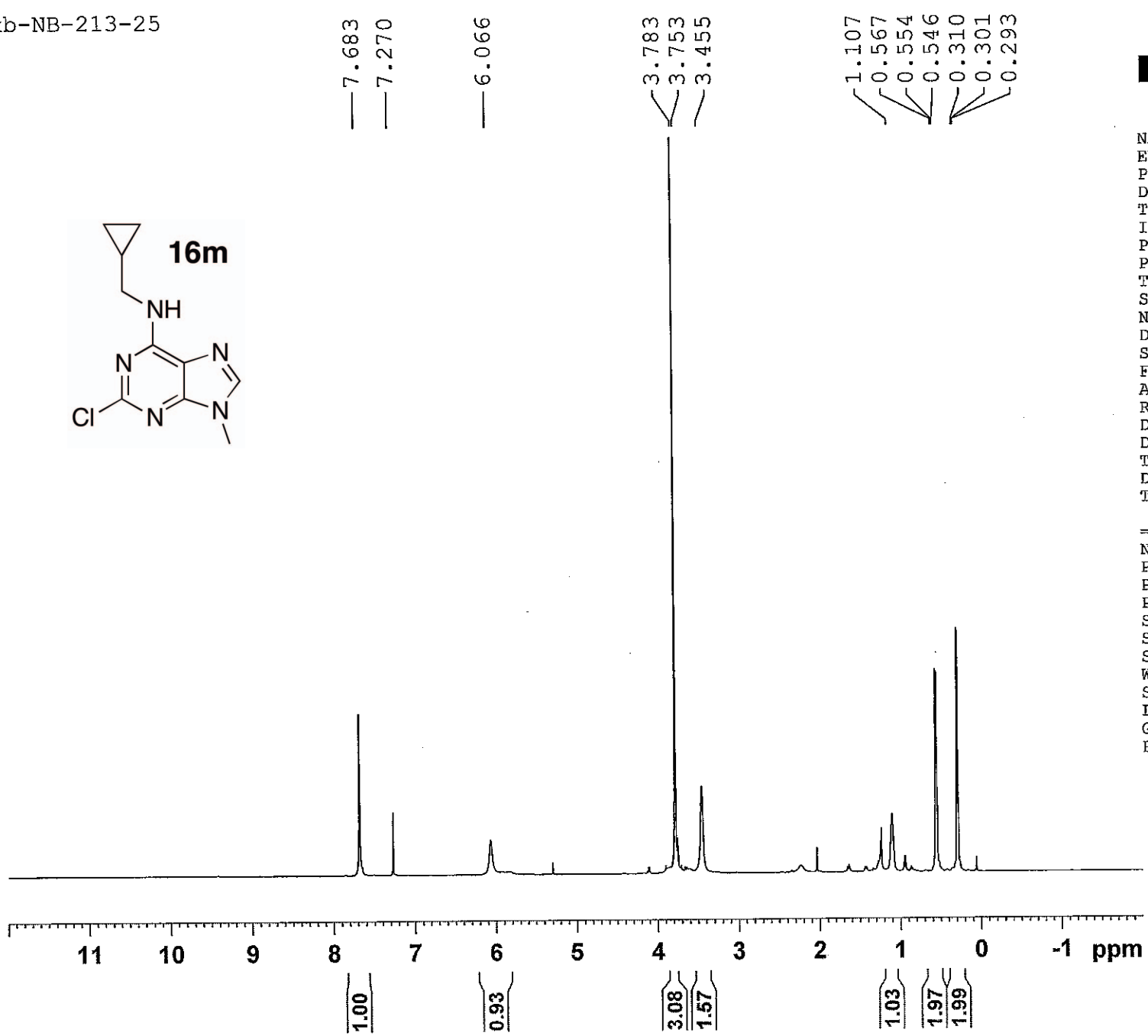
======== CHANNEL f1 ========

NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9033311 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

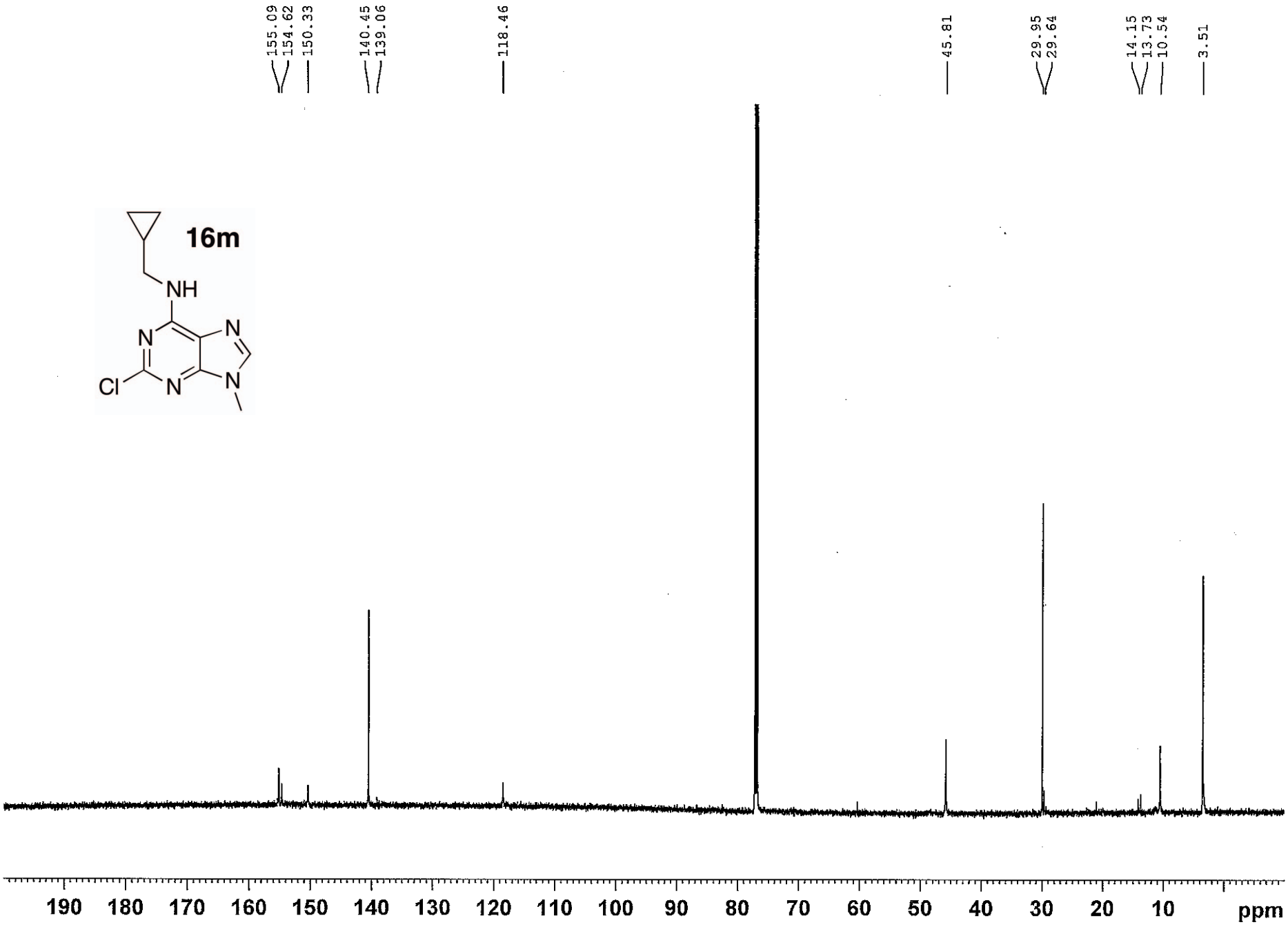
kb-NB-213-25



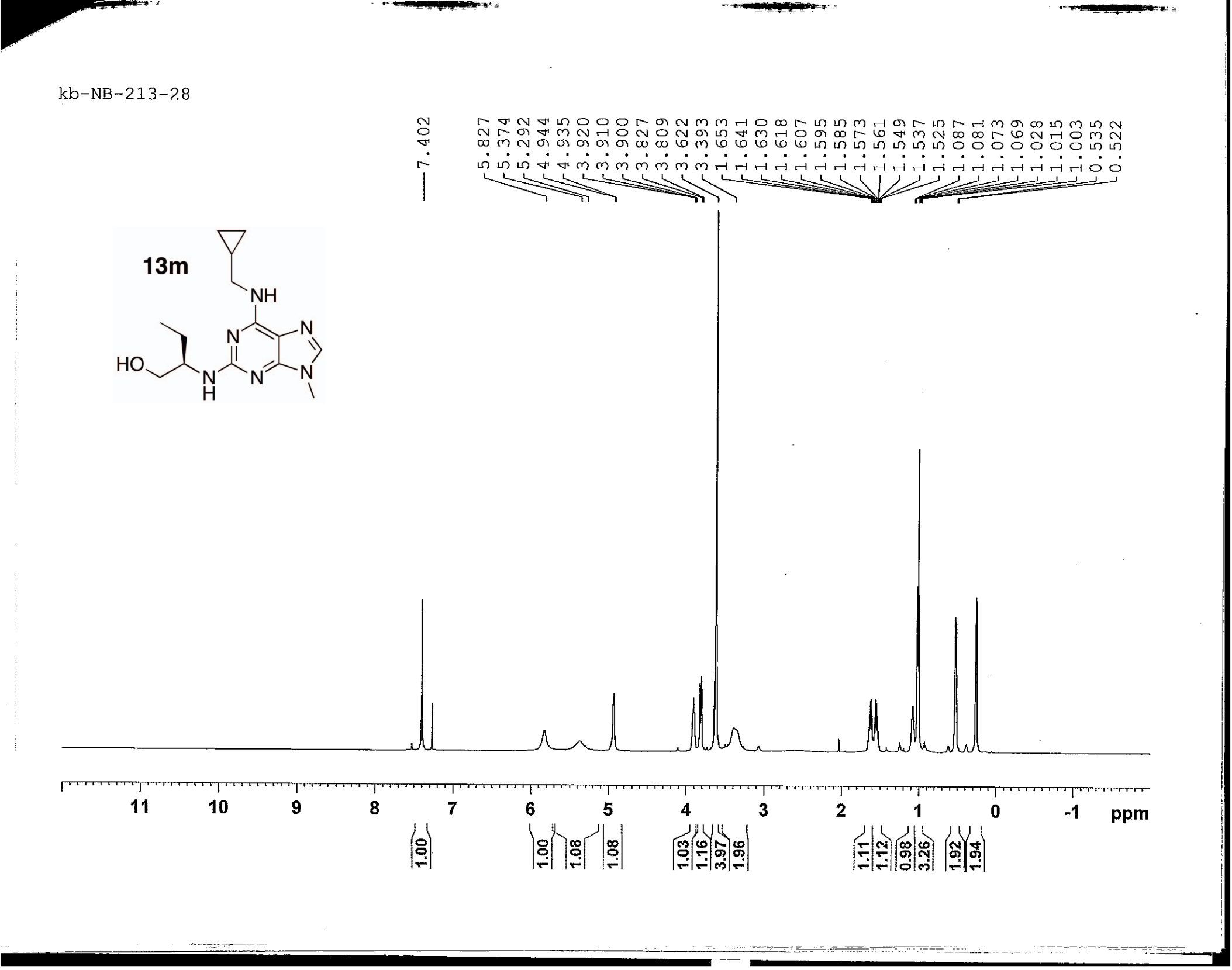
BRUKER

NAME	kb-NB-213-25
EXPNO	1
PROCNO	1
Date_	20090805
Time	19.59
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	57
DW	40.400 usec
DE	6.50 usec
TE	298.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300047 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

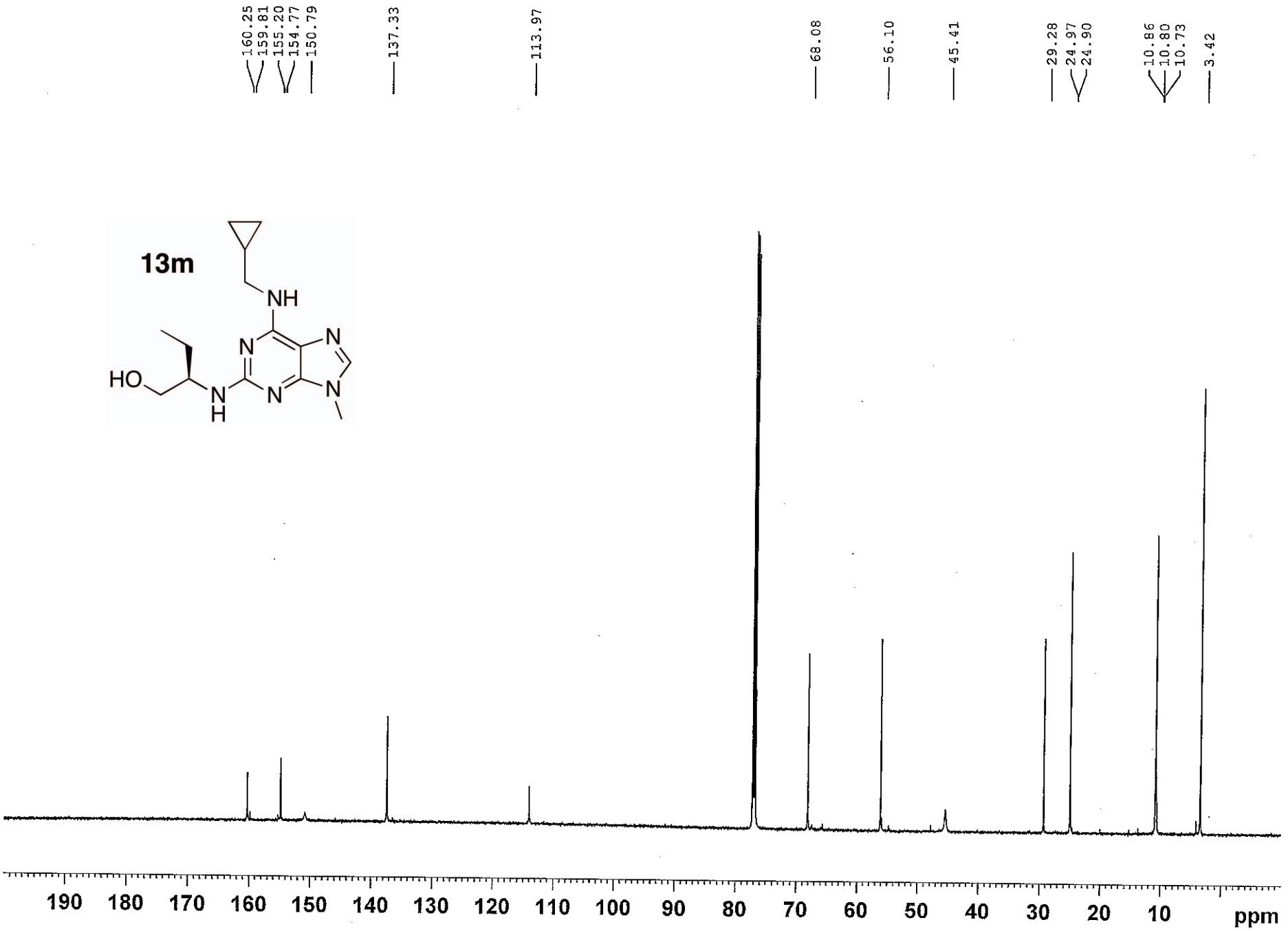
kb-NB-213-25


155.09
154.62
150.33
140.45
139.06
118.46
45.81
29.95
29.64
14.15
13.73
10.54
3.51
16m
NH
N
N
N
N
Cl
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

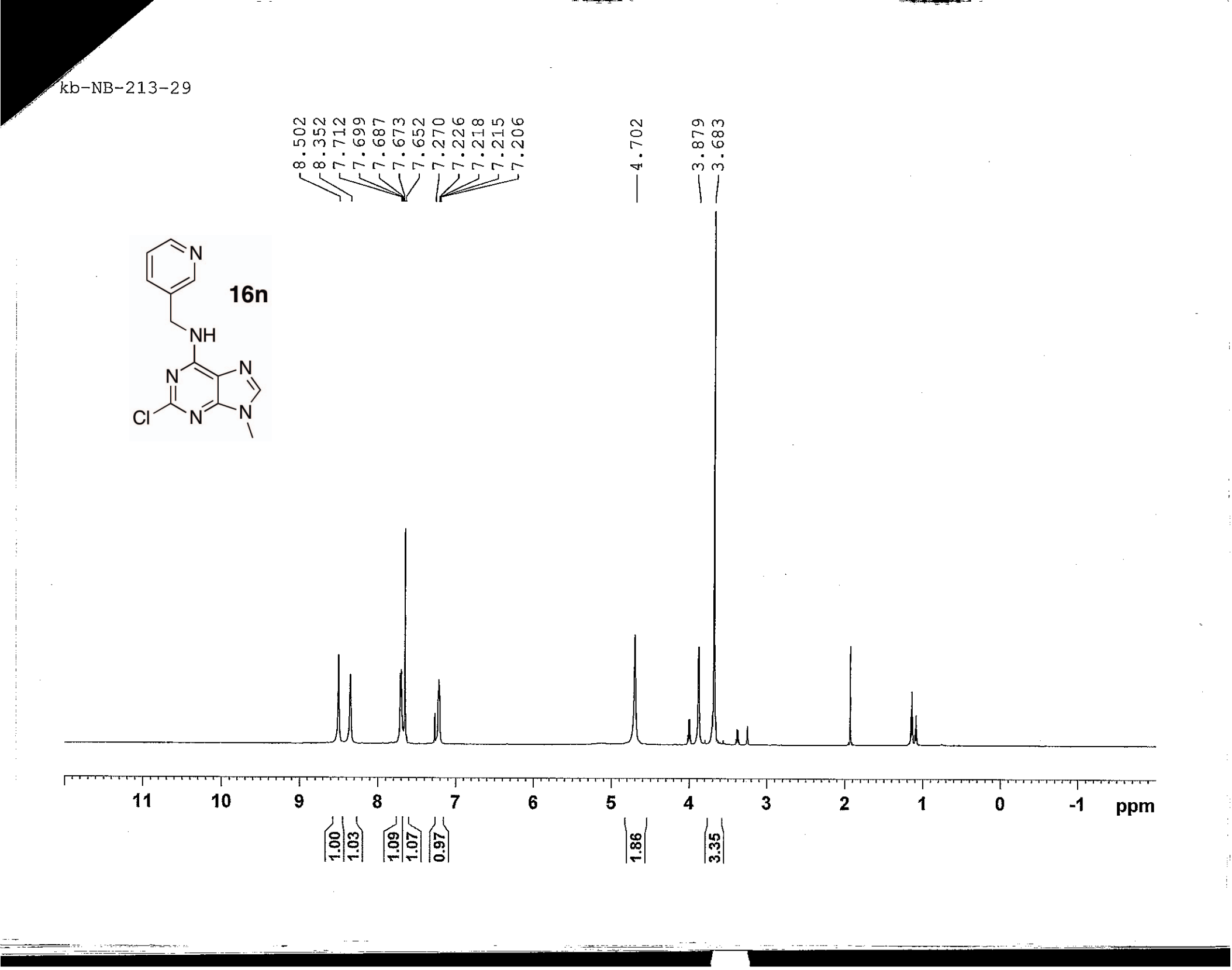
kb-NB-213-28


7.402
5.827
5.374
5.292
4.944
4.935
3.920
3.910
3.900
3.827
3.809
3.622
3.393
1.653
1.641
1.630
1.618
1.607
1.595
1.585
1.573
1.561
1.549
1.537
1.525
1.087
1.081
1.073
1.069
1.028
1.015
1.003
0.535
0.522
13m
NH
N
N
N
HO
N
H
N
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.00
1.00
1.08
1.08
1.03
1.16
3.97
1.96
1.11
1.12
0.98
3.26
1.92
1.94

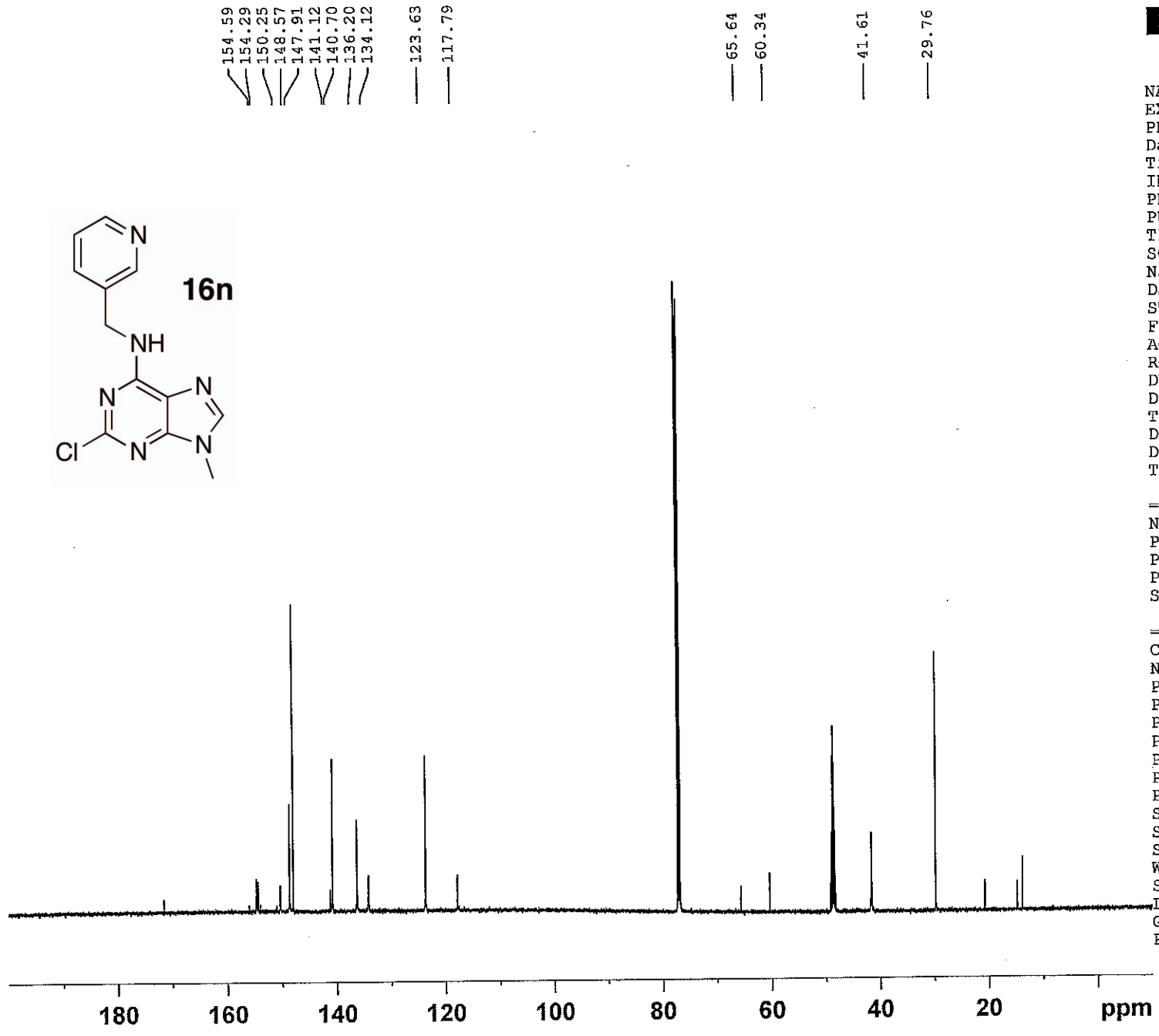
kb-NB-213-28


160.25
159.81
155.20
154.77
150.79
137.33
113.97
68.08
56.10
45.41
29.28
24.97
24.90
10.86
10.80
10.73
3.42
13m
NH
N
N
N
N
HO
N
H
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

kb-NB-213-29


8.502
8.352
7.712
7.699
7.687
7.673
7.652
7.270
7.226
7.218
7.215
7.206
4.702
3.879
3.683
16n
NH
N
Cl
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.00
1.03
1.09
1.07
0.97
1.86
3.35

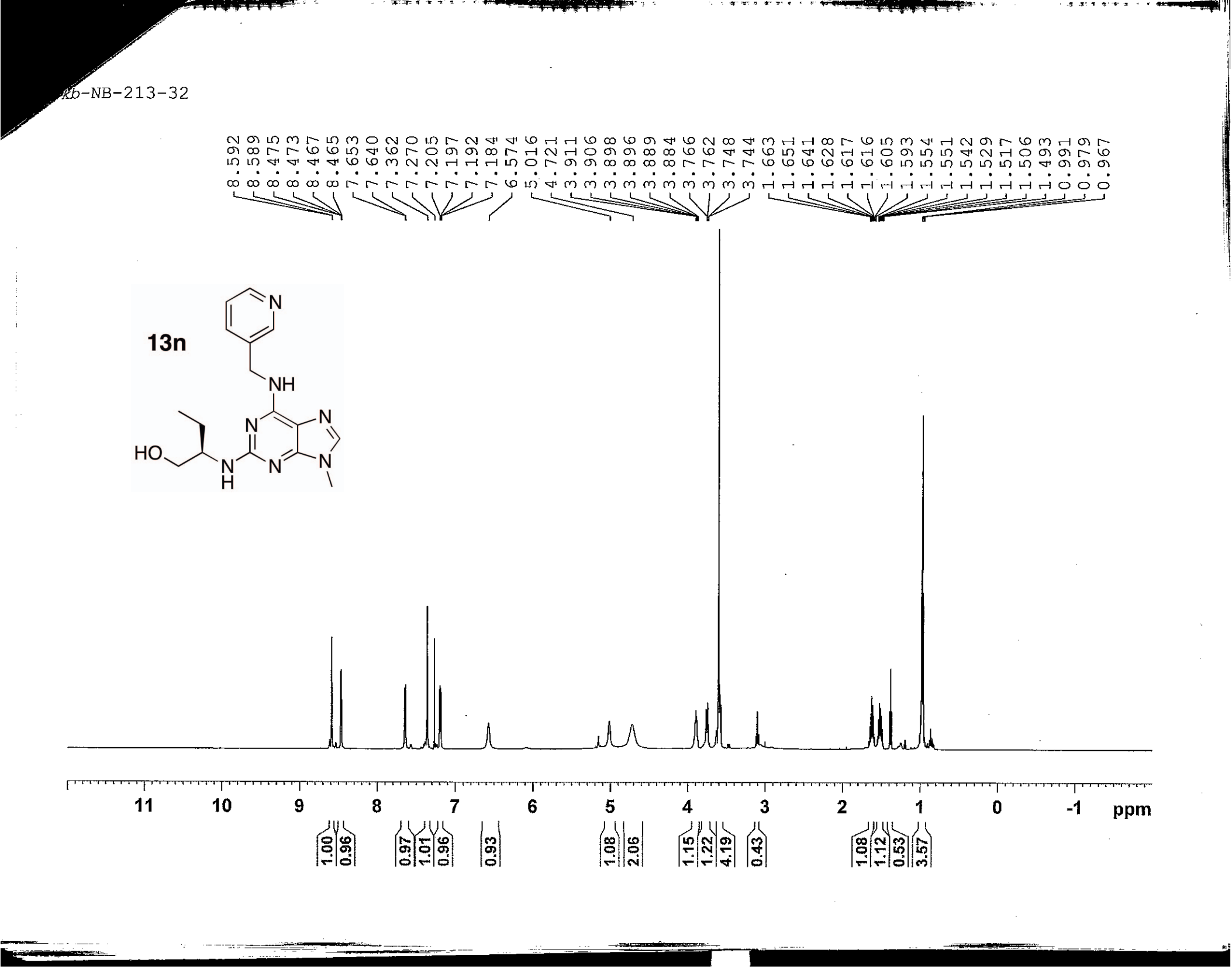
kb-NB-213-29



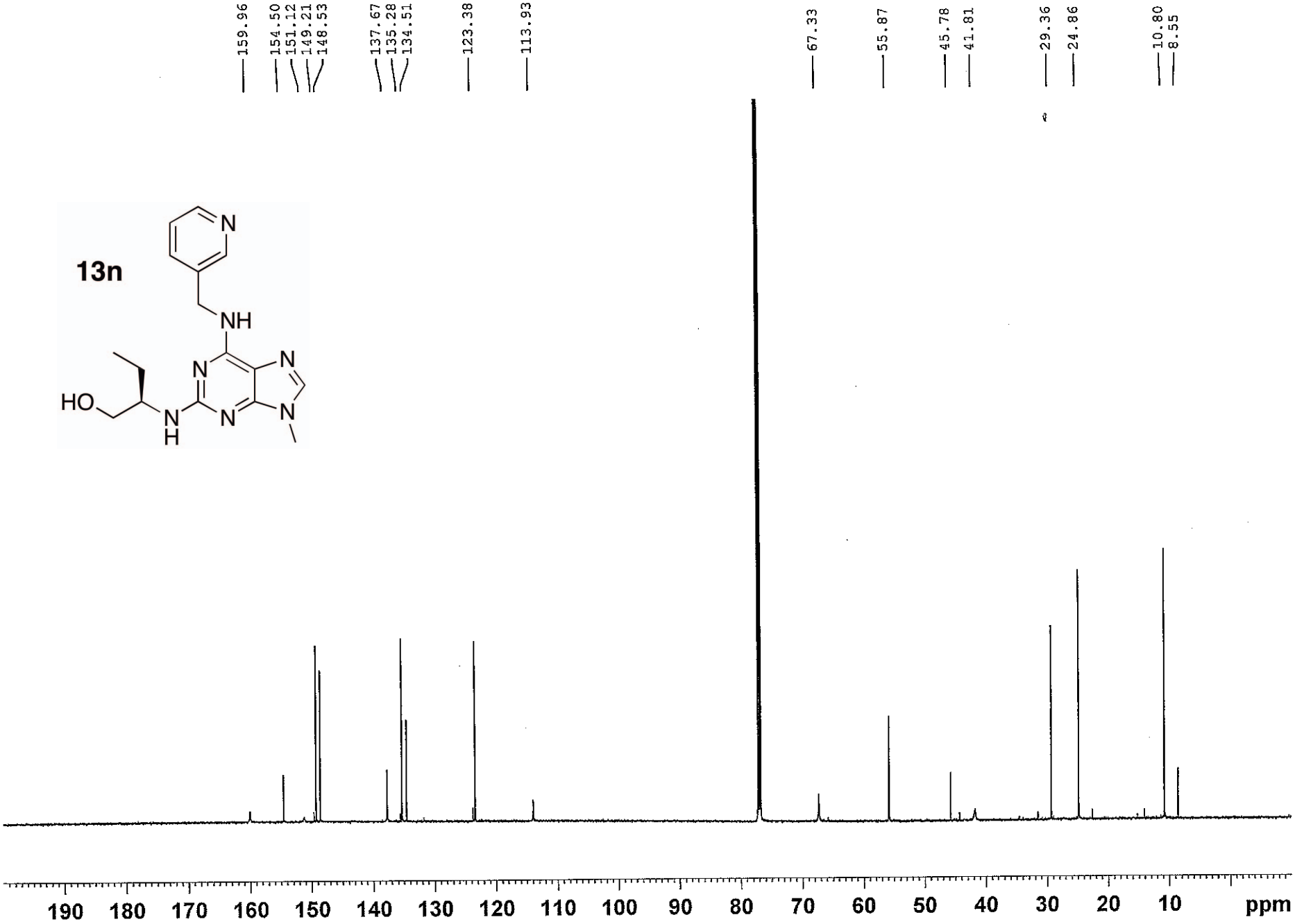
BRUKER

NAME	kb-NB-213-29
EXPNO	2
PROCNO	1
Date_	20090807
Time	15.11
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	256
DS	4
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	13004
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028324 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

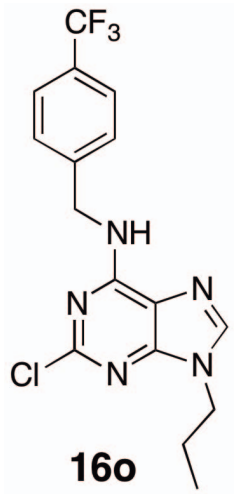
Rb-NB-213-32


8.592
8.589
8.475
8.473
8.467
8.465
7.653
7.640
7.362
7.270
7.205
7.197
7.192
7.184
6.574
5.016
4.721
3.911
3.906
3.898
3.896
3.889
3.884
3.766
3.762
3.748
3.744
1.663
1.651
1.641
1.628
1.617
1.616
1.605
1.593
1.554
1.551
1.542
1.529
1.517
1.506
1.493
0.991
0.979
0.967
13n
N
NH
N
N
N
HO
N
H
N
N
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
1.00
0.96
0.97
1.01
0.96
0.93
1.08
2.06
1.15
1.22
4.19
0.43
1.08
1.12
0.53
3.57

kb-NB-213-32


159.96
154.50
151.12
149.21
148.53
137.67
135.28
134.51
123.38
113.93
67.33
55.87
45.78
41.81
29.36
24.86
10.80
8.55
13n
NH
N
N
N
N
N
HO
N
H
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

kb-NB-213-33



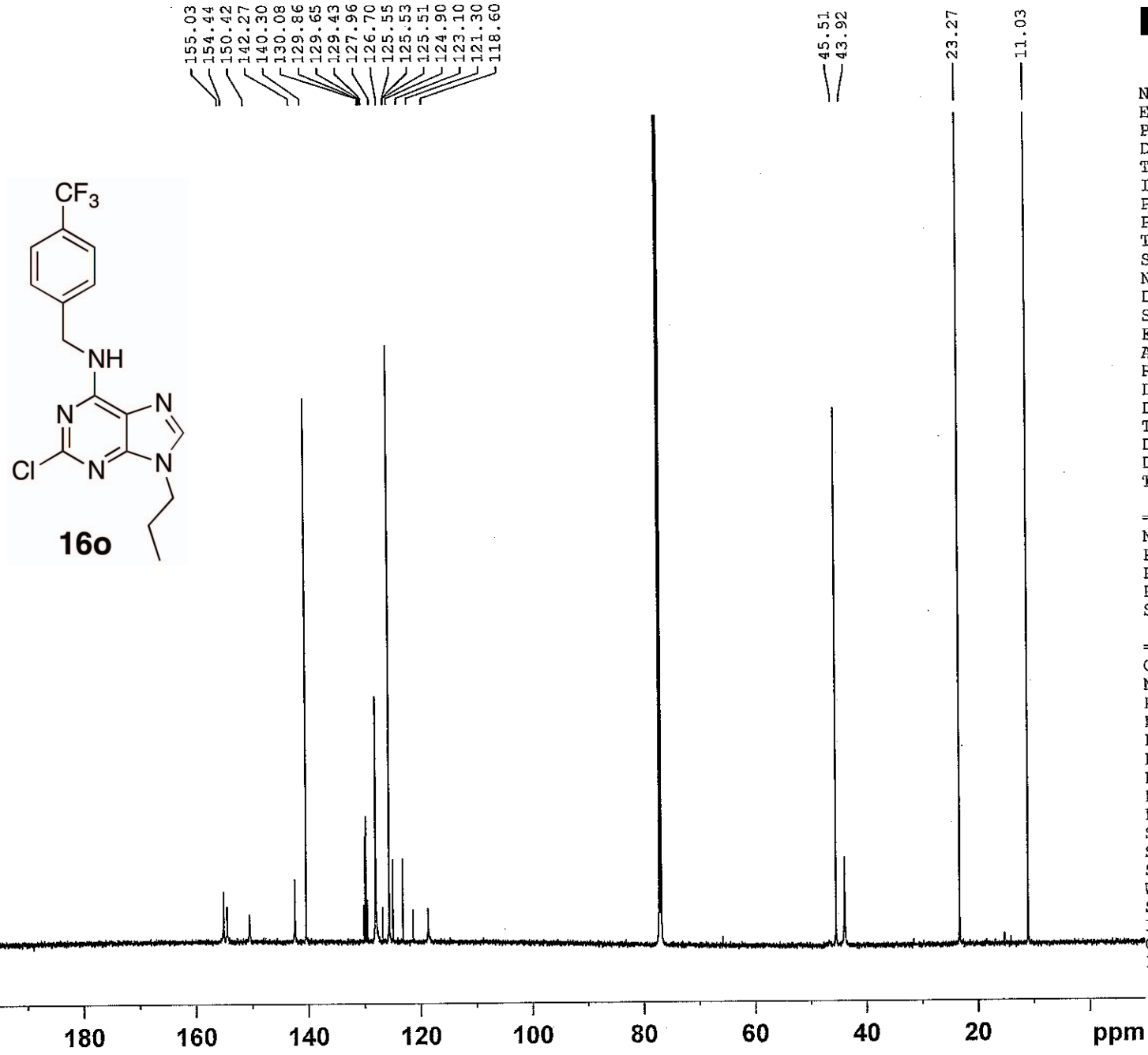
16o

7.582
7.569
7.485
7.472
6.961
4.898
4.097
4.085
4.073
2.374
1.887
1.875
1.863
1.851
0.947
0.935
0.922

11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm

2.11 2.56 0.93 1.72 1.99 1.99 3.03

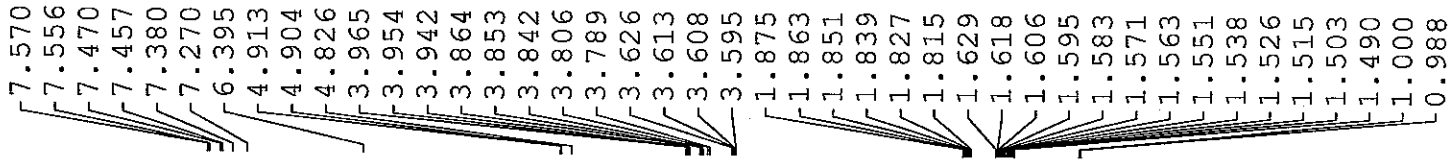
kb-NB-213-33

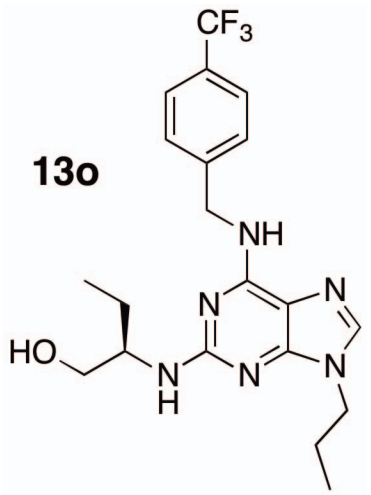


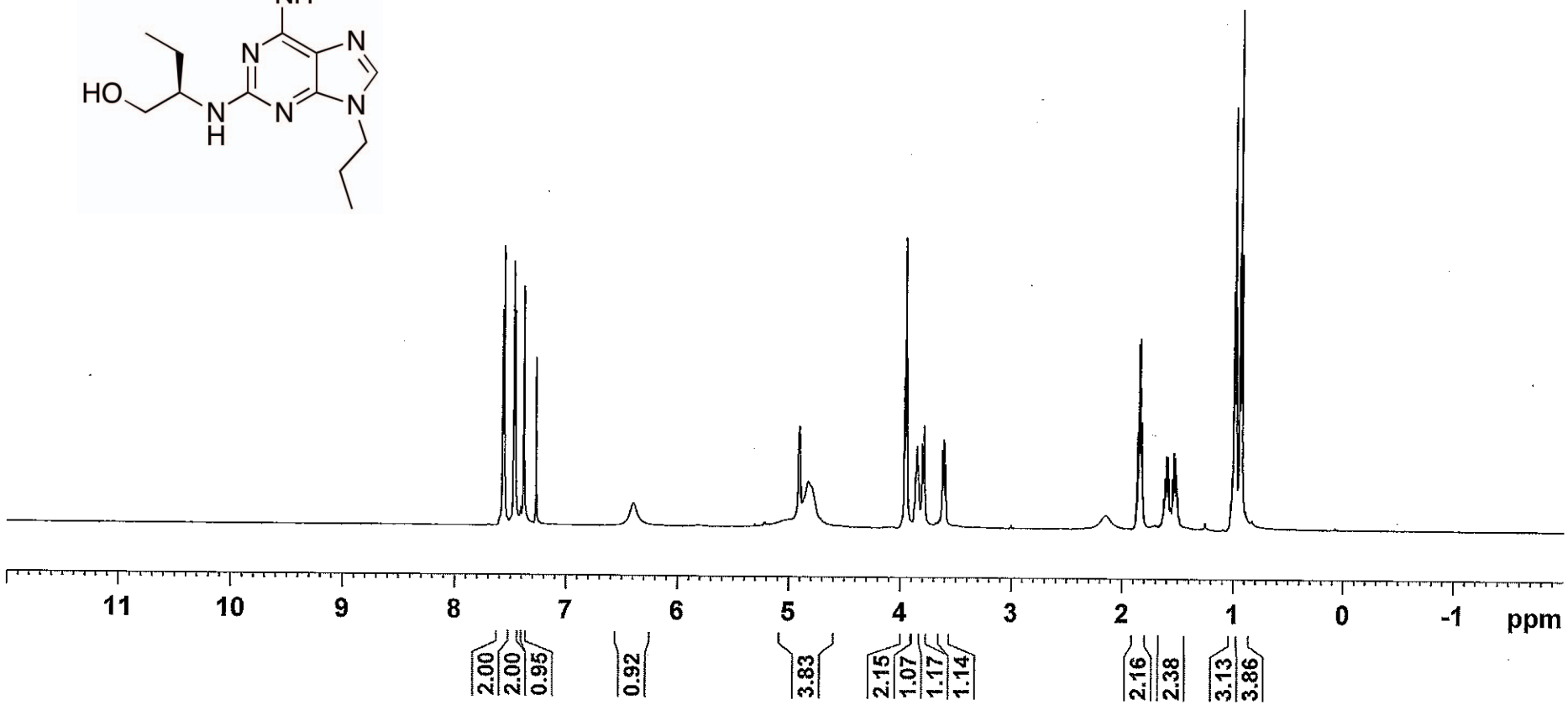
BRUKER

NAME	kb-NB-213-33
EXPNO	2
PROCNO	1
Date_	20090812
Time	23.14
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	13004
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028160 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

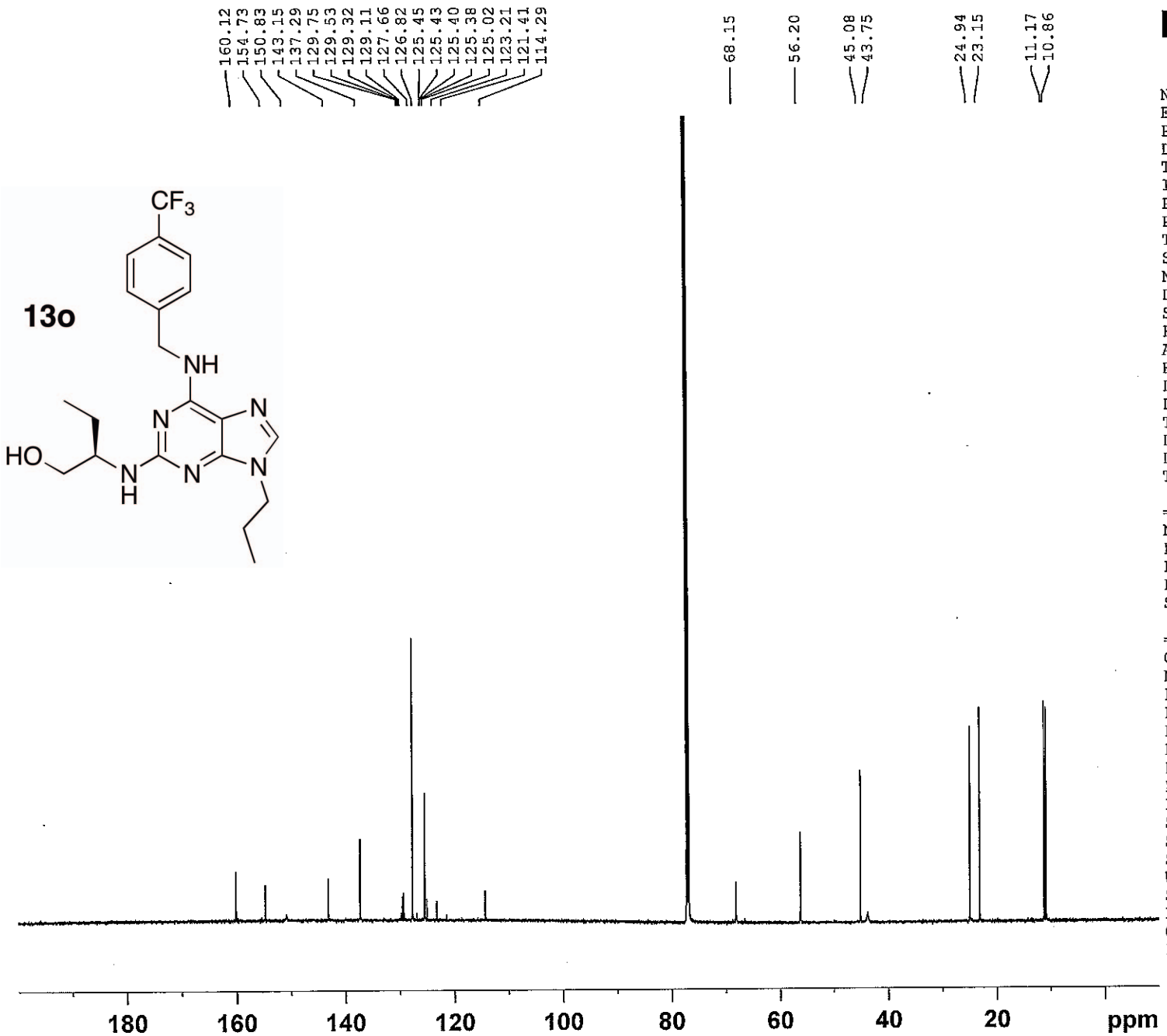
kb-NB-213-35


7.570
7.556
7.470
7.457
7.380
7.270
6.395
4.913
4.904
4.826
3.965
3.954
3.942
3.864
3.853
3.842
3.806
3.789
3.626
3.613
3.608
3.595
1.875
1.863
1.851
1.839
1.827
1.815
1.629
1.618
1.606
1.595
1.583
1.571
1.563
1.551
1.538
1.526
1.515
1.503
1.490
1.000
0.988


13o
CF3
NH
N
N
N
HO
N
H
N
N


11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
2.00
0.95
0.92
3.83
2.15
1.07
1.17
1.14
2.16
2.38
3.13
3.86

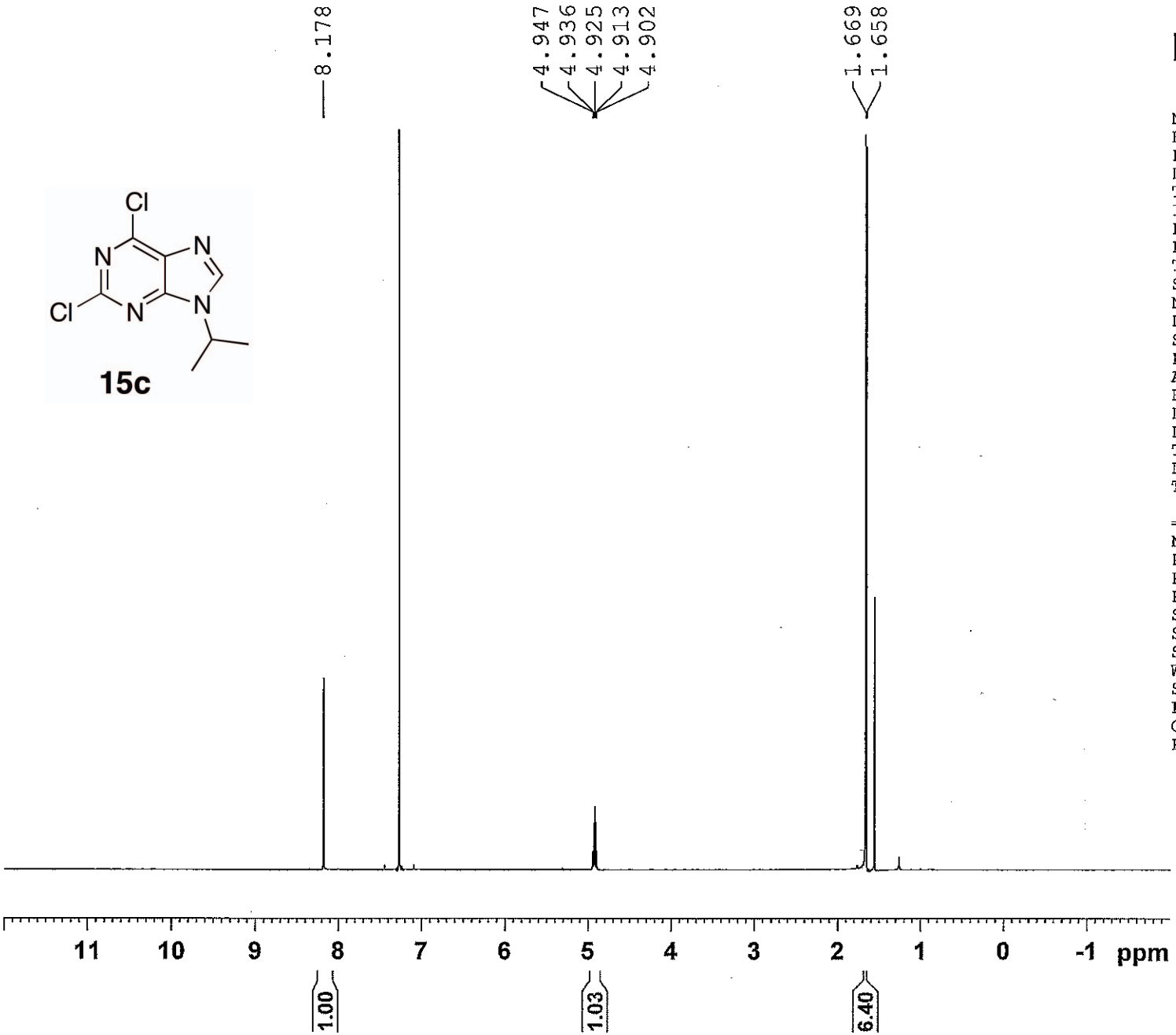
kb-NB-213-35



NAME	kb-NB-213-35	
EXPNO	2	
PROCNO	1	
Date_	20090814	
Time	23.53	
INSTRUM	spect	
PROBHD	5 mm CPTCI 1H-	
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	1024	
DS	4	
SWH	35971.223	Hz
FIDRES	0.548877	Hz
AQ	0.9110143	sec
RG	18390.4	
DW	13.900	usec
DE	6.50	usec
TE	298.0	K
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	13C	
P1	12.00	usec
PL1	-0.70	dB
PL1W	82.63385773	W
SFO1	150.9178988	MHz
======== CHANNEL f2 ========		
CPDPRG2	waltz16	
NUC2	1H	
PCPD2	80.00	usec
PL2	4.00	dB
PL12	24.00	dB
PL13	27.00	dB
PL2W	7.00000000	W
PL12W	0.07000000	W
PL13W	0.03508311	W
SFO2	600.1324005	MHz
SI	32768	
SF	150.9028138	MHz
WDW	EM	
SSB	0	
LB	1.00	Hz
GB	0	
PC	1.40	

kb-NB-184-83-1

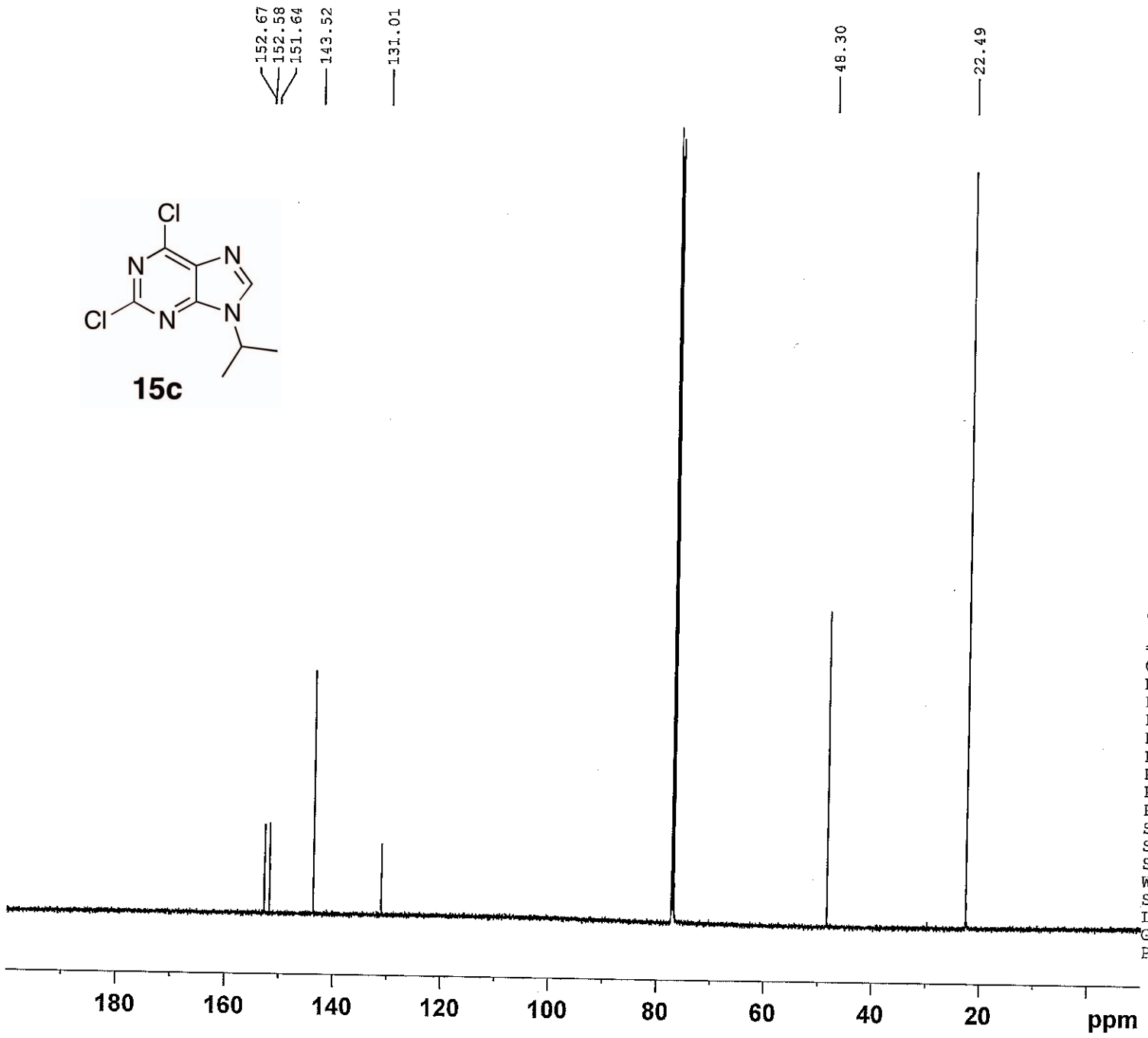
15c



BRUKER

NAME	kb-NB-184-83
EXPNO	1
PROCNO	1
Date_	20090707
Time	11.42
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	12376.237 Hz
FIDRES	0.188846 Hz
AQ	2.6477449 sec
RG	645.1
DW	40.400 usec
DE	6.50 usec
TE	297.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.00 usec
PL1	4.00 dB
PL1W	7.00000000 W
SFO1	600.1337060 MHz
SI	32768
SF	600.1300043 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

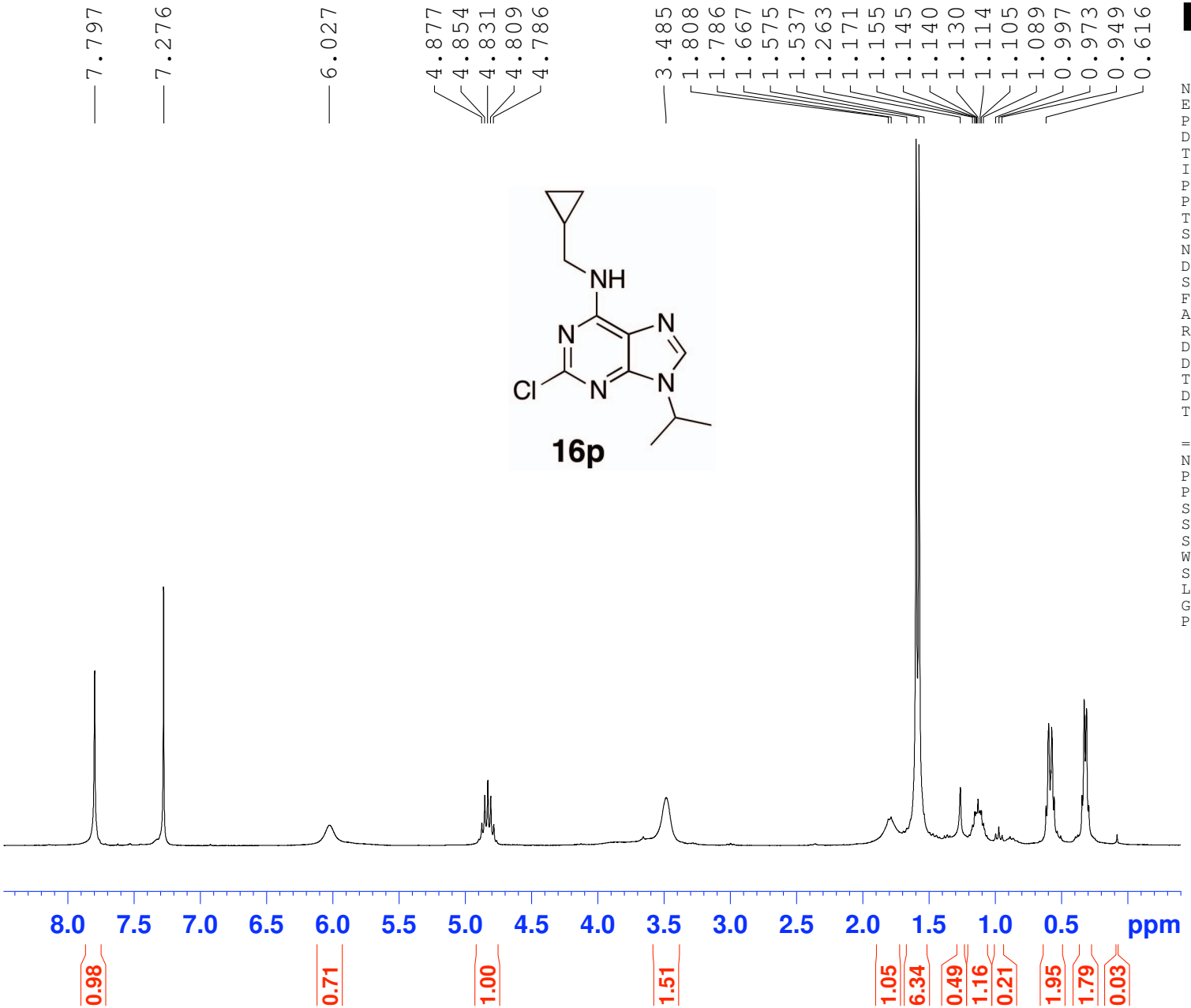
kb-NB-184-81



BRUKER

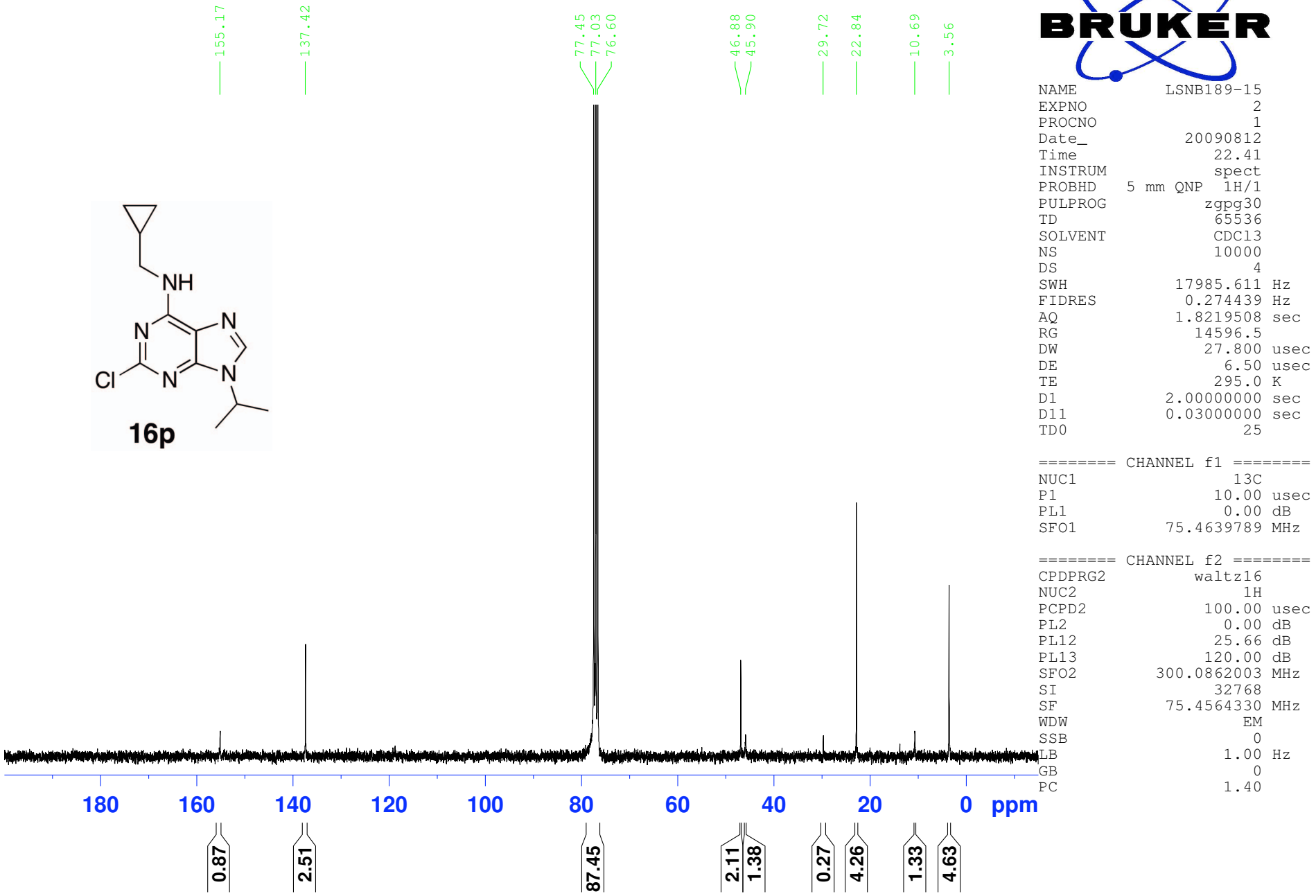
NAME	kb-NB-184-81
EXPNO	2
PROCNO	1
Date_	20090701
Time	20.55
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	254
DS	4
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	16384
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028149 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

LSNB189-15-CDCl3-301-1H



NAME	LSNB189-15-CDCl3-301-1H
EXPNO	15
PROCNO	1
Date_	20090817
Time	10.41
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	470
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	574.7
DW	81.000 usec
DE	6.50 usec
TE	295.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	5.21 usec
PL1	0.00 dB
SFO1	300.0868531 MHz
SI	32768
SF	300.0850000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

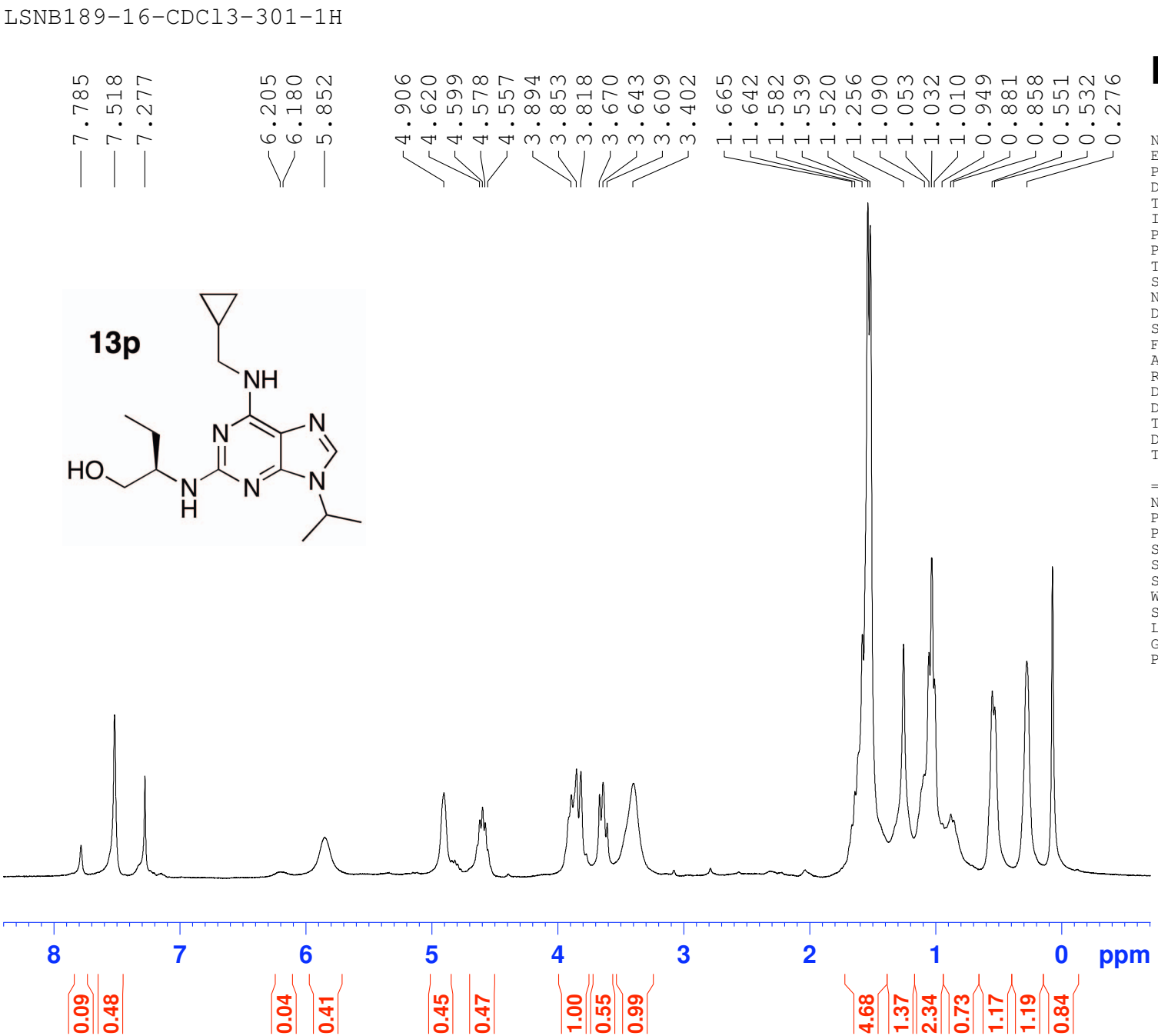
LSNB189-15 in CDCl3, 13C, 301, 8/12/09



BRUKER

NAME	LSNB189-15
EXPNO	2
PROCNO	1
Date_	20090812
Time	22.41
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	10000
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	14596.5
DW	27.800 usec
DE	6.50 usec
TE	295.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	25
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
SFO1	75.4639789 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	25.66 dB
PL13	120.00 dB
SFO2	300.0862003 MHz
SI	32768
SF	75.4564330 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

LSNB189-16-CDCl3-301-1H

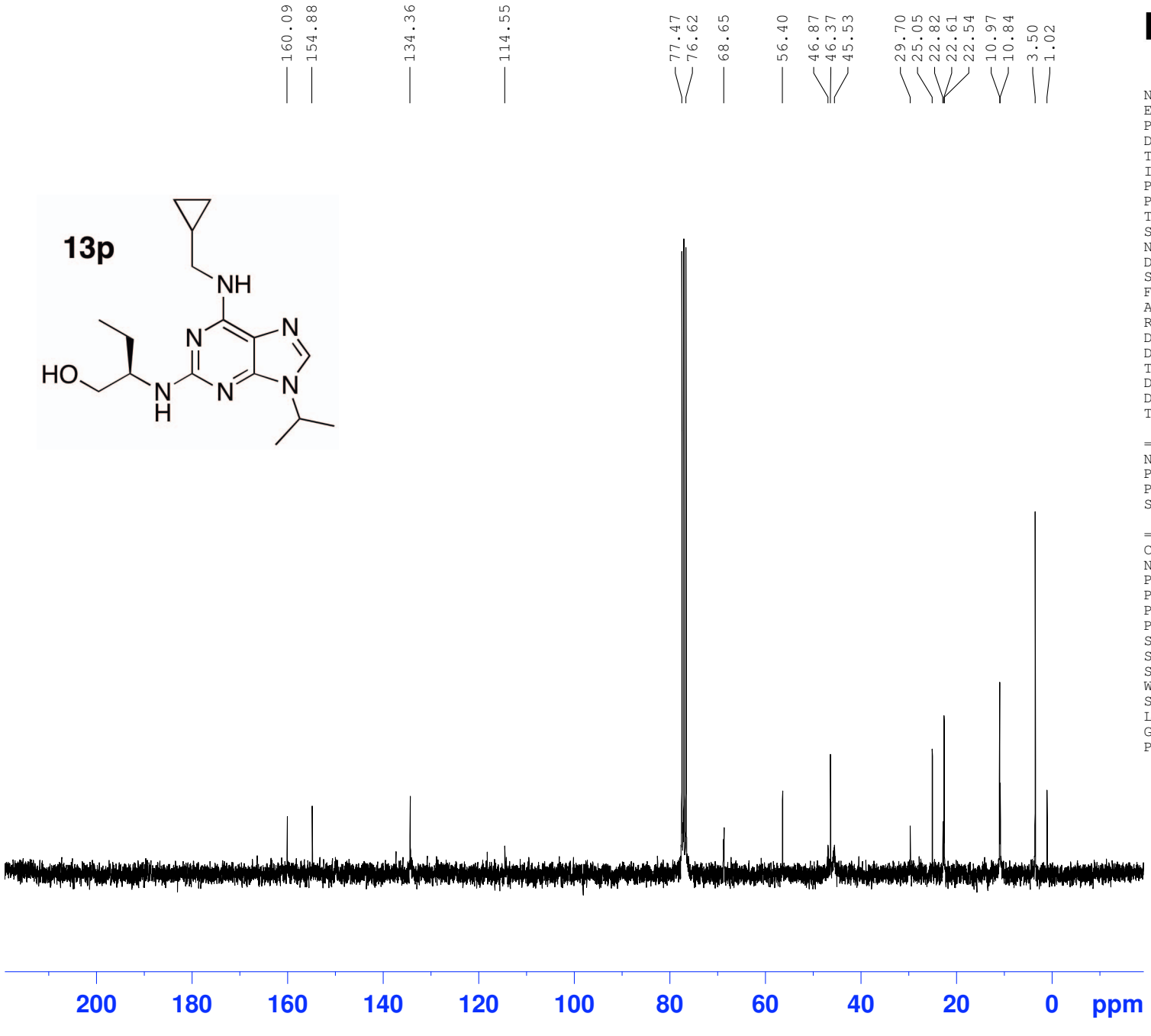


NAME	LSNB189-16-CDCl3-301-1H
EXPNO	16
PROCNO	1
Date_	20090818
Time	14.06
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	32
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	181
DW	81.000 usec
DE	6.50 usec
TE	294.9 K
D1	1.00000000 sec
TD0	1

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

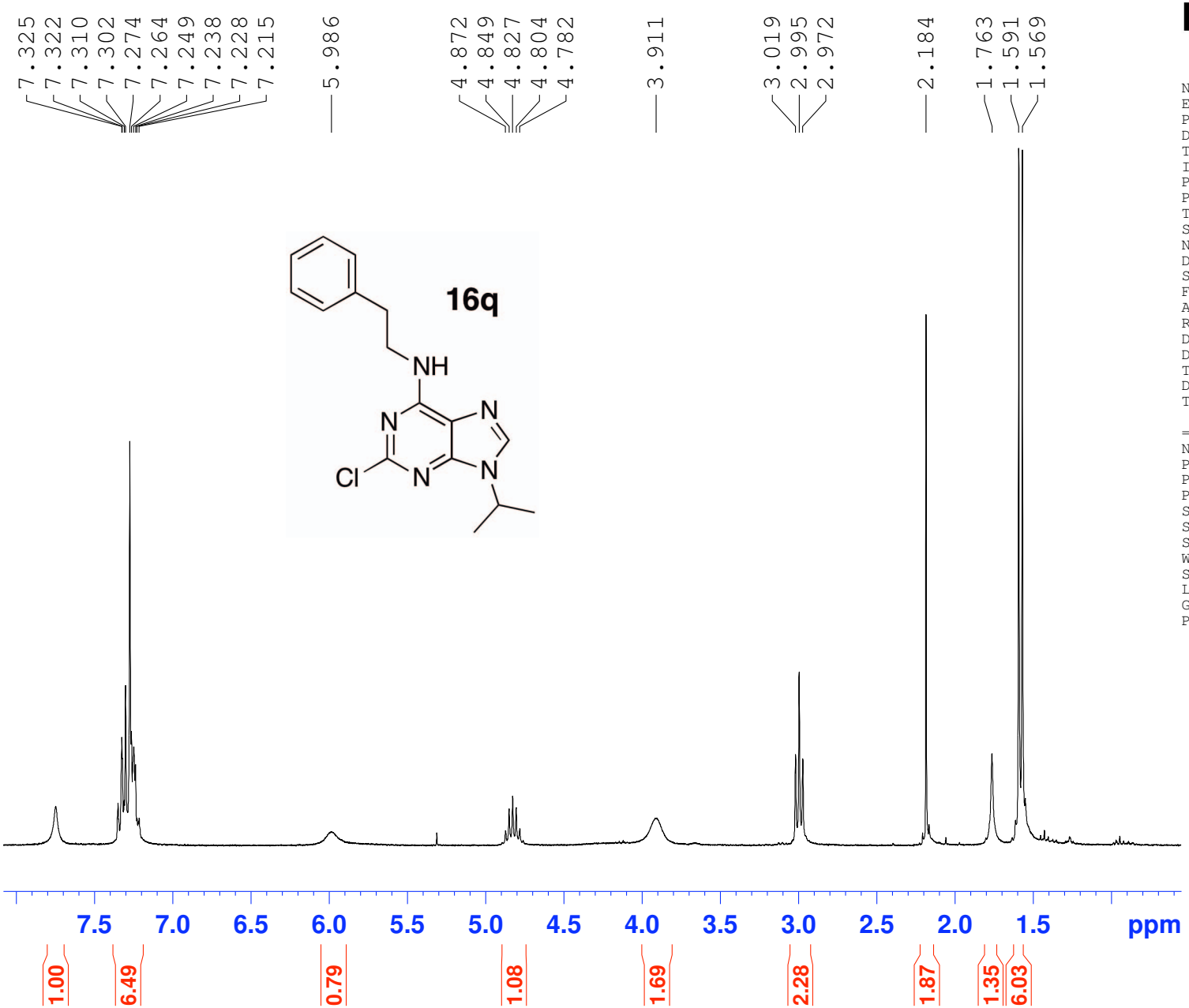
NUC1	1H
P1	5.21 usec
PL1	0.00 dB
SFO1	300.0868531 MHz
SI	32768
SF	300.0850000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

LSNB189-16-CDCl3-301-13C



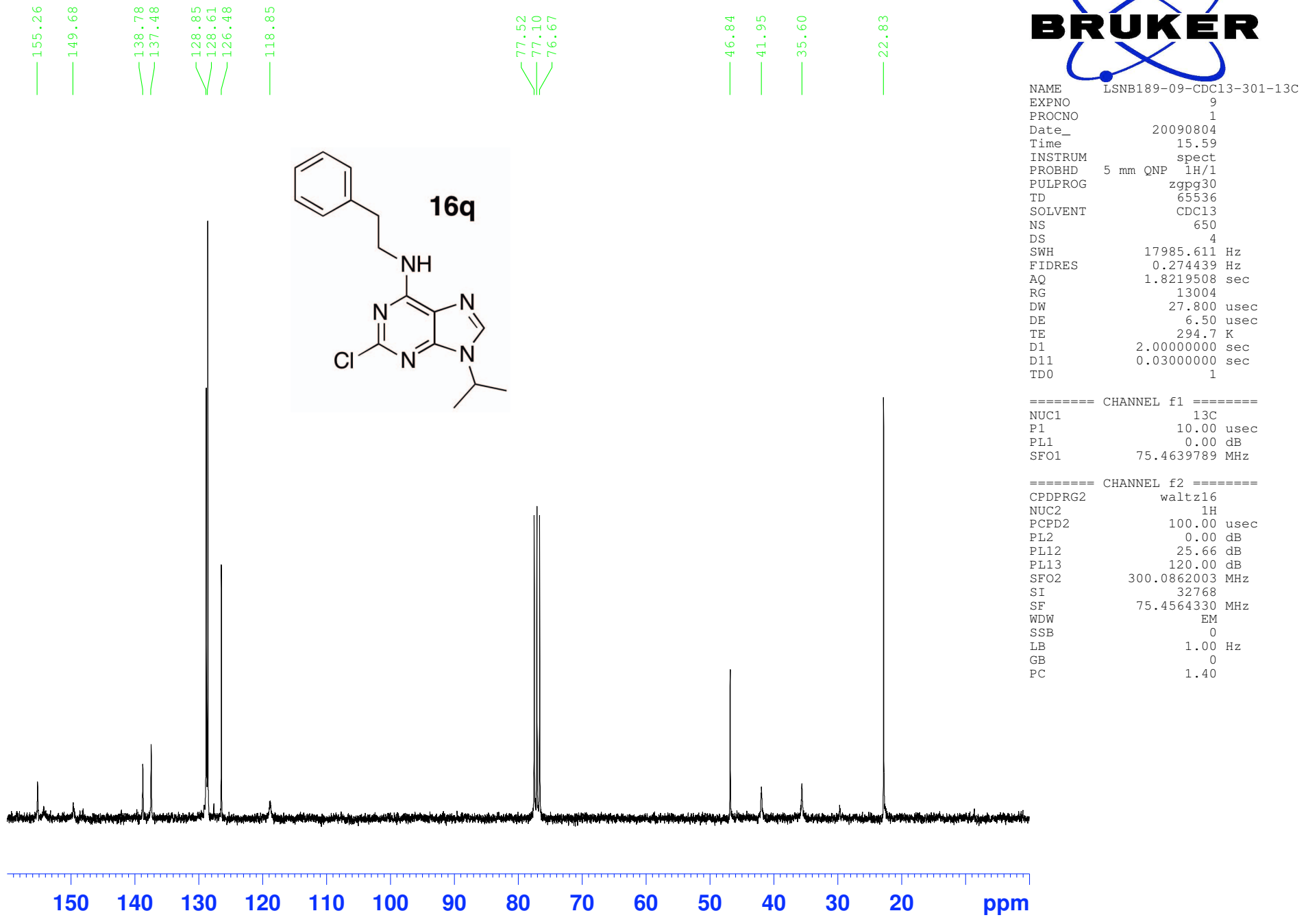
NAME	LSNB189-16-CDCl3-301-13C
EXPNO	16
PROCNO	1
Date_	20090818
Time	14.13
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	700
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	32768
DW	27.800 usec
DE	6.50 usec
TE	295.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
SFO1	75.4639789 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	25.66 dB
PL13	120.00 dB
SFO2	300.0862003 MHz
SI	32768
SF	75.4564330 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

LSNB189-09-301b-CDCl3-1H



NAME	LSNB189-09-301b-CDCl3-1H
EXPNO	9
PROCNO	1
Date_	20090720
Time	14.42
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	2
SWH	6188.119 Hz
FIDRES	0.188846 Hz
AQ	2.6477044 sec
RG	228
DW	80.800 usec
DE	6.50 usec
TE	292.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.20 usec
PL1	-2.00 dB
PL1W	18.20942116 W
SFO1	300.2318540 MHz
SI	32768
SF	300.2300000 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

LSNB189-09-CDCl3-301-13C

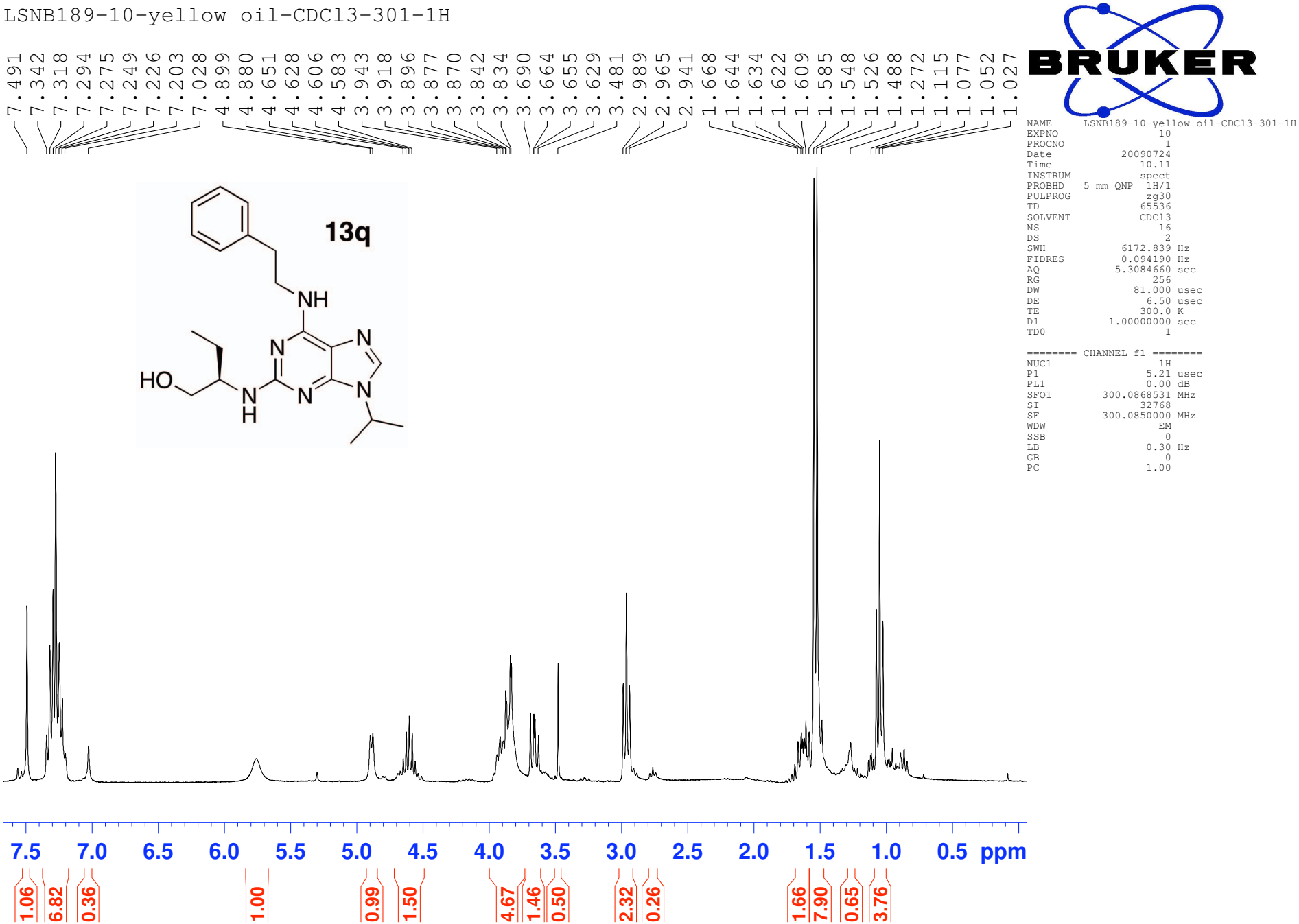



```
NAME          LSNB189-09-CDCl3-301-13C
EXPNO                                9
PROCNO                               1
Date_                         20090804
Time                             15.59
INSTRUM                          spect
PROBHD    5 mm QNP  1H/1
PULPROG                       zgpg30
TD                               65536
SOLVENT                       CDCl3
NS                                 650
DS                                   4
SWH                        17985.611 Hz
FIDRES                      0.274439 Hz
AQ                         1.8219508 sec
RG                               13004
DW                            27.800 usec
DE                             6.50 usec
TE                             294.7 K
D1                        2.00000000 sec
D11                       0.03000000 sec
TD0                                  1

======== CHANNEL f1 ========
NUC1                            13C
P1                           10.00 usec
PL1                           0.00 dB
SFO1                  75.4639789 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                            1H
PCPD2                       100.00 usec
PL2                           0.00 dB
PL12                         25.66 dB
PL13                        120.00 dB
SFO2                 300.0862003 MHz
SI                           32768
SF                    75.4564330 MHz
WDW                             EM
SSB                              0
LB                            1.00 Hz
GB                               0
PC                            1.40
```

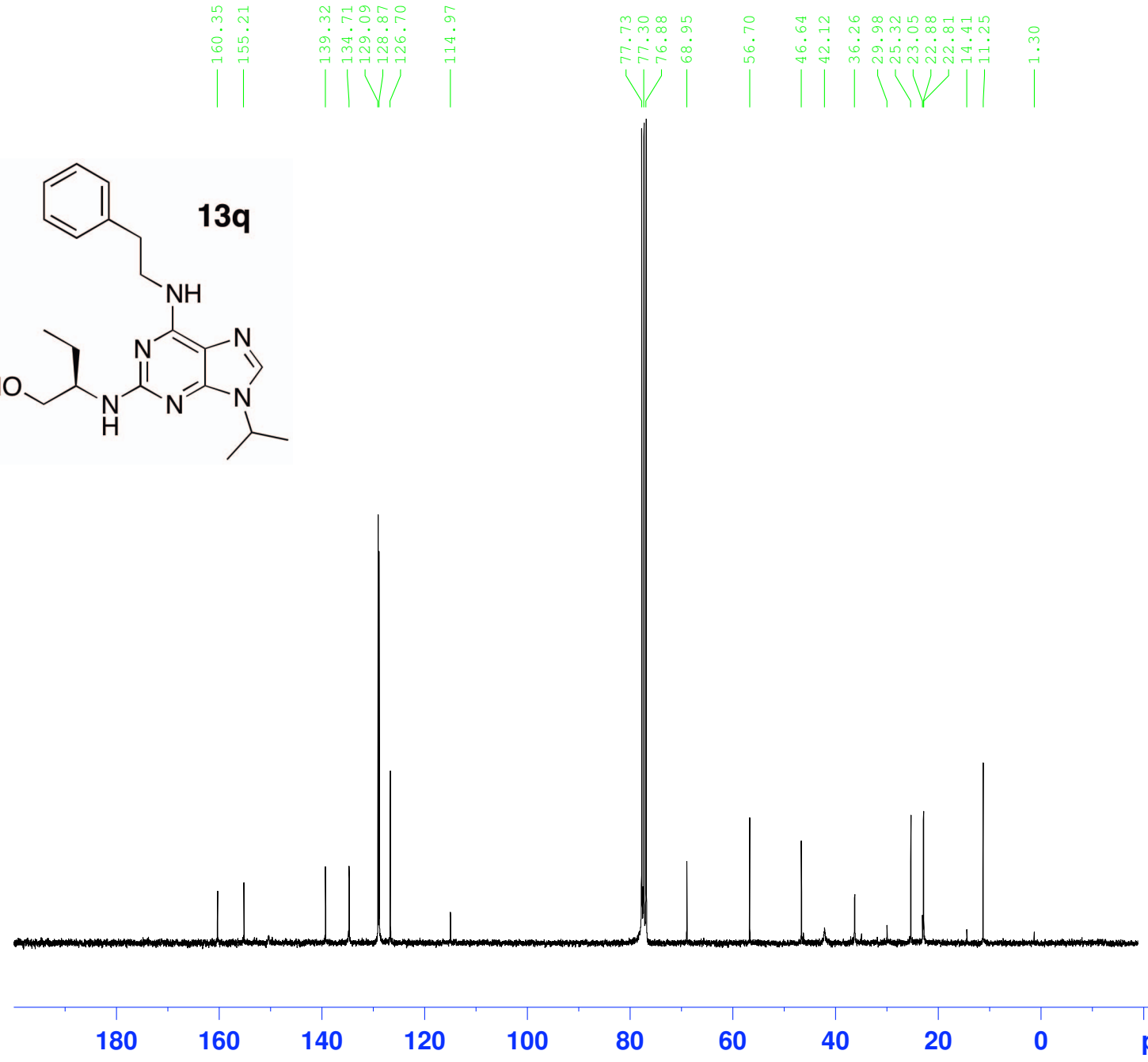
LSNB189-10-yellow oil-CDCl3-301-1H




```
NAME        LSNB189-10-yellow oil-CDCl3-301-1H
EXPNO                   10
PROCNO                   1
Date_             20090724
Time                 10.11
INSTRUM              spect
PROBHD    5 mm QNP  1H/1
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               6172.839 Hz
FIDRES               0.094190 Hz
AQ                5.3084660 sec
RG                     256
DW                  81.000 usec
DE                   6.50 usec
TE                  300.0 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   5.21 usec
PL1                  0.00 dB
SFO1           300.0868531 MHz
SI                   32768
SF             300.0850000 MHz
WDW                     EM
SSB                      0
LB                   0.30 Hz
GB                       0
PC                   1.00
```

LSNB189_compound in CDCl3, 13C, 301, 8/6/09



NAME	LSNB189_compound
EXPNO	2
PROCNO	1
Date_	20090806
Time	23.15
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	9000
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	32768
DW	27.800 usec
DE	6.50 usec
TE	294.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	10
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
SFO1	75.4639789 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	25.66 dB
PL13	120.00 dB
SFO2	300.0862003 MHz
SI	32768
SF	75.4564130 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

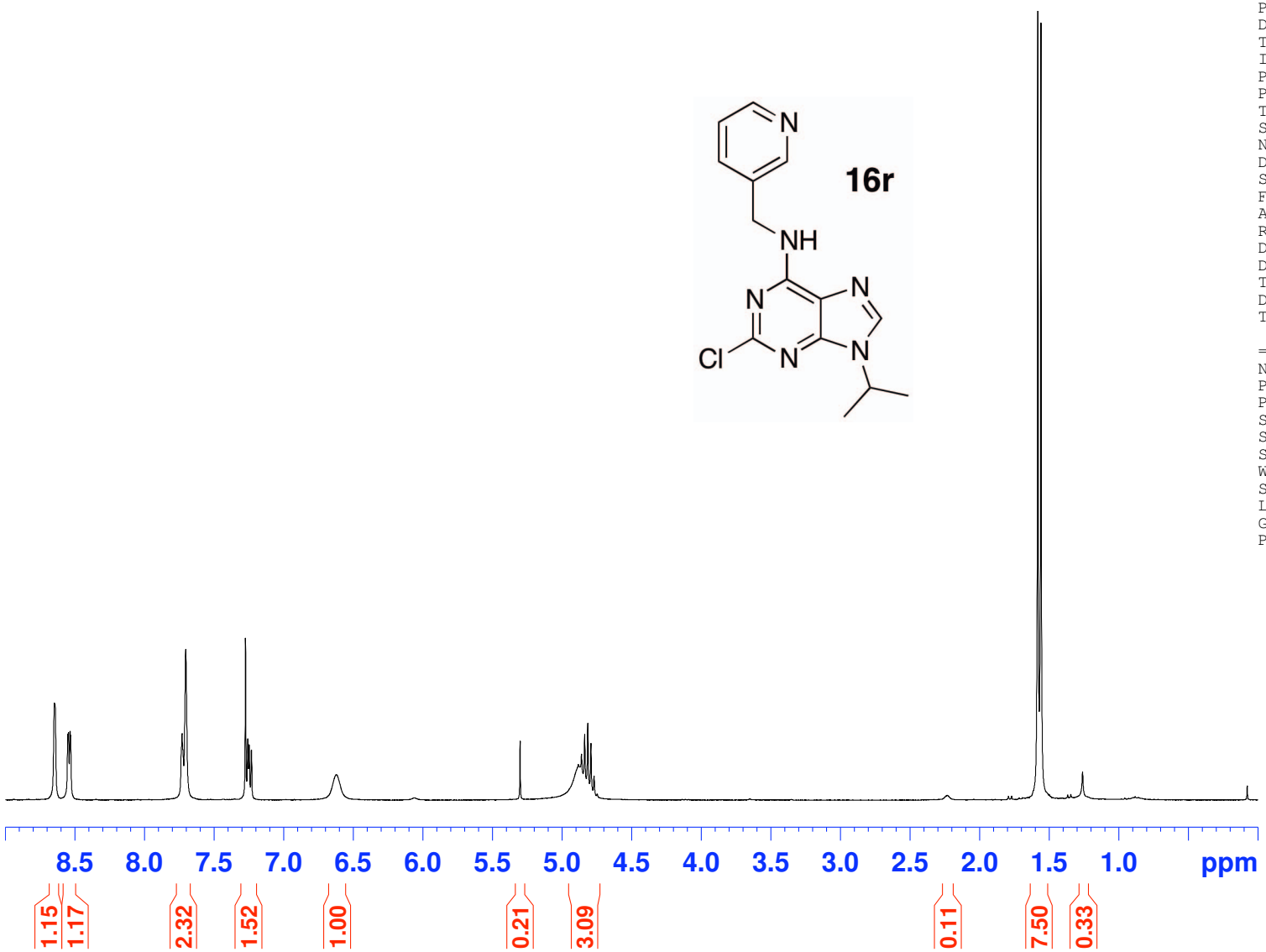
LSNB189-11-CDCl3-301-1H

8.549 8.545 8.534 8.530 7.732 7.726 7.705 7.276 7.260 7.250 7.234 6.624

5.302 4.884 4.861 4.838 4.816 4.793 4.770

2.235

1.581 1.559 1.259

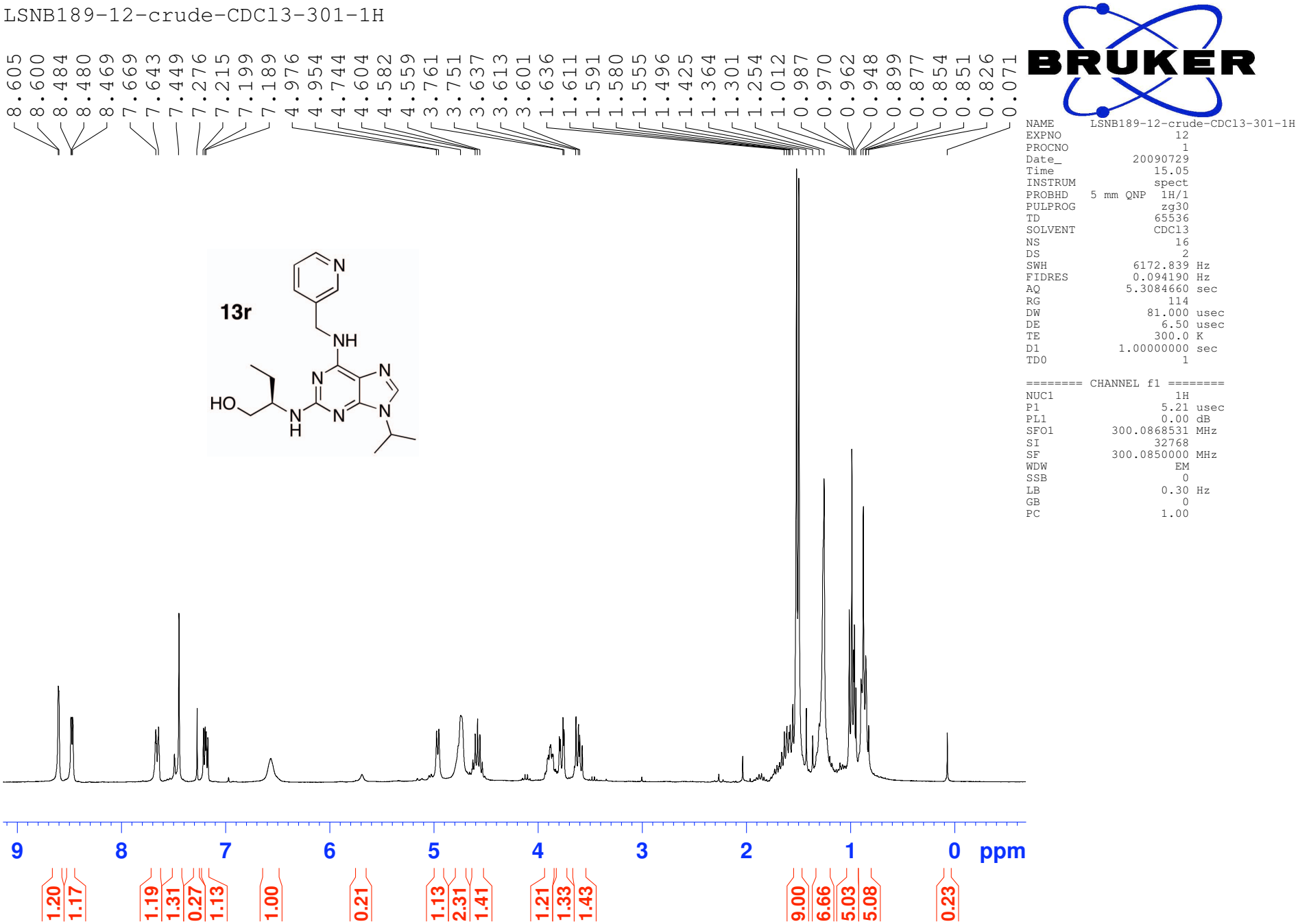


NAME	LSNB189-11-CDCl3-301-1H
EXPNO	11
PROCNO	1
Date_	20090729
Time	15.24
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	362
DW	81.000 usec
DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

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

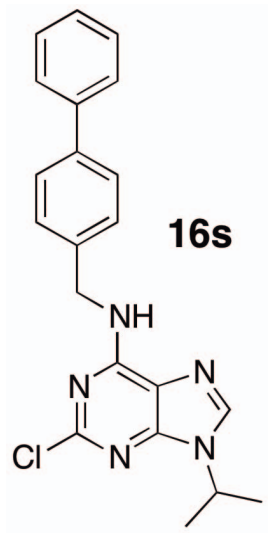
NUC1	1H
P1	5.21 usec
PL1	0.00 dB
SFO1	300.0868531 MHz
SI	32768
SF	300.0850000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

LSNB189-12-crude-CDCl3-301-1H



NAME	LSNB189-12-crude-CDCl3-301-1H
EXPNO	12
PROCNO	1
Date_	20090729
Time	15.05
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	114
DW	81.000 usec
DE	6.50 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	5.21 usec
PL1	0.00 dB
SFO1	300.0868531 MHz
SI	32768
SF	300.0850000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

kb-NB-184-85



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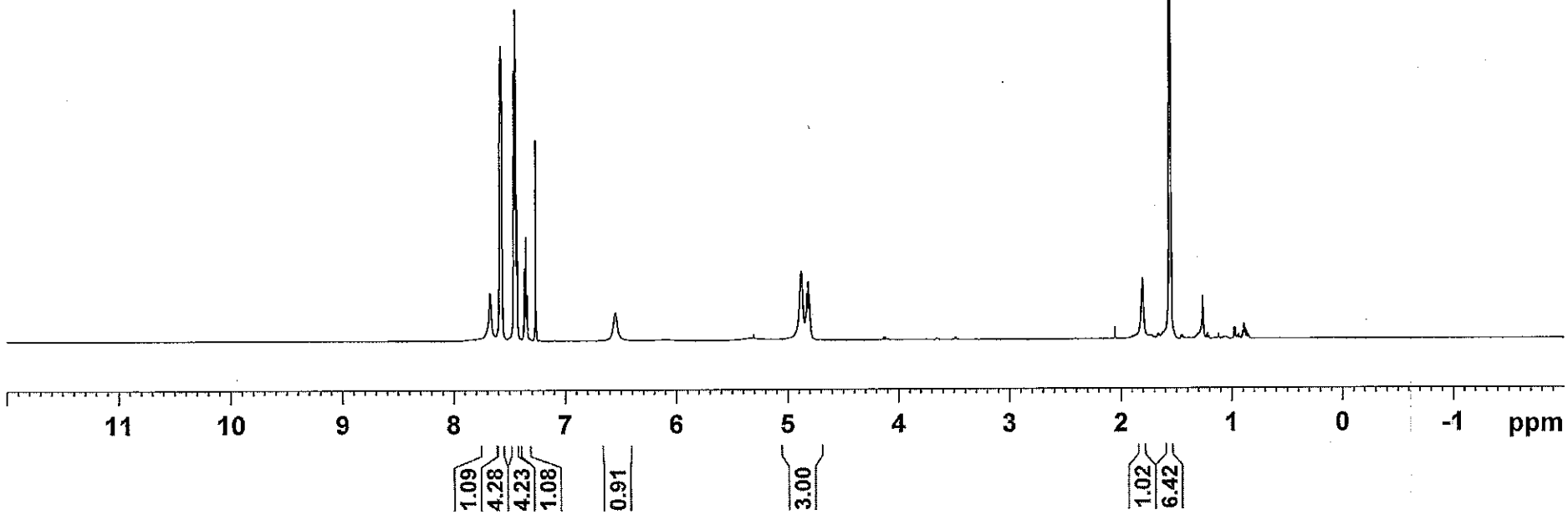
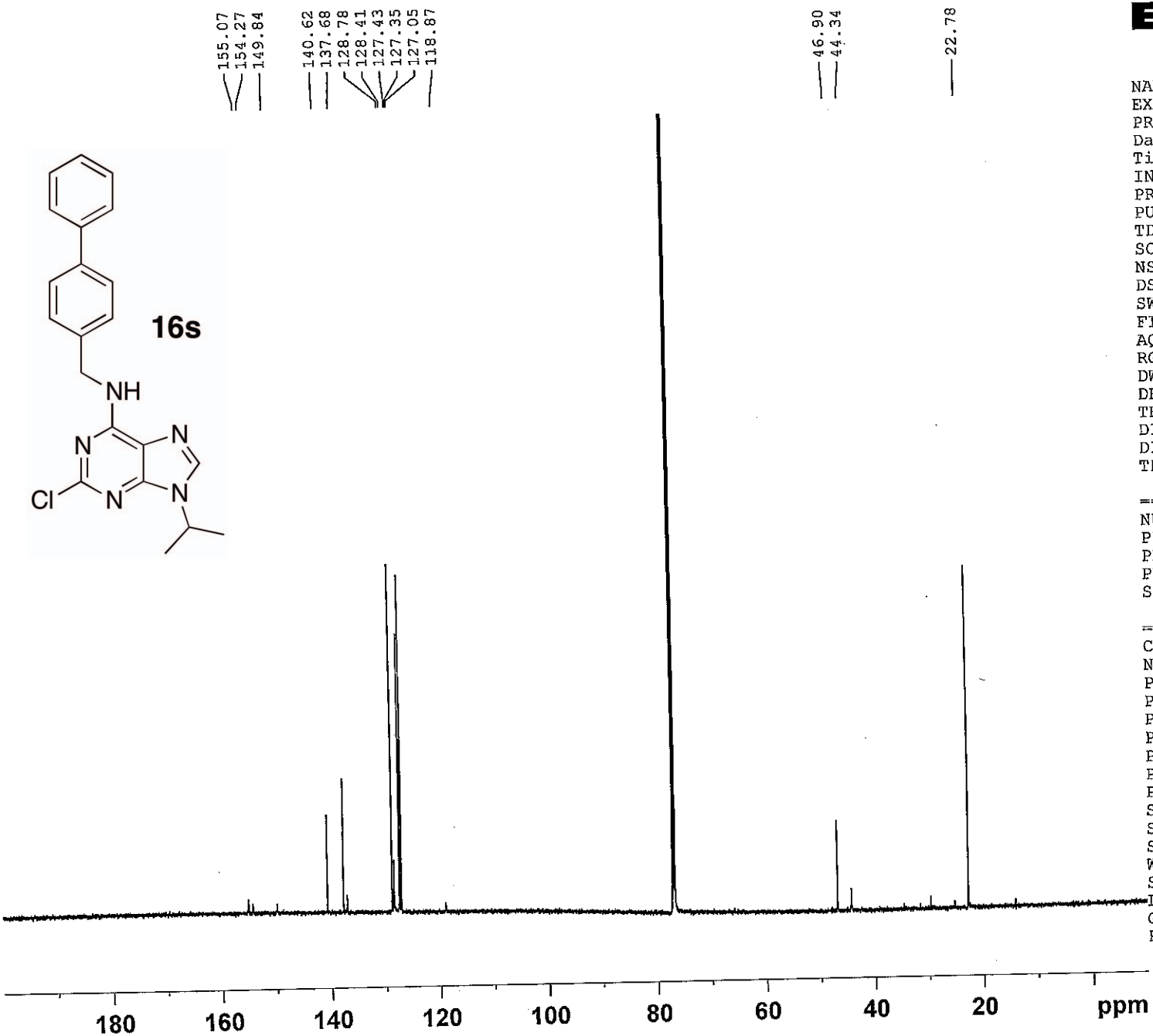
7.346
7.345
6.552
4.881
4.841
4.830
4.819
4.808
4.797

1.809
1.565
1.554



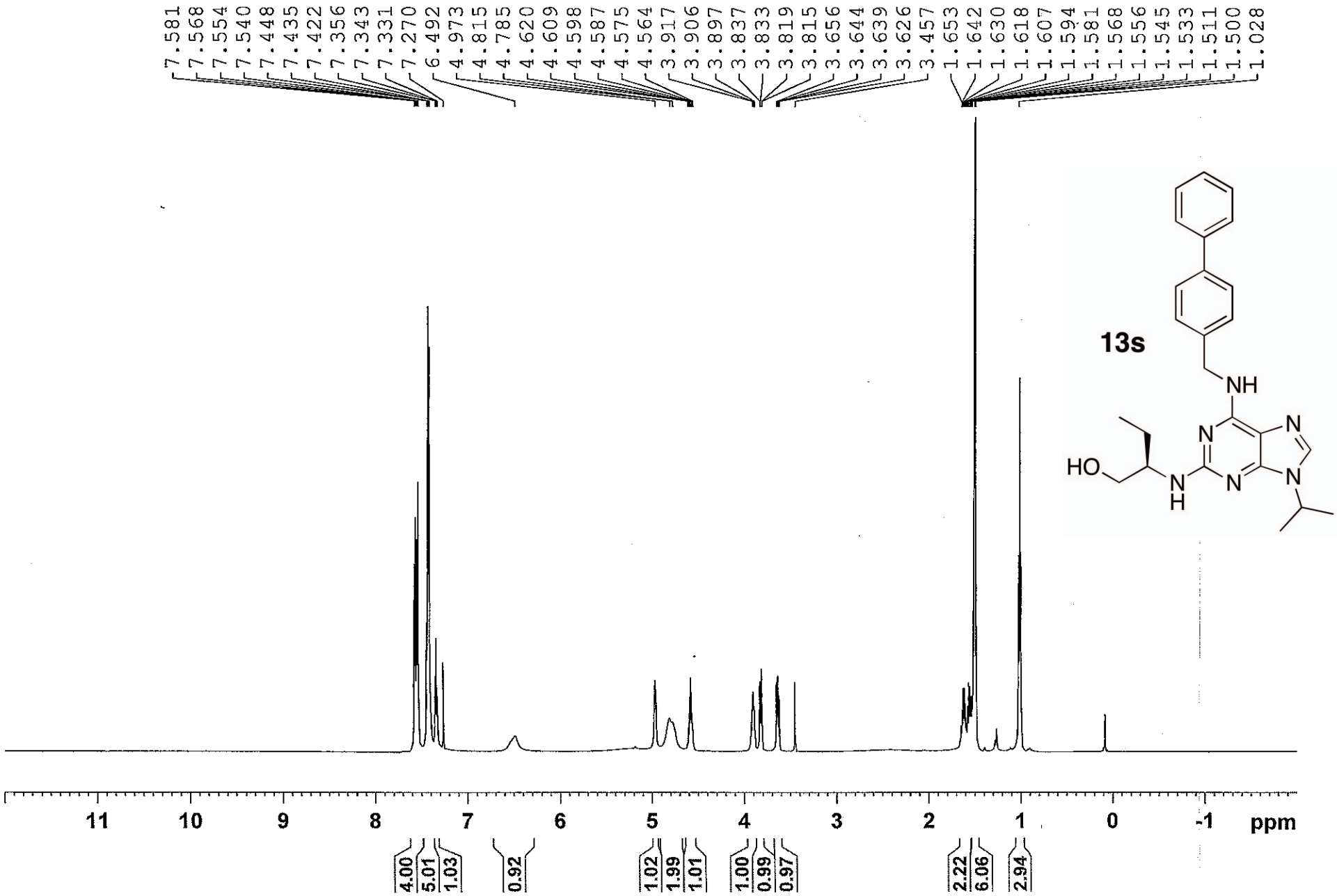
kb-NB-184-85



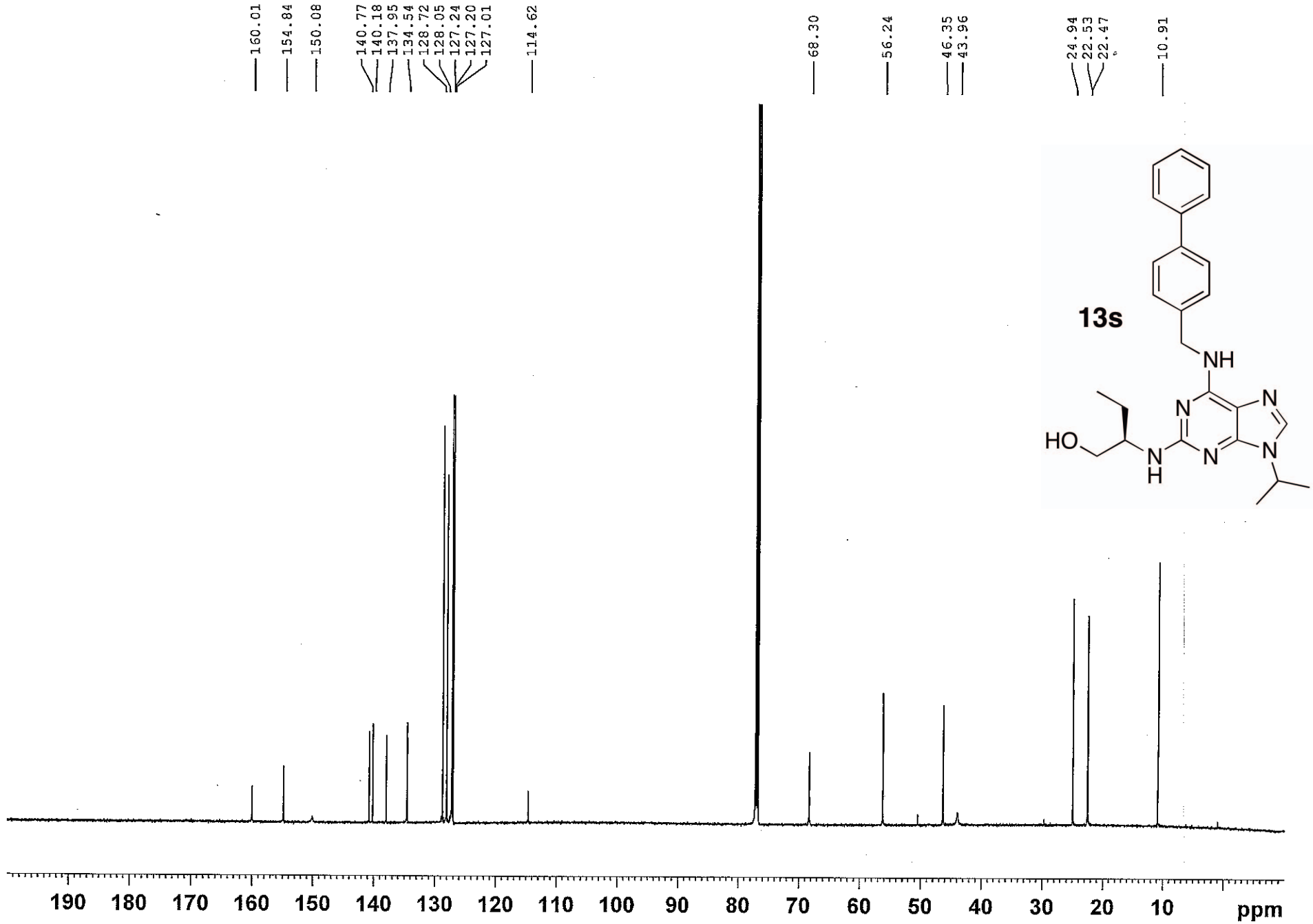
BRUKER

NAME	kb-NB-184-85
EXPNO	3
PROCNO	1
Date_	20090702
Time	20.55
INSTRUM	spect
PROBHD	5 mm CPTCI 1H-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	35971.223 Hz
FIDRES	0.548877 Hz
AQ	0.9110143 sec
RG	18390.4
DW	13.900 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.00 usec
PL1	-0.70 dB
PL1W	82.63385773 W
SFO1	150.9178988 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	4.00 dB
PL12	24.00 dB
PL13	27.00 dB
PL2W	7.00000000 W
PL12W	0.07000000 W
PL13W	0.03508311 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028137 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

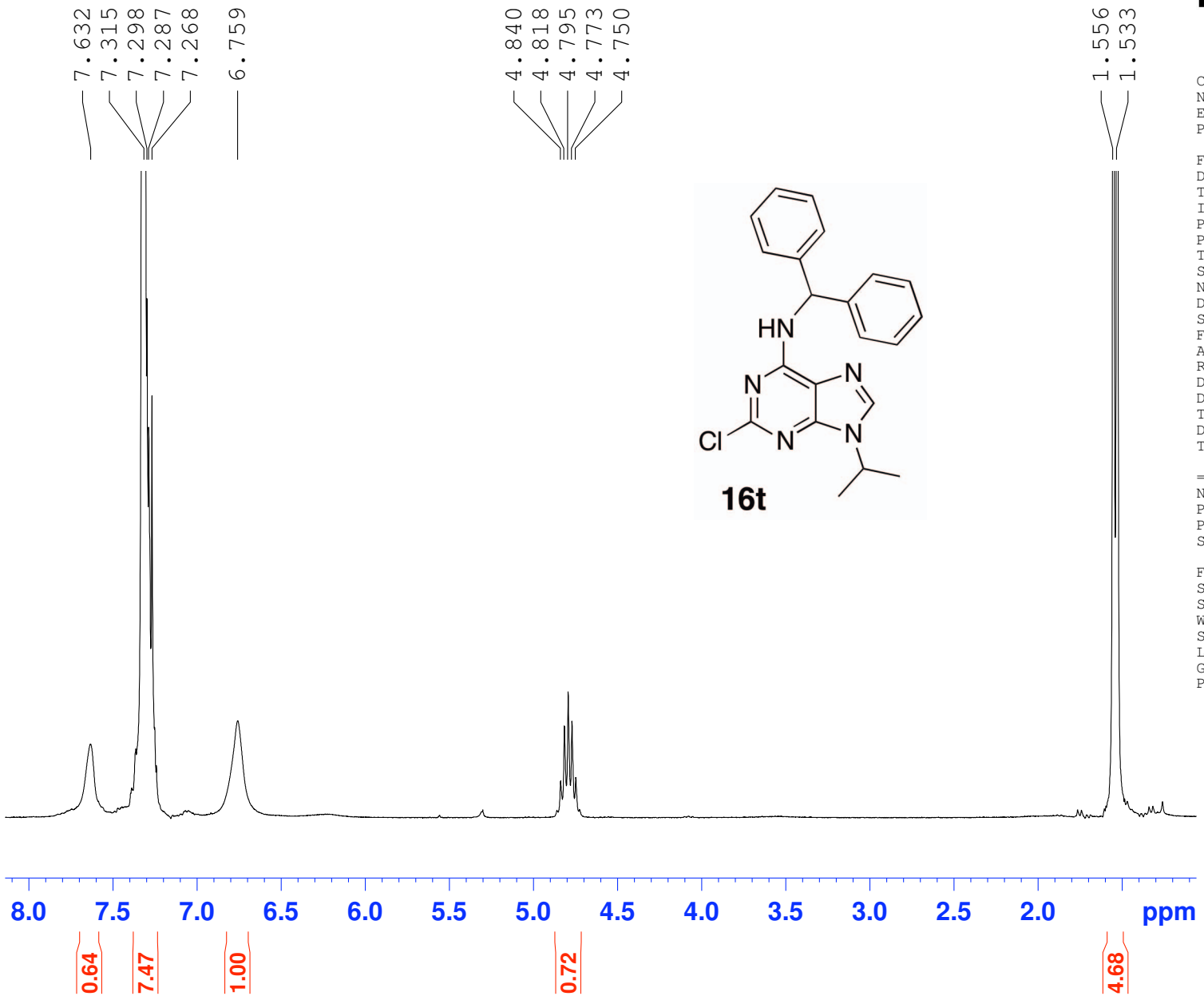
kb-NB-184-91


7.581
7.568
7.554
7.540
7.448
7.435
7.422
7.356
7.343
7.331
7.270
6.492
4.973
4.815
4.785
4.620
4.609
4.598
4.587
4.575
4.564
3.917
3.906
3.897
3.837
3.833
3.819
3.815
3.656
3.644
3.639
3.626
3.457
1.653
1.642
1.630
1.618
1.607
1.594
1.581
1.568
1.556
1.545
1.533
1.511
1.500
1.028
13s
NH
N
N
N
HO
N
H
N
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
4.00
5.01
1.03
0.92
1.02
1.99
1.01
1.00
0.99
0.97
2.22
6.06
2.94

kb-NB-184-91


160.01
154.84
150.08
140.77
140.18
137.95
134.54
128.72
128.05
127.24
127.20
127.01
114.62
68.30
56.24
46.35
43.96
24.94
22.53
22.47
10.91
13s
NH
N
N
N
N
HO
N
H
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

LSNB189-13-CDCl3-301a-1H



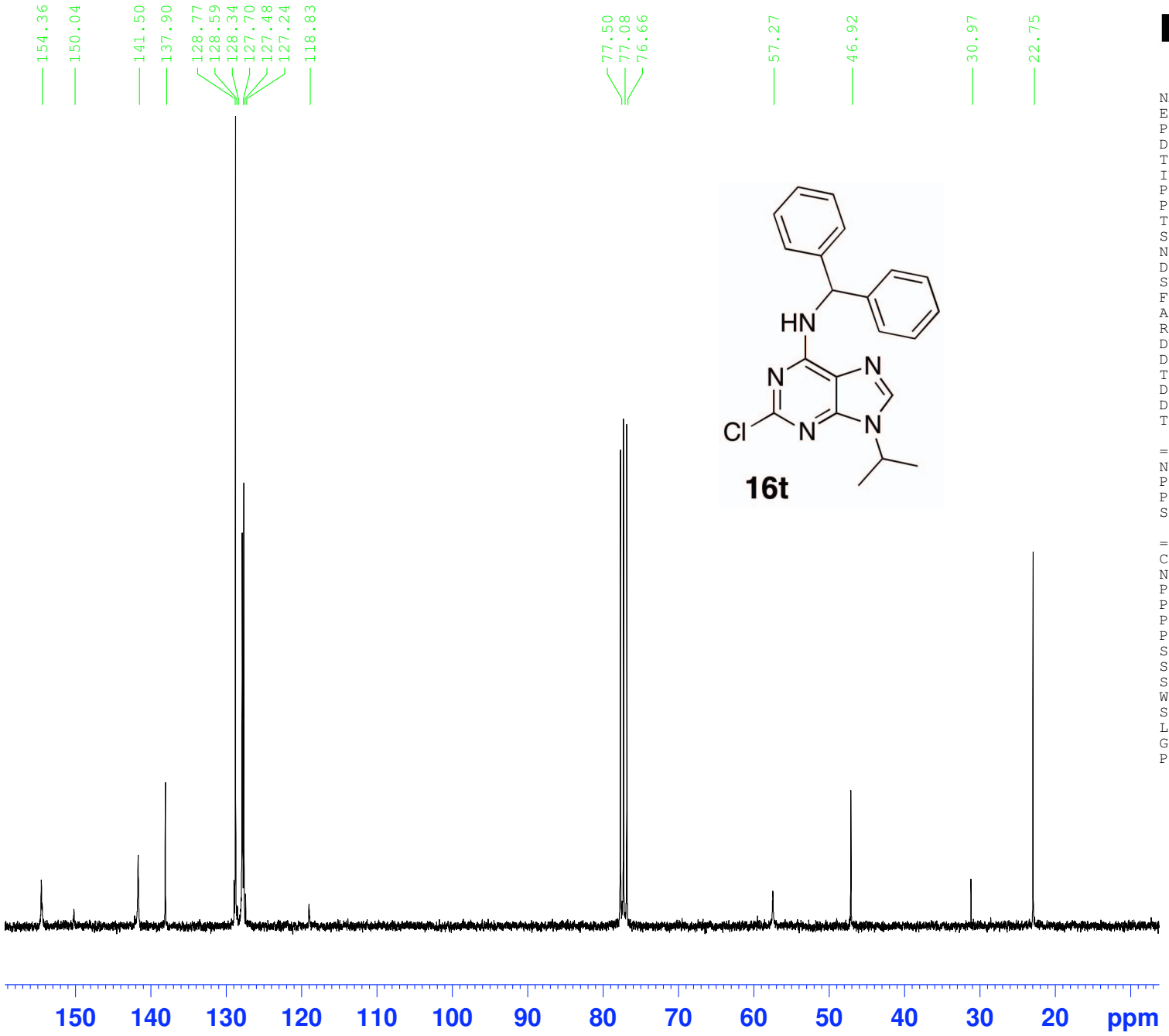
Current Data Parameters
NAME LSNB189-13-CDCl3-301a-1H
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090731
Time 17.40
INSTRUM spect
PROBHD 5 mm DUL 1H-13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 6218.905 Hz
FIDRES 0.189786 Hz
AQ 2.6345973 sec
RG 181
DW 80.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 1.00 dB
SFO1 300.3818550 MHz

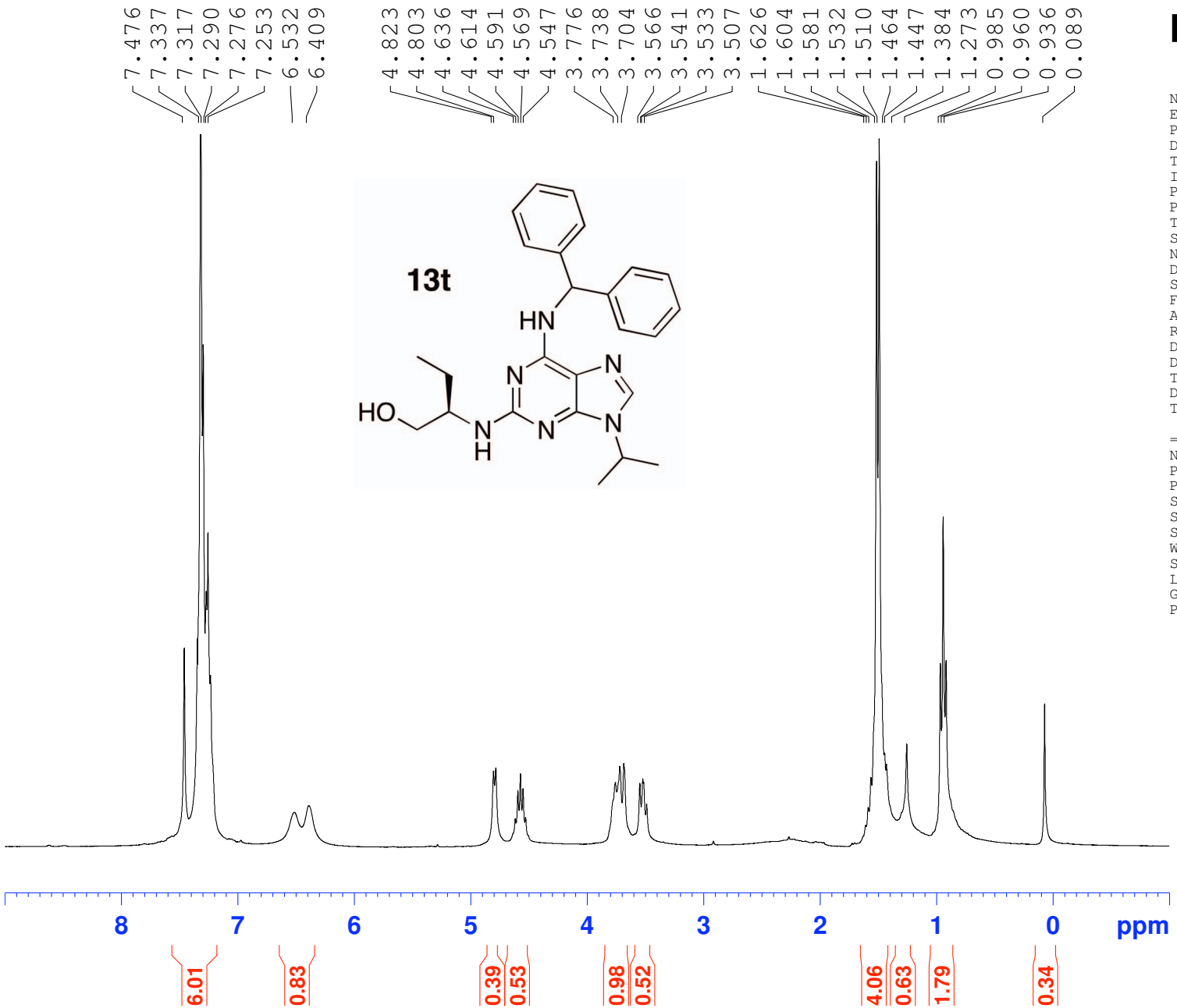
F2 - Processing parameters
SI 16384
SF 300.3800000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

LSNB189-13-CDCl3-301-13C



NAME	LSNB189-13-CDCl3-301-13C
EXPNO	13
PROCNO	1
Date_	20090810
Time	15.01
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	870
DS	4
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219508 sec
RG	16384
DW	27.800 usec
DE	6.50 usec
TE	295.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	0.00 dB
SFO1	75.4639789 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	25.66 dB
PL13	120.00 dB
SFO2	300.0862003 MHz
SI	32768
SF	75.4564172 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

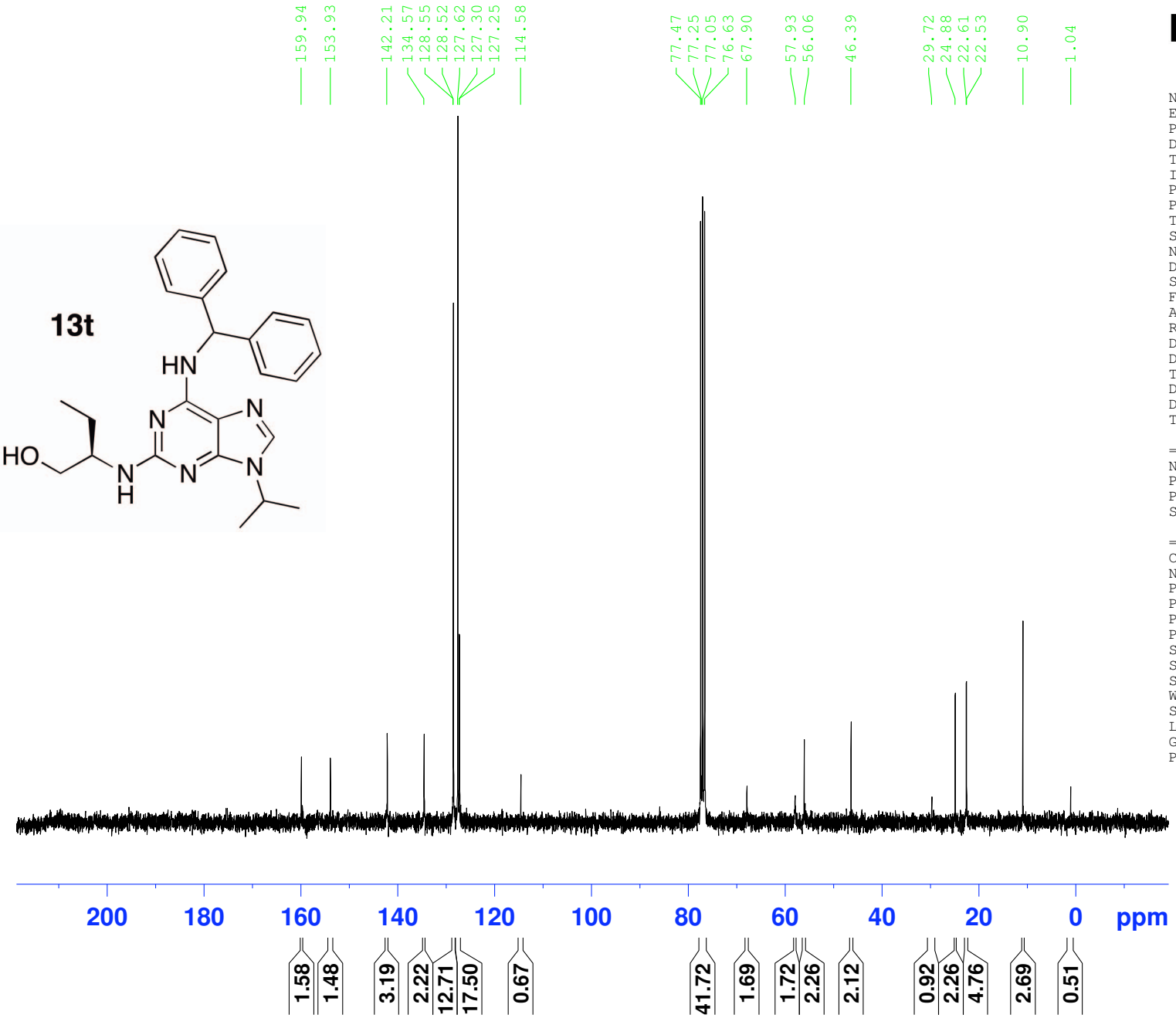
LSNB189-14-CDCl3-301-1H



BRUKER

NAME	LSNB189-14-CDCl3-301-13C
EXPNO	14
PROCNO	1
Date_	20090811
Time	15.55
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	450
DS	2
SWH	6172.839 Hz
FIDRES	0.094190 Hz
AQ	5.3084660 sec
RG	256
DW	81.000 usec
DE	6.50 usec
TE	300.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	5.21 usec
PL1	0.00 dB
SFO1	300.0868531 MHz
SI	32768
SF	300.0850050 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

LSNB189-14-CDCl3-301-13C

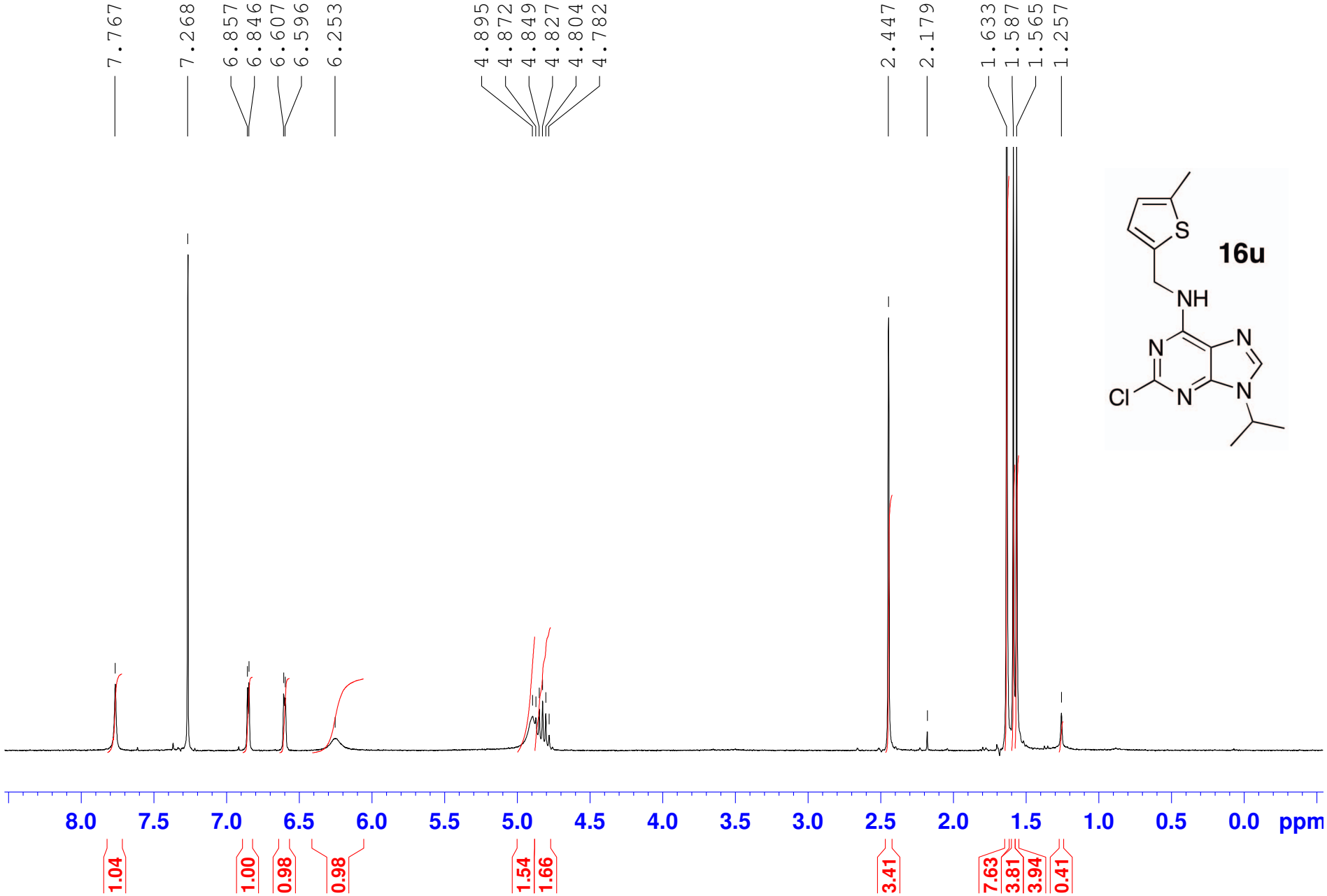


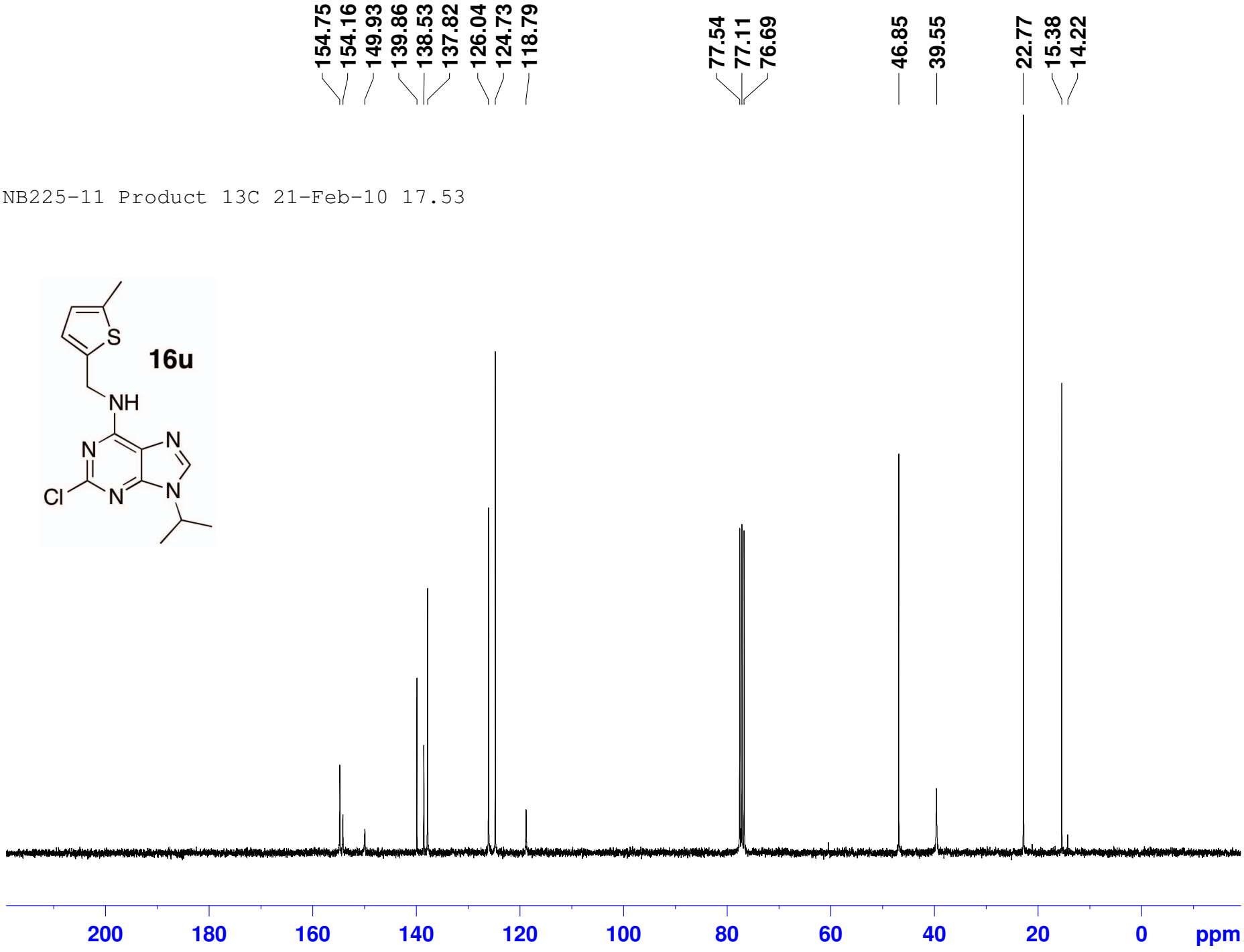

```
NAME          LSNB189-14-CDCl3-301-13C
EXPNO                               14
PROCNO                               1
Date_                         20090812
Time                             13.59
INSTRUM                          spect
PROBHD    5 mm QNP  1H/1
PULPROG                       zgpg30
TD                                65536
SOLVENT                         CDCl3
NS                                  780
DS                                    4
SWH                        17985.611 Hz
FIDRES                      0.274439 Hz
AQ                         1.8219508 sec
RG                           14596.5
DW                            27.800 usec
DE                             6.50 usec
TE                            294.8 K
D1                       2.00000000 sec
D11                      0.03000000 sec
TD0                                  1

======== CHANNEL f1 ========
NUC1                             13C
P1                            10.00 usec
PL1                            0.00 dB
SFO1                    75.4639789 MHz

======== CHANNEL f2 ========
CPDPRG2                     waltz16
NUC2                             1H
PCPD2                        100.00 usec
PL2                            0.00 dB
PL12                          25.66 dB
PL13                         120.00 dB
SFO2                   300.0862003 MHz
SI                               32768
SF                      75.4564330 MHz
WDW                                EM
SSB                                 0
LB                             1.00 Hz
GB                                  0
PC                             1.40
```

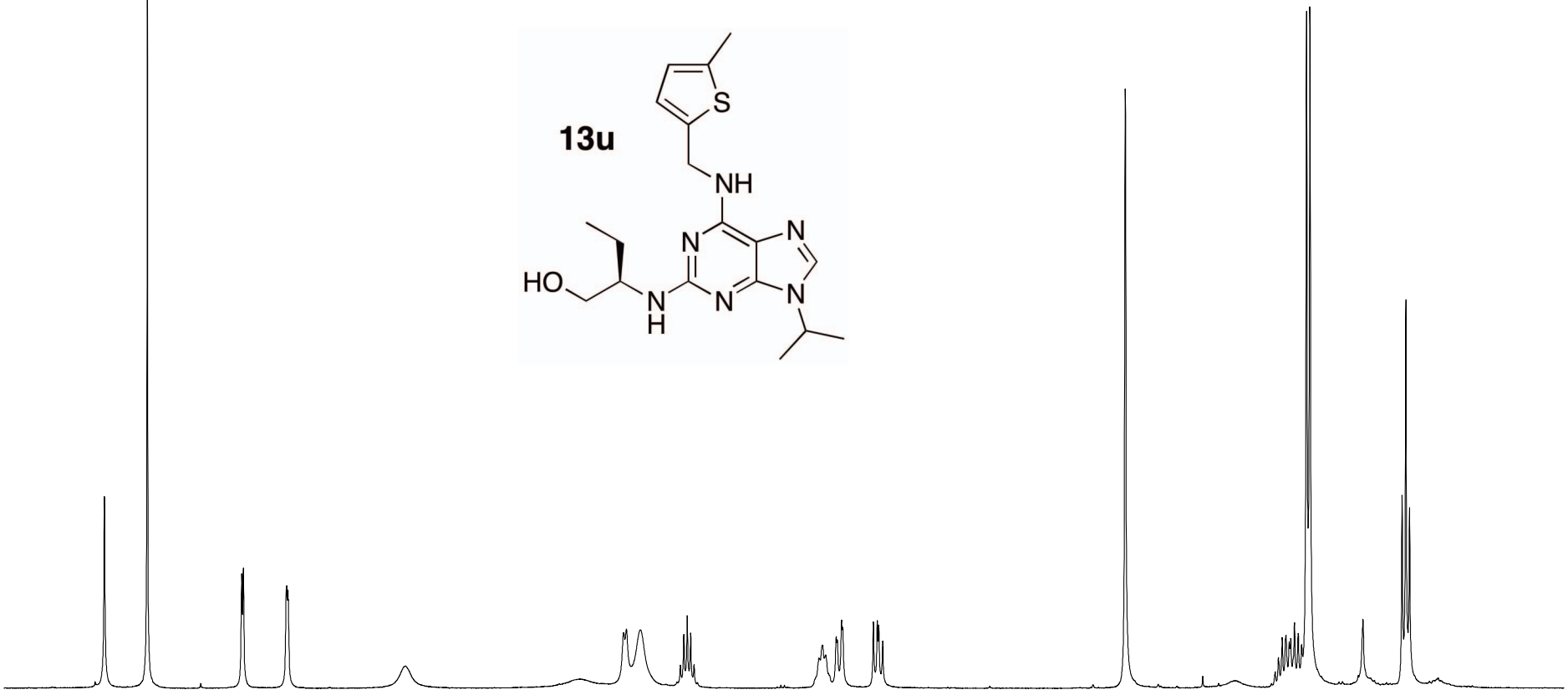
ml.282.10 in CDCl3, 1H, 301a, 7/5/10




154.75
154.16
149.93
139.86
138.53
137.82
126.04
124.73
118.79
77.54
77.11
76.69
46.85
39.55
22.77
15.38
14.22
NB225-11 Product 13C 21-Feb-10 17.53
16u
S
NH
N
N
N
N
Cl
200
180
160
140
120
100
80
60
40
20
0
ppm

ml282-50_Product in CDCl3, 1H, 400b, 1/7/11

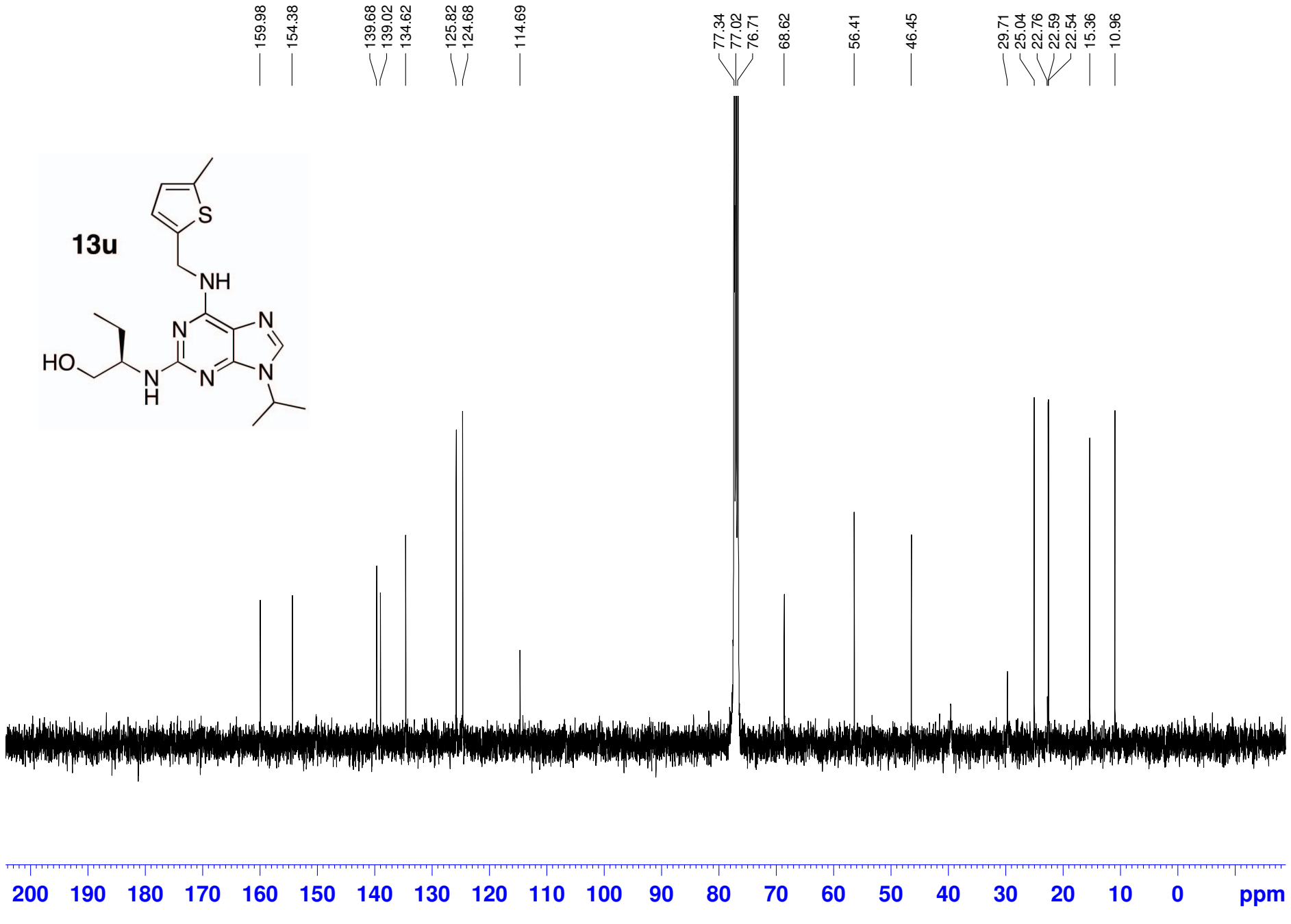
7.472 7.260 6.794 6.785 6.574 6.571 6.568 6.565 5.983 5.223 5.122 4.905 4.891 4.821 4.624 4.607 4.590 4.573 4.556 3.939 3.921 3.906 3.853 3.848 3.827 3.822 3.670 3.650 3.643 3.624 2.424 1.685 1.669 1.650 1.631 1.616 1.608 1.589 1.571 1.554 1.530 1.513 1.251 1.134 1.058 1.039 1.020

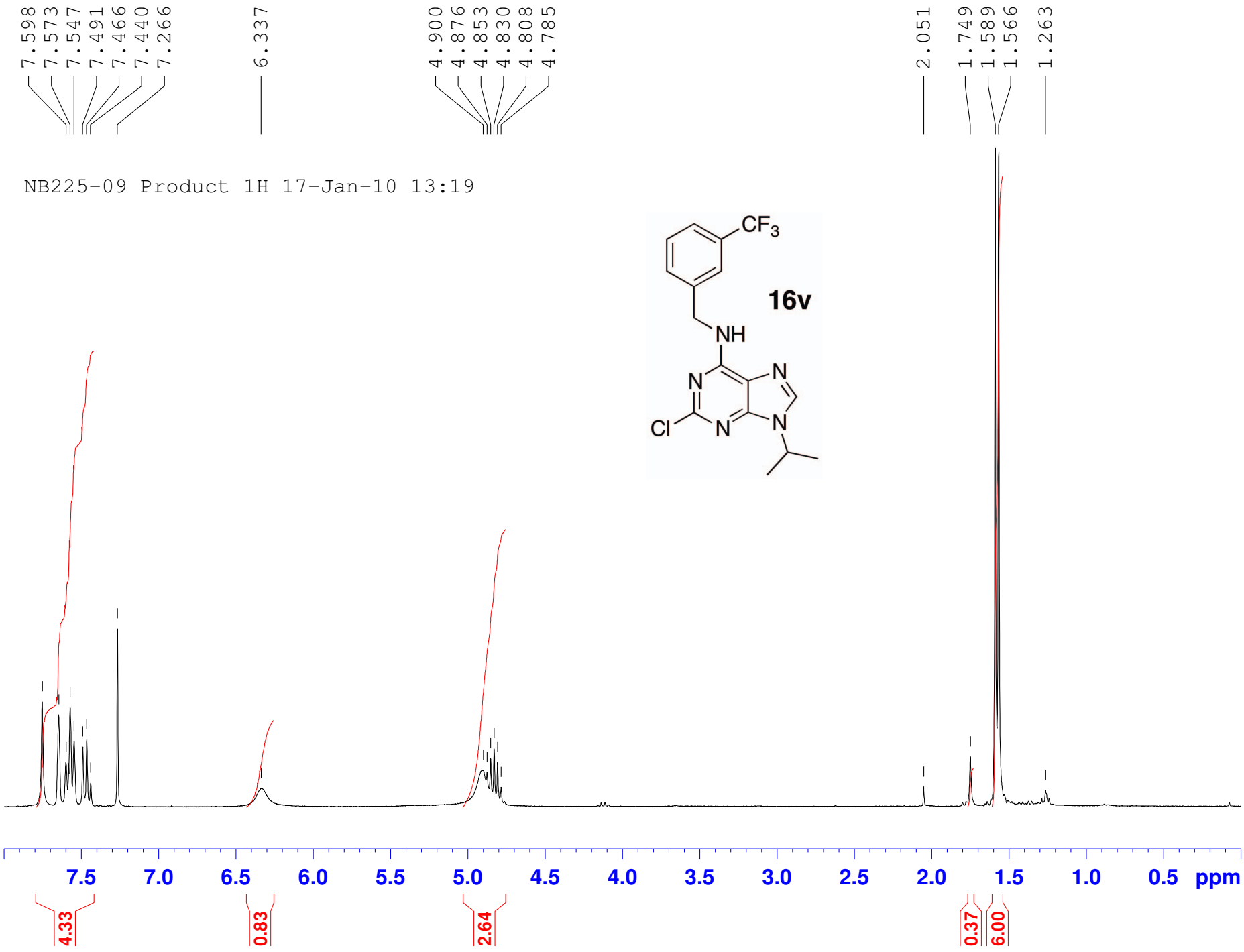


7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm

1.00 1.01 1.00 0.95 0.81 3.28 1.14 1.09 1.09 1.14 3.30 2.18 6.55 3.29

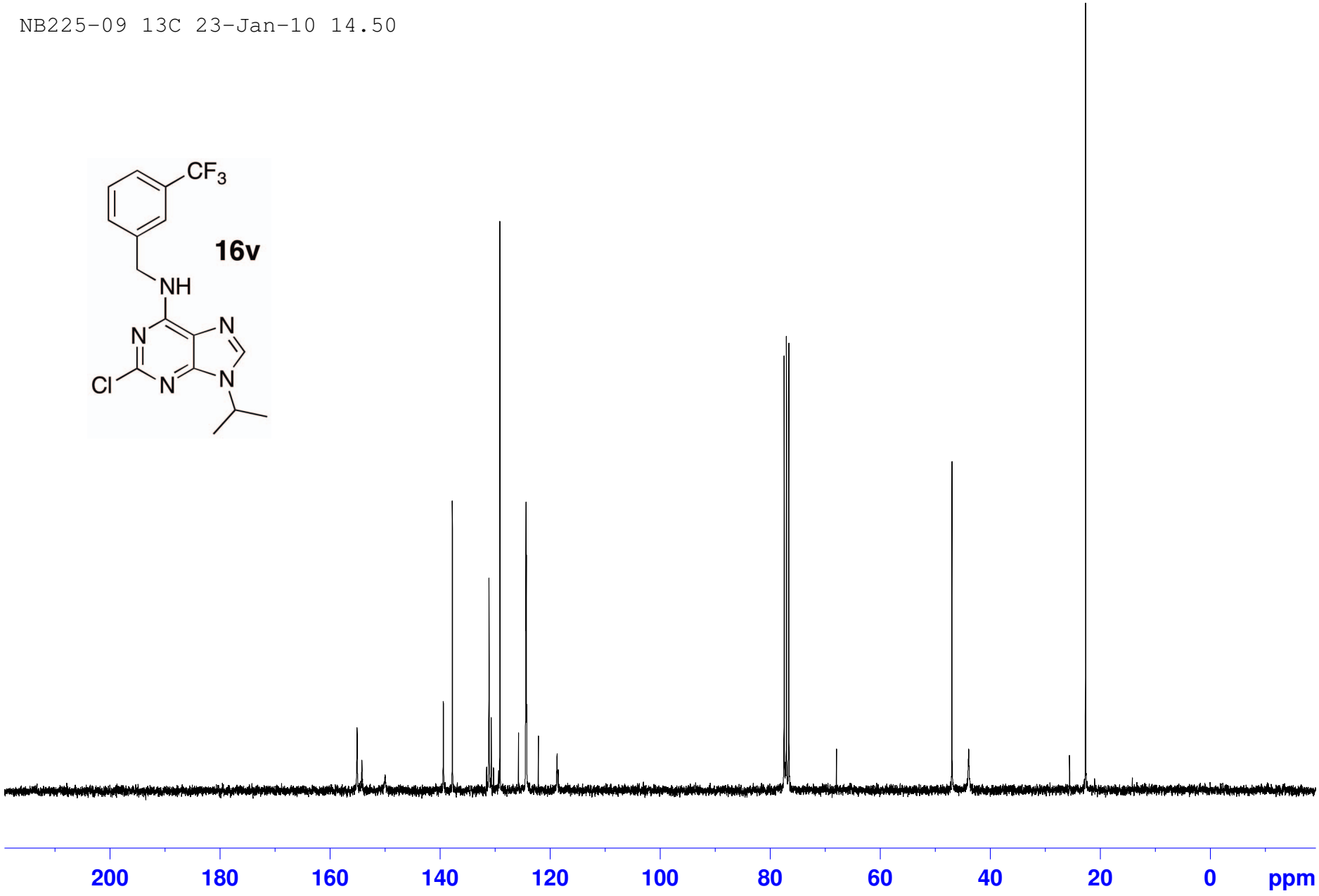
ml.282.11_f14 in CDCl3, 13C, 400a, 7/13/10

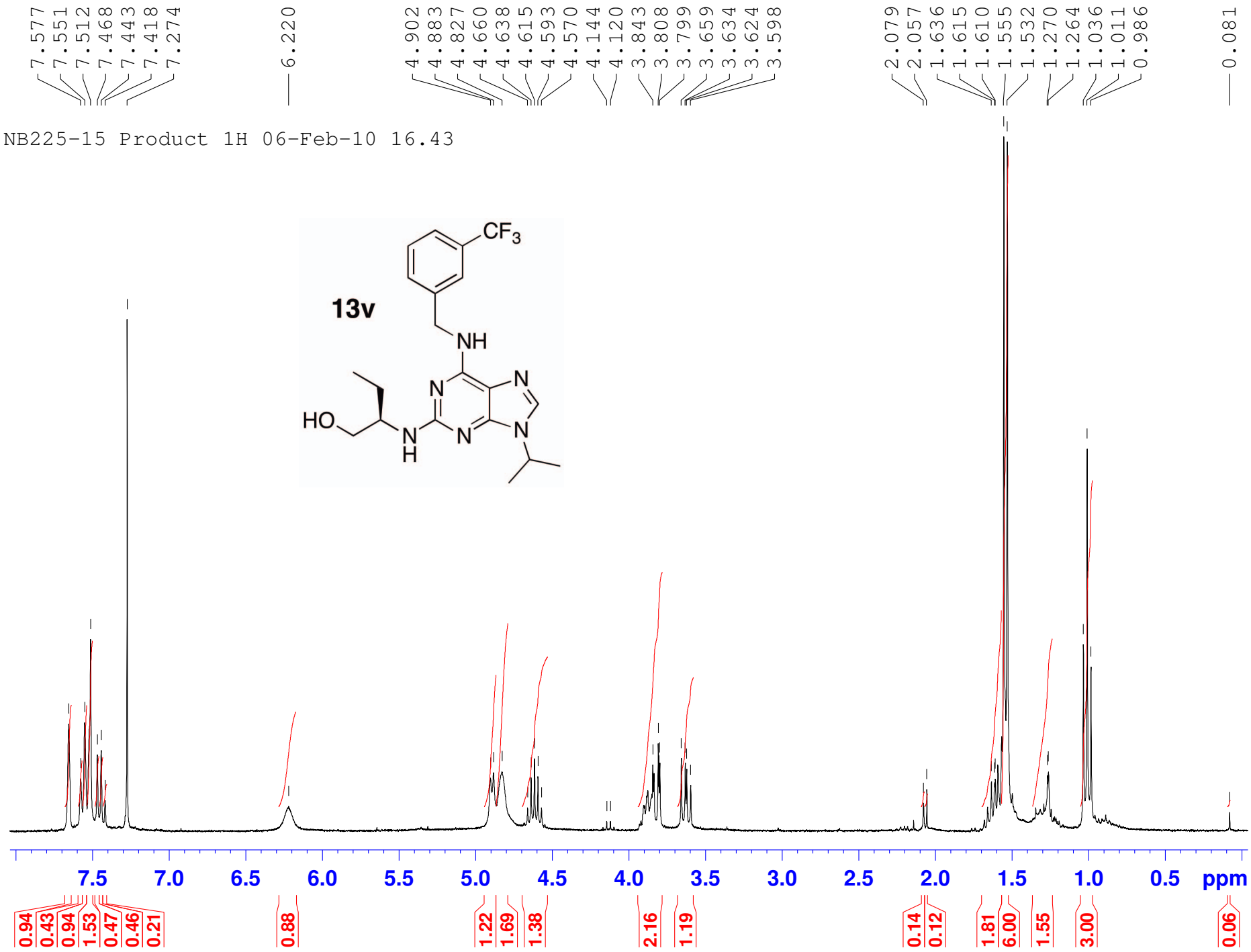



7.598
7.573
7.547
7.491
7.466
7.440
7.266
6.337
4.900
4.876
4.853
4.830
4.808
4.785
2.051
1.749
1.589
1.566
1.263
NB225-09 Product 1H 17-Jan-10 13:19
CF3
16v
NH
N
N
Cl
N
N
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.33
0.83
2.64
0.37
6.00

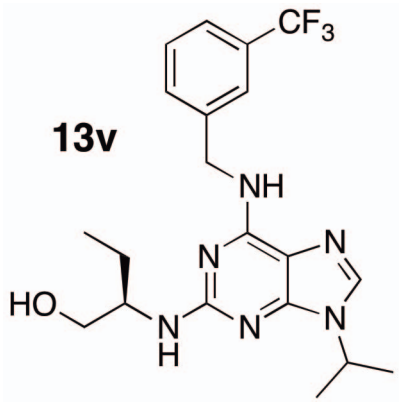
155.13
154.24
150.02
139.43
137.79
131.59
131.16
130.73
130.31
129.38
129.16
125.77
124.44
124.39
124.34
124.29
122.16
118.77
118.56
77.49
77.07
76.64
67.97
46.99
43.94
25.60
22.71

NB225-09 13C 23-Jan-10 14.50




7.577
7.551
7.512
7.468
7.443
7.418
7.274
6.220
4.902
4.883
4.827
4.660
4.638
4.615
4.593
4.570
4.144
4.120
3.843
3.808
3.799
3.659
3.634
3.624
3.598
2.079
2.057
1.636
1.615
1.610
1.555
1.532
1.270
1.264
1.036
1.011
0.986
0.081
NB225-15 Product 1H 06-Feb-10 16.43
13v
CF3
NH
N
N
N
N
HO
N
H
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
0.94
0.43
0.94
1.53
0.47
0.46
0.21
0.88
1.22
1.69
1.38
2.16
1.19
0.14
0.12
1.81
6.00
1.55
3.00
0.06

mf-00417-030_CSC700_13C



159.88
154.63
150.25
140.04
134.69
131.03
130.89
130.84
130.66
130.48
128.98
126.37
124.82
124.45
124.43
124.41
124.39
124.07
124.05
123.28
121.73
114.53

77.18
77.00
76.82

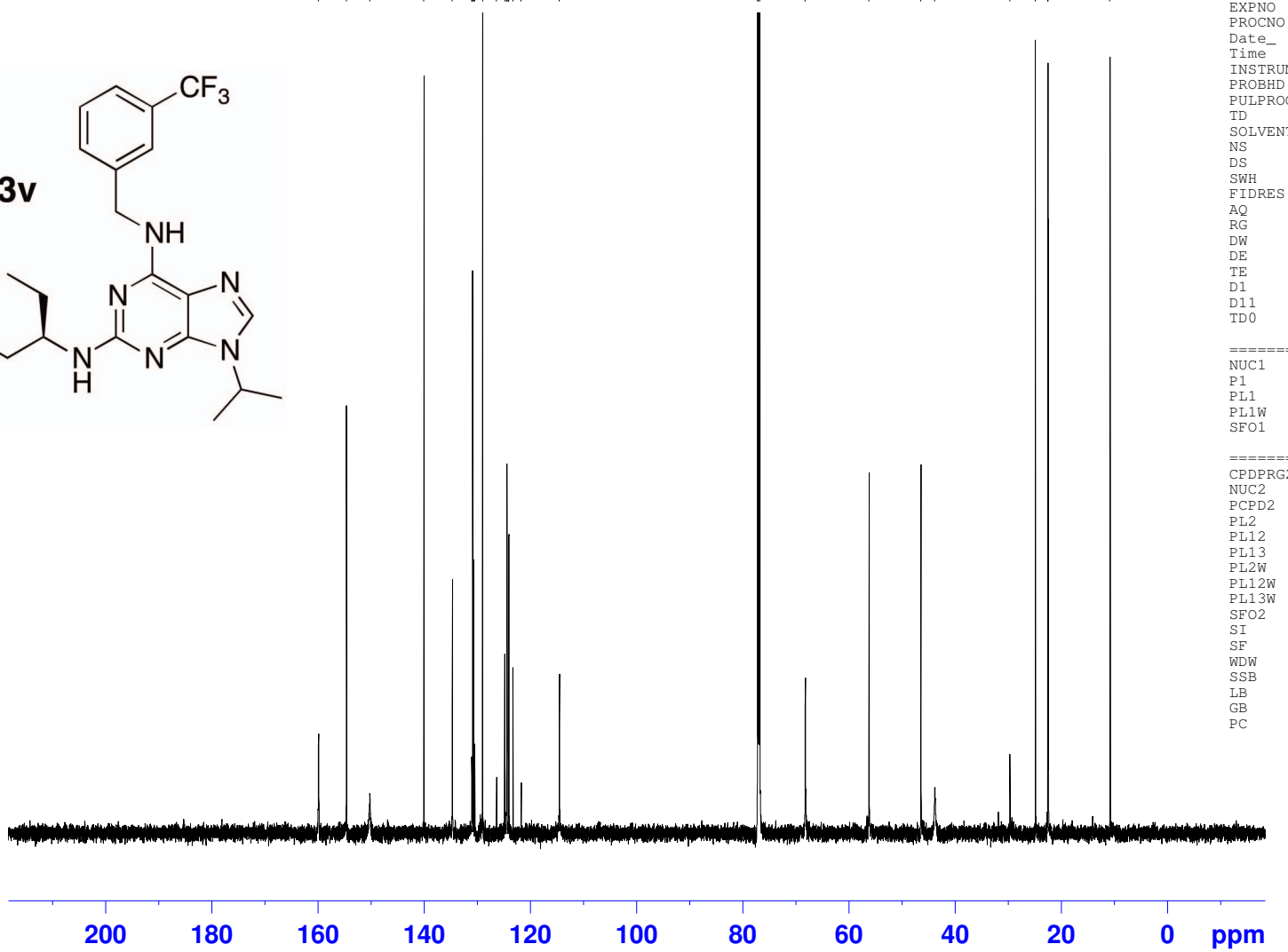
68.21

56.21

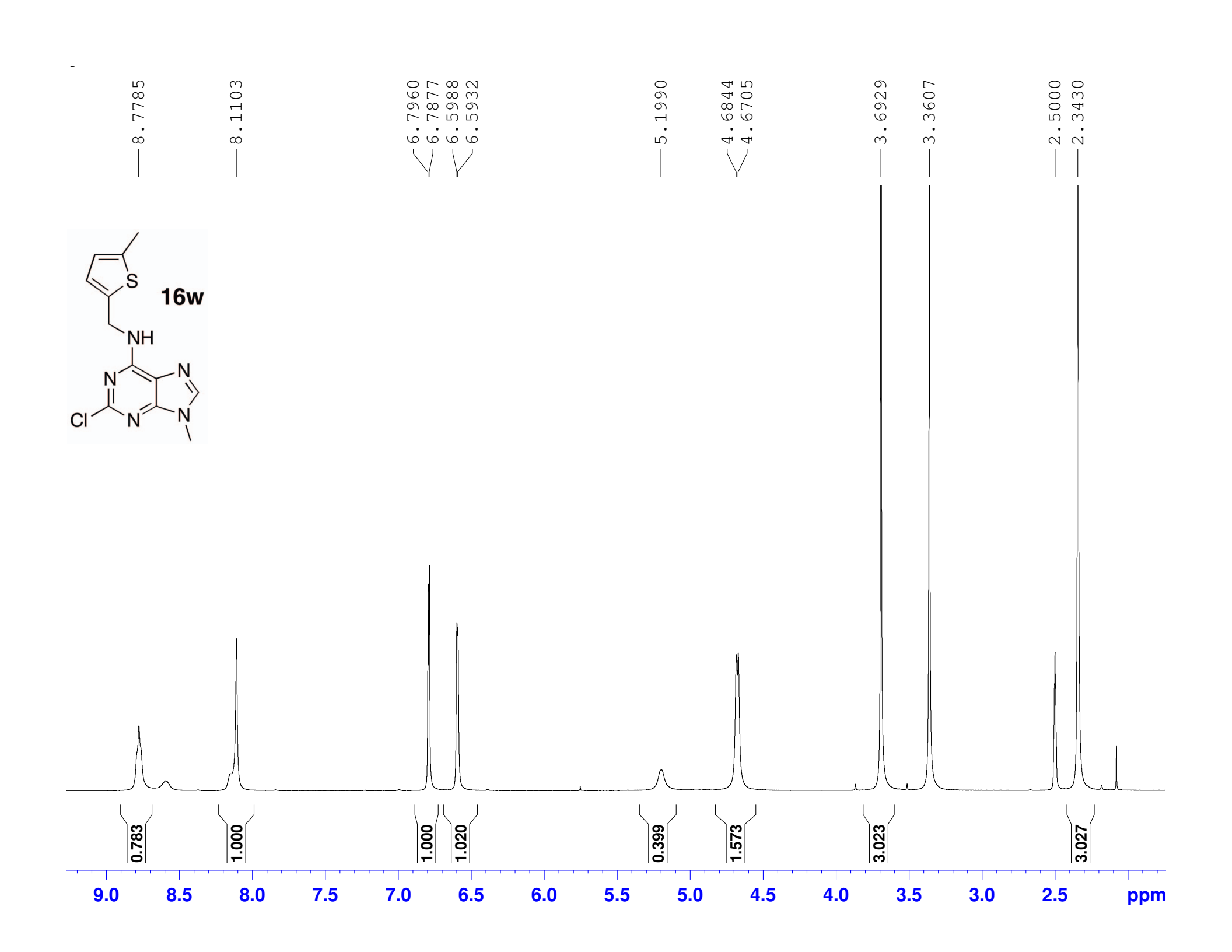
46.45
43.84

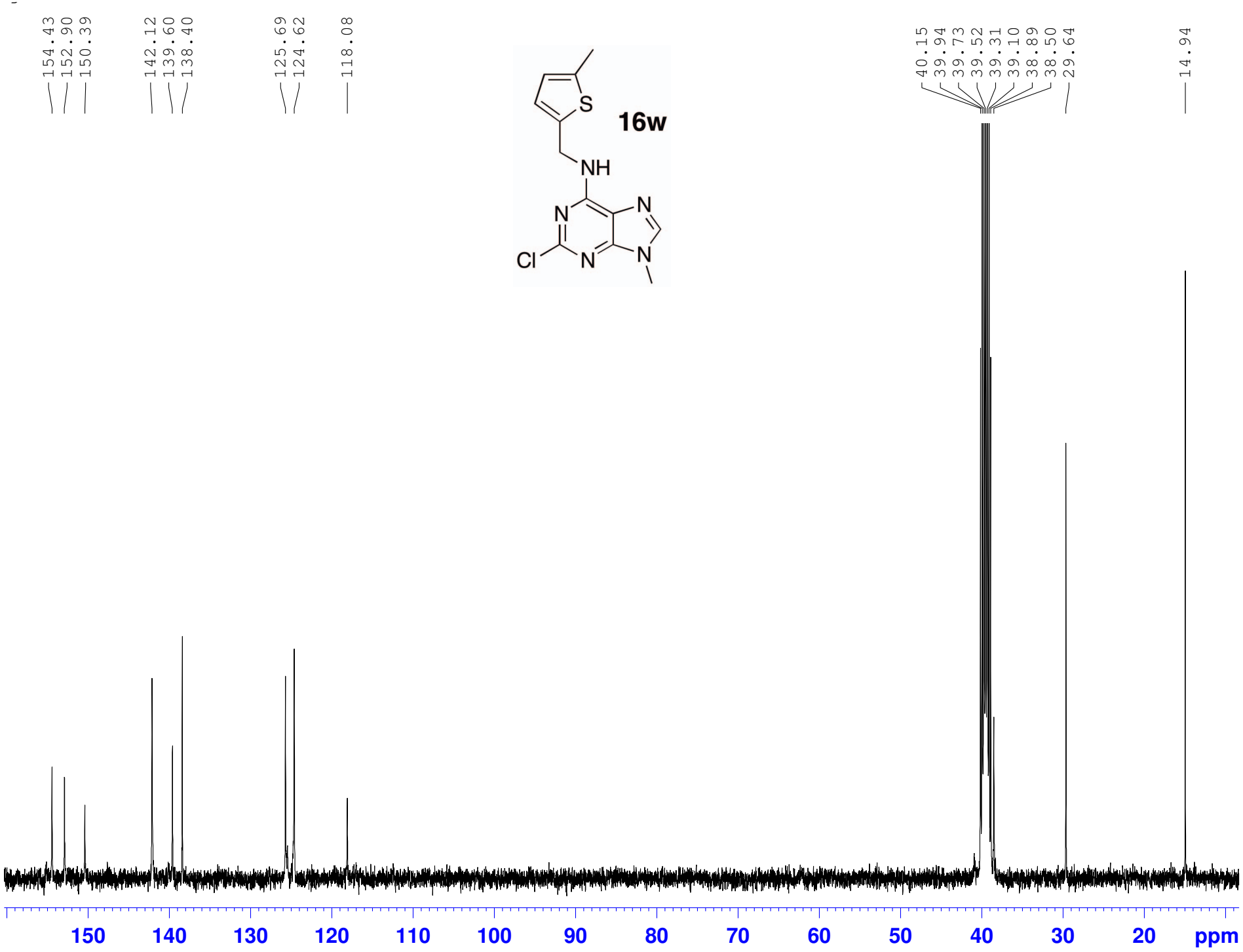
29.68
24.89
22.54
22.47

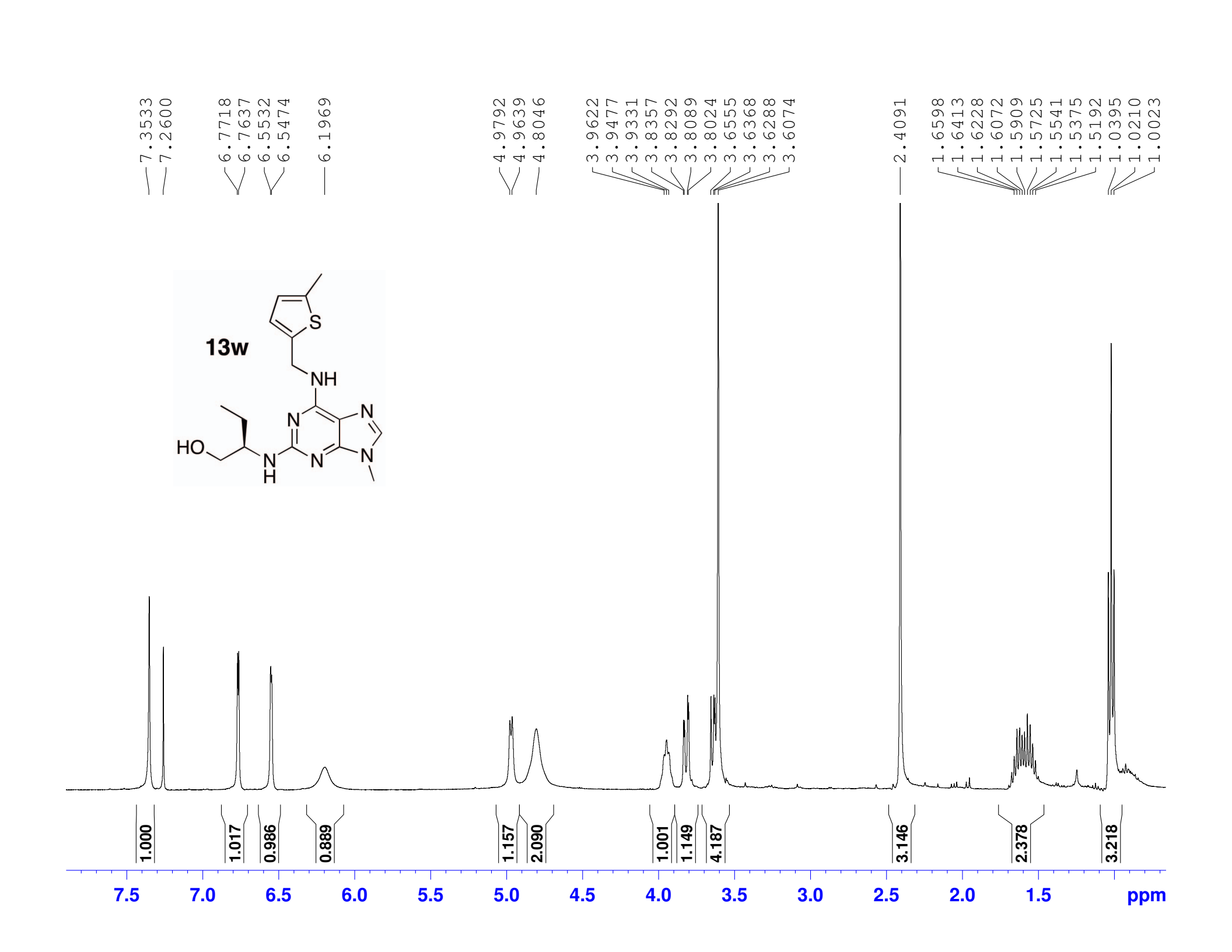
10.84



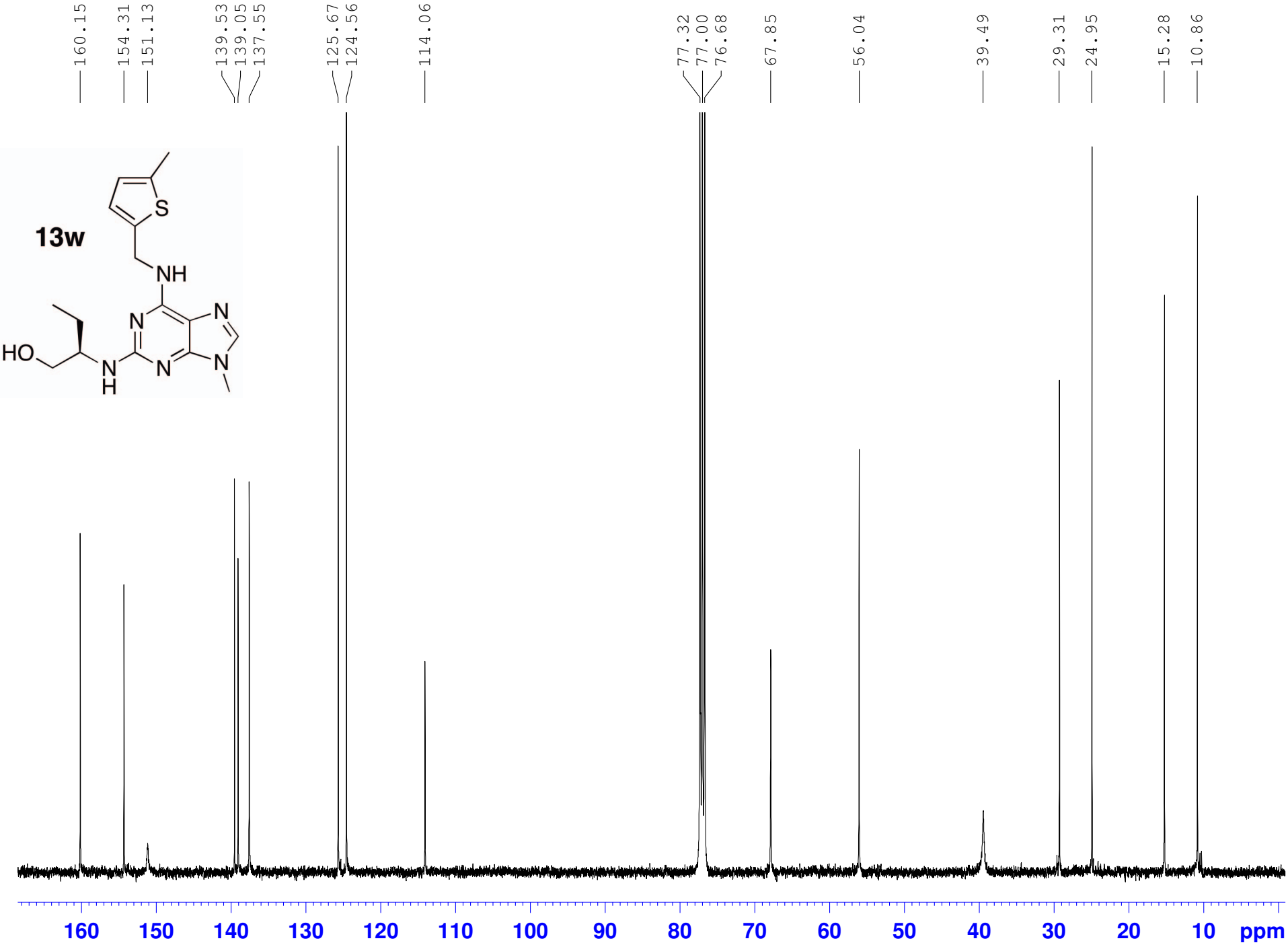
NAME	mf-00417-030
EXPNO	2
PROCNO	1
Date_	20120801
Time	20.13
INSTRUM	spect
PROBHD	5 mm PATXI 1H-
PULPROG	zgpg
TD	65536
SOLVENT	CDCl3
NS	11297
DS	4
SWH	41666.668 Hz
FIDRES	0.635783 Hz
AQ	0.7864820 sec
RG	2050
DW	12.000 usec
DE	6.50 usec
TE	293.0 K
D1	6.00000000 sec
D11	0.03000000 sec
TD0	18
======== CHANNEL f1 ========	
NUC1	13C
P1	16.13 usec
PL1	-3.00 dB
PL1W	242.57461548 W
SFO1	176.0453139 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	2.40 dB
PL12	23.41 dB
PL13	120.00 dB
PL2W	13.05232048 W
PL12W	0.10343982 W
PL13W	0.00000000 W
SFO2	700.0528002 MHz
SI	32768
SF	176.0277205 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


8.7785
8.1103
6.7960
6.7877
6.5988
6.5932
5.1990
4.6844
4.6705
3.6929
3.3607
2.5000
2.3430
16w
S
NH
N
N
Cl
N
N
0.783
1.000
1.000
1.020
0.399
1.573
3.023
3.027
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm

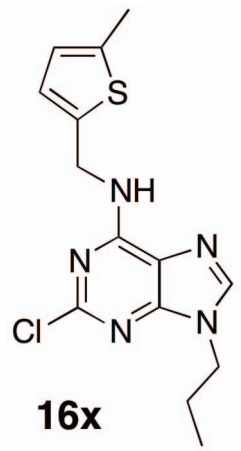

154.43
152.90
150.39
142.12
139.60
138.40
125.69
124.62
118.08
40.15
39.94
39.73
39.52
39.31
39.10
38.89
38.50
29.64
14.94
16w
S
NH
N
N
N
N
Cl
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


7.3533
7.2600
6.7718
6.7637
6.5532
6.5474
6.1969
4.9792
4.9639
4.8046
3.9622
3.9477
3.9331
3.8357
3.8292
3.8089
3.8024
3.6555
3.6368
3.6288
3.6074
2.4091
1.6598
1.6413
1.6228
1.6072
1.5909
1.5725
1.5541
1.5375
1.5192
1.0395
1.0210
1.0023
13w
S
NH
N
N
N
N
HO
N
H
1.000
1.017
0.986
0.889
1.157
2.090
1.001
1.149
4.187
3.146
2.378
3.218
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm

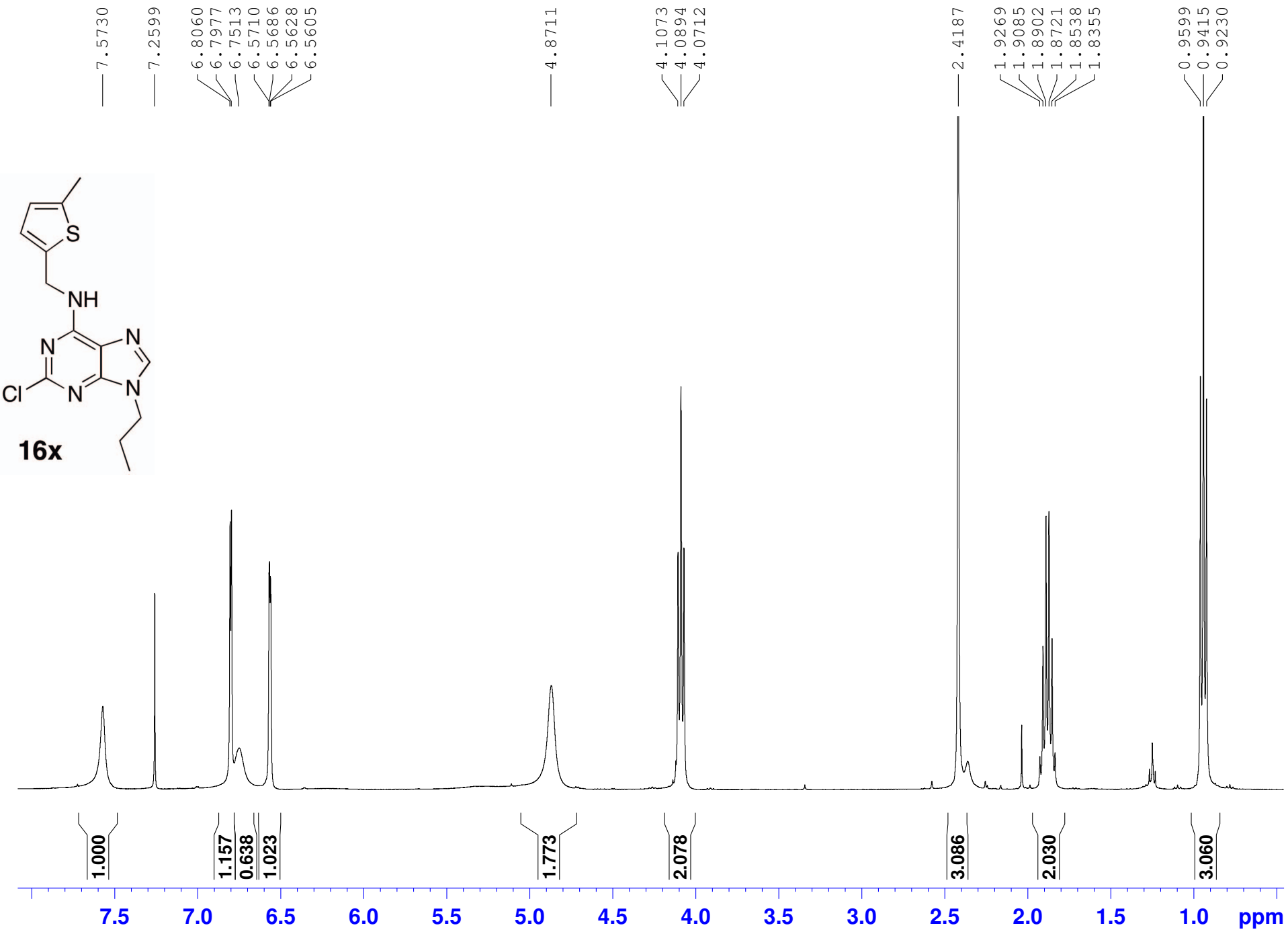
gv-NB-359.05 night 13C CDCl3 400 B 100 MHz



gv-NB-359.56, Column, CDCl3, 1H, 400 B

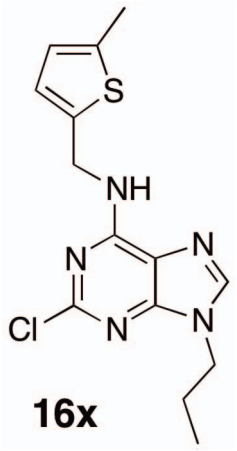


16x



gv-NB-359.56, Column, CDCl3, 13C, 400 B

154.7571
154.4831
150.5363
140.3667
140.1563
138.1263
126.4279
124.8907
118.7210
77.4782
77.1602
76.8428
45.6378
39.7688
23.4537
15.4684
11.2341

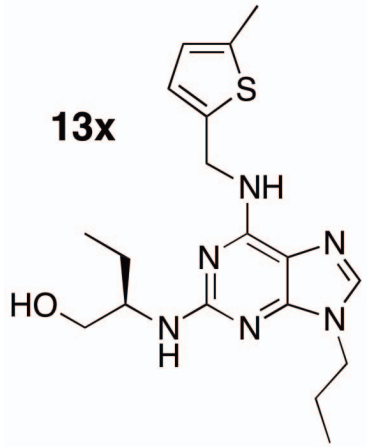


16x

150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

gv-NB-359.58, Column, CDCl3, 1H, 400 B

7.3964
7.2600
6.7958
6.7874
6.5731
6.5704
6.5675
6.5647
6.5621
6.0422
4.9640
4.9502
4.8231
3.9694
3.9518
3.9341
3.9247
3.9050
3.8918
3.8851
3.8469
3.8406
3.8201
3.8138
3.6641
3.6449
3.6373
3.6181
2.4237
1.8895
1.8711
1.8529
1.8350
1.8169
1.7986
1.6999
1.6809
1.6654
1.6465
1.6308
1.6277
1.6123
1.6033
1.5938
1.5846
1.5662
1.5497
1.5313
1.5128
1.0528
1.0342

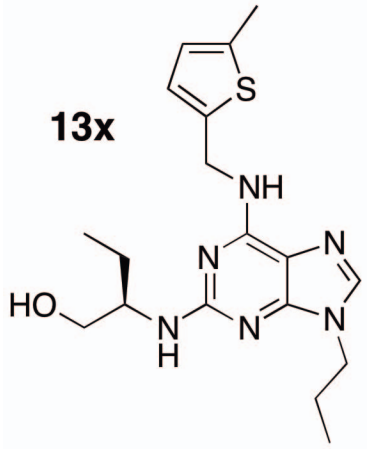


1.000
1.010
1.013
0.938
1.167
2.071
3.141
1.077
1.064
3.192
2.145
2.212
3.132
3.324

7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm

gv-NB-359.58, Column, CDCl3, 13C, 400 B

160.1432
154.4487
150.9924
139.8323
139.0881
137.4043
125.9806
124.8093
114.5117
77.4779
77.1601
76.8431
68.5713
56.5129
45.2448
39.6642
25.1768
23.3342
15.4890
11.3475
11.0841



160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm