A Pd(0)-Catalyzed Diamination of Terminal Olefins at Allylic and Homoallylic Carbons via Formal C-H Activation under Solvent-Free Conditions

Haifeng Du, Weicheng Yuan, Baoguo Zhao, and Yian Shi*

Department of Chemistry
Colorado State University
Fort Collins, CO 80523

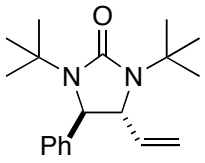
Supporting Information

Representative diamination procedure (Table 1, entry 6). A 1.5-mL vial charged with $Pd(PPh_3)_4$ (0.0924 g, 0.08 mmol) was evacuated and then filled with argon followed by addition of 1,5-hexadiene (0.131 g, 1.6 mmol). The resulting mixture was immersed into an oil bath (65 °C) with stirring. Di-*t*-butyldiaziridinone (0.748 g, 4.4 mmol) was added by syringe pump at the rate of 0.4 mmol/h. Upon completion of addition (11 h), the reaction mixture was stirred for another hour and purified by flash chromatography (silica gel, hexane:ethyl ether = 5:1) to give the product as a colorless oil (0.303 g, 76% yield).

Deprotection procedure (Scheme 3). A 3.0-mL vial charged with compound **5a** (0.240 g, 0.8 mmol) and CF_3CO_2H (1.6 mL) was heated at 80 °C with stirring for 1 h. The solvent was then removed under reduced pressure, and the resulting residue was transferred to a flask and conc. HCl (20 mL) was added. Upon stirring at reflux for 24 h, the reaction mixture was concentrated under reduced pressure, diluted with water (10 mL), and washed with CH_2Cl_2 (3x10 mL). The aqueous solution was adjusted to basic (pH >12) with NaOH (1N) and extracted with CH_2Cl_2 (3x10 mL). The organic layers were dried over Na_2SO_4, filtered, and concentrated to give diamine **6** as dark yellow oil (0.1084 g, 84% yield).

Representative bisdiamination procedure (Scheme 4). A 1.5-mL vial charged with $Pd(PPh_3)_4$ (0.0924 g, 0.08 mmol) was evacuated and then filled with argon followed by addition of 1,7-octadiene (0.088 g, 0.8 mmol). The resulting mixture was immersed into an oil bath (65 °C) with stirring. Di-*t*-butyldiaziridinone (0.748 g, 4.4 mmol) was added by syringe pump at rate of 0.4 mmol/h. Upon completion of addition (11 h), the reaction mixture was stirred for another hour and purified by flash chromatography (silica gel, hexane:ethyl acetate = 5:1) to give the product as a white solid (0.167 g, 47% yield).

Table 1, Entry 1



Colorless oil; IR (film) 1689 cm^{-1}; ^1H NMR (300 MHz, benzene-d_6) δ 7.27 (d, J = 7.2 Hz, 2H), 7.16-7.02 (m, 3H), 5.92 (ddd, J = 17.4, 9.9, 8.4 Hz, 1H), 4.91 (d, J = 17.4 Hz, 1H), 4.88 (d, J = 9.9 Hz, 1H), 4.05 (s, 1H), 3.55 (d, J = 8.4 Hz, 1H), 1.39 (s, 9H), 1.35 (s, 9H); ^{13}C NMR (75 MHz, benzene-d_6) δ 159.2, 145.0, 141.7, 129.4, 126.5, 115.6, 65.5, 63.8, 53.9, 53.7, 29.2, 29.1.

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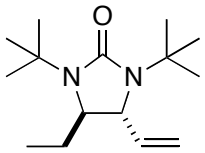
Table 1, Entry 2



Colorless oil; IR (film) 1689 cm^{-1}; ^1H NMR (300 MHz, benzene-d_6) δ 5.86 (ddd, J = 17.1, 9.9, 8.1 Hz, 1H), 4.97 (d, J = 17.1 Hz, 1H), 4.86 (d, J = 9.9 Hz, 1H), 3.49 (d, J = 8.1 Hz, 1H), 2.89 (dd, J = 8.4, 2.4 Hz, 1H), 1.54-1.22 (m, 2H), 1.44 (s, 9H), 1.39 (s, 9H), 0.72 (t, J = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, benzene-d_6) δ 158.2, 141.6, 115.0, 61.2, 60.7, 53.4, 52.8, 29.3, 29.2, 27.7, 9.4.

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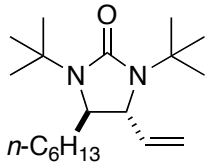
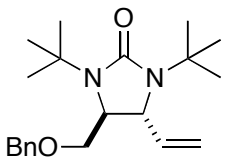
Table 1, Entry 3



Colorless oil; IR (film) 1690 cm^{-1}; ^1H NMR (300 MHz, benzene-d_6) δ 5.90 (ddd, J = 17.4, 10.2, 8.1 Hz, 1H), 5.03 (dd, J = 17.4 Hz, 1H), 4.88 (dd, J = 10.2 Hz, 1H), 3.59 (d, J = 8.1 Hz, 1H), 3.04 (dd, J = 8.4, 1.8 Hz, 1H), 1.70-1.50 (m, 2H), 1.47 (s, 9H), 1.43 (s, 9H), 1.40-1.10 (m, 8H), 0.91 (t, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, benzene-d_6) δ 158.2, 141.6, 115.1, 61.2, 60.1, 53.4, 52.8, 34.9, 32.5, 30.1, 29.4, 29.3, 25.4, 23.4, 14.7; Anal. Calcd for $C_{19}H_{36}N_2O$: C, 73.97; H,

11.76; N, 9.08; Found: C, 73.68; H, 11.96; N, 9.30; HRMS Calcd for $C_{19}H_{37}N_2O$ (M+1): 309.2906; Found: 309.2906.

Table 1, Entry 4



Colorless oil; IR (film) 1689 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.41-7.27 (m, 5H), 5.91 (ddd, *J* = 17.4, 10.2, 8.1 Hz, 1H), 5.26 (d, *J* = 17.4 Hz, 1H), 5.13 (d, *J* = 10.2 Hz, 1H), 4.60 (d, *J* = 12.0 Hz, 1H), 4.48 (d, *J* = 12.0 Hz, 1H), 3.97 (d, *J* = 8.1 Hz, 1H), 3.50-3.30 (m, 3H), 1.32 (s, 9H), 1.29 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.4, 139.7, 137.9, 128.6, 128.0, 127.9, 115.8, 73.3, 70.0, 58.7, 58.5, 53.0, 52.7, 29.1, 28.8.

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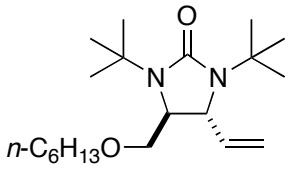
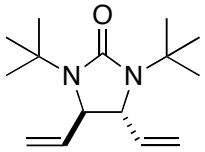
Table 1, Entry 5



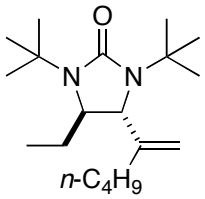
Colorless oil; IR (film) 1693 cm^{-1}; ^1H NMR (300 MHz, benzene-d_6) δ 5.94 (ddd, *J* = 17.1, 10.5, 8.1 Hz, 1H), 5.20 (d, *J* = 17.1 Hz, 1H), 4.93 (d, *J* = 10.5 Hz, 1H), 4.07 (d, *J* = 8.1 Hz, 1H), 3.40-3.17 (m, 5H), 1.54-1.46 (m, 2H), 1.48 (s, 9H), 1.40 (s, 9H), 1.38-1.16 (m, 6H), 0.89 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (75 MHz, benzene-d_6) δ 158.4, 140.9, 115.7, 71.9, 71.8, 59.4, 59.2, 53.4, 52.9, 32.3, 30.3, 29.3, 29.2, 26.6, 23.4, 14.6; HRMS Calcd for $C_{20}H_{39}N_2O_2$ (M+1): 339.3011; Found: 339.3015.

Table 1, Entry 6



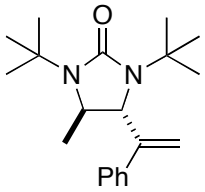
Colorless oil; IR (film) 1691 cm^{-1}; ^1H NMR (300 MHz, benzene-d_6) δ 5.88 (ddd, J = 17.1, 10.2, 8.4 Hz, 2H), 4.95 (dd, J = 17.1, 1.2 Hz, 2H), 4.86 (dd, J = 10.2, 1.2 Hz, 2H), 3.47 (d, J = 8.4 Hz, 2H), 1.42 (s, 18H); ^{13}C NMR (75 MHz, benzene-d_6) δ 158.3, 140.4, 116.0, 63.4, 53.6, 29.1; Anal. Calcd for $C_{15}H_{26}N_2O$: C, 71.95; H, 10.47; N, 11.19; Found: C, 71.79; H, 10.70; N, 11.40; HRMS Calcd for $C_{15}H_{27}N_2O$ (M+1): 251.2123; Found: 251.2125.

Table 1, Entry 7



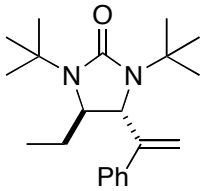
Colorless oil; IR (film) 1688 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.00 (s, 1H), 4.85 (s, 1H), 3.61 (s, 1H), 3.02 (dd, J = 8.4, 2.4 Hz, 1H), 2.10-2.00 (m, 2H), 1.70-1.20 (m, 6H), 1.40 (s, 18H), 0.98-0.86 (m, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.8, 150.8, 109.4, 61.4, 60.5, 53.2, 52.4, 31.2, 29.8, 29.0, 28.8, 28.7, 22.8, 14.1, 8.8; Anal. Calcd for $C_{19}H_{36}N_2O$: C, 73.97; H, 11.76; N, 9.08; Found: C, 74.13; H, 11.36; N, 9.25; HRMS Calcd for $C_{19}H_{37}N_2O$ (M+1): 309.2906; Found: 309.2901.

Table 1, Entry 8



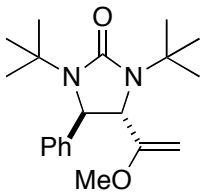
White solid; mp. 66-67 °C; IR (film) 1687 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.40-7.30 (m, 5H), 5.43 (s, 1H), 5.35 (s, 1H), 4.04 (s, 1H), 3.27 (q, *J* = 6.0 Hz, 1H), 1.43 (s, 9H), 1.32 (s, 9H), 1.24 (d, *J* = 6.0 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.6, 148.7, 139.7, 128.8, 128.0, 126.5, 113.0, 62.4, 55.4, 53.4, 52.4, 29.1, 29.0, 23.3; HRMS Calcd for $C_{20}H_{31}N_2O$ (M+1): 315.2436; Found: 315.2440.

Table 1, Entry 9



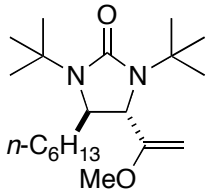
White solid; mp. 65-66 °C; IR (film) 1686 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.40-7.30 (m, 5H), 5.35 (s, 1H), 5.33 (s, 1H), 4.16 (s, 1H), 3.04 (dd, *J* = 7.5, 3.9 Hz, 1H), 1.60-1.48 (m, 2H), 1.45 (s, 9H), 1.32 (s, 9H), 0.75 (t, *J* = 7.5 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.8, 149.8, 139.7, 128.6, 127.9, 126.9, 113.2, 60.4, 59.6, 53.3, 52.2, 28.9, 28.6, 8.6; Anal. Calcd for $C_{21}H_{32}N_2O$: C, 76.78; H, 9.82; N, 8.53; Found: C, 77.18; H, 9.74; N, 8.31; HRMS Calcd for $C_{21}H_{33}N_2O$ (M+1): 329.2593; Found: 329.2589.

Table 1, Entry 10



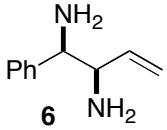
Colorless oil; IR (film) 1691 cm^{-1}; ^1H NMR (300 MHz, benzene-d_6) δ 7.42-7.36 (m, 2H), 7.22-7.14 (m, 2H), 7.10-7.04 (m, 1H), 4.37 (d, *J* = 1.5 Hz, 1H), 4.18 (d, *J* = 2.4 Hz, 1H), 3.83 (d, *J* = 2.4 Hz, 1H), 3.67 (d, *J* = 1.5 Hz, 1H), 3.17 (s, 3H), 1.42 (s, 9H), 1.41 (s, 9H); ^{13}C NMR (75 MHz, benzene-d_6) δ 166.3, 159.8, 146.3, 129.5, 128.2, 126.1, 81.5, 65.0, 62.9, 55.2, 53.9, 53.7, 29.2, 28.9; Anal. Calcd for $C_{20}H_{30}N_2O_2$: C, 72.69; H, 9.15; N, 8.48; Found: C, 72.60; H, 9.29; N, 8.40; HRMS Calcd for $C_{20}H_{31}N_2O_2$ (M+1): 331.2386; Found: 331.2376.

Table 1, Entry 11



Colorless oil; IR (film) 1691 cm^{-1}; ^{1}H NMR (300 MHz, benzene-d_6) δ 4.31 (d, J = 1.8 Hz, 1H), 3.85 (d, J = 1.8 Hz, 1H), 3.69 (s, 1H), 3.31 (dd, J = 6.3, 4.2 Hz, 1H), 3.13 (s, 3H), 1.62-1.38 (m, 2H), 1.51 (s, 9H), 1.47 (s, 9H), 1.36-1.16 (m, 8H), 0.90 (t, J = 6.6 Hz, 3H); ^{13}C NMR (75 MHz, benzene-d_6) δ 166.3, 158.9, 80.9, 60.6, 59.5, 55.1, 53.5, 52.9, 37.0, 32.6, 30.0, 29.3, 29.1, 25.0, 23.4, 14.7; HRMS Calcd for $C_{20}H_{39}N_2O_2$ (M+1): 339.3011; Found: 339.3007.

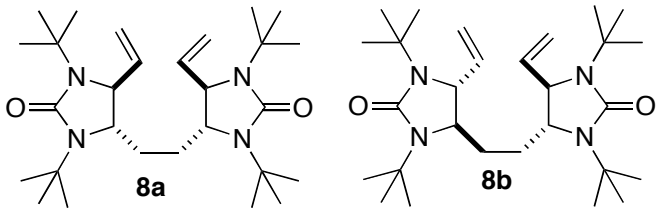
Scheme 3



6

Dark yellow oil; IR (film) 3363, 3300 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.20 (m, 5H), 5.80 (ddd, J = 16.8, 10.5, 6.0 Hz, 1H), 5.18 (d, J = 16.8 Hz, 1H), 5.18 (d, J = 10.5 Hz, 1H), 3.88 (d, J = 5.7 Hz, 1H), 3.50 (dd, J = 6.0, 5.7 Hz, 1H), 1.46 (bs, 4H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.6, 140.3, 128.5, 127.3, 127.2, 115.3, 60.5, 59.9; HRMS Calcd. for $C_{10}H_{15}N_2$ (M+1): 163.1230; Found: 163.1223; HRMS Calcd. for $C_{10}H_{12}N$ (M-NH_2): 146.0964; Found: 146.0967.

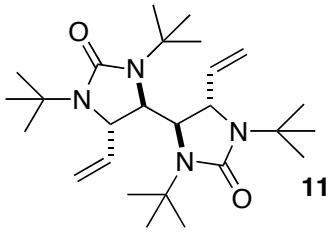
Scheme 4



8a **8b**

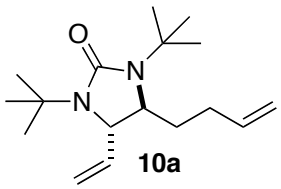
Mixture of **8a** and **8b** (**8a/8b** = 1/1): IR (film) 1688 cm^{-1}; Anal. Calcd for $C_{28}H_{50}N_4O_2$: C, 70.84; H, 10.62; N, 11.80; Found: C, 71.00; H, 10.60; N, 11.63; HRMS Calcd for $C_{28}H_{51}N_4O_2$ (M+1): 475.4012; Found: 475.4012. **8a**: white solid; mp. 160-161 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.92 (ddd, *J* = 17.1, 10.2, 7.8 Hz, 2H), 5.19 (d, *J* = 17.1 Hz, 2H), 5.13 (d, *J* = 10.2 Hz, 2H), 3.63 (d, *J* = 7.8 Hz, 2H), 3.20-3.10 (m, 2H), 1.64-1.58 (m, 4H), 1.40 (s, 18H), 1.35 (s, 18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.2, 140.3, 115.7, 61.2, 59.3, 53.3, 52.9, 29.6, 29.2, 29.0; **8b**: white solid; mp. 161-162 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.92 (ddd, *J* = 17.1, 10.2, 8.7 Hz, 2H), 5.20 (d, *J* = 17.1 Hz, 2H), 5.13 (d, *J* = 10.2 Hz, 2H), 3.65 (d, *J* = 8.7 Hz, 2H), 3.14-3.11 (m, 2H), 1.73-1.56 (m, 2H), 1.55-1.40 (m, 2H), 1.38 (s, 18H), 1.36 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.2, 140.2, 115.8, 61.0, 59.2, 53.3, 52.9, 29.6, 29.3, 29.0.

Scheme 5



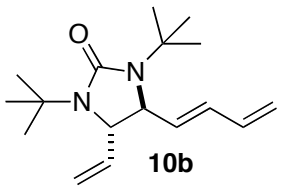
11

White solid; mp. 151-152 °C; IR (film) 1690 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.80 (ddd, *J* = 17.1, 10.2, 9.3 Hz, 2H), 5.26 (d, *J* = 17.1 Hz, 2H), 5.12 (d, *J* = 10.2 Hz, 2H), 4.15 (d, *J* = 9.3 Hz, 2H), 3.33 (s, 2H), 1.42 (s, 18H), 1.37 (s, 18H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.3, 141.7, 116.1, 60.6, 56.7, 54.3, 54.0, 28.9, 28.8; Anal. Calcd for $C_{26}H_{46}N_4O_2$: C, 69.91; H, 10.38; N, 12.54; Found: C, 69.75; H, 10.32; N, 12.37; HRMS Calcd for $C_{26}H_{47}N_4O_2$ (M+1): 447.3699; Found: 447.3712.



10a

Colorless oil; IR (film) 1689 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 5.92 (ddd, *J* = 17.4, 10.2, 8.4 Hz, 1H), 5.9-5.7 (m, 1H), 5.20 (d, *J* = 17.4 Hz, 1H), 5.10 (d, *J* = 10.2, Hz, 1H), 5.08-5.00 (m, 2H), 3.70 (d, *J* = 8.4 Hz, 1H), 3.17 (dd, *J* = 8.4, 2.7 Hz, 1H), 2.24-1.98 (m, 2H), 1.80-1.56 (m, 2H), 1.37 (s, 9H), 1.36 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.2, 140.3, 137.7, 115.5, 115.3, 60.5, 59.0, 53.1, 52.7, 33.3, 29.2, 29.1, 28.9; HRMS Calcd for $C_{17}H_{31}N_2O_1$ (M+1): 279.2436; Found: 279.2438.



10b

Colorless oil; IR (film) 1691 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 6.35 (dt, *J* = 16.8, 10.2 Hz, 1H), 6.18 (dd, *J* = 15.0, 10.2 Hz, 1H), 5.98 (ddd, *J* = 18.0, 10.2, 8.4 Hz, 1H), 5.80 (dd, *J* = 15.0, 8.4 Hz, 1H), 5.28-5.10 (m, 4H), 3.65 (d, *J* = 8.4 Hz, 1H), 3.64 (d, *J* = 8.4 Hz, 1H), 1.35 (s, 9H), 1.34 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.2, 139.4, 136.1, 134.4, 131.8, 118.0, 116.0, 62.9, 62.0, 53.3, 53.1, 28.8, 28.7; HRMS Calcd for $C_{17}H_{29}N_2O_1$ (M+1): 277.2280; Found: 277.2277.

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: Benzene
Ambient temperature
File: dhf2112-1-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592336 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

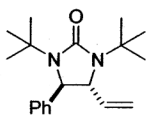


Table 1, Entry 1

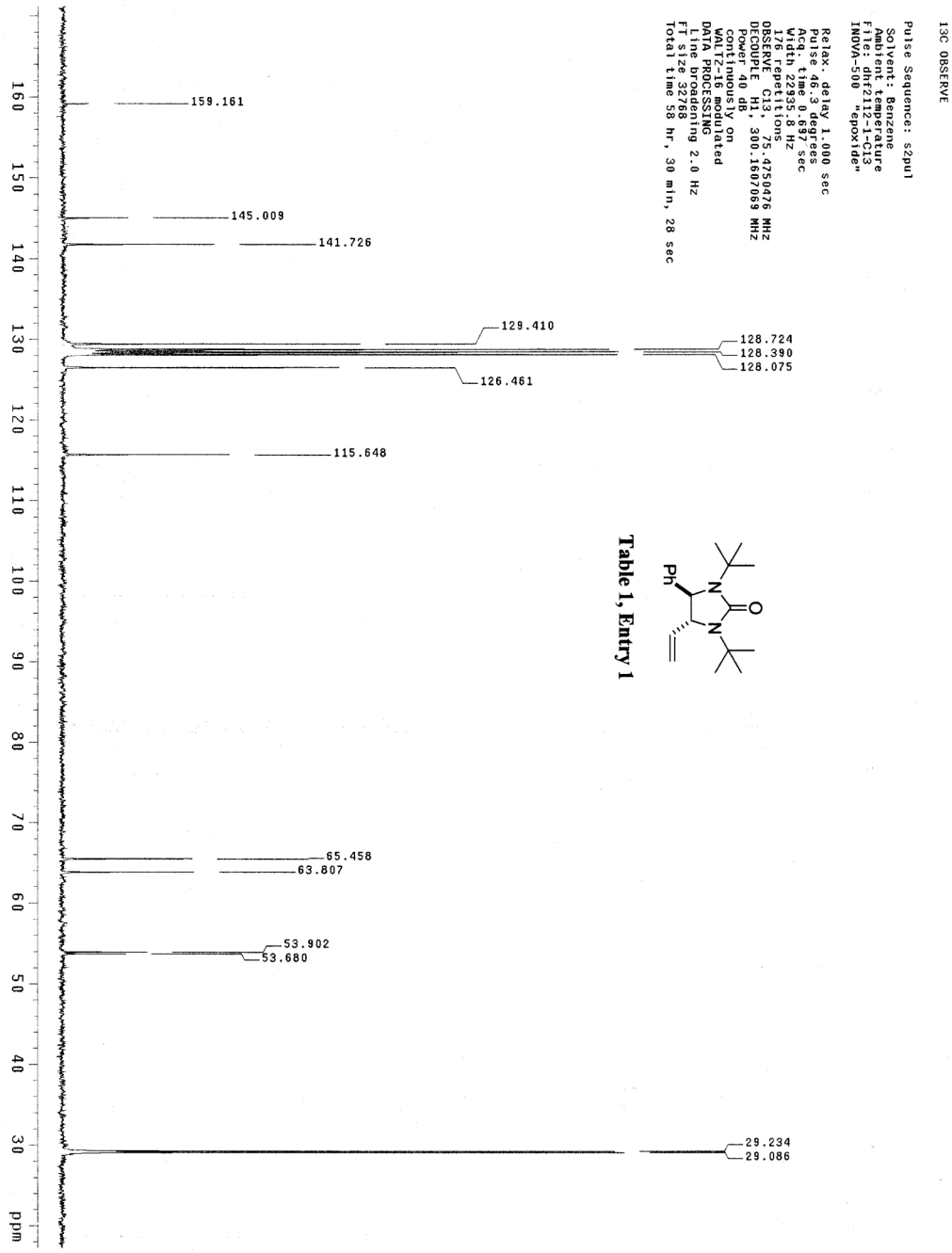


Table 1, Entry 1

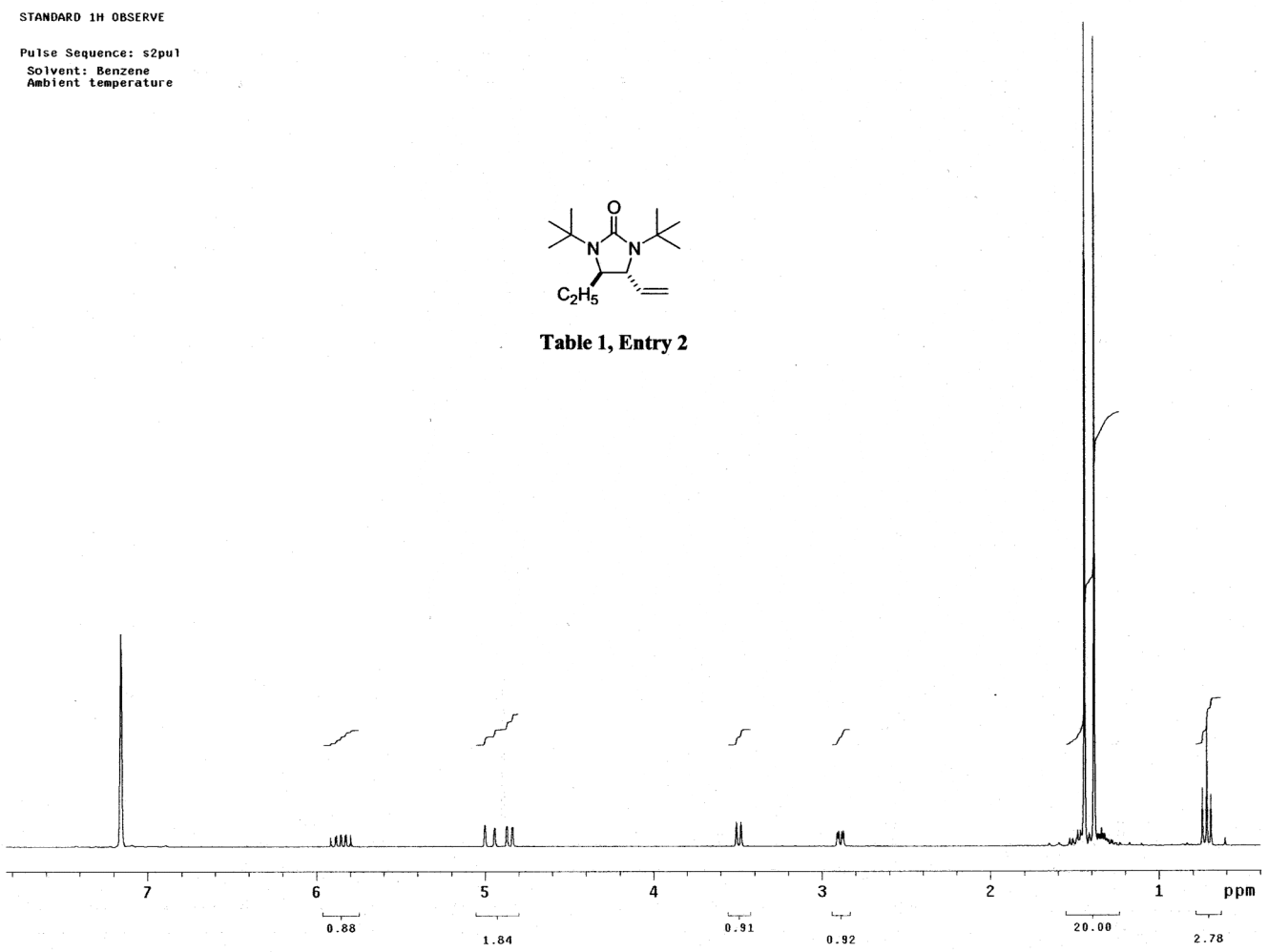


Table 1, Entry 2

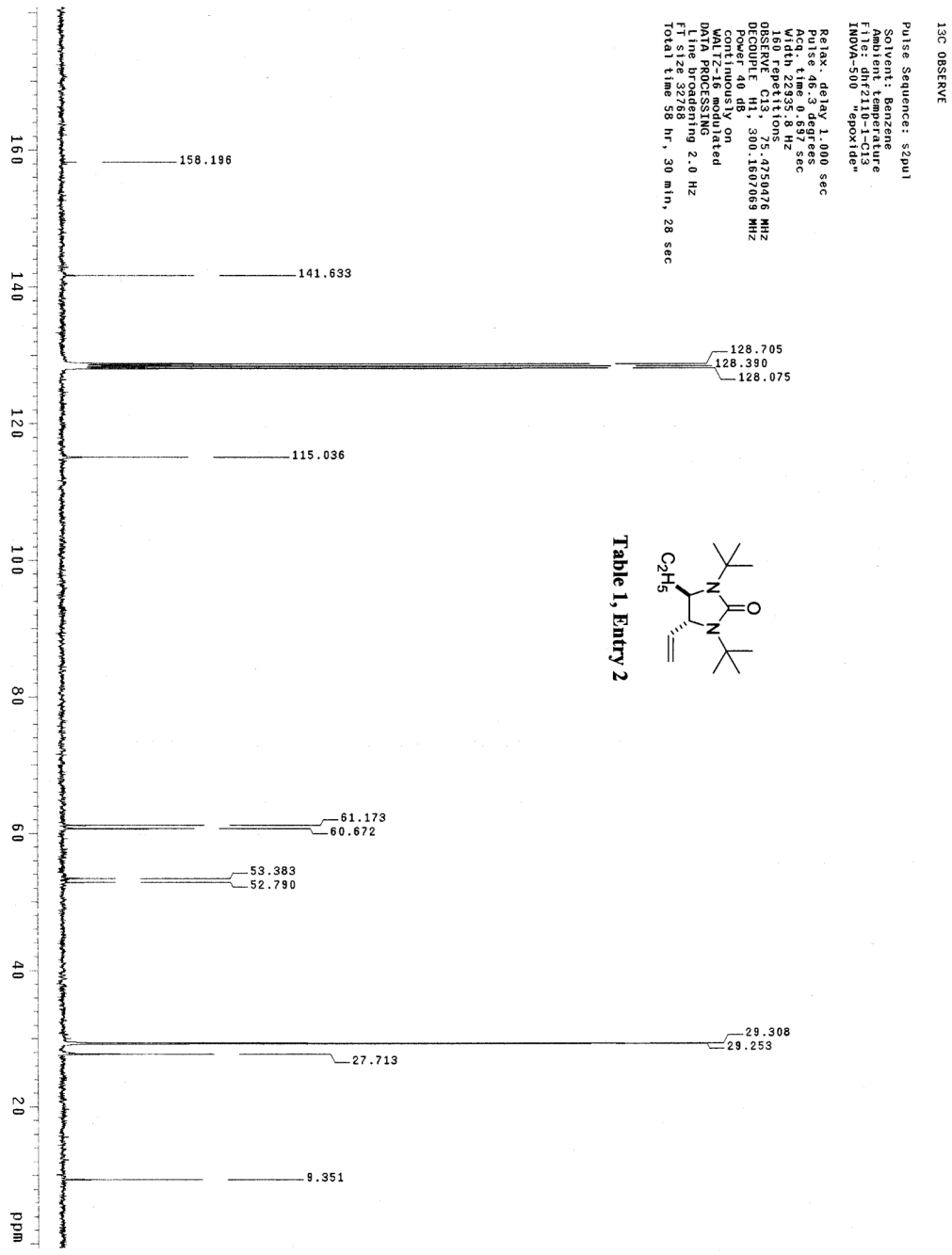


Table 1, Entry 2

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: Benzene
Ambient temperature
File: dhf2110-2-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592332 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

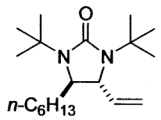


Table 1, Entry 3

13C OBSERVE

Pulse Sequence: s2pul
Solvent: Benzene
Ambient temperature
File: dhf2110-2-C13
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
224 repetitions
OBSERVE C13, 75.4750476 MHz
DECOUPLE H1, 300.1607069 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 45 min, 29 sec

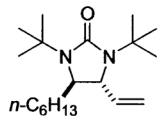
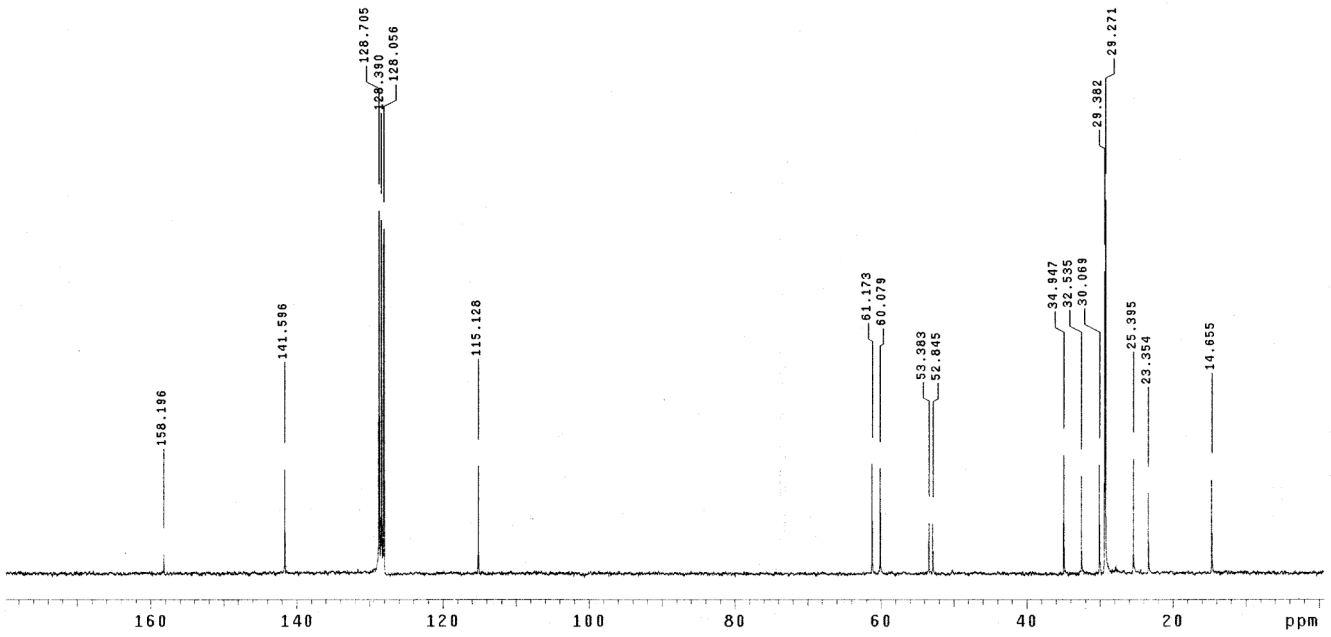


Table 1, Entry 3



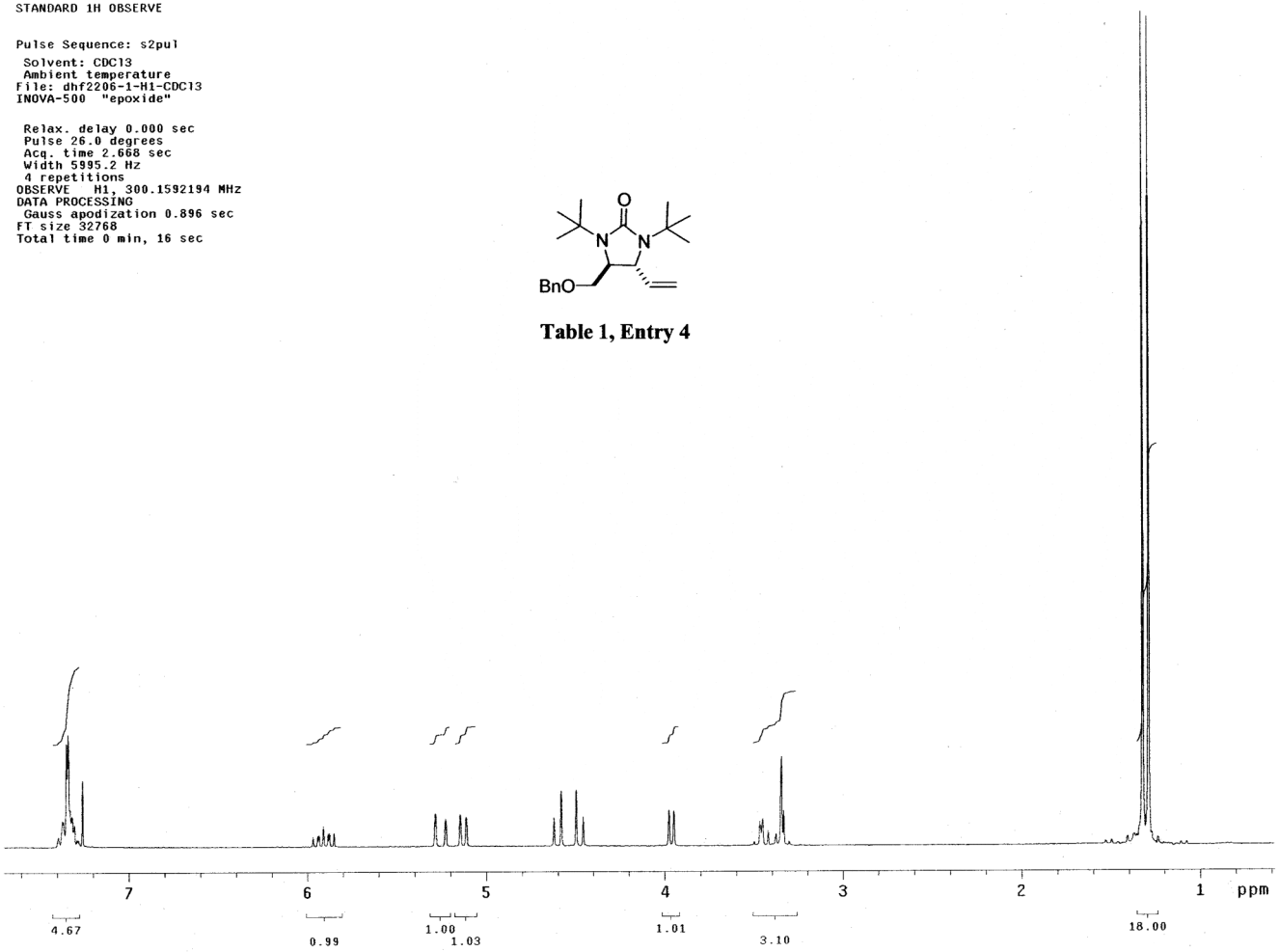


Table 1, Entry 4

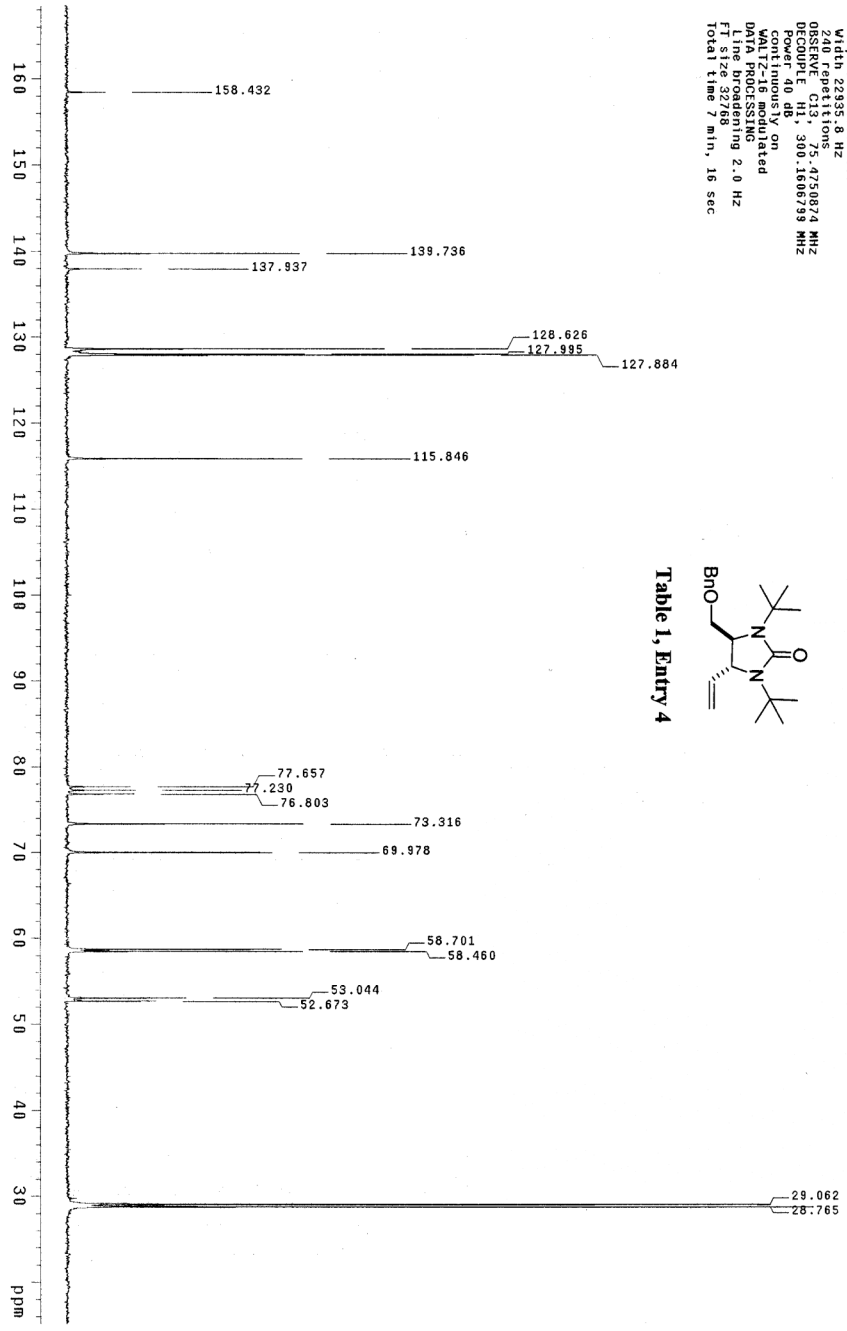


Table 1, Entry 4

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: Benzene
Ambient temperature
File: dhf2206-2-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592336 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

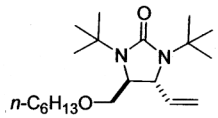


Table 1, Entry 5

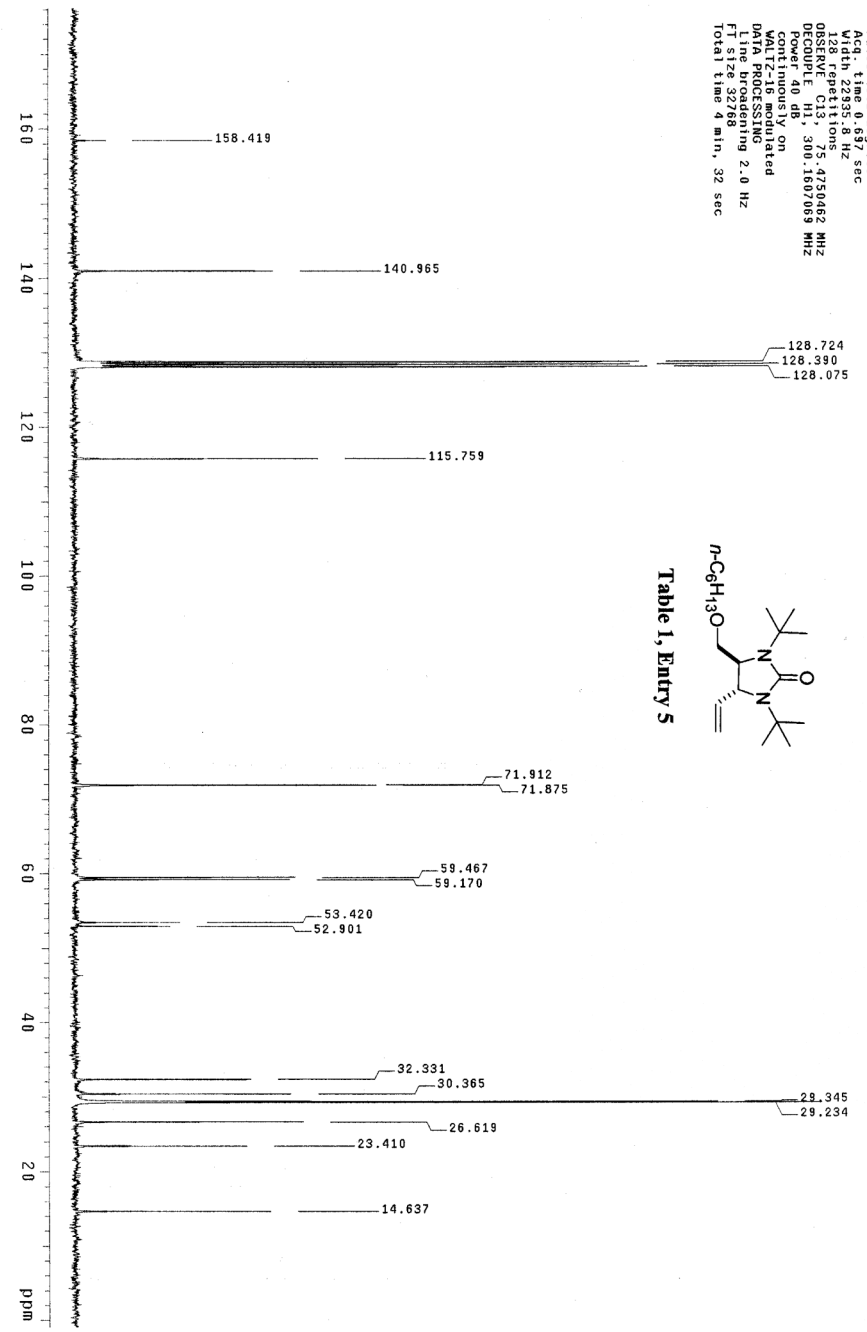


Table 1, Entry 5

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: Benzene
Ambient temperature
File: dhf2109-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592336 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

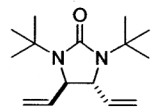


Table 1, Entry 6

13C OBSERVE

Pulse Sequence: s2pul
Solvent: Benzene
Ambient temperature
File: dhf2109-C13
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
96 repetitions
OBSERVE C13, 75.4750476 MHz
DECOUPLE H1, 300.1607069 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 58 hr, 30 min, 28 sec

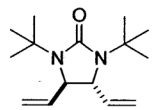
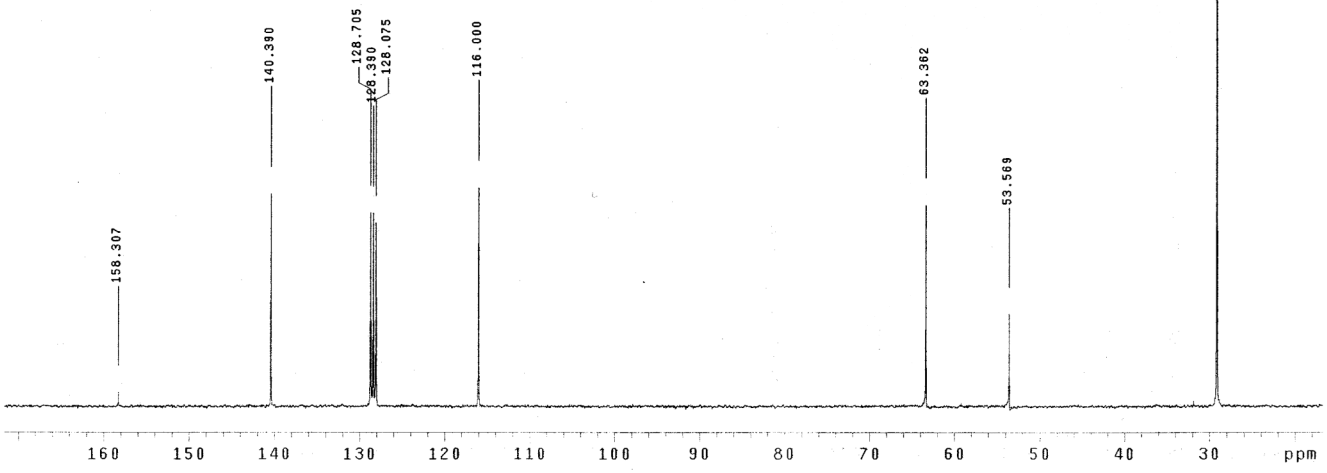


Table 1, Entry 6



STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2213-1-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592197 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

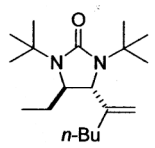
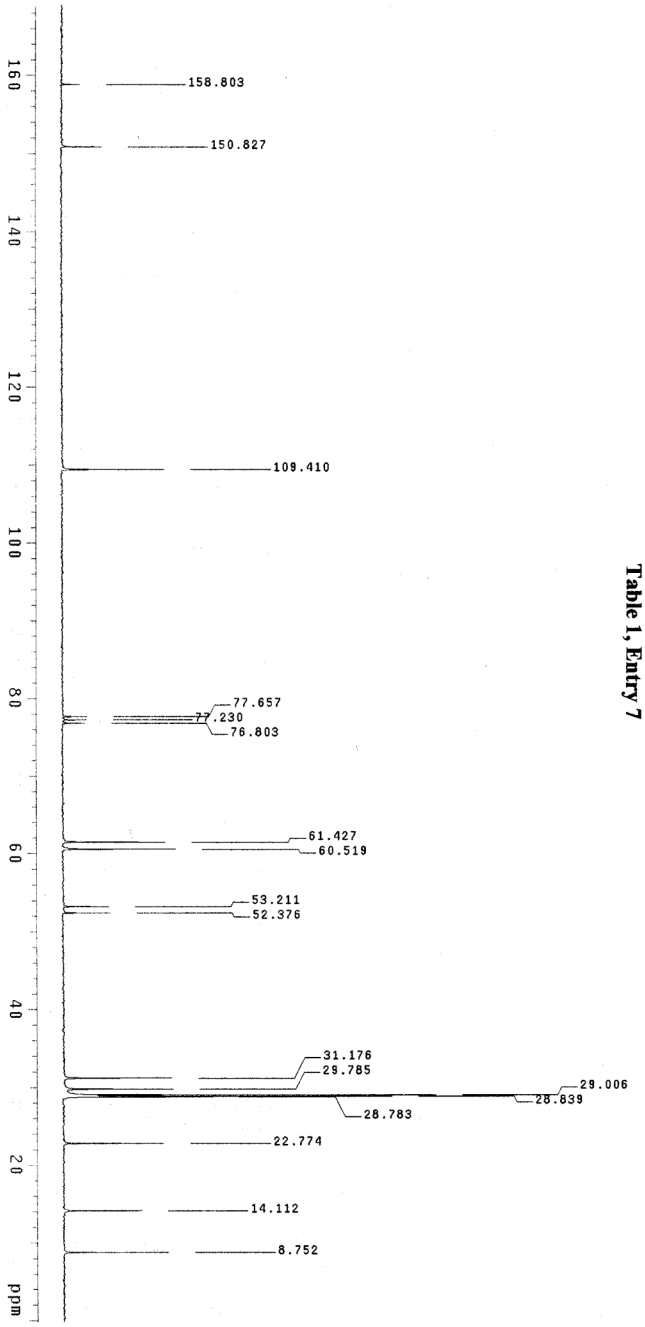


Table 1, Entry 7



13C OBSERVE

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
File: dhf2213-1-C13
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
16000 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 4 min, 32 sec

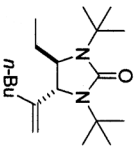


Table 1, Entry 7

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2205-1-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592197 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

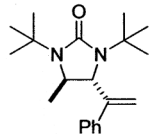


Table 1, Entry 8

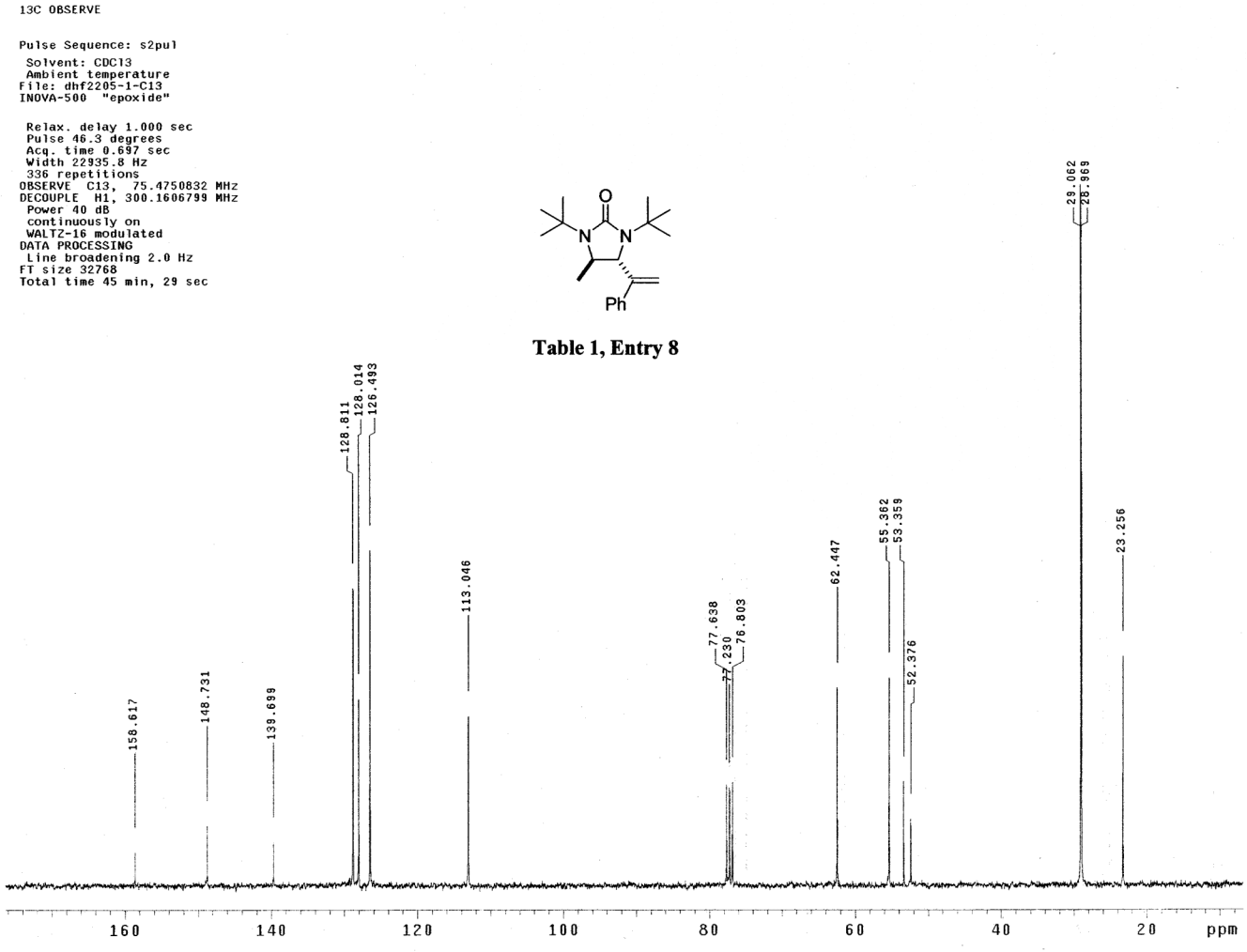


Table 1, Entry 8

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2149-3-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592194 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

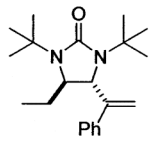


Table 1, Entry 9

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2149-3-C13
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
80 repetitions
OBSERVE C13, 75.4750902 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 4 min, 32 sec

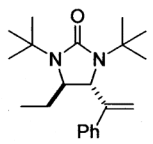


Table 1, Entry 9

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: Benzene
Ambient temperature
File: dhf2220-1-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592336 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

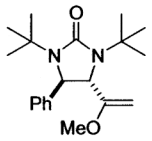
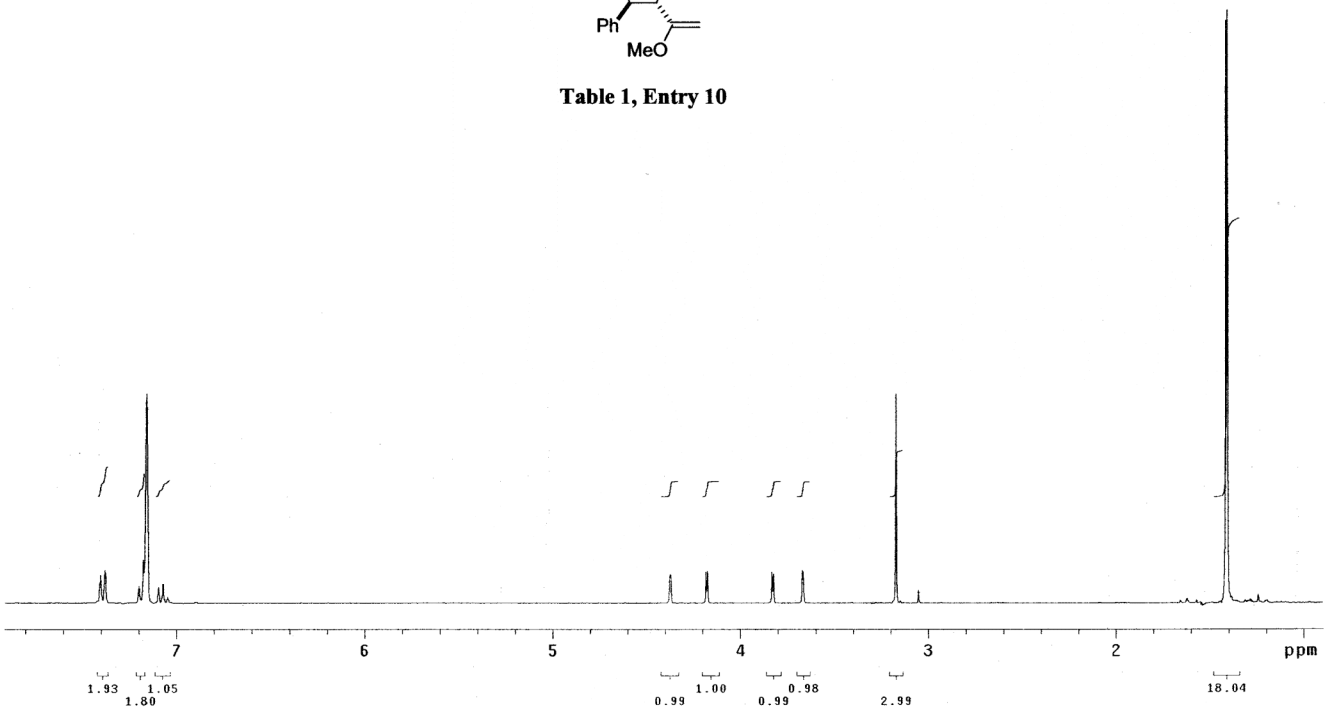


Table 1, Entry 10



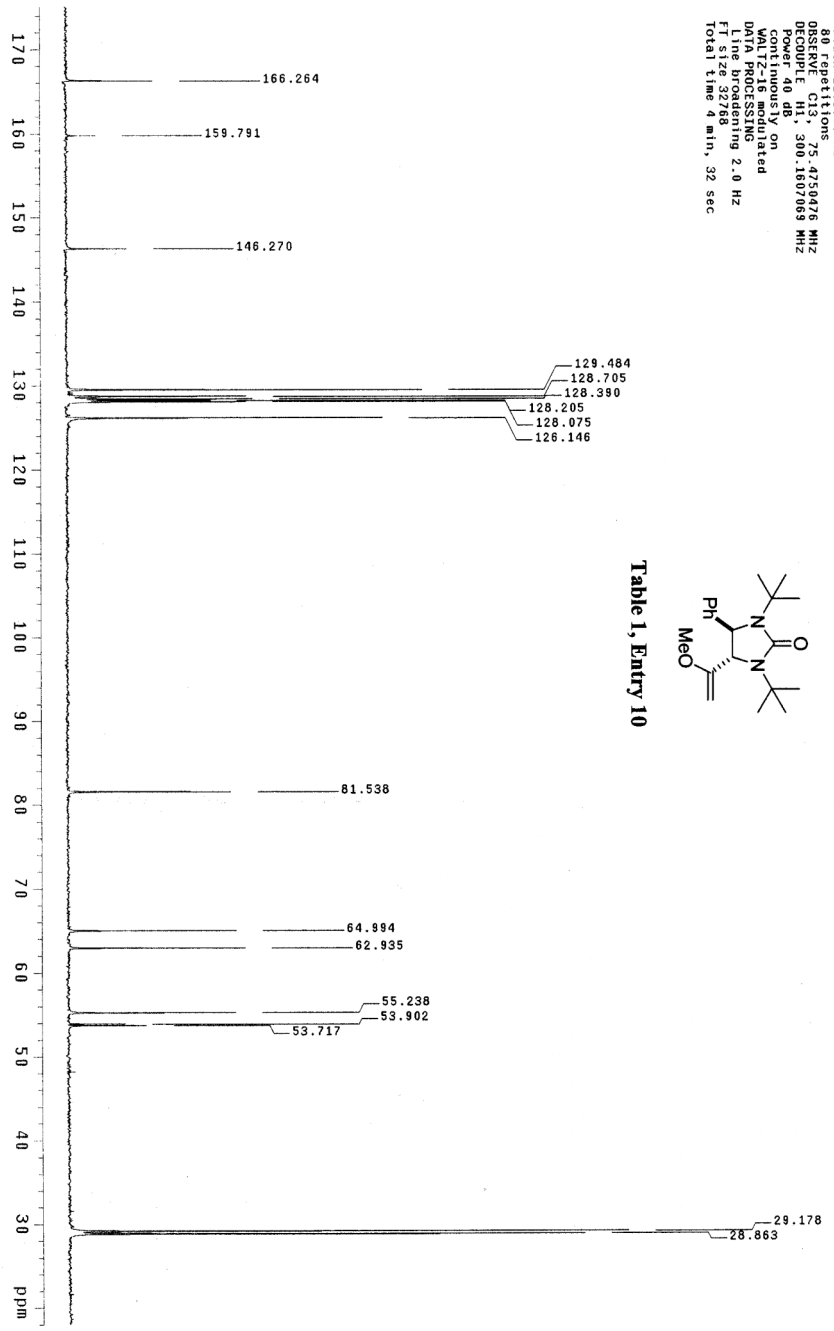


Table 1, Entry 10

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: Benzene
Ambient temperature
File: dhf2220-2-H
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592339 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

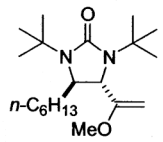


Table 1, Entry 11

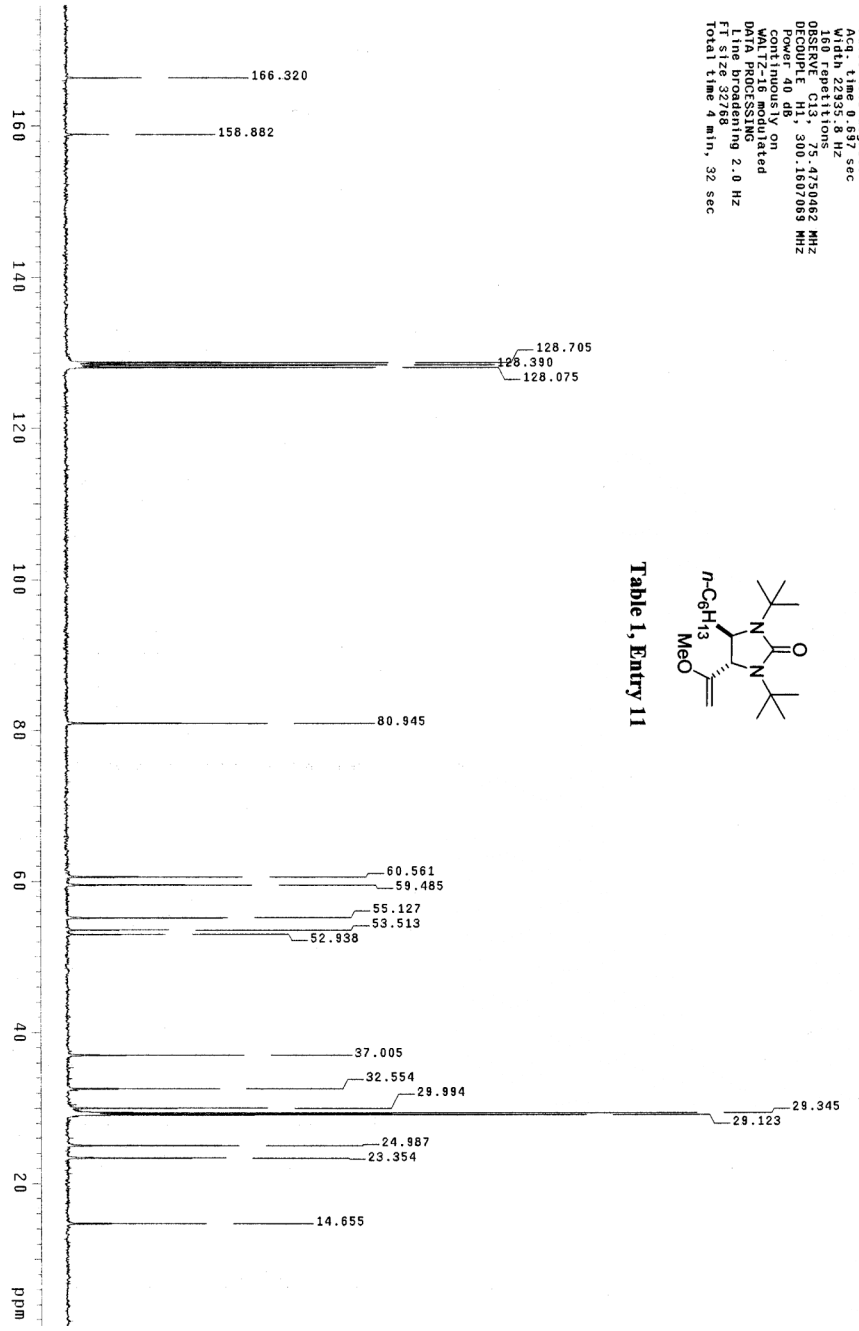
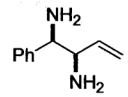


Table 1, Entry 11

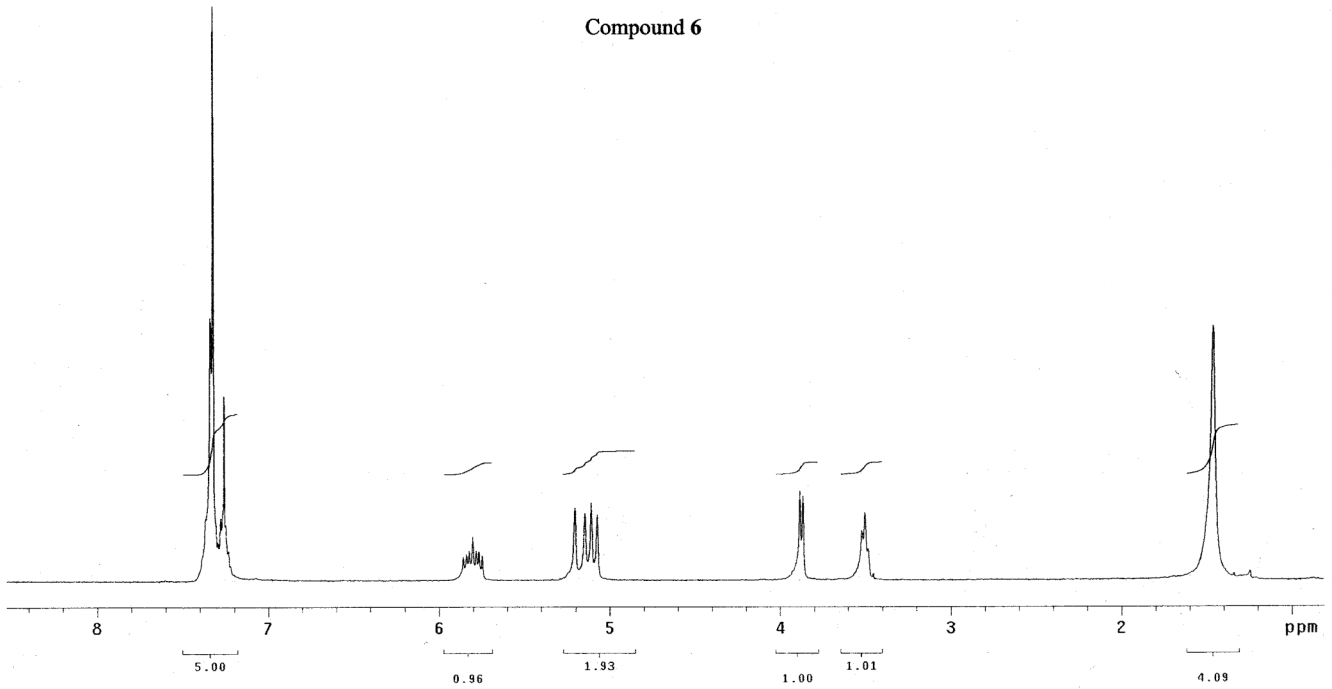
STANDARD 1H OBSERVE

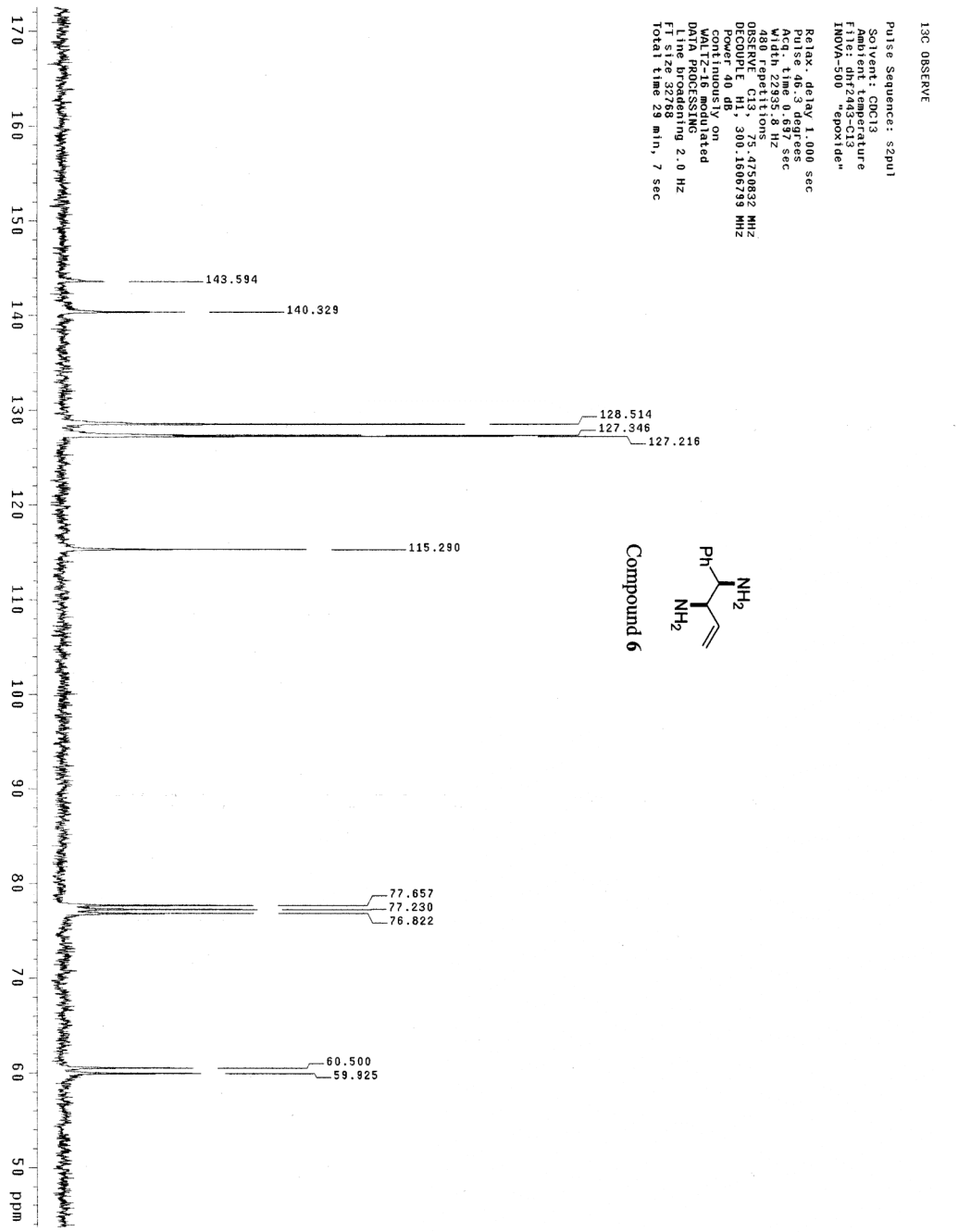
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2443-diamine-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec



Compound **6**



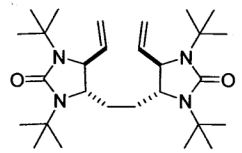

13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dnf243-c13
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 45.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
480 repetitions
OBSERVE C13, 75.4750832 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 29 min, 7 sec
143.594
140.329
128.514
127.346
127.216
115.290
77.657
77.230
76.822
60.500
59.925
170
160
150
140
130
120
110
100
90
80
70
60
50 ppm
Ph
NH2
NH2

Compound **6**

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2212-2-meso-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592201 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

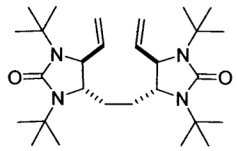


Compound **8a**

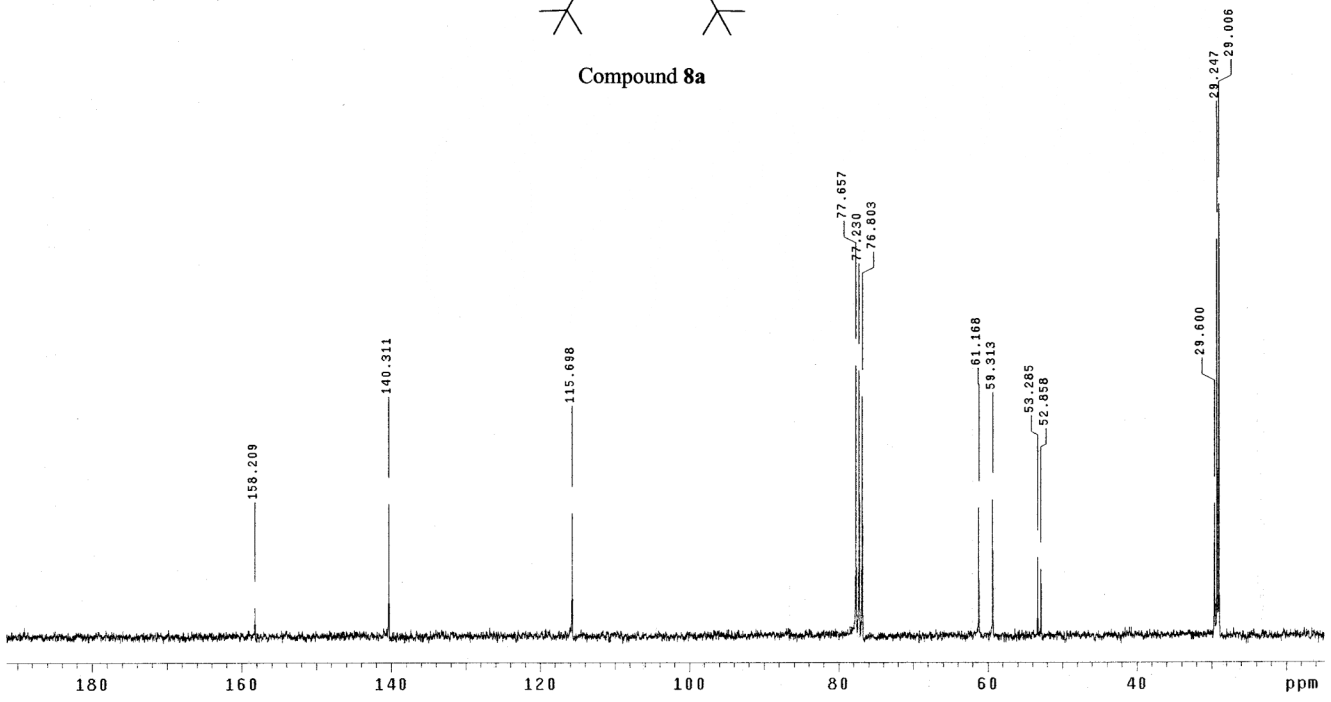
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2212-2-meso-C13
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
1344 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 58 hr, 30 min, 28 sec



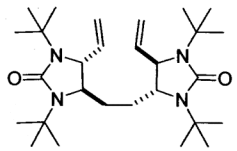
Compound **8a**



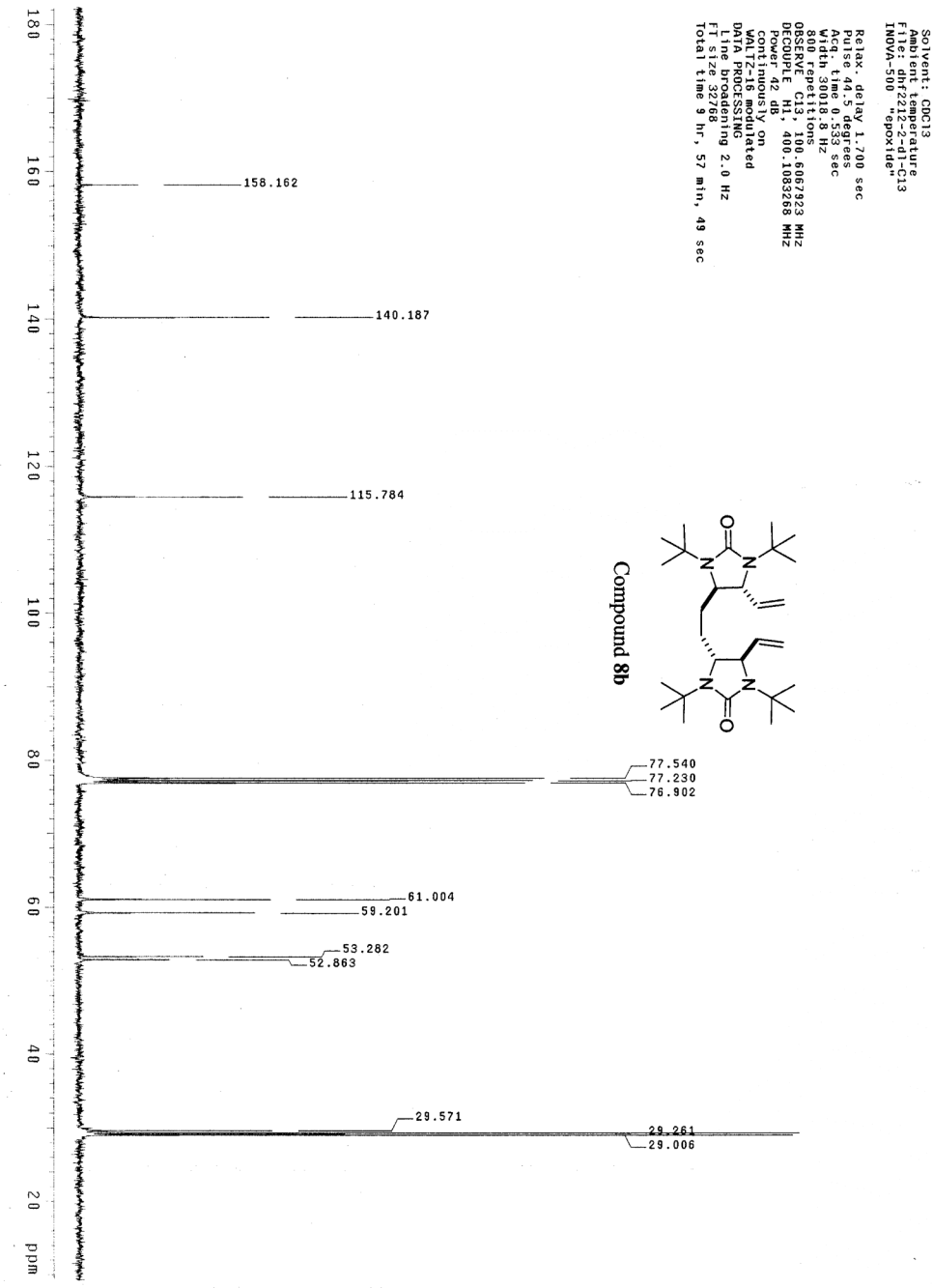
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2212-d1-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
20 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 55 min, 9 sec



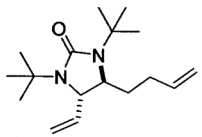
Compound **8b**


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2212-2-d1-C13
INOVA-500 "epoxide"
Relax. delay 1.700 sec
Pulse 44.5 degrees
Acq. time 0.533 sec
Width 30018.8 Hz
800 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 9 hr, 57 min, 49 sec
Compound 8b
158.162
140.187
115.784
77.540
77.230
76.902
61.004
59.201
53.282
52.863
29.571
29.261
29.006
180
160
140
120
100
80
60
40
20
ppm

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2235-2-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592197 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

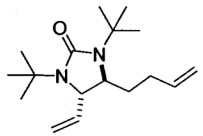


Compound **10a**

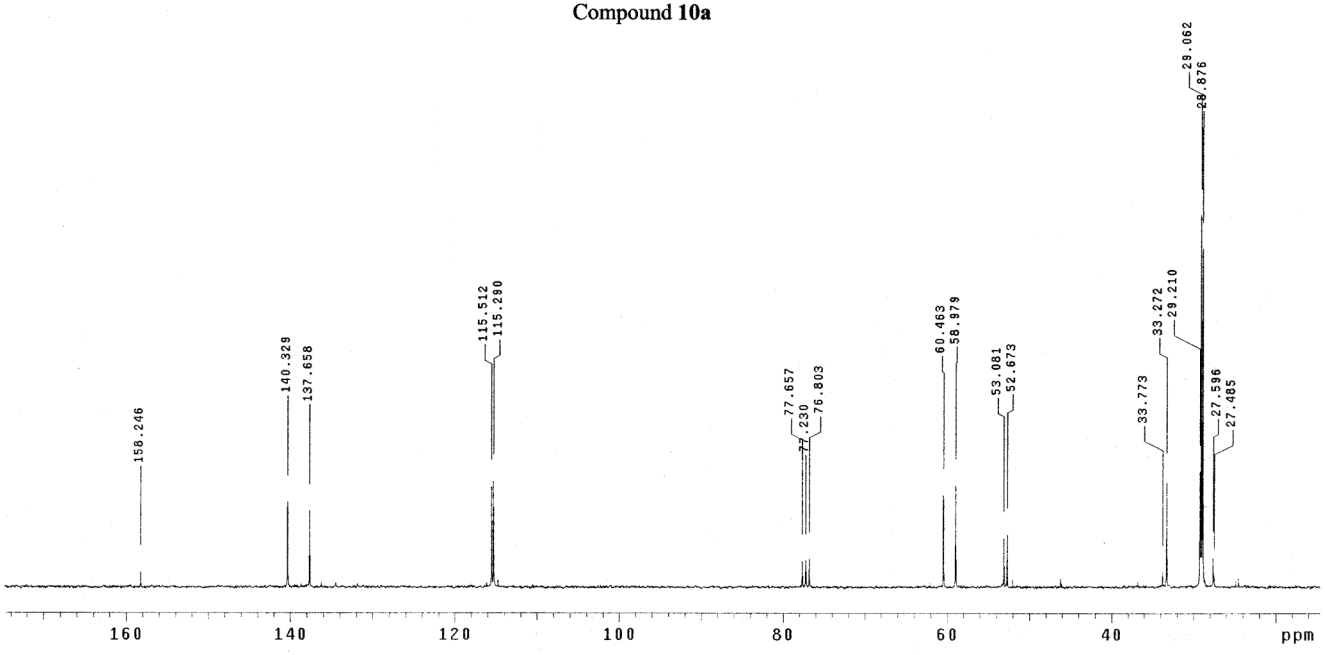
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2235-2-C13
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
128 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 7 min, 16 sec



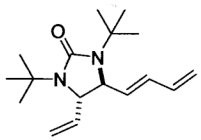
Compound **10a**



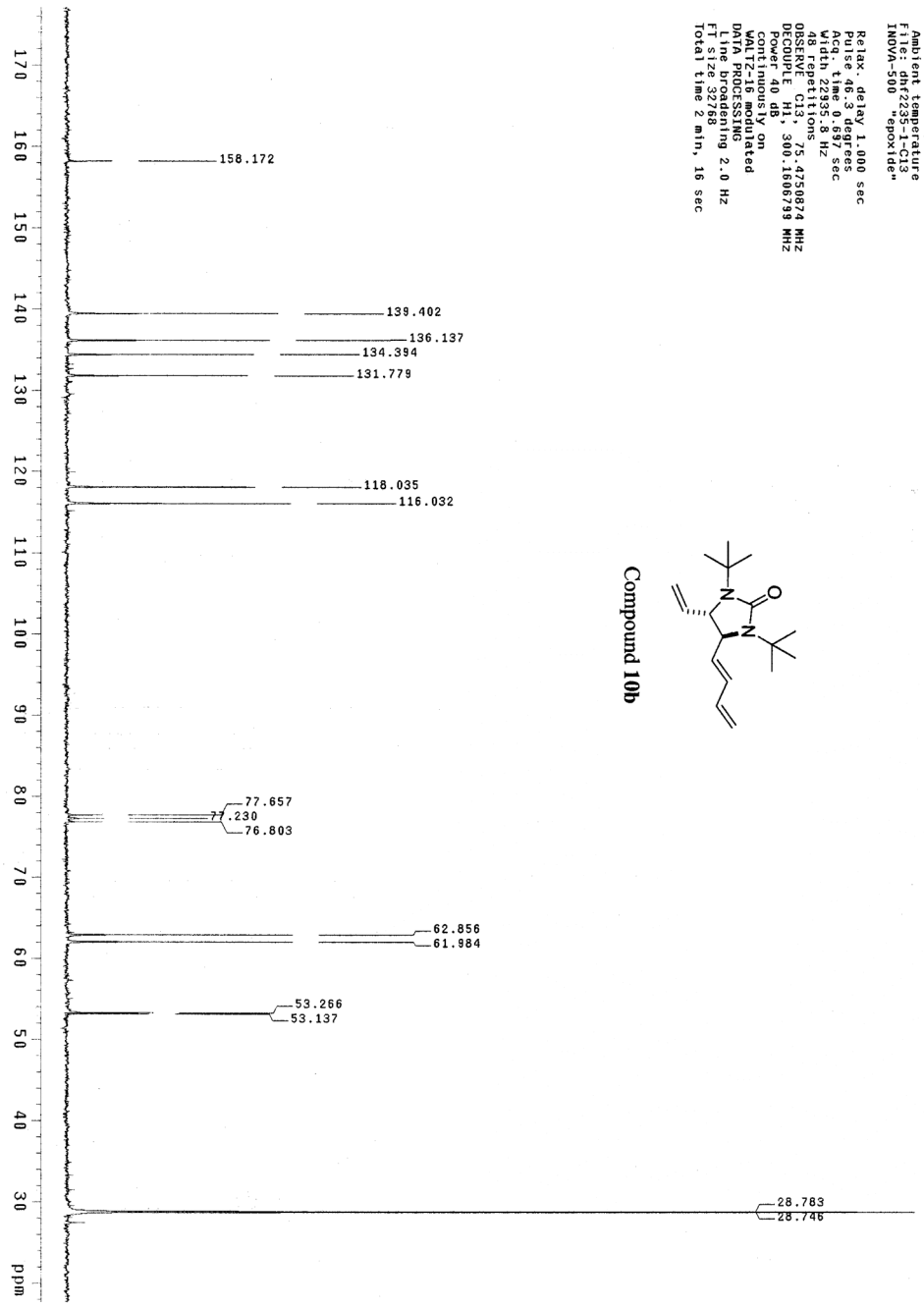
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2235-1-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec



Compound **10b**

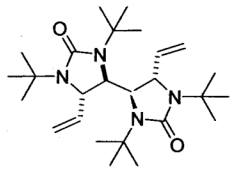


Compound 10b

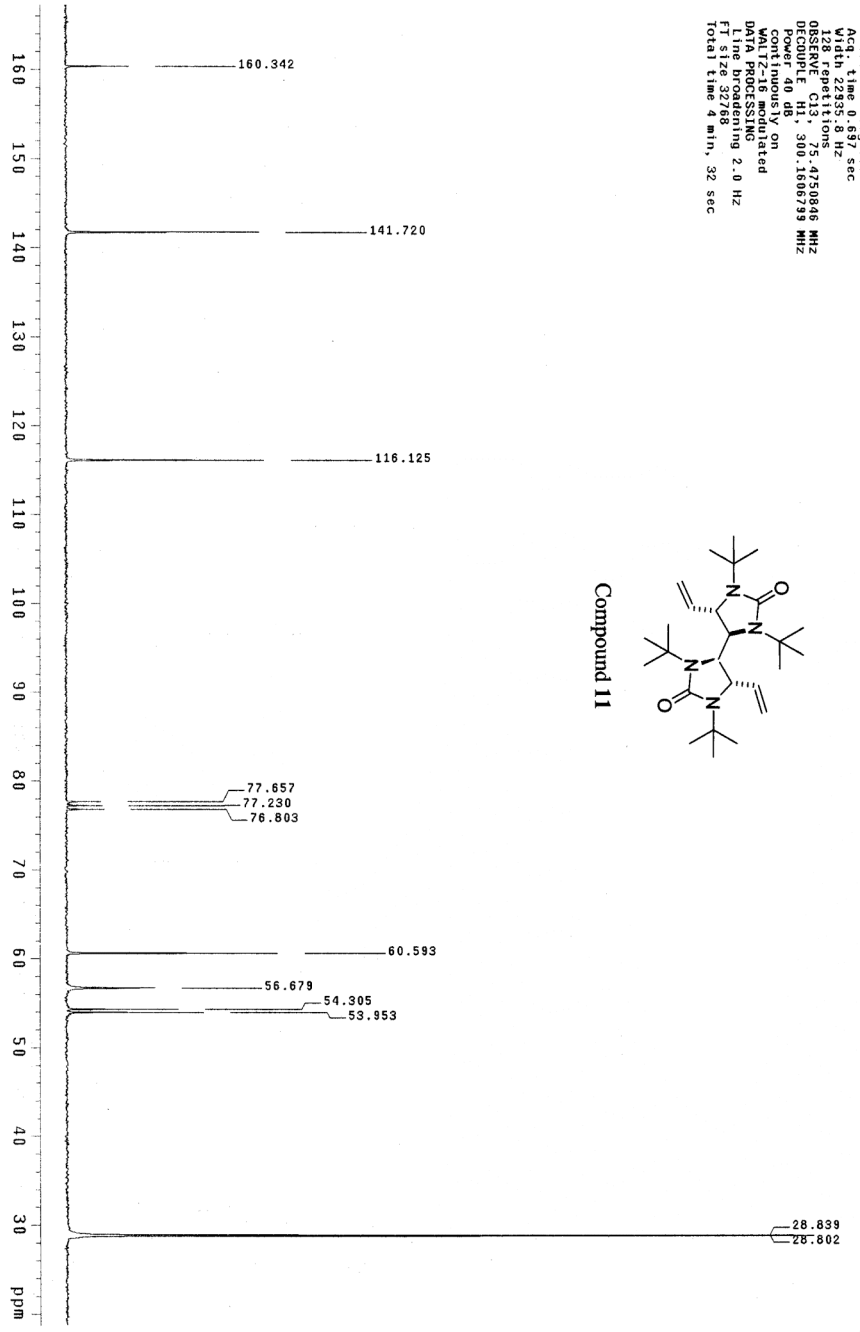
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf2209-2-H1
INOVA-500 "epoxide"

Relax. delay 0.000 sec
PuIse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
4 repetitions
OBSERVE H1, 300.1592197 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 16 sec

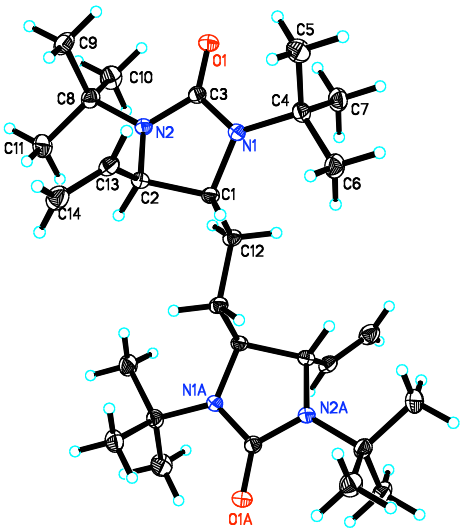


Compound **11**


13C OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: dhf220-2-C13
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
128 repetitions
OBSERVE C13, 75.4750846 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 4 min, 32 sec
160.342
141.720
116.125
77.657
77.230
76.803
60.593
56.679
54.305
53.953
28.839
28.802
ppm

Compound 11

X-ray structure for 8a



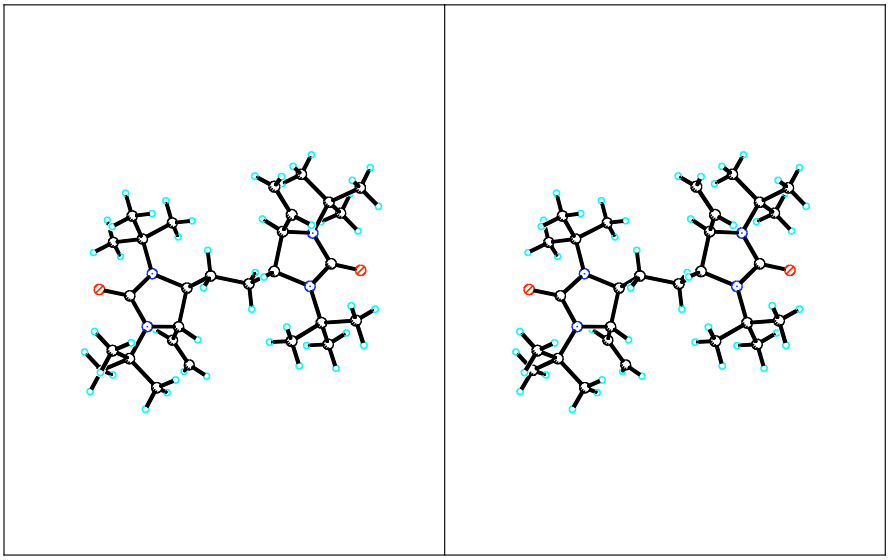


Table 1. Crystal data and structure refinement for **8a**.

Identification code	ys157_0m	
Empirical formula	C28 H50 N4 O2	
Formula weight	474.72	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 10.4248(4) Å	α= 90°.
	b = 12.8075(4) Å	β= 112.968(2)°.
	c = 11.7988(4) Å	γ = 90°.
Volume	1450.44(9) $Å^3$	
Z	2	
Density (calculated)	1.087 Mg/m^3	
Absorption coefficient	0.068 mm^{-1}	
F(000)	524	
Crystal size	0.20 x 0.13 x 0.07 mm^3	
Theta range for data collection	2.12 to 30.37°.	
Index ranges	-14<=h<=14, -17<=k<=18, -10<=l<=16	
Reflections collected	13979	
Independent reflections	4336 [R(int) = 0.0538]	
Completeness to theta = 30.37°	99.0 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9950 and 0.9862	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4336 / 0 / 154	
Goodness-of-fit on F^2	1.006	
Final R indices [I>2sigma(I)]	R1 = 0.0520, wR2 = 0.1199	
R indices (all data)	R1 = 0.0874, wR2 = 0.1377	
Largest diff. peak and hole	0.344 and -0.245 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for ys157_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	7132(1)	1732(1)	3960(1)	20(1)
N(1)	8870(1)	2802(1)	3846(1)	15(1)
N(2)	6810(1)	3508(1)	3534(1)	15(1)
C(1)	9063(1)	3926(1)	3778(1)	14(1)
C(2)	7541(1)	4274(1)	3077(1)	15(1)
C(3)	7557(1)	2589(1)	3795(1)	15(1)
C(4)	9962(1)	1993(1)	4097(1)	18(1)
C(5)	9433(2)	1157(1)	3096(1)	27(1)
C(6)	11272(1)	2487(1)	4035(2)	23(1)
C(7)	10321(2)	1517(1)	5372(1)	24(1)
C(8)	5297(1)	3543(1)	3281(1)	19(1)
C(9)	4460(1)	2910(1)	2127(1)	25(1)
C(10)	5081(2)	3109(1)	4401(2)	26(1)
C(11)	4819(2)	4679(1)	3097(2)	25(1)
C(12)	9739(1)	4451(1)	5039(1)	16(1)
C(13)	7149(1)	4221(1)	1707(1)	18(1)
C(14)	6677(1)	5015(1)	948(1)	25(1)

Table 3. Bond lengths [Å] and angles [°] for ys157_0m.

O(1)-C(3)	1.2269(15)
N(1)-C(3)	1.3745(16)
N(1)-C(1)	1.4600(16)
N(1)-C(4)	1.4809(17)
N(2)-C(3)	1.3788(16)
N(2)-C(2)	1.4685(16)
N(2)-C(8)	1.4870(16)
C(1)-C(12)	1.5307(18)
C(1)-C(2)	1.5416(17)
C(2)-C(13)	1.5068(19)
C(4)-C(7)	1.529(2)
C(4)-C(5)	1.5295(19)
C(4)-C(6)	1.5312(19)
C(8)-C(11)	1.5253(19)
C(8)-C(10)	1.530(2)
C(8)-C(9)	1.532(2)
C(12)-C(12)#1	1.525(2)
C(13)-C(14)	1.3163(19)
C(3)-N(1)-C(1)	110.48(10)
C(3)-N(1)-C(4)	122.99(10)
C(1)-N(1)-C(4)	126.08(10)
C(3)-N(2)-C(2)	108.90(10)
C(3)-N(2)-C(8)	122.25(10)
C(2)-N(2)-C(8)	124.61(10)
N(1)-C(1)-C(12)	113.62(11)
N(1)-C(1)-C(2)	100.35(9)
C(12)-C(1)-C(2)	112.43(10)
N(2)-C(2)-C(13)	113.64(10)
N(2)-C(2)-C(1)	100.54(10)
C(13)-C(2)-C(1)	110.76(11)
O(1)-C(3)-N(1)	125.90(12)
O(1)-C(3)-N(2)	126.27(12)
N(1)-C(3)-N(2)	107.83(10)

N(1)-C(4)-C(7)	110.48(11)
N(1)-C(4)-C(5)	108.53(11)
C(7)-C(4)-C(5)	110.86(12)
N(1)-C(4)-C(6)	109.37(11)
C(7)-C(4)-C(6)	109.34(11)
C(5)-C(4)-C(6)	108.21(12)
N(2)-C(8)-C(11)	108.63(11)
N(2)-C(8)-C(10)	108.52(11)
C(11)-C(8)-C(10)	108.64(12)
N(2)-C(8)-C(9)	110.73(11)
C(11)-C(8)-C(9)	109.72(12)
C(10)-C(8)-C(9)	110.54(12)
C(12)#1-C(12)-C(1)	112.67(13)
C(14)-C(13)-C(2)	124.61(13)

Symmetry transformations used to generate equivalent atoms:
#1 -x+2,-y+1,-z+1

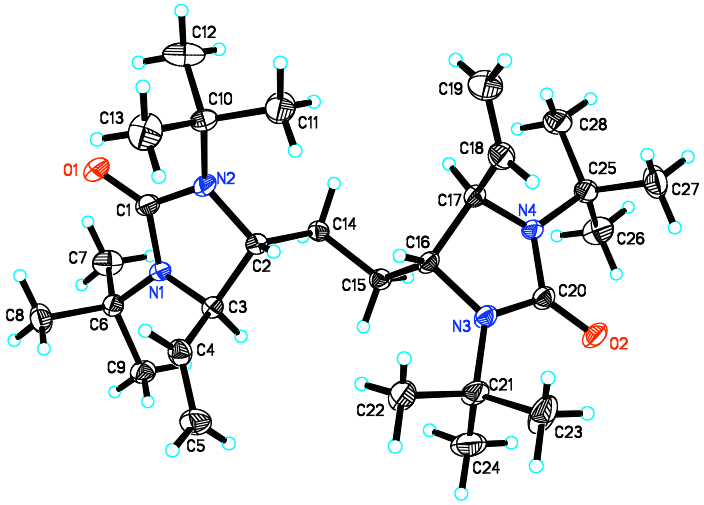
Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for ys157_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	19(1)	14(1)	25(1)	1(1)	8(1)	-3(1)
N(1)	14(1)	13(1)	19(1)	0(1)	6(1)	0(1)
N(2)	14(1)	13(1)	18(1)	1(1)	6(1)	-1(1)
C(1)	15(1)	13(1)	14(1)	1(1)	5(1)	-2(1)
C(2)	16(1)	12(1)	16(1)	0(1)	5(1)	-1(1)
C(3)	15(1)	15(1)	14(1)	-1(1)	5(1)	-1(1)
C(4)	17(1)	16(1)	19(1)	0(1)	7(1)	2(1)
C(5)	27(1)	23(1)	29(1)	-7(1)	10(1)	3(1)
C(6)	18(1)	25(1)	29(1)	2(1)	11(1)	3(1)
C(7)	21(1)	25(1)	25(1)	7(1)	6(1)	4(1)
C(8)	14(1)	21(1)	23(1)	-1(1)	8(1)	0(1)
C(9)	15(1)	30(1)	25(1)	-4(1)	3(1)	-2(1)
C(10)	23(1)	30(1)	30(1)	2(1)	15(1)	1(1)
C(11)	19(1)	24(1)	33(1)	1(1)	12(1)	6(1)
C(12)	16(1)	16(1)	12(1)	1(1)	3(1)	-4(1)
C(13)	15(1)	21(1)	16(1)	-1(1)	4(1)	-4(1)
C(14)	21(1)	30(1)	22(1)	6(1)	5(1)	-4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for ys157_0m.

	x	y	z	U(eq)
H(1A)	9622	4073	3273	17
H(2A)	7398	4996	3328	18
H(5A)	9204	1479	2288	40
H(5B)	8598	827	3125	40
H(5C)	10158	627	3235	40
H(6A)	11046	2787	3216	35
H(6B)	11992	1951	4194	35
H(6C)	11614	3038	4657	35
H(7A)	9484	1203	5415	37
H(7B)	10672	2064	5999	37
H(7C)	11037	979	5521	37
H(9A)	4607	3200	1418	38
H(9B)	3467	2945	1975	38
H(9C)	4769	2181	2248	38
H(10A)	5621	3523	5130	39
H(10B)	5392	2381	4534	39
H(10C)	4090	3144	4257	39
H(11A)	4946	4966	2378	37
H(11B)	5372	5085	3830	37
H(11C)	3832	4716	2966	37
H(12A)	9051	4485	5426	19
H(12B)	10531	4018	5574	19
H(13A)	7249	3569	1366	21
H(14A)	6564	5679	1256	30
H(14B)	6450	4924	92	30

X-ray structure for 8b



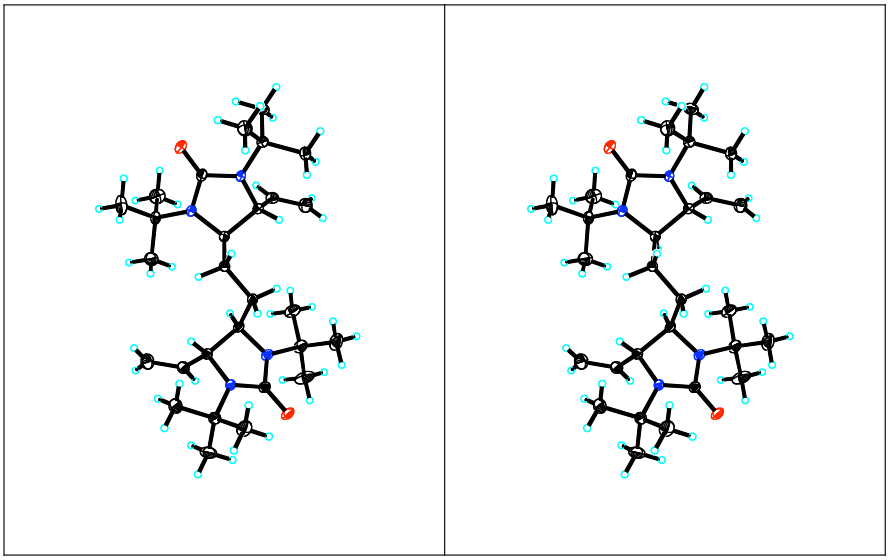


Table 1. Crystal data and structure refinement for **8b**.

Identification code	ys161r_0m	
Empirical formula	C28 H50 N4 O2	
Formula weight	474.72	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 15.5328(6) Å	α= 90°.
	b = 15.6066(6) Å	β= 90°.
	c = 24.5182(8) Å	γ = 90°.
Volume	5943.6(4) $Å^3$	
Z	8	
Density (calculated)	1.061 Mg/m^3	
Absorption coefficient	0.067 mm^{-1}	
F(000)	2096	
Crystal size	0.52 x 0.34 x 0.29 mm^3	
Theta range for data collection	1.85 to 31.51°.	
Index ranges	-22<=h<=22, -21<=k<=22, -36<=l<=33	
Reflections collected	121000	
Independent reflections	19737 [R(int) = 0.0556]	
Completeness to theta = 31.51°	99.8 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9807 and 0.9663	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	19737 / 0 / 613	
Goodness-of-fit on F^2	1.026	
Final R indices [I>2sigma(I)]	R1 = 0.0542, wR2 = 0.1176	
R indices (all data)	R1 = 0.0966, wR2 = 0.1358	
Largest diff. peak and hole	0.346 and -0.211 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for ys161r_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	-5321(1)	1416(1)	3639(1)	28(1)
O(2)	23(1)	854(1)	1369(1)	28(1)
N(1)	-4919(1)	1603(1)	2734(1)	20(1)
N(2)	-4052(1)	857(1)	3284(1)	25(1)
N(3)	-1236(1)	331(1)	1759(1)	22(1)
N(4)	-340(1)	1093(1)	2278(1)	19(1)
C(1)	-4818(1)	1298(1)	3259(1)	21(1)
C(2)	-3635(1)	839(1)	2744(1)	19(1)
C(3)	-4373(1)	1112(1)	2357(1)	19(1)
C(4)	-4832(1)	349(1)	2116(1)	24(1)
C(5)	-4861(1)	181(1)	1588(1)	32(1)
C(6)	-5666(1)	2136(1)	2566(1)	21(1)
C(7)	-5682(1)	2949(1)	2913(1)	32(1)
C(8)	-6514(1)	1639(1)	2625(1)	31(1)
C(9)	-5554(1)	2386(1)	1968(1)	27(1)
C(10)	-3824(1)	267(1)	3740(1)	25(1)
C(11)	-2916(1)	-81(2)	3663(1)	47(1)
C(12)	-3862(2)	753(1)	4283(1)	41(1)
C(13)	-4460(1)	-476(1)	3740(1)	38(1)
C(14)	-2872(1)	1462(1)	2711(1)	19(1)
C(15)	-2297(1)	1306(1)	2215(1)	19(1)
C(16)	-1709(1)	519(1)	2265(1)	18(1)
C(17)	-948(1)	683(1)	2656(1)	19(1)
C(18)	-605(1)	-144(1)	2893(1)	26(1)
C(19)	-588(1)	-327(1)	3418(1)	35(1)
C(20)	-465(1)	766(1)	1758(1)	21(1)
C(21)	-1621(1)	-53(1)	1260(1)	29(1)
C(22)	-2452(1)	-523(1)	1417(1)	35(1)
C(23)	-989(1)	-717(2)	1033(1)	44(1)
C(24)	-1818(2)	643(2)	834(1)	43(1)

C(25)	481(1)	1505(1)	2449(1)	24(1)
C(26)	614(1)	2304(1)	2096(1)	35(1)
C(27)	1231(1)	877(1)	2391(1)	37(1)
C(28)	403(1)	1792(1)	3042(1)	34(1)
O(1A)	6542(1)	7903(1)	-1162(1)	27(1)
O(2A)	6262(1)	2586(1)	1123(1)	28(1)
N(1A)	6935(1)	6565(1)	-827(1)	26(1)
N(2A)	6286(1)	7485(1)	-263(1)	20(1)
N(3A)	6971(1)	3715(1)	692(1)	23(1)
N(4A)	6041(1)	2927(1)	209(1)	21(1)
C(1A)	6586(1)	7375(1)	-791(1)	21(1)
C(2A)	6863(1)	6116(1)	-299(1)	19(1)
C(3A)	6700(1)	6865(1)	100(1)	20(1)
C(4A)	7532(1)	7189(1)	346(1)	25(1)
C(5A)	7699(1)	7166(1)	873(1)	35(1)
C(6A)	5887(1)	8298(1)	-74(1)	25(1)
C(7A)	5090(1)	8485(2)	-421(1)	39(1)
C(8A)	6538(1)	9037(1)	-110(1)	33(1)
C(9A)	5600(1)	8188(1)	520(1)	30(1)
C(10A)	7458(1)	6267(1)	-1297(1)	26(1)
C(11A)	6939(1)	6369(1)	-1827(1)	37(1)
C(12A)	7694(2)	5325(1)	-1231(1)	42(1)
C(13A)	8287(1)	6797(1)	-1320(1)	33(1)
C(14A)	6125(1)	5470(1)	-288(1)	21(1)
C(15A)	6161(1)	4888(1)	217(1)	22(1)
C(16A)	6837(1)	4173(1)	174(1)	21(1)
C(17A)	6525(1)	3433(1)	-193(1)	21(1)
C(18A)	7269(1)	2952(1)	-437(1)	27(1)
C(19A)	7433(1)	2906(1)	-967(1)	37(1)
C(20A)	6410(1)	3029(1)	721(1)	21(1)
C(21A)	7471(1)	4042(1)	1168(1)	25(1)
C(22A)	8099(1)	4729(1)	968(1)	34(1)
C(23A)	7996(1)	3302(1)	1408(1)	33(1)
C(24A)	6871(1)	4406(1)	1605(1)	35(1)
C(25A)	5478(1)	2191(1)	67(1)	22(1)
C(26A)	4680(1)	2213(1)	434(1)	32(1)

C(27A)	5957(1)	1340(1)	135(1)	35(1)
C(28A)	5186(1)	2282(1)	-527(1)	29(1)

Table 3. Bond lengths [Å] and angles [°] for ys161r_0m.

O(1)-C(1)	1.230(2)
O(2)-C(20)	1.2274(19)
N(1)-C(1)	1.381(2)
N(1)-C(3)	1.469(2)
N(1)-C(6)	1.486(2)
N(2)-C(1)	1.377(2)
N(2)-C(2)	1.475(2)
N(2)-C(10)	1.490(2)
N(3)-C(20)	1.376(2)
N(3)-C(16)	1.471(2)
N(3)-C(21)	1.489(2)
N(4)-C(20)	1.387(2)
N(4)-C(17)	1.469(2)
N(4)-C(25)	1.488(2)
C(2)-C(14)	1.535(2)
C(2)-C(3)	1.547(2)
C(3)-C(4)	1.509(2)
C(4)-C(5)	1.321(3)
C(6)-C(7)	1.528(2)
C(6)-C(9)	1.529(2)
C(6)-C(8)	1.536(2)
C(10)-C(13)	1.523(3)
C(10)-C(11)	1.524(3)
C(10)-C(12)	1.533(3)
C(14)-C(15)	1.530(2)
C(15)-C(16)	1.535(2)
C(16)-C(17)	1.543(2)
C(17)-C(18)	1.513(2)
C(18)-C(19)	1.318(3)
C(21)-C(23)	1.533(3)
C(21)-C(22)	1.535(2)
C(21)-C(24)	1.538(3)
C(25)-C(28)	1.525(2)
C(25)-C(27)	1.530(3)

C(25)-C(26)	1.533(3)
O(1A)-C(1A)	1.228(2)
O(2A)-C(20A)	1.227(2)
N(1A)-C(1A)	1.379(2)
N(1A)-C(2A)	1.477(2)
N(1A)-C(10A)	1.484(2)
N(2A)-C(1A)	1.387(2)
N(2A)-C(3A)	1.464(2)
N(2A)-C(6A)	1.485(2)
N(3A)-C(20A)	1.382(2)
N(3A)-C(16A)	1.472(2)
N(3A)-C(21A)	1.493(2)
N(4A)-C(20A)	1.388(2)
N(4A)-C(17A)	1.470(2)
N(4A)-C(25A)	1.484(2)
C(2A)-C(14A)	1.528(2)
C(2A)-C(3A)	1.544(2)
C(3A)-C(4A)	1.514(2)
C(4A)-C(5A)	1.318(3)
C(6A)-C(7A)	1.531(3)
C(6A)-C(9A)	1.532(2)
C(6A)-C(8A)	1.537(3)
C(10A)-C(12A)	1.525(3)
C(10A)-C(13A)	1.532(3)
C(10A)-C(11A)	1.538(3)
C(14A)-C(15A)	1.536(2)
C(15A)-C(16A)	1.535(2)
C(16A)-C(17A)	1.542(2)
C(17A)-C(18A)	1.503(2)
C(18A)-C(19A)	1.326(3)
C(21A)-C(24A)	1.528(3)
C(21A)-C(22A)	1.531(3)
C(21A)-C(23A)	1.532(2)
C(25A)-C(26A)	1.531(2)
C(25A)-C(27A)	1.532(2)
C(25A)-C(28A)	1.533(2)

C(1)-N(1)-C(3)	109.95(13)
C(1)-N(1)-C(6)	122.59(13)
C(3)-N(1)-C(6)	124.67(13)
C(1)-N(2)-C(2)	110.43(13)
C(1)-N(2)-C(10)	123.24(14)
C(2)-N(2)-C(10)	123.88(14)
C(20)-N(3)-C(16)	109.75(13)
C(20)-N(3)-C(21)	123.20(14)
C(16)-N(3)-C(21)	124.91(13)
C(20)-N(4)-C(17)	109.12(13)
C(20)-N(4)-C(25)	122.60(13)
C(17)-N(4)-C(25)	124.19(13)
O(1)-C(1)-N(2)	126.10(16)
O(1)-C(1)-N(1)	125.73(15)
N(2)-C(1)-N(1)	108.16(14)
N(2)-C(2)-C(14)	111.99(13)
N(2)-C(2)-C(3)	102.65(12)
C(14)-C(2)-C(3)	111.42(13)
N(1)-C(3)-C(4)	112.69(13)
N(1)-C(3)-C(2)	100.77(12)
C(4)-C(3)-C(2)	111.90(13)
C(5)-C(4)-C(3)	123.92(17)
N(1)-C(6)-C(7)	108.92(14)
N(1)-C(6)-C(9)	108.62(13)
C(7)-C(6)-C(9)	108.87(15)
N(1)-C(6)-C(8)	111.20(14)
C(7)-C(6)-C(8)	110.68(15)
C(9)-C(6)-C(8)	108.49(14)
N(2)-C(10)-C(13)	108.46(15)
N(2)-C(10)-C(11)	110.30(15)
C(13)-C(10)-C(11)	109.21(17)
N(2)-C(10)-C(12)	109.65(15)
C(13)-C(10)-C(12)	110.62(17)
C(11)-C(10)-C(12)	108.60(18)
C(15)-C(14)-C(2)	113.11(13)

C(14)-C(15)-C(16)	114.20(13)
N(3)-C(16)-C(15)	112.86(13)
N(3)-C(16)-C(17)	99.97(12)
C(15)-C(16)-C(17)	111.87(13)
N(4)-C(17)-C(18)	112.78(13)
N(4)-C(17)-C(16)	100.06(12)
C(18)-C(17)-C(16)	111.56(13)
C(19)-C(18)-C(17)	124.60(17)
O(2)-C(20)-N(3)	126.45(15)
O(2)-C(20)-N(4)	125.93(15)
N(3)-C(20)-N(4)	107.63(13)
N(3)-C(21)-C(23)	108.30(16)
N(3)-C(21)-C(22)	108.92(14)
C(23)-C(21)-C(22)	107.92(16)
N(3)-C(21)-C(24)	110.72(15)
C(23)-C(21)-C(24)	110.95(17)
C(22)-C(21)-C(24)	109.95(17)
N(4)-C(25)-C(28)	109.12(14)
N(4)-C(25)-C(27)	110.40(14)
C(28)-C(25)-C(27)	109.75(16)
N(4)-C(25)-C(26)	107.96(14)
C(28)-C(25)-C(26)	108.10(16)
C(27)-C(25)-C(26)	111.44(16)
C(1A)-N(1A)-C(2A)	110.45(13)
C(1A)-N(1A)-C(10A)	123.48(14)
C(2A)-N(1A)-C(10A)	125.05(14)
C(1A)-N(2A)-C(3A)	109.74(13)
C(1A)-N(2A)-C(6A)	122.51(14)
C(3A)-N(2A)-C(6A)	123.92(13)
C(20A)-N(3A)-C(16A)	109.40(13)
C(20A)-N(3A)-C(21A)	123.56(14)
C(16A)-N(3A)-C(21A)	125.56(13)
C(20A)-N(4A)-C(17A)	109.44(13)
C(20A)-N(4A)-C(25A)	122.96(13)
C(17A)-N(4A)-C(25A)	124.04(13)
O(1A)-C(1A)-N(1A)	126.19(15)

O(1A)-C(1A)-N(2A)	126.03(15)
N(1A)-C(1A)-N(2A)	107.78(14)
N(1A)-C(2A)-C(14A)	112.70(14)
N(1A)-C(2A)-C(3A)	102.04(12)
C(14A)-C(2A)-C(3A)	111.41(13)
N(2A)-C(3A)-C(4A)	113.34(13)
N(2A)-C(3A)-C(2A)	100.86(12)
C(4A)-C(3A)-C(2A)	111.41(14)
C(5A)-C(4A)-C(3A)	123.36(18)
N(2A)-C(6A)-C(7A)	109.04(15)
N(2A)-C(6A)-C(9A)	108.81(14)
C(7A)-C(6A)-C(9A)	108.29(15)
N(2A)-C(6A)-C(8A)	110.40(14)
C(7A)-C(6A)-C(8A)	110.93(16)
C(9A)-C(6A)-C(8A)	109.32(15)
N(1A)-C(10A)-C(12A)	110.49(15)
N(1A)-C(10A)-C(13A)	108.57(15)
C(12A)-C(10A)-C(13A)	108.76(17)
N(1A)-C(10A)-C(11A)	109.75(15)
C(12A)-C(10A)-C(11A)	108.44(16)
C(13A)-C(10A)-C(11A)	110.83(16)
C(2A)-C(14A)-C(15A)	112.20(13)
C(16A)-C(15A)-C(14A)	113.50(14)
N(3A)-C(16A)-C(15A)	112.98(14)
N(3A)-C(16A)-C(17A)	100.51(12)
C(15A)-C(16A)-C(17A)	111.63(13)
N(4A)-C(17A)-C(18A)	113.10(14)
N(4A)-C(17A)-C(16A)	99.95(13)
C(18A)-C(17A)-C(16A)	111.38(14)
C(19A)-C(18A)-C(17A)	124.50(18)
O(2A)-C(20A)-N(3A)	126.56(16)
O(2A)-C(20A)-N(4A)	125.80(15)
N(3A)-C(20A)-N(4A)	107.64(14)
N(3A)-C(21A)-C(24A)	110.98(14)
N(3A)-C(21A)-C(22A)	108.72(14)
C(24A)-C(21A)-C(22A)	110.63(16)

N(3A)-C(21A)-C(23A)	108.69(14)
C(24A)-C(21A)-C(23A)	109.56(15)
C(22A)-C(21A)-C(23A)	108.18(15)
N(4A)-C(25A)-C(26A)	108.78(14)
N(4A)-C(25A)-C(27A)	111.06(14)
C(26A)-C(25A)-C(27A)	110.48(15)
N(4A)-C(25A)-C(28A)	108.98(13)
C(26A)-C(25A)-C(28A)	108.40(15)
C(27A)-C(25A)-C(28A)	109.08(15)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for ys161r_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}
O(1)	28(1)	35(1)	20(1)	-1(1)	5(1)	0(1)
O(2)	26(1)	35(1)	23(1)	-7(1)	9(1)	-10(1)
N(1)	20(1)	23(1)	17(1)	-1(1)	2(1)	1(1)
N(2)	21(1)	37(1)	18(1)	8(1)	4(1)	3(1)
N(3)	20(1)	29(1)	17(1)	-7(1)	4(1)	-5(1)
N(4)	18(1)	24(1)	16(1)	-3(1)	1(1)	-2(1)
C(1)	22(1)	24(1)	18(1)	1(1)	-1(1)	-3(1)
C(2)	19(1)	22(1)	16(1)	1(1)	1(1)	0(1)
C(3)	19(1)	20(1)	17(1)	0(1)	2(1)	-1(1)
C(4)	22(1)	23(1)	26(1)	-2(1)	0(1)	0(1)
C(5)	37(1)	30(1)	29(1)	-7(1)	-4(1)	-1(1)
C(6)	18(1)	22(1)	22(1)	1(1)	0(1)	1(1)
C(7)	38(1)	27(1)	31(1)	-5(1)	-2(1)	7(1)
C(8)	21(1)	34(1)	39(1)	4(1)	0(1)	0(1)
C(9)	28(1)	31(1)	23(1)	4(1)	-2(1)	5(1)
C(10)	28(1)	29(1)	19(1)	7(1)	2(1)	1(1)
C(11)	33(1)	66(2)	41(1)	26(1)	4(1)	14(1)
C(12)	59(1)	38(1)	26(1)	1(1)	-12(1)	5(1)
C(13)	41(1)	27(1)	47(1)	6(1)	4(1)	1(1)
C(14)	19(1)	20(1)	18(1)	-2(1)	-1(1)	0(1)
C(15)	20(1)	20(1)	16(1)	2(1)	0(1)	-2(1)
C(16)	19(1)	21(1)	15(1)	-2(1)	3(1)	-2(1)
C(17)	19(1)	21(1)	16(1)	-1(1)	1(1)	-1(1)
C(18)	25(1)	23(1)	28(1)	2(1)	2(1)	3(1)
C(19)	39(1)	33(1)	33(1)	9(1)	-2(1)	4(1)
C(20)	20(1)	22(1)	20(1)	-3(1)	1(1)	-3(1)
C(21)	28(1)	38(1)	21(1)	-13(1)	5(1)	-13(1)
C(22)	29(1)	43(1)	33(1)	-18(1)	6(1)	-16(1)
C(23)	35(1)	48(1)	48(1)	-31(1)	19(1)	-16(1)
C(24)	45(1)	60(1)	22(1)	-4(1)	-5(1)	-21(1)
C(25)	19(1)	28(1)	24(1)	-5(1)	-1(1)	-4(1)

C(26)	40(1)	32(1)	33(1)	-2(1)	0(1)	-16(1)
C(27)	20(1)	44(1)	46(1)	-6(1)	-2(1)	0(1)
C(28)	30(1)	47(1)	25(1)	-9(1)	-3(1)	-13(1)
O(1A)	39(1)	25(1)	17(1)	5(1)	2(1)	2(1)
O(2A)	34(1)	26(1)	23(1)	8(1)	0(1)	-4(1)
N(1A)	43(1)	18(1)	18(1)	2(1)	9(1)	2(1)
N(2A)	26(1)	20(1)	14(1)	0(1)	1(1)	1(1)
N(3A)	29(1)	20(1)	20(1)	4(1)	-3(1)	-4(1)
N(4A)	25(1)	20(1)	18(1)	3(1)	1(1)	-3(1)
C(1A)	26(1)	20(1)	17(1)	-1(1)	2(1)	-2(1)
C(2A)	25(1)	16(1)	16(1)	2(1)	0(1)	-1(1)
C(3A)	23(1)	19(1)	17(1)	2(1)	1(1)	-1(1)
C(4A)	28(1)	20(1)	27(1)	-1(1)	-5(1)	-1(1)
C(5A)	43(1)	32(1)	30(1)	-2(1)	-12(1)	0(1)
C(6A)	29(1)	24(1)	22(1)	1(1)	2(1)	6(1)
C(7A)	34(1)	49(1)	35(1)	4(1)	0(1)	16(1)
C(8A)	45(1)	21(1)	33(1)	-4(1)	9(1)	1(1)
C(9A)	37(1)	31(1)	23(1)	0(1)	8(1)	6(1)
C(10A)	37(1)	24(1)	19(1)	-2(1)	6(1)	1(1)
C(11A)	43(1)	42(1)	27(1)	-12(1)	0(1)	0(1)
C(12A)	68(2)	23(1)	35(1)	-1(1)	21(1)	6(1)
C(13A)	30(1)	30(1)	40(1)	5(1)	4(1)	4(1)
C(14A)	25(1)	19(1)	19(1)	1(1)	-4(1)	-1(1)
C(15A)	24(1)	20(1)	20(1)	2(1)	1(1)	-2(1)
C(16A)	25(1)	19(1)	18(1)	3(1)	0(1)	-2(1)
C(17A)	25(1)	19(1)	18(1)	2(1)	0(1)	-2(1)
C(18A)	25(1)	27(1)	28(1)	1(1)	2(1)	1(1)
C(19A)	33(1)	44(1)	33(1)	-6(1)	8(1)	2(1)
C(20A)	23(1)	19(1)	21(1)	2(1)	2(1)	2(1)
C(21A)	27(1)	24(1)	25(1)	3(1)	-9(1)	-2(1)
C(22A)	36(1)	30(1)	37(1)	7(1)	-15(1)	-8(1)
C(23A)	34(1)	31(1)	35(1)	9(1)	-10(1)	1(1)
C(24A)	42(1)	36(1)	26(1)	-6(1)	-9(1)	4(1)
C(25A)	23(1)	17(1)	24(1)	0(1)	-1(1)	-2(1)
C(26A)	28(1)	35(1)	32(1)	-3(1)	4(1)	-6(1)
C(27A)	35(1)	19(1)	50(1)	2(1)	-2(1)	-1(1)

C(28A)	34(1)	25(1)	28(1)	-3(1)	-4(1)	-7(1)

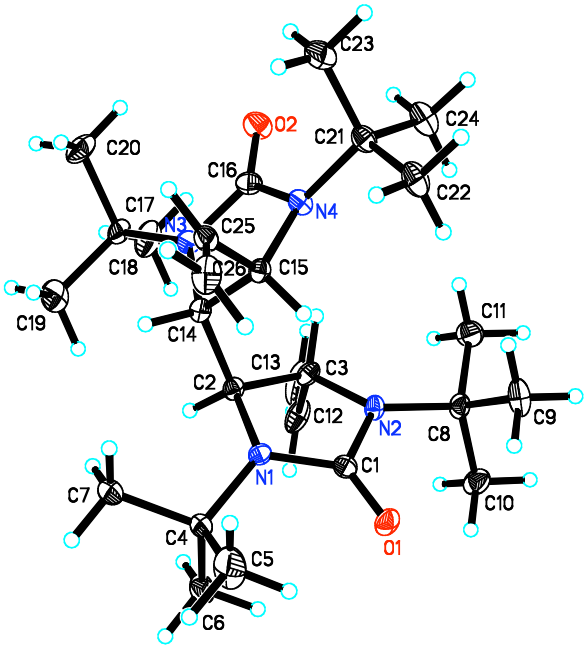
Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for ys161r_0m.

	x	y	z	U(eq)
H(2A)	-3441	244	2655	23
H(3A)	-4148	1492	2062	22
H(4A)	-5116	-33	2357	28
H(5A)	-4583	552	1336	38
H(5B)	-5161	-310	1460	38
H(7A)	-5134	3253	2873	48
H(7B)	-5770	2797	3296	48
H(7C)	-6153	3320	2790	48
H(8A)	-6595	1473	3007	47
H(8B)	-6494	1124	2397	47
H(8C)	-6995	2002	2509	47
H(9A)	-5015	2705	1923	41
H(9B)	-6039	2745	1853	41
H(9C)	-5536	1867	1742	41
H(11A)	-2883	-394	3317	70
H(11B)	-2775	-468	3965	70
H(11C)	-2506	396	3658	70
H(12A)	-3440	1220	4279	61
H(12B)	-3730	359	4583	61
H(12C)	-4441	990	4334	61
H(13A)	-4425	-780	3391	58
H(13B)	-5045	-256	3791	58
H(13C)	-4318	-871	4037	58
H(14A)	-3095	2055	2697	23
H(14B)	-2521	1405	3046	23
H(15A)	-1934	1819	2156	22
H(15B)	-2666	1236	1889	22
H(16A)	-2047	7	2385	22
H(17A)	-1119	1088	2952	22
H(18A)	-386	-561	2647	31

H(19A)	-801	74	3676	42
H(19B)	-362	-861	3538	42
H(22A)	-2874	-109	1555	52
H(22B)	-2327	-947	1701	52
H(22C)	-2688	-814	1096	52
H(23A)	-453	-431	925	66
H(23B)	-1244	-998	714	66
H(23C)	-864	-1147	1313	66
H(24A)	-2220	1061	989	64
H(24B)	-2076	378	510	64
H(24C)	-1283	933	731	64
H(26A)	119	2688	2136	52
H(26B)	671	2134	1713	52
H(26C)	1139	2602	2212	52
H(27A)	1284	700	2009	55
H(27B)	1124	372	2619	55
H(27C)	1765	1155	2508	55
H(28A)	-71	2203	3076	51
H(28B)	942	2064	3157	51
H(28C)	288	1293	3274	51
H(2AA)	7419	5826	-208	23
H(3AA)	6292	6684	394	24
H(4AA)	7955	7422	109	30
H(5AA)	7287	6936	1119	42
H(5AB)	8232	7378	1007	42
H(7AA)	4686	8004	-393	59
H(7AB)	5262	8561	-802	59
H(7AC)	4811	9008	-289	59
H(8AA)	6719	9112	-490	49
H(8AB)	7042	8904	115	49
H(8AC)	6270	9566	21	49
H(9AA)	5174	7726	543	45
H(9AB)	5343	8723	650	45
H(9AC)	6099	8044	746	45
H(11D)	6416	6018	-1807	56
H(11E)	7289	6181	-2137	56

H(11F)	6780	6972	-1876	56
H(12D)	7167	4979	-1219	63
H(12E)	8016	5247	-891	63
H(12F)	8049	5142	-1540	63
H(13D)	8608	6721	-979	50
H(13E)	8143	7403	-1367	50
H(13F)	8641	6604	-1627	50
H(14C)	6150	5111	-621	25
H(14D)	5570	5782	-291	25
H(15C)	6289	5243	541	26
H(15D)	5587	4624	272	26
H(16B)	7395	4409	35	25
H(17B)	6133	3652	-485	25
H(18B)	7647	2662	-196	32
H(19C)	7068	3188	-1220	44
H(19D)	7916	2590	-1093	44
H(22D)	7775	5213	817	51
H(22E)	8472	4487	685	51
H(22F)	8451	4928	1274	51
H(23D)	7605	2853	1538	50
H(23E)	8343	3513	1714	50
H(23F)	8377	3066	1127	50
H(24D)	6474	3957	1727	52
H(24E)	6542	4885	1452	52
H(24F)	7213	4608	1915	52
H(26D)	4856	2166	816	47
H(26E)	4300	1733	341	47
H(26F)	4372	2754	378	47
H(27D)	6471	1339	-98	52
H(27E)	5578	866	28	52
H(27F)	6130	1269	516	52
H(28D)	5690	2271	-768	44
H(28E)	4880	2827	-573	44
H(28F)	4801	1807	-621	44

X-ray structure for 11



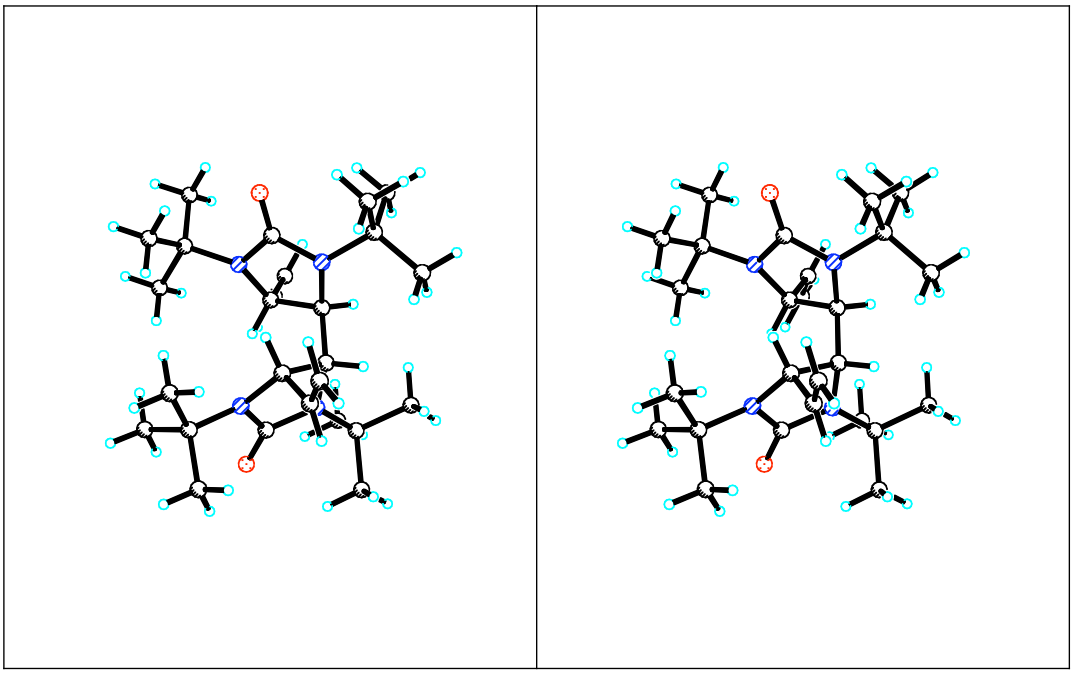


Table 1. Crystal data and structure refinement for **11**.

Identification code	156r	
Empirical formula	C26 H46 N4 O2	
Formula weight	446.67	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 17.640 Å	α= 90°.
	b = 17.3703(12) Å	β= 90°.
	c = 17.6396(13) Å	γ = 90°.
Volume	5404.9(5) $Å^3$	
Z	8	
Density (calculated)	1.098 Mg/m^3	
Absorption coefficient	0.070 mm^{-1}	
F(000)	1968	
Crystal size	0.34 x 0.31 x 0.06 mm^3	
Theta range for data collection	2.01 to 30.67°.	
Index ranges	-25<=h<=12, -21<=k<=17, -22<=l<=24	
Reflections collected	23888	
Independent reflections	13025 [R(int) = 0.1155]	
Completeness to theta = 30.67°	77.8 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9957 and 0.9767	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	13025 / 0 / 578	
Goodness-of-fit on F^2	0.632	
Final R indices [I>2sigma(I)]	R1 = 0.0583, wR2 = 0.1060	
R indices (all data)	R1 = 0.1831, wR2 = 0.1471	
Extinction coefficient	0.00074(15)	
Largest diff. peak and hole	0.266 and -0.198 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for 156r. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1A)	4254(1)	2029(1)	2759(1)	24(1)
O(2A)	1406(1)	-132(1)	4640(1)	28(1)
N(1A)	3824(1)	1088(1)	3577(1)	17(1)
N(2A)	3294(1)	1193(1)	2399(1)	15(1)
N(3A)	1746(1)	24(1)	3361(1)	18(1)
N(4A)	1802(1)	1027(1)	4156(1)	18(1)
C(1A)	3840(1)	1487(2)	2895(1)	19(1)
C(2A)	3451(1)	350(2)	3462(1)	18(1)
C(3A)	2948(1)	509(2)	2740(1)	15(1)
C(4A)	4361(1)	1248(2)	4222(1)	20(1)
C(5A)	4153(2)	739(2)	4911(1)	28(1)
C(6A)	4264(2)	2069(2)	4466(2)	34(1)
C(7A)	5185(2)	1093(2)	3977(2)	28(1)
C(8A)	3357(2)	1288(2)	1544(1)	25(1)
C(9A)	3417(2)	2131(2)	1350(2)	55(1)
C(10A)	4057(2)	854(2)	1271(2)	50(1)
C(11A)	2642(2)	980(2)	1147(1)	25(1)
C(12A)	4009(2)	-270(2)	3288(2)	27(1)
C(13A)	4068(2)	-913(2)	3683(2)	47(1)
C(14A)	2090(1)	628(2)	2903(1)	15(1)
C(15A)	1888(1)	1343(2)	3376(1)	16(1)
C(16A)	1625(1)	265(2)	4108(1)	20(1)
C(17A)	1483(1)	-731(2)	3081(1)	21(1)
C(18A)	1545(2)	-764(2)	2202(1)	36(1)
C(19A)	653(2)	-851(2)	3288(2)	33(1)
C(20A)	1973(2)	-1356(2)	3431(2)	36(1)
C(21A)	1573(2)	1494(2)	4839(1)	22(1)
C(22A)	1840(2)	2310(2)	4714(2)	28(1)
C(23A)	711(2)	1478(2)	4945(2)	36(1)
C(24A)	1965(2)	1184(2)	5563(1)	31(1)

C(25A)	1182(2)	1698(2)	3044(1)	20(1)
C(26A)	1159(2)	2353(2)	2681(1)	30(1)
O(1)	748(1)	7030(1)	2758(1)	23(1)
O(2)	3593(1)	4866(1)	4640(1)	28(1)
N(1)	1708(1)	6193(1)	2399(1)	17(1)
N(2)	1177(1)	6088(1)	3576(1)	17(1)
N(3)	3252(1)	5027(1)	3362(1)	19(1)
N(4)	3198(1)	6030(1)	4158(1)	18(1)
C(1)	1161(1)	6489(2)	2897(1)	18(1)
C(2)	2050(1)	5510(2)	2741(1)	16(1)
C(3)	1549(1)	5351(2)	3458(1)	19(1)
C(4)	1643(2)	6290(2)	1546(1)	26(1)
C(5)	1584(2)	7130(2)	1351(2)	55(1)
C(6)	942(2)	5856(2)	1270(2)	48(1)
C(7)	2358(2)	5982(2)	1149(1)	24(1)
C(8)	638(1)	6248(2)	4224(1)	20(1)
C(9)	731(2)	7077(2)	4462(2)	33(1)
C(10)	-185(1)	6087(2)	3978(2)	27(1)
C(11)	849(2)	5740(2)	4907(1)	29(1)
C(12)	989(2)	4729(2)	3291(2)	29(1)
C(13)	930(2)	4088(2)	3681(2)	47(1)
C(14)	2914(1)	5629(2)	2903(1)	16(1)
C(15)	3110(1)	6347(2)	3372(1)	16(1)
C(16)	3373(1)	5267(2)	4108(1)	21(1)
C(17)	3514(1)	4267(2)	3079(1)	19(1)
C(18)	3024(2)	3642(2)	3430(2)	35(1)
C(19)	3451(2)	4236(2)	2204(2)	35(1)
C(20)	4349(2)	4153(2)	3293(2)	33(1)
C(21)	3426(2)	6495(2)	4839(1)	22(1)
C(22)	3156(2)	7307(2)	4714(2)	29(1)
C(23)	4289(2)	6478(2)	4946(2)	34(1)
C(24)	3036(2)	6182(2)	5560(1)	31(1)
C(25)	3820(1)	6699(2)	3045(1)	21(1)
C(26)	3839(2)	7351(2)	2679(1)	28(1)

Table 3. Bond lengths [Å] and angles [°] for 156r.

O(1A)-C(1A)	1.216(3)
O(2A)-C(16A)	1.226(3)
N(1A)-C(1A)	1.389(3)
N(1A)-C(2A)	1.455(3)
N(1A)-C(4A)	1.506(3)
N(2A)-C(1A)	1.398(3)
N(2A)-C(3A)	1.465(3)
N(2A)-C(8A)	1.522(3)
N(3A)-C(16A)	1.399(3)
N(3A)-C(14A)	1.456(3)
N(3A)-C(17A)	1.476(3)
N(4A)-C(16A)	1.363(3)
N(4A)-C(15A)	1.489(3)
N(4A)-C(21A)	1.507(3)
C(2A)-C(12A)	1.491(4)
C(2A)-C(3A)	1.576(3)
C(3A)-C(14A)	1.556(3)
C(4A)-C(6A)	1.500(4)
C(4A)-C(7A)	1.539(3)
C(4A)-C(5A)	1.546(4)
C(8A)-C(9A)	1.508(4)
C(8A)-C(10A)	1.525(4)
C(8A)-C(11A)	1.539(4)
C(12A)-C(13A)	1.321(4)
C(14A)-C(15A)	1.537(3)
C(15A)-C(25A)	1.510(3)
C(17A)-C(20A)	1.518(4)
C(17A)-C(19A)	1.524(3)
C(17A)-C(18A)	1.555(4)
C(21A)-C(22A)	1.511(4)
C(21A)-C(23A)	1.533(4)
C(21A)-C(24A)	1.549(4)
C(25A)-C(26A)	1.306(4)
O(1)-C(1)	1.215(3)

O(2)-C(16)	1.231(3)
N(1)-C(1)	1.403(3)
N(1)-C(2)	1.460(3)
N(1)-C(4)	1.519(3)
N(2)-C(1)	1.385(3)
N(2)-C(3)	1.452(3)
N(2)-C(8)	1.512(3)
N(3)-C(16)	1.398(3)
N(3)-C(14)	1.451(3)
N(3)-C(17)	1.483(3)
N(4)-C(16)	1.364(3)
N(4)-C(15)	1.500(3)
N(4)-C(21)	1.501(3)
C(2)-C(14)	1.565(3)
C(2)-C(3)	1.567(3)
C(3)-C(12)	1.494(4)
C(4)-C(5)	1.503(4)
C(4)-C(6)	1.527(4)
C(4)-C(7)	1.539(4)
C(8)-C(9)	1.509(4)
C(8)-C(10)	1.540(4)
C(8)-C(11)	1.540(4)
C(12)-C(13)	1.313(4)
C(14)-C(15)	1.535(4)
C(15)-C(25)	1.509(3)
C(17)-C(18)	1.520(4)
C(17)-C(20)	1.534(4)
C(17)-C(19)	1.550(4)
C(21)-C(22)	1.505(4)
C(21)-C(23)	1.534(4)
C(21)-C(24)	1.545(4)
C(25)-C(26)	1.304(4)

C(1A)-N(1A)-C(2A)	109.1(2)
C(1A)-N(1A)-C(4A)	123.3(2)
C(2A)-N(1A)-C(4A)	123.6(2)

C(1A)-N(2A)-C(3A)	109.00(19)
C(1A)-N(2A)-C(8A)	122.08(19)
C(3A)-N(2A)-C(8A)	121.7(2)
C(16A)-N(3A)-C(14A)	111.7(2)
C(16A)-N(3A)-C(17A)	122.2(2)
C(14A)-N(3A)-C(17A)	125.83(19)
C(16A)-N(4A)-C(15A)	108.9(2)
C(16A)-N(4A)-C(21A)	120.7(2)
C(15A)-N(4A)-C(21A)	124.6(2)
O(1A)-C(1A)-N(1A)	124.7(2)
O(1A)-C(1A)-N(2A)	124.9(2)
N(1A)-C(1A)-N(2A)	110.3(2)
N(1A)-C(2A)-C(12A)	111.5(2)
N(1A)-C(2A)-C(3A)	102.3(2)
C(12A)-C(2A)-C(3A)	109.4(2)
N(2A)-C(3A)-C(14A)	111.9(2)
N(2A)-C(3A)-C(2A)	103.85(19)
C(14A)-C(3A)-C(2A)	114.94(18)
C(6A)-C(4A)-N(1A)	108.6(2)
C(6A)-C(4A)-C(7A)	110.8(2)
N(1A)-C(4A)-C(7A)	110.5(2)
C(6A)-C(4A)-C(5A)	106.9(2)
N(1A)-C(4A)-C(5A)	109.8(2)
C(7A)-C(4A)-C(5A)	110.2(2)
C(9A)-C(8A)-N(2A)	109.6(2)
C(9A)-C(8A)-C(10A)	110.7(3)
N(2A)-C(8A)-C(10A)	108.6(2)
C(9A)-C(8A)-C(11A)	107.0(2)
N(2A)-C(8A)-C(11A)	110.66(19)
C(10A)-C(8A)-C(11A)	110.3(2)
C(13A)-C(12A)-C(2A)	123.7(3)
N(3A)-C(14A)-C(15A)	100.64(17)
N(3A)-C(14A)-C(3A)	114.3(2)
C(15A)-C(14A)-C(3A)	115.6(2)
N(4A)-C(15A)-C(25A)	115.13(19)
N(4A)-C(15A)-C(14A)	103.2(2)

C(25A)-C(15A)-C(14A)	108.04(19)
O(2A)-C(16A)-N(4A)	124.8(2)
O(2A)-C(16A)-N(3A)	126.9(3)
N(4A)-C(16A)-N(3A)	108.3(2)
N(3A)-C(17A)-C(20A)	108.7(2)
N(3A)-C(17A)-C(19A)	110.1(2)
C(20A)-C(17A)-C(19A)	110.5(2)
N(3A)-C(17A)-C(18A)	110.1(2)
C(20A)-C(17A)-C(18A)	109.8(2)
C(19A)-C(17A)-C(18A)	107.6(2)
C(22A)-C(21A)-N(4A)	107.8(2)
C(22A)-C(21A)-C(23A)	110.2(2)
N(4A)-C(21A)-C(23A)	110.7(2)
C(22A)-C(21A)-C(24A)	107.9(2)
N(4A)-C(21A)-C(24A)	110.6(2)
C(23A)-C(21A)-C(24A)	109.6(2)
C(26A)-C(25A)-C(15A)	124.9(3)
C(1)-N(1)-C(2)	108.83(19)
C(1)-N(1)-C(4)	121.9(2)
C(2)-N(1)-C(4)	122.0(2)
C(1)-N(2)-C(3)	109.21(19)
C(1)-N(2)-C(8)	123.2(2)
C(3)-N(2)-C(8)	123.6(2)
C(16)-N(3)-C(14)	111.8(2)
C(16)-N(3)-C(17)	122.3(2)
C(14)-N(3)-C(17)	125.59(19)
C(16)-N(4)-C(15)	108.7(2)
C(16)-N(4)-C(21)	120.9(2)
C(15)-N(4)-C(21)	124.7(2)
O(1)-C(1)-N(2)	125.2(2)
O(1)-C(1)-N(1)	124.7(2)
N(2)-C(1)-N(1)	110.0(2)
N(1)-C(2)-C(14)	111.8(2)
N(1)-C(2)-C(3)	104.11(19)
C(14)-C(2)-C(3)	115.14(19)
N(2)-C(3)-C(12)	111.5(2)

N(2)-C(3)-C(2)	102.4(2)
C(12)-C(3)-C(2)	110.0(2)
C(5)-C(4)-N(1)	109.8(2)
C(5)-C(4)-C(6)	110.5(3)
N(1)-C(4)-C(6)	108.8(2)
C(5)-C(4)-C(7)	106.9(2)
N(1)-C(4)-C(7)	110.5(2)
C(6)-C(4)-C(7)	110.3(2)
N(2)-C(8)-C(9)	108.5(2)
N(2)-C(8)-C(10)	110.3(2)
C(9)-C(8)-C(10)	110.7(2)
N(2)-C(8)-C(11)	109.6(2)
C(9)-C(8)-C(11)	107.6(2)
C(10)-C(8)-C(11)	110.1(2)
C(13)-C(12)-C(3)	124.2(3)
N(3)-C(14)-C(15)	101.12(18)
N(3)-C(14)-C(2)	114.0(2)
C(15)-C(14)-C(2)	115.1(2)
N(4)-C(15)-C(25)	114.6(2)
N(4)-C(15)-C(14)	102.9(2)
C(25)-C(15)-C(14)	108.08(19)
O(2)-C(16)-N(4)	124.9(2)
O(2)-C(16)-N(3)	126.7(3)
N(4)-C(16)-N(3)	108.5(2)
N(3)-C(17)-C(18)	108.8(2)
N(3)-C(17)-C(20)	109.4(2)
C(18)-C(17)-C(20)	110.6(2)
N(3)-C(17)-C(19)	110.2(2)
C(18)-C(17)-C(19)	109.9(2)
C(20)-C(17)-C(19)	108.0(2)
N(4)-C(21)-C(22)	107.6(2)
N(4)-C(21)-C(23)	110.8(2)
C(22)-C(21)-C(23)	110.5(2)
N(4)-C(21)-C(24)	110.5(2)
C(22)-C(21)-C(24)	108.0(2)
C(23)-C(21)-C(24)	109.4(2)

C(26)-C(25)-C(15)	124.2(3)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for 156r. 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}
O(1A)	28(1)	16(1)	29(1)	3(1)	-3(1)	-9(1)
O(2A)	39(1)	21(1)	23(1)	3(1)	6(1)	-4(1)
N(1A)	22(1)	9(1)	19(1)	1(1)	-2(1)	-2(1)
N(2A)	21(1)	11(1)	14(1)	0(1)	1(1)	-3(1)
N(3A)	25(1)	10(2)	20(1)	-2(1)	2(1)	-6(1)
N(4A)	22(1)	14(2)	19(1)	0(1)	2(1)	1(1)
C(1A)	23(1)	17(2)	16(1)	-2(1)	2(1)	4(1)
C(2A)	20(1)	11(2)	24(1)	2(1)	-1(1)	-2(1)
C(3A)	18(1)	7(2)	21(1)	-3(1)	3(1)	-1(1)
C(4A)	24(1)	18(2)	18(1)	4(1)	-5(1)	-1(1)
C(5A)	25(2)	37(2)	23(1)	7(1)	-4(1)	-2(1)
C(6A)	41(2)	35(2)	27(2)	-7(1)	-11(1)	2(2)
C(7A)	23(1)	31(2)	29(2)	4(1)	-4(1)	-5(1)
C(8A)	30(2)	30(2)	15(1)	0(1)	2(1)	-10(1)
C(9A)	84(3)	49(3)	32(2)	20(2)	-26(2)	-35(2)
C(10A)	22(2)	100(3)	29(2)	-29(2)	9(1)	-14(2)
C(11A)	25(1)	31(2)	19(1)	2(1)	-1(1)	-3(1)
C(12A)	24(2)	13(2)	45(2)	-7(1)	-9(1)	-2(1)
C(13A)	39(2)	19(2)	82(3)	-5(2)	-32(2)	1(2)
C(14A)	17(1)	11(2)	16(1)	-1(1)	0(1)	-3(1)
C(15A)	16(1)	14(2)	17(1)	-2(1)	0(1)	-2(1)
C(16A)	23(1)	16(2)	20(1)	0(1)	1(1)	1(1)
C(17A)	20(1)	13(2)	30(1)	-3(1)	0(1)	-4(1)
C(18A)	54(2)	22(2)	30(2)	-7(1)	3(1)	-17(2)
C(19A)	22(2)	27(2)	51(2)	-8(2)	2(1)	-6(1)
C(20A)	36(2)	15(2)	57(2)	-5(2)	-10(1)	4(1)
C(21A)	29(2)	18(2)	19(1)	-3(1)	3(1)	3(1)
C(22A)	38(2)	17(2)	28(2)	-8(1)	-1(1)	2(1)
C(23A)	32(2)	44(2)	31(2)	-11(2)	8(1)	6(2)

C(24A)	40(2)	31(2)	22(1)	-4(1)	-2(1)	9(1)
C(25A)	22(1)	16(2)	23(1)	-4(1)	0(1)	3(1)
C(26A)	32(2)	23(2)	34(2)	-4(1)	-9(1)	6(1)
O(1)	26(1)	17(1)	25(1)	3(1)	2(1)	8(1)
O(2)	39(1)	22(1)	24(1)	3(1)	-5(1)	6(1)
N(1)	19(1)	14(2)	17(1)	2(1)	-1(1)	3(1)
N(2)	20(1)	12(1)	18(1)	1(1)	3(1)	1(1)
N(3)	26(1)	11(2)	20(1)	-1(1)	-4(1)	5(1)
N(4)	27(1)	8(2)	19(1)	0(1)	-2(1)	0(1)
C(1)	18(1)	18(2)	19(1)	-4(1)	-2(1)	-3(1)
C(2)	18(1)	12(2)	20(1)	-2(1)	0(1)	1(1)
C(3)	17(1)	14(2)	26(1)	3(1)	2(1)	2(1)
C(4)	30(2)	34(2)	15(1)	-1(1)	-1(1)	13(1)
C(5)	84(3)	52(3)	28(2)	25(2)	21(2)	35(2)
C(6)	23(2)	93(3)	29(2)	-29(2)	-6(1)	9(2)
C(7)	27(2)	29(2)	17(1)	2(1)	0(1)	3(1)
C(8)	20(1)	23(2)	18(1)	1(1)	6(1)	3(1)
C(9)	43(2)	29(2)	28(2)	-7(1)	13(1)	0(2)
C(10)	20(1)	33(2)	29(2)	5(1)	3(1)	3(1)
C(11)	26(2)	38(2)	23(1)	5(1)	3(1)	1(1)
C(12)	23(1)	16(2)	50(2)	-2(2)	10(1)	0(1)
C(13)	40(2)	24(2)	76(2)	-6(2)	31(2)	-6(2)
C(14)	18(1)	11(2)	17(1)	3(1)	3(1)	-1(1)
C(15)	18(1)	13(2)	18(1)	0(1)	1(1)	1(1)
C(16)	19(1)	21(2)	22(1)	1(1)	-2(1)	-2(1)
C(17)	20(1)	10(2)	28(1)	-3(1)	0(1)	5(1)
C(18)	36(2)	16(2)	54(2)	-5(2)	11(1)	2(1)
C(19)	49(2)	21(2)	34(2)	-8(1)	-2(1)	15(2)
C(20)	22(2)	25(2)	50(2)	-8(2)	-3(1)	9(1)
C(21)	27(1)	18(2)	21(1)	-4(1)	-1(1)	-3(1)
C(22)	42(2)	18(2)	27(2)	-8(1)	1(1)	-3(1)
C(23)	32(2)	40(2)	30(2)	-9(1)	-5(1)	-4(2)
C(24)	44(2)	28(2)	22(1)	-2(1)	3(1)	-5(2)
C(25)	21(1)	22(2)	19(1)	-5(1)	-1(1)	-1(1)
C(26)	30(2)	21(2)	34(2)	-2(1)	10(1)	-7(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 156r.

	x	y	z	U(eq)
H(2AA)	3130	210	3908	22
H(3AA)	3007	67	2381	18
H(5AA)	4505	844	5328	43
H(5AB)	4188	195	4767	43
H(5AC)	3635	856	5073	43
H(6AA)	4396	2412	4045	51
H(6AB)	4597	2175	4898	51
H(6AC)	3736	2157	4614	51
H(7AA)	5313	1423	3545	41
H(7AB)	5239	551	3830	41
H(7AC)	5528	1205	4400	41
H(9AA)	2959	2399	1523	83
H(9AB)	3468	2191	800	83
H(9AC)	3862	2352	1602	83
H(10D)	4508	1056	1527	76
H(10E)	4112	921	722	76
H(10F)	4001	305	1387	76
H(11D)	2198	1266	1325	37
H(11E)	2579	432	1266	37
H(11F)	2694	1044	598	37
H(12B)	4340	-199	2869	33
H(13A)	3744	-1000	4105	56
H(13B)	4435	-1289	3546	56
H(14B)	1814	658	2409	18
H(15B)	2315	1721	3362	19
H(18D)	1228	-359	1979	53
H(18E)	1372	-1268	2023	53
H(18F)	2074	-685	2051	53
H(19D)	597	-843	3841	50
H(19E)	481	-1350	3092	50

H(19F)	346	-439	3065	50
H(20D)	1923	-1340	3984	53
H(20E)	2504	-1271	3290	53
H(20F)	1807	-1860	3244	53
H(22D)	2392	2313	4644	41
H(22E)	1709	2625	5156	41
H(22F)	1595	2523	4262	41
H(23D)	464	1674	4485	53
H(23E)	571	1801	5378	53
H(23F)	545	947	5038	53
H(24D)	2515	1189	5492	46
H(24E)	1795	655	5659	46
H(24F)	1830	1510	5996	46
H(25B)	719	1425	3102	24
H(26A)	1610	2643	2611	36
H(26B)	691	2540	2488	36
H(2A)	1991	5069	2382	20
H(3A)	1871	5212	3904	22
H(5A)	2036	7400	1535	82
H(5B)	1132	7348	1592	82
H(5C)	1546	7189	800	82
H(6A)	491	6059	1525	72
H(6B)	997	5307	1388	72
H(6C)	890	5922	721	72
H(7A)	2803	6264	1331	36
H(7B)	2308	6052	600	36
H(7C)	2418	5433	1263	36
H(9A)	1255	7166	4623	50
H(9B)	386	7188	4884	50
H(9C)	611	7415	4034	50
H(10A)	-315	6417	3547	41
H(10B)	-528	6196	4401	41
H(10C)	-235	5546	3830	41
H(11A)	1372	5847	5060	43
H(11B)	801	5196	4767	43
H(11C)	506	5853	5331	43

H(12A)	653	4802	2876	35
H(13C)	1257	3997	4100	56
H(13D)	560	3715	3545	56
H(14A)	3190	5657	2409	19
H(15A)	2683	6725	3355	20
H(18A)	3071	3661	3983	53
H(18B)	3192	3137	3247	53
H(18C)	2494	3724	3287	53
H(19A)	2922	4311	2053	52
H(19B)	3628	3733	2023	52
H(19C)	3765	4643	1981	52
H(20A)	4402	4162	3846	49
H(20B)	4654	4567	3072	49
H(20C)	4526	3656	3098	49
H(22A)	3393	7516	4256	44
H(22B)	3297	7625	5152	44
H(22C)	2604	7309	4655	44
H(23A)	4536	6674	4488	51
H(23B)	4454	5947	5039	51
H(23C)	4427	6801	5380	51
H(24A)	2485	6186	5489	47
H(24B)	3169	6508	5994	47
H(24C)	3207	5654	5655	47
H(25A)	4284	6429	3110	25
H(26C)	3385	7636	2604	34
H(26D)	4306	7542	2487	34