Asymmetric Epoxidation of 1,1-Disubstituted Terminal Olefins by Chiral Dioxirane via a Planar-like Transition State

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Supporting Information

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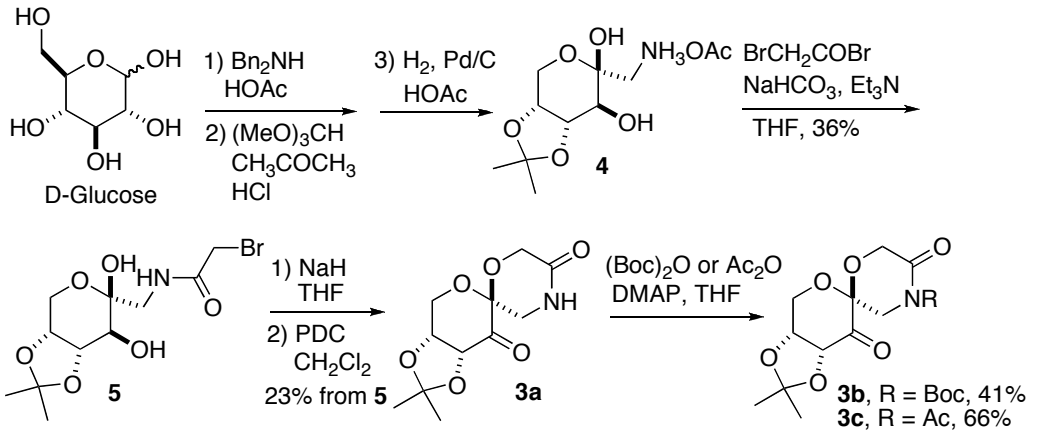
Synthesis and characterization of ketones 3a-c



To a mixture of the crude amino salt **4** (prepared from D-glucose as previously reported[a]) (14.47 g, 52.0 mmol) in THF (250 mL) was added $NaHCO_3$ (8.7 g, 104.0 mmol). After the resulting mixture was stirred at rt for 1 h, Et_3N (6.83 g, 9.46 mL, 67.6 mmol) was added, followed by a dropwise addition of 2-bromoacetyl bromide (13.65 g, 67.6 mmol) in THF (20 mL) at rt within 1 h. Upon stirring at rt for 6 h, the reaction mixture was filtered and concentrated to give a brown crude syrup which was purified by flash chromatography (silica gel, hexanes /EtOAc = 1/4 to 0/1) to give diol **5** as a light brown solid (6.4 g, 36% yield): mp 89-91 °C; $[\alpha]_D^{25}$ = -105.0 (*c* 0.60, MeOH); IR (film) 3343, 1733 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 4.28-4.17 (m, 4H), 3.98 (d, *J* = 13.6 Hz, 1H), 3.92 (s, 2H), 3.62 (dd, *J* = 14.0, 7.2 Hz, 1H), 3.57-3.48 (m, 2H), 1.53 (s, 3H), 1.38 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.7, 109.4, 96.8, 76.4, 73.4, 71.7, 60.3, 47.4, 28.6, 28.2, 26.2; HRMS Calcd for $C_{11}H_{16}BrNO_5$ (M-H_2O): 321.0212; Found: 321.0211. ([a] Shu, L.; Shen, Y-M.; Burke, C.; Goeddel, D.; Shi, Y. *J. Org. Chem.* **2003**, *68*, 4963.)

To a solution of diol **5** (1.85 g, 5.4 mmol) in THF (30 mL) was carefully added NaH (95%, 0.301 g, 11.9 mmol) in portions. Upon stirring at rt for 0.5 h, the reaction mixture was quenched with MeOH (0.58 mL), concentrated, and dried under vaccum to give a yellow syrup.

To a mixture of the above yellow syrup in dry DCM (50 mL) was added PDC (4.06 g, 10.8 mmol), 3Å MS (2.5 g), and 4 drops of AcOH. Upon stirring at rt for 4 d (TLC showed no

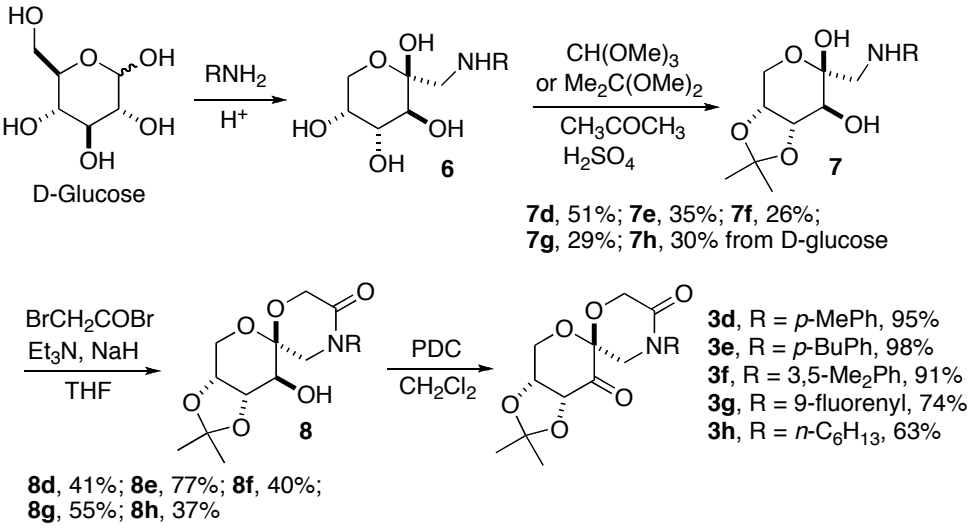
alcohol left), the reaction mixture was filtered through a pad of silica gel, and the filter cake was washed by EtOAC/MeOH (10/1). Upon removal of solvent, the mixture was purified by flash chromatography (silica gel, EtOAc) to give ketone **3a** as a white solid (0.327 g, 23% yield): mp 186-187 ºC; $[\alpha]_D^{25}$ = -61.3 (*c* 0.50, $CHCl_3$); IR (film) 3200, 1749, 1692 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 6.01 (s, 1H), 4.82 (d, *J* = 5.6 Hz, 1H), 4.63 (d, *J* = 5.6, Hz, 1H), 4.32 (d, *J* = 16.4 Hz, 1H), 4.26 (d, *J* = 16.4 Hz, 1H), 4.21 (s, 2H), 4.03 (d, *J* = 13.2 Hz, 1H), 3.37 (dd, *J* = 13.2, 4.4 Hz, 1H), 1.48 (s, 3H), 1.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.6, 166.7, 111.0, 95.1, 78.4, 75.6, 62.5, 59.9, 44.1, 27.4, 26.3; Anal. Calcd. for $C_{11}H_{15}NO_6$: C, 51.36; H, 5.88. Found: C, 51.52; H, 5.88.

To a solution of ketone **3a** (0.171 g, 0.67 mmol) and DMAP (0.0008 g, 0.0065 mmol) in THF (10 mL) was added Boc anhydride (0.160 g, 0.737 mmol). After the resulting mixture was stirred at rt for 1 d, additional amount of Boc anhydride (0.160 g, 0.737 mmol) was added, and the mixture was stirred for another day at rt (TLC showed most of SM disappeared). The reaction mixture was concentrated and purified by flash chromatography (silica gel, first hexanes/EtOAc = 2/1, then EtOAc) to give ketone **3b** as a colorless syrup (0.1 g, 41% yield) plus some recovered ketone **3a**: $[\alpha]_D^{25}$ = -68.0 (*c* 2.0, $CHCl_3$); IR (film) 1781, 1731 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 4.80 (d, *J* = 5.6 Hz, 1H), 4.60 (ddd, *J* = 5.6, 2.0, 0.8 Hz, 1H), 4.35 (d, *J* = 14.4 Hz, 1H), 4.33 (d, *J* = 16.4 Hz, 1H), 4.28 (d, *J* = 16.4 Hz, 1H), 4.27 (dd, *J* = 12.4, 2.0 Hz, 1H), 4.19 (d, *J* = 12.4 Hz, 1H), 3.75 (d, *J* = 14.4 Hz, 1H), 1.55 (s, 9H) 1.49 (s, 3H), 1.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.1, 166.8, 150.4, 111.1, 97.0, 84.7, 78.2, 75.6, 64.0, 60.1, 46.0, 28.1, 27.4, 26.3; HRMS Calcd for $C_{16}H_{24}NO_8$ (M+H): 358.1502; Found: 358.1498.

To a solution of ketone **3a** (0.138 g, 0.54 mmol) and DMAP (0.0066 g, 0.054 mmol) in THF (20 mL) was added acetic anhydride (1.1 g, 10.8 mmol). Upon stirring at rt for 12 h, the reaction mixture was concentrated and purified by flash chromatography (silica gel, hexanes/EtOAc = 1/1) to give ketone **3c** as a white solid (0.106 g, 66% yield): mp 159-160 ºC; $[\alpha]_D^{25}$ = -109.1 (*c* 0.80, $CHCl_3$); IR (film) 1753, 1732, 1693 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 4.78 (d, *J* = 5.6 Hz, 1H), 4.59 (d, *J* = 5.6 Hz, 1H), 4.39 (d, *J* = 16.4 Hz, 1H), 4.33 (d, *J* = 16.4

Hz, 1H), 4.23 (dd, *J* = 13.2, 2.0 Hz, 1H), 4.22 (d, *J* = 14.8 Hz, 1H), 4.18 (d, *J* = 13.2 Hz, 1H), 3.99 (d, *J* = 14.8 Hz, 1H), 2.60 (s, 3H), 1.48 (s, 3H), 1.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.0, 171.8, 168.2, 111.2, 96.9, 77.9, 75.5, 63.8, 60.2, 44.1, 27.4, 27.3, 26.2; Anal.Calcd. for $C_{13}H_{17}NO_7$: C, 52.17; H, 5.73. Found: C, 52.06; H, 5.89.

Synthesis and characterization of ketones 3d-h



Ketone **3d**: To a solution of amino alcohol **7d** (prepared from D-glucose in two steps)[a] (3.09 g, 10.0 mmol) and Et_3N (1.11 g, 1.54 mL, 11.0 mmol) in dry THF (50 mL), a solution of 2-bromoacetyl bromide (2.22 g, 0.95 mL, 11.0 mmol) in dry THF (10 mL) was added dropwise at rt over 2 h. After the resulting mixture was stirred at rt for 3 h, NaH (95%, 0.6 g, 23.7 mmol) was added into the reaction mixture carefully. Upon stirring at rt for 0.5 h, the reaction mixture was quenched with MeOH (0.25 mL) and filtered. The filtrate was concentrated and purified by flash chromatography (silica gel, hexanes/EtOAc = 1/6) to give lactam **8d** as a white solid (1.42 g, 41% yield): mp 198-199 ºC; $[\alpha]_D^{25}$ = -144.6 (*c* 1.0, $CHCl_3$); IR (film) 3410, 1661 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21-7.14 (m, 4H), 4.40-4.36 (m, 1H), 4.30-4.21 (m, 4H), 4.12 (d, *J* = 13.2 Hz, 1H), 3.96 (dd, *J* = 13.2, 2.8 Hz, 1H), 3.62-3.59 (m, 1H), 3.53-3.48 (m, 1H), 3.10-2.88 (m, 1H), 2.33 (s, 3H), 1.51 (s, 3H), 1.37 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.6, 138.4, 137.4, 130.1, 125.8, 109.7, 96.2, 76.5, 73.4, 71.7, 62.7, 60.5, 54.2, 28.2, 26.2, 21.2;

HRMS Calcd for $C_{18}H_{24}O_6N$ (M+H): 350.1604; Found: 350.1607. ([a] Shu, L.; Wang, P.; Gan, Y.; Shi, Y. *Org. Lett.* **2003**, *5*, 293.)

AcOH (0.15 mL) was added to a slurry of lactam **8d** (4.8 g, 13.76 mmol), PDC (10.3 g, 27.5 mmol), and 3Å MS (6.5 g) in CH_2Cl_2 (300 mL). Upon stirring at rt for 3 d (no SM left as judged by TLC), the reaction mixture was filtered through a pad of silica gel, and the filter cake was washed with EtOAc. The filtrate was concentrated and purified by flash chromatography (silica gel, hexanes/EtOAc = 3/1) to give ketone **3d** as a white solid (4.5 g, 95% yield): mp 184-185 ºC; $[\alpha]_D^{25}$ = -86.5 (*c* 1.0, $CHCl_3$); IR (film) 1753, 1674 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.24-7.18 (m, 4H), 4.86 (d, *J* = 5.7 Hz, 1H), 4.66-4.64 (m, 1H), 4.49-4.23 (m, 5H), 3.64 (d, *J* = 13.8 Hz, 1H), 2.36 (s, 3H), 1.47 (s, 3H), 1.43 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.7, 165.2, 138.2, 137.7, 130.2, 125.8, 111.0, 96.1, 78.4, 75.7, 63.2, 59.9, 51.9, 27.3. 26.2, 21.3; HRMS Calcd for $C_{18}H_{22}NO_6$ (M+H): 348.1447; Found: 348.1447; Anal. Calcd. for $C_{18}H_{21}NO_6$: C, 62.24; H, 6.09. Found: C, 62.02; H, 6.01.

Ketone **3e** prepared by a reaction sequence similar to **3d**: White solid; mp 84-86 ºC; $[\alpha]_D^{25}$ = -87.0 (*c* 1.1, $CHCl_3$); IR (film) 1753, 1675 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.23-7.22 (m, 4H), 4.85 (d, *J* = 5.6 Hz, 1H), 4.65 (d, *J* = 5.6 Hz, 1H), 4.48-4.23 (m, 5H), 3.64 (d, *J* = 13.6 Hz, 1H), 2.61 (t, *J* = 8.0 Hz, 2H), 1.63-1.55 (m, 2H), 1.46 (s, 3H), 1.42 (s, 3H), 1.40-1.31 (m, 2H), 0.93 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.7, 165.2, 142.6, 138.3, 129.6, 125.7, 111.0, 96.2, 78.5, 75.7, 63.3, 59.9, 51.9, 35.4, 33.7, 27.3, 26.3, 22.5, 14.1; Anal. Calcd. for $C_{21}H_{27}NO_6$: C, 64.77; H, 6.99; N, 3.60. Found: C, 64.57; H, 6.86; N, 3.50.

Ketone **3f** prepared by a reaction sequence similar to **3d**: White solid; mp 153-155 ºC; $[\alpha]_D^{25}$ = -95.1 (*c* 0.70, $CHCl_3$); IR (film) 1754, 1675 cm^{-1}; ^1H NMR (300 M Hz, $CDCl_3$) δ 6.94 (s, 1H), 6.93 (s, 2H), 4.85 (d, *J* = 5.4 Hz, 1H), 4.65 (d, *J* = 5.4 Hz, 1H), 4.48-4.23 (m, 5H), 3.62 (d, *J* = 13.5 Hz, 1H), 2.32 (s, 6H), 1.46 (s, 3H), 1.42 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.7, 165.1, 140.6, 139.4, 129.7, 123.8, 111.0, 96.1, 78.5, 75.7, 63.2, 59.9, 52.0, 27.4, 26.3, 21.4; Anal. Calcd. for $C_{19}H_{23}NO_6$: C, 63.15; H, 6.41. Found: C, 63.41; H, 6.60.

Ketone **3g**: To a slurry of 9-aminoflurene hydrochloride (25.0 g, 115.0 mmol) in $CHCl_3$ (500 mL), a solution of NaOH (5.52 g, 138.0 mmol) in water (50 mL) was added. The resulting mixture was stirred at rt overnight. The layers were separated, and the aqueous layer was extracted by $CHCl_3$ (80 mL). The combined organic layers were dried over Na_2SO_4 and concentrated to give a light yellow solid (20.3 g, 98% yield) which was directly used for next step without further purification.

To a mixture of D-Glucose (24.1 g, 134.0 mmol) and 9-aminoflurene (20.3 g, 112.0 mmol) were added acetic acid (3.2 g, 3.1 mL, 53.0 mmol), EtOH (21 mL), and water (13.4 mL). The mixture was rotated on a rotary evaporator open to air at rt for 6 h (a spatula was occasionally used to break the hard clumps). Upon standing at rt overnight, the mixture was diluted with EtOAc (70 mL) and stirred at rt for 1 h. The resulting slurry was filtered and washed by a mixture of hexanes and EtOAc (1/1, v/v, 60 mL). The filter cake was dried under vacuum to give a white solid (28.0 g, 72% yield) which is directly used in next step without further purification.

To a solution of the above white solid (27.0 g, 78.6 mmol) in isopropanol (195 mL), a solution of oxalic acid (10.6 g, 117.9 mmol) in isopropanol (130 mL) was added. Upon stirring at 70 ºC for 5 h, the reaction mixture was cooled to rt, filtered, and washed with ether. The filter cake was dried under vacuum to get a crude light brown solid (26.0 g, 96% yield) which is directly used in next step without further purification. (Hodge, J. E.; Fisher, B. E. *Methods Carbohydr. Chem.* **1963**, *2*, 99.)

Concentrated H_2SO_4 (3.64 mL, 65.6 mmol) was added to a suspension of the above compound (15.0 g, 43.7 mmol) and trimethyl orthoformate (9.26 g, 9.56 mL, 87.4 mmol) in acetone (300 mL) at 0 ºC. Upon stirring at 0 ºC (ice-water bath) for 40 min, the reaction mixture was quenched with NH_4OH (9.0 mL), diluted with acetone (about 500 mL), and dried over excess Na_2SO_4 with stirring at rt for 1 h. The reaction mixture was filtered through a pad of silica gel, and the filtrate was concentrated until small amount of solution was left (10 mL). A solid was crystallized, filtered, and washed by acetone to give diol **7g** as a white solid (7.0 g,

42% yield): mp 139-140 ºC (decompose); $[\alpha]_D^{25}$ = -136.0 (*c* 1.0, $CHCl_3$); IR (film) 3068, 1601 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.97 (t, *J* = 7.2 Hz, 2H), 7.70 (t, *J* = 7.2 Hz, 2H), 7.45 (t, *J* = 7.2 Hz, 1H), 7.42 (t, *J* = 7.2 Hz, 1H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.25 (t, *J* = 7.2 Hz, 1H), 5.83 (s, 1H), 4.45 (t, *J* = 6.0 Hz, 1H), 4.15-4.12 (m, 2H), 3.72 (d, *J* = 13.2 Hz, 1H), 3.56 (d, *J* = 6.4 Hz, 1H), 2.69 (d, *J* = 12.8 Hz, 1H), 2.45 (d, *J* = 12.8 Hz, 1H), 2.18 (s, 1H), 1.29 (s, 3H), 1.23 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.9, 142.0, 141.8, 138.2, 137.9, 130.1, 128.5, 128.4, 126.4, 126.1, 120.6, 109.1, 93.9, 75.8, 73.2, 60.6, 60.0, 48.6, 27.7, 25.8; HRMS. Calcd. for $C_{22}H_{26}NO_5$ (M+H): 384.1805; Found: 384.1810.

To a solution of diol **7g** (1.5 g, 3.92 mmol) and Et_3N (0.435 g, 0.60 mL, 4.31 mmol) in dry THF (125 mL), 2-bromoacetyl bromide (0.87 g, 0.375 mL, 4.31 mmol) was added dropwise at rt within 5 min. After the resulting mixture was stirred at rt for 2 h and 10 min (TLC showed diol **7g** gone), NaH (60%, 0.627 g, 15.7 mmol) was added slowly. Upon stirring at rt for 2 d, the slurry mixture was quenched with saturated aqueous $NaHCO_3$ (5 mL), diluted with Et_2O (100 mL), washed with H_2O (3×15 mL). The organic layers was dried over Na_2SO_4, concentrated, dissolved in DCM, washed with H_2O, and dried over Na_2SO_4, filtered, concentrated, and recrystallized in DCM-Et_2O-Hexanes to give alcohol **8g** as a white solid (0.905 g, 55% yield): mp 197-198 ºC; $[\alpha]_D^{25}$ = -163.5 (*c* 0.5, $CHCl_3$); IR (film) 3364, 1646 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.74-7.69 (m, 2H), 7.52-7.29 (m, 6H), 6.91 (s, 1H), 4.50 (d, *J* = 16.5 Hz, 1H), 4.38 (d, *J* = 16.5 Hz, 1H), 4.22 (dd, *J* = 6.0, 2.1 Hz, 1H), 4.15 (t, *J* = 6.3 Hz, 1H), 4.00 (d, *J* = 13.5 Hz, 1H), 3.90 (dd, *J* = 13.5, 2.4 Hz, 1H), 3.34 (t, *J* = 6.3 Hz, 1H), 3.32 (d, *J* = 12.6 Hz, 1H), 2.43 (d, *J* = 12.6 Hz, 1H), 2.20 (d, *J* = 6.3 Hz, 1H), 1.29 (s, 3H), 1.23 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.4, 141.6, 141.1, 140.9, 129.1, 129.0, 128.3, 127.9, 125.7, 125.1, 120.5, 120.1, 109.7, 95.6, 75.9, 73.2, 71.0, 62.5, 60.7, 58.1, 45.8, 27.6, 26.0; HRMS. Calcd. for $C_{24}H_{26}NO_6$ (M+H): 424.1755; Found: 424.1766.

To a slurry of alcohol **8g** (0.905 g, 2.14 mmol), PDC (1.609 g, 4.28 mmol), and 3Å MS (1.127 g) in DCM (80 mL), AcOH (0.05 mL) was added. Upon stirring at rt for 2 d (TLC showed no alcohol left), the reaction mixture was filtered through a pad of silica gel, and the

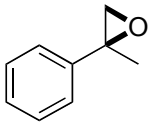
filter cake was washed by EtOAc. The filtrate was concentrated and purified by flash chromatography (silica gel, hexanes/EtOAc = 2/1 to 1/1) to give ketone **3g** as a white solid (0.67 g, 74% yield): mp 225-226 ºC; $[\alpha]_D^{25}$ = -114.4 (*c* 0.80, $CHCl_3$); IR (film) 1755, 1661 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.73 (t, *J* = 6.8 Hz, 2H), 7.48-7.41 (m, 4H), 7.33 (t, *J* = 7.3 Hz, 2H), 6.91 (s, 1H), 4.72 (d, *J* = 5.6 Hz, 1H), 4.58 (d, *J* = 5.6 Hz, 1H), 4.54 (d, *J* = 16.4 Hz, 1H), 4.45 (d, *J* = 16.4 Hz, 1H), 4.22-4.10 (m, 2H), 3.40 (d, *J* = 13.2 Hz, 1H), 4.51 (d, *J* = 13.2 Hz, 1H), 1.33 (s, 3H), 1.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.6, 165.8, 141.7, 141.6, 140.8, 140.6, 129.3, 129.2, 128.2, 128.0, 125.5, 125.1, 120.6, 120.3, 111.0, 95.9, 78.4, 75.6, 62.6, 60.1, 58.1, 43.5, 27.1, 26.1; Anal. Calcd. for $C_{24}H_{23}NO_6$: C, 68.40; H, 5.50. Found: C, 68.55; H, 5.71.

Ketone **3h** prepared by a reaction sequence similar to **3g**: White solid; mp 74-75 ºC; $[\alpha]_D^{25}$ = -72.9 (*c* 0.70, $CHCl_3$); IR (film) 1755, 1663 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 4.82 (d, *J* = 5.4 Hz, 1H), 4.62 (dd, *J* = 5.4, 1.5 Hz, 1H), 4.32-4.16 (m, 4H), 3.98 (d, *J* = 13.5 Hz, 1H), 3.41 (t, *J* = 7.5 Hz, 2H), 3.25 (d, *J* = 13.5 Hz, 1H), 1.62-1.26 (m, 8H), 1.49 (s, 3H), 1.42 (s, 3H), 0.89 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.9, 164.7, 110.9, 95.9, 78.5, 75.7, 62.6, 59.7, 48.1, 46.6, 31.7, 27.4, 26.8, 26.5, 26.3, 22.7, 14.2; Anal. Calcd. for $C_{17}H_{27}NO_6$: C, 59.81; H, 7.97. Found: C, 59.87; H, 8.12.

Representative Epoxidation Procedure (Table 2, Entry 19). To a solution of the olefin (0.032 g, 0.20 mmol), tetrabutylammonium hydrogen sulfate (0.0038 g, 0.010 mmol), and ketone (0.0208 g, 0.06 mmol) in dioxane (3 mL) was added buffer (0.1 M K_2CO_3-AcOH in 4 x 10^{-4} M aqueous EDTA, pH = 9.3)(2 mL) with stirring. After the mixture was cooled to -10 ºC (bath temperature), a solution of Oxone (0.20 M in 4 x 10^{-4} M aqueous EDTA,1.6 mL) (0.197 g, 0.32 mmol) and a solution of K_2CO_3 (0.84 M in 4 x 10^{-4} M aqueous EDTA, 1.6 mL) (0.185 g, 1.344 mmol) were added separately and simultaneously via a syringe pump over a period of 2 h. The reaction mixture was quenched with hexanes, extracted with EtOAc, dried over Na_2SO_4, filtered, concentrated, and purified by flash chromatography (silica gel was buffered with 1% Et_3N in

organic solvent; hexanes/Et_2O=5/1 as eluent) to give the epoxide as white solid (0.027 g, 76% yield, 87% ee).

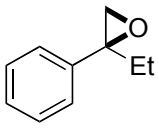
Table 2, Entry 1



Colorless oil; $[\alpha]_D^{25}$ = +13.0 (*c* 0.80, $CHCl_3$) (62% ee); ^1H NMR (300 MHz, $CDCl_3$) δ 7.41-7.27 (m, 5H), 2.99 (d, *J* = 5.4 Hz, 1H), 2.82 (d, *J* = 5.4 Hz, 1H), 1.74 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.4, 128.5, 127.7, 125.5, 57.3, 57.0, 22.0.

Capriati, V.; Florio, S.; Luisi, R.; Salomone, A. *Org. Lett.* **2002**, *4*, 2445.

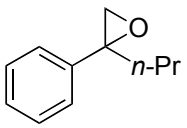
Table 2, Entry 2



Colorless oil; $[\alpha]_D^{25}$ = +26.1 (*c* 0.70, $CHCl_3$) (78% ee); IR (film) 1496, 1463, 1448 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.41-7.29 (m, 5H), 3.00 (d, *J* = 5.7 Hz, 1H), 2.76 (d, *J* = 5.7 Hz, 1H), 2.28-2.16 (m, 1H), 1.89-1.77 (m, 1H), 0.96 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.2, 128.5, 127.5, 126.2, 61.1, 55.6, 28.5, 9.2; HRMS Calcd for $C_{10}H_{12}O$ (M): 148.0888; found: 148.0889.

Capriati, V.; Florio, S.; Luisi, R.; Salomone, A. *Org. Lett.* **2002**, *4*, 2445.

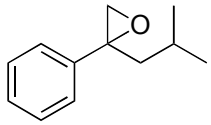
Table 2, Entry 3



Colorless oil; $[\alpha]_D^{25}$ = +26.1 (*c* 1.4, $CHCl_3$) (75% ee); IR (film) 1496, 1465, 1448 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.40-7.25 (m, 5H), 2.96 (d, *J* = 5.4 Hz, 1H), 2.74 (d, *J* = 5.4 Hz, 1H),

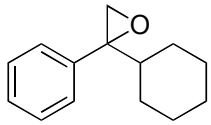
2.21-2.11 (m, 1H), 1.77-1.67 (m, 1H), 1.47-1.32 (m, 2H), 0.93 (t, J = 7.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.4, 128.5, 127.5, 126.2, 60.6, 55.6, 37.8, 18.5, 14.4; HRMS Calcd for $C_{11}H_{14}O$ (M): 162.1045; found: 162.1046; Anal. Calcd. for $C_{11}H_{14}O$: C, 81.44; H, 8.70. Found: C, 81.22; H, 8.80.

Table 2, Entry 4



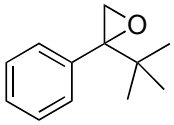
Colorless oil; $[\alpha]_D^{25}$ = +31.1 (*c* 0.90, $CHCl_3$) (74% ee); IR (film) 1496, 1466, 1448 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.25 (m, 5H), 2.87 (d, J = 5.7 Hz, 1H), 2.72 (d, J = 5.7 Hz, 1H), 2.14 (dd, J = 13.8, 6.0 Hz, 1H), 1.66 (septet, J = 6.6 Hz, 1H), 1.55 (dd, J = 13.8, 8.1 Hz, 1H), 0.93 (d, J = 6.6 Hz, 3H), 0.90 (d, J = 6.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.4, 128.5, 127.5, 126.3, 60.2, 55.1, 44.8, 25.6, 23.6, 22.9. HRMS Calcd for $C_{12}H_{16}O$ (M): 176.1201; found: 176.1206; Anal. Calcd. for $C_{12}H_{16}O$: C, 81.77; H, 9.15. Found: C, 81.69; H, 9.30.

Table 2, Entry 5



Colorless oil; $[\alpha]_D^{25}$ = +35.2 (*c* 1.0, $CHCl_3$) (77% ee); IR (film) 1495, 1447 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37-7.26 (m, 5 H), 3.02 (d, J = 5.7 Hz, 1 H), 2.70 (d, J = 5.7 Hz, 1H), 1.82-1.56 (m, 6H), 1.26-0.95 (m, 5H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.9, 128.1, 127.51, 127.46, 64.5, 53.1, 43.2, 29.0, 28.3, 26.5, 26.3, 26.2; Anal. Calcd. for $C_{14}H_{18}O$: C, 83.12; H, 8.97. Found: C, 83.33; H, 8.74.

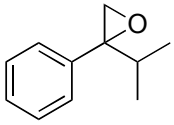
Table 2, Entry 6



Colorless oil; $[\alpha]_D^{25}$ = +53.3 (*c* 0.90, $CHCl_3$) (86% ee); IR (film) 1480, 1462, 1447 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.38-7.25 (m, 5H), 3.12 (d, *J* = 5.2 Hz, 1H), 2.66 (d, *J* = 5.2 Hz, 1H), 0.99 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.7, 129.0, 127.5, 127.4, 67.0, 51.0, 34.0, 26.5; Anal. Calcd. for $C_{12}H_{16}O$: C, 81.77; H, 9.15. Found: C, 81.53; H, 9.10.

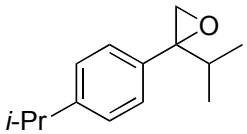
Lodge, E. P.; Heathcock, C. H. *J. Am. Chem. Soc*. **1987**, *109*, 3353.

Table 2, Entry 7



Colorless oil; $[\alpha]_D^{25}$ = +33.5 (*c* 1.1, $CHCl_3$) (84% ee); IR (film) 1496, 1468 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.39-7.26 (m, 5H), 3.00 (d, *J* = 5.4 Hz, 1H), 2.73 (d, *J* = 5.4 Hz, 1H), 2.10 (septet, *J* = 6.9 Hz, 1H), 0.98 (d, *J* = 3.6 Hz, 3H), 0.95 (d, *J* = 3.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.6, 128.1, 127.6, 127.5, 64.7, 53.4, 33.3, 18.7, 18.0; Anal. Calcd. for $C_{11}H_{14}O$: C, 81.44; H, 8.70. Found: C, 81.62; H, 8.62.

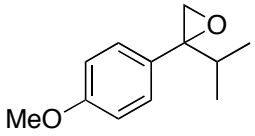
Table 2, Entry 8



Colorless oil; $[\alpha]_D^{25}$ = +23.6 (*c* 1.0, $CHCl_3$) (82% ee); IR (film) 1512, 1464 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 2.98 (d, *J* = 5.1 Hz, 1H), 2.90 (septet., *J* = 6.9 Hz, 1H), 2.72 (d, *J* = 5.1 Hz, 1H), 2.08 (septet., *J* = 6.9 Hz, 1H), 1.25 (d, *J* = 6.9 Hz, 6H), 0.96 (d, *J* = 6.9 Hz, 3H), 0.95 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.2, 136.9, 127.4, 126.1, 64.6, 53.3, 34.0, 33.4, 24.2, 18.7, 18.1; HRMS Calcd for $C_{14}H_{21}O$ (M+H): 205.1592; found: 205.1588. Anal. Calcd. for $C_{14}H_{20}O$: C, 82.30; H, 9.87.

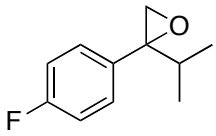
Found: C, 82.42; H, 9.69.

Table 2, Entry 9



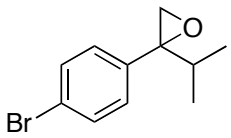
Colorless oil; $[\alpha]_D^{25}$= +22.2 (*c* 1.1, $CHCl_3$) (84% ee); IR (film) 1612 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.30-7.27 (m, 2H), 6.88-6.86 (m, 2H), 3.81 (s, 3H), 2.97 (d, *J* = 5.2 Hz, 1H), 2.71 (d, *J* = 5.2 Hz, 1H), 2.03 (septet, *J* = 6.8 Hz, 1H), 0.95 (d, *J* = 6.8 Hz, 3H), 0.94 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.0, 131.6, 128.7, 113.5, 64.4, 55.5, 53.5, 33.6, 18.8, 18.1; Anal. Calcd. for $C_{12}H_{16}O_2$: C, 74.97; H, 8.39. Found: C, 74.78; H, 8.22.

Table 2, Entry 10



Colorless oil; $[\alpha]_D^{25}$= +28.2 (*c* 1.1 $CHCl_3$) (74% ee); IR (film) 1606 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.35-7.32 (m, 2H), 7.04-7.00 (m, 2H), 2.99 (d, *J* = 5.2 Hz, 1H), 2.69 (d, *J* = 5.2 Hz, 1H), 2.04 (septet, *J* = 6.8 Hz, 1H), 0.95 (d, *J* = 6.8 Hz, 3H), 0.94 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.5, 161.0, 135.3, 129.2, 129.1, 115.1, 114.9, 64.3, 53.5, 33.4, 18.7, 18.0; Anal. Calcd. for $C_{11}H_{13}FO$: C, 73.31; H, 7.27. Found: C, 73.53; H, 7.42.

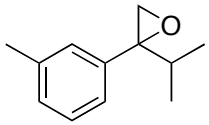
Table 2, Entry 11



Colorless oil; $[\alpha]_D^{25}$= +21.7 (*c* 1.2, $CHCl_3$) (78% ee); IR (film) 1593 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.48-7.45 (m, 2H), 7.26-7.23 (m, 2H), 3.00 (d, *J* = 5.2 Hz, 1H), 2.66 (d, *J* = 5.2 Hz, 1H), 2.07 (septet, *J* = 6.8 Hz, 1H), 0.95 (d, *J* = 6.8 Hz, 3H), 0.93 (d, *J* = 6.8 Hz, 3H); ^{13}C

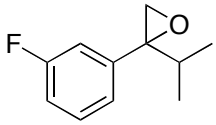
NMR (100 MHz, $CDCl_3$ δ 138.7, 131.3, 129.2, 121.6, 64.2, 53.5, 33.1, 18.7, 17.9; Anal. Calcd. for $C_{11}H_{13}BrO$: C, 54.79; H, 5.43. Found: C, 54.72; H, 5.34.

Table 2, Entry 12



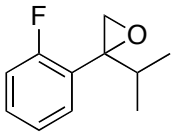
Colorless oil; $[\alpha]_D^{25}$ = +30.9 (*c* 1.0, $CHCl_3$) (82% ee); IR (film) 1608 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.24-7.09 (m, 4H), 2.99 (d, *J* = 5.2 Hz, 1H), 2.71 (d, *J* = 5.2 Hz, 1H), 2.36 (s, 3H), 2.09 (septet, *J* = 6.8 Hz, 1H), 0.96 (d, *J* = 7.2 Hz, 3H), 0.95 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.5, 137.7, 128.3, 128.1, 128.0, 124.6, 64.7, 53.4, 33.3, 21.7, 18.7, 18.1; Anal. Calcd. for $C_{12}H_{16}O$: C, 81.77; H, 9.15. Found: C, 81.96; H, 8.97.

Table 2, Entry 13



Colorless oil; $[\alpha]_D^{25}$ = +35.7 (*c* 1.4, $CHCl_3$) (81% ee); IR (film) 1616 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.33-7.27 (m, 1H), 7.15 (dd, *J* = 7.6, 0.8 Hz, 1H), 7.10-7.07 (m, 1H), 7.00-6.95 (ddd, *J* = 8.4, 2.8, 0.8 Hz, 1H), 3.01 (d, *J* = 5.2 Hz, 1H), 2.69 (d, *J* = 5.2 Hz, 1H), 2.12 (septet, *J* = 6.8 Hz, 1H), 0.97 (d, *J* = 7.2 Hz, 3H), 0.95 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.0, 161.6, 142.43, 142.36, 129.8, 129.7, 122.9, 114.6, 114.5, 114.4, 114.3, 64.1, 53.6, , 32.9, 18.7, 17.8; Anal. Calcd. for $C_{11}H_{13}FO$: C, 73.31; H, 7.27. Found: C, 73.50; H, 7.39.

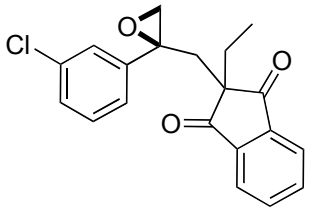
Table 2, Entry 14



Colorless oil; $[\alpha]_D^{25}$ = +53.1 (*c* 1.5, $CHCl_3$) (88% ee); IR (film) 1617 cm^{-1}; ^1H NMR (400

MHz, $CDCl_3$) δ 7.41-7.37 (td, *J* = 7.2, 1.2 Hz, 1H), 7.32-7.26 (m, 1H), 7.15-7.11 (td, *J* = 7.6, 1.2 Hz, 1H), 7.06-7.01 (m, 1H), 3.05 (d, *J* = 5.2 Hz, 1H), 2.81 (d, *J* = 5.2 Hz, 1H), 2.02 (septet, *J* = 6.8 Hz, 1H), 0.98-0.94 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.8, 159.4, 130.5, 130.4, 129.6, 129.5, 126.8, 126.7, 123.9, 123.9, 115.5, 115.3, 61.4, 52.5, 33.9, 18.2, 17.9; Anal. Calcd. for $C_{11}H_{13}FO$: C, 73.31; H, 7.27. Found: C, 73.12; H, 6.93.

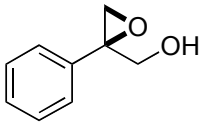
Table 2, Entry 15



Colorless oil; $[\alpha]_D^{25}$ = -34.9 (*c* 0.80, $CHCl_3$) (66% ee); IR (film) 1744, 1708 cm^{-1}; ^1H NMR (400 MHz) δ 7.93-7.73 (m, 4H), 7.11-6.99 (m, 3H), 6.82 (s, 1H), 2.84 (d, *J* = 5.2 Hz, 1H), 2.77 (d, *J* = 14.0 Hz, 1H), 2.56 (d, *J* = 14.0 Hz, 1H), 2.48 (d, *J* = 5.2 Hz, 1H), 1.80 (q, *J* = 7.6 Hz, 2H), 0.65 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz) δ 203.4, 203.1, 142.4, 141.4, 135.7, 135.6, 134.2, 129.6, 128.1, 126.3, 124.9, 123.0, 122.9, 58.0, 57.2, 56.3, 40.1, 30.1, 8.9; Anal. Calcd. for $C_{20}H_{17}O_3Cl$: C, 70.49; H, 5.03. Found: C, 70.26; H, 5.21.

Tanaka, K.; Yoshida, K.; Sasaki, C.; Osano, Y. T. *J. Org. Chem.* **2002**, *67*, 3131.

Table 2, Entry 16

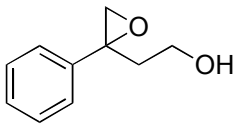


Colorless oil; $[\alpha]_D^{25}$ = +27.4 (*c* 1.3, $CHCl_3$) (77% ee); IR (film) 3420 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.41-7.33 (m, 5H), 4.12 (dd, *J* = 12.3, 4.5 Hz, 1H), 4.03 (dd, *J* = 12.6, 9.0 Hz, 1H), 3.29 (d, *J* = 5.1 Hz, 1H), 2.84 (d, *J* = 5.1 Hz, 1H), 1.91 (dd, *J* = 9.0, 6.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.5, 128.8, 128.4, 126.2, 63.2, 60.6, 52.7; HRMS Calcd for $C_9H_9O_2$ (M-H): 149.0603. Found: 149.0601

Adam, W.; Alsters, P. L.; Neumann, R.; Saha-Möller, C. R.; Seebach, D.; Zhang, R. *Org. Lett.*

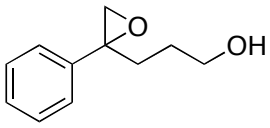
2003, *5*, 725.

Table 2, Entry 17



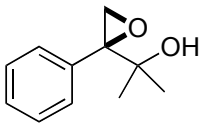
Colorless oil; $[\alpha]_D^{25}$ = +17.5 (*c* 1.3, $CHCl_3$) (72% ee); IR (film) 3411 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.41-7.28 (m, 5 H), 3.80-3.68 (m, 2H), 3.14 (d, *J* = 4.8 Hz, 1H), 2.79 (d, *J* = 4.8 Hz, 1 H), 2.52 (ddd, *J* = 14.4, 6.8, 5.6 Hz, 1H), 2.12 (ddd, *J* = 14.4, 6.8, 5.6 Hz, 1H), 2.04 (t, *J* = 5.6 Hz, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.9, 128.7, 128.0, 125.9, 59.7, 59.4, 55.0, 37.2; HRMS Calcd for $C_{10}H_{12}O_2$ (M): 164.0837. Found: 164.0836.

Table 2, Entry 18



Colorless oil; $[\alpha]_D^{25}$ = +19.8 (*c* 2.1, $CHCl_3$) (74% ee); IR (film) 3396 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.39-7.20 (m, 5H), 3.62 (t, *J* = 6.0 Hz, 2H), 2.99 (d, *J* = 5.1 Hz, 1H), 2.75 (d, *J* = 5.1 Hz, 1H), 2.45-2.36 (m, 1H), 2.09 (s, 1H), 1.82-1.72 (m, 1H), 1.67-1.58 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.6, 128.6, 127.6, 126.0, 62.4, 60.3, 56.3, 31.8, 28.0; HRMS Calcd for $C_{11}H_{14}O_2$ (M): 178.0994. Found: 178.0991; Anal. Calcd. for $C_{11}H_{14}O_2$: C, 74.13; H, 7.92. Found: C, 74.33 ; H, 7.90.

Table 2, Entry 19

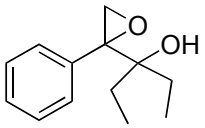


White solid; mp 55-56 °C; $[\alpha]_D^{25}$ = +55.3 (*c* 1.1, $CHCl_3$) (87% ee); IR (film) 3477 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.44-7.31 (m, 5 H), 3.37 (d, *J* = 5.2 Hz, 1H), 2.75 (d, *J* = 5.2 Hz, 1 H), 2.14 (s, 1 H), 1.36 (s, 3 H), 1.22 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.1, 128.5,

128.1, 128.0, 70.4, 67.0, 51.1, 26.9, 25.7; HRMS Calcd for $C_{11}H_{12}O$ (M-H_2O): 160.0888; found: 160.0890.

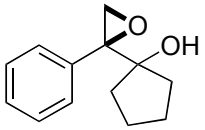
Capriati, V.; Florio, S.; Luisi, R.; Salomone, A. *Org. Lett.* **2002**, *4*, 2445.

Table 2, Entry 20



Colorless oil; $[\alpha]_D^{25}$ = +38.1 (*c* 1.4, $CHCl_3$) (87% ee); IR (film) 3505 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.45-7.40 (m, 2H), 7.40-7.30 (m, 3H), 3.33 (d, *J* = 5.6 Hz, 1H), 2.68 (d, *J* = 5.6 Hz, 1H), 2.06 (s, 1H), 1.83-1.73 (m, 1H), 1.70-1.61 (m, 1H), 1.57-1.48 (m, 1H), 1.46-1.37 (m, 1H), 1.10 (t, *J* = 7.6 Hz, 3H), 0.94 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.4, 128.2, 128.0, 127.8, 73.7, 64.8, 50.6, 30.9, 28.9, 8.4, 7.6; Anal. Calcd. for $C_{13}H_{18}O_2$: C, 75.69; H, 8.80. Found: C, 75.55; H, 8.60.

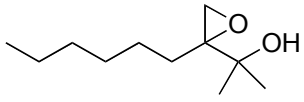
Table 2, Entry 21



Colorless oil; $[\alpha]_D^{25}$ = +48.6 (*c* 1.0, $CHCl_3$) (88% ee); IR (film) 3465 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.48-7.45 (m, 2H), 7.37-7.29 (m, 3H), 3.30 (d, *J* = 5.6 Hz, 1H), 2.78 (d, *J* = 5.6 Hz, 1H), 1.92-1.70 (m, 4H), 1.65-1.52 (m, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.2, 128.7, 128.1, 82.5, 64.5, 51.4, 36.30, 36.27, 23.6, 23.5; Anal. Calcd. for $C_{13}H_{16}O_2$: C, 76.44; H, 7.90. Found: C, 76.33; H, 7.76.

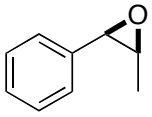
Capriati, V.; Florio, S.; Luisi, R.; Salomone, A. *Org. Lett.* **2002**, *4*, 2445.

Table 2, Entry 22



Colorless oil; $[\alpha]_D^{25}$ = +4.3 (*c* 0.80, $CHCl_3$) (60% ee); IR (film) 3473 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 2.99 (d, *J* = 4.8 Hz, 1H), 2.69 (d, *J* = 4.8 Hz, 1H), 2.07 (s, 1H), 1.88-1.60 (m, 2H), 1.31 (s, 3H), 1.27 (s, 3H), 1.34-1.23 (m, 8H), 0.90 (t, *J* = 6.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 70.3, 64.6, 48.4, 31.9, 29.8, 29.4, 26.5, 25.6, 24.6, 22.8, 14.3; Anal. Calcd. for $C_{11}H_{22}O_2$: C, 70.92; H, 11.90. Found: C, 71.09; H, 11.94.

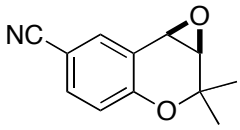
Table 3, Entry 1



Colorless oil; $[\alpha]_D^{25}$ = -40.8 (*c* 0.75, $CHCl_3$) (85% ee); ^1H NMR (300 MHz, $CDCl_3$) δ 7.38-7.26 (m, 5H), 3.07 (d, *J* = 4.2 Hz, 1H), 3.34 (qd, *J* = 5.4, 4.5 Hz, 1H), 1.09 (d, *J* = 5.4 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 135.7, 128.2, 127.7, 126.8, 57.8, 55.4, 12.7.

(1) Tian, H.; She, X.; Shu, L.; Yu, H.; Shi, Y. *J. Am. Chem. Soc.* **2000**, *122*, 11551. (2) Tian, H.; She, X.; Yu, H.; Shu, L.; Shi, Y. *J. Org. Chem.* **2002**, *67*, 2435.

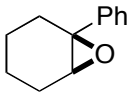
Table 3, Entry 2



Colorless oil; $[\alpha]_D^{25}$ = +61.3 (*c* 1.6, $CHCl_3$) (84% ee); ^1H NMR (300 MHz, $CDCl_3$) δ 7.66 (d, *J* = 2.1 Hz, 1H), 7.53 (dd, *J* = 8.4, 2.1 Hz, 1H), 6.87 (d, *J* = 8.4 Hz, 1H), 3.91 (d, *J* = 4.2 Hz, 1H), 3.54 (d, *J* = 4.5 Hz, 1H), 1.60 (s, 3H). 1.30 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 156.7, 134.6, 134.0, 121.3, 119.2, 118.9, 104.5, 74.9, 62.5, 50.1, 25.7, 23.2.

Tian, H.; She, X.; Yu, H.; Shu, L.; Shi, Y. *J. Org. Chem.* **2002**, *67*, 2435.

Table 3, Entry 3

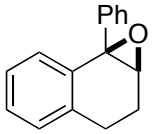


Colorless oil; $[\alpha]_D^{25}$ = -56.3 (*c* 1.4, $CHCl_3$) (80% ee); ^1H NMR (300 MHz, $CDCl_3$)

δ 7.40-7.22 (m, 5H), 3.07 (s, 1H), 2.33-2.24 (m, 1H), 2.12 (td, *J* = 14.7, 5.4 Hz, 1H), 2.02-1.96 (m, 2H), 1.64-1.41 (m, 3H), 1.38-1.26 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.7, 128.5, 127.4, 125.5, 62.1, 60.4, 29.0, 24.9, 20.3, 20.0.

Tian, H.; She, X.; Yu, H.; Shu, L.; Shi, Y. *J. Org. Chem.* **2002**, *67*, 2435.

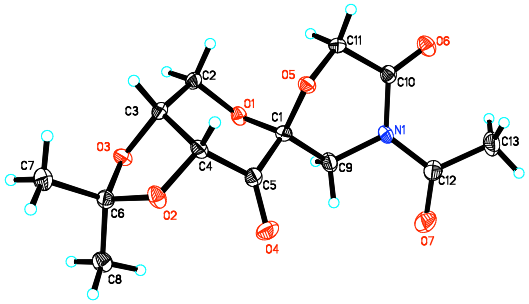
Table 3, Entry 4



Colorless oil; $[\alpha]_D^{25}$ = +54.0 (*c* 1.2, $CHCl_3$) (89% ee); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.53-7.49 (m, 2H), 7.46-7.38 (m, 3H), 7.24 (dd, *J* = 7.2, 1.2 Hz, 1H), 7.18 (d, *J* = 7.2 Hz, 1H), 7.09 (td, *J* = 7.8, 1.2 Hz, 1H), 7.01 (dd, *J* = 7.8, 1.2 Hz, 1H), 3.65 (d, *J* = 3.0 Hz, 1H), 3.03-2.92 (m, 1H), 2.72 (dd, *J* = 15.9, 5.7 Hz, 1H), 2.55-2.46 (m, 1H), 2.06 (td, *J* = 13.8, 6.0 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.8, 137.3, 135.0, 130.0, 128.8, 128.4, 128.3, 128.1, 127.9, 126.1, 63.2, 60.7, 25.6, 22.3.

Wang, Z. X.; Tu, Y.; Frohn, M.; Zhang, J. R.; Shi, Y. *J. Am. Chem. Soc.* **1997**, *119*, 11224.

The X-ray structure of ketone 3c



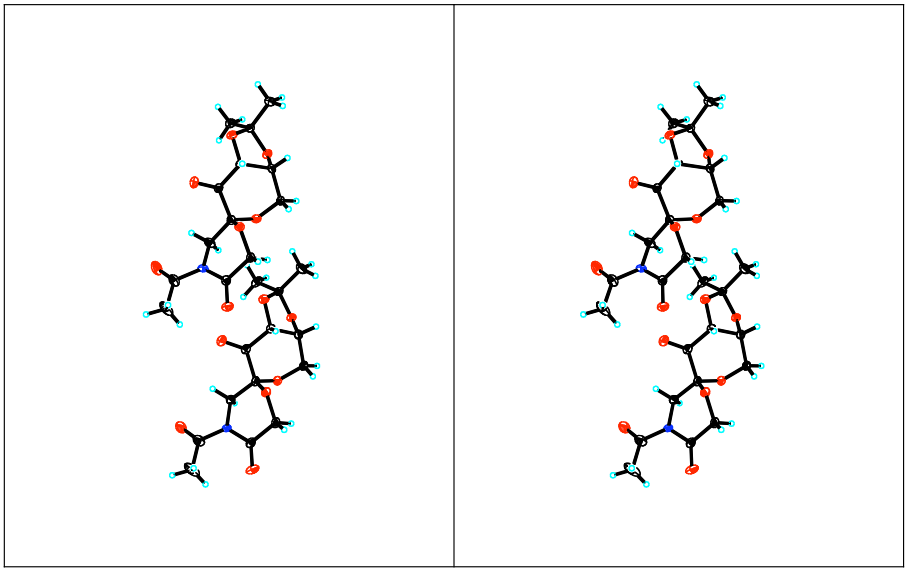


Table 1. Crystal data and structure refinement for **3c**.

Identification code	ys143	
Empirical formula	C13 H17 N O7	
Formula weight	299.28	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 7.6742(2) Å	α= 90°.
	b = 13.4254(3) Å	β= 94.302(2)°.
	c = 13.2089(3) Å	γ = 90°.
Volume	1357.07(6) $Å^3$	
Z	4	
Density (calculated)	1.465 Mg/m^3	
Absorption coefficient	0.120 mm^{-1}	
F(000)	632	
Crystal size	0.09 x 0.09 x 0.04 mm^3	
Theta range for data collection	2.17 to 30.50°.	
Index ranges	-10<=h<=10, -19<=k<=17, -8<=l<=18	
Reflections collected	18744	
Independent reflections	7871 [R(int) = 0.0653]	
Completeness to theta = 30.50°	99.9 %	
Absorption correction	multi-scans	
Max. and min. transmission	0.9955 and 0.9894	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7871 / 1 / 379	
Goodness-of-fit on F^2	0.855	
Final R indices [I>2sigma(I)]	R1 = 0.0545, wR2 = 0.1298	
R indices (all data)	R1 = 0.0929, wR2 = 0.1590	
Largest diff. peak and hole	0.315 and -0.256 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ys143. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	-3182(3)	861(2)	3436(2)	16(1)
O(1)	944(2)	-28(2)	4181(1)	16(1)
O(2)	2894(3)	1545(2)	6581(2)	18(1)
O(3)	4190(2)	458(1)	5521(1)	16(1)
O(4)	580(3)	2236(2)	5068(2)	27(1)
O(5)	-1554(2)	83(2)	5105(1)	16(1)
O(6)	-5066(3)	-492(2)	3306(2)	21(1)
O(7)	-3609(3)	2413(2)	2812(2)	32(1)
C(1)	-391(3)	601(2)	4523(2)	15(1)
C(2)	1802(4)	-630(2)	4972(2)	17(1)
C(3)	2645(3)	-20(2)	5834(2)	16(1)
C(4)	1598(3)	887(2)	6152(2)	16(1)
C(5)	569(4)	1356(2)	5237(2)	16(1)
C(6)	4569(4)	1249(2)	6231(2)	16(1)
C(7)	5729(4)	888(2)	7135(2)	22(1)
C(8)	5352(4)	2105(2)	5676(2)	20(1)
C(9)	-1308(4)	1084(2)	3574(2)	21(1)
C(10)	-3751(3)	-94(2)	3679(2)	16(1)
C(11)	-2590(4)	-610(2)	4491(2)	17(1)
C(12)	-4248(4)	1611(2)	2989(2)	22(1)
C(13)	-6167(4)	1395(3)	2760(3)	36(1)
N(1A)	-8060(3)	-1567(2)	-1054(2)	15(1)
O(1A)	-3994(2)	-2448(1)	-460(1)	16(1)
O(2A)	-1819(3)	-1168(2)	2088(1)	19(1)
O(3A)	-648(2)	-2068(2)	832(1)	16(1)
O(4A)	-4290(3)	-310(2)	764(2)	24(1)
O(5A)	-6388(2)	-2461(1)	545(2)	18(1)
O(6A)	-9803(3)	-2951(2)	-1346(2)	24(1)
O(7A)	-8565(3)	-30(2)	-1687(2)	28(1)
C(1A)	-5250(4)	-1868(2)	7(2)	15(1)
C(2A)	-3051(4)	-3127(2)	232(2)	17(1)

C(3A)	-2127(3)	-2617(2)	1135(2)	16(1)
C(4A)	-3134(4)	-1779(2)	1614(2)	16(1)
C(5A)	-4256(4)	-1203(2)	808(2)	16(1)
C(6A)	-190(4)	-1392(2)	1643(2)	16(1)
C(7A)	528(4)	-455(2)	1198(2)	24(1)
C(8A)	1054(4)	-1867(3)	2446(2)	25(1)
C(9A)	-6237(4)	-1258(2)	-832(2)	20(1)
C(10A)	-8556(4)	-2554(2)	-905(2)	17(1)
C(11A)	-7403(4)	-3113(2)	-127(2)	21(1)
C(12A)	-9162(4)	-842(2)	-1515(2)	24(1)
C(13A)	-11048(5)	-1069(3)	-1744(4)	51(1)

Table 3. Bond lengths [Å] and angles [°] for ys143.

N(1)-C(10)	1.399(4)
N(1)-C(12)	1.401(4)
N(1)-C(9)	1.467(3)
O(1)-C(1)	1.427(3)
O(1)-C(2)	1.441(3)
O(2)-C(4)	1.416(3)
O(2)-C(6)	1.453(3)
O(3)-C(6)	1.432(3)
O(3)-C(3)	1.436(3)
O(4)-C(5)	1.203(4)
O(5)-C(1)	1.405(3)
O(5)-C(11)	1.435(3)
O(6)-C(10)	1.213(3)
O(7)-C(12)	1.213(4)
C(1)-C(5)	1.534(4)
C(1)-C(9)	1.534(4)
C(2)-C(3)	1.508(4)
C(3)-C(4)	1.535(4)
C(4)-C(5)	1.528(4)
C(6)-C(8)	1.511(4)

C(6)-C(7)	1.515(4)
C(10)-C(11)	1.510(4)
C(12)-C(13)	1.509(4)
N(1A)-C(10A)	1.397(4)
N(1A)-C(12A)	1.399(4)
N(1A)-C(9A)	1.468(3)
O(1A)-C(1A)	1.416(3)
O(1A)-C(2A)	1.446(3)
O(2A)-C(4A)	1.410(3)
O(2A)-C(6A)	1.452(3)
O(3A)-C(6A)	1.428(3)
O(3A)-C(3A)	1.435(3)
O(4A)-C(5A)	1.200(3)
O(5A)-C(1A)	1.412(3)
O(5A)-C(11A)	1.435(3)
O(6A)-C(10A)	1.207(3)
O(7A)-C(12A)	1.211(4)
C(1A)-C(9A)	1.531(4)
C(1A)-C(5A)	1.543(4)
C(2A)-C(3A)	1.506(4)
C(3A)-C(4A)	1.529(4)
C(4A)-C(5A)	1.528(4)
C(6A)-C(7A)	1.509(4)
C(6A)-C(8A)	1.514(4)
C(10A)-C(11A)	1.505(4)
C(12A)-C(13A)	1.488(5)
C(10)-N(1)-C(12)	125.0(2)
C(10)-N(1)-C(9)	118.6(2)
C(12)-N(1)-C(9)	116.2(2)
C(1)-O(1)-C(2)	113.7(2)
C(4)-O(2)-C(6)	108.29(19)
C(6)-O(3)-C(3)	105.97(19)
C(1)-O(5)-C(11)	111.05(19)
O(5)-C(1)-O(1)	112.3(2)
O(5)-C(1)-C(5)	106.6(2)
O(1)-C(1)-C(5)	105.2(2)

O(5)-C(1)-C(9)	112.7(2)
O(1)-C(1)-C(9)	106.7(2)
C(5)-C(1)-C(9)	113.1(2)
O(1)-C(2)-C(3)	112.9(2)
O(3)-C(3)-C(2)	110.1(2)
O(3)-C(3)-C(4)	100.6(2)
C(2)-C(3)-C(4)	115.7(2)
O(2)-C(4)-C(5)	111.4(2)
O(2)-C(4)-C(3)	103.8(2)
C(5)-C(4)-C(3)	111.1(2)
O(4)-C(5)-C(4)	122.7(3)
O(4)-C(5)-C(1)	123.0(2)
C(4)-C(5)-C(1)	114.2(2)
O(3)-C(6)-O(2)	105.7(2)
O(3)-C(6)-C(8)	108.4(2)
O(2)-C(6)-C(8)	109.9(2)
O(3)-C(6)-C(7)	110.9(2)
O(2)-C(6)-C(7)	108.5(2)
C(8)-C(6)-C(7)	113.2(2)
N(1)-C(9)-C(1)	113.6(2)
O(6)-C(10)-N(1)	125.1(3)
O(6)-C(10)-C(11)	121.0(3)
N(1)-C(10)-C(11)	113.9(2)
O(5)-C(11)-C(10)	112.1(2)
O(7)-C(12)-N(1)	119.2(3)
O(7)-C(12)-C(13)	122.3(3)
N(1)-C(12)-C(13)	118.5(3)
C(10A)-N(1A)-C(12A)	124.0(2)
C(10A)-N(1A)-C(9A)	120.3(2)
C(12A)-N(1A)-C(9A)	115.3(2)
C(1A)-O(1A)-C(2A)	113.3(2)
C(4A)-O(2A)-C(6A)	108.02(19)
C(6A)-O(3A)-C(3A)	105.87(19)
C(1A)-O(5A)-C(11A)	111.1(2)
O(5A)-C(1A)-O(1A)	112.1(2)
O(5A)-C(1A)-C(9A)	112.0(2)

O(1A)-C(1A)-C(9A)	107.2(2)
O(5A)-C(1A)-C(5A)	105.8(2)
O(1A)-C(1A)-C(5A)	107.5(2)
C(9A)-C(1A)-C(5A)	112.3(2)
O(1A)-C(2A)-C(3A)	113.4(2)
O(3A)-C(3A)-C(2A)	110.4(2)
O(3A)-C(3A)-C(4A)	99.9(2)
C(2A)-C(3A)-C(4A)	116.1(2)
O(2A)-C(4A)-C(5A)	111.2(2)
O(2A)-C(4A)-C(3A)	104.2(2)
C(5A)-C(4A)-C(3A)	111.1(2)
O(4A)-C(5A)-C(4A)	123.3(3)
O(4A)-C(5A)-C(1A)	122.4(3)
C(4A)-C(5A)-C(1A)	114.1(2)
O(3A)-C(6A)-O(2A)	105.3(2)
O(3A)-C(6A)-C(7A)	108.4(2)
O(2A)-C(6A)-C(7A)	109.6(2)
O(3A)-C(6A)-C(8A)	111.4(2)
O(2A)-C(6A)-C(8A)	108.7(2)
C(7A)-C(6A)-C(8A)	113.2(2)
N(1A)-C(9A)-C(1A)	114.0(2)
O(6A)-C(10A)-N(1A)	124.4(3)
O(6A)-C(10A)-C(11A)	120.9(3)
N(1A)-C(10A)-C(11A)	114.6(2)
O(5A)-C(11A)-C(10A)	112.5(2)
O(7A)-C(12A)-N(1A)	118.9(3)
O(7A)-C(12A)-C(13A)	121.5(3)
N(1A)-C(12A)-C(13A)	119.5(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for ys143. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	12(1)	19(1)	16(1)	-1(1)	-2(1)	-3(1)
O(1)	13(1)	19(1)	16(1)	-4(1)	1(1)	0(1)
O(2)	14(1)	21(1)	20(1)	-9(1)	1(1)	-1(1)
O(3)	15(1)	15(1)	20(1)	-3(1)	3(1)	-3(1)
O(4)	30(1)	14(1)	37(1)	-1(1)	-6(1)	3(1)
O(5)	15(1)	16(1)	16(1)	1(1)	-1(1)	-3(1)
O(6)	18(1)	24(1)	22(1)	-6(1)	1(1)	-4(1)
O(7)	29(1)	29(1)	36(1)	15(1)	1(1)	4(1)
C(1)	14(1)	14(1)	18(1)	2(1)	2(1)	-1(1)
C(2)	16(1)	12(1)	23(1)	-2(1)	-3(1)	2(1)
C(3)	15(1)	14(1)	17(1)	2(1)	-2(1)	2(1)
C(4)	13(1)	16(1)	17(1)	-3(1)	0(1)	-3(1)
C(5)	14(1)	15(1)	18(1)	-5(1)	2(1)	2(1)
C(6)	16(1)	15(1)	18(1)	-4(1)	-2(1)	1(1)
C(7)	20(1)	24(2)	22(1)	-1(1)	-5(1)	-1(1)
C(8)	20(1)	20(2)	19(1)	-4(1)	2(1)	-1(1)
C(9)	14(1)	25(2)	22(1)	6(1)	-1(1)	-2(1)
C(10)	14(1)	17(1)	18(1)	-2(1)	4(1)	2(1)
C(11)	16(1)	11(1)	23(1)	1(1)	0(1)	-1(1)
C(12)	20(1)	26(2)	20(1)	8(1)	4(1)	2(1)
C(13)	16(2)	43(2)	48(2)	21(2)	-3(1)	2(1)
N(1A)	14(1)	14(1)	17(1)	-1(1)	-2(1)	-4(1)
O(1A)	16(1)	15(1)	18(1)	-2(1)	1(1)	1(1)
O(2A)	15(1)	22(1)	19(1)	-8(1)	2(1)	1(1)
O(3A)	16(1)	16(1)	14(1)	-3(1)	0(1)	-2(1)
O(4A)	29(1)	15(1)	28(1)	0(1)	-2(1)	-2(1)
O(5A)	17(1)	15(1)	22(1)	2(1)	2(1)	-3(1)
O(6A)	22(1)	26(1)	23(1)	-9(1)	5(1)	-10(1)
O(7A)	29(1)	25(1)	29(1)	13(1)	-5(1)	-2(1)
C(1A)	16(1)	11(1)	18(1)	1(1)	3(1)	0(1)
C(2A)	16(1)	13(1)	22(1)	-2(1)	2(1)	0(1)

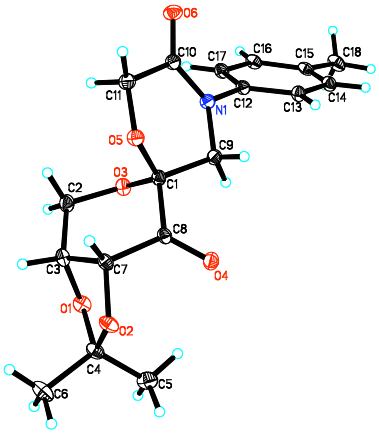
C(3A)	18(1)	15(1)	16(1)	0(1)	1(1)	2(1)
C(4A)	16(1)	16(1)	16(1)	-1(1)	2(1)	-1(1)
C(5A)	14(1)	15(1)	20(1)	-2(1)	6(1)	-3(1)
C(6A)	16(1)	16(1)	16(1)	-6(1)	1(1)	1(1)
C(7A)	27(2)	19(2)	28(2)	-9(1)	7(1)	-4(1)
C(8A)	24(2)	32(2)	19(1)	-2(1)	-3(1)	11(1)
C(9A)	18(1)	14(1)	26(1)	4(1)	-3(1)	-4(1)
C(10A)	18(1)	16(1)	19(1)	-4(1)	7(1)	-1(1)
C(11A)	18(1)	12(1)	32(2)	2(1)	2(1)	-2(1)
C(12A)	24(2)	29(2)	20(1)	11(1)	-1(1)	-3(1)
C(13A)	24(2)	50(3)	77(3)	40(2)	-19(2)	-11(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for ys143.

	x	y	z	U(eq)
H(2A)	2688	-1035	4685	21
H(2B)	952	-1077	5237	21
H(3A)	2941	-448	6423	19
H(4A)	803	693	6664	19
H(7A)	5174	340	7450	33
H(7B)	5916	1420	7616	33
H(7C)	6831	674	6913	33
H(8A)	4566	2299	5111	29
H(8B)	6446	1901	5435	29
H(8C)	5542	2659	6131	29
H(9A)	-1156	1801	3619	25
H(9B)	-750	856	2981	25
H(11A)	-3309	-994	4921	20
H(11B)	-1822	-1069	4172	20
H(13A)	-6733	1970	2453	53
H(13B)	-6687	1237	3379	53

H(13C)	-6303	841	2302	53
H(2AA)	-2197	-3486	-133	20
H(2AB)	-3865	-3611	470	20
H(3AA)	-1748	-3111	1652	20
H(4AA)	-3869	-2052	2123	20
H(7AA)	-313	-188	696	36
H(7AB)	1589	-608	889	36
H(7AC)	766	27	1728	36
H(8AA)	529	-2456	2700	38
H(8AB)	1301	-1405	2993	38
H(8AC)	2122	-2041	2154	38
H(9AA)	-6216	-563	-633	23
H(9AB)	-5629	-1315	-1447	23
H(11C)	-8125	-3533	268	25
H(11D)	-6621	-3542	-473	25
H(13D)	-11621	-504	-2066	77
H(13E)	-11573	-1216	-1124	77
H(13F)	-11168	-1634	-2189	77

The X-ray structure of ketone 3d



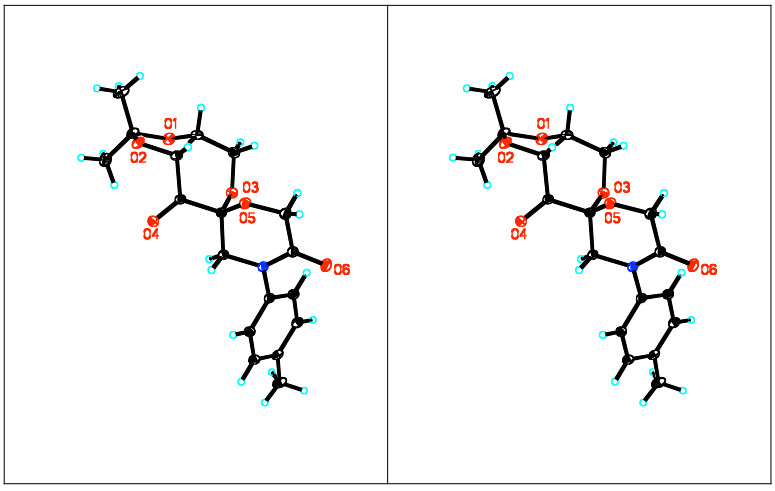


Table 1. Crystal data and structure refinement for **3d**.

Identification code	ys113	
Empirical formula	C18 H21 N O6	
Formula weight	347.36	
Temperature	373(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 9.5204(3) Å	α= 90°.
	b = 6.5582(2) Å	β= 91.632(2)°.
	c = 13.2707(4) Å	γ = 90°.
Volume	828.24(4) $Å^3$	
Z	2	
Density (calculated)	1.393 Mg/m^3	
Absorption coefficient	0.105 mm^{-1}	
F(000)	368	
Crystal size	0.40 x 0.31 x 0.16 mm^3	
Theta range for data collection	2.60 to 49.01°.	
Index ranges	-20<=h<=20, -13<=k<=13, -27<=l<=24	
Reflections collected	28445	
Independent reflections	14322 [R(int) = 0.0247]	
Completeness to theta = 49.01°	96.5 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9835 and 0.9597	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	14322 / 1 / 226	
Goodness-of-fit on F^2	0.825	
Final R indices [I>2sigma(I)]	R1 = 0.0382, wR2 = 0.1066	
R indices (all data)	R1 = 0.0492, wR2 = 0.1184	
Largest diff. peak and hole	0.471 and -0.262 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ys113. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	11973(1)	3563(1)	4665(1)	16(1)
O(2)	14032(1)	4785(1)	5387(1)	15(1)
O(3)	10314(1)	3072(1)	6537(1)	14(1)
O(4)	12435(1)	7059(1)	6685(1)	17(1)
O(5)	11824(1)	2872(1)	7974(1)	14(1)
O(6)	8883(1)	2038(1)	9582(1)	18(1)
C(1)	11136(1)	4151(1)	7262(1)	12(1)
C(2)	11063(1)	1496(1)	6024(1)	16(1)
C(3)	12348(1)	2281(1)	5501(1)	14(1)
C(4)	13207(1)	4731(1)	4447(1)	15(1)
C(5)	12765(1)	6870(1)	4153(1)	20(1)
C(6)	14067(1)	3676(1)	3658(1)	22(1)
C(7)	13260(1)	3767(1)	6125(1)	13(1)
C(8)	12319(1)	5222(1)	6703(1)	12(1)
C(9)	10169(1)	5643(1)	7776(1)	15(1)
C(10)	9566(1)	2823(1)	8912(1)	14(1)
C(11)	10873(1)	1724(1)	8566(1)	16(1)
C(12)	7863(1)	5524(1)	8608(1)	13(1)
C(13)	7843(1)	7525(1)	8958(1)	14(1)
C(14)	6558(1)	8507(1)	9091(1)	14(1)
C(15)	5284(1)	7519(1)	8874(1)	14(1)
C(16)	5327(1)	5484(1)	8554(1)	15(1)
C(17)	6602(1)	4491(1)	8413(1)	14(1)
C(18)	3904(1)	8636(1)	8945(1)	19(1)
N(1)	9178(1)	4549(1)	8411(1)	13(1)

Table 3. Bond lengths [Å] and angles [°] for ys113.

O(1)-C(3)	1.4298(8)
O(1)-C(4)	1.4386(7)
O(2)-C(7)	1.4090(7)
O(2)-C(4)	1.4547(7)
O(3)-C(1)	1.4131(7)
O(3)-C(2)	1.4379(7)
O(4)-C(8)	1.2099(7)
O(5)-C(1)	1.4103(7)
O(5)-C(11)	1.4300(8)
O(6)-C(10)	1.2292(8)
C(1)-C(9)	1.5182(7)
C(1)-C(8)	1.5361(7)
C(2)-C(3)	1.5136(9)
C(2)-H(2A)	0.9700
C(2)-H(2B)	0.9700
C(3)-C(7)	1.5324(8)
C(3)-H(3A)	0.9800
C(4)-C(5)	1.5131(9)
C(4)-C(6)	1.5148(9)
C(5)-H(5A)	0.9600
C(5)-H(5B)	0.9600
C(5)-H(5C)	0.9600
C(6)-H(6A)	0.9600
C(6)-H(6B)	0.9600
C(6)-H(6C)	0.9600
C(7)-C(8)	1.5302(7)
C(7)-H(7A)	0.9800
C(9)-N(1)	1.4700(7)
C(9)-H(9A)	0.9700
C(9)-H(9B)	0.9700
C(10)-N(1)	1.3584(7)
C(10)-C(11)	1.5202(8)
C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700

C(12)-C(13)	1.3931(8)
C(12)-C(17)	1.3958(8)
C(12)-N(1)	1.4359(7)
C(13)-C(14)	1.3980(7)
C(13)-H(13A)	0.9300
C(14)-C(15)	1.3975(8)
C(14)-H(14A)	0.9300
C(15)-C(16)	1.4015(9)
C(15)-C(18)	1.5097(8)
C(16)-C(17)	1.3947(8)
C(16)-H(16A)	0.9300
C(17)-H(17A)	0.9300
C(18)-H(18A)	0.9600
C(18)-H(18B)	0.9600
C(18)-H(18C)	0.9600
C(3)-O(1)-C(4)	106.36(4)
C(7)-O(2)-C(4)	107.77(4)
C(1)-O(3)-C(2)	114.20(4)
C(1)-O(5)-C(11)	113.10(4)
O(5)-C(1)-O(3)	113.26(4)
O(5)-C(1)-C(9)	111.05(5)
O(3)-C(1)-C(9)	107.29(4)
O(5)-C(1)-C(8)	105.22(4)
O(3)-C(1)-C(8)	107.43(5)
C(9)-C(1)-C(8)	112.63(4)
O(3)-C(2)-C(3)	113.02(5)
O(3)-C(2)-H(2A)	109.0
C(3)-C(2)-H(2A)	109.0
O(3)-C(2)-H(2B)	109.0
C(3)-C(2)-H(2B)	109.0
H(2A)-C(2)-H(2B)	107.8
O(1)-C(3)-C(2)	111.67(5)
O(1)-C(3)-C(7)	99.81(5)
C(2)-C(3)-C(7)	114.91(5)
O(1)-C(3)-H(3A)	110.0
C(2)-C(3)-H(3A)	110.0

C(7)-C(3)-H(3A)	110.0
O(1)-C(4)-O(2)	105.35(5)
O(1)-C(4)-C(5)	108.80(5)
O(2)-C(4)-C(5)	109.76(5)
O(1)-C(4)-C(6)	110.86(6)
O(2)-C(4)-C(6)	108.38(5)
C(5)-C(4)-C(6)	113.38(6)
C(4)-C(5)-H(5A)	109.5
C(4)-C(5)-H(5B)	109.5
H(5A)-C(5)-H(5B)	109.5
C(4)-C(5)-H(5C)	109.5
H(5A)-C(5)-H(5C)	109.5
H(5B)-C(5)-H(5C)	109.5
C(4)-C(6)-H(6A)	109.5
C(4)-C(6)-H(6B)	109.5
H(6A)-C(6)-H(6B)	109.5
C(4)-C(6)-H(6C)	109.5
H(6A)-C(6)-H(6C)	109.5
H(6B)-C(6)-H(6C)	109.5
O(2)-C(7)-C(8)	112.17(5)
O(2)-C(7)-C(3)	102.97(5)
C(8)-C(7)-C(3)	109.67(4)
O(2)-C(7)-H(7A)	110.6
C(8)-C(7)-H(7A)	110.6
C(3)-C(7)-H(7A)	110.6
O(4)-C(8)-C(7)	123.79(5)
O(4)-C(8)-C(1)	122.20(5)
C(7)-C(8)-C(1)	113.90(4)
N(1)-C(9)-C(1)	110.50(5)
N(1)-C(9)-H(9A)	109.6
C(1)-C(9)-H(9A)	109.6
N(1)-C(9)-H(9B)	109.6
C(1)-C(9)-H(9B)	109.6
H(9A)-C(9)-H(9B)	108.1
O(6)-C(10)-N(1)	124.10(5)
O(6)-C(10)-C(11)	118.29(5)

N(1)-C(10)-C(11)	117.46(5)
O(5)-C(11)-C(10)	116.91(5)
O(5)-C(11)-H(11A)	108.1
C(10)-C(11)-H(11A)	108.1
O(5)-C(11)-H(11B)	108.1
C(10)-C(11)-H(11B)	108.1
H(11A)-C(11)-H(11B)	107.3
C(13)-C(12)-C(17)	119.90(5)
C(13)-C(12)-N(1)	120.06(5)
C(17)-C(12)-N(1)	120.00(5)
C(12)-C(13)-C(14)	119.79(5)
C(12)-C(13)-H(13A)	120.1
C(14)-C(13)-H(13A)	120.1
C(15)-C(14)-C(13)	121.18(5)
C(15)-C(14)-H(14A)	119.4
C(13)-C(14)-H(14A)	119.4
C(14)-C(15)-C(16)	118.11(5)
C(14)-C(15)-C(18)	120.93(6)
C(16)-C(15)-C(18)	120.93(6)
C(17)-C(16)-C(15)	121.22(5)
C(17)-C(16)-H(16A)	119.4
C(15)-C(16)-H(16A)	119.4
C(16)-C(17)-C(12)	119.74(5)
C(16)-C(17)-H(17A)	120.1
C(12)-C(17)-H(17A)	120.1
C(15)-C(18)-H(18A)	109.5
C(15)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(15)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(10)-N(1)-C(12)	120.56(5)
C(10)-N(1)-C(9)	121.14(5)
C(12)-N(1)-C(9)	117.63(4)

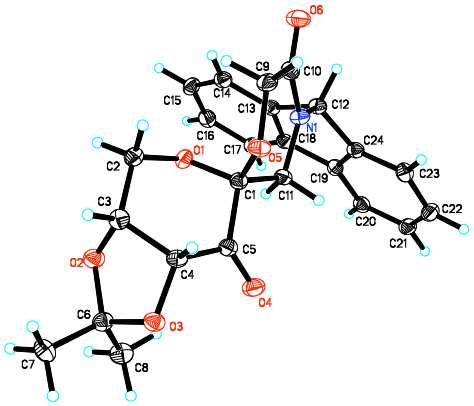
Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for ys113. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	12(1)	20(1)	15(1)	-1(1)	-1(1)	-1(1)
O(2)	9(1)	22(1)	13(1)	-1(1)	1(1)	0(1)
O(3)	10(1)	15(1)	18(1)	-2(1)	0(1)	0(1)
O(4)	17(1)	13(1)	22(1)	-2(1)	5(1)	-1(1)
O(5)	11(1)	16(1)	16(1)	2(1)	2(1)	3(1)
O(6)	16(1)	19(1)	20(1)	5(1)	6(1)	2(1)
C(1)	10(1)	12(1)	15(1)	-1(1)	2(1)	1(1)
C(2)	16(1)	13(1)	19(1)	-3(1)	1(1)	-1(1)
C(3)	14(1)	14(1)	15(1)	-3(1)	0(1)	2(1)
C(4)	11(1)	21(1)	13(1)	-1(1)	1(1)	2(1)
C(5)	14(1)	23(1)	23(1)	5(1)	2(1)	2(1)
C(6)	19(1)	33(1)	15(1)	-4(1)	3(1)	6(1)
C(7)	10(1)	16(1)	13(1)	-2(1)	1(1)	2(1)
C(8)	10(1)	13(1)	13(1)	-2(1)	1(1)	0(1)
C(9)	13(1)	12(1)	19(1)	1(1)	6(1)	2(1)
C(10)	12(1)	14(1)	15(1)	1(1)	2(1)	2(1)
C(11)	15(1)	16(1)	18(1)	3(1)	4(1)	5(1)
C(12)	10(1)	14(1)	14(1)	0(1)	2(1)	2(1)
C(13)	11(1)	14(1)	16(1)	-1(1)	2(1)	1(1)
C(14)	13(1)	15(1)	15(1)	-1(1)	2(1)	3(1)
C(15)	11(1)	20(1)	11(1)	0(1)	2(1)	3(1)
C(16)	11(1)	20(1)	14(1)	-1(1)	1(1)	0(1)
C(17)	12(1)	16(1)	15(1)	-2(1)	2(1)	1(1)
C(18)	13(1)	28(1)	17(1)	-2(1)	2(1)	7(1)
N(1)	11(1)	13(1)	16(1)	2(1)	4(1)	3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for ys113.

	x	y	z	U(eq)
H(2A)	10436	861	5529	19
H(2B)	11352	459	6509	19
H(3A)	12919	1134	5275	17
H(5A)	12224	7455	4680	30
H(5B)	12205	6824	3540	30
H(5C)	13585	7689	4052	30
H(6A)	14314	2331	3886	33
H(6B)	14906	4446	3548	33
H(6C)	13524	3583	3038	33
H(7A)	13892	3022	6590	15
H(9A)	10723	6582	8188	18
H(9B)	9651	6429	7270	18
H(11A)	11386	1231	9160	19
H(11B)	10574	539	8178	19
H(13A)	8681	8206	9103	16
H(14A)	6550	9842	9327	17
H(16A)	4490	4783	8434	18
H(17A)	6612	3147	8191	17
H(18A)	4074	10002	9177	29
H(18B)	3317	7940	9410	29
H(18C)	3443	8675	8292	29

The X-ray structure of ketone 3g



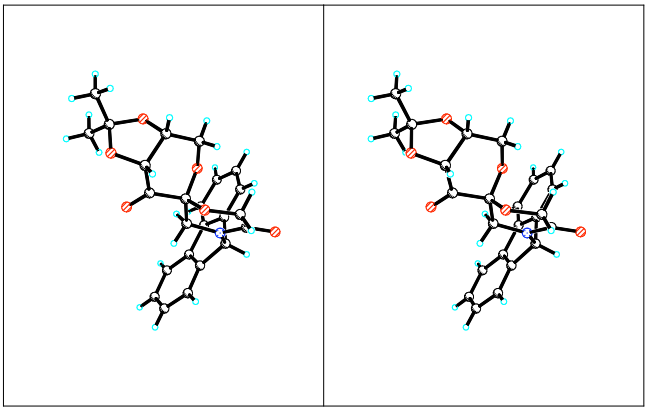


Table 1. Crystal data and structure refinement for **3g**.

Identification code	ys155r_0m	
Empirical formula	C24 H23 N O6	
Formula weight	421.43	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 6.7640(3) Å	α= 90°.
	b = 10.3317(3) Å	β= 90°.
	c = 28.5745(12) Å	γ = 90°.
Volume	1996.89(14) $Å^3$	
Z	4	
Density (calculated)	1.402 Mg/m^3	
Absorption coefficient	0.101 mm^{-1}	
F(000)	888	
Crystal size	0.20 x 0.14 x 0.10 mm^3	
Theta range for data collection	3.47 to 33.12°.	
Index ranges	-10<=h<=4, -12<=k<=15, -37<=l<=43	
Reflections collected	15723	
Independent reflections	7334 [R(int) = 0.0831]	
Completeness to theta = 33.12°	98.0 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9899 and 0.9799	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7334 / 0 / 280	
Goodness-of-fit on F^2	0.967	
Final R indices [I>2sigma(I)]	R1 = 0.0678, wR2 = 0.1194	
R indices (all data)	R1 = 0.1245, wR2 = 0.1442	
Absolute structure parameter	1.1(11)	
Largest diff. peak and hole	0.370 and -0.335 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ys155r_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	4172(3)	1002(2)	783(1)	17(1)
O(1)	353(2)	2092(2)	1079(1)	17(1)
O(2)	-2644(3)	2875(2)	1810(1)	21(1)
O(3)	-503(3)	3892(2)	2318(1)	19(1)
O(4)	2347(3)	2072(2)	2122(1)	24(1)
O(5)	3118(2)	3470(2)	1091(1)	19(1)
O(6)	4462(3)	1731(2)	36(1)	26(1)
C(1)	2184(4)	2387(2)	1295(1)	15(1)
C(2)	-998(4)	3168(3)	1065(1)	19(1)
C(3)	-1432(4)	3732(3)	1541(1)	19(1)
C(4)	354(4)	3877(2)	1868(1)	16(1)
C(5)	1741(3)	2732(2)	1804(1)	17(1)
C(6)	-2372(4)	3202(2)	2295(1)	19(1)
C(7)	-3953(4)	4111(3)	2468(1)	23(1)
C(8)	-2269(4)	1944(3)	2565(1)	24(1)
C(9)	3497(4)	3279(2)	598(1)	19(1)
C(10)	4082(4)	1920(3)	451(1)	19(1)
C(11)	3473(3)	1204(3)	1262(1)	17(1)
C(12)	4671(4)	-329(2)	651(1)	16(1)
C(13)	2884(4)	-1195(2)	589(1)	17(1)
C(14)	1218(4)	-1008(3)	316(1)	19(1)
C(15)	-198(4)	-1974(3)	298(1)	21(1)
C(16)	51(4)	-3123(3)	548(1)	23(1)
C(17)	1704(4)	-3318(3)	828(1)	19(1)
C(18)	3119(4)	-2338(2)	850(1)	18(1)
C(19)	4956(3)	-2229(2)	1125(1)	17(1)
C(20)	5785(4)	-3049(3)	1457(1)	19(1)
C(21)	7467(4)	-2647(3)	1693(1)	22(1)
C(22)	8330(4)	-1455(3)	1600(1)	23(1)
C(23)	7539(4)	-649(2)	1259(1)	20(1)
C(24)	5840(4)	-1037(2)	1024(1)	16(1)

Table 3. Bond lengths [Å] and angles [°] for ys155r_0m.

N(1)-C(10)	1.343(3)
N(1)-C(11)	1.463(3)
N(1)-C(12)	1.466(3)
O(1)-C(1)	1.417(3)
O(1)-C(2)	1.439(3)
O(2)-C(3)	1.431(3)
O(2)-C(6)	1.438(3)
O(3)-C(4)	1.411(3)
O(3)-C(6)	1.453(3)
O(4)-C(5)	1.209(3)
O(5)-C(1)	1.411(3)
O(5)-C(9)	1.444(3)
O(6)-C(10)	1.231(3)
C(1)-C(11)	1.505(3)
C(1)-C(5)	1.527(3)
C(2)-C(3)	1.509(3)
C(3)-C(4)	1.534(3)
C(4)-C(5)	1.521(3)
C(6)-C(7)	1.507(4)
C(6)-C(8)	1.514(3)
C(9)-C(10)	1.518(4)
C(12)-C(13)	1.514(3)
C(12)-C(24)	1.517(3)
C(13)-C(14)	1.384(3)
C(13)-C(18)	1.405(3)
C(14)-C(15)	1.385(4)
C(15)-C(16)	1.395(4)
C(16)-C(17)	1.390(3)
C(17)-C(18)	1.395(3)
C(18)-C(19)	1.474(3)
C(19)-C(20)	1.390(3)
C(19)-C(24)	1.399(3)
C(20)-C(21)	1.387(4)

C(21)-C(22)	1.389(4)
C(22)-C(23)	1.388(3)
C(23)-C(24)	1.390(3)
C(10)-N(1)-C(11)	123.1(2)
C(10)-N(1)-C(12)	119.42(19)
C(11)-N(1)-C(12)	116.72(19)
C(1)-O(1)-C(2)	113.66(18)
C(3)-O(2)-C(6)	107.37(18)
C(4)-O(3)-C(6)	108.12(17)
C(1)-O(5)-C(9)	111.94(17)
O(5)-C(1)-O(1)	112.49(18)
O(5)-C(1)-C(11)	111.05(18)
O(1)-C(1)-C(11)	107.78(19)
O(5)-C(1)-C(5)	107.23(18)
O(1)-C(1)-C(5)	107.06(18)
C(11)-C(1)-C(5)	111.20(19)
O(1)-C(2)-C(3)	113.33(18)
O(2)-C(3)-C(2)	110.9(2)
O(2)-C(3)-C(4)	100.66(18)
C(2)-C(3)-C(4)	115.7(2)
O(3)-C(4)-C(5)	111.79(19)
O(3)-C(4)-C(3)	103.42(19)
C(5)-C(4)-C(3)	109.70(19)
O(4)-C(5)-C(4)	123.9(2)
O(4)-C(5)-C(1)	121.3(2)
C(4)-C(5)-C(1)	114.61(19)
O(2)-C(6)-O(3)	105.69(18)
O(2)-C(6)-C(7)	111.9(2)
O(3)-C(6)-C(7)	107.30(19)
O(2)-C(6)-C(8)	107.2(2)
O(3)-C(6)-C(8)	111.0(2)
C(7)-C(6)-C(8)	113.6(2)
O(5)-C(9)-C(10)	116.26(19)
O(6)-C(10)-N(1)	124.0(2)
O(6)-C(10)-C(9)	118.0(2)
N(1)-C(10)-C(9)	118.03(19)

N(1)-C(11)-C(1)	111.13(19)
N(1)-C(12)-C(13)	113.6(2)
N(1)-C(12)-C(24)	112.99(19)
C(13)-C(12)-C(24)	102.32(19)
C(14)-C(13)-C(18)	120.6(2)
C(14)-C(13)-C(12)	129.3(2)
C(18)-C(13)-C(12)	110.1(2)
C(13)-C(14)-C(15)	118.9(2)
C(14)-C(15)-C(16)	120.7(2)
C(17)-C(16)-C(15)	121.0(2)
C(16)-C(17)-C(18)	118.2(2)
C(17)-C(18)-C(13)	120.6(2)
C(17)-C(18)-C(19)	131.1(2)
C(13)-C(18)-C(19)	108.3(2)
C(20)-C(19)-C(24)	120.3(2)
C(20)-C(19)-C(18)	131.1(2)
C(24)-C(19)-C(18)	108.5(2)
C(21)-C(20)-C(19)	118.8(2)
C(20)-C(21)-C(22)	121.1(2)
C(23)-C(22)-C(21)	120.4(2)
C(22)-C(23)-C(24)	119.0(2)
C(23)-C(24)-C(19)	120.6(2)
C(23)-C(24)-C(12)	129.2(2)
C(19)-C(24)-C(12)	110.2(2)

Symmetry transformations used to generate equivalent atoms:

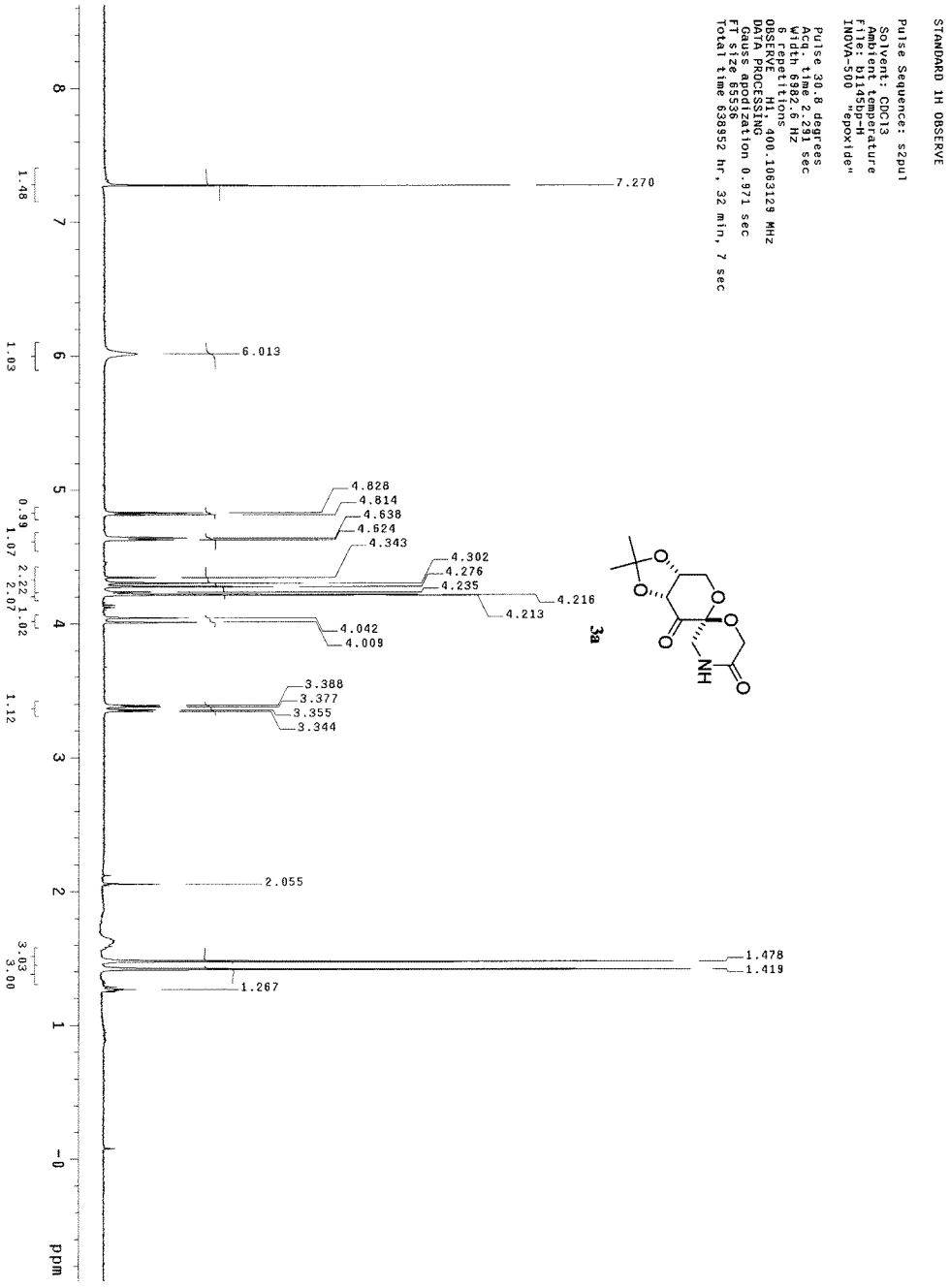
Table 4. Anisotropic displacement parameters ($Å^2$x 10^3)for ys155r_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

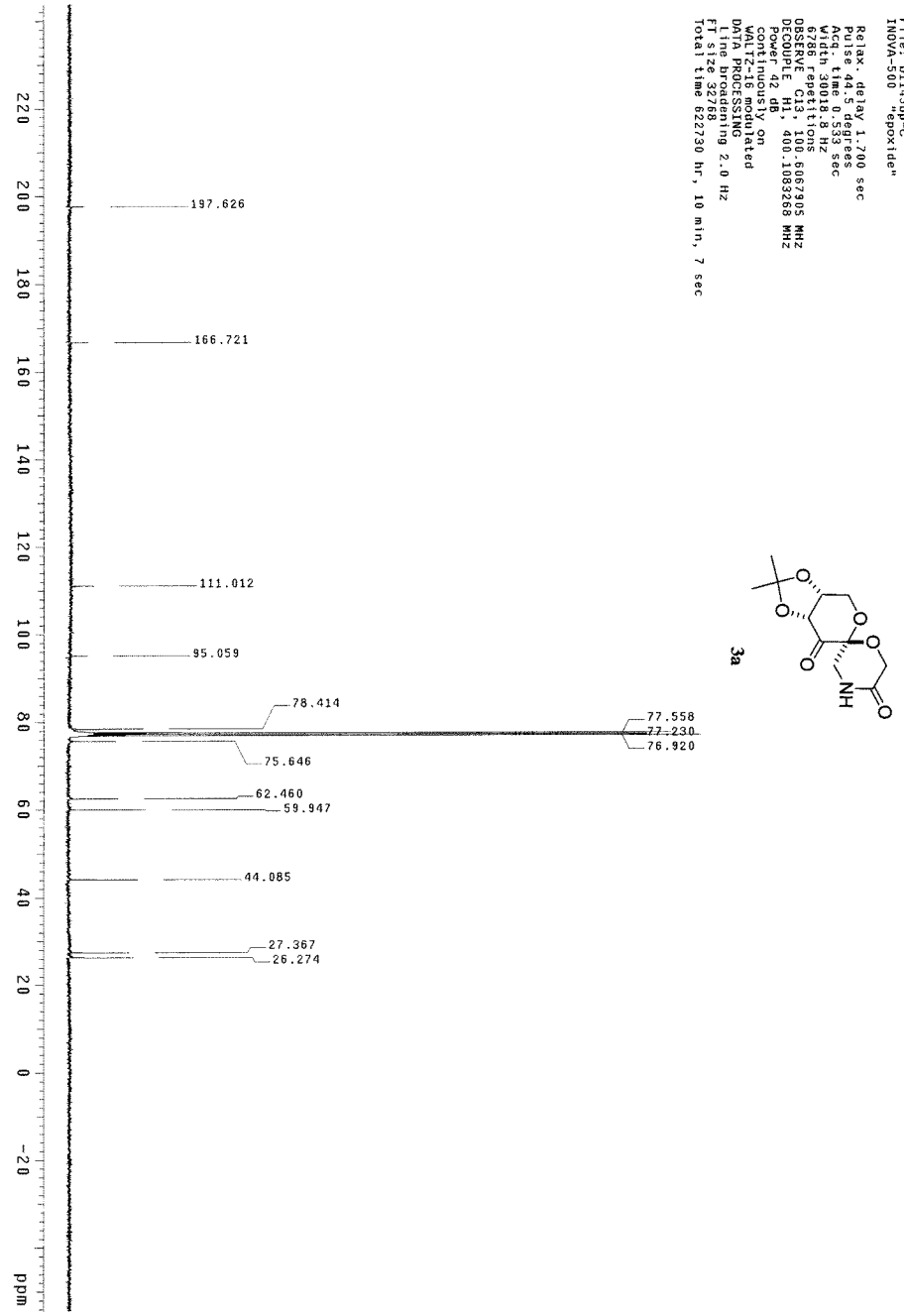
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	22(1)	15(1)	14(1)	1(1)	0(1)	1(1)
O(1)	16(1)	18(1)	18(1)	-1(1)	-4(1)	1(1)
O(2)	21(1)	25(1)	18(1)	-3(1)	-1(1)	-4(1)
O(3)	20(1)	21(1)	17(1)	-3(1)	1(1)	-4(1)

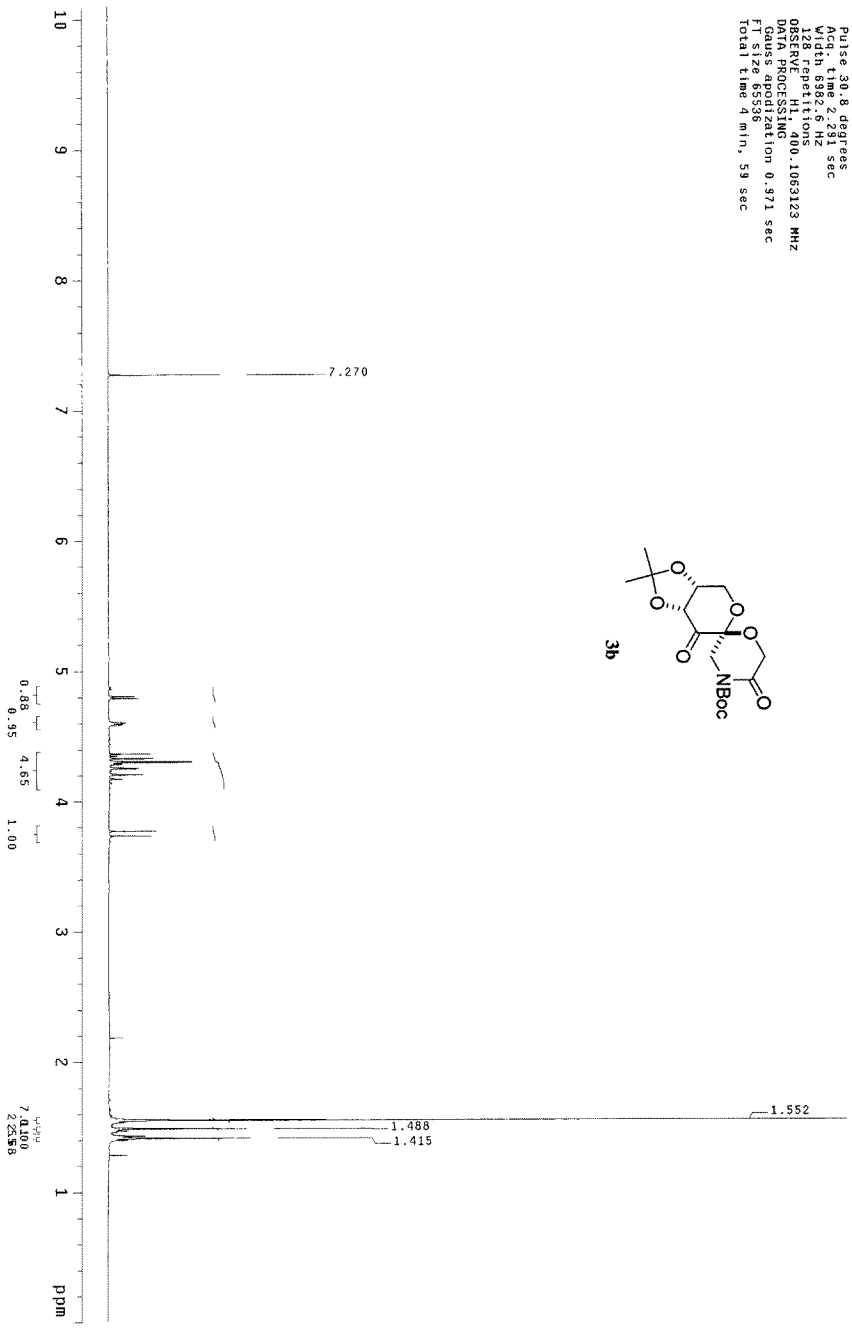
O(4)	28(1)	26(1)	16(1)	2(1)	-2(1)	4(1)
O(5)	24(1)	16(1)	16(1)	2(1)	2(1)	-2(1)
O(6)	32(1)	28(1)	16(1)	3(1)	4(1)	6(1)
C(1)	16(1)	15(1)	14(1)	1(1)	0(1)	-1(1)
C(2)	20(1)	20(1)	18(1)	2(1)	-1(1)	3(1)
C(3)	21(1)	16(1)	20(1)	0(1)	0(1)	1(1)
C(4)	20(1)	15(1)	14(1)	-1(1)	1(1)	-1(1)
C(5)	16(1)	19(1)	15(1)	-3(1)	0(1)	-6(1)
C(6)	20(1)	19(1)	17(1)	-1(1)	-1(1)	-4(1)
C(7)	23(1)	22(2)	25(1)	-3(1)	5(1)	3(1)
C(8)	28(1)	21(1)	23(1)	2(1)	0(1)	-3(1)
C(9)	21(1)	20(1)	17(1)	5(1)	-1(1)	-1(1)
C(10)	18(1)	19(1)	19(1)	2(1)	0(1)	-2(1)
C(11)	18(1)	20(1)	13(1)	-1(1)	2(1)	2(1)
C(12)	20(1)	14(1)	15(1)	-1(1)	1(1)	1(1)
C(13)	19(1)	17(1)	14(1)	-4(1)	1(1)	1(1)
C(14)	22(1)	19(1)	17(1)	-2(1)	1(1)	5(1)
C(15)	21(1)	25(2)	18(1)	-5(1)	-4(1)	4(1)
C(16)	21(1)	25(2)	23(1)	-6(1)	1(1)	-2(1)
C(17)	20(1)	18(1)	18(1)	-1(1)	1(1)	1(1)
C(18)	19(1)	19(1)	14(1)	-3(1)	2(1)	3(1)
C(19)	17(1)	18(1)	17(1)	-4(1)	1(1)	4(1)
C(20)	19(1)	19(1)	19(1)	1(1)	3(1)	3(1)
C(21)	22(1)	28(2)	17(1)	3(1)	-1(1)	9(1)
C(22)	21(1)	29(2)	19(1)	-2(1)	-3(1)	5(1)
C(23)	18(1)	21(1)	22(1)	-1(1)	2(1)	1(1)
C(24)	19(1)	16(1)	15(1)	-2(1)	2(1)	3(1)

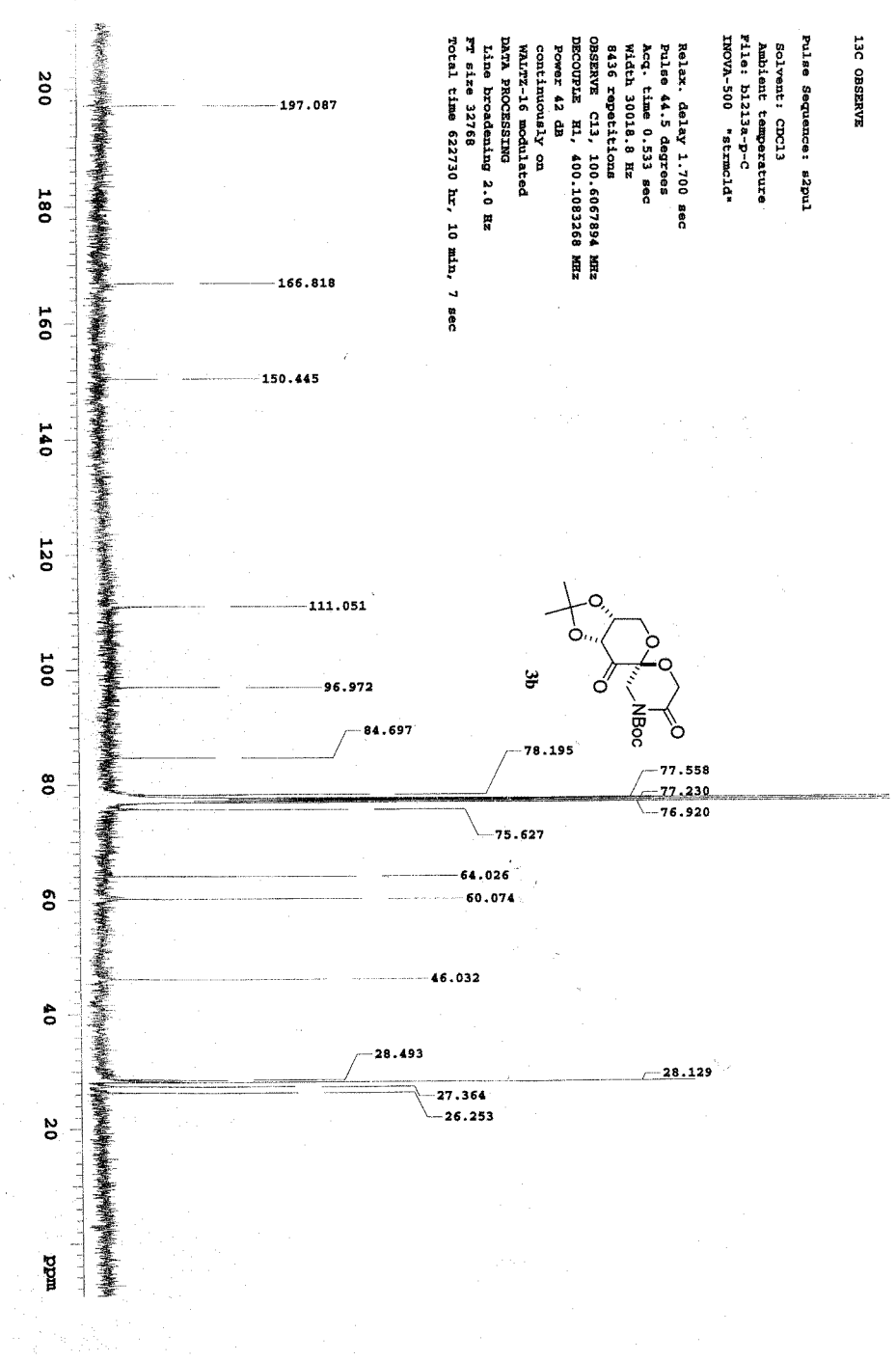
Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for ys155r_0m.

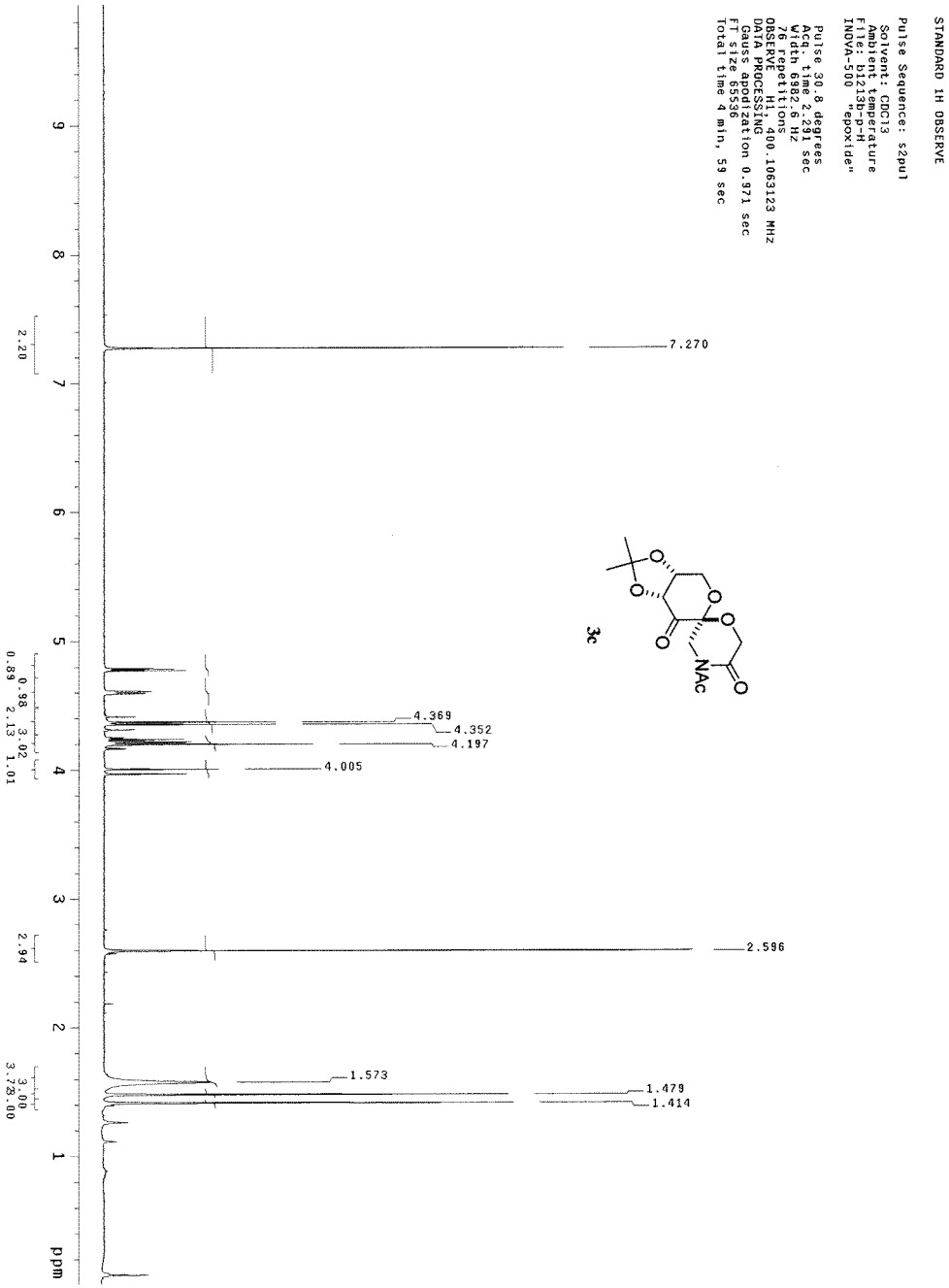
	x	y	z	U(eq)
H(2A)	-2255	2880	921	23
H(2B)	-434	3854	863	23
H(3A)	-2110	4586	1505	23
H(4A)	1062	4708	1805	20
H(7A)	-3939	4903	2280	35
H(7B)	-3700	4327	2797	35
H(7C)	-5248	3692	2441	35
H(8A)	-1215	1400	2436	37
H(8B)	-3536	1489	2539	37
H(8C)	-1991	2128	2895	37
H(9A)	4565	3879	503	23
H(9B)	2294	3526	423	23
H(11A)	4623	1304	1474	20
H(11B)	2715	436	1366	20
H(12A)	5450	-317	353	20
H(14A)	1048	-229	144	23
H(15A)	-1352	-1854	114	25
H(16A)	-923	-3783	526	27
H(17A)	1865	-4097	1000	23
H(20A)	5210	-3869	1520	23
H(21A)	8038	-3196	1923	27
H(22A)	9468	-1191	1769	28
H(23A)	8148	156	1188	24


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1145Dp-H
INOVA-500 "epoxide"
Pulse 30.8 degrees
Acq. time 2.291 sec
Width 6982.6 HZ
6 repetitions
OBSERVE H1, 400.1083129 MHZ
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 85536
Total time 838952 hr, 32 min, 7 sec
7.270
6.013
4.828
4.814
4.638
4.624
4.343
4.302
4.276
4.235
4.216
4.213
4.042
4.009
3.388
3.377
3.355
3.344
2.055
1.478
1.419
1.267
1.48
1.03
0.99
1.07
2.22
2.07
1.02
1.12
3.03
3.00
ppm
3a
NH


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1145bp-C
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
6786 repetitions
OBSERVE C13, 100.6067905 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec
197.626
166.721
111.012
95.059
78.414
77.558
77.230
76.920
75.646
62.460
59.947
44.085
27.367
26.274
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm
3a


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b123a-H
INOVA-500 "epoxide"
Pulse 30.8 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
128 repetitions
OBSERVE H1, 400.1063123 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 4 min, 59 sec
3b
NBoc
7.270
1.552
1.488
1.415
0.88
0.95
4.65
1.00
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1213a-p-C
INOVA-500 "strmcld"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
8436 repetitions
OBSERVE C13, 100.6067894 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec
197.087
166.818
150.445
111.051
96.972
84.697
78.195
77.558
77.230
76.920
75.627
64.026
60.074
46.032
28.493
28.129
27.364
26.253
3b
NBoc
200
180
160
140
120
100
80
60
40
20
ppm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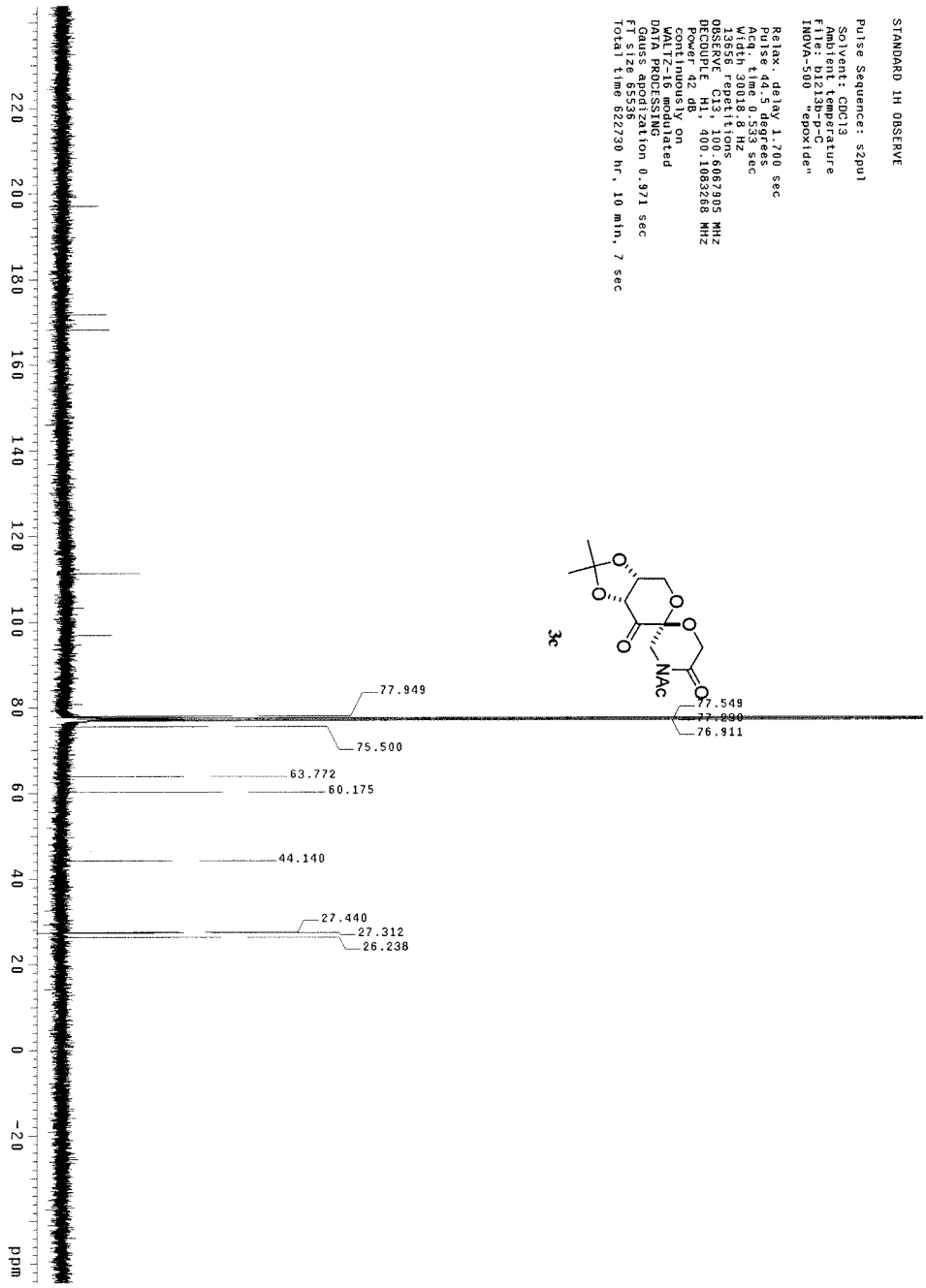
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1213b-p-H
INOVA-500 "epoxide"
Pulse 30.8 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
76 repetitions
OBSERVE H1, 400.1063123 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 4 min, 59 sec
7.270
4.369
4.352
4.197
4.005
2.596
1.573
1.479
1.414
2.20
0.89
0.98
2.13
3.02
1.01
2.94
3.72
3.00
1.00
3c
NAc
ppm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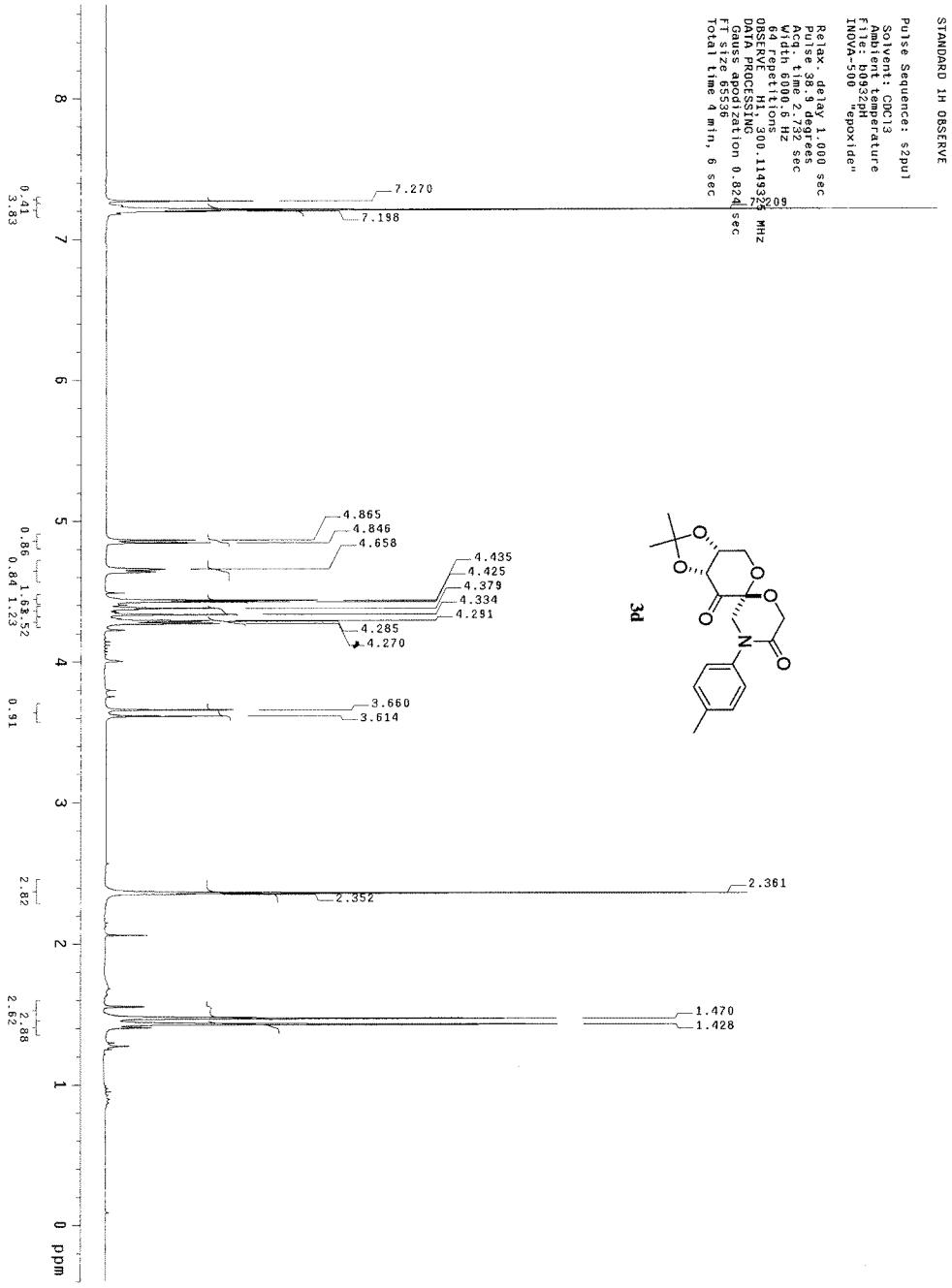
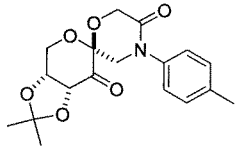
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1213b-p-C
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
13656 repetitions
OBSERVE C13, 100.6067905 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 622730 hr, 10 min, 7 sec
3c
NAc
77.949
77.549
77.230
76.911
75.500
63.772
60.175
44.140
27.440
27.312
26.238
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b0932pH
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 38.9 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
64 repetitions
OBSERVE H1, 300.1149325 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 4 min, 6 sec
7.270
7.198
4.865
4.846
4.658
4.435
4.425
4.379
4.334
4.291
4.285
4.270
3.660
3.614
2.361
2.352
1.470
1.428
3d
0.41
3.83
0.86
0.84
1.63
1.52
1.23
0.91
2.82
2.88
2.62
8
7
6
5
4
3
2
1
0 ppm

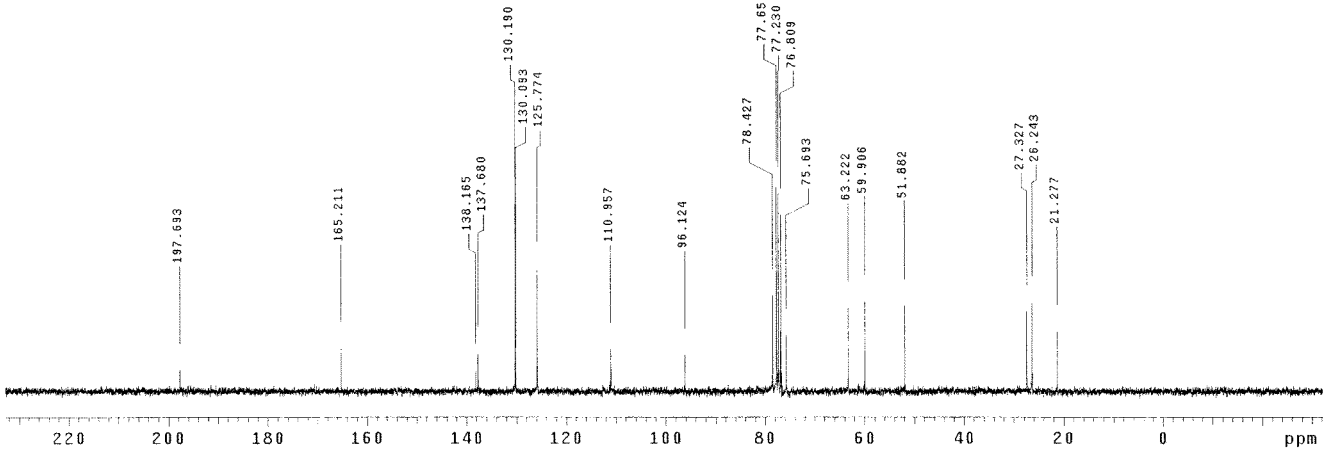
13C OBSERVE

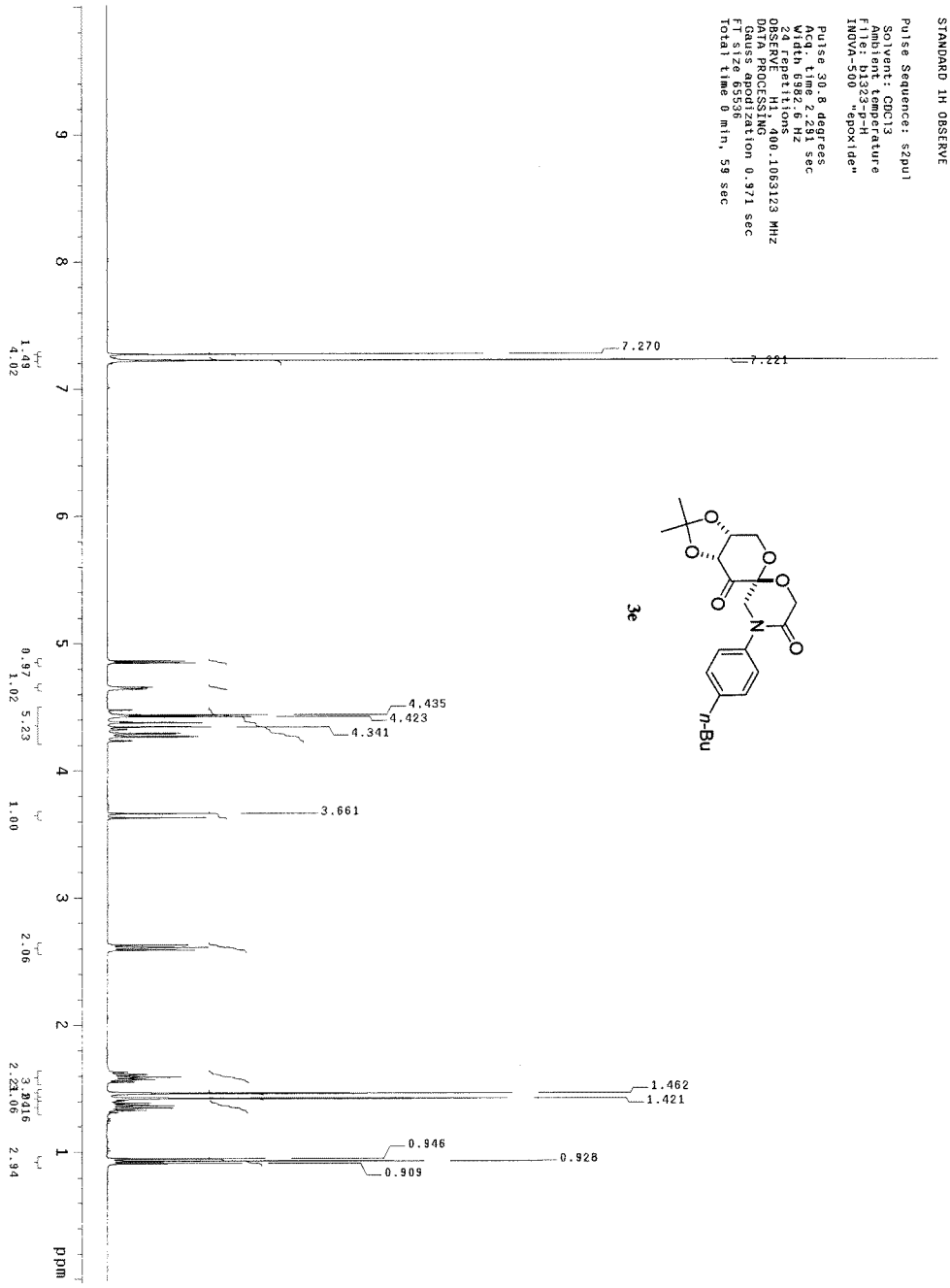
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b0932pC
INOVA-500 "epoxide"

Relax. delay 1.500 sec
Pulse 42.4 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
552 repetitions
OBSERVE C13, 75.4639380 MHz
DECOUPLE H1, 300.1184227 MHz
Power 32 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 641343 hr, 30 min, 7 sec



3d

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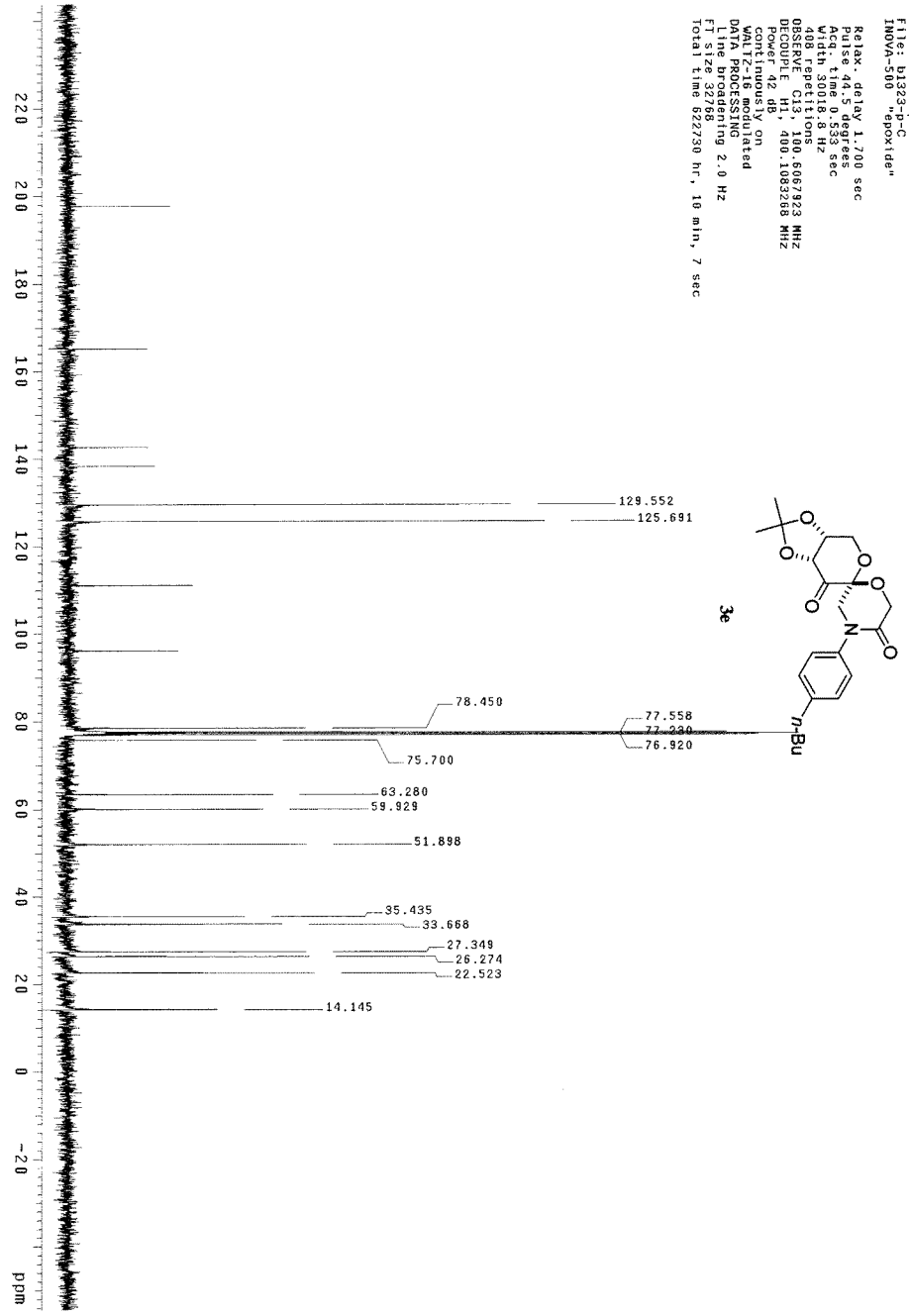
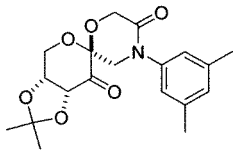
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temprature
File: b1323-p-H
INOVA-500 "epoxide"
Pulse 30.8 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
24 repetitions
OBSERVE H1, 400.1063123 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 59 sec
7.270
7.221
4.435
4.423
4.341
3.661
1.462
1.421
0.946
0.928
0.909
1.49
4.02
0.97
1.02
5.23
1.00
2.06
3.16
2.23
1.06
2.94
3e
n-Bu
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temprature
File: b1323-p-C
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
408 repetitions
OBSERVE C13, 100.6067923 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec
129.552
125.691
78.450
77.558
77.230
76.920
75.700
63.280
59.929
51.898
35.435
33.668
27.349
26.274
22.523
14.145
3e
n-Bu
ppm

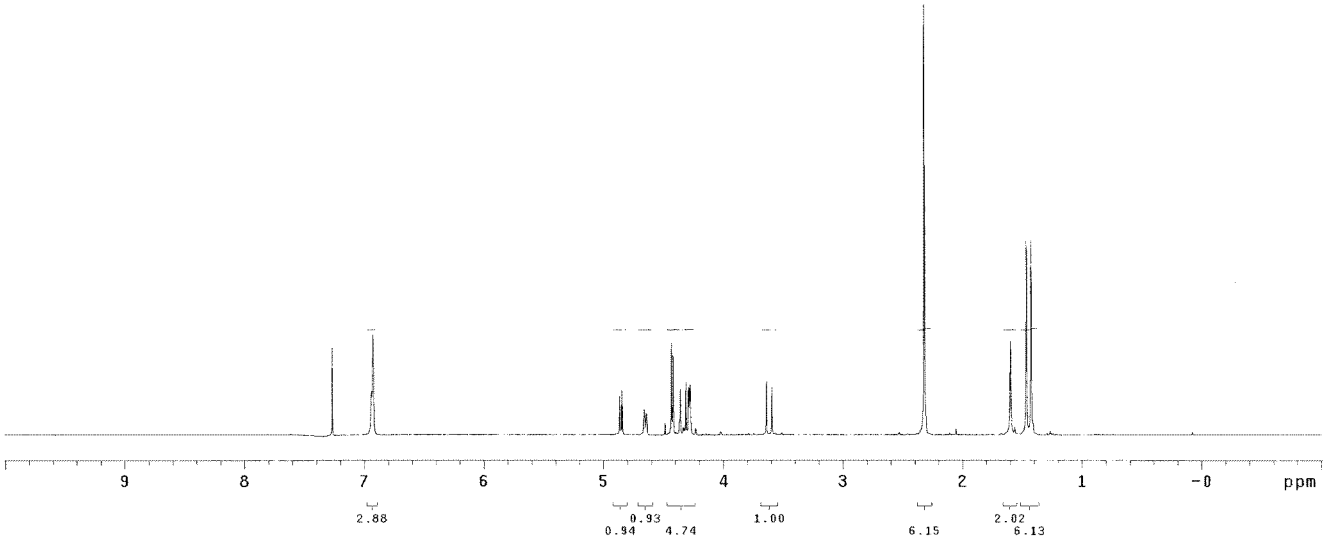
STANDARD 1H OBSERVE

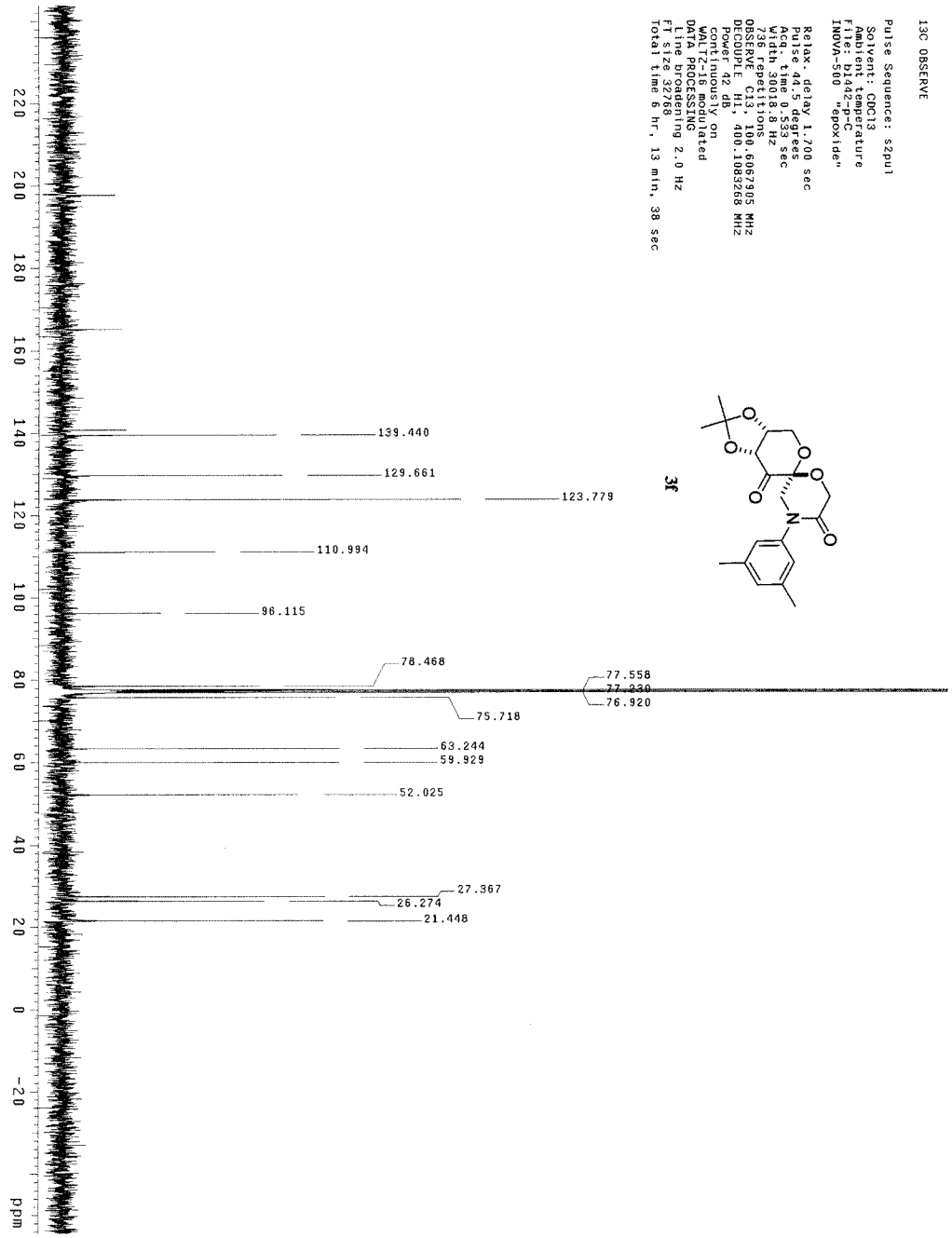
Pulse Sequence: s2pul
 Solvent: cdcl3
 Ambient temperature
File: b1442-p-H
INOVA-500 "epoxide"

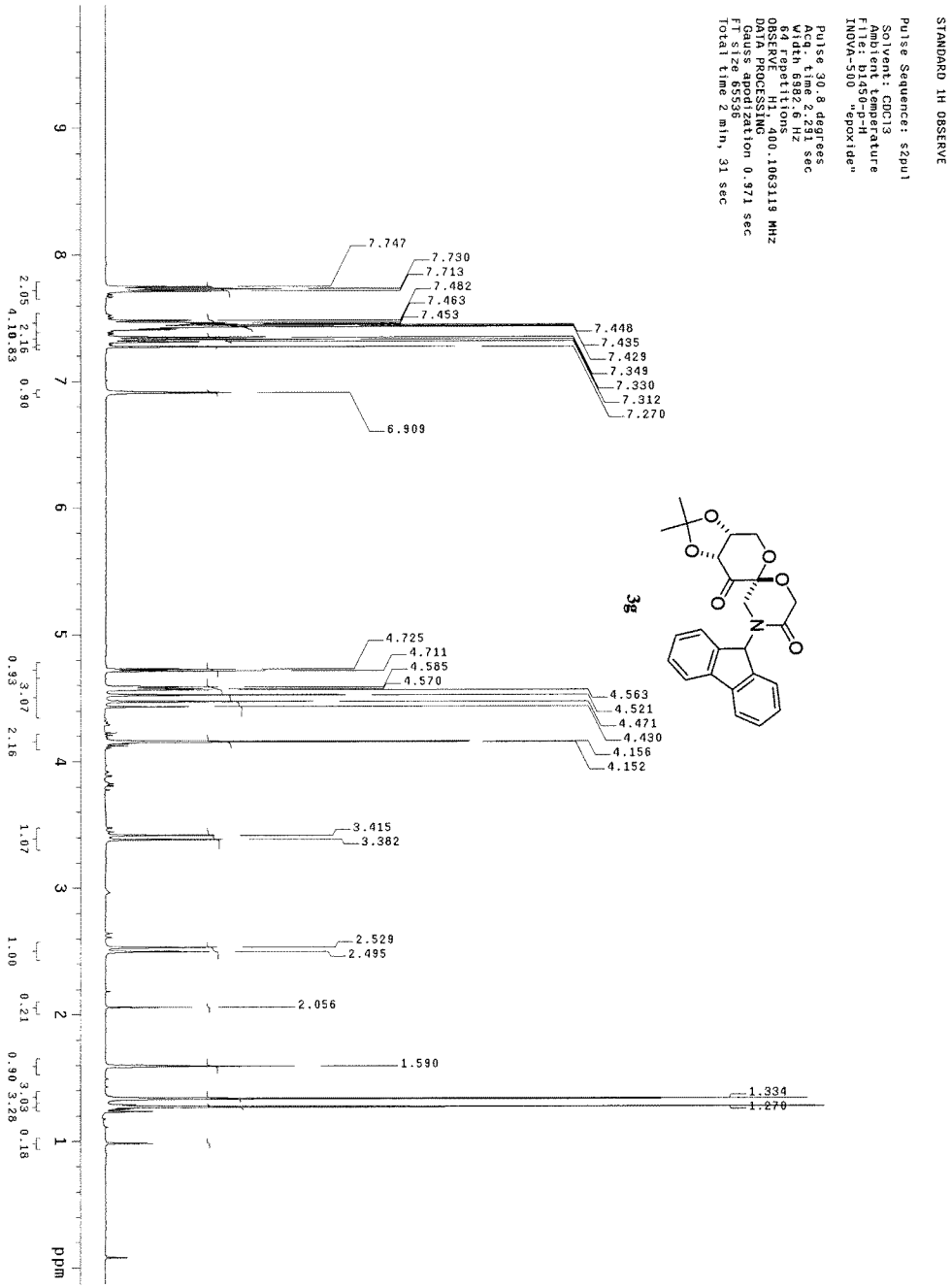
 Relax. delay 1.000 sec
 Pulse 34.0 degrees
 Acq. time 2.732 sec
 Width 6000.6 Hz
 28 repetitions
OBSERVE H1, 299.9533664 MHz
DATA PROCESSING
 Gauss apodization 0.824 sec
FT size 65536
Total time 4 min, 6 sec

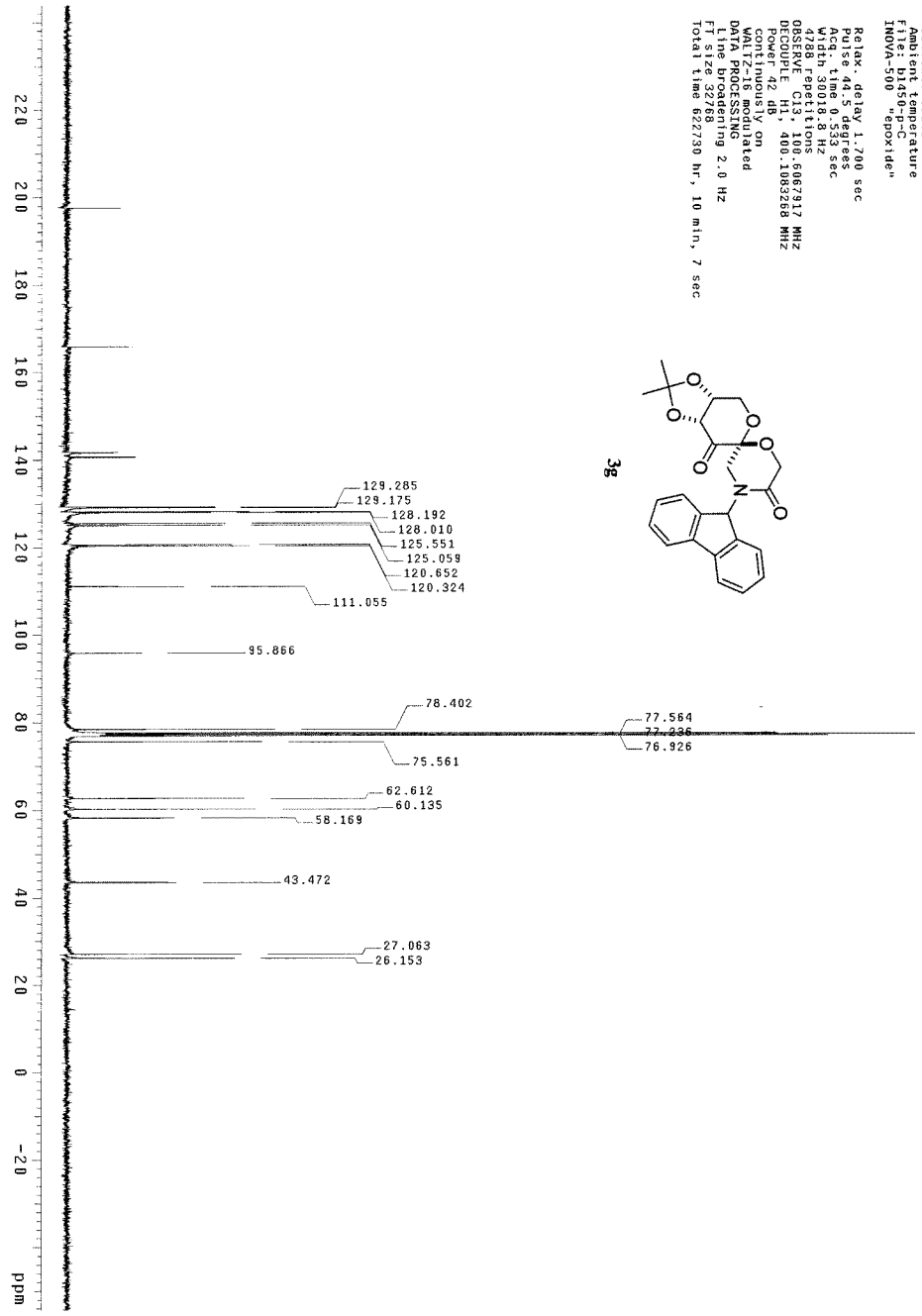


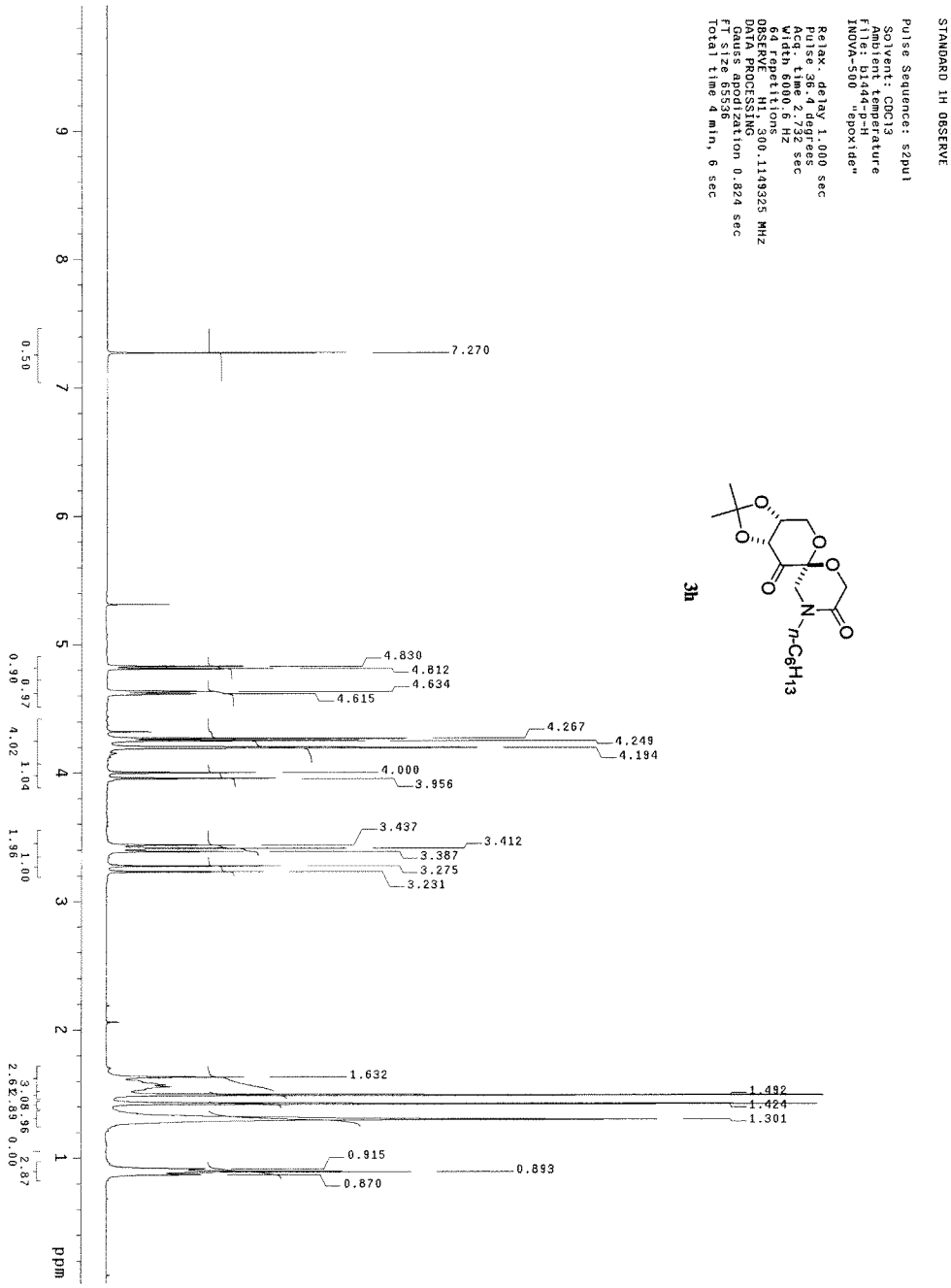
3f

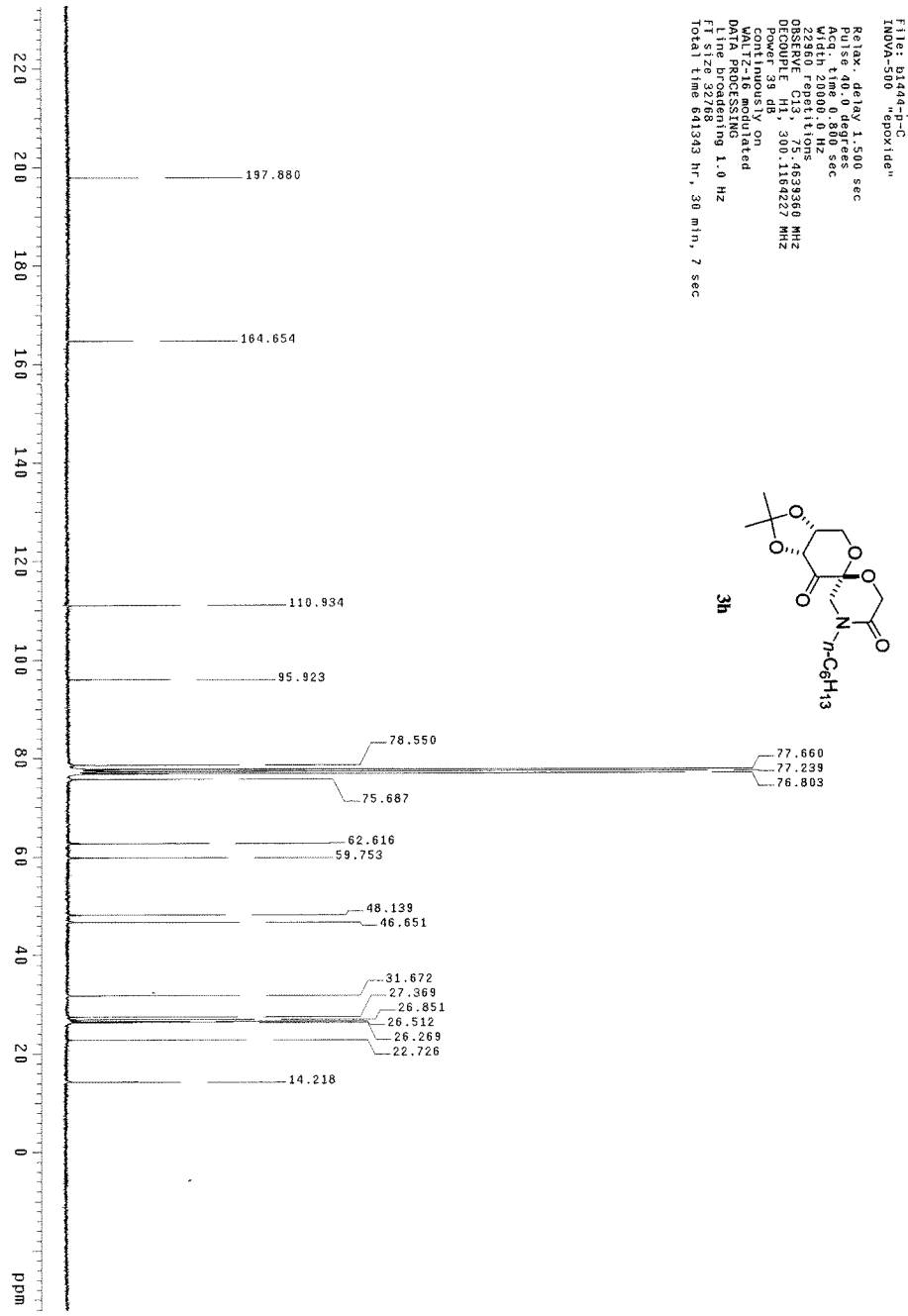



13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: bl442-p-C
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
736 repetitions
OBSERVE C13, 100.6067905 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 6 hr, 13 min, 38 sec
3f
139.440
129.661
123.779
110.994
96.115
78.468
77.558
77.230
76.920
75.718
63.244
59.929
52.025
27.367
26.274
21.448
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1450-p-H
INOVA-500 "epoxide"
Pulse 30.8 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
64 repetitions
OBSERVE H1, 400.1063119 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 2 min, 31 sec
7.747
7.730
7.713
7.482
7.463
7.453
7.448
7.435
7.429
7.349
7.330
7.312
7.270
6.909
4.725
4.711
4.585
4.570
4.563
4.521
4.471
4.430
4.156
4.152
3.415
3.382
2.529
2.495
2.056
1.590
1.334
1.270
3g
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1450-p-C
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
4788 repetitions
OBSERVE C13, 100.6067917 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec
3g
129.285
129.175
128.192
128.010
125.551
125.059
120.652
120.324
111.055
95.866
78.402
77.564
77.236
76.926
75.561
62.612
60.135
58.169
43.472
27.063
26.153
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1444-p-H
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 36.4 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
64 repetitions
OBSERVE H1, 300.1149325 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 4 min, 6 sec
3h
n-C6H13
7.270
4.830
4.812
4.634
4.615
4.267
4.249
4.194
4.000
3.956
3.437
3.412
3.387
3.275
3.231
1.632
1.492
1.424
1.301
0.915
0.893
0.870
0.50
0.90
0.97
4.02
1.04
1.96
1.00
2.61
2.89
3.08
0.96
0.00
2.87
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1444-p-C
INOVA-500 "epoxide"
Relax. delay 1.500 sec
Pulse 40.0 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
22960 repetitions
OBSERVE C13, 75.4639360 MHz
DECOUPLE H1, 300.1164227 MHz
Power 39 dB
Continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 641343 hr, 30 min, 7 sec
197.880
164.654
110.934
95.923
78.550
77.660
77.239
76.803
75.687
62.616
59.753
48.139
46.651
31.672
27.369
26.851
26.512
26.269
22.726
14.218
220
200
180
160
140
120
100
80
60
40
20
0
ppm
3h
n-C6H13

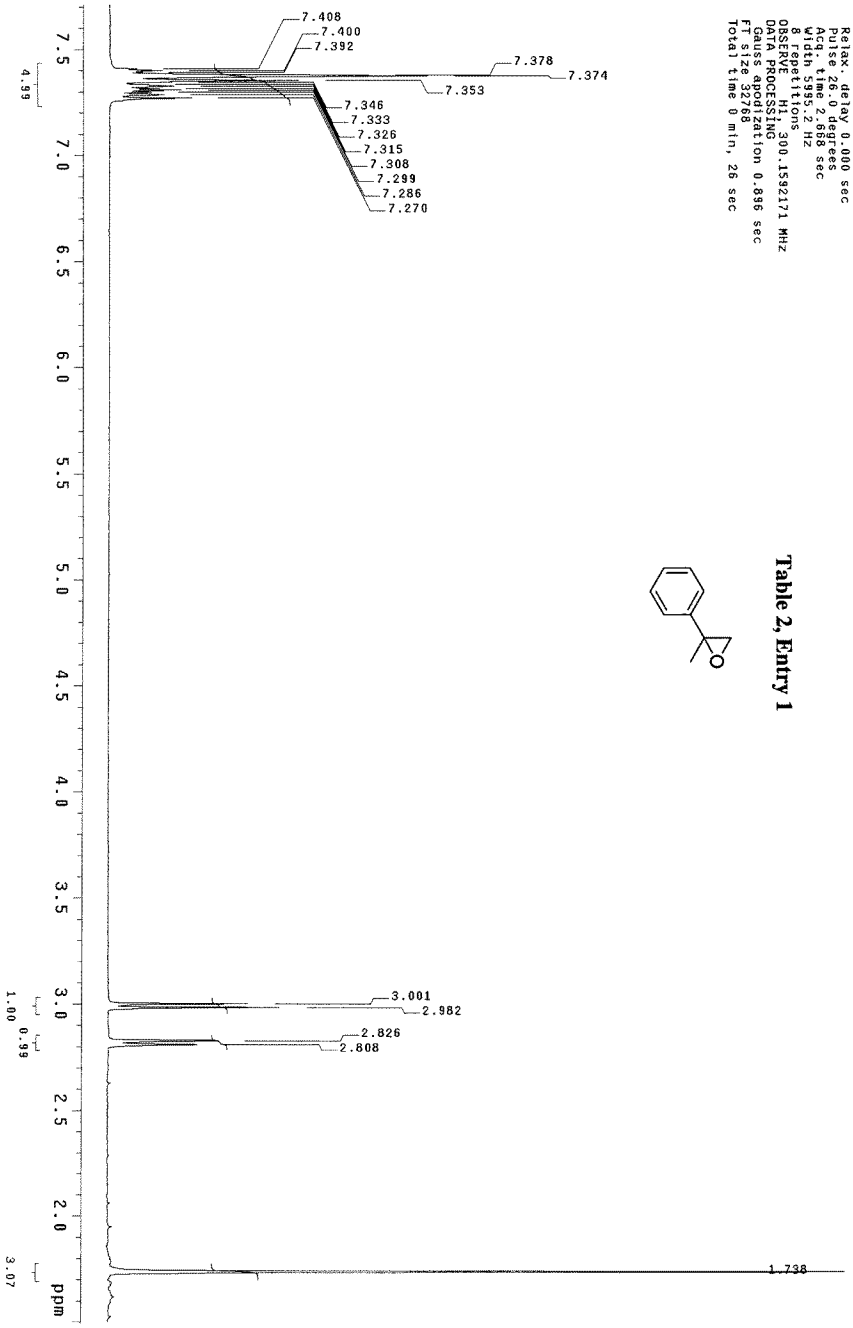


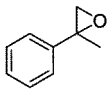
Table 2, Entry 1

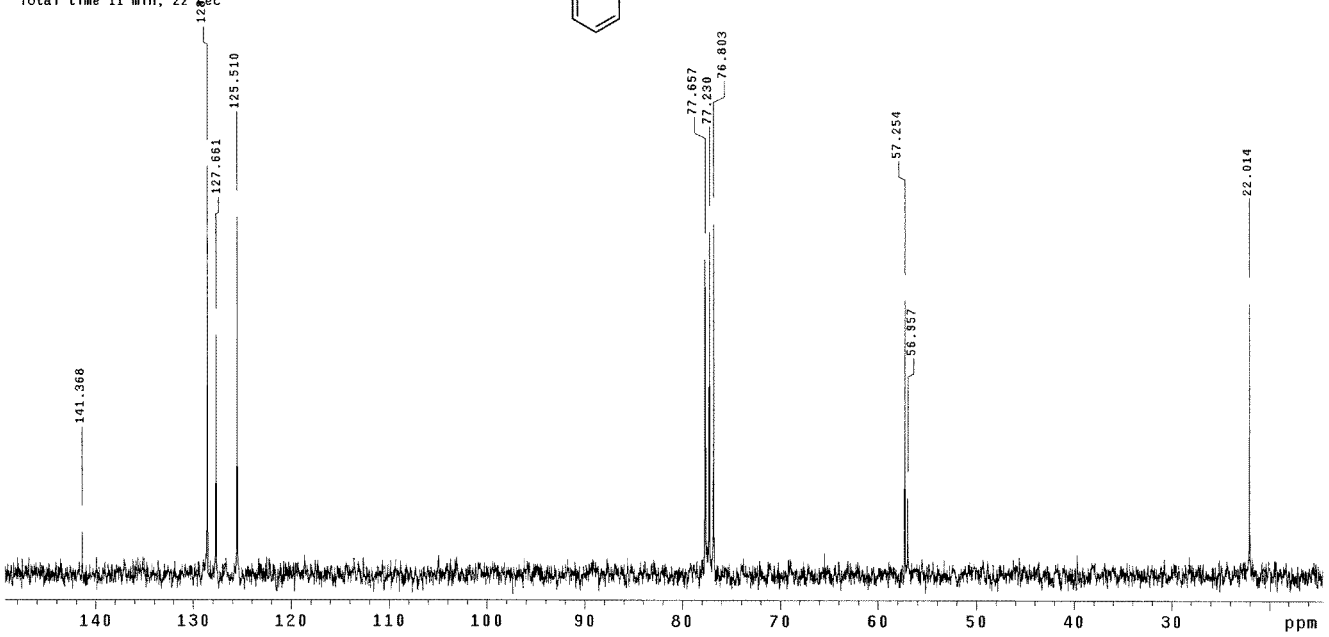
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-17-14-3-13C
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
144 repetitions
OBSERVE C13, 75.4750804 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 11 min, 22 sec

Table 2, Entry 1





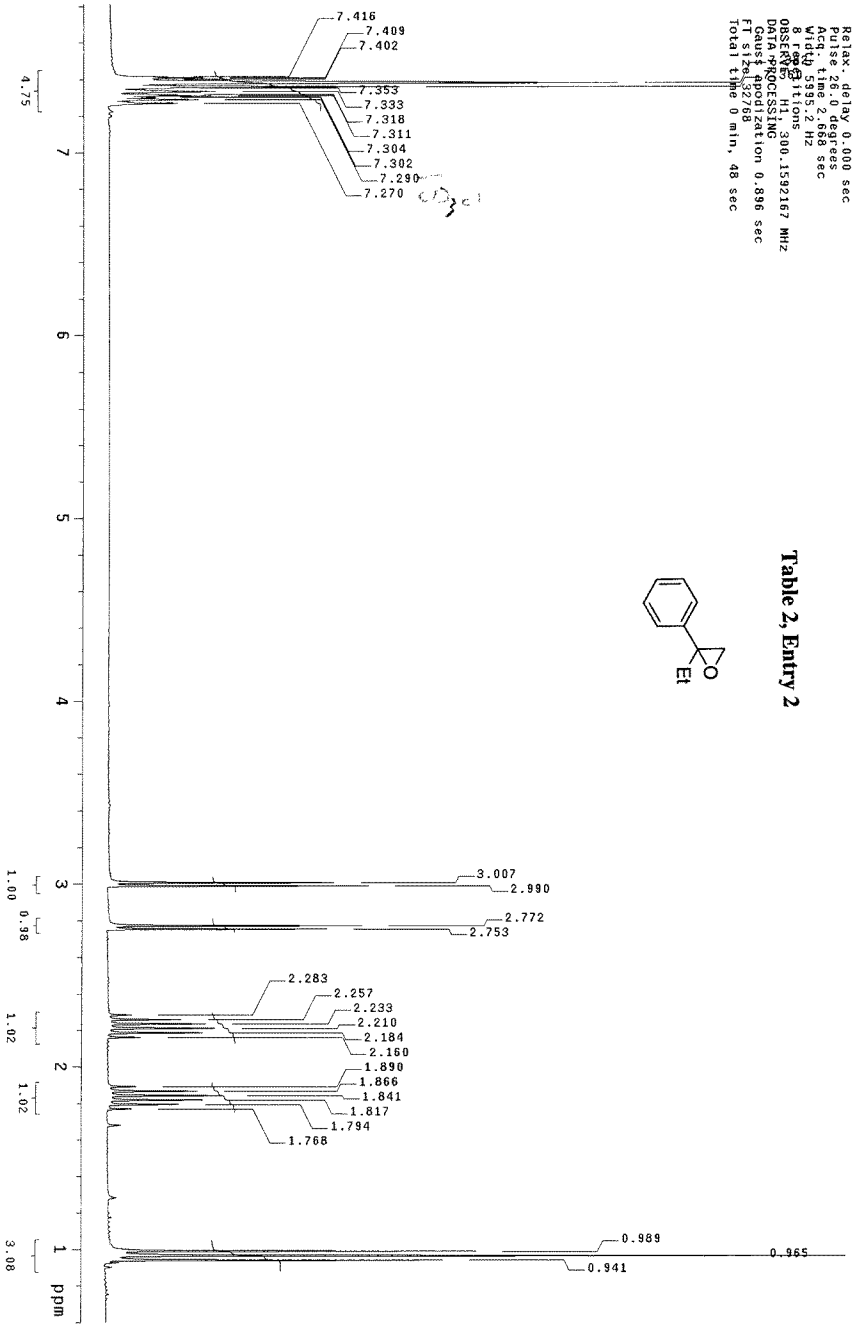


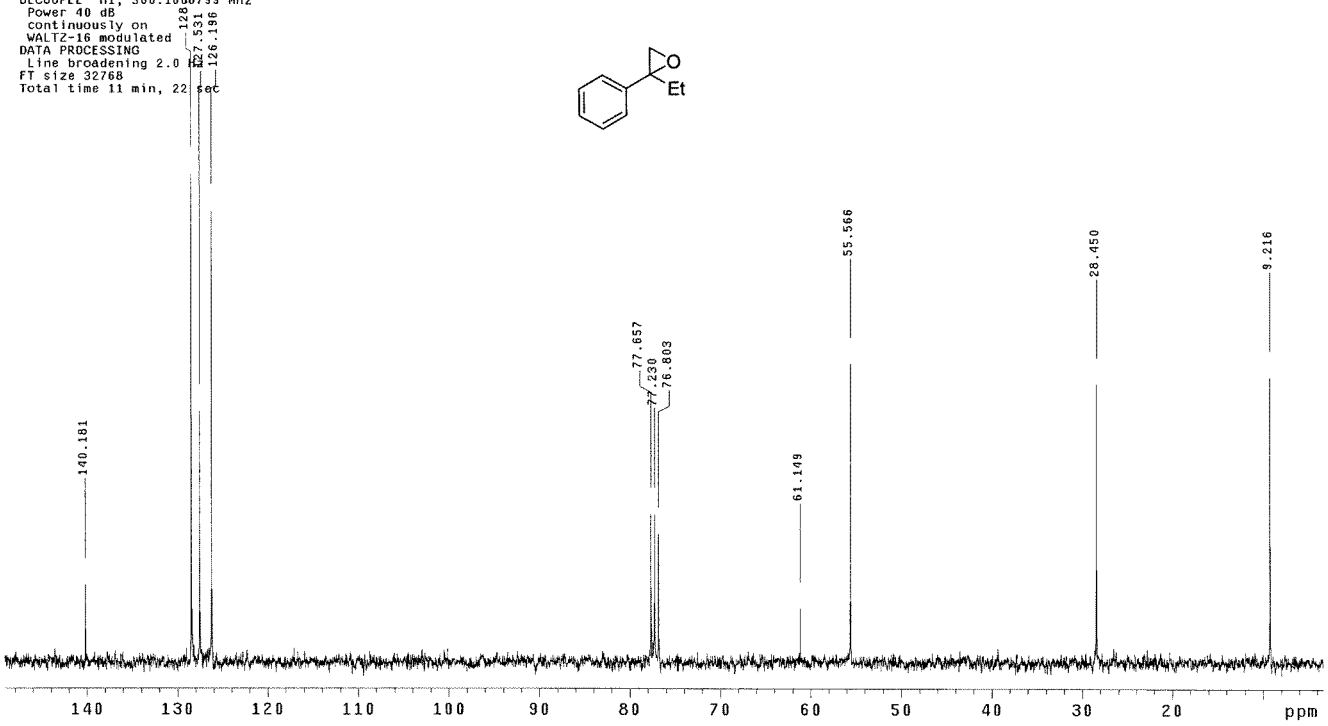
Table 2, Entry 2

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-18-39-1-13C-2
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
40 repetitions
OBSERVE C13, 75.4750832 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 11 min, 22 sec

Table 2, Entry 2

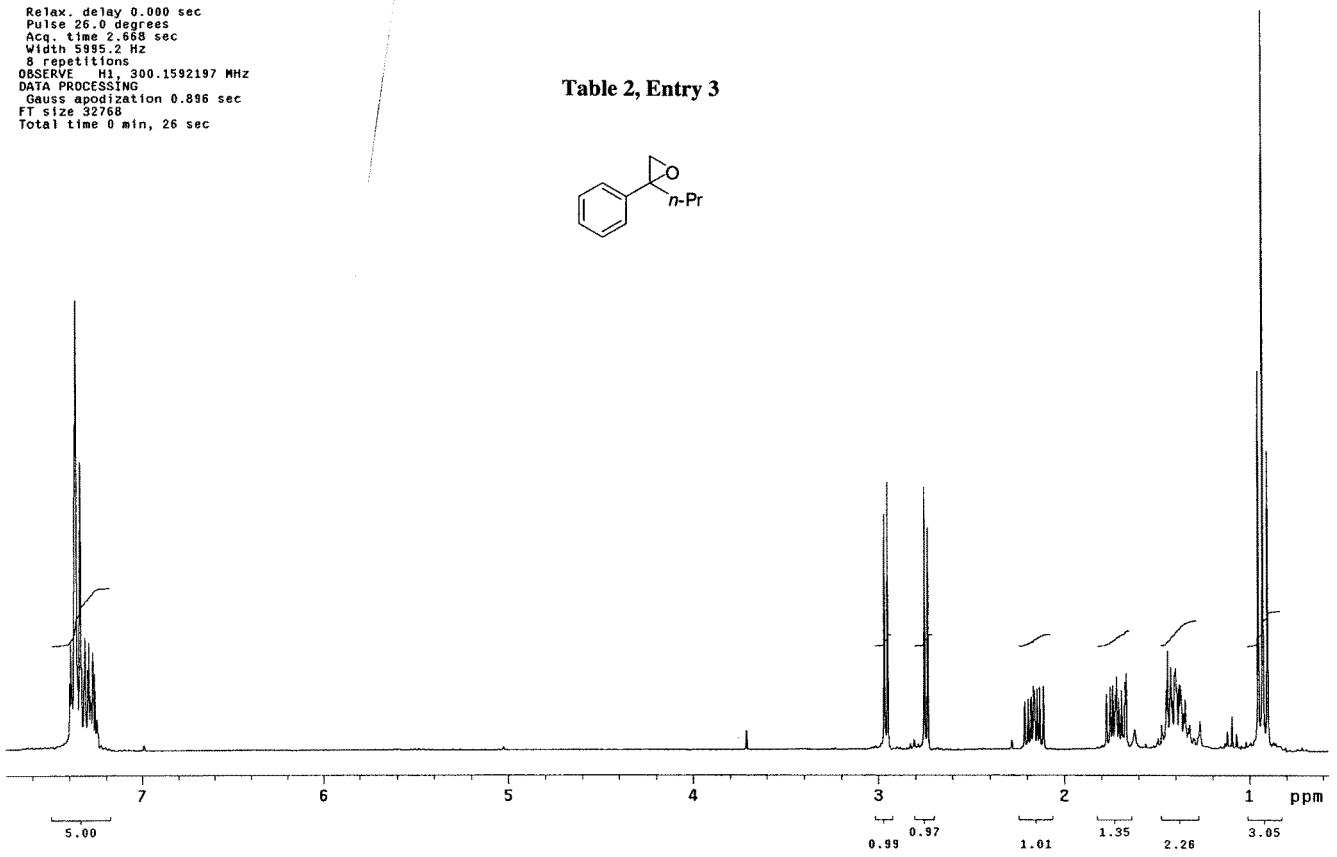


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-18-15-1H
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 300.1592197 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 26 sec

Table 2, Entry 3

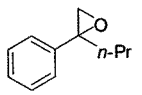


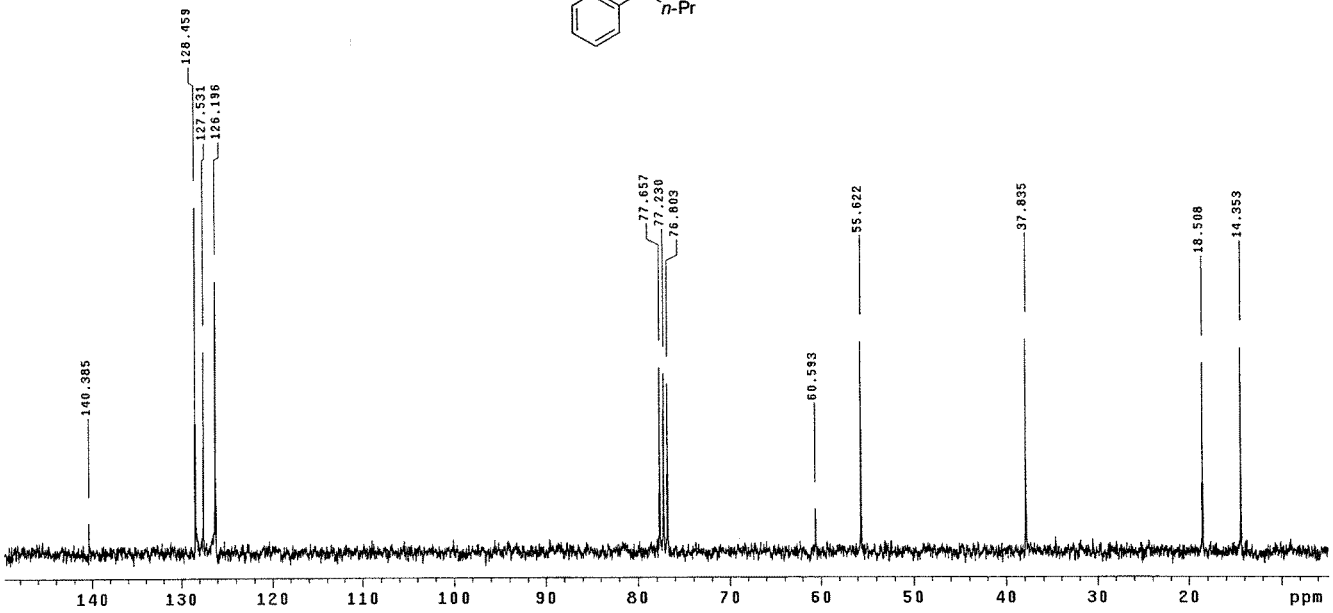
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-18-15-13C-1
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
88 repetitions
OBSERVE C13, 75.4750804 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 11 min, 22 sec

Table 2, Entry 3



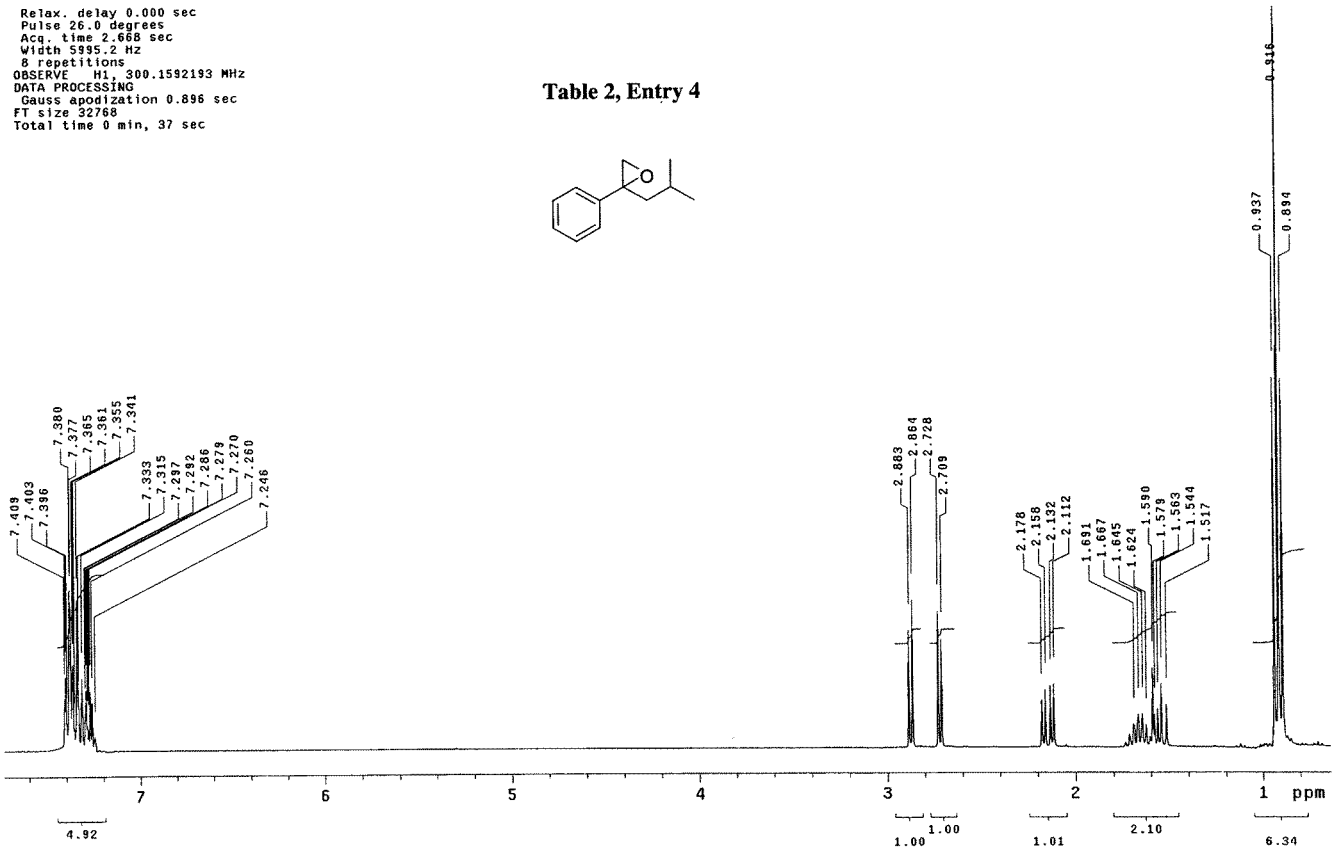


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-18-13-1H-1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
8 repetitions
OBSERVE H1, 300.1592193 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 37 sec

Table 2, Entry 4

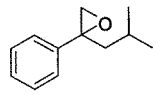


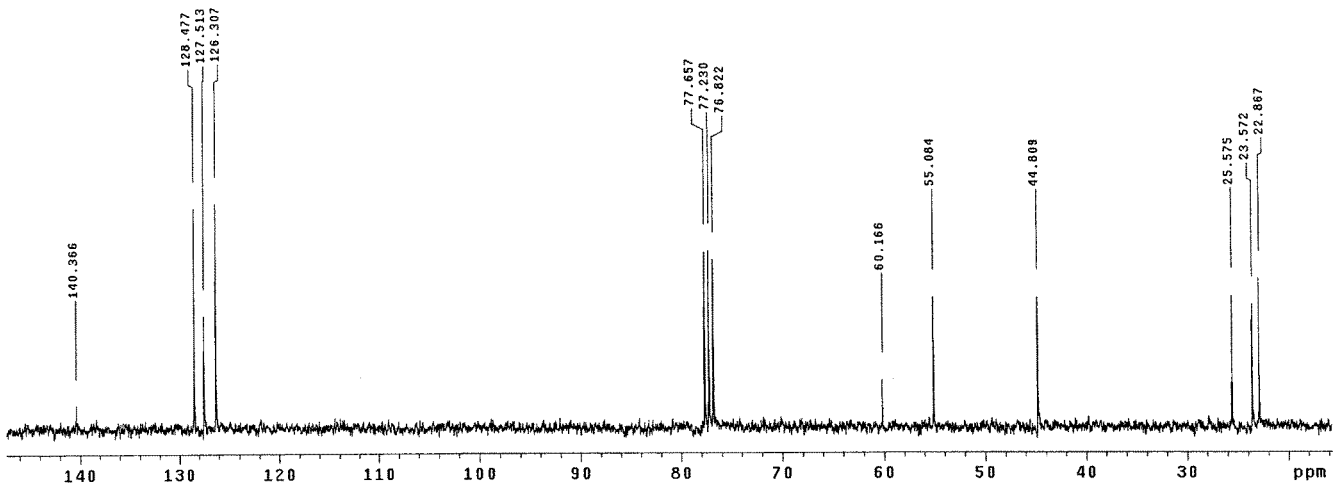
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-18-18-13C
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
176 repetitions
OBSERVE C13, 75.4750780 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 22 min, 44 sec

Table 2, Entry 4





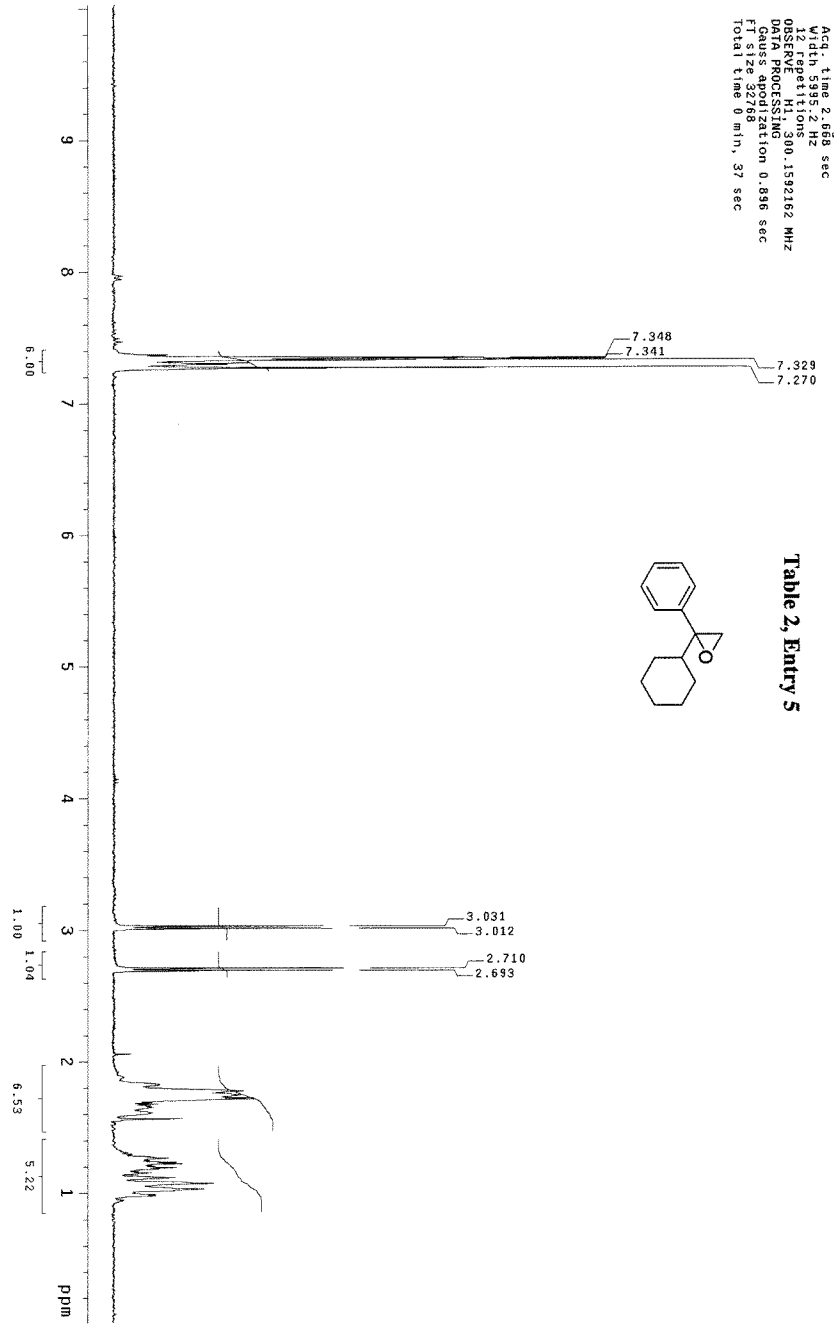


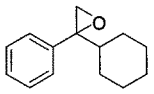
Table 2, Entry 5

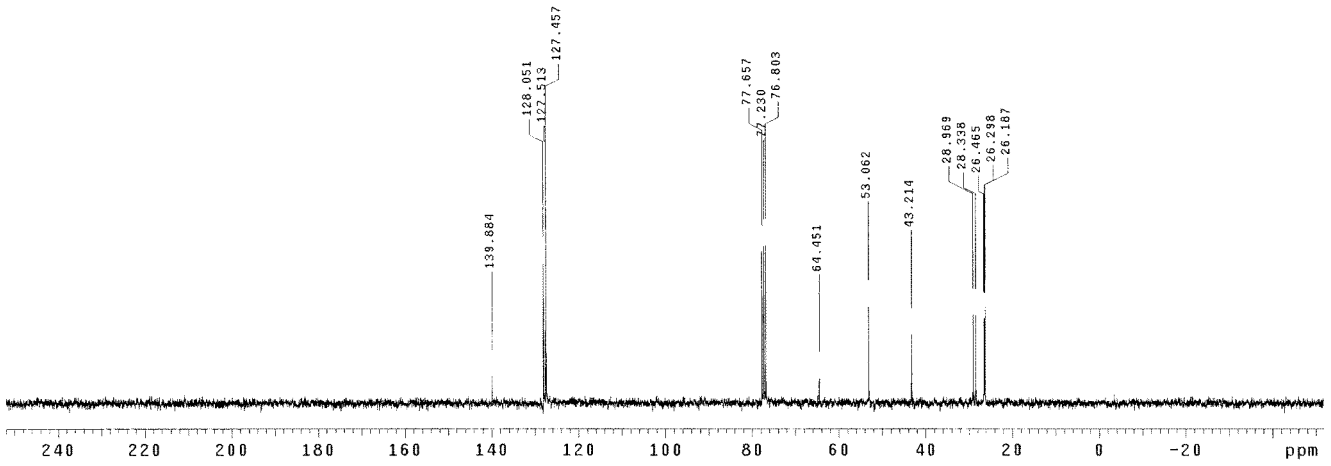
13C OBSERVE

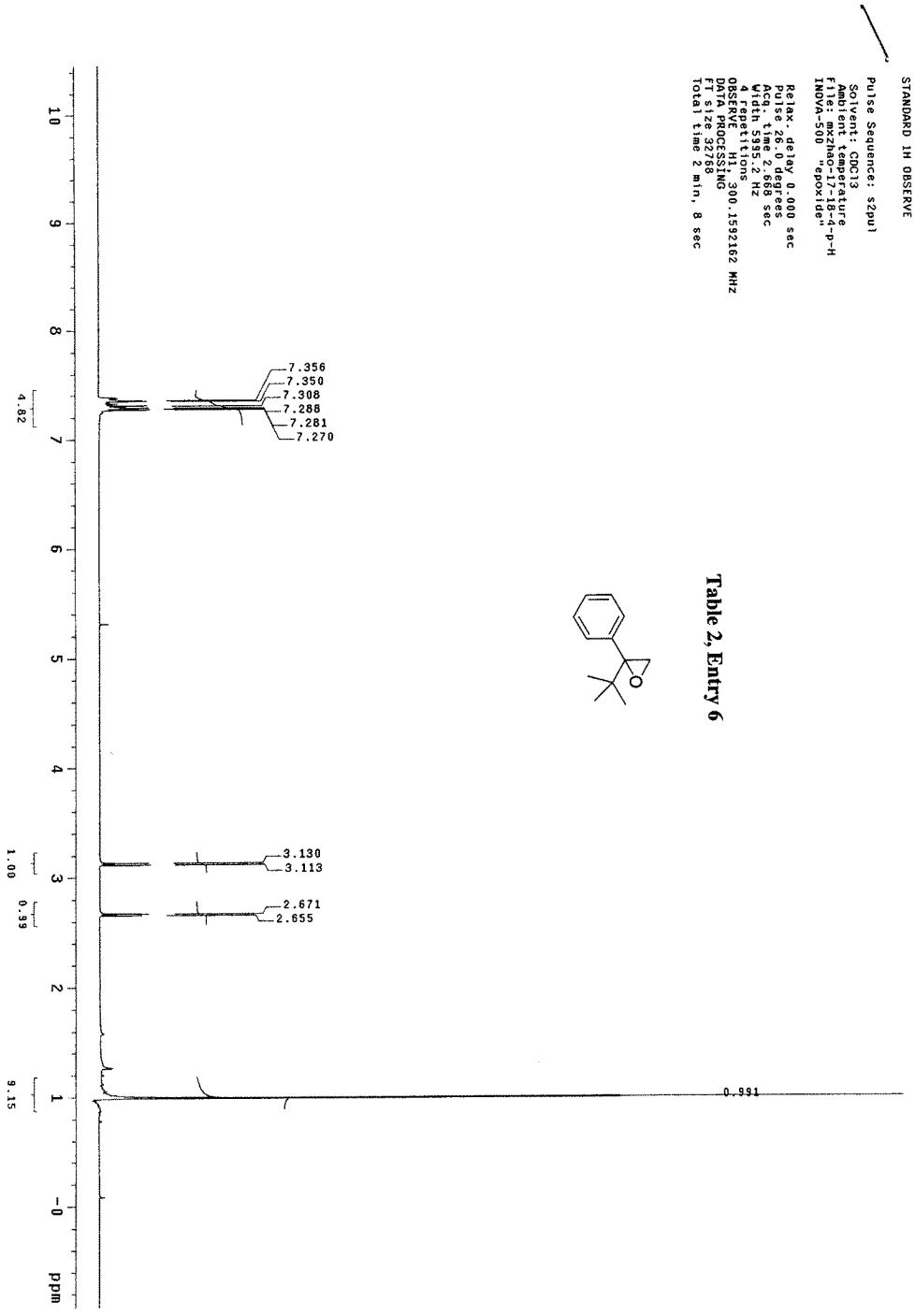
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1519a-p-C
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
168 repetitions
OBSERVE C13, 75.4750804 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 473916 hr, 13 min, 52 sec

Table 2, Entry 5





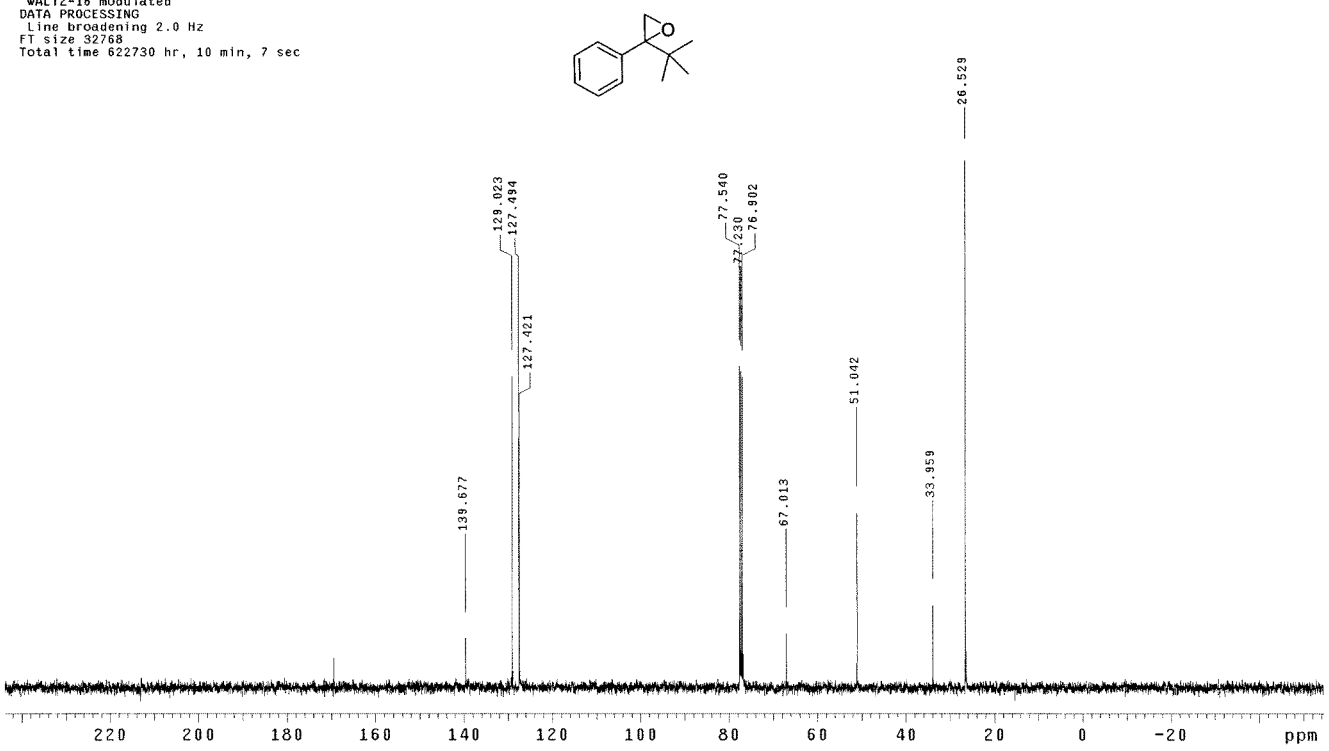

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-17-18-4-p-H
INOVA-500 "epoxide"
Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.000 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592162 MHz
DATA PROCESSING
FT size 32768
Total time 2 min, 8 sec
Table 2, Entry 6
7.356
7.350
7.308
7.288
7.281
7.270
3.130
3.113
2.671
2.655
0.991
4.82
1.00
0.99
9.15
ppm

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: oaw-08-34-C
INOVA-500 "epoxide"

Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
160 repetitions
OBSERVE C13, 100.6067923 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec

Table 2, Entry 6



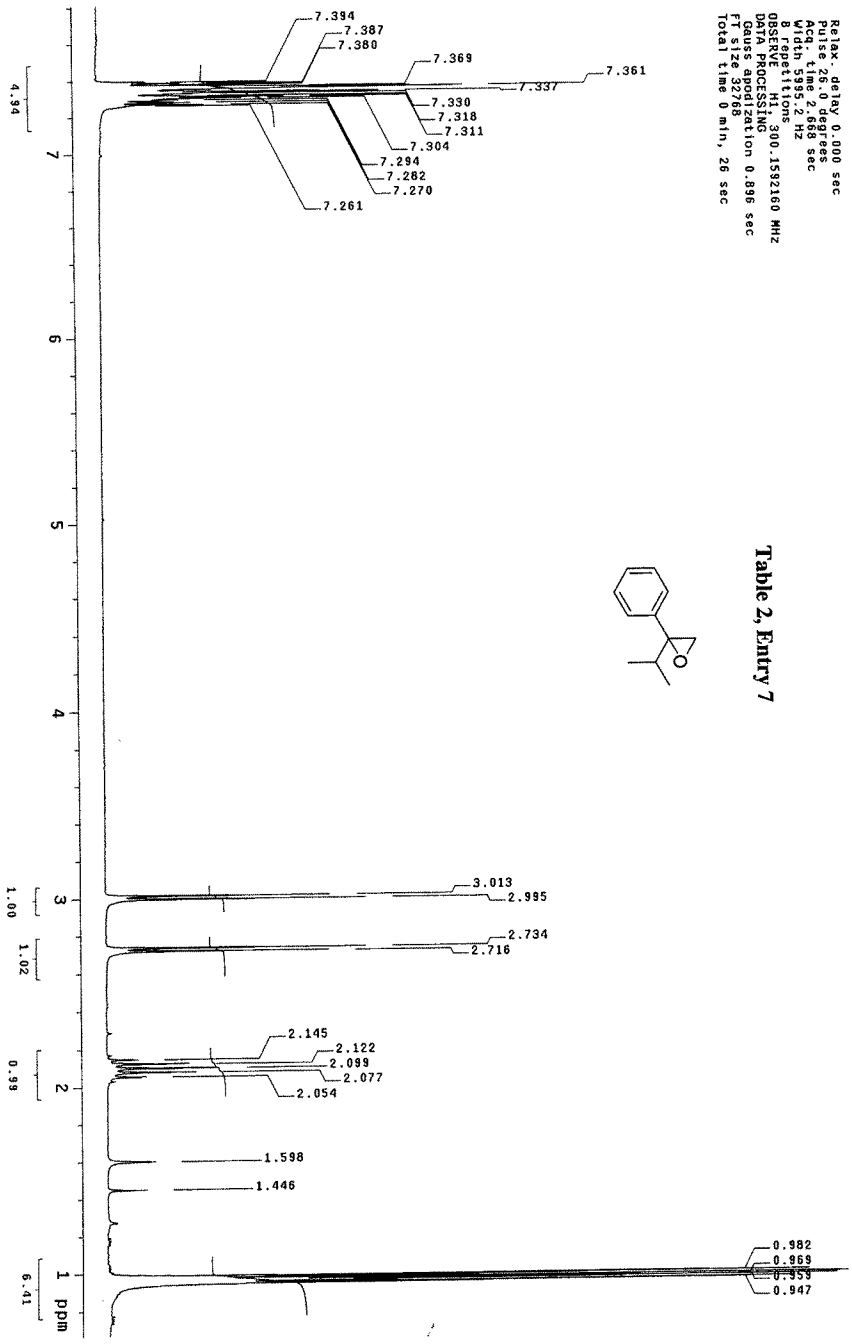


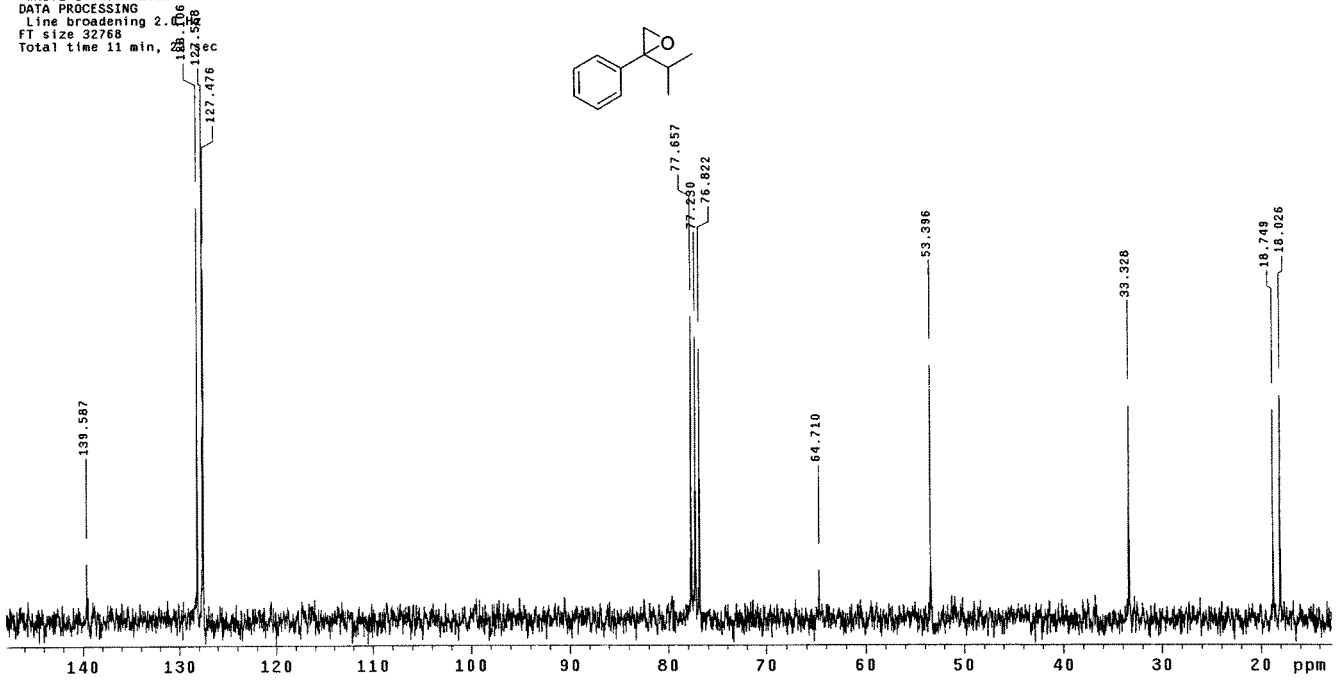
Table 2, Entry 7

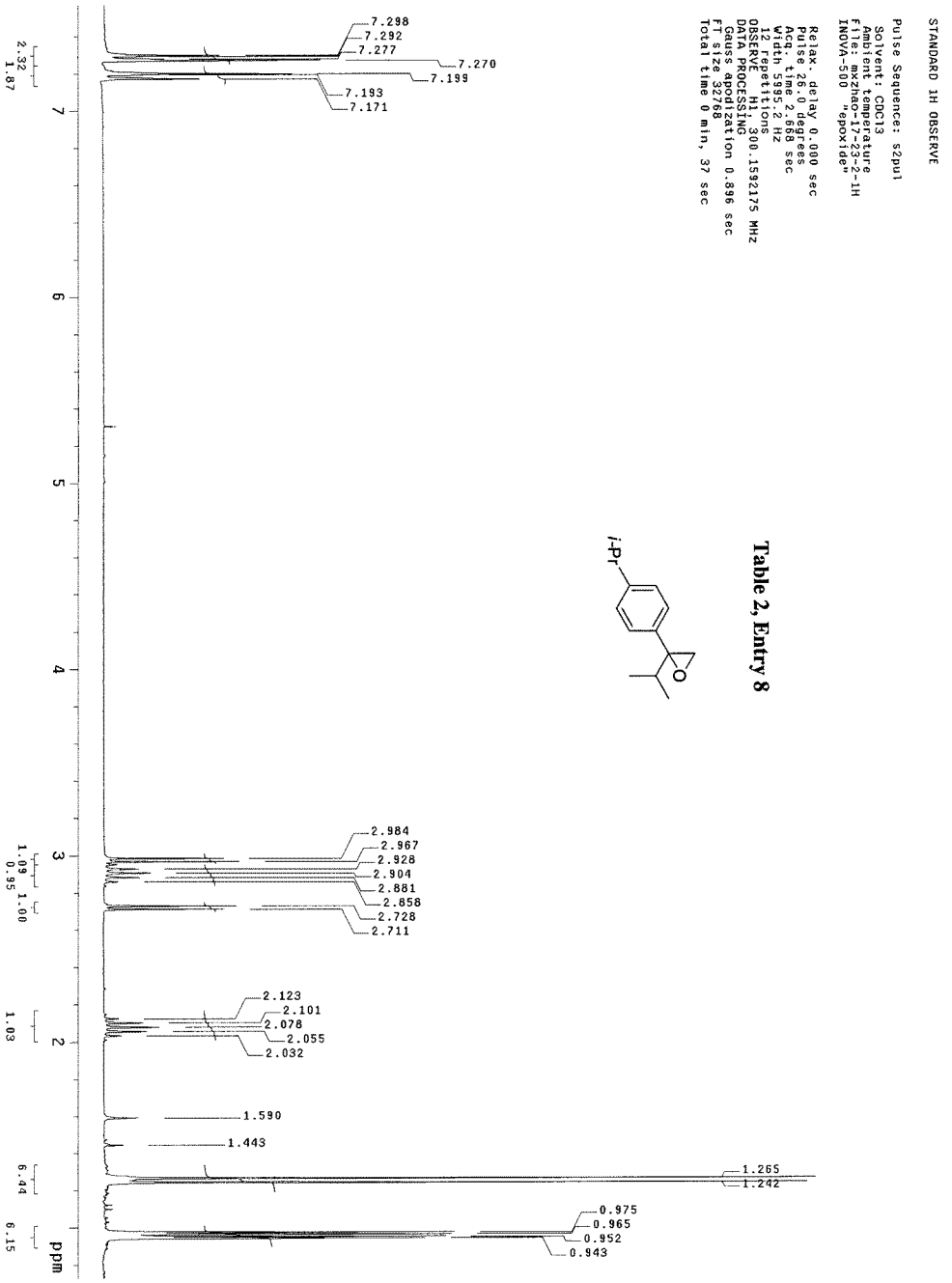
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-17-16-4-13C
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
72 repetitions
OBSERVE C13, 75.4750790 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 11 min, 22 sec

Table 2, Entry 7



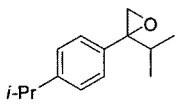

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mx2heo-17-23-r 1H
INOVA-500 "epoxide"
Relax. delay 0.000 sec
Pulse 45.0 degrees
Acq. time 2.999 sec
Width 5995.2 Hz
12 repetitions
OBSERVE H1, 300.1592175 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 37 sec
Table 2, Entry 8
i-Pr
O
7.298
7.292
7.277
7.270
7.199
7.193
7.171
2.984
2.967
2.928
2.904
2.881
2.858
2.728
2.711
2.123
2.101
2.078
2.055
2.032
1.590
1.443
1.265
1.242
0.975
0.965
0.952
0.943
2.32
1.87
1.09
0.95
1.00
1.03
6.44
6.15
ppm

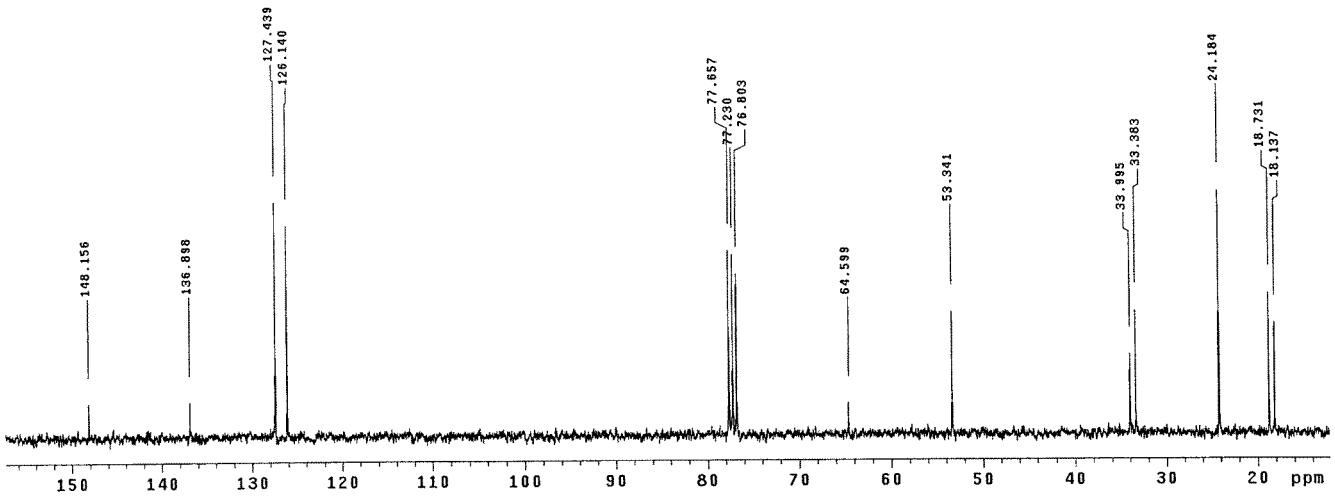
13C OBSERVE

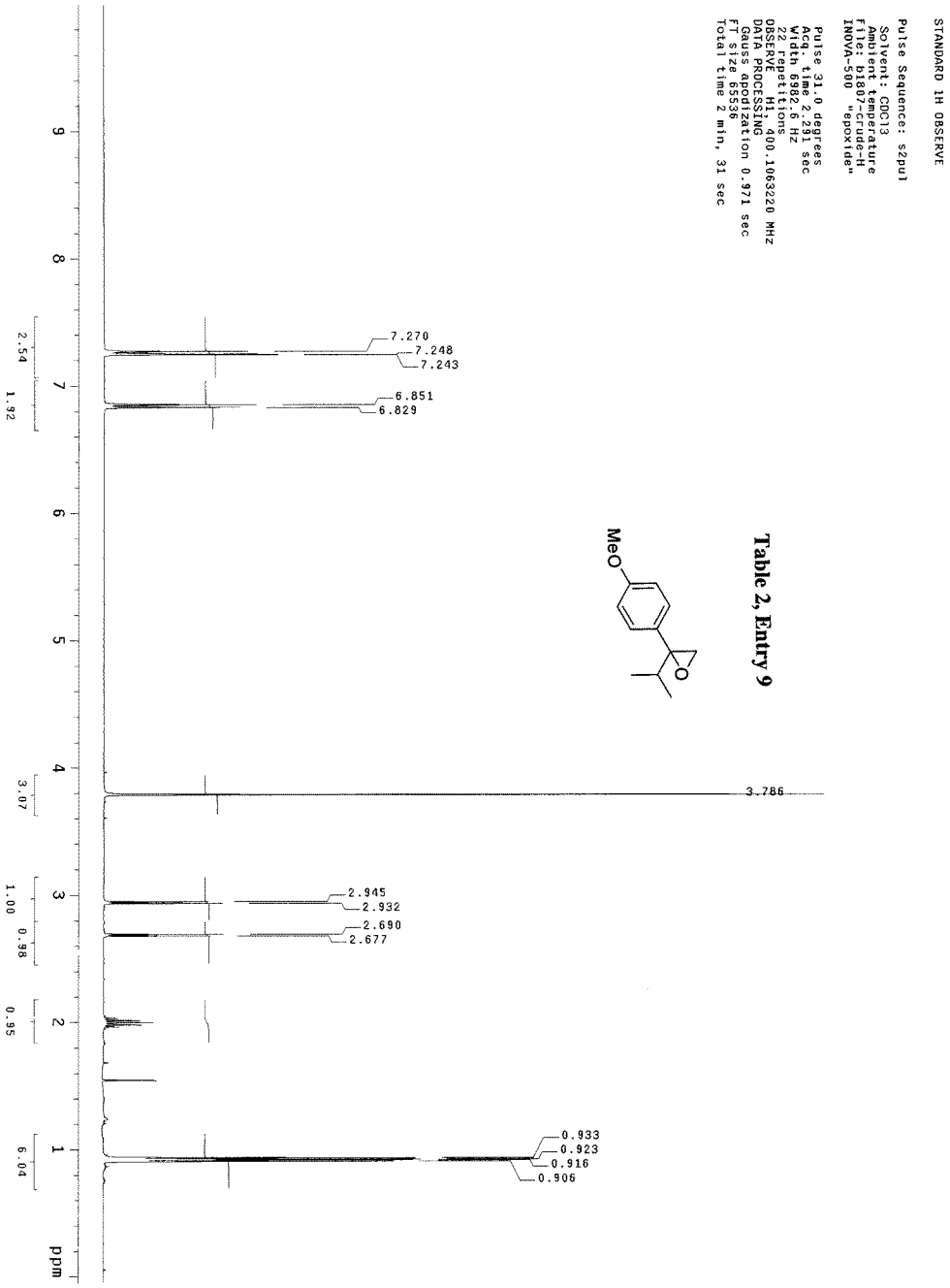
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-17-23-2-13C
INOVA-500 "epoxide"

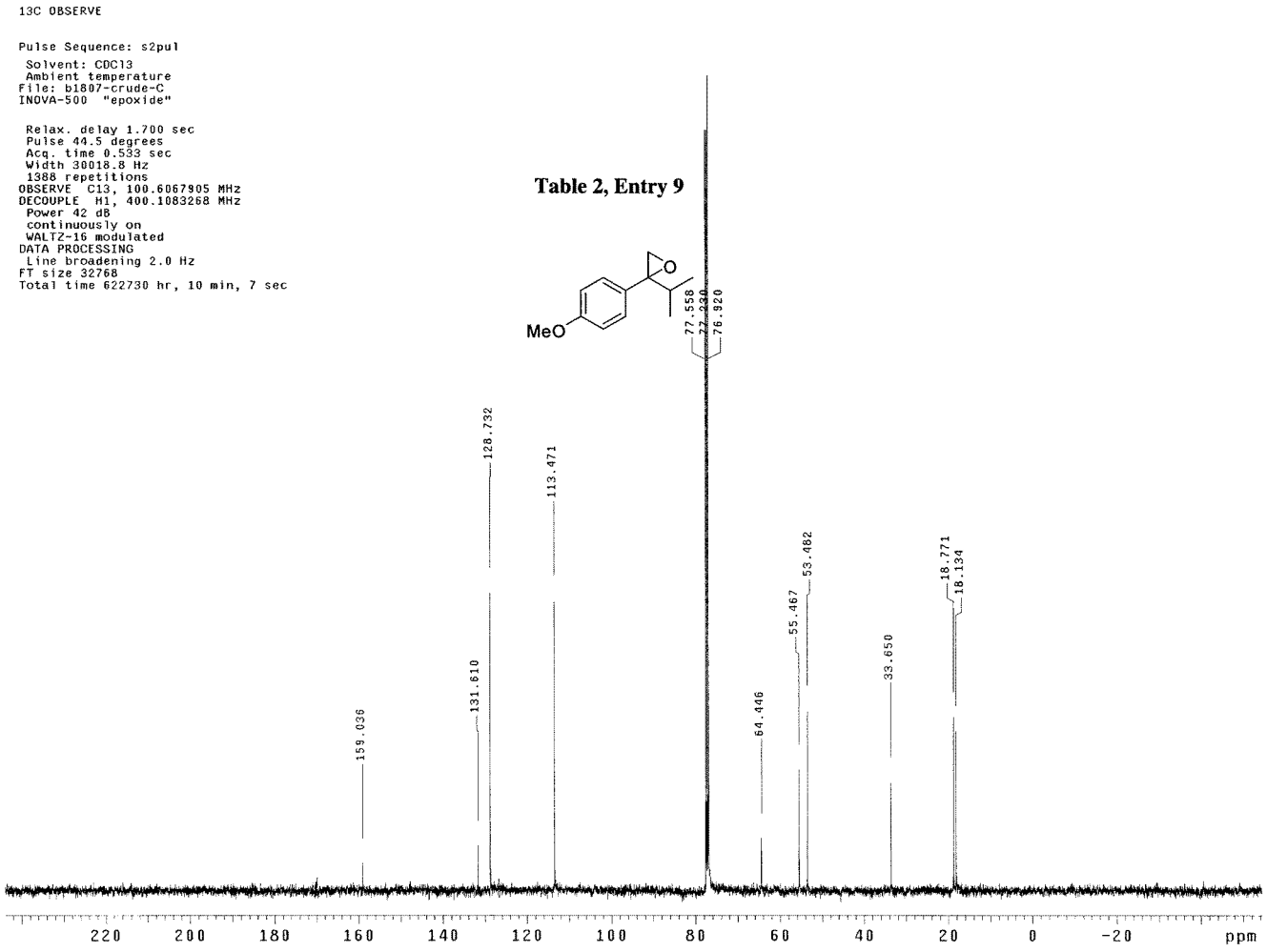
Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
192 repetitions
OBSERVE C13, 75.4750790 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 11 min, 22 sec

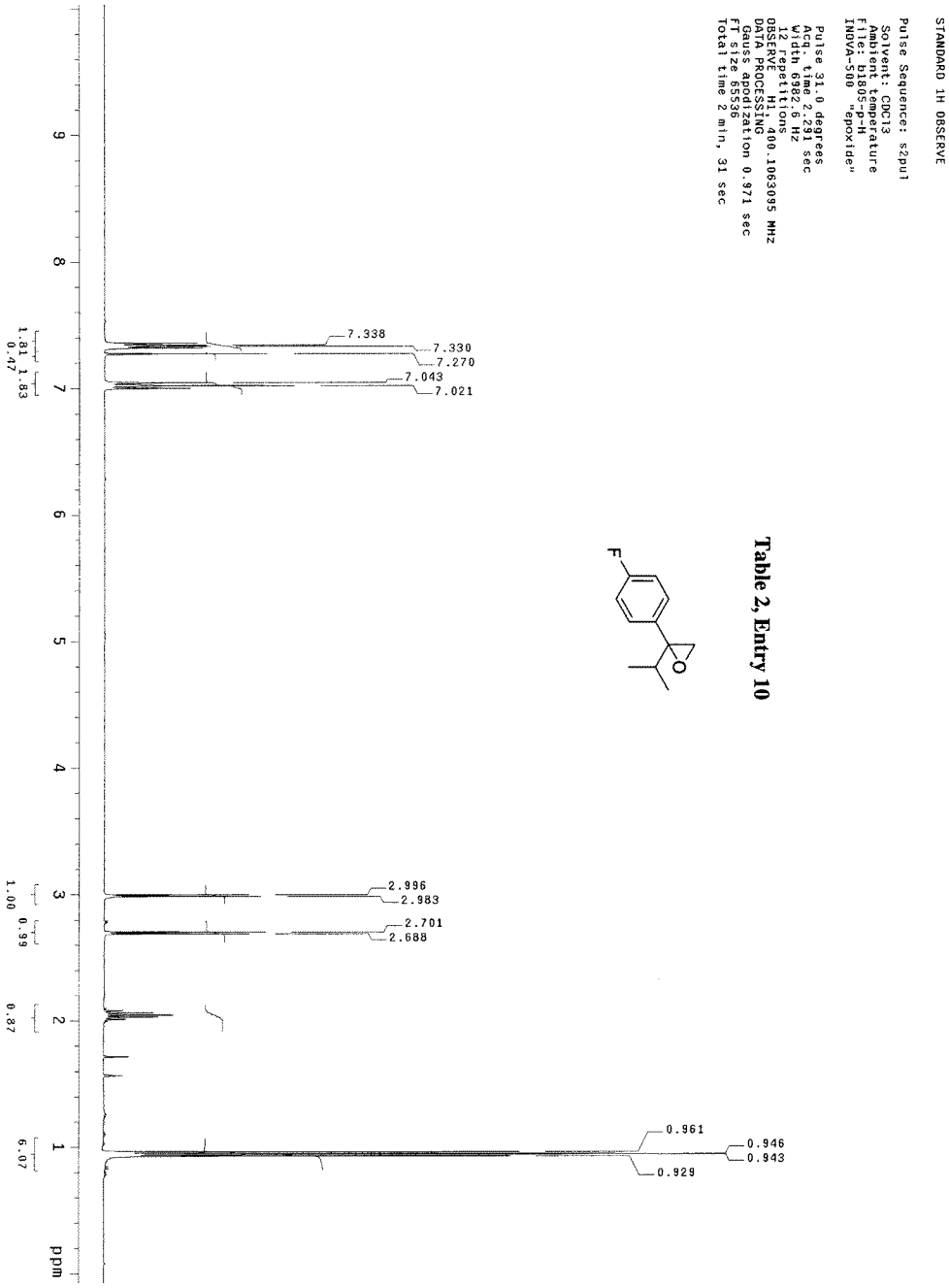
Table 2, Entry 8






STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1807-crude-H
INOVA-500 "epoxide"
Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
22 repetitions
OBSERVE H1, 400.1063220 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 2 min, 31 sec
Table 2, Entry 9
MeO
7.270
7.248
7.243
6.851
6.829
3.786
2.945
2.932
2.690
2.677
0.933
0.923
0.916
0.906
2.54
1.92
3.07
1.00
0.98
0.95
6.04
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1807-crude-C
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
1388 repetitions
OBSERVE C13, 100.6067905 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec
Table 2, Entry 9
MeO
O
77.558
77.230
76.920
159.036
131.610
128.732
113.471
64.446
55.467
53.482
33.650
18.771
18.134
220
200
180
160
140
120
100
80
60
40
20
0
-20
ppm

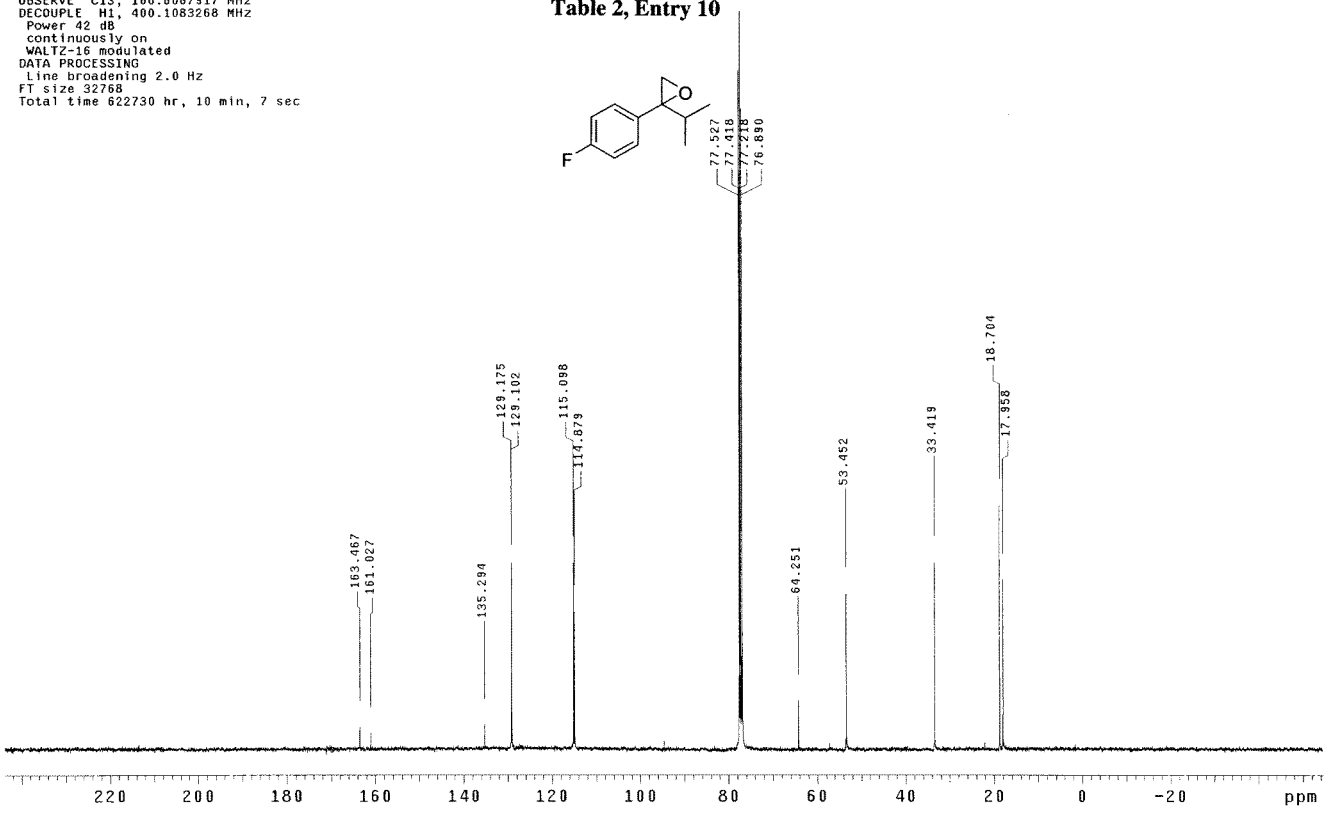

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1805-p-H
INOVA-500 "epoxide"
Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
12 repetitions
OBSERVE H1, 400.1083095 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 2 min, 31 sec
F
O
Table 2, Entry 10
7.338
7.330
7.270
7.043
7.021
2.996
2.983
2.701
2.688
0.961
0.946
0.943
0.929
1.81
1.83
0.47
1.00
0.99
0.87
6.07
ppm

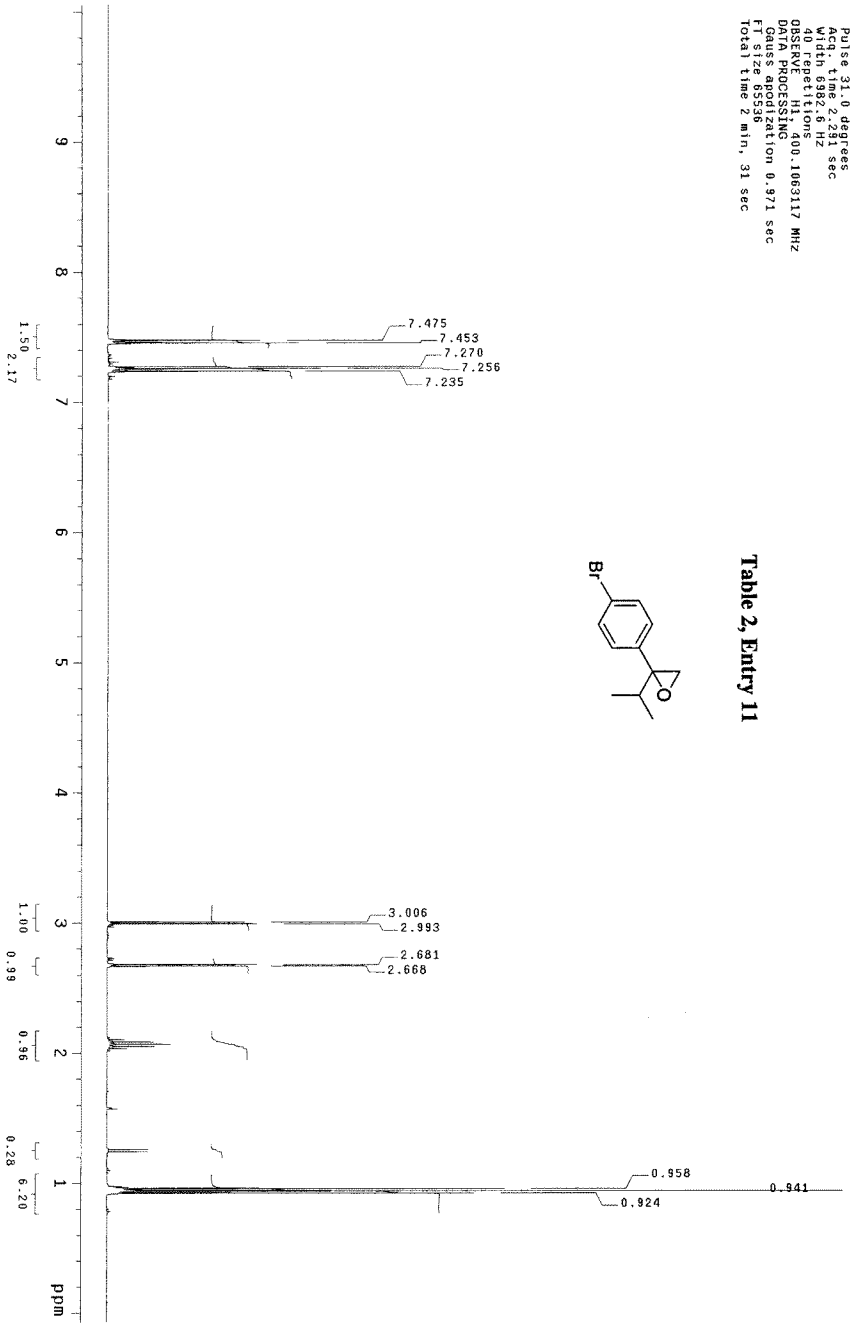
b1805-p-H

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1805-p-C
INOVA-500 "epoxide"

Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
9604 repetitions
OBSERVE C13, 100.6067917 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec

Table 2, Entry 10




STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1812-H
INOVA-500 "epoxide"
Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
40 repetitions
OBSERVE H1, 400.1063117 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 2 min, 31 sec
Br
O
Table 2, Entry 11
7.475
7.453
7.270
7.256
7.235
3.006
2.993
2.681
2.668
0.958
0.941
0.924
1.50
2.17
1.00
0.99
0.96
0.28
6.20
ppm

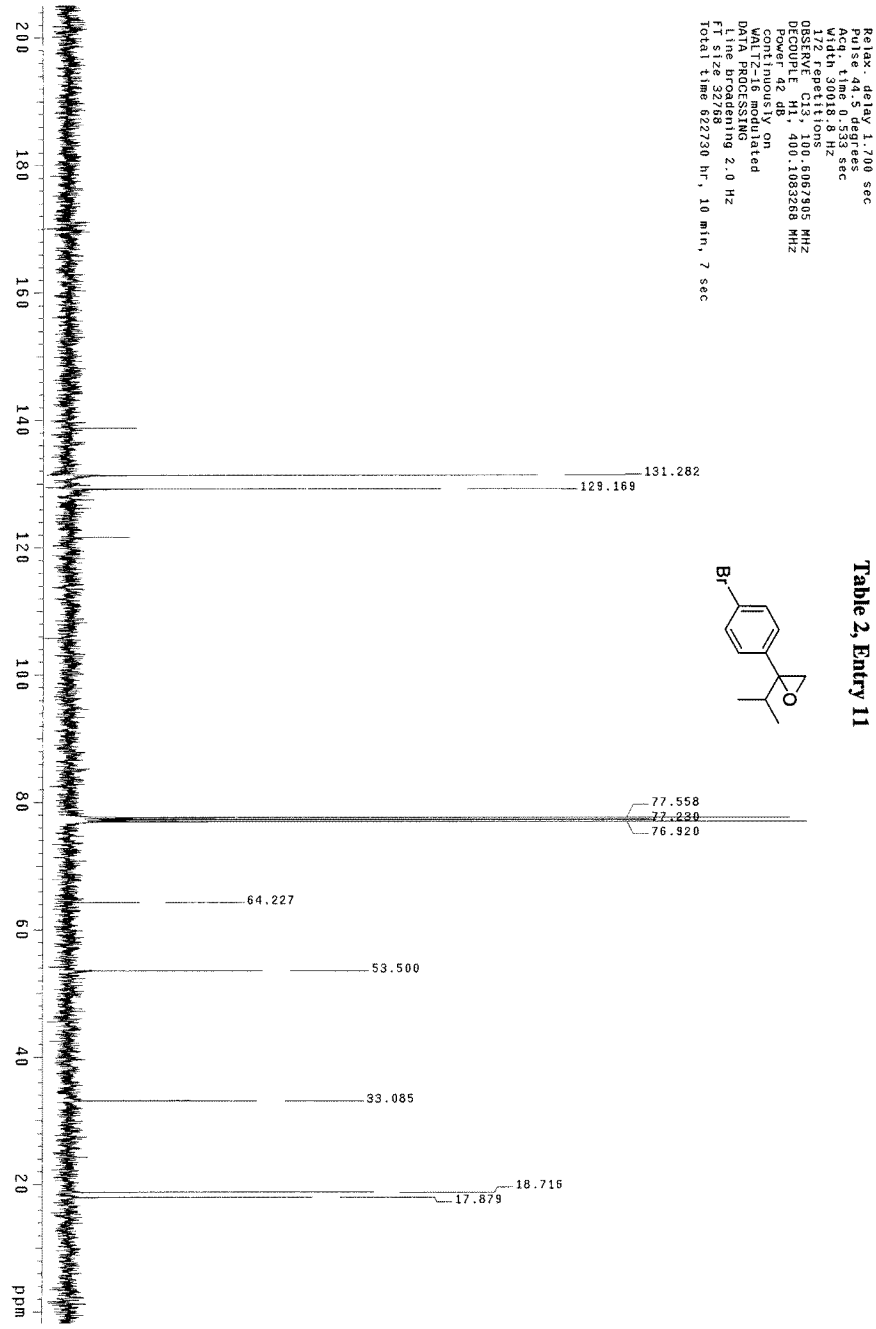
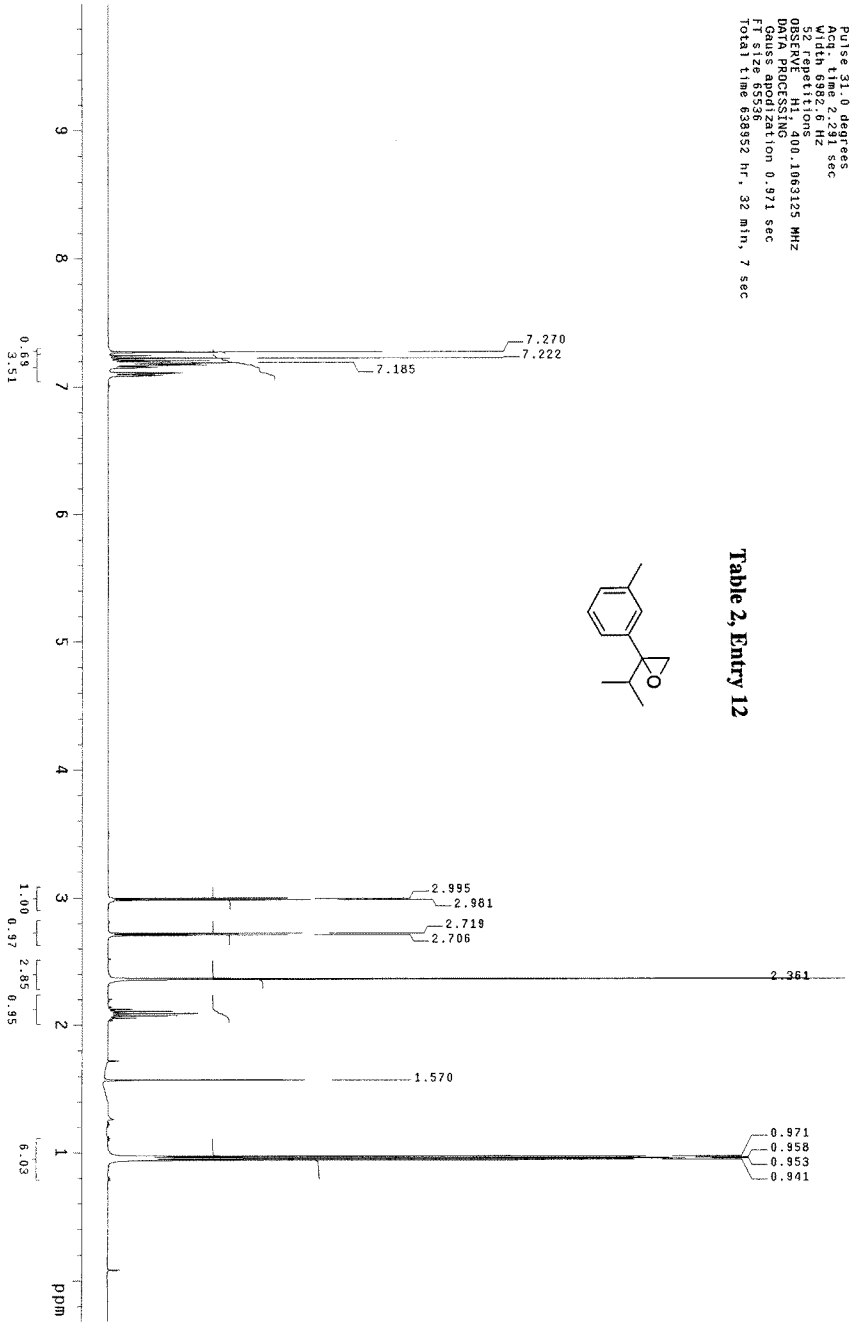


Table 2, Entry 11


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1742-H
INOVA-500 "epoxide"
Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
52 repetitions
OBSERVE H1, 400.1063125 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 638952 hr, 32 min, 7 sec
Table 2, Entry 12
7.270
7.222
7.185
2.995
2.981
2.719
2.706
2.361
1.570
0.971
0.958
0.953
0.941
0.69
3.51
1.00
0.97
2.85
0.95
6.03
ppm

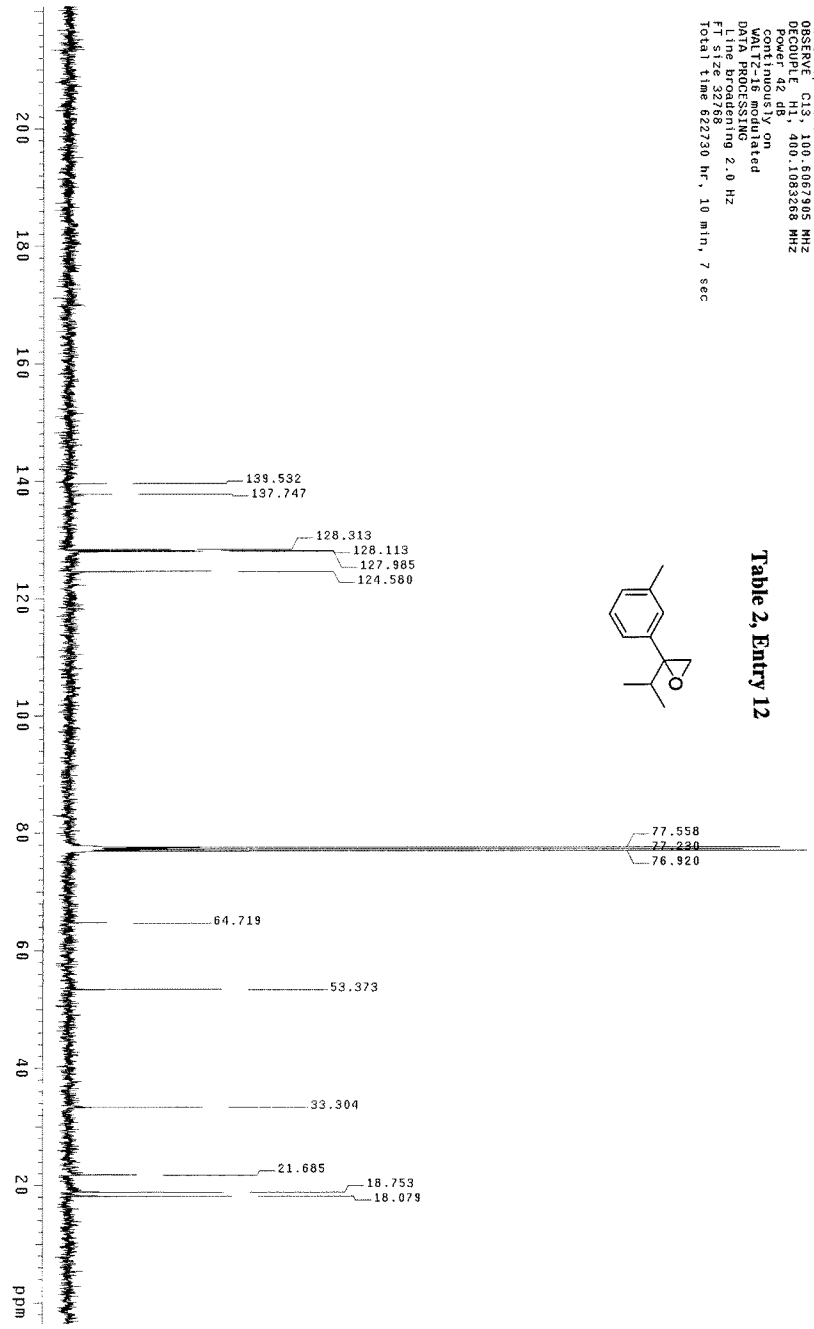


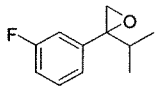
Table 2, Entry 12

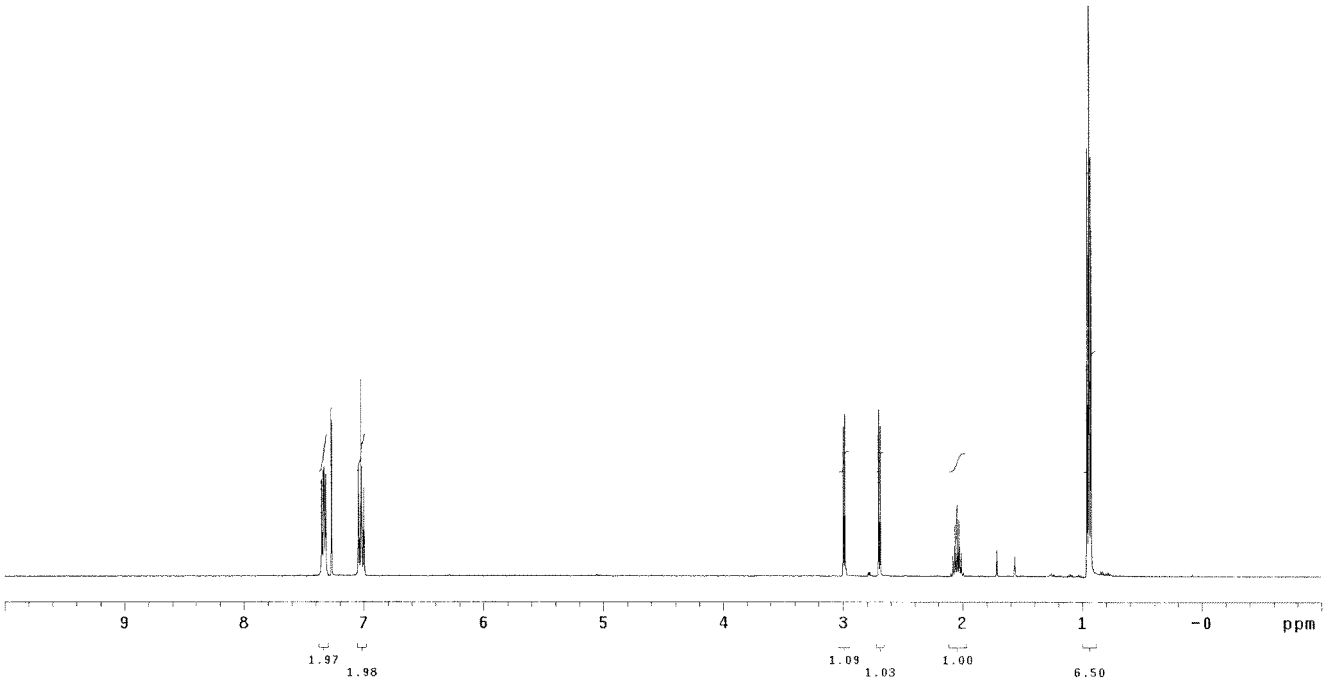
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
File: b1805-p-H
INOVA-500 "epoxide"

 Pulse 31.0 degrees
 Acq. time 2.291 sec
 Width 6982.6 Hz
 12 repetitions
OBSERVE   H1, 400.1063096 MHz
DATA PROCESSING
 Gauss apodization 0.971 sec
FT size 65536
Total time 2 min, 31 sec

Table 2, Entry 13



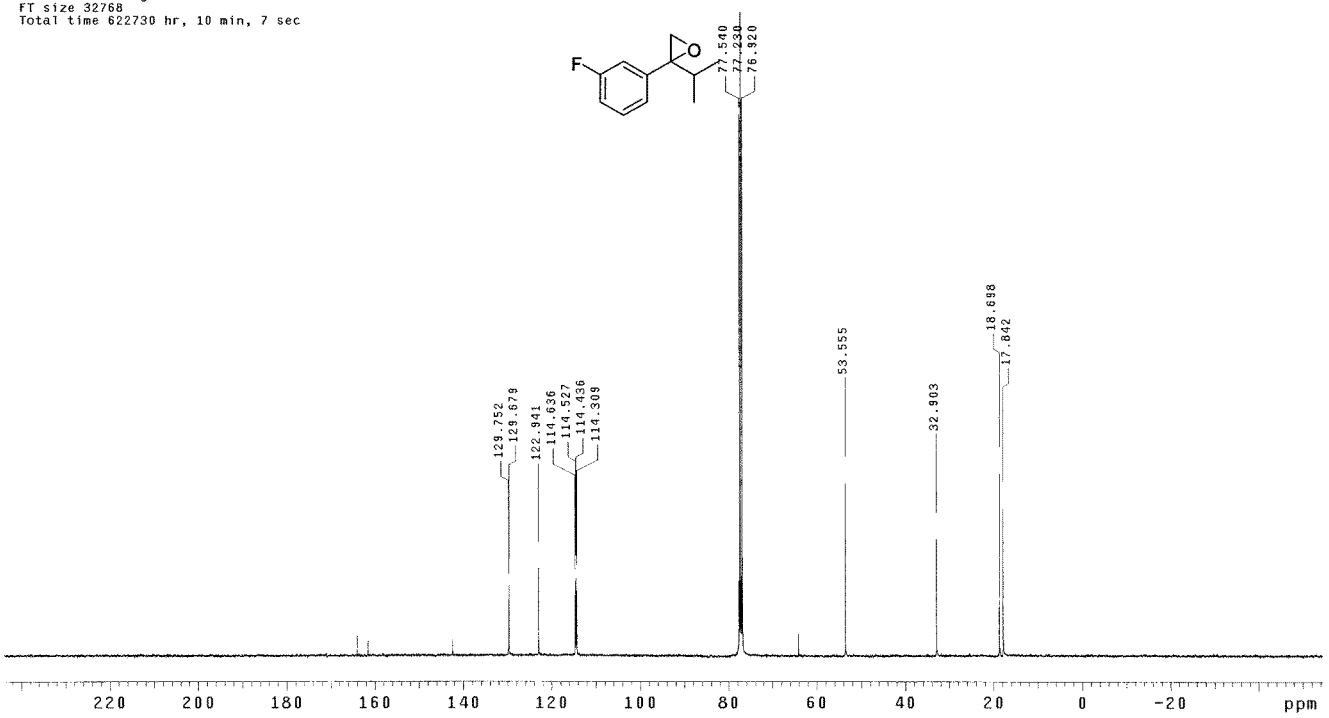


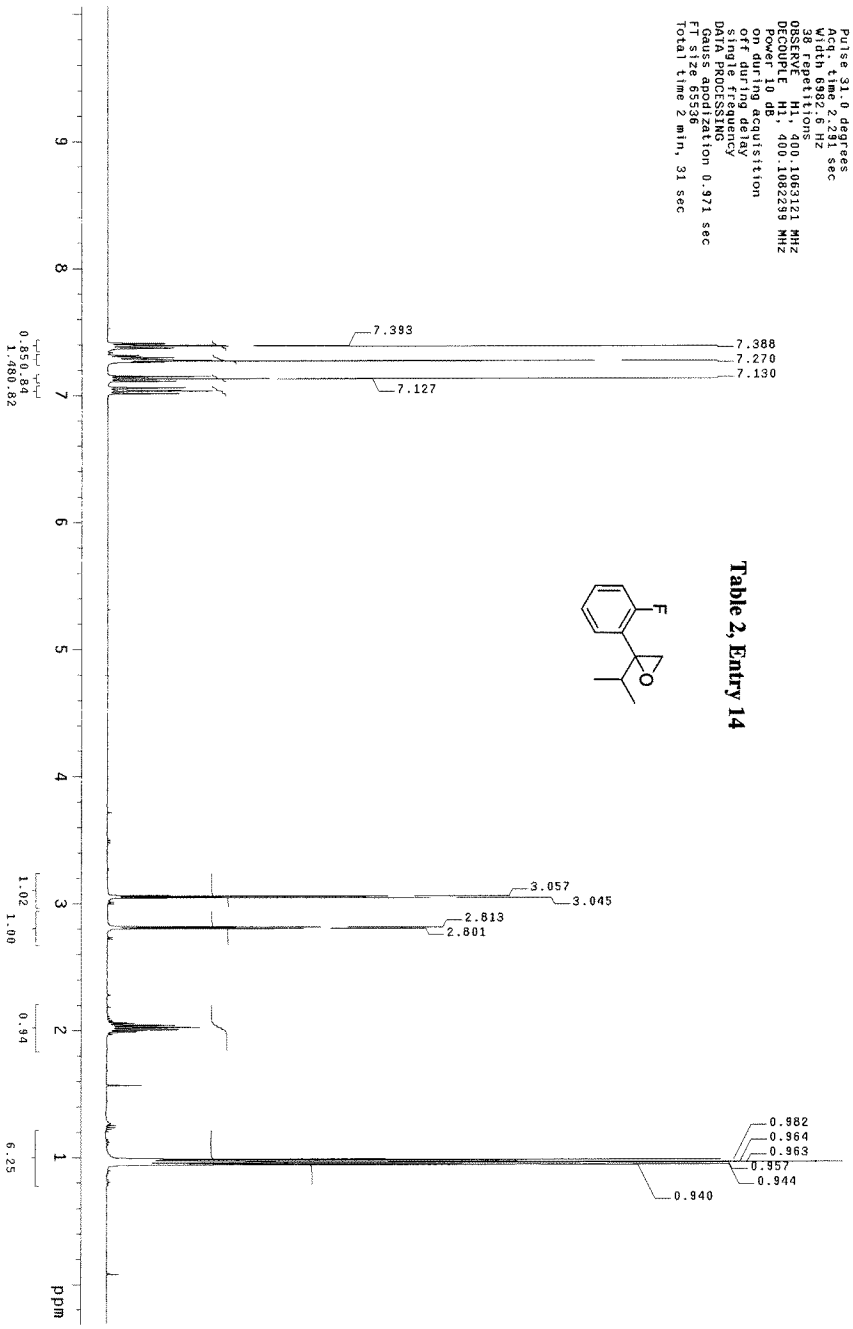
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1825-p-C
INOVA-500 "epoxide"

Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
15216 repetitions
OBSERVE C13, 100.6067905 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec

Table 2, Entry 13



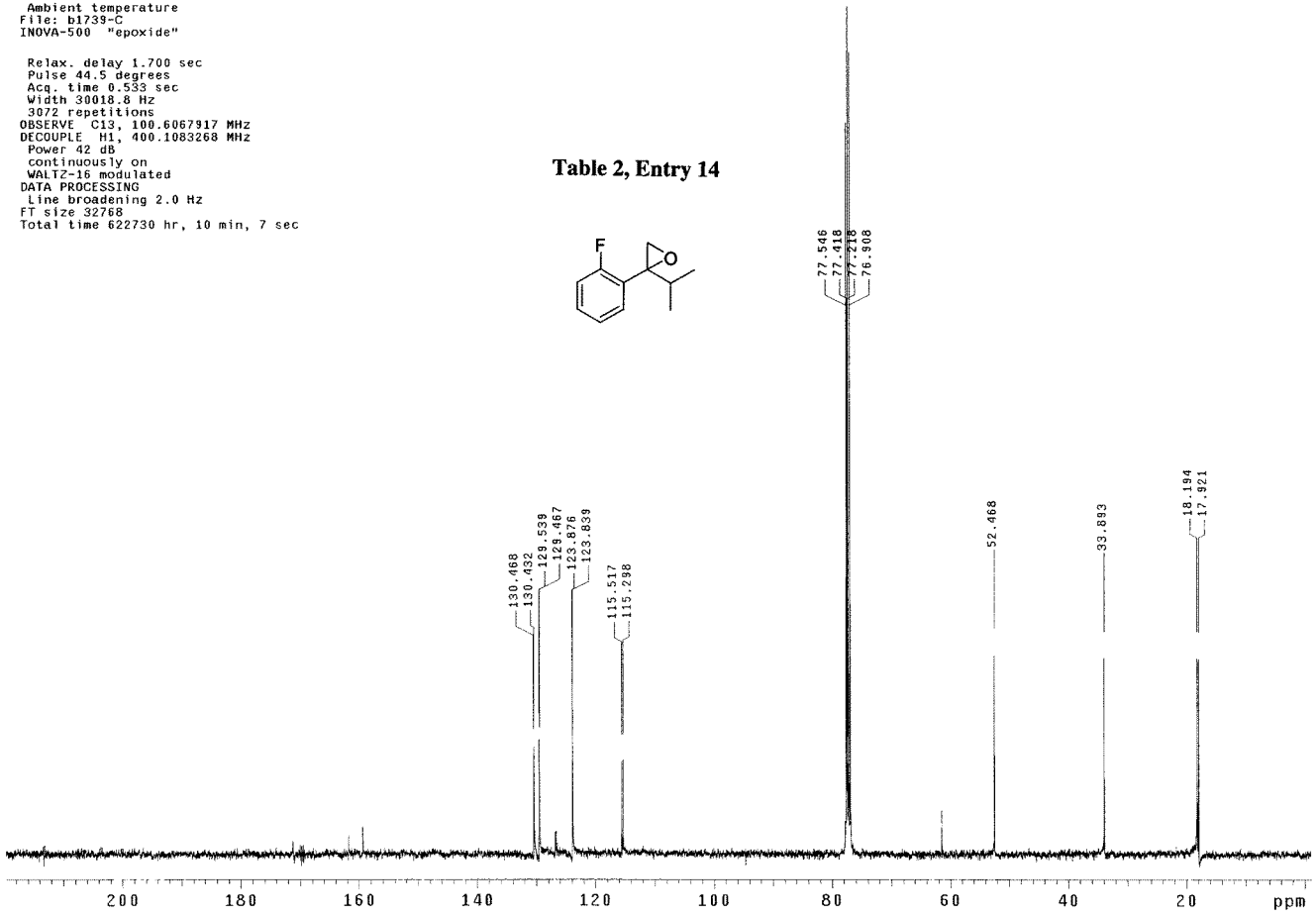

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1739-H
INOVA-500 "epoxide"
Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
38 repetitions
OBSERVE H1, 400.1063121 MHz
DECOUPLE H1, 400.1082239 MHz
Power 10 dB
on during acquisition
off during delay
single frequency
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 2 min, 31 sec
7.393
7.388
7.270
7.130
7.127
3.057
3.045
2.813
2.801
0.982
0.964
0.963
0.957
0.944
0.940
F
O
Table 2, Entry 14
0.85
0.84
1.48
0.82
1.02
1.00
0.94
6.25
ppm

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1739-C
INOVA-500 "epoxide"

Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
3072 repetitions
OBSERVE C13, 100.6067917 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec

Table 2, Entry 14



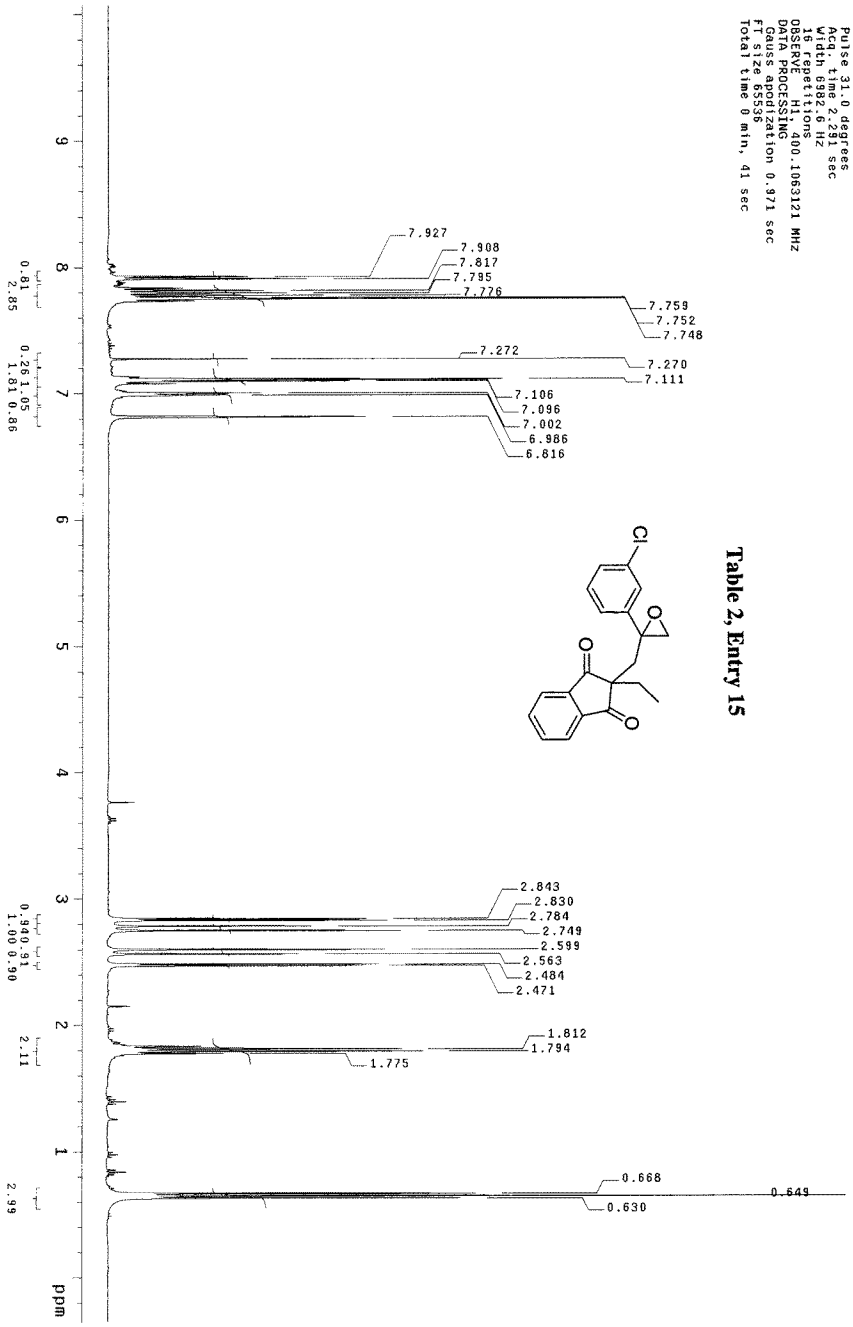


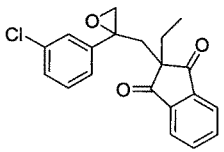
Table 2, Entry 15

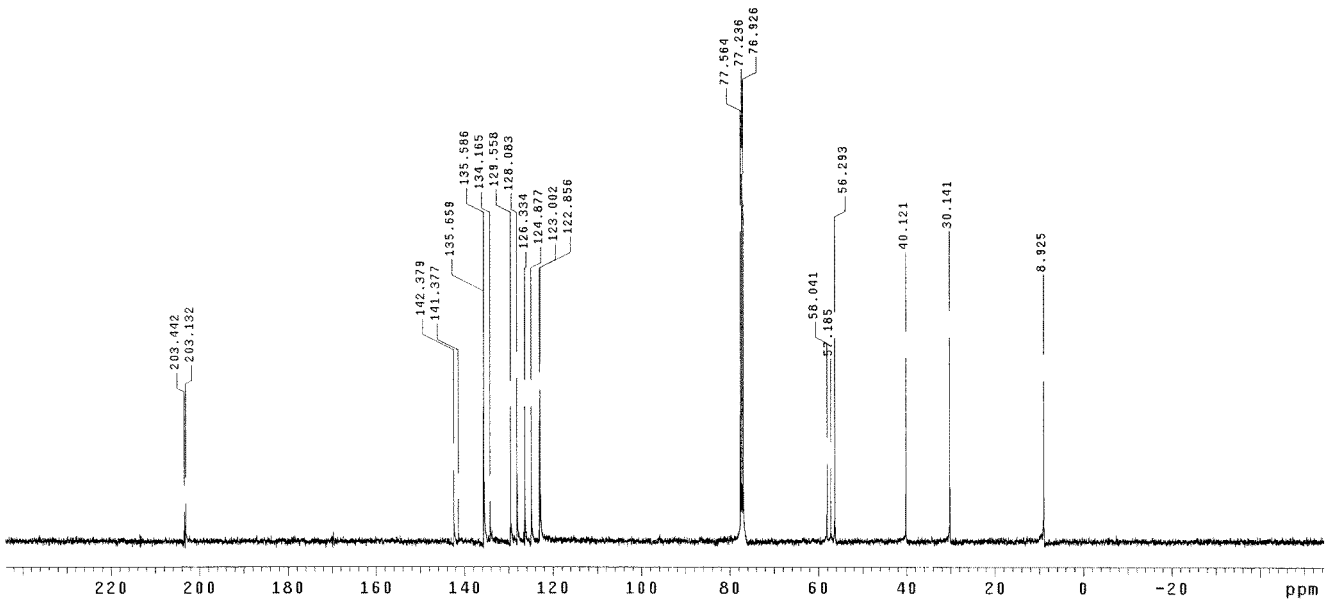
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1609-p-C
INOVA-500 "epoxide"

Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
1328 repetitions
OBSERVE C13, 100.6067917 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec

Table 2, Entry 15





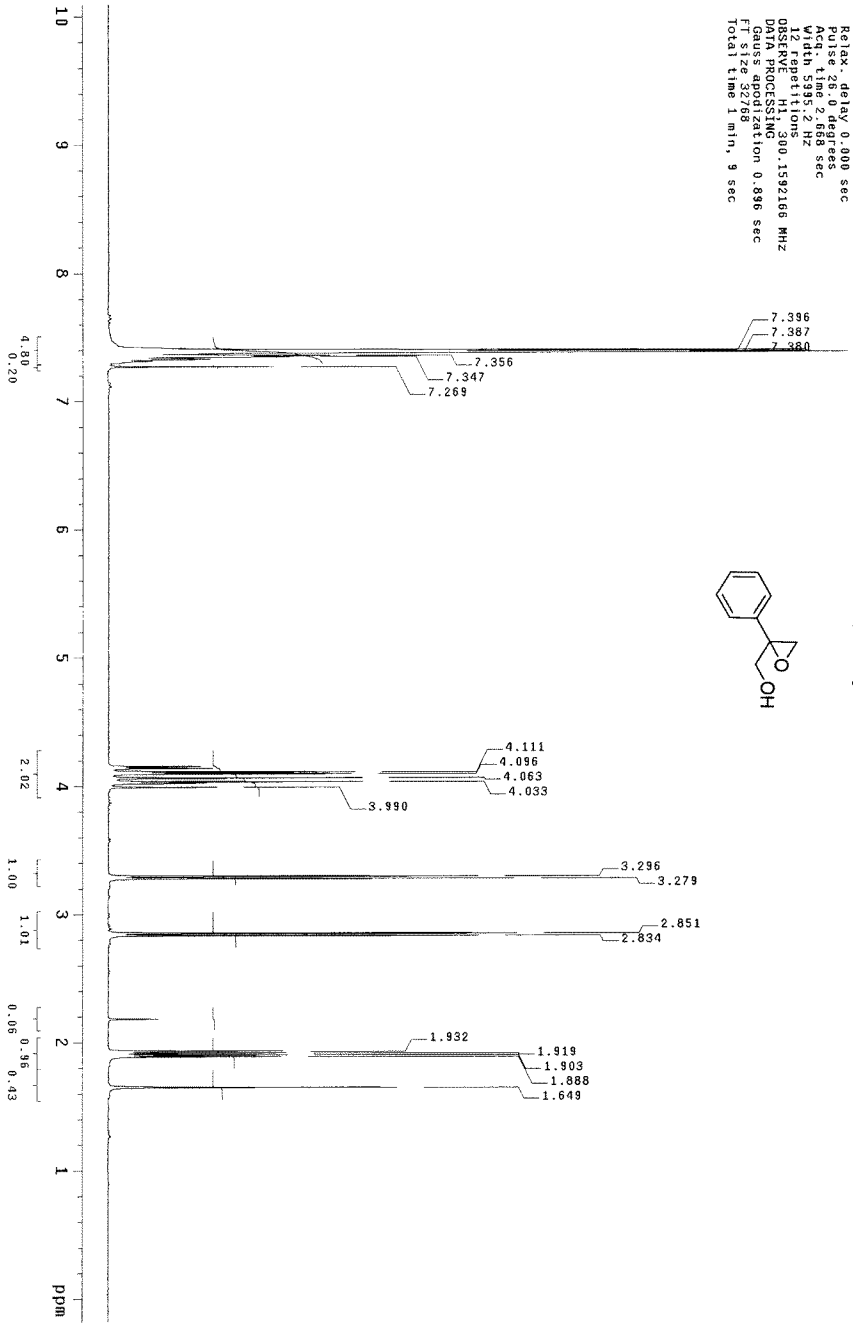


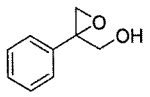
Table 2, Entry 16

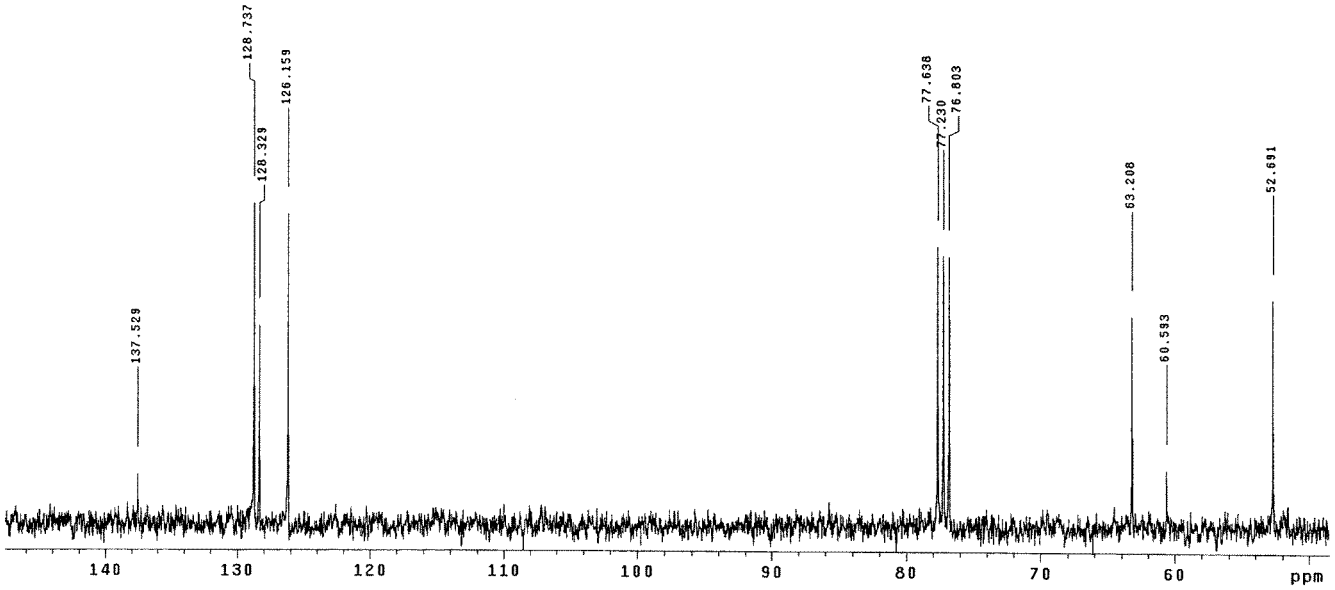
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-18-26-1-13C
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
48 repetitions
OBSERVE C13, 75.4750818 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 11 min, 22 sec

Table 2, Entry 16



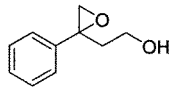


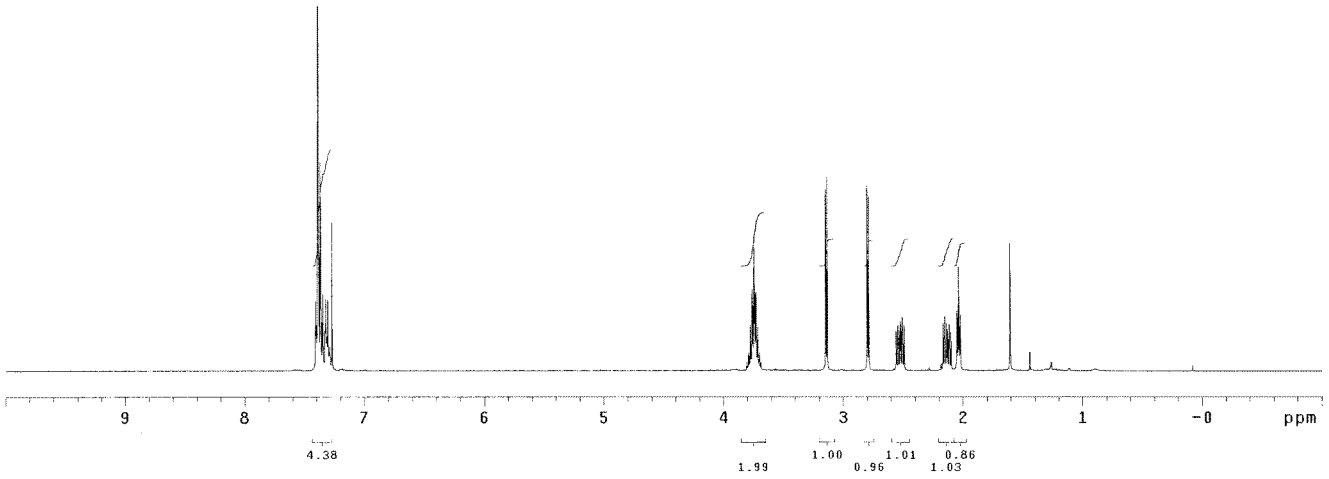
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
File: b1419c-p-H
INOVA-500 "epoxide"

 Pulse 30.8 degrees
 Acq. time 2.291 sec
 Width 6982.6 Hz
 16 repetitions
OBSERVE   H1, 400.1063122 MHz
DATA PROCESSING
 Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 41 sec

Table 2, Entry 17



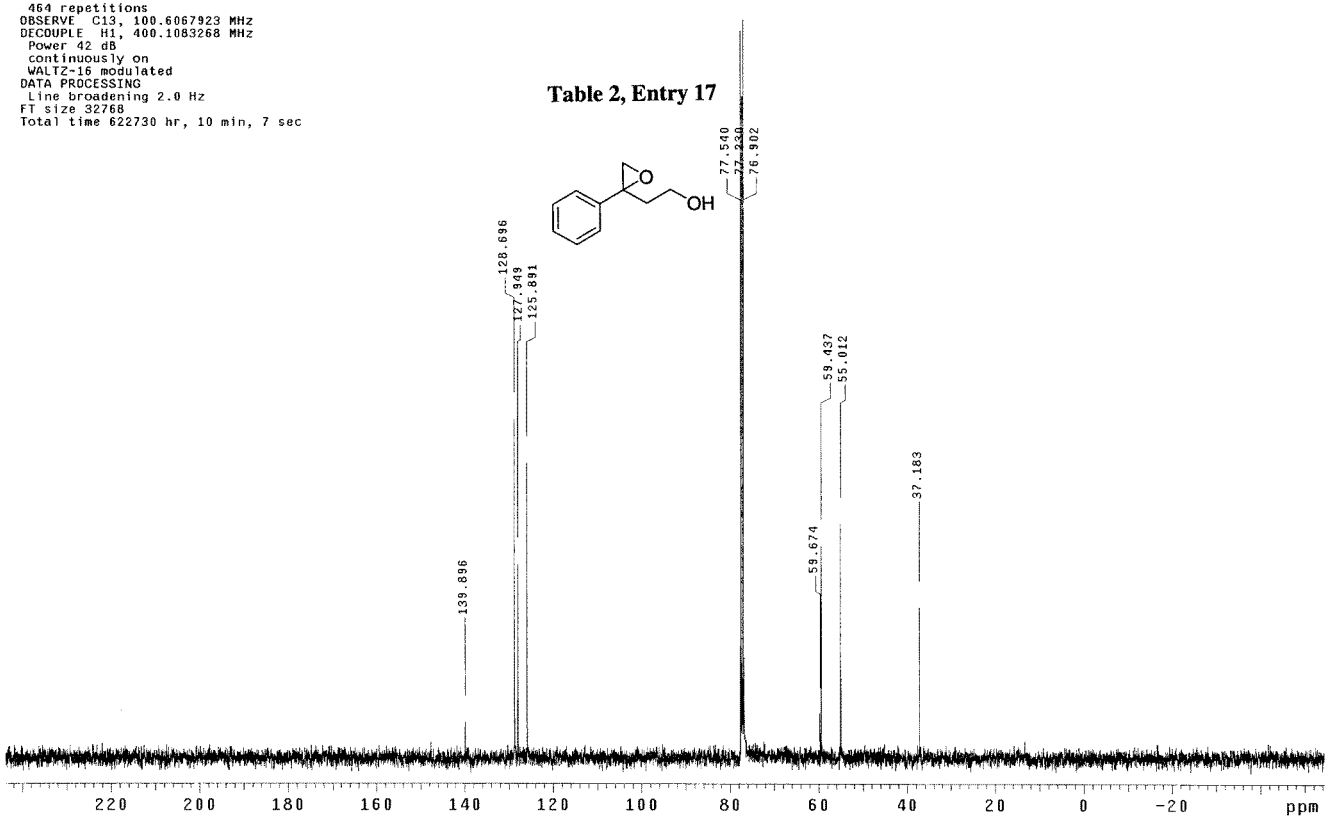


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1419-p--C
INOVA-500 "epoxide"

Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
464 repetitions
OBSERVE C13, 100.6067923 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec

Table 2, Entry 17



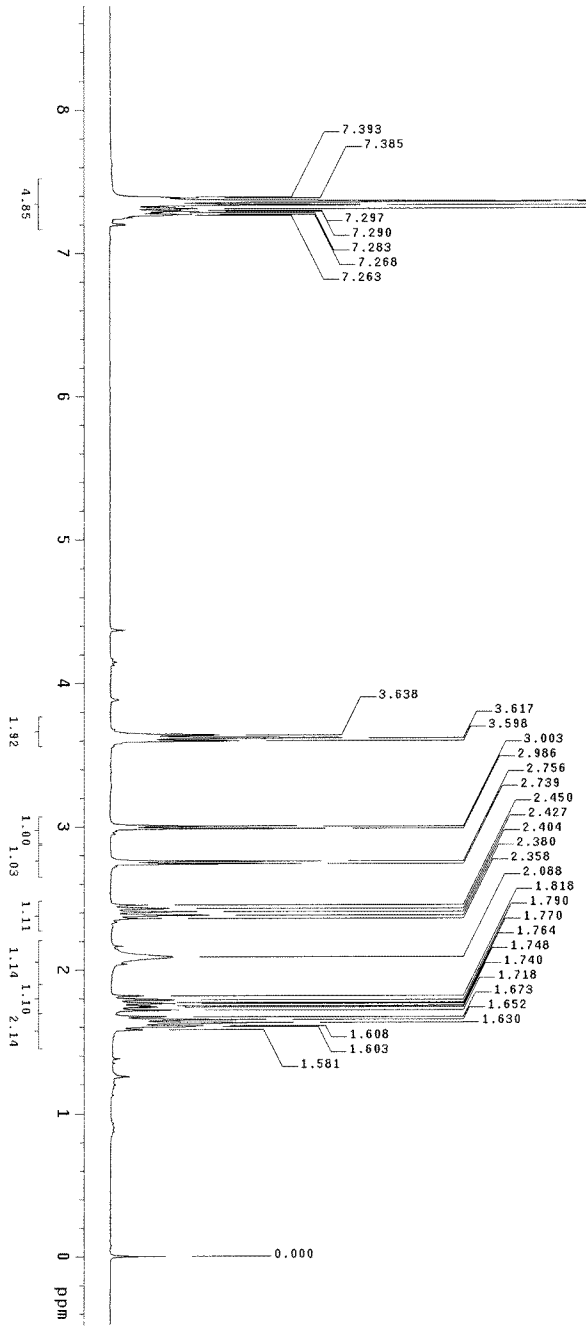


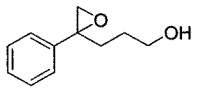
Table 2, Entry 18

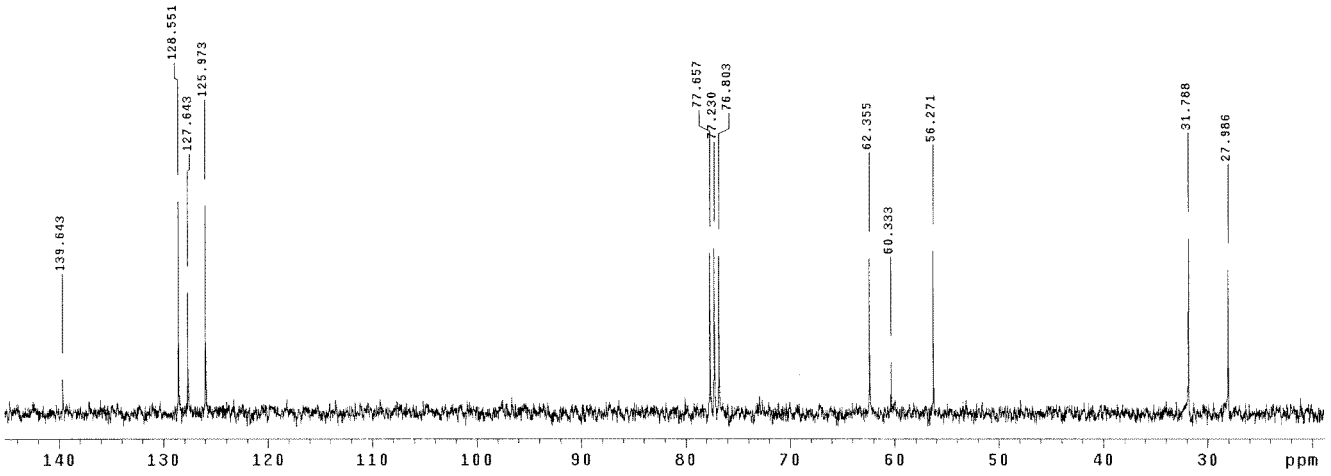
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-18-26-2-13C
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
16 repetitions
OBSERVE C13, 75.4750846 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 11 min, 22 sec

Table 2, Entry 18



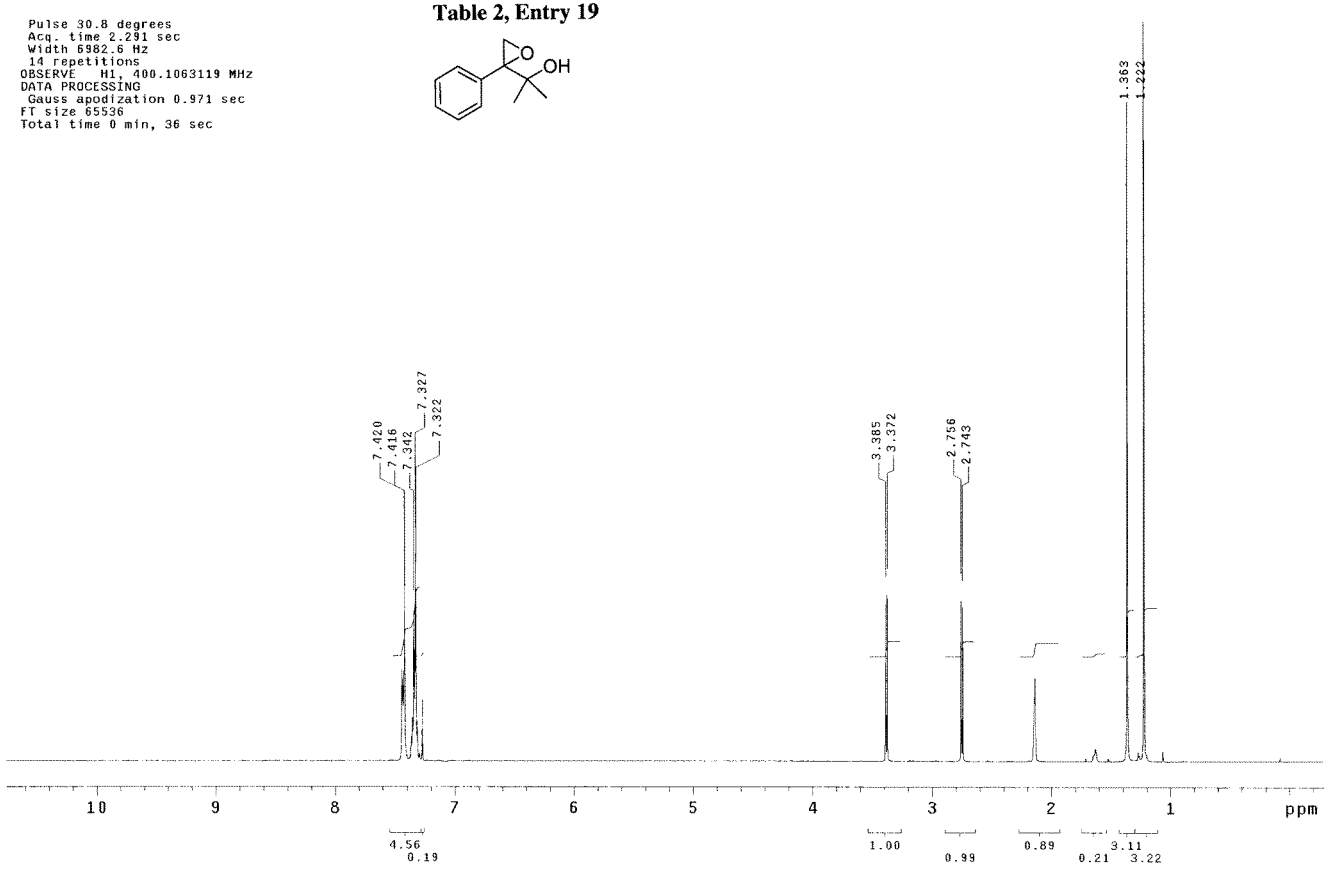


STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: bl419d-p-H
INOVA-500 "epoxide"

Pulse 30.8 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
14 repetitions
OBSERVE H1, 400.1063119 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 36 sec

Table 2, Entry 19

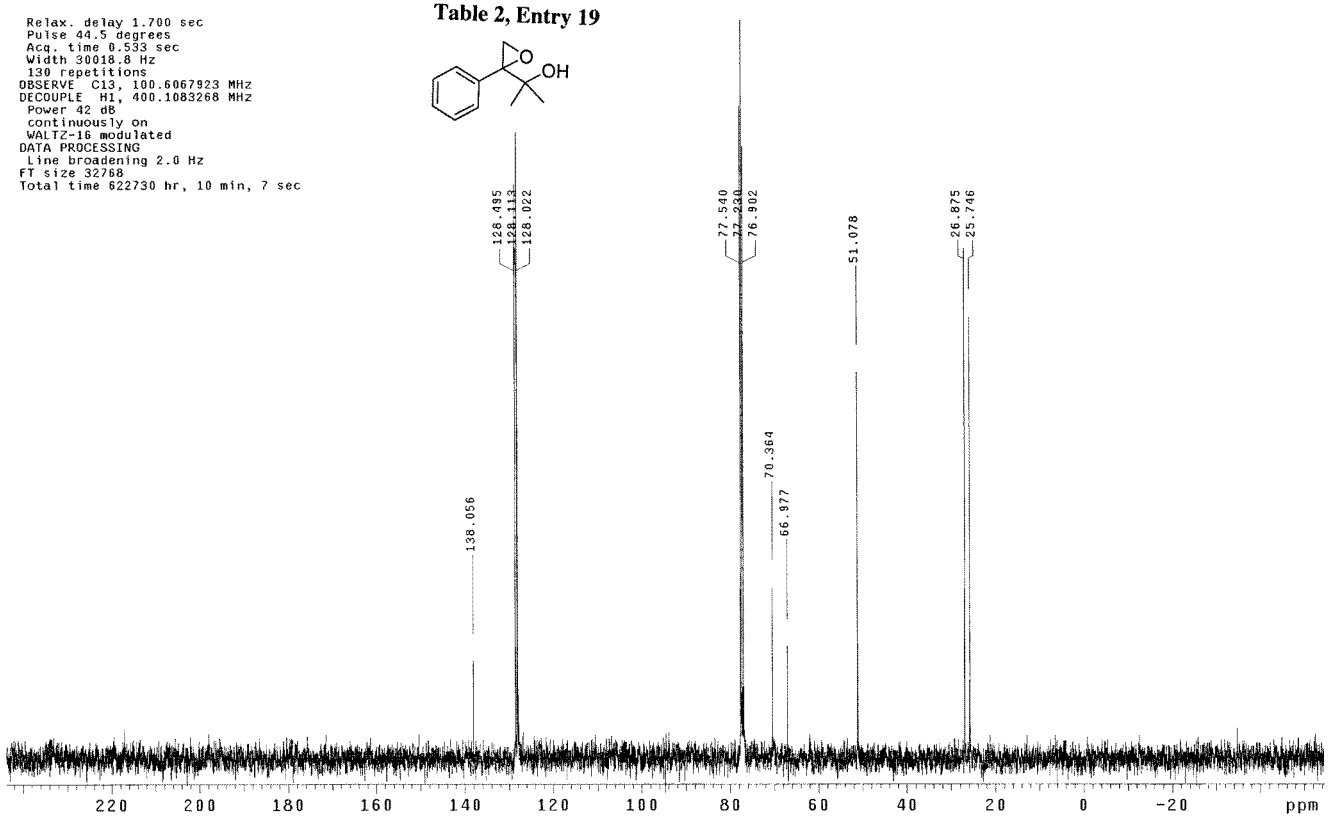


13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1419d-p-C
INOVA-500 "epoxide"

Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
130 repetitions
OBSERVE C13, 100.6067923 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec

Table 2, Entry 19

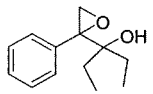


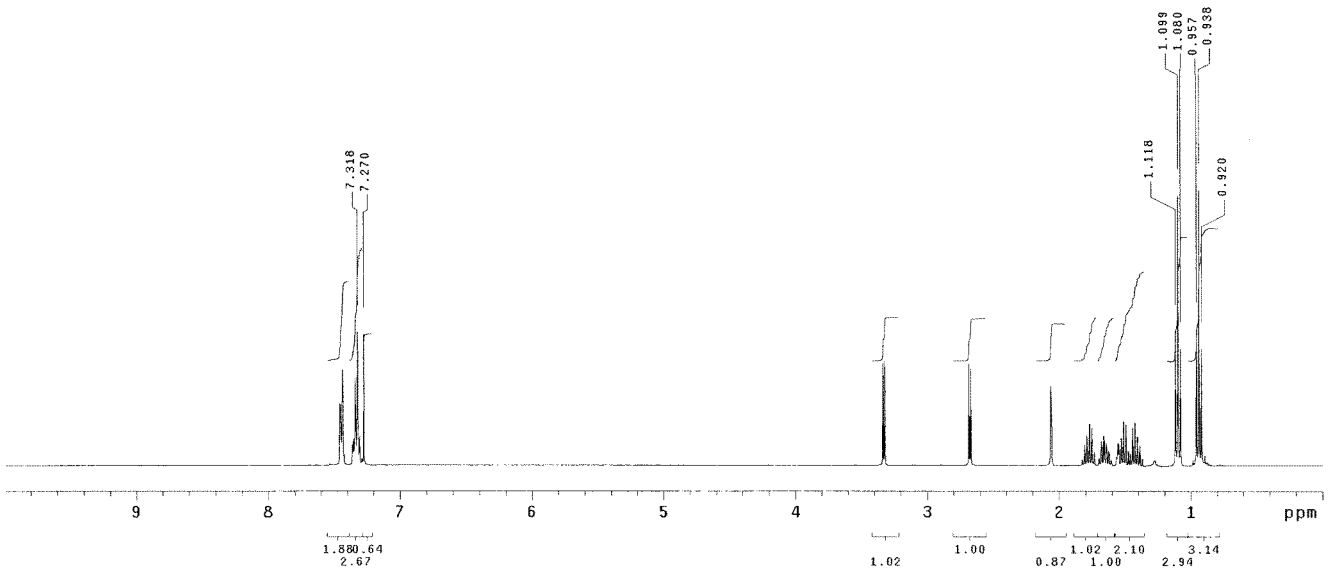
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: b1521b-p-H
INOVA-500 "epoxide"

Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
52 repetitions
OBSERVE H1, 400.1063117 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 2 min, 31 sec

Table 2, Entry 20



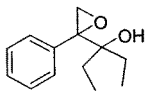


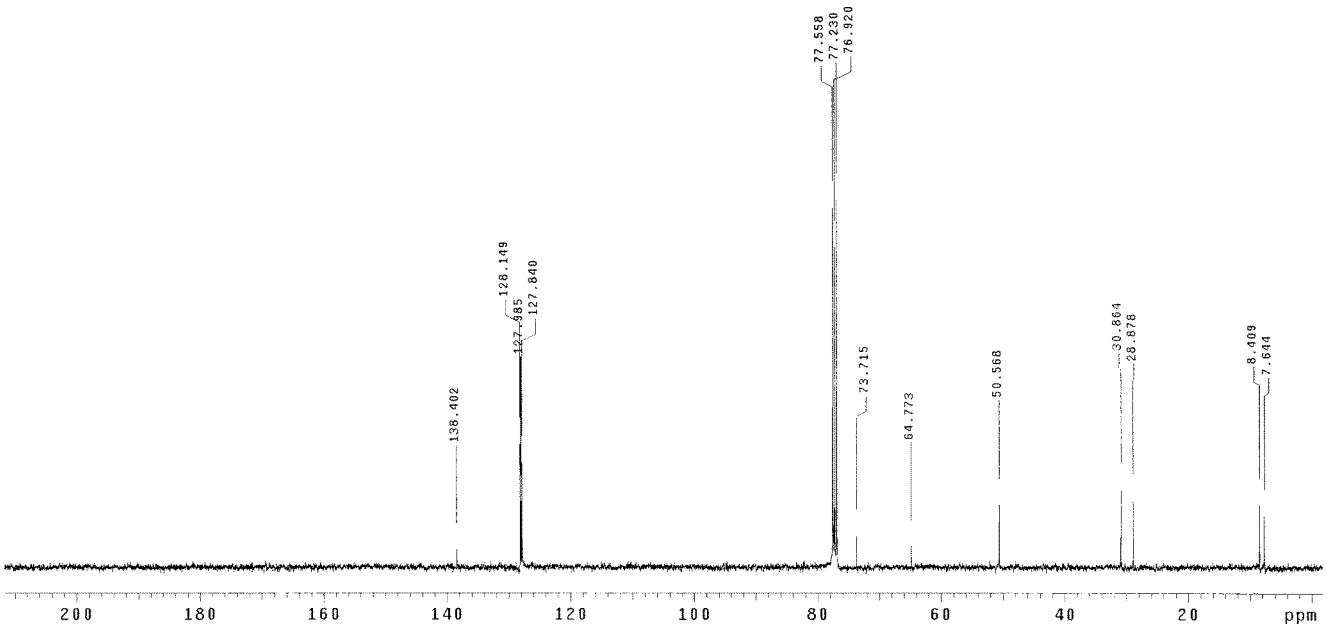
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: b1521b-p-C
INOVA-500 "epoxide"

Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
760 repetitions
OBSERVE C13, 100.6067886 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec

Table 2, Entry 20





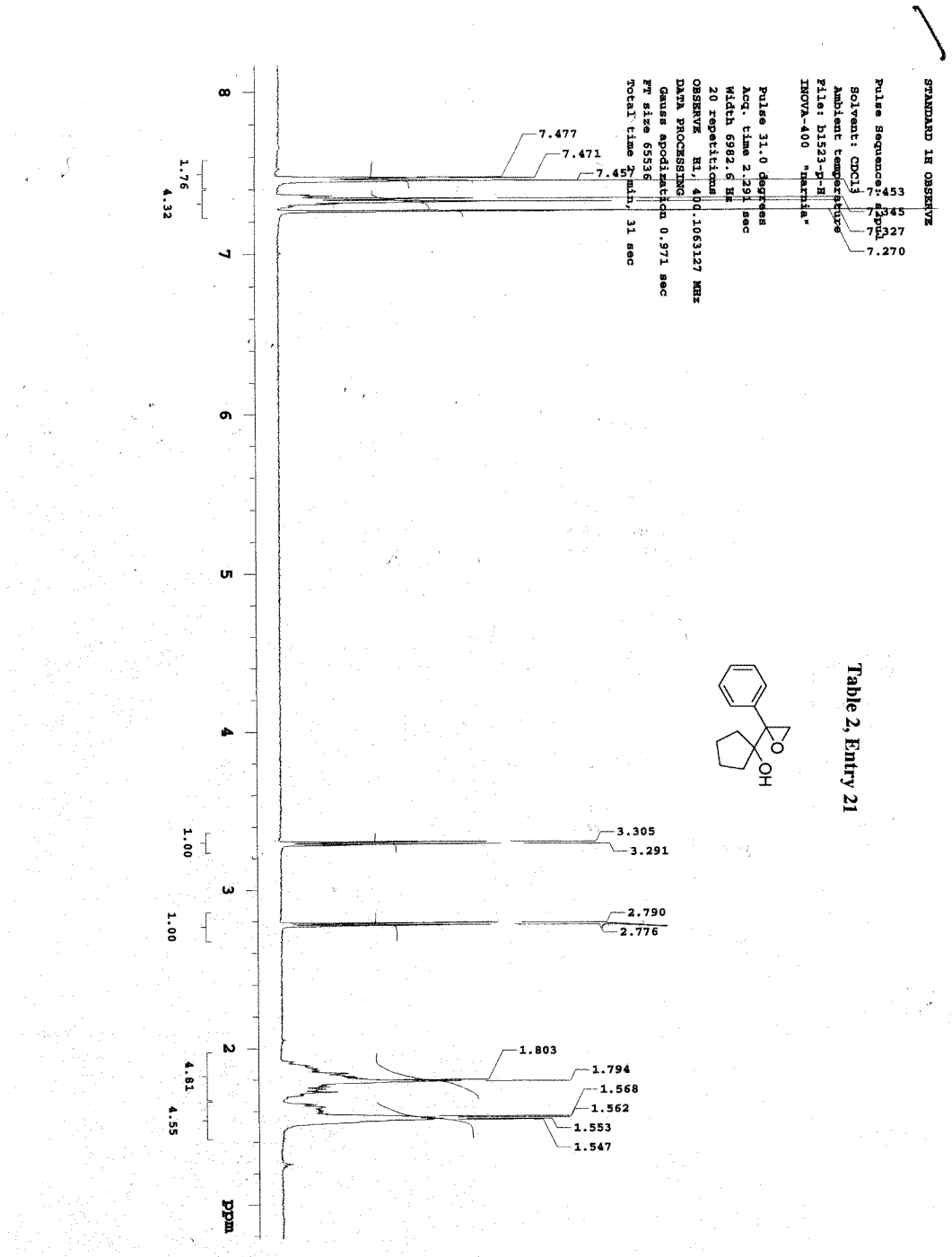


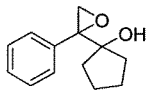
Table 2, Entry 21

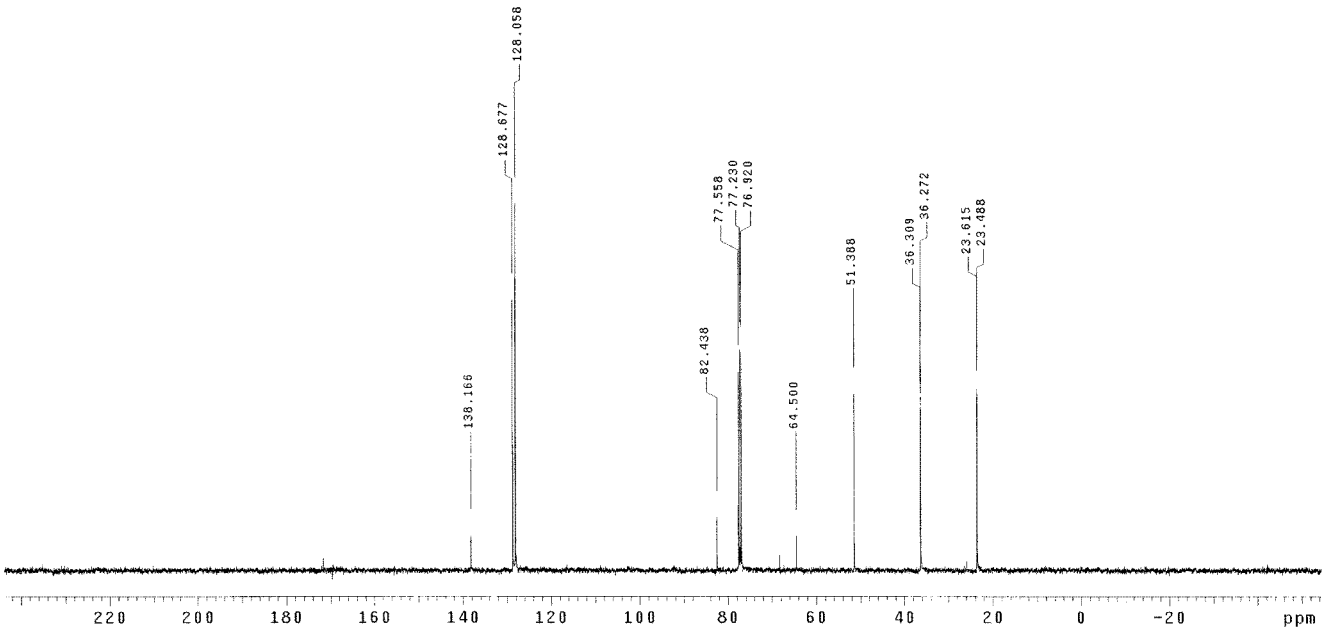
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1523-p-chiral-C
INOVA-500 "epoxide"

Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
320 repetitions
OBSERVE C13, 100.6067923 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec

Table 2, Entry 21



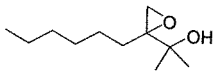


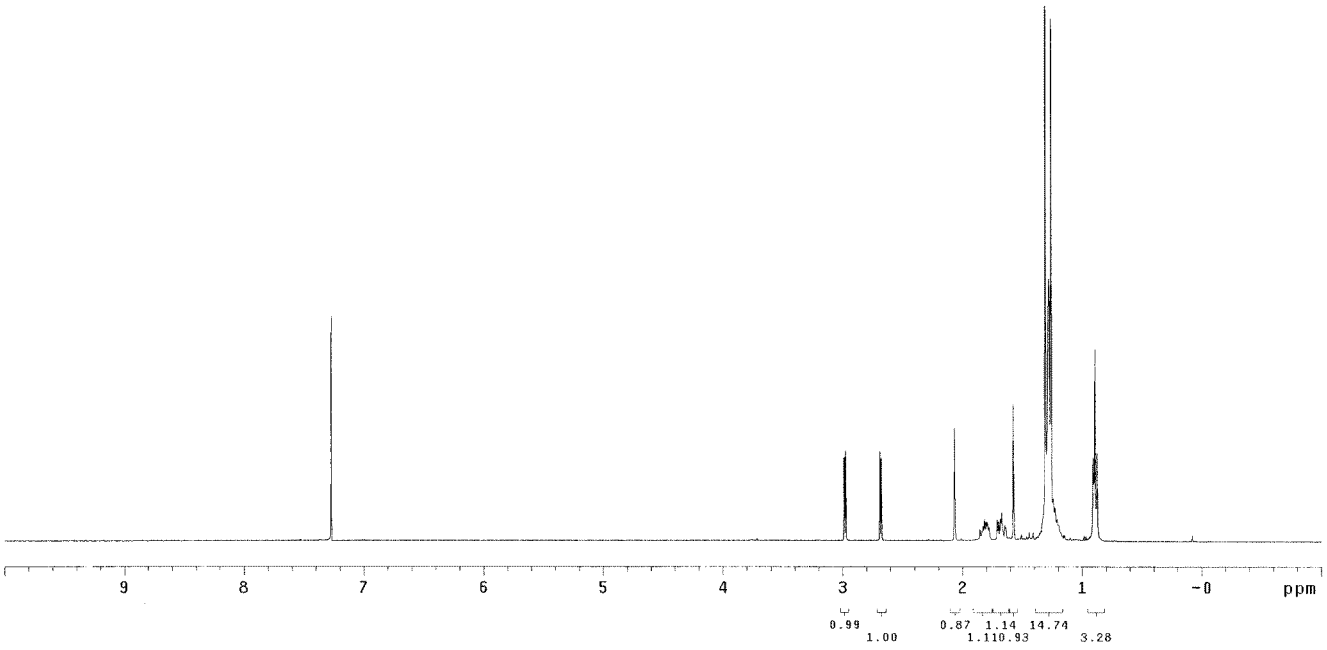
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1548-p-H
INOVA-500 "epoxide"

Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
24 repetitions
OBSERVE H1, 400.1063122 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 59 sec

Table 2, Entry 22



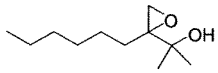


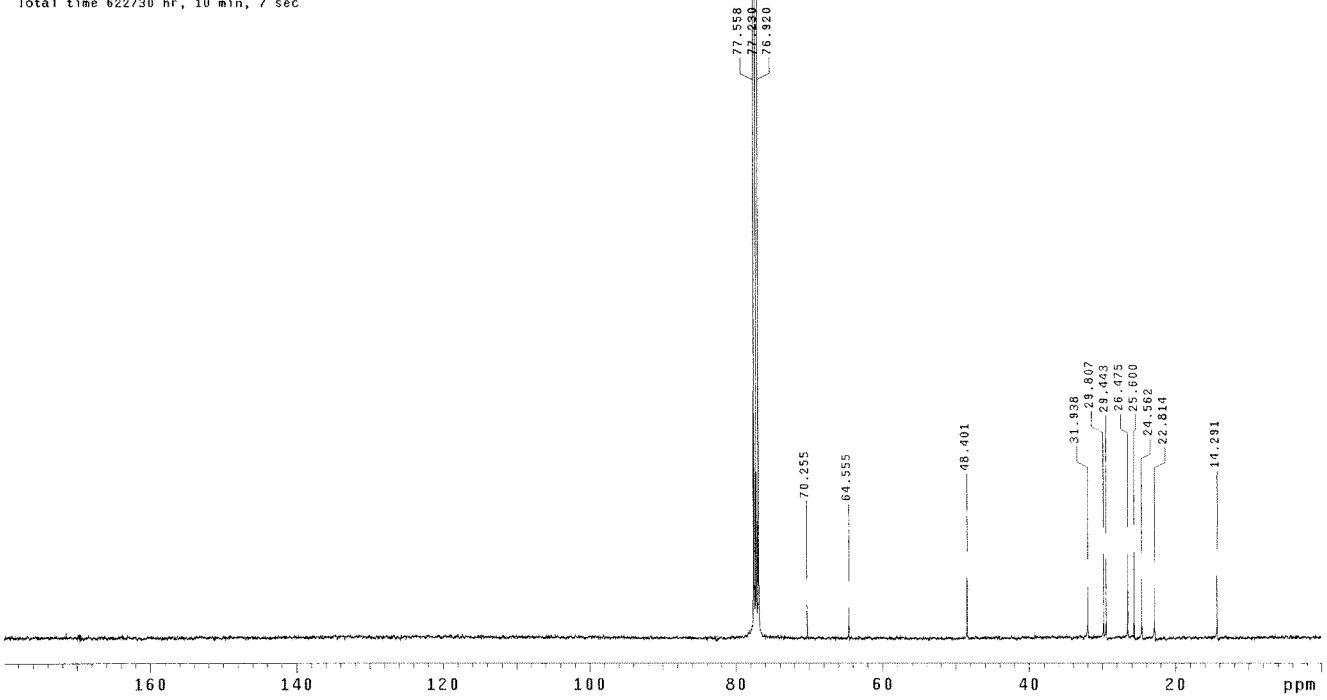
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: b1548-p-C
INOVA-500 "epoxide"

Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
9012 repetitions
OBSERVE C13, 100.6067905 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 622730 hr, 10 min, 7 sec

Table 2, Entry 22





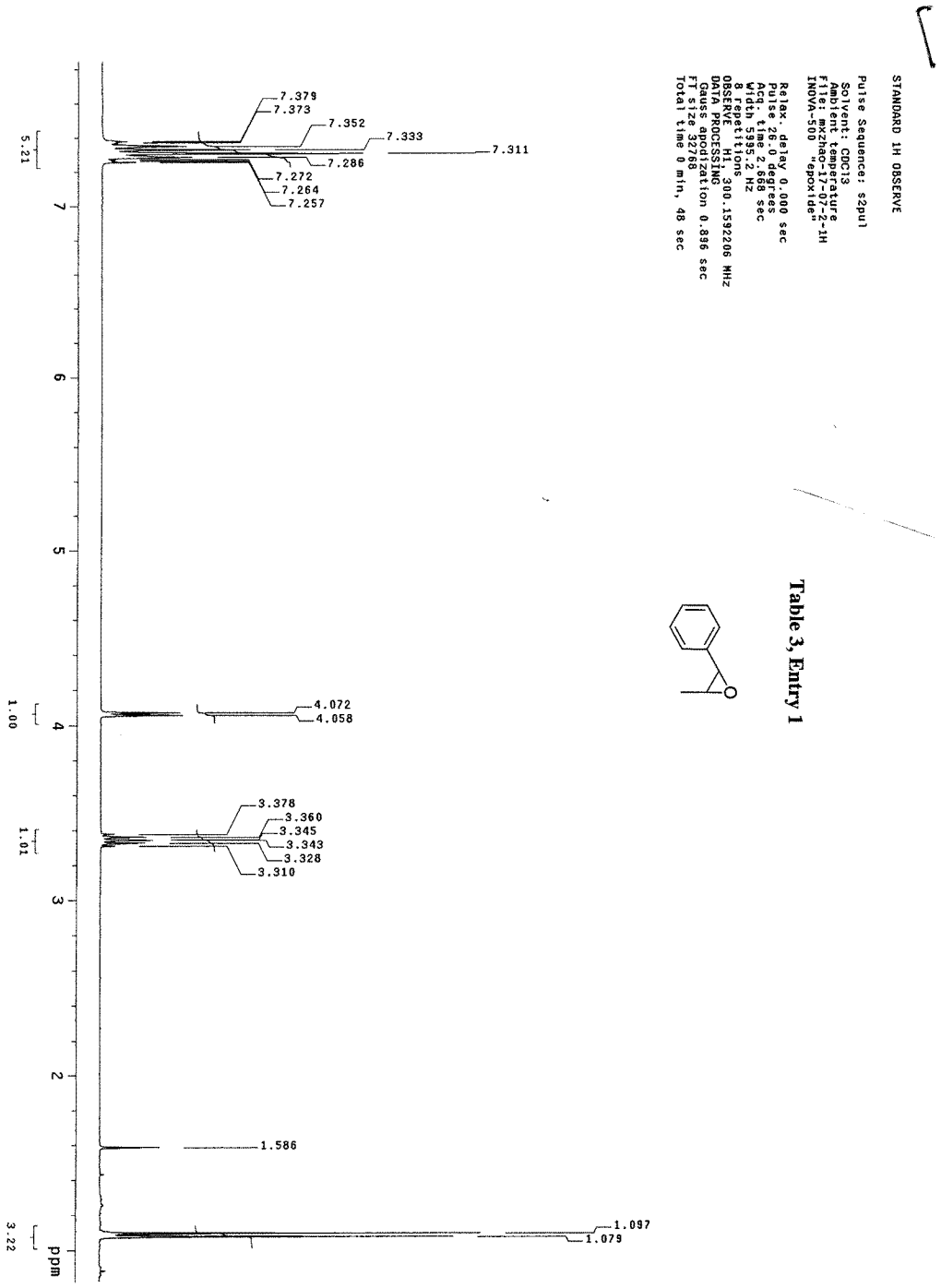


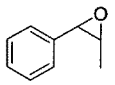
Table 3, Entry 1

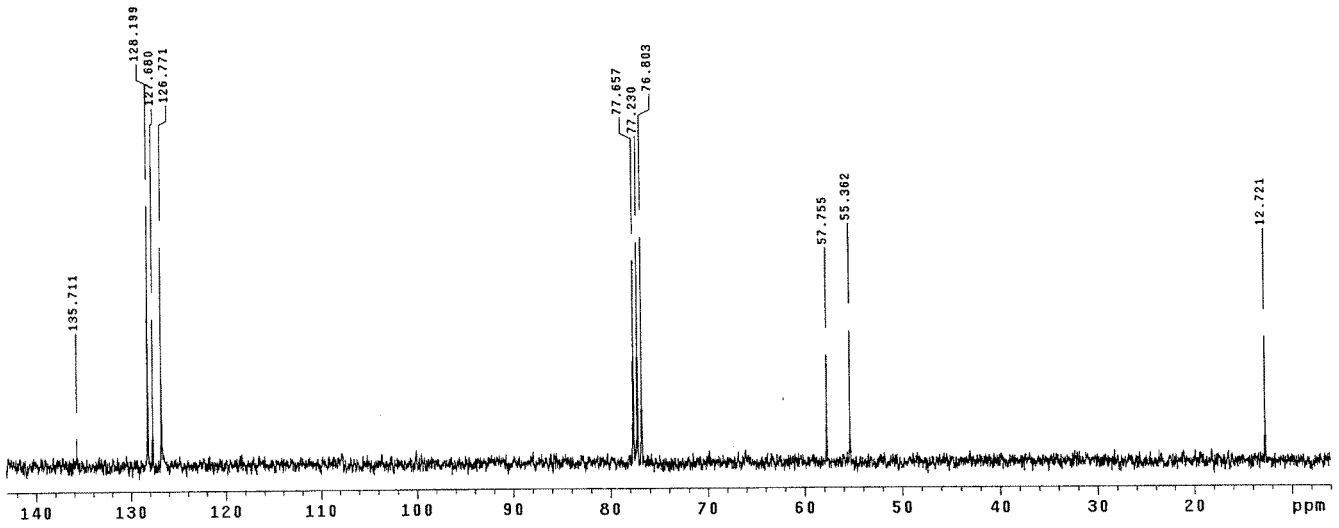
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mwzhao-17-07-2-13C
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
160 repetitions
OBSERVE C13, 75.4750804 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 56 min, 52 sec

Table 3, Entry 1





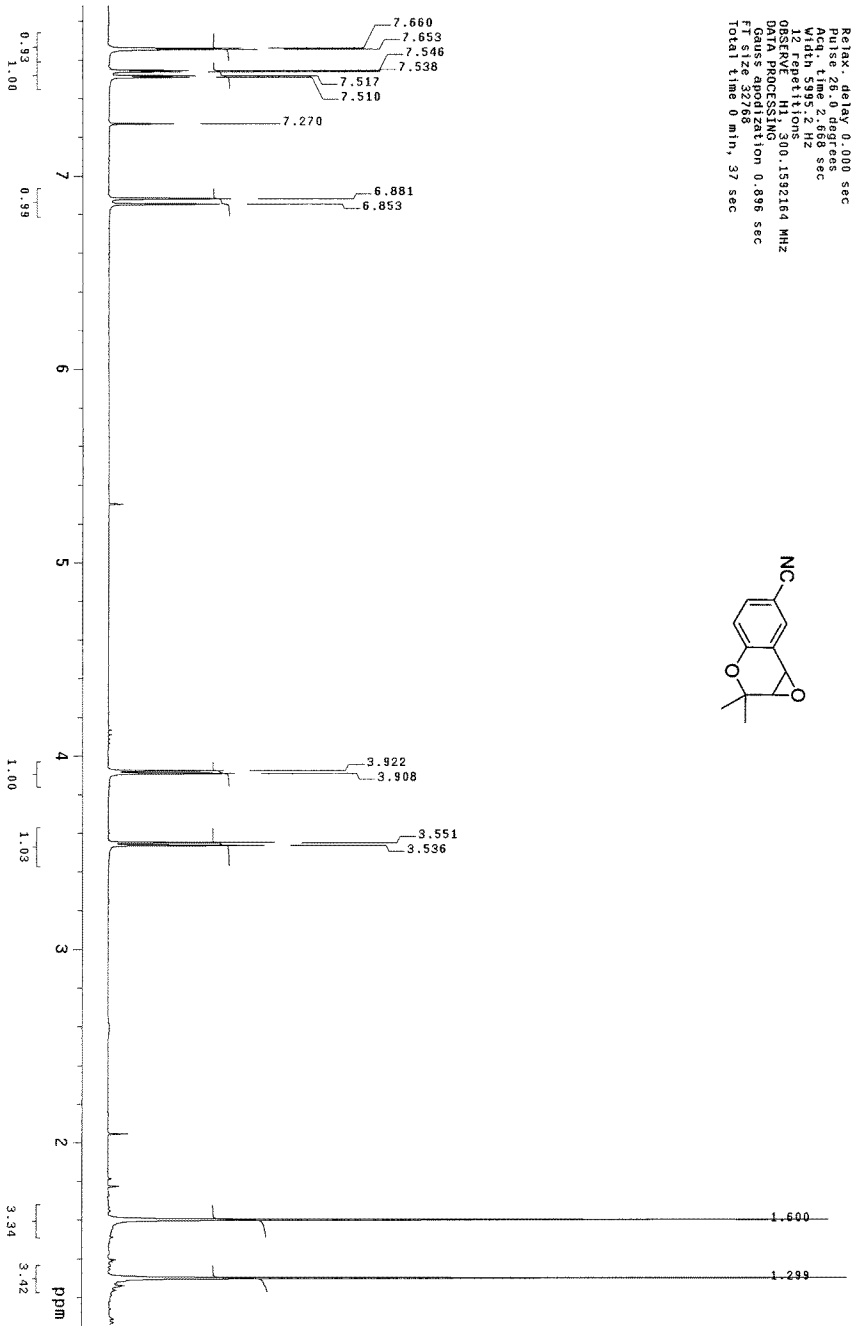


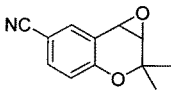
Table 3, Entry 2

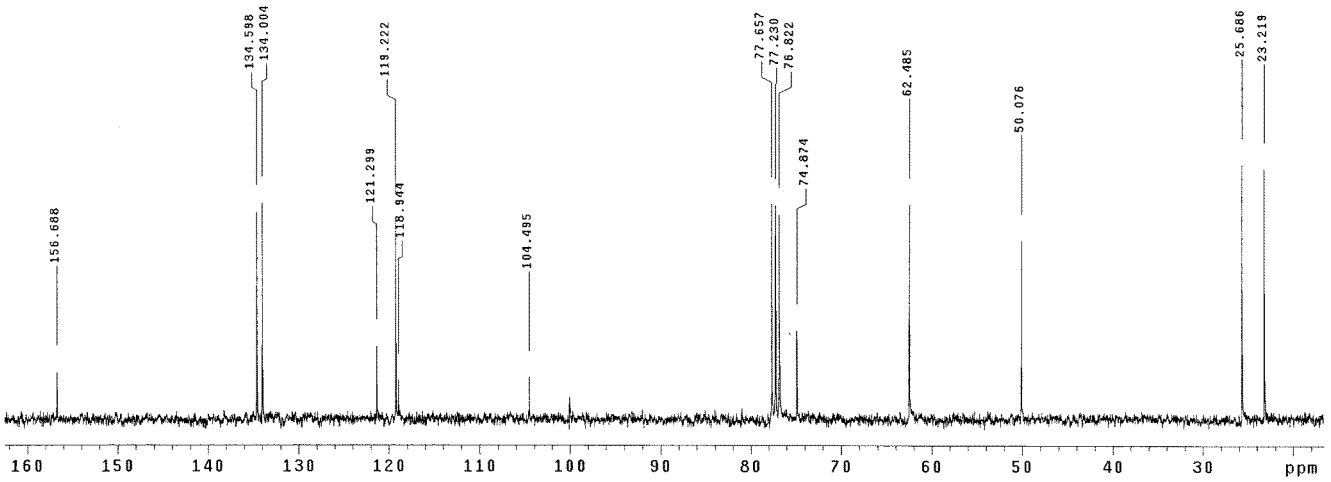
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-17-11-2-13C
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
272 repetitions
OBSERVE C13, 75.4750804 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 11 min, 22 sec

Table 3, Entry 2



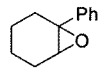


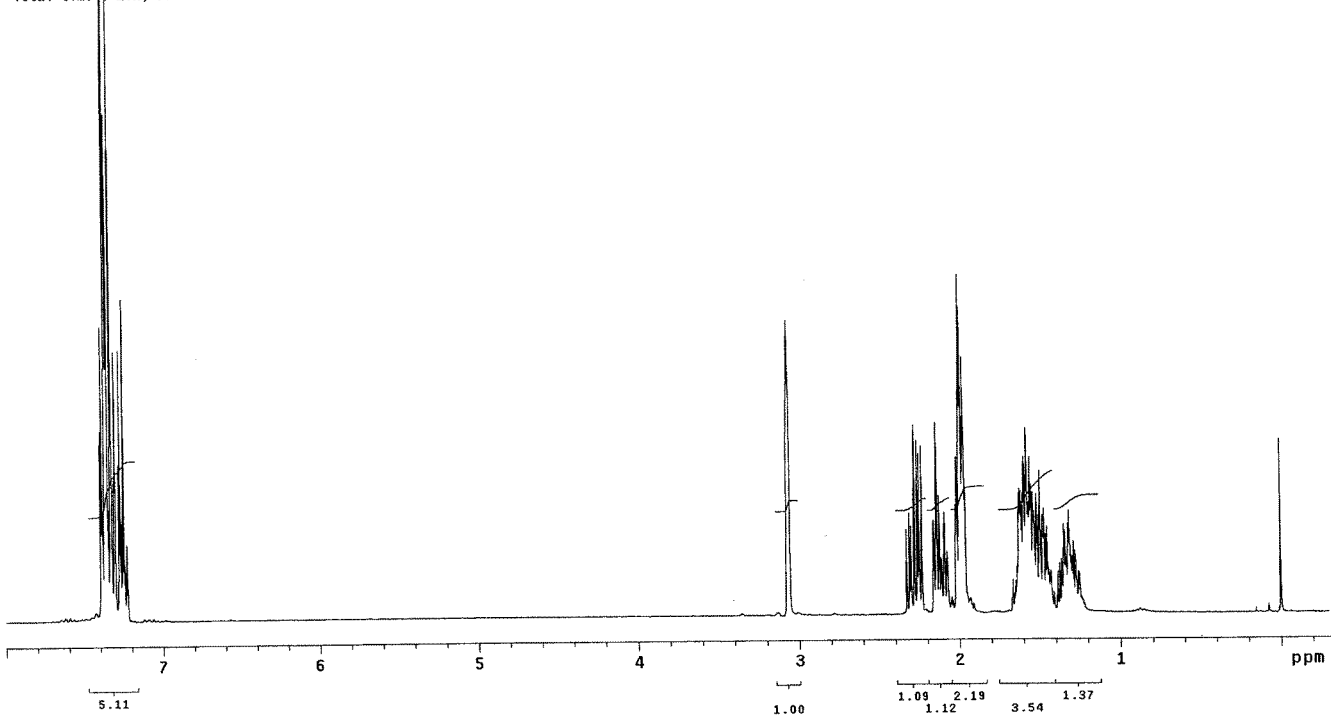
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-17-13-1-1H-2
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
12 repetitions
OBSERVE H1, 300.1592231 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 37 sec

Table 3, Entry 3



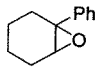


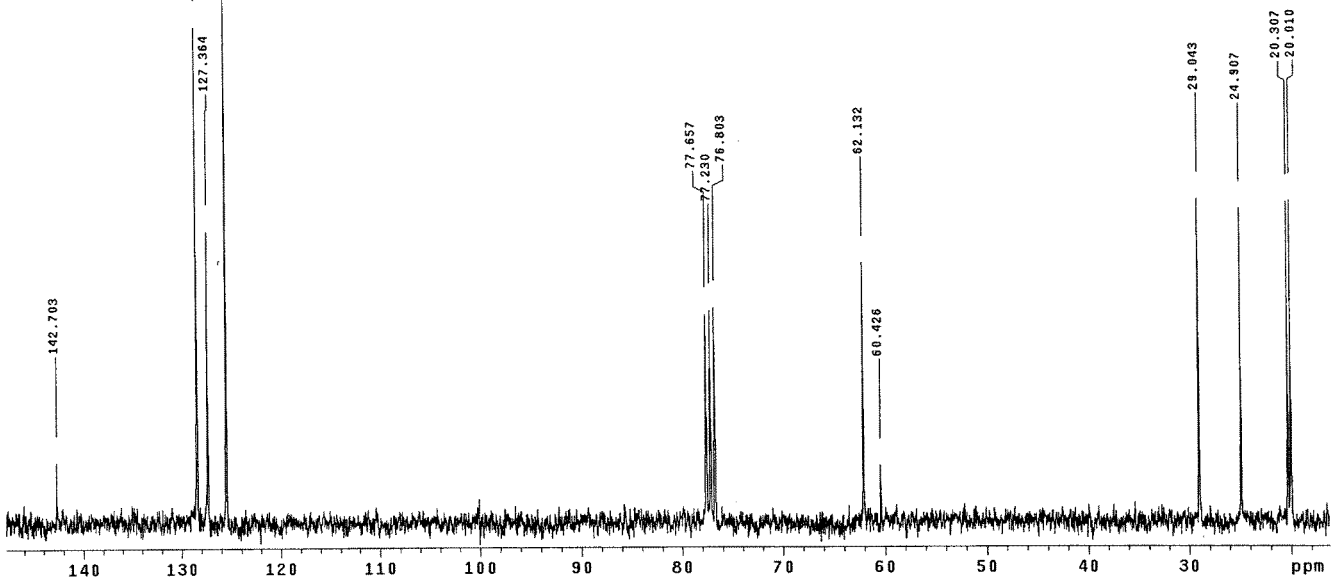
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-17-13-1-13C
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
192 repetitions
OBSERVE C13, 75.4753804 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 11 min, 22 sec

Table 3, Entry 3



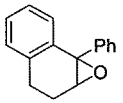


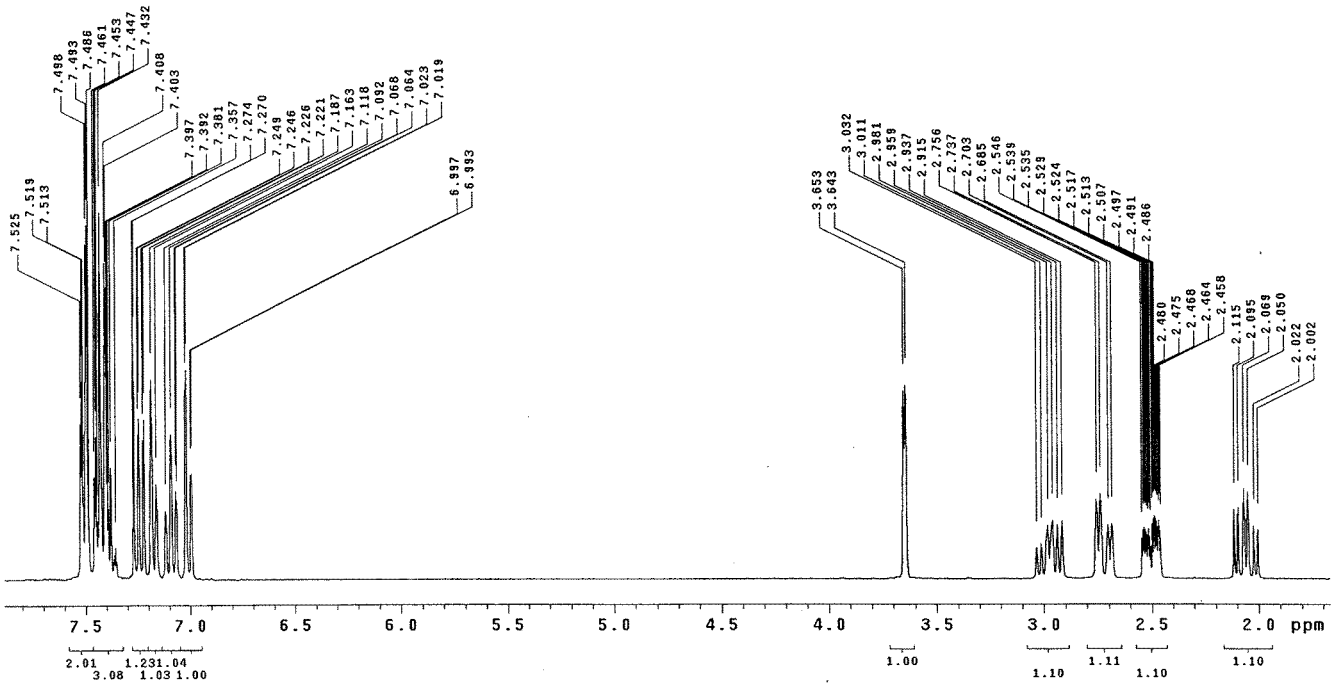
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: mxzhao-17-13-2-1H
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5985.2 Hz
12 repetitions
OBSERVE H1, 300.1592167 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 37 sec

Table 3, Entry 4





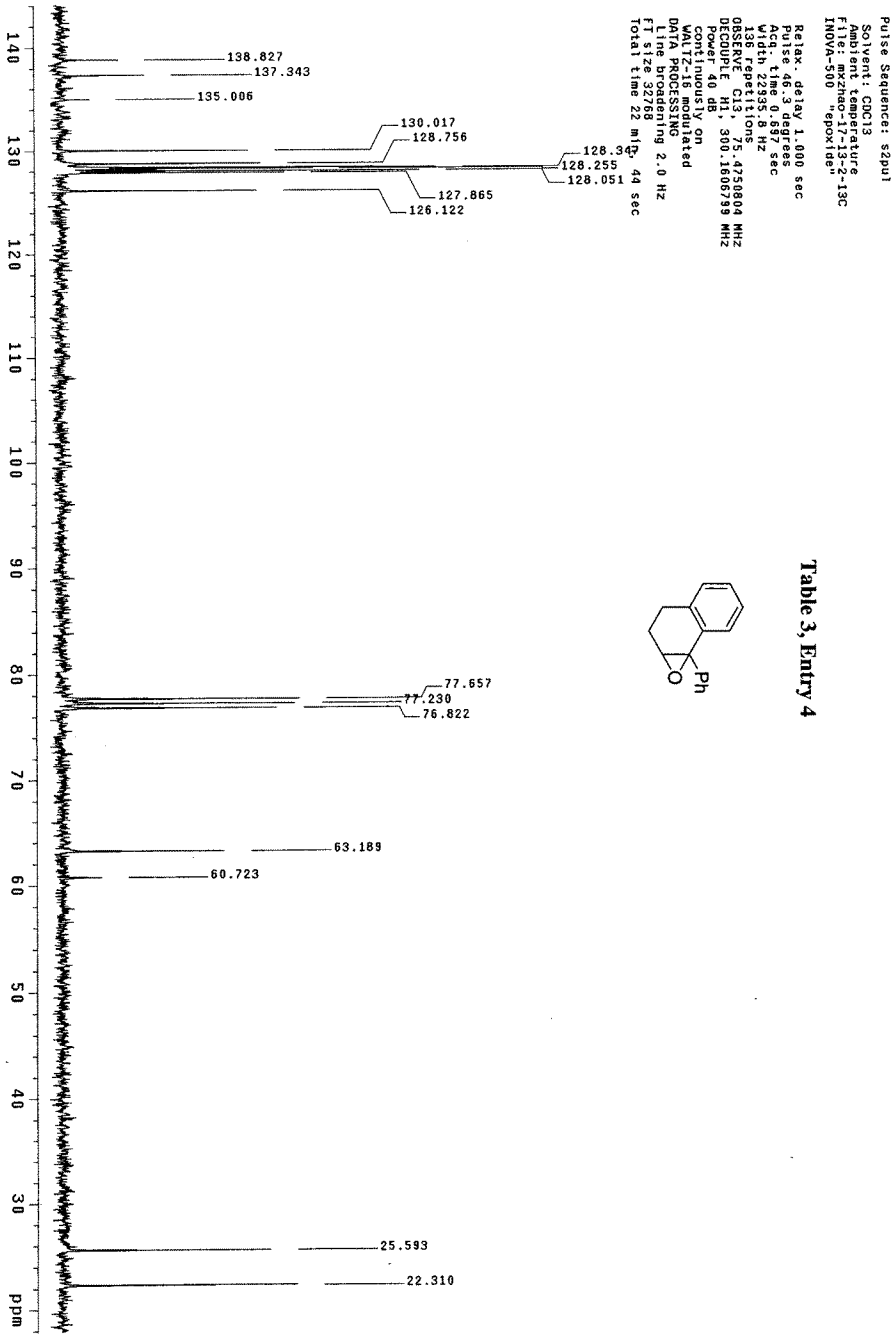
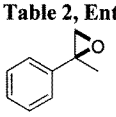


Table 3, Entry 4

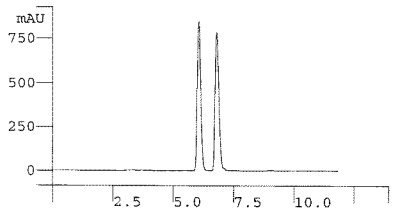
The chromatograms for the determination of enantioselectivity

Table 2, Entry 1



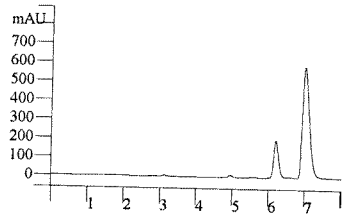
HPLC Cond.: Column: Chiralcel OD (Column No. OD00CE-DL010), Daicel Chemical Industries, Ltd.
Eluent: Hexanes/IPA (95/5); **Flow rate:** 1.0 mL/min; **Detection:** UV220 nm.

Racemic



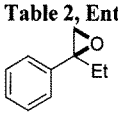
Peak No	Ret. Time (min)	Result ()	Area (counts)
1	6.037	50.3640	4463937
2	6.779	49.6360	4399408
		100.0000	**8863345**

Chiral



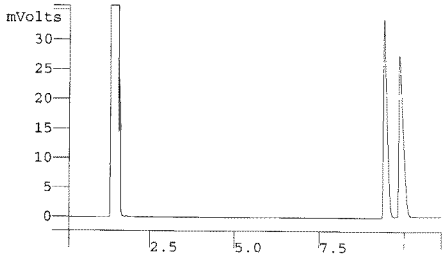
Peak No	Ret. Time (min)	Result ()	Area (counts)
1	6.193	18.8644	944562
2	6.979	81.1356	4062558
		100.0000	**5007120**

Table 2, Entry 2



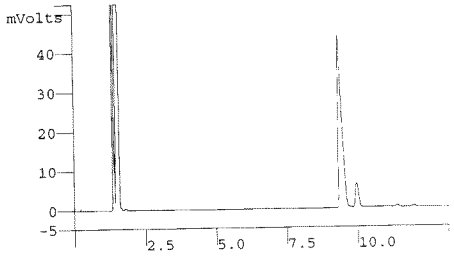
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 100 °C; **Carrier:** Helium, head pressure 25 psi; **Detection:** FID 250 °C.

Racemic



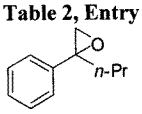
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		50.0868	9.382	236820
2		49.9132	9.833	236000
	Totals	**100.0000**		**472820**

Chiral



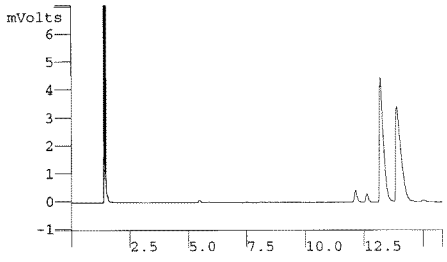
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		89.2228	9.339	385583
2		10.7772	9.959	46575
	Totals	**100.0000**		**432158**

Table 2, Entry 3



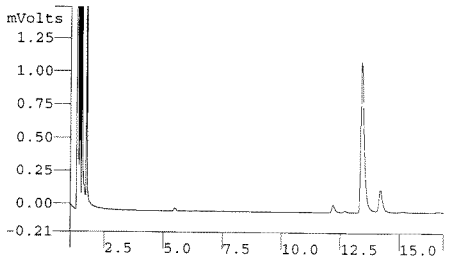
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 100 °C; **Carrier:** Helium, head pressure 25 psi; **Detection:** FID 250 °C.

Racemic



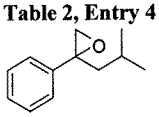
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		49.7145	13.186	51657
2		50.2855	13.877	52250
	Totals	**100.0000**		**103907**

Chiral



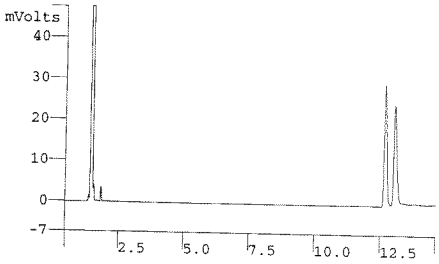
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		87.3705	13.384	11521
2		12.6295	14.184	1665
	Totals	**100.0000**		**13186**

Table 2, Entry 4



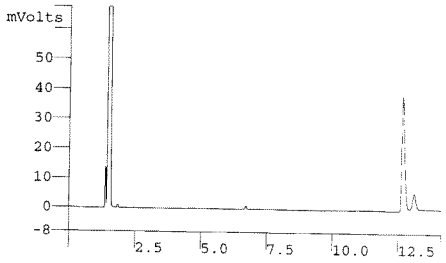
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 105 °C; **Carrier:** Helium, head pressure 25 psi; **Detection:** FID 250 °C.

Racemic



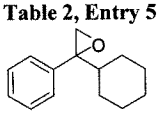
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		50.5073	12.648	186641
2		49.4927	13.016	182892
	Totals	**100.0000**		**369533**

Chiral



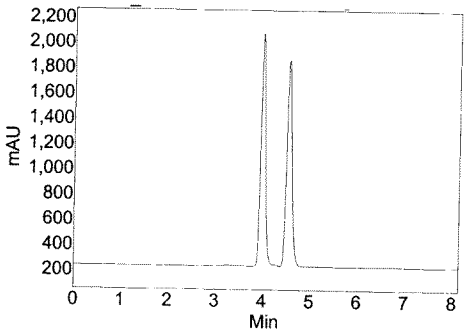
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		87.1355	12.637	277867
2		12.8645	13.091	41024
	Totals	**100.0000**		**318891**

Table 2, Entry 5



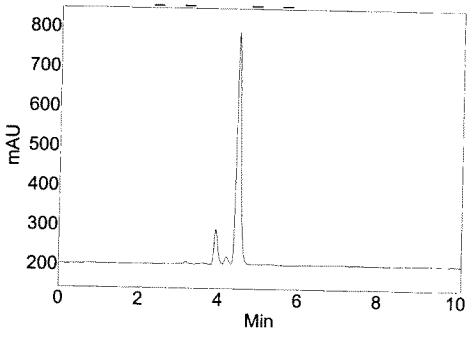
HPLC Cond.: Column: Chiralcel OD (Column No. OD00CE-DL010), Daicel Chemical Industries, Ltd.
Eluent: Hexanes/IPA (90/10); **Flow rate:** 1.0 mL/min; **Detection:** UV210 nm.

Racemic



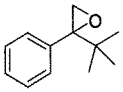
Index	Time [Min]	Area % [%]
1	3.99	49.876
2	4.54	50.124
Total		100.000

Chiral



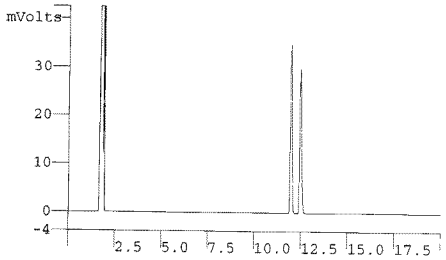
Index	Time [Min]	Area % [%]
1	3.93	11.489
2	4.47	88.511
Total		100.000

Table 2, Entry 6



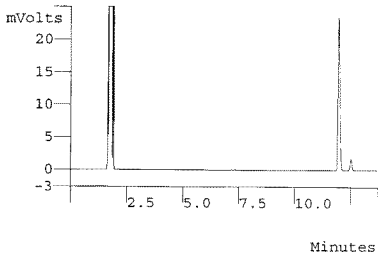
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 110 °C; **Carrier:** Helium, head pressure 20 psi; **Detection:** FID 250 °C.

Racemic



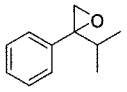
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		50.0457	11.934	207370
2		49.9543	12.439	206991
	Totals	**100.0000**		**414361**

Chiral



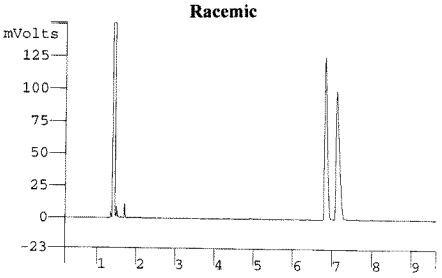
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		92.9477	11.948	134771
2		7.0523	12.519	10226
	Totals	**100.0000**		**144997**

Table 2, Entry 7



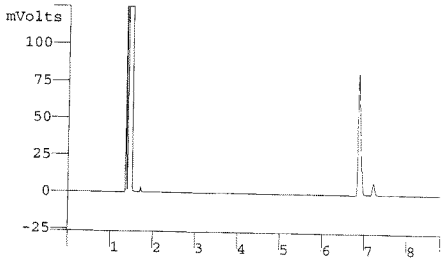
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 115 °C; **Carrier:** Helium, head pressure 25 psi; **Detection:** FID 250 °C.

Racemic



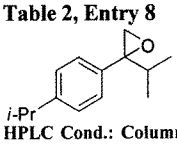
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		50.0425	6.787	574087
2		49.9575	7.084	573112
	Totals	**100.0000**		**1147199**

Chiral



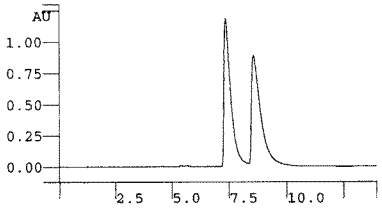
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		92.0163	6.839	344204
2		7.9837	7.202	29865
	Totals	**100.0000**		**374069**

Table 2, Entry 8



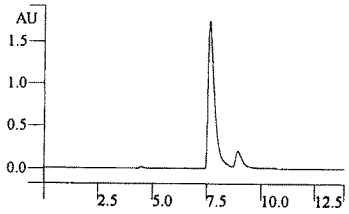
HPLC Cond.: Column: Chiralcel OD (Column No. OD00CE-DL010), Daicel Chemical Industries, Ltd.
Eluent: Hexanes/IPA (98/2); **Flow rate:** 0.75 mL/min; **Detection:** UV220 nm.

Racemic



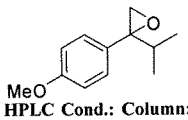
Peak No	Ret. Time (min)	Result ()	Area (counts)
1	7.347	49.7765	12321675
2	8.561	50.2235	12432322
		100.0000	**24753996**

Chiral



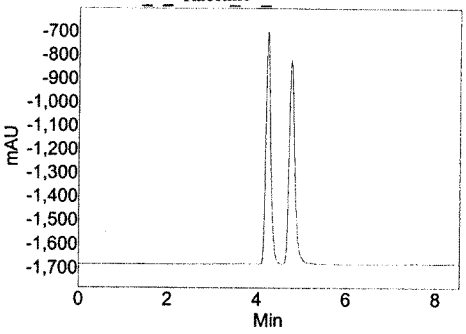
Peak No	Ret. Time (min)	Result ()	Area (counts)
1	7.589	90.7719	18586268
2	8.924	9.2281	1889533
		100.0000	**20475800**

Table 2, Entry 9



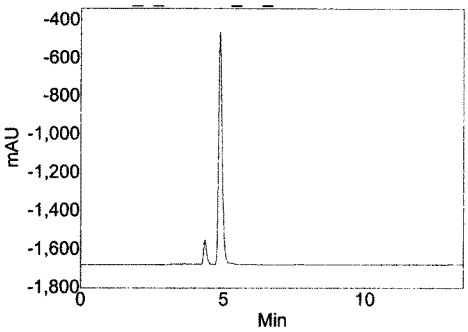
HPLC Cond.: Column: Chiralcel OD (Column No. OD00CE-DL010), Daicel Chemical Industries, Ltd.
Eluent: Hexanes/IPA (90/10); **Flow rate:** 1.0 mL/min; **Detection:** UV220 nm.

Racemic



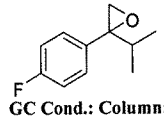
Index	Time [Min]	Area % [%]
1	4.23	49.760
2	4.76	50.240
Total		100.000

Chiral



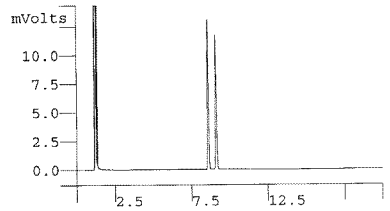
Index	Time [Min]	Area % [%]
1	4.34	7.975
2	4.87	92.025
Total		100.000

Table 2, Entry 10



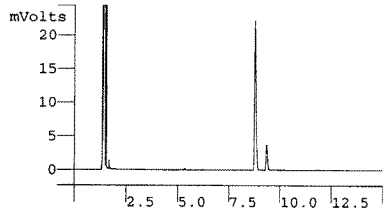
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 110 °C; **Carrier:** Helium, head pressure 25 psi; **Detection:** FID 250 °C.

Racemic



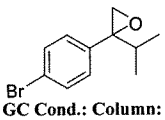
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		49.9955	8.568	60211
2		50.0045	9.083	60222
	Totals	**100.0000**		**120433**

Chiral



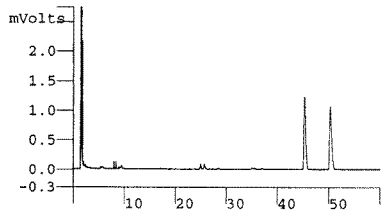
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		86.9589	8.744	113854
2		13.0411	9.341	17074
	Totals	**100.0000**		**130928**

Table 2, Entry 11



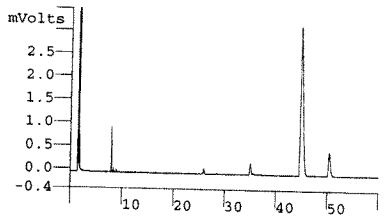
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 110 °C; **Carrier:** Helium, head pressure 25 psi; **Detection:** FID 250 °C.

Racemic



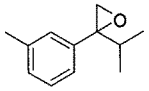
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		50.0490	45.237	29396
2		49.9510	50.234	29339
	Totals	**100.0000**		**58735**

Chiral



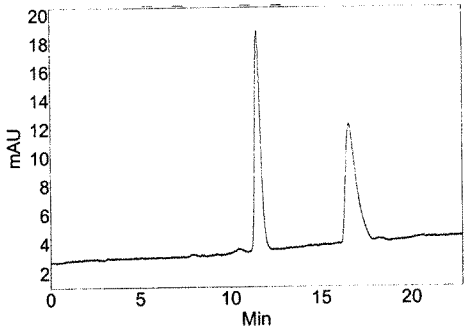
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		89.1383	44.686	108110
2		10.8617	50.329	13173
	Totals	**100.0000**		**121283**

Table 2, Entry 12



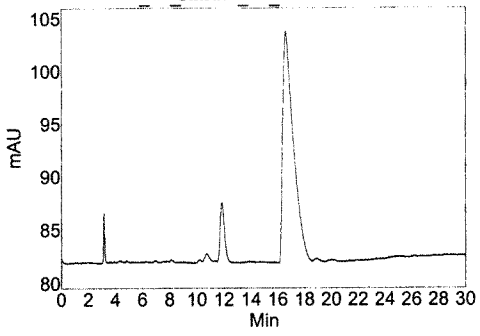
HPLC Cond.: Column: Chiralcel OD (Column No. OD00CE-DL010), Daicel Chemical Industries, Ltd.
Eluent: Hexanes/IPA (98/2); **Flow rate:** 1.0 mL/min; **Detection:** UV254 nm.

Racemic



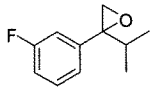
Index	Time [Min]	Area % [%]
1	11.46	50.169
2	16.54	49.831
Total		100.000

Chiral



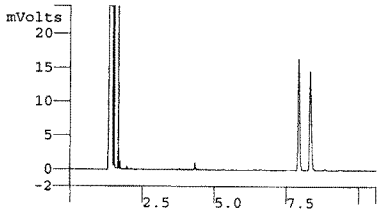
Index	Time [Min]	Area % [%]
1	11.83	9.154
2	16.57	90.846
Total		100.000

Table 2, Entry 13



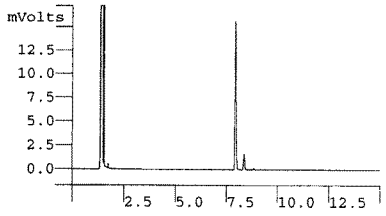
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 110 °C; **Carrier:** Helium, head pressure 25 psi; **Detection:** FID 250 °C.

Racemic



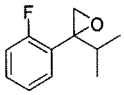
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		50.0133	7.916	62841
2		49.9867	8.311	62808
	Totals	**100.0000**		**125649**

Chiral



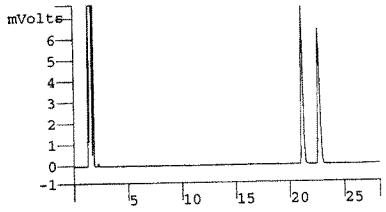
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		90.3992	7.914	61745
2		9.6008	8.352	6558
	Totals	**100.0000**		**68303**

Table 2, Entry 14



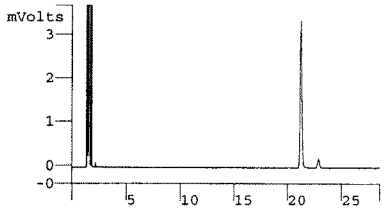
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 80 °C; **Carrier:** Helium, head pressure 25 psi; **Detection:** FID 250 °C.

Racemic



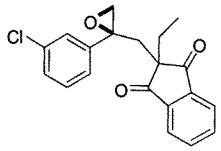
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		50.0358	21.103	91032
2		49.9642	22.608	90902
	Totals	**100.0000**		**181934**

Chiral



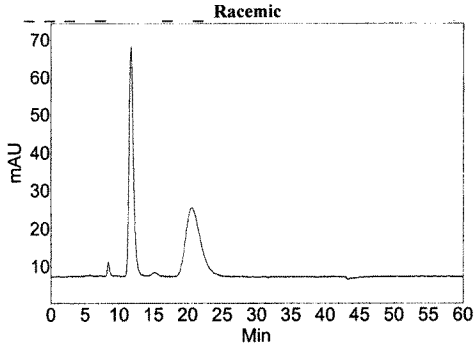
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		94.1314	21.194	38159
2		5.8686	22.849	2379
	Totals	**100.0000**		**40538**

Table 2, Entry 15



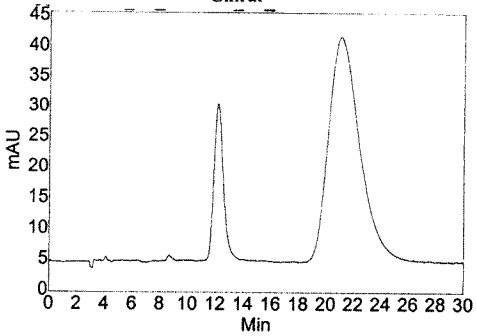
HPLC Cond.: Column: Chiralcel OD (Column No. OD00CE-DL010), Daicel Chemical Industries, Ltd.
Eluent: Hexanes/IPA (80/20); **Flow rate:** 1.0 mL/min; **Detection:** UV254 nm.

Racemic



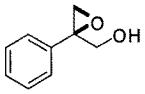
Index	Time [Min]	Area % [%]
1	11.73	49.945
2	20.54	50.055
Total		100.000

Chiral



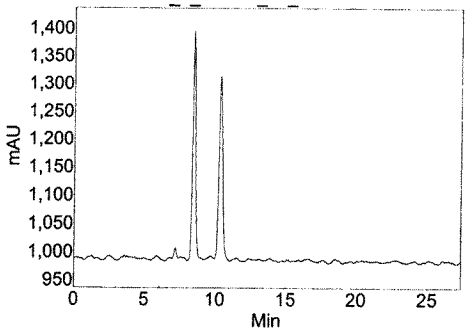
Index	Time [Min]	Area % [%]
1	12.14	17.183
2	21.02	82.817
Total		100.000

Table 2, Entry 16



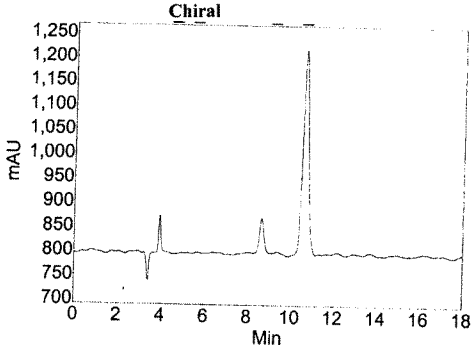
HPLC Cond.: Column: Chiralcel OD (Column No. OD00CE-DL010), Daicel Chemical Industries, Ltd.
Eluent: Hexanes/IPA (90/10); **Flow rate:** 1.0 mL/min; **Detection:** UV210 nm.

Racemic



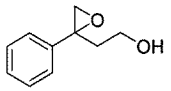
Index	Time [Min]	Area % [%]
1	8.44	50.566
2	10.34	49.434
Total		100.000

Chiral



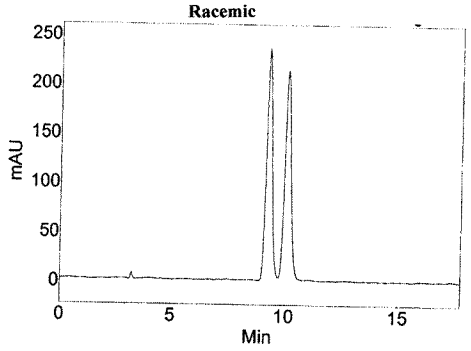
Index	Time [Min]	Area % [%]
2	8.63	11.632
1	10.60	88.368
Total		100.000

Table 2, Entry 17



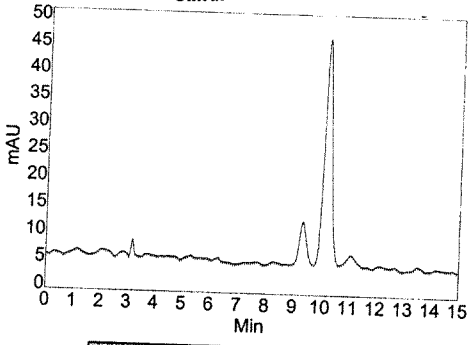
HPLC Cond.: Column: Chiralcel OD (Column No. OD00CE-DL010), Daicel Chemical Industries, Ltd.
Eluent: Hexanes/IPA (90/10); **Flow rate:** 1.0 mL/min; **Detection:** UV220 nm.

Racemic



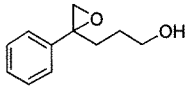
Index	Time [Min]	Area % [%]
1	9.26	50.055
2	10.08	49.945
Total		100.000

Chiral



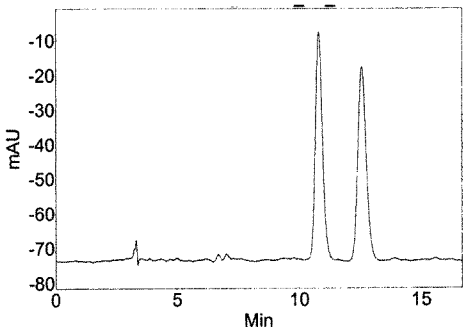
Index	Time [Min]	Area % [%]
1	9.33	14.067
2	10.14	85.933
Total		100.000

Table 2, Entry 18



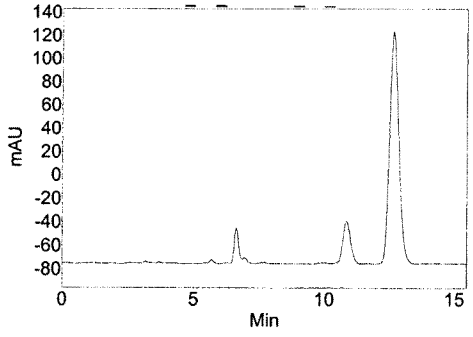
HPLC Cond.: Column: Chiralcel OD (Column No. OD00CE-DL010), Daicel Chemical Industries, Ltd.
Eluent: Hexanes/IPA (90/10); **Flow rate:** 1.0 mL/min; **Detection:** UV220 nm.

Racemic



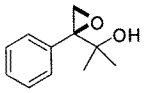
Index	Time [Min]	Area % [%]
1	10.81	49.646
2	12.58	50.354
Total		100.000

Chiral



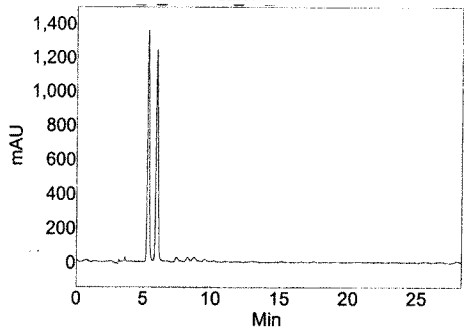
Index	Time [Min]	Area % [%]
1	10.86	13.048
2	12.65	86.952
Total		100.000

Table 2, Entry 19



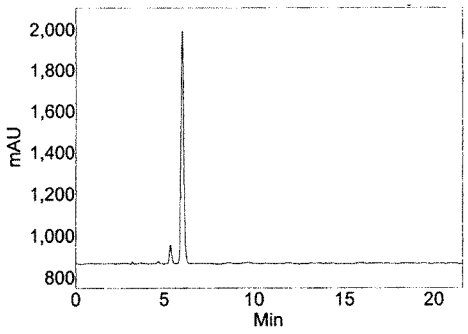
HPLC Cond.: Column: Chiralcel OD (Column No. OD00CE-DL010), Daicel Chemical Industries, Ltd.
Eluent: Hexanes/IPA (90/10); **Flow rate:** 1.0 mL/min; **Detection:** UV210 nm.

Racemic



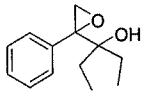
Index	Time [Min]	Area % [%]
1	5.36	49.675
2	6.00	50.325
Total		100.000

Chiral



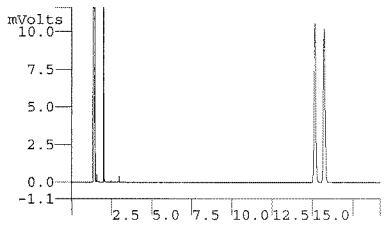
Index	Time [Min]	Area % [%]
1	5.35	6.279
2	6.01	93.721
Total		100.000

Table 2, Entry 20



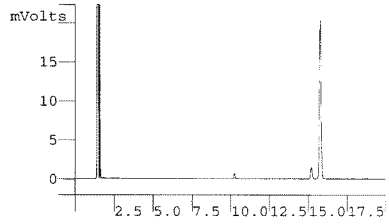
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 130 °C; **Carrier:** Helium, head pressure 25 psi; **Detection:** FID 250 °C.

Racemic



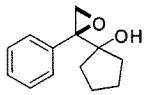
Peak No	Peak Name	Ret. Time (min)	Result ()	Area (counts)
1		15.146	49.9656	75665
2		15.719	50.0344	75769
	Tota		100.0000	151434

Chiral



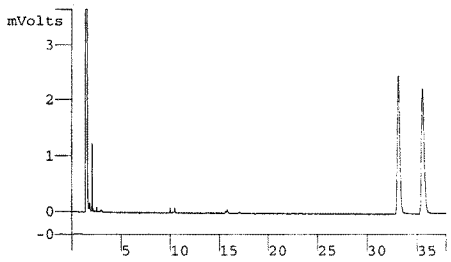
Peak No	Peak Name	Ret. Time (min)	Result ()	Area (counts)
1		15.189	6.6247	10795
2		15.734	93.3753	152151
	Tota		100.0000	162946

Table 2, Entry 21



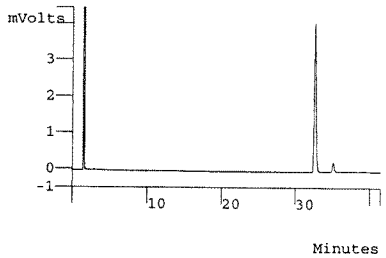
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 130 °C; **Carrier:** Helium, head pressure 25 psi; **Detection:** FID 250 °C.

Racemic



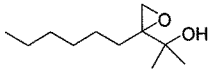
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		50.3469	33.088	42312
2		49.6530	35.494	41728
	Totals	**99.9999**		**84040**

Chiral



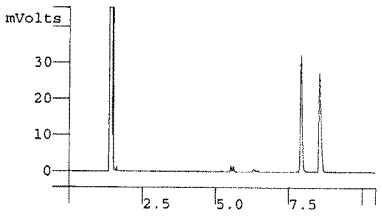
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		93.9829	32.524	69460
2		6.0171	35.061	4447
	Totals	**100.0000**		**73907**

Table 2, Entry 22



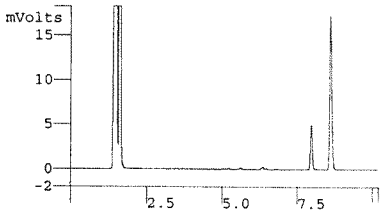
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 125 °C; **Carrier:** Helium, head pressure 25 psi; **Detection:** FID 250 °C.

Racemic



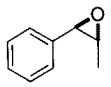
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		50.0562	7.894	136401
2		49.9438	8.526	136095
	Totals	**100.0000**		**272496**

Chiral



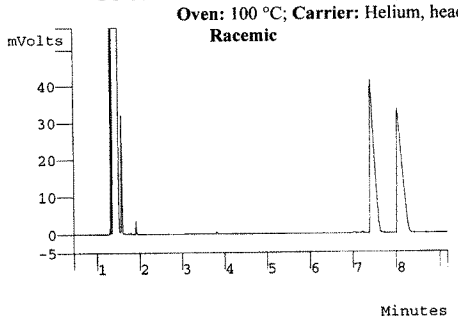
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		20.0045	7.959	20973
2		79.9955	8.574	83869
	Totals	**100.0000**		**104842**

Table 3, Entry 1



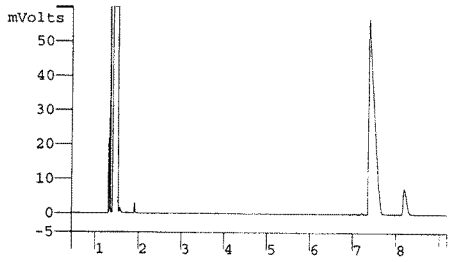
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 100 °C; **Carrier:** Helium, head pressure 25 psi; **Detection:** FID 250 °C.

Racemic



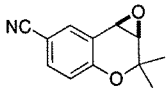
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		50.1725	7.404	276460
2		49.8275	8.029	274558
	Totals	**100.0000**		**551018**

Chiral



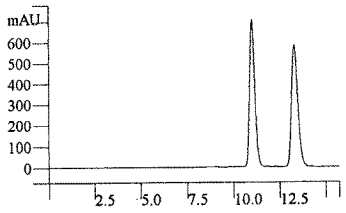
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		92.5831	7.364	488892
2		7.4169	8.189	39166
	Totals	**100.0000**		**528058**

Table 3, Entry 2



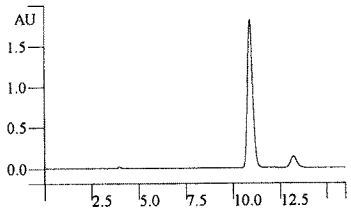
HPLC Cond.: Column: Chiralcel OD (Column No. OD00CE-DL010), Daicel Chemical Industries, Ltd.
Eluent: Hexanes/IPA (90/10); **Flow rate:** 1.0 mL/min; **Detection:** UV254 nm.

Racemic



Peak No	Ret. Time (min)	Result ()	Area (counts)
1	10.977	49.7986	7251947
2	13.266	50.2014	7310608
		100.0000	**14562555**

Chiral

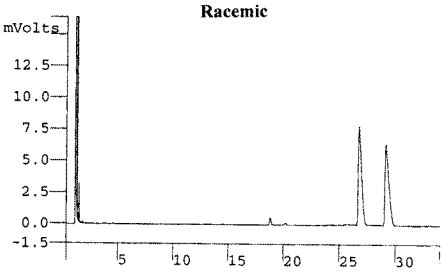


Peak No	Ret. Time (min)	Result ()	Area (counts)
1	10.861	92.1968	18571752
2	13.199	7.8032	1571838
		100.0000	**20143590**

Table 3, Entry 3

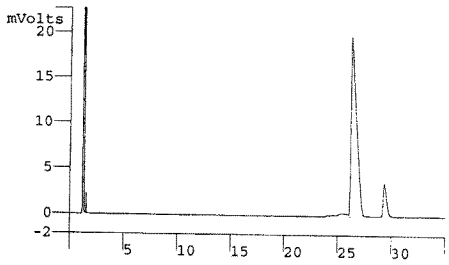
GC Cond.: Column: Chiraldex B-DM (Cat. No. 77023), Adv. Separation Technologies, Inc.
Oven: 110 °C; **Carrier:** Helium, head pressure 30 psi; **Detection:** FID 250 °C.

Racemic



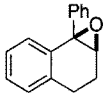
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		50.3459	26.722	153915
2		49.6541	29.106	151800
	Totals	**100.0000**		**305715**

Chiral



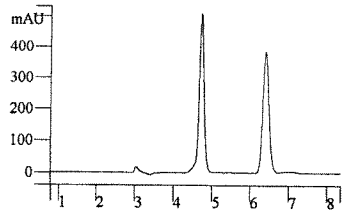
Peak No	Peak Name	Result ()	Ret. Time (min)	Area (counts)
1		90.0401	26.166	662100
2		9.9599	29.296	73239
	Totals	**100.0000**		**735339**

Table 3, Entry 4



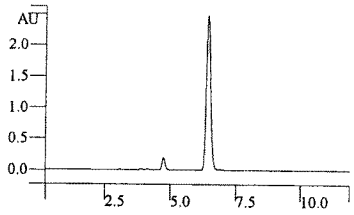
HPLC Cond.: Column: Chiralcel OD (Column No. OD00CE-DL010), Daicel Chemical Industries, Ltd.
Eluent: Hexanes/IPA (90/10); **Flow rate:** 1.0 mL/min; **Detection:** UV220 nm.

Racemic



Peak No	Ret. Time (min)	Result ()	Area (counts)
1	4.707	50.2704	2250342
2	6.378	49.7296	2226135
		100.0000	**4476477**

Chiral



Peak No	Ret. Time (min)	Result ()	Area (counts)
1	4.727	5.1522	816046
2	6.411	94.8478	15022684
		100.0000	**15838730**