# Rh(I)-Catalyzed Formal [2+2+2] Cycloadditions of 1,6-Diynes with Potassium (*Z*)-(2-bromovinyl)trifluoroborate: a New Strategy and a Facile Entry to Polysubstituented Benzene Derivatives

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## Supplementary Information

## Contents

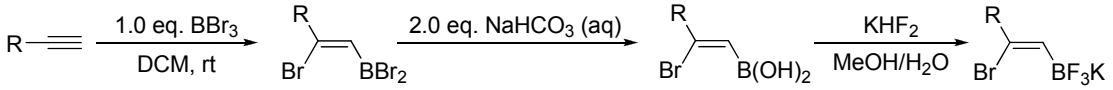
# Ⅰ. General Information

**Nuclear magnetic resonance spectra.** $^{1}H$ and $^{13}C$ spectra were recorded on a Bruker AVANCE 400 spectrometer, operating at 400 MHz for $^{1}H$ NMR, 100 MHz for $^{13}C$. NMR were reported downfield from $CDCl_3$ (δ: 7.27 ppm) for $^{1}H$ NMR. For $^{13}C$ NMR, chemical shifts were reported in the scale relative to the solvent of $CDCl_3$ (δ: 77.0 ppm) used as an internal reference.

**Mass spectroscopy.** Mass spectra were in general recorded on an AMD 402/3 or a HP 5989A mass selective detector.

**Chromatography.** Column chromatography was performed with silica gel (200-300 mesh ASTM).

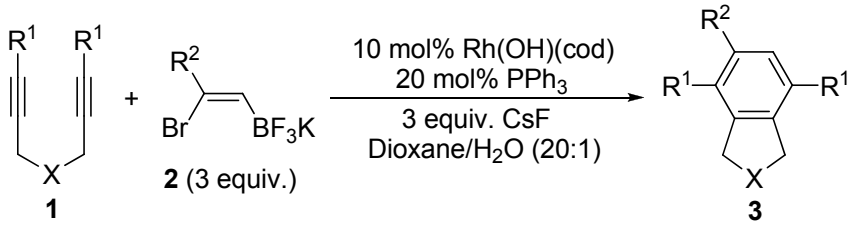
# Ⅱ. Procedure for Synthesis of Compound 2




**2**

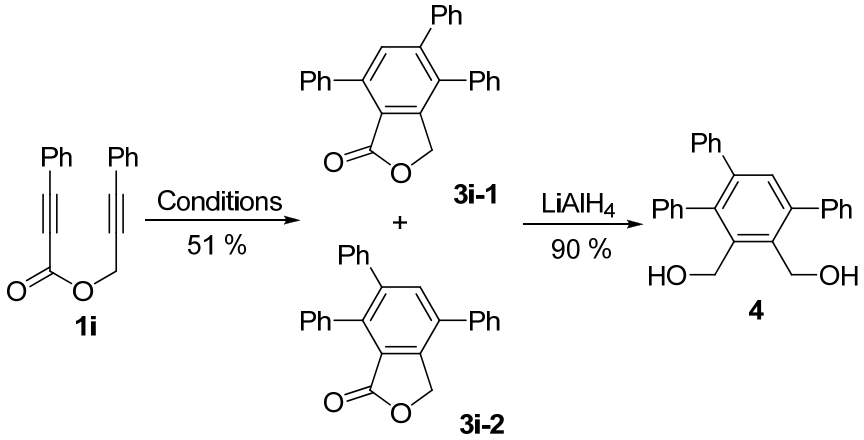
To a solution of phenylacetylene or 1-hexyne (50 mmol) in $CH_2Cl_2$ (100 mL) at $0^{o}C$ was added $BBr_3$ (50 mmol, 4.7 mL) in $CH_2Cl_2$ (50 mL). The mixture was stirred at room temperature for 15 min. The resulting red-purple reaction mixture was transferred by a double-ended needle to a mixture of diethyl ether (25 mL) and aqueous $NaHCO_3$ (8.5 g of $NaHCO_3$ in 75 mL of water) at $0^{o}C$. The mixture was stirred at room temperature for 15 min. The aqueous layer was separated and extracted with $CH_2Cl_2$, and the combined organic phase was washed with cold water and then brine. The organic phase was subsequently dried over anhydrous sodium sulfate. Removal of solvents under vacuum followed by washing with hexanes (15 mL) yielded crude boronic acid as a pale yellow solid. Crude boronic acid was dissolved in 50 mL of methanol and aqueous $KHF_2$ (150 mmol, 11.7 g, in 75 mL of water) was added slowly with vigorous stirring. After 15 min, the precipitated product was collected and washed with cold methanol. A white solid **2** was obtained (8.5 g, 59%). [Reference: M.-L. Yao, M. S. Reddy, W. Zeng, K. Hall, I. Walfish, G. W. Kabalka, *J. Org. Chem.* 2009, **74**, 1385–1387.]

## III. General procedure for the Rh(I)-catalyzed reaction of 1,6-Diynes with Potassium (*Z*)-(2-bromovinyl)trifluoroborate




1,6-Diynes **1** (0.3 mmol), Potassium (*Z*)-(2-bromovinyl)trifluoroborate **2** (0.9 mmol), $[Rh(OH)(cod)]_2$ (6.8 mg, 5 mol%), $PPh_3$ (15.7 mg, 20 mol%) and CsF (136.8 mg, 0.9 mmol) were added to 1,4-dioxane/$H_2O$ (20/1, 3 mL) in Schlenk tube. The mixture was stirred at 100°C and monitored by TLC. When the starting material was disappeared the reaction was cooled to room temperature. The contents were transferred to a round-bottom flask, and volatiles were removed in vacuo. Then, the mixture was purified by flash column chromatography (hexane: ethyl acetate = 30:1 to 5:1) to obtain the product **3**.

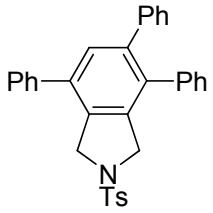
## IV. Procedure for Synthesis of Compound 4




The compound 3i was obtained under the conditions for Rh(I)-catalyzed cycloaddition. To a stirring solution of $LiAlH_4$ (0.18 mmol, 6.9 mg) in 1 mL of THF at 0°C was slowly added the solution of **3i** (0.15 mmol, 54.3 mg) in 3 mL THF. The mixture was brought to room temperature and heated at refluxed for another 3 hrs. Then 5 ml of 10% aqueous NaOH and 2 mL water was cautiously added to the reaction mixture,

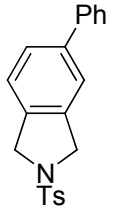
the resulting salts were filtered, and filtrate was extracted with 3 × 10 mL portions of ethyl acetate. The ethyl acetate solutions were combined, dried over anhydrous sodium sulfate, and the solvent was removed under vacuum. Then, the mixture was purified by flash column chromatography (hexane: ethyl acetate = 2:1) to obtain the product **4** (49.5 mg, 90%). **$^1$H NMR** (400 MHz, $CDCl_3$): δ 2.87 (br, 1H), 3.22 (br, 1H), 4.71 (s, 2H), 4.82 (s, 2H), 7.05-7.07 (m, 2H), 7.12-7.18 (m, 5H), 7.23-7.26 (m, 3H), 7.39-7.42 (m, 2H), 7.47 (t, *J* = 7.2 Hz, 2H), 7.54 (d, *J* = 6.8 Hz, 2H), ; **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 60.62, 60.97, 126.36, 126.84, 127.35, 127.53, 127.77, 128.26, 129.54, 129.70, 130.67, 131.91, 136.64, 138.73, 139.18, 140.79, 140.86, 141.28, 141.68, 142.62; MS (m/z): 366($M^+$); HRMS Calcd for $C_{26}H_{22}O_2$ 366.1620, Found 366.1623.

## V. The Data for Products 3 and Compound 5.




**3aa** (114mg, 76%)

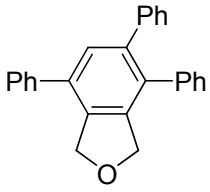
**$^1$H NMR** (400 MHz, $CDCl_3$): δ 2.41 (s, 3H), 4.52 (s, 2H), 4.77 (s, 2H), 7.03-7.08 (m, 4H), 7.11-7.18 (m, 3H), 7.21-7.25 (m, 3H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.36 (s, 1H), 7.39-7.48 (m, 5H), 7.71 (d, *J* = 8.0 Hz, 2H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 21.48, 53.82, 53.90, 126.63, 127.12, 127.16, 127.58, 127.77, 128.03, 128.27, 128.77, 129.42, 129.74, 129.79, 130.61, 133.26, 133.56, 134.83, 136.38, 136.57, 138.15, 139.24, 140.28, 141.38, 143.62; MS (m/z): 501($M^+$); HRMS Calcd for $C_{33}H_{27}NO_2S$ 501.1762, Found 501.1757.




**3ba** (21mg, 20%)

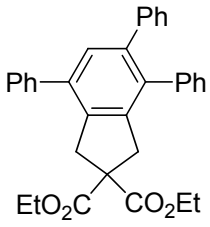
**$^1$H NMR** (400 MHz, $CDCl_3$): δ 2.42 (s, 3H), 4.67 (s, 2H), 4.68 (s, 2H), 7.24 (d, *J* = 8.0 Hz, 1H), 7.32-7.38 (m, 4H), 7.41-7.47 (m, 3H), 7.52 (d, *J* = 7.6 Hz, 2H), 7.80 (d, *J*

= 7.8 Hz, 2H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 21.49, 53.53, 53.71, 121.31, 122.91, 126.90, 127.09, 127.53, 127.61, 128.83, 129.83, 133.69, 135.12, 136.83, 140.53, 141.26, 143.70; MS (m/z): 349($M^+$); HRMS Calcd for $C_{21}H_{19}NO_2S$ 349.1136, Found 349.1129.



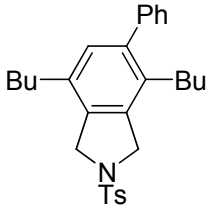

**3ca** (78mg, 75%)

**$^1$H NMR** (400 MHz, $CDCl_3$): δ 5.14 (s, 2H), 5.40 (s, 2H), 7.17-7.33 (m, 10H), 7.43 (t, *J* = 7.2 Hz, 1H), 7.50-7.57 (m, 5H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 73.85, 73.90, 126.50, 126.90, 127.56, 127.77, 127.84, 128.09, 128.70, 129.45, 129.90, 130.12, 133.44, 134.97, 136.37, 138.77, 139.69, 139.84, 140.64, 140.96; MS (m/z): 348($M^+$); HRMS Calcd for $C_{26}H_{20}O$ 348.1514, Found 348.1515.



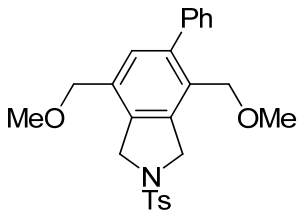

**3da** (122mg, 83%)

**$^1$H NMR** (400 MHz, $CDCl_3$): δ 1.28 (t, *J* = 7.2 Hz, 6H), 3.61 (s, 2H), 3.85 (s, 2H), 4.24 (q, *J* = 7.2 Hz, 4H), 7.20-7.23 (m, 7H), 7.29-7.34 (m, 3H), 7.43 (t, *J* = 7.2 Hz, 2H), 7.52 (t, *J* = 7.6 Hz, 2H), 7.62 (d, *J* = 7.2 Hz, 2H ); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 13.93, 40.35, 40.54, 60.33, 61.63, 126.19, 126.53, 127.16, 127.57, 127.92, 128.40, 128.49, 129.84, 129.98, 130.07, 135.96, 136.86, 137.28, 139.30, 140.11, 140.33, 140.47, 141.12, 171.51; MS (m/z): 490($M^+$); HRMS Calcd for $C_{33}H_{30}O_4$ 490.2144, Found 490.2146.



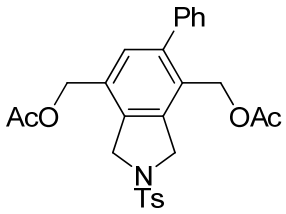

**3ea** (122mg, 88%)

**$^1$H NMR** (400 MHz, $CDCl_3$): δ 0.75 (t, *J* = 7.2 Hz, 3H), 0.93 (t, *J* = 7.2 Hz, 3H), 1.16 (q, *J* = 7.2 Hz, 2H), 1.23-1.31 (m, 2H), 1.36 (q, *J* = 7.2 Hz, 2H), 1.49-1.57 (m, 2H), 2.38-2.49 (m, 4H), 2.44 (s, 3H), 4.66 (s, 2H), 4.68 (s, 2H), 6.93 (s, 1H), 7.24 (d, *J* = 6.8 Hz, 2H ), 7.33-7.41 (m, 5H), 7.83 (d, *J* = 8.0 Hz, 2H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 13.59, 13.89, 21.47, 22.55, 22.68, 29.90, 32.06, 32.10, 32.48, 53.03, 53.36, 126.85, 127.55, 127.97, 129.13, 129.80, 129.92, 132.54, 133.77, 133.80, 134.40, 135.20, 141.30, 141.94, 143.62; MS (m/z): 461($M^+$); HRMS Calcd for $C_{29}H_{35}NO_2S$ 461.2389, Found 461.2381.



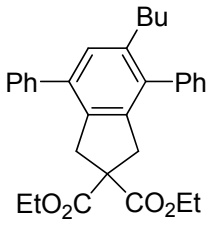

**3fa** (79mg, 60%)

**$^1$H NMR** (400 MHz, $CDCl_3$): δ 2.42 (s, 3H), 3.28 (s, 3H), 3.36 (s, 3H), 4.23 (s, 2H), 4.39 (s, 2H), 4.68 (s, 2H), 4.76 (s, 2H), 7.15 (s, 1H), 7.29-7.41 (m, 7H), 7.82 (d, *J* = 8.4 Hz, 2H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 21.49, 52.55, 52.99, 58.32, 58.36, 69.82, 72.43, 127.33, 127.58, 128.12, 129.24, 129.27, 129.34, 129.81, 132.28, 133.69, 134.51, 137.66, 139.90, 142.25, 143.60; MS (m/z): 437($M^+$); HRMS Calcd for $C_{25}H_{27}NO_4S$ 437.1661, Found 437.1665.



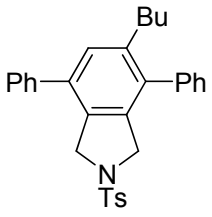

**3ga** (59mg, 40%)

**$^1$H NMR** (400 MHz, $CDCl_3$): δ 2.03 (s, 3H), 2.10 (s, 3H), 2.44 (s, 3H), 4.71 (s, 4H), 4.90 (s, 2H), 5.03 (s, 2H), 7.21-7.24 (m, 3H), 7.35-7.41 (m, 5H), 7.82 (d, *J* = 8.0 Hz, 2H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 20.66, 20.76, 21.50, 52.50, 52.94, 61.74, 63.54, 127.60, 127.72, 127.74, 128.34, 129.05, 129.87, 130.10, 130.66, 133.48, 134.81, 137.73, 139.13, 143.24, 143.92, 170.28, 170.44; MS (m/z): 493($M^+$); HRMS Calcd for $C_{27}H_{27}NO_6S$ 493.1559, Found 493.1565.



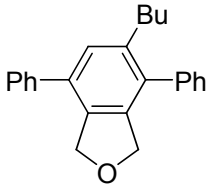

**3db** (120mg, 85%)

**$^1$H NMR** (400 MHz, $CDCl_3$): δ 0.82 (t, *J* = 7.2 Hz, 3H), 1.24 (t, *J* = 7.2 Hz, 3H), 1.28-1.41 (m, 2H), 1.45-1.52 (m, 2H), 2.51 (t, *J* = 8.0 Hz, 2H), 3.39 (s, 2H), 3.76 (s, 2H), 4.19 (q, *J* = 7.2 Hz, 4H), 7.23 (s, 1H), 7.32 (d, *J* = 6.8 Hz, 2H), 7.38-7.42 (m, 2H), 7.49 (t, *J* = 8.4 Hz, 4H), 7.56 (d, *J* = 7.2 Hz, 2H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 13.73, 13.90, 22.43, 32.50, 33.62, 40.28, 40.32, 60.22, 61.50, 126.81, 126.92, 128.24, 128.30, 128.44, 128.61, 129.04, 131.59, 134.64, 137.02, 137.12, 139.62, 139.67, 139.94, 140.75, 171.56; MS (m/z): 470($M^+$); HRMS Calcd for $C_{31}H_{34}O_4$ 470.2457, Found 470.2457.




**3ab** (108mg, 75%)

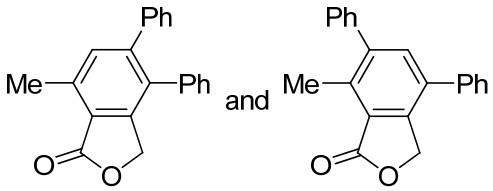
**$^1$H NMR** (400 MHz, $CDCl_3$): δ 0.77 (t, *J* = 7.2 Hz, 3H), 1.15-1.24 (m, 2H), 1.38-1.45 (m, 2H), 2.42 (s, 3H), 2.46 (t, *J* = 8.0 Hz, 2H), 4.37 (s, 2H), 4.72 (s, 2H), 7.19 (d, *J* = 6.8 Hz, 2H), 7.23 (s, 1H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.38-7.50 (m, 8H), 7.70 (d, *J* = 8.0 Hz, 2H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 13.70, 21.44, 22.33, 32.27, 33.57, 53.72, 53.82, 127.45, 127.52, 127.55, 127.97, 128.56, 128.60, 128.67, 129.31, 129.71, 131.11, 133.65, 135.88, 136.07, 136.13, 138.32, 139.66, 141.13, 143.50; MS (m/z): 481($M^+$); HRMS Calcd for $C_{31}H_{31}NO_2S$ 481.2075, Found 481.2071.




**3cb** (69mg, 70%)

**$^1$H NMR** (400 MHz, $CDCl_3$): δ 0.82 (t, *J* = 7.2 Hz, 3H), 1.23-1.31 (m, 2H), 1.47-1.54

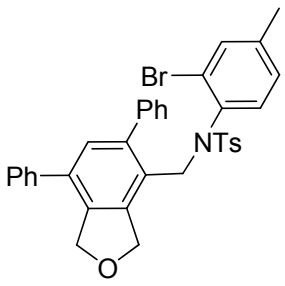
(m, 2H), 2.57 (t, *J* = 8.0 Hz, 2H), 4.92 (s, 2H), 5.30 (s, 2H), 7.29-7.34 (m, 3H), 7.39-7.51 (m, 8H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 13.78, 22.45, 32.21, 33.76, 73.73, 73.98, 127.23, 127.36, 127.84, 128.41, 128.65, 128.73, 134.24, 134.57, 134.78, 139.02, 139.35, 140.20, 140.67; MS (m/z): 328 ($M^+$); HRMS Calcd for $C_{24}H_{24}O$ 328.1827, Found 328.1824.




**3h** (48 mg, 53%)

Two isomers with the ratio of 2:1. MS (m/z): 300 ($M^+$); HRMS Calcd for $C_{21}H_{16}O_2$ 300.1150, Found 300.1152

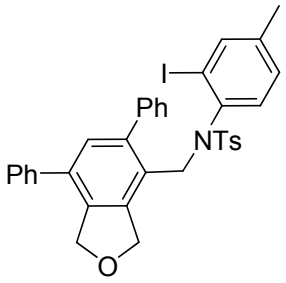
For minor isomer: **$^1$H NMR** (400 MHz, $CDCl_3$): δ 2.67 (s, 1.5H), 5.39 (s, 1H), 7.07-7.09 (m, 0.67H), 7.13-7.15 (m, 0.67H), 7.22-7.24 (m, 1H), 7.25-7.27 (m, 0.33H), 7.28-7.29 (m, 0.33H), 7.35-7.38 (m, 0.33H), 7.40 (s, 0.33H), 7.42-7.51 (m, 1.67H), 7.60 (s, 0.5H). For major isomer: **$^1$H NMR** (400 MHz, $CDCl_3$): δ 2.78 (s, 3H), 5.14 (s, 2H), 7.07-7.09 (m, 1.33H), 7.13-7.15 (m, 1.33H), 7.22-7.24 (m, 2H), 7.25-7.27 (m, 0.67H), 7.28-7.29 (m, 0.67H), 7.35-7.38 (m, 0.67H), 7.40 (s, 0.67H), 7.42-7.51 (m, 3.33H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 14.62, 17.13, 68.33, 68.90, 122.44, 124.19, 127.31, 127.51, 127.54, 127.71, 127.97, 128.17, 128.37, 128.52, 129.07, 129.33, 129.69, 132.70, 133.22, 133.77, 135.26, 136.33, 136.50, 137.58, 138.48, 139.58, 139.81, 143.67, 144.24, 146.14, 146.81, 171.33, 171.37.




**3ja-1** (105mg, 56%)

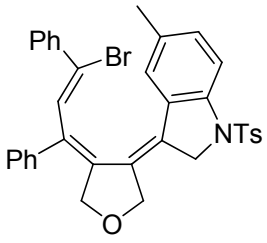
**$^1$H NMR** (400 MHz, $CDCl_3$): δ 2.33 (s, 3H), 2.44 (s, 3H), 4.69 (d, *J* = 14.0 Hz, 1H), 4.80 (d, *J* = 14.0 Hz, 1H), 4.92 (s, 2H), 5.07-5.08 (m, 2H), 6.89-6.92 (m, 2H), 6.97-7.00 (m, 3H), 7.04-7.06 (m, 2H), 7.13-7.15(m, 3H), 7.19-7.20 (m, 3H), 7.30 (d, *J*

= 8.0 Hz, 2H), 7.40 (s, 1H), 7.70 (d, *J* = 8.0 Hz, 2H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 20.77, 21.58, 52.07, 72.94, 73.77, 125.27, 126.38, 126.87, 127.64, 127.76, 127.98, 128.00, 128.79, 129.33, 129.55, 129.80, 131.39, 131.88, 134.12, 134.46, 134.77, 136.65, 138.29, 138.68, 139.20, 140.31, 140.42, 140.49, 143.73; MS (m/z): 623 ($M^+$); HRMS Calcd for $C_{35}H_{30}BrNO_3S$ 623.1130, Found 623.1128




**3ja-2** (99mg, 50%)

**$^{1}$H NMR** (400 MHz, $CDCl_3$): δ 2.32 (s, 3H), 2.45 (s, 3H), 4.47 (d, *J* = 14.0 Hz, 1H), 4.87 (d, *J* = 14.0 Hz, 2H), 4.99 (dd, *J* = 12.8 Hz, *J* = 21.2 Hz, 2H), 5.12 (d, *J* = 12.8 Hz, 1H), 6.83-6.87 (m, 3H), 6.91 (s, 1H), 6.99-7.01 (m, 2H), 7.07-7.12 (m, 4H), 7.19-7.20 (m, 3H), 7.32 (d, *J* = 8.2 Hz, 2H), 7.72 (d, *J* = 8.2 Hz, 3H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 20.53, 21.62, 52.93, 73.11, 73.84, 102.86, 126.36, 126.89, 127.17, 127.61, 128.02, 128.26, 129.34, 129.61, 129.69, 129.80, 130.23, 131.79, 134.33, 136.24, 138.45, 138.70, 138.75, 139.29, 140.24, 140.41, 140.48, 140.98, 143.89; MS (m/z): 671 ($M^+$); HRMS Calcd for $C_{35}H_{30}INO_3S$ 671.0991, Found 671.0993.

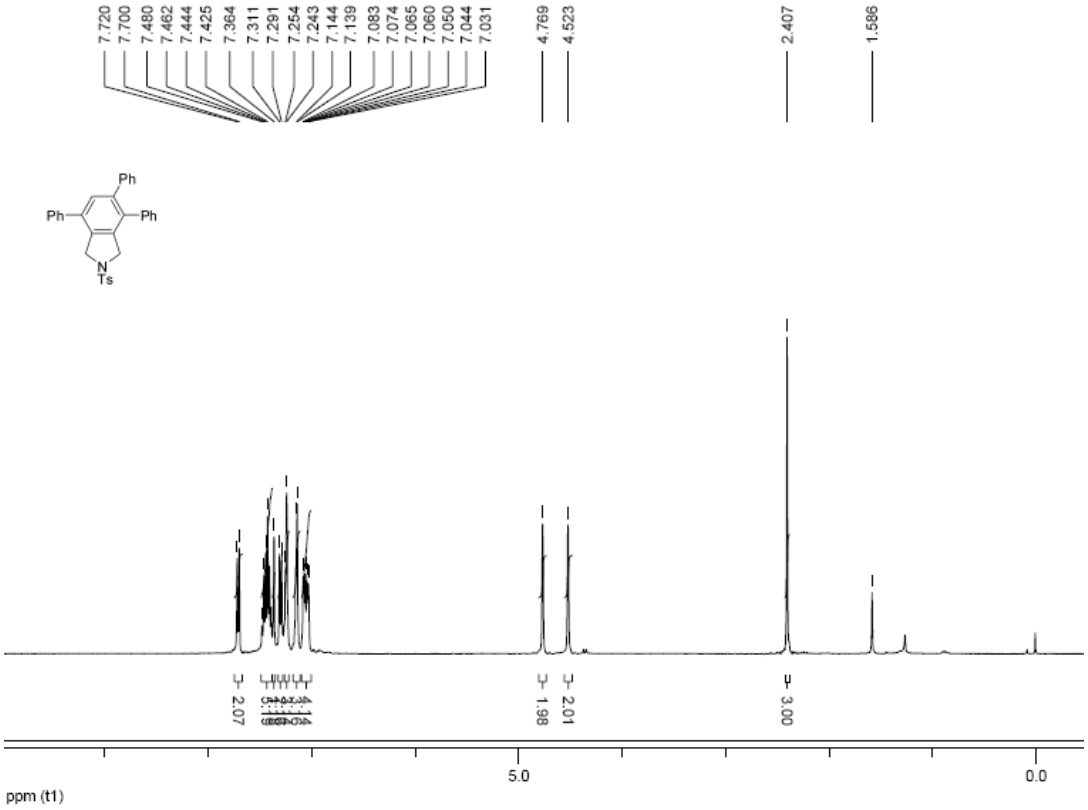


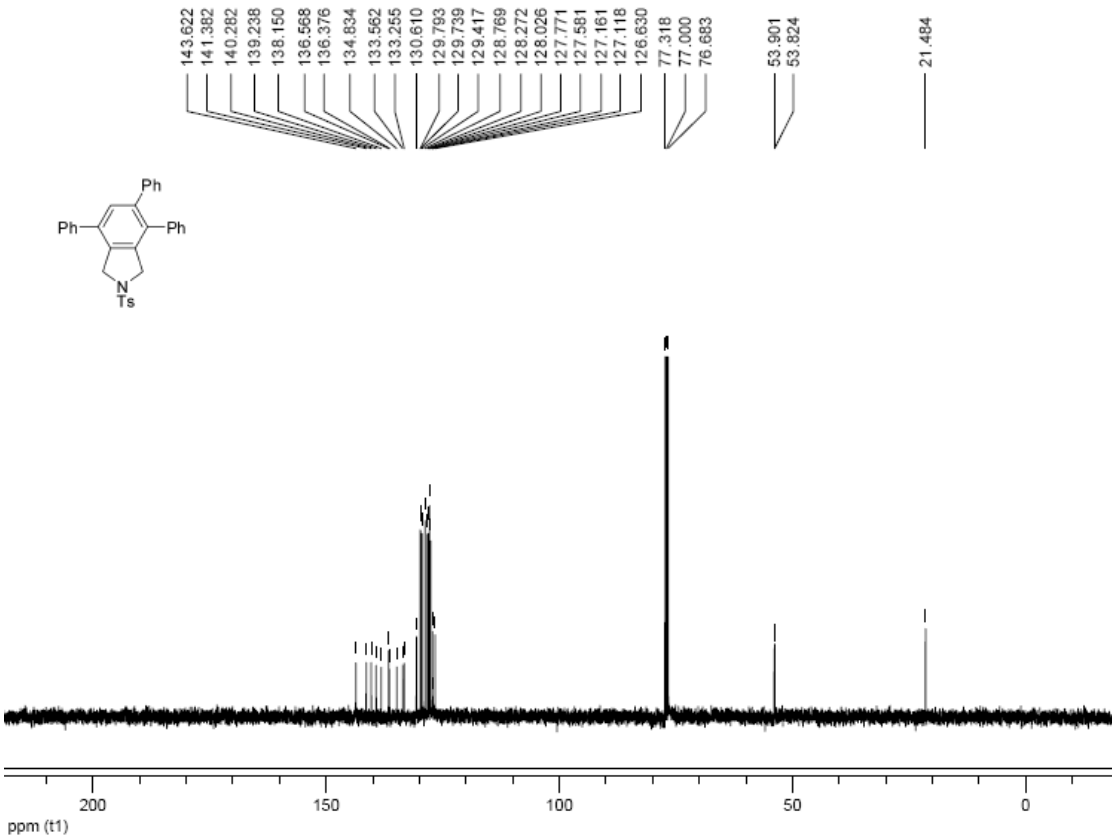

**5**

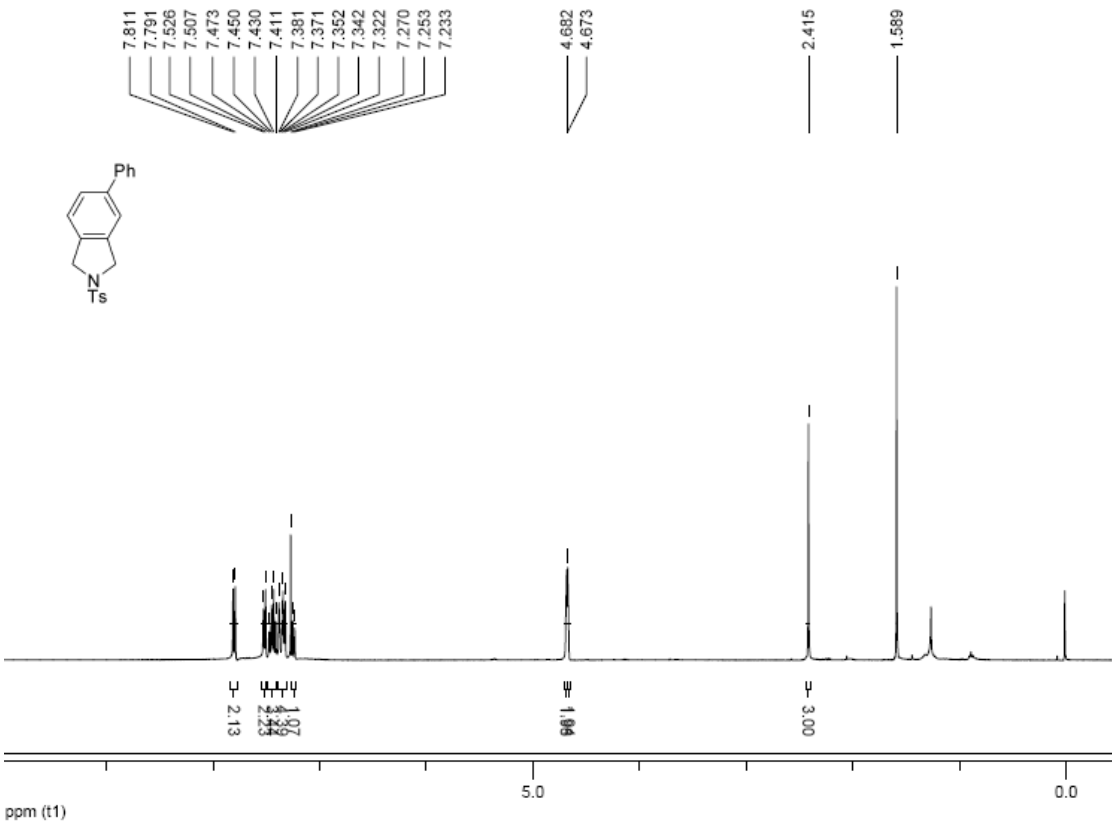
**$^{1}$H NMR** (400 MHz, $CDCl_3$): δ 2.26 (s, 3H), 2.42 (s, 3H), 4.71 (d, *J* = 13.6 Hz, 1H), 4.99 (d, *J* = 13.2 Hz, 1H), 5.23-5.30 (m, 2H), 5.39 (d, *J* = 13.6 Hz, 1H), 5.70 (d, *J* = 13.2 Hz, 1H), 6.18 (d, *J* = 8.0 Hz, 1H), 6.63 (d, *J* = 8.0 Hz, 1H), 6.68 (d, *J* = 6.8 Hz, 2H), 7.00 (s, 1H), 7.14-7.18 (m, 3H), 7.22-7.24 (m, 3H), 7.37-7.43 (m, 5H), 7.51 (d, *J* = 8.0 Hz, 2H); **$^{13}$C NMR** (100 MHz, $CDCl_3$): δ 20.72, 21.60, 48.52, 73.44, 73.95, 124.74, 125.91, 126.75, 127.59, 127.69, 127.81, 128.21, 128.55, 128.64, 129.41,

129.51, 129.71, 130.78, 133.94, 134.15, 134.30, 135.27, 135.76, 136.91, 139.53, 139.83, 142.53, 143.04, 143.69; MS (m/z): 623 ($M^+$); HRMS Calcd for $C_{35}H_{30}BrNO_3S$ 623.1130, Found 623.1133.

# VI. The Spectra of Products 3, Compounds 4 and 5

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7.450
7.430
7.411
7.381
7.371
7.352
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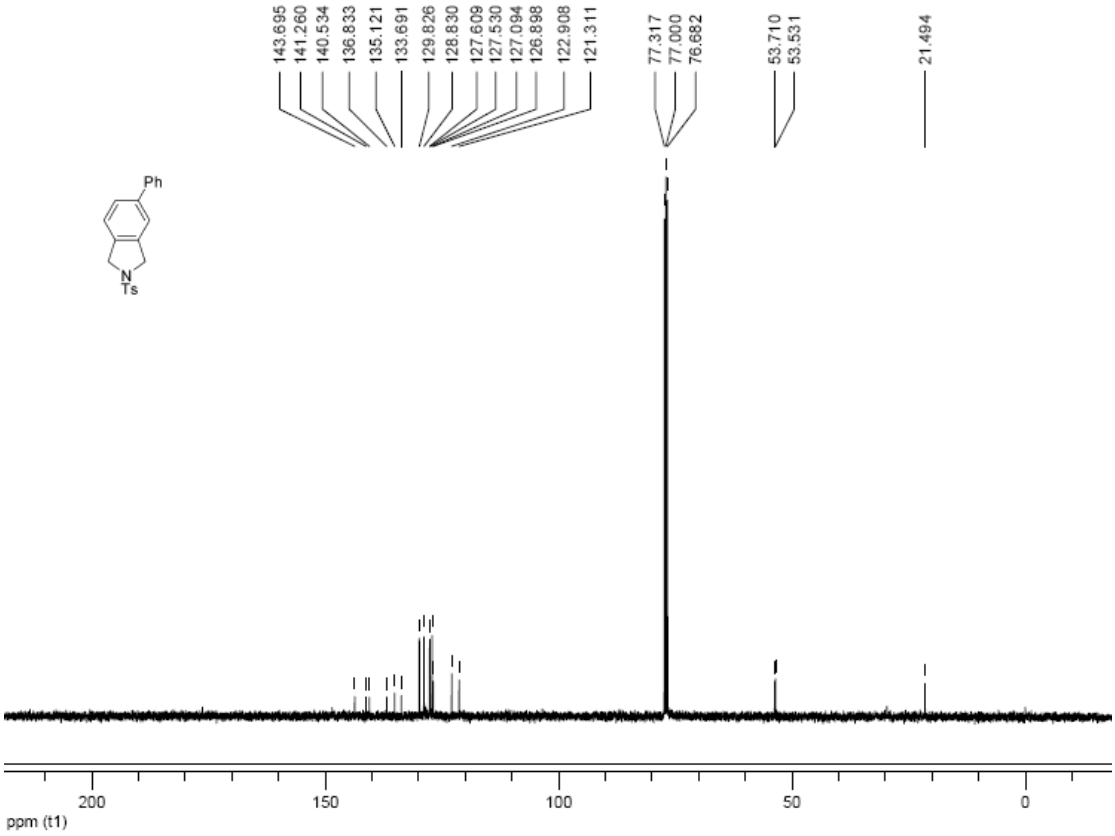
7.322
7.270
7.253
7.233
4.682
4.673
2.415
1.589
Ph
N
Ts
2.13
3.00
5.0
0.0
ppm (t1)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141.260
140.534
136.833
135.121
133.691
129.826
128.830
127.609
127.530
127.094
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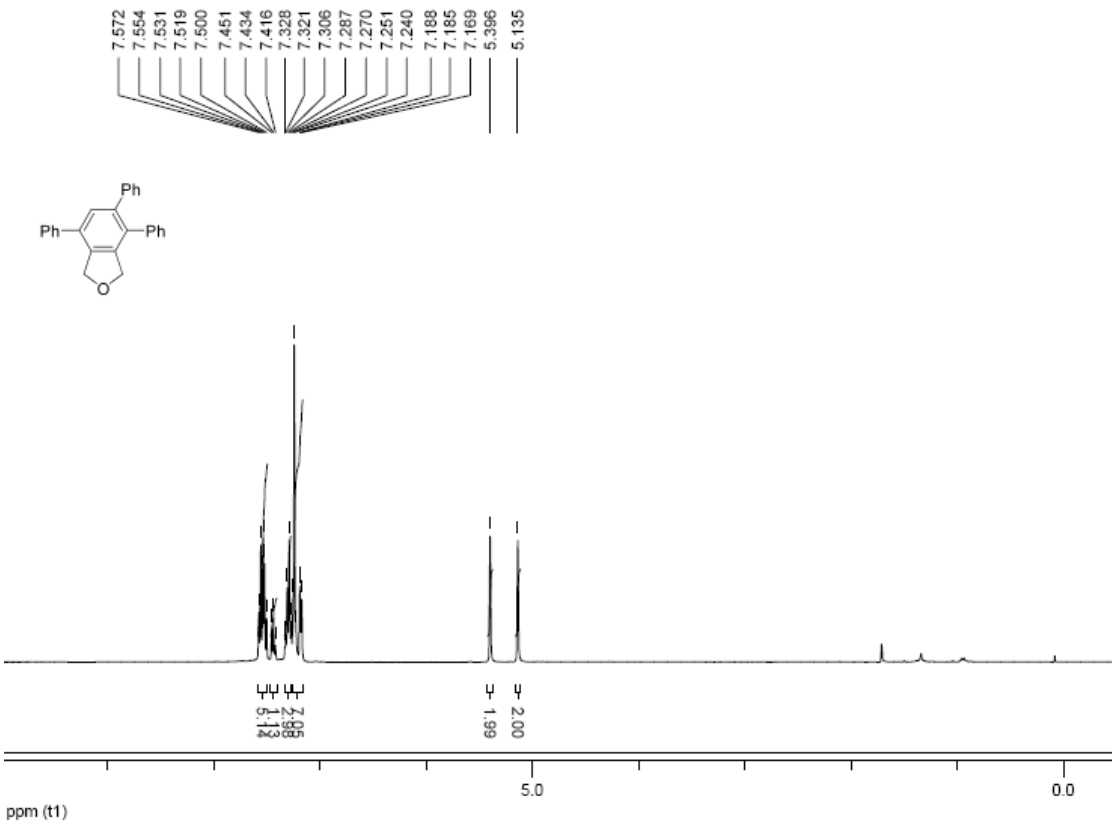
122.908
121.311
77.317
77.000
76.682
53.710
53.531
21.494
Ph
N
Ts
200
150
100
50
0
ppm (t1)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7.554
7.531
7.519
7.500
7.451
7.434
7.416
7.328
7.321
7.306
7.287
7.270
7.251
7.240
7.188
7.185
7.169
5.396
5.135
Ph
Ph
Ph
O
5.14
5.13
2.88
2.02
1.99
2.00
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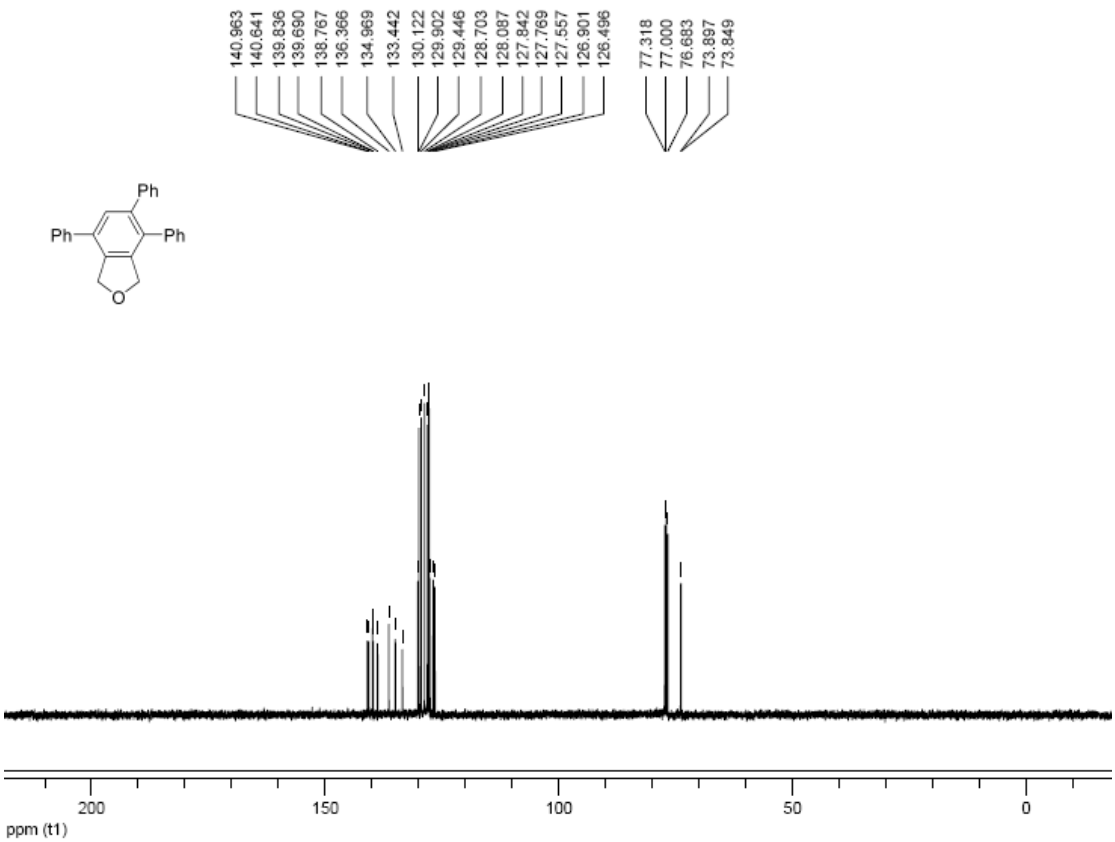
ppm (t1)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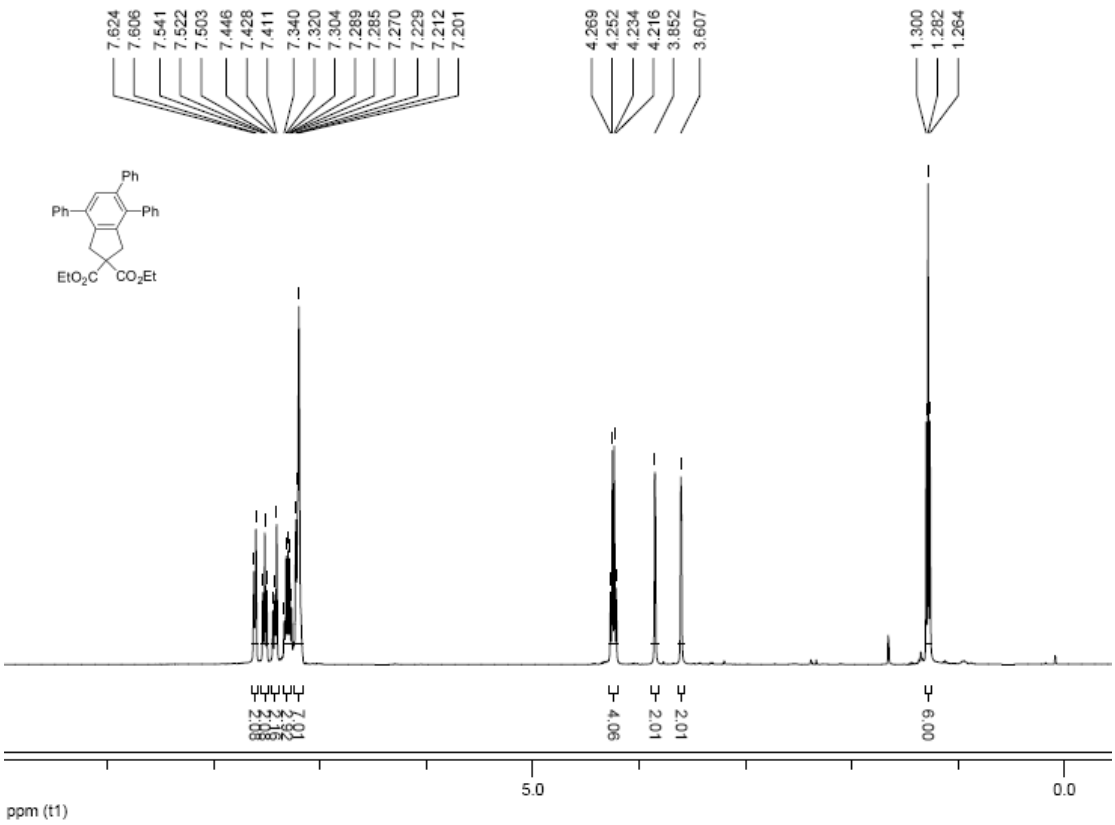
140.641
139.836
139.690
138.767
136.366
134.969
133.442
130.122
129.902
129.446
128.703
128.087
127.842
127.769
127.557
126.901
126.496
77.318
77.000
76.683
73.897
73.849
Ph
Ph
Ph
O
200
150
100
50
0
ppm (t1)


7.624
7.606
7.541
7.522
7.503
7.446
7.428
7.411
7.340
7.320
7.304
7.289
7.285
7.270
7.229
7.212
7.201
4.269
4.252
4.234
4.216
3.852
3.607
1.300
1.282
1.264
Ph
Ph
Ph
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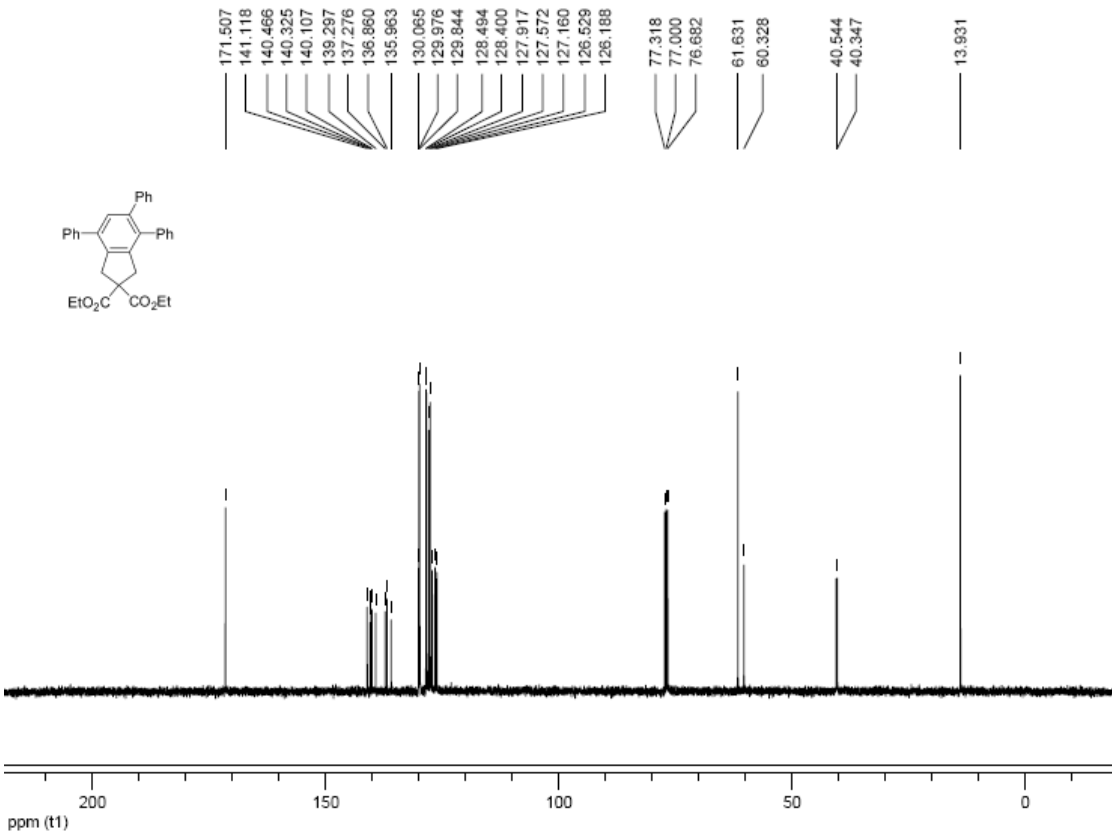
CO2Et
7.01
4.06
2.01
2.01
6.00
5.0
0.0
ppm (t1)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140.466
140.325
140.107
139.297
137.276
136.860
135.963
130.065
129.976
129.844
128.494
128.400
127.917
127.572
127.160
126.529
126.188
77.318
77.000
76.682
61.631
60.328
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13.931
Ph
Ph
Ph
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CO2Et
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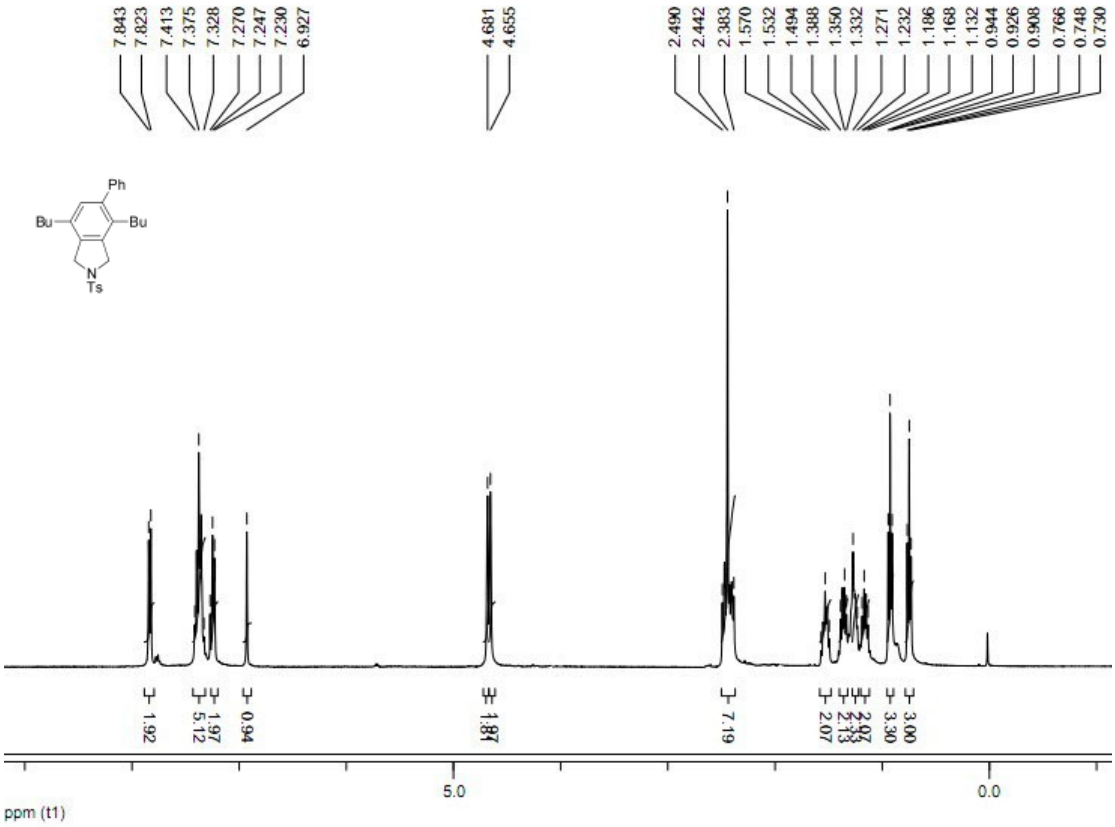
150
100
50
0
ppm (t1)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7.413
7.375
7.328
7.270
7.247
7.230
6.927
4.681
4.655
2.490
2.442
2.383
1.570
1.532
1.494
1.388
1.350
1.332
1.271
1.232
1.186
1.168
1.132
0.944
0.926
0.908
0.766
0.748
0.730
Ph
Bu
Bu
N
Ts
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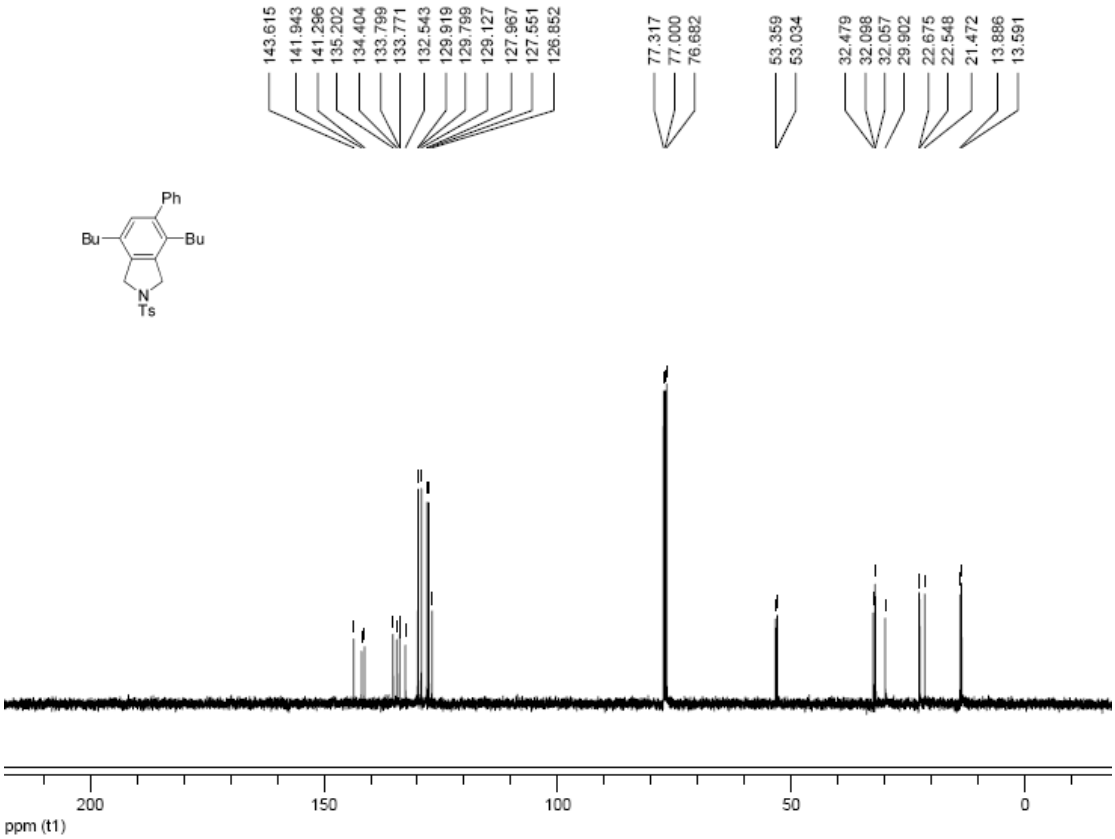
5.12
1.97
0.94
1.87
7.19
2.07
3.30
3.00
5.0
0.0
ppm (t1)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134.404
133.799
133.771
132.543
129.919
129.799
129.127
127.967
127.551
126.852
77.317
77.000
76.682
53.359
53.034
32.479
32.098
32.057
29.902
22.675
22.548
21.472
13.886
13.591
Ph
Bu
Bu
N
Ts
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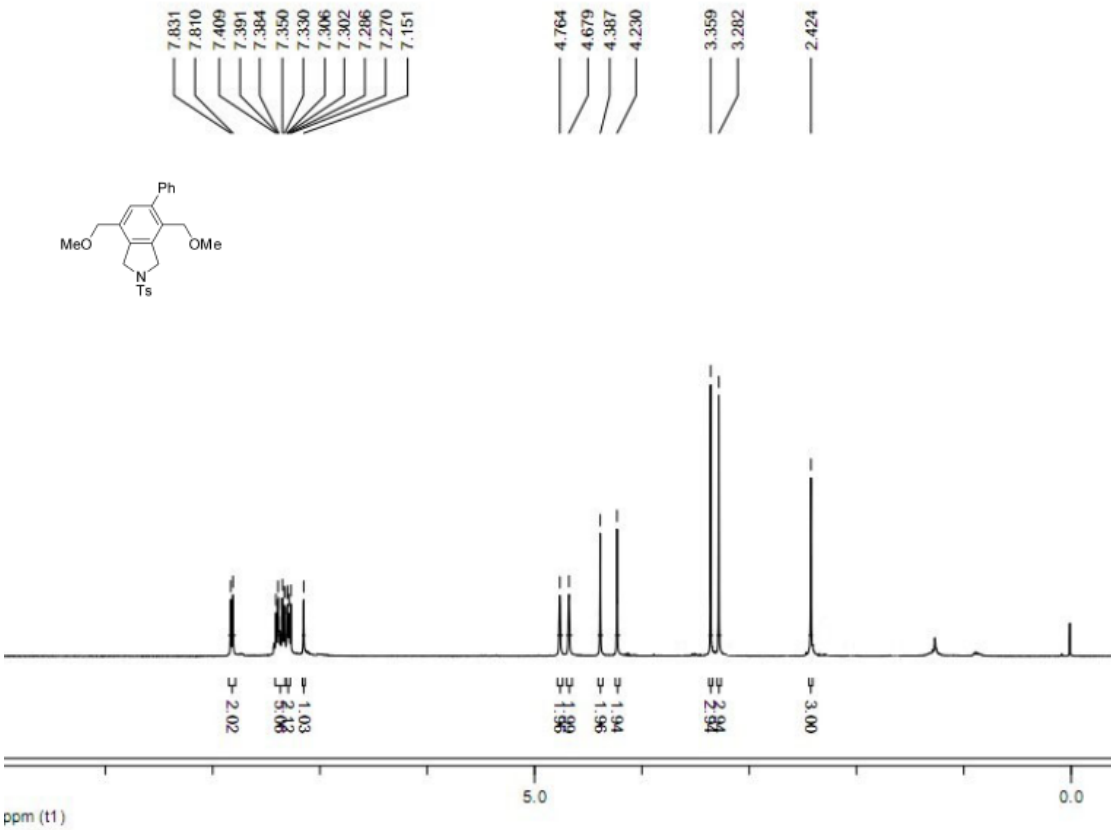
150
100
50
0
ppm (t1)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7.391
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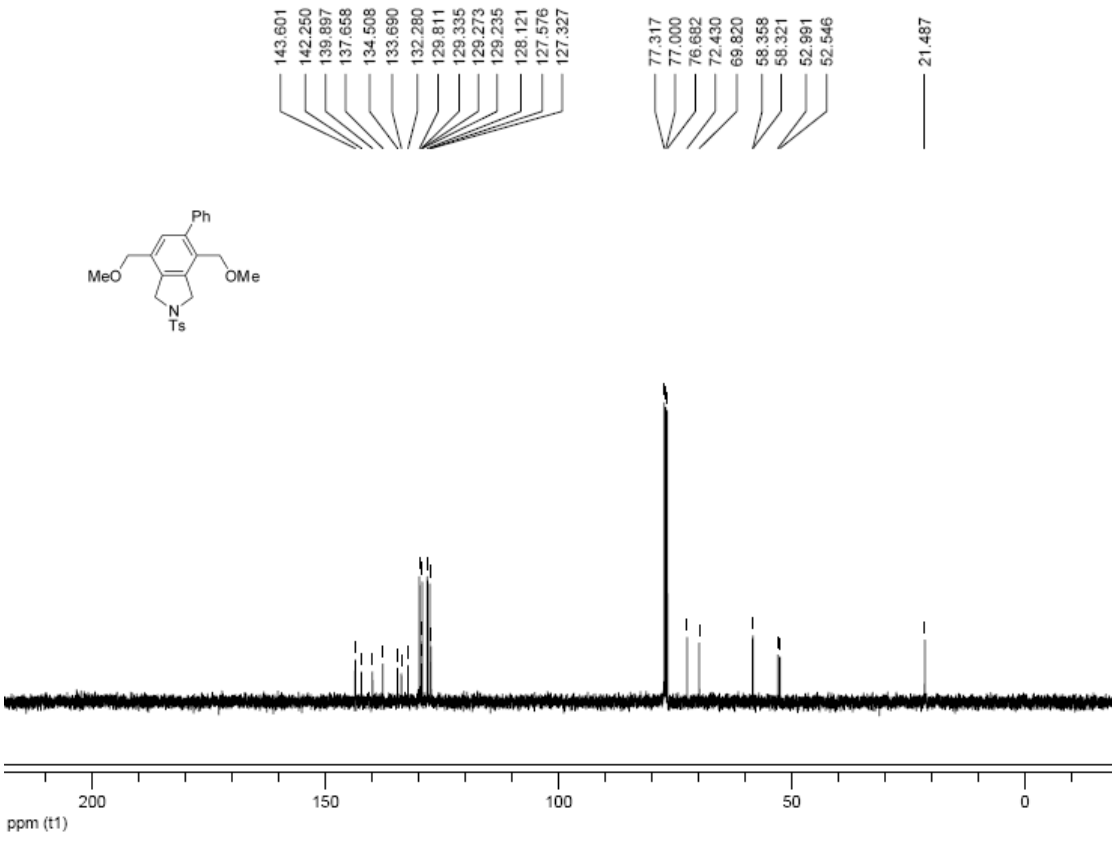
7.350
7.330
7.306
7.302
7.286
7.270
7.151
4.764
4.679
4.387
4.230
3.359
3.282
2.424
Ph
MeO
OMe
N
Ts
2.02
5.06
2.03
1.96
1.94
2.04
3.00
5.0
0.0
ppm (t1)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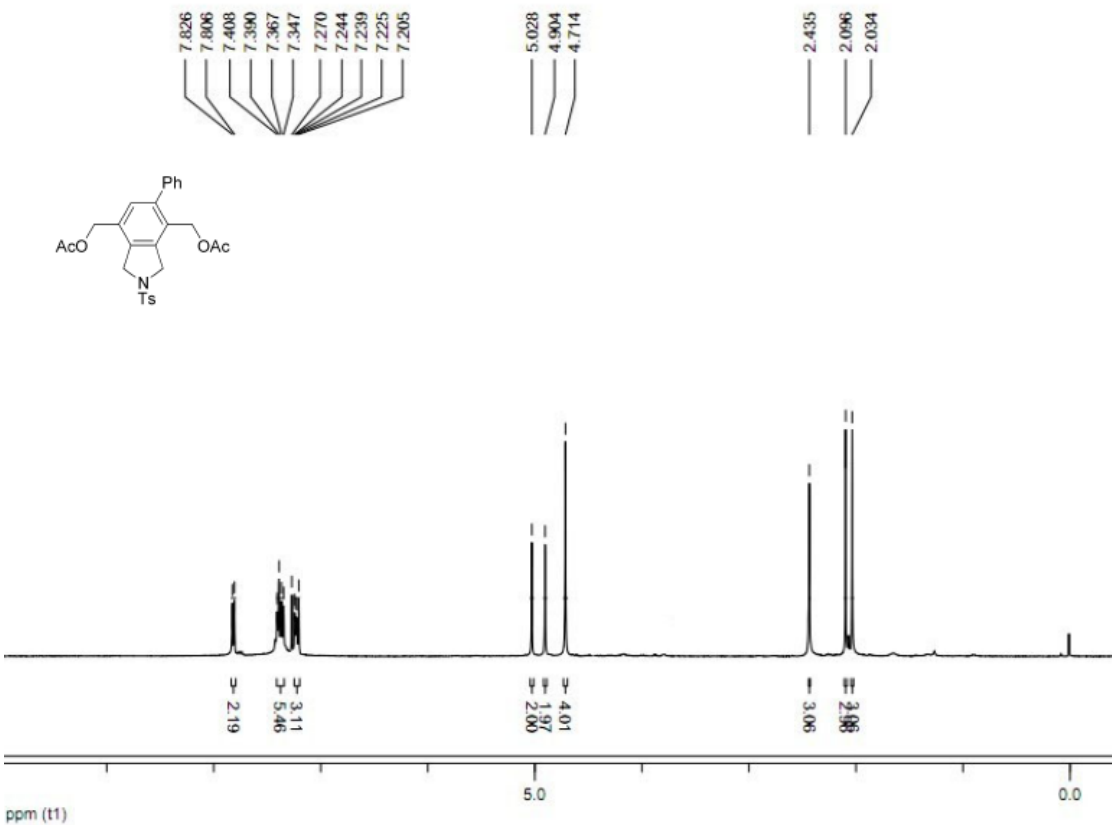
137.658
134.508
133.690
132.280
129.811
129.335
129.273
129.235
128.121
127.576
127.327
77.317
77.000
76.682
72.430
69.820
58.358
58.321
52.991
52.546
21.487
Ph
MeO
OMe
N
Ts
200
150
100
50
0
ppm (t1)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7.367
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7.244
7.239
7.225
7.205
5.028
4.904
4.714
2.435
2.096
2.034
Ph
AcO
OAc
N
Ts
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2.00
1.97
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3.06
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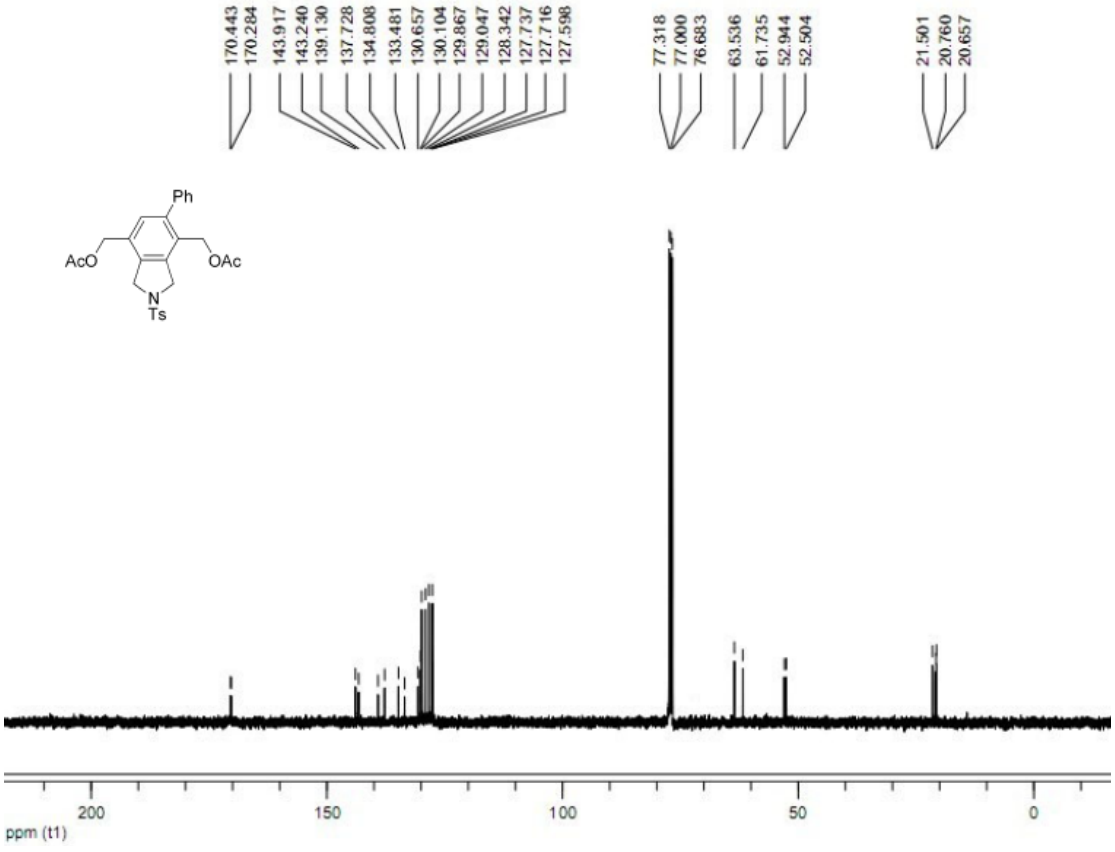
ppm (t1)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143.240
139.130
137.728
134.808
133.481
130.657
130.104
129.867
129.047
128.342
127.737
127.716
127.598
77.318
77.000
76.683
63.536
61.735
52.944
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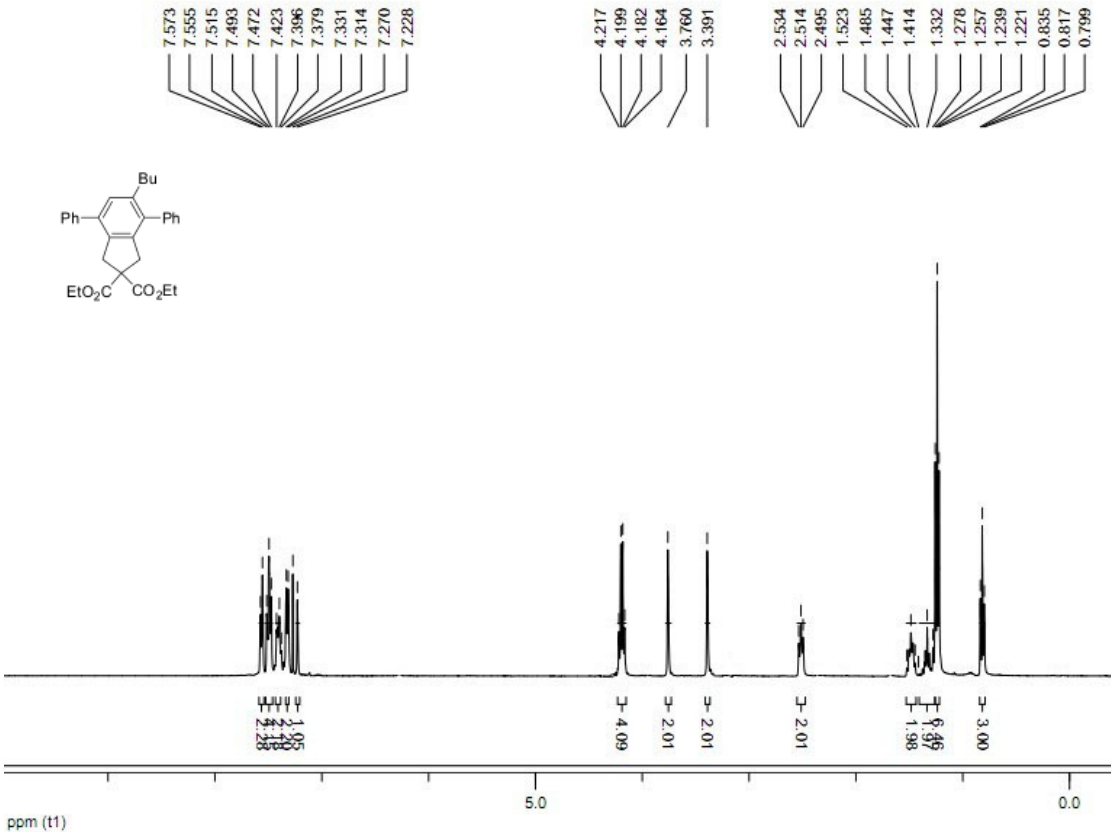
20.760
20.657
Ph
AcO
OAc
N
Ts
200
150
100
50
0
ppm (t1)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7.472
7.423
7.396
7.379
7.331
7.314
7.270
7.228
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4.199
4.182
4.164
3.760
3.391
2.534
2.514
2.495
1.523
1.485
1.447
1.414
1.332
1.278
1.257
1.239
1.221
0.835
0.817
0.799
Bu
Ph
Ph
EtO2C
CO2Et
4.09
2.01
2.01
2.01
1.98
3.00
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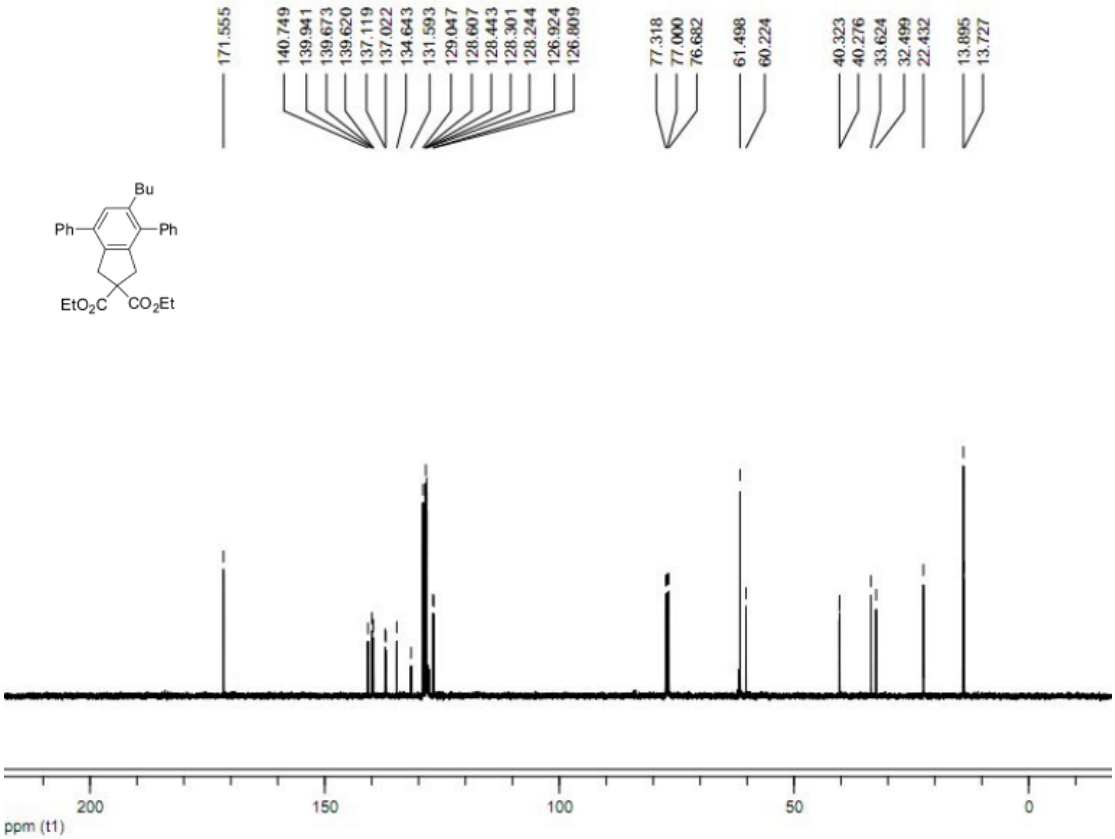
ppm (t1)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134.643
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129.047
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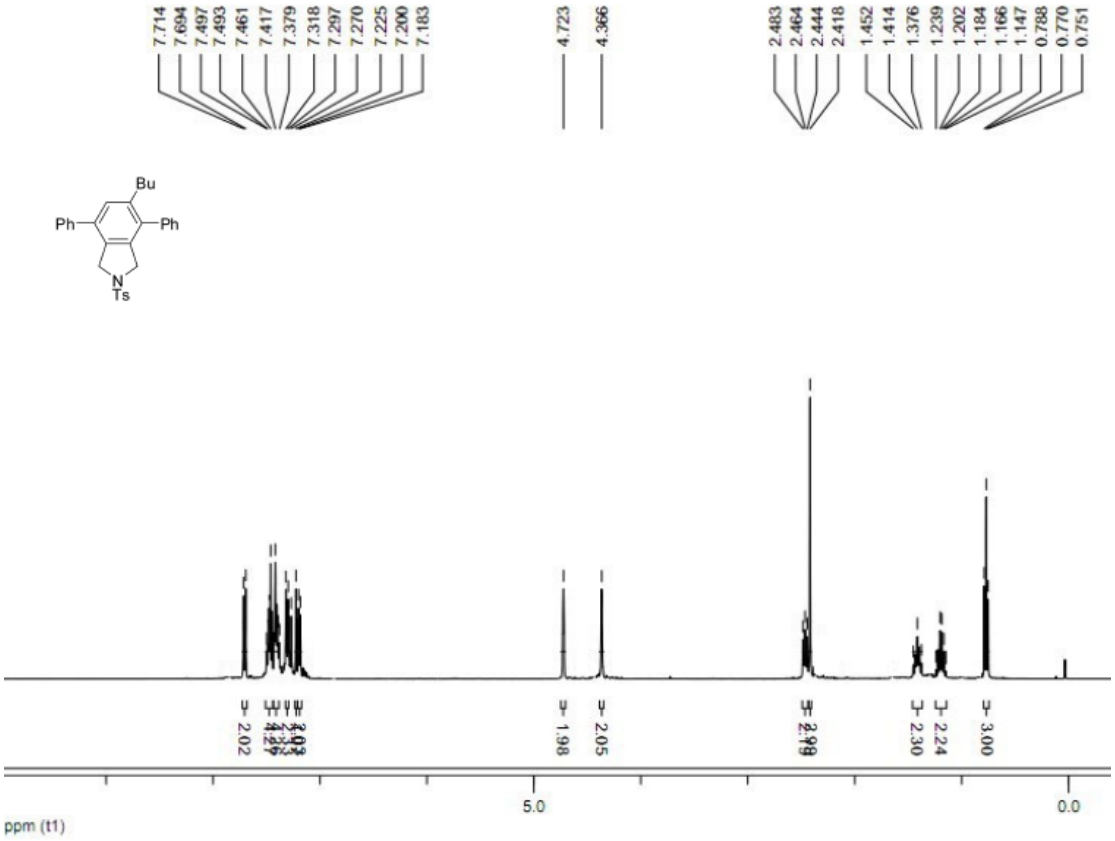
128.443
128.301
128.244
126.924
126.809
77.318
77.000
76.682
61.498
60.224
40.323
40.276
33.624
32.499
22.432
13.895
13.727
Bu
Ph
Ph
EtO2C
CO2Et
200
150
100
50
0
ppm (t1)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7.379
7.318
7.297
7.270
7.225
7.200
7.183
4.723
4.366
2.483
2.464
2.444
2.418
1.452
1.414
1.376
1.239
1.202
1.184
1.166
1.147
0.788
0.770
0.751
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Ph
Ph
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Ts
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2.05
2.30
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3.00
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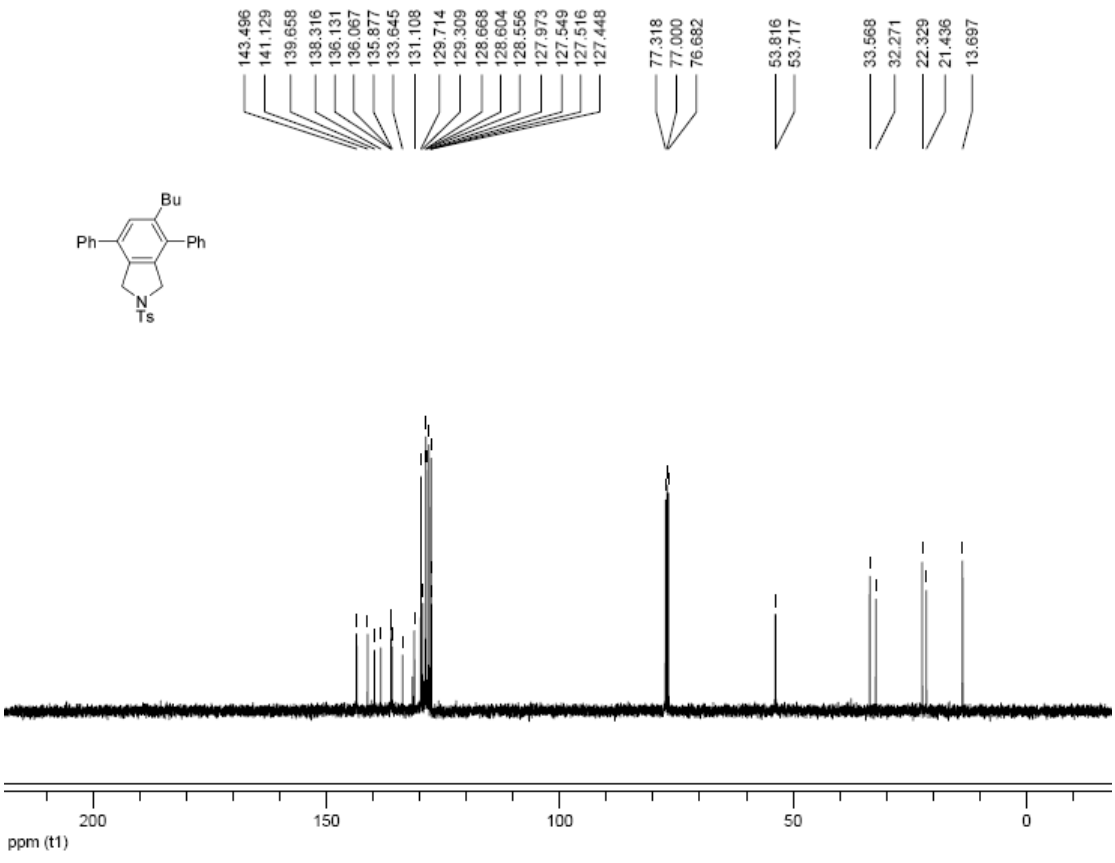
ppm (t1)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128.604
128.556
127.973
127.549
127.516
127.448
77.318
77.000
76.682
53.816
53.717
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32.271
22.329
21.436
13.697
Bu
Ph
Ph
N
Ts
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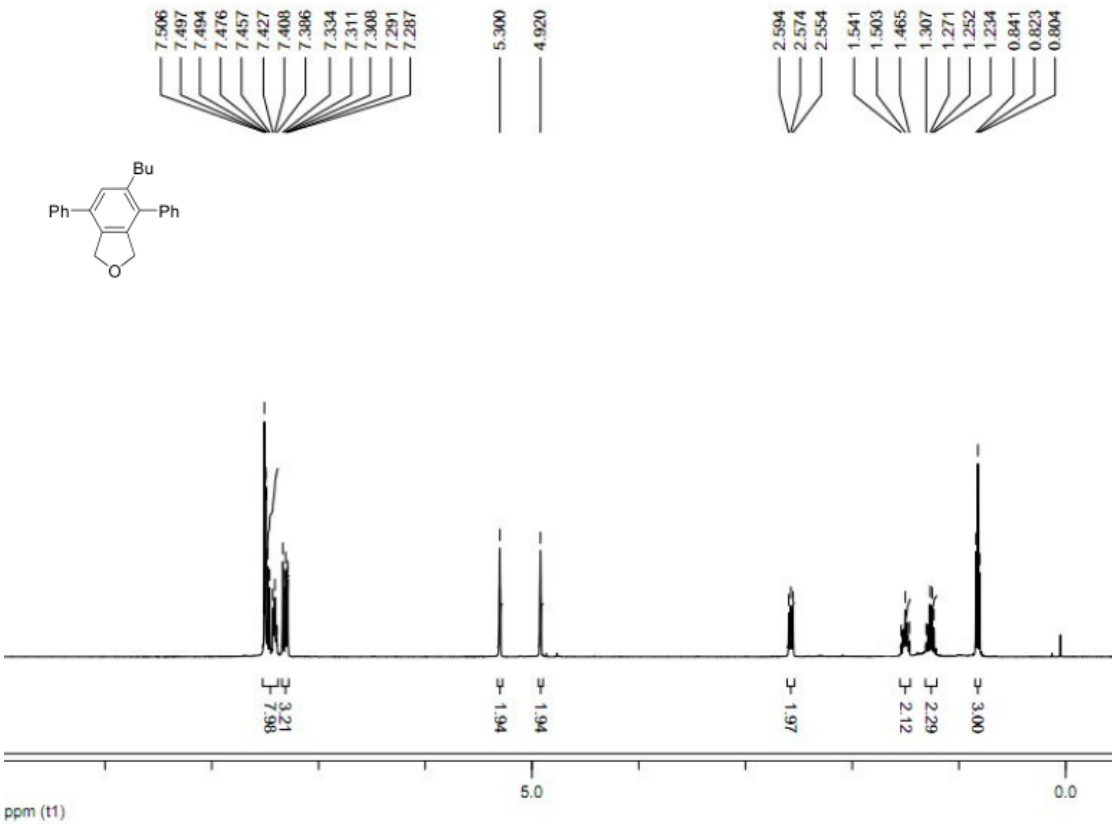
50
0
ppm (t1)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7.287
5.300
4.920
2.594
2.574
2.554
1.541
1.503
1.465
1.307
1.271
1.252
1.234
0.841
0.823
0.804
Bu
Ph
Ph
O
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1.97
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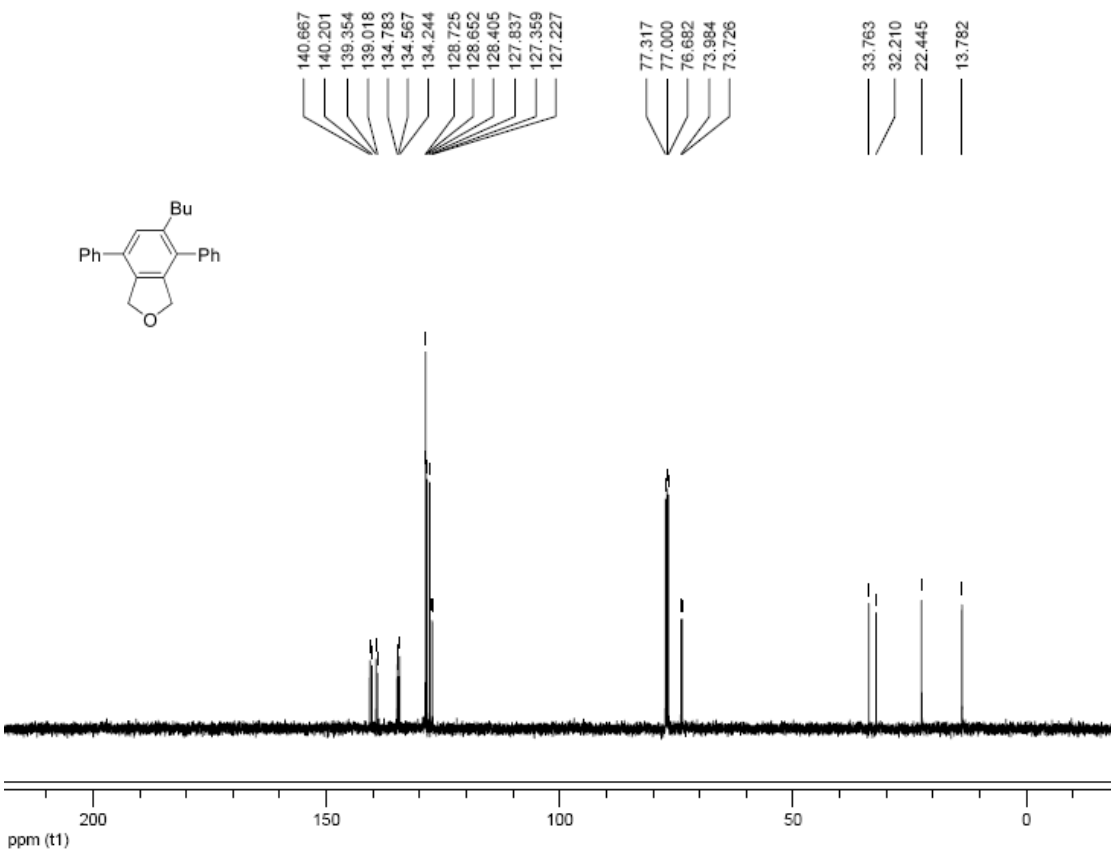
ppm (t1)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134.567
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127.359
127.227
77.317
77.000
76.682
73.984
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32.210
22.445
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Ph
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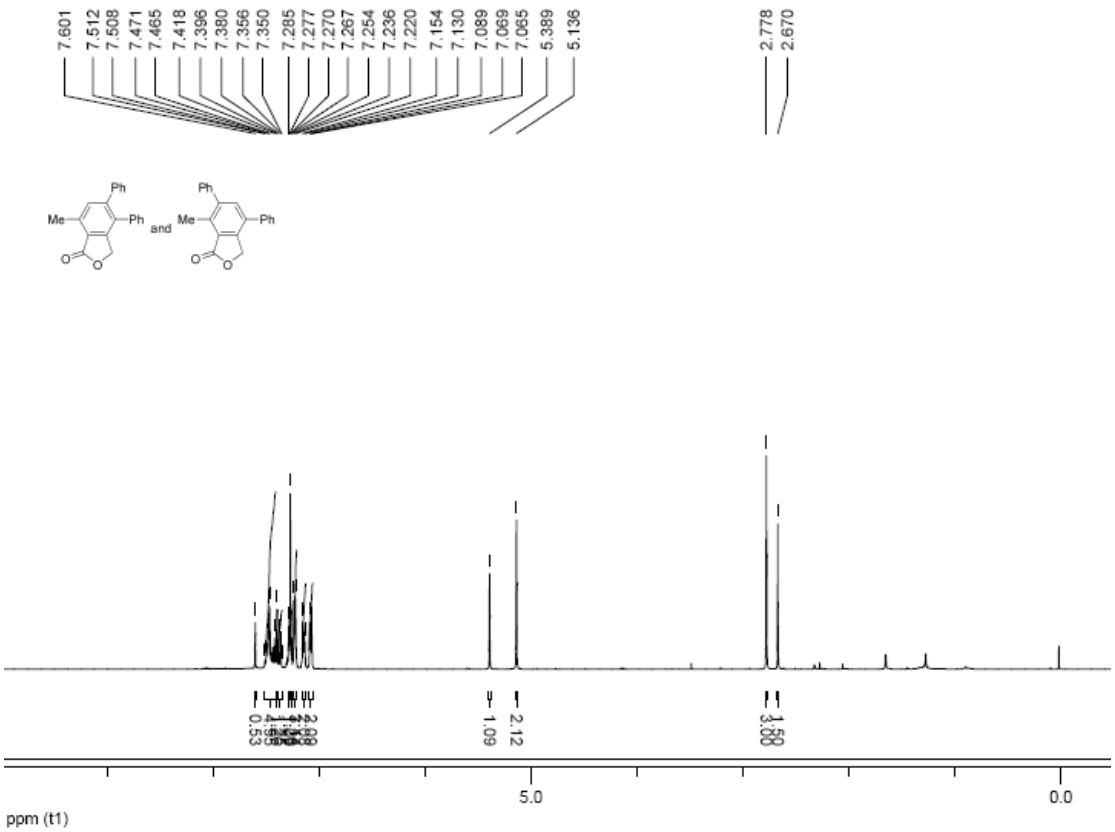
ppm (t1)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7.285
7.277
7.270
7.267
7.254
7.236
7.220
7.154
7.130
7.089
7.069
7.065
5.389
5.136
2.778
2.670
Ph
Me
Ph
and
Me
Ph
O
O
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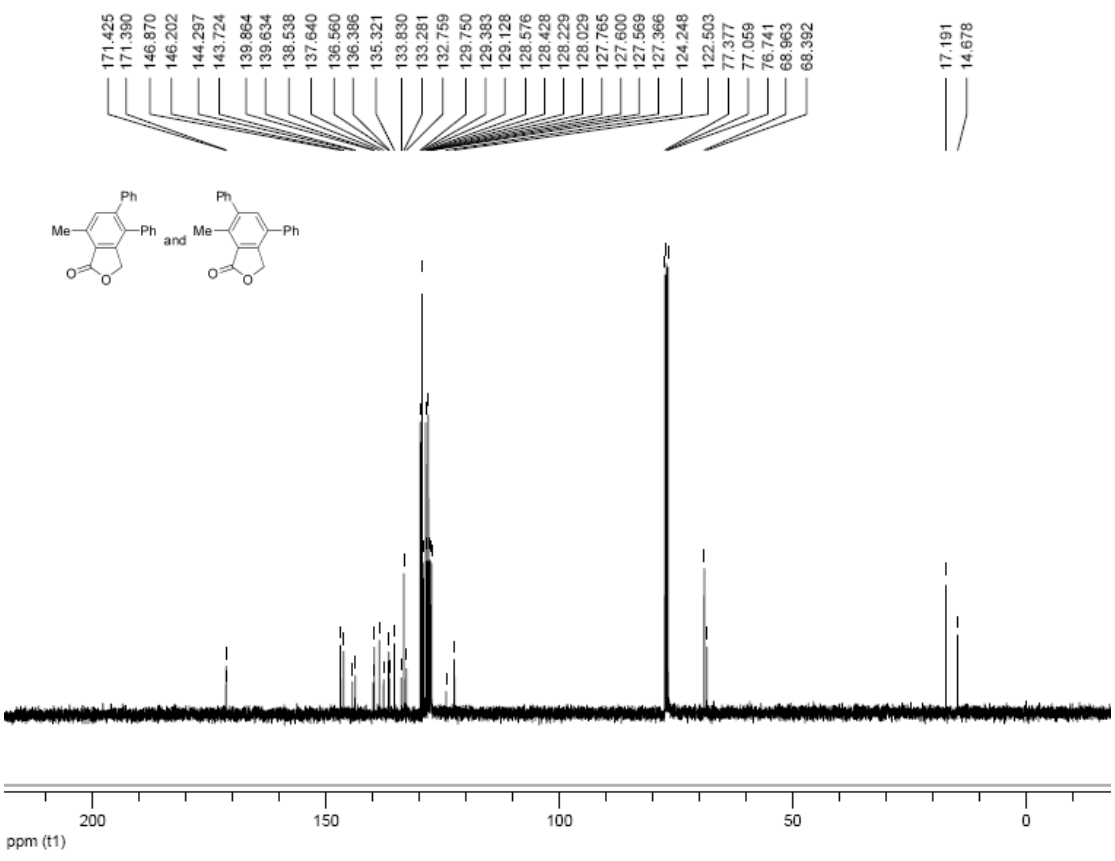
1.09
2.12
3.00
1.50
5.0
0.0
ppm (t1)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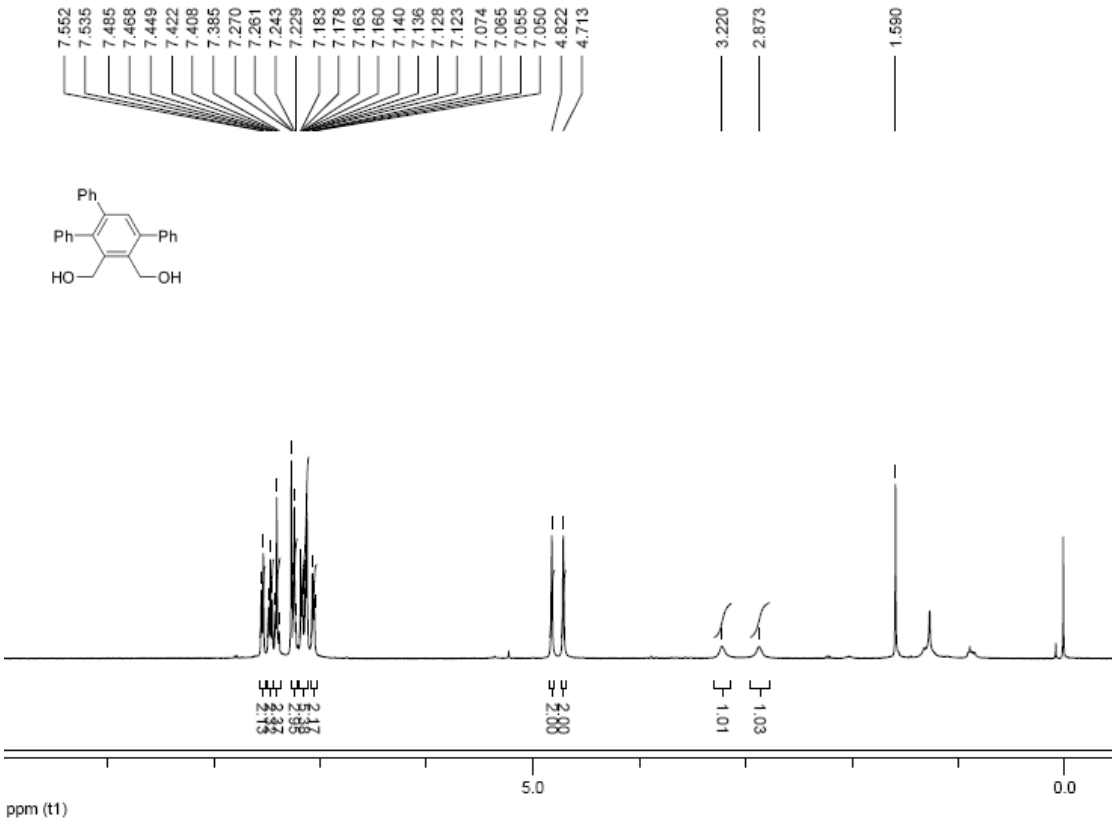
135.321
133.830
133.281
132.759
129.750
129.383
129.128
128.576
128.428
128.229
128.029
127.765
127.600
127.569
127.366
124.248
122.503
77.377
77.059
76.741
68.963
68.392
17.191
14.678
Ph
Me
Ph
and
Me
Ph
O
O
200
150
100
50
0
ppm (t1)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7.183
7.178
7.163
7.160
7.140
7.136
7.128
7.123
7.074
7.065
7.055
7.050
4.822
4.713
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Ph
Ph
Ph
HO
OH
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1.03
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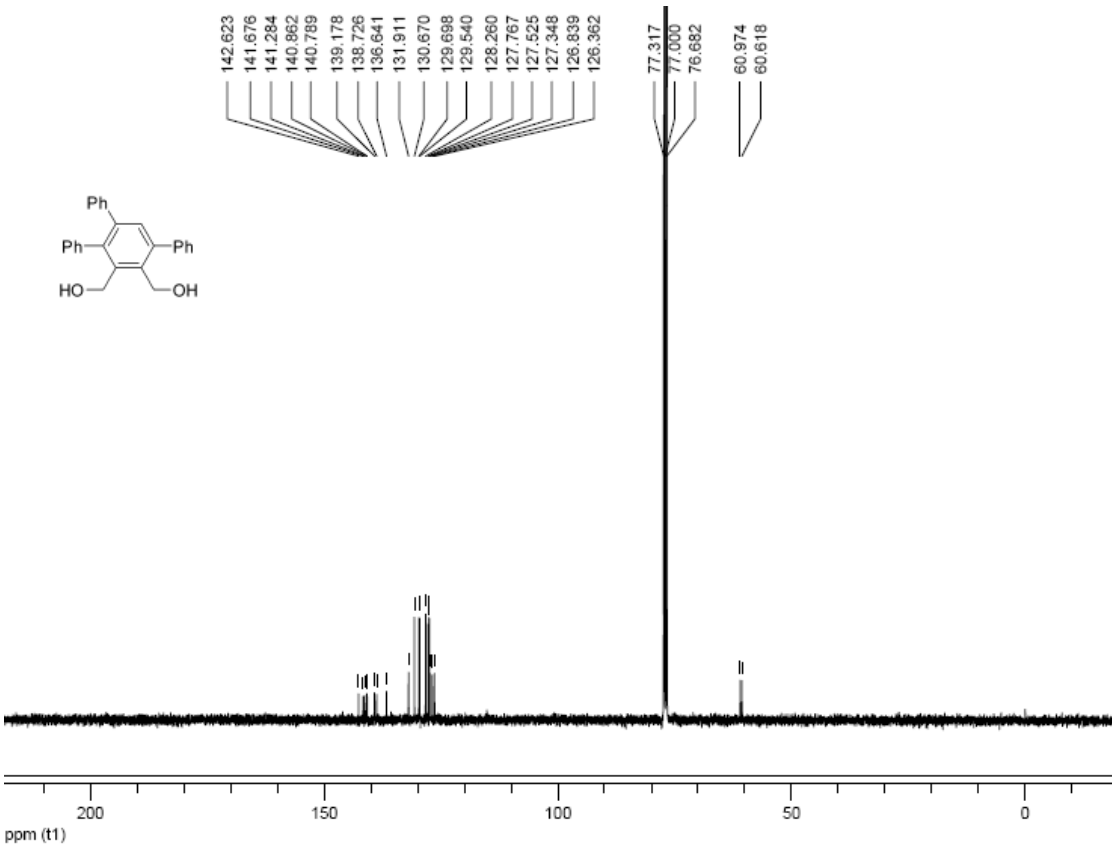
ppm (t1)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128.260
127.767
127.525
127.348
126.839
126.362
77.317
77.000
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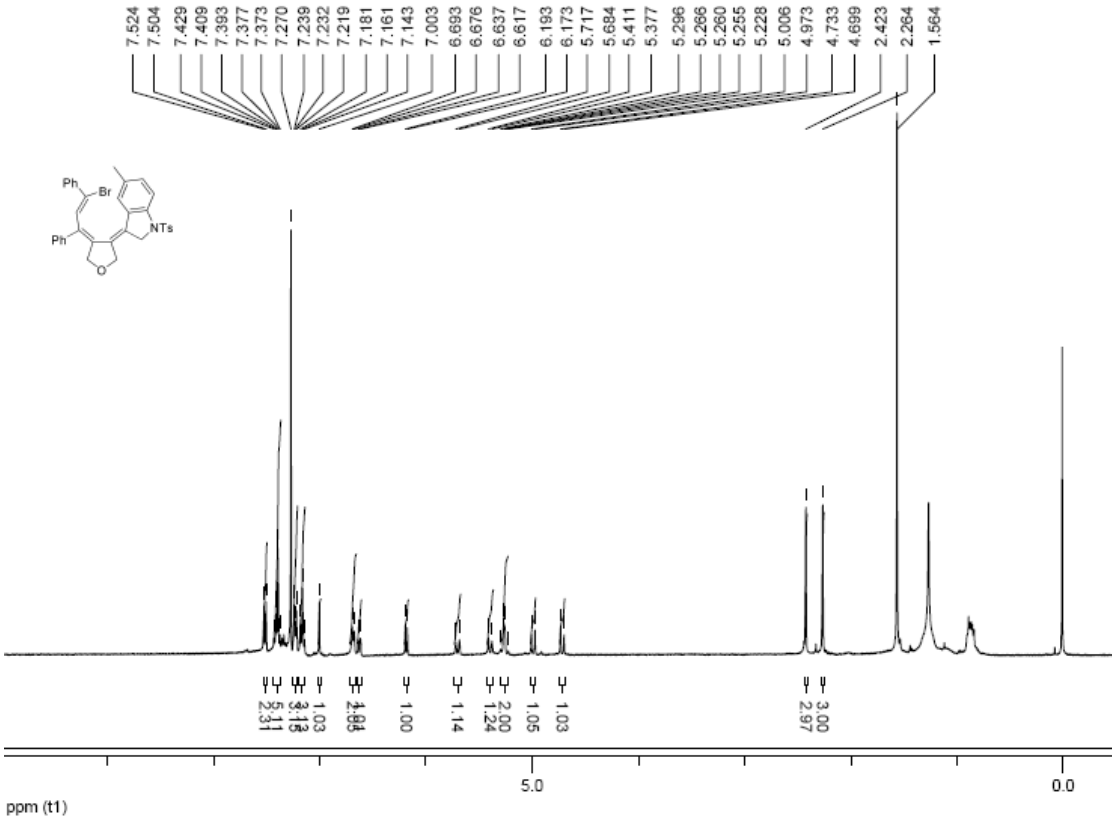
60.974
60.618
Ph
Ph
Ph
HO
OH
200
150
100
50
0
ppm (t1)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7.377
7.373
7.270
7.239
7.232
7.219
7.181
7.161
7.143
7.003
6.693
6.676
6.637
6.617
6.193
6.173
5.717
5.684
5.411
5.377
5.296
5.266
5.260
5.255
5.228
5.006
4.973
4.733
4.699
2.423
2.264
1.564
Ph
Br
Ph
NTs
O
2.31
5.11
3.18
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1.14
2.24
1.05
1.03
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3.00
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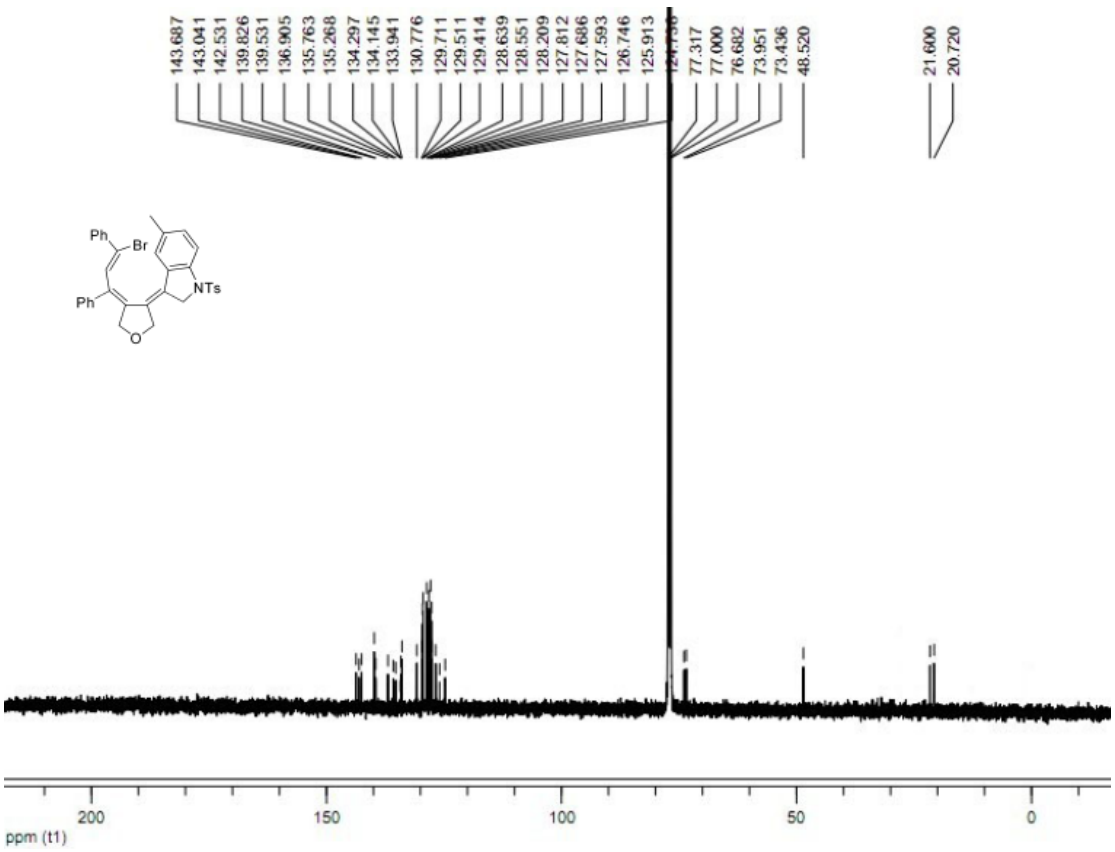
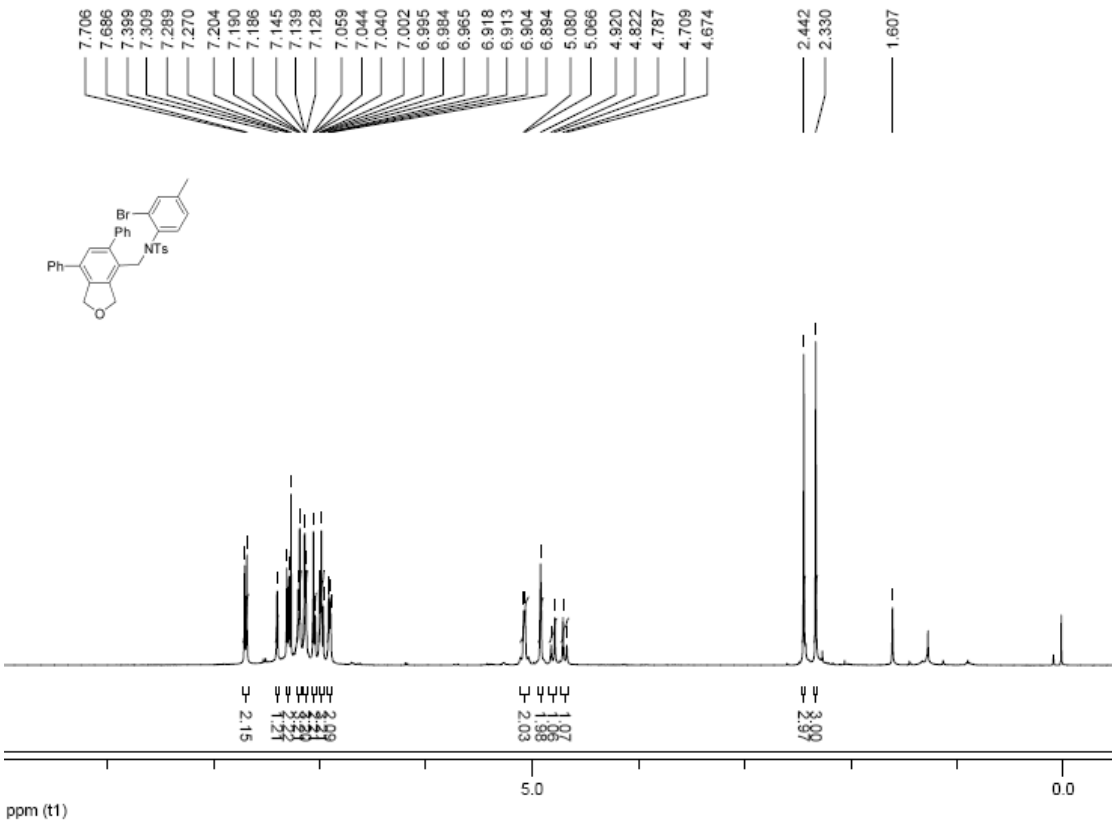
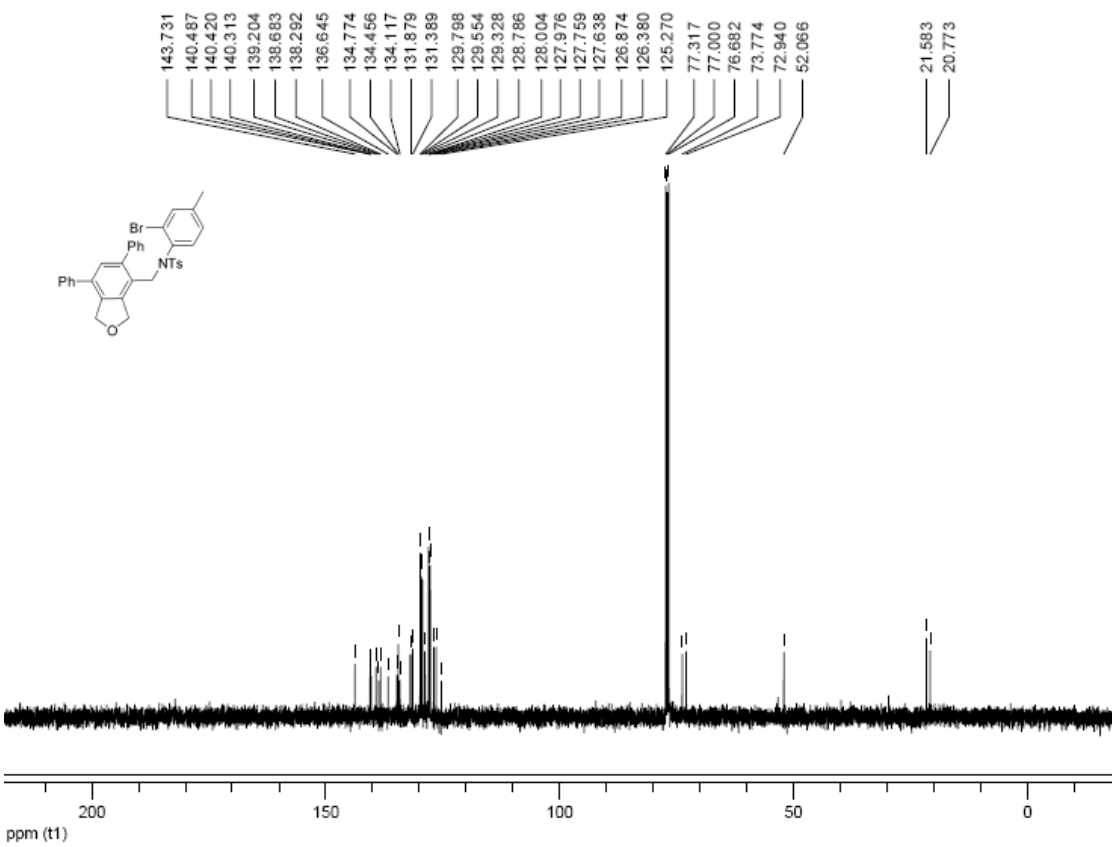
0.0
ppm (t1)


143.687
143.041
142.531
139.826
139.531
136.905
135.763
135.268
134.297
134.145
133.941
130.776
129.711
129.511
129.414
128.639
128.551
128.209
127.812
127.686
127.593
126.746
125.913
77.317
77.000
76.682
73.951
73.436
48.520
21.600
20.720
Ph
Br
Ph
NTs
O
200
150
100
50
0
ppm (t1)


7.706
7.686
7.399
7.309
7.289
7.270
7.204
7.190
7.186
7.145
7.139
7.128
7.059
7.044
7.040
7.002
6.995
6.984
6.965
6.918
6.913
6.904
6.894
5.080
5.066
4.920
4.822
4.787
4.709
4.674
2.442
2.330
1.607
Br
Ph
NTs
Ph
O
2.15
2.03
3.09
5.0
0.0
ppm (t1)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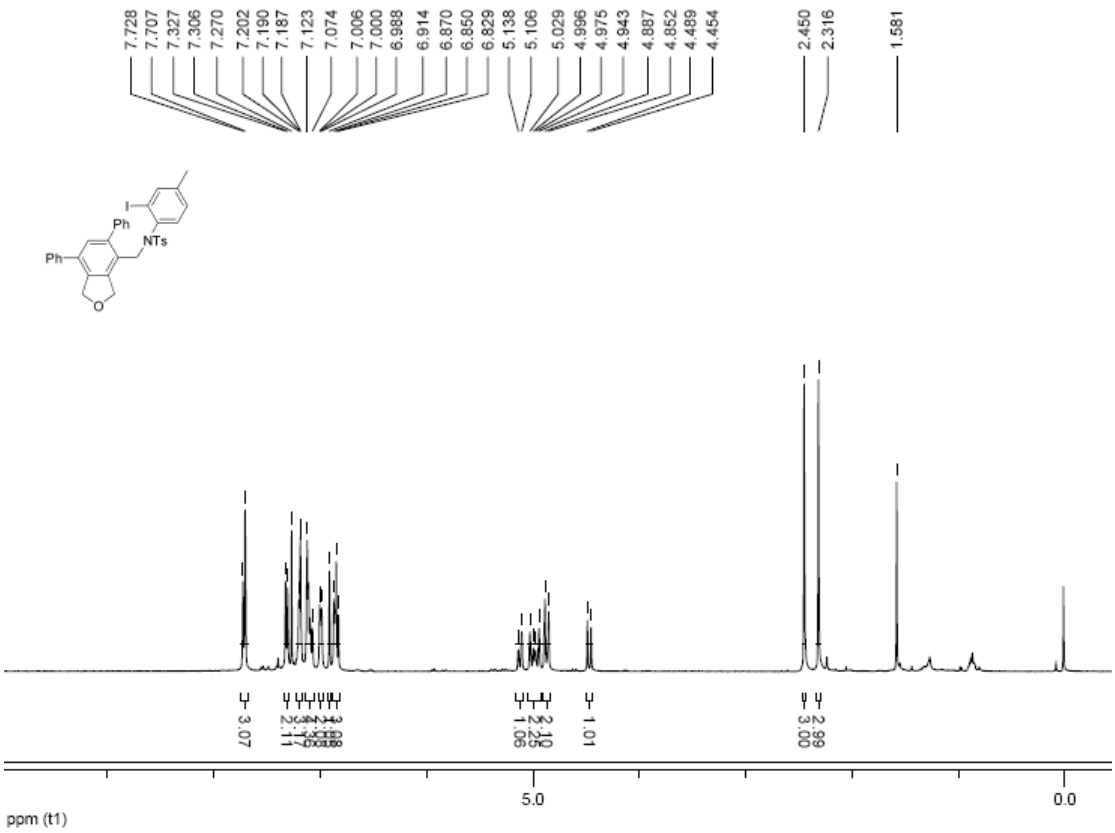
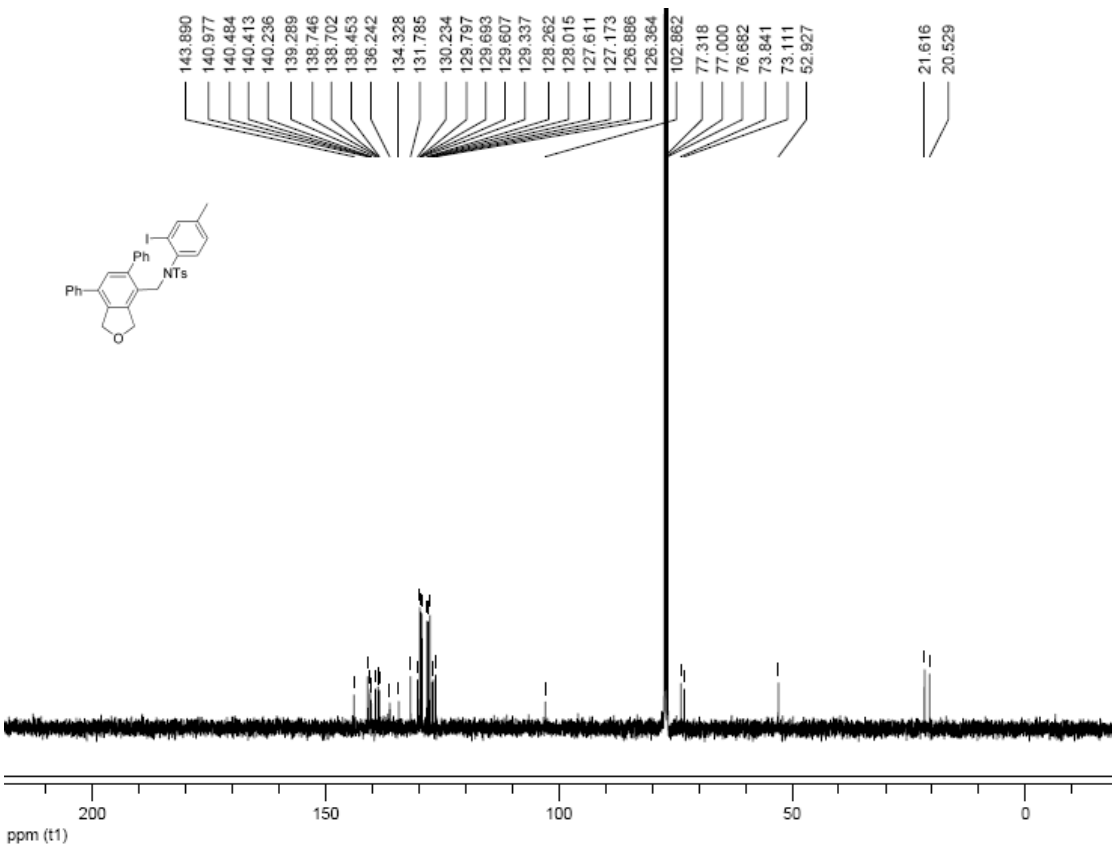
140.487
140.420
140.313
139.204
138.683
138.292
136.645
134.774
134.456
134.117
131.879
131.389
129.798
129.554
129.328
128.786
128.004
127.976
127.759
127.638
126.874
126.380
125.270
77.317
77.000
76.682
73.774
72.940
52.066
21.583
20.773
Br
Ph
NTs
Ph
O
200
150
100
50
0
ppm (t1)


7.728
7.707
7.327
7.306
7.270
7.202
7.190
7.187
7.123
7.074
7.006
7.000
6.988
6.914
6.870
6.850
6.829
5.138
5.106
5.029
4.996
4.975
4.943
4.887
4.852
4.489
4.454
2.450
2.316
1.581
I
Ph
NTs
Ph
O
3.07
2.11
1.06
1.01
3.00
2.99
5.0
0.0
ppm (t1)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138.453
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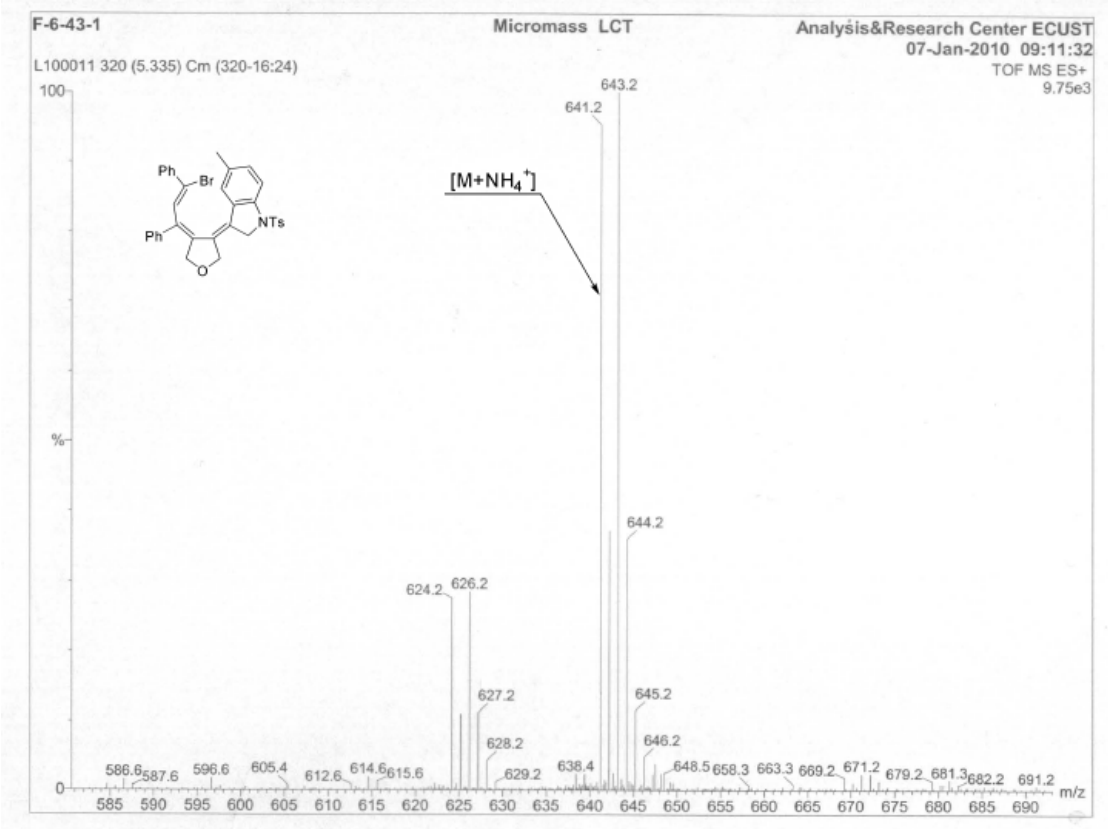
130.234
129.797
129.693
129.607
129.337
128.262
128.015
127.611
127.173
126.886
126.364
102.862
77.318
77.000
76.682
73.841
73.111
52.927
21.616
20.529
I
Ph
NTs
Ph
O
200
150
100
50
0
ppm (t1)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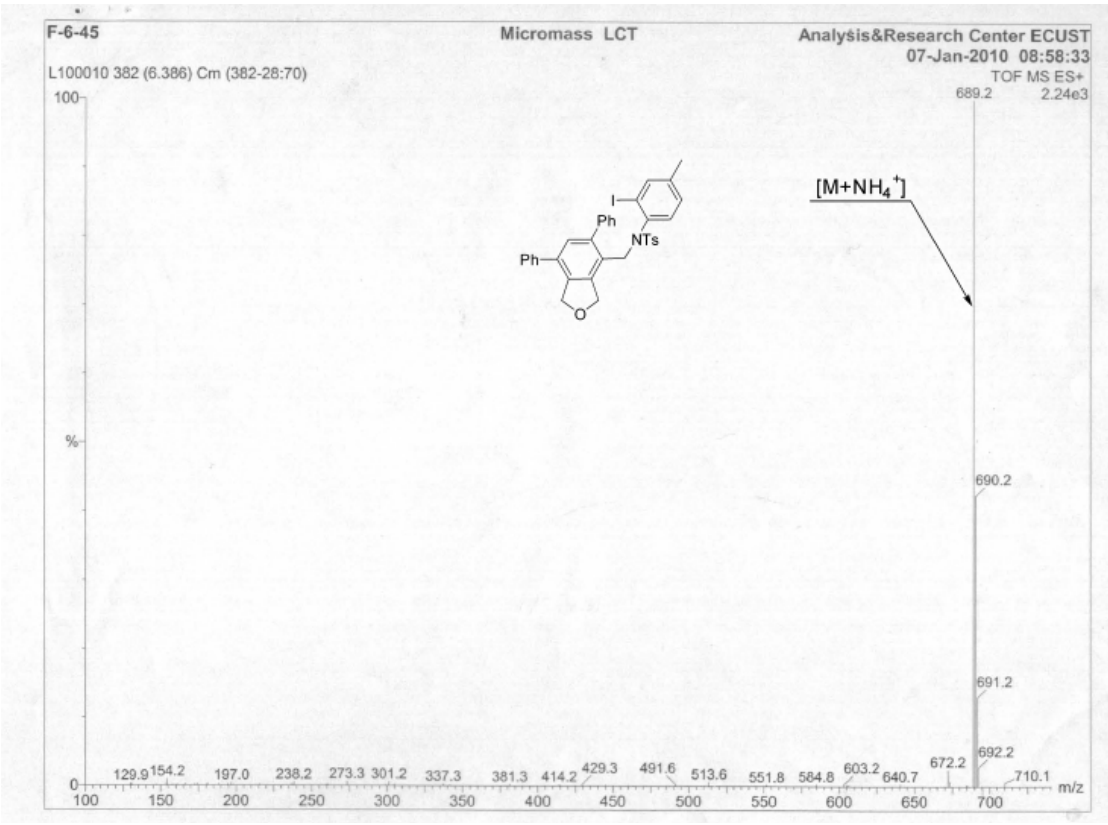
Analysis&Research Center ECUST
07-Jan-2010 09:11:32
L100011 320 (5.335) Cm (320-16:24)
TOF MS ES+
9.75e3
643.2
641.2
[M+NH4+]
Ph
Br
NTs
Ph
O
644.2
624.2
626.2
627.2
645.2
628.2
646.2
586.6
587.6
596.6
605.4
612.6
614.6
615.6
629.2
638.4
648.5
658.3
663.3
669.2
671.2
679.2
681.3
682.2
691.2
%
m/z


F-6-45
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Analysis&Research Center ECUST
07-Jan-2010 08:58:33
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TOF MS ES+
689.2
2.24e3
[M+NH4+]
I
Ph
NTs
Ph
O
690.2
691.2
692.2
129.9
154.2
197.0
238.2
273.3
301.2
337.3
381.3
414.2
429.3
491.6
513.6
551.8
584.8
603.2
640.7
672.2
710.1
%
m/z