Synthesis of Nitrogen-Containing Fused-Polycyclic Compounds from Tyramine Derivatives Using Phenol Dearomatization and Cascade Cyclization

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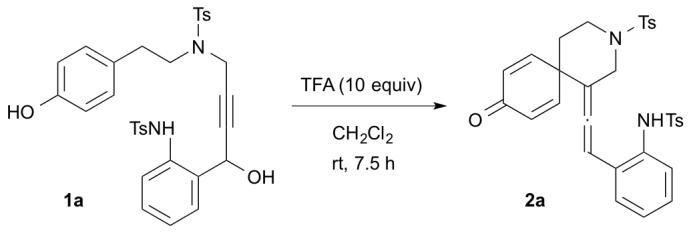
Electronic Supplementary Information (ESI)

1. General

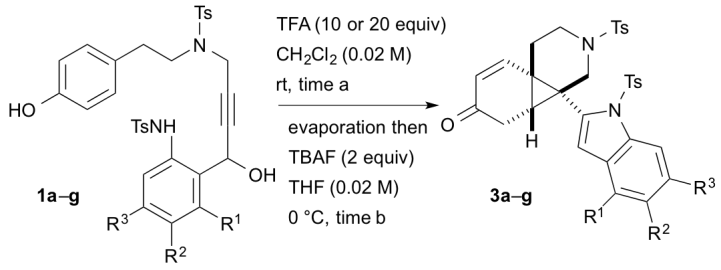
Infrared (IR) spectra were recorded on a JASCO FT/IR 230 Fourier transform infrared spectrophotometer, equipped with ATR (Smiths Detection, DuraSample IR II). NMR spectra were recorded on a JEOL ecs 400 spectrometer. Chemical shifts in $CDCl_3$, were reported downfield from TMS (= 0 ppm) for ^{1}H NMR. For ^{13}C NMR, chemical shifts were reported in the scale relative to the solvent signal [$CHCl_3$ (77.0 ppm)] as an internal reference. 2D-NMR experiments and NOE experiments were performed on a JEOL ecp 600 spectrometer. ESI mass spectra were measured on JEOL AccuTOF LC-plus JMS-T100LP. Melting points were measured with a SIBATA NEL-270 melting point apparatus. Analytical thin layer chromatography was performed on Merck Art. 5715, Kieselgel 60F254/0.25 mm thickness plates. Column chromatography was performed with silica gel 60 N (spherical, neutral 63-210 mesh). Reactions were carried out in dry solvent. Other reagents were purified by the usual methods.

2. TFA-promoted intramolecular *ipso*-Friedel-Crafts allenylation of 1a (Table 1, Entry 5)

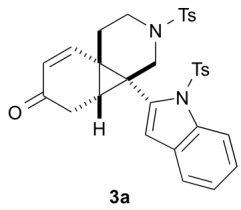


To a stirred solution of **1a** (30.2 mg, 0.05 mmol) in CH_2Cl_2 (2.0 mL) at room temperature was added TFA (0.5 mL, 1.0 M in CH_2Cl_2, 0.5 mmol). After being stirred for 7.5 h at the same temperature, the reaction mixture was concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAc = 2/1) to give the desired product **2a** (28.5 mg, 97%) as white solid: melting point 40 °C; Rf 0.33 (*n*-hexane/EtOAc = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.94–2.07 (m, 2H), 2.41 (s, 3H), 2.47 (s, 3H), 3.29–3.34 (m, 1H), 3.38–3.44 (m, 1H), 3.91 (d, *J* = 13.2 Hz, 1H), 3.97 (d, *J* = 13.2 Hz, 1H), 6.22 (dd, *J* = 10.0, 1.2 Hz, 1H), 6.27 (dd, *J* = 10.0, 1.2 Hz, 1H), 6.41 (s, 1H), 6.87 (dd, *J* = 10.0, 3.2 Hz, 1H), 7.00 (s, 1H), 7.06–7.12 (m, 5H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.39 (d, *J* = 8.0 Hz, 2H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.71 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 21.6, 35.0, 42.0, 42.2, 46.2, 94.6, 101.3, 123.6, 125.5, 126.0, 127.3 (2C), 127.7 (2C), 128.7, 129.0, 129.1, 129.3, 129.8 (2C), 130.0 (2C), 133.0, 134.1, 136.3, 144.1, 144.3, 149.1, 149.8, 185.0, 201.0; IR (ATR) ν 1662, 1624, 1493, 1330, 1156, 1090, 972, 918, 814, 732, 659 cm^{-1}; HRMS (ESI^+) calcd for $C_{32}H_{30}N_2NaO_5S_2^+$ 609.1488 ($M+Na^+$) found 609.1485.

3. General procedure for the synthesis of penta-substituted cyclopropane derivatives using a one-pot dearomatization of phenol–cascade cyclization sequence and product characterization



General Procedure: To a stirred solution of **1** (0.05 mmol) in CH_2Cl_2 (2.0 mL) at room temperature was added TFA (0.5 mL, 1.0 M in CH_2Cl_2, 0.5 mmol). After being stirred for required time at the same temperature, the reaction mixture was evaporated *in vacuo*, and the obtained mixture was azeotropic dried with toluene. TBAF (0.10 mL, 1.0 M in THF, 0.10 mmol) was added to a stirred solution of the crude product in THF (2.4 mL) at 0 °C. After required time, the reaction mixture was quenched with saturated aqueous NH_4Cl, and extracted twice with EtOAc. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography to give the desired products **3**.

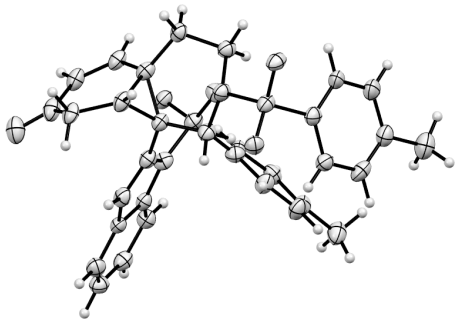


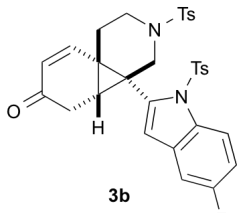
3a

Compound 3a. White solid; melting point 108 °C; Rf 0.46 (*n*-hexane/EtOAc = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.22 (dd, *J* = 13.6, 5.2 Hz, 1H), 2.31–2.39 (m, 2H), 2.32 (s, 3H), 2.38 (s, 3H), 2.79–2.89 (m, 2H), 2.93 (d, *J* = 19.2 Hz, 1H), 3.15 (d, *J* = 11.2 Hz, 1H), 3.66 (td, *J* = 8.0, 3.2 Hz, 1H), 3.95 (d, *J* = 11.2 Hz, 1H), 5.50 (d, *J* = 10.0 Hz, 1H), 6.66 (s, 1H), 6.79 (d, *J* = 10.0 Hz, 1H), 7.08 (d, *J* = 8.4 Hz, 2H), 7.11–7.15 (m, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.26 (d, *J* = 8.4 Hz, 2H), 7.44 (dd, *J* = 6.8, 2.4 Hz, 1H), 7.51 (dd, *J* = 6.8, 2.4 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 21.4, 25.4, 26.7, 28.3, 32.4, 33.3, 41.7, 49.4, 113.9, 117.3, 121.1, 123.5, 125.1, 125.8 (2C), 126.9, 127.2 (2C), 127.7, 129.6 (2C), 129.7 (2C), 133.4, 135.7, 136.4, 136.6, 143.3, 144.4, 152.9, 194.2; IR (ATR) ν 1671, 1452, 1341, 1167, 1123, 1090, 941, 814, 749, 670 cm^{-1}; HRMS (ESI^+) calcd for $C_{32}H_{30}N_2NaO_5S_2^+$ 609.1488 ($M+Na^+$) found 609.1500.

Structural determination of **3a** based on X-ray crystal structure analysis. (CCDC 1008194)
Crystal Data: Colorless platelet crystal, $C_{32}H_{30}N_2O_5S_2$, *M* = 586.72, triclinic, *a* = 11.7659(3), *b* = 13.1216(4), *c* = 19.8185(5) Å, *V* = 2821.3(2) $Å^3$, *T* = 93K, space group P–1, *Z* = 4, 29210 reflections measured, 10086 unique (Rint = 0.1012). R1 = 0.0782, wR2 = 0.1915.

ORTEP of **3a**

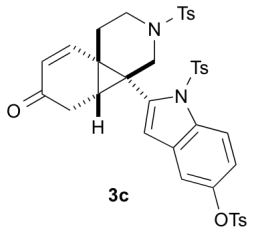




3b

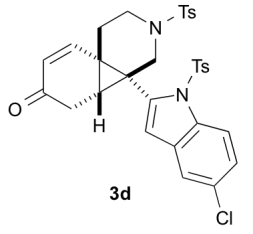
Compound 3b. White solid; melting point 113 °C; Rf 0.24 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.22 (dd, *J* = 13.6, 6.0 Hz, 1H), 2.32–2.41 (m, 2H), 2.37 (s, 3H), 2.41 (s, 3H), 2.81–2.88 (m, 2H), 2.92 (d, *J* = 19.2 Hz, 1H), 3.13 (d, *J* = 11.2 Hz, 1H), 3.66 (br-dd, *J* = 10.0, 8.0 Hz, 1H), 3.94 (d, *J* = 11.2 Hz,

1H), 5.52 (d, *J* = 10.4 Hz, 1H), 6.61 (s, 1H), 6.79 (d, *J* = 10.4 Hz, 1H), 6.88 (td, *J* = 9.2, 2.4 Hz, 1H), 7.11 (d, *J* = 8.0 Hz, 2H), 7.11 (dd, *J* = 8.0, 2.4 Hz, 1H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.46 (dd, *J* = 9.2, 4.4 Hz, 1H), 7.58 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 21.6, 25.5, 26.8, 28.4, 32.5, 33.4, 41.8, 49.4, 106.8 (d, *J* = 23.9 Hz), 113.2 (d, *J* = 24.8 Hz), 115.2 (d, *J* = 9.6 Hz), 117.1 (d, *J* = 3.8 Hz), 126.0 (2C), 127.2, 127.3 (2C), 128.8 (d, *J* = 10.5 Hz), 129.7 (2C), 129.9 (2C), 132.8, 133.6, 136.6, 137.7, 143.5, 144.8, 153.0, 159.5 (d, *J* = 240.2 Hz), 194.4; IR (ATR) ν 1671, 1596, 1466, 1339, 1251, 1161, 1089, 940, 809, 735, 713, 673 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{32}H_{29}FN_2NaO_5S_2^+$ 627.1394 (M+Na$^+$) found 627.1401.



3c

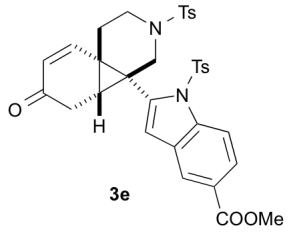
Compound 3c. White solid; melting point 115 °C; Rf 0.61 (*n*-hexane/EtOAc = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.21 (dd, *J* = 14.4, 5.2 Hz, 1H), 2.31–2.40 (m, 2H), 2.39 (s, 3H), 2.42 (s, 3H), 2.47 (s, 3H), 2.80–2.91 (m, 3H), 3.13 (d, *J* = 11.2 Hz, 1H), 3.66 (br-dd, *J* = 12.0, 7.2 Hz, 1H), 3.92 (d, *J* = 11.2 Hz, 1H), 5.51 (d, *J* = 10.0 Hz, 1H), 6.54 (s, 1H), 6.76 (d, *J* = 10.0 Hz, 1H), 6.84 (dd, *J* = 8.8, 1.6 Hz, 1H), 6.99 (d, *J* = 2.0 Hz, 1H), 7.13 (d, *J* = 8.4 Hz, 2H), 7.23–7.32 (m, 6H), 7.39 (d, *J* = 9.2 Hz, 1H), 7.58 (d, *J* = 7.6 Hz, 2H), 7.66 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 21.6, 21.7, 25.4, 26.8, 28.3, 32.5, 33.4, 41.8, 49.5, 114.5, 114.9, 116.8, 119.9, 126.1 (2C), 127.2, 127.3 (2C), 128.4, 128.4 (2C), 129.8 (2C), 129.8 (2C), 130.0 (2C), 132.2, 133.6, 134.7, 136.4, 137.8, 143.6, 145.0, 145.5, 145.8, 152.9, 194.3; IR (ATR) ν 1671, 1596, 1458, 1366, 1163, 1090, 956, 885, 813, 737, 662 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{39}H_{36}N_2NaO_8S_3^+$ 779.1526 (M+Na$^+$) found 779.1520.



3d

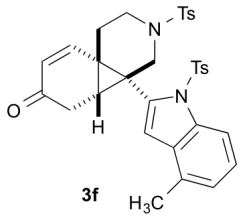
Compound 3d. Pale yellow solid; melting point 110–112 °C; Rf 0.27 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.22 (dd, *J* = 14.0, 5.2 Hz, 1H), 2.34–2.45 (m, 2H), 2.38 (s, 3H), 2.42 (s, 3H), 2.81–2.87 (m, 2H), 2.92 (d, *J* = 18.8 Hz, 1H), 3.12 (d, *J* = 11.6 Hz, 1H), 3.66 (br-t, *J* = 8.0 Hz, 1H), 3.94 (d, *J* = 11.6 Hz, 1H), 5.52 (d, *J* = 10.0 Hz, 1H), 6.58 (s, 1H), 6.80 (d, *J* = 10.0 Hz, 1H), 7.09–7.12 (m, 3H), 7.23–7.28 (m, 4H), 7.41–7.45 (m, 2H), 7.58 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 21.6, 25.5, 26.8, 28.4, 32.4, 33.4, 41.8, 49.4, 115.1, 116.6, 120.8, 125.5, 126.0 (2C), 127.2, 127.3 (2C), 129.0, 129.5, 129.8 (2C), 129.9 (2C), 133.6, 134.8, 136.4, 137.4, 143.6, 144.9, 153.1, 194.5; IR (ATR) ν 1671, 1448, 1340, 1250, 1163, 1090, 940, 876, 811, 734, 668 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{32}H_{29}ClN_2NaO_5S_2^+$

643.1099 ($M+Na^+$) found 643.1100.



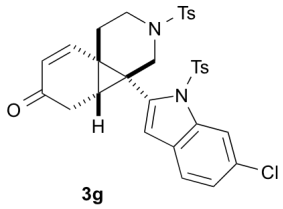
3e

Compound 3e. White solid; melting point 145–147 °C; Rf 0.54 (*n*-hexane/EtOAc = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.24 (dd, *J* = 14.0, 4.8 Hz, 1H), 2.33–2.44 (m, 2H), 2.37 (s, 3H), 2.42 (s, 3H), 2.82–2.88 (m, 2H), 2.94 (d, *J* = 19.2 Hz, 1H), 3.16 (d, *J* = 11.2 Hz, 1H), 3.67 (br-dd, *J* = 11.2, 8.0 Hz, 1H), 3.89 (s, 3H), 3.97 (d, *J* = 11.2 Hz, 1H), 5.53 (d, *J* = 10.0 Hz, 1H), 6.72 (s, 1H), 6.81 (d, *J* = 10.0 Hz, 1H), 7.11 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.54 (d, *J* = 8.4 Hz, 1H), 7.59 (d, *J* = 8.0 Hz, 2H), 7.84 (dd, *J* = 8.4, 1.2 Hz, 1H), 8.17 (d, *J* = 1.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 21.7, 25.5, 26.8, 28.4, 32.4, 33.4, 41.8, 49.4, 52.1, 113.8, 117.5, 123.5, 125.7, 126.1 (2C), 126.4, 127.2, 127.3 (2C), 127.6, 129.7 (2C), 129.9 (2C), 133.5, 136.3, 137.4, 138.9, 143.6, 145.0, 152.9, 166.8, 194.3; IR (ATR) ν 1716, 1671, 1341, 1253, 1163, 1088, 936, 813, 732, 700, 666 cm^{-1}; HRMS (ESI^+) calcd for $C_{34}H_{32}N_2NaO_7S_2^+$ 667.1543 ($M+Na^+$) found 667.1549.



3f

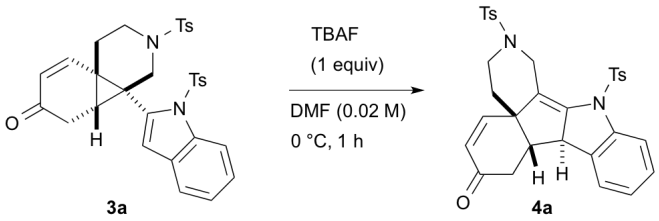
Compound 3f. White solid; melting point 119–120 °C; Rf 0.49 (*n*-hexane/EtOAc = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.21 (dd, *J* = 14.0, 5.2 Hz, 1H), 2.18–2.39 (m, 2H), 2.36 (s, 3H), 2.41 (s, 3H), 2.44 (s, 3H), 2.81–2.90 (m, 2H), 2.99 (d, *J* = 19.2 Hz, 1H), 3.14 (d, *J* = 11.2 Hz, 1H), 3.66 (br-dd, *J* = 11.2, 8.0 Hz, 1H), 3.95 (d, *J* = 11.2 Hz, 1H), 5.50 (d, *J* = 10.4 Hz, 1H), 6.68 (s, 1H), 6.76 (d, *J* = 10.4 Hz, 1H), 6.95 (d, *J* = 8.0 Hz, 1H), 7.03 (t, *J* = 8.0 Hz, 1H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.3, 21.5, 21.6, 25.4, 26.9, 28.5, 32.7, 33.6, 41.8, 49.7, 111.6, 115.8, 124.0, 125.3, 126.0 (2C), 127.2, 127.3 (2C), 127.5, 129.8 (2C), 129.8 (2C), 130.9, 133.7, 135.2, 136.4, 136.9, 143.5, 144.5, 153.1, 194.6; IR (ATR) ν 1669, 1597, 1340, 1164, 1091, 944, 813, 733, 702, 658 cm^{-1}; HRMS (ESI^+) calcd for $C_{33}H_{32}N_2NaO_5S_2^+$ 623.1645 ($M+Na^+$) found 623.1669.



3g

Compound 3g. White solid; melting point 127–129 °C; Rf 0.40 (*n*-hexane/EtOAc = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.22 (dd, *J* = 13.6, 4.8 Hz, 1H), 2.32–2.39 (m, 2H), 2.39 (s, 3H), 2.42 (s, 3H), 2.79–2.83 (m, 1H), 2.85 (d, *J* = 6.4 Hz, 1H), 2.92 (d, *J* = 18.8 Hz, 1H), 3.15 (d, *J* = 11.2 Hz, 1H), 3.66 (dd, *J* = 11.2, 8.0 Hz, 1H), 3.93 (d, *J* = 11.2 Hz, 1H), 5.51 (d, *J* = 10.0 Hz, 1H), 6.62 (s, 1H), 6.78 (d, *J* = 10.0 Hz, 1H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.14 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.27 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.56 (d, *J* = 1.6 Hz, 1H), 7.57 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 21.6, 25.5, 26.8, 28.4, 32.5, 33.5, 41.8, 49.4, 114.4, 117.1, 122.0, 124.5, 126.0 (2C), 126.4, 127.2, 127.3 (2C), 129.8 (2C), 130.0 (2C), 131.4, 133.6, 136.4, 136.7, 136.9, 143.6, 145.0, 153.0, 194.3; IR (ATR) ν 1671, 1596, 1340, 1294, 1164, 1089, 932, 812, 735, 665 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{32}H_{19}ClN_2NaO_5S_2^+$ 643.1099 (M+Na$^+$) found 643.1090.

4. Synthesis of a fused-indoline derivative 4a using 3a



To a stirred solution of **3a** (29.3 mg, 0.05 mmol) in DMF (2.5 mL) at 0 °C was added TBAF (50 μL, 1.0 M in THF). After being stirred for 1 h at 0 °C, the reaction mixture was quenched with saturated aqueous NH_4Cl, and extracted twice with EtOAc. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography to give the desired product **4a** (24.6 mg, 84%) as white solid: melting point 109–111 °C; Rf 0.24 (*n*-hexane/EtOAc = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.73 (td, *J* = 13.2, 4.0 Hz, 1H), 1.87–1.95 (m, 2H), 2.25 (s, 3H), 2.31 (s, 3H), 2.55–2.64 (m, 2H), 2.89 (t, *J* = 13.2 Hz, 1H), 2.99 (dd, *J* = 9.6, 4.0 Hz, 1H), 3.56 (dd, *J* = 16.4, 4.0 Hz, 1H), 3.98 (d, *J* = 13.2 Hz, 1H), 5.14 (d, *J* = 16.4 Hz, 1H), 6.02 (d, *J* = 10.4 Hz, 1H), 6.75 (d, *J* = 10.4 Hz, 1H), 6.97 (d, *J* = 7.2 Hz, 1H), 7.02 (d, *J* = 8.0 Hz, 2H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.12 (t, *J* = 7.2 Hz, 1H), 7.28 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 7.2 Hz, 1H), 7.73 (d, *J* = 8.0 Hz, 2H), 7.73 (d, *J* = 7.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.4, 21.6, 35.0, 37.6, 42.3, 42.9, 49.9, 51.4, 51.7, 118.8, 123.6, 125.8, 126.1, 126.8 (2C), 127.0, 127.5 (2C), 128.4, 129.5 (2C), 129.7 (2C), 133.3, 134.1, 134.6, 142.2, 143.6, 144.6, 145.0, 145.0, 196.2; IR (ATR) ν 1677, 1456, 1347, 1157, 1088, 976, 933, 815, 715, 667 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{32}H_{30}N_2NaO_5S_2^+$ 609.1488 (M+Na$^+$) found 609.1500.

Structural determination of **4a** based on X-ray crystal structure analysis. (CCDC 1007338)

Crystal Data: Colorless prism crystal, $C_{32}H_{30}N_2O_5S_2$, *M* = 586.72, monoclinic, *a* = 10.7668(2), *b* = 17.8920(4), *c* = 14.4006(3) Å, *V* = 2772.38(9) Å^3, *T* = 93K, space group $P2_1/c$, *Z* = 4, 29210 reflections measured, 5055 unique (Rint = 0.0755). R1 = 0.0593, wR2 = 0.1480.

ORTEP of **4a**

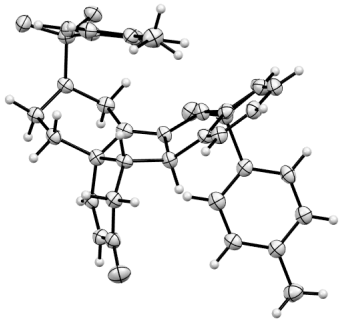
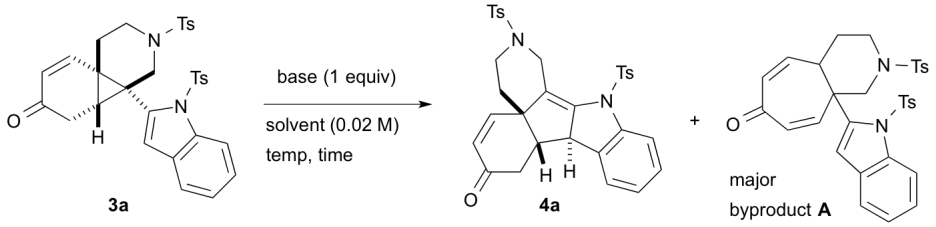
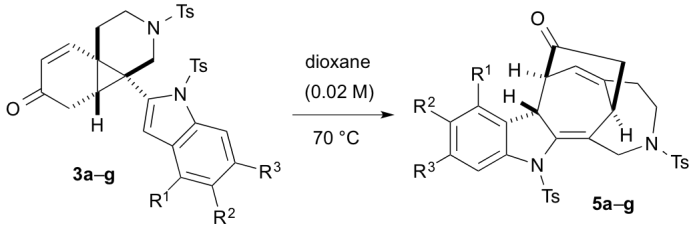


Table S-1. Optimization of the reaction conditions

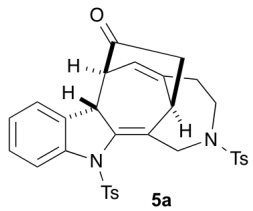


Entry	Base (1 equiv)	Solvent	Temp.	Time	Results
1	LHMDS	THF	–78 °C to rt	6 h	no reaction
2	KHMDS	THF	–78 °C to rt	20 h	**3a**: 28% recovery, Byproduct **A**: 34% yield
3	DBU	THF	0 °C to rt	20 h	no reaction
4	CsF	THF	0 °C to rt	20 h	no reaction
5	TBAF	THF	0 °C	20 h	**4a**: 61% yield, Byproduct **A**: 26% yield
6	TBAF	DME	0 °C	24 h	**3a** : **4a** : Byproduct **A** = 1.8 : 3.3 : 1 (1H NMR of the crude sample)
7	TBAF	CH_2Cl_2	0 °C to rt	4 h	no reaction
8	TBAF	CH_3CN	0 °C to rt	24 h	no reaction
9	**TBAF**	**DMF**	**0 °C**	**1 h**	**4a: 84% yield**
10	TBAF	DMSO	0 °C	1.5 h	**4a**: 71% yield

5. General procedure for the Cope rearrangement



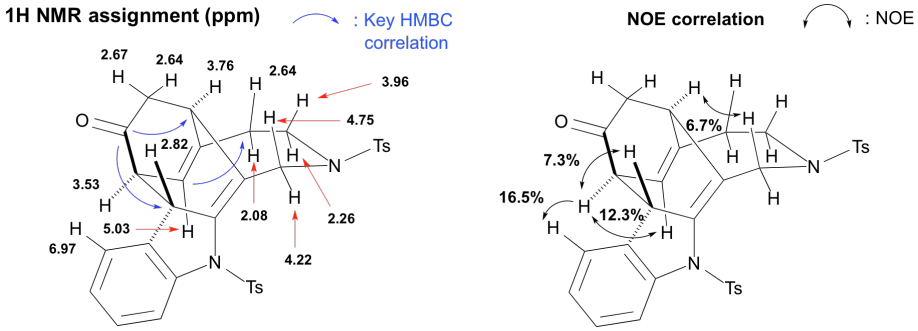
A solution of **3** (0.04 mmol) in 1,4-dioxane (2.0 mL) was heated to 70 °C and stirred for required time at the same temperature. After the consumption of **3** (monitored by TLC), the reaction mixture was cooled down to room temperature and concentrated *in vacuo*. The obtained residue was purified by silica gel column chromatography to give the desired products **5**.



5a

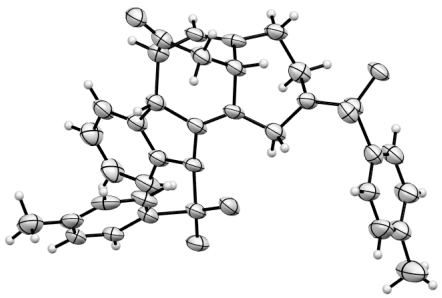
Compound 5a. White solid; melting point 124 °C; Rf 0.29 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.08 (d, *J* = 12.8 Hz, 1H), 2.26 (t, *J* = 12.8 Hz, 1H), 2.35 (s, 3H), 2.46 (s, 3H), 2.53–2.69 (m, 3H), 2.82 (br-s, 1H), 3.53 (d, *J* = 6.8 Hz, 1H), 3.76 (d, *J* = 6.8 Hz, 1H), 3.96 (br-d, *J* = 14.0 Hz, 1H), 4.22 (d, *J* = 15.2 Hz, 1H), 4.75 (dd, *J* = 15.2, 4.4 Hz, 1H), 5.02 (d, *J* = 6.4 Hz, 1H), 7.01 (d, *J* = 6.8 Hz, 1H), 7.08 (d, *J* = 7.6 Hz, 2H), 7.14 (t, *J* = 7.6 Hz, 1H), 7.23–7.26 (m, 1H), 7.26 (d, *J* = 7.6 Hz, 2H), 7.37 (d, *J* = 7.6 Hz, 2H), 7.61 (d, *J* = 8.4 Hz, 1H), 7.76 (d, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 21.6, 35.2, 37.5, 37.6, 42.7, 50.0, 50.2, 51.1, 113.6, 119.5, 122.5, 126.4, 126 7 .7 (2C), 127.4 (2C), 128.3, 129.5 (2C), 130.0 (2C), 132.1, 133.7, 134.9, 136.8, 137.8, 140.3, 143.4, 145.0, 153.6, 209.0; IR (ATR) ν 2924, 1727, 1360, 1162, 1092, 964, 880, 815, 726, 695, 675 cm^{-1}; HRMS (ESI^{+}) calcd for $C_{32}H_{30}N_2NaO_5S_2^{+}$ 609.1488 (M+Na^{+}) found 609.1516.

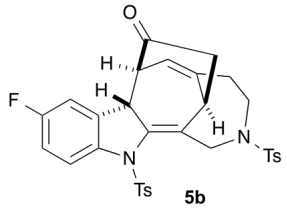
Structural determination of **5a** based on 2D-NMR and NOE experiments.



Although X-ray crystal structure analysis of **5a** was examined several times, we could not refine the structure completely because of the disordered solvent(s). However, the calculated X-ray structure except for the disordered solvent(s) shown below strongly supported our conclusion based on the NMR experiments.

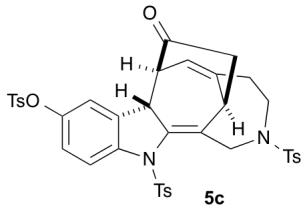
ORTEP of **5a**





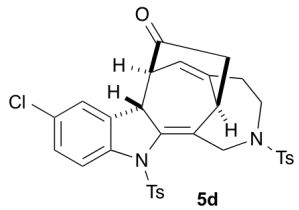
5b

Compound 5b. White solid; melting point >200 °C; Rf 0.50 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.10 (d, *J* = 12.8 Hz, 1H), 2.26 (t, *J* = 12.8 Hz, 1H), 2.37 (s, 3H), 2.46 (s, 3H), 2.53–2.67 (m, 3H), 2.78 (br-s, 1H), 3.44 (d, *J* = 6.8 Hz, 1H), 3.77 (d, *J* = 6.8 Hz, 1H), 3.97 (br-d, *J* = 14.4 Hz, 1H), 4.20 (d, *J* = 15.2 Hz, 1H), 4.75 (dd, *J* = 15.2, 3.2 Hz, 1H), 5.03 (d, *J* = 6.4 Hz, 1H), 6.72 (d, *J* = 7.6 Hz, 1H), 6.94 (t, *J* = 8.8 Hz, 1H), 7.12 (d, *J* = 8.0 Hz, 2H), 7.29 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.56 (dd, *J* = 8.8, 4.8 Hz, 1H), 7.76 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.6, 21.6, 35.1, 37.4, 37.5, 42.8, 50.0, 50.2, 51.1, 110.2 (d, *J* = 24.8 Hz), 113.3, 115.1 (d, *J* = 23.8 Hz), 120.8 (d, *J* = 8.5 Hz), 126.7 (2C), 127.4 (2C), 129.6 (2C), 130.0 (2C), 133.4, 134.5 (d, *J* = 7.6 Hz), 134.7, 136.2 (d, *J* = 2.9 Hz), 136.7, 138.4, 143.5, 145.3, 153.9, 161.1 (d, *J* = 246.0 Hz), 208.5; IR (ATR) ν 1725, 1474, 1339, 1159, 1092, 968, 877, 815, 723, 695, 671 cm^{-1}; HRMS (ESI^{+}) calcd for $C_{32}H_{29}FN_2NaO_5S_2^{+}$ 627.1394 (M+Na^{+}) found 627.1400.



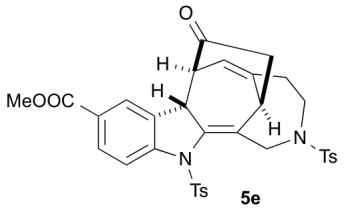
5c

Compound 5c. White solid; melting point 94 °C; Rf 0.24 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.09 (d, *J* = 13.2 Hz, 1H), 2.26 (t, *J* = 13.2 Hz, 1H), 2.38 (s, 3H), 2.46 (s, 3H), 2.48 (s, 3H), 2.51–2.67 (m, 3H), 2.72 (br-s, 1H), 3.31 (d, *J* = 7.2 Hz, 1H), 3.76 (d, *J* = 7.2 Hz, 1H), 3.98 (br-d, *J* = 13.6 Hz, 1H), 4.16 (d, *J* = 15.6 Hz, 1H), 4.74 (dd, *J* = 15.6, 4.0 Hz, 1H), 4.92 (d, *J* = 6.4 Hz, 1H), 6.63 (s, 1H), 6.91 (d, *J* = 8.8 Hz, 1H), 7.11 (d, *J* = 8.4 Hz, 2H), 7.24 ((d, *J* = 8.4 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.52 (d, *J* = 8.8 Hz, 1H), 7.68 (d, *J* = 8.0 Hz, 2H), 7.76 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 21.7, 21.8, 35.1, 37.3, 37.5, 42.7, 50.0, 50.2, 51.0, 113.2, 117.2, 120.3, 122.6, 126.7 (2C), 127.3 (2C), 128.5 (2C), 129.6 (2C), 129.8 (2C), 130.0 (2C), 132.1, 133.4, 134.0, 134.3, 136.6, 138.6, 139.0, 143.5, 145.4, 145.8, 147.6, 153.9, 208.3; IR (ATR) ν 1724, 1596, 1469, 1362, 1161, 1091, 968, 873, 813, 731, 696 cm^{-1}; HRMS (ESI^{+}) calcd for $C_{39}H_{36}N_2NaO_8S_3^{+}$ 779.1526 (M+Na^{+}) found 779.1500.



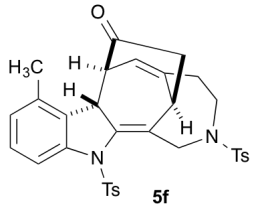
5d

Compound 5d. White solid; melting point 90 °C; Rf 0.33 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.10 (d, *J* = 13.6 Hz, 1H), 2.24 (td, *J* = 13.6, 2.4 Hz, 1H), 2.37 (s, 3H), 2.46 (s, 3H), 2.53–2.68 (m, 3H), 2.79 (br-s, 1H), 3.47 (d, *J* = 6.8 Hz, 1H), 3.77 (d, *J* = 6.8 Hz, 1H), 3.97 (br-d, *J* = 14.0 Hz, 1H), 4.18 (dd, *J* = 15.6, 0.8 Hz, 1H), 4.75 (dd, *J* = 15.6, 4.0 Hz, 1H), 5.05 (d, *J* = 6.8 Hz, 1H), 6.99 (t, *J* = 1.6 Hz, 1H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.23 (ddd, *J* = 8.8, 2.4, 0.8 Hz, 1H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 7.54 (d, *J* = 8.8 Hz, 1H), 7.76 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 21.6, 35.1, 37.4, 37.5, 42.7, 50.0, 50.2, 51.0, 113.4, 120.5, 123.0, 126.7 (2C), 127.3 (2C), 128.5, 129.7 (2C), 130.0 (2C), 132.0, 133.5, 134.1, 134.3, 136.7, 138.4, 138.9, 143.5, 145.4, 153.8, 208.4; IR (ATR) ν 1721, 1462, 1338, 1159, 1092, 965, 878, 815, 731, 691, 670 cm^{-1}; HRMS (ESI^{+}) calcd for $C_{32}H_{29}ClN_2NaO_5S_2^{+}$ 643.1099 (M+Na^{+}) found 643.1103.



5e

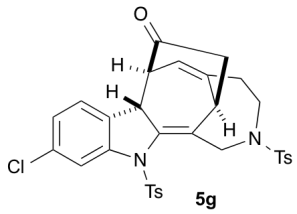
Compound 5e. White solid; melting point 129–130 °C; Rf 0.31 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.09 (d, *J* = 12.8 Hz, 1H), 2.21 (t, *J* = 12.8 Hz, 1H), 2.35 (s, 3H), 2.47 (s, 3H), 2.55–2.70 (m, 3H), 2.86 (br-s, 1H), 3.61 (d, *J* = 6.8 Hz, 1H), 3.78 (d, *J* = 6.8 Hz, 1H), 3.90 (s, 3H), 3.96 (br-d, *J* = 14.0 Hz, 1H), 4.17 (d, *J* = 15.2 Hz, 1H), 4.77 (dd, *J* = 15.2, 4.4 Hz, 1H), 5.05 (d, *J* = 6.6 Hz, 1H), 7.11 (d, *J* = 8.4 Hz, 2H), 7.30 (d, *J* = 8.4 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 7.67 (d, *J* = 8.4 Hz, 1H), 7.70 (s, 1H), 7.76 (d, *J* = 8.0 Hz, 2H), 7.96 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.6, 21.6, 35.1, 37.4, 37.6, 42.6, 50.0, 50.2, 50.9, 52.3, 113.6, 119.0, 124.0, 126.7 (2C), 127.2 (2C), 128.2, 129.7 (2C), 130.0 (2C), 130.4, 132.5, 133.6, 134.3, 136.7, 138.5, 143.5, 144.2, 145.4, 153.7, 166.1, 208.5; IR (ATR) ν 1719, 1439, 1363, 1291, 1162, 1093, 880, 828, 686, 670 cm^{-1}; HRMS (ESI^{+}) calcd for $C_{34}H_{32}N_2NaO_7S_2^{+}$ 667.1543 (M+Na^{+}) found 667.1523.



5f

Compound 5f. White solid; melting point 116–118 °C; Rf 0.54 (*n*-hexane/EtOAc = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.10 (d, *J* = 12.8 Hz, 1H), 2.26 (s, 3H), 2.29 (t, *J* = 12.8 Hz, 1H), 2.36 (s, 3H), 2.47 (s, 3H), 2.54 (dd, *J* = 19.2, 6.4 Hz, 1H), 2.62–2.70 (m, 2H), 2.95 (br-s, 1H), 3.77 (d, *J* = 6.8 Hz, 1H), 3.95–4.01 (m, 2H), 4.24 (d, *J* = 15.6 Hz, 1H), 4.78 (dd, *J* = 15.6, 3.6 Hz, 1H), 5.03 (d, *J* = 7.2 Hz, 1H), 6.89 (d, *J* = 8.0 Hz, 1H), 7.08 (d, *J* = 8.0 Hz, 2H), 7.14 (t, *J* = 8.0 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.77 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.0, 21.6, 21.6, 35.1, 37.1,

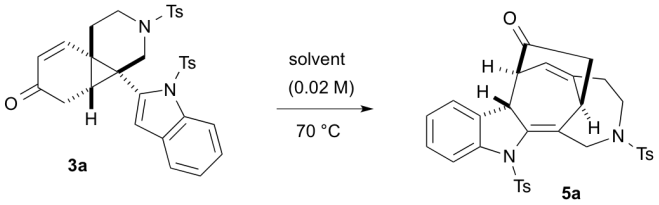
37.4, 43.8, 50.1, 50.2, 50.8, 114.0, 117.2, 126.7 (2C), 127.4 (2C), 128.1, 129.0, 129.3 (2C), 130.0 (2C), 130.3, 133.4, 133.9, 134.0, 136.8, 138.1, 140.4, 143.4, 144.9, 153.5, 209.4; IR (ATR) ν 1721, 1359, 1160, 1093, 965, 881, 815, 752, 723, 675 cm^{-1}; HRMS (ESI^+) calcd for $C_{33}H_{32}N_2NaO_5S_2^+$ 623.1645 ($M+Na^+$) found 623.1671.



5g

Compound 5g. White solid; melting point 148–149 °C; Rf 0.66 (*n*-hexane/EtOAc = 1/1); ^1H NMR (400 MHz, $CDCl_3$) δ 2.09 (d, *J* = 13.2 Hz, 1H), 2.24 (t, *J* = 13.2 Hz, 1H), 2.37 (s, 3H), 2.47 (s, 3H), 2.53–2.67 (m, 3H), 2.77 (br-s, 1H), 3.49 (d, *J* = 6.4 Hz, 1H), 3.77 (d, *J* = 6.4 Hz, 1H), 3.97 (br-d, *J* = 14.0 Hz, 1H), 4.16 (d, *J* = 15.6 Hz, 1H), 4.75 (dd, *J* = 15.6, 4.0 Hz, 1H), 5.01 (d, *J* = 6.4 Hz, 1H), 6.93 (d, *J* = 8.0 Hz, 1H), 7.11 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 7.63 (d, *J* = 1.6 Hz, 1H), 7.76 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.6, 21.7, 35.1, 37.4, 37.5, 42.4, 50.0, 50.2, 51.0, 113.4, 119.9, 123.4, 126.5, 126.7 (2C), 127.3 (2C), 129.7 (2C), 130.0 (2C), 130.6, 133.5, 133.9, 134.5, 136.6, 138.3, 141.4, 143.5, 145.4, 153.8, 208.7; IR (ATR) ν 1727, 1339, 1159, 1093, 964, 879, 749, 727, 694, 671 cm^{-1}; HRMS (ESI^+) calcd for $C_{32}H_{29}ClN_2NaO_5S_2^+$ 643.1099 ($M+Na^+$) found 643.1121.

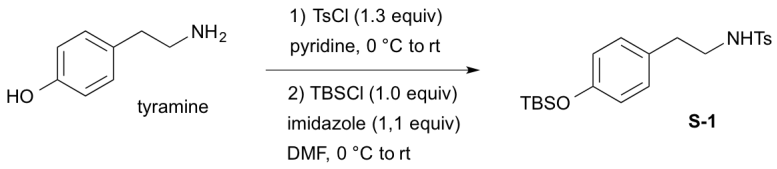
Table S-2. Optimization of the reaction conditions



Entry	Solvent	Time	Isolated Yield
1	$(CH_2Cl)_2$	5 h	42% yield
2	**1,4-dioxane**	**18 h**	**88% yield**
3	CH_3CN	7 h	37% yield
4	DMF	22 h	80% yield
5	AcOEt	6.5 h	51% yield
6	cyclopentyl methyl ether	22 h	69% yield
7	DME	7 h	64% yield

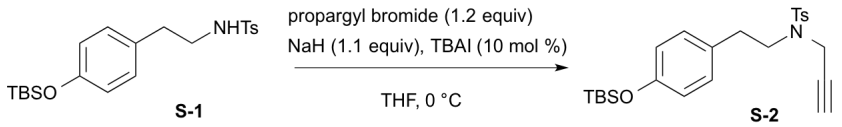
6. Substrate Syntheses and Compound Characterizations

Preparation of Compounds 1a-1g

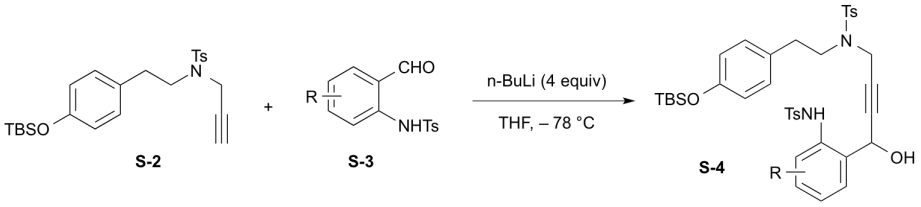


S-1

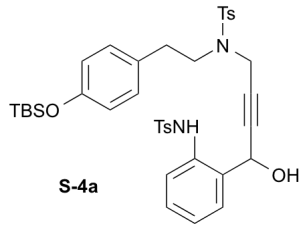
To a stirred solution of tyramine (8.22 g, 60 mmol) in pyridine (60 mL) at 0 °C was added TsCl (1.49 g, 78 mmol). After being stirred for 14 h at room temperature, the reaction mixture was diluted with EtOAc, and washed twice with aqueous 1 M $KHSO_4$ and brine. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The obtained sulfonamide was utilized for the next reaction without further purification. TBSCl (9.04 g, 60 mmol) was added to the solution of the obtained residue and imidazole (4.49 g, 66 mmol) in DMF (30 mL) at 0 °C. After being stirred for 2 h at room temperature, the reaction mixture was quenched with aqueous 1 M $KHSO_4$ at 0 °C, and extracted twice with Et_2O. The combined organic layers were washed with aqueous 1 M $KHSO_4$ and brine, dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAc = 6/1) to give the desired product **S-1** (10.3 g, 43% in 2 steps) as white solid: melting point 55 °C; Rf 0.46 (*n*-hexane/EtOAc = 3/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.18 (s, 6H), 0.98 (s, 9H), 2.43 (s, 3H), 2.68 (t, *J* = 7.2 Hz, 2H), 3.17 (q, *J* = 7.2 Hz, 2H), 4.37 (t, *J* = 7.2 Hz, 1H), 6.73 (d, *J* = 8.4 Hz, 2H), 6.92 (d, *J* = 8.4 Hz, 2H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 8.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ –4.4 (2C), 18.2, 21.5, 25.6 (3C), 34.9, 44.3, 120.3 (2C), 127.1 (2C), 129.6 (2C), 129.7 (2C), 130.1, 136.9, 143.4, 154.5; IR (ATR) ν 2929, 1608, 1509, 1324, 1253, 1156, 1093, 910, 837, 779, 662 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{21}H_{31}NNaO_3SSi^+$ 428.1686 (M+Na$^+$) found 428.1685.



To a stirred solution of **S-1** (1.0 g, 3.30 mmol) in THF (16 mL) at 0 °C was added NaH (60% in oil, 144 mg, 3.6 mmol). After being stirred for 30 min at 0 °C, propargyl bromide (0.40 mL, 3.90 mmol) and TBAI (121 mg, 0.33 mmol) were added to the reaction mixture at the same temperature. After being stirred for 1 h at 0 °C, the reaction mixture was quenched with saturated aqueous NH_4Cl at 0 °C, and extracted twice with EtOAc. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (*n*-hexane/EtOAc = 4/1) to give the desired product **S-2** (1.02 g, 92%) as yellow oil: Rf 0.39 (*n*-hexane/EtOAc = 6/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.18 (s, 6H), 0.98 (s, 9H), 2.05 (t, *J* = 2.4 Hz, 1H), 2.40 (s, 3H), 2.83 (t, *J* = 7.6 Hz, 2H), 3.38 (t, *J* = 7.6 Hz, 2H), 4.07 (d, *J* = 2.4 Hz, 2H), 6.75 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 7.27 (d, *J* = 8.0 Hz, 2H), 7.71 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ –4.5 (2C), 18.1, 21.5, 25.6 (3C), 34.0, 36.8, 48.1, 73.7, 76.7, 120.1 (2C), 127.6 (2C), 129.4 (2C), 129.7 (2C), 130.8, 135.9, 143.4, 154.3; IR (ATR) ν 2929, 1737, 1509, 1348, 1252, 1158, 1092, 908, 837, 779, 747 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{24}H_{33}NNaO_3SSi^+$ 466.1843 (M+Na$^+$) found 466.1847.

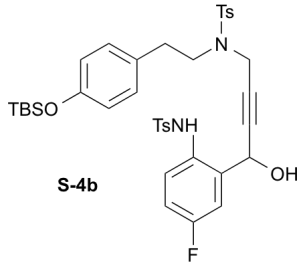


General Procedure: To a stirred solution of alkyne **S-2** (560 mg, 1.30 mmol) in THF (5 mL) at –78 °C was added *n*-BuLi (0.79 mL, 1.6 M in THF, 1.30 mmol). After being stirred for 30 min at –78 °C, a THF solution of aldehyde **S-3** (0.32 mmol in 5 mL of THF) was added to the reaction mixture at the same temperature. After being stirred for required time, the reaction mixture was quenched with saturated aqueous NH_4Cl, and extracted twice with EtOAc. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography to give the desired products **S-4**.



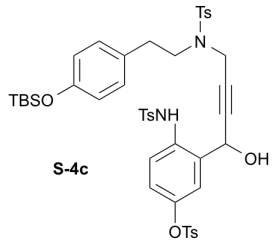
S-4a

Compound S-4a. 60% yield; White solid; melting point 36 °C; Rf 0.29 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.18 (s, 6H), 0.96 (s, 9H), 2.30 (s, 3H), 2.37 (s, 3H), 2.77 (br-s, 1H), 2.79 (t, *J* = 7.6 Hz, 2H), 3.37 (t, *J* = 7.6 Hz, 2H), 4.10 (s, 2H), 5.16 (s, 1H), 6.73 (d, *J* = 8.4 Hz, 2H), 7.00 (d, *J* = 8.4 Hz, 2H), 7.03 (t, *J* = 8.4 Hz, 1H), 7.16–7.28 (m, 7H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.66 (d, *J* = 8.4 Hz, 2H), 7.72 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ –4.5 (2C), 18.2, 21.4, 21.5, 25.6 (3C), 34.0, 37.2, 48.4, 62.4, 81.6, 83.1, 120.1 (2C), 123.0, 125.3, 127.1 (2C), 127.6 (2C), 128.1, 129.5 (2C), 129.6 (2C), 129.7 (2C), 129.7, 130.7, 130.9, 135.2, 135.8, 136.7, 143.7, 144.0, 154.3; IR (ATR) ν 2928, 1599, 1509, 1338, 1254, 1157, 1091, 913, 840, 781, 755, 661 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{38}H_{46}N_2NaO_6S_2Si^+$ 741.2459 (M+Na$^+$) found 741.2450.



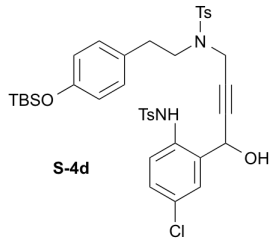
S-4b

Compound S-4b. 79% yield; White solid; melting point 46 °C; Rf 0.29 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.18 (s, 6H), 0.97 (s, 9H), 2.30 (s, 3H), 2.38 (s, 3H), 2.79 (t, *J* = 7.6 Hz, 2H), 3.03 (br-s, 1H), 3.37 (t, *J* = 7.6 Hz, 2H), 4.09 (s, 2H), 5.13 (s, 1H), 6.73 (d, *J* = 8.4 Hz, 2H), 6.88–6.94 (m, 2H), 7.01 (d, *J* = 8.4 Hz, 2H), 7.11 (dd, *J* = 8.4, 5.2 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.0 Hz, 2H), 7.44 (s, 1H), 7.61 (d, *J* = 8.0 Hz, 2H), 7.65 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ –4.5 (2C), 18.1, 21.4, 21.5, 25.6 (3C), 34.0, 37.1, 48.4, 61.4, 81.7, 82.6, 115.2 (d, *J* = 23.9 Hz), 116.1 (d, *J* = 21.9 Hz), 120.2 (2C), 126.6 (d, *J* = 8.6 Hz), 127.2 (2C), 127.6 (2C), 129.5 (2C), 129.7 (2C), 129.8 (2C), 130.5, 130.6, 135.4 (d, *J* = 6.6 Hz), 135.6, 136.2, 143.8, 144.2, 154.3, 160.3 (d, *J* = 245.1 Hz); IR (ATR) ν 2928, 1509, 1331, 1258, 1158, 1092, 912, 839, 668 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{38}H_{45}FN_2NaO_6S_2Si^+$ 759.2365 (M+Na$^+$) found 759.2361.



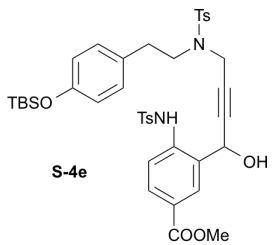
S-4c

Compound S-4c. 84% yield; White solid; melting point 100 °C; Rf 0.22 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.17 (s, 6H), 0.97 (s, 9H), 2.29 (s, 3H), 2.39 (s, 3H), 2.44 (s, 3H), 2.80 (t, *J* = 7.6 Hz, 2H), 3.39 (t, *J* = 7.6 Hz, 2H), 3.46 (br-s, 1H), 4.04 (s, 2H), 5.00 (s, 1H), 6.74 (d, *J* = 8.0 Hz, 2H), 6.75 (dd, *J* = 8.8, 2.8 Hz, 1H), 7.00 (s, 1H), 7.03 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.23 (d, *J* = 8.4 Hz, 2H), 7.23 (d, *J* = 8.4 Hz, 1H), 7.29 (d, *J* = 8.4 Hz, 2H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.65 (d, *J* = 8.4 Hz, 2H), 7.65 (d, *J* = 8.4 Hz, 2H), 7.79 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ –4.5 (2C), 18.2, 21.4, 21.5, 21.7, 25.6 (3C), 34.1, 37.1, 48.4, 62.1, 81.9, 82.5, 120.2 (2C), 122.3, 123.0, 123.6, 127.1 (2C), 127.7 (2C), 128.4 (2C), 129.5 (2C), 129.7 (2C), 129.8 (2C), 129.8 (2C), 130.7, 132.0, 132.1, 134.2, 135.8, 136.5, 143.8, 144.3, 145.7, 146.0, 154.3; IR (ATR) ν 1509, 1339, 1255, 1159, 1091, 912, 814, 662 cm^{-1}; HRMS (ESI^{+}) calcd for $C_{45}H_{52}N_2NaO_9S_3Si^{+}$ 911.2496 (M+Na^{+}) found 911.2505.



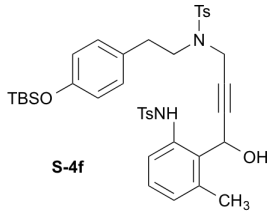
S-4d

Compound S-4d. 82% yield; Pale yellow solid; melting point 59 °C; Rf 0.34 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.18 (s, 6H), 0.97 (s, 9H), 2.27 (s, 3H), 2.38 (s, 3H), 2.80 (t, *J* = 7.6 Hz, 2H), 2.99 (br-s, 1H), 3.37 (t, *J* = 7.6 Hz, 2H), 4.08 (s, 2H), 5.07 (s, 1H), 6.74 (d, *J* = 8.4 Hz, 2H), 7.01 (d, *J* = 8.4 Hz, 2H), 7.15–7.19 (m, 4H), 7.20–7.26 (m, 3H), 7.64 (d, *J* = 7.6 Hz, 2H), 7.64 (d, *J* = 7.6 Hz, 2H), 7.74 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ –4.5 (2C), 18.1, 21.3, 21.5, 25.6 (3C), 34.0, 37.2, 48.4, 61.8, 82.1, 82.3 120.2 (2C), 124.4, 127.1 (2C), 127.6 (2C), 128.1, 129.4, 129.5 (2C), 129.7 (2C), 129.8 (2C), 130.6, 130.7, 132.7, 133.7, 135.6, 136.3, 143.8, 144.2, 154.3; IR (ATR) ν 2929, 1509, 1334, 1254, 1158, 1091, 912, 813, 781, 661 cm^{-1}; HRMS (ESI^{+}) calcd for $C_{38}H_{45}ClN_2NaO_6S_2Si^{+}$ 775.2069 (M+Na^{+}) found 775.2066.



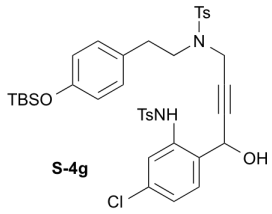
S-4e

Compound S-4e. 76% yield; White solid; melting point 56 °C; Rf 0.20 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.17 (s, 6H), 0.96 (s, 9H), 2.16 (s, 3H), 2.33 (s, 3H), 2.78 (t, *J* = 7.6 Hz, 2H), 3.37 (t, *J* = 7.6 Hz, 2H), 3.74 (s, 3H), 4.07 (s, 2H), 5.20 (s, 1H), 6.71 (d, *J* = 7.6 Hz, 2H), 6.99 (d, *J* = 7.6 Hz, 2H), 7.06 (d, *J* = 7.6 Hz, 2H), 7.22 (d, *J* = 7.6 Hz, 2H), 7.48 (d, *J* = 8.4 Hz, 1H), 7.59 (d, *J* = 7.6 Hz, 2H), 7.72 (s, 1H), 7.73 (d, *J* = 7.6 Hz, 2H), 7.84 (d, *J* = 8.4 Hz, 1H), 8.54 (br-s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ –4.6 (2C), 18.0, 21.1, 21.4, 25.5 (3C), 33.8, 37.0, 48.3, 51.9, 62.8, 81.8, 82.3, 119.3, 120.0 (2C), 125.0, 127.1 (2C), 127.5 (2C), 127.8, 129.3 (2C), 129.4, 129.6 (2C), 129.7 (2C), 130.5, 130.8, 135.3, 136.1, 140.1, 143.6, 144.3, 154.1, 166.0; IR (ATR) ν 2928, 1719, 1610, 1509, 1261, 1160, 1091, 914, 840, 661 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{40}H_{48}N_2NaO_8S_2Si^+$ 799.2514 (M+Na$^+$) found 799.2524.



S-4f

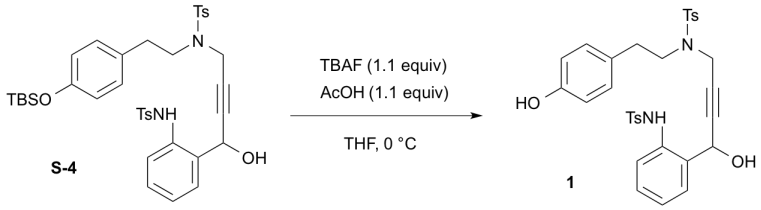
Compound S-4f. 90% yield; White solid; melting point 56–57 °C; Rf 0.29 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.17 (s, 6H), 0.97 (s, 9H), 2.15 (s, 3H), 2.34 (s, 3H), 2.36 (s, 3H), 2.71 (t, *J* = 7.2 Hz, 2H), 3.24–3.33 (m, 2H), 3.45 (br-s, 1H), 3.96 (d, *J* = 18.4 Hz, 1H), 4.95 (dd, *J* = 18.4, 2.0 Hz, 1H), 5.70 (s, 1H), 6.69 (d, *J* = 8.8 Hz, 2H), 6.84 (d, *J* = 7.6 Hz, 1H), 6.90 (d, *J* = 8.8 Hz, 2H), 7.04 (t, *J* = 7.6 Hz, 1H), 7.08 (d, *J* = 7.6 Hz, 1H), 7.14 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.59 (d, *J* = 8.4 Hz, 2H), 7.69 (d, *J* = 8.4 Hz, 2H), 8.33 (br-s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ –4.5 (2C), 18.1, 19.7, 21.4, 21.4, 25.6 (3C), 33.8, 37.1, 48.2, 59.7, 79.3, 83.6, 119.7, 120.0 (2C), 127.4 (2C), 127.4, 127.5 (2C), 128.6, 128.7, 129.4 (2C), 129.5 (2C), 129.7 (2C), 130.7, 135.5, 135.6, 136.5, 136.6, 143.5, 143.8, 154.1; IR (ATR) ν 1509, 1325, 1254, 1156, 1091, 908, 811, 780, 729, 660 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{39}H_{48}N_2NaO_6S_2Si^+$ 755.2615 (M+Na$^+$) found 755.2622.



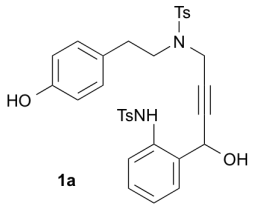
S-4g

Compound S-4g. 91% yield; White solid; melting point 56–57 °C; Rf 0.30 (*n*-hexane/EtOAc = 2/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.18 (s, 6H), 0.97 (s, 9H), 2.31 (s, 3H), 2.37 (s, 3H), 2.77 (t, *J* = 7.2 Hz, 2H), 3.02 (br-s, 1H), 3.34 (t, *J* = 7.2 Hz, 2H), 4.05 (d, *J* = 18.8 Hz, 1H), 4.11 (d, *J* = 18.8 Hz, 1H), 5.10 (s, 1H), 6.73 (d, *J* = 8.4 Hz, 2H), 6.94 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.97 (d, *J* = 8.4 Hz, 2H), 7.04 (d, *J* = 8.8 Hz, 1H), 7.16 (d, *J* = 8.4 Hz, 2H), 7.24 (d, *J* = 8.4 Hz, 2H), 7.35 (d, *J* = 2.4 Hz, 1H), 7.63 (d, *J* = 8.4 Hz, 2H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.96 (br-s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ –4.5 (2C), 18.1, 21.4, 21.5, 25.6 (3C), 33.9, 37.1, 48.4, 62.2, 82.0, 82.5, 120.1 (2C), 122.0, 124.7, 127.1 (2C), 127.6 (2C), 128.3, 129.1, 129.5 (2C),

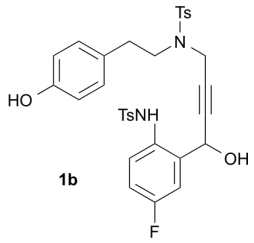
129.6 (2C), 129.8 (2C), 130.5, 135.1, 135.6, 136.3, 136.6, 143.7, 144.3, 154.3; IR (ATR) ν 1599, 1509, 1331, 1254, 1157, 1090, 907, 812, 730, 659 cm^{-1}; HRMS (ESI^+) calcd for $C_{38}H_{45}ClN_2NaO_6S_2Si^+$ 775.2069 ($M+Na^+$) found 775.2075.



General Procedure: To a stirred solution of **S-4** (0.24 mmol) and AcOH (15 μL, 0.27 mmol) in THF (6 mL) at 0 °C was added TBAF (0.27 mL, 1.0 M in THF). After being stirred for required time at 0 °C, the reaction mixture was quenched with saturated aqueous NH_4Cl, and extracted twice with EtOAc. The combined organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated *in vacuo*. The residue was purified by silica gel column chromatography to give the desired product **1**.

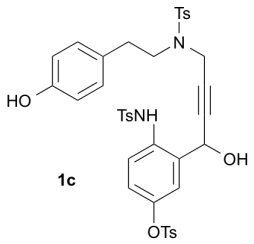


Compound 1a. 97% yield; White solid; melting point 42 °C; Rf 0.24 (*n*-hexane/EtOAc = 1/1); ^1H NMR (400 MHz, $CDCl_3$) δ 2.26 (s, 3H), 2.32 (s, 3H), 2.73 (t, *J* = 7.6 Hz, 2H), 3.32 (t, *J* = 7.6 Hz, 2H), 3.68 (br-s, 1H), 4.04 (s, 2H), 5.23 (s, 1H), 6.40 (br-s, 1H), 6.70 (d, *J* = 8.0 Hz, 2H), 6.92 (d, *J* = 8.0 Hz, 2H), 6.98–7.02 (m, 1H), 7.11–7.19 (m, 7H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.62 (d, *J* = 8.0 Hz, 2H), 7.91 (br-s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 21.4, 33.7, 37.1, 48.3, 62.1, 81.0, 83.3, 115.4 (2C), 123.0, 125.5, 127.1 (2C), 127.4 (2C), 128.2, 129.4, 129.5 (2C), 129.6, 129.7 (2C), 129.8 (2C), 131.4, 134.8, 135.2, 136.2, 143.8, 144.1, 154.5; IR (ATR) ν 3427, 1597, 1515, 1326, 1153, 1089, 915, 813, 735, 660 cm^{-1}; HRMS (ESI^+) calcd for $C_{32}H_{32}N_2NaO_6S_2^+$ 627.1594 ($M+Na^+$) found 627.1601.



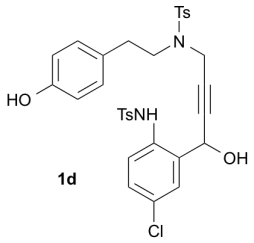
Compound 1b. 90% yield; White solid; melting point 48 °C; Rf 0.10 (*n*-hexane/EtOAc = 2/1); ^1H NMR (400 MHz, $CDCl_3$) δ 2.26 (s, 3H), 2.35 (s, 3H), 2.73 (t, *J* = 7.2 Hz, 2H), 3.32 (t, *J* = 7.2 Hz, 2H), 3.86 (br-s, 1H), 4.04 (s, 2H), 5.21 (s, 1H), 6.44 (br-s, 1H), 6.71 (d, *J* = 8.0 Hz, 2H), 6.84 (td, *J* = 8.0, 1.6 Hz, 1H),

6.90–6.95 (m, 1H), 6.93 (d, *J* = 8.0 Hz, 2H), 7.00 (dd, *J* = 8.0, 5.2 Hz, 1H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.61 (br-s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 21.5, 33.7, 37.1, 48.4, 61.1, 81.2, 82.9, 115.2 (d, *J* = 23.8 Hz), 115.5 (2C), 116.0 (d, *J* = 22.0 Hz), 126.7 (d, *J* = 8.6 Hz), 127.1 (2C), 127.4 (2C), 129.6 (2C), 129.7, 129.8 (2C), 129.8 (2C), 130.2 (d, *J* = 2.9 Hz), 135.1, 135.9, 135.9, 143.9, 144.3, 154.5, 160.3 (d, *J* = 245.0 Hz); IR (ATR) ν 3422, 1597, 1494, 1328, 1154, 1091, 913, 815, 661 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{32}H_{31}FN_2NaO_6S_2^+$ 645.1500 (M+Na$^+$) found 645.1515.



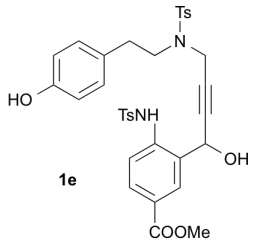
1c

Compound 1c. 91% yield; White solid; melting point 67 °C; Rf 0.12 (*n*-hexane/EtOAc = 1.5/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.25 (s, 3H), 2.34 (s, 3H), 2.39 (s, 3H), 2.74 (t, *J* = 7.2 Hz, 2H), 3.35 (t, *J* = 7.2 Hz, 2H), 3.98 (s, 2H), 5.08 (s, 1H), 6.71 (d, *J* = 8.4 Hz, 2H), 6.71 (d, *J* = 8.4 Hz, 1H), 6.94 (d, *J* = 8.4 Hz, 2H), 6.97 (s, 1H), 7.15 (d, *J* = 8.0 Hz, 2H), 7.15 (d, *J* = 8.4 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.60 (d, *J* = 8.0 Hz, 2H), 7.60 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 21.4, 21.6, 33.7, 37.0, 48.3, 61.7, 81.7, 82.3, 115.4 (2C), 122.3, 122.7, 123.5, 127.0 (2C), 127.4 (2C), 128.2 (2C), 129.6 (2C), 129.7, 129.7 (2C), 129.8 (2C), 129.8 (2C), 131.6, 132.5, 134.0, 135.1, 136.0, 143.9, 144.3, 145.8, 145.9, 154.5; IR (ATR) ν 3466, 1597, 1493, 1333, 1157, 1091, 913, 815, 738, 661 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{39}H_{38}N_2NaO_9S_3^+$ 797.1632 (M+Na$^+$) found 797.1631.

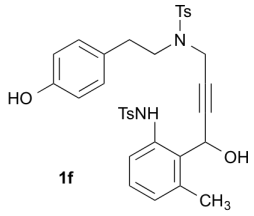


1d

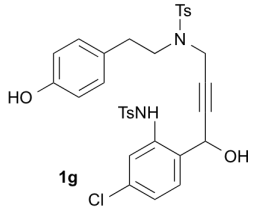
Compound 1d. 99% yield; White solid; melting point 60–62 °C; Rf 0.24 (*n*-hexane/EtOAc = 1.5/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.31 (s, 3H), 2.40 (s, 3H), 2.47 (br-s, 1H), 2.83 (t, *J* = 7.6 Hz, 2H), 3.40 (t, *J* = 7.6 Hz, 2H), 4.08 (s, 2H), 4.90 (s, 1H), 5.06 (s, 1H), 6.75 (d, *J* = 8.0 Hz, 2H), 7.05 (d, *J* = 8.0 Hz, 2H), 7.18–7.22 (m, 5H), 7.26 (d, *J* = 8.0 Hz, 2H), 7.56 (s, 1H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.67 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 21.5, 33.7, 37.2, 48.4, 61.6, 81.7, 82.6, 115.5 (2C), 124.5, 127.1 (2C), 127.5 (2C), 128.2, 129.3, 129.6 (2C), 129.7, 129.8 (2C), 129.8 (2C), 130.8, 133.2, 133.4, 135.1, 136.0, 143.9, 144.4, 154.4; IR (ATR) ν 3442, 1597, 1516, 1487, 1327, 1155, 1090, 814, 736, 661 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{32}H_{31}ClN_2NaO_6S_2^+$ 661.1204 (M+Na$^+$) found 661.1214.



Compound 1e. 84% yield; White solid; melting point 68–70 °C; Rf 0.52 (*n*-hexane/EtOAc = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.19 (s, 3H), 2.34 (s, 3H), 2.75 (t, *J* = 7.6 Hz, 2H), 3.36 (t, *J* = 7.6 Hz, 2H), 3.77 (s, 3H), 4.01 (s, 2H), 5.19 (s, 1H), 6.71 (d, *J* = 8.4 Hz, 2H), 6.95 (d, *J* = 8.4 Hz, 2H), 7.07 (d, *J* = 8.0 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 7.44 (d, *J* = 8.4 Hz, 1H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.0 Hz, 2H), 7.73 (s, 1H), 7.83 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.2, 21.5, 33.8, 37.2, 48.4, 52.2, 62.9, 81.9, 82.4, 115.4 (2C), 119.4, 125.0, 127.1 (2C), 127.5 (2C), 127.9, 129.5 (2C), 129.6, 129.7, 129.9 (2C), 129.9 (2C), 131.0, 135.1, 136.1, 140.2, 143.8, 144.5, 154.5, 166.3; IR (ATR) ν 1717, 1613, 1515, 1264, 1156, 1089, 914, 814, 732, 659 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{34}H_{34}N_2NaO_8S_2^+$ 685.1649 (M+Na$^+$) found 685.1688.



Compound 1f. 87% yield; White solid; melting point 79–80 °C; Rf 0.32 (*n*-hexane/EtOAc = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.11 (s, 3H), 2.30 (s, 3H), 2.32 (s, 3H), 2.67 (t, *J* = 7.2 Hz, 2H), 3.27 (t, *J* = 7.2 Hz, 2H), 3.86 (br-s, 1H), 3.92 (d, *J* = 18.8 Hz, 1H), 3.99 (d, *J* = 18.8 Hz, 1H), 5.70 (s, 1H), 6.38 (s, 1H), 6.70 (d, *J* = 8.4 Hz, 2H), 6.81 (d, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 8.4 Hz, 2H), 7.01 (t, *J* = 7.6 Hz, 1H), 7.04 (d, *J* = 7.6 Hz, 1H), 7.10 (d, *J* = 8.0 Hz, 2H), 7.16 (d, *J* = 8.0 Hz, 2H), 7.54 (d, *J* = 8.0 Hz, 2H), 7.66 (d, *J* = 8.0 Hz, 2H), 8.47 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.6, 21.4, 21.4, 33.6, 37.1, 48.2, 59.6, 79.2, 83.7, 115.4 (2C), 119.6, 127.3 (2C), 127.4 (2C), 127.4, 128.6, 126.7, 129.5 (2C), 129.6 (2C), 129.7, 129.8 (2C), 135.1, 135.4, 136.2, 136.6, 143.6, 144.0, 154.4; IR (ATR) ν 3432, 1597, 1515, 1323, 1154, 1090, 974, 813, 735, 659 cm^{-1}; HRMS (ESI$^+$) calcd for $C_{33}H_{34}N_2NaO_6S_2^+$ 641.1750 (M+Na$^+$) found 641.1772.



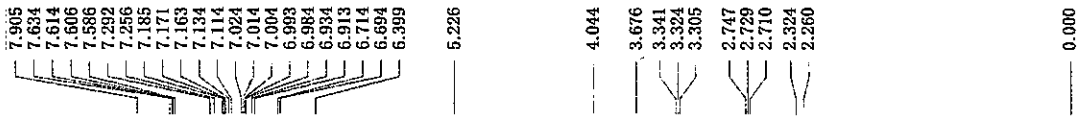
Compound 1g. 95% yield; White solid; melting point 71–72 °C; Rf 0.32 (*n*-hexane/EtOAc = 1/1); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.27 (s, 3H), 2.33 (s, 3H), 2.73 (t, *J* = 6.8 Hz, 2H), 3.32 (t, *J* = 6.8 Hz, 2H), 3.76 (br-s,

1H), 4.00 (d, J = 20.0 Hz, 1H), 4.05 (d, J = 20.0 Hz, 1H), 5.14 (s, 1H), 6.36 (br-s, 1H), 6.71 (d, J = 8.8 Hz, 2H), 6.89–6.95 (m, 3H), 7.01 (d, J = 8.4 Hz, 1H), 7.12 (d, J = 7.6 Hz, 2H), 7.20 (d, J = 7.6 Hz, 2H), 7.26 (br-s, 1H), 7.57 (d, J = 7.6 Hz, 2H), 7.64 (d, J = 7.6 Hz, 2H), 8.08 (br-d, J = 12.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 21.4, 33.6, 37.0, 48.3, 61.9, 81.5, 82.7, 115.4 (2C), 122.0, 124.9, 127.0 (2C), 127.4 (2C), 128.8, 129.2, 129.5 (2C), 129.6, 129.8 (2C), 129.8 (2C), 134.8, 135.0, 135.8, 136.2, 143.9, 144.4, 154.4; IR (ATR) ν 1598, 1515, 1327, 1264, 1154, 1089, 938, 813, 733, 658 cm^{-1}; HRMS (ESI^+) calcd for $C_{32}H_{31}ClN_2NaO_6S_2^+$ 661.1204 ($M+Na^+$) found 661.1235.

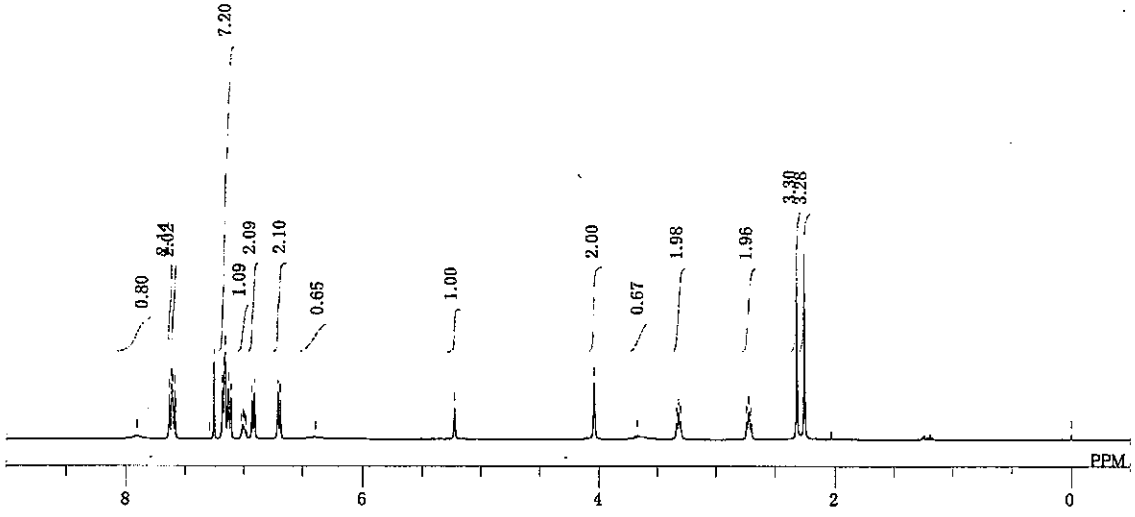
7. ^{1}H and ^{13}C NMR Charts of New Compounds (page 20–53)

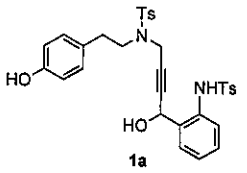
single_pulse

G:¥NMR¥横坂¥34¥YKTY34-53-Fr8-15_proton-2-1.als



DFILE	YKTY34-53-Fr8-15_proton-2-1.
COMNT	single_pulse
DATIM	2013-08-16 15:53:32
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.40 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	23.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	28

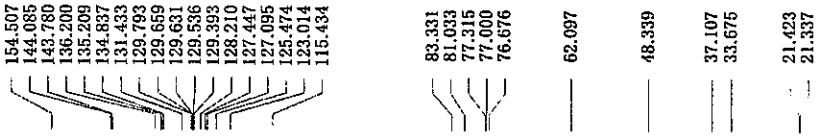




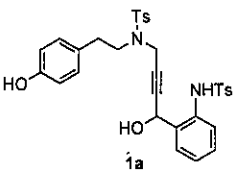
1a

single pulse decoupled gated NOE

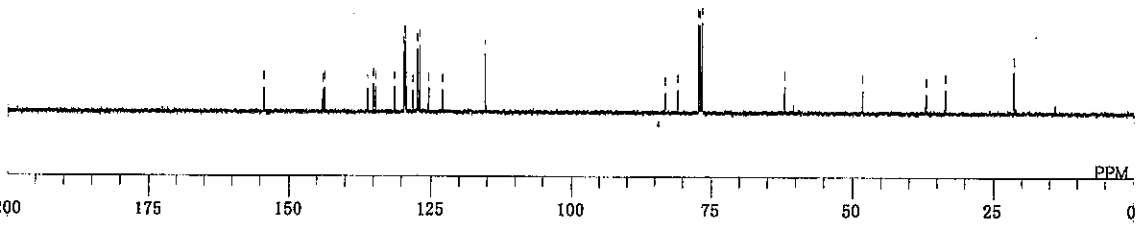
G:¥NMR¥横坂¥34¥YKTY34-53-Fr8-15_carbon-1-1.jdf



DFILE	YKTY34-53-Fr8-15_carbon-1-1
COMNT	single pulse decoupled gated NO
DATIM	2013-08-16 14:51:50
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	27
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

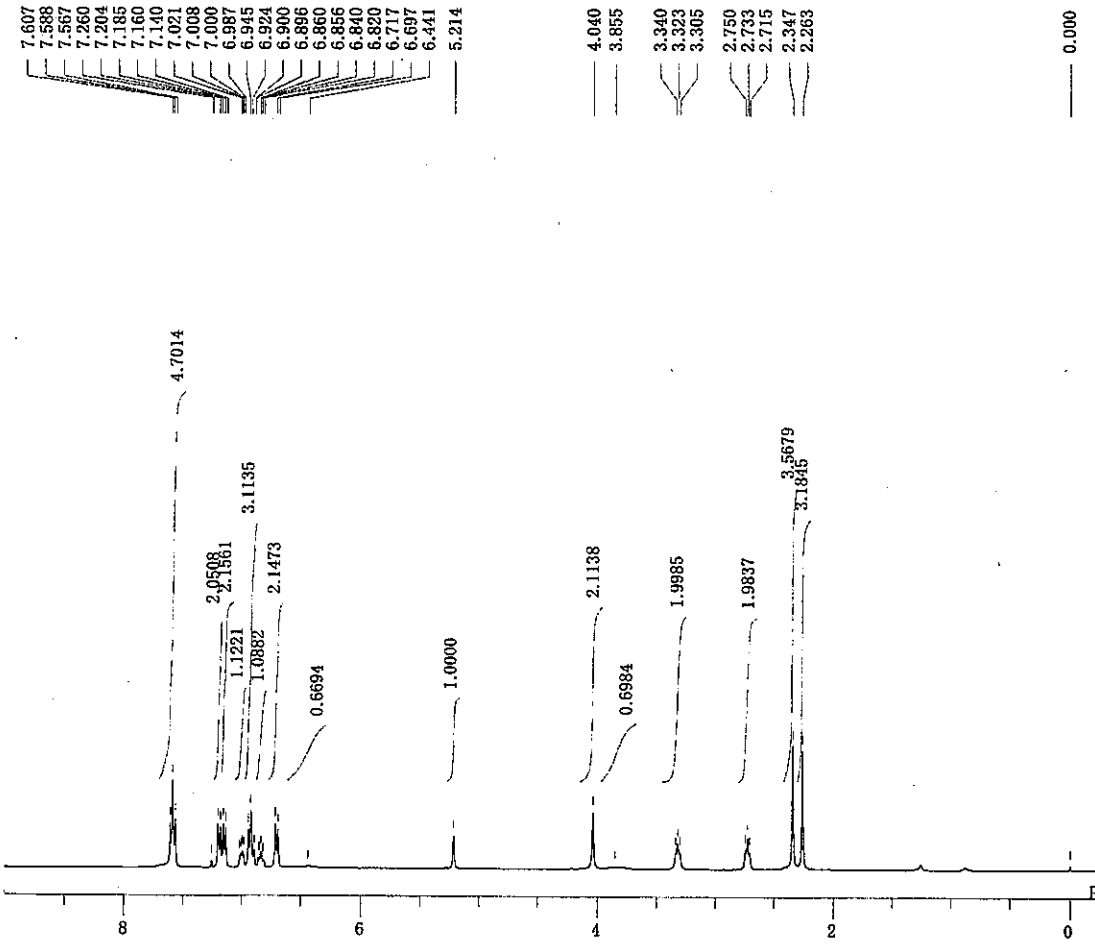


1a

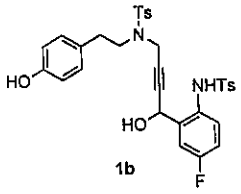


single_pulse

G:¥NMR¥横坂¥35¥YKTY35-28-Fr17-25_proton-2-1.jdf

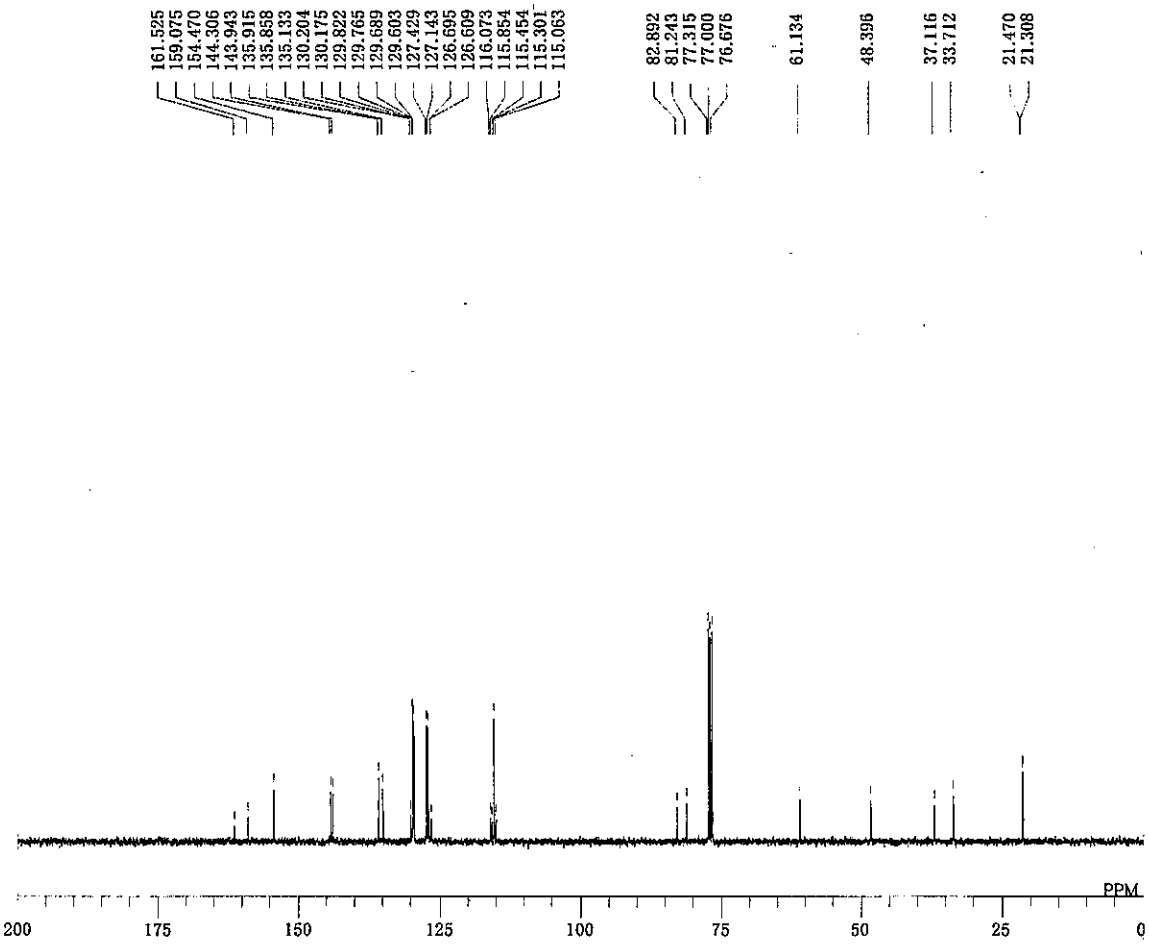


DFILE	YKTY35-28-Fr17-25_proton-2-
COMNT	single_pulse
DATIM	2013-08-26 18:52:11
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	28

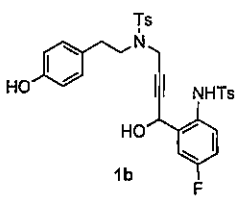


single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-28-Fr17-25_carbon-2-1.als

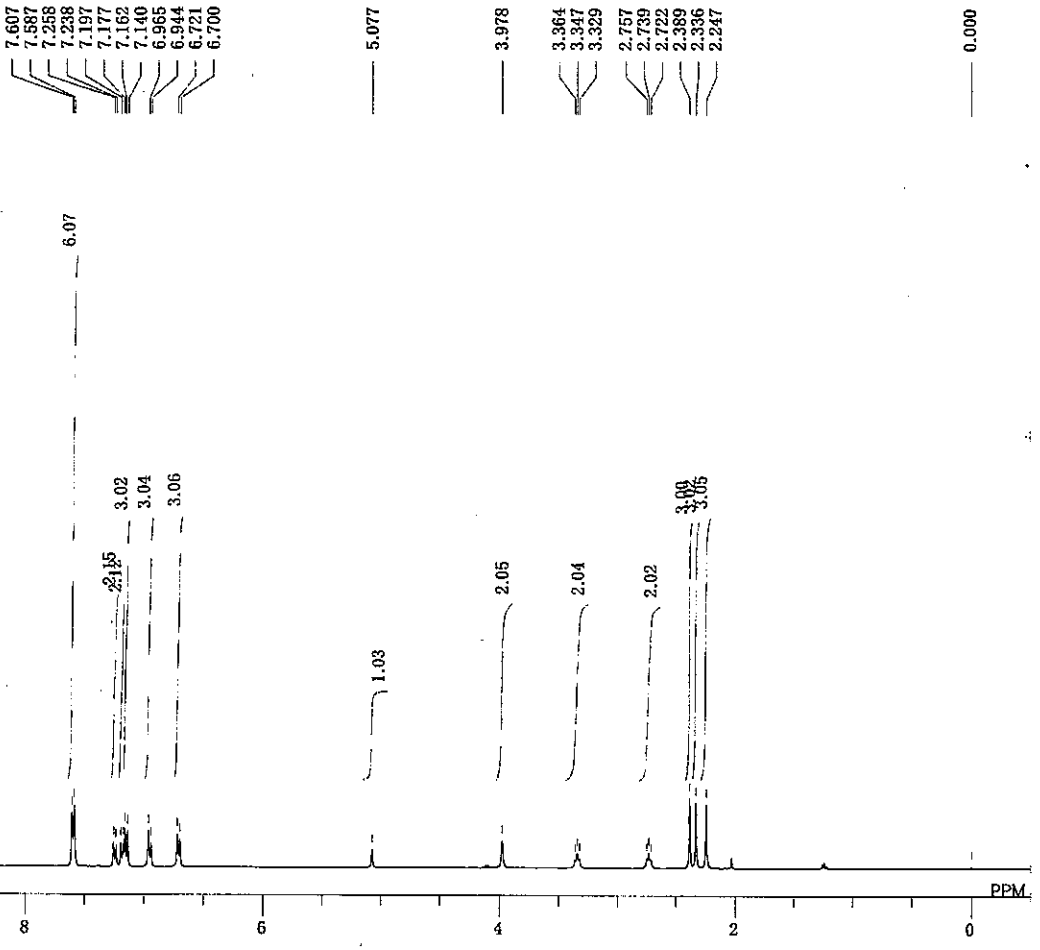


DFILE	YKTY35-28-Fr17-25_carbon-2-
COMNT	single pulse decoupled gated NOI
DATIM	2013-08-26 18:55:30
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.63 Hz
SCANS	79
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

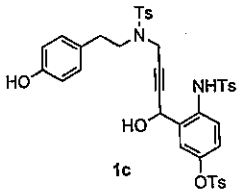


single_pulse

G:¥NMR¥横坂¥35¥YKTY35-61-Fr14-29_proton-1-1.jdf



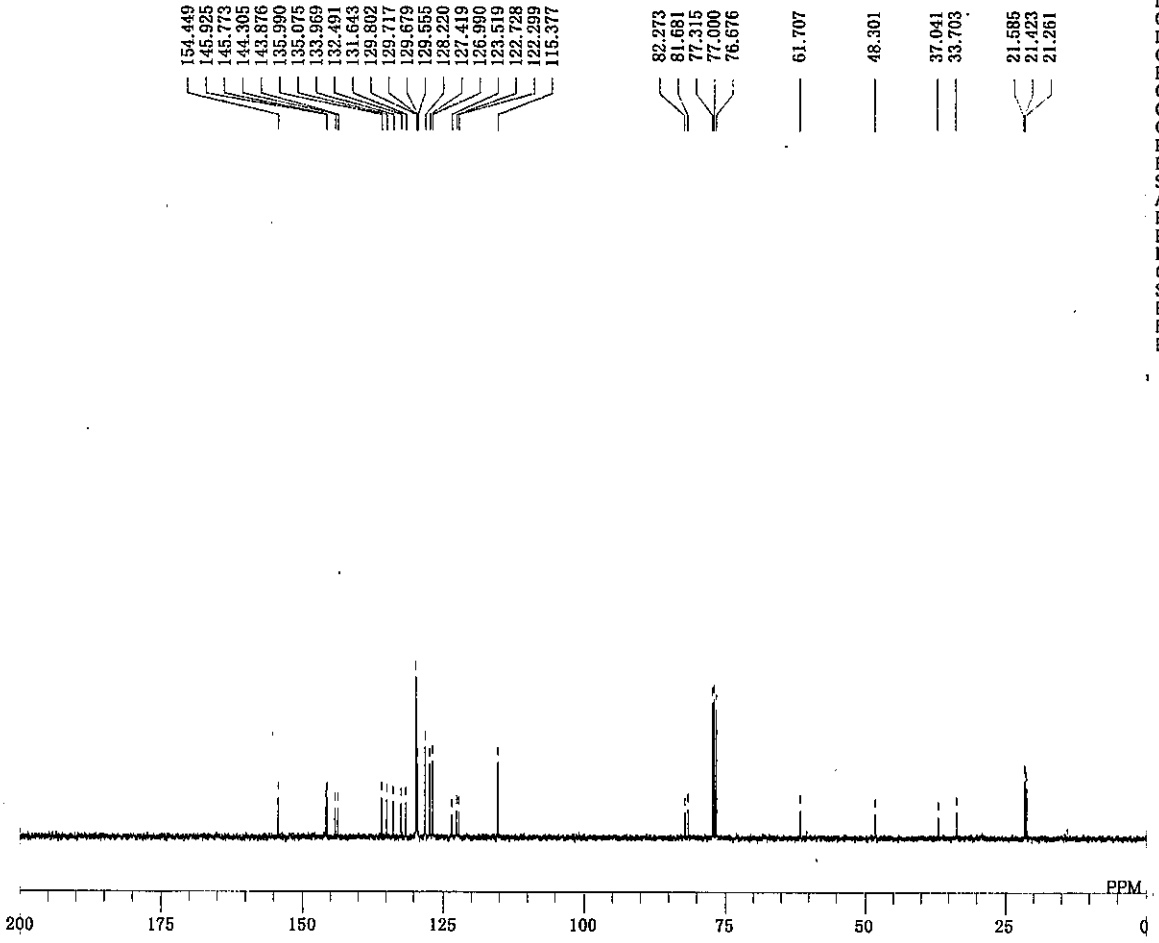
DFILE	YKTY35-61-Fr14-29_proton-1-1
COMNT	single_pulse
DATIM	2013-09-12 23:43:54
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	24



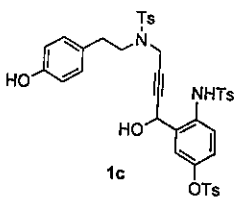
1c

single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-61-Fr14-29_carbon-1-1.jdf



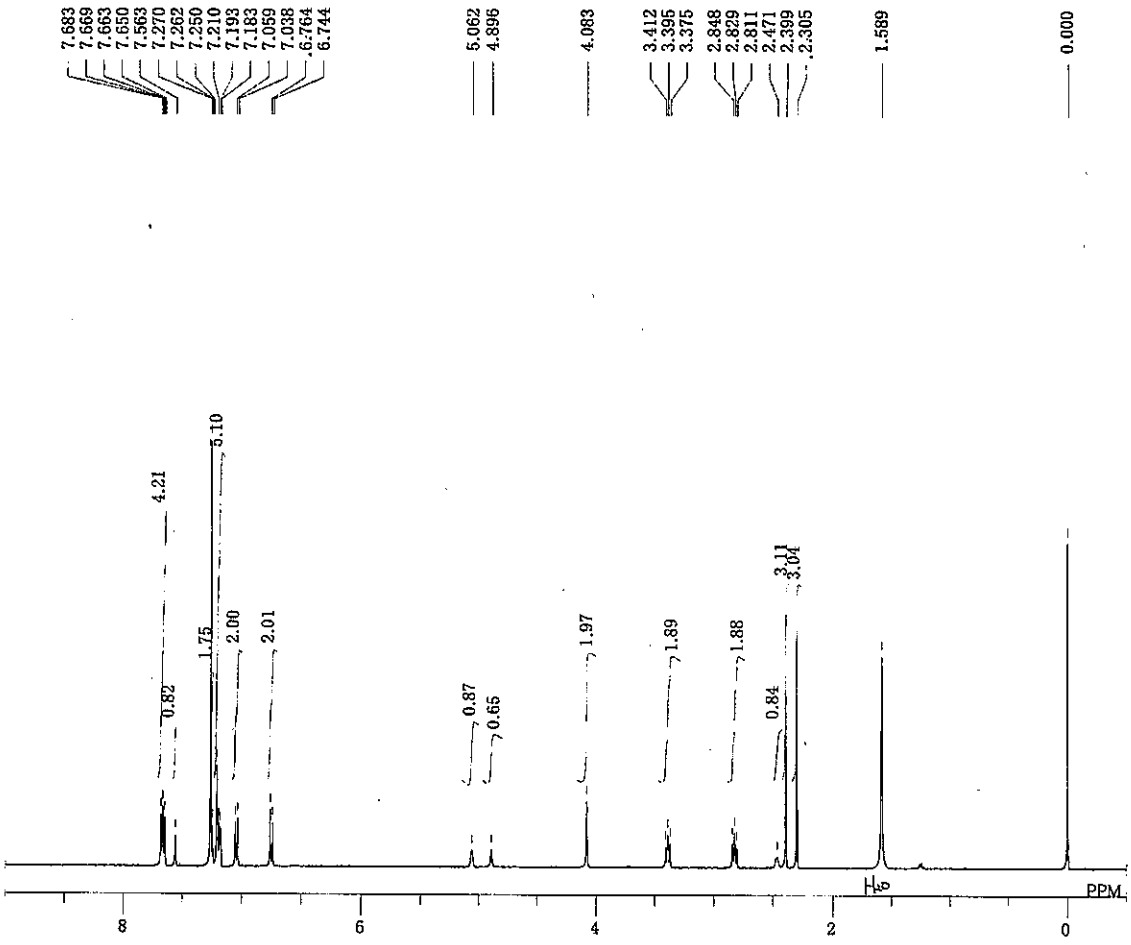
DFILE	YKTY35-61-Fr14-29_carbon-1-1
COMNT	single pulse decoupled gated NOE
DATIM	2013-09-12 23:46:54
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	30
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	20.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



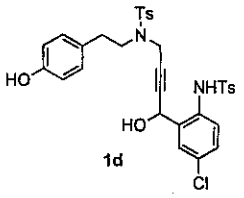
1c

single_pulse

G:¥NMR¥横坂¥35¥YKTY35-80-Fr8-15_proton-2-1.jdf



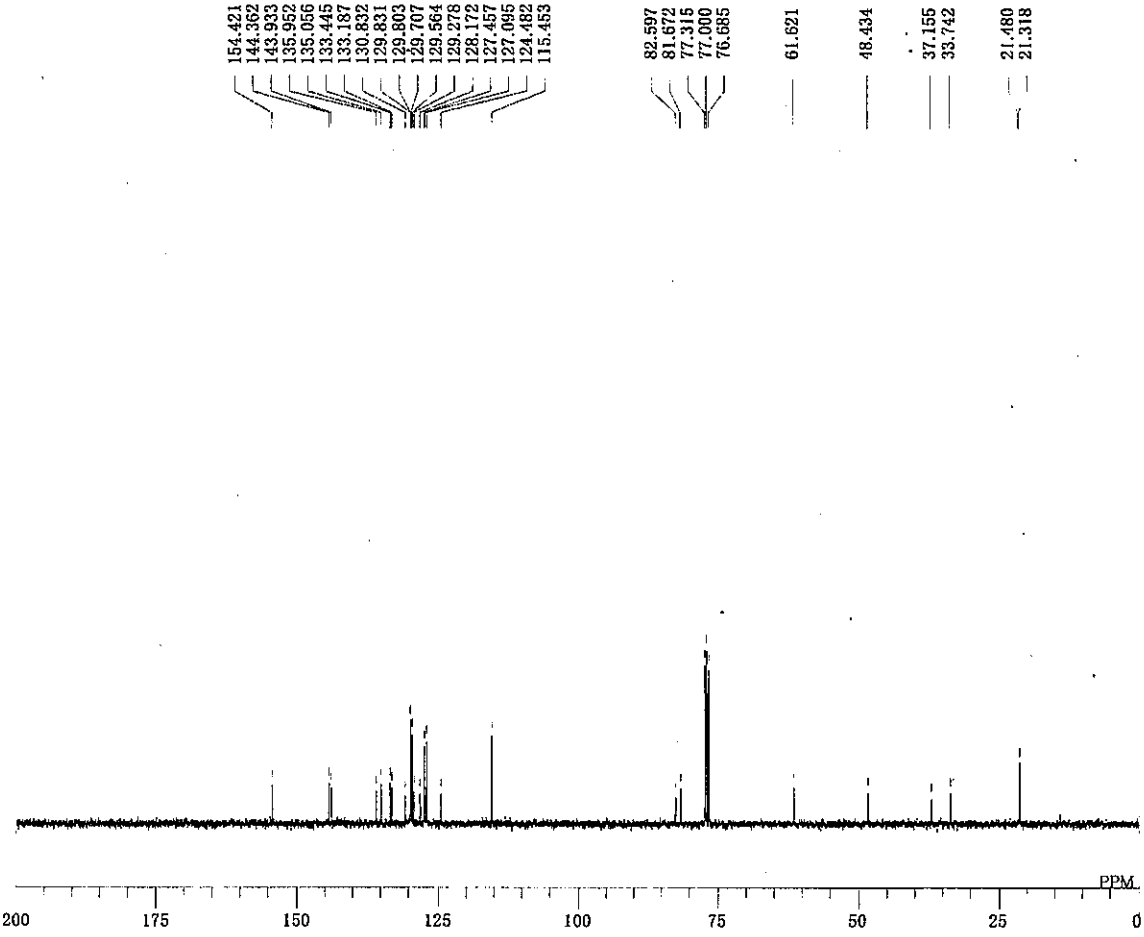
DFILE	YKTY35-80-Fr8-15_proton-2-1.
COMNT	single_pulse
DATIM	2013-09-25 12:37:19
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	50



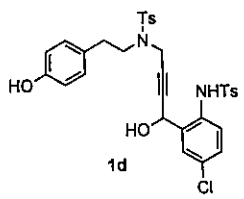
1d

single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-80-Fr8-15_carbon-1-1.jdf



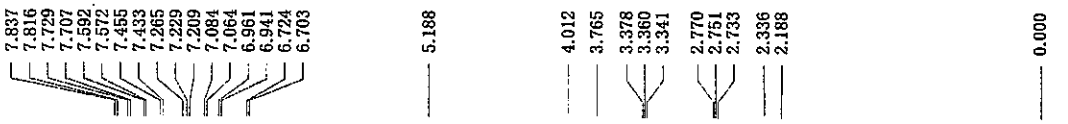
DFILE	YKTY35-80-Fr8-15_carbon-1-1.
COMNT	single pulse decoupled gated NOI
DATIM	2013-09-25 11:35:56
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	24
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



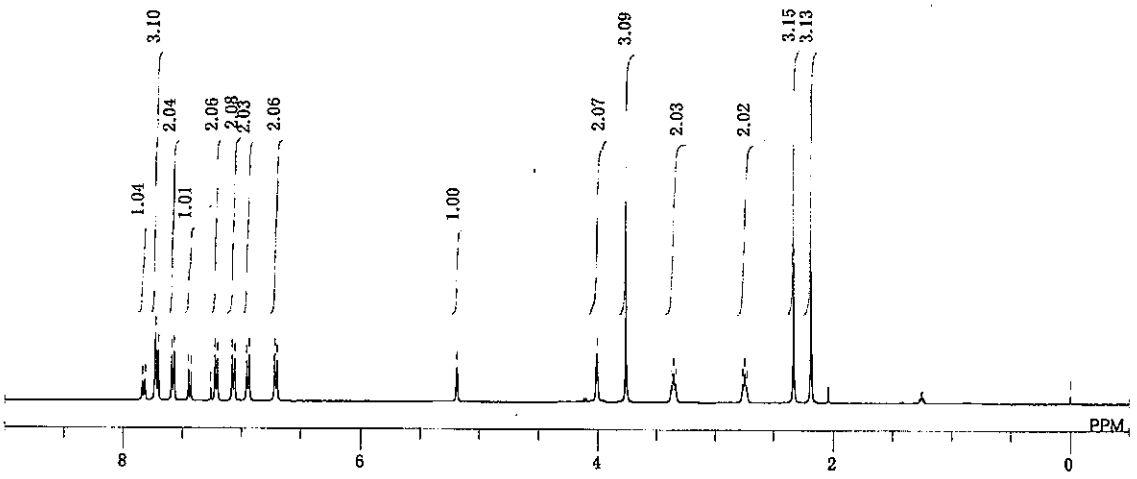
1d

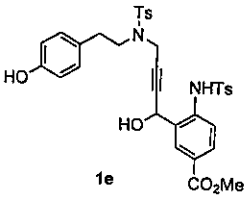
single_pulse

G:¥NMR¥横坂¥35¥YKTY35-71-Fr37-45_proton-1-1.jdf



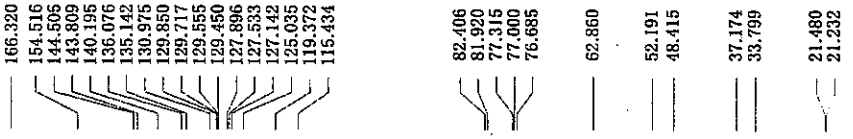
DFILE	YKTY35-71-Fr37-45_proton-1-1
COMNT	single_pulse
DATIM	2013-09-19 23:20:02
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30



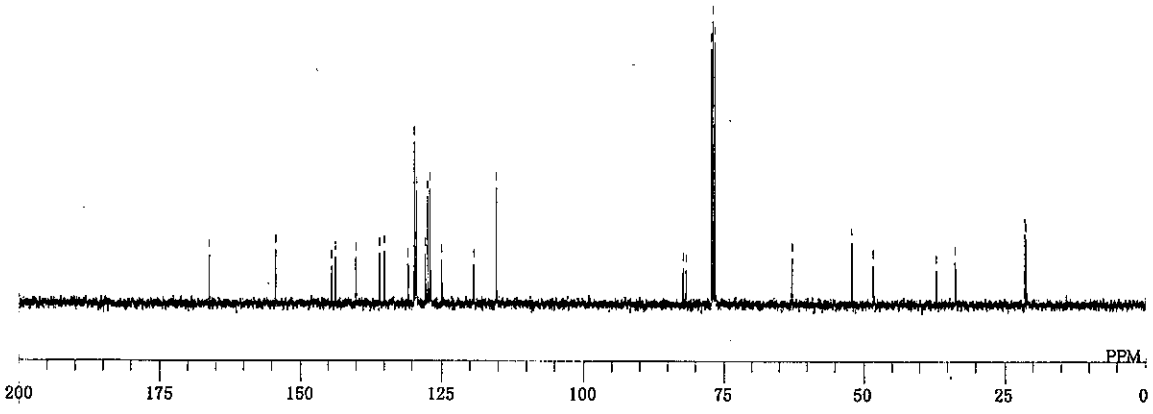


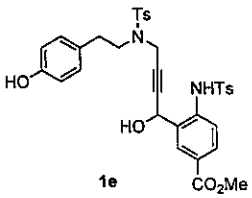
single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-71-Fr37-45_carbon-1-1.jdf



DFILE	YKTY35-71-Fr37-45_carbon-1-1
COMNT	single pulse decoupled gated NOE
DATIM	2013-09-19 23:23:05
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	48
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	19.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

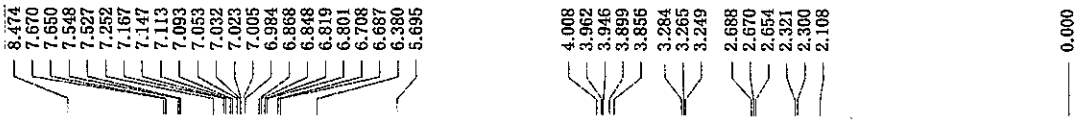




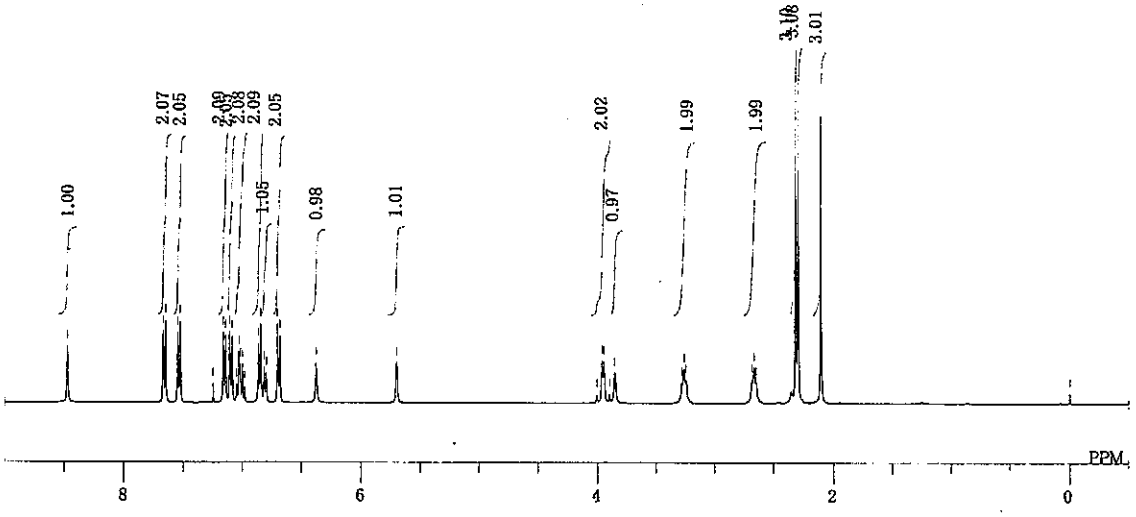
2X

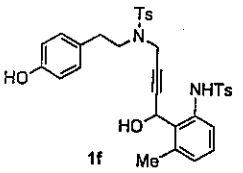
yk-hn-07-51_

G:¥NMR¥中山¥07¥yk-hn-07-51_proton-1-1.jdf



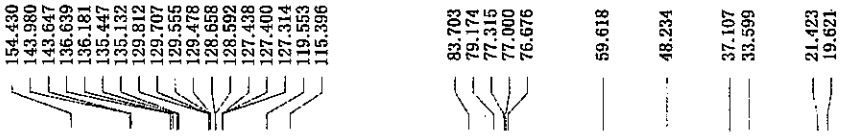
DFILE	yk-hn-07-51_proton-1-1.jdf
COMNT	yk-hn-07-51_
DATIM	2013-10-08 15:28:49
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	26



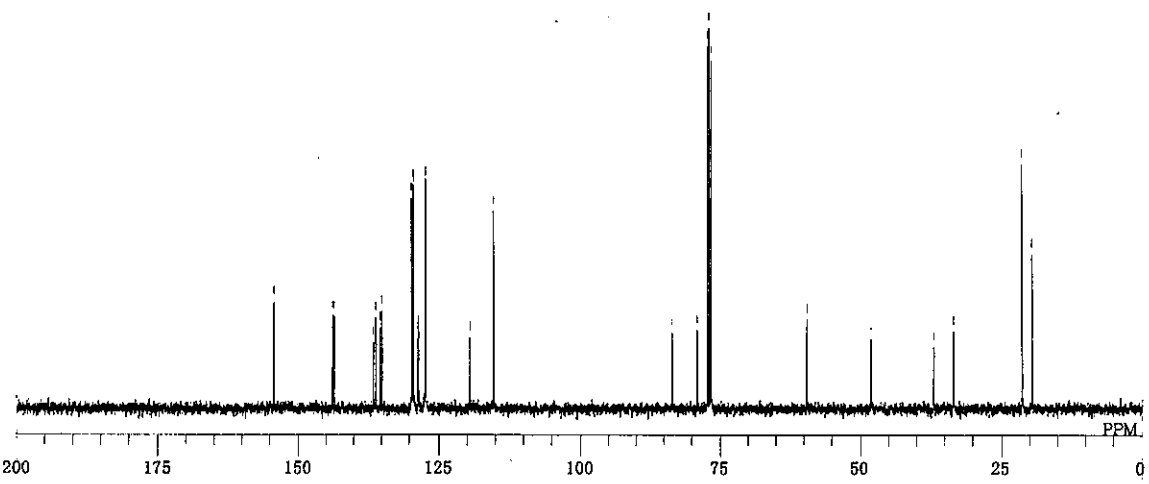


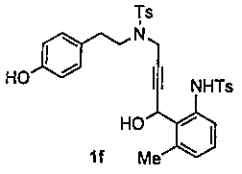
yk-hn-07-51_yk-hn-07-51_proton

G:¥NMR¥中山¥07¥yk-hn-07-51_carbon-1-1.jdf



DFILE	yk-hn-07-51_carbon-1-1.jdf
COMNT	yk-hn-07-51_yk-hn-07-51_proto
DATIM	2013-10-08 15:30:15
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	61
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60





yk-hn-07-50_

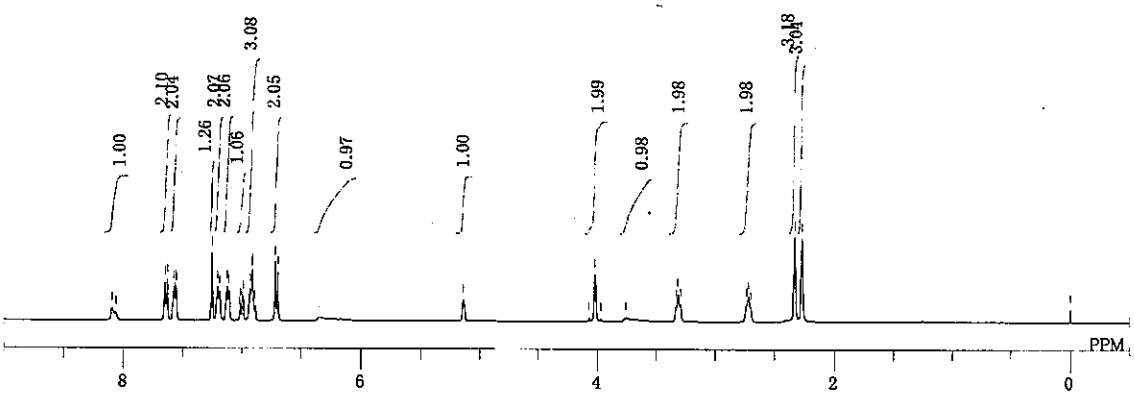
G:¥NMR¥中山¥07¥yk-hn-07-50-2_proton-1-1.als

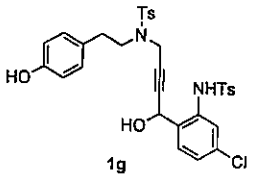
8.096 8.064 7.652 7.634 7.576 7.568 7.560 7.557 7.258 7.211 7.194 7.132 7.115 7.024 7.015 7.004 6.994 6.935 6.926 6.919 6.914 6.898 6.894 6.723 6.701 6.355 5.141

4.073 4.023 3.974 3.763 3.333 3.317 3.298 2.740 2.725 2.708 2.333 2.272

0.000

DFILE	yk-hn-07-50-2_proton-1-1.als
COMNT	yk-hn-07-50_
DATIM	2013-10-08 15:22:47
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.40 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	28





yk-hn-07-50_yk-hn-07-50_proton

G:¥NMR¥中山¥07¥yk-hn-07-50_carbon-1-1.jdf

154.421 144.429 143.895 136.238 135.819 135.018 134.827 129.774 129.583 129.536 129.164 128.763 127.381 127.009 124.883 121.965 115.415

82.721 81.462 77.315 77.000 76.685

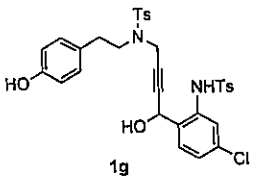
61.859

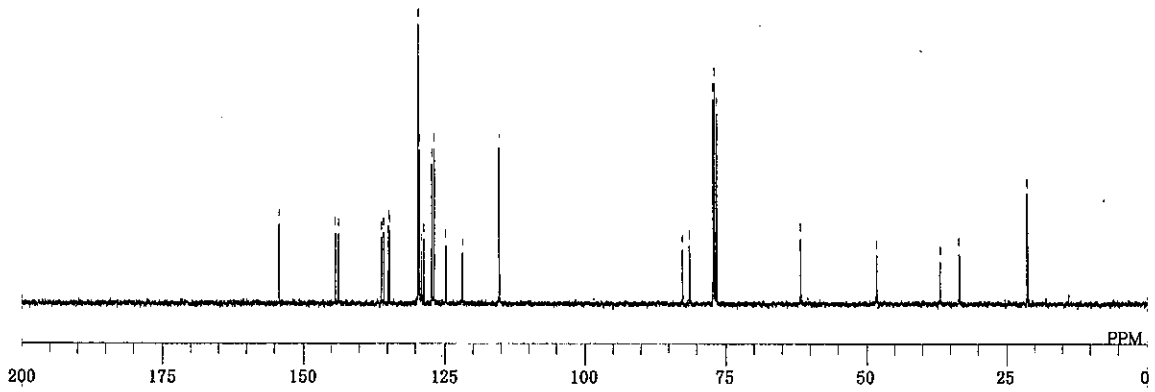
48.330

37.031 33.608

21.423 21.309

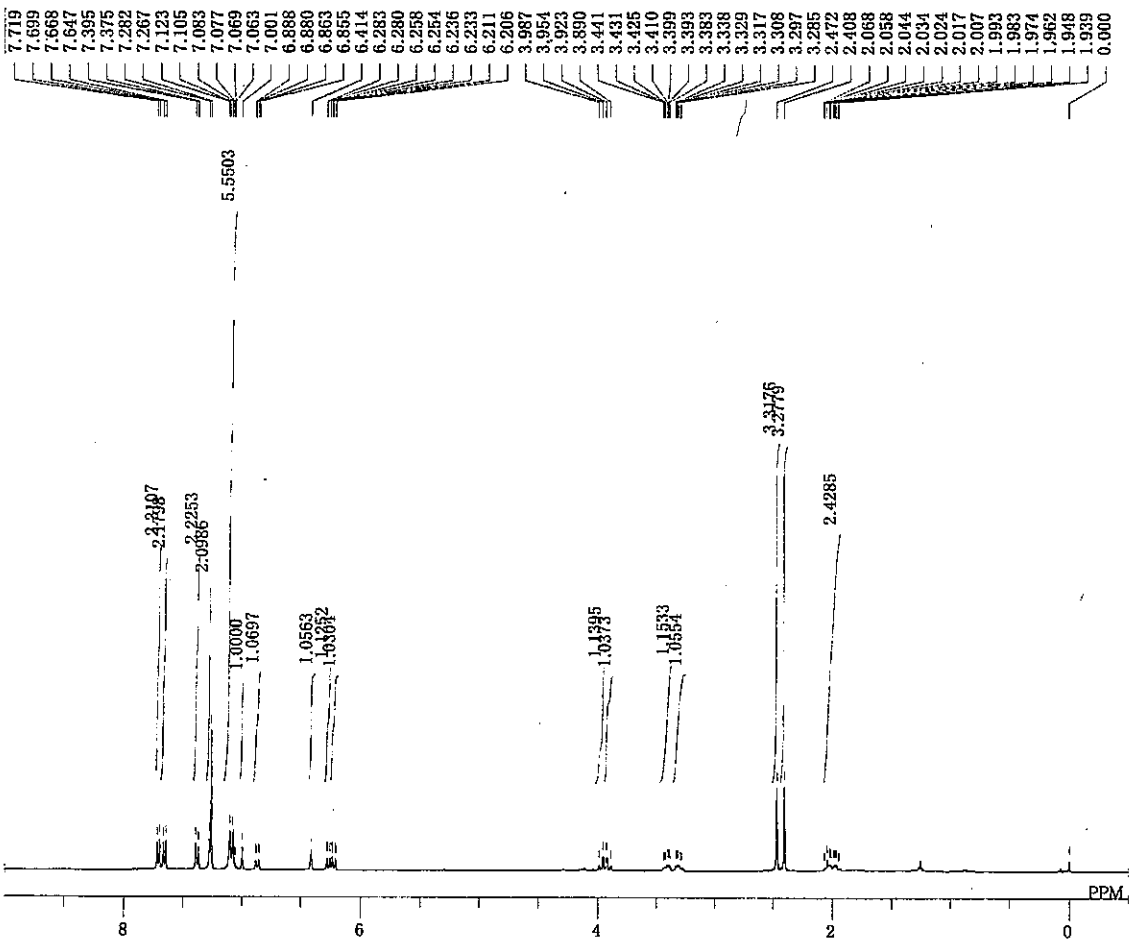
DFILE	yk-hn-07-50_carbon-1-1.jdf
COMNT	yk-hn-07-50_yk-hn-07-50_proto
DATIM	2013-10-08 12:08:49
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	84
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



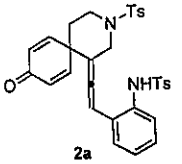


single_pulse

G:¥NMR¥横坂¥34¥YKTY34-33-Fr14-27_proton-3-1.als



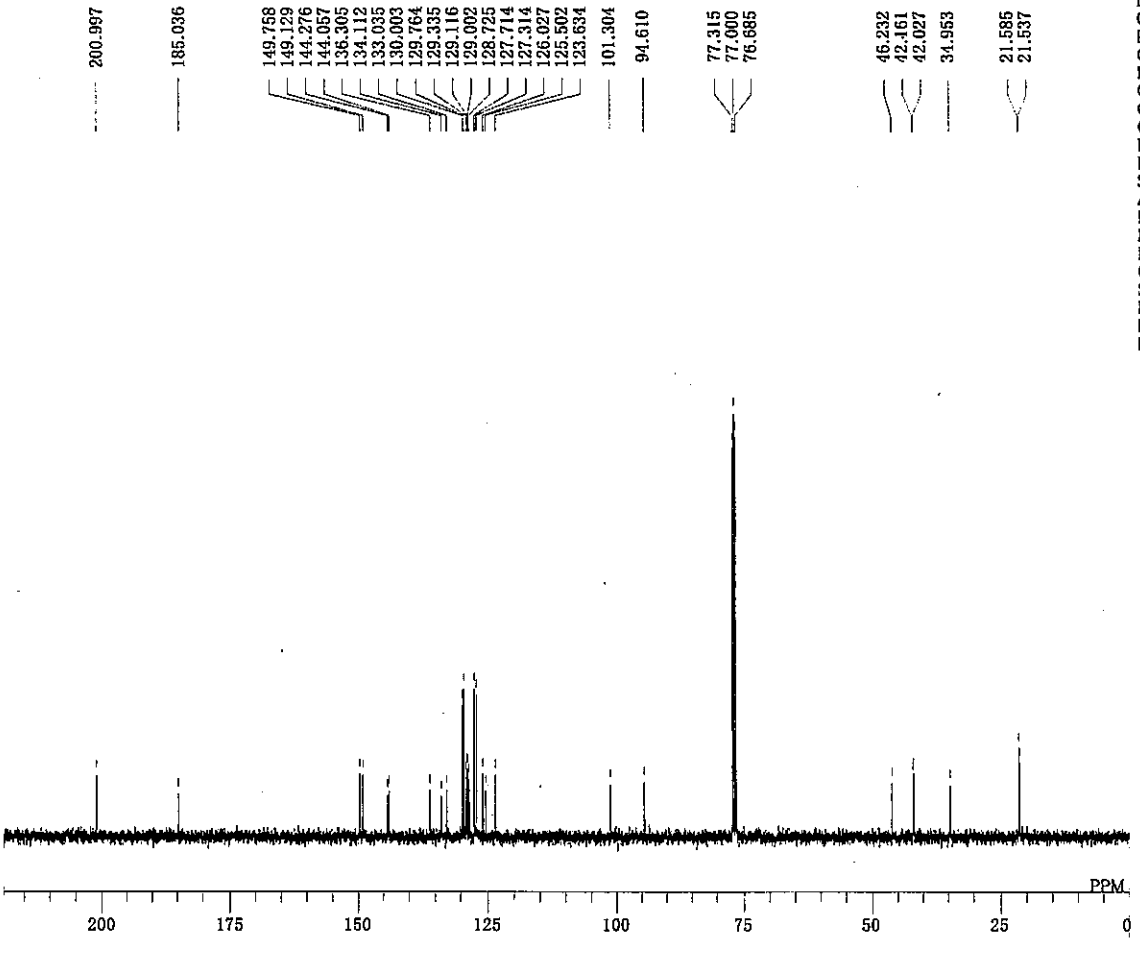
DFILE	YKTY34-33-Fr14-27_proton-3-1
COMNT	single_pulse
DATIM	2013-06-15 17:13:52
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	24.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36



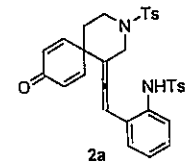
2a

single pulse decoupled gated NOE

G:¥NMR¥横坂¥34¥YKTY34-33-Fr14-27_carbon-1-1.jdf



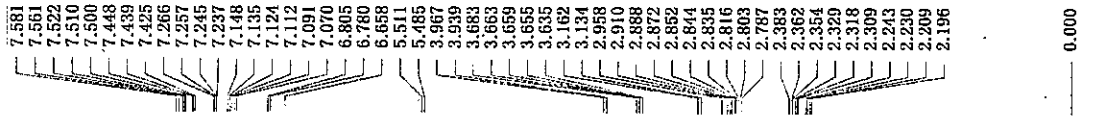
DFILE	YKTY34-33-Fr14-27_carbon-1-1
COMNT	single pulse decoupled gated NOE
DATIM	2013-06-15 15:44:53
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	75
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	24.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



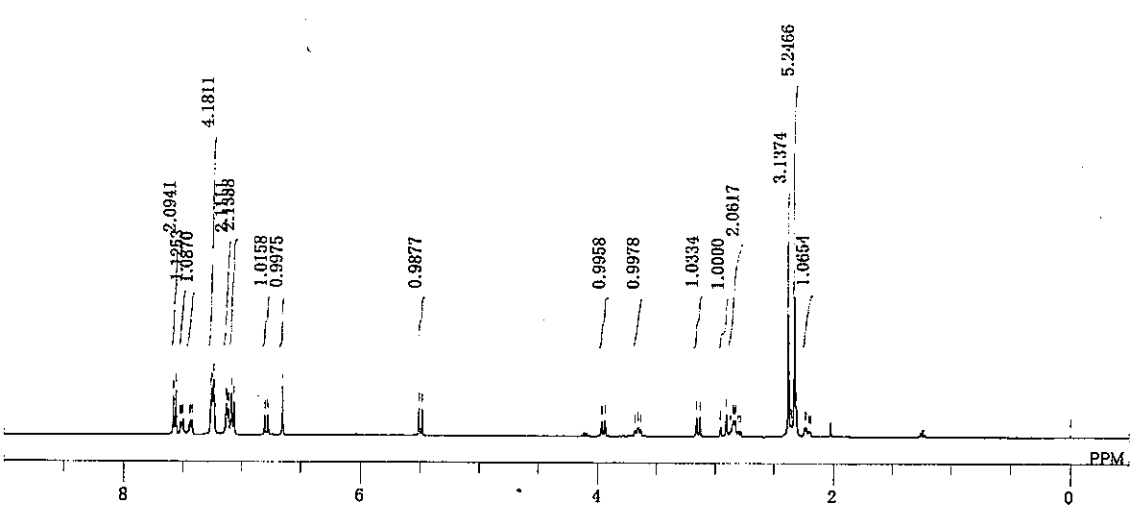
2a

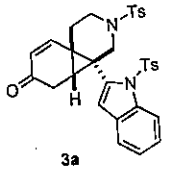
single_pulse

G:¥NMR¥横坂¥34¥YKTY34-38-Fr19-33_proton-1-1.jdf



DFILE	YKTY34-38-Fr19-33_proton-1-1
COMNT	single_pulse
DATIM	2013-06-19 20:56:18
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	25.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	24

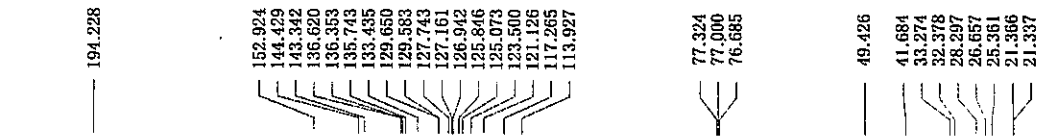




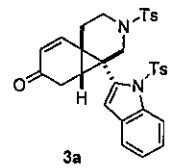
3a

single pulse decoupled gated NOE

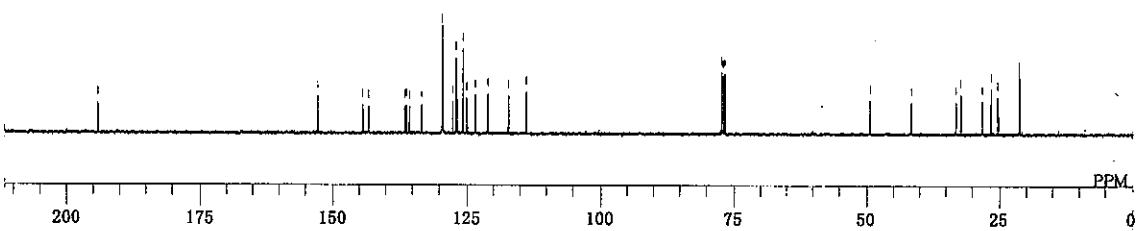
G:¥NMR¥横坂¥34¥YKTY34-38-Fr19-33_carbon-1-1.jdf



DFILE	YKTY34-38-Fr19-33_carbon-1-1
COMNT	single pulse decoupled gated NOE
DATIM	2013-06-19 20:59:19
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	28
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	25.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

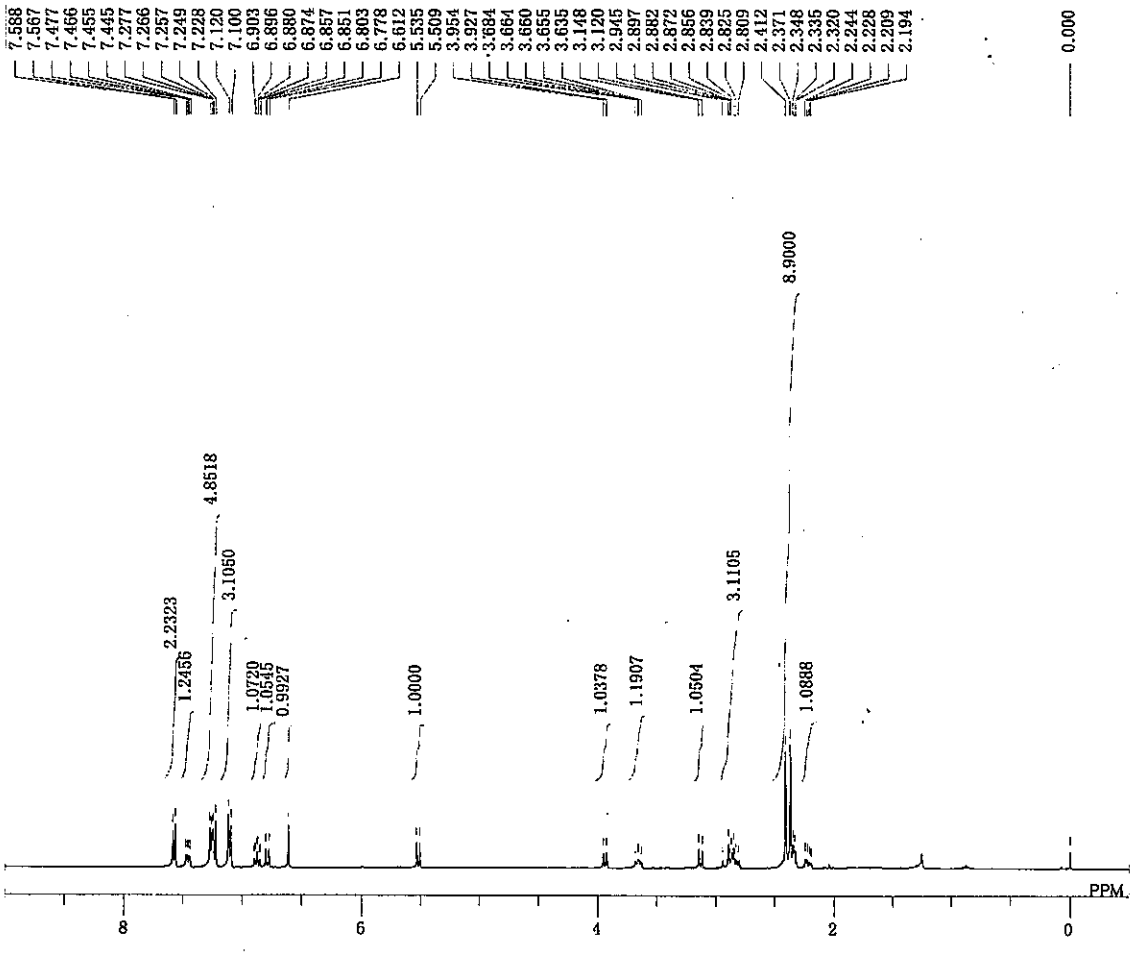


3a

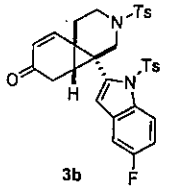


single_pulse

G:¥NMR¥横坂¥35¥YKTY35-30-Fr8-19_proton-1-1.jdf



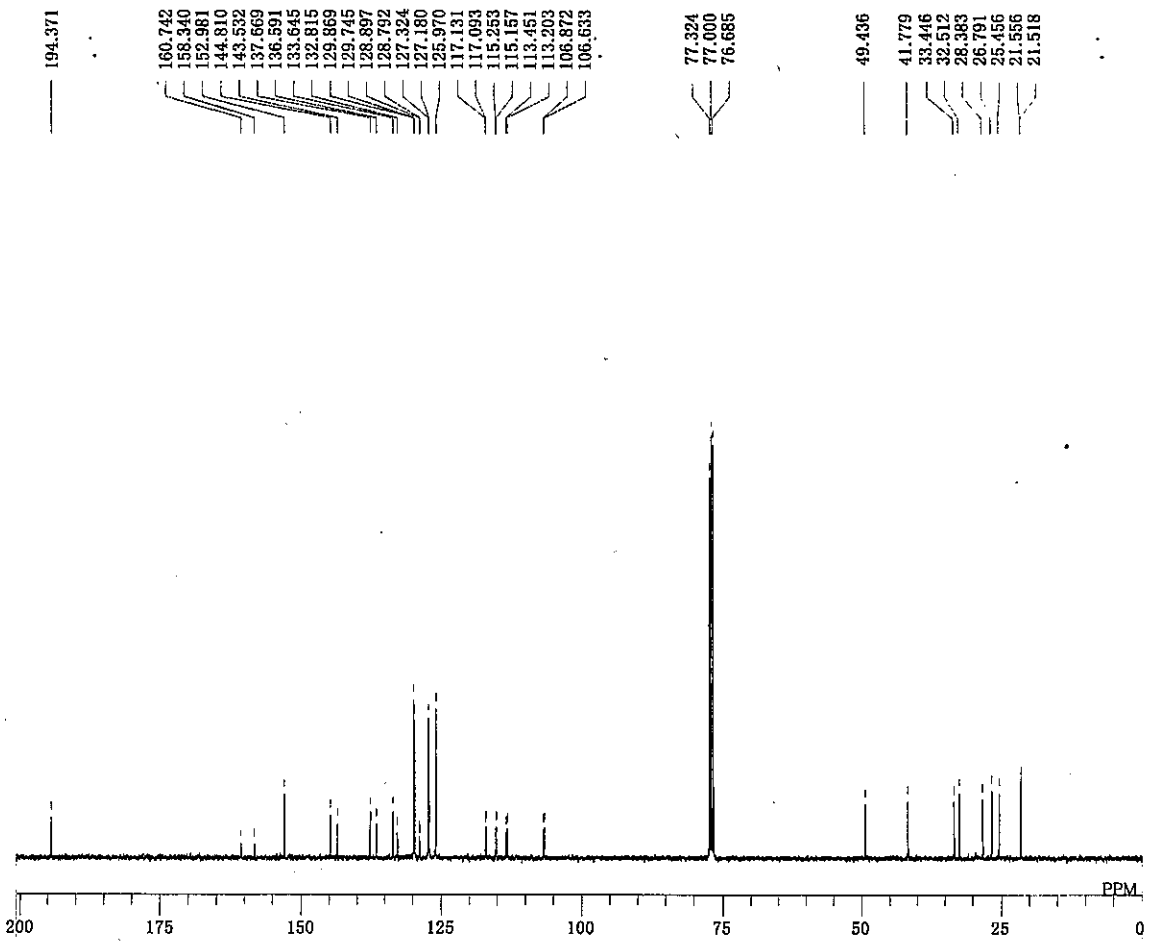
DFILE	YKTY35-30-Fr8-19_proton-1-1.
COMNT	single_pulse
DATIM	2013-09-01 13:20:25
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	22.9 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38



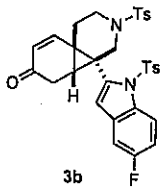
3b

single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-30-Fr8-19_carbon-1-1.jdf



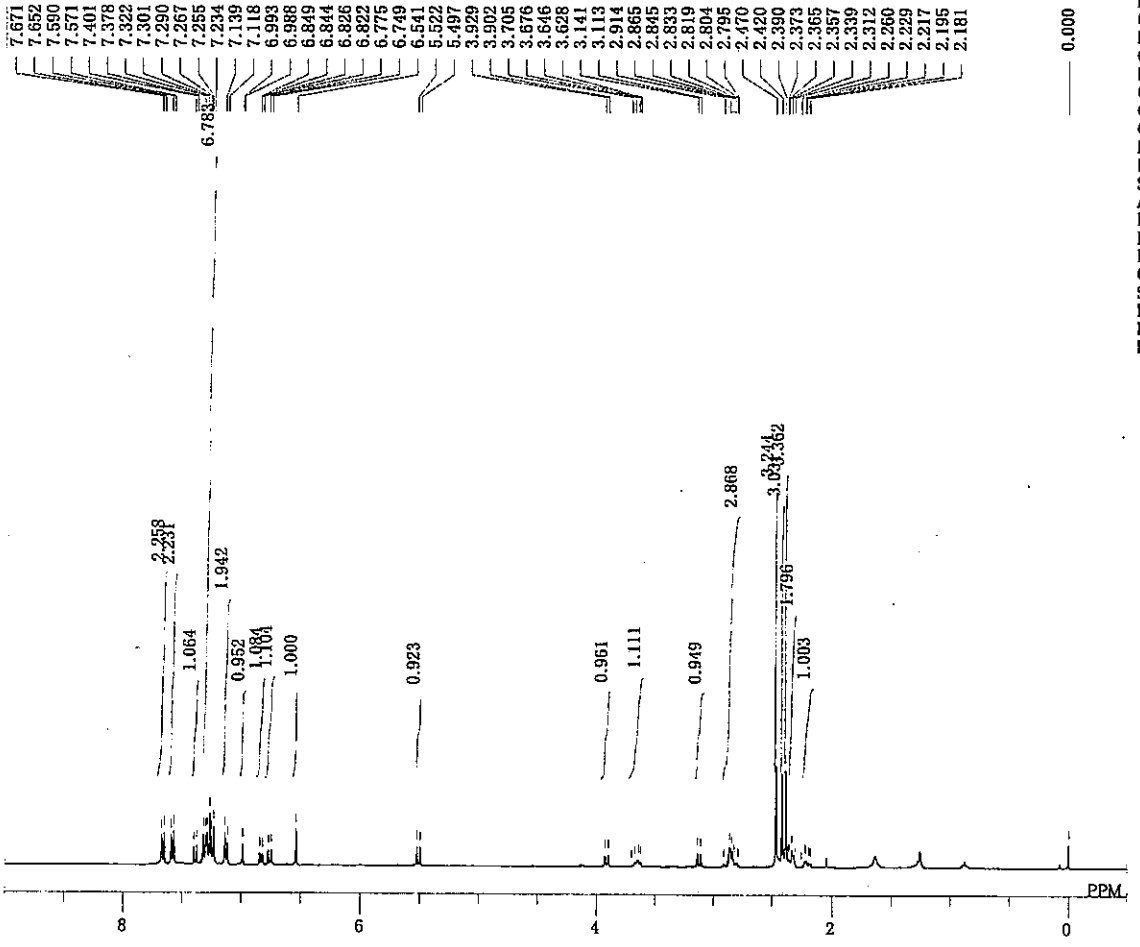
DFILE	YKTY35-30-Fr8-19_carbon-1-1.
COMNT	single pulse decoupled gated NOI
DATIM	2013-09-01 13:23:23
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	670
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	23.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



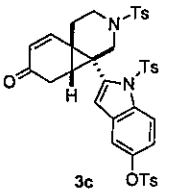
3b

single_pulse

G:¥NMR¥横坂¥35¥YKTY35-45-Fr18-30_proton-1-1.als



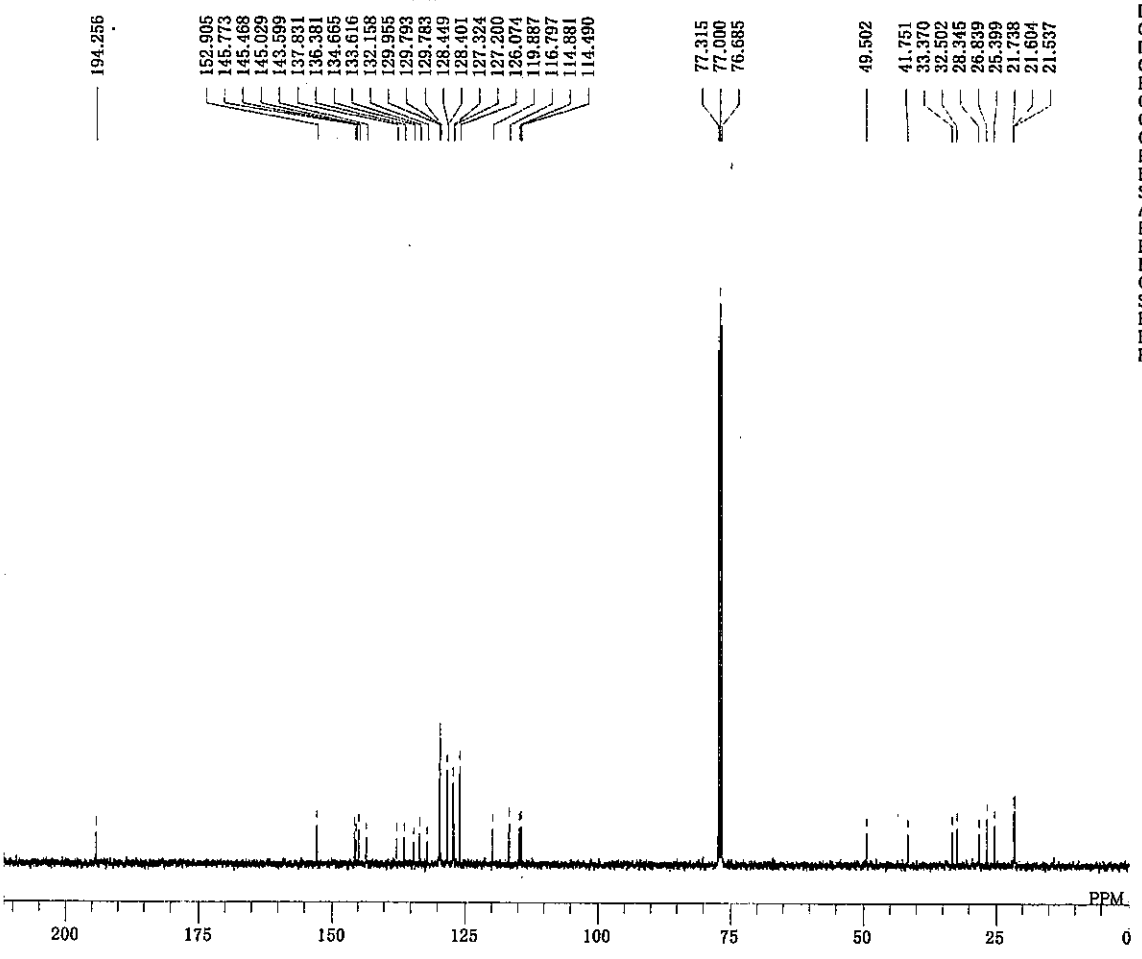
DFILE YKTY35-45-Fr18-30_proton-1-1
COMNT single_pulse
DATIM 2013-09-07 21:44:18
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.01 usec
IRNUC 1H
CTEMP 21.3 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 40



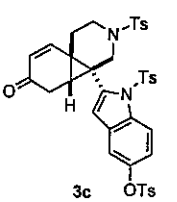
3c

single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-45-Fr18-30_carbon-1-1.jdf



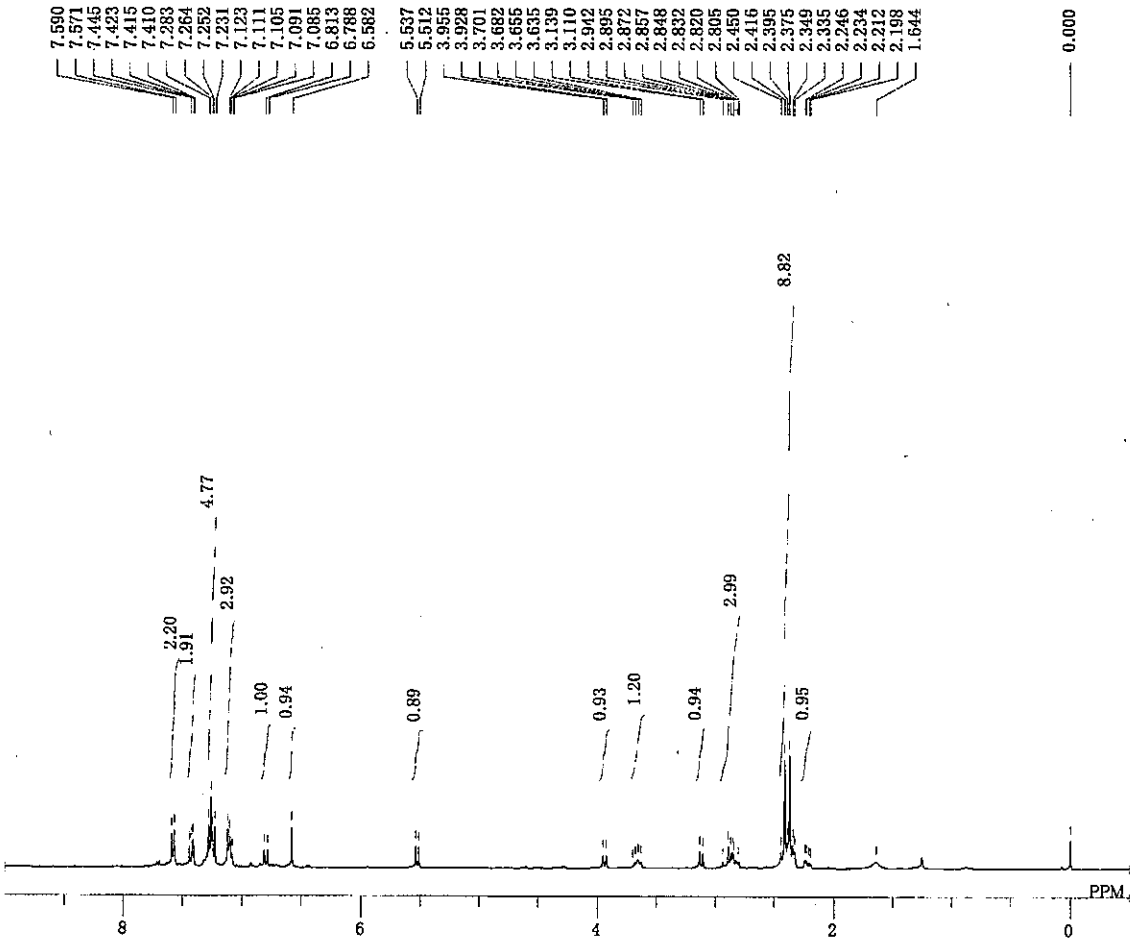
DFILE YKTY35-45-Fr18-30_carbon-1-1
COMNT single pulse decoupled gated NOE
DATIM 2013-09-07 21:47:17
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 32767
FREQU 31407.04 Hz
SCANS 346
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.02 usec
IRNUC 1H
CTEMP 21.6 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60



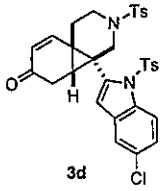
3c

single_pulse

G:¥NMR¥横坂¥35¥YKTY35-67_proton-1-1.jdf



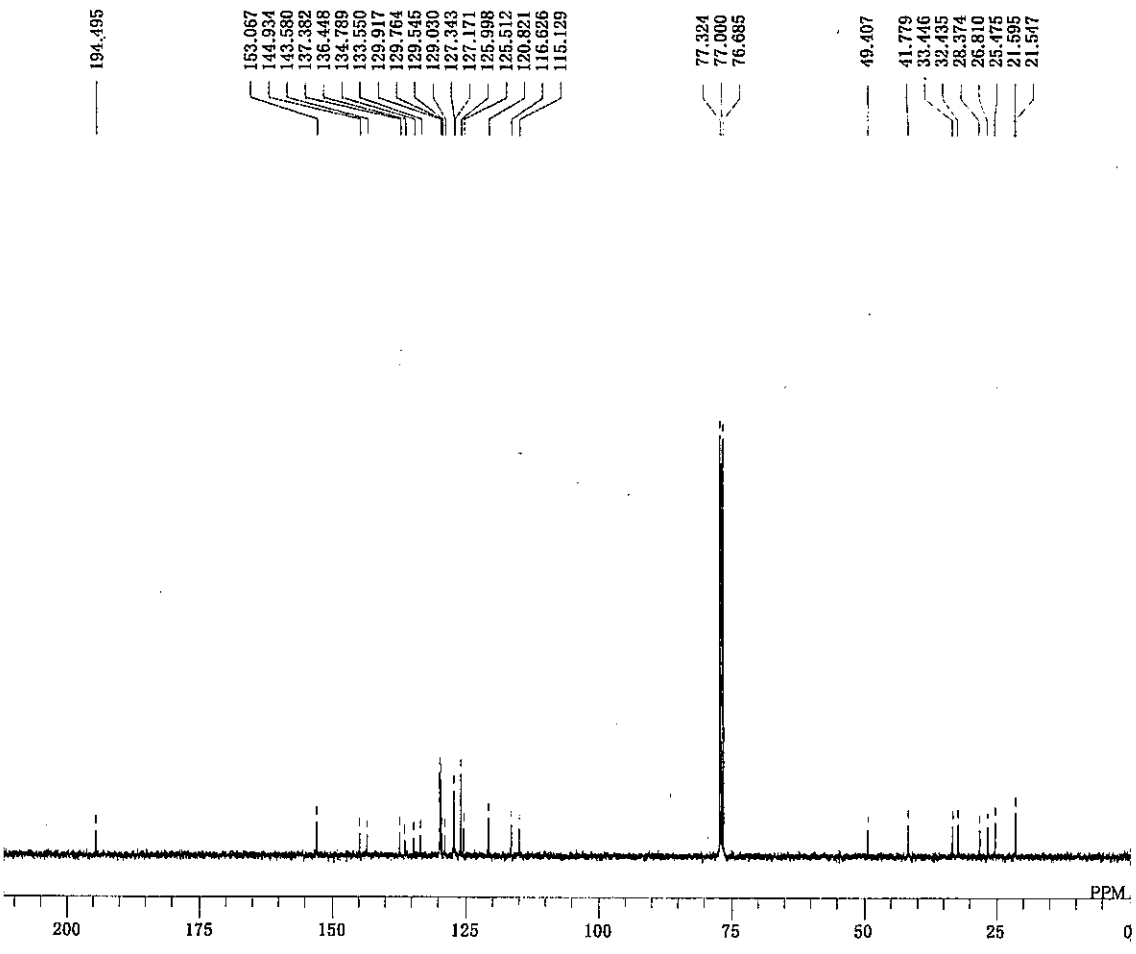
DFILE	YKTY35-67_proton-1-1.jdf
COMNT	single_pulse
DATIM	2013-09-23 16:07:31
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	19.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	42



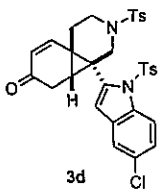
3d

single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-67_carbon-1-1.als



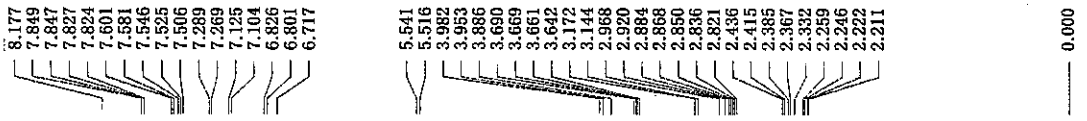
DFILE	YKTY35-67_carbon-1-1.als
COMNT	single pulse decoupled gated NOI
DATIM	2013-09-23 16:10:33
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	337
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	19.2 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



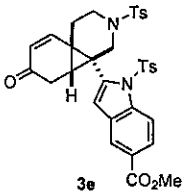
3d

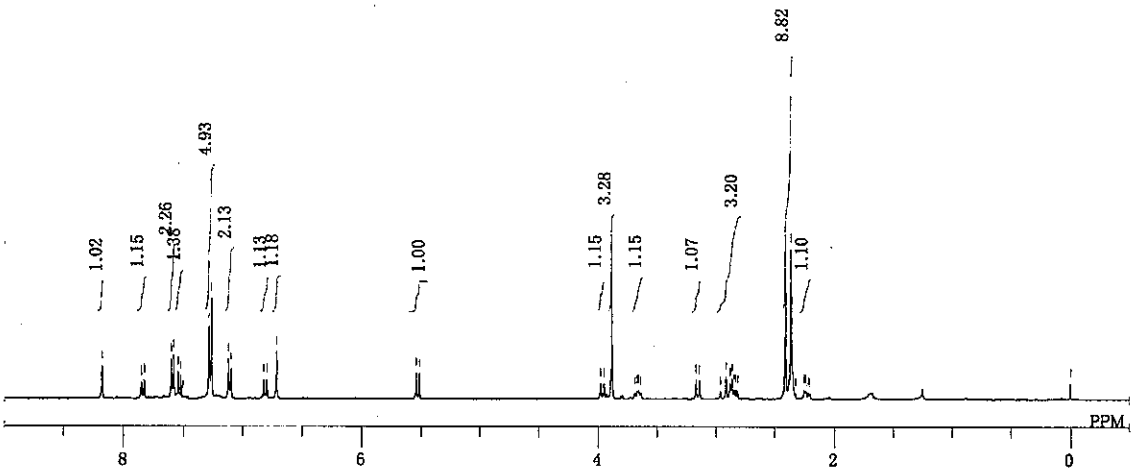
single_pulse

G:¥NMR¥横坂¥35¥YKTY35-72-2-Fr8-21_proton-2-1.jdf



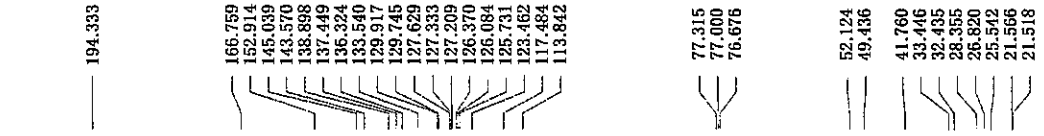
DFILE	YKTY35-72-2-Fr8-21_proton-2-
COMNT	single_pulse
DATIM	2013-09-21 23:45:23
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36



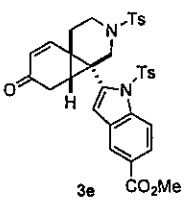


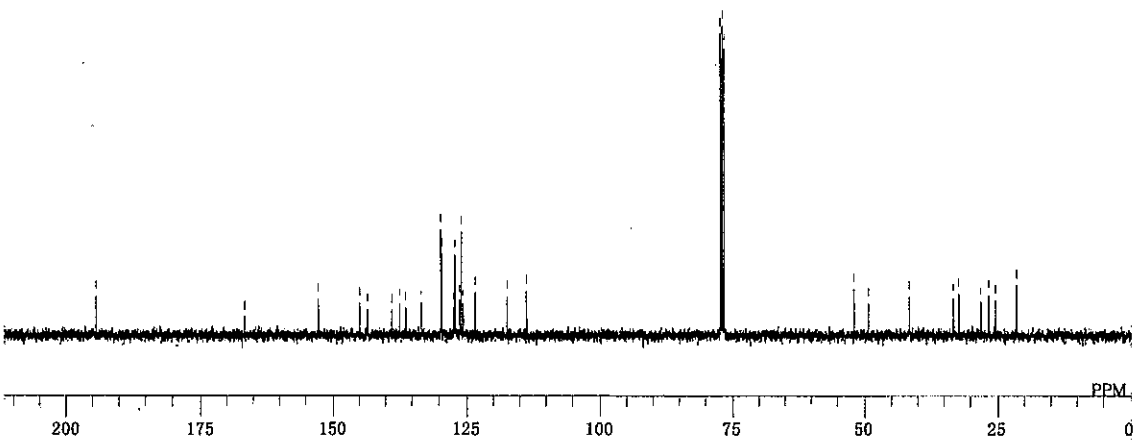
single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-72-2-Fr8-21_carbon-2-1.jdf



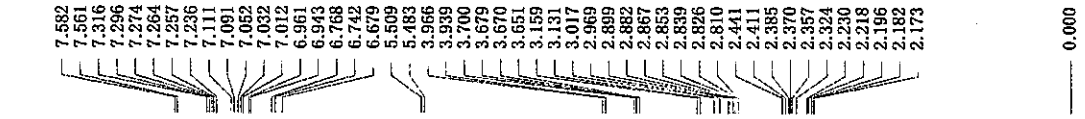
DFILE	YKTY35-72-2-Fr8-21_carbon-2-
COMNT	single pulse decoupled gated NOE
DATIM	2013-09-21 23:48:44
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	53
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



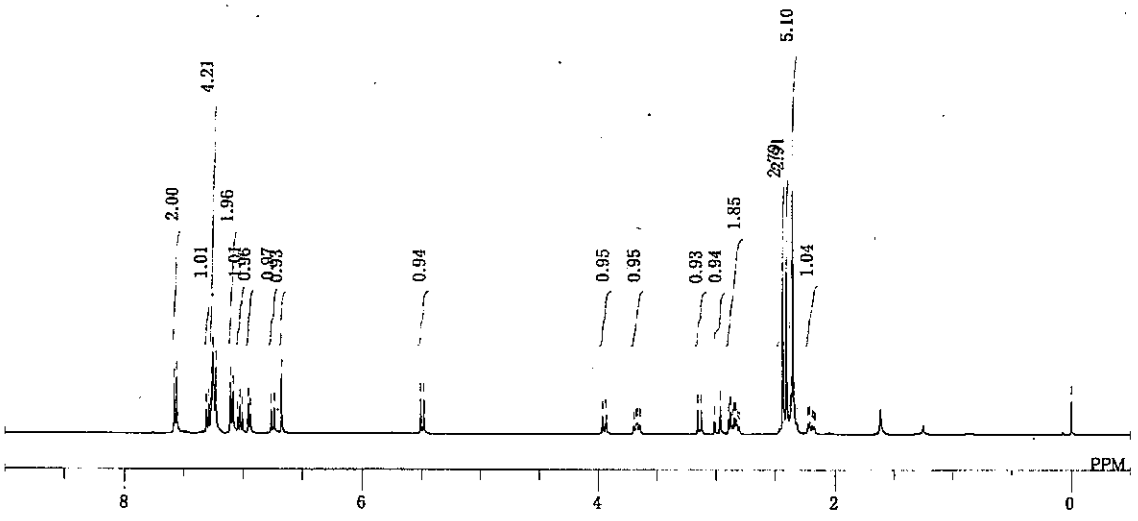


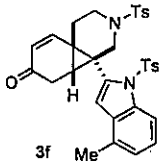
yk-hn-07-58-2_

G:¥NMR¥中山¥07¥yk-hn-07-58-2_proton-1-1.jdf



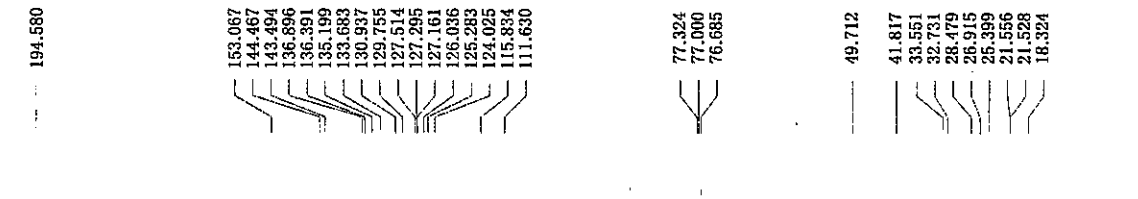
DFILE	yk-hn-07-58-2_proton-1-1.jdf
COMNT	yk-hn-07-58-2_
DATIM	2013-10-12 15:10:40
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



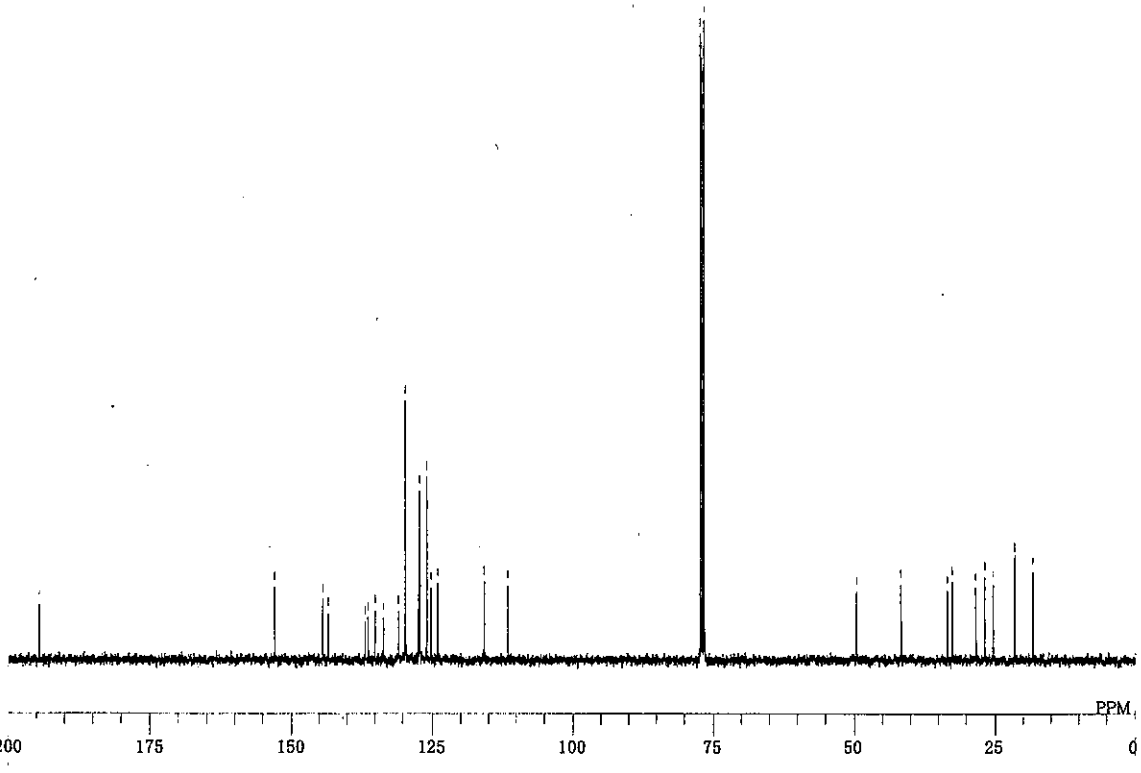


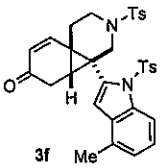
yk-hn-07-58-2_yk-hn-07-58-2_proton

G:¥NMR¥中山¥07¥yk-hn-07-58-2_carbon-1-1.jdf



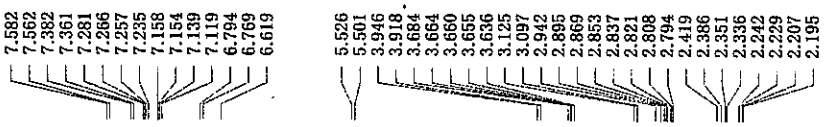
DFILE	yk-hn-07-58-2_carbon-1-1.jdf
COMNT	yk-hn-07-58-2_yk-hn-07-58-2_
DATIM	2013-10-12 15:12:07
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	390
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	20.1 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



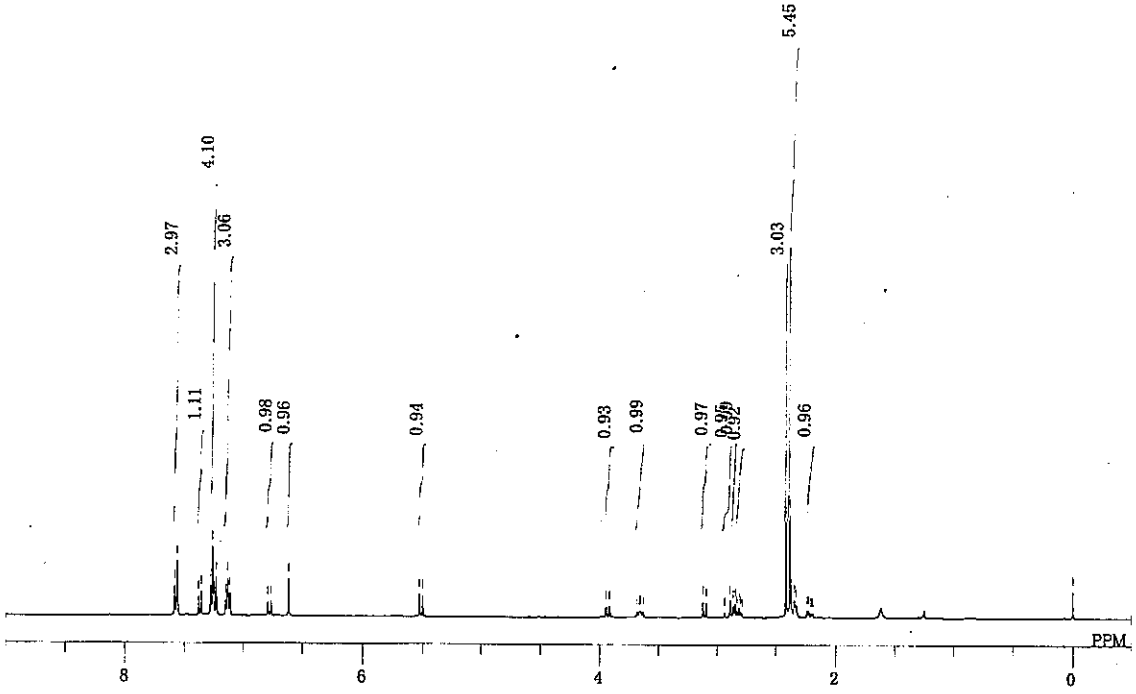


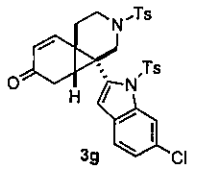
yk-hn-07-63_

G:¥NMR¥中山¥07¥yk-hn-07-63-2_proton-1-1.als



DFILE	yk-hn-07-63-2_proton-1-1.als
COMNT	yk-hn-07-63_
DATIM	2013-10-21 12:34:23
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.40 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	19.1 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

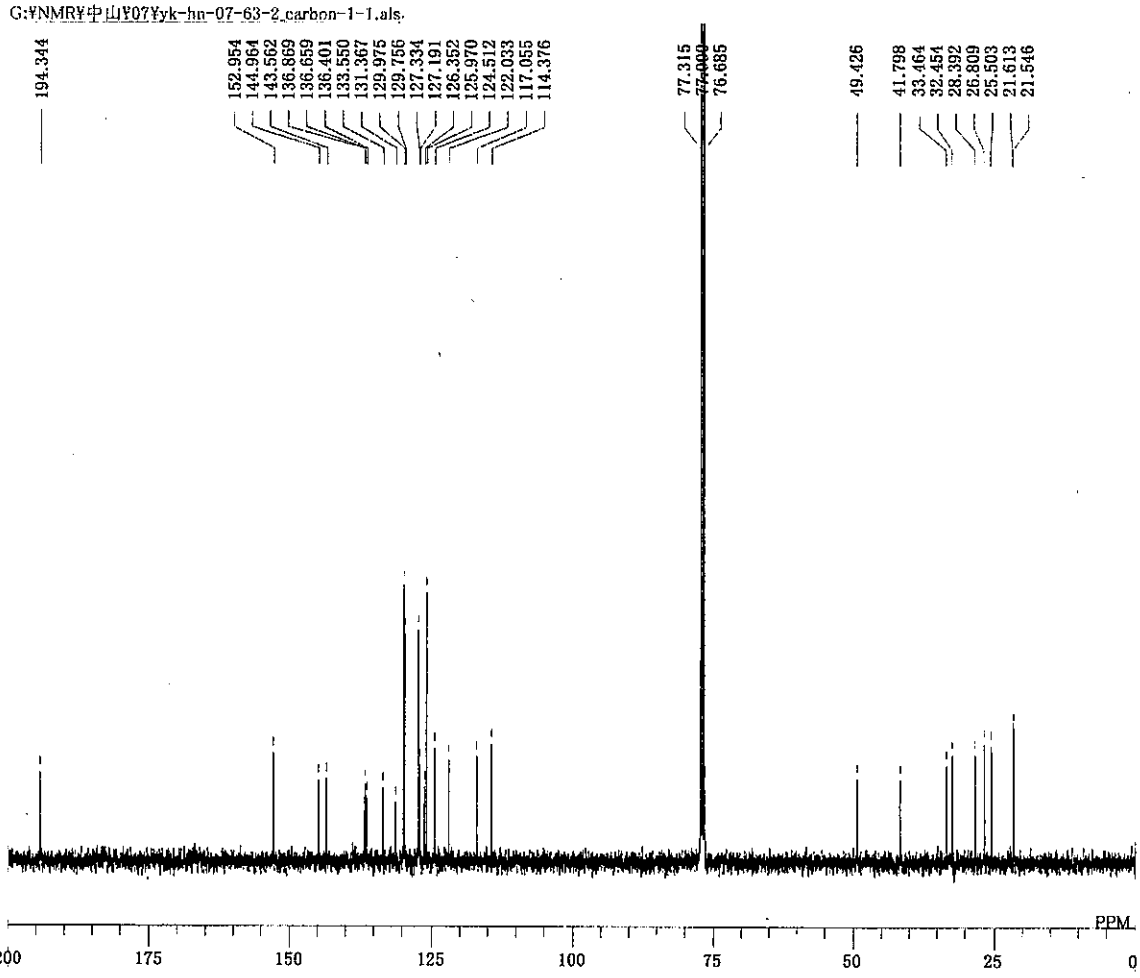




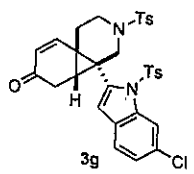
3g

yk-hn-07-63-2_yk-hn-07-63-2_proton

G:¥NMR¥中山¥07¥yk-hn-07-63-2_carbon-1-1.als



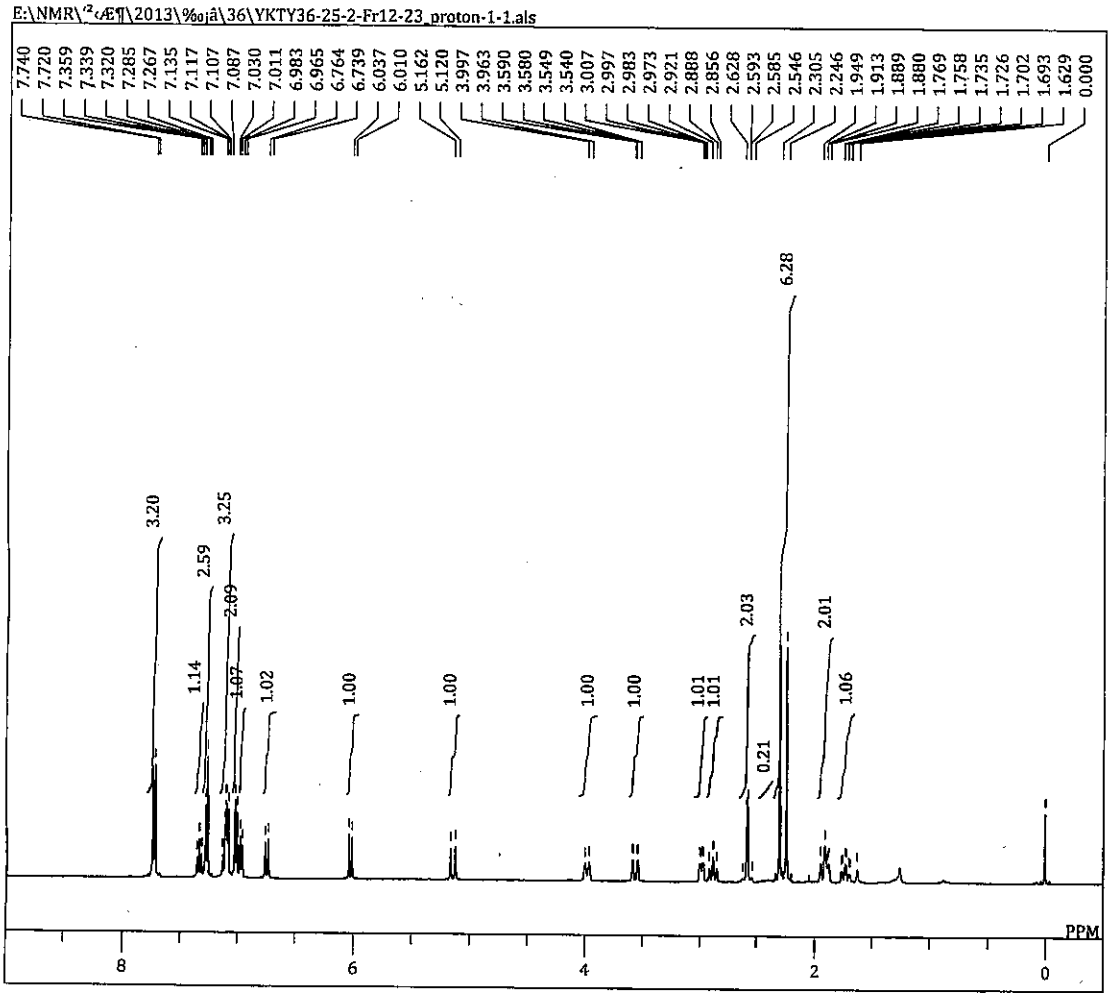
DFILE	yk-hn-07-63-2_carbon-1-1.als
COMNT	yk-hn-07-63-2_yk-hn-07-63-2_
DATIM	2013-10-19 18:11:20
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.63 Hz
SCANS	450
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



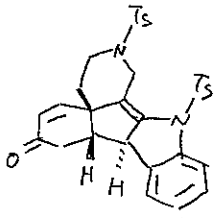
3g

single_pulse

E:\NMR\´²‹Æ¶\2013\‰¡â\36\YKTY36-25-2-Fr12-23_proton-1-1.als



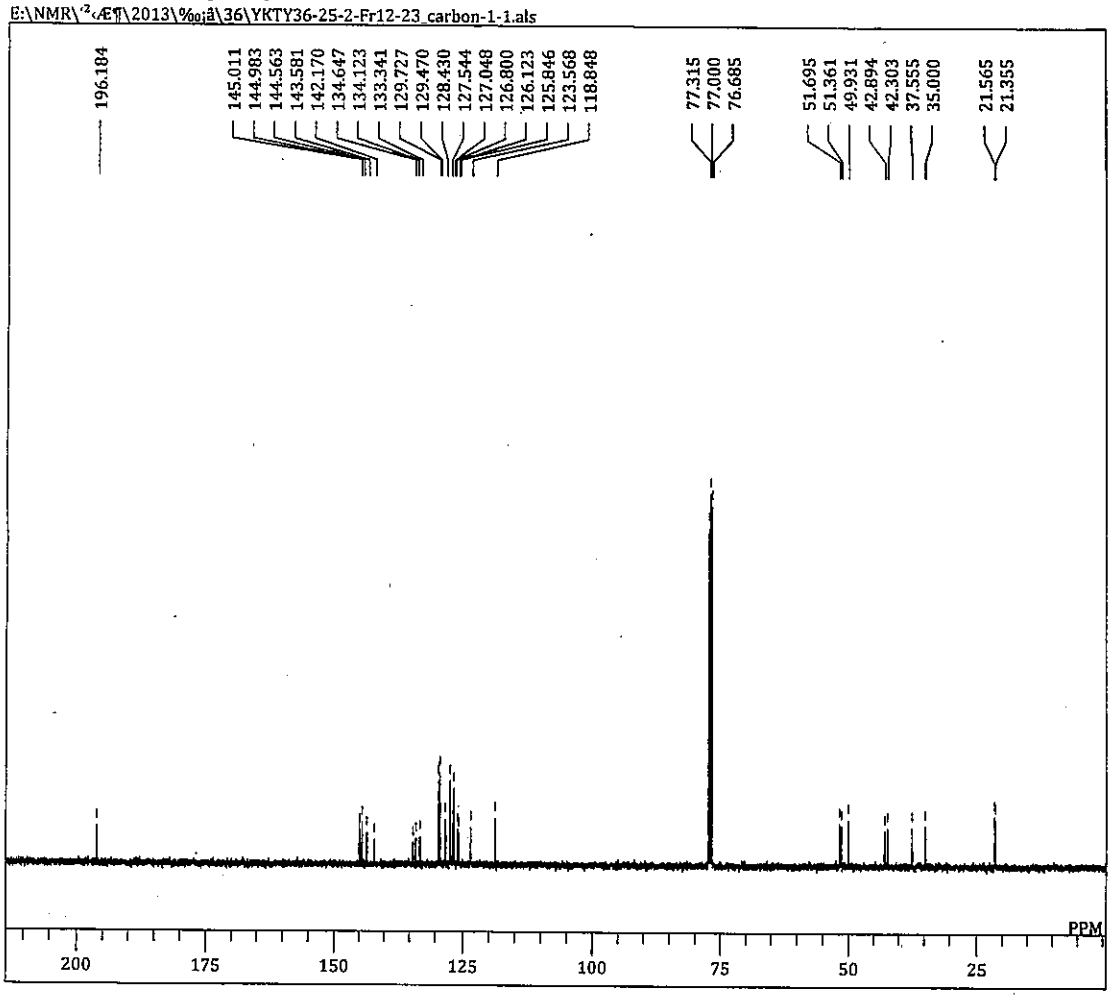
DFILE YKTY36-25-2-Fr12-23_proton-1-1.
COMNT single_pulse
DATIM 2014-03-07 13:56:57
OBNUC 1H
EXMOD proton.jxp
OBFRQ 399.78 MHz
OBSET 4.19 KHz
OBFIN 7.29 Hz
POINT 13107
FREQU 6002.40 Hz
SCANS 8
ACQTM 2.1837 sec
PD 5.0000 sec
PW1 5.01 usec
IRNUC 1H
CTEMP 17.8 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.12 Hz
RGAIN 38



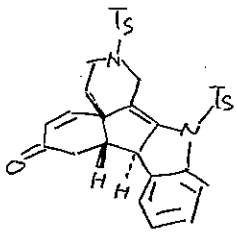
4a

single pulse decoupled gated NOE

E:\NMR\´²‹Æ¶\2013\‰¡â\36\YKTY36-25-2-Fr12-23_carbon-1-1.als



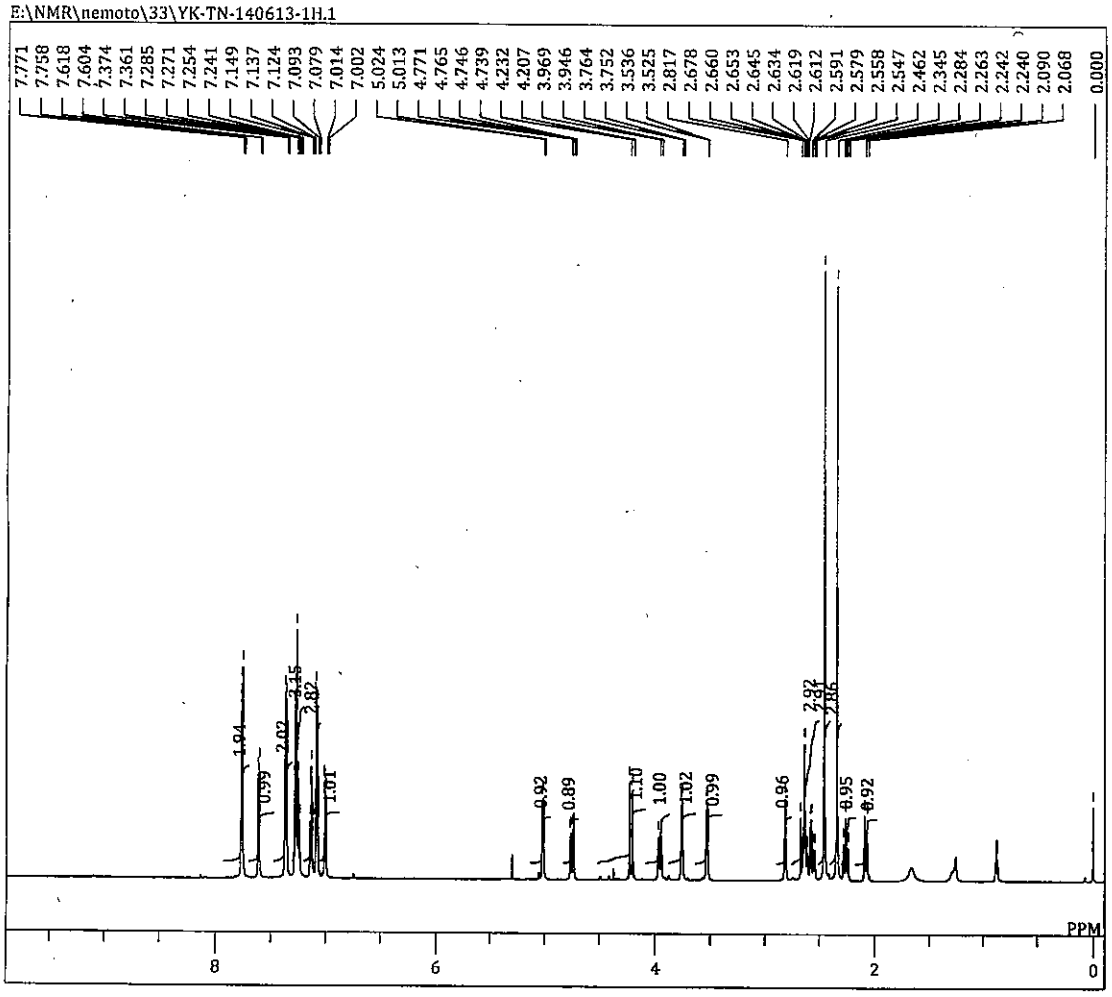
DFILE YKTY36-25-2-Fr12-23_carbon-1-1.
COMNT single pulse decoupled gated NOE
DATIM 2014-03-07 13:59:56
OBNUC 13C
EXMOD carbon.jxp
OBFRQ 100.53 MHz
OBSET 5.35 KHz
OBFIN 5.86 Hz
POINT 26214
FREQU 25125.63 Hz
SCANS 180
ACQTM 1.0433 sec
PD 2.0000 sec
PW1 3.02 usec
IRNUC 1H
CTEMP 17.8 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 1.20 Hz
RGAIN 60



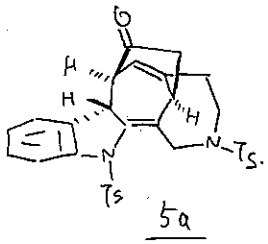
4a

YK-TN-140613-1H

E:\NMR\nemoto\33\YK-TN-140613-1H.1



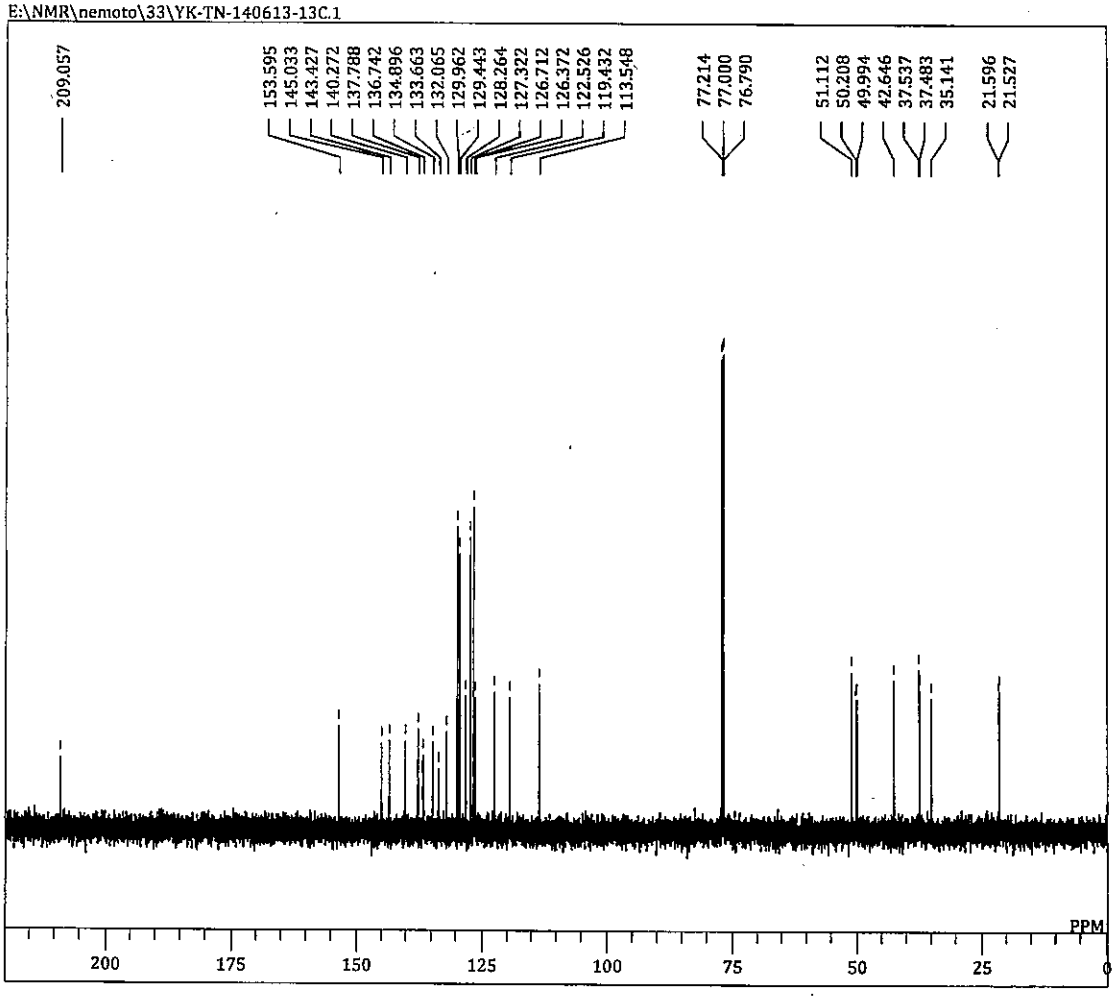
DFILE	YK-TN-140613-1H.1
COMNT	YK-TN-140613-1H
DATIM	2014-06-14 05:50:00
OBNUC	1H
EXMOD	single_pulse.exp
OBFRQ	600.17 MHz
OBSET	5.90 KHz
OBFIN	5.64 Hz
POINT	32768
FREQU	9009.01 Hz
SCANS	8
ACQTM	3.6372 sec
PD	4.0000 sec
PW1	3.45 usec
IRNUC	
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	17



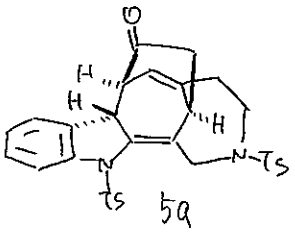
5a

YK-TN-140613-13C

E:\NMR\nemoto\33\YK-TN-140613-13C.1



DFILE	YK-TN-140613-13C.1
COMNT	YK-TN-140613-13C
DATIM	2014-06-14 05:57:04
OBNUC	13C
EXMOD	single_pulse_dec
OBFRQ	150.93 MHz
OBSET	0.03 KHz
OBFIN	0.87 Hz
POINT	65536
FREQU	37735.85 Hz
SCANS	108
ACQTM	1.7367 sec
PD	1.5000 sec
PW1	9.75 usec
IRNUC	1H
CTEMP	21.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	27



5a

JEOL

```
---- ACQUISITION PARAMETERS ----
File Name              = YK-TN-140613-COSY
Author                 =
Sample ID              = tn
Content                = YK-TN-140613-COSY
Creation Date          = 14-JUN-2014 06:22

Revision Date          = 13-JUN-2014 16:36
Spec Site              = ECP600

Spec Type              = DELTA_NMR
Data Format            = 2D REAL REAL
Dimensions             = X Y
Dim Title              = 1H 1H
Dim Size               = 512, 512
Dim Units              = [ppm][ppm]
Scans                  = 4
Mod_return             = 1
X_points               = 512
X_prescans             = 4
X_domain               = 1H
X_offset               = 5.13633[ppm]
X_freq                 = 600.1723046[MHz]
X_sweep                = 4.18060201[kHz]
X_resolution           = 8.16523829[Hz]
Y_points               = 256
Y_prescans             = 0
Y_domain               = 1H
Y_offset               = 5.13633[ppm]
Y_freq                 = 600.1723046[MHz]
Y_sweep                = 4.18060201[kHz]
Y_resolution           = 16.33047659[Hz]
X_acq_duration         = 0.1224704[s]
Digital_filter         = FALSE
Filter_factor          = 1
Delay_of_start         = 1[s]
Actual_start_time      = 14-JUN-2014 05:57
Acq_delay              = 0.2358[ms]
X90                    = 6.9[us]
Y90                    = 6.25[us]
Irr90                  = 6.9[us]
Tri90                  = 10[us]
Qua90                  = 10[us]
X90_hi                 = 11.5[us]
Y90_hi                 = 10[us]
Irr90_hi               = 11.5[us]
Tri90_hi               = 10[us]
Qua90_hi               = 10[us]
X90_lo                 = 55[us]
Y90_lo                 = 50[us]
Irr90_lo               = 55[us]
Tri90_lo               = 10[us]
Qua90_lo               = 10[us]
Spin_lock_90           = 25[us]
Spin_lock_attn         = 13[dB]
Deut_grad_shim_90      = 0.14[ms]
Deut_grad_shim_attn    = 12.8[dB]
Adc_card               = 16/1MHz/20
Field_strength         = 14.09636928[T]
Filter_mode            = BUTTERWORTH
Filter_width           = 2.090301[kHz]
Recvr_gain             = 17
Irr_code               = 128
Obs_pwidth             = 1[us]
Obs_noise              = WAUGH
Irr_pwidth             = 55[us]
Irr_noise              = WALTZ
Tri_pwidth             = 1[us]
Tri_noise              = WALTZ
Qua_pwidth             = 1[us]
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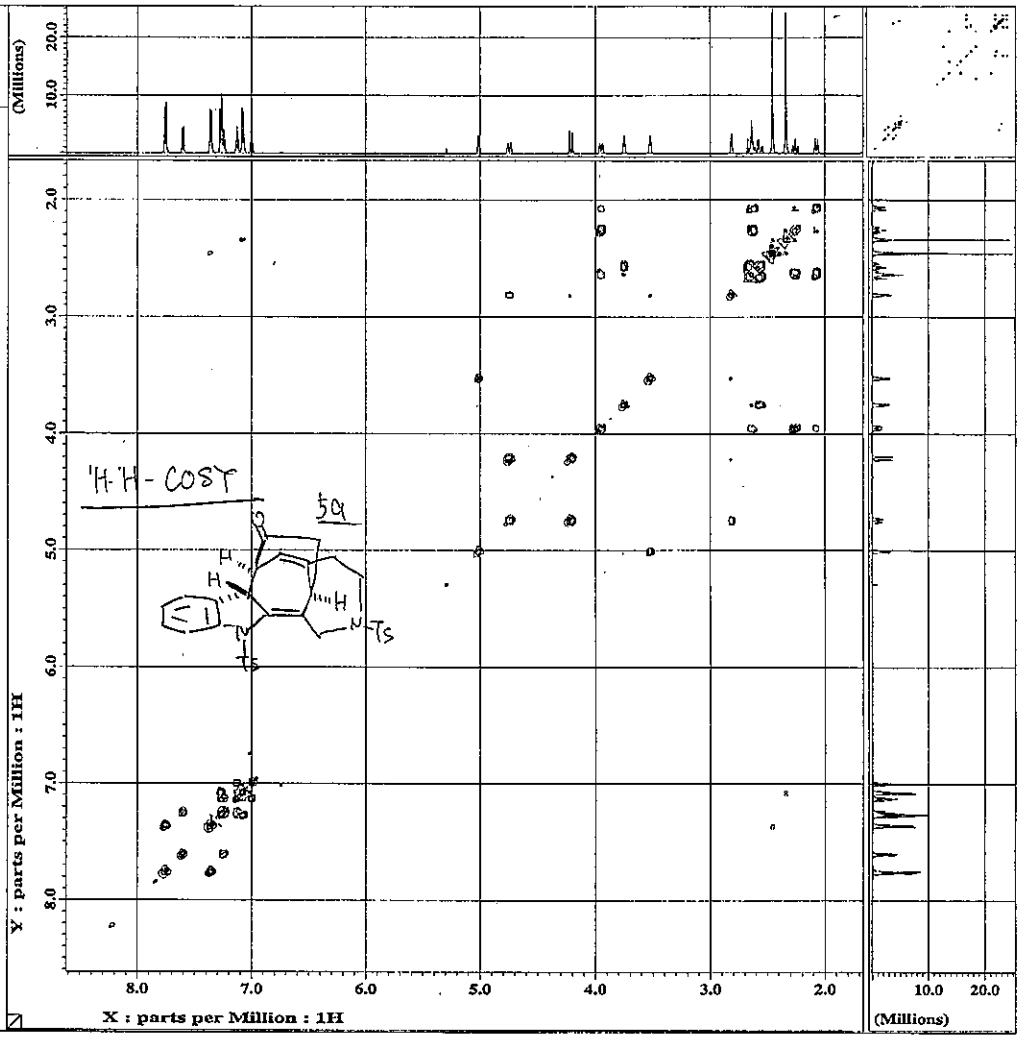
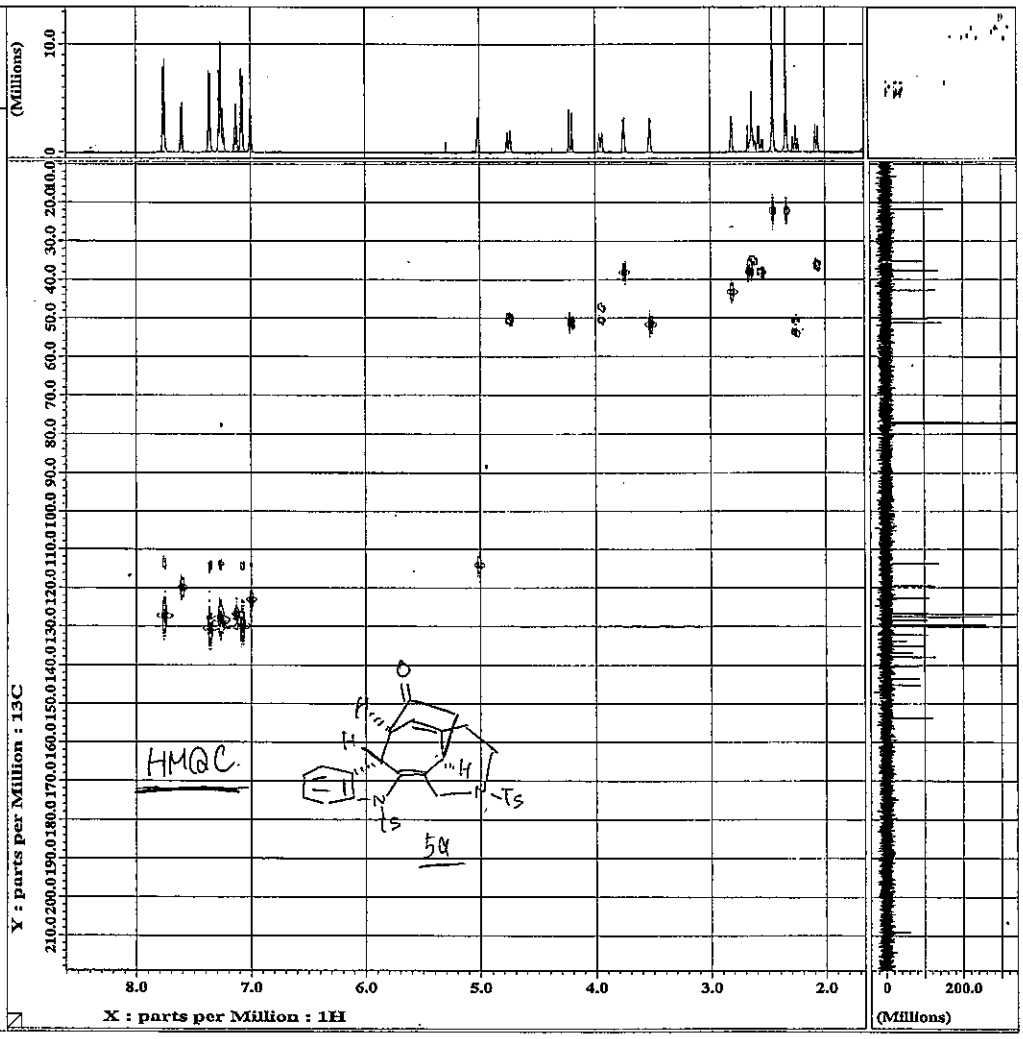


JEOL

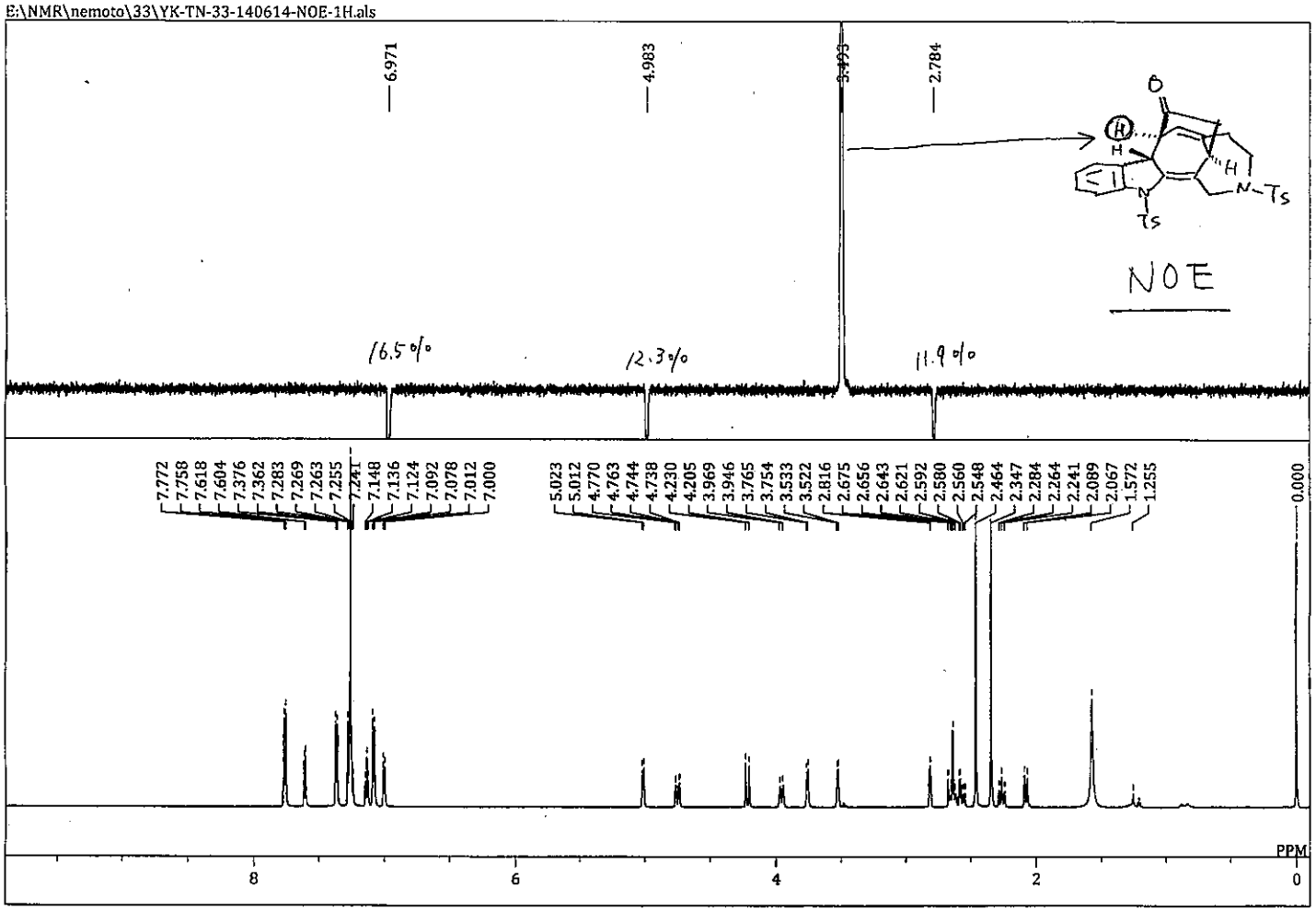
```
---- ACQUISITION PARAMETERS ----
File Name              = YK-TN-140613-HMQC
Author                 =
Sample ID              = tn
Content                = YK-TN-140613-HMQC
Creation Date          = 14-JUN-2014 07:12

Revision Date          = 13-JUN-2014 17:11
Spec Site              = ECP600

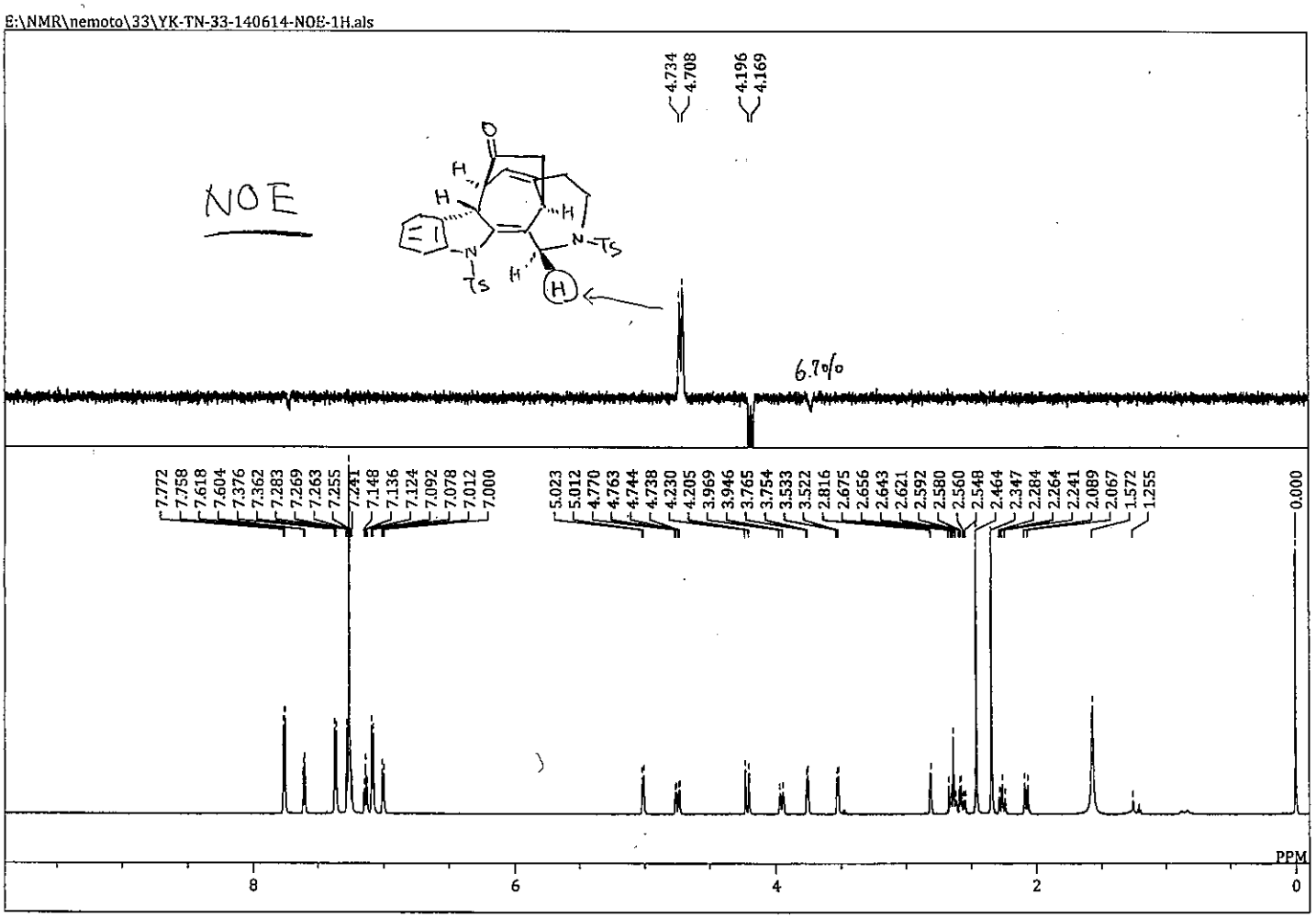
Spec Type              = DELTA_NMR
Data Format            = 2D REAL REAL
Dimensions             = X Y
Dim Title              = 1H 13C
Dim Size               = 1024, 512
Dim Units              = [ppm][ppm]
Scans                  = 8
Mod_return             = 1
X_points               = 1024
X_prescans             = 4
X_domain               = 1H
X_offset               = 5.13633[ppm]
X_freq                 = 600.1723046[MHz]
X_sweep                = 4.18060201[kHz]
X_resolution           = 4.08261915[Hz]
Y_points               = 256
Y_prescans             = 0
Y_domain               = 13C
Y_offset               = 113.59969[ppm]
Y_freq                 = 150.91343039[MHz]
Y_sweep                = 31.84713376[kHz]
Y_resolution           = 124.40286624[Hz]
X_acq_duration         = 0.2449408[s]
Digital_filter         = FALSE
Filter_factor          = 1
Delay_of_start         = 1[s]
Actual_start_time      = 14-JUN-2014 06:22
Acq_delay              = 0.2358[ms]
X90                    = 6.9[us]
Y90                    = 19.5[us]
Irr90                  = 6.9[us]
Tri90                  = 10[us]
Qua90                  = 10[us]
X90_hi                 = 11.5[us]
Y90_hi                 = 19.5[us]
Irr90_hi               = 11.5[us]
Tri90_hi               = 10[us]
Qua90_hi               = 10[us]
X90_lo                 = 55[us]
Y90_lo                 = 70[us]
Irr90_lo               = 55[us]
Tri90_lo               = 10[us]
Qua90_lo               = 10[us]
Spin_lock_90           = 25[us]
Spin_lock_attn         = 13[dB]
Deut_grad_shim_90      = 0.14[ms]
Deut_grad_shim_attn    = 12.8[dB]
Adc_card               = 16/1MHz/20
Field_strength         = 14.09636928[T]
Filter_mode            = BUTTERWORTH
Filter_width           = 2.090301[kHz]
Recvr_gain             = 32
Irr_code               = 128
Obs_pwidth             = 1[us]
Obs_noise              = WAUGH
Irr_pwidth             = 70[us]
Irr_noise              = MPF6
Tri_pwidth             = 1[us]
Tri_noise              = WALTZ
Qua_pwidth             = 1[us]
```



E:\NMR\nemoto\33\YK-TN-33-140614-NOE-1H.als

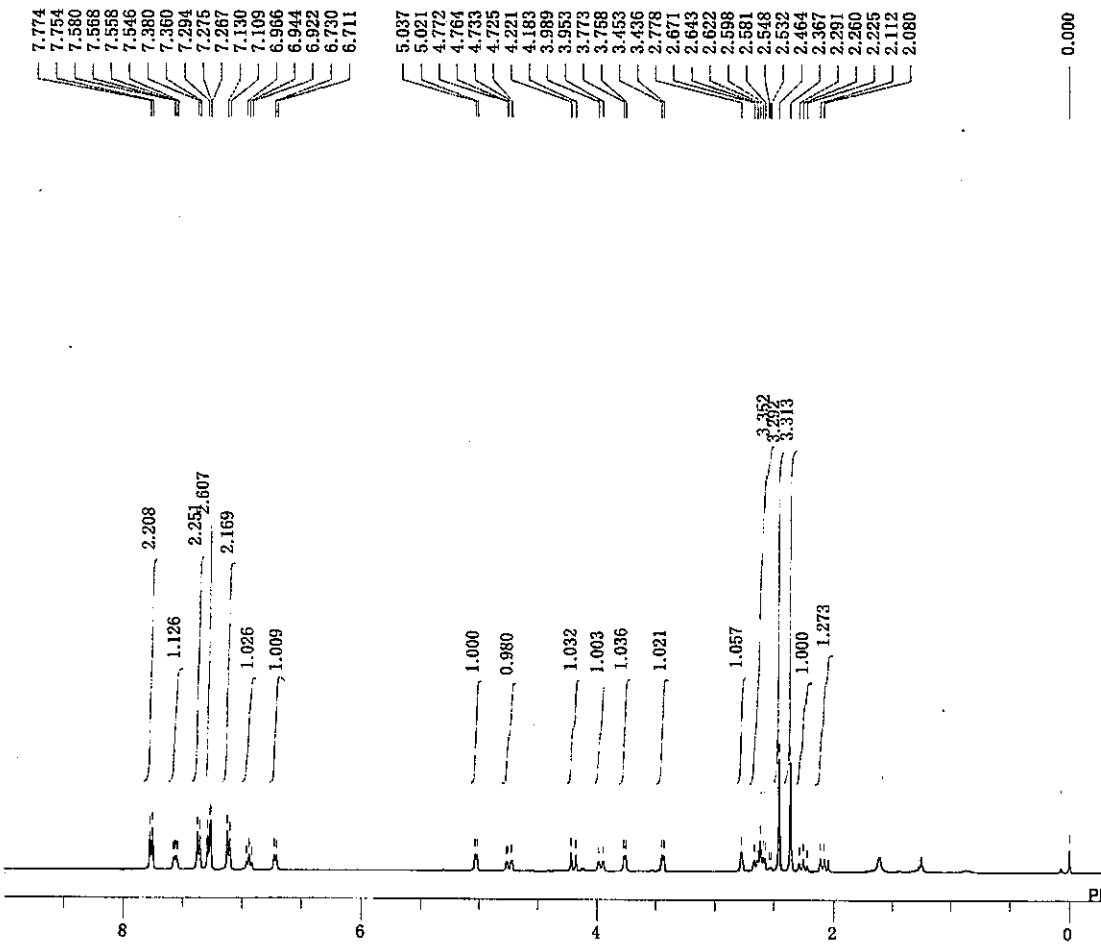


E:\NMR\nemoto\33\YK-TN-33-140614-NOE-1H.als

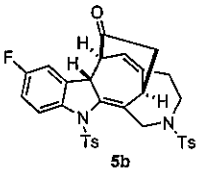


single_pulse

G:¥NMR¥横坂¥35¥YKTY35-47-Fr7-15_proton-2-1.jdf



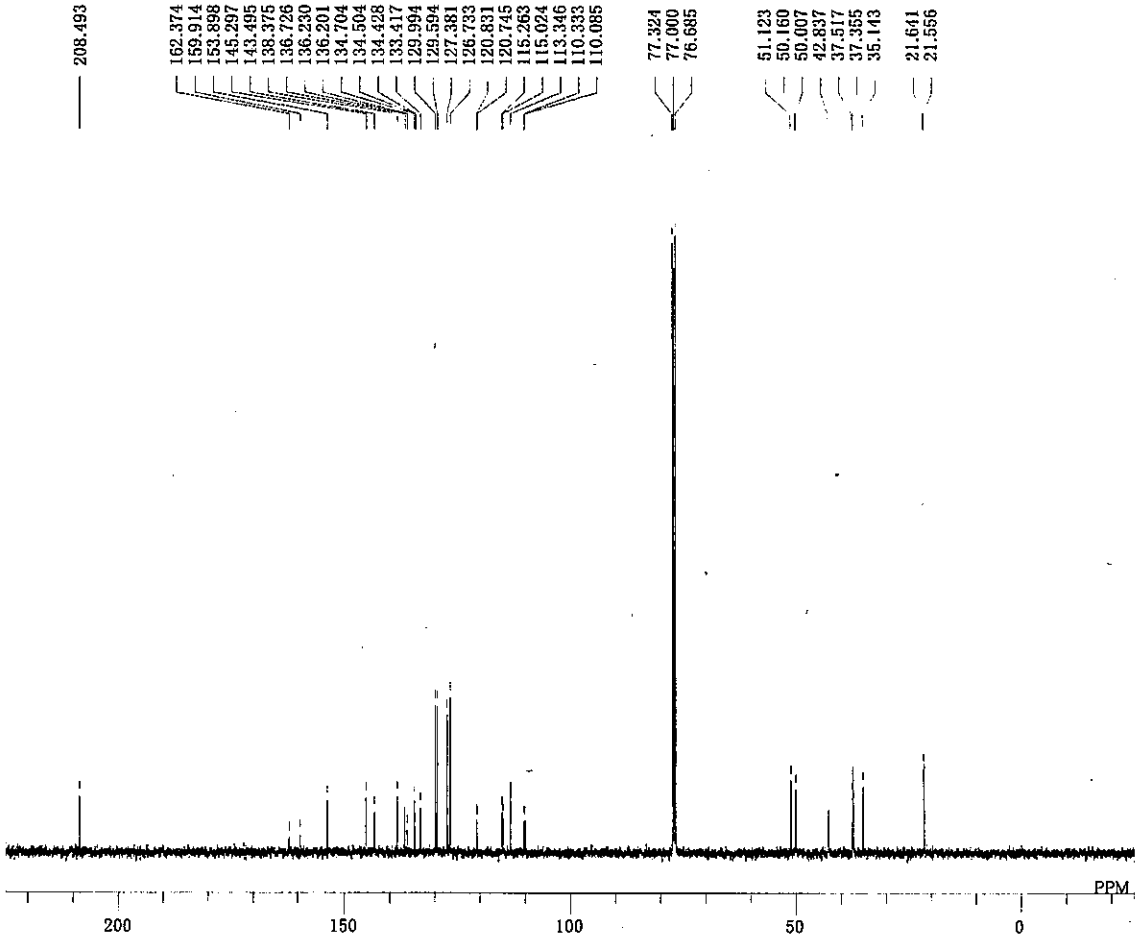
DFILE	YKTY35-47-Fr7-15_proton-2-1.
COMNT	single_pulse
DATIM	2013-09-07 21:17:60
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



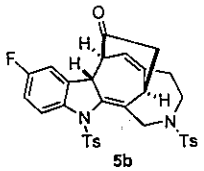
5b

single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-47-Fr7-15_carbon-2-1.als



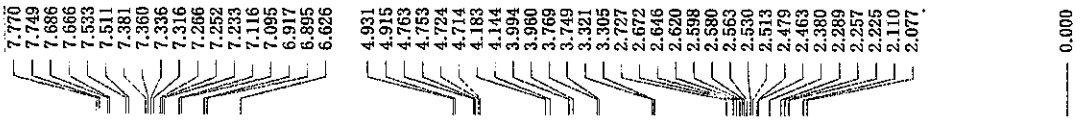
DFILE	YKTY35-47-Fr7-15_carbon-2-1.
COMNT	single pulse decoupled gated NOI
DATIM	2013-09-07 21:21:19
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.63 Hz
SCANS	357
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	21.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



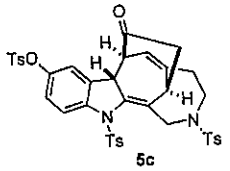
5b

single_pulse

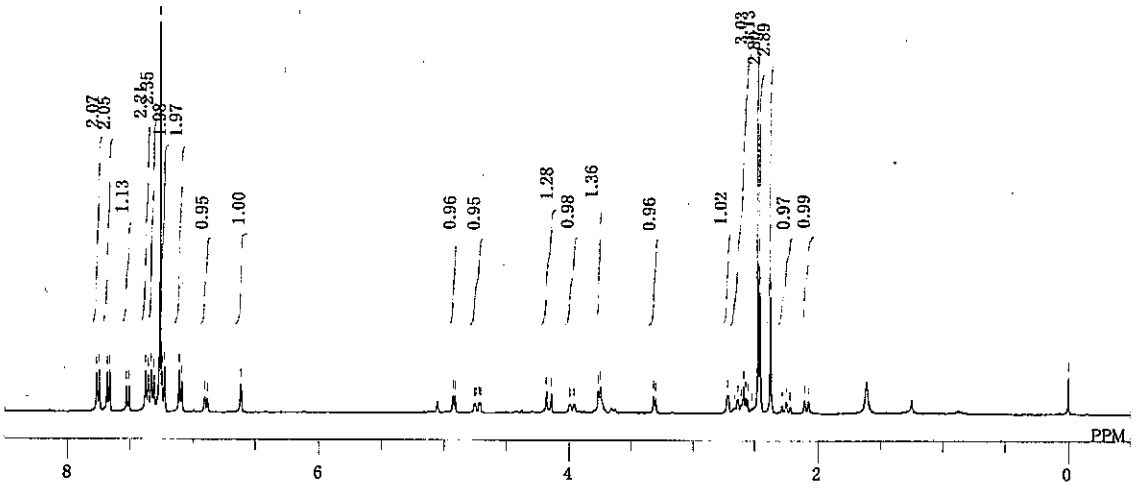
G:¥NMR¥横坂¥35¥YKTY35-56-Fr7-12_proton-2-1.als



DFILE	YKTY35-56-Fr7-12_proton-2-1
COMNT	single_pulse
DATIM	2013-09-12 15:07:54
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.40 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	21.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40

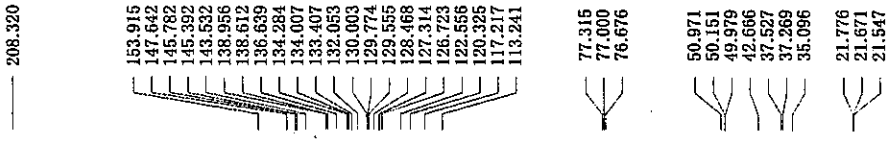


5c

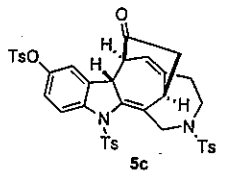


single pulse decoupled gated NOE

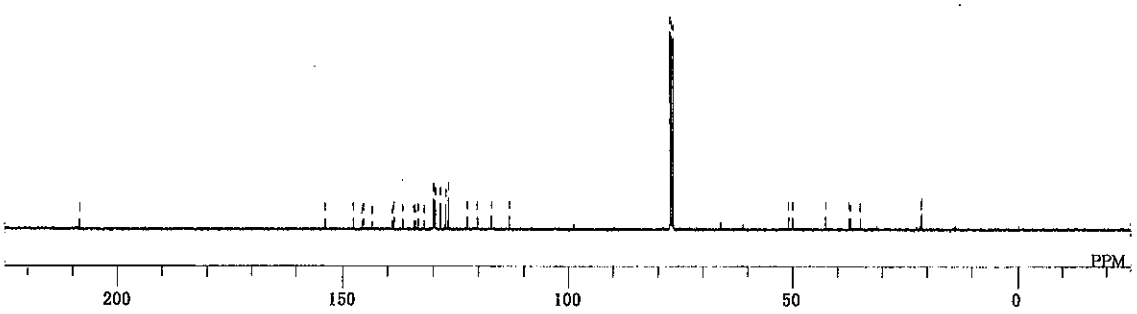
G:¥NMR¥横坂¥35¥YKTY35-56-Fr7-12_carbon-1-1.jdf



DFILE	YKTY35-56-Fr7-12_carbon-1-1
COMNT	single pulse decoupled gated NOE
DATIM	2013-09-12 00:10:41
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	613
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	20.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

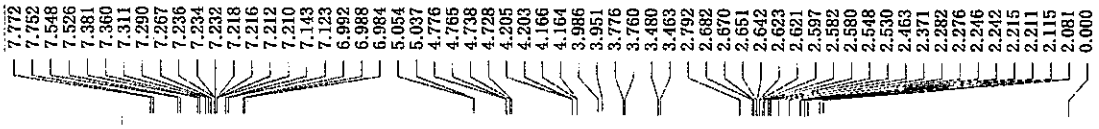


5c

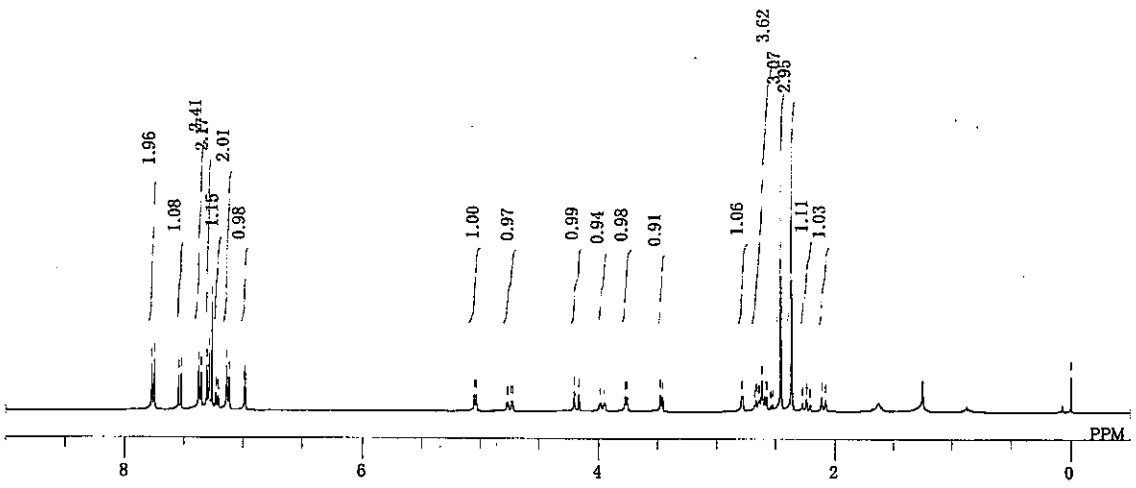


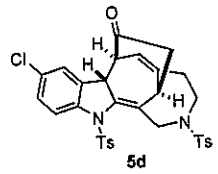
single_pulse

G:¥NMR¥横坂¥35¥YKTY35-85-Fr1-10_proton-2-1.als



DFILE	YKTY35-85-Fr1-10_proton-2-1.
COMNT	single_pulse
DATIM	2013-09-28 16:07:31
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.40 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	19.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38

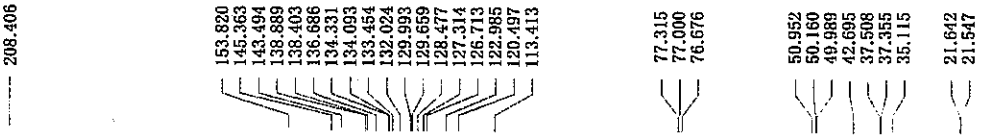




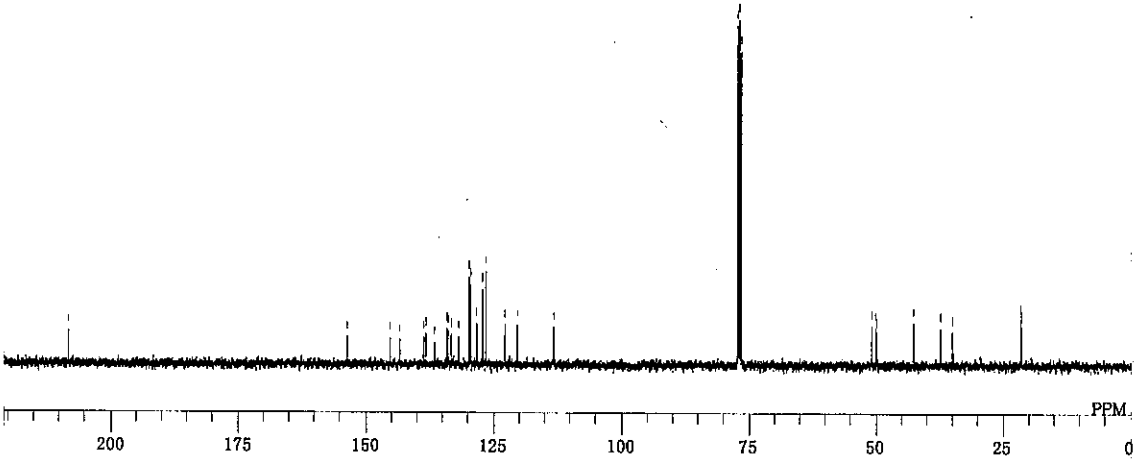
5d

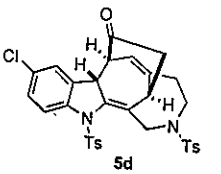
single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-85-Fr1-10_carbon-1-1.jdf



DFILE	YKTY35-85-Fr1-10_carbon-1-1
COMNT	single pulse decoupled gated NO
DATIM	2013-09-28 13:25:16
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	76
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	19.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

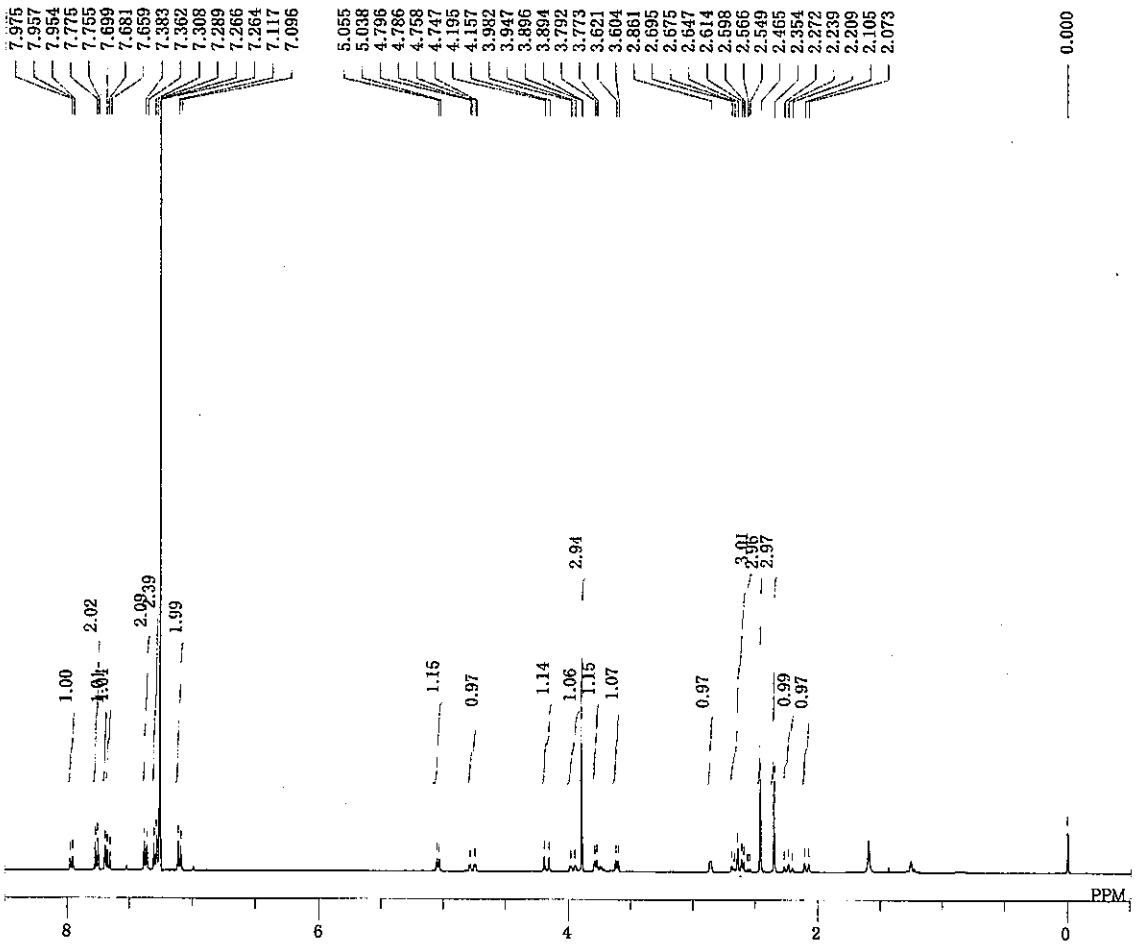




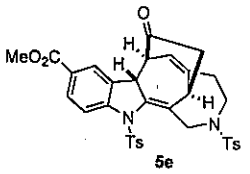
5d

single_pulse

G:¥NMR¥横坂¥35¥YKTY35-84_proton-2-1.als



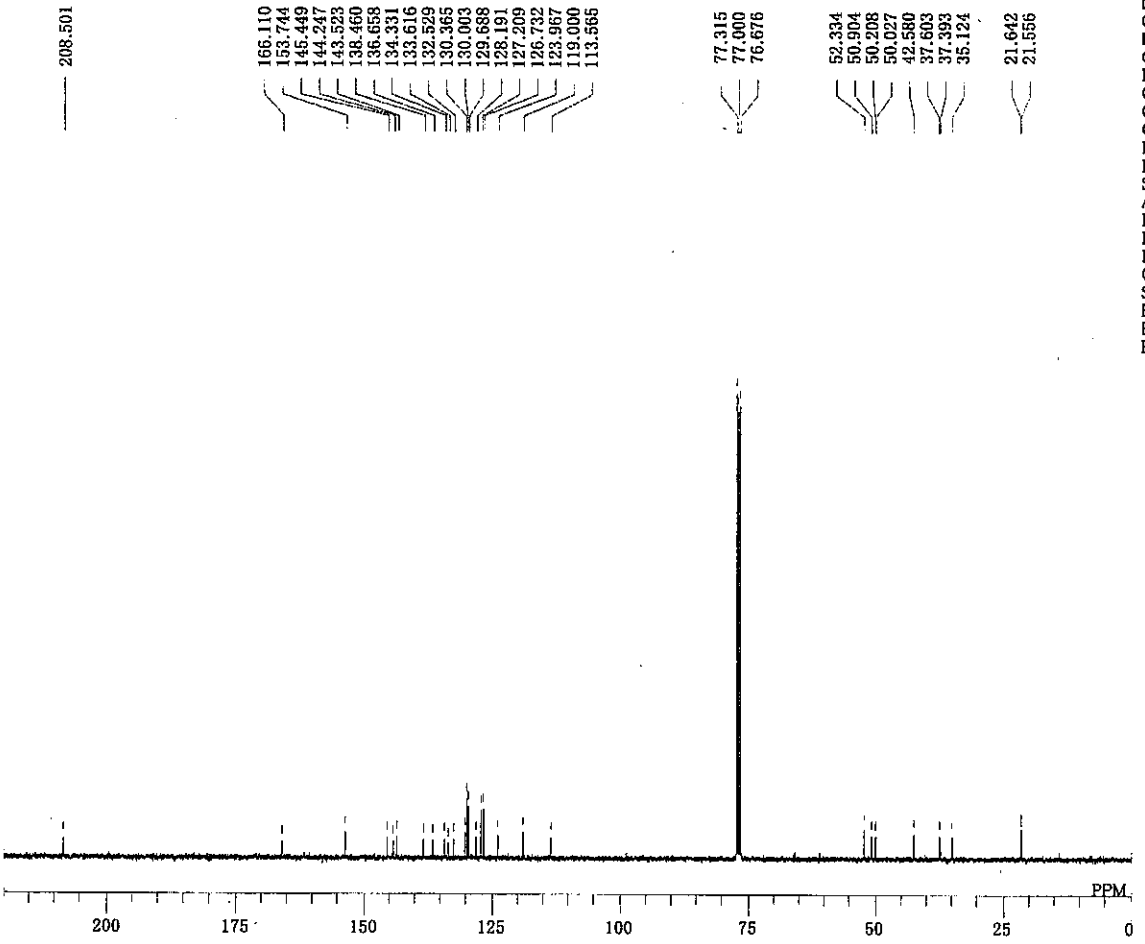
DFILE	YKTY35-84_proton-2-1.als
COMNT	single_pulse
DATIM	2013-09-28 23:14:35
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.40 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	42



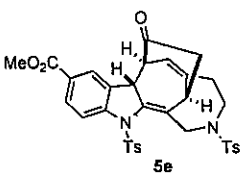
5e

single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-84_carbon-1-1.jdf



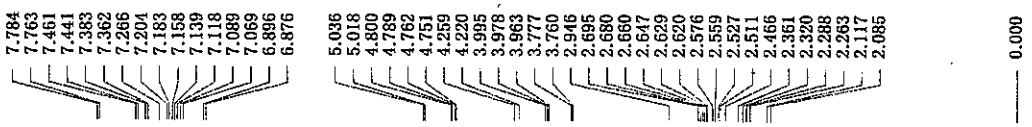
DFILE	YKTY35-84_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2013-09-28 21:53:54
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	507
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	19.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



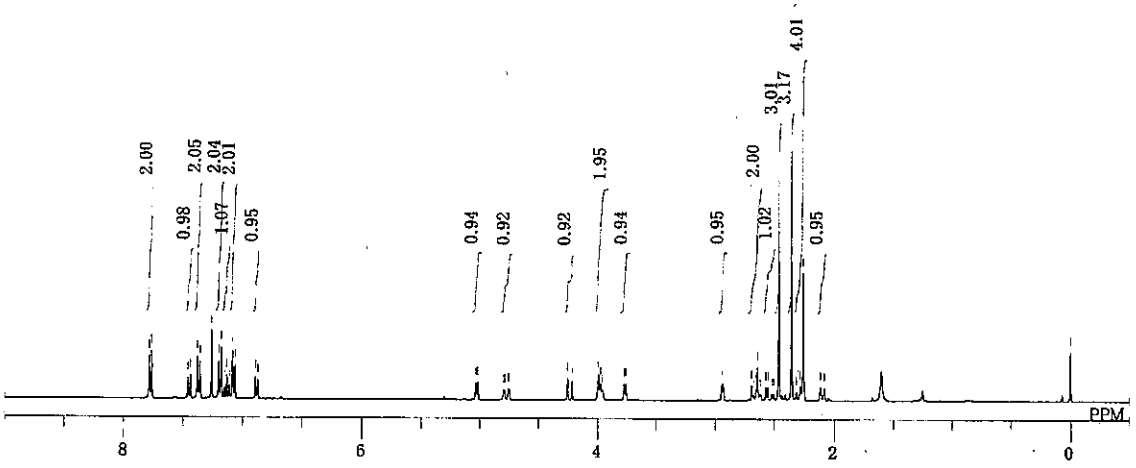
5e

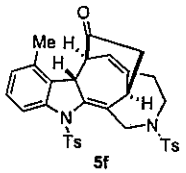
yk-hn-07-56-2_

G:¥NMR¥中山¥07¥yk-hn-07-56-2_proton-1-1.jdf



DFILE	yk-hn-07-56-2_proton-1-1.jdf
COMNT	yk-hn-07-56-2_
DATIM	2013-10-22 19:56:02
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	18.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	44

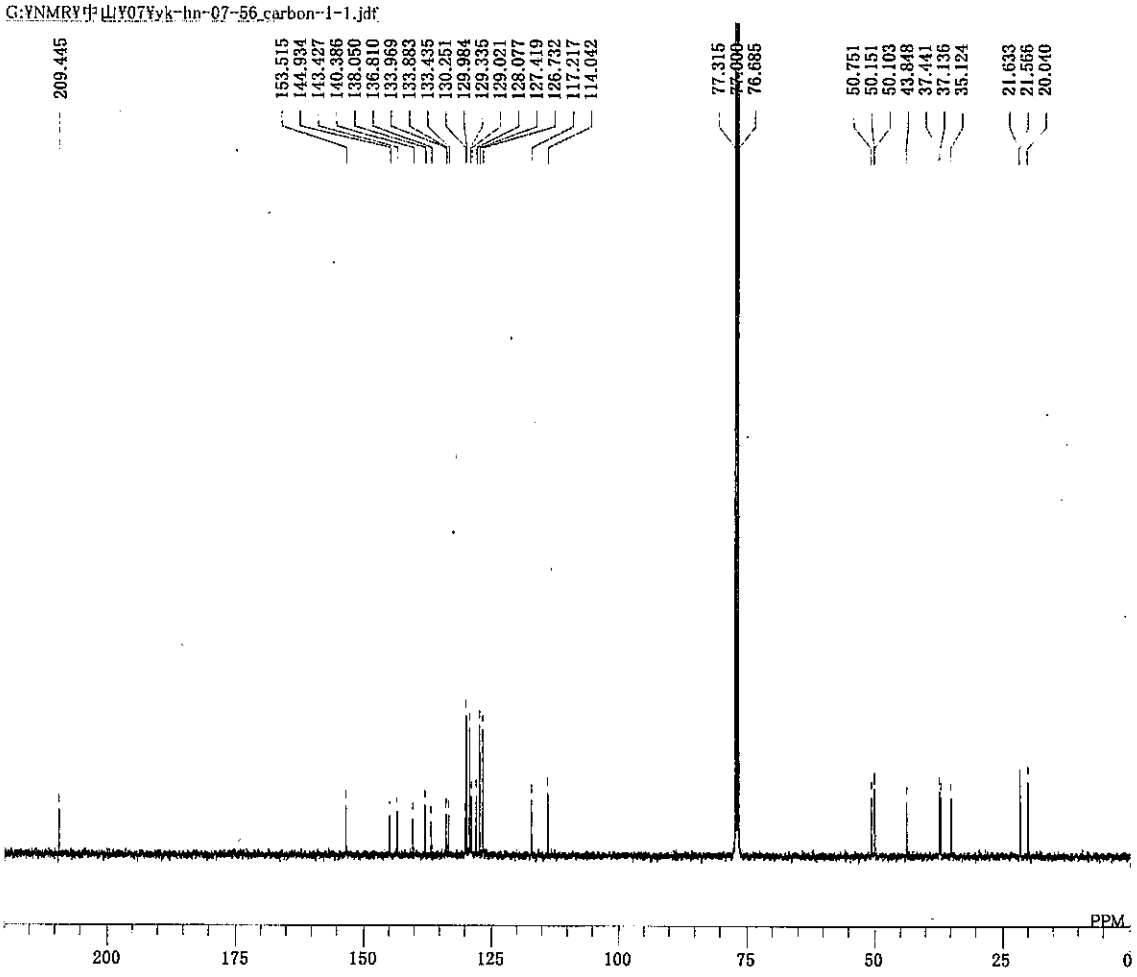




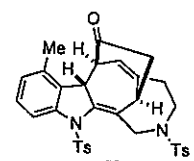
5f

yk-hn-07-56_

G:¥NMR¥中山¥07¥yk-hn-07-56_carbon-1-1.jdf



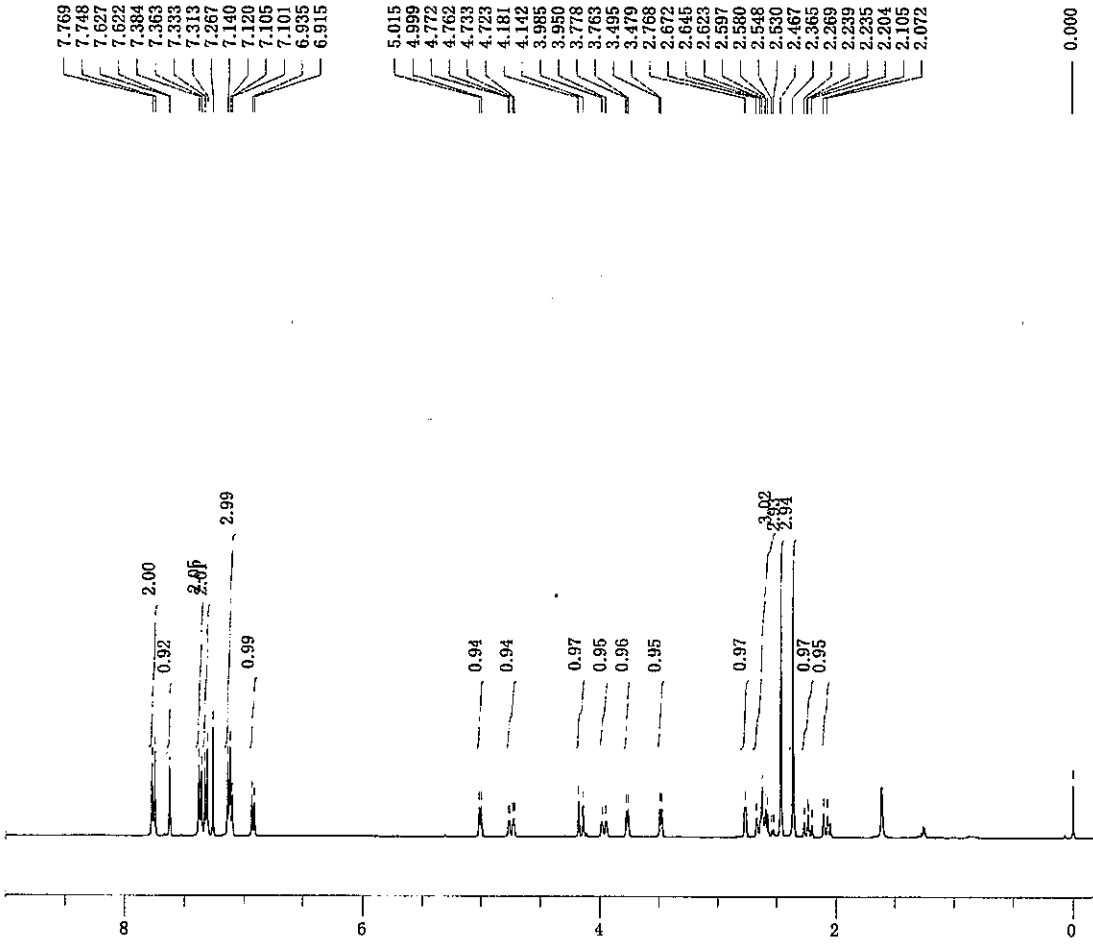
DFILE	yk-hn-07-56_carbon-1-1.jdf
COMNT	yk-hn-07-56_
DATIM	2013-10-21 23:22:02
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	2048
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	18.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



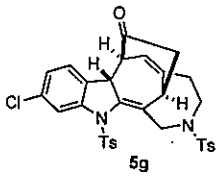
5f

yk-hn-07-53_

G:¥NMR¥中山¥07¥yk-hn-07-53-2_proton-1-1.jdf



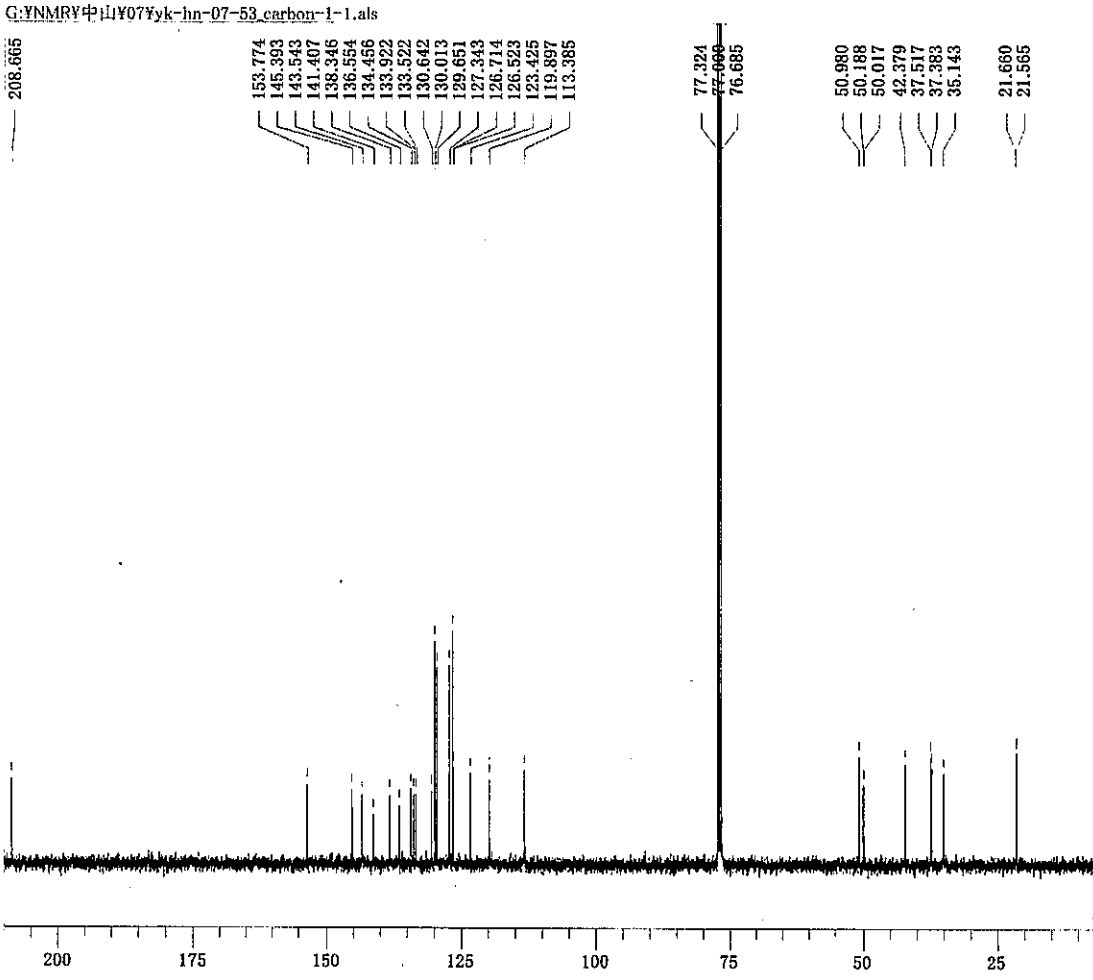
DFILE	yk-hn-07-53-2_proton-1-1.jdf
COMNT	yk-hn-07-53_
DATIM	2013-10-21 12:40:24
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	19.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	40



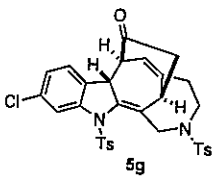
5g

yk-hn-07-53_yk-hn-07-53_proton

G:¥NMR¥中山¥07¥yk-hn-07-53_carbon-1-1.als



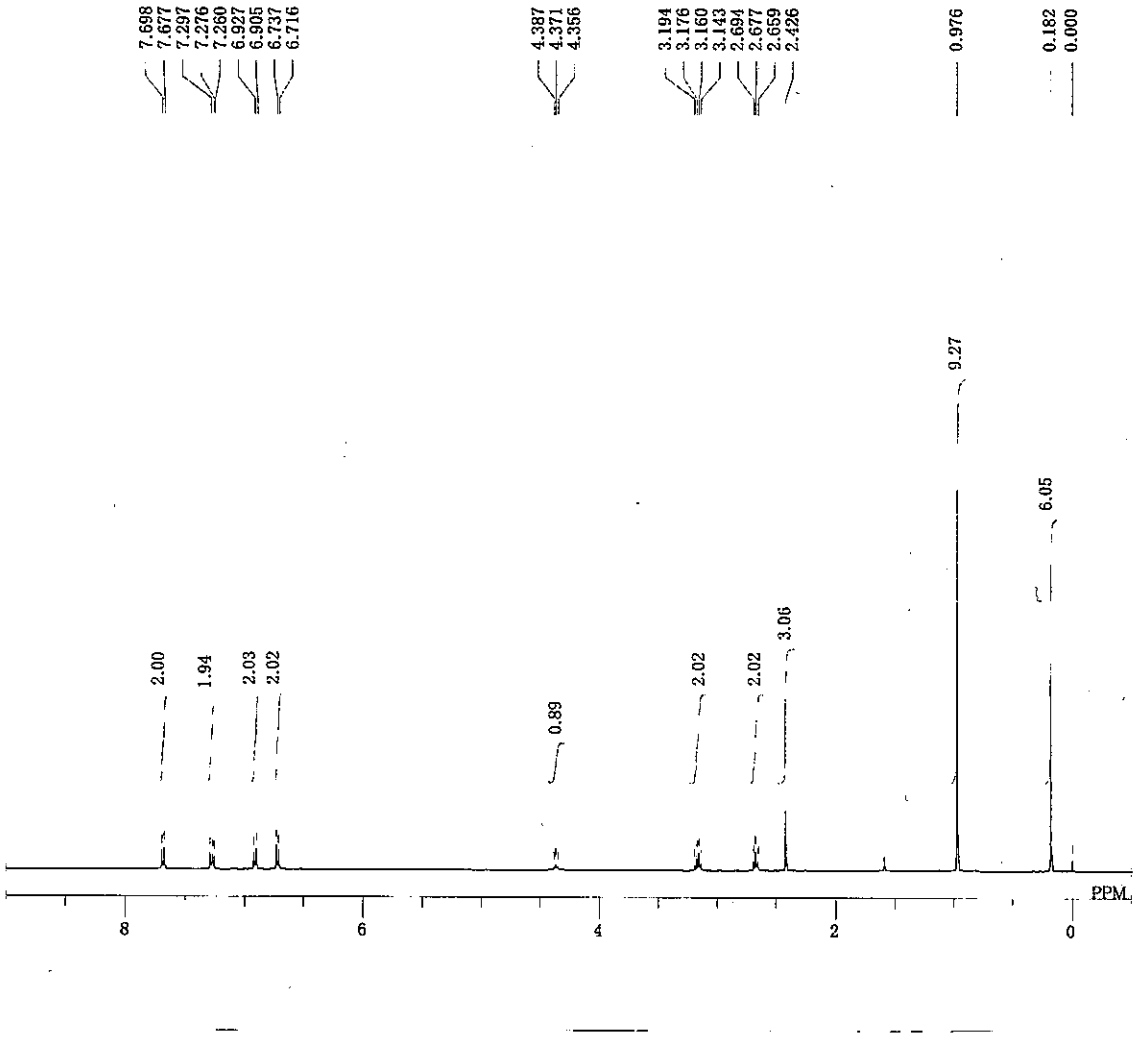
DFILE	yk-hn-07-53_carbon-1-1.als
COMNT	yk-hn-07-53_yk-hn-07-53_proto
DATIM	2013-10-21 12:42:30
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.63 Hz
SCANS	339
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	19.3 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



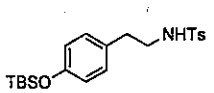
5g

single_pulse

G:¥NMR¥横坂¥28¥YKTY28-32_proton-3-1.als



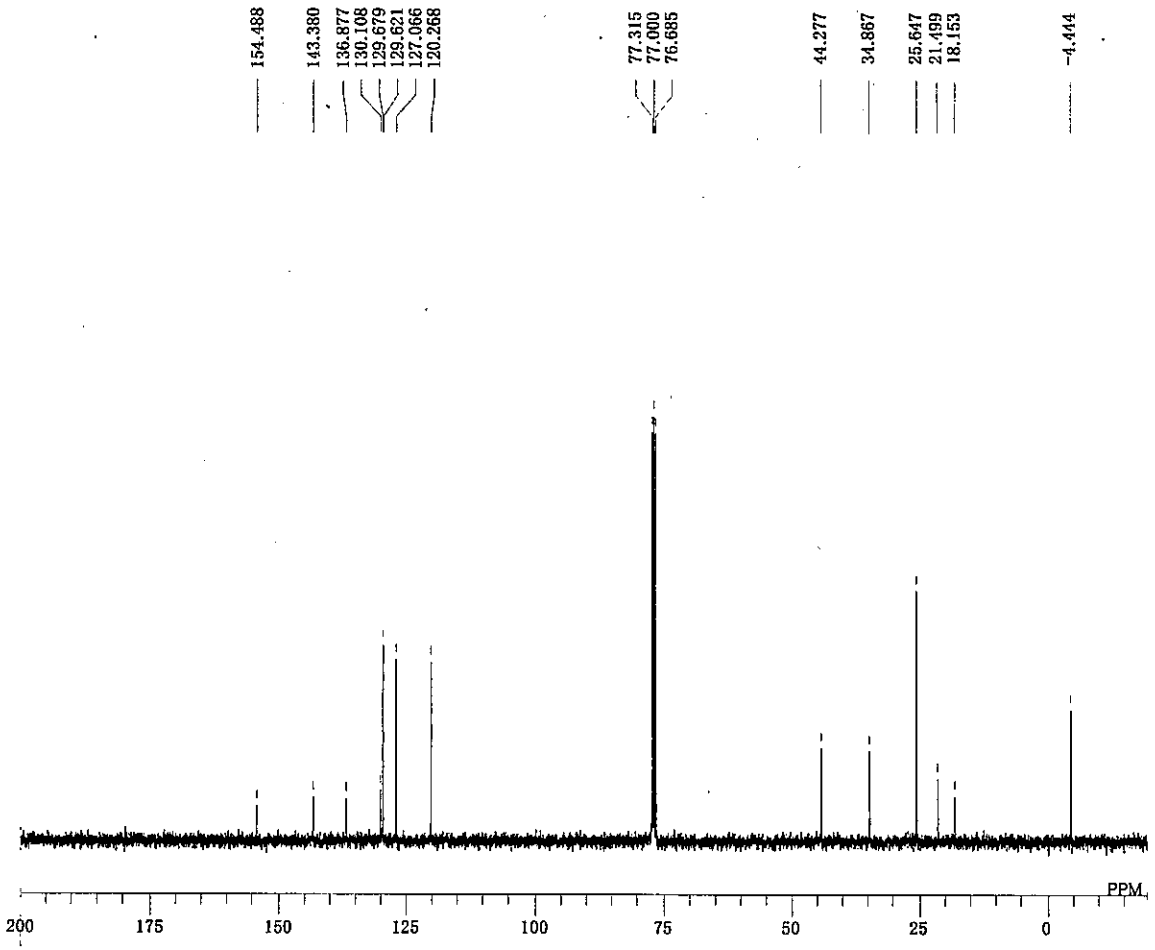
DFILE	YKTY28-32_proton-3-1.als
COMNT	single_pulse
DATIM	2013-06-13 23:55:36
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.40 Hz
SCANS	4
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	23.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	38



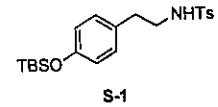
S-1

single pulse decoupled gated NOE

G:¥NMR¥横坂¥28¥YKTY28-32_carbon-1-1.jdf



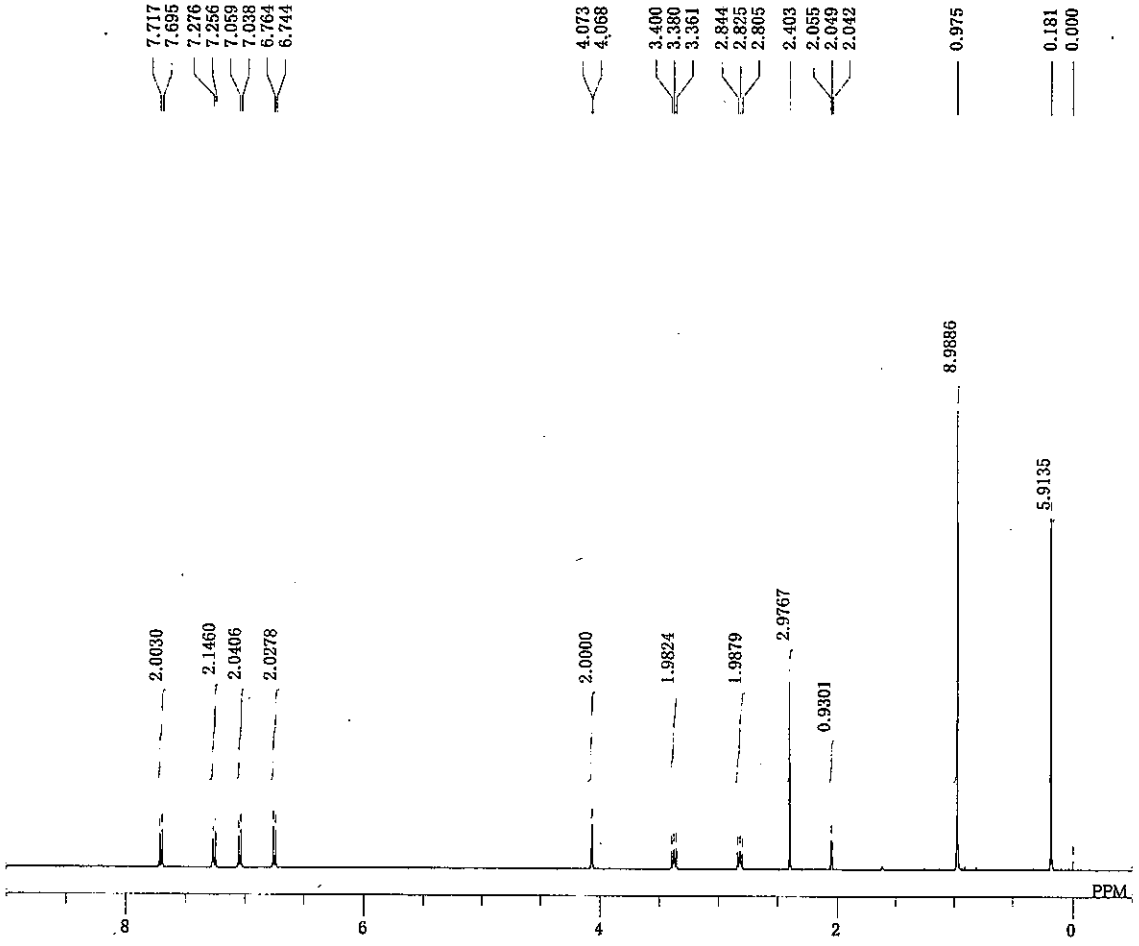
DFILE	YKTY28-32_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOI
DATIM	2013-06-13 23:20:41
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	96
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	23.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



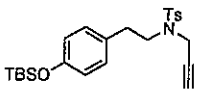
S-1

single_pulse

G:¥NMR¥横坂¥32¥YKTY32-75_proton-1-1.jdf



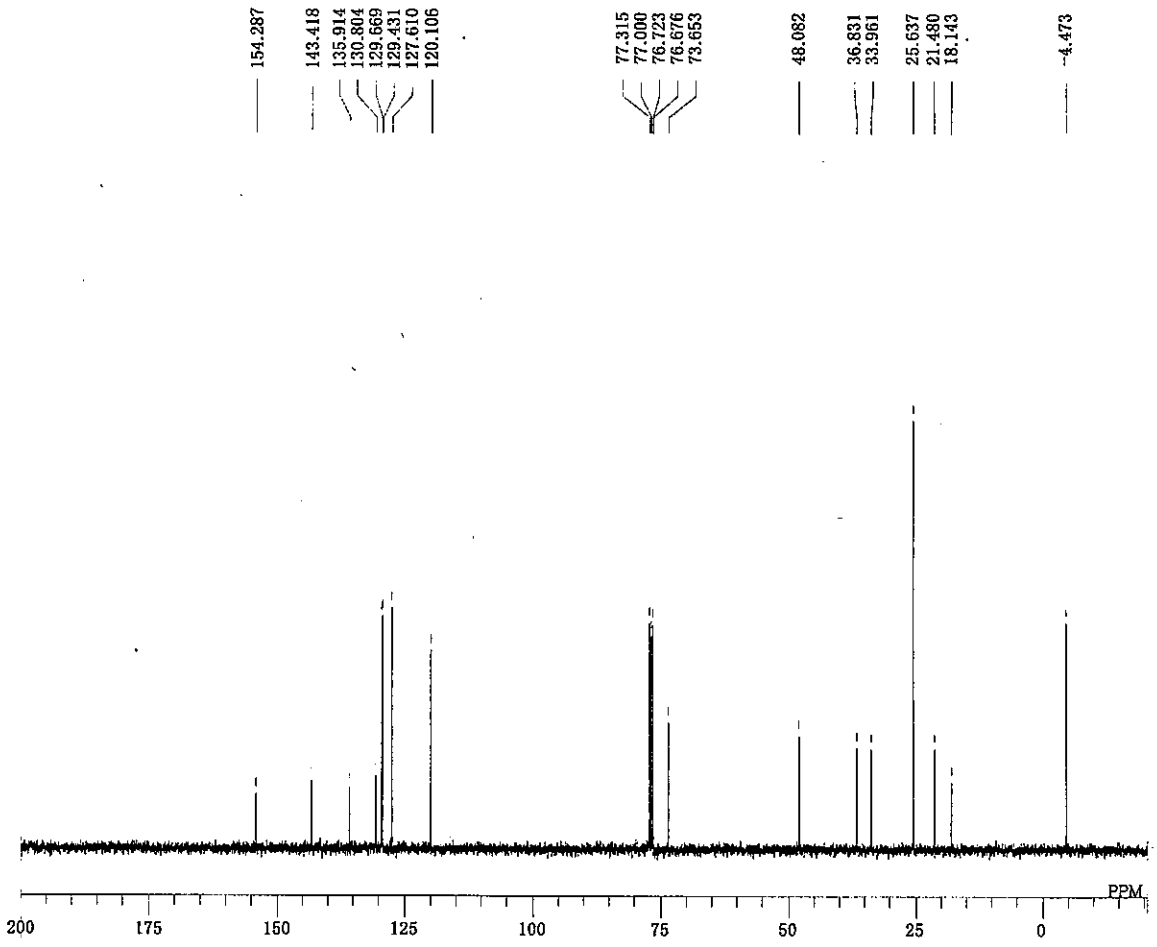
DFILE	YKTY32-75_proton-1-1.jdf
COMNT	single_pulse
DATIM	2013-06-10 14:19:27
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	4
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	24.2 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30



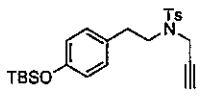
S-2

single pulse decoupled gated NOE

G:¥NMR¥横坂¥32¥YKTY32-75_carbon-1-1.jdf



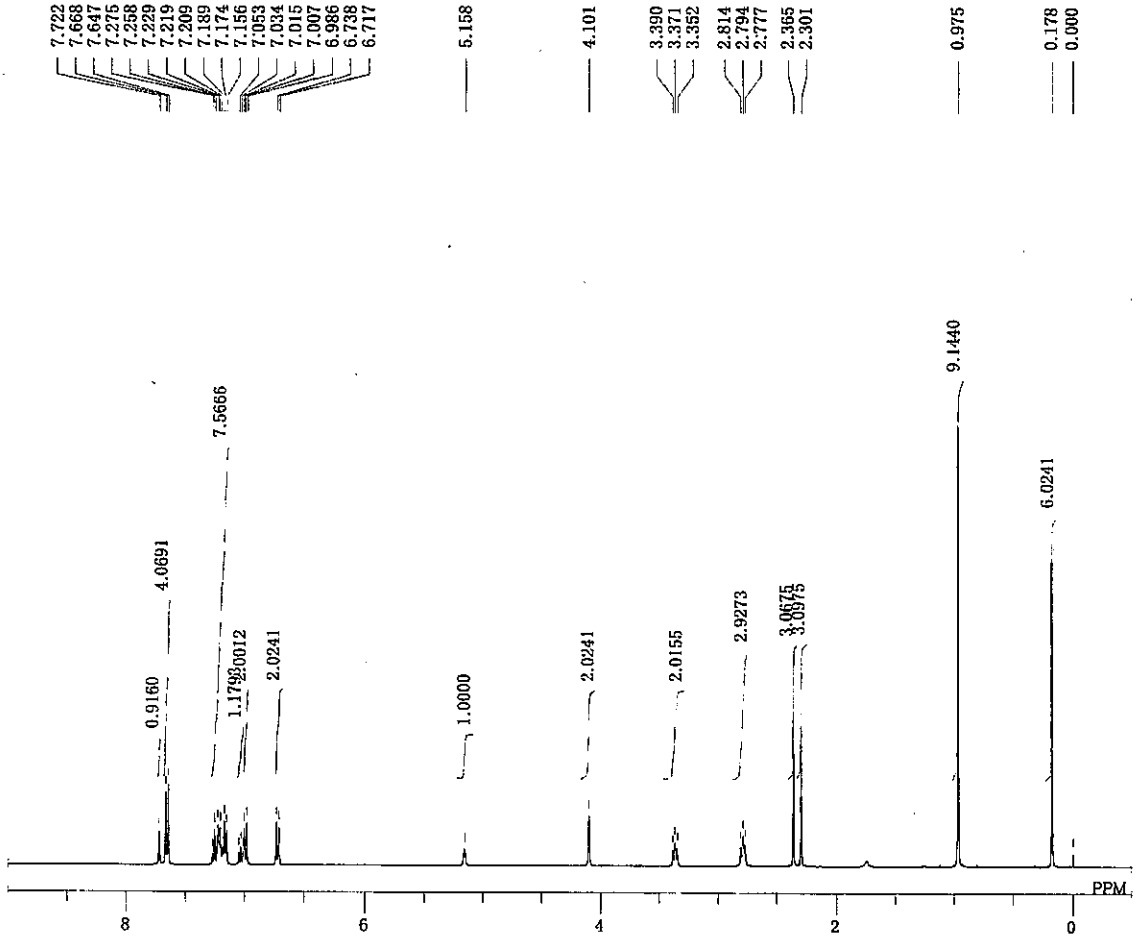
DFILE	YKTY32-75_carbon-1-1.jdf
COMNT	single pulse decoupled gated NO
DATIM	2013-06-10 14:22:01
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	28
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	24.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



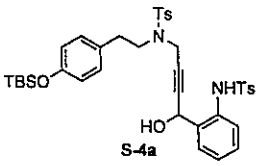
S-2

single_pulse

G:¥NMR¥横坂¥33¥YKTY33-52_proton-1-1.jdf



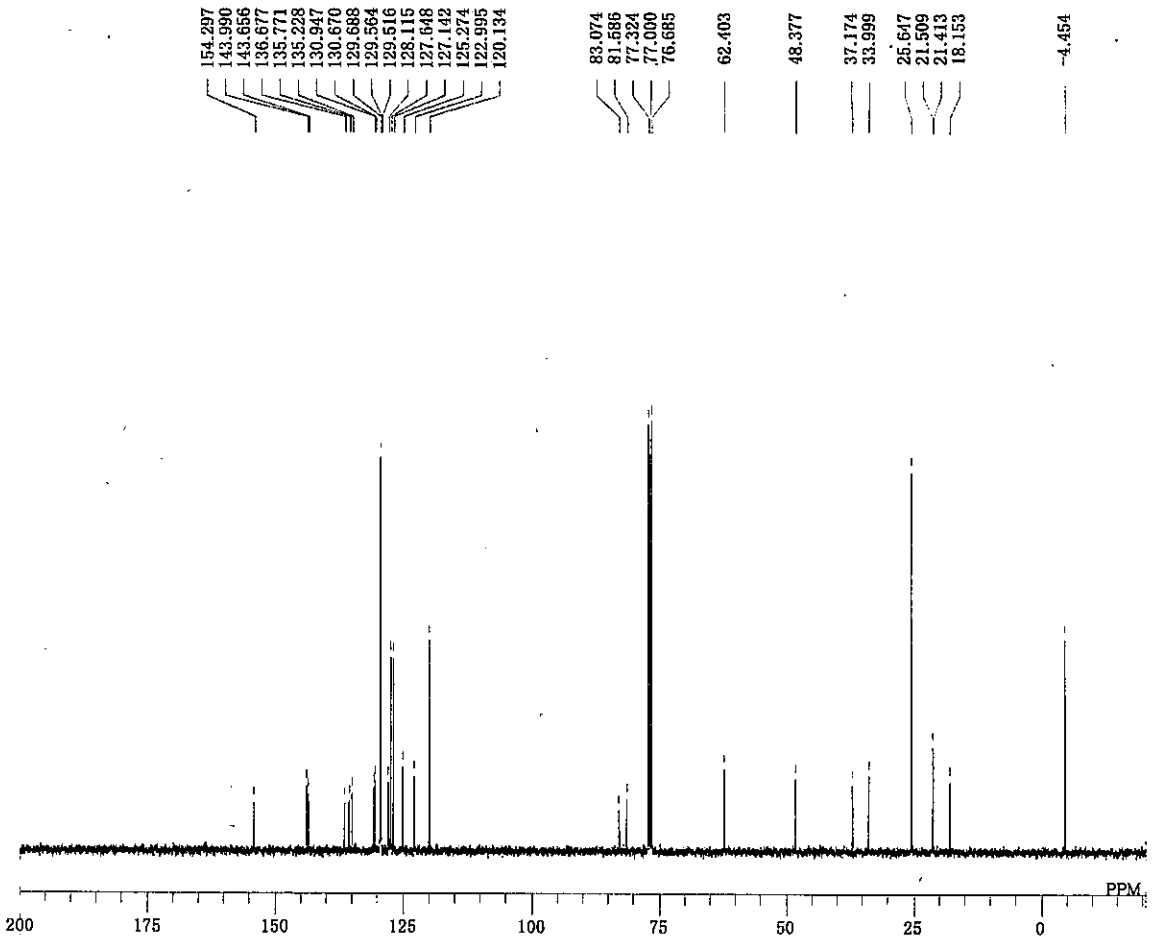
DFILE	YKTY33-52_proton-1-1.jdf
COMNT	single_pulse
DATIM	2013-06-14 11:53:07
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	23.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	32



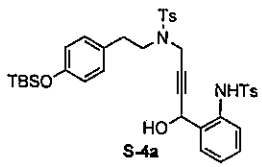
S-4a

single pulse decoupled gated NOE

G:¥NMR¥横坂¥33¥YKTY33-52_carbon-1-1.jdf



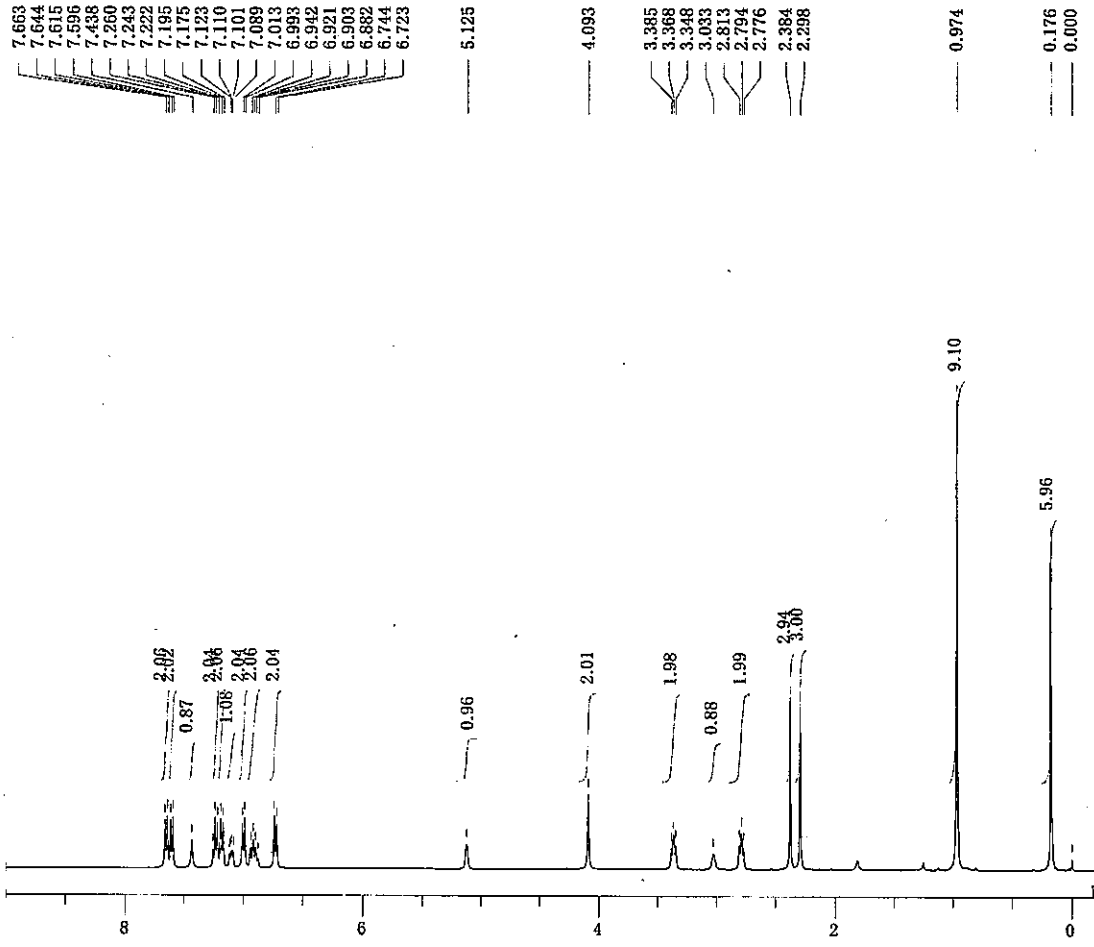
DFILE	YKTY33-52_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOI
DATIM	2013-06-14 11:56:09
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	162
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	23.8 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



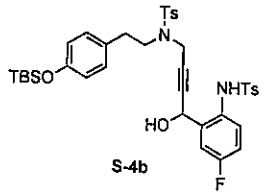
S-4a

single_pulse

G:¥NMR¥横坂¥35¥YKTY35-63_proton-1-1.jdf



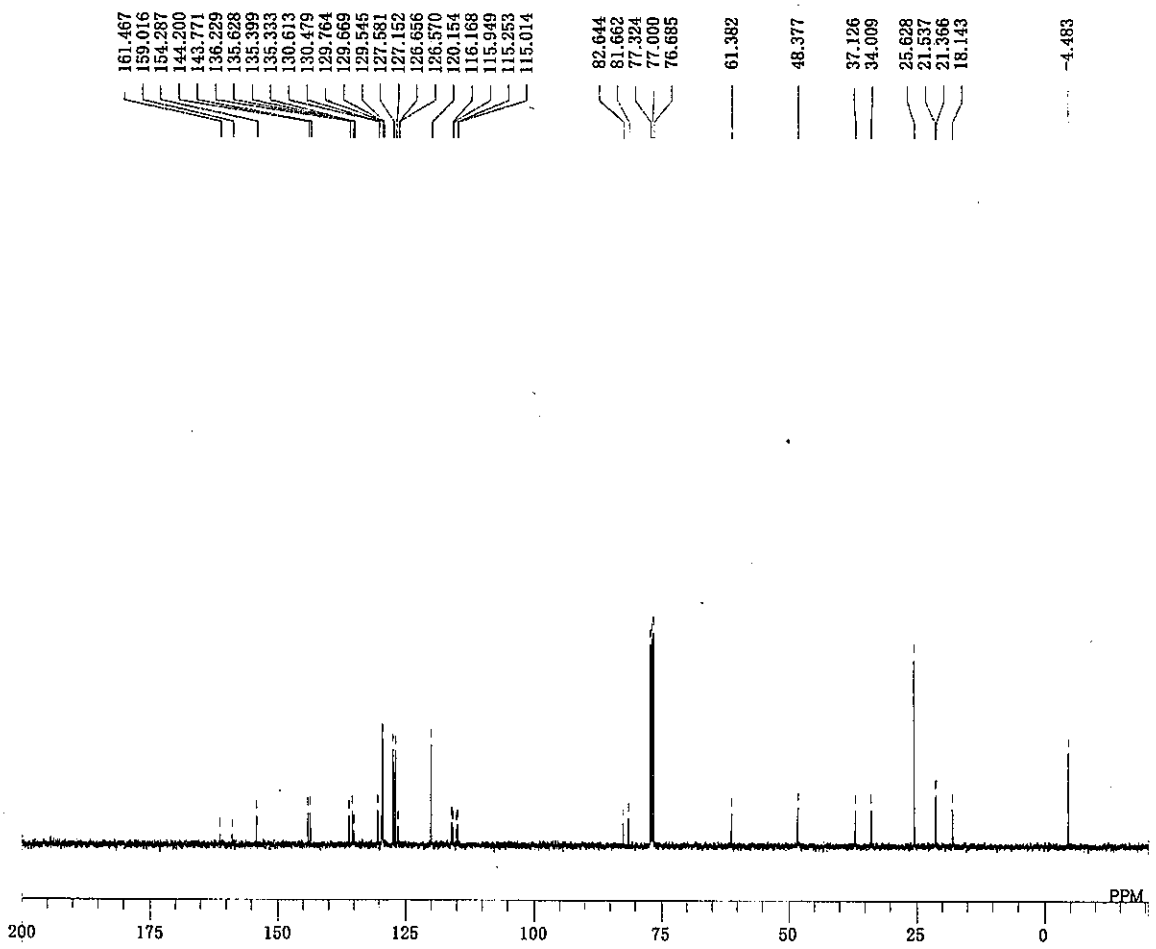
DFILE	YKTY35-63_proton-1-1.jdf
COMNT	single_pulse
DATIM	2013-09-17 13:16:06
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30



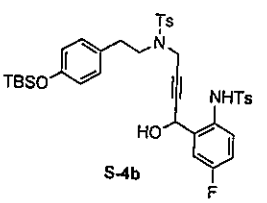
S-4b

single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-63_carbon-1-1.jdf



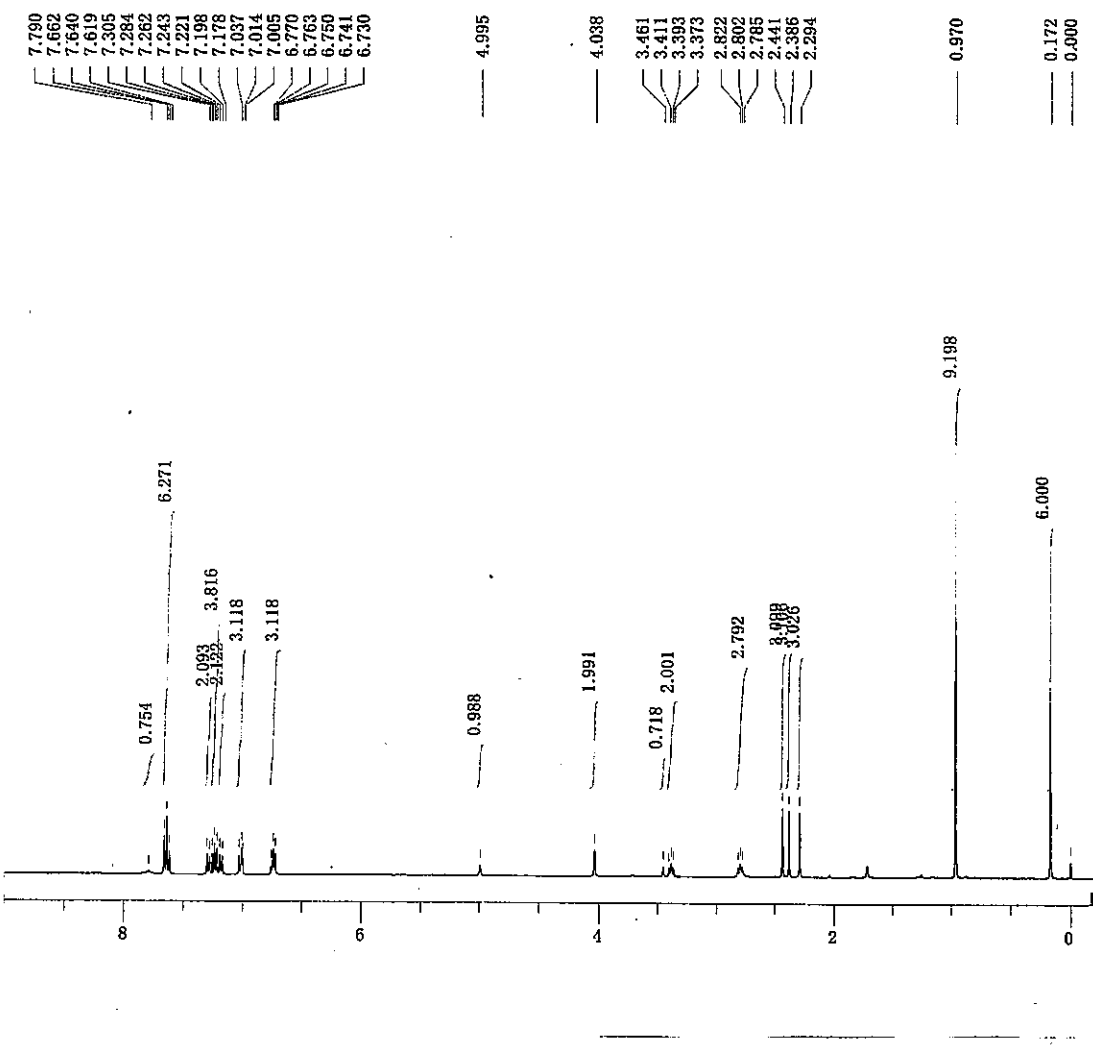
DFILE	YKTY35-63_carbon-1-1.jdf
COMNT	single pulse decoupled gated NOE
DATIM	2013-09-17 13:19:07
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	71
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



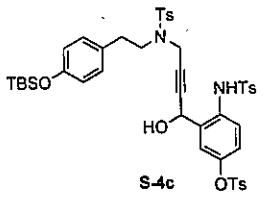
S-4b

single_pulse

G:¥NMR¥横坂¥35¥YKTY35-32_proton-1-1.jdf

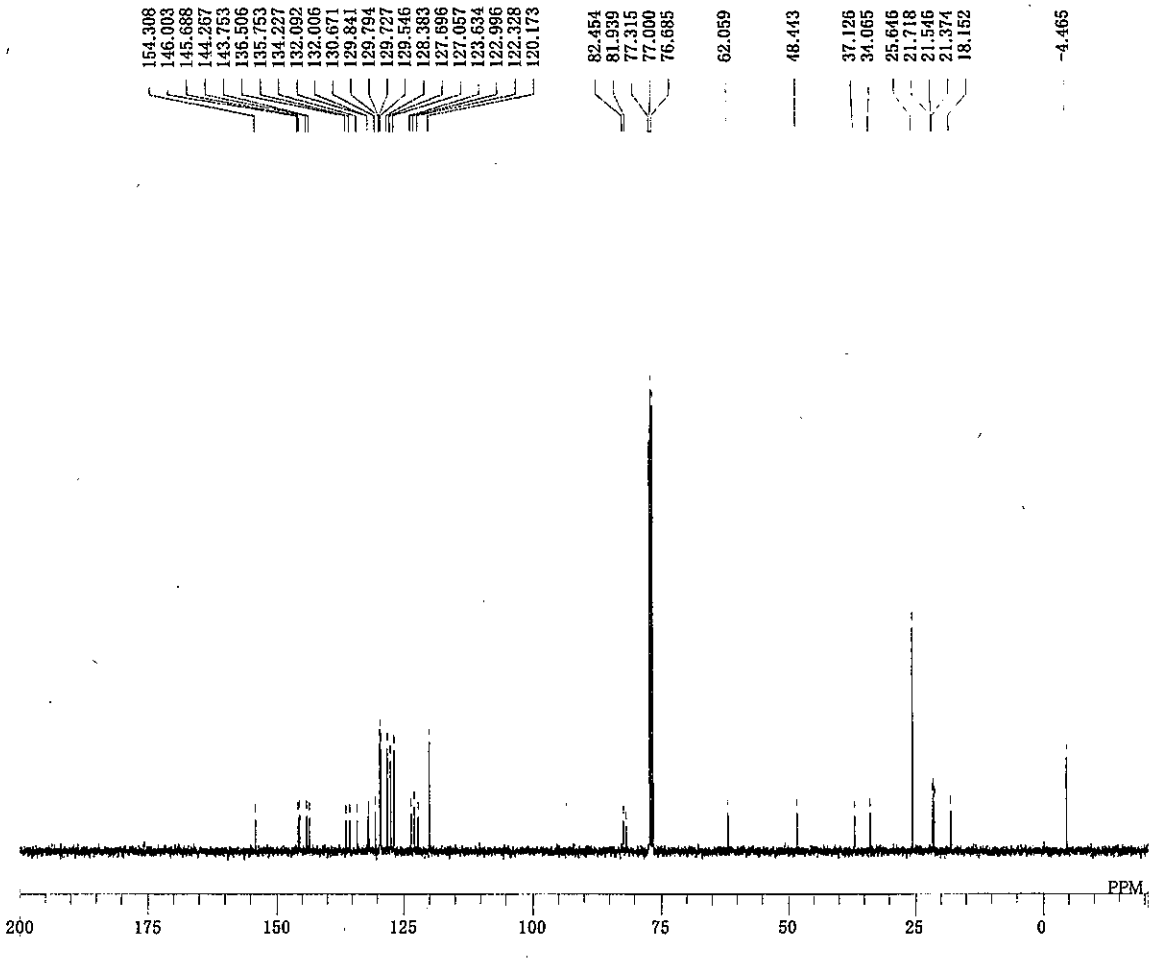


DFILE	YKTY35-32_proton-1-1.jdf
COMNT	single_pulse
DATIM	2013-09-05 16:47:45
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	22.0 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	36

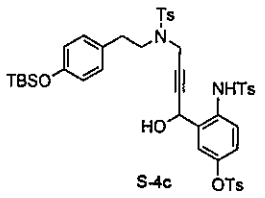


single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-32_carbon-1-1.als

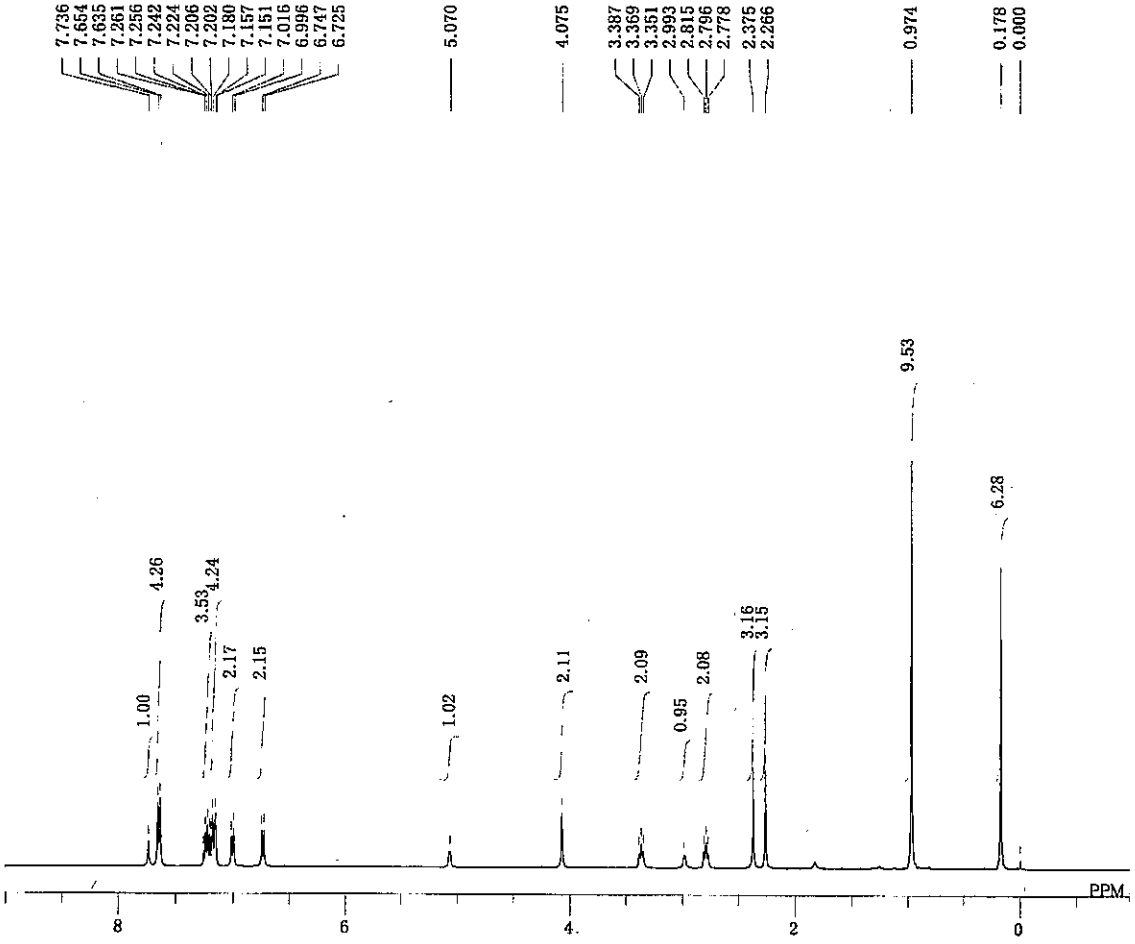


DFILE	YKTY35-32_carbon-1-1.als
COMNT	single pulse decoupled gated NOE
DATIM	2013-09-05 16:50:44
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.63 Hz
SCANS	148
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	22.4 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60

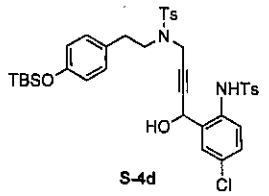


single_pulse

G:¥NMR¥横坂¥35¥YKTY35-58_proton-2-1.als



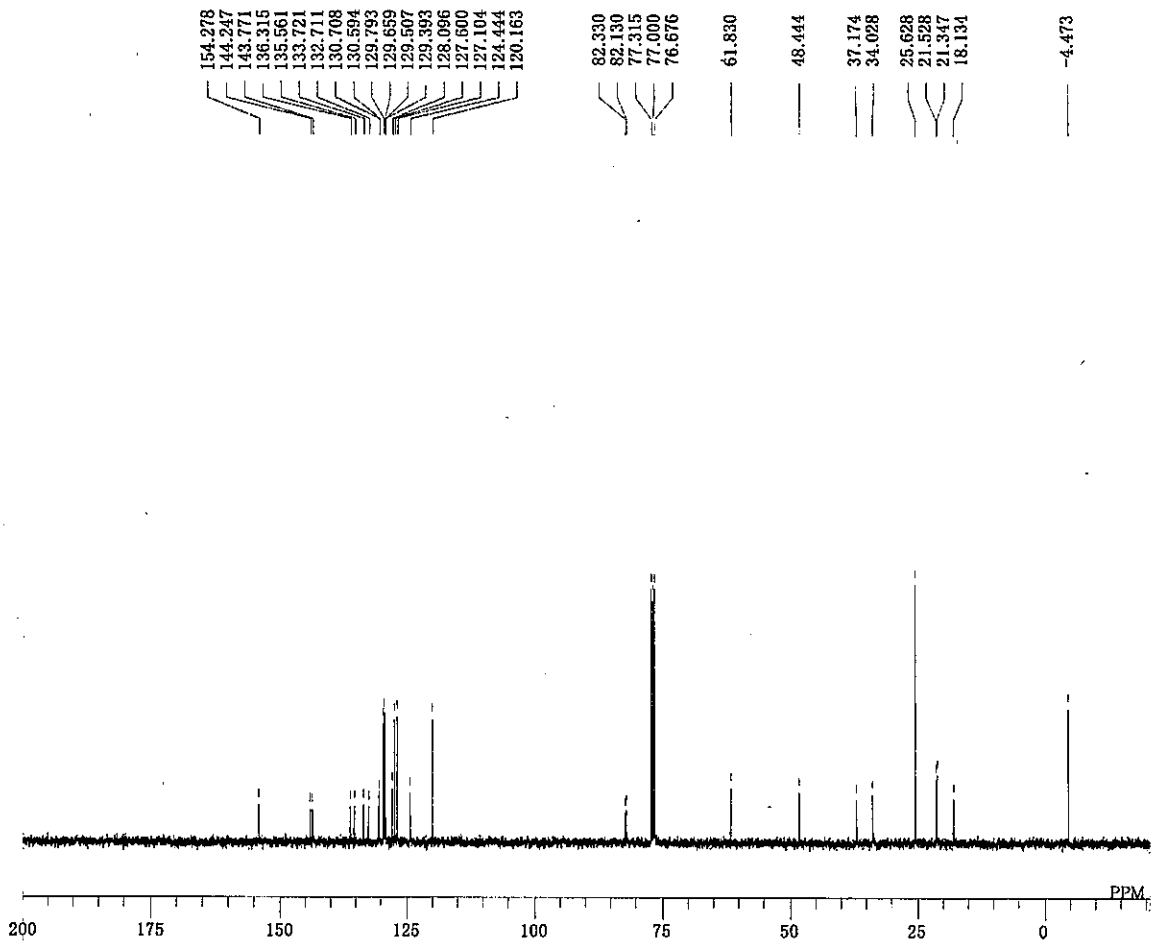
DFILE	YKTY35-58_proton-2-1.als
COMNT	single_pulse
DATIM	2013-09-17 13:33:38
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.40 Hz
SCANS	4
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30



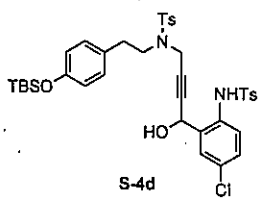
S-4d

single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-58_carbon-1-1.jdf



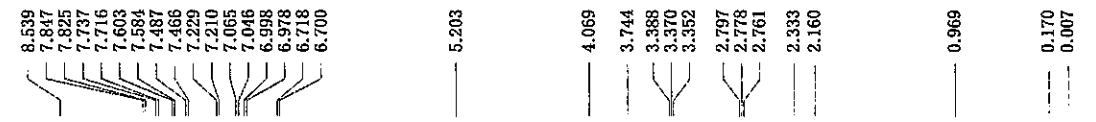
DFILE	YKTY35-58_carbon-1-1.jdf
COMNT	single pulse decoupled gated NO
DATIM	2013-09-17 13:28:21
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	58
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	20.5 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



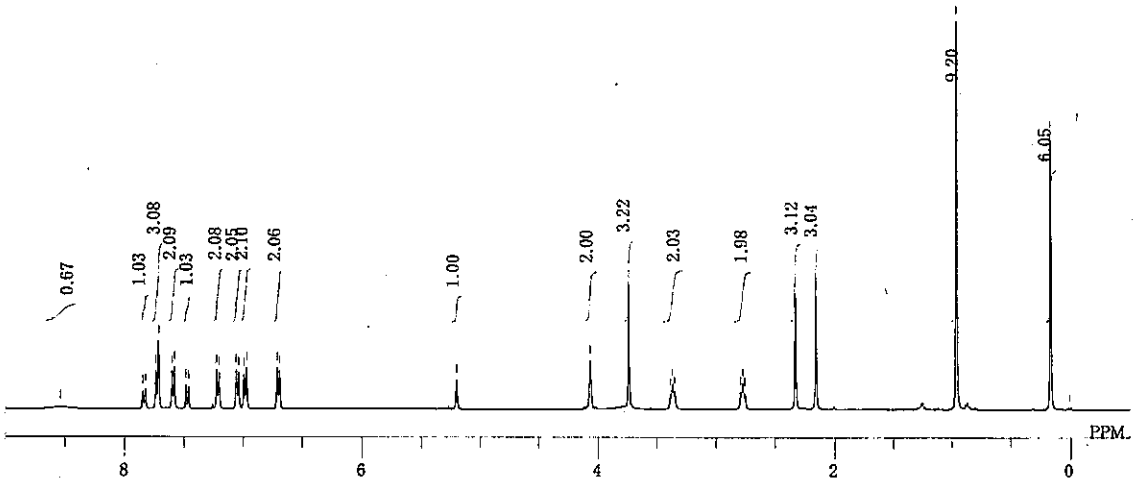
S-4d

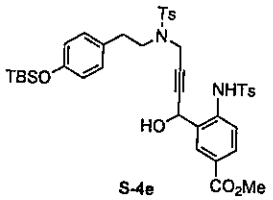
single_pulse

G:¥NMR¥横坂¥35¥YKTY35-69-Fr32-42_proton-1-1.als



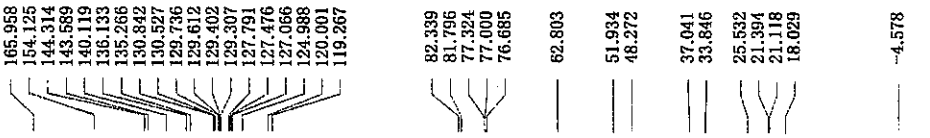
DFILE	YKTY35-69-Fr32-42_proton-1-1
COMNT	single_pulse
DATIM	2013-09-19 11:57:50
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.40 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	19.7 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	20



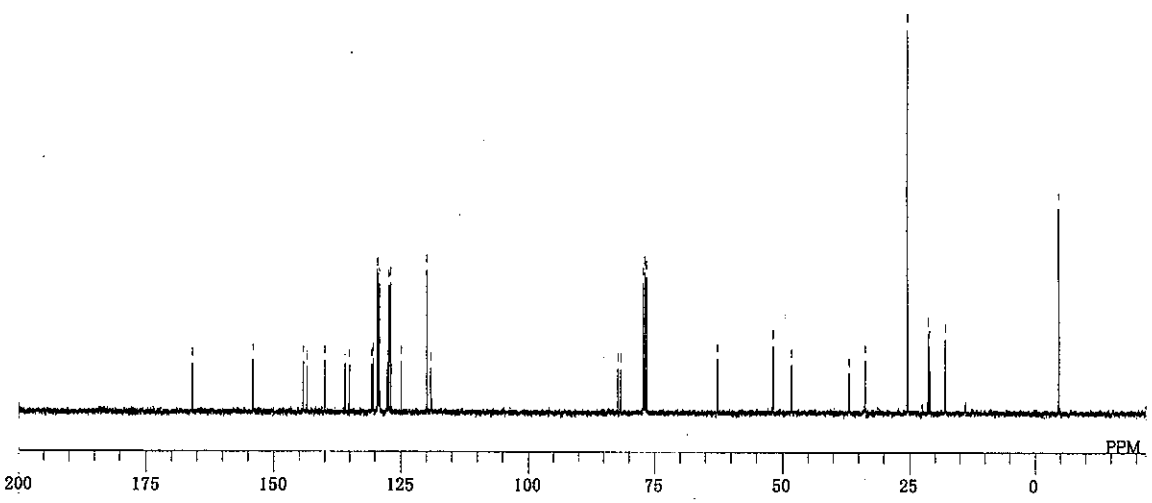


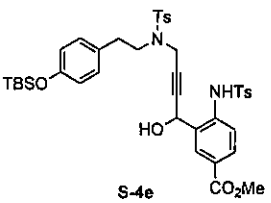
single pulse decoupled gated NOE

G:¥NMR¥横坂¥35¥YKTY35-69-Fr32-42_carbon-1-1.jdf



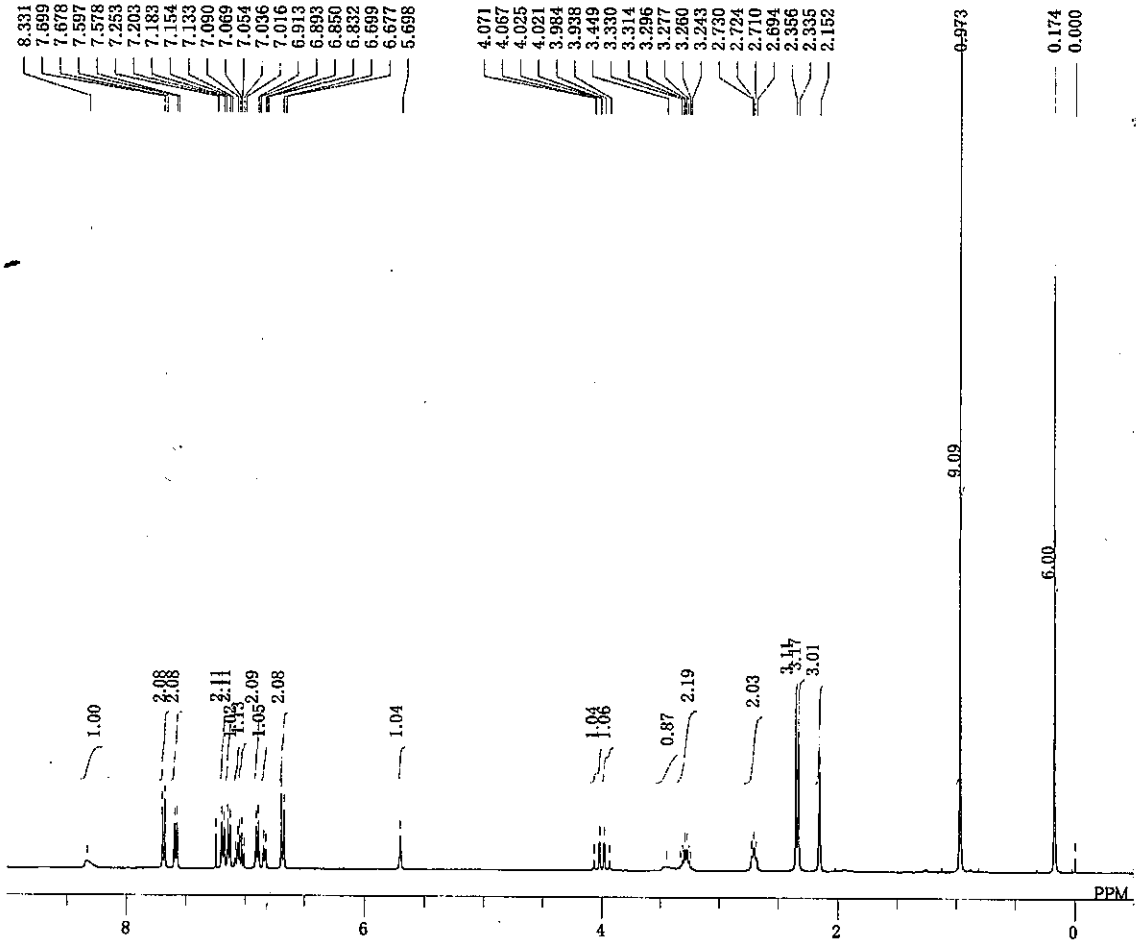
DFILE	YKTY35-69-Fr32-42_carbon-1-1
COMNT	single pulse decoupled gated NOE
DATIM	2013-09-19 12:00:52
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	32
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	19.9 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



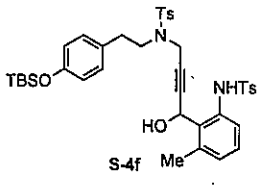


yk-hn-07-45_

G:¥NMR¥中山¥07¥yk-hn-07-45-2_proton-1-1.als



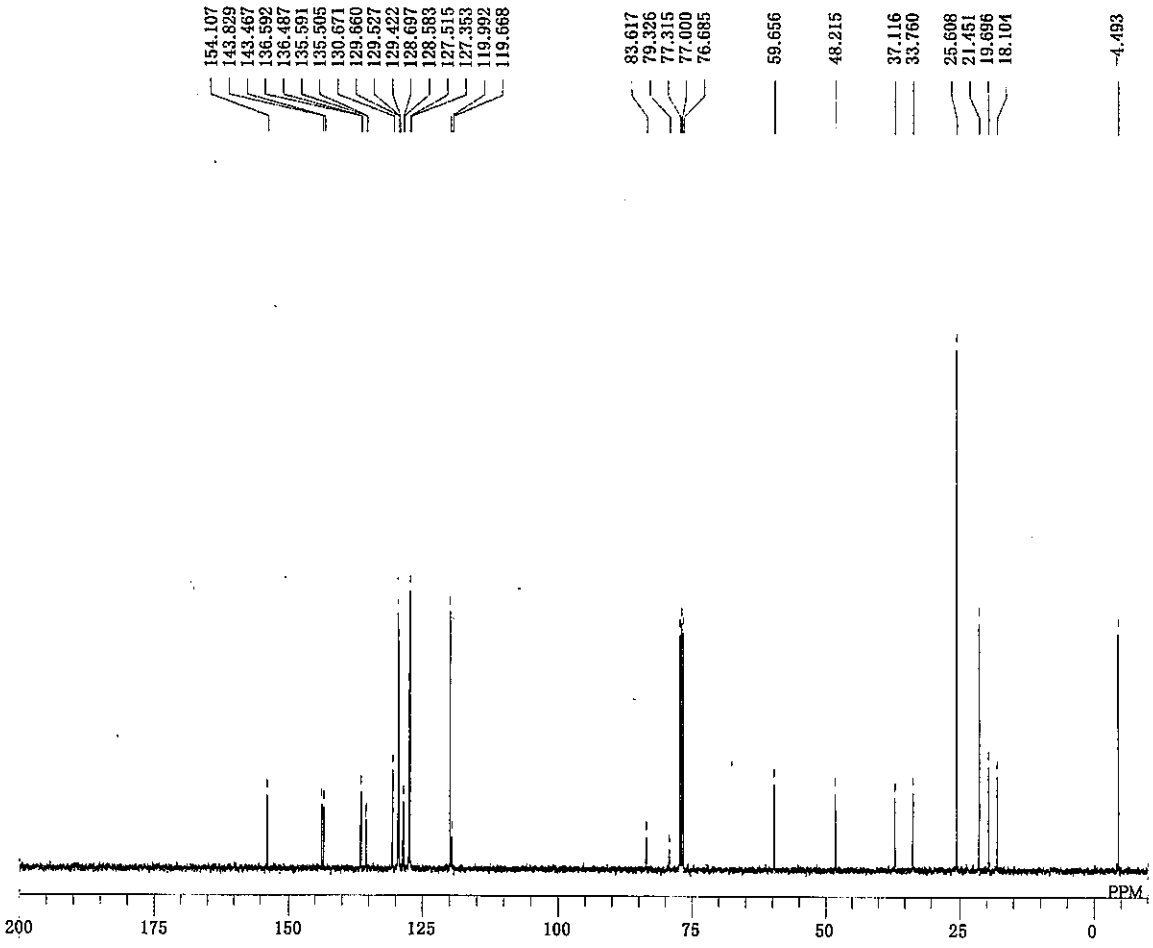
DFILE	yk-hn-07-45-2_proton-1-1.als
COMNT	yk-hn-07-45_
DATIM	2013-09-27 19:59:09
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	13107
FREQU	6002.40 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	26



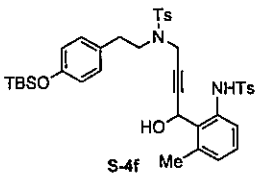
S-4f

yk-hn-07-45_yk-hn-07-45_proton

G:¥NMR¥中山¥07¥yk-hn-07-45_carbon-1-1.als



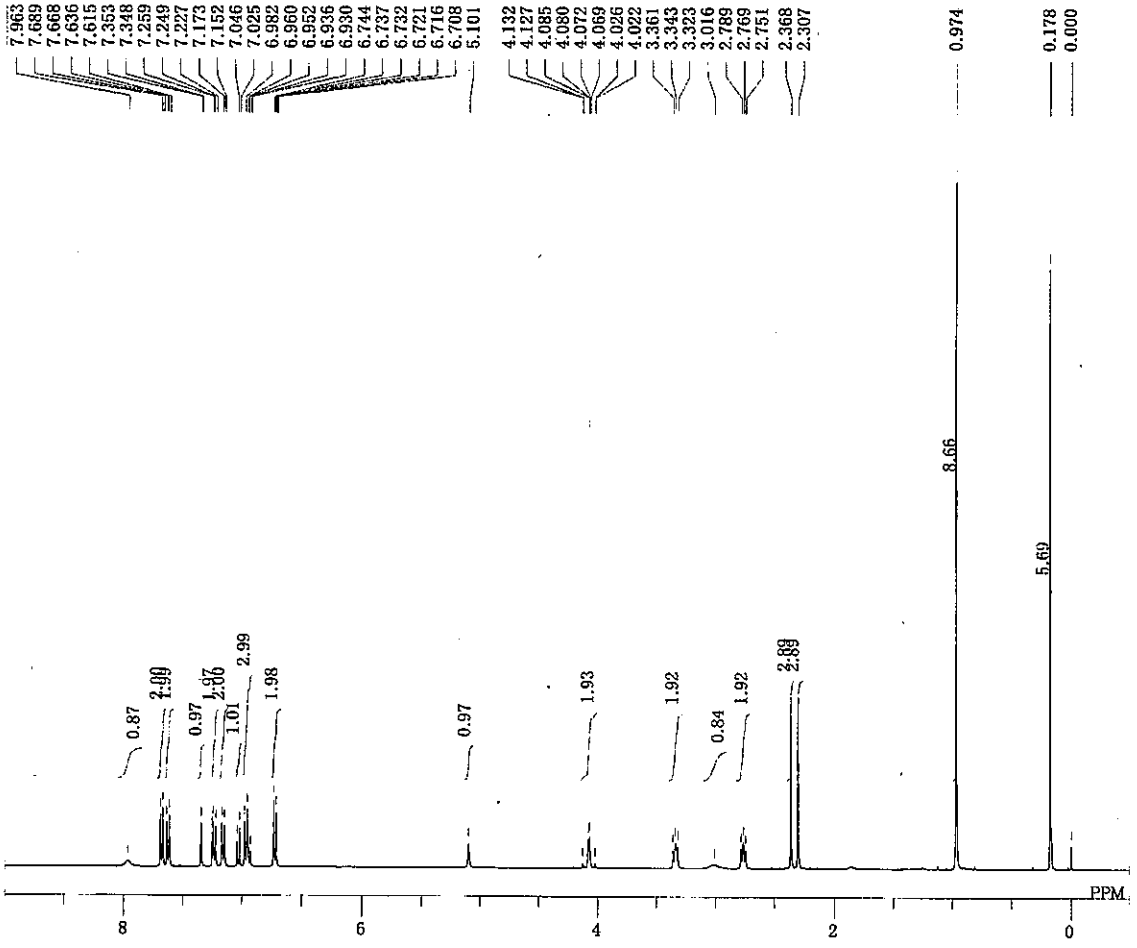
DFILE	yk-hn-07-45_carbon-1-1.als
COMNT	yk-hn-07-45_yk-hn-07-45_proto
DATIM	2013-09-27 20:02:10
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	26214
FREQU	25125.63 Hz
SCANS	124
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	19.6 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



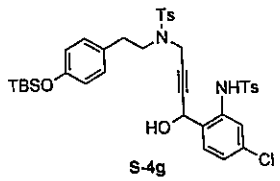
S-4f

yk-hn-07-44_

G:¥NMR¥中山¥07¥yk-hn-07-44-2_proton-1-1.jdf



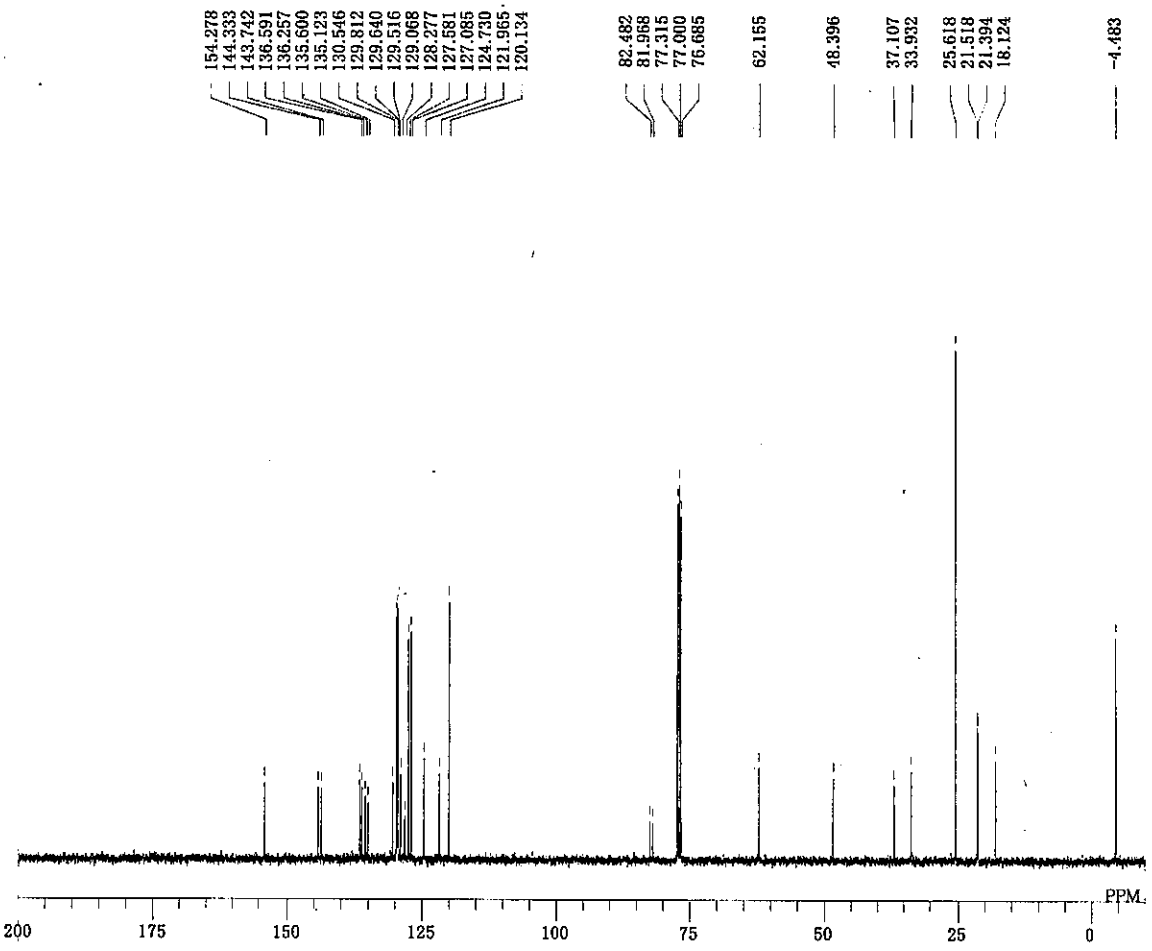
DFILE	yk-hn-07-44-2_proton-1-1.jdf
COMNT	yk-hn-07-44_
DATIM	2013-09-27 19:45:52
OBNUC	1H
EXMOD	proton.jxp
OBFRQ	399.78 MHz
OBSET	4.19 KHz
OBFIN	7.29 Hz
POINT	16384
FREQU	7503.00 Hz
SCANS	8
ACQTM	2.1837 sec
PD	5.0000 sec
PW1	5.01 usec
IRNUC	1H
CTEMP	20.3 c
SLVNT	CDCL3
EXREF	0.00 ppm
BF	0.12 Hz
RGAIN	30



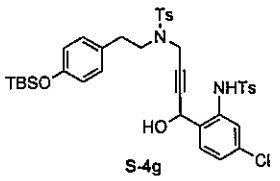
S-4g

yk-hn-07-44_yk-hn-07-44_proton

G:¥NMR¥中山¥07¥yk-hn-07-44_carbon-1-1.jdf



DFILE	yk-hn-07-44_carbon-1-1.jdf
COMNT	yk-hn-07-44_yk-hn-07-44_protc
DATIM	2013-09-27 19:48:54
OBNUC	13C
EXMOD	carbon.jxp
OBFRQ	100.53 MHz
OBSET	5.35 KHz
OBFIN	5.86 Hz
POINT	32767
FREQU	31407.04 Hz
SCANS	124
ACQTM	1.0433 sec
PD	2.0000 sec
PW1	3.02 usec
IRNUC	1H
CTEMP	20.0 c
SLVNT	CDCL3
EXREF	77.00 ppm
BF	1.20 Hz
RGAIN	60



S-4g