A New Use of Wittig-Type Reagents as 1,3-Dipolar Cycloaddition Precursors and in Pyrrole Synthesis

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Supporting Information

I. General Procedures

All reactions were performed in a Vacuum Atmospheres 553-2 dry box or under nitrogen using standard schlenk techniques. All reagents were purchased from Aldrich® and used as received, including $PhPCl_2$, catechol, DBU (1,8-diazabicyclo[5.4.0]undec-7-ene), LiHMDS (Lithium bis(trimethylsilyl)amide), dimethyl acetylenedicarboxylate, aldehydes, amines, and acid chlorides. Dichloromethane and acetonitrile were distilled from CaH_2 under nitrogen. Diethyl ether was distilled from Sodium benzophenone ketyl. Chloroform and deuterated chloroform were dried over 4Å molecular sieves. Imines[1] and dibenzoylacetylene[2] were prepared as per standard literature procedures. ^{1}H, ^{13}C, and ^{31}P NMR spectra were recorded on Varian Mercury 200, 300, 400 MHz, and Unity 500 MHz spectrometers. Reported J_{CP} and J_{CF} values were verified by obtaining ^{13}C NMR spectra at both 300 and 500 MHz. Mass spectra were obtained from the McGill University mass spectral facilities.

II. Synthesis

Synthesis of 2-phenyl-benzo[1,3,2]dioxaphosphole [PhP(catechol)]

A modified version of the literature procedure was followed.[3] A septum-sealed 500 mL schenk flask charged with catechol (11.01 g, 100 mmol) and placed under a nitrogen atmosphere. 100 mL of freshly distilled Et_2O was injected into the flask, followed by pyridine (16.2 mL, 200 mmol). The mixture was cooled to 0 °C and $PhPCl_2$ (13.6 mL, 100 mmol) was added dropwise over 15 min, stirred for 1 hr at this temperature, then warmed to ambient temperature for 2 hr. The pyridine hydrochloride was removed by filtration over a coarse (40-60 μM porosity) frit and washed with several portions of anhydrous Et_2O (N_2 degassed, non-distilled, total volume ca. 250 mL) directly into a 500 mL round bottom flask. An inverted funnel connected to a flow of nitrogen was used to cap the filtration apparatus in order to minimize exposure to air. The filtrate was concentrated *in vacuo* to yield an oil, which was distilled (84 – 88 °C, 115 mTorr) to yield 14.9 g (69%) of a clear colorless oil. This was stored in the glovebox over 4 Å molecular sieves. Alternatively, the product is also reasonably stable when stored in the freezer outside the glovebox. It was noted that with aging batches of PhP(catechol) can become quite malodorous. This does not however compromise its reactivity.

Synthesis of 2a'

In the glovebox, (4-$CH_3C_6H_4$)HC=N($CH_2C_6H_5$) (210 mg, 1.00 mmol) and *p*-methoxybenzoyl chloride (171 mg, 1.00 mmol) were mixed in minimal acetonitrile and allowed to stand for 20 min. Triphenylphosphine (263 mg, 1.00 mmol) was added and the mixture concentrated *in vacuo* to replace the solvent with THF (ca. 10 mL). Outside the glovebox, a 2.5 M solution of butylithium in hexanes (0.44 mL, 1.10 mmol) was added dropwise at -78 °C to the stirred suspension. The mixture was concentrated *in vacuo*, the solid was dissolved in dichloromethane, precipitated with diethyl ether and dried under high vacuum to yield the product as an orange solid (542 mg, 84% yield).

Synthesis of 2b

In the glovebox, (4-$CH_3C_6H_4$)HC=N($CH_2C_6H_5$) (210 mg, 1.00 mmol) and *p*-methoxybenzoyl chloride (171 mg, 1.00 mmol) were mixed in minimal acetonitrile and allowed to stand for 20 min. PhP(catechol) (216 mg, 1.00 mmol) was added followed by DBU (304 mg, 2.00 mmol), and the mixture diluted to ca. 5 mL with acetonitrile yielding precipitated product within a few minutes. Cooling in the freezer overnight improves yield, after which the precipitate is filtered, washed with acetonitrile, and dried under high vacuum to yield the pure product as a yellow solid (508 mg, 91% yield).

Typical procedure for the synthesis of pyrroles (Table 2)

In the glovebox, (4-$CH_3C_6H_4$)HC=N($CH_2C_6H_5$) (105 mg, 0.50 mmol) and *p*-methoxybenzoyl chloride (85 mg, 0.50 mmol) were mixed in minimal chloroform and allowed to stand for 30 min. PhP(catechol) (162 mg, 0.75 mmol), DBU (152 mg, 1 mmol), and dimethyl acetylenedicarboxylate (184 μL, 1.5 mmol) were added in order as solutions in chloroform, bringing the final volume to 1 mL. The reaction is complete once all additions have been made. The solution was concentrated *in vacuo* to give the crude product as an oily mixture which was purified by column chromatography using ethyl acetate / hexanes as eluent. The pyrrole products are generally fluorescent blue spots with RF ca. 0.2 on TLC with 5-30% ethyl acetate: hexanes as eluent.

Synthesis of alkyl-acid chloride derived pyrroles (Table 2, entry 7)

In the glovebox PhHC=N($CH_2C_6H_5$) (97.6 mg, 0.50 mmol) and isobutyryl chloride (53.3 mg, 0.50 mmol) 0.5 mL were mixed in minimal dichloromethane allowed to stand for 30 min. PhP(catechol) (108 mg, 0.5 mmol) was added. Outside the glovebox, the mixture was cooled to -78 °C under nitrogen, and LiHMDS (500 μL, 0.5 mmol, 1.0M solution in THF) was added dropwise. Methyl propiolate was injected (125 μL, 1.5 mmol), the cooling bath removed, and the mixture allowed to warm to rt over 0.5 h. The solution was concentrated *in vacuo* to give the crude product as an oily mixture which was purified by column chromatography using ethyl acetate / hexanes as eluent.

Synthesis of *C*-alkyl imine derived pyrroles (Table 2, entry 6)

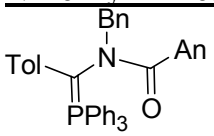
To $(CH_3)_2$CHHC=N($CH_2C_6H_5$) (80.5 mg, 0.50 mmol) in dichloromethane (0.5 mL) under nitrogen at -78 °C was added *p*-toluoyl chloride (66 μL, 0.5 mmol) via syringe. The -78 °C bath was then replaced for a -15 °C bath (ethylene glycol/ dry ice) and PhP(catechol) (89 μL, 0.55 mmol, d = 1.34 g/ mL) was injected, followed by AgOTf (128.5 mg, 0.5 mmol, acetonitrile solution). The mixture was warmed to room temperature, then recooled to – 78 °C and LiHMDS (500 μL, 0.5 mmol, 1.0M solution in THF) was added dropwise. Dimethyl acetylene dicarboxylate was injected (184 μL, 1.5 mmol), and the mixture allowed to warm to rt over 0.5 h. The solution was concentrated *in vacuo* to give the crude product as an oily mixture, which was purified by column chromatography using ethyl acetate / hexanes as eluent.

III. References

1. Layer, R. *Chem. Rev.* **1963**, 63, 489-510.
2. Zhang, J.-J.; Schuster, G. B. *J. Am. Chem. Soc.* **1989**, 111, 7149.
3. Previously reported syntheses of this compound did not include NMR and mass spectral data (a) Berlin, K. D.; Nagabhushanam, M. *J. Org. Chem.* **1964**, 29, 2056. (b) Wieber, M.; Hoos, W., R. *Tetrahedron Lett.* **1968**, 51, 5333. (c) Weiber, M.; Hoos, W. R. *Monatsh. Chem.* **1970**, 101, 776.

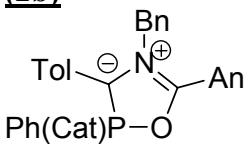
IV. Spectroscopic Data

N-Benzyl-4-methoxy-N-[p-tolyl-(triphenyl-l5-phosphanylidene)-methyl]-benzamide (2a)



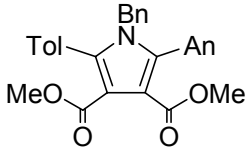
Isolated Yield: 84%. ^{1}H NMR (500 MHz, 25 °C, $CDCl_3$): δ (ppm) 7.46 (m, 6H), 7.60-6.95 (broad m, 10H), 7.16-7.08 (m, 4H), 7.05-6.98 (m, 2H), 6.84-6.76 (m, 2H), 6.72-6.58 (m, 4H), 5.15 (d, J = 13.0 Hz, 1H), 4.54 (d, J = 13.5 Hz, 1H), 3.77 (s, 3H), 2.22 (s, 3H). ^{13}C NMR (125 MHz, - 19 °C, $CDCl_3$): δ (ppm) 175.7, 160.0, 142.3, 142.1, 138.5, 134.1 (broad), 134.0 (broad), 133.8 (broad), 133.7 (broad), 133.6, 133.4_4 (broad), 133.3_7 (broad), 131.9 (broad), 131.6, 131.4 (broad), 130.9, 130.2, 128.8-128.2 (m, broad), 128.5, 128.0 (broad), 127.9 (broad), 127.4, 126.9, 126.6, 125.8, 124.3, 123.9, 123.1, 119.1, 111.8, 77.2, 56.8, 56.5, 55.6, 55.1, 20.5. ^{31}P NMR (81.0 MHz, $CDCl_3$): δ (ppm) 10.2. HRMS (ESI^+) for $C_{41}H_{37}NO_2P^+$; calculated: 606.25564, found: 606.25627 (error m/z = 1.0 ppm).

4′-benzyl-5′-(4-methoxyphenyl)-3′-(4-methylphenyl)-2-phenyl-2′,3′-dihydro-2λ^5-spiro[[1,3,2]-benzodioxaphosphole-2,2′-[1,4 λ^5,2]oxazaphosphol-4′-ylium-3′-ide] (2b)



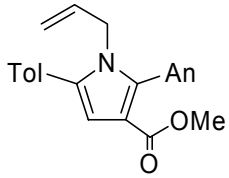
Isolated Yield: 91%. ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 7.68 (dd, J = 16.5, 7.0 Hz, 2H), 7.45 (d, J = 8.5 Hz, 2H), 7.40-7.20 (m, 4H), 7.05 (d, J = 7.5 Hz, 3H), 7.02-6.97 (m, 2H), 6.92 (d, J = 8.0 Hz, 2H), 6.85 (d, J = 9.0 Hz, 2H), 6.73 (dd, J = 5.5, 3.0), 6.46 (broad s, 1H), 4.99 (broad dd, 2H), 3.79 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 160.9, 149.6 ($O\underline{C}C_6H_5OMe$, based on 2D NMR analysis), 148.0 , 145.7, 142.9 (d, $^{1}J_{C\text{-}P}$ = 226.7 Hz), 135.7, 135.3 (d, $^{5}J_{C\text{-}P}$ = 1.9 Hz), 135.1 (d, $^{3}J_{C\text{-}P}$ = 6.2 Hz) 132.2 (d, $^{2}J_{C\text{-}P}$ = 17.6 Hz), 129.5, 128.5, 128.5 (d, $^{4}J_{C\text{-}P}$ = 3.8 Hz), 128.0 (d, $^{2\text{ or }3}J_{C\text{-}P}$ = 11.4 Hz), 127.9, 127.8 (d, $^{2\text{ or }3}J_{C\text{-}P}$ = 12.3 Hz), 127.5, 126.1, 121.6, 119.2, 118.9, 114.1, 110.7, 73.0 (d, $^{1}J_{C\text{-}P}$ = 261.6 Hz, P=$\underline{C}$), 55.3, 50.9 (d, $^{3}J_{C\text{-}P}$ = 8.4 Hz), 21.1. ^{31}P NMR (81.0 MHz, $CDCl_3$): δ (ppm) – 16.9. HRMS (ESI^+) for $C_{35}H_{31}NO_4P^+$; calculated: 560.19852, found: 560.19895 (error m/z = 0.8 ppm).

1-Benzyl-2-(4-methoxy-phenyl)-5-p-tolyl-1H-pyrrole-3,4-dicarboxylic acid dimethyl ester.



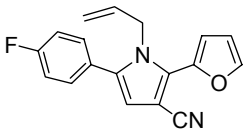
Isolated Yield: 85%. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.24-7.02 (m, 9H), 6.88-6.76 (m, 2H), 6.63-6.51 (m, 2H), 4.89 (s, 2H), 3.79 (s, 3H), 3.67 (s, 6H), 2.33 (s, 3H). ^{13}C NMR (75.5 MHz, $CDCl_3$): δ (ppm) 165.6_9, 165.6_5, 159.9, 138.7, 137.6, 137.3, 137.2, 132.1, 130.6, 129.1, 128.5, 127.8, 127.4, 126.3, 122.9, 122.9, 114.8_1, 114.8_0, 113.8, 55.5, 52.0, 48.7, 21.8. HRMS (ESI^+) for $C_{29}H_{28}NO_5^+$; calculated: 470.19620, found: 470.19541 (error m/z = 1.7 ppm).

1-Allyl-5-(4-methoxy-phenyl)-2-p-tolyl-1H-pyrrole-3-carboxylic acid methyl ester



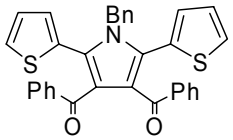
Isolated Yield: 89%. ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 7.38-7.26 (m, 4H), 7.21 (d, *J* = 7.0 Hz, 2H), 6.95 (d, *J* = 8.0 Hz, 2H), 6.69 (s, 1H), 5.65-5.55 (m, 1H), 5.01 (d, *J* = 10.0, 1H), 4.65 (d, *J* = 17.5 Hz, 1H), 4.39 (s, 2H), 3.85 (s, 3H), 3.68 (s, 3H), 2.39 (s, 3H). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ (ppm) 165.5, 159.9, 139.8, 137.8, 134.8, 134.7, 132.2, 130.1, 129.4$_5$, 129.3$_6$, 124.4, 116.7, 113.6, 113.4, 110.3, 55.4, 51.0, 47.2, 21.5. HRMS (ESI$^+$) for $C_{23}H_{24}NO_3^+$; calculated: 362.17507, found: 362.17471 (error m/z = 1.0 ppm).

1-Allyl-5-(4-fluoro-phenyl)-2-furan-2-yl-1H-pyrrole-3-carbonitrile



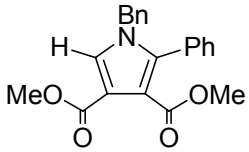
Isolated Yield: 74%. ^{1}H NMR (300 MHz, $CDCl_3$): δ (ppm) 7.53 (s, 1H), 7.42-7.31 (m, 2H), 7.16-7.04 (m, 2H), 6.87 (d, *J* = 3.4 Hz, 1H), 6.54-6.50 (m, ^{1}H), 6.48 (s, 1H), 5.92-5.76 (m, 1H), 5.14 (d, *J* = 10.6 Hz, 1H), 4,79 (d, *J* = 17.3 Hz, 1H), 4.72-4.63 (m, 2H). ^{13}C NMR (75.5 MHz, $CDCl_3$): δ (ppm) 164.7, 161.4, 143.8, 143.0, 136.0, 133.8, 131.6, 131.5, 127.3$_0$, 127.2$_6$, 117.1, 117.0, 116.1, 115.8, 112.5, 111.9, 111.1, 92.4, 48.8.

(4-Benzoyl-1-benzyl-2,5-di-thiophen-2-yl-1H-pyrrol-3-yl)-phenyl-methanone



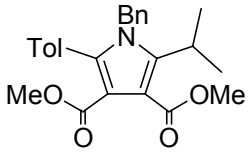
Isolated Yield: 60%. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.54 (d, *J* = 7.6 Hz, 2H), 7.40-7.10 (m, 11H), 7.05-6.95 (m, 2H), 6.93-6.80 (m, 4H), 5.26 (s, 2H). ^{13}C NMR (75.5 MHz, $CDCl_3$): δ (ppm) 192.0, 139.2, 137.8, 136.1, 132.3, 131.4, 130.1, 130.0, 129.2, 128.9, 128.5, 128.1, 127.7, 127.1, 126.1, 49.1. HRMS (ESI$^+$) for $C_{32}H_{20}NO_7^+$; calculated: 530.12343, found: 530.12378 (error m/z = 0.7 ppm).

1-Benzyl-2-phenyl-1H-pyrrole-3,4-dicarboxylic acid dimethyl ester



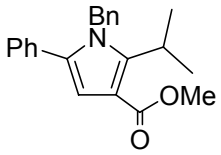
Isolated Yield: 44%. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.42-7.33 (m, 3H), 7.32-7.23 (m, 6H), 6.98-6.92 (m, 2H), 4.93 (m, 2H), 3.81 (m, 3H), 3.67 (m, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 165.5, 164.1, 137.3, 136.3, 130.4, 130.0, 128.8, 128.3, 128.0, 127.0, 126.9, 115.5, 115.0, 51.7, 51.5, 51.2. HRMS (ESI$^+$) for $C_{21}H_{20}NO_4^+$; calculated: 350.13869, found: 350.13827 (error m/z = 1.2 ppm).

1-Benzyl-2-isopropyl-5-p-tolyl-1H-pyrrole-3,4-dicarboxylic acid dimethyl ester



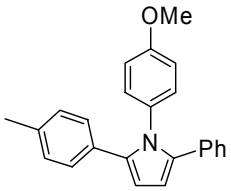
Isolated Yield: 83%. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.32-7.20 (m, 3H), 7.18-7.07 (m, 4H), 6.86 (d, J = 7.2 Hz, 2H), 4.97 (s, 2H), 3.86 (s, 3H), 3.62 (s, 3H), 3.02-2.90 (m, 1H), 2.32 (s, 3H), 1.20 (d, J = 7.2 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 167.6, 165.3, 140.6, 138.7, 137.7, 136.9, 130.6, 129.1, 129.0, 128.1, 127.7, 125.7, 113.8, 113.4, 52.1, 51.6, 48.0, 26.7, 21.5_9, 21.5_5. HRMS (ESI^+) for $C_{25}H_{28}NO_4^+$; calculated: 406.20129, found: 406.20093 (error m/z = 0.9 ppm).

1-Benzyl-2-isopropyl-5-phenyl-1H-pyrrole-3-carboxylic acid methyl ester



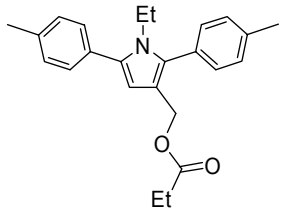
Isolated Yield: 67%. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.35-7.22 (m, 8H), 6.93 (d, J = 7.2 Hz, 2H), 5.19 (s, 2H), 3.83 (s, 3H), 3.25-3.13 (m, 1H), 1.29 (d, J = 7.2 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) 165.3, 146.2, 138.4, 133.6, 132.7, 129.3, 128.8, 128.4, 127.6, 127.3, 125.5, 111.6, 111.4, 50.9, 48.3, 26.7, 20.1. HRMS (ESI^+) for $C_{22}H_{24}NO_2^+$; calculated: 334.18016, found: 334.17984 (error m/z = 1.0 ppm).

1-(4-Methoxy-phenyl)-2-phenyl-5-p-tolyl-1H-pyrrole



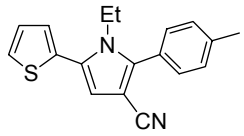
Isolated Yield: 42%. ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 7.22-7.07 (m, 5H), 7.04-6.93 (m, 6H), 6.77 (d, J = 9.0 Hz, 2H), 6.47 (s, 1H), 6.45 (s, 1H), 3.79 (s, 3H), 2.30 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 158.4, 136.0, 135.8, 135.6, 133.7, 132.0, 130.4, 129.8, 128.6_2, 128.5_6, 127.8, 126.0, 113.9, 109.5, 109.3, 55.3, 21.0. HRMS (ESI^+) for $C_{24}H_{22}NO^+$; calculated: 340.16959, found: 340.16952 (error m/z = 0.2 ppm). N.B.: rapid H → D exchange in $CDCl_3$ yielded extra carbon and Mass Spec. signals: ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 135.9, 135.5, 109.4, 109.2. HRMS (ESI^+) for $C_{24}H_{21}DNO^+$; calculated: 341.17587, found: 341.17587 (error m/z = 0.0 ppm).

1-Ethyl-2,5-di-p-tolyl-1H-pyrrole



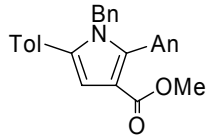
Isolated Yield: 24%. ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 7.36 (d, J = 8.0 Hz, 2H), 7.32-7.00 (m, 6H), 6.30 (s, 1H), 4.92 (s, 3H), 3.97 (q, J = 7.5 Hz, 2H), 2.42 (s, 3H), 2.41 (s, 3H), 2.34 (q, J = 7.5 Hz, 2H), 1.14 (t, J = 7.5 Hz, 3H), 0.87 (t, J = 7.0 Hz). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 174.7, 137.6, 136.8, 134.4, 130.7_6, 130.7_4, 130.5, 129.1, 129.0, 116.3, 116.2, 109.8, 60.0_8, 60.0_6, 39.7, 27.7, 21.2_4, 21.1_7, 16.2, 9.1. HRMS (ESI^+) for $C_{24}H_{27}NO_2Na^+$; calculated: 384.19340, found: 384.19341 (error m/z = 0.1 ppm).

1-Ethyl-5-thiophen-2-yl-2-p-tolyl-1H-pyrrole-3-carbonitrile



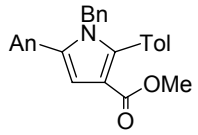
Isolated Yield: 69%. ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 7.42-7.34 (m, 3H), 7.30 (d, J = 7.5 Hz, 2H), 7.15-7.09 (m, 2H), 6.58 (s, 1H), 4.05 (q, J = 7.5 Hz, 2H), 2.42 (s, 3H), 1.03 (t, J = 7.4 Hz). ^{13}C NMR (125 MHz, $CDCl_3$): δ (ppm) 142.5, 139.3, 132.4, 129.6, 129.5, 127.5, 127.4, 127.3, 126.7, 126.5, 117.0, 113.3, 92.8, 40.4, 21.3, 16.2. HRMS (ESI^+) for $C_{18}H_{17}N_2S^+$; calculated: 293.11070, found: 293.11030 (error m/z = 1.3 ppm).

1-Benzyl-2-(4-methoxy-phenyl)-5-p-tolyl-1H-pyrrole-3-carboxylic acid methyl ester



Isolated Yield: 88%. ^{1}H NMR (500 MHz, $CDCl_3$): δ (ppm) 7.26 (d, J = 6.5 Hz, 2H), 7.21 (d, J = 8.0 Hz, 2H), 7.19-7.11 (m, 5H), 6.88 (d, J = 6.5 Hz, 2H), 6.79 (s, 1H), 6.69-6.65 (m, 2H), 5.06 (s, 2H), 3.81 (s, 3H), 3.71 (s, 3H), 2.36 (s, 3H). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ (ppm) 165.5, 159.8, 140.2, 138.6, 137.8, 135.3, 132.1, 130.0, 129.4_5, 129.4_2, 128.6, 127.3, 126.2, 124.3, 113.8, 113.6, 110.7, 55.4, 51.1, 48.6, 21.5. HRMS (ESI^+) for $C_{27}H_{26}NO_3^+$; calculated: 412.19072, found: 412.19040 (error m/z = 0.8 ppm).

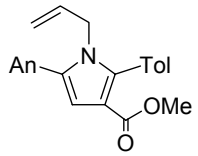
1-Benzyl-5-(4-methoxy-phenyl)-2-p-tolyl-1H-pyrrole-3-carboxylic acid methyl ester



Isolated Yield: 91%. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.24 (d, J = 8.4 Hz, 2H), 7.20-7.10 (m, 7H), 6.86 (d, J = 7.6 Hz, 2H), 6.74 (s, 1H), 6.69-6.61 (m, 2H), 5.02 (s, 2H), 3.80 (s, 3H), 3.70 (s, 3H), 2.36 (s, 3H). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ (ppm) 165.5, 159.5, 140.2, 138.6, 138.3, 135.0, 130.9, 130.7, 129.1, 128.9, 128.5, 127.2, 126.1, 125.3, 114.1, 113.6, 110.5, 55.5,

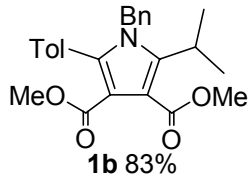
51.1, 48.6, 21.6. HRMS (ESI^+) for $C_{27}H_{26}NO_3^+$; calculated: 412.19072, found: 412.19030 (error m/z = 1.0 ppm).

1-Allyl-2-(4-methoxy-phenyl)-5-p-tolyl-1H-pyrrole-3-carboxylic acid methyl ester



Isolated Yield: 90%. ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.38 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 7.6 Hz, 2H), 6.95 (d, *J* = 8.4 Hz, 2H), 6.69 (s, 1H), 5.68-5.55 (m, 1H), 5.03 (d, *J* = 10.4 Hz, 1H), 4.67 (d, *J* = 17.2 Hz, 1H), 4.39 (s, 2H), 3.85 (s, 3H), 3.69 (s, 3H), 2.42 (s, 3H). ^{13}C NMR (100.6 MHz, $CDCl_3$): δ (ppm) 165.2, 159.3, 139.6, 138.2, 134.4_4, 134.3_8, 130.7, 130.5, 129.1, 128.7, 125.1, 116.4, 113.8, 113.0, 109.9, 55.3, 50.8, 46.9, 21.5. HRMS (ESI^+) for $C_{23}H_{24}NO_3^+$; calculated: 362.17507, found: 362.17487 (error m/z = 0.6 ppm).

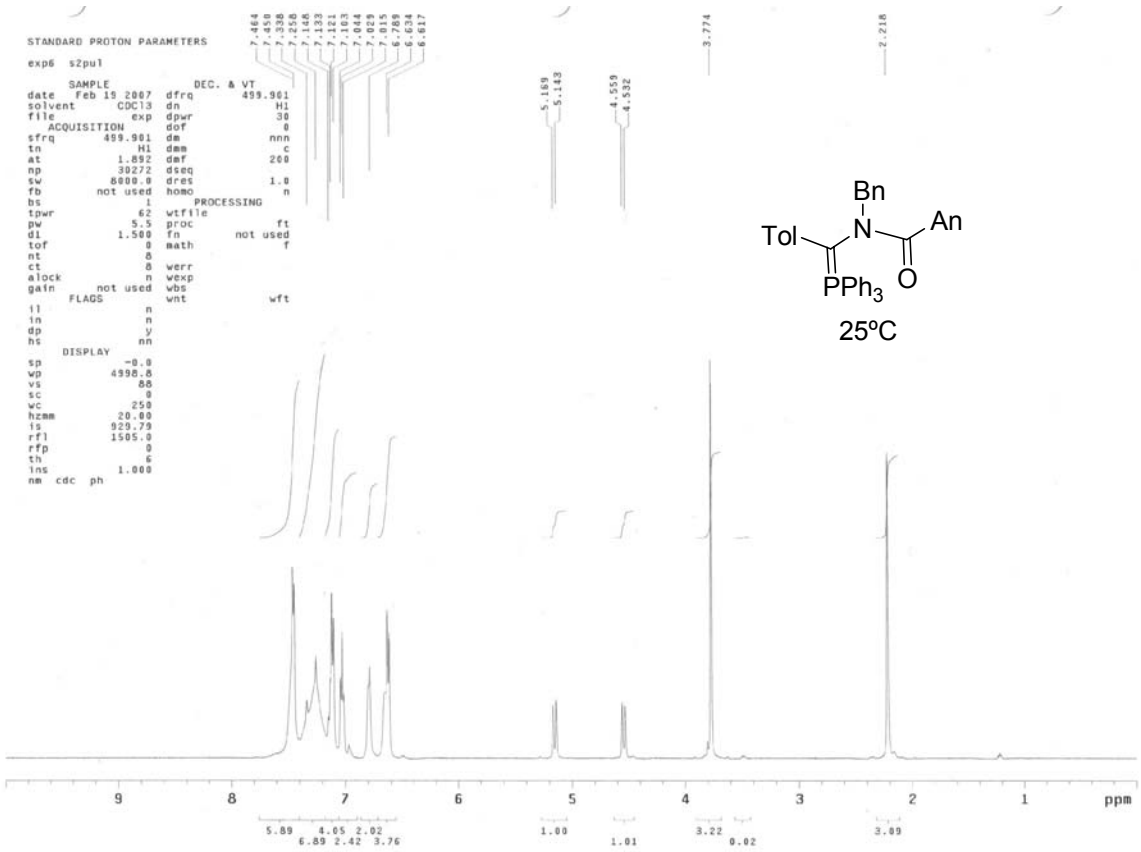
1-Benzyl-2-isopropyl-5-p-tolyl-1H-pyrrole-3,4-dicarboxylic acid dimethyl ester

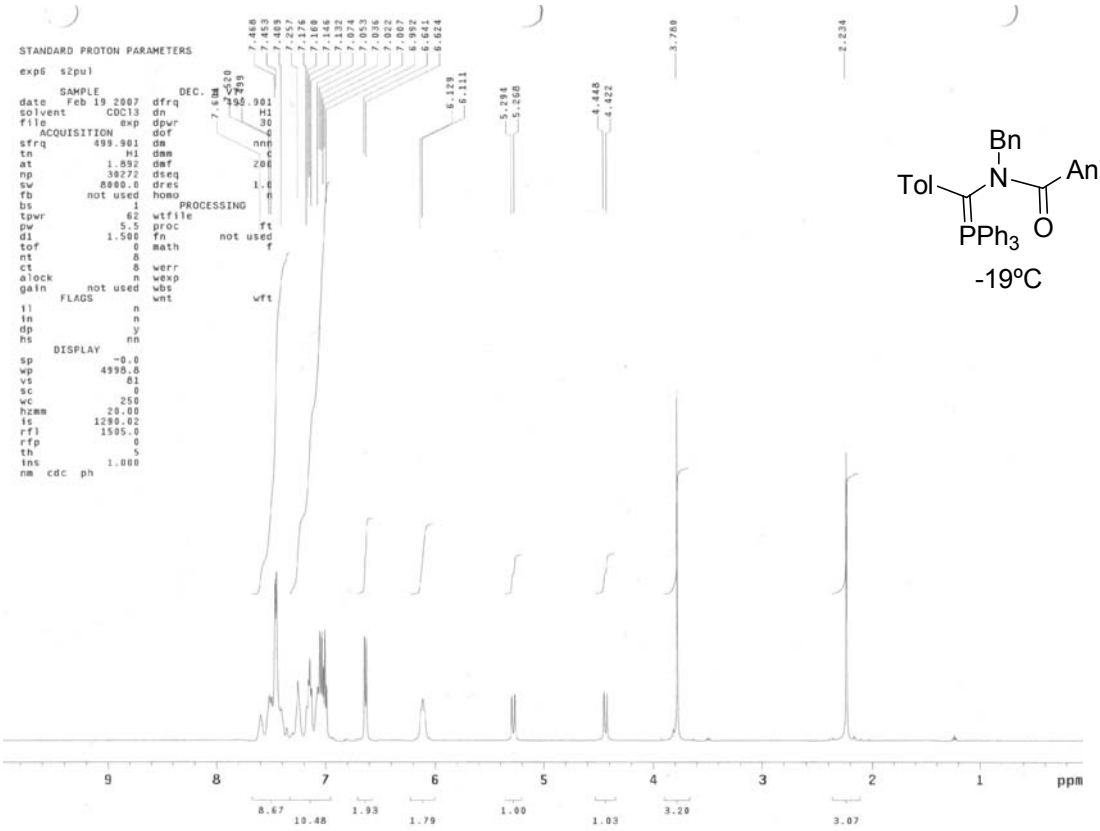


1b 83%

Isolated Yield: 83%. ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) 7.32-7.20 (m, 3H), 7.18-7.07 (m, 4H), 6.86 (d, *J* = 7.2 Hz, 2H), 4.97 (s, 2H), 3.86 (s, 3H), 3.62 (s, 3H), 3.02-2.90 (m, 1H), 2.32 (s, 3H), 1.20 (d, *J* = 7.2 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ (ppm) 167.6, 165.3, 140.6, 138.7, 137.7, 136.9, 130.6, 129.1, 129.0, 128.1, 127.7, 125.7, 113.8, 113.4, 52.1, 51.6, 48.0, 26.7, 21.5_9, 21.5_5. HRMS (ESI^+) for $C_{25}H_{28}NO_4^+$; calculated: 406.20129, found: 406.20093 (error m/z = 0.9 ppm).

V. ^{1}H and ^{13}C NMR Spectra

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date Feb 19 2007
solvent CDCl3
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sw 8000.8
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tpwr 62
pw 5.5
d1 1.500
tof 0
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
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vs 81
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wc 250
hzmm 20.00
is 1290.02
rfl 1505.0
rfp 0
th 5
ins 1.000
nm cdc ph
DEC.
dfrq 499.901
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.6
homo n
PROCESSING
wtfile
proc ft
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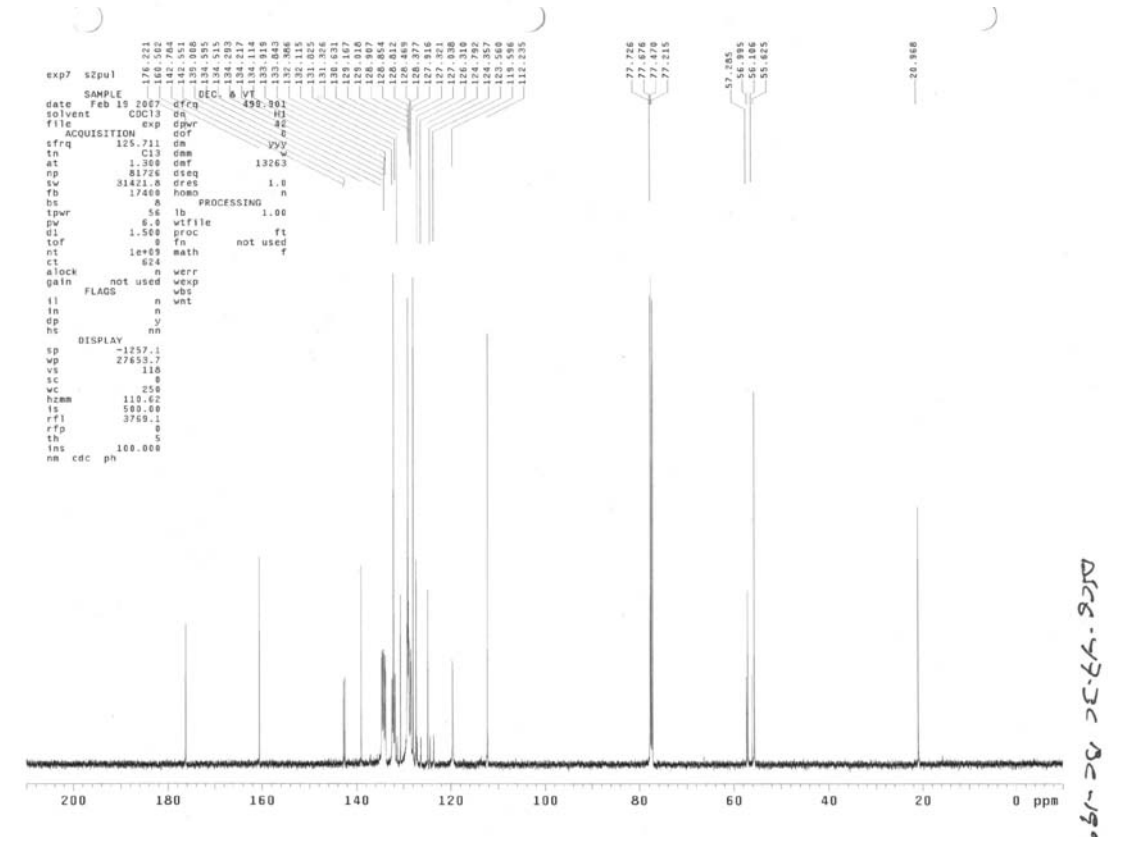
math f
werr
wexp
wbs
wnt wft
Bn
N
An
Tol
PPh3
O
-19ºC
ppm


exp7 s2pul
SAMPLE
date Feb 19 2007
solvent CDCl3
file exp
ACQUISITION
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tn C13
at 1.308
np 81726
sw 31421.8
fb 17460
bs 8
tpwr 56
pw 6.0
d1 1.500
tof 0
nt 1e+09
ct 624
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1257.1
wp 27653.7
vs 118
sc 8
wc 250
hzmm 110.62
is 500.00
rfl 3769.1
rfp 0
th 5
ins 100.000
nm cdc ph
DEC. @ f1
dfrq 499.901
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 13263
dseq
dres 1.8
homo n
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wtfile
proc ft
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math f
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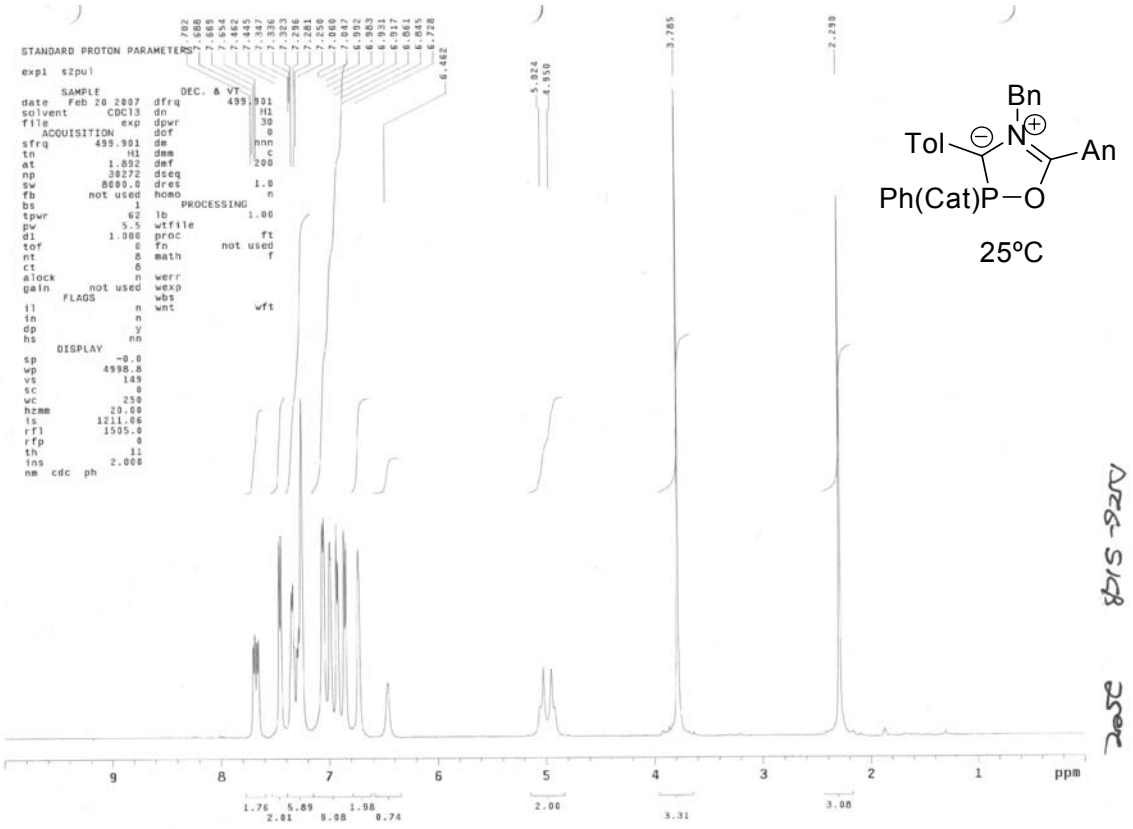
wexp
wbs
wnt
ppm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Tol
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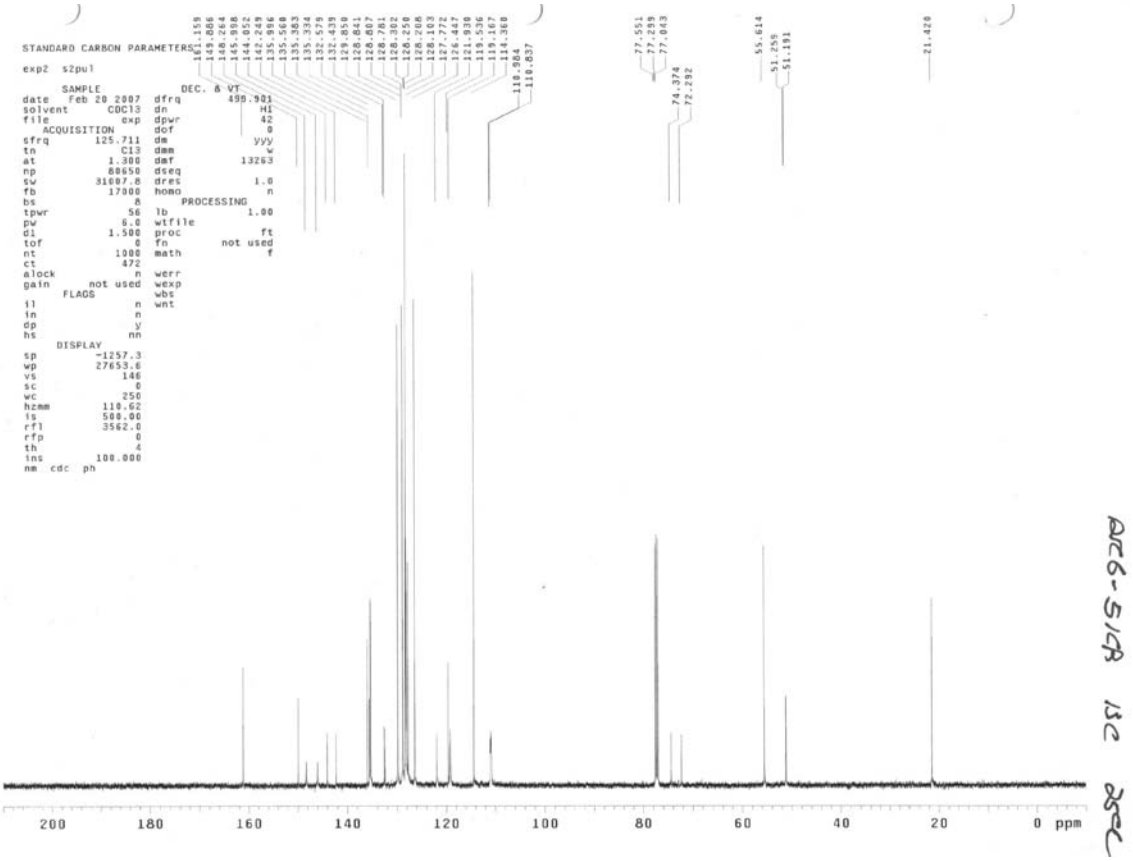
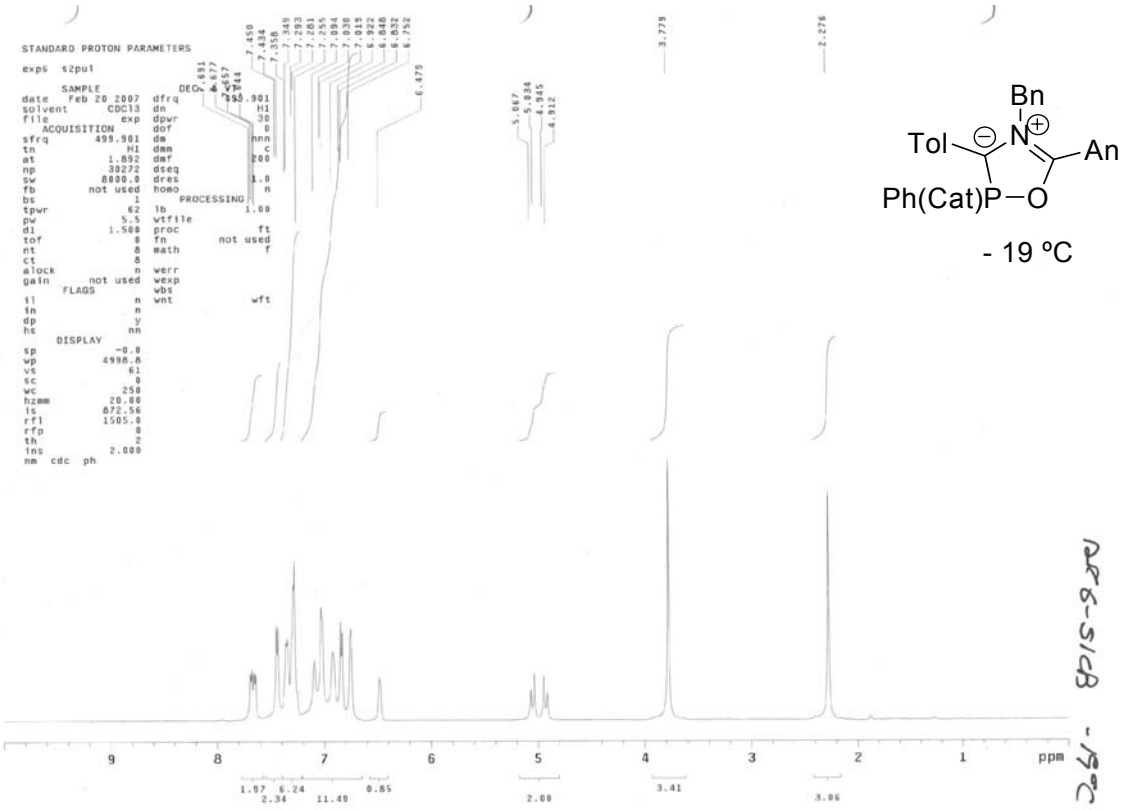
An
Ph(Cat)P
O
25ºC
ppm


STANDARD CARBON PARAMETERS
ppm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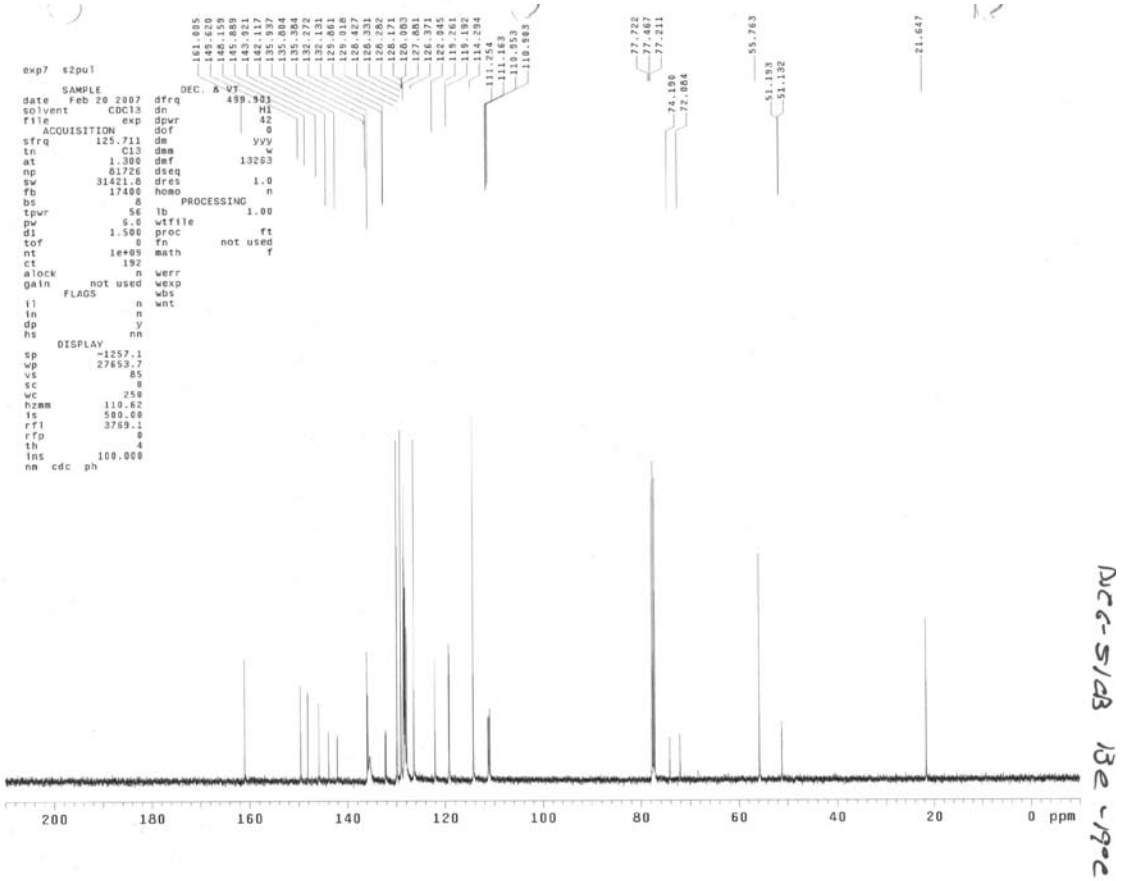
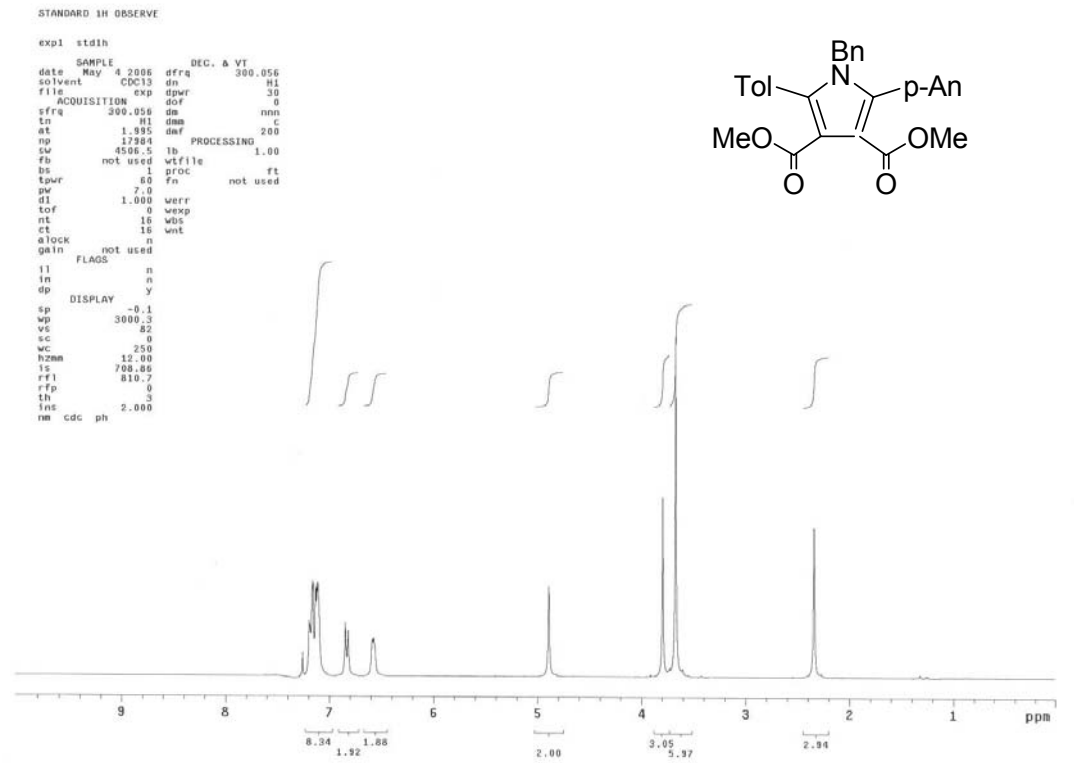
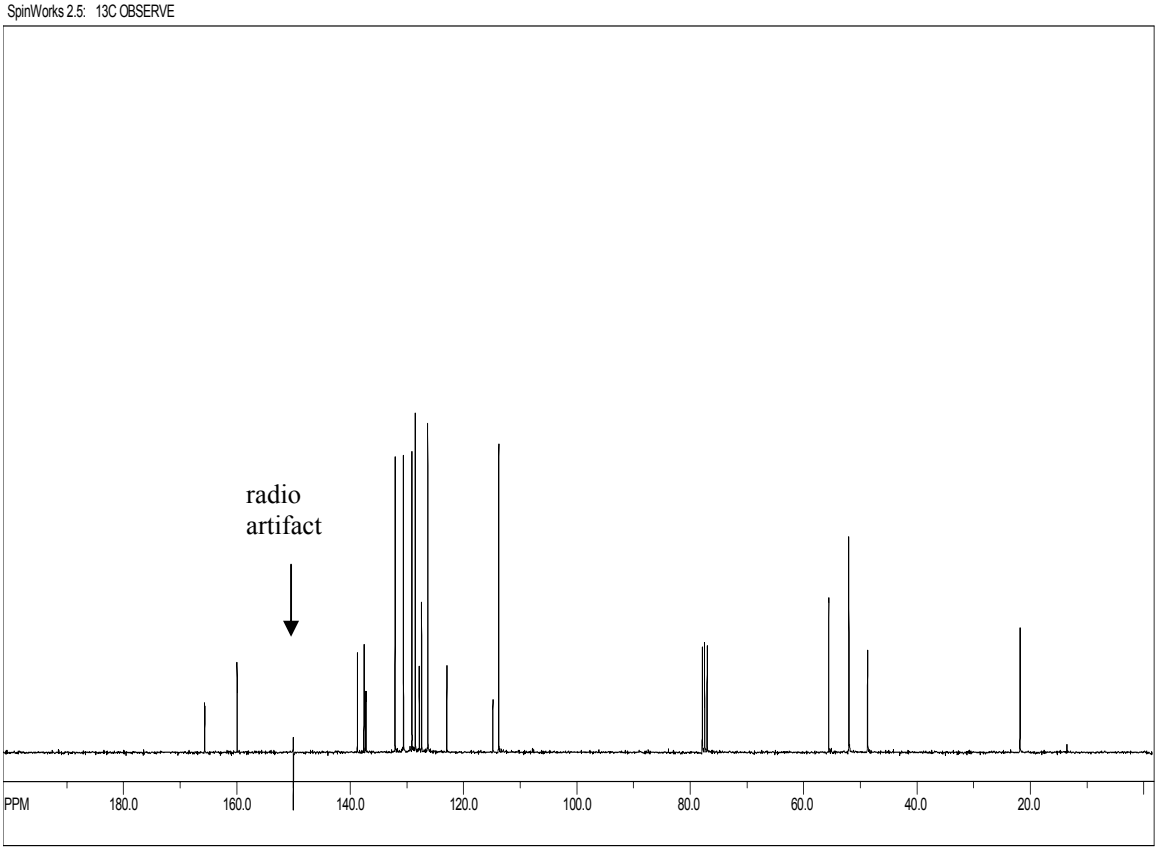
Bn
Tol
N
An
Ph(Cat)P
O
- 19 ºC
ppm


ppm



SpinWorks 2.5: 13C OBSERVE



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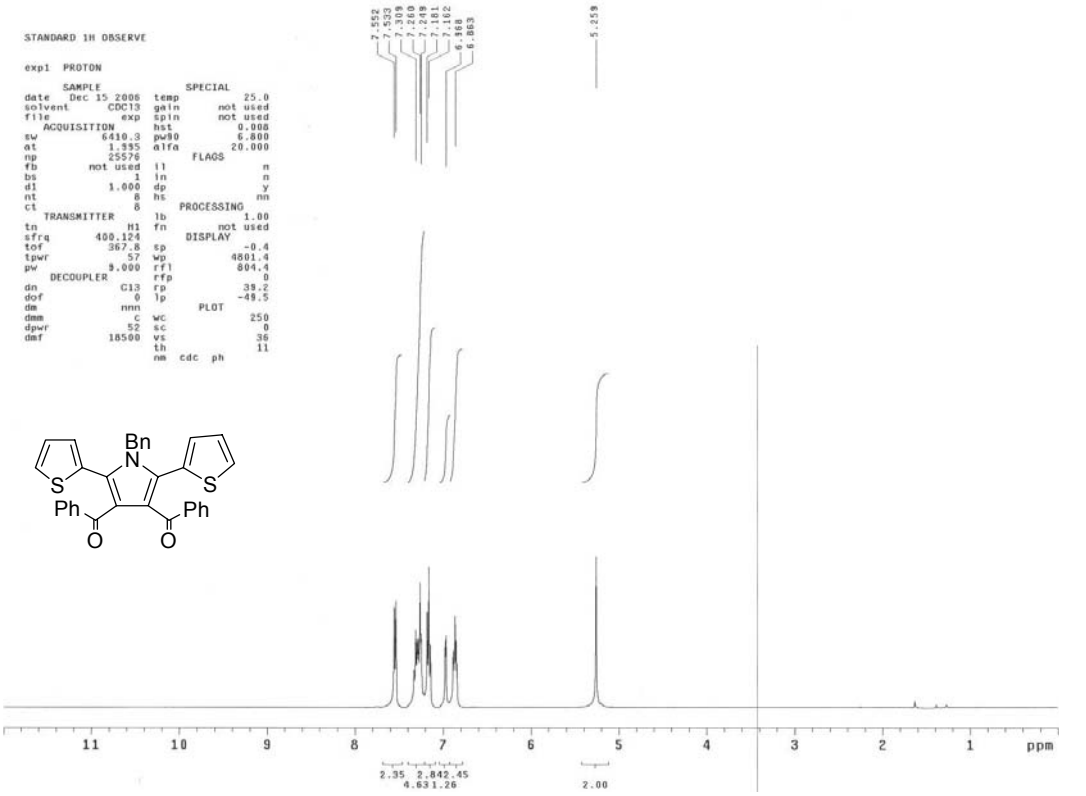
time domain size: 68492 points
width: 18867.92 Hz = 250.044338 ppm = 0.275476 Hz/pt
number of scans: 688

freq. of 0 ppm: 75.450016 MHz
processed size: 131072 complex points
LB: 0.000 GB: 0.0000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date Dec 15 2006
solvent CDCl3
file exp
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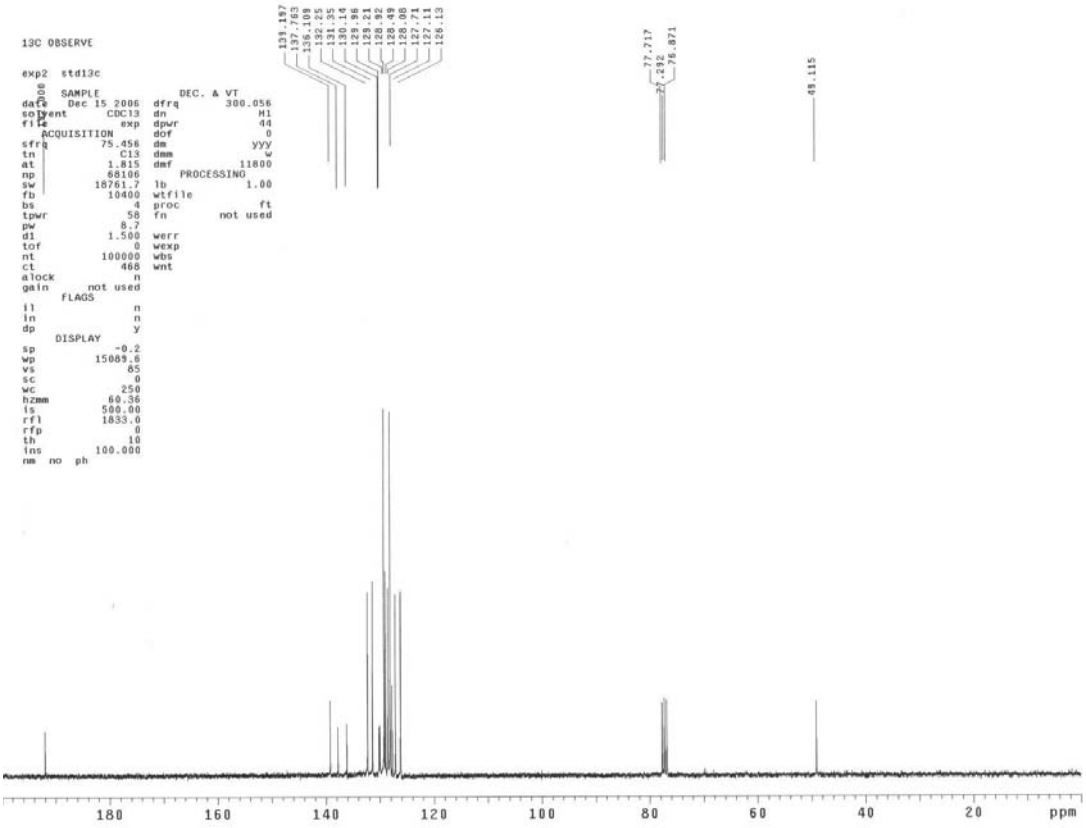
np 25576
fb not used
bs 1
d1 1.000
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 400.124
tof 367.8
tpwr 57
pw 9.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 52
dmf 18500
SPECIAL
temp 25.0
gain not used
spin not used
hst 0.008
pw90 6.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -0.4
wp 4801.4
rfl 804.4
rfp 0
rp 39.2
lp -49.5
PLOT
wc 250
sc 0
vs 36
th 11
nm cdc ph
7.552
7.533
7.518
7.260
7.249
7.181
7.162
6.868
6.863
5.259
Bn
N
S
S
Ph
Ph
O
O
11
10
9
8
7
6
5
4
3
2
1
ppm
2.35
2.84
2.45
4.63
1.26
2.00


13C OBSERVE
exp2 std13c
SAMPLE
date Dec 15 2006
solvent CDCl3
file exp
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tn C13
at 1.815
np 68106
sw 18761.7
fb 10400
bs 4
tpwr 58
pw 8.7
d1 1.500
tof 0
nt 100000
ct 468
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -0.2
wp 15089.6
vs 85
sc 0
wc 250
hzmm 60.36
is 500.00
rfl 1833.0
rfp 0
th 10
ins 100.000
nm no ph
DEC. & VT
dfrq 300.056
dn H1
dpwr 44
dof 0
dm yyy
dmm w
dmf 11800
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
131.197
137.763
136.109
132.25
131.35
130.54
129.96
129.21
128.92
128.49
128.08
127.71
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77.292
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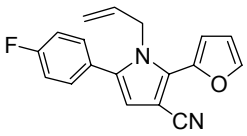
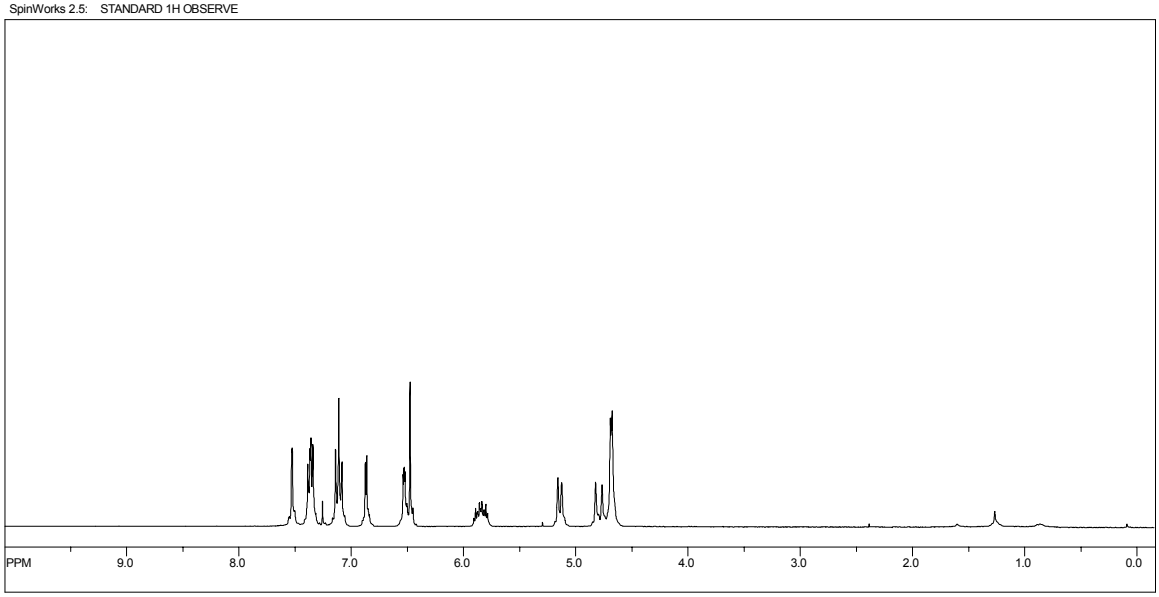
180
160
140
120
100
80
60
40
20
ppm



SpinWorks 2.5: STANDARD 1H OBSERVE



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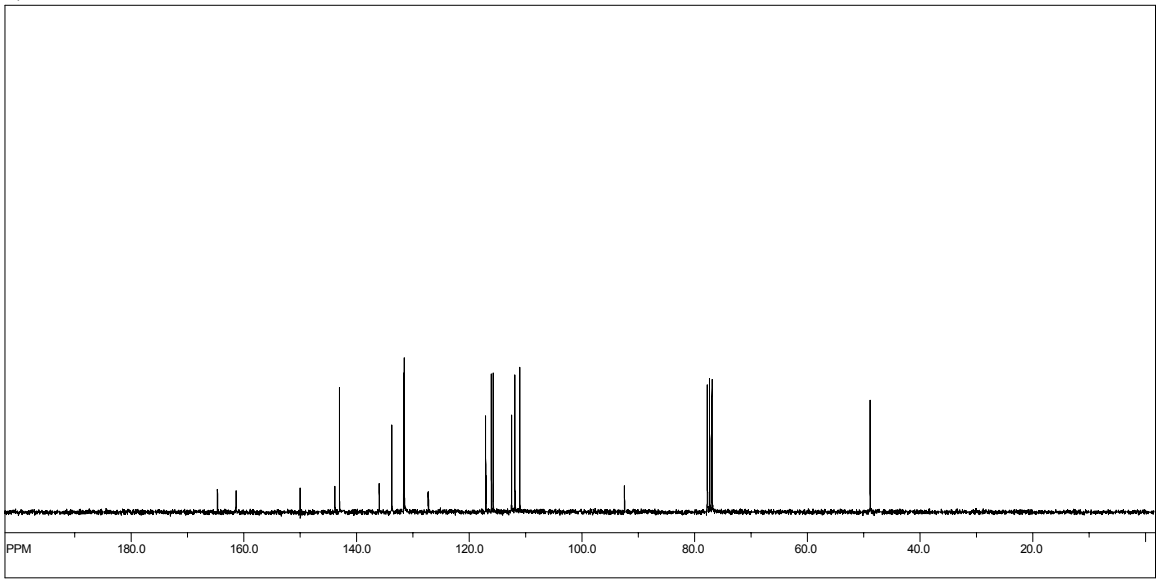
number of scans: 4

freq. of 0 ppm: 300.059550 MHz
processed size: 32768 complex points
LB: 0.000 GB: 0.0000

SpinWorks 2.5: 13C OBSERVE



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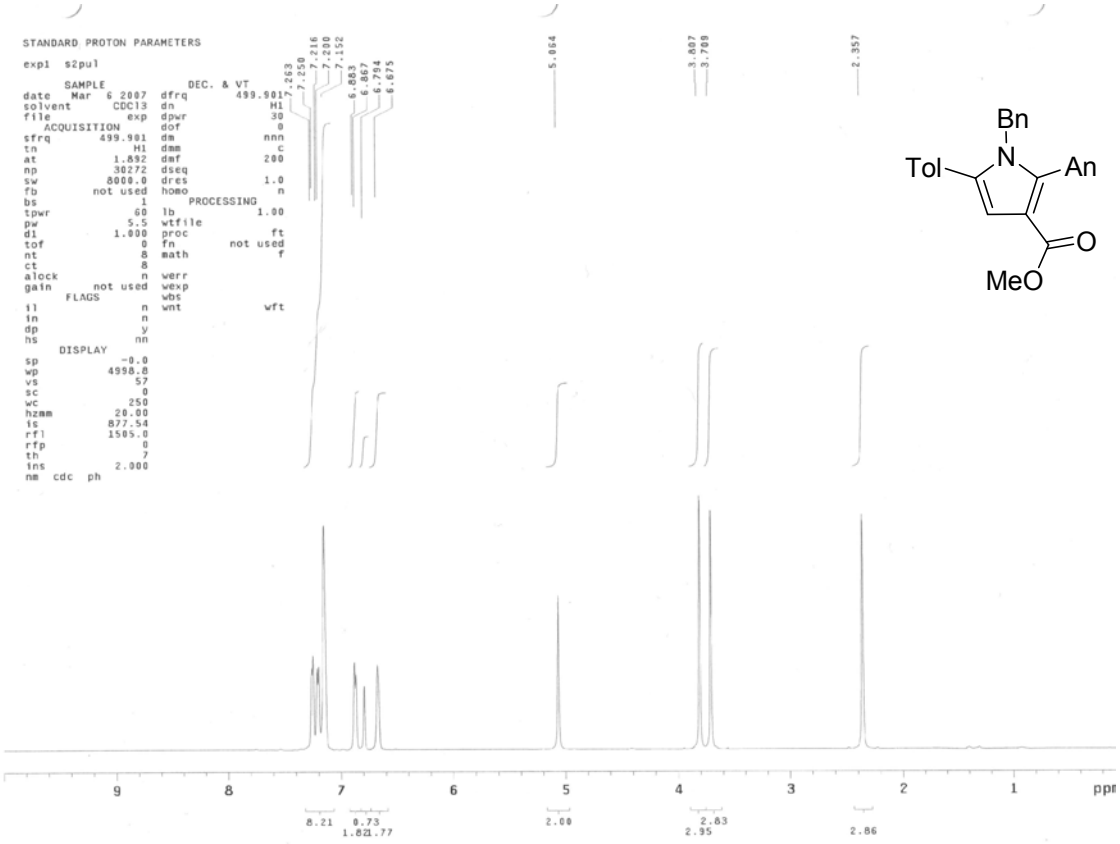
transmitter freq.: 75.458315 MHz
time domain size: 68492 points
width: 18867.92 Hz = 250.044338 ppm = 0.275476 Hz/pt
number of scans: 1352

freq. of 0 ppm: 75.450016 MHz
processed size: 131072 complex points
LB: 1.000 GB: 0.0000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exp1 s2pul
SAMPLE
date Mar 6 2007
solvent CDCl3
file exp
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tn H1
at 1.892
np 30272
sw 8000.0
fb not used
bs 1
tpwr 60
pw 5.5
d1 1.000
tof 0
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -0.8
wp 4998.8
vs 57
sc 0
wc 250
hzmm 20.00
is 877.54
rfl 1505.0
rfp 0
th 7
ins 2.000
nm cdc ph
DEC. & VT
dfrq 499.901
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
7.263
7.250
7.216
7.180
7.152
6.883
6.867
6.794
6.675
5.064
3.807
3.788
2.357
Bn
N
Tol
An
O
MeO
9
8
7
6
5
4
3
2
1
ppm
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1.77
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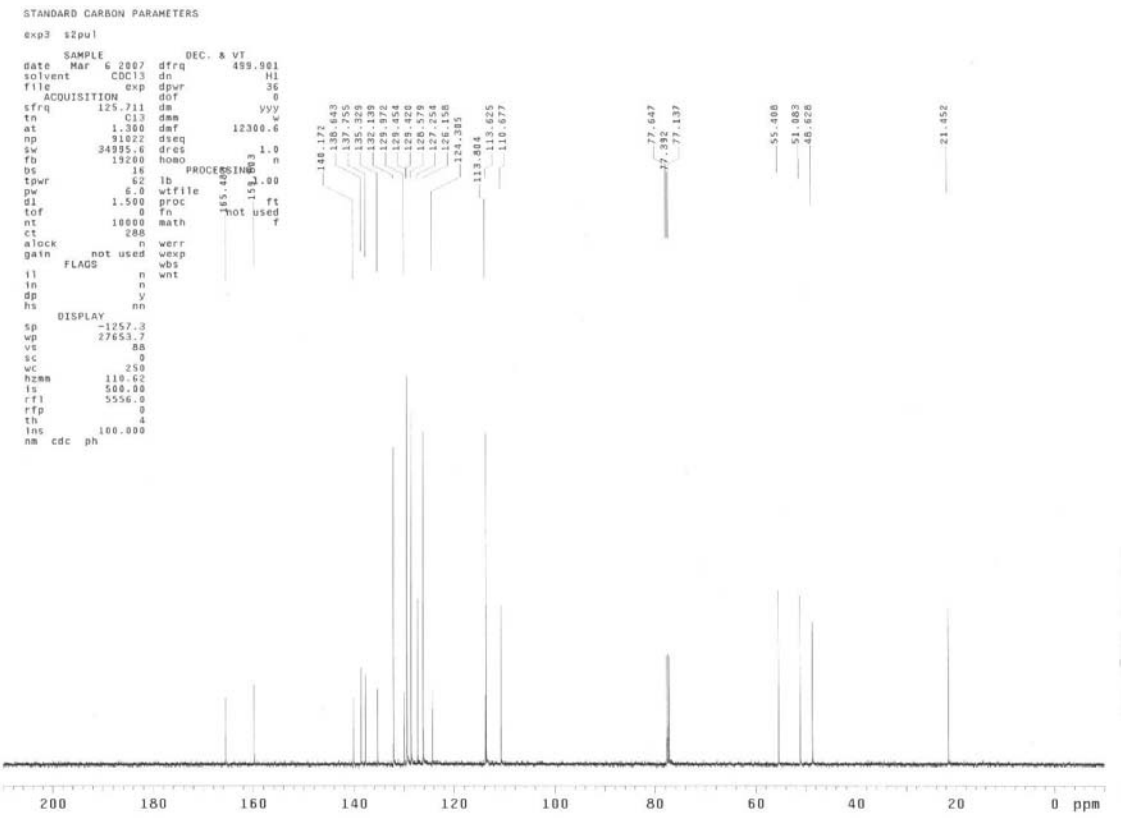
2.95
2.86


STANDARD CARBON PARAMETERS
exp3 s2pul
SAMPLE
date Mar 6 2007
solvent CDCl3
file exp
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tn C13
at 1.300
np 91022
sw 34995.6
fb 19200
bs 16
tpwr 62
pw 6.0
d1 1.500
tof 0
nt 10000
ct 288
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1257.3
wp 27653.7
vs 88
sc 0
wc 250
hzmm 110.62
is 500.00
rfl 5556.0
rfp 0
th 4
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.901
dn H1
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dof 0
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dmm c
dmf 12300.6
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
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116.158
124.385
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77.647
77.392
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51.083
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60
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20
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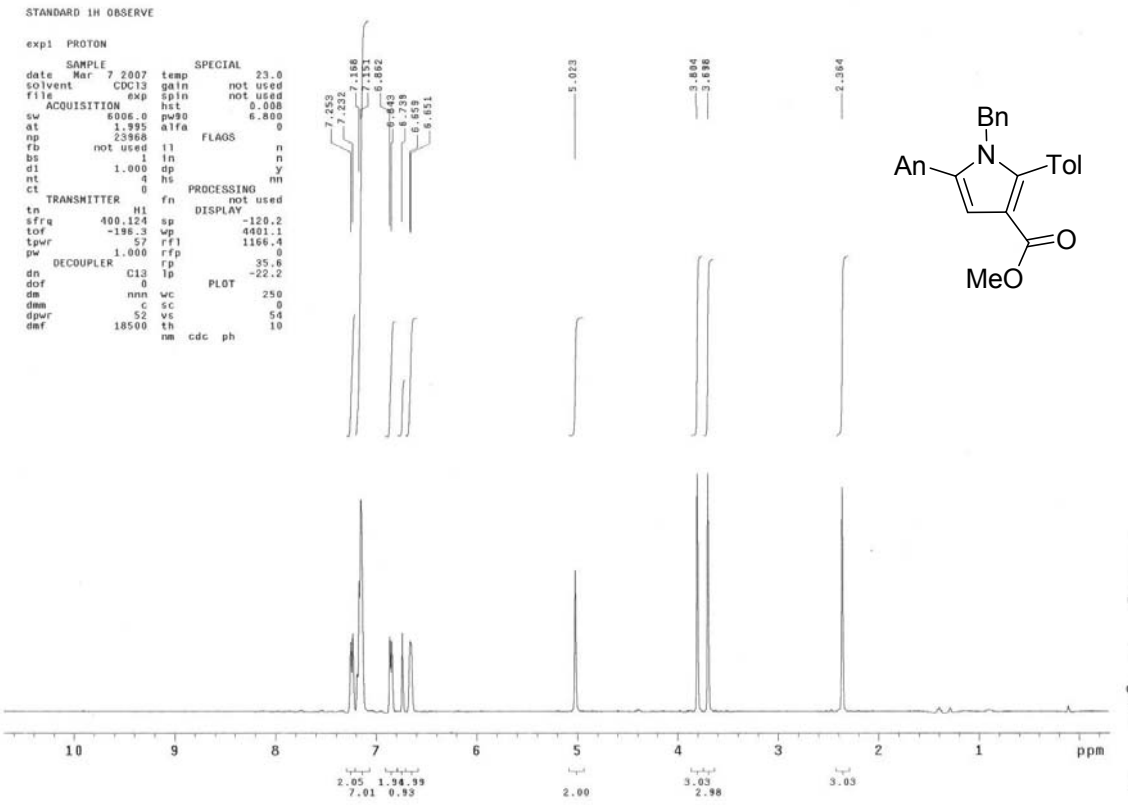
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date Mar 7 2007
solvent CDCl3
file exp
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d1 1.000
nt 4
ct 0
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sfrq 400.124
tof -196.3
tpwr 57
pw 1.000
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 52
dmf 18500
SPECIAL
temp 23.0
gain not used
spin not used
hst 0.008
pw90 6.800
alfa 0
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -120.2
wp 4401.1
rfl 1166.4
rfp 0
rp 35.6
lp -22.2
PLOT
wc 250
sc 0
vs 54
th 10
nm cdc ph
7.259
7.232
7.166
7.151
6.682
6.643
6.739
6.659
6.651
5.023
3.684
3.698
2.364
Bn
N
An
Tol
O
MeO
10
9
8
7
6
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ppm
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0.93
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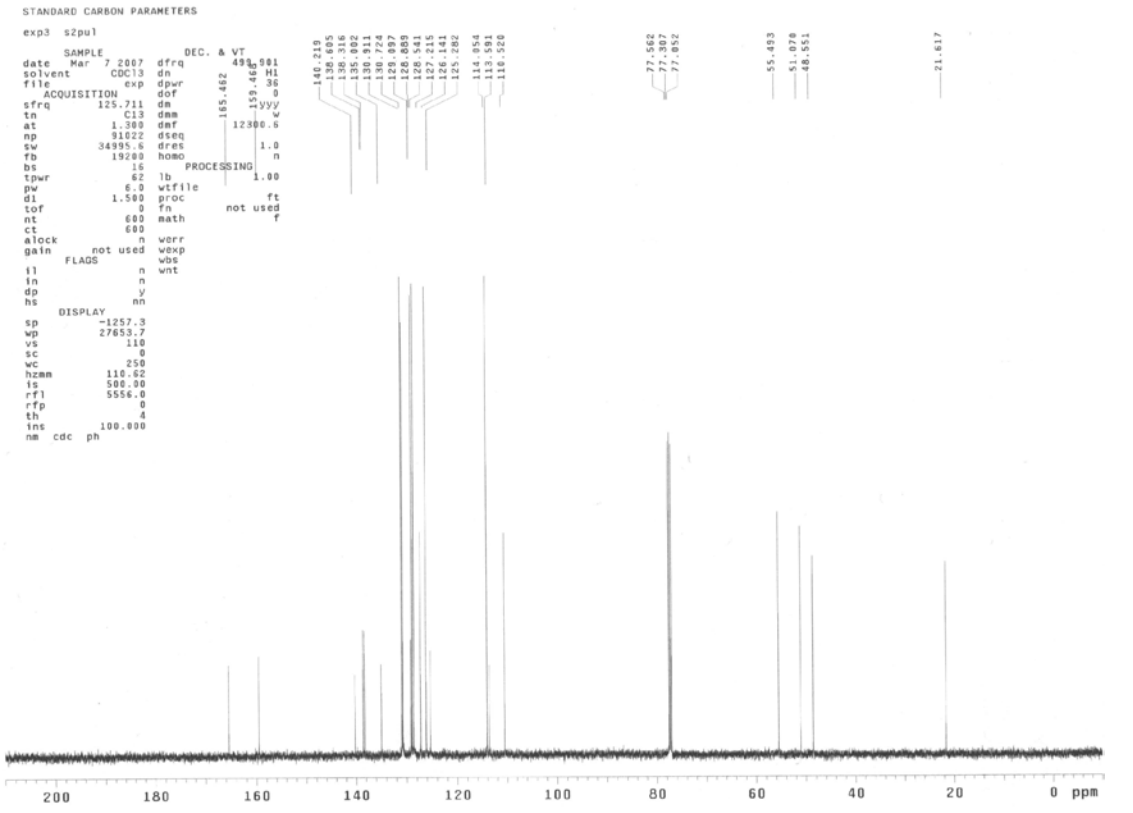
3.03


STANDARD CARBON PARAMETERS
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SAMPLE
date Mar 7 2007
solvent CDCl3
file exp
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tn C13
at 1.300
np 91622
sw 34995.6
fb 19200
bs 16
tpwr 62
pw 6.0
d1 1.500
tof 0
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ct 600
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
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sc 0
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is 500.00
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th 4
ins 100.800
nm cdc ph
DEC. & VT
dfrq 499.901
dn H1
dpwr 36
dof 0
dm yyy
dmm c
dmf 12300.6
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
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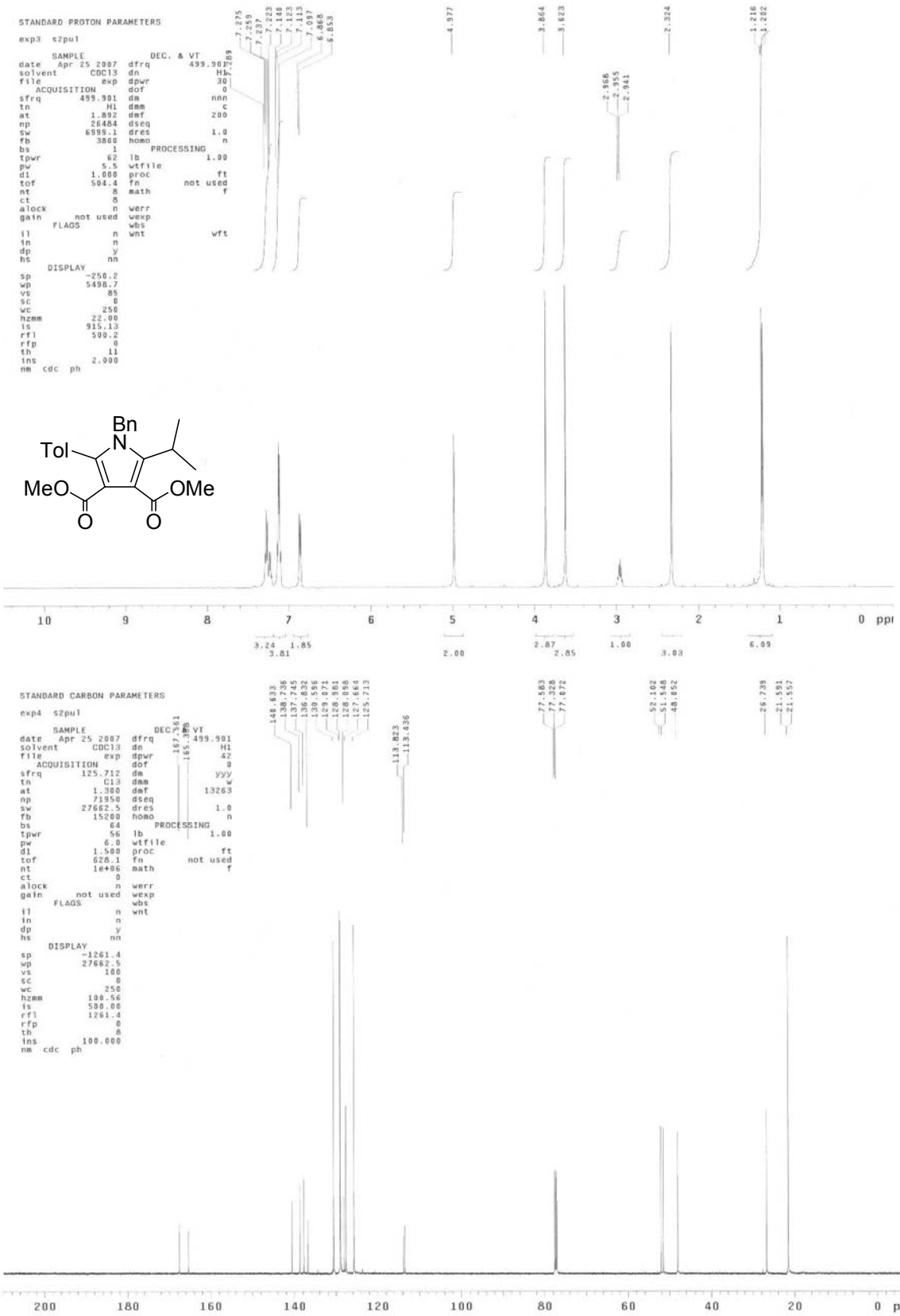
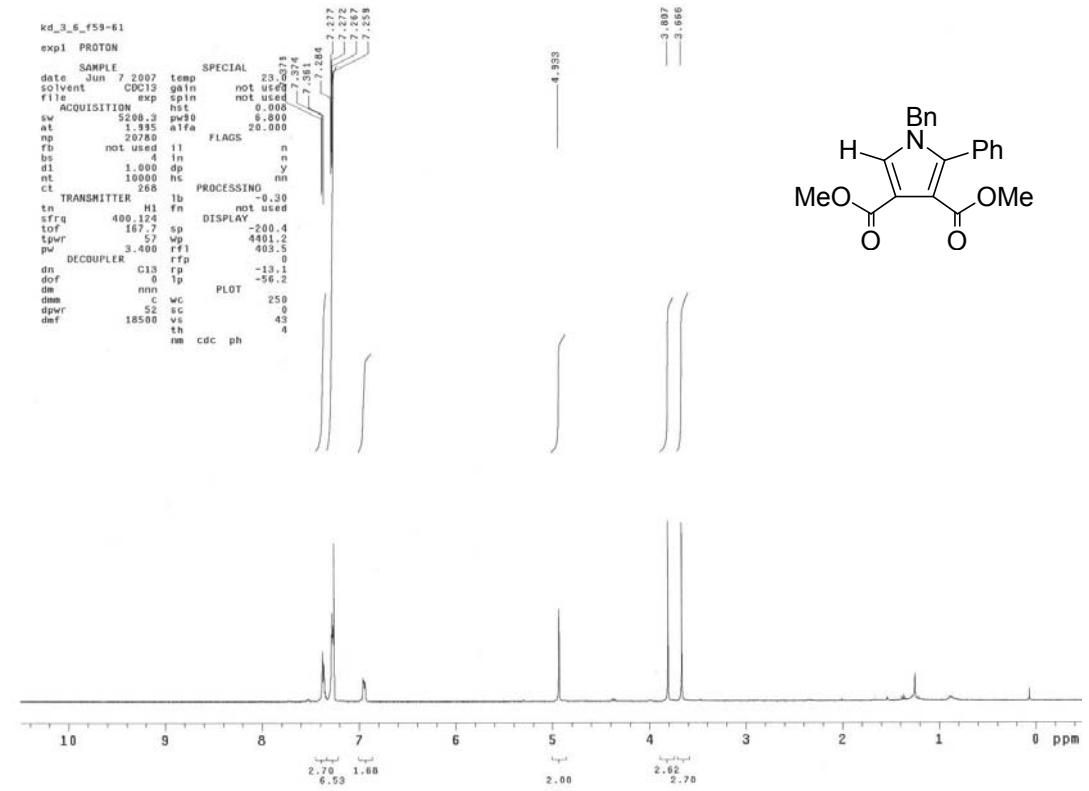
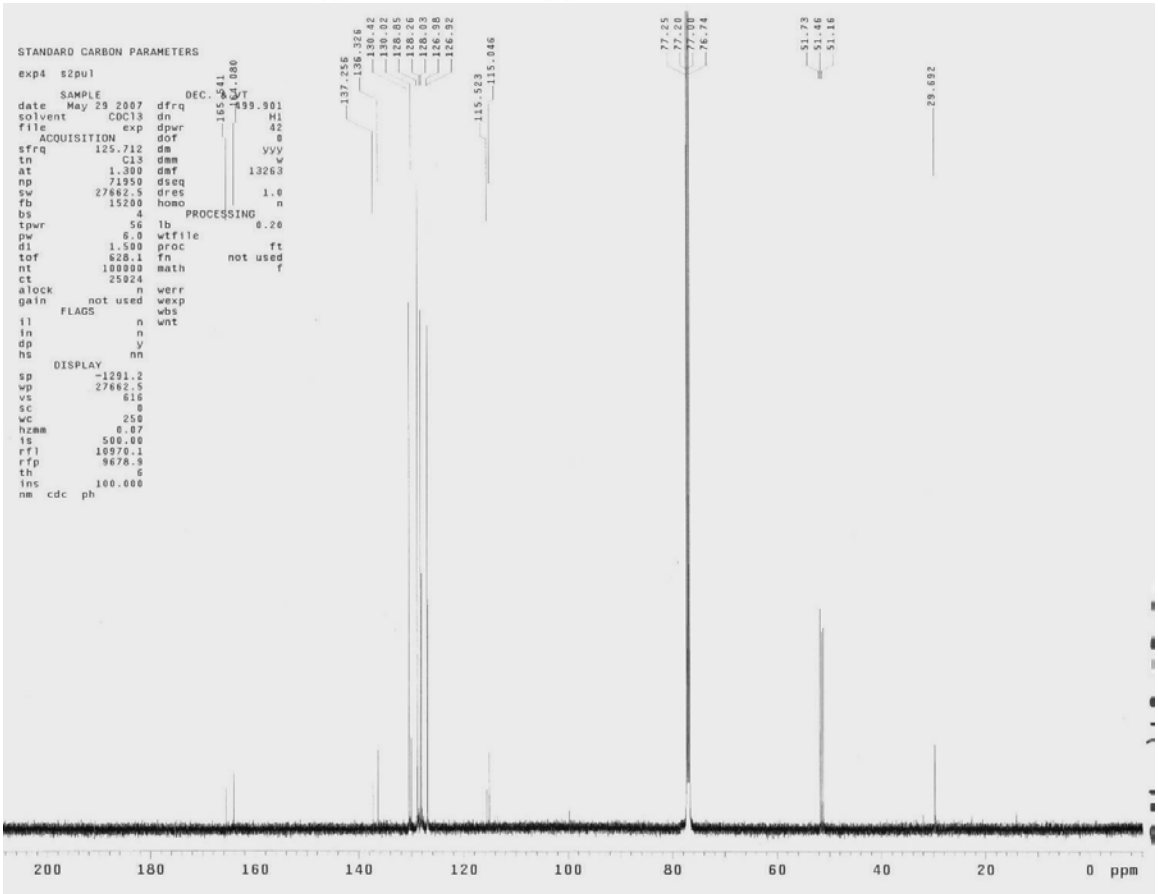
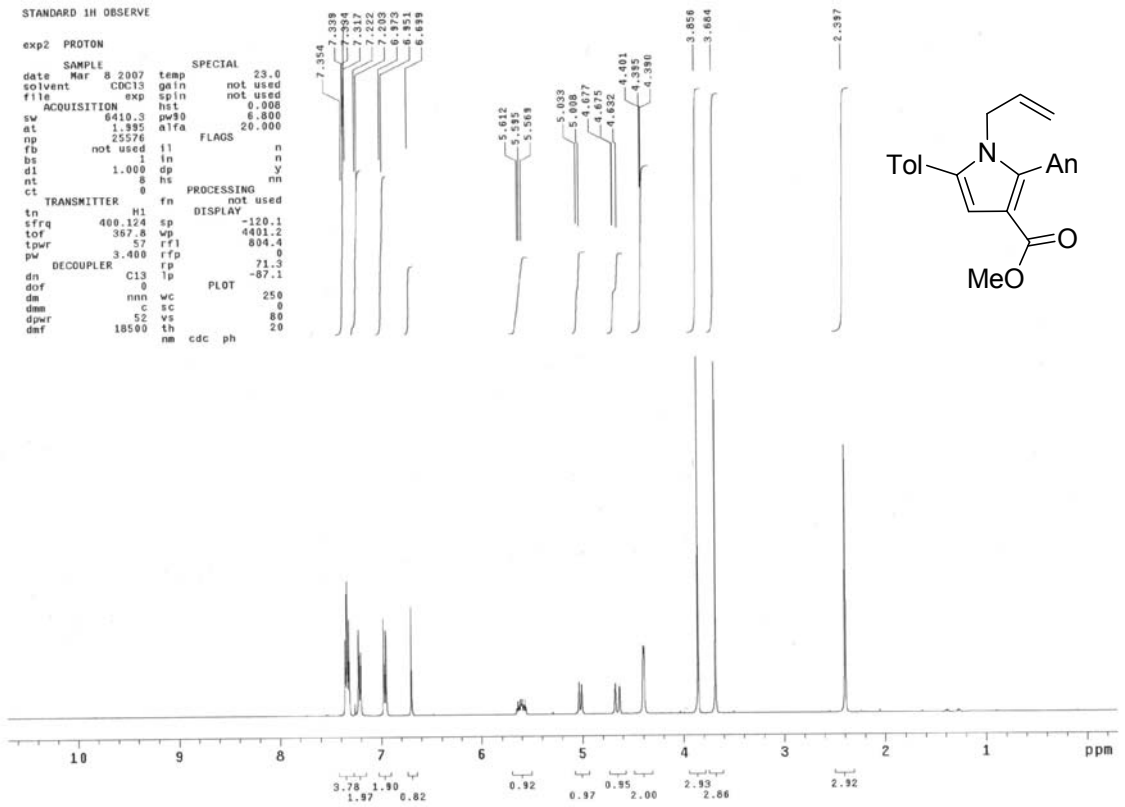
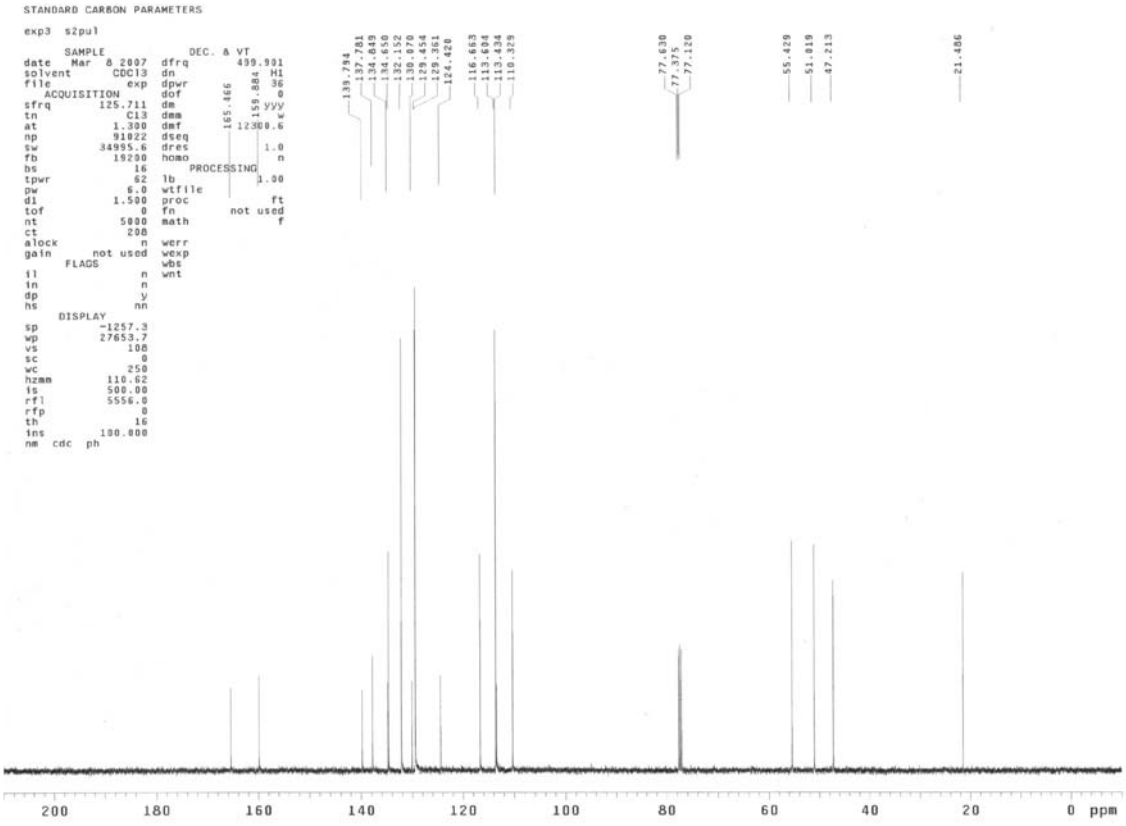
138.316
135.902
130.911
130.724
129.897
128.859
128.541
127.215
126.141
125.282
114.854
113.591
110.520
77.562
77.307
77.052
55.493
51.070
48.551
21.617
200
180
160
140
120
100
80
60
40
20
0 ppm

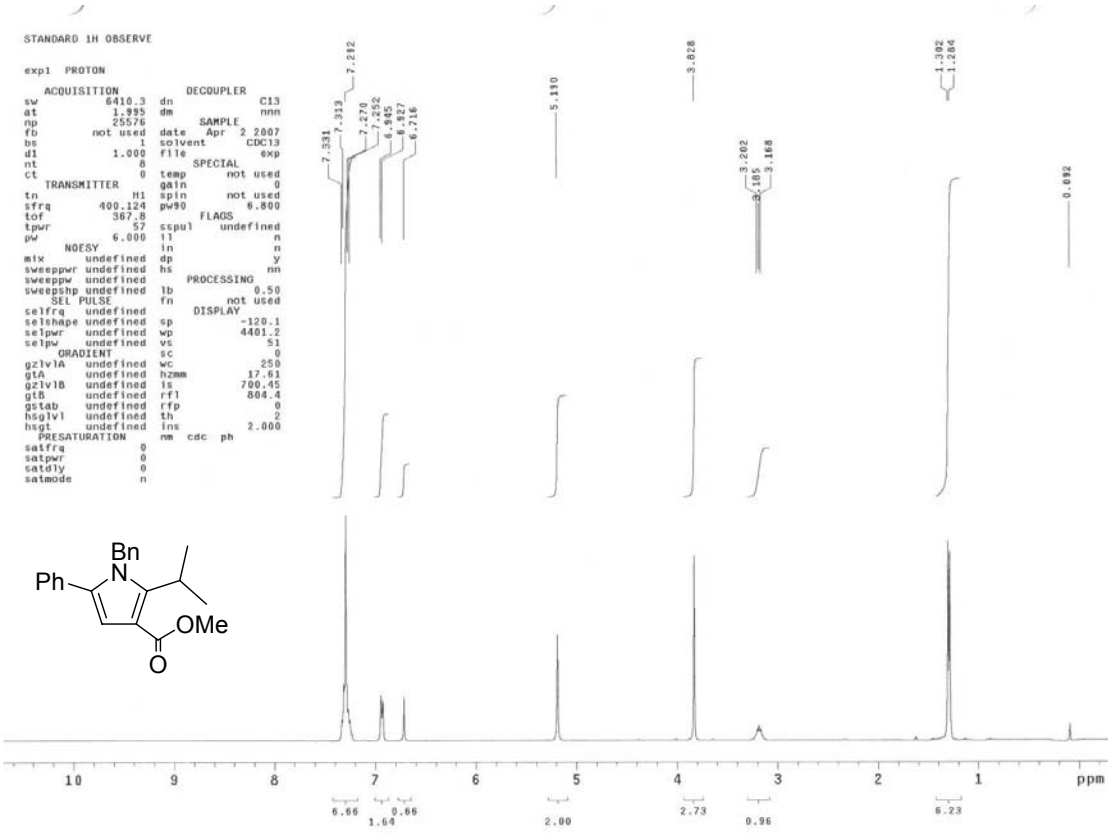

STANDARD PROTON PARAMETERS
exp3 s2pul
SAMPLE
date Apr 25 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 499.901
tn H1
at 1.892
np 26484
sw 6999.1
fb 3800
bs 1
tpwr 62
pw 5.5
d1 1.000
tof 504.4
nt 8
ct 6
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.2
wp 5498.7
vs 85
sc 0
wc 250
hzmm 22.00
is 915.13
rfl 509.2
rfp 0
th 11
ins 2.000
nm cdc ph
DEC. & VT
dfrq 499.901
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
7.275
7.259
7.237
7.223
7.148
7.138
7.113
7.097
6.868
6.853
4.977
3.864
3.623
2.966
2.955
2.941
2.324
1.216
1.202
Bn
N
Tol
MeO
OMe
O
O
10 9 8 7 6 5 4 3 2 1 0 ppm
3.24 1.85
3.81
2.00
2.87
2.85
1.00
3.03
6.09
STANDARD CARBON PARAMETERS
exp4 s2pul
SAMPLE
date Apr 25 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 125.712
tn C13
at 1.360
np 71950
sw 27662.5
fb 15200
bs 64
tpwr 56
pw 6.0
d1 1.500
tof 628.1
nt 1e+06
ct 0
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1261.4
wp 27662.5
vs 100
sc 0
wc 250
hzmm 100.56
is 500.00
rfl 1261.4
rfp 0
th 8
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.901
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 13263
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
167.361
165.386
140.633
138.736
137.745
136.832
130.596
129.071
128.581
128.068
127.666
125.713
113.823
113.436
77.583
77.328
77.072
52.102
51.548
48.852
26.739
21.591
21.537
200 180 160 140 120 100 80 60 40 20 0 ppm

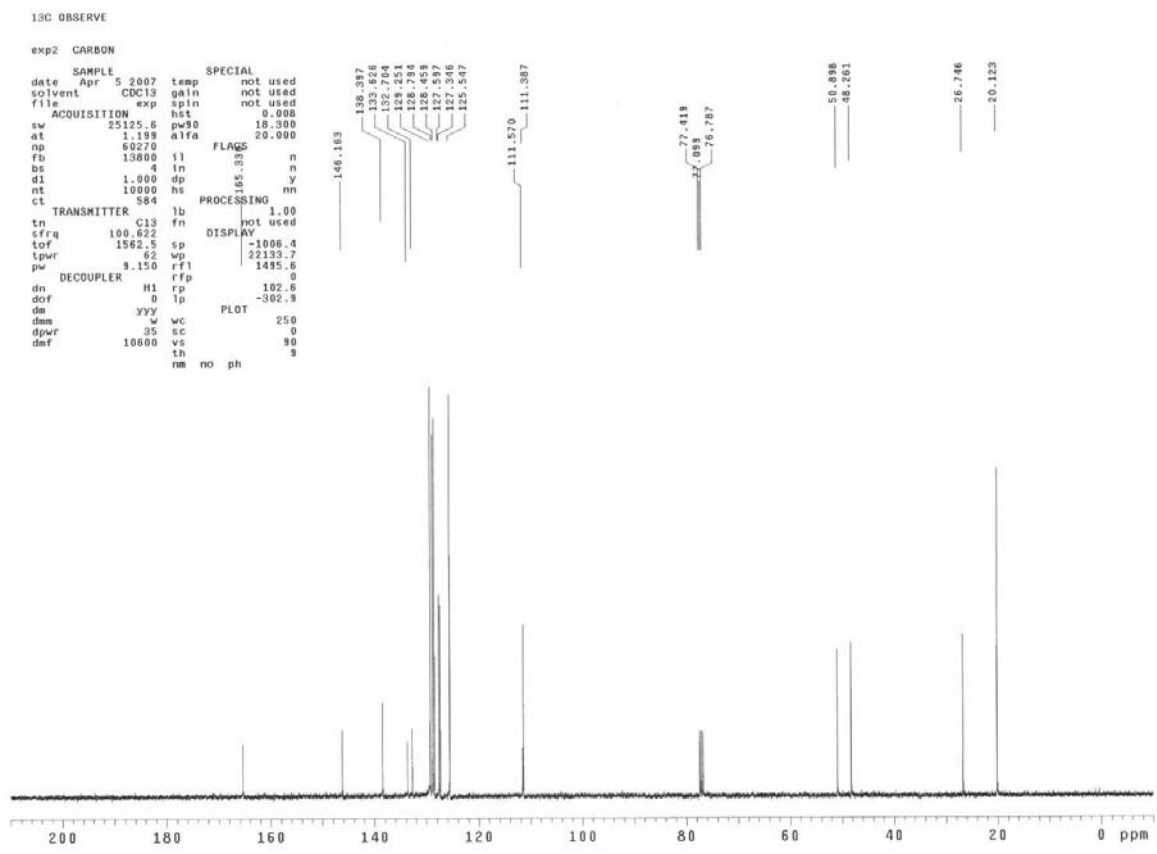

kd_3_6_f59-61
exp1 PROTON
SAMPLE
date Jun 7 2007
solvent CDCl3
file exp
ACQUISITION
sw 5208.3
at 1.995
np 20780
fb not used
bs 4
d1 1.000
nt 10000
ct 268
TRANSMITTER
tn H1
sfrq 400.124
tof 167.7
tpwr 57
pw 3.400
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 52
dmf 18500
SPECIAL
temp 23.0
gain not used
spin not used
hst 0.008
pw90 8.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb -0.30
fn not used
DISPLAY
sp -200.4
wp 4401.2
rfl 403.5
rfp 0
rp -13.1
lp -56.2
PLOT
wc 250
sc 0
vs 43
th 4
nm cdc ph
7.377
7.374
7.361
7.284
7.217
7.212
7.205
7.259
4.933
3.887
3.666
Bn
N
H
Ph
MeO
OMe
O
O
10
9
8
7
6
5
4
3
2
1
0 ppm
2.70
6.53
1.68
2.00
2.62
2.70

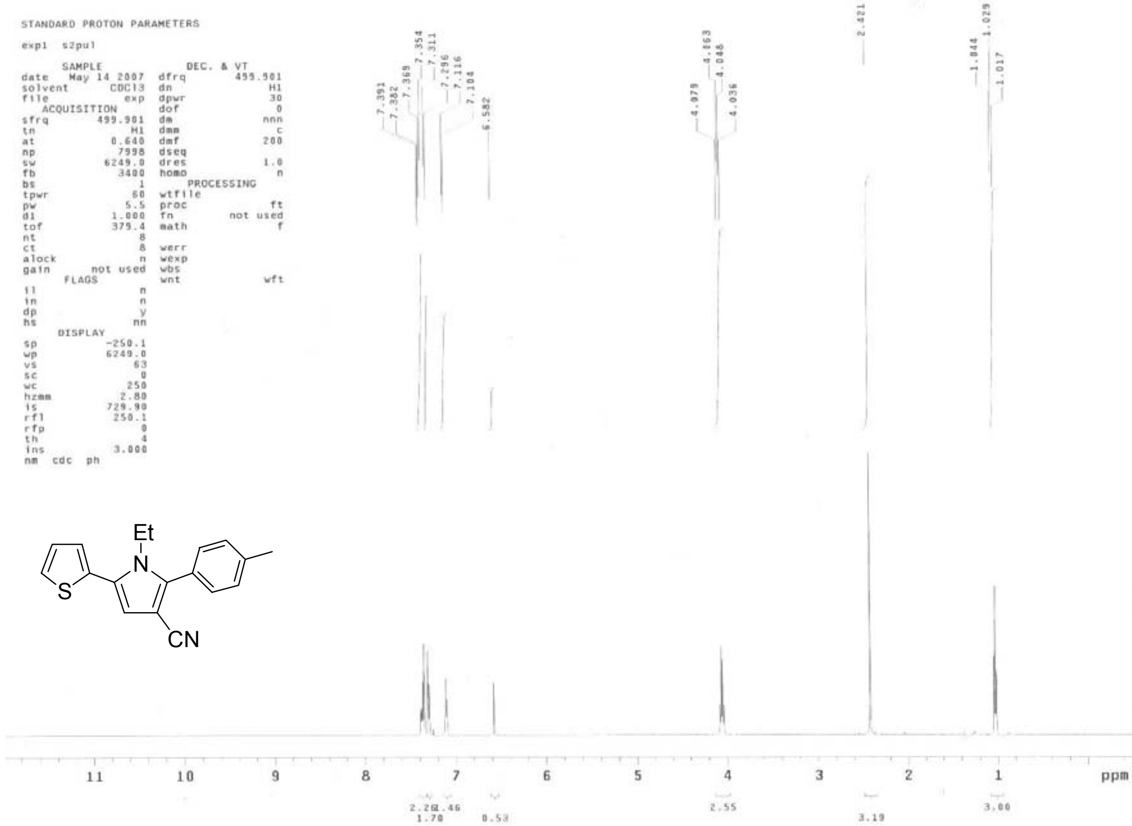

STANDARD CARBON PARAMETERS
exp4 s2pul
SAMPLE
date May 29 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 125.712
tn C13
at 1.300
np 71950
sw 27662.5
fb 15200
bs 4
tpwr 56
pw 6.0
d1 1.500
tof 628.1
nt 100000
ct 25024
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1291.2
wp 27662.5
vs 616
sc 0
wc 250
hzmm 0.87
ts 500.00
rfl 10970.1
rfp 9678.9
th 6
ins 100.000
nm cdc ph
DEC. SAT
dfrq -499.901
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 13263
dseq
dres 1.0
homo n
PROCESSING
lb 0.20
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
137.256
136.336
130.42
130.02
128.85
128.26
128.03
126.98
126.92
115.523
115.046
77.25
77.20
77.00
76.74
51.73
51.46
51.16
29.692
200
180
160
140
120
100
80
60
40
20
0 ppm

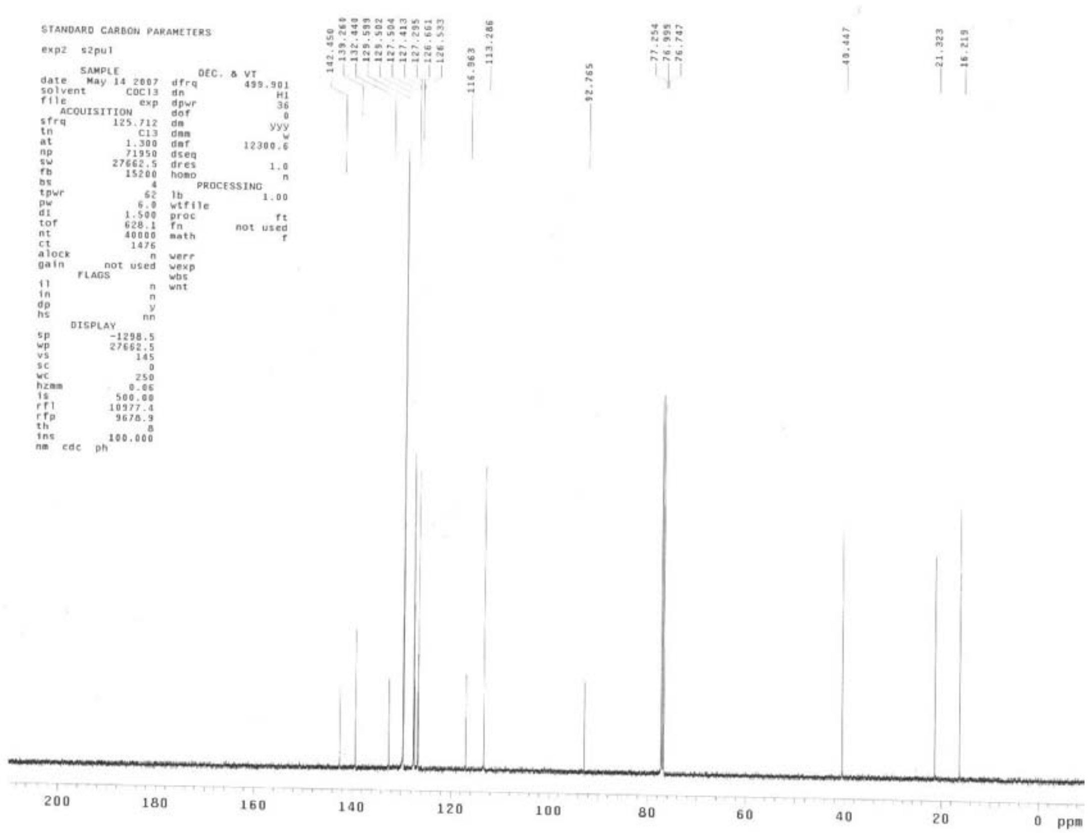

STANDARD 1H OBSERVE
exp2 PROTON
SAMPLE
date Mar 8 2007
solvent CDCl3
file exp
ACQUISITION
sw 6410.3
at 1.995
np 25576
fb not used
bs 1
d1 1.000
nt 8
ct 0
TRANSMITTER
tn H1
sfrq 400.124
tof 367.8
tpwr 57
pw 3.400
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 52
dmf 18500
SPECIAL
temp 23.0
gain not used
spin not used
hst 0.008
pw90 6.800
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
sp -120.1
wp 4401.2
rfl 804.4
rfp 0
rp 71.3
lp -87.1
PLOT
wc 250
sc 0
vs 80
th 20
nm cdc ph
7.354
7.338
7.334
7.317
7.222
7.203
6.973
6.951
6.699
5.612
5.595
5.568
5.033
5.008
4.677
4.675
4.632
4.401
4.395
4.390
3.856
3.684
2.397
Tol
N
An
O
MeO
10
9
8
7
6
5
4
3
2
1
ppm
3.78
1.90
1.97
0.82
0.92
0.97
0.95
2.00
2.93
2.86
2.92

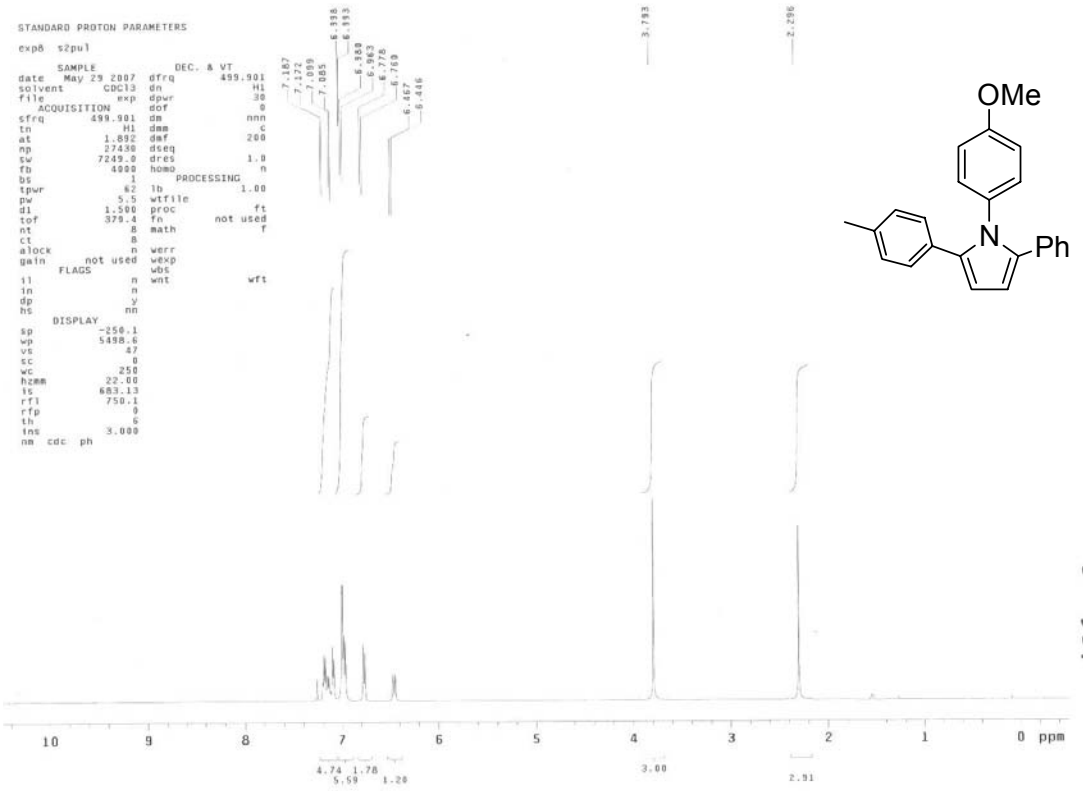

STANDARD CARBON PARAMETERS
exp3 s2pul
SAMPLE
date Mar 8 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 125.711
tn C13
at 1.300
np 91022
sw 34995.6
fb 19200
bs 16
tpwr 52
pw 6.0
d1 1.500
tof 0
nt 5000
ct 208
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1257.3
wp 27653.7
vs 108
sc 0
wc 250
hzmm 110.62
is 500.00
rfl 5556.0
rfp 0
th 16
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.901
dn H1
dpwr 36
dof 0
dm yyy
dmm w
dmf 12300.6
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
139.794
137.781
134.649
131.456
130.152
130.070
129.454
129.361
124.420
116.663
113.694
113.434
110.329
77.630
77.375
77.120
55.429
51.019
47.213
21.486
200
180
160
140
120
100
80
60
40
20
0 ppm

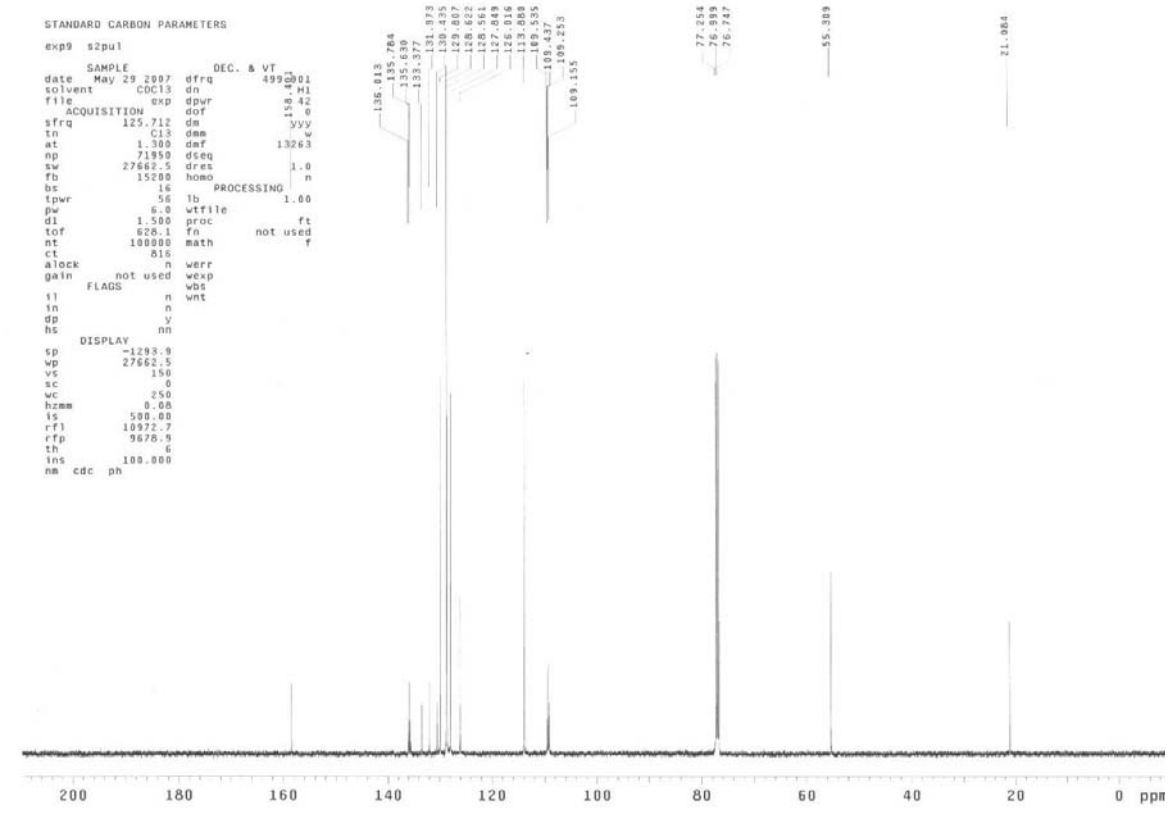

STANDARD 1H OBSERVE
exp1 PROTON
ACQUISITION
sw 6410.3
at 1.995
np 25576
fb not used
bs 1
d1 1.000
nt 8
ct 0
TRANSMITTER
tn H1
sfrq 400.124
tof 367.8
tpwr 57
pw 6.000
NOESY
mix undefined
sweeppwr undefined
sweeppw undefined
sweepshp undefined
SEL PULSE
selfrq undefined
selshape undefined
selpwr undefined
selpw undefined
GRADIENT
gzlvlA undefined
gtA undefined
gzlvlB undefined
gtB undefined
gstab undefined
hsglvl undefined
hsgt undefined
PRESATURATION
satfrq 0
satpwr 0
satdly 0
satmode n
DECOUPLER
dn C13
dm nnn
SAMPLE
date Apr 2 2007
solvent CDCl3
file 6xp
SPECIAL
temp not used
gain 0
spin not used
pw90 6.800
FLAGS
sspul undefined
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
fn not used
DISPLAY
sp -120.1
wp 4401.2
vs 51
sc 0
wc 250
hzmm 17.61
is 706.45
rfl 804.4
rfp 0
th 2
ins 2.000
nm cdc ph
7.331
7.313
7.282
7.270
7.252
6.945
6.927
6.716
5.110
3.828
3.202
3.185
3.168
1.392
1.284
0.692
Bn
N
Ph
OMe
O
10
9
8
7
6
5
4
3
2
1
ppm
6.66
1.64
0.66
2.00
2.73
0.96
6.23

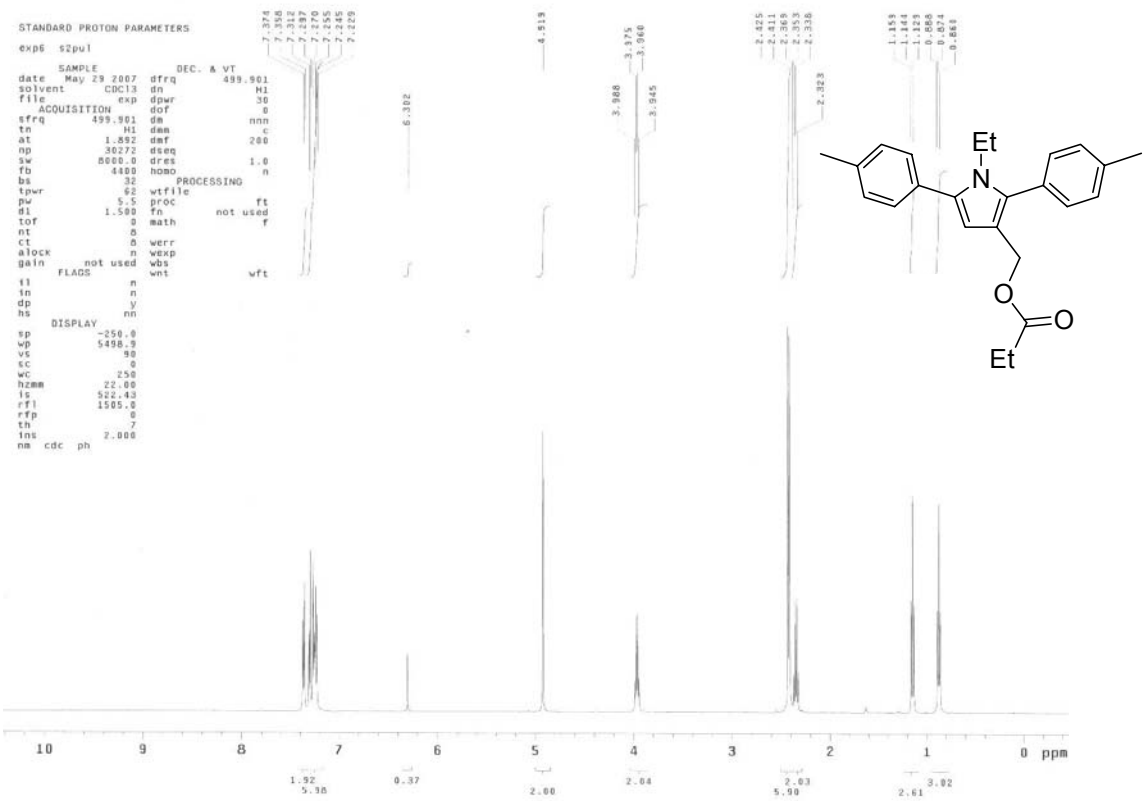

13C OBSERVE
exp2 CARBON
SAMPLE
date Apr 5 2007
solvent CDCl3
file exp
ACQUISITION
sw 25125.6
at 1.199
np 60270
fb 13800
bs 4
d1 1.000
nt 10000
ct 584
TRANSMITTER
tn C13
sfrq 100.622
tof 1562.5
tpwr 62
pw 9.150
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 35
dmf 10600
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 18.300
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -1006.4
wp 22133.7
rfl 1495.6
rfp 0
rp 102.6
lp -302.9
PLOT
wc 250
sc 0
vs 90
th 9
nm no ph
146.163
138.397
133.626
132.764
132.251
128.734
128.459
127.597
127.346
125.547
111.570
111.387
77.419
77.099
76.787
50.898
48.261
26.746
20.123
200
180
160
140
120
100
80
60
40
20
0 ppm

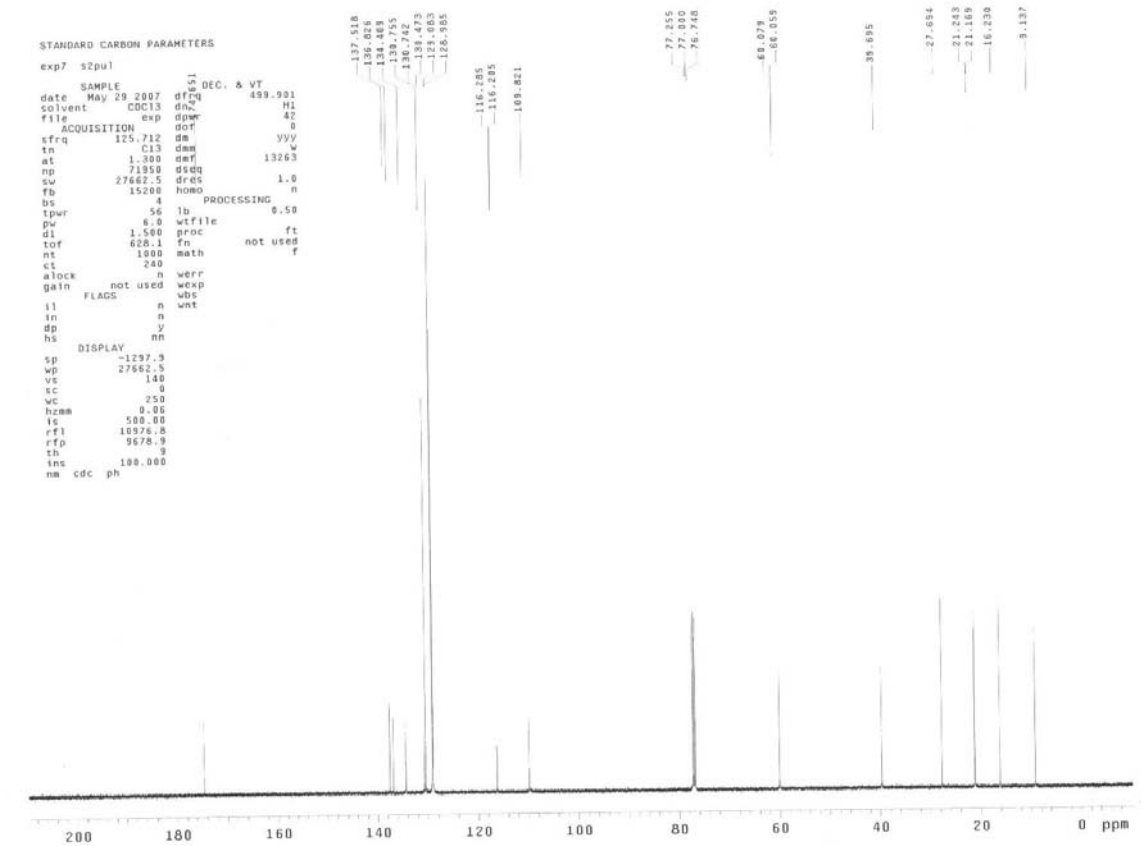

STANDARD PROTON PARAMETERS
exp1 s2pul
7.391
7.382
7.369
7.354
7.311
7.296
7.116
7.104
6.582
4.079
4.063
4.048
4.036
2.421
1.044
1.029
1.017
11
10
9
8
7
6
5
4
3
2
1
ppm
2.26
1.46
1.70
0.53
2.55
3.19
3.00
Et
N
S
CN


STANDARD CARBON PARAMETERS
exp2 s2pul
142.450
139.260
132.440
129.599
129.502
127.504
127.413
127.295
126.661
126.533
116.963
113.286
92.765
77.254
76.999
76.747
40.447
21.323
16.219
200
180
160
140
120
100
80
60
40
20
0
ppm


STANDARD PROTON PARAMETERS
exp8 s2pul
SAMPLE
date May 29 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 499.901
tn H1
at 1.892
np 27430
sw 7249.0
fb 4000
bs 1
tpwr 62
pw 5.5
d1 1.500
tof 379.4
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.1
wp 5498.6
vs 47
sc 0
wc 250
hzmm 22.00
is 683.13
rfl 750.1
rfp 0
th 6
ins 3.000
nm cdc ph
DEC. & VT
dfrq 499.901
dn H1
dpwr 38
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.8
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
7.187
7.172
7.099
7.085
6.998
6.993
6.980
6.963
6.778
6.769
6.467
6.446
3.783
2.286
OMe
N
Ph
10
9
8
7
6
5
4
3
2
1
0 ppm
4.74
1.78
5.59
1.20
3.00
2.91


STANDARD CARBON PARAMETERS
exp9 s2pul
SAMPLE
date May 29 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 125.712
tn C13
at 1.300
np 71950
sw 27662.5
fb 15200
bs 16
tpwr 56
pw 6.0
d1 1.500
tof 628.1
nt 100000
ct 816
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1293.9
wp 27662.5
vs 150
sc 0
wc 250
hzmm 8.68
is 500.00
rfl 10972.7
rfp 9678.9
th 6
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.901
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 13263
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
136.013
135.784
135.630
133.377
131.973
130.435
129.807
128.622
128.561
127.848
126.016
119.888
109.535
109.437
109.253
109.155
77.254
76.999
76.747
55.309
21.084
200
180
160
140
120
100
80
60
40
20
0 ppm


STANDARD PROTON PARAMETERS
exp6 s2pul
SAMPLE
date May 29 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 499.901
tn H1
at 1.892
np 30272
sw 8000.0
fb 4400
bs 32
tpwr 62
pw 5.5
dl 1.500
tof 0
nt 8
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -250.0
wp 5498.9
vs 90
sc 0
wc 250
hzmm 22.00
is 522.43
rfl 1505.0
rfp 0
th 7
ins 2.000
nm cdc ph
DEC. & VT
dfrq 499.901
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 280
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
7.374
7.358
7.312
7.297
7.276
7.255
7.245
7.229
6.382
4.919
3.988
3.975
3.960
3.945
2.425
2.411
2.369
2.353
2.336
2.323
1.159
1.144
1.128
0.888
0.874
0.860
Et
N
O
O
Et
10
9
8
7
6
5
4
3
2
1
0 ppm
1.92
5.98
0.37
2.00
2.04
2.03
5.90
2.61
3.02


STANDARD CARBON PARAMETERS
exp7 s2pul
SAMPLE
date May 29 2007
solvent CDCl3
file exp
ACQUISITION
sfrq 125.712
tn C13
at 1.300
np 71950
sw 27662.5
fb 15200
bs 4
tpwr 56
pw 6.0
dl 1.500
tof 628.1
nt 1000
ct 240
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -1297.9
wp 27662.5
vs 140
sc 0
wc 250
hzmm 0.06
is 500.00
rfl 10976.8
rfp 9678.9
th 9
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.901
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 13263
dseq
dres 1.0
homo n
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
137.518
136.026
134.469
130.755
130.742
130.473
129.053
126.955
116.285
116.285
109.821
77.255
77.000
76.748
68.079
66.055
39.695
27.694
21.243
21.169
16.230
9.137
200
180
160
140
120
100
80
60
40
20
0 ppm