Angewandte Chemie
Eine Zeitschrift der Gesellschaft Deutscher Chemiker

Supporting Information

69451 Weinheim, Germany

Supporting Information

A General Palladium-Catalyzed Suzuki-Miyaura Coupling of Aryl Mesylates**

Chau Ming So, Chak Po Lau, Albert S. C. Chan and Fuk Yee Kwong*

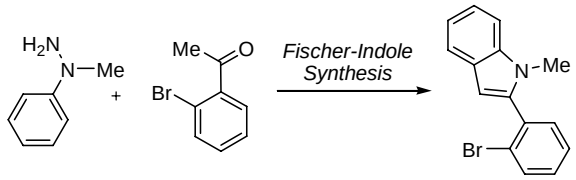
Open Laboratory of Chirotechnology of the Institute of Molecular Technology for Drug Discovery and Synthesis, Department of Applied Biology and Chemical Technology, The Hong Kong Polytechnic University, Hung Hom, Hong Kong.
E-mail: bcfyk@inet.polyu.edu.hk

Table of Contents

1. General considerations.

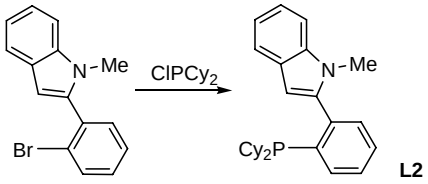
Unless otherwise noted, all reagents were purchased from commercial suppliers and used without purification. All Suzuki-Miyaura reactions were performed in Rotaflo® (England) resealable screw cap Schlenk flask (approx. 20 mL volume) in the presence of Teflon coated magnetic stirrer bar (3 mm × 10 mm). Toluene and tetrahydrofuran (THF) were distilled from sodium and sodium benzophenone ketyl under nitrogen, respectively.[1] *tert*-Butanol was distilled from sodium under nitrogen.[1] Chlorodiphenylphosphine (Tech grade from Aldrich) was distilled under vacuum prior to use. Most commercially available arylboronic acids were used as received. Some arylboronic acids may require further recrystallization depending on the received conditions. 1-Naphthylboronic acid and 2,4-di-*tert*-butyl-6-methoxyphenylboronic acid were synthesized according to literature methods. New bottle of *n*-butyllithium was used (*Note*: since the concentration of *n*-BuLi from old bottle may vary, we recommend to perform a titration prior to use). $K_3PO_4 \bullet H_2O$ and K_3PO_4 were purchased from Fluka. Thin layer chromatography was performed on Merck precoated silica gel 60 F_{254} plates. Silica gel (Merck, 70-230 and 230-400 mesh) was used for column chromatography. Melting points were recorded on an uncorrected Büchi Melting Point B-545 instrument. ^{1}H NMR spectra were recorded on a Bruker (400 MHz) spectrometer. Spectra were referenced internally to the residual proton resonance in $CDCl_3$ (δ 7.26 ppm), or with tetramethylsilane (TMS, δ 0.00 ppm) as the internal standard. Chemical shifts (δ) were reported as part per million (ppm) in δ scale downfield from TMS. ^{13}C NMR spectra were referenced to $CDCl_3$ (δ 77.0 ppm, the middle peak). ^{31}P NMR spectra were referenced to 85% H_3PO_4 externally. Coupling constants (*J*) were reported in Hertz (Hz). Mass spectra (EI-MS and ES-MS) were recorded on a HP 5989B Mass Spectrometer. High-resolution mass spectra (HRMS) were obtained on a Brüker APEX 47e FT-ICR mass spectrometer (ESIMS). GC-MS analysis was conducted on a HP 5973 GCD system using a HP5MS column (30 m × 0.25 mm). The products described in GC yield were accorded to the authentic samples/dodecane calibration standard from HP 6890 GC-FID system. All yields reported refer to isolated yield of compounds estimated to be greater than 95% purity as determined by capillary gas chromatography (GC) or ^{1}H NMR. Compounds described in the literature were characterized by comparison of their ^{1}H, and/or ^{13}C NMR spectra to the previously reported data. The procedures in this section are representative, and thus the yields may differ from those reported in tables.

2. Preparation of new indolyl phosphine ligands L1-L4



General procedure for Fischer-indole synthesis: 2'-Bromoacetophenone (1.31 mL, 10 mmol) was mixed with *N*-methylphenylhydrazine (1.3 mL, 11 mmol) in phosphoric acid (5 mL) and stirred at room temperature for 30 min. PPA (25-30 g) was added to the mixture and an exothermic reaction ensured whereupon the mixture was heated slowly to 120 °C and kept at this temperature for 1 h. The mixture was poured into ice water and then extracted with Et_2O (3 × ~150 mL). The organic phases were combined, dried over Na_2SO_4 and concentrated under reduced pressure. The crude product was filtered through a short silica pad (3 × ~10 cm) and washed with hexane then EA/Hexane (1:9). The solution was evaporated to yield a light yellow solid. Small amount of cold hexane was used to further wash the product. The product was then dried under vacuum to afford *N*-methyl-2-(2'-bromophenyl)indole (2.35 g, 95%) as a light yellow solid. Melting point. 85.5-87.5 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.61 (s, 3H), 6.56 (s, 1H), 7.20-7.45 (m, 6H), 7.70-7.75 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 30.5, 102.0, 109.4, 119.7, 120.6, 121.6, 125.0, 127.1, 127.5 130.0, 132.6 132.7, 134.1, 137.1, 139.5; IR (cm^{-1}) 3049.43, 2933.63, 1536.35, 1457.76, 1431.08, 1382.94, 1337.55, 1309.58, 1164.78, 1061.55, 1023.56, 791.60, 749.44, 662.83, 578.63, 536.74, 454.92; MS (EI): *m/z* (relative intensity) 285 (M^+, 100), 204 (75), 190 (10), 178 (20); HRMS: calcd. for $C_{15}H_{12}BrNH^+$: 286.0231, found 286.0280.

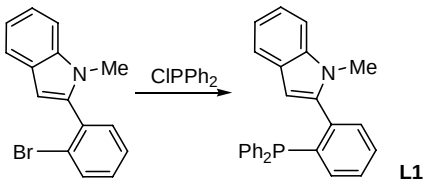
_N_-Methyl-2-(2'- Dicyclohexylphosphinophenyl)indole (L2)



L2

General procedure for ligand synthesis: *N*-methyl-2-(2'-bromophenyl)indole (2.2 g, 7.7 mmol) was dissolved in freshly distilled THF (25 mL) at room temperature under a nitrogen atmosphere. The solution was cooled to -78 °C in dry ice/acetone bath. Titrated *n*-BuLi (8.47 mmol) was added dropwise by syringe. After the reaction mixture was stirred for 30 min at -78 °C, chlorodicyclohexylphosphine (1.87 mL, 8.47 mmol) in THF (5 mL) was added. The reaction was allowed to warm to room temperature and stirred overnight. Solvent was removed under reduced pressure. After the solvent was removed under vacuum, the product was successively washed with cold MeOH/EtOH mixture. The product was then dried under vacuum. White solid of *N*-methyl-2-(2'-dicyclohexylphosphinophenyl)indole **(L2)** (2.75g, 88%) were obtained. Melting point. 171.9-174.9 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.20-1.80 (m, 22H), 3.53 (s, 3H), 6.44 (s, 1H), 7.15(t, *J*=7.4 Hz, 1H), 7.24-7.27 (m, 1H), 7.36-7.50 (m, 4H), 7.66 (d, *J*=7.7 Hz, 2H); ^{13}C NMR (100MHz, $CDCl_3$) δ 25.5, 26.2, 27.1, 28.9, 29.3, 29.5, 30.1, 30.7, 103.2, 109.3, 119.3, 120.2 121.0, 127.6, 127.8, 128.0, 128.2, 128.4, 131.8, 131.9, 132.7, 136.6 (unresolved complex C-P splittings were observed); ^{31}P NMR (162 MHz, C_6D_6) δ -9.87; IR (cm^{-1}) 2422.67, 3050.84, 2924.05, 2846.58, 1445.74, 1384.98, 1338.55, 1309.68, 1264.17, 1123.17, 1001.01, 886.58, 848.87, 769.86, 746.53, 523.77; MS (EI): *m/z* (relative intensity) 403(M^+, 25), 348 (5), 321 (30), 238 (100), 222 (30), 207 (20); HRMS: calcd. for $C_{27}H_{34}NP$: 403.2423, found 403.2414.

_N_-Methyl-2-(2'-Diphenylphosphinophenyl)indole (L1)

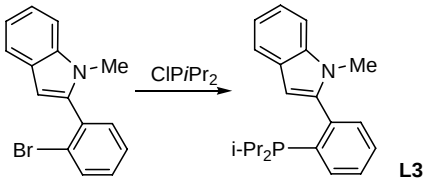


L1

General procedures for the synthesis of ligand **L2** were followed. *N*-Methyl-2-(2'-bromophenyl)indole (1.89 g, 6.6 mmol), *n*-BuLi (6.9 mmol), chlorodiphenylphosphine (1.25 mL, 7.0 mmol) were used to afford *N*-methyl-2-(2'-diphenylphosphinophenyl)indole (**L1**) (1.65 g, 64%) as a white crystal. Melting point. 122.0-125.5 ºC; ^{1}H NMR (400 MHz, C_6D_6) δ 3.18 (s, 3H), 6.55 (s,1H), 7.08-7.66 (m, 18H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 30.5, 103.7, 103.8, 109.5, 119.5, 120.4, 121.5, 127.6, 128.5 (overlapped), 128.6, 128.7, 128.8, 131.6 (overlapped), 133.7, 133.8, 133.9, 137.2, 137.4, 137.5, 139.6 (overlapped) (unresolved complex C-P splittings were

observed); ^{31}P NMR (162 MHz, C_6D_6) δ -12.02; IR (cm^{-1}) 3446.06, 3048.19, 2931.48, 1582.52, 1461.53, 1429.92, 1377.43, 1359.35, 1336.25, 1308.54, 1235.94, 1176.72, 1123.94, 1093.80, 1025.26, 999.97, 924.13, 943.98 ,769.18, 745.33, 695.27, 539.41, 495.26; MS (EI): *m/z* (relative intensity) 391 (M^+, 100), 376 (50), 314 (30), 298 (27), 281 (5), 261 (5), 236 (50), 222 (40), 204 (25); HRMS: calcd. for $C_{27}H_{22}NPH^+$: 392.1568, found 392.1579.

_N_-Methyl-2-(2'-Diisopropylphosphinophenyl)indole (L3)

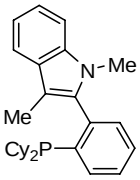


L3

General procedures for the synthesis of ligand **L2** were followed. *N*-Methyl-2-(2'-bromophenyl)indole (1.66 g, 5.8 mmol), *n*-BuLi (6.38 mmol), chlorodiisopropylphosphine (1.03 mL, 6.4 mmol) were used to afford *N*-methyl-2-(2'-diisopropylphosphinophenyl)indole (**L3**) (1.41 g, 75%) as white crystal. Melting point. 146.1-147.8 ºC; ^{1}H NMR (400 MHz, CD_2Cl_2) δ 1.06-1.15 (m, 12H), 1.80-2.80 (m, 2H), 3.61 (s, 3H), 6.52 (s, 1H), 7.23-7.77 (m, 8H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 20.0, 20.2, 23.3, 25.7 (overlapped), 30.8 (overlapped), 103.3 (overlapped), 109.4, 119.4, 120.1, 121.1, 127.8, 128.2, 128.4, 131.7, 131.8, 132.5, 132.6, 136.7, 137.9, 138.1, 140.6, 140.9, 141.5, 141.5 (overlapped) (unresolved complex C-P splitting was observed); ^{31}P NMR (162 MHz, CD_2Cl_2) δ -1.29; IR (cm^{-1}) 3445.89, 3050.50, 2941.77, 2860.55, 1542.40, 1454.90, 1420.35, 1380.67, 1336.26, 1309.58, 1237.42, 1119.75, 1005.64, 880.09, 778.80, 747.64, 676.90, 653.14, 606.16, 583.14, 531.39, 459.25; MS (EI): *m/z* (relative intensity) 323 (M^+, 20), 280 (100), 236 (40), 222 (30), 204 (5); HRMS: calcd. for $C_{21}H_{26}NP$: 323.1797, found 323.1803.

Independent synthesis of ligand **L4** for a **L2/L4** ligand comparison study.

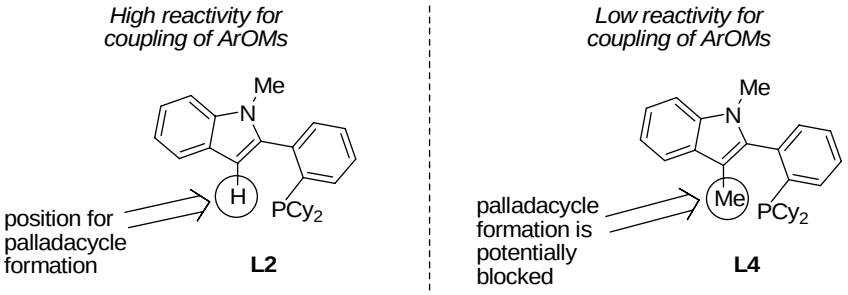
1,3-Dimethyl-2-(2'-dicyclohexylphosphinophenyl)indole (L4).



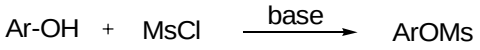
1,3-Dimethylindole (1.45 g, 10 mmol) was dissolved in freshly distilled THF (20 mL) at room temperature under a nitrogen atmosphere. The solution was cooled to 0 °C in ice water bath. Titrated *t*-BuLi (10 mmol) was added dropwise by syringe. After the reaction mixture was stirred for 10 min at 0 °C, 2-bromochlorobenzene (1.2 mL, 10 mmol) was added dropwise by syringe and the solution was stirred for further 30 min. The solvent was removed under vacuum. Diethyl ether (150 mL) was added and washed with brine. The solution was concentrated and subjected to column chromatography and eluted with DCM:Hexane = 2:8. The factions that are containing the 1,3-dimethylindole and 1,3-dimethyl 2-(2-bromophenyl)indole were concentrated and dried by vacuum. The amount of 1,3-dimethylindole and 2-(2-bromophenyl)indole in the mixture was judged by GC. The viscous mixture was dissolved in freshly distilled THF (20 mL) at room temperature under a nitrogen atmosphere. The solution was cooled to -78 °C in dry ice/acetone bath. Titrated *n*-BuLi (1.1 equiv.) was added dropwise by syringe. After the reaction mixture was stirred for 10 min at -78 °C, chlorodicyclohexylphosphine (1.1 equiv.) was added. The reaction was allowed to warm to room temperature and stirred overnight. Solvent was removed under reduced pressure. After the solvent was removed under vacuum, the product was washed with cold EtOH. The product was then dried under vacuum. White solid of 1,3-dimethyl-2-(2'-dicyclohexylphosphinophenyl)indole (20% overall yield in 2 steps) were obtained. Melting point. 199.8-201.9 °C; ^{1}H NMR (400 MHz, CD_2Cl_2) δ 1.22-2.12 (m, 22H), 2.23 (s, 3H), 3.54 (s, 3H), 7.20 (t, *J*=7.3, 1H), 7.28-7.57 (m, 5H), 7.65 (d, *J*=7.6 Hz, 1H), 7.74-7.76 (m, 1H); ^{13}C NMR (100 MHz,CD_2Cl_2) δ 10.5, 27.2, 27.3, 27.8, 27.9, 28.0, 28.2, 28.3, 28.4, 29.8, 30.7, 30.9, 31.1, 31.2, 31.7, 33.8, 34.0, 35.7, 35.8, 109.1, 109.7, 119.1, 119.3, 121.8, 128.6, 129.0, 129.1, 133.0, 133.1, 133.7, 137.1, 138.8, 139.0, 140.6, 140.9 (unresolved complex C-P splittings were observed); ^{31}P NMR (162 MHz, CD_2Cl_2) δ -8.96; IR (cm^{-1}) 3046.64, 2923.29, 2846.57, 1573.98, 1459.63, 1445.76, 1421.75, 1381.21, 1357.91, 1327.96, 1265.92, 1243.64,

1178.72, 1156.81, 1129.37, 1080.41, 1001.15, 918.68, 881.88, 848.60, 820.68, 760.63, 738.96, 614.63, 552.33, 526.21, 484.54, 460.05; MS (EI): *m/z* (relative intensity) 417(M^+,15), 402 (100), 334 (25), 252 (30), 236 (29), 222 (10), 207 (3); HRMS: calcd. for $C_{27}H_{34}NPH^+$: 404.2507, found 404.2511.

The structural fine-tunings on ligand skeleton were reported to give significant impacts on the effectiveness of a catalytic system. In order to have a better insight on the relationship between the ligand reactivity and the structure of the indolyl ligand family, we performed additional probing experiments. Mechanistic studies showed that the substitution pattern on indole moiety is important to the reactivity. ^{1}H NMR studies indicated that the signal of the C-H group (from 3-position of the indole ring, **L2**) rapidly disappeared before the catalysis starts. These spectroscopic data directly provide the evidence for the palladacycle formation. We are intrigued to investigate the importance of the indole 3-C-H position, we therefore independently synthesized a structurally similar ligand **L4** (Figure 2). Ligand **L4** contains all features of ligand **L2** except the C-H position from the indolyl scaffold is blocked by a methyl group. Surprisingly, a significantly lower reactivity (only 24% conv.) was observed when **L4** was subjected to the coupling of unactivated 4-*tert*-butylphenyl mesylate with 4-tolylboronic acid. From the ligand structural comparison, it seems to show that the ease of the palladacycle formation is important to accomplish mesylate catalysis.

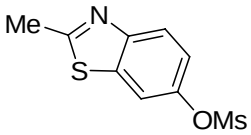


3. Preparation of aryl mesylates



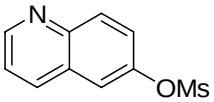
4-*tert*-Butylphenyl mesylate[3], 4-methoxyphenyl mesylate[4], 4-acetylphenyl mesylate[4], 4-benzoylphenyl mesylate[5], 4-acetyl-2-methoxyphenyl mesylate[6], 1,3-Benzodioxol-5-ol mesylate[7], 4-formyl-2-methoxyphenyl mesylate[8], 3-pyridyl mesylate[9], 4-cyanophenyl mesylate[4], 4-(methoxycarbonyl)phenyl mesylate[4] were prepared from their corresponding phenols with MsCl in the presence of triethylamine in CH_2Cl_2 according to the literature method.[10] Unknown aryl mesylates were prepared according the the literature procedures without modification. Characterizations data of unknown aryl mesylates are shown below.[10]

2-Methyl-5-benzothiazol mesylate



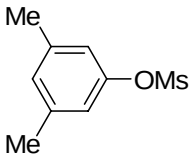
Obtained in 38% yield as white crystals: mp 119.5-120.8 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 2.76 (s, 3H), 3.12 (s, 3H), 7.24 (dd, *J*=2.3Hz, 8.7Hz, 1H), 7.74-7.78 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.0, 37.1, 115.4, 118.9, 122.1, 134.4, 147.4, 153.7, 169.6; MS (EI): *m/z* (relative intensity) 243 (M^+, 35), 164 (100), 136 (28), 122 (15), 95 (18). HRMS: calcd. for $C_9H_9N_1O_3S_2$: 243.0018, found 243.0023.

6-(Mesyloxy)quinoline



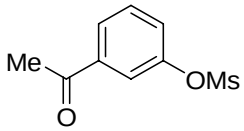
Obtained in 58% yield as deep purple crystals: mp 84.6-86.2 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ 3.15 (s, 3H), 7.32-7.35 (m, 1H), 7.52-7.55 (m, 1H), 7.67 (d, *J*=2.6Hz, 1H), 8.04-8.07 (m, 2H), 8.83-8.85 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 37.4, 119.2, 121.8, 124.0,128.1, 131.6, 135.7, 146.4, 146.4, 150.7; MS (EI): *m/z* (relative intensity) 223 (M^+, 63), 145 (60), 116 (100), 89 (30), 63 (10). HRMS: calcd. for $C_{10}H_9N_1O_3S_1$: 223.0298, found 223.0292.

3,5-Dimethylphenyl mesylate.



Obtained in 95% yield as white crystals: mp 41.7-43.4 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.30 (s, 3H), 3.07 (s, 3H), 6.88 (s, 2H), 6.93 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.8, 36.8, 119.1, 128.7, 139.7, 149.0; MS (EI): *m/z* (relative intensity) 200 (M^+, 65), 122 (100), 107 (20), 91 (30), 77 (30). HRMS: calcd. for $C_9H_{12}O_3S_1$: 200.0502, found 200.0507.

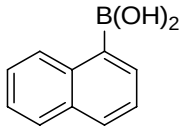
3-Acetylphenyl mesylate



Obtained in 83% yield as white crystals: mp 50.1-52.4 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.56 (s, 3H), 3.14 (s, 3H), 7.44-7.48 (m, 2H), 7.78 (s, 1H), 7.86 (d, *J*=7.1Hz, 1H) ; ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.5, 37.4, 121.4, 126.5, 127.0, 130.2, 138.7, 149.2, 196.3; MS (EI): *m/z* (relative intensity) 214 (M^+, 28), 199 (100), 171 (5), 121 (35), 92 (10). HRMS: calcd. for $C_9H_{10}O_4S_1$: 214.2940, found 214.0304.

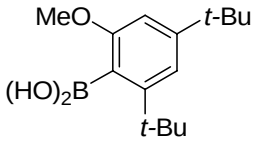
4. Procedures for preparation arylboronic acid substrates

1-Naphthylboronic acid[11]



Anhydrous THF (20 mL) was added to 1-bromonaphthalene (5.56 mL, 40 mmol) under nitrogen at room temperature. *n*-Butylithium (44 mmol) was added dropwise to the solution at -78 ^{o}C and stirred for 30min. The reaction mixture was transferred to a solution of B(O*i*-Pr)$_3$ (18.6 mL, 80 mmol) in THF (10 mL) at -78 °C *via* cannular under nitrogen. The solution was warm to room temperature and stirred overnight. Diluted HCl (10%, 20 mL) was added to the mixture and stirred for 30 min. The organic layer was separated and the aqueous phase was extracted by diethyl ether (50 mL × 3). The combined organic extracts were washed with brine and dried over $MgSO_4$. The solvent was removed and the residue was crystallized under ether/hexane to give colorless crystals (5.0 g, 72%). The product was used without further characterization.

2,4-Di-*tert*-butyl-6-methoxyphenylboronic acid[12]

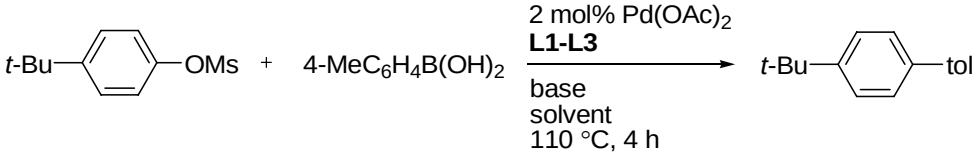


n-Butyllithium (1.6 M in hexane, 25 mL, 40 mmol) was added to a solution of 2-bromo-3,5-di *tert*-butylanisole (12 g, 40 mmol) in THF (20 mL) at -78 °C under nitrogen. The mixture was stirred for 1 h and then transferred to a solution of distilled B(OMe)$_3$ (9.1 mL, 80 mmoL) in THF (10 mL) at -78 °C under nitrogen. It was allowed to warm to room temperature and water was added. The organic layer was separated, and the aqueous phase was extracted with ether (3 × ~100 mL). The combined organic phases were washed with brine. After removing of solvent, the residue was redissolved in ethanol (60 mL). A solution of NaOH (2 M) was added and the mixture was stirred for 2 h. Solvent was rotary-evaporated, and the remaining aqueous layer was extracted with ether (3 × ~100 mL). The etheral layer was then washed with brine and dried over $MgSO_4$. The solvent was removed, and the residue was recrystallized from ether/hexane to give

2,4-di-*tert*-butyl-6-methoxyphenylboronic acid as colorless crystals (7.8 g, 74%). The product was used without further characterization.

5. General procedures/data for initial ligand and reaction condition screenings

General procedure for screening: $Pd(OAc)_2$ (6.8 mg, 0.030 mmol) and ligand (Pd:L = 1:4) were loaded into a Schlenk tube equipped with a Teflon-coated magnetic stir bar. The tube was evacuated and flushed with nitrogen for three times. Precomplexation was applied by adding freshly distilled dichloromethane (0.5 mL) and Et_3N (50 μL) into the tube. The palladium complex stock solution was stirred and warmed using hair drier for about 1 to 2 minutes until the solvent started boiling. The solvent was then evaporated under high vacuum. 4-*tert*-Butylphenyl mesylate (1.0 mmol), 4-methylphenylboronic acid (2.0 mmol) and base (3.0 mmol) were loaded into the tube, and the system was further evacuated and flushed with nitrogen for three times. The solvent (3.0 mL) was added with stirring at room temperature for several minutes. The tube was then placed into a preheated oil bath (110 ^oC) and stirred for the time as indicated. After completion of reaction, the reaction tube was allowed to cool to room temperature. Ethyl acetate (~10 mL), dodecane (227 μL, internal standard) and water were added. The organic layer was subjected to GC analysis. The GC yield obtained was previously calibrated by authentic sample/dodecane calibration curve.



entry	Ligand	base	Solvent	GC yield (%)
1	L1	$K_3PO_4.H_2O$	^{t}BuOH	<1%
2	L3	$K_3PO_4.H_2O$	^{t}BuOH	84%
3	L2	$K_3PO_4.H_2O$	^{t}BuOH	89%
4	L2	$K_3PO_4.H_2O$	Toluene	42%
5	L2	$K_3PO_4.H_2O$	DMF	50%
6	L2	$K_3PO_4.H_2O$	^{t}BuOH	89%
7	L2	$K_3PO_4.H_2O$	THF	33%
8	L2	Cs_2CO_3	^{t}BuOH	83%
9	L2	CsF	^{t}BuOH	93%

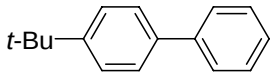
10	L2	K_2CO_3	^tBuOH	79%
11	L2	^tBuOK	^tBuOH	<1%
12	L2	K_3PO_4	^tBuOH	97%
13	L2	K_3PO_4	DMF	81%

6. General procedures for Suzuki-Miyaura couplings of aryl mesylates

General procedure for Suzuki-Miyaura coupling of aryl mesylates: $Pd(OAc)_2$ (2.3 mg, 0.010 mmol) and ligand (Pd:L = 1:4) were loaded into a Schlenk tube equipped with a Teflon-coated magnetic stir bar. The tube was evacuated and flushed with nitrogen for several times. Precomplexation was applied by adding freshly distilled dichloromethane (0.5 mL) and Et_3N (50 μL) into the tube. The solution was stirred and warmed using hair drier for about 1 to 2 minutes until the solvent started boiling. The solvent was then evaporated under high vacuum. Aryl mesylate (1.0 mmol), arylboronic acid (2.0 mmol) and K_3PO_4 (3.0 mmol) were loaded into the tube, and the system was further evacuated and flushed with nitrogen for several times. The solvent *tert*-butanol (3.0 mL) was then added. The tube was stirred at room temperature for several minutes and then placed into a preheated oil bath (110 ^oC) for the time period as indicated in Table 2. After completion of reaction as judged by GC analysis, the reaction tube was allowed to cool to room temperature and quenched with water and diluted with EtOAc. The organic layer was separated and the aqueous layer was washed with EtOAc. The filtrate was concentrated under reduced pressure. The crude products were purified by flash column chromatography on silica gel (230-400 mesh) to afford the desired product.

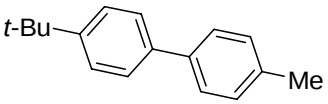
7. Characterization data for coupling products

4-*tert*-Butylbiphenyl (Table 2, entries 1 and 2, Scheme 2).[14]



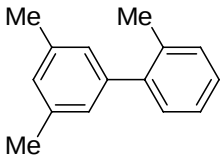
Hexane, R_f=0.55; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.72 (s, 9H), 7.64 (t, *J*=7.4 Hz, 1H), 7.72-7.82 (m, 4H), 7.89-7.96 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 31.3, 34.4, 125.6, 126.7, 126.9, 128.6, 138.2, 140.9, 150.0; MS (EI): *m/z* (relative intensity) 210 (M^+, 35), 195 (100), 178 (20), 167 (30).

4-Methyl-4'-*tert*-butylbiphenyl (Table 2, entry 3).[13]



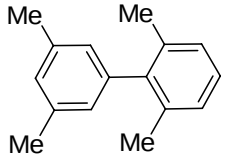
Hexane, R_f=0.5; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.65 (s, 9H), 2.65 (s, 3H), 7.49 (d, *J*=8.0 Hz, 2H), 7.73 (*J*=8.4 Hz, 2H), 7.78 (*J*=8.0 Hz, 2H), 7.82 (*J*=8.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.0, 31.3, 34.4, 125.6, 126.5, 126.7, 129.4, 136.5, 138.1, 138.2, 149.7; MS (EI): *m/z* (relative intensity) 224 (M^+, 33), 209 (100), 193 (5), 181 (14), 165 (10).

3, 5-Dimethyl-2'-methylbiphenyl (Table 2, entry 4).[15]



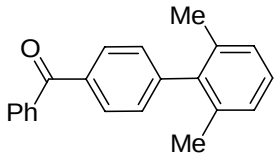
Hexane, R_f=0.55; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.59 (s, 3H), 2.65 (s, 6H), 7.27 (m, 3H), 7.50-7.54 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.4, 21.2, 125.6, 126.9, 128.3, 129.6, 130.1, 135.1, 137.3, 141.9, 142.1; MS (EI): *m/z* (relative intensity) 196 (M^+, 70), 181 (100), 165 (45), 152 (10).

3, 5-Dimethyl-2',6'-dimethylbiphenyl (Table 2, entry 5).[16]



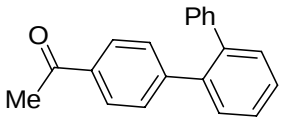
Hexane, R_f=0.55; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.29 (s, 6H), 2.57 (s, 6H), 7.01 (s, 2H), 7.20 (s, 1H), 7.30-7.40 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.8, 21.3, 126.6, 127.1, 128.1, 135.8, 137.6, 11.0, 142.0; MS (EI): *m/z* (relative intensity) 210 (M^+, 65), 195 (100), 180 (30), 152 (25).

4-(2,6-Dimethylphenyl)benzophenone (Table 2, entry 6).



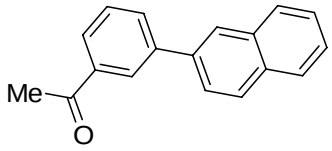
EA:Hexane = 1:20, R_f=0.4; white solid, m.p. = 76.5-78.9 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.13 (s, 6H), 7.18-7.25 (m, 3H), 7.33 (d, *J*=8.2 Hz, 2H), 7.54 (t, *J*=7.7Hz, 2H), 7.62-7.63 (m, 1H), 7.93-7.98 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.5, 127.1, 127.2, 127.9, 128.7, 129.6, 130.0, 131.9, 135.1, 135.5, 137.3, 140.3, 145.4, 195.8; MS (EI): *m/z* (relative intensity) 286 (M^+, 80), 209 (100), 181 (25), 165 (50), 105 (45). HRMS: calcd. for $C_{21}H_{18}O$: 287.1436, found 287.1442.

4-(2-Biphenyl)acetophenone (Table 2, entry 7).[18]



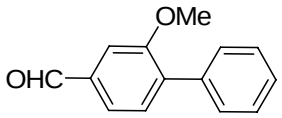
EA:Hexane = 1:20, R_f=0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.56 (s, 3H), 7.20-7.31 (m, 7H), 7.46-7.49 (m, 4H), 7.86 (d, *J*=8.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.2, 126.5, 127.4, 127.7, 127.8, 127.9, 129.0, 129.8, 130.1, 130.5, 134.8, 139.0, 140.3, 140.7, 146.3; MS (EI): *m/z* (relative intensity) 272 (M^+, 70), 257 (100), 228 (50), 202 (20).

3-(2-Naphthyl)acetophenone (Table 2, entry 8).



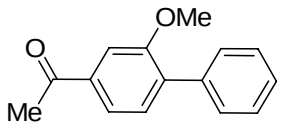
EA:Hexane = 1:9, R_f=0.35; White solid, m.p. = 69.5-71.8 °C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.60 (s, 3H), 7.45-7.50 (m, 3H), 7.68 (dd, *J*=1.6, 8.5 Hz, 1H), 7.78-7.89 (m, 5H), 8.00 (bs, 1H), 8.30 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.2, 124.7, 125.5, 125.8, 126.0, 126.5, 126.8, 127.2, 127.8, 128.2, 128.6, 131.3, 132.4, 133.2, 136.8, 137.2, 140.9, 197.5; MS (EI): *m/z* (relative intensity) 246 (M^+, 100), 231 (90), 202 (95). HRMS: calcd. for $C_{18}H_{14}O$: 247.1123, found 247.1106.

3'-Methoxy-4'-phenylbenzaldehyde (Table 2, entry 9).[24]



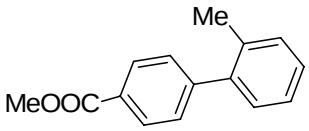
EA:Hexane = 1:9, R_f=0.35; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.86 (s, 3H), 7.39-7.59 (m, 8H), 10.00 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.3, 109.4, 124.0, 127.6, 127.8, 129.1, 130.9, 136.4, 136.8, 136.9, 156.7, 191.5; MS (EI): *m/z* (relative intensity) 212 (M^+, 100), 169 (20), 152 (10), 139 (20), 115(15).

3'-Methoxy-4'-phenylacetophenone (Table 2, entry 10).[19]



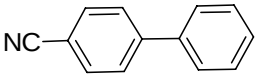
EA:Hexane = 1:9, R_f=0.3; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.61 (s, 3H), 3.82 (s, 3H), 7.37-7.46 (m, 4H), 7.57-7.62 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 26.1, 55.1, 109.5, 121.3, 127.2, 129.0, 130.3, 135.1, 136.8, 137.0, 156.2, 197.0; MS (EI): *m/z* (relative intensity) 226 (M^+, 80), 211 (100), 183 (10), 168 (30).

Methyl 4-(2'-tolyl)benzoate (Table 2, entry 11).



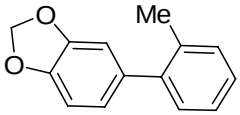
EA:Hexane = 1:9, R_f=0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.3 (s, 3H), 3.96 (s, 3H), 7.24-7.32 (m, 4H), 7.42(d, *J* = 8.0 Hz, 2H), 8.14 (d, *J* = 8.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.1, 125.6, 127.5, 128.3, 129.0, 129.1, 129.2, 130.2, 134.8, 140.5, 146.4, 166.6; MS (EI): *m/z* (relative intensity) 226 (M^+, 100), 195 (98), 168 (60), 152 (50), 139 (7).

4-Cyanobiphenyl (Table 2, entry 12).[28]



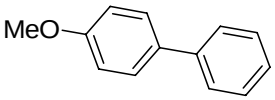
DCM:Hexane = 1:20, R_f=0.35; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43-7.50 (m, 3H), 7.57-7.59 (m, 2H), 7.64-7.70 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 110.6, 118.6, 126.9, 127.4, 128.4, 128.8, 132.3, 138.8, 145.2; MS (EI): *m/z* (relative intnsity) 179 (M^+, 100), 151 (20).

2'-Methyl- 3,4-methylenedioxy biphenyl (Table 2, entry 13).[23]



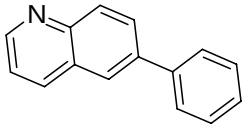
EA:Hexane = 1:4, R_f=0.7; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.44 (s, 3H), 6.07 (s, 2H), 6.91-7.01 (m, 3H), 7.37-7.39 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.3, 100.8, 107.8, 109.6, 122.3, 125.6, 127.0, 129.6, 130.1, 135.2, 135.7, 141.4, 146.3, 147.1; MS (EI): *m/z* (relative intnsity) 212 (M^+, 100), 181 (20), 153 (30).

4-Phenylanisole (Table 2, entry 14).[17]



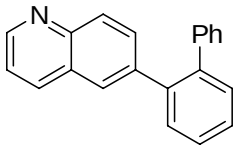
EA:Hexane = 1:20, R_f=0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.97 (s, 3H), 7.17 (d, *J*=8.7 Hz, 2H), 7.51-7.54 (m, 1H), 7.62 (t, *J*=7.8 Hz, 2H), 7.73-7.79 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.0, 114.1, 126.5, 127.9, 128.3, 128.6, 133.5, 140.6, 159.0; MS (EI): *m/z* (relative intensity) 184 (M^+, 100), 169 (50), 141 (50), 115 (40).

6-Phenylquinoline (Table 3, entry 1).[22]



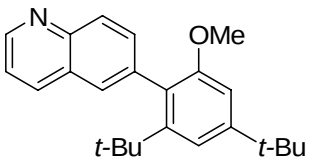
EA:Hexane = 1:4, R_f=0.25; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21-7.33 (m, 2H), 7.37-7.71 (m, 2H), 7.58-7.60 (m, 2H), 7.82-7.87 (m, 2H), 7.96-8.15 (m, 2H), 8.83 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 121.0, 125.0, 127.0, 127.3, 128.0, 128.5, 128.7, 129.4, 135.7, 138.7, 139.7, 147.2, 149.9; MS (EI): *m/z* (relative intnsity) 205 (M^+, 100), 176 (10).

5-(2-Biphenyl)quinoline (Table 3, entry 2).



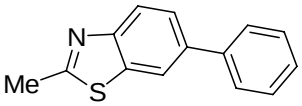
DCM, R_f=0.2; light yellow viscous liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.18 (bs, 5H), 7.25-7.54 (m, 6H), 7.69 (s, 1H), 7.94-7.98 (m, 2H), 8.85 (d, *J*= 4.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 120.8, 126.4, 127.4, 127.7, 127.8, 127.9, 128.3, 129.6, 130.4, 130.5, 131.7, 135.6, 139.2, 139.7, 140.4, 140.8, 146.8, 149.9; MS (EI): *m/z* (relative intensity) 281 (M^+, 100), 266 (30), 252 (20); HRMS: calcd. for $C_{21}H_{15}N$: 281.1199, found 281.1203.

6-(2,4-Di-*tert*-butyl-6-methoxyphenyl)quinoline (Table 3, entry 3).



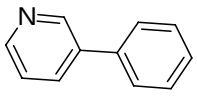
EA:Hexane = 1:9, R_f=0.15; white solid, m.p. = 136.4-138.1 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.19 (s, 9H), 1.42 (s, 9H), 3.56 (s, 3H), 6.92 (s, 1H); 7.24-7.31 (m, 4H), 8.02 (d, *J*=7.9 Hz, 1H), 8.14 (d, *J*=8.5 Hz, 1H), 8.87 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 31.1, 32.5, 34.8, 36.6, 55.5, 105.7, 116.0, 120.5, 127.0, 127.2, 127.5, 129.2, 133.9, 135.5, 138.3, 147.0, 148.4, 149.6, 150.6, 157.3; MS (EI): *m/z* (relative intensity) 347 (M^+, 100), 332 (75), 317 (5), 302 (3), 290 (5), 276(75), 261(30); HRMS: calcd. for $C_{24}H_{29}NO$: 347.2244, found 347.2253.

2-Methyl-5-phenylbenzothiazole (Table 3, entry 4).[20]



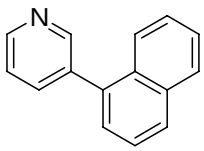
EA:Hexane = 1:4, R_f=0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.73 (s, 3H), 7.22-7.24 (m, 1H), 7.40 (t, *J*=8.3Hz, 2H), 7.62 (d, *J*=7.3Hz, 1H), 7.69 (d, *J*=8.3Hz, 2H), 8.21 (d, *J*=1.1Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.5, 120.1, 121.0, 123.5, 126.8, 126.9, 128.4, 134.1, 138.9, 140.2, 153.6, 167.0; MS (EI): *m/z* (relative intensity) 225 (M^+, 100), 184 (10), 152 (10).

3-Phenyl pyridine (Table 3, entry 5). [25]



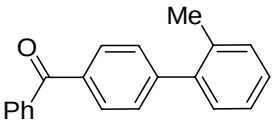
EA:Hexane = 3:7, R_f=0.5; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.83 (m, 7H), 8.5-8.58 (m, 1H), 8.85 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 123.3, 126.8, 127.9, 128.8, 134.2, 136.4, 137.4, 147.8, 147.9; MS (EI): *m/z* (relative intensity) 155 (M^+, 100), 127 (10), 102 (7).

3-(1-Naphthalenyl) pyridine (Table 3, entry 6).[29]



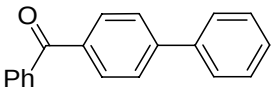
EA:Hexane = 1:4, R_f=0.3; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34-7.48 (m, 5H), 7.50-7.86 (m, 4H), (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 122.7, 124.8, 125.0, 125.7, 126.1, 127.0, 128.1, 131.0, 133.4, 135.8, 135.9, 136.9, 148.1, 150.1; MS (EI): *m/z* (relative intensity) 205 (M^+, 90), 204 (100), 176 (18), 151 (10).

4-(2'-Tolyl)benzophenone (Scheme 2).[21]



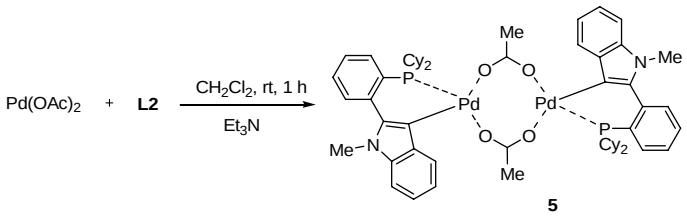
EA:Hexane = 1:20, R_f=0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.36 (s, 3H,), 7.30-7.33 (m, 4H), 7.46-7.61 (m, 5H), 7.90-7.93 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.2, 125.7, 127.6, 128.0, 128.9, 129.3, 129.7, 130.3, 132.0, 135.6, 137.4, 140.4, 146.0, 195.9; MS (EI): *m/z* (relative intensity) 272 (M^+, 70), 195 (100), 164 (40), 152 (40), 105 (40).

4-Phenylbenzophenone (Scheme 2).[26]

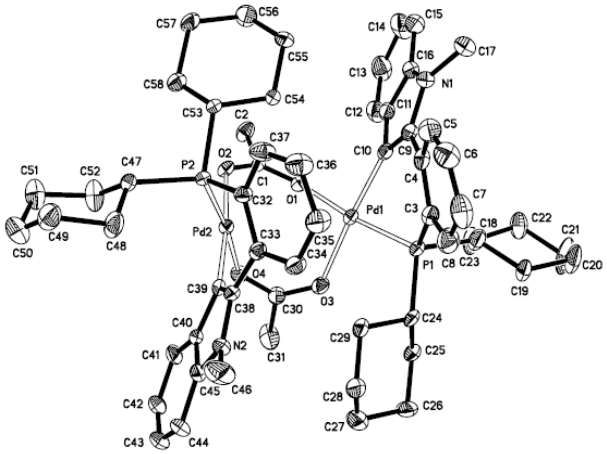


EA:Hexane = 1:20, R_f=0.4; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.42 (m, 1H,), 7.46-7.51 (m, 4H), 7.57-7.61 (m, 1H), 7.65-7.71 (m, 4H), 7.82-7.92 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 126.6, 126.9, 127.9, 128.0,128.6, 129.6, 130.4, 132.0, 135.8, 137.4, 139.5, 144.7, 195.7; MS (EI): *m/z* (relative intensity) 258 (M^+, 75), 181 (100), 152 (40), 105 (20), 77 (19).

8. Preparation of single-component complex 5

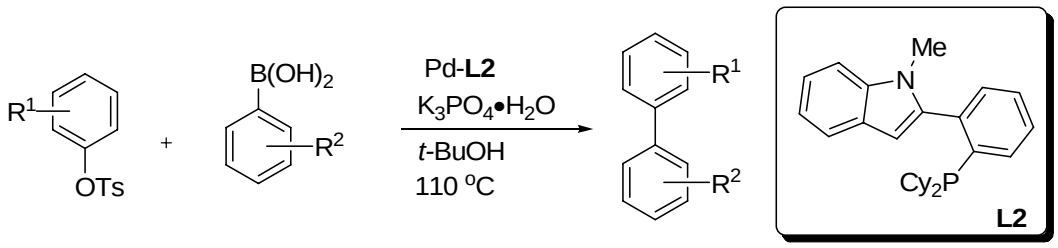


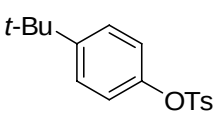
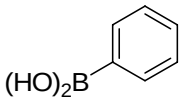
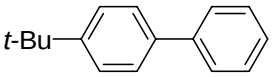
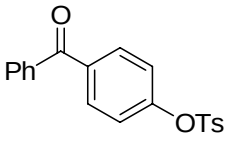
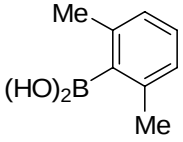
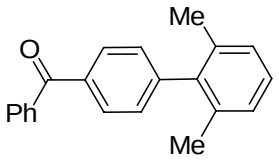
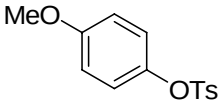
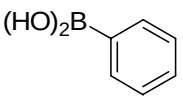
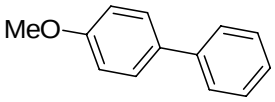
Palladium(II) acetate (224 mg, 1.0 mmol) and **L2** (1.0 mmol) were dissolved in freshly distilled dichloromethane (10 mL) and anhydrous Et_3N (2.0 mmol) under nitrogen at room temperature. The red solution was stirred for one hour. Solvent was partially removed under reduced pressure. Anhydrous diethyl ether was then slowly added and reddish brown precipitates were obtained (70%). Crystals for X-ray diffraction were obtained by recrystallization of the pure product from dichloromethane/hexane layers.



^{1}H NMR (400 MHz, CD_2Cl_2) δ 8.01 (d, J = 7.0 Hz, 1 H), 7.54-7.62 (m, 2 H), 7.45-7.49 (m, 1 H), 7.20-7.34 (m, 3 H), 7.07 (t, J = 7.2 Hz, 1 H), 3.83 (s, 3H), 1.11-2.07 (m, 30H); ^{13}C NMR (100 MHz, CD_2Cl_2) δ 143.1, 141.2 (d, J_{CP} = 10.6 Hz), 137.6 (d, J_{CP} = 26.8 Hz), 134.2, 133.7, 131.4, 128.6, 126.4, 126.3, 124.9, 123.1, 122.4, 119.9, 110.2, 104.0 (d, J_{CP} = 14.5 Hz), 35.6 (d, J_{CP} = 28.1 Hz), 34.5, 29.3, 28.2, 27.7, 26.5, 24.6; ^{31}P NMR (162 MHz, CD_2Cl_2) δ +44.0. Full X-ray data were reported in supplied cif file.

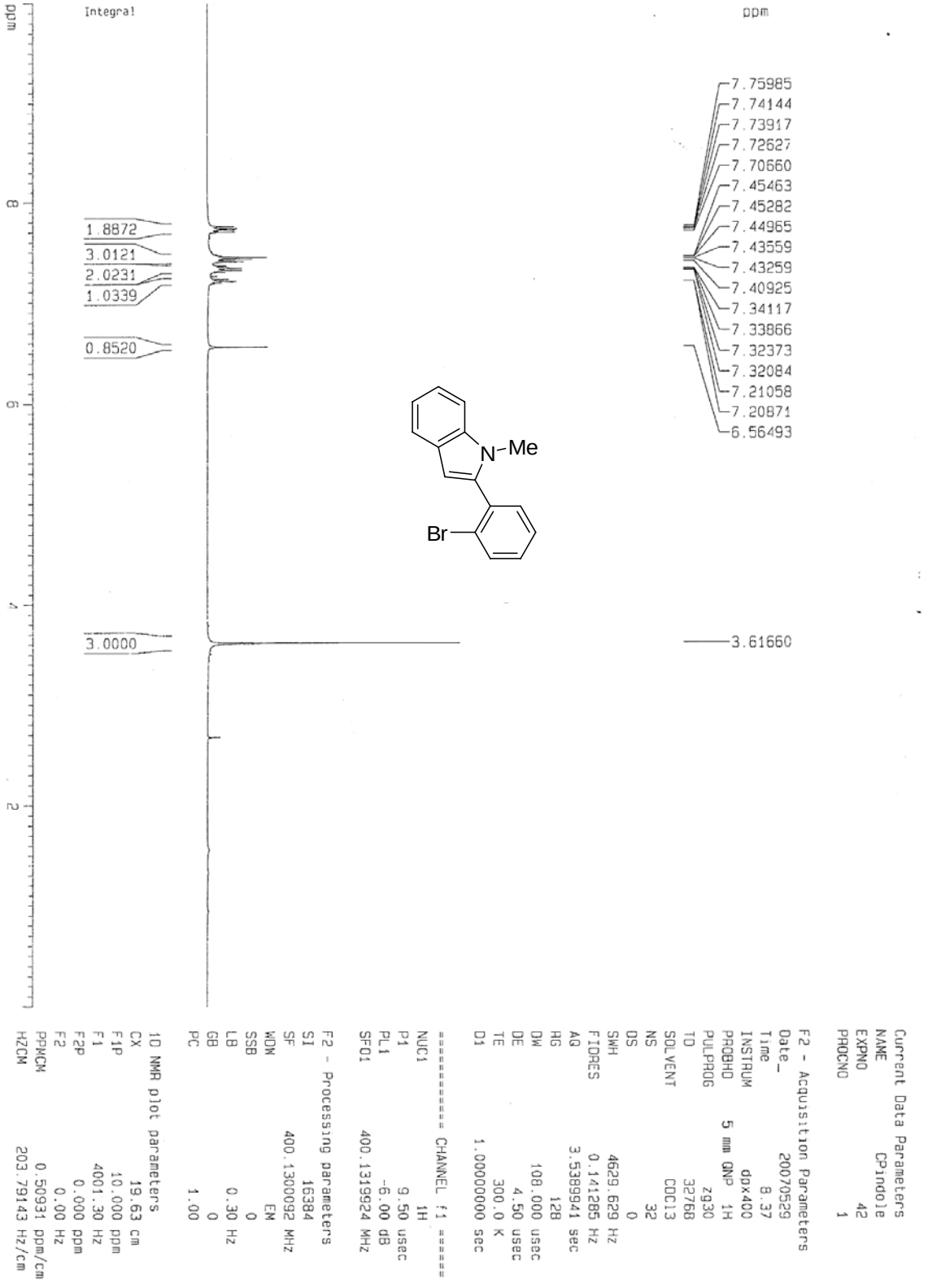
9. Data from aryl tosylate couplings (for comparison)

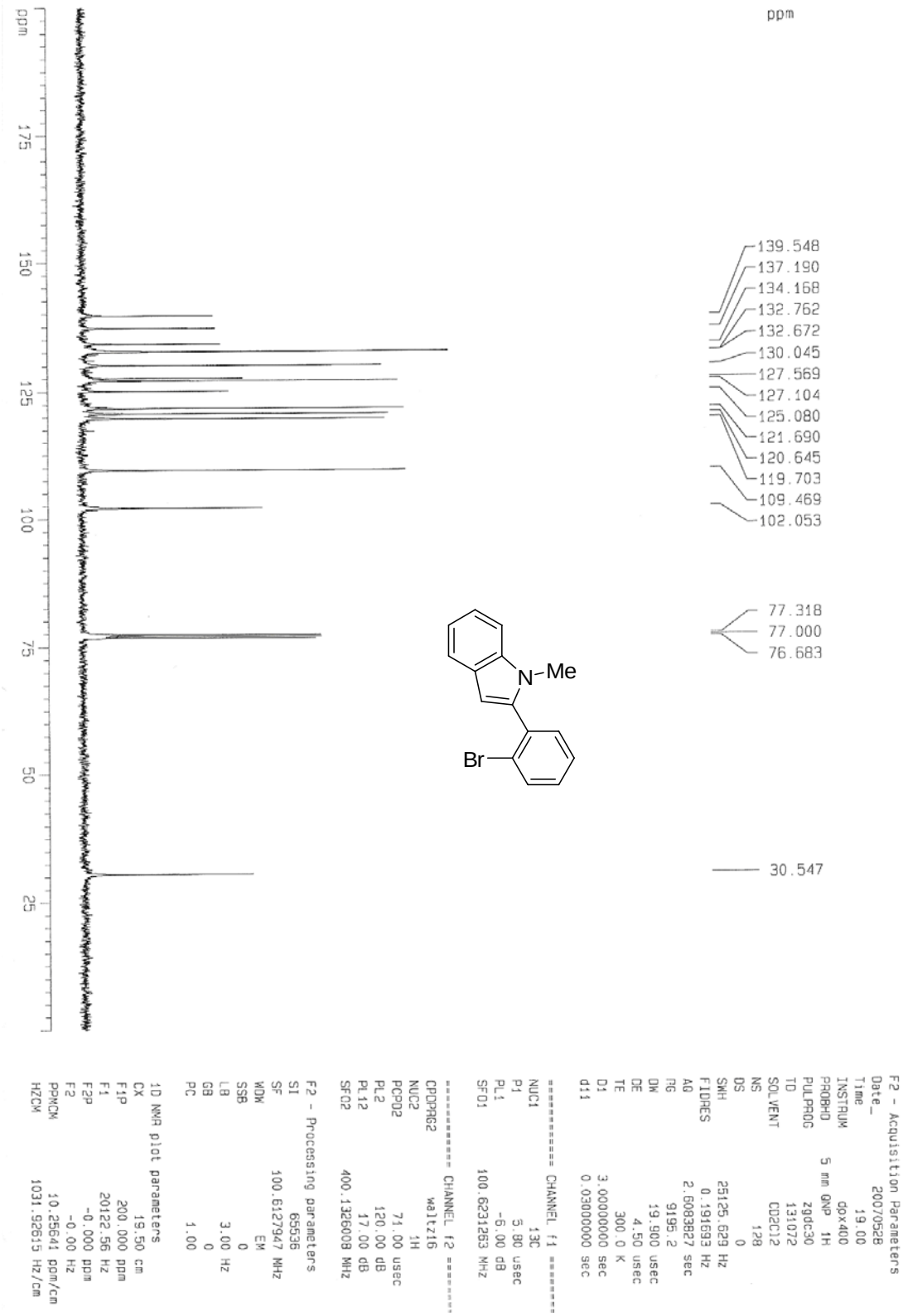


entry	ArOTs	Ar'B(OH)$_2$	product	mol% Pd	% yield
1				0.2%, 12.5 h	85
2				0.5%, 2 h	90
3				3%, 4 h	90

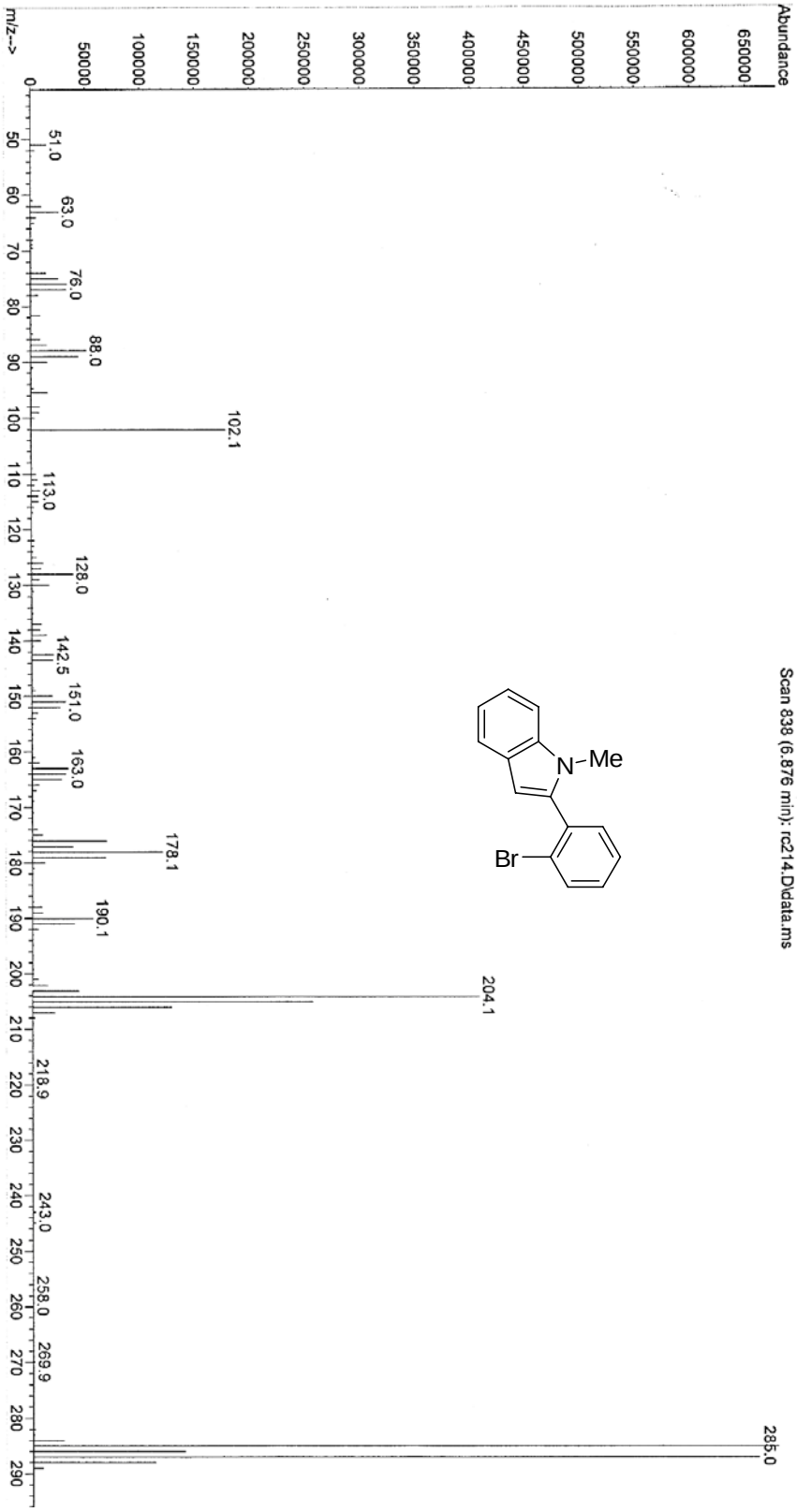
Reaction conditions: ArOTs (1.0 mmol), Ar'B(OH)$_2$ (2.0 mmol), K_3PO_4 (3.0 mmol), Pd(OAc)$_2$/**L2** (mol% as indicated), *t*-BuOH (3.0 mL), at 110 °C under N_2 for indicated period of time.

10. ^{1}H, ^{13}C, ^{31}P NMR, MS, HRMS and IR spectra

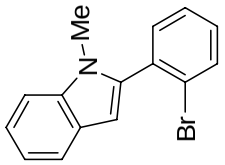





```
F2 - Acquisition Parameters
Date_              20070528
Time                   19.00
INSTRUM              dpx400
PROBHD     5 mm QNP 1H
PULPROG             zgdc30
TD                  131072
SOLVENT             CDCl3
NS                     128
DS                       0
SWH                25125.629 Hz
FIDRES                0.191693 Hz
AQ                 2.6083827 sec
RG                      9195.2
DW                     19.900 usec
DE                      4.50 usec
TE                     300.0 K
D1                  3.00000000 sec
d11                 0.03000000 sec

============ CHANNEL f1 =======
NUC1                      13C
P1                       5.80 usec
PL1                     -6.00 dB
SFO1              100.6231263 MHz

============ CHANNEL f2 =======
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   71.00 usec
PL2                    120.00 dB
PL12                    17.00 dB
SFO2              400.1326008 MHz

F2 - Processing parameters
SI                        65536
SF                100.6127947 MHz
WDW                          EM
SSB                           0
LB                         3.00 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                        19.50 cm
F1P                     200.000 ppm
F1                    20122.56 Hz
F2P                    -0.000 ppm
F2                      -0.00 Hz
PPMCM                  10.25641 ppm/cm
HZCM                1031.92615 Hz/cm
```


Abundance
m/z-->
Scan 838 (6.876 min): rc214.D\data.ms
N-Me
Br
51.0
63.0
76.0
88.0
102.1
113.0
128.0
142.5
151.0
163.0
178.1
190.1
204.1
218.9
243.0
258.0
269.9
285.0

Elemental Composition Report



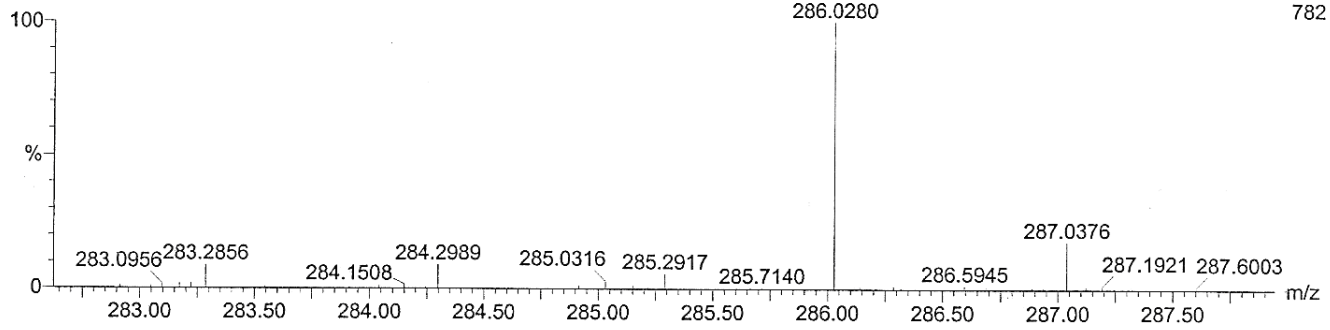
Single Mass Analysis

Tolerance = 100.0 PPM / DBE: min = -1.5, max = 60.0
Selected filters: None

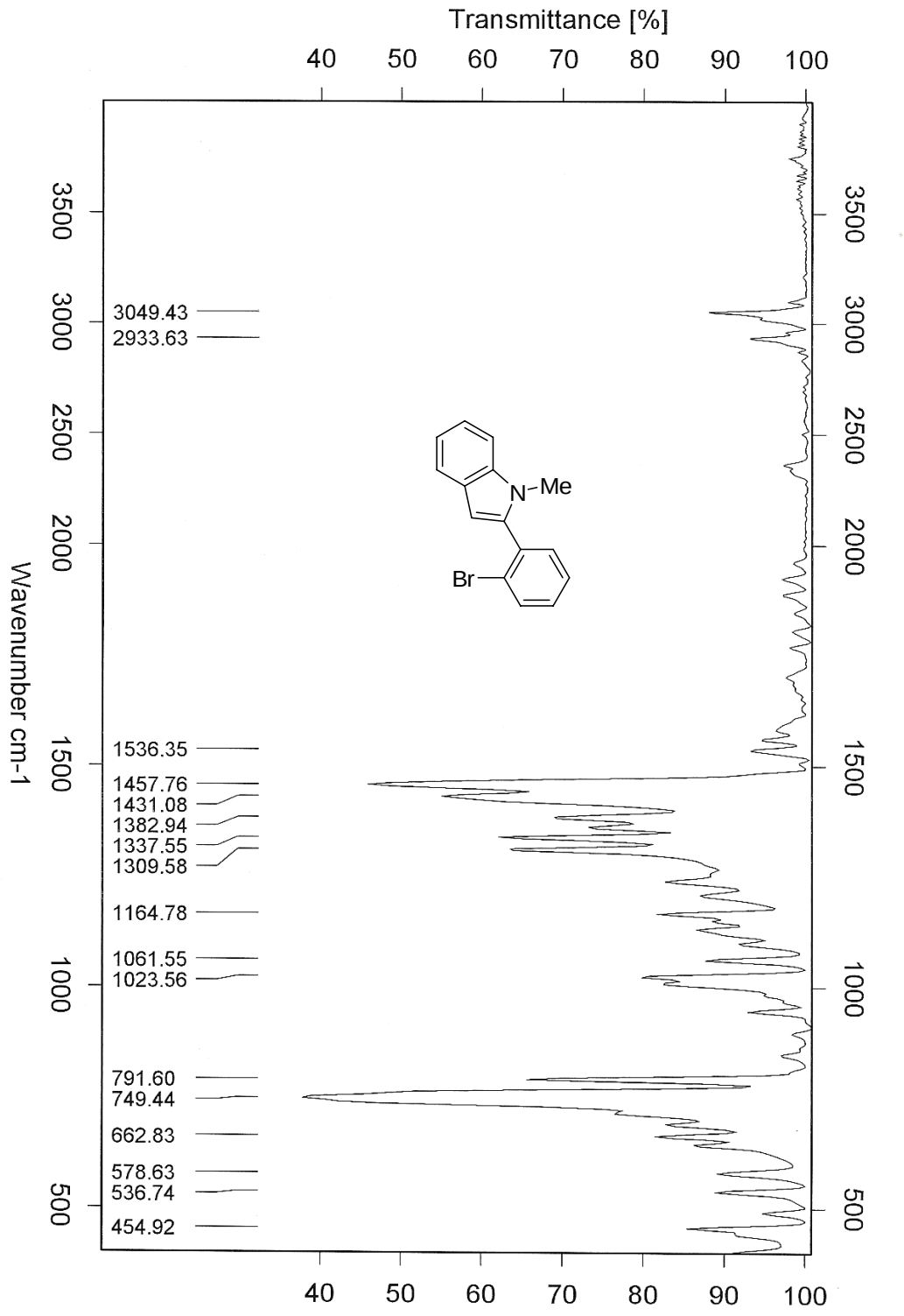
Monoisotopic Mass, Even Electron Ions
7 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-15 H: 0-1000 N: 0-1 Br: 0-1

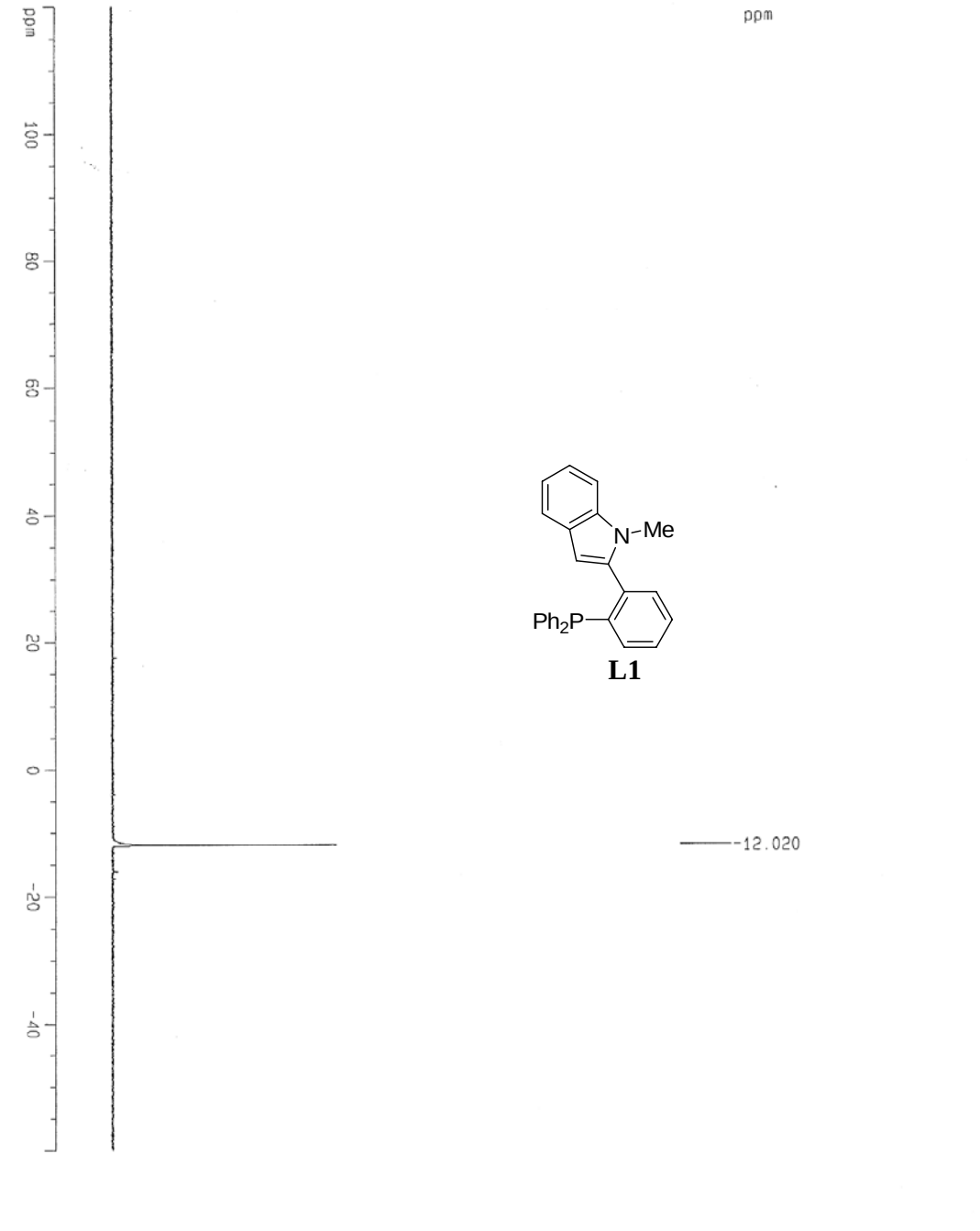
So Chau Ming, N-methyl 2(2Bromophenyl)indole
HR07_0615_4B 41 (0.778) AM (Cen,5, 80.00, Ht,10000.0,0.00,0.70); Sm (SG, 3x2.00); Cm (41:59)

15-Jun-2007
TOF MS ES+
782



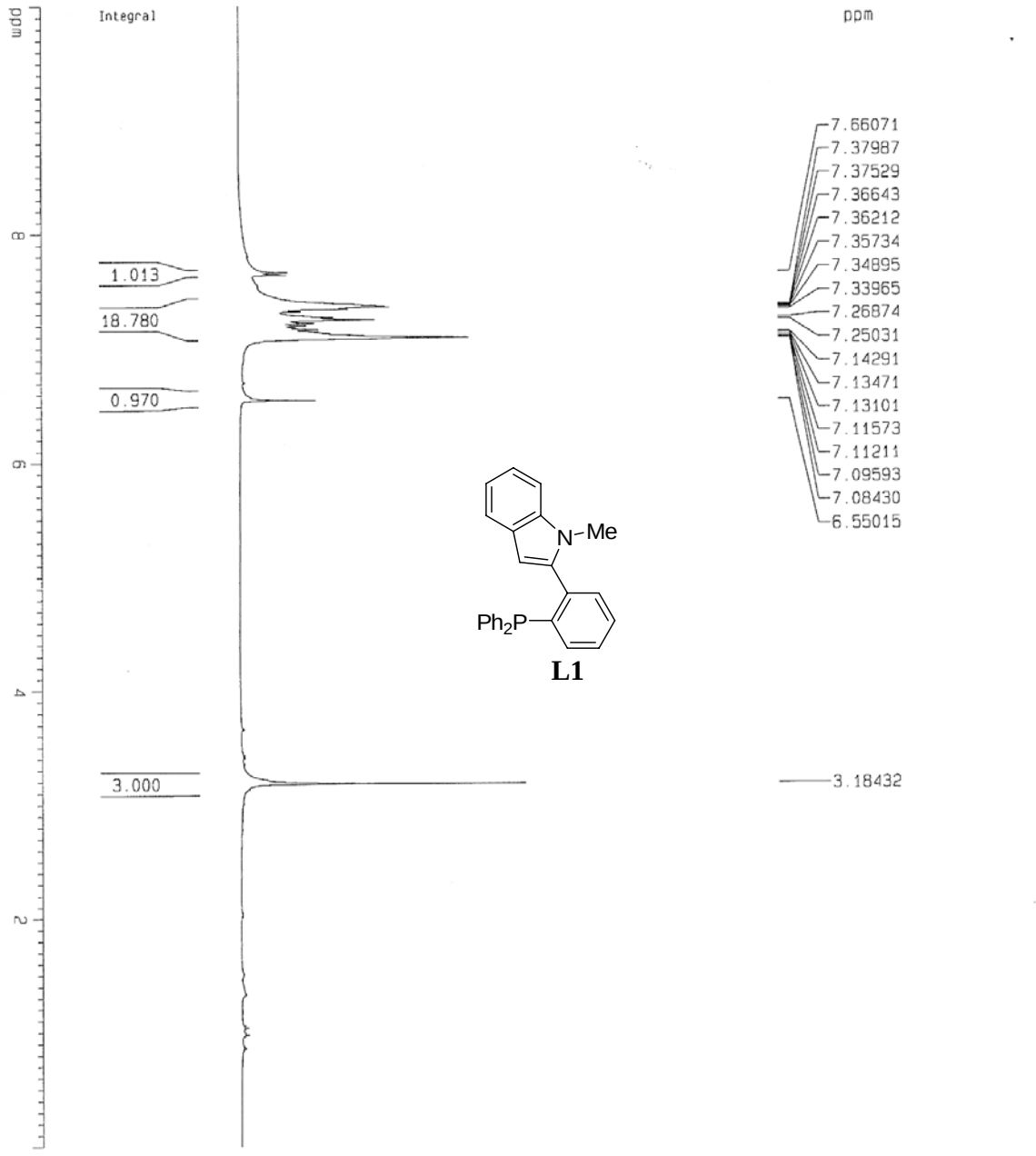
Minimum:				-1.5		
Maximum:		5.0	100.0	60.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
286.0280	286.0231	4.9	17.1	9.5	463.5	C15 H13 N Br


Transmittance [%]
40
50
60
70
80
90
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3049.43
2933.63
1536.35
1457.76
1431.08
1382.94
1337.55
1309.58
1164.78
1061.55
1023.56
791.60
749.44
662.83
578.63
536.74
454.92
N-Me
Br

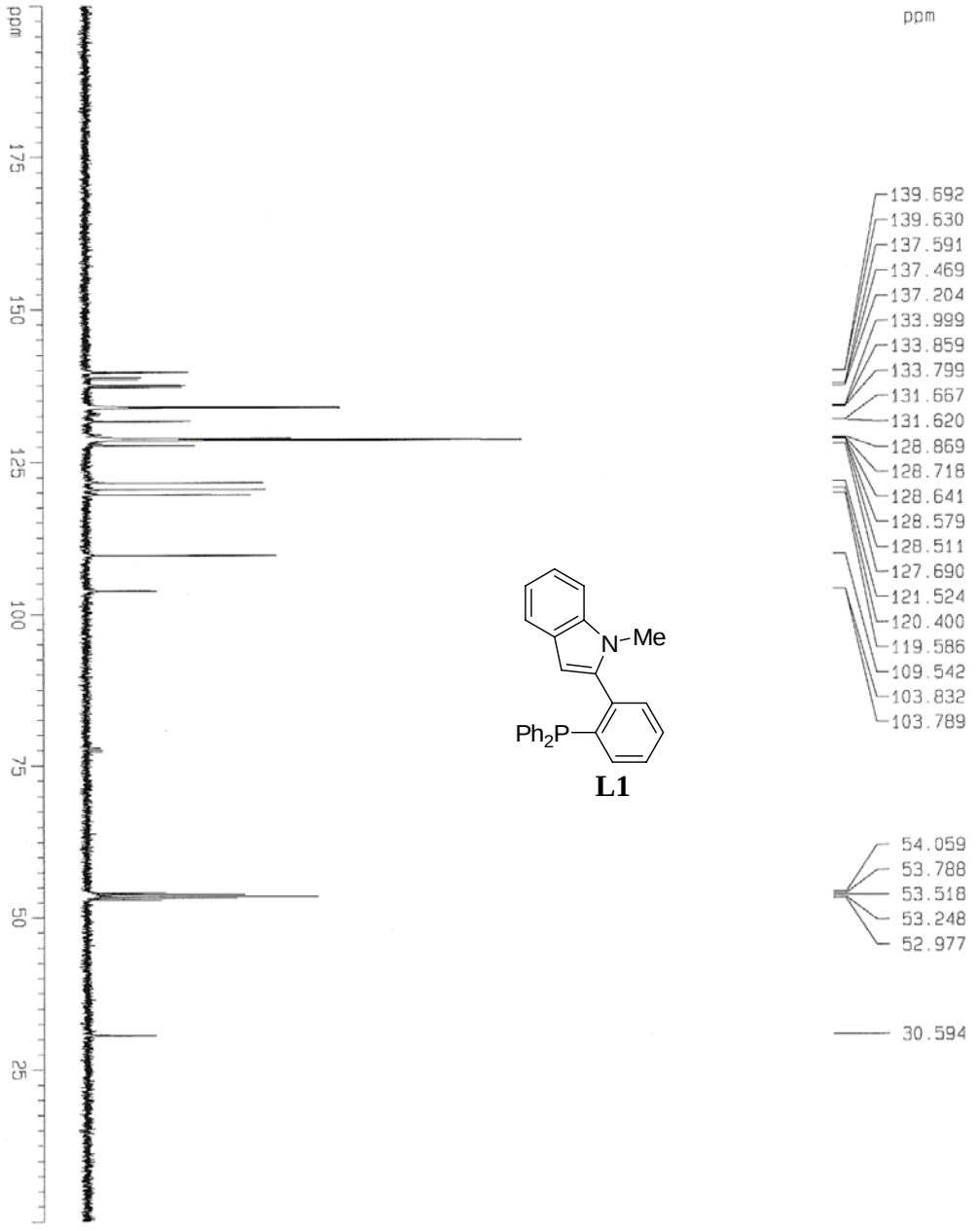


L1

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

F2 - Acquisition Parameters
Date_              20070529
Time                   13.06
INSTRUM                dpx400
PROBHD     5 mm GNP 1H
PULPROG                zg0c30
TD                     65536
SOLVENT                C606
NS                        32
DS                         0
SWH                48651.901 Hz
FIDRES             0.742520 Hz
AQ                 0.6734324 sec
RG                      13004
DW                     10.275 usec
DE                      4.50 usec
TE                     300.0 K
D1                 1.00000000 sec
D11                0.03000000 sec

============== CHANNEL f1 ======
NUC1                      31P
P1                       7.20 usec
PL1                     -5.00 dB
SF01           161.9851115 MHz

============== CHANNEL f2 ======
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  71.00 usec
PL2                    120.00 dB
PL12                    17.00 dB
SF02           400.1324008 MHz

F2 - Processing parameters
SI                        32768
SF             161.9755831 MHz
WDW                          EM
SSB                           0
LB                         3.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                        20.00 cm
F1P                      120.000 ppm
F1                     19437.07 Hz
F2P                     -60.000 ppm
F2                     -9718.54 Hz
PPMCM                  9.00000 ppm/cm
HZCM                1457.78027 Hz/cm
```

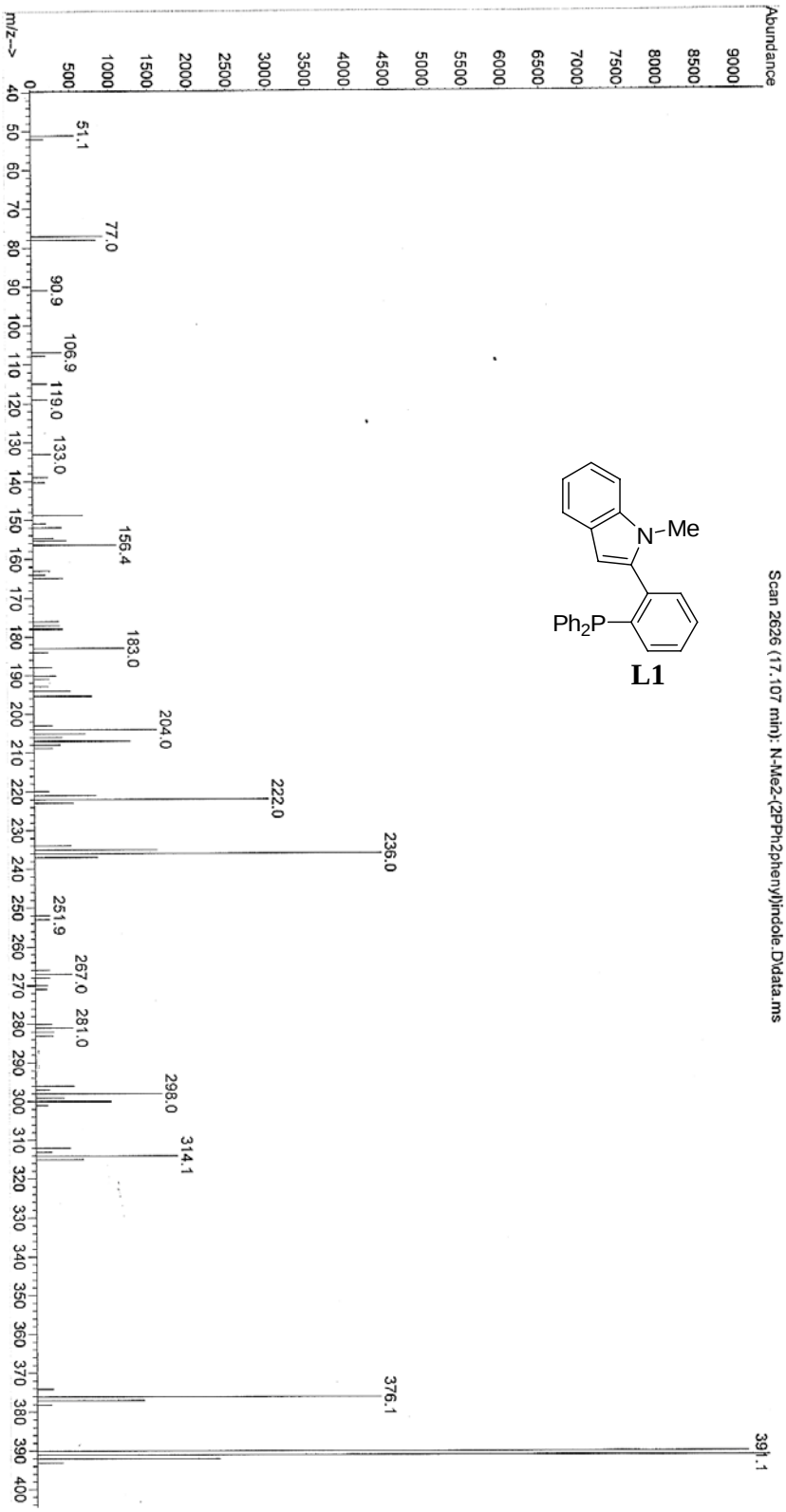



```
Current Data Parameters
NAME                  CPindole
EXPNO                       44
PROCNO                       1

F2 - Acquisition Parameters
Date_              20070529
Time                    13.12
INSTRUM                 dpx400
PROBHD     5 mm QNP 1H
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                         32
DS                          0
SWH                4629.629 Hz
FIDRES             0.141285 Hz
AQ                 3.5389941 sec
RG                       90.5
DW                 108.000 usec
DE                   4.50 usec
TE                  300.0 K
D1                 1.00000000 sec

======== CHANNEL f1 ======
NUC1                       1H
P1                   9.50 usec
PL1                  -6.00 dB
SFO1           400.1319924 MHz

F2 - Processing parameters
SI                       16384
SF                 400.1300092 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                    19.63 cm
F1P                 10.000 ppm
F1                  4001.30 Hz
F2P                  0.000 ppm
F2                     0.00 Hz
PPMCM              0.50931 ppm/cm
HZCM             203.79143 Hz/cm
```



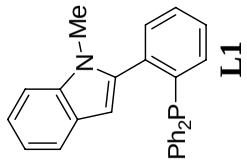
L1

```
Current Data Parameters
NAME                  CCPindole
EXPNO                        25
PROCNO                        1

F2 - Acquisition Parameters
Date_              20070529
Time               12.53
INSTRUM            dpx400
PROBHD     5 mm QNP 1H
PULPROG            zgdc30
TD                 131072
SOLVENT            CD2Cl2
NS                 96
DS                 0
SWH                25125.629 Hz
FIDRES             0.191693 Hz
AQ                 2.6083827 sec
RG                 9195.2
DW                 19.900 usec
DE                 4.50 usec
TE                 300.0 K
D1                 3.00000000 sec
d11                0.03000000 sec

============ CHANNEL f1 =======
NUC1               13C
P1                 5.80 usec
PL1                -6.00 dB
SF01               100.6231263 MHz

============ CHANNEL f2 =======
CPDPRG2            waltz16
NUC2               1H
PCPD2              71.00 usec
PL2                120.00 dB
PL12               17.00 dB
SF02               400.1326008 MHz

F2 - Processing parameters
SI                 65536
SF                 100.6127679 MHz
WDW                EM
SSB                0
LB                 1.00 Hz
GB                 0
PC                 1.00

1D NMR plot parameters
CX                 19.50 cm
F1P                200.000 ppm
F1                 20122.55 Hz
F2P                -0.000 ppm
F2                 -0.00 Hz
PPMCM              10.25641 ppm/cm
HZCM               1031.92590 Hz/cm
```


Abundance
m/z-->
Scan 2626 (17.107 min): N-Me2-(2PPh2phenyl)indole.D\data.ms
51.1
77.0
90.9
106.9
119.0
133.0
155.4
183.0
204.0
222.0
236.0
251.9
267.0
281.0
298.0
314.1
376.1
391.1
N-Me
Ph2P

L1

Elemental Composition Report



L1

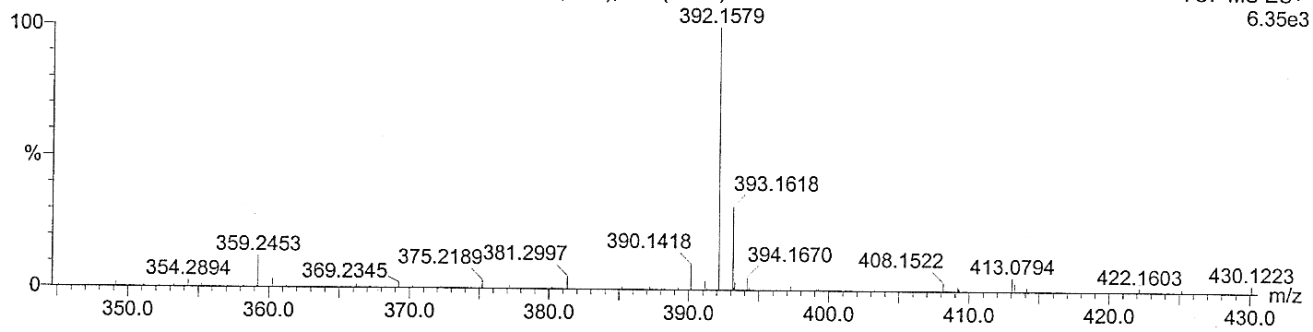
Single Mass Analysis

Tolerance = 100.0 PPM / DBE: min = -1.5, max = 60.0
Selected filters: None

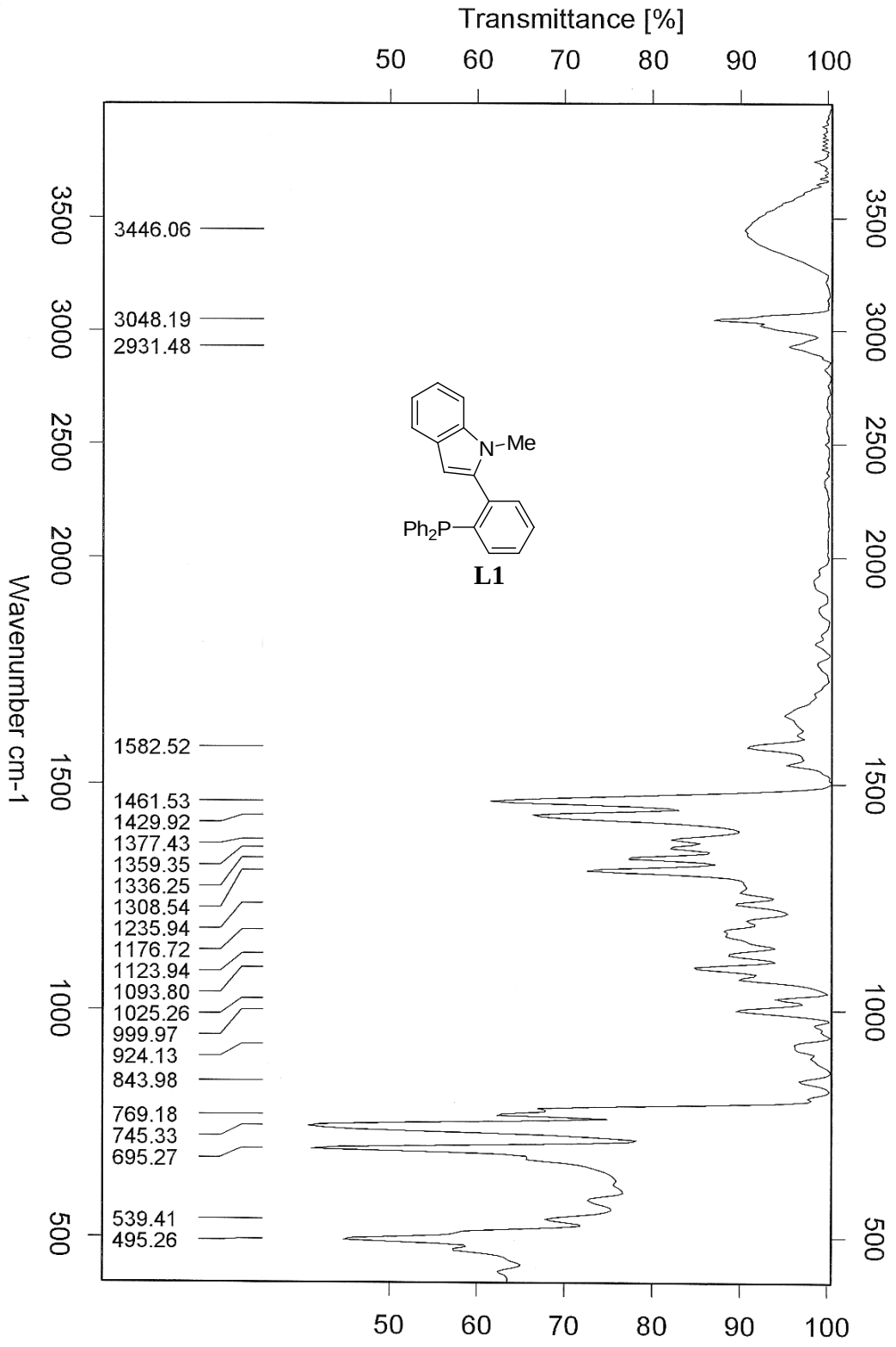
Monoisotopic Mass, Even Electron Ions
9 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-27 H: 0-1000 N: 0-1 P: 0-1

So Chau Ming, 2-(N-methylindolylphenylPh2phosphine
HR07_0615_7 66 (1.249) AM (Cen,5, 80.00, Ar,10000.0,0.00,0.70); Cm (66:77)

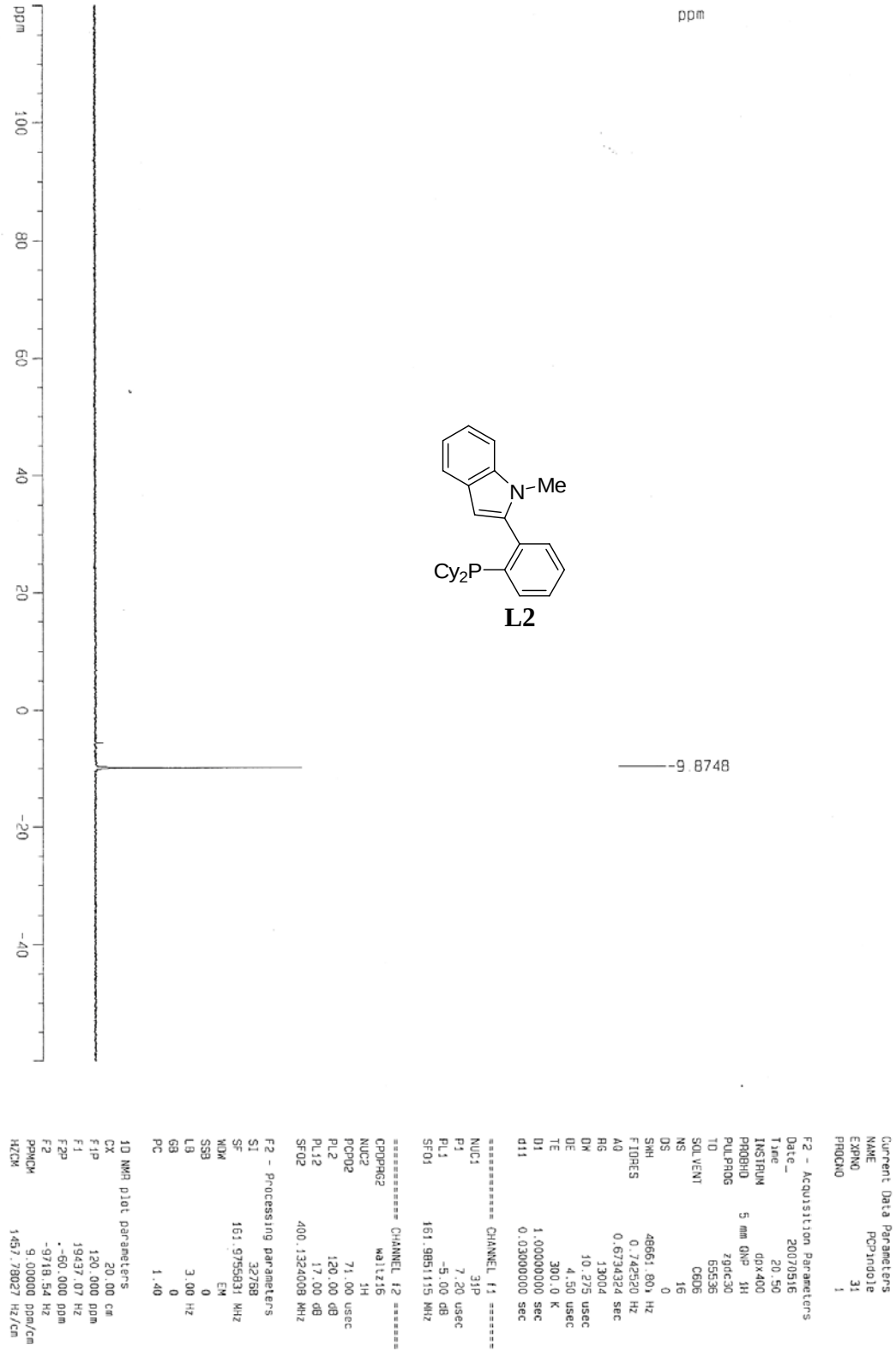
15-Jun-2007
TOF MS ES+
6.35e3



Minimum:				-1.5		
Maximum:		5.0	100.0	60.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
392.1579	392.1568	1.1	2.8	17.5	1.2	C27 H23 N P

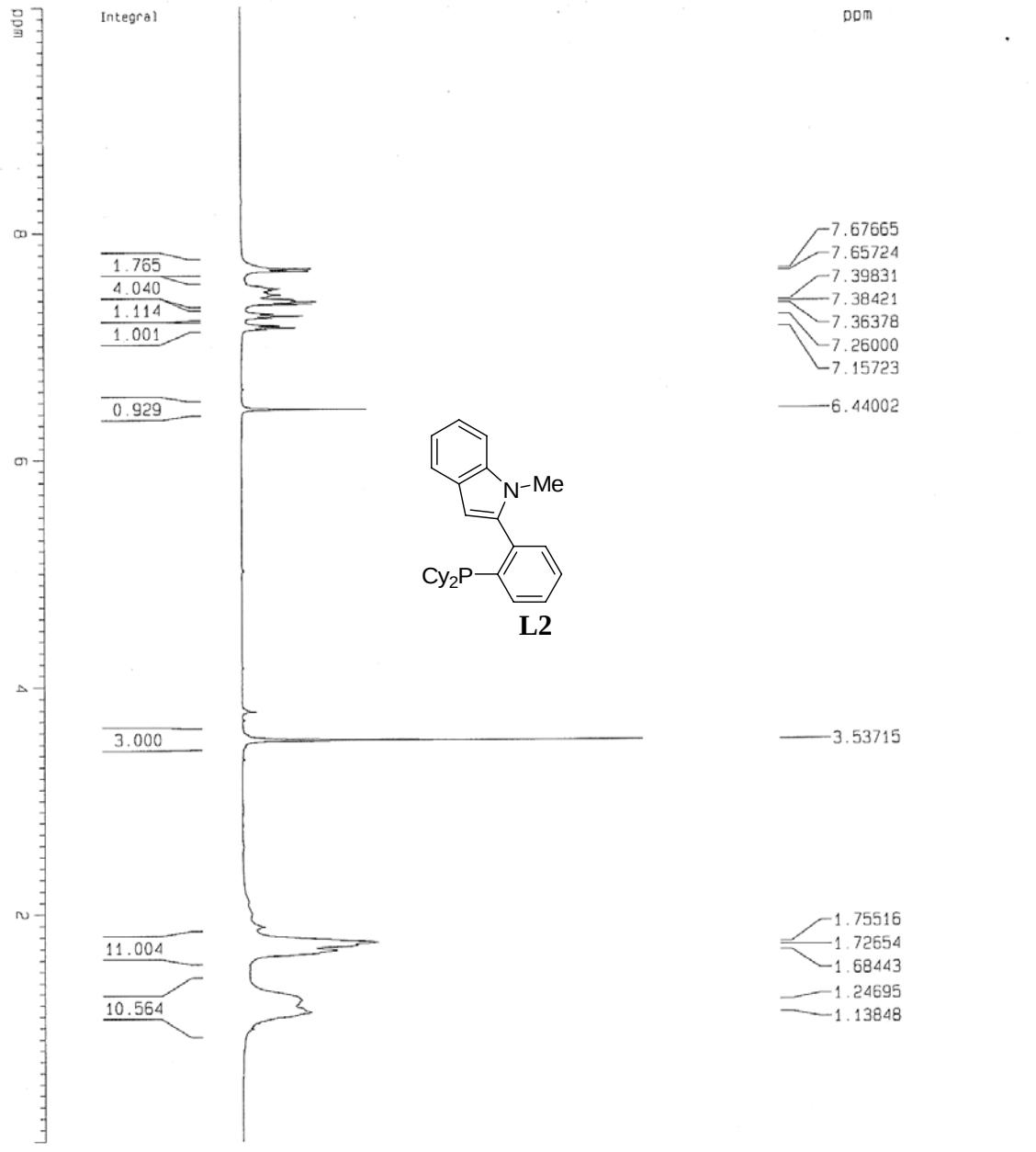

Transmittance [%]
50
60
70
80
90
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3446.06
3048.19
2931.48
1582.52
1461.53
1429.92
1377.43
1359.35
1336.25
1308.54
1235.94
1176.72
1123.94
1093.80
1025.26
999.97
924.13
843.98
769.18
745.33
695.27
539.41
495.26
N-Me
Ph2P

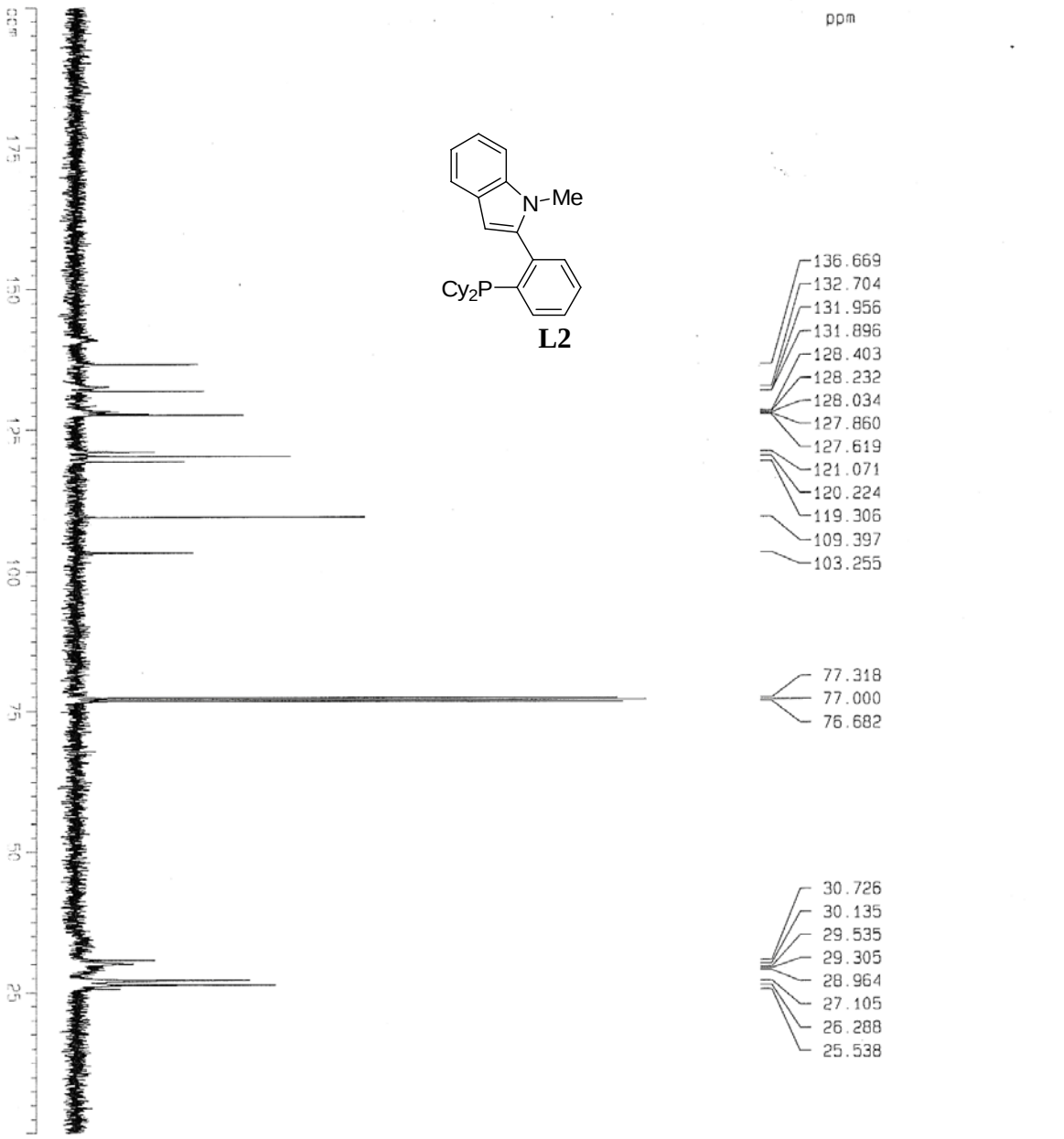
L1



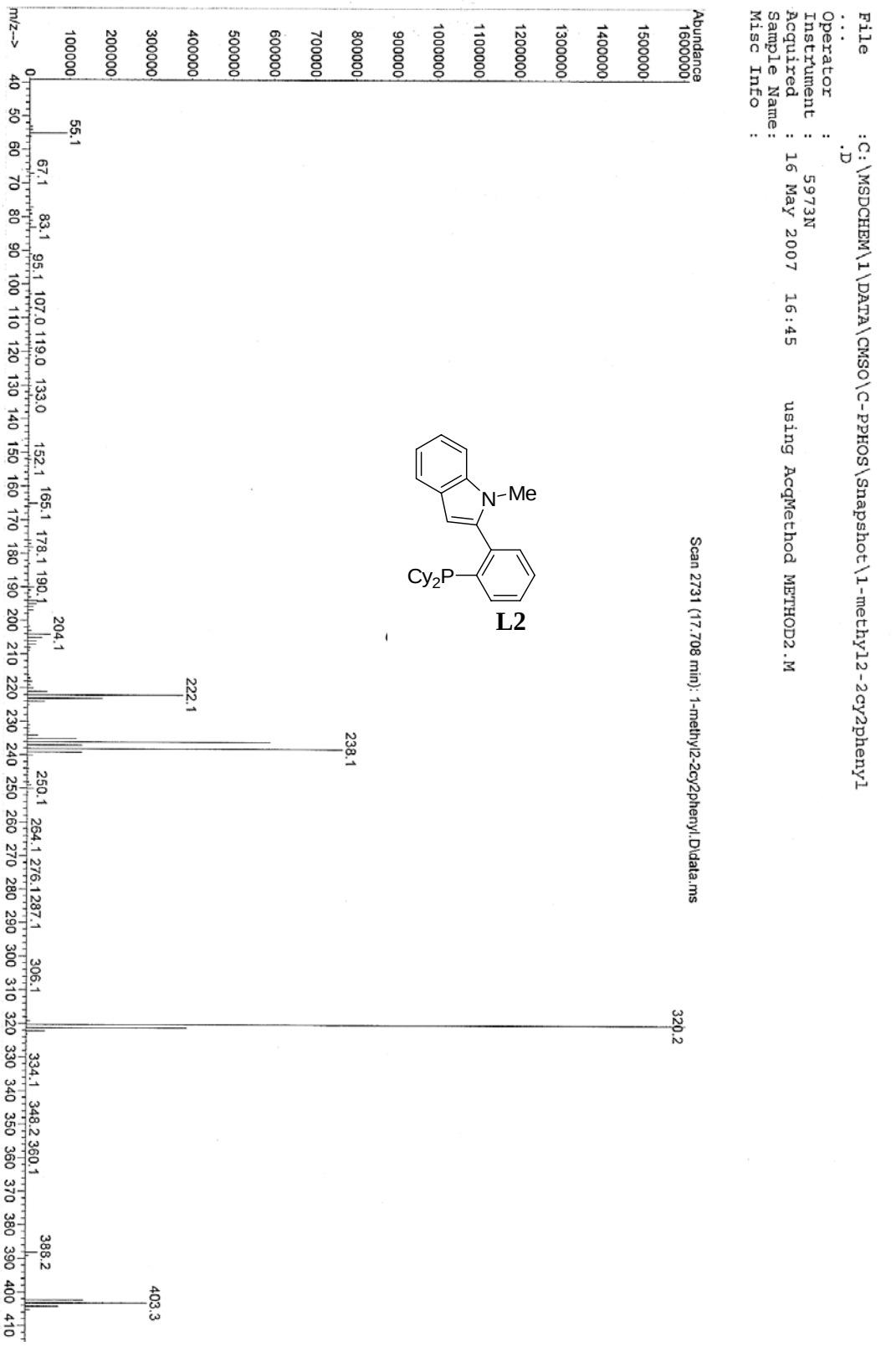
L2


```
Current Data Parameters
NAME                  PCPindole
EXPNO                        31
PROCNO                        1

F2 - Acquisition Parameters
Date_              20070516
Time                   20.50
INSTRUM              dpx400
PROBHD     5 mm BNP 1H
PULPROG              zgdc30
TD                    65536
SOLVENT               C606
NS                       16
DS                        0
SWH              48661.80 Hz
FIDRES               0.742520 Hz
AQ                0.6734324 sec
RG                      13004
DW                   10.275 usec
DE                    4.50 usec
TE                   300.0 K
D1                1.00000000 sec
d11               0.03000000 sec

======== CHANNEL f1 =======
NUC1                     31P
P1                    7.20 usec
PL1                  -5.00 dB
SFO1           161.9851115 MHz

======== CHANNEL f2 =======
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 71.00 usec
PL2                  120.00 dB
PL12                  17.00 dB
SFO2           400.1324008 MHz

F2 - Processing parameters
SI                        32768
SF             161.9755831 MHz
WDW                          EM
SSB                           0
LB                         3.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                       20.00 cm
F1P                     120.000 ppm
F1                     19437.07 Hz
F2P                    -60.000 ppm
F2                     -9718.54 Hz
PPMCM                  9.00000 ppm/cm
HZCM                1457.78027 Hz/cm
```

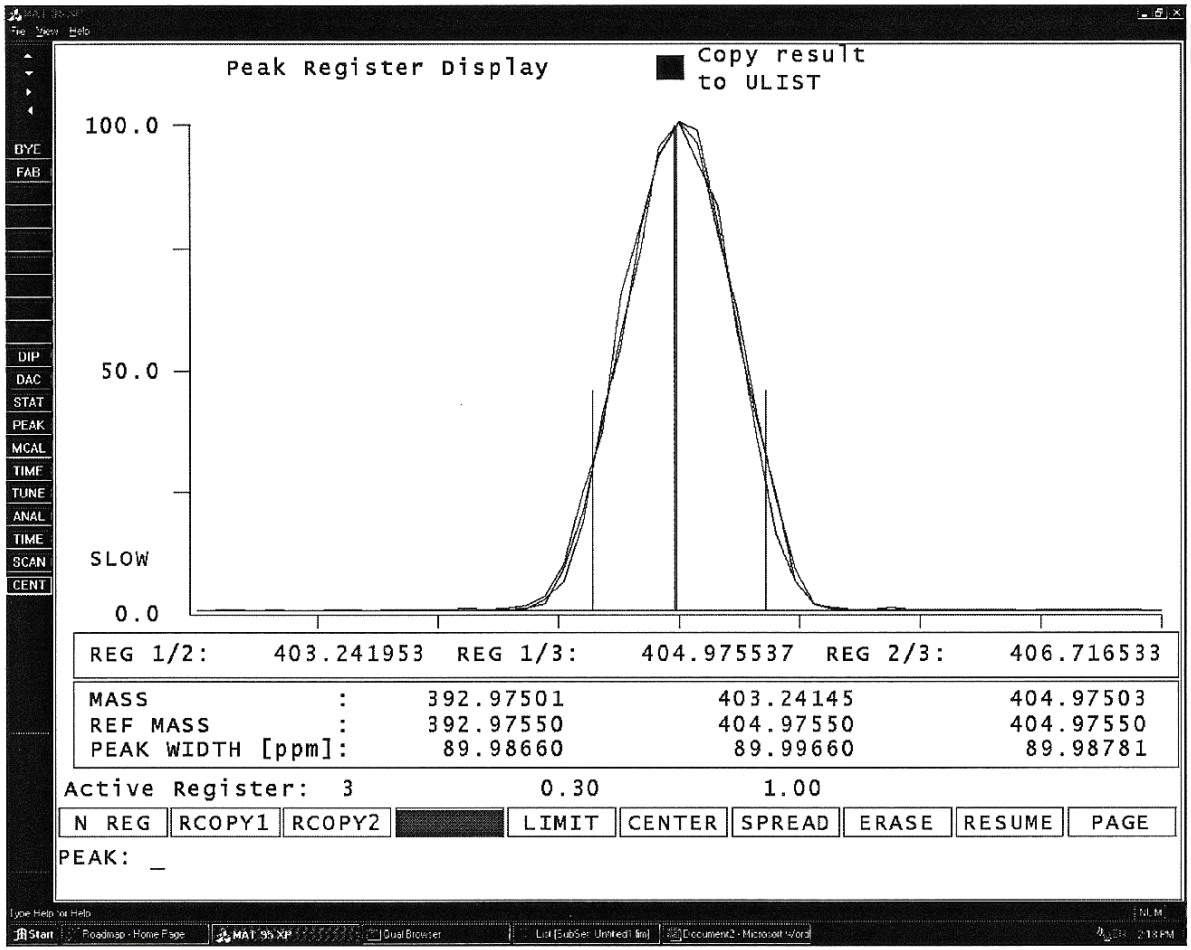

ppm
Integral
ppm
1.765
4.040
1.114
1.001
0.929
3.000
11.004
10.564
7.67665
7.65724
7.39831
7.38421
7.36378
7.26000
7.15723
6.44002
3.53715
1.75516
1.72654
1.68443
1.24695
1.13848
N-Me
Cy2P
L2
Current Data Parameters
NAME CPindole
EXPNO 34
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070516
Time 20.10
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 32
DS 0
SWH 4629.629 Hz
FIDRES 0.141285 Hz
AQ 3.5389941 sec
RG 35.9
DW 108.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 ======
NUC1 1H
P1 9.50 usec
PL1 -6.00 dB
SFO1 400.1319924 MHz
F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 19.63 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50931 ppm/cm
HZCM 203.79143 Hz/cm



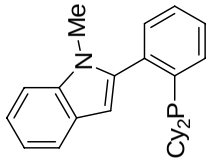

```
Current Data Parameters
NAME                  CCPindole
EXPNO                        21
PROCNO                        1

F2 - Acquisition Parameters
Date_               20070515
Time                      20.14
INSTRUM                 dpx400
PROBHD     5 mm GNP 1H
PULPROG                 zgdc30
TD                      131072
SOLVENT                 CD2Cl2
NS                          128
DS                            0
SWH                 25125.629 Hz
FIDRES                0.191693 Hz
AQ                  2.6083827 sec
RG                      9195.2
DW                     19.900 usec
DE                      4.50 usec
TE                     300.0 K
D1                  3.00000000 sec
d11                 0.03000000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                        5.80 usec
PL1                      -6.00 dB
SFO1              100.6231263 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                   71.00 usec
PL2                     120.00 dB
PL12                     17.00 dB
SFO2              400.1326008 MHz

F2 - Processing parameters
SI                         65536
SF                100.6127828 MHz
WDW                           EM
SSB                            0
LB                      1.00 Hz
GB                             0
PC                      1.00

1D NMR plot parameters
CX                      19.50 cm
F1P                   200.000 ppm
F1                  20122.56 Hz
F2P                    -0.000 ppm
F2                     -0.00 Hz
PPMCM                10.25641 ppm/cm
HZCM                 1031.92603 Hz/cm
```


File :C:\MSDCHEM\1\DATA\CMSO\C-PPHOS\Snapshot\1-methyl2-2cy2phenyl
.D
Operator :
Instrument : 5973N
Acquired : 16 May 2007 16:45 using AcqMethod METHOD2.M
Sample Name:
Misc Info :
Scan 2731 (17.708 min): 1-methyl2-2cy2phenyl.D\data.ms
Abundance
m/z-->
N-Me
Cy2P
L2

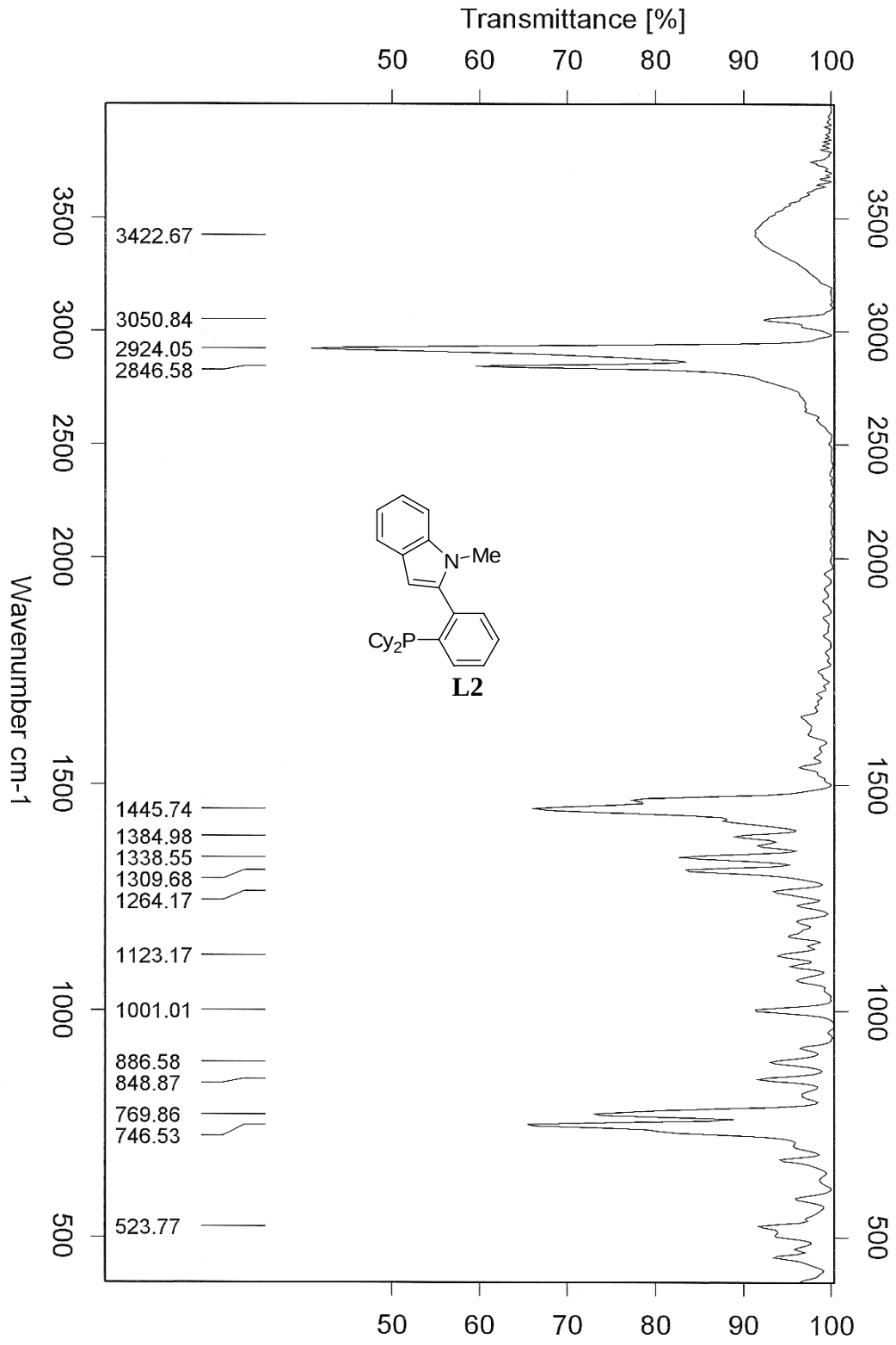
Accurate Mass Measurement:

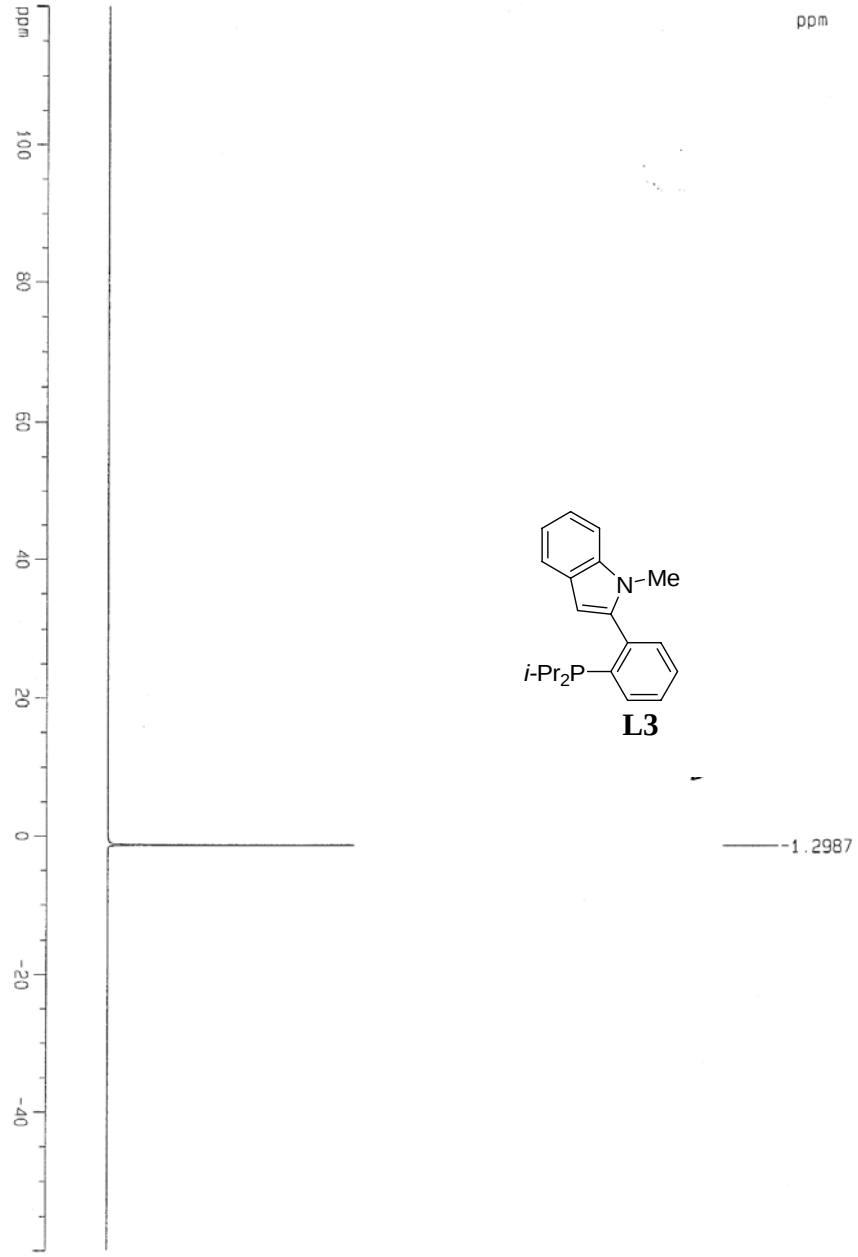


Molecular formula: $C_{27}H_{34}N_1P_1$
$[M]^+$ (theoretical) = 403.2423



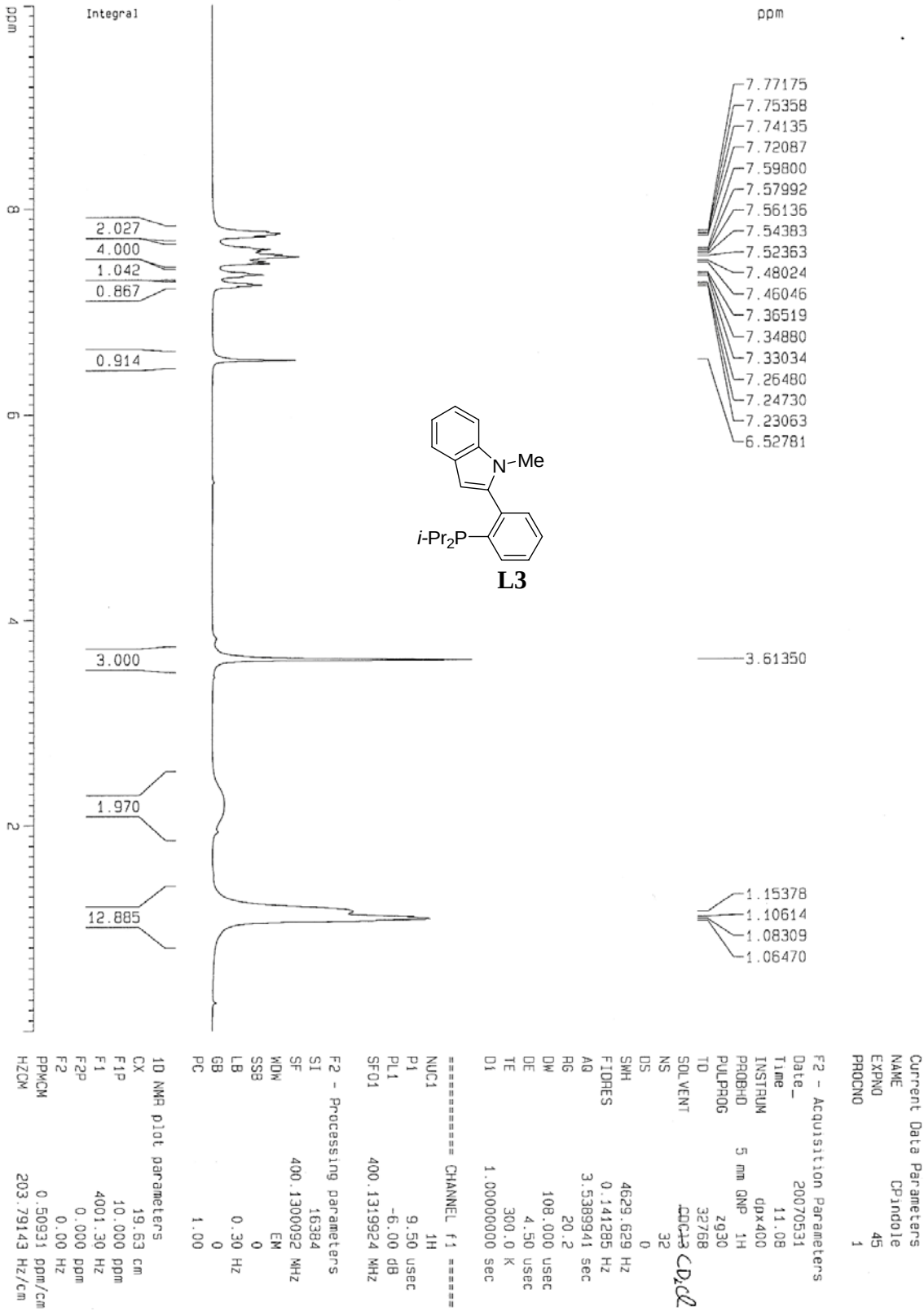
L2

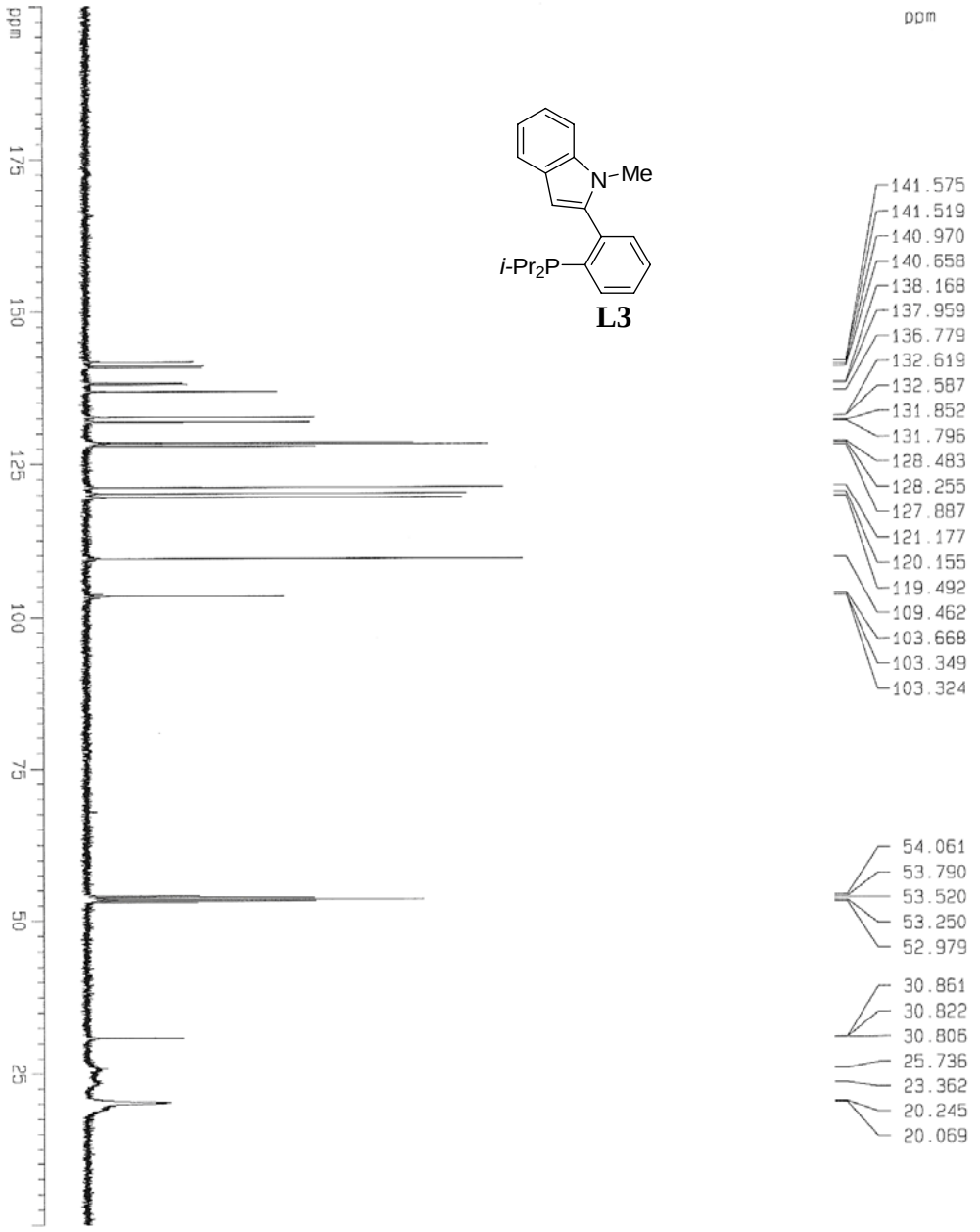

Transmittance [%]
50
60
70
80
90
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3422.67
3050.84
2924.05
2846.58
1445.74
1384.98
1338.55
1309.68
1264.17
1123.17
1001.01
886.58
848.87
769.86
746.53
523.77
N-Me
Cy2P
L2



L3

```
Current Data Parameters
NAME                  PCPindole
EXPNO                        42
PROCNO                        1

F2 - Acquisition Parameters
Date_              20070531
Time                   11.06
INSTRUM                dqx400
PROBHD     5 mm QNP  1H
PULPROG                zgdc30
TD                      65536
SOLVENT                CD2Cl2
NS                          32
DS                           0
SWH                49661.801 Hz
FIDRES             0.742520 Hz
AQ                 0.6734324 sec
RG                        6502
DW                      10.075 usec
DE                       4.50 usec
TE                      300.0 K
D1                 1.00000000 sec
d11                0.03000000 sec

========== CHANNEL f1 ========
NUC1                       31P
P1                        7.20 usec
PL1                      -5.00 dB
SFO1             161.9851115 MHz

========== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                   71.00 usec
PL2                     120.00 dB
PL12                     17.00 dB
SFO2             400.1324009 MHz

F2 - Processing parameters
SI                         32768
SF               161.9755931 MHz
WDW                           EM
SSB                            0
LB                      3.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                         20.00 cm
F1P                      120.000 ppm
F1                     19437.07 Hz
F2P                      -60.000 ppm
F2                     -9718.54 Hz
PPMCM                  9.00000 ppm/cm
HZCM                 1457.78027 Hz/cm
```

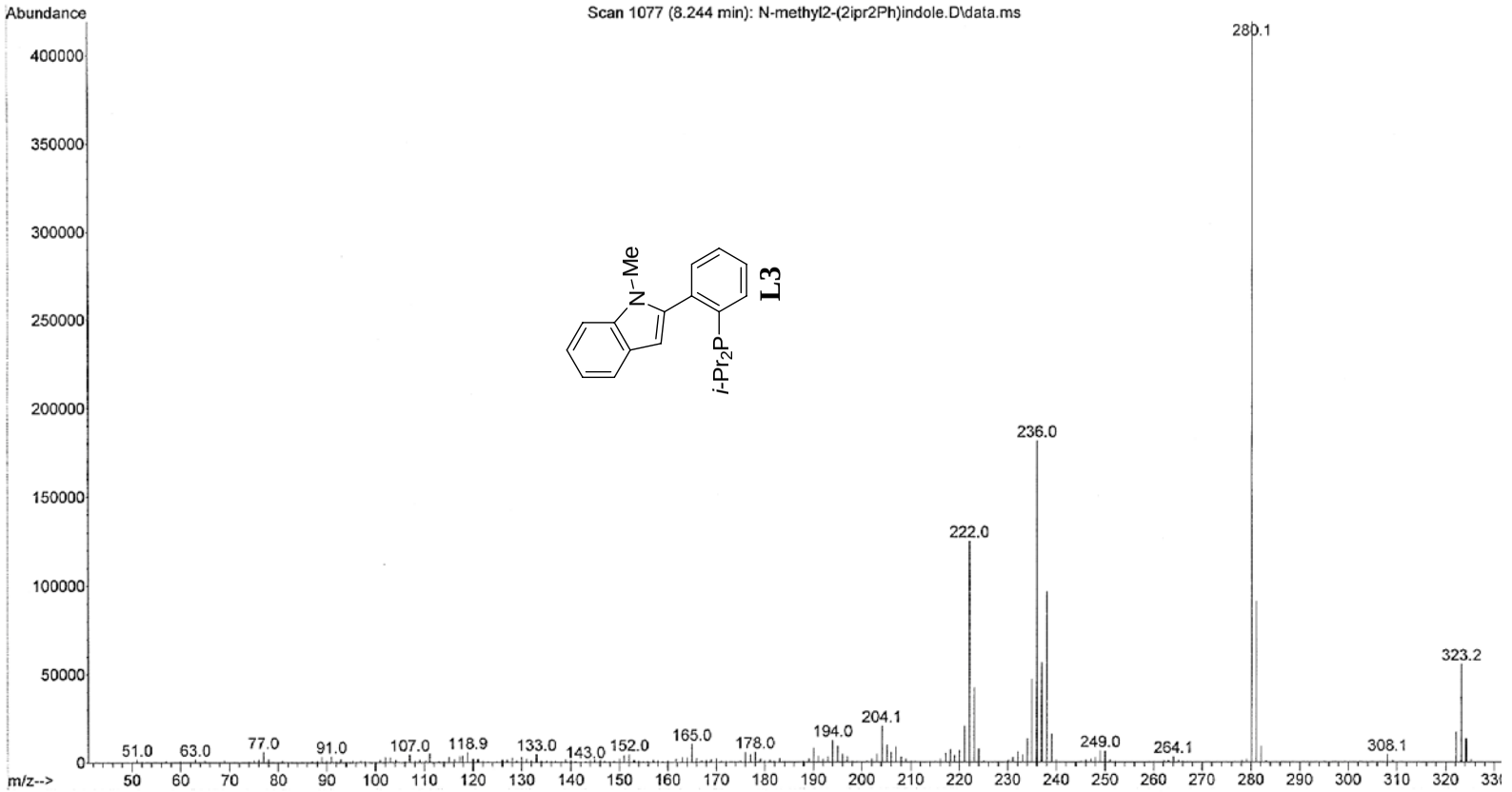

ppm
Integral
2.027
4.000
1.042
0.867
0.914
3.000
1.970
12.885
7.77175
7.75358
7.74135
7.72087
7.59800
7.57992
7.56136
7.54383
7.52363
7.48024
7.46046
7.36519
7.34880
7.33034
7.26480
7.24730
7.23063
6.52781
3.61350
1.15378
1.10614
1.08309
1.06470
N-Me
i-Pr2P
L3
Current Data Parameters
NAME CPindole
EXPNO 45
PROCNO 1
F2 - Acquisition Parameters
Date_ 20070531
Time 11.08
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3 CD2Cl2
NS 32
DS 0
SWH 4629.629 Hz
FIDRES 0.141285 Hz
AQ 3.5389941 sec
RG 20.2
DW 108.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 ======
NUC1 1H
P1 9.50 usec
PL1 -6.00 dB
SFO1 400.1319924 MHz
F2 - Processing parameters
SI 16384
SF 400.1300092 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 19.63 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.50931 ppm/cm
HZCM 203.79143 Hz/cm



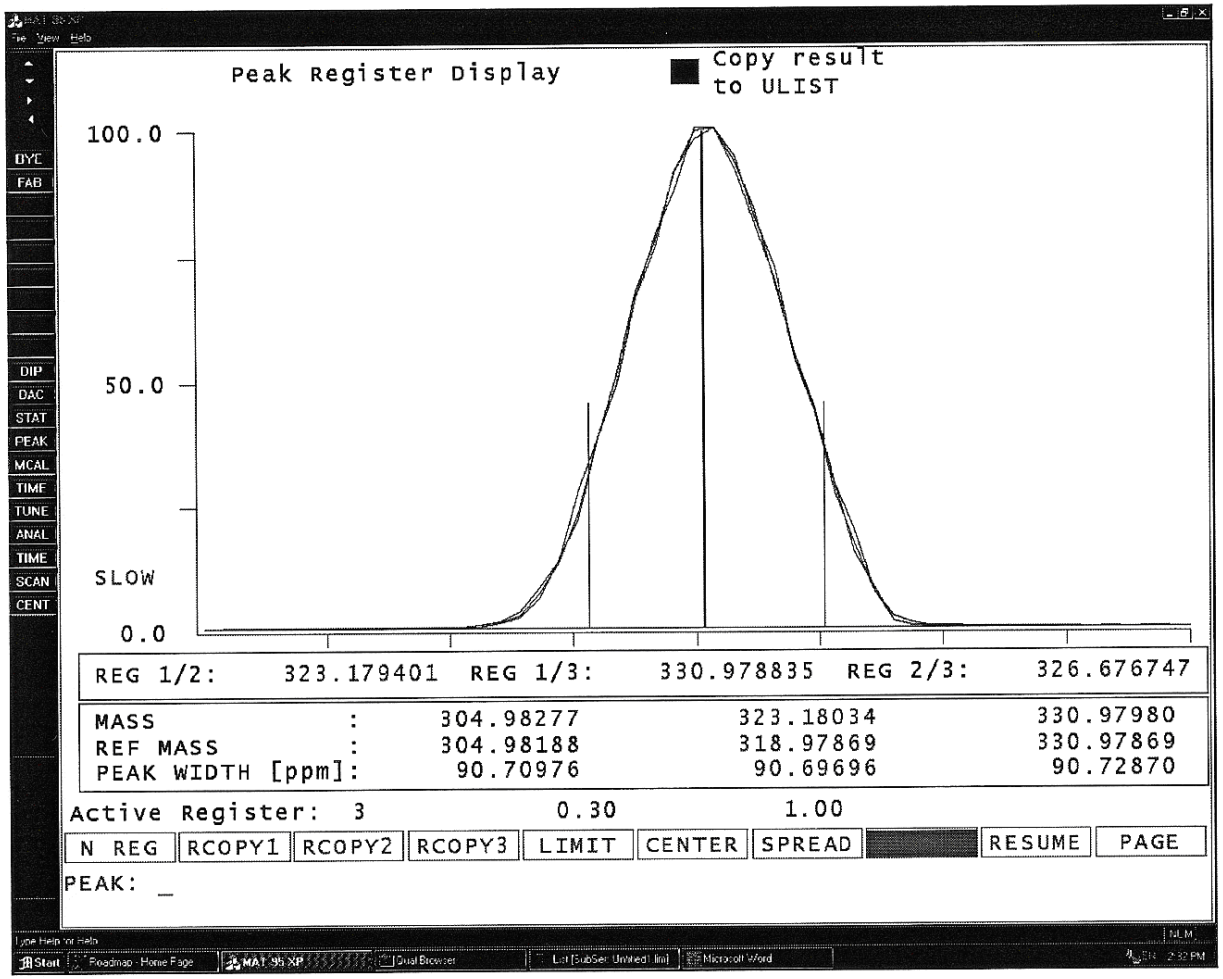
L3

```
Current Data Parameters
NAME                  CCPindole
EXPNO                        26
PROCNO                        1

F2 - Acquisition Parameters
Date_               20070531
Time                    11.20
INSTRUM                 dpx400
PROBHD     5 mm BNP 1H
PULPROG                 zgdc30
TD                      131072
SOLVENT                 CDCl3
NS                         128
DS                           0
SWH                 25125.629 Hz
FIDRES                  0.191693 Hz
AQ                  2.6083827 sec
RG                       9195.2
DW                      19.900 usec
DE                       4.50 usec
TE                      300.0 K
D1                  3.00000000 sec
d11                 0.03000000 sec

========== CHANNEL f1 ========
NUC1                       13C
P1                        5.80 usec
PL1                      -6.00 dB
SFO1                100.6231263 MHz

========== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    71.00 usec
PL2                      120.00 dB
PL12                      17.00 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                         65536
SF                  100.6127679 MHz
WDW                            EM
SSB                             0
LB                       1.00 Hz
GB                              0
PC                       1.00

1D NMR plot parameters
CX                       19.50 cm
F1P                    200.000 ppm
F1                   20122.55 Hz
F2P                     -0.000 ppm
F2                       -0.00 Hz
PPMCM                  10.25641 ppm/cm
HZCM                1031.92590 Hz/cm
```

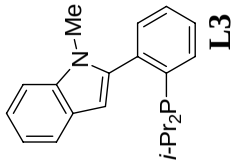
```
File       :C:\msdchem\1\DATA\cmso\C-Pphos\N-methyl2-(2ipr2Ph)indole.D
Operator   :
Acquired   : 30 May 2007  20:57     using AcqMethod METHOD2.M
Instrument :   5973N
Sample Name:
Misc Info  :
Vial Number: 8
```



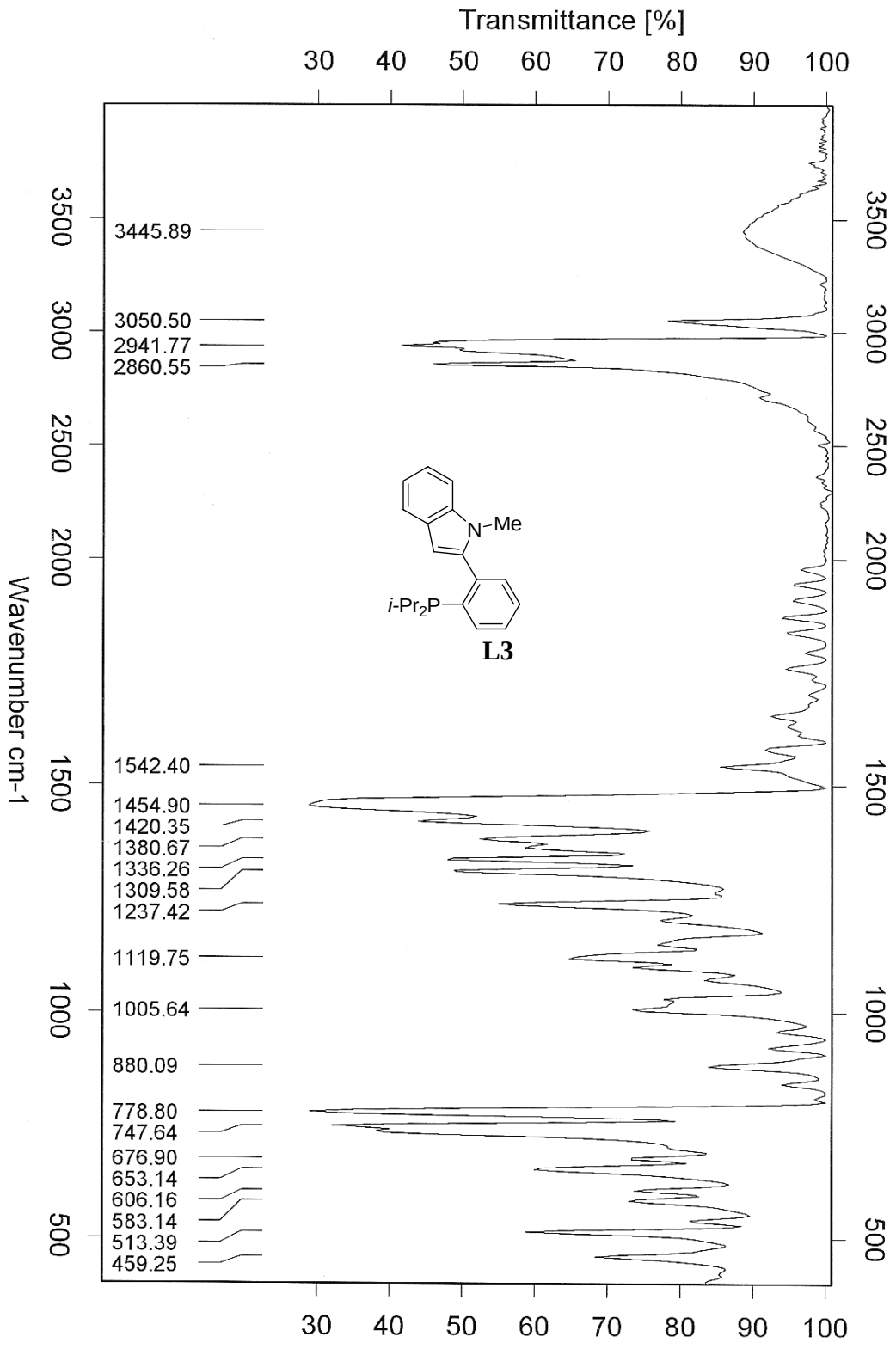
Accurate Mass Measurement:

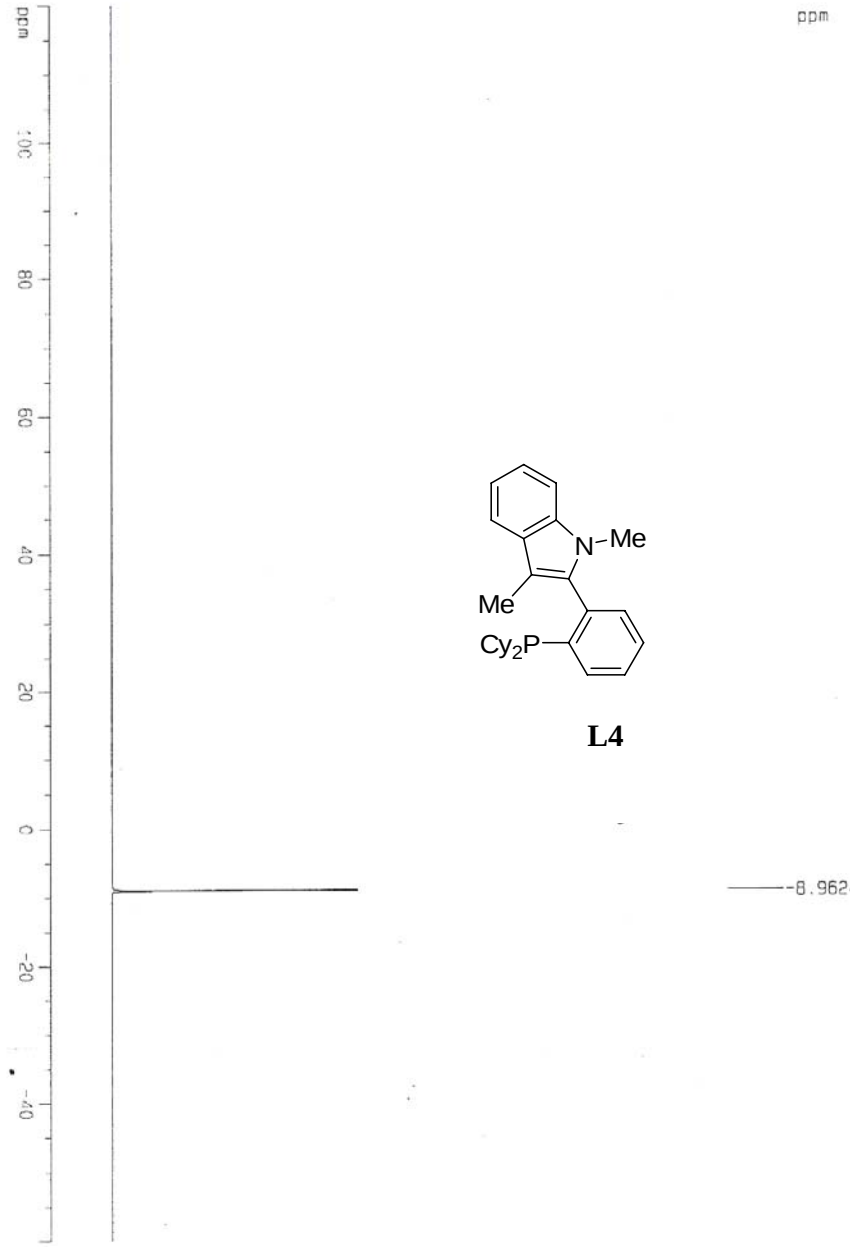


Molecular formula:
$C_{21}H_{26}N_1P_1$
$[M]^+$ (theoretical)
= 323.1797



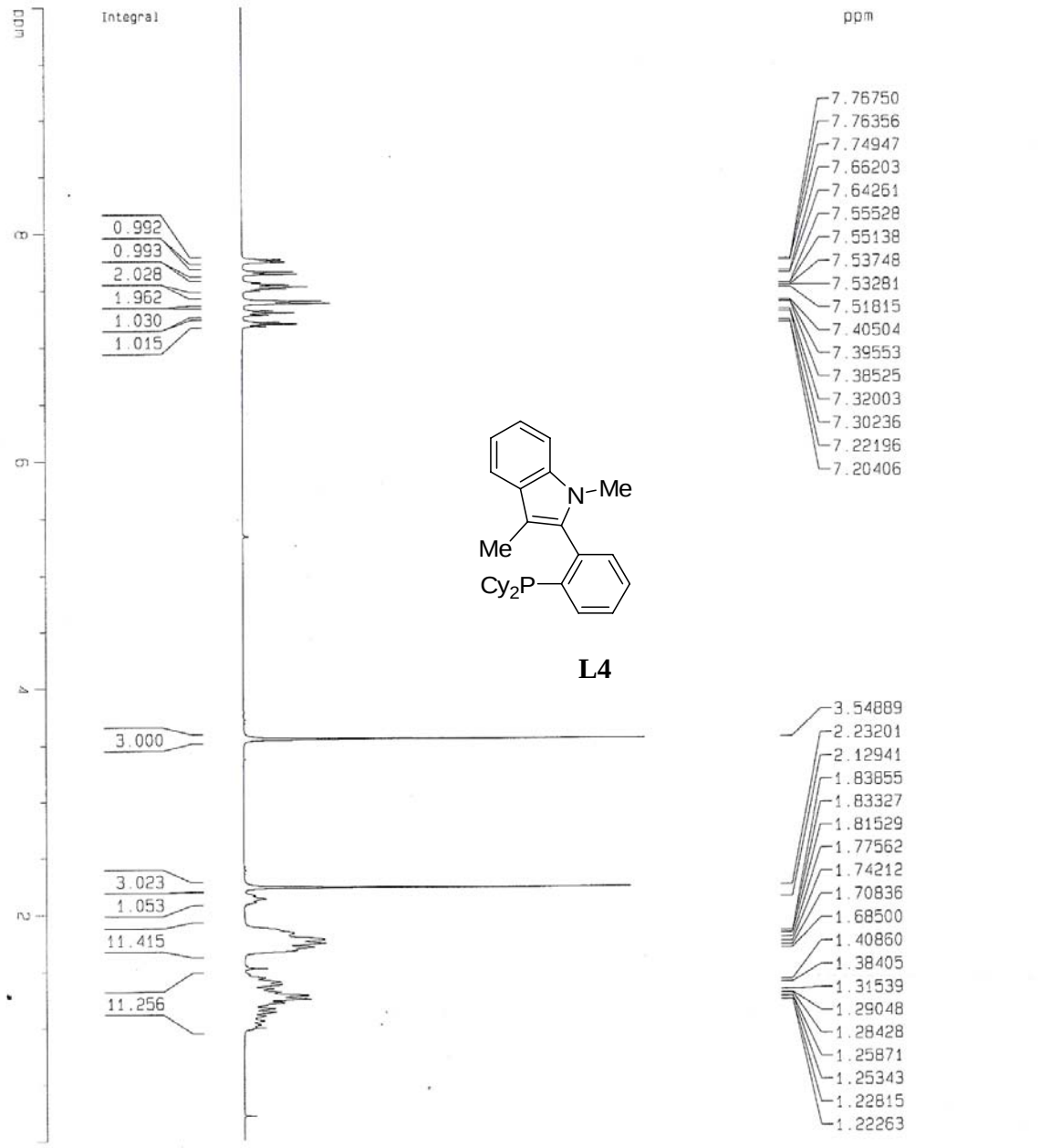
L3

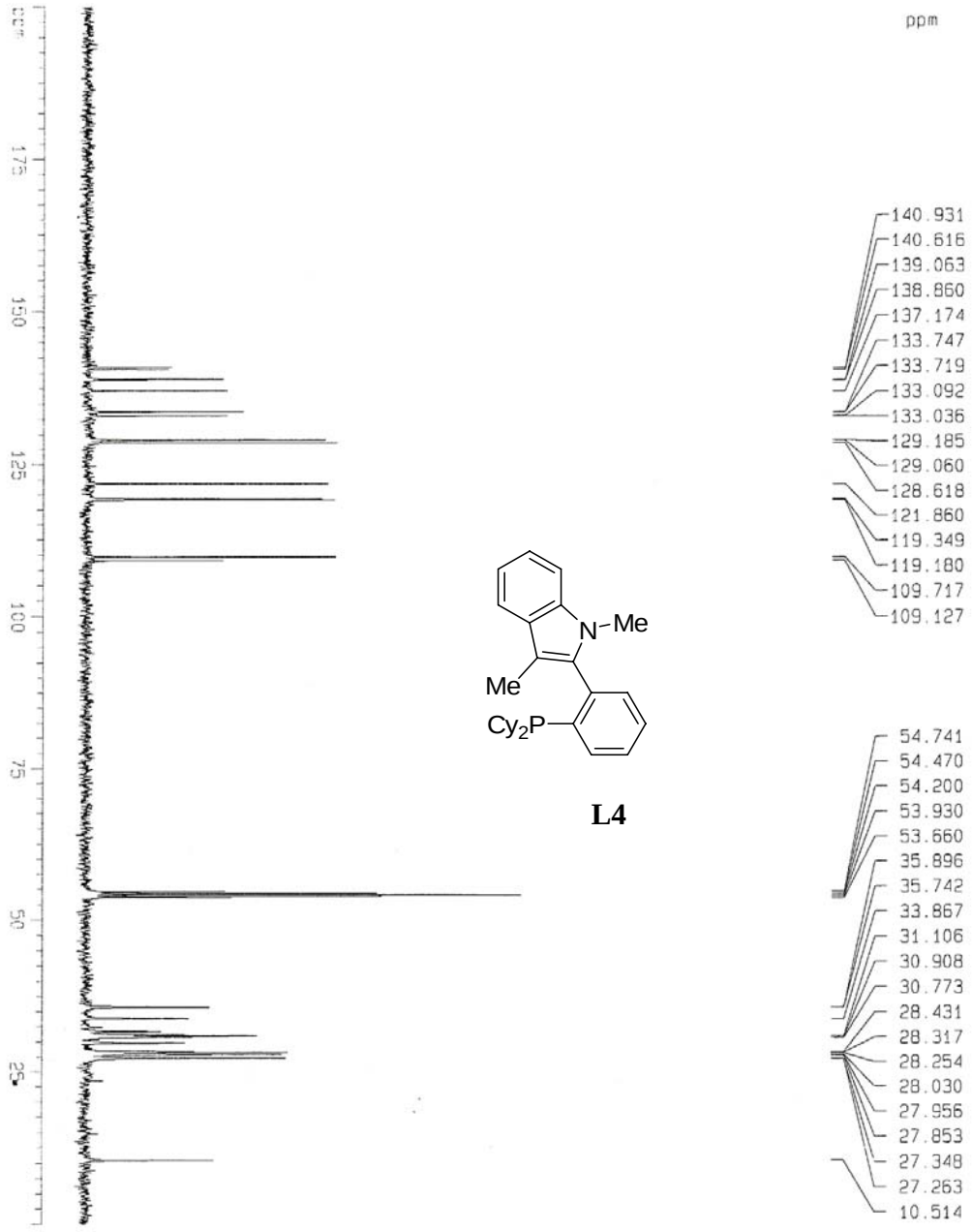

Transmittance [%]
30 40 50 60 70 80 90 100
Wavenumber cm-1
3500 3000 2500 2000 1500 1000 500
3445.89
3050.50
2941.77
2860.55
1542.40
1454.90
1420.35
1380.67
1336.26
1309.58
1237.42
1119.75
1005.64
880.09
778.80
747.64
676.90
653.14
606.16
583.14
513.39
459.25
N-Me
i-Pr2P
L3



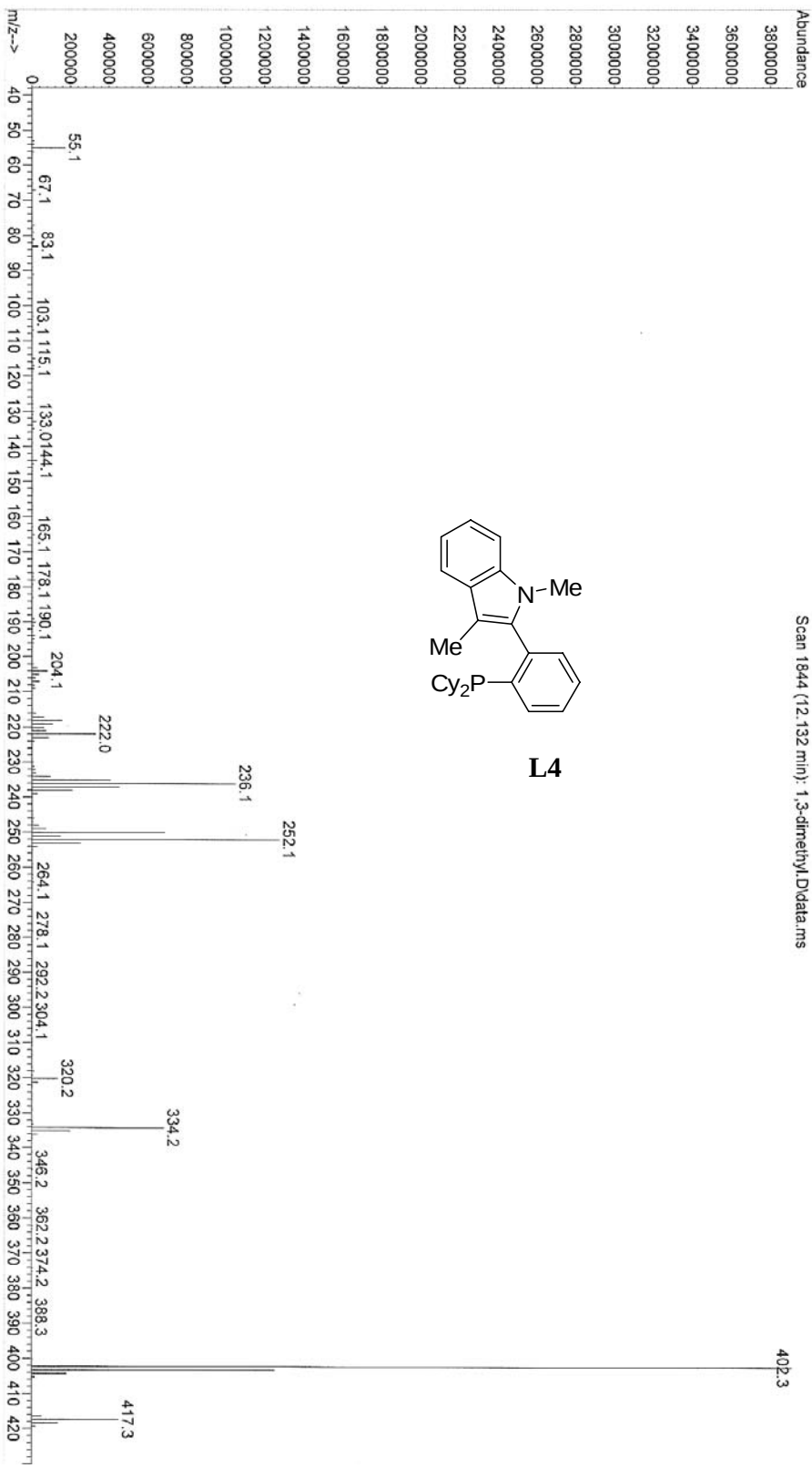
L4


```
Current Data Parameters
NAME                  PCPrindole
EXPNO                         80
PROCNO                         1

F2 - Acquisition Parameters
Date_              20071210
Time                   15.16
INSTRUM          qdx400
PROBHD     5 mm QNP 1H
PULPROG          zgdc30
TD                  65536
SOLVENT           CD2Cl2
NS                    32
DS                     0
SWH              4866.801 Hz
FIDRES           0.742520 Hz
AQ               0.6734324 sec
RG                  6502
DW                 10.275 usec
DE                  4.50 usec
TE                300.0 K
D1           1.00000000 sec
d11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                  31P
P1                   7.20 usec
PL1                 -5.00 dB
SFO1         161.9851115 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2               71.00 usec
PL2                120.00 dB
PL12                17.00 dB
SFO2         400.1324008 MHz

F2 - Processing parameters
SI                   32768
SF           161.9755831 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                   20.00 cm
F1P                 120.000 ppm
F1                19437.07 Hz
F2P                -60.000 ppm
F2                -9718.54 Hz
PPMCM              9.00000 ppm/cm
HZCM             1457.78027 Hz/cm
```


ppm
Integral
7.76750
7.76356
7.74947
7.66203
7.64261
7.55528
7.55138
7.53748
7.53281
7.51815
7.40504
7.39553
7.38525
7.32003
7.30236
7.22196
7.20406
0.992
0.993
2.028
1.962
1.030
1.015
3.000
3.023
1.053
11.415
11.256
N-Me
Me
Cy2P
L4
3.54889
2.23201
2.12941
1.83855
1.83327
1.81529
1.77562
1.74212
1.70836
1.68500
1.40860
1.38405
1.31539
1.29048
1.28428
1.25871
1.25343
1.22815
1.22263
Current Data Parameters
NAME phosphinenido
EXPNO 79
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071210
Time 15.18
INSTRUM dpx400
PROBHD 5 mm BNP 1H
PULPROG z930
TD 32768
SOLVENT CD2Cl2
NS 32
DS 0
SWH 4629.629 Hz
FIDRES 0.141285 Hz
AQ 3.5389941 sec
RG 35.9
DW 108.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 -6.00 dB
SFO1 400.1319924 MHz
F2 - Processing parameters
SI 16384
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0.70 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 19.63 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 0.50930 ppm/cm
HZCM 203.78769 Hz/cm

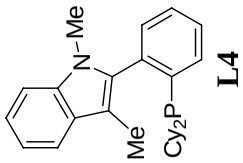



```
Current Data Parameters
NAME                  CCPindole
EXPNO                        34
PROCNO                        1

F2 - Acquisition Parameters
Date_               20071210
Time                    15.21
INSTRUM                 dpx400
PROBHD     5 mm QNP  1H
PULPROG                 zgdc30
TD                      131072
SOLVENT                 CD2Cl2
NS                         116
DS                           0
SWH                 25125.629 Hz
FIDRES               0.191693 Hz
AQ                   2.6083827 sec
RG                      5792.6
DW                      19.900 usec
DE                       4.50 usec
TE                      300.0 K
D1                   3.00000000 sec
d11                  0.03000000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                        5.80 usec
PL1                      -6.00 dB
SF01               100.6231263 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    71.00 usec
PL2                     120.00 dB
PL12                     17.00 dB
SF02               400.1320008 MHz

F2 - Processing parameters
SI                        65536
SF                 100.6126960 MHz
WDW                          EM
SSB                           0
LB                         3.00 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                        19.50 cm
F1P                      200.000 ppm
F1                     20122.54 Hz
F2P                     -0.000 ppm
F2                     -0.00 Hz
PPMCM                  10.25641 ppm/cm
HZCM                1031.92505 Hz/cm
```


File :C:\msdchem\1\DATA\cmso\C-Pphos\1,3-dimethyl.D
Operator :
Acquired : 8 Dec 2007 11:20 using AcqMethod METHOD2.M
Instrument : 5973N
Sample Name:
Misc Info :
Vial Number: 8
Abundance
Scan 1844 (12.132 min): 1,3-dimethyl.D\data.ms
m/z-->

N-Me
Me
Cy2P

L4



L4

Elemental Composition Report

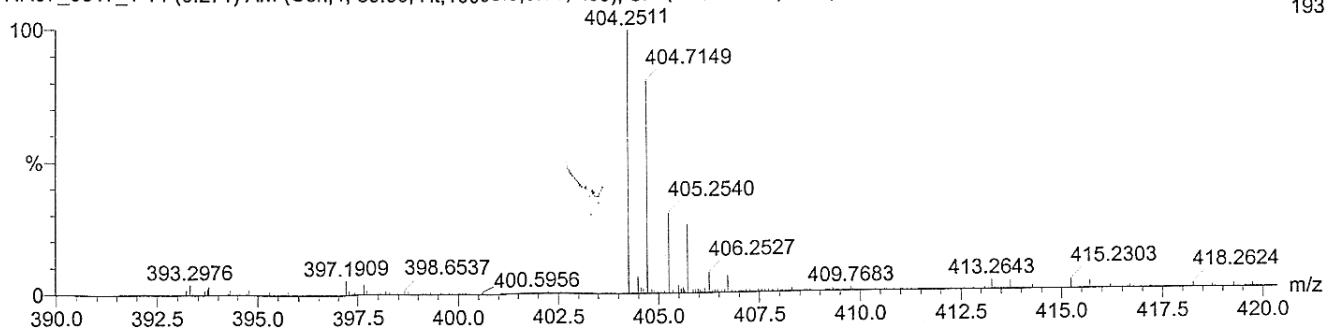
Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 60.0
Selected filters: None

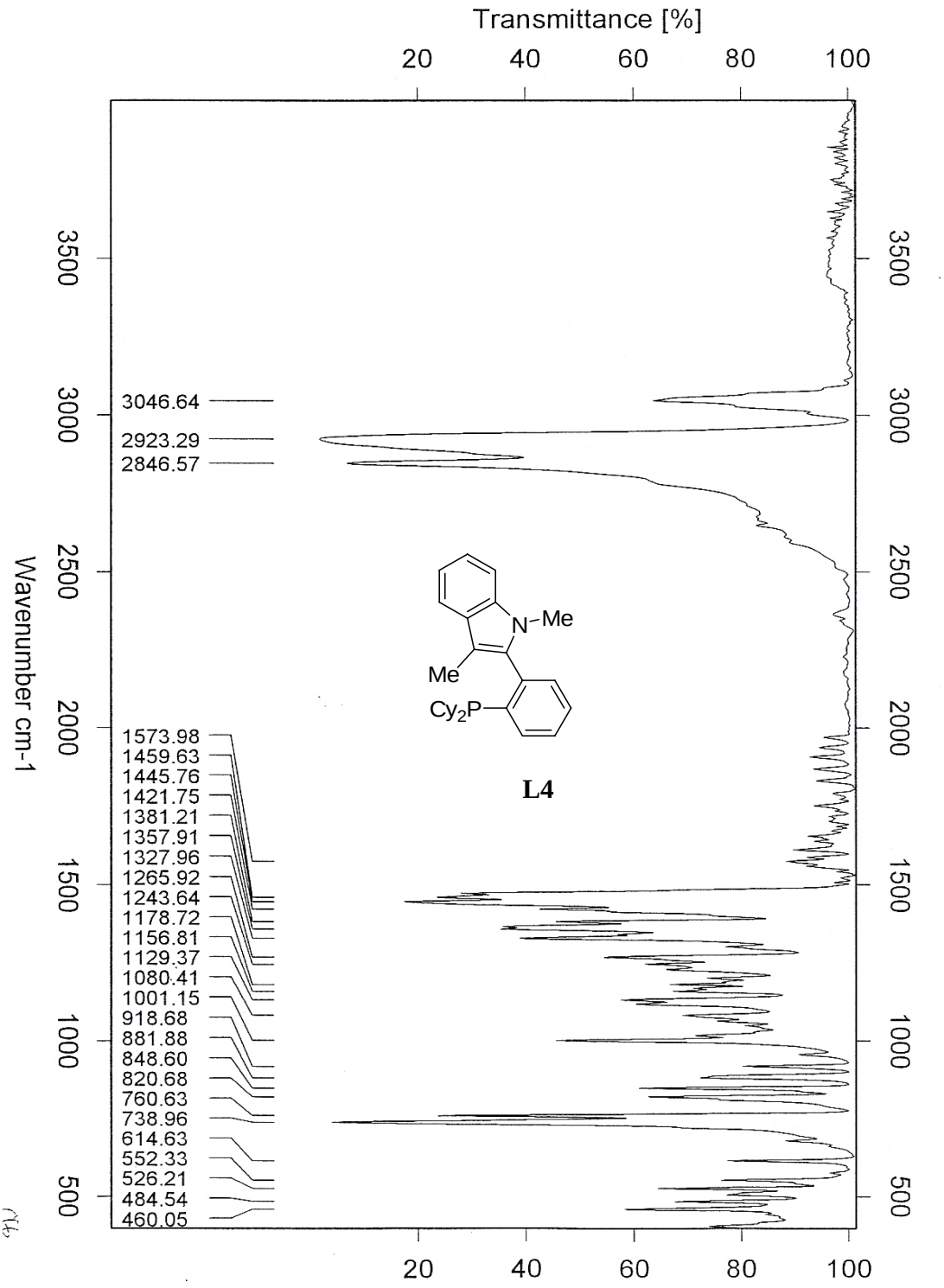
Monoisotopic Mass, Even Electron Ions
24 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-28 H: 0-1000 N: 0-1 O: 0-1 P: 0-1

So Chau Ming, N-methyl 2-phenylindole 3-cy2Phosphine
HR07_0517_1 14 (0.271) AM (Cen,4, 80.00, Ht,10000.0,0.00,1.00); Sm (SG, 2x3.00); Sb (15,10.00); Cm (13:19)

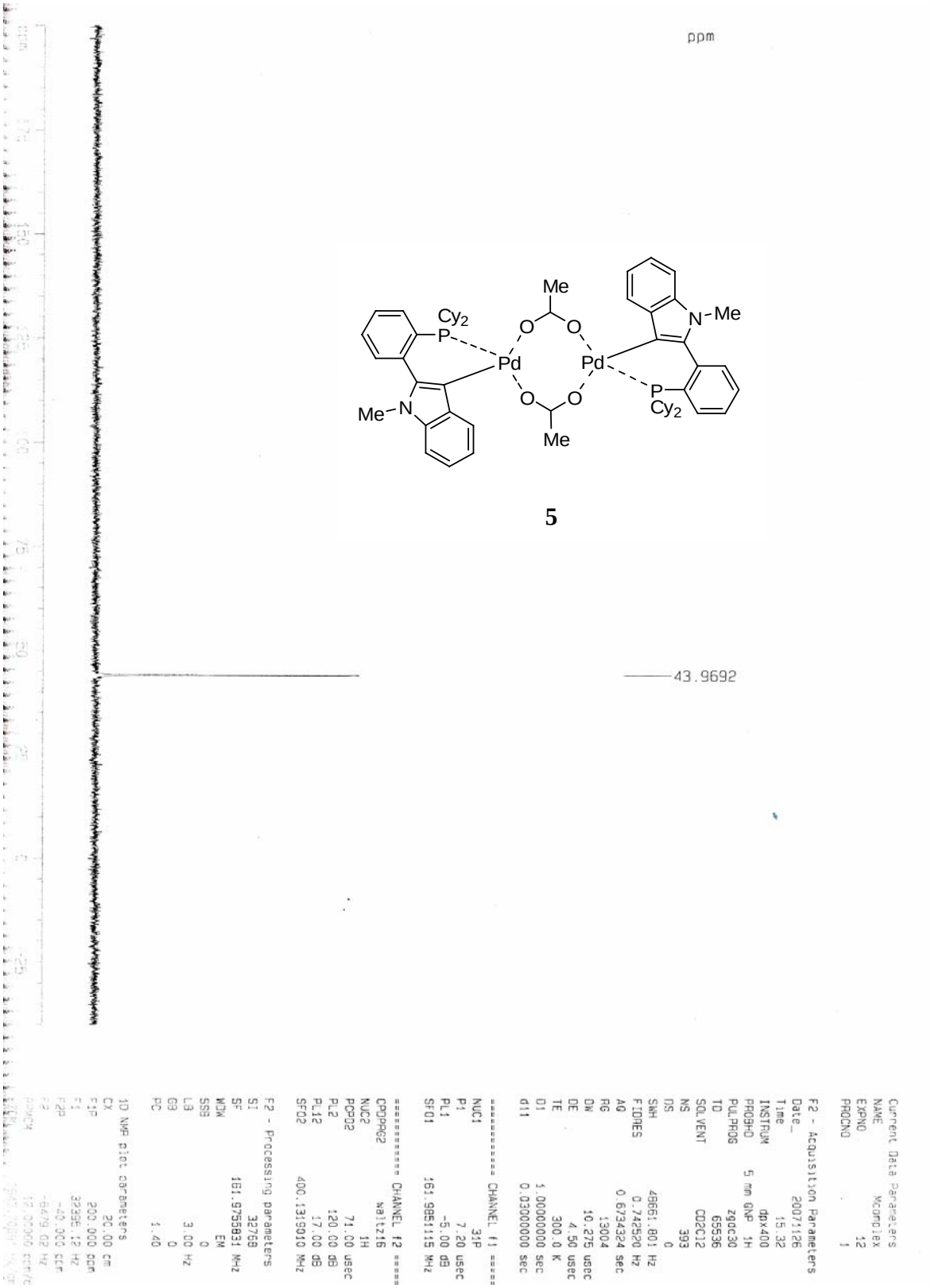
TOF MS ES+
193



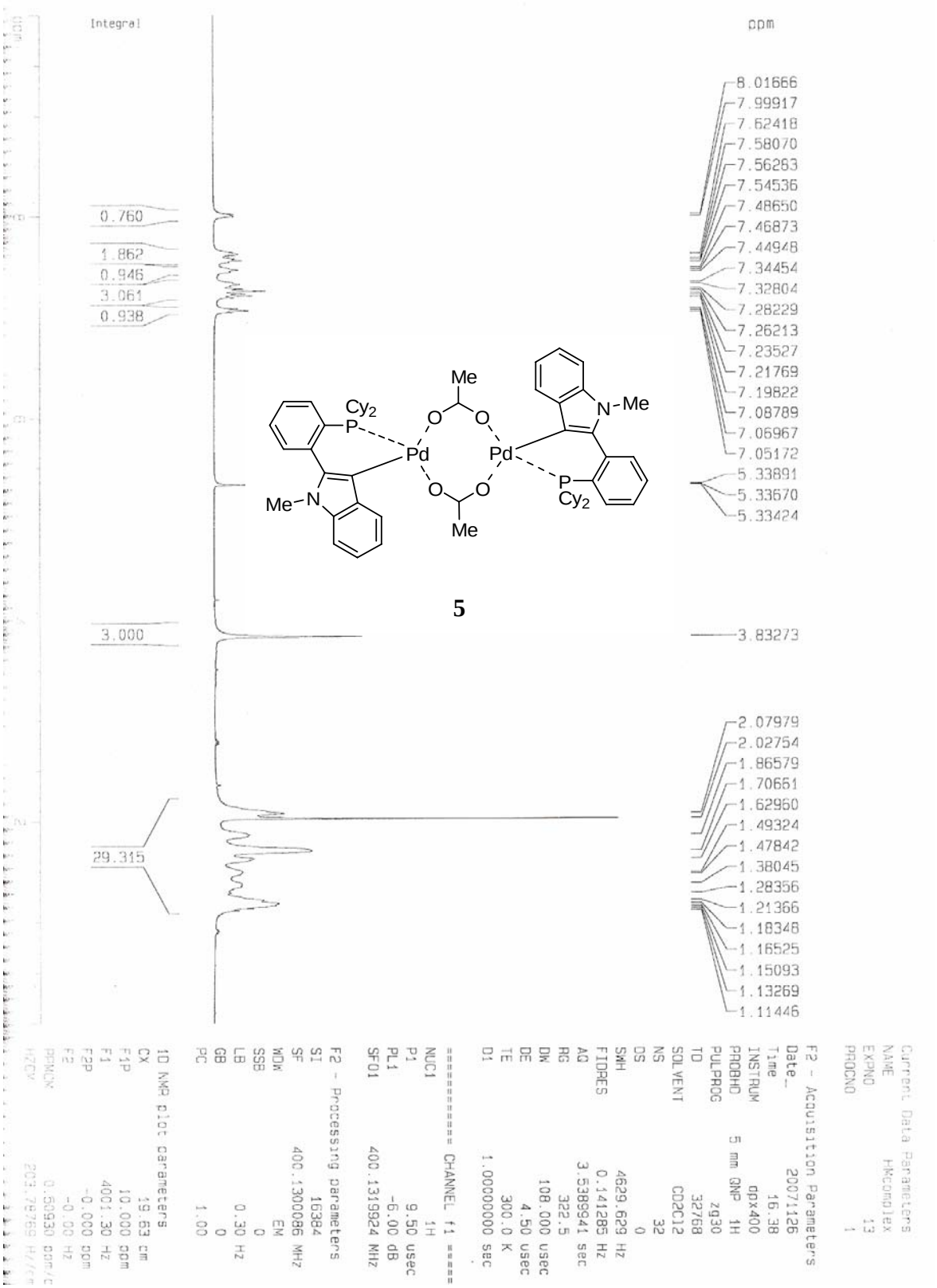
Minimum:				-1.5		
Maximum:		5.0	5.0	60.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
404.2511	404.2507	0.4	1.0	11.5	1.1	C27 H35 N P


Transmittance [%]
20
40
60
80
100
Wavenumber cm-1
3500
3000
2500
2000
1500
1000
500
3046.64
2923.29
2846.57
1573.98
1459.63
1445.76
1421.75
1381.21
1357.91
1327.96
1265.92
1243.64
1178.72
1156.81
1129.37
1080.41
1001.15
918.68
881.88
848.60
820.68
760.63
738.96
614.63
552.33
526.21
484.54
460.05
N-Me
Me
Cy2P

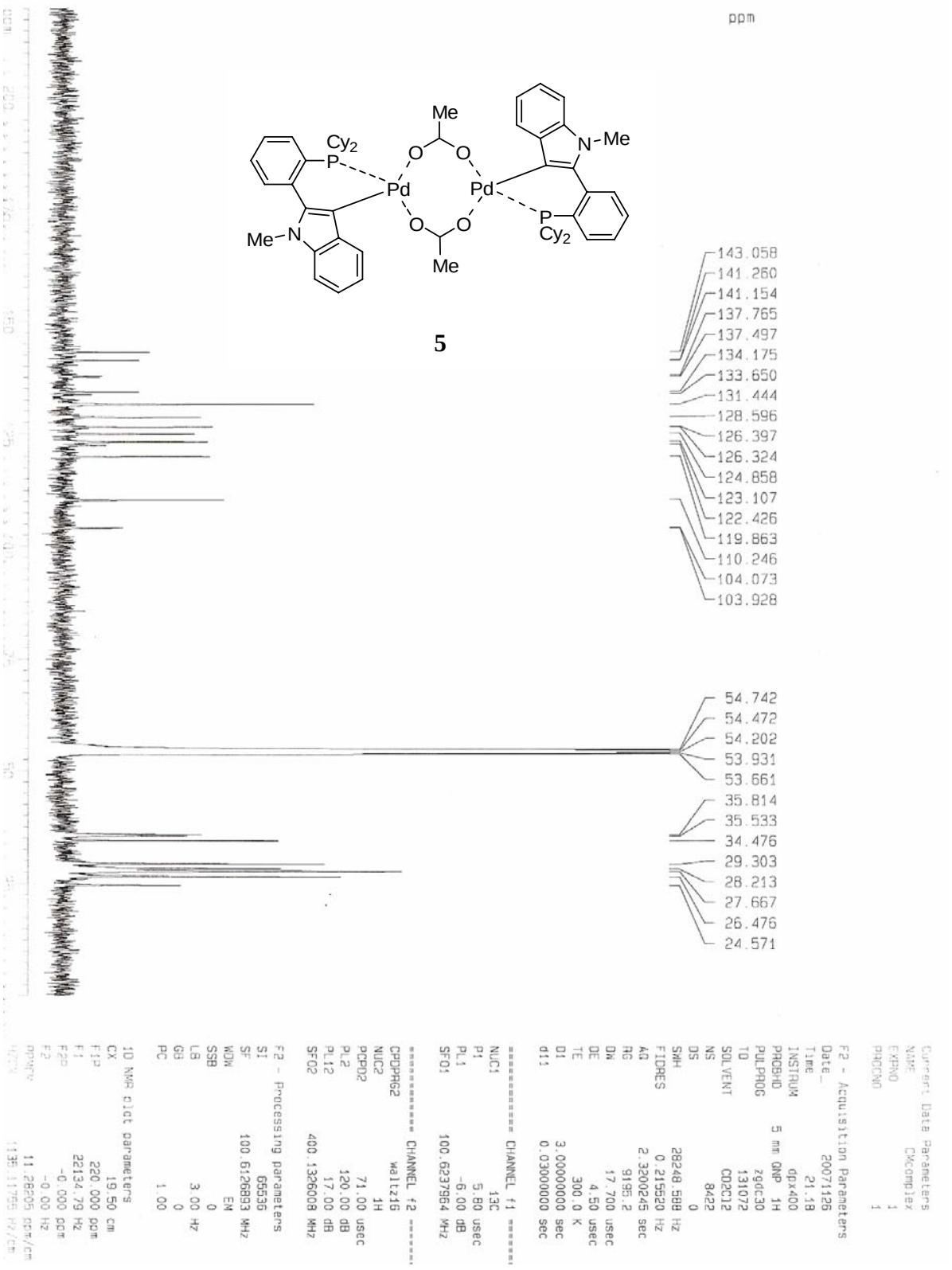
L4



5


Integral
ppm
8.01666
7.99917
7.62418
7.58070
7.56283
7.54536
7.48650
7.46873
7.44948
7.34454
7.32804
7.28229
7.26213
7.23527
7.21769
7.19822
7.08789
7.06967
7.05172
5.33891
5.33670
5.33424
0.760
1.862
0.946
3.061
0.938
3.000
3.83273
2.07979
2.02754
1.86579
1.70661
1.62960
1.49324
1.47842
1.38045
1.28356
1.21366
1.18348
1.16525
1.15093
1.13269
1.11446
29.315
Me
Cy2
P
Pd
O
N-Me
Me-N
Cy2
Me
Current Data Parameters
NAME HMcomplex
EXPNO 13
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071126
Time 16.38
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CD2Cl2
NS 32
DS 0
SWH 4629.629 Hz
FIDRES 0.141285 Hz
AQ 3.5389941 sec
RG 322.5
DW 108.000 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
========== CHANNEL f1 =====
NUC1 1H
P1 9.50 usec
PL1 -6.00 dB
SF01 400.1319924 MHz
F2 - Processing parameters
SI 16384
SF 400.1300086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 19.63 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 0.50930 ppm/c
HZCM 203.79769 Hz/cm

5


ppm
Me
Cy2
P
O
O
N-Me
Pd
Pd
Me-N
P
Cy2
O
O
Me
143.058
141.260
141.154
137.765
137.497
134.175
133.650
131.444
128.596
126.397
126.324
124.858
123.107
122.426
119.863
110.246
104.073
103.928
54.742
54.472
54.202
53.931
53.661
35.814
35.533
34.476
29.303
28.213
27.667
26.476
24.571
Current Data Parameters
NAME CMcomplex
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071126
Time 21.18
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zgdc30
TD 131072
SOLVENT CD2Cl2
NS 8422
DS 0
SWH 28248.588 Hz
FIDRES 0.215520 Hz
AQ 2.3200245 sec
RG 9195.2
DW 17.700 usec
DE 4.50 usec
TE 300.0 K
D1 3.00000000 sec
d11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 5.80 usec
PL1 -6.00 dB
SFO1 100.6237964 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 71.00 usec
PL2 120.00 dB
PL12 17.00 dB
SFO2 400.1326008 MHz
F2 - Processing parameters
SI 65536
SF 100.6126893 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 19.50 cm
F1P 220.000 ppm
F1 22134.79 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 11.28205 ppm/cm
HZCM 1135.11755 Hz/cm

5

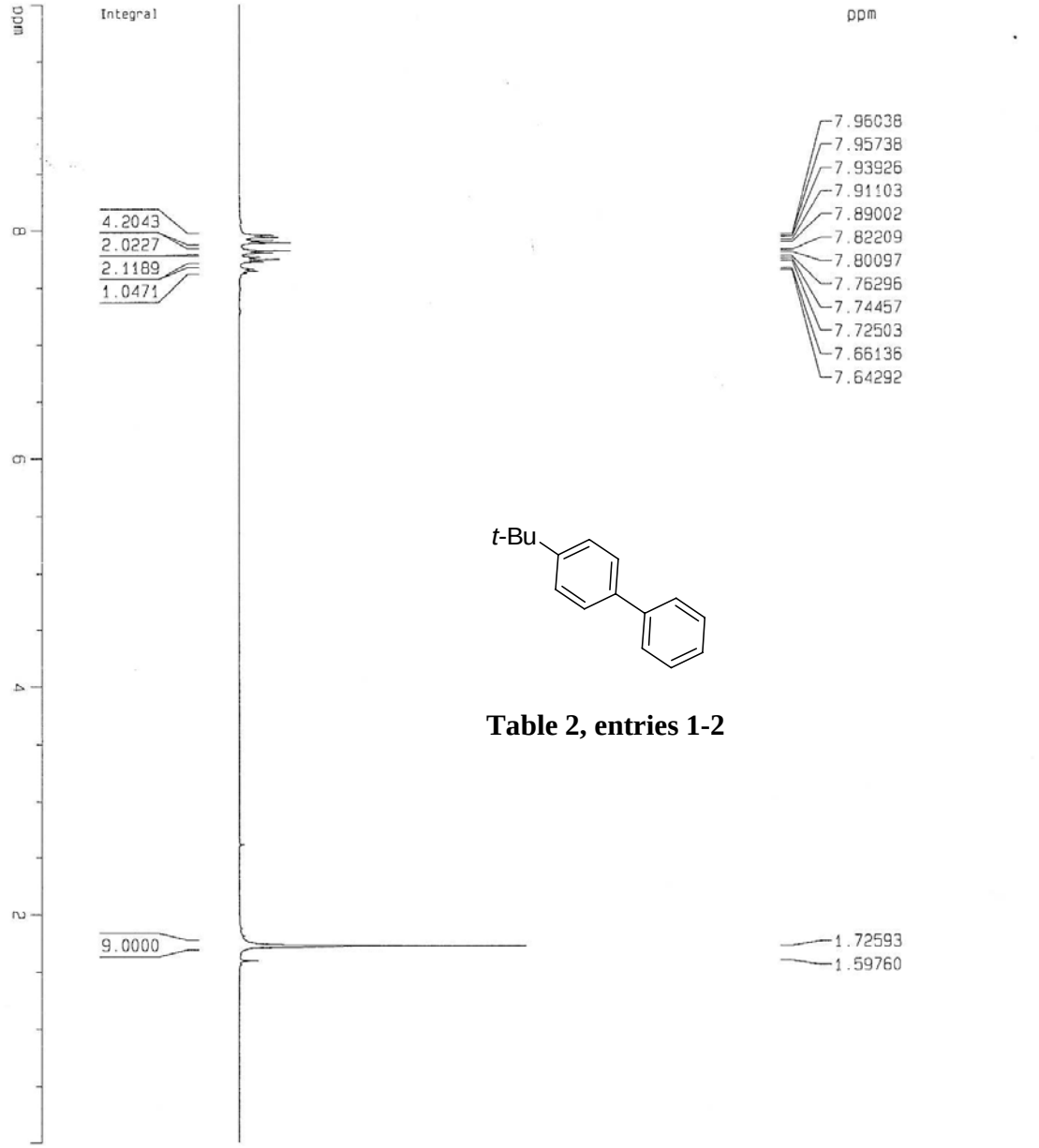


Table 2, entries 1-2

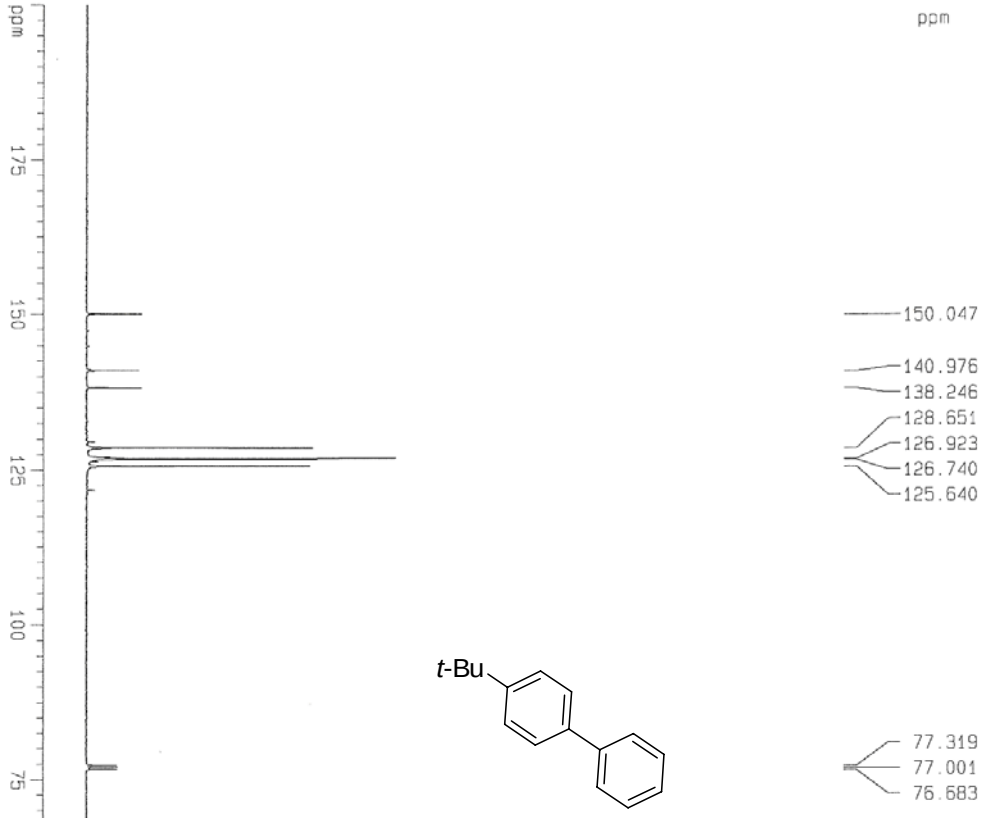
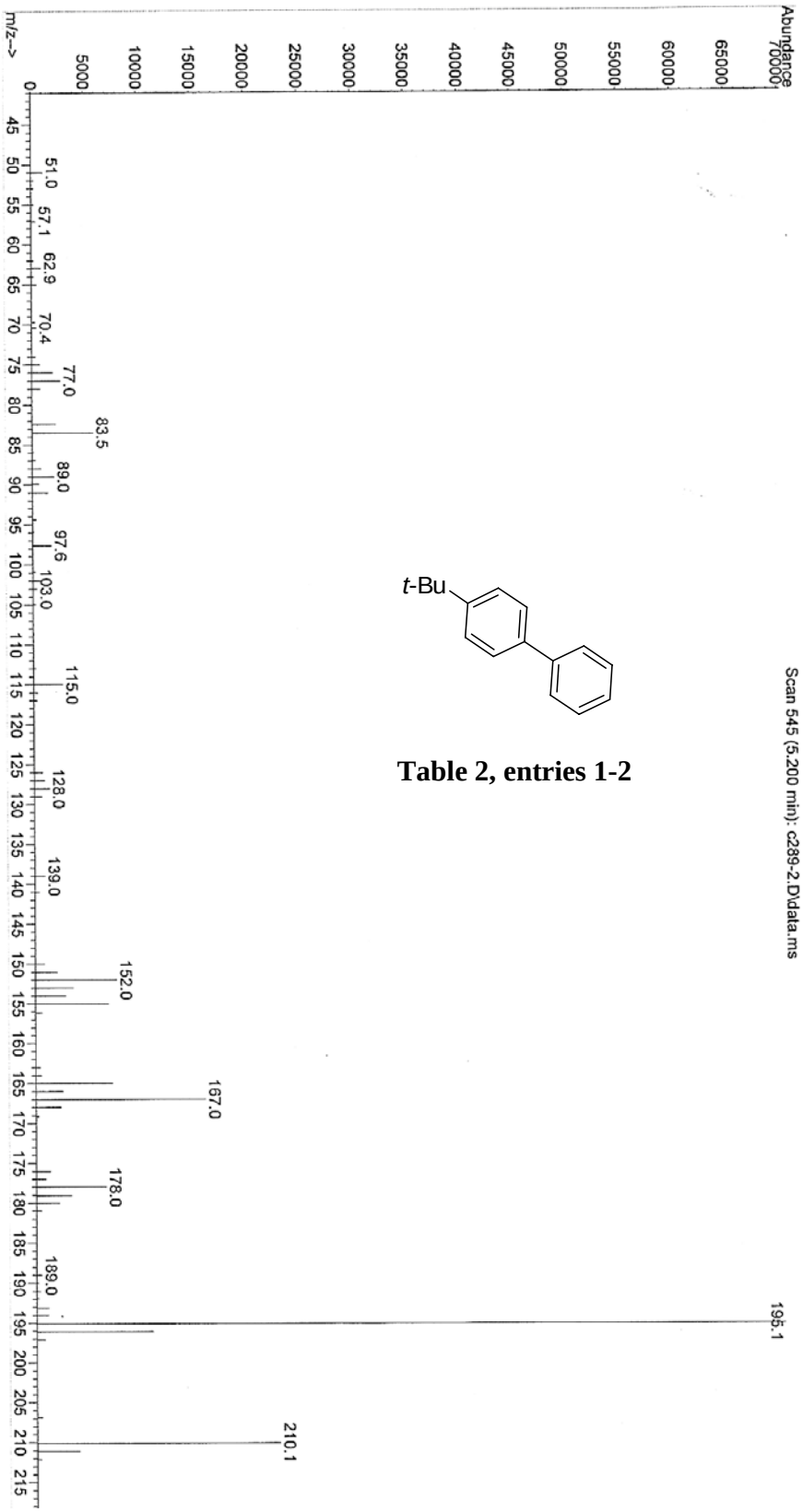


Table 2, entries 1-2



t-Bu

Table 2, entries 1-2

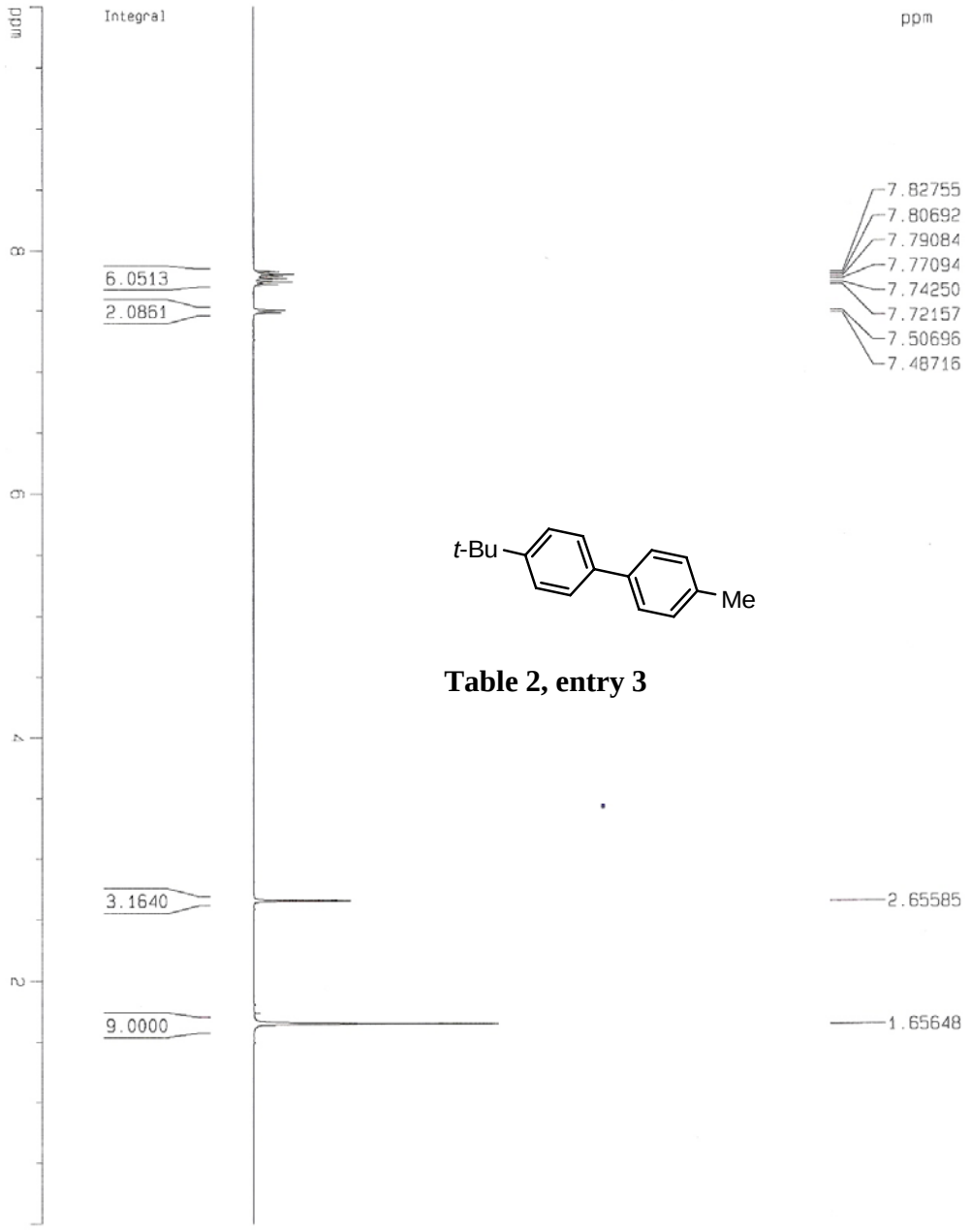


Table 2, entry 3

Current Data Parameters
NAME coupling
EXPNO 146
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071115
Time 21.10
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 4789.272 Hz
FIDRES 0.146157 Hz
AQ 3.4210291 sec
RG 35.9
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec

========== CHANNEL f1 ======
NUC1 1H
P1 9.50 usec
PL1 -6.00 dB
SFO1 400.1320007 MHz

F2 - Processing parameters
SI 16384
SF 400.1300089 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 19.63 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 0.50930 ppm/cm
HZCM 203.78769 Hz/cm

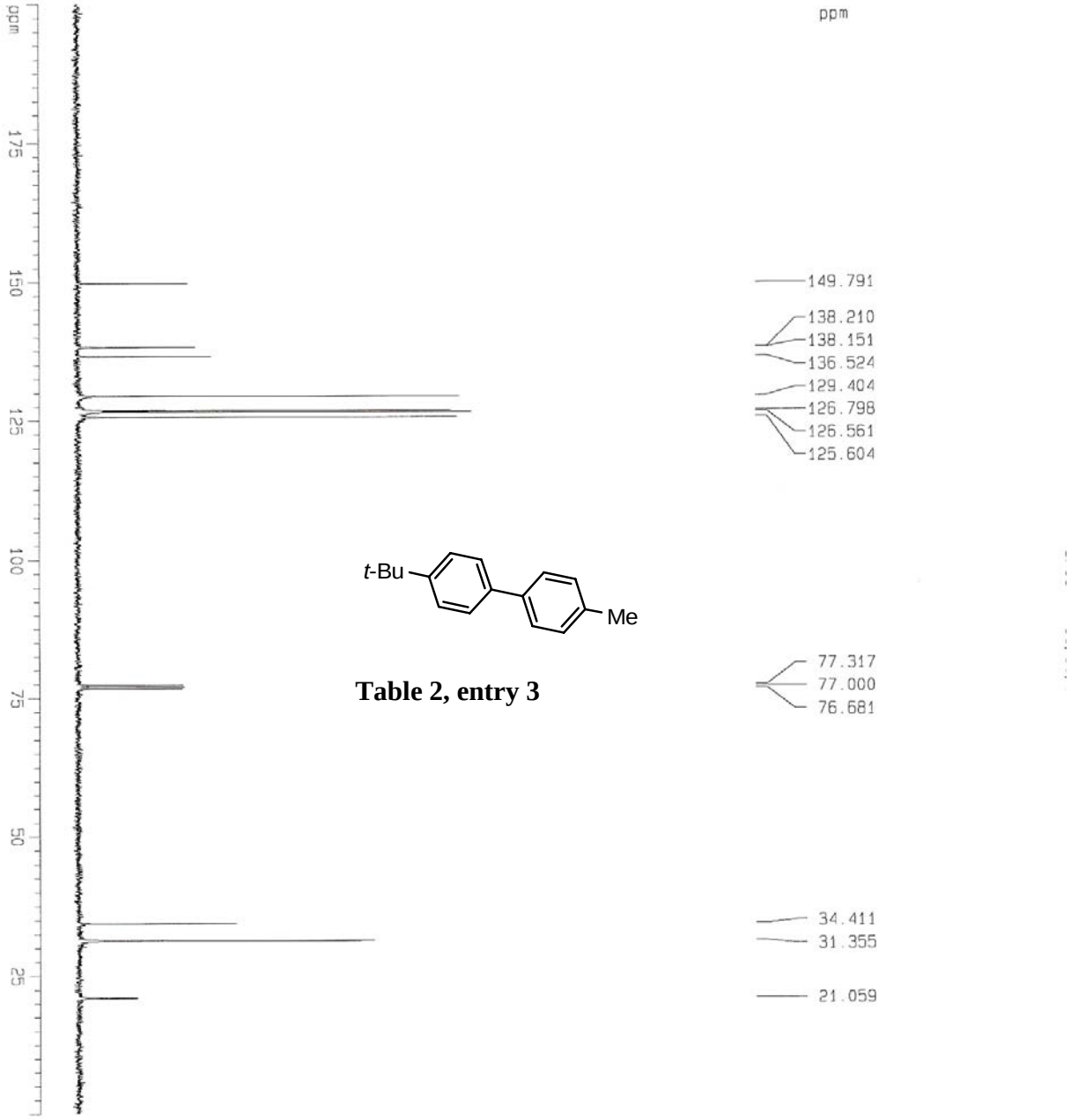
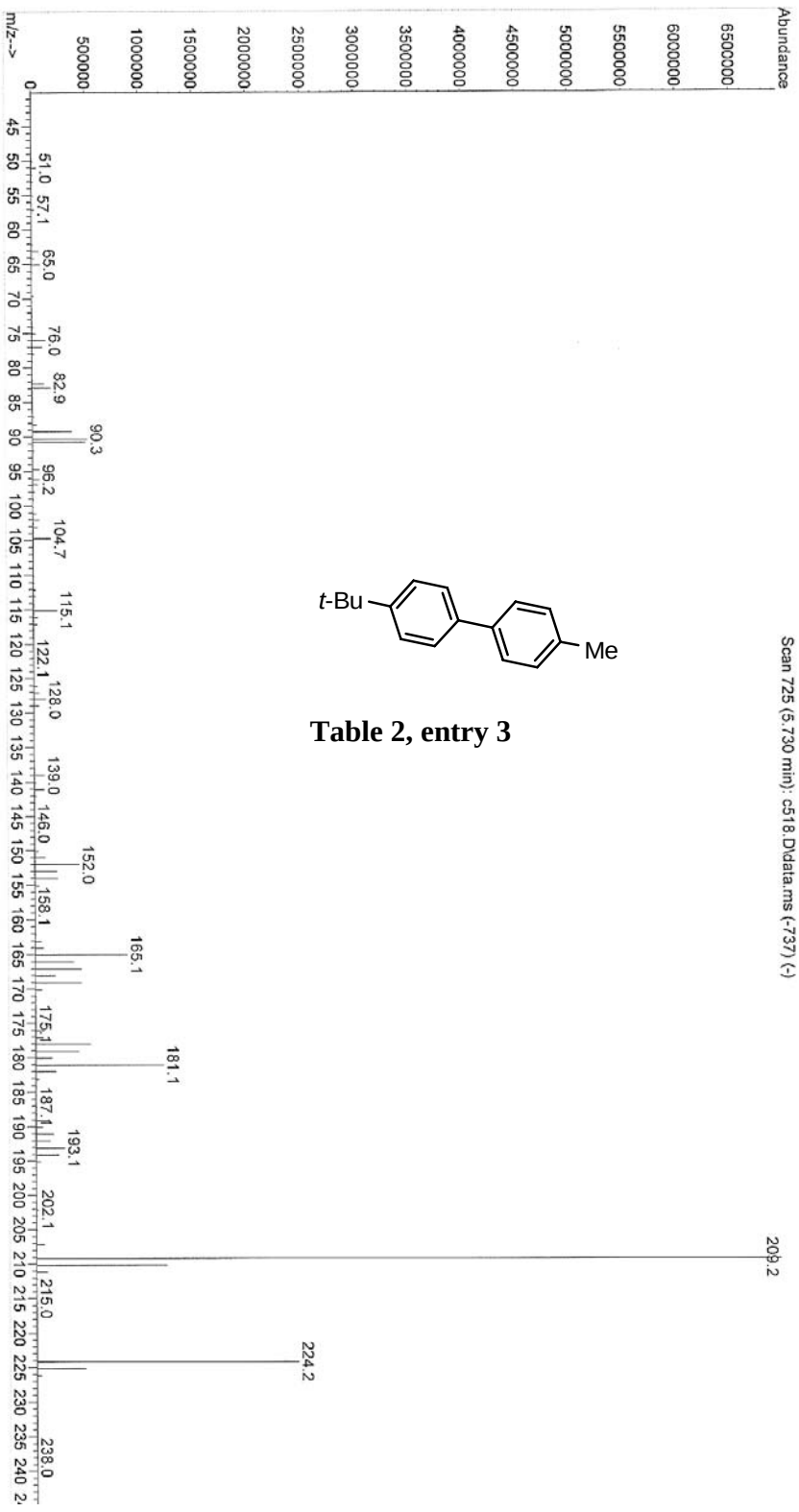


Table 2, entry 3


```
Current Data Parameters
NAME                  Ccoupling
EXPNO                        99
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20071115
Time                       21.11
INSTRUM                  dpx400
PROBHD     5 mm QNP 1H
PULPROG                zgdc30
TD                         131072
SOLVENT                    CDCl3
NS                         16
DS                         0
SWH                        25125.629 Hz
FIDRES                     0.191693 Hz
AQ                         2.6083827 sec
RG                         8192
DW                         19.900 usec
DE                         4.50 usec
TE                         300.0 K
D1                         3.00000000 sec
d11                        0.03000000 sec

========== CHANNEL f1 =====
NUC1                       13C
P1                         5.80 usec
PL1                        -6.00 dB
SF01                       100.6231263 MHz

========== CHANNEL f2 =====
CPDPRG2                    waltz16
NUC2                       1H
PCPD2                      71.00 usec
PL2                        120.00 dB
PL12                       17.00 dB
SFO2                       400.1326008 MHz

F2 - Processing parameters
SI                         65536
SF                         100.6128039 MHz
WDW                        EM
SSB                        0
LB                         3.00 Hz
GB                         0
PC                         1.00

1D NMR plot parameters
CX                         19.50 cm
F1P                        200.000 ppm
F1                         20122.56 Hz
F2P                        -0.000 ppm
F2                         -0.00 Hz
PPMCM                      10.25641 ppm/c
HZCM                       1031.92615 Hz/cm
```



t-Bu

Me

Table 2, entry 3

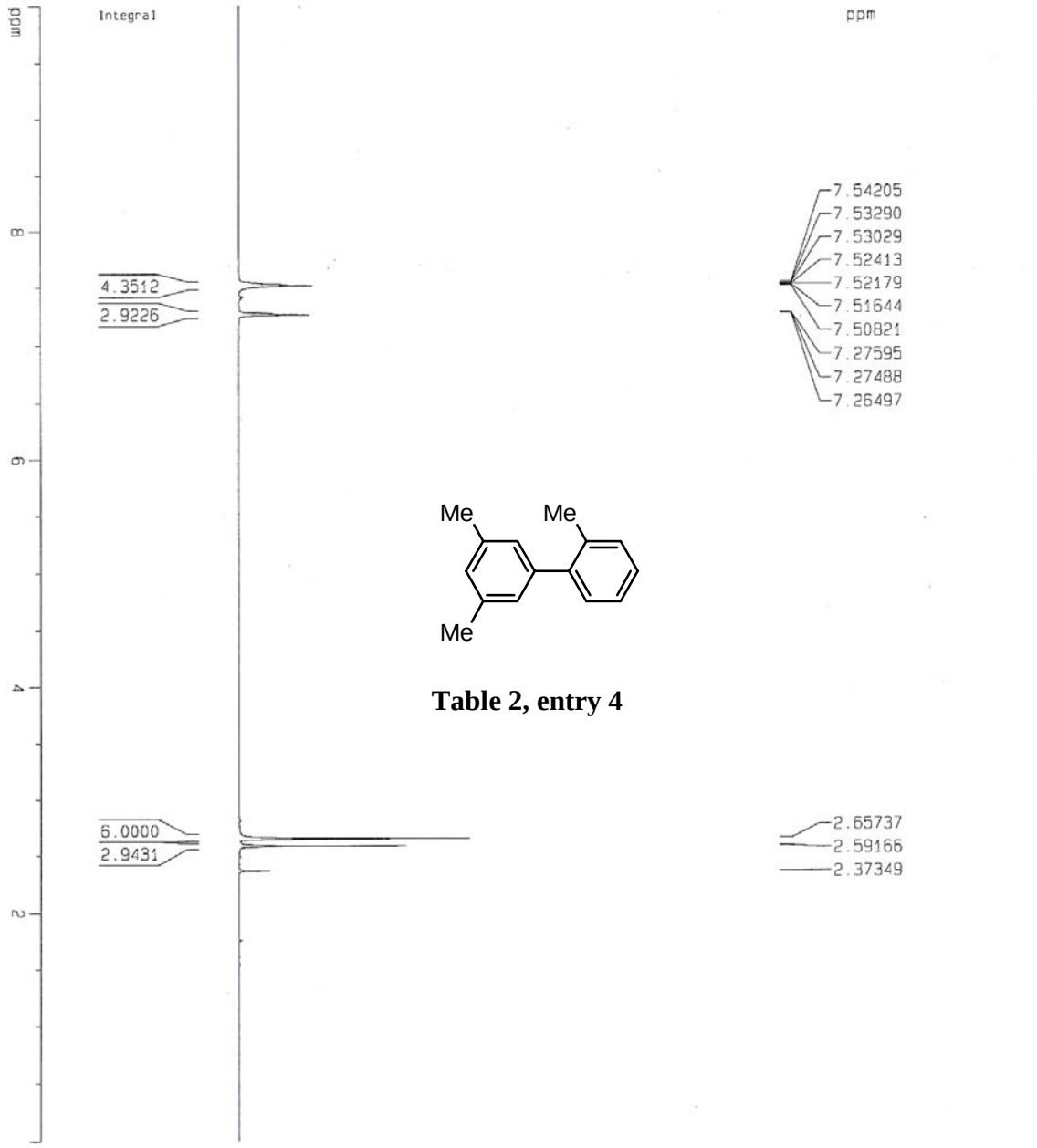


Table 2, entry 4


```
Current Data Parameters
NAME                  coupling
EXPNO                      141
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20071115
Time                      11.17
INSTRUM                 dpx400
PROBHD     5 mm QNP 1H
PULPROG                   zg30
TD                       32768
SOLVENT                  CDCl3
NS                           4
DS                           0
SWH                   4789.272 Hz
FIDRES                0.146157 Hz
AQ                    3.4210291 sec
RG                        28.5
DW                      104.400 usec
DE                        4.50 usec
TE                       300.0 K
D1                    1.00000000 sec

======== CHANNEL f1 ========
NUC1                         1H
P1                         9.50 usec
PL1                       -6.00 dB
SFO1                  400.1320007 MHz

F2 - Processing parameters
SI                        16384
SF                    400.1300089 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                        19.63 cm
F1P                    10.000 ppm
F1                     4001.30 Hz
F2P                    -0.000 ppm
F2                      -0.00 Hz
PPMCM                  0.50930 ppm/cm
HZCM                  203.78769 Hz/cm
```

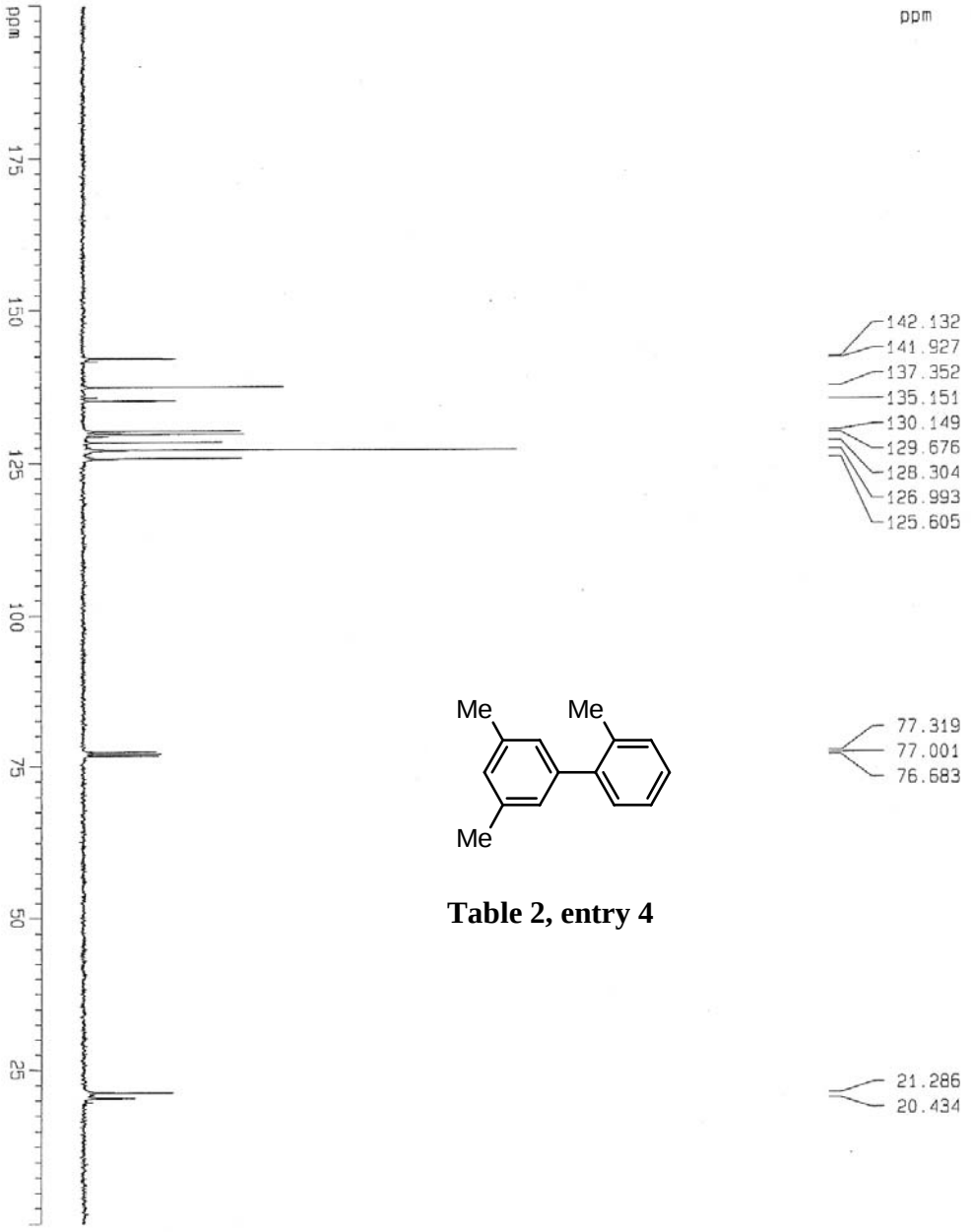


Table 2, entry 4


```
Current Data Parameters
NAME                  Ccoupling
EXPNO                        94
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20071115
Time                         11.19
INSTRUM                  dpx400
PROBHD     5 mm QNP 1H
PULPROG                  zgdc30
TD                       131072
SOLVENT                  CDCl3
NS                           16
DS                            0
SWH                   25125.629 Hz
FIDRES                0.191693 Hz
AQ                    2.6083827 sec
RG                         8192
DW                       19.900 usec
DE                        4.50 usec
TE                       300.0 K
D1                    3.00000000 sec
d11                   0.03000000 sec

========== CHANNEL f1 =======
NUC1                        13C
P1                         5.80 usec
PL1                       -6.00 dB
SF01                  100.6231263 MHz

========== CHANNEL f2 =======
CPDPRG2                   waltz16
NUC2                         1H
PCPD2                     71.00 usec
PL2                       120.00 dB
PL12                       17.00 dB
SFO2                  400.1320008 MHz

F2 - Processing parameters
SI                         65536
SF                    100.6128074 MHz
WDW                           EM
SSB                            0
LB                          3.50 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         19.50 cm
F1P                      200.000 ppm
F1                      20122.56 Hz
F2P                       -0.000 ppm
F2                        -0.00 Hz
PPMCM                   10.25641 ppm/cm
HZCM                  1031.92627 Hz/cm
```

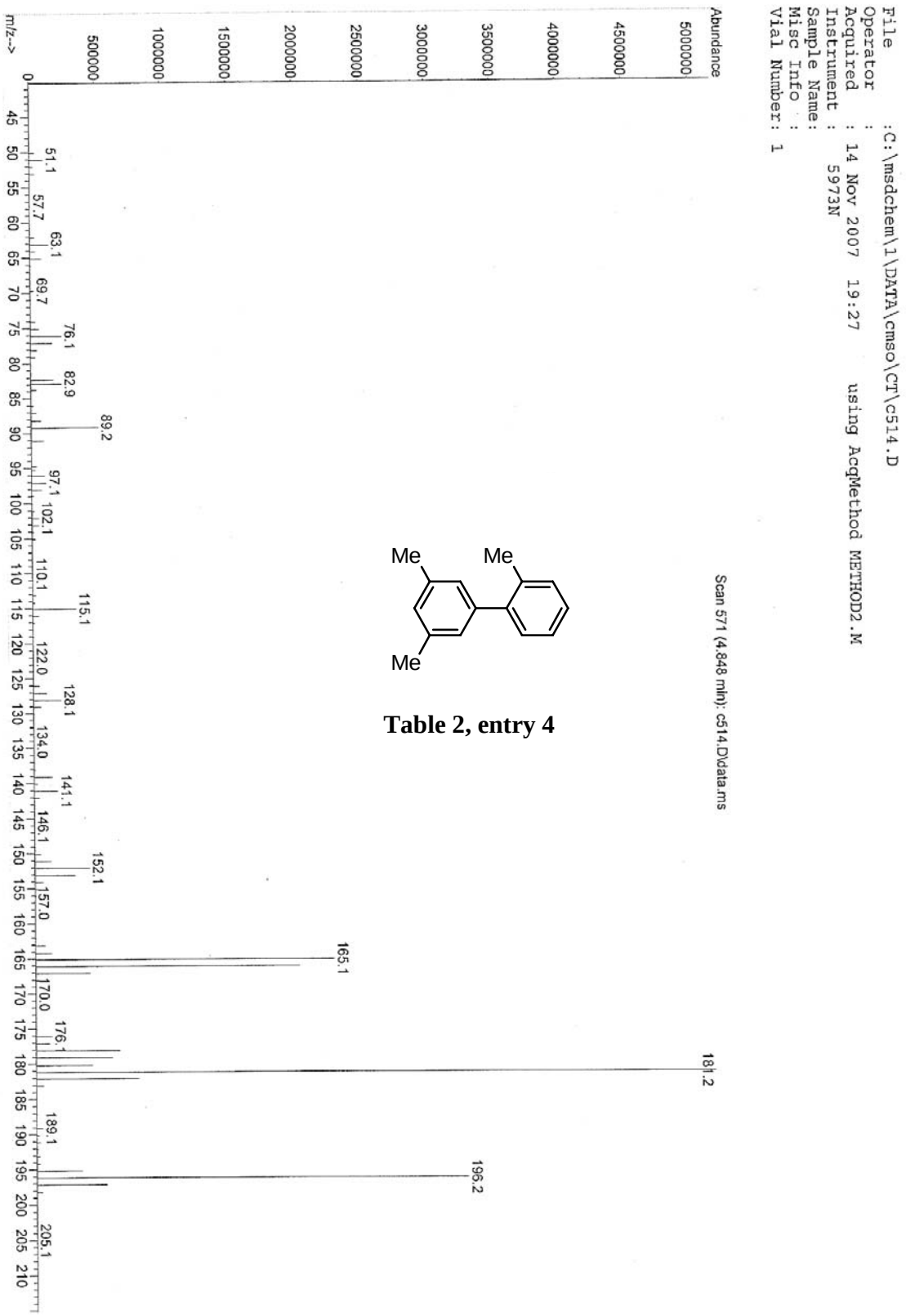


Table 2, entry 4

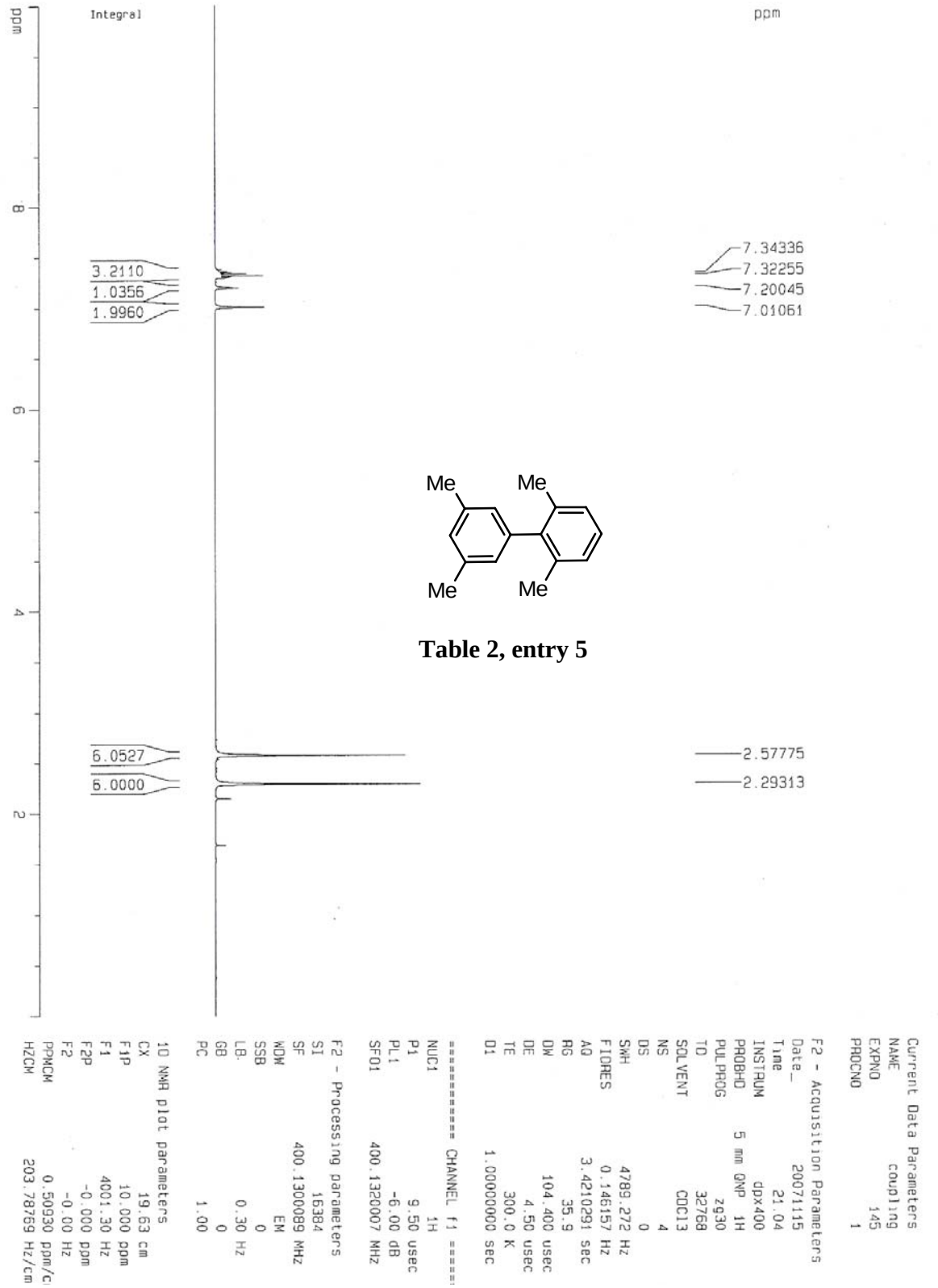


Table 2, entry 5


```
Current Data Parameters
NAME                  coupling
EXPNO                      145
PROCNO                       1

F2 - Acquisition Parameters
Date_               20071115
Time                   21.04
INSTRUM                dpx400
PROBHD     5 mm QNP  1H
PULPROG                zg30
TD                     32768
SOLVENT                CDCl3
NS                         4
DS                         0
SWH                 4789.272 Hz
FIDRES              0.146157 Hz
AQ                  3.4210291 sec
RG                      35.9
DW                 104.400 usec
DE                  4.50 usec
TE                  300.0 K
D1                  1.00000000 sec

======== CHANNEL f1 ======
NUC1                    1H
P1                   9.50 usec
PL1                  -6.00 dB
SFO1           400.1320007 MHz

F2 - Processing parameters
SI                      16384
SF             400.1300089 MHz
WDW                        EM
SSB                         0
LB                   0.30 Hz
GB                          0
PC                   1.00

1D NMR plot parameters
CX                   19.63 cm
F1P                  10.000 ppm
F1                   4001.30 Hz
F2P                  -0.000 ppm
F2                    -0.00 Hz
PPMCM                0.50930 ppm/cm
HZCM                 203.78769 Hz/cm
```

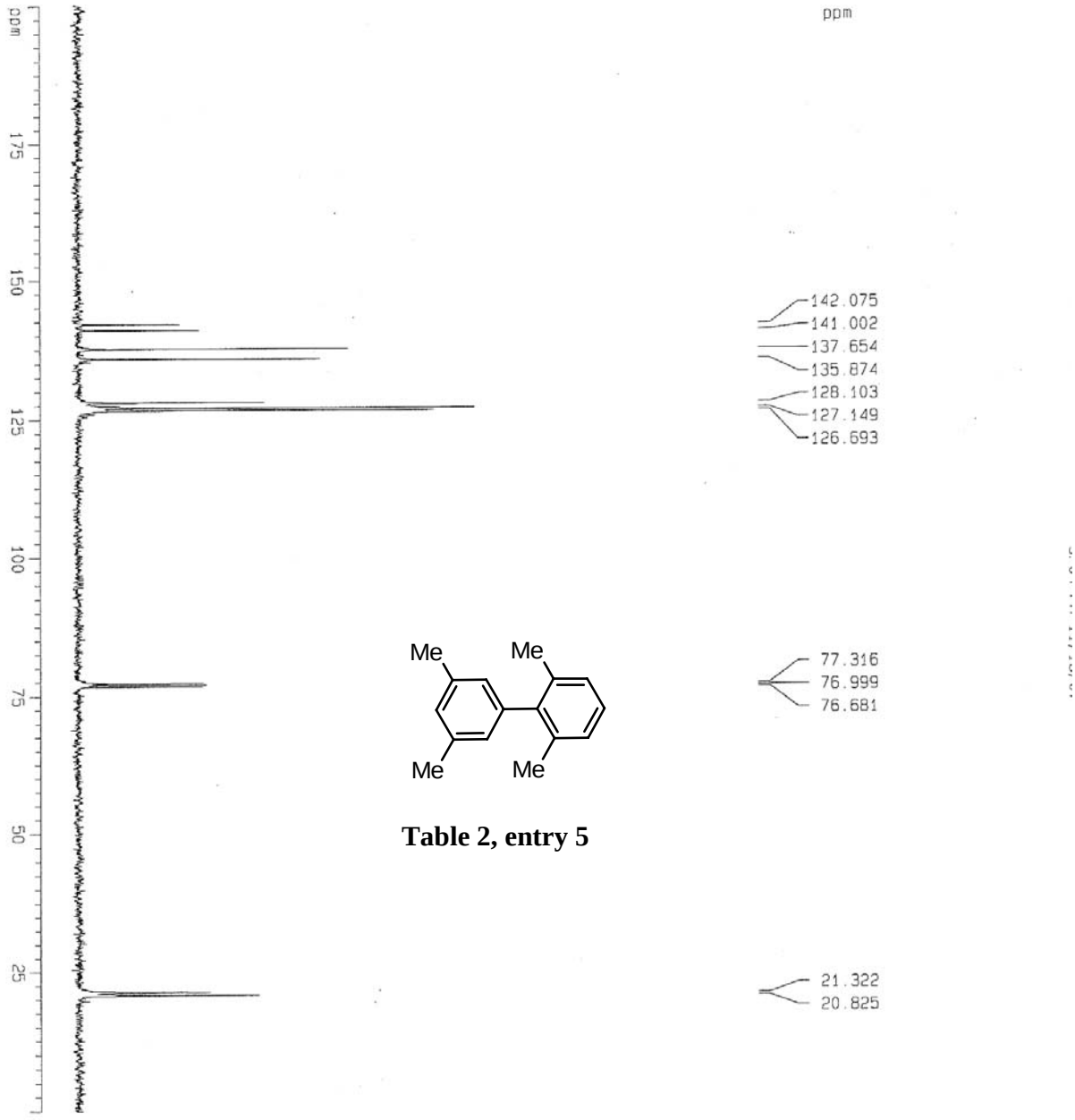


Table 2, entry 5


```
Current Data Parameters
NAME                  Ccoupling
EXPNO                        98
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20071115
Time                         21.05
INSTRUM                    dpx400
PROBHD     5 mm QNP  1H
PULPROG                   zgdc30
TD                        131072
SOLVENT                   CDCl3
NS                            16
DS                             0
SWH                    25125.629 Hz
FIDRES                     0.191693 Hz
AQ                         2.6083827 sec
RG                          8192
DW                         19.900 usec
DE                          4.50 usec
TE                           300.0 K
D1                     3.00000000 sec
D11                    0.03000000 sec

======== CHANNEL f1 ========
NUC1                          13C
P1                           5.80 usec
PL1                         -6.00 dB
SFO1                  100.6231263 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                          1H
PCPD2                       71.00 usec
PL2                         120.00 dB
PL12                         17.00 dB
SFO2                  400.1325008 MHz

F2 - Processing parameters
SI                           65536
SF                    100.6127986 MHz
WDW                             EM
SSB                              0
LB                            5.00 Hz
GB                               0
PC                            1.00

1D NMR plot parameters
CX                           19.50 cm
F1P                         200.000 ppm
F1                        20122.56 Hz
F2P                        -0.000 ppm
F2                          -0.00 Hz
PPMCM                     10.25641 ppm/cm
HZCM                  1031.92615 Hz/cm
```

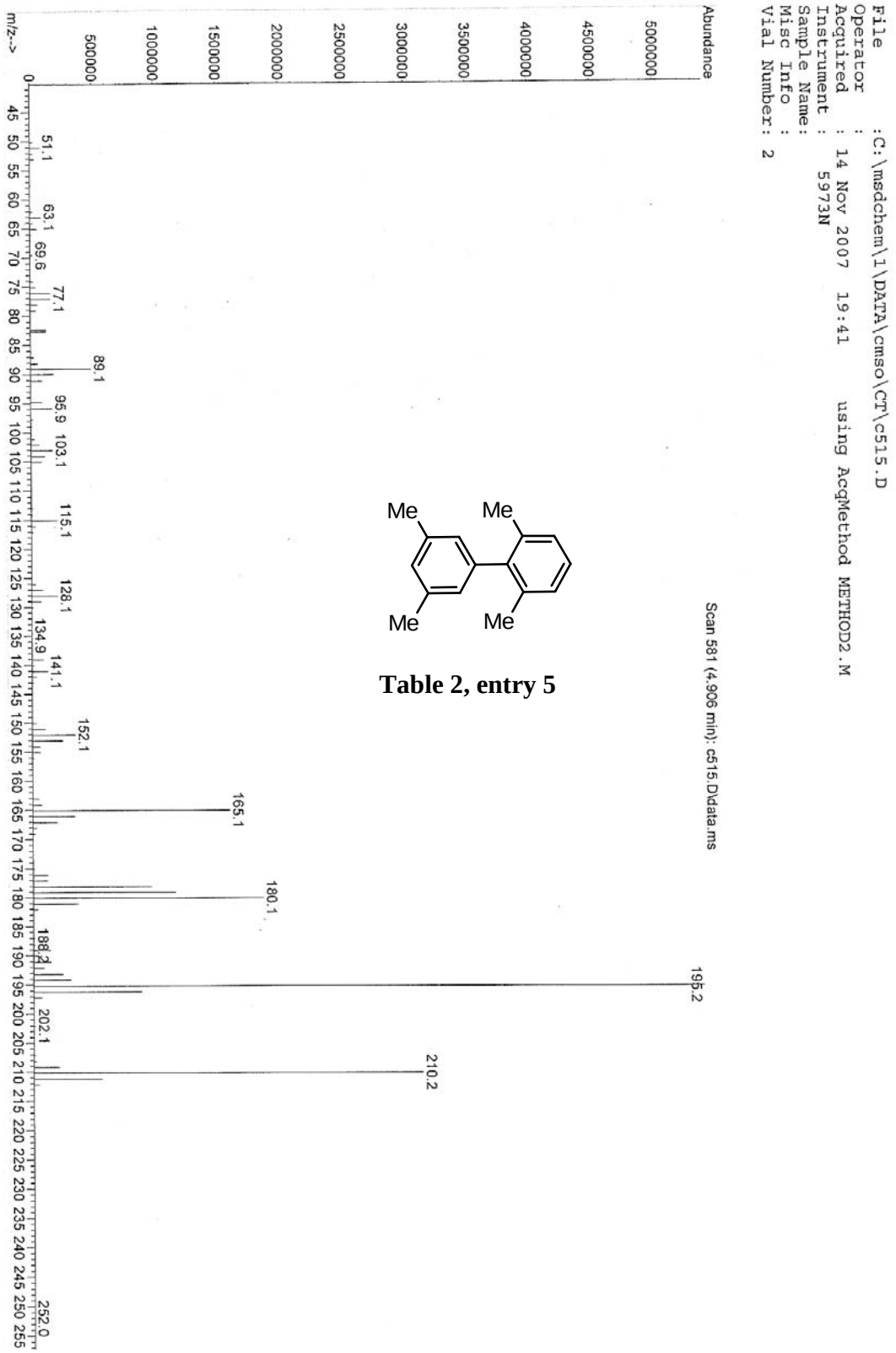


Table 2, entry 5

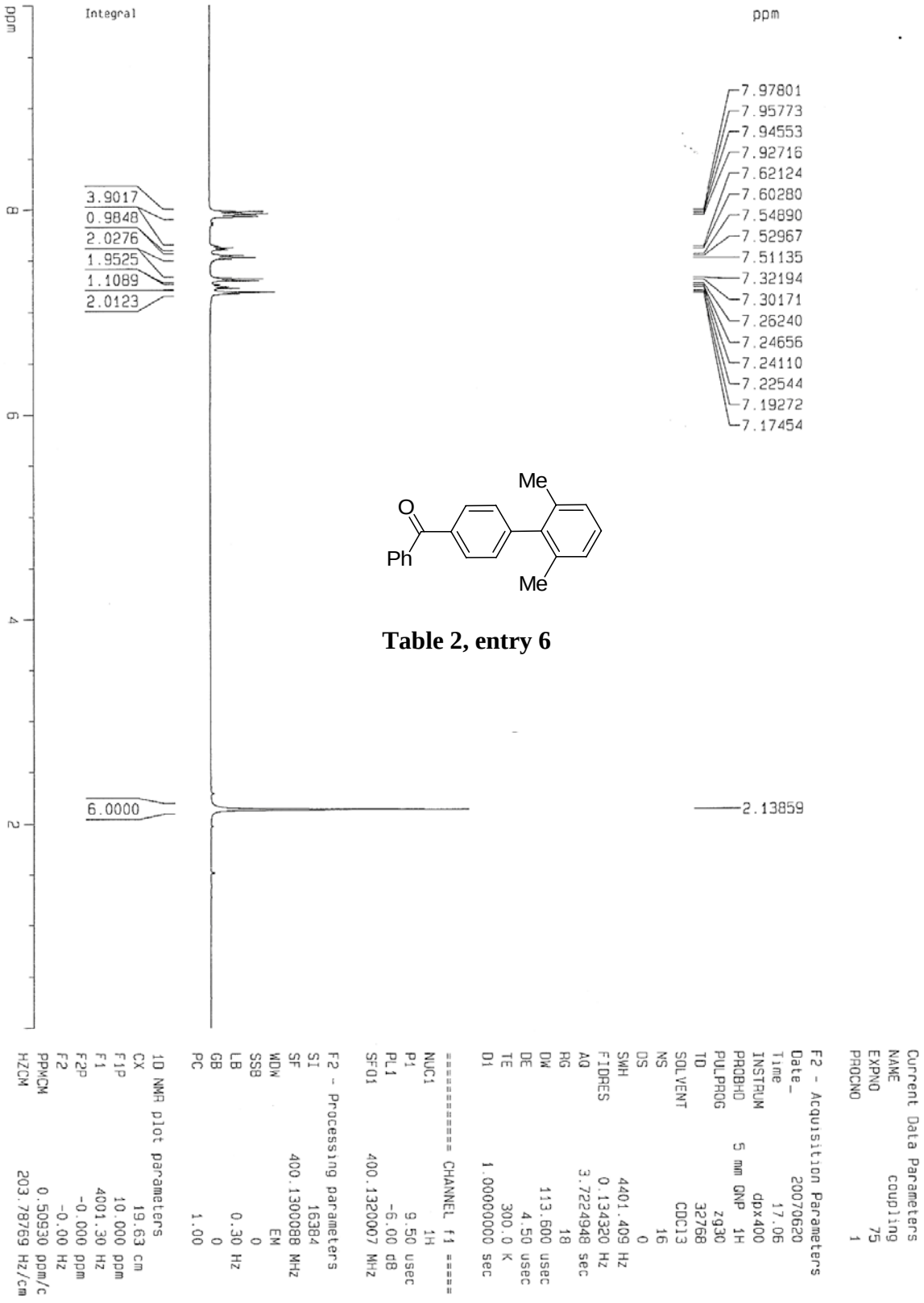


Table 2, entry 6


```
Current Data Parameters
NAME                  coupling
EXPNO                       75
PROCNO                       1

F2 - Acquisition Parameters
Date_              20070620
Time                   17.06
INSTRUM              dpx400
PROBHD     5 mm QNP  1H
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH                4401.409 Hz
FIDRES             0.134320 Hz
AQ                 3.7224948 sec
RG                     18
DW                 113.600 usec
DE                   4.50 usec
TE                 300.0 K
D1                 1.00000000 sec

======== CHANNEL f1 =====
NUC1                   1H
P1                   9.50 usec
PL1                 -6.00 dB
SFO1           400.1320007 MHz

F2 - Processing parameters
SI                     16384
SF             400.1300088 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                   19.63 cm
F1P                10.000 ppm
F1                 4001.30 Hz
F2P                -0.000 ppm
F2                 -0.00 Hz
PPMCM              0.50930 ppm/c
HZCM               203.78769 Hz/cm
```

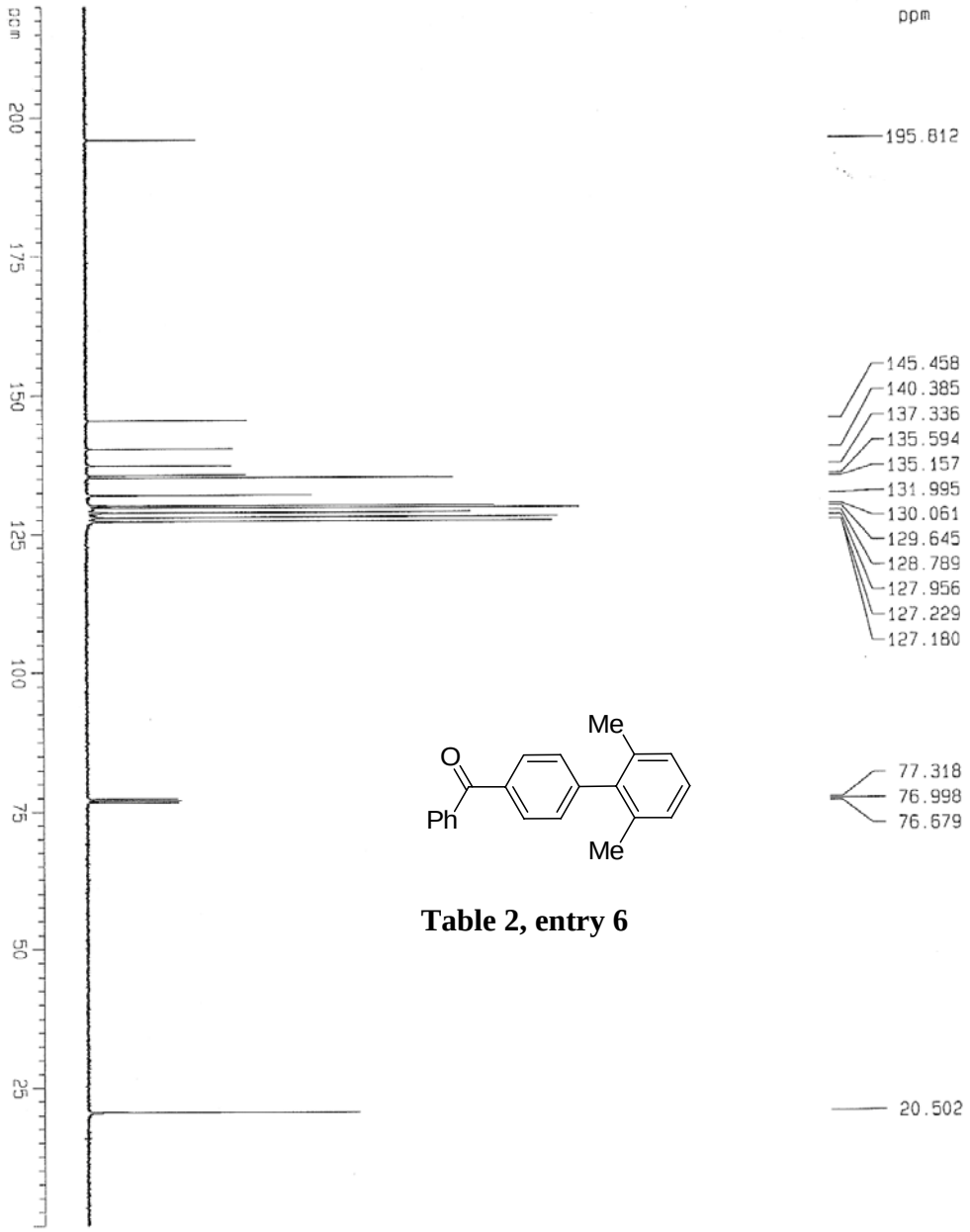


Table 2, entry 6

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

F2 - Acquisition Parameters
Date_              20070620
Time                   17.08
INSTRUM              dpx400
PROBHD     5 mm QNP 1H
PULPROG              zgdc30
TD                   131072
SOLVENT              CDCl3
NS                       64
DS                        0
SWH                25125.629 Hz
FIDRES                0.191693 Hz
AQ                 2.6083827 sec
RG                      8192
DW                    19.900 usec
DE                     4.50 usec
TE                    300.0 K
D1                 3.00000000 sec
d11                0.03000000 sec

========== CHANNEL f1 =======
NUC1                      13C
P1                       5.80 usec
PL1                     -6.00 dB
SFO1             100.6231263 MHz

========== CHANNEL f2 =======
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   71.00 usec
PL2                     120.00 dB
PL12                     17.00 dB
SF02             400.1326009 MHz

F2 - Processing parameters
SI                        65536
SF               100.6128177 MHz
WDW                          EM
SSB                           0
LB                         1.50 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                        19.50 cm
F1P                     220.000 ppm
F1                    22134.82 Hz
F2P                    -0.000 ppm
F2                      -0.00 Hz
PPMCM                 11.28205 ppm/cm
HZCM                1135.11902 Hz/cm
```

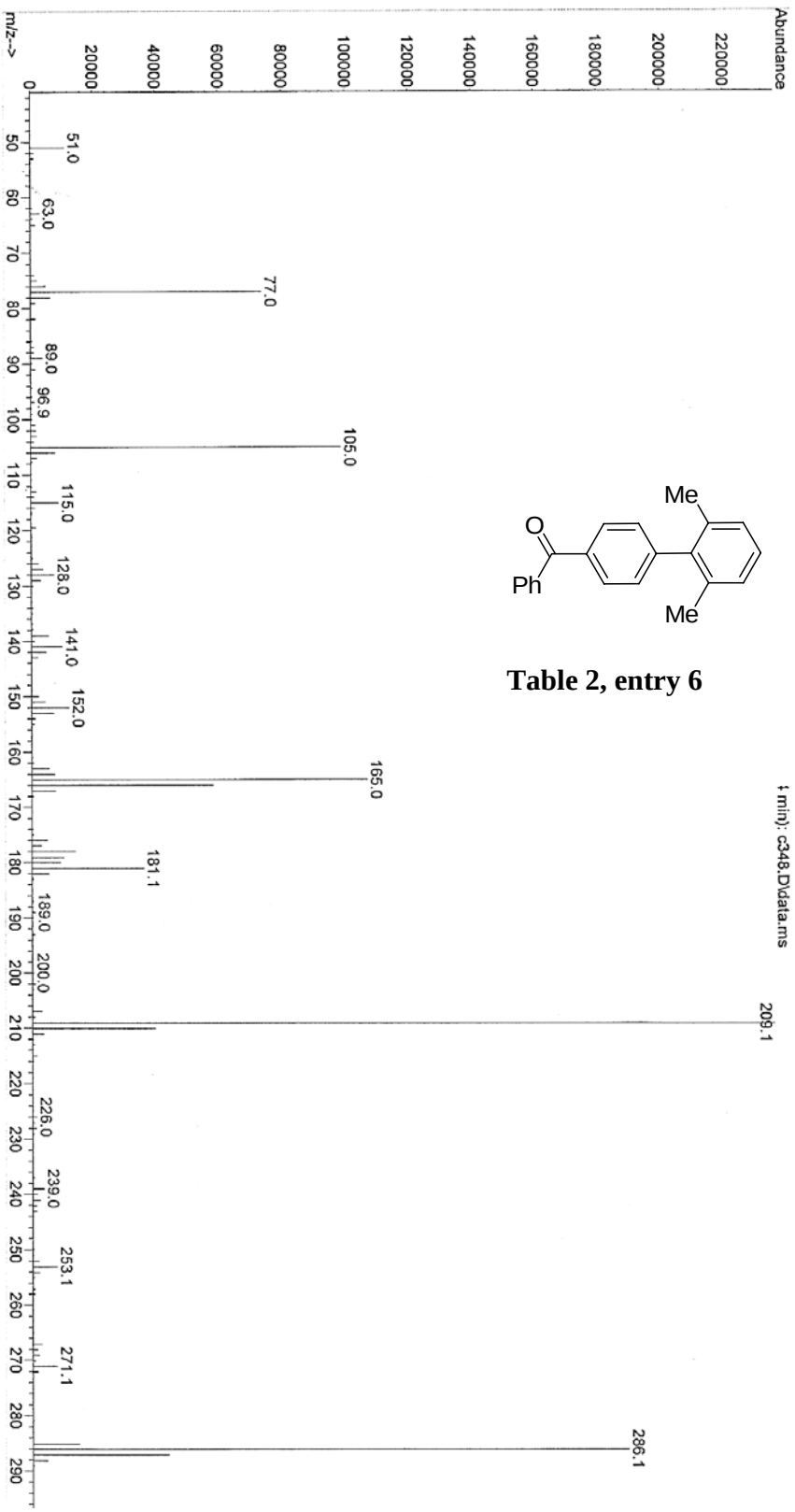


Table 2, entry 6

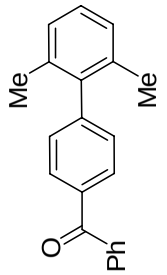


Table 2, entry 6

Elemental Composition Report

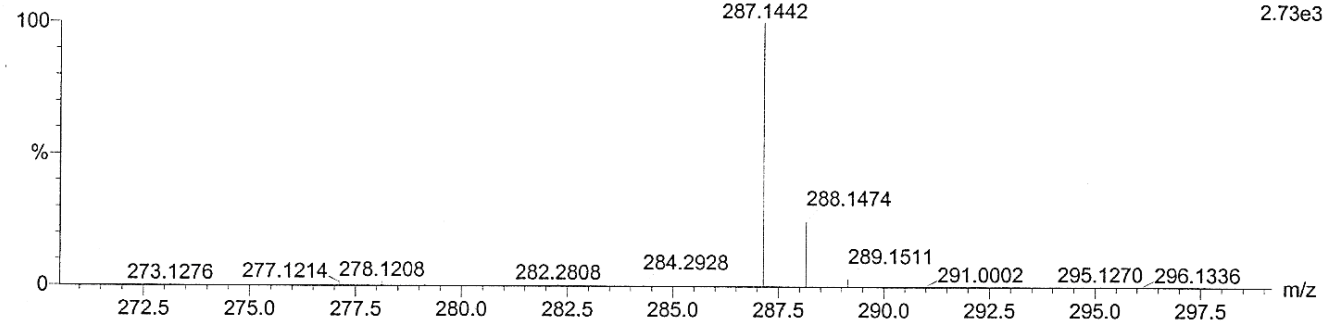
Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
5 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-21 H: 0-1000 O: 0-1

So chau Ming, 4-(2'6'-dimethylphenyl)benzophenone
HR07_0724_4 47 (0.891) AM (Cen,5, 80.00, Ht,10000.0,0.00,1.00); Sm (SG, 2x3.00); Cm (31:57)

TOF MS ES+
2.73e3



Minimum:				-1.5		
Maximum:		5.0	10.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
287.1442	287.1436	0.6	2.1	12.5	0.7	C21 H19 O

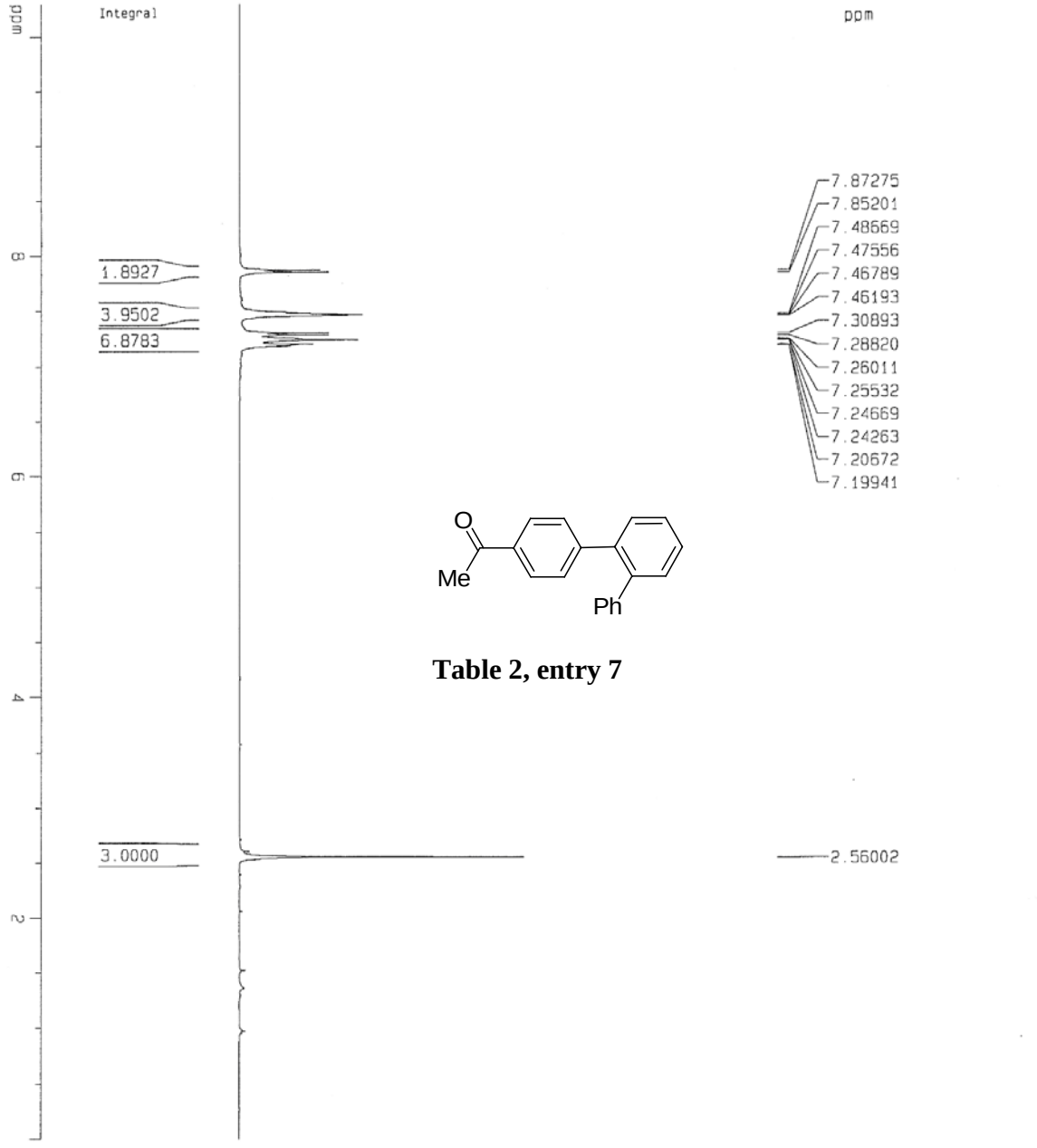


Table 2, entry 7


```
Current Data Parameters
NAME                  coupling
EXPNO                      101
PROCNO                       1

F2 - Acquisition Parameters
Date_              20070825
Time                   11.25
INSTRUM                dpx400
PROBHD     5 mm QNP 1H
PULPROG                zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                4401.409 Hz
FIDRES             0.134320 Hz
AQ                 3.7224948 sec
RG                      25.4
DW                 113.600 usec
DE                   4.50 usec
TE                   300.0 K
D1                 1.00000000 sec
============== CHANNEL f1 ======
NUC1                       1H
P1                       9.50 usec
PL1                     -6.00 dB
SF01           400.1320006 MHz

F2 - Processing parameters
SI                       16384
SF             400.1300077 MHz
WDW                         EM
SSB                          0
LB                        0.70 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       19.63 cm
F1P                   10.300 ppm
F1                  4121.34 Hz
F2P                   0.000 ppm
F2                     0.00 Hz
PPMCM                 0.52458 ppm/cm
HZCM               209.90131 Hz/cm
```

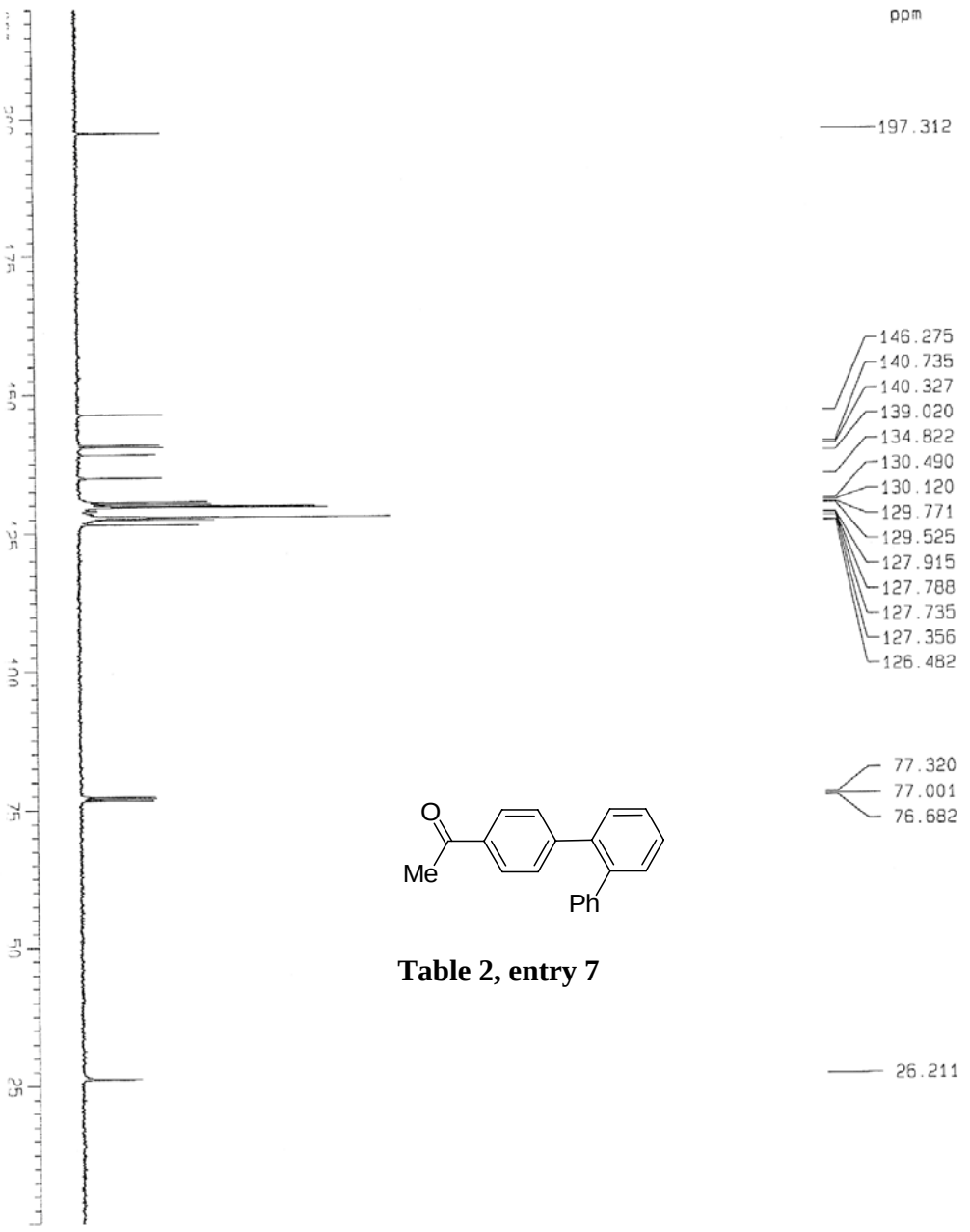


Table 2, entry 7

```
Current Data Parameters
NAME                  Ccoupling
EXPNO                        58
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20070825
Time                      11.27
INSTRUM                 dpx400
PROBHD     5 mm QNP 1H
PULPROG                 zgdc30
TD                      131072
SOLVENT                 CDCl3
NS                          32
DS                           0
SWH                  25125.629 Hz
FIDRES                0.191693 Hz
AQ                    2.6083827 sec
RG                         8192
DW                       19.900 usec
DE                        4.50 usec
TE                       300.0 K
D1                    3.00000000 sec
d11                   0.03000000 sec

========== CHANNEL f1 =======
NUC1                        13C
P1                         5.80 usec
PL1                       -6.00 dB
SFO1                100.6231263 MHz

========== CHANNEL f2 =======
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     71.00 usec
PL2                      120.00 dB
PL12                      17.00 dB
SFO2                400.1326009 MHz

F2 - Processing parameters
SI                         65536
SF                  100.6128116 MHz
WDW                            EM
SSB                             0
LB                         4.00 Hz
GB                              0
PC                         1.00

1D NMR plot parameters
CX                        19.50 cm
F1P                     220.000 ppm
F1                     22134.82 Hz
F2P                     -0.000 ppm
F2                       -0.00 Hz
PPMCM                 11.28205 ppm/cm
HZCM                  1135.11690 Hz/cm
```

Table 2, entry 7

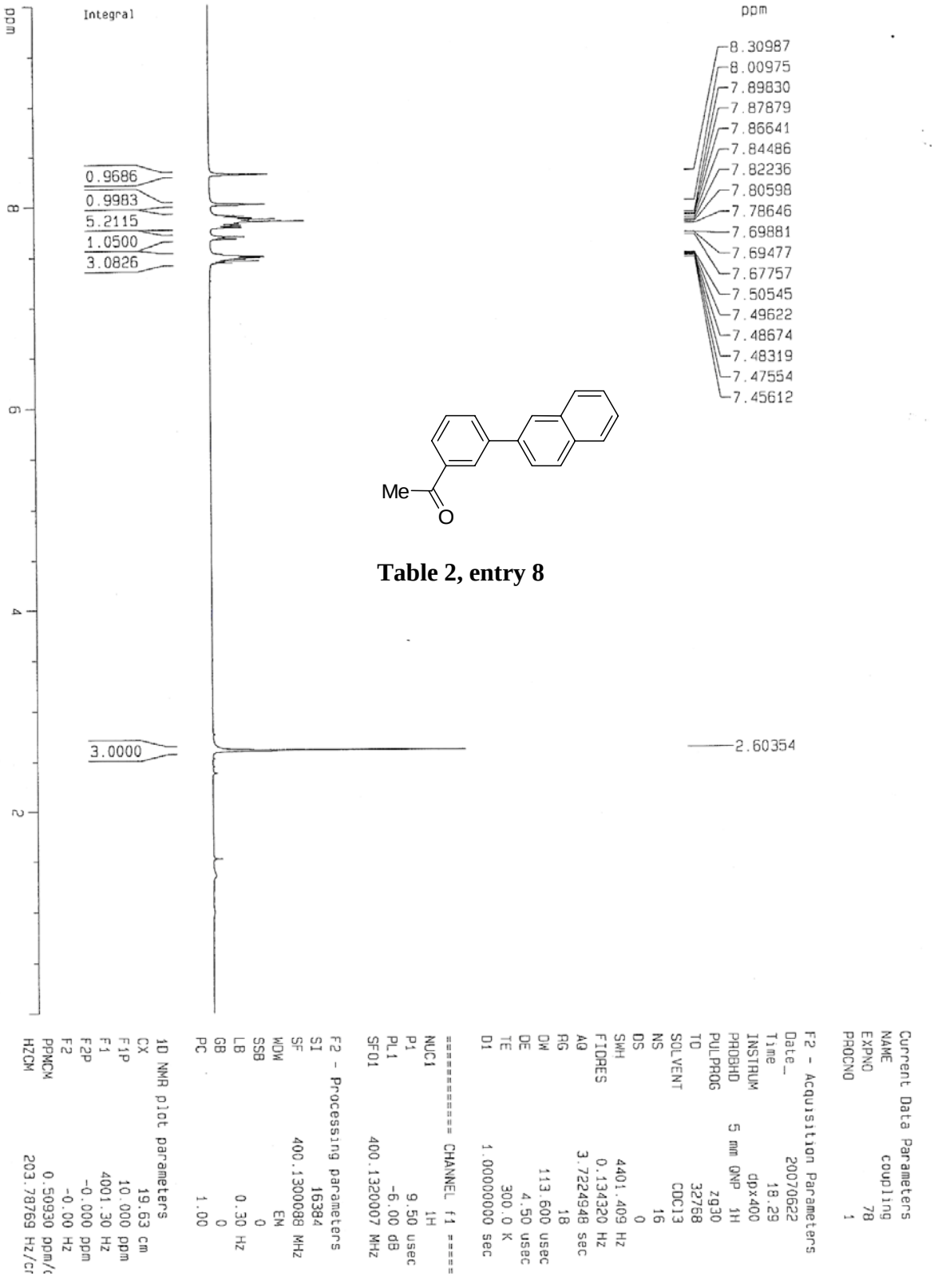


Table 2, entry 8

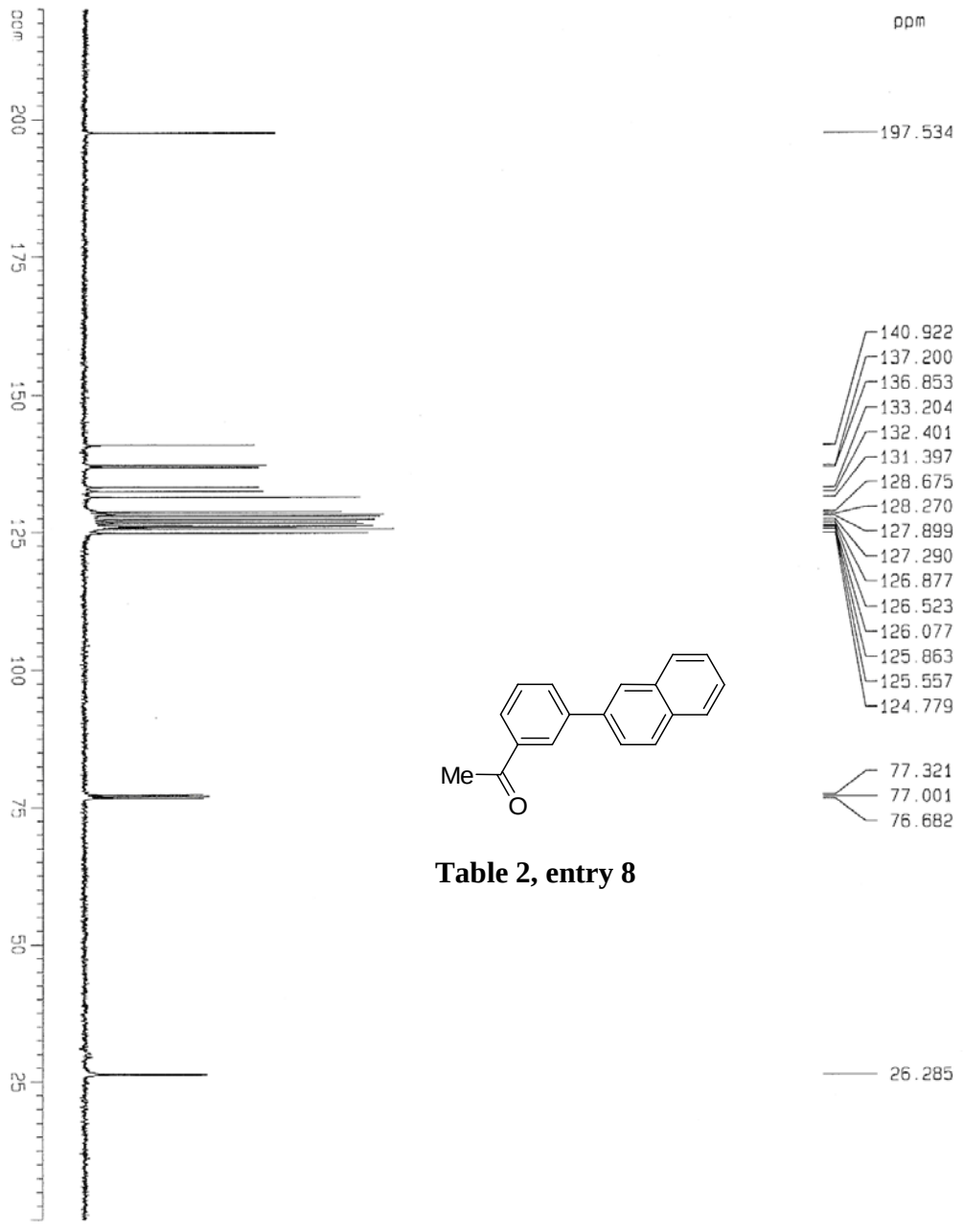


Table 2, entry 8


```
Current Data Parameters
NAME                  Ccoupling
EXPNO                        43
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20070522
Time                      18.31
INSTRUM                 dpx400
PROBHD     5 mm QNP 1H
PULPROG                zgdc30
TD                     131072
SOLVENT                CDCl3
NS                            32
DS                             0
SWH                    25125.629 Hz
FIDRES                 0.191693 Hz
AQ                     2.6083827 sec
RG                          8192
DW                       19.900 usec
DE                        4.50 usec
TE                       300.0 K
D1                     3.00000000 sec
d11                    0.03000000 sec

========== CHANNEL f1 =====
NUC1                         13C
P1                         5.80 usec
PL1                       -6.00 dB
SF01           100.6231263 MHz

========== CHANNEL f2 =====
CPDPRG2                  waltz16
NUC2                         1H
PCPD2                     71.00 usec
PL2                       120.00 dB
PL12                       17.00 dB
SF02           400.1326008 MHz

F2 - Processing parameters
SI                         65536
SF             100.6128181 MHz
WDW                           EM
SSB                            0
LB                          3.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         19.50 cm
F1P                      220.000 ppm
F1                     22134.82 Hz
F2P                     -0.000 ppm
F2                      -0.00 Hz
PPMCM                  11.28205 ppm/c
HZCM                   1135.11902 Hz/cm
```

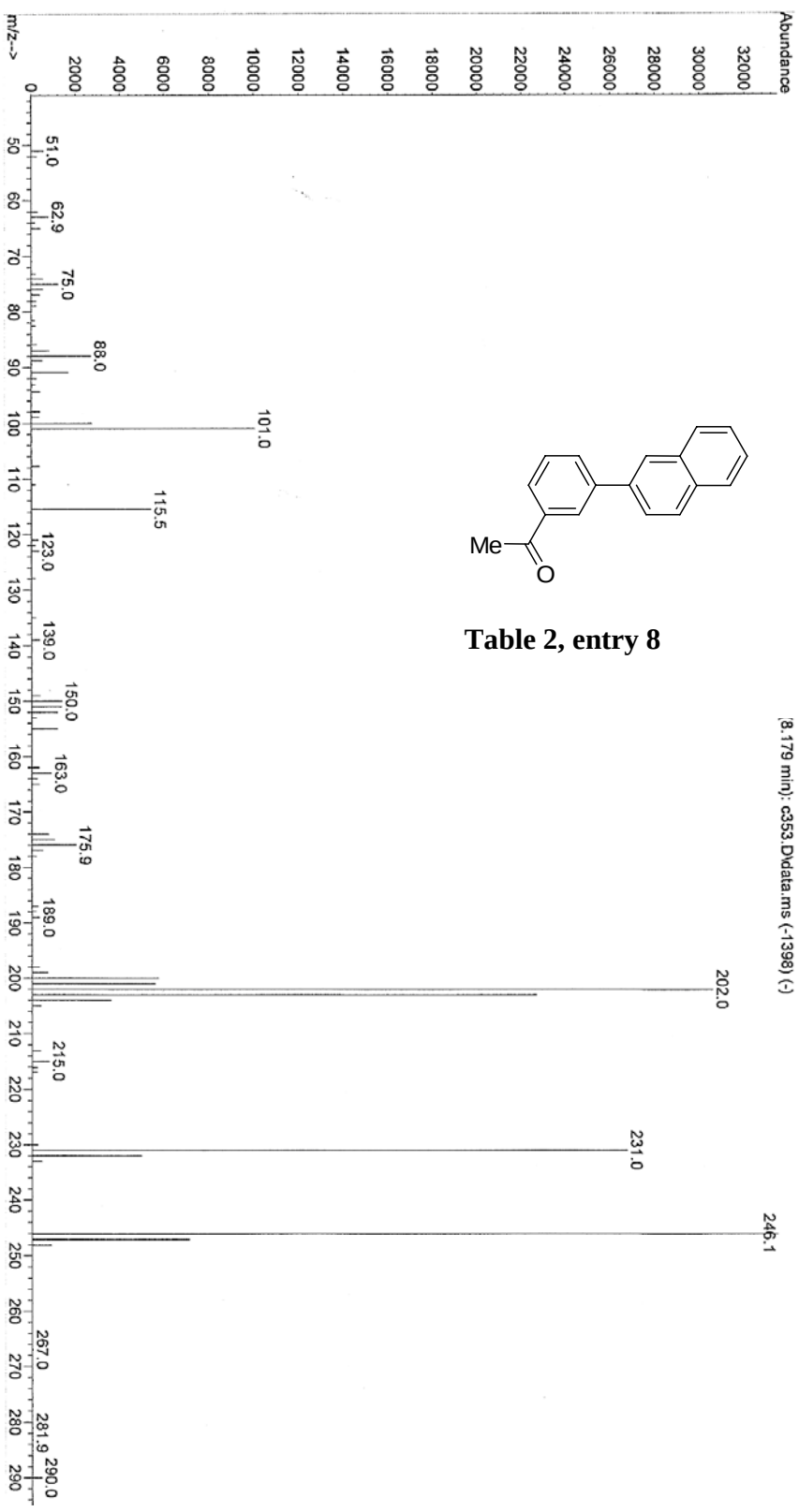


Table 2, entry 8

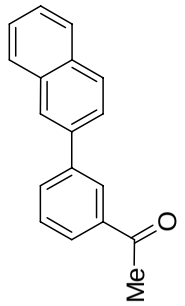


Table 2, entry 8

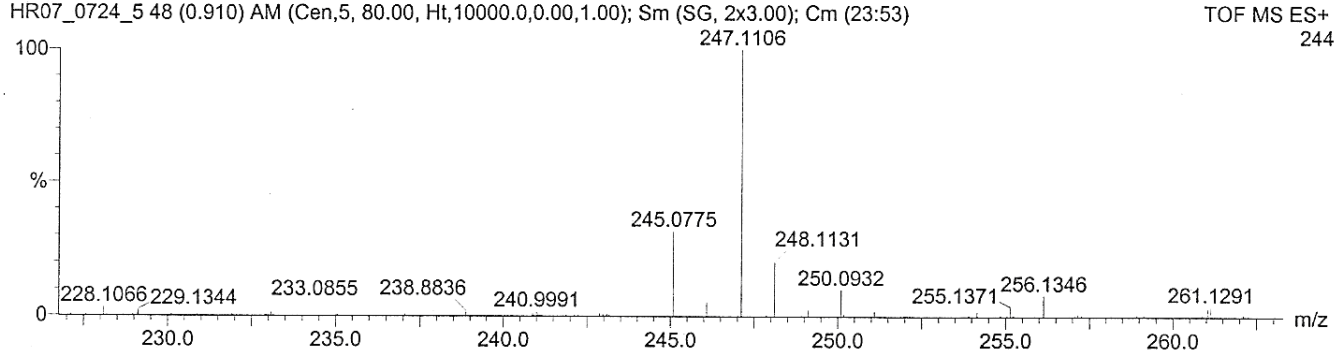
Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
8 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-21 H: 0-1000 O: 0-1

So Chau Ming, 3-naphthylacetophenone
HR07_0724_5 48 (0.910) AM (Cen,5, 80.00, Ht,10000.0,0.00,1.00); Sm (SG, 2x3.00); Cm (23:53)



Minimum:				-1.5		
Maximum:		5.0	10.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
247.1106	247.1123	-1.7	-6.9	11.5	0.1	C18 H15 O

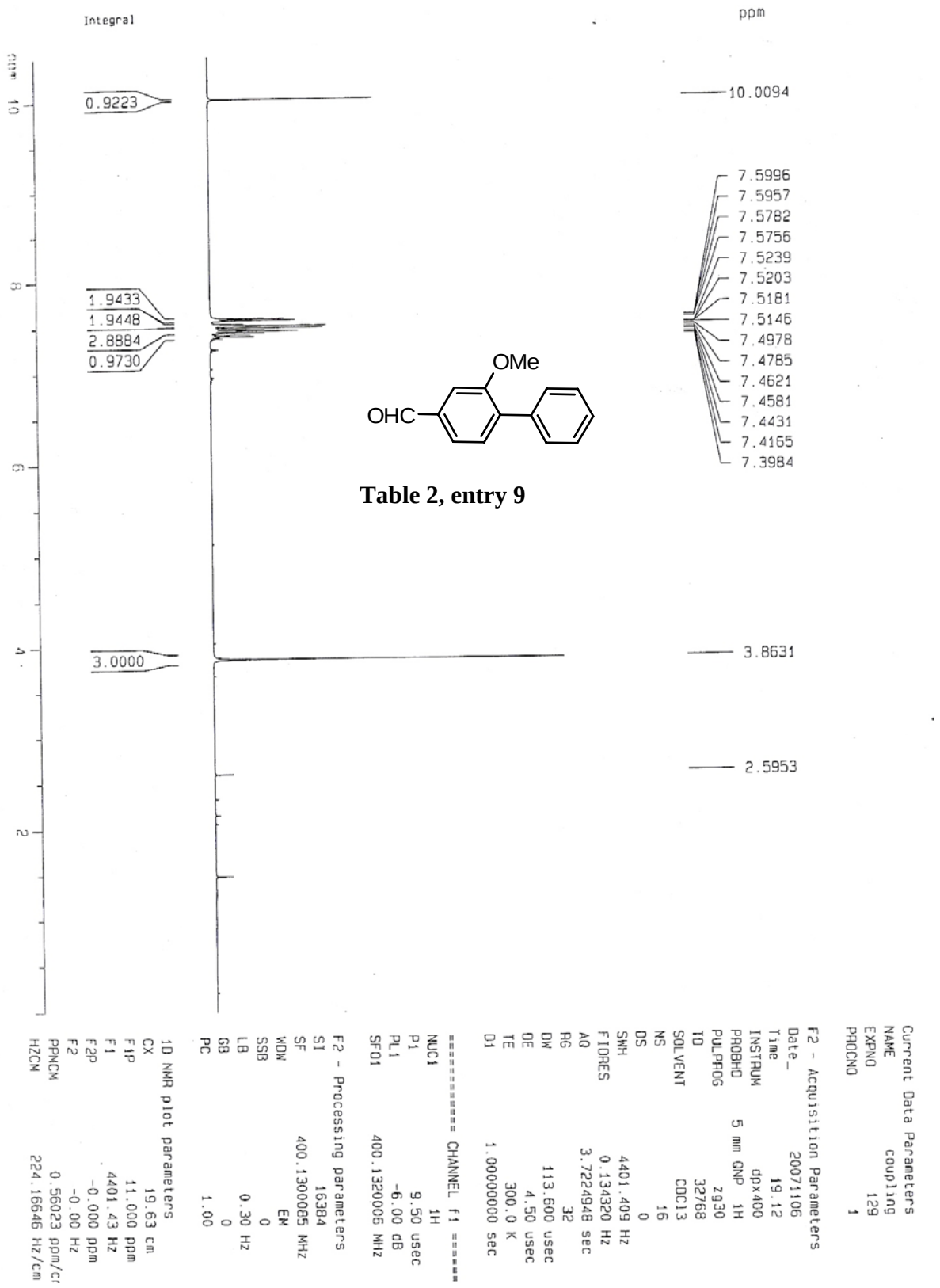


Table 2, entry 9

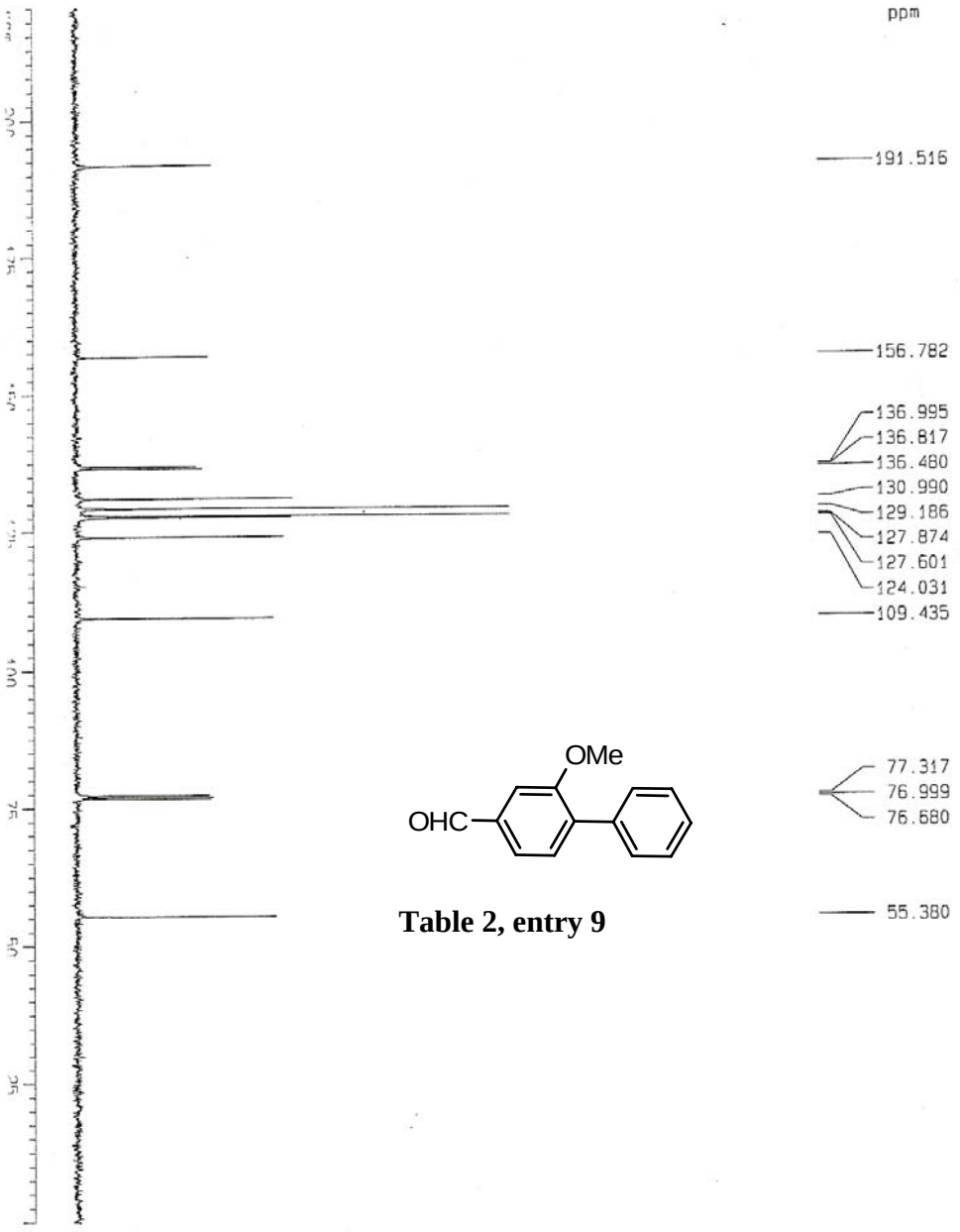


Table 2, entry 9


```
Current Data Parameters
NAME                  Ccoupling
EXPNO                        83
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20071106
Time                      19.16
INSTRUM                 dpx400
PROBHD     5 mm QNP 1H
PULPROG                 zgdc30
TD                      131072
SOLVENT                  CDCl3
NS                           16
DS                            0
SWH                   25125.629 Hz
FIDRES                0.191693 Hz
AQ                    2.6083827 sec
RG                         8192
DW                       19.900 usec
DE                        4.50 usec
TE                       300.0 K
D1                    3.00000000 sec
d11                   0.03000000 sec

========== CHANNEL f1 ========
NUC1                        13C
P1                         5.80 usec
PL1                       -6.00 dB
SFO1                100.6231263 MHz

========== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                     71.00 usec
PL2                       20.00 dB
PL12                      17.00 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                         65536
SF                  100.6128047 MHz
WDW                            EM
SSB                             0
LB                         4.00 Hz
GB                              0
PC                         1.00

1D NMR plot parameters
CX                        19.50 cm
F1P                     220.000 ppm
F1                     22134.82 Hz
F2P                      -0.000 ppm
F2                       -0.00 Hz
PPMCM                  11.28205 ppm/cm
HZCM                1135.11690 Hz/cm
```

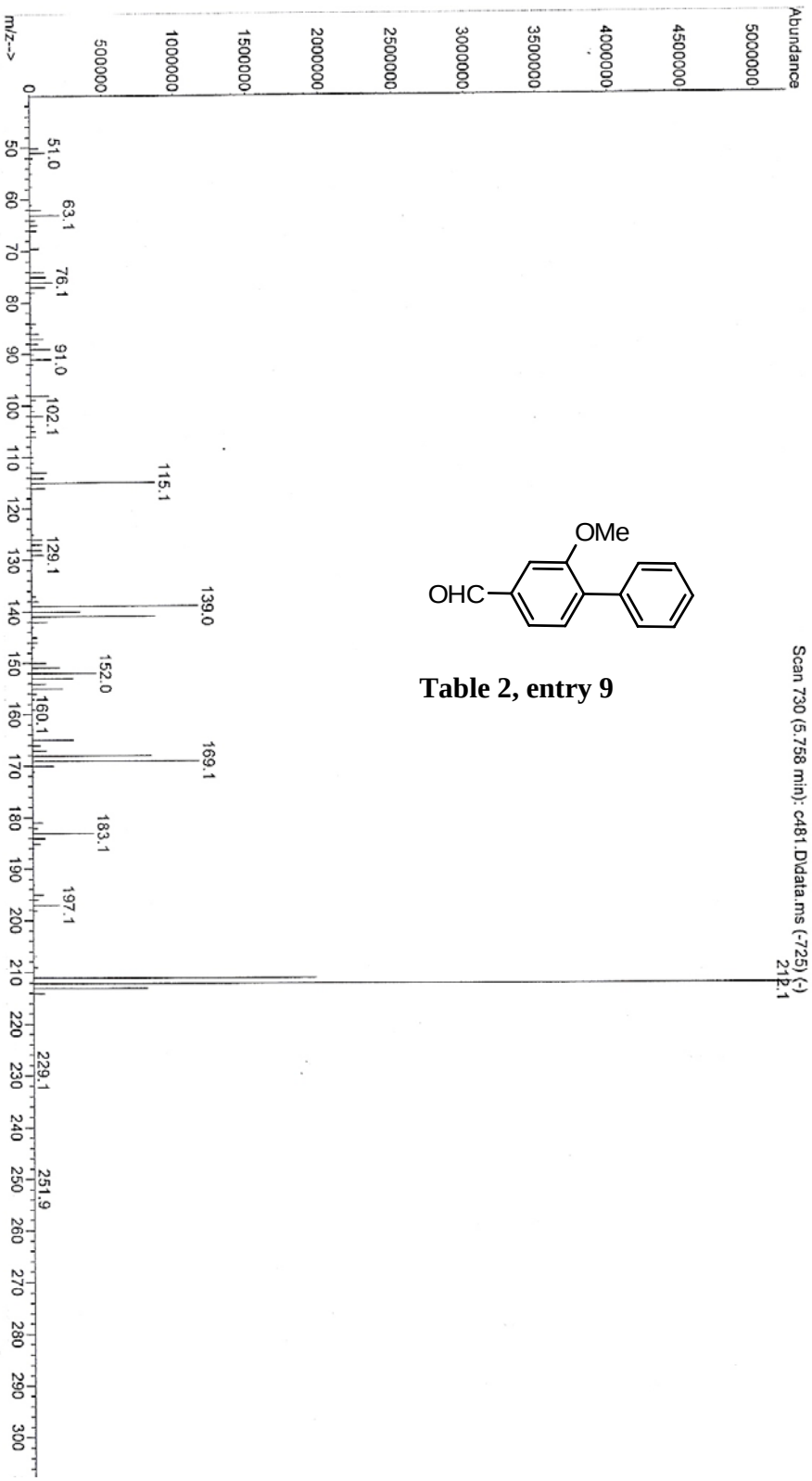


Table 2, entry 9

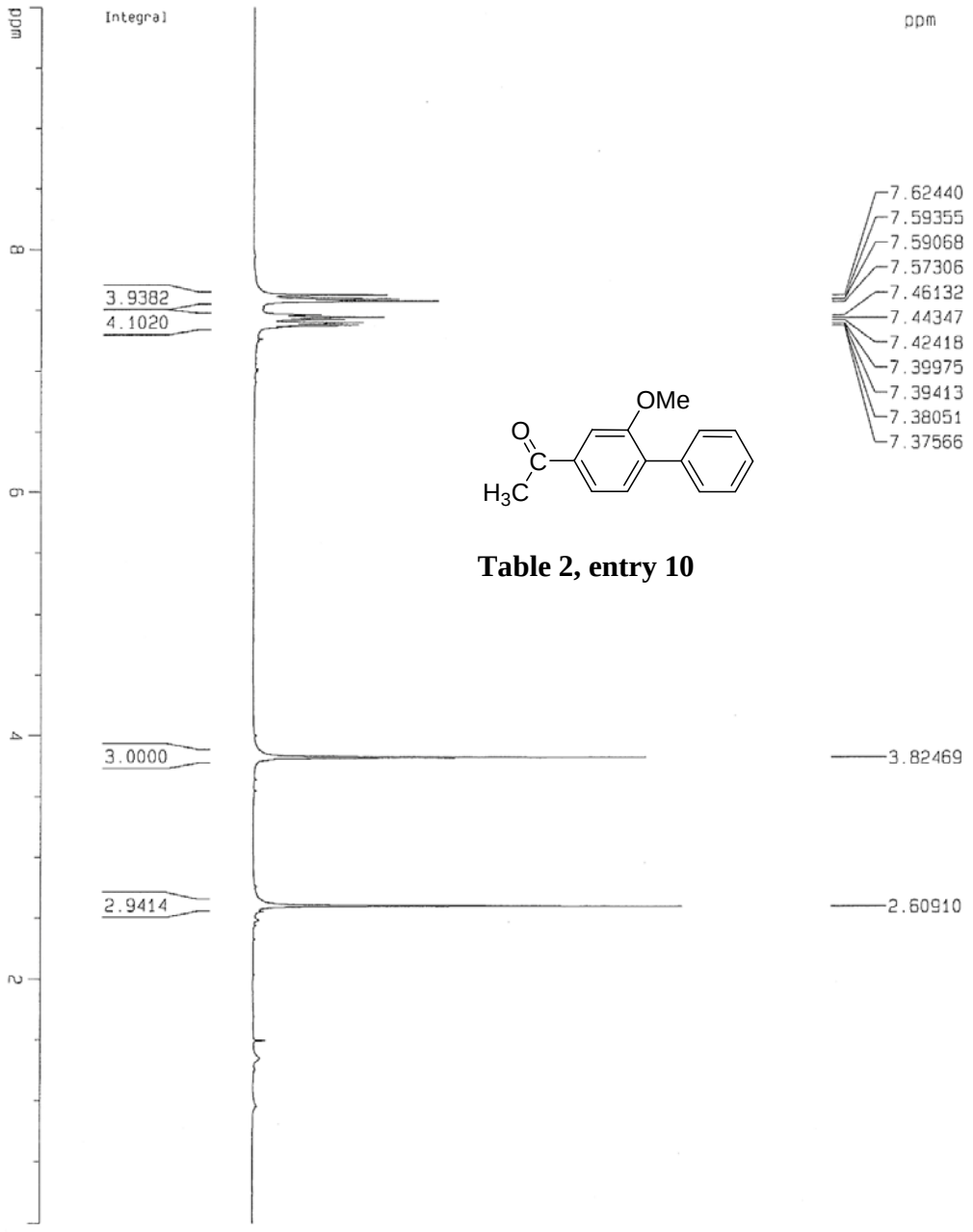


Table 2, entry 10


```
Current Data Parameters
NAME                  coupling
EXPNO                       94
PROCNO                       1

F2 - Acquisition Parameters
Date_               20070818
Time                   11.31
INSTRUM                dpx400
PROBHD     5 mm QNP 1H
PULPROG                zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                 4401.409 Hz
FIDRES              0.134320 Hz
AQ                  3.722494 sec
RG                        16
DW                 113.600 usec
DE                   4.50 usec
TE                   300.0 K
D1                  1.00000000 sec

======== CHANNEL f1 =====
NUC1                       1H
P1                      9.50 usec
PL1                     -6.00 dB
SFO1               400.1320006 MHz

F2 - Processing parameters
SI                        16384
SF                 400.1300077 MHz
WDW                        EM
SSB                         0
LB                      0.70 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      19.63 cm
F1P                  10.000 ppm
F1                   4001.30 Hz
F2P                  -0.000 ppm
F2                    -0.00 Hz
PPMCM                0.50930 ppm/c
HZCM               203.78769 Hz/cm
```

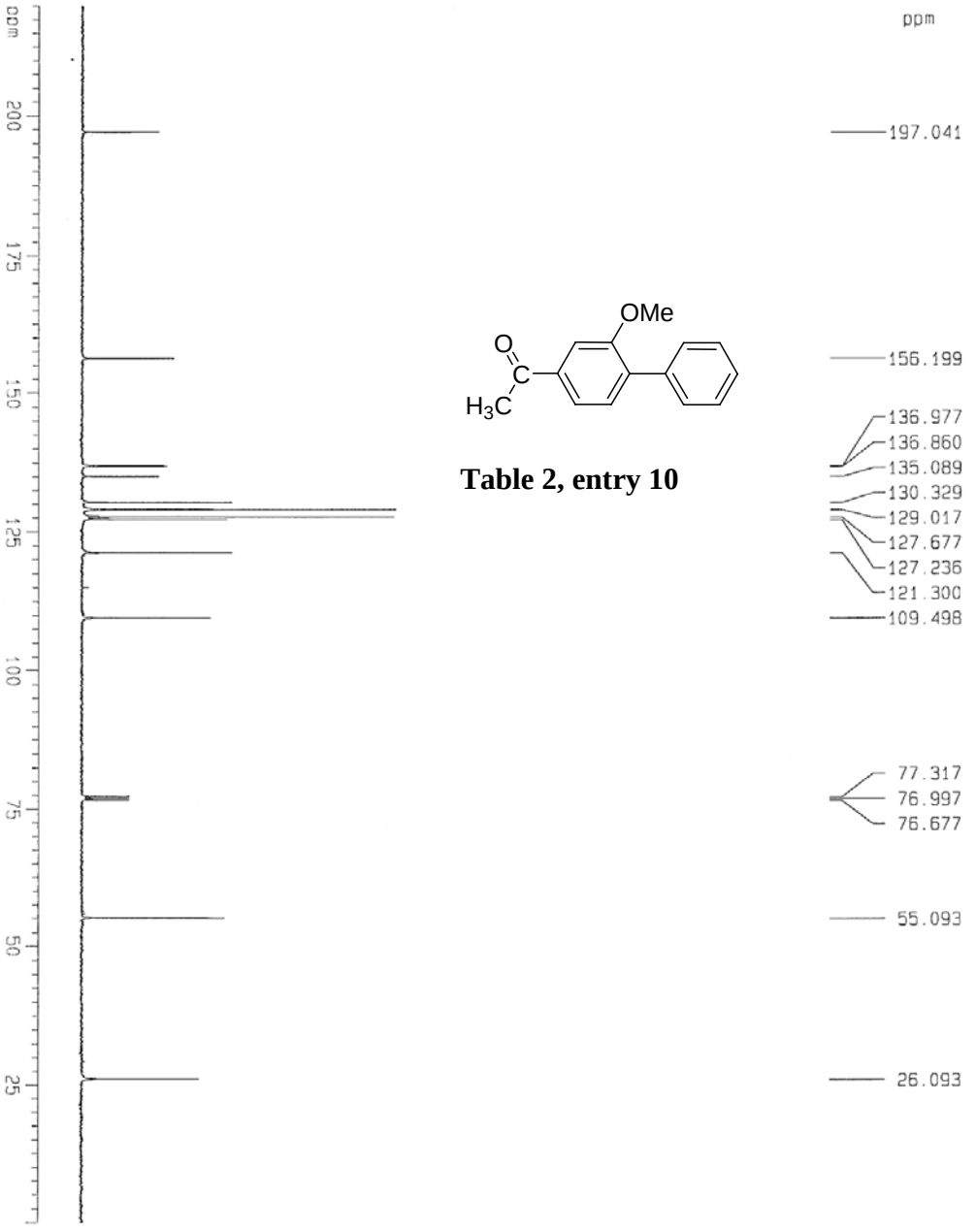


Table 2, entry 10

Current Data Parameters
NAME Ccoupling
EXPNO 54
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070818
Time 11.33
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zgdc30
TD 131072
SOLVENT CDCl3
NS 32
DS 0
SWH 25125.629 Hz
FIDRES 0.191693 Hz
AQ 2.6083827 sec
RG 8192
DW 19.900 usec
DE 4.50 usec
TE 300.0 K
D1 3.00000000 sec
d11 0.03000000 sec

========== CHANNEL f1 ======
NUC1 13C
P1 5.80 usec
PL1 -6.00 dB
SFO1 100.6231263 MHz

========== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 71.00 usec
PL2 120.00 dB
PL12 17.00 dB
SFO2 400.1326008 MHz

F2 - Processing parameters
SI 65536
SF 100.6128219 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 19.50 cm
F1P 220.000 ppm
F1 22134.82 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 11.28205 ppm/cm
HZCM 1135.11902 Hz/cm

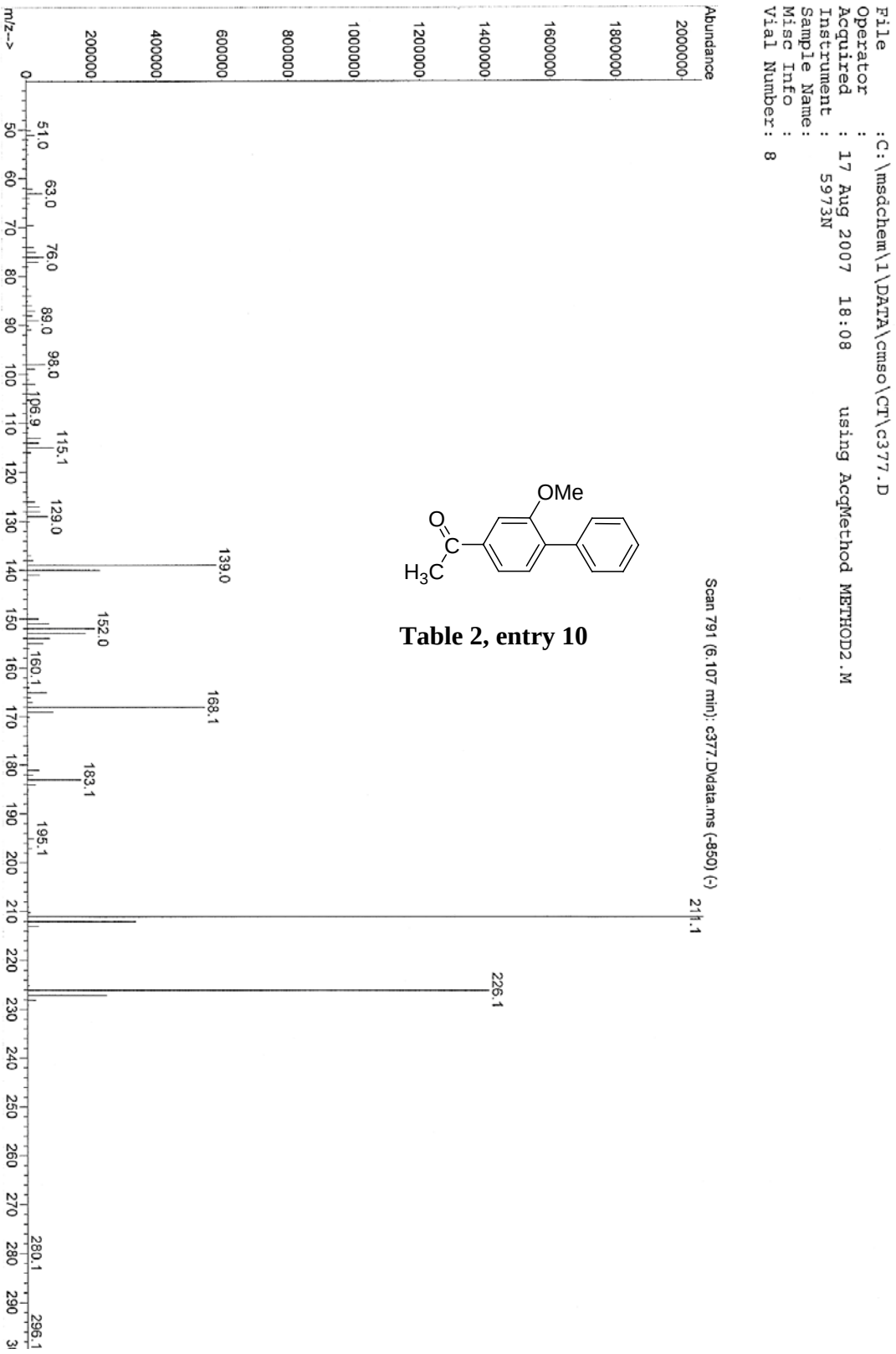


Table 2, entry 10

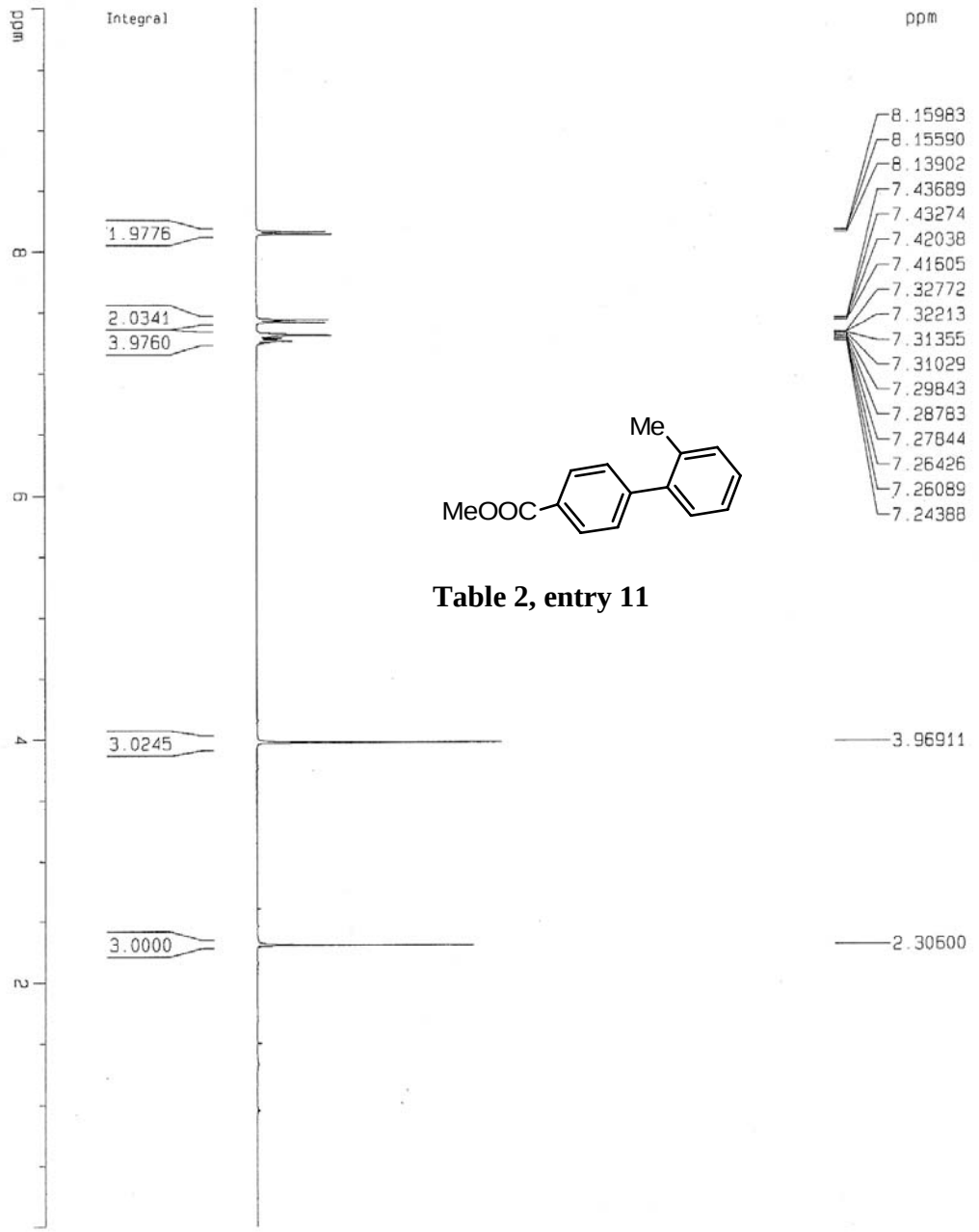


Table 2, entry 11


```
Current Data Parameters
NAME                  coupling
EXPNO                      139
PROCNO                       1

F2 - Acquisition Parameters
Date_              20071113
Time                   20.41
INSTRUM              dpx400
PROBHD     5 mm QNP  1H
PULPROG              zg30
TD                   32768
SOLVENT              CDCl3
NS                       4
DS                       0
SWH                4789.272 Hz
FIDRES            0.146157 Hz
AQ                3.4210291 sec
RG                      28.5
DW                 104.400 usec
DE                   4.50 usec
TE                    300.0 K
D1                1.00000000 sec

========== CHANNEL f1 ========
NUC1                     1H
P1                    9.50 usec
PL1                  -6.00 dB
SFO1          400.1320007 MHz

F2 - Processing parameters
SI                     16384
SF            400.1300089 MHz
WDW                       EM
SSB                        0
LB                  0.30 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                   19.63 cm
F1P                 10.000 ppm
F1                4001.30 Hz
F2P                -0.000 ppm
F2                   -0.00 Hz
PPMCM             0.50930 ppm/
HZCM            203.78769 Hz/c
```

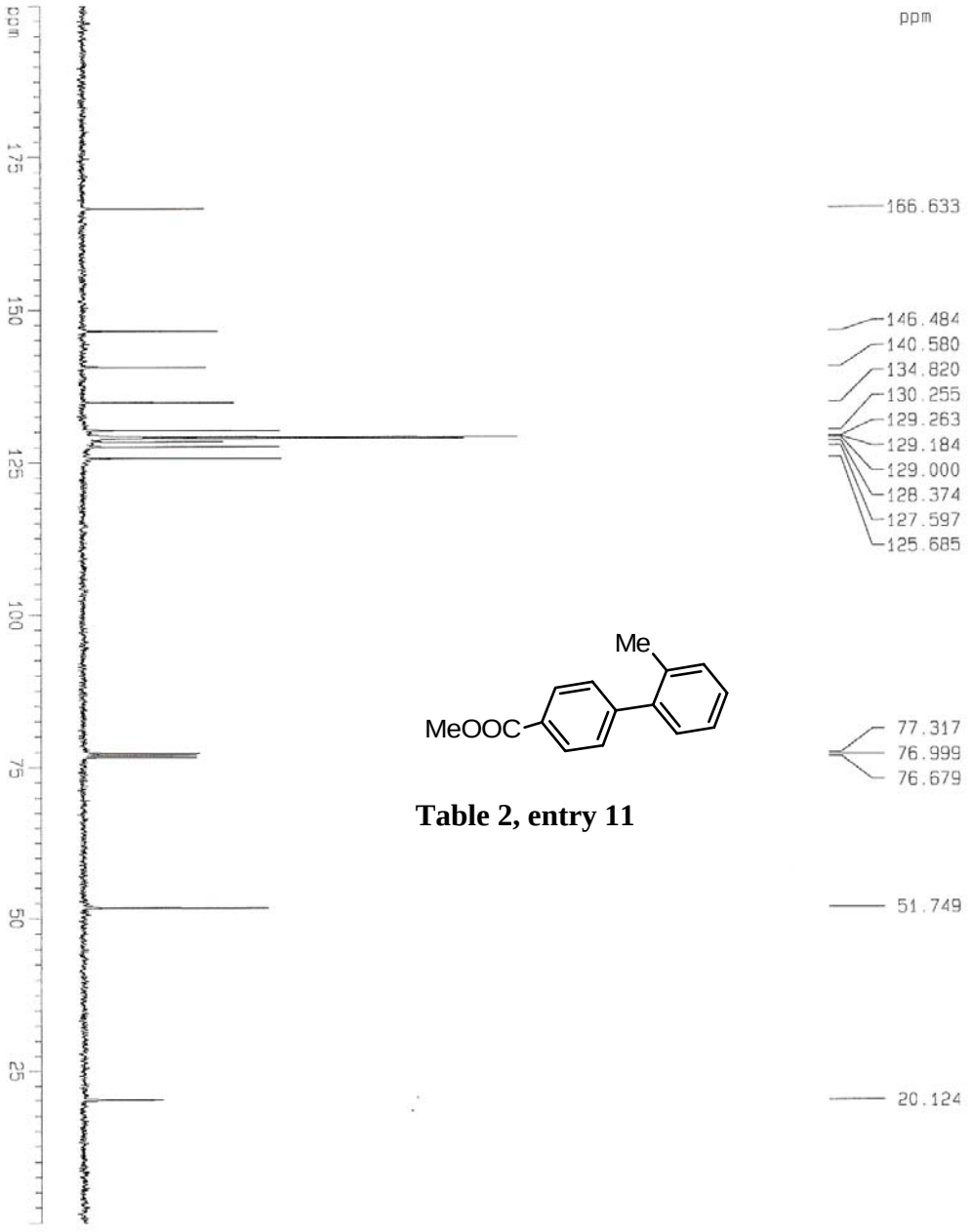


Table 2, entry 11


```
Current Data Parameters
NAME                  tcoupling
EXPNO                        93
PROCNO                        1

F2 - Acquisition Parameters
Date_               20071113
Time_                   20.43
INSTRUM                 qdx400
PROBHD     5 mm QNP 1H
PULPROG                 zgqc30
TD                      131072
SOLVENT                 CDCl3
NS                          16
DS                           0
SWH                 25125.629 Hz
FIDRES               0.191693 Hz
AQ                   2.6083827 sec
RG                        8192
DW                      19.900 usec
DE                       4.50 usec
TE                     300.0 K
D1                   3.00000000 sec
d11                  0.03000000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                        5.80 usec
PL1                      -6.00 dB
SFO1             100.6231263 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    71.00 usec
PL2                     120.00 dB
PL12                     17.00 dB
SF02             400.1326008 MHz

F2 - Processing parameters
SI                         65536
SF               100.6128020 MHz
WDW                           EM
SSB                            0
LB                          3.50 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         19.50 cm
F1P                     200.000 ppm
F1                     20122.56 Hz
F2P                     -0.000 ppm
F2                      -0.00 Hz
PPMCM                  10.25641 ppm/cm
HZCM                 1031.92615 Hz/cm
```

Table 2, entry 11

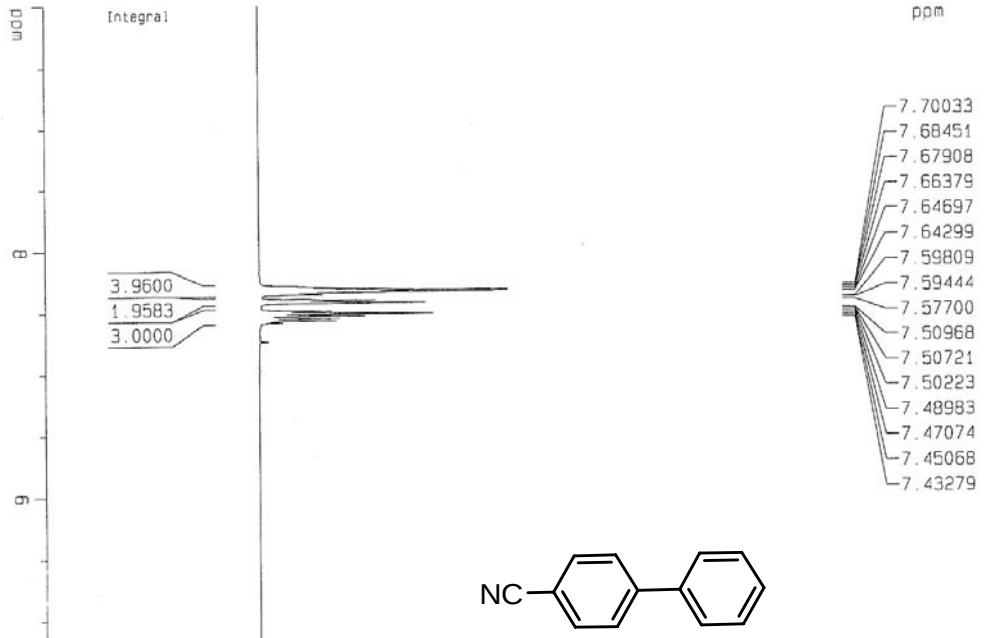


Table 2, entry 12

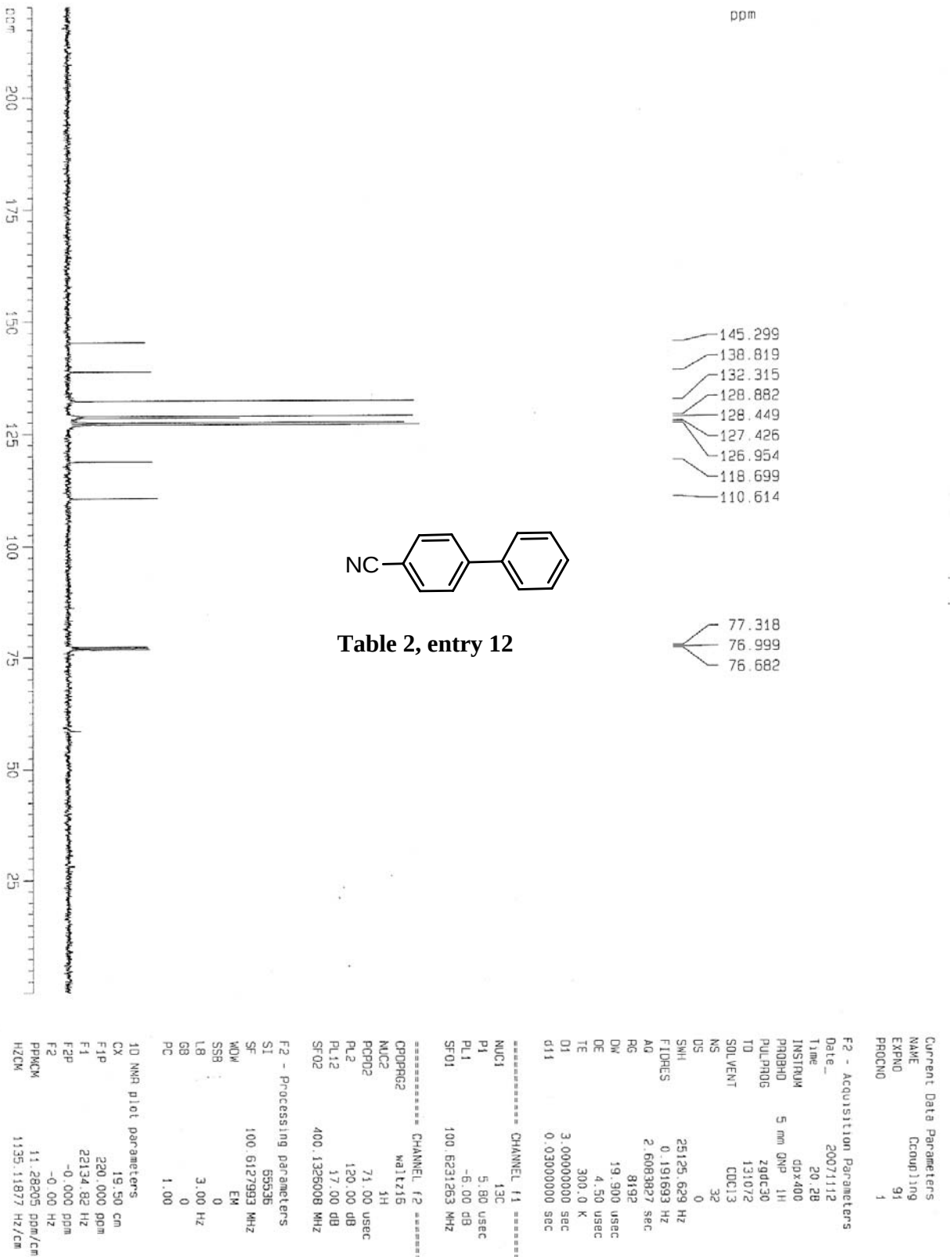


Table 2, entry 12

Current Data Parameters
NAME Ccoupling
EXPNO 91
PROCNO 1

F2 - Acquisition Parameters
Date_ 20071112
Time 20.28
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zgdc30
TD 131072
SOLVENT CDCl3
NS 32
DS 0
SWH 25125.629 Hz
FIDRES 0.191693 Hz
AQ 2.6083827 sec
RG 8192
DW 19.900 usec
DE 4.50 usec
TE 300.0 K
D1 3.00000000 sec
d11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 5.80 usec
PL1 -6.00 dB
SFO1 100.6231263 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 71.00 usec
PL2 120.00 dB
PL12 17.00 dB
SFO2 400.1326008 MHz

F2 - Processing parameters
SI 65536
SF 100.6127993 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 19.50 cm
F1P 220.000 ppm
F1 22134.82 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 11.28205 ppm/cm
HZCM 1135.11877 Hz/cm

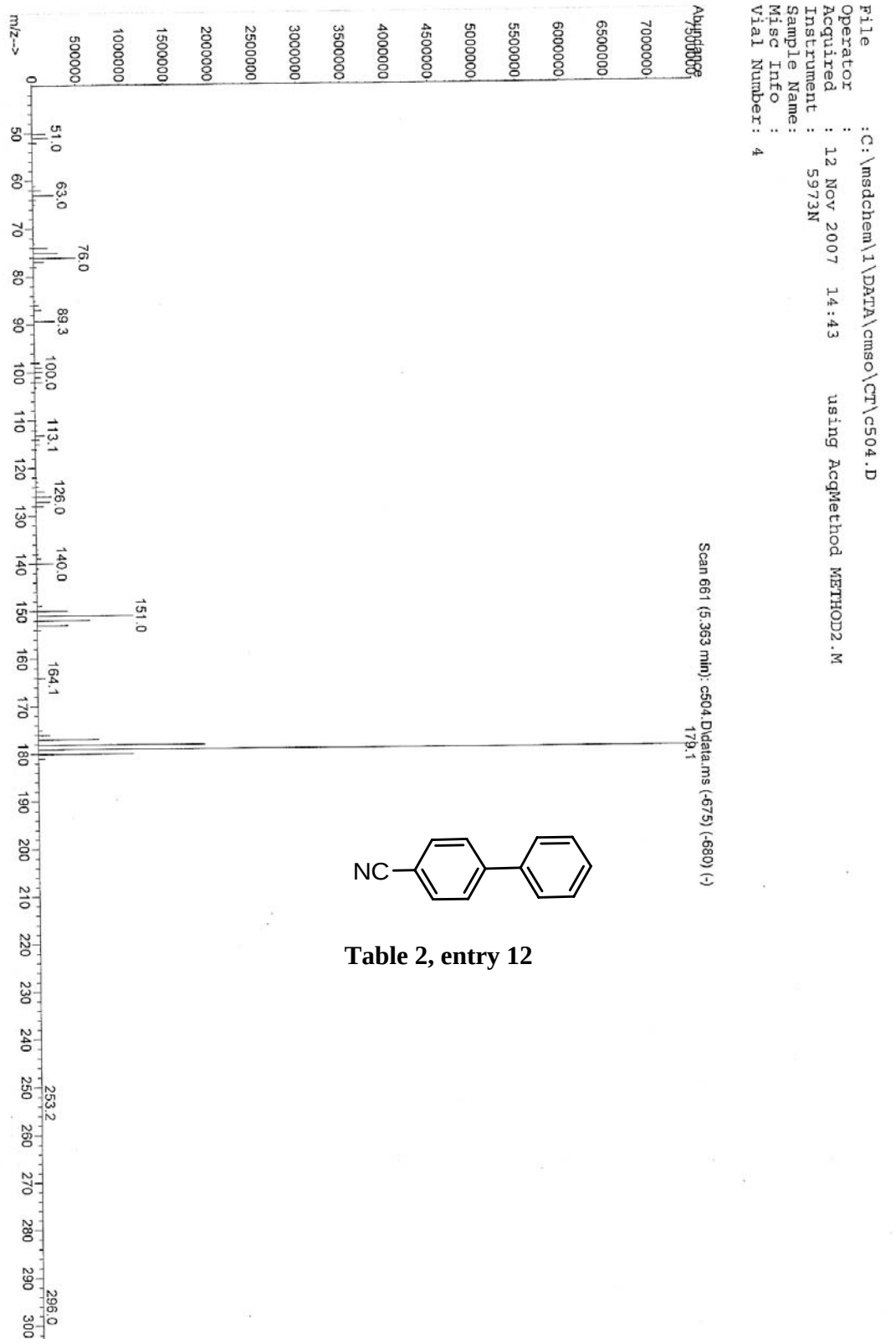


Table 2, entry 12

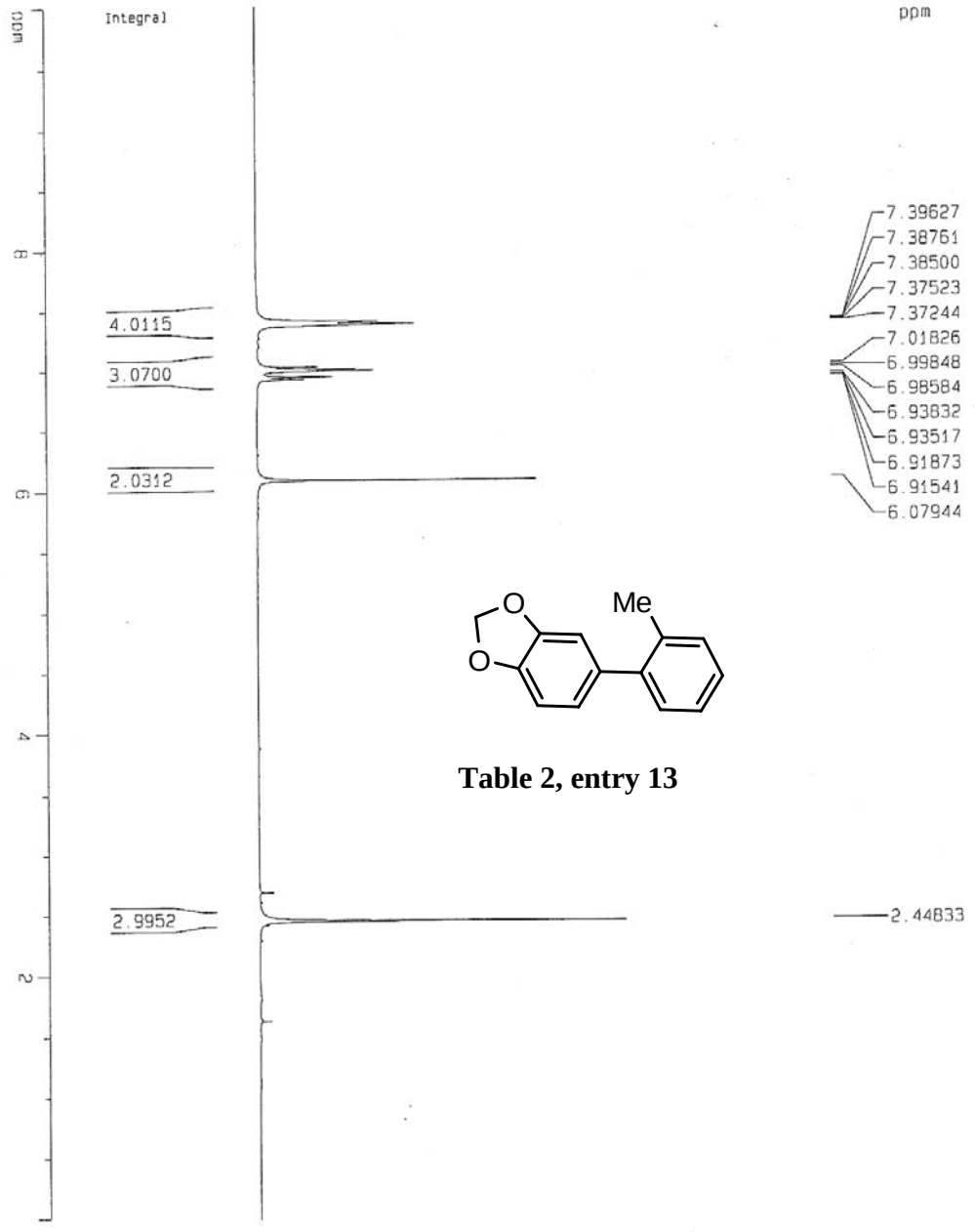


Table 2, entry 13


```
Current Data Parameters
NAME                  coupling
EXPNO                      126
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20071102
Time_                    14.43
INSTRUM                dpx400
PROBHD     5 mm QNP 1H
PULPROG                  zg30
TD                       32768
SOLVENT                  CDCl3
NS                          16
DS                           0
SWH                   4401.409 Hz
FIDRES                0.134320 Hz
AQ                    3.7224948 sec
RG                        20.2
DW                    113.600 usec
DE                      4.50 usec
TE                     300.0 K
D1                    1.00000000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      9.50 usec
PL1                     -6.00 dB
SFO1           400.1320006 MHz

F2 - Processing parameters
SI                        16384
SF                400.1300085 MHz
WDW                          EM
SSB                           0
LB                     0.40 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                     19.63 cm
F1P                   10.000 ppm
F1                  4001.30 Hz
F2P                   -0.000 ppm
F2                    -0.00 Hz
PPMCM                 0.50930 ppm/cm
HZCM                203.78759 Hz/cm
```

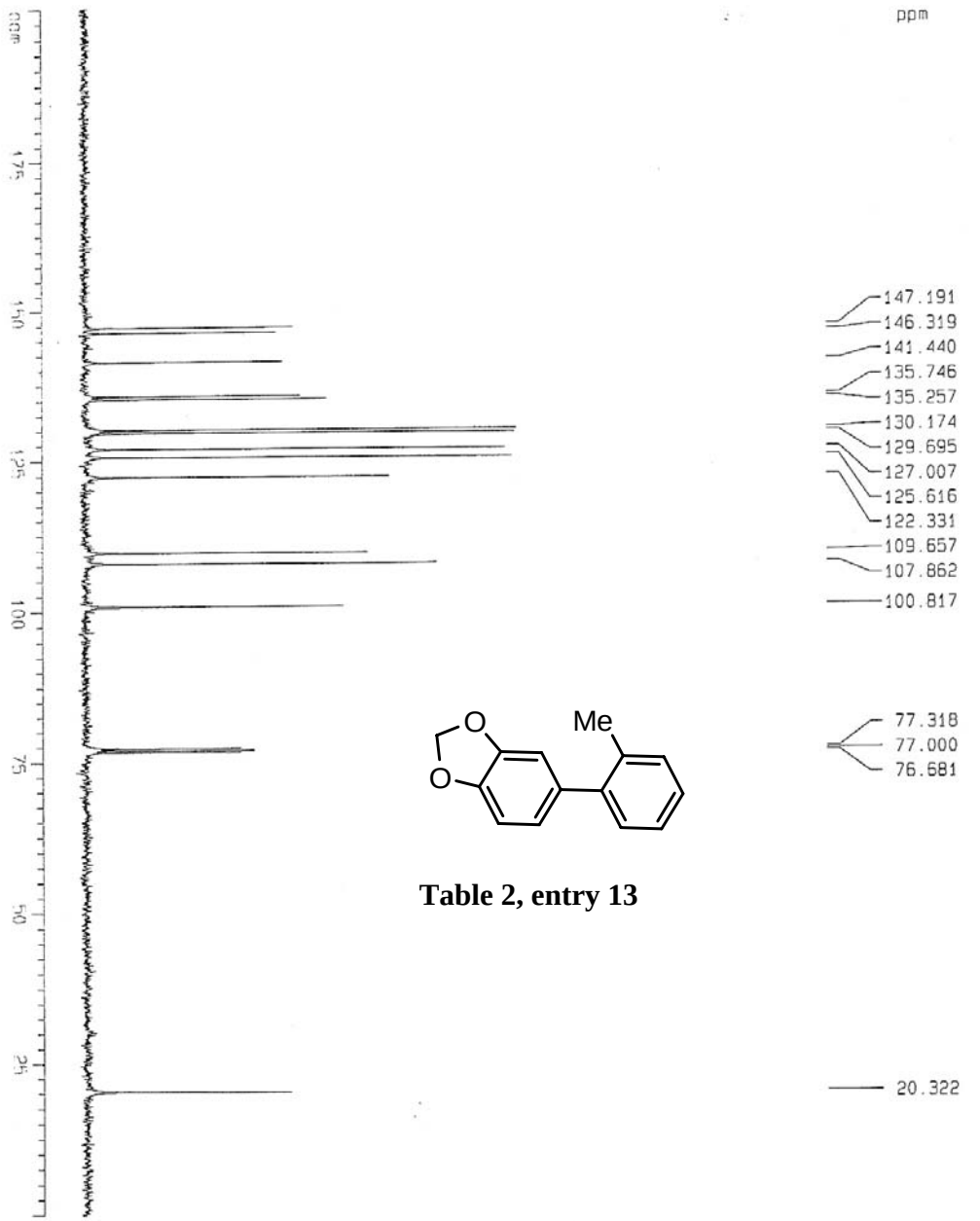


Table 2, entry 13


```
Current Data Parameters
NAME                  Ccoupling
EXPNO                        B1
PROCNO                        1

F2 - Acquisition Parameters
Date_                   20071102
Time                       14.46
INSTRUM                  dpx400
PROBHD     5 mm QNP  1H
PULPROG                 zgdc30
TD                      131072
SOLVENT                   CDCl3
NS                           32
DS                            0
SWH                  25125.629 Hz
FIDRES                0.191693 Hz
AQ                    2.6083827 sec
RG                         8192
DW                       19.900 usec
DE                        4.50 usec
TE                       300.0 K
D1                    3.00000000 sec
D11                   0.03000000 sec

========== CHANNEL f1 ==========
NUC1                        13C
P1                         5.80 usec
PL1                       -6.00 dB
SFO1                100.6231263 MHz

========== CHANNEL f2 ==========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                     71.00 usec
PL2                      120.00 dB
PL12                      17.00 dB
SFO2                400.1326009 MHz

F2 - Processing parameters
SI                         65536
SF                  100.6128032 MHz
WDW                           EM
SSB                            0
LB                         4.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                        19.50 cm
F1P                      200.000 ppm
F1                     20122.56 Hz
F2P                      -0.000 ppm
F2                       -0.00 Hz
PPMCM                  10.25641 ppm/cm
HZCM                1031.92615 Hz/cm
```

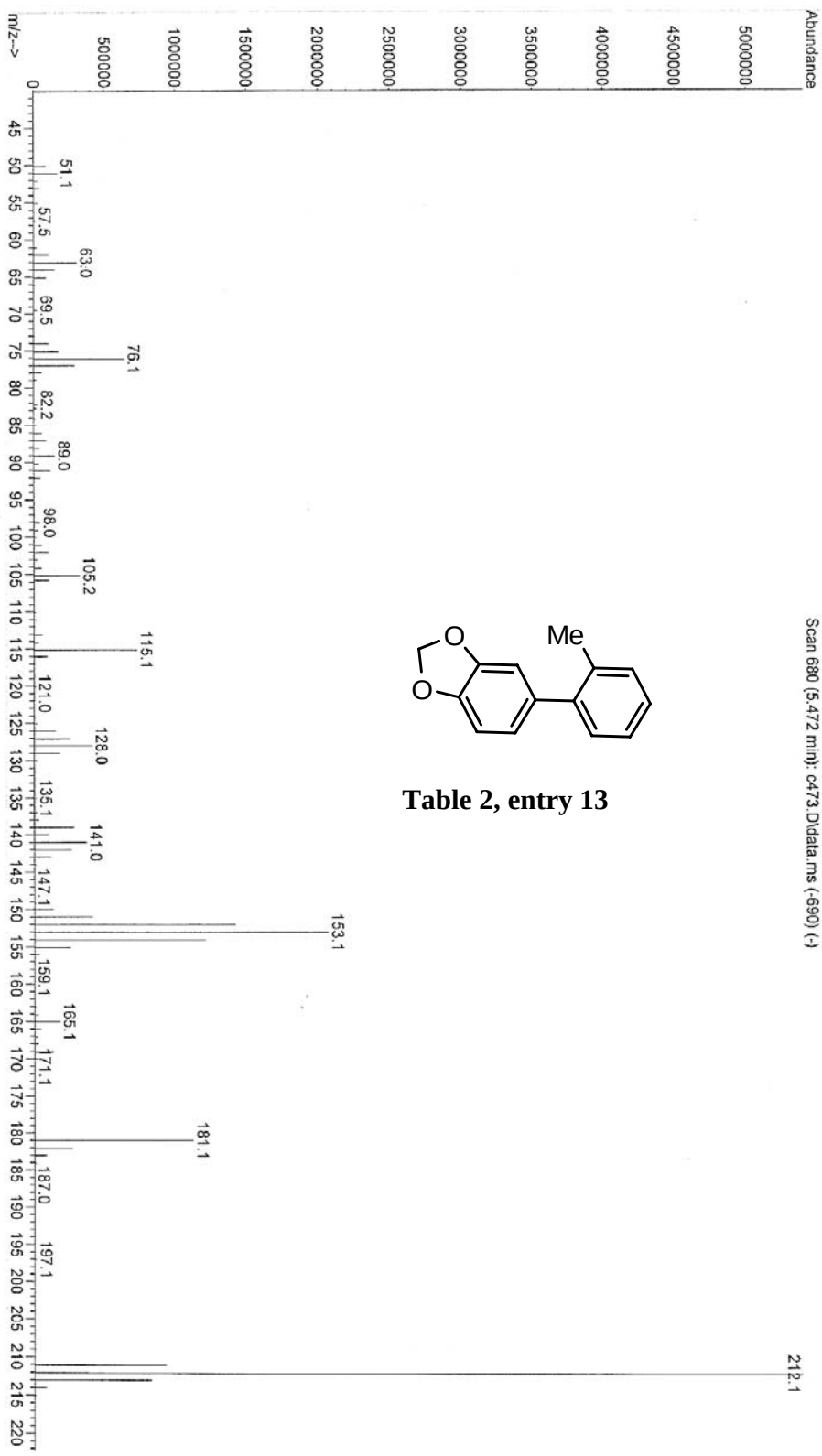


Table 2, entry 13

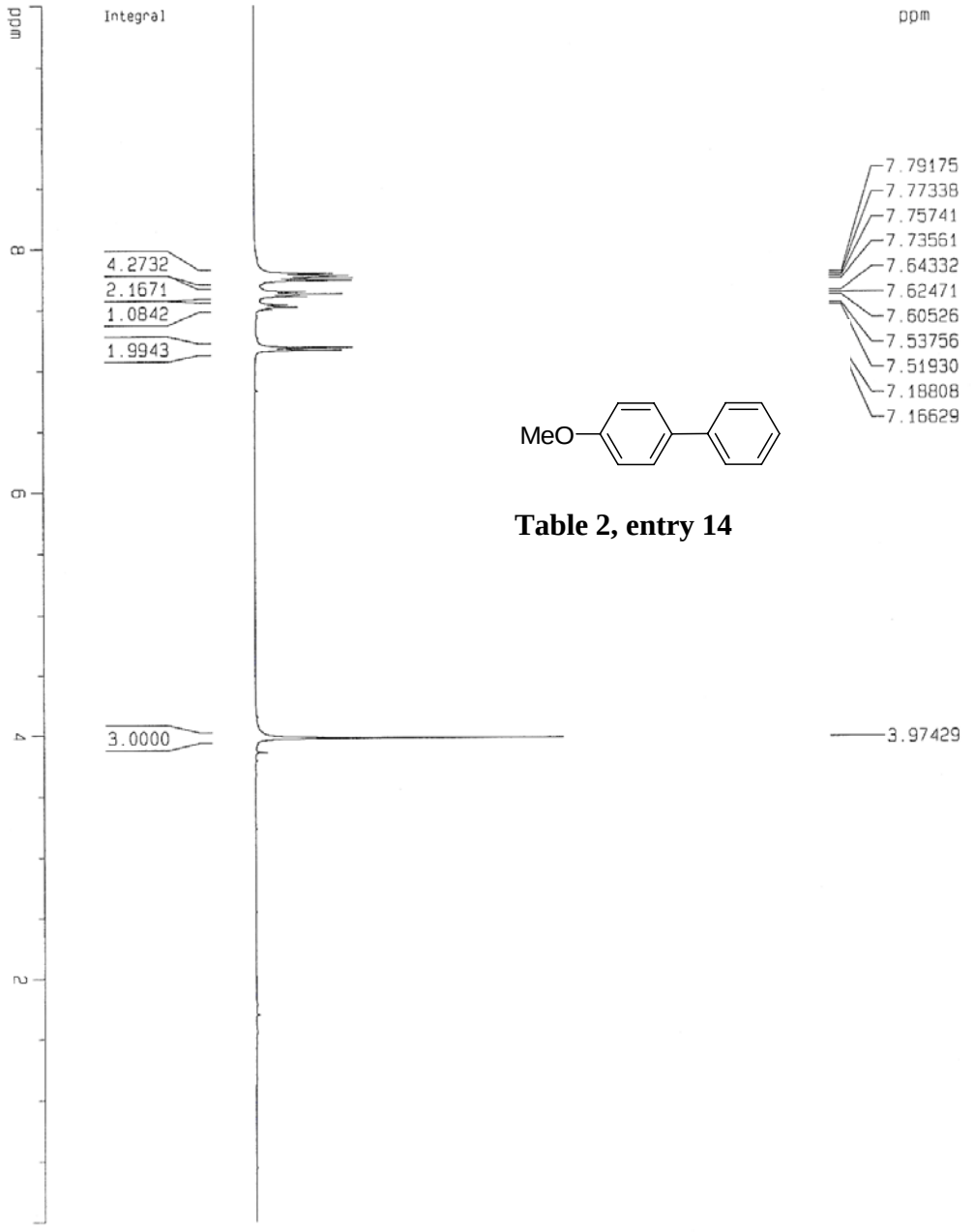


Table 2, entry 14


```
Current Data Parameters
NAME                  coupling
EXPNO                       96
PROCNO                       1

F2 - Acquisition Parameters
Date_              20070822
Time                   17.09
INSTRUM             dpx400
PROBHD     5 mm QNP  1H
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH              4401.409 Hz
FIDRES              0.134320 Hz
AQ               3.7224948 sec
RG                    25.4
DW                 113.600 usec
DE                   4.50 usec
TE                 300.0 K
D1                 1.00000000 sec

======== CHANNEL f1 ======
NUC1                  1H
P1                  9.50 usec
PL1                -6.00 dB
SFO1         400.1320006 MHz

F2 - Processing parameters
SI                 16384
SF           400.1300077 MHz
WDW                   EM
SSB                    0
LB                  0.70 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 19.63 cm
F1P                10.000 ppm
F1                4001.30 Hz
F2P                -0.000 ppm
F2                 -0.00 Hz
PPMCM              0.50930 ppm/cm
HZCM          203.78769 Hz/cm
```

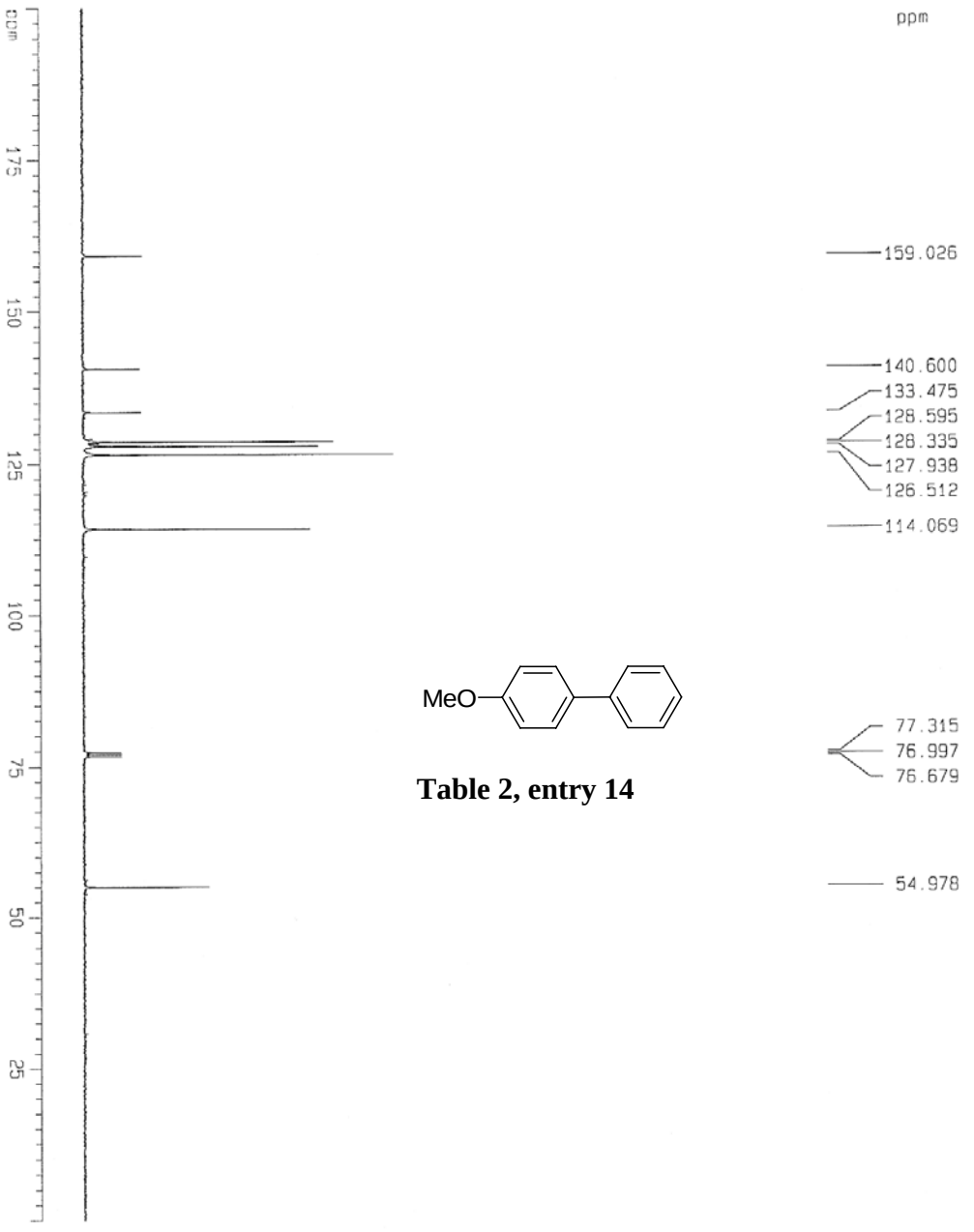


Table 2, entry 14


```
Current Data Parameters
NAME                 Ccoupling
EXPNO                      56
PROCNO                      1

F2 - Acquisition Parameters
Date_               20070822
Time                    17.11
INSTRUM                 dpx400
PROBHD     5 mm QNP 1H
PULPROG             zgdc30
TD                  131072
SOLVENT             CDCl3
NS                      32
DS                       0
SWH                 25125.629 Hz
FIDRES                0.191693 Hz
AQ                  2.6083827 sec
RG                       8192
DW                     19.900 usec
DE                      4.50 usec
TE                     300.0 K
D1                  3.00000000 sec
d11                 0.03000000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                       5.80 usec
PL1                     -6.00 dB
SF01             100.6231263 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    71.00 usec
PL2                     120.00 dB
PL12                     17.00 dB
SFO2             400.1326008 MHz

F2 - Processing parameters
SI                       65536
SF               100.6128128 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       19.50 cm
F1P                     200.000 ppm
F1                    20122.56 Hz
F2P                    -0.000 ppm
F2                     -0.00 Hz
PPMCM                 10.25641 ppm/cm
HZCM              1031.92639 Hz/cm
```

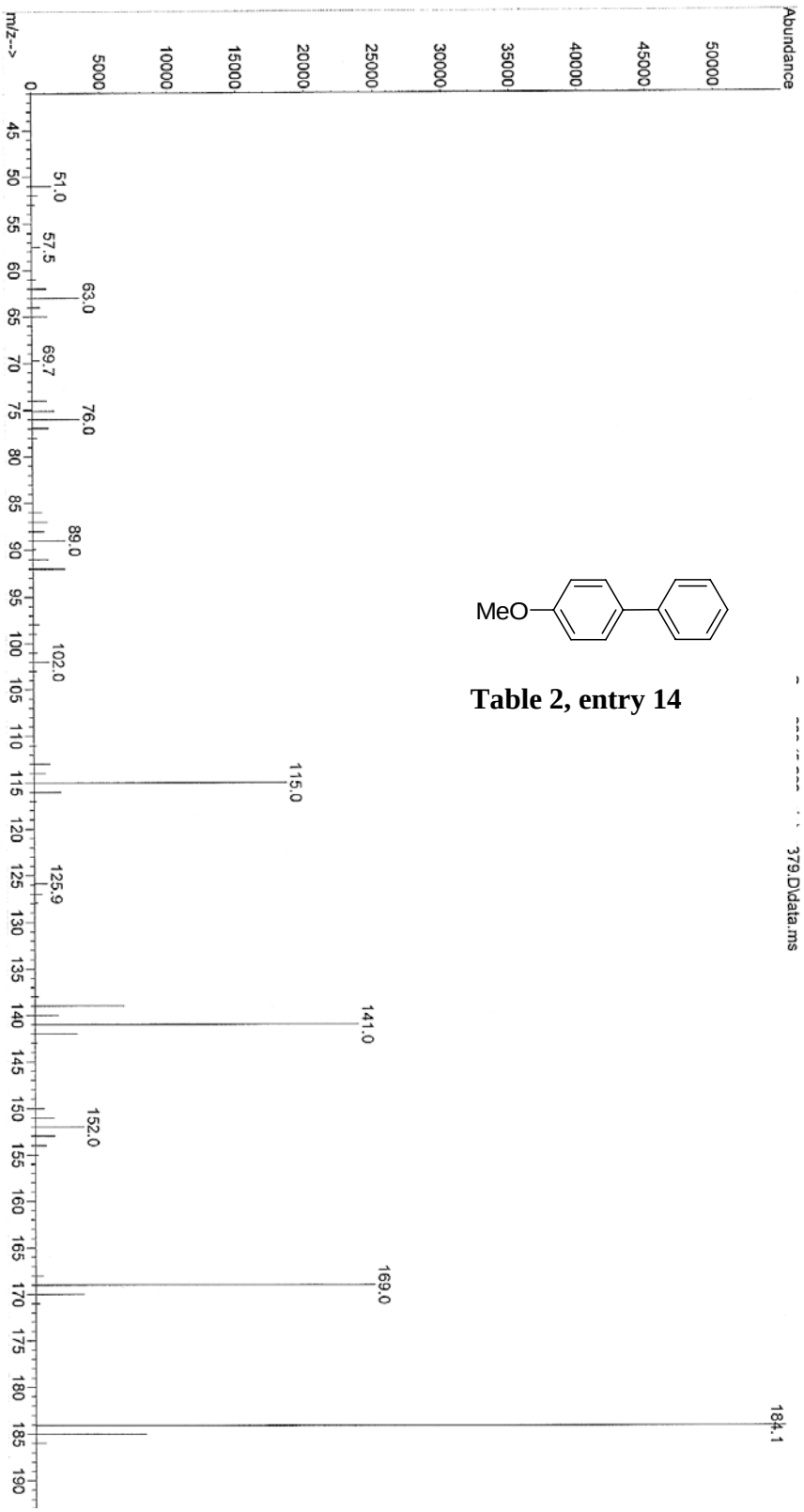
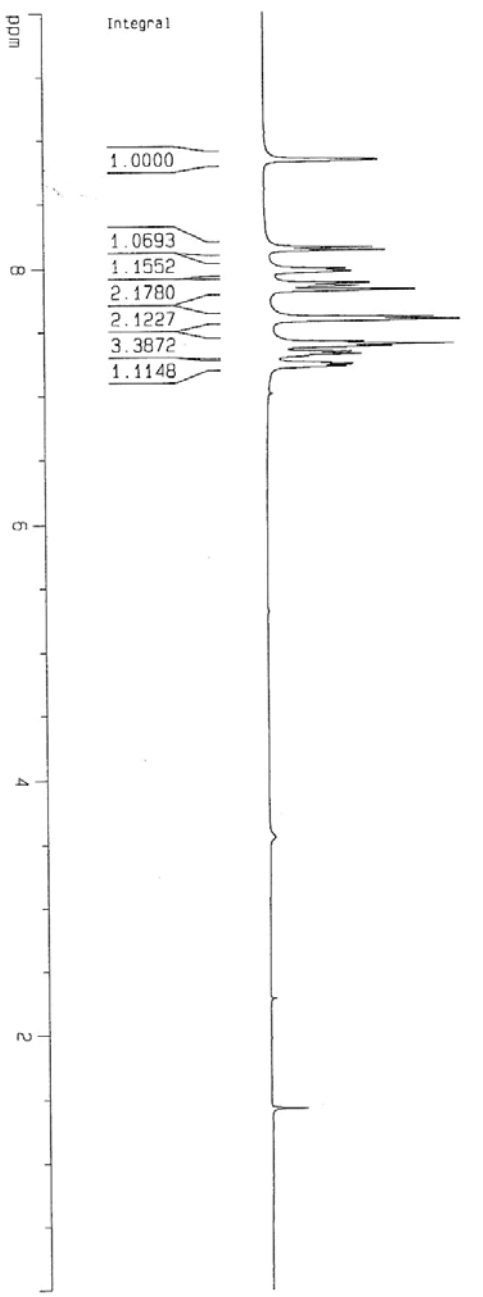
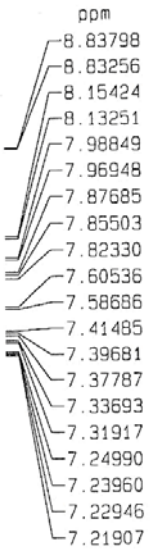


Table 2, entry 14





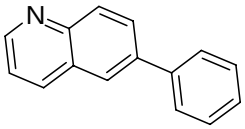


Table 3, entry 1

```
Current Data Parameters
NAME                  coupling
EXPNO                       86
PROCNO                       1

F2 - Acquisition Parameters
Date_              20070629
Time                   18.53
INSTRUM                dpx400
PROBHD     5 mm QNP  1H
PULPROG                zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                4401.409 Hz
FIDRES             0.134320 Hz
AQ                 3.722494 sec
RG                      35.9
DW                 113.600 usec
DE                   4.50 usec
TE                    300.0 K
D1                 1.00000000 sec

============== CHANNEL f1 ======
NUC1                      1H
P1                      9.50 usec
PL1                    -6.00 dB
SFO1           400.1320006 MHz

F2 - Processing parameters
SI                        16384
SF             400.1300077 MHz
WDW                          EM
SSB                           0
LB                         0.70 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                        19.63 cm
F1P                   10.000 ppm
F1                  4001.30 Hz
F2P                   -0.000 ppm
F2                     -0.00 Hz
PPMCM                 0.50930 ppm/cm
HZCM                203.78769 Hz/cm
```

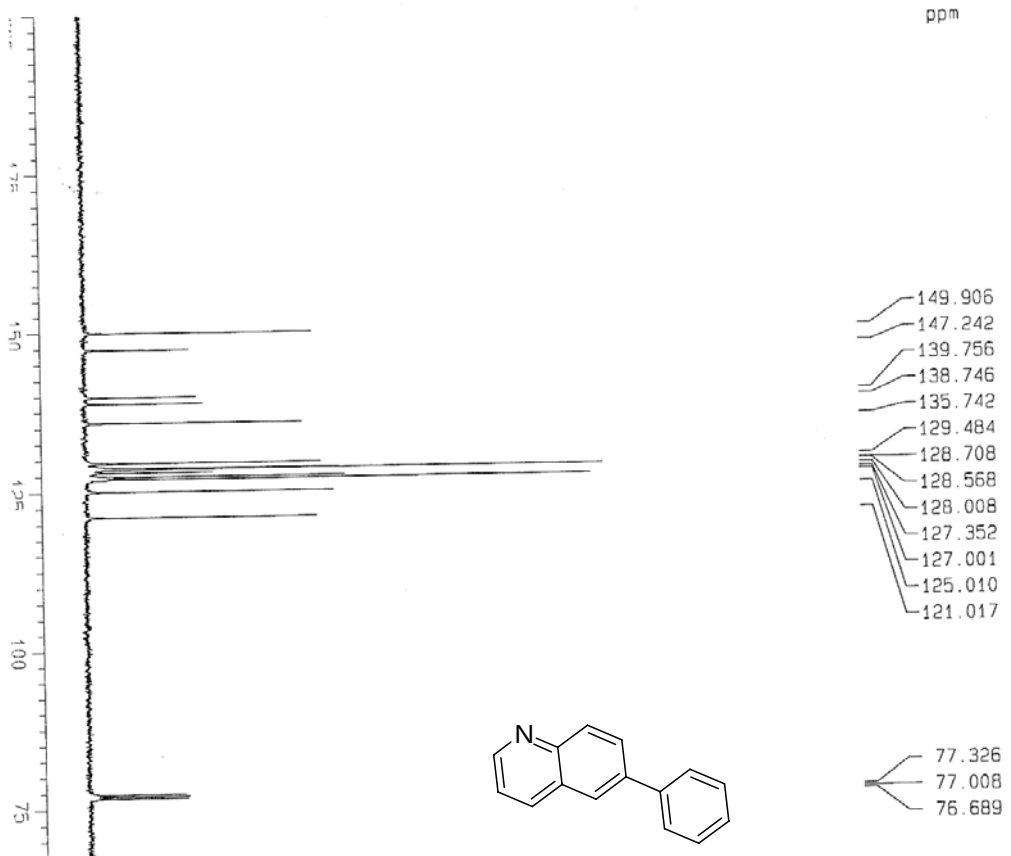


Table 3, entry 1


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

F2 - Acquisition Parameters
Date_               20070629
Time                    18.55
INSTRUM                dpx400
PROBHD     5 mm QNP  1H
PULPROG                zgdc30
TD                     131072
SOLVENT                CDCl3
NS                         32
DS                          0
SWH                 25125.629 Hz
FIDRES                0.191693 Hz
AQ                    2.6083827 sec
RG                       8192
DW                    19.900 usec
DE                     4.50 usec
TE                    300.0 K
D1                    3.00000000 sec
d11                   0.03000000 sec

========== CHANNEL f1 ====
NUC1                       13C
P1                        5.80 usec
PL1                      -6.00 dB
SFO1                100.6231263 MHz

========== CHANNEL f2 ====
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                    71.00 usec
PL2                     120.00 dB
PL12                     17.00 dB
SFO2                400.1326008 MHz

F2 - Processing parameters
SI                         65536
SF                  100.6128089 MHz
WDW                           EM
SSB                            0
LB                          3.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         19.50 cm
F1P                      200.000 ppm
F2P                    20122.56 Hz
F1                       -0.000 ppm
F2                        -0.00 Hz
PPMCM                   10.25641 ppm
HZCM                 1031.92627 Hz/cm
```

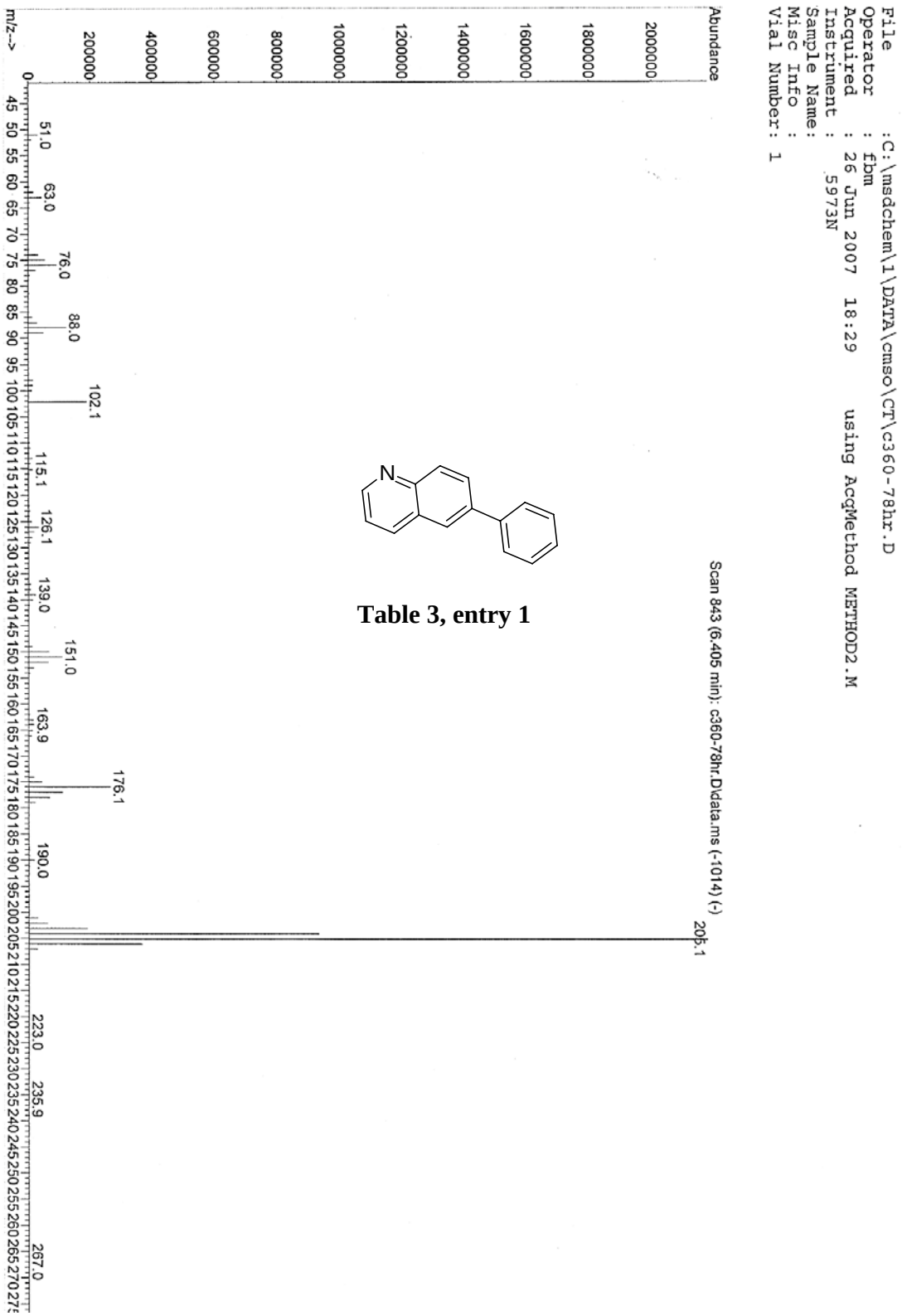


Table 3, entry 1

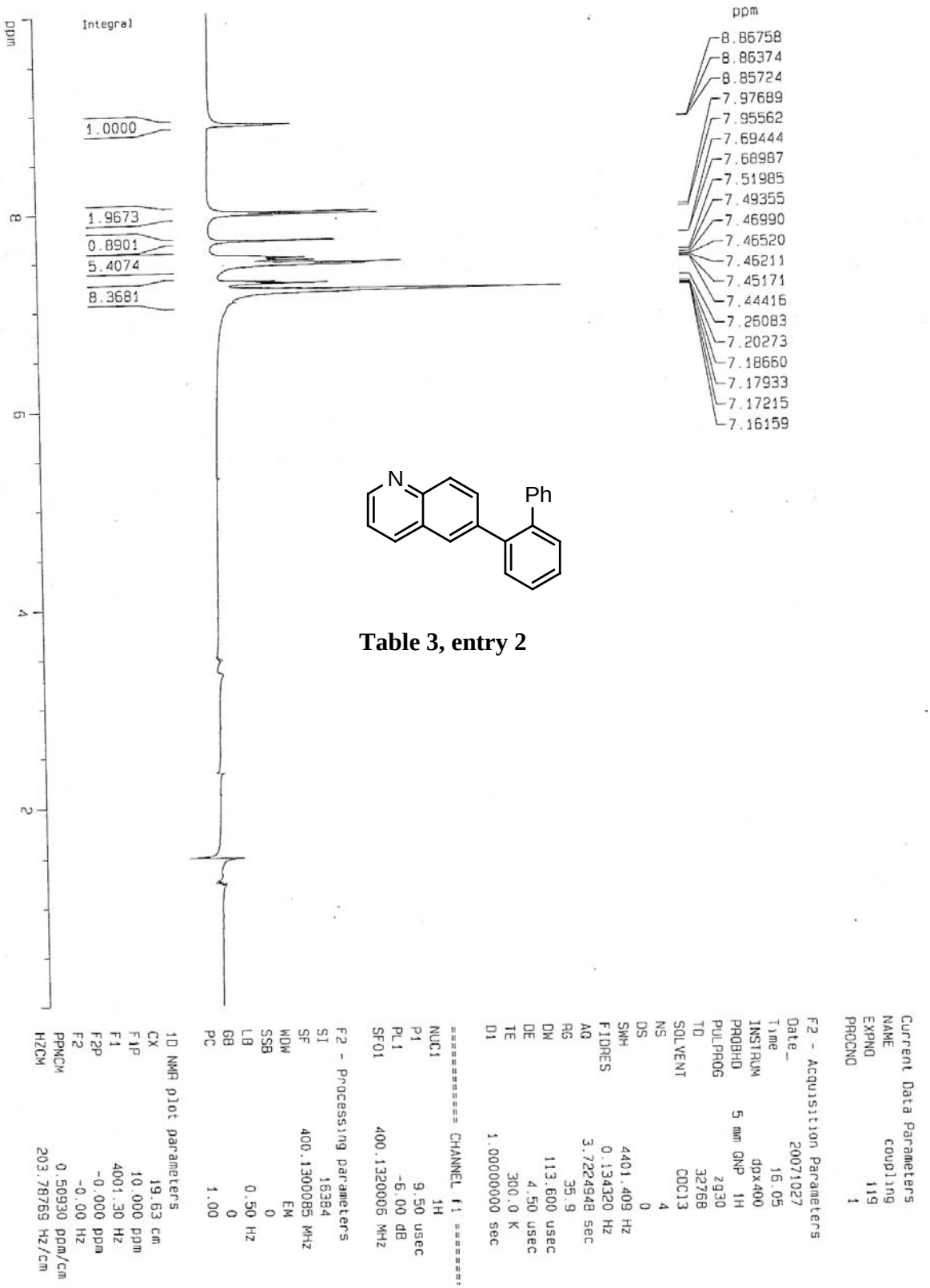


Table 3, entry 2

```
Current Data Parameters
NAME                  coupling
EXPNO                      119
PROCNO                       1

F2 - Acquisition Parameters
Date_              20071027
Time                   16.05
INSTRUM                dpx400
PROBHD      5 mm QNP  1H
PULPROG                zg30
TD                     32768
SOLVENT                CDCl3
NS                         4
DS                         0
SWH                 4401.409 Hz
FIDRES                 0.134320 Hz
AQ                  3.722948 sec
RG                        35.9
DW                    113.600 usec
DE                      4.50 usec
TE                     300.0 K
D1                  1.00000000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                       9.50 usec
PL1                     -6.00 dB
SF01               400.1320006 MHz

F2 - Processing parameters
SI                        16384
SF                 400.1300085 MHz
WDW                        EM
SSB                         0
LB                       0.50 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      19.63 cm
F1P                   10.000 ppm
F1                   4001.30 Hz
F2P                   -0.000 ppm
F2                    -0.00 Hz
PPMCM                 0.50930 ppm/cm
HZCM                203.78769 Hz/cm
```

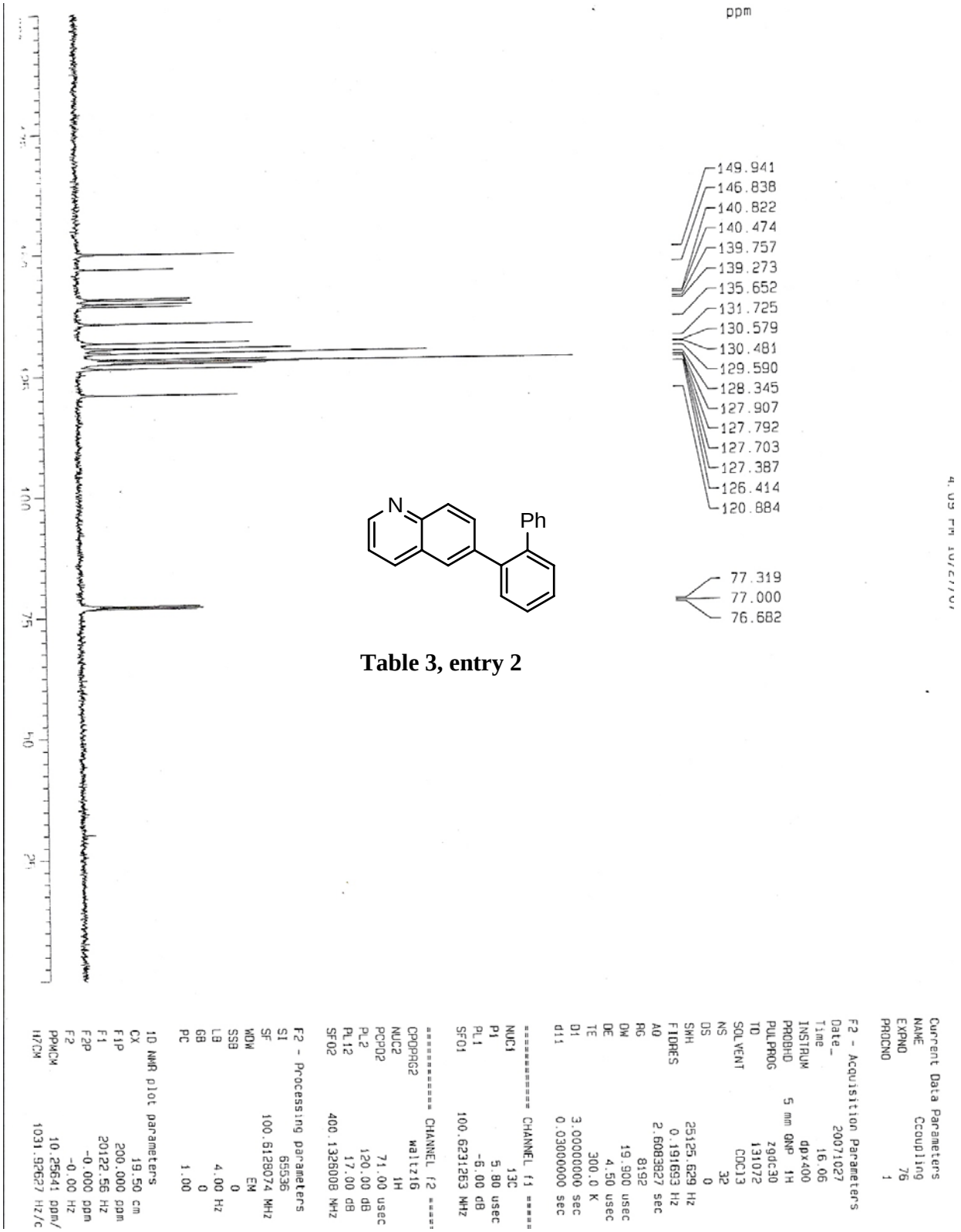


Table 3, entry 2

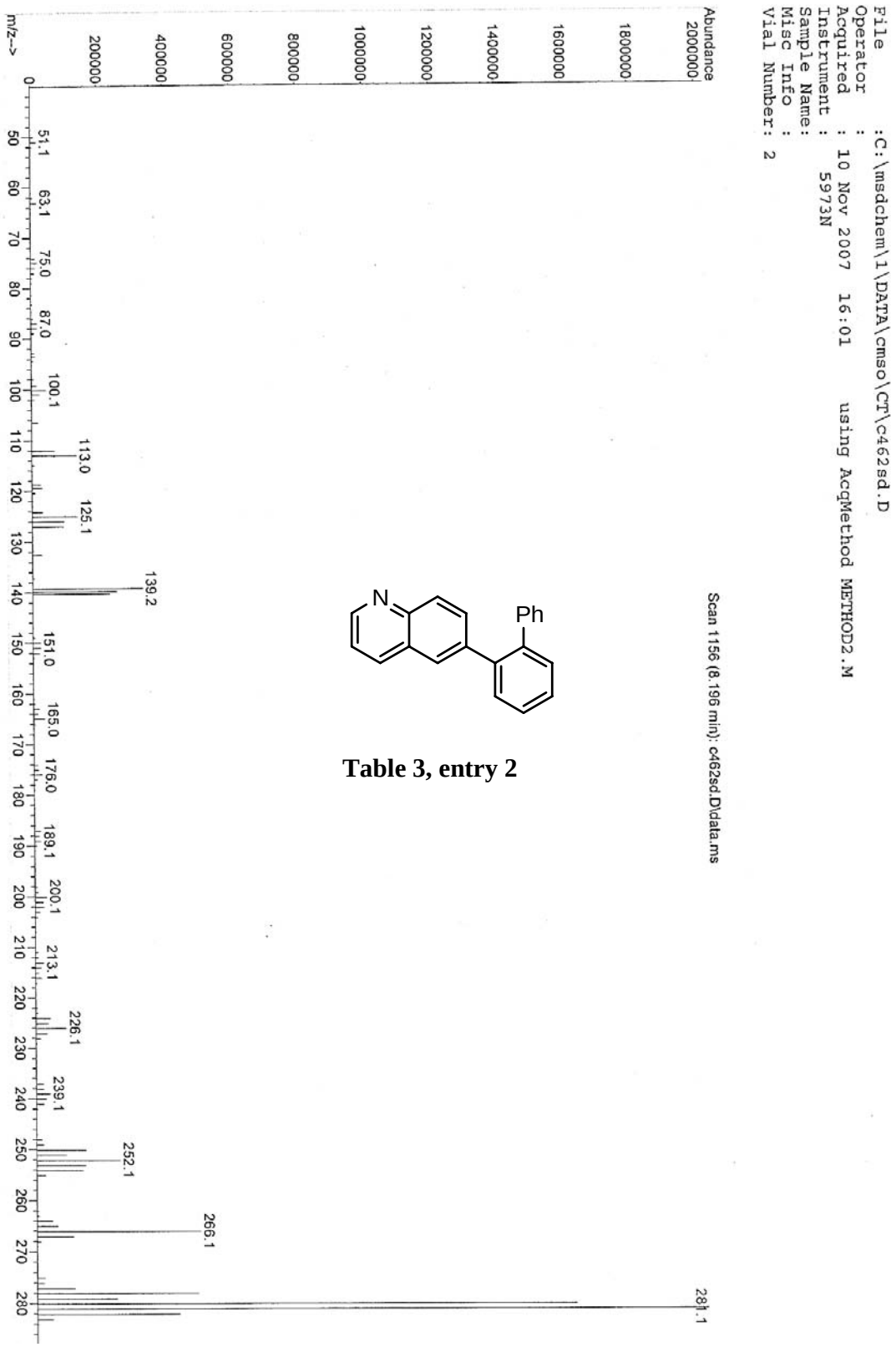


Table 3, entry 2

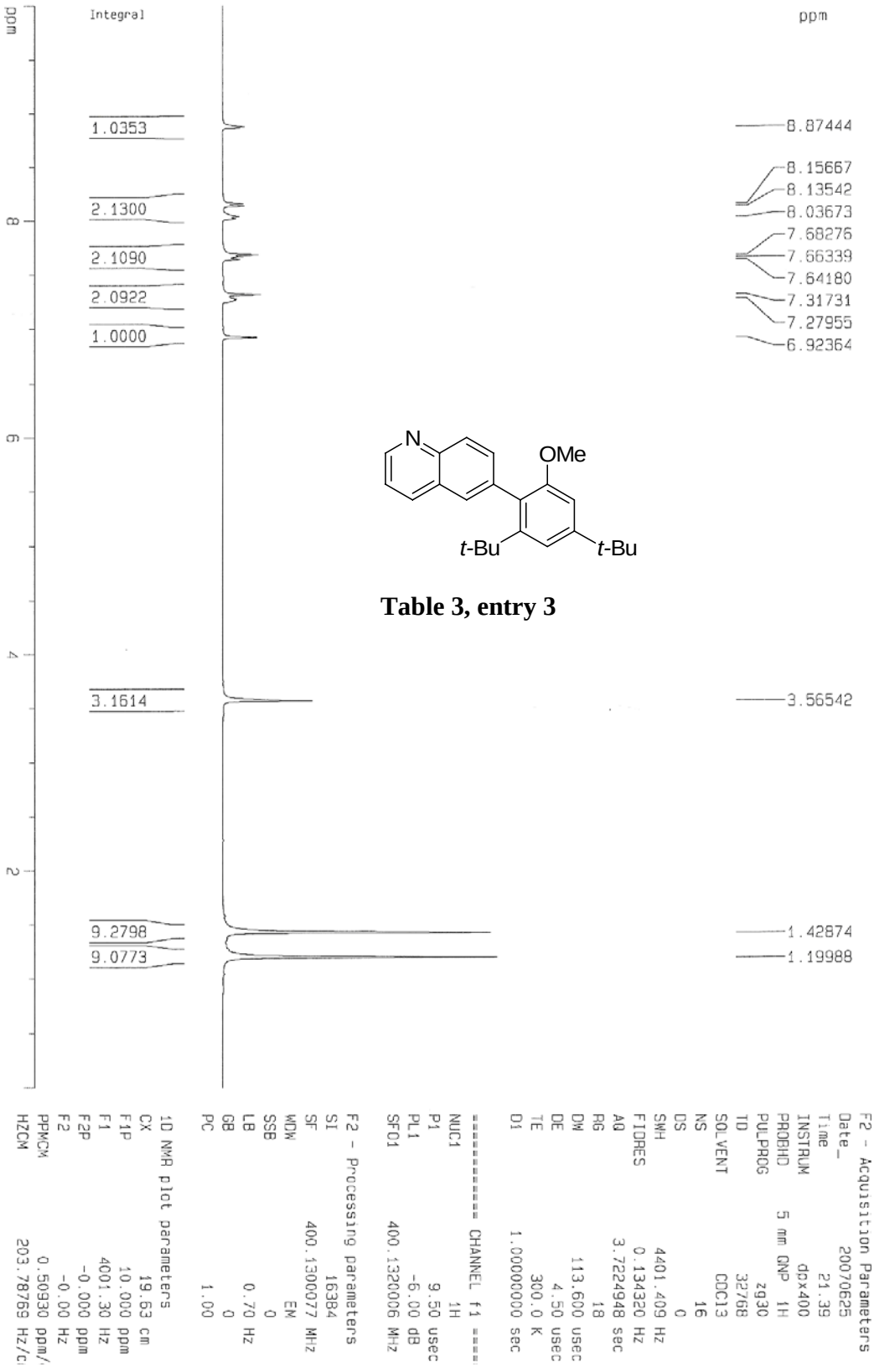


Table 3, entry 3

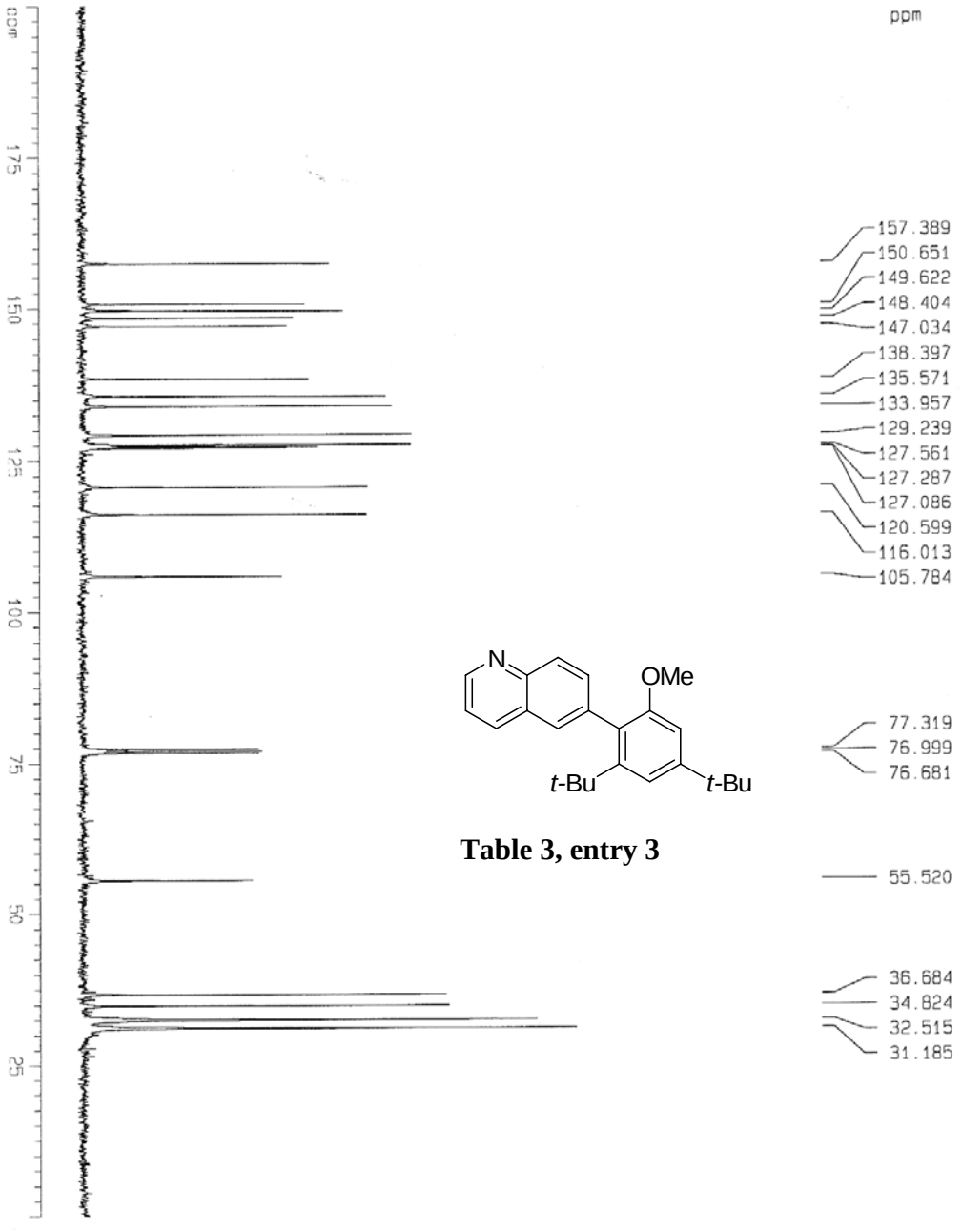


Table 3, entry 3


```
Current Data Parameters
NAME                  Ccoupling
EXPNO                        45
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20070625
Time_                     21.41
INSTRUM                 dpx400
PROBHD     5 mm QNP 1H
PULPROG                 zgdc30
TD                      131072
SOLVENT                 CDCl3
NS                          64
DS                           0
SWH                  25125.629 Hz
FIDRES                0.191693 Hz
AQ                    2.6083827 sec
RG                         8192
DW                       19.900 usec
DE                        4.50 usec
TE                       300.0 K
D1                    3.00000000 sec
d11                   0.03000000 sec

========== CHANNEL f1 ========
NUC1                       13C
P1                        5.80 usec
PL1                      -6.00 dB
SFO1                100.6231263 MHz

========== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    71.00 usec
PL2                      120.00 dB
PL12                     17.00 dB
SF02                400.1325008 MHz

F2 - Processing parameters
SI                         65536
SF                  100.6128082 MHz
WDW                           EM
SSB                            0
LB                          4.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         19.50 cm
F1P                    200.000 ppm
F1                     20122.56 Hz
F2P                     -0.000 ppm
F2                      -0.00 Hz
PPMCM                  10.25641 ppm/
HZCM                1031.92627 Hz/c
```

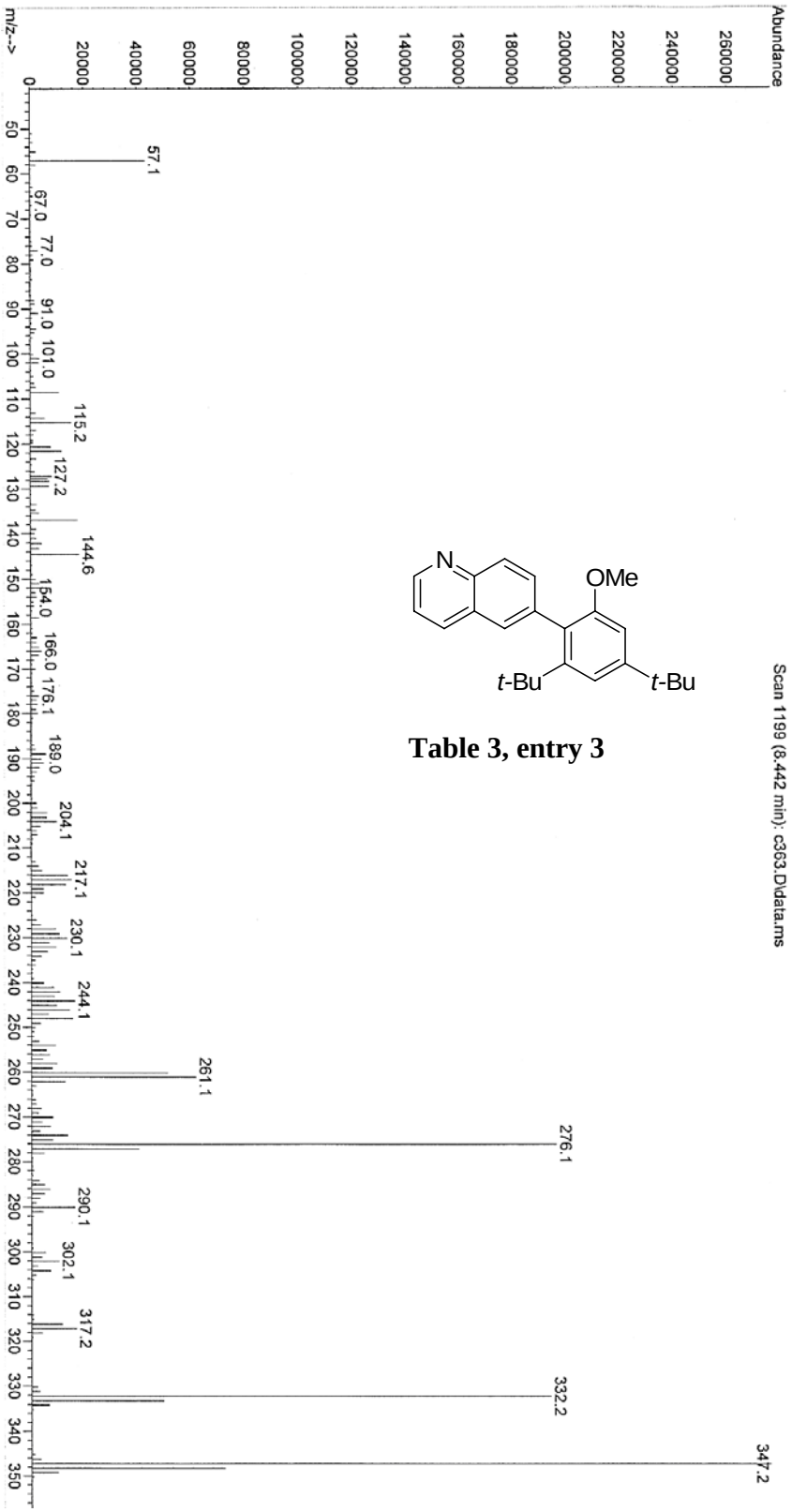
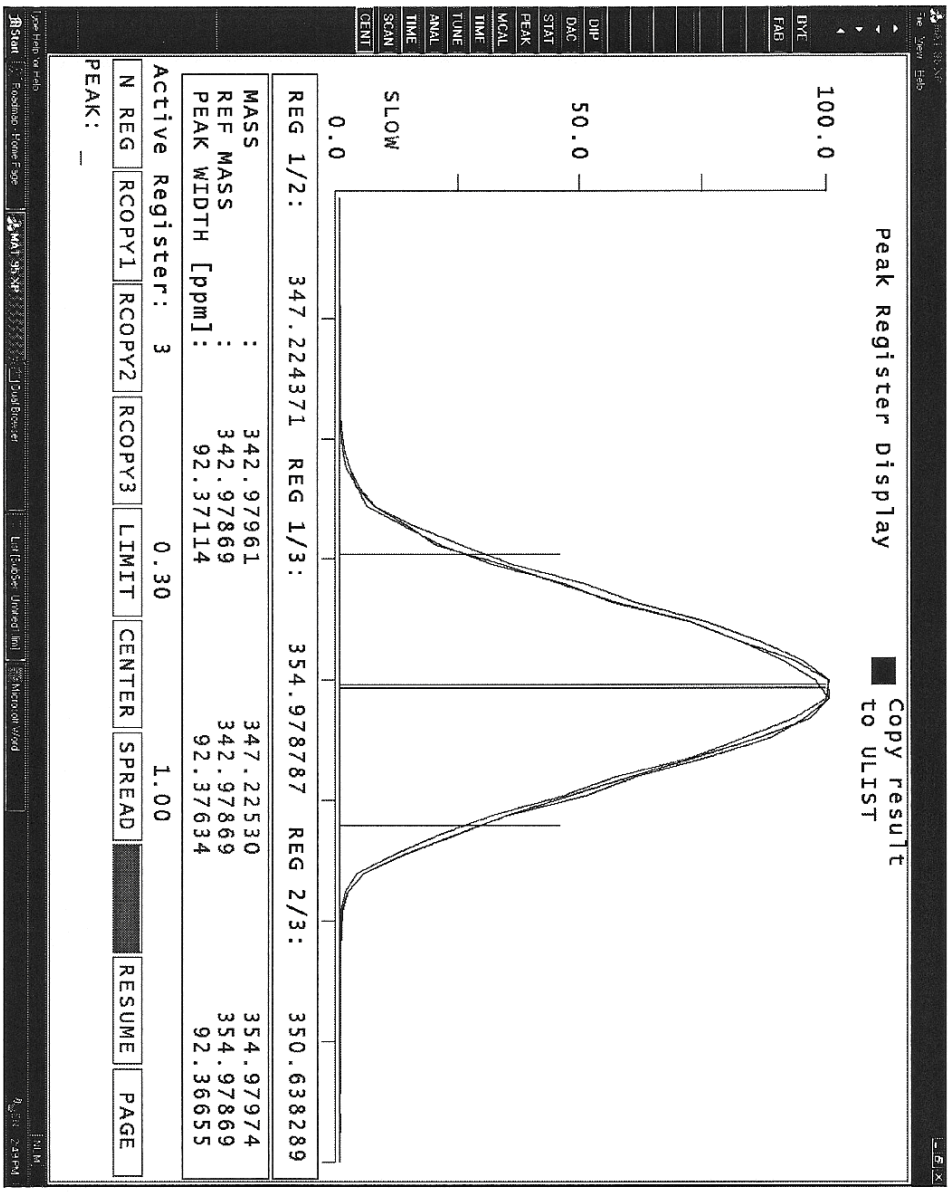


Table 3, entry 3

Accurate Mass Measurement:



Molecular formula: $C_{24}H_{29}N_1O_1$
$[M]^+$ (theoretical) = 347.2244

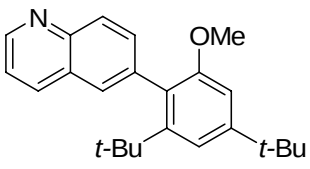


Table 3, entry 3

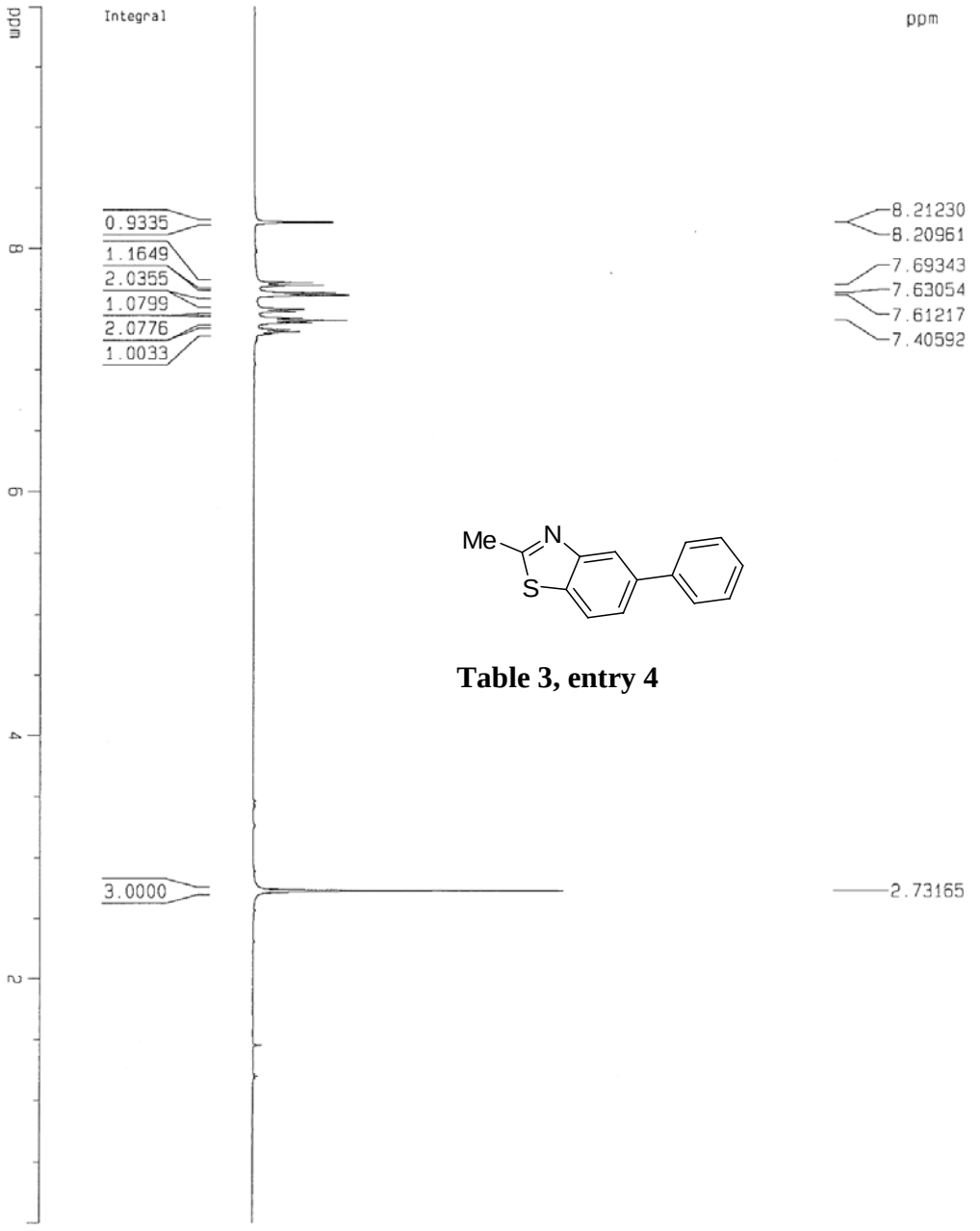


Table 3, entry 4


```
Current Data Parameters
NAME                  coupling
EXPNO                      102
PROCNO                       1

F2 - Acquisition Parameters
Date_              20070830
Time                  14.54
INSTRUM             dpx400
PROBHD     5 mm QNP  1H
PULPROG             zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH                4401.409 Hz
FIDRES             0.134320 Hz
AQ                 3.7224948 sec
RG                    25.4
DW                 113.600 usec
DE                   4.50 usec
TE                  300.0 K
D1                 1.00000000 sec

============== CHANNEL f1 ======
NUC1                   1H
P1                   9.50 usec
PL1                 -6.00 dB
SFO1           400.1320006 MHz

F2 - Processing parameters
SI                    16384
SF             400.1300077 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  19.63 cm
F1P                10.000 ppm
F1                 4001.30 Hz
F2P                -0.000 ppm
F2                   -0.00 Hz
PPMCM              0.50930 ppm/c
HZCM             203.78769 Hz/cm
```

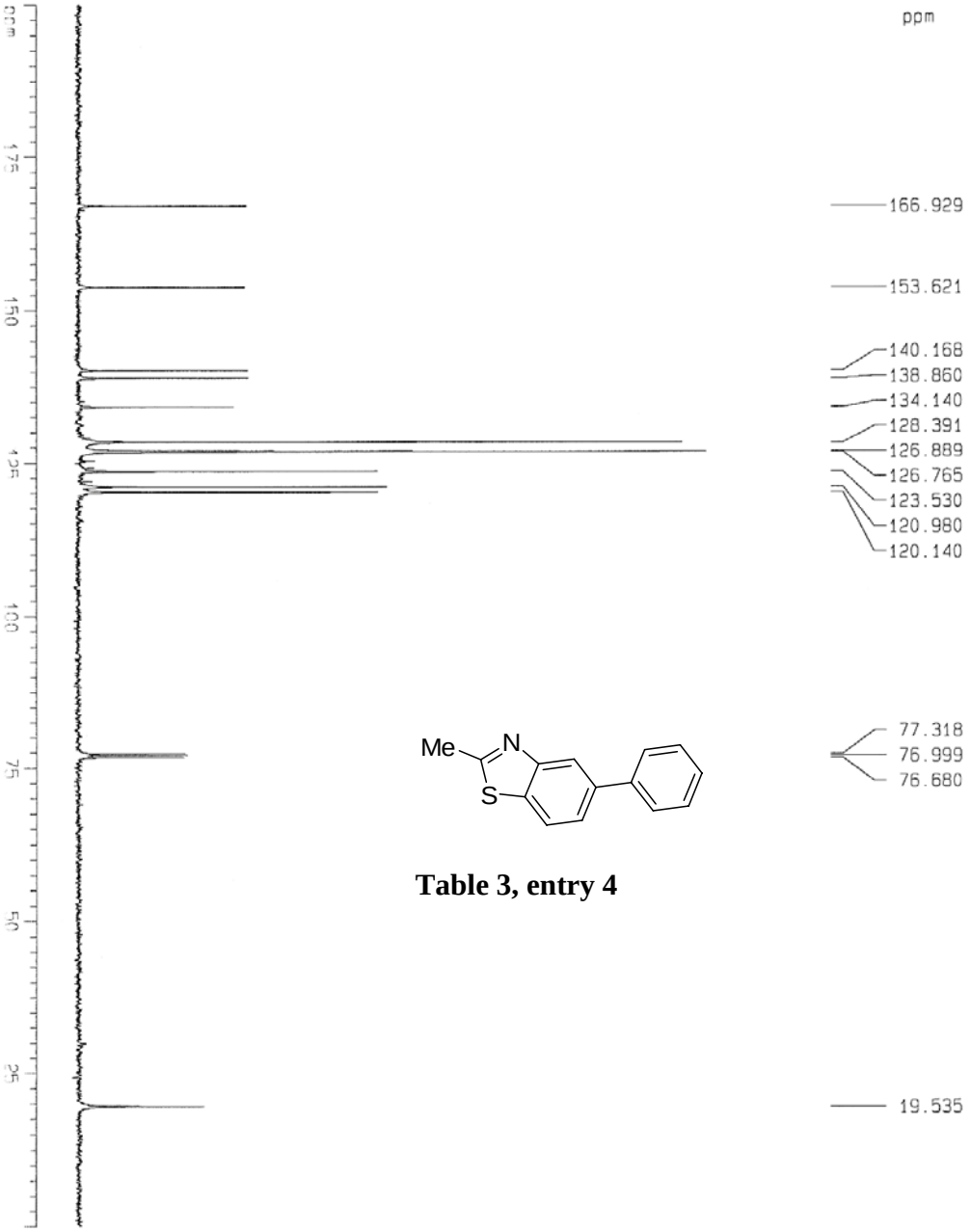


Table 3, entry 4

Current Data Parameters
NAME
EXPNO 59
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070830
Time 14.56
INSTRUM dpx400
PROBHD 5 mm GNP 1H
PULPROG zgdc30
TD 131072
SOLVENT CDCl3
NS 32
DS 0
SWH 25125.629 Hz
FIDRES 0.191693 Hz
AQ 2.6083827 sec
RG 8192
DW 19.900 usec
DE 4.50 usec
TE 300.0 K
D1 3.00000000 sec
d11 0.03000000 sec

========== CHANNEL f1 ======
NUC1 13C
P1 5.80 usec
PL1 -6.00 dB
SFO1 100.6231263 MHz

========== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 71.00 usec
PL2 120.00 dB
PL12 17.00 dB
SFO2 400.1326008 MHz

F2 - Processing parameters
SI 65536
SF 100.6128262 MHz
WDW EM
SSB 0
LB 4.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 19.50 cm
F1P 200.000 ppm
F1 20122.56 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 10.25641 ppm/cm
HZCM 1031.92639 Hz/cm

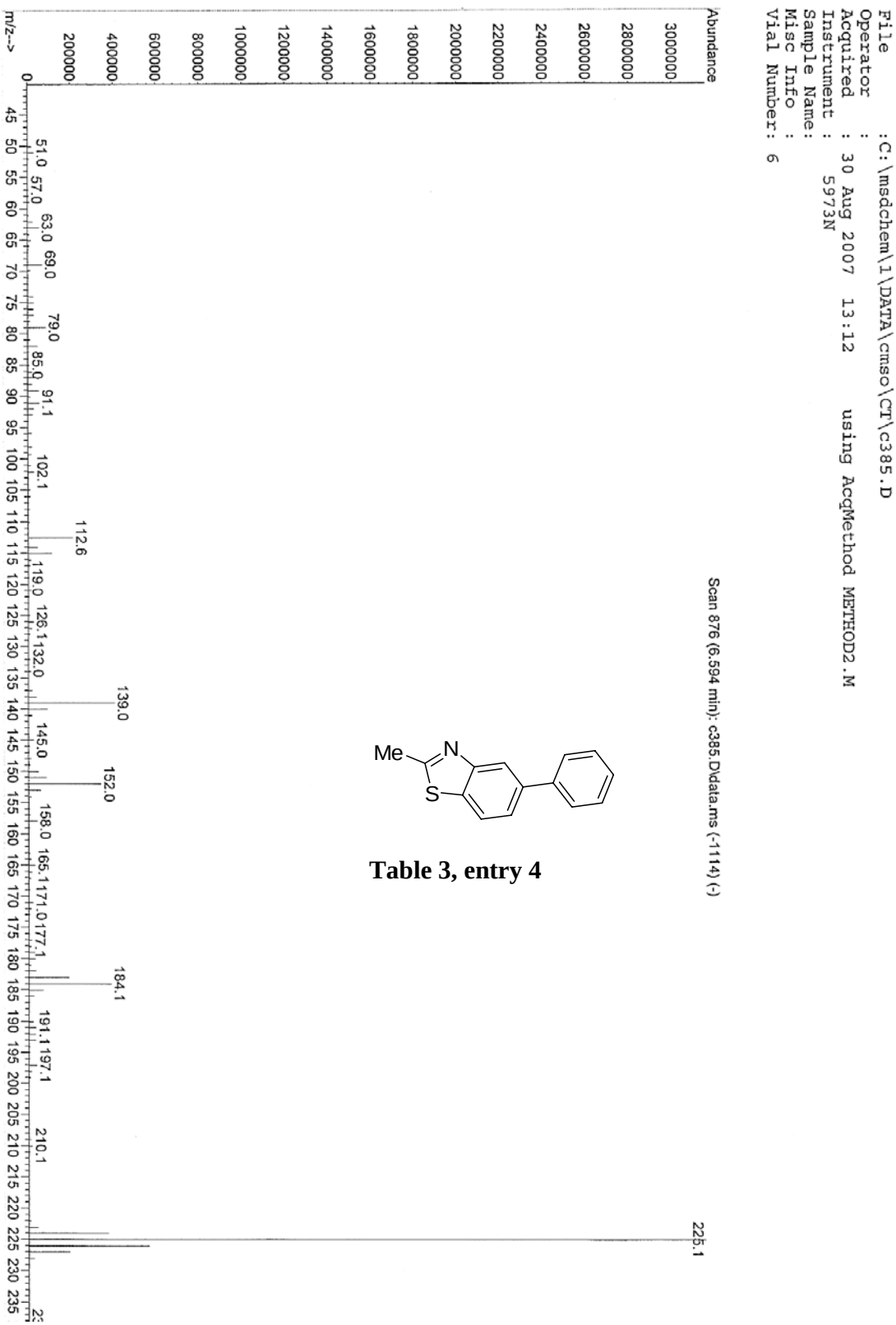


Table 3, entry 4

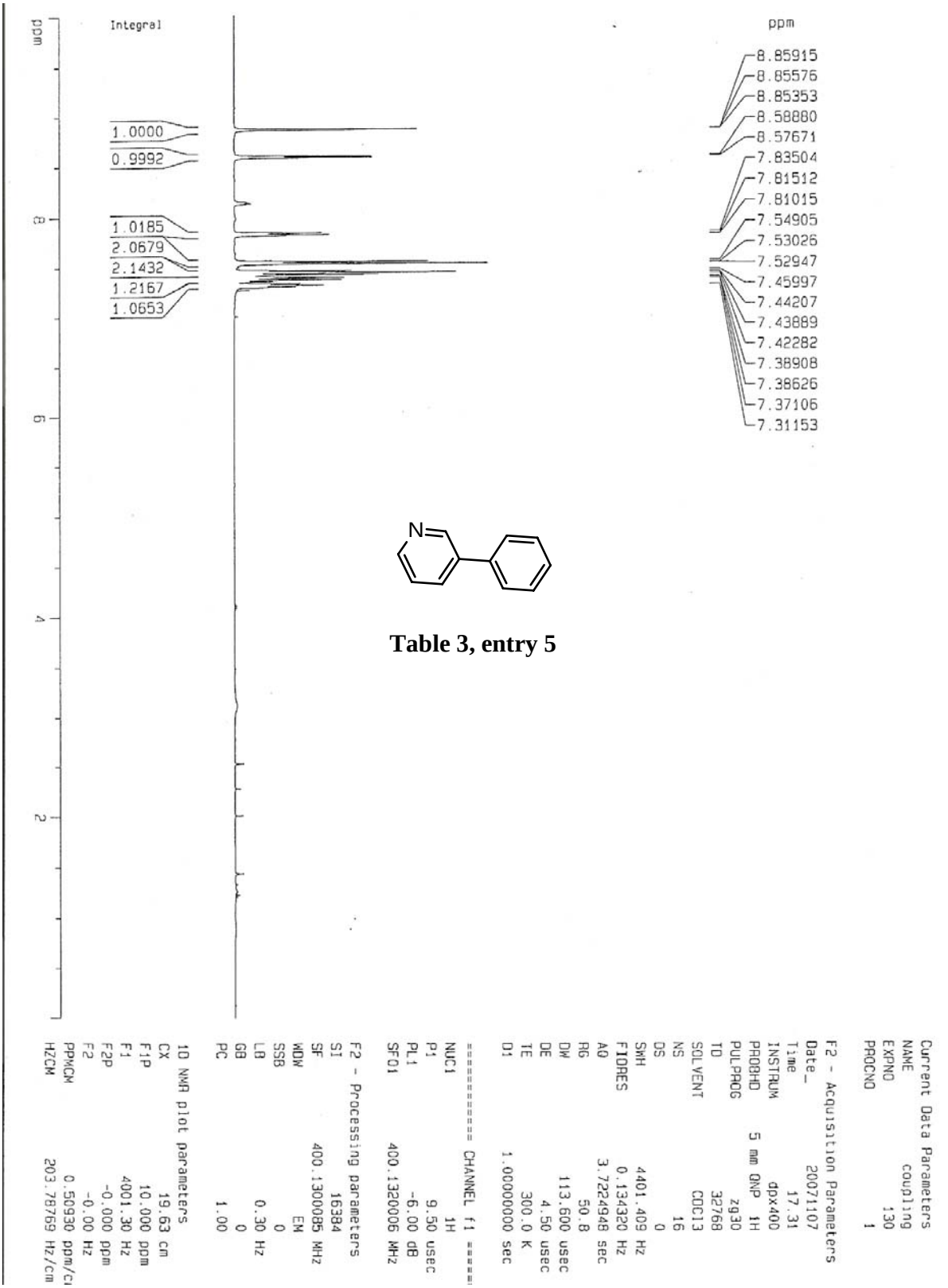


Table 3, entry 5

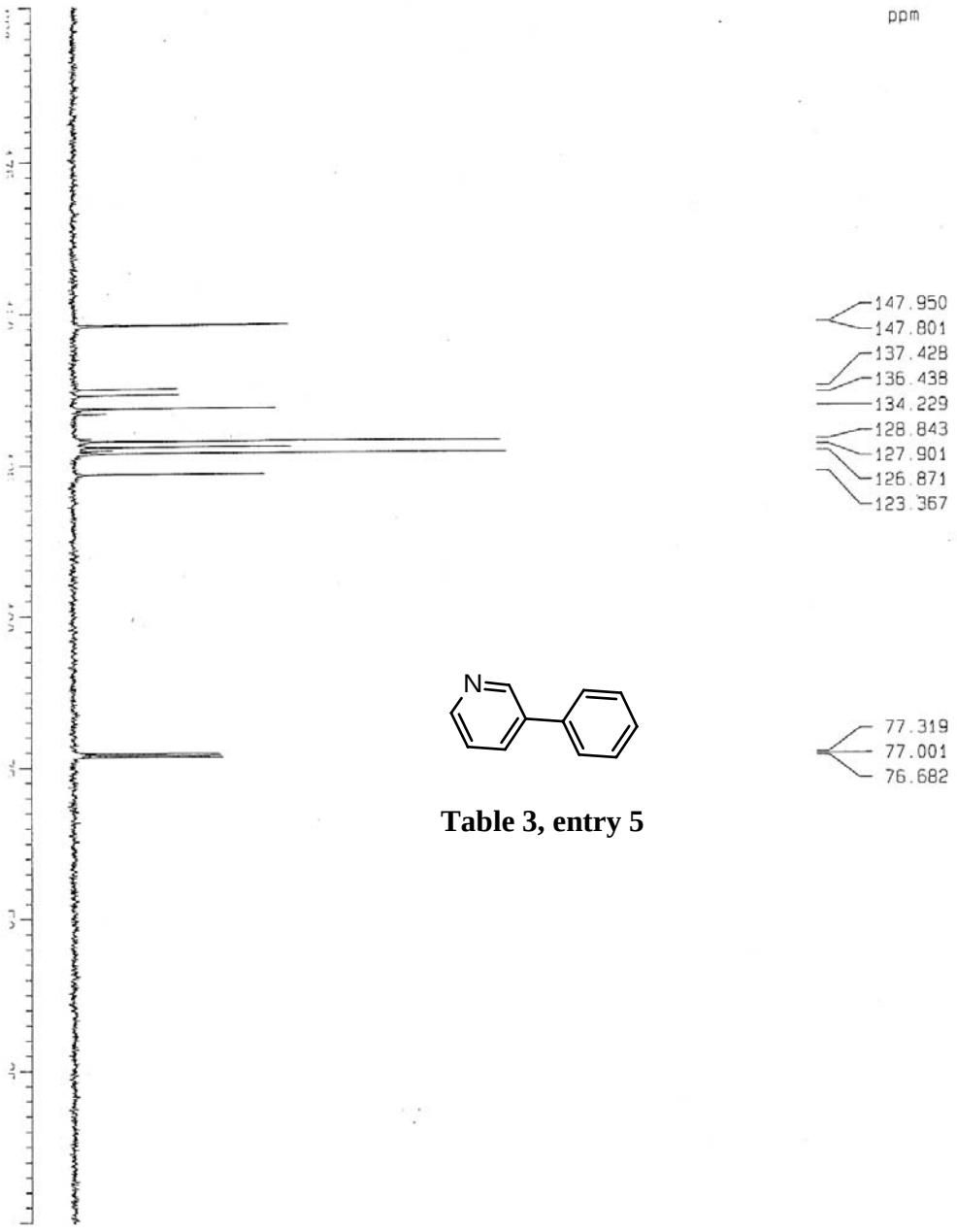


Table 3, entry 5


```
Current Data Parameters
NAME                  Ccoupling
EXPNO                        84
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20071107
Time                      17.33
INSTRUM                 dpx400
PROBHD     5 mm QNP 1H
PULPROG                 zgdc30
TD                      131072
SOLVENT                 CDCl3
NS                            32
DS                             0
SWH                    25125.629 Hz
FIDRES                 0.191693 Hz
AQ                     2.6083827 sec
RG                          8192
DW                        19.900 usec
DE                         4.50 usec
TE                        300.0 K
D1                     3.00000000 sec
d11                    0.03000000 sec

============== CHANNEL f1 ========
NUC1                         13C
P1                         5.80 usec
PL1                       -6.00 dB
SF01             100.6231263 MHz

============== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                     71.00 usec
PL2                       120.00 dB
PL12                       17.00 dB
SF02             400.1326008 MHz

F2 - Processing parameters
SI                         65536
SF               100.6127959 MHz
WDW                           EM
SSB                            0
LB                          4.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         19.50 cm
F1P                      200.000 ppm
F1                     20122.56 Hz
F2P                       -0.000 ppm
F2                        -0.00 Hz
PPMCM                  10.25641 ppm/cm
HZCM                   1051.92515 Hz/cm
```

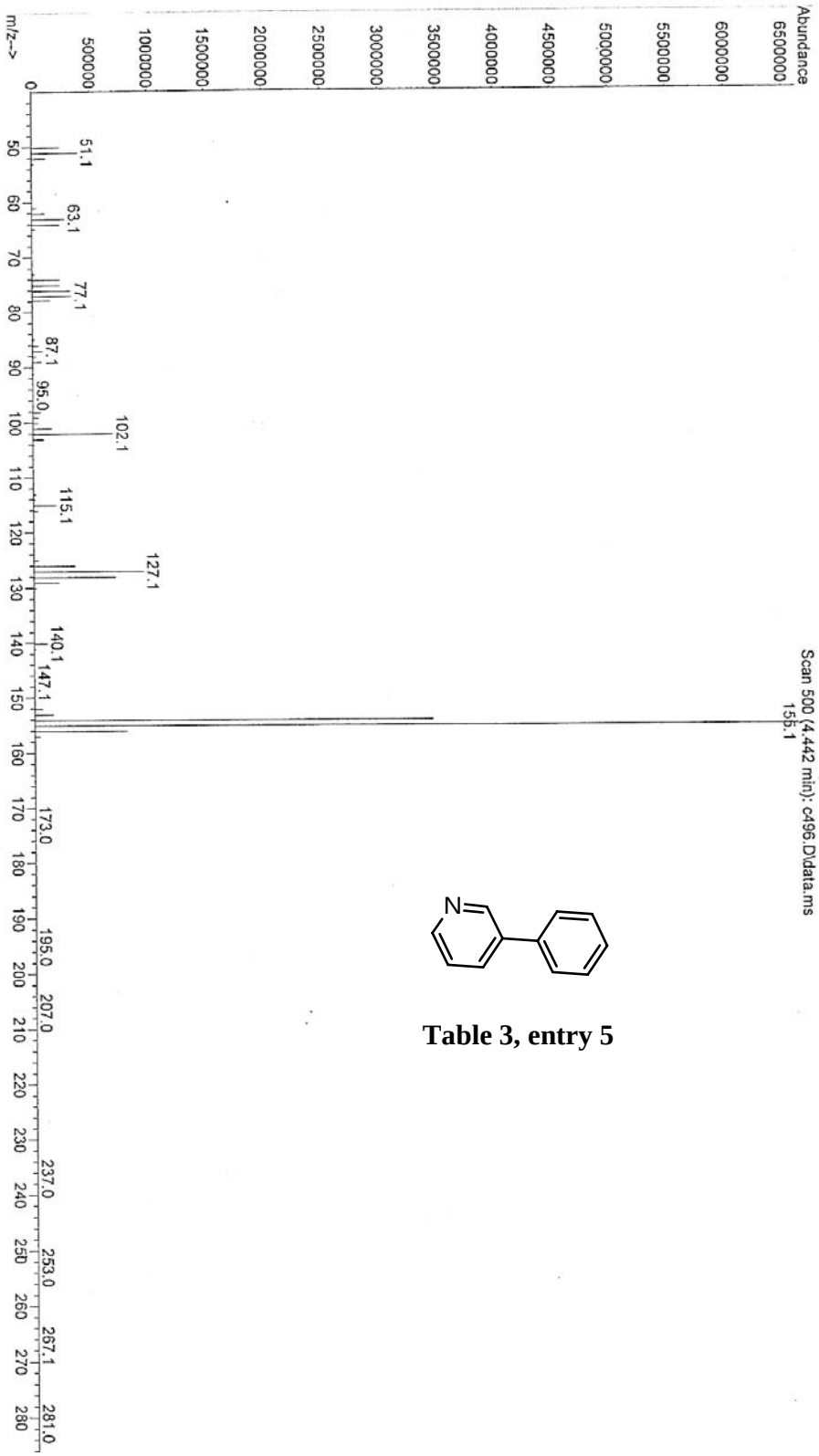


Table 3, entry 5

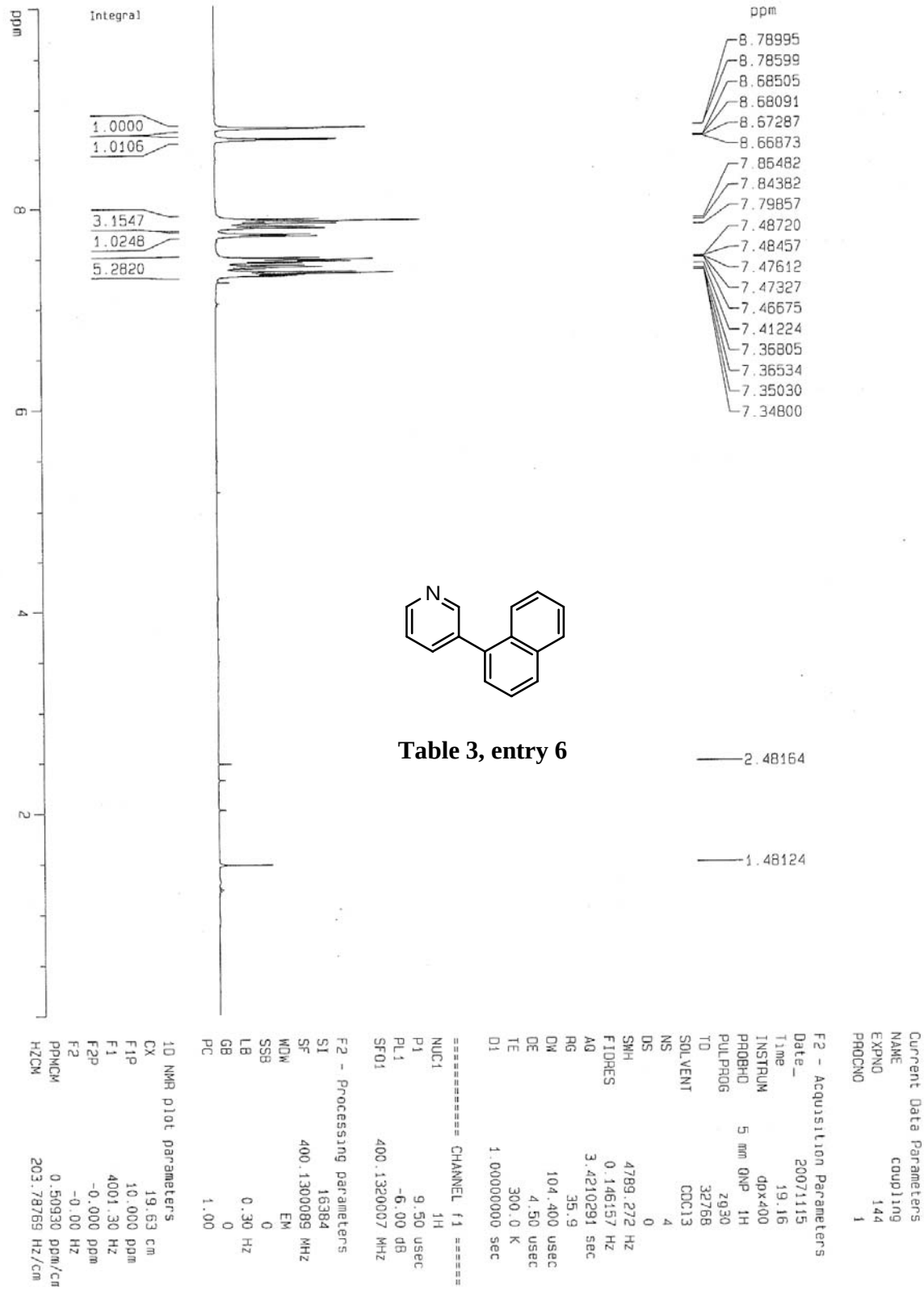


Table 3, entry 6

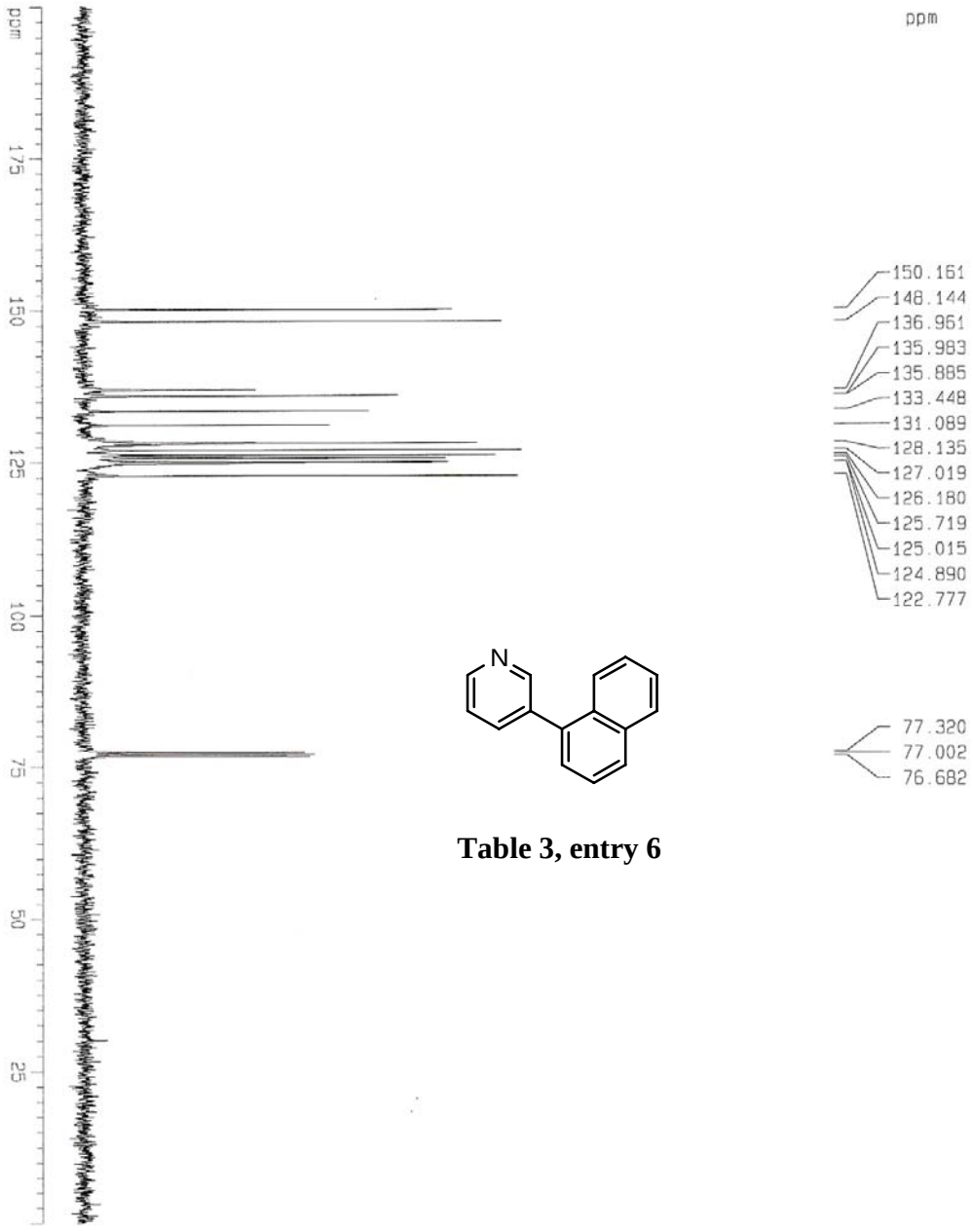


Table 3, entry 6

```
Current Data Parameters
NAME                  Ccoupling
EXPNO                       101
PROCNO                        1

F2 - Acquisition Parameters
Date_              20071117
Time                   14.40
INSTRUM                dpx400
PROBHD     5 mm QNP 1H
PULPROG                zgdc30
TD                     131072
SOLVENT                CDCl3
NS                         16
DS                          0
SWH                25125.629 Hz
FIDRES                 0.191693 Hz
AQ                 2.6083827 sec
RG                        8192
DW                      19.900 usec
DE                       4.50 usec
TE                      300.0 K
D1                 3.00000000 sec
d11                0.03000000 sec

========== CHANNEL f1 ========
NUC1                      13C
P1                       5.80 usec
PL1                     -6.00 dB
SFO1             100.6231263 MHz

========== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    71.00 usec
PL2                     120.00 dB
PL12                     17.00 dB
SFO2             400.1332010 MHz

F2 - Processing parameters
SI                         65536
SF               100.6128116 MHz
WDW                           EM
SSB                            0
LB                          3.50 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         19.50 cm
F1P                      200.000 ppm
F1                     20122.56 Hz
F2P                     -0.000 ppm
F2                       -0.00 Hz
PPMCM                   10.25641 ppm/cm
HZCM                 1031.92527 Hz/cm
```

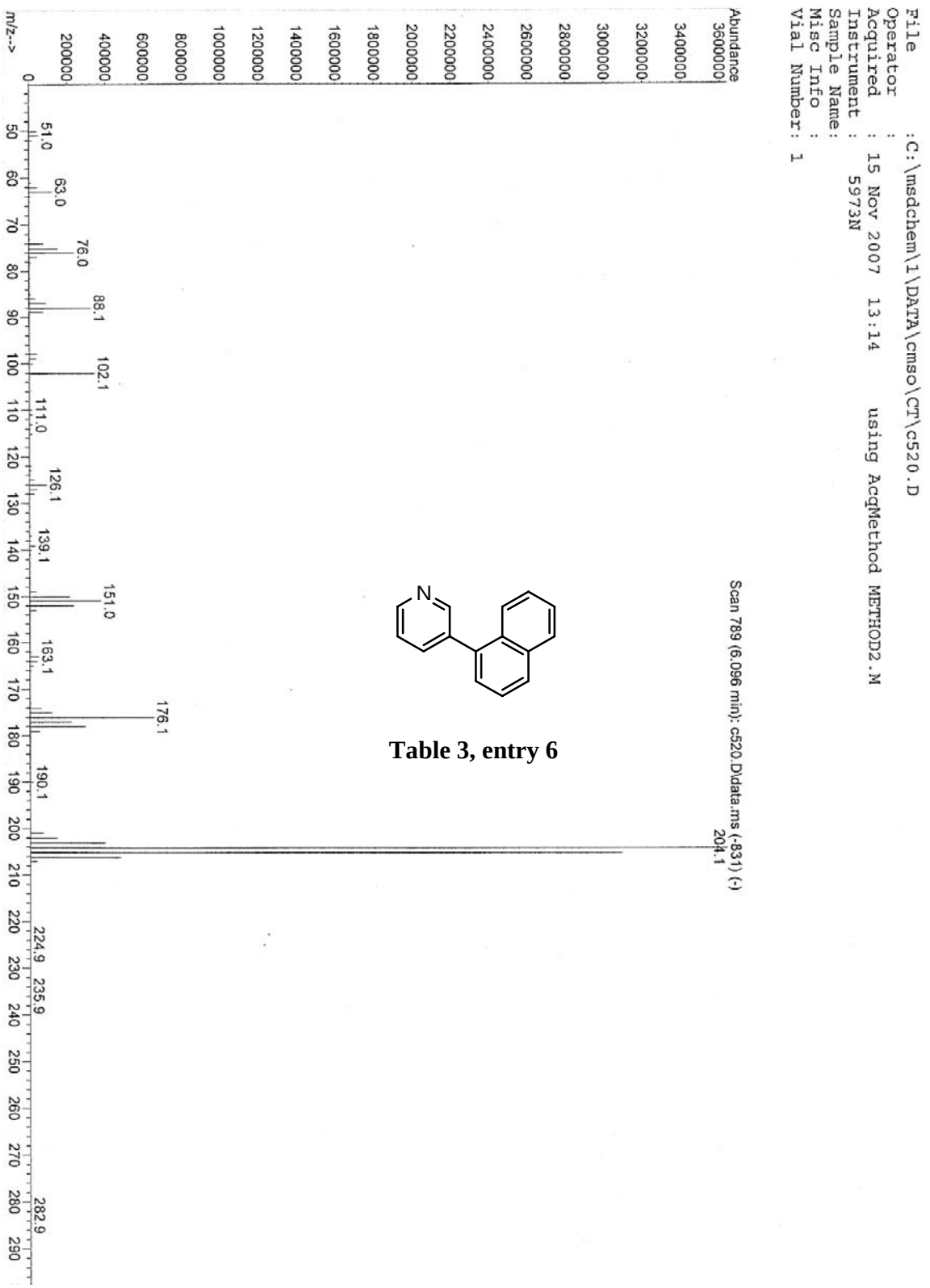
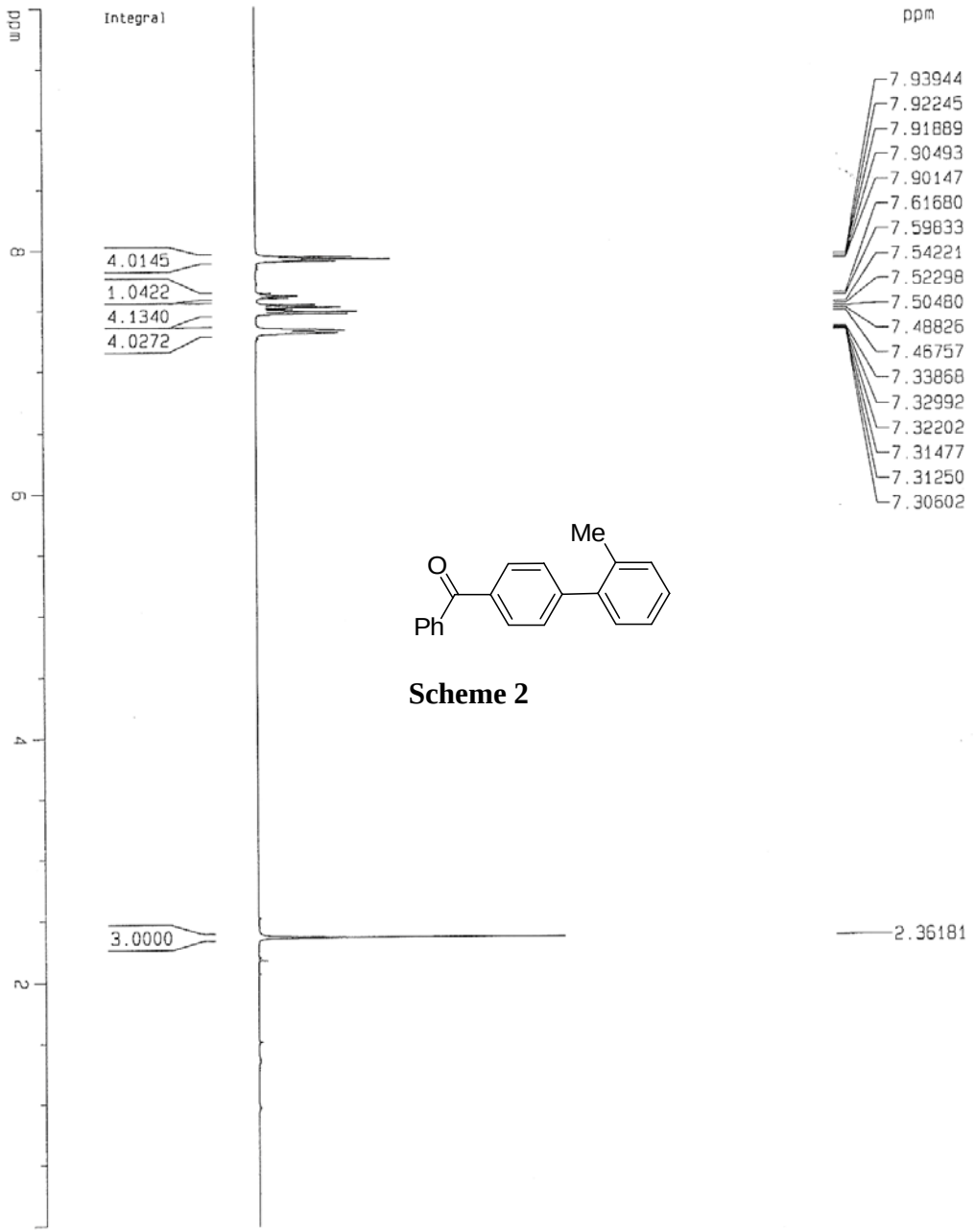
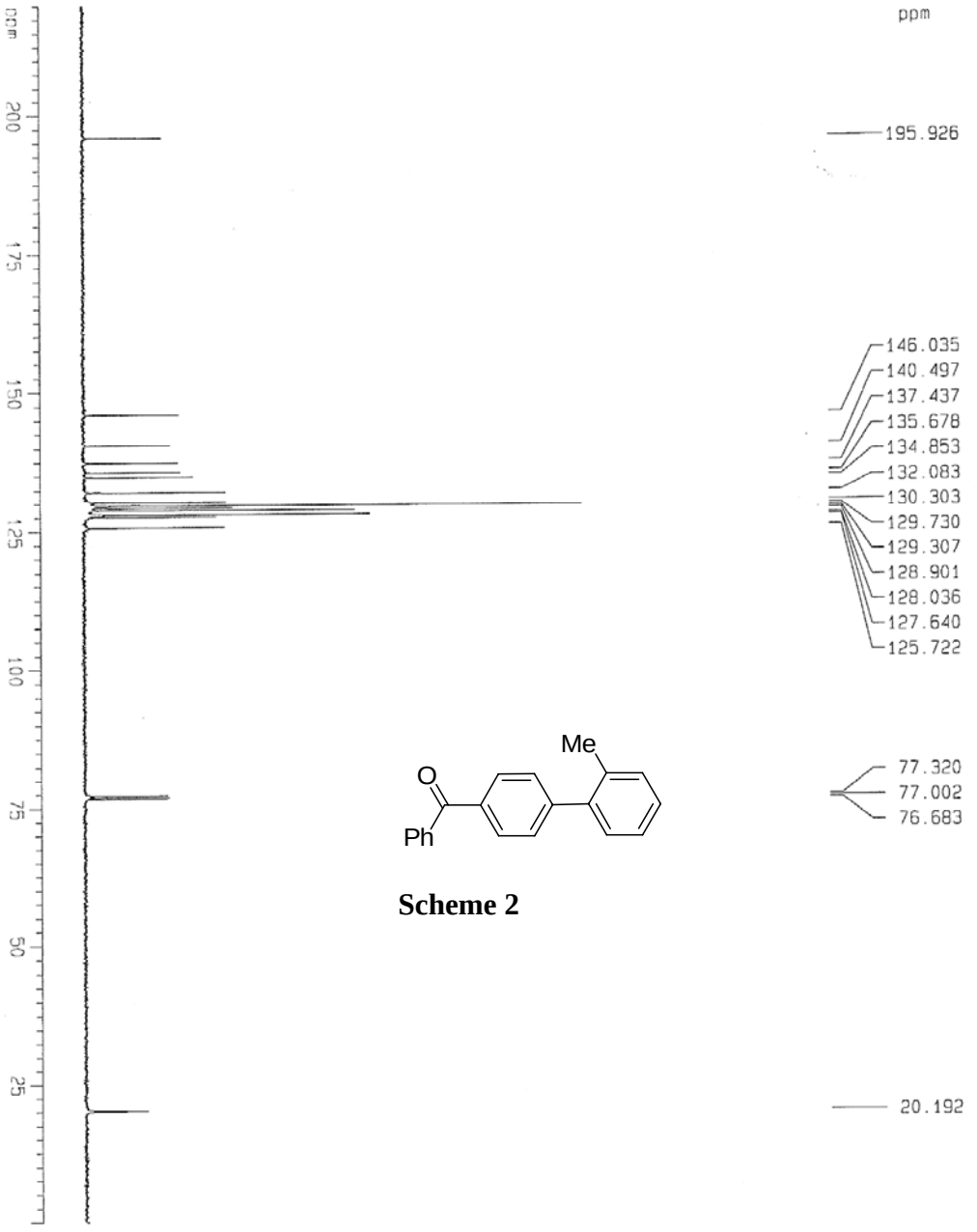


Table 3, entry 6



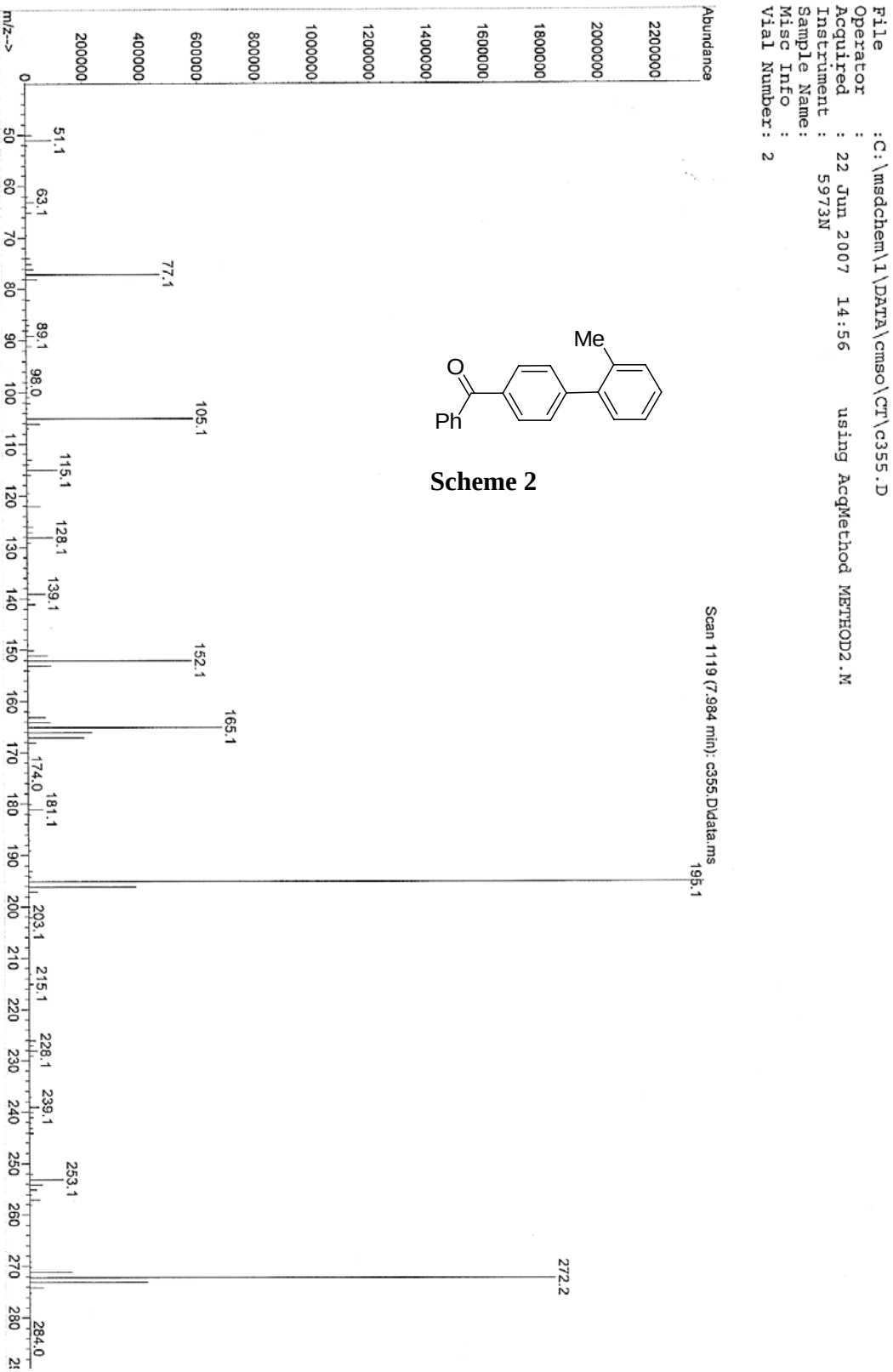
Scheme 2


```
Current Data Parameters
NAME                  coupling
EXPNO                       79
PROCNO                       1

F2 - Acquisition Parameters
Date_              20070625
Time                   18.14
INSTRUM                dpx400
PROBHD     5 mm QNP 1H
PULPROG                 zg30
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                4401.409 Hz
FIDRES             0.134320 Hz
AQ                 3.7224948 sec
RG                        18
DW                 113.600 usec
DE                   4.50 usec
TE                   300.0 K
D1                 1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.50 usec
PL1                   -6.00 dB
SFO1           400.1320007 MHz

F2 - Processing parameters
SI                     16384
SF             400.1300077 MHz
WDW                       EM
SSB                        0
LB                  0.30 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                    19.63 cm
F1P                 10.000 ppm
F1                 4001.30 Hz
F2P                -0.000 ppm
F2                  -0.00 Hz
PPMCM             0.50930 ppm/
HZCM           203.78769 Hz/c
```

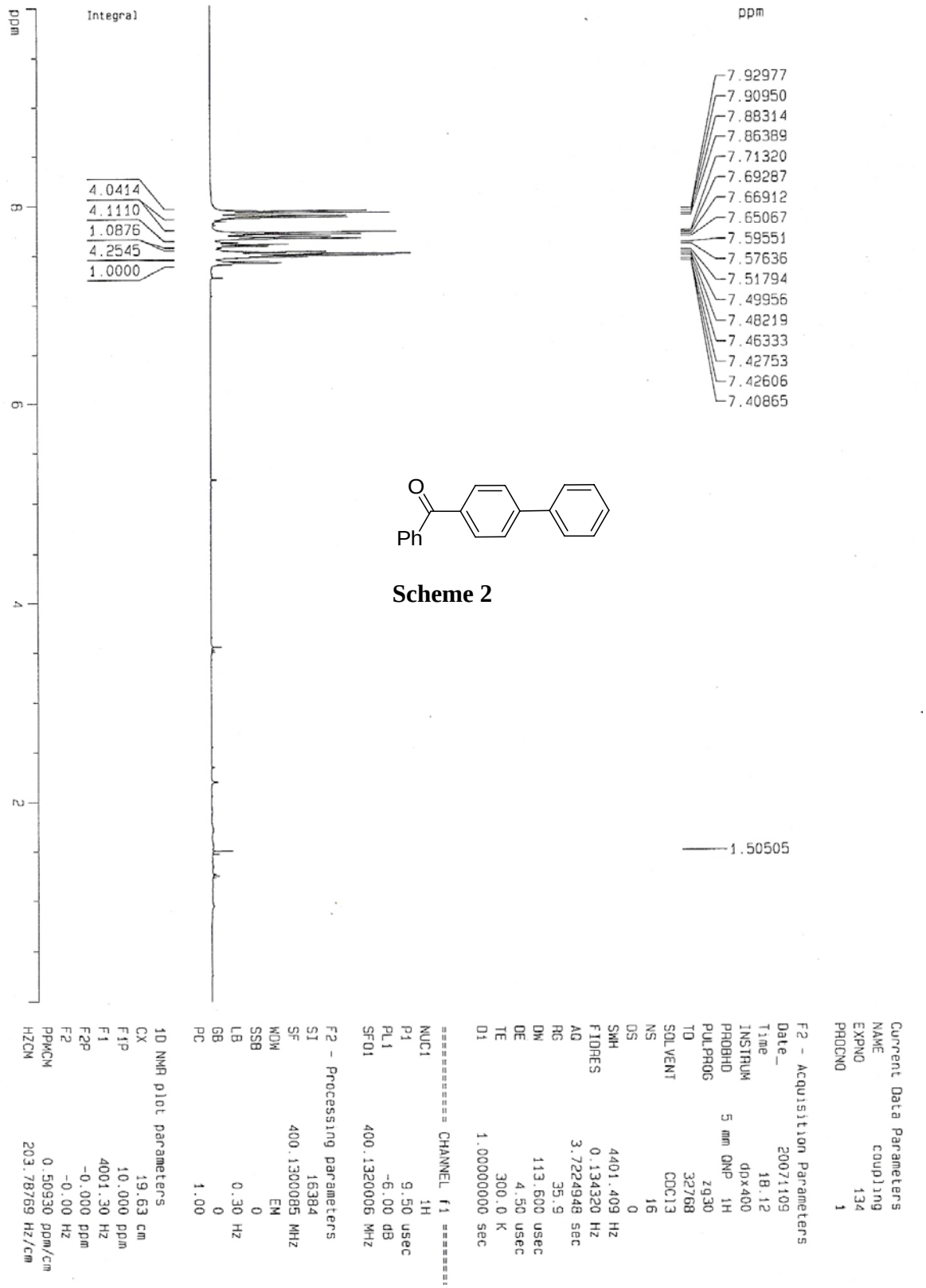


Scheme 2

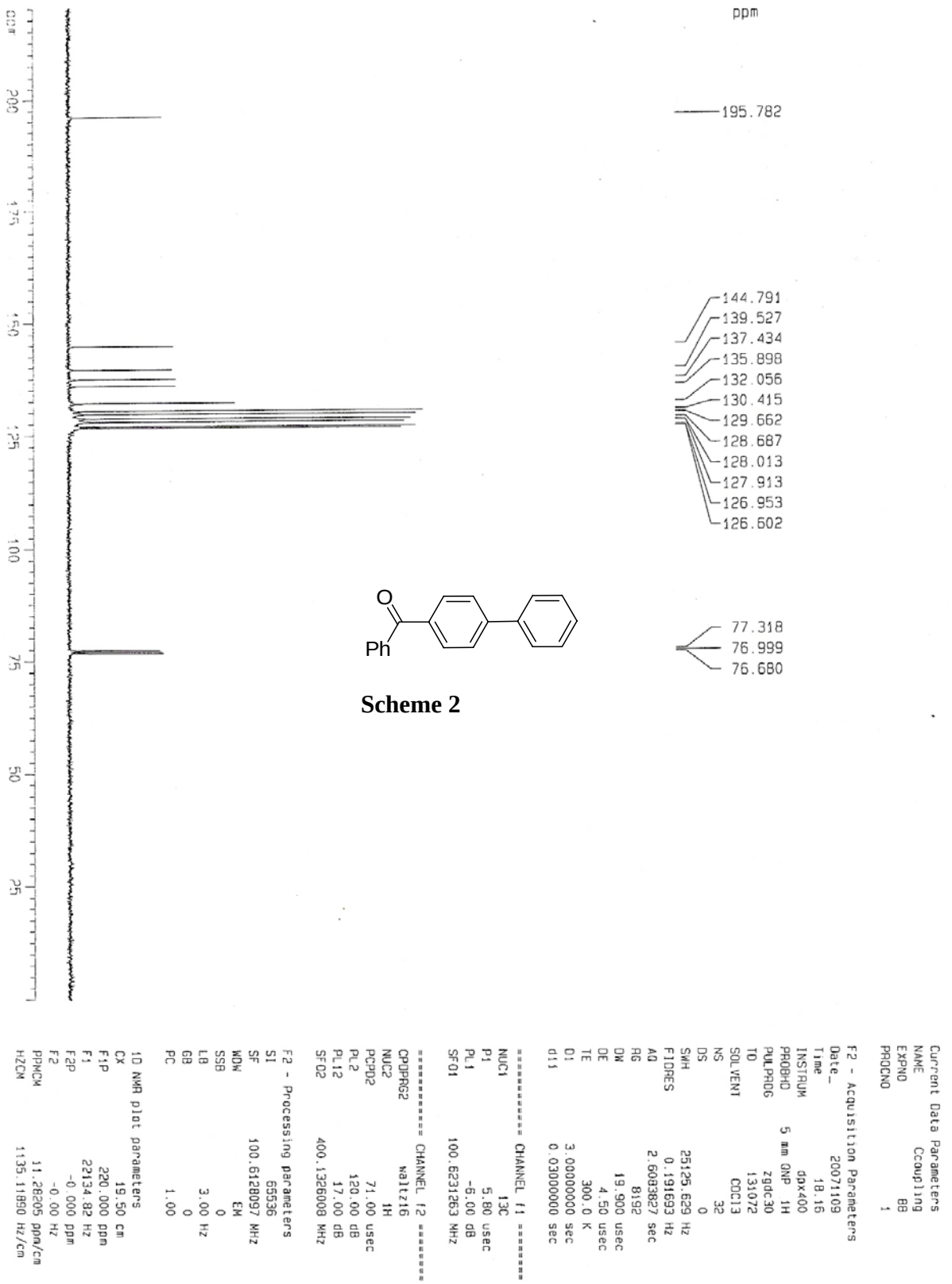

```
Current Data Parameters
NAME                  Ccoupling
EXPNO                        44
PROCNO                        1

F2 - Acquisition Parameters
Date_              20070625
Time                   18.16
INSTRUM                dpx400
PROBHD     5 mm BNP 1H
PULPROG                zgdc30
TD                     131072
SOLVENT                CDCl3
NS                         32
DS                          0
SWH                25125.629 Hz
FIDRES                 0.191693 Hz
AQ                 2.6083827 sec
RG                        8192
DW                    19.900 usec
DE                     4.50 usec
TE                     300.0 K
D1                 3.00000000 sec
d11                0.03000000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                        5.80 usec
PL1                      -6.00 dB
SFO1               100.6231263 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   71.00 usec
PL2                     120.00 dB
PL12                     17.00 dB
SF02               400.1326008 MHz

F2 - Processing parameters
SI                         65536
SF                 100.6128078 MHz
WDW                           EM
SSB                            0
LB                          3.00 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         19.50 cm
F1P                   220.000 ppm
F1                   22134.82 Hz
F2P                   -0.000 ppm
F2                    -0.00 Hz
PPMCM                 11.28205 ppm/
HZCM                1135.11890 Hz/c
```


File :C:\msdchem\1\DATA\cmso\CT\c355.D
Operator :
Acquired : 22 Jun 2007 14:56 using AcqMethod METHOD2.M
Instrument : 5973N
Sample Name:
Misc Info :
Vial Number: 2
Abundance
m/z-->
Scan 1119 (7.984 min): c355.D\data.ms
195.1
272.2
77.1
105.1
152.1
165.1
51.1
63.1
89.1
98.0
115.1
128.1
139.1
174.0
181.1
203.1
215.1
228.1
239.1
253.1
284.0
Me
O
Ph

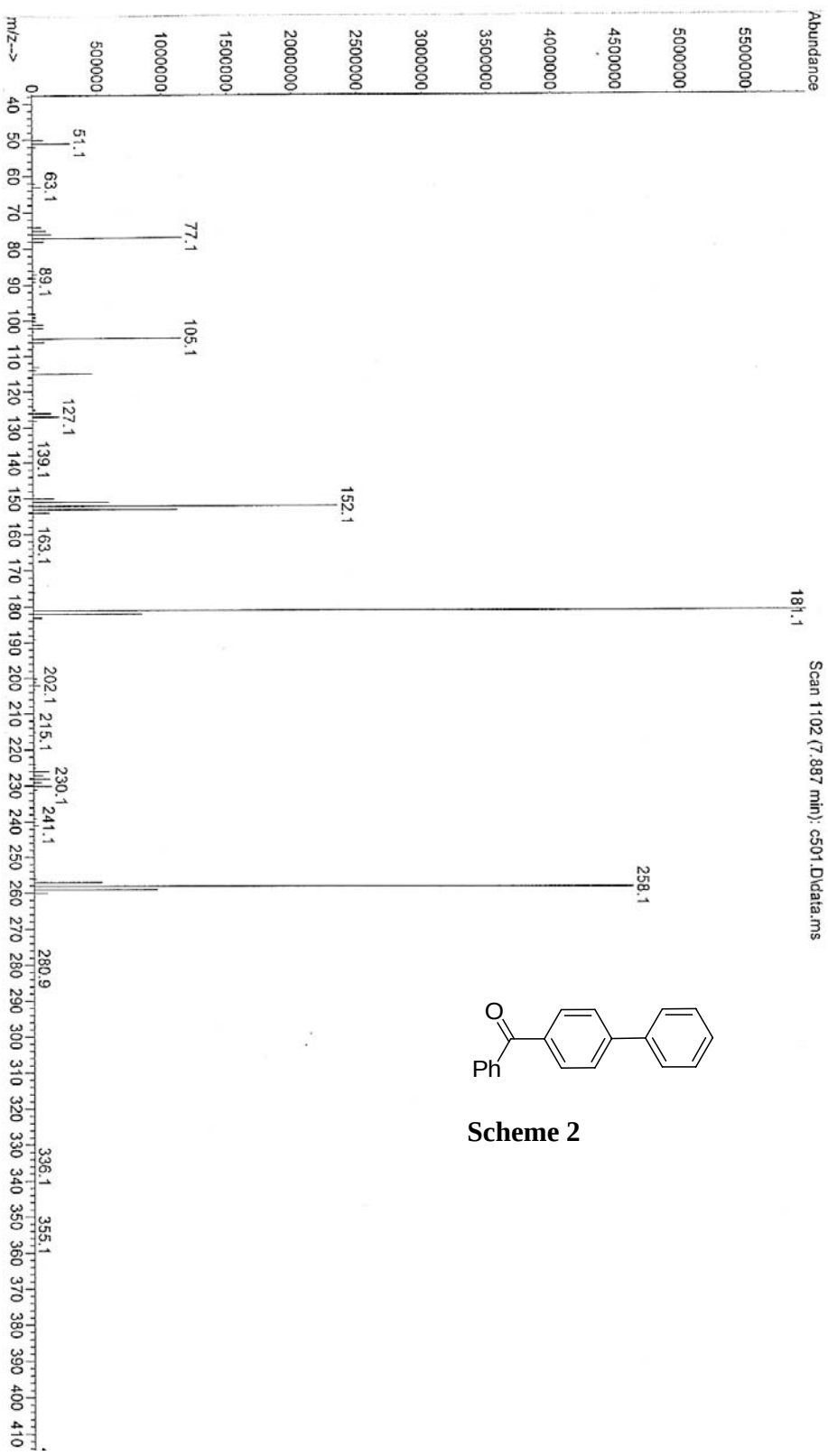
Scheme 2


ppm
Integral
4.0414
4.1110
1.0876
4.2545
1.0000
ppm
7.92977
7.90950
7.88314
7.86389
7.71320
7.69287
7.66912
7.65067
7.59551
7.57636
7.51794
7.49956
7.48219
7.46333
7.42753
7.42606
7.40865
1.50505
O
Ph
Current Data Parameters
NAME coupling
EXPNO 134
PROCNO 1
F2 - Acquisition Parameters
Date_ 20071109
Time 18.12
INSTRUM dpx400
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 4401.409 Hz
FIDRES 0.134320 Hz
AQ 3.7224948 sec
RG 35.9
DW 113.600 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 9.50 usec
PL1 -6.00 dB
SFO1 400.1320006 MHz
F2 - Processing parameters
SI 16384
SF 400.1300085 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 19.63 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 0.50930 ppm/cm
HZCM 203.78759 Hz/cm

Scheme 2


ppm
195.782
144.791
139.527
137.434
135.898
132.056
130.415
129.662
128.687
128.013
127.913
126.953
126.602
77.318
76.999
76.680
O
Ph
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM
PROBHD
PULPROG
TD
SOLVENT
NS
DS
SWH
FIDRES
AQ
RG
DW
DE
TE
D1
d11
======== CHANNEL f1 ========
NUC1
P1
PL1
SFO1
======== CHANNEL f2 ========
CPDPRG2
NUC2
PCPD2
PL2
PL12
SFO2
F2 - Processing parameters
SI
SF
WDW
SSB
LB
GB
PC
1D NMR plot parameters
CX
F1P
F1
F2P
F2
PPMCM
HZCM

Scheme 2


File :C:\msdchem\1\DATA\cmso\CT\c501.D
Operator :
Acquired : 9 Nov 2007 16:00 using AcqMethod METHOD2.M
Instrument : 5973N
Sample Name: c501
Misc Info :
Vial Number: 1
Abundance
m/z-->
Scan 1102 (7.887 min): c501.D\data.ms
181.1
258.1
152.1
77.1
105.1
51.1
63.1
89.1
127.1
139.1
163.1
202.1
215.1
230.1
241.1
280.9
336.1
355.1

O
Ph

Scheme 2

11. References.

(1) Armarego, W. L. F.; Perrin, D. D. *Purification of Laboratory Chemicals.*, 4 Ed. ed., Butterworth-Heinemann: Oxford UK, **1996**.

(2) Guo, M. J.; Varady, L.; Fokas, D.; Baldino, C.; Yu, L. B. *Tetrahedron Lett.* **2006**, *47*, 3889.

(3) Carnahan, J. C.; Closson, W. D.; Ganson, J. R.; Juckett, D. A.; Quaal, K. S. *J. Am. Chem. Soc.* **1976**, *98*, 2526.

(4) Percec, V.; Bae, J. Y.; Zhao, M. Y.; Hill, D. H. *J. Org. Chem.* **1995**, *60*, 176.

(5) Kaboudin, B. *Tetrahedron* **1999**, *55*, 12865.

(6) Bazile, Y.; Cointet, P. D.; Pigerol, C. *J. Het. Chem.***1978**, *15*, 859.

(7) Cerfontain, H.; Ansink, H. R. W.; Coenjaarts, N. J.; Degraaf, E. J.; Koebergtelder, A. *Recueil Des Travaux Chimiques Des Pays-Bas-Journal of the Royal Netherlands Chemical Society* **1989**, *108*, 445.

(8) Swain, N. A.; Brown, R. C. D.; Bruton, G. *J. Org. Chem.* **2004**, *69*, 122.

(9) Delgiudice, M. R.; Settimj, G.; Delfini, M. *Tetrahedron* **1984**, *40*, 4067.

(10) Tang, Z. Y.; Hu, Q. S. *J. Am. Chem. Soc.* **2004**, *126*, 3058.

(11) Moleele, S. S.; Michael, J. P.; de Koning, C. B. *Tetrahedron* **2006**, *62*, 2831.

(12) Zhang, H. C.; Kwong, F. Y.; Tian, Y.; Chan, K. S. *J. Org. Chem.* **1998**, *63*, 6886.

(13) Cho, C. H.; Yun, H. S.; Park, K. *J. Org. Chem.* **2003**, *68*, 3017.

(14) Wolfe, J. P.; Singer, R. A.; Yang, B. H.; Buchwald, S. L. *J. Am. Chem. Soc.* **1999**, *121*, 9550.

(15) Cooper, S. D.; Moseley, M. A.; Pellizzari, E. D. *Anal. Chem.* **1985**, *57*, 2469.

(16) Schultheiss, N.; Barnes, C. L.; Bosch, E. *Synth. Commun.* **2004**, *34*, 1499.

(17) Percec, V.; Golding, G. M.; Smidrkal, J.; Weichold, O. *J. Org. Chem.* **2004**, *69*, 3447.

(18) Miguez, J. M. A.; Adrio, L. A.; Sousa-Pedrares, A.; Vila, J. M.; Hii, K. K. *J. Org. Chem.* **2007**, *72*, 7771.

(19) Lacefield, W. B. M.; Winston, S., *7 pp. Division of U.S. 3,928,604. CODEN: USXXAM US 3968251 19760706 Patent* **1976**.

(20) Fedorova, O. A.; Andryukhina, E. N.; Gromov, S. P. *Synthesis* **2003**, 371.

(21) Kataoka, N.; Shelby, Q.; Stambuli, J. P.; Hartwig, J. F., *J. Org. Chem.* **2002**, *67*, 5553.

(22) Wu, L.; Li, B. L.; Huang, Y. Y.; Zhou, H. F.; He, Y. M.; Fan, Q. H. *Org. Lett.* **2006**, *8*, 3605.

(23) Kataoka, N.; Shelby, Q.; Stambuli, J. P.; Hartwig, J. F., *J. Org. Chem.* **2002**, *67*, 5553.

(24) Nguyen, H. N.; Huang, X. H.; Buchwald, S. L. *J. Am. Chem. Soc.* **2003**, *125*, 11818.

(25) Shi, S. Y.; Zhang, Y. H. *J. Org. Chem.* **2007**, *72*, 5927.

(26) Sapountzis, I.; Lin, W. W.; Kofink, C. C.; Despotopoulou, C.; Knochel, P. *Angew. Chem. Int. Ed.* **2005**, *44*, 1654.

(27) Grabowsk, E.; Autrey, R. L. *Tetrahedron* **1969**, *25*, 4315.

(28) Leadbeater, N. E.; Smith, R. J. *Org. Lett.* **2006**, *8*, 4588.

(29) Cioffi, C. L.; Spencer, W. T.; Richards, J. J.; Herr, R. J. *J. Org. Chem.* **2004**, *69*, 2210.