A Mild Cu(I)-Catalyzed Regioselective Diamination of Conjugated Dienes

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Supporting Information

Representative diamination procedure (Table 1, entry 1). A 10 mL test tube containing CuCl (0.006 g, 0.06 mmol) was evacuated and then filled with argon three times, followed by the addition of benzene-*d*₆ (dried over sodium metal) (1.5 mL), P(OPh)₃ (0.0186 g, 0.06 mmol), and *trans*-1-phenylbutadiene (0.078 g, 0.6 mmol) successively with stirring. Upon addition of di-*t*-butyldiaziridinone (0.153 g, 0.9 mmol), the reaction mixture was stirred at room temperature for 6 h and purified by flash chromatography (silica gel, hexanes:ether = 7:1) to give the diamination product as a white solid (0.135 g, 75% yield).

Table 1, Entry 1

White solid; mp. 154-155 °C; IR (film) 1677 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.40-7.26 (m, 5H), 6.50 (d, *J* = 15.9 Hz, 1H), 6.37 (dd, *J* = 15.9, 8.4 Hz, 1H), 4.17 (ddd, *J* = 8.4, 8.4, 2.4 Hz, 1H), 3.44 (dd, *J* = 8.4, 8.1 Hz, 1H), 3.01 (dd, *J* = 8.1, 2.4 Hz, 1H), 1.38 (s, 9H), 1.33 (s, 9H); ¹³C NMR (75 MHz, CDCl₃) δ 160.7, 136.7, 131.7, 130.8, 128.9, 128.0, 126.5, 55.4, 53.9, 53.1, 48.3, 29.0, 27.6; Anal. Calcd for C₁₉H₂₈N₂O: C, 75.96; H, 9.39; N, 9.32. Found: C, 75.79; H, 9.47; N, 9.38.

Table 1, Entry 2

White solid; mp. 100-101 °C; IR (film) 1687 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 7.31 (d, *J* = 8.7 Hz, 2H), 6.87 (d, *J* = 8.7 Hz, 2H), 6.43 (d, *J* = 15.9 Hz, 1H), 6.22 (dd, *J* = 15.9, 8.4 Hz, 1H), 4.14 (ddd, *J* = 8.4, 8.4, 2.4 Hz, 1H), 3.81 (s, 3H), 3.42 (dd, *J* = 8.4, 8.4 Hz, 1H), 3.00 (dd, *J* = 8.4,

2.4 Hz, 1H), 1.37 (s, 9H), 1.32 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 160.7, 159.5, 130.2, 129.4, 127.7, 114.2, 55.6, 55.5, 53.9, 53.1, 48.4, 29.0, 27.6; Anal. Calcd for $\text{C}_{20}\text{H}_{30}\text{N}_2\text{O}_2$: C, 72.69; H, 9.15; N, 8.48. Found: C, 73.02; H, 9.40; N, 8.67.

Table 1, Entry 3

Yellow solid. mp. 123-124 °C; IR (film) 1689 cm^{-1} ; ^1H NMR (300 MHz, C_6D_6) δ 7.81 (d, J = 8.7 Hz, 2H), 6.66 (d, J = 8.7 Hz, 2H), 6.25 (dd, J = 16.2, 8.7 Hz, 1H), 5.94 (d, J = 16.2 Hz, 1H), 3.71 (ddd, J = 8.7, 7.8, 2.1 Hz, 1H), 3.01 (dd, J = 8.4, 7.8 Hz, 1H), 2.62 (dd, J = 8.4, 2.1 Hz, 1H), 1.42 (s, 9H), 1.36 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 160.5, 147.1, 143.0, 136.7, 128.8, 127.1, 124.2, 55.1, 54.0, 53.2, 47.9, 29.0, 27.5; Anal. Calcd for $\text{C}_{19}\text{H}_{27}\text{N}_3\text{O}_3$: C, 66.06; H, 7.88; N, 12.16. Found: C, 66.22; H, 8.06; N, 12.27.

Table 1, Entry 4

White solid; mp. 142-143 °C; IR (film): 1674 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.35 (d, J = 1.8 Hz, 1H), 6.37 (dd, J = 3.3, 1.8 Hz, 1H), 6.30-6.28 (m, 2H), 6.23 (d, J = 3.3 Hz, 1H), 4.14-4.06 (m, 1H), 3.41 (dd, J = 8.4, 8.4 Hz, 1H), 2.99 (dd, J = 8.4, 2.7 Hz, 1H), 1.36 (s, 9H), 1.31 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 160.6, 152.2, 142.3, 130.3, 119.2, 111.5, 108.3, 55.0, 53.9, 53.1, 48.3, 28.9, 27.5. Anal. Calcd for $\text{C}_{17}\text{H}_{26}\text{N}_2\text{O}_2$: C, 70.31; H, 9.02; N, 9.65. Found: C, 70.39; H, 9.10; N, 9.73.

Table 1, Entry 5

White solid; mp. 63-64 °C; IR (film): 1728, 1694 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.04 (dd, $J = 15.9, 9.0$ Hz, 1H), 5.96 (d, $J = 15.9$ Hz, 1H), 4.15 (ddd, $J = 9.0, 8.4, 2.7$ Hz, 1H), 3.75 (s, 3H), 3.41 (dd, $J = 8.4, 8.4$ Hz, 1H), 2.95 (dd, $J = 8.4, 2.7$ Hz, 1H), 1.33 (s, 9H), 1.30 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 166.6, 160.4, 149.6, 121.6, 54.1, 53.7, 53.3, 52.0, 47.2, 28.9, 27.5. Anal. Calcd for $\text{C}_{15}\text{H}_{26}\text{N}_2\text{O}_3$: C, 63.80; H, 9.28; N, 9.92. Found: C, 64.00; H, 9.50; N, 10.16.

Table 1, Entry 6

Colorless oil; IR (film): 1692 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 5.34 (m, 1H), 4.15 (ddd, $J = 9.6, 8.4, 3.0$ Hz, 1H), 3.30 (dd, $J = 8.4, 8.1$ Hz, 1H), 2.80 (dd, $J = 8.1, 3.0$ Hz, 1H), 1.67 (d, $J = 1.2$ Hz, 3H), 1.60 (d, $J = 1.2$ Hz, 3H), 1.28 (s, 9H), 1.27 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 160.9, 132.0, 127.7, 53.4, 53.0, 50.6, 48.3, 28.8, 27.5, 25.9, 18.0; HRMS Calcd for $\text{C}_{15}\text{H}_{29}\text{N}_2\text{O}$ ($M+1$): 253.2274. Found: 253.2278.

Table 1, Entry 7

White solid; mp. 136-137 °C; IR (film): 1690 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.44-7.36 (m, 3H), 7.33- 7.22 (m, 5H), 7.11 (dd, $J = 8.1, 1.5$ Hz, 2H), 6.33 (d, $J = 9.3$ Hz, 1H), 4.06 (ddd, J

= 9.3, 8.4, 3.0 Hz, 1H), 3.42 (dd, J = 8.4, 8.1 Hz, 1H), 3.17 (dd, J = 8.1, 3.0 Hz, 1H), 1.34 (s, 9H), 1.30 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 160.6, 141.6, 141.5, 139.0, 131.2, 129.7, 128.5, 128.4, 127.8, 127.7, 127.5, 53.4, 53.1, 51.4, 48.4, 29.0, 27.5; Anal. Calcd for $\text{C}_{25}\text{H}_{32}\text{N}_2\text{O}$: C, 79.75; H, 8.57; N, 7.44. Found: C, 79.62; H, 8.58; N, 7.52.

Table 1, Entry 8

Colorless oil; IR (film): 1694 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 6.11-5.94 (m, 2H), 5.73-5.61 (m, 2H), 3.96 (ddd, J = 8.4, 8.4, 2.1 Hz, 1H), 3.33 (dd, J = 8.4, 7.8 Hz, 1H), 2.90 (dd, J = 7.8, 2.1 Hz, 1H), 2.05 (dt, J = 7.2, 7.2 Hz, 2H), 1.32 (s, 9H), 1.28 (s, 9H), 1.41-1.25 (m, 6H), 0.86 (t, J = 6.9 Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ 160.6, 135.8, 132.7, 131.3, 129.3, 55.1, 53.8, 53.0, 48.4, 32.8, 31.6, 29.0, 28.9, 27.5, 22.7, 14.2; Anal. Calcd for $\text{C}_{20}\text{H}_{36}\text{N}_2\text{O}$: C, 74.95; H, 11.32; N, 8.74. Found: C, 75.14; H, 11.24; N, 8.74.

Table 1, Entry 9

Mixture of **A** and **B** (A/B = 1/1.3): Colorless oil; IR (film) 1690 cm^{-1} . **A**: ^1H NMR (300 MHz, CDCl_3) δ 5.68-5.03 (m, 2H), 3.93 (ddd, J = 8.1, 8.1, 2.1 Hz, 1H), 3.32 (dd, J = 8.1, 8.1 Hz, 1H), 2.88 (dd, J = 8.1, 2.1 Hz, 1H), 1.68 (d, J = 5.1 Hz, 3H), 1.35 (s, 9H), 1.34 (s, 9H). **B**: ^1H NMR (300 MHz, CDCl_3) 5.92 (ddd, J = 17.4, 10.2, 8.4 Hz, 1H), 5.18 (d, J = 17.4 Hz, 1H), 5.08 (d, J = 10.2 Hz, 1H), 3.50 (d, J = 8.4 Hz, 1H), 3.26 (q, J = 6.3 Hz, 1H), 1.33 (s, 9H), 1.29 (s, 9H), 1.20 (d, J = 6.3 Hz, 3H). **A+B**: ^{13}C NMR (75 MHz, CDCl_3) δ 160.7, 157.9, 140.1, 133.5, 126.6,

115.5, 63.6, 55.4, 55.2, 53.8, 53.1, 52.9, 52.6, 48.5, 29.1, 28.9, 28.9, 27.6, 21.2, 17.8; Anal. Calcd for $C_{19}H_{28}N_2O$: C, 70.54; H, 10.99; N, 11.75. Found: C, 70.36; H, 10.79; N, 12.00.

Table 1, Entry 10

White solid; mp. 53-54 °C; IR (film) 1690 cm^{-1} ; ^1H NMR (300 MHz, $CDCl_3$) δ 5.71-5.61 (m, 1H), 5.46 (dq, J = 11.1, 6.9 Hz, 1H), 4.31 (ddd, J = 8.7, 8.4, 2.7 Hz, 1H), 3.36 (dd, J = 8.4, 8.1 Hz, 1H), 2.85 (dd, J = 8.1, 2.7 Hz, 1H), 1.65 (dd, J = 6.9, 1.5 Hz, 3H), 1.33 (s, 9H), 1.30 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.9, 133.6, 124.2, 53.6, 53.0, 49.3, 48.2, 28.8, 27.5, 13.1. Anal. Calcd for $C_{14}H_{26}N_2O$: C, 70.54; H, 10.99; N, 11.75. Found: C, 70.60; H, 11.13; N, 12.00.

Table 1, Entry 11

White solid; IR (film) 1693 cm^{-1} ; ^1H NMR (300 MHz, $CDCl_3$) δ 7.40-7.12 (m, 5H), 6.49 (d, J = 11.7 Hz, 1H), 5.95 (dd, J = 11.7, 9.6 Hz, 1H), 4.47 (ddd, J = 9.6, 8.4, 2.4 Hz, 1H), 3.51 (dd, J = 8.4, 8.1 Hz, 1H), 3.13 (dd, J = 8.1, 2.4 Hz, 1H), 1.34 (s, 9H), 1.23 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 160.6, 136.5, 135.5, 129.5, 128.6, 128.5, 127.4, 53.6, 53.1, 49.9, 48.3, 28.8, 27.6; Anal. Calcd for $C_{19}H_{28}N_2O$: C, 75.96; H, 9.39; N, 9.32. Found: C, 75.90; H, 9.27; N, 9.55.

Table 1, Entry 12

Colorless oil; IR (film): 1686 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 5.26-5.20 (m, 1H), 3.18 (d, J = 8.4 Hz, 1H), 2.89 (d, J = 8.4 Hz, 1H), 1.70 (d, J = 1.2 Hz, 3H), 1.64 (d, J = 0.9 Hz, 3H), 1.38 (s, 3H), 1.33 (s, 9H), 1.27 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 160.1, 135.8, 131.7, 58.0, 56.4, 54.7, 52.9, 29.7, 29.3, 27.7, 27.0, 19.4; Anal. Calcd for $\text{C}_{16}\text{H}_{30}\text{N}_2\text{O}$: C, 72.13; H, 11.35; N, 10.51. Found: C, 71.90; H, 11.40; N, 10.70.

Table 1, Entry 13

White solid; mp. 68-69 $^{\circ}\text{C}$; IR (film): 1687 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 4.94 (s, 1H), 4.85 (s, 1H), 3.05 (d, J = 8.4 Hz, 1H), 2.74 (d, J = 8.4 Hz, 1H), 1.82 (s, 3H), 1.56 (s, 3H), 1.36 (s, 9H), 1.29 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 160.5, 149.7, 112.2, 62.2, 55.6, 54.4, 53.0, 29.2, 27.6, 25.0, 19.7; HRMS Calcd for $\text{C}_{15}\text{H}_{29}\text{N}_2\text{O}$ ($M+1$): 253.2274. Found: 253.2280.

Table 1, Entry 14

White solid; mp. 70-71 $^{\circ}\text{C}$; IR (film) 1697 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.45-7.42 (m, 2H), 7.39-7.30 (m, 3H), 7.23-7.17 (m, 3H), 7.05-7.02 (m, 2H), 5.58 (s, 1H), 5.39 (s, 1H), 3.73 (d,

$J = 8.4$ Hz, 1H), 3.53 (d, $J = 8.4$ Hz, 1H), 1.32 (s, 9H), 1.22 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 150.9, 146.7, 142.5, 139.8, 128.6, 128.4, 127.9, 127.8, 127.5, 126.1, 115.7, 83.5, 53.9, 53.4, 51.9, 31.5, 27.0; Anal. Calcd for $\text{C}_{25}\text{H}_{32}\text{N}_2\text{O}$: C, 79.75; H, 8.57; N, 7.44. Found: C, 79.80; H, 8.70; N, 7.60.

Table 1, Entry 15

Colorless oil; IR (film) 1698 cm^{-1} ; ^1H NMR (300 MHz, C_6D_6) δ 4.82 (d, $J = 1.8$ Hz, 1H), 3.91 (d, $J = 1.8$ Hz, 1H), 3.22 (d, $J = 10.2$ Hz, 1H), 3.14 (s, 3H), 3.11 (s, 3H), 3.04 (d, $J = 10.2$ Hz, 1H), 1.62 (s, 9H), 1.28 (s, 9H); ^{13}C NMR (75 MHz, C_6D_6) δ 163.7, 159.8, 89.7, 82.4, 55.2, 54.5, 53.0, 50.6, 49.0, 28.7, 27.6; Anal. Calcd for $\text{C}_{15}\text{H}_{28}\text{N}_2\text{O}_3$: C, 63.35; H, 9.92; N, 9.85. Found: C, 63.47; H, 9.75; N, 10.00.

Scheme 4

Mixture of **9a** and **9b** (**9a/9b** = 65/35): IR (film) 1675 cm^{-1} ; **9a** + **9b**: ^1H NMR (300 MHz, CDCl_3) δ 7.40-7.21 (m, 5H), 6.50 (d, $J = 15.9$ Hz, 1H), 6.37 (d, $J = 15.9$ Hz, 1H), 3.41 (s, 0.35H), 2.99 (s, 0.65H), 1.38 (s, 9H), 1.33 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 160.7, 136.6, 131.6, 130.7, 128.8, 128.0, 126.5, 54.9 (t, $J = 20.9$ Hz), 53.8, 53.1, 47.8 (t, $J = 20.9$ Hz), 28.9, 27.5. HRMS Calcd for $\text{C}_{19}\text{H}_{27}\text{D}_2\text{N}_2\text{O}$ ($\text{M}+1$): 303.2405. Found: 303.2406.

X-ray structure of 4b

Table 1. Crystal data and structure refinement for **4b**.

Identification code	ys160_0m	
Empirical formula	C ₂₀ H ₃₀ N ₂ O ₂	
Formula weight	330.46	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 8.0156(5) Å	$\alpha = 90^\circ$.
	b = 10.2134(6) Å	$\beta = 95.475(3)^\circ$.
	c = 23.4505(13) Å	$\gamma = 90^\circ$.
Volume	1911.1(2) Å ³	
Z	4	
Density (calculated)	1.149 Mg/m ³	
Absorption coefficient	0.074 mm ⁻¹	
F(000)	720	
Crystal size	0.28 x 0.14 x 0.06 mm ³	
Theta range for data collection	1.74 to 29.57°.	
Index ranges	-8 ≤ h ≤ 11, -11 ≤ k ≤ 14, -32 ≤ l ≤ 32	
Reflections collected	17507	
Independent reflections	5351 [R(int) = 0.0692]	
Completeness to theta = 29.57°	99.9 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9954 and 0.9796	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	5351 / 0 / 217	
Goodness-of-fit on F ²	1.000	
Final R indices [I > 2σ(I)]	R1 = 0.0568, wR2 = 0.1112	
R indices (all data)	R1 = 0.1188, wR2 = 0.1333	
Largest diff. peak and hole	0.252 and -0.263 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ys160_0m. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
O(1)	4918(2)	9730(1)	1422(1)	24(1)
O(2)	13025(2)	8774(1)	4946(1)	30(1)
N(1)	7456(2)	10838(1)	1420(1)	19(1)
N(2)	5756(2)	11321(1)	2099(1)	19(1)
C(1)	5926(2)	10546(2)	1627(1)	19(1)
C(2)	8118(2)	12023(2)	1708(1)	21(1)
C(3)	7388(2)	11925(2)	2285(1)	19(1)
C(4)	7727(2)	10594(2)	808(1)	24(1)
C(5)	9407(2)	11188(2)	689(1)	32(1)
C(6)	6316(2)	11229(2)	416(1)	30(1)
C(7)	7789(3)	9119(2)	707(1)	31(1)
C(8)	4427(2)	11097(2)	2492(1)	21(1)
C(9)	4515(2)	12192(2)	2936(1)	28(1)
C(10)	2711(2)	11155(2)	2148(1)	29(1)
C(11)	4658(2)	9771(2)	2792(1)	28(1)
C(12)	8455(2)	11100(2)	2708(1)	20(1)
C(13)	9221(2)	11554(2)	3195(1)	20(1)
C(14)	10279(2)	10831(2)	3633(1)	19(1)
C(15)	11018(2)	11495(2)	4106(1)	24(1)
C(16)	11948(2)	10851(2)	4554(1)	26(1)
C(17)	12155(2)	9506(2)	4527(1)	22(1)
C(18)	11474(2)	8823(2)	4046(1)	23(1)
C(19)	10552(2)	9477(2)	3607(1)	22(1)
C(20)	13432(3)	9395(2)	5487(1)	32(1)

Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for ys160_0m.

O(1)-C(1)	1.227(2)
O(2)-C(17)	1.371(2)
O(2)-C(20)	1.429(2)
N(1)-C(1)	1.393(2)
N(1)-C(2)	1.460(2)
N(1)-C(4)	1.493(2)
N(2)-C(1)	1.379(2)
N(2)-C(3)	1.474(2)
N(2)-C(8)	1.491(2)
C(2)-C(3)	1.529(2)
C(3)-C(12)	1.504(2)
C(4)-C(7)	1.527(2)
C(4)-C(5)	1.527(2)
C(4)-C(6)	1.531(3)
C(8)-C(10)	1.528(2)
C(8)-C(9)	1.524(2)
C(8)-C(11)	1.528(2)
C(12)-C(13)	1.328(2)
C(13)-C(14)	1.466(2)
C(14)-C(15)	1.386(2)
C(14)-C(19)	1.402(2)
C(15)-C(16)	1.393(2)
C(16)-C(17)	1.386(2)
C(17)-C(18)	1.392(2)
C(18)-C(19)	1.380(2)
C(17)-O(2)-C(20)	116.85(14)
C(1)-N(1)-C(2)	108.05(13)
C(1)-N(1)-C(4)	120.82(14)
C(2)-N(1)-C(4)	120.17(13)
C(1)-N(2)-C(3)	108.93(13)
C(1)-N(2)-C(8)	122.61(14)
C(3)-N(2)-C(8)	123.26(13)
O(1)-C(1)-N(2)	126.39(15)

O(1)-C(1)-N(1)	125.28(16)
N(2)-C(1)-N(1)	108.33(14)
N(1)-C(2)-C(3)	101.64(13)
N(2)-C(3)-C(12)	113.18(14)
N(2)-C(3)-C(2)	99.75(13)
C(12)-C(3)-C(2)	112.27(14)
N(1)-C(4)-C(7)	108.91(14)
N(1)-C(4)-C(5)	108.74(14)
C(7)-C(4)-C(5)	108.70(16)
N(1)-C(4)-C(6)	109.85(14)
C(7)-C(4)-C(6)	110.97(15)
C(5)-C(4)-C(6)	109.62(15)
N(2)-C(8)-C(10)	109.17(14)
N(2)-C(8)-C(9)	108.58(14)
C(10)-C(8)-C(9)	108.22(15)
N(2)-C(8)-C(11)	110.97(14)
C(10)-C(8)-C(11)	109.93(15)
C(9)-C(8)-C(11)	109.91(15)
C(13)-C(12)-C(3)	123.96(16)
C(12)-C(13)-C(14)	127.94(17)
C(15)-C(14)-C(19)	117.36(16)
C(15)-C(14)-C(13)	119.25(16)
C(19)-C(14)-C(13)	123.35(15)
C(14)-C(15)-C(16)	121.98(17)
C(17)-C(16)-C(15)	119.49(17)
O(2)-C(17)-C(16)	124.28(16)
O(2)-C(17)-C(18)	116.13(16)
C(16)-C(17)-C(18)	119.58(16)
C(19)-C(18)-C(17)	120.14(17)
C(18)-C(19)-C(14)	121.38(16)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ys160_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	24(1)	22(1)	27(1)	-3(1)	2(1)	-7(1)
O(2)	38(1)	27(1)	24(1)	4(1)	-4(1)	3(1)
N(1)	21(1)	18(1)	20(1)	-2(1)	4(1)	-3(1)
N(2)	16(1)	19(1)	20(1)	0(1)	2(1)	0(1)
C(1)	20(1)	15(1)	23(1)	4(1)	0(1)	1(1)
C(2)	22(1)	20(1)	21(1)	-1(1)	4(1)	-3(1)
C(3)	20(1)	17(1)	20(1)	-2(1)	3(1)	1(1)
C(4)	31(1)	21(1)	19(1)	-2(1)	6(1)	-4(1)
C(5)	36(1)	38(1)	26(1)	-5(1)	13(1)	-8(1)
C(6)	39(1)	30(1)	21(1)	4(1)	2(1)	-2(1)
C(7)	43(1)	24(1)	27(1)	-5(1)	12(1)	0(1)
C(8)	18(1)	24(1)	23(1)	5(1)	7(1)	2(1)
C(9)	27(1)	30(1)	28(1)	-1(1)	10(1)	4(1)
C(10)	20(1)	33(1)	34(1)	4(1)	5(1)	2(1)
C(11)	26(1)	28(1)	33(1)	8(1)	8(1)	3(1)
C(12)	19(1)	18(1)	24(1)	0(1)	6(1)	1(1)
C(13)	22(1)	16(1)	22(1)	-1(1)	5(1)	0(1)
C(14)	17(1)	20(1)	20(1)	0(1)	4(1)	-1(1)
C(15)	26(1)	19(1)	28(1)	-4(1)	1(1)	3(1)
C(16)	27(1)	28(1)	22(1)	-5(1)	-2(1)	4(1)
C(17)	22(1)	23(1)	22(1)	4(1)	3(1)	3(1)
C(18)	25(1)	17(1)	26(1)	1(1)	3(1)	0(1)
C(19)	22(1)	21(1)	23(1)	-4(1)	1(1)	-3(1)
C(20)	35(1)	37(1)	23(1)	6(1)	-1(1)	0(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ys160_0m.

	x	y	z	U(eq)
H(2A)	7721	12821	1496	25
H(2B)	9358	12020	1755	25
H(3A)	7225	12818	2446	23
H(5A)	9382	12136	752	49
H(5B)	9614	11011	291	49
H(5C)	10305	10797	947	49
H(6A)	6298	12173	490	45
H(6B)	5240	10845	493	45
H(6C)	6509	11075	15	45
H(7A)	6714	8727	780	46
H(7B)	8688	8733	967	46
H(7C)	8006	8950	309	46
H(9A)	4363	13038	2741	42
H(9B)	5611	12173	3160	42
H(9C)	3630	12068	3191	42
H(10A)	2571	12007	1957	43
H(10B)	1831	11037	2407	43
H(10C)	2629	10457	1860	43
H(11A)	4606	9072	2504	43
H(11B)	3766	9643	3045	43
H(11C)	5750	9748	3018	43
H(12A)	8589	10200	2621	24
H(13A)	9064	12458	3270	24
H(15A)	10886	12418	4127	29
H(16A)	12437	11330	4874	31
H(18A)	11644	7905	4020	27
H(19A)	10094	9000	3282	26
H(20A)	14051	8781	5750	48
H(20B)	12398	9662	5647	48
H(20C)	14126	10168	5436	48

X-ray structure of 4e

Table 1. Crystal data and structure refinement for **4e**.

Identification code	ys159_0m	
Empirical formula	C ₁₅ H ₂₆ N ₂ O ₃	
Formula weight	282.38	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 6.6652(3) Å	α = 109.931(2)°.
	b = 10.3560(5) Å	β = 91.021(3)°.
	c = 12.7813(6) Å	γ = 104.591(3)°.
Volume	797.41(6) Å ³	
Z	2	
Density (calculated)	1.176 Mg/m ³	
Absorption coefficient	0.082 mm ⁻¹	
F(000)	308	
Crystal size	0.49 x 0.16 x 0.08 mm ³	
Theta range for data collection	1.71 to 31.00°.	
Index ranges	-9 ≤ h ≤ 9, -14 ≤ k ≤ 14, -18 ≤ l ≤ 18	
Reflections collected	15010	
Independent reflections	4842 [R(int) = 0.0302]	
Completeness to theta = 31.00°	95.0 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.9938 and 0.9611	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	4842 / 0 / 182	
Goodness-of-fit on F ²	1.122	
Final R indices [I > 2σ(I)]	R1 = 0.0822, wR2 = 0.2410	
R indices (all data)	R1 = 0.0988, wR2 = 0.2495	
Extinction coefficient	0.002(5)	
Largest diff. peak and hole	0.609 and -0.380 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ys159_0m. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
O(1)	4072(3)	4135(2)	1478(2)	21(1)
O(2)	-3680(4)	748(3)	3751(2)	34(1)
O(3)	-6588(3)	1470(2)	3684(2)	25(1)
N(1)	940(3)	4409(2)	2132(2)	18(1)
N(2)	872(3)	2585(2)	531(2)	16(1)
C(1)	2157(4)	3755(3)	1389(2)	16(1)
C(2)	-1263(4)	2694(3)	601(2)	18(1)
C(3)	-1255(4)	3571(3)	1848(2)	18(1)
C(4)	1821(4)	5605(3)	3206(2)	21(1)
C(5)	3080(6)	5122(4)	3950(2)	34(1)
C(6)	3219(5)	6854(3)	2955(3)	30(1)
C(7)	40(5)	6093(3)	3816(3)	29(1)
C(8)	1564(4)	2070(3)	-604(2)	18(1)
C(9)	3498(4)	1561(3)	-541(2)	25(1)
C(10)	2022(5)	3281(3)	-1077(2)	25(1)
C(11)	-183(4)	813(3)	-1364(2)	23(1)
C(12)	-1950(4)	2617(3)	2520(2)	20(1)
C(13)	-3880(4)	2349(3)	2791(2)	20(1)
C(14)	-4637(4)	1429(3)	3447(2)	21(1)
C(15)	-7555(5)	645(4)	4343(3)	32(1)

Table 3. Bond lengths [Å] and angles [°] for ys159_0m.

O(1)-C(1)	1.228(3)
O(2)-C(14)	1.208(4)
O(3)-C(14)	1.349(3)
O(3)-C(15)	1.444(3)
N(1)-C(1)	1.372(3)
N(1)-C(3)	1.467(3)
N(1)-C(4)	1.487(3)
N(2)-C(1)	1.394(3)
N(2)-C(2)	1.457(3)
N(2)-C(8)	1.491(3)
C(2)-C(3)	1.541(4)
C(3)-C(12)	1.514(4)
C(4)-C(6)	1.526(4)
C(4)-C(7)	1.529(4)
C(4)-C(5)	1.538(4)
C(8)-C(9)	1.521(4)
C(8)-C(11)	1.526(4)
C(8)-C(10)	1.537(4)
C(12)-C(13)	1.324(4)
C(13)-C(14)	1.478(4)
C(14)-O(3)-C(15)	117.0(2)
C(1)-N(1)-C(3)	110.7(2)
C(1)-N(1)-C(4)	123.0(2)
C(3)-N(1)-C(4)	124.9(2)
C(1)-N(2)-C(2)	108.0(2)
C(1)-N(2)-C(8)	120.5(2)
C(2)-N(2)-C(8)	117.85(19)
O(1)-C(1)-N(1)	125.2(2)
O(1)-C(1)-N(2)	125.7(2)
N(1)-C(1)-N(2)	109.0(2)
N(2)-C(2)-C(3)	103.21(19)
N(1)-C(3)-C(12)	114.2(2)
N(1)-C(3)-C(2)	100.52(19)

C(12)-C(3)-C(2)	111.7(2)
N(1)-C(4)-C(6)	109.0(2)
N(1)-C(4)-C(7)	109.3(2)
C(6)-C(4)-C(7)	108.6(2)
N(1)-C(4)-C(5)	110.3(2)
C(6)-C(4)-C(5)	110.2(3)
C(7)-C(4)-C(5)	109.5(2)
N(2)-C(8)-C(9)	110.1(2)
N(2)-C(8)-C(11)	108.4(2)
C(9)-C(8)-C(11)	108.6(2)
N(2)-C(8)-C(10)	109.6(2)
C(9)-C(8)-C(10)	110.4(2)
C(11)-C(8)-C(10)	109.8(2)
C(13)-C(12)-C(3)	121.1(2)
C(12)-C(13)-C(14)	123.3(3)
O(2)-C(14)-O(3)	123.8(3)
O(2)-C(14)-C(13)	126.5(3)
O(3)-C(14)-C(13)	109.7(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ys159_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	15(1)	24(1)	21(1)	7(1)	-1(1)	3(1)
O(2)	37(1)	36(1)	42(1)	28(1)	12(1)	13(1)
O(3)	24(1)	24(1)	28(1)	13(1)	10(1)	2(1)
N(1)	17(1)	18(1)	18(1)	6(1)	3(1)	5(1)
N(2)	14(1)	20(1)	14(1)	7(1)	2(1)	4(1)
C(1)	18(1)	16(1)	16(1)	7(1)	1(1)	5(1)
C(2)	12(1)	22(1)	18(1)	8(1)	1(1)	4(1)
C(3)	16(1)	19(1)	21(1)	9(1)	4(1)	5(1)
C(4)	25(1)	21(1)	17(1)	4(1)	2(1)	9(1)
C(5)	52(2)	34(2)	17(1)	3(1)	-4(1)	23(2)
C(6)	29(2)	19(1)	32(2)	4(1)	0(1)	0(1)
C(7)	34(2)	27(1)	25(1)	4(1)	8(1)	11(1)
C(8)	16(1)	22(1)	15(1)	7(1)	3(1)	5(1)
C(9)	20(1)	29(1)	26(1)	7(1)	5(1)	11(1)
C(10)	25(1)	30(2)	22(1)	15(1)	4(1)	3(1)
C(11)	22(1)	23(1)	19(1)	4(1)	-1(1)	3(1)
C(12)	23(1)	20(1)	20(1)	9(1)	5(1)	8(1)
C(13)	24(1)	20(1)	19(1)	9(1)	6(1)	7(1)
C(14)	27(1)	18(1)	17(1)	5(1)	5(1)	4(1)
C(15)	33(2)	30(2)	28(2)	14(1)	11(1)	-2(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ys159_0m.

	x	y	z	U(eq)
H(2A)	-2273	1738	382	21
H(2B)	-1612	3193	116	21
H(3A)	-2175	4219	1927	21
H(5A)	4228	4818	3561	51
H(5B)	2166	4323	4107	51
H(5C)	3648	5919	4655	51
H(6A)	2399	7155	2484	44
H(6B)	4366	6555	2561	44
H(6C)	3788	7653	3659	44
H(7A)	-755	6408	3345	44
H(7B)	617	6887	4522	44
H(7C)	-883	5297	3973	44
H(9A)	4635	2360	-56	37
H(9B)	3916	1200	-1293	37
H(9C)	3187	796	-231	37
H(10A)	3144	4086	-587	37
H(10B)	761	3594	-1118	37
H(10C)	2452	2935	-1829	37
H(11A)	-463	41	-1061	34
H(11B)	241	468	-2117	34
H(11C)	-1448	1121	-1401	34
H(12A)	-986	2203	2751	24
H(13A)	-4825	2770	2552	24
H(15A)	-8963	754	4455	47
H(15B)	-6720	987	5072	47
H(15C)	-7639	-364	3949	47

STANDARD 1H OBSERVE

Pulse Sequence: szpul

Solvent: CDCl3

Ambient temperature

File: ypc-02-22p-H

INOVA-500 "epoxide"

Relax. delay 0.000 sec

Pulse 26.0 degrees

Acq. time 2.69 sec

Width 5985.0 Hz

2 repetitions

OBSERVE H1, 300.1592136 MHz

DATA PROCESSING

Gauss apodization 0.896 sec

FT size 32768

Total time 0 min, 10 sec

Tabel, Entry 1

```
Pulse Sequence: s2pu1
Solvent: CDC13
Ambient temperature
File: YWC-02-22P-C13
INDVA-500 "epoxide"
```

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22995.8 Hz
382 repetitions
OBSERVE G13, 75.475082 MHz
DECOUPLE H1, 500.160679 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 1

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: yvc-02-37ap-H
INOVA-500 "epoxide"
Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
# of repetitions 300.1592196 MHz
OBSERVED F1
DATA PROCESSING
Gauss apodization 0.896 sec
FT size 32768
Total time 0 min, 10 sec

Table1, Entry 2

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: ywc-02-37a
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.657 sec
Width 22935.8 Hz
208 repetitions
OBSERVE C13 300.1606794 MHz
DECOUPLE H1 300.1606794 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FI size 32768
Total time 45 min, 29 sec

Table1, Entry 2

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: Benzene

Ambient temperature

File: yvc-02-47-01benzene

INOVA-500 "epoxide"

Relax. delay 0.000 sec

Pulse 26.0 degrees

Acq. time 2.065 sec

Acq. date 05/24/02

2 repetitions

OBSERVE H1 300.1592466 MHz

DATA PROCESSING

Gauss apodization 0.896 sec

FT size 32768

Total time 0 min, 10 sec

Table1, Entry 3

13C OBSERVE

Pulse Sequence: szpu1

Solvent: CDCl3

Sample Temperature: 25.00 °C

File: WY-0247-01PC13

INOVA-500 "epoxide"

Relax. delay: 1.000 sec

Pulse: 45.3 degrees

Acq. time: 0.697 sec

Width: 22935.8 Hz

88 repetitions

OBSERVE C13, 75.4750889 MHz

DECOUPLE H1, 300.1606793 MHz

Power: 40 dB

continuously on

WALTZ16 modulated

DATA PROCESSING

Time Shifting: 2.0 Hz

FI size: 32768

Total time: 45 min, 29 sec

Table1, Entry 3

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: ywc-02-50-01PH1good
INOVA-500 "epoxide"
Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
2 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FI size 32768
Total time 0 min, 10 sec

Tabel, Entry 4

13C OBSERVE

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
File: ywc-02-50-01PC13good
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.897 sec
NUC1 125.0 MHz
96 repetitions
OBSERVE C13 75.4750855 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 45 min, 29 sec

Table1, Entry 4

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: ywc-02-d6Phligood
INOVA-500 *epoxide
Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.066 sec
Wdth 1385.242 Hz
2 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
FI size 32768
Total time 0 min, 10 sec

Table1, Entry 5

Pulse Sequence: szpu1
Solvent: CDC13
Ambient temperature
File: ywc-02-46-02PC13good
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
680 repetitions
OBSERVE C13, 705.4750813 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
F1 size 32768
Total time 45 min, 29 sec

Table 1, Entry 5

STANDARD 1H OBSERVE

Pulse Sequence: szpu1

Solvent: CDCl3

Sample Temperature

File: YWC-02-25P-H1

INOVIA-500 "epoxide"

Relax: delay 0.000 sec

Pulse: 28.0 degrees

Acq. time 2.666 sec

Width 5995.2 Hz

2 repetitions

OBSERVE H1, 300.1592196 MHz

DATA PROCESSING

Gauss apodization 0.896 sec

FI size 32768

Total time 0 min, 10 sec

Table1, Entry 6

13C OBSERVE

Pulse Sequence: zgpg30
Solvent: CDCl3
Ambient temperature
F1: YWC-02-25P-C13
INNOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Date_ 20130808
Time 12:11:03
56.46411000 Hz
OBSERVE C13 75.4750827 MHz
DECOUPLE H1 300.106799 MHz
Power 40 dB
continuously on
SALTZMAN modulated
DATA ACQUISITION
Line broadening 2.0 Hz
F1 size 32768
Total time 45 min, 29 sec

Tabel, Entry 6

STANDARD 1H OBSERVE

Pulse Sequence: szpu1

Solvent: CDCl3

Ambient Temperature

F1: 200-02-30-02PM

INOVA-300 600186

Relax. delay 0.000 sec

Pulse 20.0 degrees

Acq time 2.00 sec

Width 5995.2 Hz

2 repetitions

OBSERVE H1, 300.1592196 MHz

DATA PROCESSING

Gauss: apodization 0.896 sec

FT size 32768

Total time 0 min, 10 sec

Table1, Entry 7

13C OBSERVE

Pulse Sequence: szpul
Solvent: CDCl₃
Ambient temperature
File: ywc-02-50-02PC13good
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
48 repetitions
OBSERVE C13, 75.4750911 MHz
DECUPLE H1, 300.1606799 MHz
Power 40 dB
Continuously on
Waltz16 decoupled
DATA PROCESSING
Line broadening 2.0 Hz
FI size 32768
Total time 45 min, 29 sec

Table1, Entry 7

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: YWC-02-23repeat
INOVA-500 "epoxide"
Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5395.2 Hz
2 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
F2 size 32768
Total time 0 min, 10 sec

Table1, Entry 8

13C OBSERVE

Pulse Sequence: s2pu1
Solvent: CDCl3
Acquisition Temperature
File: YWC-0223repeatC
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
72 repetitions
OBSERVE C13, 75.4750841 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
F1 size 32768
Total time 45 min, 29 sec

Table1, Entry 8

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: VWC-02-02-02P
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
F1 size 32768
Total time 0 min, 16 sec

Table1, Entry 9

13C OBSERVE

Pulse Sequence: s2put
Solvent: CDCl3
Ambient temperature
File: ymc-02-02-02PC13
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
NUC1 13C
OBSERVE excitations 4750813 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 45 min, 29 sec

Table1, Entry 9

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: CDCl3

Sample Temperature

File: 140C020215P-H1

INOVA-500 "epoxide"

Relax. delay 0.000 sec

Pulse 28.000000000000000

Acq. time 2.668 sec

Width 5995.2 Hz

2 repetitions

OBSERVE H1, 300.1592196 MHz

DATA PROCESSING

Gauss apodization 0.896 sec

FT size 32768

Total time 0 min, 10 sec

Table1, Entry 10

¹³C OBSERVE

Pulse Sequence: zgpg30
Solvent: CDCl₃
Ambient temperature
File: ymc-02-05cisp-c13
INNOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Sweep rate 228.000 Hz
288 repetitions
OBSERVE C13, 75.4750827 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DPA 100.000000
line broadening 2.0 Hz
FT size 32768
Total time 45 min, 29 sec

Tablet, Entry 10

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl3
 Ambient temperature
 File: YWC-02-29P-H1
 INOVA-500 "epoxide"
 Relax. delay 0.000 sec
 Pulse 26.0 degrees
 Acq. time 2.668 sec
 Width 5995.2 Hz
 2 repetitions
 OBSERVE H1, 300.1592196 MHz
 DATA PROCESSING
 Gain: 43276
 Phase: 0.896 sec
 Total time 0 min, 10 sec

Table1, Entry 11

¹³C OBSERVE

Pulse Sequence: s2pul
 Solvent: CDCl₃
 Ambient temperature
 File: ymc-02-29C13repeat
 INOVA-500 "epoxide"

Relax. delay 1.000 sec
 Pulse 46.3 degrees
 Acq. time 0.697 sec
 Scan 1283.8 Hz
 1200.699105
 OBSERVE C13: 75.4750778 MHz
 DECOUPLE H1: 300.1606799 MHz
 Power 40 dB
 continuously on
 SALTZ-16 modulated
 DPA 10000000
 Line broadening 2.0 Hz
 FT size 32768
 Total time 45 min, 29 sec

Table1, Entry 11

STANDARD 1H OBSERVE

Pulse Sequence: szpul
Solvent: CDCl3
Ambient temperature
File: yw6-02-44-02-01
INOVA-500 "cpoct16"
Relax. delay 0.000 sec
Pulse 28.0 degrees
Acq. time 2.85 sec
Width 5985.2 Hz
2 repetitions
OBSERVE: H1, 300.1592196 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
Ft size 32768
Total time 0 min, 10 sec

Table1, Entry 12

13C OBSERVE

Pulse Sequence: szpul
Solvent: CDCl3
Ambient temperature
File: ymc-02-44-02P-C13
INOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.487 sec
Width 22835.8 Hz
64 repetitions
OBSERVE C13, 75.4750855 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
Continuously on
Waltz16
Data processing
Line broadening 2.0 Hz
FT size 32768
Total time 45 min, 29 sec

Tabel, Entry 12

```
Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
file: YWC-02-26P-H1
INOVA-500 "epoxide"
```

```
Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
2 repetitions
OBSERVE H1, 300.1592196 MHz
DATA PROCESSING
Gauss adp. lation 0.896 sec
FI size 32768
Total time 0 min, 10 sec
```

Pulse 26.0 degrees
 Acq. time 2.665 sec
 Nucleus 13C
 Observed 125.2 MHz
 Observed 1H 300.1592196 MHz
 DATA PROCESSING
 Gauss apodization 0.896 sec
 FT size 32768
 Total time 0 min, 10 sec

Tablet, Entry 13

CC(C)N(C(=O)N(C)C(C)=C)C(C)=C

7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm

1.04 0.88 1.00 0.98 2.76 2.91 8.54 8.85

13C OBSERVE

Pulse Sequence: szpul
Solvent: CDCl3
Ambient temperature
File: ywc-02-38p-C13
INOVA-500 "epoxide"
Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Acq. in 228.8 Hz
Sf 125.761 MHz
OBSERVE C13 on 75.4750827 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 45 min, 29 sec

Table1, Entry 13

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1
Solvent: CDCl3
Ambient temperature
File: ywc-02-46-01Phigood
INDVA-500 "epoxide"
Relax. delay 0.000 sec
Pulse 26.0 degrees
Acq. time 2.668 sec
Width 5995.2 Hz
2 repetitions
OBSERVE H1, 300.1592186 MHz
DATA PROCESSING
Gauss apodization 0.896 sec
F1 size 32768
Total time 0 min, 10 sec

Table1, Entry 14

13C OBSERVE

Pulse Sequence: szpu1

Solvent: CDCl3

Ambient temperature

File: ywc-02-46-01PC13good

INOVA-500 "epoxide"

Relax. delay 1.000 sec

Pulse 46.3 degrees

Acq. time 0.897 sec

Width 22935.847

168 repetitions

OBSERVE C13, 75.4750827 MHz

DECOUPLE H1, 300.1606799 MHz

Power 40 dB

continuously on

DATA PROCESSING

Line broadening 2.0 Hz

FT size 32768

Total time 45 min, 29 sec

Table1, Entry 14

STANDARD 1H OBSERVE

Pulse Sequence: s2pu1

Solvent: Benzene

Ambient temperature

File: ymc-02-44-1p-4h

INOVA-500 "epoxide"

Relax. delay 0.000 sec

Pulse 26.0 degrees

Acq. time 2.085 sec

Acq. date 1995/2/2

2 repetitions

OBSERVE H1 300.1592466 MHz

DATA PROCESSING

Gauss apodization 0.896 sec

FI size 32768

Total time 0 min, 10 sec

Tablet, Entry 15

¹³C OBSERVE

Pulse Sequence: szpul
Solvent: Benzene
Ambient temperature
File: ywc-02-44-1PC13
INOVA-500 "epoxide"

Relax: delay 1.000 sec
Pulse: 46.3 degrees
Acq: 120.628.897 sec
Waltz: 2235.8 Hz
136 repetitions
OBSERVE C13: 75.4750710 MHz
DECOUPLE H1: 300.1607069 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
F1 size 32768
Total time 45 min, 29 sec

Table1, Entry 15

STANDARD 1H OBSERVE

Pulse Sequence: szpul

Solvent: CDCl3
Ambient Temperature
F1: 200.632534 MHz
INOVA-500 "epoxide"

Relax. delay 0.000 sec

Pulse 16.000 sec

Acq time 2.068 sec

Width 5995.2 Hz

2 repetitions

OBSERVE H1, 300.1592196 MHz

DATA PROCESSING

Gauss apodization 0.896 sec

FT size 32768

Total time 0 min, 10 sec

Scheme 4

13C OBSERVE

Pulse Sequence: szpu1
Solvent: CDCl3
Ambient temperature
File: YWC-03-25-3C13
INDOVA-500 "epoxide"

Relax. delay 1.000 sec
Pulse 46.3 degrees
Acq. time 0.697 sec
Width 22935.8 Hz
256 repetitions
OBSERVE C13, 75.4750889 MHz
DECOUPLE H1, 300.1606799 MHz
Power 40 dB
Sensitivity on
Waltz16 simulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 32768
Total time 45 min, 29 sec

Scheme 4