A New Method for N–N Bond Cleavage of *N*,*N*-Disubstituted Hydrazines to Secondary Amines and Direct *ortho*-Amination of Naphthol and Its analogues

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General information

^{1}H NMR spectra were recorded on Bruker instruments (600 MHz, 400 MHz). Chemical shifts are reported in ppm with the tetramethylsilane as the internal standard. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration. ^{13}C NMR data were collected on Bruker instruments (150, 100 MHz) with complete proton decoupling. Chemical shifts are reported in ppm with the tetramethylsilane as the internal standard. IR spectra were recorded on a Perkin-Elmer FT-IR spectrometer. Elemental analyses were performed on a CARLO ERBA-1106 apparatus. High-resolution mass spectra were obtained on a Bruker Daltoics Bio TOF-Q mass spectrometer (ESI, MALDI-TOF). GC–MS were measured on Agilent Technologies 6890-5973N. DSC tests were performed on USA / TA-Q100 instrument. Flash column chromatography was performed with silica gel (300–400 mesh). All reactions were carried out under an argon atmosphere and other measures had been worked preferentially under argon because most of the products are especially unstable under air. Commercially available materials were used without further purification unless otherwise noted. For the structure determination, COSY, HSQC and HMBC experiments were also employed.

Experimental procedures

Preparation of substrates

6-Bromo-2-naphthol,[1] 6-*tert*-butyl-2-naphthol,[2] 7-methoxy-2-naphthol,[3] 7-allyloxy-2-naphthol[4] and ethyl 6-hydroxy-2-naphthoate[5] were prepared following the procedures previously reported. *N*-Alkyl-*N*-arylhydrazines[6] were prepared from the corresponding arylhydrazines, while *N*,*N*-diarylhydrazines[7] and *N*,*N*-dialkylhydrazines[8] were prepared from the appropriate secondary amines.

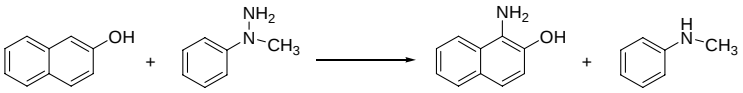
***N*-Methyl-*N*-(naphthalen-2-yl)hydrazine:** Following the procedure elucidated by Lerch[6], under an argon atmosphere and at 0 ^{o}C, to a mixture of sodium amide (0.43 g, 11 mmol) and THF (50 mL) was added dropwise a solution of 2-naphthalenylhydrazine (1.58 g, 10 mmol) in THF (50 mL). After the

addition, the reaction mixture was stirred for 6 h at room temperature and then a gentle stream of argon was passed through the brownish solution for 1 h. With ice cooling, methyl iodide (1.42 g, 10 mmol) was added and then stirred at room temperature for 2 h. Water (20 mL) was introduced and stirring was continued for 0.5 h. The THF was removed in vaccum and the residue diluted with water (100 mL), extracted with dichloromethane. The combined extracts was washed with water and dried with anhydrous sodium sulfate. The solvent was removed in vaccum and the residue was purified by flash column chromatography on silica gel (20:1 / hexanes : ethyl acetate) to provide *N*-methyl-*N*-(naphthalen-2-yl)hydrazine as a white solid (1.18 g, 69% yield). Mp: 50-52 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.73-7.68 (m, 3H), 7.44 (dd, *J* = 9.2 Hz, 2.4 Hz, 1H), 7.39 (t, *J* = 7.2 Hz, 1H), 7.26 (t, *J* = 8.0 Hz, 1H), 7.18 (d, *J* = 1.6 Hz, 1H), 3.89 (brs, 2H), 3.22 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 150.6, 134.7, 128.7, 128.0, 127.6, 126.7, 126.4, 123.0, 117.4, 107.3, 44.8; IR (KBr, cm^{-1}): 3333.8, 3052.6, 2862.5, 1906.0, 1629.1, 1506.3,1102.6, 834.4, 746.4; GC-MS (EI, *m/z*): 172 [M^{+}].

***N*-Heptyl-*N*-(naphthalen-2-yl)hydrazine:** Following the procedure elucidated by Lerch[6], *N*-heptyl-*N*-(naphthalen-2-yl)hydrazine was attained as colorless oil which solidified upon standing (62% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.71-7.65 (m, 3H), 7.38 (t, *J* = 7.2 Hz, 1H), 7.33 (dd, *J* = 9.2 Hz, 2.4 Hz, 1H), 7.24 (t, *J* = 7.6 Hz, 1H), 7.19 (d, *J* = 2.4 Hz, 1H), 3.72 (brs, 2H), 3.48 (t, *J* = 7.2 Hz, 2H), 1.70-1.66 (m, 2H), 1.38-1.28 (m, 8H), 0.89 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 149.8, 135.0, 128.8, 127.8, 127.7, 126.7, 126.4, 122.7, 117.1, 106.9, 56.0, 32.1, 29.5, 27.3, 26.1, 22.9, 14.4; IR (film, cm^{-1}): 3337.6, 3054.9, 2925.9, 2854.7, 1897.8, 1628.9, 1467.4, 830.7; HRMS (ESI) calcd. for: $C_{17}H_{25}N_2$ $[M+H]^{+}$ 257.2018, found *m/z* 257.2011. Anal. Calcd. for $C_{17}H_{24}N_2$: C, 79.64; H, 9.44; N, 10.93. Found: C, 79.53; H, 9.52; N, 10.87.

Procedure for the reaction of *N,N*-disubstituted hydrazines with naphthol and its analogues

Reaction of 2-naphthol with *N*-methyl-*N*-phenylhydrazine (Table 1, entry 1)

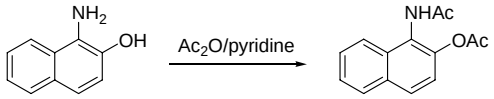


A mixture of 2-naphthol (0.72 g, 5 mmol) and *N*-methyl-*N*-phenylhydrazine (0.61 g, 5 mmol) was heated at 80 ^{o}C for 4 h (tracked by TLC) to attain a brown solid. The brown solid was washed with ethyl acetate (3 × 5 mL) and then dried to attain the first portion of 1-amino-2-naphthol as white solid (0.31 g). The combined ethyl acetate was evaporated and the residue was separated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-methylaniline as colorless oil (0.50 g, 95% yield) and another portion of 1-amino-2-naphthol (0.43 g, 94% total yield). 1-Amino-2-naphthol: Mp: >142 ^{o}C (decomp.); ^{1}H NMR (600 MHz, DMSO-d^6) δ (ppm): 9.15 (brs, 1H, OH), 7.93 (d, J = 8.4 Hz, 1H, H-8), 7.65 (d, J = 7.8 Hz, 1H, H-5), 7.30 (t, J = 7.8 Hz, 1H, H-7), 7.20 (t, J = 7.2 Hz, 1H, H-6), 7.08 (s, 2H, H-3, H-4), 4.95 (brs, 2H, NH_2); ^{13}C NMR (100 MHz, DMSO-d^6) δ (ppm): 139.4 (C-2), 129.3 (C-1), 129.0(C-10), 128.2 (C-5), 124.21(C-6), 124.19 (C-9), 122.7(C-7), 121.9(C-8), 118.0(C-3), 116.5(C-4); IR (KBr, cm^{-1}): 3385.4, 2942.3, 2622.2, 1605.0, 1275.1, 797.7; HRMS (ESI) calcd. for $C_{10}H_{10}NO$ $[M+H]^+$ 160.0762, found *m/z* 160.0757. *N*-Methylaniline: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.18 (t, J = 7.2 Hz, 2H), 6.70 (t, J = 7.2 Hz, 1H), 6.60 (d, J = 8.0 Hz, 2H), 3.58 (brs, 1H), 2.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 149.4, 129.3, 117.3, 112.5, 30.8; GC-MS (EI, *m/z*): 107 [M^+]; Anal. Calcd. for C_7H_9N: C, 78.46; H, 8.47; N, 13.07. Found: C, 78.72; H, 8.48; N, 13.14.

Three cross peaks (pp.20) were observed from couplings between the doublet at 7.93 ppm and the triplet at 7.30 ppm, the doublet at 7.65 ppm and the triplet at 7.20 ppm, the triplet at 7.30 ppm and the triplet at 7.20 ppm. These results show that these four peaks must therefore be assigned to the protons at the aromatic ring without the hydroxyl group, and the singlet at 7.08 ppm should be assigned to the other two aromatic protons at the aromatic ring with the hydroxyl group. The amino group should not be attached to C-3 by analysis of the HMQC and HSQC spectra (pp.21-22). After acetylation of the amino and hydroxyl groups, all of the signals for aromatic protons are splitted. These information, in addition

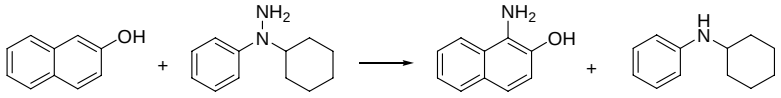
to the relatively large coupling constants, demonstrates that the amino group should be attached to C-1. The structure is further absolutely confirmed by comparing the NMR spectra with that of an authentic sample prepared according to the method described in literature.[9]

Acetylation of 1-amino-2-naphthol with acetic anhydride



A mixture of 1-amino-2-naphthol (0.40 g, 2.5 mmol) and acetic anhydride (10 mL) in pyridine (10 mL) was placed under argon atmosphere and stirred at room temperature for 1 day. The reaction mixture was diluted with water, extracted with dichloromethane. The organic extract was successively washed with saturated sodium bicarbonate and dried with sodium sulfate. Removal of solvent in vaccum and purified by flash column chromatography on silica gel (4:1 / hexanes : ethyl acetate) to provide a white solid (0.54 g, 89%). 1-Acetamidonaphthalen-2-yl acetate: Mp: 188-190 ^{o}C; ^{1}H NMR (600 MHz, DMSO-d^6) δ (ppm): 9.72 (s, 1H), 7.97 (d, J = 7.8 Hz, 1H), 7.90 (d, J = 3.6 Hz, 1H), 7.89 (d, J = 4.2 Hz, 1H), 7.57 (t, J =7.2 Hz, 1H), 7.54 (t, J = 6.6 Hz, 1H), 7.37 (d, J = 9.0 Hz, 1H), 2.27 (s, 3H), 2.15 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d^6) δ (ppm): 169.2, 169.1, 144.2, 132.0, 131.1, 128.4, 127.6, 127.0, 126.2, 125.3, 124.0, 122.7, 23.1, 21.3; GC-MS (EI, *m/z*): 243 [M^+].

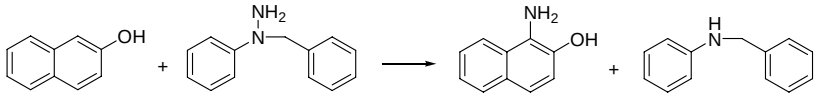
Reaction of 2-naphthol with *N*-cyclohexyl-*N*-phenylhydrazine (Table 1, entry 2)



A mixture of 2-naphthol (0.72 g, 5 mmol) and *N*-cyclohexyl-*N*-phenylhydrazine (0.95 g, 5 mmol) was heated at 90 ^{o}C for 4 h (tracked by TLC) to attain a gray solid. The gray solid was washed with ethyl ether (3×5 mL) and then dried to attain the first portion of 1-amino-2-naphthol as white solid (0.29 g). The collection of ethyl ether was evaporated and separated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-cyclohexylaniline

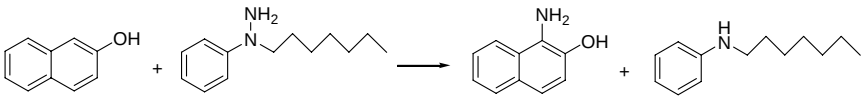
as colorless oil (0.84 g, 96% yield) and another portion of 1-amino-2-naphthol (0.44 g, 93% total yield). *N*-Cyclohexylaniline: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.17-7.12 (m, 2H), 6.67-6.65 (m, 1H), 6.60-6.57 (m, 2H), 3.54 (brs, 1H), 3.28-3.21 (m, 1H), 2.08-2.04 (m, 2H), 1.78-1.73 (m, 2H), 1.67-1.62 (m, 1H), 1.42-1.35 (m, 2H), 1.35-1.27 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 147.5, 129.3, 116.9, 113.2, 51.7, 33.6, 26.0, 25.1; GC-MS (EI, *m/z*): 175 [M^+]; Anal. Calcd. For $C_{12}H_{17}N$: C, 82.23; H, 9.78; N, 7.99. Found: C, 82.73; H, 9.78; N, 8.48.

Reaction of 2-naphthol with *N*-benzyl-*N*-phenylhydrazine (Table 1, entry 3)



A mixture of 2-naphthol (0.72 g, 5 mmol) and *N*-benzyl-*N*-phenylhydrazine (0.99 g, 5 mmol) was heated at 120 °C for 20 h (tracked by TLC) to attain a brown solid. The brown solid was dissolved in a small amount of DMSO and then isolated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-benzylaniline as colorless oil (0.84 g, 92% yield) and 1-amino-2-naphthol (0.67 g, 85% yield). *N*-Benzylaniline: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.38-7.31 (m, 4H), 7.29-7.24 (m, 1H), 7.19-7.15 (m, 2H), 6.74-6.69 (m, 1H), 6.65-6.62 (m, 2H), 4.33 (s, 2H), 4.05 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 148.1, 139.4, 129.3, 128.7, 127.6, 127.3, 117.6, 112.9, 48.4; GC-MS (EI, *m/z*): 183 [M^+].

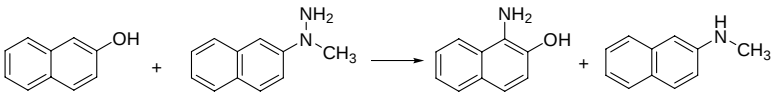
Reaction of 2-naphthol with *N*-heptyl-*N*-phenylhydrazine (Table 1, entry 4)



A mixture of 2-naphthol (0.72 g, 5 mmol) and *N*-heptyl-*N*-phenylhydrazine (1.03 g, 5 mmol) was heated at 90 °C for 4 h (tracked by TLC) to attain a brown slurry. The brown slurry was dissolved in a small amount of DMSO and then isolated by flash column chromatography on silica gel (gradient

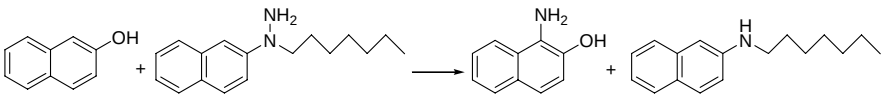
elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-heptylaniline as colorless oil (0.90 g, 95% yield) and 1-amino-2-naphthol (0.74 g, 94%). *N*-Heptylaniline: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.17 (t, *J* = 8.0 Hz, 2H), 6.68 (t, *J* = 7.2 Hz, 1H), 6.60 (d, *J* = 8.0 Hz, 2H), 3.61 (brs, 1H), 3.10 (t, *J* = 7.2 Hz, 2H), 1.65-1.57 (m, 2H), 1.43-1.25 (m, 8H), 0.89 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 148.7, 129.3, 117.2, 112.8, 44.1, 32.0, 29.8, 29.3, 27.3, 22.8, 14.3; GC-MS (EI, *m/z*): 191 [M^+].

Reaction of 2-naphthol with *N*-methyl-*N*-(naphthalene-2-yl)hydrazine (Table 1, entry 5)



A mixture of 2-naphthol (0.72 g, 5 mmol) and *N*-methyl-*N*-(naphthalene-2-yl)hydrazine (0.85 g, 5 mmol) was heated at 90 °C for 4 h (tracked by TLC) to attain a slurry material. The slurry was dissolved in a small amount of DMSO and then separated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-methylnaphthalen-2-amine as colorless oil (0.75 g, 95% yield) and 1-amino-2-naphthol (0.74 g, 93% yield). *N*-Methylnaphthalen-2-amine: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.80-7.70 (m, 3H), 7.49 (t, *J* = 7.2 Hz, 1H), 7.32 (t, *J* = 7.2 Hz, 1H), 6.93-6.90 (m, 1H), 6.88 (s, 1H), 3.81 (brs, 1H), 2.96 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 147.1, 135.4, 128.9, 127.8, 127.6, 126.5, 126.1, 122.0, 118.1, 103.8, 30.8; GC-MS (EI, *m/z*): 157 [M^+]; Anal. Calcd. for $C_{11}H_{11}N$: C, 84.04; H, 7.05; N, 8.91. Found: C, 84.72; H, 7.48; N, 8.14.

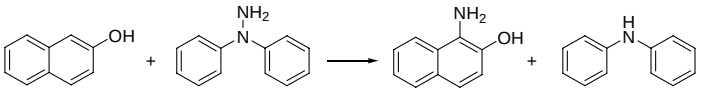
Reaction of 2-naphthol with *N*-heptyl-*N*-(naphthalene-2-yl)hydrazine (Table 1, entry 6)



A mixture of 2-naphthol (0.72 g, 5 mmol) and *N*-heptyl-*N*-(naphthalene-2-yl)hydrazine (1.28 g, 5 mmol) was heated at 90 °C for 4 h (tracked by TLC) to attain a slurry material. The slurry was dissolved in a small amount of DMSO and then isolated by flash column chromatography on silica gel (gradient

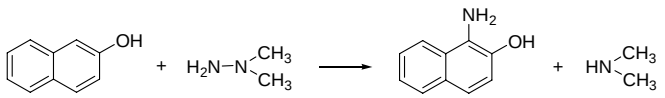
elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-heptylnaphthalen-2-amine as colorless oil (1.14 g, 95% yield) and 1-amino-2-naphthol (0.74 g, 93% yield). N-Heptylnaphthalen-2-amine: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.65 (d, *J* = 8.0 Hz, 1H), 7.60 (d, *J* = 8.4 Hz, 2H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.17 (t, *J* = 7.2 Hz, 1H), 6.85 (dd, *J* = 8.4 Hz, 2.0 Hz, 1H), 6.78 (d, *J* = 2.0 Hz, 1H), 3.76 (brs, 1H), 3.19 (t, *J* = 7.2 Hz, 2H), 1.69-1.63 (m, 2H), 1.48-1.25 (m, 8H), 0.90 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 146.4, 135.6, 129.0, 127.9, 127.6, 126.5, 126.1, 122.0, 118.3, 104.3, 44.2, 32.1, 29.7, 29.5, 27.5, 23.0, 14.4; IR (film, cm^{-1}): 3412.5, 3050.5, 2926.5, 2855.0, 1897.8, 1630.1, 1522.2, 1225.3, 827.4, 743.5； HRMS (MALDI-TOF) calcd. for: $C_{17}H_{23}N$ [M^+] 241.1830, found *m/z* 241.1825; Anal. Calcd. for $C_{17}H_{23}N$: C, 84.59; H, 9.60; N, 5.80. Found: C, 84.62; H, 9.63; N, 5.78.

Reaction of 2-naphthol with *N,N*-diphenylhydrazine (Table 1, entry 7)



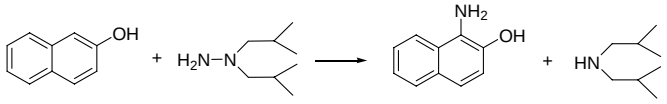
A mixture of 2-naphthol (0.72 g, 5 mmol) and *N,N*-diphenylhydrazine (0.92 g, 5 mmol) was heated at 120 ^{o}C for 4h (tracked by TLC). After cooled, the reaction mixture was dissolved in a small amount of DMSO and then separated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide diphenylamine as white solid (0.80 g, 95% yield) and 1-amino-2-naphthol (0.71 g, 91% yield). Diphenylamine: Mp: 52-54 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.26 (m, 4H), 7.07 (d, *J* = 7.6 Hz, 4H), 6.92 (t, *J* = 7.2 Hz, 2H), 5.71(brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 143.3, 129.5, 121.2, 118.0; GC-MS (EI, *m/z*): 169 [M^+].

Reaction of 2-naphthol with *N,N*-dimethylhydrazine (Table 1, entry 8)



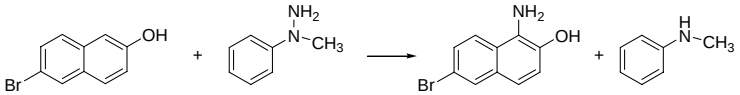
A mixture of 2-naphthol (0.72 g, 5 mmol) and *N*,*N*-dimethylhydrazine (0.60 g, 10 mmol) was heated at 80 ^{o}C for 4 h (tracked by TLC) to attain a brown slurry material. After cooled, the reaction mixture was dissolved in a small amount of DMSO and then isolated by flash column chromatography on silica gel (1:1 / hexanes : ethyl acetate) to provide 1-amino-2-naphthol (0.71 g, 91% yield).

Reaction of 2-naphthol with *N*,*N*-diisobutylhydrazine (Table 1, entry 9)



A mixture of 2-naphthol (0.72 g, 5 mmol) and *N*,*N*-diisobutylhydrazine (0.72 g, 5 mmol) was heated at 80 ^{o}C for 4 h (tracked by TLC) to attain a black slurry material. After cooled, the reaction material was dissolved in a small amount of DMSO and then isolated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide diisobutylamine (0.45 g, 70%) and 1-amino-2-naphthol (0.51 g, 65% yield). Diisobutylamine: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 2.39 (d, *J* = 6.8 Hz, 4H), 1.75 (m, 2H), 1.33 (brs, 1H), 0.98 (d, *J* = 6.8 Hz, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 58.0, 28.1, 20.5; GC-MS (EI, *m/z*): 129 [M^+].

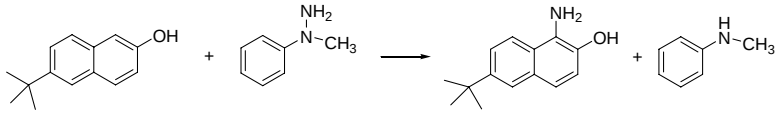
Reaction of 6-bromo-2-naphthol with *N*-methyl-*N*-phenylhydrazine (Table 2, entry 1)



A mixture of 6-bromo-2-naphthol (1.12 g, 5 mmol) and *N*-methyl-*N*-phenylhydrazine (0.61 g, 5 mmol) was heated at 90 ^{o}C for 4 h (measured by TLC). After cooled, the reaction mixture was dissolved in a small amount of DMSO and then separated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-methylaniline as colorless oil (0.51 g, 95% yield) and 1-amino-6-bromo-2-naphthol as white solid (1.12 g, 94% yield). 1-Amino-6-bromo-2-naphthol: Mp: >162 ^{o}C (decomp.); ^{1}H NMR (400 MHz, DMSO-d^6) δ (ppm): 9.32 (brs, 1H), 7.92 (d, *J*

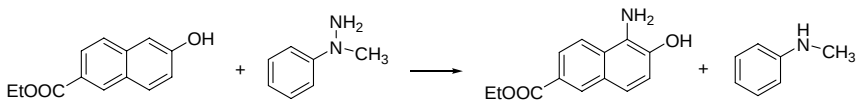
= 9.2 Hz, 1H), 7.89 (d, *J* = 2.0 Hz, 1H), 7.37 (dd, *J* = 8.8 Hz, 2.0 Hz, 1H), 7.10 (d, *J* = 8.4 Hz, 1H), 7.04 (d, *J* = 8.4 Hz, 1H), 5.08 (brs, 2H); ^{13}C NMR (100 MHz, DMSO-*d*^{6}) δ (ppm): 140.0, 130.2, 129.94, 129.86, 126.8, 124.5, 122.4, 119.0, 115.8, 115.6; IR (KBr, cm^{-1}): 3383.7, 3303.6, 2961.4, 1576.5, 1366.2, 1277.1, 870.0; HRMS (MALDI-TOF) calcd. for: $C_{10}H_8BrNO$ [M^+] 236.9789, found *m/z* 236.9766.

Reaction of 6-*tert*-butyl-2-naphthol with *N*-methyl-*N*-phenylhydrazine (Table 2, entry 2)



A mixture of 6-*tert*-butyl-2-naphthol (1.00 g, 5 mmol) and *N*-methyl-*N*-phenylhydrazine (0.61 g, 5 mmol) was heated at 90 ^{o}C for 4 h (tracked by TLC) to attain a yellow solid. The yellow solid was dissolved in a small amount of DMSO and then isolated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-methylaniline as colorless oil (0.51 g, 95% yield) and 1-amino-6-*tert*-butyl-2-naphthol as white solid (1.00 g, 93% yield). 1-Amino-6-*tert*-butyl-2-naphthol: Mp: 154-156 ^{o}C; ^{1}H NMR (600 MHz, DMSO-*d*^{6}) δ (ppm): 8.99 (brs. 1H), 7.87 (d, *J* = 9.0 Hz, 1H), 7.54 (d, *J* = 1.8 Hz, 1H), 7.40 (dd, *J* = 9.0 Hz, 1.8 Hz, 1H), 7.02 (s, 2H), 4.85 (brs. 2H), 1.34 (s, 9H); ^{13}C NMR (150 MHz, DMSO-*d*^{6}) δ (ppm): 144.6, 138.9, 129.1, 128.9, 123.0, 122.9, 122.5, 121.8, 118.0, 116.5, 34.6, 31.6; IR (KBr, cm^{-1}): 3389.6, 295835, 2631.2, 1865.8, 1570.2, 1283.8, 803.6; HRMS (MALDI-TOF) calcd. for: $C_{14}H_{17}NO$ [M^+] 215.1310, found *m/z* 215.1331； Anal. Calcd. for $C_{11}H_{17}NO$: C, 78.10; H, 7.96; N, 6.51. Found: C, 78.12; H, 7.92; N, 6.56.

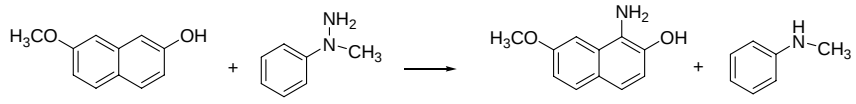
Reaction of ethyl 6-hydroxy-2-naphthoate with *N*-methyl-*N*-phenylhydrazine (Table 2, entry 6)



A mixture of ethyl 6-hydroxy-2-naphthoate (1.08 g, 5 mmol) and *N*-methyl-*N*-phenylhydrazine (0.61 g, 5 mmol) was heated at 90 ^{o}C for 8 h (tracked by TLC) to attain a yellow solid. The yellow solid was dissolved in a small amount of DMSO and then isolated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-methylaniline as colorless oil (0.50 g, 94% yield) and ethyl 5-amino-6-hydroxy-2-naphthoate as a yellow solid (1.05 g, 91% yield). Ethyl 5-amino-6-hydroxy-2-naphthoate: Mp: 131-133 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d^6) δ (ppm): 9.50 (brs. 1H), 8.37 (d, J = 1.2 Hz, 1H), 8.04 (d, J = 9.2 Hz, 1H), 7.75 (dd, J = 8.8 Hz, 1.6 Hz, 1H), 7.29 (d, J = 8.4 Hz, 1H), 7.17 (d, J = 8.4 Hz, 1H), 5.15 (brs, 2H), 4.34 (q, J = 7.2 Hz, 2H), 1.35 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d^6) δ (ppm): 166.7, 142.0, 131.4, 129.7, 127.7, 125.7, 123.9, 122.9, 122.5, 118.7, 118.5, 60.9, 14.8; IR (KBr, cm^{-1}): 3396.4, 3338.9, 2984.5, 2637.4, 1691.2, 1628.9, 1285.2, 1112.7, 803.1; HRMS (MALDI-TOF) calcd. for: $C_{13}H_{13}NO_3$ [M^+] 231.0895, found *m/z* 231.0882; Anal. Calcd. for $C_{13}H_{13}NO_3$: C, 67.52; H, 5.67; N, 6.06. Found: C, 67.65; H, 5.72; N, 6.01.

Two cross peaks (pp.34) were observed from couplings between the doublet at 8.37 ppm and the double-doublet at 7.75 ppm, the doublet at 8.04 ppm and the double-doublet at 7.75 ppm. These results show that these three peaks must therefore be assigned to the protons at the aromatic ring with the ester group, and the doublets at 7.29 ppm and 7.17 ppm should be assigned to the other two protons at the aromatic ring without the ester group. The coupling and the large coupling constants (8.4 Hz) of these two protons indicate that the amino group is situated at C-5.

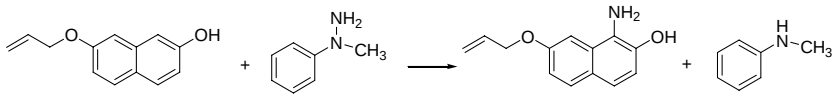
Reaction of 7-methoxy-2-naphthol with *N*-methyl-*N*-phenylhydrazine (Table 2, entry 4)



A mixture of 7-methoxy-2-naphthol (0.87 g, 5 mmol) and *N*-methyl-*N*-phenylhydrazine (0.61 g, 5 mmol) was heated at 90 ^{o}C for 2 h (tracked by TLC) to attain a grey solid. The grey solid was dissolved in a small amount of DMSO and then isolated by flash column chromatography on silica gel

(gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-methylaniline as colorless oil (0.50 g, 94% yield) and 1-amino-7-methoxy-2-naphthol as a white solid (0.87 g, 92% yield). 1-Amino-7-methoxy-2-naphthol: Mp: 120-121 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d^6) δ (ppm): 9.09 (brs. 1H), 7.55 (d, J = 8.8 Hz, 1H), 7.26 (d, J = 2.4 Hz, 1H), 6.99 (d, J = 8.8 Hz, 1H), 6.91 (d, J = 8.4 Hz, 1H), 6.85 (dd, J = 8.8 Hz, 2.0Hz, 1H), 4.85 (brs, 2H), 2.50 (t, J = 2.0 Hz, 3H); ^{13}C NMR (100 MHz, DMSO-d^6) δ (ppm): 156.8, 140.1, 129.9, 128.4, 125.2, 124.5, 116.7, 115.6, 115.2, 100.9, 55.6; IR (KBr, cm^{-1}): 3344.6, 3266.8, 2994.4, 2650.6, 1618.3, 1516.9, 1237.1, 819.4; The NMR spectra are in accordance with that of a sample prepared following the literature procedure.[10] HRMS (MALDI-TOF) calcd. for: $C_{11}H_{11}NO_2$ [M^+] 189.0790, found *m/z* 189.0769.

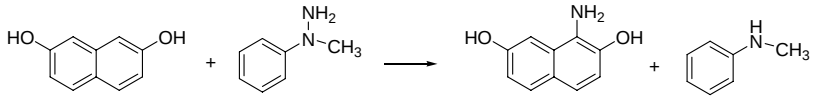
Reaction of 7-allyloxy-2-naphthol with *N*-methyl-*N*-phenylhydrazine (Table 2, entry 5)



A mixture of 7-allyloxy-2-naphthol (1.00 g, 5 mmol) and *N*-methyl-*N*-phenylhydrazine (0.61 g, 5 mmol) was heated at 90 ^{o}C for 2 h (tracked by TLC) to attain a grey solid. The grey solid was dissolved in a small amount of DMSO and then isolated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-methylaniline as colorless oil (0.51 g, 95% yield) and 1-amino-7-allyloxy-2-naphthol as a white solid (0.98 g, 92% yield). 1-Amino-7-allyloxy-2-naphthol: Mp: 105-106 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d^6) δ (ppm): 9.05 (brs. 1H), 7.56 (d, J = 8.8 Hz, 1H), 7.30 (d, J = 2.4 Hz, 1H), 6.99 (d, J = 8.4 Hz, 1H), 6.91 (d, J = 8.8 Hz, 1H), 6.88 (dd, J = 8.8 Hz, 2.4Hz, 1H), 6.12 (m, 1H), 5.45 (dd, J = 17.6Hz, 1.6 Hz, 1H), 5.27 (d, J = 10.8 Hz, 1H) 4.79 (brs, 2H), 4.66 (d, J = 5.2Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d^6) δ (ppm): 155.6, 140.0, 134.4, 129.9, 128.3, 125.1, 124.5, 117.8, 116.6, 115.7, 115.4, 102.1, 68.7; IR (KBr, cm^{-1}): 3387.3, 2919.3, 2637.4, 1617.8, 1517.6, 1213.2, 1113.9, 820.6; HRMS (MALDI-TOF) calcd. for: $C_{13}H_{13}NO_2$ [M^+] 215.0946,

found *m/z* 215.0952； Anal. Calcd. for $C_{13}H_{13}NO_2$: C, 72.54; H, 6.09; N, 6.51. Found: C, 72.46; H, 6.10; N, 6.55.

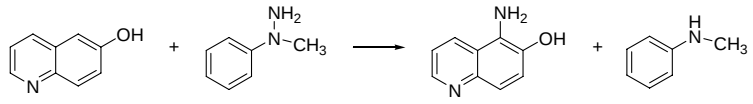
Reaction of 2,7-naphthalendiol with *N*-methyl-*N*-phenylhydrazine (Table 2, entry 3)



To a mixture of 2,7-naphthalendiol (0.80 g, 5 mmol) and chlorobenzene (5.0 mL) was added *N*-methyl-*N*-phenylhydrazine (0.61 g, 5 mmol). The mixture was then refluxed for 3 h (tracked by TLC). After cooled down, the reaction mixture was dissolved in a small amount of DMSO and then separated by flash column chromatography on silica gel to provide *N*-methylaniline (gradient elution 10:1 / hexanes : ethyl acetate →4:1 / hexanes : ethyl acetate) as colorless oil (0.49 g, 93% yield) and 1-amino-2,7-naphthalenediol (gradient elution 10:1 / dichloromethane : methanol→4:1 / dichloromethane : methanol) as grey solid (0.82 g, 93% yield,). 1-Amino-2,7-naphthalenediol: Mp: >300 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d^6) δ (ppm): 9.28 (s, 1H), 8.95 (brs. 1H), 7.49 (d, *J* = 8.8 Hz, 1H), 7.08 (d, *J* = 2.0 Hz, 1H), 6.96 (d, *J* = 8.4 Hz, 1H), 6.84 (d, *J* = 8.4 Hz, 1H), 6.80 (dd, *J* = 8.4 Hz, 2.0Hz, 1H), 4.49 (brs, 2H); ^{13}C NMR (100 MHz, DMSO-d^6) δ (ppm): 154.6, 139.9, 129.8, 127.3, 126.2, 123.9, 117.2, 115.5, 115.0, 103.6; IR (KBr, cm^{-1}): 3394.0, 3322.6, 3273.6, 2605.4, 1635.9, 1330.5, 1207.1, 823.6; HRMS (MALDI-TOF) calcd. for: $C_{10}H_9NO_2$ [M^+] 175.0633, found *m/z* 175.0647.

Two cross peaks (pp.44) were observed from couplings between the doublet at 7.49 ppm and the double-doublet at 6.80 ppm, the doublet at 7.08 ppm and the double-doublet at 6.80 ppm. These results show that these three peaks must therefore be assigned to the protons at the aromatic ring without the amino group, and the doublets at 6.96 ppm and 6.84 ppm should be assigned to the other two protons at the aromatic ring with the amino group. The coupling and the large coupling constants (8.4 Hz) of these two protons suggest that the amino group is situated at C-1. The structure is further confirmed by comparing the NMR spectra with that of a sample prepared according to the literature procedure.[11]

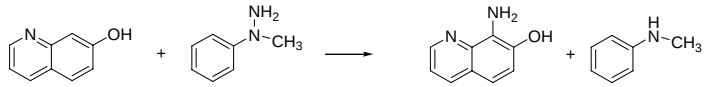
Reaction of 6-hydroxyquinoline with *N*-methyl-*N*-phenylhydrazine (Table 2, entry 7)



To a mixture of 6-hydroxyquinoline (0.73 g, 5 mmol) and chlorobenzene (5 mL) was added *N*-methyl-*N*-phenylhydrazine (0.61 g, 5 mmol). The mixture was then refluxed for 12 h (tracked by TLC). After cooled down, the reaction mixture was dissolved in a small amount of DMSO and then separated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-methylaniline as colorless oil (0.48 g, 89% yield) and 5-amino-6-hydroxylquinoline as yellow solid (0.46 g, 58% yield,). 5-Amino-6-hydroxylquinoline: Mp: 165-168 ^{o}C; ^{1}H NMR (600 MHz, DMSO-d^6) δ (ppm): 9.42(brs, 1H), 8.60 (d, J = 4.2 Hz, 1H), 8.44 (d, J = 8.4 Hz, 1H), 7.31-7.28 (m, 2H), 7.18 (d, J = 9.0 Hz, 1H), 5.23 (brs, 2H); ^{13}C NMR (150 MHz, DMSO-d^6) δ (ppm): 147.2, 143.8, 139.3, 130.4, 129.6, 120.9, 119.1, 118.7, 117.1; IR (KBr, cm^{-1}): 3407.5, 3309.1, 2962.0, 2919.9, 2637.7, 2553.3, 1739.6, 1626.7, 1510.8, 1343.0, 1116.5, 803.6; HRMS (MALDI-TOF) calcd. for: $C_9H_8N_2O$ [M^+] 160.0637, found m/z 160.0641.

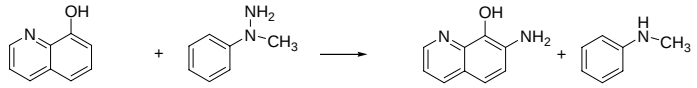
Two cross peaks (pp.48) were observed from couplings between the multiplet at 7.31-7.28 ppm and the doublet at 8.60 ppm, the multiplet at 7.31-7.28 ppm and the doublet at 8.44 ppm. These results show that these three peaks must therefore be assigned to the protons at the pyridine ring, and the multiplet at 7.31-7.28 ppm and the doublet at 7.18 ppm should be assigned to the other two protons at the benzene ring. The coupling and the large coupling constants (9.0 Hz) of these two protons indicate that the amino group is situated at C-5.

Reaction of 7-hydroxyquinoline with *N*-methyl-*N*-phenylhydrazine (Table 2, entry 8)



To a mixture of 7-hydroxyquinoline (0.73 g, 5 mmol) and chlorobenzene (5 mL) was added *N*-methyl-*N*-phenylhydrazine (0.61 g, 5 mmol). The mixture was then refluxed for 48 h (tracked by TLC). After cooled down, the reaction mixture was dissolved in a small amount of DMSO and then separated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-methylaniline as colorless oil (0.47 g, 88% yield) and 8-amino-7-hydroxylquinoline as a yellow solid (0.36 g, 45% yield,). 8-Amino-7-hydroxylquinoline: Mp: 168-170 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d^6) δ (ppm): 9.41(brs, 1H), 8.66 (dd, J = 8.4 Hz, 2.0 Hz, 1H), 8.10 (dd, J = 8.4 Hz, 1.6 Hz, 1H), 7.25 (q, J = 4.0 Hz, 1H), 7.16 (d, J = 8.4 Hz, 1H), 7.07 (d, J = 8.8 Hz, 1H), 5.08 (brs, 2H); ^{13}C NMR (100 MHz, DMSO-d^6) δ (ppm): 147.9, 141.7, 138.5, 136.2, 130.3, 123.2, 118.8, 118.7, 114.8; IR (KBr, cm^{-1}): 3350.9, 2925.7, 2707.9, 1623.3, 1506.1, 1347.7, 823.2; HRMS (MALDI-TOF) calcd. for: $C_9H_8N_2O$ [M^+] 160.0637, found m/z 160.0626.

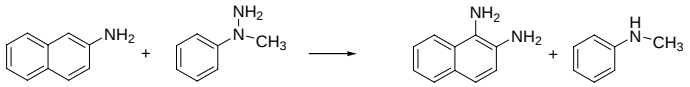
Reaction of 8-hydroxyquinoline with *N*-methyl-*N*-phenylhydrazine (Table 2, entry 9)



A mixture of 8-hydroxyquinoline (0.73 g, 5 mmol) and *N*-methyl-*N*-phenylhydrazine (0.61 g, 5 mmol) was heated at 90 ^{o}C for 30 h (tracked by TLC). After cooled, the reaction material was dissolved in a small amount of DMSO and then separated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-methylaniline as colorless oil (0.38 g, 71% yield) and 7-amino-8-hydroxyquinoline as colorless oil (0.25 g, 31% yield). 7-Amino-8-hydroxyquinoline: ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.71 (dd, J = 4.0 Hz, 1.2 Hz, 1H), 8.04 (dd, J = 8.0 Hz, 1.6 Hz, 1H), 7.28-7.25 (m, 1H), 7.19 (q, J = 4.0 Hz, 1H), 7.10 (d, J = 8.4 Hz, 1H), 4.05 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 148.4, 138.5, 136.3, 136.1, 131.5, 122.4, 119.4, 118.4, 118.0; IR (film, cm^{-1}): 3365.2, 3196.4, 3020.0, 1630.1, 1507.0, 1307.7, 1184.7, 824.1; The NMR spectra

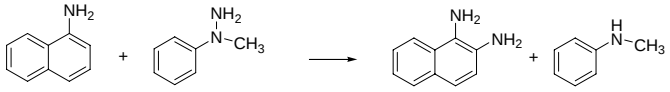
is in accordance with that of an authentic sample prepared according to the method described in literature.[12] HRMS (ESI) calcd. for: $C_9H_9N_2O$ $[M+H]^+$ 161.0715, found *m/z* 161.0709.

Reaction of 2-naphthylamine with *N*-methyl-*N*-phenylhydrazine (Table 2, entry 10)



A mixture of 2-naphthylamine (0.72 g, 5 mmol) and *N*-methyl-*N*-phenylhydrazine (0.61 g, 5 mmol) was heated at 90 ^oC for 10 h (tracked by TLC). After cooled, the reaction mixture was dissolved in a small amount of DMSO and then separated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-methylaniline as colorless oil (0.30 g, 56% yield) and naphthalene-1,2-diamine as a yellow solid (0.31 g, 40% yield). Naphthalene-1,2-diamine: Mp:195-197 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.73 (d, *J* = 3.6 Hz, 1H), 7.71 (d, *J* = 4.0 Hz, 1H), 7.43 (t, *J* = 8.4 Hz, 1H), 7.29 (t, *J* = 7.2 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 1H), 3.70 (brs, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 129.8, 129.4, 128.6, 127.0, 125.4, 124.9, 122.7, 120.1, 119.5, 119.4; IR (KBr, cm^{-1}): 3401.7, 3287.9, 2957.3, 2853.1, 1724.7, 1624.2, 1599.2, 1113.0, 806.6; HRMS (ESI) calcd. for: $C_{10}H_{11}N_2$ $[M+H]^+$ 159.0922, found *m/z* 159.0921.

Reaction of 1-naphthalenamine with *N*-methyl-*N*-phenylhydrazine (Table 2, entry 11)



A mixture of 1-naphthalenamine (0.72 g, 5 mmol) and *N*-methyl-*N*-phenylhydrazine (0.61 g, 5 mmol) was heated at 90 ^oC for 10 h (tracked by TLC). After cooled, the reaction material was dissolved in a small amount of DMSO and then separated by flash column chromatography on silica gel (gradient elution hexanes →1:1 / hexanes : ethyl acetate) to provide *N*-methylaniline as colorless oil (0.19 g, 36% yield) and naphthalene-1,2-diamine as yellow solid (0.08 g, 10% yield).

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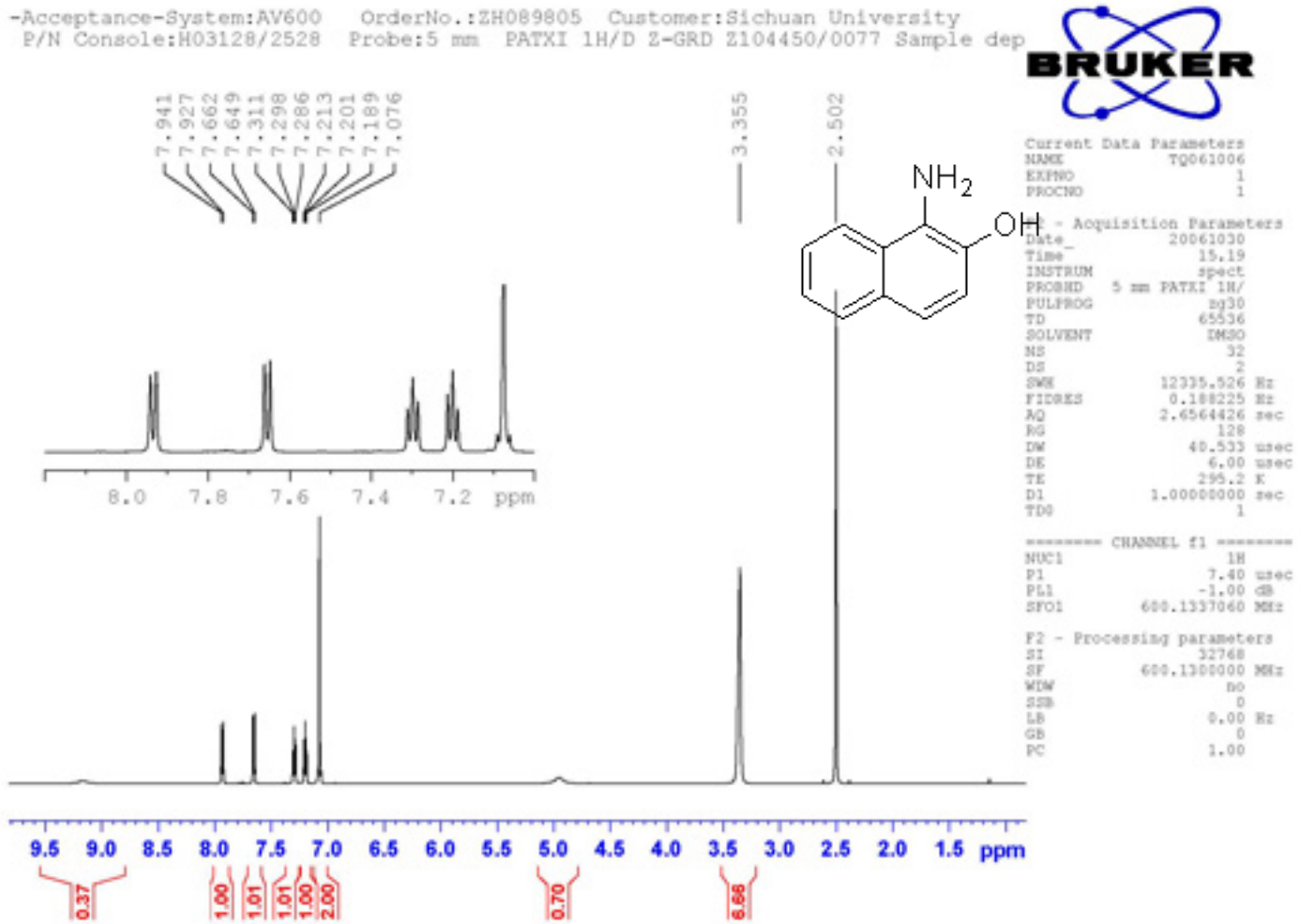
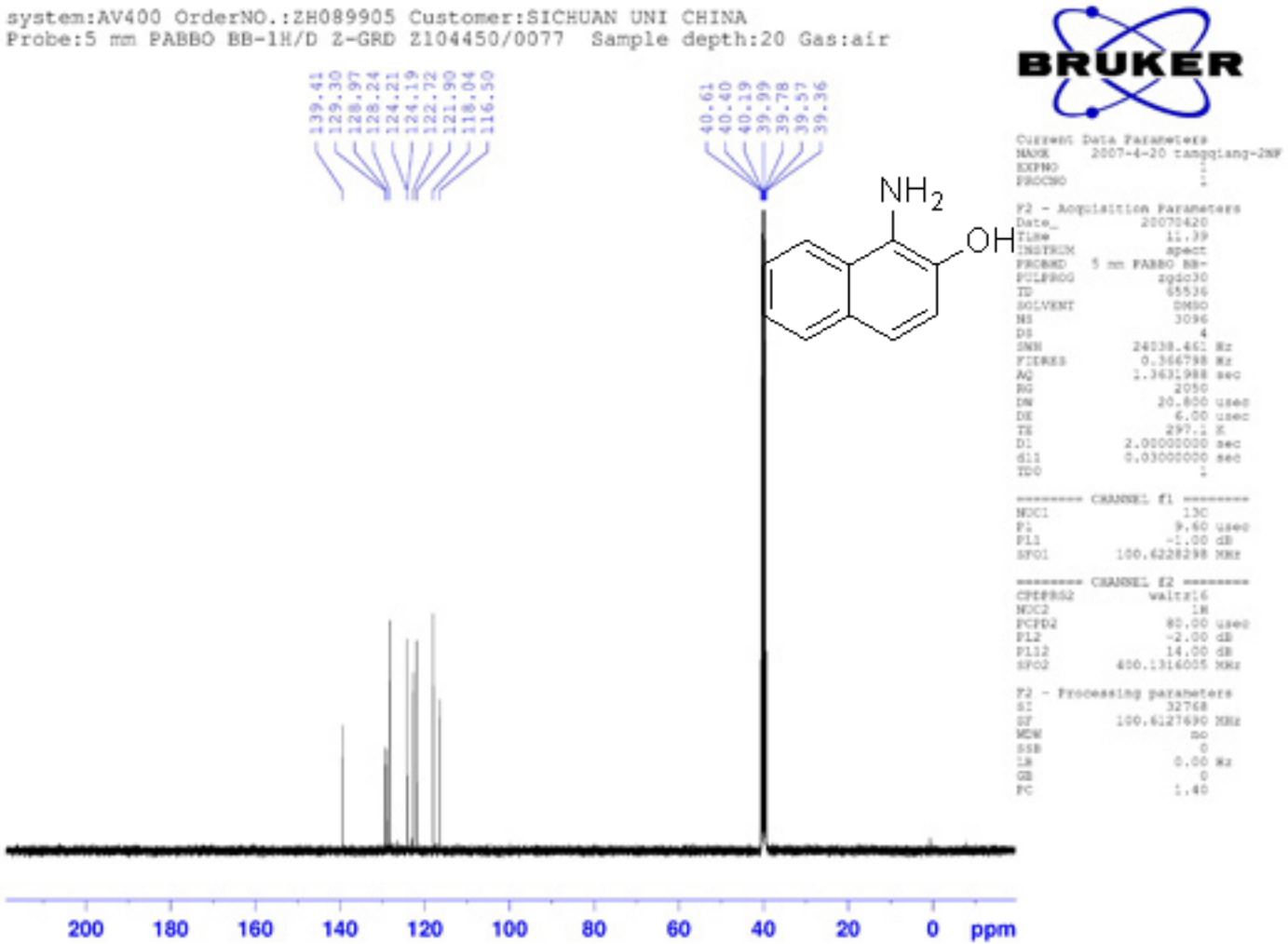
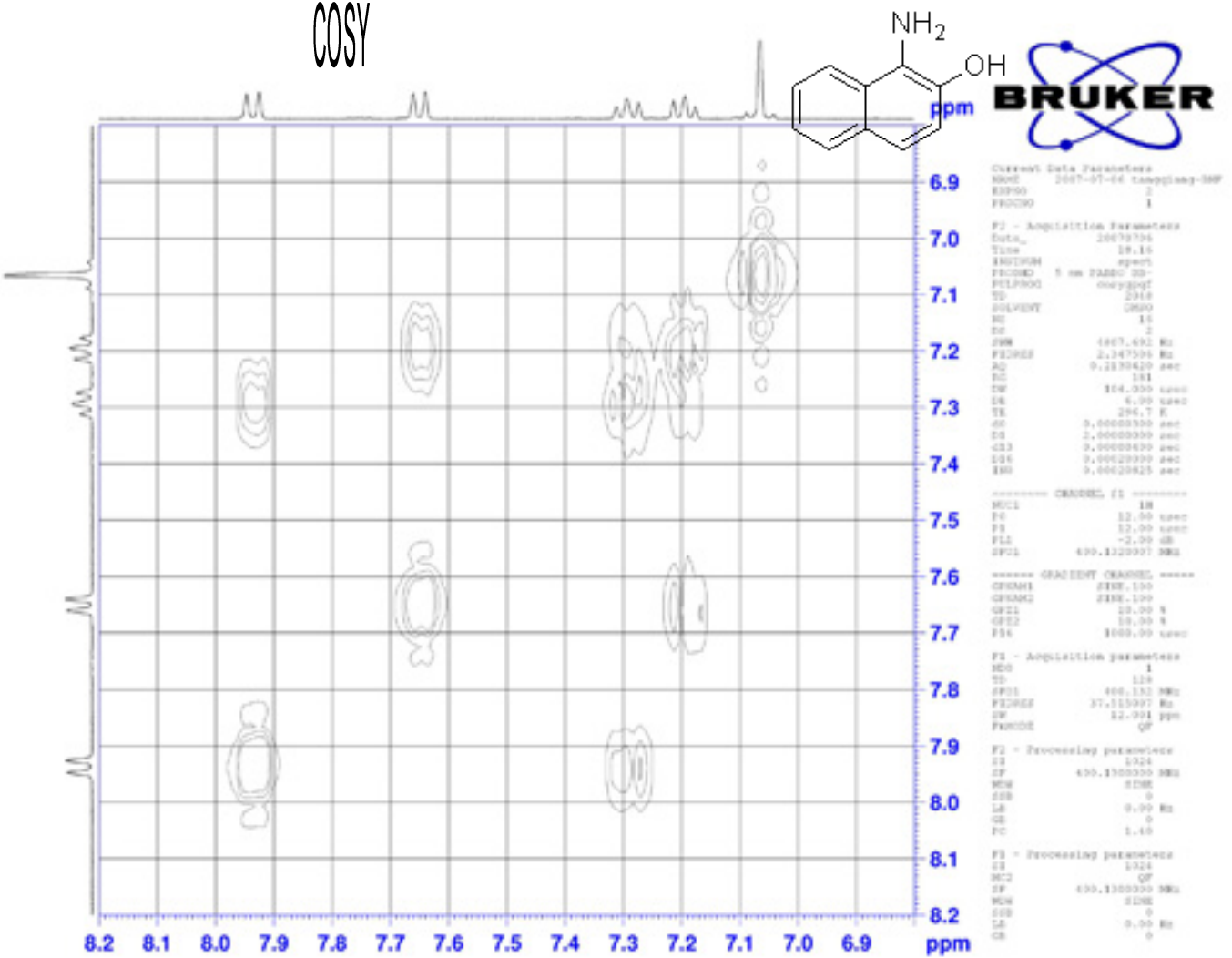
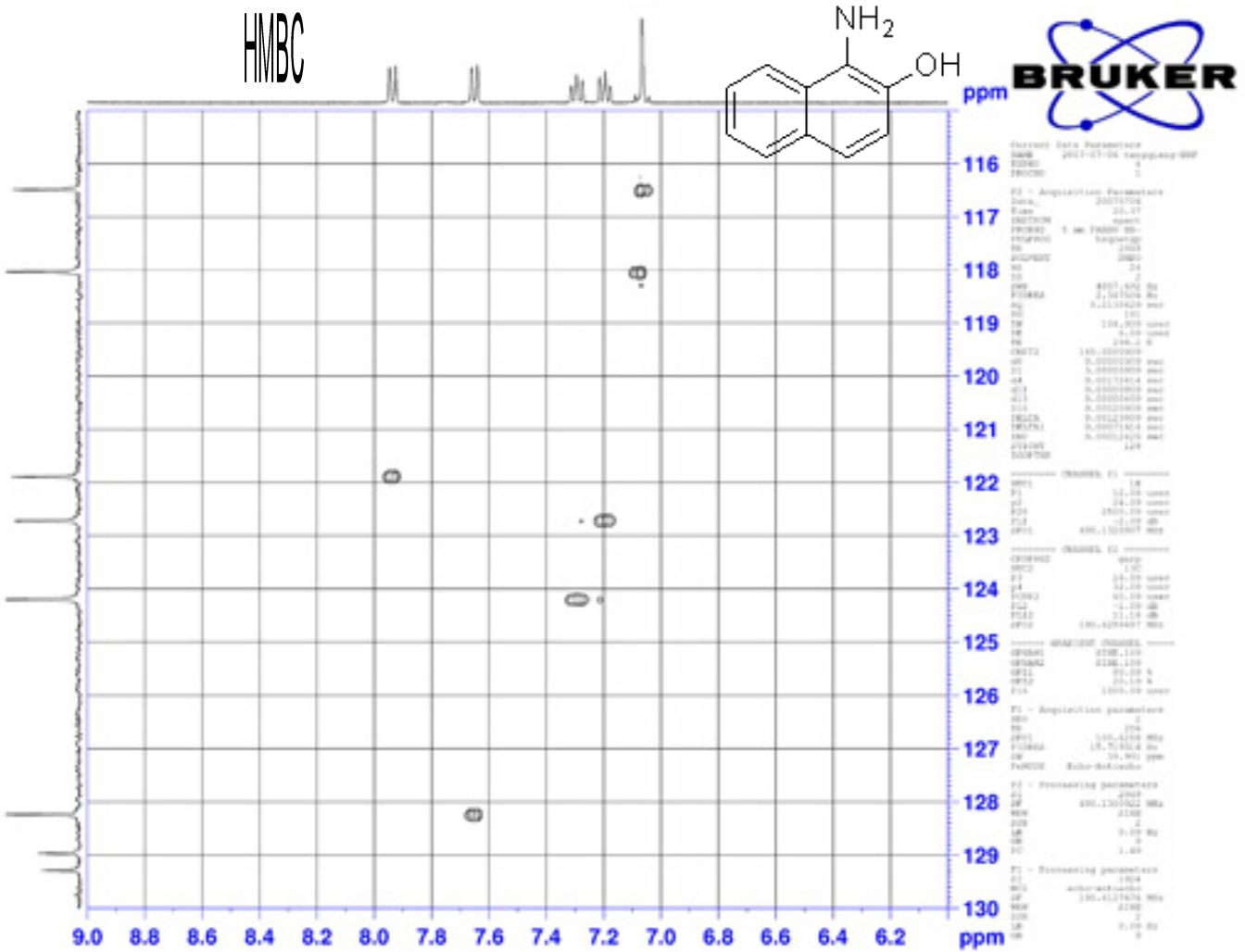
(12) Musser, J. H.; Jones, H.; Sciortino, S.; Bailey, K.; Coutts, S. M.; Khandwala, A.; Sonnino-Goldman, P.; Leibowitz, M.; Wolf, P.; Neiss, E. S. *J. Med. Chem.* **1985**, *28*, 1255-1259.

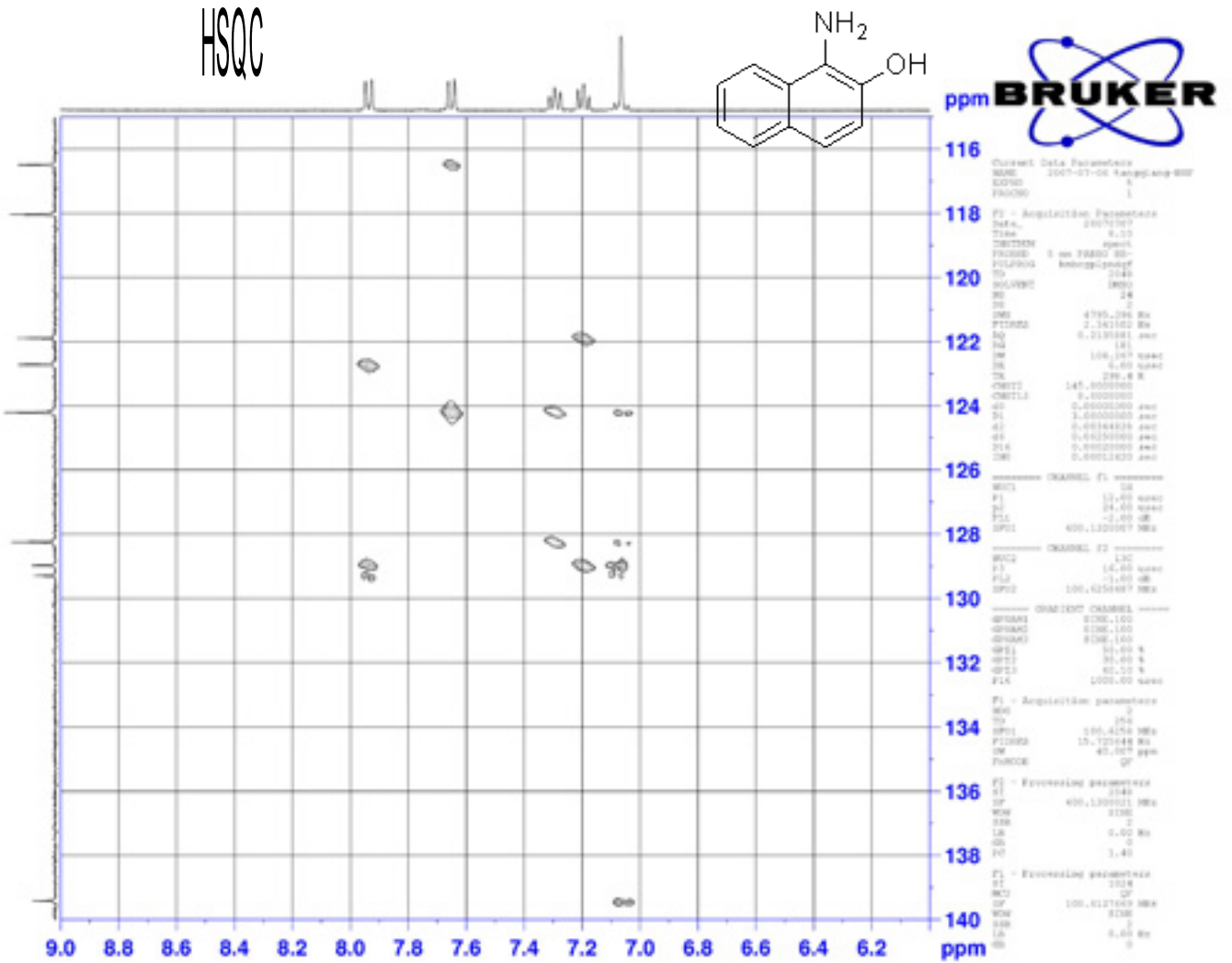

-Acceptance-System:AV600 OrderNo.:ZH089805 Customer:Sichuan University
P/N Console:H03128/2528 Probe:5 mm PATXI 1H/D Z-GRD Z104450/0077 Sample dep
BRUKER
7.941
7.927
7.662
7.649
7.311
7.298
7.286
7.213
7.201
7.189
7.076
3.355
2.502
NH2
OH
8.0
7.8
7.6
7.4
7.2
ppm
Current Data Parameters
NAME TQ061006
EXPNO 1
PROCNO 1
F1 - Acquisition Parameters
Date_ 20061030
Time 15.19
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 32
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 128
DW 40.533 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.40 usec
PL1 -1.00 dB
SFO1 600.1337060 MHz
F2 - Processing parameters
SI 32768
SF 600.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9.5 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 2.0 1.5 ppm
0.37
1.00
1.01
1.01
1.00
2.00
0.70
6.66

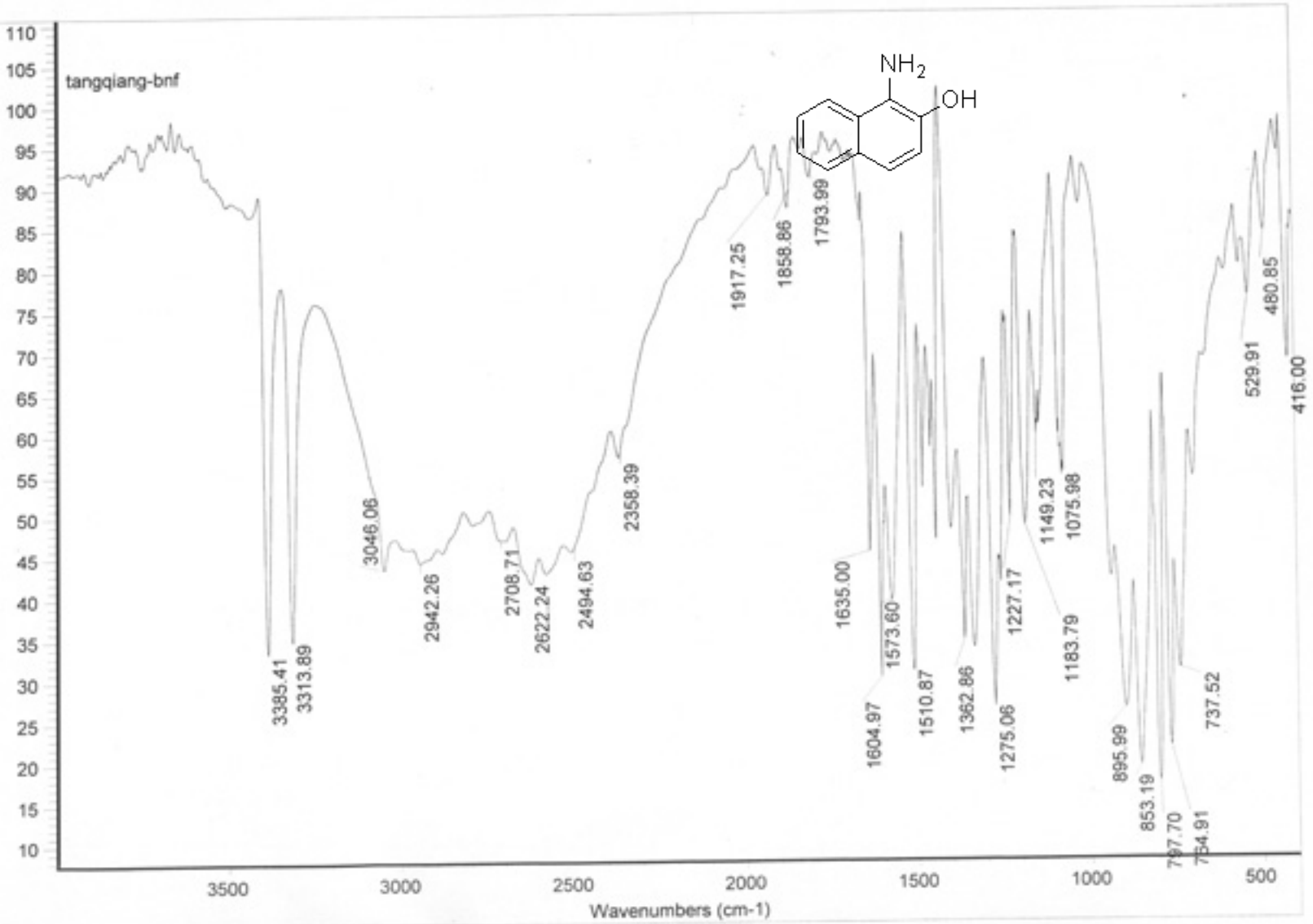

system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
139.41
129.30
128.97
128.24
124.21
124.19
122.72
121.90
118.04
116.50
40.61
40.40
40.19
39.99
39.78
39.57
39.36
NH2
OH
BRUKER
Current Data Parameters
NAME 2007-4-20 tangqiang-2NF
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070420
Time 11.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT DMSO
NS 3096
DS 4
SWH 24038.462 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 297.1 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

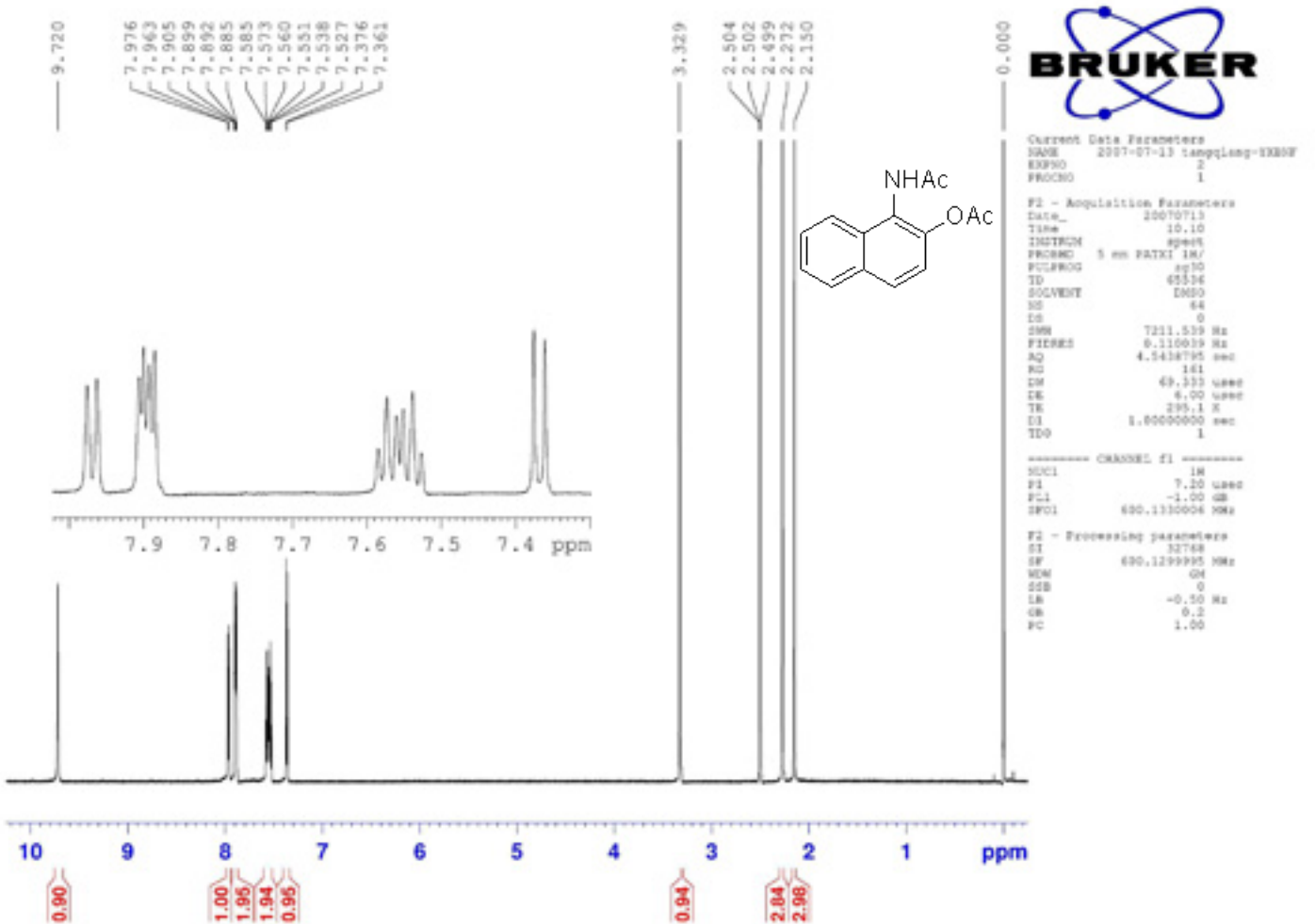
COSY


NH2
OH
BRUKER
ppm
6.9
7.0
7.1
7.2
7.3
7.4
7.5
7.6
7.7
7.8
7.9
8.0
8.1
8.2
8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 ppm


HMBC
NH2
OH
BRUKER
ppm
116
117
118
119
120
121
122
123
124
125
126
127
128
129
130
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
ppm


HSQC
NH2
OH
BRUKER
ppm
116
118
120
122
124
126
128
130
132
134
136
138
140
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
ppm


tangqiang-bnf
NH2
OH
3385.41
3313.89
3046.06
2942.26
2708.71
2622.24
2494.63
2358.39
1917.25
1858.86
1793.99
1635.00
1604.97
1573.60
1510.87
1362.86
1275.06
1227.17
1183.79
1149.23
1075.98
895.99
853.19
797.70
754.91
737.52
529.91
480.85
416.00
Wavenumbers (cm-1)



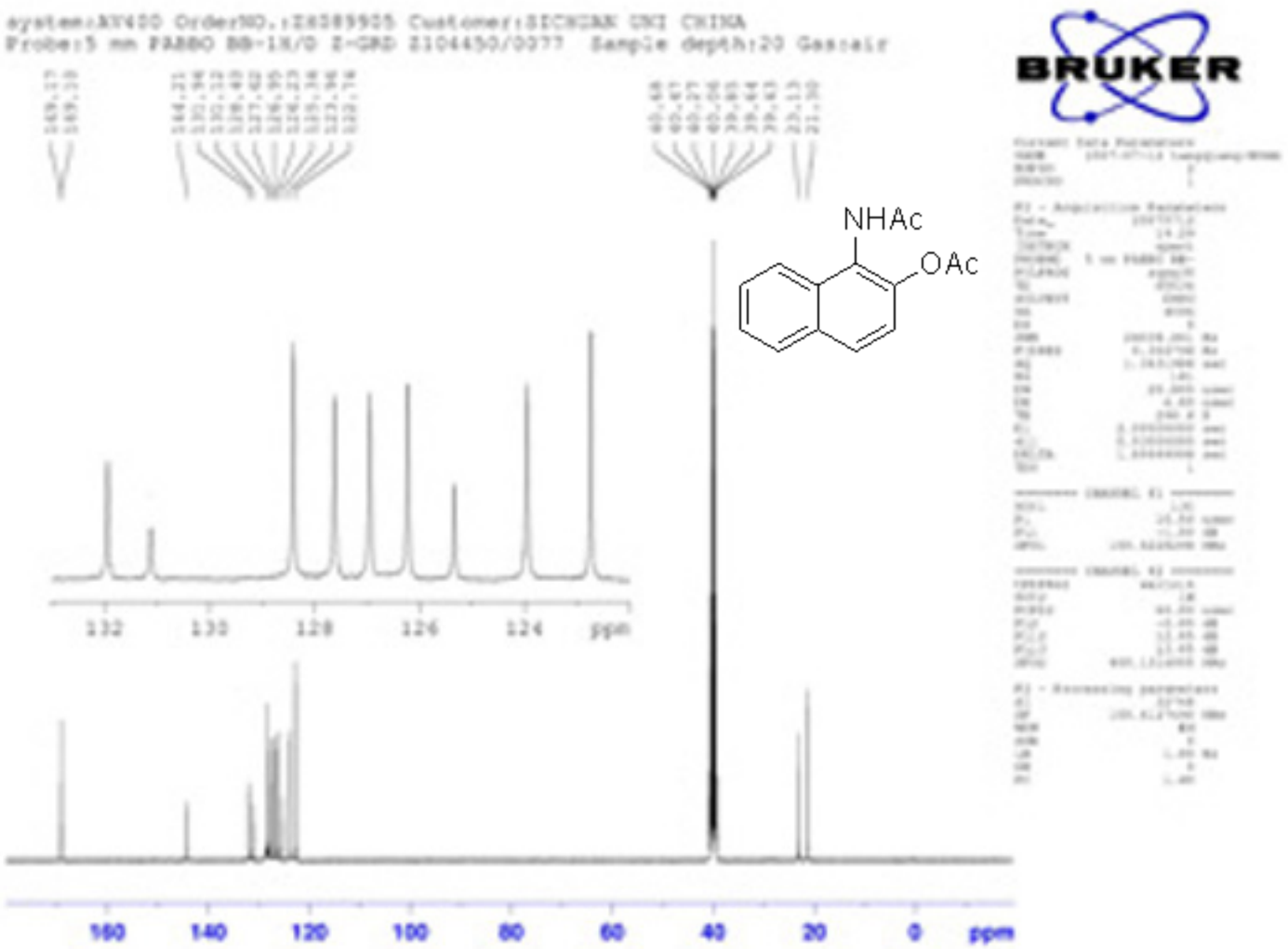
BRUKER

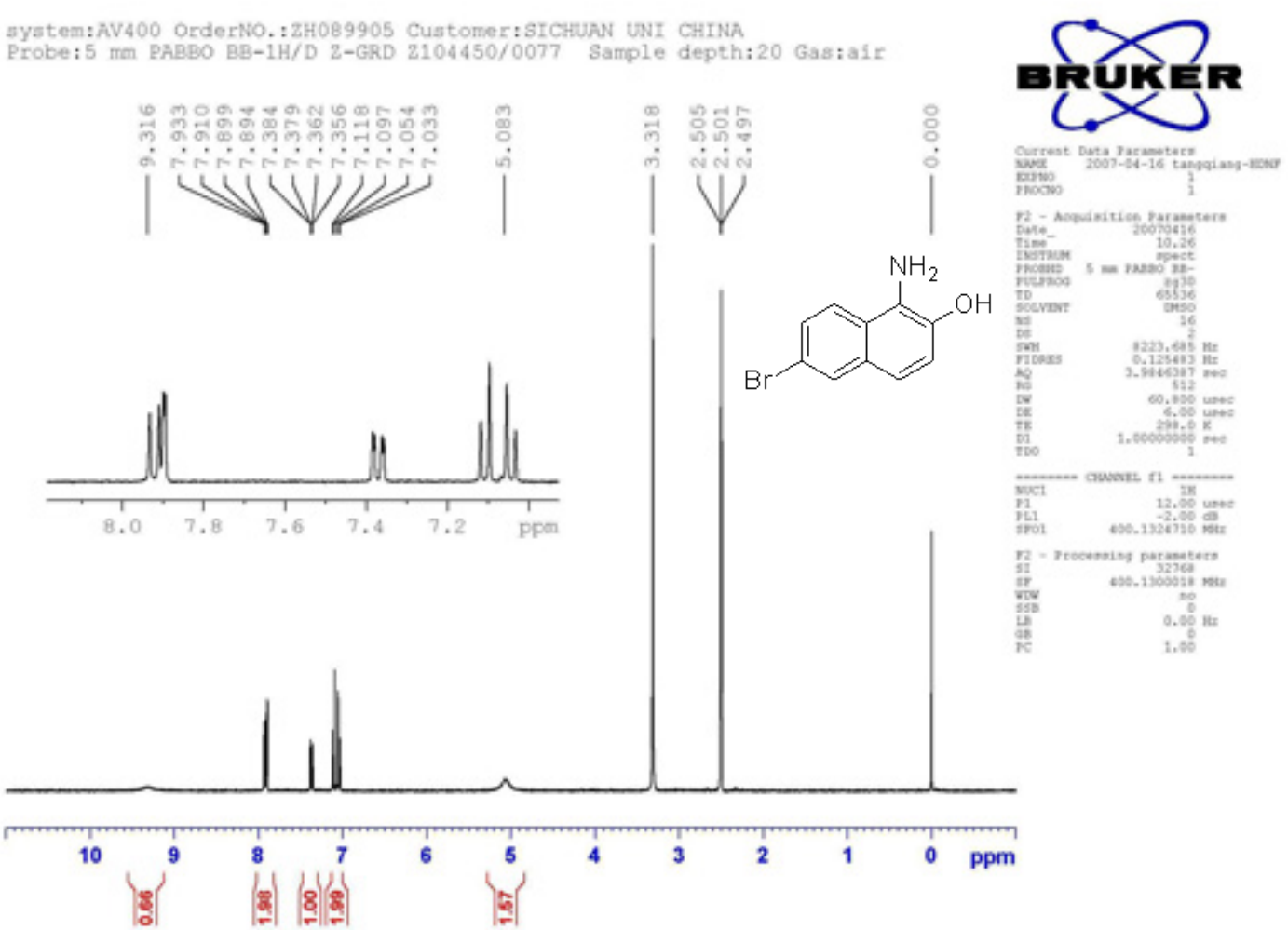
```
Current Data Parameters
NAME        2007-07-13 tangqiang-YXBOY
EXPNO                2
PROCNO               1

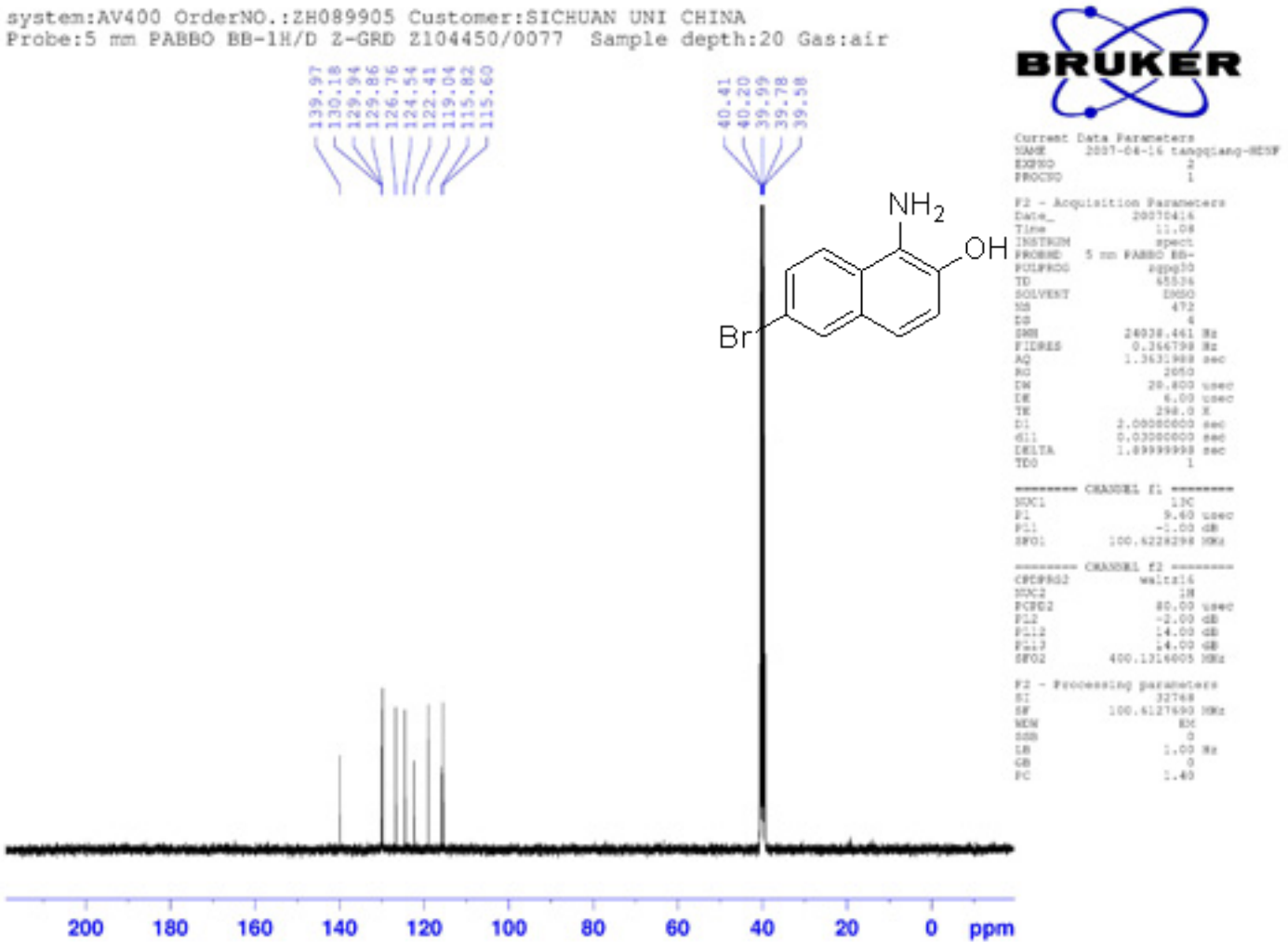
F2 - Acquisition Parameters
Date_           20070713
Time              10.10
INSTRM              spect
PROBHD   5 mm PATXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    64
DS                     0
SWH             7211.539 Hz
FIDRES          0.110039 Hz
AQ              4.5438795 sec
RG                   161
DW                49.333 usec
DE                 6.00 usec
TE                295.1 K
D1              1.00000000 sec
TD0                    1

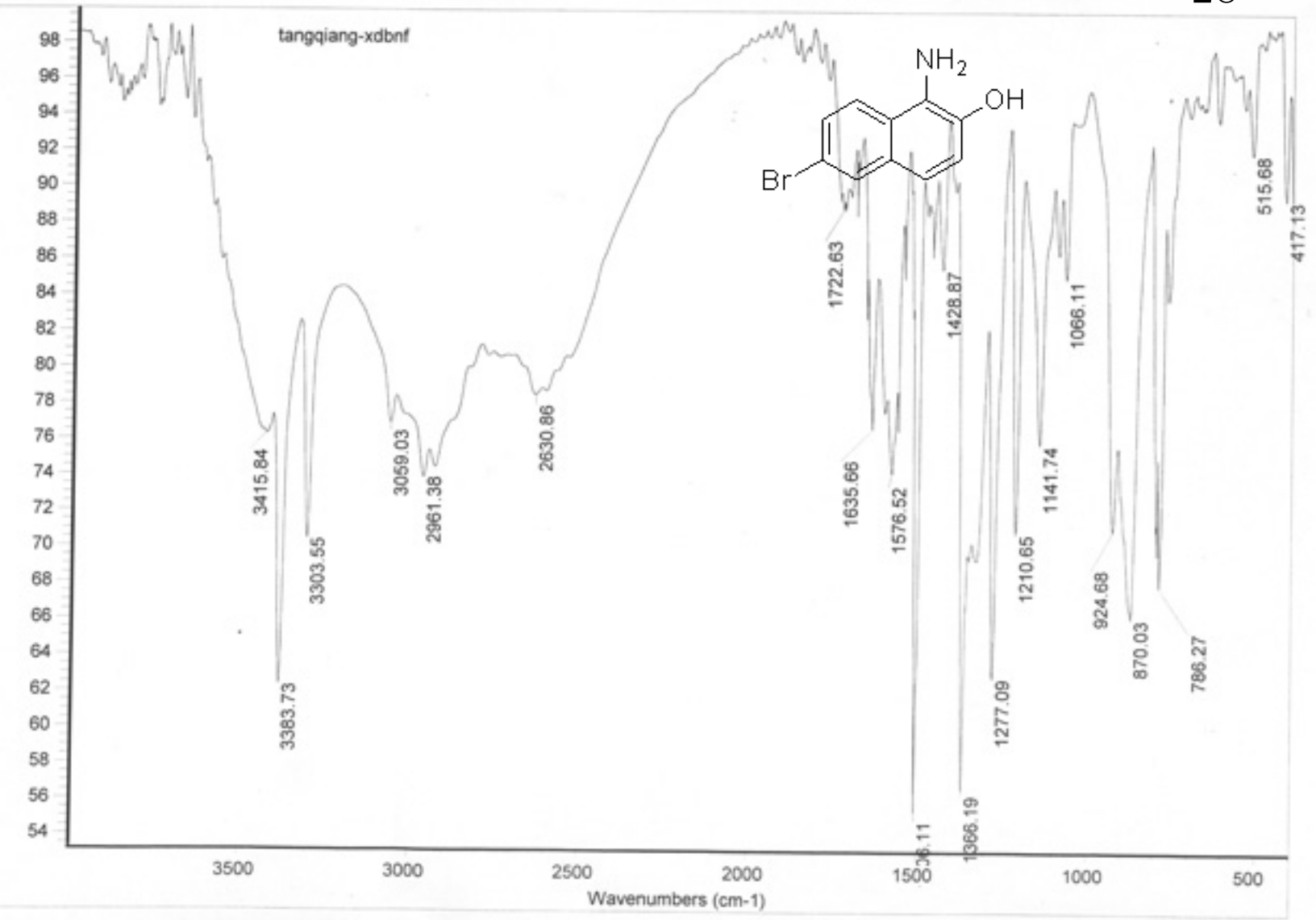
======== CHANNEL f1 ========
NUC1                  1H
P1                 7.20 usec
PL1               -1.00 dB
SFO1        600.1330006 MHz

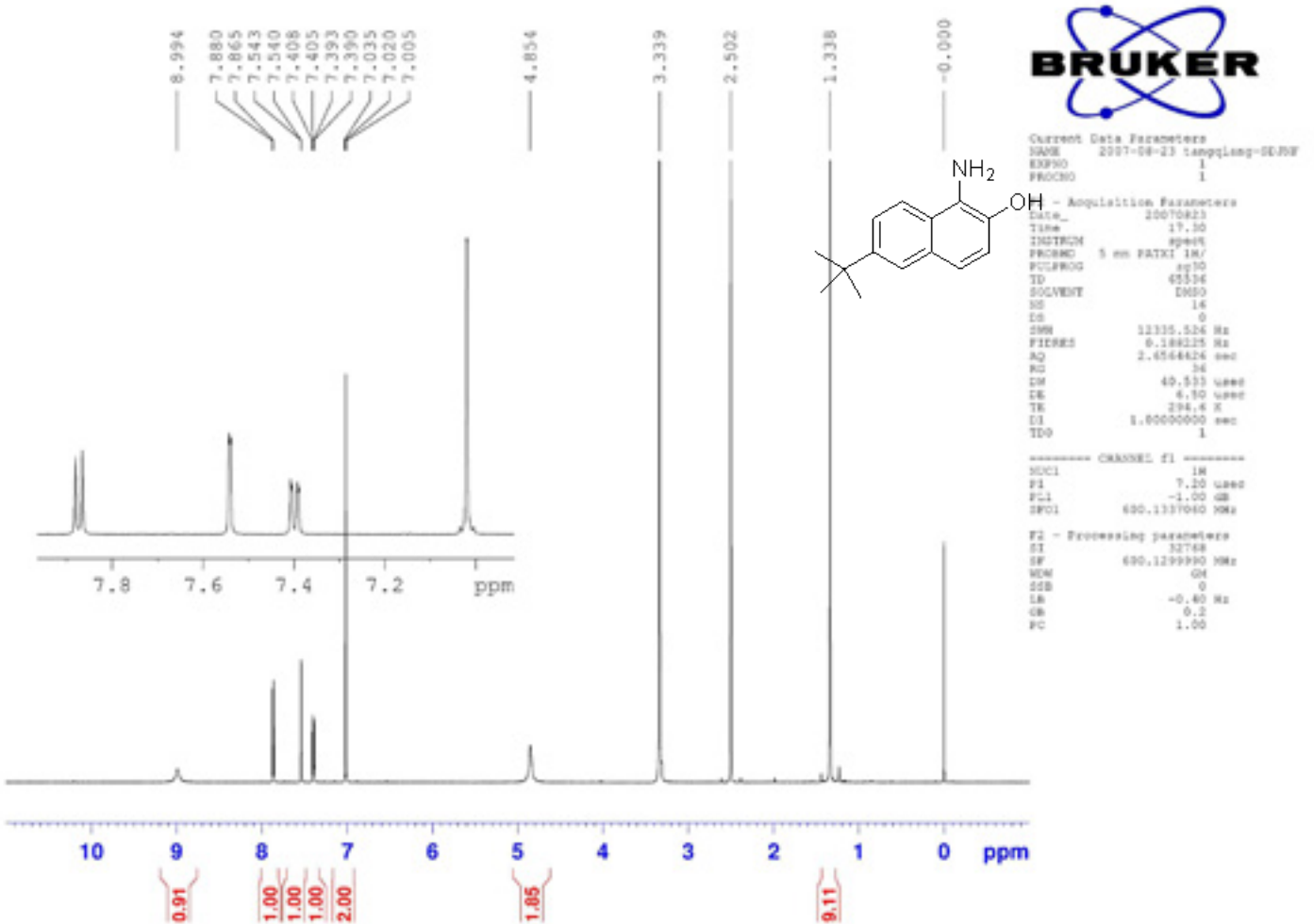
F2 - Processing parameters
SI                 32768
SF          600.1299995 MHz
WDW                   EM
SSB                    0
LB                -0.50 Hz
GB                   0.2
PC                  1.00
```


NHAc
OAc
132
130
128
126
124
ppm
160
140
120
100
80
60
40
20
0
ppm


system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
BRUKER
9.316
7.933
7.910
7.899
7.894
7.384
7.379
7.362
7.356
7.118
7.097
7.054
7.033
5.083
3.318
2.505
2.501
2.497
0.000
NH2
OH
Br
8.0
7.8
7.6
7.4
7.2
ppm
10
9
8
7
6
5
4
3
2
1
0
ppm
0.66
1.98
1.00
1.99
1.67
Current Data Parameters
NAME 2007-04-16 tangqiang-HDNF
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070416
Time 10.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 512
DW 60.800 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300019 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00


system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
BRUKER
139.97
130.18
129.94
129.86
126.76
124.54
122.41
119.04
115.82
115.60
40.41
40.20
39.99
39.78
39.58
NH2
OH
Br
Current Data Parameters
NAME 2007-04-16 tangqiang-HESF
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070416
Time 11.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 472
DS 4
SWH 24038.461 Hz
FIDRES 0.366799 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.00 dB
SFO1 100.6224298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.00 dB
PL13 14.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


tangqiang-xdbnf
NH2
OH
Br
3415.84
3383.73
3303.55
3059.03
2961.38
2630.86
1722.63
1635.66
1576.52
1506.11
1428.87
1366.19
1277.09
1210.65
1141.74
1066.11
924.68
870.03
786.27
515.68
417.13
Wavenumbers (cm-1)



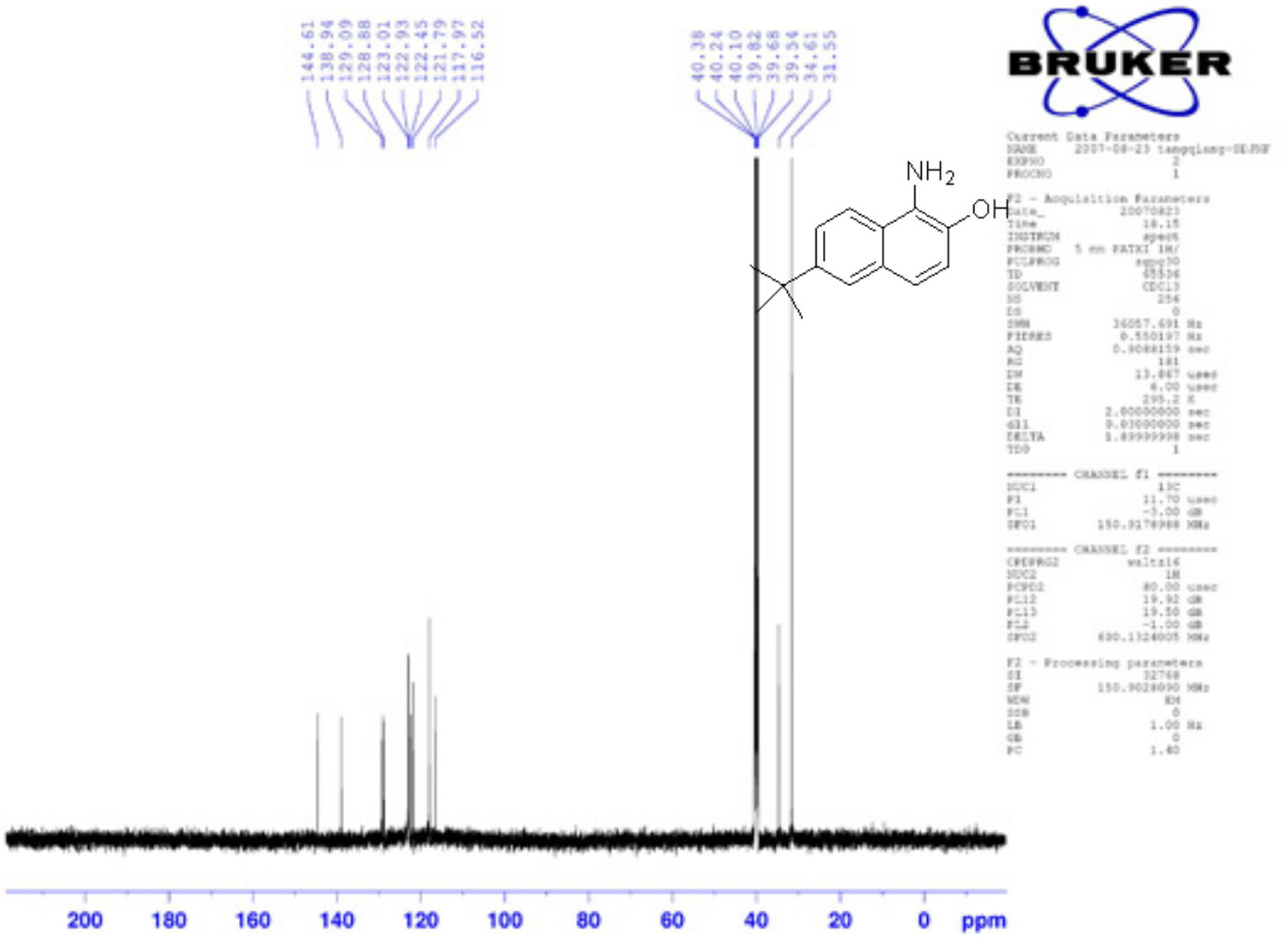
BRUKER

Current Data Parameters
NAME 20070823 tangqiang-SDJW
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070823
Time 17.30
INSTRUM spect
PROBHD 5 mm PATXI 1H/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564624 sec
RG 36
DW 40.533 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 7.20 usec
PL1 -1.00 dB
SFO1 600.1337060 MHz

F2 - Processing parameters
SI 32768
SF 600.1299990 MHz
WDW EM
SSB 0
LB -0.40 Hz
GB 0.2
PC 1.00



BRUKER

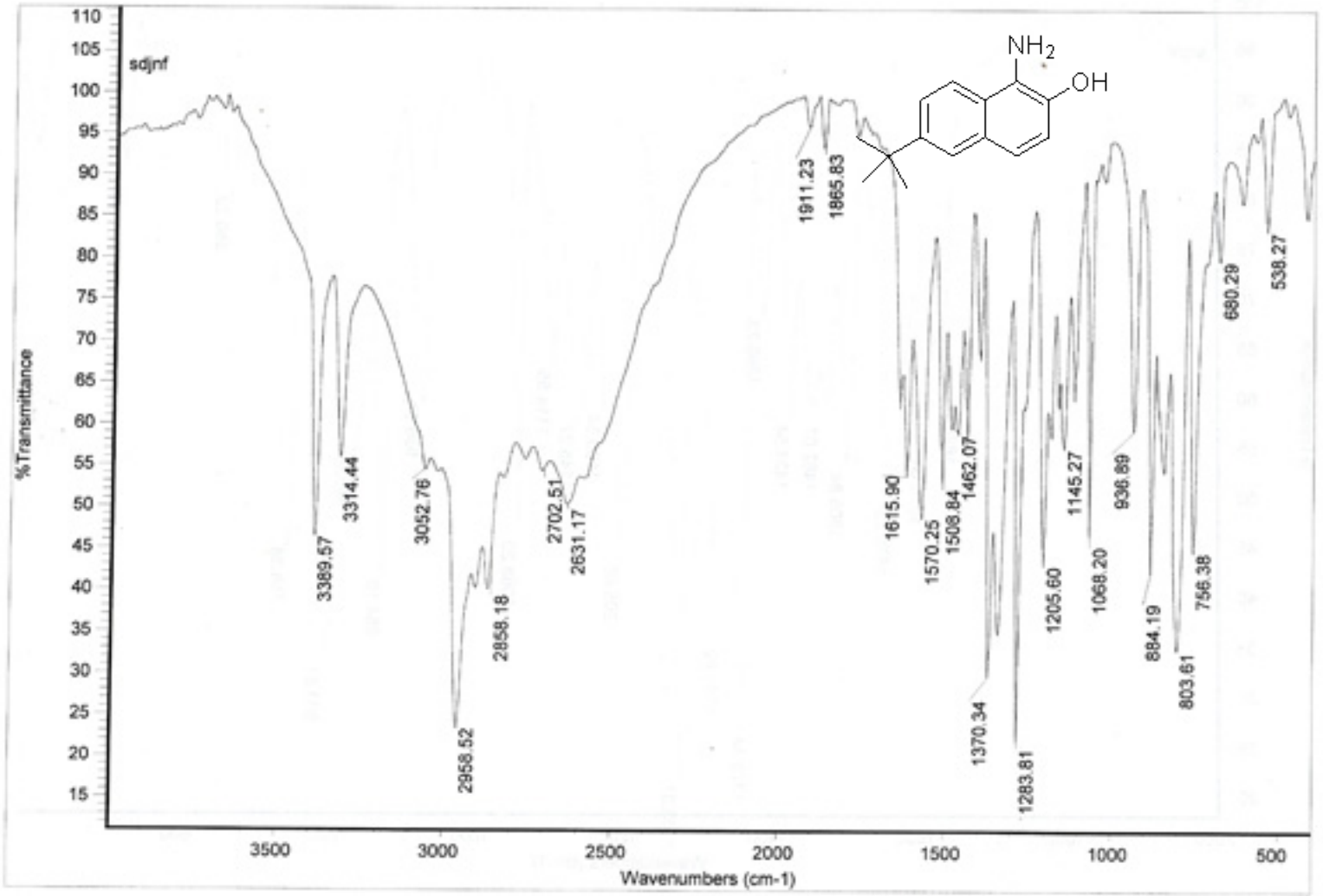
```
Current Data Parameters
NAME      2007-08-23 tangqiang-SD-NW
EXPNO                2
PROCNO               1

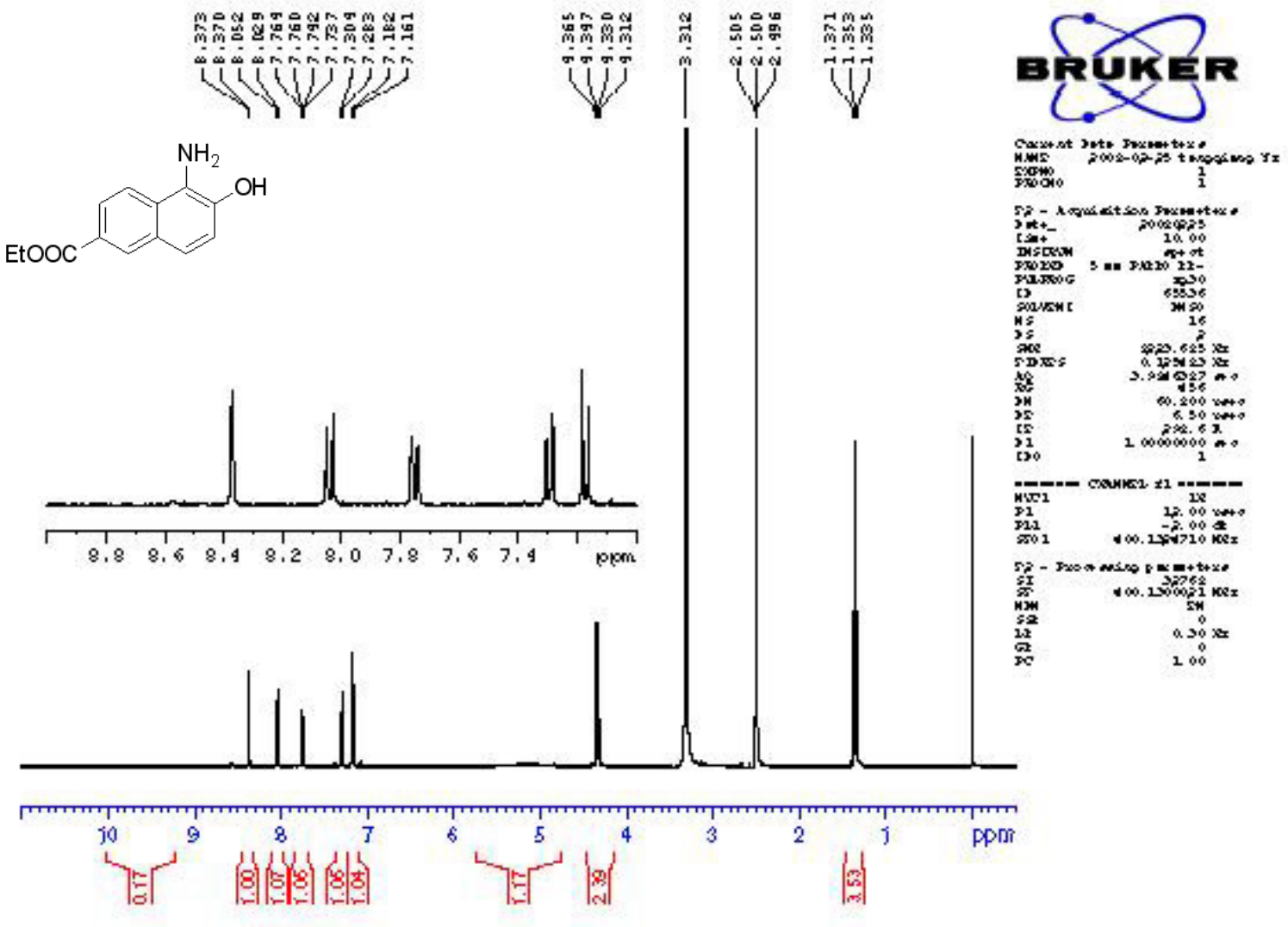
F2 - Acquisition Parameters
Date_          20070823
Time              18.15
INSTRM              spect
PROBHD   5 mm PATXI 1H/
PULPROG             zgpg30
TD                 65536
SOLVENT             CDCl3
NS                    256
DS                      0
SWH           36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9088159 sec
RG                    181
DW               13.867 usec
DE                  6.00 usec
TE                295.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

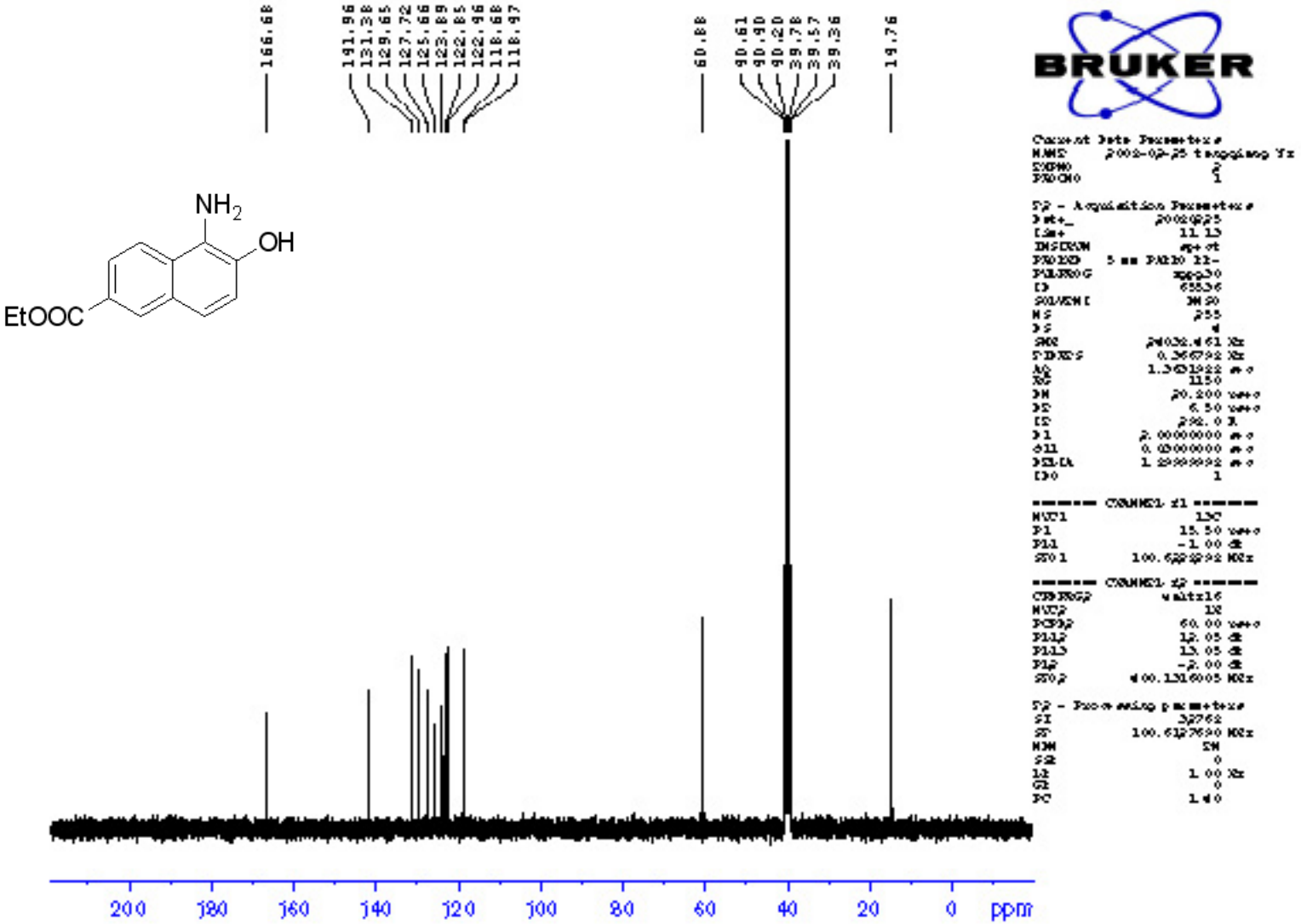
======== CHANNEL f1 ========
NUC1                 13C
P1                 11.70 usec
PL1                -3.00 dB
SFO1         150.9178988 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL12               19.92 dB
PL13               19.50 dB
PL2                -1.00 dB
SFO2         600.1324005 MHz

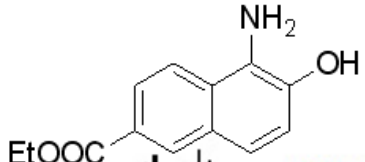
F2 - Processing parameters
SI                  32768
SF           150.9028490 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

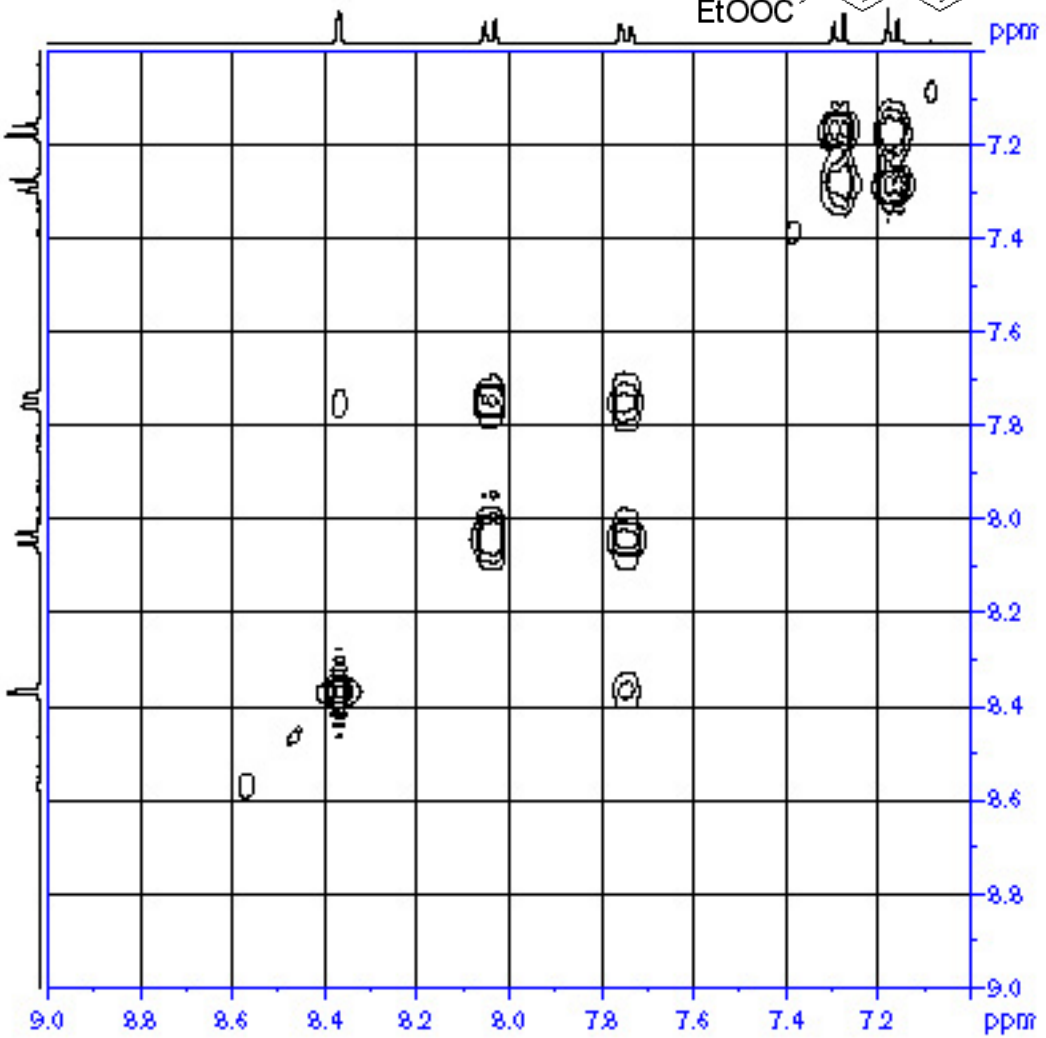

sdjnf
%Transmittance
Wavenumbers (cm-1)
3389.57
3314.44
3052.76
2958.52
2858.18
2702.51
2631.17
1911.23
1865.83
1615.90
1570.25
1508.84
1462.07
1370.34
1283.81
1205.60
1145.27
1068.20
936.89
884.19
803.61
756.38
680.29
538.27
NH2
OH


8.373
8.370
8.052
8.029
7.769
7.760
7.742
7.737
7.309
7.283
7.182
7.161
9.365
9.347
9.330
9.312
3.312
2.505
2.500
2.496
1.371
1.353
1.335
NH2
OH
EtOOC
8.8 8.6 8.4 8.2 8.0 7.8 7.6 7.4 ppm
10 9 8 7 6 5 4 3 2 1 ppm
0.17
1.00
1.07
1.08
1.06
1.04
1.17
2.39
3.63
BRUKER

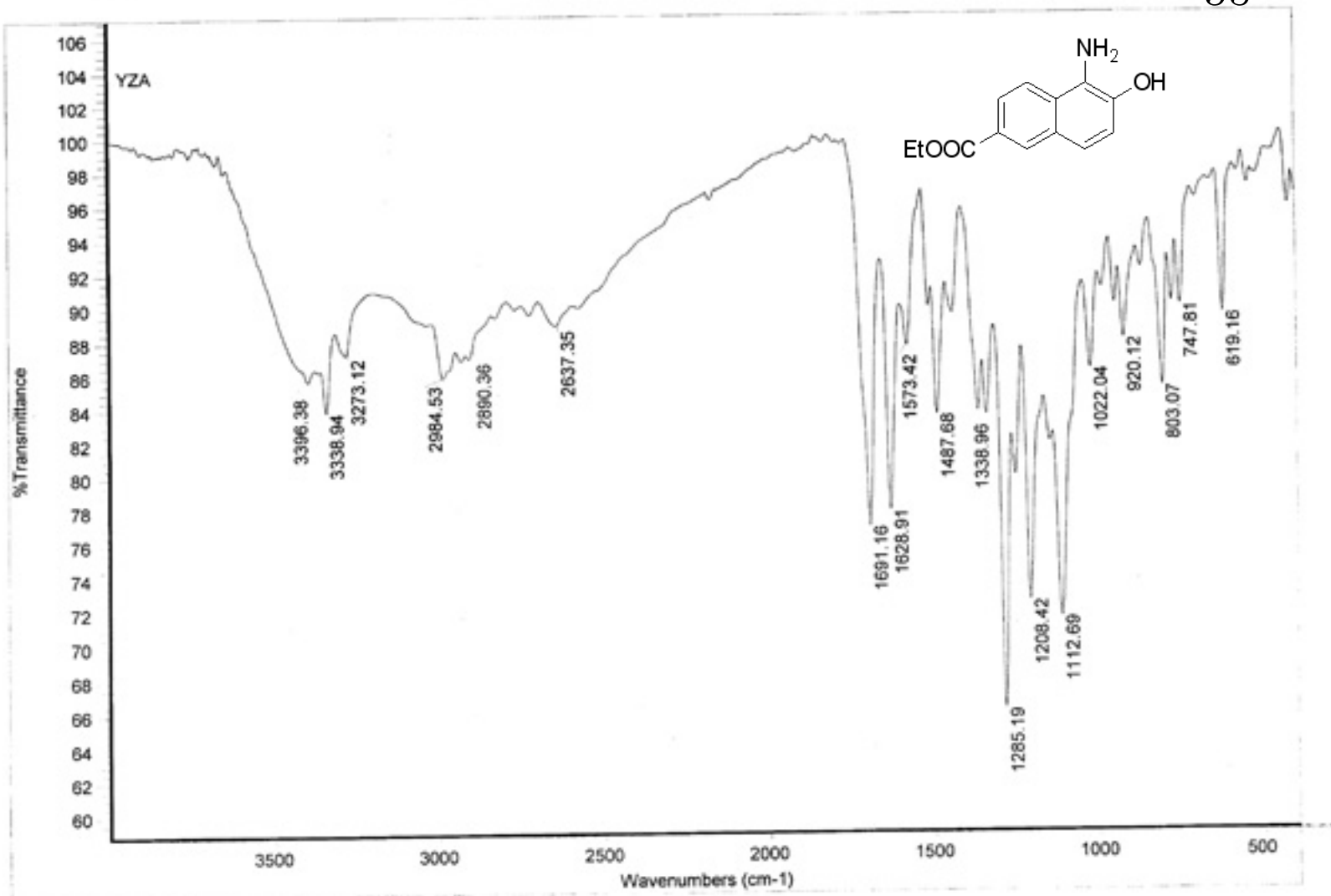

166.68
191.96
131.38
129.65
127.72
125.66
123.89
122.85
122.96
118.68
118.97
60.88
90.61
90.90
90.20
39.78
39.57
39.36
19.76
NH2
OH
EtOOC
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER

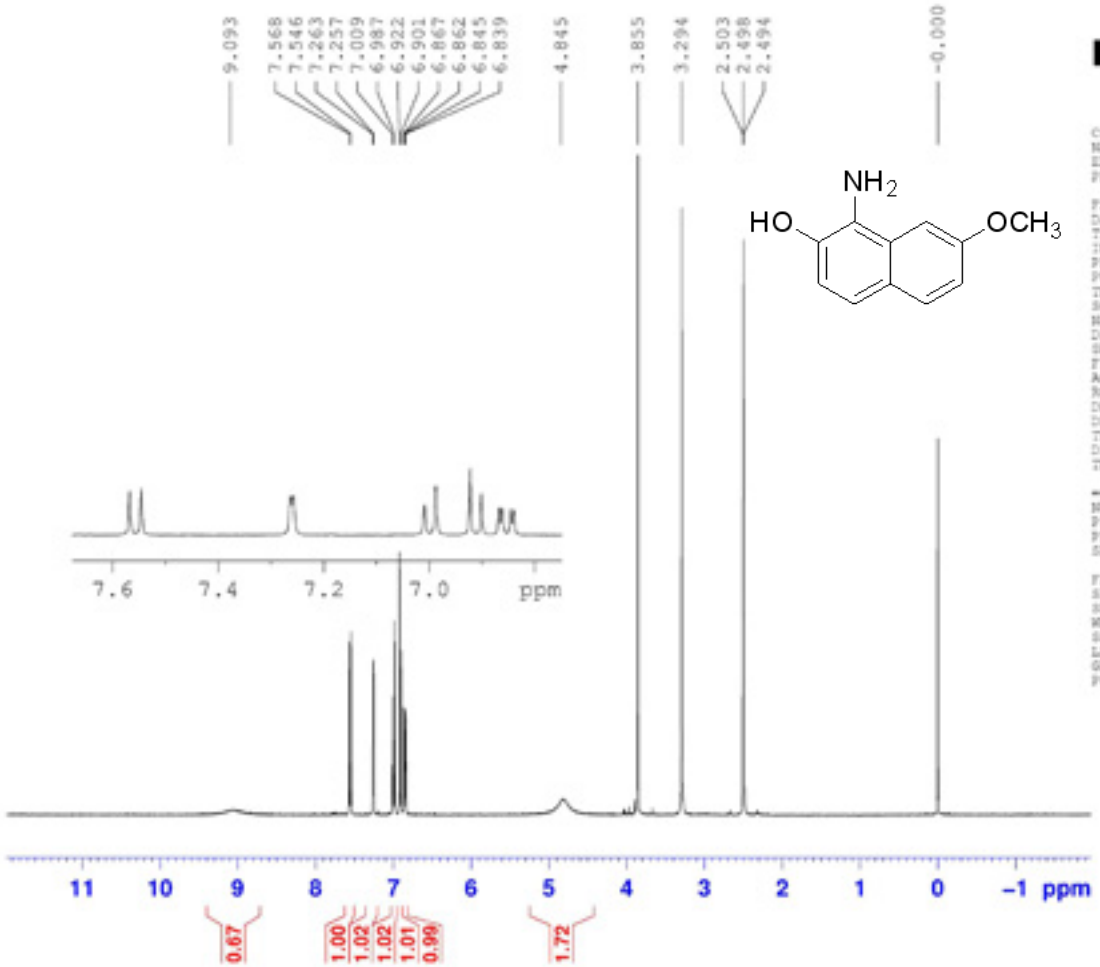
COSY


NH2
OH
EtOOC


ppm
7.2
7.4
7.6
7.8
8.0
8.2
8.4
8.6
8.8
9.0
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
ppm

BRUKER


YZA
%Transmittance
Wavenumbers (cm-1)
3396.38
3338.94
3273.12
2984.53
2890.36
2637.35
1691.16
1628.91
1573.42
1487.68
1338.96
1285.19
1208.42
1112.69
1022.04
920.12
803.07
747.81
619.16
NH2
OH
EtOOC



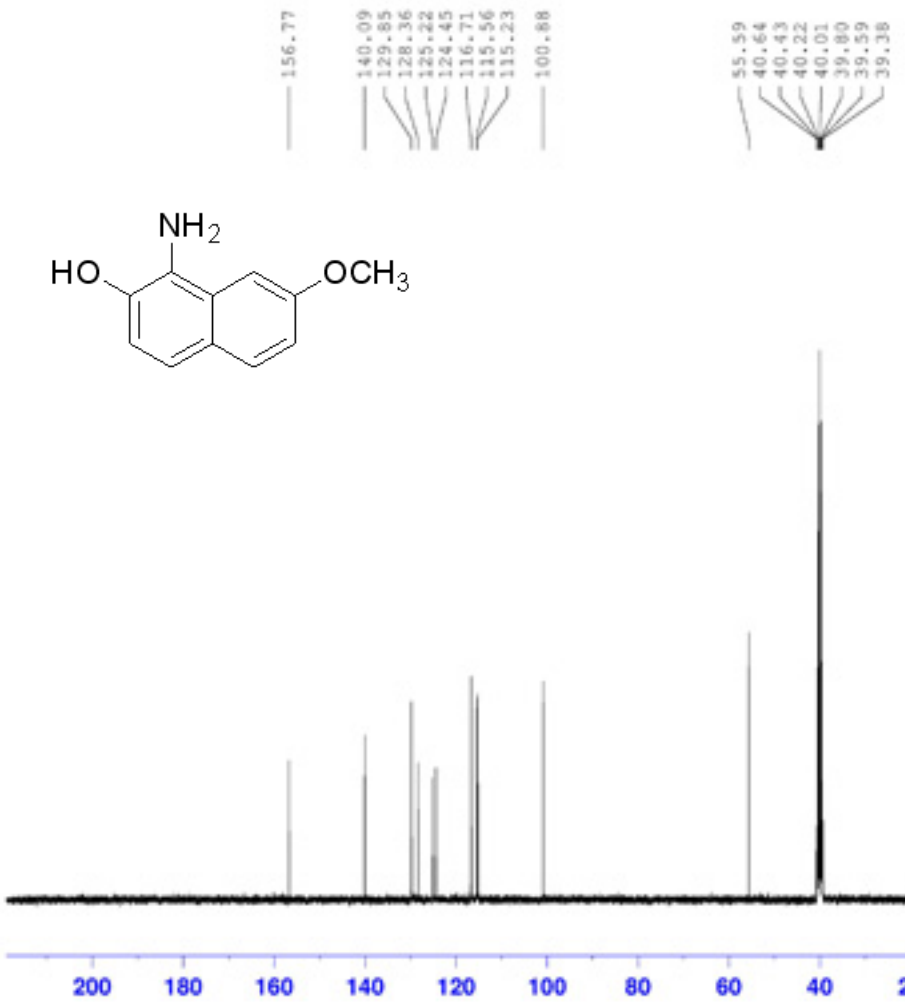
BRUKER

```
Current Data Parameters
NAME      2008-02-19 tangqiang-JJK
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20080219
Time              10.16
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   406
DW                60.800 usec
DE                 6.50 usec
TE                302.5 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300029 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



BRUKER

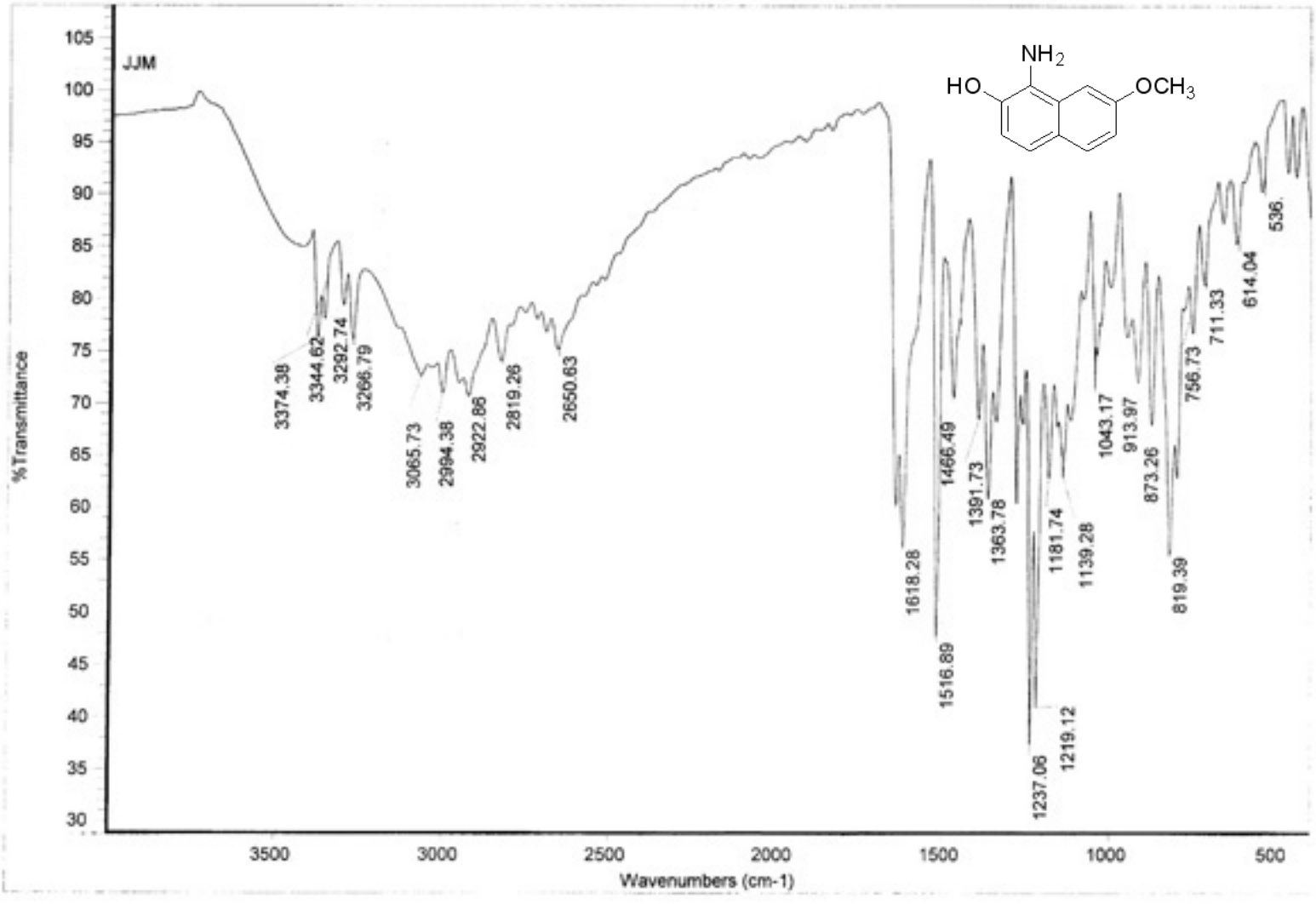
```
Current Data Parameters
NAME      2008-02-19 tangqiang-JJK
EXPNO                 2
PROCNO                1

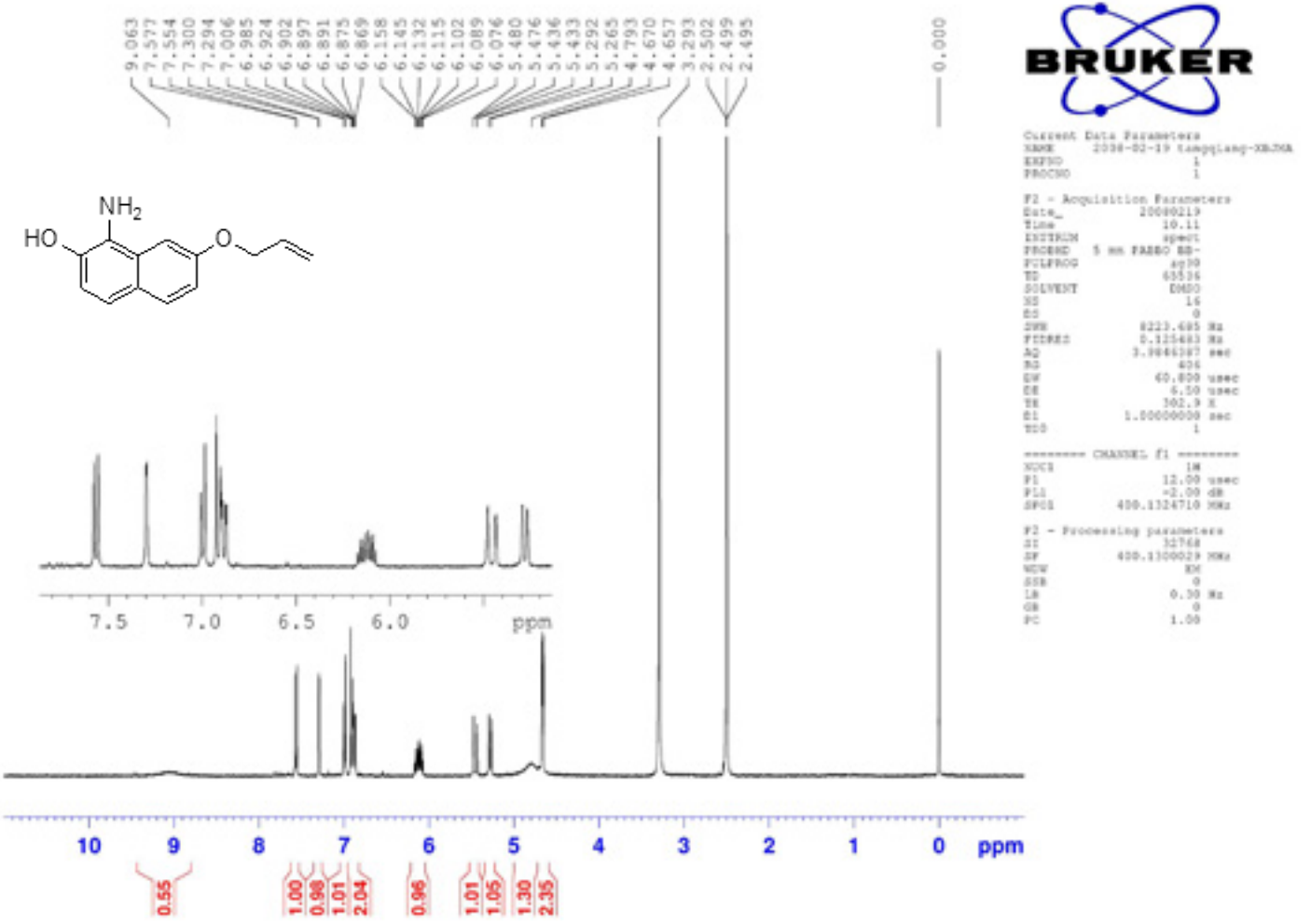
F2 - Acquisition Parameters
Date_           20080219
Time              11.24
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                   149
DS                     0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  2050
DW                20.800 usec
DE                 6.50 usec
TE                302.5 K
D1           2.00000000 sec
GL1          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
PL1               -1.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             80.00 usec
PL12              12.05 dB
PL13              13.05 dB
PL2               -2.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


JJM
%Transmittance
Wavenumbers (cm-1)
3374.38
3344.62
3292.74
3266.79
3065.73
2994.38
2922.86
2819.26
2650.63
1618.28
1516.89
1466.49
1391.73
1363.78
1237.06
1219.12
1181.74
1139.28
1043.17
913.97
873.26
819.39
756.73
711.33
614.04
536.
NH2
HO
OCH3



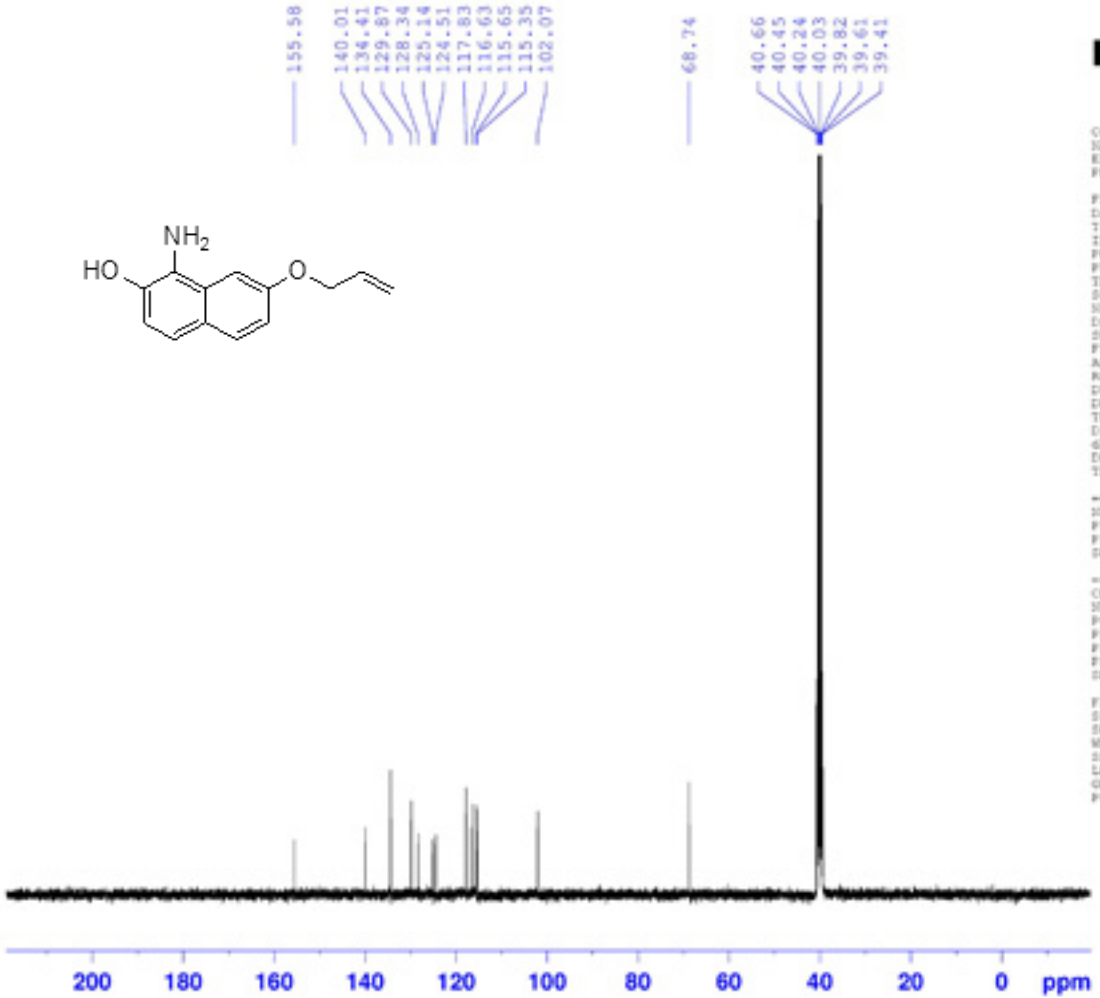
BRUKER

```
Current Data Parameters
NAME      2008-02-19 tangqiang-XBJMA
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080219
Time              10.11
INSTRUM           spect
PROBHD  5 mm PABBO BB-
PULPROG          zg30
TD               65536
SOLVENT           DMSO
NS                   16
DS                    0
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                 406
DW               60.800 usec
DE                6.50 usec
TE              302.9 K
D1              1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                32768
SF           400.1300029 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



BRUKER

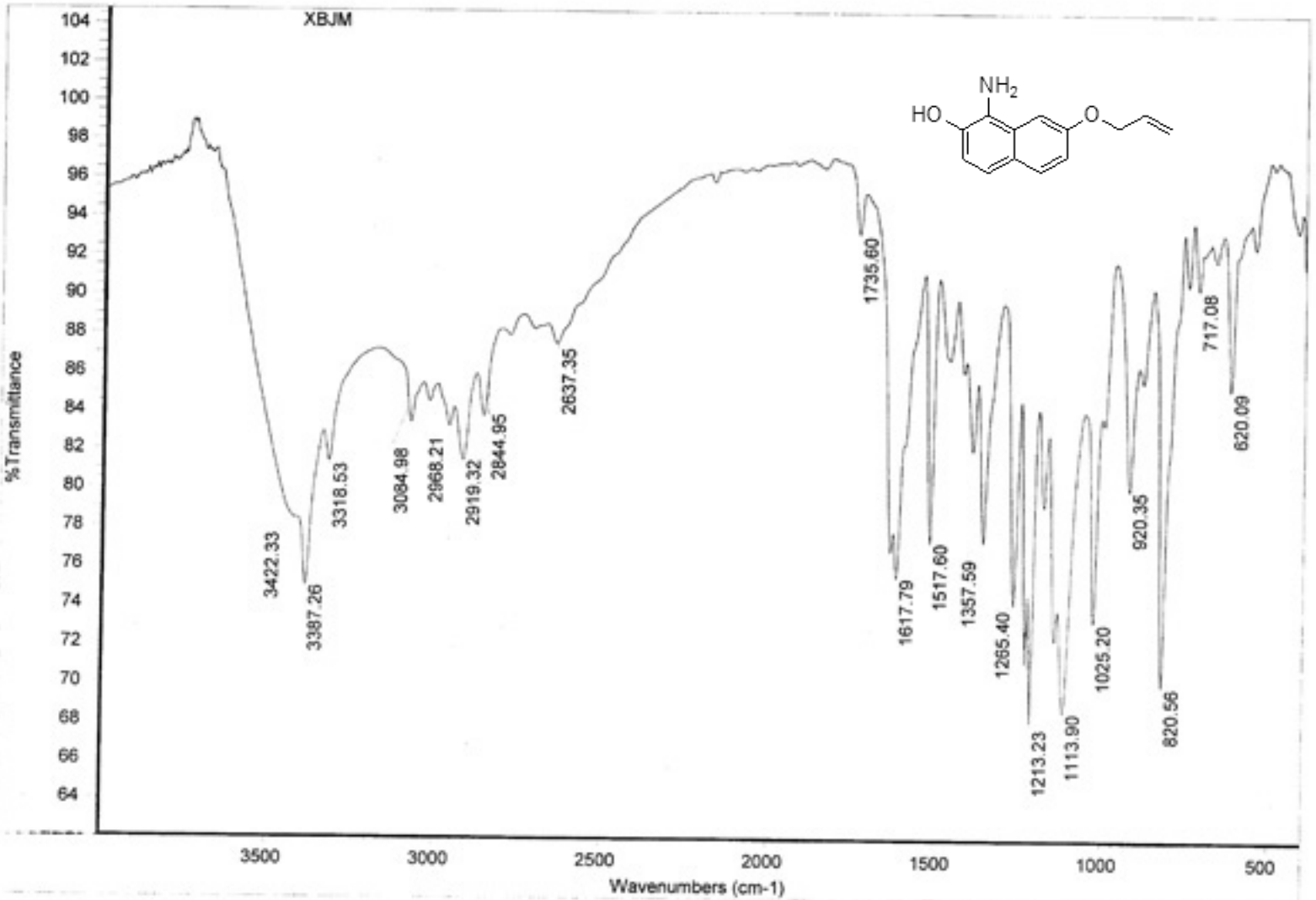
```
Current Data Parameters
NAME      2008-02-19 tangqiang-XBJWA
EXPNO                2
PROCNO               1

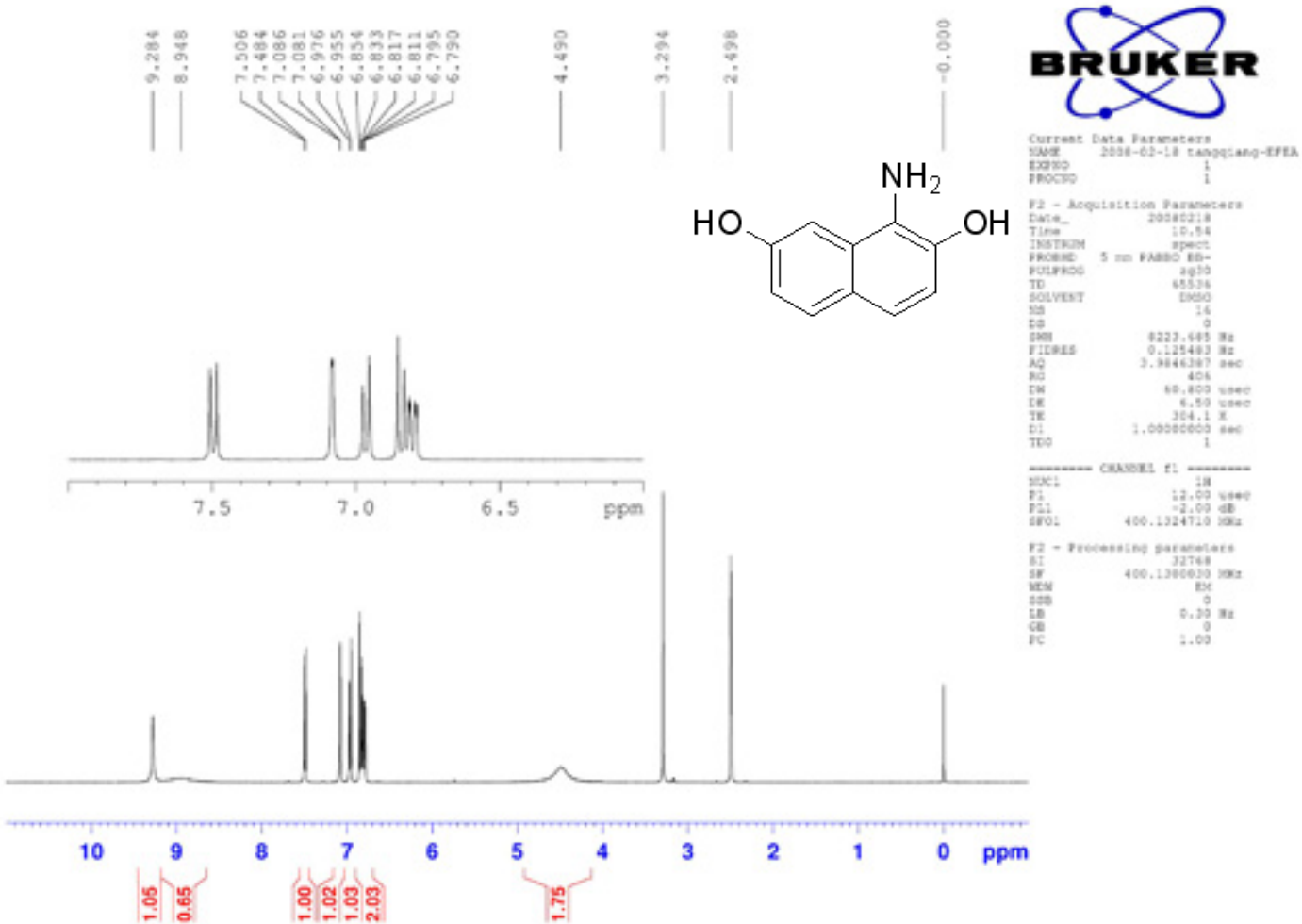
F2 - Acquisition Parameters
Date_           20080219
Time              11.12
INSTRM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                65536
SOLVENT             DMSO
NS                   508
DS                     0
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  2050
DW               20.800 usec
DE                6.50 usec
TE                302.3 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                  15.50 usec
PL1                 -1.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2               60.00 usec
PL12                12.05 dB
PL13                13.05 dB
PL2                 -2.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF            100.6127690 MHz
WDW                    EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


XBJM
%Transmittance
Wavenumbers (cm-1)
3422.33
3387.26
3318.53
3084.98
2668.21
2919.32
2844.95
2637.35
1735.60
1617.79
1517.60
1357.59
1265.40
1213.23
1113.90
1025.20
920.35
820.56
717.08
620.09
NH2
HO
O



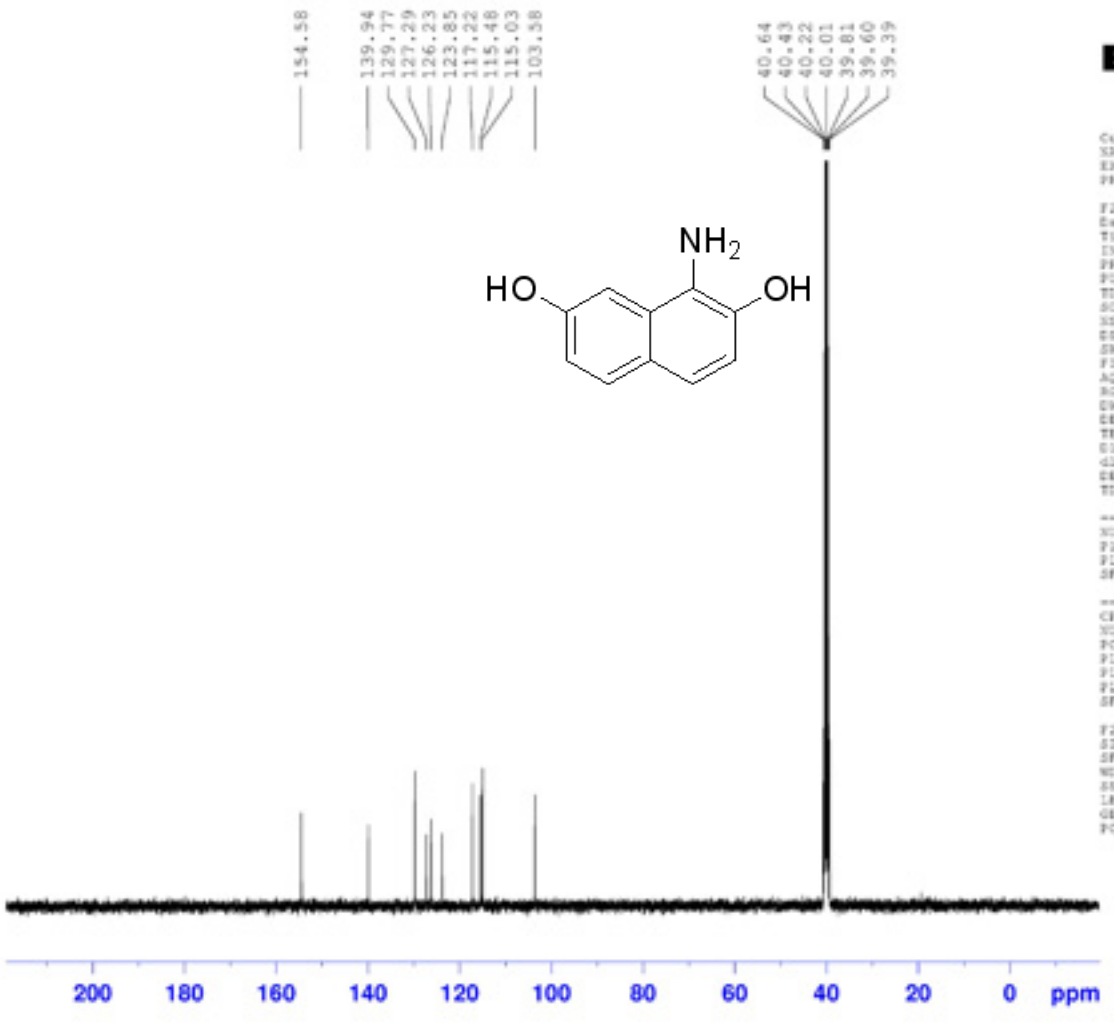
BRUKER

```
Current Data Parameters
NAME      20080218 tangqiang-EFEA
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_          20080218
Time               10.54
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                   406
DW              60.800 usec
DE               6.50 usec
TE              304.1 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -2.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 32768
SW          400.1300000 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



BRUKER

```
Current Data Parameters
NAME      2008-02-18 tangqiang-EFRA
EXPNO                 2
PROCNO                1

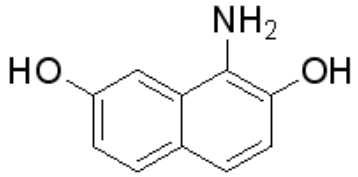
F2 - Acquisition Parameters
Date_          20080218
Time              11.29
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD               65536
SOLVENT           DMSO
NS                  257
DS                    0
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                  181
DW               20.800 usec
DE                6.50 usec
TE              363.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                15.50 usec
PL1               -1.00 dB
SFO1        100.6228298 MHz

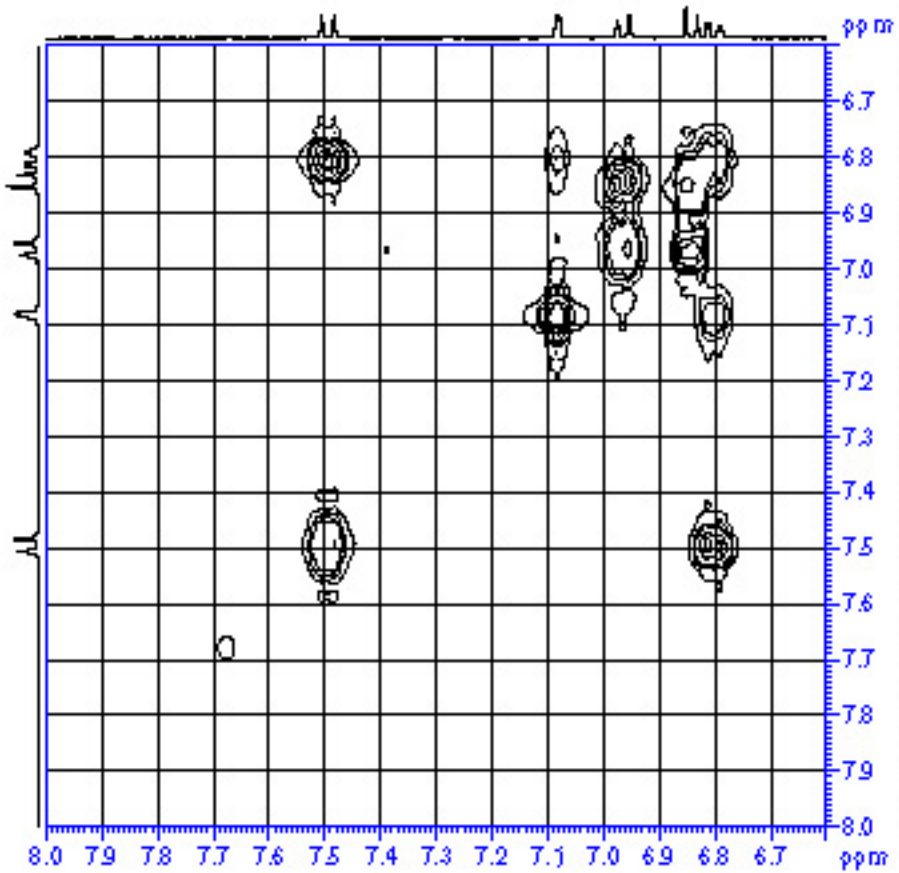
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             60.00 usec
PL12              12.05 dB
PL13              13.05 dB
PL2               -2.00 dB
SFO2        400.1316005 MHz

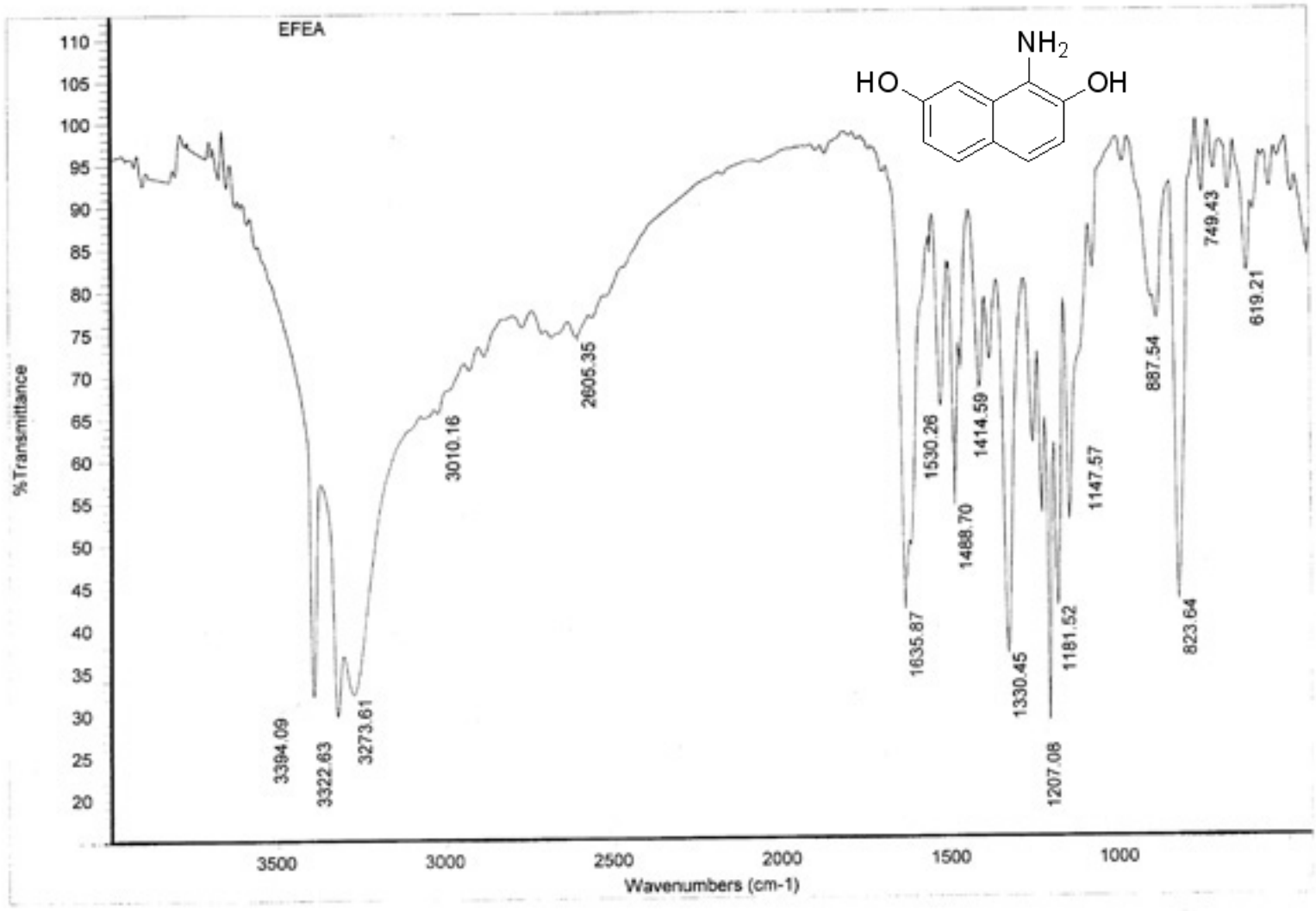
F2 - Processing parameters
SI                32768
SF          100.6127690 MHz
WDW                 EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

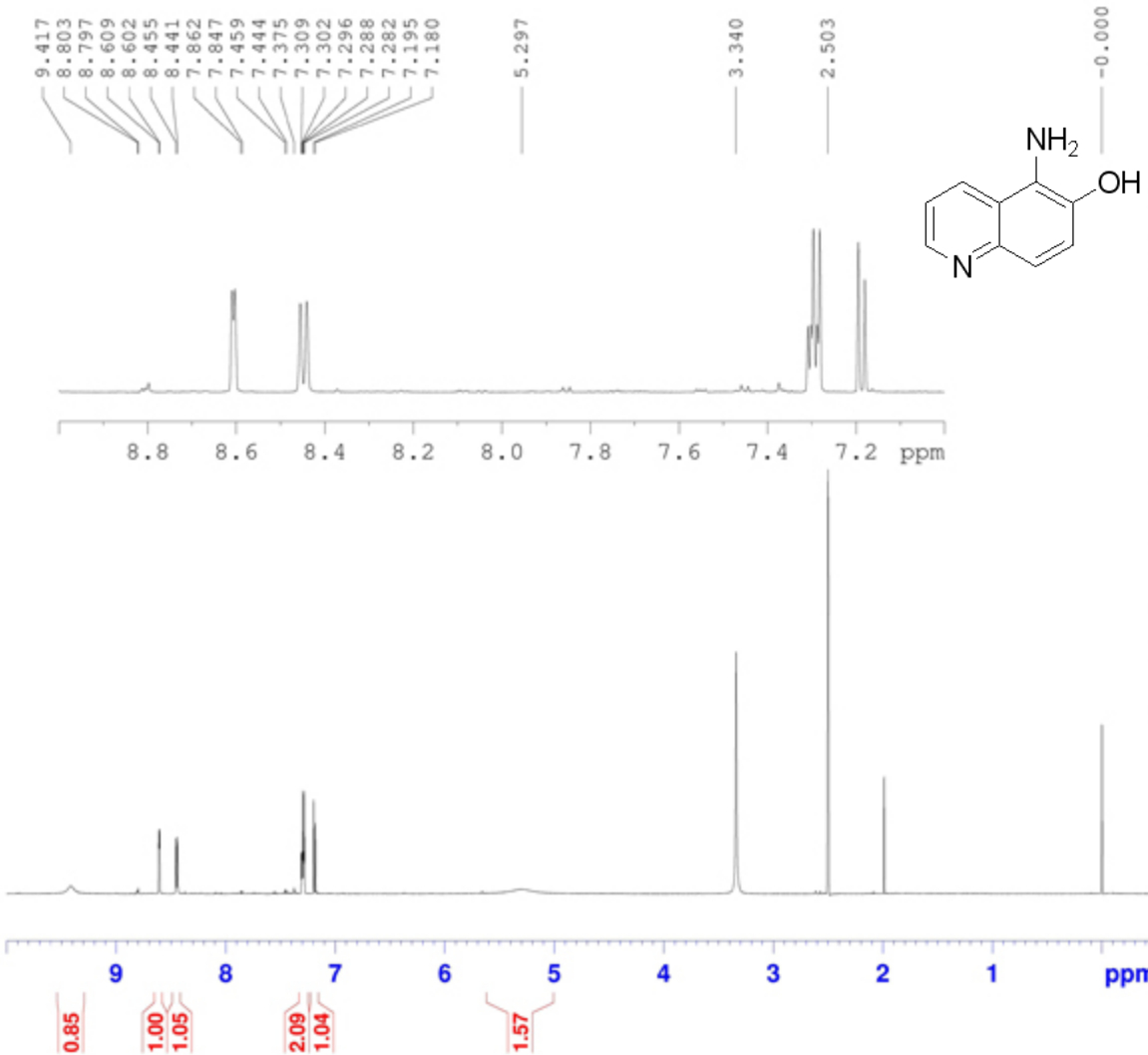
COSY


NH2
HO
OH

BRUKER


ppm
6.7
6.8
6.9
7.0
7.1
7.2
7.3
7.4
7.5
7.6
7.7
7.8
7.9
8.0
8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7
ppm


EFEA
%Transmittance
Wavenumbers (cm-1)
3500
3000
2500
2000
1500
1000
3384.09
3322.63
3273.61
3010.16
2605.35
1635.87
1530.26
1488.70
1414.59
1330.45
1207.08
1181.52
1147.57
887.54
823.64
749.43
619.21
NH2
HO
OH



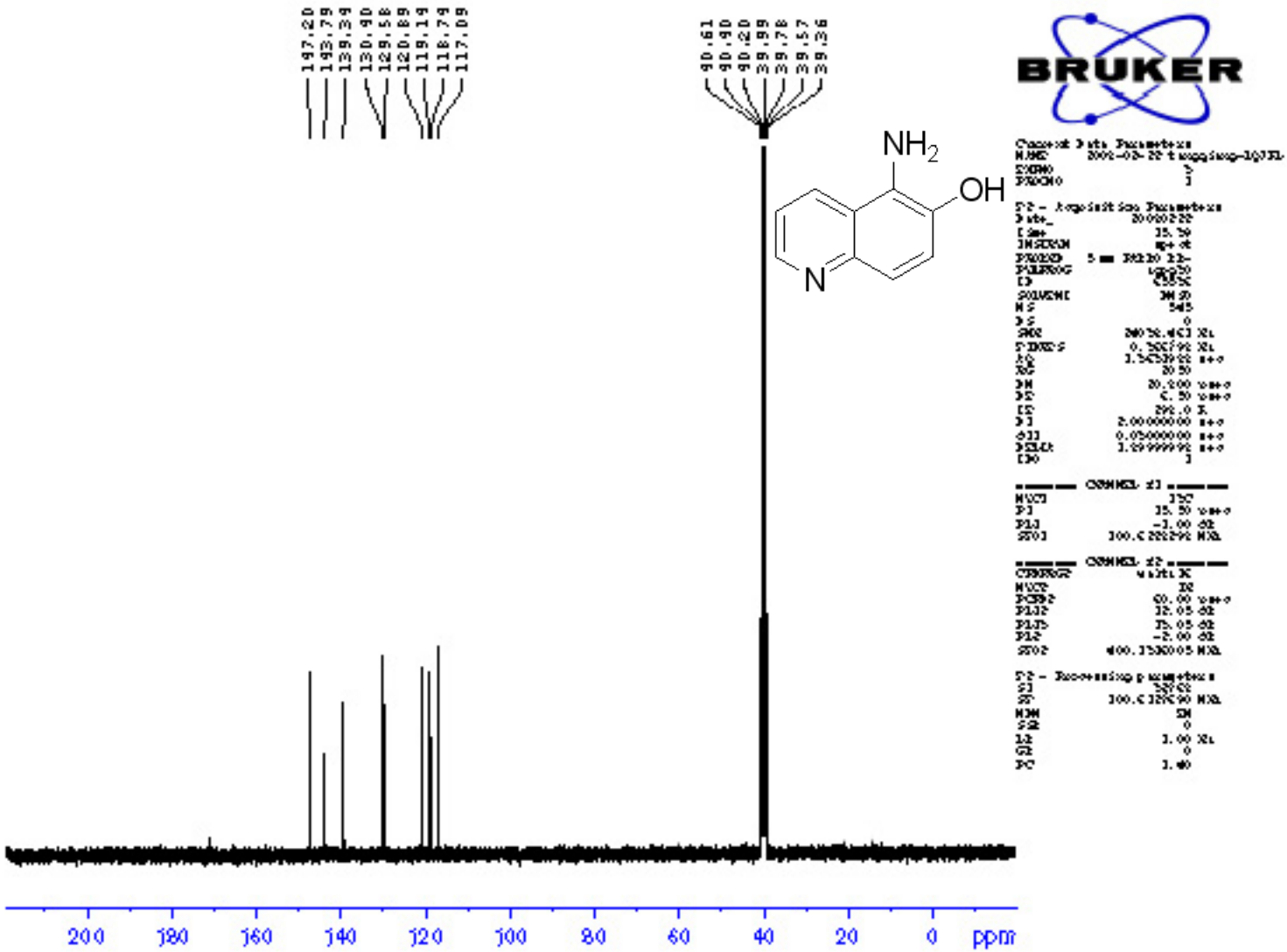
BRUKER

```
Current Data Parameters
NAME      2008-02-29 tangqiang-LQJKL
EXPNO                 1
PROCNO                1

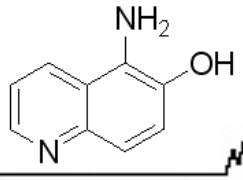
F2 - Acquisition Parameters
Date_           20080229
Time                8.49
INSTRUM            spect
PROBHD   5 mm PATXI 1H/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     0
SWH            12335.526 Hz
FIDRES             0.188225 Hz
AQ             2.6564426 sec
RG                   161
DW                40.533 usec
DE                 6.50 usec
TE                293.0 K
D1             1.00000000 sec
TD0                   1

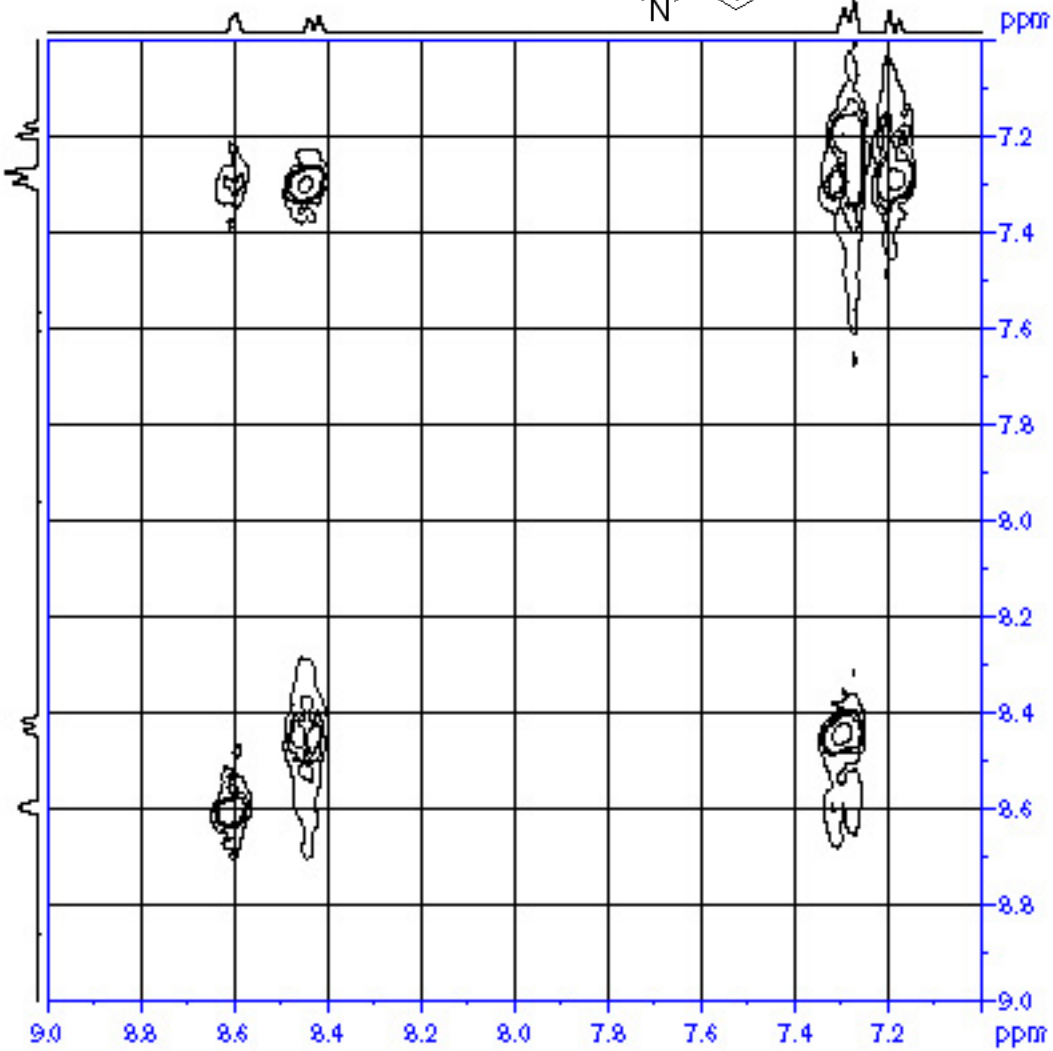
======== CHANNEL f1 ========
NUC1                  1H
P1                 7.20 usec
PL1               -1.00 dB
SFO1          600.1337060 MHz

F2 - Processing parameters
SI                32768
SF            600.1299982 MHz
WDW                  GM
SSB                   0
LB                -0.60 Hz
GB                  0.1
PC                 1.00
```

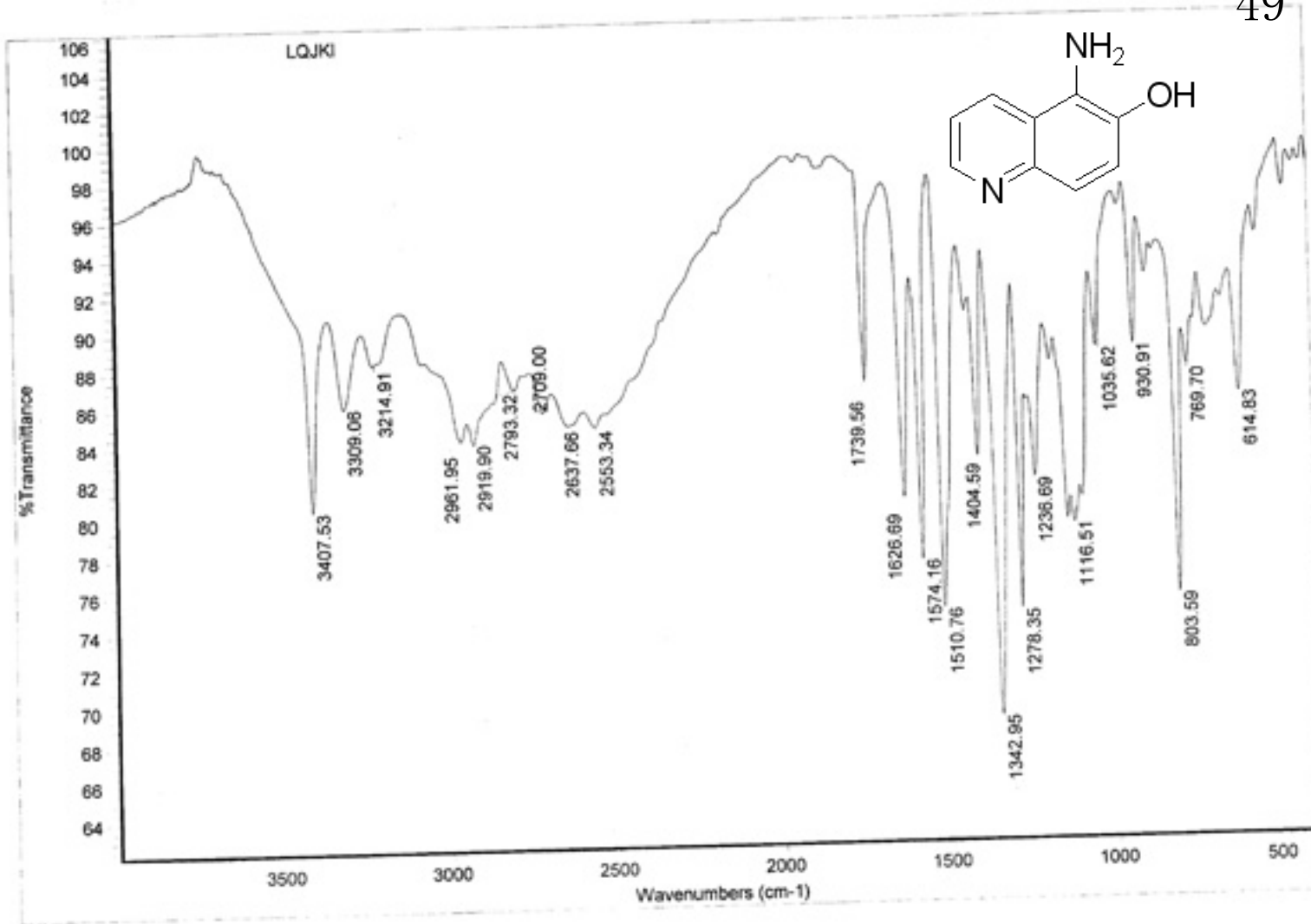

197.20
193.79
139.39
130.90
129.58
120.89
119.19
118.79
117.09
90.61
90.90
90.20
39.99
39.78
39.57
39.36
NH2
OH
N
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER

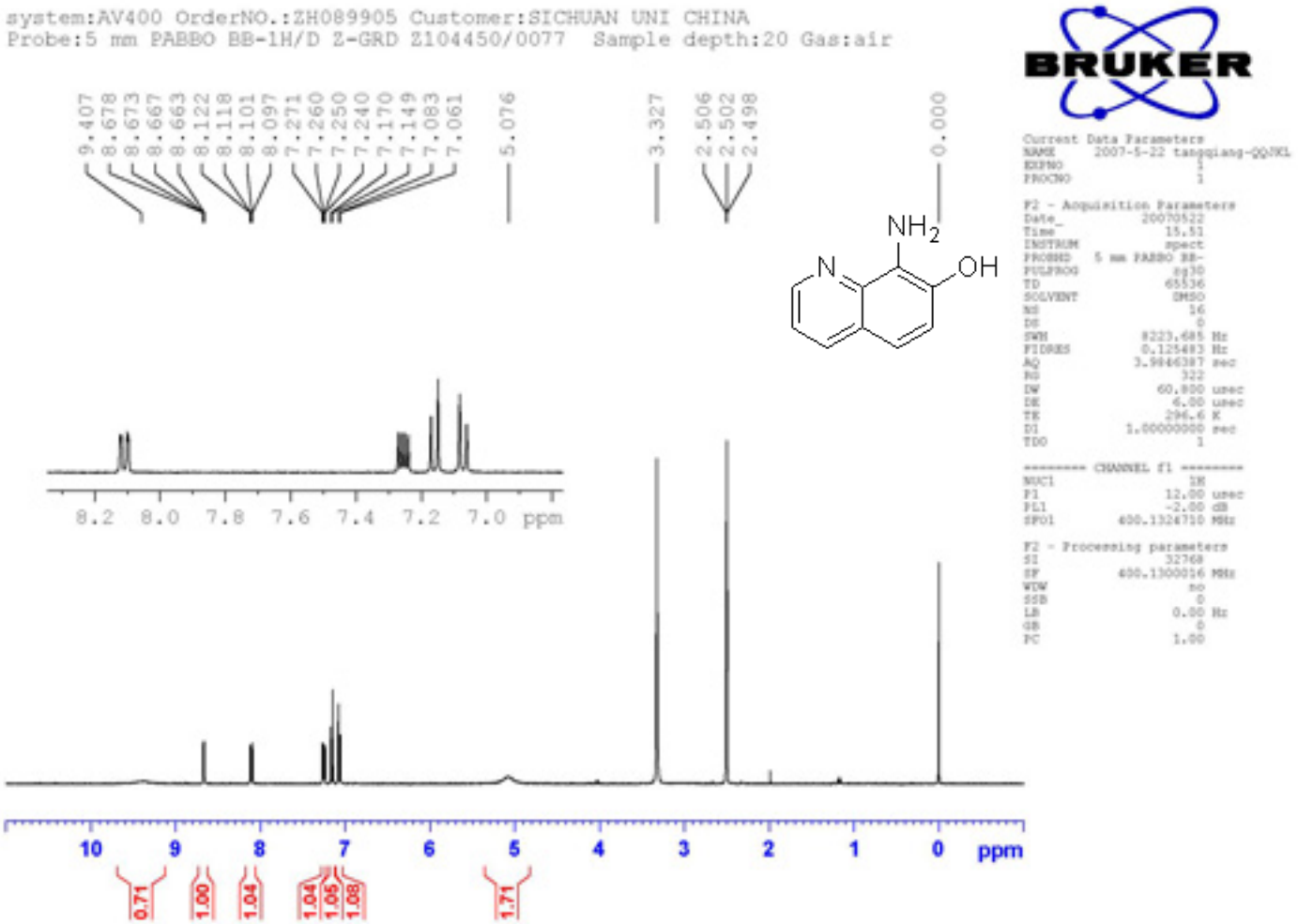
COSY

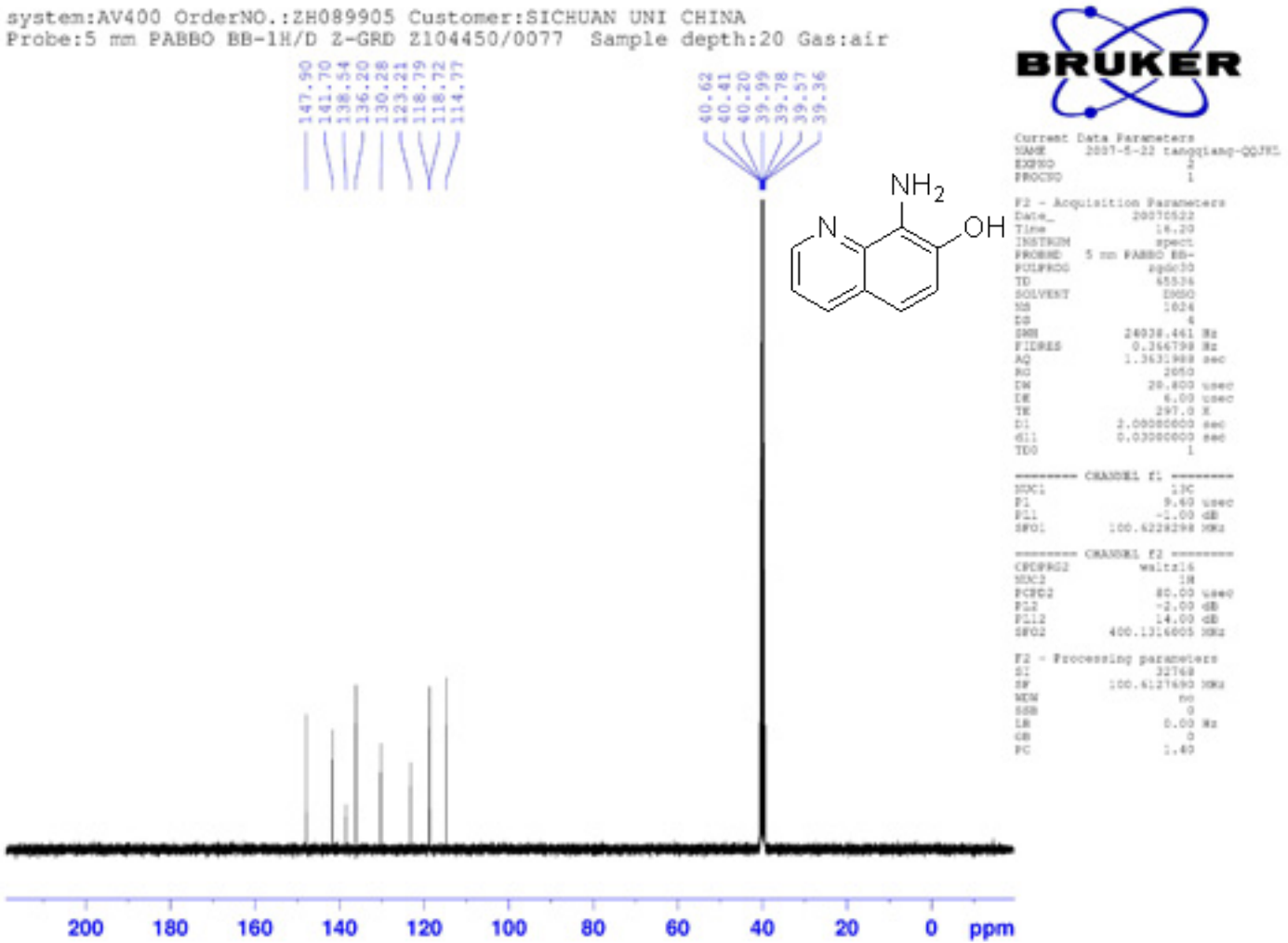

NH2
OH
N

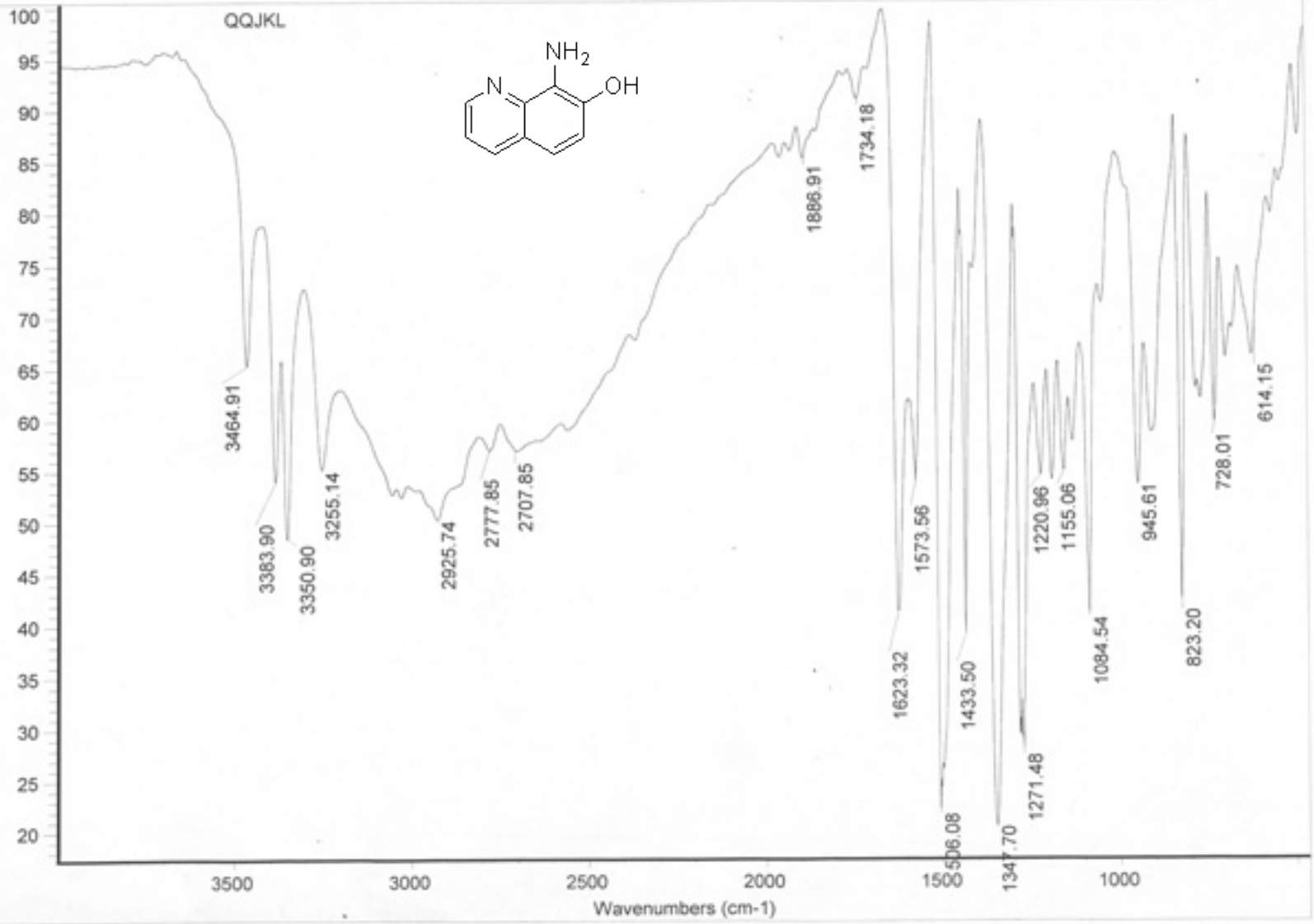

ppm
7.2
7.4
7.6
7.8
8.0
8.2
8.4
8.6
8.8
9.0
9.0
8.8
8.6
8.4
8.2
8.0
7.8
7.6
7.4
7.2
ppm


BRUKER

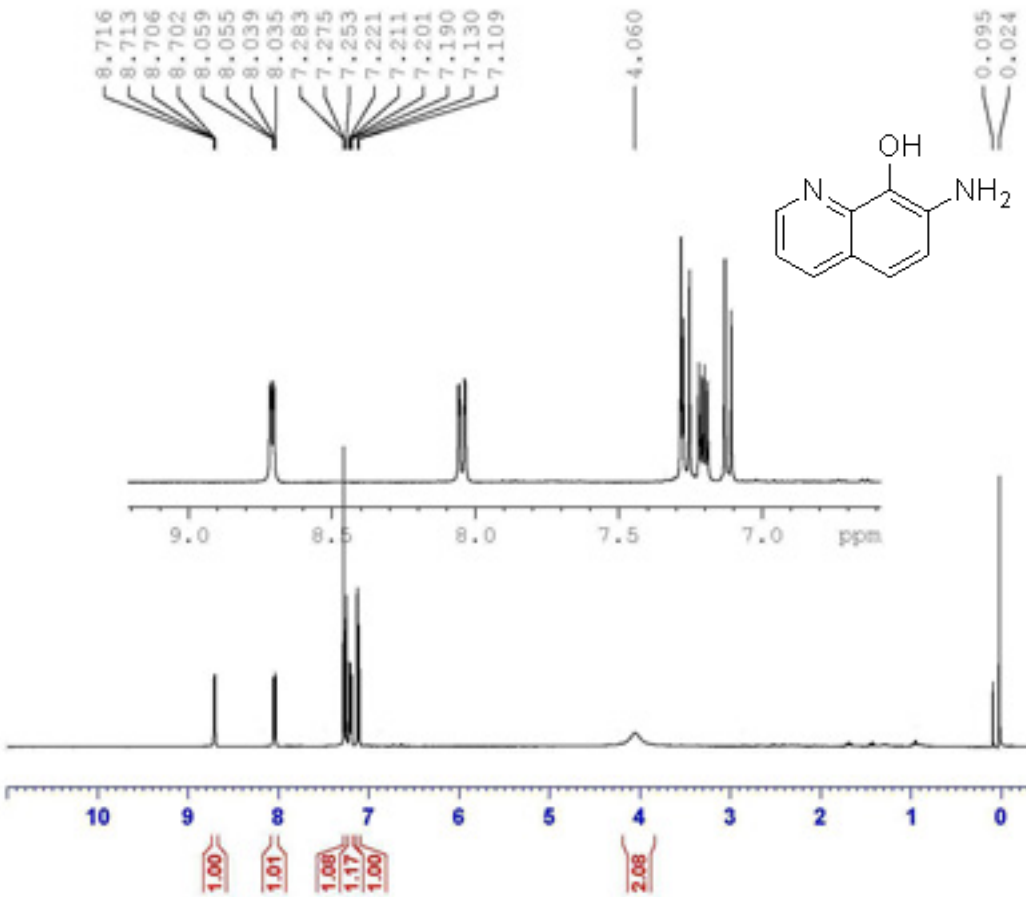

LQJKI
%Transmittance
Wavenumbers (cm-1)
3407.53
3309.06
3214.91
2961.95
2919.90
2793.32
2709.00
2637.66
2553.34
1739.56
1626.69
1574.16
1510.76
1404.59
1342.95
1278.35
1236.69
1116.51
1035.62
930.91
803.59
769.70
614.83
NH2
OH
N


system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
9.407
8.678
8.673
8.667
8.663
8.122
8.118
8.101
8.097
7.271
7.260
7.250
7.240
7.170
7.149
7.083
7.061
5.076
3.327
2.506
2.502
2.498
0.000
NH2
N
OH
8.2 8.0 7.8 7.6 7.4 7.2 7.0 ppm
10 9 8 7 6 5 4 3 2 1 0 ppm
0.71
1.00
1.04
1.04
1.05
1.08
1.71
BRUKER
Current Data Parameters
NAME 2007-5-22 tangqiang-QQ7K1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070522
Time 15.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 322
DW 60.800 usec
DE 6.00 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 600.1324710 MHz
F2 - Processing parameters
SI 32768
SF 600.1300016 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00


system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
147.90
141.70
138.54
136.20
130.28
123.21
118.79
118.72
114.77
40.62
40.41
40.20
39.99
39.78
39.57
39.36
NH2
N
OH
BRUKER
Current Data Parameters
NAME 2007-5-22 tangqiang-QQJYL
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070522
Time 14.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366799 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


QQJKL
NH2
N
OH
3464.91
3383.90
3350.90
3255.14
2925.74
2777.85
2707.85
1886.91
1734.18
1623.32
1573.56
1506.08
1433.50
1347.70
1271.48
1220.96
1155.06
1084.54
945.61
823.20
728.01
614.15
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
3500
3000
2500
2000
1500
1000
Wavenumbers (cm-1)

system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air



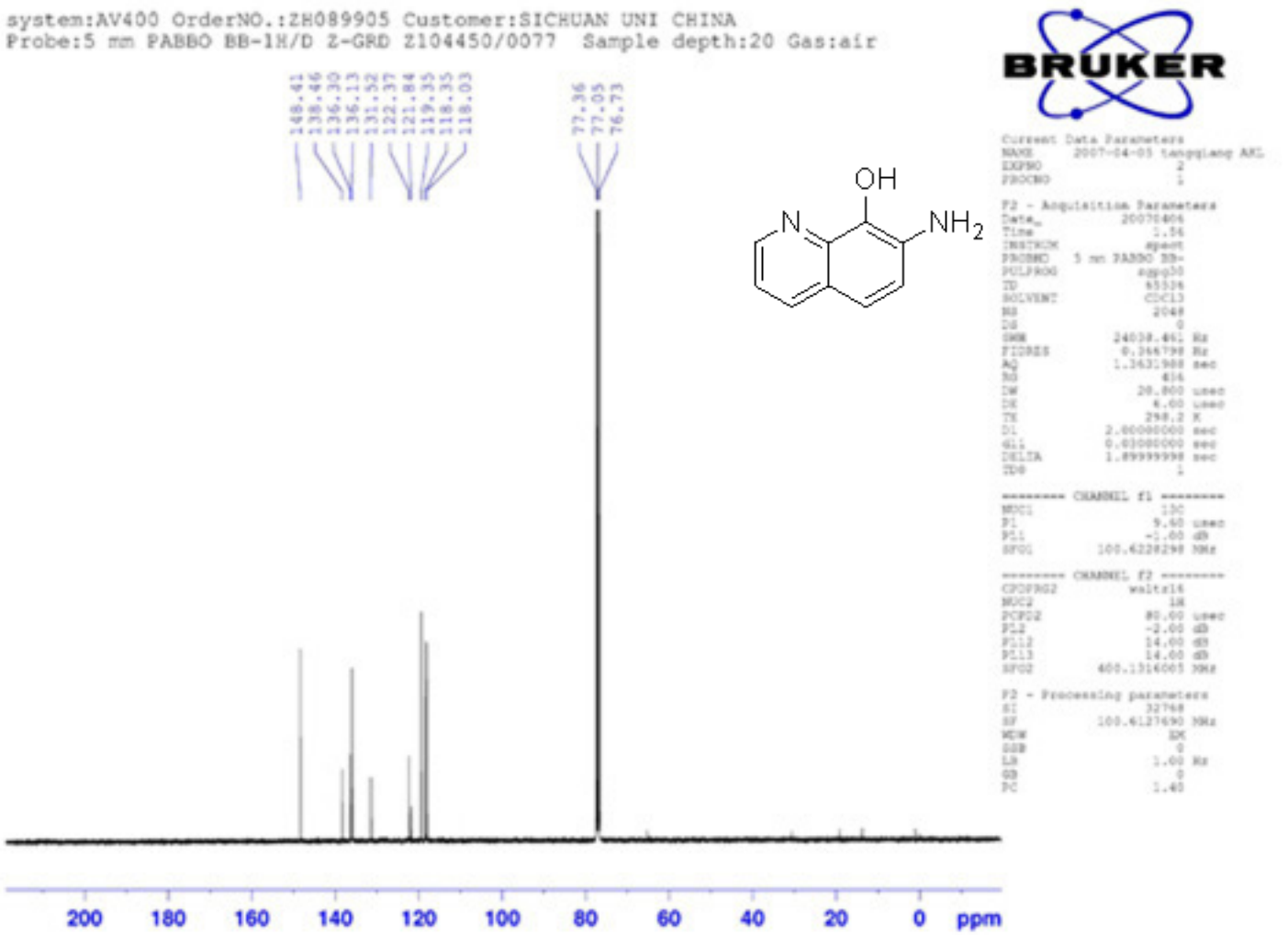
BRUKER

Current Data Parameters
NAME 2007-04-05 tangqiang AEL
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070405
Time 23.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846307 sec
RG 181
DW 60.800 usec
DE 6.00 usec
TE 296.4 K
D1 2.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

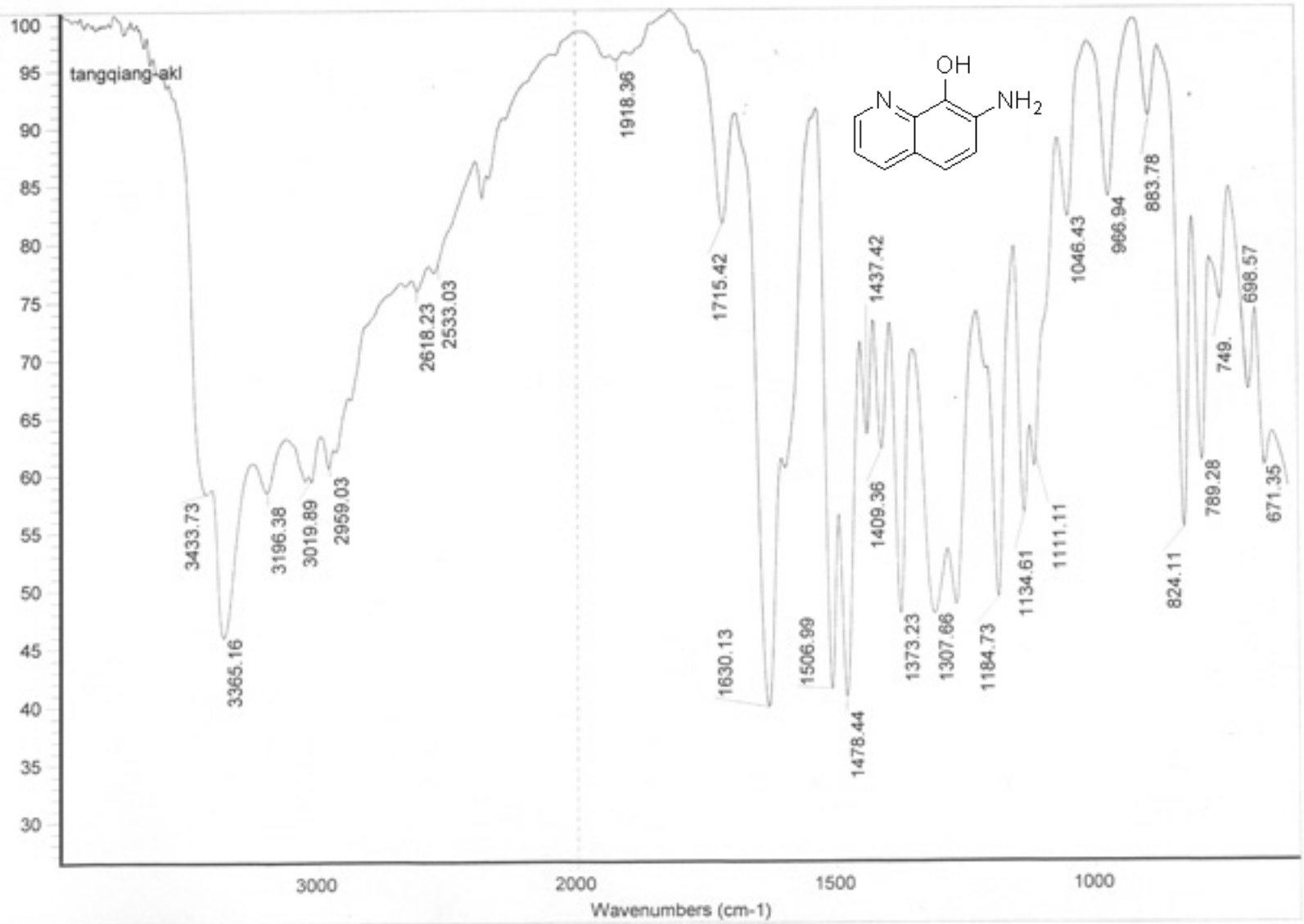
Current Data Parameters
NAME 2007-04-05 tangqiang AXL
EXPNO 2
PROCNO 1

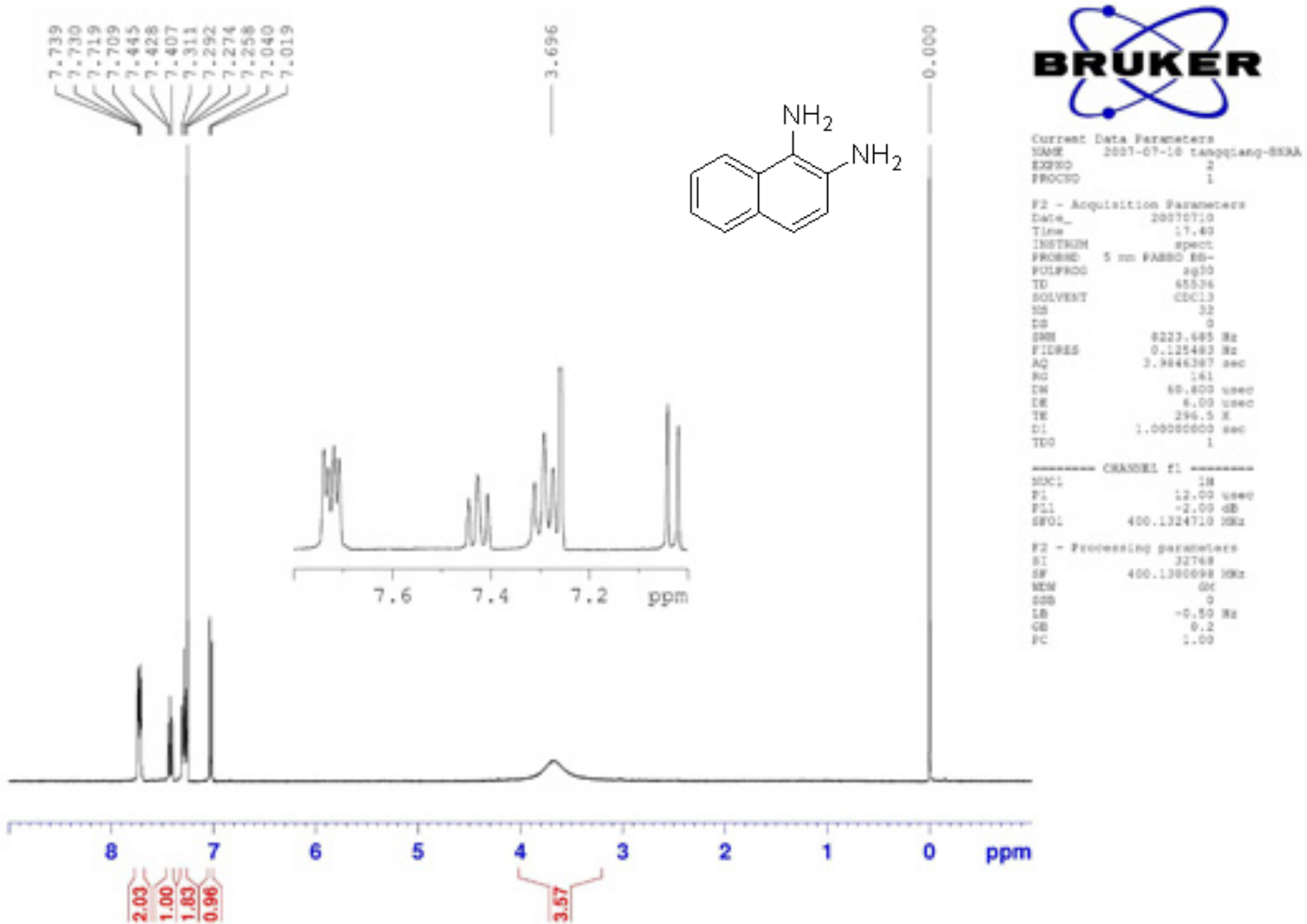
F2 - Acquisition Parameters
Date_ 20070406
Time 1.56
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 456
DW 20.800 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
GL1 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.00 dB
PL13 14.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


tangqiang-akl
100
95
90
85
80
75
70
65
60
55
50
45
40
35
30
3433.73
3365.16
3196.38
3019.89
2959.03
2618.23
2533.03
1918.36
1715.42
1630.13
1506.99
1478.44
1437.42
1409.36
1373.23
1307.66
1184.73
1134.61
1111.11
1046.43
966.94
883.78
824.11
789.28
749.
698.57
671.35
OH
N
NH2
3000
2000
1500
1000
Wavenumbers (cm-1)



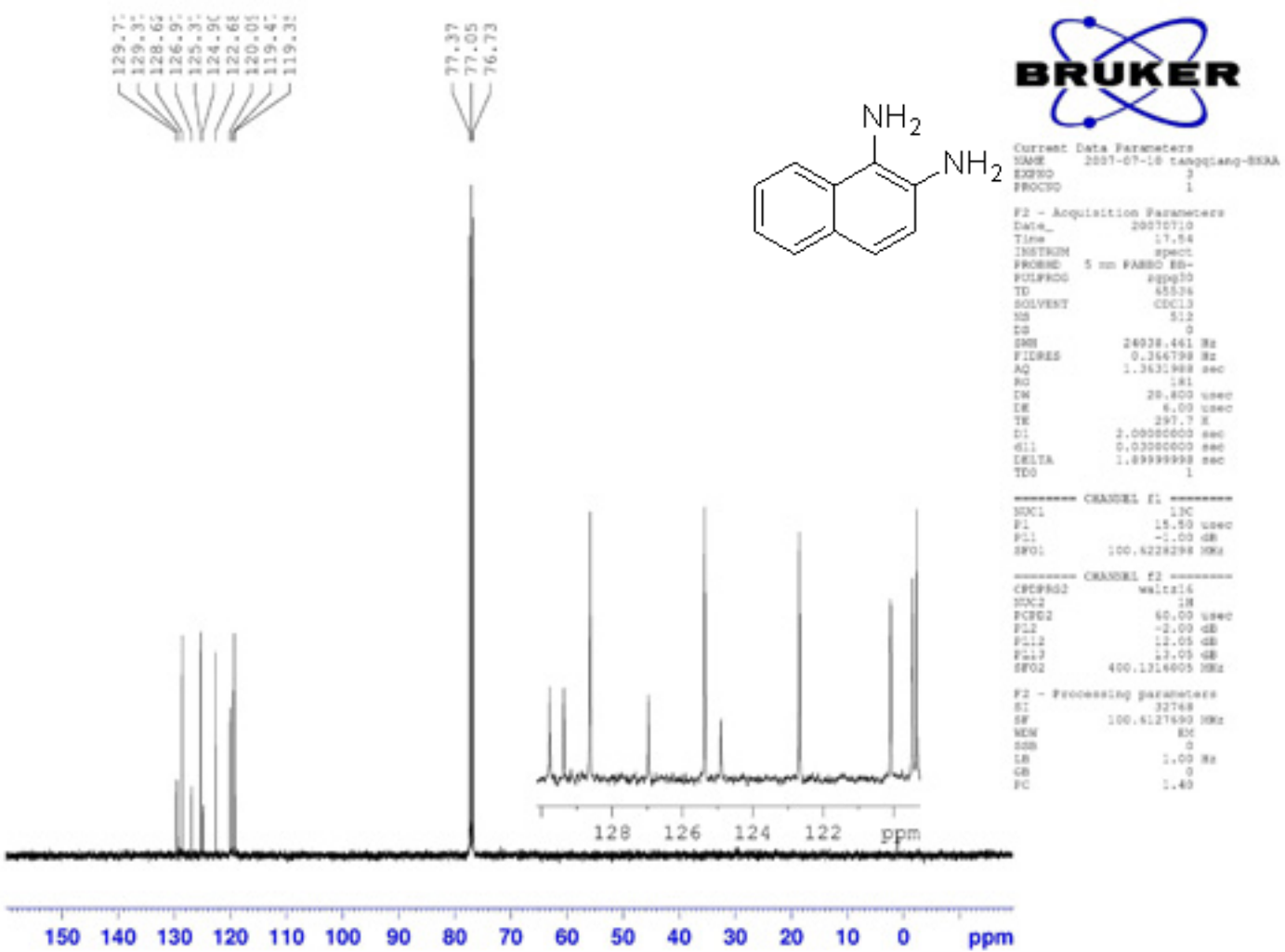
BRUKER

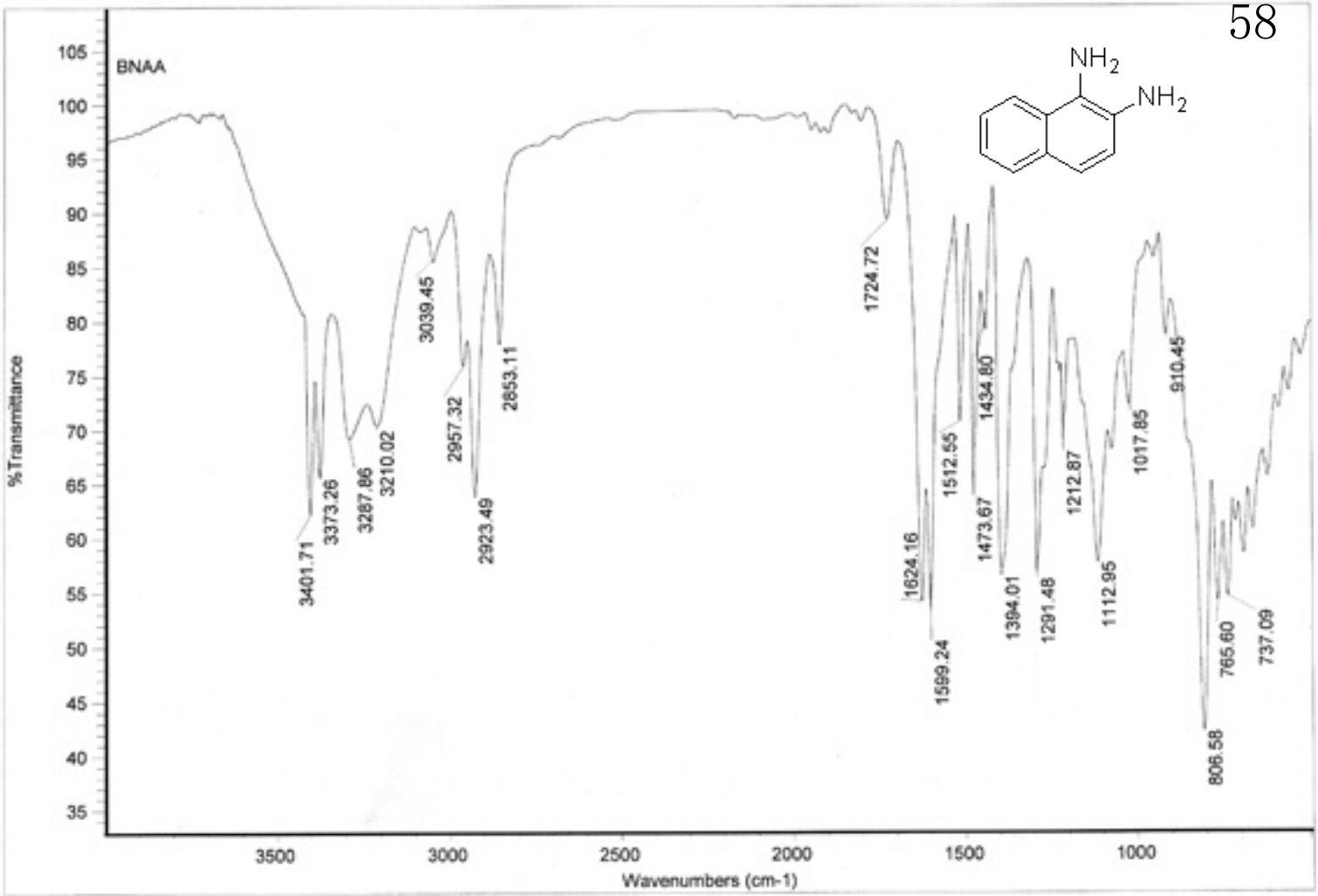
```
Current Data Parameters
NAME      2007-07-10 tangqiang-BXAA
EXPNO                2
PROCNO               1

F2 - Acquisition Parameters
Date_           20070710
Time              17.40
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  32
DS                   0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                 161
DW               60.800 usec
DE                6.00 usec
TE              296.5 K
D1            1.00000000 sec
TD0                  1

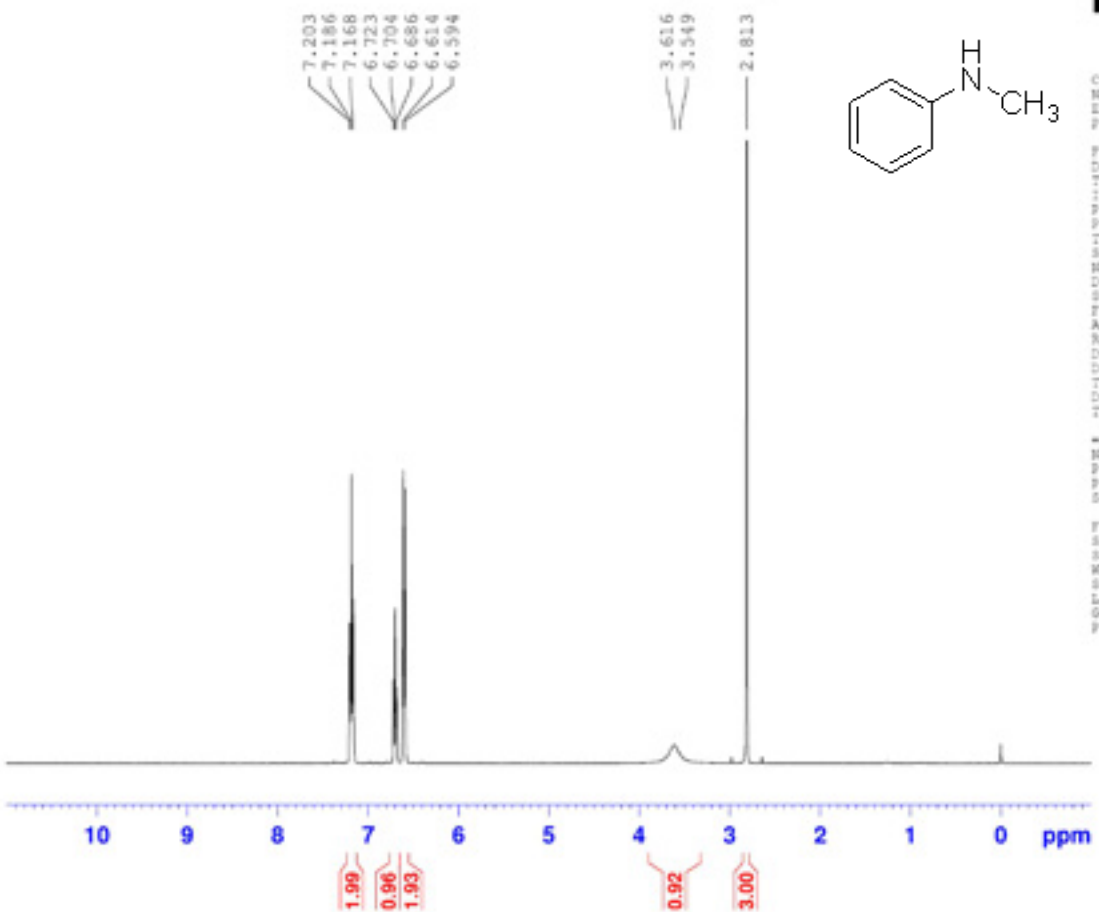
======== CHANNEL f1 ========
NUC1               1H
P1               12.00 usec
PL1              -2.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300099 MHz
WDW                EM
SSB                 0
LB              -0.50 Hz
GB                 0.2
PC                1.00
```


129.7
129.3
128.6
126.9
125.3
124.9
122.6
120.0
119.4
119.3
77.37
77.05
76.73
NH2
NH2
BRUKER
Current Data Parameters
NAME 2007-07-10 tangqiang-BXAA
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070710
Time 17.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 24038.461 Hz
FIDRES 0.366799 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.00 usec
TE 297.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 18.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 -2.00 dB
PL12 12.05 dB
PL13 23.05 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
128 126 124 122 ppm
150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


BNAA
NH2
NH2
%Transmittance
105
100
95
90
85
80
75
70
65
60
55
50
45
40
35
3500
3000
2500
2000
1500
1000
Wavenumbers (cm-1)
3401.71
3373.26
3287.86
3210.02
3039.45
2957.32
2923.49
2853.11
1724.72
1624.16
1599.24
1512.55
1473.67
1434.80
1394.01
1291.48
1212.87
1112.95
1017.85
910.45
806.58
765.60
737.09

system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air



BRUKER

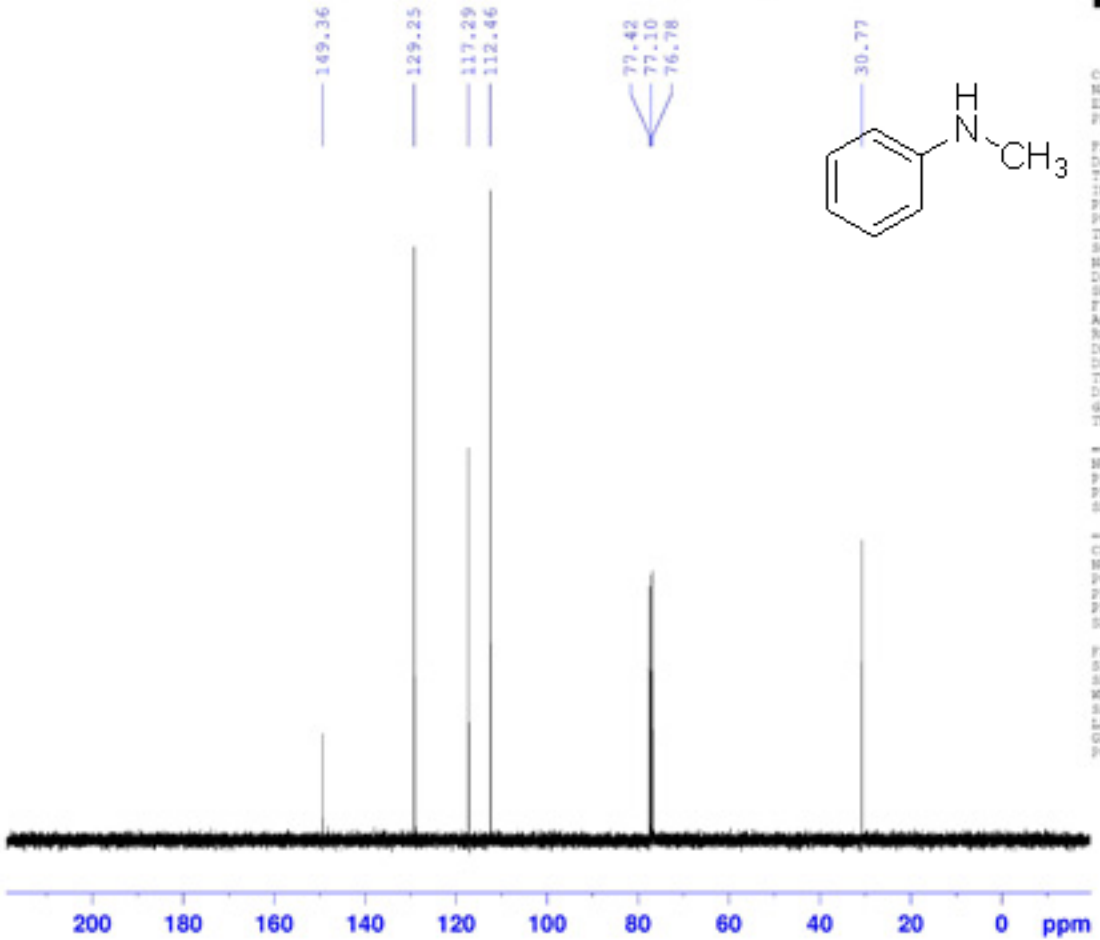
Current Data Parameters
NAME 2007-3-22 tangqiang-HJBA
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070322
Time 14.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 114
DW 62.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 32768
SF 400.1300277 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air



BRUKER

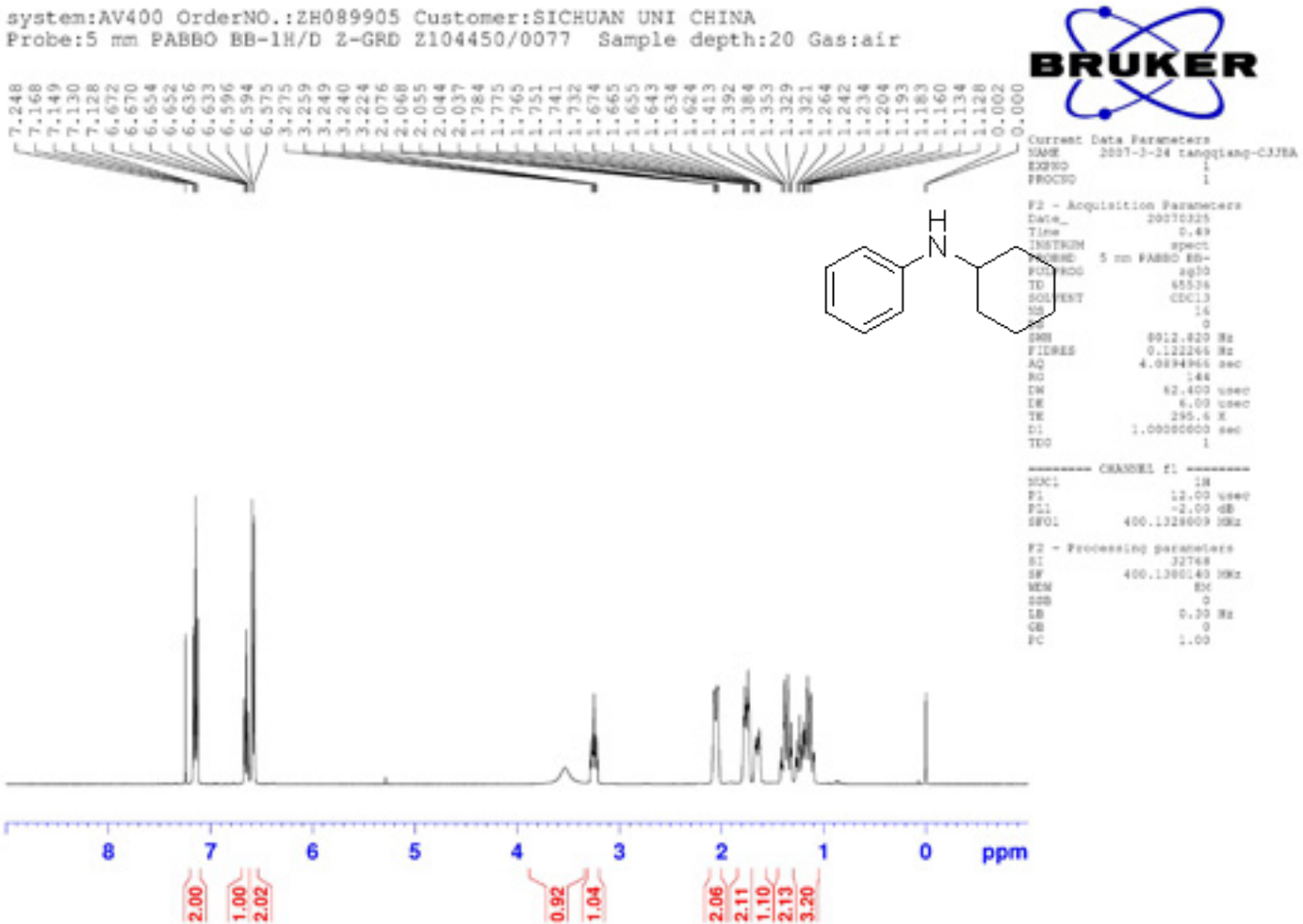
Current Data Parameters
NAME 2007-3-22 tangqiang-NJBA
EXPNO 2
PROCNO 1

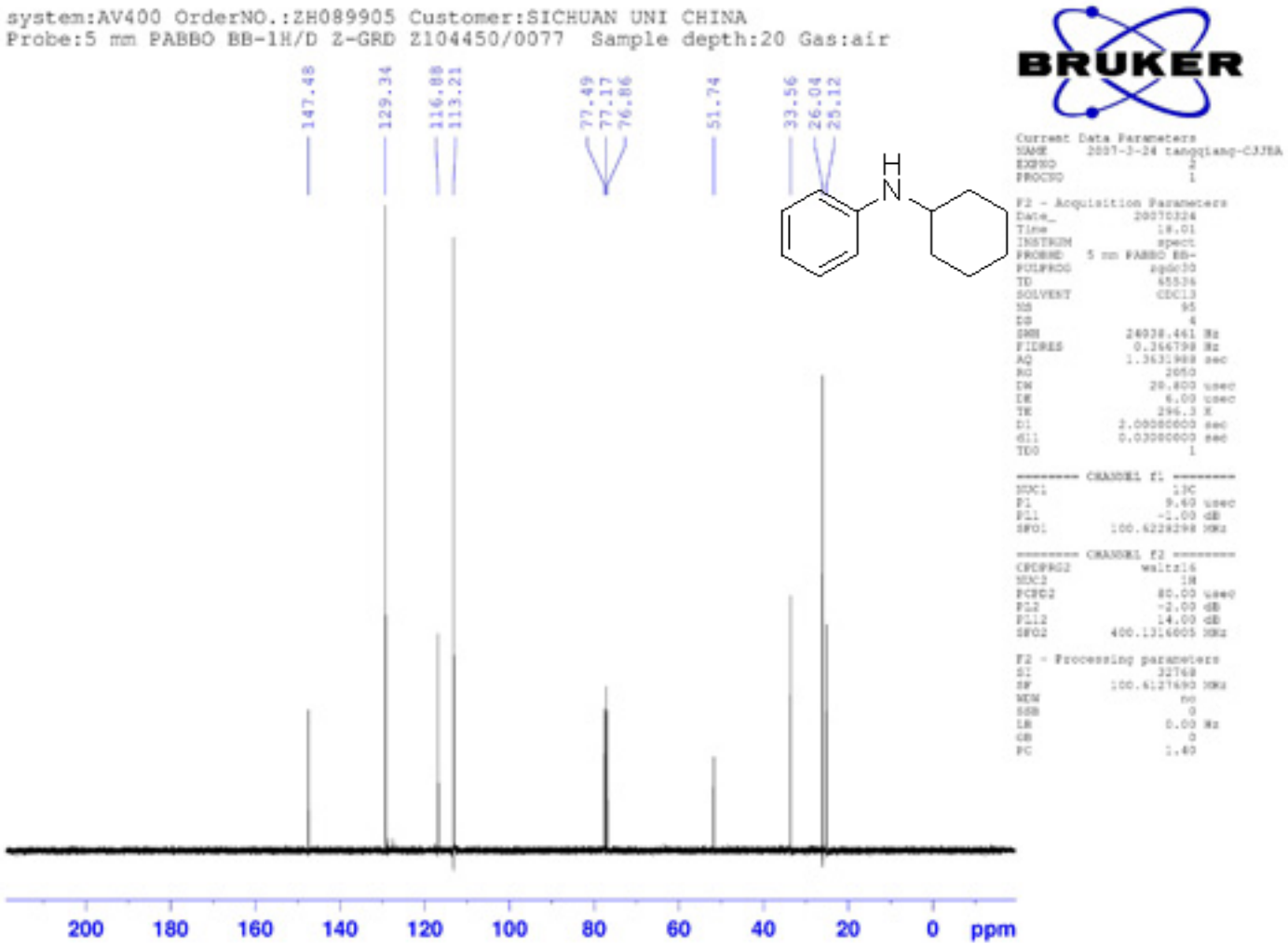
F2 - Acquisition Parameters
Date_ 20070322
Time 14.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 101
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.8 K
D1 2.00000000 sec
GIL 0.03000000 sec
TD0 1

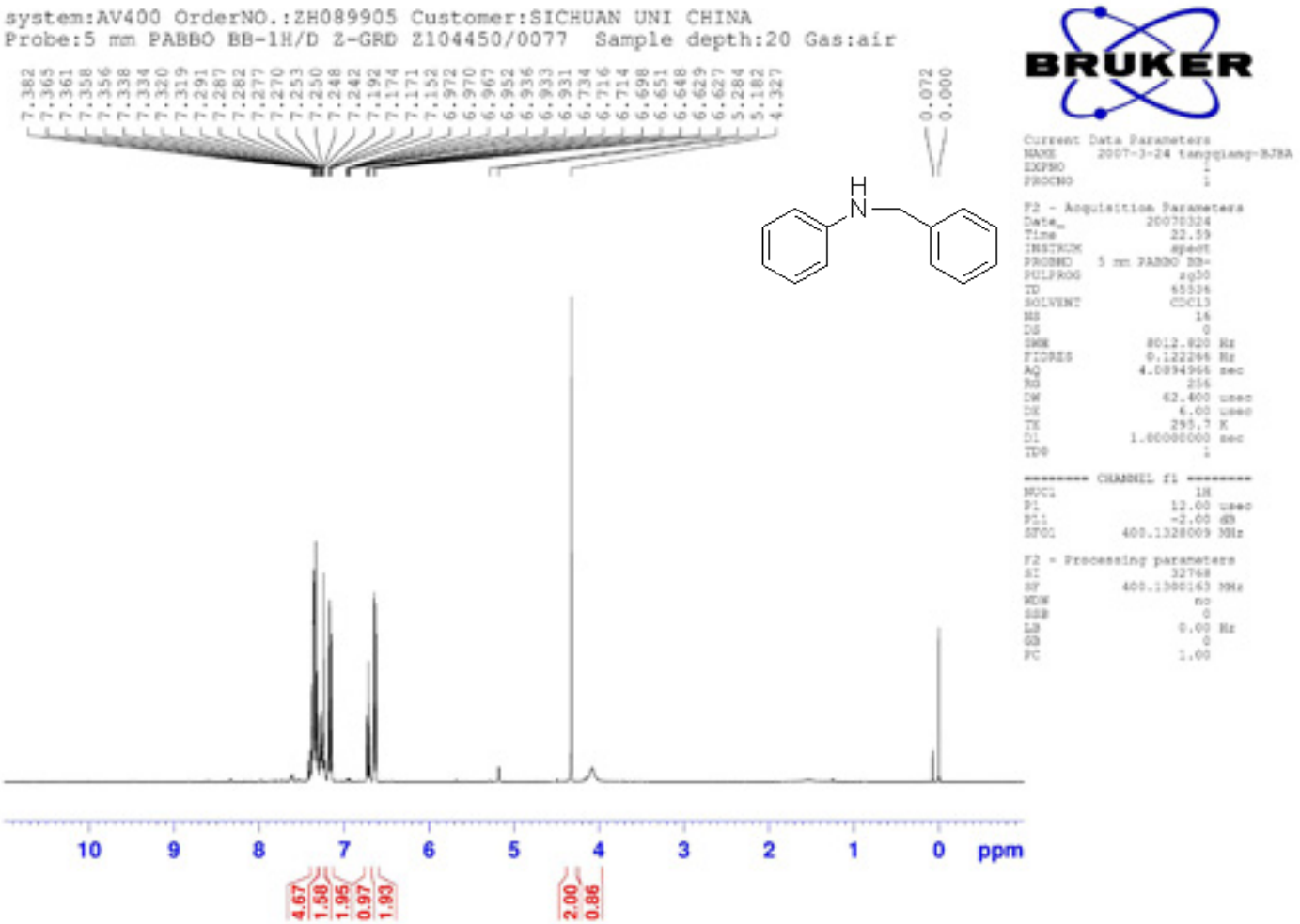
======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40


system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
BRUKER
7.248 7.168 7.149 7.130 7.128 6.672 6.670 6.654 6.652 6.636 6.633 6.596 6.594 6.575 3.275 3.259 3.249 3.240 3.224 3.076 2.068 2.055 2.044 2.037 1.784 1.775 1.765 1.751 1.741 1.732 1.674 1.665 1.655 1.643 1.638 1.624 1.413 1.392 1.384 1.353 1.329 1.321 1.264 1.242 1.234 1.204 1.193 1.183 1.160 1.134 1.128 0.002 0.000
H
N
Current Data Parameters
NAME 2007-3-24 tangqiang-CJJBA
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070325
Time 0.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894964 sec
RG 144
DW 62.400 usec
DE 6.00 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 32768
SW 400.1300140 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8 7 6 5 4 3 2 1 0 ppm
2.00 1.00 2.02 0.92 1.04 2.06 2.11 1.10 2.13 3.20


system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
147.48
129.34
116.88
113.21
77.49
77.17
76.86
51.74
33.56
26.04
25.12
H
N
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME 2007-3-24 tangqiang-CJJBA
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070324
Time 18.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 95
DS 4
SWH 24038.461 Hz
FIDRES 0.366799 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 296.3 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40



BRUKER

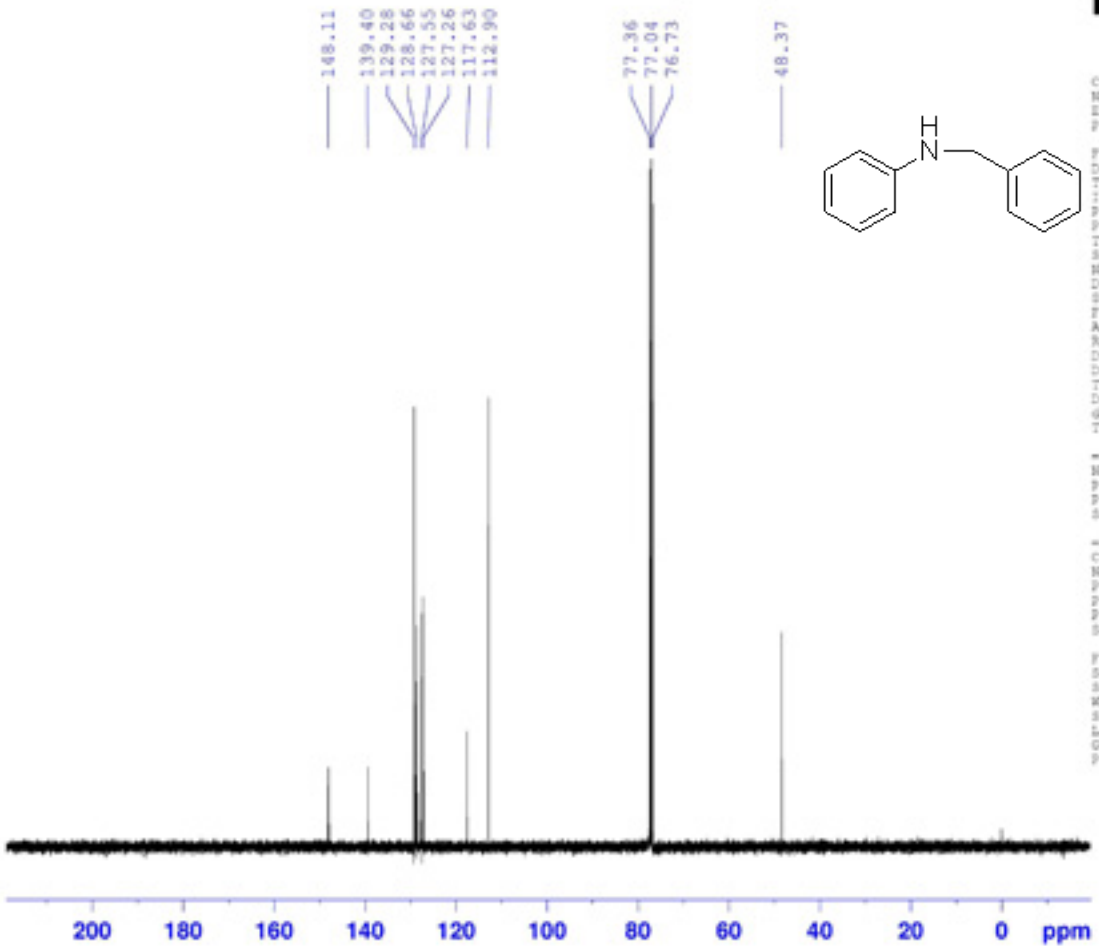
Current Data Parameters
NAME 2007-3-24 tangqiang-BJBA
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070324
Time 22.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 256
DW 62.400 usec
DE 6.00 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1328009 MHz

F2 - Processing parameters
SI 32768
SF 400.1300163 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air



BRUKER

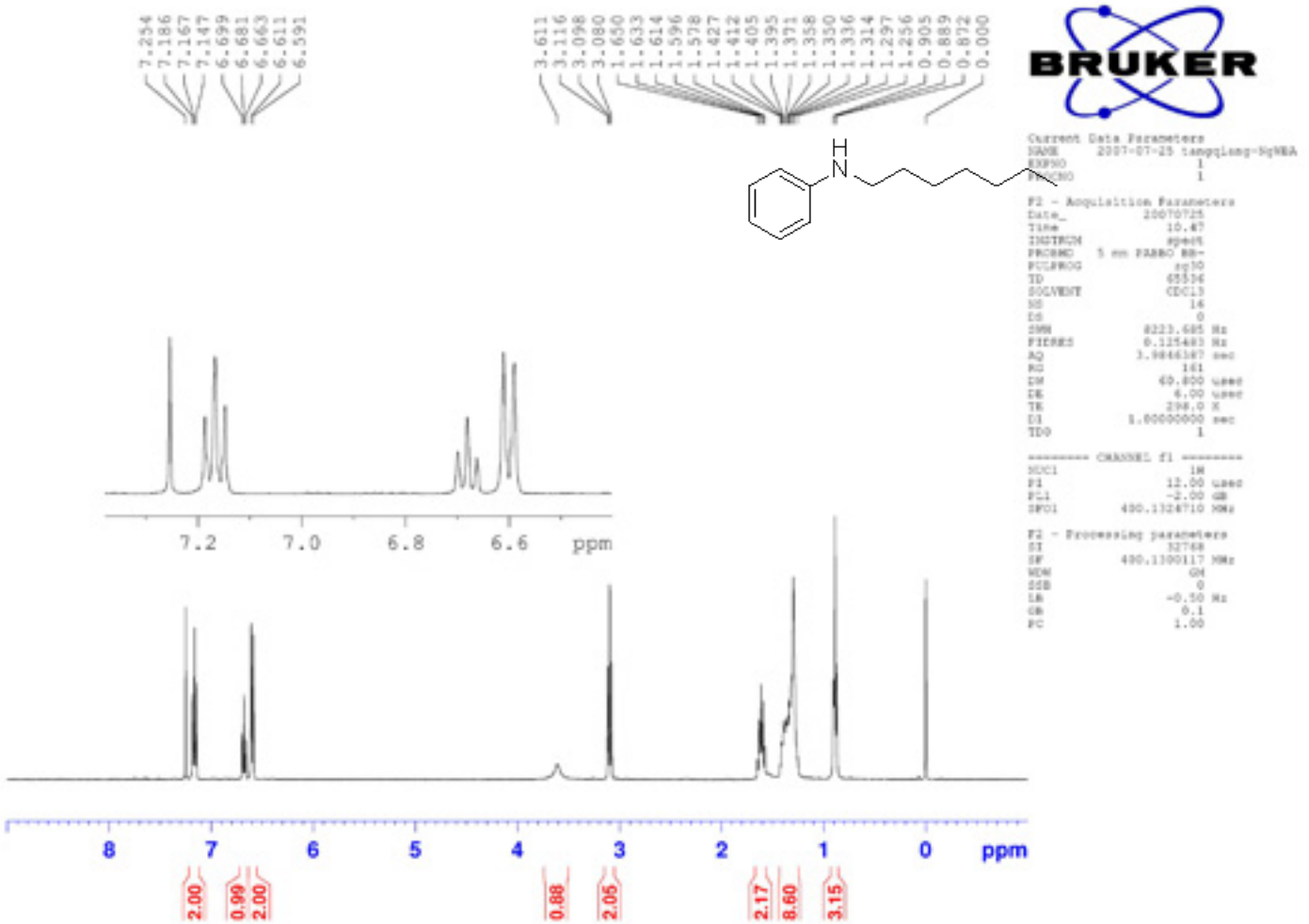
```
Current Data Parameters
NAME      2007-3-24 tangqiang-BJBA
EXPNO                 2
PROCNO                1

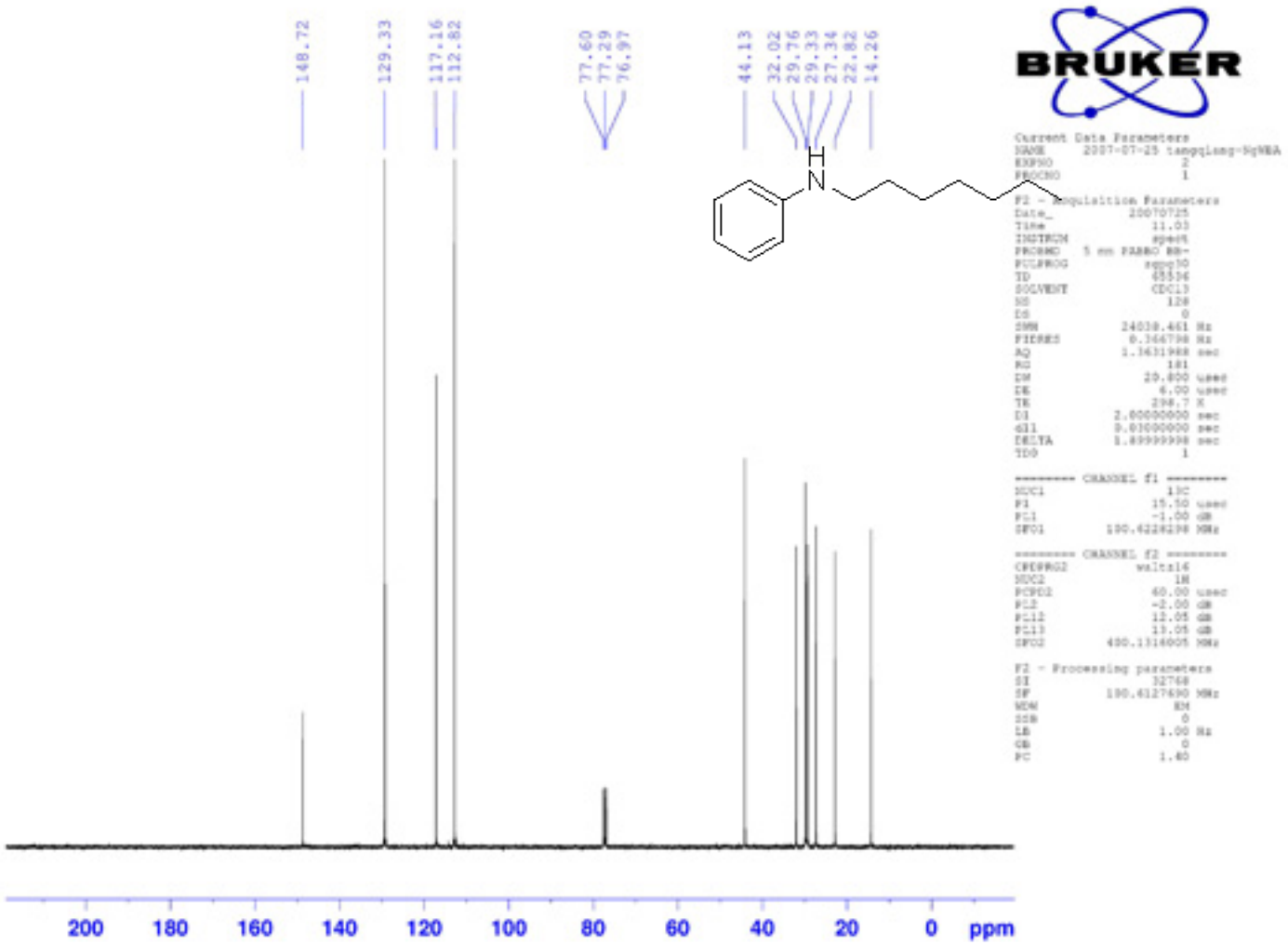
F2 - Acquisition Parameters
Date_          20070324
Time              16.16
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG          zgdc30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                 2050
DW               20.800 usec
DE                6.00 usec
TE               296.5 K
D1            2.00000000 sec
GL1           0.03000000 sec
TD0                   1

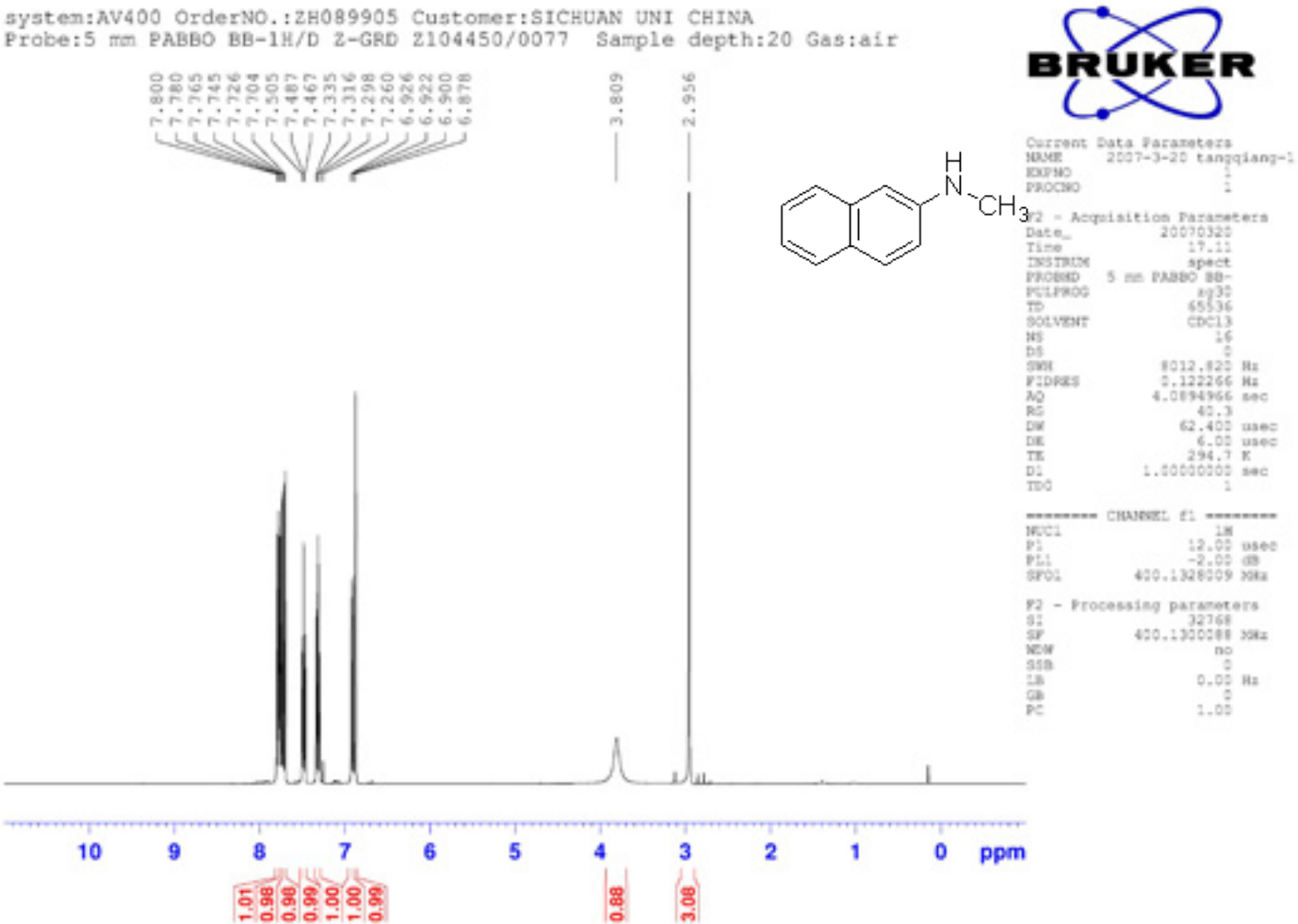
======== CHANNEL f1 ========
NUC1                13C
P1                 9.60 usec
PL1               -1.00 dB
SFO1        100.6228298 MHz

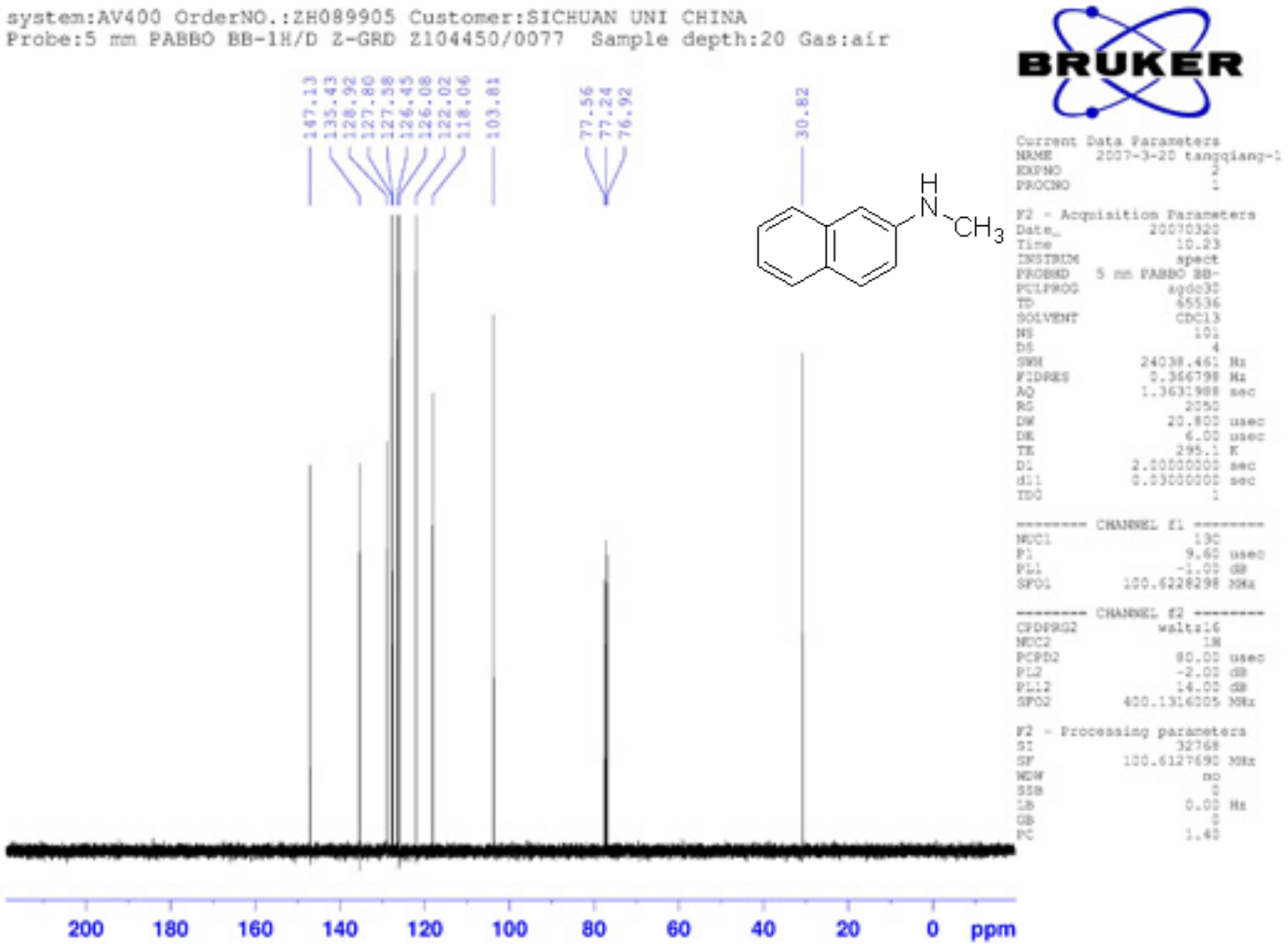
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              14.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
```

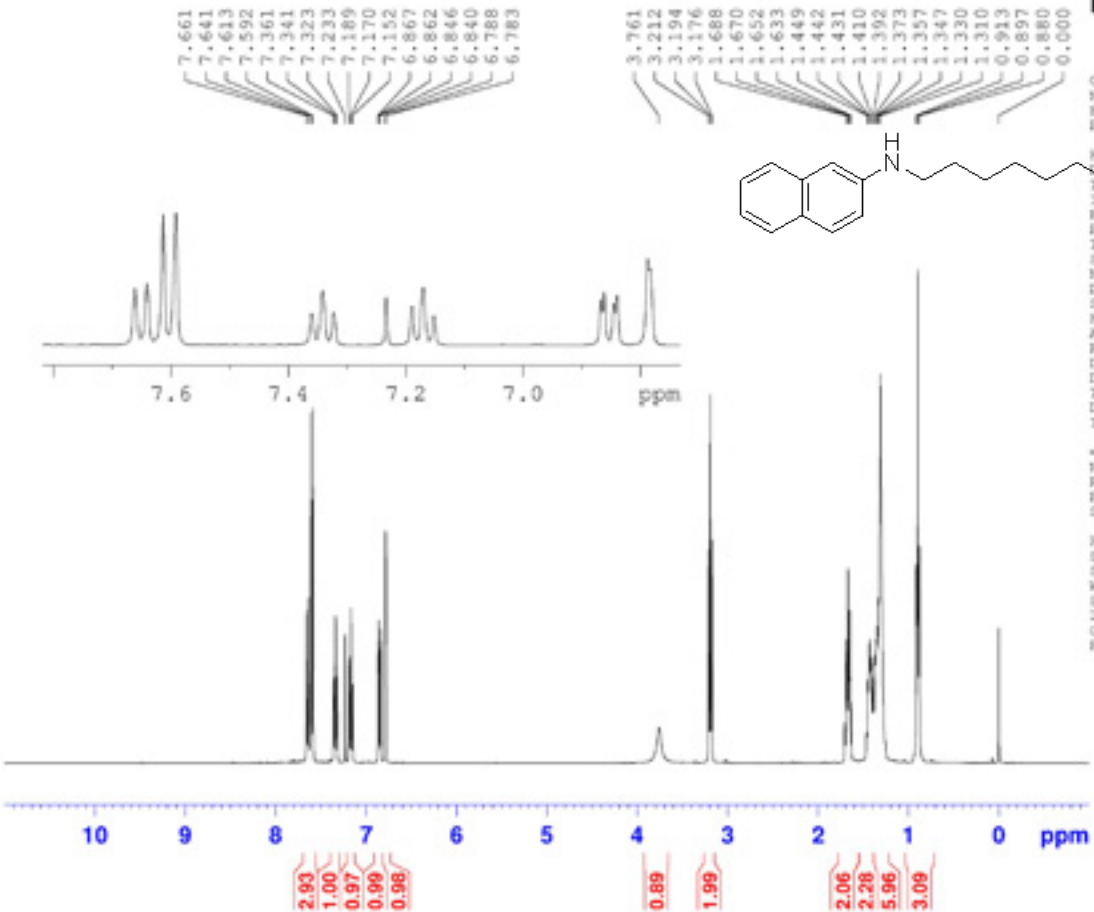

7.254
7.186
7.167
7.147
6.699
6.681
6.663
6.611
6.591
3.611
3.116
3.098
3.080
1.650
1.633
1.614
1.596
1.578
1.427
1.412
1.405
1.395
1.371
1.358
1.350
1.336
1.314
1.297
1.256
0.905
0.889
0.872
0.000
BRUKER
N
H
Current Data Parameters
NAME 20070725 tangqiang-HgNBA
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070725
Time 10.47
INSTRM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846587 sec
RG 161
DW 60.800 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300117 MHz
WDW EM
SSB 0
LB -0.50 Hz
GB 0.1
PC 1.00
7.2
7.0
6.8
6.6
ppm
8
7
6
5
4
3
2
1
0
ppm
2.00
0.99
2.00
0.88
2.05
2.17
8.60
3.15


148.72
129.33
117.16
112.82
77.60
77.29
76.97
44.13
32.02
29.76
29.33
27.34
22.82
14.26
H
N
BRUKER
Current Data Parameters
NAME 20070725 tangqiang-HgNBA
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070725
Time 11.03
INSTRM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.00 usec
TE 298.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 -2.00 dB
PL12 12.05 dB
PL13 13.05 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm


system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
BRUKER
7.800
7.780
7.765
7.745
7.726
7.704
7.505
7.487
7.467
7.335
7.316
7.298
7.260
6.926
6.922
6.900
6.878
3.809
2.956
H
N
CH3
Current Data Parameters
NAME 2007-3-20 tangqiang-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070320
Time 17.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 40.3
DW 62.400 usec
DE 6.00 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1328009 MHz
F2 - Processing parameters
SI 32768
SF 400.1300088 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.01
0.98
0.98
0.99
1.00
1.00
0.99
0.88
3.08


system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
147.13
135.43
128.92
127.80
127.58
126.45
126.08
122.02
118.06
103.81
77.56
77.24
76.92
30.82
H
N
CH3
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
Current Data Parameters
NAME 2007-3-20 tangqiang-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070320
Time 10.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 101
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.1 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 9.60 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air



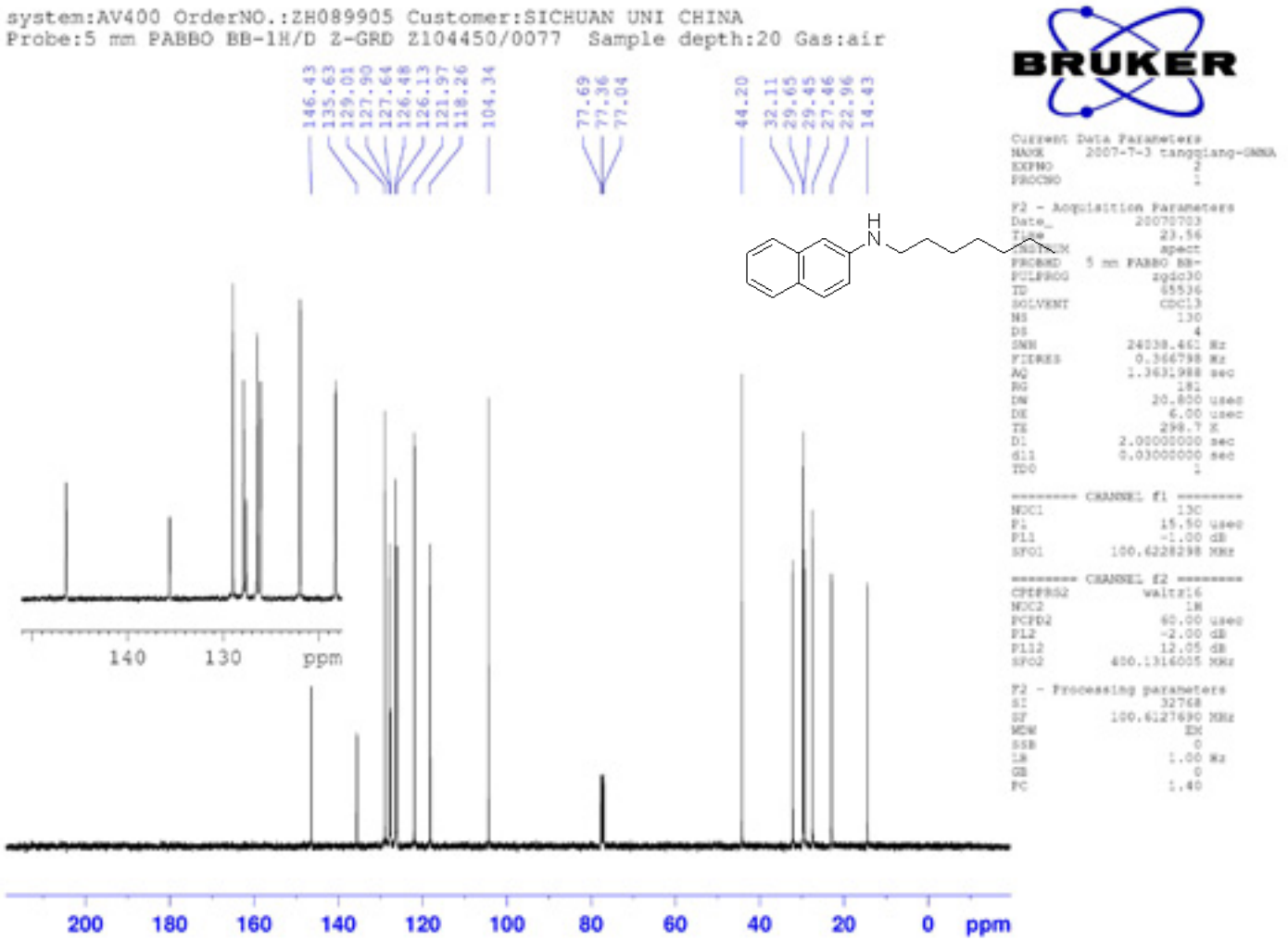
BRUKER

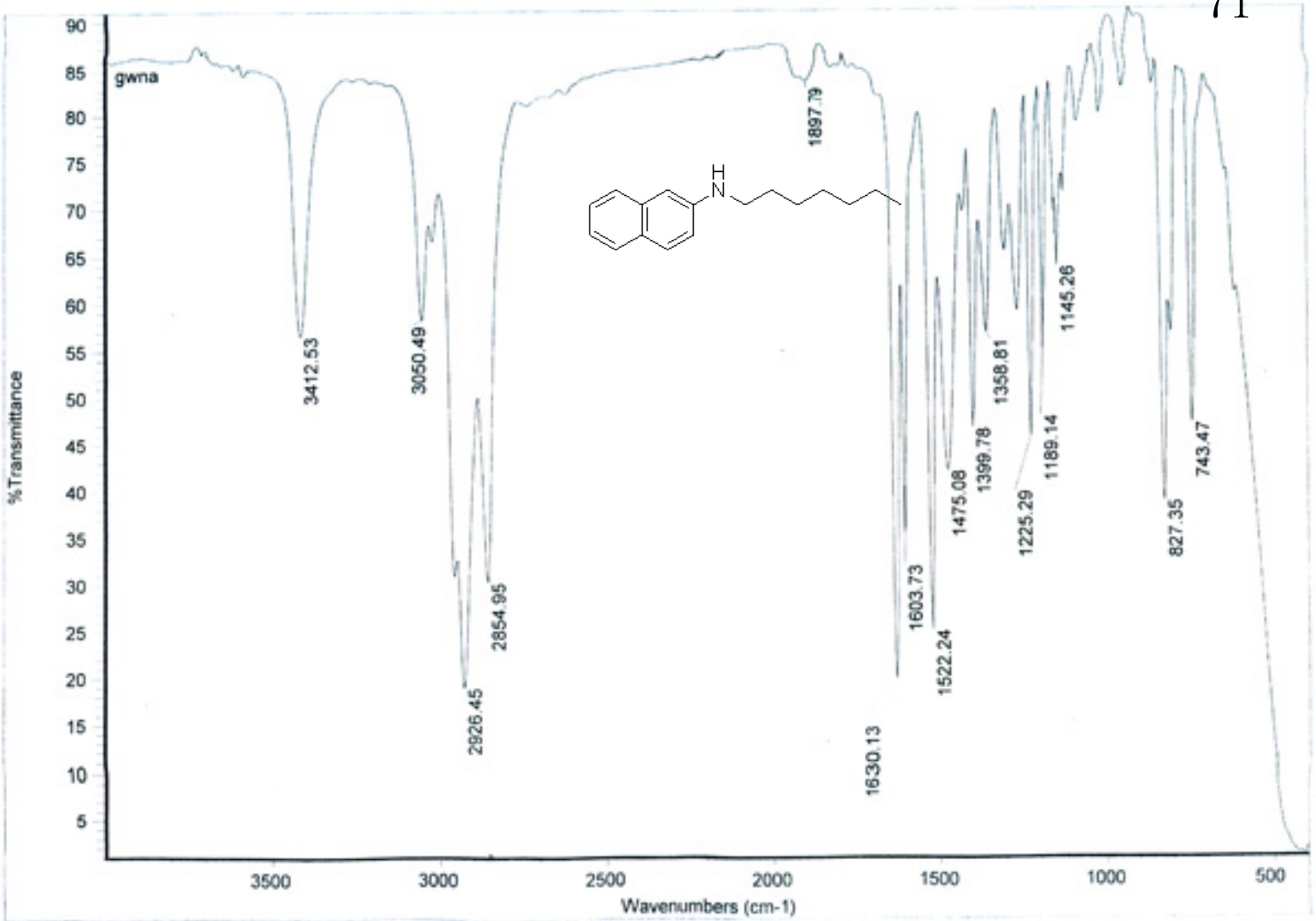
```
Current Data Parameters
NAME      2007-7-3 tangqiang-GNNA
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_           20070703
Time              23.32
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                 144
DW              60.800 usec
DE               6.00 usec
TE              296.9 K
D1          1.00000000 sec
TD0                  1

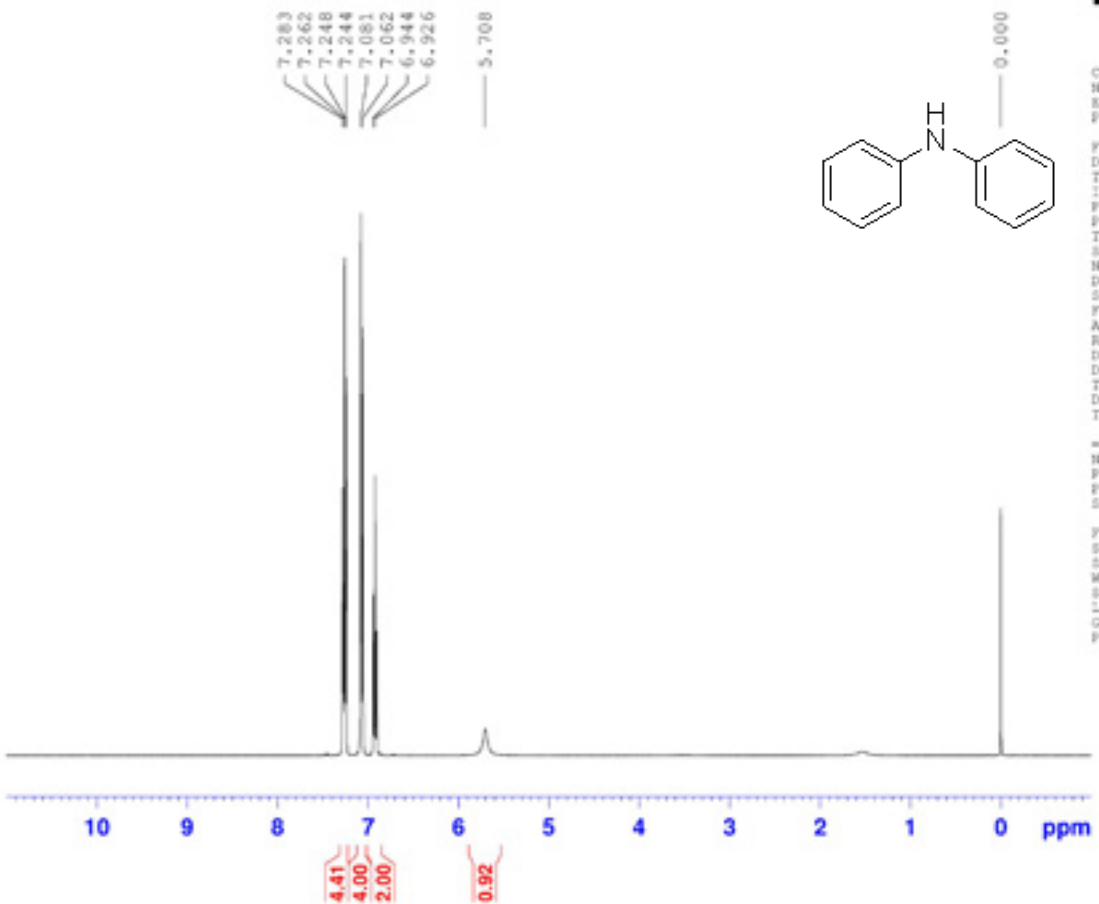
======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -2.00 dB
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                32768
SF          400.1300199 MHz
WDW                  EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```


system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
146.43
135.63
129.01
127.90
127.64
126.48
126.13
121.97
118.26
104.34
77.69
77.36
77.04
44.20
32.11
29.65
29.45
27.46
22.96
14.43
H
N
BRUKER
Current Data Parameters
NAME 2007-7-3 tangqiang-GNNA
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070703
Time 23.56
INSTRUM apect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 130
DS 4
SWH 24038.462 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.00 usec
TE 298.7 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 -2.00 dB
PL12 12.05 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
140
130
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm


gwna
%Transmittance
Wavenumbers (cm-1)
3412.53
3050.49
2926.45
2854.95
1897.79
1630.13
1603.73
1522.24
1475.08
1399.78
1358.81
1225.29
1189.14
1145.26
827.35
743.47
H
N

system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air



BRUKER

Current Data Parameters
NAME 2007-5-31 tangqiang-ERA
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070531
Time 23.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 322
DW 60.800 usec
DE 6.00 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 32768
SF 400.1300141 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air

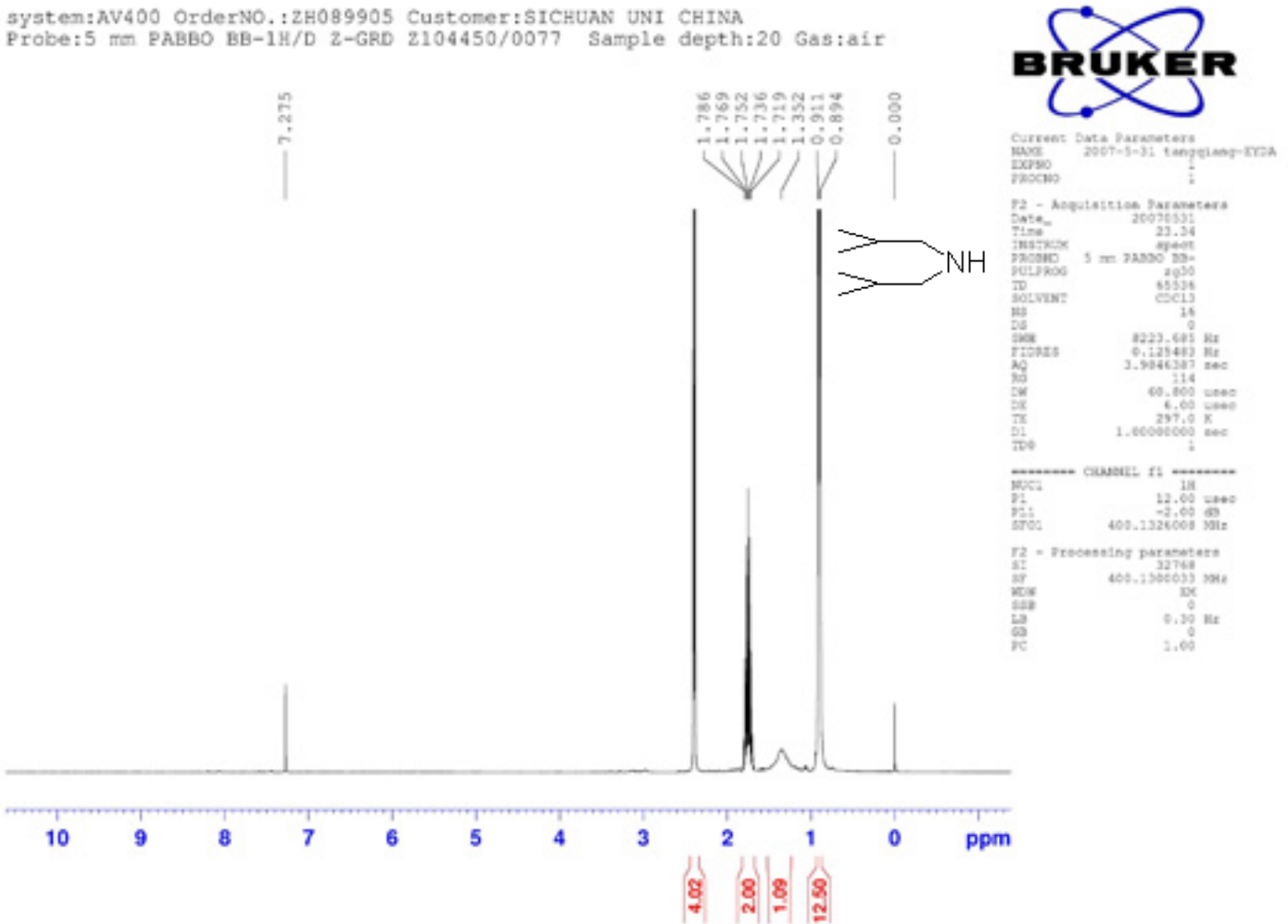
Current Data Parameters
NAME 2007-5-31 tangqiang-ERA
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070531
Time 18.12
INSTRM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 65
DS 4
SWH 24038.462 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 297.7 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1

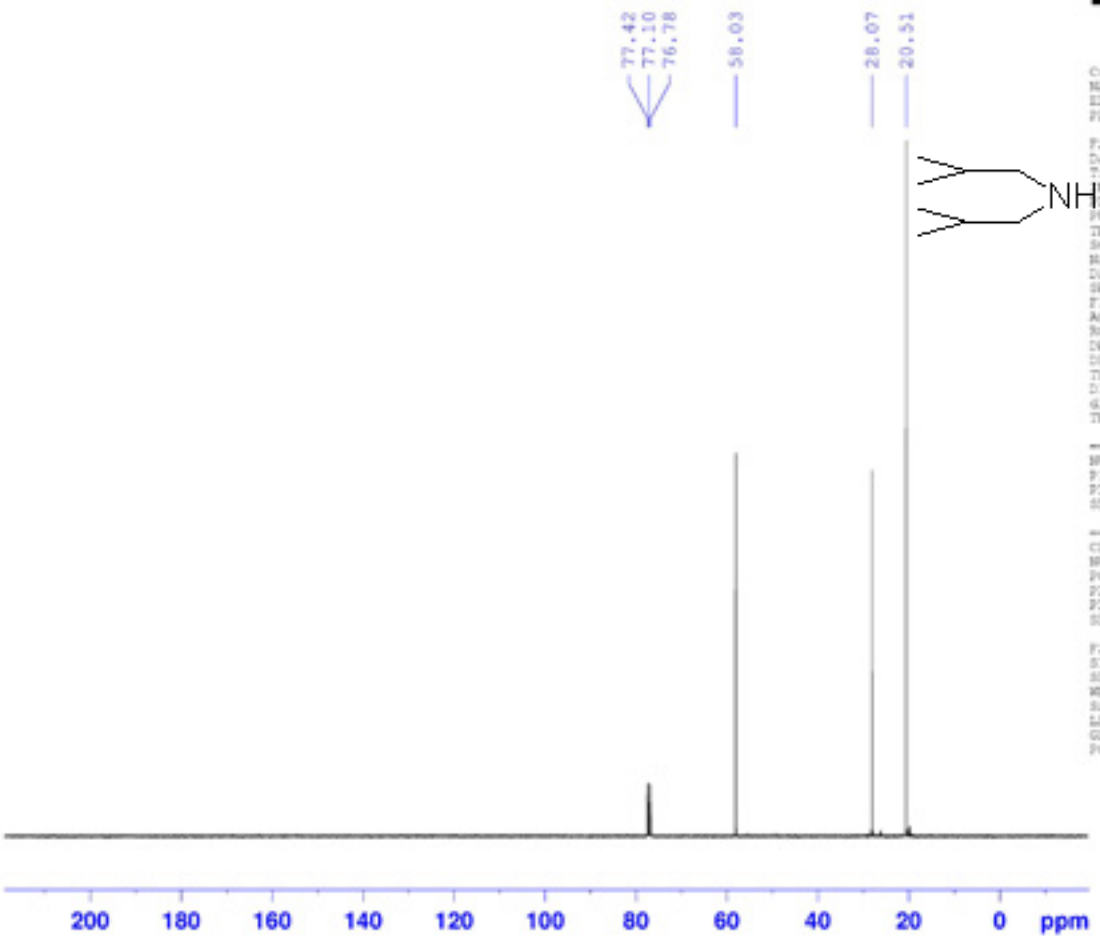
======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
BRUKER
7.275
1.786
1.769
1.752
1.736
1.719
1.352
0.911
0.894
0.000
NH
10
9
8
7
6
5
4
3
2
1
0
ppm
4.02
2.00
1.09
12.50
Current Data Parameters
NAME 2007-5-31 tangqiang-EYDA
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070531
Time 23.34
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 114
DW 60.800 usec
DE 6.00 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1326008 MHz
F2 - Processing parameters
SI 32768
SF 400.1300033 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air



BRUKER

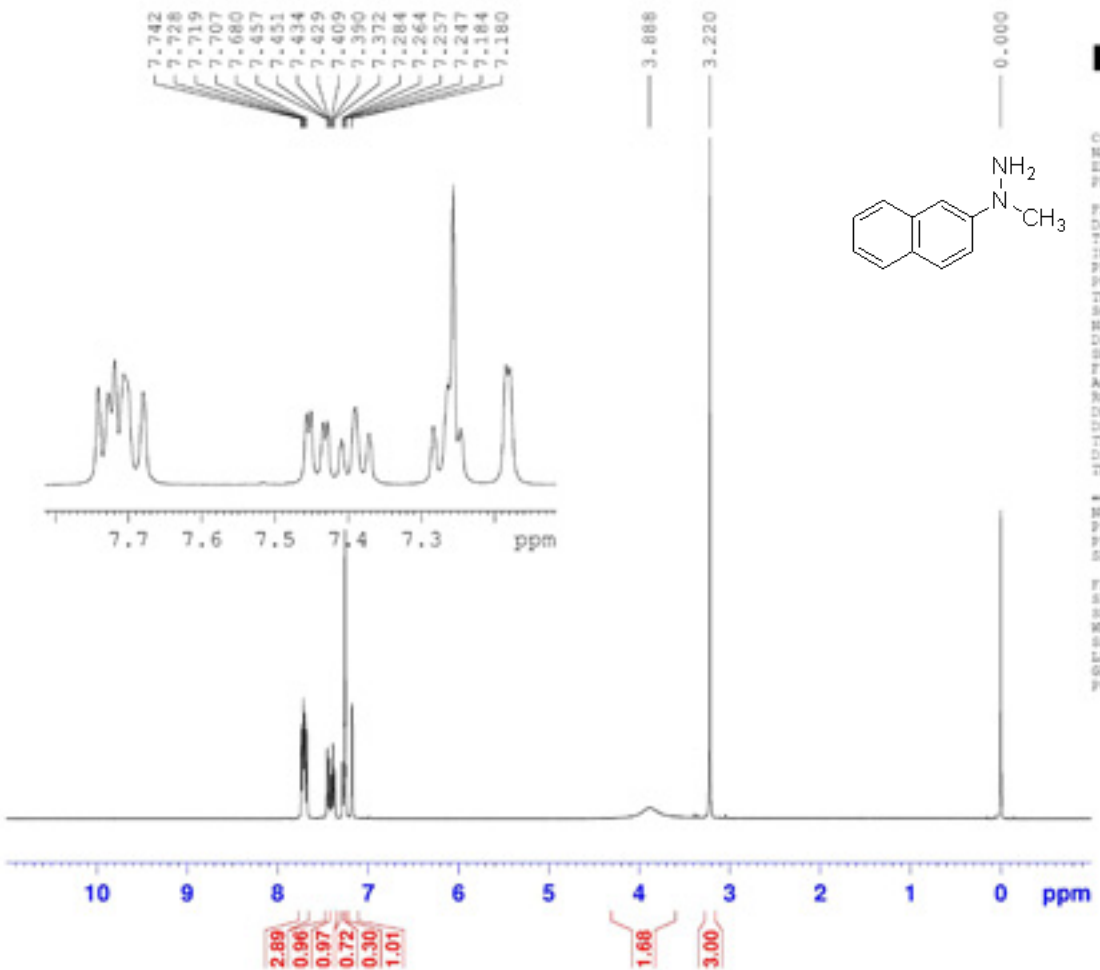
Current Data Parameters
NAME 2007-5-31 tangqiang-EYDA
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070531
Time 18.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 297.8 K
D1 2.00000000 sec
GL1 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 9.60 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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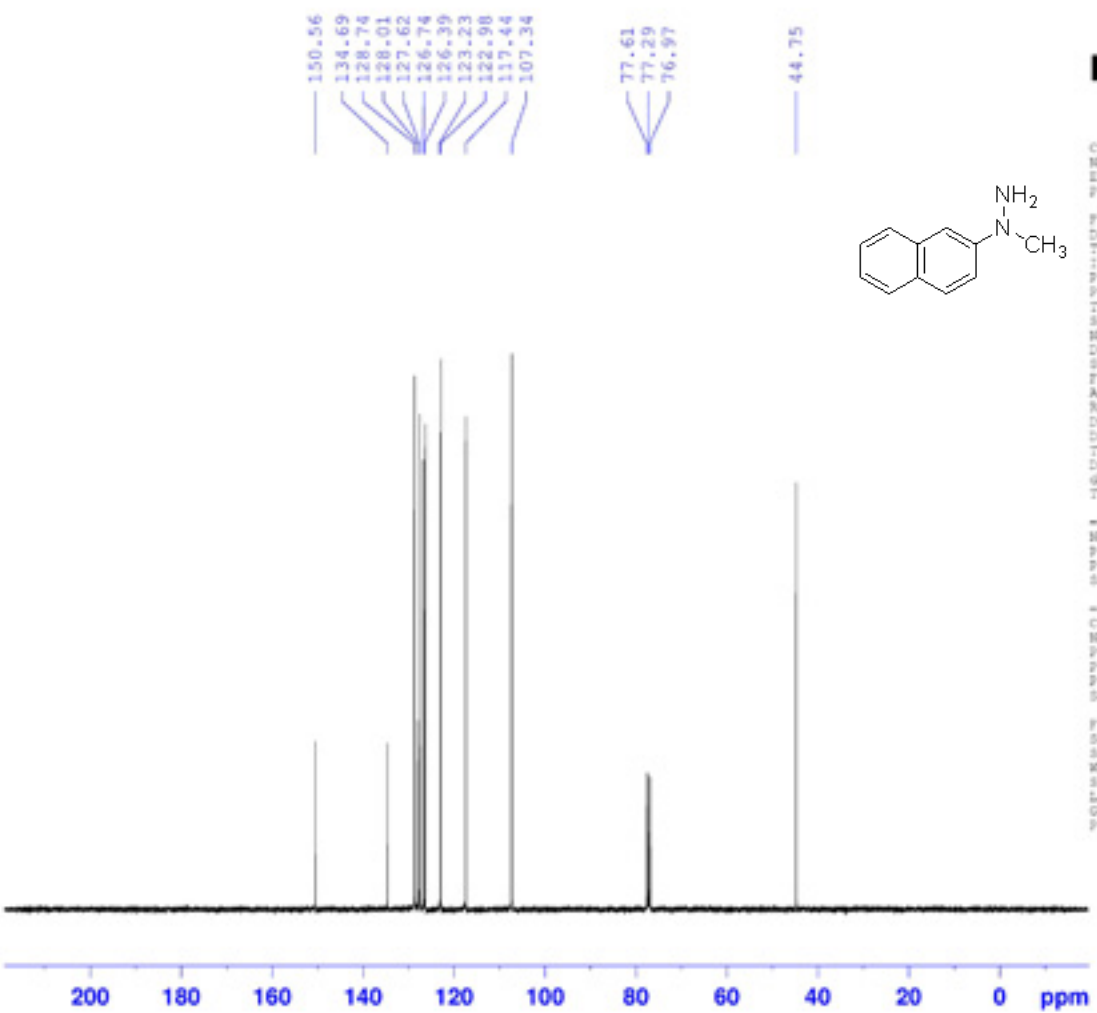
Current Data Parameters
NAME      2007-7-18 tangqiang-NJNU
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20070718
Time              16.53
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                    16
DS                     0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   362
DW                60.800 usec
DE                 6.00 usec
TE                296.2 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                32768
SF           400.1300105 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



BRUKER

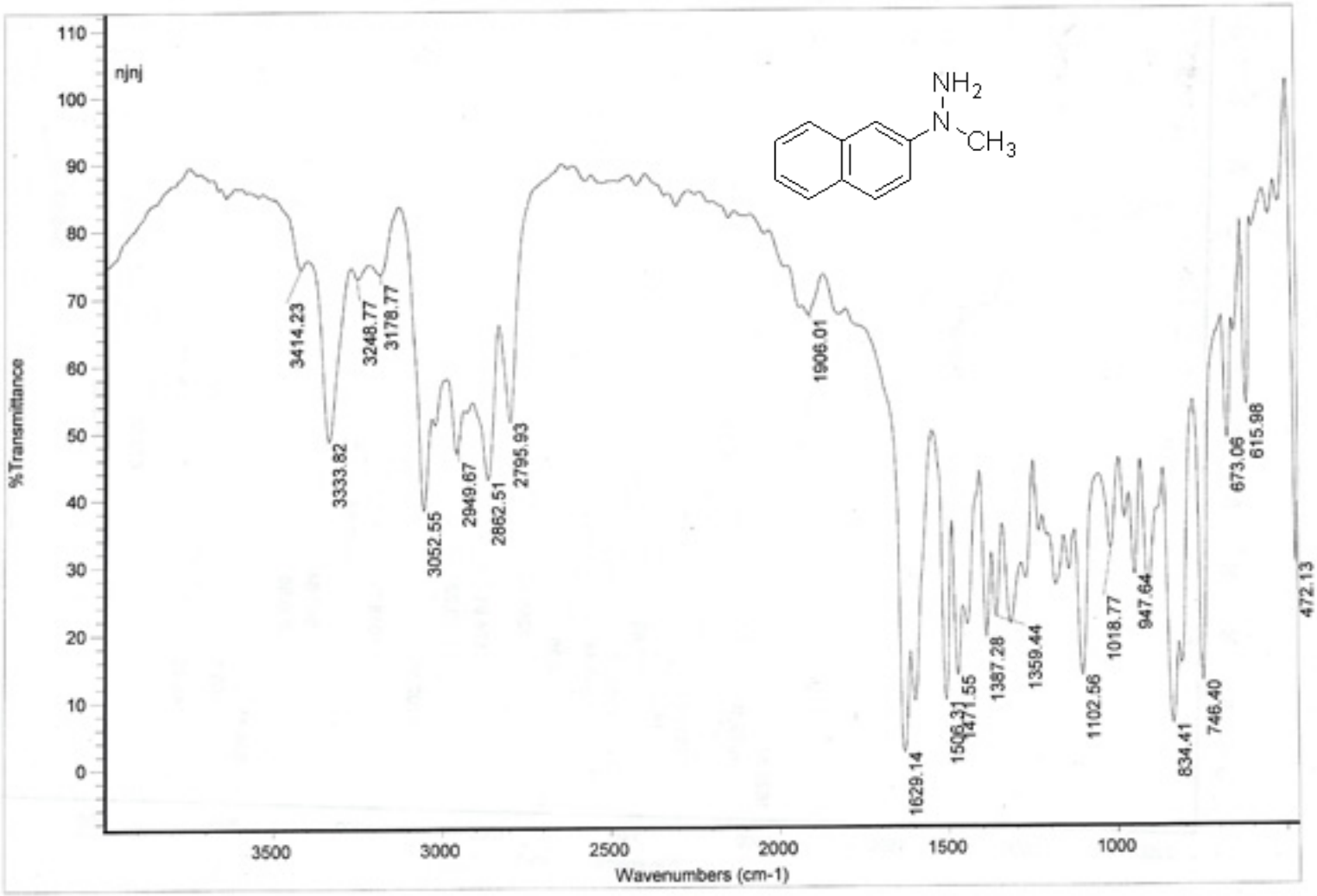
Current Data Parameters
NAME 2007-7-18 tangqiang-WJSJ
EXPNO 2
PROCNO 1

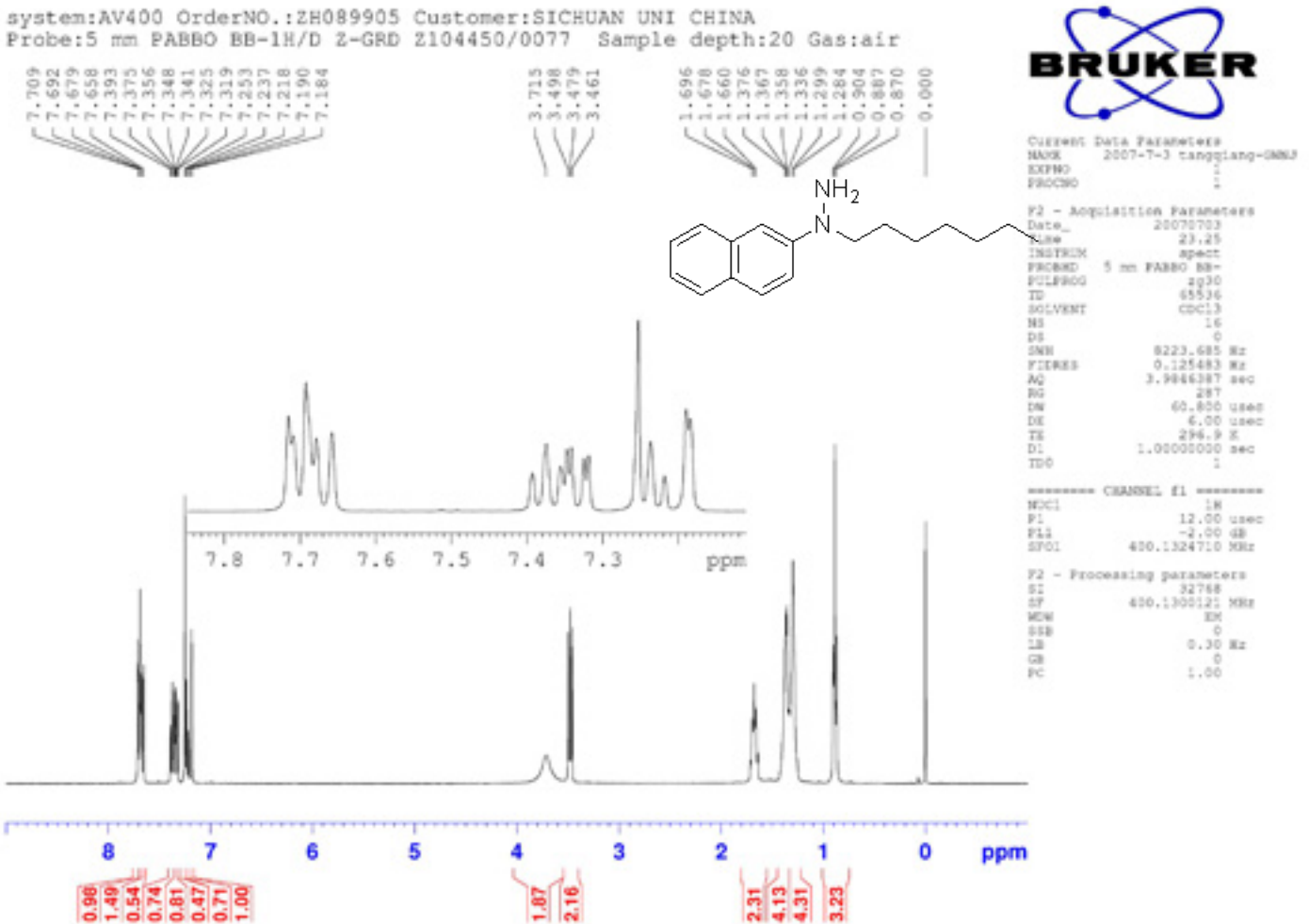
F2 - Acquisition Parameters
Date_ 20070718
Time 17.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 130
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
GL1 0.03000000 sec
TD0 1

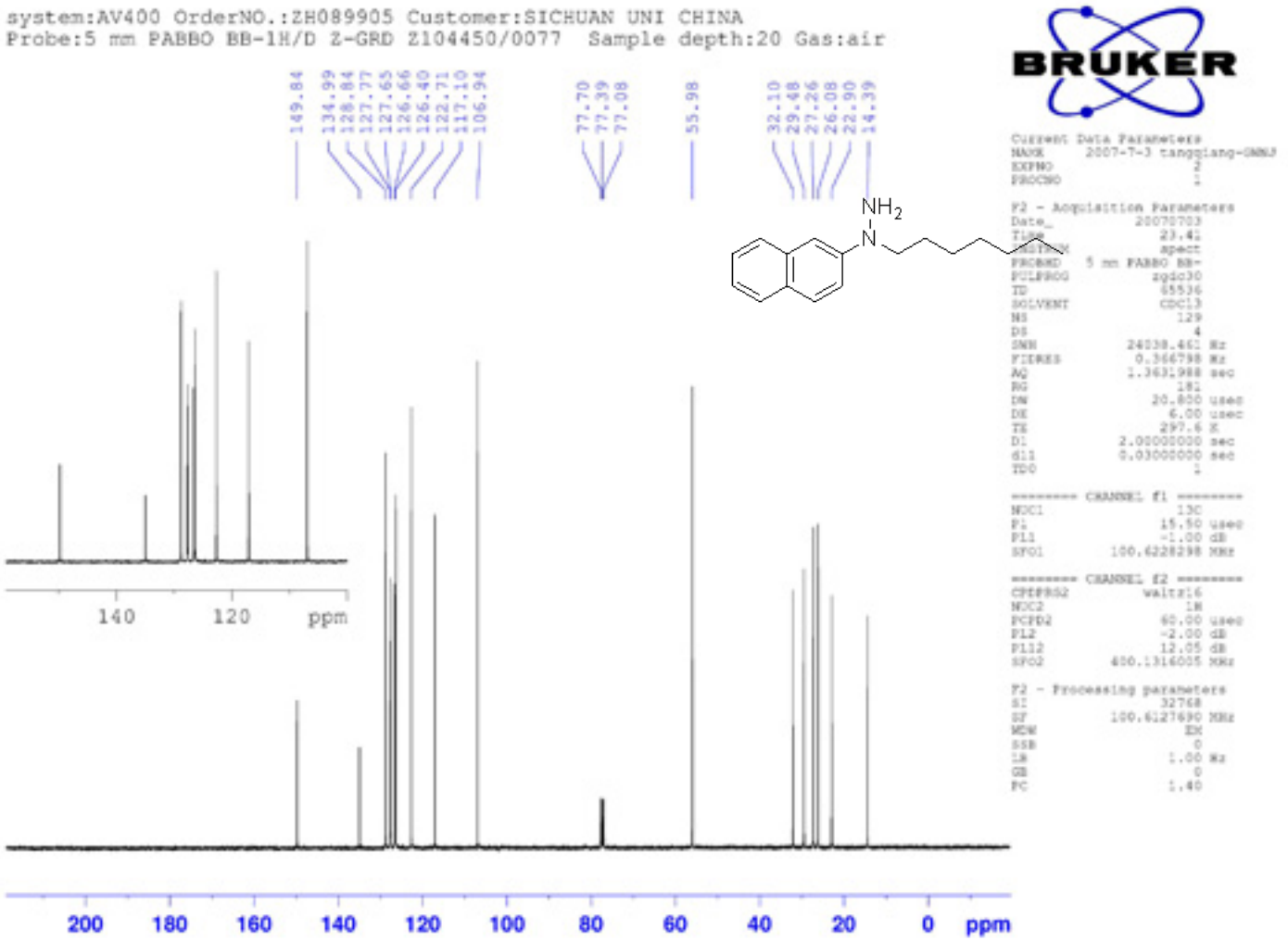
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz

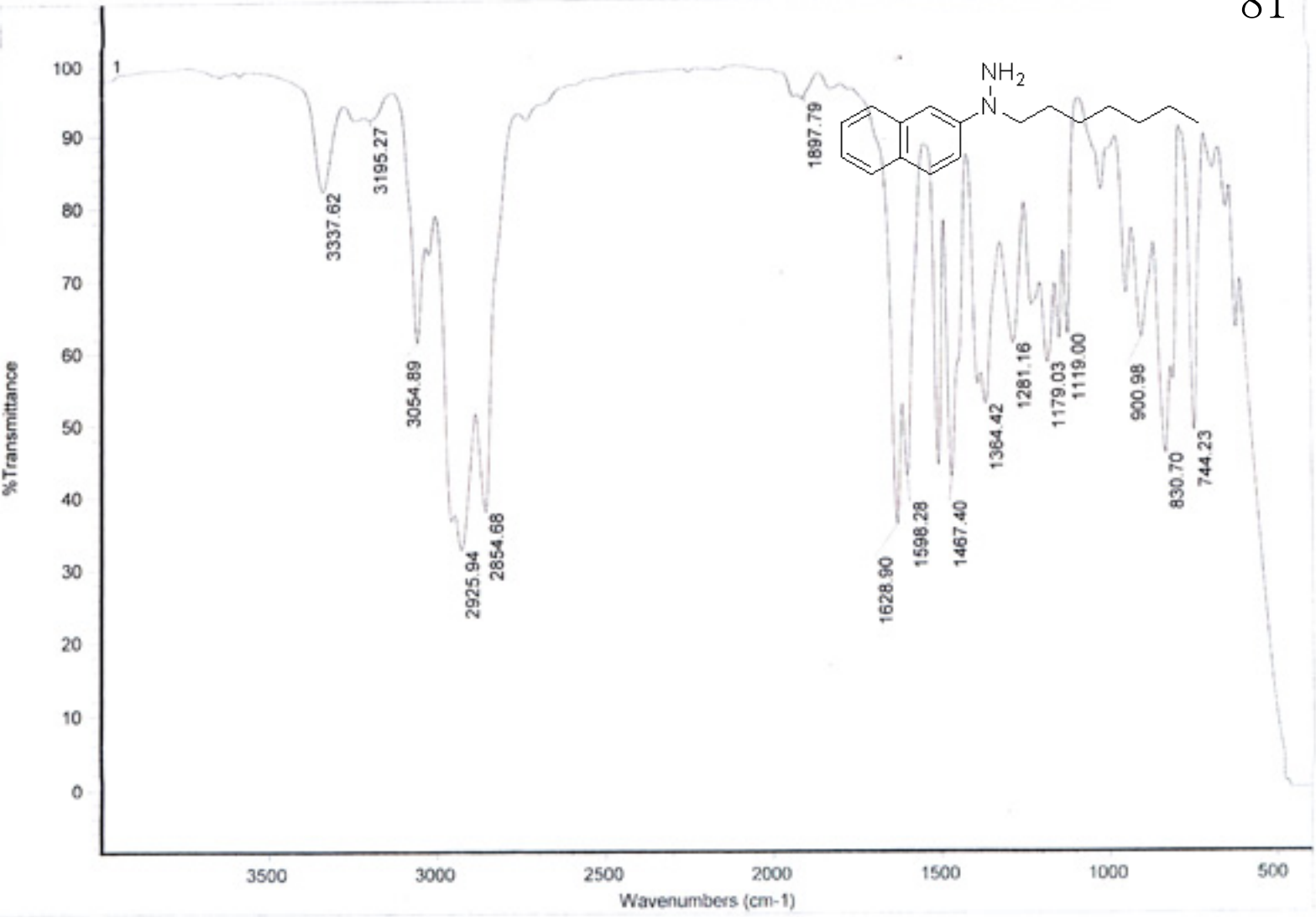
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 -2.00 dB
PL12 12.05 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


njnj
%Transmittance
Wavenumbers (cm-1)
3414.23
3333.82
3248.77
3178.77
3052.55
2949.67
2862.51
2795.93
1906.01
1629.14
1506.31
1471.55
1387.28
1359.44
1102.56
1018.77
947.64
834.41
746.40
673.06
615.98
472.13
NH2
N
CH3


system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
7.709
7.692
7.679
7.658
7.393
7.375
7.356
7.348
7.341
7.325
7.319
7.253
7.237
7.218
7.190
7.184
3.715
3.498
3.479
3.461
1.696
1.678
1.660
1.376
1.367
1.358
1.336
1.299
1.284
0.904
0.887
0.870
0.000
NH2
N
7.8
7.7
7.6
7.5
7.4
7.3
ppm
8
7
6
5
4
3
2
1
0
ppm
0.98
1.49
0.54
0.74
0.81
0.47
0.71
1.00
1.87
2.16
2.31
4.13
4.31
3.23
BRUKER
Current Data Parameters
NAME 2007-7-3 tangqiang-GNNJ
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070703
Time 23.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 287
DW 60.800 usec
DE 6.00 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


system:AV400 OrderNO.:ZH089905 Customer:SICHUAN UNI CHINA
Probe:5 mm PABBO BB-1H/D Z-GRD Z104450/0077 Sample depth:20 Gas:air
149.84
134.99
128.84
127.77
127.65
126.66
126.40
122.71
117.10
106.94
77.70
77.39
77.08
55.98
32.10
29.48
27.26
26.08
22.90
14.39
NH2
N
BRUKER
Current Data Parameters
NAME 2007-7-3 tangqiang-GNNJ
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070703
TIME 23.41
INSTRUM apect
PROBHD 5 mm PABBO BB-
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 129
DS 4
SWH 24038.462 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 181
DW 20.800 usec
DE 6.00 usec
TE 297.6 K
D1 2.00000000 sec
d11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 15.50 usec
PL1 -1.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 -2.00 dB
PL12 12.05 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
140
120
ppm
200
180
160
140
120
100
80
60
40
20
0
ppm


%Transmittance
100
90
80
70
60
50
40
30
20
10
0
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
3337.62
3195.27
3054.89
2925.94
2854.68
1897.79
1628.90
1598.28
1467.40
1364.42
1281.16
1179.03
1119.00
900.98
830.70
744.23
NH2
N

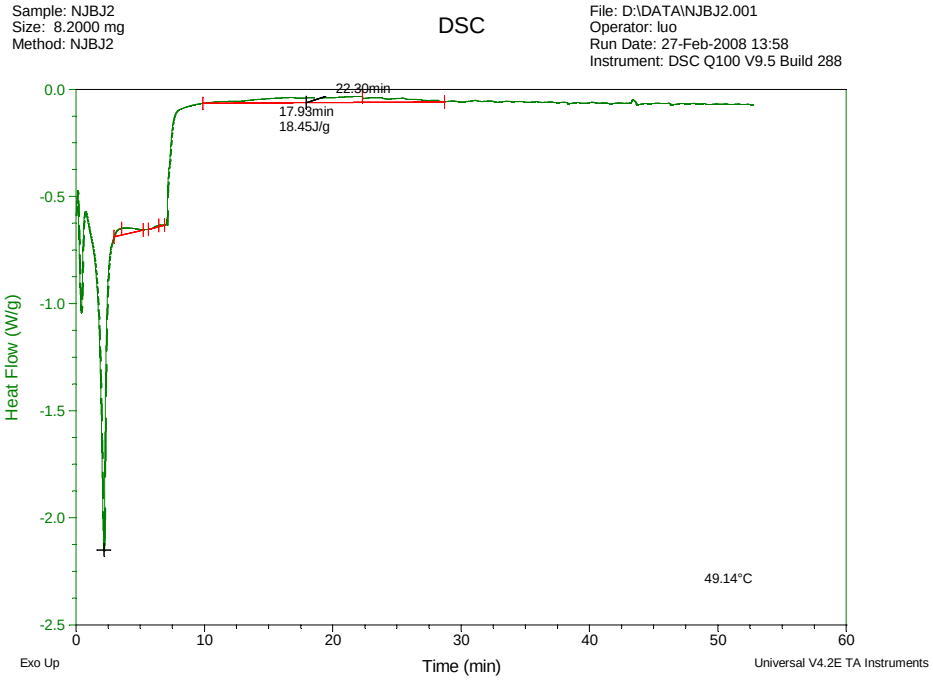


Figure 1.1: DSC trace of a mixture of 2-naphthol (4.4 mmg, 3.1 mmol) and *N*-methyl-*N*-phenylhydrazine (3.8 mmg, 3.1 mmol). The temperature was increased from 30 ^{o}C to 100 ^{o}C (10 ^{o}C / min) and then held at 100 ^{o}C for 1 hour. (Heat Flow-Time curves)

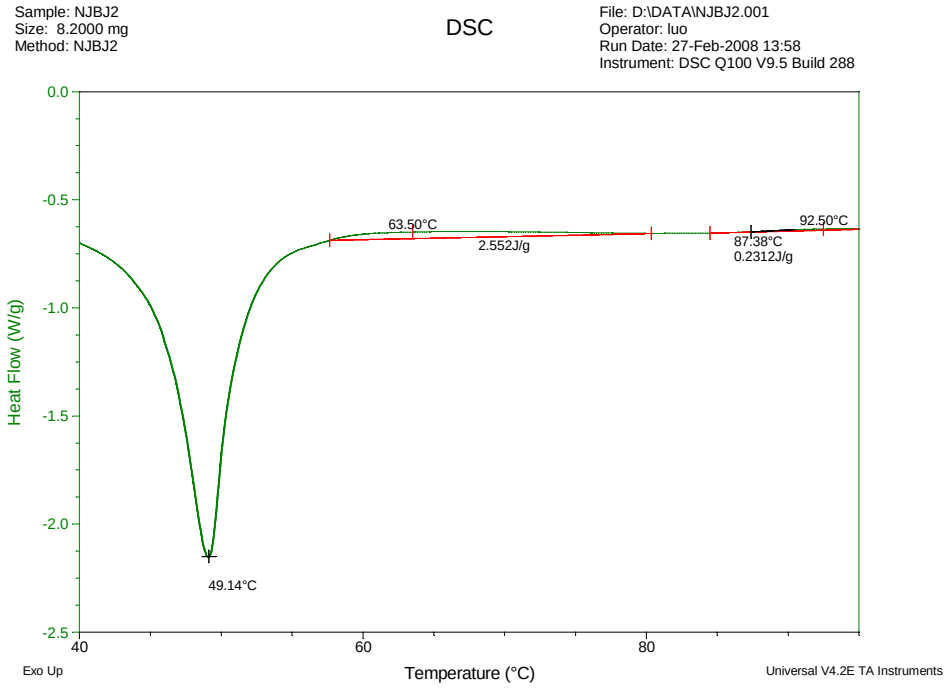


Figure 1.2: DSC trace of a mixture of 2-naphthol (4.4 mmg, 3.1 mmol) and N-methyl-N-phenylhydrazine (3.8 mmg, 3.1 mmol). The temperature was increased from 30 ^{o}C to 100 ^{o}C (10 ^{o}C / min) and then held at 100 ^{o}C for 1 hour. (Magnified Heat Flow-Temperature curves)

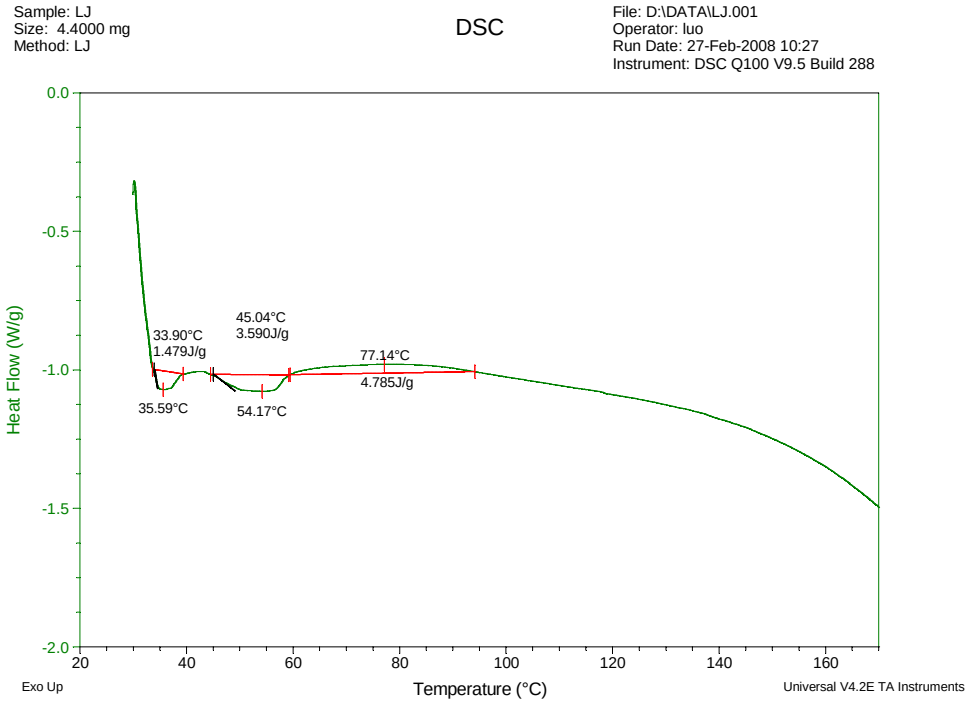


Figure 2: DSC trace of a mixture of 2-naphthol (2.0 mmg, 1.4 mmol) and N-methyl-N-(naphthalene-2-yl)hydrazine (2.4 mmg, 1.4mmol). The temperature was increased from 30 ^{o}C to 170 ^{o}C (10 ^{o}C / min).

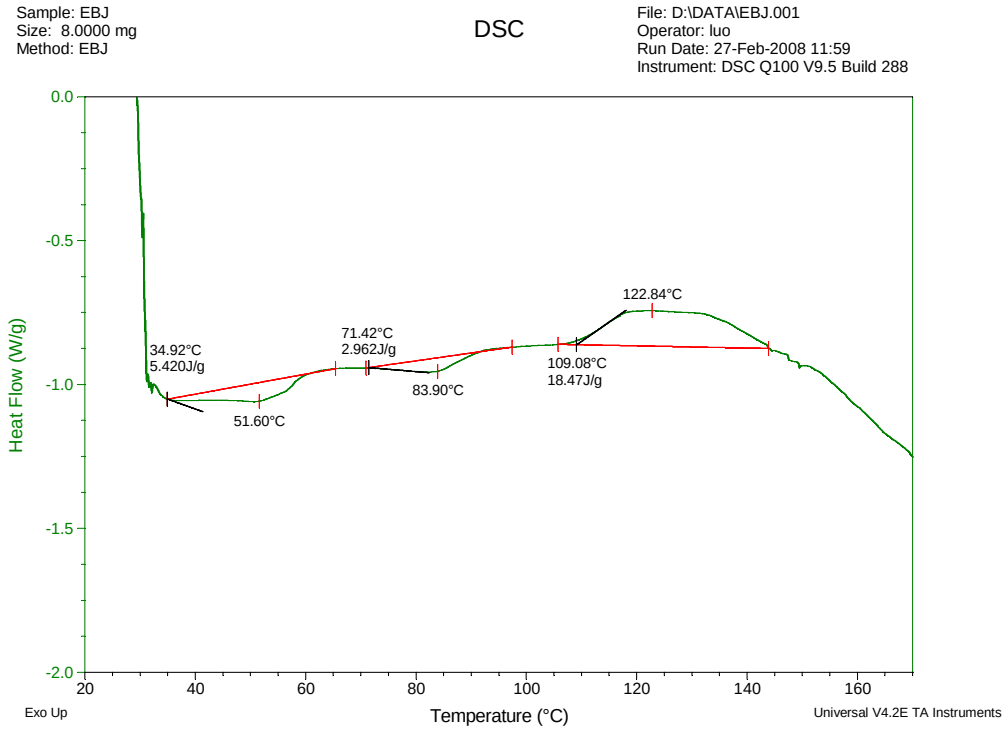


Figure 3: DSC trace of a mixture of 2-naphthol (3.5 mmg, 2.4 mmol) and N,N-diphenylhydrazine (4.5 mmg, 2.4 mmol) . The temperature was increased from 30 ^{o}C to 170 ^{o}C (10 ^{o}C / min).