

One-pot Synthesis of 2,4-Disubstituted Indoles from *N*-Tosyl-2,3-dichloroaniline Using Palladium-Dihydroxyterphenylphosphine Catalyst

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1. General method and materials

General. All reactions were performed under argon atmosphere. Reactions were monitored by TLC on Merck silica gel 60 F254 plates visualized by UV lamp at 254 nm. Column chromatography was performed on Kanto Chemicals silica gel 60N (neutral, spherical) and preparative TLC was performed on Merck silica gel 60 F254 0.5 mm plates. NMR spectra were measured on a JEOL AL-400 NMR spectrometer at 400 MHz for ^1H spectra and 100 MHz for ^{13}C spectra, and for ^1H NMR, tetramethylsilane (TMS) ($\delta = 0$) in CDCl_3 served as an internal standard. For ^{13}C NMR, CDCl_3 ($\delta = 77.0$) served as an internal standard. Infrared spectra were measured on a SHIMADZU IR Prestige-21 spectrometer (ATR). High-resolution mass spectra (HRMS) were measured on a Bruker MicrOTOF time-of-flight mass spectrometer (ESI) and a JEOL JMS-T100TD time-of-flight mass spectrometer (DART). Melting points were measured using Stanford Research Systems MPA 100 OptiMelt and uncorrected.

Materials. Ligands **1b**• HBF_4 and **1c** were prepared according to the reported procedure.¹ 2-Chloroaniline derivatives **2a**,² **2c**,³ **2d**,⁴ and **2f**⁵ were prepared according to the reported procedure. **2e**⁶ was prepared by using the preparation method of *N*-(4-chlorophenyl)-2-nitrobenzenesulfonamide.⁷ Dichloroaniline derivatives **7**,⁸ *N*-(2,4-dichlorophenyl)-4-methylbenzenesulfonamide **32**,⁹ and *N*-(2,5-dichlorophenyl)-4-methylbenzenesulfonamide **33** were prepared by using the preparation method of **2a**.^{2a} Other reagents and anhydrous solvents were purchased from commercial suppliers and used without further purification.

2. Optimization of reaction conditions for indole synthesis

Table S1. Ligand Screening.

Reaction scheme for Table S1:

entry	ligand	time (h)	yield (%) ^a	
			4a	5a
1	1b ·HBF ₄	19	trace	38
2	1a ·HBF ₄	17	8	28
3	PCy ₃	21	nd ^b	nd ^b
4	P(<i>t</i> -Bu) ₃ ·HBF ₄	21	nd ^b	nd ^b
5	XPhos	19	nd ^b	nd ^b

^aIsolated yield. ^bNot detected.

Table S2. Optimization of Reaction Conditions.

Reaction scheme for Table S2:

entry	solvent	base	yield (%) ^a
1	toluene	<i>t</i> -BuOLi	68
2	toluene	Li ₃ PO ₄	<5 ^b
3	toluene	LiOH	<5 ^b
4	toluene	K ₃ PO ₄	nd ^c
5	toluene	K ₂ CO ₃	nd ^c
6	toluene	<i>t</i> -BuOK	nd ^c
7	DMF	<i>t</i> -BuOLi	nd ^{cd}
8 ^d	xylene	<i>t</i> -BuOLi	74
9 ^d	mesitylene	<i>t</i> -BuOLi	87

^aIsolated yield. ^bNMR yield. ^cNot detected. ^d140 °C, 2 h and H₂O reflux 1 h.

3. Typical experimental procedure for the synthesis of *N*-tosylated chloroanilines^{2a}

p-toluenesulfonyl chloride (4.56 g, 24 mmol) was added portionwise to 2-chloroanilines (20 mmol) and pyridine (10 mL, 120 mmol) dissolved in dichloromethane (66 mL). The reaction mixture was stirred at rt for 22 h. 1 M aqueous HCl (50 mL) was added and the organic phase was washed with aqueous HCl (50 mL × 3). Organic layer was dried over anhydrous Na₂SO₄ and evaporated in vacuo to afford the residue. The residue was purified by silica gel column chromatography or recrystallization to give the desired product.

4. Typical experimental procedure for the synthesis of 2-substituted indoles (Table 1, entry 1)

Toluene (2.0 mL) was added to PdCl₂(CH₃CN)₂ (2.6 mg, 0.01 mmol), **1b**•HBF₄ (10.9 mg, 0.02 mmol), *t*-BuOLi (96 mg, 1.2 mmol), and **2a** (140 mg, 0.50 mmol) in a two-neck flask under argon. The reaction mixture was stirred at rt for 40 min, and then ethylbenzene (112 μL, 1.0 mmol) was added. The reaction mixture was stirred at reflux for 3 h. Then, water (2 mL) was added, and the reaction mixture was stirred at reflux for 3 h. The resulting suspension was quenched with aq. NH₄Cl (5 mL) at rt and extracted with ethyl acetate (20 mL × 2). The combined organic layers were dried over anhydrous Na₂SO₄ and concentrated, and then the residue was purified by preparative TLC (hexanes/CH₂Cl₂ = 1/1) to give 2-phenyl-1-tosyl-1*H*-indole **4a** (118 mg, 68% yield) as a pale yellow solid.

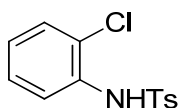
5. Typical experimental procedure for the Suzuki-Miyaura coupling of 4-chloroindole **9 (Scheme 2)**

Toluene (1.0 mL) and water (1.0 mL) were added to PdCl₂(CH₃CN)₂ (1.3 mg, 0.005 mmol), ligand (0.01 mmol), **9** (96 mg, 0.25 mmol), 4-methoxyphenylboronic acid **15** (57 mg, 0.375 mmol), and K₃PO₄ (106 mg, 0.5 mmol) in a two-neck flask under argon. The reaction mixture was stirred at reflux for 6 h. The resulting suspension was quenched with 1M aq. HCl (3 mL) at rt and filtered through the pad of Celite. The pad was washed with ethyl acetate (50 mL). The combined organic layers were dried over anhydrous Na₂SO₄, and concentrated. The residue was purified by column chromatography (SiO₂, hexanes/CH₂Cl₂ = 2/1) to give 4-(4-methoxyphenyl)-2-phenyl-1-tosyl-1*H*-indole **16** (135 mg, quant) as a pale yellow solid.

6. Typical experimental procedure for the synthesis of disubstituted indoles (Table 2, Entry 10)

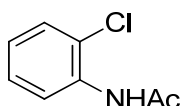
Toluene (2.0 mL) was added to PdCl₂(CH₃CN)₂ (2.6 mg, 0.01 mmol), **1b**•HBF₄ (10.9 mg, 0.02 mmol), *t*-BuOLi (96 mg, 1.20 mmol), and **7** (158 mg, 0.50 mmol) in a two-neck flask under argon. The reaction mixture was stirred at rt for 40 min, and then ethylbenzene (112 μL, 1.0 mmol) was added. The reaction mixture was stirred at reflux for 3 h. Then, water (2 mL) was added, and the reaction mixture was stirred at reflux for 3 h. After cooling down, 4-methoxyphenylboronic acid **15** (152 mg, 1.0 mmol), K₃PO₄ (318 mg, 1.5 mmol), and tetrabutylammonium chloride (69 mg, 0.25 mmol) were added, and the reaction mixture was stirred at reflux for 12 h. The resulting suspension was quenched with 1M aq. HCl (6 mL) at rt and filtered through the pad of Celite. The pad was washed with ethyl acetate (50 mL). The combined organic layers were dried over anhydrous Na₂SO₄ and concentrated, and then the residue was purified by column chromatography (SiO₂, hexanes/CH₂Cl₂ = 2/1) to give 4-(4-methoxyphenyl)-2-phenyl-1-tosyl-1*H*-indole **16** (157 mg, 69%) as a pale yellow solid.

7. Analytical data of the products



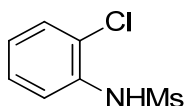
***N*-(2-Chlorophenyl)-4-methylbenzenesulfonamide (2a)²**

Prepared from 21.7 mmol of 2-chloroaniline following the typical procedure. Purification by silica gel column chromatography (hexane/ethyl acetate) afforded 4.99 g (82% yield) of **2a** as a colorless crystal. Spectral data matches that reported in the literature.



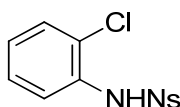
***N*-(2-Chlorophenyl)acetamide (2c)³**

Prepared from 10 mmol of 2-chloroaniline following the reported procedure.^{3a} Recrystallization from ethanol afforded 1.01 g (60% yield) of **2c** as a colorless crystal. Spectral data matches that reported in the literature.



***N*-(2-Chlorophenyl)methanesulfonamide (2d)⁴**

Prepared from 10 mmol of 2-chloroaniline following the reported procedure.^{4a} Recrystallization from ethanol afforded 1.25 g (61% yield) of **2d** as a colorless crystal. Spectral data matches that reported in the literature.



***N*-(2-Chlorophenyl)-2-nitrobenzenesulfonamide (2e)⁶**

Prepared from 12 mmol of 2-chloroaniline following the literature procedure.⁷ Recrystallization from ethyl acetate/hexane afforded 1.49 g (48% yield) of **2e** as a colorless crystal.

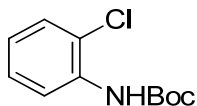
¹H NMR (400 MHz, CDCl₃) δ 7.89 (3H, ddd, J = 17.2, 7.9, 1.3 Hz), 7.75-7.71 (2H, m), 7.63 (1H, td, J = 7.7, 1.3 Hz), 7.29 (2H, dt, J = 13.8, 5.2 Hz), 7.13 (1H, td, J = 7.7, 1.8 Hz)

¹³C NMR (100 MHz, CDCl₃) δ 134.1, 133.1, 132.9, 132.7, 131.0, 129.6, 127.9, 127.0, 126.6, 125.6, 124.9

HRMS (ESI) m/z calcd for C₁₂H₉N₂O₄ClSNa ([M+Na]⁺) : 334.9864, found : 334.9871

IR (ATR) 3291, 1539, 1474, 1366, 1331, 1177, 1126, 1057, 926 cm⁻¹

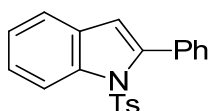
Mp. 147.0-148.0 °C



***tert*-Butyl (2-chlorophenyl)carbamate (**2f**)⁵**

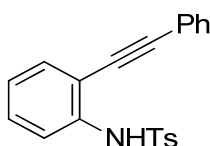
Prepared from 10 mmol of 2-chloroaniline following the reported procedure.^{5a} Purification by silica gel column chromatography (hexane/ethyl acetate) afforded 0.31 g (14% yield) of **2f** as a colorless oil. Spectral data matches that reported in the literature.

¹³C NMR (100 MHz, CDCl₃) δ 152.3, 135.1, 128.9, 127.6, 123.2, 121.8, 119.7, 81.0, 28.2



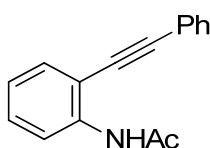
2-Phenyl-1-tosyl-1*H*-indole (4a**)¹⁰**

Prepared from **2a** and ethynylbenzene **3** following the typical procedure. The crude reaction mixture was purified by preparative TLC (hexanes/chloroform = 1/1) to yield 118 mg (68% yield) of **4a** as a pale yellow solid. Spectral data matches that reported in the literature.



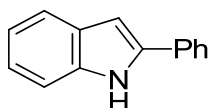
4-Methyl-*N*-(2-(phenylethynyl)phenyl)benzenesulfonamide (5a**)¹¹**

Prepared from **2a** and ethynylbenzene **3** following the typical procedure. The crude reaction mixture was purified by preparative TLC (hexanes/chloroform = 1/2) to yield **5a** as a pale yellow solid. Spectral data matches that reported in the literature.



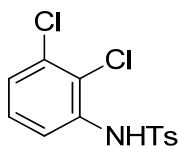
***N*-(2-(Phenylethynyl)phenyl)acetamide (**5c**)¹²**

Prepared from **2c** and ethynylbenzene **3** following the typical procedure. The crude reaction mixture was purified by column chromatography (SiO₂, hexanes/dichloromethane = 2/3) to yield **5c** as a pale yellow solid. Spectral data matches that reported in the literature.



2-Phenyl-1*H*-indole (6)¹³

Prepared from **2a** and ethynylbenzene **3** following the typical procedure. The crude reaction mixture was purified by column chromatography (SiO₂, hexanes/dichloromethane = 2/1) to yield 12 mg (12% yield) of **6** as a pale yellow solid. Spectral data matches that reported in the literature.



N-(2,3-Dichlorophenyl)-4-methylbenzenesulfonamide (**7**)⁸

Prepared from 2,3-dichloroaniline following the typical procedure. Recrystallization from ethanol afford 4.79 g (76% yield) of **7** as a colorless crystal.

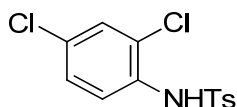
¹H NMR (400 MHz, CDCl₃) δ 7.67 (2H, d, *J* = 8.0 Hz), 7.58 (1H, d, *J* = 7.2 Hz), 7.26-7.23 (3H, m), 7.19-7.13 (1H, m), 7.10 (1H, s), 2.39 (3H, s)

¹³C NMR (100 MHz, CDCl₃) δ 144.5, 135.3, 129.8, 127.8, 127.3, 126.3, 119.6, 21.6

HRMS (ESI) *m/z* calcd for C₁₃H₁₁NO₂Cl₂SNa ([M+Na]⁺) : 337.9780, found : 337.9738

IR (ATR) 3271, 1584, 1456, 1381, 1333, 1163, 1092, 937 cm⁻¹

Mp. 124.0-126.2 °C



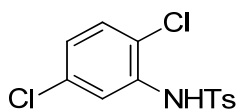
N-(2,4-Dichlorophenyl)-4-methylbenzenesulfonamide (**32**)⁹

Prepared from 2,4-dichloroaniline following the typical procedure. Purification by silica gel column chromatography (hexane/chloroform = 2/1) afforded 4.11 g (65 % yield) of **32** as a colorless crystal. Spectral data matches that reported in the literature.

HRMS (ESI) *m/z* calcd for C₁₃H₁₁NO₂Cl₂SNa ([M+Na]⁺) : 337.9780, found : 337.9790

IR (ATR) 3258, 1474, 1410, 1375, 1335, 1167, 1090, 1053, 903 cm⁻¹

Mp. 126.8-127.7 °C



N-(2,5-Dichlorophenyl)-4-methylbenzenesulfonamide (**33**)

Prepared from 2,5-dichloroaniline following the typical procedure. Recrystallization from ethanol afforded 3.81 g (60 % yield) of **33** as a colorless crystal.

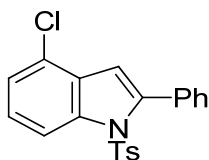
¹H NMR (400 MHz, CDCl₃) δ 7.69 (3H, t, *J* = 4.9 Hz), 7.26 (2H, t, *J* = 4.1 Hz), 7.18 (1H, d, *J* = 8.3 Hz), 7.00 (2H, dd, *J* = 8.8, 2.4 Hz), 2.39 (3H, s)

^{13}C NMR (100 MHz, CDCl_3) δ 144.6, 135.6, 134.5, 133.7, 130.1, 129.8, 127.3, 125.6, 122.7, 121.6, 21.6

HRMS (ESI) m/z calcd for $\text{C}_{13}\text{H}_{11}\text{NO}_2\text{Cl}_2\text{SNa}$ ($[\text{M}+\text{Na}]^+$) : 337.9780, found : 337.9793

IR (ATR) 3262, 1572, 1477, 1381, 1339, 1167, 1090, 1045, 924, 806 cm^{-1}

Mp. 144.9-145.9 $^\circ\text{C}$



4-Chloro-2-phenyl-1-tosyl-1H-indole (9)

Prepared from **7** and ethynylbenzene **3** following the typical procedure. The crude reaction mixture was purified by preparative TLC (hexanes/dichloromethane = 3/1) to yield 169 mg (88% yield) of **9** as a pale yellow solid.

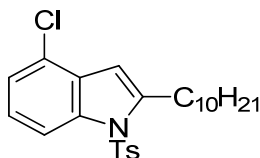
^1H NMR (400 MHz, CDCl_3) δ 8.22 (1H, d, $J = 9.3$ Hz), 7.51-7.39 (5H, m), 7.28-7.23 (4H, m), 7.04 (2H, d, $J = 7.8$ Hz), 6.66 (1H, s), 2.28 (3H, s)

^{13}C NMR (100 MHz, CDCl_3) δ 144.9, 142.7, 138.7, 134.5, 131.8, 130.4, 129.3, 129.2, 28.9, 127.5, 126.8, 125.8, 125.3, 124.0, 114.9, 111.1, 21.5

HRMS (ESI) m/z calcd for $\text{C}_{21}\text{H}_{17}\text{NO}_2\text{ClS}$ ($[\text{M}+\text{H}]^+$) : 382.0663, found : 382.0676

IR (ATR) 3059, 2924, 1595, 1491, 1464, 1423, 1369, 1163, 1051, 995 cm^{-1}

Mp. 156.5-157.3 $^\circ\text{C}$



4-Chloro-2-decyl-1-tosyl-1H-indole (10)

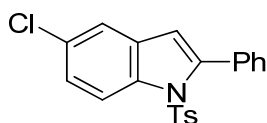
Prepared from **7** and 1-dodecyne **8** following the typical procedure. The crude reaction mixture was purified by preparative TLC (hexanes/dichloromethane = 3/1) to yield 186 mg (83% yield) of **10** as a pale yellow oil.

^1H NMR (400 MHz, CDCl_3) δ 8.08 (1H, d, $J = 3.9$ Hz), 7.61 (2H, dd, $J = 6.1, 4.1$ Hz), 7.25-7.14 (4H, m), 6.49 (1H, d, $J = 1.0$ Hz), 2.98 (2H, t, $J = 7.3$ Hz), 2.33 (3H, s), 1.75 (2H, t, $J = 7.6$ Hz), 1.43-1.27 (14H, m), 0.88 (3H, t, $J = 6.8$ Hz)

^{13}C NMR (100 MHz, CDCl_3) δ 144.9, 143.4, 137.7, 136.0, 129.9, 128.5, 126.3, 125.2, 124.3, 123.2, 113.2, 106.3, 31.9, 29.6, 29.5, 29.39, 29.36, 29.3, 29.0, 28.7, 22.7, 21.5, 14.1

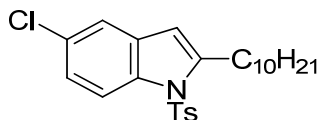
HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{33}\text{NO}_2\text{ClS}$ ($[\text{M}+\text{H}]^+$) : 446.1915, found : 446.1907

IR (ATR) 2922, 2853, 1597, 1560, 1425, 1371, 1171, 1092, 1049, 947 cm^{-1}



5-Chloro-2-phenyl-1-tosyl-1H-indole (11)¹⁴

Prepared from **32** and ethynylbenzene **3** following the typical procedure. The crude reaction mixture was purified by preparative TLC (hexanes/dichloromethane = 3/1) to yield 127 mg (67% yield) of **11** as a pale yellow solid. Spectral data matches that reported in the literature.



5-Chloro-2-decyl-1-tosyl-1H-indole (**12**)

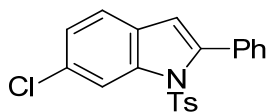
Prepared from **32** and 1-dodecyne **8** following the typical procedure. The crude reaction mixture was purified by preparative TLC (hexanes/dichloromethane = 3/1) to yield 186 mg (83% yield) of **12** as a pale yellow oil.

^1H NMR (400 MHz, CDCl_3) δ 8.09 (1H, d, $J = 9.3$ Hz), 7.58 (2H, d, $J = 8.3$ Hz), 7.34 (1H, d, $J = 2.0$ Hz), 7.18-7.16 (3H, m), 6.29 (1H, s), 2.95 (2H, t, $J = 7.6$ Hz), 2.30 (3H, s), 1.71 (2H, t, $J = 7.3$ Hz), 1.36-1.29 (14H, m), 0.88 (3H, t, $J = 6.6$ Hz)

^{13}C NMR (100 MHz, CDCl_3) δ 144.8, 144.1, 136.0, 135.5, 131.1, 129.8, 129.1, 126.2, 123.8, 19.6, 115.8, 107.5, 31.9, 29.6, 29.5, 29.4, 29.31, 29.28, 28.9, 28.7, 22.6, 21.5, 14.1

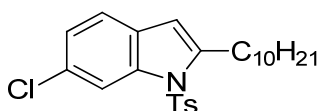
HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{33}\text{NO}_2\text{ClS}$ ($[\text{M}+\text{H}]^+$) : 446.1915, found : 446.1915

IR (ATR) 2922, 2853, 1597, 1445, 1369, 1219, 1173, 1072, 1049, 923 cm^{-1}



6-Chloro-2-phenyl-1-tosyl-1H-indole (**13**)¹⁵

Prepared from **33** and ethynylbenzene **3** following the typical procedure. The crude reaction mixture was purified by preparative TLC (hexanes/dichloromethane = 3/1) to yield 109 mg (57% yield) of **13** as a pale yellow solid. Spectral data matches that reported in the literature.



6-Chloro-2-decyl-1-tosyl-1H-indole (**14**)

Prepared from **33** and 1-dodecyne **8** following the typical procedure. The crude reaction mixture was purified by preparative TLC (hexanes/dichloromethane = 3/1) to yield 57 mg (30% yield) of **14** as a pale yellow solid.

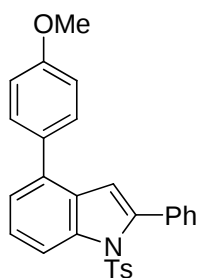
^1H NMR (400 MHz, CDCl_3) δ 8.21 (1H, s), 7.61 (2H, d, $J = 8.3$ Hz), 7.30 (1H, d, $J = 8.3$ Hz), 7.18 (3H, dt, $J = 12.2, 5.2$ Hz), 6.33 (1H, s), 2.93 (2H, t, $J = 7.6$ Hz), 2.34 (3H, s), 1.67-1.74 (2H, m), 1.37-1.25 (14H, m), 0.88 (3H, t, $J = 6.8$ Hz)

^{13}C NMR (100 MHz, CDCl_3) δ 144.9, 143.3, 137.5, 136.0, 129.9, 129.6, 128.3, 126.3, 124.0, 120.6, 115.0, 108.0, 31.9, 29.6, 29.5, 29.4, 29.33, 29.32, 28.9, 28.7, 22.7, 21.6, 14.1

HRMS (ESI) m/z calcd for $\text{C}_{25}\text{H}_{33}\text{NO}_2\text{ClS}$ ($[\text{M}+\text{H}]^+$) : 446.1915, found : 446.1906

IR (ATR) 2924, 2855, 1591, 1452, 1418, 1368, 1169, 1148, 1092, 1040, 932 cm^{-1}

Mp. 81.7-82.7 °C



4-(4-Methoxyphenyl)-2-phenyl-1-tosyl-1H-indole (**16**)

Prepared from **7**, ethynylbenzene **3**, and 4-methoxyphenylboronic acid **15** following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 157 mg (69% yield) of **16** as a pale yellow solid.

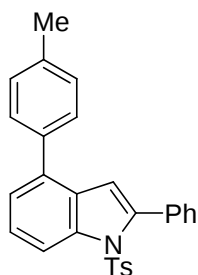
^1H NMR (400 MHz, CDCl_3) δ 8.28 (1H, d, J = 8.3 Hz), 7.48 (2H, td, J = 4.9, 1.6 Hz), 7.43-7.36 (6H, m), 7.31 (2H, d, J = 8.3 Hz), 7.26 (1H, t, J = 3.9 Hz), 7.02 (2H, d, J = 8.3 Hz), 6.93 (2H, dt, J = 9.3, 2.6 Hz), 6.71 (1H, s), 3.79 (3H, s), 2.25 (3H, s)

^{13}C NMR (100 MHz, CDCl_3) δ 159.0, 144.5, 142.0, 138.6, 134.8, 134.3, 132.5, 132.3, 130.3, 129.7, 129.2, 128.5, 127.4, 126.8, 125.0, 123.9, 114.9, 114.0, 112.9, 55.2, 21.4

HRMS (DART) m/z calcd for $\text{C}_{28}\text{H}_{24}\text{NO}_3\text{S}$ ($[\text{M}+\text{H}]^+$) : 454.1471, found : 454.1494

IR (ATR) 3065, 2994, 2930, 2832, 1609, 1595, 1516, 1474, 1429, 1366, 1298, 1238, 1163, 1109, 1090, 1057, 1026, 987 cm^{-1}

Mp. 159.4-159.9 °C



4-(4-Methylphenyl)-2-phenyl-1-tosyl-1H-indole (**17**)

Prepared from **7**, ethynylbenzene **3**, and 4-methylphenylboronic acid following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 68 mg (38 % yield) of **17** as a pale yellow solid.

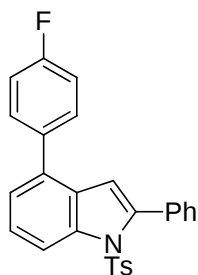
^1H NMR (400 MHz, CDCl_3) δ 8.29 (1H, d, J = 8.3 Hz), 7.48 (2H, t, J = 3.7 Hz), 7.41 (6H, t, J = 6.6 Hz), 7.31 (3H, q, J = 3.6 Hz), 7.24 (2H, t, J = 7.8 Hz), 7.05 (2H, d, J = 8.3 Hz), 6.72 (1H, s), 2.29 (3H, s), 2.38 (3H, s)

^{13}C NMR (100 MHz, CDCl_3) δ 144.5, 142.1, 138.6, 137.1, 137.0, 134.8, 134.6, 132.5, 130.3, 129.3, 129.2, 128.7, 128.6, 127.4, 126.9, 125.0, 124.1, 115.2, 113.0, 21.5, 21.1

HRMS (DART) m/z calcd for $\text{C}_{28}\text{H}_{24}\text{NO}_2\text{S}$ ($[\text{M}+\text{H}]^+$) : 438.1522, found : 438.1532

IR (ATR) 3028, 2918, 1595, 1474, 1423, 1366, 1238, 1167, 1109, 1057, 993 cm^{-1}

Mp. 148.7-149.7 °C



4-(4-Fluorophenyl)-2-phenyl-1-tosyl-1H-indole (18)

Prepared from **7**, ethynylbenzene **3**, and 4-fluorophenylboronic acid following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 62 mg (28% yield) of **18** as a pale yellow solid.

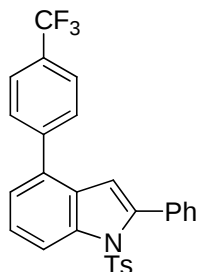
^1H NMR (400 MHz, CDCl_3) δ 8.32 (1H, d, J = 8.3 Hz), 7.67-7.37 (8H, m), 7.32 (2H, dt, J = 10.2, 3.2 Hz), 7.22 (1H, tt, J = 20.5, 6.7 Hz), 7.06 (4H, ddt, J = 24.2, 15.4, 7.4 Hz), 6.67 (1H, s), 2.28 (3H, s)

^{13}C NMR (100 MHz, CDCl_3) δ 163.5, 161.0, 144.6, 142.3, 138.5, 135.9, 135.8, 134.8, 133.5, 132.3, 131.6, 130.34, 130.28, 130.21, 129.3, 128.7, 128.5, 128.4, 127.5, 126.8, 125.0, 124.1, 115.6, 115.44, 115.37, 112.4, 21.5

HRMS (DART) m/z calcd for $\text{C}_{27}\text{H}_{21}\text{NO}_2\text{SF}$ ($[\text{M}+\text{H}]^+$) : 442.1272, found : 442.1285

IR (ATR) 3061, 2922, 1597, 1512, 1427, 1364, 1225, 1186, 1161, 1057, 986 cm^{-1}

Mp. 135.8-136.5 °C



4-(4-Trifluoromethylphenyl)-2-phenyl-1-tosyl-1H-indole (19)

Prepared from **7**, ethynylbenzene **3**, and 4-trifluoromethylphenylboronic acid following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 103 mg (42% yield) of **19** as a pale yellow solid.

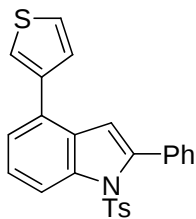
^1H NMR (400 MHz, CDCl_3) δ 8.38 (1H, d, J = 8.3 Hz), 7.64 (4H, dd, J = 22.9, 8.3 Hz), 7.49-7.38 (6H, m), 7.32 (3H, d, J = 8.3 Hz), 7.07 (2H, d, J = 7.8 Hz), 6.65 (1H, s), 2.30 (3H, s)

^{13}C NMR (100 MHz, CDCl_3) δ 144.7, 143.5, 142.7, 138.5, 134.8, 133.0, 132.2, 130.4, 129.6, 129.3, 129.2, 129.0, 128.8, 128.5, 127.5, 126.9, 125.53, 125.50, 125.45, 125.0, 124.2, 122.8, 116.2, 112.0, 21.5

HRMS (DART) m/z calcd for $\text{C}_{28}\text{H}_{21}\text{NO}_2\text{SF}_3$ ($[\text{M}+\text{H}]^+$) : 492.1240, found : 492.1237

IR (ATR) 3061, 1618, 1400, 1368, 1327, 1188, 1161, 1109, 1070, 989 cm^{-1}

Mp. 167.9-168.9 °C



4-(3-Thienyl)-2-phenyl-1-tosyl-1H-indole (20)

Prepared from **7**, ethynylbenzene **3**, and 3-thienylboronic acid following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 67 mg (31% yield) of **20** as a pale yellow solid.

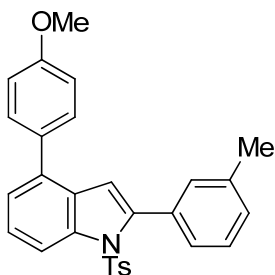
^1H NMR (400 MHz, CDCl_3) δ 8.29 (1H, dt, J = 6.8, 1.0 Hz), 7.49 (2H, td, J = 4.9, 1.8 Hz), 7.43-7.34 (7H, m), 7.32-7.29 (3H, m), 7.04 (2H, d, J = 7.8 Hz), 6.78 (1H, s), 2.27 (3H, s)

^{13}C NMR (100 MHz, CDCl_3) δ 144.6, 142.2, 140.6, 138.6, 134.7, 132.4, 130.3, 129.3, 128.7, 128.42, 128.36, 127.9, 127.5, 126.8, 125.9, 124.9, 123.7, 122.2, 115.4, 112.8, 21.5

HRMS (DART) m/z calcd for $\text{C}_{25}\text{H}_{20}\text{NO}_2\text{S}_2$ ($[\text{M}+\text{H}]^+$) : 430.0930, found : 430.0941

IR (ATR) 3059, 2924, 1595, 1489, 1464 1423, 1369, 1186, 1163, 1090, 1051, 995, 916 cm^{-1}

Mp. 155.9-157.7 $^\circ\text{C}$



4-(4-Methoxyphenyl)-2-(3-methylphenyl)-1-tosyl-1H-indole (21)

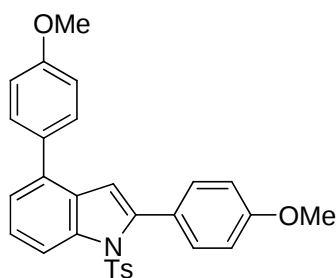
Prepared from **7**, 1-ethynyl-3-methylbenzene, and 4-methoxyphenylboronic acid **15** following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 109 mg (47% yield) of **21** as a pale yellow solid.

^1H NMR (400 MHz, CDCl_3) δ 8.27 (1H, d, J = 8.3 Hz), 7.21-7.43 (10H, m), 7.03 (2H, d, J = 7.8 Hz), 6.93 (2H, d, J = 8.3 Hz), 6.70 (1H, s), 3.80 (3H, s), 2.38 (3H, s), 2.26 (3H, s)

^{13}C NMR (100 MHz, CDCl_3) δ 159.0, 144.5, 142.2, 128.5, 136.9, 134.8, 134.2, 132.35, 132.27, 130.9, 129.7, 129.3, 129.2, 128.7, 128.5, 127.4, 127.3, 126.8, 124.9, 123.8, 114.9, 114.0, 112.6, 55.2, 21.4, 21.3

HRMS (DART) m/z calcd for $\text{C}_{29}\text{H}_{26}\text{NO}_3\text{S}$ ($[\text{M}+\text{H}]^+$) : 468.1628, found : 468.1643

IR (ATR) 2955, 2922, 1609, 1597, 1516, 1369, 1246, 1167, 887 cm^{-1}



2,4-Di(4-methoxyphenyl)-1-tosyl-1H-indole (**22**)

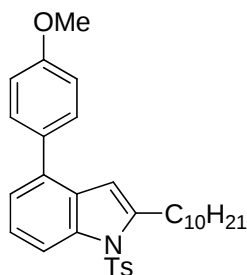
Prepared from **7**, 1-ethynyl-4-methoxybenzene, and 4-methoxyphenylboronic acid **15** following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 118 mg (49% yield) of **22** as a pale brown solid.

^1H NMR (400 MHz, CDCl_3) δ 8.27 (1H, d, J = 8.3 Hz), 7.40 (5H, tt, J = 12.7, 4.9 Hz), 7.27 (3H, dt, J = 15.9, 7.6 Hz), 7.04 (2H, d, J = 8.3 Hz), 6.94 (4H, dt, J = 9.8, 3.4 Hz), 6.65 (1H, s), 3.85 (3H, s), 3.81 (3H, s), 2.27 (3H, s)

^{13}C NMR (100 MHz, CDCl_3) δ 159.9, 159.0, 144.5, 141.9, 138.4, 14.8, 134.1, 132.3, 131.6, 129.7, 129.2, 128.6, 126.8, 124.7, 123.9, 114.9, 114.0, 112.9, 112.2, 55.3, 55.2, 21.5

HRMS (DART) m/z calcd for $\text{C}_{29}\text{H}_{26}\text{NO}_4\text{S}$ ($[\text{M}+\text{H}]^+$) : 484.1577, found : 484.1564

IR (ATR) 2932, 2835, 1609, 1503, 1369, 1244, 1173, 1030, 829 cm^{-1}



2-Decyl-4-(4-methoxyphenyl)-1-tosyl-1H-indole (**23**)

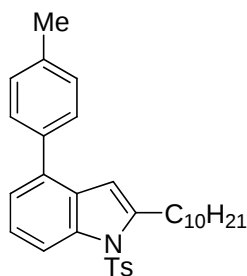
Prepared from **7**, 1-dodecyne **8**, and 4-methoxyphenylboronic acid **15** following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 226 mg (87% yield) of **23** as a yellow oil.

^1H NMR (400 MHz, CDCl_3) δ 8.14 (1H, d, J = 8.3 Hz), 7.65 (2H, d, J = 8.3 Hz), 7.44 (2H, dt, J = 9.3, 2.3 Hz), 7.28 (1H, t, J = 7.8 Hz), 7.22-7.15 (3H, m), 6.99-6.96 (2H, m), 6.52 (1H, s), 3.83 (3H, d, J = 4.4 Hz), 2.99 (2H, t, J = 7.6 Hz), 2.30 (3H, s), 1.70 (2H, q, J = 7.5 Hz), 1.29-1.11 (14H, m), 0.87 (3H, t, J = 6.8 Hz).

^{13}C NMR (100 MHz, CDCl_3) δ 158.9, 114.5, 142.6, 137.5, 136.3, 133.6, 132.6, 129.7, 129.6, 127.8, 126.3, 123.9, 123.2, 114.0, 113.2, 107.7, 55.2, 31.9, 29.6, 29.5, 29.41, 29.38, 29.3, 29.1, 29.0, 22.6, 21.5, 14.1

HRMS (DART) m/z calcd for $\text{C}_{32}\text{H}_{40}\text{NO}_3\text{S}$ ($[\text{M}+\text{H}]^+$) : 518.2723, found : 518.2724

IR (ATR) 2922, 2853, 1609, 1516, 1477, 1368, 1244, 1173, 1146, 109, 1032, 833 cm^{-1}



2-Decyl-4-(4-methylphenyl)-1-tosyl-1H-indole (**24**)

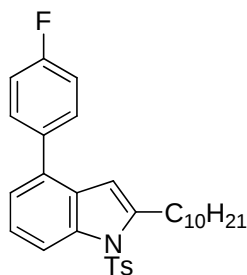
Prepared from **7**, 1-dodecyne **8**, and 4-methylphenylboronic acid following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 138 mg (55% yield) of **24** as a pale yellow oil.

^1H NMR (400 MHz, CDCl_3) δ 8.15 (1H, d, J = 8.3 Hz), 7.65 (2H, dt, J = 8.8, 2.0 Hz), 7.41 (2H, dd, J = 6.3, 2.0 Hz), 7.25 (6H, m), 6.52 (1H, d, J = 1.0 Hz), 2.97 (2H, t, J = 7.6 Hz), 2.41 (3H, s), 2.33 (3H, s), 1.74-1.67 (2H, m), 1.40-1.12 (14H, m), 0.88 (3H, t, J = 6.8 Hz)

^{13}C NMR (100 MHz, CDCl_3) δ 144.5, 142.7, 137.5, 137.3, 136.9, 136.3, 133.9, 129.7, 129.2, 128.6, 127.9, 126.3, 123.9, 123.3, 113.5, 107.7, 31.9, 29.6, 29.5, 29.41, 29.38, 29.28, 29.1, 29.0, 22.6, 21.5, 21.1, 14.1

HRMS (DART) m/z calcd for $\text{C}_{32}\text{H}_{40}\text{NO}_2\text{S}$ ($[\text{M}+\text{H}]^+$) : 502.2774, found : 502.2785

IR (ATR) 2922, 2853, 1597, 1560, 1425, 1368, 1171, 1146, 1090, 1047, 823 cm^{-1}



2-Decyl-4-(4-fluorophenyl)-1-tosyl-1H-indole (**25**)

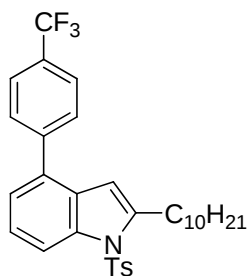
Prepared from **7**, 1-dodecyne **8**, and 4-fluorophenylboronic acid following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 122 mg (48% yield) of **25** as a yellow oil.

^1H NMR (400 MHz, CDCl_3) δ 8.18 (1H, d, J = 8.3 Hz), 7.66 (2H, t, J = 4.1 Hz), 7.46 (2H, td, J = 6.0, 2.6 Hz), 7.29 (1H, t, J = 8.0 Hz), 7.19 (3H, d, J = 7.8 Hz), 7.15-7.10 (2H, m), 6.47 (1H, s), 2.99 (2H, t, J = 7.3 Hz), 2.32 (3H, s), 1.70 (2H, q, J = 7.6 Hz), 1.41-0.86 (14H, m), 0.87 (3H, t, J = 6.8 Hz)

^{13}C NMR (100 MHz, CDCl_3) δ 163.4, 161.0, 144.7, 143.0, 137.5, 136.3, 136.20, 136.17, 132.9, 130.3, 130.2, 129.8, 127.9, 126.3, 124.0, 123.4, 115.6, 115.3, 113.8, 107.3, 31.9, 29.59, 29.56, 29.43, 29.39, 29.30, 29.1, 29.0, 22.7, 21.5, 14.1

HRMS (DART) m/z calcd for $\text{C}_{31}\text{H}_{37}\text{NO}_2\text{SF}$ ($[\text{M}+\text{H}]^+$) : 506.2524, found : 506.2521

IR (ATR) 2922, 2853, 1597, 1514, 1368, 1223, 1188, 1148, 1090, 1047, 839 cm^{-1}



2-Decyl-4-(4-trifluoromethylphenyl)-1-tosyl-1H-indole (26)

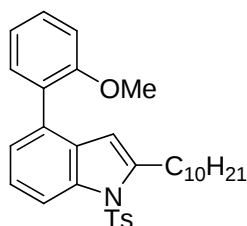
Prepared from **7**, 1-dodecyne **8**, and 4-trifluoromethylphenylboronic acid following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 201 mg (72% yield) of **26** as a yellow oil.

^1H NMR (400 MHz, CDCl_3) δ 8.23 (1H, d, J = 8.8 Hz), 7.67 (6H, td, J = 16.5, 8.1 Hz), 7.34 (1H, t, J = 8.0 Hz), 7.26-7.21 (3H, m), 6.46 (1H, s), 2.99 (2H, t, J = 7.6 Hz), 2.34 (3H, d, J = 9.3 Hz), 1.75-1.67 (2H, m), 1.56 (2H, s), 1.37-1.29 (14H, m), 0.88 (3H, t, J = 6.8 Hz)

^{13}C NMR (100 MHz, CDCl_3) δ 144.8, 143.8, 143.4, 137.5, 136.2, 132.3, 129.8, 129.0, 127.8, 126.3, 125.6, 124.0, 123.5, 114.8, 106.9, 31.9, 29.6, 29.5, 29.42, 29.37, 29.29, 29.1, 29.0, 22.6, 21.5, 14.1

HRMS (DART) m/z calcd for $\text{C}_{32}\text{H}_{37}\text{NO}_2\text{SF}_3$ ($[\text{M}+\text{H}]^+$) : 556.2491, found : 556.2493

IR (ATR) 2924, 2853, 1616, 1597, 1560, 1369, 1323, 1167, 1123, 1067, 1016, 847 cm^{-1}



2-Decyl-4-(2-methoxyphenyl)-1-tosyl-1H-indole (27)

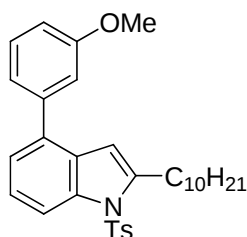
Prepared from **7**, 1-dodecyne **8**, and 4-methoxyphenylboronic acid **15** following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 150 mg (87 % yield) of **27** as a pale yellow oil.

^1H NMR (400 MHz, CDCl_3) δ 8.15 (1H, d, J = 7.8 Hz), 7.66 (2H, t, J = 4.1 Hz), 7.36-7.16 (6H, m), 7.03-6.98 (2H, m), 6.21 (1H, s), 3.71 (3H, d, J = 1.5 Hz), 2.95 (2H, t, J = 7.6 Hz), 2.32 (3H, s), 1.66 (2H, q, J = 7.0 Hz), 1.38-1.17 (14H, m), 0.87 (3H, t, J = 5.9 Hz)

^{13}C NMR (100 MHz, CDCl_3) δ 156.6, 144.4, 142.0, 137.1, 136.5, 131.5, 130.6, 129.7, 129.0, 128.8, 126.4, 124.7, 123.4, 120.6, 113.6, 111.2, 108.5, 55.4, 31.86, 29.6, 29.5, 29.4, 29.35, 29.28, 29.0, 28.9, 22.6, 21.5, 14.1

HRMS (DART) m/z calcd for $\text{C}_{32}\text{H}_{40}\text{NO}_3\text{S}$ ($[\text{M}+\text{H}]^+$) : 518.2723, found : 518.2717

IR (ATR) 2922, 2853, 1462, 1420, 1368, 1240, 1188, 1169, 1146, 1105, 1026, 812 cm^{-1}



2-Decyl-4-(3-methoxyphenyl)-1-tosyl-1H-indole (28)

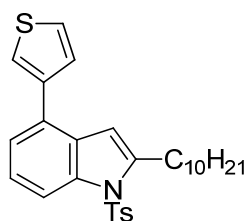
Prepared from **7**, 1-dodecyne **8**, and 4-methoxyphenylboronic acid **15** following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 150 mg (58 % yield) of **28** as a pale yellow oil.

^1H NMR (400 MHz, CDCl_3) δ 8.18 (1H, d, $J = 8.3$ Hz), 7.65 (2H, d, $J = 8.3$ Hz), 7.36-7.21 (3H, m), 7.16 (2H, d, $J = 8.3$ Hz), 7.11-7.05 (2H, m), 6.90 (1H, dd, $J = 8.3, 2.4$ Hz), 6.55 (1H, s), 3.81 (3H, s), 2.99 (2H, t, $J = 7.8$ Hz), 2.30 (3H, s), 1.71 (2H, t, $J = 7.6$ Hz), 1.39-1.26 (14H, m), 0.87 (3H, t, $J = 6.8$ Hz)

^{13}C NMR (100 MHz, CDCl_3) δ 159.7, 144.6, 142.8, 141.6, 137.5, 136.3, 133.8, 129.7, 129.5, 127.9, 126.3, 123.9, 123.4, 121.2, 114.4, 113.8, 112.6, 107.6, 55.2, 31.8, 29.6, 29.5, 29.40, 29.37, 29.28, 29.1, 29.0, 22.6, 21.4, 14.0

HRMS (DART) m/z calcd for $\text{C}_{32}\text{H}_{40}\text{NO}_3\text{S}$ ($[\text{M}+\text{H}]^+$) : 518.2723, found : 518.2714

IR (ATR) 2922, 2853, 1597, 1562, 1474, 1410, 1368, 1229, 1169, 1090, 1042, 777 cm^{-1}



2-Decyl-4-(3-thienyl)-1-tosyl-1H-indole (29)

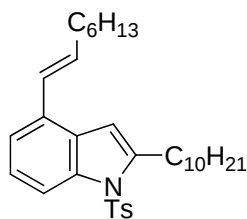
Prepared from **7**, 1-dodecyne **8**, and 3-thienylboronic acid following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 158 mg (64% yield) of **29** as a yellow oil.

^1H NMR (400 MHz, CDCl_3) δ 8.15 (1H, dd, $J = 7.1, 2.2$ Hz), 7.63 (2H, t, $J = 5.1$ Hz), 7.38 (2H, q, $J = 2.1$ Hz), 7.31 (1H, q, $J = 2.1$ Hz), 7.27 (2H, dd, $J = 7.3, 5.4$ Hz), 7.15 (2H, d, $J = 7.8$ Hz), 6.60 (1H, s), 2.99 (2H, t, $J = 7.6$ Hz), 2.29 (3H, s), 1.72 (2H, q, $J = 7.8$ Hz), 1.42-1.26 (14H, m), 0.87 (3H, t, $J = 6.8$ Hz)

^{13}C NMR (100 MHz, CDCl_3) δ 144.6, 142.8, 140.9, 137.6, 136.2, 129.7, 128.5, 127.9, 127.7, 126.3, 125.7, 123.9, 123.0, 122.0, 113.7, 107.7, 31.9, 29.6, 29.5, 29.41, 29.38, 29.3, 29.1, 29.0, 22.6, 21.5, 14.1

HRMS (DART) m/z calcd for $\text{C}_{29}\text{H}_{36}\text{NO}_2\text{S}_2$ ($[\text{M}+\text{H}]^+$) : 494.2182, found : 494.2204

IR (ATR) 2922, 2851, 1425, 1366, 1182, 1148, 1090, 1049, 772 cm^{-1}



2-Decyl 4-(1-*trans*-1-octen-1-yl)-1-tosyl-1*H*-indole (**30**)

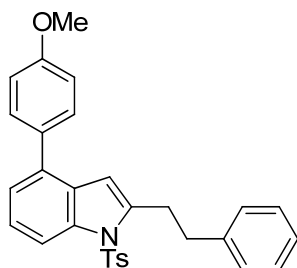
Prepared from **7**, 1-dodecyne **8**, and 1-*trans*-1-octen-1-ylboronic acid following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 93 mg (36% yield) of **30** as a pale yellow oil.

^1H NMR (400 MHz, CDCl_3) δ 8.02 (1H, d, $J = 8.3$ Hz), 7.60 (2H, d, $J = 8.3$ Hz), 7.29 (1H, d, $J = 7.8$ Hz), 7.20-7.15 (3H, m), 6.62 (1H, d, $J = 15.6$ Hz), 6.54 (1H, s), 6.28 (1H, dt, $J = 15.6, 7.1$ Hz), 2.99 (2H, t, $J = 7.6$ Hz), 2.31 (3H, s), 2.24 (1H, q, $J = 6.8$ Hz), 1.79-1.72 (2H, m), 1.39-1.13 (14H, m), 0.89 (3H, t, $J = 6.6$ Hz)

^{13}C NMR (100 MHz, CDCl_3) δ 144.5, 142.3, 137.5, 136.3, 132.9, 129.7, 127.7, 126.4, 126.2, 123.9, 119.7, 113.2, 106.8, 33.4, 31.9, 31.7, 29.60, 29.57, 29.45, 29.42, 29.40, 29.32, 29.14, 28.99, 28.91, 22.7, 22.6, 21.5, 14.1

HRMS (DART) m/z calcd for $\text{C}_{33}\text{H}_{48}\text{NO}_2\text{S}$ ($[\text{M}+\text{H}]^+$) : 522.3400, found : 522.3410

IR (ATR) 2292, 2853, 1597, 1564, 1425, 1367, 1169, 1148, 1094, 964 cm^{-1}



4-(4-Methoxyphenyl)-2-phenethyl-1-tosyl-1*H*-indole (**31**)

Prepared from **7**, 4-phenyl-1-butyne, and 4-methoxyphenylboronic acid **15** following the typical procedure. The crude reaction mixture was purified by column chromatography (hexanes/dichloromethane = 2/1) to yield 135 mg (56% yield) of **31** as a yellow solid.

^1H NMR (400 MHz, CDCl_3) δ 8.15 (1H, d, $J = 8.3$ Hz), 7.64 (2H, d, $J = 8.3$ Hz), 7.39 (2H, d, $J = 8.3$ Hz), 7.31-7.14 (9H, m), 6.96 (2H, d, $J = 8.8$ Hz), 6.51 (1H, s), 3.82 (3H, s), 3.30 (2H, t, $J = 7.8$ Hz), 3.04 (2H, t, $J = 8.0$ Hz), 2.29 (3H, s)

^{13}C NMR (100 MHz, CDCl_3) δ 158.9, 144.7, 141.24, 141.17, 137.5, 136.1, 133.8, 132.4, 129.8, 129.7, 128.5, 128.4, 127.7, 126.3, 126.1, 124.2, 123.2, 114.0, 113.3, 108.7, 55.3, 35.7, 31.3, 21.5

HRMS (DART) m/z calcd for $\text{C}_{30}\text{H}_{28}\text{NO}_3\text{S}$ ($[\text{M}+\text{H}]^+$) : 482.1784, found : 482.1780

IR (ATR) 2951, 2916, 1597, 1560, 1516, 1464, 1364, 1288, 1240, 1169, 1028, 993 cm^{-1}

Mp. 84.7-85.4 $^{\circ}\text{C}$

8. References

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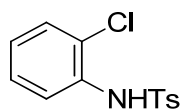
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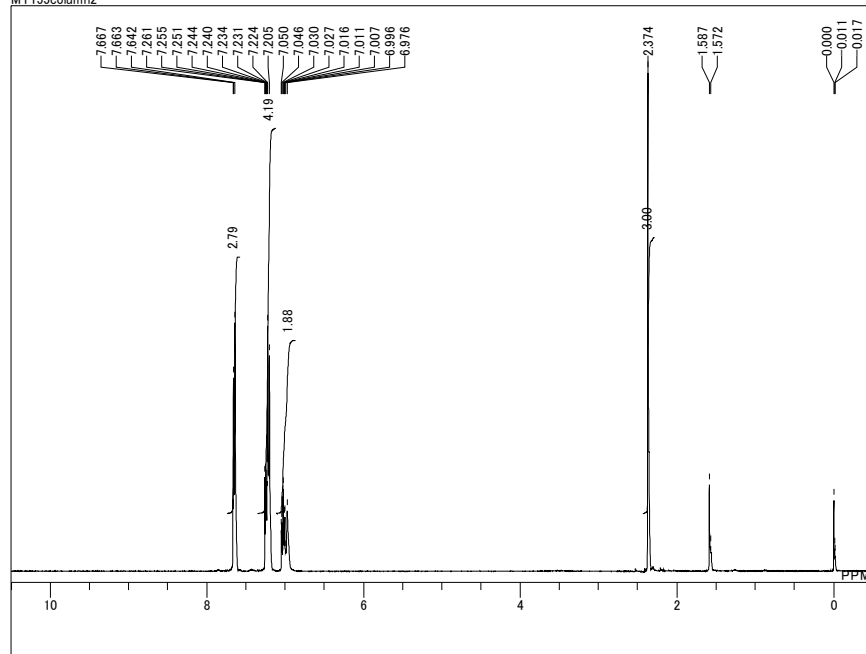
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9. NMR spectra of the products



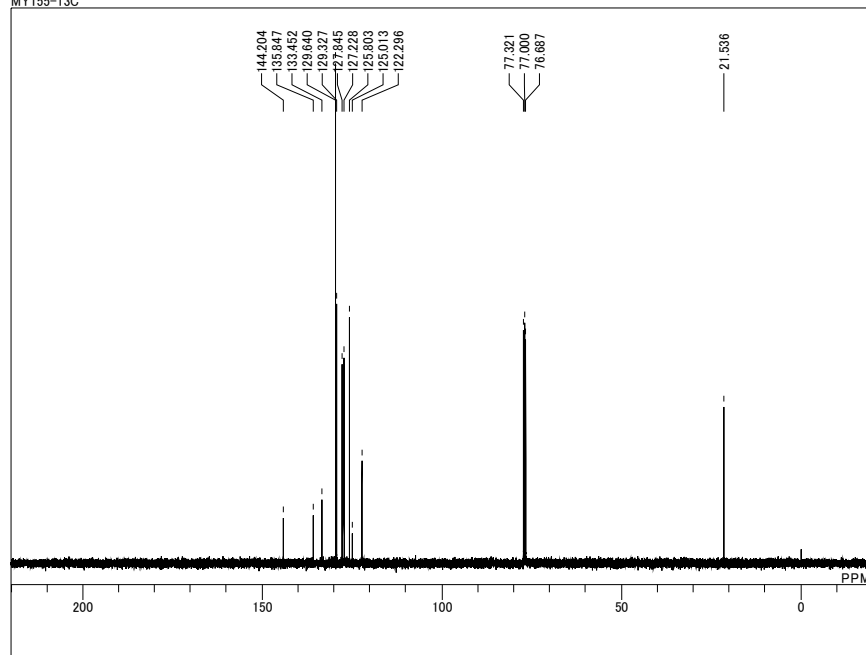
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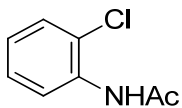


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PD 4.9500 sec
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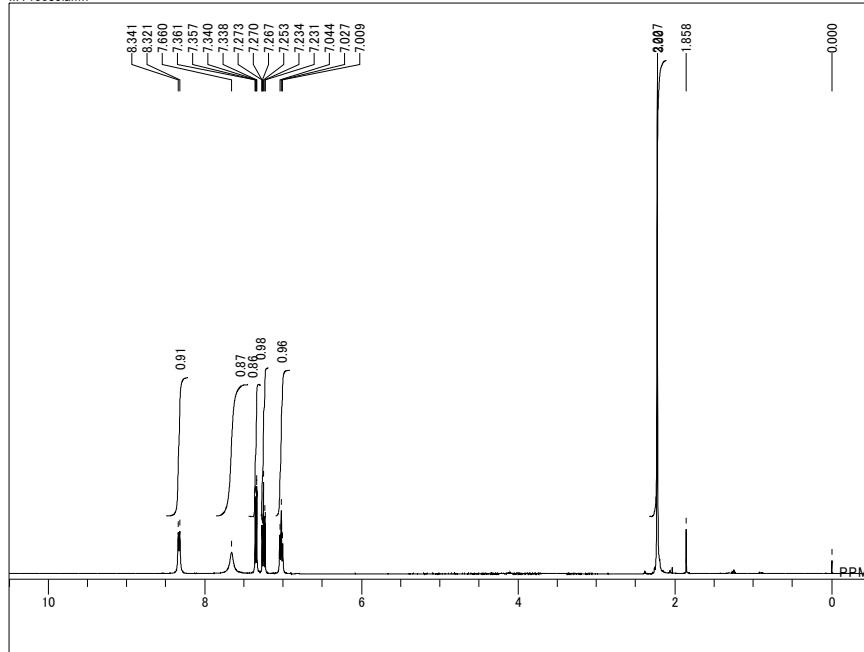


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PD 1.7920 sec
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IRNUC 1H
CTEMP 22.0 c
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EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28



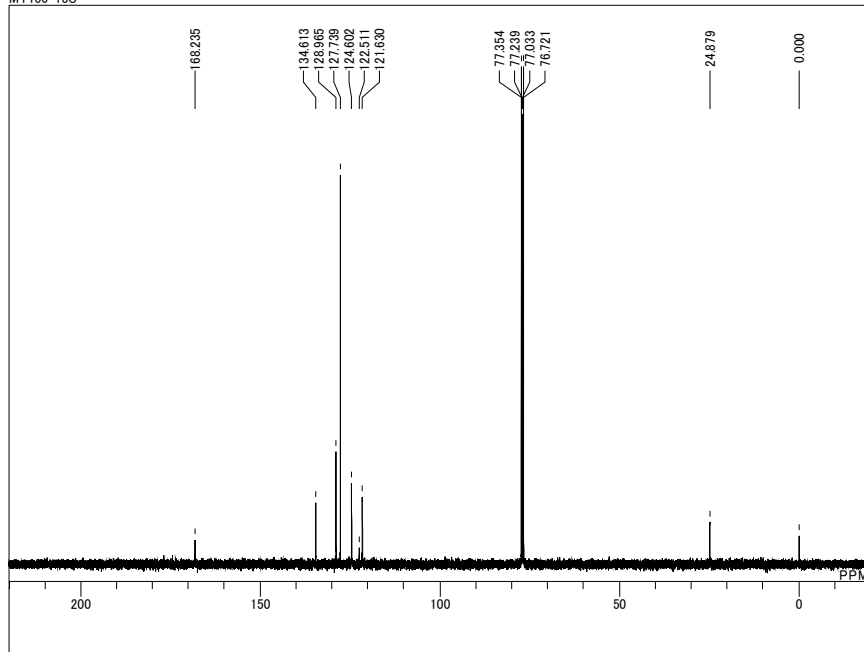
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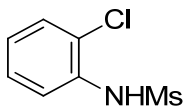


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OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
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PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 25.3 c
SLVNT CDCL3
EXREF 0.00 ppm
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RGAIN 12

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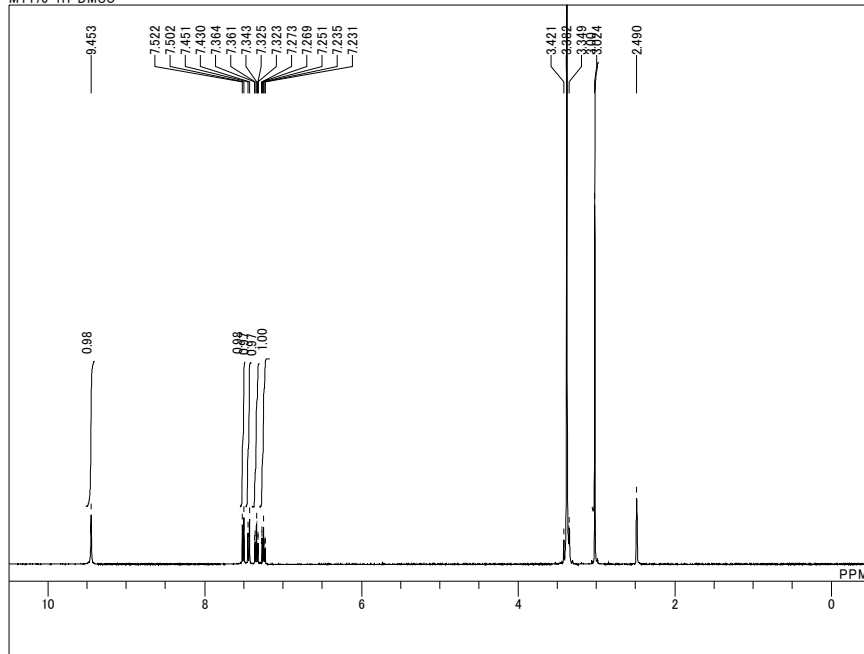


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PD 1.7920 sec
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IRNUC 1H
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RGAIN 28



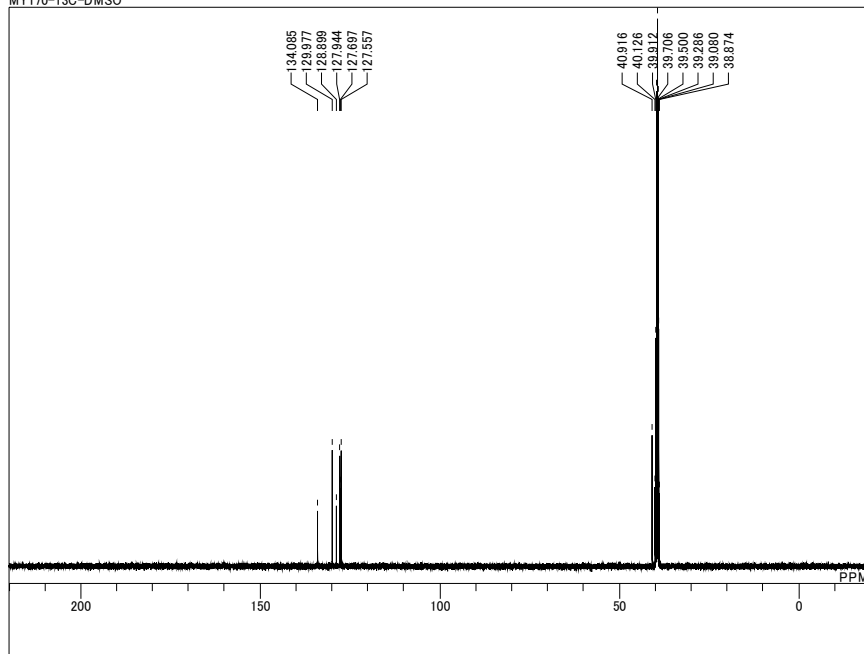
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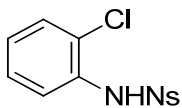


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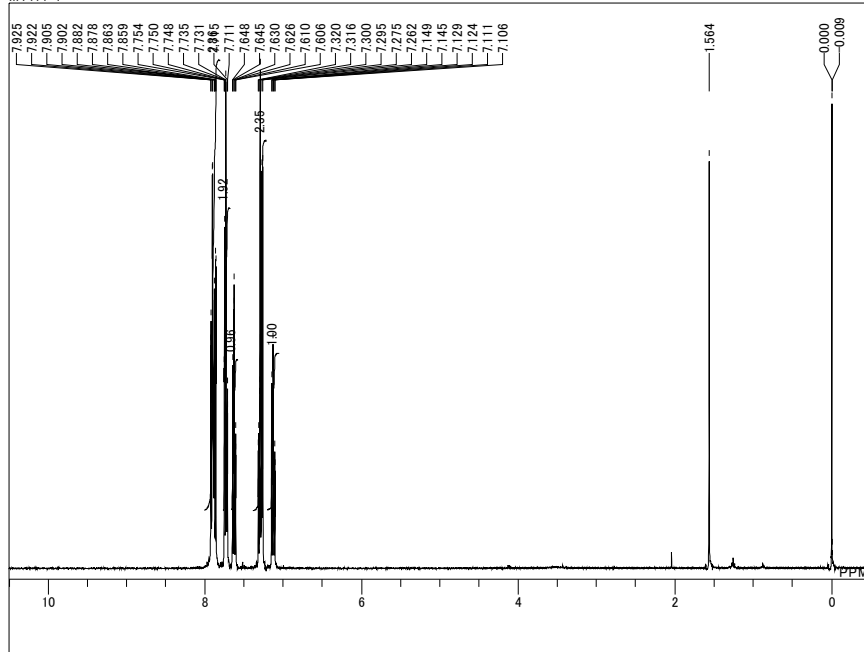


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RGAIN 25



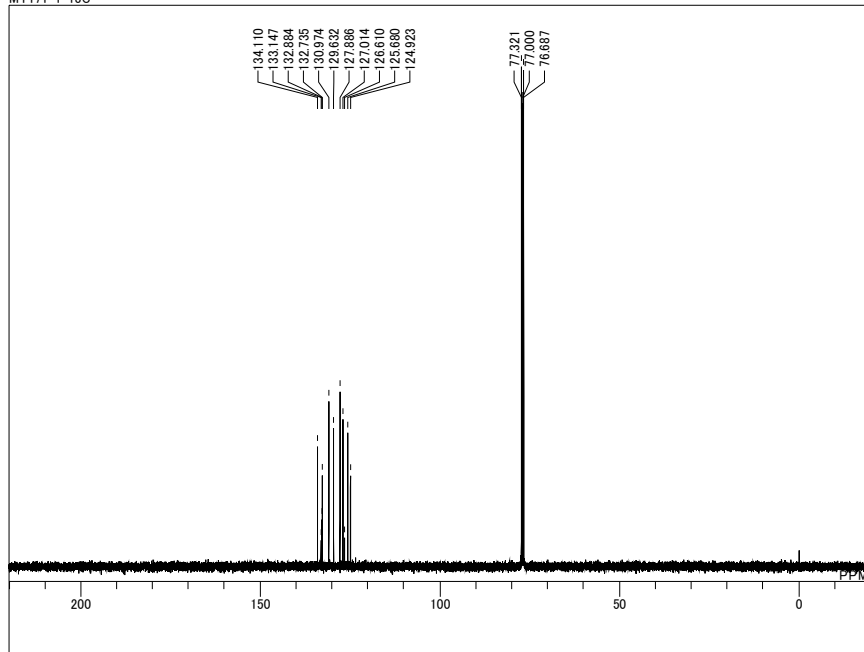
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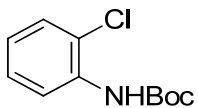


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OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 23.6 c
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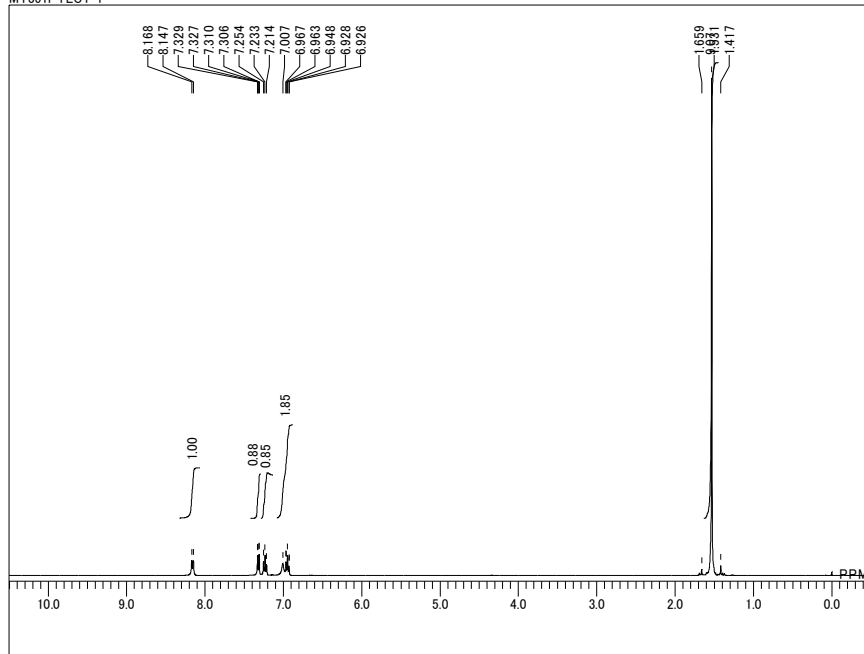


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RGAIN 28



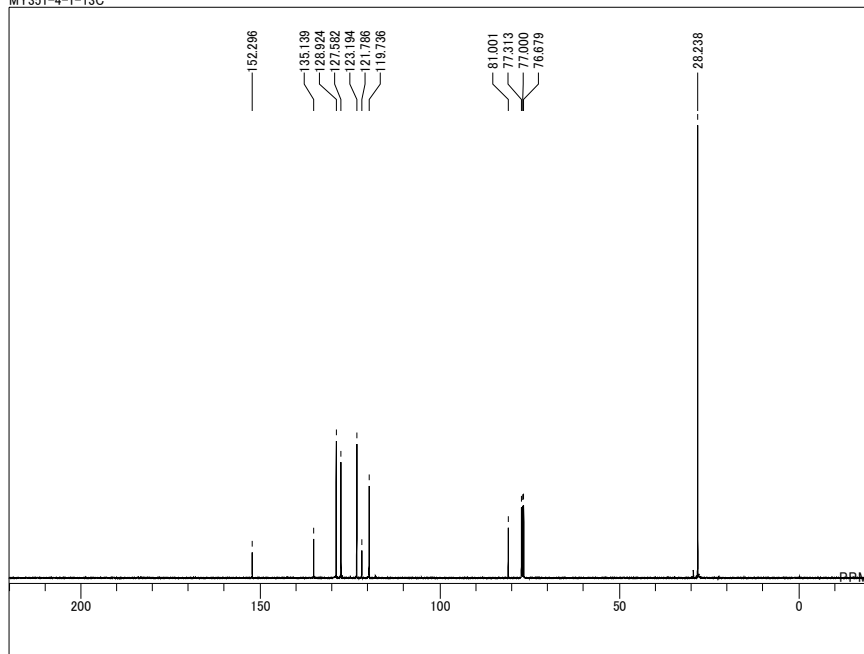
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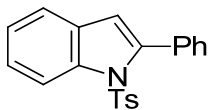


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PW1 6.20 usec
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CTEMP 22.7 c
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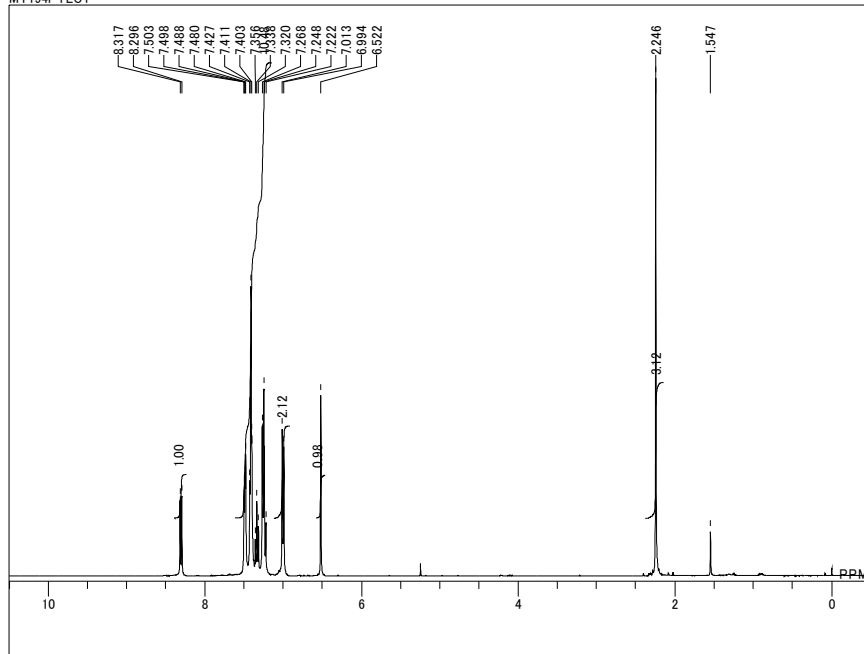


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IRNUC 1H
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BF 120 Hz
RGAIN 28



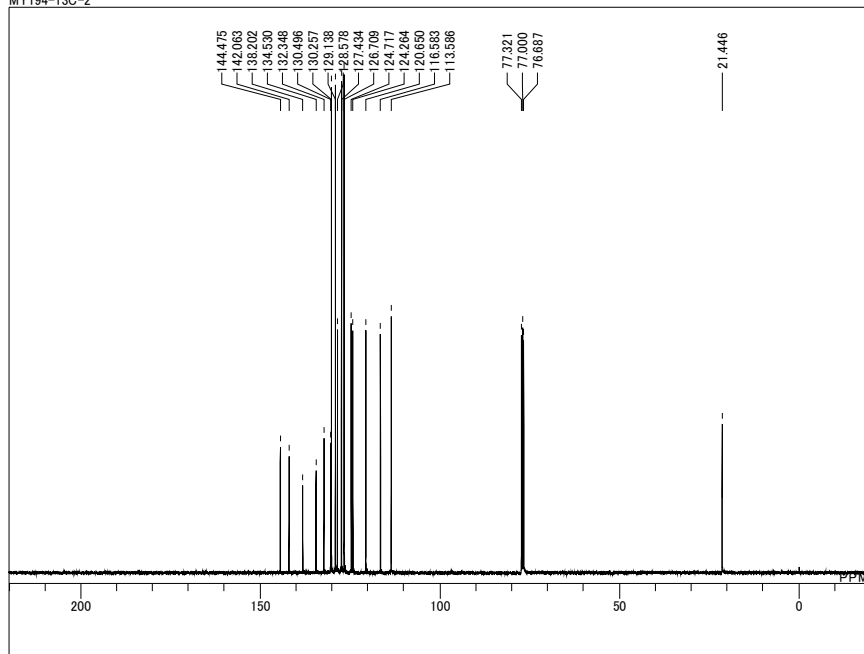
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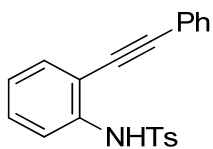


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POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 23.7 c
SLVNT CDCL3
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RGAIN 11

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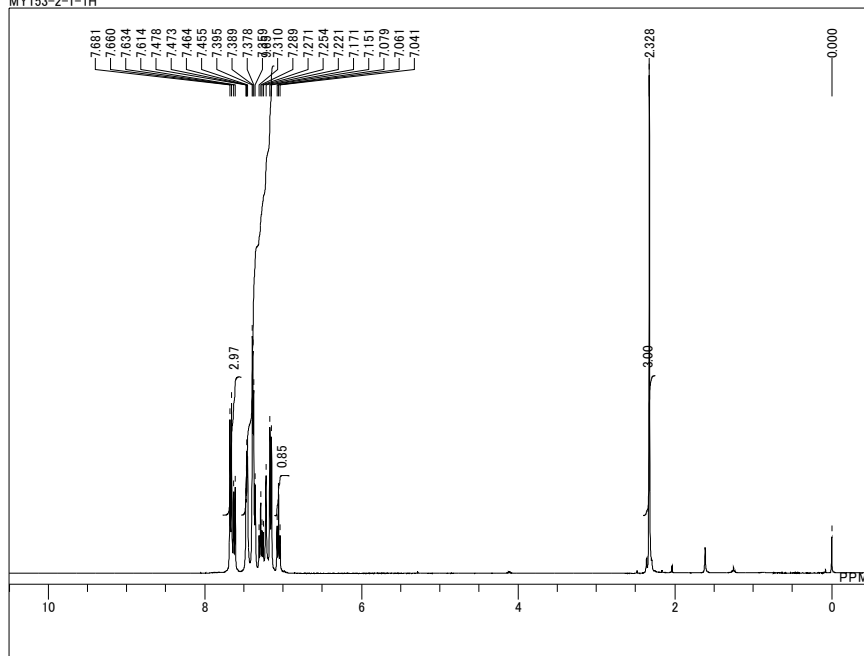


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CTEMP 21.0 c
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BF 120 Hz
RGAIN 26



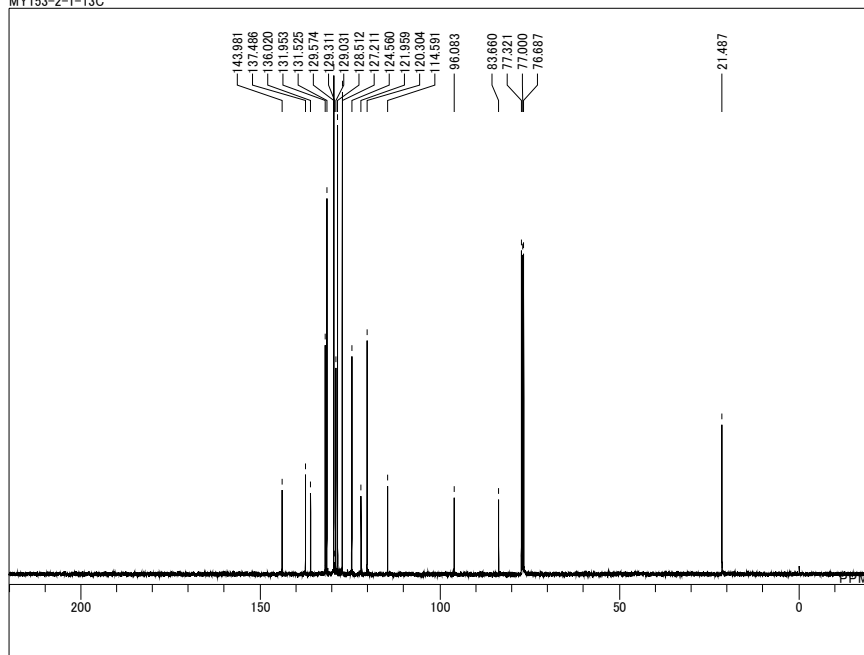
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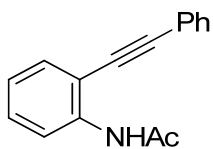


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ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 21.5 c
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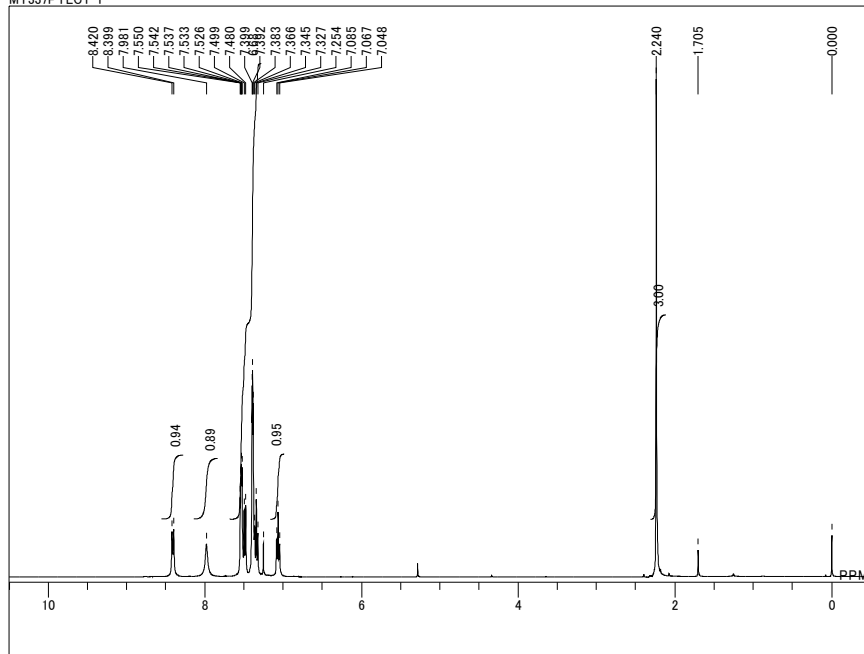


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PW1 5.50 usec
IRNUC 13C
CTEMP 21.5 c
SLVNT CDCL3
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BF 120 Hz
RGAIN 28



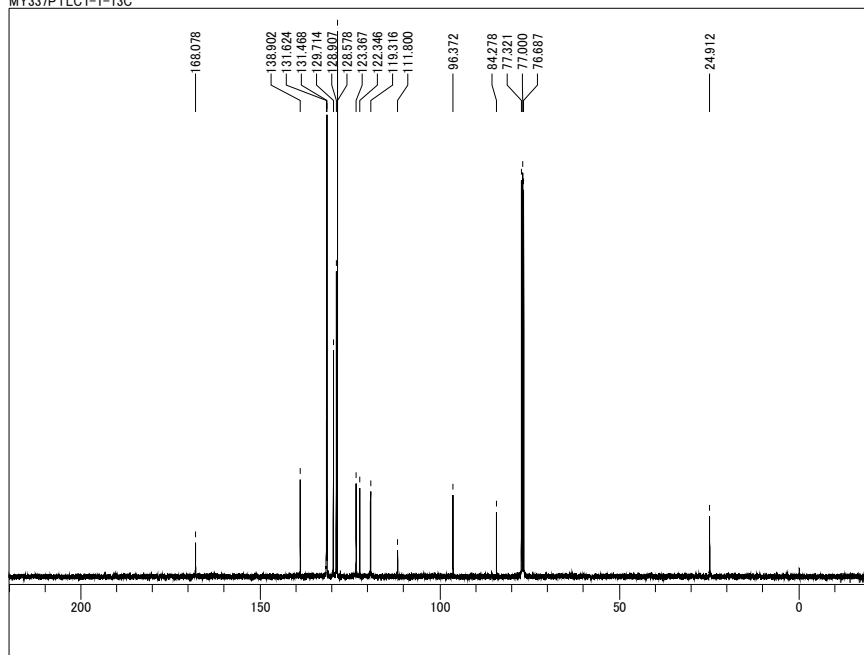
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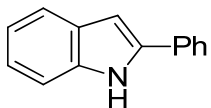


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PW1 6.20 usec
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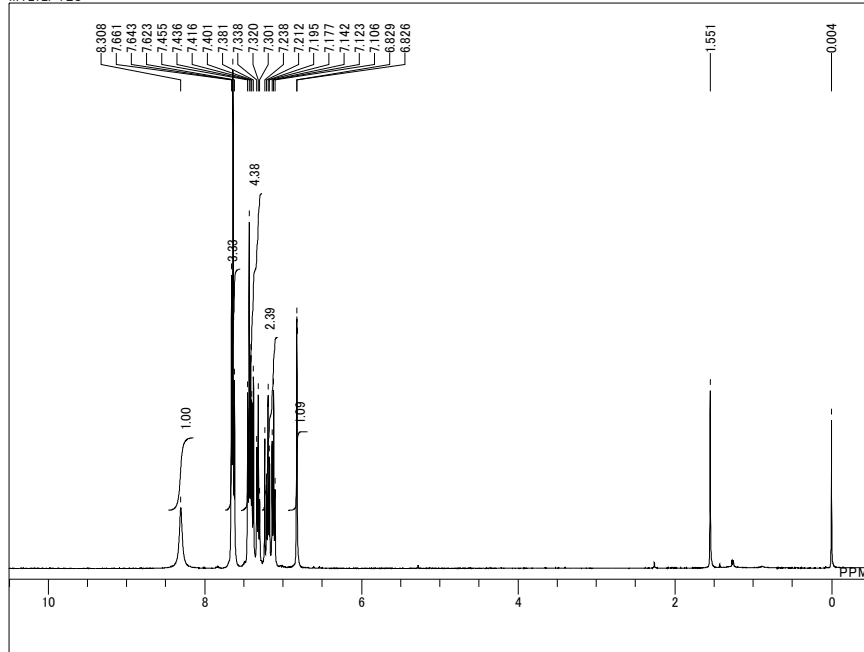


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OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
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PD 1.7920 sec
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IRNUC 1H
CTEMP 24.5 c
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BF 120 Hz
RGAIN 28



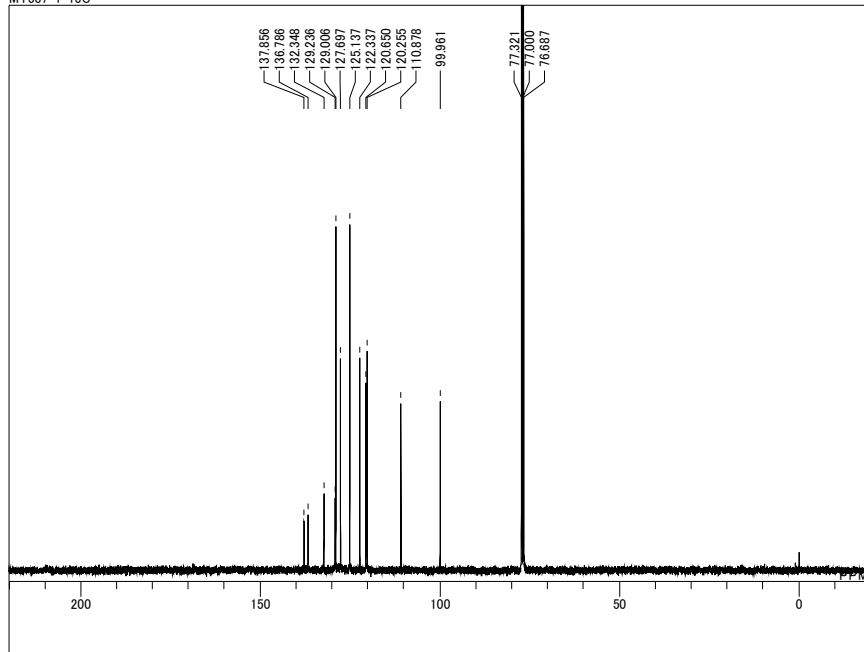
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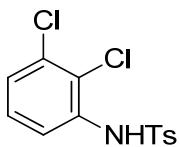


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POINT 16384
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SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 21.5 c
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RGAIN 15

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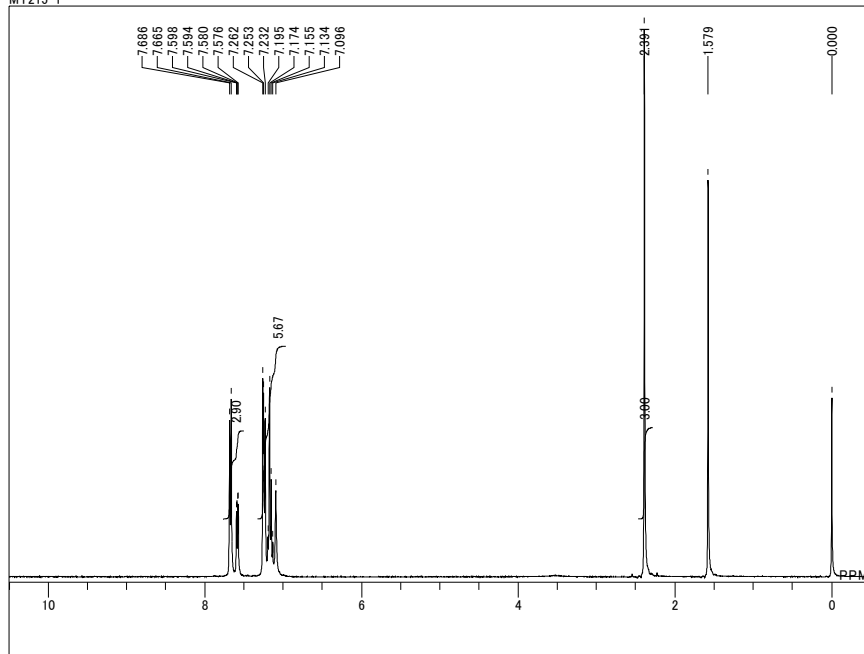


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PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 20.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 120 Hz
RGAIN 28



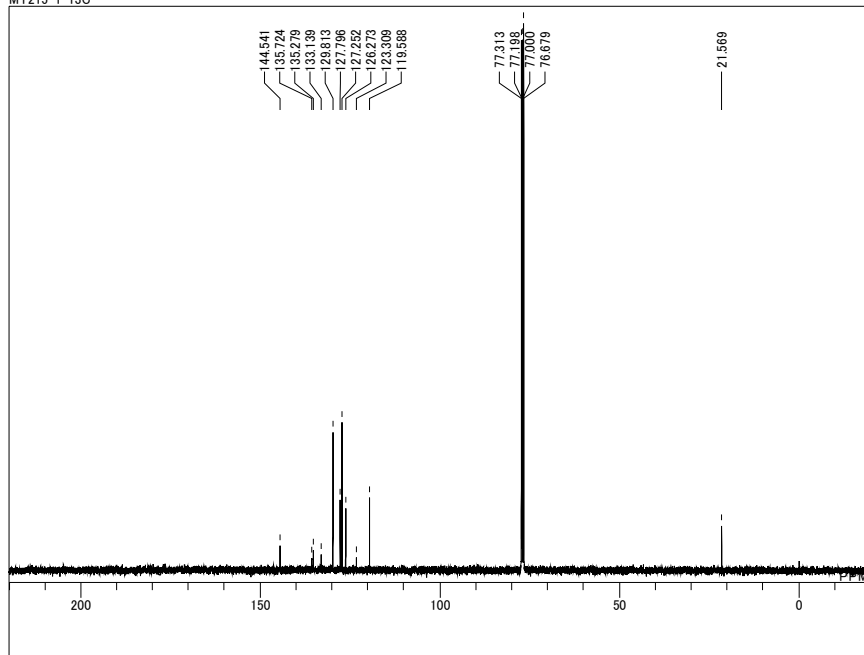
7

F:\NMR\MY215-11NON_E1.als
MY215-1

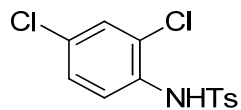


DFILE F:\NMR\MY215-11NON_E1.als
COMNT MY215-1
DATIM Tue Jul 02 09:37:37 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 120 Hz
RGAIN 18

F:\NMR\MY215-1-13C1BCM_E2.als
MY215-1-13C

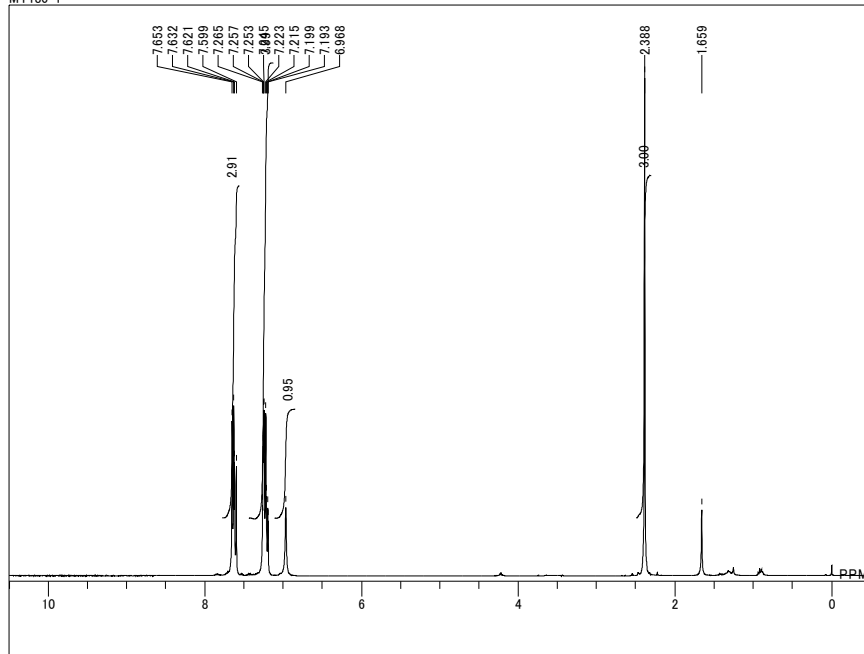


DFILE F:\NMR\MY215-1-13C1BCM_E:
COMNT MY215-1-13C
DATIM Tue Jul 02 23:17:05 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 24.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 120 Hz
RGAIN 26



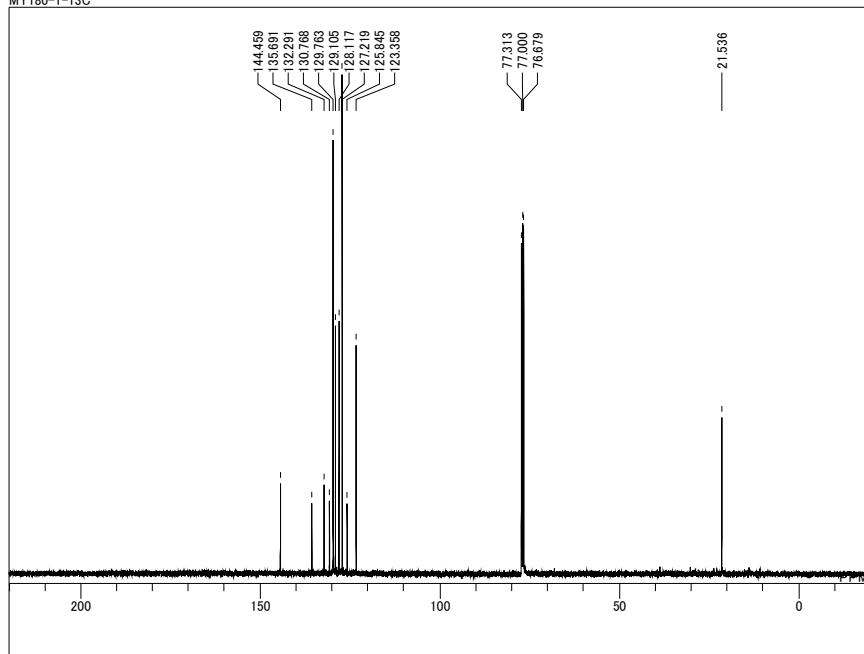
32

F:\NMR\MY180-11NON_E36.als
MY180-1

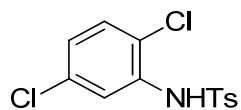


DFILE F:\NMR\MY180-11NON_E36.als
COMNT MY180-1
DATIM Thu May 30 22:48:19 2013
OBNUC 1H
EXMOD NON
OBFREQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 120 Hz
RGAIN 13

F:\NMR\MY180-1-13C1BCM_E4.als
MY180-1-13C

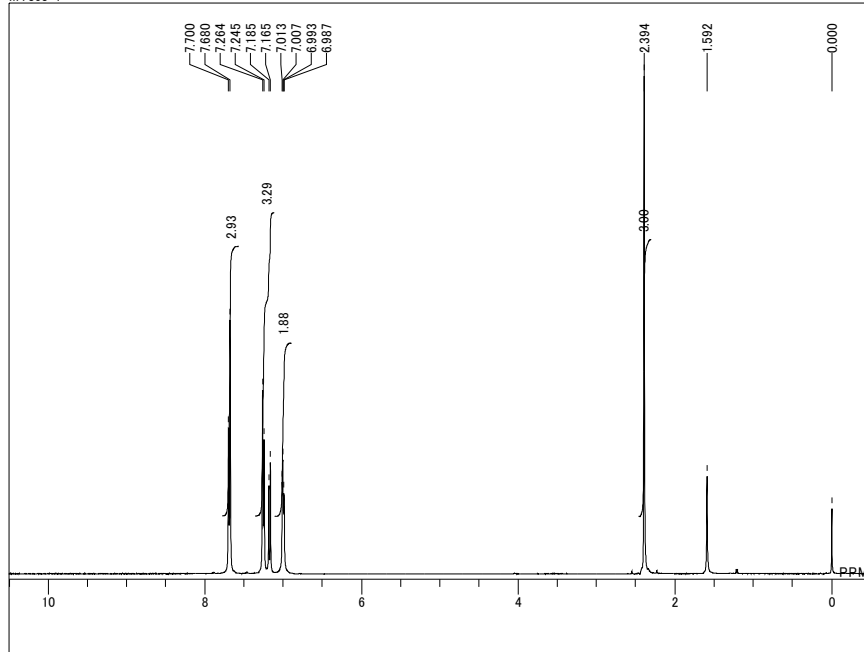


DFILE F:\NMR\MY180-1-13C1BCM_E4.als
COMNT MY180-1-13C
DATIM Fri May 31 01:00:46 2013
OBNUC 13C
EXMOD BCM
OBFREQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 25.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 120 Hz
RGAIN 28



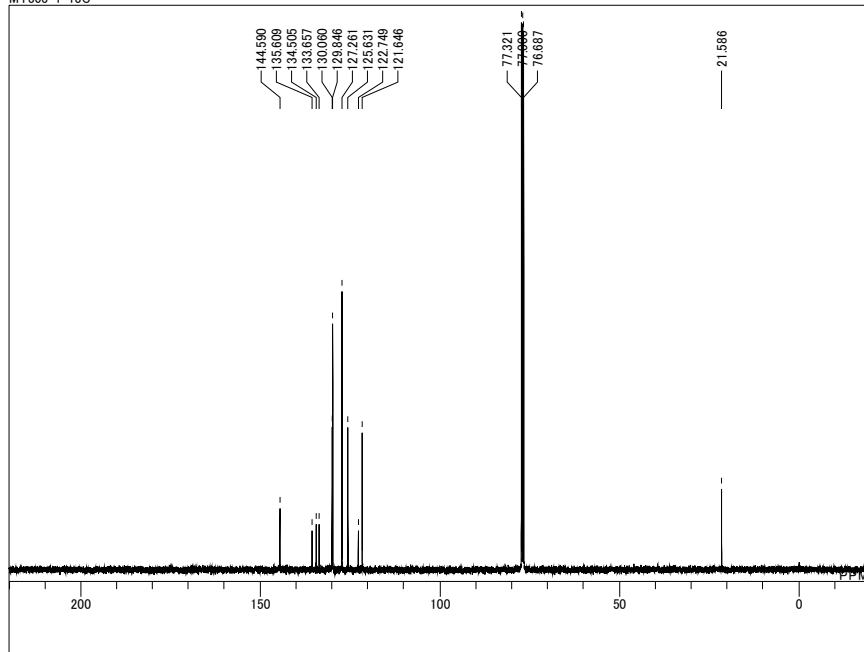
33

F:\NMR\MY303-11NON_E1.als
MY303-1

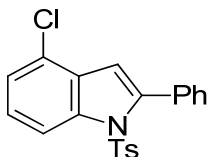


DFILE F:\NMR\MY303-11NON_E1.als
COMNT MY303-1
DATIM Mon Nov 18 09:43:45 2013
OBNUC 1H
EXMOD NON
OBFREQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 18

F:\NMR\MY303-1-13C1BCM_E1.als
MY303-1-13C

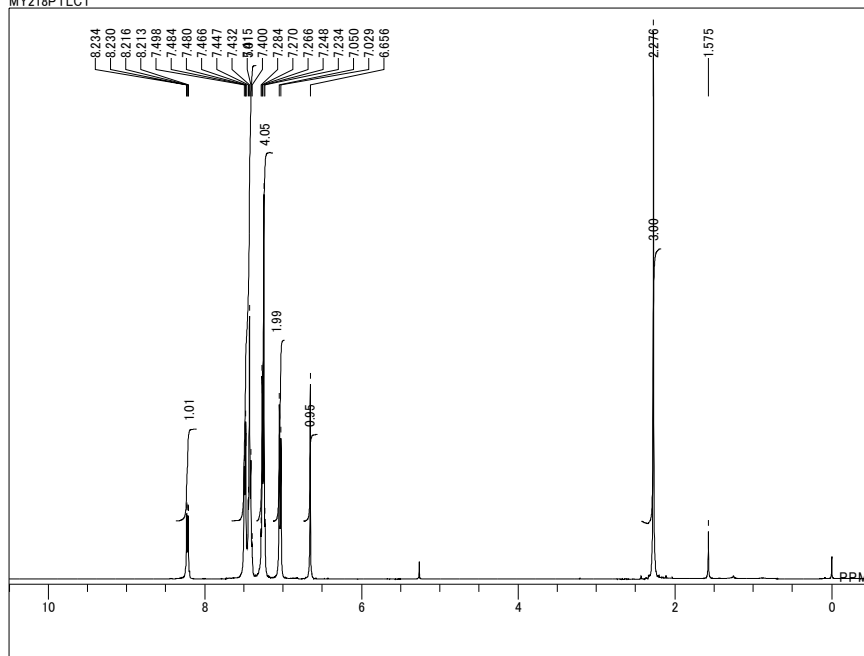


DFILE F:\NMR\MY303-1-13C1BCM_E
COMNT MY303-1-13C
DATIM Mon Nov 18 20:05:02 2013
OBNUC 13C
EXMOD BCM
OBFREQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 27



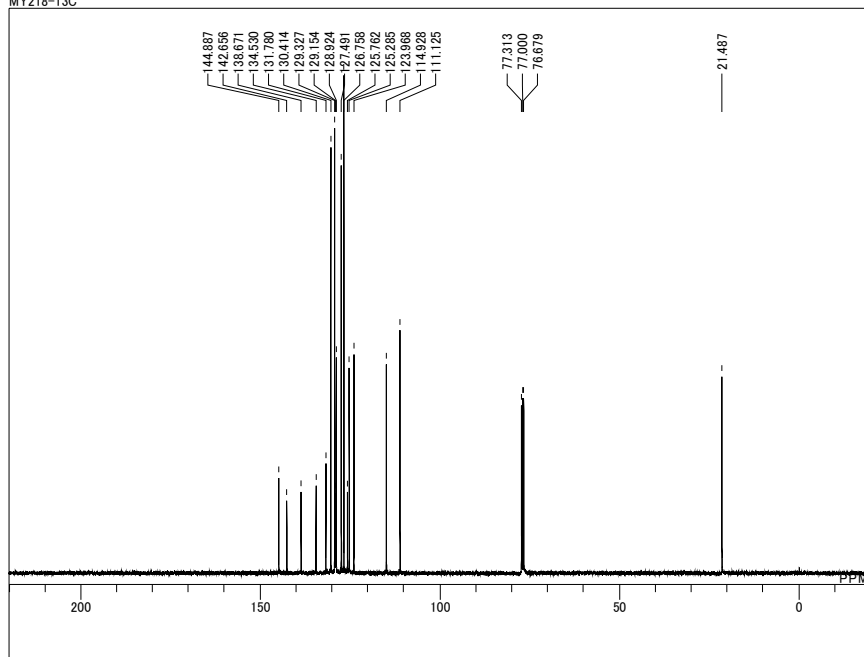
9

F:\NMR\MY218-2-11NON_E26.als
MY218PTLC1

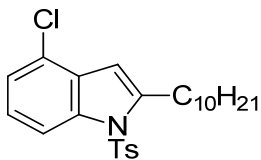


DFILE F:\NMR\MY218-2-11NON_E26.als
COMNT MY218PTLC1
DATIM Wed Jul 03 18:28:15 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 20.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 120 Hz
RGAIN 10

F:\NMR\MY218-13C1BCM_E1.als
MY218-13C

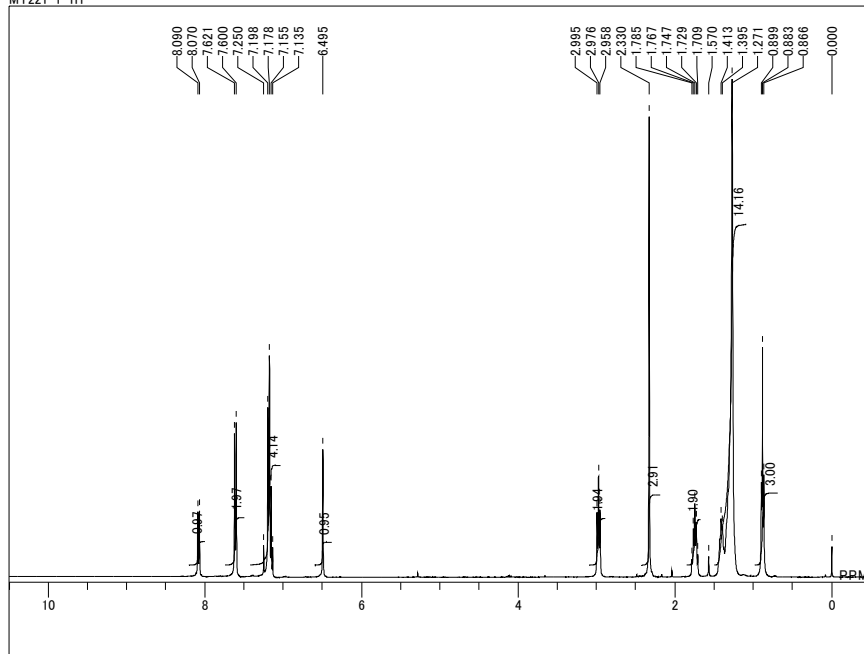


DFILE F:\NMR\MY218-13C1BCM_E1.als
COMNT MY218-13C
DATIM Tue Feb 25 16:24:09 2014
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 120 Hz
RGAIN 28



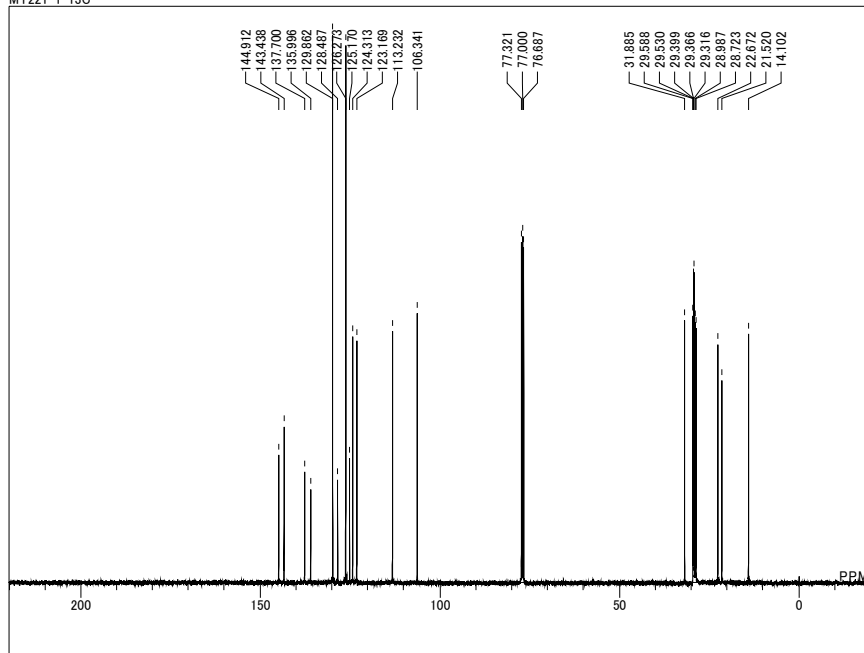
10

F:\NMR\MY221-1-1H1NON_E13.als
MY221-1-1H

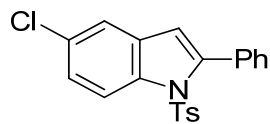


DFILE F:\NMR\MY221-1-1H1NON_E13
COMNT MY221-1-1H
DATIM Wed Feb 26 22:02:56 2014
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 120 Hz
RGAIN 12

F:\NMR\MY221-1-13C1BCM_E15.als
MY221-1-13C

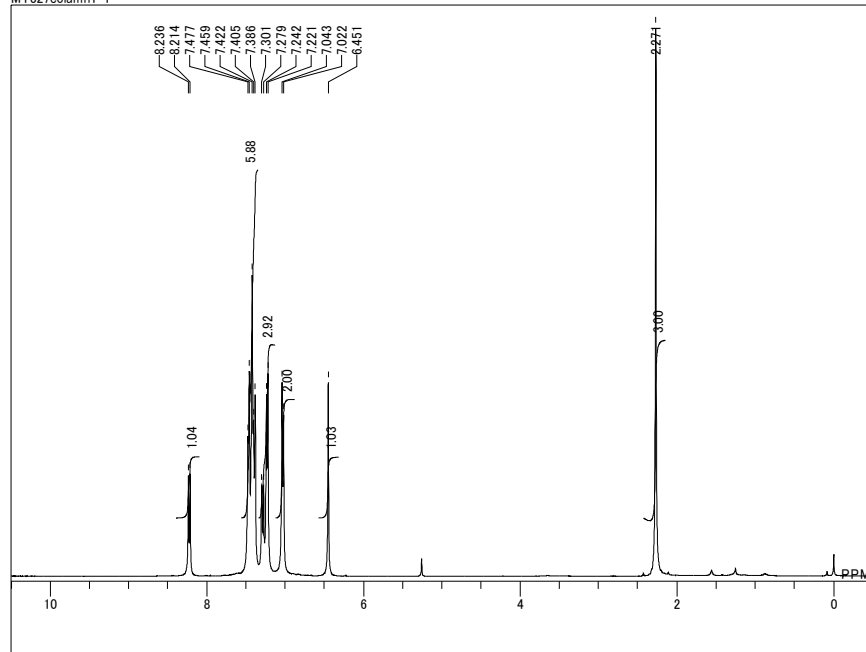


DFILE F:\NMR\MY221-1-13C1BCM_E
COMNT MY221-1-13C
DATIM Wed Feb 26 23:04:02 2014
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 120 Hz
RGAIN 28



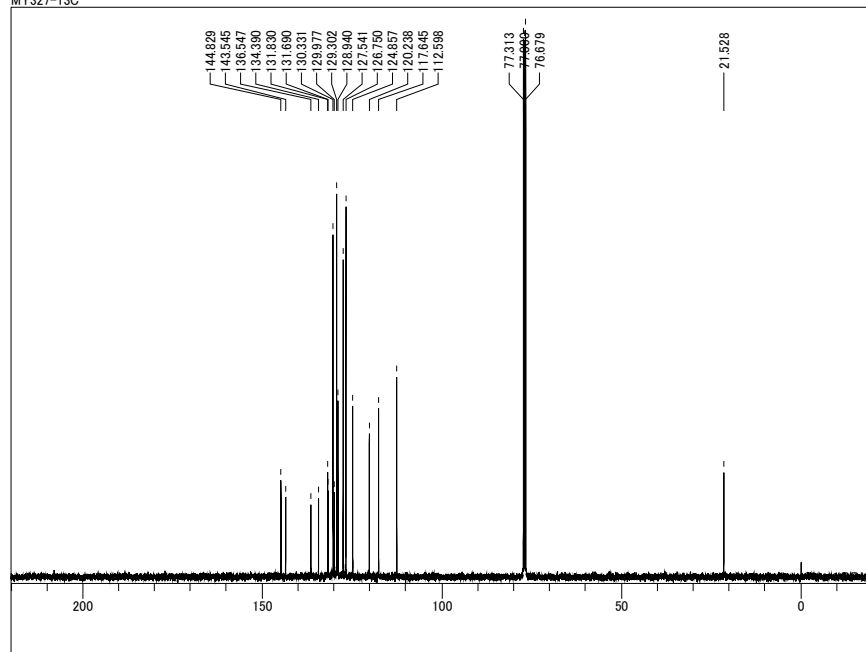
11

F:\NMR\MY327-2-11NON_E1.als
MY327column1-1

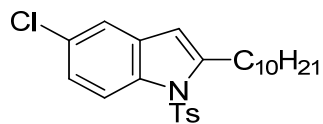


DFILE F:\NMR\MY327-2-11NON_E1.als
COMNT MY327column1-1
DATIM Wed Jan 15 09:12:00 2014
OBNUC 1H
EXMOD NON
OBFREQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQ 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 21.7 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 10

F:\NMR\MY327-13C1BCM_E3.als
MY327-13C

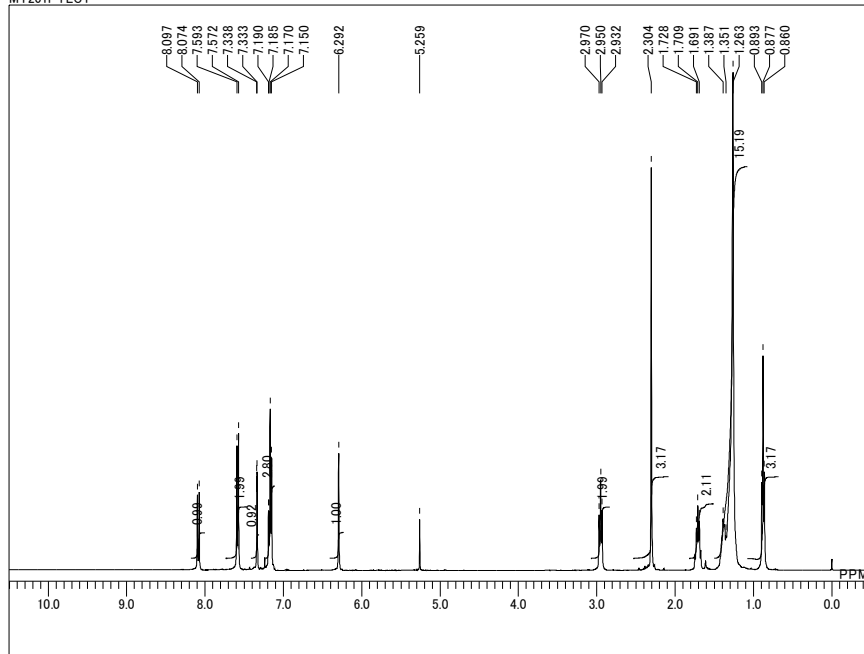


DFILE F:\NMR\MY327-13C1BCM_E3.a
COMNT MY327-13C
DATIM Wed Feb 26 00:30:02 2014
OBNUC 13C
EXMOD BCM
OBFREQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQ 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 13C
CTEMP 21.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 28



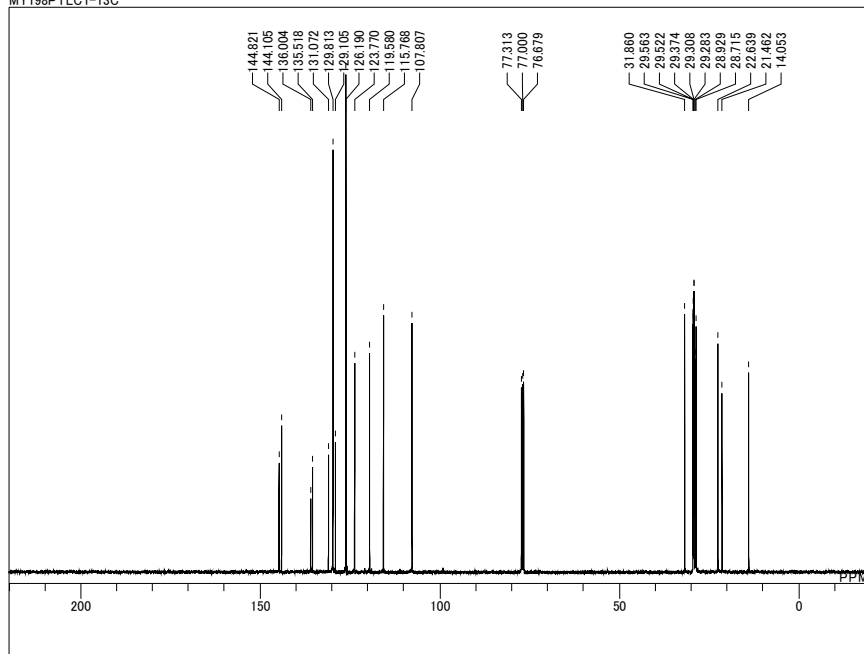
12

F:\NMR\MY201-2-11NON_E3.als
MY201PTLC1

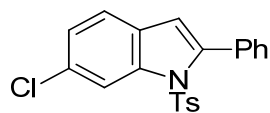


DFILE F:\NMR\MY201-2-11NON_E3.als
COMNT MY201PTLC1
DATIM Mon Jun 17 09:30:18 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 22.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 8

F:\NMR\MY198-2-1-13C1BCM_E1.als
MY198PTLC1-13C

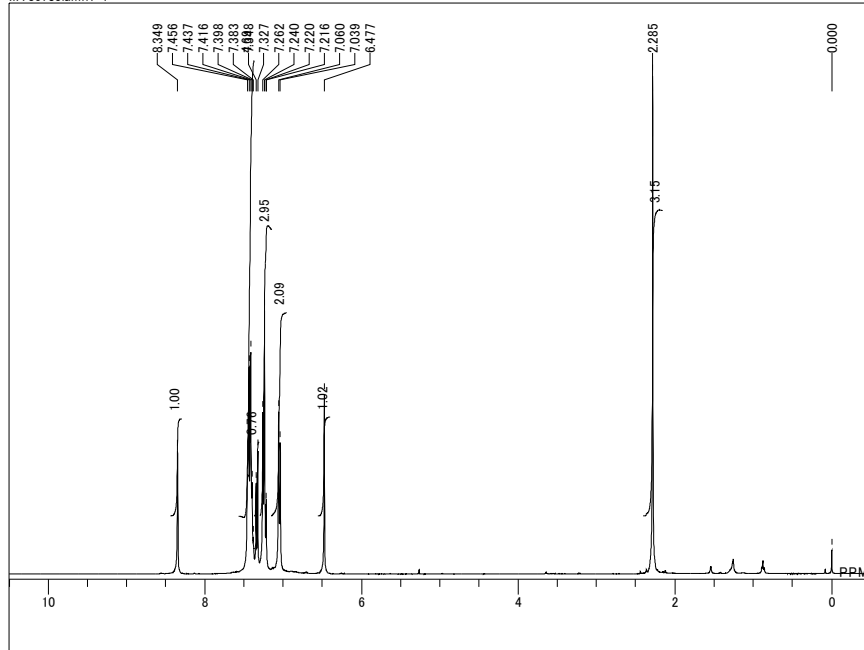


DFILE F:\NMR\MY198-2-1-13C1BCM
COMNT MY198PTLC1-13C
DATIM Thu Jun 13 11:07:25 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 25.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 27



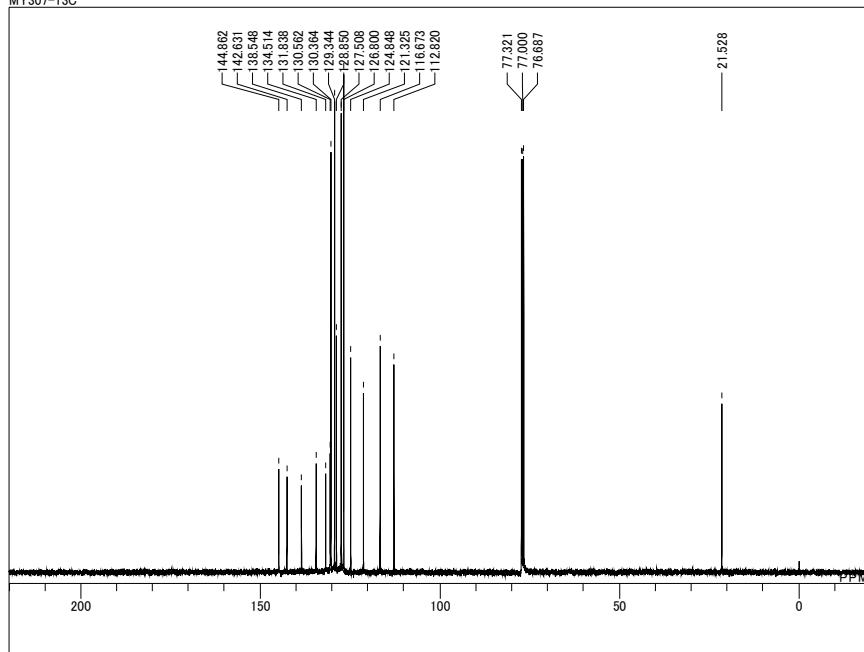
13

F:\NMR\MY307-2-11NON_E6.als
MY307column1-1

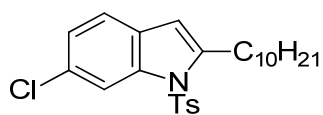


DFILE F:\NMR\MY307-2-11NON_E6.als
COMNT MY307column1-1
DATIM Wed Nov 20 13:48:36 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 120 Hz
RGAIN 14

F:\NMR\MY307-13C1BCM_E2.als
MY307-13C

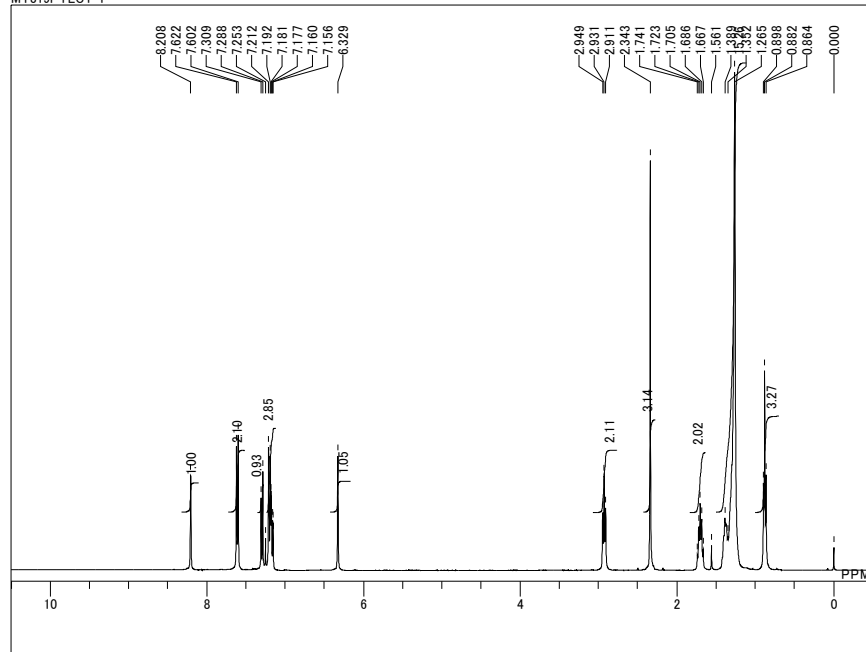


DFILE F:\NMR\MY307-13C1BCM_E2.a
COMNT MY307-13C
DATIM Tue Feb 25 23:33:12 2014
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 120 Hz
RGAIN 28



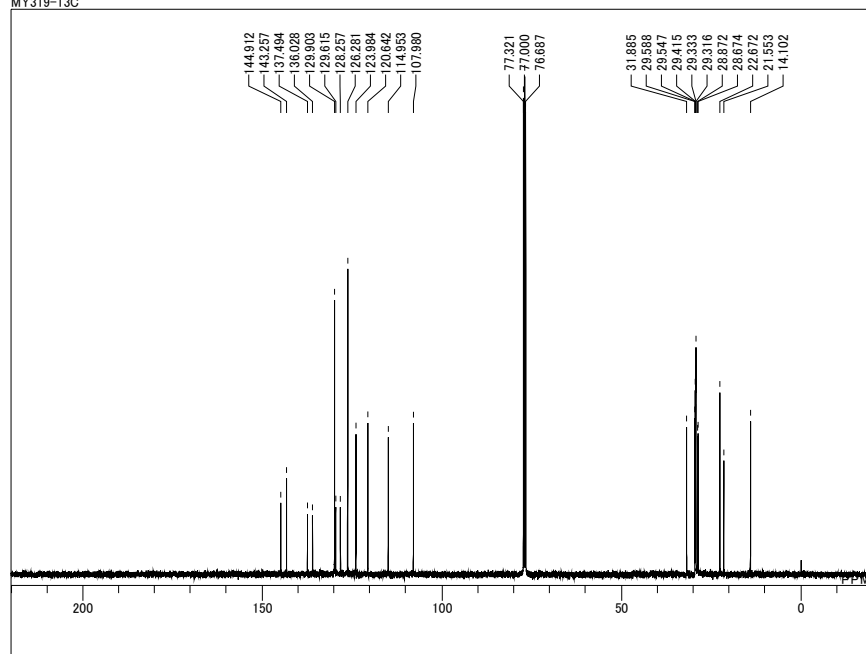
14

F:\NMR\MY319-3-11NON_E3.als
MY319PTLC1-1

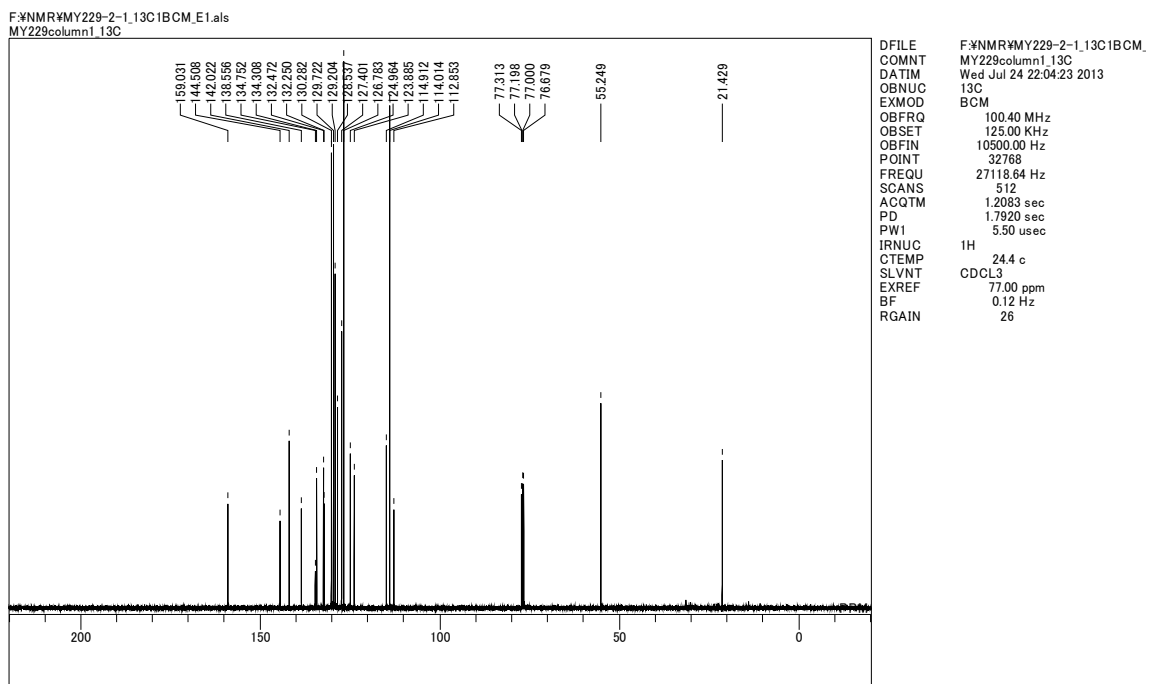


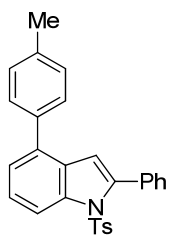
DFILE F:\NMR\MY319-3-11NON_E3.als
COMNT MY319PTLC1-1
DATIM Sat Dec 21 14:19:34 2013
OBNUC 1H
EXMOD NON
OBFREQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 1.20 Hz
RGAIN 10

F:\NMR\MY319-13C1BCM_E1.als
MY319-13C



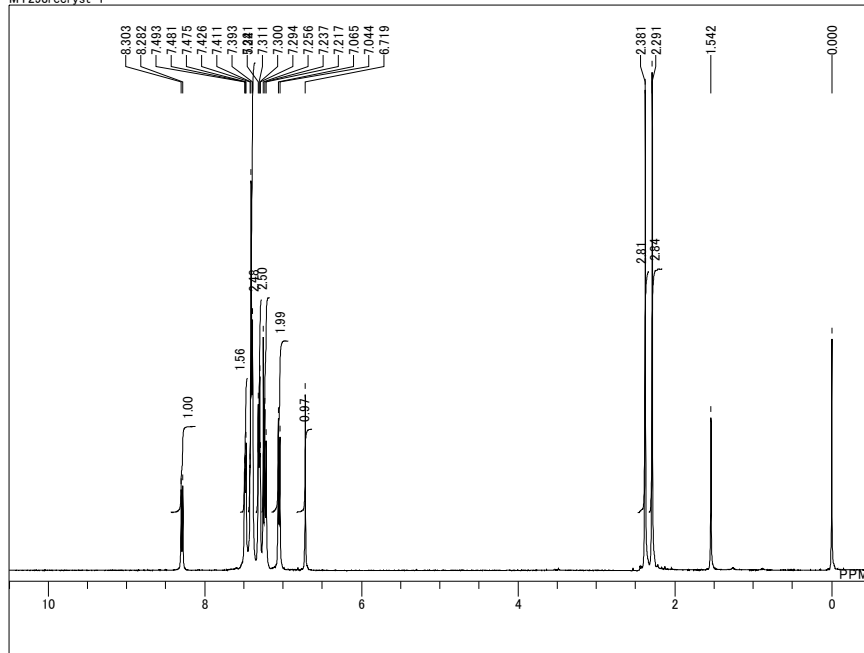
DFILE F:\NMR\MY319-13C1BCM_E1.a
COMNT MY319-13C
DATIM Tue Feb 25 22:32:40 2014
OBNUC 13C
EXMOD BCM
OBFREQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 28





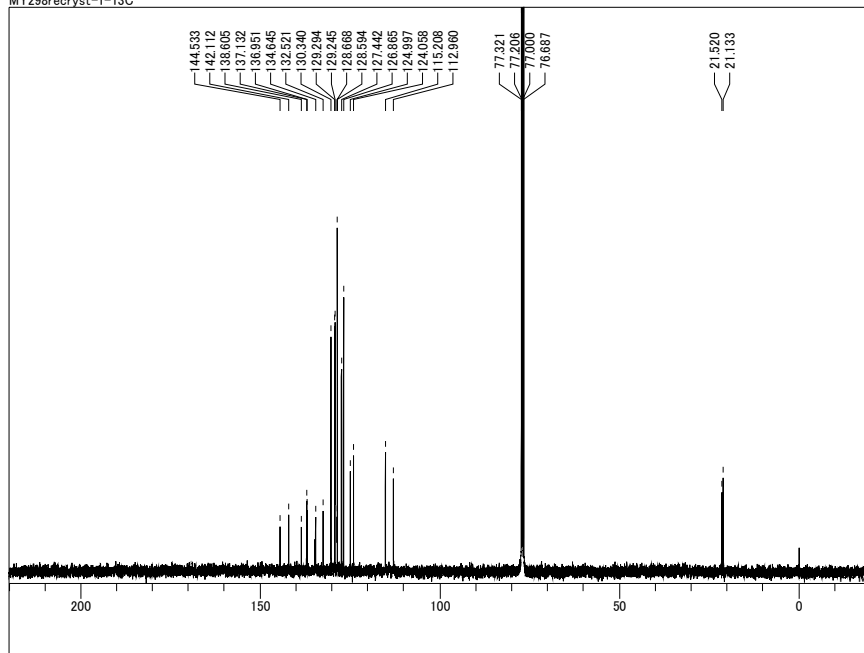
17

F:\NMR\MY298-41NON_E1.als
MY298recryst-1

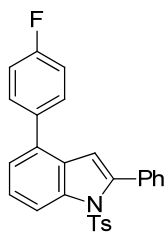


DFILE F:\NMR\MY298-41NON_E1.als
COMNT MY298recryst-1
DATIM Fri Dec 13 09:43:29 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 21.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 120 Hz
RGAIN 17

F:\NMR\MY298-4-13C1BCM_E3.als
MY298recryst-1-13C

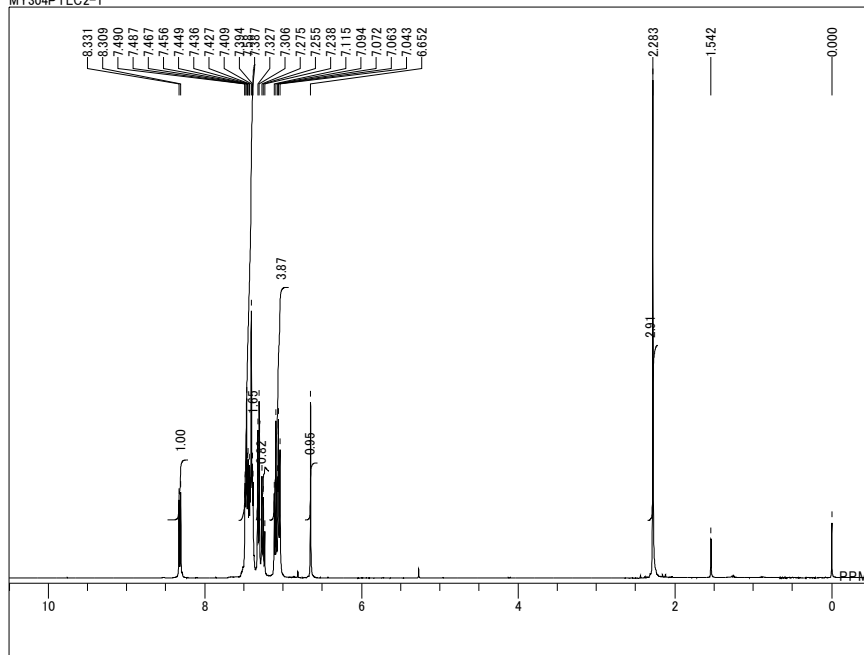


DFILE F:\NMR\MY298-4-13C1BCM_E3.als
COMNT MY298recryst-1-13C
DATIM Sat Dec 14 00:27:23 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 2048
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 120 Hz
RGAIN 27



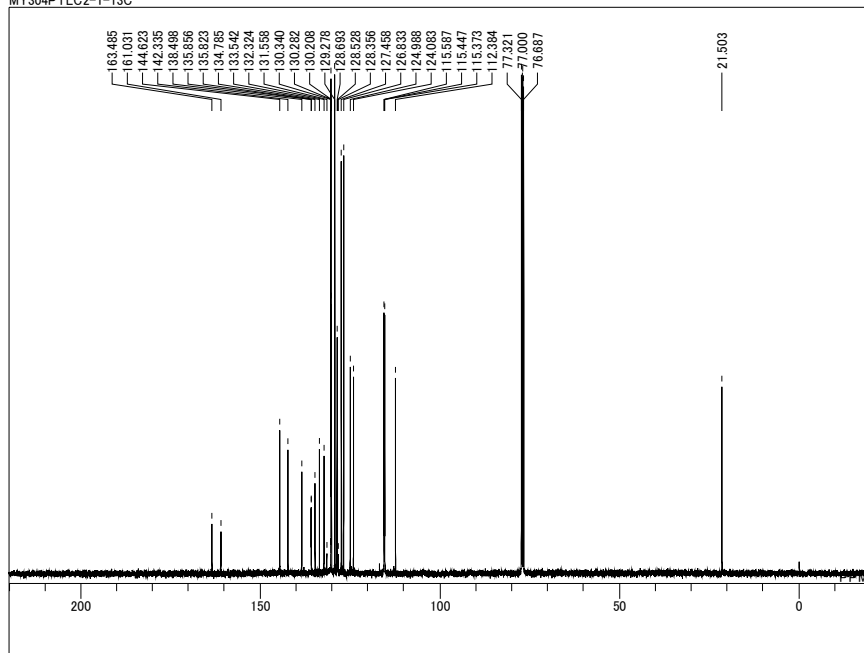
18

F:\NMR\WY304-3-11NON_E1.als
MY304PTLC2-1

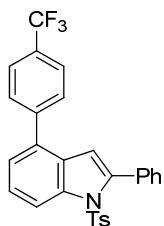


DFILE F:\NMR\WY304-3-11NON_E1.als
COMNT MY304PTLC2-1
DATIM Mon Nov 25 10:06:43 2013
OBNUC 1H
EXMOD NON
OBFREQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 120 Hz
RGAIN 14

F:\NMR\WY304-3-1-13C1BCM_E1.als
MY304PTLC2-1-13C

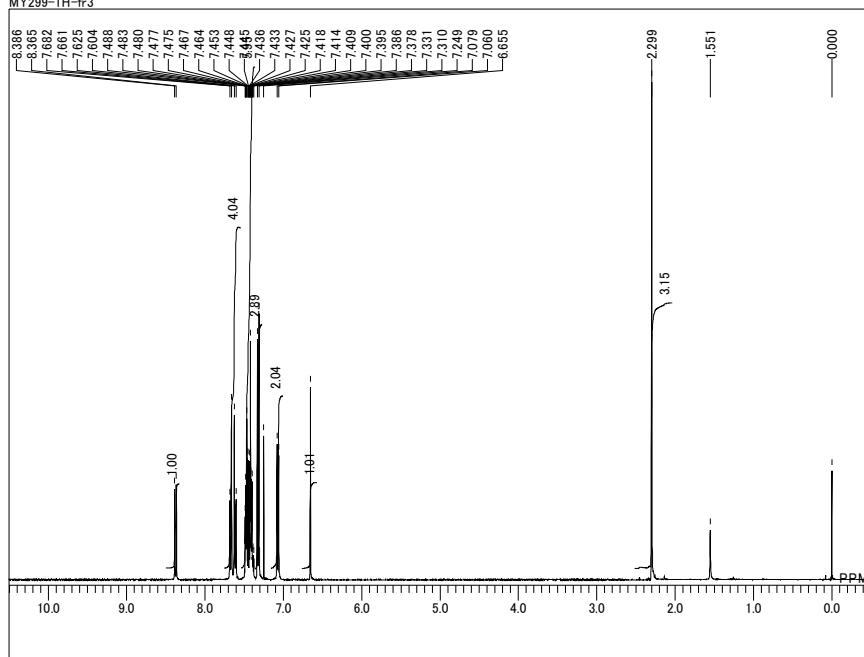


DFILE F:\NMR\WY304-3-1-13C1BCM
COMNT MY304PTLC2-1-13C
DATIM Tue Nov 26 00:34:43 2013
OBNUC 13C
EXMOD BCM
OBFREQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 120 Hz
RGAIN 28



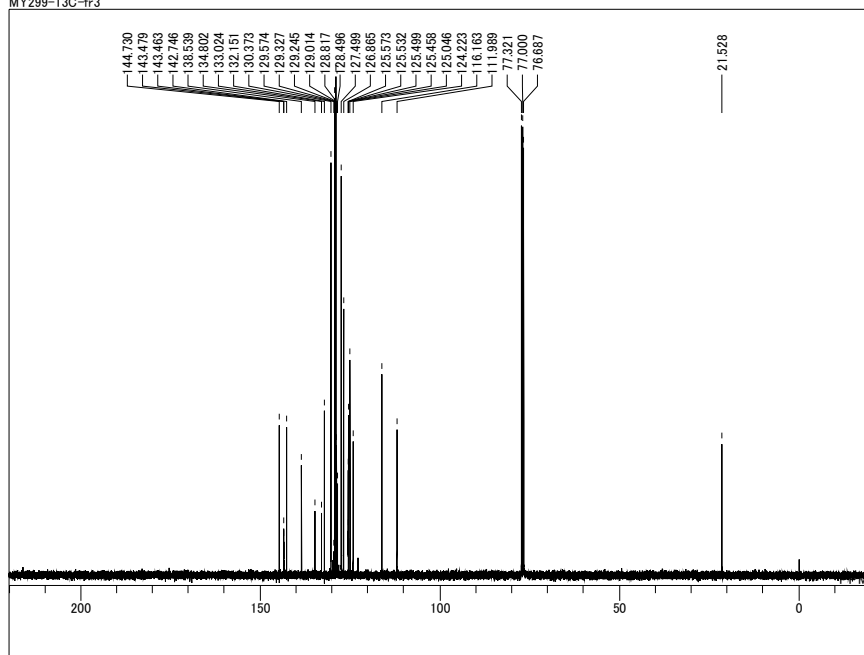
19

F:\NMR\MY299-1H-fr31NON_E2.als
MY299-1H-fr3

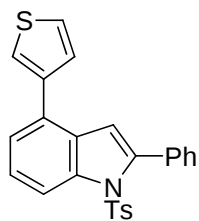


DFILE F:\NMR\MY299-1H-fr31NON_E
COMNT MY299-1H-fr3
DATIM Sat Mar 01 17:44:14 2014
OBNUC 1H
EXMOD NON
OBFREQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 22.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 15

F:\NMR\MY299-13C-fr31BCM_E4.als
MY299-13C-fr3

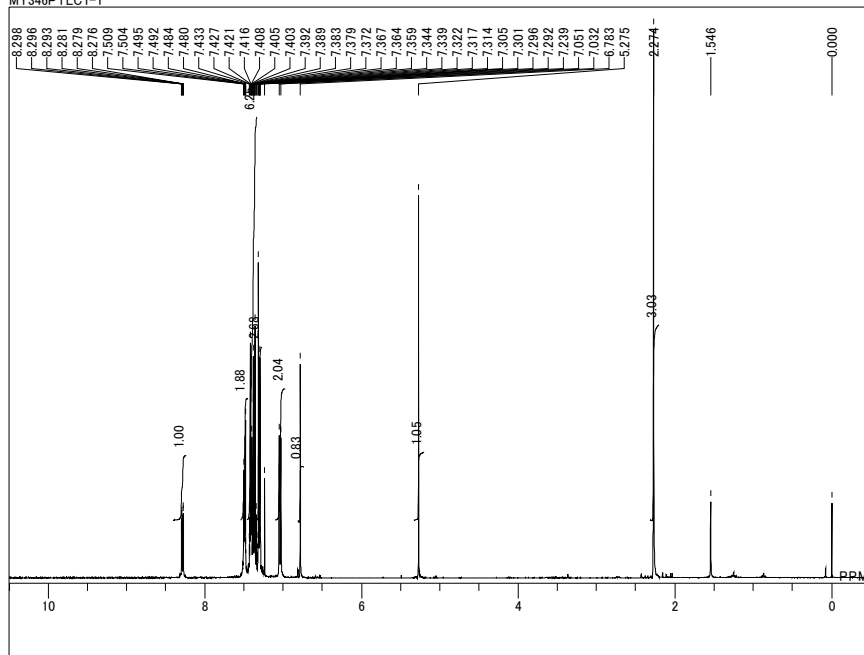


DFILE F:\NMR\MY299-13C-fr31BCM_E
COMNT MY299-13C-fr3
DATIM Sat Mar 01 19:36:45 2014
OBNUC 13C
EXMOD BCM
OBFREQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.3 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28



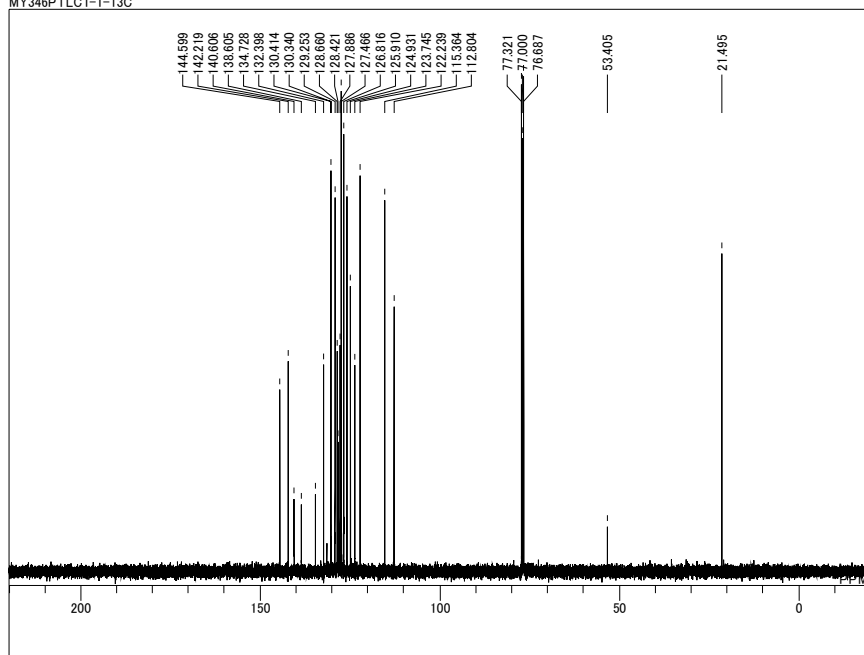
20

F:\NMR\MY346-3-11NON_E1.als
MY346PTLC1-1

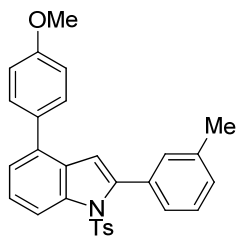


DFILE F:\NMR\MY346-3-11NON_E1.als
COMNT MY346PTLC1-1
DATIM Tue Feb 04 08:55:38 2014
OBNUC 1H
EXMOD NON
OBFREQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 23.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 14

F:\NMR\MY346-3-1-13C1BCM_E5.als
MY346PTLC1-1-13C

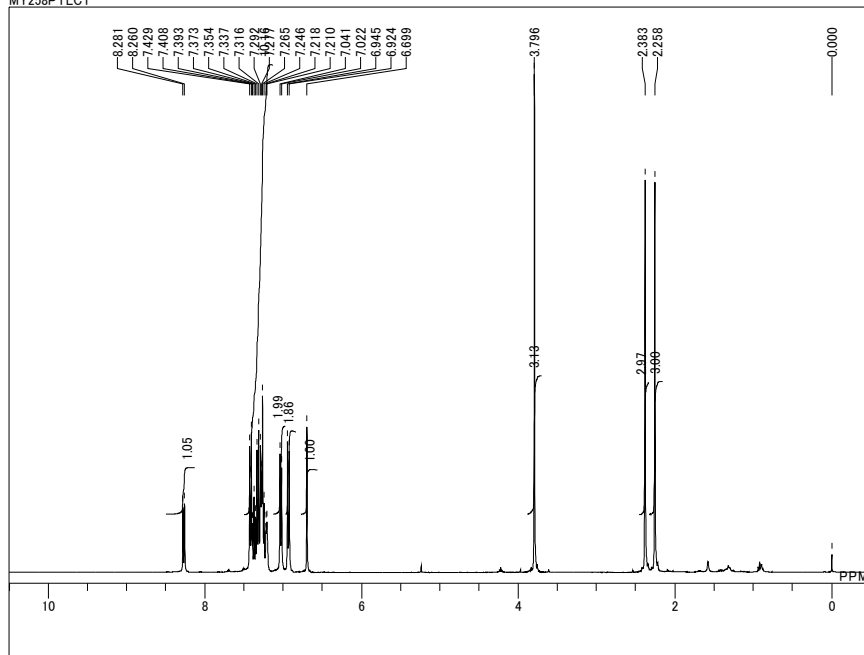


DFILE F:\NMR\MY346-3-1-13C1BCM
COMNT MY346PTLC1-1-13C
DATIM Tue Feb 04 09:48:44 2014
OBNUC 13C
EXMOD BCM
OBFREQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 512
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 13C
CTEMP 23.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28



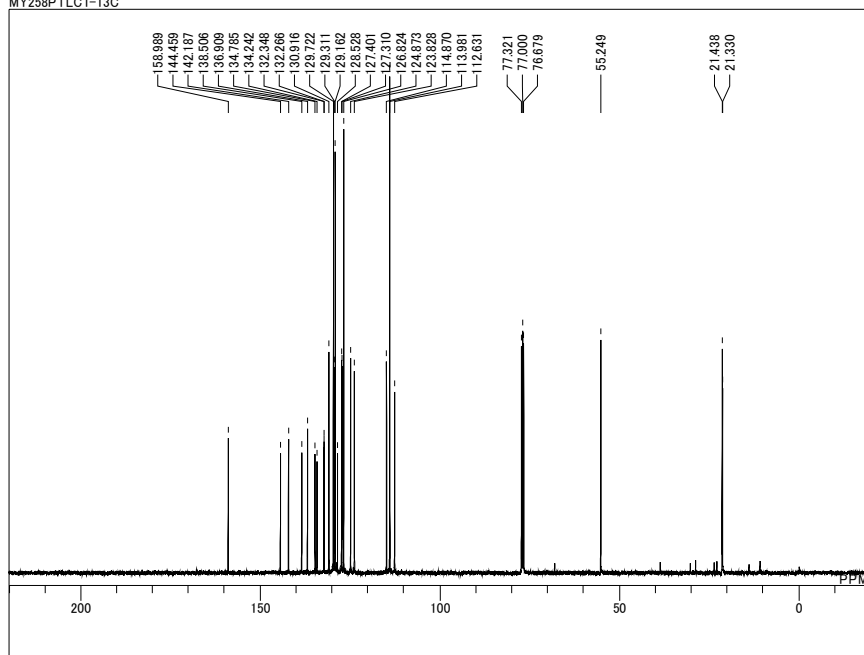
21

F:\NMR\WY258-3-11NON_E12.als
MY258PTLC1

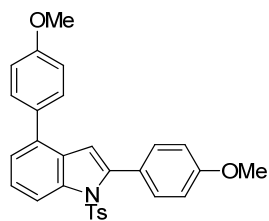


DFILE F:\NMR\WY258-3-11NON_E12.als
COMNT MY258PTLC1
DATIM Fri Sep 13 16:10:03 2013
OBNUC 1H
EXMOD NON
OBFREQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 120 Hz
RGAIN 11

F:\NMR\WY258-3-1-13C1BCM_E2.als
MY258PTLC1-13C

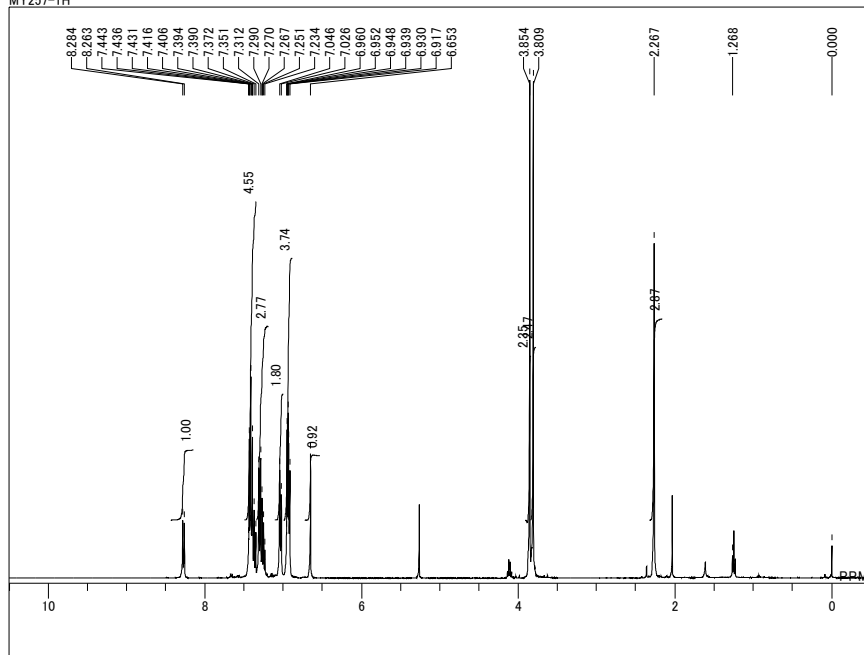


DFILE F:\NMR\WY258-3-1-13C1BCM
COMNT MY258PTLC1-13C
DATIM Sat Sep 14 00:08:02 2013
OBNUC 13C
EXMOD BCM
OBFREQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 120 Hz
RGAIN 26



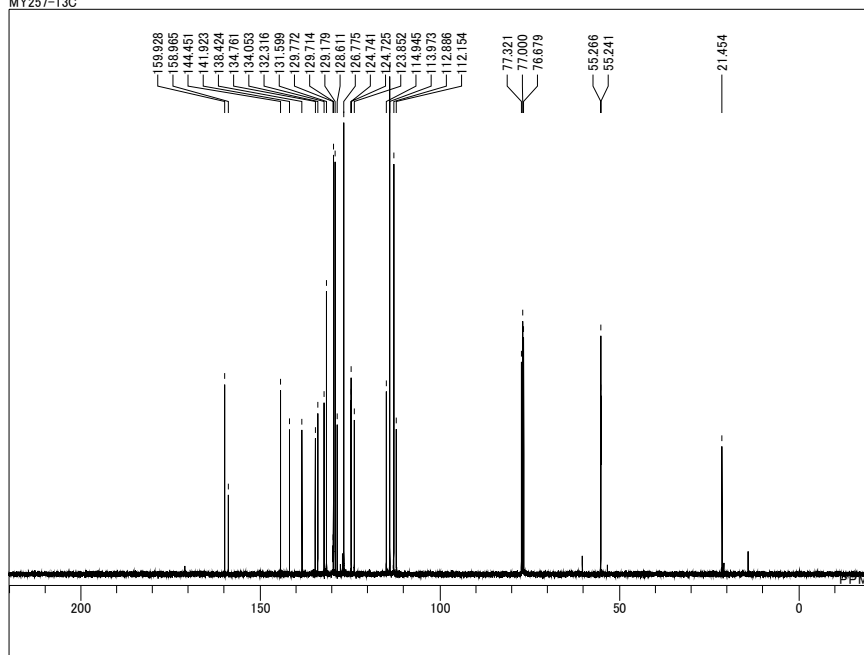
22

F:\NMR\MY257-1H1NON_E1.als
MY257-1H

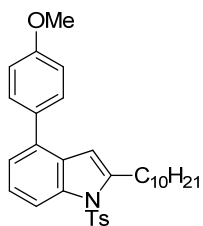


DFILE F:\NMR\MY257-1H1NON_E1.als
COMNT MY257-1H
DATIM Tue Feb 25 10:02:37 2014
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 13

F:\NMR\MY257-13C1BCM_E4.als
MY257-13C

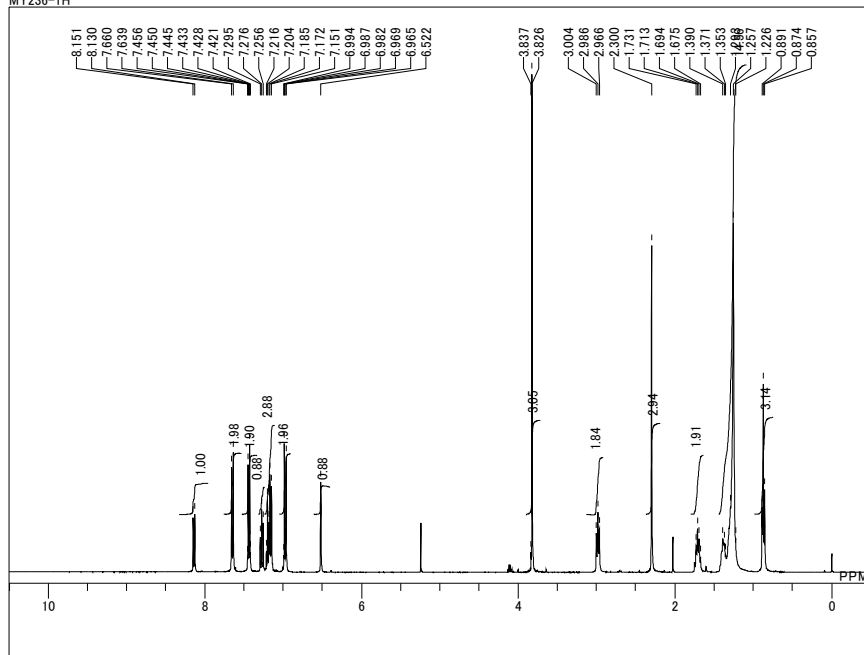


DFILE F:\NMR\MY257-13C1BCM_E4.a
COMNT MY257-13C
DATIM Tue Feb 25 12:54:33 2014
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 28



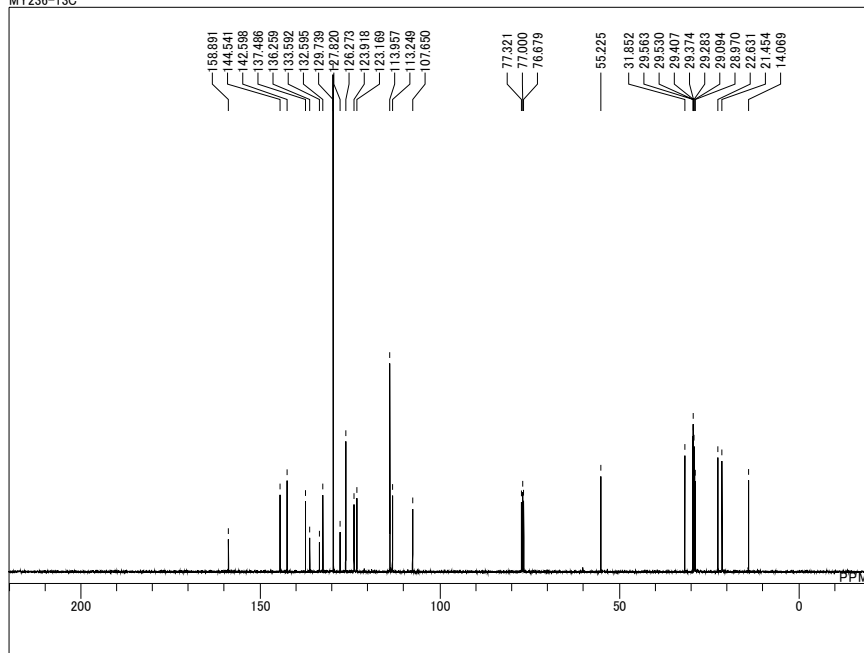
23

F:\NMR\MY236-1H1NON_E2.als
MY236-1H

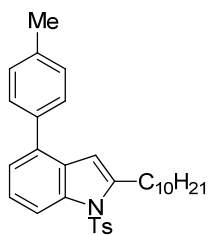


DFILE F:\NMR\MY236-1H1NON_E2.als
COMNT MY236-1H
DATIM Tue Feb 25 09:37:34 2014
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 20.9 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 11

F:\NMR\MY236-13C1BCM_E3.als
MY236-13C

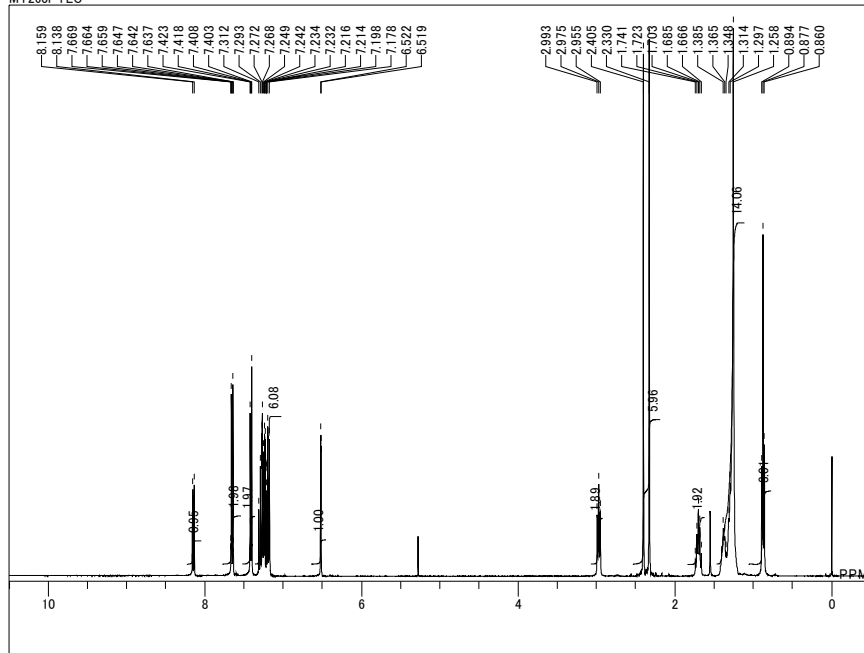


DFILE F:\NMR\MY236-13C1BCM_E3.a
COMNT MY236-13C
DATIM Tue Feb 25 11:58:08 2014
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 26



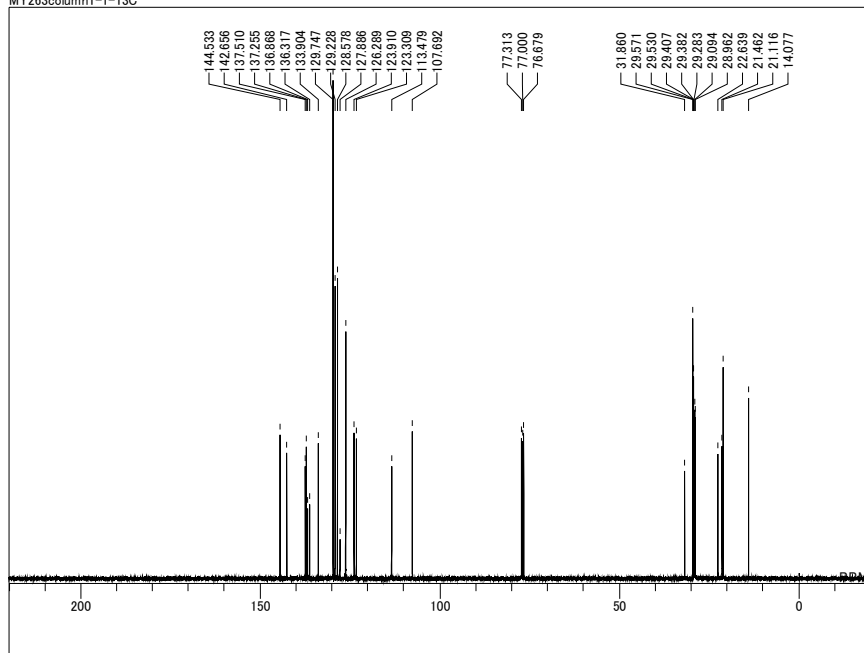
24

F:\NMR\MY263-3-11NON_E1.als
MY263PTLC

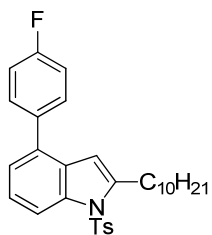


DFILE F:\NMR\MY263-3-11NON_E1.als
COMNT MY263PTLC
DATIM Sat Sep 21 11:41:13 2013
OBNUC 1H
EXMOD NON
OBFREQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 22.1 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12

F:\NMR\MY263-2-1-13C1BCM_E1.als
MY263column1-1-13C

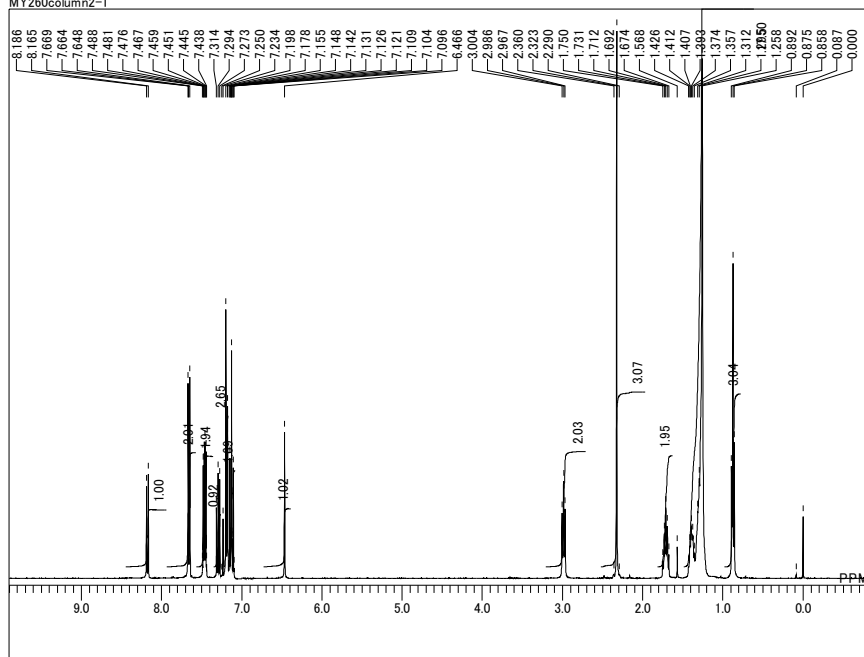


DFILE F:\NMR\MY263-2-1-13C1BCM
COMNT MY263column1-1-13C
DATIM Fri Sep 20 10:56:06 2013
OBNUC 13C
EXMOD BCM
OBFREQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 512
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 26



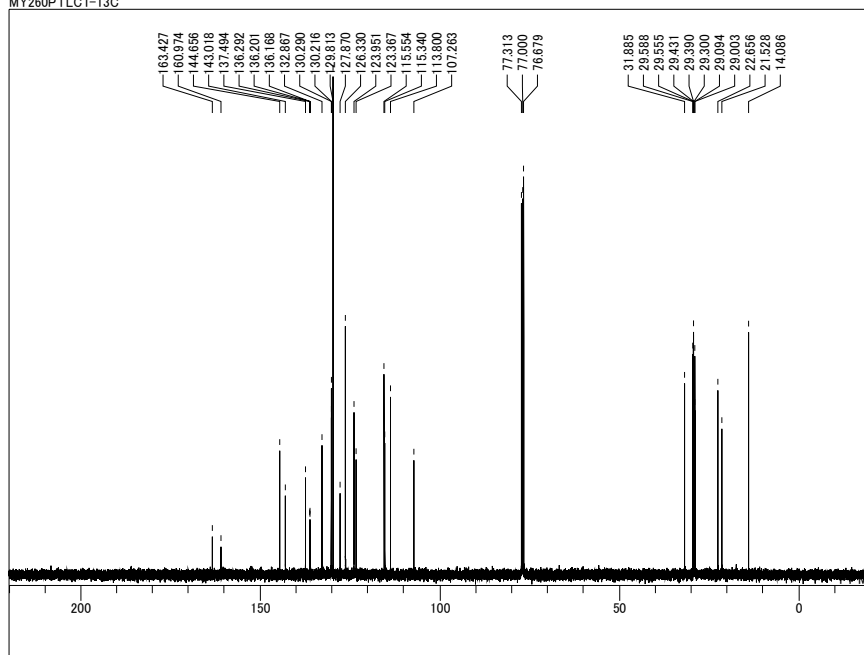
25

F:\NMR\MY260-3-11NON_E8_FT.als
MY260column2-1

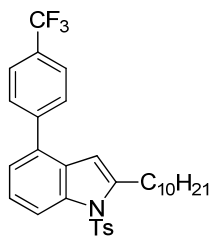


DFILE F:\NMR\MY260-3-11NON_E8_F
COMNT MY260column2-1
DATIM Thu Sep 19 15:37:13 2013
OBNUC 1H
EXMOD NON
OBFREQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 11

F:\NMR\MY260-4-1-13C1BCM_E4.als
MY260PTLC1-13C

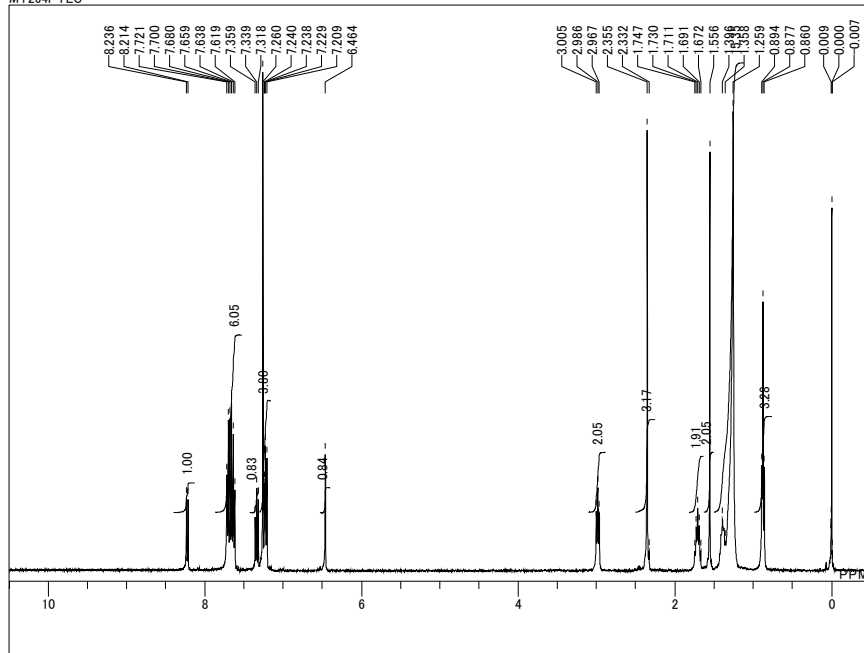


DFILE F:\NMR\MY260-4-1-13C1BCM
COMNT MY260PTLC1-13C
DATIM Fri Sep 20 10:16:46 2013
OBNUC 13C
EXMOD BCM
OBFREQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 512
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 27



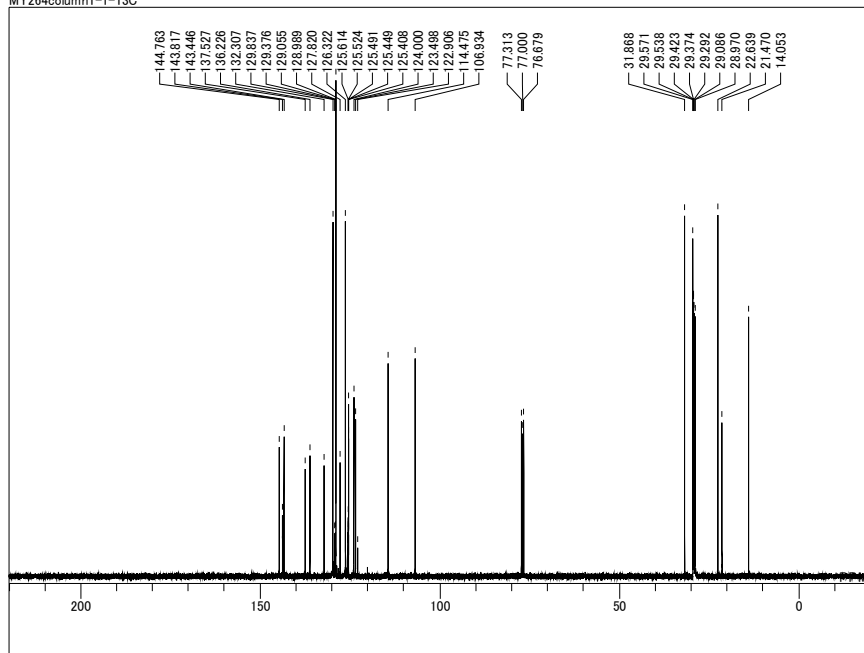
26

F:\NMR\MY264-3-11NON_E2.als
MY264PTLC

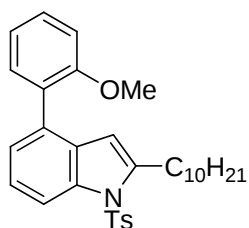


DFILE F:\NMR\MY264-3-11NON_E2.als
COMNT MY264PTLC
DATIM Sat Sep 21 11:46:11 2013
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 17

F:\NMR\MY264-2-1-13C1BCM.E1.als
MY264column1-1-13C

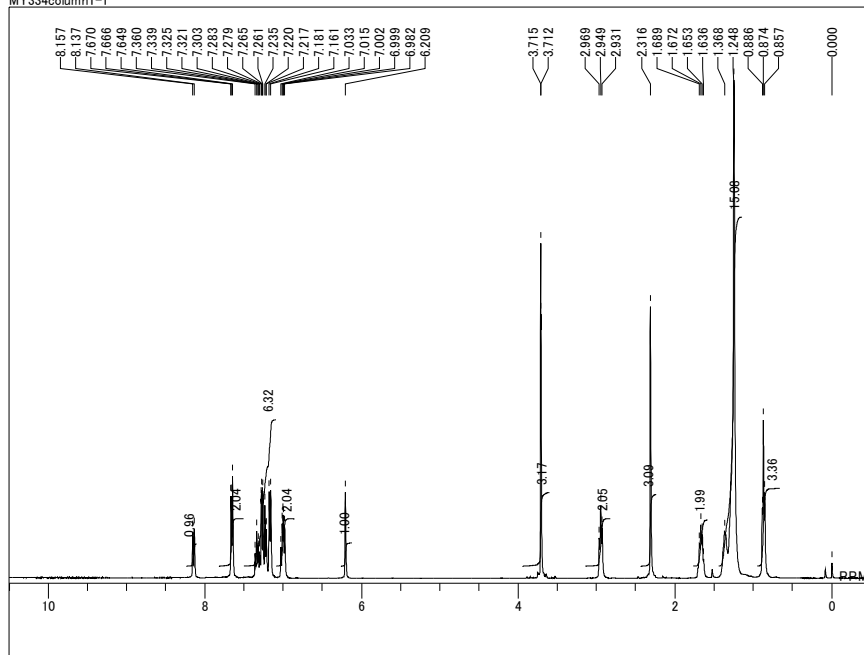


DFILE F:\NMR\MY264-2-1-13C1BCM
COMNT MY264column1-1-13C
DATIM Fri Sep 20 12:54:28 2013
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 512
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 27



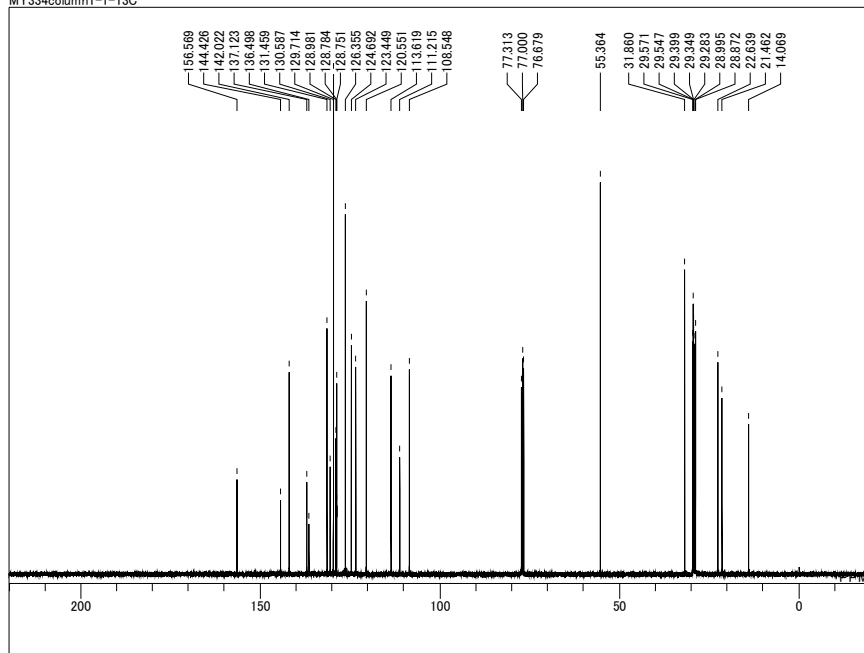
27

F:\NMR\MY334-2-11NON_E6.als
MY334column1-1

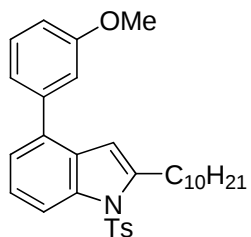


D:\NMR\MY334-2-11NON_E6.als
COMNT
MY334column1-1
DATIM
Wed Jan 22 23:12:51 2014
OBNUC
1H
EXMOD
NON
OBFREQ
399.65 MHz
OBSET
124.00 KHz
OBFIN
10500.00 Hz
POINT
16384
FREQ
7992.01 Hz
SCANS
8
ACQTM
2.0500 sec
PD
4.9500 sec
PW1
6.20 usec
IRNUC
1H
CTEMP
21.0 c
SLVNT
CDCL3
EXREF
0.00 ppm
BF
0.12 Hz
RGAIN
8

F:\NMR\MY334-2-1-13C1BCM_E11.als
MY334column1-1-13C

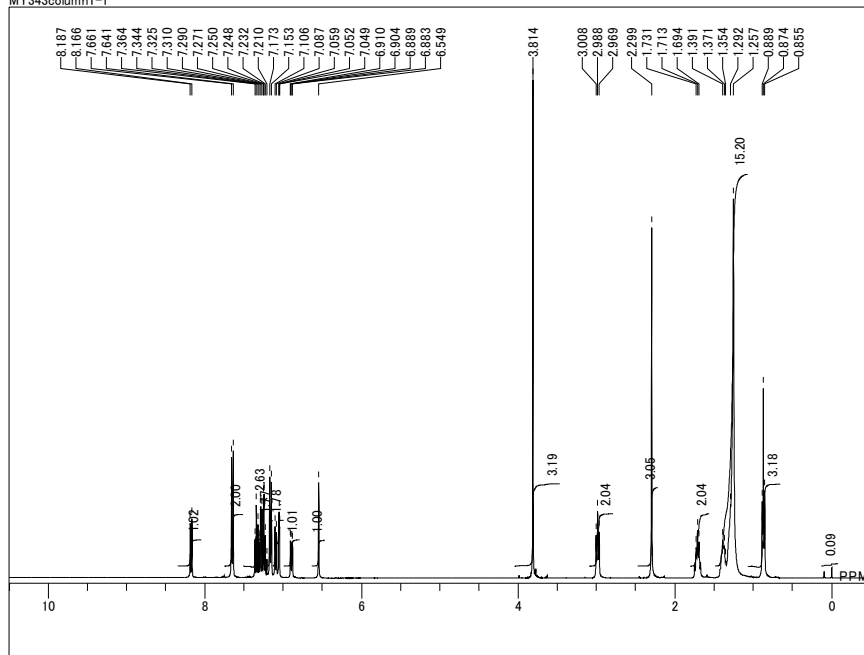


D:\NMR\MY334-2-1-13C1BCM
COMNT
MY334column1-1-13C
DATIM
Thu Jan 23 00:39:12 2014
OBNUC
13C
EXMOD
BCM
OBFREQ
100.40 MHz
OBSET
125.00 KHz
OBFIN
10500.00 Hz
POINT
32768
FREQ
27118.64 Hz
SCANS
1024
ACQTM
1.2083 sec
PD
1.7920 sec
PW1
5.50 usec
IRNUC
1H
CTEMP
24.4 c
SLVNT
CDCL3
EXREF
77.00 ppm
BF
0.12 Hz
RGAIN
26



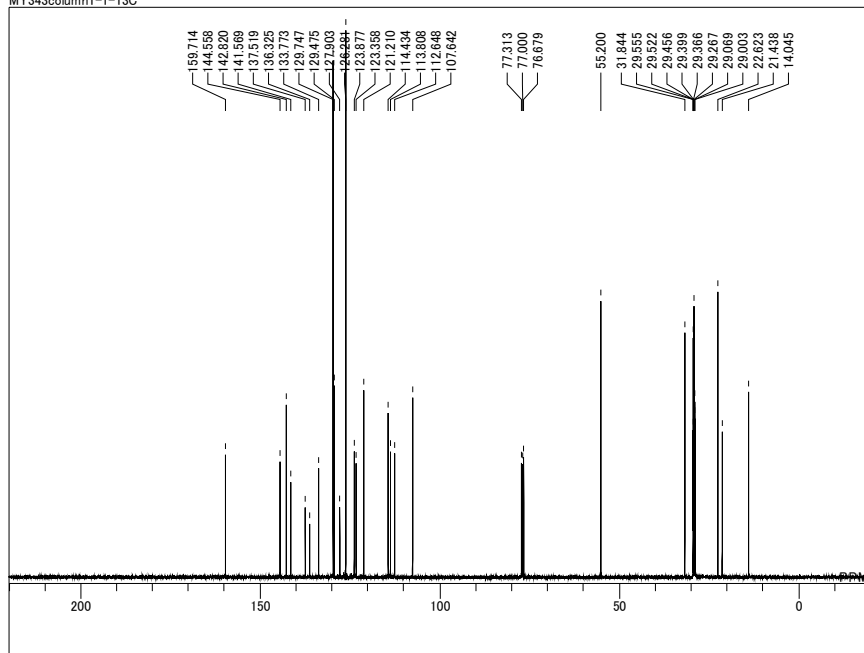
28

F:\NMR\MY343-2-11NON_E8.als
MY343column1-1

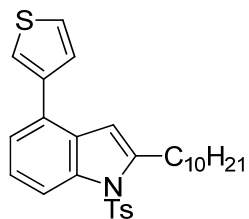


DFILE F:\NMR\MY343-2-11NON_E8.als
COMNT MY343column1-1
DATIM Tue Jan 28 20:30:43 2014
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 22.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 7

F:\NMR\MY343-2-1-13C1BCM_E1.als
MY343column1-1-13C

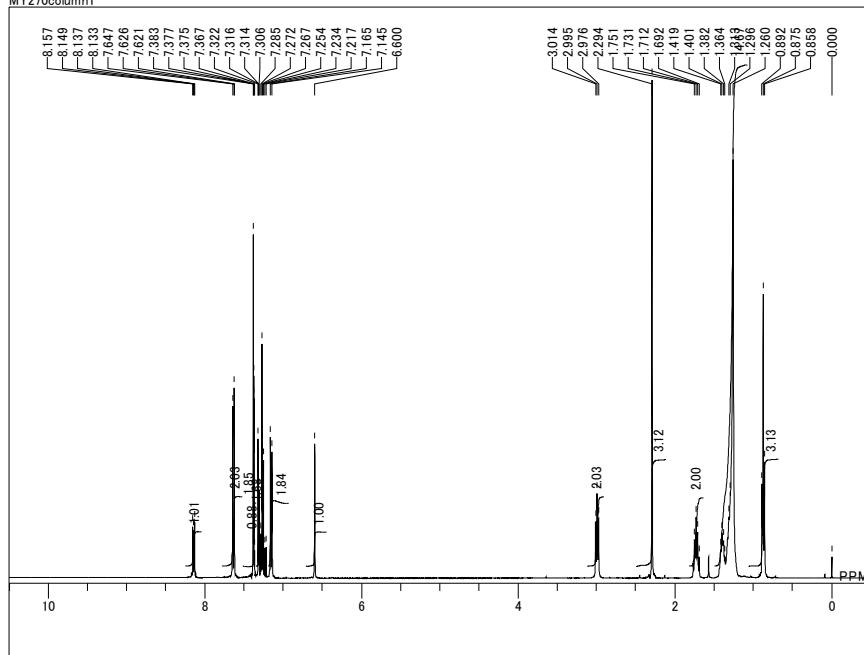


DFILE F:\NMR\MY343-2-1-13C1BCM
COMNT MY343column1-1-13C
DATIM Tue Jan 28 21:37:14 2014
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 25.2 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 26



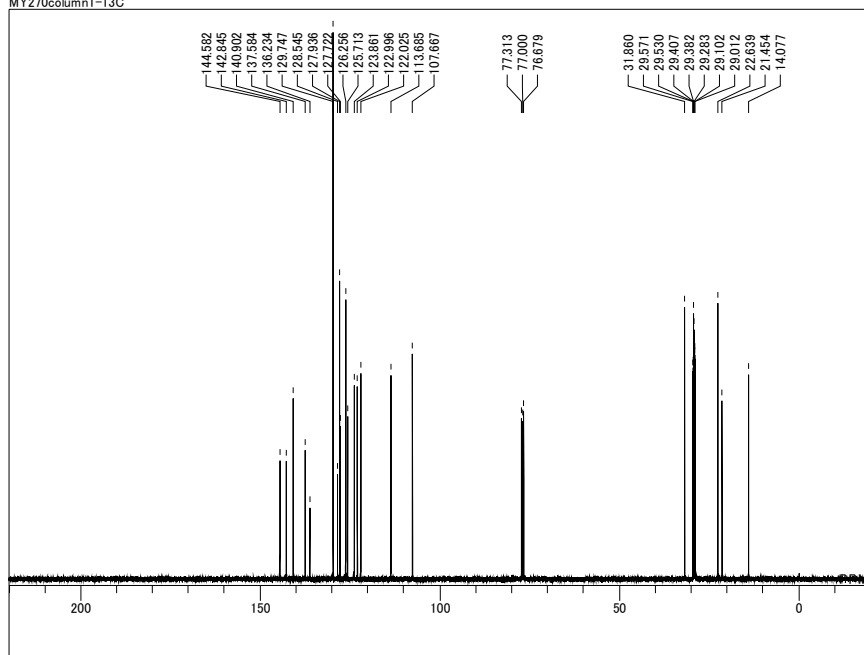
29

F:\NMR\MY270-2-11NON_E10.als
MY270column1

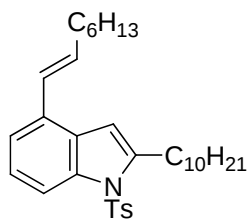


DFILE F:\NMR\MY270-2-11NON_E10.als
COMNT MY270column1
DATIM Thu Sep 26 11:30:00 2013
OBNUC 1H
EXMOD NON
OBFREQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 22.0 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 8

F:\NMR\MY270-2-1-13C1BCM_E1.als
MY270column1-13C

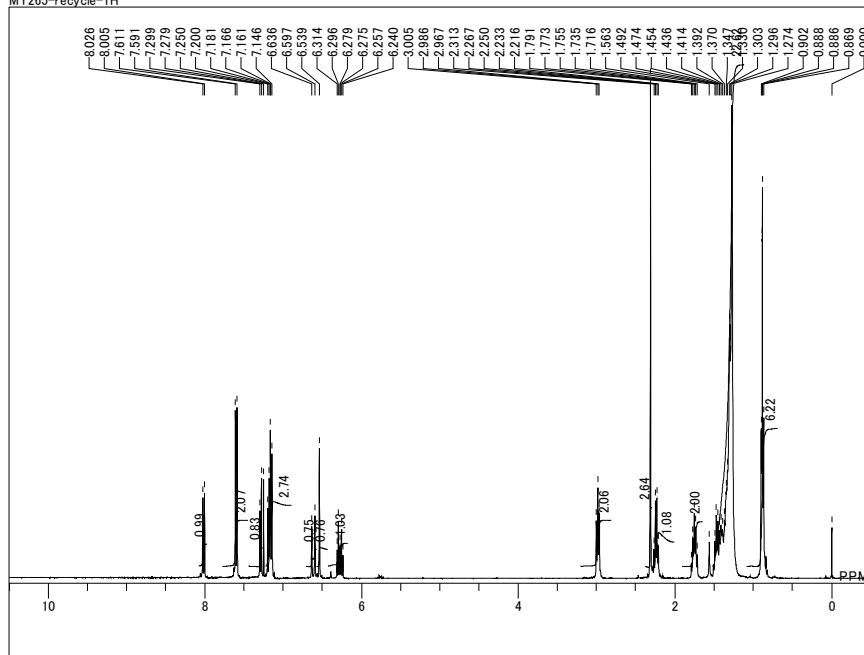


DFILE F:\NMR\MY270-2-1-13C1BCM
COMNT MY270column1-13C
DATIM Thu Sep 26 12:46:57 2013
OBNUC 13C
EXMOD BCM
OBFREQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 512
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 23.9 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 26



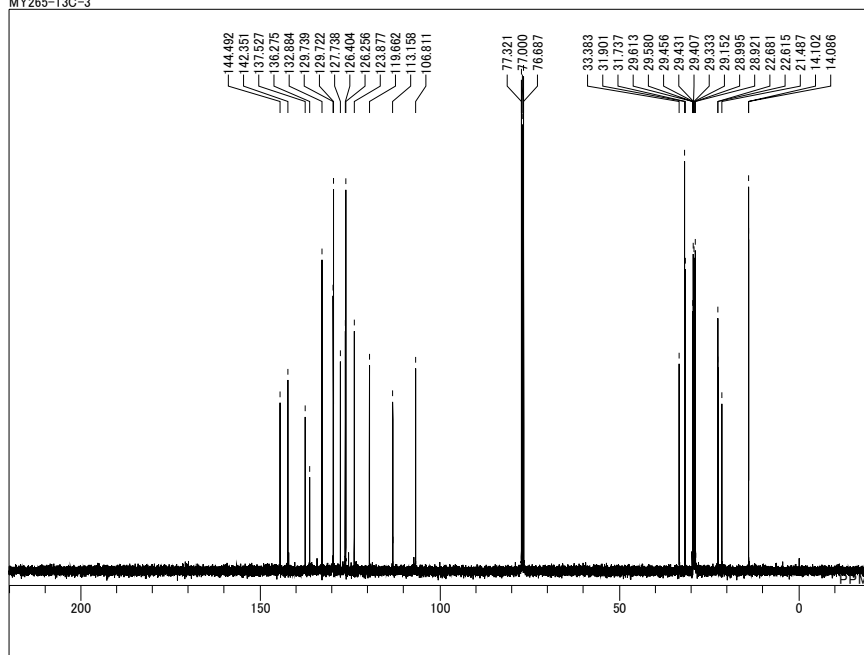
30

F:\NMR\MY265-1H1NON_E2.als
MY265-recycle-1H

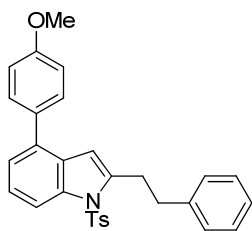


DFILE F:\NMR\MY265-1H1NON_E2.als
COMNT MY265-recycle-1H
DATIM Fri Feb 28 11:01:14 2014
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 21.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12

F:\NMR\MY265-13C-31BCM_E1.als
MY265-13C-3

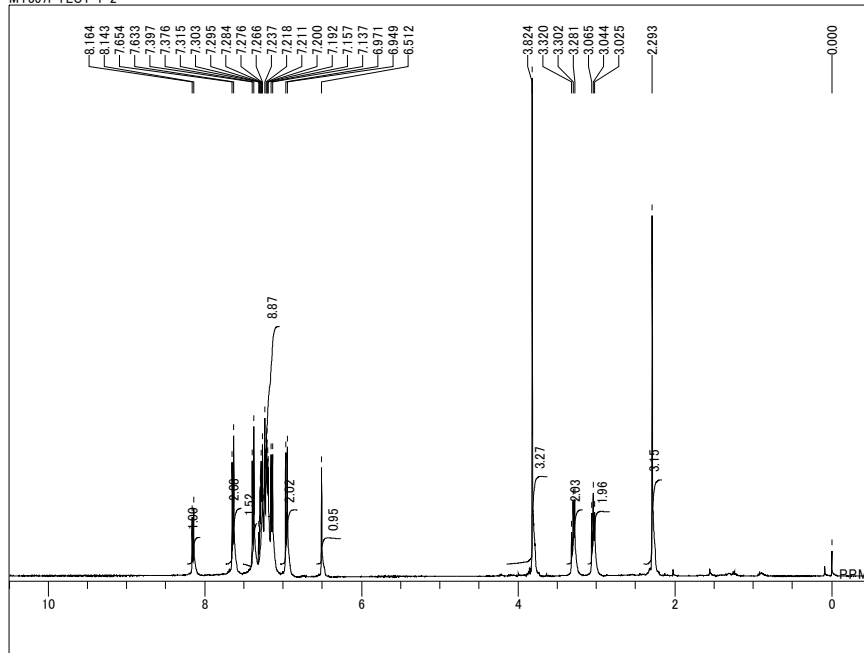


DFILE F:\NMR\MY265-13C-31BCM_E
COMNT MY265-13C-3
DATIM Fri Feb 28 12:21:07 2014
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 13C
CTEMP 23.7 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 26



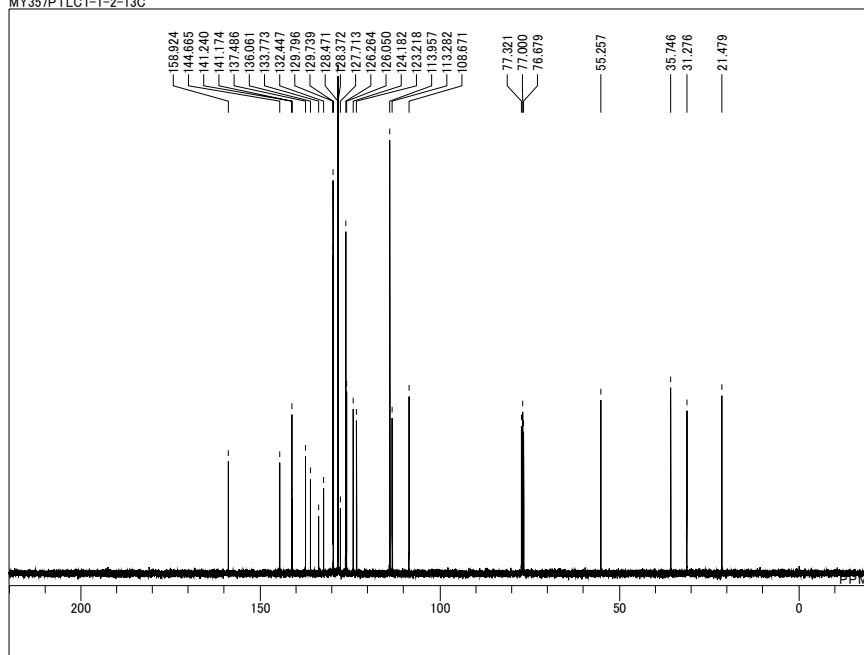
31

F:\NMR\MY357-3-1-21NON.E1.als
MY357PTLC1-1-2



DFILE F:\NMR\MY357-3-1-21NON.E1
COMNT MY357PTLC1-1-2
DATIM Thu Feb 20 09:40:59 2014
OBNUC 1H
EXMOD NON
OBFRQ 399.65 MHz
OBSET 124.00 KHz
OBFIN 10500.00 Hz
POINT 16384
FREQU 7992.01 Hz
SCANS 8
ACQTM 2.0500 sec
PD 4.9500 sec
PW1 6.20 usec
IRNUC 1H
CTEMP 21.4 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 12

F:\NMR\MY357-3-1-2-13C1BCM.E8.als
MY357PTLC1-1-2-13C



DFILE F:\NMR\MY357-3-1-2-13C1BCM
COMNT MY357PTLC1-1-2-13C
DATIM Thu Feb 20 12:23:17 2014
OBNUC 13C
EXMOD BCM
OBFRQ 100.40 MHz
OBSET 125.00 KHz
OBFIN 10500.00 Hz
POINT 32768
FREQU 27118.64 Hz
SCANS 1024
ACQTM 1.2083 sec
PD 1.7920 sec
PW1 5.50 usec
IRNUC 1H
CTEMP 21.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.12 Hz
RGAIN 20