Supporting Information

Hydrazone-Palladium Catalyzed Annulation of 1-Allyl-2-bromobenzene Derivatives with Internal Alkynes

Kohei Watanabe,[a] Takashi Mino,[a,b]* Chikako Hatta,[a] Shisei Ito[a] and Masami Sakamoto[a,b]

E-mail: tmino@faculty.chiba-u.jp

[a] Department of Applied Chemistry and Biotechnology, Graduate School of Engineering, Chiba University, 1-33, Yayoi-cho, Inage-ku, Chiba 263-8522, Japan

[b] Molecular Chirality Research Center, Chiba University, 1-33, Yayoi-cho, Inage-ku, Chiba 263-8522, Japan

Table of contents:

Preparation and characterization details for compounds 3b-j, 4b-f.

Preparation of 1-allyl-2-bromo-5-methoxybenzene (3b).

Vinyl magnesium chloride (9.25 mmol) in THF (6.8 mL, 1.36 M) was added gradually to the mixture of 2-bromo-5-methoxybenzyl bromide (1.3998 g, 5.0 mmol), CuI (95.2 mg, 0.50 mmol) and 2,2'-bipyridil (78.1 mg, 0.50 mmol) in THF (8.75 mL) at 0 °C under Ar atmosphere and the mixture was stirred at 0 °C. After 55 min, the mixture was stirred for 5 min at room temperature and the reaction was quenched with saturated NH_4Cl aq. The solution was extracted with diethyl ether, washed with saturated $NaHCO_3$ aq., dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/chloroform = 40/1) to afford **3b** in 34% yield (0.3869 g, 1.7 mmol).

1-Allyl-2-bromo-5-methoxybenzene (**3b**).[1] Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 3.46 (d, *J* = 6.5 Hz, 2H), 3.77 (s, 3H), 5.05-5.14 (m, 2H), 5.89-6.02 (m, 1H), 6.65 (dd, *J* = 8.7 and 3.1 Hz, 1H), 6.78 (d, *J* = 3.0 Hz, 1H), 7.42 (d, *J* = 8.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 40.3, 55.4, 113.4, 115.0, 116.1, 116.7, 133.2, 135.4, 140.4, 159.0; EI-MS *m/z* (rel intensity) 226 (M^+, 70).

Preparation of 1-allyl-2-bromo-5-methylbenzene (3c).

This compound was synthesized *via* three-step reactions. First step: 2-Bromo-5-methy benzoic acid (1.0753 g, 5.0 mmol) was added gradually to a THF (30 mL) solution of $LiAlH_4$ (0.4744 g, 12.5 mmol) at 0 °C under Ar atmosphere. The mixture was stirred for 25 h at room temperature. After the reaction, $Na_2SO_4 \cdot 10H_2O$ (4.0 g) and THF (20 mL) were added to the solution and the mixture was stirred at room temperature. After 24 h, the solution was filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/ethyl acetate = 8/1) to afford the desired alcohol in 42% yield (0.4227 g, 2.1 mmol).

2-Bromo-5-methyl-benzyl alcohol. White solid; m.p. 60 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.07 (br s, 1H), 2.32 (s, 3H), 4.71 (d, *J* = 6.1 Hz, 2H), 6.97 (d, *J* = 7.7 Hz, 1H), 7.27 (d, *J* = 7.0 Hz, 1H), 7.41 (d, *J* = 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 20.9, 65.1, 119.2, 129.8, 129.9, 132.3, 137.6, 139.2; EI-MS *m/z* (rel intensity) 200 (M^+, 47). HRMS (ESI) *m/z* calcd for C_8H_9OBrNa $[M + Na]^+$ 222.9729, found 222.9726.

Second step: Carbontetrabromide (0.9588 g, 3.0 mmol) was added gradually to the mixture of 2-bromo-5-methylbenzyl alcohol (0.2011 g, 1.0 mmol) and PPh_3 (0.7868 g, 3.0 mmol) in THF (5 mL) at 0 °C. The mixture was stirred for 23 h at room temperature. After the reaction, the solution was diluted with hexane and diethyl ether. The solution was filtered through silica

gel pad, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane) to afford the desired bromide in 59% yield (0.1558 g, 0.59 mmol).

2-Bromo-5-methyl-benzyl bromide.[2] olorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.30 (s, 3H), 4.57 (s, 2H), 6.96 (dd, *J* = 8.1 and 2.0 Hz, 1H), 7.26 (s, 1H), 7.44 (d, *J* = 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 20.8, 33.5, 121.0, 131.0, 131.9, 133.0, 136.6, 138.0; EI-MS *m/z* (rel intensity) 264 (M^+, 18).

Third step: Vinyl magnesium chloride (1.0 mmol) in THF (0.74 mL, 1.36 M) was added gradually to the mixture of 2-bromo-5-methylbenzyl bromide (0.15 mL, 1.0 mmol), CuI (19.0 mg, 0.10 mmol) and 2,2'-bipyridil (15.6 mg, 0.10 mmol) in THF (2 mL) at 0 °C under Ar atmosphere and the mixture was stirred at 0 °C. After 55 min, the mixture was stirred for 5 min at room temperature and the reaction was quenched with saturated NH_4Cl aq. The solution was extracted with diethyl ether, washed with saturated $NaHCO_3$ aq., dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane) to afford **3c** in 43% yield (0.0914 g, 0.43 mmol).

1-Allyl-2-bromo-5-methylbenzene (**3c**). Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.28 (s, 3H), 3.46 (d, *J* = 6.5 Hz, 2H), 5.04-5.13 (m, 2H), 5.90-6.03 (m, 1H), 6.88 (dd, *J* = 8.1 and 2.0 Hz, 1H), 7.03 (d, *J* = 1.7 Hz, 1H), 7.41 (d, *J* = 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 20.8, 40.1, 116.4, 121.1, 128.6, 131.6, 132.4, 135.7, 137.3, 139.0; EI-MS *m/z* (rel intensity) 210 (M^+, 29); HRMS (ESI) *m/z* calcd for $C_{10}H_{11}BrNa$ $[M + Na]^+$ 232.9936, found 232.9931.

Preparation of 1-allyl-2-bromo-5-chlorobenzene (3d).

Vinyl magnesium chloride (3.6 mmol) in THF (2.7 mL, 1.36 M) was added gradually to the mixture of 2-Bromo-5-chlorobenzyl bromide (0.8532 g, 3.0 mmol), CuI (57.1 mg, 0.30 mmol) and 2,2'-bipyridil (46.9 mg, 0.30 mmol) in THF (6 mL) at 0 °C under Ar atmosphere and the mixture was stirred at 0 °C. After 1 h, the mixture was stirred for 18 h at room temperature and the reaction was quenched with saturated NH_4Cl aq. The solution was extracted with diethyl ether, washed with saturated $NaHCO_3$ aq., dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/toluene = 40/1) to afford **3d** in 59% yield (0.4111 g, 1.8 mmol).

1-Allyl-2-bromo-5-chlorobenzene (**3d**). Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 3.47 (d, *J* = 6.5 Hz, 2H), 5.06-5.18 (m, 2H), 5.86-6.00 (m, 1H), 7.04-7.08 (m, 1H), 7.21 (d, *J* = 2.5 Hz, 1H), 7.46 (d, *J* = 8.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 40.0, 117.4, 122.3, 127.9, 130.3, 133.4, 133.7, 134.6, 141.2; EI-MS *m*/z (rel intensity) 230 (M^+, 28); HRMS (APPI) *m/z* calcd for C_9H_8BrCl $[M]^+$ 229.9492, found 229.9489.

Preparation of 1-allyl-2-bromo-5-fluorobenzene (3e).

Vinyl magnesium chloride (5.0 mmol) in THF (3.7 mL, 1.36 M) was added gradually to the mixture of 2-bromo-5-fluorobenzyl bromide (0.7 mL, 5.0 mmol), CuI (95.2 mg, 0.50 mmol) and 2,2'-bipyridil (78.1 mg, 0.50 mmol) in THF (10 mL) at 0 °C under Ar atmosphere and the mixture was stirred at 0 °C. After 55 min, the mixture was stirred for 5 min at room temperature and the reaction was quenched with saturated NH_4Cl aq. The solution was extracted with diethyl ether, washed with saturated $NaHCO_3$ aq., dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane) to afford **3e** in 59% yield (0.7502 g, 3.5 mmol).

1-Allyl-2-bromo-5-fluorobenzene (**3e**).[3] Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 3.48 (d, *J* = 6.5 Hz, 2H), 5.07 (t, *J* = 1.5 Hz, 2H), 5.87-6.00 (m, 1H), 6.78-6.85 (m, 1H), 6.96 (dd, *J* = 9.4 and 3.0 Hz, 1H), 7.48 (q, *J* = 3.3 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 40.2, 114.9 (d, *J* = 22.5 Hz), 117.2 (d, *J* = 22.8 Hz), 117.3, 118.5 (d, *J* = 3.3 Hz), 133.7 (d, *J* = 8.2 Hz), 134.7, 141.6 (d, *J* = 7.5 Hz), 162.0 (d, *J* = 246.7 Hz); EI-MS *m/z* (rel intensity) 214 (M^+, 46).

Preparation of 1-allyl-2-bromo-4-methylbenzene (3f).

This compound was synthesized *via* three-step reactions. First step: 2-Bromo-4-methyl benzoic acid (1.0753 g, 5.0 mmol) was added gradually to a THF (30 mL) solution of $LiAlH_4$ (0.4744 g, 12.5 mmol) at 0 °C under Ar atmosphere. The mixture was stirred for 21 h at room temperature. After the reaction, $Na_2SO_4 \cdot 10H_2O$ (4.0 g) and THF (20 mL) were added to the solution and stirred at room temperature. After 24 h, the solution was filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/ethyl acetate = 8/1) to afford the desired alcohol in 50% yield (0.5020 g, 2.5 mmol).

2-Bromo-4-methyl-benzyl alcohol.[4] White solid; m.p. 55-56 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.08 (br s, 1H), 2.33 (s, 3H), 4.70 (s, 2H), 7.12 (d, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 7.7 Hz, 1H), 7.38 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 20.7, 64.9, 122.5,128.4, 128.9, 133.1, 136.6, 136.6, 139.4; EI-MS *m/z* (rel intensity) 200 (M^+, 35).

Second step: Carbontetrabromide (8.9562 g, 27.0 mmol) was added gradually to the mixture of 2-bromo-4-methylbenzyl alcohol (1.8095 g, 9.0 mmol) and PPh_3 (7.0818 g, 27 mmol) in THF (45 mL) at 0 °C. The mixture was stirred for 28 h at room temperature. After the reaction, the solution was diluted with hexane and diethyl ether. The solution was filtered through silica gel pad, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane) to afford the desired bromide in 39% yield (0.9217 g, 3.49 mmol).

2-Bromo-4-methyl-benzylbromide.[4] Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.33 (s, 3H), 4.59 (s, 2H), 7.10 (d, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 7.8 Hz, 1H), 7.40 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 20.8, 33.5, 124.2, 128.7, 130.9, 133.7, 133.9, 140.6; EI-MS *m/z* (rel intensity) 264 (M^+, 49).

Third step: Vinyl magnesium chloride (1.0 mmol) in THF (0.74 mL, 1.36 M) was added gradually to the mixture of 2-bromo-4-methylbenzyl bromide (0.15 mL, 1.0 mmol), CuI (19.0 mg, 0.10 mmol) and 2,2'-bipyridil (15.6 mg, 0.10 mmol) in THF (2 mL) at 0 °C under Ar atmosphere and the mixture was stirred at 0 °C. After 55 min, the mixture was stirred for 5 min at room temperature and the reaction was quenched with saturated NH_4Cl aq. The solution was extracted with diethyl ether, washed with saturated $NaHCO_3$ aq., dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane) to afford **3f** in 40% yield (0.0847 g, 0.40 mmol).

1-Allyl-2-bromo-4-methylbenzene (**3f**). Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.30 (s, 3H), 3.46 (d, *J* = 6.5 Hz, 2H), 5.02-5.12 (m, 2H), 5.89-6.02 (m, 1H), 7.07 (dd, *J* = 16.2 and 7.8 Hz, 2H), 7.37 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 20.6, 39.7, 116.2, 124.2, 128.3, 130.1, 133.1, 135.8, 136.2, 137.8; EI-MS *m/z* (rel intensity) 210 (M^+, 37); HRMS (APPI) *m/z* calcd for $C_{10}H_{11}Br$ $[M]^+$ 210.0039, found 210.0035.

Preparation of 1-allyl-2-bromo-4,5-dimethoxybenzene (3g).

This compound was synthesized *via* three-step reactions. First step: 2-Bromo-4,5-dimethoxy benzoic acid (3.9161 g, 15.0 mmol) was added gradually to a THF (90 mL) solution of $LiAlH_4$ (1.4233 g, 37.5 mmol) at 0 °C under Ar atmosphere. The mixture was stirred for 31 h at room temperature. After the reaction, $Na_2SO_4 \cdot 10H_2O$ (12.0 g) and THF (60 mL) were added to the solution and stirred at room temperature. After 16 h, the solution was filtered and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/diethylether = 1/1) to afford the desired alcohol in 32% yield (1.1727 g, 4.7 mmol).

2-Bromo-4,5-dimethoxy-benzyl alcohol.[5] White solid; m.p. 95-96 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.11 (br s, 1H), 3.87 (s, 3H), 3.89 (d, *J* = 1.1 Hz, 3H), 4.68 (s, 2H), 7.01 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 56.0, 56.2, 64.9, 111.8, 112.5, 115.3, 131.8, 148.5, 148.9; EI-MS *m/z* (rel intensity) 246 (M^+, 72).

Second step: Carbontetrabromide (0.9948 g, 3.0 mmol) was added gradually to the mixture of 2-bromo-4,5-dimethoxybenzyl alcohol (0.2471 g, 1.0 mmol) and PPh_3 (0.7869 g, 3.0 mmol) in THF (5 mL) at 0 °C. The mixture was stirred for 20 h at room temperature. After the reaction, the solution was diluted with hexane and diethyl ether. The solution was filtered

through silica gel pad, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/ethyl acetate = 10/1) to afford the desired bromide in 39% yield (0.1196 g, 0.39 mmol).

2-Bromo-4,5-dimethoxybenzyl bromide.[6] White solid; m.p. 83-85 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 3.88 (d, *J* = 2.3 Hz, 6H), 4.59 (s, 2H), 6.93 (s, 1H), 7.02 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 34.1, 56.1, 56.2, 113.3, 114.9, 115.6, 128.8, 148.6, 149.8; EI-MS *m/z* (rel intensity) 310 (M^+, 10).

Third step: Vinyl magnesium chloride (1.4 mmol) in THF (1.04 mL, 1.36 M) was added gradually to the mixture of 2-bromo-4,5-dimethoxybenzyl bromide (0.3040 g, 1.0 mmol), CuI (19.0 mg, 0.10 mmol) and 2,2'-bipyridil (15.6 mg, 0.10 mmol) in THF (2 mL) at 0 °C under Ar atmosphere and the mixture was stirred at 0 °C. After 1 h, the mixture was stirred for 20 h at room temperature and the reaction was quenched with saturated NH_4Cl aq. The solution was extracted with diethyl ether, washed with saturated $NaHCO_3$ aq., dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane/ethyl acetate = 10/1) to afford **3g** in 63% yield (0.1612 g, 0.63 mmol).

1-Allyl-2-bromo-4,5-dimethoxybenzene (**3g**).[7] Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 3.42-3.45 (m, 2H), 3.86 (s, 6H), 5.03-5.13 (m, 2H), 5.88-6.01 (m, 1H), 6.72 (s, 1H), 7.02 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 39.8, 56.0, 56.1, 112.9, 114.1, 115.4, 116.3, 131.2, 135.9, 147.9, 148.4; EI-MS *m/z* (rel intensity) 256 (M^+, 100).

Preparation of 2-allyl-1-bromonaphathalene (3h).

Vinyl magnesium chloride (1.0 mmol) in THF (0.74 mL, 1.36 M) was added gradually to the mixture of 1-bromo-2-(bromomethyl)naphthalene (0.3000 g, 1.0 mmol), CuI (19.0 g, 0.10 mmol) and 2,2'-bipyridil (15.6 mg, 0.10 mmol) in THF (2 mL) at 0 °C under Ar atmosphere and the mixture was stirred at 0 °C. After 55 min, the mixture was stirred for 5 min at room temperature and the reaction was quenched with saturated NH_4Cl aq. The solution was extracted with diethyl ether, washed with saturated $NaHCO_3$ aq., dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane) to afford **3h** in 47% yield (0.1163 g, 0.47 mmol).

2-Allyl-1-bromonaphathalene (**3h**). Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 3.76 (d, *J* = 6.4 Hz, 2H), 5.07-5.14 (m, 2H), 5.97-6.10 (m, 1H), 7.35 (d, *J* = 8.4 Hz, 1H), 7.48 (ddd, *J* = 7.5, 7.4 and 0.9 Hz, 1H), 7.58 (ddd, *J* = 6.9, 7.6 and 1.2 Hz, 1H), 7.74 (d, *J* = 8.4 Hz, 1H), 7.80 (d, *J* = 8.4 Hz, 1H), 8.32 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 41.4, 116.5, 123.9, 126.0, 127.3 (2C), 127.6, 128.0 (2C), 132.6, 133.3, 135.5, 137.4; EI-MS *m/z* (rel intensity) 246 (M^+, 46); HRMS (APPI) *m/z* calcd for $C_{13}H_{11}BrNa$ $[M + Na]^+$ 268.9936, found

268.9936.

Preparation of 1-bromo-2-cinnamylbenzene (3i).

A mixture of cinnamyl acetate (0.3524 g, 2.0 mmol), *o*-bromobonic acid (0.6025 g 3.0 mmol), $Pd(OAc)_2$ (0.0180 g, 0.08 mmol), hydrazone ligand **1d** (0.0178 g, 0.08 mmol) and K_2CO_3 (0.5528 g, 4.0 mmol) in DMF/H_2O (3/1) (1 mL) at 50 °C under an Ar atmosphere was stirred for 24 h. After the reaction, the reaction was quenched with distilled water. The solution was extracted with ethyl acetate, washed with brine, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure. The residue was purified by silica gel chromatography (hexane) to afford **3i** in 38% yield (0.2067 g, 0.76 mmol).

1-Bromo-2-cinnamylbenzene (**3i**).[8] Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 3.66 (d, *J* = 6.2 Hz, 2H), 6.29-6.38 (m, 1H), 6.45 (d, *J* = 15.9 Hz, 1H), 7.05-7.11 (m, 1H), 7.17-7.37 (m, 7H), 7.56 (d, *J* = 8.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 39.4, 124.6, 126.1, 127.2, 127.3, 127.5, 127.9, 128.5, 130.5, 131.7, 132.8, 137.3, 139.6; EI-MS *m/z* (rel intensity) 272 (M^+, 31).

Preparation of 1-bromo-4-methoxy-2-prenylbenzene (3j).

Isobutenyl magnesium chloride (1.2 mmol) in THF (2.4 mL, 0.5 M) was added gradually to the mixture of 2-bromo-5-methoxybenzyl bromide (0.2730 g, 1.0 mmol), CuI (19.0 mg, 0.10 mmol) and 2,2'-bipyridil (15.6 mg, 0.10 mmol) in THF (2 mL) at 0 °C under Ar atmosphere and the mixture was stirred at 0 °C. After 1 h, the mixture was stirred for 24 h at room temperature and the reaction was quenched with saturated NH_4Cl aq. The solution was extracted with diethyl ether, washed with saturated $NaHCO_3$ aq., dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (hexane) to afford **3j** in 54% yield (0.1333 g, 0.52 mmol).

1-Bromo-4-methoxy-2-prenylbenzene (**3j**). Colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.72 (s, 3H), 1.46 (s, 3H), 3.39 (d, *J* =7.3 Hz, 2H), 3.77 (s, 3H), 5.24-5.30 (m, 1H), 6.62 (dd, *J* = 8.7 and 3.0 Hz, 1H), 6.77 (d, *J* =3.0 Hz, 1H), 7.41 (d, *J* = 8.7 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 18.0, 25.8, 34.8, 55.4, 112.7, 115.0, 116.0, 121.2, 133.0, 133.8, 142.0, 158.9; EI-MS (rel intensity) 254 (M^+, 43); HRMS (APPI) *m/z* calcd for $C_{12}H_{15}OBr$ $[M]^+$ 254.0301, found 254.0294.

Preparation of 1,2-bis(4-methoxyphenyl)ethyne (**4b**).

A mixture of *p*-bromoanisole (0.9538 g, 5.0 mmol), *p*-ethynylanisole (0.7965 g 6.0 mmol), $Pd(acac)_2$ (1.54 mg, 0.005 mmol), CuI (4.56 mg, 0.025 mmol), hydrazone ligand **1d** (5.95 mg, 0.025 mmol) and K_3PO_4 (1.0085 g, 5.0 mmol) in DMSO (20 mL) at 125 °C under an Ar atmosphere was stirred for 24 h. After the reaction, the reaction was quenched with distilled

water. The solution was extracted with ethyl acetate, washed with brine, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure. The residue was purified by silica gel chromatography (hexane/ethyl acetate = 40/1) to afford **4b** in 65% yield (0.7761 g, 3.26 mmol).

1,2-Bis(4-methoxyphenyl)ethyne (**4b**).[9] Yellow solid; m.p. 145 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 3.82 (s, 6H), 6.87 (d, *J* = 8.8 Hz, 4H), 7.45 (d, *J* = 8.9 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 55.2, 88.0, 114.0, 115.7, 132.8, 159.4; EI-MS *m/z* (rel intensity): 238 (M^+, 100).

Preparation of 1,2-di-*p*-tolylethyne (**4c**)**.**

A mixture of *p*-bromotoluene (0.8581 g, 5.0 mmol), 1-ethynyl-4-methylbenzene (0.6991 g, 6.0 mmol), $Pd(acac)_2$ (1.69 mg, 0.005 mmol), CuI (4.89 mg, 0.025 mmol), hydrazone ligand **1d** (5.58 mg, 0.025 mmol) and K_3PO_4 (1.0608 g, 5.0 mmol) in DMSO (20 mL) at 125 °C under an Ar atmosphere was stirred for 24 h. After the reaction, the reaction was quenched with distilled water. The solution was extracted with ethyl acetate, washed with brine, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure. The residue was purified by silica gel chromatography (hexane/ethyl acetate = 100/1) to afford **4c** in 65% yield (0.6650 g, 3.23 mmol).

1,2-Di-*p*-tolylethyne (**4c**).[10] White solid; m.p. 126 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 2.36 (s, 6H), 7.15 (d, *J* = 7.9 Hz, 4H), 7.41 (d, *J* = 8.1 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 21.5, 88.4, 120.3, 129.1, 131.4, 138.1; EI-MS *m/z* (rel intensity): 206 (M^+, 100).

Preparation of 1,2-bis(4-chlorophenyl)ethyne (**4d**)**.**

A mixture of 4-bromochlorobenzene (0.9546 g, 5.0 mmol), 1-ethynyl-4-chlorobenzene (0.8186 g, 6.0 mmol), $Pd(acac)_2$ (1.49 mg, 0.005 mmol), CuI (5.09 mg, 0.025 mmol), hydrazone ligand **1d** (5.50 mg, 0.025 mmol) and K_3PO_4 (1.0685 g, 5.0 mmol) in DMSO (20 mL) at 125 °C under an Ar atmosphere was stirred for 24 h. After the reaction, the reaction was quenched with distilled water. The solution was extracted with ethyl acetate, washed with brine, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure. The residue was purified by silica gel chromatography (hexane) to afford **4d** in 43% yield (0.5325 g, 2.15 mmol).

1,2-Bis(4-chlorophenyl)ethyne (**4d**).[11] White solid; m.p. 179-181 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.33 (dt, *J* = 8.7 and 1.9 Hz, 4H), 7.45 (dt, *J* = 8.7 and 1.9 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 89.2, 121.4, 128.7, 132.8, 134.5; EI-MS *m/z* (rel intensity): 246 (M^+, 100).

Preparation of 1,2-bis(4-fluorophenyl)ethyne (4e).

A mixture of 4-bromofluorobenzene (0.8754 g, 5.0 mmol), 1-ethynyl-4-fluorobenzene

(0.7325 g, 6.0 mmol), $Pd(acac)_2$ (1.78 mg, 0.005 mmol), CuI (5.32 mg, 0.025 mmol), hydrazone ligand **1d** (5.69 mg, 0.025 mmol) and K_3PO_4 (1.0985 g, 5.0 mmol) in DMSO (20 mL) at 125 °C under an Ar atmosphere was stirred for 24 h. After the reaction, the reaction was quenched with distilled water. The solution was extracted with ethyl acetate, washed with brine, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure. The residue was purified by silica gel chromatography (hexane) to afford **4e** in 59% yield (0.6298 g, 2.94 mmol).

1,2-Bis(4-fluorophenyl)ethyne (**4e**).[10] White solid; m.p. 95 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.05 (t, *J* = 8.7 Hz, 4H), 7.49 (t, *J* = 5.5 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 87.9, 115.6 (d, *J* = 2.2 Hz), 119.1 (d, *J* = 3.5 Hz), 133.4 (d, *J* = 8.4 Hz), 162.5 (d, *J* = 249 Hz); EI-MS *m/z* (rel intensity): 214 (M^+, 100).

Preparation of 1,2-bis(4-cyanophenyl)ethyne (4f).

A mixture of 4-iodebenzonitrile (0.2290 g, 1.0 mmol), 4-ethynylbenzonitrile (0.1271 g, 1.0 mmol), $Pd(PPh_3)$ (57.7 mg, 0.05 mmol), CuI (3.81 mg, 0.02 mmol) in diisopropylamine (1.0 mL) and THF (7 mL) at 40 °C under an Ar atmosphere was stirred for 24 h. After the reaction, the reaction was quenched with distilled water. The solution was extracted with ethyl acetate, washed with brine, dried over anhydrous $MgSO_4$, and concentrated under reduced pressure. The residue was washed with chloroform to afford **4e** in 41% yield (0.0935 g, 0.41 mmol).

1,2-Bis(4-cyanophenyl)ethyne (**4f**).[11] White solid; m.p. 253-255 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.63 (dd, *J* = 9.6 and 2.9 Hz, 4H), 7.69 (dd, *J* = 6.5 and 1.9 Hz, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ: 91.5, 112.4, 118.2, 127.0, 132.1, 132.2; EI-MS *m/z* (rel intensity): 228 (M^+, 100).

Reference

[1] H. Hazimeh, J.-M. Mattalia, C. Marchi-Delapierre, F. Kanoufi, C. Combellas, M. Chanon, *Eur. J. Org. Chem.* **2009**, 2775-2787.

[2] S. H. Pine, E. M. Munemo, T. M. Phillips, G. Bartorini, W. D. Cotton, G. C. Andrews, G. C. *J. Org. Chem.* **1971**, *36*, 984-991.

[3] S. Fustero, E. Rodríguez, R. Lázaro, L. Herrera, S. Catalán, P. Barrio, *Adv. Synth. Catal.* **2013**, *355*, 1058-1064.

[4] M. Chaumontet, R. Piccardi, N. Audic, J. Hitce, J.-L. Peglion, E. Clot, B. Olivier, *J. Am. Chem. Soc.* **2008**, *130*, 15157-15166.

[5] T. Beryozklna, P. Appukkuttan, N. Mont, E. V. Eycken, *Org. Lett.* **2006**, *8*, 487-490.

[6] Y. Landais, J.-P. Robin, A. Lebrun, *Tetrahedron* **1991**, *47*, 3787-3804.

[7] A. N. Kasatkin, G. Checksfield, R. J. Whitby, *J. Org. Chem.* **2000**, *65*, 3236-3238.

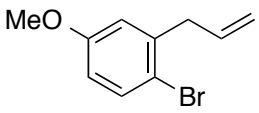
[8] J. Srogl, G. D. Allred, L. S. Liebeskind, *J. Am. Chem. Soc.* **1997**, *119*, 12376-12377.

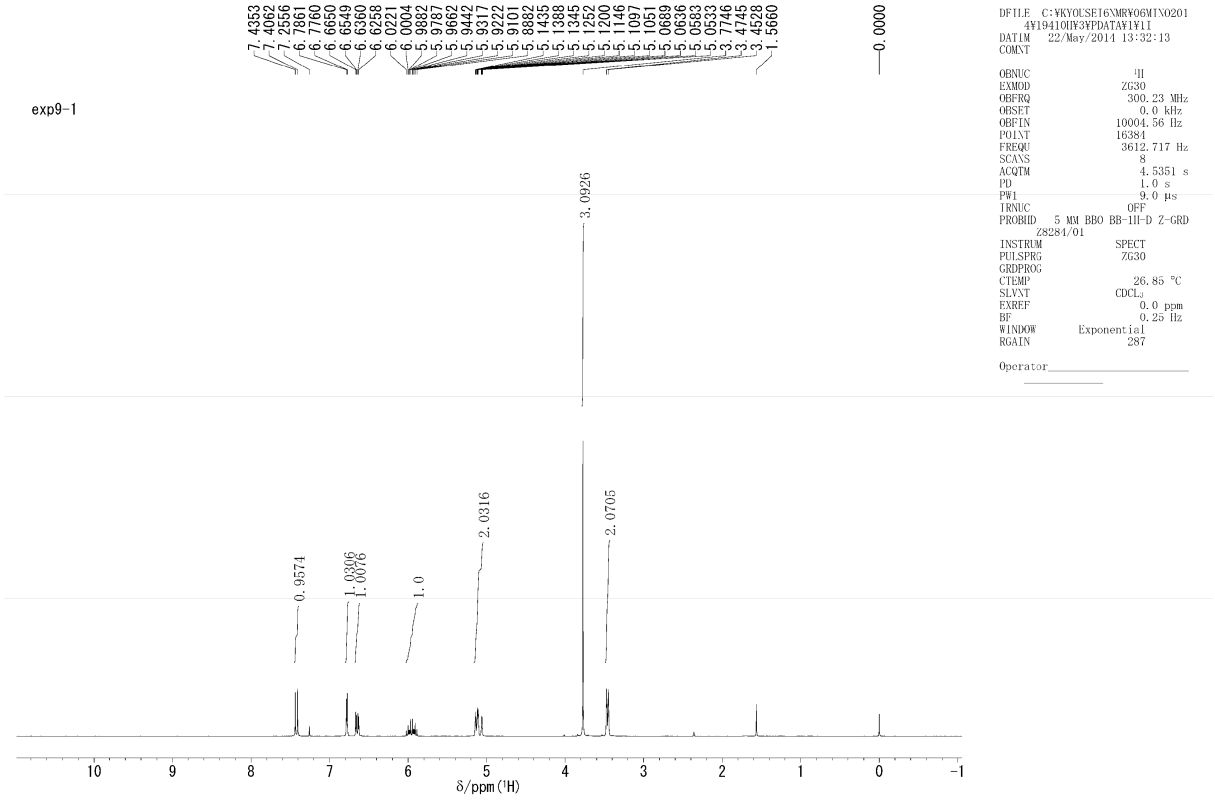
[9] M. L. N. Rao, D. N. Jadhav, P. Dasgupta, *Org. Lett.* **2010**, *12*, 2048-2051.

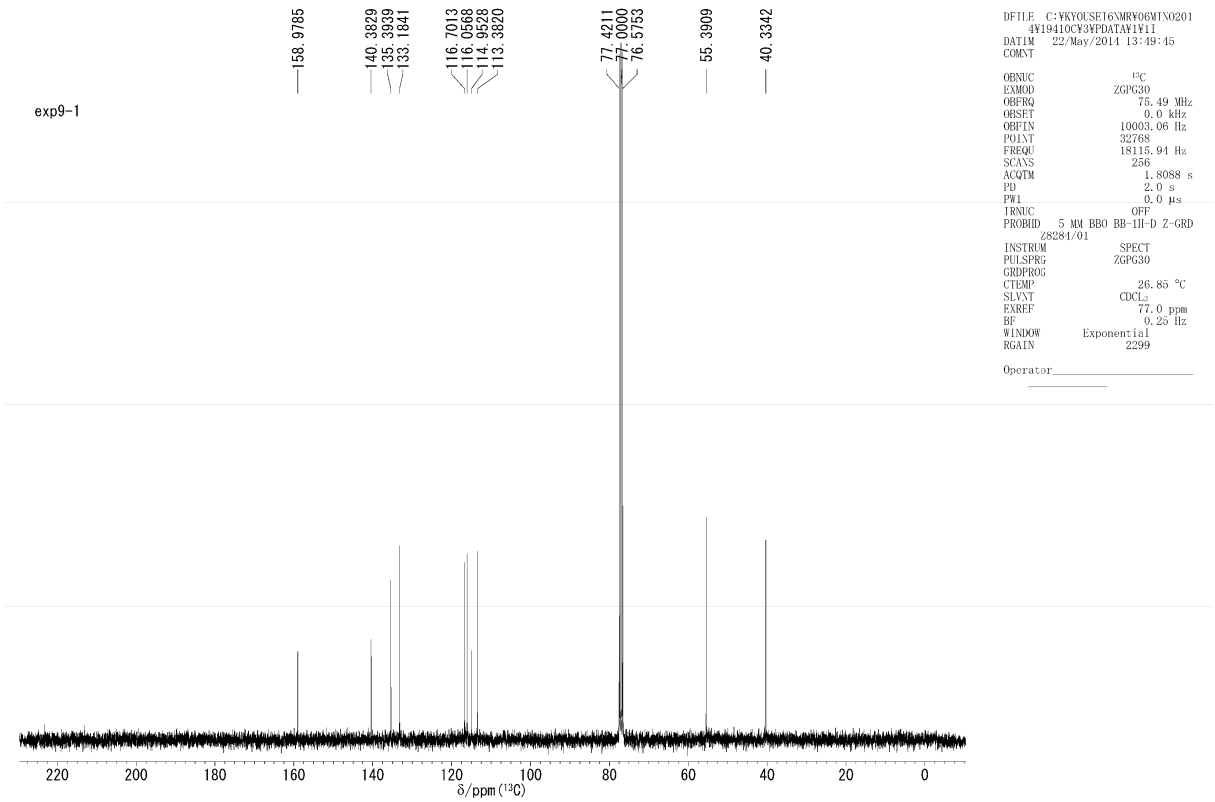
[10] N. Sakai, R. Komatsu, N. Uchida, R. Ikeda, T. Konakahara, *Org. Lett.* **2010**, *12*, 1300-1303.

[11] L. Melzig, A. Metzger, P. Knochel, *Chem. Eur. J.* **2011**, *17*, 2948-2956.

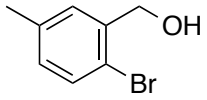
^{1}H and ^{13}C NMR of 1-Allyl-2-bromo-5-methoxybenzene (3b)

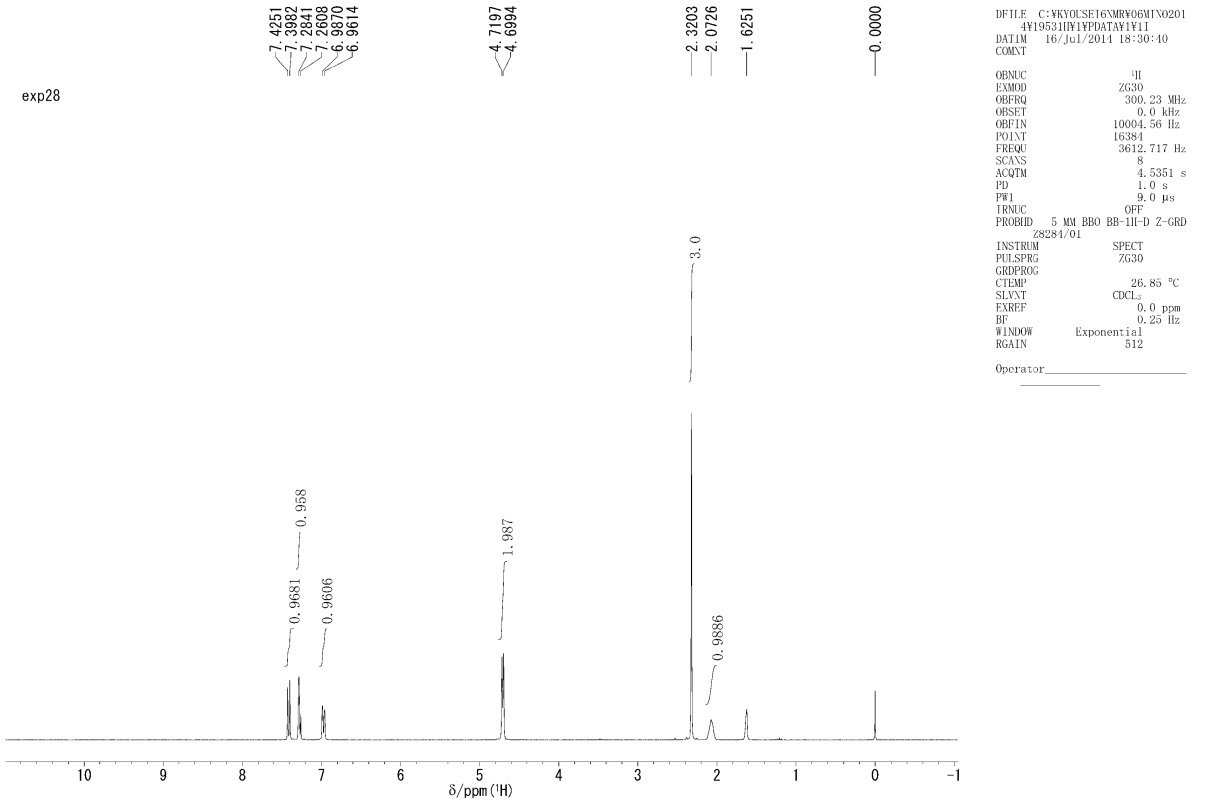


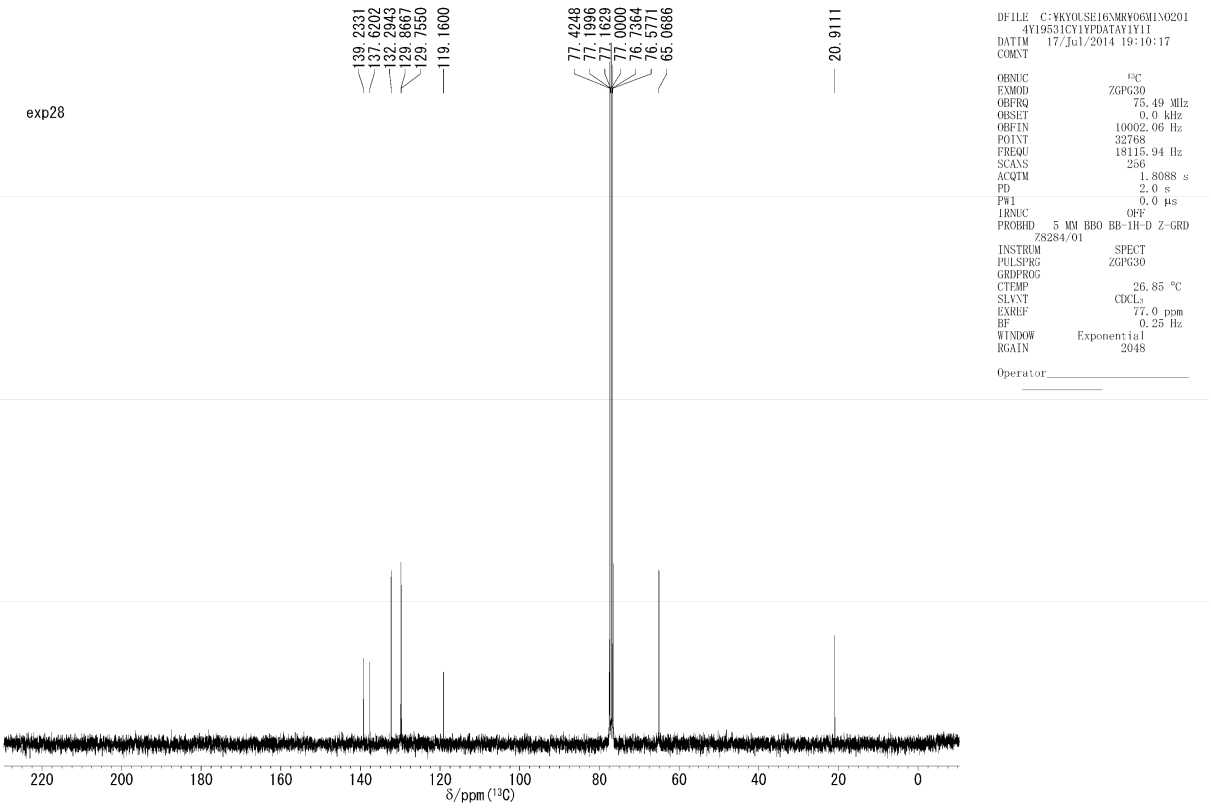




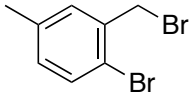
^{1}H and ^{13}C NMR of 2-Bromo-5-methyl-benzyl alcohol

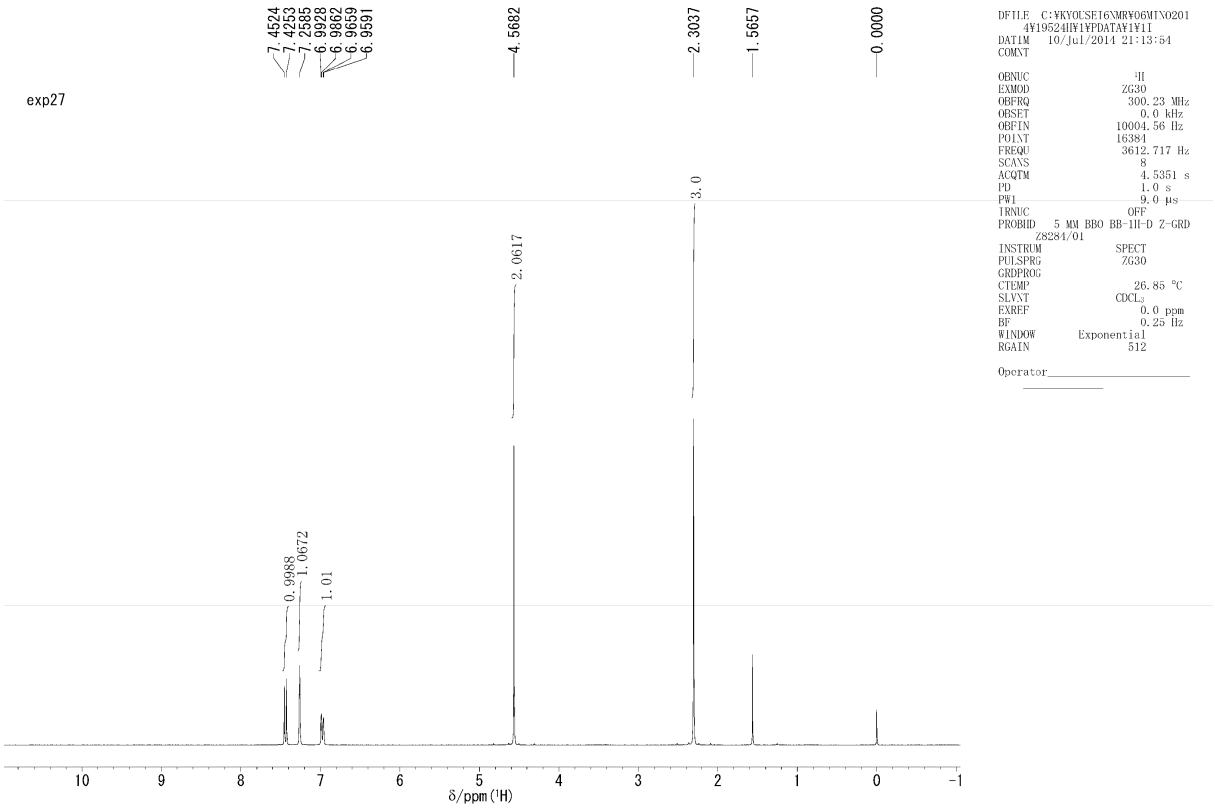


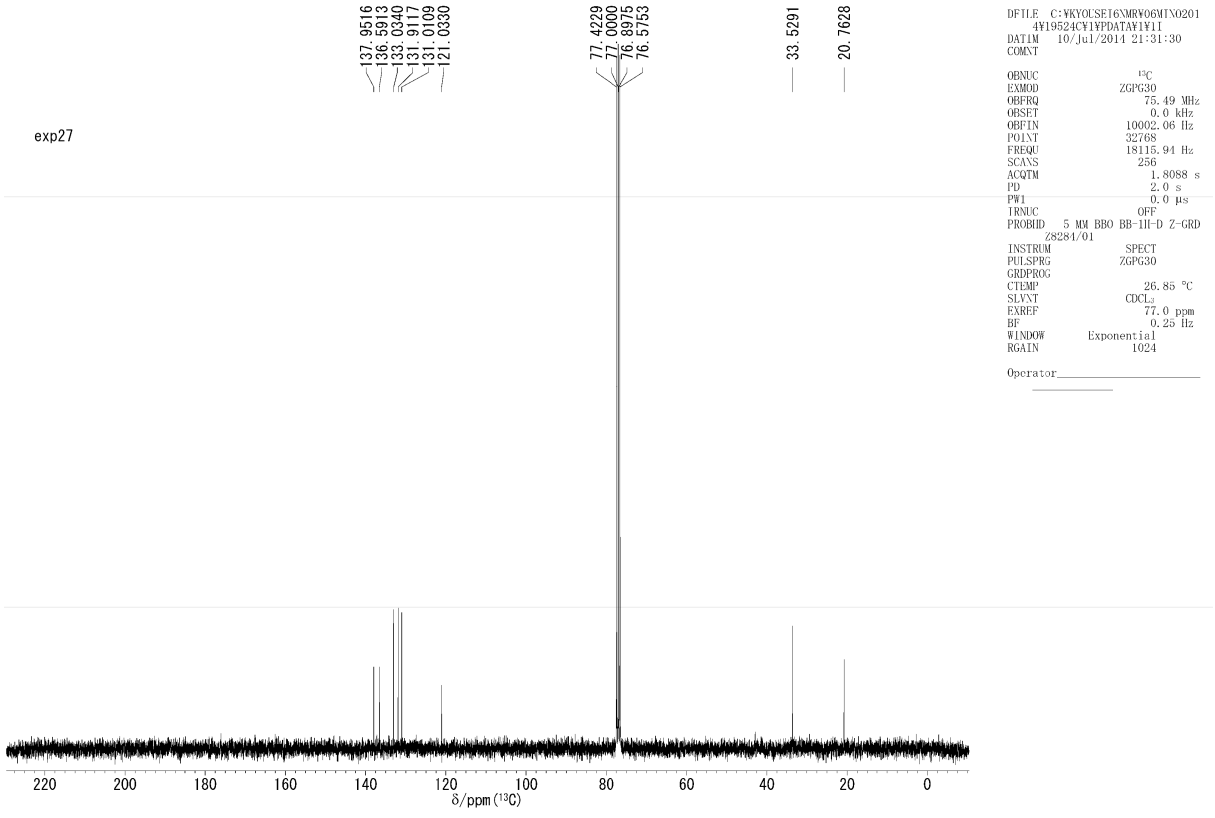




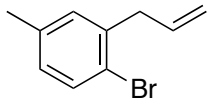
^{1}H and ^{13}C NMR of 2-Bromo-5-methyl-benzyl bromide

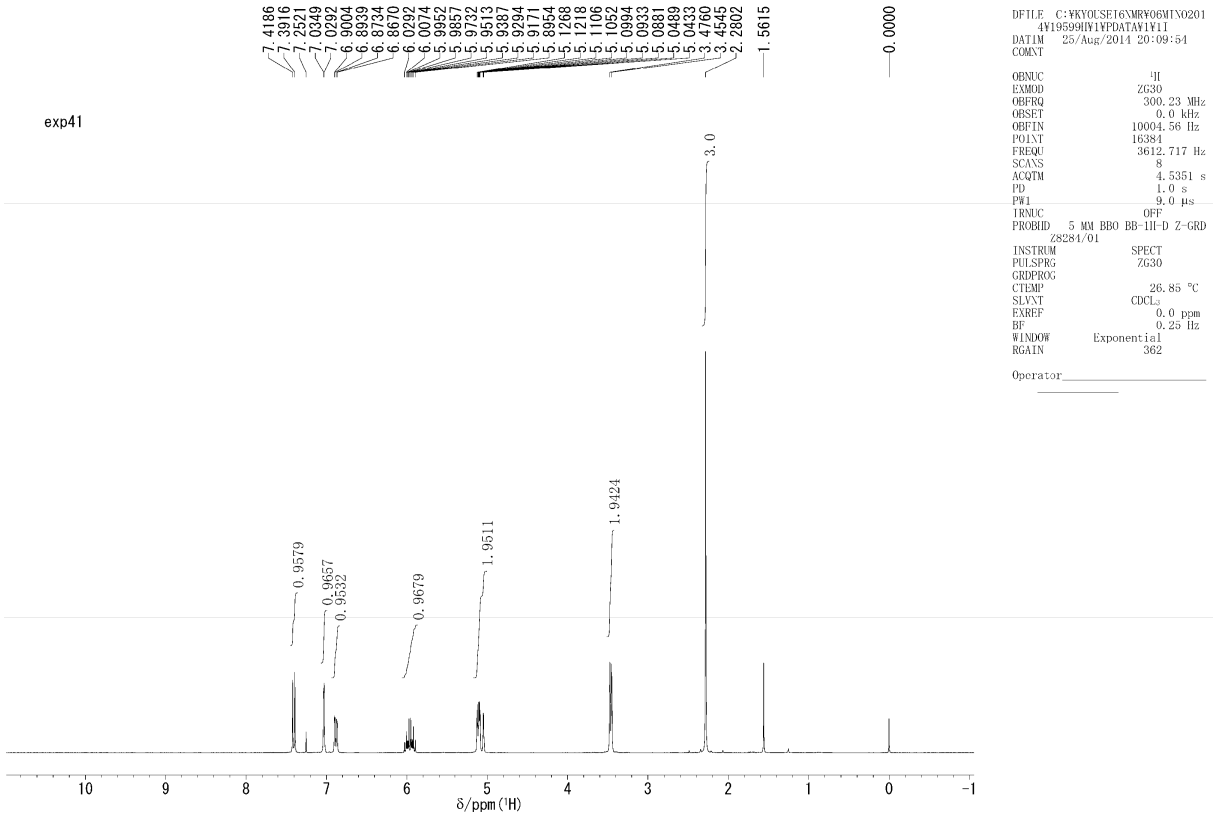






^{1}H and ^{13}C NMR of 1-Allyl-2-bromo-5-methylbenzene (3c)

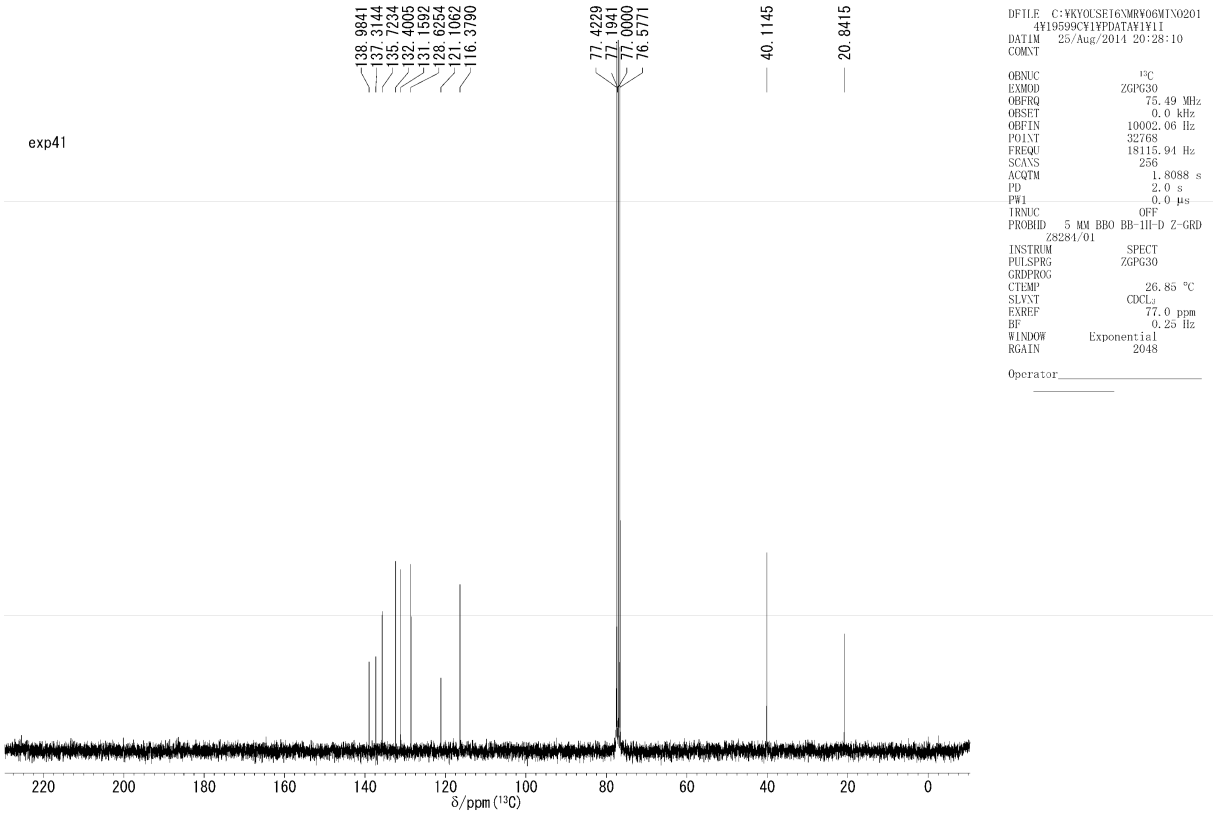




DFILE C:¥KYOUSEI6¥MR¥06MTX0201 4¥19599H¥1¥PDATA¥1¥1I
DATIM 25/Aug/2014 20:09:54
COMNT

OBNUC	^{1}H
EXMOD	ZG30
OBFRQ	300.23 MHz
OBSET	0.0 kHz
OBFIN	10004.56 Hz
PO1NT	16384
FREQU	3612.717 Hz
SCANS	8
ACQTM	4.5351 s
PD	1.0 s
PW1	9.0 μs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCl_3$
EXREF	0.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	362

Operator_____

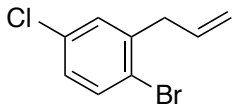


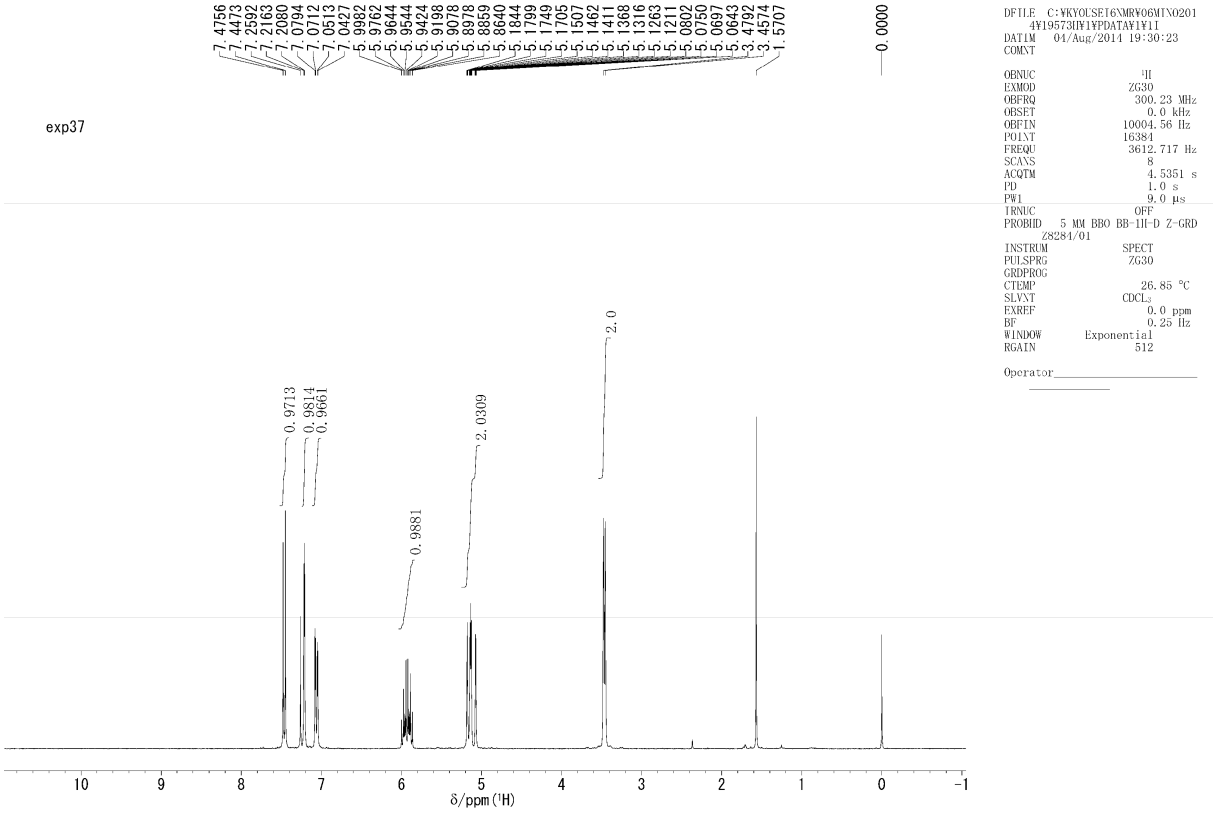
DFILE C:¥KYOUSEI6¥MR¥06MTX0201 4¥19599C¥1¥PDATA¥1¥1I
DATIM 25/Aug/2014 20:28:10
COMNT

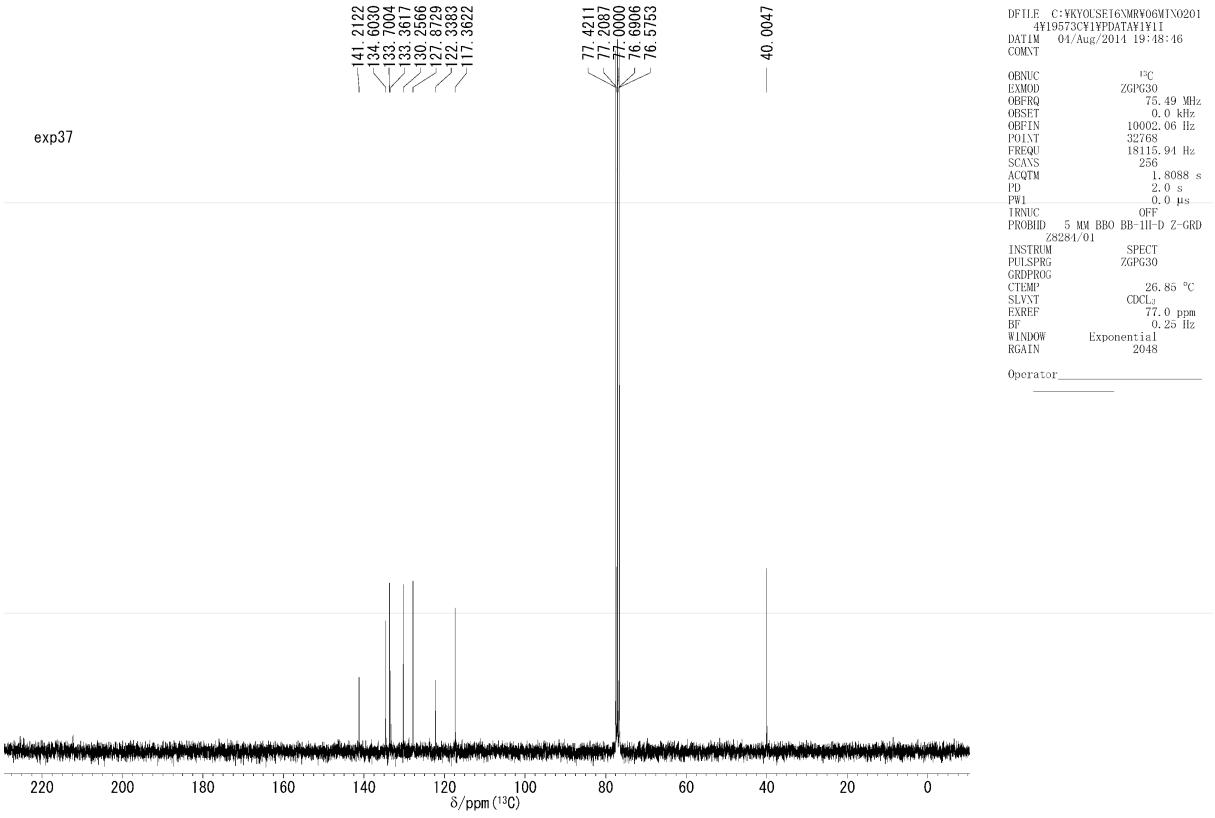
OBNUC	^{13}C
EXMOD	ZGPG30
OBFRQ	75.49 MHz
OBSET	0.0 kHz
OBFIN	10002.06 Hz
PO1NT	32768
FREQU	18115.94 Hz
SCANS	256
ACQTM	1.8088 s
PD	2.0 s
PW1	0.0 μs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZGPG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCl_3$
EXREF	77.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	2048

Operator_____

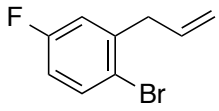
^{1}H and ^{13}C NMR of 1-Allyl-2-bromo-5-chlorobenzene (3d)

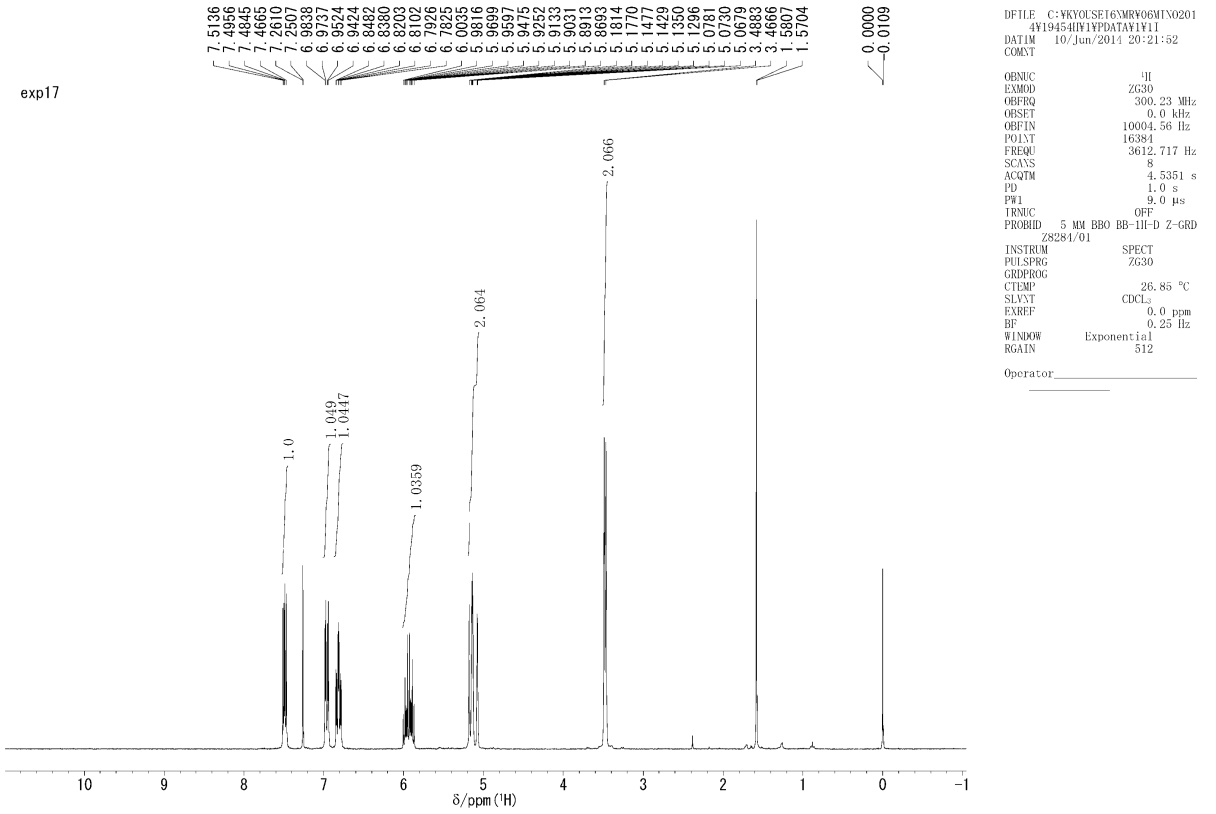






^{1}H and ^{13}C NMR of 1-Allyl-2-bromo-5-fluorobenzene (3e)

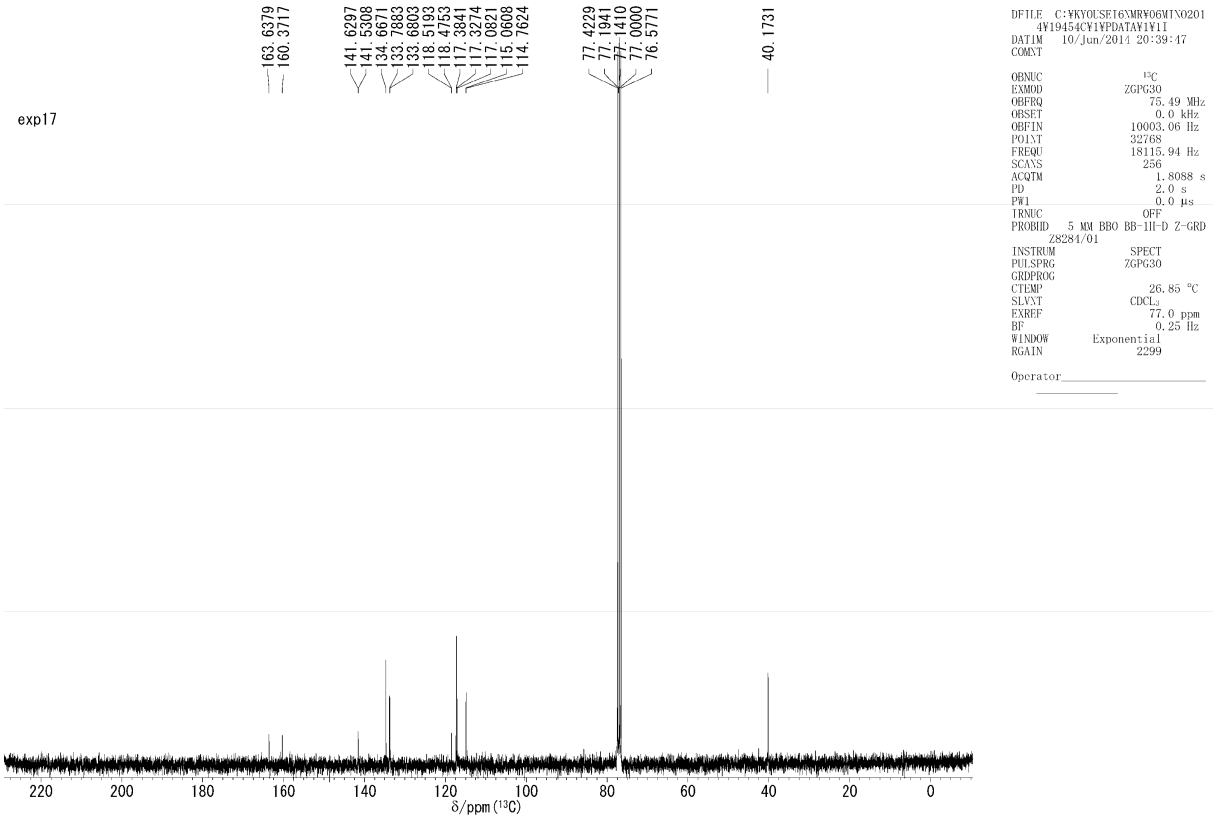




DFILE C:¥KYOUSEI6¥MR¥06MIX0201
4¥19454H¥1¥PDATA¥1¥1I
DATIM 10/Jun/2014 20:21:52
COMNT

OBNUC	¹H
EXMOD	ZG30
OBFRQ	300.23 MHz
OBSET	0.0 kHz
OBFIN	10004.56 Hz
POINT	16384
FREQU	3612.717 Hz
SCANS	8
ACQTM	4.5351 s
PD	1.0 s
PW1	9.0 μs
TRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCl_3$
EXREF	0.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	512

Operator_____

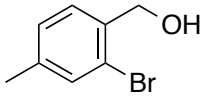


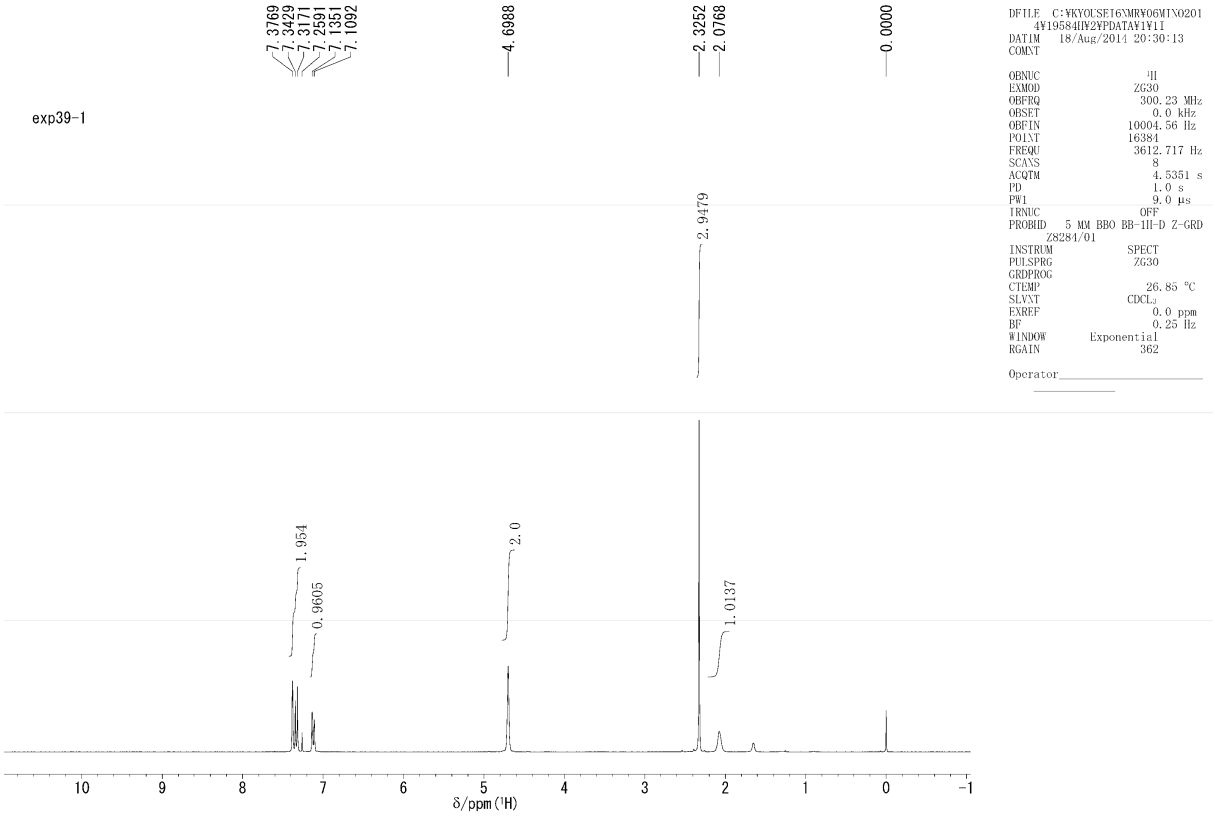
DFILE C:¥KYOUSEI6¥MR¥06MIX0201
4¥19454CY¥1¥PDATA¥1¥1I
DATIM 10/Jun/2014 20:39:47
COMNT

OBNUC	¹³C
EXMOD	ZGPG30
OBFRQ	75.49 MHz
OBSET	0.0 kHz
OBFIN	10003.06 Hz
POINT	32768
FREQU	18115.94 Hz
SCANS	256
ACQTM	1.8088 s
PD	2.0 s
PW1	0.0 μs
TRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZGPG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCl_3$
EXREF	77.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	2299

Operator_____

^{1}H and ^{13}C NMR of 2-Bromo-4-methyl-benzyl alcohol

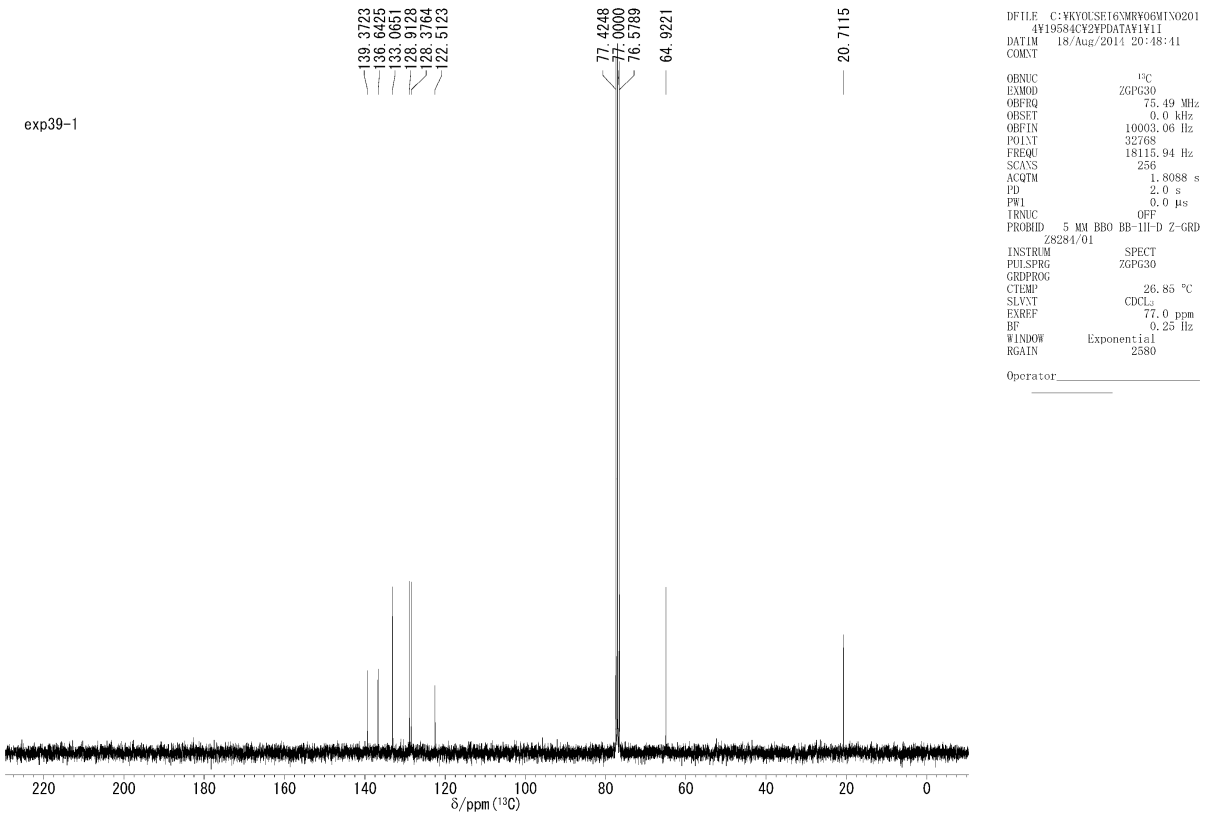




DFILE C:¥KYOUSEI6¥NMR¥06MIX0201 4¥19584H¥2¥PDATA¥1¥1I
DATIM 18/Aug/2014 20:30:13
COMNT

OBNUC	^{1}H
EXMOD	ZG30
OBFRQ	300.23 MHz
OBSET	0.0 kHz
OBFIN	10004.56 Hz
POINT	16384
FREQU	3612.717 Hz
SCANS	8
ACQTM	4.5351 s
PD	1.0 s
PW1	9.0 µs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCl_3$
EXREF	0.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	362

Operator

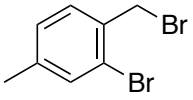


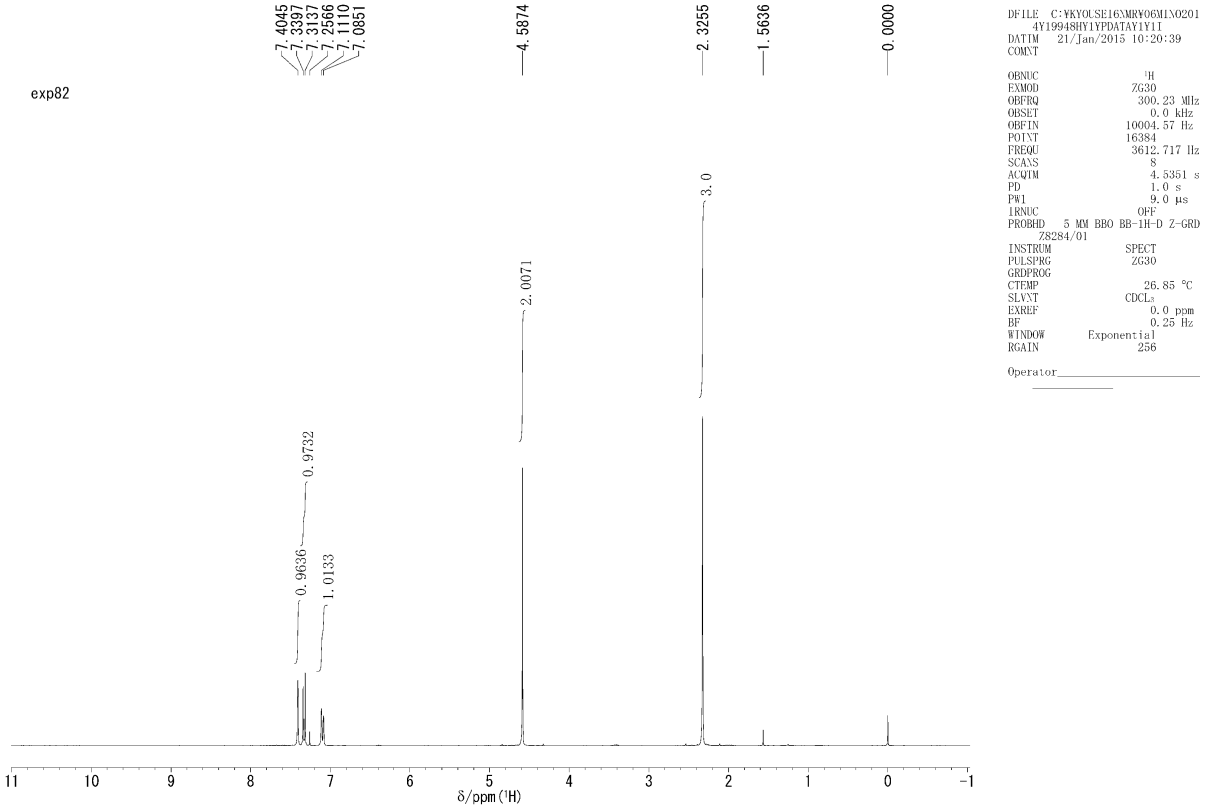
DFILE C:¥KYOUSEI6¥NMR¥06MIX0201 4¥19584C¥2¥PDATA¥1¥1I
DATIM 18/Aug/2014 20:48:41
COMNT

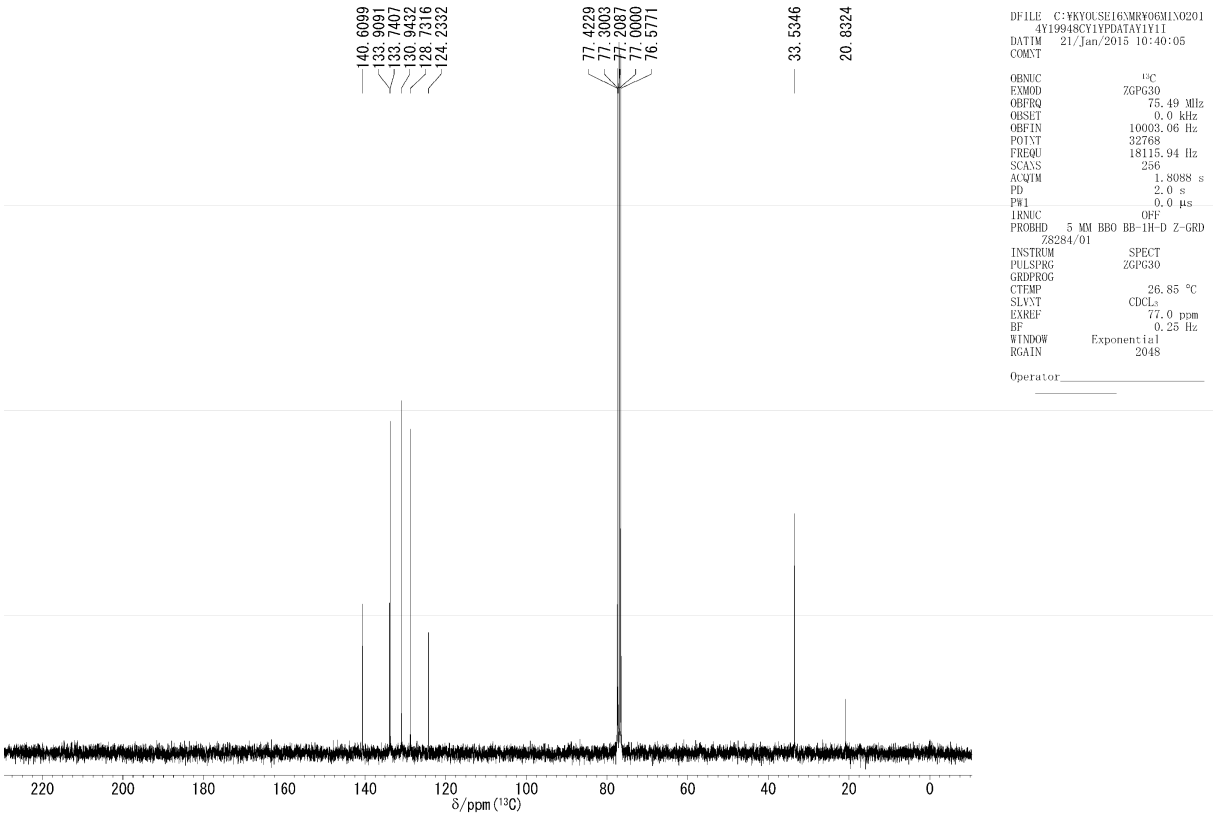
OBNUC	^{13}C
EXMOD	ZGPG30
OBFRQ	75.49 MHz
OBSET	0.0 kHz
OBFIN	10003.06 Hz
POINT	32768
FREQU	18115.94 Hz
SCANS	256
ACQTM	1.8088 s
PD	2.0 s
PW1	0.0 µs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZGPG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCl_3$
EXREF	77.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	2580

Operator

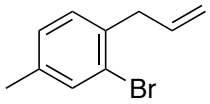
^{1}H and ^{13}C NMR of 2-Bromo-4-methyl-benzylbromide

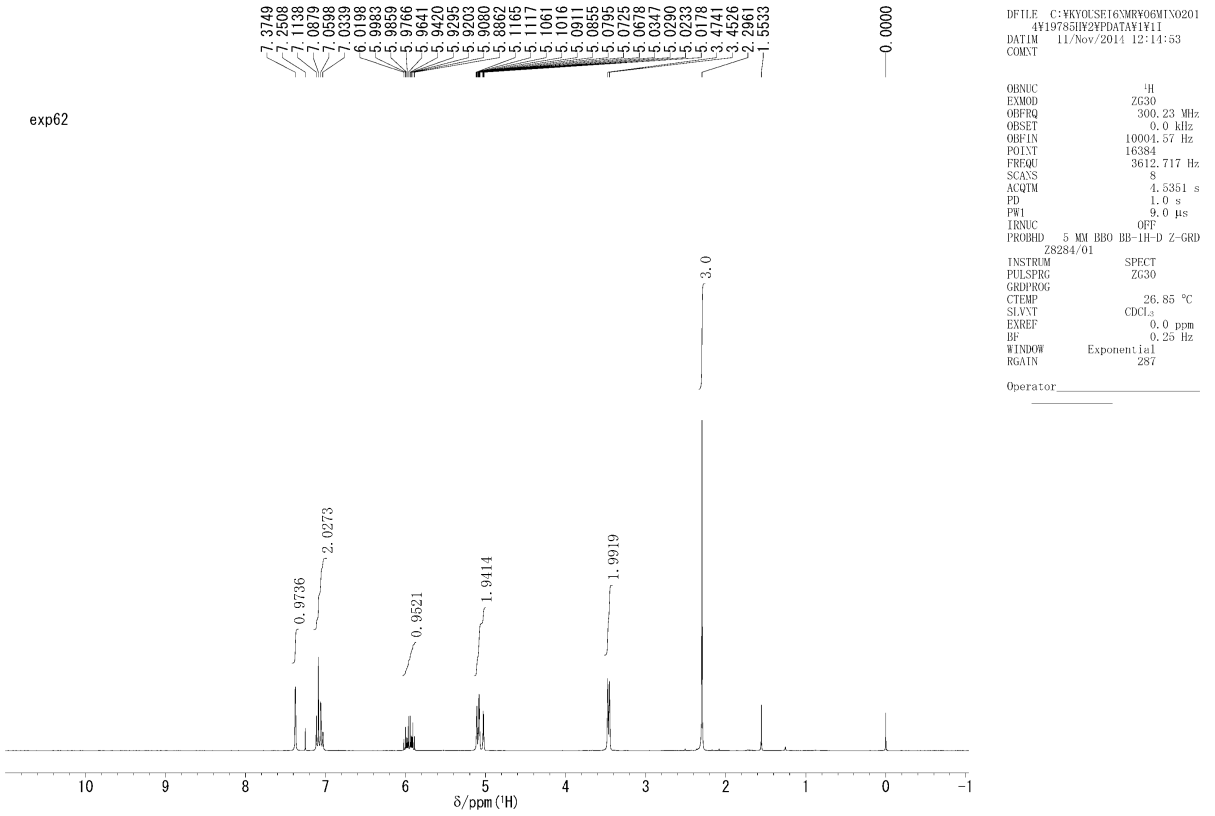




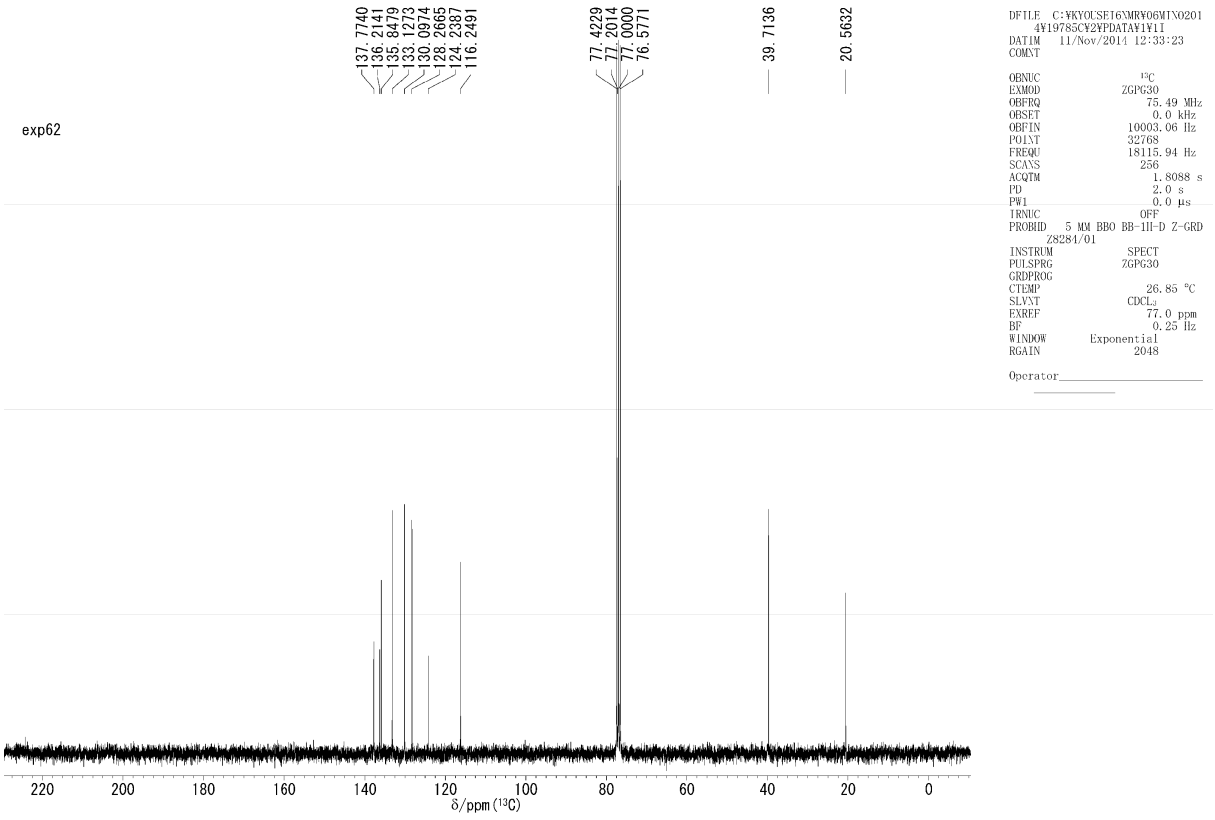


^{1}H and ^{13}C NMR of 1-Allyl-2-bromo-4-methylbenzene (3f)



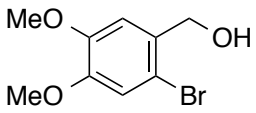


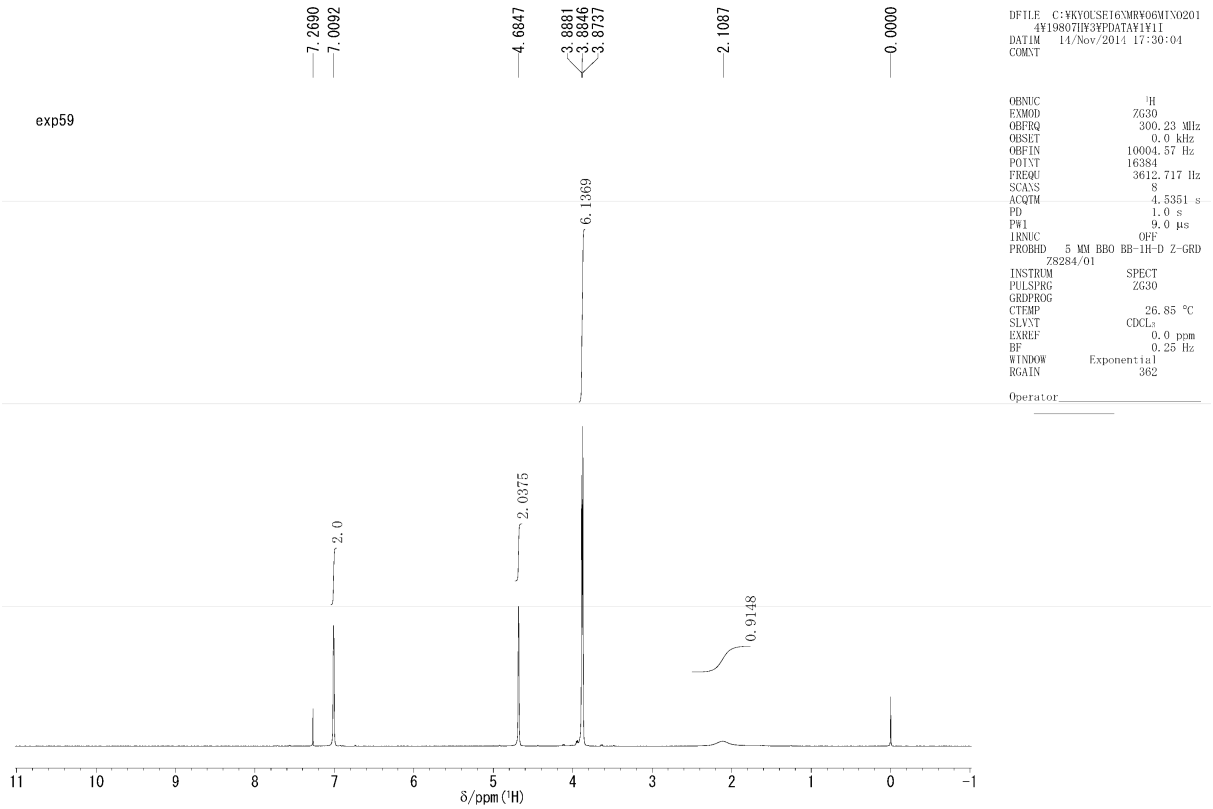

```
DFILE   C:¥KYOUSEI6¥MR¥06MIX0201
    4¥19785H¥2¥PDATA¥1¥1I
DATIM    11/Nov/2014 12:14:53
COMNT

OBNUC                   ¹H
EXMOD                 ZG30
OBFRQ               300.23 MHz
OBSET                 0.0 kHz
OBFIN              10004.57 Hz
POINT              16384
FREQU               3612.717 Hz
SCANS                    8
ACQTM                4.5351 s
PD                   1.0 s
PW1                  9.0 µs
IRNUC                  OFF
PROBHD    5 MM BBO BB-1H-D Z-GRD
        Z8284/01
INSTRUM              SPECT
PULSPRG               ZG30
GRDPROG
CTEMP               26.85 °C
SLVNT               CDCl₃
EXREF                 0.0 ppm
BF                    0.25 Hz
WINDOW         Exponential
RGAIN                  287

Operator_____________________
```

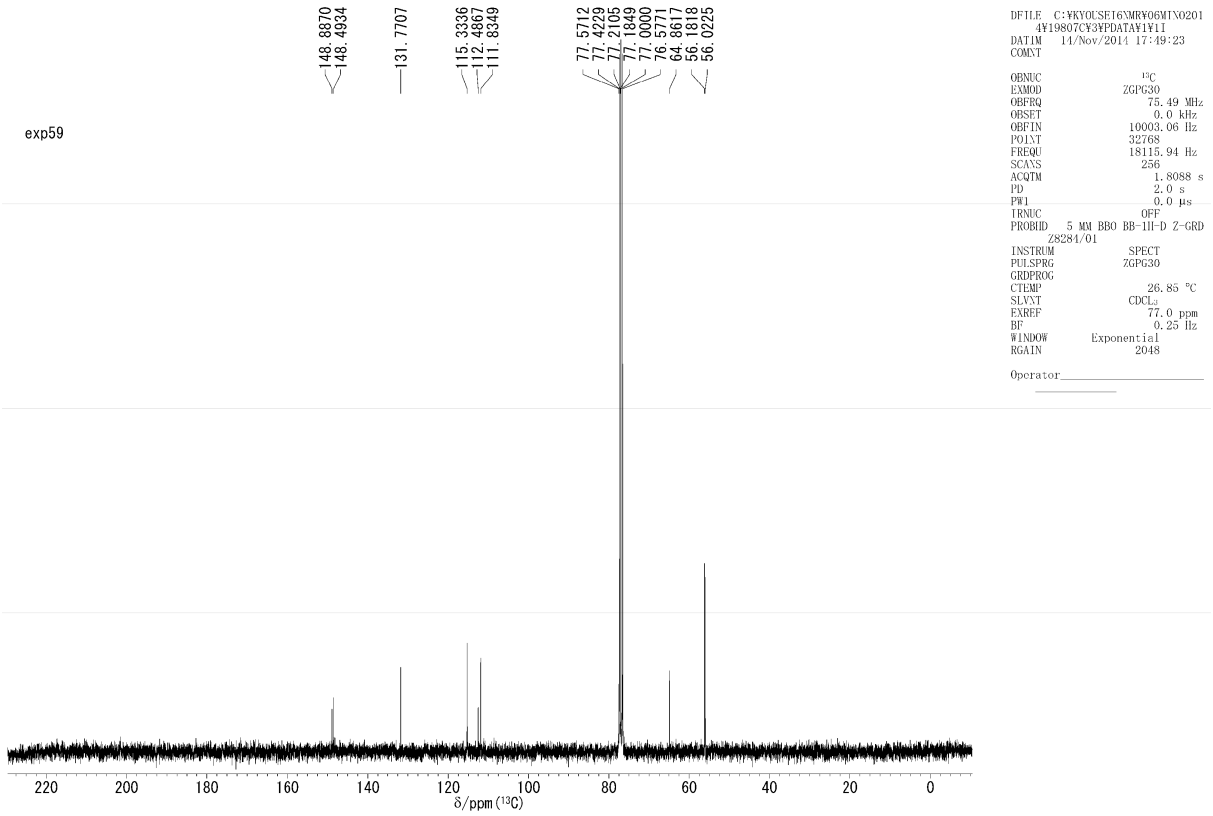



```
DFILE   C:¥KYOUSEI6¥MR¥06MIX0201
    4¥19785C¥2¥PDATA¥1¥1I
DATIM    11/Nov/2014 12:33:23
COMNT

OBNUC                  ¹³C
EXMOD                ZGPG30
OBFRQ               75.49 MHz
OBSET                 0.0 kHz
OBFIN              10003.06 Hz
POINT              32768
FREQU              18115.94 Hz
SCANS                  256
ACQTM                1.8088 s
PD                   2.0 s
PW1                  0.0 µs
IRNUC                  OFF
PROBHD    5 MM BBO BB-1H-D Z-GRD
        Z8284/01
INSTRUM              SPECT
PULSPRG              ZGPG30
GRDPROG
CTEMP               26.85 °C
SLVNT               CDCl₃
EXREF                77.0 ppm
BF                    0.25 Hz
WINDOW         Exponential
RGAIN                 2048

Operator_____________________
```

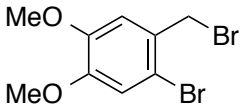
^{1}H and ^{13}C NMR of 2-Bromo-4,5-methoxy-benzyl alcohol.

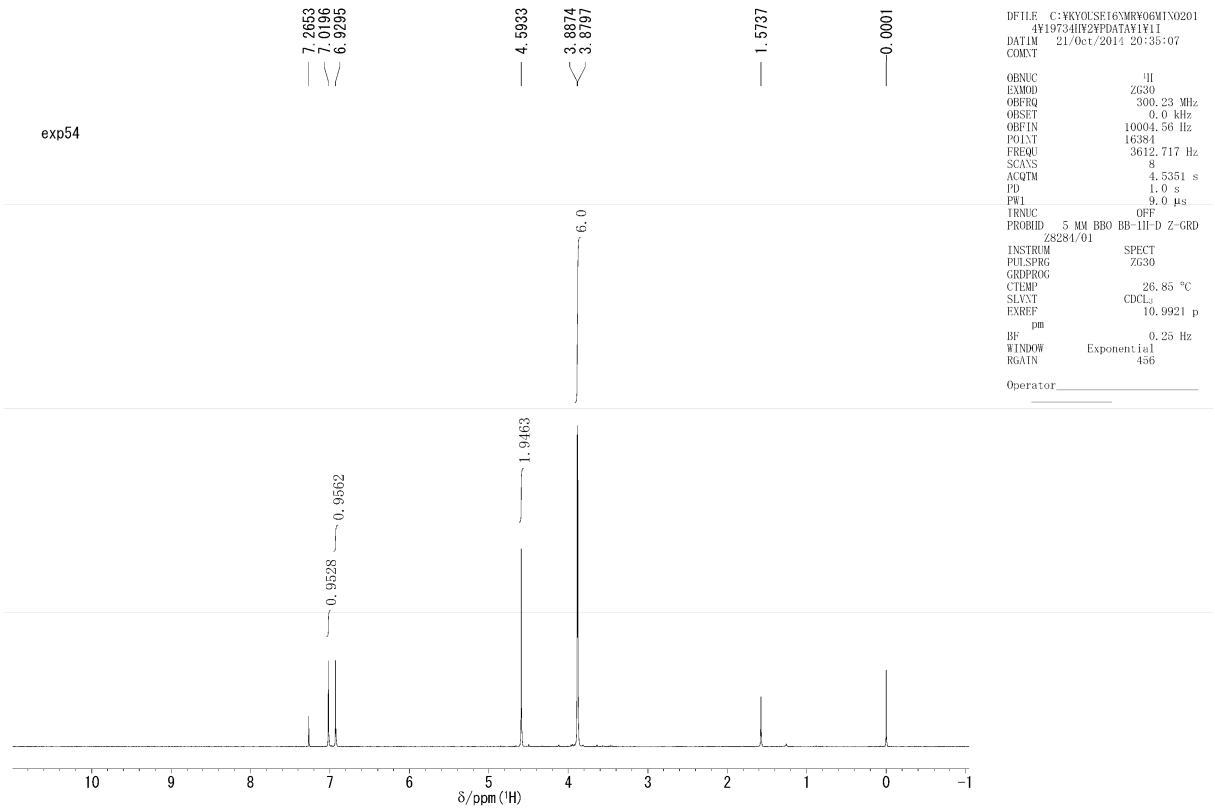


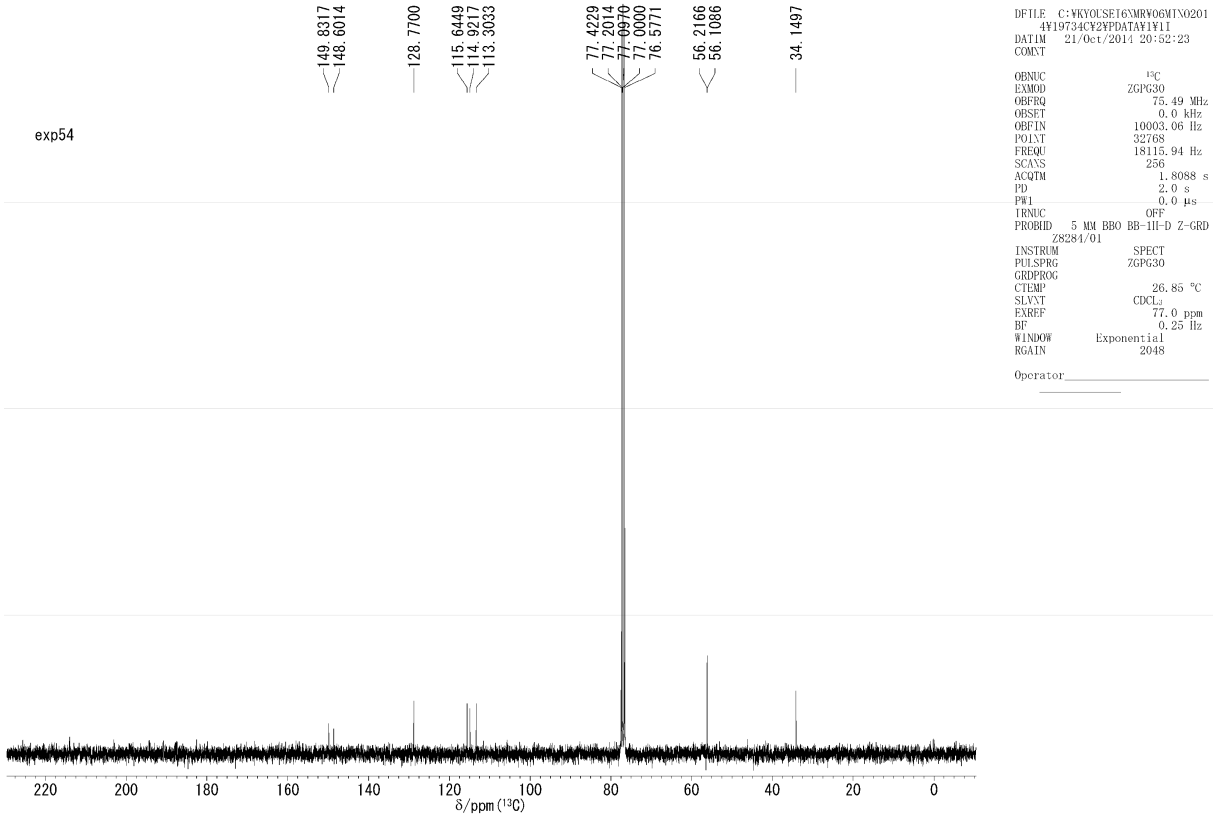




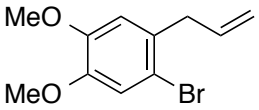
^{1}H and ^{13}C NMR of 2-Bromo-4,5-methoxybenzyl bromide

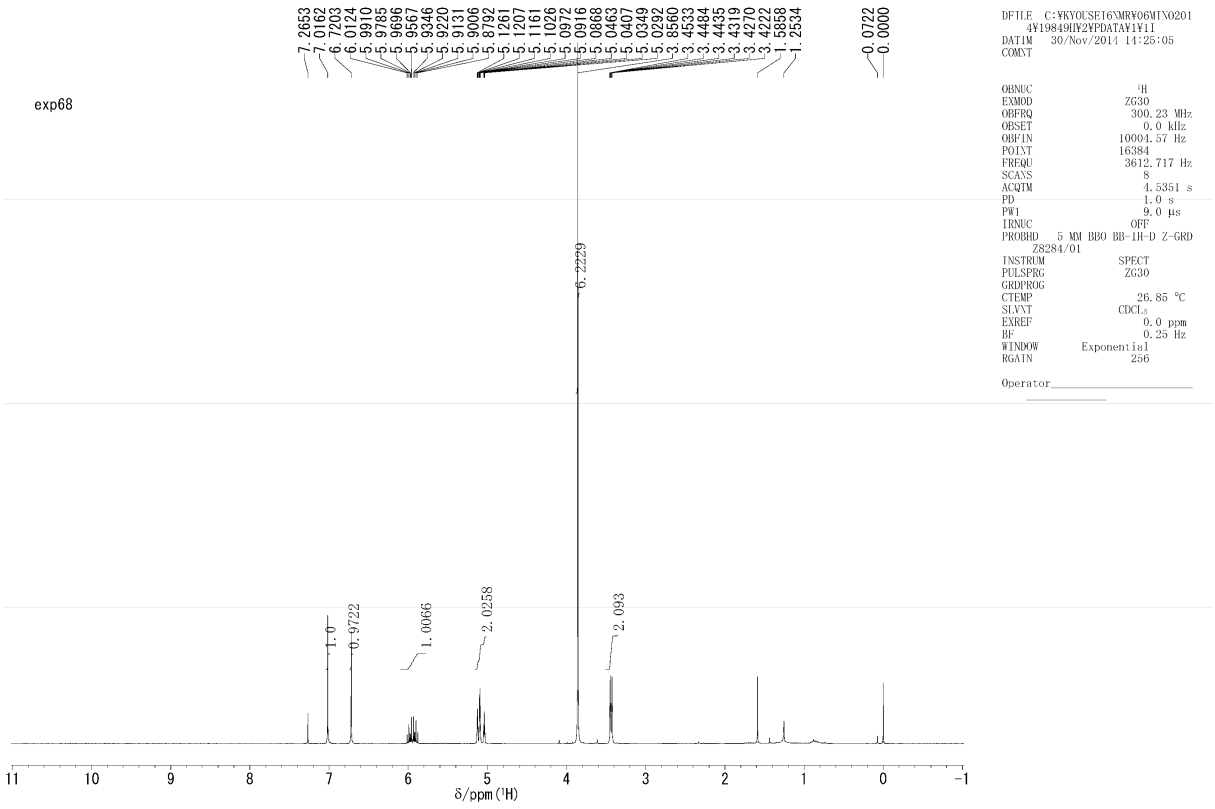


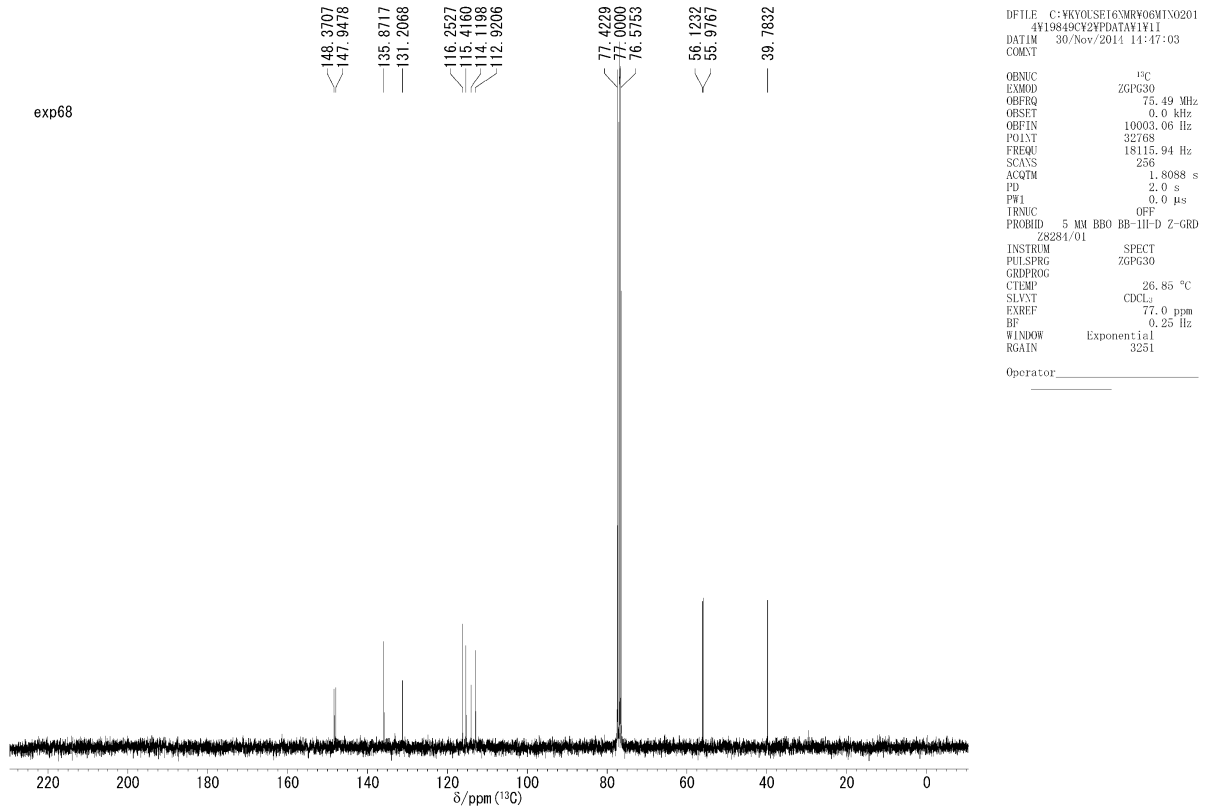




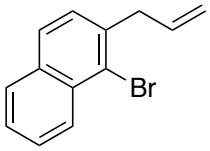
^{1}H and ^{13}C NMR of 1-Allyl-2-bromo-4,5-dimethoxybenzene (3g)

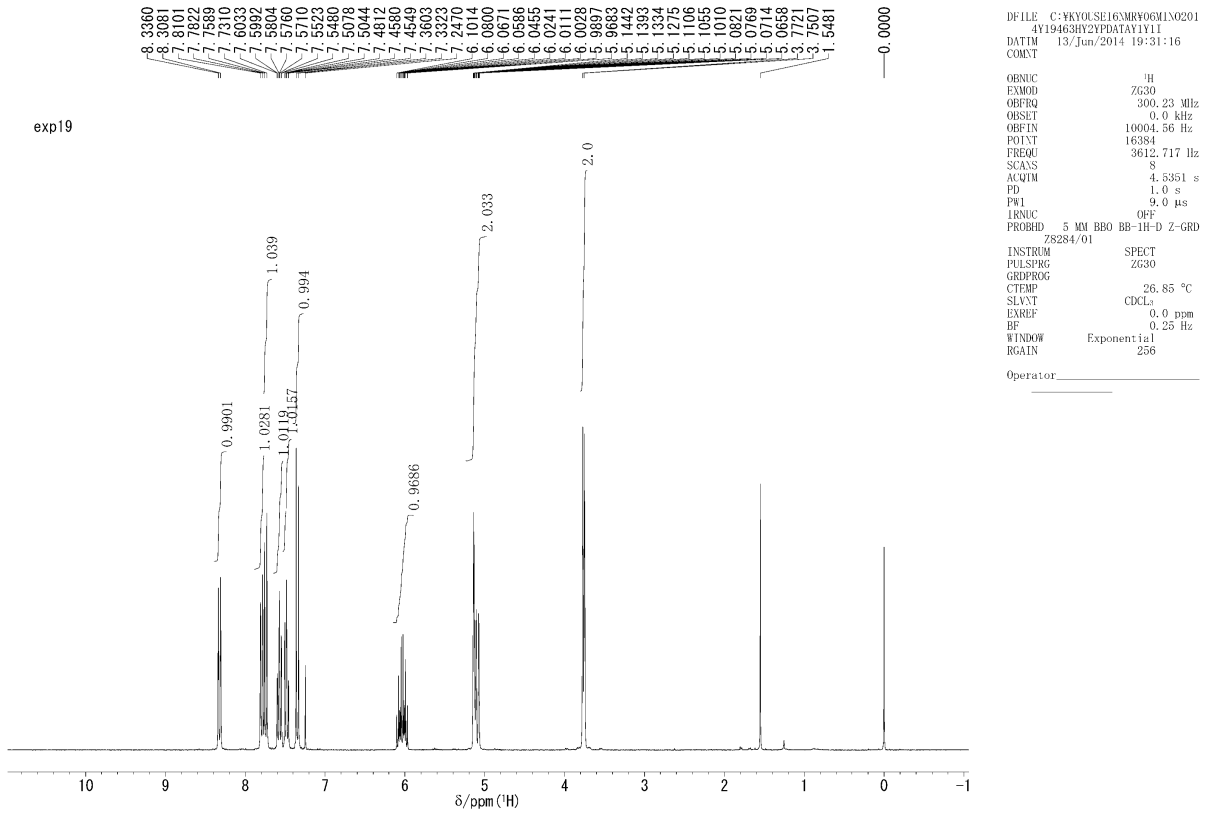






^{1}H and ^{13}C NMR of 2-Allyl-1-bromonaphathalene (3h)

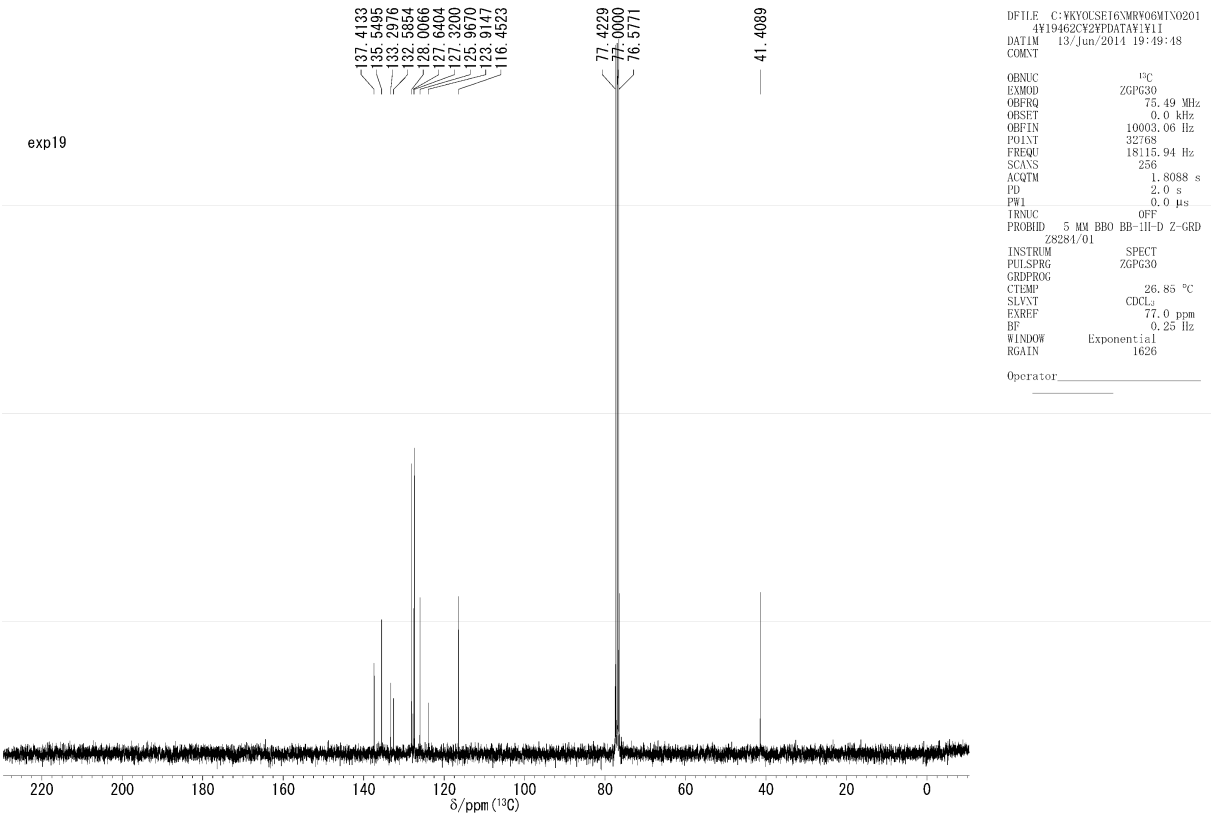




DFILE C:¥KYOUSE16¥MR¥O6M1N0201
4Y19463HY2¥PDATA¥1¥1I
DATIM 13/Jun/2014 19:31:16
COMNT

OBNUC ¹H
EXMOD ZG30
OBFRQ 300.23 MHz
OBSET 0.0 kHz
OBFIN 10004.56 Hz
POINT 16384
FREQU 3612.717 Hz
SCANS 8
ACQTM 4.5351 s
PD 1.0 s
PW1 9.0 µs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPROG
CTEMP 26.85 °C
SLVNT CDCL₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 256

Operator_____

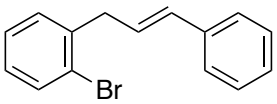


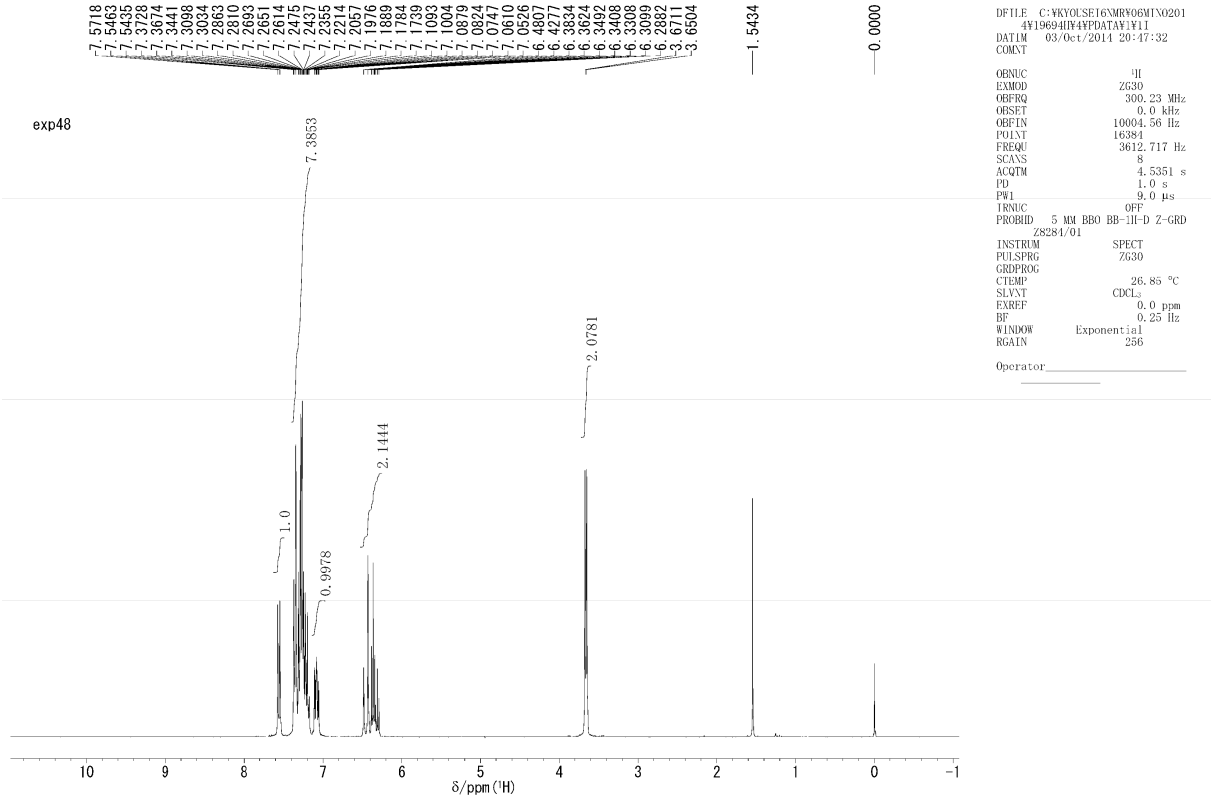
DFILE C:¥KYOUSE16¥MR¥O6M1N0201
4Y19462C¥2¥PDATA¥1¥1I
DATIM 13/Jun/2014 19:49:48
COMNT

OBNUC ¹³C
EXMOD ZGPG30
OBFRQ 75.49 MHz
OBSET 0.0 kHz
OBFIN 10003.06 Hz
POINT 32768
FREQU 18115.94 Hz
SCANS 256
ACQTM 1.8088 s
PD 2.0 s
PW1 0.0 µs
IRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM SPECT
PULSPRG ZGPG30
GRDPROG
CTEMP 26.85 °C
SLVNT CDCL₃
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 1626

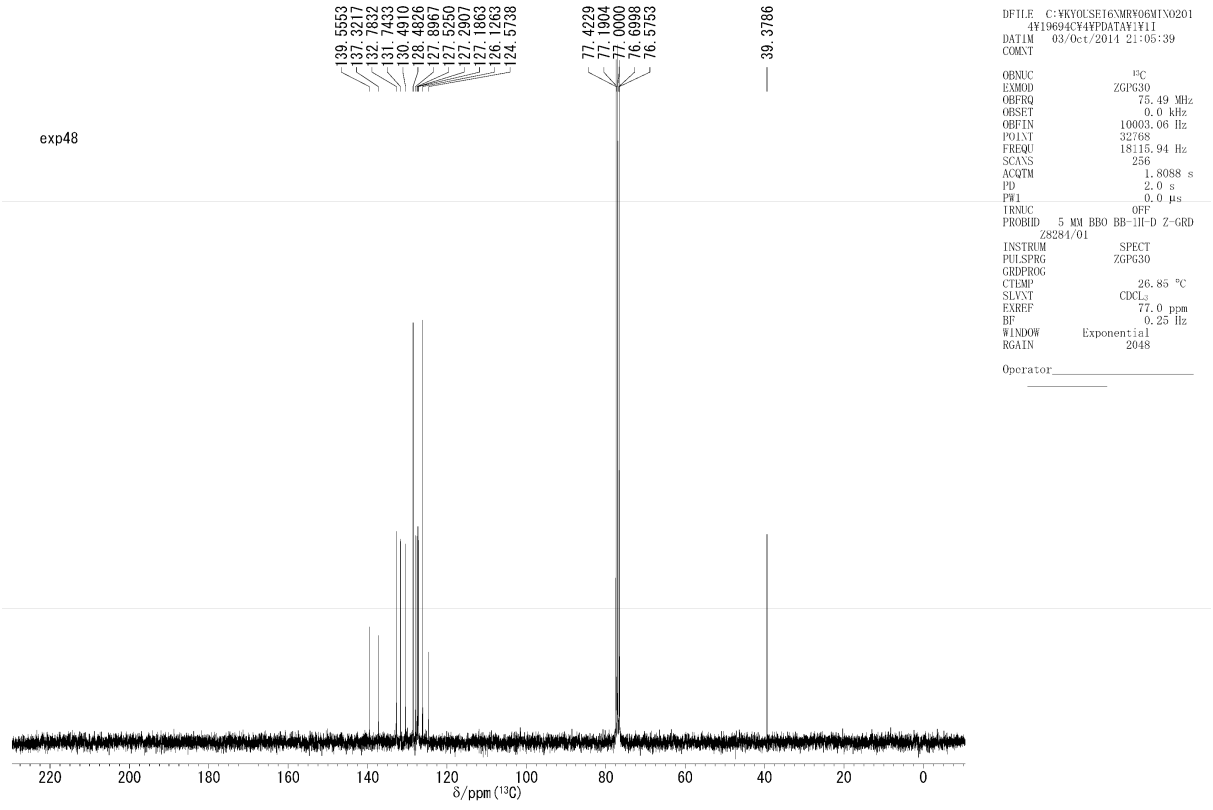
Operator_____

^{1}H and ^{13}C NMR of 1-bromo-2-cinnamylbenzene (3i)



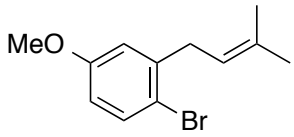


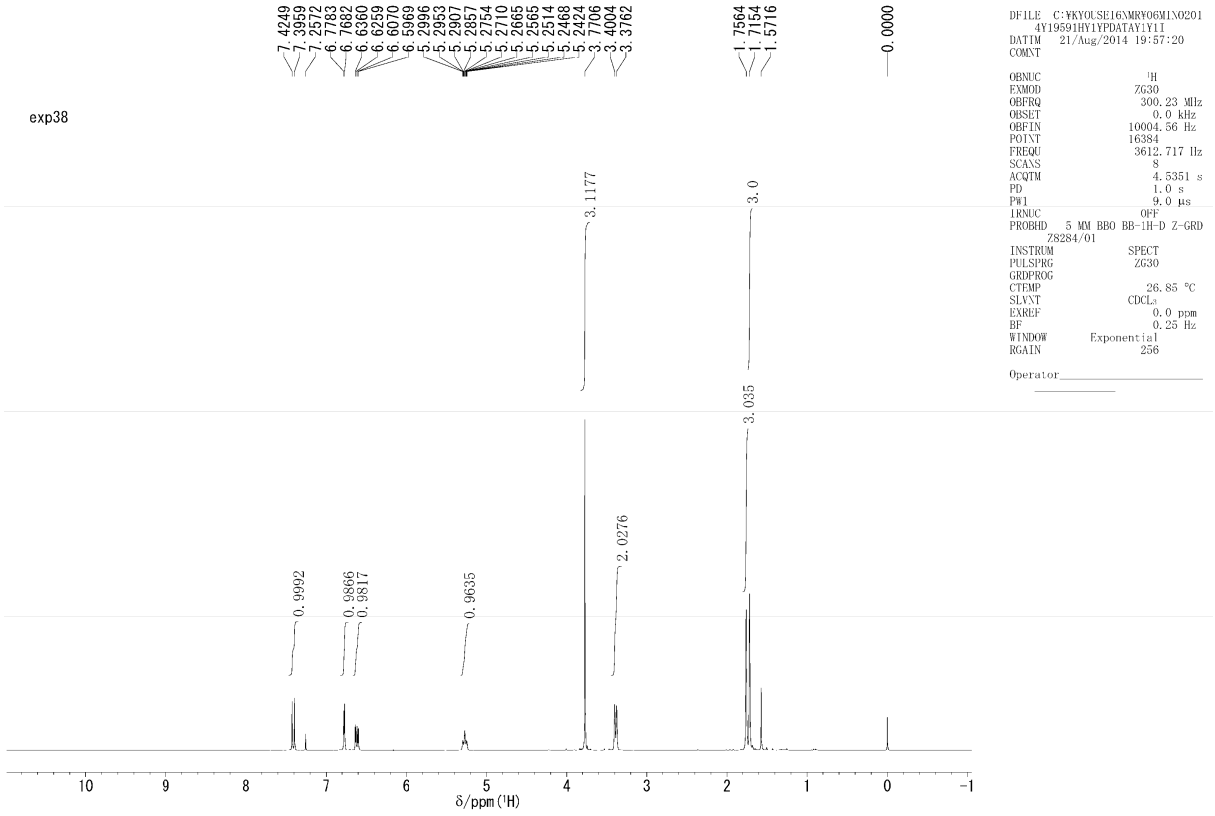

```
DFILE   C:¥KYOLSEI6¥NMR¥06MIX0201
     4¥19694H¥4¥PDATA¥1¥1I
DATIM    03/Oct/2014 20:47:32
COMNT

OBNUC                    1H
EXMOD                  ZG30
OBFRQ                300.23 MHz
OBSET                  0.0 kHz
OBFIN              10004.56 Hz
POINT                  16384
FREQU               3612.717 Hz
SCANS                     8
ACQTM                4.5351 s
PD                     1.0 s
PW1                    9.0 µs
IRNUC                   OFF
PROBHD   5 MM BBO BB-1H-D Z-GRD
     Z8284/01
INSTRUM                SPECT
PULSPRG                ZG30
GRDPROG
CTEMP                 26.85 °C
SLVNT                 CDCL3
EXREF                  0.0 ppm
BF                     0.25 Hz
WINDOW          Exponential
RGAIN                   256

Operator
```

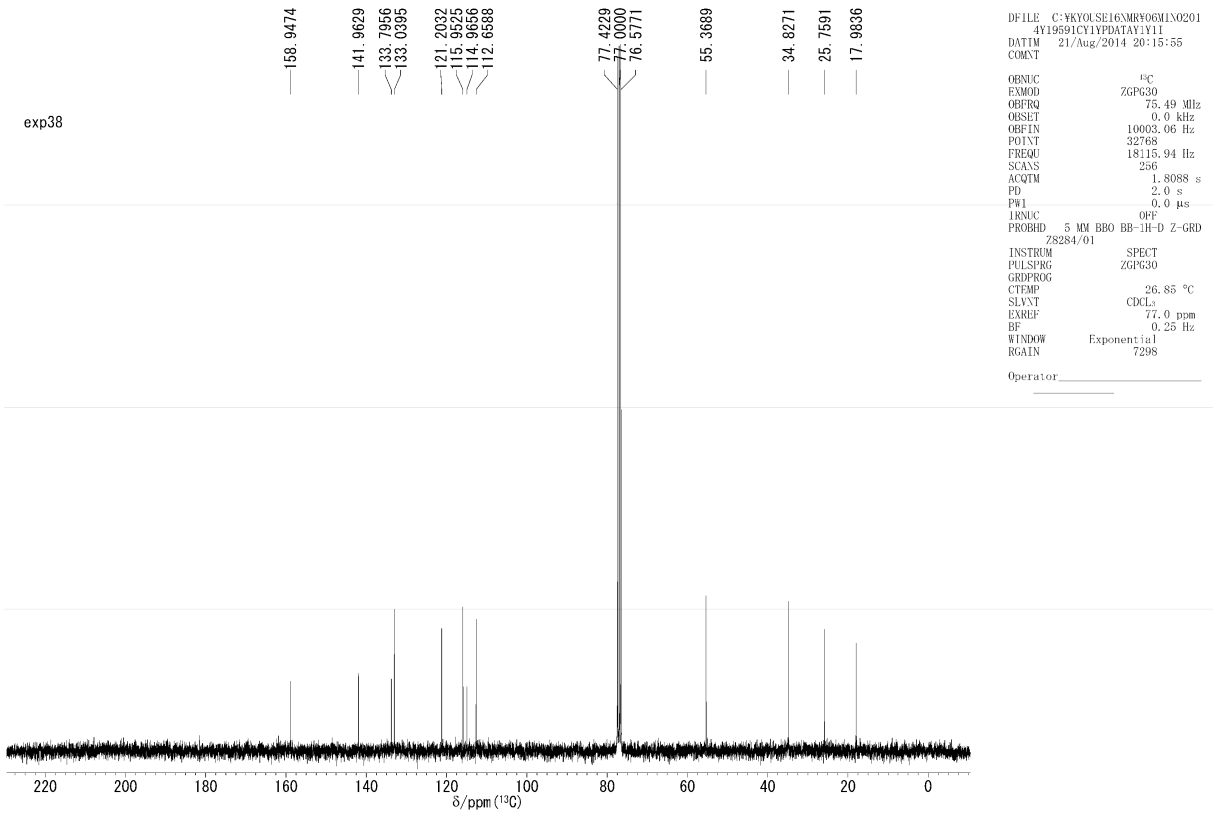



```
DFILE   C:¥KYOLSEI6¥NMR¥06MIX0201
     4¥19694C¥4¥PDATA¥1¥1I
DATIM    03/Oct/2014 21:05:39
COMNT

OBNUC                   13C
EXMOD                ZGPG30
OBFRQ                75.49 MHz
OBSET                  0.0 kHz
OBFIN              10003.06 Hz
POINT                  32768
FREQU              18115.94 Hz
SCANS                   256
ACQTM                1.8088 s
PD                     2.0 s
PW1                    0.0 µs
IRNUC                   OFF
PROBHD   5 MM BBO BB-1H-D Z-GRD
     Z8284/01
INSTRUM                SPECT
PULSPRG              ZGPG30
GRDPROG
CTEMP                 26.85 °C
SLVNT                 CDCL3
EXREF                 77.0 ppm
BF                     0.25 Hz
WINDOW          Exponential
RGAIN                  2048

Operator
```

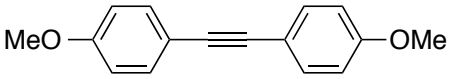
^{1}H and ^{13}C NMR of 1-Bromo-4-methoxy-2-prenylbenzene (3j)

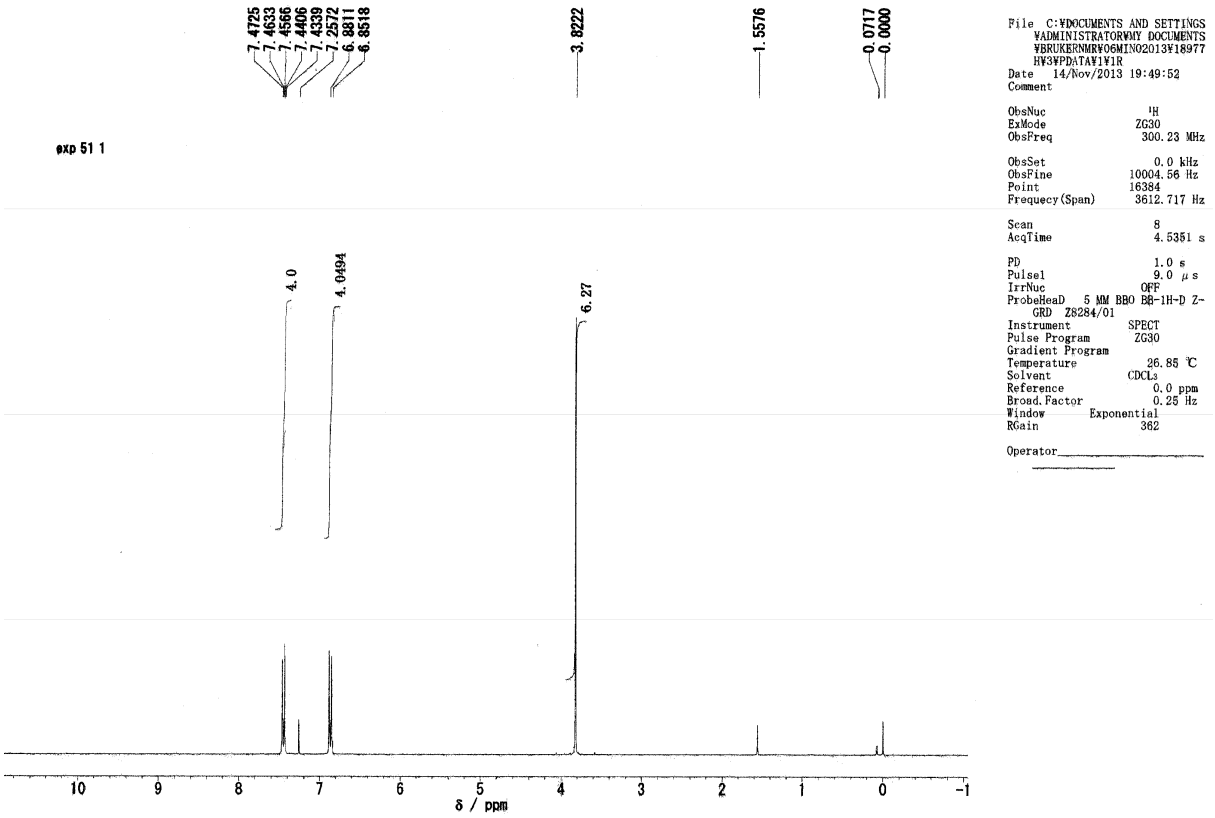






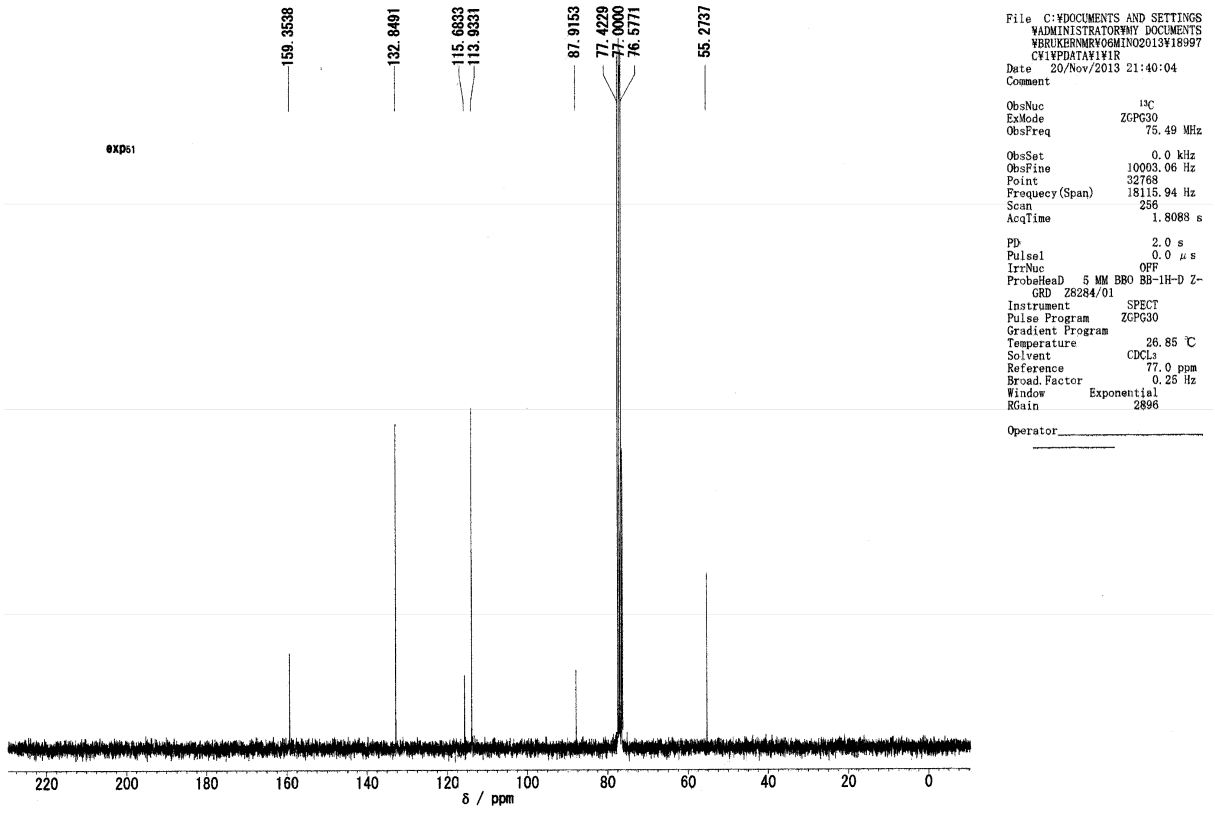
^{1}H and ^{13}C NMR of 1,2-Bis(4-methoxyphenyl)ethyne (4b)





File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MINO2013¥18977 HY3¥PDATA¥1¥1R
Date 14/Nov/2013 19:49:52
Comment

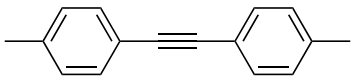
ObsNuc	^{1}H
ExMode	ZG30
ObsFreq	300.23 MHz
ObsSet	0.0 kHz
ObsFine	10004.56 Hz
Point	16384
Frequecy(Span)	3612.717 Hz
Scan	8
AcqTime	4.5351 s
PD	1.0 s
Pulse1	9.0 μs
IrrNuc	OFF
ProbeHead	5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument	SPECT
Pulse Program	ZG30
Gradient Program	
Temperature	26.85 ℃
Solvent	CDCL3
Reference	0.0 ppm
Broad.Factor	0.25 Hz
Window	Exponential
RGain	362
Operator	

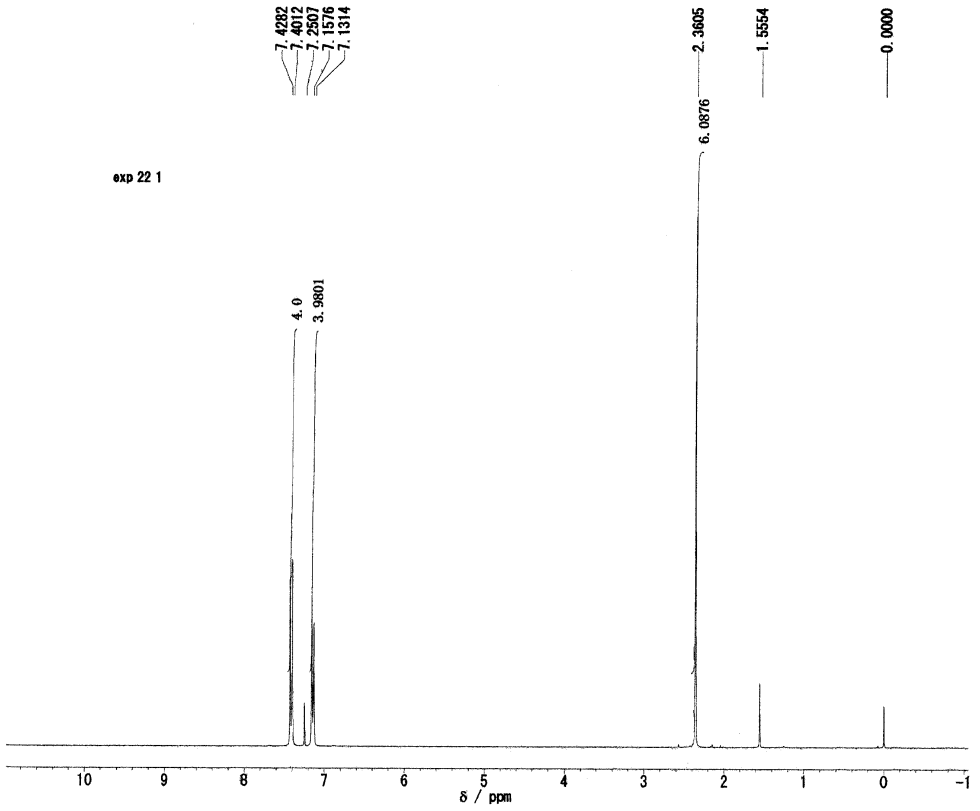


File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MINO2013¥18997 CY1¥PDATA¥1¥1R
Date 20/Nov/2013 21:40:04
Comment

ObsNuc	^{13}C
ExMode	ZGPG30
ObsFreq	75.49 MHz
ObsSet	0.0 kHz
ObsFine	10003.06 Hz
Point	32768
Frequecy(Span)	18115.94 Hz
Scan	256
AcqTime	1.8088 s
PD	2.0 s
Pulse1	9.0 μs
IrrNuc	OFF
ProbeHead	5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument	SPECT
Pulse Program	ZGPG30
Gradient Program	
Temperature	26.85 ℃
Solvent	CDCL3
Reference	77.0 ppm
Broad.Factor	0.25 Hz
Window	Exponential
RGain	2896
Operator	

^{1}H and ^{13}C NMR of 1,2-Di-*p*-tolylethyne (4c)





File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MINO2013¥18674 H¥1¥PDATA¥1¥1R
Date 28/Jun/2013 17:50:20
Comment

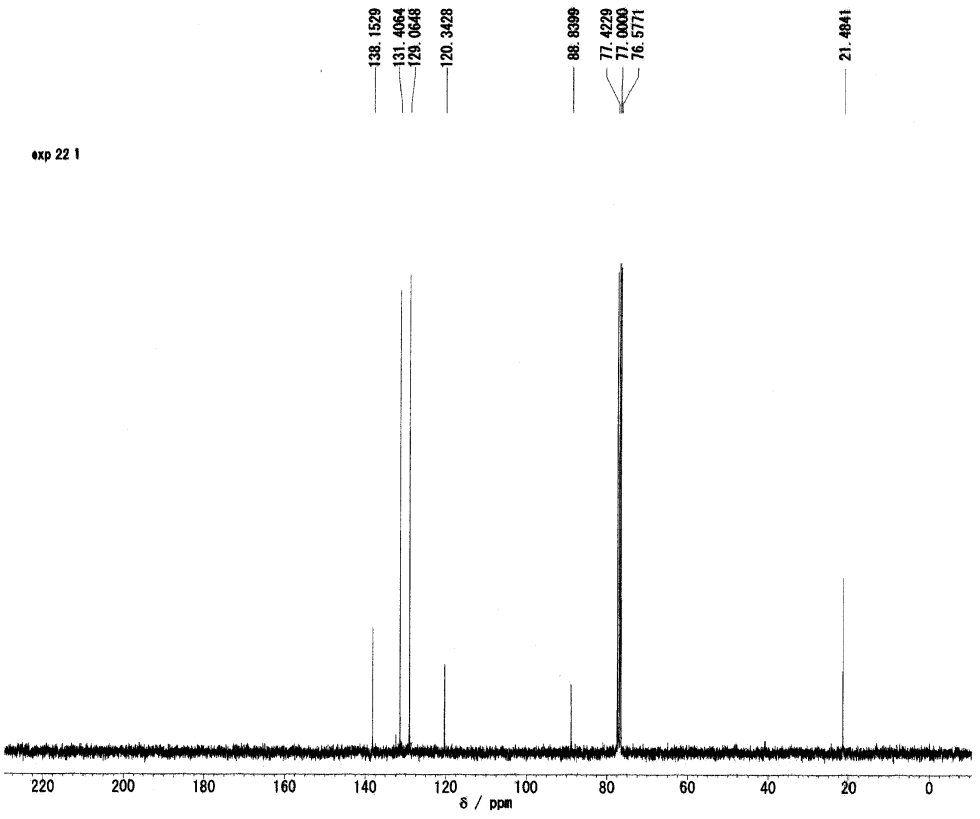
ObsNuc ^{1}H
ExMode ZG30
ObsFreq 300.23 MHz

ObsSet 0.0 kHz
ObsFine 10004.56 Hz
Point 16384
Frequecy(Span) 3612.717 Hz

Scan 8
AcqTime 4.5351 s

PD 1.0 s
Pulsel 9.0 μs
IrrNuc OFF
ProbeHeaD 5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument SPECT
Pulse Program ZG30
Gradient Program
Temperature 26.85 ℃
Solvent CDCL3
Reference 0.0 ppm
Broad.Factor 0.25 Hz
Window Exponential
RGain 362

Operator



File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MINO2013¥18674 C¥1¥PDATA¥1¥1R
Date 28/Jun/2013 18:08:01
Comment

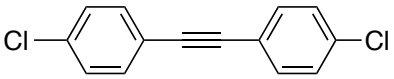
ObsNuc ^{13}C
ExMode ZGPG30
ObsFreq 75.49 MHz

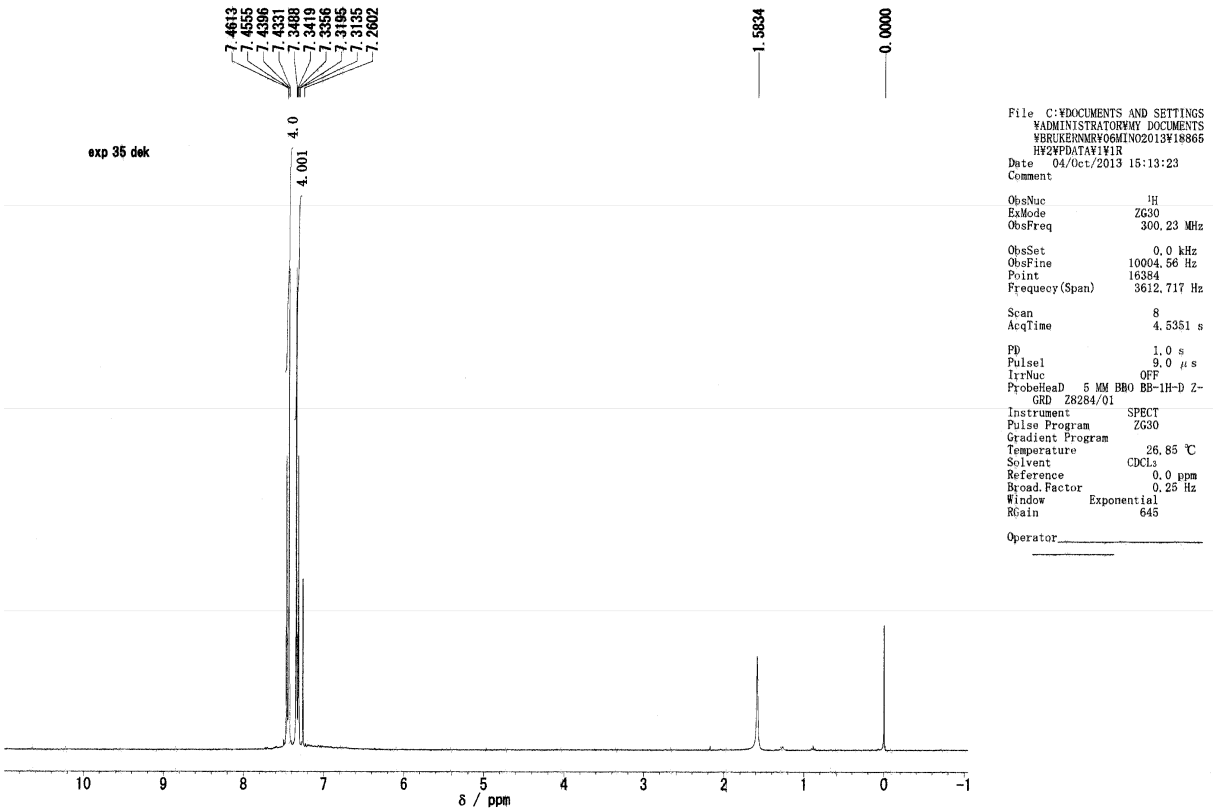
ObsSet 0.0 kHz
ObsFine 10003.06 Hz
Point 32768
Frequecy(Span) 18115.94 Hz
Scan 256
AcqTime 1.8088 s

PD 2.0 s
Pulsel 0.0 μs
IrrNuc OFF
ProbeHeaD 5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument SPECT
Pulse Program ZGPG30
Gradient Program
Temperature 26.85 ℃
Solvent CDCL3
Reference 77.0 ppm
Broad.Factor 0.25 Hz
Window Exponential
RGain 1149

Operator

^{1}H and ^{13}C NMR of 1,2-Bis(4-chlorophenyl)ethyne (4d)

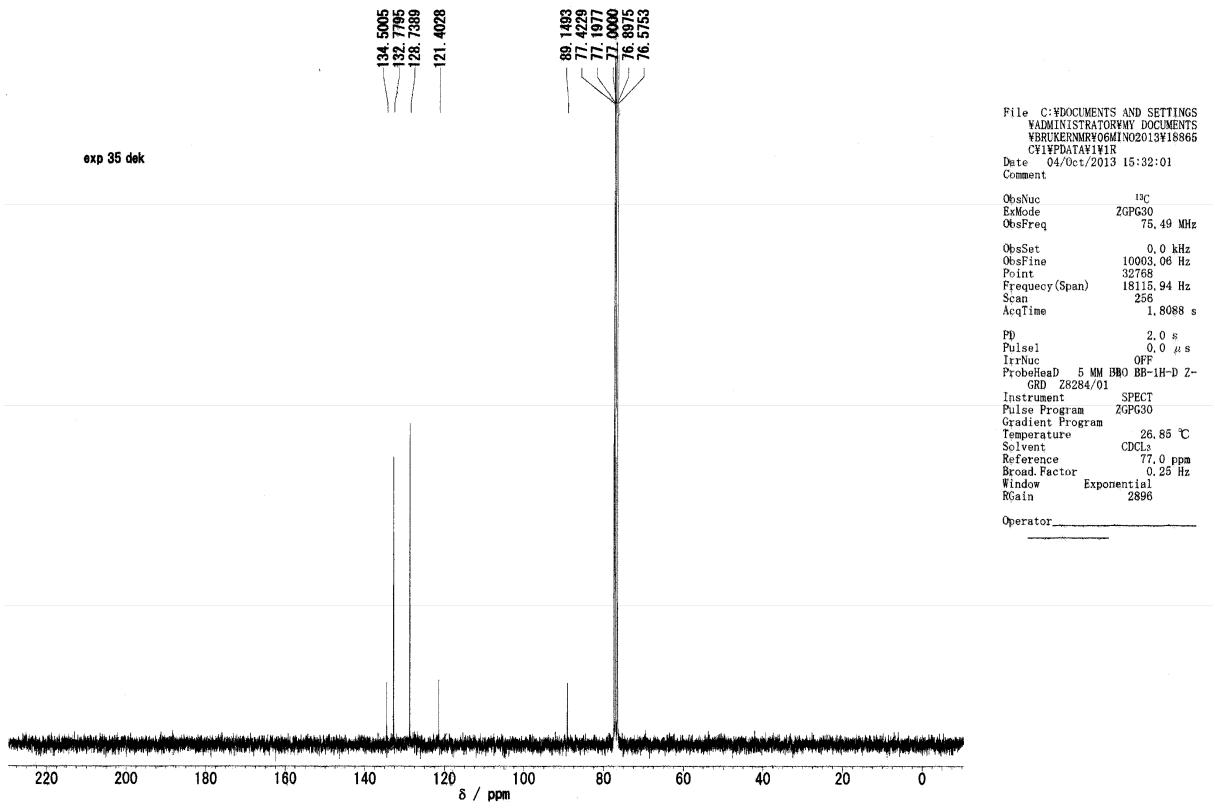




File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MIN02013¥18865 H¥2¥PDATA¥1¥1R
Date 04/Oct/2013 15:13:23
Comment

ObsNuc	^{1}H
ExMode	ZG30
ObsFreq	300.23 MHz
ObsSet	0.0 kHz
ObsFine	10004.56 Hz
Point	16384
Frequecy(Span)	3612.717 Hz
Scan	8
AcqTime	4.5351 s
PD	1.0 s
Pulse1	9.0 μs
IrrNuc	OFF
ProbeHead	5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument	SPECT
Pulse Program	ZG30
Gradient Program	
Temperature	26.85 ℃
Solvent	CDCl3
Reference	0.0 ppm
Broad.Factor	0.25 Hz
Window	Exponential
RGain	645

Operator

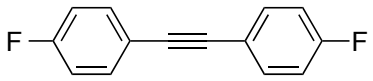


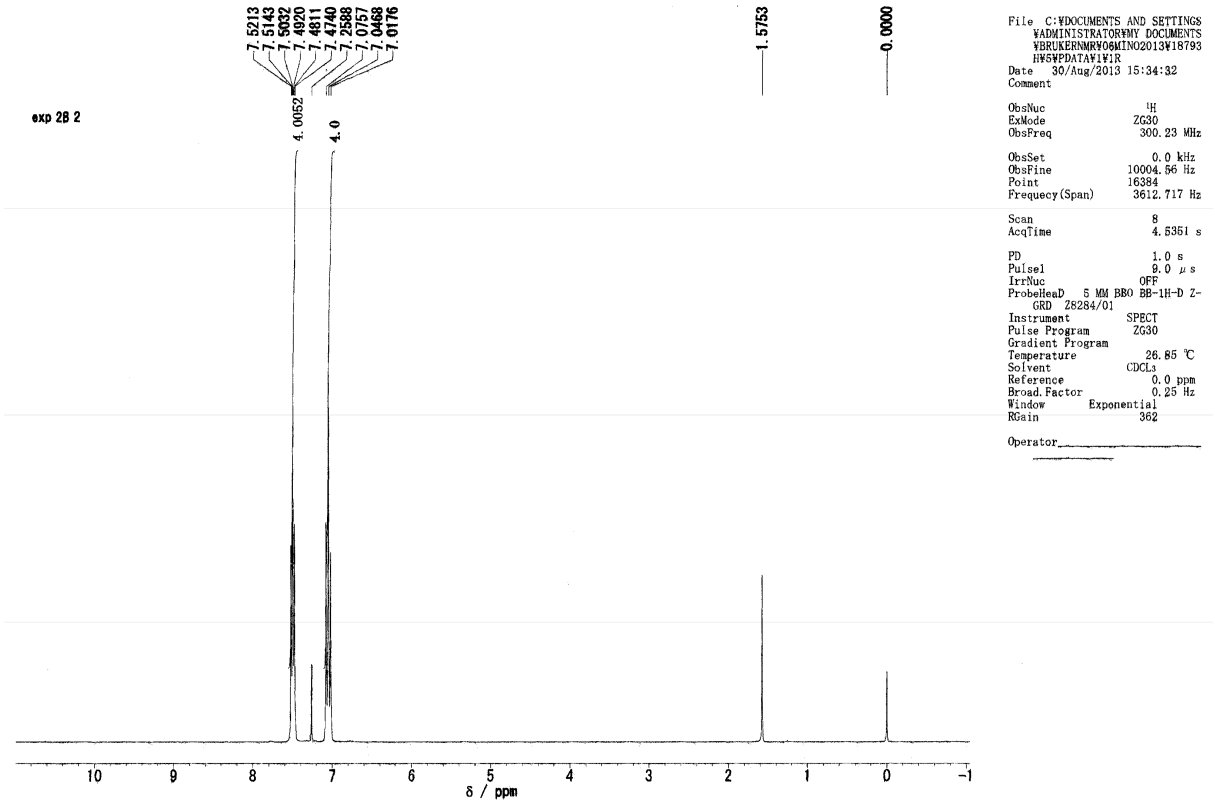
File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MIN02013¥18865 C¥1¥PDATA¥1¥1R
Date 04/Oct/2013 15:32:01
Comment

ObsNuc	^{13}C
ExMode	ZGPG30
ObsFreq	75.49 MHz
ObsSet	0.0 kHz
ObsFine	10003.06 Hz
Point	32768
Frequecy(Span)	18115.94 Hz
Scan	256
AcqTime	1.8088 s
PD	2.0 s
Pulse1	0.0 μs
IrrNuc	OFF
ProbeHead	5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument	SPECT
Pulse Program	ZGPG30
Gradient Program	
Temperature	26.85 ℃
Solvent	CDCl3
Reference	77.0 ppm
Broad.Factor	0.25 Hz
Window	Exponential
RGain	2896

Operator

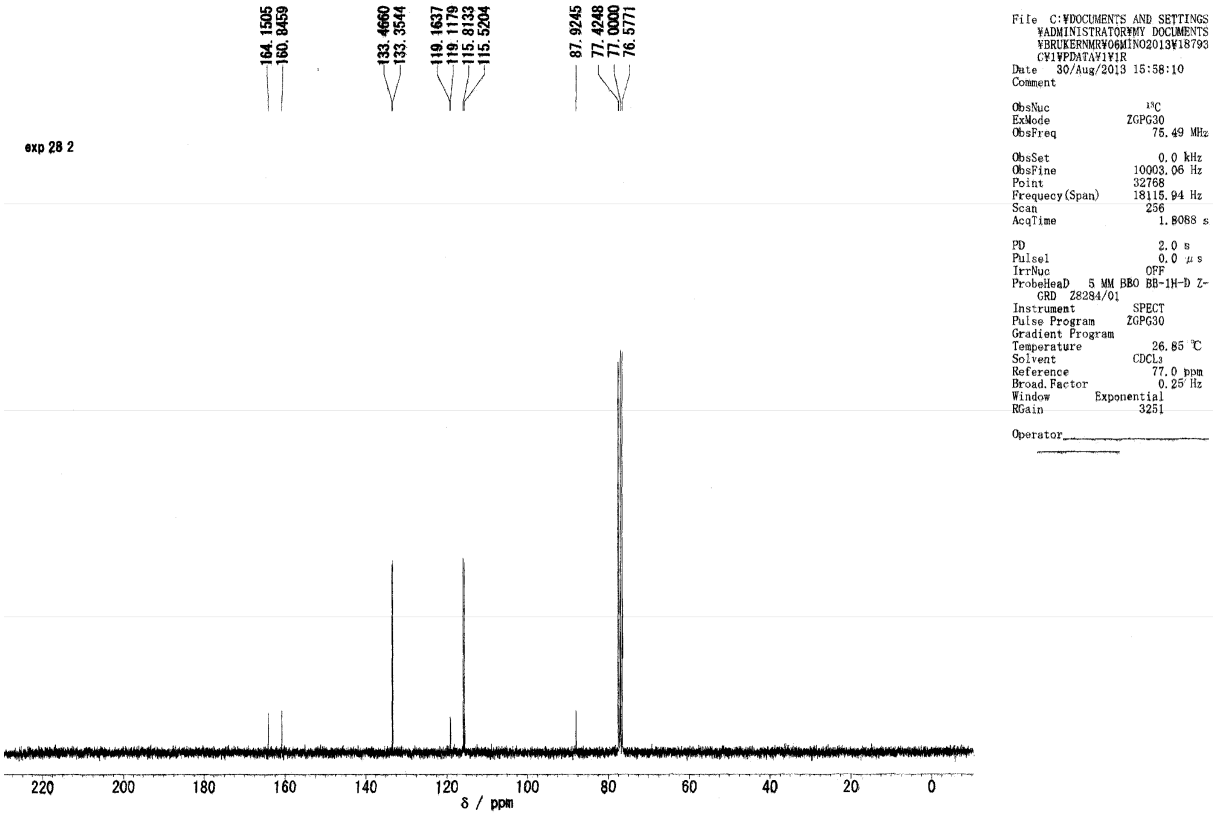
^{1}H and ^{13}C NMR of 1,2-Bis(4-fluorophenyl)ethyne (4e)





File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MINO2013¥18793 H¥5¥PDATA¥1¥1R
Date 30/Aug/2013 15:34:32
Comment

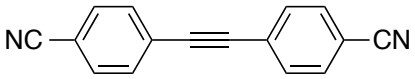
ObsNuc	^{1}H
ExMode	ZG30
ObsFreq	300.23 MHz
ObsSet	0.0 kHz
ObsFine	10004.56 Hz
Point	16384
Frequecy(Span)	3612.717 Hz
Scan	8
AcqTime	4.5351 s
PD	1.0 s
Pulsel	9.0 μs
IrrNuc	OFF
ProbeHeaD	5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument	SPECT
Pulse Program	ZG30
Gradient Program	
Temperature	26.85 ℃
Solvent	CDCL3
Reference	0.0 ppm
Broad.Factor	0.25 Hz
Window	Exponential
RGain	362
Operator	

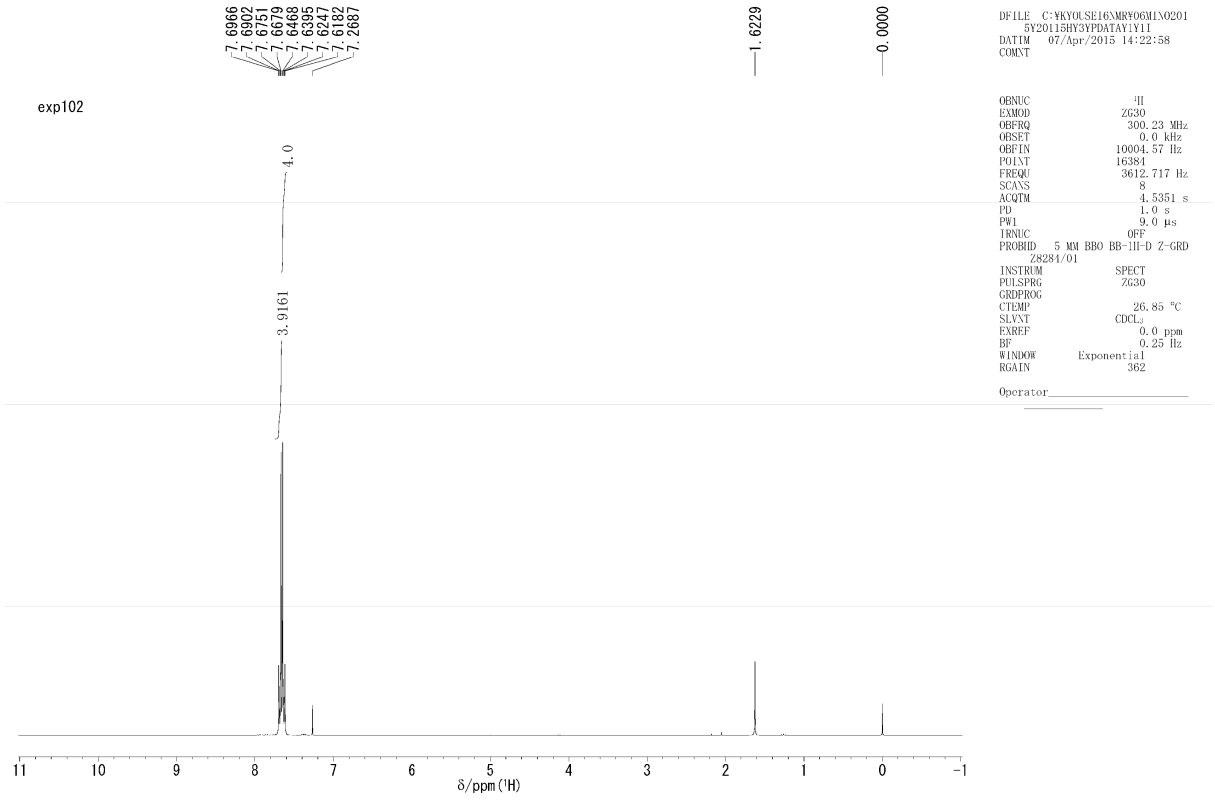


File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MINO2013¥18793 C¥1¥PDATA¥1¥1R
Date 30/Aug/2013 15:58:10
Comment

ObsNuc	^{13}C
ExMode	ZGPG30
ObsFreq	75.49 MHz
ObsSet	0.0 kHz
ObsFine	10003.06 Hz
Point	32768
Frequecy(Span)	18115.94 Hz
Scan	256
AcqTime	1.8088 s
PD	2.0 s
Pulsel	0.0 μs
IrrNuc	OFF
ProbeHeaD	5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument	SPECT
Pulse Program	ZGPG30
Gradient Program	
Temperature	26.85 ℃
Solvent	CDCL3
Reference	77.0 ppm
Broad.Factor	0.25 Hz
Window	Exponential
RGain	3251
Operator	

^{1}H and ^{13}C NMR of 1,2-Bis(4-cyanophenyl)ethyne (4f)

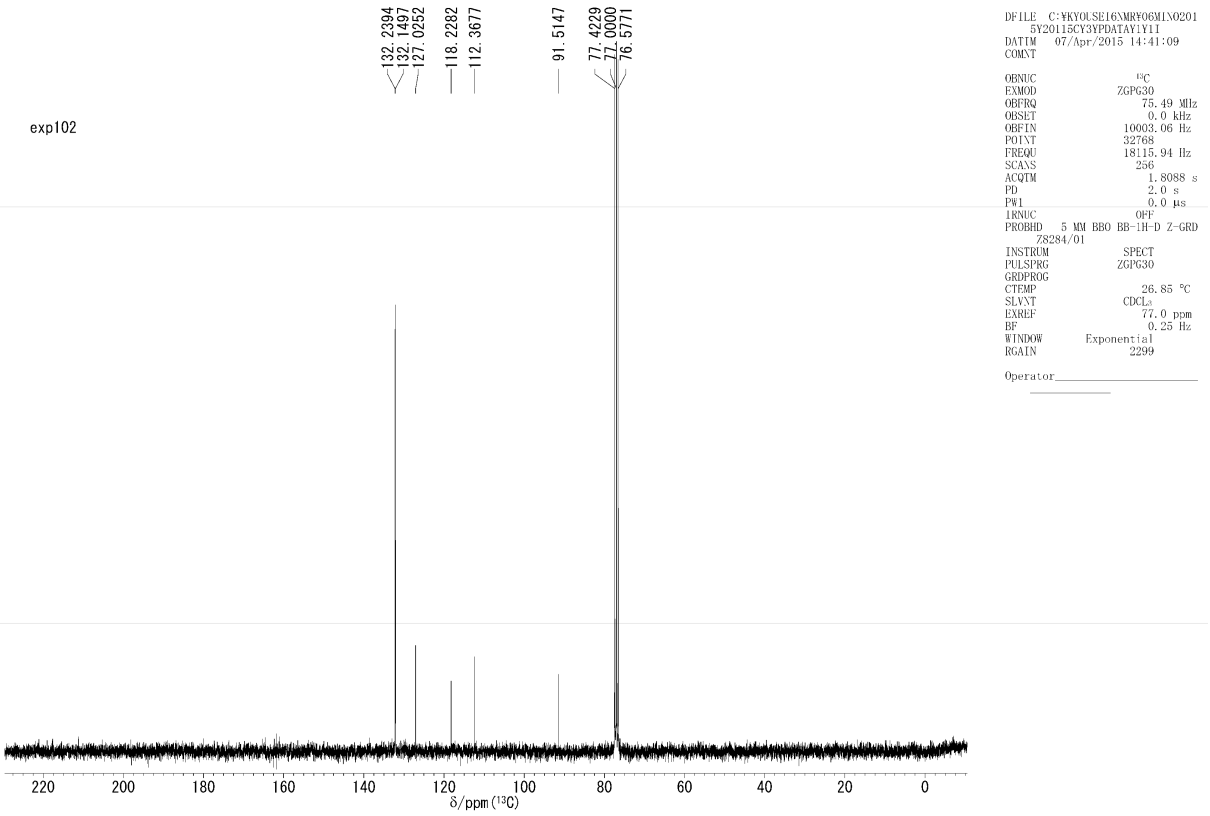




DFILE C:¥KYOUSEI6XMR¥06MINO201
5Y20115HY3YPDATAY1Y1I
DATIM 07/Apr/2015 14:22:58
COMNT

OBNUC	¹H
EXMOD	ZG30
OBFRQ	300.23 MHz
OBSET	0.0 kHz
OBFIN	10004.57 Hz
POINT	16384
FREQU	3612.717 Hz
SCANS	8
ACQTM	4.5351 s
PD	1.0 s
PW1	9.0 µs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCL_3$
EXREF	0.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	362

Operator

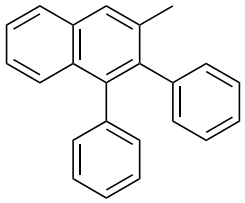


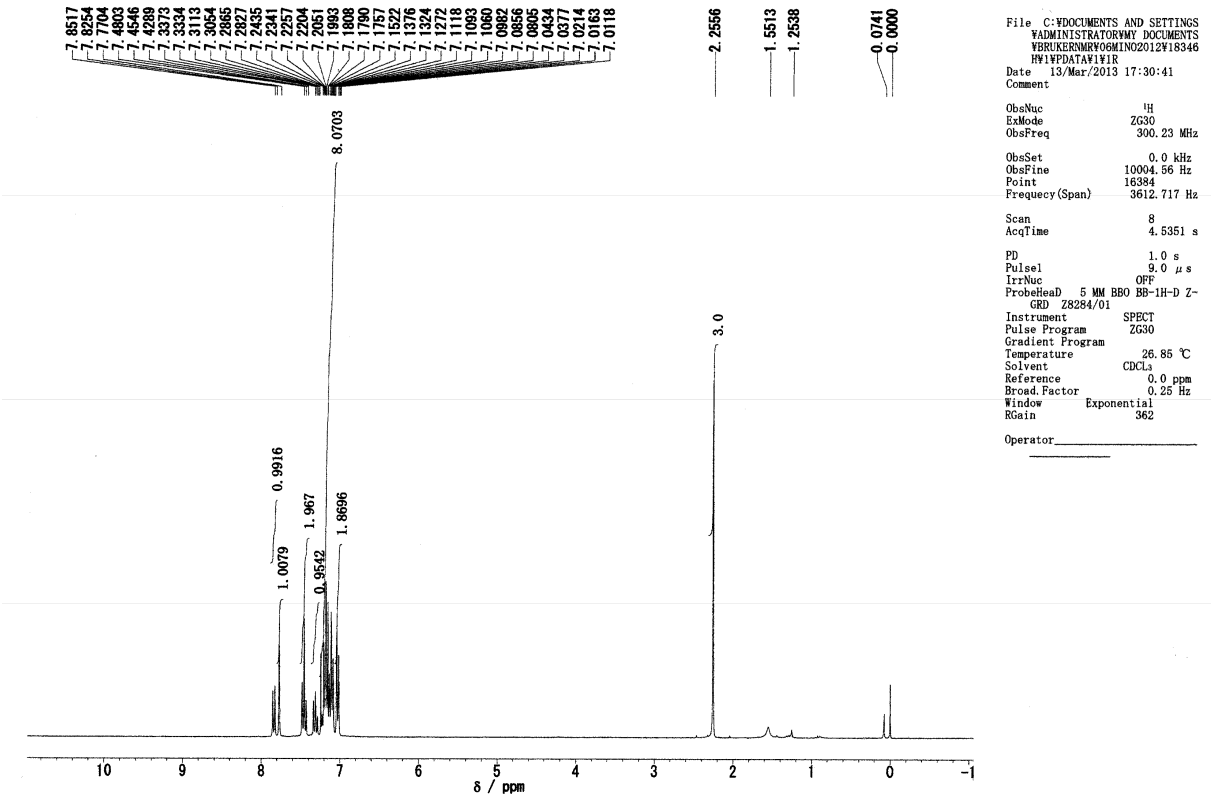
DFILE C:¥KYOUSEI6XMR¥06MINO201
5Y20115CY3YPDATAY1Y1I
DATIM 07/Apr/2015 14:41:09
COMNT

OBNUC	¹³C
EXMOD	ZGPG30
OBFRQ	75.49 MHz
OBSET	0.0 kHz
OBFIN	10003.06 Hz
POINT	32768
FREQU	18115.94 Hz
SCANS	256
ACQTM	1.8088 s
PD	2.0 s
PW1	0.0 µs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZGPG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCL_3$
EXREF	77.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	2299

Operator

^{1}H and ^{13}C NMR of 3-Methyl-1,2-diphenylnaphthalene (5aa)





File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MINO2012¥18346 H¥1¥PDATA¥1¥1R
Date 13/Mar/2013 17:30:41
Comment

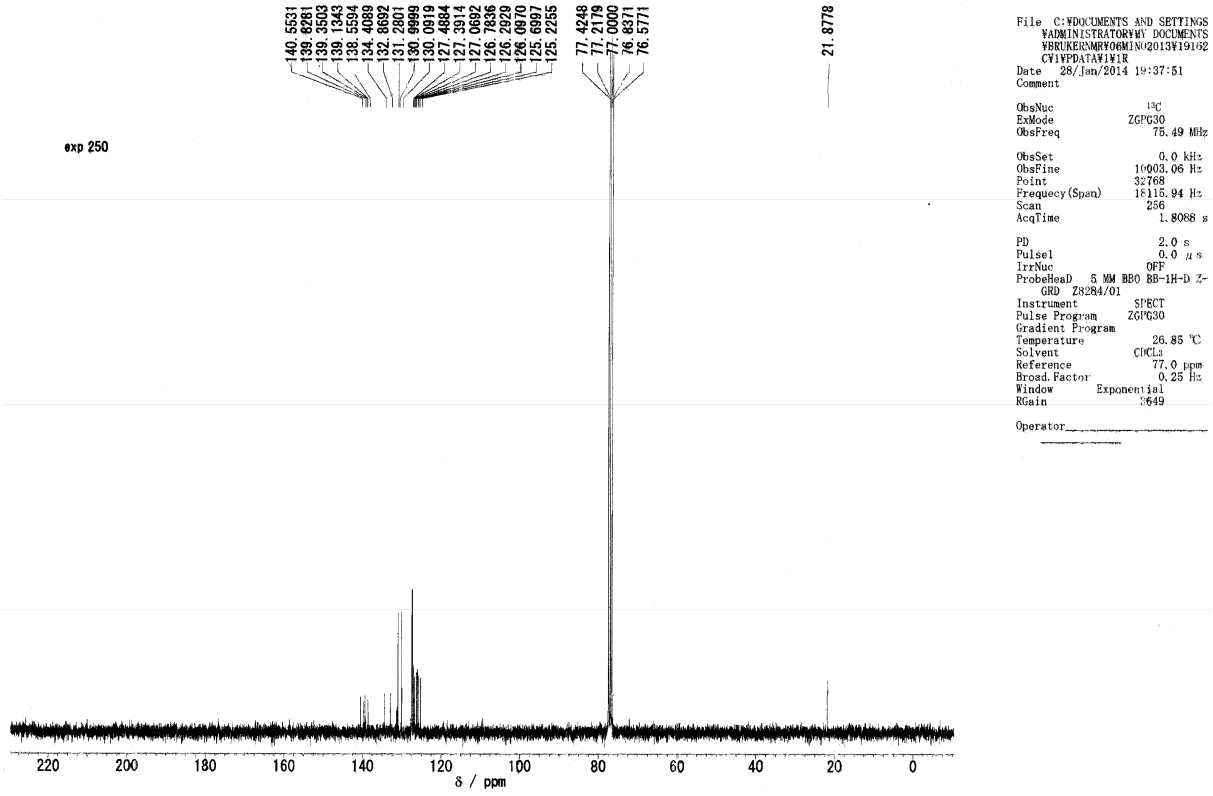
ObsNuc ^{1}H
ExMode ZG30
ObsFreq 300.23 MHz

ObsSet 0.0 kHz
ObsFine 10094.56 Hz
Point 16384
Frequecy(Span) 3612.717 Hz

Scan 8
AcqTime 4.5351 s

PD 1.0 s
Pulsel 9.0 μs
IrrNuc OFF
ProbeHead 5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument SPECT
Pulse Program ZG30
Gradient Program
Temperature 26.85 ℃
Solvent CDCL3
Reference 0.0 ppm
Broad.Factor 0.25 Hz
Window Exponential
RGain 362

Operator



File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MINO2013¥19162 CY1¥PDATA¥1¥1R
Date 28/Jan/2014 19:37:51
Comment

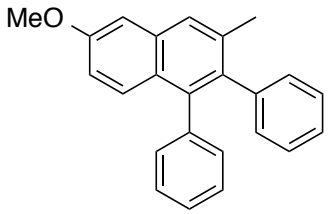
ObsNuc ^{13}C
ExMode ZGPG30
ObsFreq 75.49 MHz

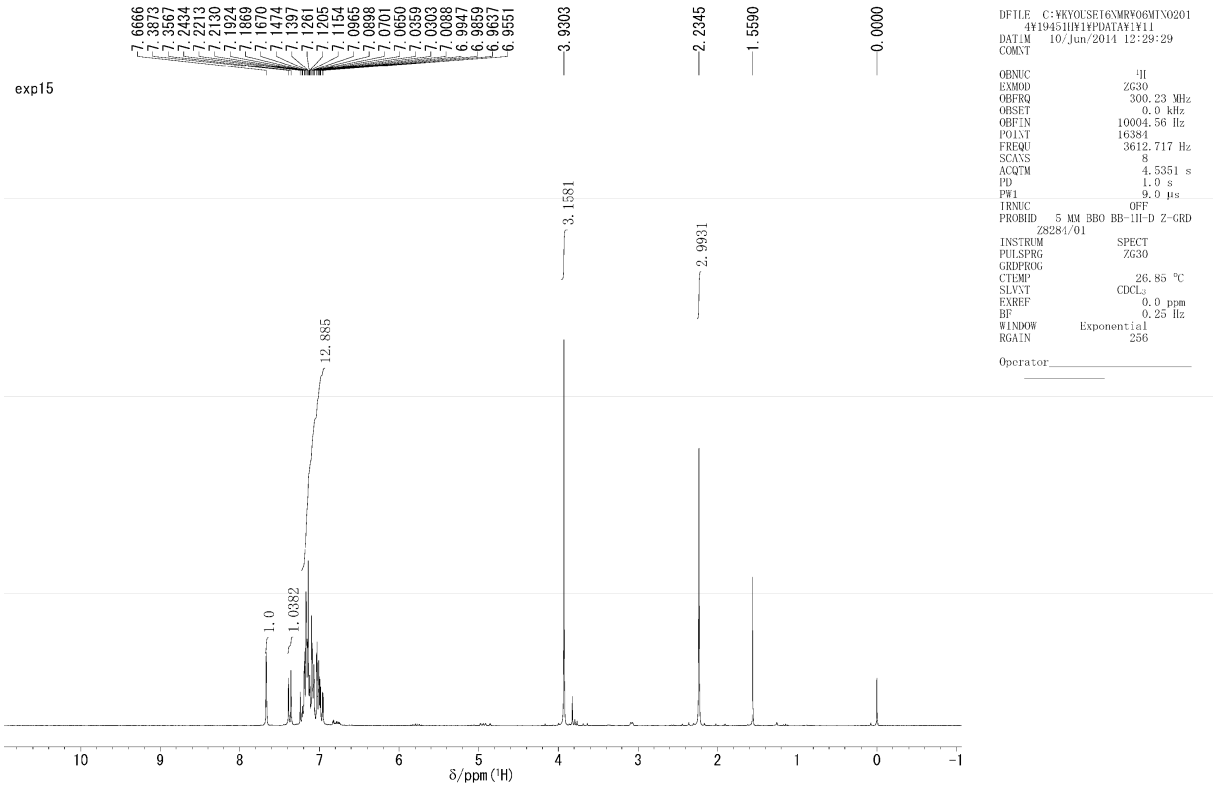
ObsSet 0.0 kHz
ObsFine 10003.06 Hz
Point 32768
Frequecy(Span) 18115.94 Hz
Scan 256
AcqTime 1.8088 s

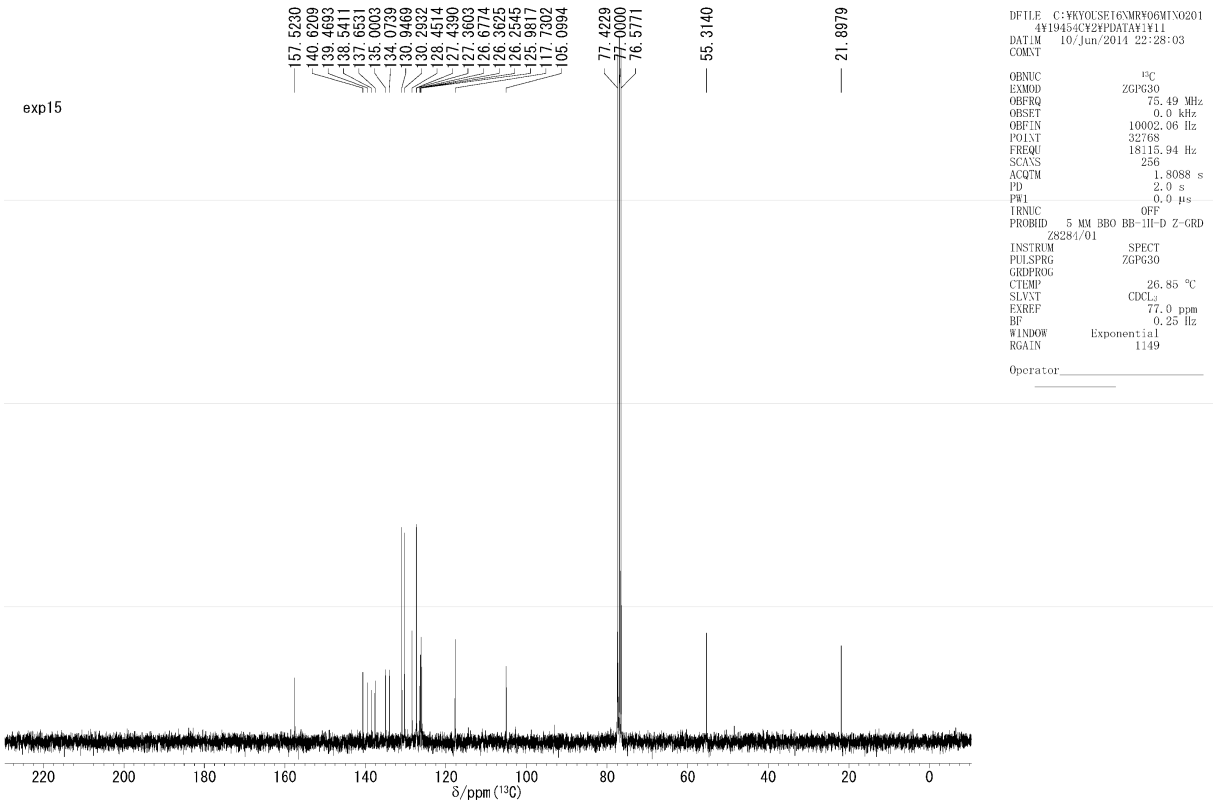
PD 2.0 s
Pulsel 0.0 μs
IrrNuc OFF
ProbeHead 5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument SPECT
Pulse Program ZGPG30
Gradient Program
Temperature 26.85 ℃
Solvent CDCL3
Reference 77.0 ppm
Broad.Factor 0.25 Hz
Window Exponential
RGain 3649

Operator

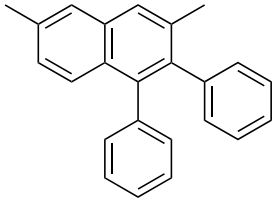
^{1}H and ^{13}C NMR of 6-Methoxy-3-methyl-1,2-diphenylnaphthalene (5ba)

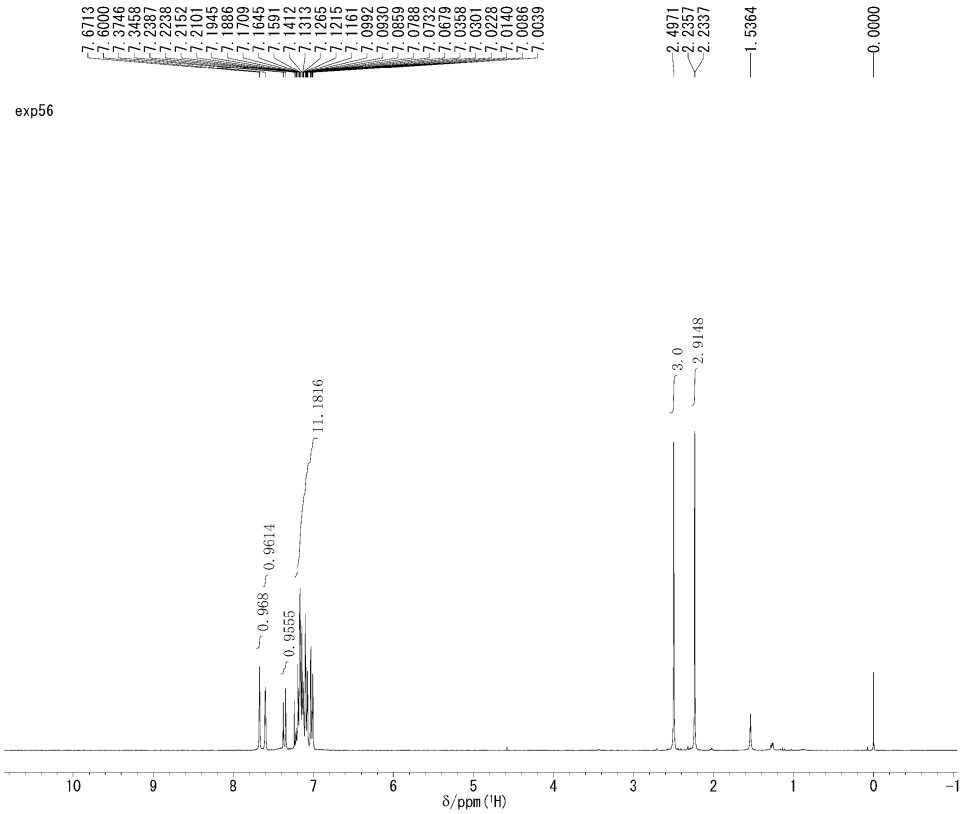




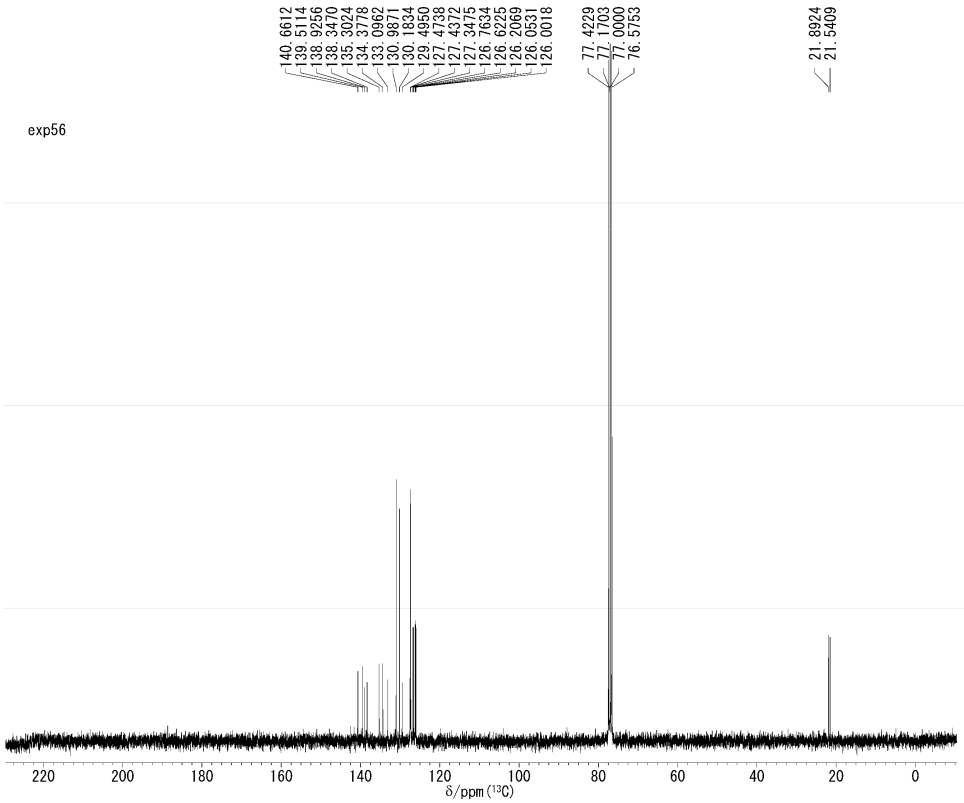


^{1}H and ^{13}C NMR of 3,6-Dimethyl-1,2-diphenylnaphthalene (5ca)



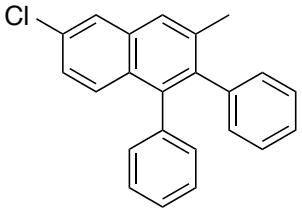


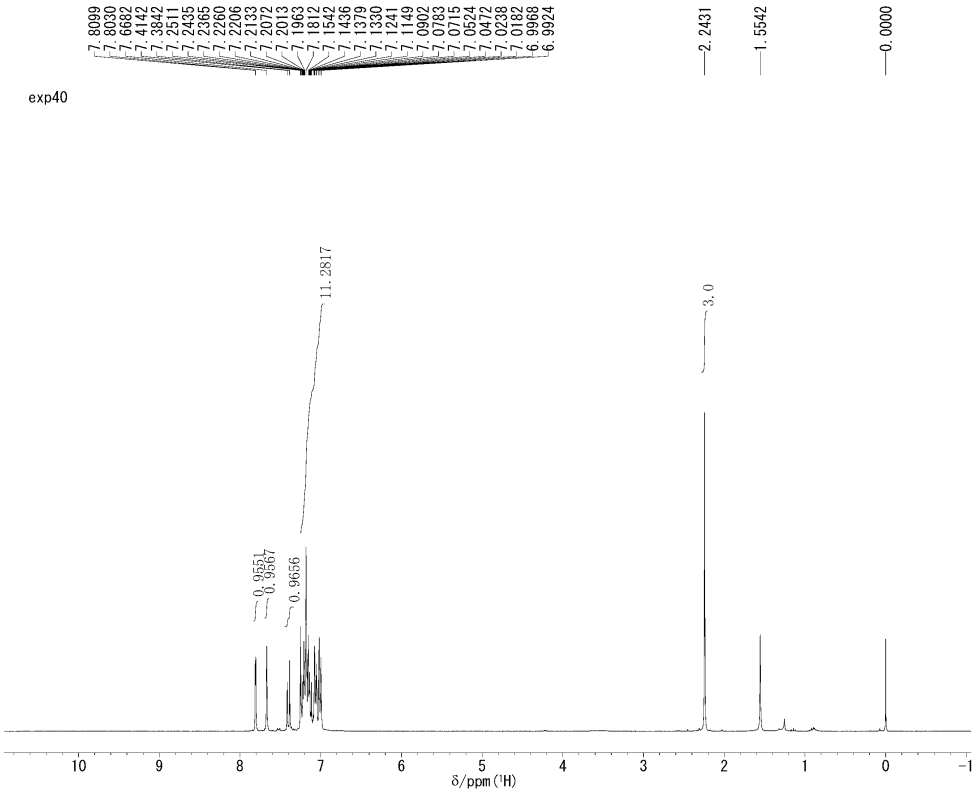

```
DFILE  C:¥KYOUSEI6¥MR¥06MTN0201
    4¥19771H¥2¥PDATA¥1¥1i
DATIM   06/Nov/2014 17:17:14
COMNT

OBNUC                  ¹H
EXMOD                ZG30
OBFRQ              300.23 MHz
OBSET                0.0 kHz
OBFIN            10004.57 Hz
PO1NT               16384
FREQU             3612.717 Hz
SCANS                   8
ACQTM               4.5351 s
PD                    1.0 s
PW1                   9.0 µs
IRNUC                 OFF
PROBHD   5 MM BBO BB-1H-D Z-GRD
      Z8284/01
INSTRUM              SPECT
PULSPRG              ZG30
GRDPROG
CTEMP              26.85 °C
SLVNT              CDCL₃
EXREF                0.0 ppm
BF                   0.25 Hz
WINDOW       Exponential
RGAIN                362

Operator_____________________
```




```
DFILE  C:¥KYOUSEI6¥MR¥06MTN0201
    4¥19771C¥2¥PDATA¥1¥1i
DATIM   06/Nov/2014 17:37:04
COMNT

OBNUC                 ¹³C
EXMOD              ZGPG30
OBFRQ              75.49 MHz
OBSET                0.0 kHz
OBFIN            10003.06 Hz
PO1NT               32768
FREQU             18115.94 Hz
SCANS                 256
ACQTM               1.8088 s
PD                    2.0 s
PW1                   0.0 µs
IRNUC                 OFF
PROBHD   5 MM BBO BB-1H-D Z-GRD
      Z8284/01
INSTRUM              SPECT
PULSPRG            ZGPG30
GRDPROG
CTEMP              26.85 °C
SLVNT              CDCL₃
EXREF               77.0 ppm
BF                   0.25 Hz
WINDOW       Exponential
RGAIN                2048

Operator_____________________
```

^{1}H and ^{13}C NMR of 6-Chloro-3-methyl-1,2-diphenylnaphthalene (5da)

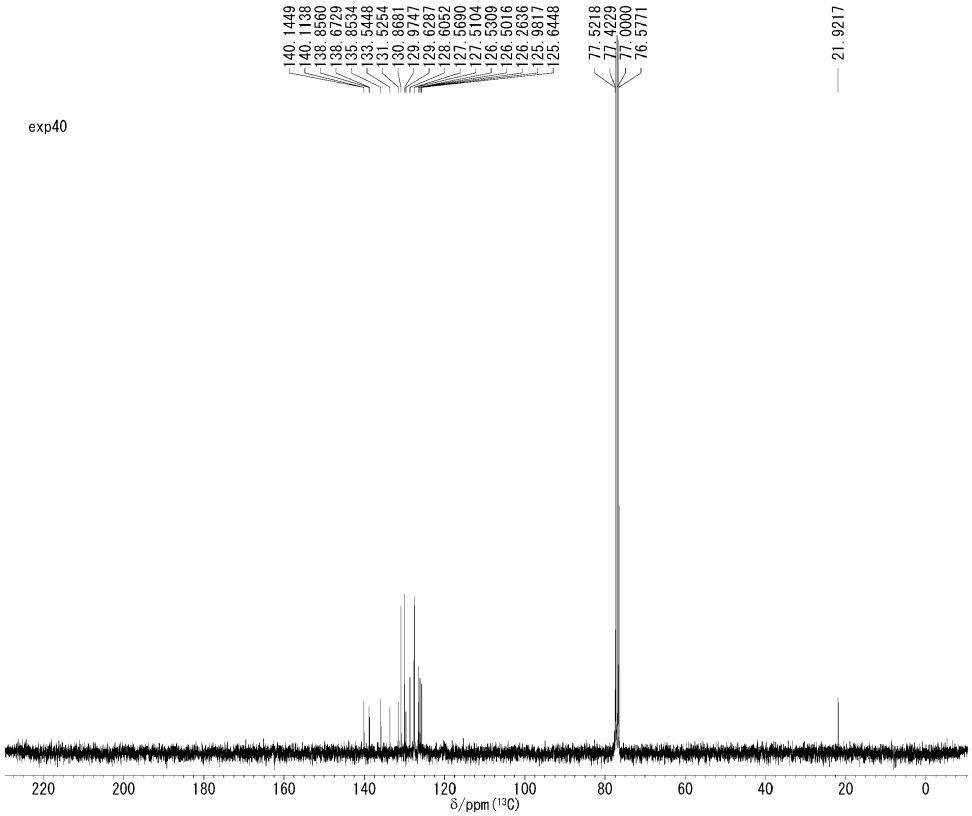




DFILE C:¥KYOUSEI6¥MR¥06MIX0201
4¥19597H¥1¥PDATA¥1¥1I
DATIM 25/Aug/2014 12:42:27
COMNT

OBNUC	^{1}H
EXMOD	ZG30
OBFRQ	300.23 MHz
OBSET	0.0 kHz
OBFIN	10004.56 Hz
PO1NT	16384
FREQU	3612.717 Hz
SCANS	8
ACQTM	4.5351 s
PD	1.0 s
PW1	9.0 µs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCl_3$
EXREF	0.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	512

Operator

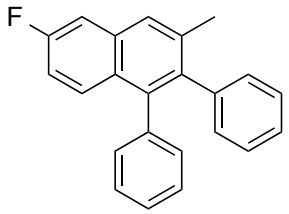


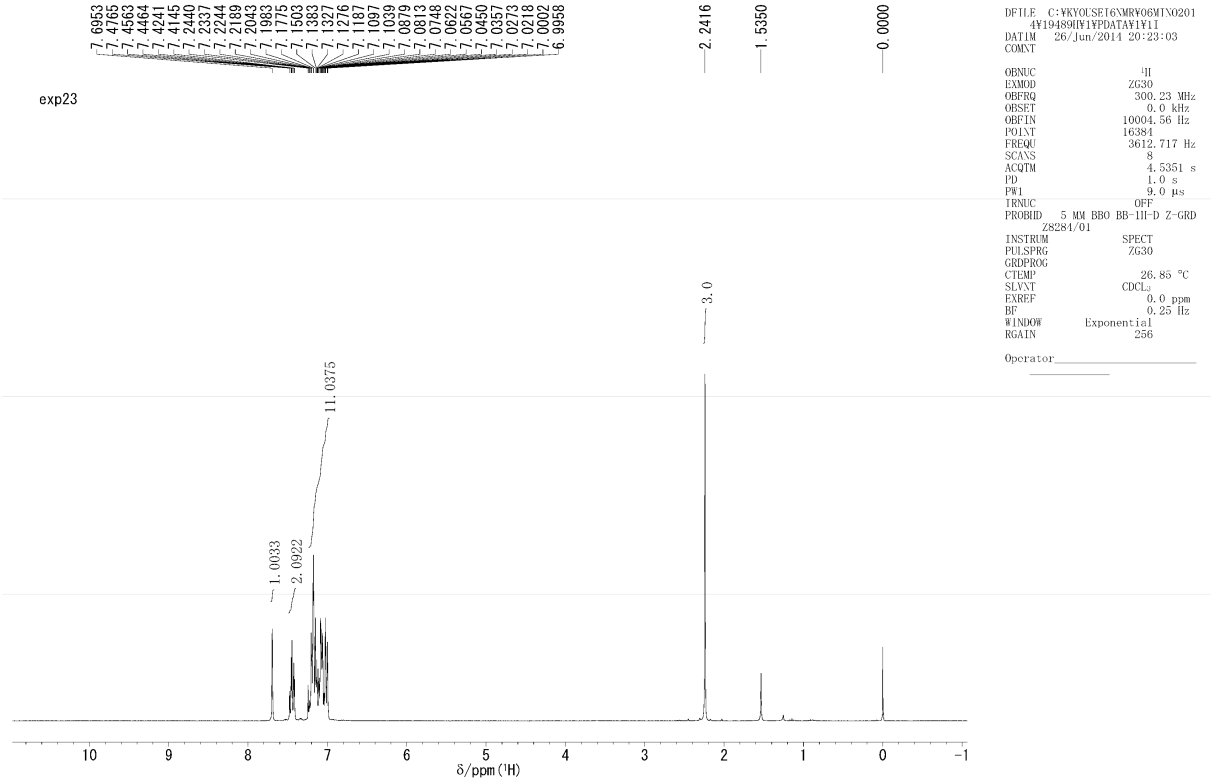
DFILE C:¥KYOUSEI6¥MR¥06MIX0201
4¥19597C¥1¥PDATA¥1¥1I
DATIM 25/Aug/2014 13:00:42
COMNT

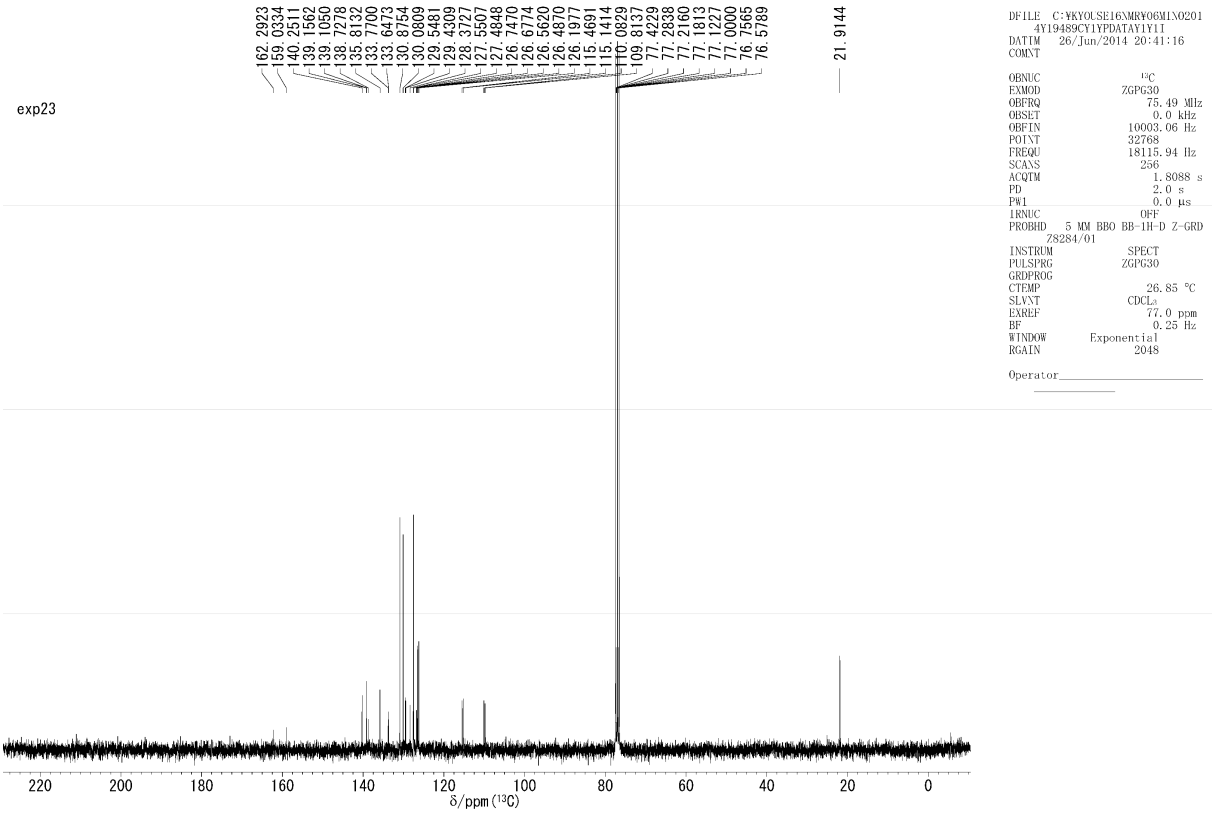
OBNUC	^{13}C
EXMOD	ZGPG30
OBFRQ	75.49 MHz
OBSET	0.0 kHz
OBFIN	10003.06 Hz
PO1NT	32768
FREQU	18115.94 Hz
SCANS	256
ACQTM	1.8088 s
PD	2.0 s
PW1	0.0 µs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZGPG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCl_3$
EXREF	77.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	2048

Operator

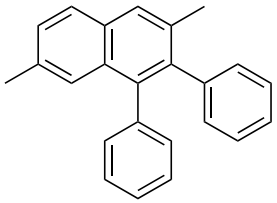
^{1}H and ^{13}C NMR of 6-Fluoro-3-methyl-1,2-diphenylnaphthalene (5ea)

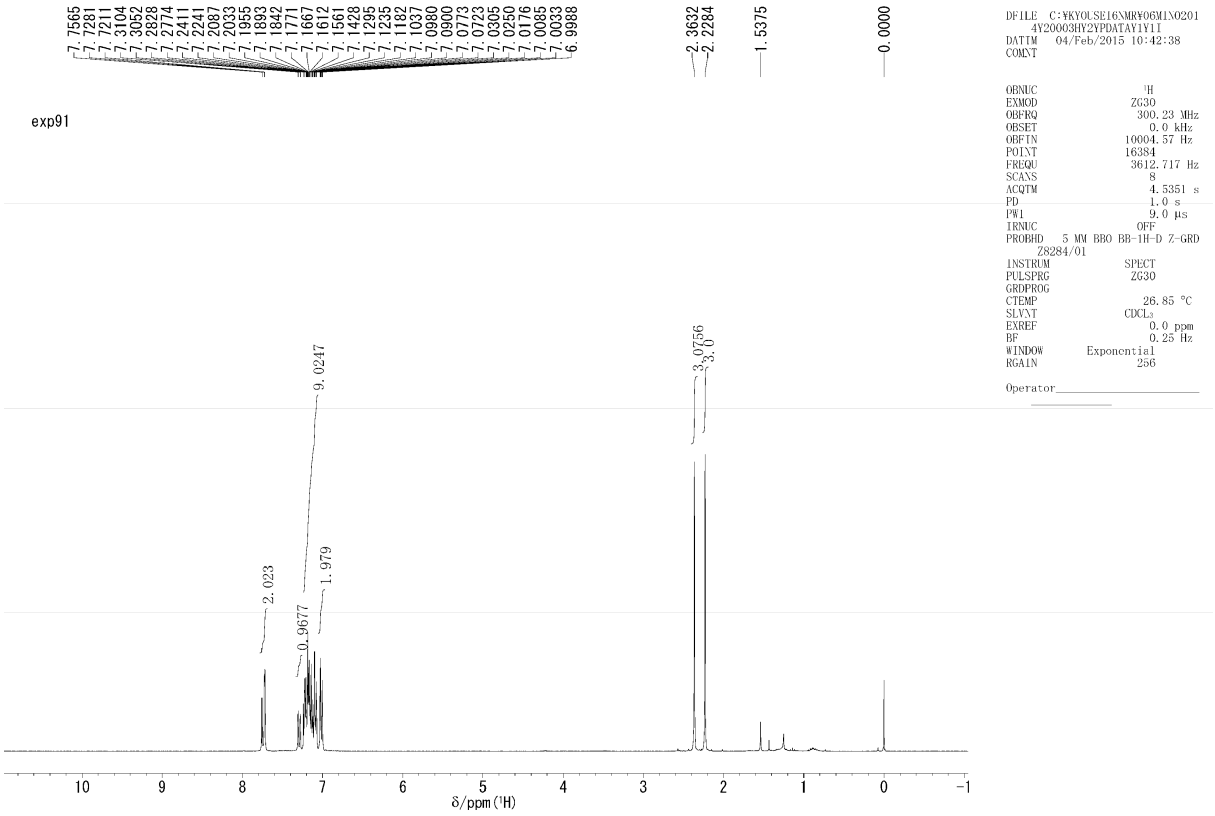




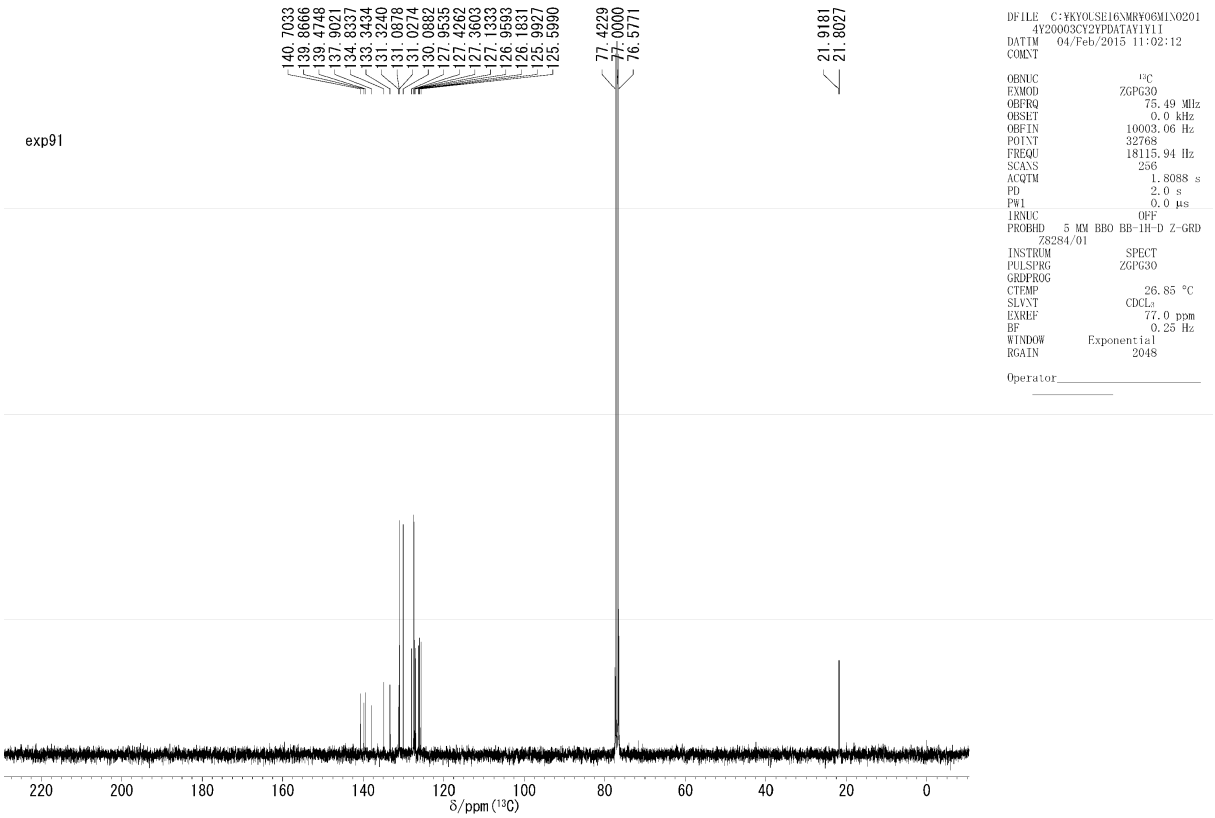


^{1}H and ^{13}C NMR of 3,7-Dimethyl-1,2-diphenylnaphthalene (5fa)



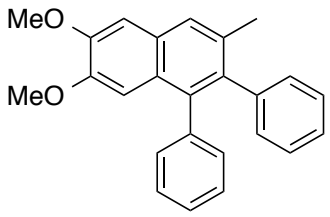


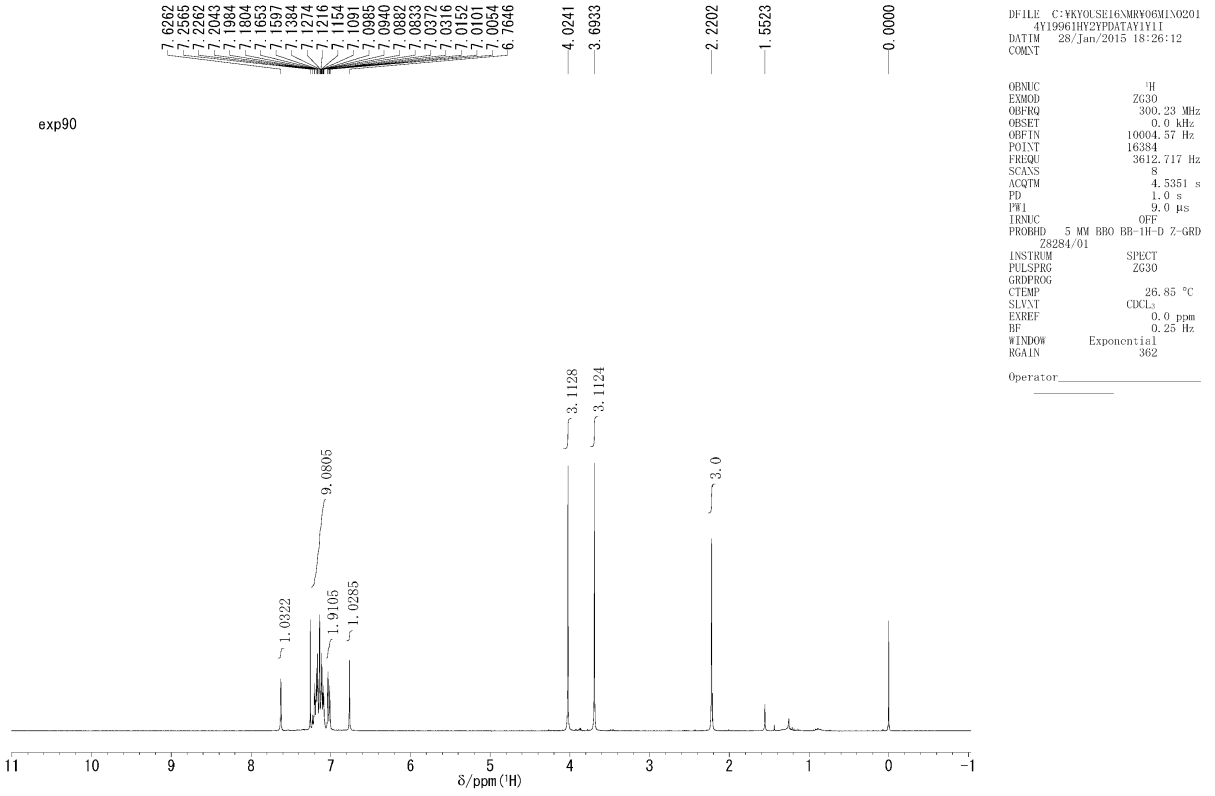

```
DFILE   C:¥KYOUSE16NMR¥O6M1NO201
    4Y20003HY2YPDATAY1Y1I
DATIM   04/Feb/2015 10:42:38
COMNT

OBNUC                   1H
EXMOD                ZG30
OBFRQ              300.23 MHz
OBSET                0.0 kHz
OBFIN            10004.57 Hz
POINT               16384
FREQU             3612.717 Hz
SCANS                   8
ACQTM               4.5351 s
PD                  1.0 s
PW1                 9.0 µs
IRNUC                OFF
PROBHD   5 MM BBO BB-1H-D Z-GRD
    Z8284/01
INSTRUM              SPECT
PULSPRG              ZG30
GRDPROG
CTEMP               26.85 °C
SLVNT               CDCL3
EXREF               0.0 ppm
BF                  0.25 Hz
WINDOW          Exponential
RGAIN                256

Operator
```




```
DFILE   C:¥KYOUSE16NMR¥O6M1NO201
    4Y20003CY2YPDATAY1Y1I
DATIM   04/Feb/2015 11:02:12
COMNT

OBNUC                  13C
EXMOD              ZGPG30
OBFRQ              75.49 MHz
OBSET                0.0 kHz
OBFIN            10003.06 Hz
POINT               32768
FREQU            18115.94 Hz
SCANS                 256
ACQTM               1.8088 s
PD                  2.0 s
PW1                 0.0 µs
IRNUC                OFF
PROBHD   5 MM BBO BB-1H-D Z-GRD
    Z8284/01
INSTRUM              SPECT
PULSPRG            ZGPG30
GRDPROG
CTEMP               26.85 °C
SLVNT               CDCL3
EXREF               77.0 ppm
BF                  0.25 Hz
WINDOW          Exponential
RGAIN               2048

Operator
```

^{1}H and ^{13}C NMR of 3-Methyl-6,7-dimethoxy-1,2-diphenylnaphthalene (5ga)

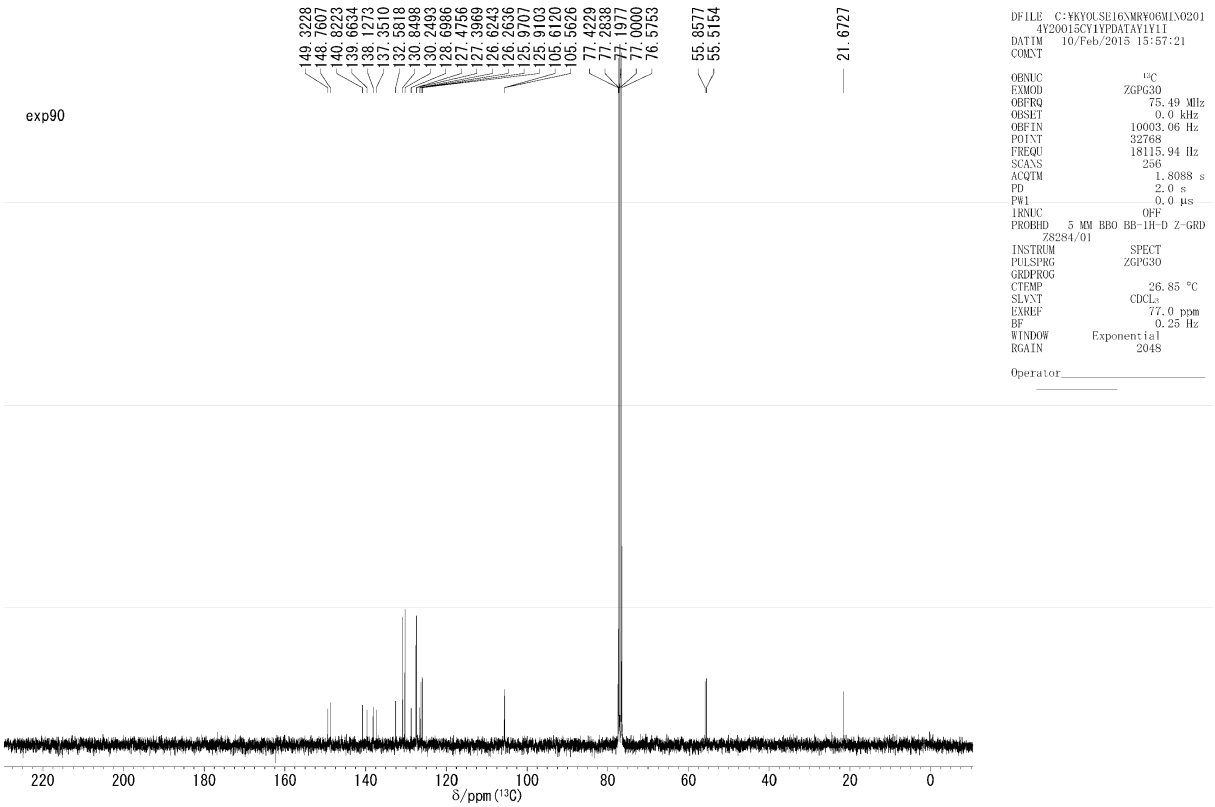




DF1LE C:¥KYOLSE16¥NMR¥06M1NO201
4Y19961HY2YPDATAY1Y1I
DATIM 28/Jan/2015 18:26:12
COMNT

OBNUC	^{1}H
EXMOD	ZG30
OBFRQ	300.23 MHz
OBSET	0.0 kHz
OBFIN	10004.57 Hz
POINT	16384
FREQU	3612.717 Hz
SCANS	8
ACQTM	4.5351 s
PD	1.0 s
PW1	9.0 µs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	CDCL₃
EXREF	0.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	362

Operator

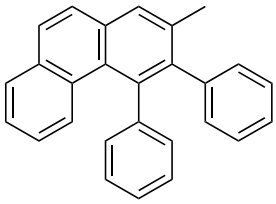


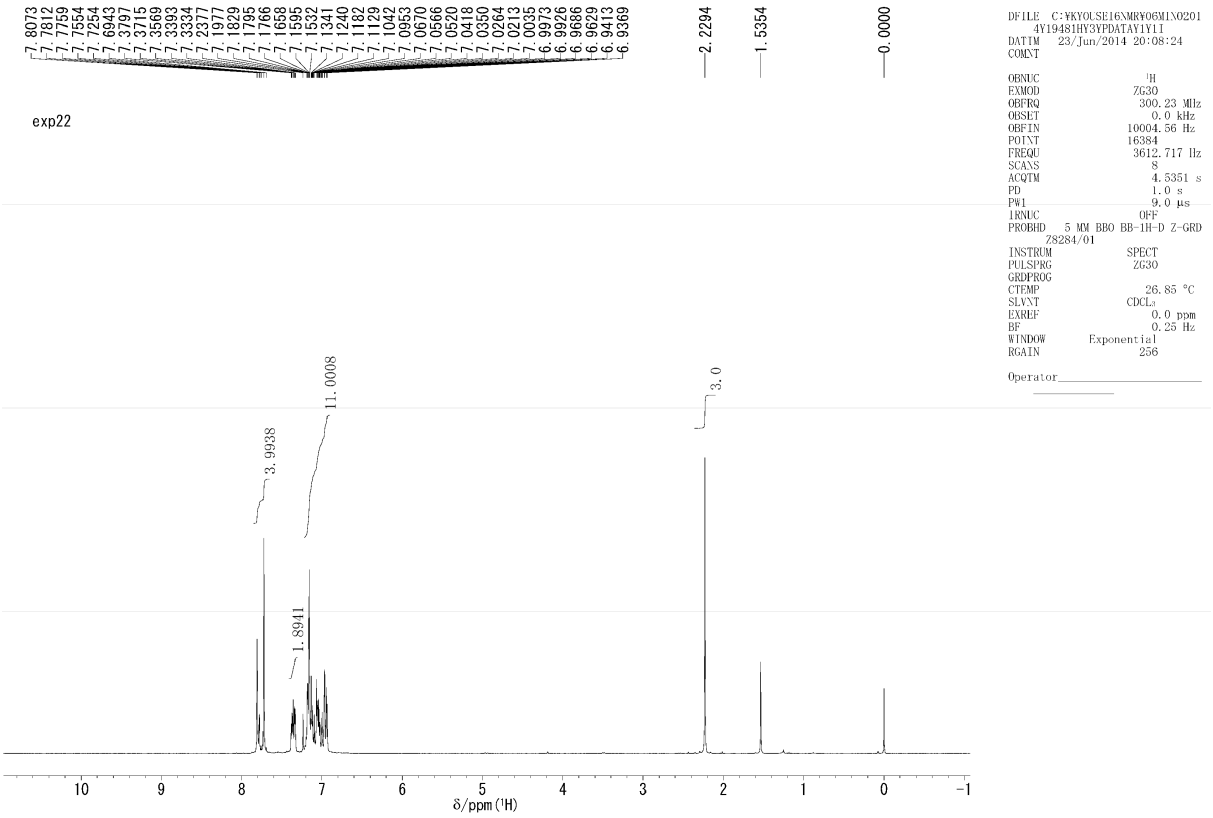
DF1LE C:¥KYOLSE16¥NMR¥06M1NO201
4Y20015CY1YPDATAY1Y1I
DATIM 10/Feb/2015 15:57:21
COMNT

OBNUC	^{13}C
EXMOD	ZGPG30
OBFRQ	75.49 MHz
OBSET	0.0 kHz
OBFIN	10003.06 Hz
POINT	32768
FREQU	18115.94 Hz
SCANS	256
ACQTM	1.8088 s
PD	2.0 s
PW1	0.0 µs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZGPG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	CDCL₃
EXREF	77.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	2048

Operator

^{1}H and ^{13}C NMR of 2-Methyl-3,4-diphenylphenanthlene (5ha)

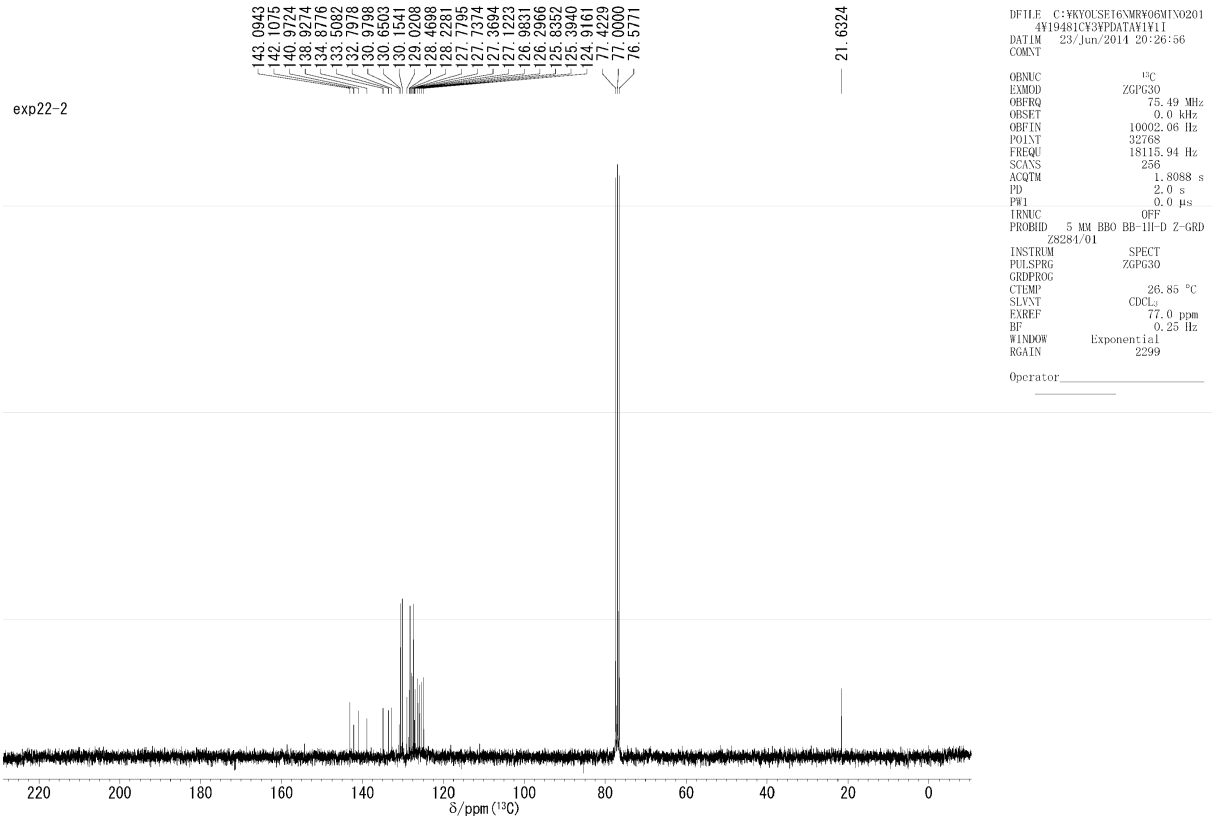




DFILE C:¥KYOUSEI6¥NMR¥06MIN0201
4Y19481HY3¥PDATAY1¥1I
DATIM 23/Jun/2014 20:08:24
COMNT

OBNUC	¹H
EXMOD	ZG30
OBFRQ	300.23 MHz
OBSET	0.0 kHz
OBFIN	10004.56 Hz
POINT	16384
FREQU	3612.717 Hz
SCANS	8
ACQTM	4.5351 s
PD	1.0 s
PW1	9.0 µs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCl_3$
EXREF	0.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	256

Operator_____

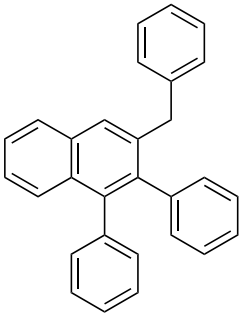


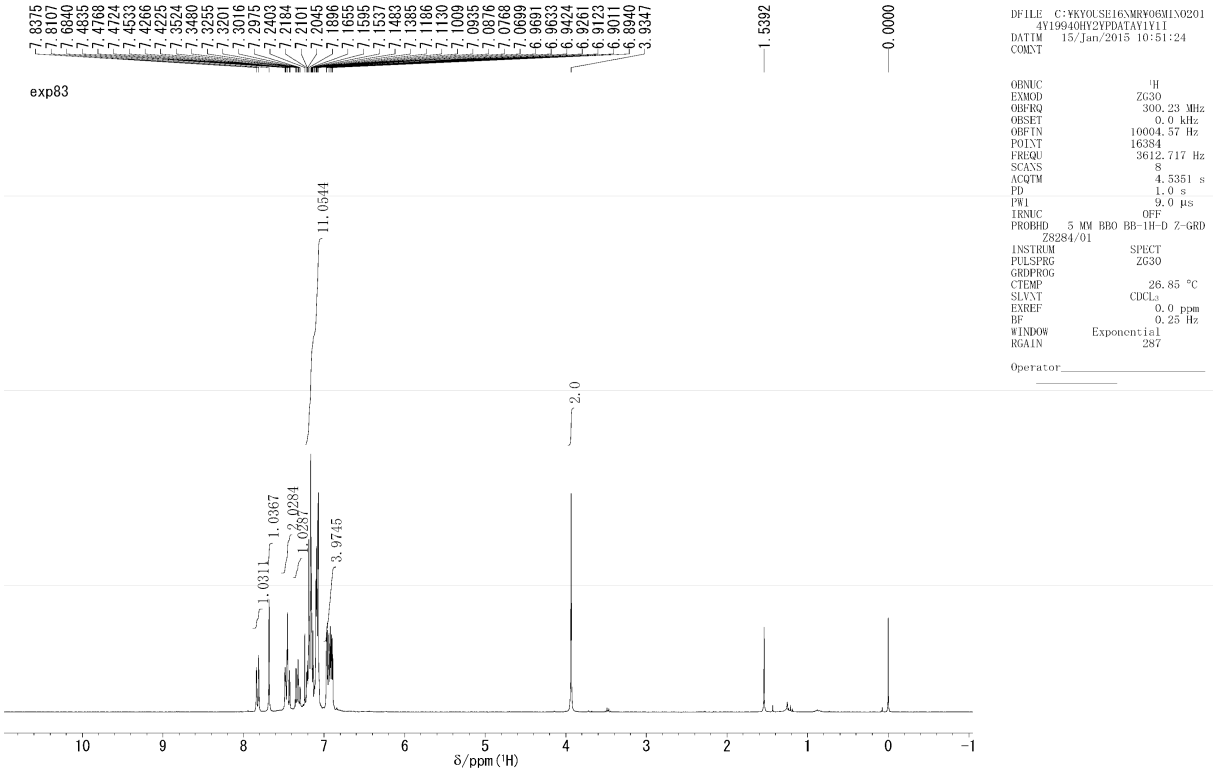
DFILE C:¥KYOUSEI6¥NMR¥06MIN0201
4Y19481CY3¥PDATAY1¥1I
DATIM 23/Jun/2014 20:26:56
COMNT

OBNUC	¹³C
EXMOD	ZGPG30
OBFRQ	75.49 MHz
OBSET	0.0 kHz
OBFIN	10002.06 Hz
POINT	32768
FREQU	18115.94 Hz
SCANS	256
ACQTM	1.8088 s
PD	2.0 s
PW1	0.0 µs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZGPG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCl_3$
EXREF	77.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	2299

Operator_____

^{1}H and ^{13}C NMR of 3-Cinnamyl-1,2-diphenylnaphthalene (5ia)

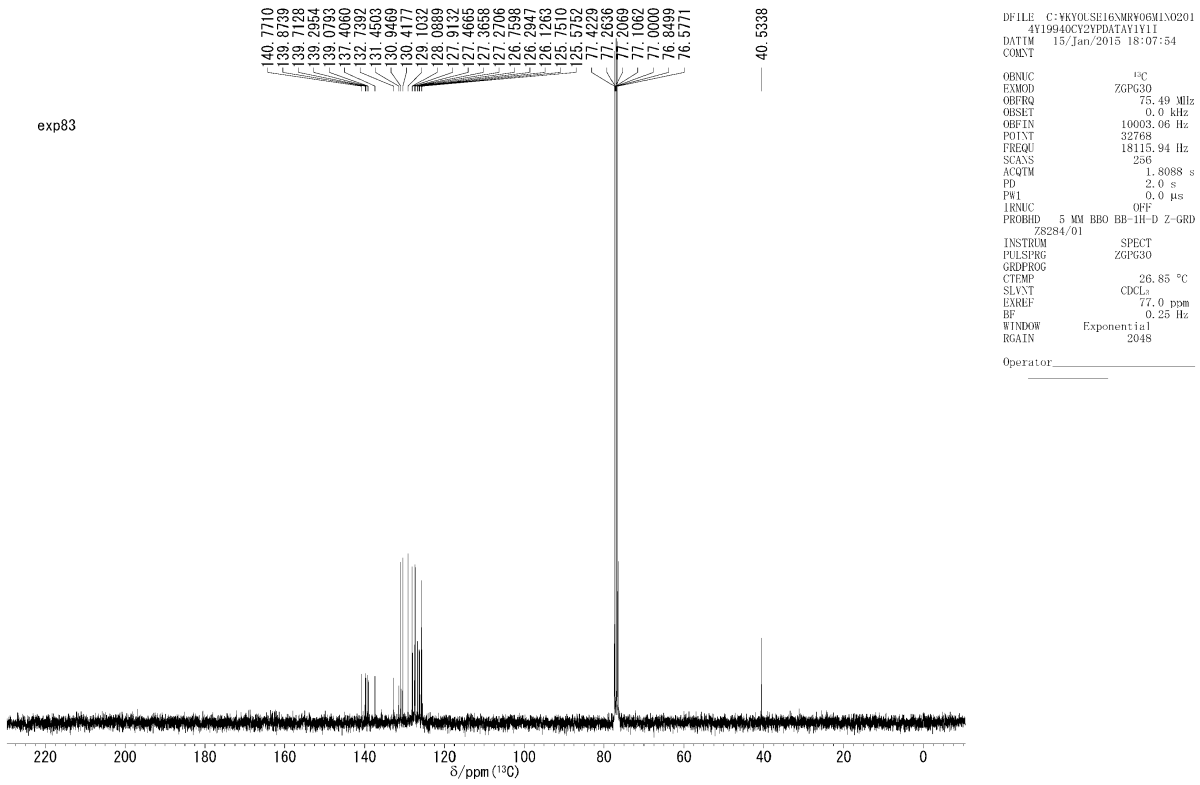




DFILE C:¥KYOUSE16NMR¥06M1NO201
4Y19940HY2YPDATAY1Y1I
DATIM 15/Jan/2015 10:51:24
COMNT

OBNUC	^{1}H
EXMOD	ZG30
OBFRQ	300.23 MHz
OBSET	0.0 kHz
OBFIN	10004.57 Hz
POINT	16384
FREQU	3612.717 Hz
SCANS	8
ACQTM	4.5351 s
PD	1.0 s
PW1	9.0 µs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCl_3$
EXREF	0.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	287

Operator_____

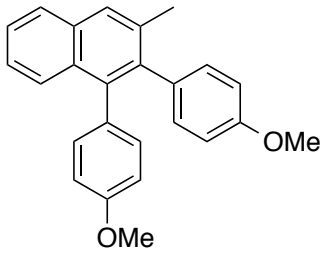


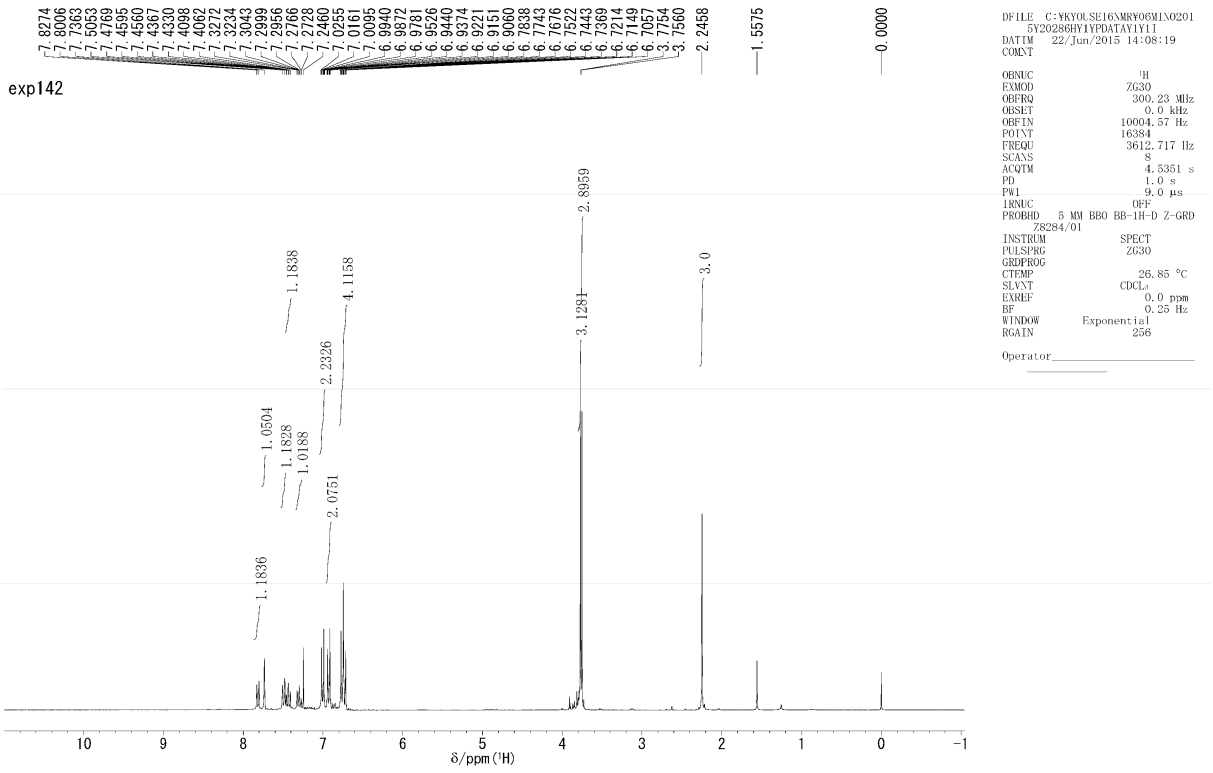
DFILE C:¥KYOUSE16NMR¥06M1NO201
4Y19940CY2YPDATAY1Y1I
DATIM 15/Jan/2015 18:07:54
COMNT

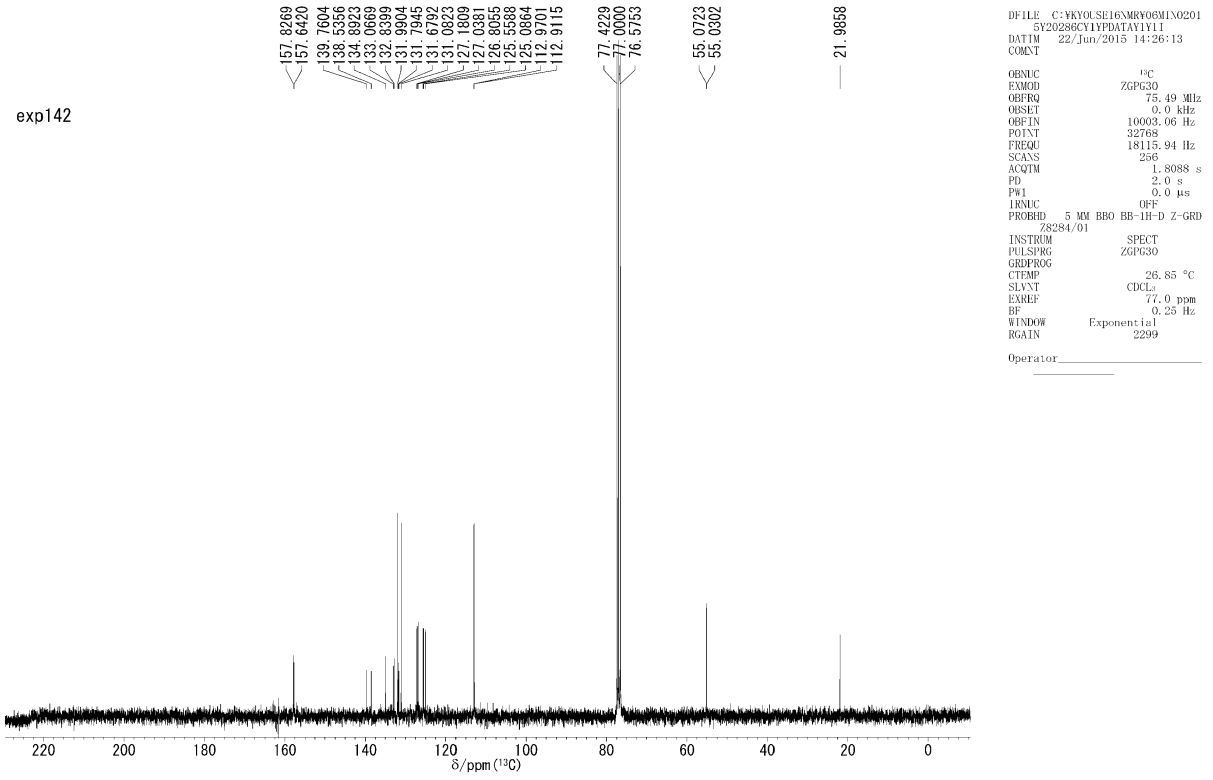
OBNUC	^{13}C
EXMOD	ZGPG30
OBFRQ	75.49 MHz
OBSET	0.0 kHz
OBFIN	10003.06 Hz
POINT	32768
FREQU	18115.94 Hz
SCANS	256
ACQTM	1.8088 s
PD	2.0 s
PW1	0.0 µs
IRNUC	OFF
PROBHD	5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM	SPECT
PULSPRG	ZGPG30
GRDPROG	
CTEMP	26.85 °C
SLVNT	$CDCl_3$
EXREF	77.0 ppm
BF	0.25 Hz
WINDOW	Exponential
RGAIN	2048

Operator_____

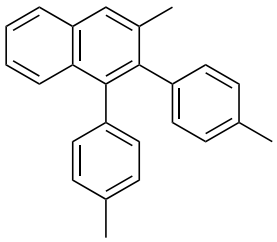
^{1}H and ^{13}C NMR of 1,2-Bis(4-methoxyphenyl)-3-methylnaphthalene (5ab)

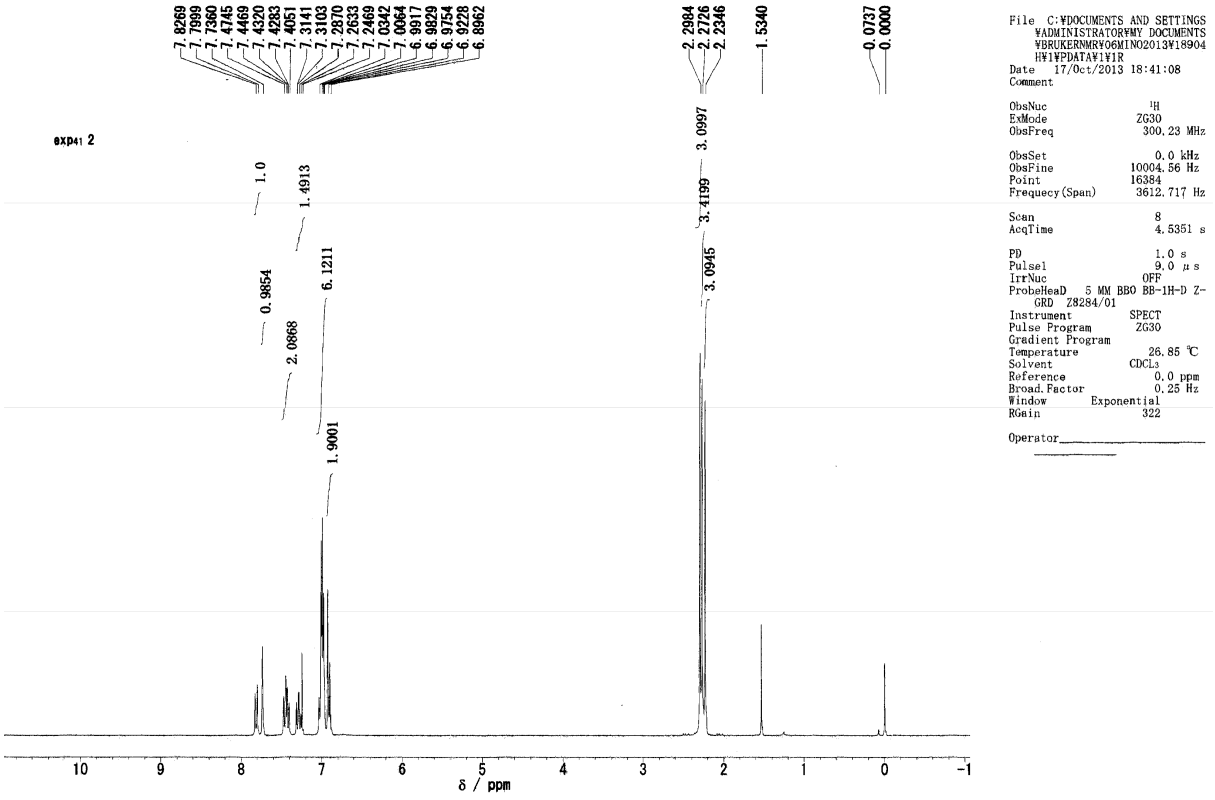






^{1}H and ^{13}C NMR of 3-Methyl-1,2-di-*p*-tolylnaphthalene (5ac)





File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MINO2013¥18904 H¥1¥PDATA¥1¥1R
Date 17/Oct/2013 18:41:08
Comment

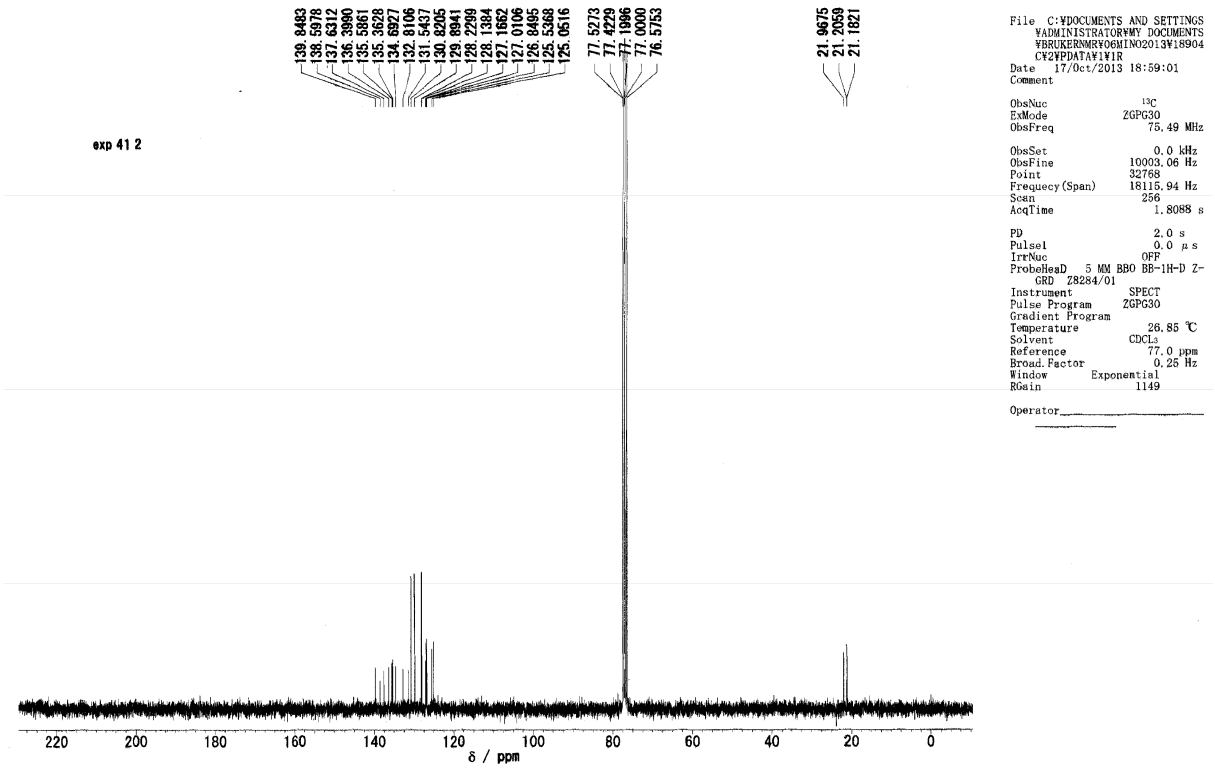
ObsNuc ^{1}H
ExMode ZG30
ObsFreq 300.23 MHz

ObsSet 0.0 kHz
ObsFine 10004.56 Hz
Point 16384
Frequecy(Span) 3612.717 Hz

Scan 8
AcqTime 4.5351 s

PD 1.0 s
Pulsel 9.0 μs
IrrNuc OFF
ProbeHeaD 5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument SPECT
Pulse Program ZG30
Gradient Program
Temperature 26.85 ℃
Solvent $CDCl_3$
Reference 0.0 ppm
Broad.Factor 0.25 Hz
Window Exponential
RGain 322

Operator



File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MINO2013¥18904 C¥2¥PDATA¥1¥1R
Date 17/Oct/2013 18:59:01
Comment

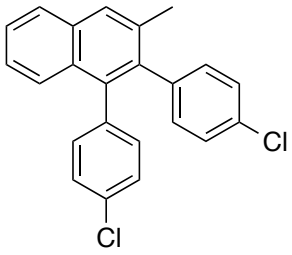
ObsNuc ^{13}C
ExMode ZGPG30
ObsFreq 75.49 MHz

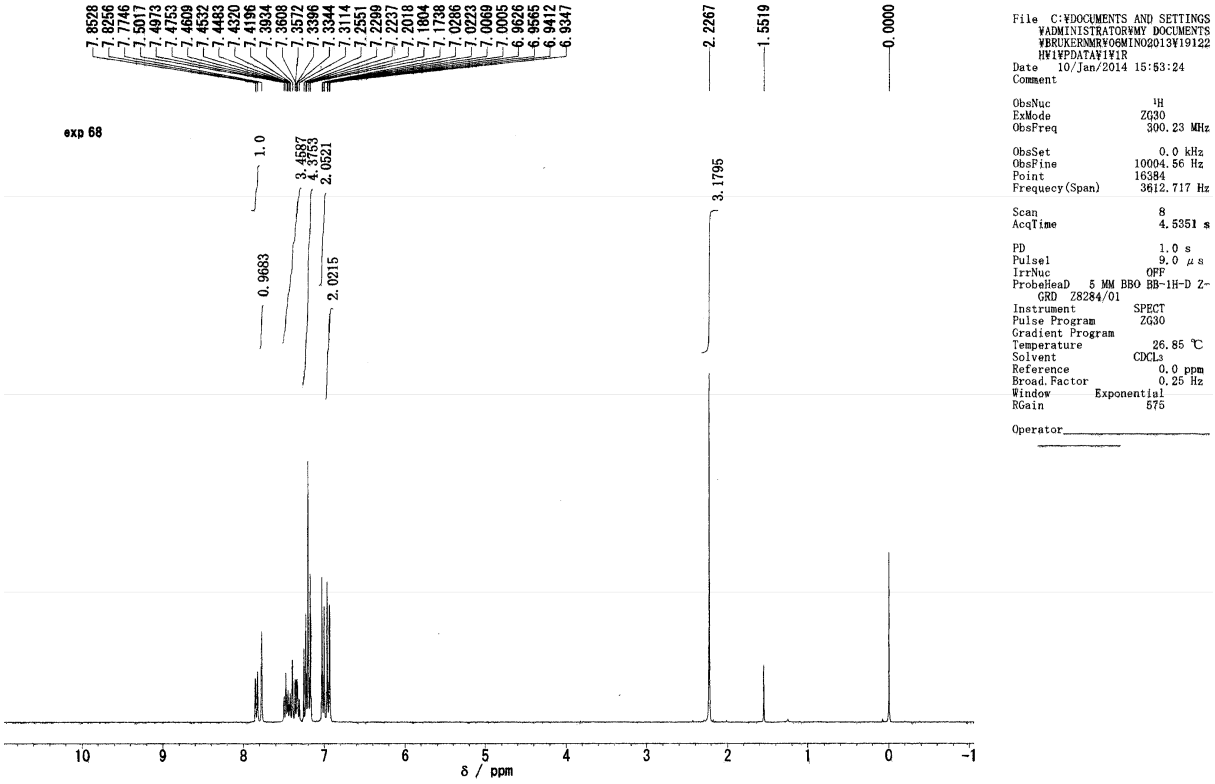
ObsSet 0.0 kHz
ObsFine 10003.06 Hz
Point 32768
Frequecy(Span) 18115.94 Hz
Scan 256
AcqTime 1.8088 s

PD 2.0 s
Pulsel 0.0 μs
IrrNuc OFF
ProbeHeaD 5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument SPECT
Pulse Program ZGPG30
Gradient Program
Temperature 26.85 ℃
Solvent $CDCl_3$
Reference 77.0 ppm
Broad.Factor 0.25 Hz
Window Exponential
RGain 1149

Operator

^{1}H and ^{13}C NMR of 1,2-Bis(4-chlorophenyl)-3-methylnaphthalene (5ad)





File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MIN02013¥19122 HW1¥PDATA¥1¥1R
Date 10/Jan/2014 15:53:24
Comment

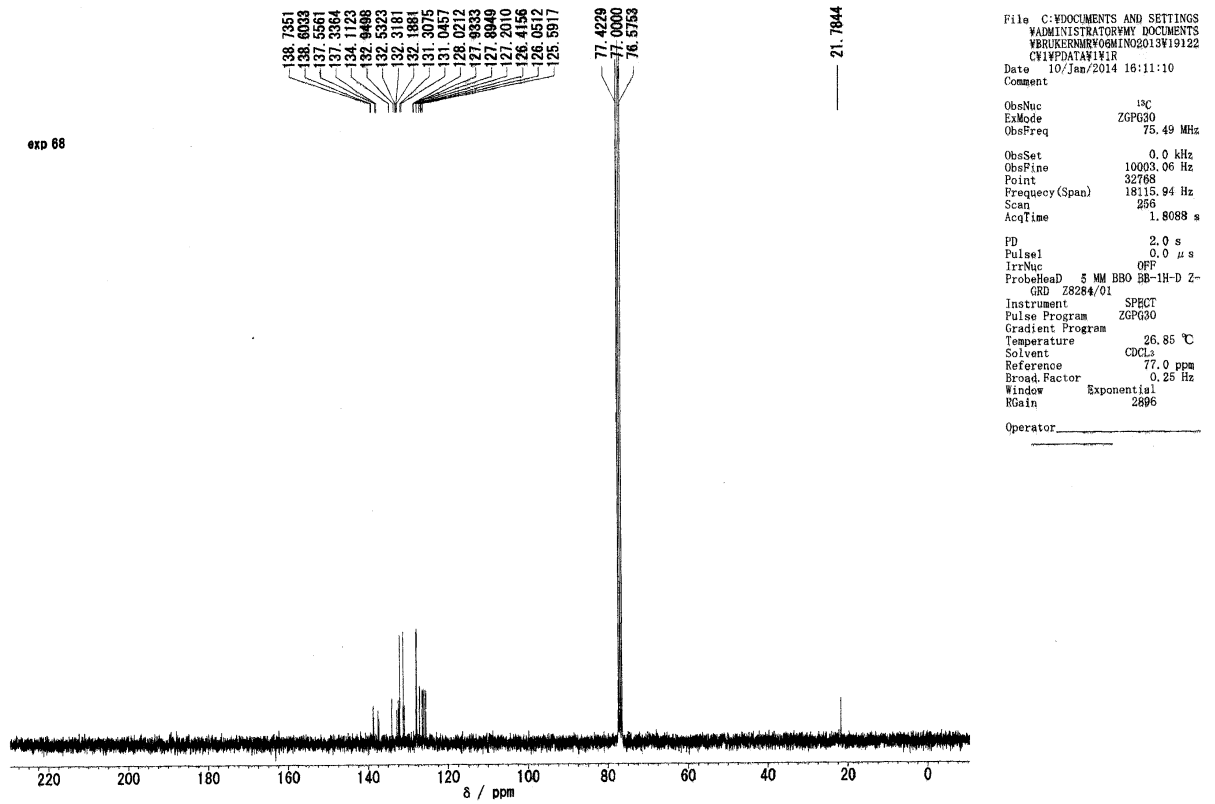
ObsNuc ^{1}H
ExMode ZG30
ObsFreq 300.23 MHz

ObsSet 0.0 kHz
ObsFine 10004.56 Hz
Point 16384
Frequecy(Span) 3612.717 Hz

Scan 8
AcqTime 4.5351 s

PD 1.0 s
Pulse1 9.0 μs
IrrNuc OFF
ProbeHeaD 5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument SPECT
Pulse Program ZG30
Gradient Program
Temperature 26.85 ℃
Solvent CDCl3
Reference 0.0 ppm
Broad.Factor 0.25 Hz
Window Exponential
RGain 575

Operator



File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MIN02013¥19122 CW1¥PDATA¥1¥1R
Date 10/Jan/2014 16:11:10
Comment

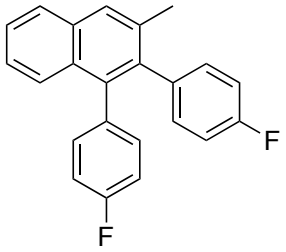
ObsNuc ^{13}C
ExMode ZGPG30
ObsFreq 75.49 MHz

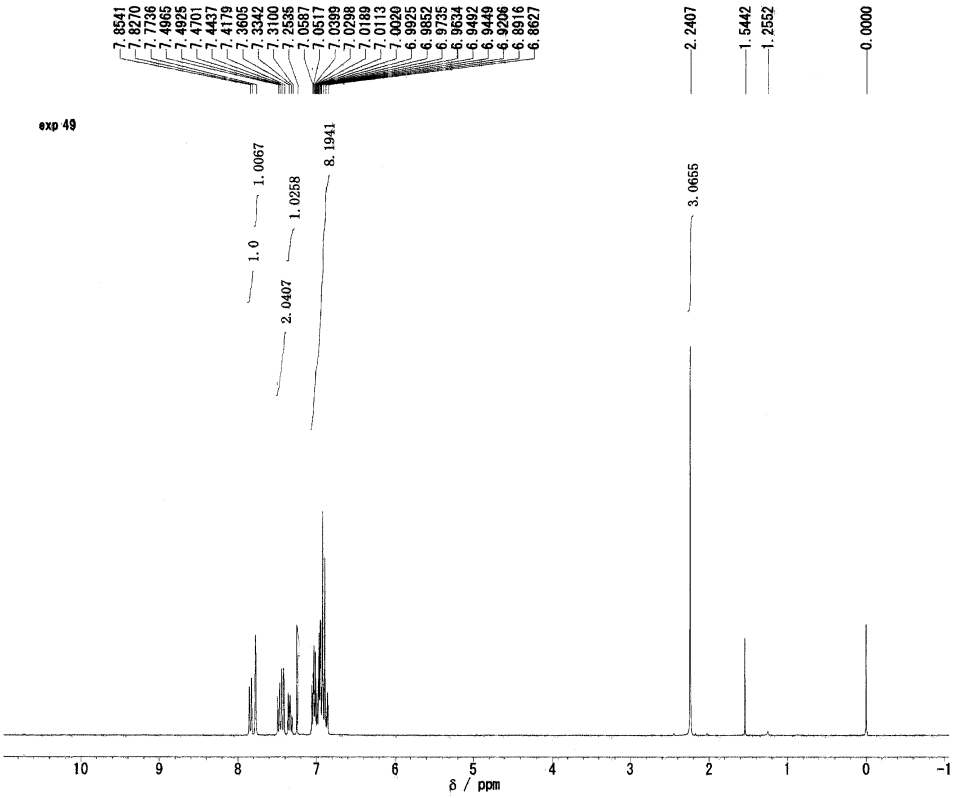
ObsSet 0.0 kHz
ObsFine 10003.06 Hz
Point 32768
Frequecy(Span) 18115.94 Hz
Scan 256
AcqTime 1.8088 s

PD 2.0 s
Pulse1 0.0 μs
IrrNuc OFF
ProbeHeaD 5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument SPECT
Pulse Program ZGPG30
Gradient Program
Temperature 26.85 ℃
Solvent CDCl3
Reference 77.0 ppm
Broad.Factor 0.25 Hz
Window Exponential
RGain 2896

Operator

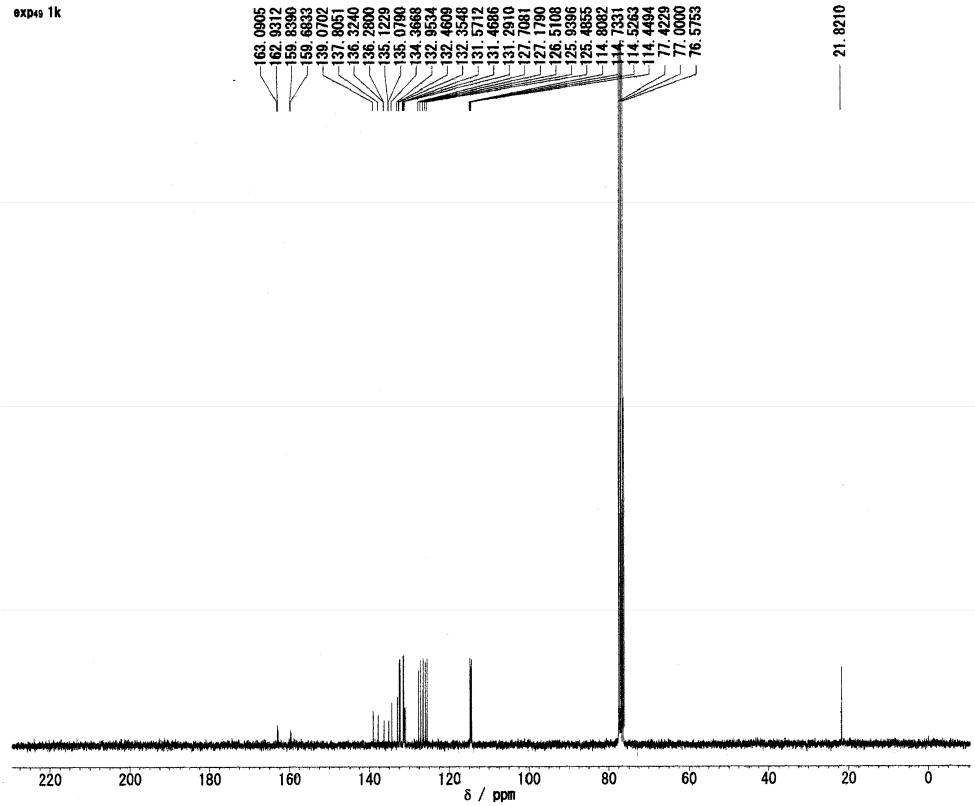
^{1}H and ^{13}C NMR of 1,2-Bis(4-fluorophenyl)-3-methylnaphthalene (5ae)





File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥¥MY DOCUMENTS ¥BRUKERNMR¥06MIN02013¥18977 H¥1¥PDATA¥1¥1R
Date 14/Nov/2013 19:21:20
Comment

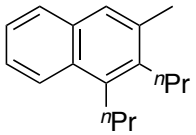
ObsNuc	^{1}H
ExMode	ZG30
ObsFreq	300.23 MHz
ObsSet	0.0 kHz
ObsFine	10004.56 Hz
Point	16384
Frequecy(Span)	3612.717 Hz
Scan	8
AcqTime	4.5351 s
PD	1.0 s
Pulse1	9.0 μs
IrrNuc	OFF
ProbeHead	5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument	SPECT
Pulse Program	ZG30
Gradient Program	
Temperature	26.85 ℃
Solvent	CDCl3
Reference	0.0 ppm
Broad.Factor	0.25 Hz
Window	Exponential
RGain	362
Operator	

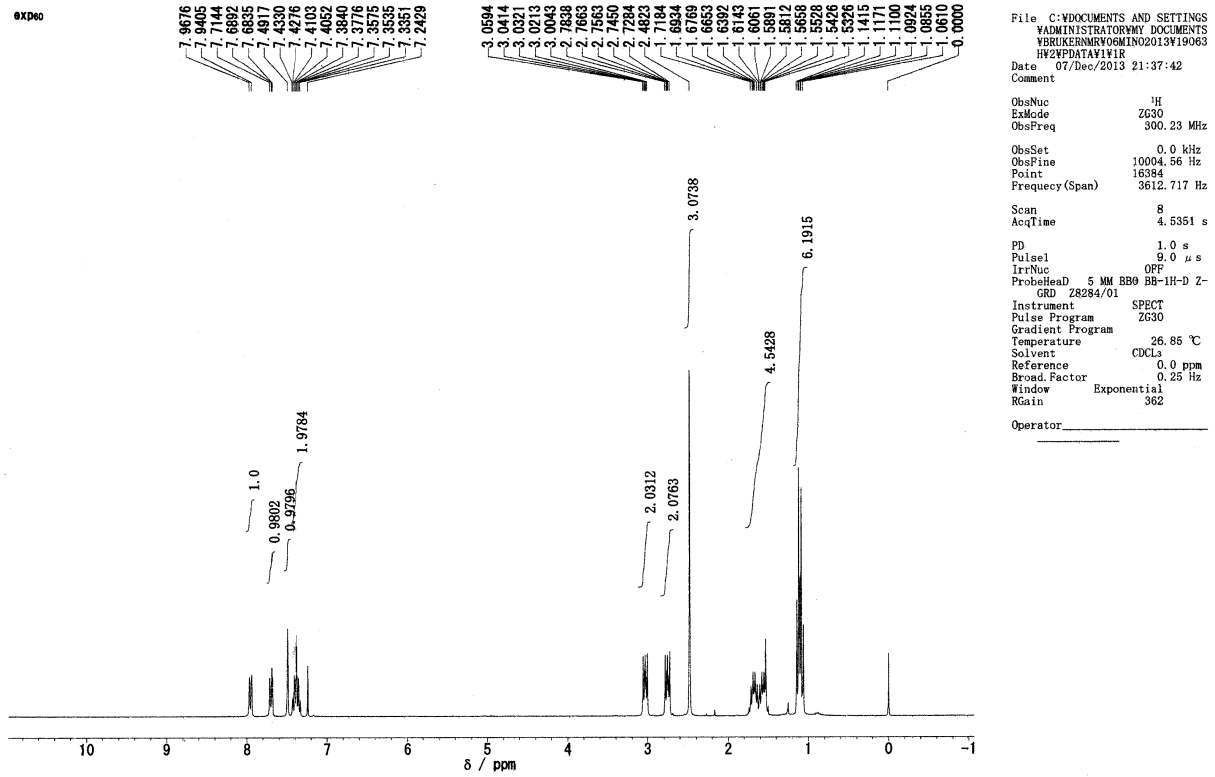


File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥¥MY DOCUMENTS ¥BRUKERNMR¥06MIN02013¥18997 C¥4¥PDATA¥1¥1R
Date 21/Nov/2013 22:47:07
Comment

ObsNuc	^{13}C
ExMode	ZGPG30
ObsFreq	75.49 MHz
ObsSet	0.0 kHz
ObsFine	10003.06 Hz
Point	32768
Frequecy(Span)	18115.94 Hz
Scan	1024
AcqTime	1.8088 s
PD	2.0 s
Pulse1	9.0 μs
IrrNuc	OFF
ProbeHead	5 MM BBO BB-1H-D Z-GRD Z8284/01
Instrument	SPECT
Pulse Program	ZGPG30
Gradient Program	
Temperature	26.85 ℃
Solvent	CDCl3
Reference	77.0 ppm
Broad.Factor	0.25 Hz
Window	Exponential
RGain	2896
Operator	

^{1}H and ^{13}C NMR of 3-Methyl-1,2-dipropylnaphthalene (5ag)

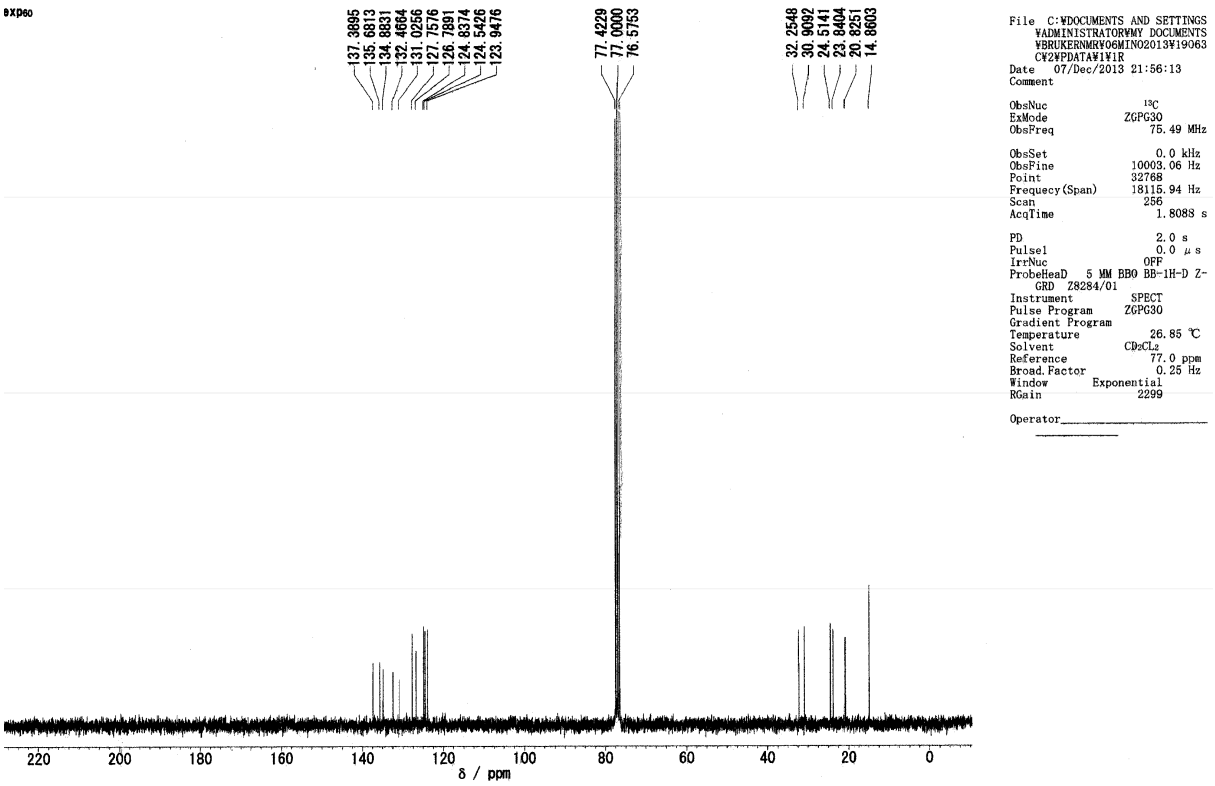




File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MIN02013¥19063 H¥2¥PDATA¥1¥1R
Date 07/Dec/2013 21:37:42
Comment

ObsNuc	^{1}H
ExMode	ZG30
ObsFreq	300.23 MHz
ObsSet	0.0 kHz
ObsPine	10004.56 Hz
Point	16384
Frequecy(Span)	3612.717 Hz
Scan	8
AcqTime	4.5351 s
PD	1.0 s
Pulse1	9.0 μs
IrrNuc	OFF
ProbeHeaD	5 MM BB0 BB-1H-D Z-GRD Z8284/01
Instrument	SPECT
Pulse Program	ZG30
Gradient Program	
Temperature	26.85 ℃
Solvent	$CDCl_3$
Reference	0.0 ppm
Broad.Factor	0.25 Hz
Window	Exponential
RGain	362

Operator

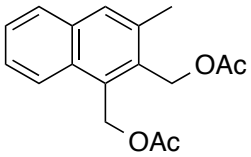


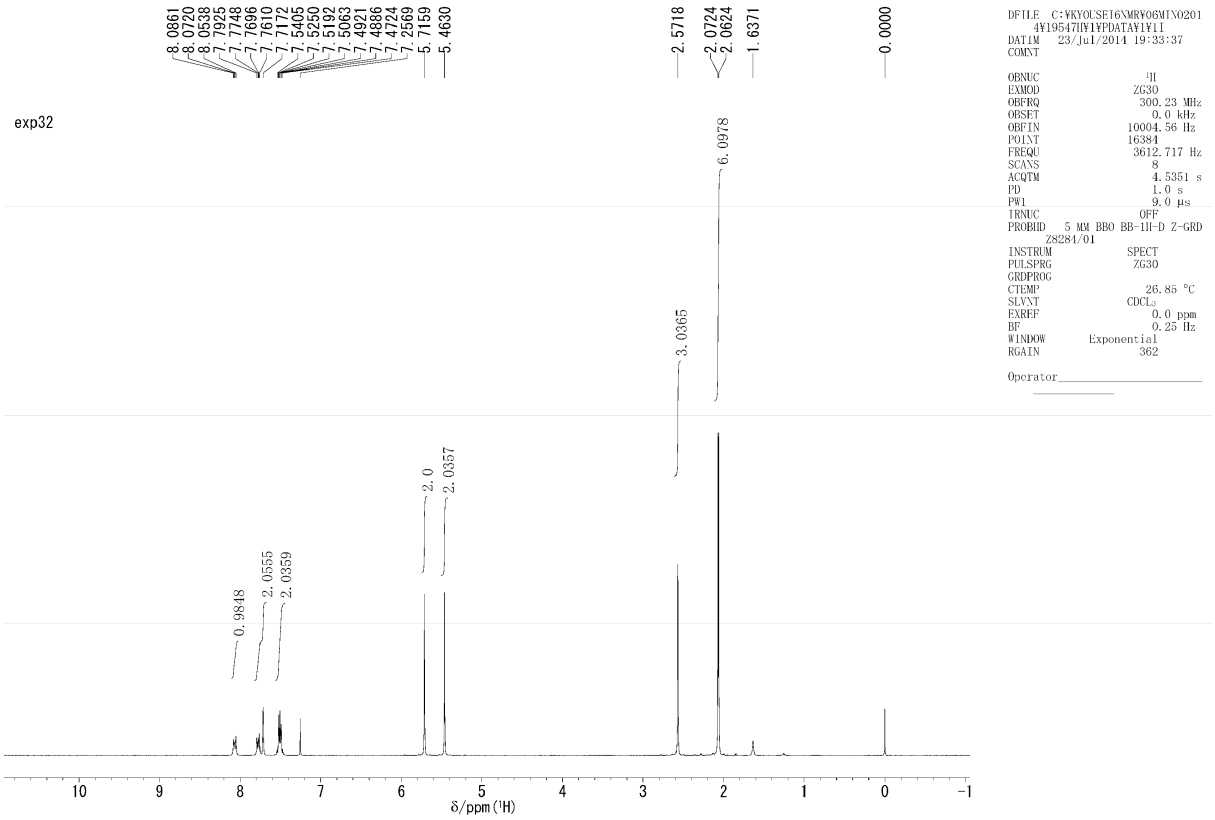
File C:¥DOCUMENTS AND SETTINGS ¥ADMINISTRATOR¥MY DOCUMENTS ¥BRUKERNMR¥06MIN02013¥19063 C¥2¥PDATA¥1¥1R
Date 07/Dec/2013 21:56:13
Comment

ObsNuc	^{13}C
ExMode	ZGPG30
ObsFreq	75.49 MHz
ObsSet	0.0 kHz
ObsPine	10003.06 Hz
Point	32768
Frequecy(Span)	18115.94 Hz
Scan	256
AcqTime	1.8088 s
PD	2.0 s
Pulse1	0.0 μs
IrrNuc	OFF
ProbeHeaD	5 MM BB0 BB-1H-D Z-GRD Z8284/01
Instrument	SPECT
Pulse Program	ZGPG30
Gradient Program	
Temperature	26.85 ℃
Solvent	CD_2CL_2
Reference	77.0 ppm
Broad.Factor	0.25 Hz
Window	Exponential
RGain	2299

Operator

^{1}H and ^{13}C NMR of 3-Methyl-1,2-naphthalene dimethanol, 1,2-diacetate (5ai)

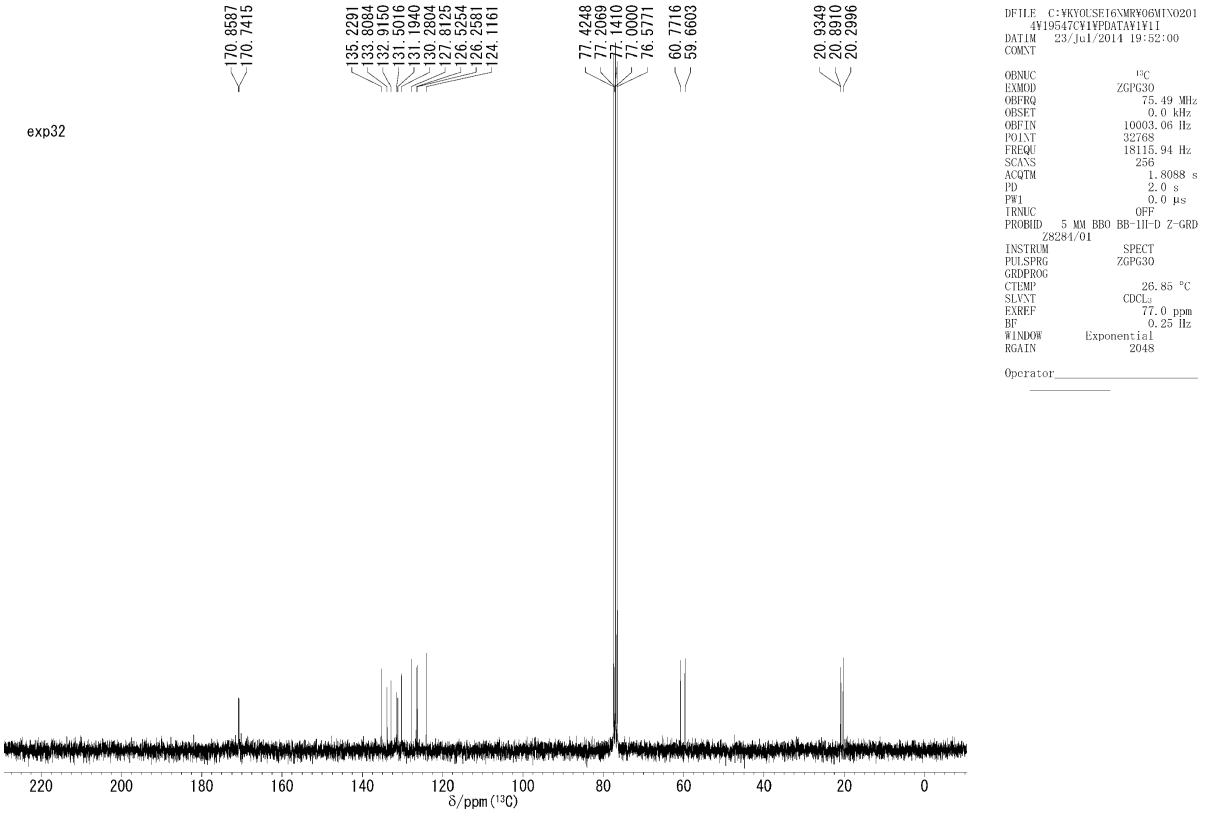




DFILE C:¥KYOUSEI6¥NMR¥06MIX0201
4¥19547H¥1¥PDATA¥1¥1I
DATIM 23/Jul/2014 19:33:37
COMNT

OBNUC ¹H
EXMOD ZG30
OBFRQ 300.23 MHz
OBSET 0.0 kHz
OBFIN 10004.56 Hz
POINT 16384
FREQU 3612.717 Hz
SCANS 8
ACQTM 4.5351 s
PD 1.0 s
PW1 9.0 µs
TRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM SPECT
PULSPRG ZG30
GRDPROG
CTEMP 26.85 °C
SLVNT CDCL₃
EXREF 0.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 362

Operator_____

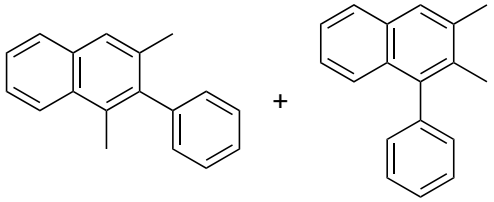


DFILE C:¥KYOUSEI6¥NMR¥06MIX0201
4¥19547C¥1¥PDATA¥1¥1I
DATIM 23/Jul/2014 19:52:00
COMNT

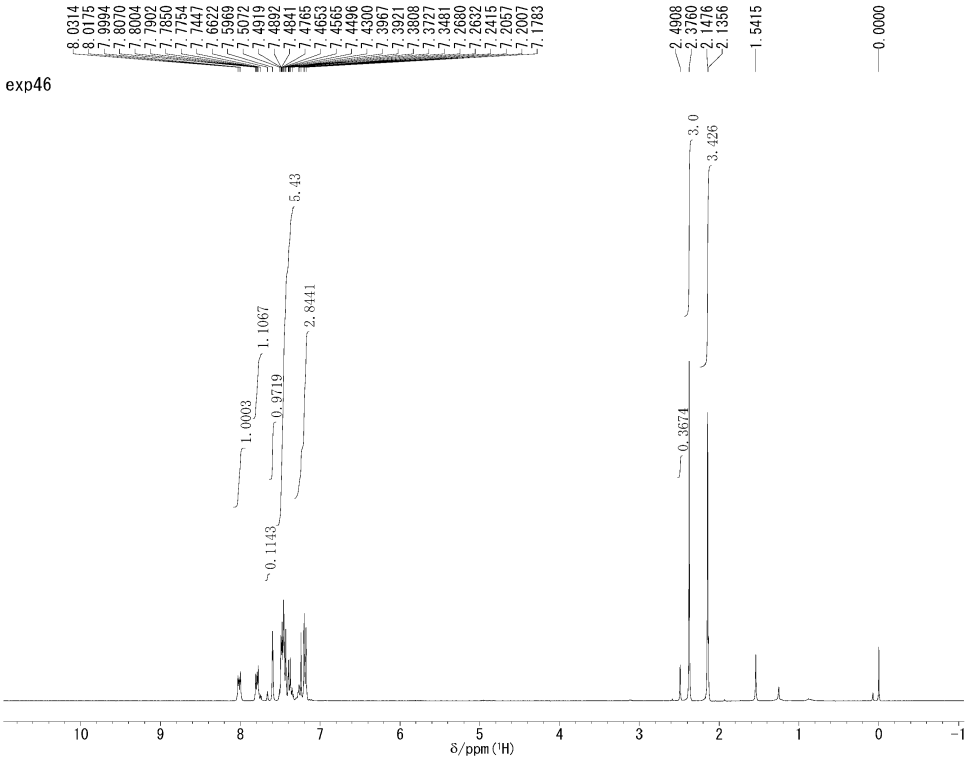
OBNUC ¹³C
EXMOD ZGPG30
OBFRQ 75.49 MHz
OBSET 0.0 kHz
OBFIN 10003.06 Hz
POINT 32768
FREQU 18115.94 Hz
SCANS 256
ACQTM 1.8088 s
PD 2.0 s
PW1 0.0 µs
TRNUC OFF
PROBHD 5 MM BBO BB-1H-D Z-GRD Z8284/01
INSTRUM SPECT
PULSPRG ZGPG30
GRDPROG
CTEMP 26.85 °C
SLVNT CDCL₃
EXREF 77.0 ppm
BF 0.25 Hz
WINDOW Exponential
RGAIN 2048

Operator_____

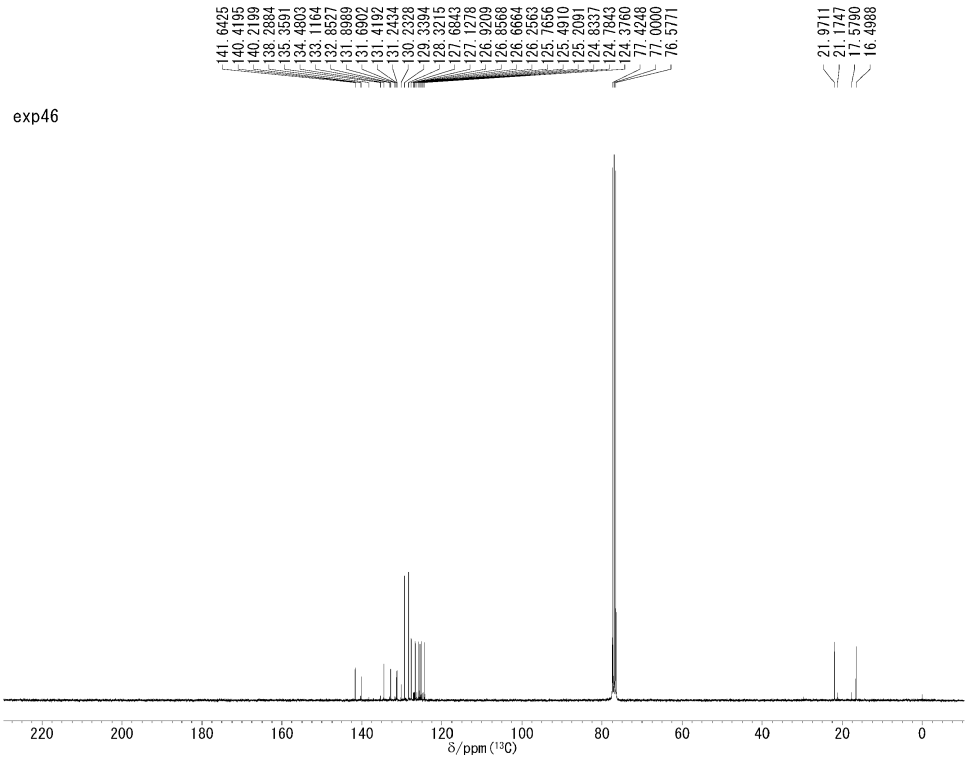
^{1}H and ^{13}C NMR of **1,3-Dimethyl-2-phenylnaphthalene** (**5aj**) and **1,2-Dimethyl-3-phenylnaphthalene** (**5aj'**)



5aj **5aj'**




```
DFILE   C:¥KYOUSEI6¥NMR¥06M1N0201
        3Y18931HY3YPDATAY1Y1I
DATIM   29/Oct/2013 15:27:23
COMNT

OBNUC                   ¹H
EXMOD                 ZG30
OBFRQ               300.23 MHz
OBSET                 0.0 kHz
OBFIN             10004.56 Hz
POINT                 16384
FREQU              3612.717 Hz
SCANS                     8
ACQTM                 4.5351 s
PD                    1.0 s
PW1                   9.0 µs
IRNUC                   OFF
PROBHD   5 MM BBO BB-1H-D Z-GRD
         Z8284/01
INSTRUM                 SPECT
PULSPRG                 ZG30
GRDPROG
CTEMP                 26.85 °C
SLVNT                 CDCL₃
EXREF              10.9681 p
       pm
BF                    0.25 Hz
WINDOW          Exponential
RGAIN                   256

Operator
```




```
DFILE   C:¥KYOUSEI6¥NMR¥06M1N0201
        3Y19257CY1YPDATAY1Y1I
DATIM   10/Mar/2014 12:02:06
COMNT

OBNUC                  ¹³C
EXMOD              ZGPG30
OBFRQ               75.49 MHz
OBSET                 0.0 kHz
OBFIN             10003.06 Hz
POINT                 32768
FREQU              18115.94 Hz
SCANS                  7168
ACQTM                 1.8088 s
PD                    2.0 s
PW1                   0.0 µs
IRNUC                   OFF
PROBHD   5 MM BBO BB-1H-D Z-GRD
         Z8284/01
INSTRUM                 SPECT
PULSPRG             ZGPG30
GRDPROG
CTEMP                 26.85 °C
SLVNT                 CDCL₃
EXREF                 77.0 ppm
BF                    0.25 Hz
WINDOW          Exponential
RGAIN                  2299

Operator
```