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Palladium-Catalyzed Stille Cross-Coupling Reaction of Aryl Chlorides using a Pre-Milled Palladium Acetate and XPhos Catalyst System.

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General. All reactions were carried out under an argon atmosphere. Dimethoxyethane (DME 99.5%, Anhydrous) and 1,4-dioxane (99.8%, Anhydrous) were purchased from Aldrich Chemical Co. Commercially available materials were used without further purification unless otherwise noted. XPhos was synthesized in our laboratories but is commercially available from Aldrich Chemical Co. or Strem Chemicals, Inc. Aryl halides were purchased from Aldrich Chemical Co. or Alfa Aesar. Phenyl- furyl- thiophenyl- and allyltributylstannanes were purchased from Aldrich Chemical Co. or Gelest, Inc. In analogy to the procedures described by Littke and Fu,^[1] other stannanes were prepared by lithiation of the corresponding aryl bromides with *n*-BuLi (2.5 M in hexanes, Aldrich) followed by quenching with tributyltin chloride; 1-lithio-2,6-dimethoxybenzene was prepared by directed lithiation^[2] and quenched with tributyltin chloride. Cesium fluoride (99.9%) was purchased from Aldrich Chemical Co. and stored in a bench-top dessicator. Palladium acetate was supplied by BASF. All products from coupling reactions were characterized by ¹H NMR, ¹³C NMR, IR spectroscopy, elemental analysis and melting points for solids. Known compounds were compared to their literature values. ¹H and ¹³C NMR spectra were recorded on a Bruker Advance 400. Infrared spectra were recorded on a Perkin-Elmer Model 2000 FT-IR using NaCl plates (thin film). Elemental analyses were performed by Atlantic Microlabs Inc., Norcross, GA. All ¹H NMR experiments are reported in parts per million (ppm) downfield of TMS and were measured relative to the signals for chloroform (7.24 ppm). All ¹³C NMR spectra were reported in ppm relative to residual chloroform (77.23 ppm) and were obtained with ¹H decoupling. Melting points were obtained on a Mel-Temp capillary melting point apparatus. Gas chromatographic analyses were performed on Hewlett-Packard 6890 gas chromatography instrument with a FID detector using 25m x 0.20 mm capillary column with cross-linked methyl siloxane as a stationary phase. The yields in tables 2

and 3 refer to isolated yields (average of two runs) of compounds estimated to be $\geq 95\%$ pure as determined by ^1H NMR and GC analysis and/or combustion analysis. The procedures described in this section are representative and may differ slightly from those given in tables 2 and 3.

$\text{Pd}(\text{OAc})_2$ (225 mg, 1.00 mmol) and XPhos (524 mg, 1.10 mmol) were ground together using a mortar and pestle until a fine beige powder was formed. This powder (effective molecular weight: 749 g/mol) was stored in a bench-top dessicator for up to six months with no loss of activity and was used as the catalyst for all reactions described in this paper except where noted.

General Procedure A:

Pre-milled $\text{Pd}(\text{OAc})_2/\text{XPhos}$ and CsF (334 mg, 2.20 mmol) were added to an oven-dried test tube. The tube was fitted with a rubber septum, sealed with electrical tape, and evacuated and backfilled with argon (this process was repeated a total of 3 times). Dimethoxyethane (DME, 1.0 mL), aryl chloride (1.00 mmol) and stannane (1.10 mmol) were added via syringe to the tube. The reaction was heated to 80 °C with stirring for 4 h. At this time the reaction was removed from the oil bath, allowed to cool to room temperature, diluted with ethyl acetate (5 mL) and filtered through a pad of silica gel, eluting with ethyl acetate (150 mL). The solvent was removed with the aid of a rotary evaporator, and the residue was taken up in dichloromethane (20 mL) and added to silica gel (~1 g). The solvent was concentrated under reduced pressure and the residue (adsorbed on silica) was loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4.

2-(4-Methoxyphenyl)furan (Table 2, entry 1).^[3] Following general procedure A, 4-chloroanisole (123 μL , 1.00 mmol), tributyl(2-furanyl)stannane (346 μL , 1.10 mmol), pre-milled $\text{Pd}(\text{OAc})_2/\text{XPhos}$ (15.0 mg, 0.020 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 °C in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 99% yield (173 mg) as a white solid, mp 55-56 °C. ^1H NMR (400 MHz, CDCl_3) δ 7.59 (d, J = 9.0 Hz, 2H), 7.41 (dd, J = 1.5, 0.5 Hz, 1H), 6.91 (d, J = 9.0 Hz, 2H), 6.50 (dd, J = 3.5, 0.5 Hz, 1H), 6.43 (dd, J = 3.5, 1.5 Hz, 1H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 159.2, 154.2, 141.6, 125.4, 124.2, 114.3, 111.7, 103.5, 55.5; IR (neat, cm^{-1}) 3450, 2958, 2837, 1515, 485, 1254, 1026, 834, 801, 734. Anal. Calc'd. for $\text{C}_{11}\text{H}_{10}\text{O}_2$: C, 75.84; H, 5.79.

Found C, 75.71; H, 5.77.

4'-Methoxy-2,4,6-trimethylbiphenyl (Table 2, entry 2).^[4] Following general procedure A, 4-chloroanisole (123 μ L, 1.00 mmol), tributyl(2,4,6-mesityl)stannane (401 μ L, 1.10 mmol), pre-milled Pd(OAc)₂/XPhos (7.5 mg, 0.010 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 °C in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 95% yield (215 mg) as a white solid, mp 75-77 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.04 (d, J = 9.0 Hz, 2H), 6.94 (d, J = 9.0 Hz, 2H), 6.93 (s, 2H), 3.84 (s, 3H), 2.37 (s, 3H), 2.00 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 158.3, 138.8, 136.6, 133.4, 130.5, 128.2, 113.9, 55.4, 21.2, 21.0; IR (neat, cm⁻¹) 2933, 2833, 1609, 1514, 1285, 1245, 1042, 854, 832, 733. Anal. Calc'd. for C₁₆H₁₈O: C, 84.91; H, 8.02. Found C, 84.81; H, 8.04.

5-(2-Furanyl)benzo[d][1,3]dioxole (Table 2, entry 3).^[5] Following general procedure A, 5-chlorobenzo[d][1,3]dioxole (117 μ L, 1.00 mmol), tributyl(2-furanyl)stannane (346 μ L, 1.10 mmol), pre-milled Pd(OAc)₂/XPhos (7.5 mg, 0.010 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 °C in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 95% yield (178 mg) as a yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.40 (d, J = 2.0 Hz, 1H), 7.16 (dd, J = 8.0, 2.0 Hz, 1H), 7.13 (d, J = 2.0 Hz, 1H), 6.81 (d, J = 8.0 Hz, 1H), 6.48 (d, J = 3.0 Hz, 1H), 6.42 (dd, J = 3.0, 2.0 Hz, 1H), 5.96 (s, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 153.9, 148.1, 147.1, 141.6, 125.5, 117.7, 111.7, 108.7, 104.7, 104.0, 101.2; IR (neat, cm⁻¹) 3457, 2894, 1507, 1479, 1451, 1257, 1227, 1040, 811, 733. Anal. Calc'd. for C₁₁H₈O₃: C, 70.21; H, 4.29. Found C, 70.16; H, 4.21.

5-*o*-tolylbenzo[d][1,3]dioxole (Table 2, entry 4).^[6] Following general procedure A, 5-chlorobenzo[d][1,3]dioxole (117 μ L, 1.00 mmol), tributyl(*o*-tolyl)stannane (373 μ L, 1.10 mmol), pre-milled Pd(OAc)₂/XPhos (7.5 mg, 0.010 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 °C in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 91% yield (192 mg) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.23 (m, 4H), 6.86 (d, J = 8.0 Hz, 1H), 6.82 (d, J = 1.5 Hz, 1H), 6.77 (dd, J = 8.0, 1.5

Hz, 1H), 5.99 (s, 2H), 2.28 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.5, 146.6, 141.7, 136.0, 135.7, 130.5, 130.0, 127.3, 125.9, 122.7, 110.0, 108.2, 101.2, 20.7; IR (neat, cm^{-1}) 2460, 2889, 1504, 1478, 1246, 1221, 1040, 760, 731. Anal. Calc'd. for $\text{C}_{14}\text{H}_{12}\text{O}_2$: C, 79.22; H, 5.70. Found C, 79.14; H, 5.84.

5-*o*-tolylbenzo[*d*][1,3]dioxole (Table 2, entry 5).^{16l} Modification to general procedure A, 5-chlorobenzo[*d*][1,3]dioxole (117 μL , 1.00 mmol), tributyl(*o*-tolyl)stannane (373 μL , 1.10 mmol), $\text{Pd}(\text{OAc})_2$ (2.3 mg 0.010 mmol), XPhos (5.2 mg, 0.011 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 $^\circ\text{C}$ in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 96% yield (203 mg) as a colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 7.25 (m, 4H), 6.88 (d, J = 8.0 Hz, 1H), 6.84 (d, J = 1.5 Hz, 1H), 6.79 (dd, J = 8.0, 1.5 Hz, 1H), 6.01 (s, 2H), 2.30 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 147.4, 146.6, 141.7, 136.0, 135.6, 130.5, 130.0, 127.3, 125.9, 122.6, 110.0, 108.2, 101.1, 20.7.

***N,N*-Dimethyl-2'-(trifluoromethyl)biphenyl-3-amine (Table 2, entry 6).** Following general procedure A, 3-chloro-*N,N*-dimethylaniline (137 μL , 1.00 mmol), tributyl(2-(trifluoromethyl)phenyl)stannane (389 μL , 1.10 mmol), pre-milled $\text{Pd}(\text{OAc})_2/\text{XPhos}$ (15.0 mg, 0.020 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 $^\circ\text{C}$ in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 87% yield (229 mg) as a pale yellow oil. ^1H NMR (400 MHz, CDCl_3) δ 7.72 (d, J = 7.5 Hz, 1H), 7.53 (t, J = 7.5 Hz, 1H), 7.43 (t, J = 7.5 Hz, 1H), 7.36 (d, J = 7.5 Hz, 1H), 7.25 (t, J = 8.0 Hz, 1H), 6.75 (d, J = 8.0 Hz, 1H), 6.66 (m, 2H), 2.95 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 150.0, 142.5, 140.8, 132.2, 131.4, 128.6, 128.6 (q, J = 30 Hz), 127.3, 126.2 (q, J = 5 Hz), 124.5 (q, J = 272 Hz), 117.6, 113.6, 111.8, 40.7; IR (neat, cm^{-1}) 2888, 1601, 1491, 1314, 1170, 1128. 1109, 768, 701. Anal. Calc'd. for $\text{C}_{15}\text{H}_{14}\text{F}_3\text{N}$: C, 67.91; H, 5.32. Found C, 67.98; H, 5.42.

***N,N*-Dimethylbiphenyl-3-amine (Table 2, entry 7).**^{17l} Following general procedure A, 3-chloro-*N,N*-dimethylaniline (137 μL , 1.00 mmol), tributyl(phenyl)stannane (359 μL , 1.10 mmol), pre-milled $\text{Pd}(\text{OAc})_2/\text{XPhos}$ (15.0 mg, 0.020 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 $^\circ\text{C}$ in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded

onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 90% yield (178 mg) as a pale yellow oil. ¹H NMR (400 MHz, CDCl₃) δ 7.68 (d, *J* = 8.0 Hz, 2H), 7.50 (t, *J* = 7.5 Hz, 2H), 7.39 (m, 2H), 7.02 (m, 2H), 6.81 (dd, *J* = 8.0, 2.5 Hz, 1H), 3.06 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 151.1, 142.4, 142.4, 129.6, 128.8, 127.5, 127.3, 116.0, 111.8, 111.7, 40.9; IR (neat, cm⁻¹) 3030, 2881, 2801, 1598, 1491, 1354, 755, 698; Anal. Calc'd. for C₁₄H₁₅N: C, 85.24; H, 7.66. Found C, 85.21; H, 7.66.

Biphenyl-2-carbonitrile (Table 3, entry 1).^[8] Following general procedure A, 2-chlorobenzonitrile (138 mg, 1.00 mmol), tributyl(phenyl)stannane (359 μL, 1.10 mmol), pre-milled Pd(OAc)₂/XPhos (7.5 mg, 0.010 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 °C in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 88% yield (158 mg) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.75 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.63 (td, *J* = 8.0, 1.0 Hz, 1H), 7.48 (m, 7H); ¹³C NMR (100 MHz, CDCl₃) δ 145.6, 128.3, 133.9, 133.0, 130.3, 128.9, 128.9, 128.9, 127.7, 118.9, 111.4; IR (neat, cm⁻¹) 3064, 2963, 2223, 1261, 1076, 1027, 799, 758; Anal. Calc'd. for C₁₃H₉N: C, 87.12; H, 5.06. Found C, 86.87; H, 5.09.

2-(2-Thiophenyl)benzonitrile (Table 3, entry 2).^[8] Following general procedure A, 2-chlorobenzonitrile (138 mg, 1.00 mmol), tributyl(2-thiophenyl)stannane (349 μL, 1.10 mmol), pre-milled Pd(OAc)₂/XPhos (7.5 mg, 0.010 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 °C in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 92% yield (171 mg) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.72 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.62 (dd, *J* = 3.5, 1.0 Hz, 1H), 7.58 (m, 2H), 7.42 (dd, *J* = 5.0, 1.0 Hz, 1H), 7.37 (td, *J* = 8.0, 1.5 Hz, 1H), 7.14 (dd, *J* = 5.0, 3.5 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 139.6, 137.7, 134.5, 133.2, 129.9, 128.4, 127.8, 127.7, 127.5, 119.1, 110.1; IR (neat, cm⁻¹) 3107, 2224, 1595, 1482, 1444, 760, 702. Anal. Calc'd. for C₁₁H₇NS: C, 71.32; H, 3.81. Found C, 71.40; H, 3.59.

2',6'-Dimethoxybiphenyl-2-carbonitrile (Table 3, entry 3).^[9] Following general procedure A, 2-chlorobenzonitrile (138 mg, 1.00 mmol), tributyl(2,6-dimethoxyphenyl)stannane (403 μL, 1.10

mmol), pre-milled Pd(OAc)₂/XPhos (7.5 mg, 0.010 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 °C in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 76% yield (181 mg) as a white solid, mp 100-101 °C. ¹H NMR (400 MHz, CDCl₃) δ 7.71 (d, *J* = 7.5 Hz, 1H), 7.59 (td, *J* = 7.5, 1.5 Hz, 1H), 7.38 (m, 2H), 7.34 (t, *J* = 8.5 Hz, 1H), 6.66 (d, *J* = 8.5 Hz, 2H), 3.75 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 157.7, 138.9, 132.7, 132.3, 132.1, 130.6, 127.3, 118.9, 115.7, 115.7, 114.6, 104.3, 56.0; IR (neat, cm⁻¹) 2940, 2838, 2225, 1590, 1473, 1250, 1108. Anal. Calc'd. for C₁₅H₁₃NO₂: C, 75.30; H, 5.48. Found C, 75.19; H, 5.32.

Methyl 4-(2-thiophenyl)benzoate (Table 3, entry 4).^[10] Following general procedure A, methyl 4-chlorobenzoate (171 mg, 1.00 mmol), tributyl(2-thiophenyl)stannane (349 μL, 1.10 mmol), pre-milled Pd(OAc)₂/XPhos (7.5 mg, 0.010 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 °C in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 89% yield (195 mg) as a white solid, mp 140-141 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.02 (d, *J* = 8.5 Hz, 2H), 7.66 (d, *J* = 8.5 Hz, 2H), 7.41 (dd, *J* = 3.5, 1.0 Hz, 1H), 7.34 (dd, *J* = 5.0, 1.0 Hz, 1H), 7.10 (dd, *J* = 5.0, 3.5 Hz, 1H), 3.91 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 166.9, 143.1, 138.7, 130.4, 128.8, 128.5, 126.4, 125.6, 124.6, 52.3; IR (neat, cm⁻¹) 1712, 1282, 1261, 1113, 769, 724, 698. Anal. Calc'd. for C₁₂H₁₀O₂S: C, 66.03; H, 4.62. Found C, 66.00; H, 4.67.

Methyl 4-allylbenzoate (Table 3, entry 5).^[11] Following general procedure A, methyl 4-chlorobenzoate (171 mg, 1.00 mmol), allyltributylstannane (341 μL, 1.10 mmol), pre-milled Pd(OAc)₂/XPhos (7.5 mg, 0.010 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 °C in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 86% yield (152 mg) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.95 (d, *J* = 8.0 Hz, 1H), 7.24 (d, *J* = 8.0 Hz, 1H), 5.94 (ddt, *J* = 17.5, 9.0, 6.5 Hz, 1H), 5.09 (dd, *J* = 9.0, 1.0 Hz, 1H), 5.08 (dd, *J* = 17.6, 1.0 Hz, 1H), 3.88 (s, 3H), 3.42 (d, *J* = 6.5 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 167.3, 145.7, 136.6, 130.0, 128.8, 128.3, 116.8, 52.2, 40.4; IR (neat, cm⁻¹) 2952, 1723, 1611, 1435, 1280, 1178, 1109, 758, 707. Anal. Calc'd. for C₁₁H₁₂O₂: C, 74.98; H, 6.02.

6.86. Found C, 75.11; H, 6.90.

Methyl 4-(2-furanyl)benzoate (Table 3, entry 6).^[12] Following general procedure A, methyl 4-chlorobenzoate (171 mg, 1.00 mmol), tributyl(2-furanyl)stannane (346 μ L, 1.10 mmol), pre-milled Pd(OAc)₂/XPhos (7.5 mg, 0.010 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 °C in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 97% yield (196 mg) as a white solid, mp 120-121 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.03 (d, *J* = 8.5 Hz, 2H), 7.71 (d, *J* = 8.5 Hz, 2H), 7.50 (d, *J* = 1.5 Hz, 1H), 6.77 (d, *J* = 3.5 Hz, 1H), 6.49 (dd, *J* = 3.5, 1.5 Hz, 1H), 3.91 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 166.9, 153.0, 143.2, 134.8, 130.2, 128.6, 123.5, 112.1, 107.4, 52.2; IR (neat, cm⁻¹) 1718, 1282, 1114, 772, 743, 701. Anal. Calc'd. for C₁₂H₁₀O₃: C, 71.28; H, 4.98. Found C, 71.09; H, 4.84.

Methyl 2',4',6'-trimethylbiphenyl-4-carboxylate (Table 3, entry 7).^[13] Following general procedure A, methyl 4-chlorobenzoate (171 mg, 1.00 mmol), tributyl(2,4,6-mesityl)stannane (401 μ L, 1.10 mmol), pre-milled Pd(OAc)₂/XPhos (7.5 mg, 0.010 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 °C in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 94% yield (238 mg) as a white solid, mp 125-126 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.08 (d, *J* = 8.0 Hz, 2H), 7.21 (d, *J* = 8.0 Hz, 2H), 6.93 (s, 2H), 3.92 (s, 3H), 2.38 (s, 3H), 1.96 (s, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 167.3, 146.5, 138.2, 137.3, 135.7, 129.9, 129.7, 128.7, 128.4, 52.3, 21.2, 20.8; IR (neat, cm⁻¹) 2948, 1720, 1437, 1285, 1116, 1099, 858. Anal. Calc'd. for C₁₇H₁₈O₂: C, 80.28; H, 7.13. Found C, 80.02; H, 7.09.

2,4,6-Trimethyl-2'-(trifluoromethyl)biphenyl (Table 3, entry 8). Following general procedure A, 2-chlorobenzotrifluoride (131 μ L, 1.00 mmol), tributyl(2,4,6-mesityl)stannane (401 μ L, 1.10 mmol), pre-milled Pd(OAc)₂/XPhos (15.0 mg, 0.020 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 °C in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 59% yield (157 mg) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.77 (d, *J* = 7.5 Hz, 1H), 7.58 (t, *J* = 7.5 Hz, 1H), 7.46 (t, *J* = 7.5 Hz, 1H), 7.16 (d, *J* =

7.5 Hz, 1H), 6.92 (s, 2H), 2.33 (s, 3H), 1.90 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 140.3, 137.3, 136.3, 136.0, 132.2, 131.7, 128.9, 127.9, 137.4, 126.5 (q, $J = 5.0$ Hz), 122.8, 21.3, 20.6; IR (neat, cm^{-1}) 3442, 2923, 1315, 1172, 1128, 1035, 763. Anal. Calc'd. for $\text{C}_{16}\text{H}_{15}\text{F}_3$: C, 72.71; H, 5.72. Found C, 72.56; H, 5.73.

4'-Fluoro-2,6-dimethoxybiphenyl (Table 3, entry 9). Following general procedure A, 1-chloro-4-fluorobenzene (107 μL , 1.00 mmol), tributyl(2,6-dimethoxyphenyl)stannane (403 μL , 1.10 mmol), pre-milled $\text{Pd}(\text{OAc})_2/\text{XPhos}$ (15.0 mg, 0.020 mmol) and CsF (334 mg, 2.20 mmol) were heated to 80 $^\circ\text{C}$ in DME with stirring for 4 h. The crude product was adsorbed on silica, loaded onto the top of a Biotage 25M column and purified by flash chromatography using a Biotage SP4 to provide the titled compound in 93% yield (215 mg) as a white solid, mp 127-128 $^\circ\text{C}$. ^1H NMR (400 MHz, CDCl_3) δ 7.28 (m, 3H), 7.07 (t, $J = 8.5$ Hz, 2H), 6.64 (d, $J = 8.5$ Hz, 2H), 3.72 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.9 (d, $J = 244$ Hz), 157.8, 132.7 (d, $J = 8$ Hz), 130.0 (d, $J = 3$ Hz), 129.0, 118.5, 114.9 (d, $J = 21$ Hz), 104.3, 56.1; IR (neat, cm^{-1}) 2835, 1585, 1517, 1472, 1245, 1114, 830, 725, 711. Anal. Calc'd. for $\text{C}_{14}\text{H}_{13}\text{FO}_2$: C, 72.40; H, 5.64. Found C, 72.38; H, 5.65.

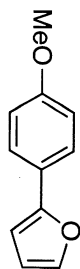
3-Phenylpyridine (Table 3, entry 10).^[14] Modification to general procedure A, 3-chloropyridine (95 μL , 1.00 mmol), tributyl(phenyl)stannane (393 μL , 1.20 mmol), pre-milled $\text{Pd}(\text{OAc})_2:\text{XPhos}$ (1:3 ratio, 33.0 mg, 0.020 mmol) and CsF (334 mg, 2.20 mmol) were heated to 100 $^\circ\text{C}$ in 1,4-dioxane with stirring for 4 h. The crude product was purified by flash chromatography using a Biotage SP4 and 25M cartridge to provide the titled compound in 91% yield (141 mg) as a colorless oil. ^1H NMR (400 MHz, CDCl_3) δ 8.82 (d, $J = 1.5$ Hz, 1H), 8.55 (dd, $J = 4.5, 1.5$ Hz, 1H), 7.81 (dt, $J = 7.5, 1.5$ Hz, 1H), 7.53 (d, $J = 7.5$ Hz, 2H), 7.43 (t, $J = 7.5$ Hz, 2H), 7.36 (t, $J = 7.5$ Hz, 1H), 7.30 (dd, $J = 7.5, 4.5$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 148.5, 148.4, 137.8, 136.6, 134.3, 129.1, 128.1, 127.2, 123.6; IR (neat, cm^{-1}) 3031, 1581, 1473, 1450, 1407, 1005, 754, 712.

6-Phenylquinoline (Table 3, entry 11).^[15] Modification to general procedure A, 6-chloroquinoline (164 mg, 1.00 mmol), tributyl(phenyl)stannane (393 μL , 1.20 mmol), pre-milled $\text{Pd}(\text{OAc})_2:\text{XPhos}$ (1:3 ratio, 33.0 mg, 0.020 mmol) and CsF (334 mg, 2.20 mmol) were heated to 100 $^\circ\text{C}$ in 1,4-dioxane with stirring for 4 h. The crude product was purified by flash

chromatography using a Biotage SP4 and 25M cartridge to provide the titled compound in 96% yield (197 mg) as a white solid, mp 110-112 °C. ¹H NMR (400 MHz, CDCl₃) δ 8.87 (dd, J = 4.0, 1.5 Hz, 1H), 8.15 (d, J = 8.5 Hz, 1H), 8.08 (d, J = 8.5 Hz, 1H), 7.92 (m, 2H), 7.65 (d, J = 7.5 Hz, 2H), 7.44 (t, J = 7.5 Hz, 2H), 7.34 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 150.4, 147.7, 140.2, 139.2, 136.2, 129.9, 129.2, 129.0, 128.4, 127.8, 127.5, 125.5, 121.5; IR (neat, cm⁻¹) 3060, 1576, 1460, 1352, 1326, 1122, 891, 844, 784, 765, 700.

References

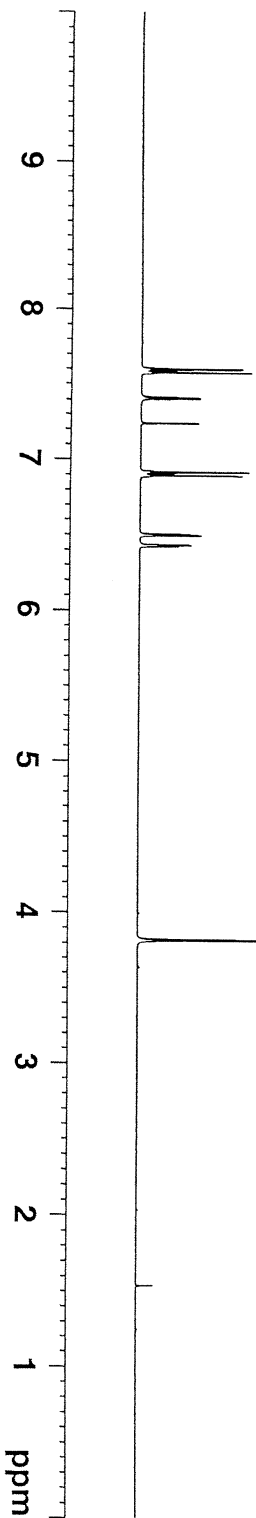
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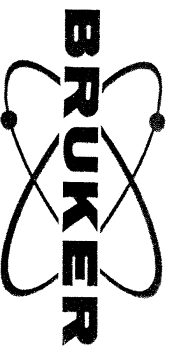
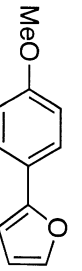


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 FIDRES 0.126314 Hz
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 RG 203.2
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 DE 6.00 usec
 TE 293.2 K
 D1 1.00000000 sec
 TD0 1

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 SF 400.1300178 MHz
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Current Data Parameters
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 PROCNO 1

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 Time 14.06

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 NS 40
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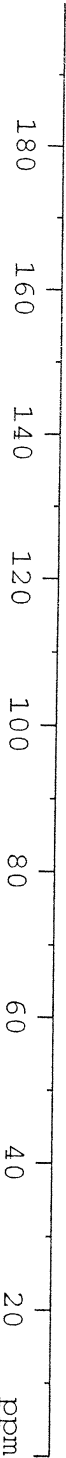
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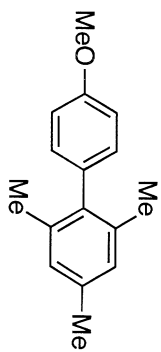
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 PL13 19.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
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 WDW EM
 SSB 0
 LB 1.00 Hz
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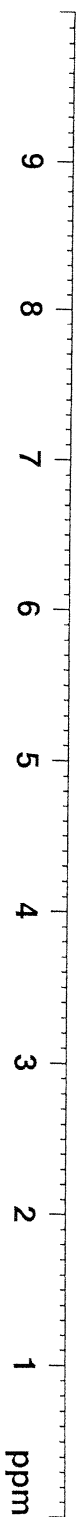


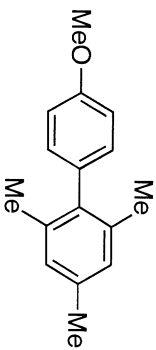
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 SOLVENT CDCl3
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 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 256
 DW 60.400 usec
 DE 6.00 usec
 TE 291.2 K
 D1 1.00000000 sec
 TD0 1

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 P1 14.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
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Current Data Parameters
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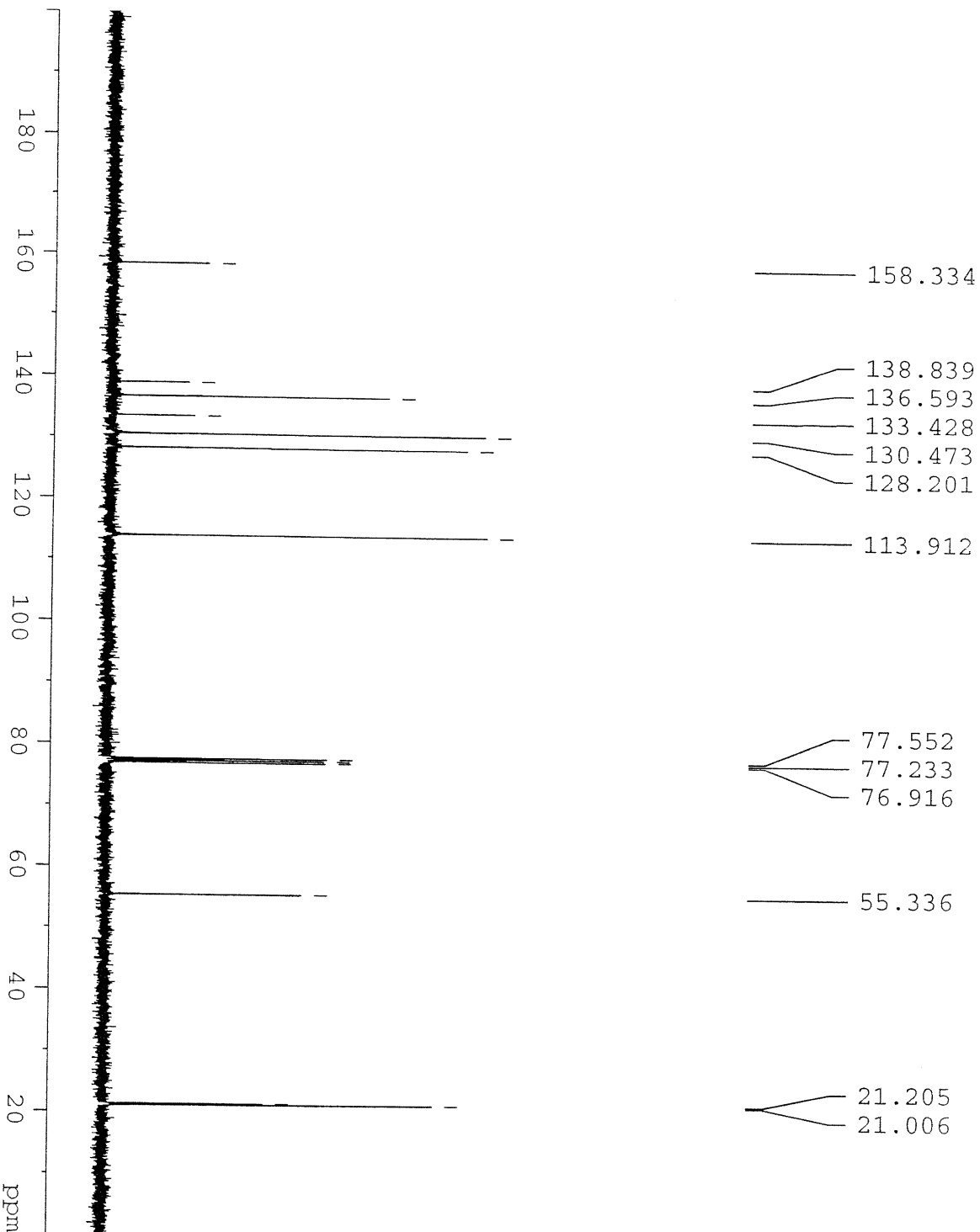
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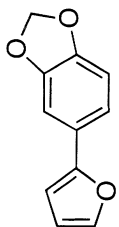
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 RG 2896.3
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 DE 6.00 usec
 TE 292.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

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 SFO1 100.6228298 MHz
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 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
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F2 - Processing Parameters

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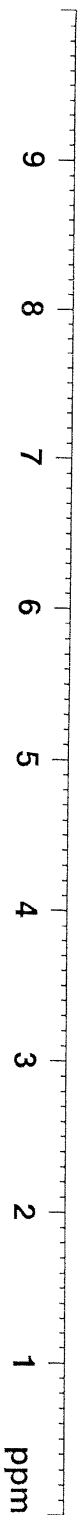


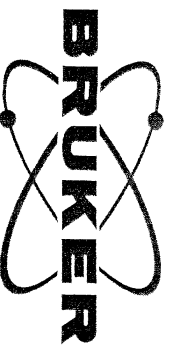
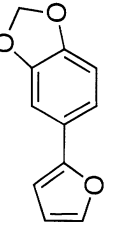
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 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 287.4
 DW 60.400 usec
 DE 6.00 usec
 TE 291.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
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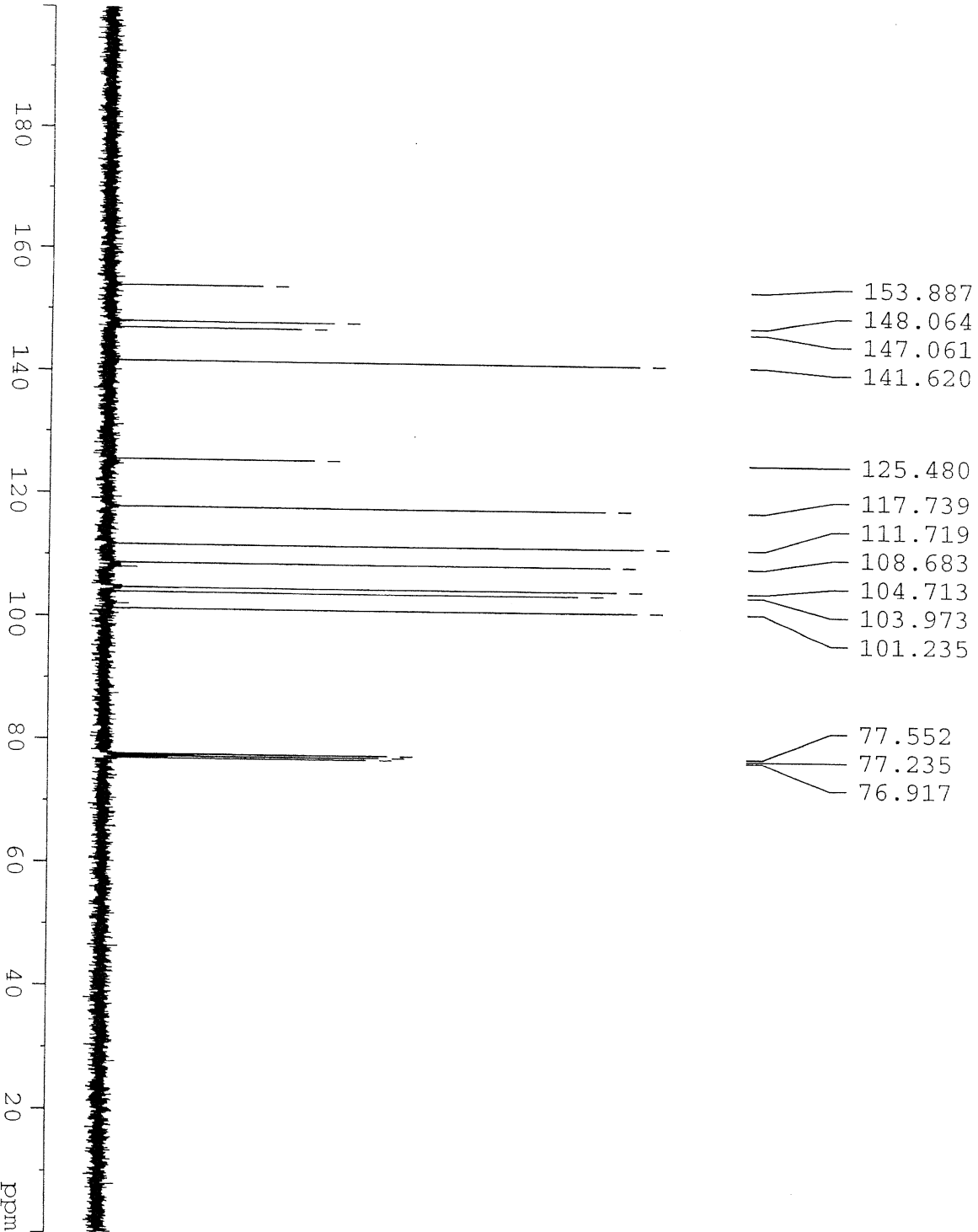
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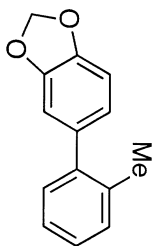
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 TD 65536
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 NS 30
 DS 4
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 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 4096
 DW 20.850 usec
 DE 6.00 usec
 TE 292.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

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 P1 9.38 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
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 NUC2 1H
 PCPD2 90.00 usec
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 PL13 19.00 dB
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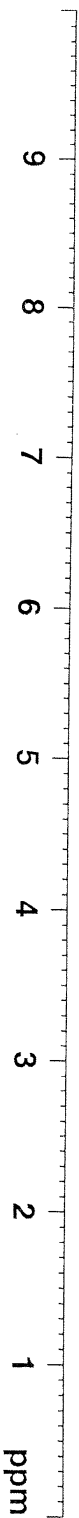


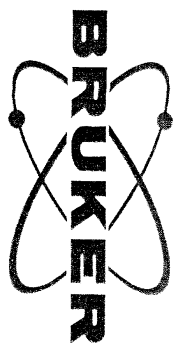
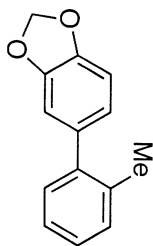
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 SOLVENT CDCl3
 NS 16
 DS 2
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 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 362
 DW 60.400 usec
 DE 6.00 usec
 TE 293.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
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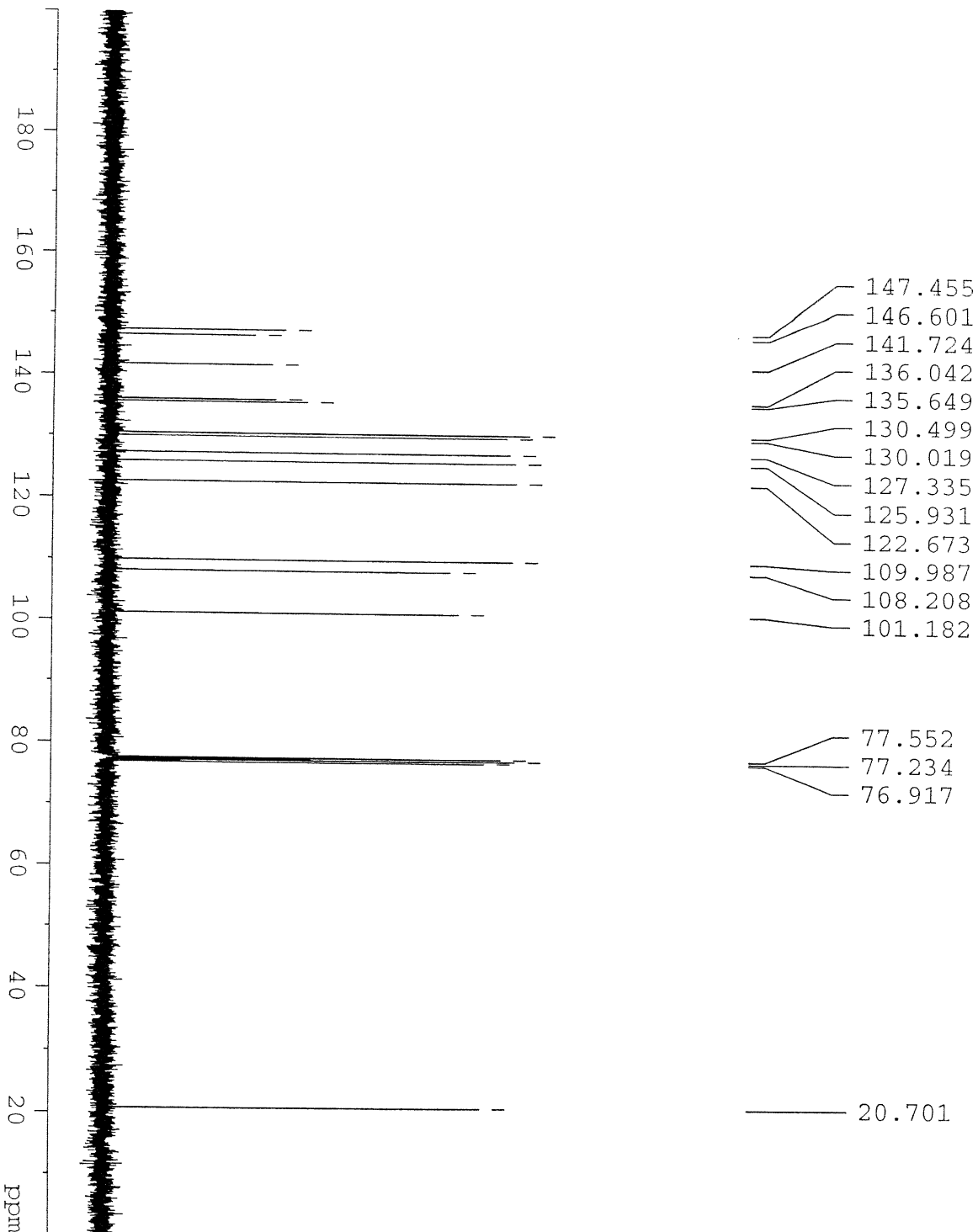
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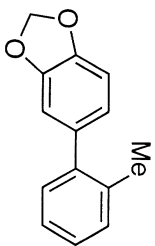
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 NS 25
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 FIDRES 0.365918 Hz
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 RG 5792.6
 DW 20.850 usec
 DE 6.00 usec
 TE 293.2 K
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 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
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 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
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 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
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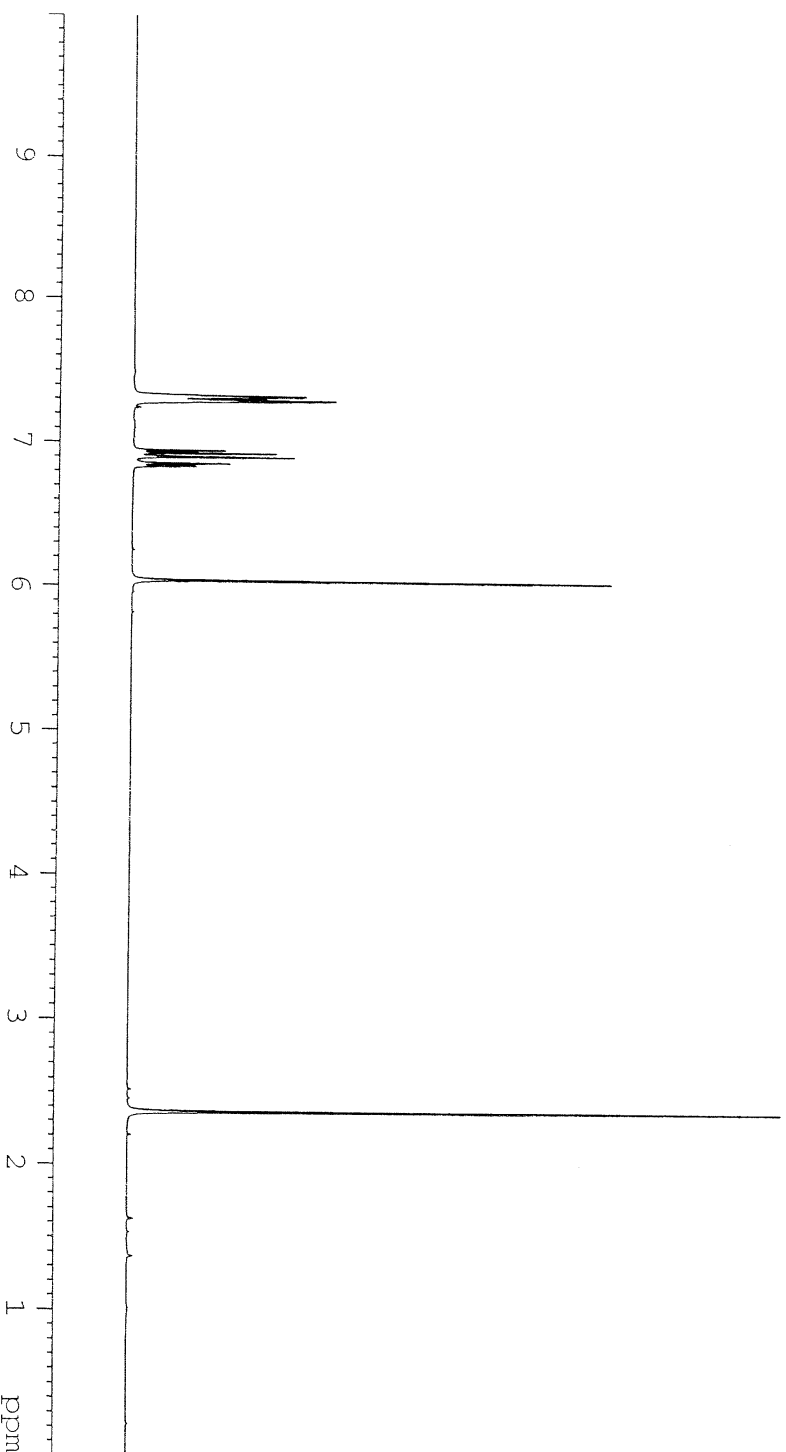


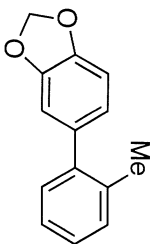
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 SOLVENT
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 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 32
 DW 60.400 usec
 DE 6.00 usec
 TE 292.2 K
 D1 1.00000000 sec
 TD0 1

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 P1 15.07 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
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 WDW EM
 SSB 0
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Current Data Parameters
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 EXPNO 13
 PROCNO 1

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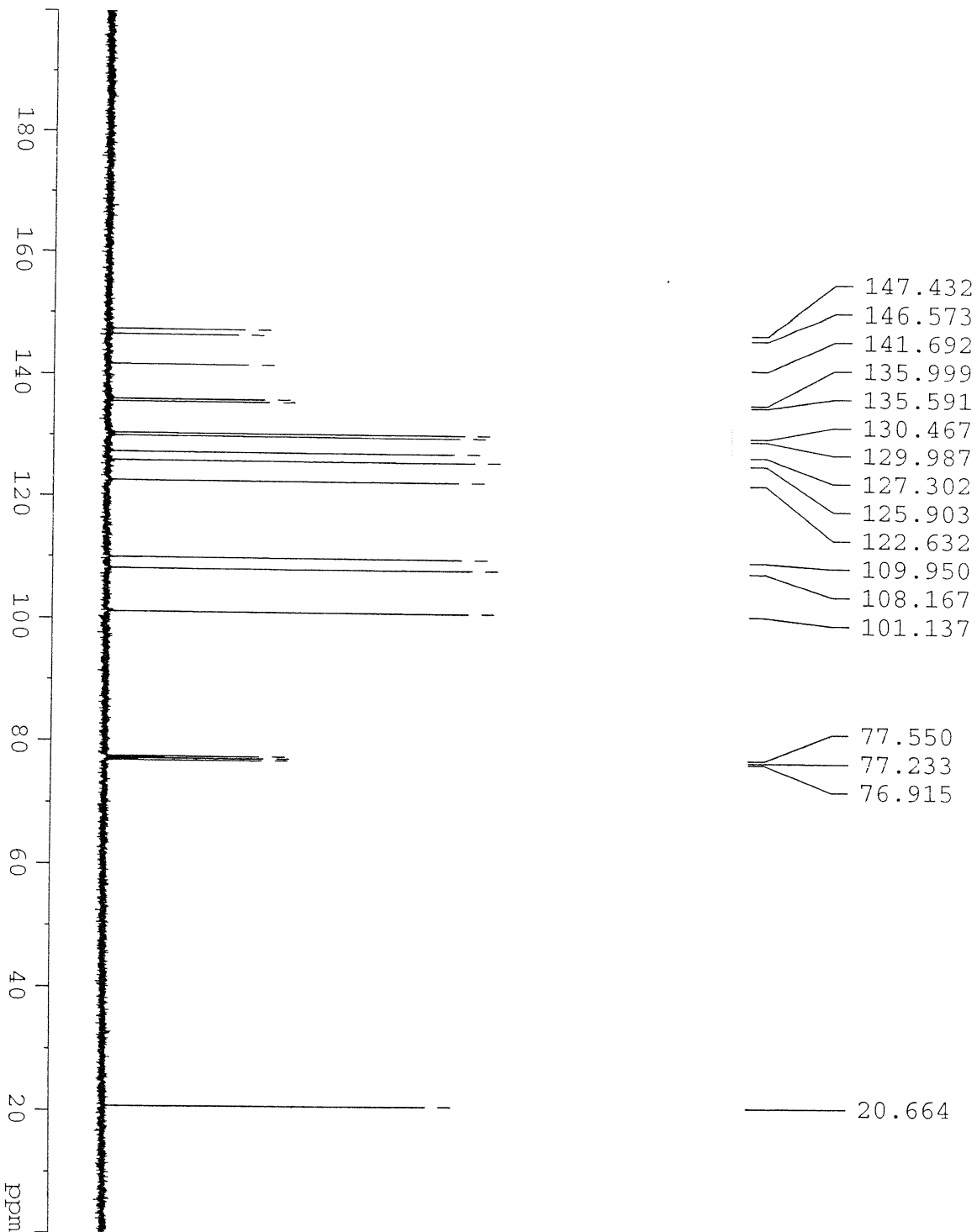
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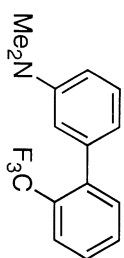
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 SFO1 100.6228298 MHz

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 PL12 14.52 dB
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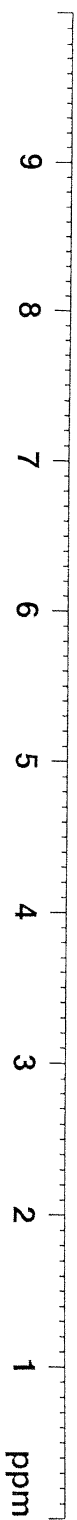


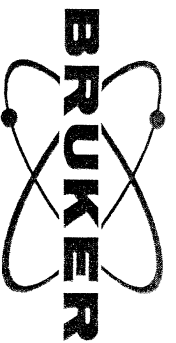
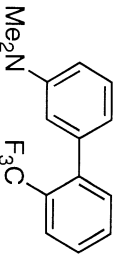


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 SOLVENT CDCl3
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 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 362
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 DE 6.00 usec
 TE 292.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
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 P1 14.00 usec
 PL1 0.00 dB
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 SI 65536
 SF 400.1300175 MHz
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Current Data Parameters
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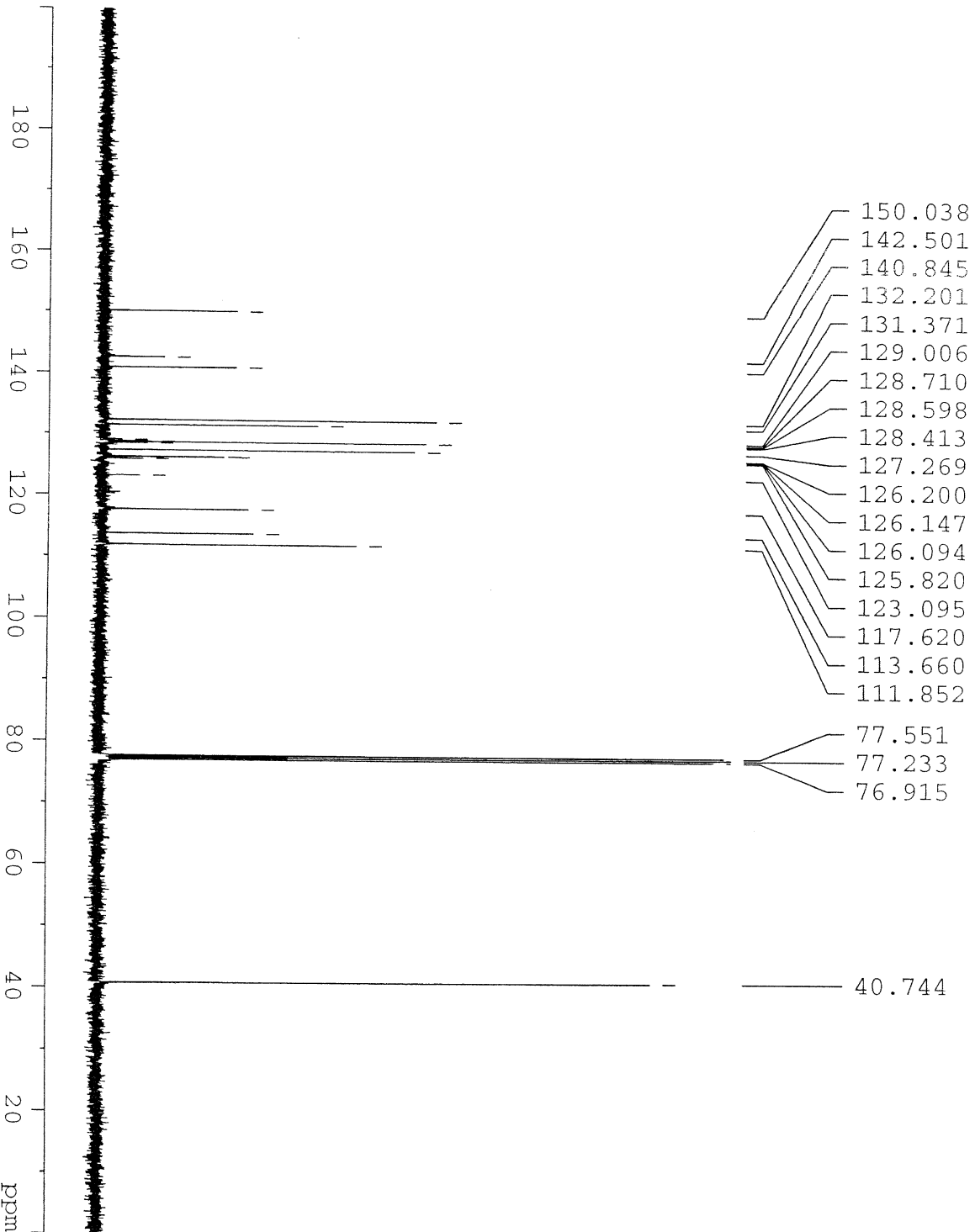
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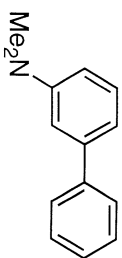
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===== CHANNEL f1 =====
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 P1 9.38 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
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 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SFO2 400.1316005 MHz

F2 - Processing Parameters
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 WDW EM
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 LB 1.00 Hz
 GB 0
 PC 1.40



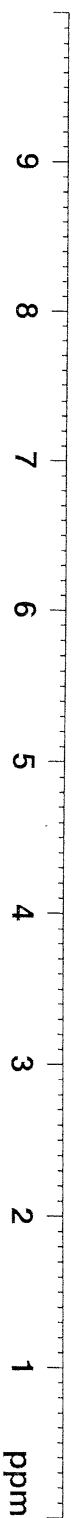


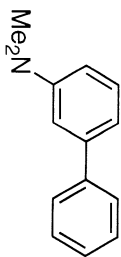
Current Data Parameters
 NAME JN2-289-010
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071020
 Time 10.18
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT
 NS 8
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 45.3
 DW 60.400 usec
 DE 6.00 usec
 TE 293.2 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 1H
 P1 15.07 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1300177 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
 NAME JN2-289-010
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071020
 Time 10.20

INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 20
 DS 2

SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 13004

DW 20.850 usec
 DE 6.00 usec
 TE 293.2 K

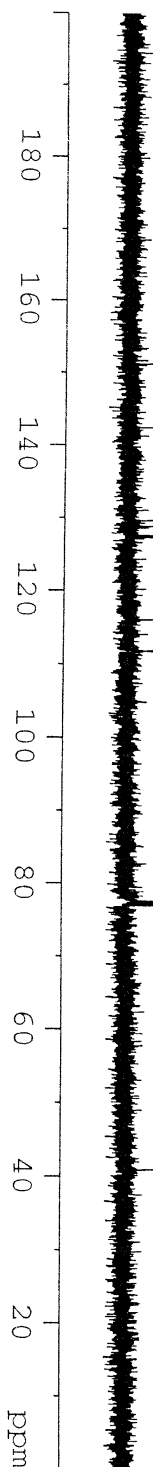
D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

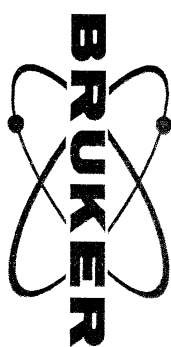
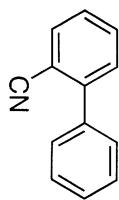
===== CHANNEL f1 =====
 NUC1 13C
 P1 8.75 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -1.00 dB
 PL12 14.52 dB
 PL13 18.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 65536
 SF 100.6127634 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

151.082
 142.431
 142.417
 129.590
 128.773
 127.514
 127.263
 115.991
 111.790
 111.713
 77.549
 77.231
 76.913
 40.875



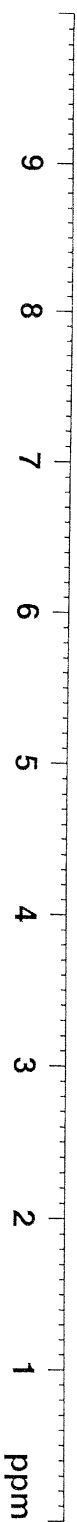


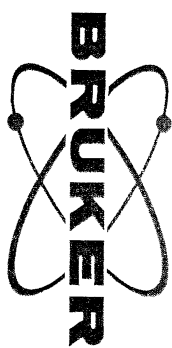
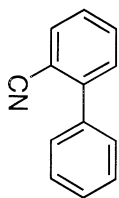
Current Data Parameters
 NAME JN2-276-010
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071014
 Time 12.55
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 362
 DW 60.400 usec
 DE 6.00 usec
 TE 291.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing Parameters
 SI 65536
 SF 400.1300175 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
 NAME JN2-276-010
 EXPNO 13
 PROCNO 1

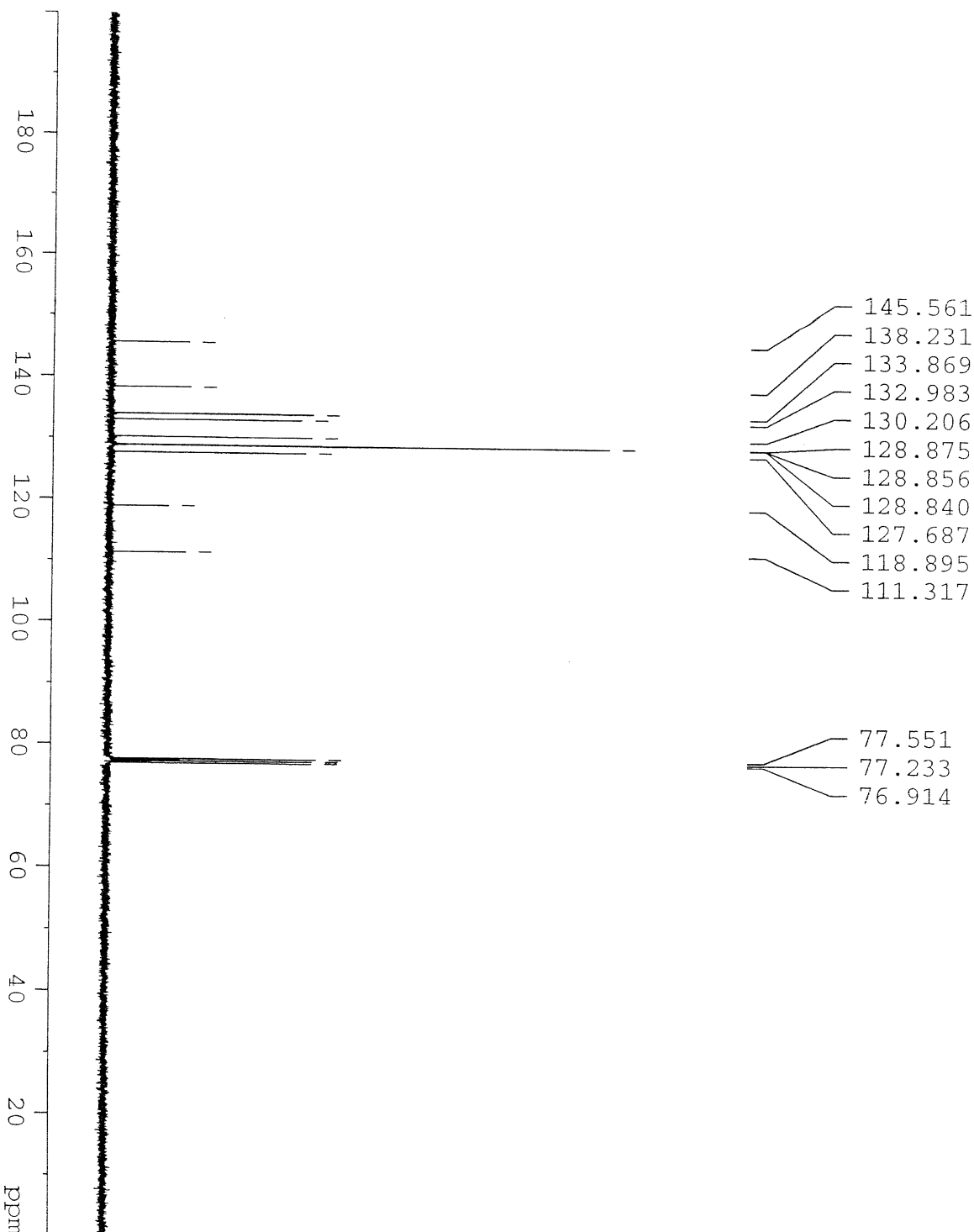
F2 - Acquisition Parameters
 Date_ 20071014
 Time 13.53

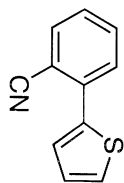
INSTRUM spect
 PROBD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 41
 DS 2
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 2896.3
 DW 20.850 usec
 DE 6.00 usec
 TE 292.2 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.45 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 65536
 SF 100.6127617 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



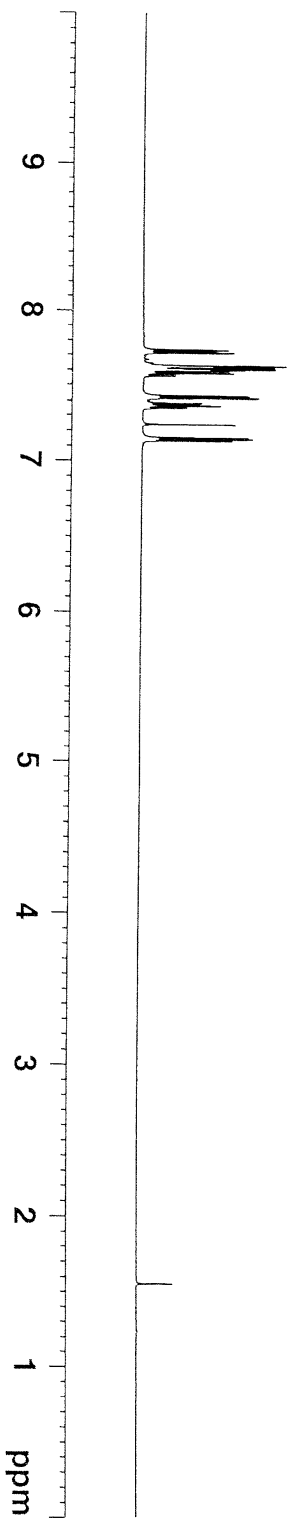


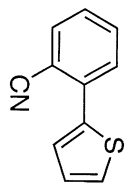
Current Data Parameters
 NAME JN2-270-010
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071012
 Time 15.30
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 181
 DW 60.400 usec
 DE 6.00 usec
 TE 293.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.07 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1300177 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
 NAME JN2-261-020
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071014
 Time 13.45

INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 76
 DS 2

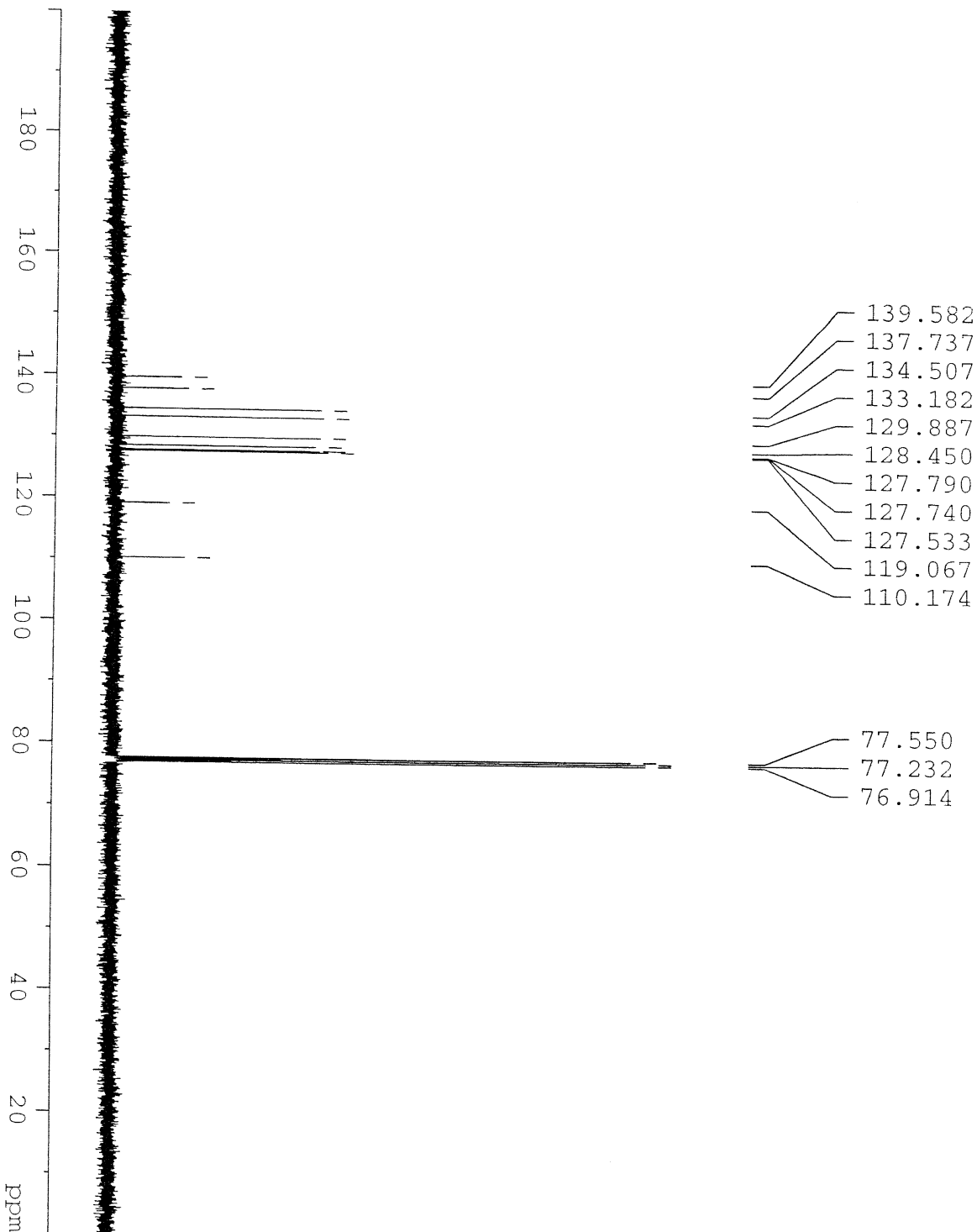
SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 1625.5
 DW 20.850 usec
 DE 6.00 usec
 TE 292.2 K

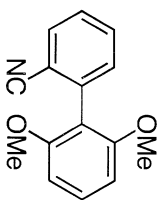
D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.45 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 65536
 SF 100.6127540 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



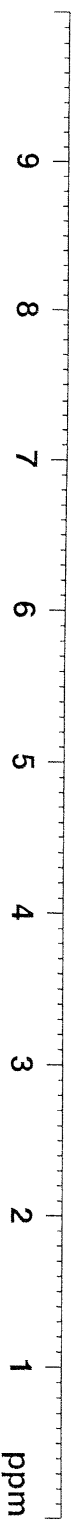


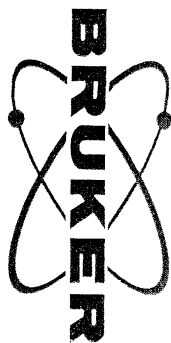
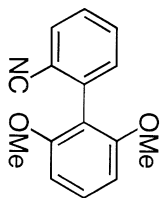
Current Data Parameters
 NAME JN2-281-010
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071016
 Time 21.32
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 203.2
 DW 60.400 usec
 DE 6.00 usec
 TE 291.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing Parameters
 SI 65536
 SF 400.1300174 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





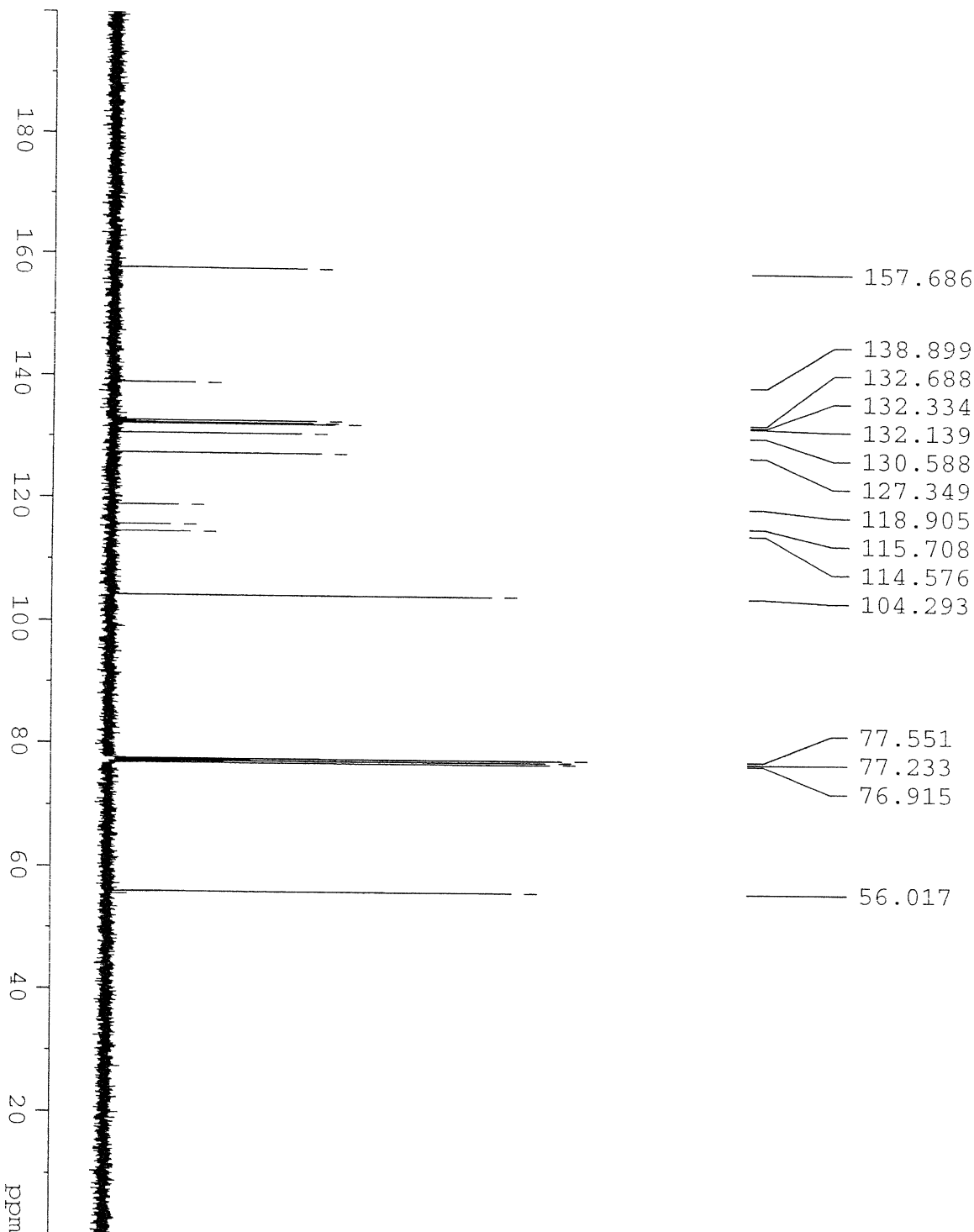
Current Data Parameters
 NAME JN2-281-010
 EXPNO 13
 PROCNO 1

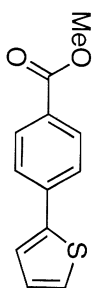
F2 - Acquisition Parameters
 Date_ 20071025
 Time 18.35
 INSTRUM spect
 PROBD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 100
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 3251
 DW 20.850 usec
 DE 6.00 usec
 TE 292.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.38 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127556 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



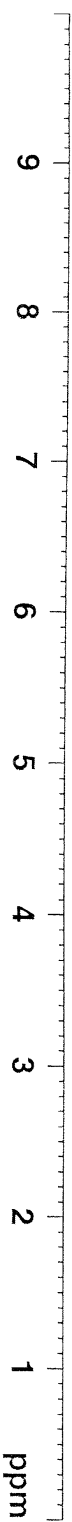


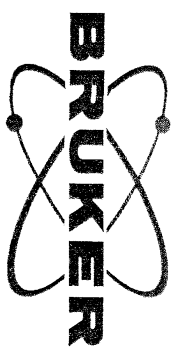
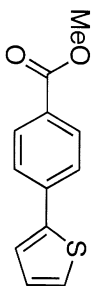
Current Data Parameters
 NAME JN2-285-010
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071025
 Time 18.14
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 90.5
 DW 60.400 usec
 DE 6.00 usec
 TE 291.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1300170 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
 NAME JN2-285-010
 EXPNO 13
 PROCNO 1

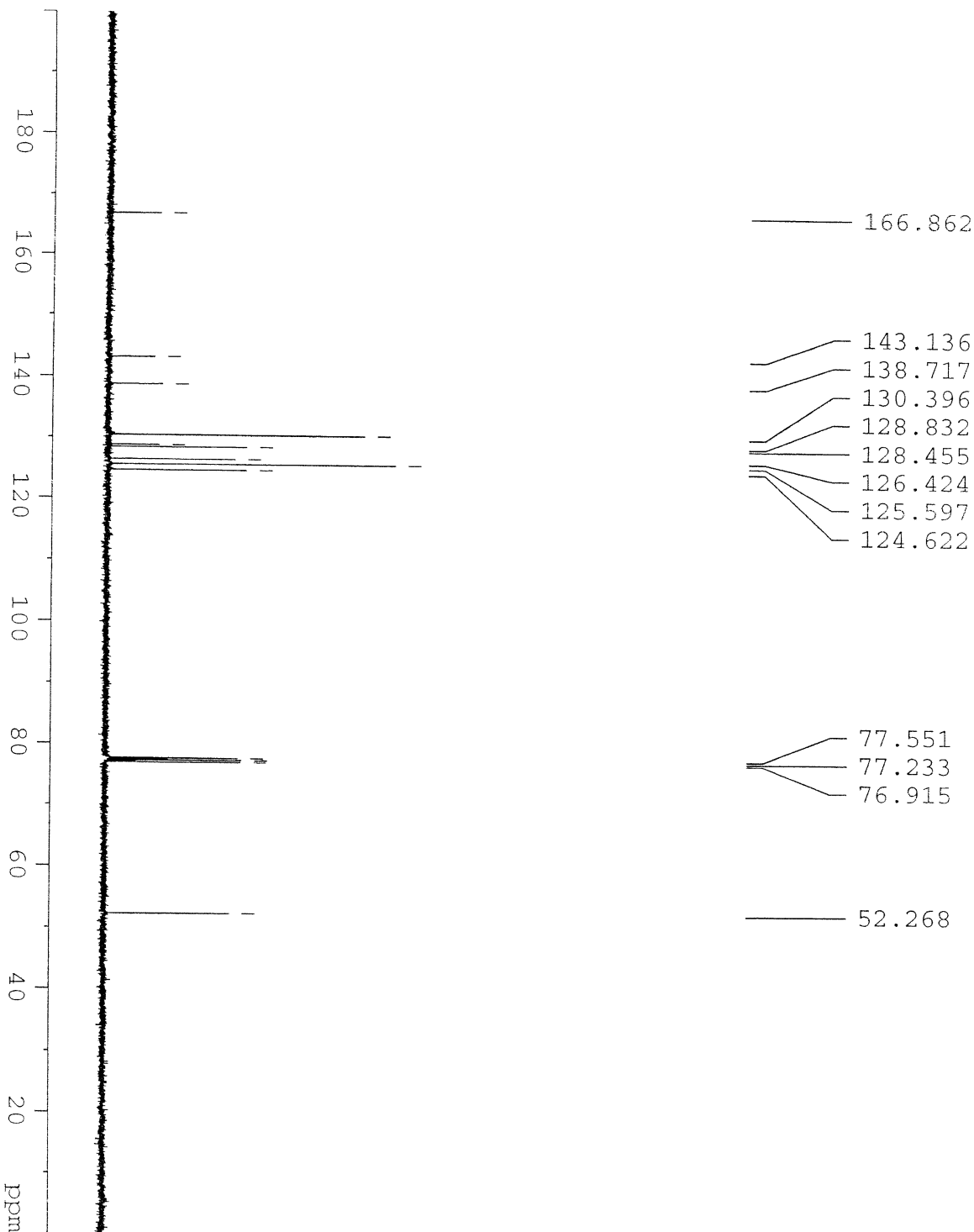
F2 - Acquisition Parameters
 Date_ 20071025
 Time 18.17

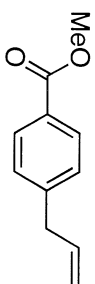
INSTRUM spect
 PROBD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 30
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 2896.3
 DW 20.850 usec
 DE 6.00 usec
 TE 291.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.38 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127596 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



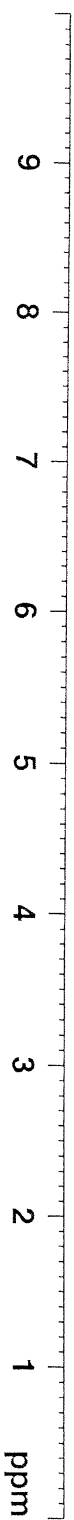


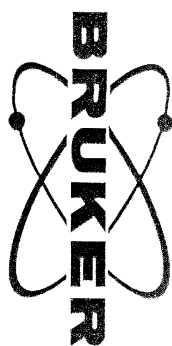
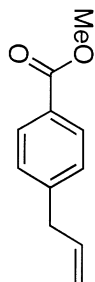
Current Data Parameters
 NAME JN2-294-010
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071105
 Time 19.33
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 256
 DW 60.400 usec
 DE 6.00 usec
 TE 292.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1300173 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
 NAME JN3-019-010
 EXPNO 13
 PROCNO 1

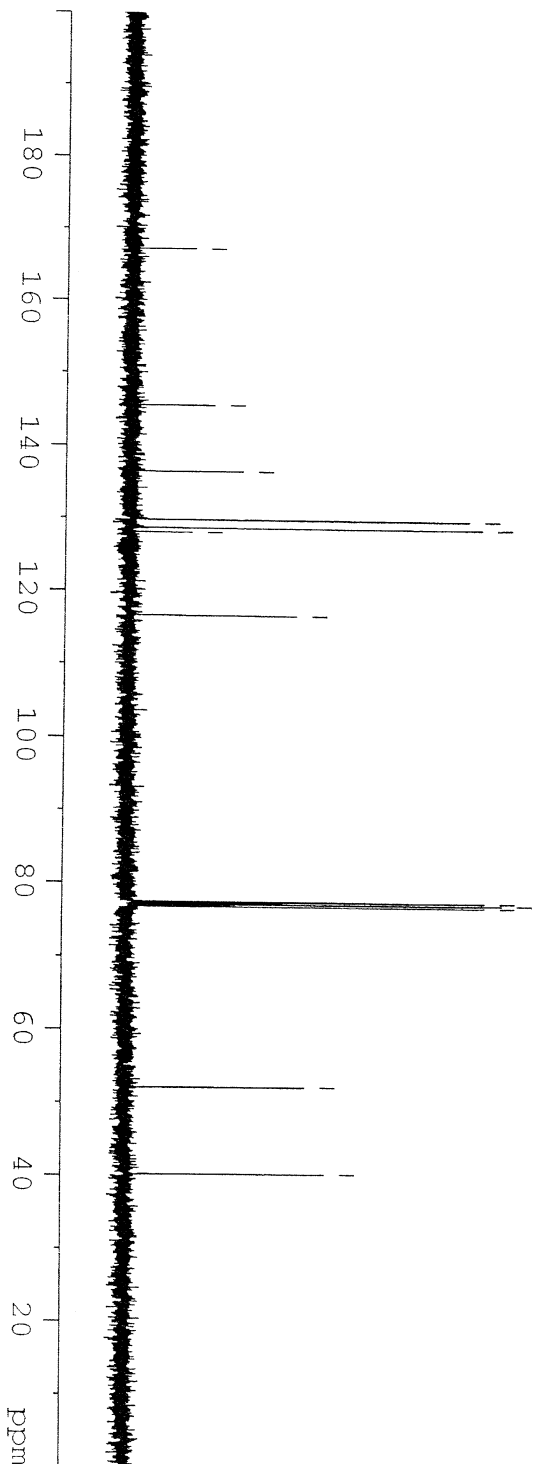
F2 - Acquisition Parameters
 Date_ 20071110
 Time 12.31
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 20
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 4096
 DW 20.850 usec
 DE 6.00 usec
 TE 293.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

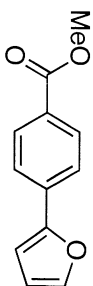
===== CHANNEL f1 =====
 NUC1 13C
 P1 9.38 usec
 PL1 0.00 dB
 SF01 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SF02 400.1316005 MHz

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

167.125
 145.508
 136.417
 129.781
 128.640
 128.078
 116.617
 77.379
 77.061
 76.743
 52.039
 40.168



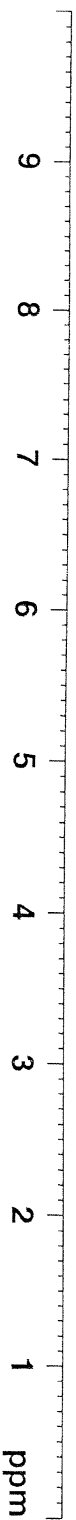


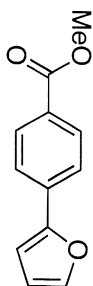
Current Data Parameters
 NAME JN2-283-010
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071016
 Time 21.43
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 322.5
 DW 60.400 usec
 DE 6.00 usec
 TE 291.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing Parameters
 SI 65536
 SF 400.1300175 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





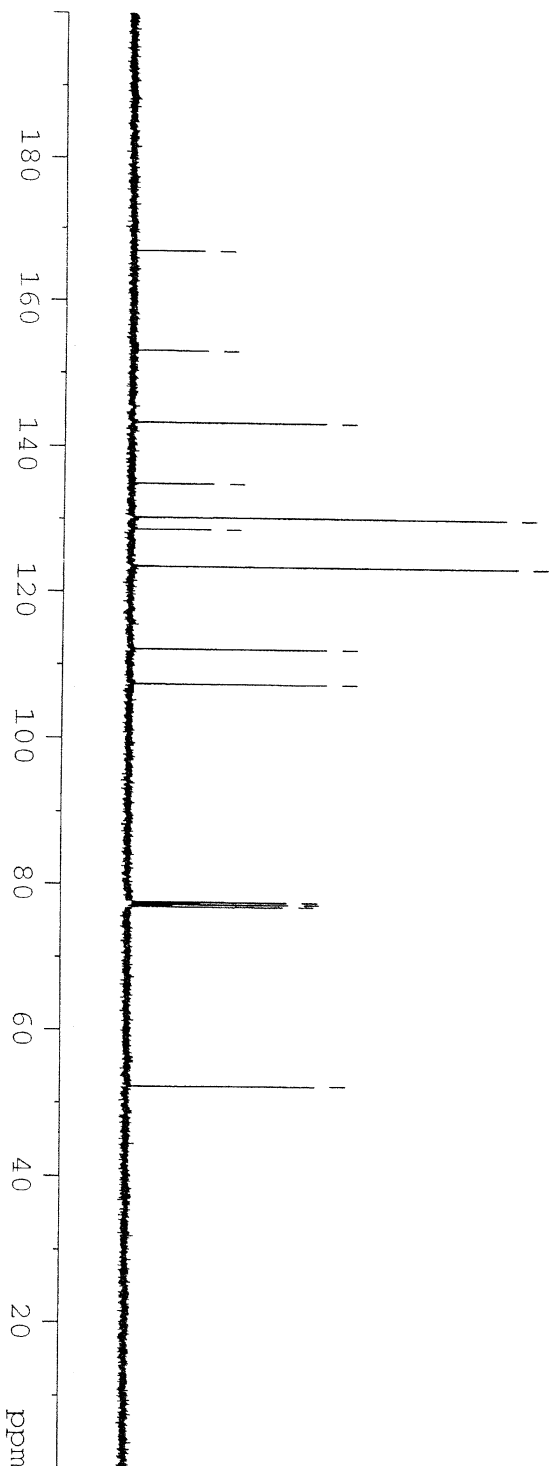
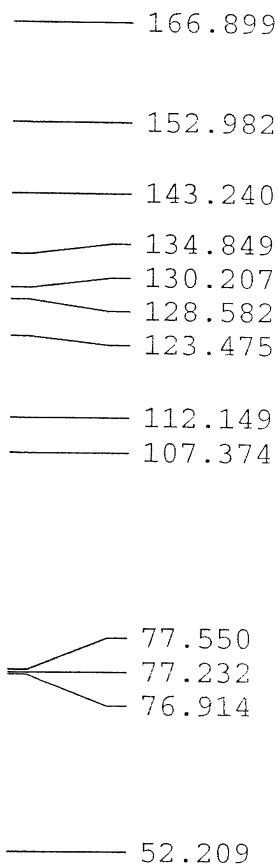
Current Data Parameters
 NAME JN2-271-010
 EXPNO 13
 PROCNO 1

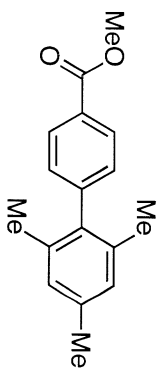
F2 - Acquisition Parameters
 Date_ 20071014
 Time 13.59
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 39
 DS 2
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 2580.3
 DN 20.850 usec
 DE 6.00 usec
 TE 292.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.45 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 65536
 SF 100.6127595 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



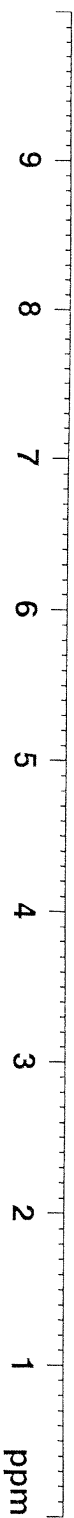


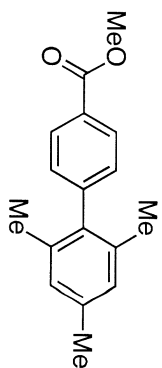
Current Data Parameters
 NAME JN2-275-010
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071014
 Time 13.00
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 287.4
 DW 60.400 usec
 DE 6.00 usec
 TE 291.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1300174 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





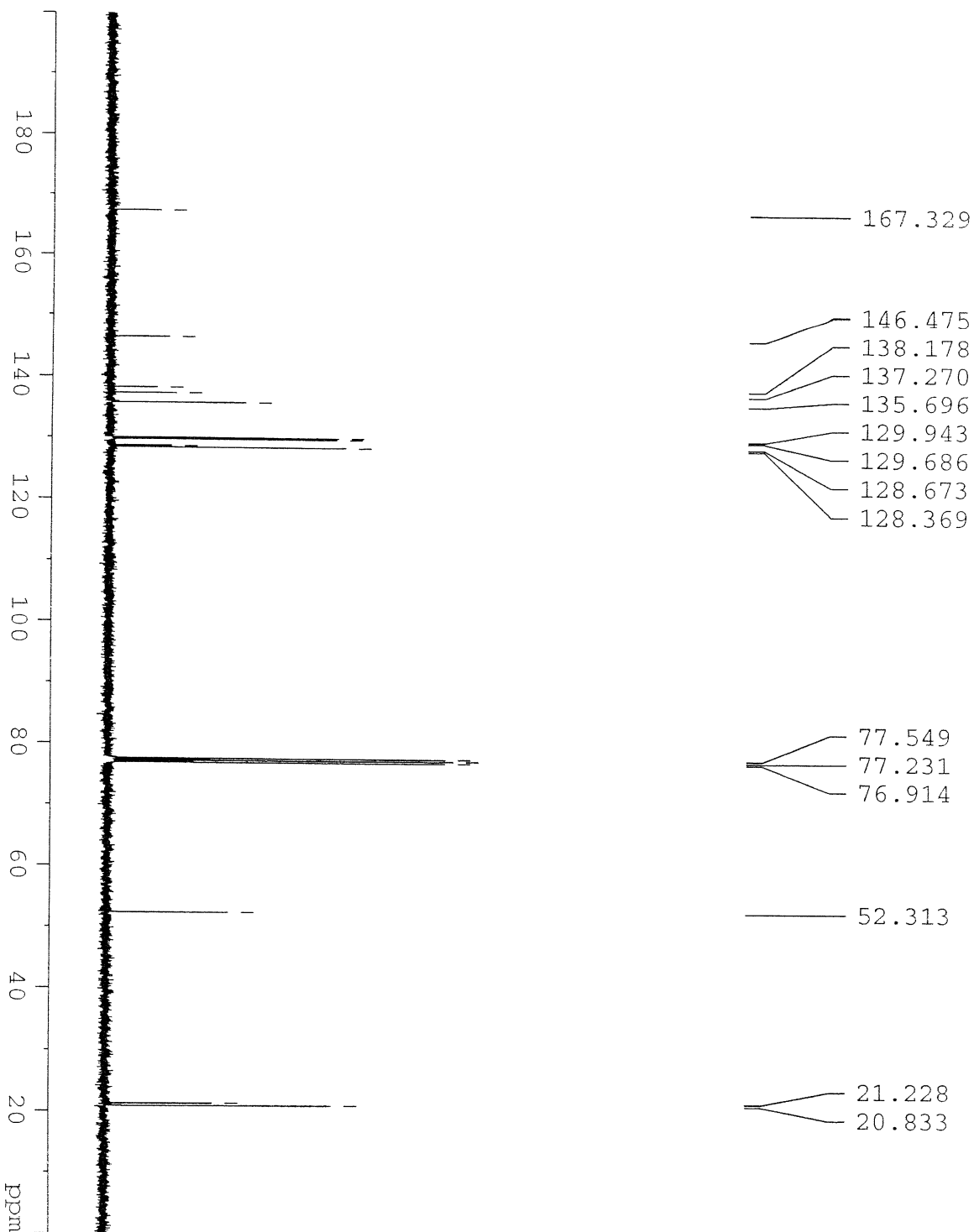
Current Data Parameters
NAME JN2-262-010
EXPNO 13
PROCNO 1

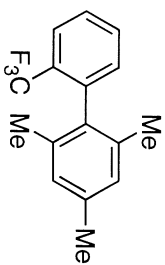
F2 - Acquisition Parameters
Date_ 20071014
Time 13.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 77
DS 2
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 2580.3
DW 20.850 usec
DE 6.00 usec
TE 291.2 K
D1 2.0000000 sec
d11 0.0300000 sec
DELTA 1.8999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.45 usec
PL1 0.00 dB
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL13 19.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127536 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



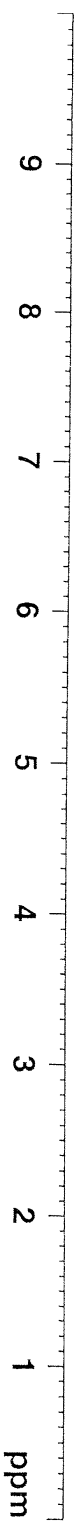


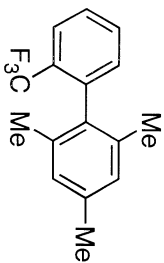
Current Data Parameters
 NAME JN3-016-010
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071110
 Time 12.39
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 256
 DW 60.400 usec
 DE 6.00 usec
 TE 293.2 K
 D1 1.0000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing Parameters
 SI 65536
 SF 400.1300173 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





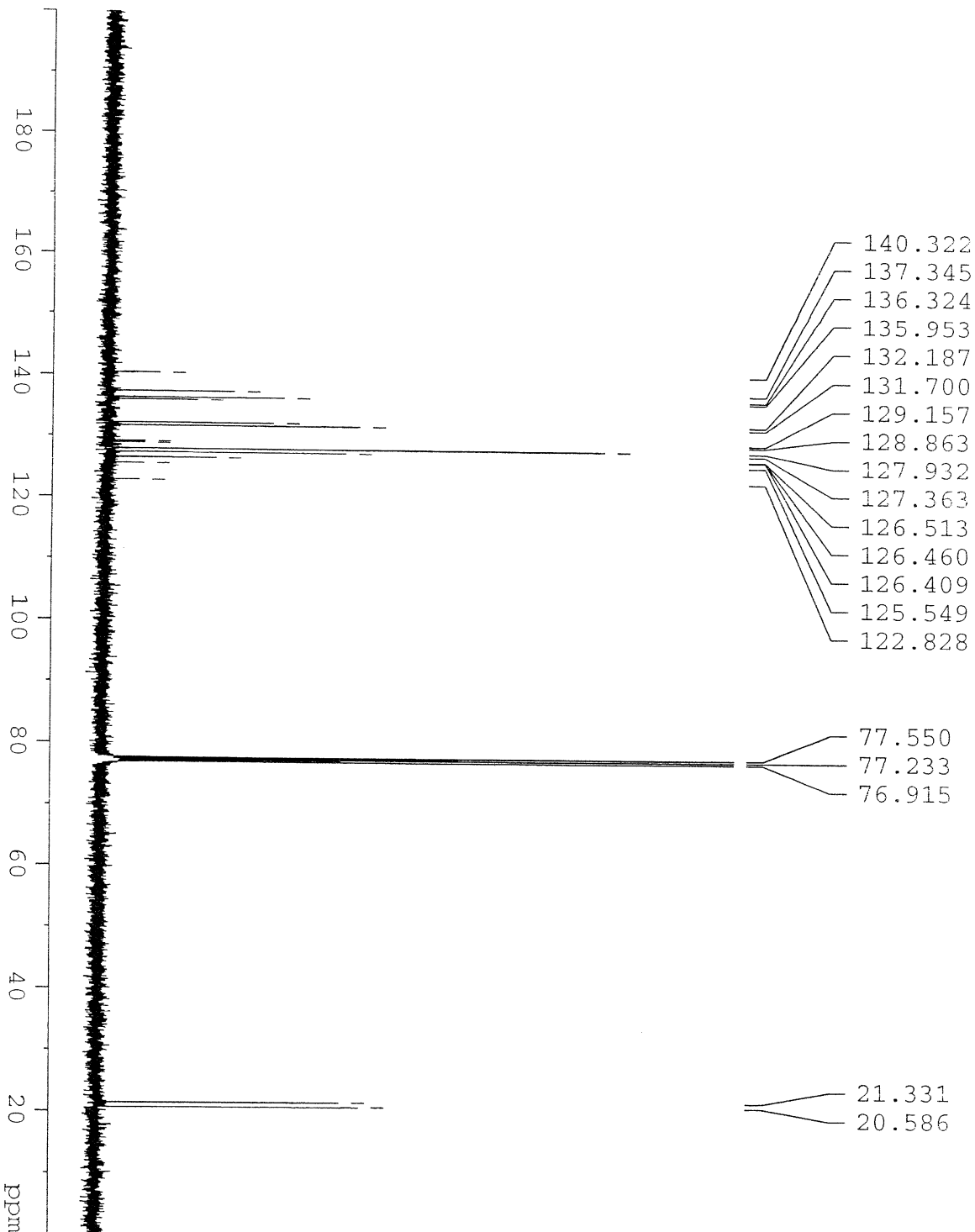
Current Data Parameters
 NAME JN3-016-010
 EXPNO 13
 PROCNO 1

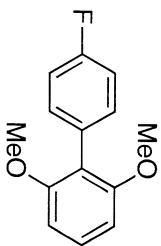
F2 - Acquisition Parameters
 Date_ 20071110
 Time 11.44
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 285
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 3649.1
 DW 20.850 usec
 DE 6.00 usec
 TE 293.2 K
 D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.38 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127498 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



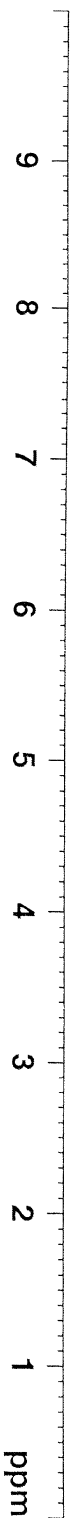


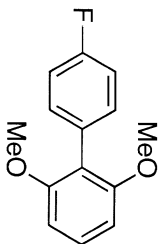
Current Data Parameters
 NAME JN3-018-010
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20071110
 Time 12.56
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 228.1
 DW 60.400 usec
 DE 6.00 usec
 TE 293.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing Parameters
 SI 65536
 SF 400.1300220 MHz
 WDM EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
 NAME JN3-018-010
 EXPNO 13
 PROCNO 1

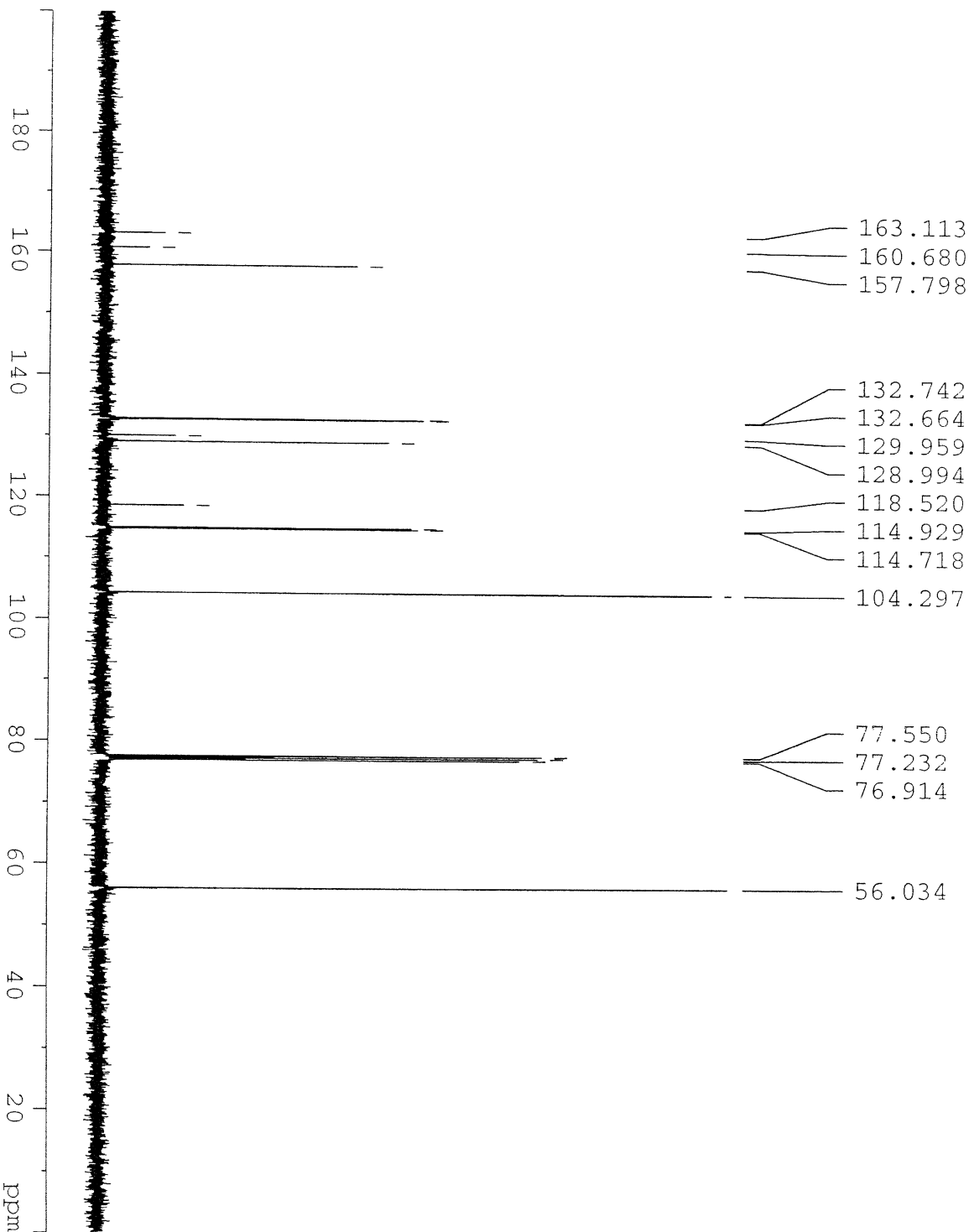
F2 - Acquisition Parameters
 Date_ 20071110

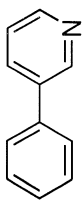
Time 12.16
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 75
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 4597.6
 DW 20.850 usec
 DE 6.00 usec
 TE 293.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.38 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127585 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



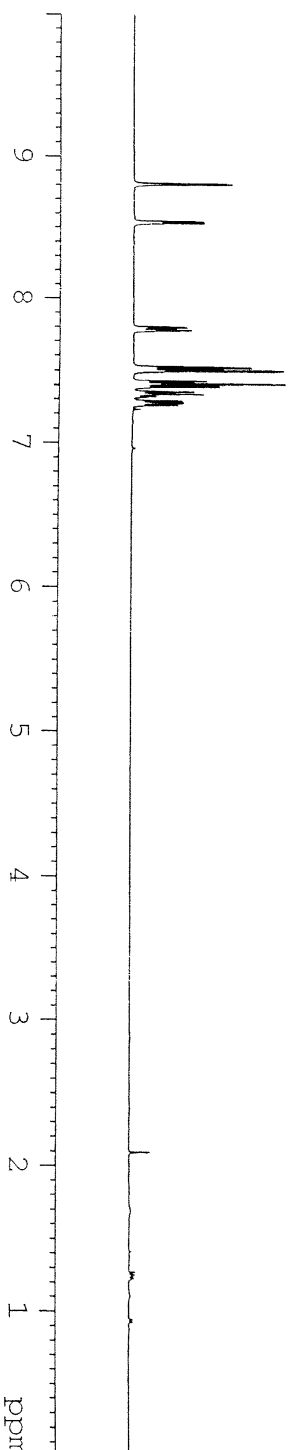


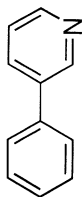
Current Data Parameters
 NAME JN3-076a-010
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080131
 Time 17.18
 INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 57
 DW 60.400 usec
 DE 6.00 usec
 TE 291.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 14.00 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 65536
 SF 400.1300220 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
 NAME JN3-076a-010
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080131
 Time 17.21

INSTRUM spect
 PROBHD 5 mm QNP 1H/13
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 17
 DS 4

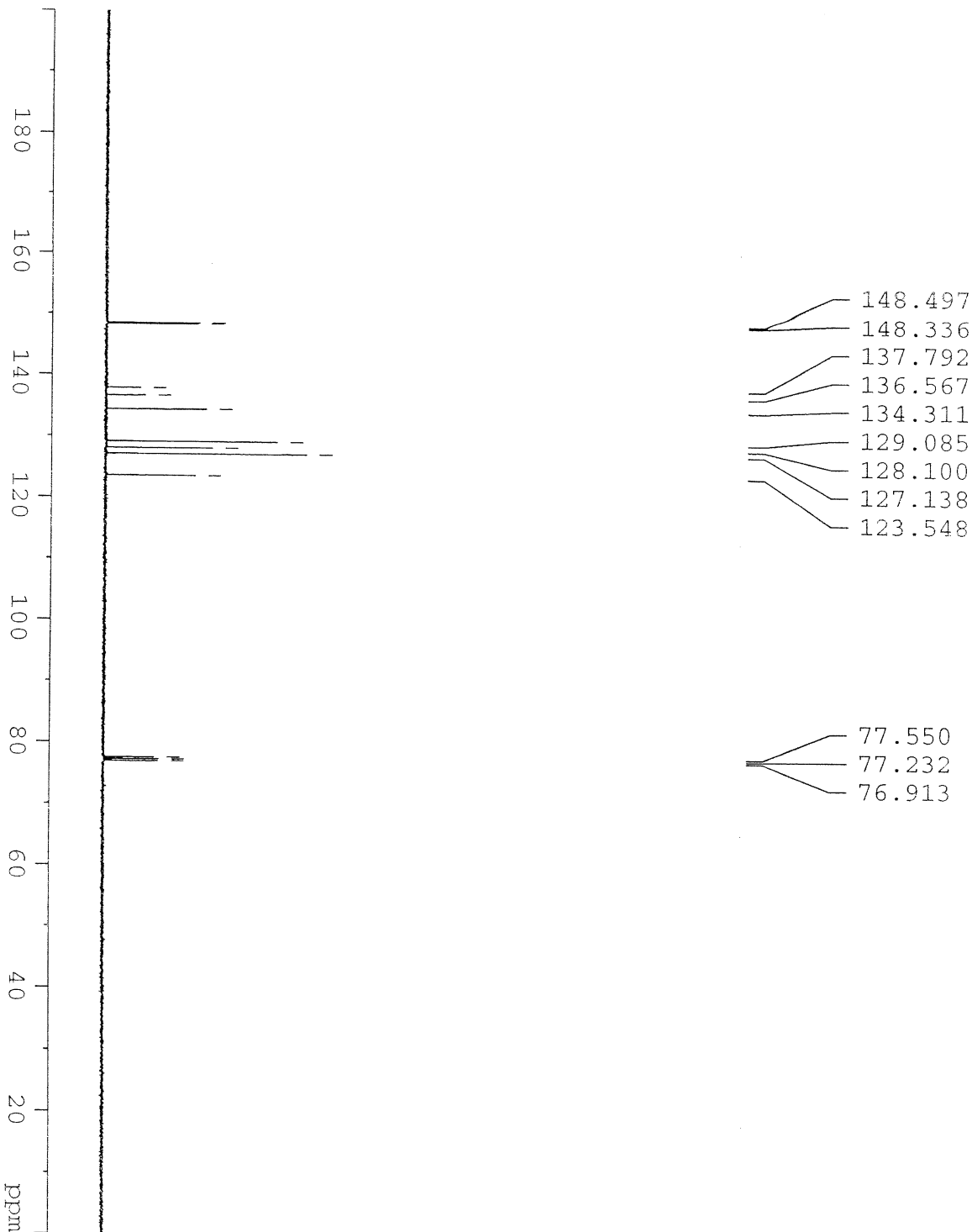
SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 1625.5
 DW 20.850 usec
 DE 6.00 usec
 TE 291.2 K

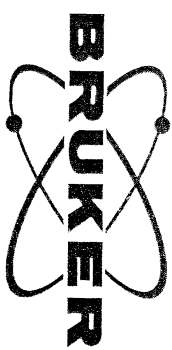
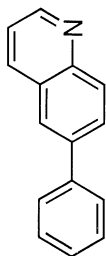
D1 2.0000000 sec
 d11 0.0300000 sec
 DELTA 1.8999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 9.38 usec
 PL1 0.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 0.00 dB
 PL12 16.10 dB
 PL13 19.00 dB
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127707 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



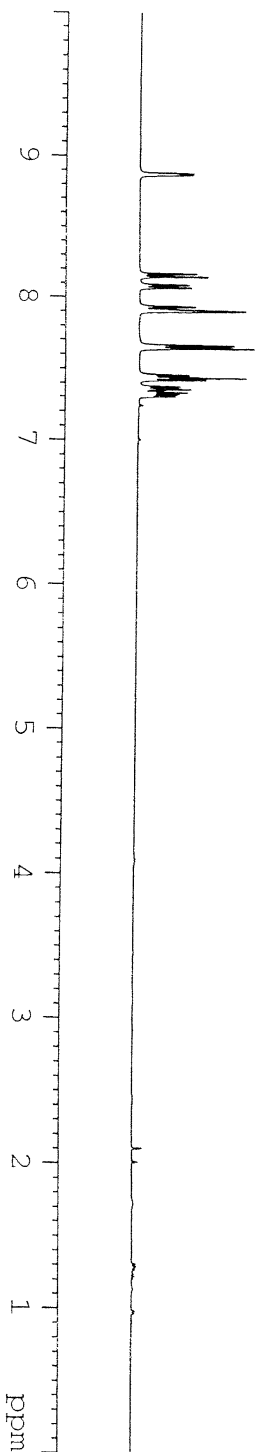


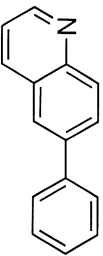
Current Data Parameters
 NAME JN3-078a-010
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080131
 Time 17.34
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 65536
 SOLVENT
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 40.3
 DW 60.400 usec
 DE 6.00 usec
 TE 292.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 15.07 usec
 PL1 0.00 dB
 SFO1 400.1324710 MHz

F2 - Processing Parameters
 SI 65536
 SF 400.1300177 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





Current Data Parameters
 NAME JN3-078a-010
 EXPNO 13
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20080131
 Time 17.37

INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 24
 DS 2

SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 8192

DM 20.850 usec
 DE 6.00 usec
 TE 292.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.75 usec
 PL1 -3.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 -1.00 dB
 PL12 14.52 dB
 PL13 18.00 dB
 SFO2 400.1316005 MHz

F2 - Processing Parameters
 SI 65536
 SF 100.6127716 MHz
 WDM EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

