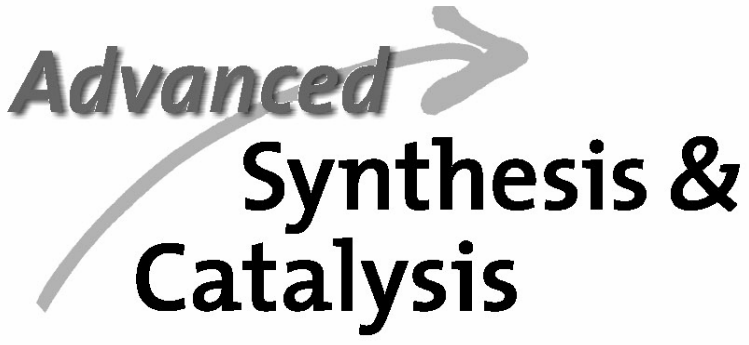


Supporting Information

Supporting Information for

Base-catalyzed and Solvent-dependent Cascade Reaction in Regioselective Synthesis of Novel Fused Polycycles

Min Xia*, Guo-Feng Xiang, Bin Wu and Yi-feng Han

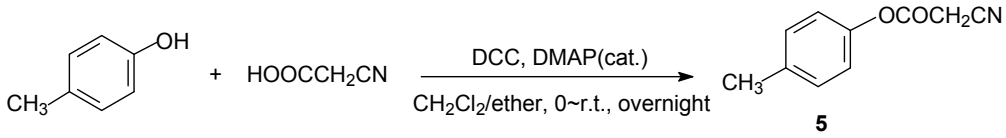
Department of Chemistry, Zhejiang Sci-Tech University,
Hangzhou 310018, P. R. China
xiamin@zstu.edu.cn

Contents:

I. General Considerations

All the compounds used are analytical reagents and some chemicals are further purified by recrystallization or distillation. Melting points are determined on X-4 micro-melting instrument and thermometer is uncorrected. The ^{1}H NMR (400MHz), ^{13}C NMR (100MHz), spectra are obtained on a Bruker Avance II DMX400 spectrometer using DMSO-d_6 or $CDCl_3$ as the solvent. Chemical shifts are reported relative to residue peaks of DMSO-d_6 (2.50 ppm for ^{1}H and 39.51ppm for ^{13}C) and $CDCl_3$ (7.26ppm for ^{1}H and 77.23ppm for ^{13}C). ^{1}H NMR signals at 3.33ppm and 1.56ppm are attributed to protons of H_2O in DMSO-d_6 and $CDCl_3$. The ^{1}H NMR and ^{13}C NMR are carried out using trimethylsilane as the internal standard. Due to the existence of H-D exchange between sample and deuterated solvents, amino groups are unable to give out their full signals. The following abbreviations are used to describe peak patterns where appropriate: b = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants are recorded in Hertz (Hz). FT-IR spectra are recorded as KBr pellets on a Nicolet Avatar spectrophotometer. Elementary analysis is executed on a CHN Micro-analyzer. X-ray crystallographic data is collected on a Bruker APEX-II apparatus. Absorption spectra are measured on a Shimadzu UV 2501(PC)S uv-vis spectrometer and fluorescence spectra are measured on a Perkin Elmer LS55 spectrophotometer. The absolute fluorescent quantum yields of compounds **3a~n** are measured with naphthacence (Φ_F = 0.6, λ= 443nm) as a standard. EI-MS spectra for **6b~c** are recorded on a GCT Pemier mass spectrometer with 70eV attack, PIP temperature range is 50~250°C. APCI-MS spectra for **6b** is measured on an Agilent 6210 mass spectrometer (nebulizer: 10psig, dry gas: 4L/min, gas tempature: 325°C, vaporizer: 350°C, Corona needle: 4.0μA, ion mode: positive). EI-MS for other compounds are executed on a HP 1105 mass spectrometer with 70eV attack. Column chromatography is carried out on silica gel (10~40 mesh) with mixed solvents (petroleum-ethyl acetate; ethyl ether-petroleum ether).

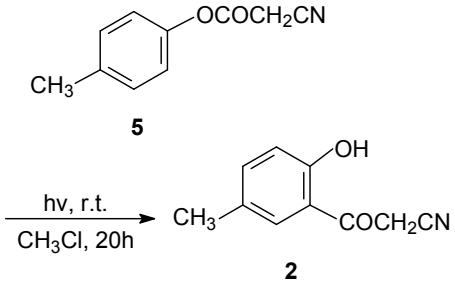
II. General Procedure for *p*-Tolyl 2-cyanoacetates (5)

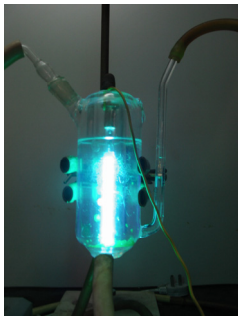


5

At room temperature, N, N-dicyclohexylcarbodiimine (DCC) (33mmol, 6.80g) and 4-(N, N-dimethyl)aminopyridine (DMAP) (1.5mmol, 0.183g) are added to the solution of substituted phenol (30mmol) in anhydrous CH_2Cl_2 (50mL) and the resulted solution is stirred and kept in an ice bath at 0 °C, cyanoacetic acid (33mmol, 2.80g) in anhydrous ether (30mL) is dropped into such mixture. Untill the dropping is finished, the ice bath is removed and the mixture is stirred at room temperature overnight. After filtration, the filtrate is evaporated under reduced pressure and the residue is separated by column chromatography using ethyl ether-petroleum ether (1:2) as the eluent to afford the pure *p*-tolyl 2-cyanoacetate **5**.

III. General Procedure for 3-(2-Hydroxy-5-methylphenyl)-3-oxo-propionitriles (2)

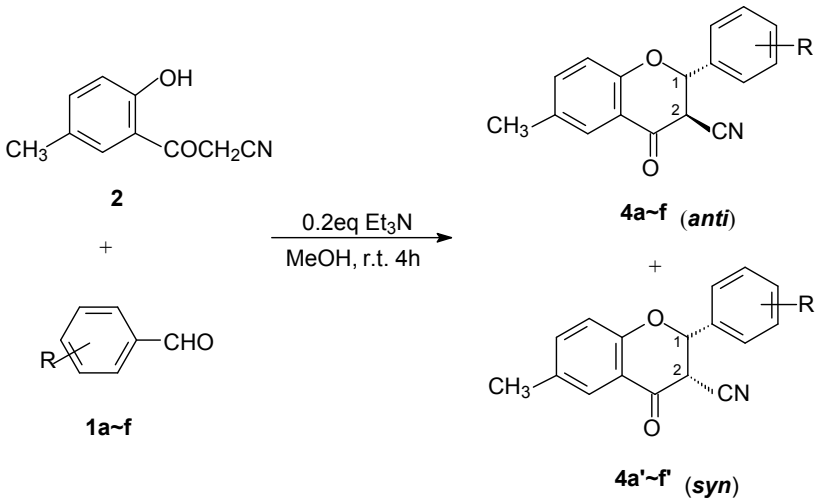




At room temperature, the CH_3Cl solution (60mL) of *p*-tolyl 2-cyanoacetate (2.3mmol, 0.402g) is poured into the quartz photo-reactor (made by ourselves, see the above picture) and then bubbled by N_2 flow. The solution is irradiated under high-pressure mercury lamp (λ=254nm) for 20h while the quartz apparatus is cooled by water flow. The solution is gradually turned from colorless to brownish yellow and then removed the solvent under reduced press by a rotating evaporator. The residue is separated on a column chromatography using petroleum ether-ethyl acetate (4:1) as the eluent to afford pure

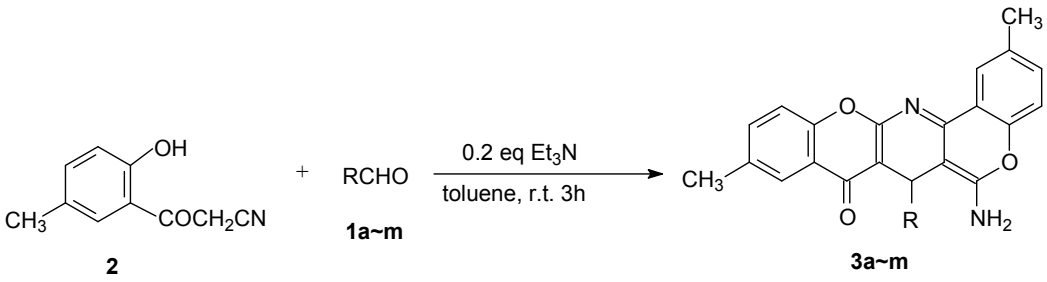
3-(2-hydroxy-5-methylphenyl)-3-oxo-propionitrile **2**.

IV. General procedure for 6-Methyl-4-oxo-2-(4-aryl)-3,4-dihydro-2*H*-chromene-3-carbonitriles (4a~f /4a′~f′)



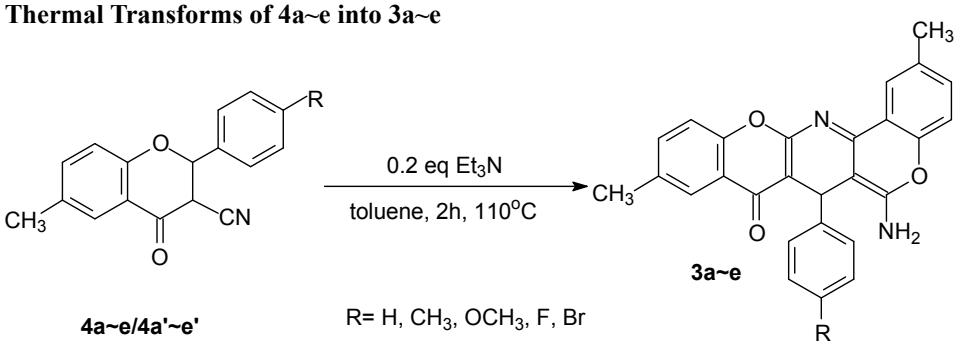
At room temperature, Et_3N (0.2mmol) is added to the solution of 3-(2-hydroxy-5-methylphenyl)-3-oxo-propanenitrile (1mmol) and aldehyde (1.2mmol) in methanol (5mL), the resulted mixture is stirred for 4h and then quenched by 0.1N HCl aqueous solution. The solution is poured into water and extracted by EtOAc for two times. The combined organic layers are dried over anhydrous Na_2SO_4 and the solvent is removed under the reduced pressure. The residue is isolated on a silica gel column chromatography using petroleum/ethyl acetate (3:1) as the eluent to offer the mixture of **4/4′** in good yields.

V. General Procedure for 6-Amino-2,10-dimethyl-7-substituted-7*H*-5,13-dioxa-14-aza-benzo[*a*] naphthacen-8-ones (3a~m)



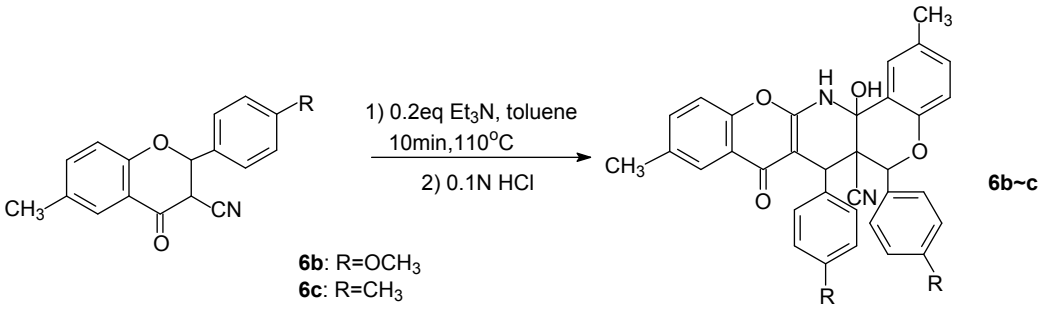
At room temperature, Et_3N (0.2mmol) is added to the solution of 3-(2-hydroxy-5-methylphenyl)-3-oxo-propionitrile (1mmol) and aldehyde (1mmol) in toluene (5mL) and the resulted solution is stirred for 3h. A yellow precipitate is gradually generated. After filtration, the solid is washed with some ether and ethyl acetate. The dried solid **3** is pure enough for spectral analysis.

VI. Thermal Transforms of 4a~e into 3a~e



At room temperature, Et_3N (0.2mmol) is added to the solution of 6-methyl-4-oxo-2-(4-aryl)-3,4-dihydro-2*H*-chromene-3-carbonitrile (1mmol) in toluene (5mL) and the resulted solution is heated at 110°C. After 15min, the colorless solution turns brightly yellow and the yellow solid is gradually generated, the reaction is continuously carried out at the same temperature for 2h. After cooling to room temperature, the precipitate is filtrated and washed with some ether and ethyl acetate. The dried solid is pure enough for spectral analysis.

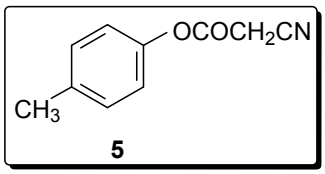
VII. General procedure for Intermediates 6b~c



6b: R=OCH_3
6c: R=CH_3

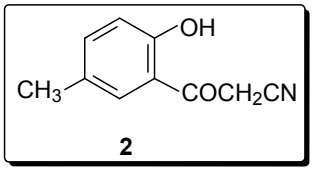
At room temperature,Et_3N (0.2mmol) is added to the solution of 6-methyl-4-oxo-2-aryl-3,4-dihydro-2*H*-chromene-3-carbonitrile (1mmol) in toluene (5mL) and the resulted solution is heated at 110°C. After 10min, 0.1N HCl (0.5mL) aqueous solution is dropped to quench the reaction. When the solution is cooled to room temperature, it is filtrated and water is poured into it. The solution is extracted by EtOAc(2x10mL), the combined organic layer is dried over anhydrous Na_2SO_4 overnight. After filtration, the solvent is removed from the filtrate at reduced pressure and the residue is isolated by a column chromatography using petroleum ether-ethyl acetate (2:1) as the eluent to provide the intermediates **6b~c** as white solids.

VIII. Characterization Data for Compounds 5, 2, 6b~c, 4a~f/4a′~f′ and 3a~m



5

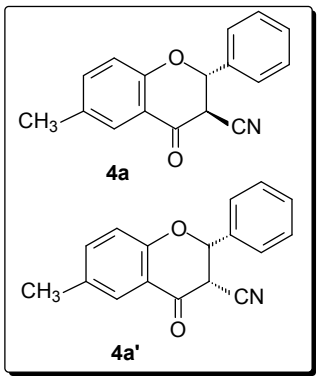
p-Tolyl 2-cyanoacetate 5: 88% yield, colorless oil; FT-IR(liquid film): υ 2966, 2263, 1770, 1507, 1337, 1198, 831cm^{-1}; ^{1}H NMR(400 MHz, $CDCl_3$): δ 2.35(s, 3H), 3.68(s, 2H), 7.01(d, *J*=8.4Hz, 2H), 7.19(d, *J*=8.4Hz, 2H); ^{13}C NMR(100MHz, $CDCl_3$): δ 20.85, 24.91, 112.70, 120.64, 130.17, 136.54, 147.90, 161.76; EI-MS(70ev): *m/z*(%)175(M^+, 45), 108(100), 91(10), 77(35), 68(9), 51(13).



2

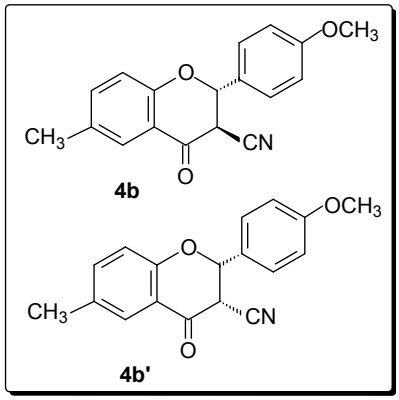
3-(2-Hydroxy-5-methylphenyl)-3-oxo-propanenitrile 2: 21% yield, white solid, m.p. 131-132°C; FT-IR(KBr): υ 3421, 2950, 2261, 1644, 1490, 1357, 1269, 1242, 761, 752 cm^{-1}; ^{1}H NMR(400MHz, $CDCl_3$): δ 2.33(s, 3H), 4.13(s, 2H), 6.96(d, *J*=8.4Hz, 1H), 7.31(s, 1H), 7.38(d, *J*=8.4Hz, 1H), 11.26(s, 1H); ^{13}C NMR(100MHz, $CDCl_3$): δ 20.51, 29.35, 113.16, 117.22, 118.96, 128.85, 129.05, 139.32, 160.78, 192.21; EI-MS(70ev): *m/z*(%) 175(M^+,80), 135(M^+-CH_2CN, 100), 107(M^+-$COCH_2CN$, 36), 77(53), 51(18).

mixture of **4a/4a'**



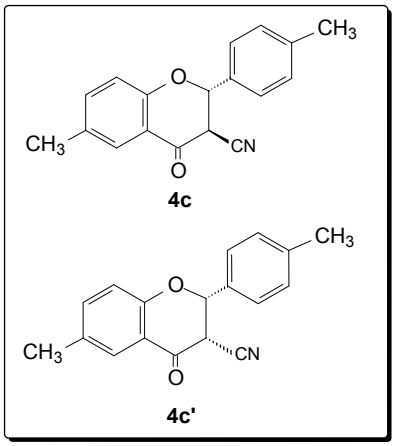
6-Methyl-4-oxo-2-phenyl-3,4-dihydro-2H-chromene-3-carbonitrile 4a/4a′: 86% all yield, white solid, FT-IR(KBr): υ 3063, 2260, 1704, 1617, 1491, 1297, 1225, 837 cm^{-1}; ^{1}H NMR(400MHz, $CDCl_3$): δ 2.36(s, 3H), 3.77(d, *J*=2.4Hz, 0.12H), 4.15(d, *J*=12.4Hz, 0.89H), 0.46(d, *J*=12.4Hz, 0.92H), 5.52(d, *J*=2.8Hz, 0.13H), 6.98(d, *J*=12.4Hz, 0.92H), 7.07(d, *J*=8.4Hz, 0.14H), 7.41(d, *J*=8.4Hz, 1H), 7.50(d, *J*=5.6Hz, 3H), 7.57(m, 2H), 7.78(s, 1H); ^{13}C NMR(100MHz, $CDCl_3$): δ 20.47, 47.39, 80.91, 113.26, 118.08, 118.42, 126.93, 127.41, 129.16, 130.16, 132.62, 135.31, 138.68, 158.86, 182.69; EI-MS(70ev): *m/z*(%) 262(M^+-1, 52), 134(100), 106(20), 77(26), 51(13). Anal. Calcd. for $C_{17}H_{13}NO_2$: C, 77.53; H, 4.98; N, 5.32. Found: C, 77.41; H, 4.91; N, 5.34.

mixture of **4b/4b'**



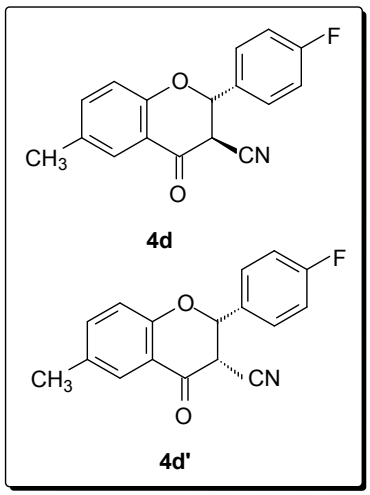
6-Methyl-4-oxo-2-(4-methoxylphenyl)-3,4-dihydro-2H-chromene-3-carbonitrile 4b/4b': 95% all yield, white solid, FT-IR(KBr): υ 3004, 2889, 2261, 1702, 1615, 1488, 1293, 1248, 1130, 830cm^{-1}; ^{1}H NMR (400MHz, $CDCl_3$): δ 2.35(s, 3H), 3.82(s, 3H), 4.14(d, *J*=12.4Hz, 1H), 5.40(d, *J*=12.0Hz, 1H), 6.96(d, *J*=8.8Hz, 1H), 7.00(d, *J*=8.4Hz, 2H), 7.39(d, *J*=8.4Hz, 1H), 7.47(d, *J*=8.4Hz, 2H), 7.75(s, 1H); ^{13}C NMR(100MHz, $CDCl_3$): δ 20.45, 47.40, 55.41, 80.62, 113.39, 114.50, 118.07, 118.39, 127.36, 127.41, 128.40, 132.48, 138.60, 158.94, 160.86, 182.94; EI-MS(70ev): *m/z* (%) 292(M^+-1, 100), 278(15), 159 (32), 134 (70), 107(11), 77(23). Anal. Calcd. for $C_{18}H_{15}NO_3$: C, 73.69; H, 5.16; N, 4.78. Found: C, 73.55; H, 5.11, N, 4.83.

mixture of **4c/4c'**



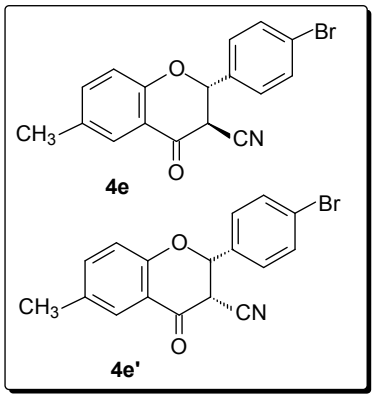
6-Methyl-4-oxo-2-(4-methylphenyl)-3,4-dihydro-2H-chromene-3-carbonitrile 4c: 91% all yield, white solid; FT-IR(KBr): υ 2885, 2259, 1701, 1617, 1489, 1298, 1224, 1131, 819 cm^{-1}; ^{1}H NMR(400MHz, $CDCl_3$): δ 2.35(s, 3H), 2.41(s, 3H), 3.75(d, *J*=3.2Hz, 0.07H), 4.14(d, *J*=12.0Hz, 0.94H), 5.42(d, *J*=12.4Hz, 0.96H), 5.49(d, *J*=2.4Hz, 0.08H), 6.96(d, *J*=8.4Hz, 0.96H), 7.06(d, *J*=8.8Hz, 0.12H), 7.29(d, *J*=7.6Hz, 2H), 7.39(d, *J*=8.4Hz, 1H), 7.43(d, *J*=7.6Hz, 2H), 7.76(s, 1H); ^{13}C NMR(100MHz, $CDCl_3$): δ 20.47, 21.36, 47.35, 80.83, 113.37, 118.10, 118.41, 126.89, 127.38, 129.82, 132.39, 132.52, 138.63, 140.26, 158.94, 182.88; EI-MS(70ev): *m/z*(%) 276(M^+-1, 62), 262(24), 134(100), 115(11), 105(21), 78(21), 52(10). Anal. Calcd. for $C_{18}H_{15}NO_2$: C, 77.94; H, 5.45; N, 5.05. Found: C, 77.82; H, 5.49; N, 4.99.

mixture of **4d/4d'**



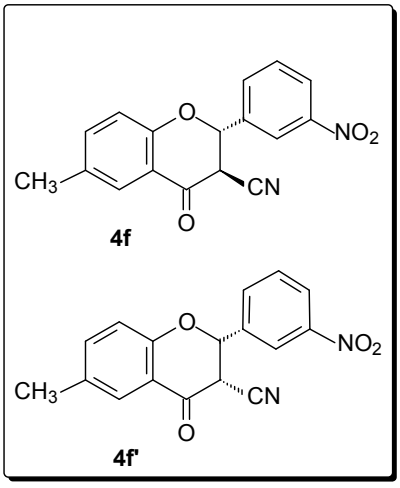
6-Methyl-4-oxo-2-(4-fluorophenyl)-3,4-dihydro-2H-chromene-3-carbonitrile 4d: 78% all yield, white solid, FT-IR(KBr): υ 2259, 1707, 1615, 1515, 1489, 1298, 845, 833 cm^{-1}; ^{1}H NMR(400MHz, $CDCl_3$): δ 2.36(s, 3H), 4.10(d, *J*=12.4Hz, 1H), 5.45(d, *J*=12.4Hz, 1H), 6.97(d, *J*=8.4Hz, 1H), 7.19(t, *J*=8.4Hz, 2H), 7.41(d, *J*=8.4Hz, 1H), 7.56(dd, *J*=5.6, 8.0Hz, 2H), 7.77(s, 1H); ^{13}C NMR(100MHz, $CDCl_3$): δ 20.48, 47.53, 80.17, 113.18, 116.48(d, $^{2}J_{CF}$ =21Hz), 118.02, 118.34, 127.43, 128.90(d, $^{3}J_{CF}$ =9Hz), 131.26, 132,78, 136.75, 158.69, 163,54(d, $^{1}J_{CF}$ =248 Hz), 182.46; EI-MS(70ev): *m/z*(%) 280(M^+-1, 94), 134(100), 106(46), 78(38), 51(18). Anal. Calcd. for $C_{17}H_{12}FNO_2$: C, 72.57; H, 4.30; N, 4.98. Found: C, 72.51; H, 4.33, N, 4.95.

mixture of **4e/4e'**

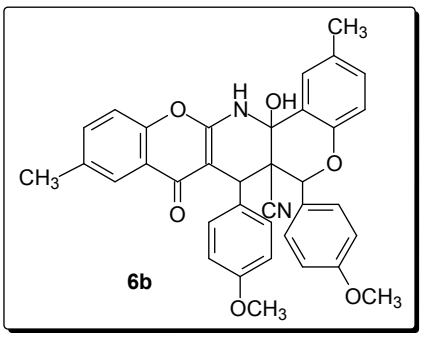


6-Methyl-4-oxo-2-(4-bromophenyl)-3,4-dihydro-2H-chromene-3-carbonitrile 4e/4e': 83% all yield, white solid; FT-IR(KBr): υ 3054, 2886, 2260, 1708, 1616, 1488, 1294, 1222, 825 cm^{-1}; ^1H NMR (400MHz, $CDCl_3$): δ 2.35(s, 3H), 3.76(d, *J*=1.6Hz, 0.27H), 4.09(d, *J*=12.4Hz, 0.69H), 5.43(d, *J*=12.0Hz, 0.73H), 5.50(s, 0.29H), 6.97(d, *J*=8.4Hz, 0.73H), 7.06(d, *J*=8.4Hz, 0.33H), 7.43(m, 3H), 7.62(m, 2H), 7.75(s, 1H); ^{13}C NMR(100MHz, $CDCl_3$): δ 20.47, 45.19, 47.37, 78.38, 80.20, 112.67, 113.15, 118.05, 118.15, 118.29, 118.36, 123.79, 124.31, 127.46, 127.75, 128.55, 132.28, 132.36, 132.83, 132.85, 133.97, 134.35, 138.80, 139.03, 158.66, 159.03, 182.12, 182.41; EI-MS (70ev): *m/z*(%) 342(M^+, 41), 262(8), 134(100), 127(10), 106(20), 71(27), 51(11). Anal. Calcd. For $C_{17}H_{12}BrNO_2$: C, 59.65; H, 3.54; N, 4.09. Found: C, 60.02; H, 3.58; N, 4.14.

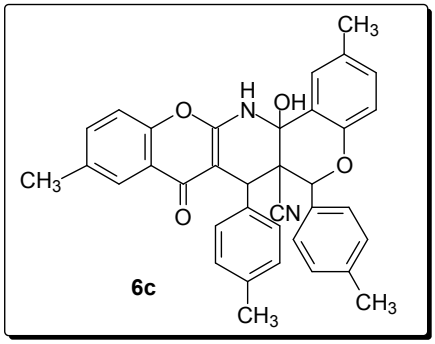
mixture of **4f/4f'**



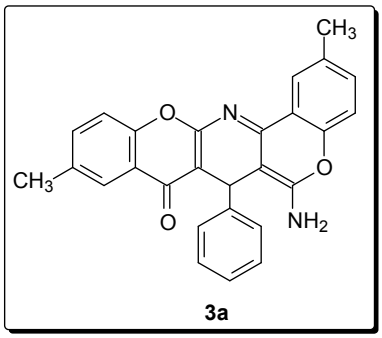
6-Methyl-4-oxo-2-(3-nitrophenyl)-3,4-dihydro-2H-chromene-3-carbonitrile 4f/4f': 74% all yield, white solid; υ 3383, 2892, 2259, 1703, 1615, 1531, 1489, 1351, 1223, 734 cm^{-1}; ^1H NMR(400MHz, $CDCl_3$): δ 2.37(s, 3H), 3.88(d, *J*=2.8Hz, 0.26H), 4.16(d, *J*=12.4Hz, 0.67H), 5.61(d, *J*=12.4Hz, 0.71H), 5.68(d, *J*=2.4Hz, 0.29H), 7.01(d, *J*=8.4Hz, 0.71H), 7.12(d, *J*=8.4Hz, 0.29H), 7.46(m, 1H), 7.71(m, 1H), 7.49(m, 1H), 7.91(d, *J*=7.6Hz, 0.72H), 7.98(d, *J*=7.6Hz, 0.30H), 8.34(m, 1H), 8.46(s, 0.29H), 8.53(s, 0.70H); ^{13}C NMR (100MHz, $CDCl_3$): δ 20.49, 44.93, 47.30, 77.75, 79.56, 112.30, 112.93, 118.05, 118.14, 118.30, 118.33, 121.33, 121.94, 124.49, 124.94, 127.53, 127.85, 130.26, 130.35, 131.91, 133.15, 133.24, 137.01, 137.40, 138.97, 139.21, 148.54, 148.65, 158.31, 158.59, 181.45, 181.76; EI-MS(70ev): *m/z* (%) 307(M^+-1, 46), 261(11), 186 (19), 134(100), 117(11), 106(17), 77(28). Anal. Calcd. for $C_{17}H_{12}N_2O_4$: C, 66.21; H, 3.92; N, 9.09. Found: C, 66.34; H, 3.96; N, 9.13.



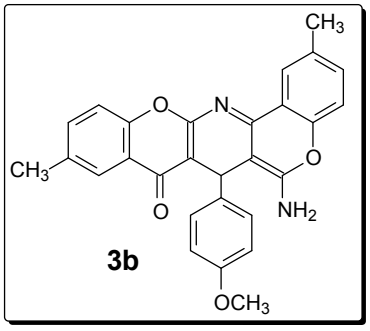
Intermediate 6b: 51% yield, white solid; m.p. 208-209°C; FT-IR (KBr): υ 3384, 3178, 2932, 1607, 1512, 1240, 1178, 1030, 836 cm^{-1}; ^1H NMR(400MHz, DMSO-d_6): δ 2.30(s, 3H), 2.37(s, 3H), 3.66(s, 3H), 3.83(s, 3H), 3.87(s, 1H), 5.13(s, 1H), 6.66(d, *J*=8.4Hz, 2H), 6.82(d, *J*=8.0Hz, 1H), 6.93(d, *J*=8.8Hz, 2H), 7.03(d, *J*=8.4Hz, 2H), 7.11(m, 2H), 7.34(d, *J*=8.4Hz, 1H), 4.46(m,3H), 7.53(s, 1H), 7.62(s, 1H), 9.53(s, 1H); ^{13}C NMR(100MHz, DMSO-d_6): δ 20.37, 20.49, 38.39, 49.62, 54.83, 55.16, 78.95, 79.82, 92.11, 112.10, 113.73, 115.87, 116.57, 117.42, 122.19, 124.42, 124.92, 127.26. 127.49, 129.30, 129.66, 130.65, 131.09, 131.14, 133.23, 133.81, 149.81, 151.49, 156.45, 157.71, 160.00, 171.95; EI-MS (70ev): *m/z* (%) 568(M-H_2O, 37), 461(M-H_2O-$CH_3C_6H_4$, 35), 292(100), 159(22), 134(49), 77(17); APCI-MS (H^+ source): *m/z* 587(M^+ +1). Anal. Calcd. for $C_{36}H_{30}N_2O_6$: C, 73.69; H, 5.16; N, 4.78. Found: C, 73.77; H, 5.20; N, 4.73.



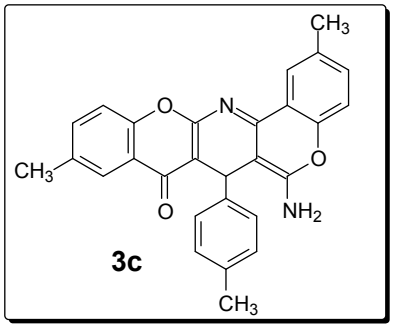
Intermediate 6c: 42% yield, white solid; m.p. 177~178°C; FT-IR (KBr): υ 3419, 2923, 2854, 1608, 1529, 1238, 1058, 807 cm^{-1}; ^1H NMR(400MHz, DMSO-d_6): δ 2.18(s, 3H), 2.30(s, 3H), 2.37(s, 3H), 2.39(s, 3H), 3.86(s, 1H), 5.14(s, 1H), 6.82(d, *J*=8.4Hz, 1H), 6.88(d, *J*=8.4Hz, 4H), 7.11(m, 1.68H), 7.28(d, *J*=8.0Hz, 2H), 7.35(d, *J*=8.4Hz, 1H), 7.40(d, *J*=7.6Hz, 2H), 7.48(m, 2H), 7.61(s, 1H), 9.55(s, 0.63H); ^{13}C NMR(100MHz, DMSO-d_6): δ 20.36, 20.47, 20.53, 20.85, 38.65, 49.41, 79.20, 79.75, 91.95, 115.86. 116.59, 117.27, 122.16, 124.40, 124.88, 127.30, 127.48, 127.89, 128.97, 129.73, 130.04, 130.68, 132.32, 133.27, 133.85, 135.20, 136.06, 138.92, 149.75, 151.51, 156.41, 171.94; EI-MS (70ev): *m/z* (%) 554(M^+, 6), 536(M-H_2O, 57), 445(M-H_2O-$CH_3OC_6H_4$, 100), 276(8), 135(8), 91(7), 77(3). Anal. Calcd. for $C_{36}H_{30}N_2O_4$: C, 77.94; H, 5.45; N, 5.05. Found: C, 78.11; H, 5.41, N, 5.03.



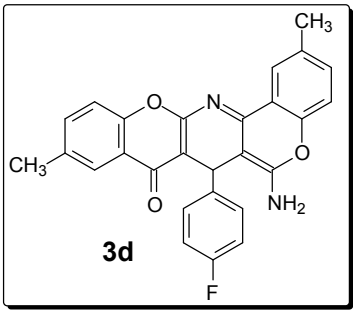
6-Amino-2,10-dimethyl-7-phenyl-7H-5,13-dioxa-14-aza-benzo[a] naphthacen-8-one 3a: 72% yield, yellow solid, m.p. 283-284 °C; FT-IR(KBr): υ 3342, 1660, 1616, 1548, 1385, 813, 732 cm^{-1}; ^1H NMR(400MHz, DMSO-d_6): δ 2.36(s, 3H), 2.45(s, 3H), 5.48(s, 1H), 7.80(t, *J*=7.2Hz, 1H), 7.19(t, *J*=7.2Hz, 2H), 7.32(d, *J*=8.4Hz, 1H), 7.39~7.50(m, 5H), 7.68(s, 1H), 8.13(s, 1H), 8.39(bs, NH_2); ^{13}C NMR(100MHz, DMSO-d_6): δ 20.37, 20.46, 34.53, 92.43, 101.05, 116.07, 117.20, 119.36, 123.25, 124.05, 124.69, 126.21, 127.29, 127.85, 133.15, 133.29, 134.68, 145.87, 149.31, 152.09, 156.82, 160.94, 164.28, 174.03; EI-MS(70ev): *m/z*(%) 420(M^+, 35), 343(M^+ -C_6H_5, 100), 111(18), 97(33), 83(34), 69(39), 57(68). Anal. Calcd. for $C_{27}H_{20}N_2O_3$: C, 77.11; H, 4.80; N, 6.67. Found: C, 77.04; H, 4.75; N, 6.71.



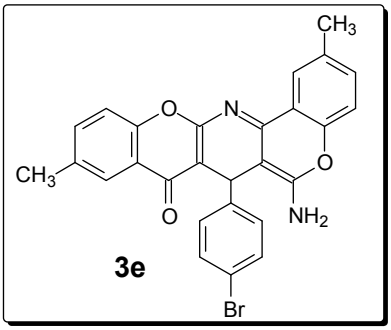
6-Amino-2,10-dimethyl-7-(4-methyoxyphenyl)-7H-5,13-dioxa-14-a za-benzo[a]naphthacen-8-one 3b: 85% yield, yellow solid, m.p. 291-292 °C; FT-IR(KBr): υ 3399, 1637, 1616, 1558, 1497, 1382, 811 cm^{-1}; ^1H NMR(400MHz, DMSO-d_6): δ 2.34(s, 3H), 2.43(s, 3H), 3.61(s, 3H), 5.40(s, 1H), 6.71(d, *J*=8.0Hz, 2H), 7.28(d, *J*=7.2Hz, 3H), 7.37~7.47(m, 3H), 7.66(s, 1H),8.11(s, 1H), 8.31(bs, NH_2); ^{13}C NMR (100MHz, DMSO-d_6): δ 20.38, 20.47, 33.72, 54.86, 92.79, 101.34, 113.21, 116.03, 117.18, 119.45, 123.32, 124.05, 124.70, 128.32, 133.03, 133.07, 133.22, 134.62, 138.32, 149.30, 152.10, 156.57, 157.67, 160.96, 164.13, 174.03; EI-MS(70ev): *m/z*(%) 450(M^+, 45), 343(M^+ -$CH_3OC_6H_4$, 100), 225(7). Anal. Calcd. for $C_{28}H_{22}N_2O_4$: C, 74.64; H, 4.92; N, 6.22. Found: C, 74.71; H, 4.95; N, 6.17.



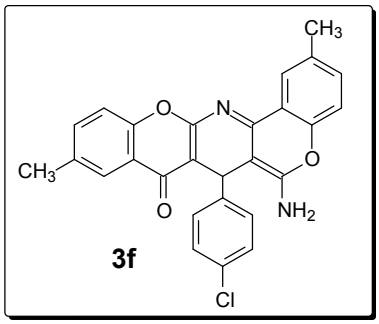
6-Amino-2,10-dimethyl-7-(4-methylphenyl)-7H-5,13-dioxa-14-aza-benzo[a]naphthacen-8-one 3c: 78% yield, yellow solid, m.p. 231-233 °C; FT-IR(KBr): υ 3400, 1616, 1556, 1496, 1384, 1263, 813 cm^{-1}; ^1H NMR(400MHz, DMSO-d_6): δ 2.15(s, 3H), 2.36(s, 3H), 2.45(s, 3H), 5.42(s, 1H), 6.96(d, *J*=8.0Hz, 2H), 7.30(dd, *J*=8.0, 14.4Hz, 3H), 7.39~7.50(m, 3H), 7.67(s, 1H), 8.12(s, 1H), 8.36(bs, NH_2); ^{13}C NMR (100MHz, DMSO-d_6): δ 20.38, 20.47, 20.51, 34.16, 92.60, 101.18, 116.06, 117.19, 119.40, 123.28, 124.04, 124.69, 127.21, 128.38, 133.12, 133.36, 134.66, 135.22, 143.02, 149.30, 152.08, 156.69, 160.93, 164.22, 174.02; EI-MS(70ev): *m/z*(%) 434(M^+, 88), 343(M^+ -$CH_3C_6H_4$, 100), 217(14), 91(7). Anal. Calcd. for $C_{28}H_{22}N_2O_3$: C, 77.38; H, 5.11; N, 6.45. Found: C, 77.45; H, 5.13; N, 6.38.



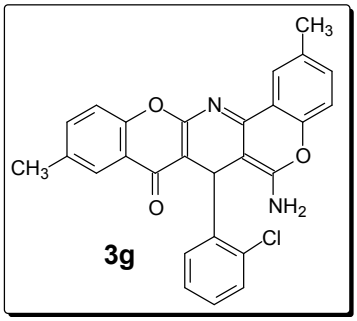
6-Amino-2,10-dimethyl-7-(4-fluorophenyl)-7H-5,13-dioxa-14-aza-benzo[a]naphthacen-8-one 3d: 58% yield, yellow solid, m.p. 294-295 ^{O}C; FT-IR(KBr): υ 3448, 1627, 1556, 1384, 1267, 701 cm^{-1}; ^{1}H NMR(400 MHz, DMSO-d_6): δ 2.33(s, 3H), 2.42(s, 3H), 5.47(s, 1H), 6.97(t, *J*=8.8Hz, 2H), 7.29(d, *J*=8.4Hz, 1H), 7.37~7.47(m, 5H), 7.65 (s, 1H), 8.09(s, 1H), 8.31(bs, NH_2); ^{13}C NMR(100 MHz, DMSO- d_6): δ 20.36, 20.45, 33.98, 92.37, 101.03, 114.42(d, $^{2}J_{CF}$=21 Hz), 116.06, 117.18, 119.40, 123.30, 124.08, 124.74, 129.12(d, $^{3}J_{CF}$=8 Hz), 133.15, 133.18, 133.30, 134.76, 142.05, 149.33, 152.11, 156.81, 159.59, 161.03, 163.04(d, $^{1}J_{CF}$=210 Hz), 174.06; EI-MS(70ev): *m/z*(%) 438(M^+, 42), 343(M^+ -FC_6H_4, 100), 95(9), 69(6), 55(12). Anal. Calcd. for $C_{27}H_{19}FN_2O_3$: C, 73.95; H, 4.37; , 6.39. Found: C, 74.08; H, 4.33; N, 6.44.



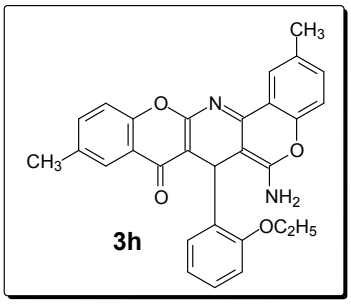
6-Amino-2,10-dimethyl-7-(4-bromophenyl)-7H-5,13-dioxa-14-aza-benzo[a]naphthacen-8-one 3e: 71% yield, yellow solid, m.p. 277-278 ^{O}C; FT-IR(KBr): υ 3420, 1644, 1555, 1422, 1385, 1266, 815 cm^{-1}; ^{1}H NMR(400MHz, DMSO-d_6): δ 2.36(s, 3H), 2.45(s, 3H), 5.49(s, 1H), 7.31(d, *J*=8.4Hz, 1H), 7.36~7.46(m, 6H), 7.49(d, *J*=8.0Hz, 1H), 7.68(s, 1H), 8.13(s, 1H), 8.34(bs, NH_2); ^{13}C NMR (100MHz, DMSO-d_6): δ 20.38, 20.47, 34.20, 91.92, 100.63, 116.12, 117.24, 119.30, 119.36, 123.18, 124.05, 124.73, 124.86, 129.59, 130.73, 133.26, 133.41, 134.76, 145.04,149.36, 152.10, 156.90, 161.09, 164.26, 174.03; EI-MS(70ev): *m/z*(%) 498(M^+-1, 20), 343(M^+-1-BrC_6H_4, 100), 155(10), 91(12), 76(17). Anal. Calcd. for $C_{27}H_{19}BrN_2O_3$: C, 64.92; H, 3.84; N, 5.61. Found: C, 65.11; H, 3.88; N, 5.57.



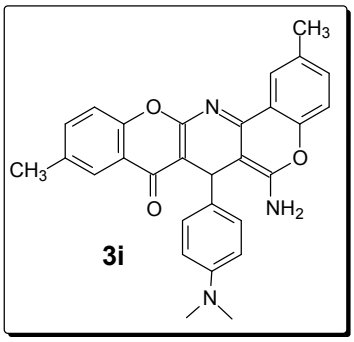
6-Amino-2,10-dimethyl-7-(4-chlorophenyl)-7H-5,13-dioxa-14-aza-benzo[a]naphthacen-8-one 3f: 68% yield, yellow solid, m.p. 298-299 ^{O}C; FT-IR(KBr): υ 3428, 1644, 1556, 1421, 1266, 816 cm^{-1}; ^{1}H NMR(400MHz, DMSO-d_6): δ 2.35(s, 3H), 2.44(s, 3H), 5.49(s, 1H), 7.23(d, *J*=8.4Hz, 2H), 7.31(d, J=8.4Hz,1H), 7.38~7.43(m, 4H), 7.48(d, *J*=8.8Hz, 1H) 7.67 (s, 1H), 8.12 (s, 1H), 8.38(bs, NH_2); ^{13}C NMR(100MHz, DMSO-d_6): δ 20.38, 20.47, 34.13, 92.04, 100.70, 116.11, 117.23, 119.30, 123.17, 124.04, 124.72, 127.81, 129.18, 130.83, 133.27, 133.41, 134.76, 144.63, 149.36, 152.09, 156.92, 161.08, 164.28, 174.05; EI-MS(70ev): *m/z*(%) 454(M^+, 60), 343(M^+ -ClC_6H_4, 100), 227(15), 157(8), 75(6). Anal. Calcd. for $C_{27}H_{19}ClN_2O_3$: C, 71.27; H, 4.21; N, 6.16. Found: C, 71.37; H, 4.24; N, 6.14.



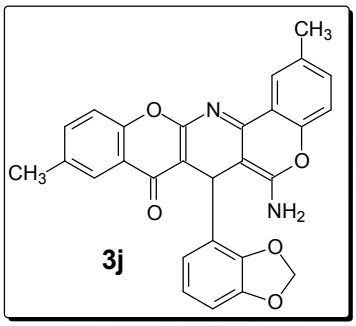
6-Amino-2,10-dimethyl-7-(2-chlorophenyl)-7H-5,13-dioxa-14-aza-benzo[a]naphthacen-8-one 3g: 47% yield, yellow solid, m.p. 263-265 ^{O}C; FT-IR(KBr): υ 3439, 1638, 1560, 1383, 1265, 817 cm^{-1}; ^{1}H NMR(400MHz, DMSO-d_6): δ2.32(s, 3H), 2.43(s, 3H), 5.69(s, 1H), 6.57(s, 1H), 7.11~7.29(m, 4H), 7.39~7.48(m, 3H), 7.57,(s, 1H), 7.75(s, *J*=8.0, 1H), 8.38(bs, NH_2); ^{13}C NMR(100MHz, DMSO-d_6): δ 20.89, 20.98, 36.45, 91.49, 99.76, 107.20, 116.55, 117.76, 119.65, 123.54, 124.40, 125.35, 127.00, 128.60, 130.20, 132.57, 133.28, 133.81, 133.91, 135.27, 141.69, 149.79, 152.61, 157.98, 161.08, 165.32, 174.55; EI-MS(70ev): *m/z*(%) 454(M^+, 18), 419(M^+ -Cl, 27), 343(M^+ -ClC_6H_4, 100), 111(22), 91(23), 75(35), 55(46). Anal. Calcd. for $C_{27}H_{19}ClN_2O_3$: C, 71.27; H, 4.21; N, 6.16. Found: C, 71.22; H, 4.17; N, 6.19.



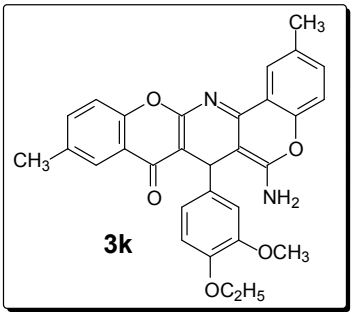
6-Amino-2,10-dimethyl-7-(2-ethyoxyphenyl)-7H-5,13-dioxa-14-aza-benzo[a]naphthacen-8-one 3h: 61% yield, yellow solid, m.p. >300 ^{O}C; FT-IR(KBr): υ 3330, 1647, 1560, 1498, 1382, 1264, 813 cm^{-1}; ^{1}H NMR(400MHz, DMSO-d_6): δ 1.33(t, *J*=6.8Hz, 3H), 2.34(s, 3H), 2.43(s, 3H), 4.02 (q, *J*=7.2Hz, 2H), 5.52(s, 1H), 6.81(t, *J*=7.2Hz, 1H), 6.93(d, *J*=8.4Hz, 1H),7.08(t, *J*=8.0Hz, 1H), 7.26(d, *J*=8.4Hz, 1H), 7.33(d, *J*=7.6Hz, 1H), 7.43~7.46(m, 3H), 7.59(s, 1H), 7.91(bs, NH_2); 8.11(s, 1H) ; ^{13}C NMR(100MHz, DMSO-d_6): δ14.57, 20.41, 20.49, 30.84, 64.56, 92.40, 100.36, 113.09, 116.02, 117.26, 119.48, 121.20, 123.26, 123.94, 124.72, 127.64, 130.14, 133.10, 133.21, 134.12, 134.60, 149.16, 152.22, 154.44, 156.65, 160.42, 164.99, 173.84; EI-MS(70ev): *m/z*(%) 464(M^+, 35), 417(12), 343(M^+ -$OC_2H_5C_6H_4$, 100), 129(11), 111(15), 97(22), 84(41), 69 (36), 55(61). Anal. Calcd. for $C_{29}H_{24}N_2O_4$: C, 74.97; H, 5.21; N, 6.03. Found: C, 75.05; H, 5.22; N, 6.06.



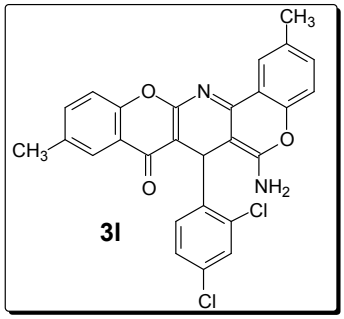
6-Amino-2,10-dimethyl-7-(4-N,N-dimethylaminophenyl)-7H-5,13-dioxa-14-aza-benzo[a]naphthacen-8-one 3i: 79% yield, yellow solid, m.p. 252-254 ^{O}C; FT-IR(KBr): υ 3431, 1637, 1557, 1498, 1384, 1265, 814 cm^{-1}; ^{1}H NMR(400MHz, DMSO-d_6): δ 2.34(s, 3H), 2.43(s, 3H), 2.73(s, 6H), 5.30(s, 1H), 6.50(d, *J*=8.8Hz, 2H), 7.17(d, *J*=8.8Hz, 2H), 7.28(d, *J*=8.4Hz, 1H), 7.36~7.46(m, 3H), 7.65(s, 1H), 8.10(s, 1H), 8.24(bs, NH_2); ^{13}C NMR(100MHz, DMSO-d_6): δ 20.88, 20.96, 34.02, 40.66, 93.62, 102.07, 112.53, 116.50, 117.65, 119.99, 123.85, 124.51, 125.15, 128.31, 133.43, 133.50, 133.65, 134.85, 135.07, 149.55, 149.76, 152.57, 156.83, 161.41, 164.55, 174.56; EI-MS(70ev): *m/z* (%) 463(M^+, 27), 343 [M^+ - $(CH_3)_2NC_6H_4$, 100], 120(39), 105(51), 91(51), 77(76), 65(23), 51(27). Anal. Calcd. for $C_{29}H_{25}N_3O_3$: C, 75.13; H, 5.44; N, 9.07. Found: C, 75.08; H, 5.41; N, 9.14.



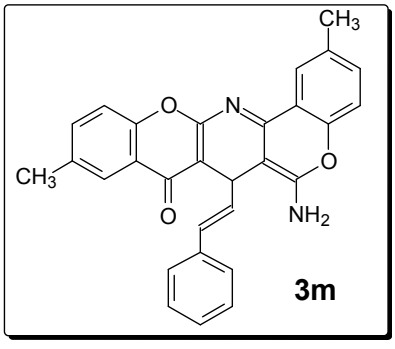
6-Amino-2,10-dimethyl-7-(2,3-oxymethyloxyphenyl)-7H-5,13-dioxa-14-aza-benzo[a]naphthacen-8-one 3j: 80% yield, yellow solid, m.p. 272-274 ^{O}C; FT-IR(KBr): υ 3394, 1651, 1556, 1498, 1381, 1268, 802 cm^{-1}; ^{1}H NMR(400MHz, DMSO-d_6): δ 2.30(s, 3H), 2.39(s, 3H), 5.34(s, 1H), 5.82(d, *J*=5.6Hz, 2H), 6.64(d, *J*=8.0Hz, 1H), 6.72(d, *J*=8.4Hz, 1H), 6.97(s, 1H), 7.26(d, *J*=8.4Hz, 1H), 7.33~7.44(m, 3H), 7.63(s, 1H), 8.06(s, 1H), 8.33(bs, NH_2); ^{13}C NMR(100MHz, DMSO-d_6): δ 20.38, 20.47, 34.27, 92.54, 100.55, 101.22, 107.72, 108.25, 116.09, 117.24, 119.38, 120.09, 123.25, 124.05, 124.73, 133.11, 133.17, 133.33, 134.78, 140.31, 145.61, 146.82, 149.23, 151.88, 156.70, 161.04, 164.18, 174.06; EI-MS(70ev): *m/z*(%) 464(M^+, 40), 343(M^+ -$OCH_2OC_6H_3$, 100). Anal. Calcd. for $C_{28}H_{20}N_2O_5$: C,72.39; H, 4.34; N, 6.03. Found: C, 72.44; H, 4.36; N, 5.98.



6-Amino-2,10-dimethyl-7-[(3-methyloxy-4-ethyloxy)phenyl]-7H-5,13-dioxa-14-aza-benzo[a]naphthacen-8-one 3k: 84% yield, yellow solid, m.p. 257-258 ^{O}C; FT-IR(KBr): υ 3368, 1615, 1550, 1496, 1374, 1263, 814cm^{-1}; ^{1}H NMR(400MHz, DMSO-d$_6$): δ 1.22(t, *J*=6.8Hz, 3H), 2.36(s, 3H), 2.45(s, 3H), 3.67(s, 3H), 3.86(q, *J*=6.4Hz, 2H), 5.39(s, 1H), 6.71(s, 2H), 7.23(s, 1H), 7.30(d, *J*= 8.0Hz, 1H), 7.73~7.74(m, 3H), 7.69(s, 1H), 8.12(s, 1H), 8.30(bs, NH_2); ^{13}C NMR(100MHz, DMSO-d$_6$): δ 14.74, 20.39, 20.49, 34.19, 55.47, 63.62, 92.79, 101.24, 112.53, 113.02, 116.05, 117.21, 119.13, 119.47, 123.33, 124.08, 124.70, 133.05, 133.10, 133.25, 134.64, 138.95, 146.42, 147.98, 149.29, 152.11, 156.49, 160.98, 164.16, 174.12; EI-MS(70ev): *m/z*(%) 494(M^+, 30), 343(M^+-$CH_3OC_2H_5OC_6H_3$, 100), 55(12). Anal. Calcd. for $C_{30}H_{26}N_2O_5$: C, 72.84; H, 5.30; N, 5.67. Found: C, 72.93; H, 5.33; N, 5.61.



6-Amino-2,10-dimethyl-7-(2,4-dichlorophenyl)-7H-5,13-dioxa-14-aza-benzo[a]naphthacen-8-one 3l: 49% yield, yellow solid, m.p. 224-225 ^{O}C; FT-IR(KBr): υ 3451, 1638, 1559, 1499, 1376, 1265, 815 cm^{-1}; ^{1}H NMR(400MHz, DMSO-d$_6$): δ 2.34(s, 3H), 2.44(s, 3H), 5.73(s, 1H), 7.27~7.33(m, 2H), 7.39~7.49(m, 4H), 7.59(s, 1H), 7.82(d, *J*=8.4, 1H), 7.82(bs, NH_2) 8.12(s, 1H); ^{13}C NMR(100 MHz, DMSO-d$_6$): δ 20.40, 20.47, 35.87, 90.49, 98.90, 116.04, 117.26, 119.17, 123.05, 123.92, 124.90, 126.47, 129.07, 131.70, 133.11, 133.33, 133.43, 134.16, 134.77, 140.08, 149.35, 152.14, 157.68, 160.72, 164.88, 173.99; EI-MS(70ev): *m/z*(%) 488(M^+-1, 11), 453(22), 368(13), 343(M^+-1-$Cl_2C_6H_3$, 85), 313(20), 239(22), 129(20), 111(23), 97(37), 83(46), 69(56), 57(100). Anal. Calcd. for $C_{27}H_{18}Cl_2N_2O_3$: C, 66.25; H, 3.71; N, 5.73. Found: C, 66.33; H, 3.74; N, 5.77.



6-Amino-2,10-dimethyl-7-styryl-7H-5,13-dioxa-14-aza-benzo [a]naphthacen-8-one 3m: 41% yield, yellow solid, m.p. 224-225 ^{O}C; FT-IR(KBr): υ 3430, 1640, 1556, 1497, 1386, 1266, 812 cm^{-1}; ^{1}H NMR(400MHz, DMSO-d$_6$): δ 2.39(s, 3H), 2.43(s, 3H), 5.09(d, *J*=5.2Hz, 1H), 6.15(d, *J*=16.0Hz, 1H), 6.38(dd, *J*=5.6, 15.4Hz, 1H), 7.13(t, *J*=6.8Hz, 1H), 7.20~7.27(m, 4H), 7.34(d, *J*=8.0Hz, 1H), 7.40~7.49(m, 3H), 7.76(s, 1H), 8.07(s, 1H), 8.46(bs, NH_2); ^{13}C NMR(100MHz, DMSO-d$_6$): δ 20.42, 20.48, 32.19, 89.98, 98.54, 116.11, 117.23, 119.39, 123.21, 124.10, 124.63, 126.00, 127.05, 128.04, 128.45, 129.86, 133.09, 133.19, 133.27, 134.54, 136.87, 149.46, 152.20, 157.05, 161.00, 164.55, 174.14; EI-MS(70ev): *m/z*(%) 446(M^+, 8), 343(M^+-CH=CHC_6H_5, 30), 154(14), 143(26), 125(20), 111(29), 97(47), 84(82), 69(68), 55(100). Anal. Calcd. for $C_{29}H_{22}N_2O_3$: C, 78.00; H, 4.97; N, 6.28. Found: C, 78.06; H, 5.01; N, 6.23.

IX. Crystallographic Data for 3b

(1) *X-ray data collection, structure determination and refinement*

A yellow single crystal (0.225 x 0.068 x 0.038mm) was attached to a glass fiber and mounted on a Bruker APEX-II quipped with a graphite momchromated Mo K_α (λ=0.71073Å) radiation source and a CCD detector. Absorption correction was applied by empirical absorption based on *ψ*-scans. The structure was solved by direct methods using SHELXS-97 and refined by full matrix least-squares on F^2 using SHELXL-97. Molecular graphics were produced using SHELXTL. Non-hydrogen atoms were refined anisotropically. H atoms of N_2 and O_5 were initially located in a difference Fourier map. The other H atoms were placed in geometrically idealised positions and constrained to ride on their parent atoms.

(2) Supplemental material

Crystallographic data, tables of atomic coordinates and thermal parameters, and full lists of bond lengths and angels have been deposited with the Cambridge Crystallographic data Center (CCDC), No. 661620. Copies of this information can be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk)

(3) Crystallographic Data

Table 1 Crystal data and structure refinement for compound **3b**

Identification code	xiamin
Empirical formula	$C_{28}H_{24}N_2O_5$
Formula weight	468.49
Temperature	295(2) K
Wavelength	0.71073 A
Crystal system, space group	Triclinic, Pī
Unit cell dimensions	a = 9.4429(13)Å α= 69.635(9)°. b = 11.1580(16)Å β= 78.846(9)°. c = 12.0491(19)Å γ= 87.735(9)°.
Volume	1167.2(3)Å^3
Z, Calculated density	2, 1.333 mg/m^3
Absorption coefficient	0.092 mm^{-1}
F(000)	492
Crystal size	0.225 x 0.068 x 0.038 mm
Theta range for data collection	1.84 to 25.60°.
Limiting indices	-11≤h≤11, -13≤k≤13, -14≤l≤14
Reflections collected / unique	7088 / 3580 [R(int) = 0.0443]
Completeness to theta = 25.60	81.3 %
Absorption correction	Empirical
Max. and min. transmission	0.9915 and 0.9819
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3580 / 1 / 335
Goodness-of-fit on F^2	0.991
Final R indices [I>2sigma(I)]	R1 = 0.0595, wR2 = 0.1129
R indices (all data)	R1 = 0.1552, wR2 = 0.1560
Largest diff. peak and hole	0.182 and -0.220 e.Å^{-3}

Table 2. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2x10^3$) for compound **3b**.

	x	y	z	U(eq)
O(1)	12016(3)	9288(3)	770(3)	52(1)
O(2)	10977(3)	6767(2)	4251(3)	56(1)
O(3)	10609(4)	14466(3)	1610(3)	85(1)
O(4)	5125(3)	9242(3)	3557(3)	51(1)
O(5)	3490(4)	1134(5)	1288(4)	82(1)
N(1)	8741(4)	7444(3)	4638(3)	49(1)
N(2)	6294(5)	10232(4)	1654(5)	57(1)
C(1)	16725(4)	6774(4)	1579(5)	78(2)
C(2)	15170(5)	6775(4)	2246(5)	55(1)
C(3)	14673(5)	5922(5)	3433(5)	66(2)
C(4)	13288(5)	5925(4)	4076(5)	67(2)
C(5)	12348(5)	6798(4)	3541(5)	49(1)
C(6)	12767(5)	7639(4)	2350(5)	43(1)
C(7)	14189(5)	7605(4)	1733(4)	53(1)
C(8)	11686(5)	8501(4)	1842(5)	43(1)
C(9)	10293(4)	8428(4)	2618(4)	36(1)
C(10)	9971(5)	7589(4)	3795(5)	47(1)
C(11)	7625(4)	8118(4)	4248(4)	41(1)
C(12)	7704(4)	8970(4)	3067(4)	38(1)
C(13)	9168(4)	9376(3)	2176(4)	37(1)
C(14)	9614(4)	10730(4)	2025(4)	38(1)
C(15)	9709(4)	11731(4)	938(4)	48(1)
C(16)	10046(4)	12959(4)	818(4)	54(1)
C(17)	10316(4)	13215(4)	1806(5)	53(1)
C(18)	10278(4)	12234(4)	2892(4)	49(1)
C(19)	9922(4)	11001(4)	2986(4)	43(1)
C(20)	10408(6)	14867(4)	2607(5)	99(2)
C(21)	6420(5)	9478(4)	2754(5)	44(1)
C(22)	5044(5)	8510(4)	4761(4)	46(1)
C(23)	6256(4)	7927(4)	5136(4)	44(1)
C(24)	6078(5)	7211(4)	6377(5)	51(1)
C(25)	4766(5)	7081(4)	7195(5)	52(1)
C(26)	4609(5)	6328(4)	8544(4)	76(2)
C(27)	3573(5)	7681(4)	6757(5)	59(1)
C(28)	3706(5)	8389(4)	5541(5)	59(1)

Table 3. Bond lengths (Å) for compound **3b**.

O(1)-C(8)	1.270(5)	C(16)-H(16)	0.9300
O(2)-C(10)	1.352(4)	C(17)-C(18)	1.378(5)
O(2)-C(5)	1.401(5)	C(18)-C(19)	1.390(5)
O(3)-C(17)	1.363(5)	C(18)-H(18)	0.9300
O(3)-C(20)	1.400(5)	C(19)-H(19)	0.9300
O(4)-C(21)	1.375(5)	C(20)-H(20A)	0.9600
O(4)-C(22)	1.383(5)	C(20)-H(20B)	0.9600
O(5)-H(51)	0.72(9)	C(20)-H(20C)	0.9600
O(5)-H(52)	1.00(6)	C(22)-C(23)	1.369(5)
N(1)-C(11)	1.334(4)	C(22)-C(28)	1.401(6)
N(1)-C(10)	1.360(5)	C(23)-C(24)	1.409(5)
N(2)-C(21)	1.325(6)	C(24)-C(25)	1.403(6)
N(2)-H(1)	0.860(10)	C(24)-H(24)	0.9300
N(2)-H(2)	0.86(5)	C(25)-C(27)	1.386(5)
C(1)-C(2)	1.531(6)	C(25)-C(26)	1.528(6)
C(1)-H(1A)	0.9600	C(26)-H(26A)	0.9600
C(1)-H(1B)	0.9600	C(26)-H(26B)	0.9600
C(1)-H(1C)	0.9600	C(26)-H(26C)	0.9600
C(2)-C(7)	1.361(5)	C(27)-C(28)	1.385(6)
C(2)-C(3)	1.414(6)	C(27)-H(27)	0.9300
C(3)-C(4)	1.386(6)	C(28)-H(28)	0.9300
C(3)-H(3)	0.9300		
C(4)-C(5)	1.366(5)		
C(4)-H(4)	0.9300		
C(5)-C(6)	1.403(6)		
C(6)-C(7)	1.410(5)		
C(6)-C(8)	1.448(5)		
C(7)-H(7)	0.9300		
C(8)-C(9)	1.445(6)		
C(9)-C(10)	1.383(5)		
C(9)-C(13)	1.507(5)		
C(11)-C(12)	1.399(5)		
C(11)-C(23)	1.480(5)		
C(12)-C(21)	1.381(5)		
C(12)-C(13)	1.551(5)		
C(13)-C(14)	1.524(5)		
C(13)-H(13)	0.9800		
C(14)-C(19)	1.378(5)		
C(14)-C(15)	1.384(5)		
C(15)-C(16)	1.371(5)		
C(15)-H(15)	0.9300		
C(16)-C(17)	1.386(5)		

Table 4. Bond angles (°) for compound **3b**

C(10)-O(2)-C(5)	119.8(4)
C(17)-O(3)-C(20)	118.2(4)
C(21)-O(4)-C(22)	121.3(3)
H(51)-O(5)-H(52)	123(7)
C(11)-N(1)-C(10)	115.8(4)
C(21)-N(2)-H(1)	115(3)
C(21)-N(2)-H(2)	121(3)
H(1)-N(2)-H(2)	123(5)
C(2)-C(1)-H(1A)	109.5
C(2)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	109.5
C(2)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5
C(7)-C(2)-C(3)	116.1(5)
C(7)-C(2)-C(1)	121.9(5)
C(3)-C(2)-C(1)	122.0(4)
C(4)-C(3)-C(2)	123.5(4)
C(4)-C(3)-H(3)	118.3
C(2)-C(3)-H(3)	118.3
C(5)-C(4)-C(3)	118.6(5)
C(5)-C(4)-H(4)	120.7
C(3)-C(4)-H(4)	120.7
C(4)-C(5)-O(2)	115.6(5)
C(4)-C(5)-C(6)	120.4(5)
O(2)-C(5)-C(6)	124.0(4)
C(5)-C(6)-C(7)	118.9(4)
C(5)-C(6)-C(8)	116.7(4)
C(7)-C(6)-C(8)	124.4(5)
C(2)-C(7)-C(6)	122.4(5)
C(2)-C(7)-H(7)	118.8
C(6)-C(7)-H(7)	118.8
O(1)-C(8)-C(9)	124.1(4)
O(1)-C(8)-C(6)	119.0(5)
C(9)-C(8)-C(6)	116.9(5)
C(10)-C(9)-C(8)	123.1(4)
C(10)-C(9)-C(13)	117.4(4)
C(8)-C(9)-C(13)	119.3(4)
O(2)-C(10)-N(1)	110.8(4)
O(2)-C(10)-C(9)	119.5(4)
N(1)-C(10)-C(9)	129.7(4)
N(1)-C(11)-C(12)	123.2(4)
N(1)-C(11)-C(23)	116.6(4)
C(12)-C(11)-C(23)	120.2(4)
C(21)-C(12)-C(11)	116.6(4)
C(21)-C(12)-C(13)	121.7(4)
C(11)-C(12)-C(13)	121.7(3)
C(9)-C(13)-C(14)	111.7(3)
C(9)-C(13)-C(12)	109.5(3)
C(14)-C(13)-C(12)	110.4(3)
C(9)-C(13)-H(13)	108.4
C(14)-C(13)-H(13)	108.4
C(12)-C(13)-H(13)	108.4
C(19)-C(14)-C(15)	117.5(4)
C(19)-C(14)-C(13)	120.5(4)

C(15)-C(14)-C(13)	122.0(4)
C(16)-C(15)-C(14)	121.9(4)
C(16)-C(15)-H(15)	119.0
C(14)-C(15)-H(15)	119.0
C(15)-C(16)-C(17)	119.5(4)
C(15)-C(16)-H(16)	120.2
C(17)-C(16)-H(16)	120.2
O(3)-C(17)-C(18)	124.5(4)
O(3)-C(17)-C(16)	115.4(4)
C(18)-C(17)-C(16)	120.1(4)
C(17)-C(18)-C(19)	118.9(4)
C(17)-C(18)-H(18)	120.5
C(19)-C(18)-H(18)	120.5
C(14)-C(19)-C(18)	122.0(4)
C(14)-C(19)-H(19)	119.0
C(18)-C(19)-H(19)	119.0
O(3)-C(20)-H(20A)	109.5
O(3)-C(20)-H(20B)	109.5
H(20A)-C(20)-H(20B)	109.5
O(3)-C(20)-H(20C)	109.5
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
N(2)-C(21)-O(4)	112.7(4)
N(2)-C(21)-C(12)	124.1(5)
O(4)-C(21)-C(12)	123.2(4)
C(23)-C(22)-O(4)	119.1(4)
C(23)-C(22)-C(28)	122.9(5)
O(4)-C(22)-C(28)	117.9(4)
C(22)-C(23)-C(24)	115.3(5)
C(22)-C(23)-C(11)	119.1(4)
C(24)-C(23)-C(11)	125.6(4)
C(25)-C(24)-C(23)	123.7(4)
C(25)-C(24)-H(24)	118.1
C(23)-C(24)-H(24)	118.1
C(27)-C(25)-C(24)	118.3(5)
C(27)-C(25)-C(26)	119.0(5)
C(24)-C(25)-C(26)	122.8(4)
C(25)-C(26)-H(26A)	109.5
C(25)-C(26)-H(26B)	109.5
H(26A)-C(26)-H(26B)	109.5
C(25)-C(26)-H(26C)	109.5
H(26A)-C(26)-H(26C)	109.5
H(26B)-C(26)-H(26C)	109.5
C(28)-C(27)-C(25)	119.6(5)
C(28)-C(27)-H(27)	120.2
C(25)-C(27)-H(27)	120.2
C(27)-C(28)-C(22)	120.2(4)
C(27)-C(28)-H(28)	119.9
C(22)-C(28)-H(28)	119.9

Table 5. Hydrogen coordinates ($x10^4$) and isotropic displacement parameters ($Å^2x10^3$) for compound **3b**.

	x	y	z	U(eq)
H(1A)	16789	7249	736	117
H(1B)	17000	5909	1685	117
H(1C)	17360	7162	1899	117
H(3)	15309	5327	3801	80
H(4)	13004	5345	4854	81
H(7)	14467	8170	947	63
H(13)	9049	9365	1389	44
H(15)	9539	11565	269	57
H(16)	10094	13617	79	65
H(18)	10488	12395	3551	59
H(19)	9890	10340	3720	52
H(20A)	9451	14621	3071	149
H(20B)	10533	15780	2335	149
H(20C)	11103	14474	3100	149
H(24)	6876	6801	6672	61
H(26A)	4692	6905	8963	115
H(26B)	5355	5712	8673	115
H(26C)	3682	5894	8843	115
H(27)	2687	7608	7277	71
H(28)	2906	8786	5241	70
H(1)	5430(20)	10450(40)	1560(40)	85(16)
H(2)	7020(50)	10380(50)	1060(50)	110(30)
H(51)	3130(90)	1690(80)	1350(90)	230(50)
H(52)	3020(60)	450(60)	1110(50)	140(20)

Table 6. Anisotropic displacement parameters (Åx10^3) for compound **3b**.

	U11	U22	U33	U23	U13	U12
O(1)	49(2)	68(2)	36(2)	-14(2)	-5(2)	2(2)
O(2)	49(2)	63(2)	46(2)	-7(2)	-12(2)	10(2)
O(3)	138(3)	52(2)	67(3)	-18(2)	-23(2)	-20(2)
O(4)	38(2)	69(2)	39(2)	-14(2)	-4(2)	1(2)
O(5)	59(2)	79(3)	108(4)	-25(3)	-31(2)	3(2)
N(1)	41(2)	62(2)	38(3)	-14(2)	-5(2)	9(2)
N(2)	46(3)	74(3)	49(3)	-16(3)	-19(3)	9(3)
C(1)	51(3)	95(4)	89(4)	-33(4)	-13(3)	18(3)
C(2)	42(3)	69(3)	54(4)	-26(3)	-6(3)	5(3)
C(3)	55(4)	77(4)	70(4)	-27(4)	-23(4)	25(3)
C(4)	60(3)	74(3)	59(4)	-11(3)	-18(3)	20(3)
C(5)	33(3)	57(3)	55(4)	-21(3)	-5(3)	2(3)
C(6)	39(3)	53(3)	40(3)	-17(3)	-11(3)	4(2)
C(7)	49(3)	61(3)	49(3)	-20(3)	-9(3)	5(2)
C(8)	48(3)	49(3)	40(3)	-20(3)	-14(3)	1(3)
C(9)	33(3)	38(3)	31(3)	-6(2)	1(3)	-2(2)
C(10)	48(3)	49(3)	46(4)	-17(3)	-15(3)	9(3)
C(11)	37(3)	49(3)	40(3)	-19(3)	-5(3)	-1(2)
C(12)	36(3)	52(3)	27(3)	-16(3)	-4(3)	-1(2)
C(13)	34(3)	46(3)	29(3)	-11(2)	-5(2)	0(2)
C(14)	36(2)	43(3)	34(3)	-10(3)	-9(2)	-1(2)
C(15)	50(3)	58(3)	36(3)	-18(3)	-7(3)	2(2)
C(16)	73(3)	47(3)	35(3)	-4(3)	-9(3)	-5(2)
C(17)	59(3)	45(3)	51(4)	-11(3)	-11(3)	-6(2)
C(18)	59(3)	51(3)	40(3)	-15(3)	-17(3)	-1(2)
C(19)	47(3)	47(3)	32(3)	-8(2)	-12(2)	1(2)
C(20)	153(5)	50(3)	116(6)	-33(4)	-71(5)	13(3)
C(21)	38(3)	53(3)	43(3)	-18(3)	-7(3)	-4(2)
C(22)	50(3)	54(3)	35(3)	-17(3)	-6(3)	-5(3)
C(23)	42(3)	48(3)	40(3)	-16(3)	-3(3)	-1(2)
C(24)	50(3)	53(3)	44(3)	-11(3)	-4(3)	-5(3)
C(25)	55(3)	53(3)	42(4)	-13(3)	4(3)	-11(3)
C(26)	85(4)	82(4)	46(4)	-12(3)	6(3)	-7(3)
C(27)	47(3)	70(3)	55(4)	-26(3)	9(3)	-3(3)
C(28)	44(3)	77(4)	51(4)	-22(3)	-2(3)	0(3)

X. 2D NMR Experiments for the intermediates *6b~c*

(1) NMR experimental parameters of HMQC data

pulse program for acquisition (PULPROG) = hmqcph

time domain size(TD) = 1024

number of scans (NS) = 140

number of dummy scans(DS) = 8

sweep width in Hz (SWH[Hz]) = 4807.69

acquisition time (AQ[s]) = 0.1065460

receiver gain (RG) = 203

dwell time (DW[µs]) =104.000

pre-scan-delay (DE[µs]) =6.00

J (XH) (CNST2) =145.0000000

d0 = 3u, d0[s] = 0.00000300

relaxation delay; 1-5*T1 (D1[s]) = 1.50000000

d2=1s/(cnst2*2), d2[s] = 0.00344828

delay for disk I/O (D11[s]) = 0.03000000

d13=3u, d13[s] = 0.00000300

1/(2*SW(X))= DW(X), IN0[s] = 0.00002613

ST1CNT= td1/(2), ST1CNT= 128

Channel f 1

Nuleus for channel 1 (NUC1) = 1H

f 1 channel: 90 degree high power pulse (P1[µs]) = 7.00

P2=p1*2, p2 [µs] = 14.00

f 1 channel: power level for pulse(default), PL1[dB] = -2.00

frequency of observe channel (SFO1[MHz]) = 400.1324024

Channel f 2

file name for cpd2 (CPDPRG2) = grap

nuleus for channel 2 (NUC2) = 13C

f 2 channel: 90 degree high power pulse (P3[µs]) = 15.00

f 2 channel: 90 degree pulse for decoupling sequence(PCPD2[µs]) = 65.00

f 2 channel: power level for pulse (default), PL2[dB] = -3.10

f 2 channel: power level for CPD/BB decoupling, PL12 [dB] = 9.64

frequency of observe channel (SFO2[MHz]) = 100.6223261

(2) NMR experimental parameters of HMBC data

pulse program for acquisition (PULPROG) = hmbclpndqf

time domain size(TD) = 4096

number of scans (NS) = 640

number of dummy scans(DS) = 16

sweep width in Hz (SWH[Hz]) = 4807.69

acquisition time (AQ[s]) = 0.4260340

receiver gain (RG) = 287

dwell time (DW[µs]) =104.000

pre-scan-delay (DE[µs]) =6.00

J (XH) (CNST2) =145.0000000

J(XH) long range (CNST13) = 8.0000000

d0 = 3u, d0[s] = 0.00000300

relaxation delay; 1-5*T1 (D1[s]) = 1.50000000

d2=1s/(cnst2*2), d2[s] = 0.00344828

d6= 1s/(cnst13*2), d6[s] = 0.06250000

1/(2*SW(X))= DW(X), IN0[s] = 0.00002613

Channel f 1

Nuleus for channel 1 (NUC1) = 1H

f 1 channel: 90 degree high power pulse (P1[µs]) = 7.00

P2=p1*2, p2 [µs] = 14.00

f 1 channel: power level for pulse(default), PL1[dB] = -2.00

frequency of observe channel (SFO1[MHz]) = 400.1324024

Channel f 2

nuleus for channel 2 (NUC2) = 13C

f 2 channel: 90 degree high power pulse (P3[µs]) = 15.00

f 2 channel: power level for pulse (default), PL2[dB] = -3.10

frequency of observe channel (SFO2[MHz]) = 100.6223261

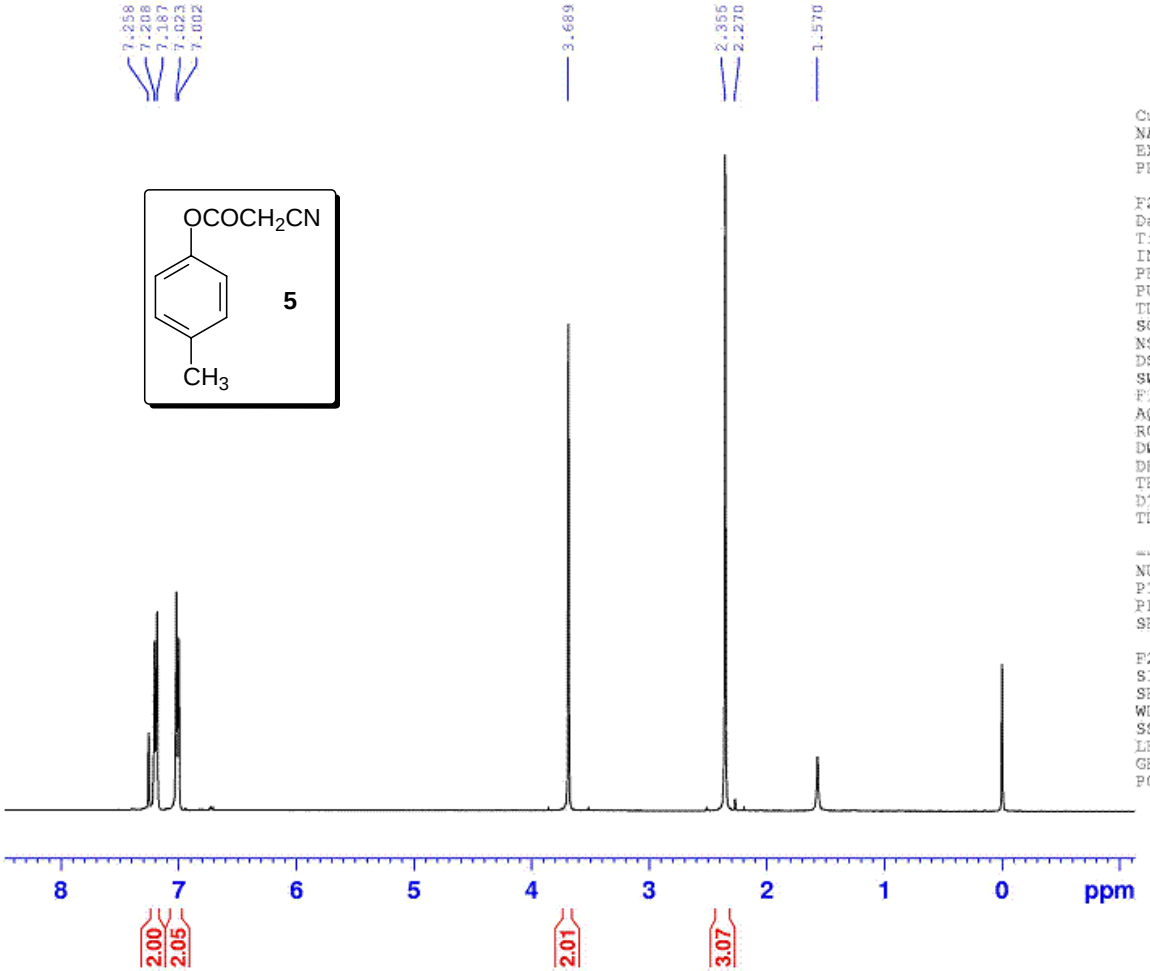
Base-catalyzed and Solvent-dependent Cascade Reaction in Regioselective Synthesis of Novel Fused Polycycles

Min Xia*, Guo-Feng Xiang, Bin Wu, Yi-Feng Han

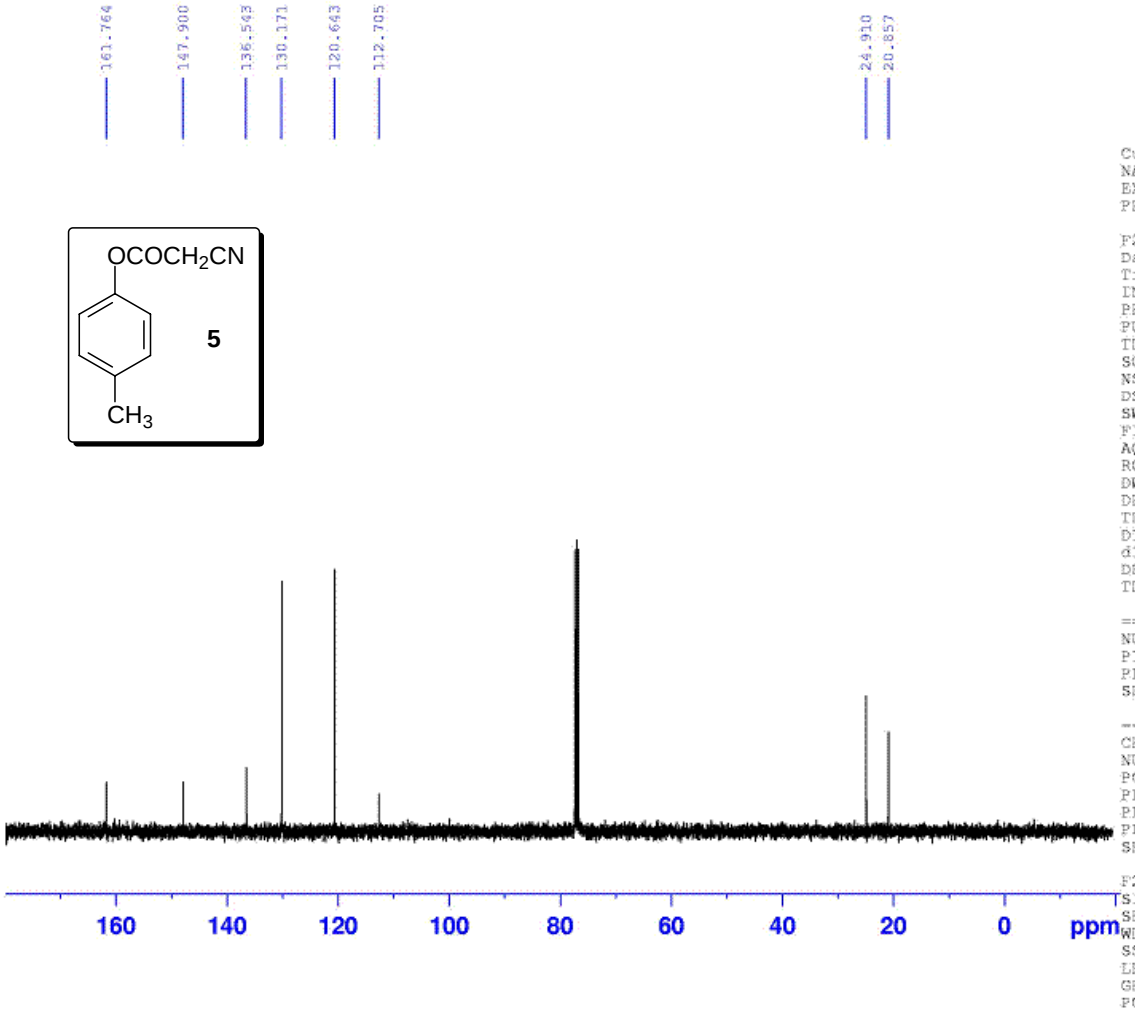
Department of Chemistry, Zhejiang Sci-Tech University, Hangzhou 310018, P. R. China

Phone/fax: +86(571)86843224; E-mail: xiamin@zstu.edu.cn

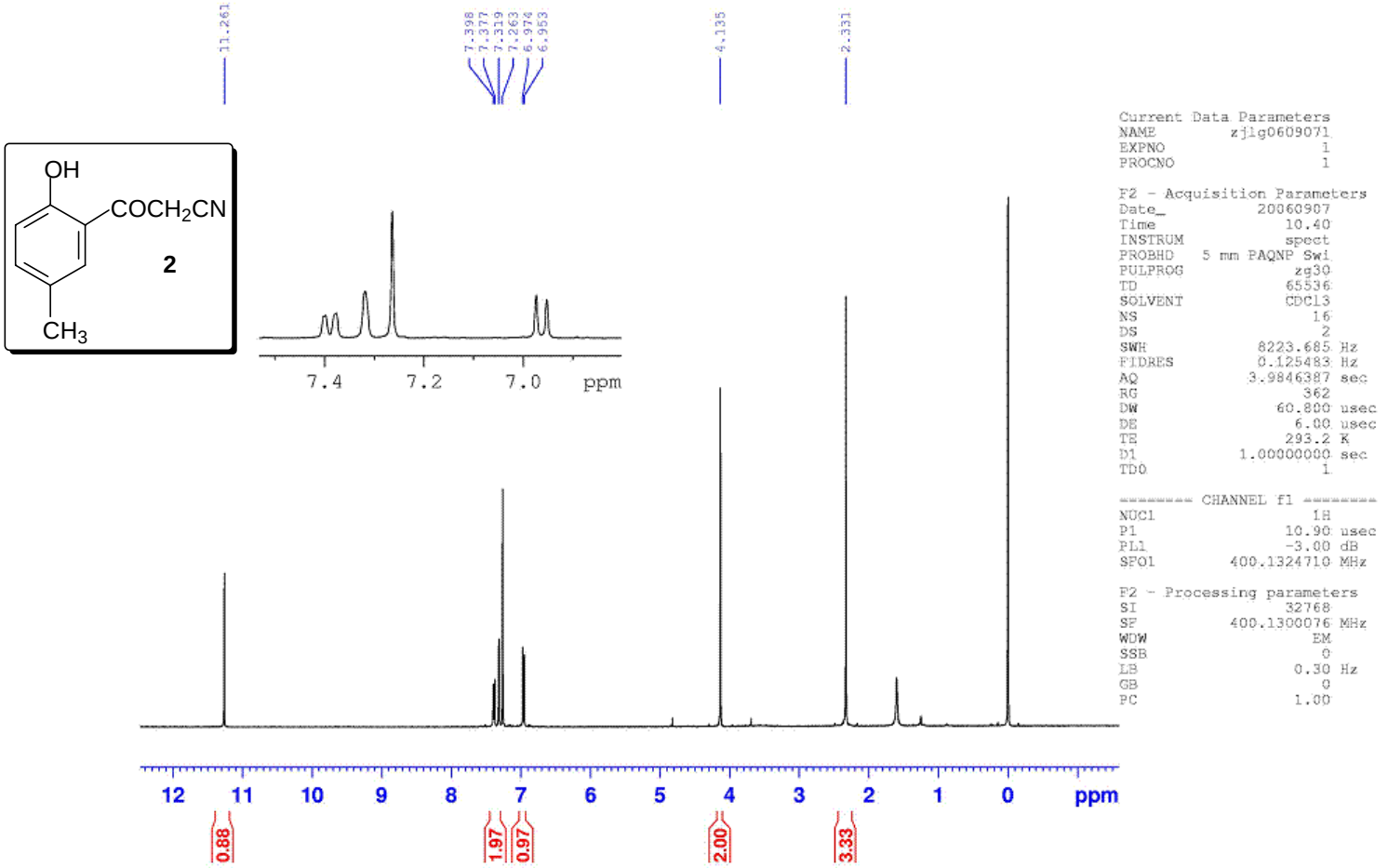
Contents:



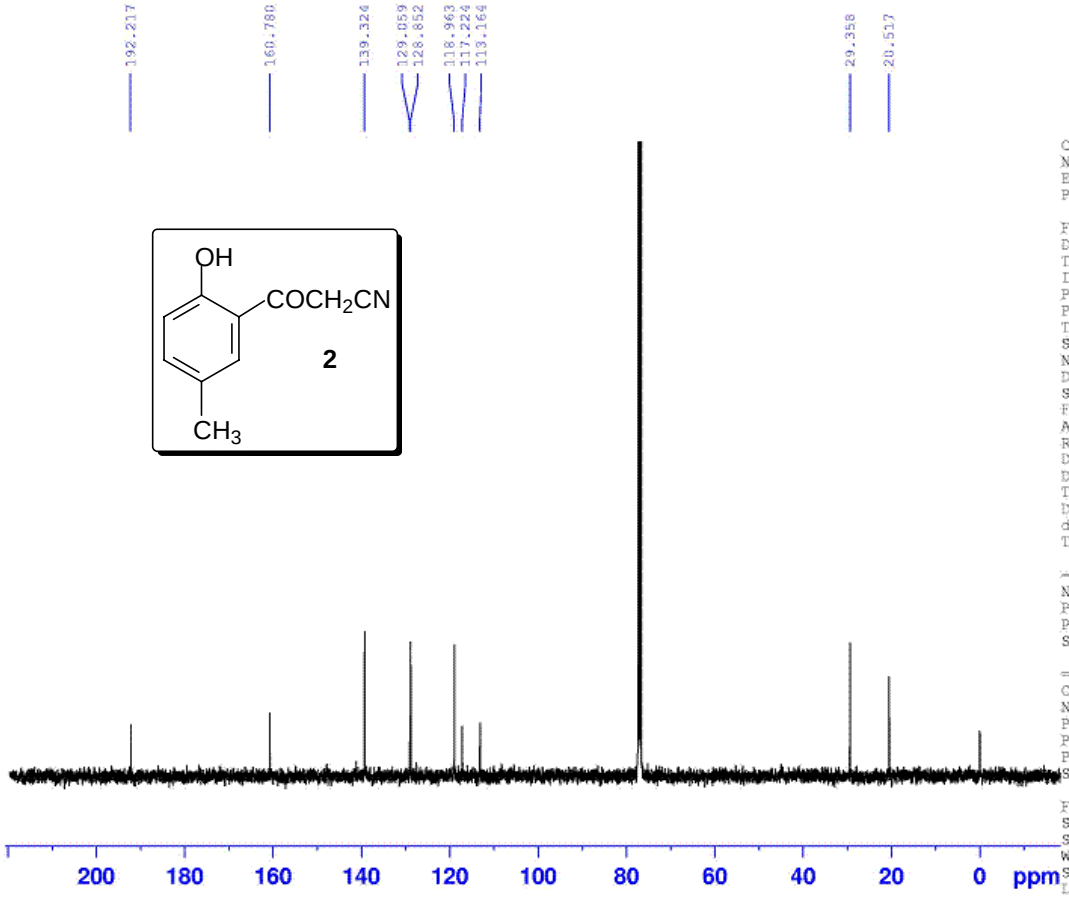

```
Current Data Parameters
NAME          zjlg0608291
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20060829
Time                14.29
INSTRUM             spect
PROBHD   5 mm PAQNP Swi
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    228
DW                 60.800 usec
DE                  6.00 usec
TE                297.6 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.90 usec
PL1                -3.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF           400.1300098 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```



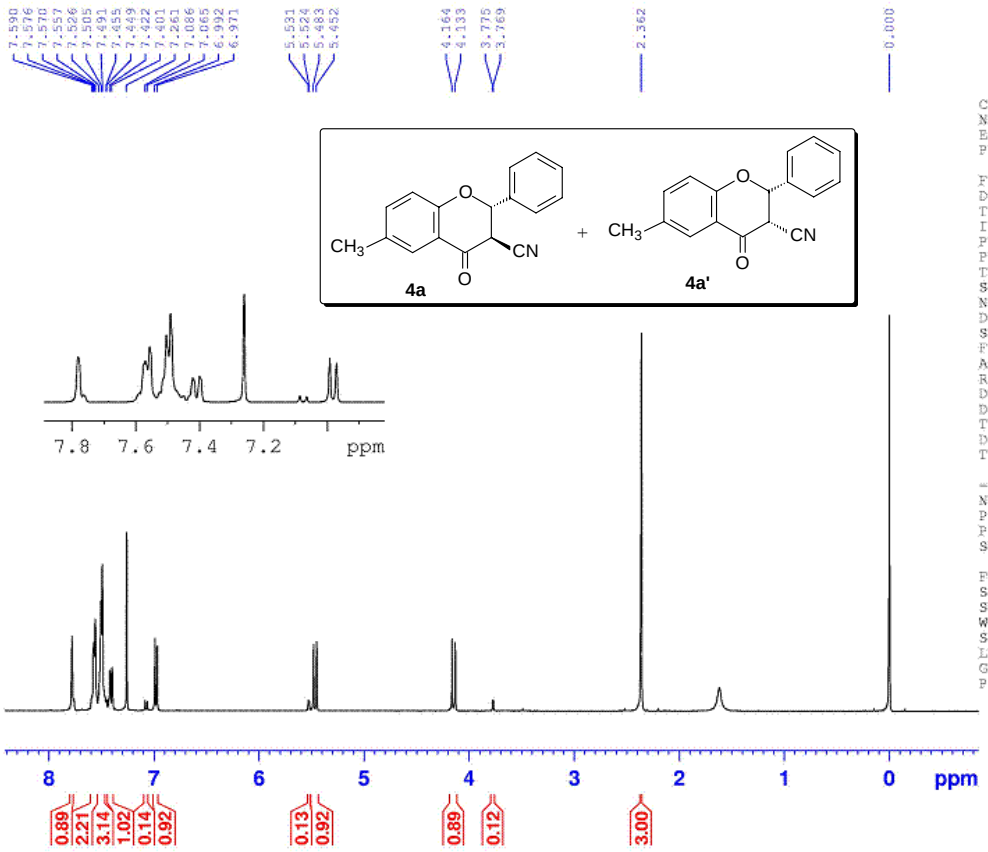

```
Current Data Parameters
NAME         zjlg0701244
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070126
Time               14.00
INSTRUM            spect
PROBHD   5 mm PAQNP Swi
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  478
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               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.00 usec
PL1                -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              13.48 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
```



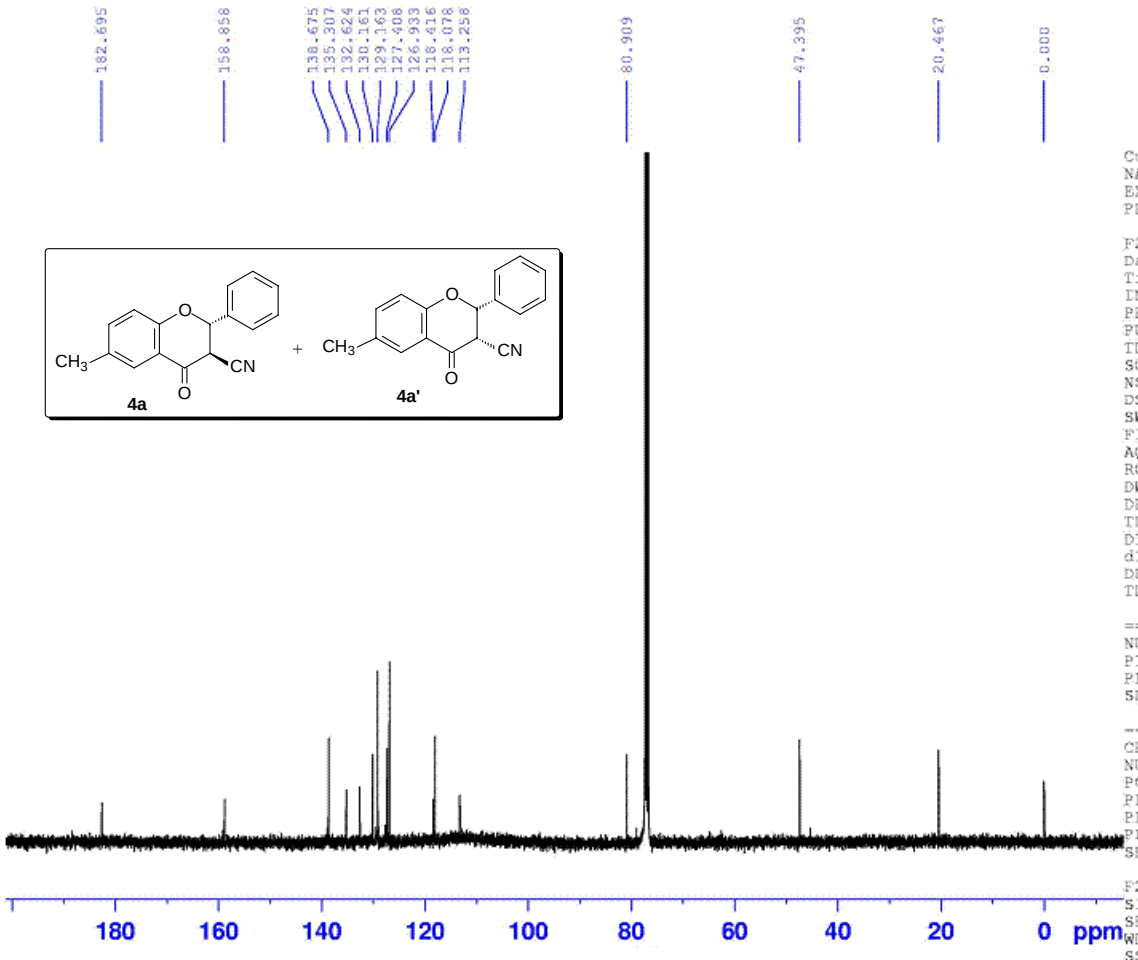

```
Current Data Parameters
NAME         zjlg0609071
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20060907
Time               10.40
INSTRUM            spect
PROBHD   5 mm PAQNP Swi
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   362
DW                60.800 usec
DE                 6.00 usec
TE                293.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.90 usec
PL1               -3.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300076 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



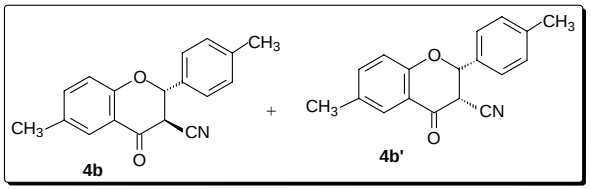

```
Current Data Parameters
NAME          zjlg0609081
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20060908
Time                 9.15
INSTRUM             spect
PROBHD   5 mm PAQNP Swi
PULPROG          zgdc30
TD                65536
SOLVENT             CDCl3
NS                    637
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                294.4 K
D1          2.00000000 sec
d11         0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.00 usec
PL1               -1.00 dB
SFO1        100.6228300 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              13.48 dB
SFO2        400.1316000 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127700 MHz
WDW                   EM
SSB                    0
LB                 2.00 Hz
GB                     0
PC                 1.40
```

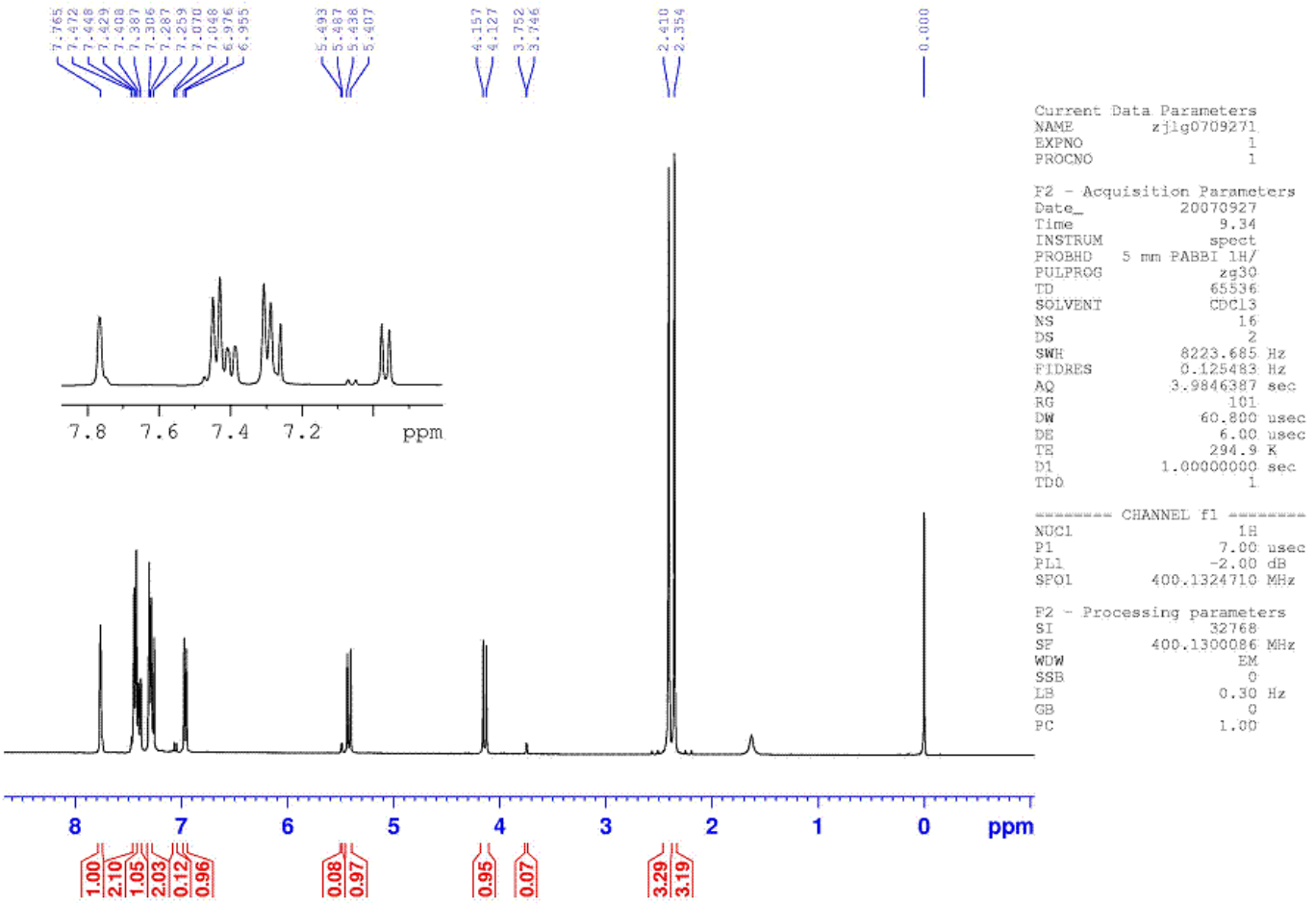



```
Current Data Parameters
NAME          zjlg0709261
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070926
Time                10.48
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   287
DW                60.800 usec
DE                 6.00 usec
TE                295.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.00 usec
PL1               -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300083 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

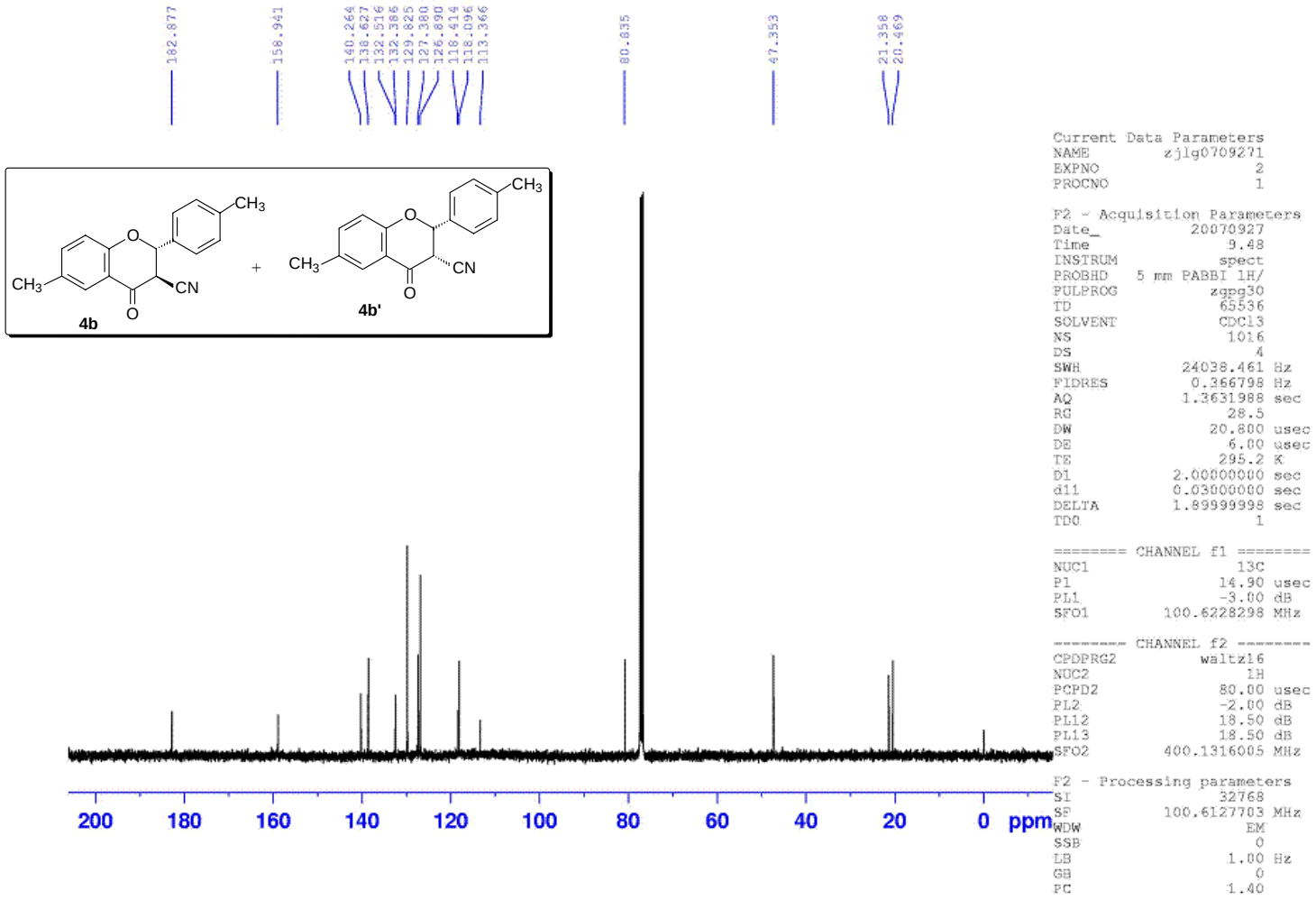



```
Current Data Parameters
NAME         zjlg0709261
EXPNO                 22
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070926
Time              11.01
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                3126
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                  36
DW               20.800 usec
DE                6.00 usec
TE               295.3 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                13C
P1                14.90 usec
PL1               -3.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              18.50 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127698 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

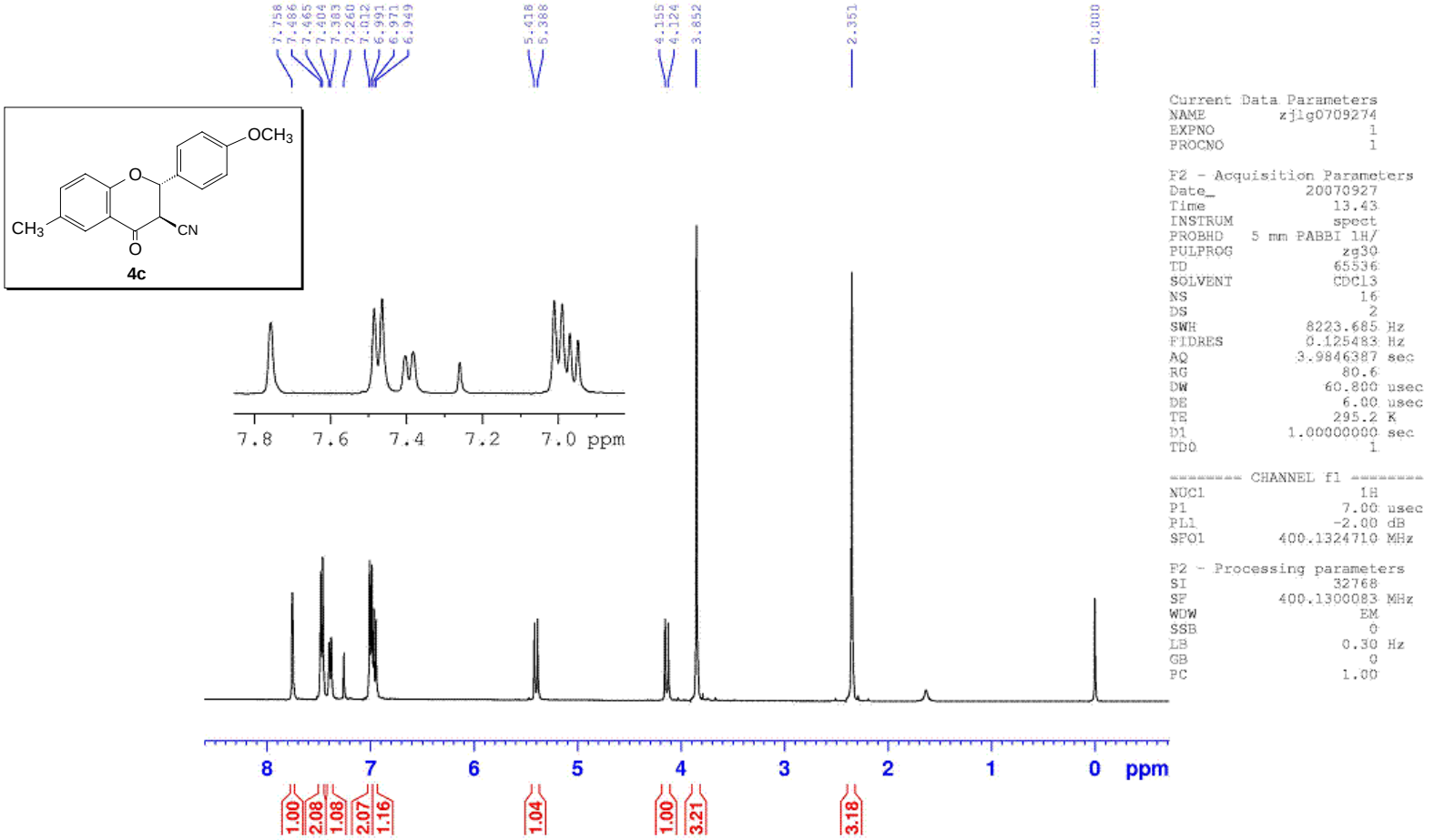




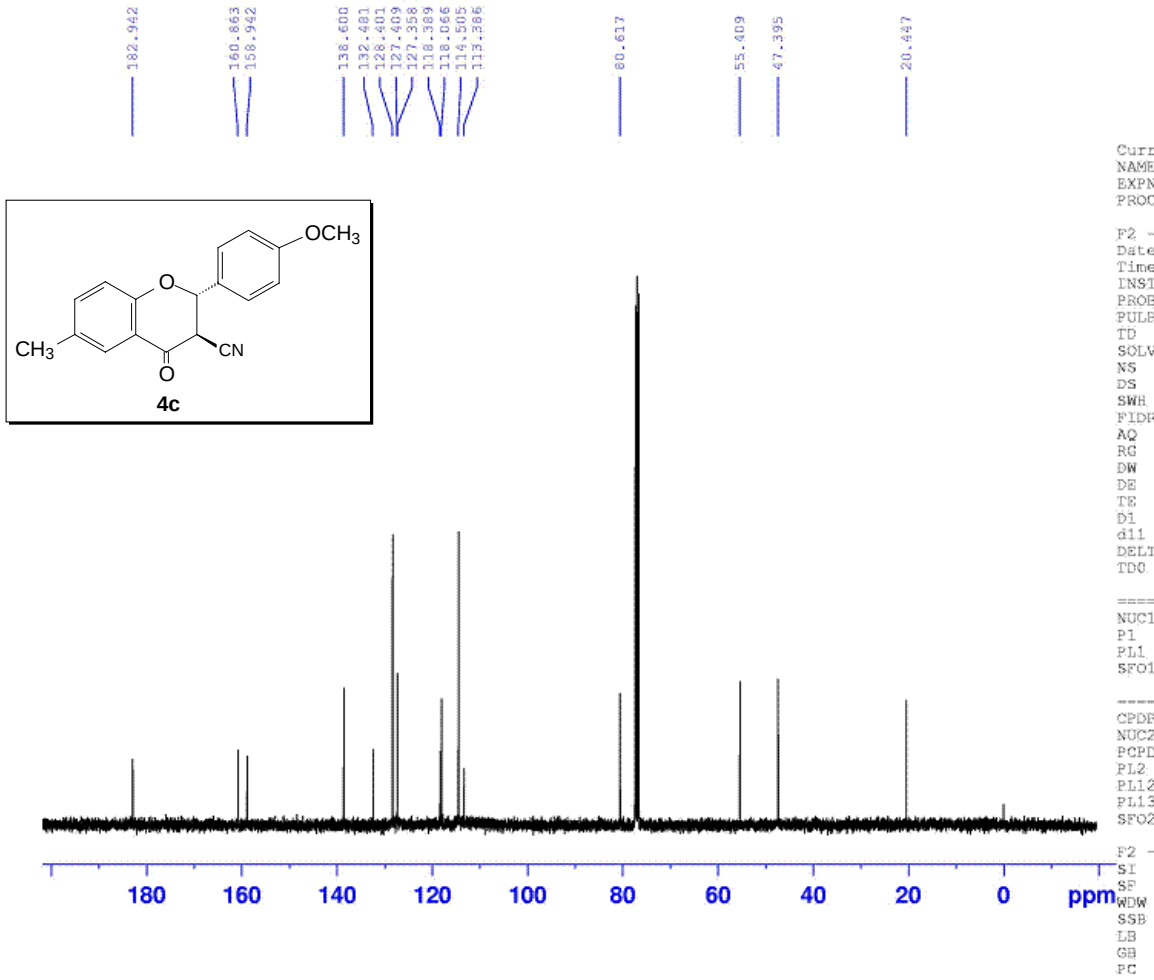

```
Current Data Parameters
NAME                  zjlg0709271
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20070927
Time                         9.34
INSTRUM                      spect
PROBHD    5 mm PABBI 1H/
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                      8223.685 Hz
FIDRES                   0.125483 Hz
AQ                       3.9846387 sec
RG                            101
DW                          60.800 usec
DE                           6.00 usec
TE                          294.9 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                          1H
P1                           7.00 usec
PL1                         -2.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                          32768
SF                400.1300086 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```



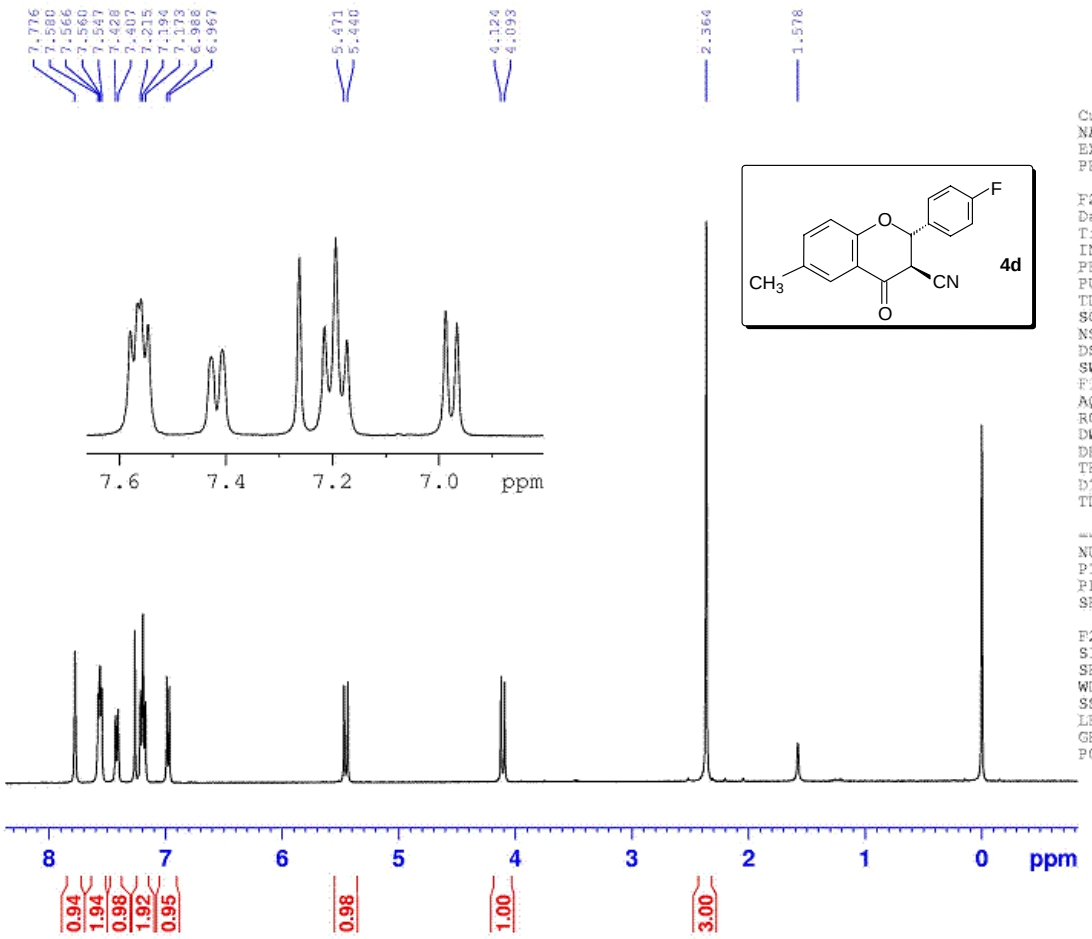

```
Current Data Parameters
NAME         zjlg0709271
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070927
Time                9.48
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1016
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                 28.5
DW               20.800 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                14.90 usec
PL1               -3.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              18.50 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127703 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



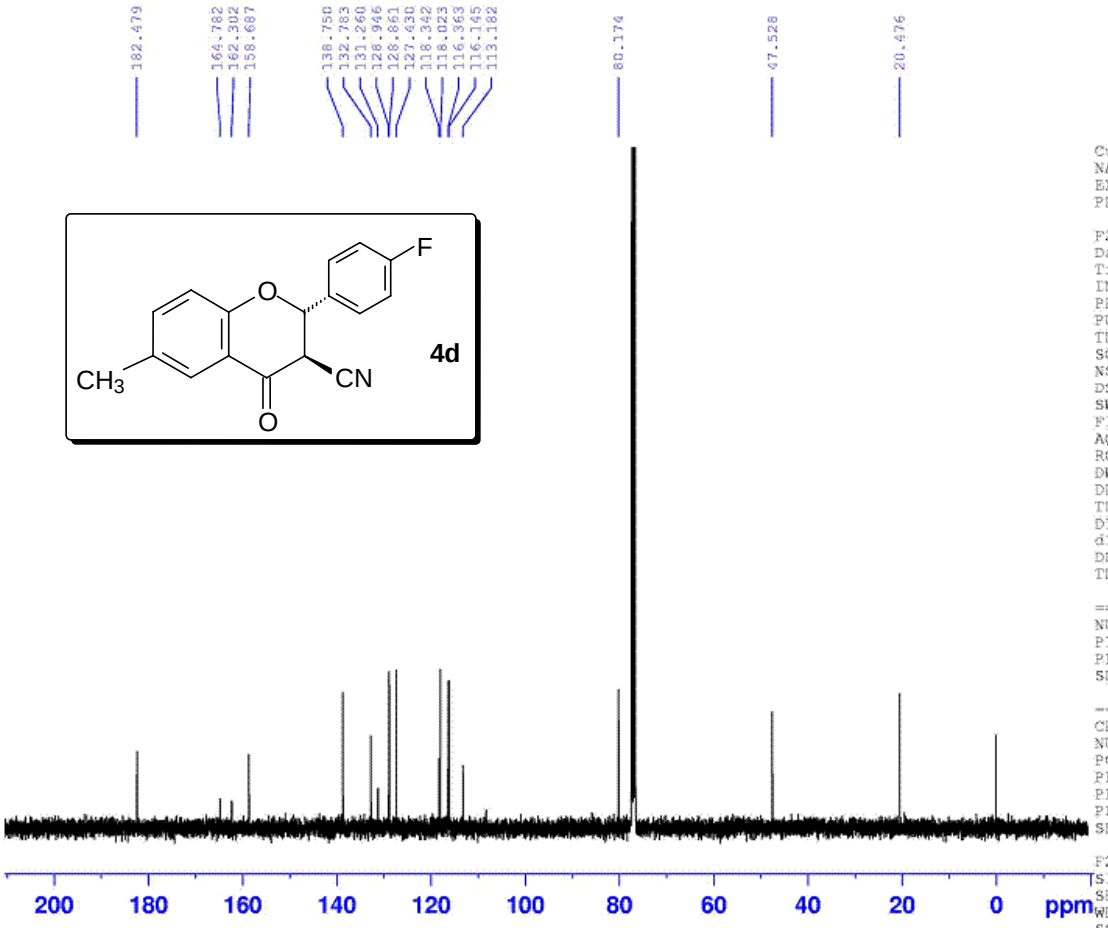

```
Current Data Parameters
NAME          zjlg0709274
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070927
Time                13.43
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ             3.9846387 sec
RG                  80.6
DW                60.800 usec
DE                 6.00 usec
TE                295.2 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.00 usec
PL1                -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300083 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



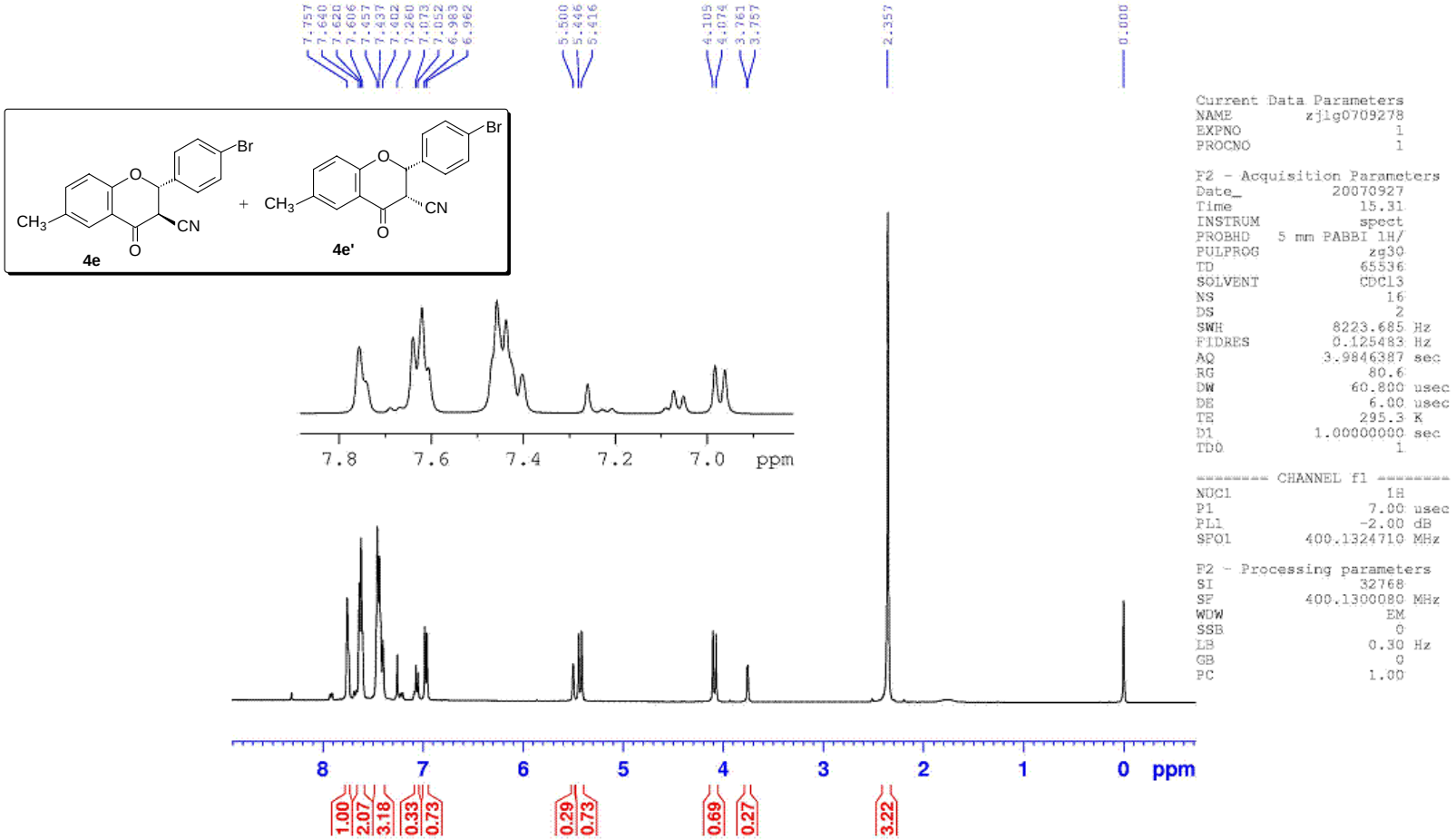

```
Current Data Parameters
NAME         zjlg0709274
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070927
Time               14.15
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1123
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                   36
DW               20.800 usec
DE                6.00 usec
TE               295.6 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                14.90 usec
PL1               -3.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              18.50 dB
PL13              18.50 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127706 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
```



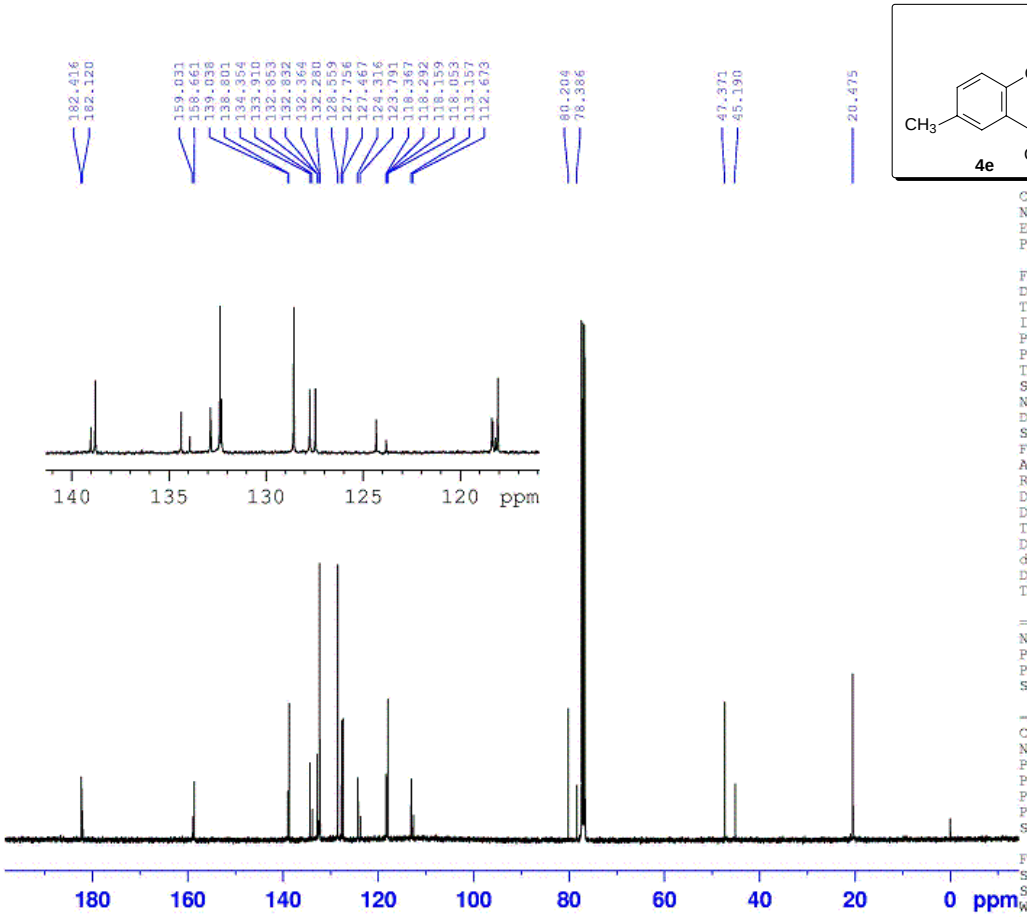

```
Current Data Parameters
NAME                  zjlg0704041
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20070404
Time                         9.52
INSTRUM                     spect
PROBHD    5 mm PAQNP Swi
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                      8223.685 Hz
FIDRES                   0.125483 Hz
AQ                       3.9846387 sec
RG                            362
DW                          60.800 usec
DE                           6.00 usec
TE                          292.3 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                          10.90 usec
PL1                          -3.00 dB
SFO1                 400.1324710 MHz

F2 - Processing parameters
SI                          32768
SF                   400.1300044 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```

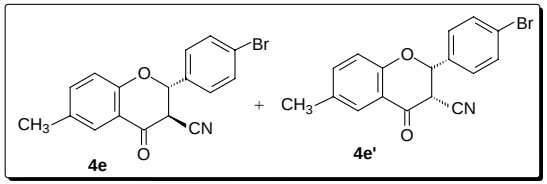



```
Current Data Parameters
NAME          zjlg0611302
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20061130
Time                11.48
INSTRUM             spect
PROBHD   5 mm PAQNP Swi
PULPROG           zgpg30
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                290.4 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.00 usec
PL1                 -1.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.48 dB
PL13               14.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127715 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

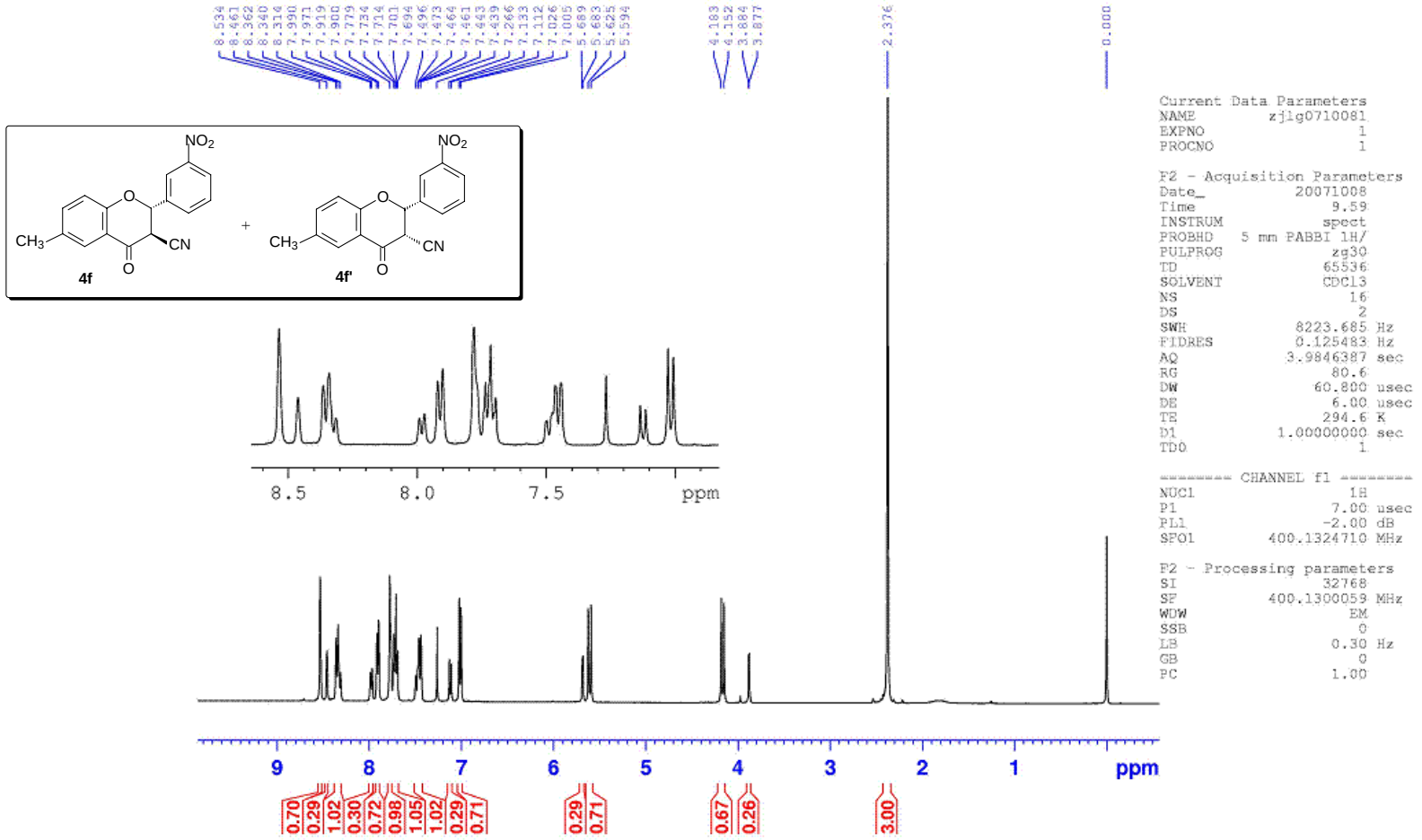



```
Current Data Parameters
NAME          zjlg0709278
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20070927
Time                 15.31
INSTRUM              spect
PROBHD   5 mm PABBI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ               3.9846387 sec
RG                    80.6
DW                  60.800 usec
DE                   6.00 usec
TE                  295.3 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   7.00 usec
PL1                 -2.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF            400.1300080 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

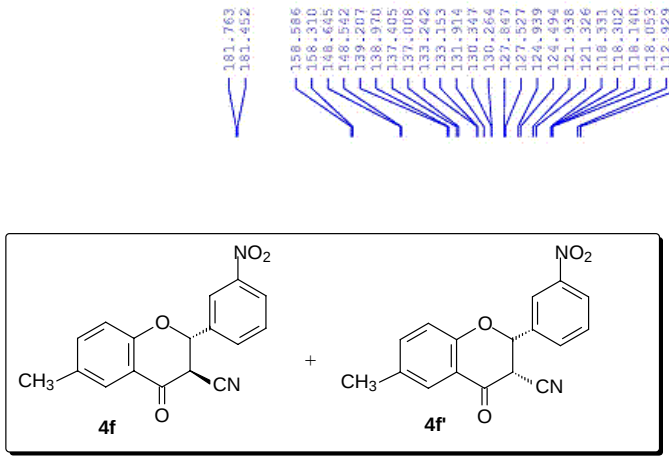


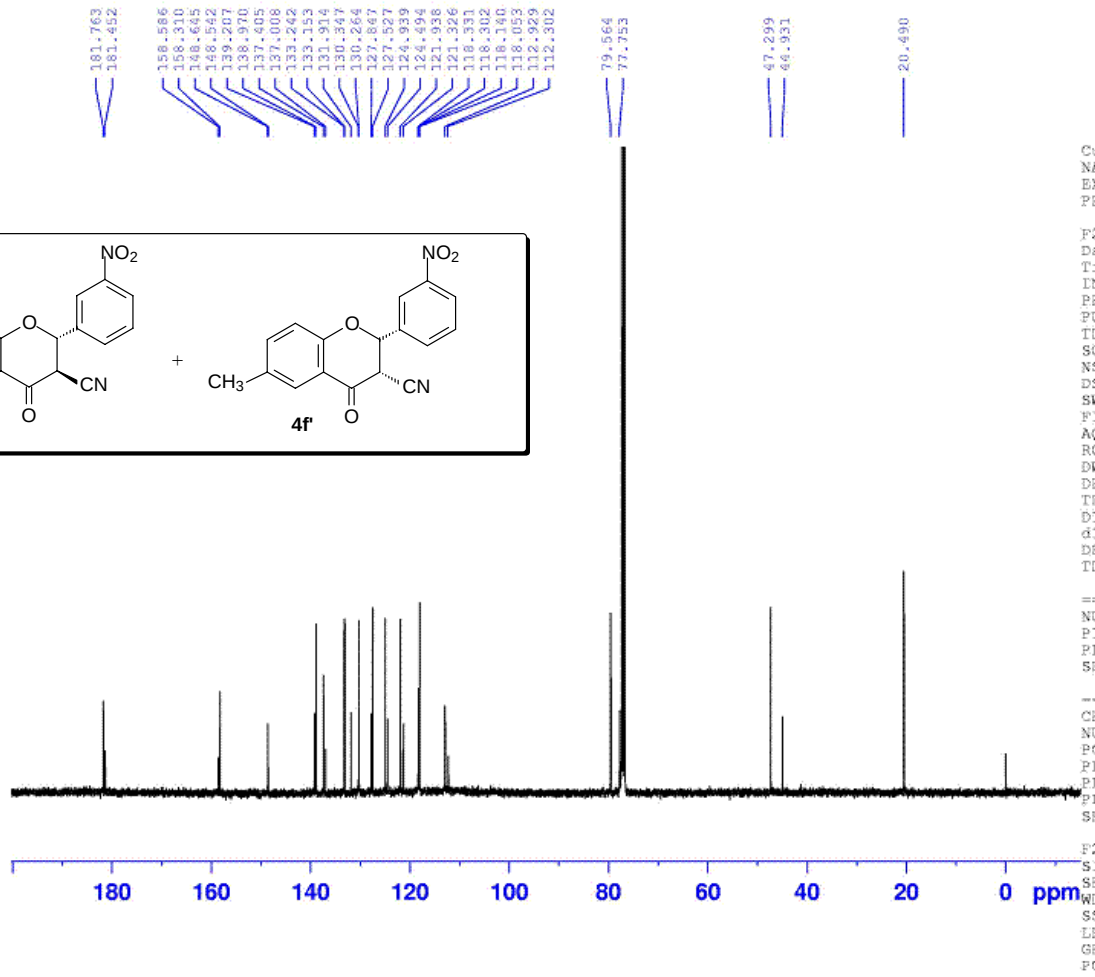



```
Current Data Parameters
NAME         zjlg0710199
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071019
Time               15.28
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3942
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                 20.2
DW                20.800 usec
DE                 6.00 usec
TE                294.4 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 14.90 usec
PL1                -3.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               18.50 dB
PL13               18.50 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
```

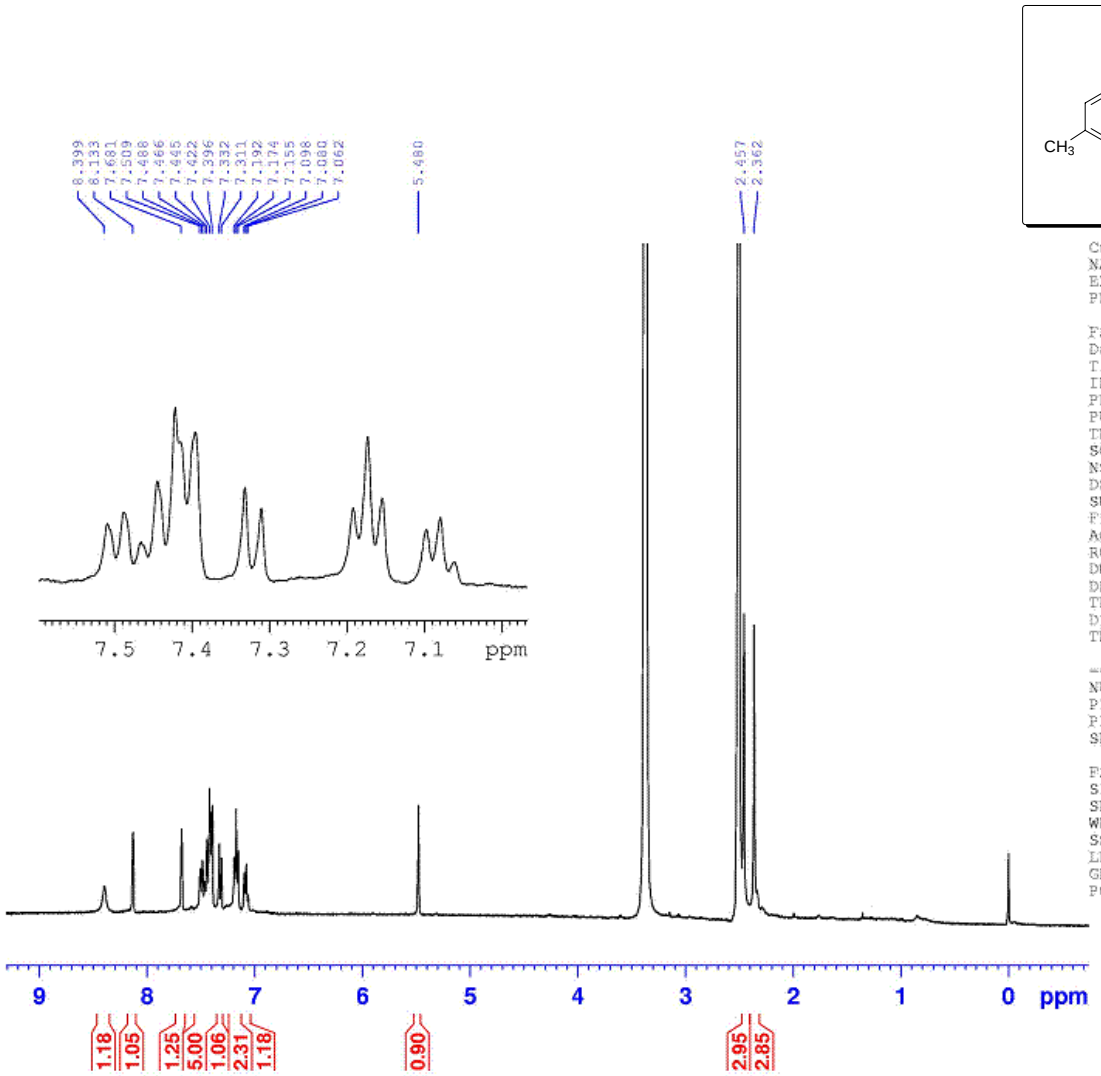


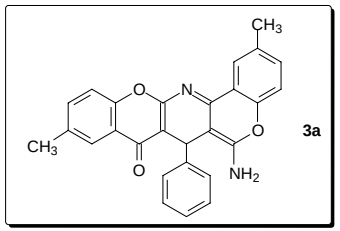

```
Current Data Parameters
NAME                  zjlg0710081
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20071008
Time                         9.59
INSTRUM                      spect
PROBHD    5 mm PABBI 1H/
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                      8223.685 Hz
FIDRES                   0.125483 Hz
AQ                       3.9846387 sec
RG                           80.6
DW                         60.800 usec
DE                          6.00 usec
TE                         294.6 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                          7.00 usec
PL1                        -2.00 dB
SFO1                 400.1324710 MHz

F2 - Processing parameters
SI                          32768
SF                   400.1300059 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```



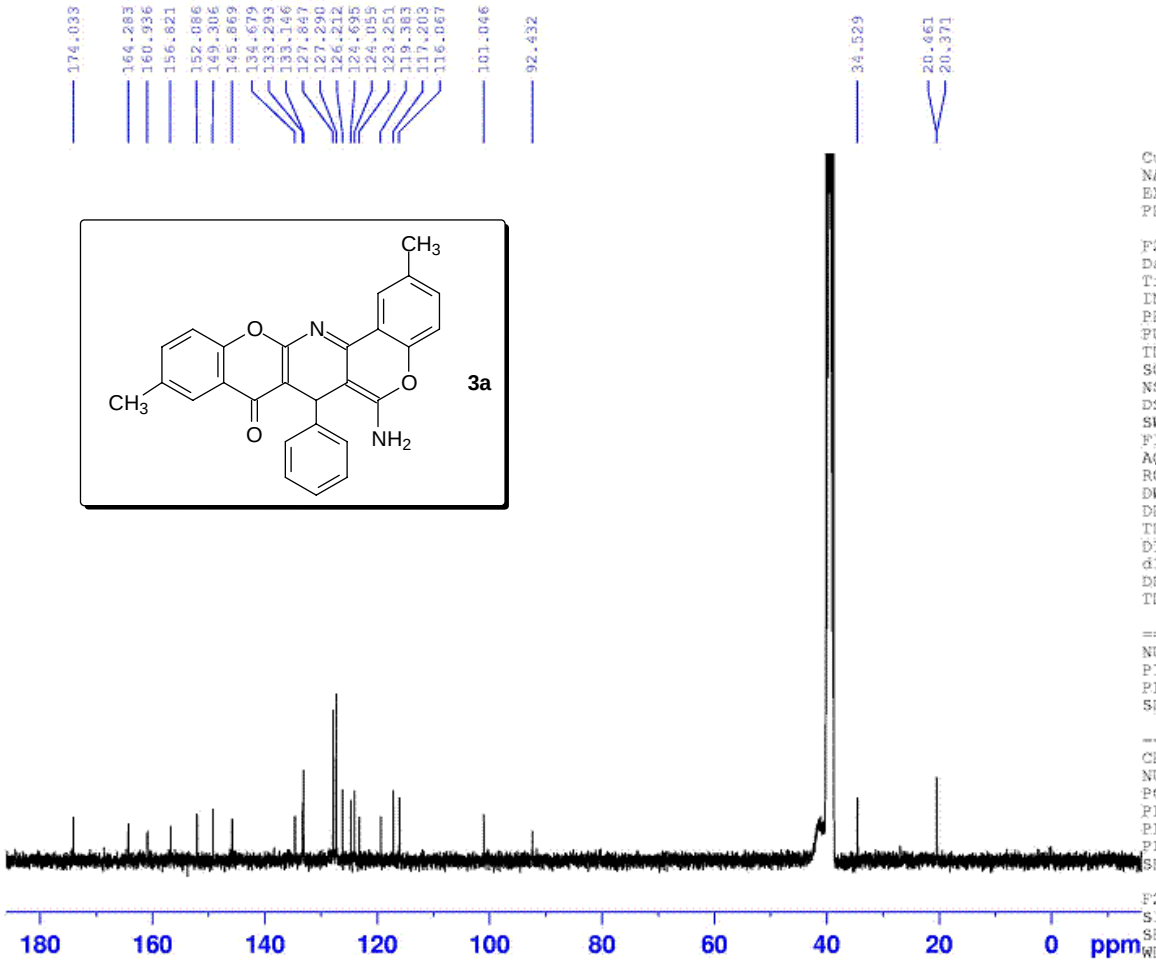



```
Current Data Parameters
NAME         zjlg0710081
EXPNO                 22
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071008
Time               10.12
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                 3124
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                 25.4
DW               20.800 usec
DE                6.00 usec
TE               294.8 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                14.90 usec
PL1               -3.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              18.50 dB
PL13              18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                32768
SF          100.6127700 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

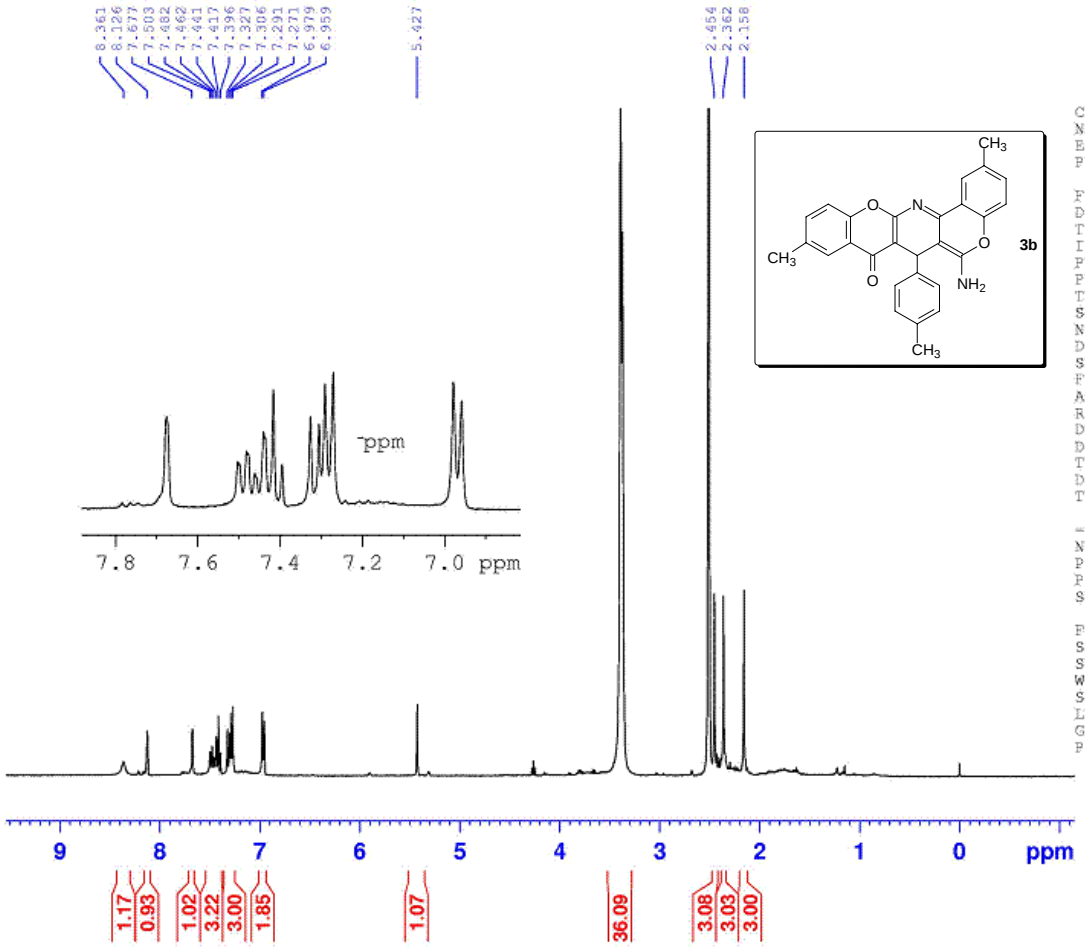




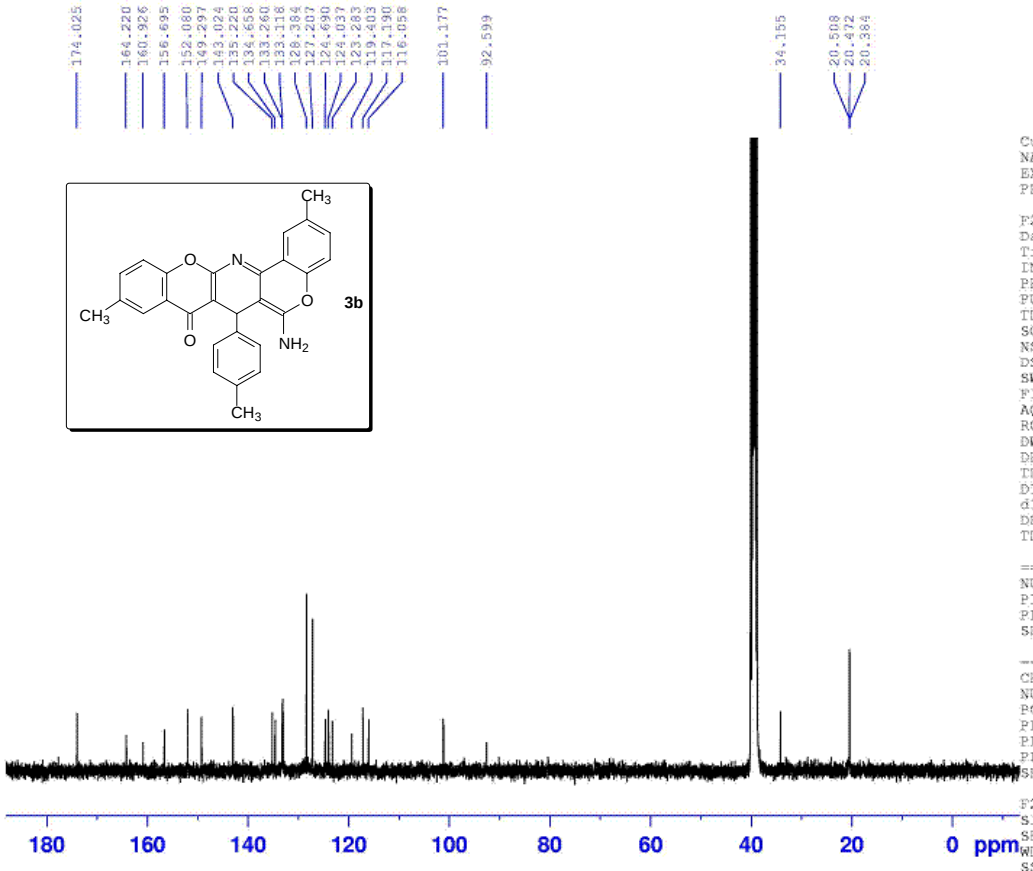

```
Current Data Parameters
NAME          zjlg0703233
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070323
Time                15.06
INSTRUM             spect
PROBHD    5 mm PAQNP Swi
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    256
DW                 60.800 usec
DE                  6.00 usec
TE                 292.9 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.90 usec
PL1                -3.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF           400.1299932 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



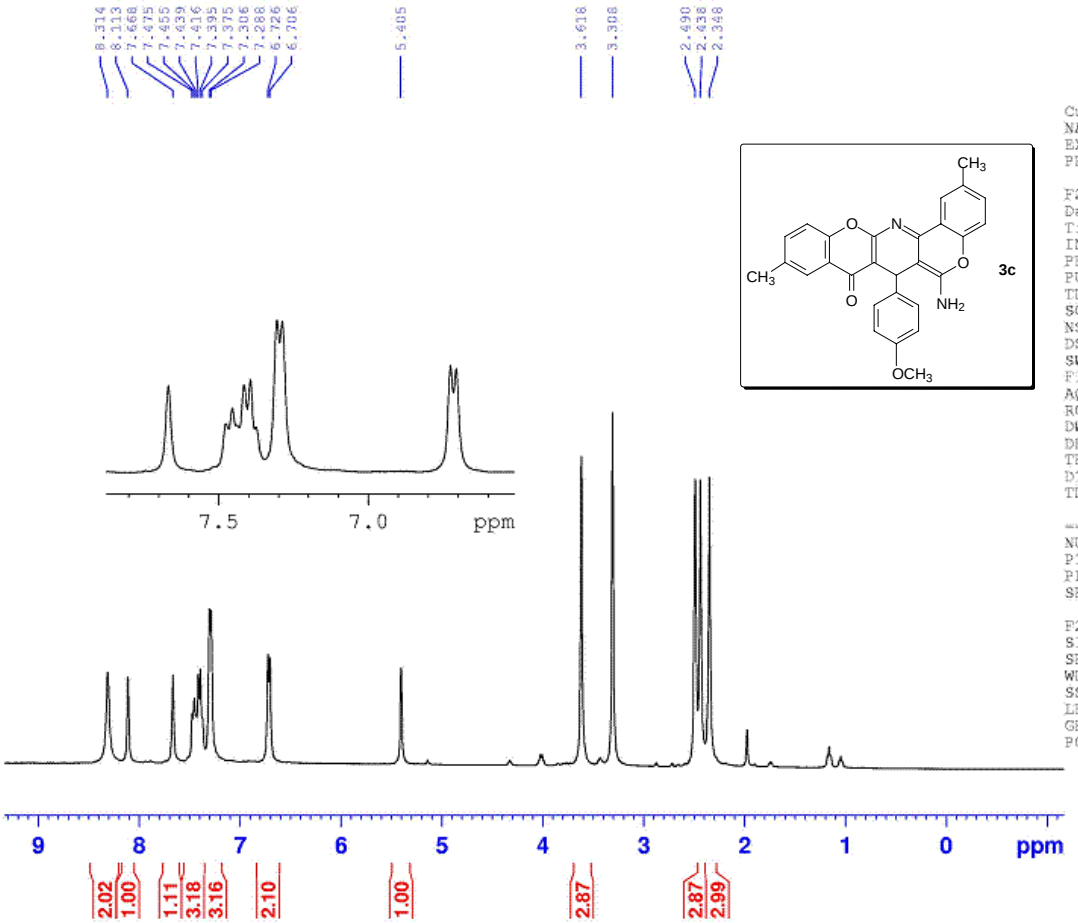

```
Current Data Parameters
NAME         zjlg0703233
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070325
Time              17.21
INSTRUM           spect
PROBHD   5 mm PAQNP Swi
PULPROG         zgpg30
TD               65536
SOLVENT           DMSO
NS                51200
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               297.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.00 usec
PL1                 -1.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               80.00 usec
PL2                 -3.00 dB
PL12                13.48 dB
PL13                14.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6128193 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



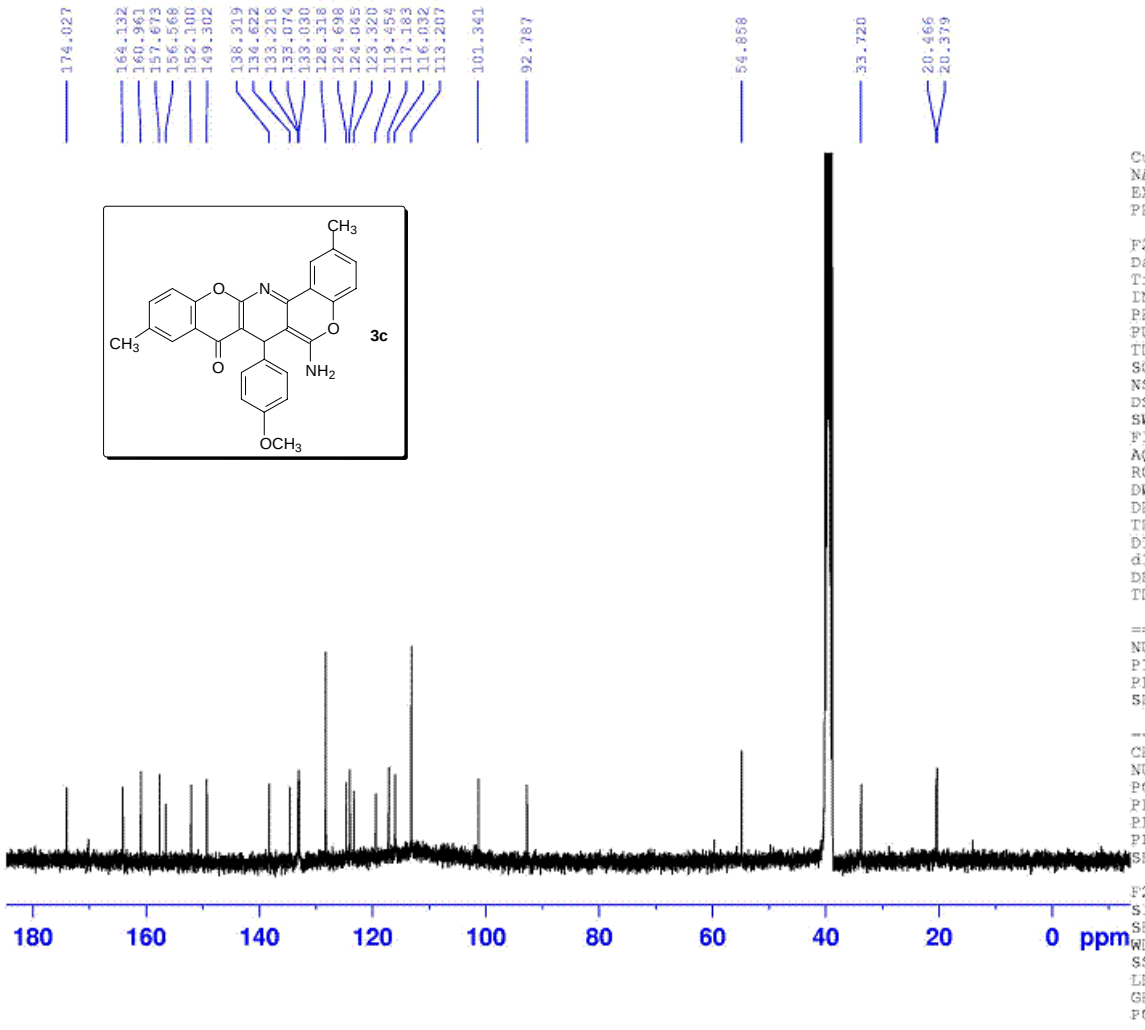

```
Current Data Parameters
NAME                  zjlg0703304
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20070330
Time                        16.10
INSTRUM                      spect
PROBHD    5 mm PAQNP Swi
PULPROG                      zg30
TD                          65536
SOLVENT                      DMSO
NS                             16
DS                              2
SWH                      8223.685 Hz
FIDRES                   0.125483 Hz
AQ                       3.9846387 sec
RG                            203
DW                         60.800 usec
DE                          6.00 usec
TE                           293.0 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                         10.90 usec
PL1                        -3.00 dB
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                          32768
SF                400.1299922 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```



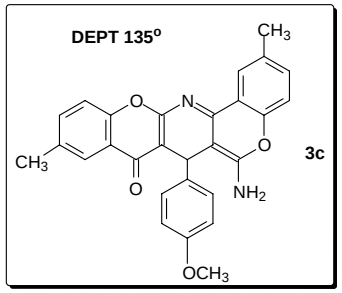

```
Current Data Parameters
NAME         zjlg0703304
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070331
Time                3.01
INSTRUM            spect
PROBHD   5 mm PAQNP Swi
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                11264
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                295.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.00 usec
PL1                -1.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.48 dB
PL13               14.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6128193 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

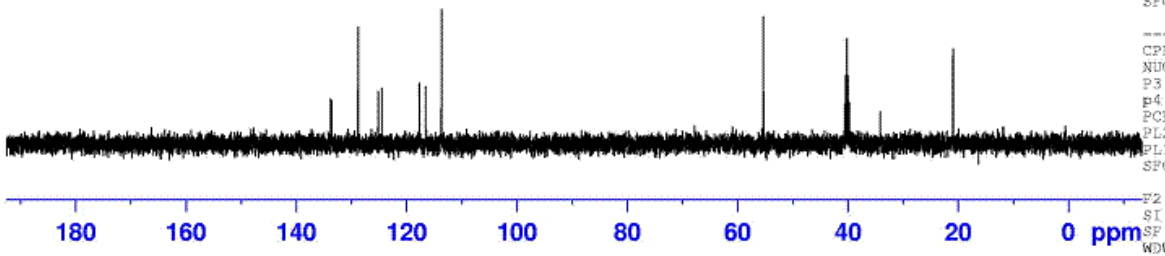



```
Current Data Parameters
NAME          zjlg07052211
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20070522
Time                  15.43
INSTRUM               spect
PROBHD   5 mm PAQNP Swi
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      114
DW                   60.800 usec
DE                    6.00 usec
TE                   300.0 K
D1                 1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                     1H
P1                   10.90 usec
PL1                  -3.00 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
```

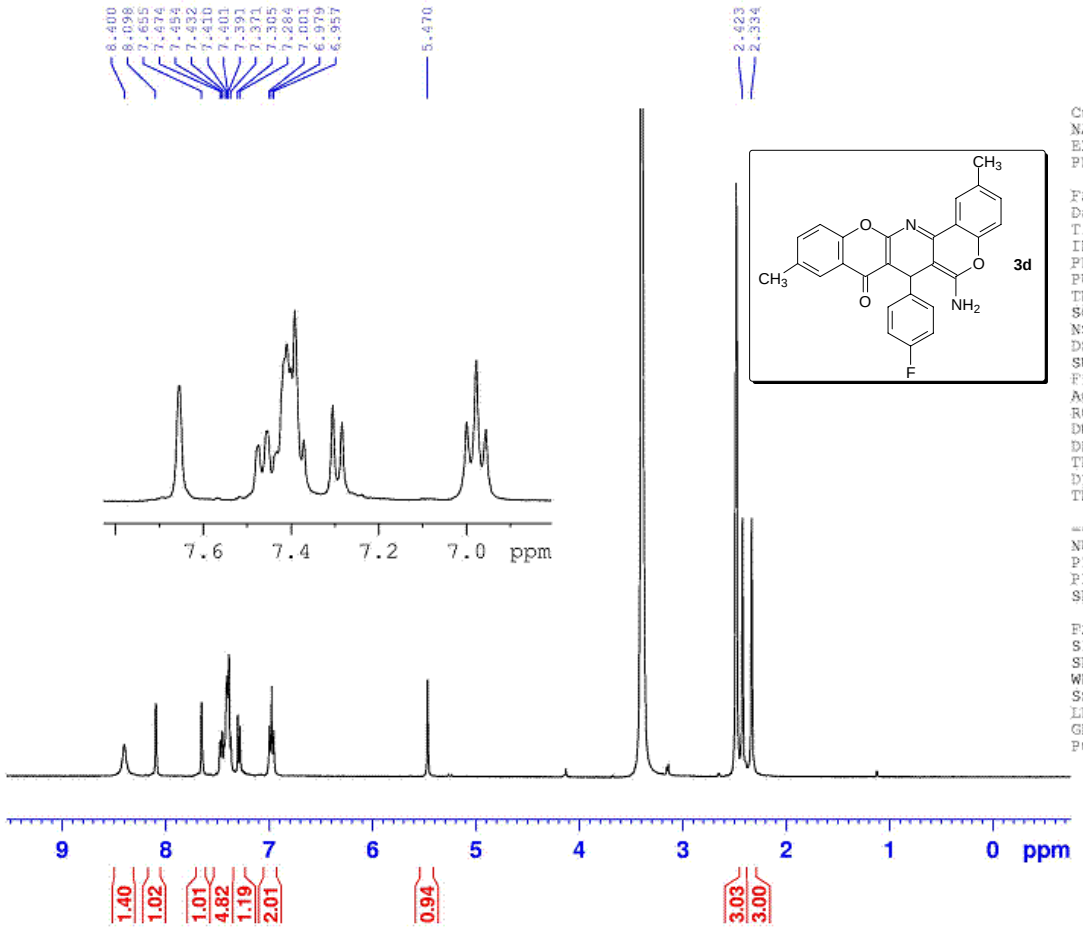



```
Current Data Parameters
NAME        zjlg07052211
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070522
Time               15.54
INSTRUM            spect
PROBHD   5 mm PAQNP Swi
PULPROG          zgpg30
TD                65536
SOLVENT            DMSO
NS                15376
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                   36
DW                20.800 usec
DE                 6.00 usec
TE                299.9 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.00 usec
PL1                -1.00 dB
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.48 dB
PL13               14.00 dB
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF          100.6128193 MHz
WDW                    EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

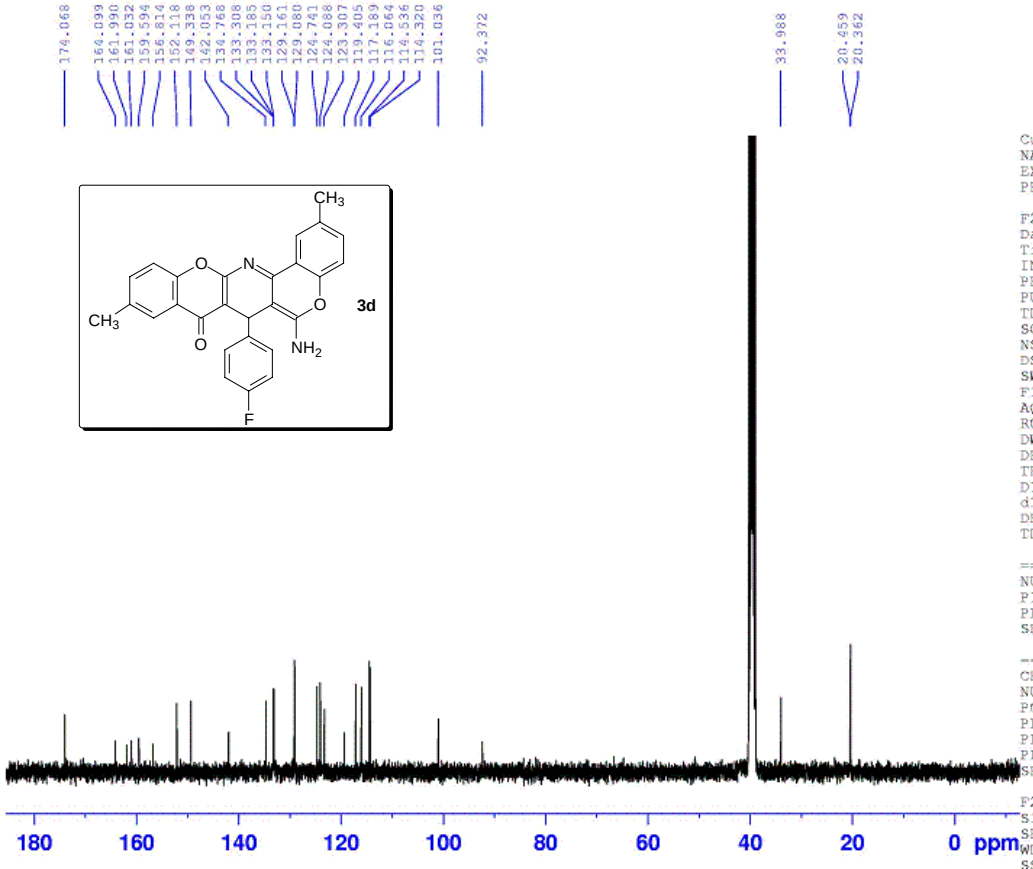




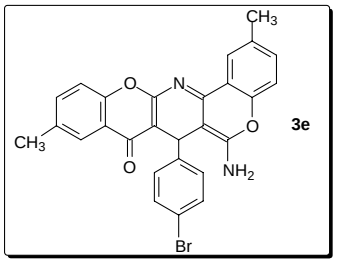

```
Current Data Parameters
NAME          zjlg0611142
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20061114
Time                12.30
INSTRUM             spect
PROBHD    5 mm PAQNP 8wi
PULPROG          dept135
TD                 65536
SOLVENT             DMSO
NS                   256
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                292.8 K
CNST2         145.0000000
D1              2.00000000 sec
d2              0.00344828 sec
d12             0.00002000 sec
DELTA           0.00001146 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.00 usec
p2                  18.00 usec
PL1                 -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
P3                  12.00 usec
p4                  24.00 usec
PCPD2               80.00 usec
PL2                 -3.00 dB
PL12                13.48 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
```



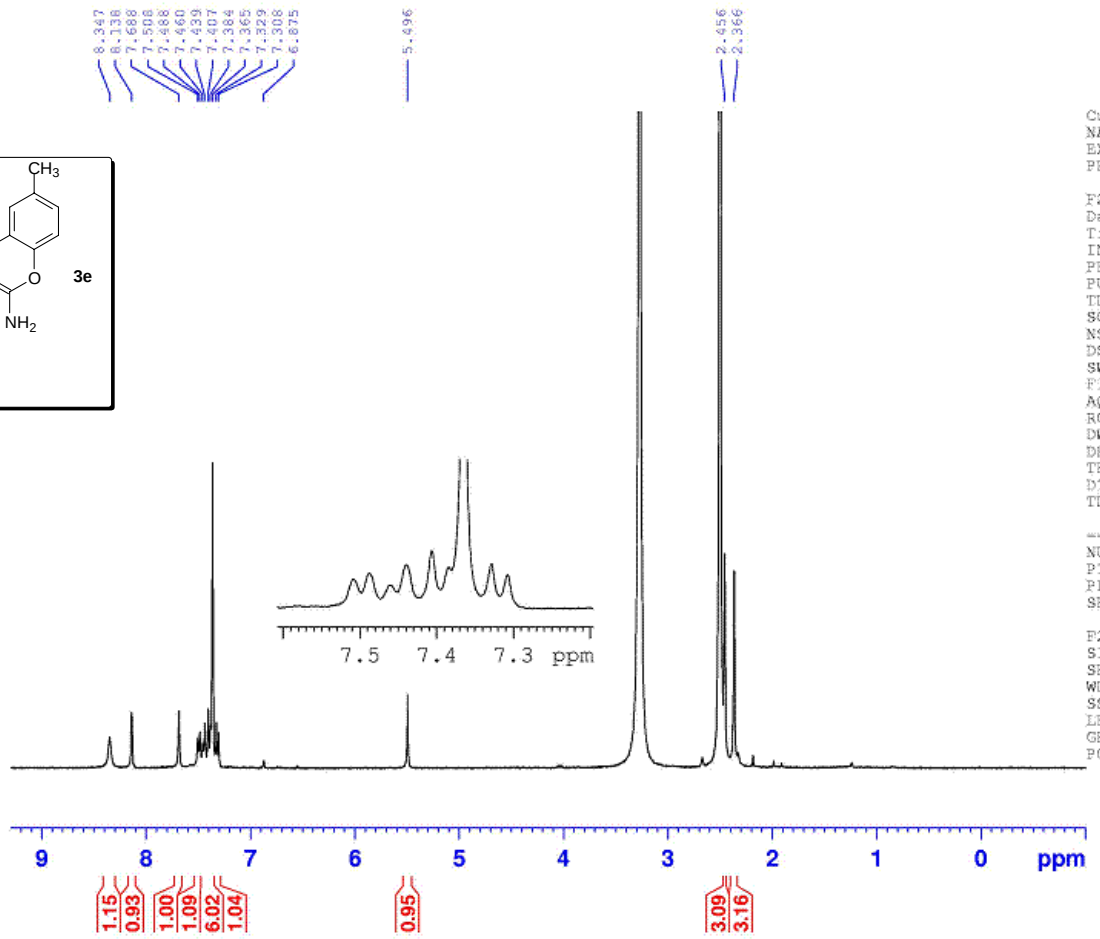

```
Current Data Parameters
NAME          zjlg0704091
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070409
Time                10.42
INSTRUM             spect
PROBHD   5 mm PAQNP Swi
PULPROG               zg30
TD                   65536
SOLVENT               DMSO
NS                      16
DS                       2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     203
DW                60.800 usec
DE                 6.00 usec
TE                 292.1 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.90 usec
PL1                -3.00 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
```



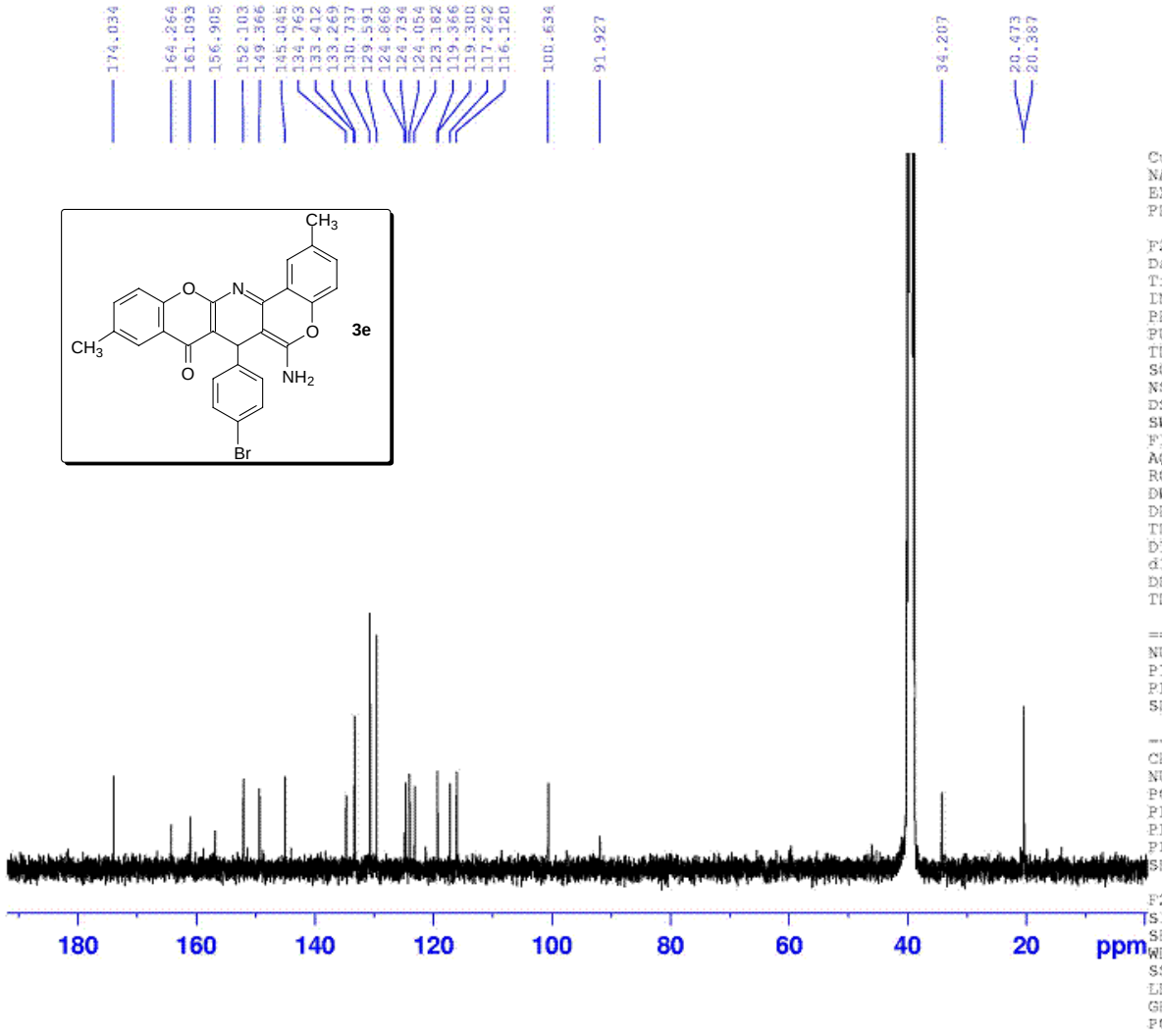

```
Current Data Parameters
NAME         zjlg0612142
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061214
Time               22.54
INSTRUM            spect
PROBHD   5 mm PAQNP Swi
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                  7168
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                 32768
DW                20.800 usec
DE                 6.00 usec
TE                315.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.00 usec
PL1                 -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -3.00 dB
PL12                13.48 dB
PL13                14.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6128193 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



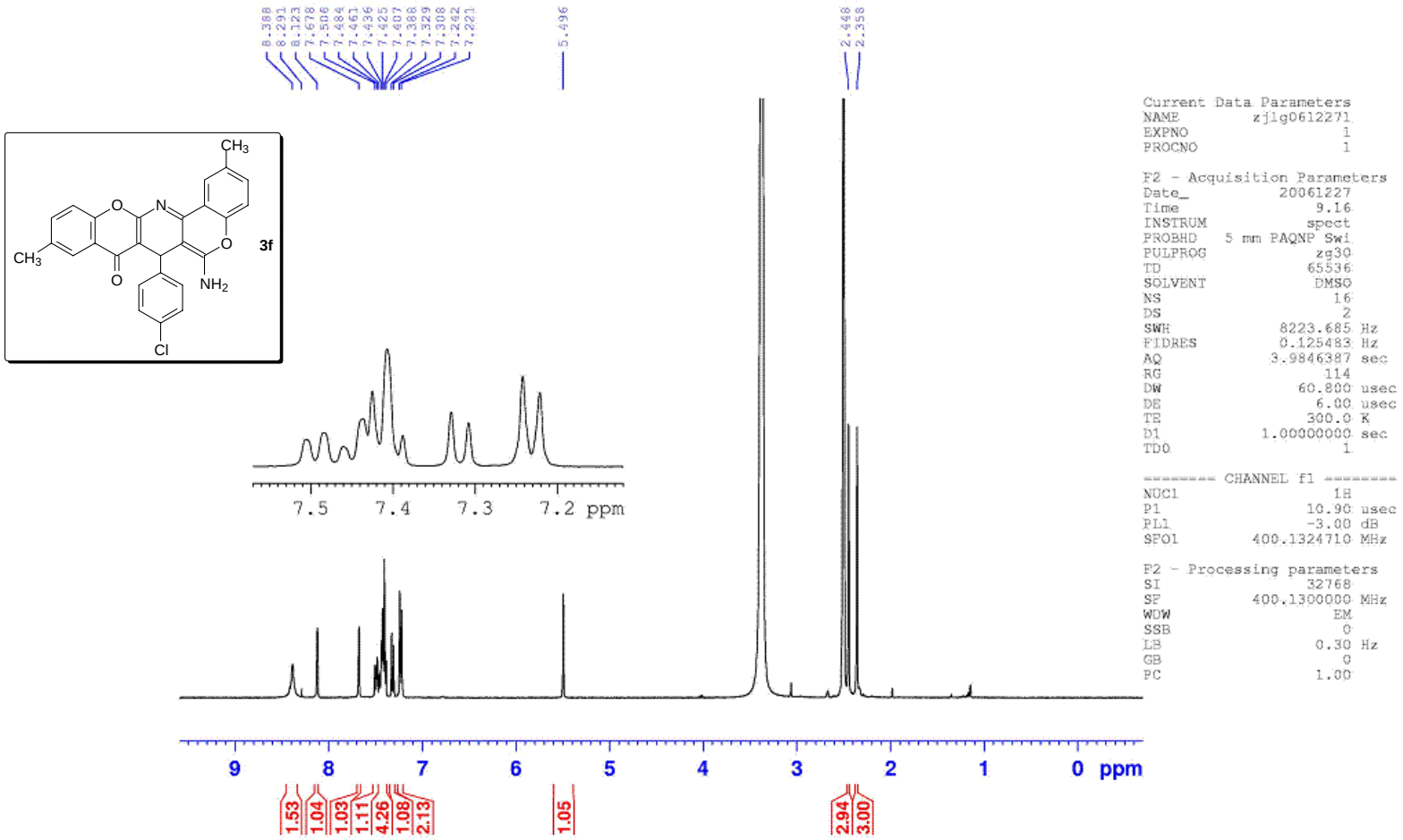
3e



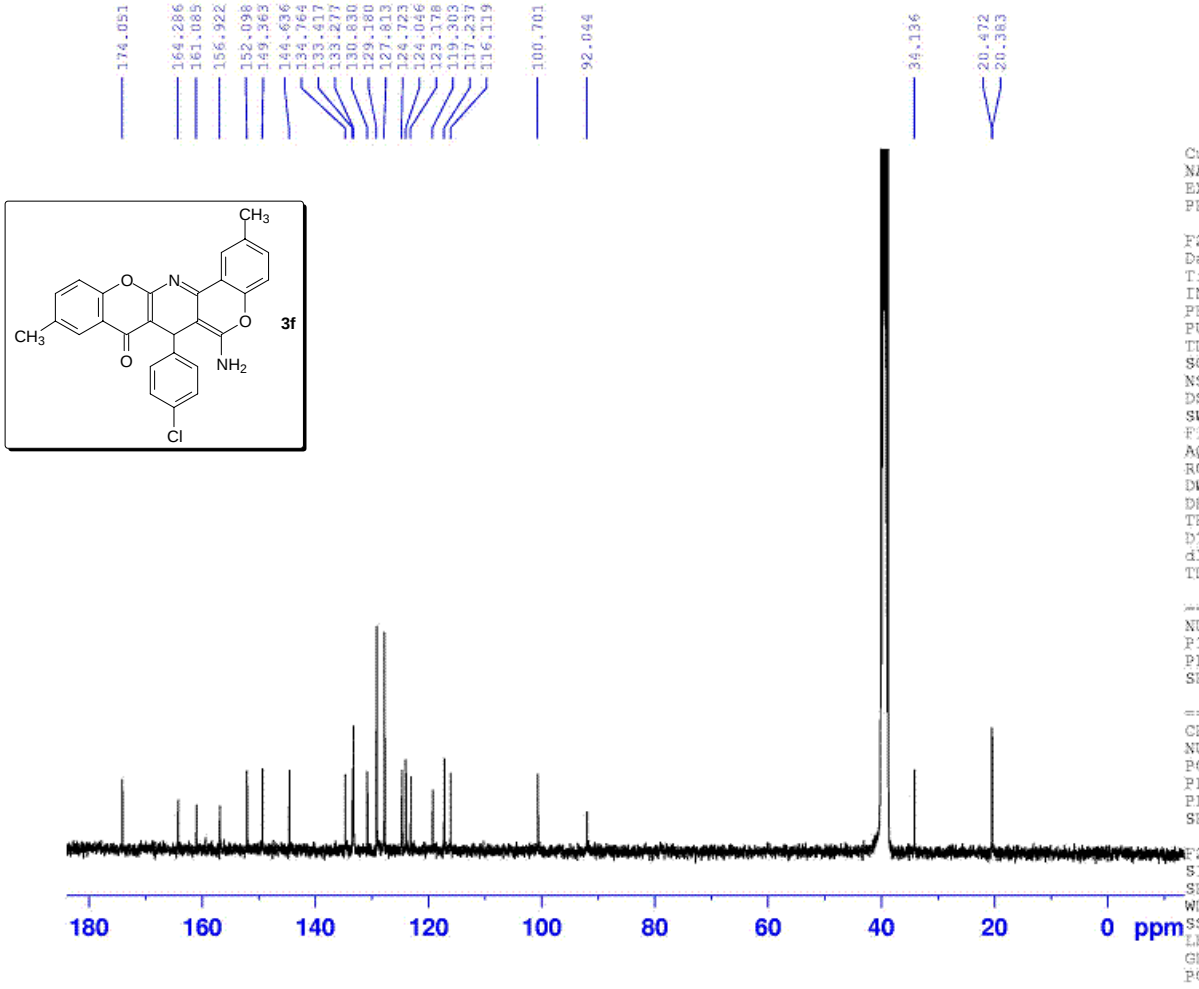

```
Current Data Parameters
NAME         zjlg0612201
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061220
Time               11.48
INSTRUM            spect
PROBHD   5 mm PAQNP Swi
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   256
DW                60.800 usec
DE                 6.00 usec
TE                310.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.90 usec
PL1                -3.00 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
```



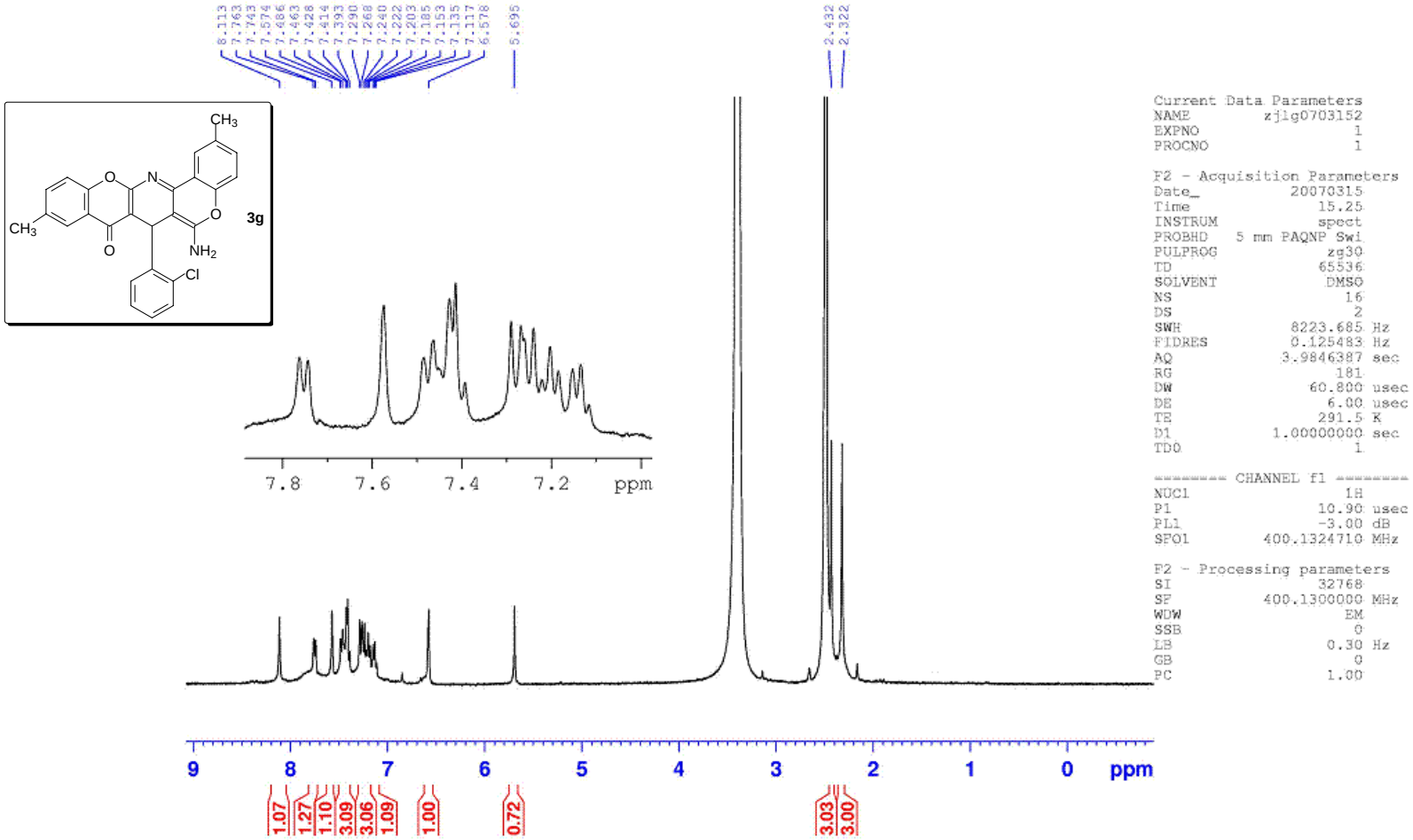

```
Current Data Parameters
NAME         zjlg0612201
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20061225
Time               16.39
INSTRUM             spect
PROBHD   5 mm PAQNP Swi
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                23552
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                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.00 usec
PL1                 -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.48 dB
PL13               14.00 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6128193 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



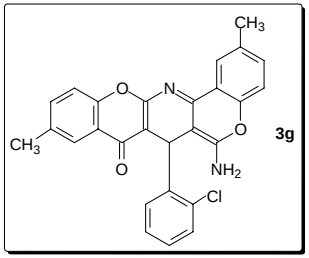

```
Current Data Parameters
NAME                 zjlg0612271
EXPNO                          1
PROCNO                         1

F2 - Acquisition Parameters
Date_               20061227
Time                    9.16
INSTRUM                spect
PROBHD    5 mm PAQNP Swi
PULPROG                 zg30
TD                     65536
SOLVENT                DMSO
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                      114
DW                   60.800 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    10.90 usec
PL1                   -3.00 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
```

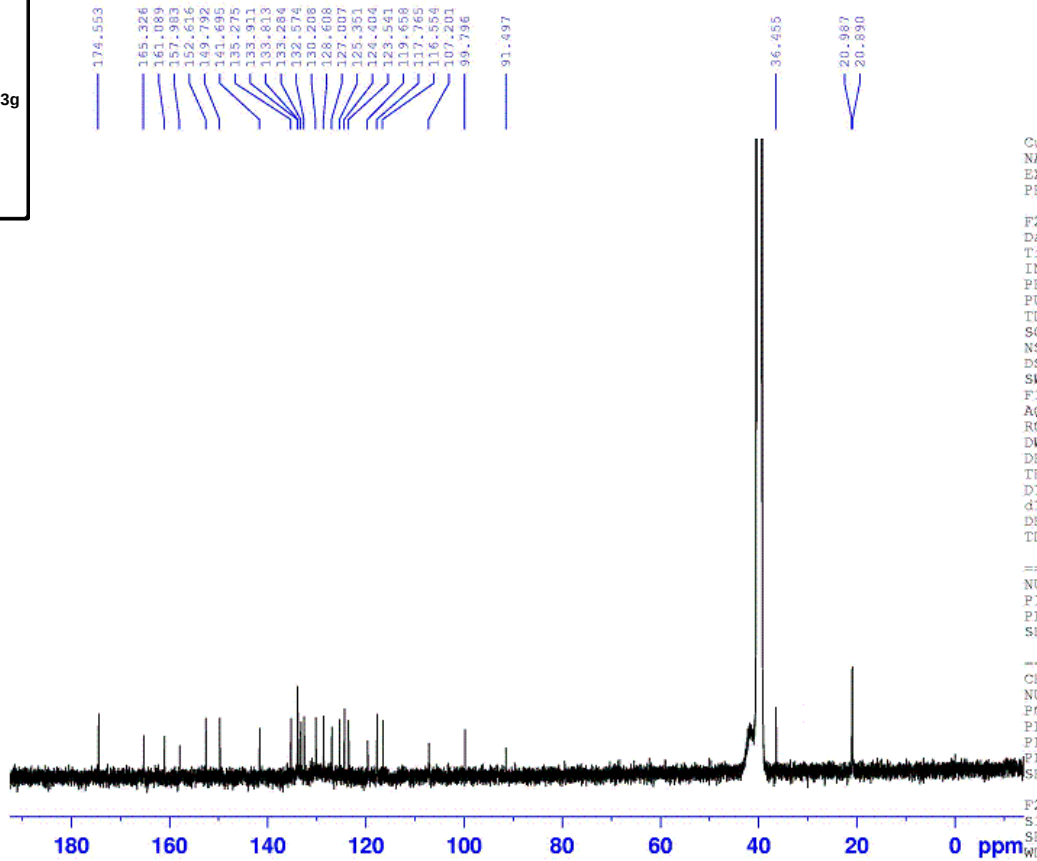



```
Current Data Parameters
NAME          zjlg0612271
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20061227
Time                11.44
INSTRUM             spect
PROBHD   5 mm PAQNP Swi
PULPROG          zgdc30
TD                 65536
SOLVENT             DMSO
NS                 12856
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                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
TD0                    1

========== CHANNEL f1 ==========
NUC1                  13C
P1                   9.00 usec
PL1                 -1.00 dB
SFO1         100.6228300 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.48 dB
SFO2         400.1316000 MHz

F2 - Processing parameters
SI                 32768
SF           100.6128193 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.40
```

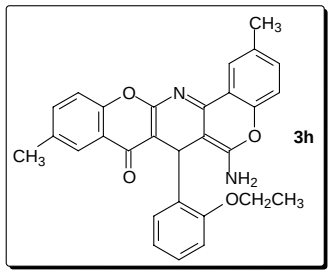


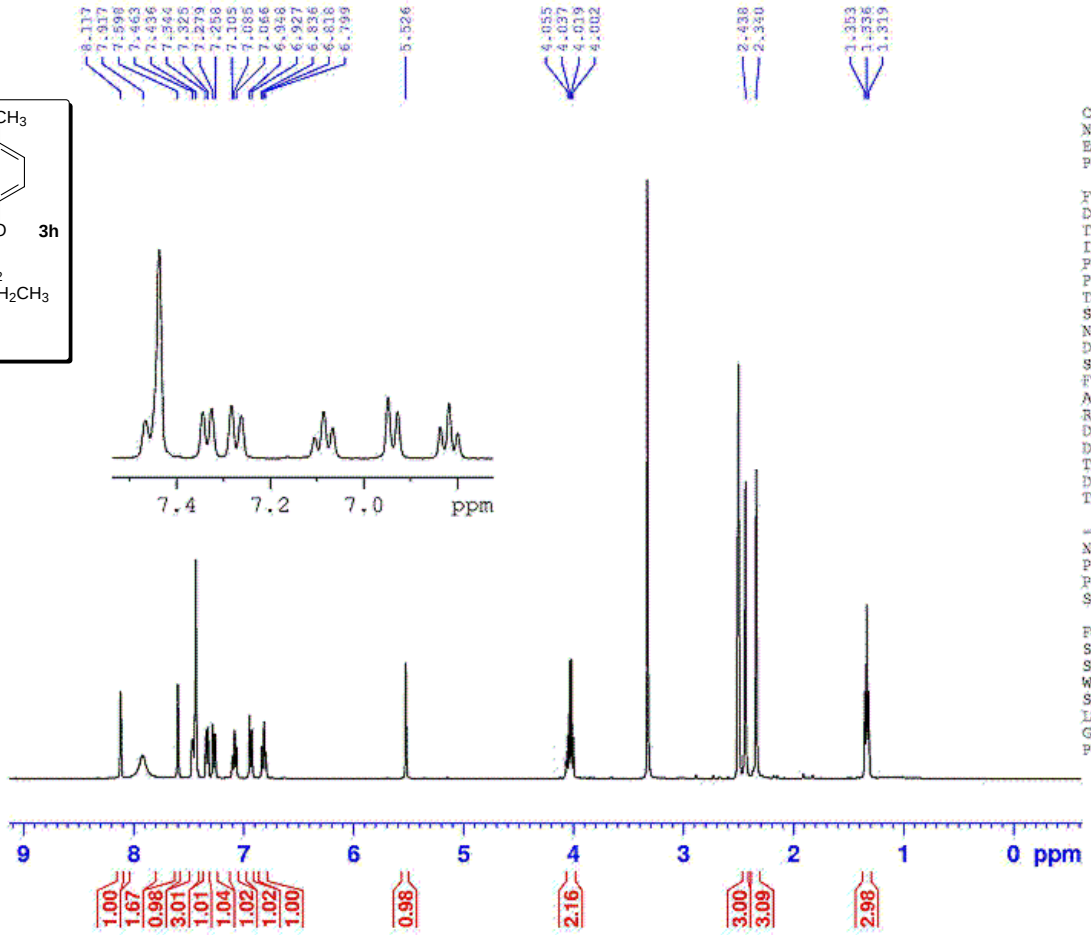

```
Current Data Parameters
NAME          zjlg0703152
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070315
Time                15.25
INSTRUM             spect
PROBHD   5 mm PAQNP Swi
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    181
DW                 60.800 usec
DE                  6.00 usec
TE                 291.5 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.90 usec
PL1                -3.00 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
```



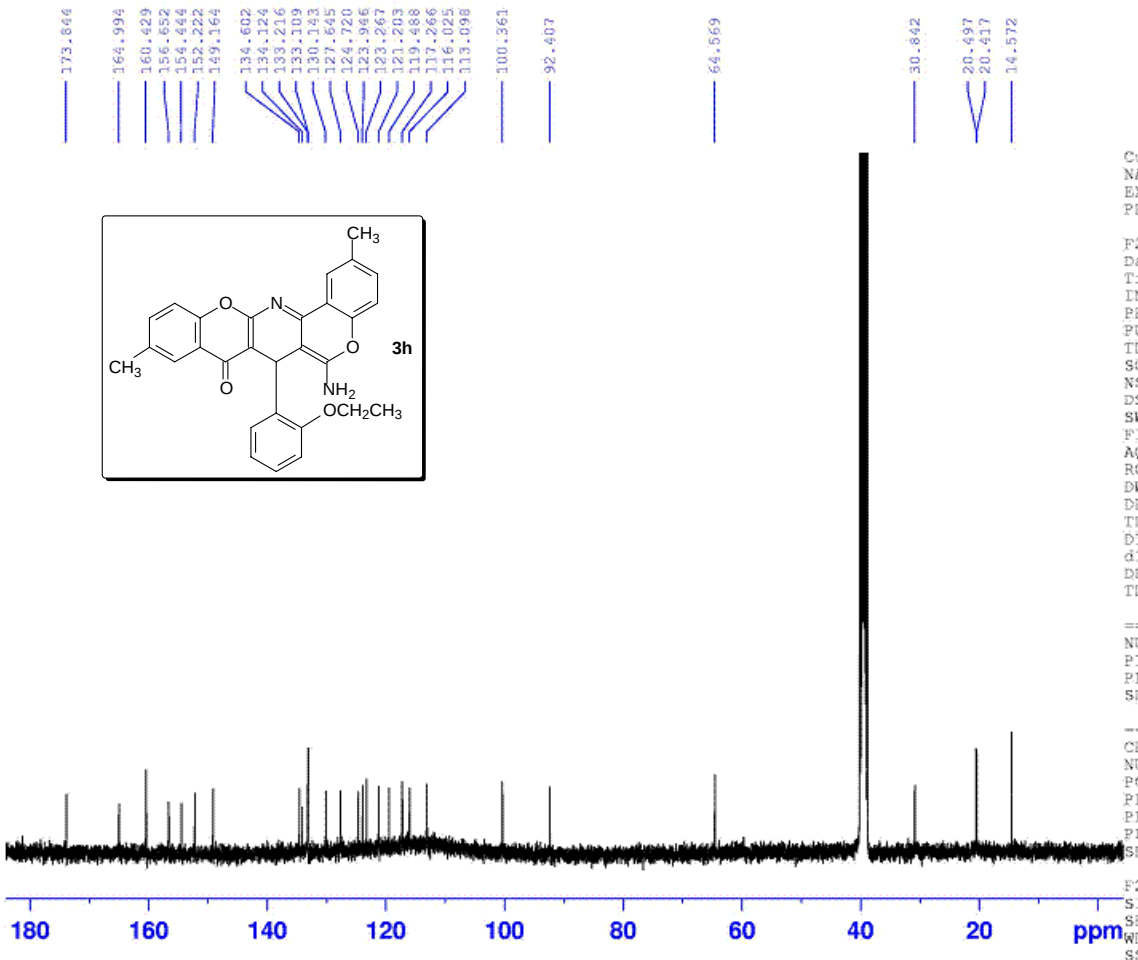



```
Current Data Parameters
NAME         zjlg0703164
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070316
Time               15.01
INSTRUM             spect
PROBHD   5 mm PAQNP Swi
PULPROG          zgpg30
TD                 65536
SOLVENT             DMSO
NS                 51206
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                291.1 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.00 usec
PL1                 -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -3.00 dB
PL12                13.48 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
```

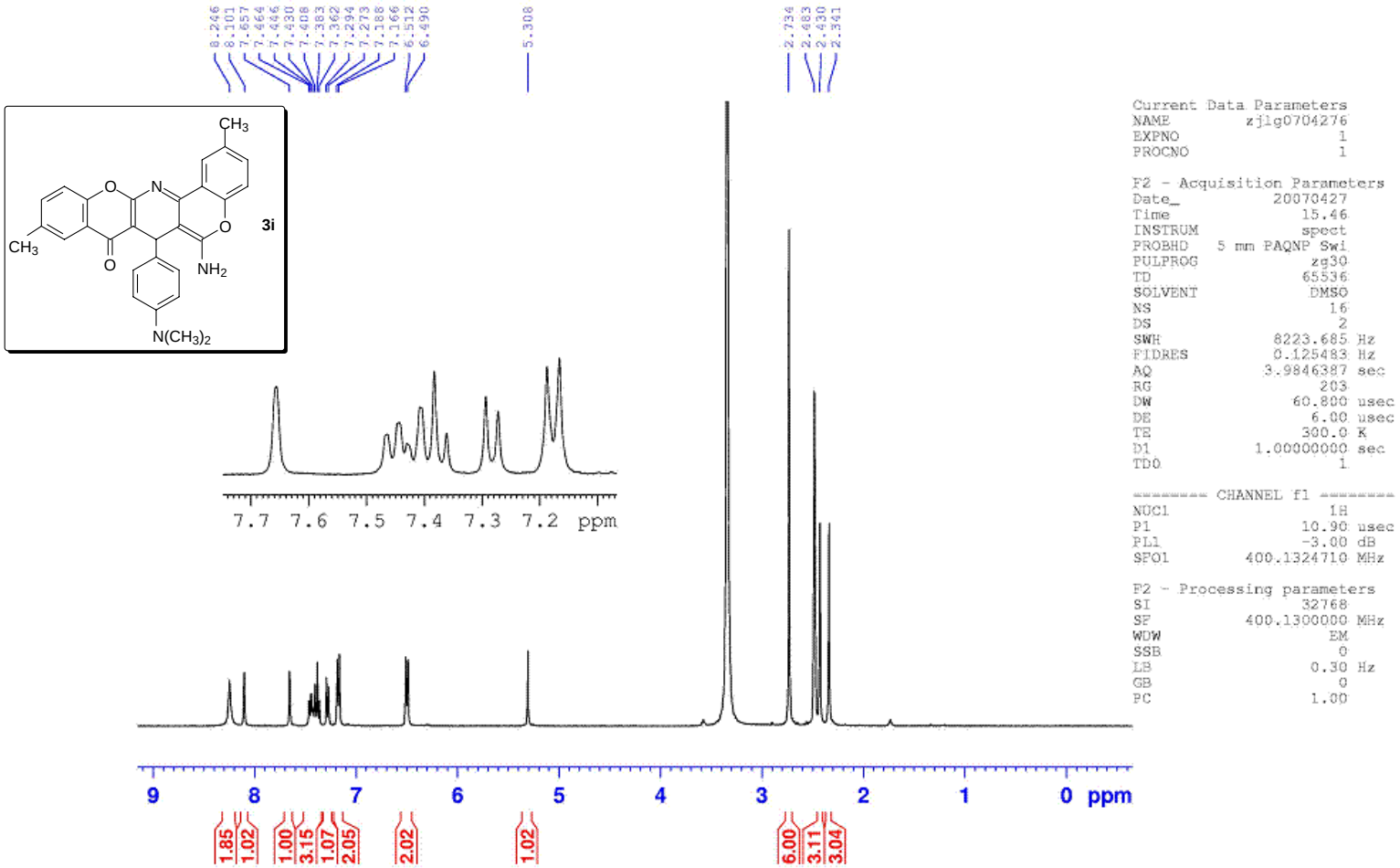




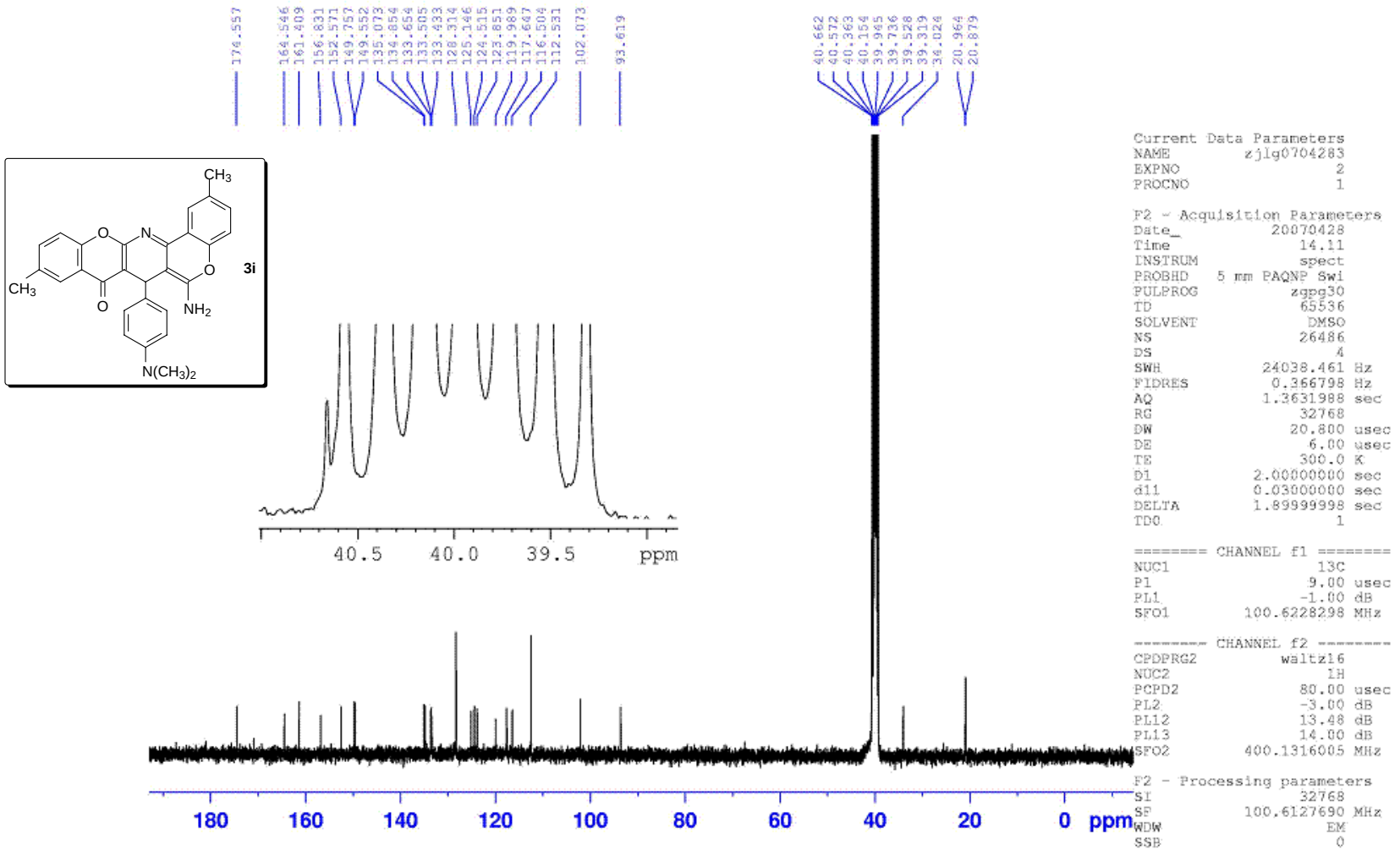

```
Current Data Parameters
NAME                  zjlg0707253
EXPNO                           1
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20070726
Time                         0.26
INSTRUM                      spect
PROBHD     5 mm PABBI 1H/
PULPROG                      zg30
TD                          65536
SOLVENT                      DMSO
NS                           12505
DS                               2
SWH                       8223.685 Hz
FIDRES                    0.125483 Hz
AQ                        3.9846387 sec
RG                             144
DW                          60.800 usec
DE                           6.00 usec
TE                          297.2 K
D1                    1.00000000 sec
TD0                              1

======== CHANNEL f1 ========
NUC1                           1H
P1                           7.00 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
```



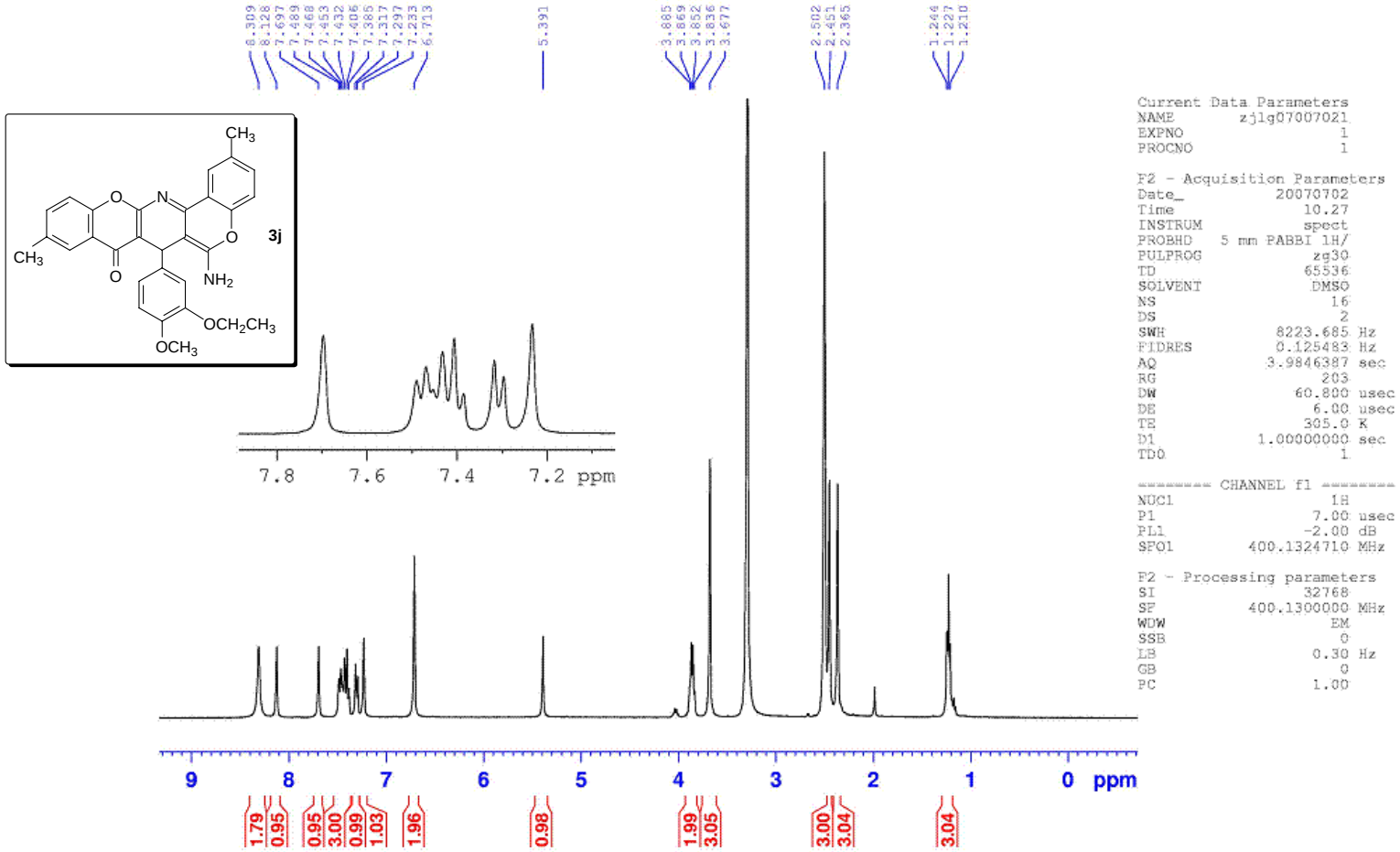

```
Current Data Parameters
NAME          zjlg0707253
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070726
Time                9.19
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                  4998
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  71.8
DW                20.800 usec
DE                 6.00 usec
TE                296.8 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 14.90 usec
PL1                -3.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               18.50 dB
PL13               18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6128193 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



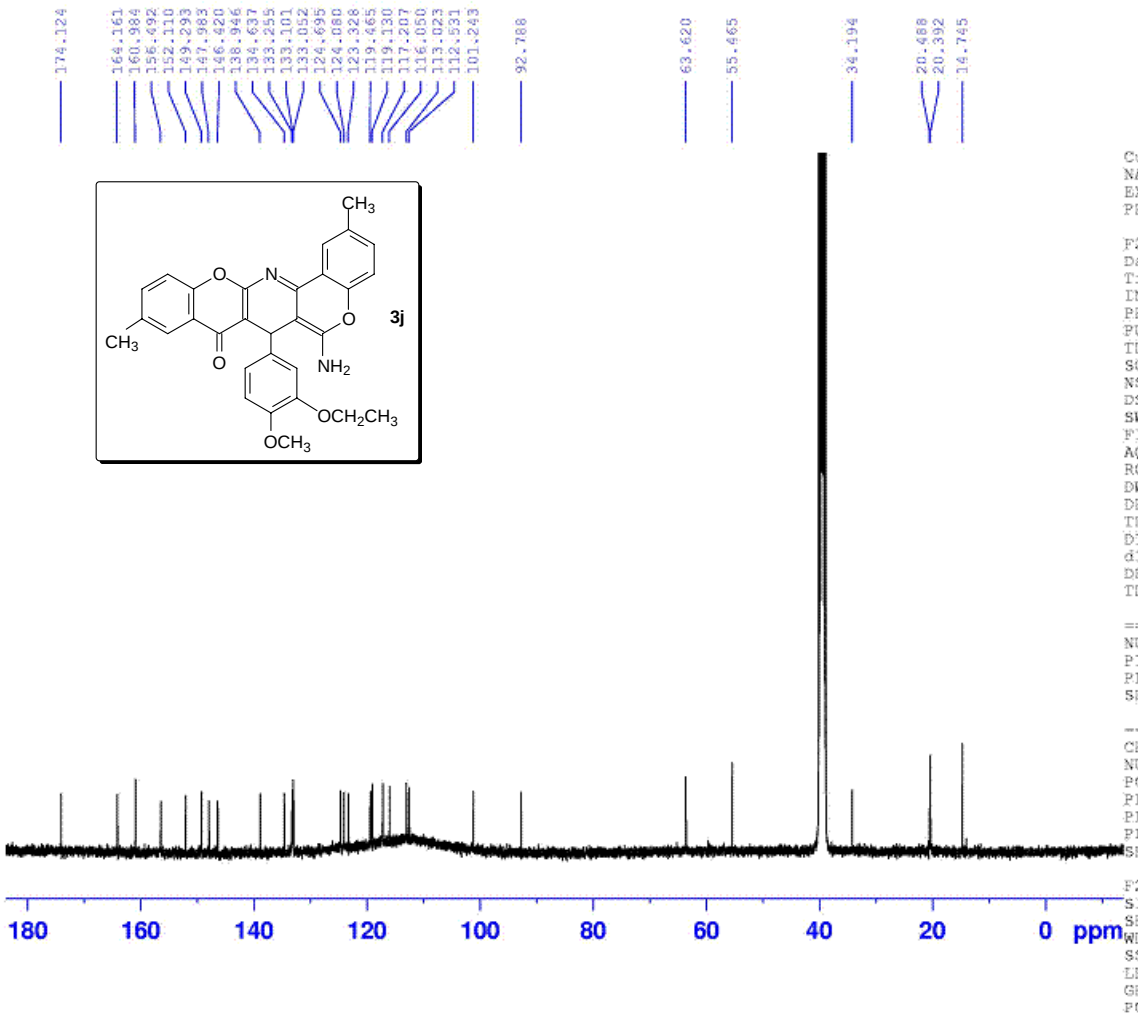

```
Current Data Parameters
NAME         zjlg0704276
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070427
Time               15.46
INSTRUM            spect
PROBHD   5 mm PAQNP Swi
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   203
DW                60.800 usec
DE                 6.00 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.90 usec
PL1                -3.00 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
```



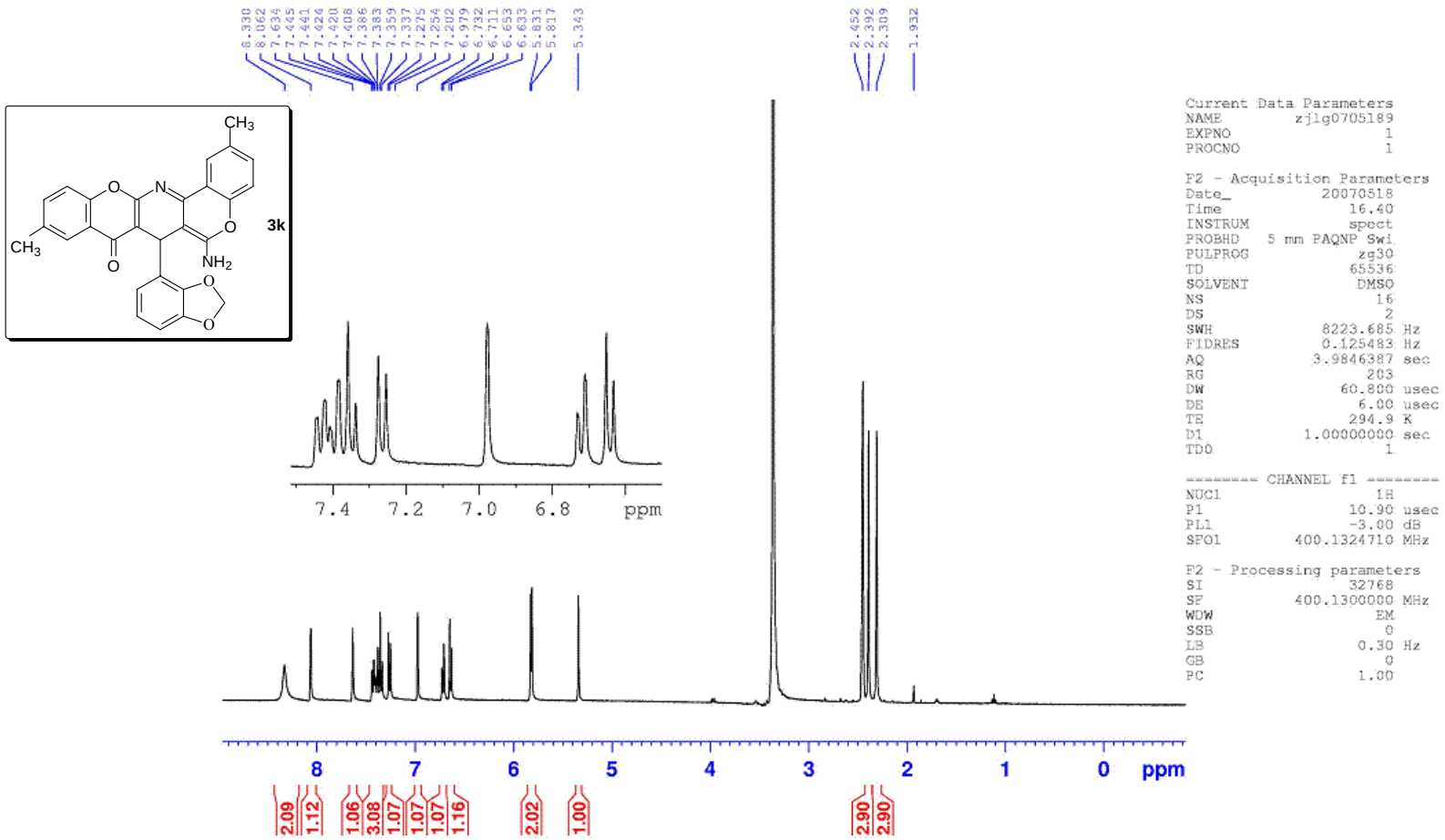

```
Current Data Parameters
NAME         zjlg0704283
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070428
Time               14.11
INSTRUM             spect
PROBHD   5 mm PAQNP Swi
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                26486
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                32768
DW               20.800 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.00 usec
PL1                -1.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -3.00 dB
PL12               13.48 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
```



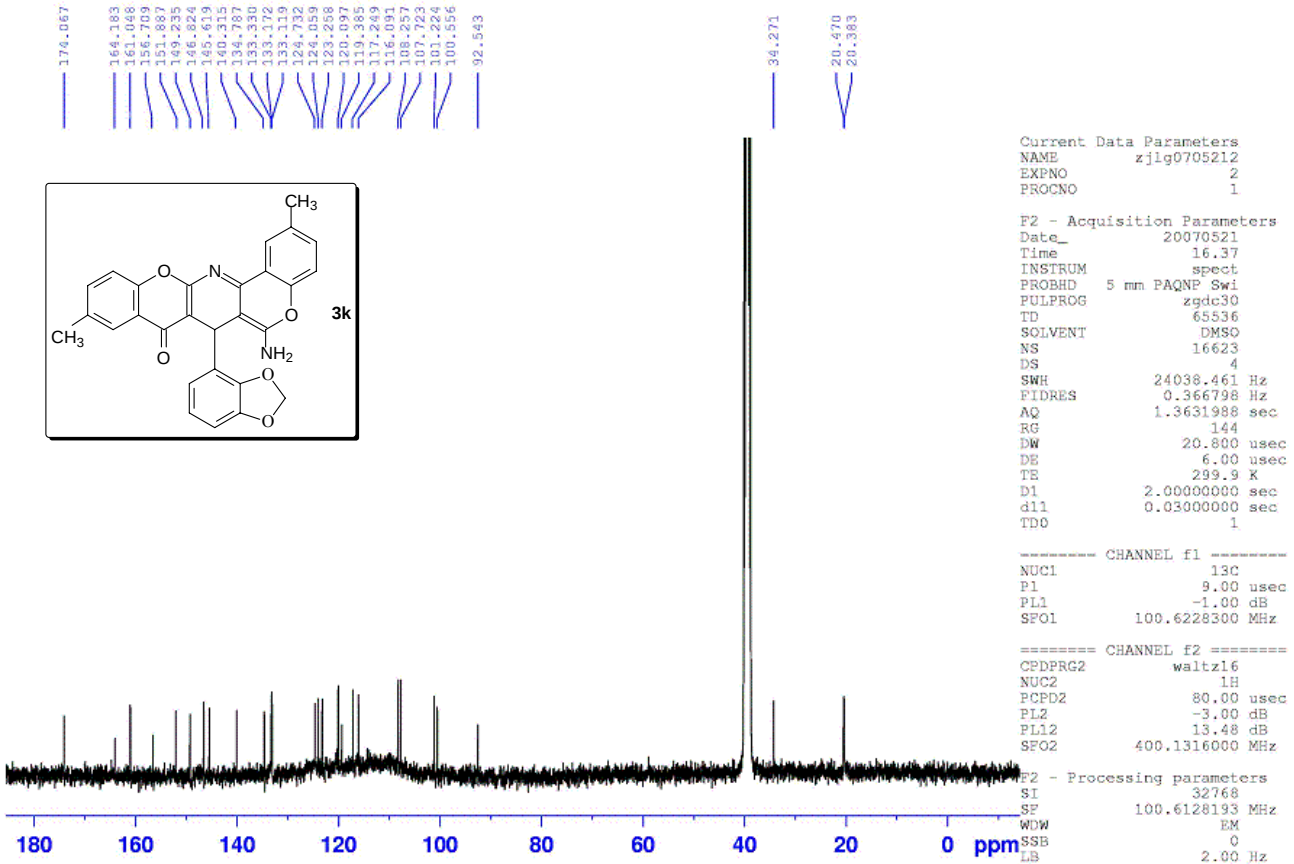

```
Current Data Parameters
NAME        zjlg07007021
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070702
Time               10.27
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG            zg30
TD                65536
SOLVENT            DMSO
NS                   16
DS                    2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                  203
DW               60.800 usec
DE                6.00 usec
TE                305.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.00 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
```



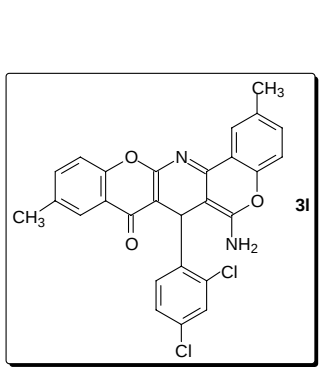

```
Current Data Parameters
NAME          zjlg0706293
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070629
Time                15.41
INSTRUM             spect
PROBHD    5 mm PABBI 1H/
PULPROG             zgpg30
TD                   65536
SOLVENT               DMSO
NS                   25761
DS                       4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631988 sec
RG                      64
DW                20.800 usec
DE                 6.00 usec
TE                297.3 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                 14.90 usec
PL1                -3.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               18.50 dB
PL13               18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF           100.6128193 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



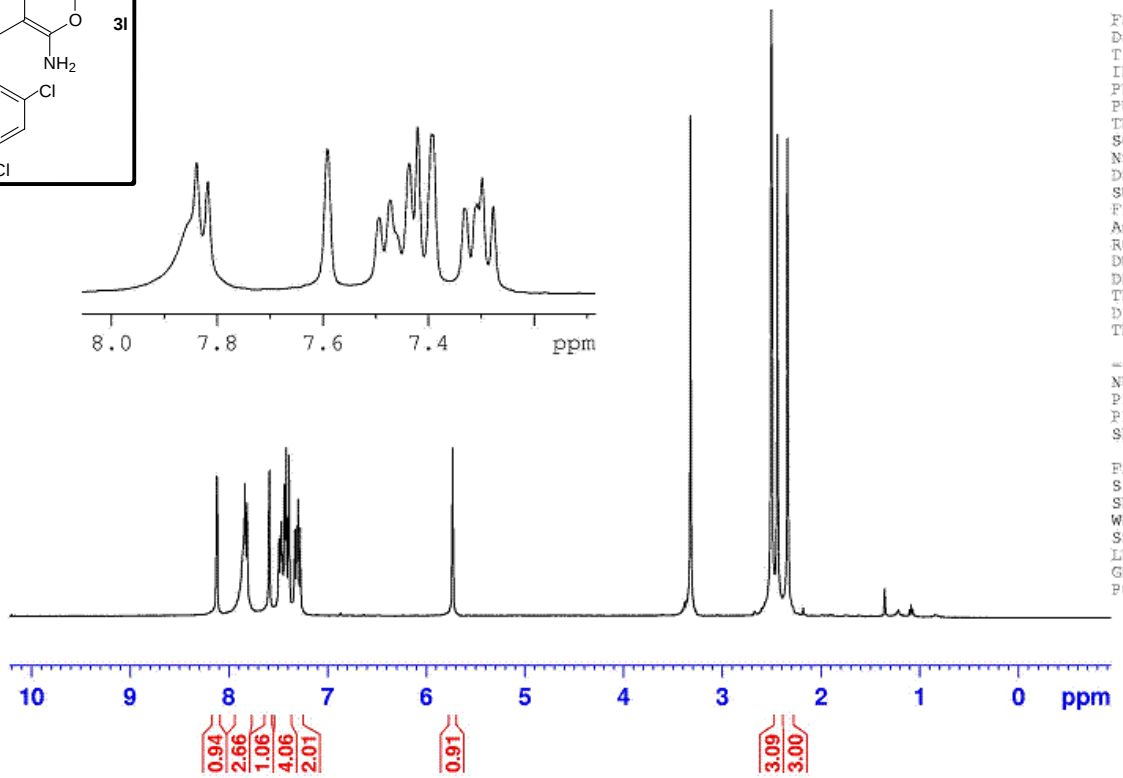

```
Current Data Parameters
NAME          zjlg0705189
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070518
Time                16.40
INSTRUM             spect
PROBHD   5 mm PAQNP Swi
PULPROG              zg30
TD                  65536
SOLVENT              DMSO
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    203
DW                 60.800 usec
DE                  6.00 usec
TE                 294.9 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.90 usec
PL1                -3.00 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
```



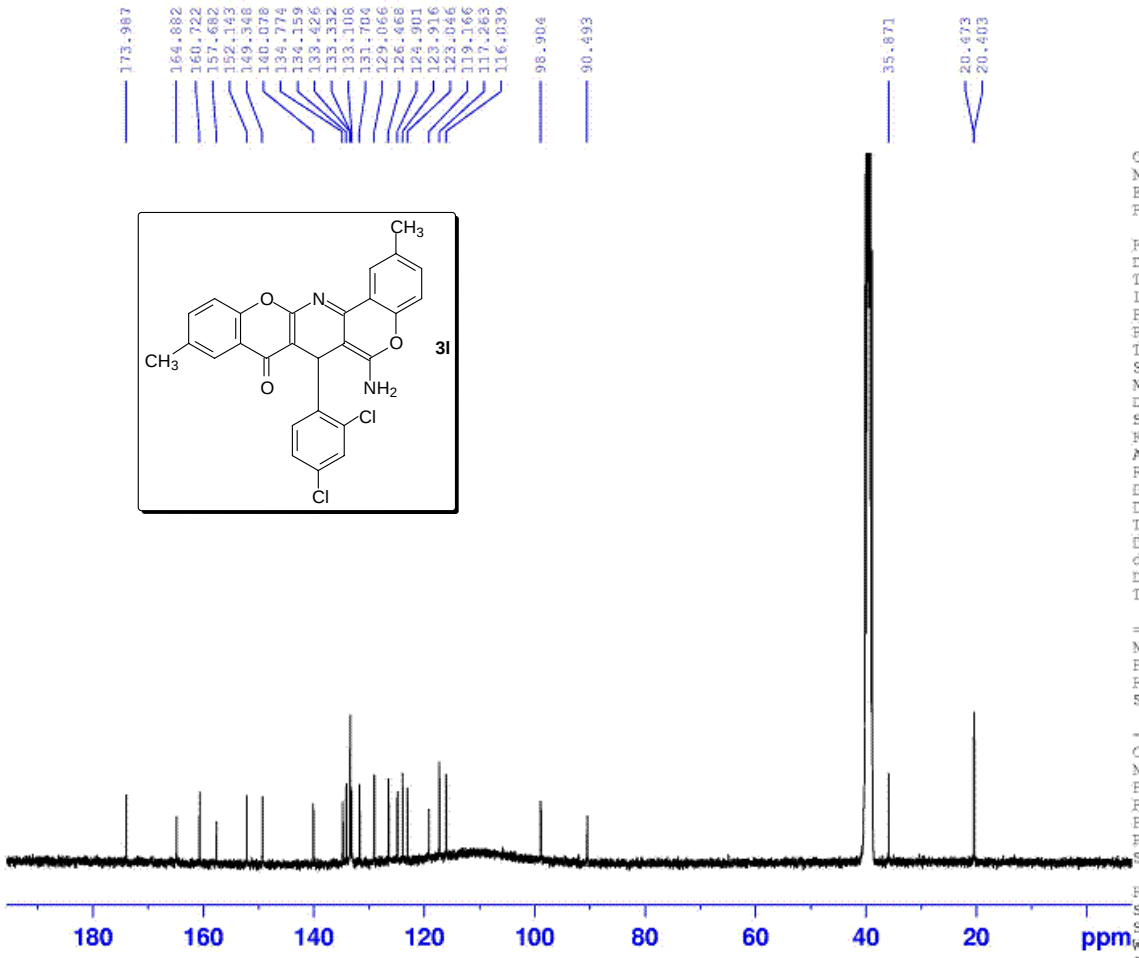

```
Current Data Parameters
NAME         zjlg0705212
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070521
Time               16.37
INSTRUM             spect
PROBHD   5 mm PAQNP Swi
PULPROG          zgdc30
TD                65536
SOLVENT            DMSO
NS                16623
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  144
DW               20.800 usec
DE                6.00 usec
TE                299.9 K
D1            2.00000000 sec
d11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.00 usec
PL1               -1.00 dB
SFO1        100.6228300 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2               -3.00 dB
PL12              13.48 dB
SFO2        400.1316000 MHz

F2 - Processing parameters
SI                 32768
SF          100.6128193 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.40
```



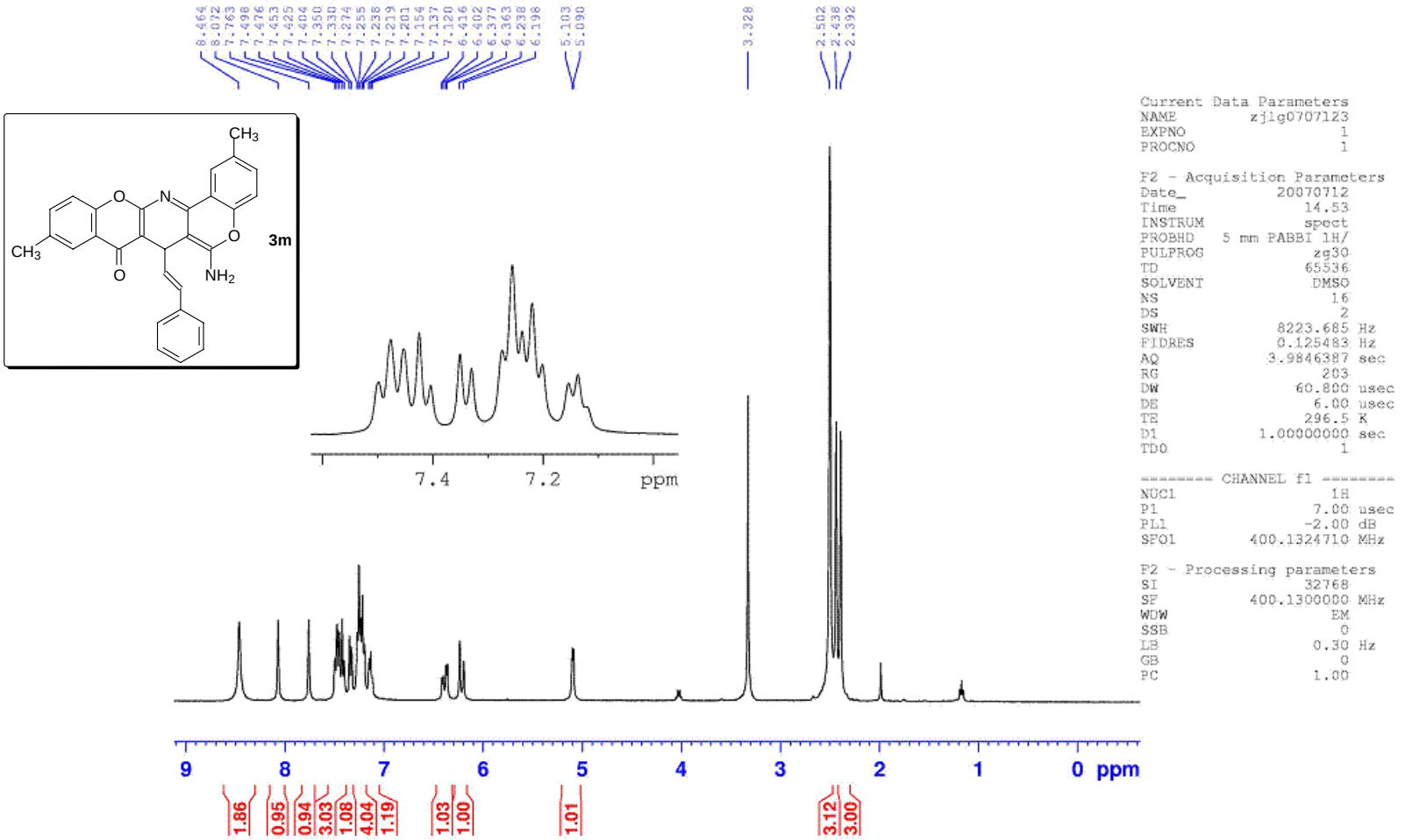
8.123
7.839
7.818
7.591
7.493
7.472
7.437
7.421
7.396
7.392
7.333
7.308
7.299
7.278
5.733
2.503
2.442
2.340



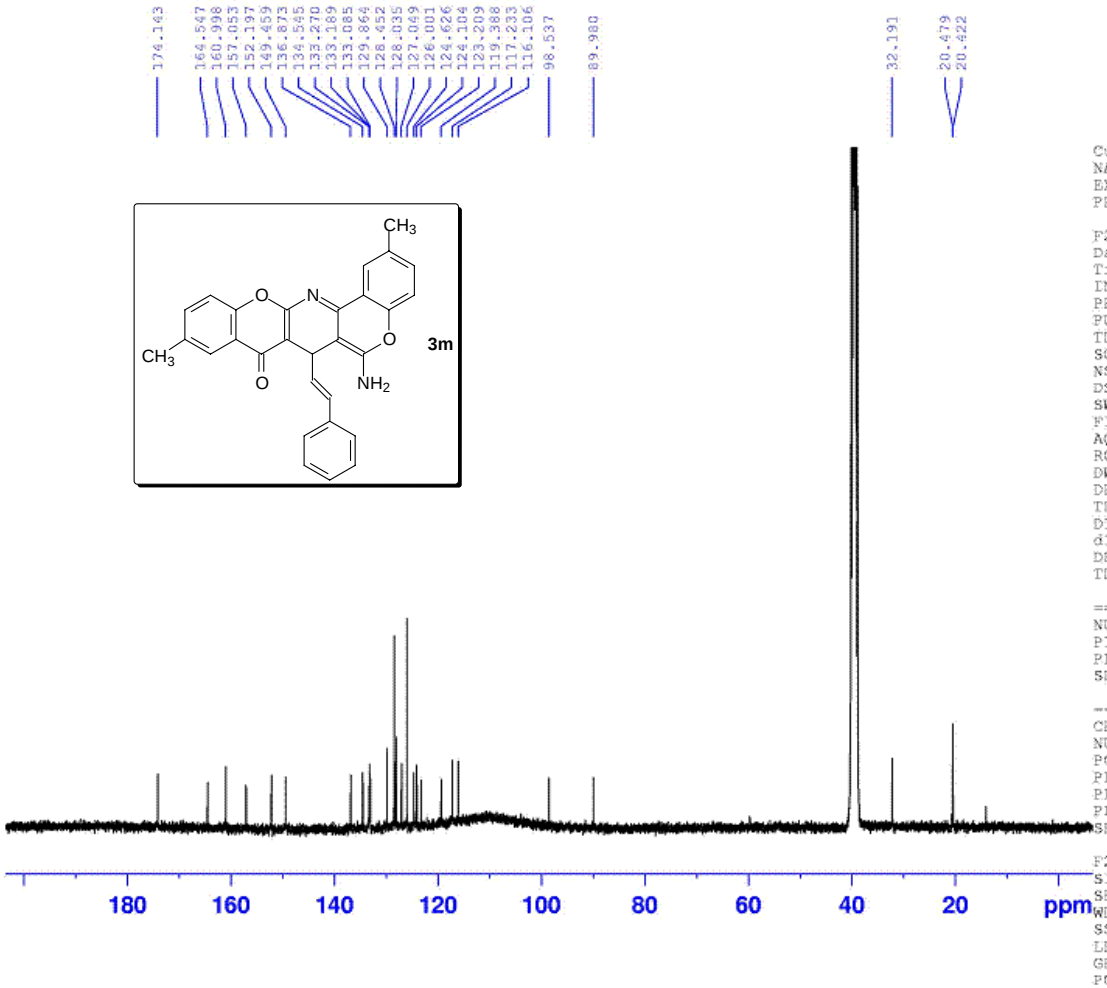

```
Current Data Parameters
NAME          zjlg070706
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070706
Time               15.43
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   203
DW                60.800 usec
DE                 6.00 usec
TE                298.5 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.00 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
```



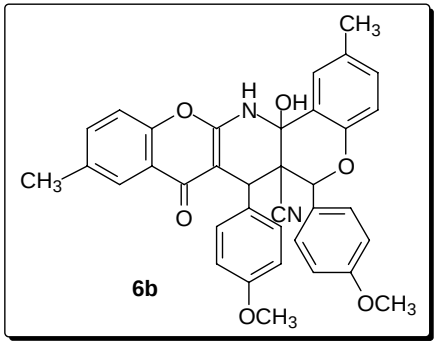

```
Current Data Parameters
NAME          zjlg070706
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070706
Time               15.52
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG         zgpg30
TD                65536
SOLVENT            DMSO
NS                20506
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                32768
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                 14.90 usec
PL1                -3.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               18.50 dB
PL13               18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6128193 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

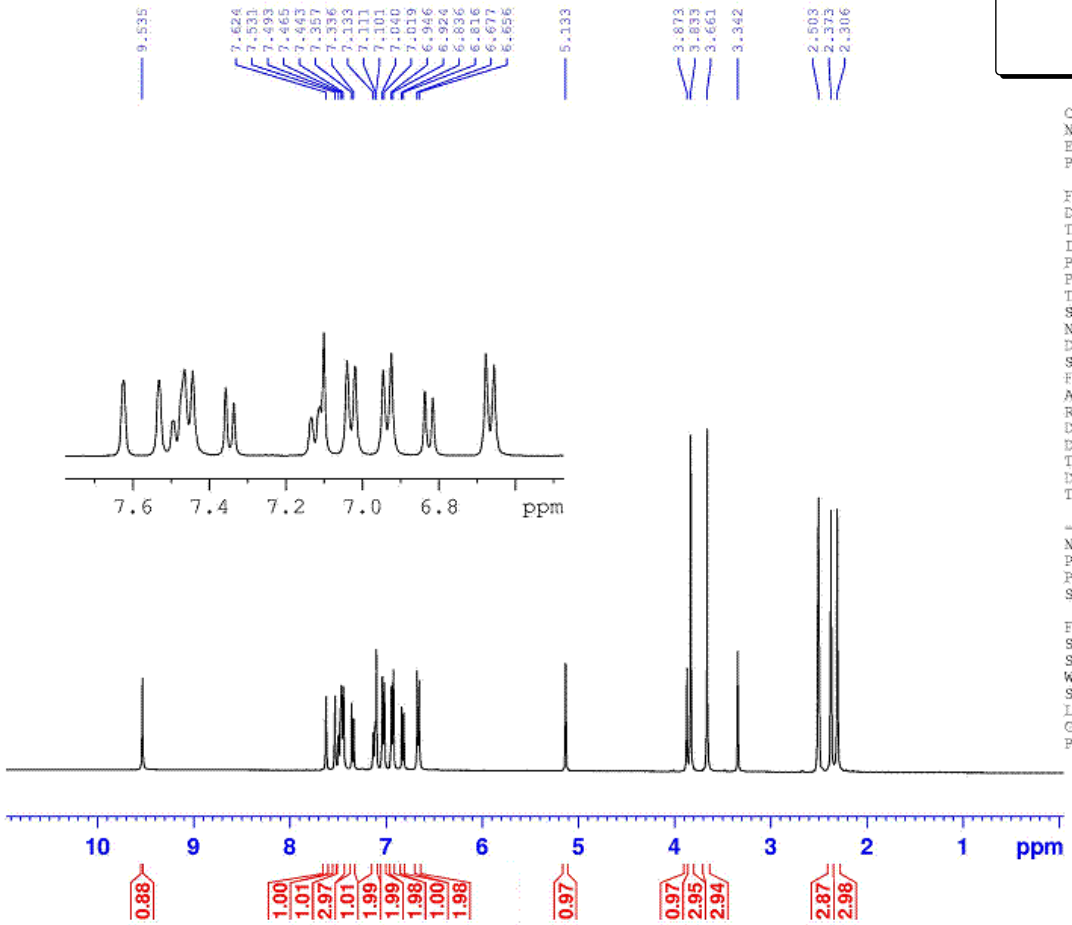



```
Current Data Parameters
NAME          zjlg0707123
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070712
Time                14.53
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   203
DW                60.800 usec
DE                 6.00 usec
TE                296.5 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.00 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
```

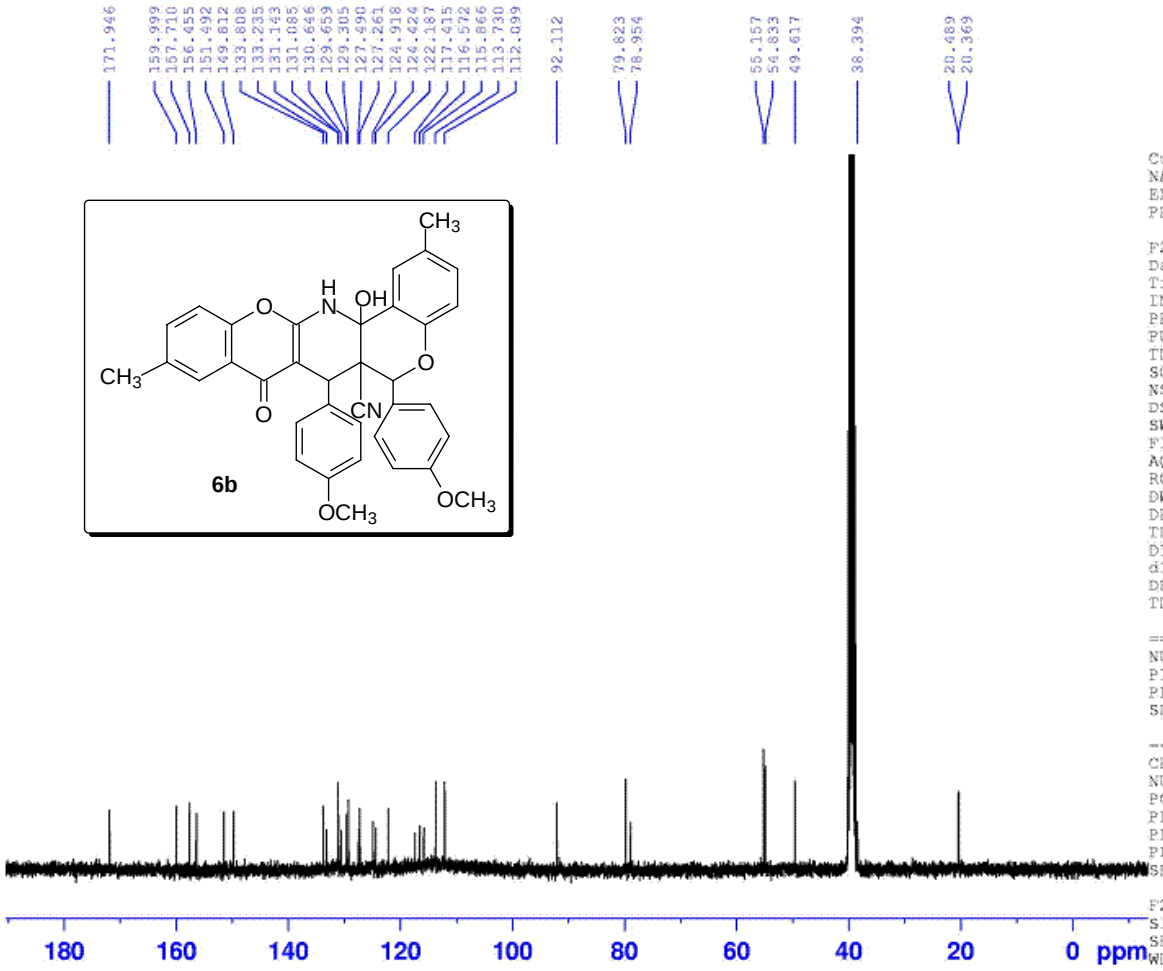



```
Current Data Parameters
NAME          zjlg0707123
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070712
Time                15.32
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG             zgpg30
TD                   65536
SOLVENT               DMSO
NS                   20480
DS                       4
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                      64
DW                 20.800 usec
DE                  6.00 usec
TE                 296.8 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   13C
P1                  14.90 usec
PL1                 -3.00 dB
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                 -2.00 dB
PL12                18.50 dB
PL13                18.50 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                     32768
SF            100.6128193 MHz
WDW                       EM
SSB                        0
LB                   1.00 Hz
GB                         0
PC                    1.40
```

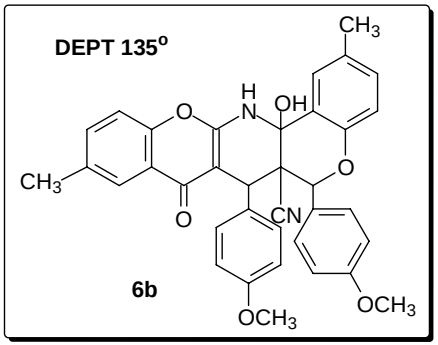




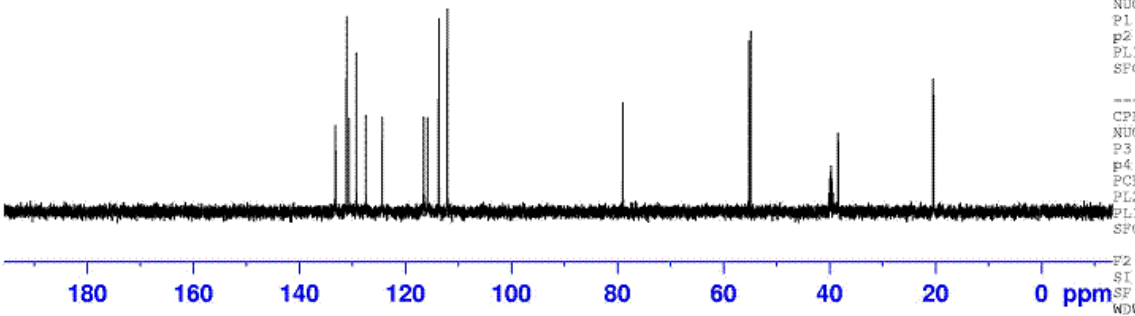

```
Current Data Parameters
NAME          zjlg0710103
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20071010
Time                15.29
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                   144
DW                60.800 usec
DE                 6.00 usec
TE                294.4 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.00 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
```



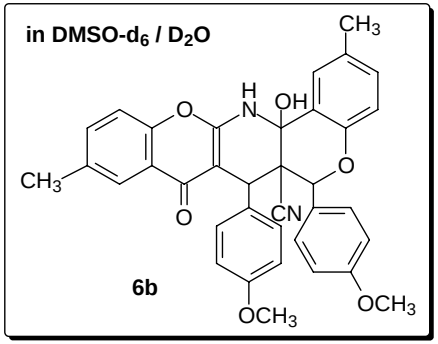

```
Current Data Parameters
NAME         zjlg0710103
EXPNO                 22
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071011
Time                8.59
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT             DMSO
NS                  2650
DS                     4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                  25.4
DW                20.800 usec
DE                 6.00 usec
TE                294.5 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 14.90 usec
PL1                -3.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               18.50 dB
PL13               18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6128193 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

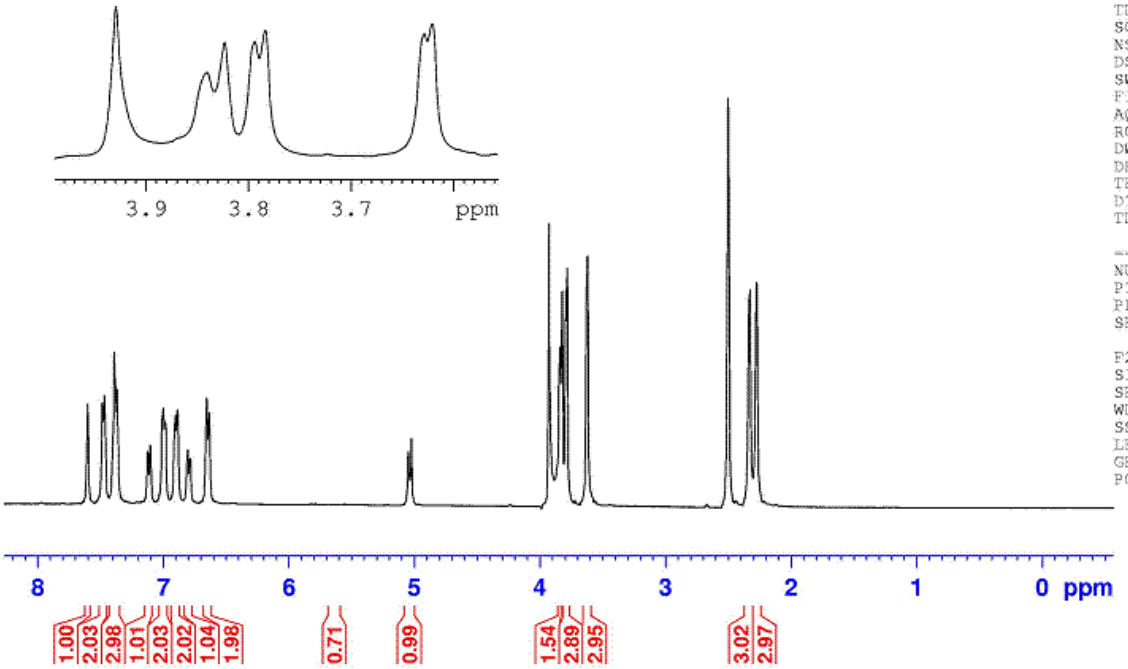


133.228
131.139
130.639
129.300
127.686
124.422
116.563
115.862
113.732
112.100
78.965
55.161
54.837
40.155
39.947
39.743
39.531
39.320
38.402
20.467
20.368

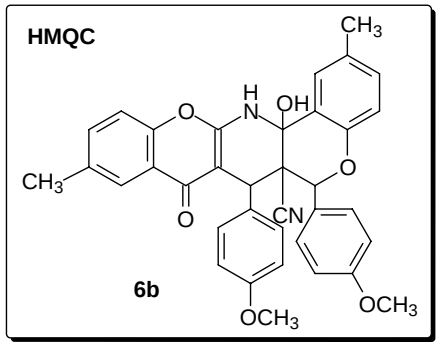


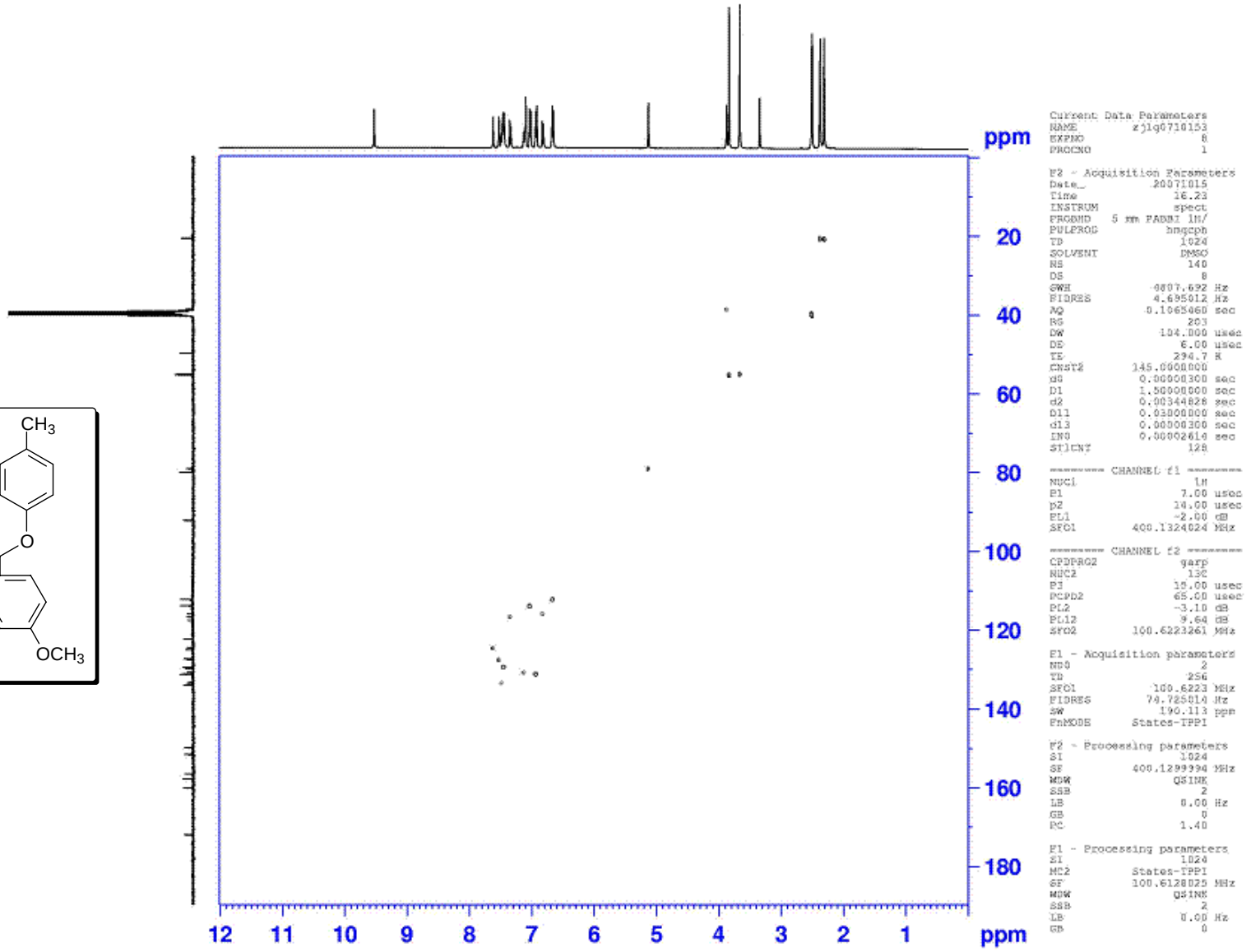

```
Current Data Parameters
NAME          zjlg0710103
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_           20071010
Time               19.24
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG          dept135
TD                 65536
SOLVENT              DMSO
NS                   1600
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
CNST2        145.0000000
D1             2.00000000 sec
d2             0.00344828 sec
d12            0.00002000 sec
DELTA          0.00001897 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  14.90 usec
p2                  29.80 usec
PL1                 -3.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
P3                  7.20 usec
p4                 14.40 usec
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                  32768
SF           100.6128193 MHz
WDW                    EM
SSB                     0
LB                  1.00 Hz
GB                      0
PC                   1.40
```





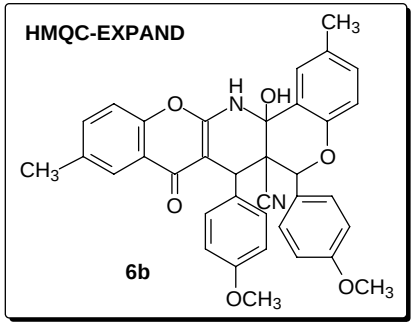

```
Current Data Parameters
NAME                 zjlg0710111
EXPNO                         11
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20071011
Time                       12.06
INSTRUM                    spect
PROBHD    5 mm PABBI 1H/
PULPROG                    zg30
TD                        65536
SOLVENT                    DMSO
NS                            16
DS                             2
SWH                     8223.685 Hz
FIDRES                  0.125483 Hz
AQ                      3.9846387 sec
RG                           161
DW                        60.800 usec
DE                         6.00 usec
TE                        294.4 K
D1                     1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                         1H
P1                         7.00 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
```



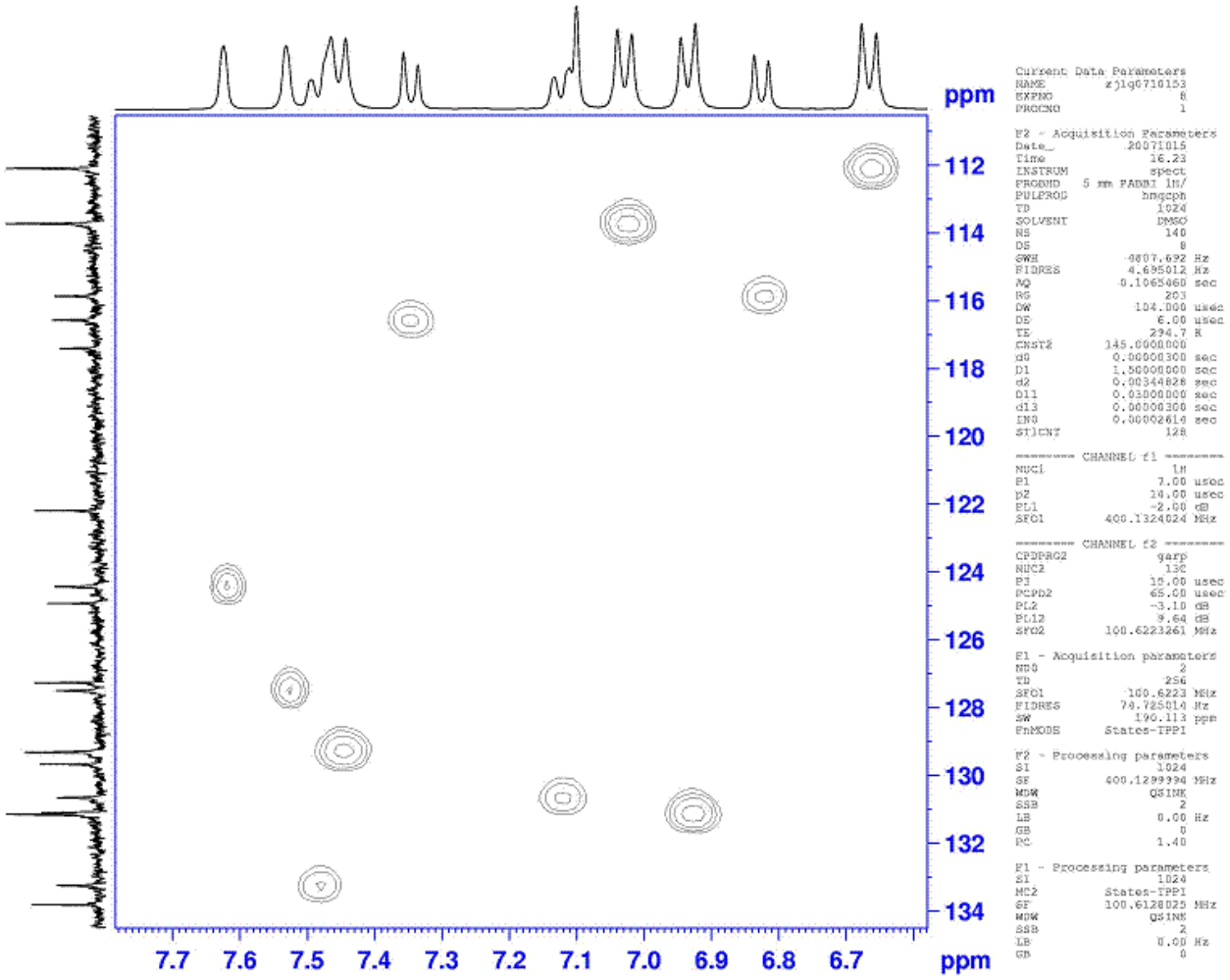



```
Current Data Parameters
NAME                 zjlg0710153
EXPNO                          8
PROCNO                         1

F2 - Acquisition Parameters
Date_               20071015
Time                     16.23
INSTRUM                  spect
PROBHD     5 mm PABBI 1H/
PULPROG                  hmqcph
TD                         1024
SOLVENT                    DMSO
NS                          140
DS                            8
SWH                 6807.692 Hz
FIDRES              6.645512 Hz
AQ                  0.1065460 sec
RG                          203
DW                    104.000 usec
DE                      6.00 usec
TE                     294.7 K
CNST2               145.0000000
d0                  0.00000300 sec
D1                  1.50000000 sec
d2                  0.00344828 sec
D11                 0.03000000 sec
d13                 0.00000300 sec
IN0                 0.00002610 sec
ST1CNT                       128

======== CHANNEL f1 ========
NUC1                         1H
P1                        7.00 usec
P2                       14.00 usec
PL1                      -2.00 dB
SFO1                400.1324024 MHz

======== CHANNEL f2 ========
CPDPRG2                   garp
NUC2                       13C
P3                       15.00 usec
PCPD2                    65.00 usec
PL2                      -3.10 dB
PL12                      9.64 dB
SFO2                100.6223261 MHz

F1 - Acquisition parameters
ND0                          2
TD                         256
SFO1                100.6223 MHz
FIDRES              78.725814 Hz
SW                    190.113 ppm
FnMODE              States-TPPI

F2 - Processing parameters
SI                        1024
SF                  400.1299994 MHz
WDW                       QSINE
SSB                          2
LB                        0.00 Hz
GB                           0
PC                        1.40

F1 - Processing parameters
SI                        1024
MC2                 States-TPPI
SF                  100.6128025 MHz
WDW                       QSINE
SSB                          2
LB                        0.00 Hz
GB                           0
```

HMQC-EXPAND

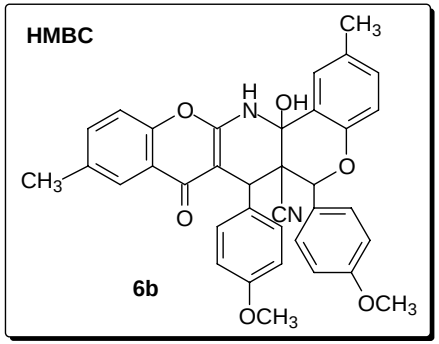


6b

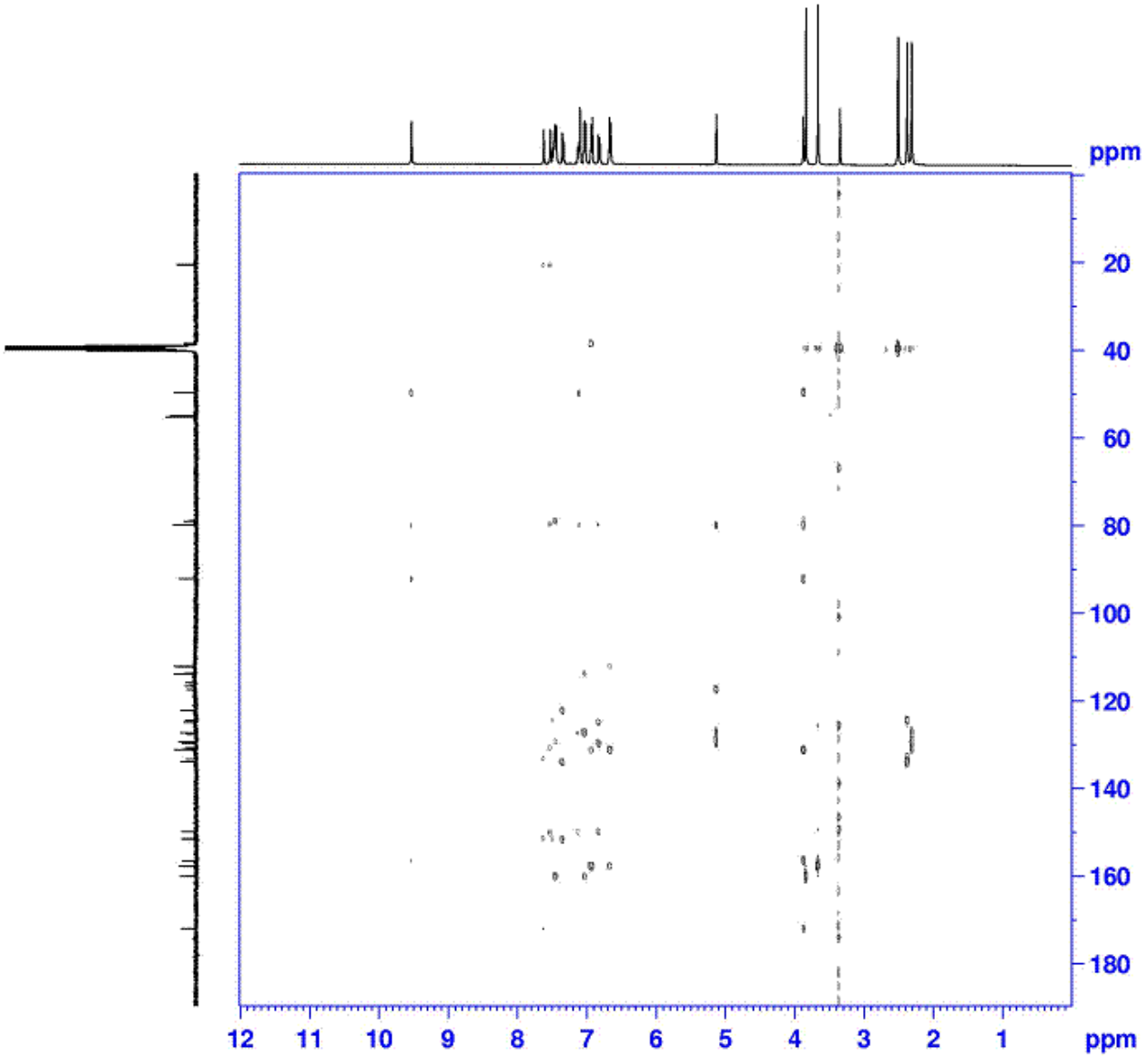



```
Current Data Parameters
NAME                 zjlq0710153
EXPNO                          8
PROCNO                         1

F2 - Acquisition Parameters
Date_               20071015
Time                     16.23
INSTRUM                  spect
PROBHD    5 mm PABBI 1H/
PULPROG                hmqcph
TD                       1024
SOLVENT                  DMSO
NS                        140
DS                          8
SWH                 9807.692 Hz
FIDRES              4.695012 Hz
AQ                  0.1065460 sec
RG                        203
DW                    104.000 usec
DE                      6.00 usec
TE                     294.7 K
CNST2               145.0000000
d0                  0.00000300 sec
D1                  1.50000000 sec
d2                  0.00344828 sec
D11                 0.03000000 sec
d13                 0.00000300 sec
IN0                 0.00002614 sec
ST1CNT                    128

======== CHANNEL f1 ========
NUC1                       1H
P1                       7.00 usec
pZ                      14.00 usec
PL1                     -2.00 dB
SFO1                400.1324024 MHz

======== CHANNEL f2 ========
CPDPRG2                  garp
NUC2                      13C
P3                      15.00 usec
PCPD2                   65.00 usec
PL2                     -3.10 dB
PL12                     9.64 dB
SFO2                100.6223261 MHz

F1 - Acquisition parameters
NS                         2
TD                       256
SFO1                100.6223 MHz
FIDRES              74.785014 Hz
SW                    190.113 ppm
FnMODE           States-TPPI

F2 - Processing parameters
SI                       1024
SF                  400.1299994 MHz
WDW                      QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
PC                       1.40

F1 - Processing parameters
SI                       1024
MC2              States-TPPI
SF                  100.6128025 MHz
WDW                      QSINE
SSB                         2
LB                       0.00 Hz
GB                          0
```

HMBC

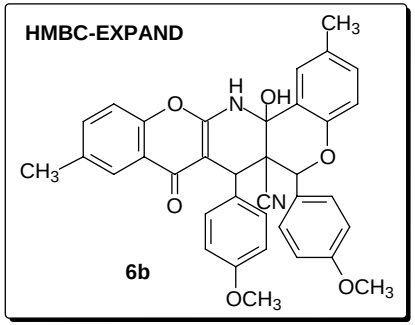


6b

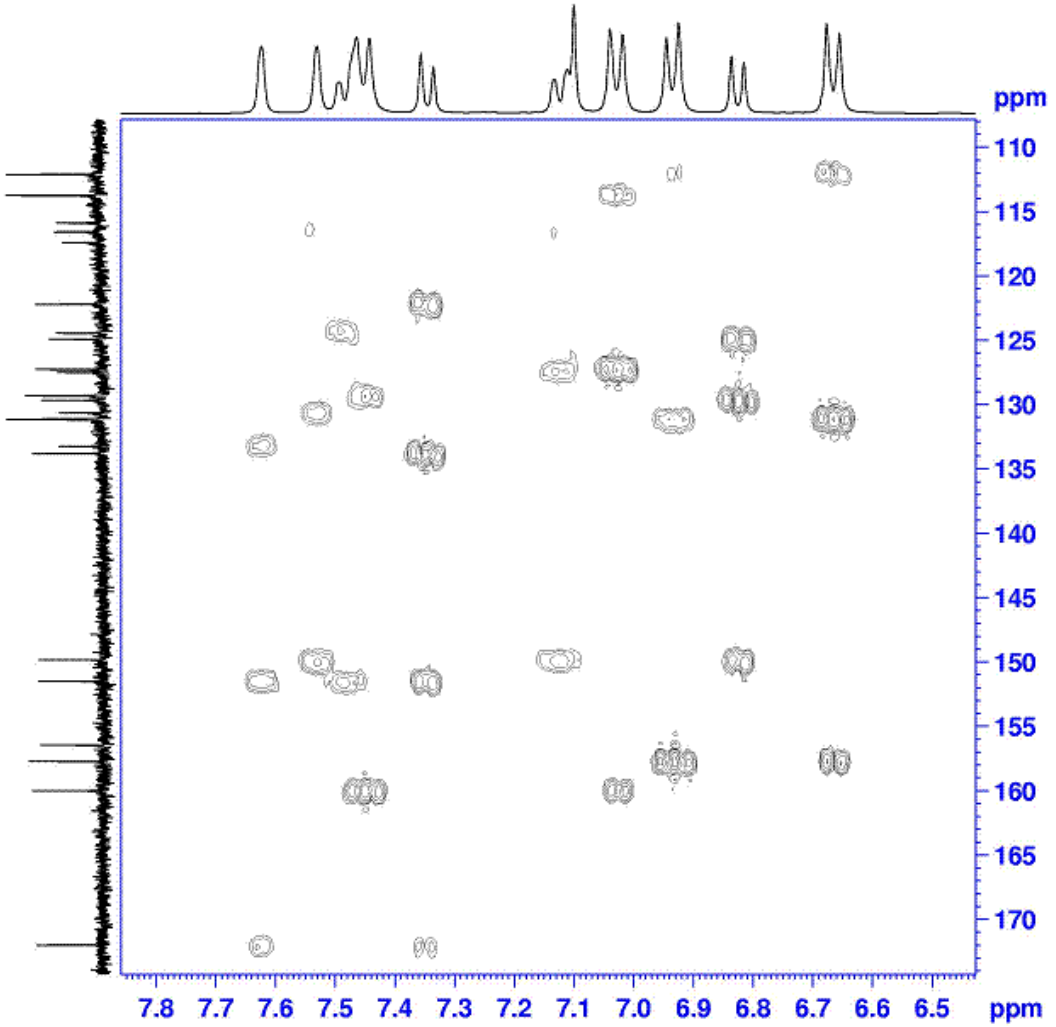



```
Current Data Parameters
NAME                  zjlg0710162
EXPNO                           9
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20071016
Time                        16.47
INSTRUM                      spect
PROBHD    5 mm PABBI 1H/
PULPROG                hmbclprndgf
TD                             4096
SOLVENT                        DMSO
NS                              640
DS                               16
SWH                      4807.692 Hz
FIDRES                   1.173753 Hz
AQ                       0.4260340 sec
RG                             287
DW                         104.000 usec
DE                           6.00 usec
TE                           293.7 K
CNST2                  145.0000000
CNST13                   8.0000000
d0                       0.00000300 sec
D1                       1.50000000 sec
d2                       0.00344828 sec
d5                       0.06250000 sec
JN0                      0.00002614 sec

======== CHANNEL f1 ========
NUC1                           1H
P1                           7.00 usec
p2                          14.00 usec
PL1                         -2.00 dB
SFO1                  400.1324024 MHz

======== CHANNEL f2 ========
NUC2                           13C
P3                          15.00 usec
PL2                         -3.10 dB
SFO2                  100.6223261 MHz

F1 - Acquisition parameters
ND0                             2
TD                            127
SFO1                    100.6223 MHz
FIDRES                150.626801 Hz
SW                      190.113 ppm
FnMODE                         QF

F2 - Processing parameters
SI                            2048
SF                    400.1299979 MHz
WDW                         SINE
SSB                            0
LB                          0.00 Hz
GB                             0
PC                          1.40

F1 - Processing parameters
SI                            1024
MC2                            QF
SF                    100.6128097 MHz
WDW                         SINE
SSB                            0
LB                          0.00 Hz
GB                             0
```

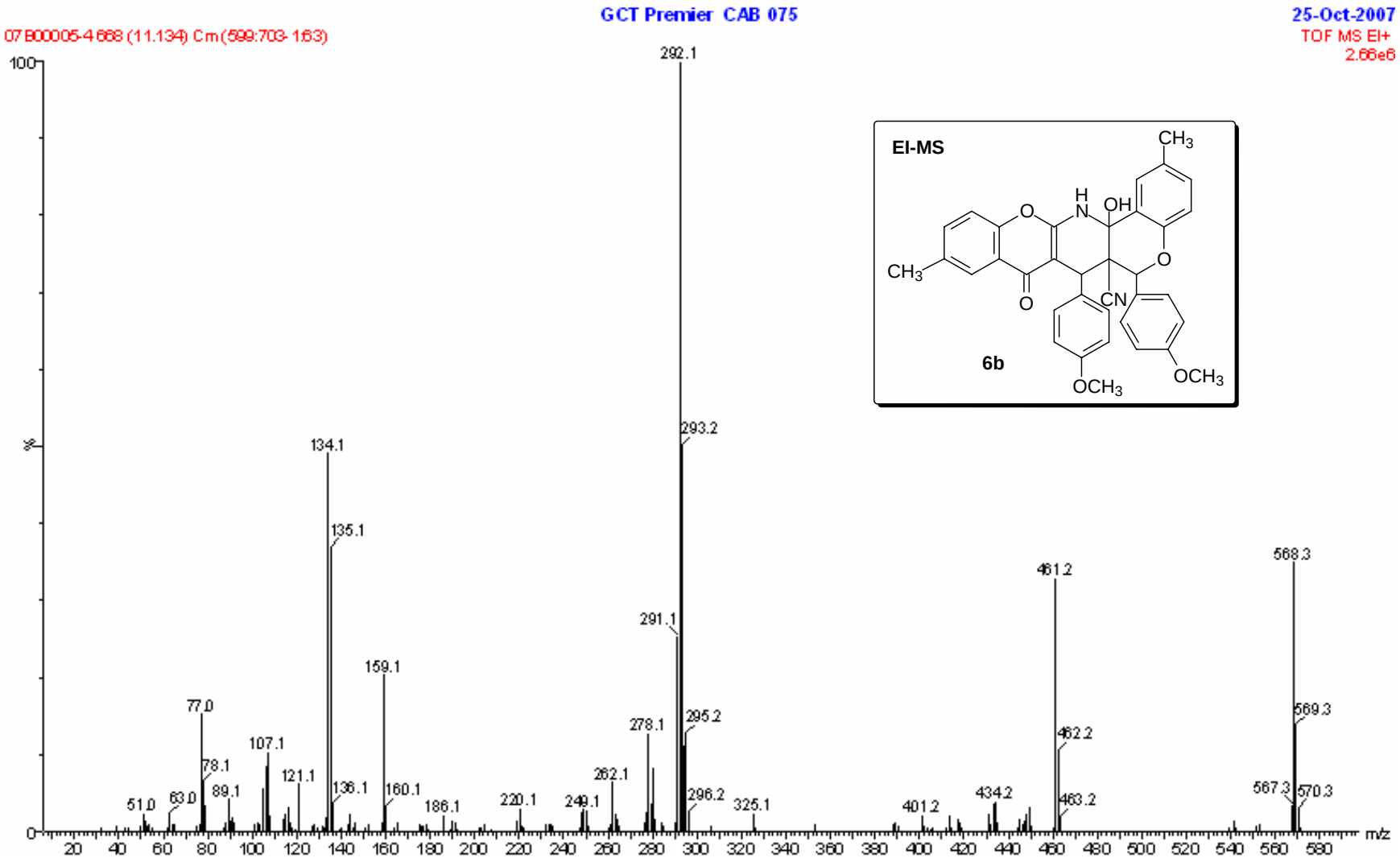
HMBC-EXPAND

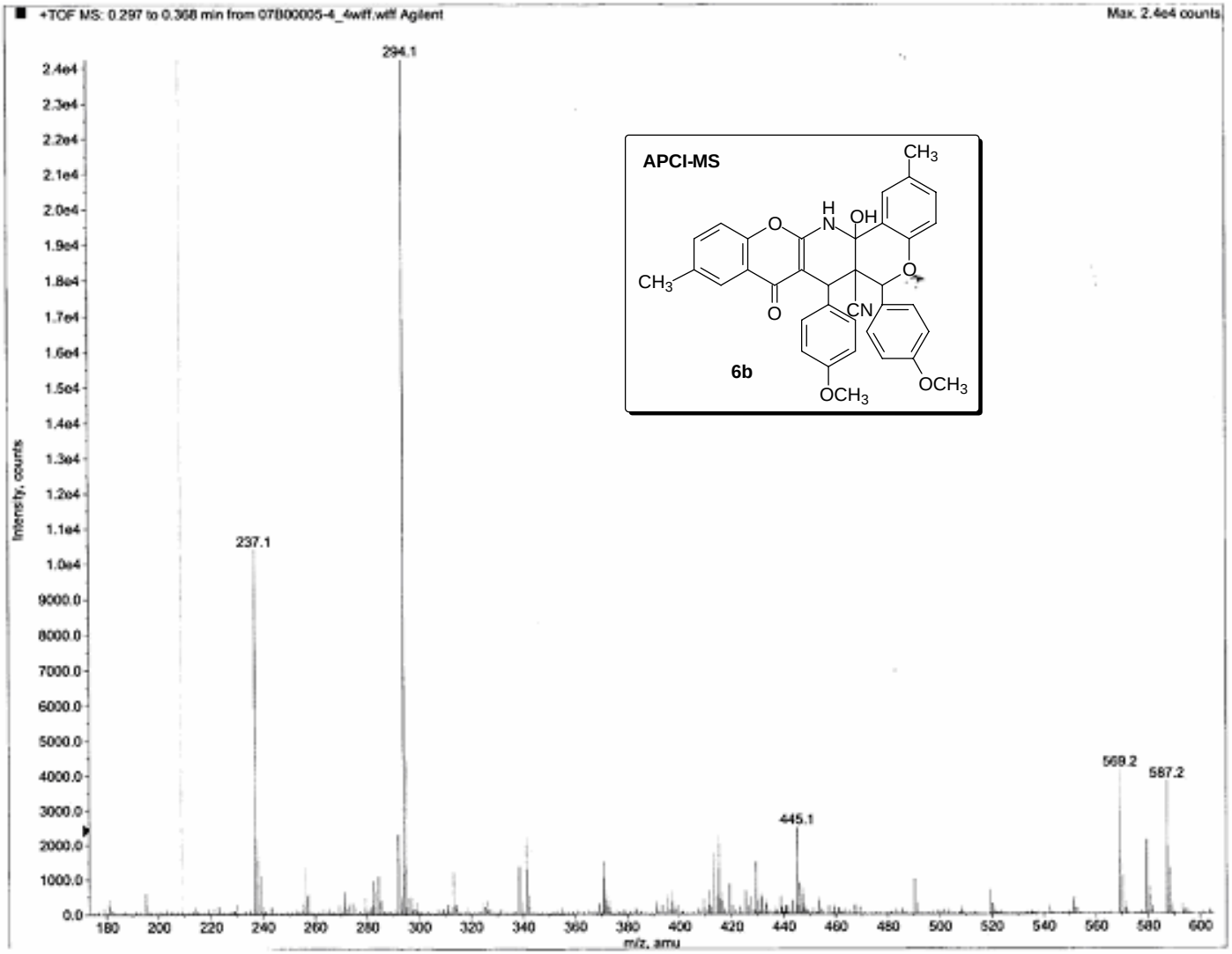


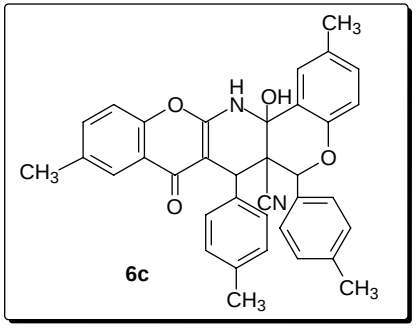
6b

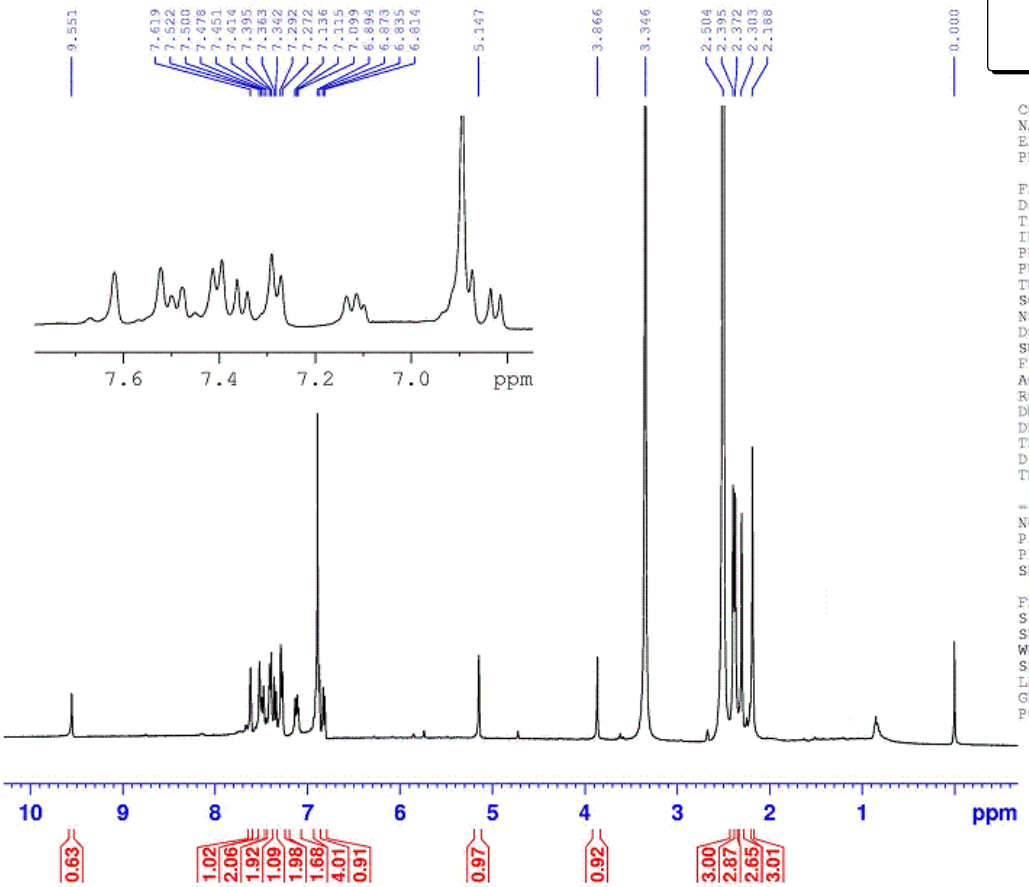



```
Current Data Parameters
NAME          zjlg0710162
EXPNO                   9
PROCNO                  1

F2 - Acquisition Parameters
Date_            20071016
Time                16.47
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG      hmbclprndqf
TD                  4096
SOLVENT             DMSO
NS                   640
DS                    16
SWH              4807.692 Hz
FIDRES           1.173753 Hz
AQ               0.4260340 sec
RG                    287
DW                104.000 usec
DE                  6.00 usec
TE                293.7 K
CNST2          145.0000000
CNST13           8.0000000
d0               0.00000300 sec
D1               1.50000000 sec
d2               0.00344828 sec
d5               0.06250000 sec
JNN              0.00002614 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                    7.00 usec
p2                   14.00 usec
PL1                  -2.00 dB
SFO1          400.1324024 MHz

======== CHANNEL f2 ========
NUC2                   13C
P3                   15.00 usec
PL2                  -3.10 dB
SFO2          100.6223261 MHz

F1 - Acquisition parameters
ND0                     2
TD                    127
SFO1          100.6223 MHz
FIDRES        150.626801 Hz
SW               190.113 ppm
FnMODE                  QF

F2 - Processing parameters
SI                    2048
SF            400.1299979 MHz
WDW                   SINE
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.40

F1 - Processing parameters
SI                    1024
MC2                     QF
SF            100.6128097 MHz
WDW                   SINE
SSB                      0
LB                    0.00 Hz
GB                       0
```

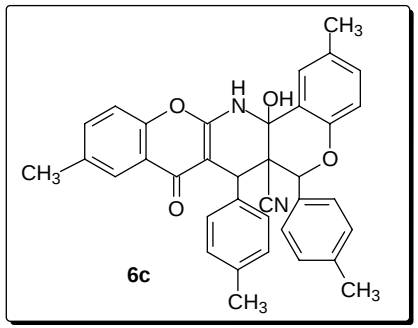

GCT Premier CAB 075
25-Oct-2007
TOF MS EI+
2.66e6
07B00005-4 668 (11.134) Cm (599:703-163)
EI-MS
6b
292.1
293.2
134.1
135.1
291.1
159.1
568.3
461.2
569.3
77.0
295.2
278.1
462.2
107.1
78.1
121.1
262.1
136.1
160.1
89.1
296.2
567.3
570.3
434.2
51.0
63.0
186.1
220.1
249.1
325.1
401.2
463.2
%
m/z

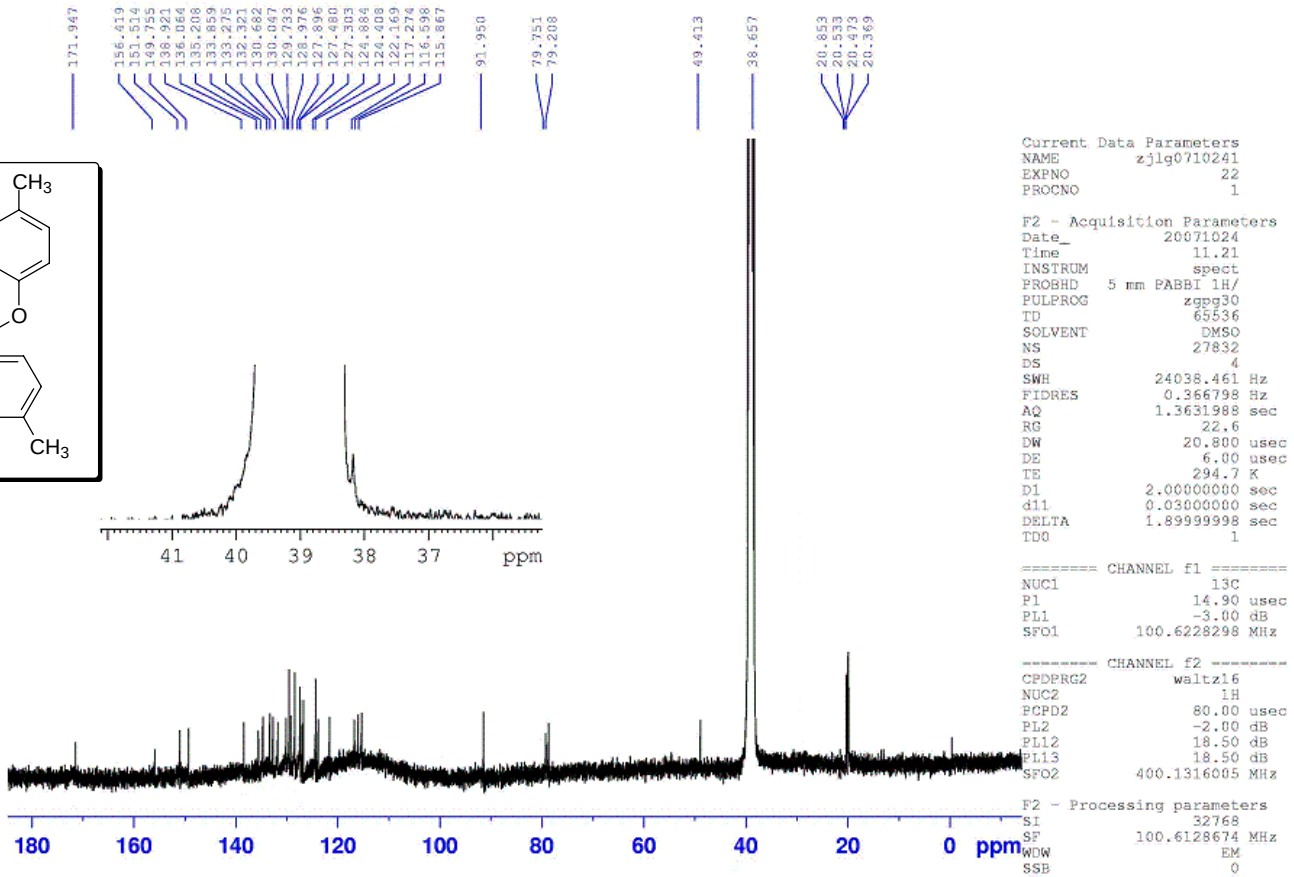

+TOF MS: 0.297 to 0.368 min from 07B00005-4_4wtf.wtf Agilent
Max. 2.4e4 counts
Intensity, counts
m/z, amu
294.1
237.1
445.1
569.2
587.2
APCI-MS
6b



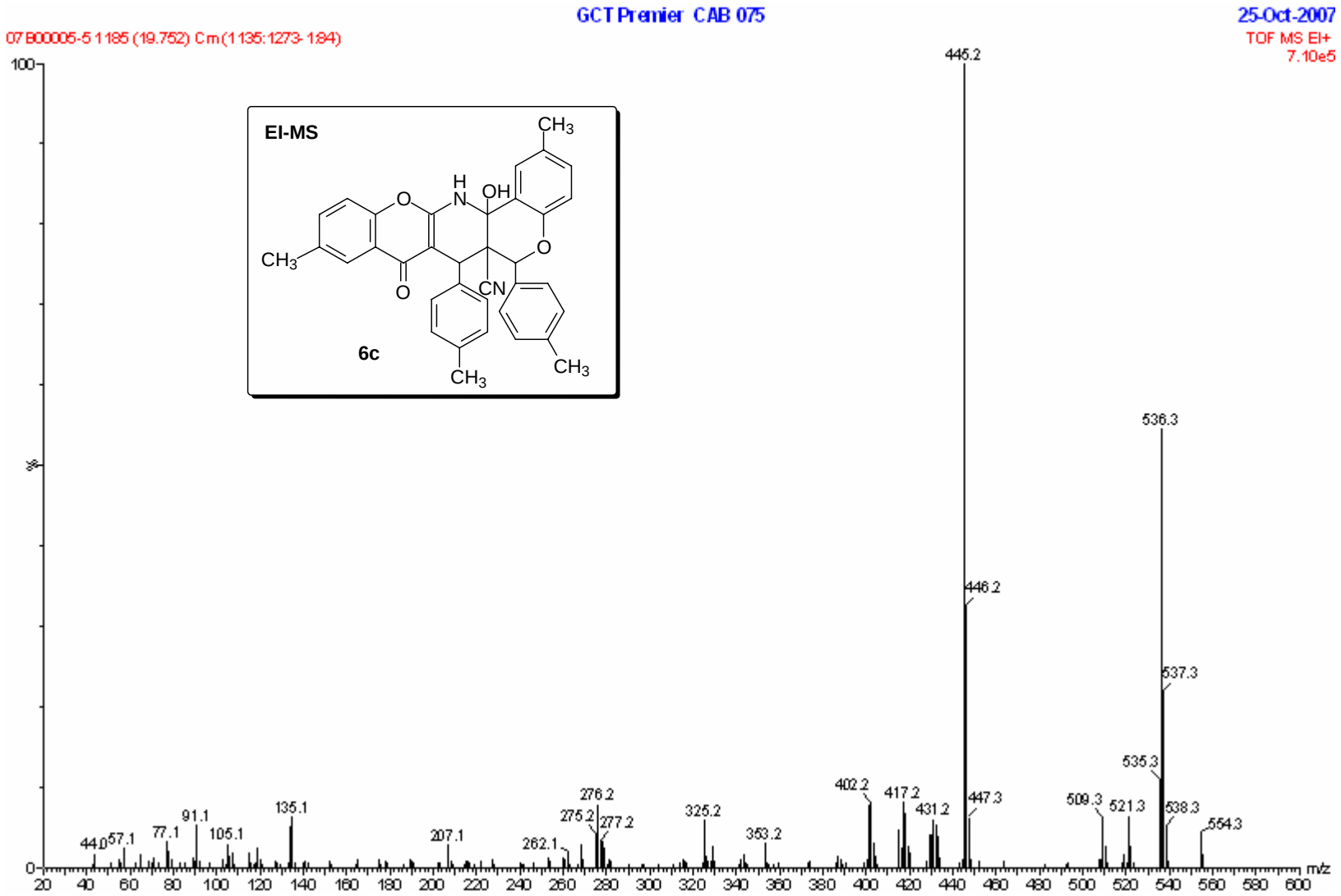



```
Current Data Parameters
NAME          zjlg0710241
EXPNO                  11
PROCNO                  1

F2 - Acquisition Parameters
Date_            20071024
Time                9.08
INSTRUM            spect
PROBHD    5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT             DMSO
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   101
DW                60.800 usec
DE                 6.00 usec
TE                294.3 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.00 usec
PL1               -2.00 dB
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                 32768
SF           400.1299987 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```






```
Current Data Parameters
NAME         zjlg0710241
EXPNO                 22
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071024
Time               11.21
INSTRUM            spect
PROBHD   5 mm PABBI 1H/
PULPROG         zgpg30
TD                65536
SOLVENT            DMSO
NS                27832
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                 22.6
DW               20.800 usec
DE                6.00 usec
TE               294.7 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  13C
P1                 14.90 usec
PL1                -3.00 dB
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               18.50 dB
PL13               18.50 dB
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6128674 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


GCT Premier CAB 075
25-Oct-2007
07B00005-5 1185 (19.752) Cm (1135:1273-184)
TOF MS EI+
7.10e5
EI-MS
6c
100
%
0
m/z
445.2
536.3
446.2
537.3
535.3
402.2
417.2
276.2
275.2
277.2
325.2
431.2
447.3
509.3
521.3
538.3
554.3
135.1
91.1
105.1
77.1
57.1
44.0
207.1
262.1
353.2
20 40 60 80 100 120 140 160 180 200 220 240 260 280 300 320 340 360 380 400 420 440 460 480 500 520 540 560 580 600

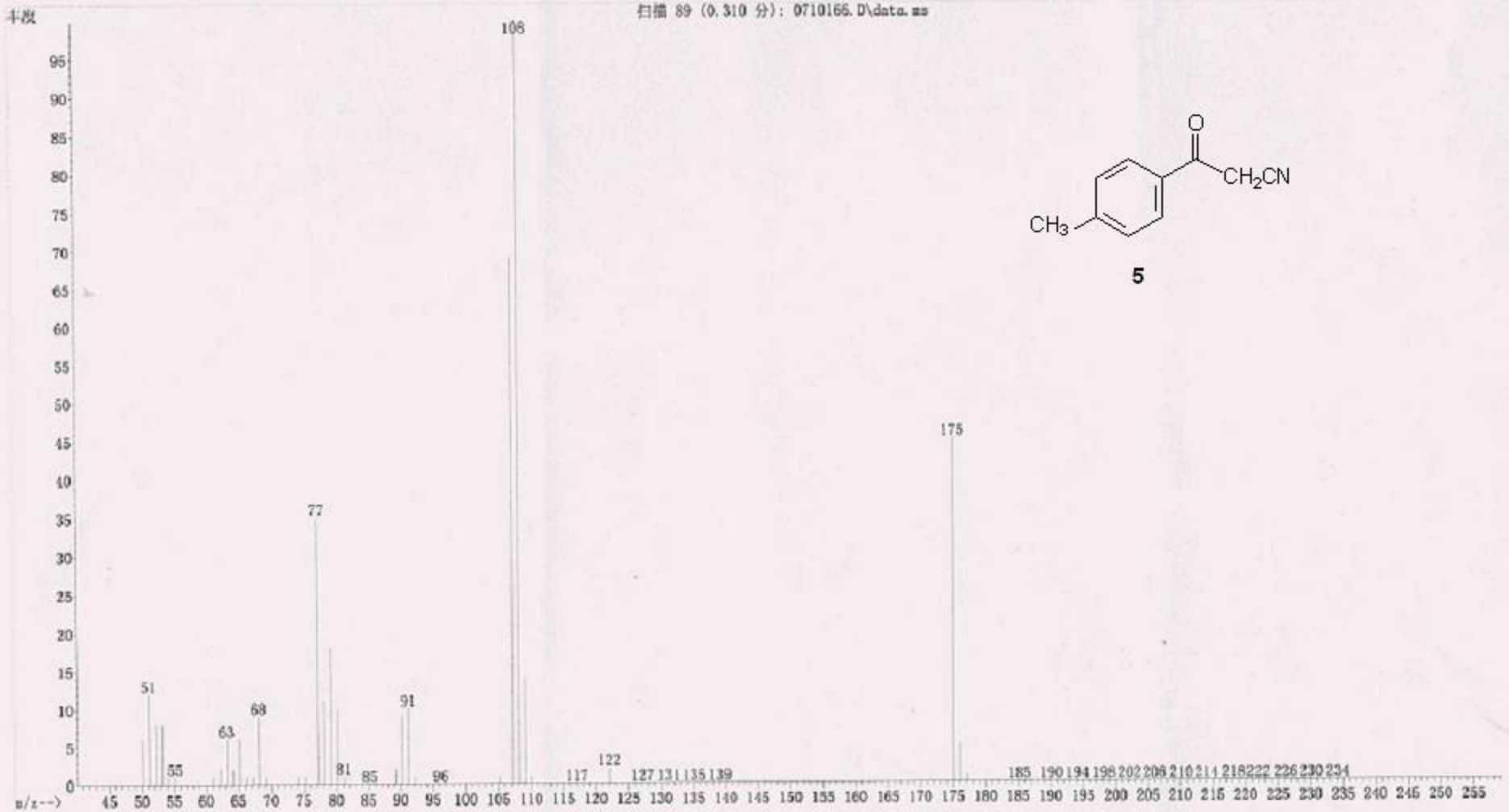
Base-catalyzed and Solvent-dependent Cascade Reaction in Regioselective Synthesis of Novel Fused Polycycles

Min Xia*, Guo-Feng Xiang, Bin Wu, Yi-Feng Han

Department of Chemistry, Zhejiang Sci-Tech University, Hangzhou 310018, P. R. China
Phone/fax: +86(571)86843224; E-mail: xiamin@zstu.edu.cn

Contents:

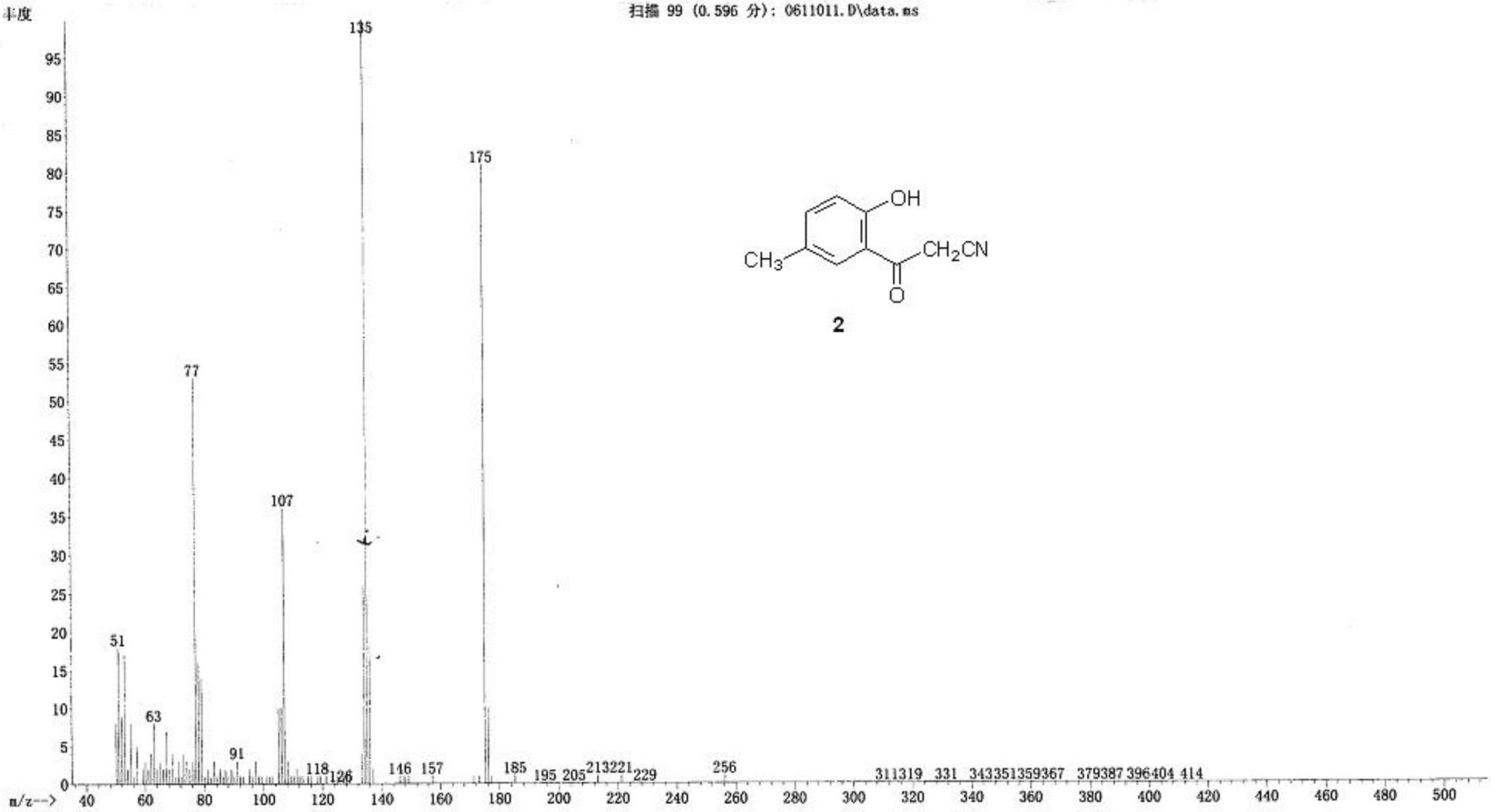
文件　　: D:\msdchem\1\DATA\071016\0710166.D
操作员　:
已采集　: 16 Oct 2007　12:31　　, 使用采集方法 DIPTEST.M
仪器:　仪器 1
样品名: XM-1
其他信息　: MW=175
样品瓶号: 1



5

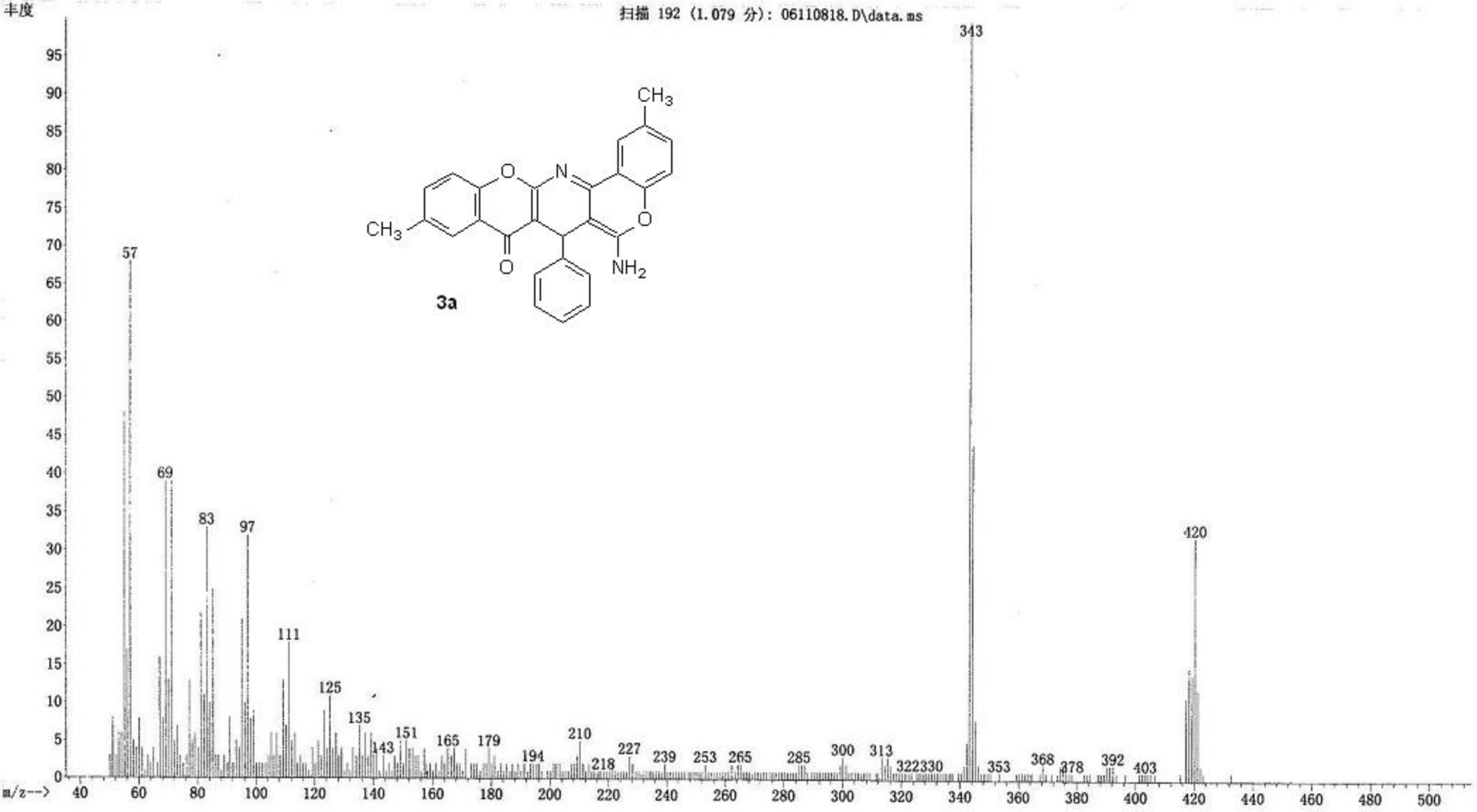
文件　　：D:\msdchem\1\DATA\061101\0611011.D
操作员　：
已采集　：1 Nov 2006　9:41　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：xm
其他信息　：MW=165
样品瓶号：1

扫描 99 (0.596 分)：0611011.D\data.ms

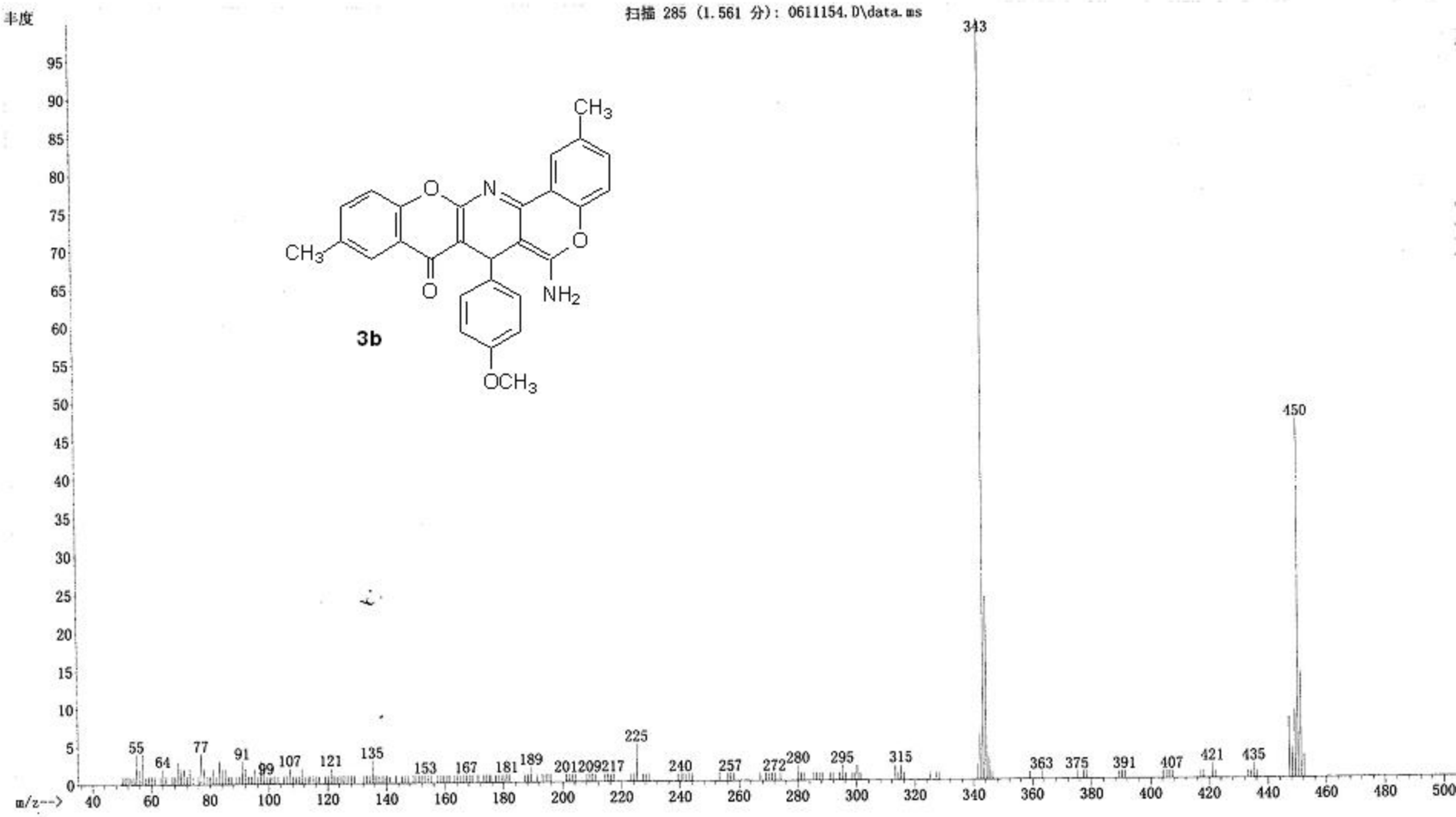


2

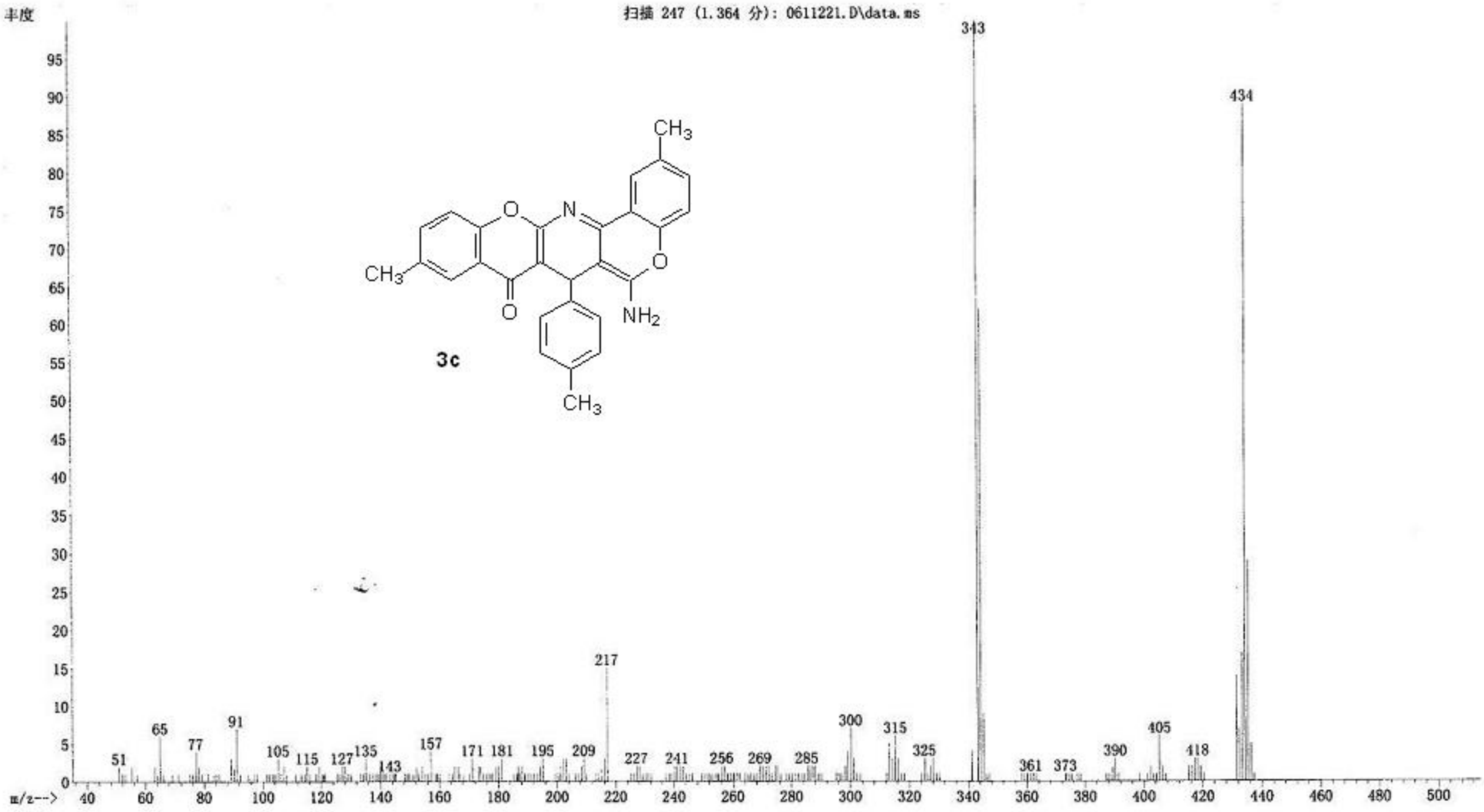
文件　　：D:\msdchem\1\DATA\061108\06110818.D
操作员　：
已采集　：8 Nov 2006　14:00　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：XM
其他信息　：MW=
样品瓶号：1



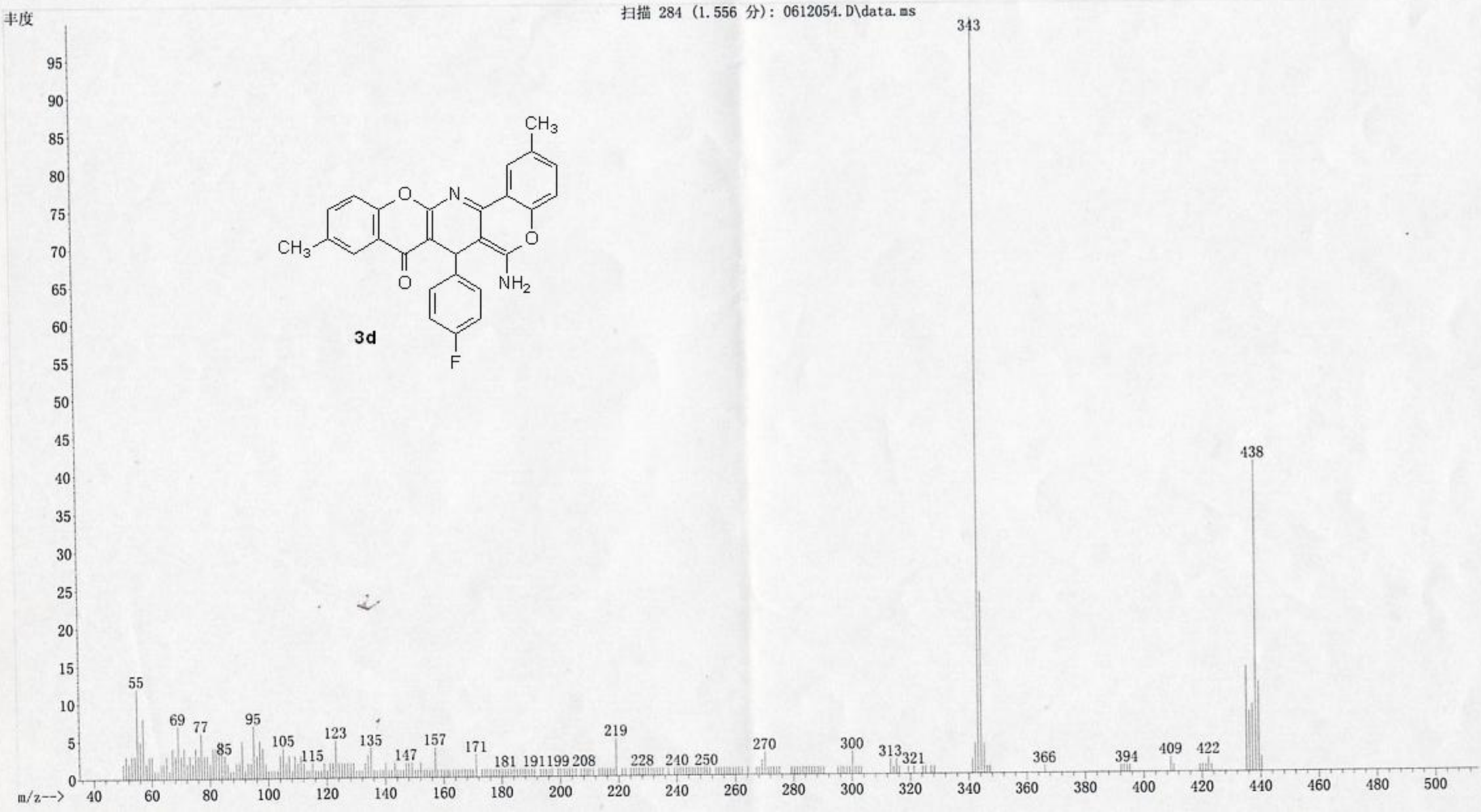
操作员 :
已采集 : 15 Nov 2006 10:29 , 使用采集方法 DIPTEST.M
仪器: 仪器 1
样品名: XM
其他信息 : MW=
样品瓶号: 1



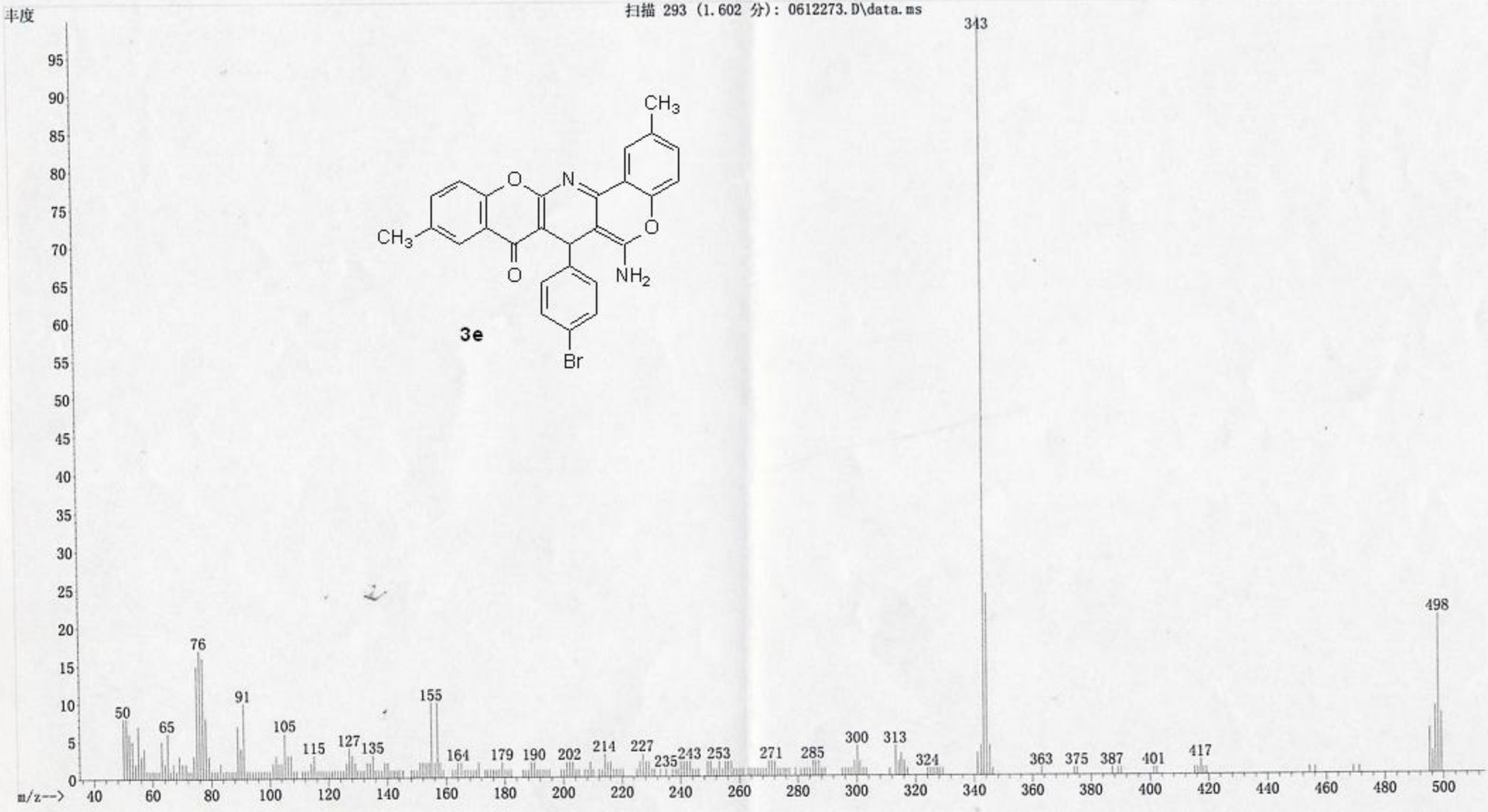
文件　　：D:\msdchem\1\DATA\061122\0611221.D
操作员　：
已采集　：22 Nov 2006　9:50　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：xm
其他信息　：MW=
样品瓶号：1



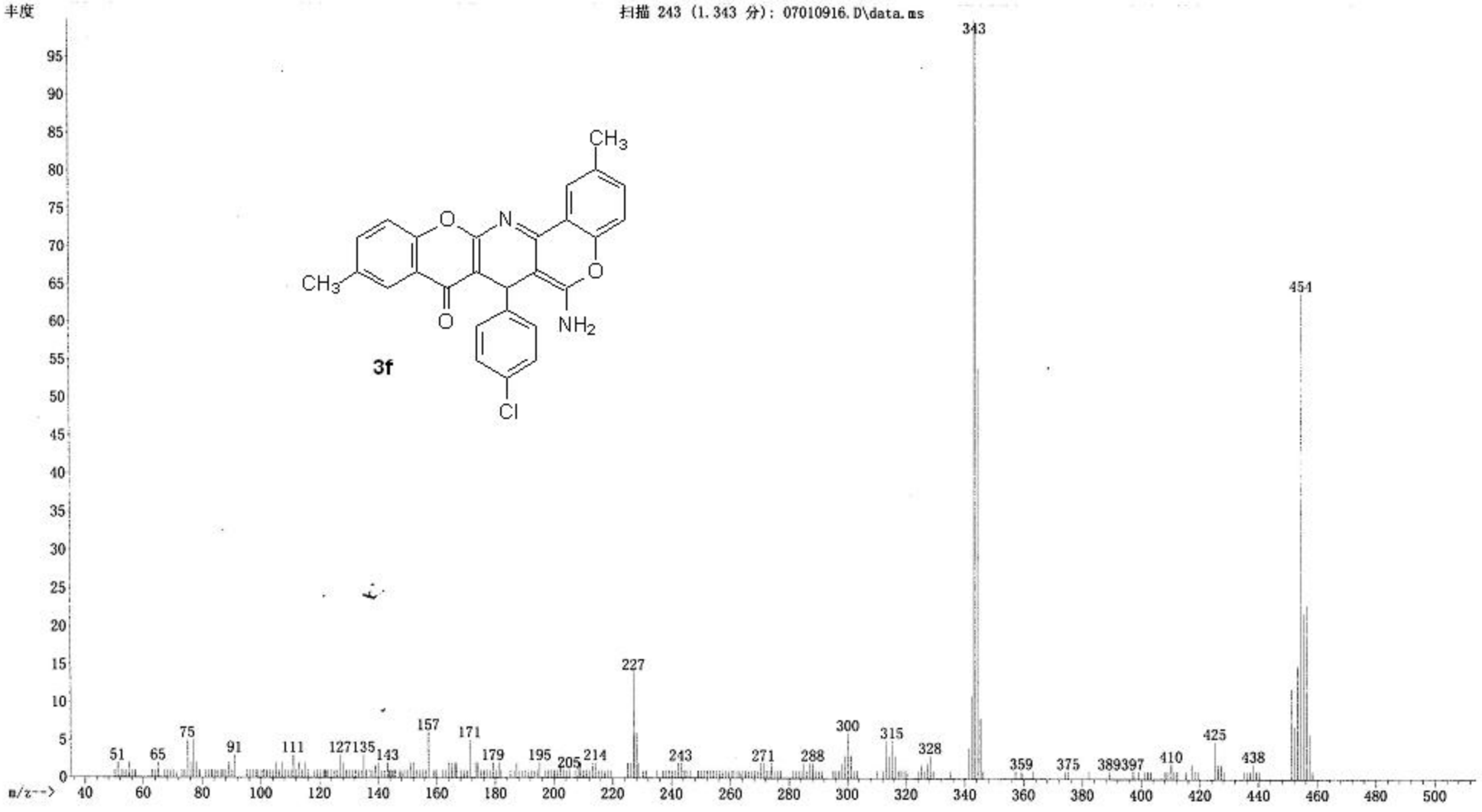
文件　　：D:\msdchem\1\DATA\061205\0612054.D
操作员　：
已采集　：5 Dec 2006　13:51　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：XM-1
其他信息　：MW=
样品瓶号：1



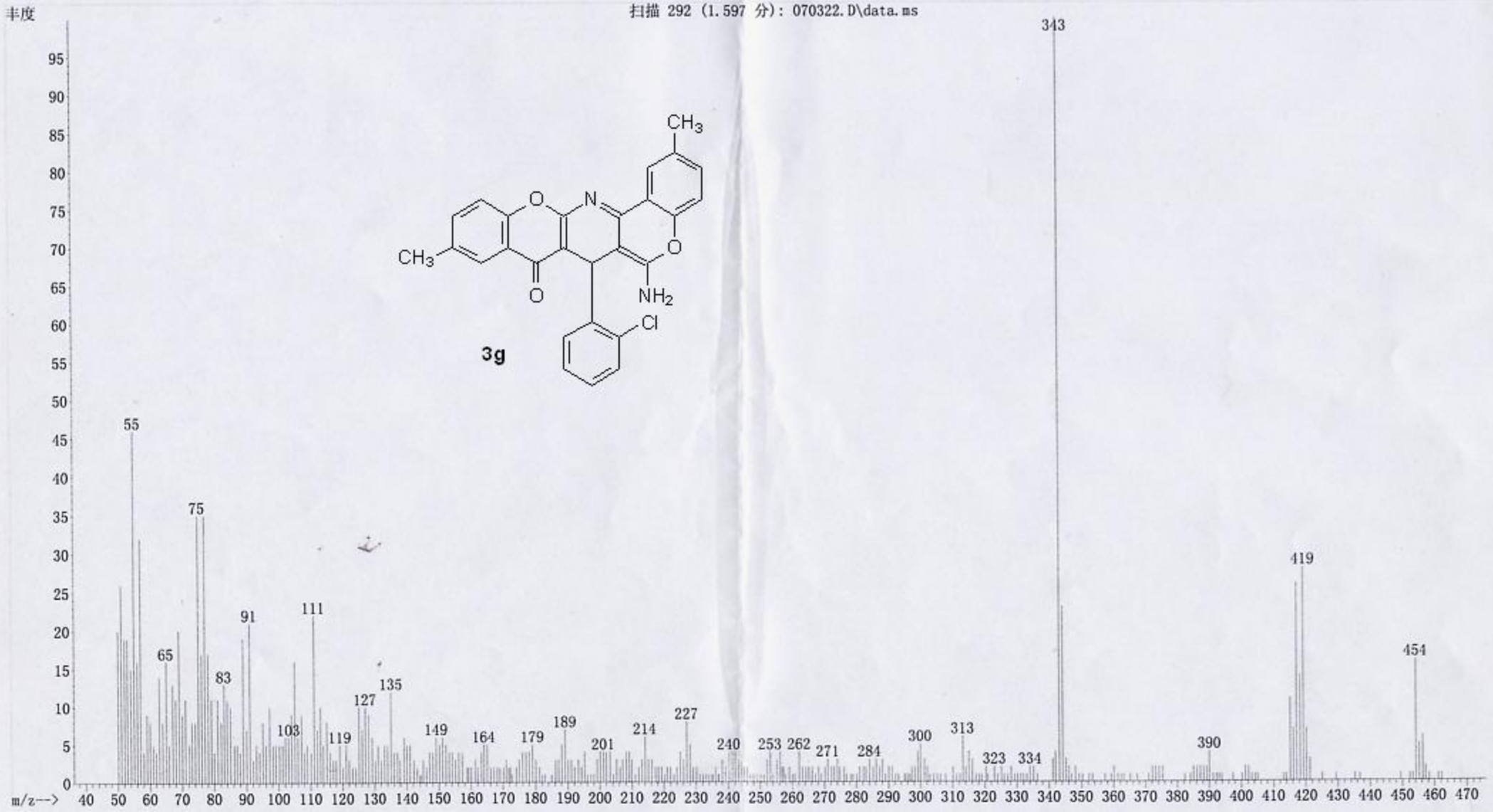
文件　　：D:\msdchem\1\DATA\061227\0612273.D
操作员　：
已采集　：27 Dec 2006　10:06　　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：XM-1
其他信息　：MW=
样品瓶号：1



文件　　：D:\msdchem\1\DATA\070109\07010916.D
操作员　：
已采集　：9 Jan 2007　15:47　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：XM-3
其他信息　：MW=454
样品瓶号：1



文件　　：D:\msdchem\1\DATA\070322\070322.D
操作员　：
已采集　：22 Mar 2007　11:09　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：XM
其他信息　：MW=454
样品瓶号：1

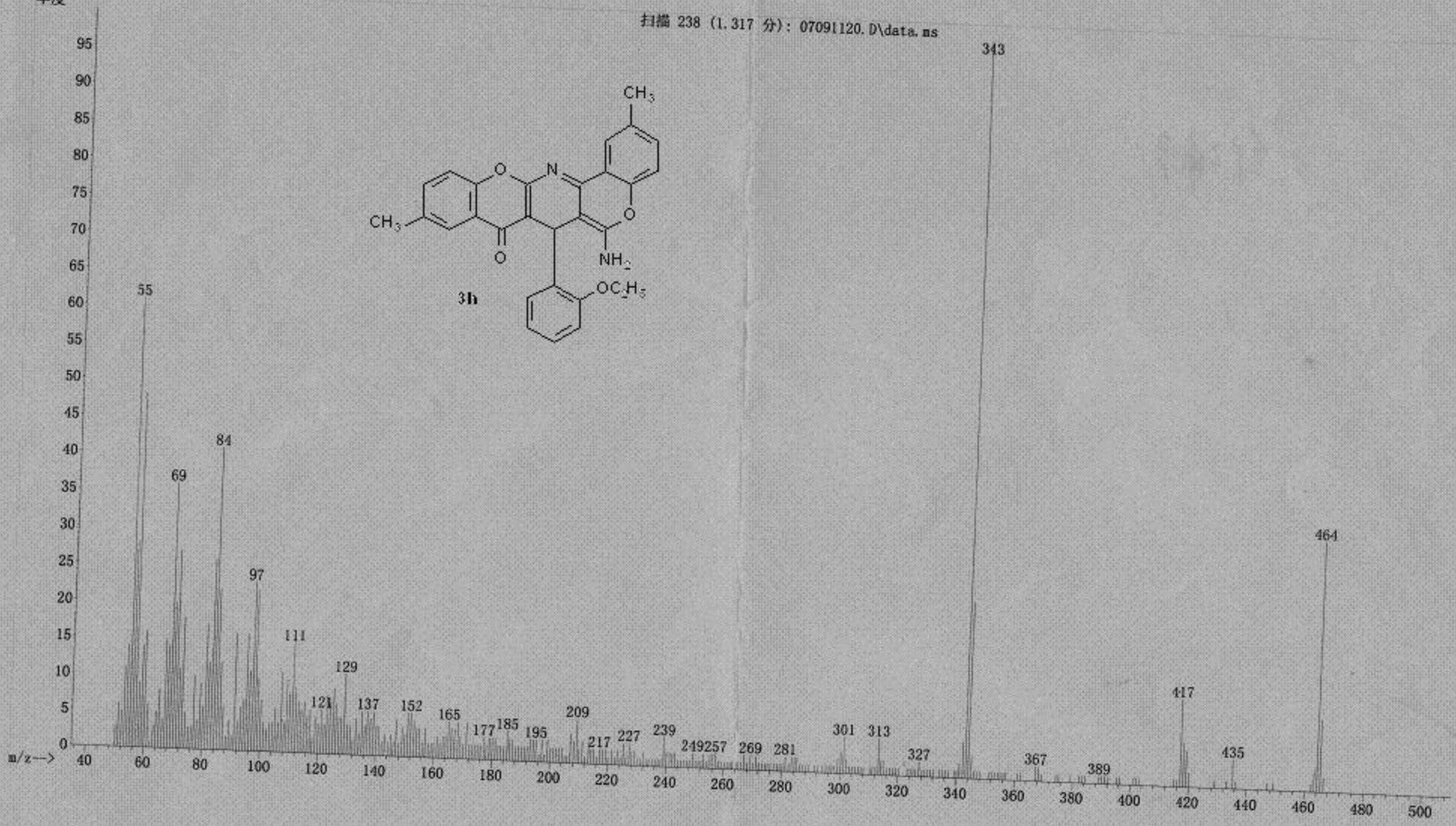


文件　　：D:\msdchem\1\DATA\070911\07091120.D
操作员　：
已采集　：12 Sep 2007　14:31　　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：XM-2
其他信息　：MW=464
样品瓶号：1

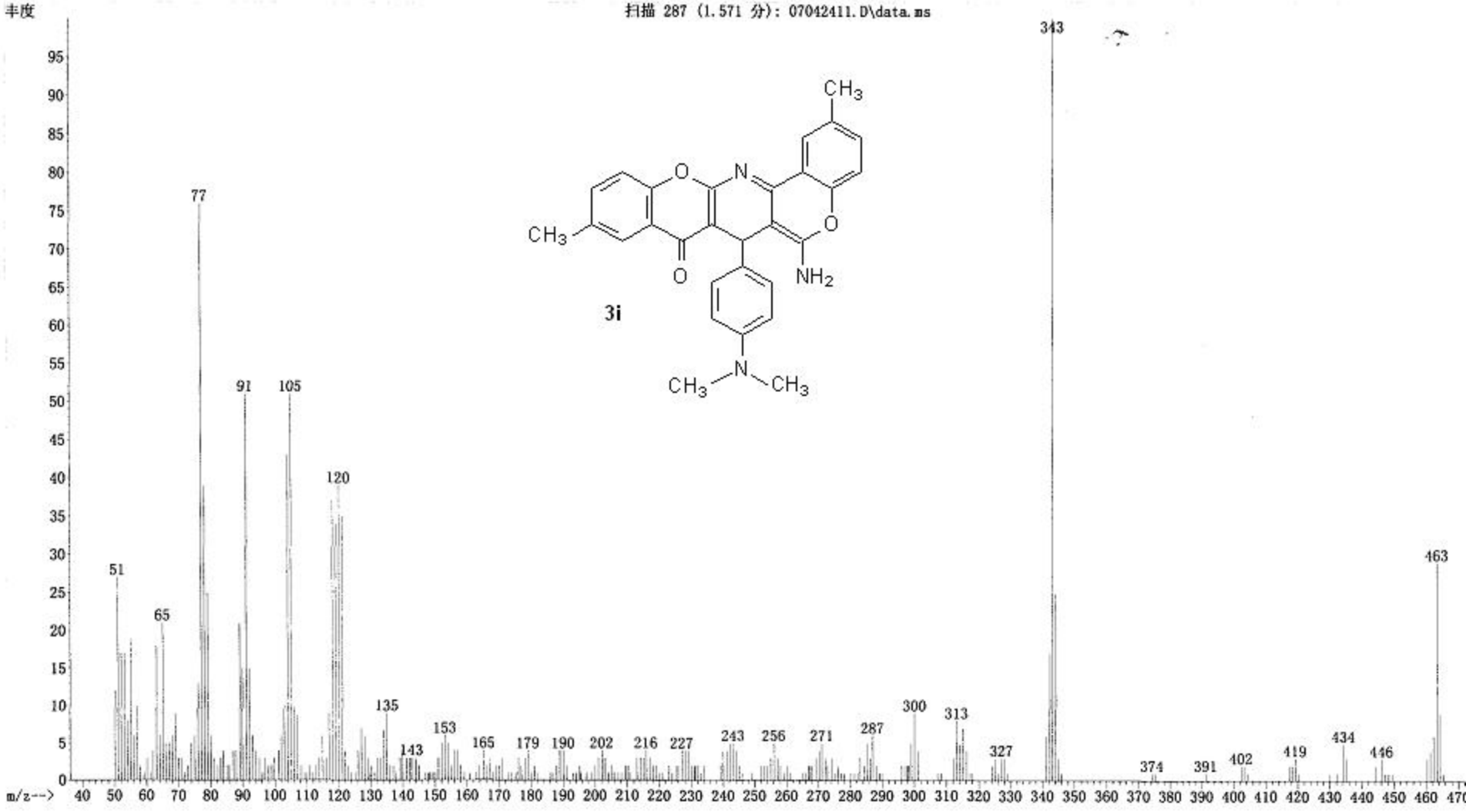
丰度

扫描 238 (1.317 分)：07091120.D\data.ms

3h

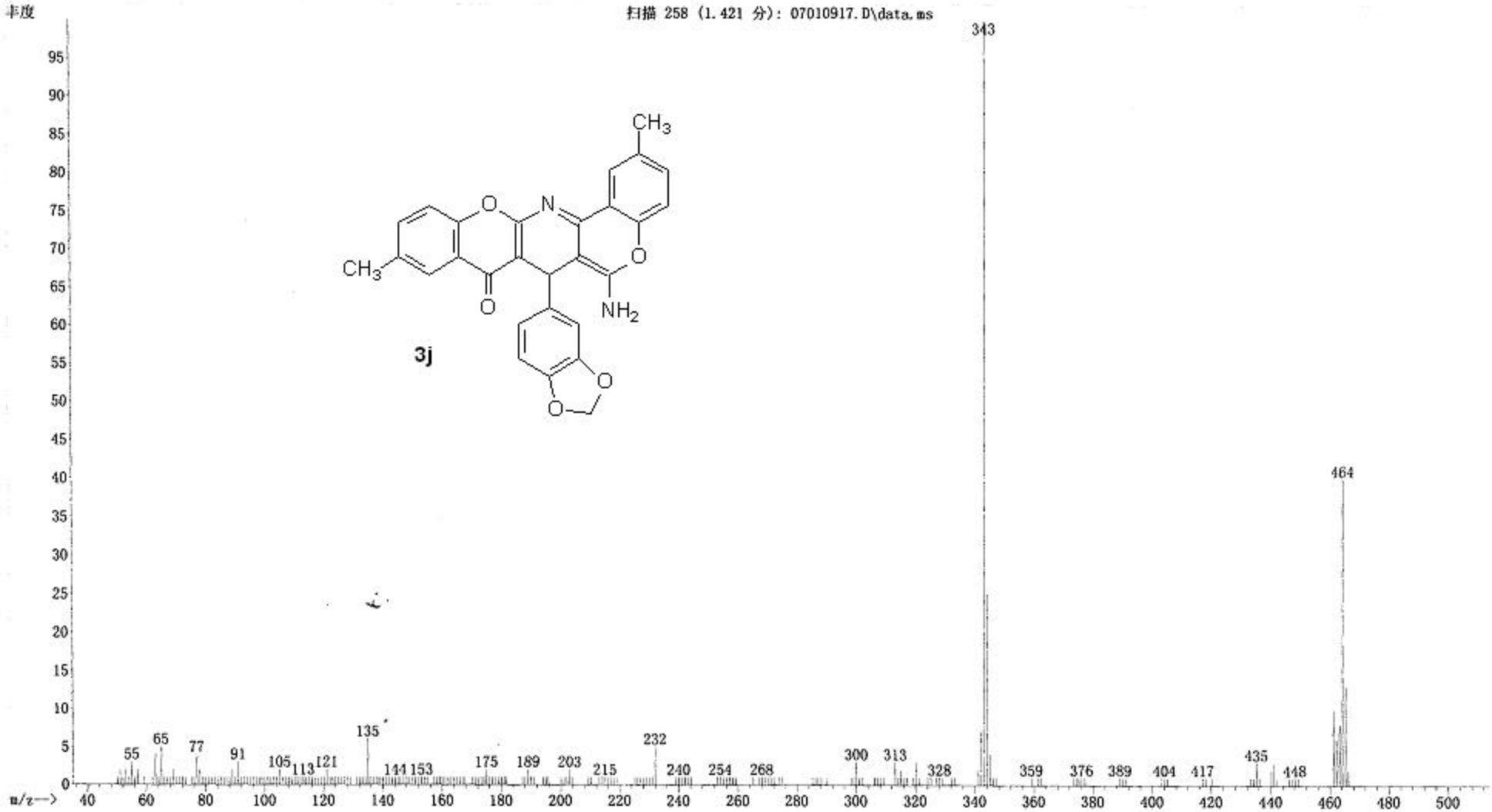


文件　　：D:\msdchem\1\DATA\070424\07042411.D
操作员　：
已采集　：24 Apr 2007　15:18　　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：XM
其他信息　：MW=463
样品瓶号：1

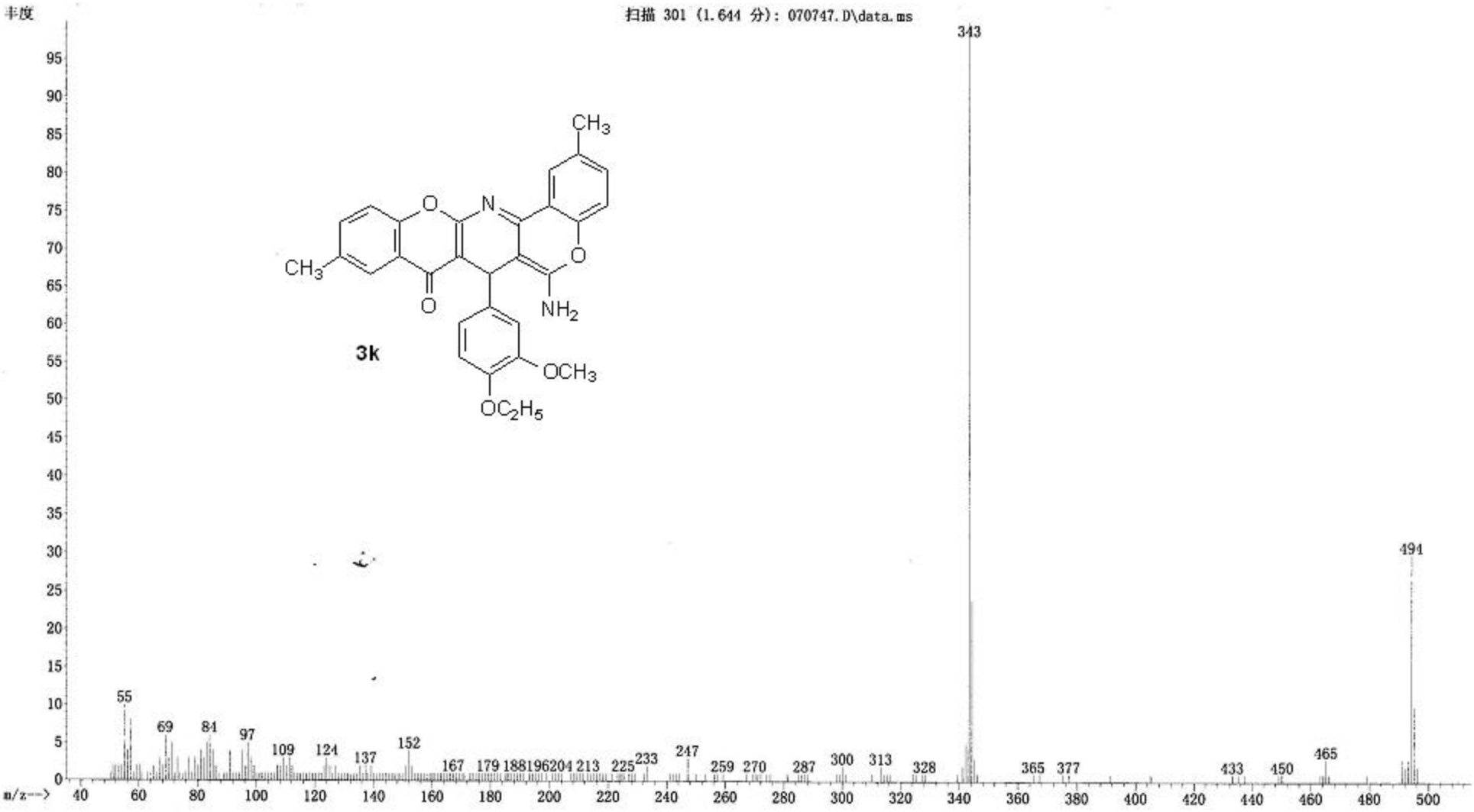


3i

文件　　：D:\msdchem\1\DATA\070109\07010917.D
操作员　：
已采集　：9 Jan 2007　15:59　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：XM-6
其他信息　：MW=464
样品瓶号：1

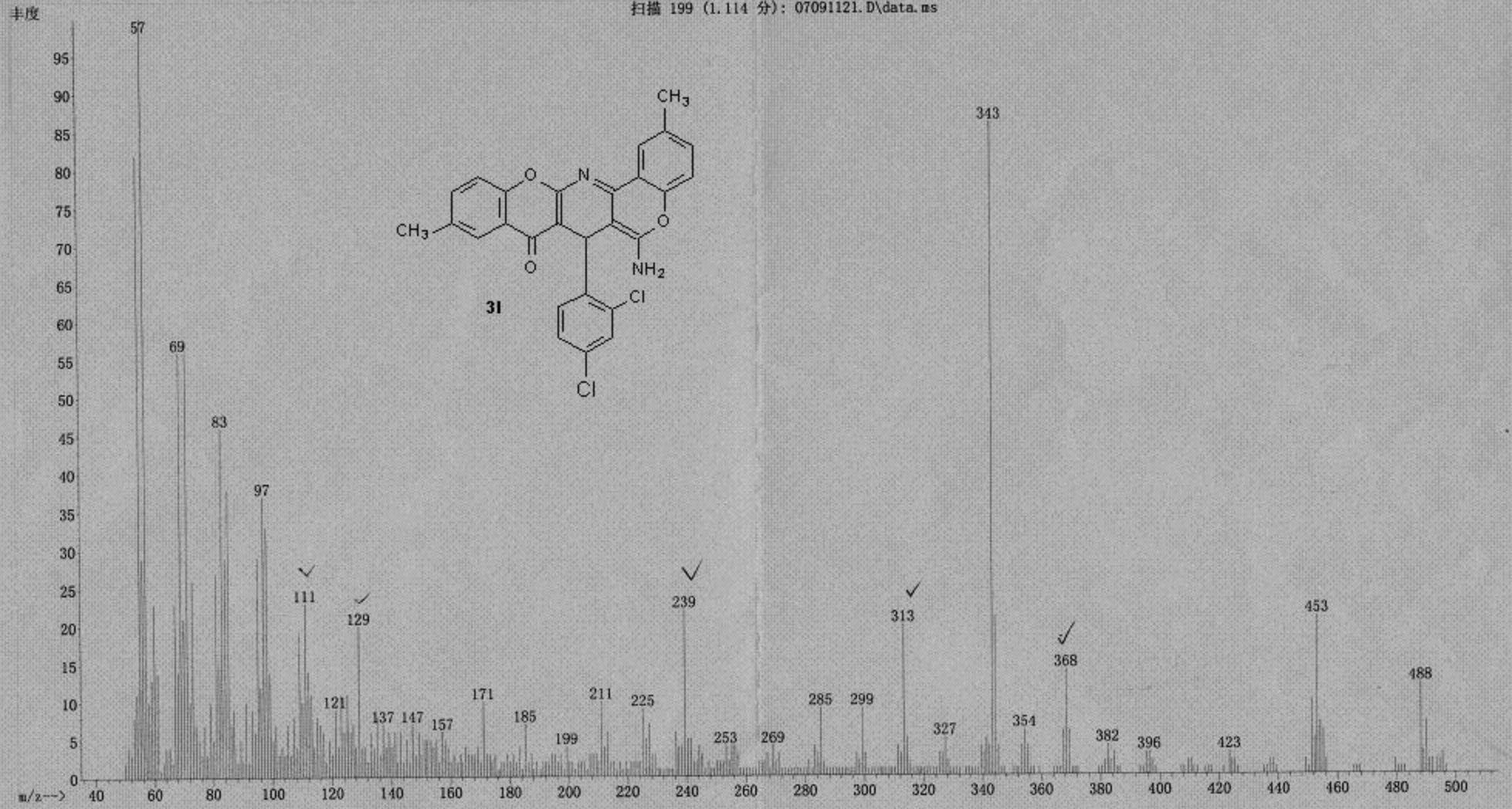


文件　　：D:\msdchem\1\DATA\07074\070747.D
操作员　：
已采集　：4 Jul 2007　10:47　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：2#
其他信息　：MW=494
样品瓶号：1



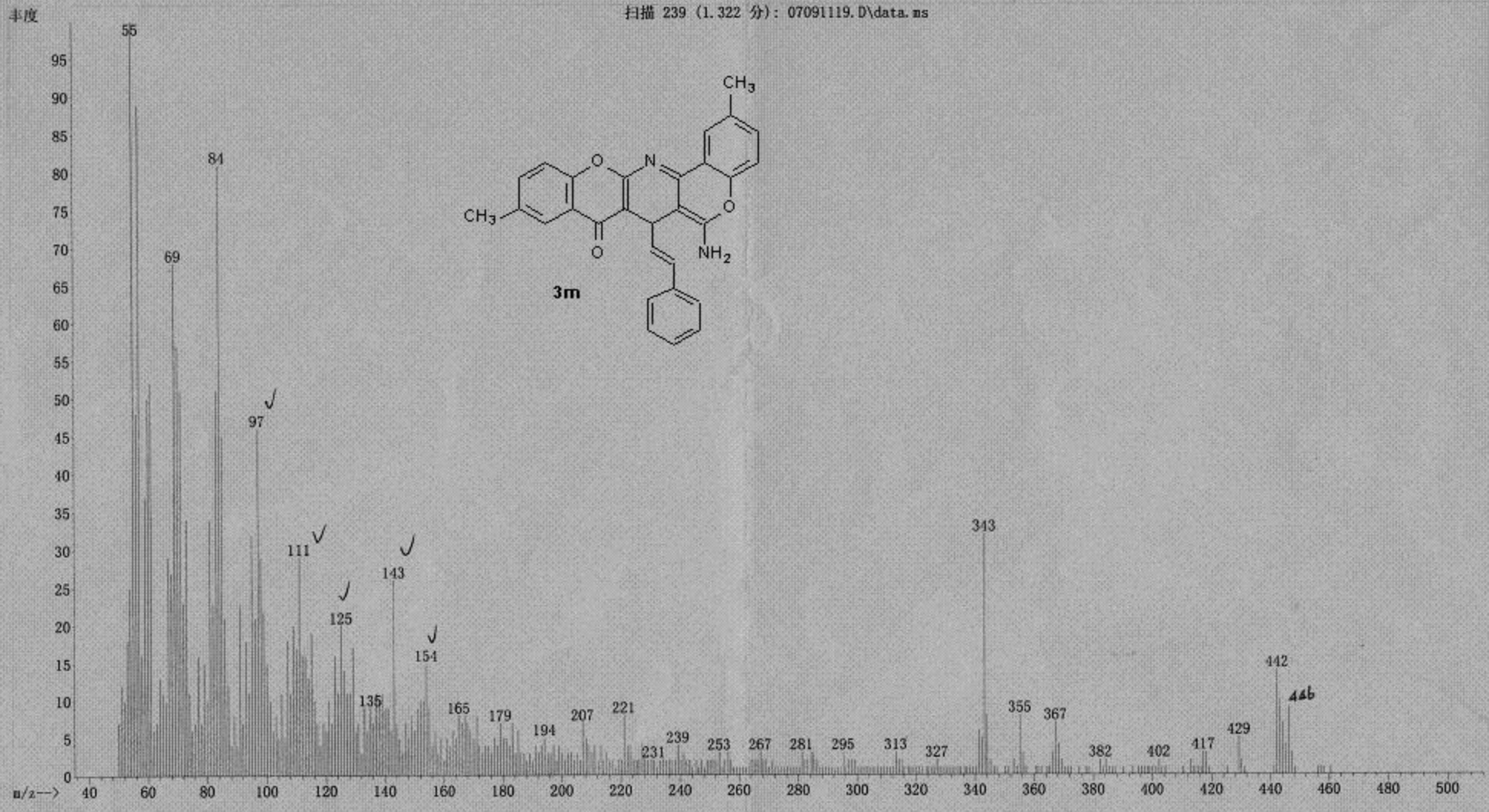
文件　　：D:\msdchem\1\DATA\070911\07091121.D
操作员　：
已采集　：12 Sep 2007　14:46　，使用采集方法 DIRTEST.M
仪器：　仪器 1
样品名：XM-3
其他信息　：MW=488
样品瓶号：1

扫描 199 (1.114 分)：07091121.D\data.ms

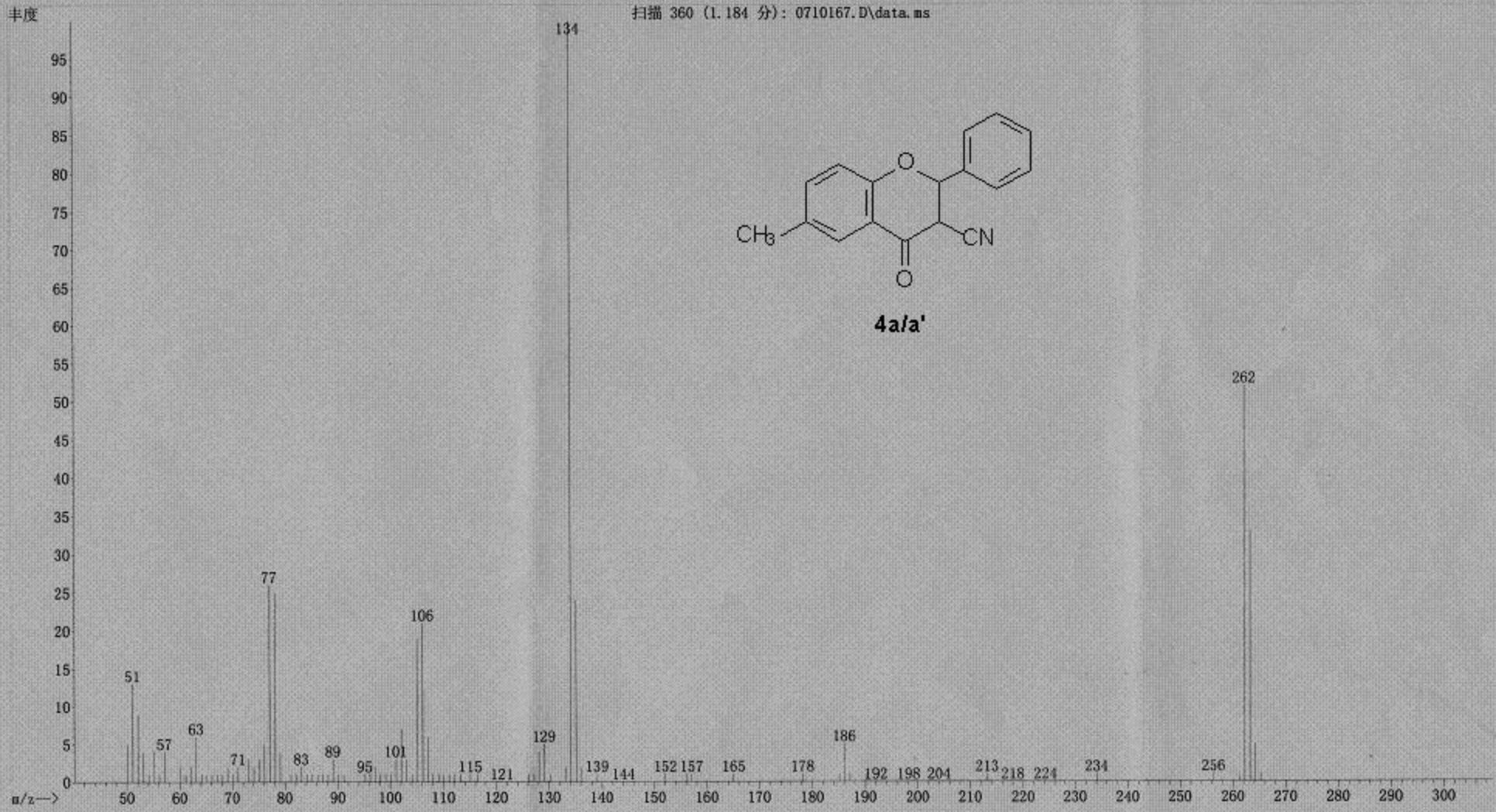


3l

文件　　：D:\msdchem\1\DATA\070911\07091119.D
操作员　：
已采集　：12 Sep 2007　14:14　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：XM-1
其他信息　：MW=446
样品瓶号：1



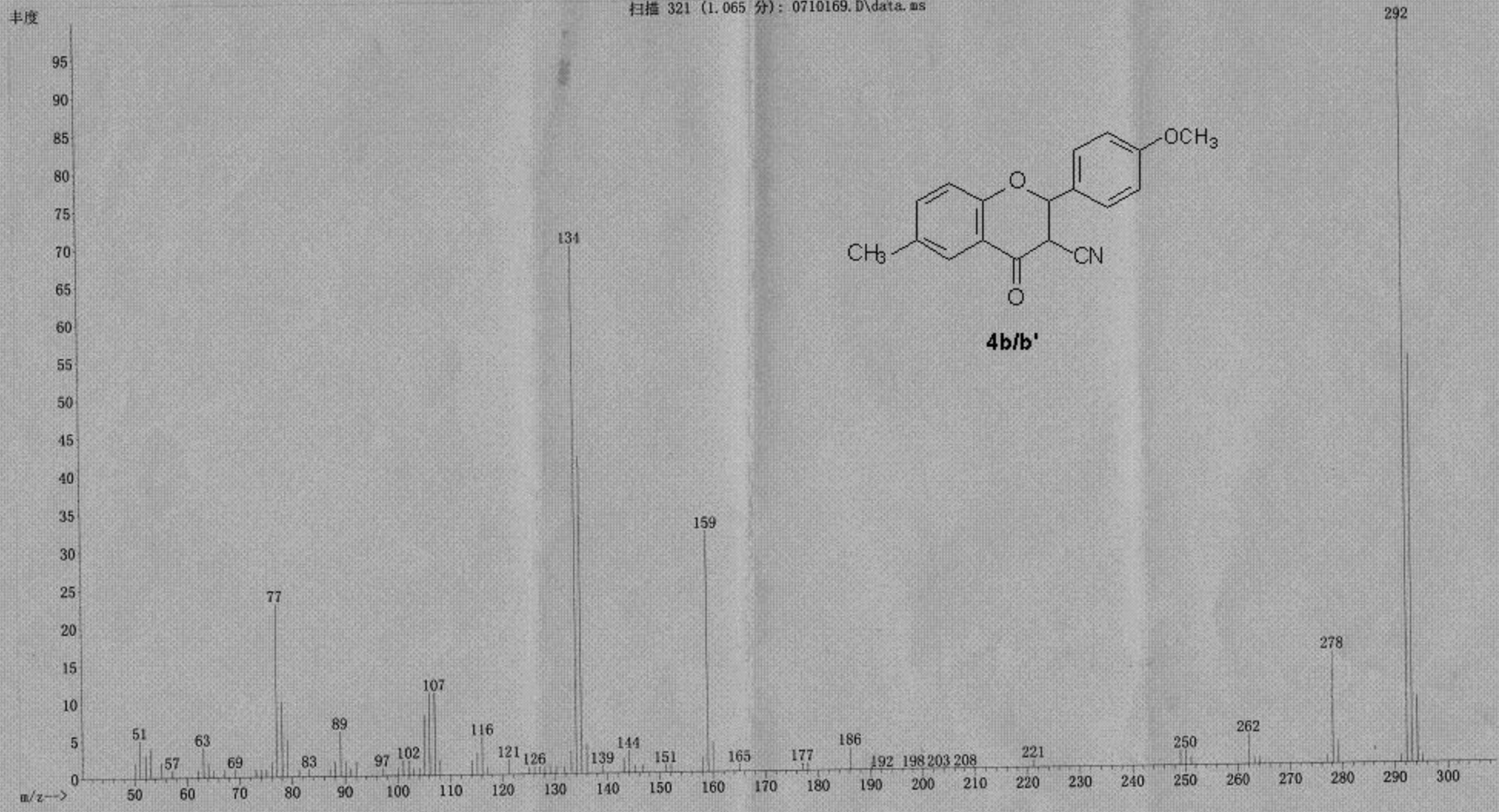
文件　　：D:\msdchem\1\DATA\071016\0710167.D
操作员　：
已采集　：16 Oct 2007　12:45　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：XM-3
其他信息　：MW=263
样品瓶号：1



4a/a'

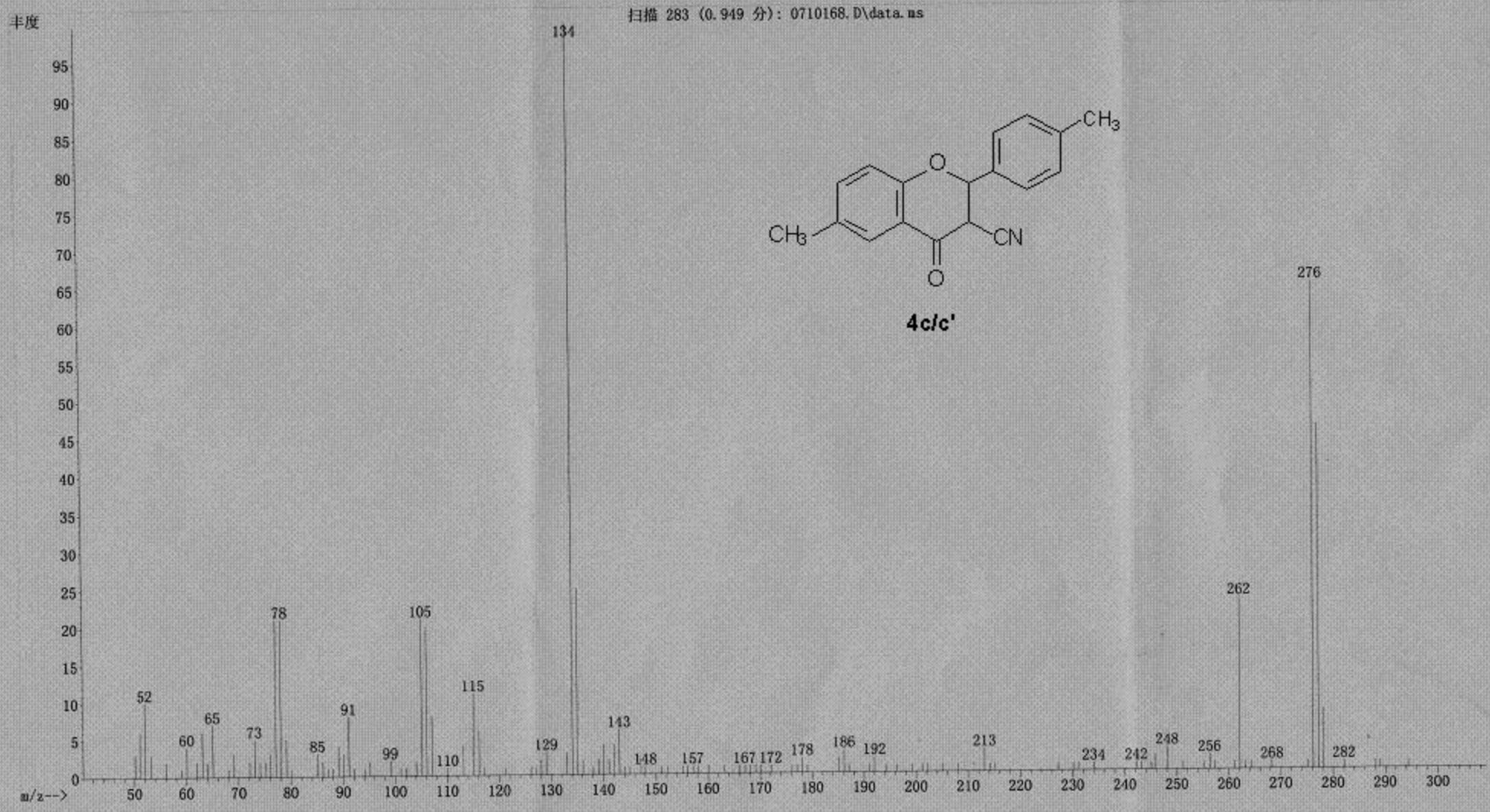
文件　　: D:\msdchem\1\DATA\071016\0710169.D
操作员　:
已采集　: 16 Oct 2007　13:19　　, 使用采集方法 DIPTEST.M
仪器:　仪器 1
样品名: XM-6
其他信息　: MW=293
样品瓶号: 1

扫描 321 (1.065 分): 0710169.D\data.ms



4b/b'

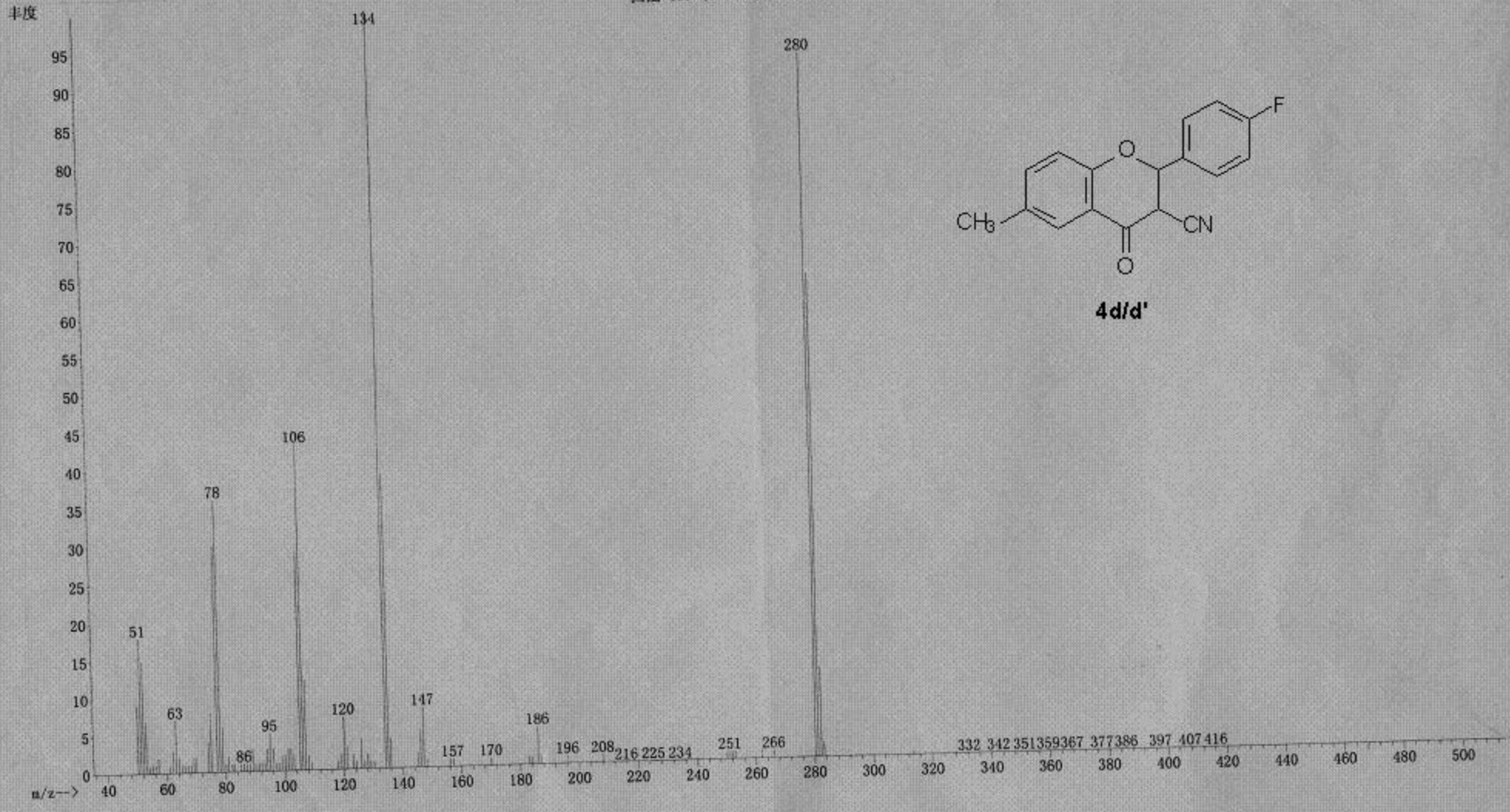
文件　　: D:\msdchem\1\DATA\071016\BSB\0710168.D
操作员　　:
已采集　　: 16 Oct 2007　13:00　　, 使用采集方法 DIPTEST.M
仪器:　仪器 1
样品名: XM-5
其他信息　　: MW=277
样品瓶号: 1



4c/c'

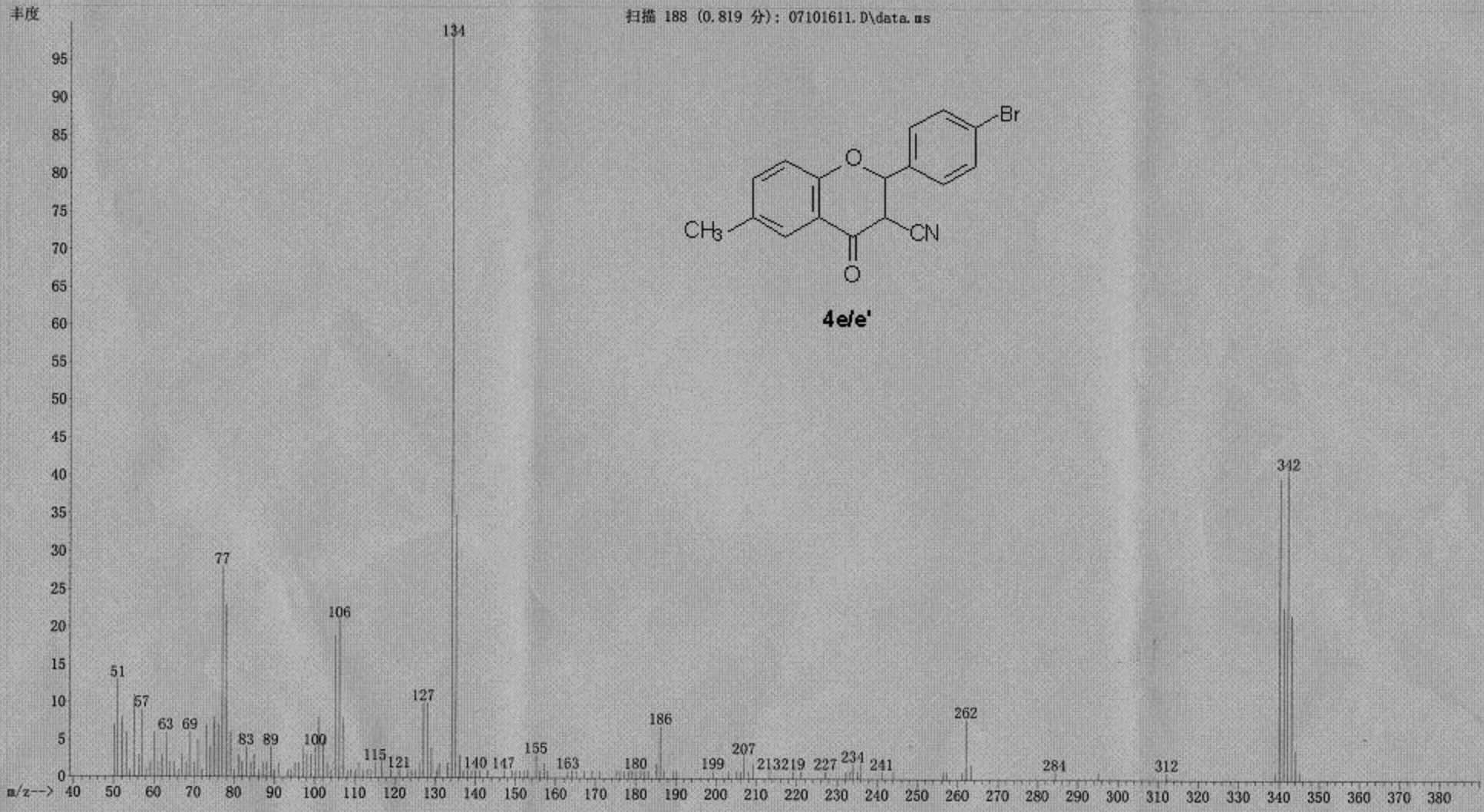
文件　　：D:\msdchem\1\DATA\061205\0612055.D
操作员　：
已采集　：5 Dec 2006　14:02　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：XM-2
其他信息　：MW=
样品瓶号：1

扫描 139 (0.804 分)：0612055.D\data.ms



4d/d'

文件　　：D:\msdchem\1\DATA\071016\07101611.D
操作员　：
已采集　：16 Oct 2007　13:53　，使用采集方法 DIPTEST.M
仪器：　仪器 1
样品名：XM-7
其他信息　：MW=342
样品瓶号：1



文件　　　: D:\msdchem\1\DATA\071016\BSB\07101610.D
操作员　 :
已采集　 : 16 Oct 2007　13:37　　, 使用采集方法 DIPTEST.M
仪器:　 仪器 1
样品名: XM-2
其他信息　: MW=308
样品瓶号: 1