Supplementary Information

For

Remote Amide-Directed Palladium-Catalyzed Benzylic C–H Amination with *N*-Fluorobenzenesulfonimide

Tao Xiong, Yan Li, Yunhe Lv, and Qian Zhang*

Department of Chemistry, Northeast Normal University, Changchun, 130024, China

zhangq651@nenu.edu.cn

Table of Contents

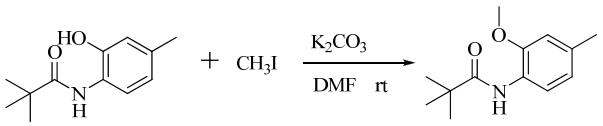
General Information:

All commercially available compounds were used as received, Palladium acetate and *N*-Fluorobenzenesulfonimide were purchased from J & K Chemical Limited. 2-amino-5-methylphenol was purchased from Alfa aesar. 1,2-dichloroethane was dryed with $CaCl_2$. All reactions were run under air with no precautions taken to exclude moisture. ^{1}H NMR and ^{13}C NMR spectra were recorded at 25ºC on a Varian 500 MHz and 126 MHz, respectively, and TMS as internal standard. IR spectra (KBr) were recorded on a Magna-560 FTIR spectrophotometer in the range of 400~4000 cm^{-1}. Melting points were obtained with a micro melting point XT4A Beijing Keyi electrooptic apparatus and are uncorrected. Elemental analyses were measured on a PE-2400 analyzer (Perkin-Elmer). High resolution mass spectra were recorded on Bruck microtof. All reactions were monitored by TLC with Taizhou GF254 silica gel coated plates. Flash column chromatography was carried out using 300-400 mesh silica gel at increased pressure.

Synthesis Procedure:

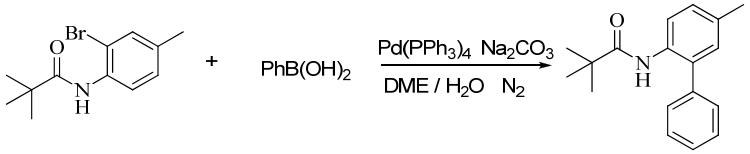
Substrates **1a-1d, 1f** and **1j** were prepared by the reaction of corresponding anilines and acyl chlorides in CH_2Cl_2 at room temperature.[1] Substrates **1e** was prepared according to literature procedure.[2]

General procedure for substrate 1g, 1h and 1i (1g as an example)



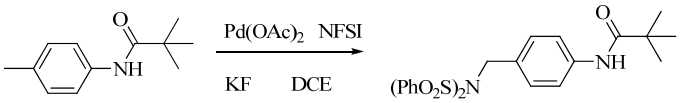
To a solution of *N*-(2-hydroxy-4-methylphenyl)pivalamide (2.0 mmol) in DMF(5 mL) at room temperature was added K_2CO_3 (2.4 mmol) in one portion. After the reaction mixture was stirred for 15 min, added CH_3I (2.4 mmol). The reactions were stirred for 1.5 h. After the reaction was stopped, the reaction mixture was concentrated *in vacuo*. The mixture was purified by column chromatography (10% ether/petro ether) afforded the product **1f** (97%).

General procedure for substrate 1k, 1l, 1m, and 1n[3] (1k as an example)



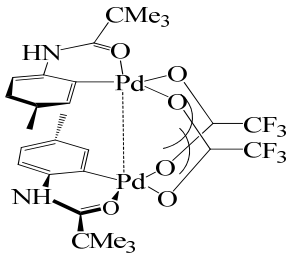
A solution of *N*-(2-bromo-4-methylphenyl)pivalamide (2 mmol) in degassed dimethoxyethane (DME) (6 mL) was stirred at room temperature with $Pd(Ph_3P)_4$ (6 mmol) for 20 min, then phenylboronic acid (2.4 mmol) and 2 M aqueous Na_2CO_3 solution (2 ml) were added and lowered into an oil bath at 80 ^oC under nitrogen. After completion of the reaction (TLC monitoring) DME was partially evaporated under reduced pressure, the mixture was poured on ice-water and extracted with dichloromethane (3×15 mL). The combined organic layers were dried (Na_2SO_4), filtered over Celite, evaporated *in vacuo*, and the residue was purified by column chromatography to give the compound **1k** (417 mg, 78%).

General procedure for Palladium-Catalyzed Benzylic C–H Amination of 1 with *N*-Fluorobenzenesulfonimide (1a as an example)



To a solution of the *N*-p-tolylpivalamide (**1a**, 0.40 mmol) in 1,2-dichloroethane (4.0 ml) was added the *N*-Fluorobenzenesulfonimide (315 mg, 1.0 mmol), KF (93 mg, 1.6 mmol) and $Pd(OAc)_2$ (9.0 mg, 0.04 mmol). The reaction was stirred for the 5.5 h at 90 ^oC under air condition. After completion of the reaction (TLC monitoring) the mixture was poured on ice-water and extracted with dichloromethane (3×15 mL). The combined organic layers were dried (Na_2SO_4), filtered over Celite, evaporated *in vacuo*, and the residue was purified by column chromatography to give the compound *N*-(4-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)phenyl)pivalamide (**2a**, 167 mg, 86%).

Palladium complex E[4]



In a one-dram vial was added *N*-*p*-tolylpivalamide (**1a**) (67.3 mg, 0.3 mmol), $Pd(OAc)_2$ (67.2 mg, 0.3 mmol), and dichloromethane (3 mL). Trifluoroacetic acid (34.2 mg, 0.3 mmol) was subsequently added into the vial and the resulting solution was heated to 40 °C for 6 h. After cooling to ambient temperature, the reaction mixture was concentrated *in vacuo* and the resulting residue was redissolved in petro ether (6 mL). Then the precipitation of the desired complex occurred. The suspension was filtered through Celite and washed with 4 x 2 mL (20% ether/petro ether). The residue was evaporated *in vacuo* to afford the bimetallic palladacycle **E** as a yellow solid (87.3 mg, 71%). ^{1}H NMR (500 MHz; CD_3COCD_3): δ = 0.89 (s, 9H), 2.19 (s, 3H), 6.84 (d, J = 8.0 Hz, 1H), 6.91 (d, J = 8.0 Hz, 2H), 9.81 (s, 1H). ^{13}C NMR (125 MHz; CD_3COCD_3): δ = 20.1, 26.1, 38.4, 114.3, 115.4, 116.1, 116.6, 126.1, 128.8, 131.9, 132.8, 173.9. Recrystallization from acetone and hexanes gave a single crystal suitable for X-ray analysis.

References:

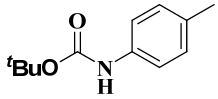
(1) Phipps, R. J.; Gaunt, M. J. *Science* **2009**, *323*, 1593.

(2) Vilaivan, T. *Tetrahedron Letters*. **2006**, *47*, 6739.

(3) Pudlo, M.; Csányi, D.; Moreau, F.; Hajós, G.; Riedlb, Z.; Sapi, J. *Tetrahedron* **2007**, *63* ,10320.

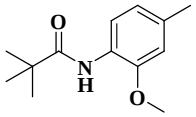
(4) Zhao, X.; Yeung, C. S.; Dong, V. M. *Am. Chem. Soc.* **2010**, *132*, 5837.

Analytical Data for New Compounds



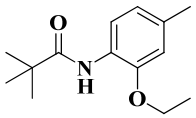
Tert-butyl p-tolylcarbamate 1e

White solid. mp: 86 ^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.51(s, 9H), 2.28 (s, 3H), 6.49 (s, 1H), 7.07 (d, J = 8.0 Hz, 2H), 7.24 (t, J = 7.5 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 20.7, 28.3, 80.3, 118.6, 129.4, 132.5, 135.7, 152.9. IR (KBr, cm^{-1}): 1697, 1529, 1157, 1050, 817, 508. MS calcd *m/z* 207.2689, $[M]^+$ found 207.2684; Anal. Calcd for: $C_{12}H_{17}NO_2$: C, 69.54; H, 8.27; N, 6.76; Found: C, 69.52; H, 8.24; N, 6.73.



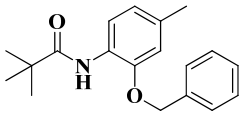
***N*-(2-methoxy-4-methylphenyl)pivalamide 1g**

Colorless liquid; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.31 (s, 9H), 2.31 (s, 3H), 3.86 (s, 3H), 6.68 (s, 1H), 6.75 (d, J = 8.0 Hz, 1H), 8.05 (s, 1H), 8.27 (d, J = 8.0 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 21.2, 27.5, 39.8, 55.6, 110.6, 119.2, 121.1, 125.2, 133.1, 147.7, 176.2. IR (KBr, cm^{-1}): 1680, 1528, 1257, 750. MS calcd *m/z* 221.2955, $[M]^+$ found 221.2959; Anal. Calcd for: $C_{13}H_{19}NO_2$: C, 70.56; H, 8.65; N, 6.33; Found: C, 70.42; H, 8.25; N, 6.77.



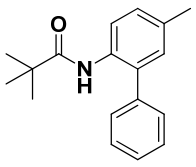
***N*-(2-ethoxy-4-methylphenyl)pivalamide 1h**

Colorless liquid; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.30 (s, 9H), 1.43 (t, J = 7.0 Hz, 3H), 2.28 (s, 3H), 4.03 (dd, J_1 = 13.5 Hz, J_2 = 7.0 Hz , 2H), 6.65 (s, 1H), 6.73 (d, J = 8.0 Hz, 1H), 8.13 (s, 1H), 8.25 (d, J = 7.5 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 14.7, 21.1, 27.4, 39.7, 63.9, 111.5, 118.9, 120.9, 125.2, 132.8, 147.1, 175.9. IR (KBr, cm^{-1}): 1680, 1526, 1259, 1123, 749. MS calcd *m/z* 235.3220, $[M]^+$ found 235.3197; Anal. Calcd for: $C_{14}H_{21}NO_2$: C, 71.46; H, 8.99; N, 5.95; Found: C, 71.43; H, 8.94; N, 5.98.



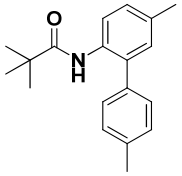
***N*-(2-(benzyloxy)-4-methylphenyl)pivalamide 1i**

Colorless liquid; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.24 (s, 9H), 2.31 (s, 3H), 5.08 (s, 2H), 6.79 (d, J = 5.5 Hz, 2H), 7.35-7.42 (m, 5H), 8.12 (s, 1H), 8.28 (d, J = 9.0 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 21.3, 27.5, 39.8, 70.8, 112.3, 119.4, 121.8, 125.6, 127.3, 128.2, 128.6, 133.2, 136.5, 147.2, 176.2. IR (KBr, cm^{-1}): 1680, 1527, 1257, 1042, 749. MS calcd *m/z* 297.3914, $[M]^+$ found 297.3921; Anal. Calcd for: $C_{19}H_{23}NO_2$: C, 76.73; H, 7.80; N, 4.71; Found: C, 76.75; H, 7.81; N, 4.69.



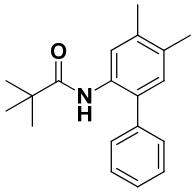
***N*-(5-methylbiphenyl-2-yl)pivalamide 1k**

White solid. mp: 107 ^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.09 (s, 9H), 2.34 (s, 3H), 7.06 (s, 1H), 7.17 (d, J = 8.0 Hz, 1H), 7.36 (t, J = 7.0 Hz, 2H), 7.39-7.42 (m, 2H), 7.47 (t, J = 7.0 Hz, 2H), 8.20 (d, J = 7.0 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 20.8, 27.4, 39.6, 121.0, 127.9, 128.9, 129.3, 130.3, 132.2, 132.5, 133.5, 138.2, 176.2. IR (KBr, cm^{-1}): 1680, 1502, 821, 577. MS calcd *m/z* 267.3654, $[M]^+$ found 267.3652; Anal. Calcd for: $C_{18}H_{21}NO$: C, 80.86; H, 7.92; N, 5.24; Found: C, 80.84; H, 7.93; N, 5.26.



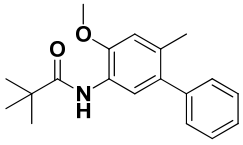
***N*-(5-methylbiphenyl-2-yl)pivalamide 1l**

White solid. mp: 102 ^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.09 (s, 9H), 2.31 (s, 3H), 2.39 (s, 3H), 7.02 (d, J = 1.0 Hz, 1H), 7.13 (d, J = 8.0 Hz, 2H), 7.22 (d, J = 8.5 Hz, 2H), 7.26 (d, J = 8.0 Hz, 2H), 7.45 (s, 1H), 8.20 (d, J = 8.0 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 20.7, 21.0, 27.3, 39.5, 120.9, 128.5, 128.9, 129.5, 130.2, 132.1, 132.5, 133.2, 135.1, 137.5, 175.9. IR (KBr, cm^{-1}): 1680, 1514, 1301, 820. MS calcd *m/z* 281.3920, $[M]^+$ found 281.3924; Anal. Calcd for: $C_{19}H_{23}NO$: C, 81.10; H, 8.24; N, 4.98; Found: C, 81.11; H, 8.25; N, 4.94.



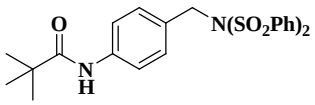
***N*-(4,5-dimethylbiphenyl-2-yl)pivalamide 1m**

White solid. mp: 105 ^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.09 (s, 9H), 2.24 (s, 3H), 2.30 (s, 3H), 7.02 (s, 1H), 7.33 (d, J = 7.5 Hz, 2H), 9.38 (s, 2H), 7.46 (t, J = 7.5 Hz, 2H), 8.14 (s, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 19.1, 19.7, 27.3, 39.6, 122.2, 127.7, 128.8, 129.3, 129.8, 130.7, 132.2, 132.5, 136.7, 138.1, 176.2. IR (KBr, cm^{-1}): 1642, 1511, 846, 820. MS calcd *m/z* 281.3920, $[M]^+$ found 281.3925; Anal. Calcd for: $C_{19}H_{23}NO$: C, 81.10; H, 8.24; N, 4.98; Found: C, 81.12; H, 8.25; N, 4.96.



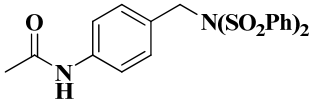
***N*-(4-methoxy-6-methylbiphenyl-3-yl)pivalamide 1n**

White solid. mp: 114 ^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.31 (s, 9H), 2.26 (s, 3H), 3.91 (s, 3H), 6.76 (s, 1H), 7.27 (t, J = 7.0 Hz, 1H), 7.31-7.36 (m, 4H), 8.06 (s, 1H), 8.34 (s, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 20.4, 27.6, 39.9, 55.9, 111.7, 121.2, 125.4, 126.4, 127.8, 129.4, 130.3, 134.5, 141.5, 147.1, 176.3. IR (KBr, cm^{-1}): 1677, 1527, 1170, 581. MS calcd *m/z* 297.3914, $[M]^+$ found 297.3910; Anal. Calcd for: $C_{19}H_{23}NO_2$: C, 76.73; H, 7.80; N, 4.71; Found: C, 76.72; H, 7.82; N, 4.70.



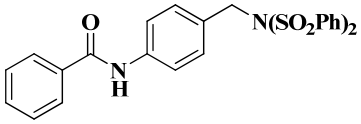
***N*-(4-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)phenyl)pivalamide 2a**

White solid. mp: 124 ^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.34 (s, 9H), 4.88 (s, 2H), 7.30 (s, 1H), 7.33 (d, *J* = 8.5 Hz, 2H), 7.41-7.47 (m, 6H), 7.59 (t, *J* = 7.5 Hz, 2H), 7.80 (d, *J* = 7.5 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 27.6, 39.7, 51.9, 119.7, 128.1, 128.9, 129.9, 130.2, 133.7, 137.9, 139.8, 176.6. IR (KBr, cm^{-1}): 1676, 1394, 1170, 788, 582. MS calcd *m/z* 486.6036, $[M]^+$ found 486.6041; Anal. Calcd for: $C_{24}H_{26}N_2O_5S_2$: C, 59.24; H, 5.39; N, 5.76. Found: C, 59.27; H, 5.34; N, 5.75.



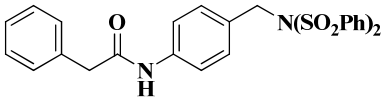
***N*-(4-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)phenyl)acetamide 2b**

White solid. mp: 156 ^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 2.19 (s, 3H), 4.89 (s, 2H), 7.31-7.33 (m, 3H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.44 (t, *J* = 8.0 Hz, 4H), 7.58 (t, *J* = 7.5 Hz, 2H), 7.79 (d, *J* = 7.5 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 24.7, 51.9, 119.5, 128.0, 128.8, 129.9, 130.1, 133.7, 137.8, 139.8, 168.3. IR (KBr, cm^{-1}): 1668, 1214, 750, 704. MS calcd *m/z* 444.5239, $[M]^+$ found 444.5241; Anal. Calcd for: $C_{21}H_{20}N_2O_5S_2$: C, 56.74; H, 4.53; N, 6.30. Found: C, 56.71; H, 4.52; N, 6.27.



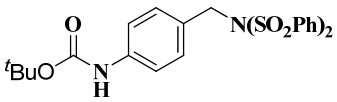
***N*-(4-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)phenyl)benzamide 2c**

White solid. mp: 131 ^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 4.91 (s, 2H), 7.36 (d, *J* = 8.5 Hz, 2H), 7.45-7.53 (m, 7H), 7.54-7.59 (m, 5H), 7.81 (t, *J* = 8.0 Hz, 3H), 7.89 (d, *J* = 8.5 Hz, 2H), 7.96 (s, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 51.9, 119.9, 127.0, 128.1, 128.8, 128.9, 129.9, 130.5, 131.9, 133.7, 134.7, 137.9, 139.9, 165.6. IR (KBr, cm^{-1}): 1684, 1513, 1165, 752. MS calcd *m/z* 506.5933, $[M]^+$ found 506.5936; Anal. Calcd for: $C_{26}H_{22}N_2O_5S_2$: C, 61.64; H, 4.38; N, 5.53; Found: C, 61.62; H, 4.34; N, 5.56.



2-phenyl-N-(4-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)phenyl)acetamide 2d

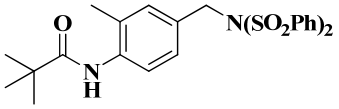
White solid. mp: 171 ^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 3.75 (s, 2H), 4.86 (s, 2H), 7.22 (s, 1H), 7.35 (t, *J* = 7.0 Hz, 3H), 7.39-7.44 (m, 5H), 7.47 (t, *J* = 7.5 Hz, 2H), 7.54-7.59 (m, 3H), 7.77 (t, *J* = 7.5 Hz, 4H), 7.93 (d, *J* = 7.5 Hz, 2H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 44.7, 51.8, 119.6, 127.7, 127.7, 128.0, 128.8, 129.0, 129.2, 129.5, 129.8, 130.5, 133.6, 133.7, 134.2, 137.4, 139.8, 169.4. IR (KBr, cm^{-1}): 1675, 1514, 1374, 1167, 752. MS calcd *m/z* 520.6199, $[M]^+$ found 520.6174; Anal. Calcd for: $C_{27}H_{24}N_2O_5S_2$: C, 62.29; H, 4.65; N, 5.38; Found: C, 62.31; H, 4.66; N, 5.36.



tert-butyl 4-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)phenylcarbamate 2e

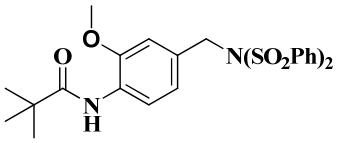
White solid. mp: 96 ^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.54 (s, 9H), 4.87 (s, 2H), 6.49 (s, 1H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 8.5 Hz, 2H), 7.44 (t, *J* = 8.0 Hz, 4H), 7.57 (t, *J* = 8.0 Hz, 2H), 7.79 (t, *J* = 7.0 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 28.3, 51.9, 80.7, 118.1, 127.8, 128.1,

128.8, 129.1, 130.1, 131.3, 133.6, 138.3, 139.9, 152.5. IR (KBr, cm^{-1}): 1631, 1170, 788, 745. MS calcd *m/z* 502.6030, $[M]^+$ found 502.6025; Anal. Calcd for: $C_{24}H_{26}N_2O_6S_2$: C, 57.35; H, 5.21; N, 5.57; Found: C, 57.38; H, 5.19; N, 5.55.



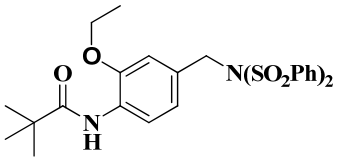
***N*-(2-methyl-4-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)phenyl)pivalamide 2f**

White solid. mp: 147 °C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.34 (s, 9H), 2.10 (s, 3H), 4.87 (s, 2H), 7.09 (s, 1H), 7.17 (dd, J_1 = 8.0 Hz, J_2 = 1.5 Hz , 1H), 7.23 (s, 1H), 7.45 (t, *J* = 8.0 Hz, 4H), 7.56 (t, *J* = 8.0 Hz, 2H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 17.4, 27.6, 39.7, 52.0, 122.6, 127.5, 128.0, 128.8, 128.9, 130.7, 130.8, 133.6, 135.7, 139.8, 176.4. IR (KBr, cm^{-1}): 1642, 1511, 844, 486, 457. MS calcd *m/z* 500.6302, $[M]^+$ found 500.6297; Anal. Calcd for: $C_{25}H_{28}N_2O_5S_2$: C, 59.98; H, 5.64; N, 5.60; Found: C, 59.96; H, 5.67; N, 5.62.



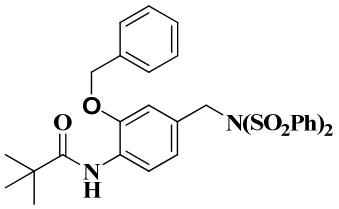
***N*-(2-methoxy-4-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)phenyl)pivalamide 2g**

White solid. mp: 158 °C;^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.34 (s, 9H), 3.67 (s, 3H), 4.89 (s, 2H), 6.83 (s, 1H), 6.94 (d, *J* = 8.5 Hz, 1H), 7.45 (t, *J* = 8.0 Hz, 4H), 7.58 (t, *J* = 7.0 Hz, 2H), 7.83 (d, *J* = 8.0 Hz, 4H), 8.08 (s, 1H), 8.32 (d, *J* = 8.5 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 27.6, 40.0, 52.4, 55.7, 110.0, 118.9, 122.0, 127.7, 128.1, 128.8, 129.4, 133.6, 139.9, 147.9, 176.5. IR (KBr, cm^{-1}): 1677, 1526, 1374, 1168, 582, 563. MS calcd *m/z* 516.6296, $[M]^+$ found: 516.6292; Anal. Calcd for: $C_{25}H_{28}N_2O_6S_2$: C, 58.12; H, 5.46; N, 5.42; Found: C, 58.14; H, 5.47; N, 5.40.



***N*-(2-ethoxy-4-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)phenyl)pivalamide 2h**

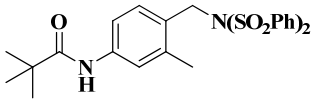
White solid. mp: 142 °C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.34 (s, 9H), 1.37 (t, *J* = 7.0 Hz, 3H), 3.84 (dd, J_1 = 14.0 Hz, J_2 = 7.0 Hz , 2H), 4.88 (s, 2H), 6.84 (s, 1H), 6.94 (d, *J* = 7.0 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 4H), 7.56-7.59 (m, 2H), 7.82 (d, *J* = 7.5 Hz, 4H), 8.17 (s, 1H), 8.30 (d, *J* = 8.0 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 14.6, 27.5, 40.0, 52.4, 64.0, 111.1, 118.8, 121.9, 127.8, 128.1, 128.2, 128.8, 129.4, 133.5, 139.9, 147.3, 176.4. IR (KBr, cm^{-1}): 1677, 1526, 1479, 1394, 1170, 582, 562. MS calcd *m/z* 530.6562, $[M]^+$ found 530.6457; Anal. Calcd for: $C_{26}H_{30}N_2O_6S_2$: C, 58.85; H, 5.70; N, 5.28; Found: C, 58.84; H, 5.73; N, 5.26.



***N*-(2-(benzyloxy)-4-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)phenyl)pivalamide 2i**

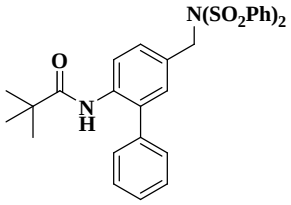
White solid. mp: 135 °C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.27 (s, 9H), 4.82 (s, 2H), 4.89 (s, 2H),

6.96 (s, 1H), 6.97 (s, 1H), 7.35-7.43 (m, 9H), 7.54 (d, *J* = 7.5 Hz, 2H), 7.82 (d, *J* = 8.0 Hz, 4H), 8.16 (s, 1H), 8.32 (d, *J* = 9.0 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 27.5, 39.9, 52.4, 70.7, 111.8, 119.1, 122.4, 127.6, 128.1, 128.2, 128.4, 128.7, 128.8, 129.5, 133.6, 136.1, 139.9, 147.2, 176.5. IR (KBr, cm^{-1}): 1681, 1525, 1375, 1168, 581, 550. MS calcd *m/z* 592.7256, $[M]^+$ found 592.7252; Anal. Calcd for: $C_{31}H_{32}N_2O_6S_2$: C, 62.82; H, 5.44; N, 4.73; Found: C, 62.81; H, 5.42; N, 4.75.



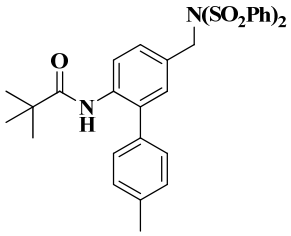
***N*-(3-methyl-4-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)phenyl)pivalamide 2j**

White solid. mp: 137 ^{o}C;^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.32 (s, 9H), 2.28 (s, 3H), 4.97 (s, 2H), 7.00 (dd, J_1 = 8.5 Hz, J_2 = 2.0 Hz, 1H), 7.14 (d, *J* = 8.5 Hz, 1H), 7.24 (s, 1H), 7.39 (s, 1H), 7.45 (t, *J* = 7.5 Hz, 4H), 7.59 (t, *J* = 7.5 Hz, 2H), 7.82 (d, *J* = 8.5 Hz, 4H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 19.3, 27.6, 39.6, 49.7, 117.2, 121.5, 126.6, 127.9, 128.0, 128.4, 128.8, 129.9, 133.6, 137.4, 137.6, 139.9, 176.6. IR (KBr, cm^{-1}): 1687, 1521, 1168, 734, 550. MS calcd *m/z* 500.6302, $[M]^+$ found 500.6293; Anal. Calcd for: $C_{25}H_{28}N_2O_5S_2$: C, 59.98; H, 5.64; N, 5.60; Found: C, 59.94; H, 5.61; N, 5.63.



***N*-(5-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)biphenyl-2-yl)pivalamide 2k**

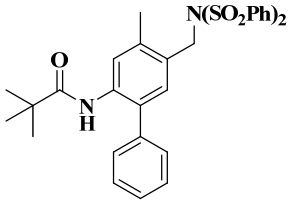
White solid. mp: 140 ^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.10 (s, 9H), 4.93 (s, 2H), 7.17 (d, *J* = 8.0 Hz, 2H), 7.22 (s, 1H), 7.35 (d, *J* = 7.5 Hz, 2H), 7.41-7.49 (m, 6H), 7.58 (t, *J* = 7.5 Hz, 2H), 7.84 (t, *J* = 8.0 Hz, 4H), 8.27 (d, *J* = 8.0 Hz, 1H),^{13}C NMR (125 MHz; $CDCl_3$): δ = 27.3, 39.8, 52.0, 120.6, 128.1, 128.2, 128.4, 128.6, 128.8, 129.1, 129.4, 129.8, 130.2, 132.2, 133.7, 134.9, 137.3, 139.9, 176.3. IR (KBr, cm^{-1}): 1669, 1567, 1159, 581, 550. MS calcd *m/z* 562.6996, $[M]^+$ found 562.6987; Anal. Calcd for: $C_{30}H_{30}N_2O_5S_2$: C, 64.03; H, 5.37; N, 4.98; Found: C, 64.23; H, 5.39; N, 4.94.



***N*-(4'-methyl-5-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)biphenyl-2-yl)pivalamide 2l**

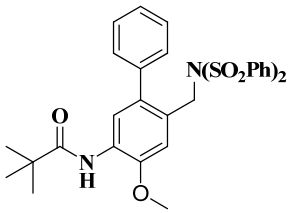
White solid. mp: 113^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.12 (s, 9H), 2.42 (s, 3H), 4.92 (s, 2H), 7.06 (d, *J* = 7.5 Hz, 2H), 7.20 (s, 1H), 7.26 (d, *J* = 7.5 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 1H), 7.45 (t, *J* = 7.5 Hz, 4H), 7.51 (s, 1H), 7.58 (t, *J* = 7.5 Hz, 2H), 7.84 (d, *J* = 7.5 Hz, 4H), 8.28 (d, *J* = 8.5 Hz, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 21.2, 27.4, 39.8, 52.1, 120.4, 128.1, 128.1, 128.8, 128.9, 129.2, 129.7, 130.4, 132.0, 133.6, 133.9, 134.3, 135.1, 138.0, 139.9, 176.3. IR (KBr, cm^{-1}): 1667, 1564, 1160, 581, 550. MS calcd *m/z* 576.7262, $[M]^+$ found 576.7254; Anal. Calcd for:

$C_{31}H_{32}N_2O_5S_2$: C, 64.56; H, 5.59; N, 4.86; Found: C, 64.54; H, 5.57; N, 4.84.



***N*-(4-methyl-5-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)biphenyl-2-yl)pivalamide 2m**

White solid. mp: 139 ^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.11 (s, 9H), 2.38 (s, 3H), 5.01 (s, 2H), 7.04-7.06 (m, 3H), 7.39-7.44 (m, 8H), 7.54 (d, J = 7.5 Hz, 2H), 7.84 (d, J = 7.5 Hz, 4H), 8.16 (s, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 19.2, 27.3, 39.8, 49.6, 122.3, 127.8, 127.9, 128.0, 128.8, 128.9, 129.1, 129.8, 130.2, 133.6, 134.3, 136.7, 137.2, 139.9, 176.4. IR (KBr, cm^{-1}): 1669, 1450, 1400, 1159, 581, 550. MS calcd *m/z* 576.7262, $[M]^+$ found 576.7269; Anal. Calcd for: $C_{31}H_{32}N_2O_5S_2$: C, 64.56; H, 5.59; N, 4.86; Found: C, 64.54; H, 5.57; N, 4.88.

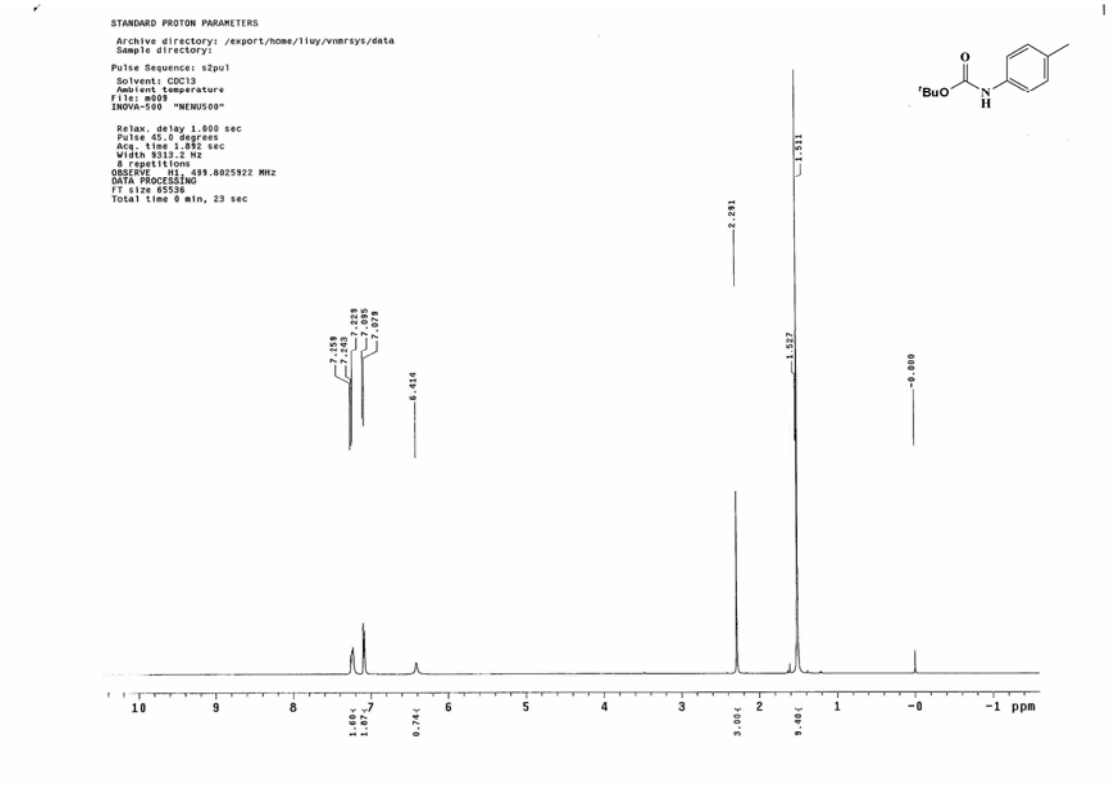


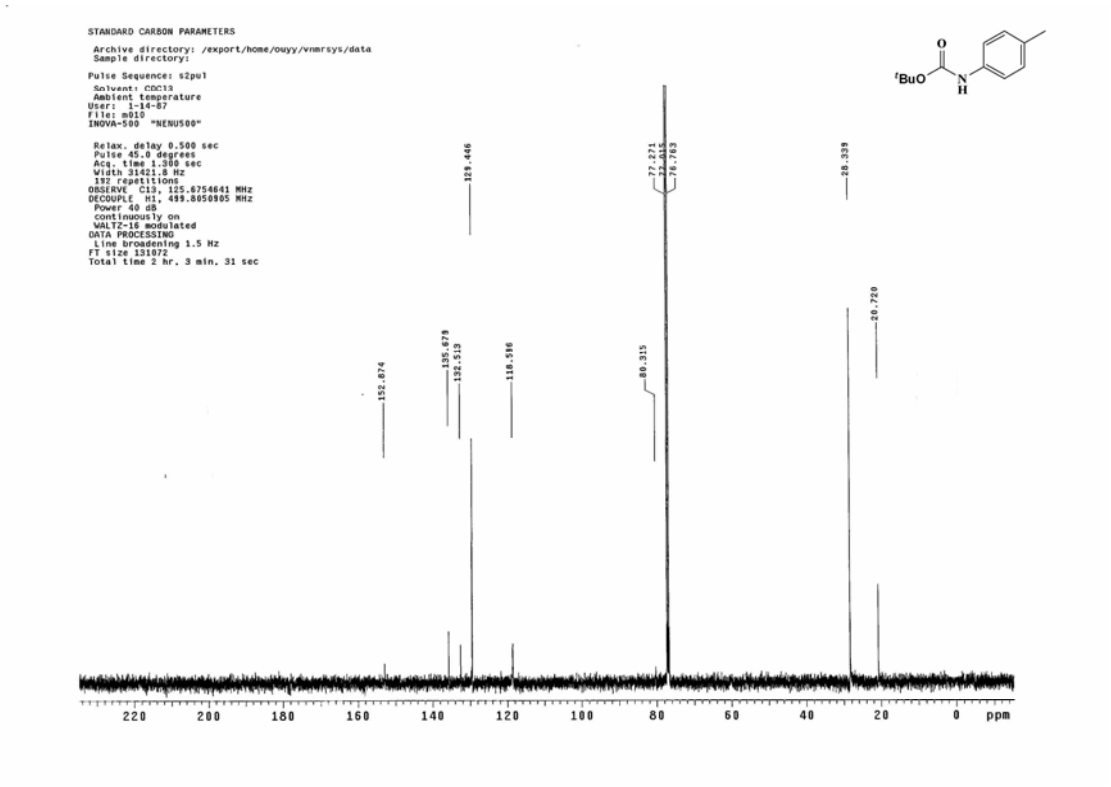
***N*-(4-methoxy-6-((*N*-(phenylsulfonyl)phenylsulfonamido)methyl)biphenyl-3-yl)pivalamide 2n**

White solid. mp: 158^{o}C; ^{1}H NMR (500 MHz; $CDCl_3$): δ = 1.33 (s, 9H), 3.51 (s, 3H), 4.96 (s, 2H), 6.77 (s, 1H), 7.34 (d, J = 7.5 Hz, 3H), 7.38-7.45 (m, 6H), 7.56-7.62 (m, 2H), 7.79 (d, J = 7.5 Hz, 4H), 8.02 (t, J = 7.5 Hz, 2H), 8.36 (s, 1H). ^{13}C NMR (125 MHz; $CDCl_3$): δ = 27.6, 39.9, 50.2, 55.4, 109.4, 120.9, 126.5, 126.7, 127.1, 128.1, 128.2, 128.7, 129.4, 129.6, 129.8, 133.5, 134.7, 135.8, 139.8, 139.9, 147.1, 176.6. IR (KBr, cm^{-1}): 1667, 1452, 1400, 1160, 581, 550. MS calcd *m/z* 592.7256, $[M]^+$ found 592.7249; Anal. Calcd for: $C_{31}H_{32}N_2O_6S_2$: C, 62.82; H, 5.44; N, 4.73; Found: C, 62.84; H, 5.43; N, 4.70.

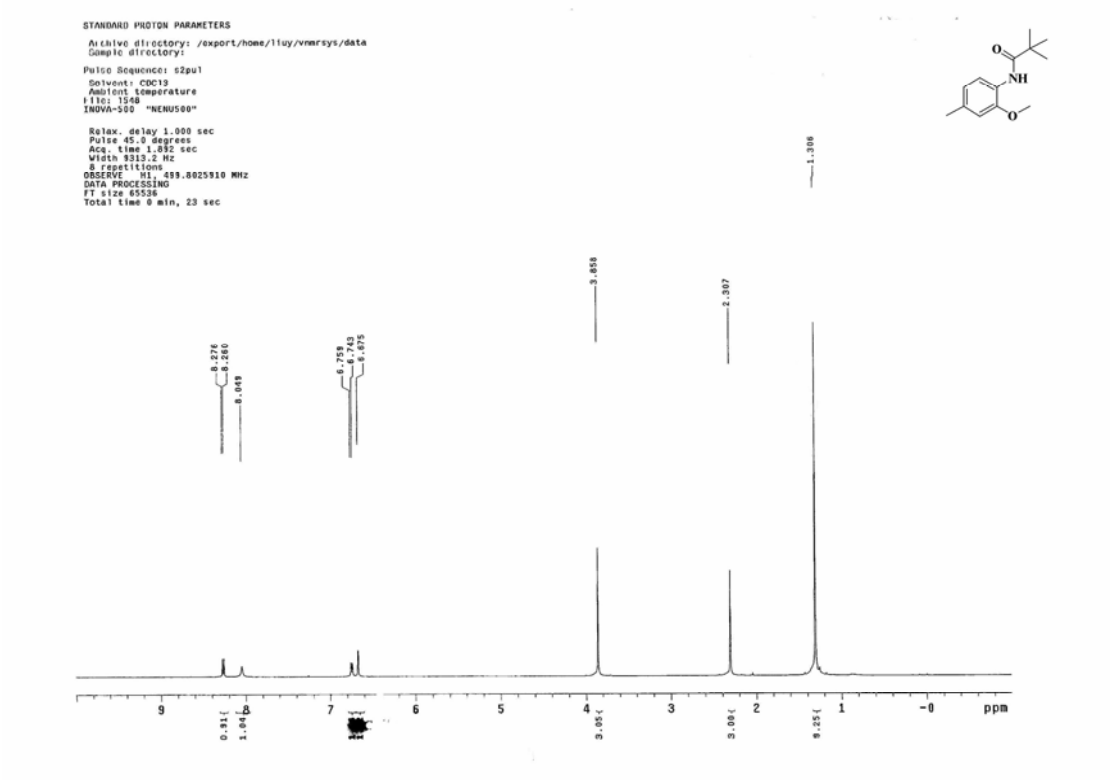
^{1}H and ^{13}C Spectra of New Compounds

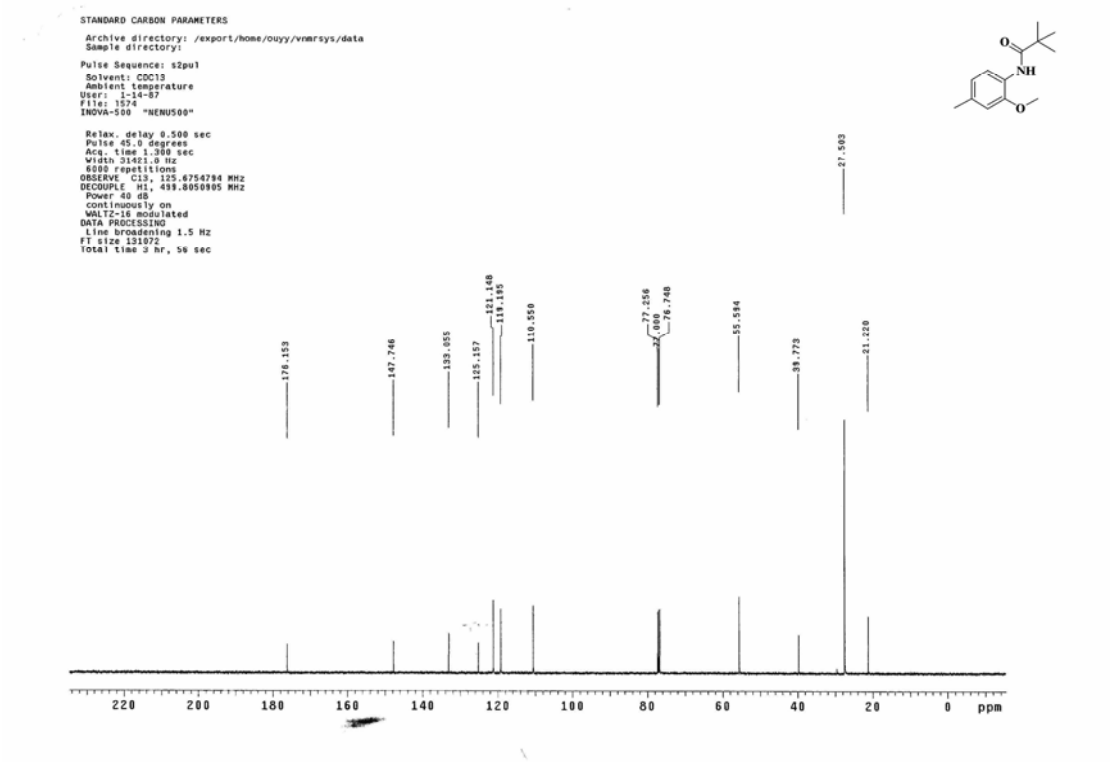
Compound 1e



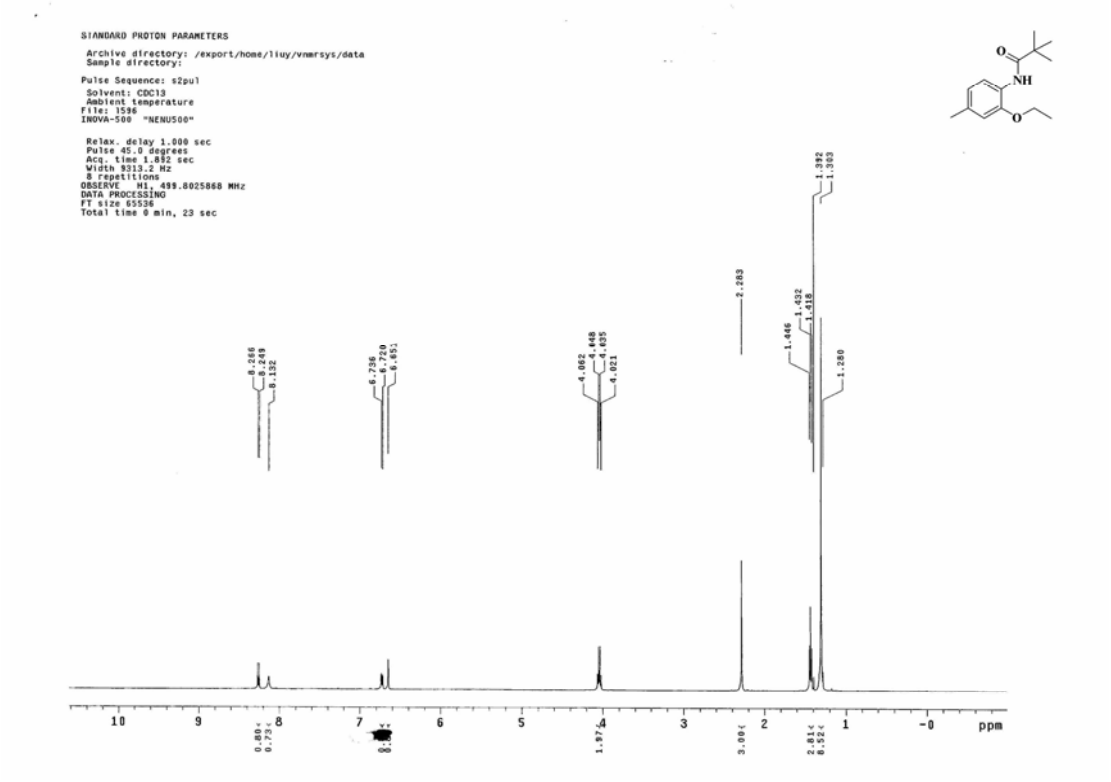


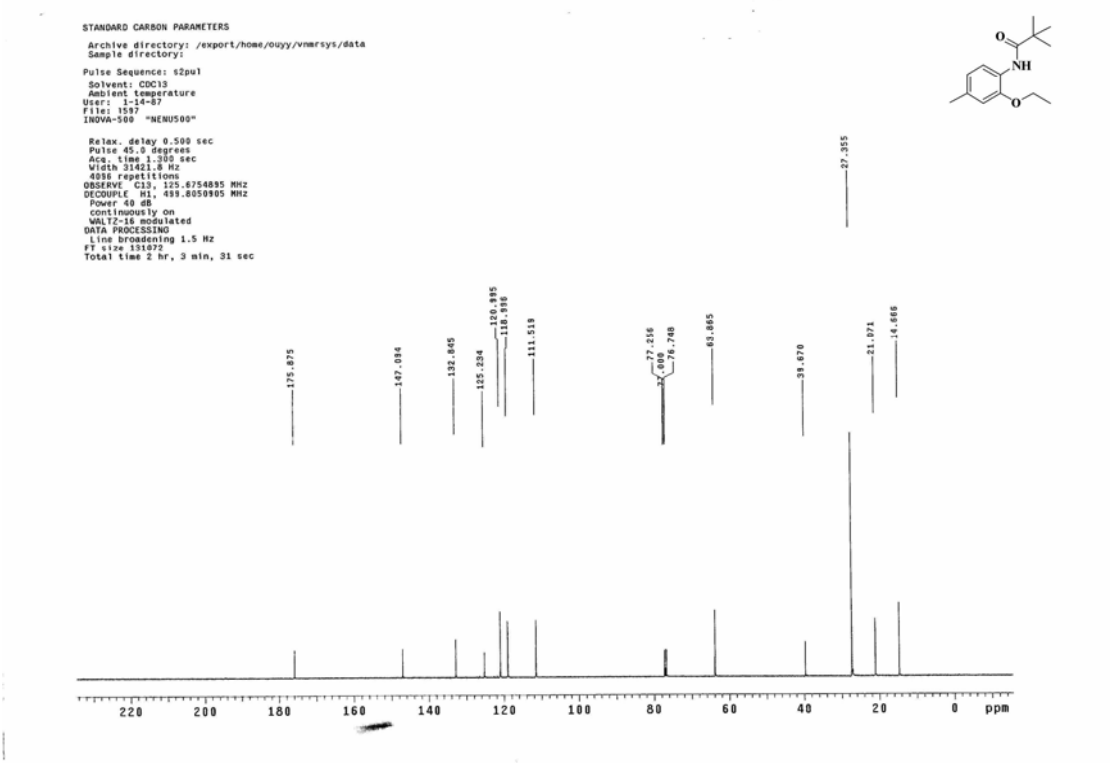
Compound 1g


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1548
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025910 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
8.276
8.260
8.048
6.754
6.743
6.675
3.858
2.307
1.306
ppm

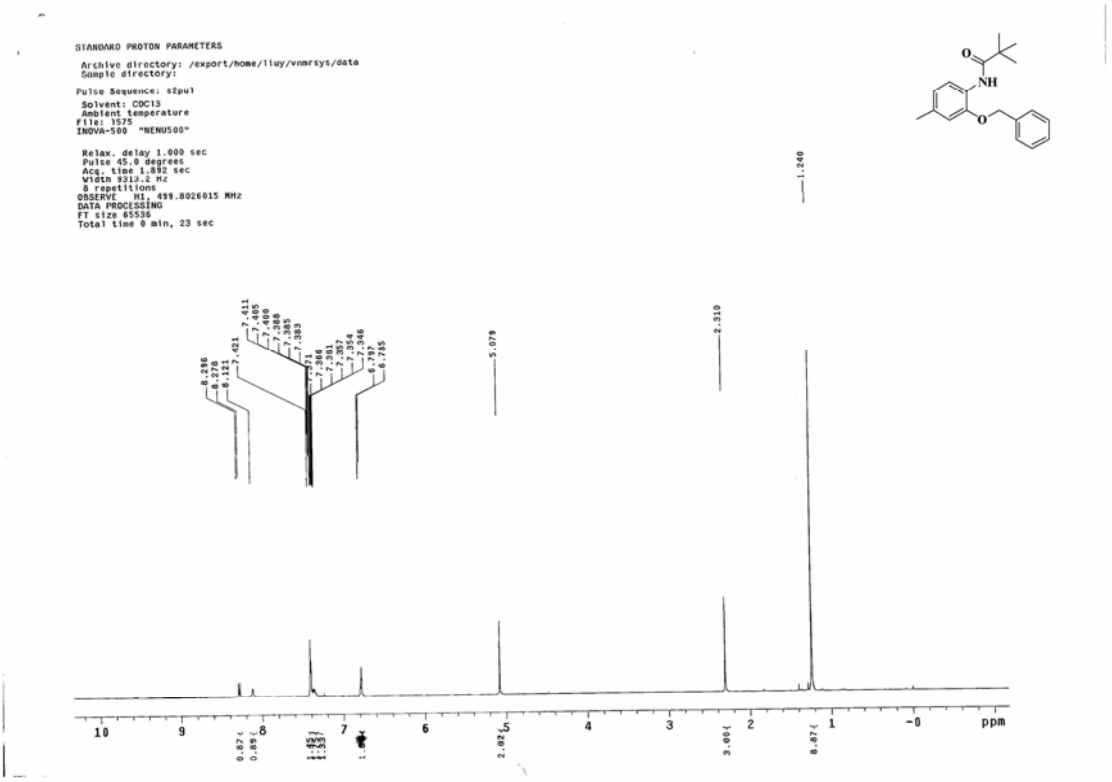

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
File: 1574
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.0 Hz
6000 repetitions
OBSERVE C13, 125.6754794 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
176.153
147.746
139.655
125.157
121.148
119.185
110.550
77.256
77.000
76.748
55.594
39.773
27.593
21.220
ppm

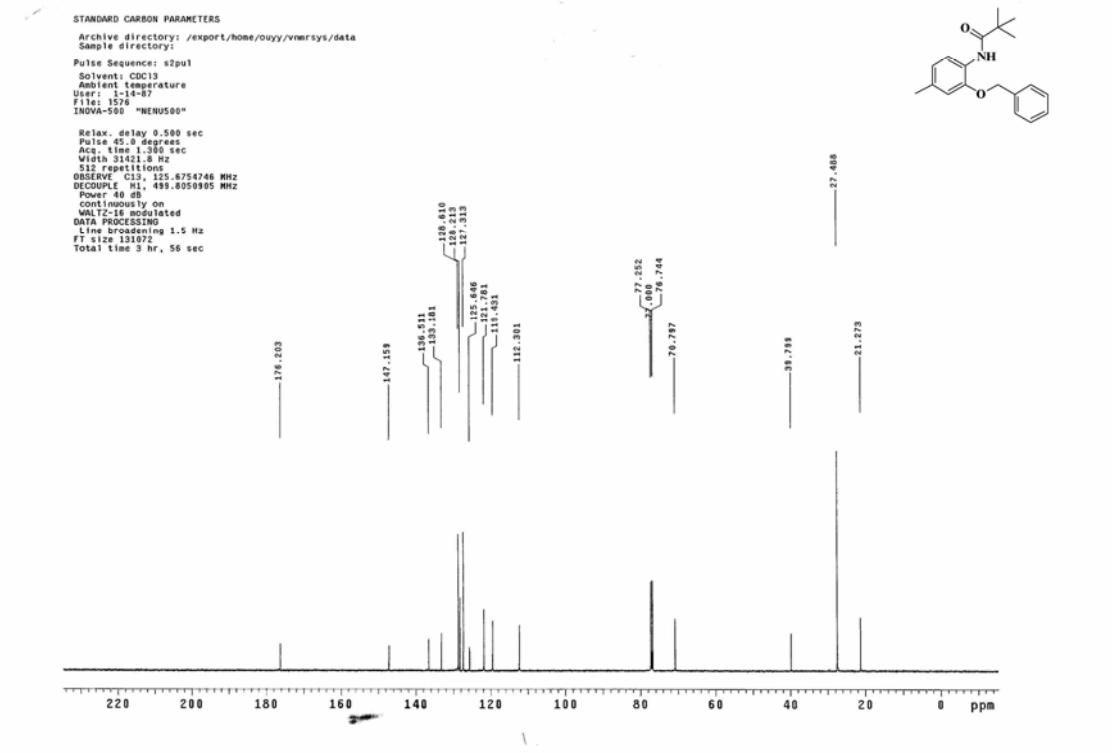
Compound 1h


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1596
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025868 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
8.266
8.249
8.132
6.736
6.720
6.651
4.062
4.048
4.035
4.021
2.283
1.446
1.432
1.418
1.392
1.383
1.280
ppm

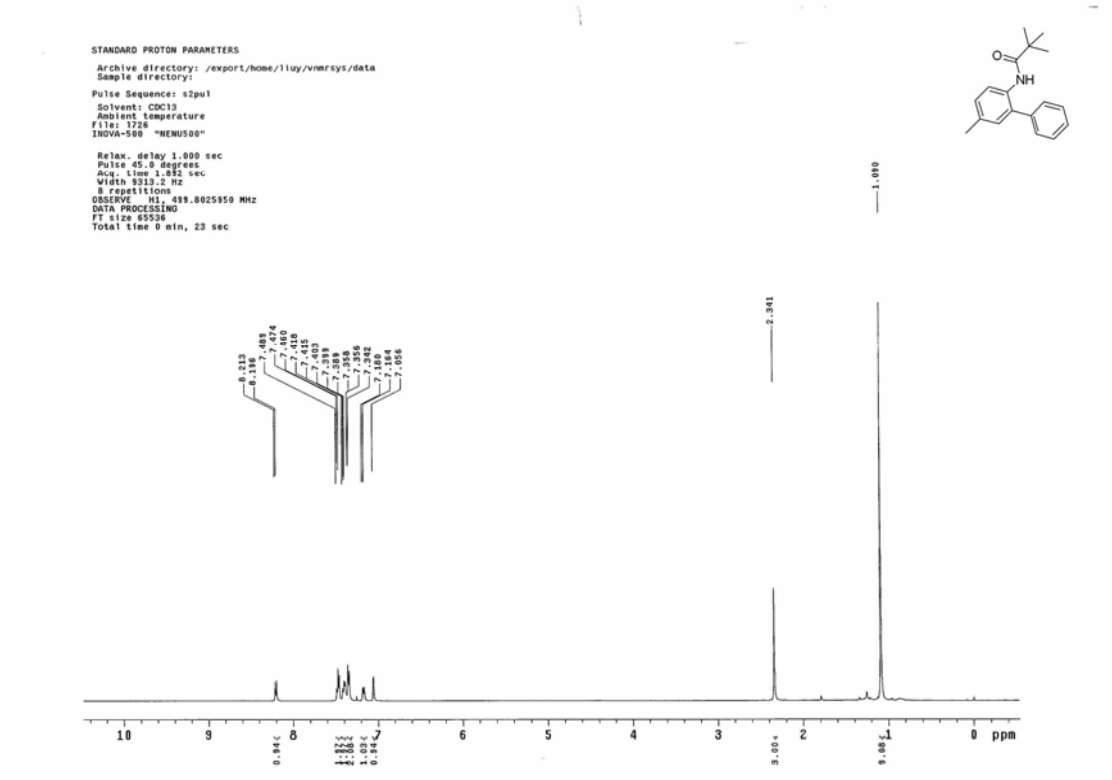

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
File: 1597
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754895 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
175.875
147.084
132.845
125.234
120.995
118.906
111.519
77.256
77.000
76.748
63.865
39.670
27.355
21.971
14.666
ppm

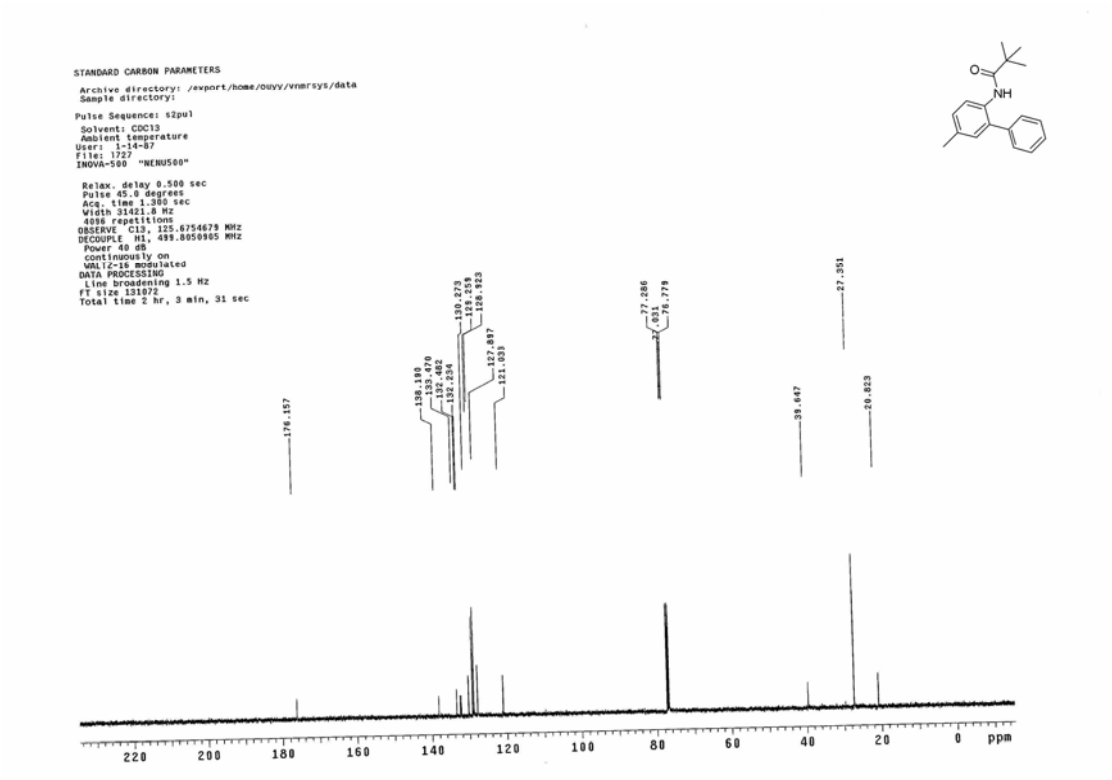
Compound 1i


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1575
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9213.2 Hz
8 repetitions
OBSERVE H1, 499.8026015 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
ppm

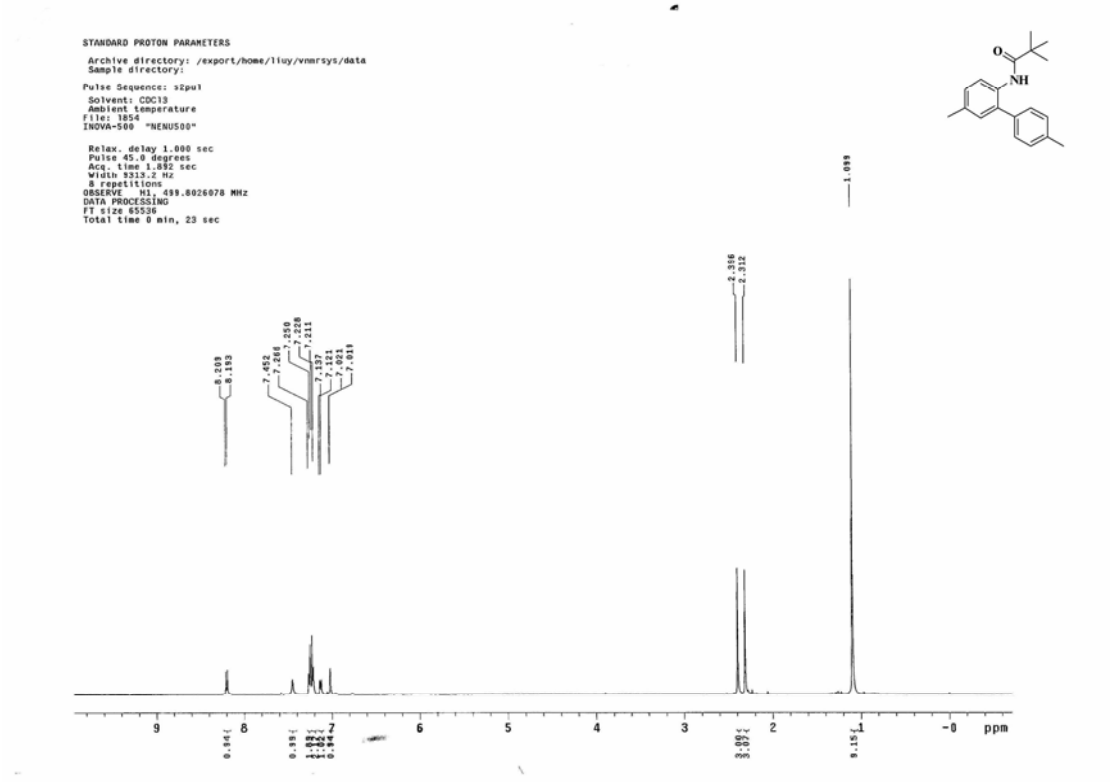

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1578
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
512 repetitions
OBSERVE C13, 125.6754746 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
ppm

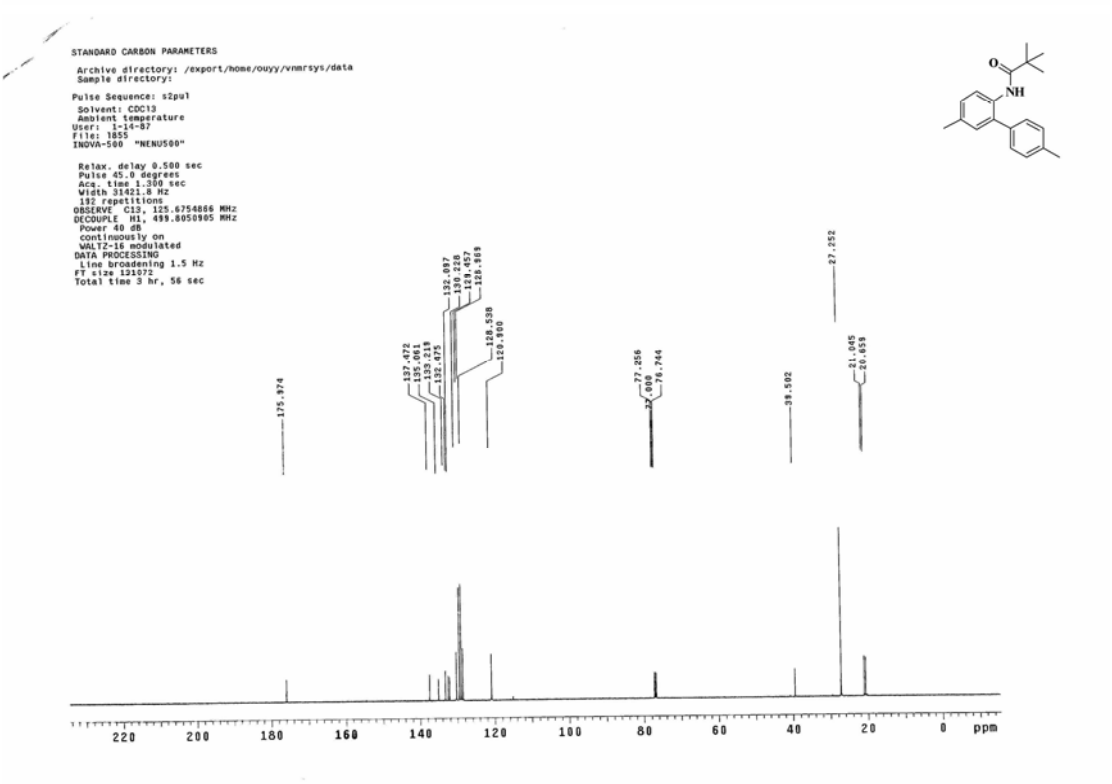
Compound 1k


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1726
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025950 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH
ppm

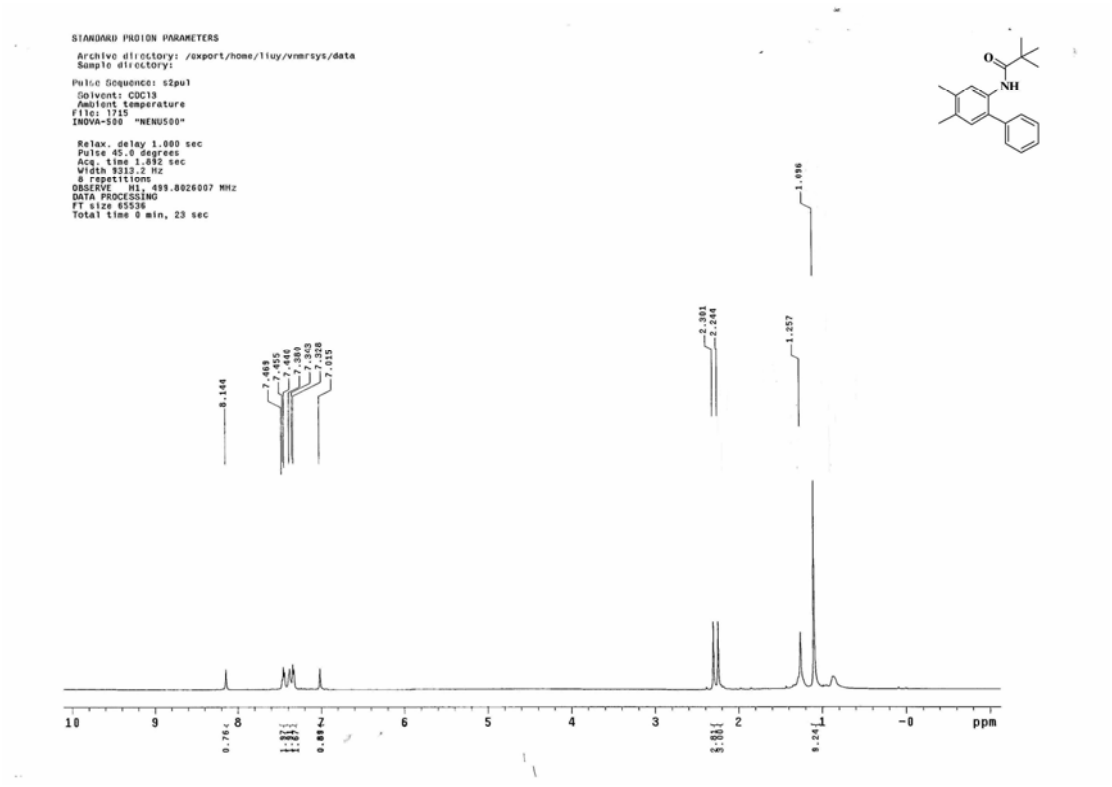

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1727
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754679 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
NH
ppm

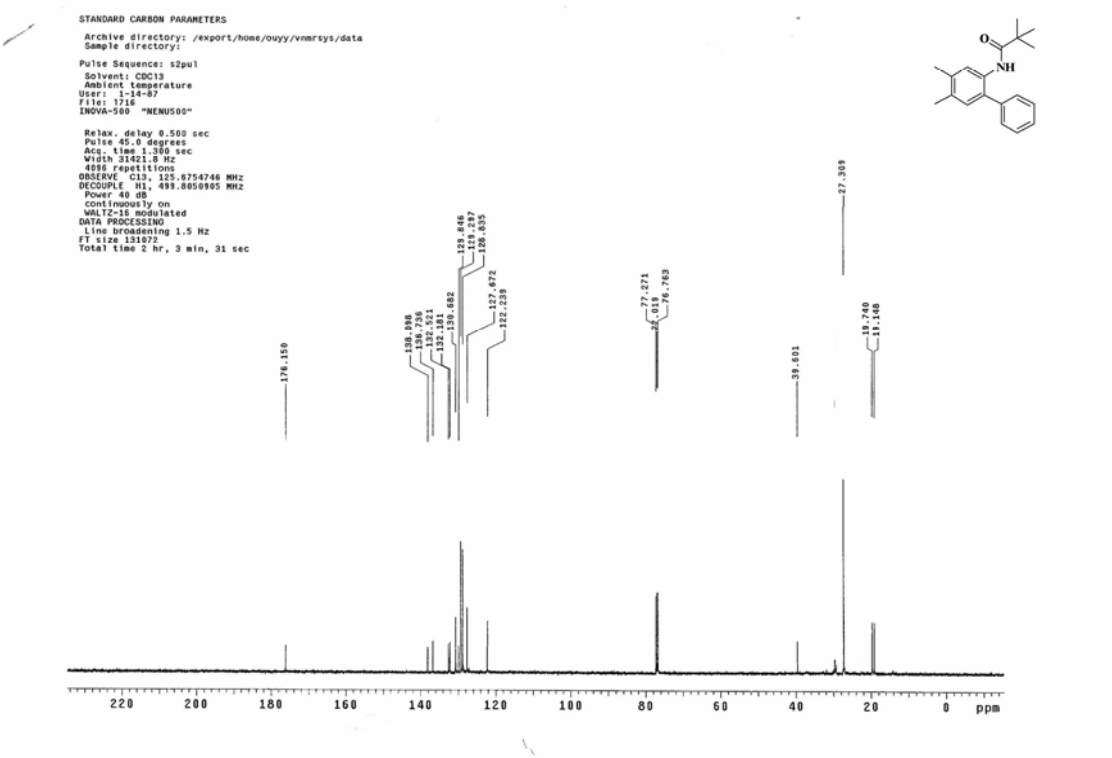
Compound 1l


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1854
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8026078 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
ppm

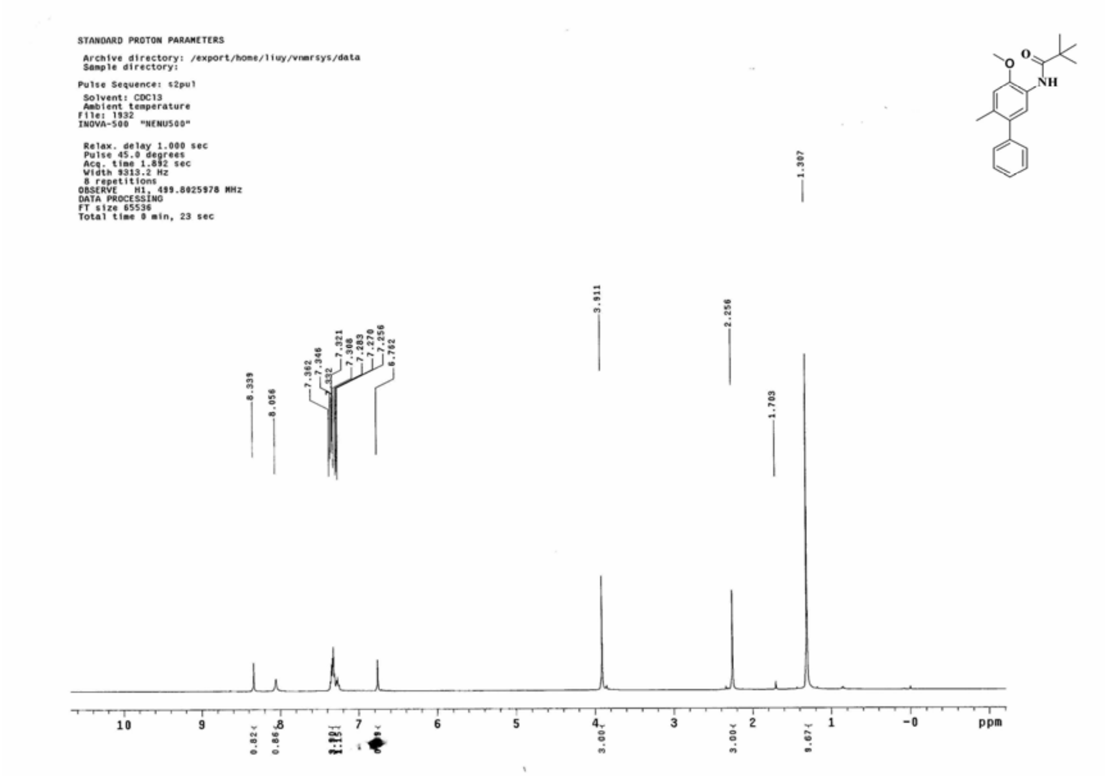

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1855
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754886 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
ppm

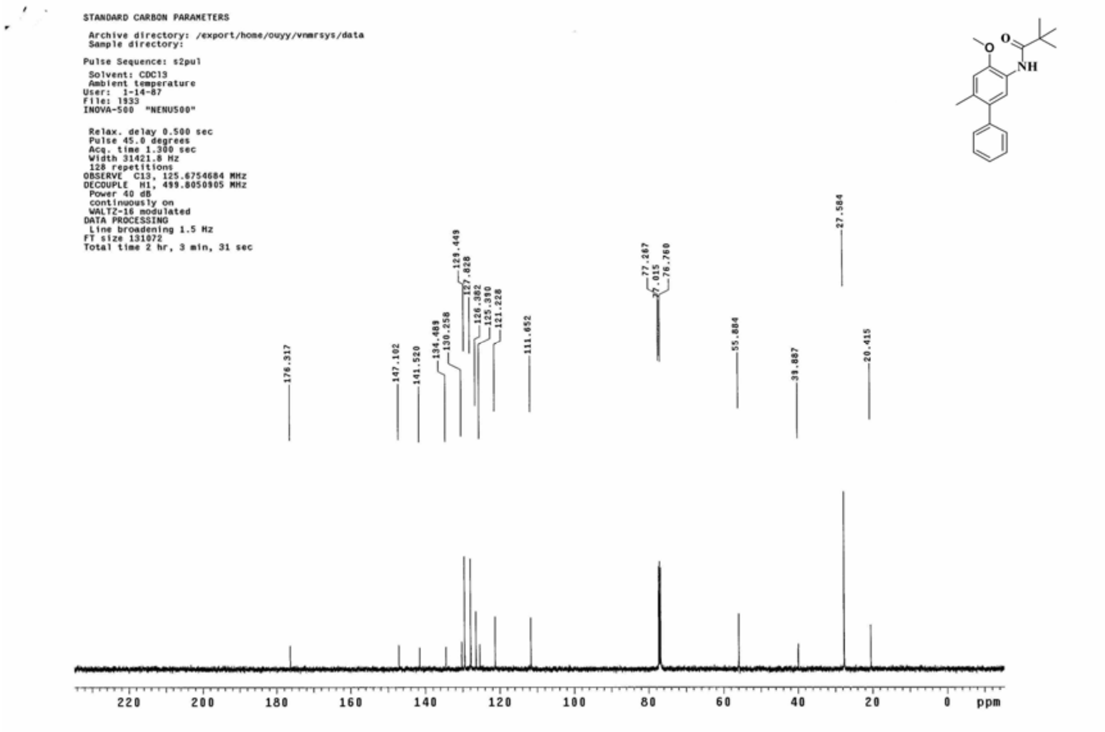
Compound 1m


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1715
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8026007 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
8.144
7.468
7.455
7.444
7.380
7.365
7.328
7.015
2.301
2.244
1.257
1.056
ppm

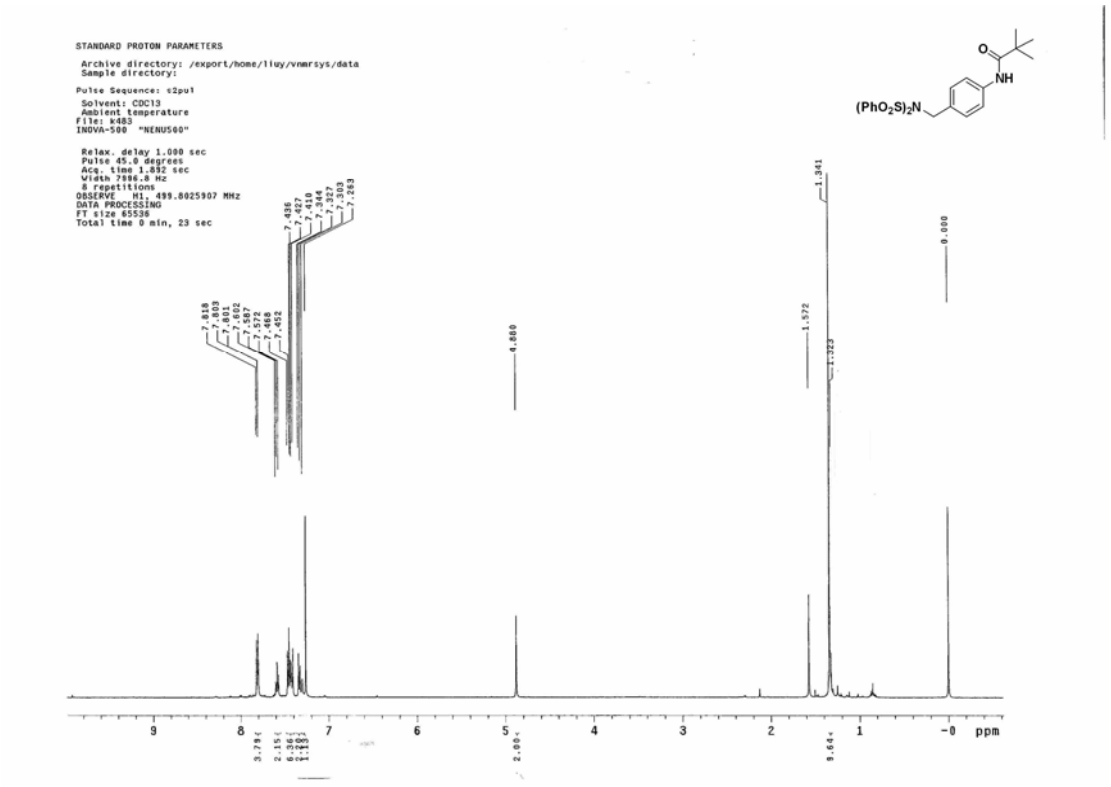

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1716
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754746 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
176.150
138.188
136.766
132.521
132.181
130.882
129.846
129.297
128.835
127.672
122.238
77.271
77.019
76.763
39.601
27.309
19.740
19.148
ppm

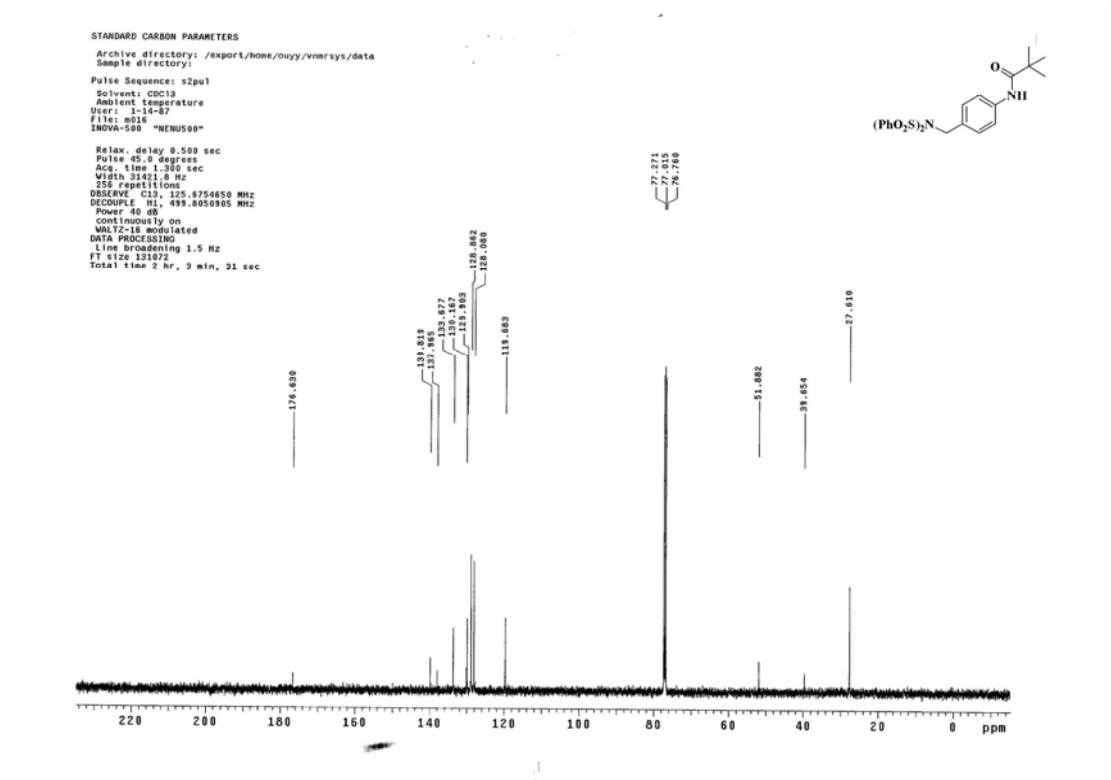
Compound 1n


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1932
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025978 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
ppm

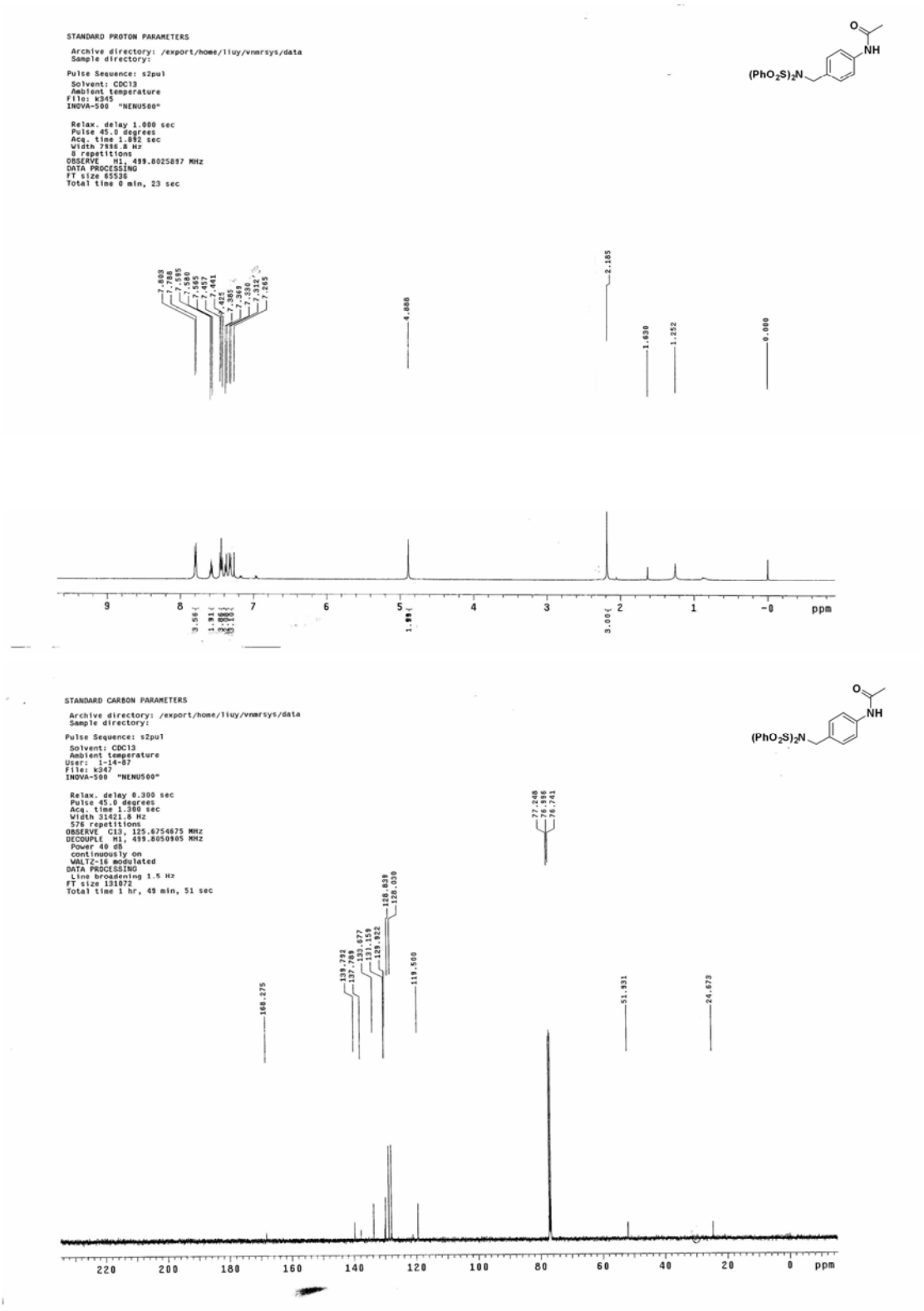

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1933
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
128 repetitions
OBSERVE C13, 125.6754684 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
ppm

Compound 2a

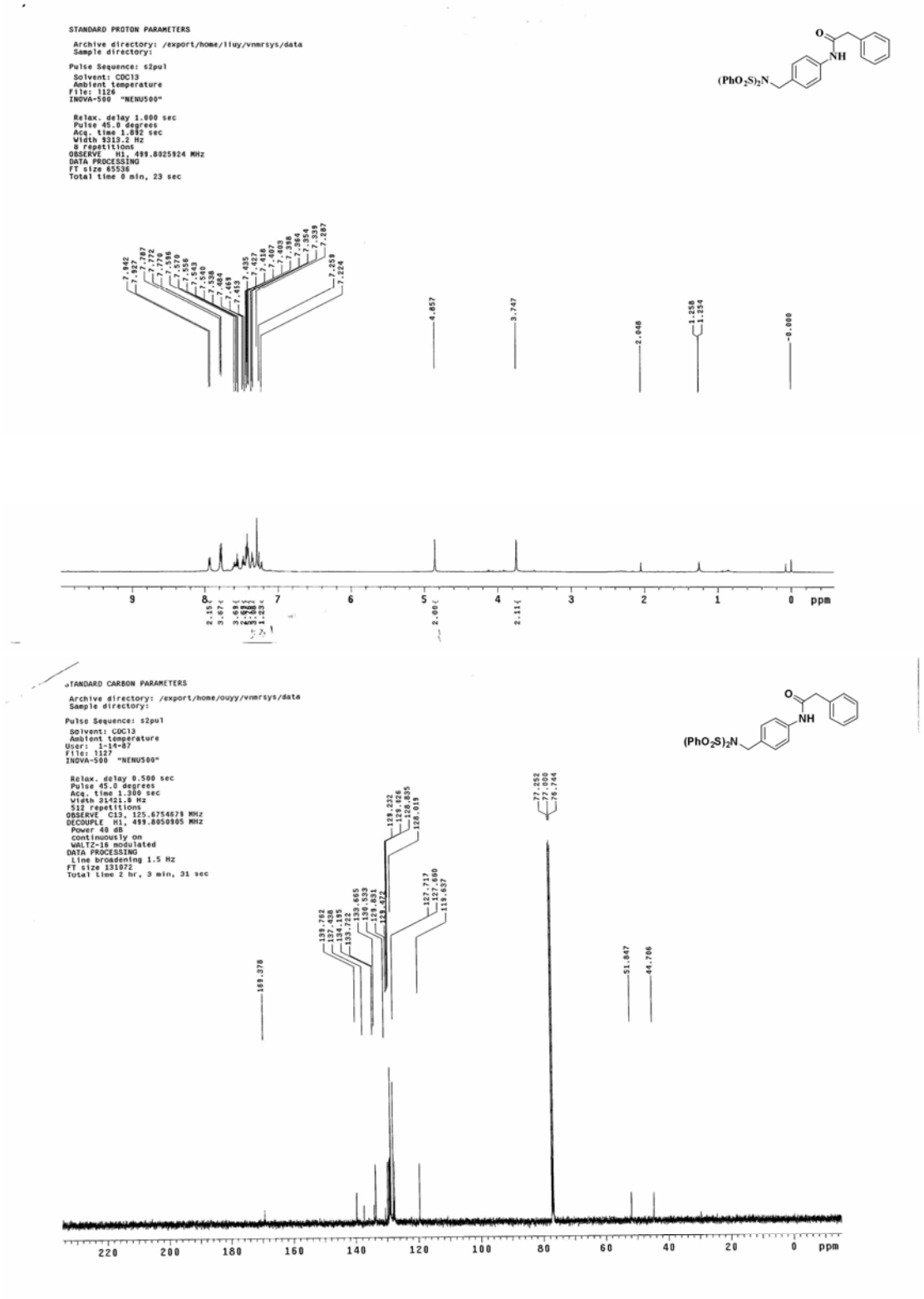

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k483
INOVA-500 "NENUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025907 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.818
7.803
7.801
7.603
7.587
7.572
7.468
7.452
7.436
7.427
7.410
7.384
7.327
7.303
7.283
4.880
1.572
1.341
1.323
0.000
ppm
O
NH
(PhO2S)2N


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: m016
INOVA-500 "NENUS00"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754650 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 9 min, 31 sec
77.271
77.015
76.760
176.630
141.818
137.965
133.677
130.167
128.903
126.862
126.080
119.083
51.682
39.654
27.610
ppm
O
NH
(PhO2S)2N

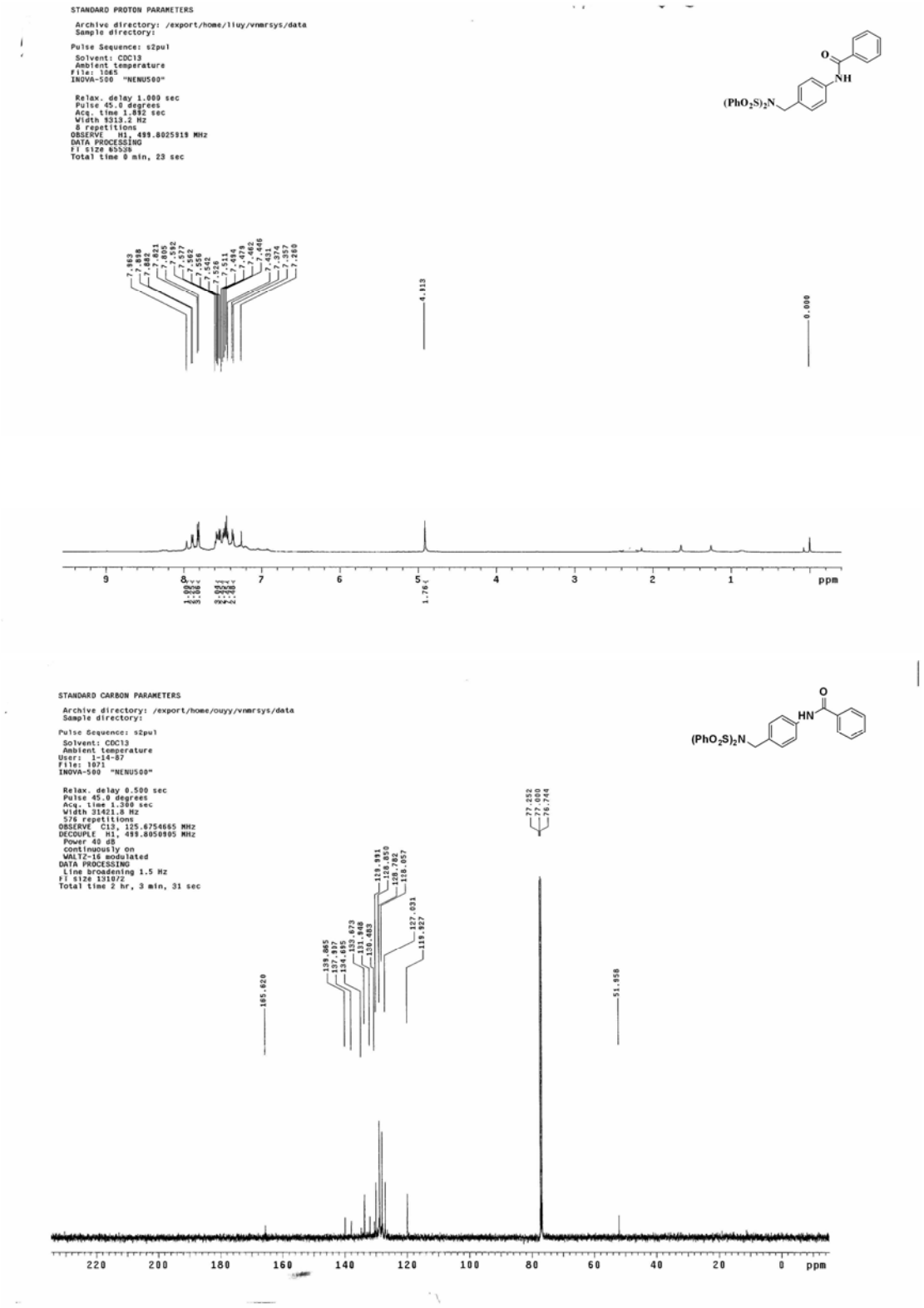
Compound 2b


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k345
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8025897 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
(PhO2S)2N
NH
O
7.803
7.788
7.595
7.580
7.565
7.457
7.441
7.385
7.369
7.330
7.315
7.265
4.888
2.185
1.630
1.252
0.000
9
8
7
6
5
4
3
2
1
-0
ppm
3.56
1.91
1.89
3.00
1.89
STANDARD CARBON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: k347
INOVA-500 "NENU500"
Relax. delay 0.300 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
576 repetitions
OBSERVE C13, 125.6754675 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
(PhO2S)2N
NH
O
77.248
76.996
76.741
130.838
130.588
139.792
137.788
133.677
131.159
128.922
119.580
168.275
51.931
24.673
220
200
180
160
140
120
100
80
60
40
20
0
ppm

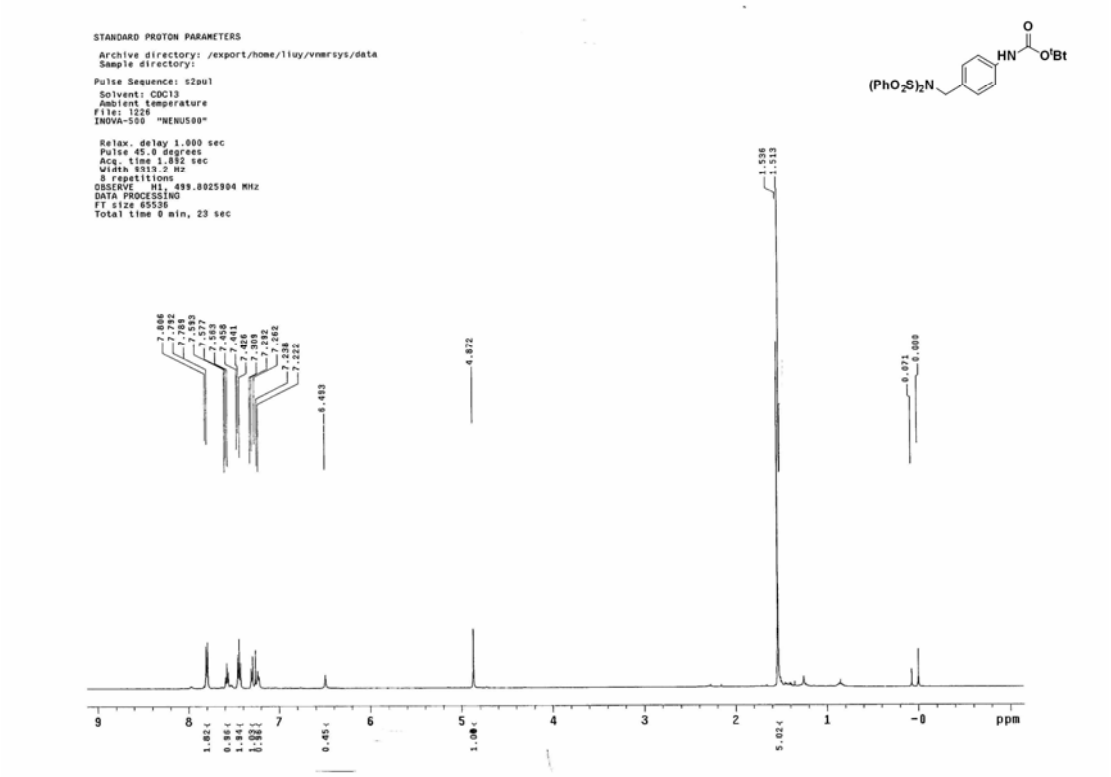
Compound 2c

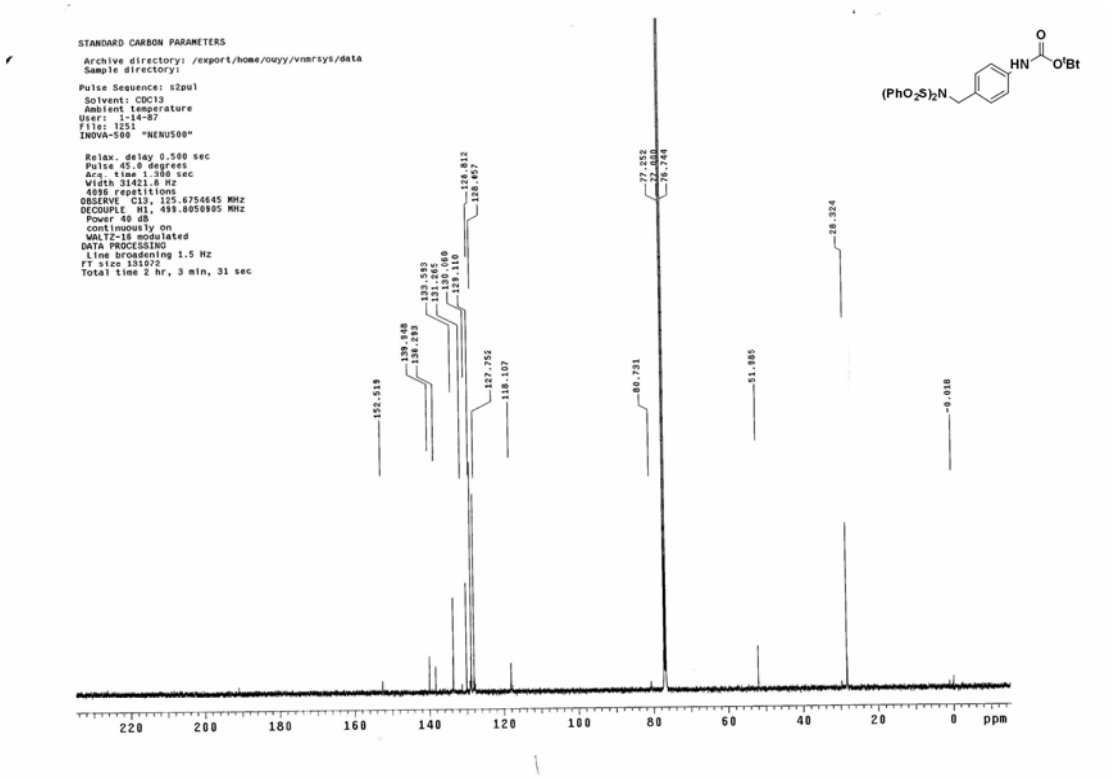

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1126
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8313.2 Hz
8 repetitions
OBSERVE H1, 499.8025924 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
(PhO2S)2N
NH
O
ppm
STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1127
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
512 repetitions
OBSERVE C13, 125.6754675 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
(PhO2S)2N
NH
O
ppm

Compound 2d

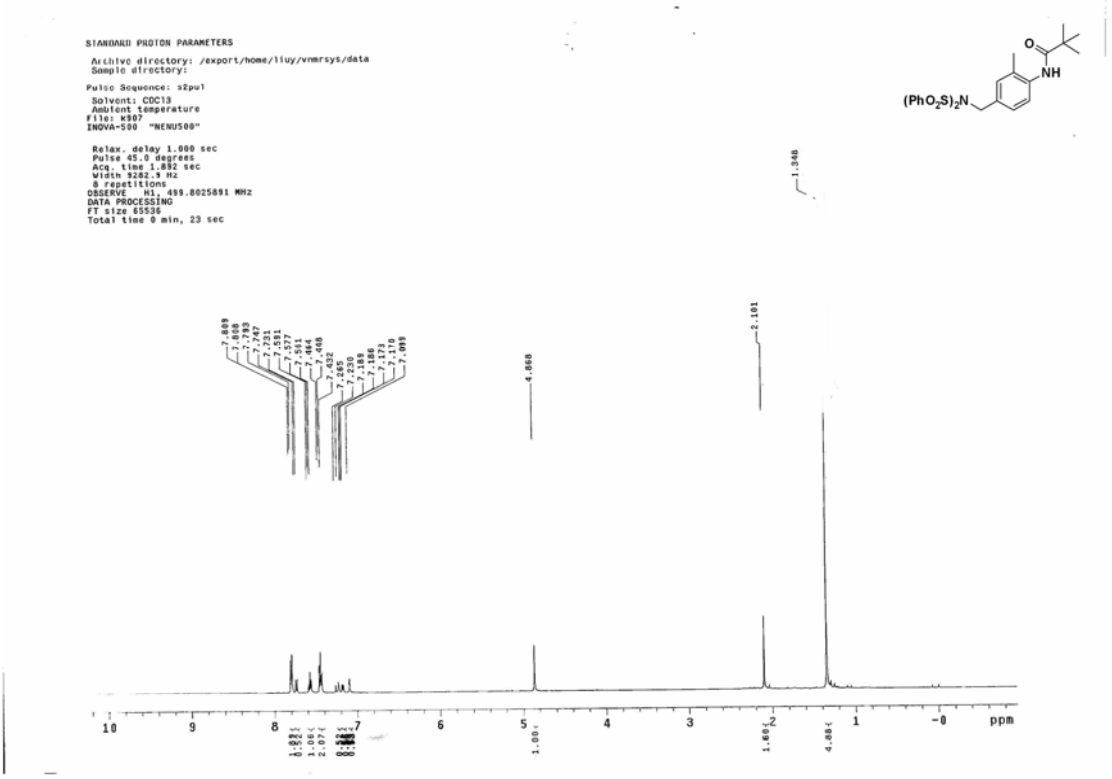

STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1065
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 8313.2 Hz
8 repetitions
OBSERVE H1, 499.8025919 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
7.868
7.858
7.852
7.821
7.809
7.802
7.577
7.562
7.556
7.552
7.526
7.511
7.484
7.478
7.462
7.446
7.431
7.426
7.357
7.260
4.913
0.000
(PhO2S)2N
O
NH
9
8
7
6
5
4
3
2
1
ppm
1.764
STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1071
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
576 repetitions
OBSERVE C13, 125.6754665 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
HN
O
(PhO2S)2N
77.252
77.000
76.744
119.991
128.850
129.288
119.057
127.031
119.927
109.065
137.937
134.695
133.673
131.448
130.483
185.620
51.958
220
200
180
160
140
120
100
80
60
40
20
0
ppm

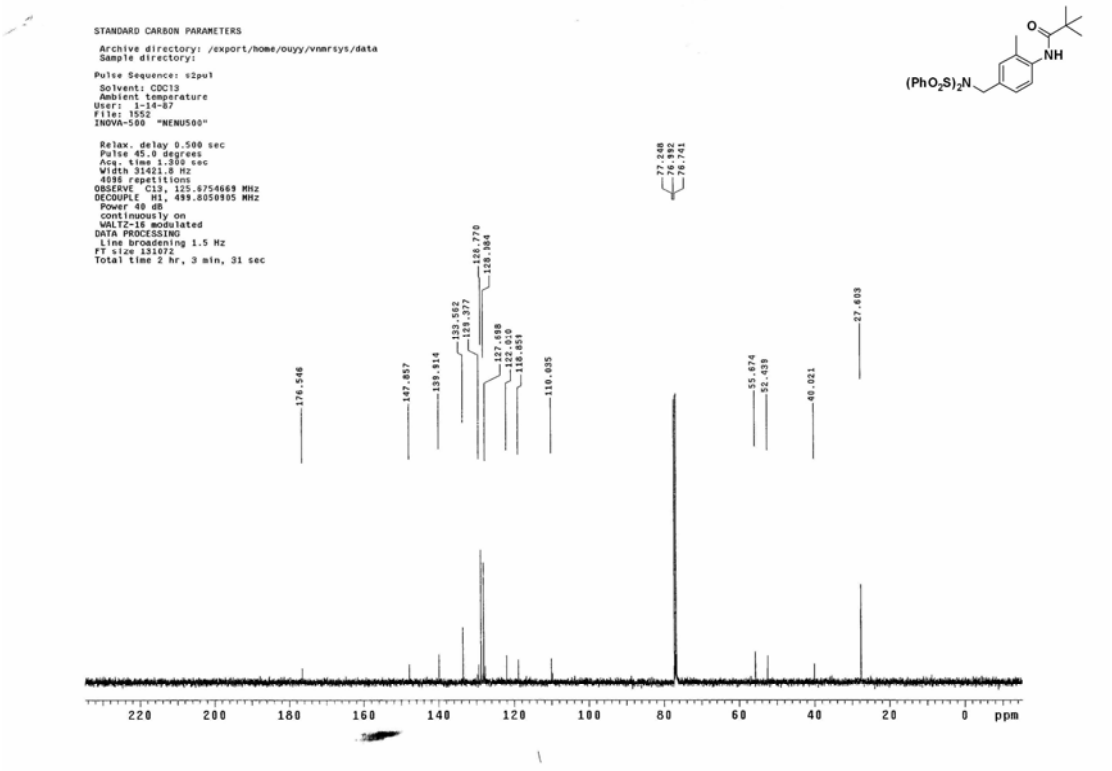
Compound 2e


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1226
INOVA-500 "NENUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.832 sec
Width 6319.2 Hz
8 repetitions
OBSERVE H1, 499.8025904 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
HN
O
O^{t}Bt
(PhO$_2$S)$_2$N
ppm

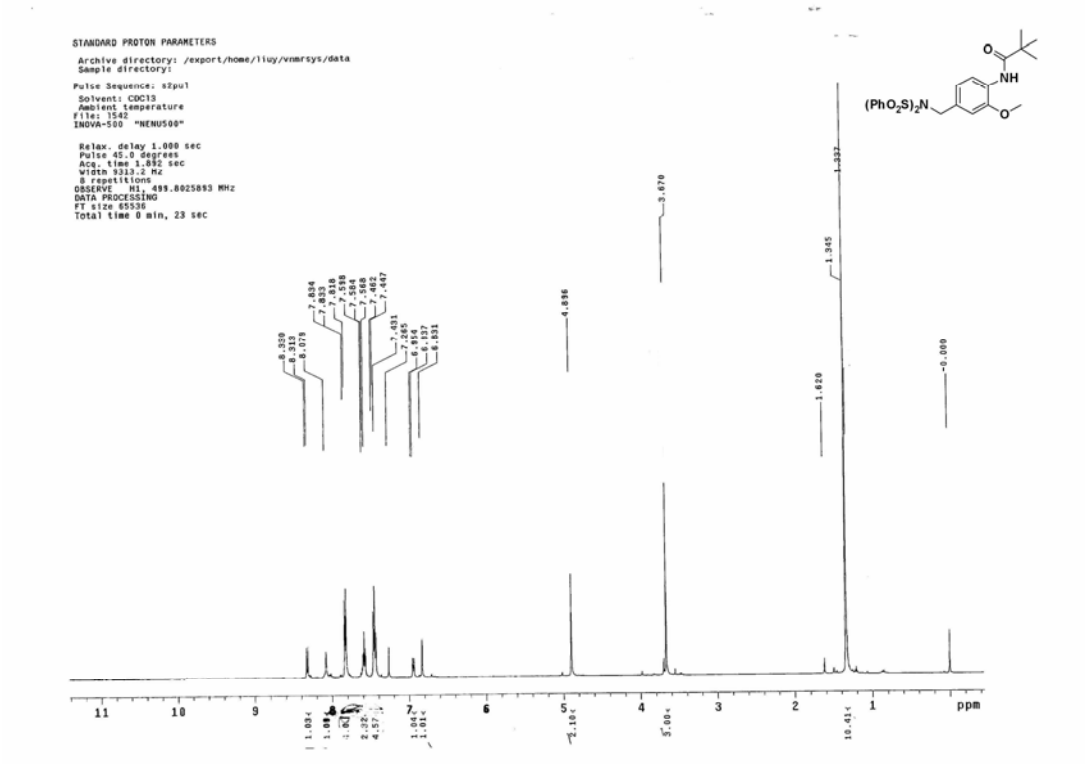

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1251
INOVA-500 "NENUS00"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754645 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
HN
O
O^{t}Bt
(PhO$_2$S)$_2$N
ppm

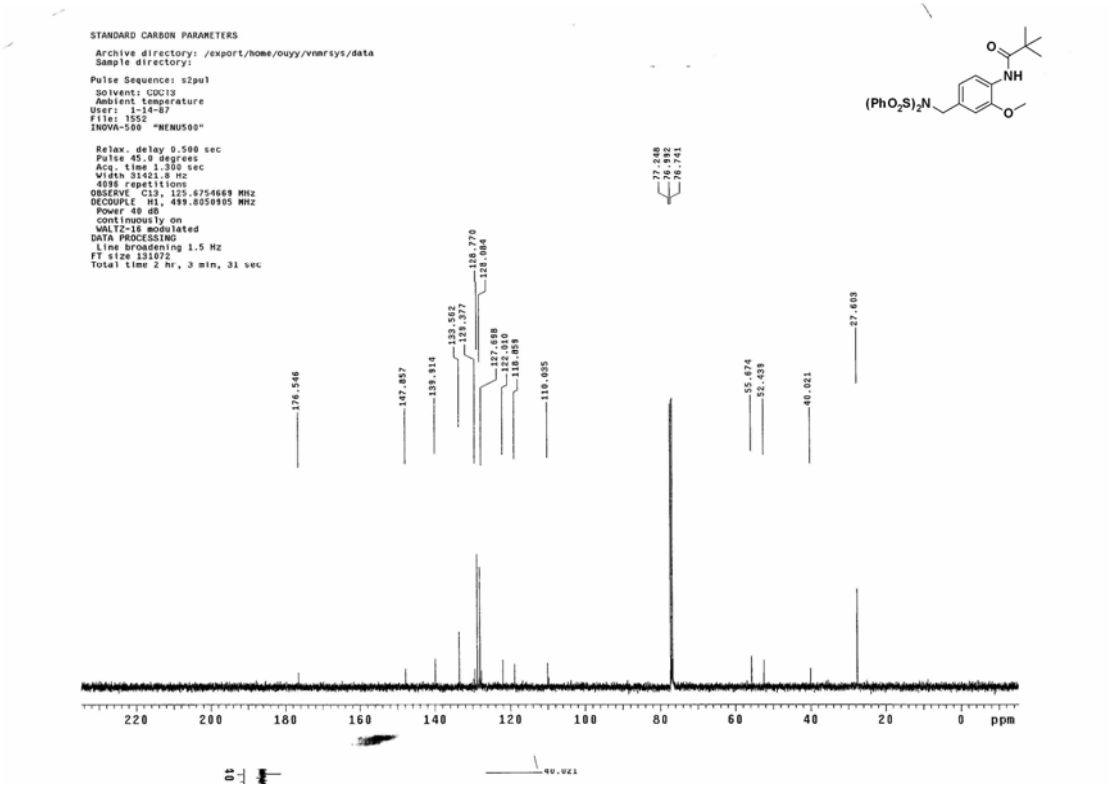
Compound 2f


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: k907
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9282.9 Hz
8 repetitions
OBSERVE H1, 499.8025891 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
(PhO2S)2N
NH
O
ppm

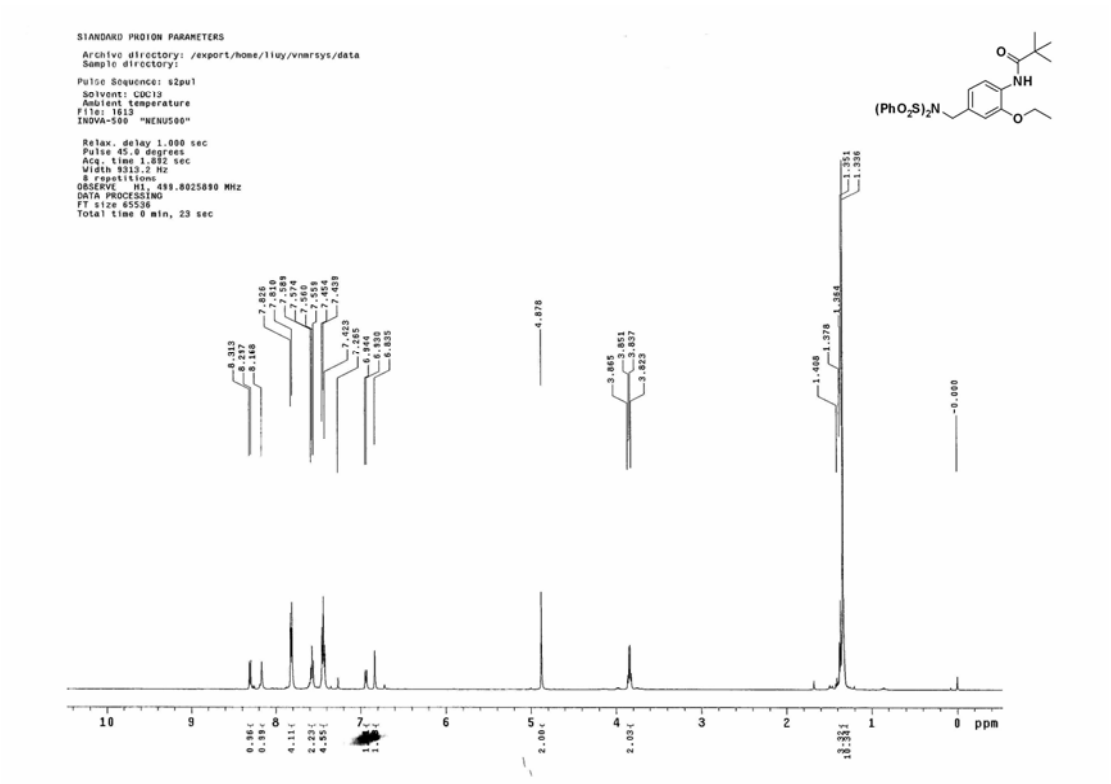

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1552
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754669 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
(PhO2S)2N
NH
O
ppm

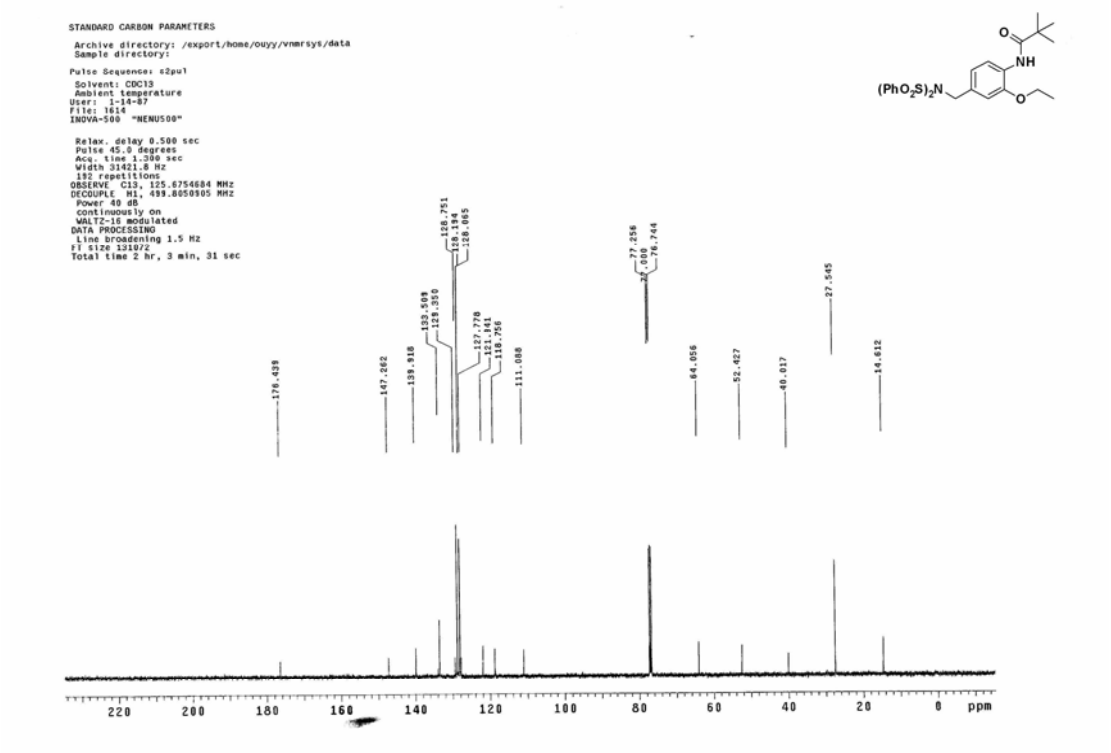
Compound 2g


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1542
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025893 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH
(PhO2S)2N
O
ppm

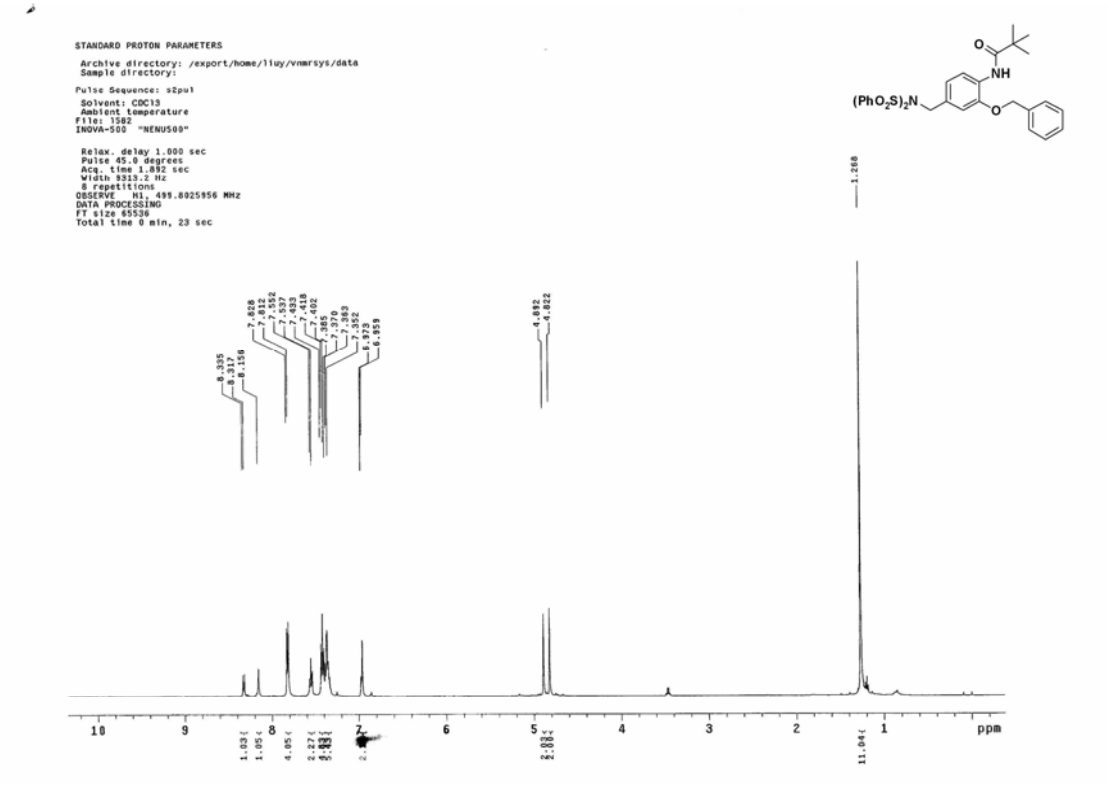

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1552
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.8754669 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
NH
(PhO2S)2N
O
ppm

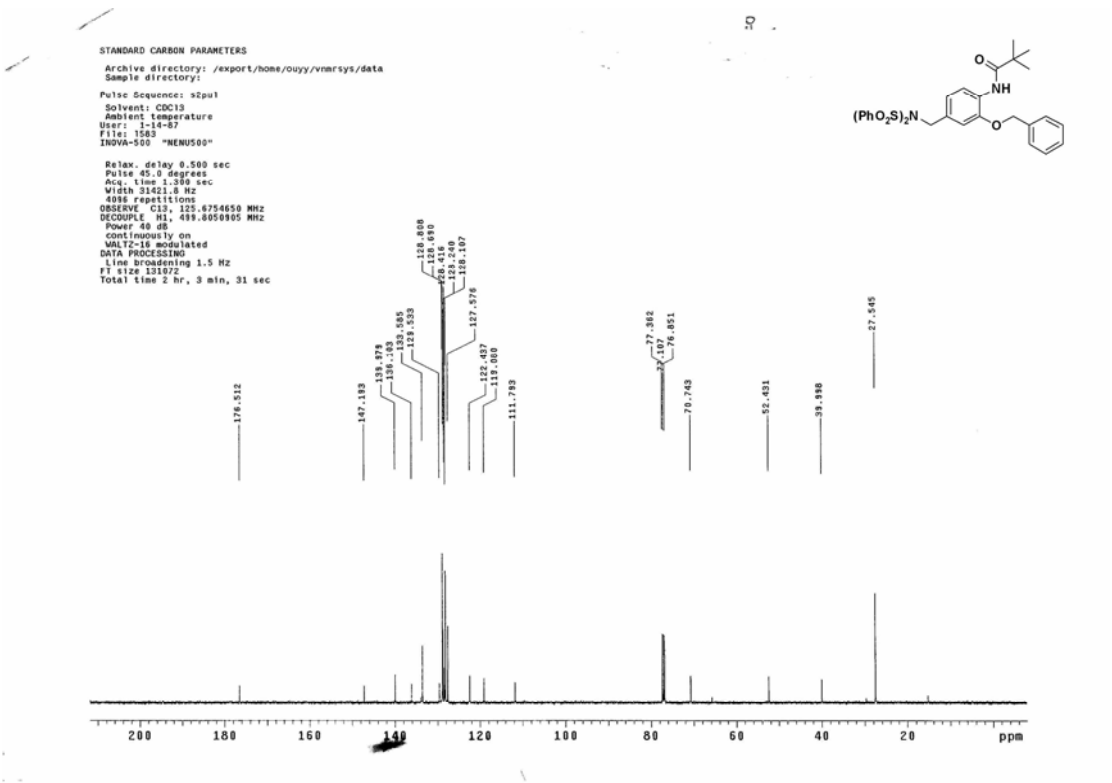
Compound 2h


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1613
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025890 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
NH
(PhO2S)2N
O
ppm

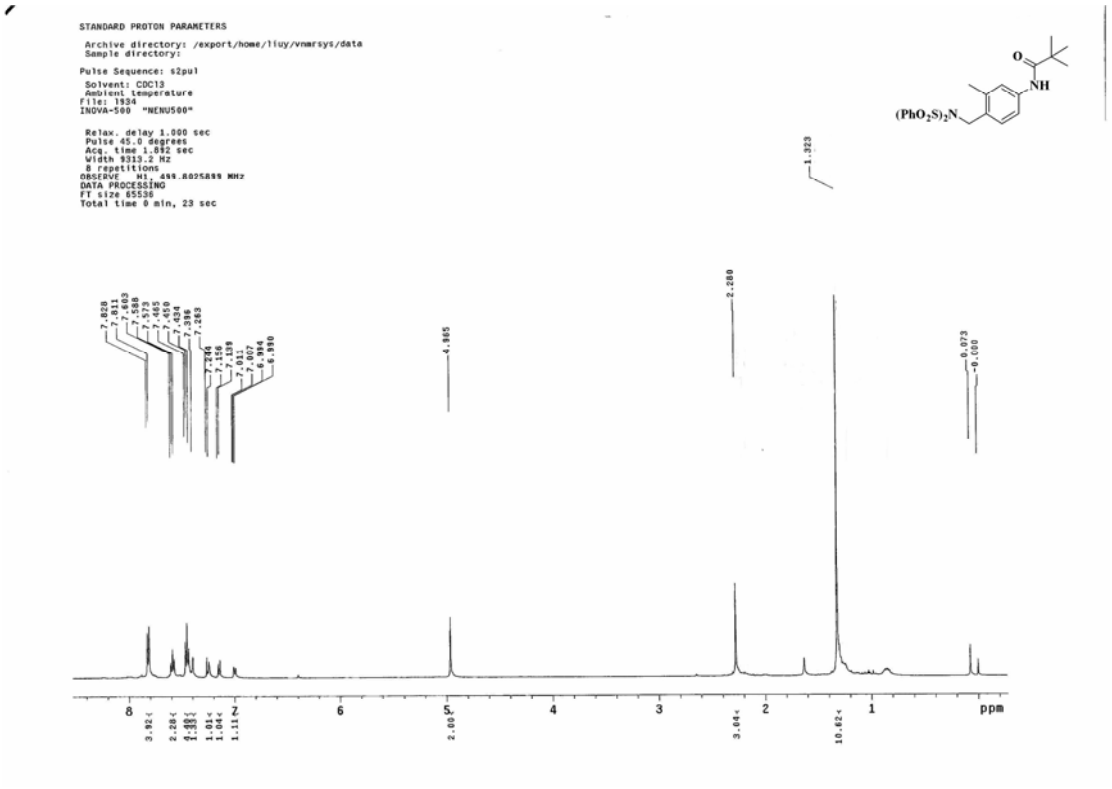

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-07
File: 1614
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754684 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
NH
(PhO2S)2N
O
ppm

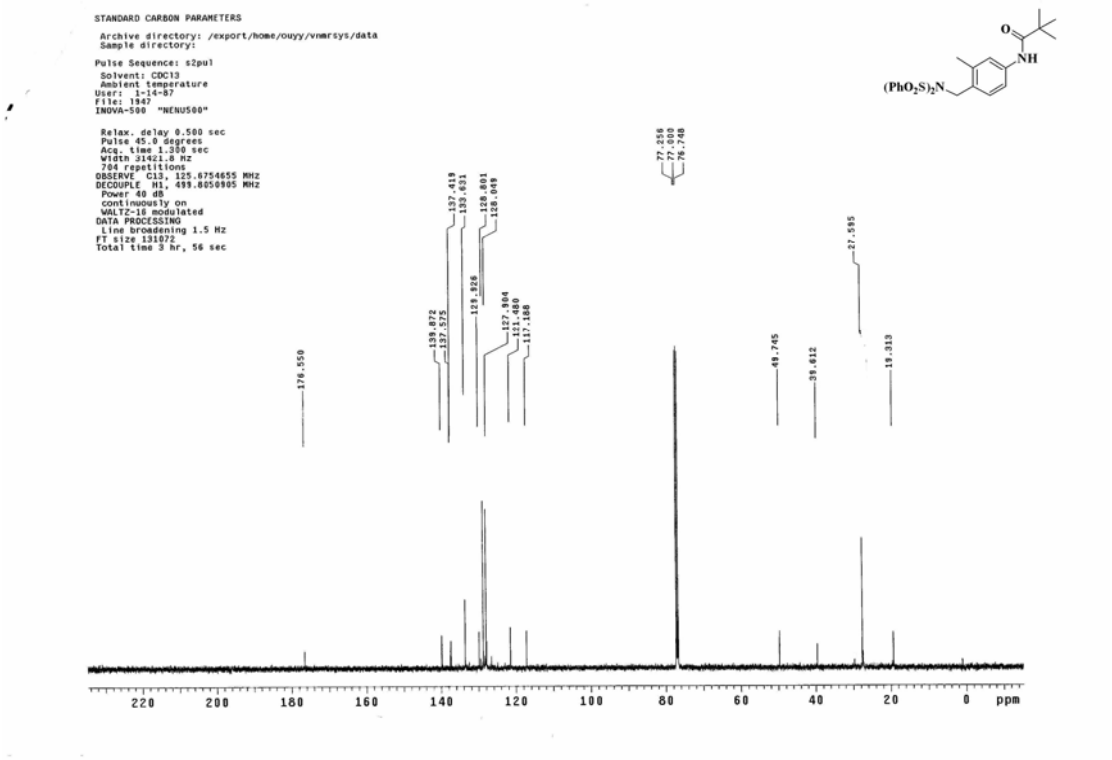
Compound 2i


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1582
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025956 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
(PhO2S)2N
NH
O
ppm

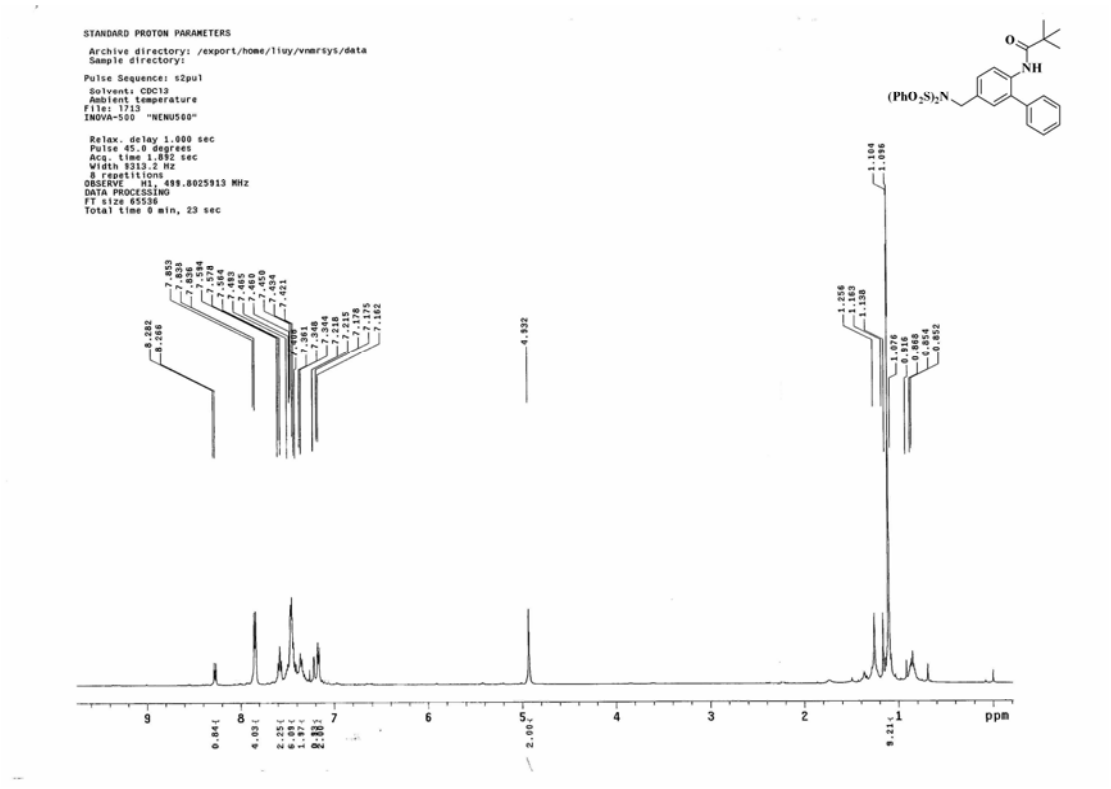

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1583
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
4096 repetitions
OBSERVE C13, 125.6754650 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
(PhO2S)2N
NH
O
ppm

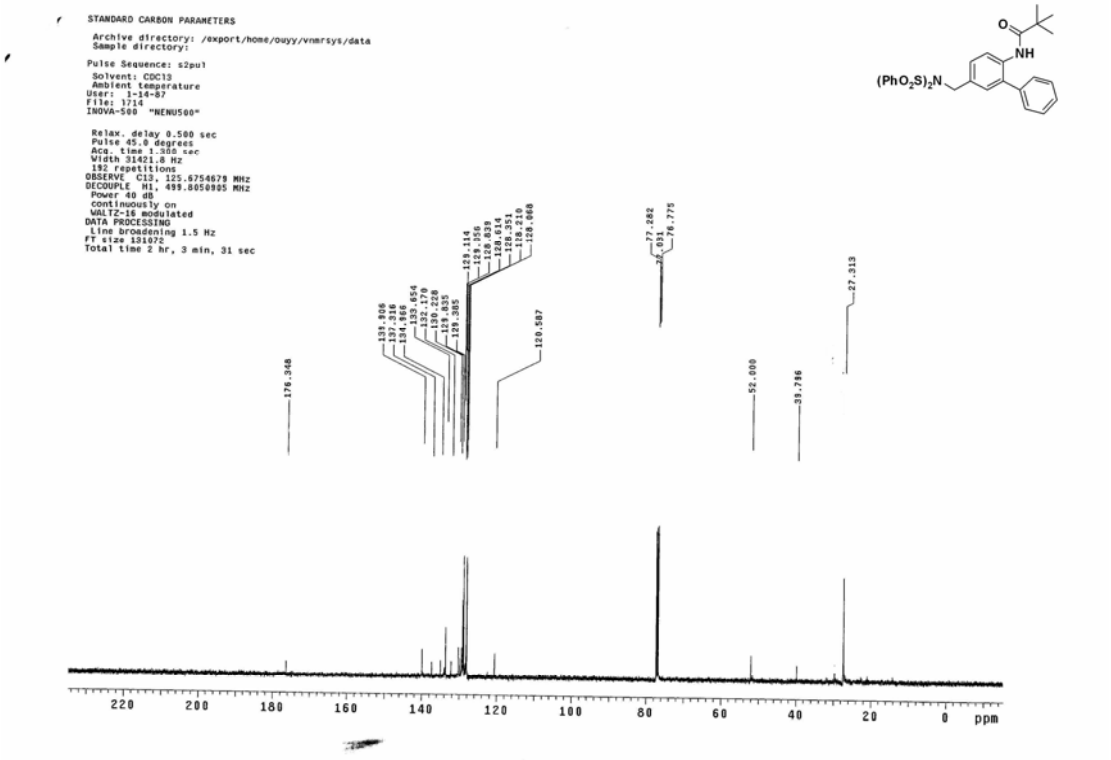
Compound 2j


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1934
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025899 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
O
NH
(PhO2S)2N
ppm

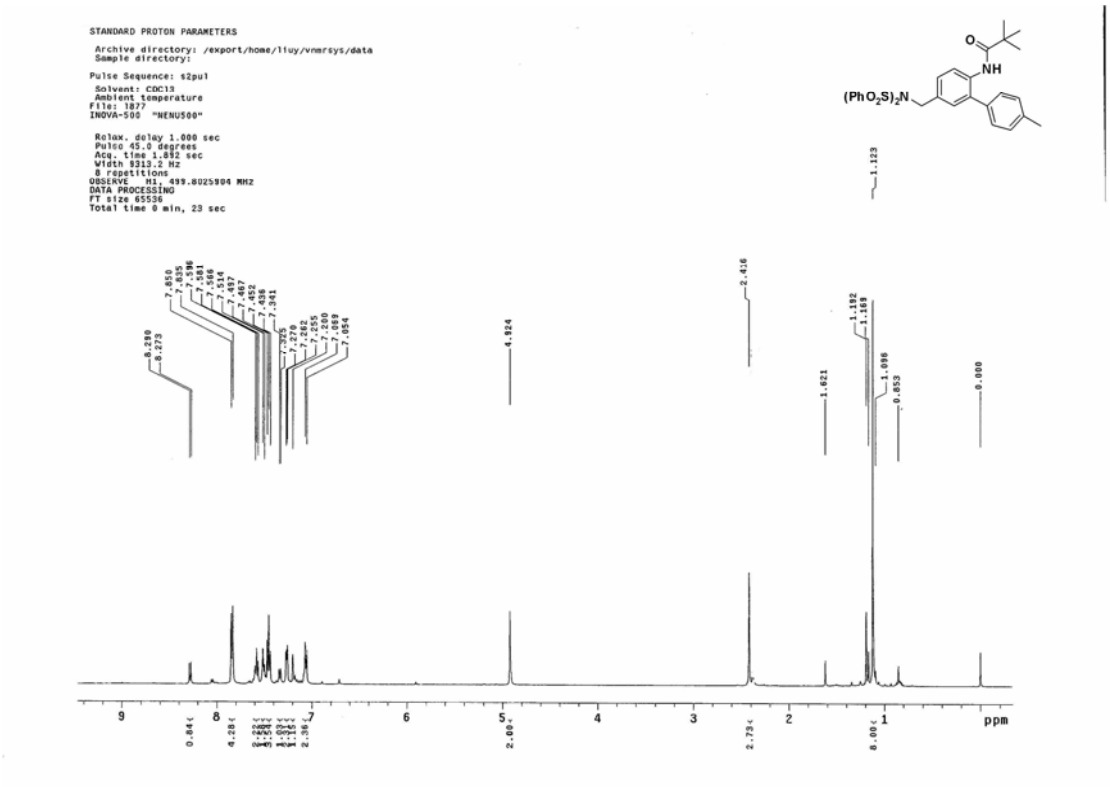

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1947
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.380 sec
Width 21421.8 Hz
704 repetitions
OBSERVE C13, 125.6754655 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 3 hr, 56 sec
O
NH
(PhO2S)2N
ppm

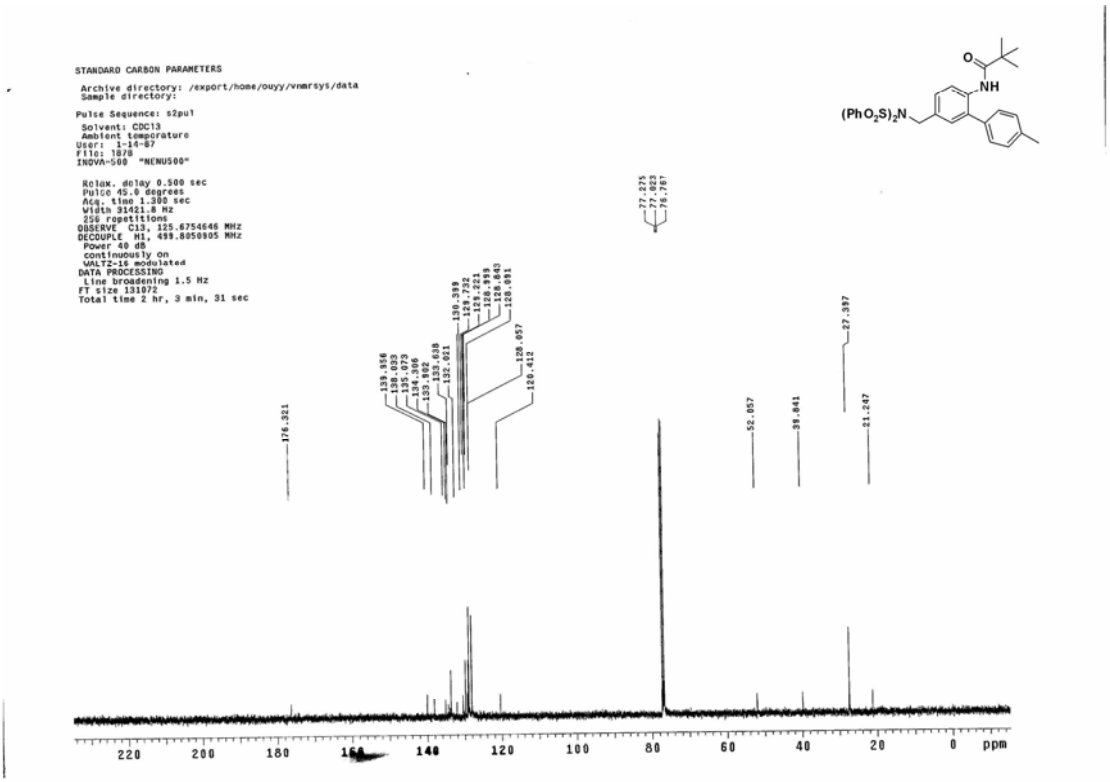
Compound 2k


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1713
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025913 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
ppm

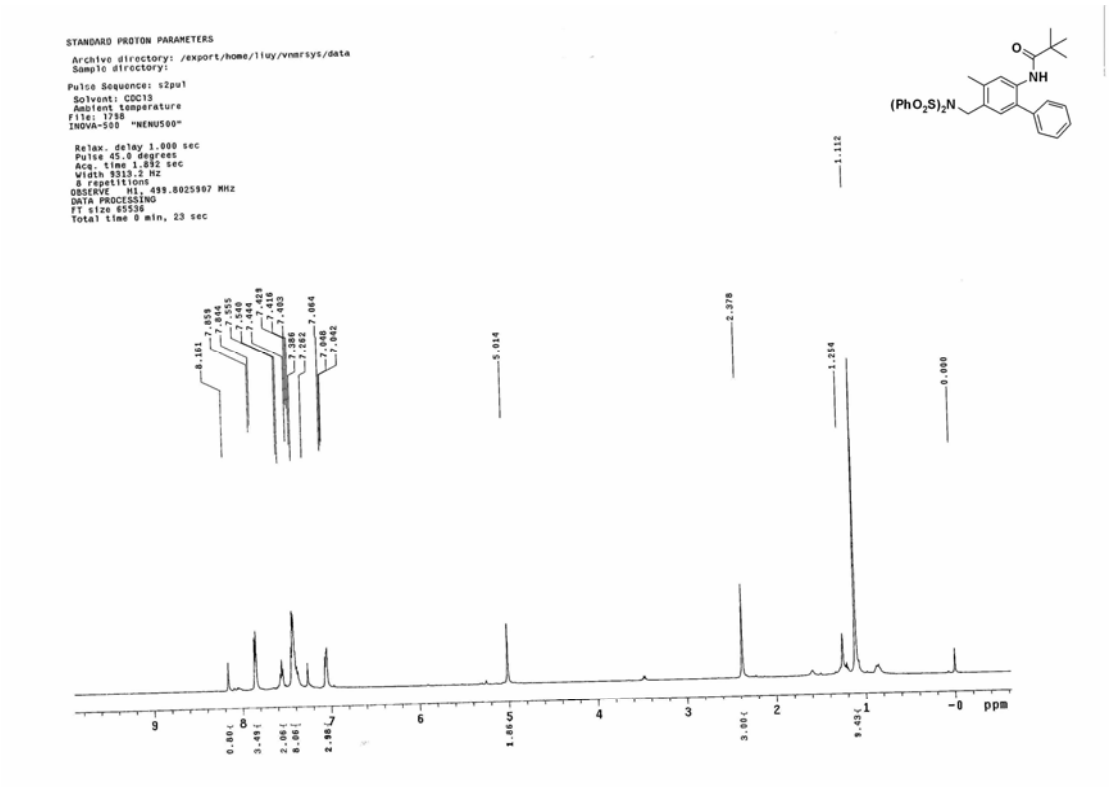

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1714
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.306 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754679 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
ppm

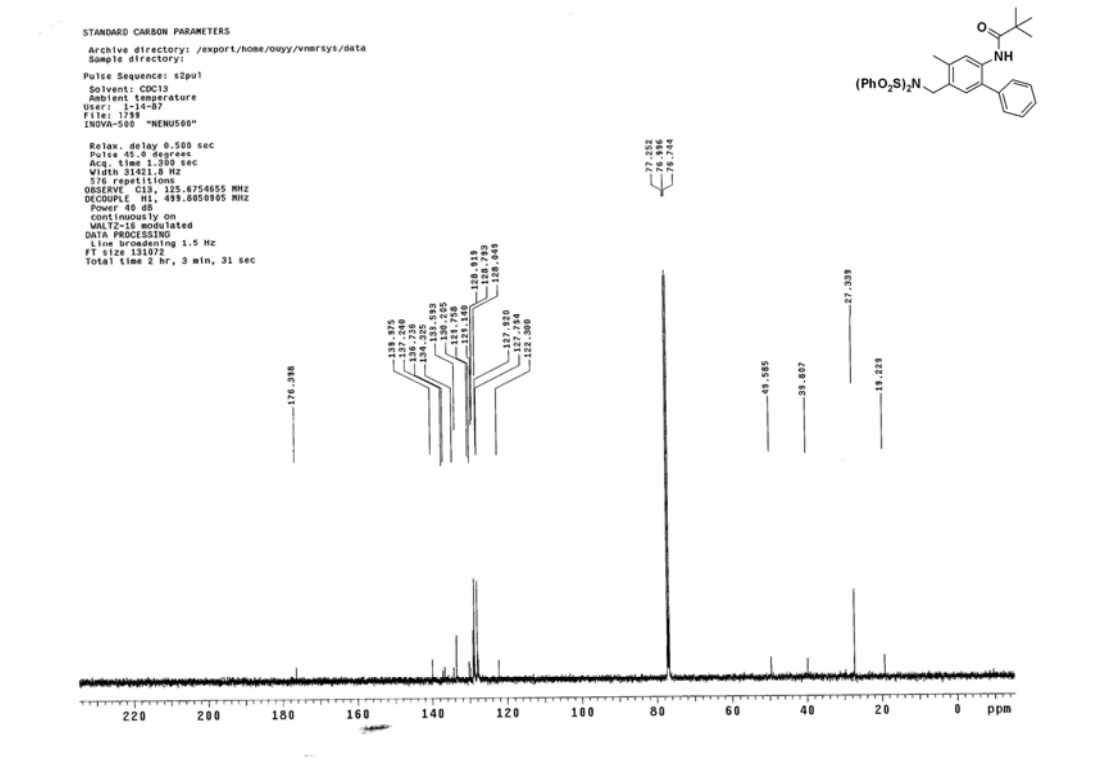
Compound 2l


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1877
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025904 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
(PhO2S)2N
NH
O
ppm

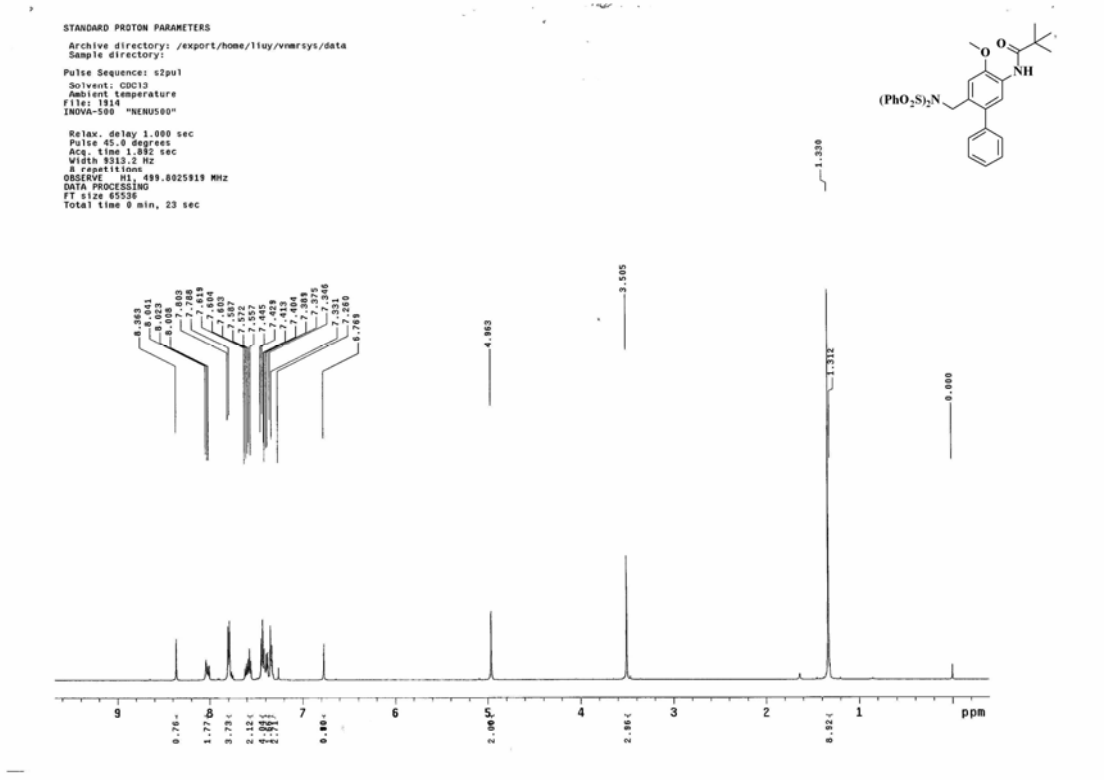

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1878
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
256 repetitions
OBSERVE C13, 125.6754646 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
(PhO2S)2N
NH
O
ppm

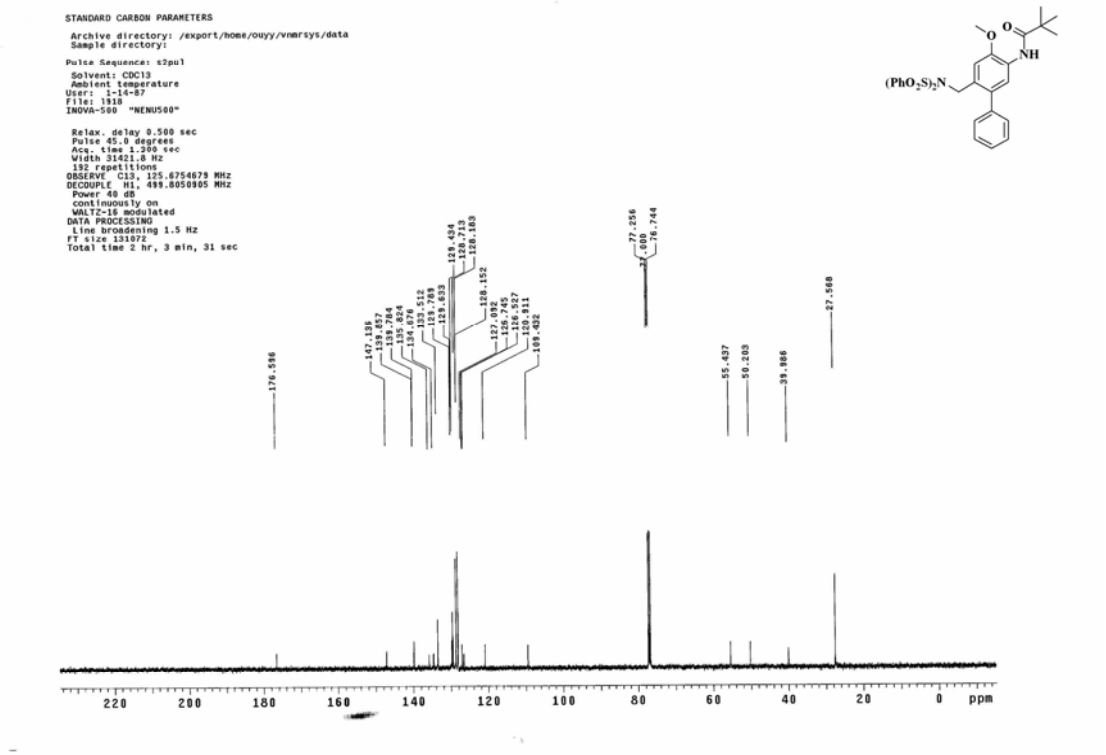
Compound 2m


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1798
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025907 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
(PhO2S)2N
NH
O
8.161
7.859
7.844
7.555
7.540
7.466
7.428
7.416
7.403
7.366
7.262
7.064
7.048
7.042
5.014
2.378
1.254
1.112
0.000
ppm

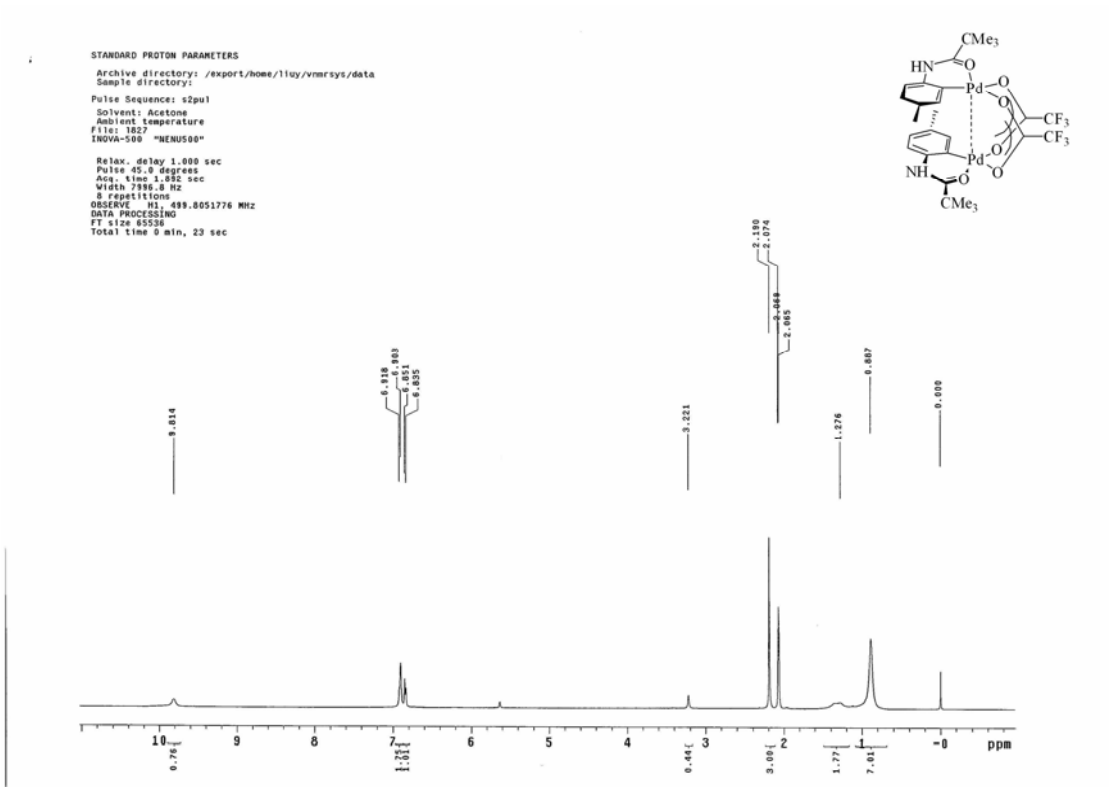

STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1799
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
576 repetitions
OBSERVE C13, 125.6754655 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
(PhO2S)2N
NH
O
176.398
139.975
137.240
136.736
134.325
131.583
130.205
121.758
121.140
128.919
128.793
128.649
127.920
127.744
122.309
77.252
76.996
76.744
69.585
39.857
27.339
18.228
ppm

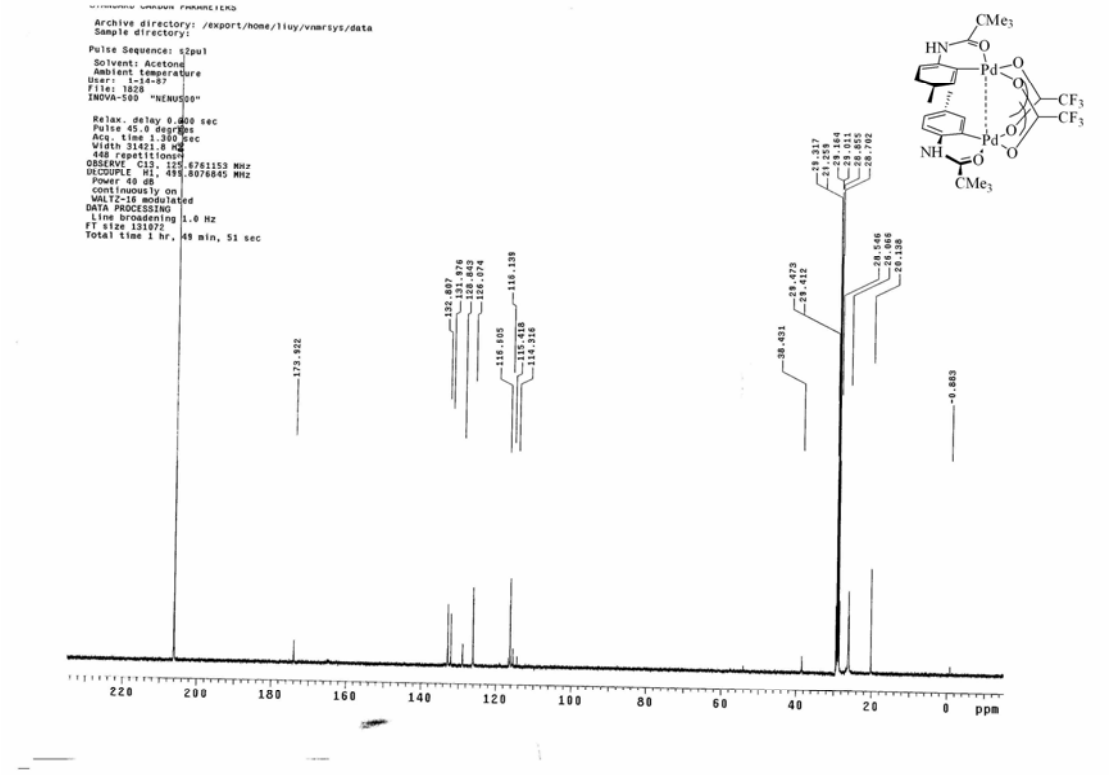
Compound 2n


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: 1914
INOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.882 sec
Width 9313.2 Hz
8 repetitions
OBSERVE H1, 499.8025919 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
(PhO2S)2N
NH
ppm


STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: 1-14-87
File: 1918
INOVA-500 "NENU500"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
192 repetitions
OBSERVE C13, 125.6754679 MHz
DECOUPLE H1, 499.8050905 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec
(PhO2S)2N
NH
ppm

Compound E


STANDARD PROTON PARAMETERS
Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: Acetone
Ambient temperature
File: 1827
INOVA-500 "NENUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7986.8 Hz
8 repetitions
OBSERVE H1, 499.8051776 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec
CMe3
HN
Pd
O
CF3
NH
ppm


Archive directory: /export/home/liuy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: Acetone
Ambient temperature
User: 1-14-07
File: 1828
INOVA-500 "NENUS00"
Relax. delay 0.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31421.8 Hz
448 repetitions
OBSERVE C13, 125.6761153 MHz
DECOUPLE H1, 499.8076845 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 1 hr, 49 min, 51 sec
CMe3
HN
Pd
O
CF3
NH
ppm

Ball-and-stick Representation of Bimetallic Pd Complex E

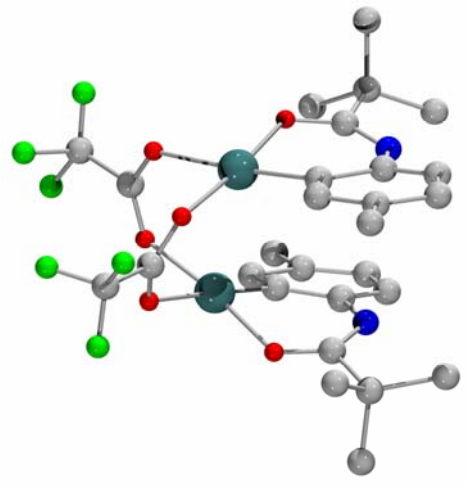


Table 1. Crystal data and structure refinement for 1.

Identification code	**1**
Empirical formula	**C H F0.01 N O Pd**
Formula weight	**149.64**
Temperature	**293(2) K**
Wavelength	**0.71073 A**
Crystal system, space group	**Orthorhombic, Pbcn**
Unit cell dimensions	**a = 10.9301(6) A alpha = 90 deg.** **b = 16.8303(8) A beta = 90 deg.** **c = 40.821(2) A gamma = 90 deg.**
Volume	**7509.3(7) A^3**
Z, Calculated density	**90, 2.978 Mg/m^3**
Absorption coefficient	**5.303 mm^-1**
F(000)	**6129**

Crystal size	**0.21 x 0.24 x 0.26 mm**
Theta range for data collection	**2.00 to 25.04 deg.**
Limiting indices	**-12<=h<=9, -19<=k<=20, -48<=l<=36**
Reflections collected / unique	**31157 / 6615 [R(int) = 0.0832]**
Completeness to theta = 25.04	**99.8 %**
Absorption correction	**Numerical**
Refinement method	**Full-matrix least-squares on F^2**
Data / restraints / parameters	**6615 / 0 / 191**
Goodness-of-fit on F^2	**1.925**
Final R indices [I>2sigma(I)]	**R1 = 0.1261, wR2 = 0.3407**
R indices (all data)	**R1 = 0.1909, wR2 = 0.3617**
Largest diff. peak and hole	**3.809 and -0.766 e.A^-3**

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 1.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Pd(1)	4064(1)	837(1)	1195(1)	62(1)
Pd(2)	5074(1)	2352(1)	1456(1)	72(1)
O(3)	5610(12)	884(7)	907(3)	79(3)
O(5)	5167(11)	420(7)	1591(3)	79(3)
O(2)	2627(12)	669(7)	1481(3)	81(3)
N(1)	2830(14)	3488(8)	1245(3)	74(4)
O(1)	4715(13)	3106(8)	1105(3)	94(4)
O(4)	6477(12)	1920(7)	1143(3)	86(3)
O(6)	5627(11)	1584(7)	1832(3)	81(3)
F(1)	5727(12)	-250(7)	2165(3)	118(4)
C(9)	3017(16)	1200(9)	840(4)	66(4)
C(10)	5593(16)	856(10)	1801(4)	72(5)
C(12)	3813(17)	2836(10)	1744(5)	81(5)
F(3)	6263(16)	834(9)	2351(4)	160(5)
C(14)	2835(15)	3328(9)	1568(4)	66(4)
N(2)	1398(13)	1522(8)	1245(3)	72(4)
F(5)	7982(16)	1960(10)	608(4)	175(6)
F(2)	7288(16)	210(9)	2000(4)	167(5)
C(18)	6427(18)	1381(11)	952(4)	75(5)
C(19)	1904(17)	1534(10)	919(4)	76(5)
C(20)	3590(20)	3860(12)	701(5)	99(6)
C(21)	2250(20)	4032(12)	596(5)	106(7)
F(4)	8400(20)	993(11)	866(5)	226(8)
C(23)	1990(20)	3659(12)	1750(5)	95(6)
C(24)	820(20)	946(12)	1752(6)	102(7)
C(25)	3790(20)	2735(12)	2074(5)	103(6)
C(26)	1150(20)	1839(12)	660(5)	99(6)
C(27)	6200(20)	414(14)	2090(6)	104(7)
F(6)	7477(17)	734(11)	498(4)	196(6)
C(30)	1910(20)	3534(13)	2103(6)	117(7)
C(31)	210(20)	141(14)	1714(5)	111(7)
C(32)	2857(19)	3076(11)	2258(5)	96(6)
C(33)	3390(20)	1096(12)	518(5)	99(6)

C(34)	**1726(18)**	**1064(11)**	**1478(4)**	**79(5)**
C(35)	**4320(20)**	**4595(13)**	**733(5)**	**108(7)**
C(37)	**3713(19)**	**3435(11)**	**1040(5)**	**81(5)**
C(38)	**-220(30)**	**1599(17)**	**1790(6)**	**146(9)**
C(39)	**2590(20)**	**1434(14)**	**265(6)**	**122(8)**
C(40)	**7470(30)**	**1337(17)**	**684(6)**	**136(8)**
C(41)	**1520(20)**	**1811(13)**	**331(5)**	**108(7)**
C(42)	**1570(30)**	**879(16)**	**2082(7)**	**172(11)**
C(43)	**3030(30)**	**1332(15)**	**-92(6)**	**145(9)**
C(44)	**4160(20)**	**3252(12)**	**414(5)**	**104(6)**
C(45)	**2910(30)**	**2941(15)**	**2658(7)**	**145(9)**

Table 3. Bond lengths [A] and angles [deg] for 1.

Pd(1)-C(9)	**1.945(16)**
Pd(1)-O(2)	**1.978(12)**
Pd(1)-O(3)	**2.060(13)**
Pd(1)-O(5)	**2.135(12)**
Pd(1)-Pd(2)	**2.9750(18)**
Pd(2)-C(12)	**1.987(18)**
Pd(2)-O(1)	**1.953(13)**
Pd(2)-O(6)	**2.095(12)**
Pd(2)-O(4)	**2.123(13)**
O(3)-C(18)	**1.237(19)**
O(5)-C(10)	**1.222(18)**
O(2)-C(34)	**1.19(2)**
N(1)-C(14)	**1.346(18)**
N(1)-C(37)	**1.28(2)**
O(1)-C(37)	**1.26(2)**
O(4)-C(18)	**1.196(18)**
O(6)-C(10)	**1.232(18)**
F(1)-C(27)	**1.27(2)**
C(9)-C(33)	**1.39(2)**
C(9)-C(19)	**1.38(2)**
C(10)-C(27)	**1.55(3)**
C(12)-C(25)	**1.36(2)**
C(12)-C(14)	**1.53(2)**
F(3)-C(27)	**1.28(2)**
C(14)-C(23)	**1.31(2)**
N(2)-C(34)	**1.276(19)**
N(2)-C(19)	**1.44(2)**
F(5)-C(40)	**1.23(3)**
F(2)-C(27)	**1.29(2)**
C(18)-C(40)	**1.58(3)**
C(19)-C(26)	**1.44(2)**
C(20)-C(37)	**1.56(3)**
C(20)-C(35)	**1.48(3)**
C(20)-C(21)	**1.55(3)**
C(20)-C(44)	**1.68(3)**
F(4)-C(40)	**1.39(3)**
C(23)-C(30)	**1.46(3)**
C(24)-C(31)	**1.52(3)**
C(24)-C(42)	**1.58(3)**
C(24)-C(38)	**1.59(3)**

C(24)-C(34)	**1.51(3)**
C(25)-C(32)	**1.39(3)**
C(26)-C(41)	**1.40(3)**
C(26)-C(54)	**1.85(8)**
F(6)-C(40)	**1.27(3)**
C(30)-C(32)	**1.43(3)**
C(32)-C(45)	**1.65(3)**
C(33)-C(39)	**1.47(3)**
C(39)-C(41)	**1.36(3)**
C(39)-C(43)	**1.55(3)**

C(9)-Pd(1)-O(2)	**91.1(6)**
C(9)-Pd(1)-O(3)	**92.6(6)**
O(2)-Pd(1)-O(3)	**173.7(5)**
C(9)-Pd(1)-O(5)	**178.3(6)**
O(2)-Pd(1)-O(5)	**87.3(5)**
O(3)-Pd(1)-O(5)	**88.9(4)**
C(9)-Pd(1)-Pd(2)	**102.5(5)**
O(2)-Pd(1)-Pd(2)	**101.9(3)**
O(3)-Pd(1)-Pd(2)	**82.3(3)**
O(5)-Pd(1)-Pd(2)	**78.6(3)**
C(12)-Pd(2)-O(1)	**91.6(7)**
C(12)-Pd(2)-O(6)	**91.1(6)**
O(1)-Pd(2)-O(6)	**174.6(5)**
C(12)-Pd(2)-O(4)	**175.8(6)**
O(1)-Pd(2)-O(4)	**85.8(5)**
O(6)-Pd(2)-O(4)	**91.2(5)**
C(12)-Pd(2)-Pd(1)	**107.8(5)**
O(1)-Pd(2)-Pd(1)	**102.7(4)**
O(6)-Pd(2)-Pd(1)	**80.8(3)**
O(4)-Pd(2)-Pd(1)	**76.1(3)**
C(18)-O(3)-Pd(1)	**122.2(12)**
C(10)-O(5)-Pd(1)	**123.4(11)**
C(34)-O(2)-Pd(1)	**124.8(13)**
C(14)-N(1)-C(37)	**128.5(17)**
C(37)-O(1)-Pd(2)	**128.0(13)**
C(18)-O(4)-Pd(2)	**128.1(13)**
C(10)-O(6)-Pd(2)	**122.1(12)**
C(33)-C(9)-C(19)	**122.3(17)**
C(33)-C(9)-Pd(1)	**119.6(14)**
C(19)-C(9)-Pd(1)	**118.1(13)**
O(5)-C(10)-O(6)	**132.8(17)**
O(5)-C(10)-C(27)	**114.3(16)**
O(6)-C(10)-C(27)	**112.9(17)**

C(25)-C(12)-C(14)	**121.3(17)**
C(25)-C(12)-Pd(2)	**123.4(15)**
C(14)-C(12)-Pd(2)	**115.3(12)**
N(1)-C(14)-C(23)	**117.9(17)**
N(1)-C(14)-C(12)	**124.8(15)**
C(23)-C(14)-C(12)	**117.2(16)**
C(34)-N(2)-C(19)	**126.1(16)**
O(3)-C(18)-O(4)	**130.0(19)**
O(3)-C(18)-C(40)	**112.5(18)**
O(4)-C(18)-C(40)	**117.1(19)**
N(2)-C(19)-C(9)	**123.4(16)**
N(2)-C(19)-C(26)	**117.7(17)**
C(9)-C(19)-C(26)	**118.6(17)**
C(37)-C(20)-C(35)	**105.0(17)**
C(37)-C(20)-C(21)	**114.3(18)**
C(35)-C(20)-C(21)	**112.0(18)**
C(37)-C(20)-C(44)	**108.0(15)**
C(35)-C(20)-C(44)	**112.0(18)**
C(21)-C(20)-C(44)	**105.6(16)**
C(30)-C(23)-C(14)	**122(2)**
C(31)-C(24)-C(42)	**104.7(19)**
C(31)-C(24)-C(38)	**108(2)**
C(42)-C(24)-C(38)	**110(2)**
C(31)-C(24)-C(34)	**109.4(18)**
C(42)-C(24)-C(34)	**108(2)**
C(38)-C(24)-C(34)	**116.7(19)**
C(12)-C(25)-C(32)	**120(2)**
C(41)-C(26)-C(19)	**122(2)**
C(41)-C(26)-C(54)	**135(3)**
C(19)-C(26)-C(54)	**102(3)**
F(1)-C(27)-F(3)	**107.9(19)**
F(1)-C(27)-F(2)	**102(2)**
F(3)-C(27)-F(2)	**110(2)**
F(1)-C(27)-C(10)	**115(2)**
F(3)-C(27)-C(10)	**113.0(19)**
F(2)-C(27)-C(10)	**107.8(19)**
C(23)-C(30)-C(32)	**118(2)**
C(25)-C(32)-C(30)	**120.7(19)**
C(25)-C(32)-C(45)	**116.9(19)**
C(30)-C(32)-C(45)	**122(2)**
C(9)-C(33)-C(39)	**116(2)**
O(2)-C(34)-N(2)	**125.4(18)**
O(2)-C(34)-C(24)	**117.5(18)**
N(2)-C(34)-C(24)	**116.7(18)**

O(1)-C(37)-N(1)	**123.5(18)**
O(1)-C(37)-C(20)	**117.6(18)**
N(1)-C(37)-C(20)	**118.8(18)**
C(33)-C(39)-C(41)	**124(2)**
C(33)-C(39)-C(43)	**115(2)**
C(41)-C(39)-C(43)	**121(2)**
F(5)-C(40)-F(6)	**122(3)**
F(5)-C(40)-F(4)	**99(2)**
F(6)-C(40)-F(4)	**89(2)**
F(5)-C(40)-C(18)	**118(2)**
F(6)-C(40)-C(18)	**117(2)**
F(4)-C(40)-C(18)	**100(2)**
C(26)-C(41)-C(39)	**117(2)**

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 1.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Pd(1)	54(1)	64(1)	68(1)	-2(1)	-1(1)	-2(1)
Pd(2)	62(1)	57(1)	98(1)	-14(1)	-2(1)	11(1)