Cu(I)-Catalyzed Diamination of Disubstituted Terminal Olefins: An Approach to Potent NK_1 Antagonist

Yuehong Wen, Baoguo Zhao, and Yian Shi*

Department of Chemistry
Colorado State University
Fort Collins, CO 80523

Supporting Information

Table of Contents

General methods	S-2
Diamination procedure	S-2
Deprotection procedure	S-2
Synthesis of **Sch 425078**	S-4
Characterization data of compounds **3**	S-7
NOE studies of compound **4a**	S-13
NMR spectra of compounds **3**, **4a**, **5a**, **6a**, **9-14**	S-14
The X-ray structure of compound **14**	S-64

General methods. All commercially reagents were used without further purification. Column chromatography was performed with silica gel (200-400 mesh). ^{1}H NMR was recorded on 300 or 400 MHz spectrometers at ambient temperature. ^{13}C NMR was recorded at 75 or 100 MHz spectrometers at ambient temperature. IR spectra were recorded on a FT-IR spectrometer. Melting points were uncorrected.

Olefins **1a**, **1f**, **1h**, **1i**, **1j**, and **1p** were purchased and used directly. Olefins **1b-1e, 1g**, and **1k-1n** were prepared from ketones using the Tebbe or Wittig reagents according to the reported procedures.[1] Olefin **1o** was prepared by following the reported procedure.[2]

(1) (a) Ohsugi, S.; Nishide, K.; Node, M. *Tetrahedron* **2003,** *59*, 1859. (b) Pine, S. H.; Shen, G. S.; Hoang, H. *Synthesis* **1991**, 165. (c) Tebbe, F. N.; Parshall, G. W.; Reddy, G. S. *J. Am. Chem. Soc.* **1978**, *100*, 3611.

(2) Beddow, J. E.; Davies, S. G.; Ling, K. B.; Roberts, P. M.; Russell, A. J.; Smith, A. D.; Thomson, J. E. *Org. Biomol. Chem.* **2007**, *5*, 2812.

Representative diamination procedure (Table 1, entry 1). To a 1.5 mL vial equipped with a stir bar was added CuCl (0.002 g, 0.02 mmol), triphenylphosphine (0.0052 g, 0.02 mmol), and $CDCl_3$ (0.3 mL). After the mixture was stirred at room temperature for 10 min, 2-phenylpropene (**1a**) (0.047 g, 0.4 mmol) was added. The reaction mixture was warmed to 65 ^{o}C using an oil bath with stirring, and di-*tert*-butyldiaziridinone (**2**) (0.136 g, 0.8 mmol) was added by syringe pump over 8 h. The reaction mixture was stirred at this temperature for an additional 1 h and purified by flash chromatography (silica gel, hexane:ether = 10:1, v/v) to give the diamination product **3a** as a white solid (0.105 g, 91%).

Removal of one *tert*-butyl group (Scheme 2). A mixture of compound **3a** (0.075 g, 0.26 mmol) and methanesulfonic acid (0.075 mL) in hexane (0.75 mL) was stirred at room temperature for 3.5 h. Water (7 mL) was then added. The mixture was extracted with chloroform (10 mL × 3), washed with brine (10 mL), dried (Na_2SO_4), filtered, concentrated, and purified by column chromatography (silica gel, hexane:ethyl acetate = 2:1, v/v) to give

compound **4a** as a white solid (0.060 g, 99%). mp 88-90 ^{o}C; IR (film) 3226, 1693 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38-7.31 (m, 4H), 7.29-7.22 (m, 1H), 4.92 (s, 1H), 3.48 (d, *J* = 8.8 Hz, 1H), 3.40 (d, *J* = 8.8 Hz, 1H), 1.61 (s, 3H), 1.32 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.4, 146.1, 128.6, 127.2, 124.8, 57.7, 56.3, 52.9, 28.2, 27.7; HRMS Calcd for $C_{14}H_{21}N_2O$ ($M+H^+$): 233.1648. Found: 233.1651.

Removal of both of the *tert*-butyl groups (Scheme 2). A mixture of compound **3a** (0.150 g, 0.52 mmol) and methanesulfonic acid (0.15 mL) in hexane (1.5 mL) was stirred at 65 ^{o}C for 3.5 h. Water (10 mL) was then added. The mixture was extracted with chloroform (20 mL × 3), washed with brine (10 mL), dried (Na_2SO_4), filtered, concentrated, and purified by column chromatography (silica gel, ethyl acetate then ethyl acetate/methanol = 20/1) to give compound **5a** as a white solid (0.078 g, 85%). mp 197-198 ^{o}C; IR (film) 3193, 1699 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.39-7.32 (m, 4H), 7.25-7.24 (m, 1H), 7.02 (s, 1H), 6.29 (s, 1H), 3.43 (d, *J* = 8.8 Hz, 1H), 3.26 (d, *J* = 8.4 Hz, 1H), 1.49 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 162.4, 147.1, 128.2, 126.6, 124.8, 59.6, 54.1, 28.7; Anal. Calcd for $C_{10}H_{12}N_2O$: C, 68.16; H, 6.86; N, 15.90. Found: C, 68.09; H, 6.84; N, 15.78.

Preparation of diamine 6a (Scheme 2). A mixture of compounds **3a** (0.116 g, 0.4 mmol) and conc. HCl (6.0 mL) was stirred at reflux for 30 h, washed with CH_2Cl_2 (10 mL x 3), concentrated under reduced pressure, diluted with water (5 mL), and adjusted to basic (pH >12) with 15% aqueous NaOH. The mixture was extracted with CH_2Cl_2 (10 mL × 3), washed with brine (10 mL), dried (Na_2SO_4), filtered, and concentrated to give diamine **6a** as a dark yellow oil (0.052 g, 87% yield). IR (film) 3355, 3287 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43 (d, *J* = 8.1 Hz, 2H), 7.35-7.30 (m, 2H), 7.23-7.18 (m, 1H), 2.91 (d, *J* = 12.3 Hz, 1H), 2.72 (d, *J* = 12.3 Hz, 1H), 1.41 (s, 3H), 1.34 (brs, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.1, 128.3, 126.4, 125.5, 56.1, 54.7, 28.8; HRMS Calcd for $C_9H_{14}N_2$ (M^+): 150.1154. Found: 150.1157.

Preparation of α-bromomethylstyrene (7). *N*-Bromosuccinimide (8.90 g, 50.0 mmol) was added to a solution of α-methylstyrene (10.4 mL, 80.0 mmol) in CCl_4 (5 mL), and the mixture was rapidly heated in an oil bath at 170 °C until the solids were dissolved. The reaction mixture was allowed to cool to room temperture and filtered to remove the precipitates. The filtrate was concentrated under reduced pressure and purified by flash chromatography (hexane) to give **7** as a colorless oil (5.0 g, 51%). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.59-7.56 (m, 2H), 7.45-7.42 (m, 3H), 5.63 (s, 1H), 5.56 (s, 1H), 4.45 (s, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.3, 137.6, 128.6, 128.4, 126.2, 117.3, 34.3.

Reed, S. F. *J. Org. Chem.* **1965**, *30*, 3258.

Preparation of olefin 9. To a stirred suspension of NaH (60% dispersion in mineral oil) (0.40 g, 10.0 mmol) in THF (20.0 mL) at 0 ºC was added a solution of (*R*)-1-(3,5-bis(trifluoromethyl)phenyl)ethanol (2.582 g, 10.0 mmol) in THF (10.0 mL). After stirring at room temperature for 30 min, a solution of α-bromomethylstyrene (1.97 g, 10.0 mmol) in THF (10.0 mL) was added. The resulting mixture was heated at reflux for 18 h, cooled to room temperature, filtered through Celite, concentrated, and purified by flash chromatography (silical gel, hexane:ethyl acetate = 100:1, v/v) to afford compound **9** as a colorless oil (2.90 g, 77%). $[\alpha]^{20}_D$ = +43.8 (*c* 1.1, CH_2Cl_2); IR (film) 1279, 1177, 1134 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (d, *J* = 9.6 Hz, 3H), 7.38-7.35 (m, 2H), 7.31-7.24 (m, 3H), 5.49 (s, 1H), 5.27 (d, *J* = 1.2 Hz, 1H), 4.60 (q, *J* = 6.4 Hz, 1H), 4.31 (d, *J* = 12.8 Hz, 1H), 4.20 (d, *J* = 12.4 Hz, 1H), 1.41 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.8, 144.2, 138.6, 132.4, 132.1, 131.8, 131.4, 128.6, 128.1, 126.6, 126.2, 124.9, 122.1, 121.7, 121.6, 115.0, 76.2, 71.1, 24.0.

Diamination of compound 9. To a 1.5 mL vial equipped with a stir bar was added CuCl (0.004 g, 0.04 mmol), triphenylphosphite (0.0124 g, 0.04 mmol), and $CDCl_3$ (0.3 mL). After the mixture was stirred at room temperature for 10 min, compound **9** (0.075 g, 0.20 mmol) was added. The reaction mixture was warmed to 65 °C using an oil bath with stirring, and di-*tert*-butyldiaziridinone (**2**) (0.068 g, 0.40 mmol) was added by syringe pump over 8 h. The

reaction mixture was stirred at this temperature for an additional 2 h and purified by flash chromatography (silica gel, hexane:ether = 10:1, v/v) to give compound **10** as a sticky colorless oil (0.038 g, 35%) and more polar compound **11** as a sticky colorless oil (0.033 g, 30%).

10: $[\alpha]^{20}_{D}$ = +19.8 (*c* 2.5, CH_2Cl_2); IR (film) 1682 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.87 (s, 3H), 7.56 (d, *J* = 8.1 Hz, 2H), 7.42-7.32 (m, 3H), 4.77 (q, *J* = 6.6 Hz, 1H), 4.16 (d, *J* = 9.0 Hz, 1H), 3.80 (d, *J* = 9.0 Hz, 1H), 3.73 (d, *J* = 8.7 Hz, 1H), 3.21 (d, *J* = 8.7 Hz, 1H), 1.64 (d, *J* = 6.6 Hz, 3H), 1.37 (s, 9H), 1.20 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.4, 146.1, 145.2, 132.8, 132.4, 132.0, 131.5, 128.8, 128.4, 127.6, 126.9, 126.3, 125.2, 122.1, 122.0, 121.6, 78.4, 72.0, 64.0, 55.8, 54.5, 53.2, 29.8, 27.4, 24.0; HRMS Calcd for $C_{28}H_{35}F_6N_2O_2$ ($M+H^+$): 545.2597. Found: 545.2601.

11: $[\alpha]^{20}_{D}$ = +31.1 (*c* 2.85, CH_2Cl_2); IR (film) 1685 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.90-7.89 (m, 3H), 7.45 (d, *J* = 6.9 Hz, 2H), 7.38-7.27 (m, 3H), 4.72 (q, *J* = 6.3 Hz, 1H), 4.03 (d, *J* = 9.0 Hz, 1H), 3.98 (d, *J*= 9.3 Hz, 1H), 3.71 (d, *J* = 8.7 Hz, 1H), 3.21 (d, *J* = 8.7 Hz, 1H), 1.62 (d, *J* = 6.6 Hz, 3H), 1.43 (s, 9H), 1.24 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.5, 146.4, 145.3, 132.8, 132.4, 131.9, 131.5, 128.4, 127.6, 126.4, 125.2, 121.9, 121.88, 121.8, 121.6, 78.3, 72.3, 63.9, 55.8, 54.8, 53.1, 29.8, 27.6, 23.7; HRMS Calcd for $C_{28}H_{35}F_6N_2O_2$ ($M+H^+$): 545.2597. Found: 545.2601.

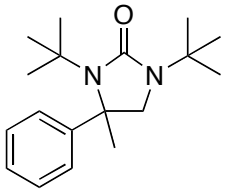
Preparation of compound 12. A mixture of compound **10** (0.140 g, 0.26 mmol) and CF_3CO_2H (0.52 mL) in a 3 mL vial was stirred at 80 ºC for 2 h, concentrated, and purified by flash chromatography (silica gel, ethyl acetate) to give compound **12** as a white solid (0.083 g, 74%). $[\alpha]^{20}_{D}$ = -52.9 (*c* 1.2, CH_2Cl_2); mp 131-132 ^oC; IR (film) 3200, 1713 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.80 (s, 1H), 7.50 (s, 2H), 7.44-7.34 (m, 3H), 7.30-7.28 (m, 2H), 5.60 (s, 1H), 4.90 (s, 1H), 4.52 (q, *J* = 6.6 Hz, 1H), 3.78 (d, *J* = 8.7 Hz, 1H), 3.68 (d, *J* = 8.4 Hz, 1H), 3.58 (d, *J* = 9.0 Hz, 1H), 3.51 (d, *J* = 9.0 Hz, 1H), 1.43 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.9, 146.1, 142.3, 132.6, 132.2, 131.8, 131.3, 128.9, 127.9, 126.2, 125.3, 125.1, 125.0, 121.8, 121.5,78.0, 74.8, 63.4, 50.9, 24.1; Anal. Calcd for $C_{20}H_{18}F_6N_2O_2$: C, 55.56; H, 4.20; N, 6.48. Found: C, 55.63; H, 4.42; N, 6.33; HRMS Calcd for $C_{20}H_{19}F_6N_2O_2$

($M+H^+$): 433.1345. Found: 433.1347.

Preparation of compound 13. A mixture of compound **10** (0.168 g, 0.31 mmol) and CF_3CO_2H (0.6 mL) in a 3 mL vial was stirred at rt for 1 h, concentrated, and purified by flash chromatography (silica gel, ethyl acetate:hexane = 1:2, v/v) to give compound **13** as a sticky colorless oil (0.142 g, 94%). $[\alpha]^{20}_D$ = -31.6 (*c* 3.8, CH_2Cl_2); IR (film) 1694 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.45 (s, 2H), 7.40-7.29 (m, 3H), 7.26-7.24 (m, 2H), 5.26 (brs, 1H), 4.46 (q, *J* = 6.6 Hz, 1H), 3.66 (d, *J* = 8.4 Hz, 1H), 3.57 (d, *J* = 8.4 Hz, 1H), 3.49 (d, *J* = 8.7 Hz, 1H), 3.35 (d, *J* = 9.0 Hz, 1H), 1.36 (d, *J* = 6.3 Hz, 3H), 1.33 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.2, 146.2, 142.6, 132.6, 132.2, 131.7, 131.3, 128.8, 127.7, 126.3, 126.2, 125.1, 121.8, 121.7, 121.5, 78.0, 74.7, 59.2, 53.4, 53.3, 27.7, 24.2; HRMS Calcd for $C_{24}H_{27}F_6N_2O_2$ ($M+H^+$): 489.1971. Found: 489.1979.

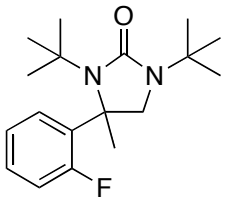
Preparation of compound 14. To a solution of **13** (0.090 g, 0.184 mmol) in anhydrous THF (2 mL) at 0 °C under Ar, was added a solution of *n*-BuLi in pentane (2.0 M, 0.14 mL, 0.28 mmol). After the mixture was stirred at 0 °C for 15 min, a solution of benzoyl chloride (0.042 mL, 0.051 g, 0.36 mmol) in THF (1 mL) was added. Upon stirring at 0 °C for an additional 2 h, the reaction mixture was quenched with saturated aqueous ammonium chloride solution, extracted with CH_2Cl_2 (15 mL × 3), washed with saturated aqueous $NaHCO_3$ and brine, dried (Na_2SO_4), filtered, concentrated, and purified by flash chromatography (silica gel, ethyl acetate:hexane = 1:8, v/v) to give compound **14** as a white solid (0.068 g, 62%). $[\alpha]^{20}_D$ = -15.1 (*c* 1.45, CH_2Cl_2); mp 162-163 °C; IR (film) 1727, 1668 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.10-8.08 (m, 1H), 7.74 (s, 1H), 7.66 (s, 2H), 7.48-7.43 (m, 2H), 7.41-7.33 (m, 4H), 7.31-7.23 (m, 3H), 4.65 (q, *J* = 6.4 Hz, 1H), 4.21 (d, *J* = 9.2 Hz, 1H), 4.18 (d, *J* = 9.2 Hz, 1H), 4.07 (d, *J* = 9.2 Hz, 1H), 3.50 (d, *J* = 8.8 Hz, 1H), 1.53 (d, *J* = 6.4 Hz, 3H), 1.34 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.3, 154.4, 145.9, 141.3, 135.8, 133.9, 132.1, 131.8, 131.4, 130.3, 128.9, 128.6, 128.5, 128.0, 127.7, 126.3, 125.0, 124.7, 121.9, 78.1, 69.8, 63.7, 54.3, 52.9, 27.5, 23.8; HRMS Calcd for $C_{31}H_{31}F_6N_2O_3$ ($M+H^+$): 593.2233. Found: 593.2241.

Table 1, Entry 1



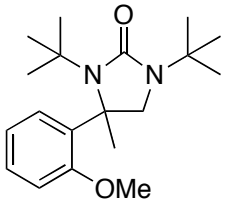
White solid; mp 74-75 °C; IR (film) 1688 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.47-7.45 (m, 2H), 7.33-7.29 (m, 2H), 7.24-7.23 (m, 1H), 3.16 (d, *J* = 8.8 Hz, 1H), 3.06 (d, *J* = 8.8 Hz, 1H), 1.80 (s, 3H), 1.31 (s, 9H), 1.19 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.5, 147.8, 128.3, 127.1, 126.3, 61.3, 60.3, 55.6, 53.0, 29.8, 27.5, 24.3; Anal. Calcd for $C_{18}H_{28}N_2O$: C, 74.96; H, 9.78; N, 9.71. Found: C, 75.19; H, 9.56; N, 9.94.

Table 1, Entry 2



White solid; mp 110-111 °C; IR (film) 1689 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.30-7.26 (m, 1H), 7.20-7.18 (m, 1H), 7.03-6.93 (m, 2H), 3.33 (d, *J* = 8.4 Hz, 1H), 3.01 (dd, *J* = 8.4, 2.8 Hz, 1H), 1.75 (s, 3H), 1.25 (s, 9H), 1.12 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.5, 159.9 (*J* = 29.2 Hz), 133.8 (*J* = 7.3 Hz), 129.5 (*J* = 9.1 Hz), 128.3 (*J* = 3.6 Hz), 123.9 (*J* = 2.7 Hz), 117.1 (*J* = 21.9 Hz), 59.5, 57.7, 55.4, 53.2, 29.6, 27.6, 26.1; Anal. Calcd for $C_{18}H_{27}FN_2O$: C, 70.55; H, 8.88; N, 9.14. Found: C, 70.33; H, 8.65; N, 9.06.

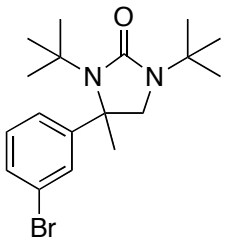
Table 1, Entry 3



White solid; mp 56-57 °C; IR (film) 1681 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.32-7.26 (m, 2H), 6.93-6.88 (m, 2H), 3.83 (s, 3H), 3.58 (d, *J* = 8.1 Hz, 1H), 2.97 (d, *J* = 7.5 Hz, 1H), 1.83

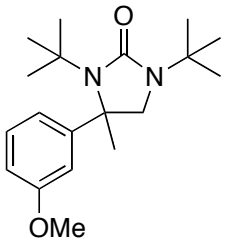
(s, 3H), 1.37 (s, 9H), 1.18 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.1, 158.0, 133.6, 128.9, 127.6, 120.0, 111.3, 59.6, 56.4, 55.0, 54.5, 53.0, 29.5, 27.7, 27.4; Anal. Calcd for $C_{19}H_{30}N_2O_2$: C, 71.66; H, 9.50; N, 8.80. Found: C, 71.45; H, 9.61; N, 8.83.

Table 1, Entry 4



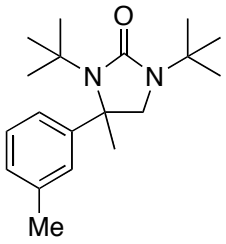
White solid; mp 90-91 °C; IR (film) 1690 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60-7.59 (m, 1H), 7.44-7.36 (m, 2H), 7.23-7.18 (m, 1H), 3.12 (d, *J* = 8.4 Hz, 1H), 3.08 (d, *J* = 8.7 Hz, 1H), 1.80 (s, 3H), 1.32 (s, 9H), 1.21 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.3, 150.4, 130.3, 130.0, 129.3, 125.0, 122.6, 61.2, 60.0, 55.7, 53.1, 29.8, 27.5, 24.5; Anal. Calcd for $C_{18}H_{27}BrN_2O$: C, 58.86; H, 7.41; N, 7.63. Found: C, 58.77; H, 7.50; N, 7.54.

Table 1, Entry 5



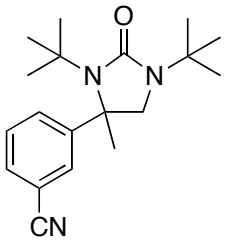
White solid; mp 113-115 °C; IR (film) 1683 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25-7.21 (m, 1H), 7.05-7.01 (m, 2H), 6.78-6.76 (m, 1H), 3.79 (s, 3H), 3.16 (d, *J* = 8.4 Hz, 1H), 3.06 (d, *J* = 8.4 Hz, 1H), 1.79 (s, 3H), 1.31 (s, 9H), 1.21 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.5, 159.6, 149.7, 129.4, 118.7, 112.6, 112.0, 61.4, 60.2, 55.6, 55.3, 53.1, 29.7, 27.5, 24.6; Anal. Calcd for $C_{19}H_{30}N_2O_2$: C, 71.66; H, 9.50; N, 8.80. Found: C, 72.02; H, 9.27; N, 8.97.

Table 1, Entry 6



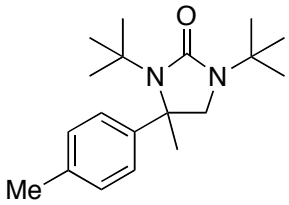
Colorless oil; IR (film) 1690 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.30-7.28 (m, 2H), 7.25-7.19 (m, 1H), 7.08-7.05 (m, 1H), 3.18 (d, *J* = 8.4 Hz, 1H), 3.08 (d, *J* = 8.4 Hz, 1H), 2.36 (s, 3H), 1.82 (s, 3H), 1.34 (s, 9H), 1.22 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.5, 147.8, 137.8, 128.2, 127.8, 126.9, 123.4, 61.3, 60.2, 55.5, 53.0, 29.8, 27.5, 24.4, 21.7; Anal. Calcd for $C_{19}H_{30}N_2O$: C, 75.45; H, 10.00; N, 9.26. Found: C, 75.58; H, 9.89; N, 9.09.

Table 1, Entry 7



White solid; mp 175-176 ^oC; IR (film) 1686 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.72-7.67 (m, 2H), 7.50-7.48 (m, 1H), 7.42-7.38 (m, 1H), 3.05-3.02 (m, 2H), 1.76 (s, 3H), 1.24 (s, 9H), 1.11 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.1, 149.6, 131.0, 129.6, 129.3, 118.8, 112.5, 61.1, 59.8, 55.8, 53.2, 29.9, 27.5, 24.3; Anal. Calcd for $C_{19}H_{27}N_3O$: C, 72.81; H, 8.68; N, 13.41. Found: C, 73.15; H, 8.61; N, 13.66.

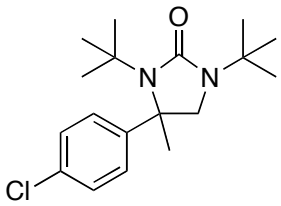
Table 1, Entry 8



Colorless oil; IR (film) 1690 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.34 (d, *J* = 8.0 Hz, 2H), 7.11 (d, *J* = 8.4 Hz, 2H), 3.14 (d, *J* = 8.4 Hz, 1H), 3.04 (d, *J* = 8.0 Hz, 1H), 2.32 (s, 3H), 1.78 (s,

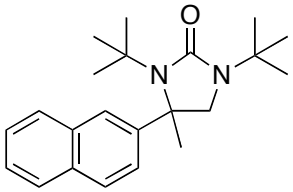
3H), 1.31 (s, 9H), 1.19 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.6, 144.8, 136.8, 129.0, 126.2, 61.1, 60.3, 55.5, 53.0, 29.8, 27.5, 24.4, 21.1; Anal. Calcd for $C_{19}H_{30}N_2O$: C, 75.45; H, 10.00; N, 9.26. Found: C, 75.18; H, 9.89; N, 9.06.

Table 1, Entry 9



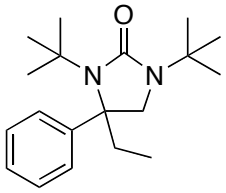
Colorless oil; IR (film) 1689 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, J = 8.4 Hz, 2H), 7.26 (d, J = 8.8 Hz, 2H), 3.08 (d, J = 8.4 Hz, 1H), 3.04 (d, J = 8.8 Hz, 1H), 1.77 (s, 3H), 1.28 (s, 9H), 1.17 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 160.3, 146.4, 133.0, 128.5, 127.7, 61.0, 60.0, 55.6, 53.0, 29.8, 27.5, 24.4; Anal. Calcd for $C_{18}H_{27}ClN_2O$: C, 66.96; H, 8.43; N, 8.68. Found: C, 66.94; H, 8.52; N, 8.69.

Table 1, Entry 10



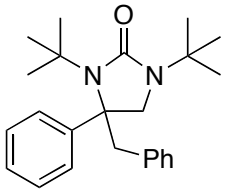
White solid; mp 103-104 ^oC; IR (film) 1689 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88-7.81 (m, 4H), 7.75-7.72 (m, 1H), 7.52-7.49 (m, 2H), 3.28 (d, J = 8.4 Hz, 1H), 3.13 (d, J = 8.7 Hz, 1H), 1.98 (s, 3H), 1.38 (s, 9H), 1.26 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.6, 145.0, 133.1, 132.5, 128.2, 128.16, 127.7, 126.5, 126,2, 125.2, 124.0, 61.6, 59.6, 55.7, 53.1, 29.8, 27.6, 24.5; Anal. Calcd for $C_{22}H_{30}N_2O$: C, 78.06; H, 8.93; N, 8.28. Found: C, 78.15; H, 8.70; N, 8.14.

Table 1, Entry 11



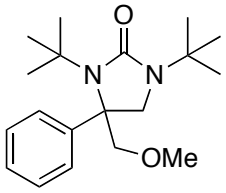
Colorless oil; IR (film) 1685 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.49-7.46 (m, 2H), 7.36-7.29 (m, 2H), 7.27-7.22 (m, 1H), 3.30 (d, *J* = 8.7 Hz, 1H), 3.25 (d, *J* = 9.0 Hz, 1H), 2.40-2.28 (m, 1H), 2.13-2.01 (m, 1H), 1.36 (s, 9H), 1.22 (s, 9H), 1.14 (t, *J* = 7.2, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.7, 148.8, 128.3, 127.1, 126.3, 63.1, 57.1, 55.6, 53.1, 31.2, 29.4, 27.6, 8.6; HRMS Calcd for $C_{19}H_{31}N_2O$ ($M+H^+$): 303.2431. Found: 303.2435.

Table 1, Entry 12



White solid; mp 115-117 °C; IR (film) 1684 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.65-7.63 (m, 2H), 7.43-7.33 (m, 4H), 7.31-7.23 (m, 4H), 3.61 (s, 2H), 3.53 (d, *J* = 9.0 Hz, 1H), 3.31 (d, *J* = 9.0 Hz, 1H), 1.28 (s, 9H), 1.16 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.1, 148.3, 137.4, 130.6, 128.4, 128.3, 127.4, 127.0, 126.7, 63.3, 56.5, 56.1, 52.8, 43.5, 29.8, 27.3; Anal. Calcd for $C_{24}H_{32}N_2O$: C, 79.08; H, 8.85; N, 7.68. Found: C, 78.87; H, 8.61; N, 7.40.

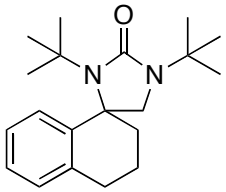
Table 1, Entry 13



Colorless oil; IR (film) 1690 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.43-7.40 (m, 2H), 7.25-7.22 (m, 2H), 7.18-7.16 (m, 1H), 4.02 (d, *J* = 9.2 Hz, 1H), 3.67 (d, *J* = 9.2 Hz, 1H), 3.61 (d, *J* = 9.2 Hz, 1H), 3.41 (s, 3H), 3.00 (d, *J* = 9.2 Hz, 1H), 1.26 (s, 9H), 1.09 (s, 9H); ^{13}C NMR

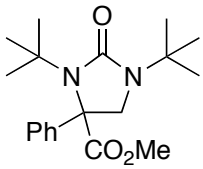
(100 MHz, $CDCl_3$) δ 160.5, 145.5, 128.2, 127.3, 126.8, 75.3, 64.1, 59.2, 55.7, 53.9, 53.1, 29.8, 27.6; Anal. Calcd for $C_{19}H_{30}N_2O_2$: C, 71.66; H, 9.50; N, 8.80. Found: C, 71.42; H, 9.40; N, 8.61.

Table 1, Entry 14



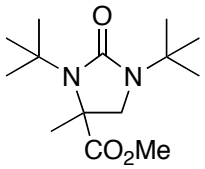
White solid; mp 124-125 °C; IR (film) 1688 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.7 (d, J = 7.8 Hz, 1H), 7.28-7.12 (m, 2H), 7.05-7.02 (m, 1H), 3.35 (d, J = 8.7 Hz, 1H), 3.11 (d, J = 8.7 Hz, 1H), 2.78-2.76 (m, 2H), 2.19-2.16 (m, 2H), 2.03-1.98 (m, 1H), 1.83-1.76 (m, 1H), 1.36 (s, 9H), 1.25 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.8, 142.7, 137.6, 128.8, 128.3, 1226.8, 126.3, 61.7, 58.3, 55.2, 53.0, 31.9, 29.9, 29.8, 27.5, 21.4; Anal. Calcd for $C_{20}H_{30}N_2O$: C, 76.39; H, 9.62; N, 8.91. Found: C, 76.34; H, 9.46; N, 8.88.

Table 1, Entry 15



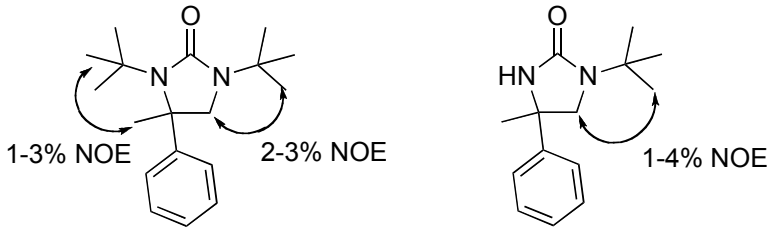
Yellow oil; IR (film) 1751, 1697 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.53-7.50 (m, 2H), 7.34-7.31 (m, 3H), 3.91 (s, 3H), 3.85 (d, J = 9.0 Hz, 1H), 3.38 (d, J = 9.0 Hz, 1H), 1.33 (s, 9H), 1.24 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.8, 159.8, 142.4, 128.3, 128.0, 127.3, 69.2, 57.7, 56.6, 53.4, 52.6, 29.0, 27.3; Anal. Calcd for $C_{19}H_{28}N_2O_3$: C, 68.65; H, 8.49; N, 8.43. Found: C, 68.90; H, 8.28; N, 8.17.

Table 1, Entry 16



White solid; mp 66-68 ^{o}C; IR (film) 1743, 1697 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.74 (s, 3H), 3.31 (d, J = 8.1 Hz, 1H), 2.99 (d, J = 7.8 Hz, 1H), 1.57 (s, 3H), 1.34 (s, 9H), 1.29 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 175.5, 159.4, 61.8, 55.2, 54.1, 53.2, 52.6, 28.8, 27.4, 23.7; HRMS Calcd for $C_{14}H_{27}N_2O_3$ ($M+H^+$): 271.2016. Found: 271.2021.

NOE studies of compound 4a.



STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
File: wen-1-30AH
INOVA-500 "epoxide"

 Pulse 31.0 degrees
 Acq. time 2.291 sec
 Width 6982.6 Hz
 8 repetitions
OBSERVE H1, 400.1063260 MHz
DATA PROCESSING
 Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 23 sec

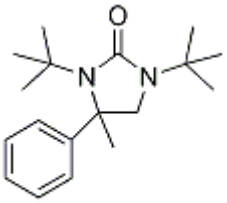
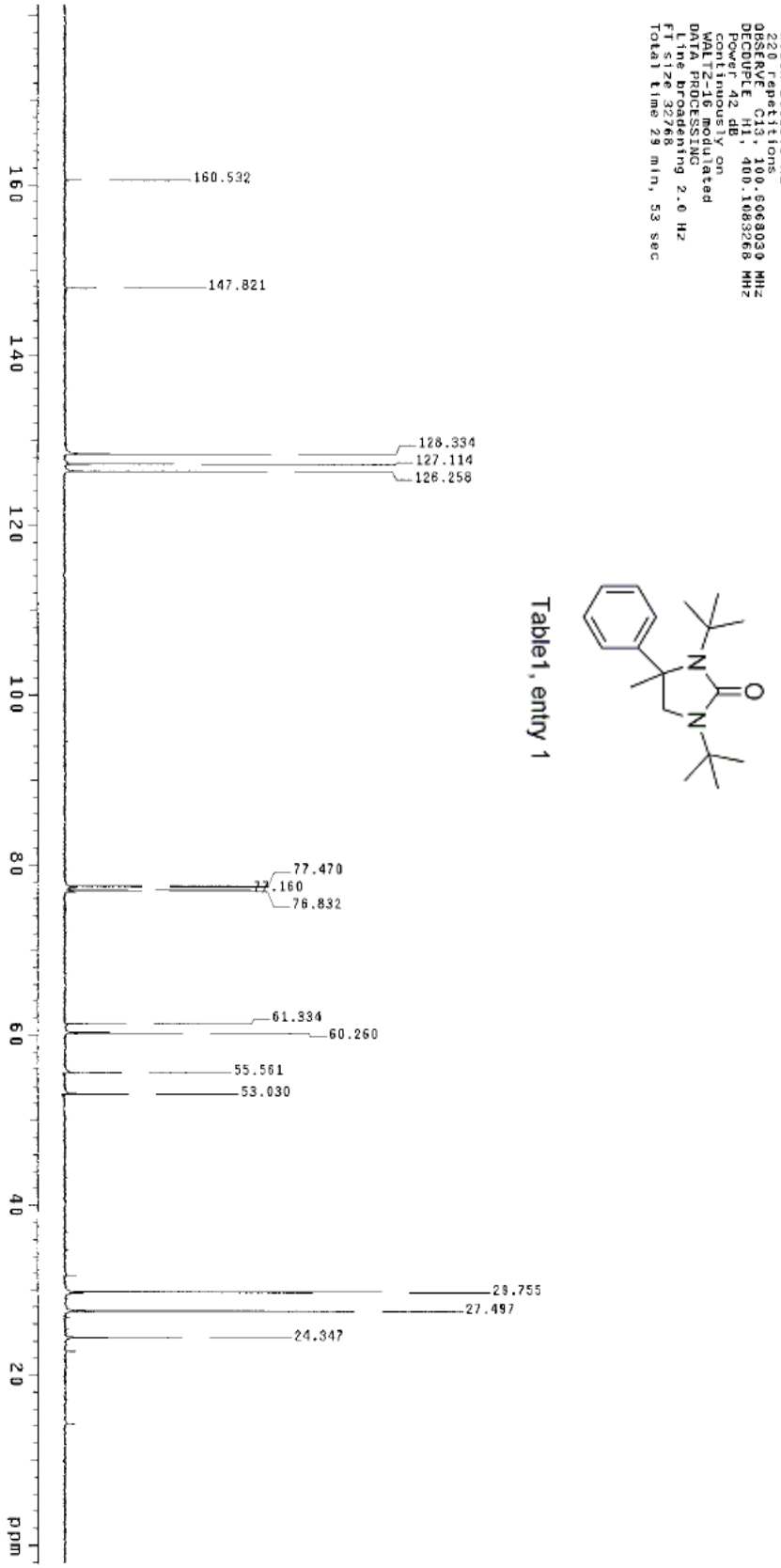


Table1, entry 1


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-1-30AC
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.555 sec
Width 30018.8 Hz
220 repetitions
OBSERVE C13, 100.6068030 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 29 min, 53 sec
160.532
147.821
128.334
127.114
126.258
77.470
77.160
76.832
61.934
60.260
55.561
53.030
28.755
27.497
24.347
160
140
120
100
80
60
40
20
ppm
Table1, entry 1
N
O
N

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-18AHH
INOVA-500 "epoxide"

Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
8 repetitions
OBSERVE H1, 400.1063260 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 23 sec

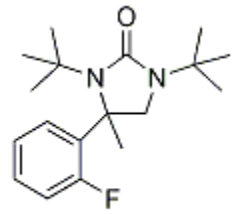
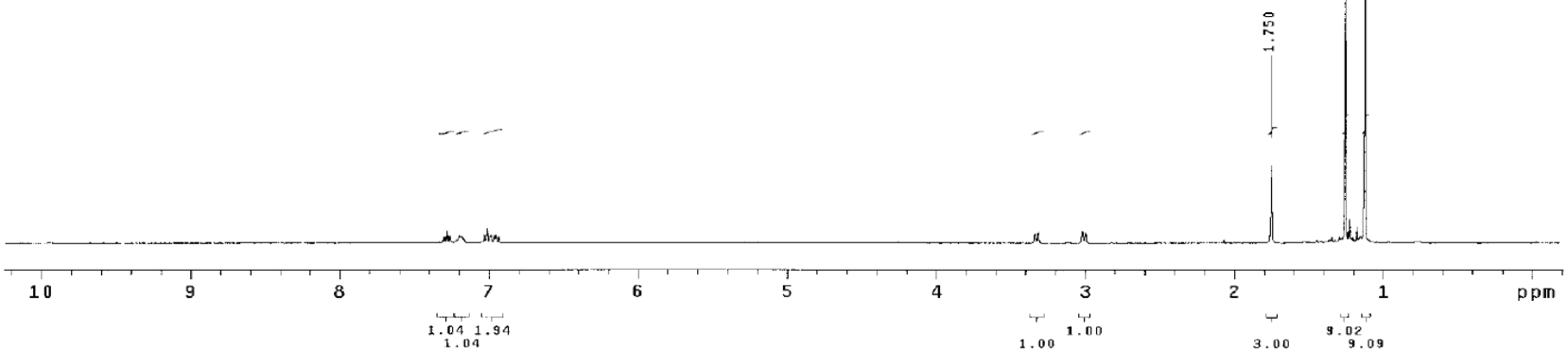
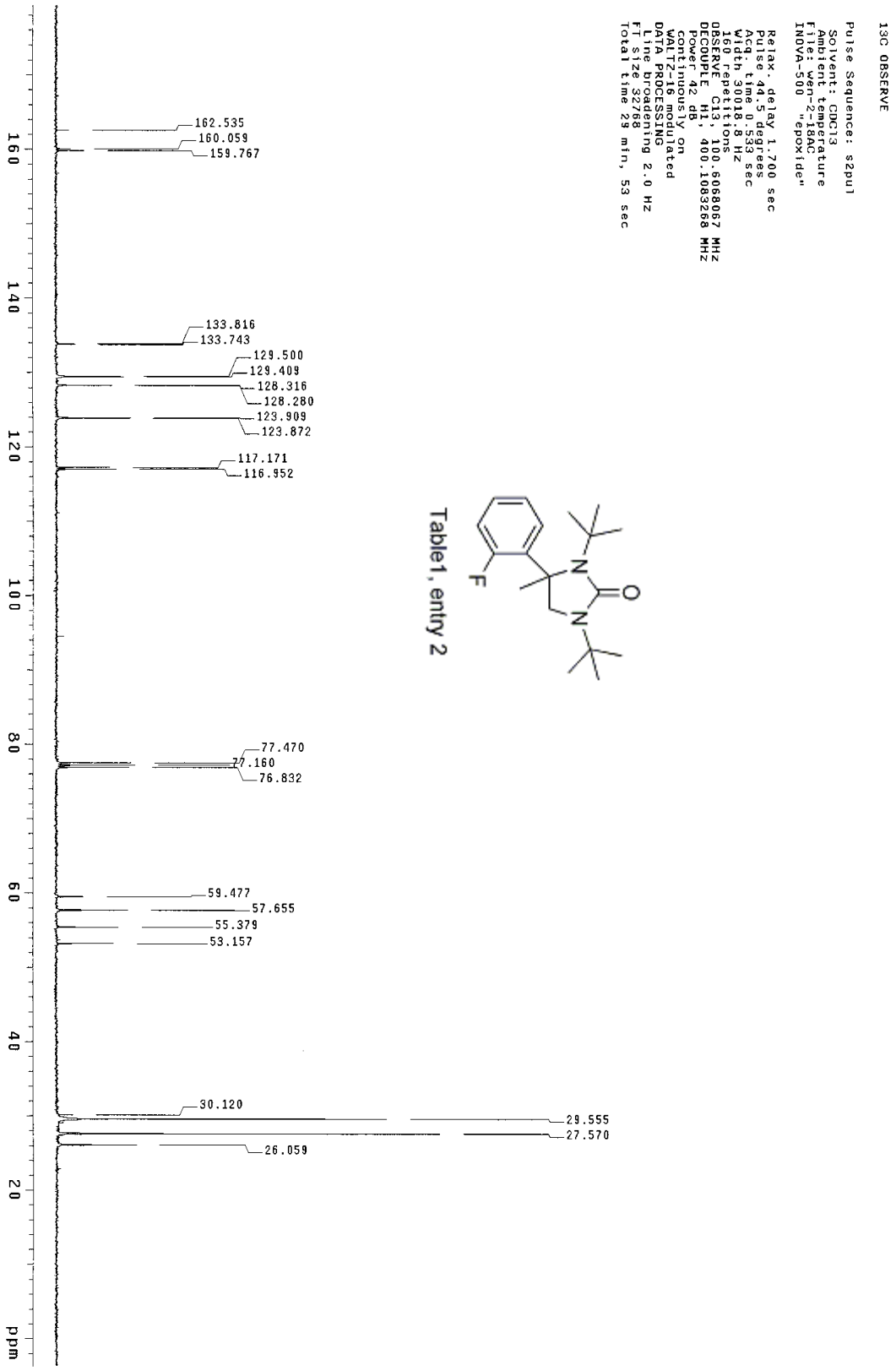


Table1, entry 2




13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-18AC
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, 100.6068067 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 29 min, 53 sec
Table1, entry 2
F
N
O
N
162.535
160.059
159.767
133.816
133.743
129.500
129.409
128.316
128.280
123.909
123.872
117.171
116.952
77.470
77.160
76.832
59.477
57.655
55.379
53.157
30.120
29.555
27.570
26.059
160
140
120
100
80
60
40
20
ppm

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
File: wen-2-22AH
INOVA-500 "epoxide"

 Relax. delay 1.000 sec
 Pulse 34.0 degrees
 Acq. time 2.732 sec
 Width 6000.6 Hz
 8 repetitions
OBSERVE   H1, 299.9533661 MHz
DATA PROCESSING
 Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 37 sec

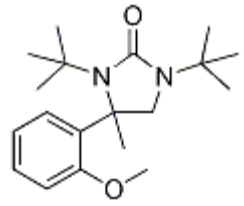


Table 1, entry 3

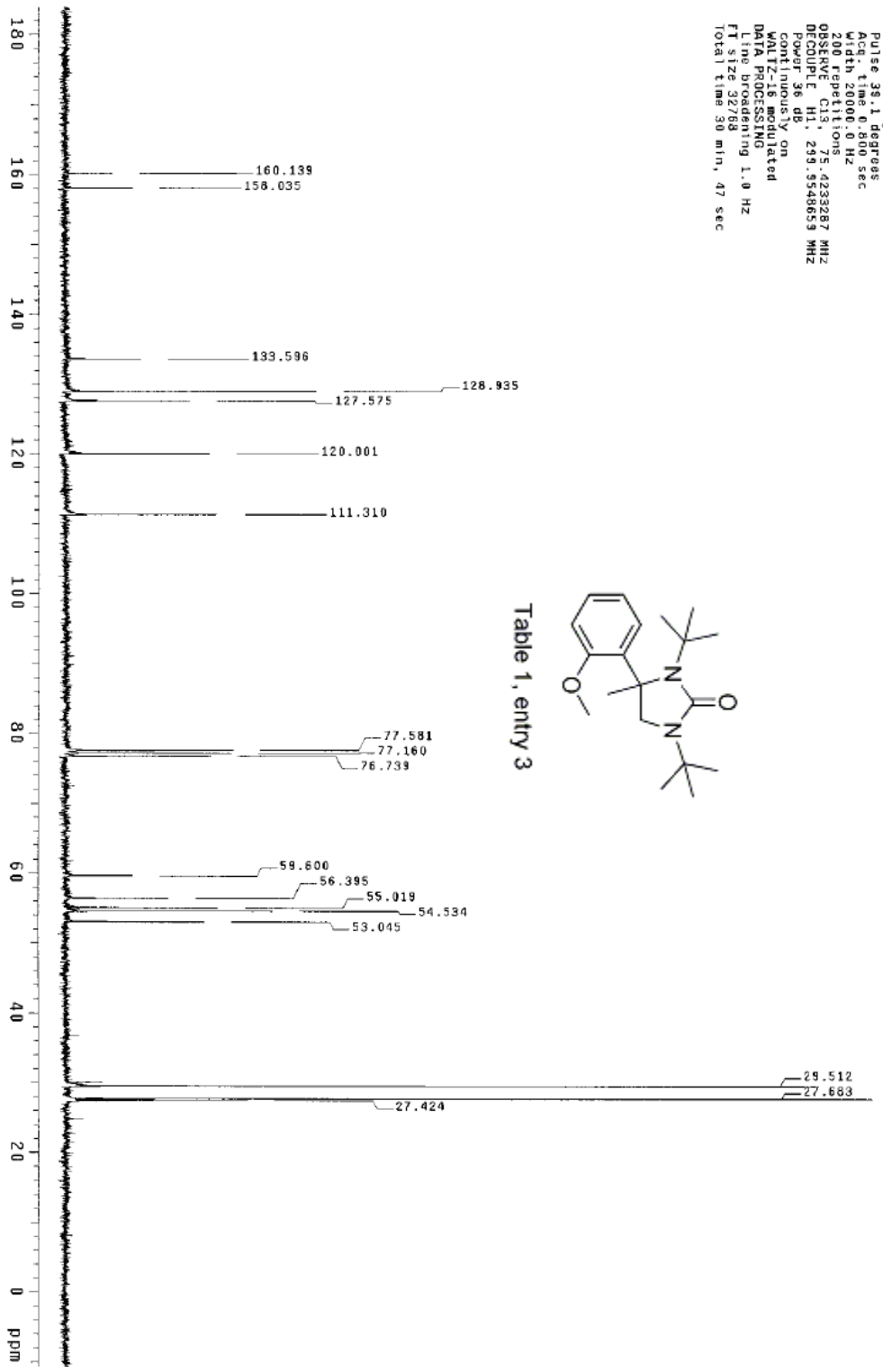


Table 1, entry 3

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-17AH
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
8 repetitions
OBSERVE H1, 299.9533661 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 37 sec

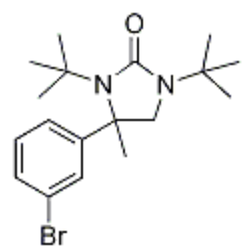
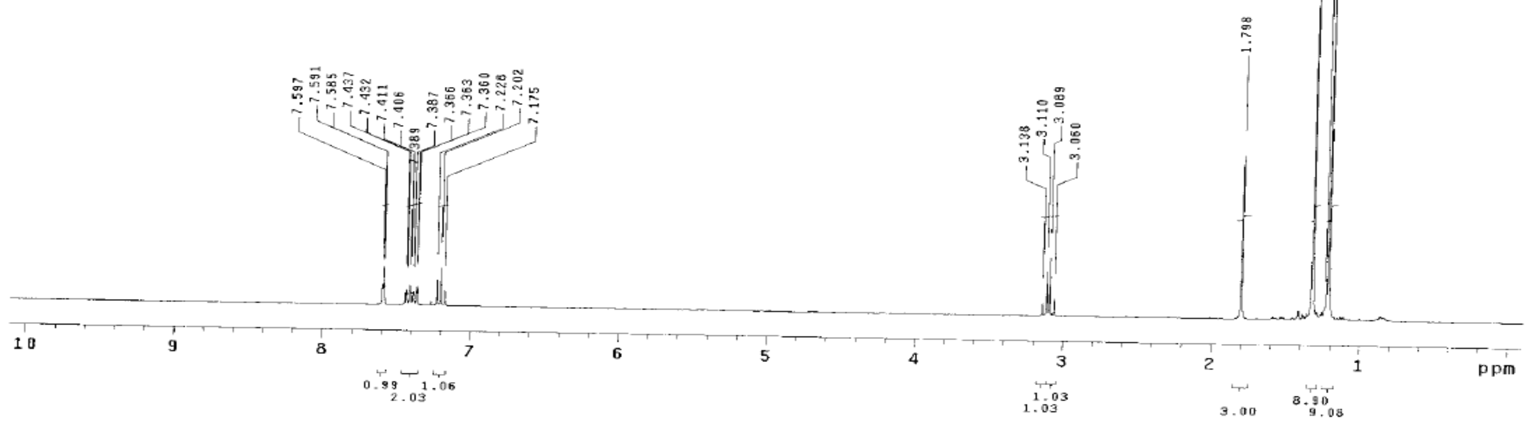


Table 1, entry 4



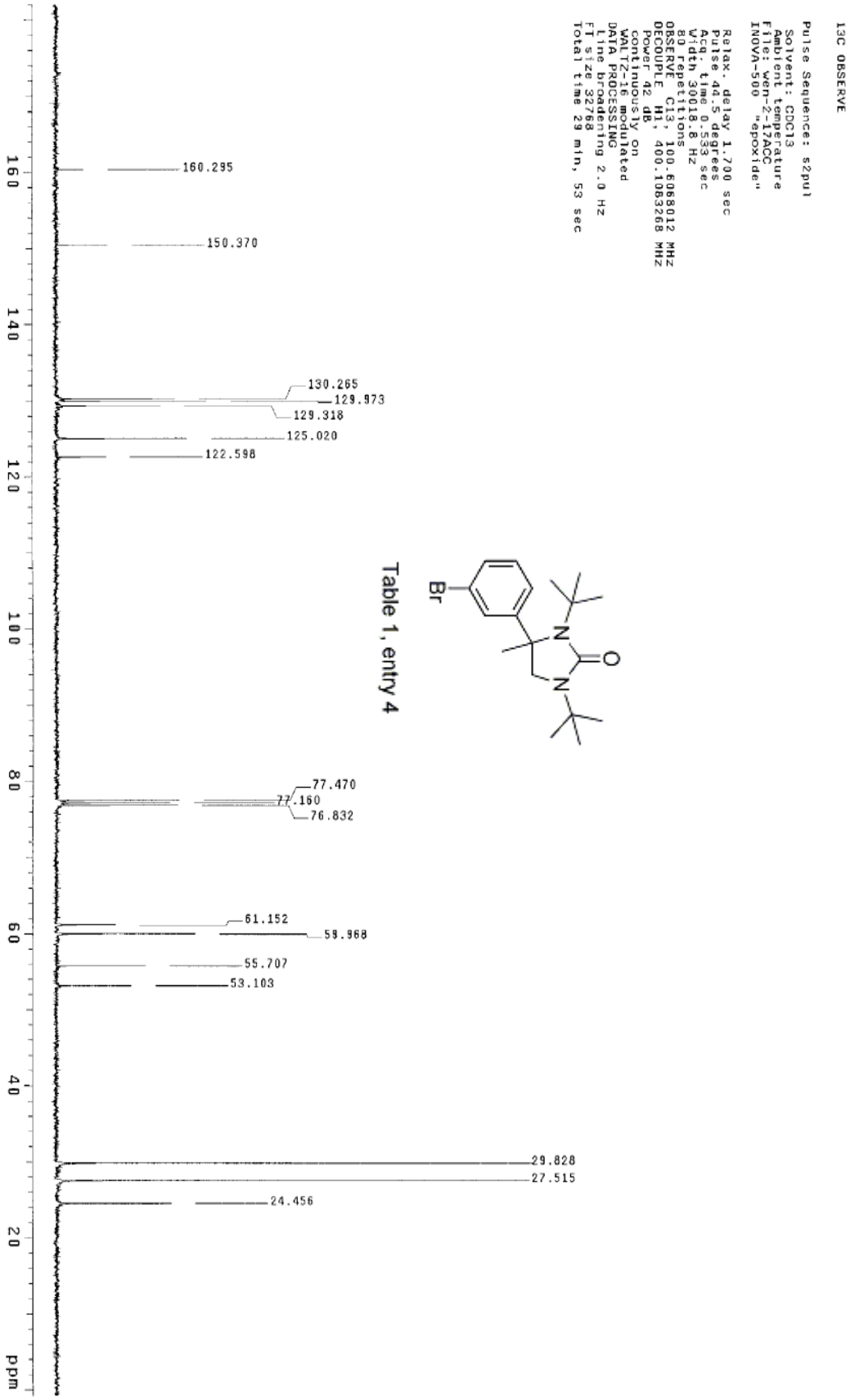
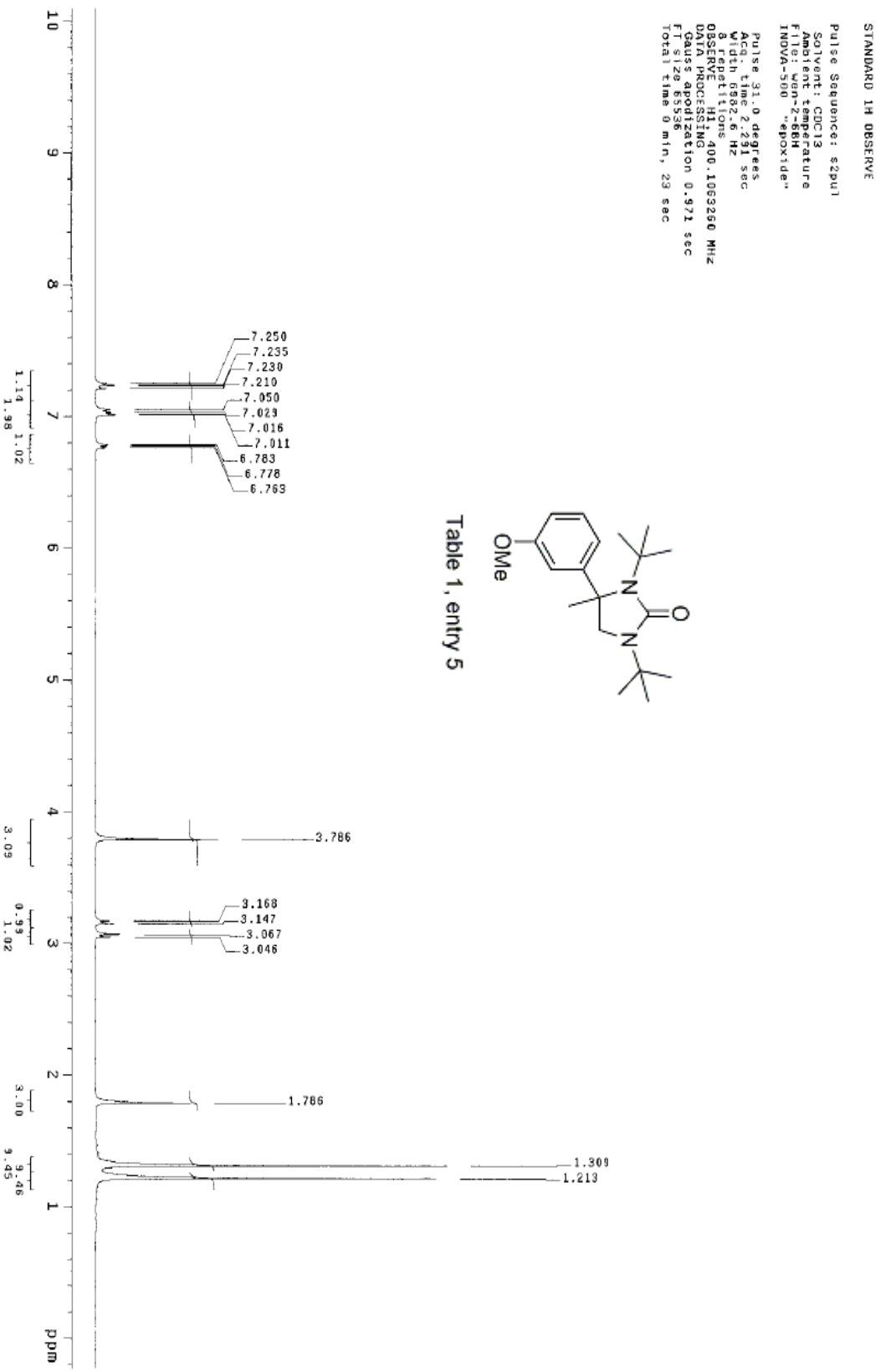


Table 1, entry 4


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wgn-2-66H
INOVA-500 "epoxide"
Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6802.6 Hz
8 repetitions
OBSERVE H1, 400.1063260 MHz
DATA PROCESSING
Gauss apodization 0.921 sec
FT size 65536
Total time 0 min, 23 sec
OMe
Table 1, entry 5
7.250
7.235
7.230
7.210
7.050
7.029
7.016
7.011
6.783
6.778
6.769
3.786
3.168
3.147
3.067
3.046
1.786
1.309
1.213
1.14
1.98
1.02
3.09
0.99
1.02
3.00
9.46
9.45
ppm

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-6BC
INOVA-500 "epoxide"

Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
420 repetitions
OBSERVE C13, 100.6067993 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 29 min, 53 sec

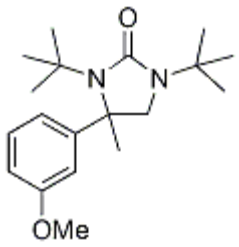
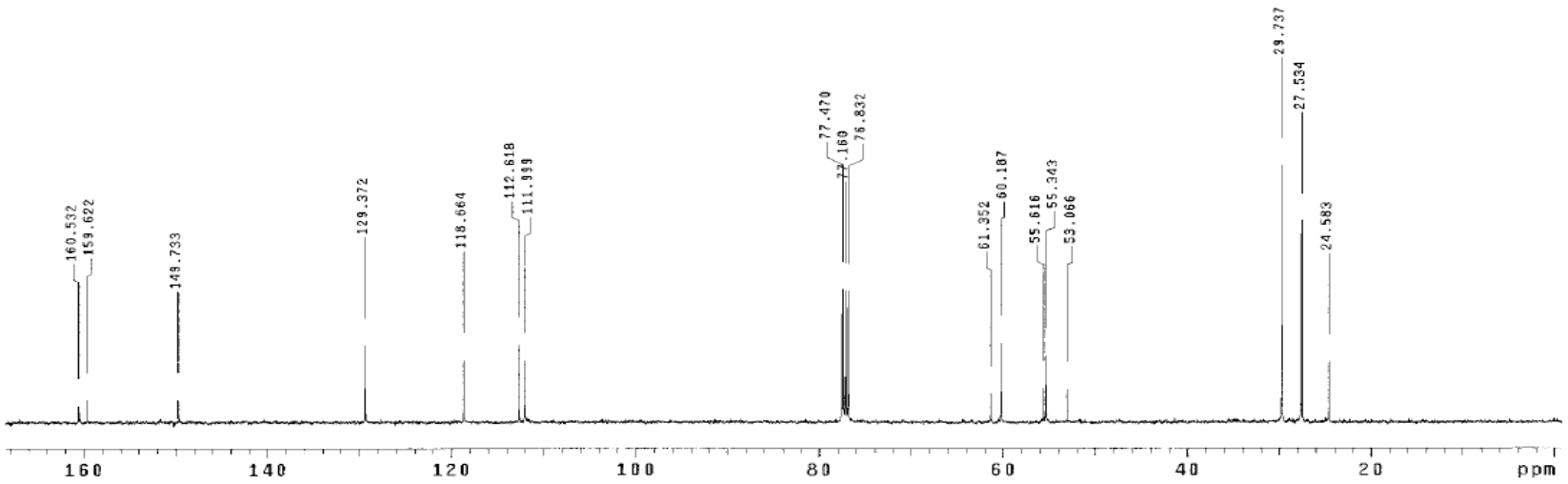


Table 1, entry 5



STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-17BH
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
8 repetitions
OBSERVE H1, 299.9533661 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 37 sec

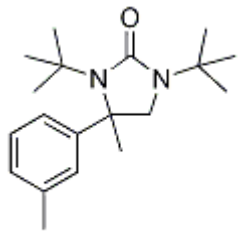
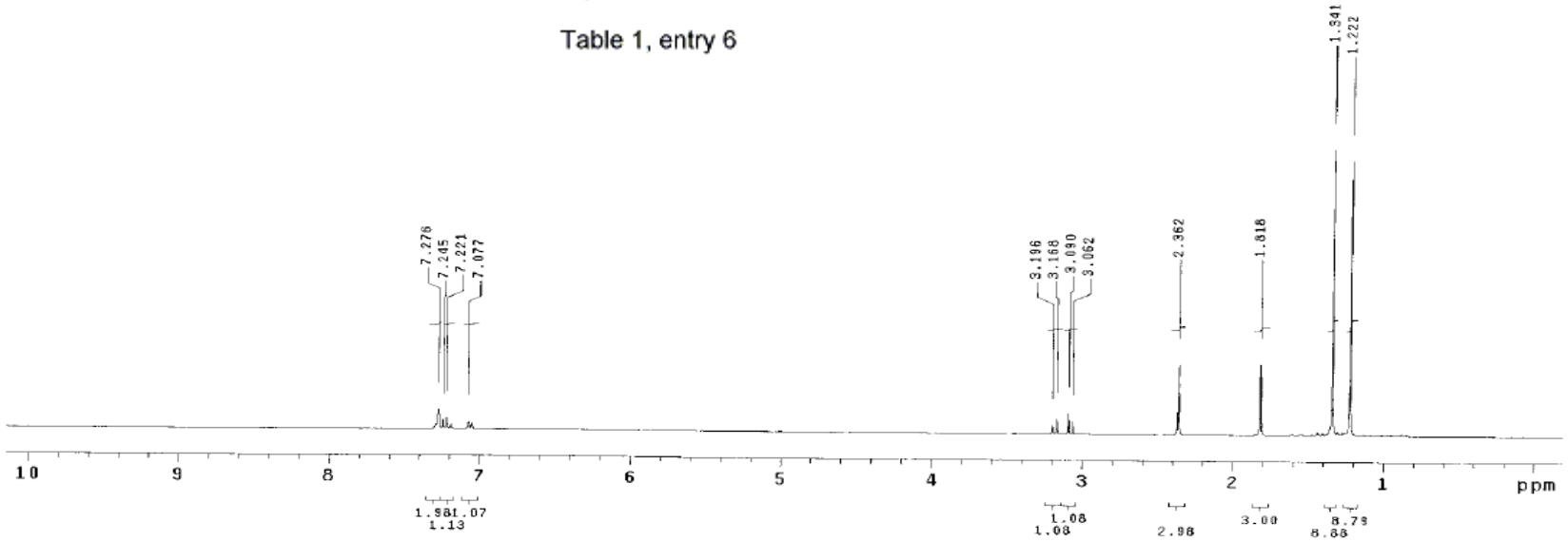


Table 1, entry 6



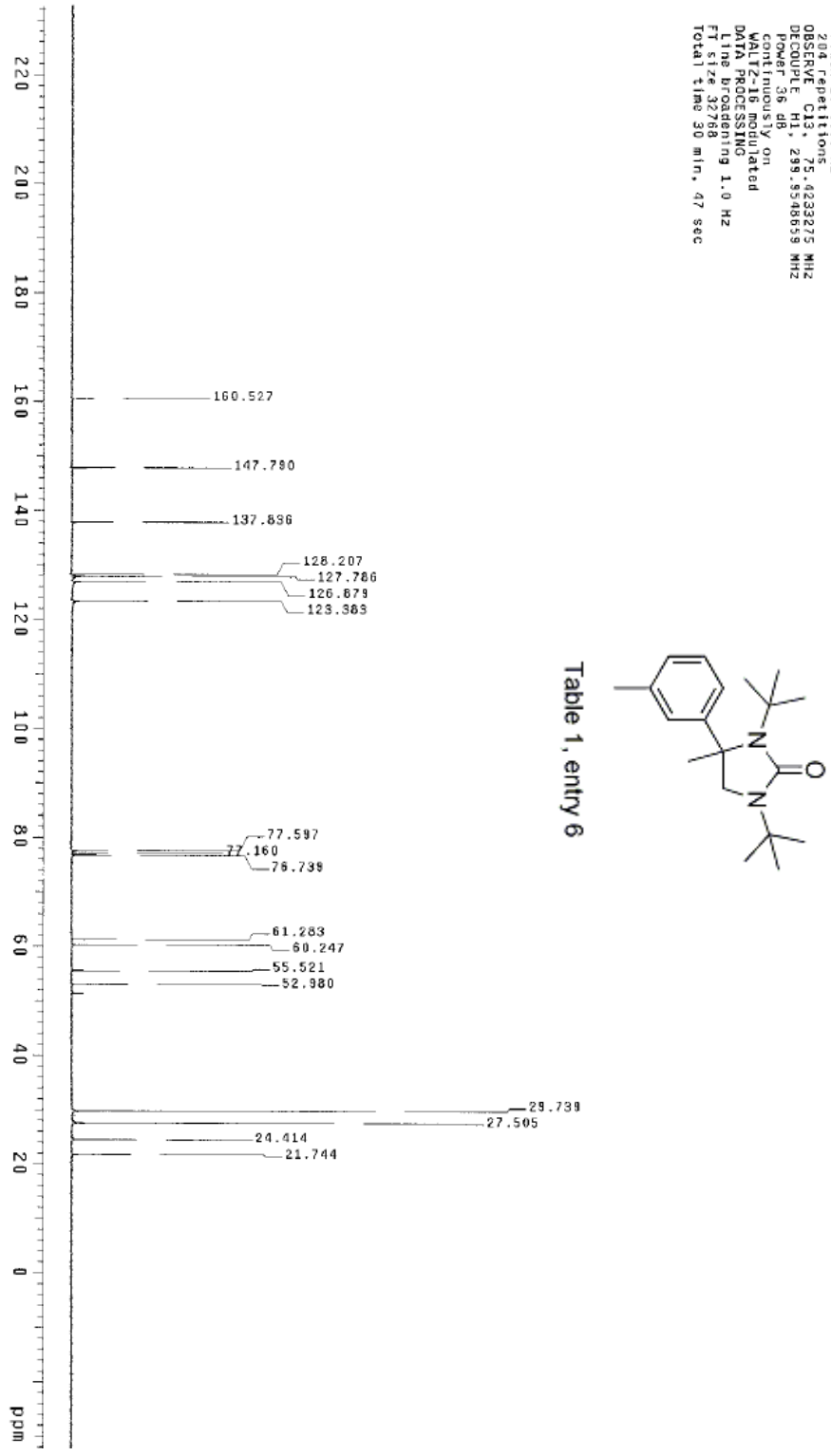


Table 1, entry 6

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-20AH
INOVA-500 "epoxide"

Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
8 repetitions
OBSERVE H1, 400.1063260 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 23 sec

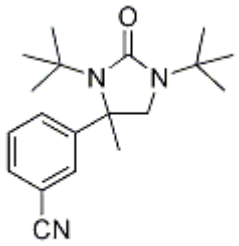
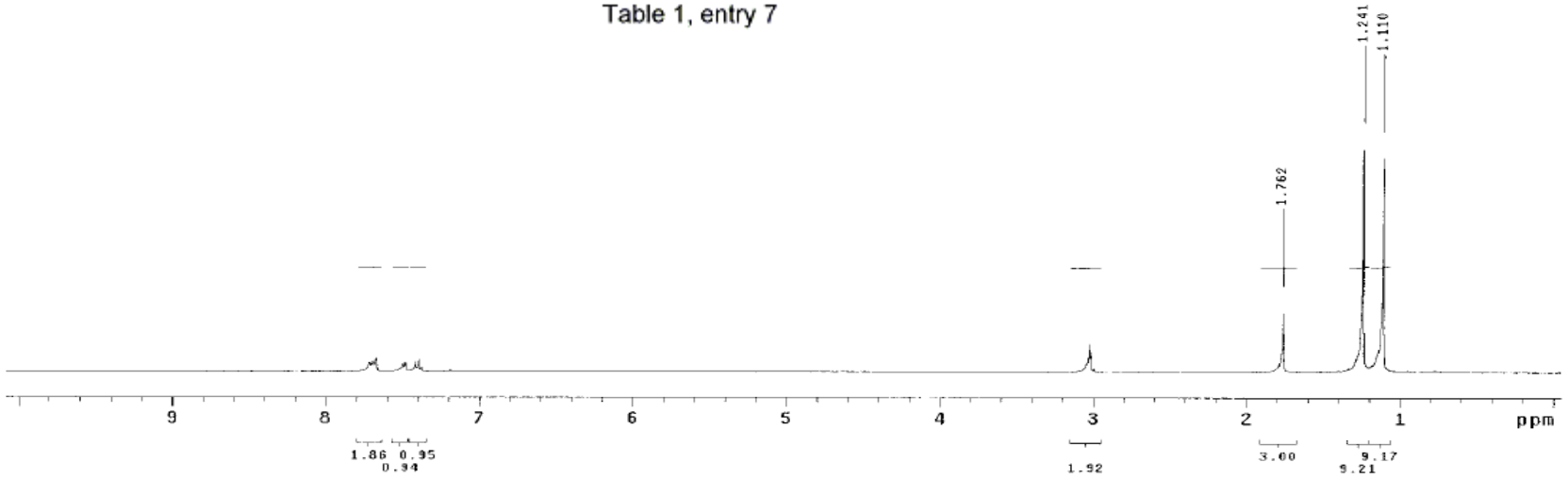
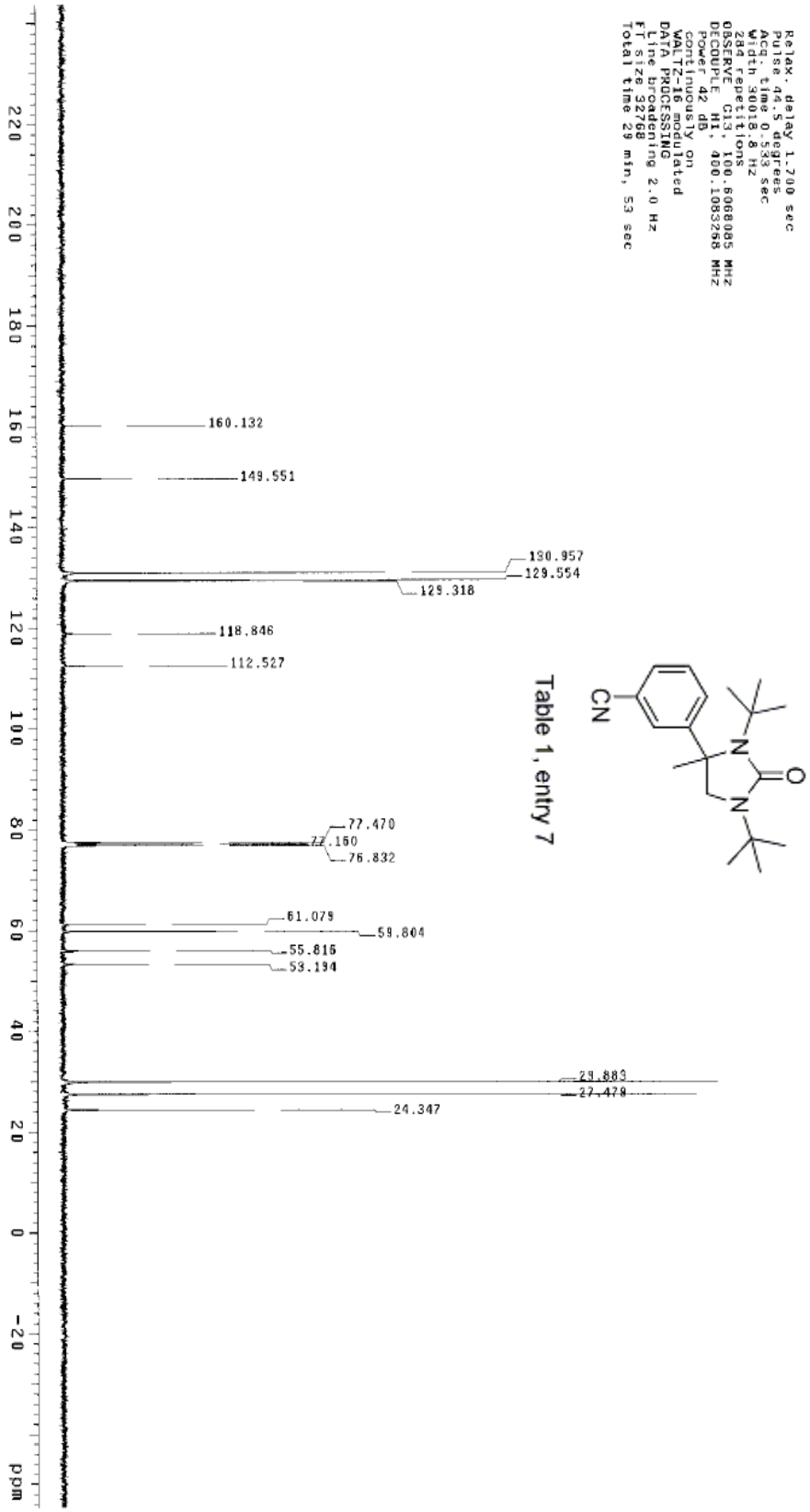


Table 1, entry 7




13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-20AC
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.583 sec
Width 30018.8 Hz
284 repetitions
OBSERVE C13, 100.6068085 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 29 min, 53 sec
160.132
149.551
130.957
129.554
129.318
118.848
112.527
77.470
77.160
76.832
61.079
59.804
55.815
53.194
29.889
27.479
24.347
CN
N
O
N
Table 1, entry 7
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: wen-2-58H
INOVA-500 "epoxide"

Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
8 repetitions
OBSERVE H1, 400.1063260 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 23 sec

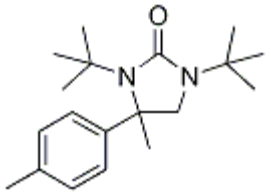
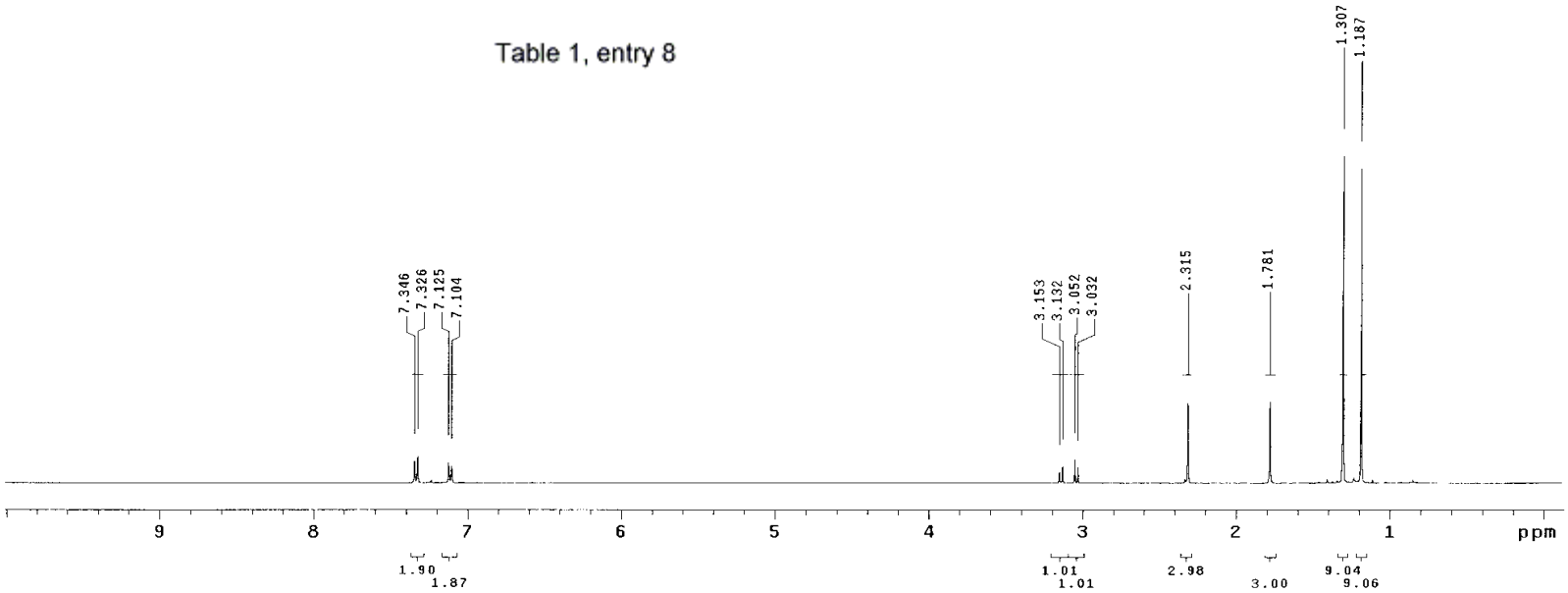
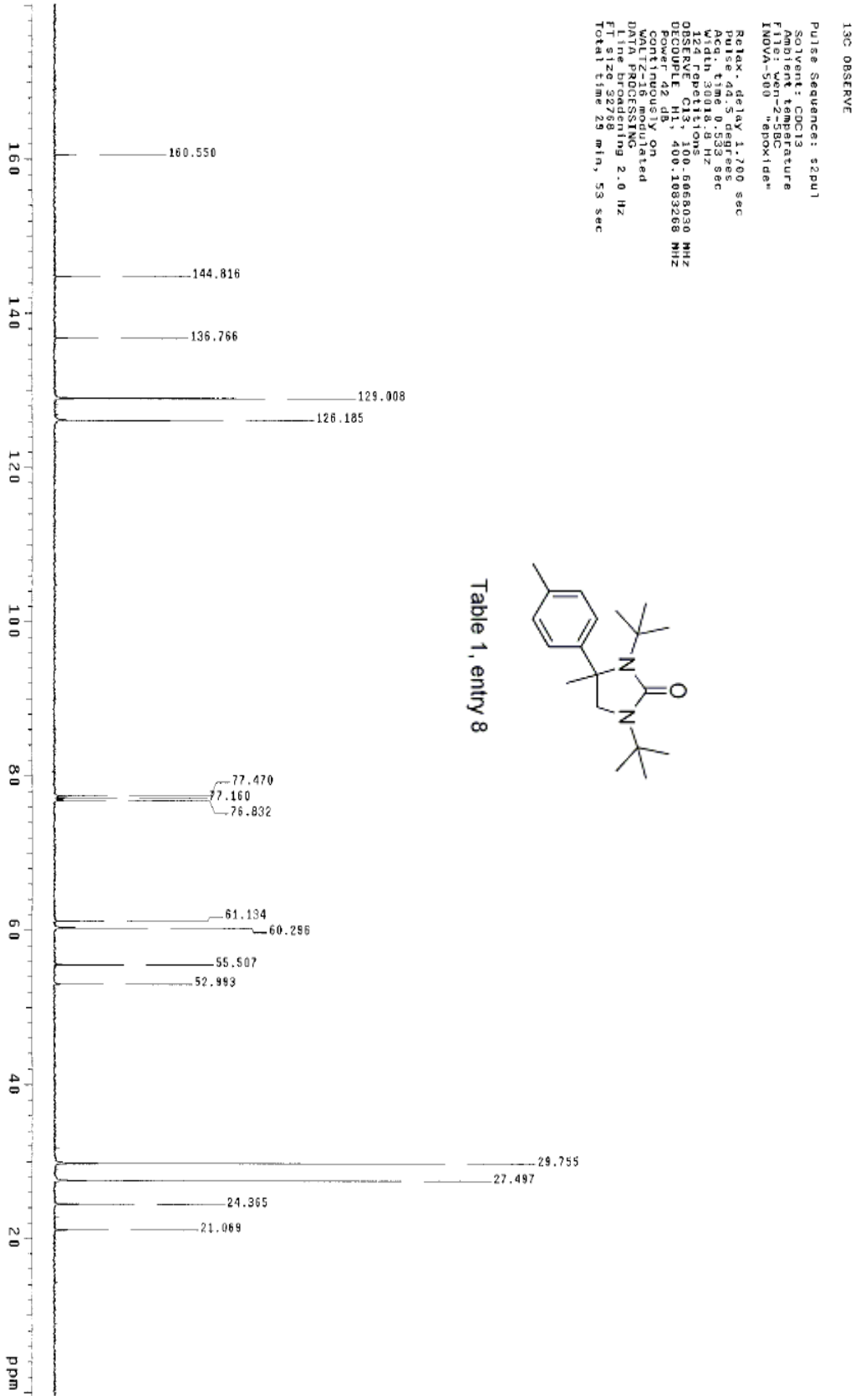
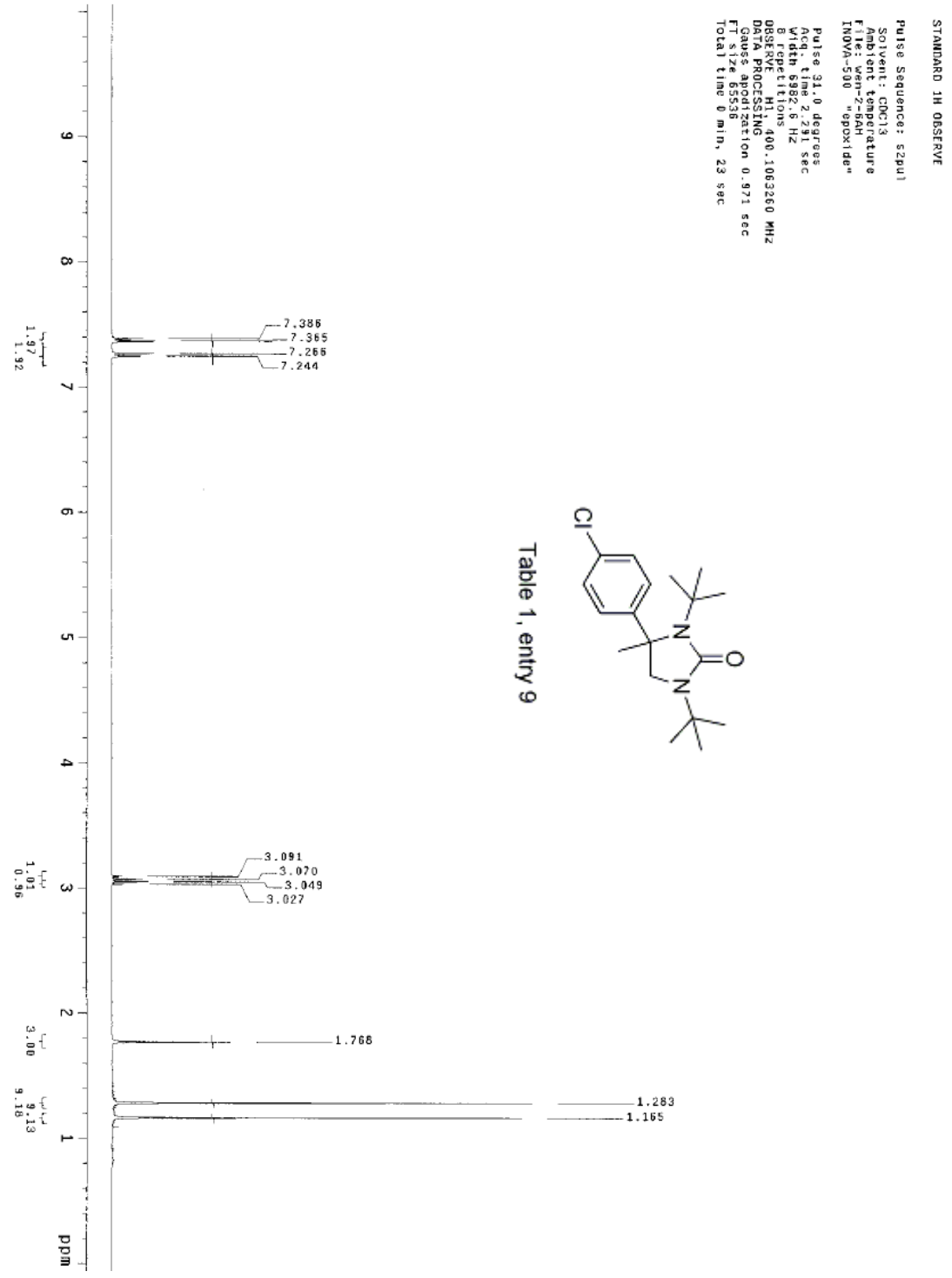


Table 1, entry 8




13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: ven-2-58C-
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.933 sec
Width 30811.0 Hz
124 repetitions
OBSERVE C13, 100.6668030 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 29 min, 53 sec
Table 1, entry 8
N
O
N
160.550
144.816
136.766
129.008
126.185
77.470
77.160
76.832
61.134
60.286
55.507
52.993
29.755
27.497
24.365
21.069
160
140
120
100
80
60
40
20
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-6AH
INOVA-500 "epoxide"
Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 HZ
8 repetitions
OBSERVE H1, 400.1063260 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 23 sec
Table 1, entry 9
Cl
N
O
N
7.386
7.365
7.266
7.244
3.091
3.070
3.049
3.027
1.768
1.283
1.165
1.97
1.92
1.01
0.96
3.00
9.13
9.18
ppm

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-6AC
INOVA-500 "epoxide"

Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
152 repetitions
OBSERVE C13, 100.6068030 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 29 min, 53 sec

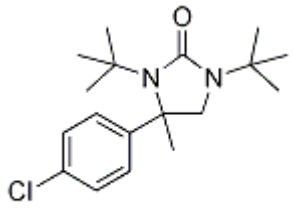
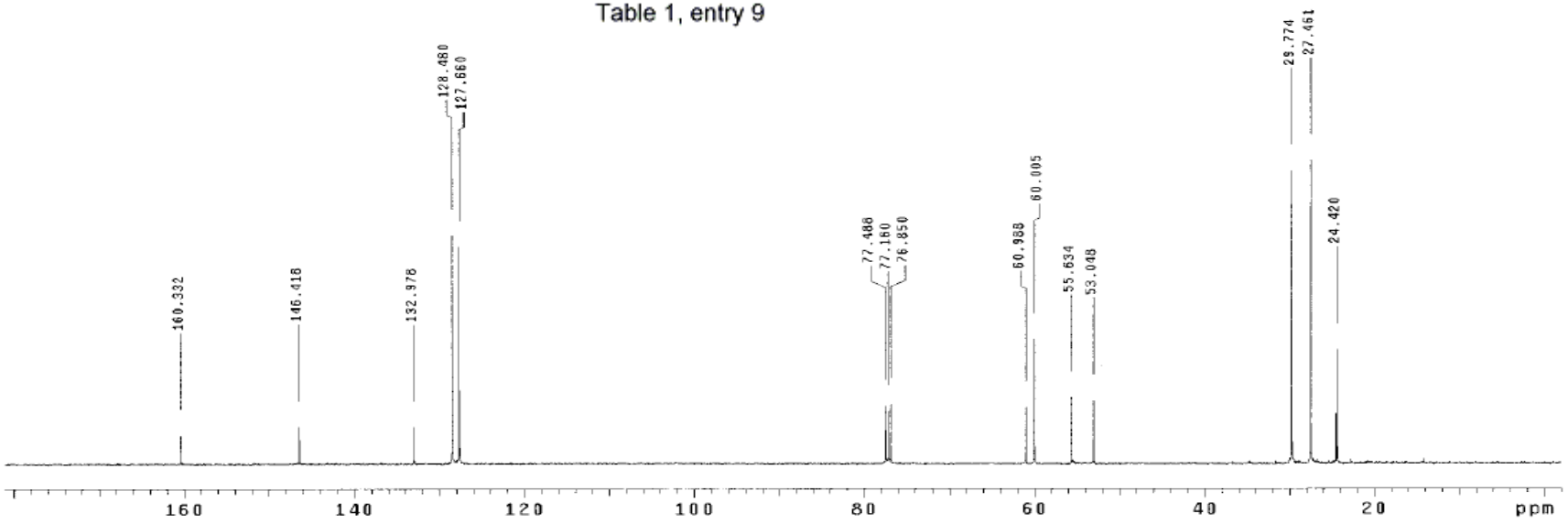
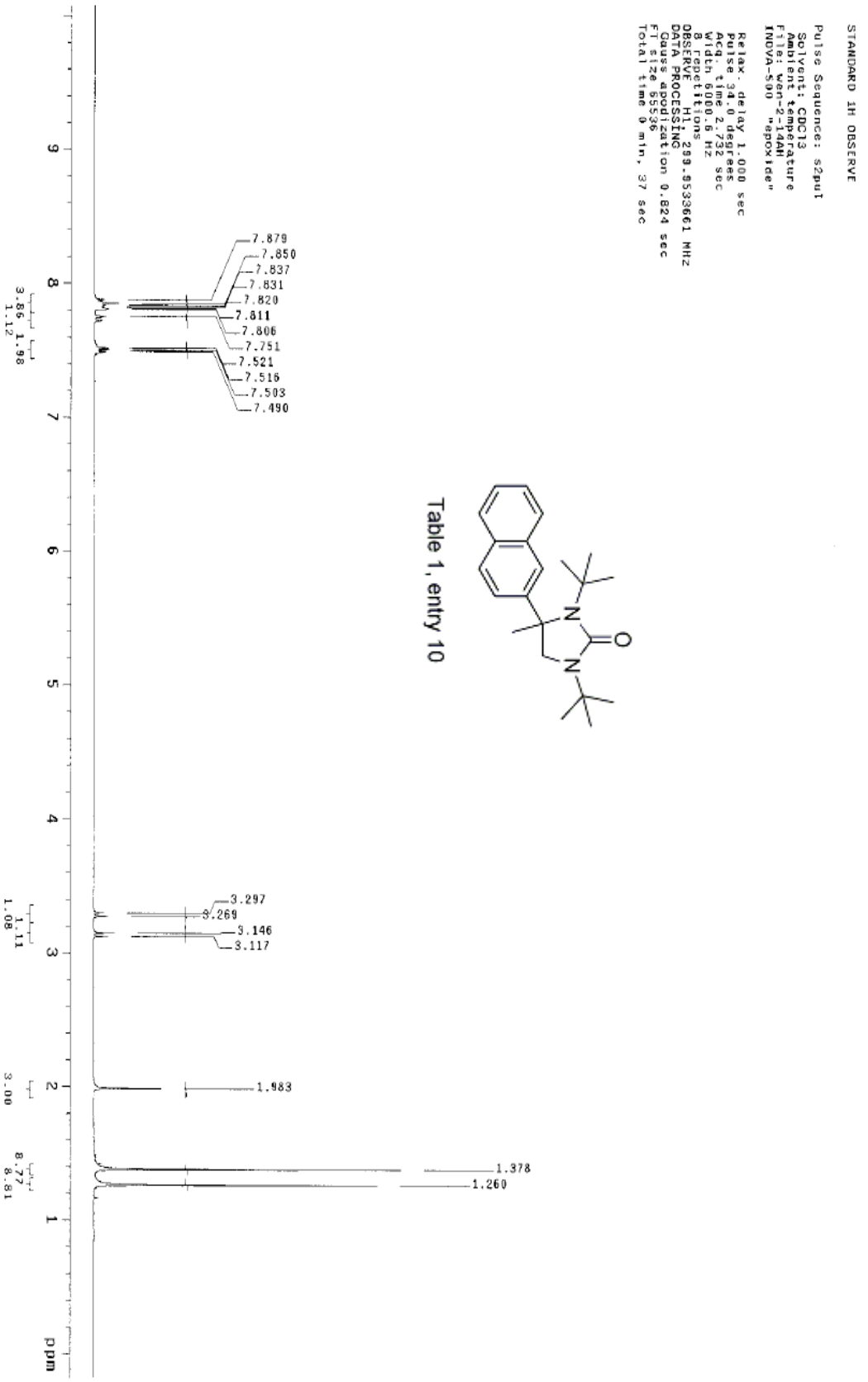


Table 1, entry 9




STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-14AH
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
8 repetitions
OBSERVE H1, 299.9533661 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 37 sec
Table 1, entry 10
7.879
7.850
7.837
7.831
7.820
7.811
7.806
7.751
7.521
7.516
7.503
7.490
3.297
3.269
3.146
3.117
1.983
1.378
1.260
3.85
1.98
3.12
1.11
1.08
3.00
8.77
8.81
ppm

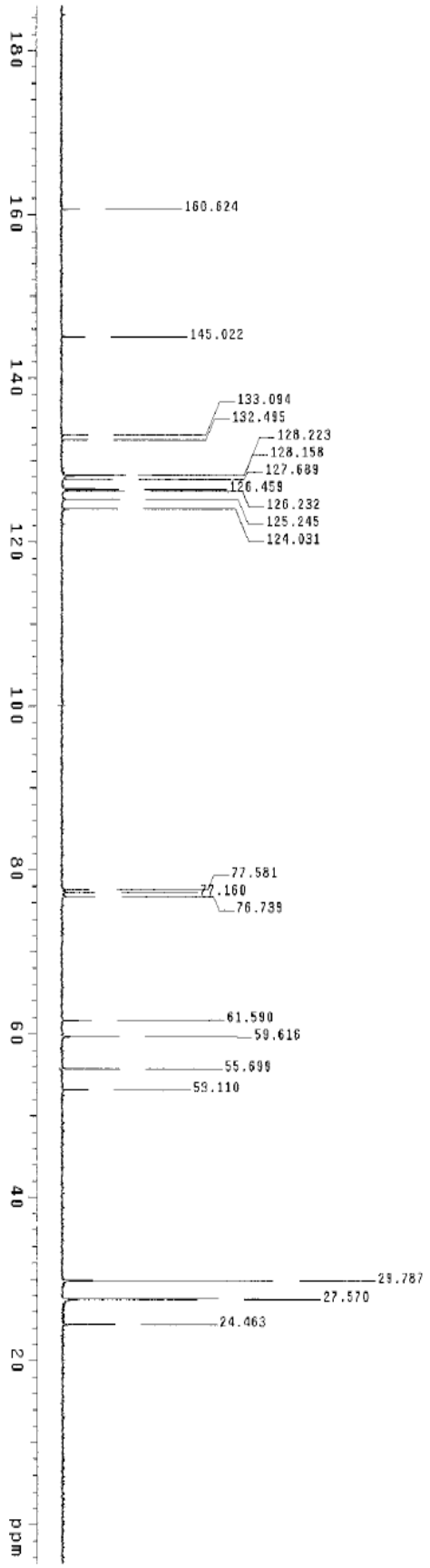
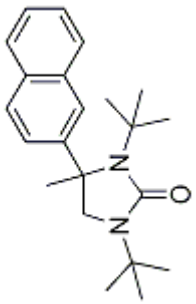


Table 1, entry 10



STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
File: wen-2-7AH
INOVA-500 "epoxide"

 Relax. delay 1.000 sec
 Pulse 34.0 degrees
 Acq. time 2.732 sec
 Width 6000.6 Hz
 16 repetitions
OBSERVE   H1, 299.9533661 MHz
DATA PROCESSING
 Gauss apodization 0.824 sec
FT size 65536
Total time 1 min, 7 sec

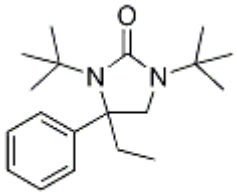
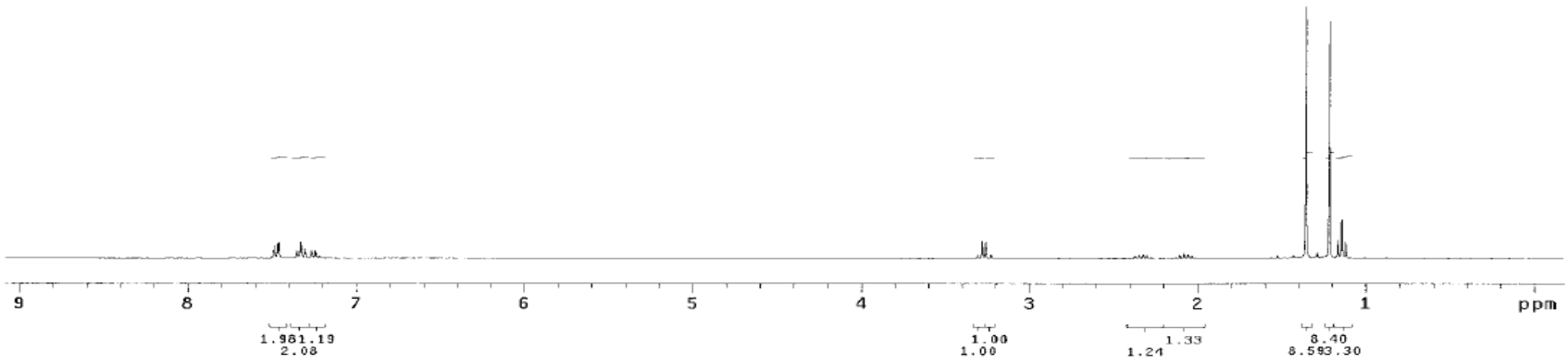
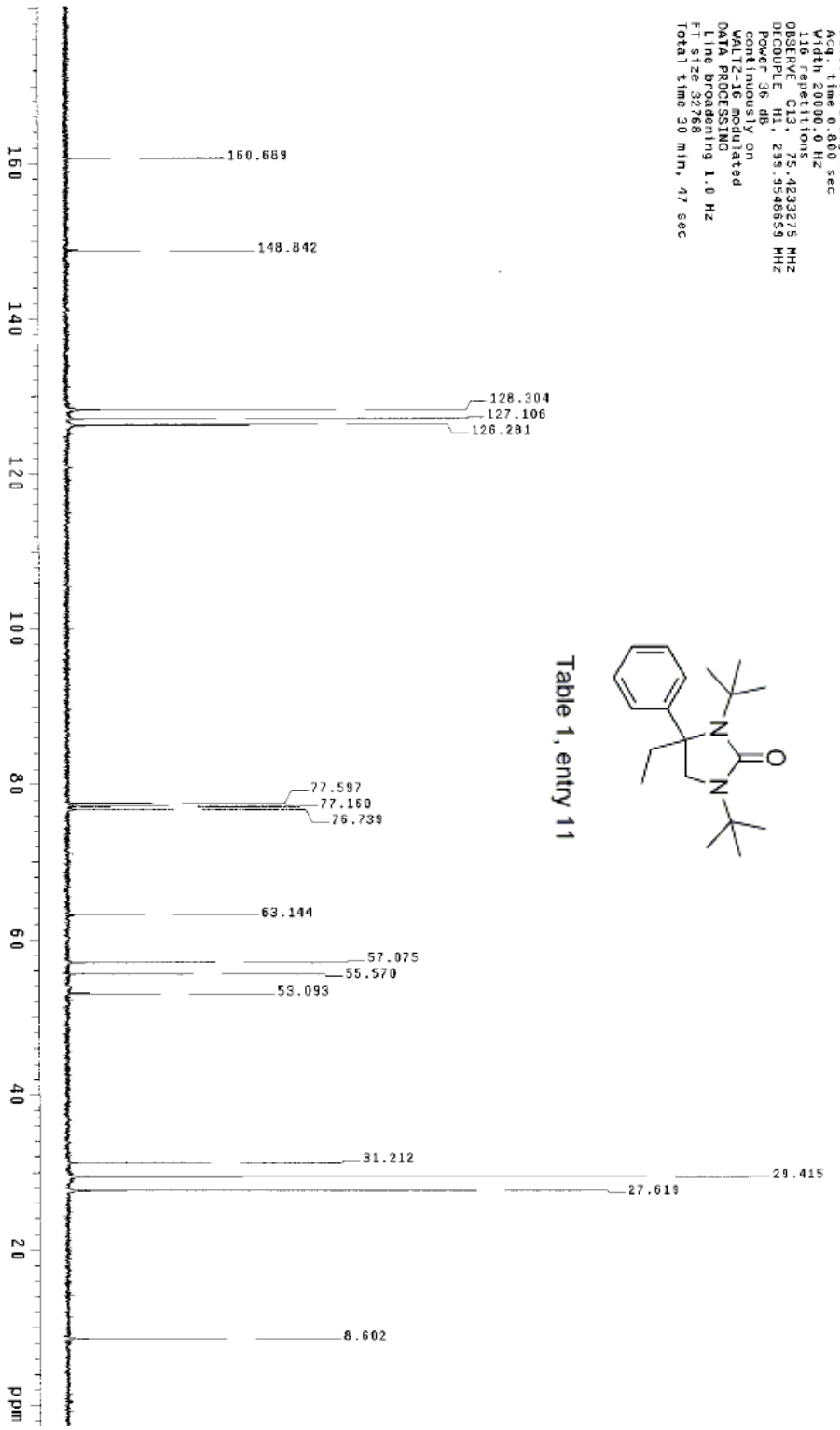
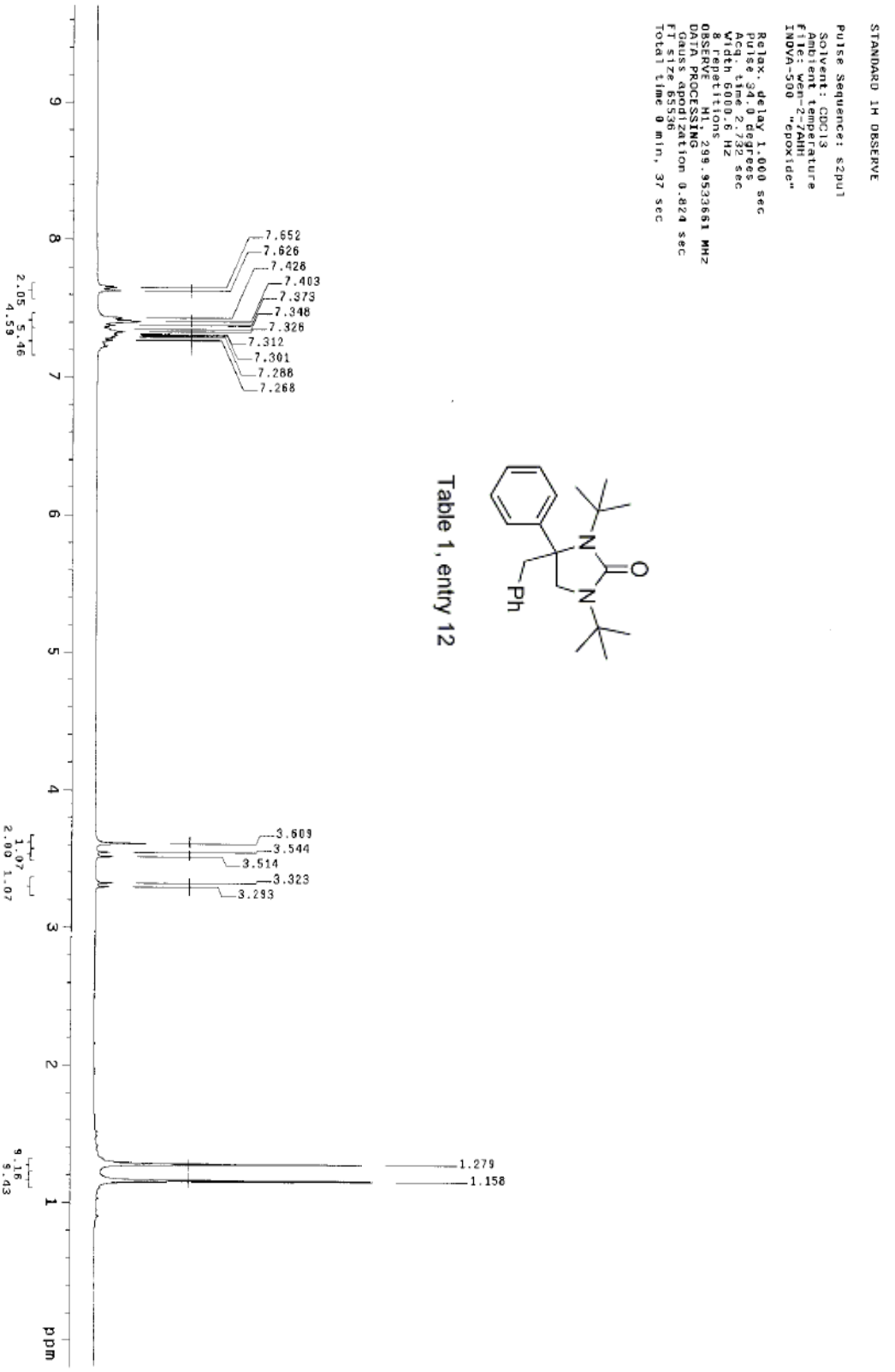


Table 1, entry 11




13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-7AC
INOVA-500 "epoxide"
Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
116 repetitions
OBSERVE C13, 75.4233275 MHz
DECOUPLE H1, 299.9486659 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 30 min, 47 sec
160.689
148.842
128.304
127.106
126.281
77.597
77.160
76.739
63.144
57.075
55.570
53.093
31.212
29.415
27.619
8.602
160
140
120
100
80
60
40
20
ppm
Table 1, entry 11
N
O
N


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-7AHH
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 HZ
8 repetitions
OBSERVE H1, 299.9533661 MHZ
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 37 sec
Table 1, entry 12
Ph
N
O
N
7.652
7.626
7.426
7.403
7.373
7.348
7.326
7.312
7.301
7.288
7.268
3.609
3.544
3.514
3.323
3.293
1.279
1.158
2.05
5.46
4.59
1.07
2.00
1.07
9.16
9.43
ppm

13C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
File: wen-2-78C
INOVA-500 "epoxide"

 Relax. delay 1.500 sec
 Pulse 39.1 degrees
 Acq. time 0.800 sec
 Width 20000.0 Hz
 420 repetitions
OBSERVE C13, 75.4233251 MHz
DECOUPLE H1, 299.9548659 MHz
 Power 36 dB
 continuously on
 WALTZ-16 modulated
DATA PROCESSING
 Line broadening 1.0 Hz
FT size 32768
Total time 30 min, 47 sec

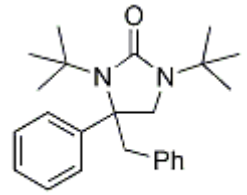
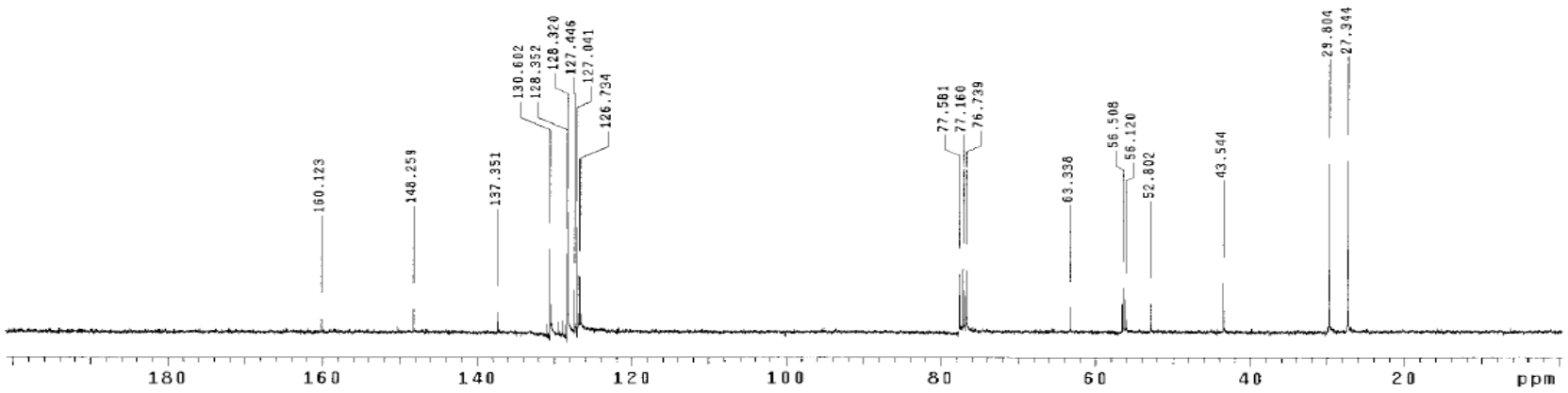


Table 1, entry 12



STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
File: wen-2-18BH
INOVA-500 "epoxide"

 Pulse 31.0 degrees
 Acq. time 2.291 sec
 Width 6982.6 Hz
 8 repetitions
OBSERVE H1, 400.1063260 MHz
DATA PROCESSING
 Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 23 sec

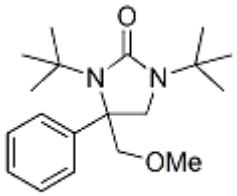
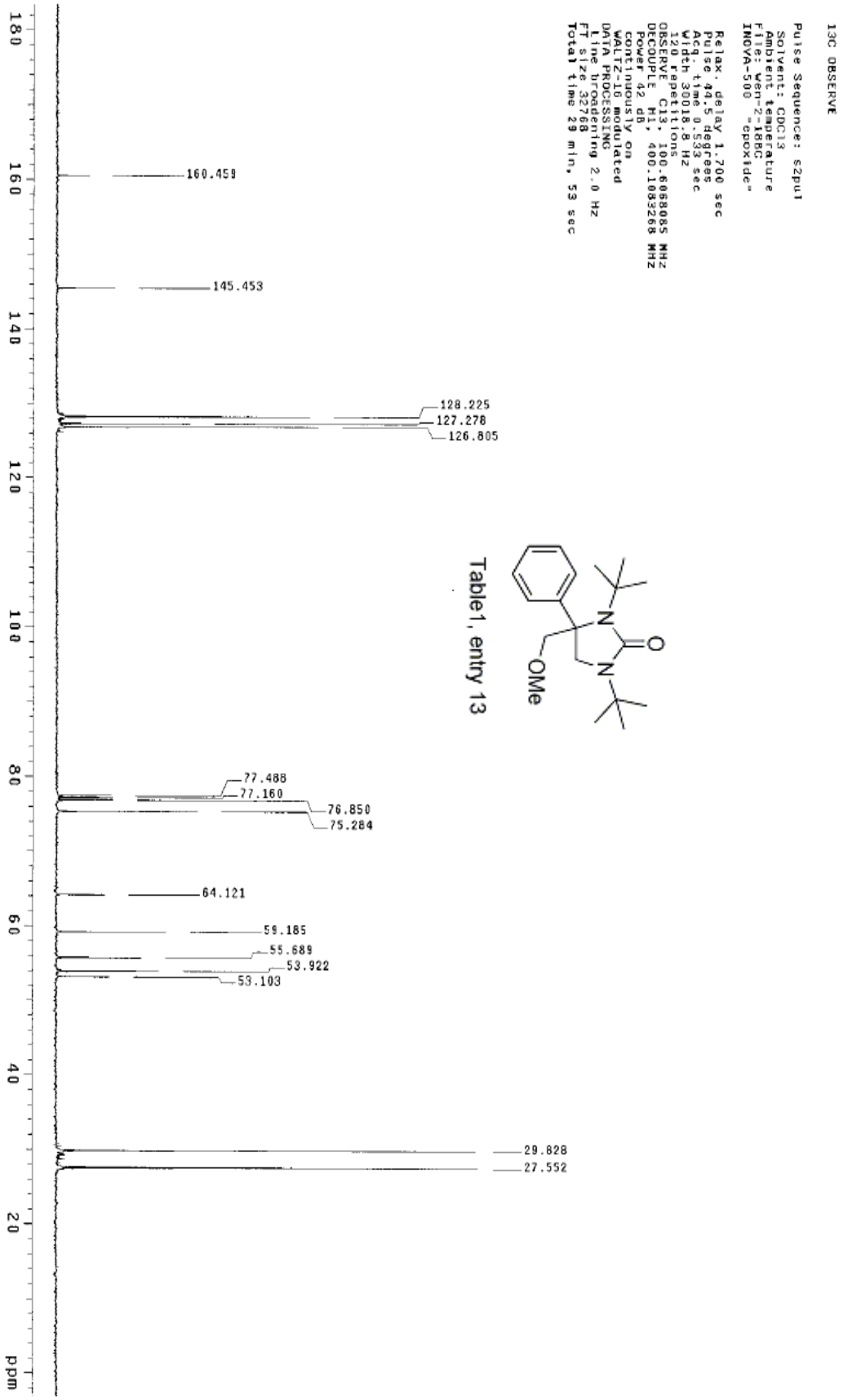


Table1, entry 13


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-18BC
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
120 repetitions
OBSERVE C13, 100.6668085 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 29 min, 53 sec
160.459
145.453
128.225
127.278
126.805
77.488
77.160
76.850
75.284
64.121
59.185
55.689
53.922
53.103
29.828
27.552
Table1, entry 13
OMe
N
O
N
180
160
140
120
100
80
60
40
20
ppm

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
File: wen-2-22AHH
INOVA-500 "epoxide"

 Relax. delay 1.000 sec
 Pulse 34.0 degrees
 Acq. time 2.732 sec
 Width 6000.6 Hz
 8 repetitions
OBSERVE   H1, 299.9533661 MHz
DATA PROCESSING
 Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 37 sec

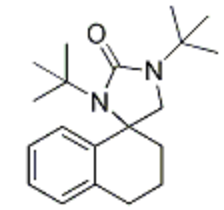
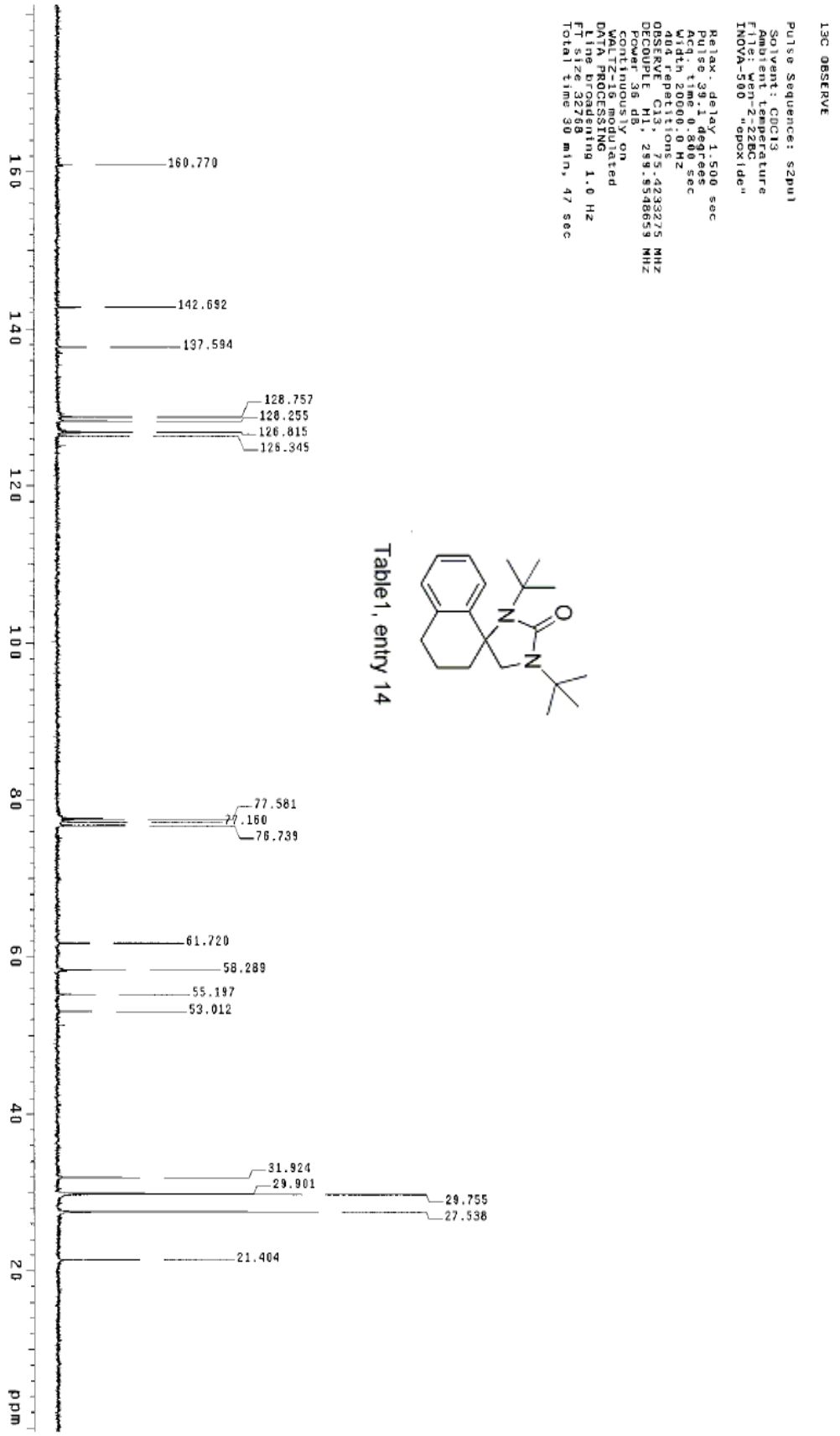


Table1, entry 14


13C OBSERVE
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: exp-2_22sc
INOVA-500 "epoxide"
Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
4 repetitions
OBSERVE C13, 75.4233275 MHz
DECOUPLE H1, 299.9548659 MHz
Power 56 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 30 min, 47 sec
160.770
142.692
137.594
128.757
128.255
126.815
126.345
77.581
77.160
76.739
61.720
58.289
55.197
53.012
31.924
29.901
29.755
27.538
21.404
160
140
120
100
80
60
40
20
ppm
Table1, entry 14
N
O
N

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
File: wen-2-148H
INOVA-500 "epoxide"

 Relax. delay 1.000 sec
 Pulse 34.0 degrees
 Acq. time 2.732 sec
 Width 6000.6 Hz
 8 repetitions
OBSERVE H1, 299.9533661 MHz
DATA PROCESSING
 Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 37 sec

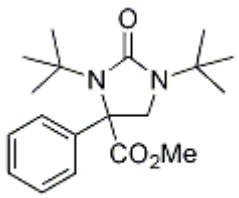
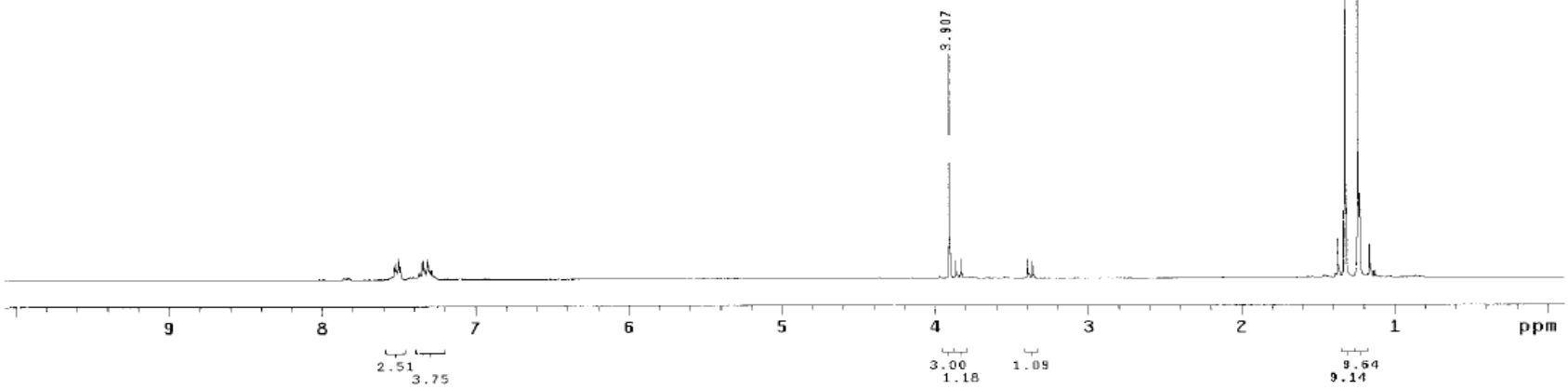
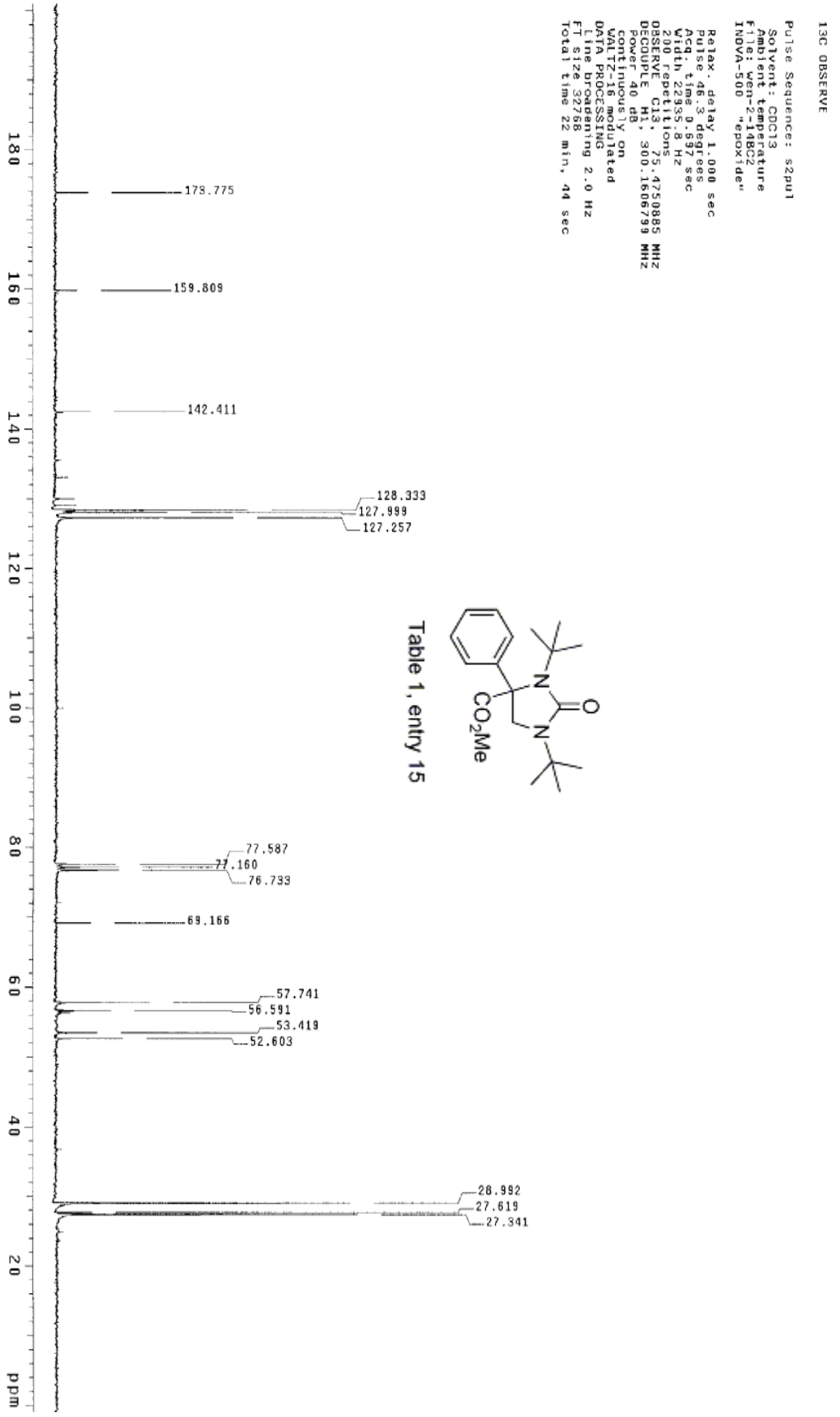


Table 1, entry 15




13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-14BC2
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 45.3 degrees
Acq. time 0.597 sec
Width 22935.8 Hz
200 repetitions
OBSERVE C13, 75.4750885 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
178.775
159.809
142.411
128.333
127.999
127.257
77.587
77.160
76.733
69.166
57.741
56.591
53.419
52.603
28.992
27.619
27.341
180
160
140
120
100
80
60
40
20
ppm
Table 1, entry 15
CO2Me
N
O
N

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-9AH
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
8 repetitions
OBSERVE H1, 299.9533661 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 37 sec

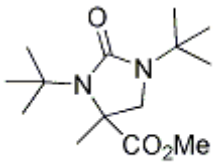
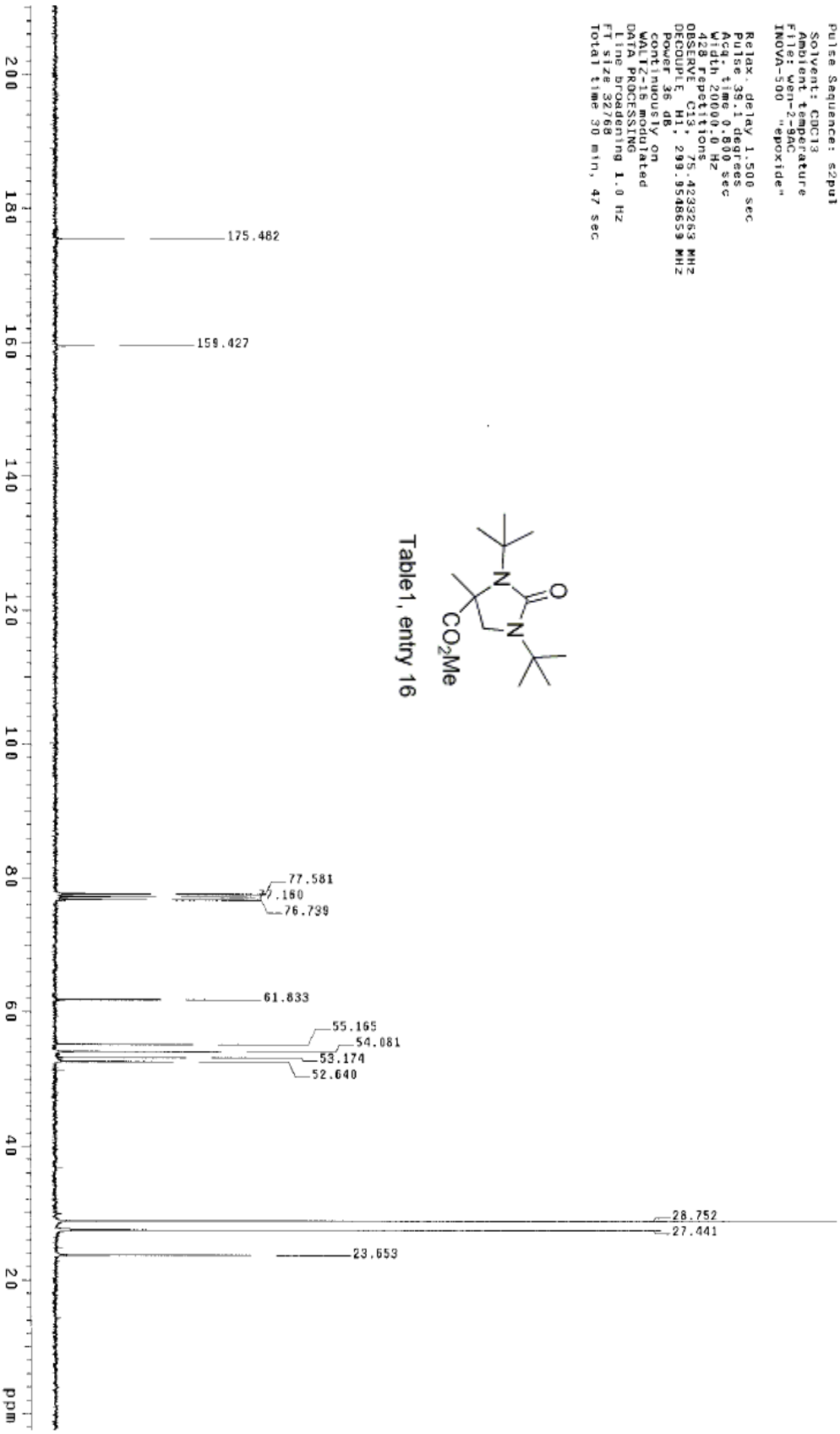
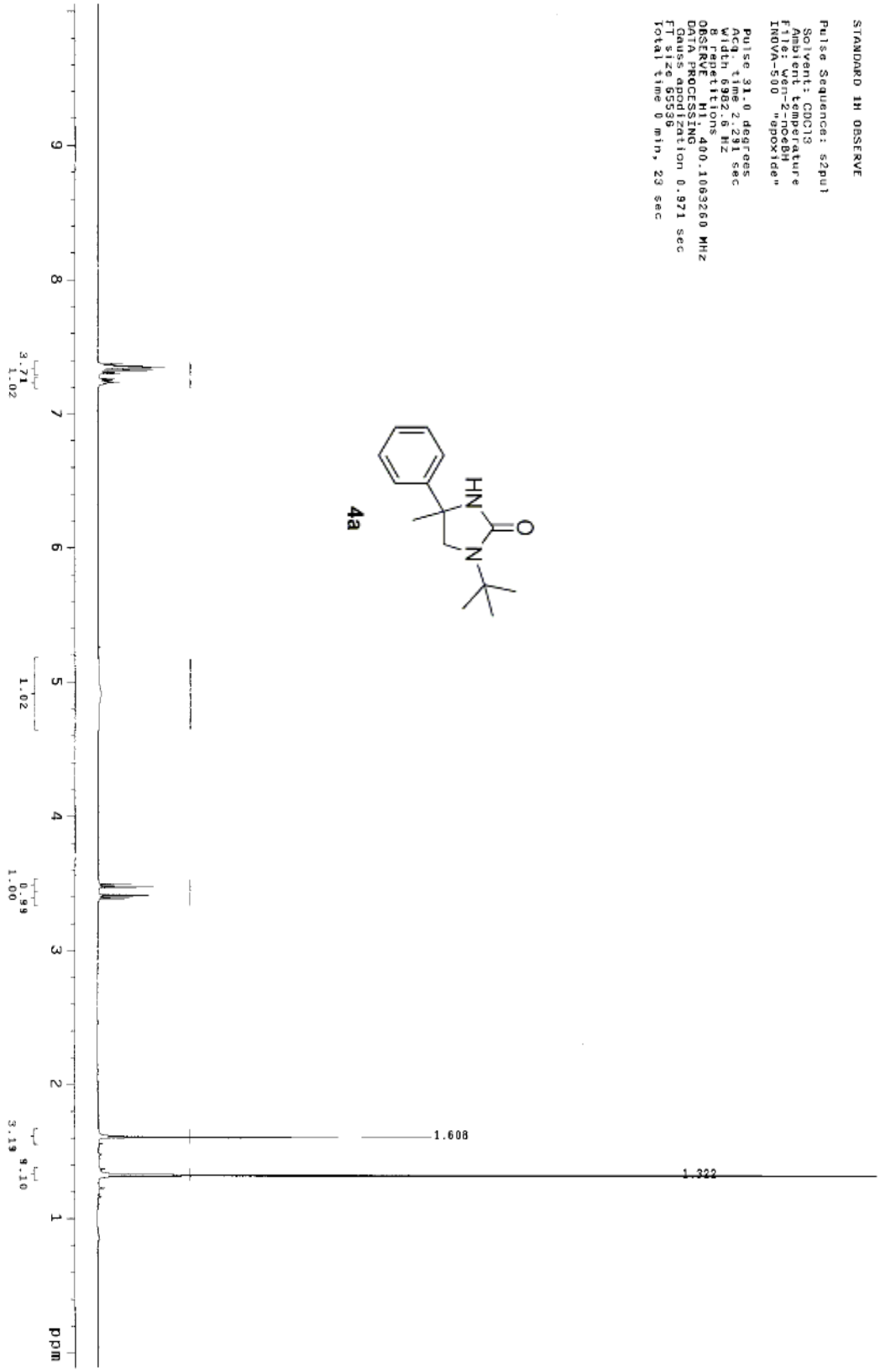


Table1, entry 16

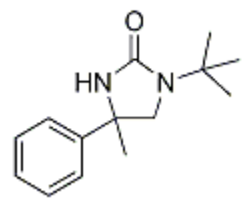

13C OBSERVE
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: wen-2-8AC
INOVA-500 "epoxide"
Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
428 repetitions
OBSERVE C13, 75.4233263 MHz
DECOUPLE H1, 299.9548659 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 30 min, 47 sec
Table1, entry 16
CO2Me
175.482
159.427
77.581
77.160
76.739
61.833
55.165
54.081
53.174
52.640
28.752
27.441
23.653
200
180
160
140
120
100
80
60
40
20
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-noeBH
INOVA-500 "epoxide"
Pulse 31.0 degrees
Acq. time 2.291 sec
width 6982.6 HZ
8 repetitions
OBSERVE H1, 400.1063260 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 23 sec
4a
HN
O
N
1.608
1.322
3.71
1.02
1.02
0.99
1.00
3.19
9.10
ppm

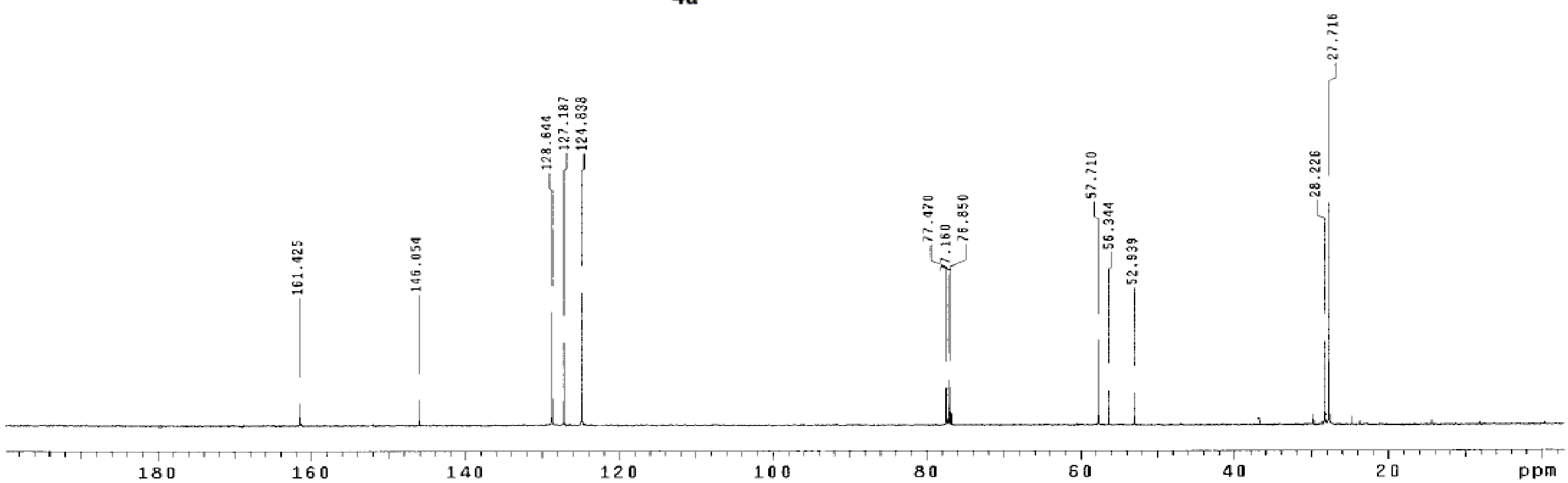
13C OBSERVE

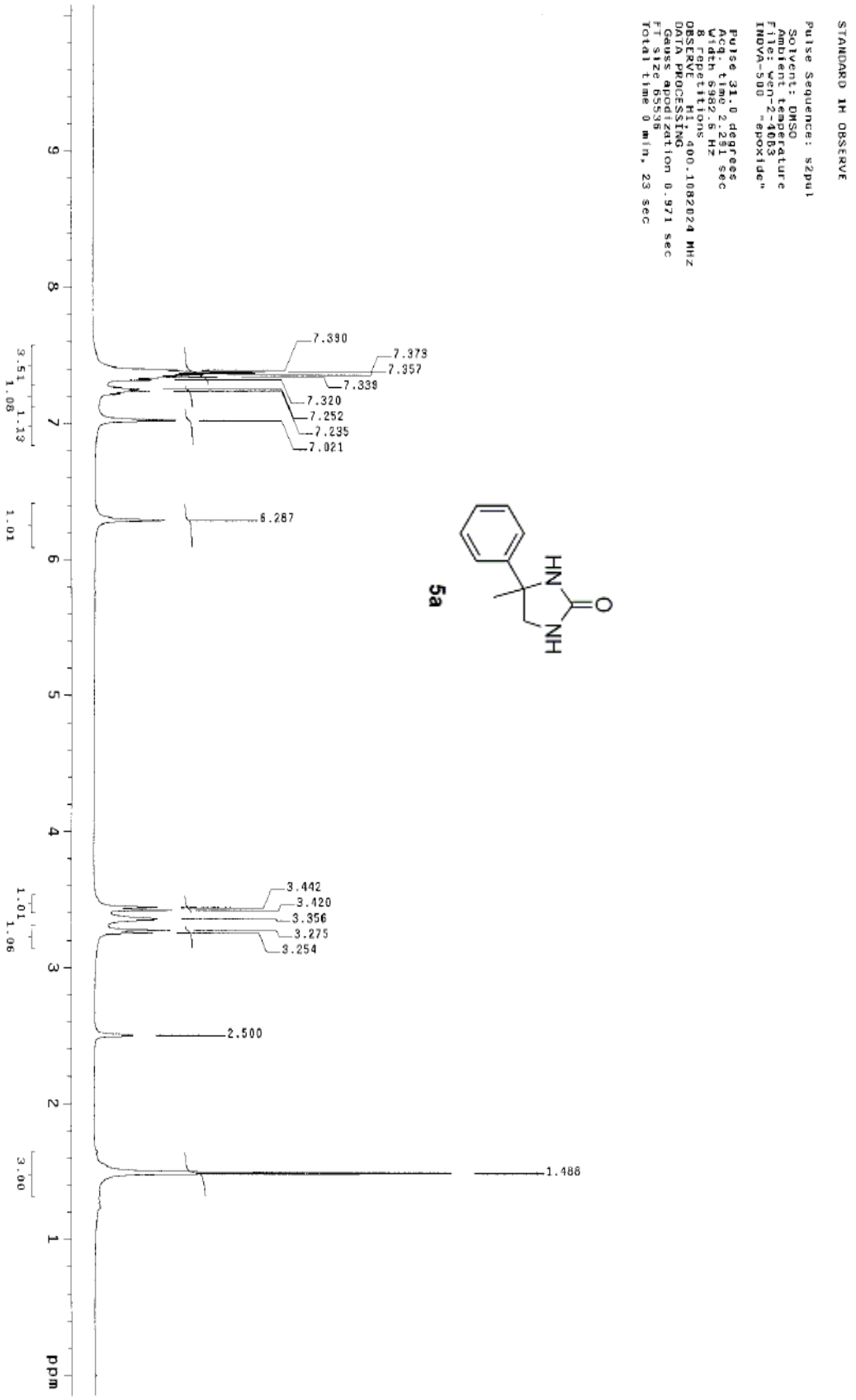
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-40A1C
INOVA-500 "epoxide"

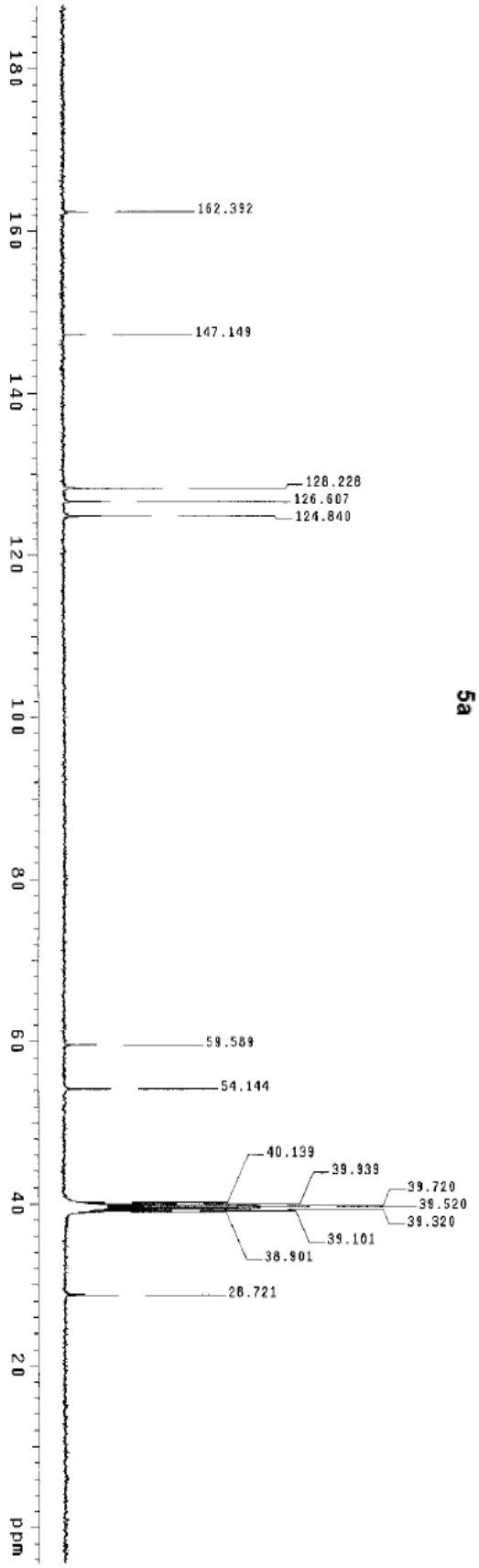
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
48 repetitions
OBSERVE C13, 100.6068067 MHz
DECOUPLE H1, 400.1083268 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 29 min, 53 sec

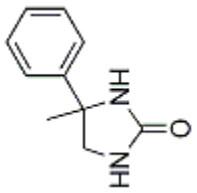


4a

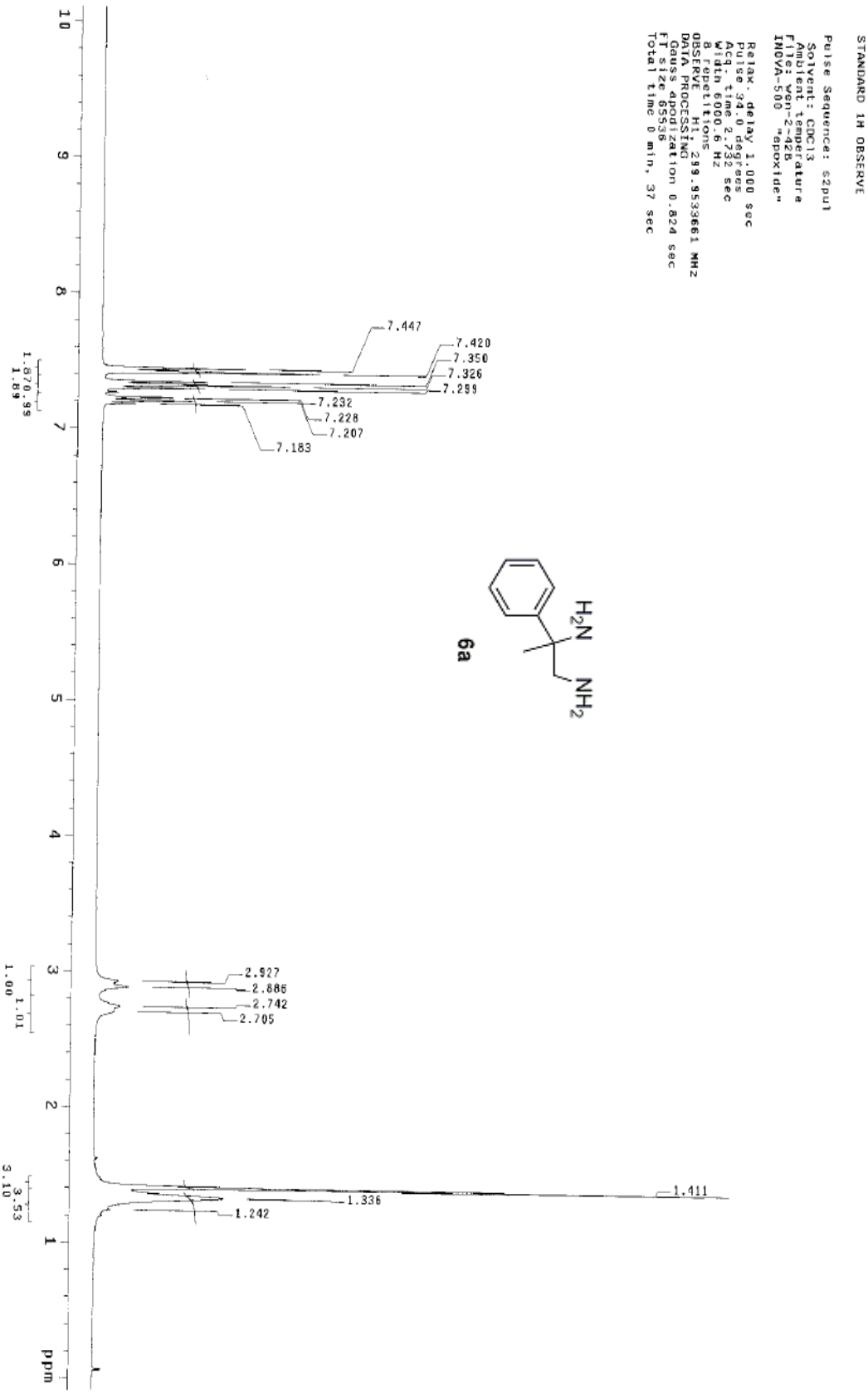


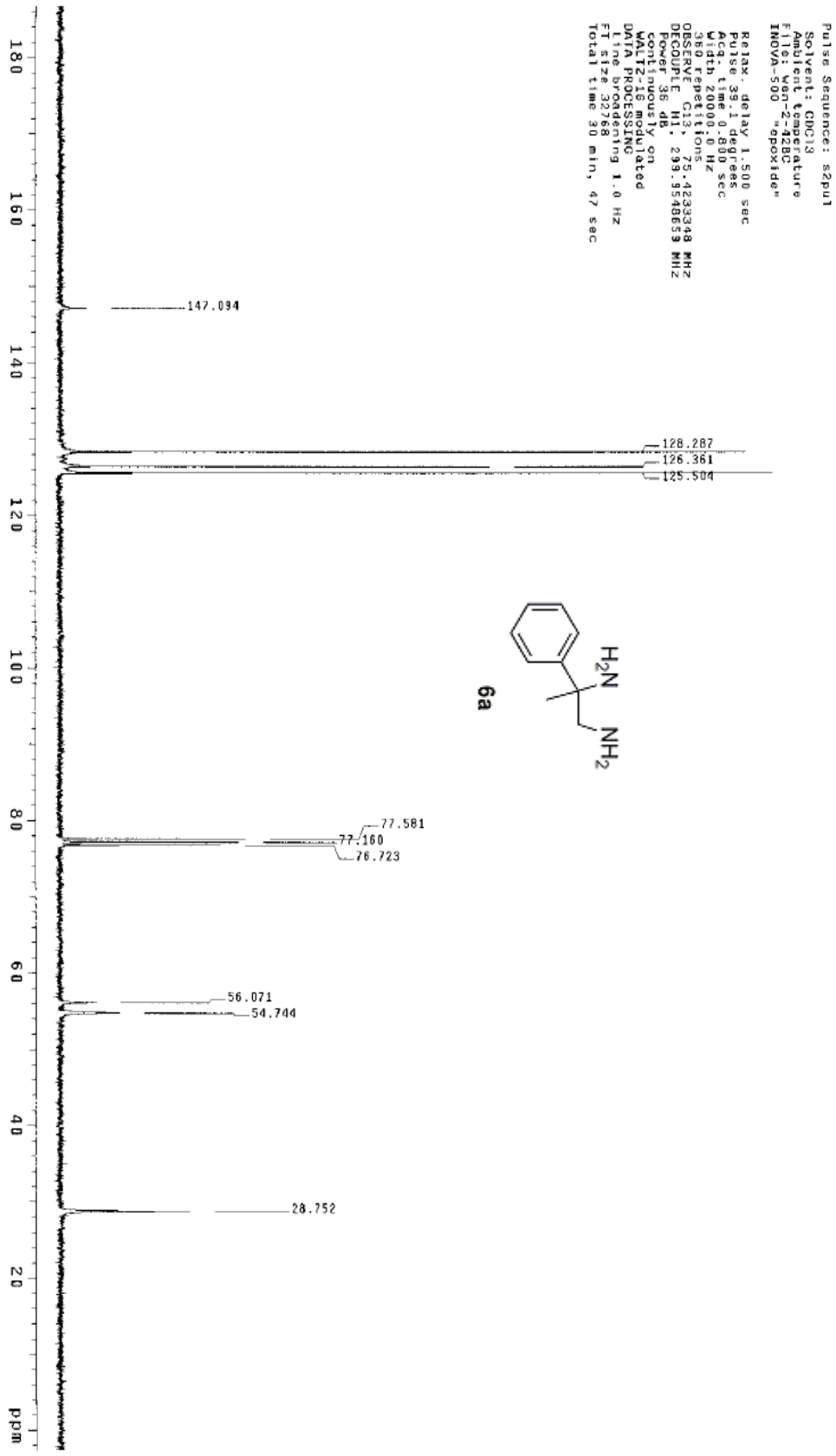

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: wen-2-4083
INOVA-500 "epoxide"
Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.5 Hz
8 repetitions
OBSERVE H1, 400.1082024 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 23 sec
5a
7.390
7.378
7.357
7.339
7.320
7.252
7.235
7.021
6.287
3.442
3.420
3.356
3.275
3.254
2.500
1.486
3.51
1.08
1.13
1.01
1.01
1.06
3.00
ppm

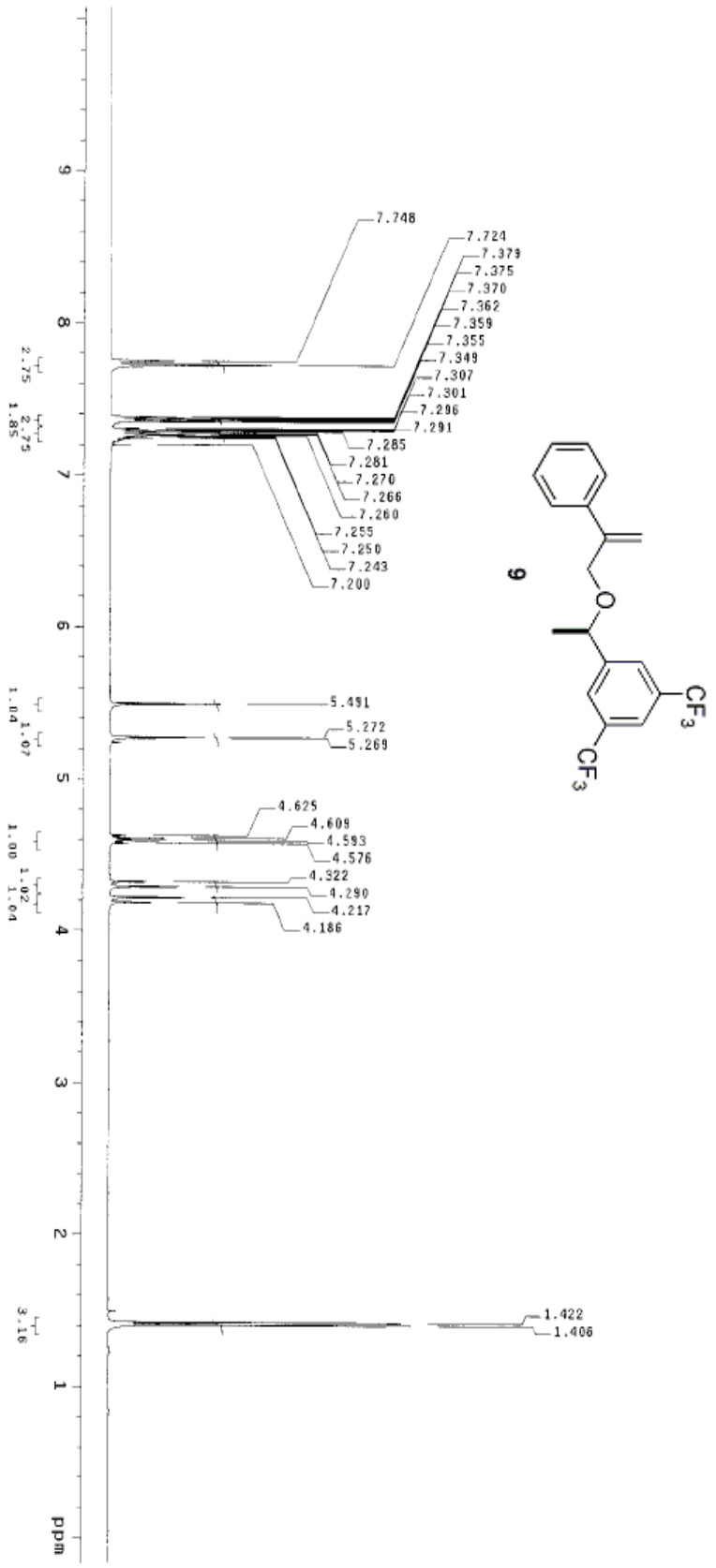

13C OBSERVE
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: wen-2-4083C
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
240 repetitions
OBSERVE C13, 100.6073320 MHz
DECOUPLE H1, 400.1102274 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 59 min, 46 sec
162.392
147.149
128.228
126.607
124.840
59.589
54.144
40.139
39.939
39.720
39.520
39.320
39.101
38.901
28.721
180
160
140
120
100
80
60
40
20
ppm

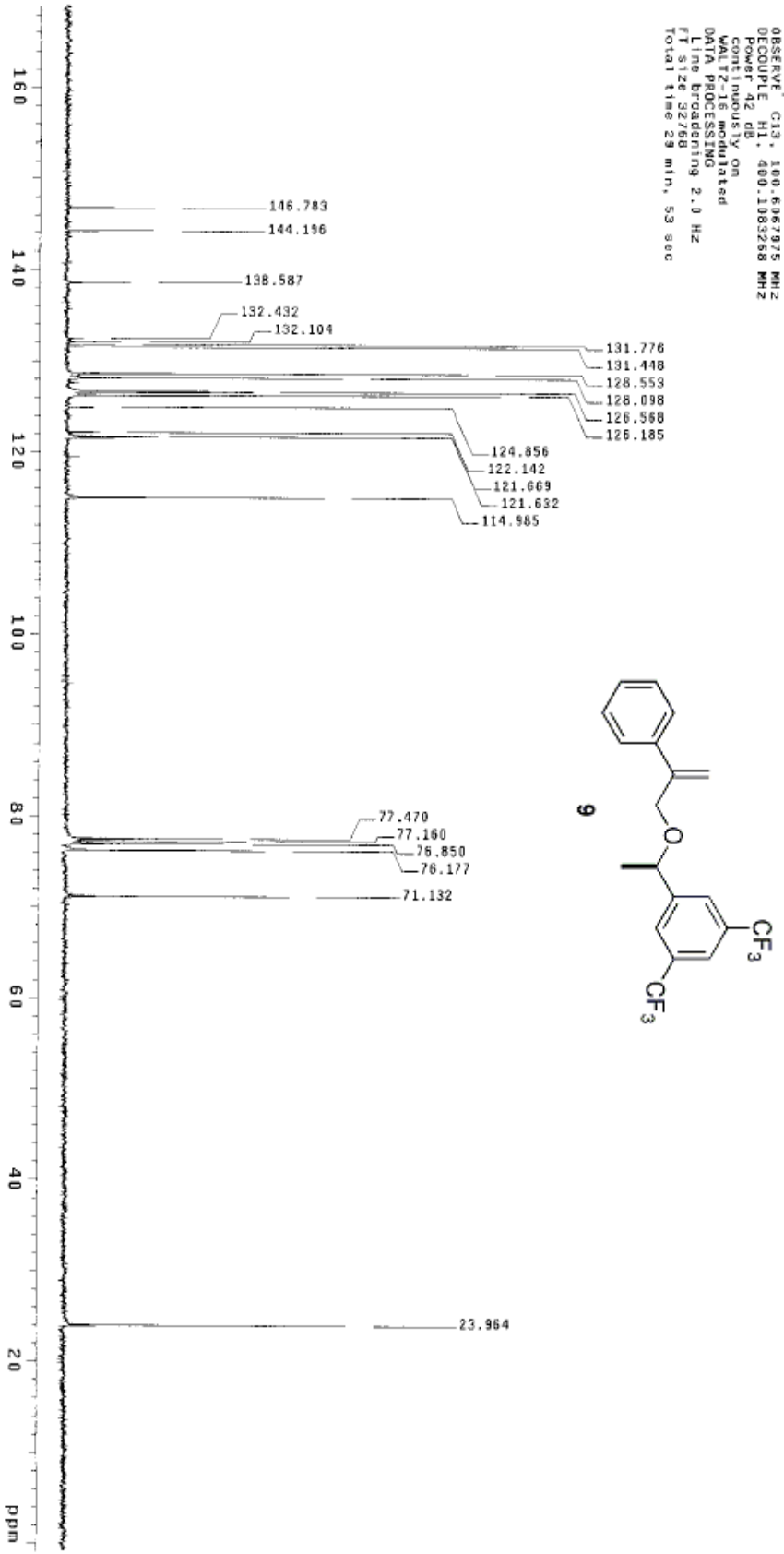

HN
O
NH

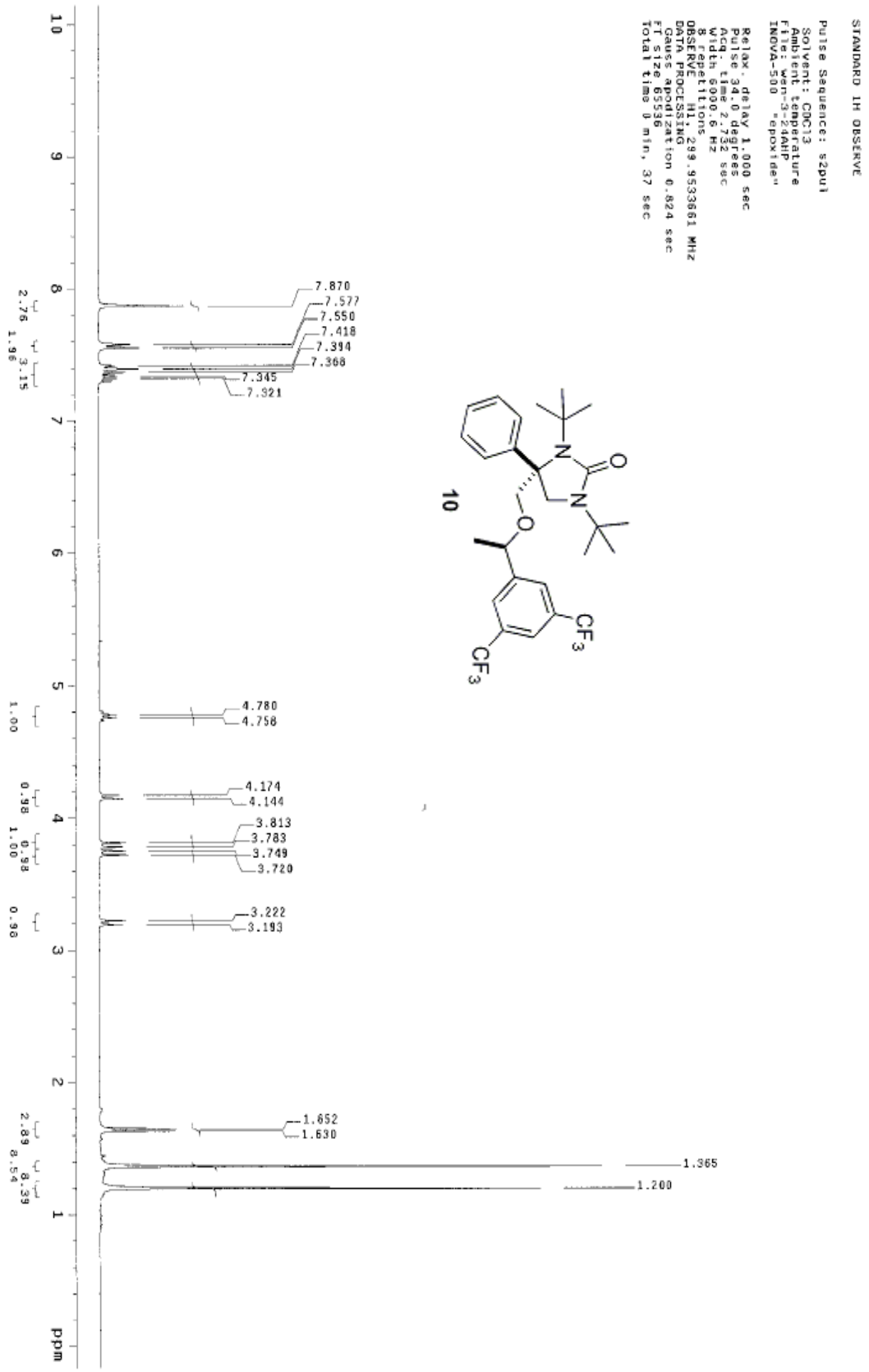
5a

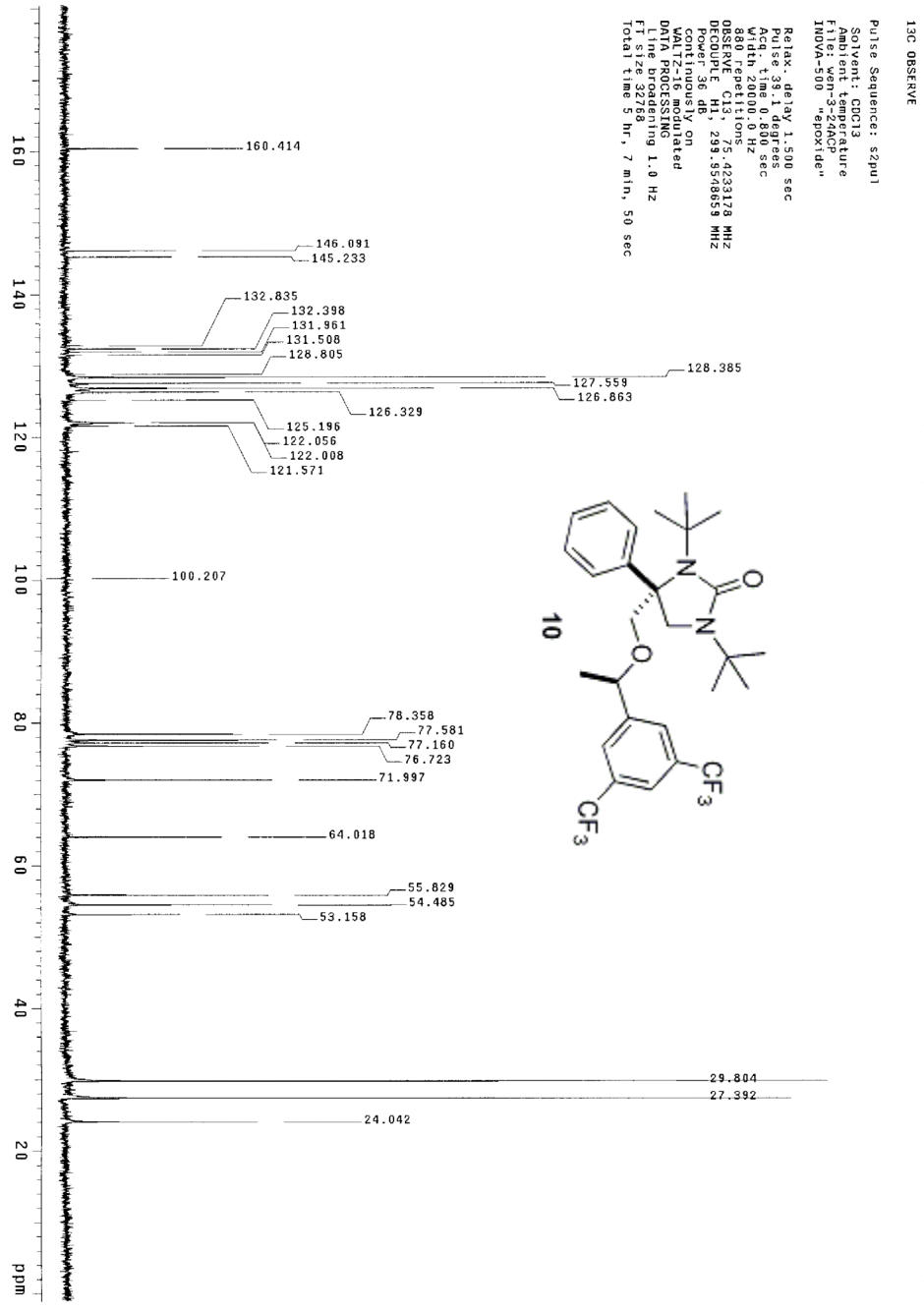

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-2-42B
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
8 repetitions
OBSERVE H1, 299.9533661 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 37 sec
7.447
7.420
7.350
7.326
7.299
7.232
7.228
7.207
7.183
1.870.99
1.89
2.927
2.886
2.742
2.705
1.00
1.01
1.411
1.336
1.242
3.10
3.53
ppm
6a
H2N
NH2


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: ryan-2-228C
INOVA-500 "epoxide"
Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
32 repetitions
OBSERVE C13, 75.4233348 MHz
DECOUPLE H1, 299.9540859 MHz
Power 56 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 30 min, 47 sec
147.094
128.287
126.361
125.504
77.581
77.160
76.723
56.071
54.744
28.752
6a
H2N
NH2
180
160
140
120
100
80
60
40
20
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-3-05AH
INOVA-500 "epoxide"
Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
8 repetitions
OBSERVE H1, 500.1063397 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 23 sec
7.748
7.724
7.379
7.375
7.370
7.362
7.359
7.355
7.349
7.307
7.301
7.296
7.291
7.285
7.281
7.270
7.266
7.260
7.255
7.250
7.243
7.200
5.491
5.272
5.269
4.625
4.609
4.593
4.576
4.322
4.290
4.217
4.186
1.422
1.406
2.75
2.75
1.85
1.04
1.07
1.00
1.02
1.04
3.16
ppm
9
CF3
CF3


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-3-05AC
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
280 repetitions
OBSERVE C13, 100.6067975 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 29 min, 53 sec
146.783
144.196
138.587
132.432
132.104
131.776
131.448
128.553
128.098
126.568
126.185
124.856
122.142
121.669
121.632
114.985
77.470
77.160
76.850
76.177
71.132
23.964
160
140
120
100
80
60
40
20
ppm
9
O
CF3
CF3

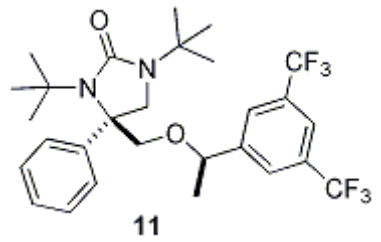

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-3-24AHP
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
width 6000.6 Hz
8 repetitions
OBSERVE H1, 299.9533661 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 37 sec
10
CF3
CF3
7.870
7.577
7.550
7.418
7.394
7.368
7.345
7.321
4.780
4.758
4.174
4.144
3.813
3.783
3.749
3.720
3.222
3.193
1.652
1.630
1.365
1.200
2.76
1.96
3.15
1.00
0.98
1.00
0.98
0.96
2.89
8.54
8.39
ppm


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-S-24ACP
INOVA-500 "epoxide"
Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 1.000 sec
Width 28000.8 Hz
880 repetitions
OBSERVE C13, 75.4233178 MHz
DECOUPLE H1, 299.9548659 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 5 hr, 7 min, 50 sec
160.414
146.091
145.233
132.835
132.398
131.961
131.508
128.805
128.385
127.559
126.863
126.329
125.196
122.056
122.008
121.571
100.207
78.358
77.581
77.160
76.723
71.997
64.018
55.829
54.485
53.158
29.804
27.392
24.042
160
140
120
100
80
60
40
20
ppm
10
N
O
N
O
CF3
CF3

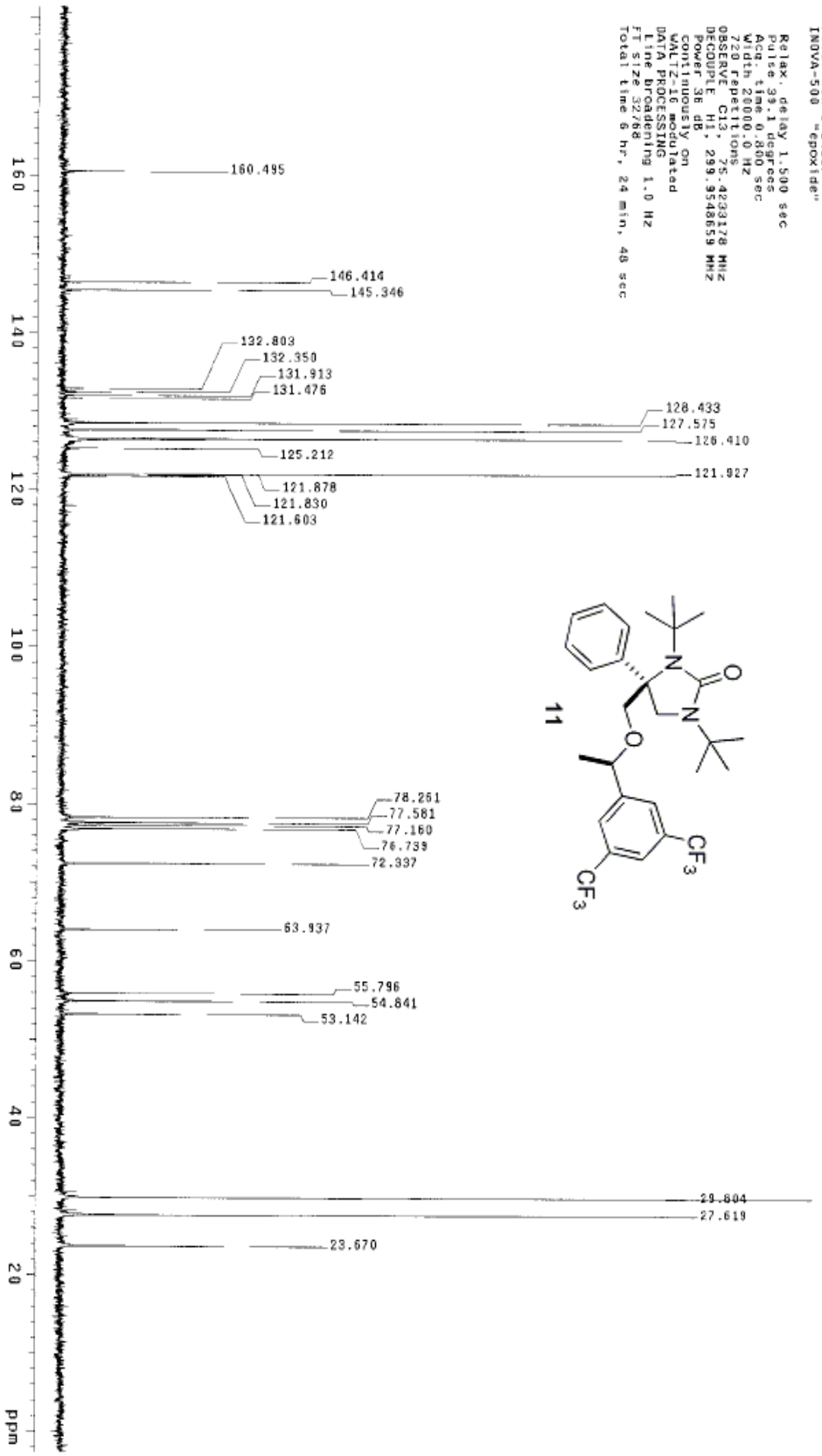
STANDARD 1H OBSERVE

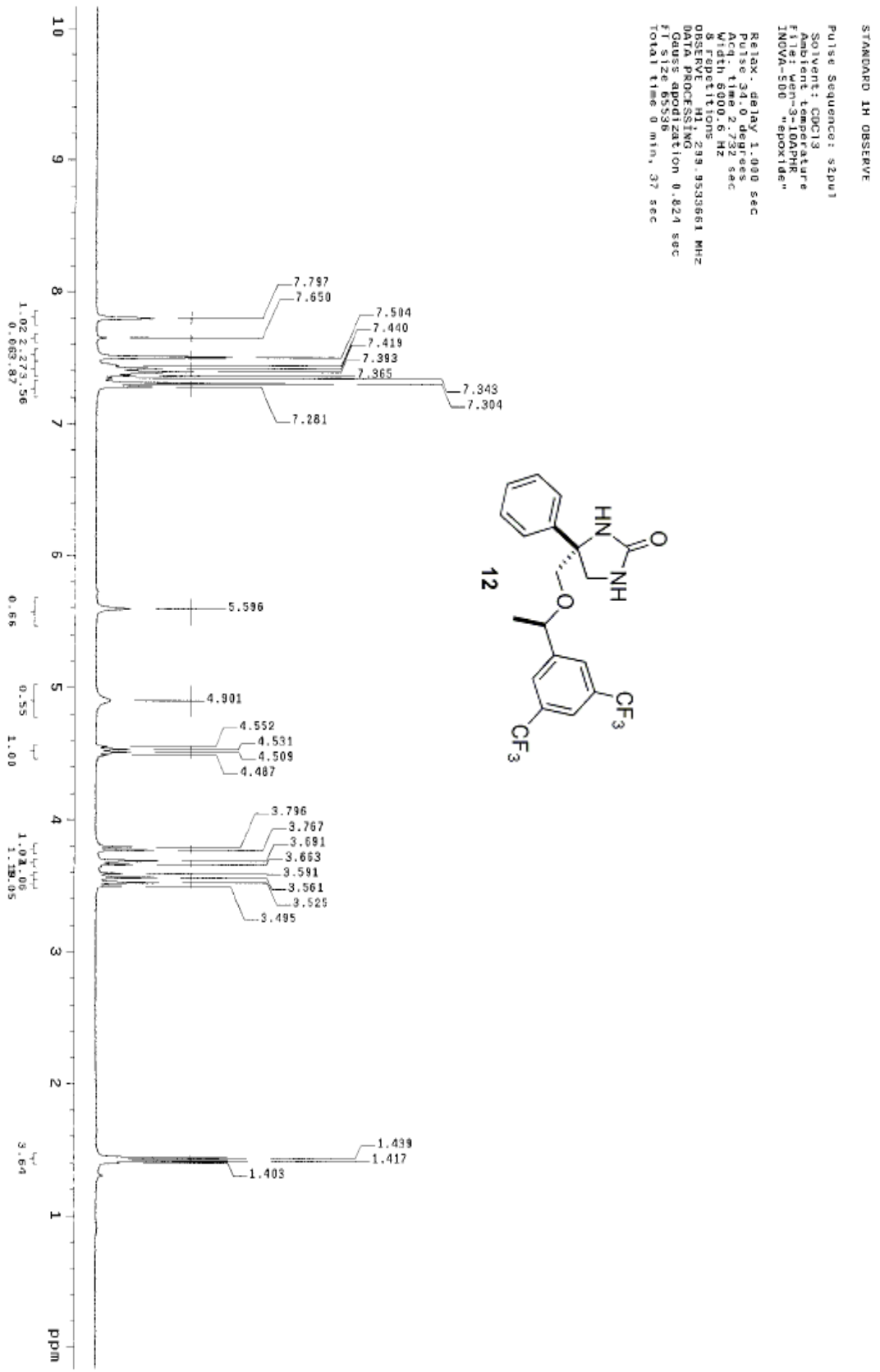
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-3-24BMP
INOVA-500 "epoxide"

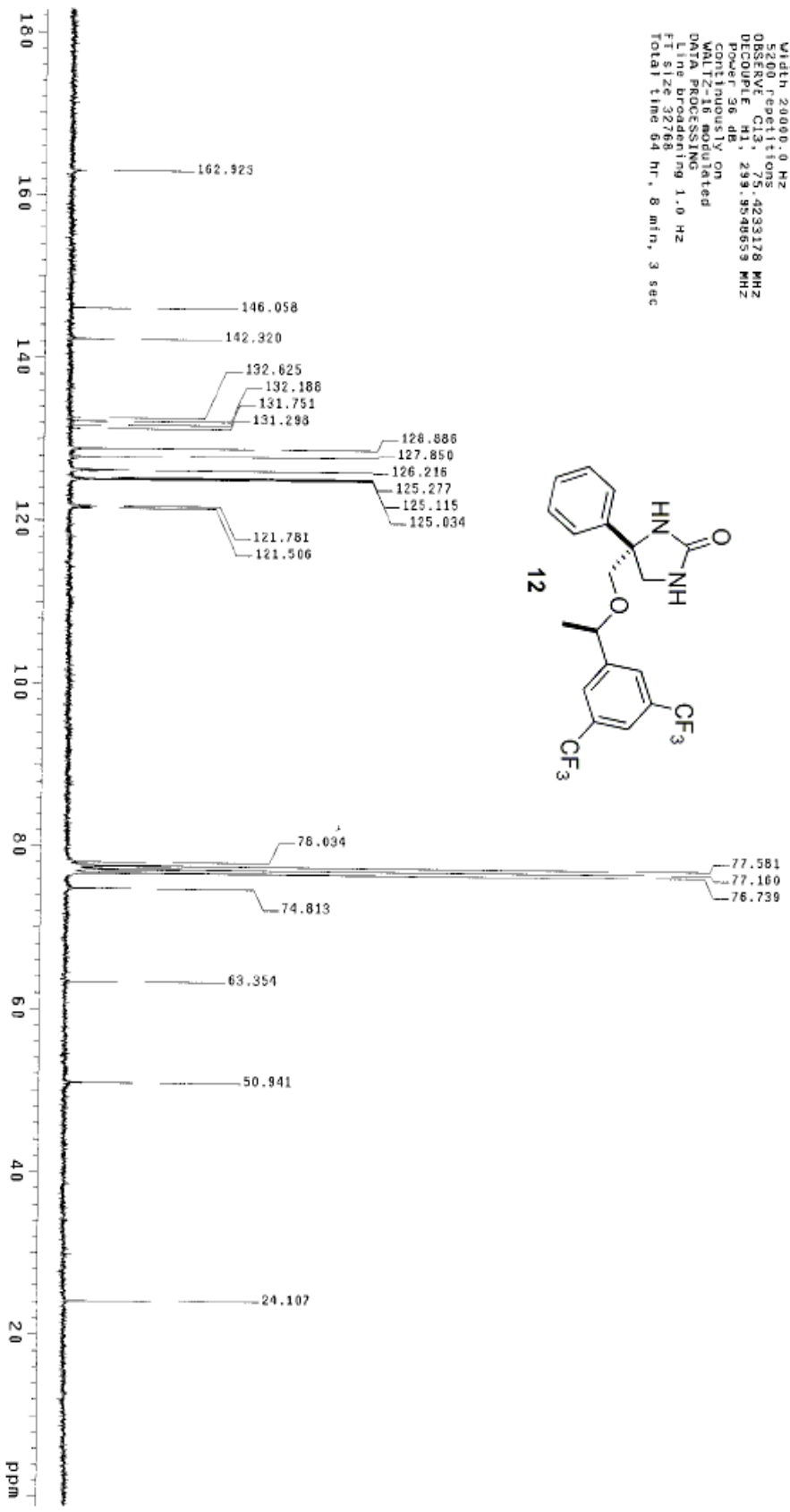
Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
8 repetitions
OBSERVE H1, 299.9533661 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 37 sec



11


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-3-48CP
INOVA-500 "epoxide"
Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 26000.0 Hz
720 repetitions
OBSERVE C13, 75.423178 MHz
DECOUPLE H1, 299.9548659 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 6 hr, 24 min, 48 sec
160.495
146.414
145.346
132.803
132.350
131.913
131.476
128.433
127.575
126.410
125.212
121.927
121.878
121.830
121.603
78.261
77.581
77.160
76.739
72.337
63.937
55.796
54.841
53.142
29.804
27.619
23.670
11
CF3
CF3
ppm

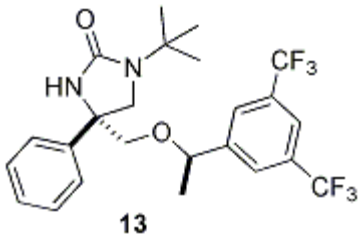

STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-3-10APHR
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 34.0 degrees
Acq. time 2.732 sec
Width 6000.6 Hz
8 repetitions
OBSERVE H1, 299.9533661 MHz
DATA PROCESSING
Gauss apodization 0.824 sec
FT size 65536
Total time 0 min, 37 sec
7.797
7.650
7.504
7.440
7.419
7.393
7.365
7.343
7.304
7.281
5.596
4.901
4.552
4.531
4.509
4.487
3.796
3.767
3.691
3.663
3.591
3.561
3.525
3.495
1.439
1.417
1.403
HN
NH
O
O
CF3
CF3
12
ppm


Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-3-100GC
INOVA-500 "epoxide"
Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.800 sec
Width 20000.0 Hz
5200 repetitions
OBSERVE C13, 75.4233178 MHz
DECOUPLE H1, 299.9548659 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 64 hr, 8 min, 3 sec
162.923
146.058
142.320
132.625
132.188
131.751
131.298
128.886
127.850
126.216
125.277
125.115
125.034
121.781
121.506
78.034
77.581
77.160
76.739
74.813
63.354
50.941
24.107
ppm
12

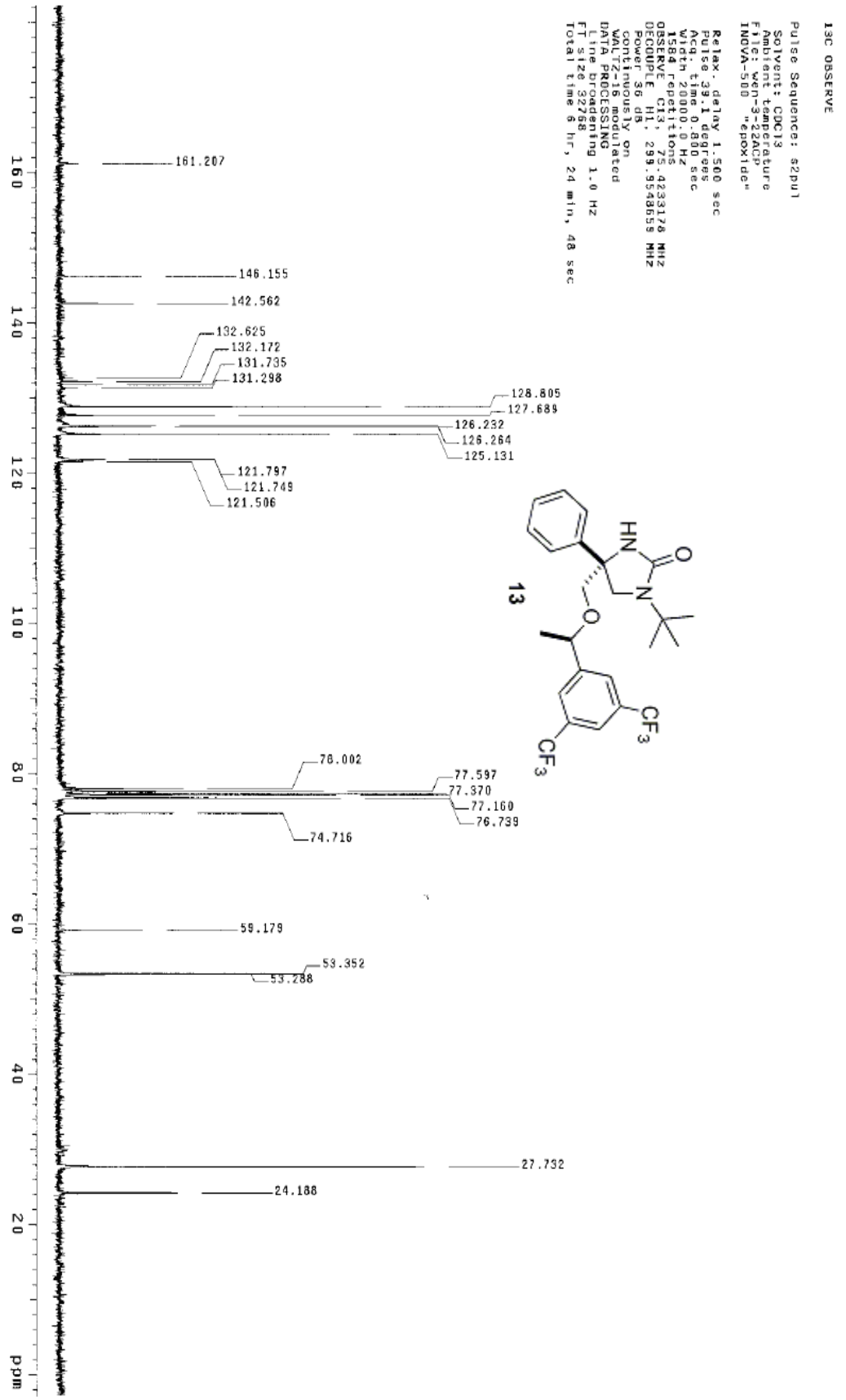
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
File: wen-S-22AHP2
INOVA-500 "epoxide"

 Relax. delay 0.000 sec
 Pulse 26.0 degrees
 Acq. time 2.668 sec
 Width 5995.2 Hz
 32 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
 Gauss apodization 0.896 sec
FT size 32768
Total time 1 min, 31 sec



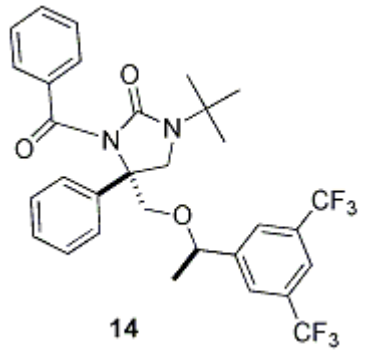
13


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-3-22ACP
INOVA-500 "epoxide"
Relax. delay 1.500 sec
Pulse 39.1 degrees
Acq. time 0.880 sec
Width 20000.0 Hz
1584 repetitions
OBSERVE C13, 75.4233178 MHz
DECOUPLE H1, 299.9548658 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 6 hr, 24 min, 48 sec
161.207
146.155
142.562
132.625
132.172
131.735
131.298
128.805
127.689
126.232
126.264
125.131
121.797
121.749
121.506
78.002
77.597
77.370
77.160
76.739
74.716
59.179
53.352
53.288
27.732
24.188
ppm
13

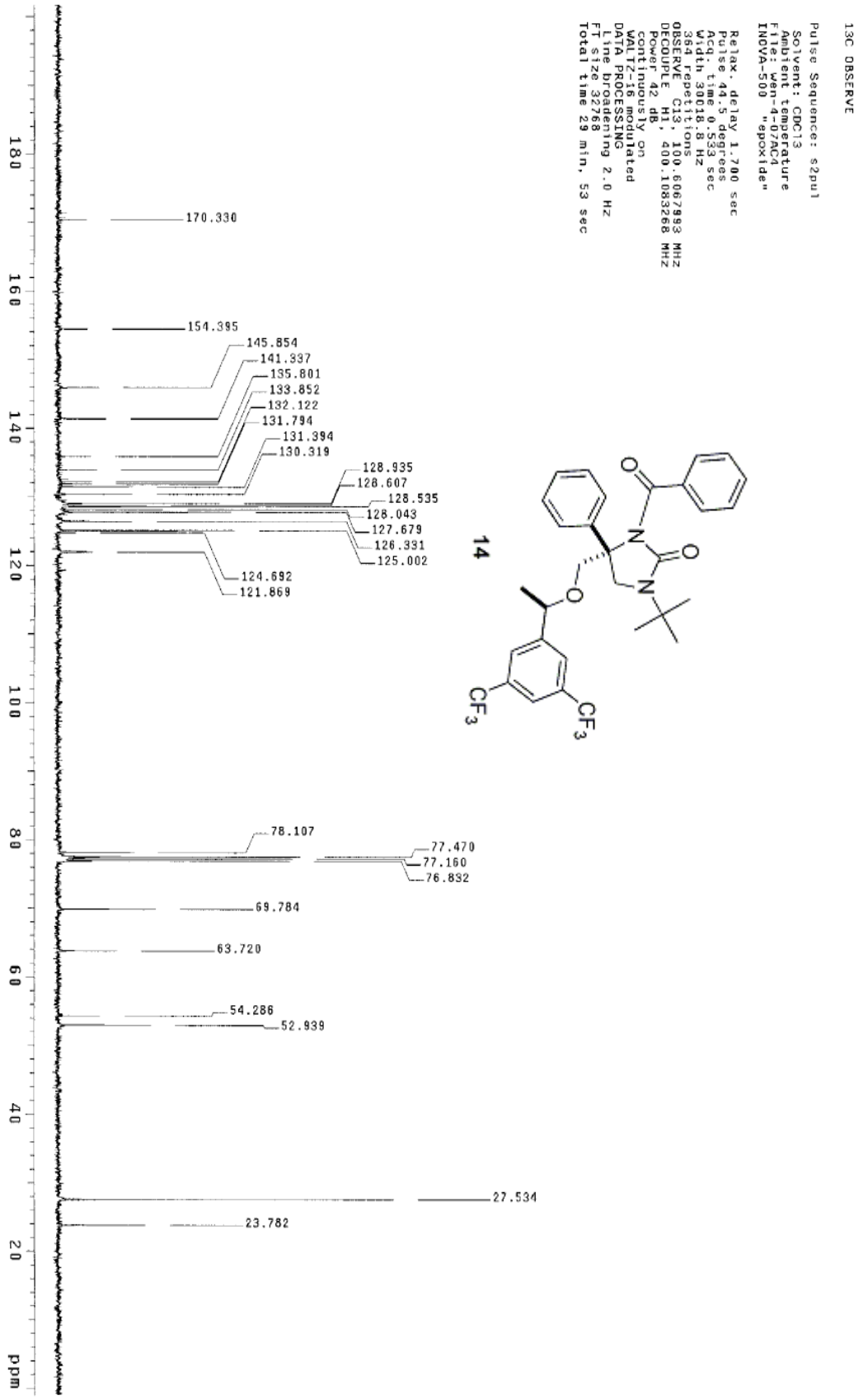
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-4-07A
INOVA-500 "epoxide"

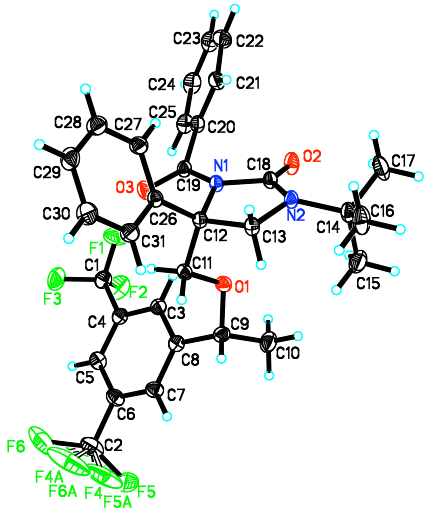
Pulse 31.0 degrees
Acq. time 2.291 sec
Width 6982.6 Hz
8 repetitions
OBSERVE H1, 400.1063250 MHz
DATA PROCESSING
Gauss apodization 0.971 sec
FT size 65536
Total time 0 min, 23 sec



14


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: wen-4-07AC4
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
364 repetitions
OBSERVE C13, 100.6067993 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 29 min, 53 sec
170.330
154.395
145.854
141.337
135.801
133.852
132.122
131.794
131.394
130.319
128.935
128.607
128.535
128.043
127.679
126.331
125.002
124.692
121.869
78.107
77.470
77.160
76.832
69.784
63.720
54.286
52.939
27.534
23.782
14
CF3
CF3
180
160
140
120
100
80
60
40
20
ppm

The X-ray structure of compound **14**



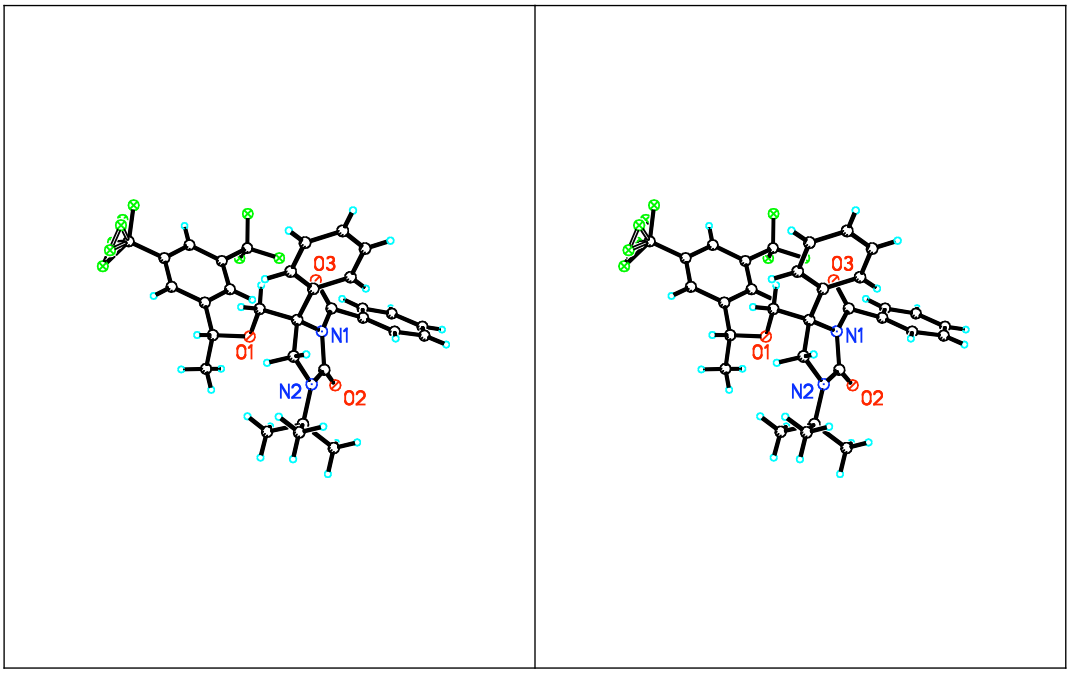


Table 1. Crystal data and structure refinement for **14**.

Identification code	ys190_0m	
Empirical formula	C31 H30 F6 N2 O3	
Formula weight	592.57	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 9.5388(6) Å	□= 90°.
	b = 14.5600(10) Å	□= 109.057(4)°.
	c = 10.9484(7) Å	□ = 90°.
Volume	1437.23(16) $Å^3$	
Z	2	
Density (calculated)	1.369 Mg/m^3	
Absorption coefficient	0.114 mm^{-1}	
F(000)	616	
Crystal size	0.52 x 0.34 x 0.24 mm^3	
Theta range for data collection	2.26 to 45.37°.	
Index ranges	-19<=h<=18, -22<=k<=28, -21<=l<=21	
Reflections collected	40750	
Independent reflections	18762 [R(int) = 0.0555]	
Completeness to theta = 45.37°	98.7 %	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9728 and 0.9433	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	18762 / 1 / 408	
Goodness-of-fit on F^2	0.967	
Final R indices [I>2sigma(I)]	R1 = 0.0562, wR2 = 0.1185	
R indices (all data)	R1 = 0.1293, wR2 = 0.1478	
Absolute structure parameter	-0.1(4)	
Extinction coefficient	0.0167(18)	
Largest diff. peak and hole	0.382 and -0.337 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)

for ys190_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	6812(1)	7867(1)	10838(1)	16(1)
N(2)	4784(1)	7863(1)	11440(1)	18(1)
O(1)	4867(1)	8660(1)	8719(1)	19(1)
O(2)	5466(1)	6505(1)	10675(1)	26(1)
O(3)	8443(1)	8044(1)	9734(1)	24(1)
F(1)	8541(1)	7593(1)	6800(1)	40(1)
F(2)	7622(1)	7563(1)	4731(1)	41(1)
F(3)	9255(1)	8567(1)	5656(1)	41(1)
F(4)	4655(10)	10724(3)	2884(3)	101(2)
F(4A)	5660(6)	10935(3)	3193(5)	66(2)
F(5)	3541(3)	10963(4)	3402(5)	83(2)
F(5A)	4215(7)	11504(3)	4334(5)	78(2)
F(6)	6357(4)	11405(3)	4242(6)	98(2)
F(6A)	5309(7)	11713(2)	4641(3)	81(2)
C(1)	8063(2)	8123(1)	5756(1)	25(1)
C(2)	5041(2)	10922(1)	4063(2)	34(1)
C(3)	6066(1)	8624(1)	6636(1)	20(1)
C(4)	6870(1)	8779(1)	5799(1)	20(1)
C(5)	6558(2)	9527(1)	4958(1)	22(1)
C(6)	5406(2)	10111(1)	4955(1)	22(1)
C(7)	4589(2)	9961(1)	5784(1)	22(1)
C(8)	4919(1)	9221(1)	6633(1)	19(1)
C(9)	4006(1)	9058(1)	7525(1)	20(1)
C(10)	2749(2)	8385(1)	6919(1)	30(1)
C(11)	5952(1)	9276(1)	9515(1)	17(1)
C(12)	6485(1)	8854(1)	10874(1)	16(1)
C(13)	5179(1)	8830(1)	11427(1)	18(1)
C(14)	3290(1)	7604(1)	11489(1)	21(1)
C(15)	2179(2)	7680(1)	10130(2)	33(1)

C(16)	2887(2)	8274(1)	12402(2)	33(1)
C(17)	3301(2)	6631(1)	12017(2)	37(1)
C(18)	5635(1)	7315(1)	10965(1)	18(1)
C(19)	7891(1)	7539(1)	10344(1)	18(1)
C(20)	8464(1)	6586(1)	10689(1)	18(1)
C(21)	8739(2)	6233(1)	11934(1)	23(1)
C(22)	9446(2)	5390(1)	12268(2)	28(1)
C(23)	9854(2)	4885(1)	11361(2)	30(1)
C(24)	9574(2)	5227(1)	10123(2)	28(1)
C(25)	8903(2)	6084(1)	9792(1)	24(1)
C(26)	7784(1)	9406(1)	11754(1)	17(1)
C(27)	9056(2)	8985(1)	12589(1)	22(1)
C(28)	10194(2)	9514(1)	13399(1)	25(1)
C(29)	10087(2)	10461(1)	13408(1)	27(1)
C(30)	8826(2)	10884(1)	12595(2)	29(1)
C(31)	7686(2)	10363(1)	11771(1)	24(1)

—

Table 3. Bond lengths [Å] and angles [°] for ys190_0m.

N(1)-C(19)	1.3936(15)
N(1)-C(18)	1.4239(15)
N(1)-C(12)	1.4746(15)
N(2)-C(18)	1.3574(16)
N(2)-C(13)	1.4583(16)
N(2)-C(14)	1.4922(15)
O(1)-C(9)	1.4208(15)
O(1)-C(11)	1.4311(15)
O(2)-C(18)	1.2179(16)
O(3)-C(19)	1.2223(15)
F(1)-C(1)	1.3296(17)
F(2)-C(1)	1.3385(17)
F(3)-C(1)	1.3433(18)
F(4)-C(2)	1.254(4)
F(4A)-C(2)	1.274(3)
F(5)-C(2)	1.377(3)
F(5A)-C(2)	1.256(3)
F(6)-C(2)	1.396(4)
F(6A)-C(2)	1.298(3)
C(1)-C(4)	1.4982(18)
C(2)-C(6)	1.500(2)
C(3)-C(4)	1.3923(17)
C(3)-C(8)	1.3965(18)
C(4)-C(5)	1.3941(19)
C(5)-C(6)	1.3880(19)
C(6)-C(7)	1.3936(18)
C(7)-C(8)	1.3904(19)
C(8)-C(9)	1.5234(17)
C(9)-C(10)	1.523(2)
C(11)-C(12)	1.5353(17)
C(12)-C(26)	1.5260(17)
C(12)-C(13)	1.5522(16)
C(14)-C(15)	1.523(2)
C(14)-C(17)	1.528(2)
C(14)-C(16)	1.533(2)

C(19)-C(20)	1.4942(17)
C(20)-C(25)	1.3939(17)
C(20)-C(21)	1.3989(18)
C(21)-C(22)	1.3901(19)
C(22)-C(23)	1.391(2)
C(23)-C(24)	1.386(2)
C(24)-C(25)	1.395(2)
C(26)-C(31)	1.3965(18)
C(26)-C(27)	1.3997(18)
C(27)-C(28)	1.389(2)
C(28)-C(29)	1.383(2)
C(29)-C(30)	1.385(2)
C(30)-C(31)	1.390(2)

C(19)-N(1)-C(18)	123.41(10)
C(19)-N(1)-C(12)	122.25(10)
C(18)-N(1)-C(12)	111.52(9)
C(18)-N(2)-C(13)	112.02(9)
C(18)-N(2)-C(14)	124.22(11)
C(13)-N(2)-C(14)	119.86(10)
C(9)-O(1)-C(11)	112.95(10)
F(1)-C(1)-F(2)	107.07(13)
F(1)-C(1)-F(3)	106.68(12)
F(2)-C(1)-F(3)	105.49(11)
F(1)-C(1)-C(4)	113.16(11)
F(2)-C(1)-C(4)	112.38(12)
F(3)-C(1)-C(4)	111.58(12)
F(4)-C(2)-F(5A)	113.7(4)
F(4)-C(2)-F(4A)	44.5(3)
F(5A)-C(2)-F(4A)	130.3(2)
F(4)-C(2)-F(6A)	130.6(3)
F(5A)-C(2)-F(6A)	47.6(3)
F(4A)-C(2)-F(6A)	106.6(3)
F(4)-C(2)-F(5)	64.2(5)
F(5A)-C(2)-F(5)	58.1(3)
F(4A)-C(2)-F(5)	105.3(4)
F(6A)-C(2)-F(5)	102.9(4)

F(4)-C(2)-F(6)	102.1(4)
F(5A)-C(2)-F(6)	103.4(4)
F(4A)-C(2)-F(6)	58.8(3)
F(6A)-C(2)-F(6)	57.3(3)
F(5)-C(2)-F(6)	141.7(2)
F(4)-C(2)-C(6)	114.6(2)
F(5A)-C(2)-C(6)	114.05(17)
F(4A)-C(2)-C(6)	115.60(19)
F(6A)-C(2)-C(6)	114.46(18)
F(5)-C(2)-C(6)	110.87(18)
F(6)-C(2)-C(6)	107.33(19)
C(4)-C(3)-C(8)	119.85(12)
C(3)-C(4)-C(5)	121.07(12)
C(3)-C(4)-C(1)	120.35(12)
C(5)-C(4)-C(1)	118.55(11)
C(6)-C(5)-C(4)	118.59(12)
C(5)-C(6)-C(7)	120.86(12)
C(5)-C(6)-C(2)	120.00(12)
C(7)-C(6)-C(2)	119.14(12)
C(8)-C(7)-C(6)	120.30(12)
C(7)-C(8)-C(3)	119.31(11)
C(7)-C(8)-C(9)	119.84(11)
C(3)-C(8)-C(9)	120.83(11)
O(1)-C(9)-C(10)	105.93(11)
O(1)-C(9)-C(8)	111.93(10)
C(10)-C(9)-C(8)	110.78(11)
O(1)-C(11)-C(12)	106.50(9)
N(1)-C(12)-C(26)	113.26(9)
N(1)-C(12)-C(11)	111.73(9)
C(26)-C(12)-C(11)	110.15(9)
N(1)-C(12)-C(13)	101.08(9)
C(26)-C(12)-C(13)	110.82(9)
C(11)-C(12)-C(13)	109.47(9)
N(2)-C(13)-C(12)	105.59(9)
N(2)-C(14)-C(15)	108.36(11)
N(2)-C(14)-C(17)	111.07(11)
C(15)-C(14)-C(17)	110.70(13)

N(2)-C(14)-C(16)	107.92(11)
C(15)-C(14)-C(16)	110.32(13)
C(17)-C(14)-C(16)	108.42(12)
O(2)-C(18)-N(2)	128.60(11)
O(2)-C(18)-N(1)	124.26(11)
N(2)-C(18)-N(1)	107.14(10)
O(3)-C(19)-N(1)	120.72(11)
O(3)-C(19)-C(20)	120.64(10)
N(1)-C(19)-C(20)	118.37(10)
C(25)-C(20)-C(21)	119.34(12)
C(25)-C(20)-C(19)	118.58(11)
C(21)-C(20)-C(19)	121.60(11)
C(22)-C(21)-C(20)	120.10(13)
C(21)-C(22)-C(23)	120.26(14)
C(24)-C(23)-C(22)	119.90(13)
C(23)-C(24)-C(25)	120.11(13)
C(20)-C(25)-C(24)	120.24(13)
C(31)-C(26)-C(27)	118.36(11)
C(31)-C(26)-C(12)	119.32(11)
C(27)-C(26)-C(12)	122.27(11)
C(28)-C(27)-C(26)	120.33(12)
C(29)-C(28)-C(27)	120.84(13)
C(28)-C(29)-C(30)	119.31(13)
C(29)-C(30)-C(31)	120.34(13)
C(30)-C(31)-C(26)	120.81(13)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for ys190_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	16(1)	11(1)	23(1)	0(1)	10(1)	0(1)
N(2)	16(1)	14(1)	26(1)	0(1)	11(1)	0(1)
O(1)	20(1)	16(1)	19(1)	0(1)	5(1)	-1(1)
O(2)	24(1)	13(1)	46(1)	-4(1)	19(1)	-3(1)
O(3)	25(1)	20(1)	32(1)	5(1)	18(1)	2(1)
F(1)	45(1)	43(1)	40(1)	14(1)	23(1)	25(1)
F(2)	39(1)	42(1)	44(1)	-19(1)	16(1)	7(1)
F(3)	24(1)	43(1)	64(1)	4(1)	22(1)	3(1)
F(4)	217(7)	49(2)	26(1)	14(1)	23(3)	43(3)
F(4A)	111(3)	44(2)	76(3)	34(2)	78(3)	37(2)
F(5)	37(1)	111(4)	84(3)	76(3)	-2(2)	1(2)
F(5A)	130(4)	43(2)	99(4)	43(2)	91(4)	54(2)
F(6)	92(3)	58(3)	157(5)	61(3)	59(3)	3(2)
F(6A)	174(5)	22(1)	38(1)	4(1)	23(2)	5(2)
C(1)	23(1)	27(1)	28(1)	0(1)	13(1)	4(1)
C(2)	50(1)	25(1)	37(1)	8(1)	26(1)	9(1)
C(3)	19(1)	19(1)	22(1)	1(1)	7(1)	1(1)
C(4)	19(1)	20(1)	20(1)	-2(1)	7(1)	0(1)
C(5)	26(1)	20(1)	22(1)	-2(1)	11(1)	0(1)
C(6)	29(1)	19(1)	23(1)	1(1)	12(1)	2(1)
C(7)	24(1)	19(1)	23(1)	1(1)	9(1)	4(1)
C(8)	19(1)	19(1)	19(1)	-1(1)	7(1)	0(1)
C(9)	18(1)	22(1)	21(1)	2(1)	8(1)	4(1)
C(10)	21(1)	43(1)	24(1)	1(1)	5(1)	-7(1)
C(11)	19(1)	13(1)	20(1)	1(1)	7(1)	-1(1)
C(12)	16(1)	12(1)	21(1)	-1(1)	8(1)	1(1)
C(13)	18(1)	14(1)	25(1)	-2(1)	11(1)	-1(1)
C(14)	18(1)	17(1)	34(1)	1(1)	16(1)	0(1)
C(15)	20(1)	34(1)	44(1)	-1(1)	9(1)	-4(1)
C(16)	34(1)	29(1)	47(1)	-8(1)	30(1)	-4(1)

C(17)	39(1)	25(1)	59(1)	12(1)	34(1)	1(1)
C(18)	17(1)	14(1)	24(1)	0(1)	10(1)	-1(1)
C(19)	16(1)	16(1)	23(1)	0(1)	9(1)	1(1)
C(20)	16(1)	14(1)	27(1)	-1(1)	10(1)	0(1)
C(21)	23(1)	19(1)	29(1)	3(1)	13(1)	3(1)
C(22)	28(1)	19(1)	37(1)	6(1)	12(1)	4(1)
C(23)	24(1)	16(1)	51(1)	2(1)	14(1)	3(1)
C(24)	26(1)	20(1)	43(1)	-7(1)	17(1)	2(1)
C(25)	23(1)	20(1)	31(1)	-5(1)	13(1)	0(1)
C(26)	18(1)	14(1)	19(1)	-1(1)	8(1)	-1(1)
C(27)	22(1)	18(1)	23(1)	0(1)	5(1)	0(1)
C(28)	23(1)	26(1)	24(1)	0(1)	4(1)	0(1)
C(29)	26(1)	25(1)	28(1)	-5(1)	5(1)	-8(1)
C(30)	31(1)	17(1)	36(1)	-5(1)	6(1)	-5(1)
C(31)	23(1)	15(1)	32(1)	0(1)	5(1)	0(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for ys190_0m.

	x	y	z	U(eq)
H(3A)	6297	8114	7208	24
H(5A)	7122	9635	4400	26
H(7A)	3803	10367	5769	26
H(9A)	3584	9655	7693	24
H(10A)	2176	8291	7506	44
H(10B)	3162	7797	6764	44
H(10C)	2100	8634	6097	44
H(11A)	5509	9889	9533	21
H(11B)	6792	9345	9179	21
H(13A)	5488	9088	12311	22
H(13B)	4327	9189	10873	22
H(15A)	2184	8308	9811	49
H(15B)	1184	7530	10149	49
H(15C)	2452	7250	9557	49
H(16A)	2876	8902	12077	49
H(16B)	3624	8228	13267	49
H(16C)	1904	8121	12445	49
H(17A)	3556	6193	11442	55
H(17B)	2317	6484	12063	55
H(17C)	4038	6592	12882	55
H(21A)	8442	6569	12551	27
H(22A)	9651	5159	13120	33
H(23A)	10324	4305	11589	36
H(24A)	9840	4878	9499	34
H(25A)	8745	6326	8952	28
H(27A)	9142	8335	12601	26
H(28A)	11058	9221	13954	30
H(29A)	10870	10818	13966	32
H(30A)	8740	11534	12601	35
H(31A)	6830	10660	11213	29