Supporting Information

Tetrahydrothiophene-catalyzed Synthesis of Benzo[*n*.1.0] bicycloalkanes

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General Information. All reaction flasks were dried by flame. And all reactions were carried out under N₂ unless otherwise noted. All solvents were purified according to standard methods unless otherwise noted.

¹H NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer in chloroform-d₃. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data is being reported as (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, brs = broad singlet, coupling constant(s) in Hz, integration).

¹³C NMR spectra were recorded on a VARIAN Mercury 75 MHz spectrometer in chloroform-d₃. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard.

2a: a white solid, m.p. 53-54°C; ¹H NMR (300 MHz, CDCl₃, TMS): δ 7.25-7.34 (m, 2H), 7.13 (dt, *J* = 15.6 and 3.8 Hz, 1H), 6.95 (t, *J* = 8.4 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 6.30 (dt, *J* = 15.9 and 2.1 Hz, 1H), 4.79 (dd, *J* = 2.1 and 3.9 Hz, 2H), 4.59 (s, 2H), 3.77 (s, 3H); ¹³C NMR (75 MHz, CDCl₃, TMS): δ 166.5, 155.7, 142.3, 131.0, 130.2, 126.4, 121.5, 121.3, 111.8, 66.5, 51.7, 28.8. IR ν/cm⁻¹ 1723 (s), 1669 (m), 1599 (m), 1494 (m), 758 (m); MS (EI, *m/z*, rel. intensity) 284 (M⁺, 6.24), 145 (100). Anal. calcd for C₁₂H₁₃O₃Br: C, 50.55; H, 4.60. Found: C, 50.57; H, 4.63.

2b: ¹H NMR (300 MHz, CDCl₃, TMS): δ 7.25-7.37 (m, 2H), 7.11 (dt, *J* = 15.9 and 4.1 Hz, 1H), 6.95 (t, *J* = 7.5 Hz, 1H), 6.82 (d, *J* = 8.1 Hz, 1H), 6.27 (dt, *J* = 15.9 and 2.0 Hz, 1H), 4.78 (dd, *J* = 2.1 and 3.3 Hz, 2H), 4.60 (s, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 1.31 (t, *J* = 7.1 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃, TMS): δ 166.1, 155.8, 142.0, 131.0, 130.2, 126.4, 122.0, 121.3, 111.9, 66.6, 60.6, 28.8, 14.2. IR ν/cm⁻¹ 1719

(s), 1664 (m), 1601 (m), 1493 (m), 753 (m); MS (EI, m/z , rel. intensity) 298 (M^+ , 11.08), 78 (100), 145 (84.18). Anal. calcd for $C_{13}H_{15}O_3Br$: C, 52.19; H, 5.05. Found: C, 52.12; H, 5.26.

2b': 1H NMR (300 MHz, $CDCl_3$, TMS): δ 7.25-7.35 (m, 2H), 6.87-6.96 (m, 2H), 6.56 (dt, $J = 11.4$ and 4.7 Hz, 1H), 5.94 (dt, $J = 11.4$ and 2.6 Hz, 1H), 5.25 (dd, $J = 4.5$ and 2.7 Hz, 2H), 4.59 (s, 2H), 4.22 (q, $J = 7.2$ Hz, 2H), 1.32 (t, $J = 6.9$ Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 166.0, 156.0, 146.7, 131.0, 130.2, 126.2, 121.0, 120.2, 112.0, 66.5, 60.4, 29.0, 14.2. IR ν/cm^{-1} 1716 (s), 1651 (m), 1602 (m), 1589 (w), 1493 (s), 753 (m); MS (EI, m/z , rel. intensity) 298 (M^+ , 3.86), 113 (100), 85 (93.18). Anal. calcd for $C_{13}H_{15}O_3Br$: C, 52.19; H, 5.05. Found: C, 52.43; H, 5.17.

2c: 1H NMR (300 MHz, $CDCl_3$, TMS): δ 7.11-7.37 (m, 5H), 5.76 (dt, $J = 15.6$ and 2.0 Hz, 1H), 4.47 (s, 2H), 3.72 (s, 3H), 3.68 (dd, $J = 6.0$ and 1.5 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 166.7, 146.7, 136.6, 135.7, 130.7, 130.5, 129.4, 127.5, 122.3, 51.5, 35.0, 31.3. IR ν/cm^{-1} 1721 (s), 1655 (m), 1435 (m), 762 (m), 607 (m); MS (EI, m/z , rel. intensity) 268 (M^+ , 0.78), 129 (100). Anal. calcd for $C_{12}H_{13}BrO_2$: C, 53.55; H, 4.87. Found: C, 53.80; H, 4.90.

2d: 1H NMR (300 MHz, $CDCl_3$, TMS): δ 7.16-7.34 (m, 4H), 7.05 (dt, $J = 15.6$ and 6.9 Hz, 1H), 5.89 (dt, $J = 15.9$ and 1.4 Hz, 1H), 4.51 (s, 2H), 4.18 (q, $J = 7.1$ Hz, 2H), 2.85-2.91 (m, 2H), 2.53-2.61 (m, 2H), 1.28 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$, TMS): δ 166.3, 147.5, 139.6, 135.3, 130.6, 129.4, 129.0, 126.7, 121.9, 60.1, 32.9, 31.4, 30.4, 14.2. IR ν/cm^{-1} 2980 (m), 1717 (s), 1655 (m), 1492 (m), 761 (m), 607 (m); MS (EI, m/z , rel. intensity) 297 (M^+ , 0.07), 49 (100). Anal. calcd

for C₁₄H₁₇O₂Br: C, 56.58; H, 5.77. Found: C, 56.68; H, 5.69.

2e: ¹H NMR (300 MHz, CDCl₃, TMS): δ 7.17-7.35 (m, 4H), 6.94 (dt, *J* = 15.9 and 6.9 Hz, 1H), 5.83 (dt, *J* = 15.3 and 1.4 Hz, 1H), 4.52 (s, 2H), 2.85-2.90 (m, 2H), 2.51-2.59 (m, 2H), 1.49 (s, 9H); ¹³C NMR (75 MHz, CDCl₃, TMS): δ 165.8, 146.3, 139.8, 135.3, 130.6, 129.5, 129.1, 126.8, 123.7, 80.2, 32.9, 31.4, 30.6, 28.1. IR ν/cm⁻¹ 2977 (m), 1712 (s), 1653 (m), 1455 (m), 761 (m), 607 (m); MS (EI, *m/z*, rel. intensity) 267 (M⁺-^{*t*}Bu, 0.33), 251 (M⁺-O^{*t*}Bu, 0.79), 188 (M⁺-^{*t*}Bu-Br, 36.42), 57 (100). Anal. calcd for C₁₆H₂₁O₂Br: C, 59.09; H, 6.51. Found: C, 59.53; H, 6.11.

2f: ¹H NMR (300 MHz, CDCl₃, TMS): δ 9.53 (d, *J* = 7.8 Hz, 1H), 7.21-7.37 (m, 4H), 6.92 (dt, *J* = 15.3 and 6.9 Hz, 1H), 6.18 (ddt, *J* = 15.9, 7.8 and 1.5 Hz, 1H), 4.63 (s, 2H), 2.94-2.99 (m, 2H), 2.69-2.77 (m, 2H); ¹³C NMR (75 MHz, CDCl₃, TMS): δ 193.9, 156.9, 139.1, 135.4, 133.4, 130.8, 129.5, 129.2, 127.0, 33.3, 31.4, 30.2. IR ν/cm⁻¹ 2924 (m), 1687 (s), 1636 (w), 1455 (m), 762 (m), 604 (m); MS (EI, *m/z*, rel. intensity) 173 (M⁺-Br, 33.58), 104 (100). HRMS (EI) calcd for C₁₂H₁₃OBr (M⁺): 252.01498; Found: 252.01484.

2g: ¹H NMR (300 MHz, CDCl₃, TMS): δ 7.89-7.92 (m, 2H), 7.08-7.58 (m, 8H), 6.92 (dt, *J* = 15.6 and 1.2 Hz, 1H), 4.54 (s, 2H), 2.94-2.99 (m, 2H), 2.67-2.74 (m, 2H); ¹³C NMR (75 MHz, CDCl₃, TMS): δ 190.7, 147.9, 139.7, 137.7, 135.4, 132.7, 130.7, 129.7, 129.2, 128.5, 126.9, 126.6, 33.6, 31.5, 30.7. IR ν/cm⁻¹ 2926 (m), 1669 (s), 1620 (s), 1492 (m), 1447 (m), 763 (m), 693 (m), 606 (m); MS (EI, *m/z*, rel. intensity) 249 (M⁺-Br, 13.63), 105 (100). HRMS (MALDI) calcd for C₁₈H₁₈OBr⁺ (M⁺+H): 329.05490; Found: 329.05355.

2h: ^1H NMR (300 MHz, CDCl_3 , TMS): δ 7.20-7.36 (m, 4H), 6.86 (dt, $J = 16.2$ and 6.9 Hz, 1H), 6.13 (dt, $J = 15.9$ and 1.5 Hz, 1H), 4.63 (s, 2H), 2.89-2.94 (m, 2H), 2.56-2.64 (m, 2H), 2.25 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3 , TMS): δ 198.6, 146.7, 139.5, 135.3, 131.8, 130.7, 129.5, 129.2, 126.9, 33.2, 31.4, 30.5, 27.0. IR ν/cm^{-1} 1696 (m), 1673 (s), 1625 (m), 1492 (w), 1454 (w), 762 (m), 606 (m); MS (EI, m/z , rel. intensity) 187 ($\text{M}^+ - \text{Br}$, 23.86), 43 (100). HRMS (EI) calcd for $\text{C}_{13}\text{H}_{15}\text{O}^{+1}$ ($\text{M}^+ - \text{Br}$): 187.11229; Found: 187.11319.

1,1a,2,7b-Tetrahydrocyclopropa[c]chromene-1-carboxylic Acid, Ethyl Ester

(5b). Method A: 61% yield (oil, 16 h); ^1H NMR (300 MHz, CDCl_3 , TMS): δ 7.24 (dd, $J = 1.8$ Hz, 7.8 Hz, 1H), 7.10 (dt, $J = 1.8$, 7.8 Hz, 1H), 6.93 (dt, $J = 0.9$, 6.9 Hz, 1H), 6.80 (dd, $J = 0.9$, 7.8 Hz, 1H), 4.37 (ABd, $J = 11.1$ Hz, 1H), 4.17 (q, $J = 7.2$ Hz, 2H), 3.92 (ABd, $J = 10.8$ Hz, 1H), 2.56-2.60 (m, 1H), 2.30-2.34 (m, 2H), 1.27 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (75 MHz, CDCl_3 , TMS): δ 172.2, 152.7, 128.7, 127.3, 124.0, 121.8, 117.3, 61.6, 60.8, 26.9, 24.3, 22.7, 14.2. IR (ν/cm^{-1}) 2980 (m), 1721 (s), 1583 (m), 1491 (m), 1433 (w), 757 (m); MS (EI, m/z , rel. intensity) 218 (72.43, M^+), 189 (89.36), 131 (84.75), 115 (81.54). HRMS (EI) calcd for $\text{C}_{13}\text{H}_{15}\text{O}_3^{+1}$ ($\text{M}^+ + \text{H}$): 219.10220; Found: 219.10157.

1a,2,3,7b-Tetrahydro-1H-cyclopropa[a]naphthalene-1-carboxylic Acid, Ethyl

Ester 5d¹ . Method A: 63% yield (oil, 32 h); ¹H NMR (300 MHz, CDCl₃, TMS): δ 7.29-7.32 (m, 1H), 7.10-7.19 (m, 2H), 7.02-7.05 (m, 1H), 4.18 (q, *J* = 7.2 Hz, 2H), 2.41-2.70 (m, 3H), 2.17-2.25 (m, 2H), 2.09 (t, *J* = 3.9 Hz, 1H), 1.75-1.87 (m, 1H), 1.29 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 173.1, 135.0, 133.7, 128.7, 128.5, 126.2, 125.9, 60.5, 26.3, 25.3, 24.2, 23.0, 18.5, 14.2.

1a,2,3,7b-Tetrahydro-1H-cyclopropa[a]naphthalene-1-carboxylic Acid,

tert-Butyl Ester 5e. Method A: 77% yield, 86% conversion (oil, 72 h); ¹H NMR (300 MHz, CDCl₃, TMS): δ 7.28-7.32 (m, 1H), 7.08-7.17 (m, 2H), 7.00 (d, *J* = 6.9 Hz, 1H), 2.64 (dd, *J* = 5.4, 15.9 Hz, 1H), 2.39-2.51 (m, 2H), 2.07-2.23 (m, 2H), 1.98 (t, *J* = 3.6 Hz, 1H), 1.71-1.99 (m, 1H), 1.58 (s, 9H); ¹³C NMR (75 MHz, CDCl₃): δ 172.4, 135.4, 133.8, 128.8, 128.5, 126.1, 125.8, 80.4, 28.2, 26.0, 25.4, 24.0, 23.8, 18.5. IR (ν/cm⁻¹) 2929 (m), 1716 (s), 1493 (m), 1419 (w), 850 (m); MS (EI, *m/z*, rel. intensity) 244 (2.04, M⁺), 128 (100), 57 (63.49), 188 (51.34). HRMS (EI) calcd for C₁₆H₂₀O₂ (M⁺): 244.14633; Found: 244.14585.

1a,2,3,7b-Tetrahydro-1H-cyclopropa[a]naphthalene-1-carboxaldehyde 5f¹ .

Method B: 64% yield (oil, 72 h); ¹H NMR (300 MHz, CDCl₃, TMS): δ 9.38 (d, *J* =

4.2 Hz, 1H), 7.26-7.29 (m, 1H), 7.12-7.19 (m, 2H), 7.03-7.06 (m, 1H), 2.66-2.73 (m, 2H), 2.40-2.55 (m, 2H), 2.31-2.35 (m, 1H), 2.17-2.26 (m, 1H), 1.81-1.93 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3): δ 199.4, 134.0, 133.7, 128.6 (8), 128.6 (7), 128.7, 126.4, 126.3, 33.0, 27.3, 25.2, 24.8, 18.6.

Phenyl-(1a,2,3,7b-tetrahydro-1H-cyclopropa[a]naphthalen-1-yl)methanone 5g²

. **Method B:** 64% yield (solid, 65 h); ^1H NMR (300 MHz, CDCl_3 , TMS): δ 7.96-7.99 (m, 2H), 7.43-7.56 (m, 3H), 7.30-7.33 (m, 1H), 7.06-7.19 (m, 3H), 3.17 (t, J = 3.9 Hz, 1H), 2.60-2.82 (m, 3H), 2.47-2.51 (m, 1H), 2.21-2.29 (m, 1H), 1.84-1.97 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3): δ 198.3, 137.8, 135.4, 133.9, 132.7, 128.9, 128.7, 128.5, 128.0, 126.4, 126.1, 30.5, 28.2, 27.6, 25.6, 18.9.

1-(1a,2,3,7b-Tetrahydro-1H-cyclopropa[a]naphthalen-1-yl)ethanone 5h.

Method A: 66% yield, 80% conversion (oil, 72 h); ^1H NMR (300 MHz, CDCl_3 , TMS): δ 7.24-7.27 (m, 1H), 7.09-7.18 (m, 2H), 7.02-7.04 (m, 1H), 2.44-2.71 (m, 4H), 2.27 (s, 3H), 2.13-2.27 (m, 2H), 1.74-1.86 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3): δ 206.5, 135.1, 133.7, 128.7, 128.6, 126.2, 126.0, 31.7, 31.0, 29.6, 26.9, 25.3, 18.7. IR (ν/cm^{-1}) 2927 (m), 1693 (s), 1493 (m), 1408 (w), 752 (m); MS (EI, m/z , rel. intensity) 186 (4.61, M^+), 84 (100), 49 (99.44), 86 (74.53). Anal. calcd for $\text{C}_{13}\text{H}_{14}\text{O}$: C, 83.83; H, 7.58. Found: C, 83.87; H, 7.76.

5c'³: ¹H NMR (300 MHz, CDCl₃, TMS): δ 7.12-7.42 (m, 4H), 6.70 (brs, 1H), 3.72 (s, 3H), 3.54 (brs, 2H), 3.45 (brs, 2H); ¹³C NMR (75 MHz, CDCl₃, TMS): δ 171.5, 144.7, 143.4, 140.9, 130.1, 126.3, 124.4, 123.5, 120.6, 52.0, 41.3, 36.8. IR ν/cm⁻¹ 1739 (s), 2952 (m), 1461 (m), 1436 (m), 754 (m); MS (EI, *m/z*, rel. intensity) 188 (M⁺, 29), 129 (100), 128 (81). HRMS (EI) calcd for C₁₂H₁₂O₂ (M⁺): 188.08373; Found: 188.08506.

References:

1. Cordi, A. A.; Berque-Bestel, I.; Persigand, T.; Lacoste, J. M.; Newman-Tancredi, A.; Audinot, V.; Millan, M. J. *J. Med. Chem.* **2001**, *44*, 787.
2. Podder, R. K.; Sarkar, T. K.; Sarkar, R. K.; Ray, S. C. *Indian J. Chem. Sect. B*, **1988**, *27*, 217.
3. Ohkita, M.; Nishida, S.; Tsuji, T. *J. Am. Chem. Soc.* **1999**, *121*, 4589.

¹H NMR 2a

¹³C NMR **2a**

¹H NMR **2b**

¹³C NMR **2b**

^1H NMR **2b'**

^{13}C NMR **2b'**

¹H NMR 2c

^{13}C NMR 2c

¹H NMR 2d

^{13}C NMR **2d**

¹H NMR 2e

^{13}C NMR 2e

¹H NMR 2f

^{13}C NMR 2f

¹H NMR 2g

^{13}C NMR 2g

¹H NMR 2h

^{13}C NMR 2h

¹H NMR **5a**

¹³C NMR 5a

¹H NMR **5b**

¹³C NMR **5b**

¹H NMR 5c

^{13}C NMR **5c**

¹H NMR 5c'

¹³C NMR 5c'

¹H NMR 5d

¹³C NMR 5d

¹H NMR 5e

¹³C NMR 5e

¹H NMR 5f

^{13}C NMR 5f

^{13}C NMR 5g

¹H NMR 5h

¹³C NMR 5h

Figure 1. X-ray crystal structure of compound **5g**.

Crystal data for C₂₀H₂₂O₃ (5g**):**

Table 1. Crystal data and structure refinement for cd26337.

Identification code	cd26337
Empirical formula	C ₂₀ H ₂₂ O ₃
Formula weight	310.38
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)/n
Unit cell dimensions	a = 16.3784(17) Å alpha = 90 deg. b = 6.4995(6) Å beta = 111.877(2) deg. c = 16.8446(17) Å gamma = 90 deg.
Volume	1664.0(3) Å ³
Z, Calculated density	4, 1.239 Mg/m ³
Absorption coefficient	0.082 mm ⁻¹
F(000)	664
Crystal size	0.427 x 0.330 x 0.290 mm
Theta range for data collection	1.48 to 26.99 deg.
Limiting indices	-14<=h<=20, -8<=k<=8, -21<=l<=21

Reflections collected / unique	9374 / 3629 [R(int) = 0.0667]
Completeness to theta = 26.99	99.8 %
Absorption correction	Empirical
Max. and min. transmission	1.00000 and 0.62005
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3629 / 0 / 218
Goodness-of-fit on F ²	0.915
Final R indices [I>2sigma(I)]	R1 = 0.0520, wR2 = 0.1258
R indices (all data)	R1 = 0.0801, wR2 = 0.1374
Extinction coefficient	0.0068(17)
Largest diff. peak and hole	0.248 and -0.167 e.Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd26337.
 $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
O(1)	-296(1)	2361(2)	702(1)	66(1)
O(2)	-568(1)	2956(3)	3729(1)	97(1)
O(3)	869(1)	2303(2)	4208(1)	65(1)
C(1)	360(1)	1374(3)	1090(1)	45(1)
C(2)	1205(1)	1789(3)	982(1)	45(1)
C(3)	1272(1)	3544(3)	532(1)	55(1)
C(4)	2037(2)	4008(3)	426(1)	69(1)
C(5)	2750(2)	2704(4)	748(1)	74(1)
C(6)	2690(1)	960(3)	1184(1)	65(1)
C(7)	1924(1)	473(3)	1315(1)	49(1)
C(8)	1873(1)	-1401(3)	1811(1)	57(1)
C(9)	941(1)	-2073(3)	1642(1)	53(1)
C(10)	354(1)	-316(2)	1721(1)	42(1)
C(11)	655(1)	693(2)	2596(1)	44(1)
C(12)	-5(1)	1125(3)	2836(1)	47(1)
C(13)	-877(1)	398(3)	2201(1)	52(1)
C(14)	-595(1)	-947(3)	1590(1)	50(1)
C(15)	-720(1)	-3141(3)	1856(1)	69(1)
C(16)	-1535(2)	-2989(4)	2061(2)	89(1)
C(17)	-1403(1)	-1005(4)	2566(1)	74(1)
C(18)	50(1)	2216(3)	3622(1)	59(1)
C(19)	975(1)	3374(4)	4995(1)	78(1)
C(20)	1890(1)	3140(4)	5590(1)	83(1)

Table 3. Bond lengths [Å] and angles [deg] for cd26337.

O(1)–C(1)	1.2126(18)
O(2)–C(18)	1.194(2)
O(3)–C(18)	1.337(2)
O(3)–C(19)	1.449(2)
C(1)–C(2)	1.486(2)
C(1)–C(10)	1.532(2)
C(2)–C(7)	1.393(2)
C(2)–C(3)	1.396(2)
C(3)–C(4)	1.364(3)
C(3)–H(3)	0.9300
C(4)–C(5)	1.379(3)
C(4)–H(4)	0.9300
C(5)–C(6)	1.373(3)
C(5)–H(5)	0.9300
C(6)–C(7)	1.389(2)
C(6)–H(6)	0.9300
C(7)–C(8)	1.496(2)
C(8)–C(9)	1.510(2)
C(8)–H(8A)	0.9700
C(8)–H(8B)	0.9700
C(9)–C(10)	1.531(2)
C(9)–H(9A)	0.9700
C(9)–H(9B)	0.9700
C(10)–C(11)	1.517(2)
C(10)–C(14)	1.542(2)
C(11)–C(12)	1.319(2)
C(11)–H(11)	0.9300
C(12)–C(18)	1.475(2)
C(12)–C(13)	1.506(2)
C(13)–C(17)	1.532(3)
C(13)–C(14)	1.545(2)
C(13)–H(13)	0.970(17)
C(14)–C(15)	1.531(3)
C(14)–H(14)	0.936(16)
C(15)–C(16)	1.500(3)
C(15)–H(15A)	0.9700
C(15)–H(15B)	0.9700
C(16)–C(17)	1.515(3)
C(16)–H(16A)	0.9700
C(16)–H(16B)	0.9700

C(17)–H(17A)	0.9700
C(17)–H(17B)	0.9700
C(19)–C(20)	1.468(3)
C(19)–H(19A)	0.9700
C(19)–H(19B)	0.9700
C(20)–H(20A)	0.9600
C(20)–H(20B)	0.9600
C(20)–H(20C)	0.9600

C(18)–O(3)–C(19)	116.18(14)
O(1)–C(1)–C(2)	121.10(15)
O(1)–C(1)–C(10)	121.31(15)
C(2)–C(1)–C(10)	117.57(14)
C(7)–C(2)–C(3)	119.70(16)
C(7)–C(2)–C(1)	121.65(16)
C(3)–C(2)–C(1)	118.65(15)
C(4)–C(3)–C(2)	120.89(19)
C(4)–C(3)–H(3)	119.6
C(2)–C(3)–H(3)	119.6
C(3)–C(4)–C(5)	119.7(2)
C(3)–C(4)–H(4)	120.1
C(5)–C(4)–H(4)	120.1
C(6)–C(5)–C(4)	119.97(19)
C(6)–C(5)–H(5)	120.0
C(4)–C(5)–H(5)	120.0
C(5)–C(6)–C(7)	121.41(19)
C(5)–C(6)–H(6)	119.3
C(7)–C(6)–H(6)	119.3
C(6)–C(7)–C(2)	118.26(17)
C(6)–C(7)–C(8)	121.02(17)
C(2)–C(7)–C(8)	120.71(16)
C(7)–C(8)–C(9)	113.10(14)
C(7)–C(8)–H(8A)	109.0
C(9)–C(8)–H(8A)	109.0
C(7)–C(8)–H(8B)	109.0
C(9)–C(8)–H(8B)	109.0
H(8A)–C(8)–H(8B)	107.8
C(8)–C(9)–C(10)	113.06(14)
C(8)–C(9)–H(9A)	109.0
C(10)–C(9)–H(9A)	109.0
C(8)–C(9)–H(9B)	109.0
C(10)–C(9)–H(9B)	109.0
H(9A)–C(9)–H(9B)	107.8
C(11)–C(10)–C(9)	114.83(13)

C(11)–C(10)–C(1)	106.28(12)
C(9)–C(10)–C(1)	107.87(13)
C(11)–C(10)–C(14)	101.55(13)
C(9)–C(10)–C(14)	114.93(14)
C(1)–C(10)–C(14)	111.02(13)
C(12)–C(11)–C(10)	112.49(14)
C(12)–C(11)–H(11)	123.8
C(10)–C(11)–H(11)	123.8
C(11)–C(12)–C(18)	126.48(16)
C(11)–C(12)–C(13)	112.54(14)
C(18)–C(12)–C(13)	120.97(15)
C(12)–C(13)–C(17)	115.26(15)
C(12)–C(13)–C(14)	102.22(14)
C(17)–C(13)–C(14)	106.42(15)
C(12)–C(13)–H(13)	108.2(10)
C(17)–C(13)–H(13)	111.6(10)
C(14)–C(13)–H(13)	113.0(10)
C(15)–C(14)–C(10)	116.34(15)
C(15)–C(14)–C(13)	103.09(15)
C(10)–C(14)–C(13)	106.81(13)
C(15)–C(14)–H(14)	108.6(9)
C(10)–C(14)–H(14)	110.7(9)
C(13)–C(14)–H(14)	111.0(9)
C(16)–C(15)–C(14)	103.51(17)
C(16)–C(15)–H(15A)	111.1
C(14)–C(15)–H(15A)	111.1
C(16)–C(15)–H(15B)	111.1
C(14)–C(15)–H(15B)	111.1
H(15A)–C(15)–H(15B)	109.0
C(15)–C(16)–C(17)	103.39(17)
C(15)–C(16)–H(16A)	111.1
C(17)–C(16)–H(16A)	111.1
C(15)–C(16)–H(16B)	111.1
C(17)–C(16)–H(16B)	111.1
H(16A)–C(16)–H(16B)	109.0
C(16)–C(17)–C(13)	105.38(17)
C(16)–C(17)–H(17A)	110.7
C(13)–C(17)–H(17A)	110.7
C(16)–C(17)–H(17B)	110.7
C(13)–C(17)–H(17B)	110.7
H(17A)–C(17)–H(17B)	108.8
O(2)–C(18)–O(3)	123.04(16)
O(2)–C(18)–C(12)	123.85(17)
O(3)–C(18)–C(12)	113.11(15)

O(3)-C(19)-C(20)	108.59(16)
O(3)-C(19)-H(19A)	110.0
C(20)-C(19)-H(19A)	110.0
O(3)-C(19)-H(19B)	110.0
C(20)-C(19)-H(19B)	110.0
H(19A)-C(19)-H(19B)	108.4
C(19)-C(20)-H(20A)	109.5
C(19)-C(20)-H(20B)	109.5
H(20A)-C(20)-H(20B)	109.5
C(19)-C(20)-H(20C)	109.5
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd26337.
The anisotropic displacement factor exponent takes the form:
 $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
O(1)	62(1)	67(1)	68(1)	24(1)	25(1)	19(1)
O(2)	63(1)	155(2)	70(1)	-37(1)	21(1)	28(1)
O(3)	53(1)	97(1)	42(1)	-23(1)	16(1)	3(1)
C(1)	54(1)	44(1)	34(1)	-3(1)	15(1)	4(1)
C(2)	57(1)	47(1)	32(1)	-9(1)	18(1)	-2(1)
C(3)	73(1)	50(1)	45(1)	-6(1)	25(1)	-6(1)
C(4)	91(2)	63(1)	64(1)	-12(1)	42(1)	-21(1)
C(5)	73(1)	94(2)	66(1)	-24(1)	39(1)	-23(1)
C(6)	61(1)	83(2)	54(1)	-12(1)	26(1)	2(1)
C(7)	55(1)	56(1)	36(1)	-12(1)	18(1)	2(1)
C(8)	61(1)	59(1)	51(1)	0(1)	21(1)	16(1)
C(9)	67(1)	46(1)	45(1)	1(1)	21(1)	7(1)
C(10)	47(1)	44(1)	34(1)	-2(1)	13(1)	1(1)
C(11)	45(1)	50(1)	34(1)	-1(1)	11(1)	2(1)
C(12)	47(1)	55(1)	36(1)	-2(1)	14(1)	3(1)
C(13)	46(1)	63(1)	43(1)	2(1)	14(1)	5(1)
C(14)	53(1)	56(1)	35(1)	0(1)	10(1)	-4(1)
C(15)	77(1)	64(1)	70(1)	-1(1)	32(1)	-15(1)
C(16)	81(2)	95(2)	96(2)	6(1)	39(1)	-24(1)
C(17)	59(1)	103(2)	66(1)	-1(1)	30(1)	-8(1)
C(18)	53(1)	78(1)	45(1)	-8(1)	18(1)	10(1)
C(19)	75(1)	111(2)	48(1)	-29(1)	25(1)	1(1)
C(20)	77(2)	127(2)	43(1)	-18(1)	19(1)	-7(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for cd26337.

	x	y	z	U(eq)
H(3)	788	4409	302	66
H(4)	2078	5198	137	82
H(5)	3270	3006	671	88
H(6)	3173	86	1395	78
H(8A)	2168	-1121	2417	68
H(8B)	2184	-2519	1665	68
H(9A)	950	-3152	2043	64
H(9B)	689	-2647	1070	64
H(11)	1241	976	2926	53
H(15A)	-802	-4104	1393	83
H(15B)	-220	-3573	2353	83
H(16A)	-1584	-4154	2400	106
H(16B)	-2058	-2921	1543	106
H(17A)	-1079	-1257	3170	88
H(17B)	-1965	-386	2494	88
H(19A)	579	2801	5246	93
H(19B)	836	4820	4879	93
H(20A)	2011	1713	5733	125
H(20B)	1980	3915	6100	125
H(20C)	2279	3639	5325	125
H(13)	-1216(10)	1600(30)	1921(10)	50(5)
H(14)	-971(10)	-750(20)	1021(10)	47(4)

Table 6. Torsion angles [deg] for cd26337.

O(1)-C(1)-C(2)-C(7)	-169.77(16)
C(10)-C(1)-C(2)-C(7)	11.4(2)
O(1)-C(1)-C(2)-C(3)	10.0(2)
C(10)-C(1)-C(2)-C(3)	-168.78(14)
C(7)-C(2)-C(3)-C(4)	-1.1(2)
C(1)-C(2)-C(3)-C(4)	179.13(15)
C(2)-C(3)-C(4)-C(5)	1.5(3)
C(3)-C(4)-C(5)-C(6)	-0.8(3)
C(4)-C(5)-C(6)-C(7)	-0.5(3)
C(5)-C(6)-C(7)-C(2)	0.9(3)
C(5)-C(6)-C(7)-C(8)	-178.33(17)
C(3)-C(2)-C(7)-C(6)	-0.2(2)
C(1)-C(2)-C(7)-C(6)	179.65(14)
C(3)-C(2)-C(7)-C(8)	179.08(15)
C(1)-C(2)-C(7)-C(8)	-1.1(2)
C(6)-C(7)-C(8)-C(9)	-160.58(15)
C(2)-C(7)-C(8)-C(9)	20.2(2)
C(7)-C(8)-C(9)-C(10)	-49.93(19)
C(8)-C(9)-C(10)-C(11)	-60.31(19)
C(8)-C(9)-C(10)-C(1)	57.97(17)
C(8)-C(9)-C(10)-C(14)	-177.60(13)
O(1)-C(1)-C(10)-C(11)	-93.59(18)
C(2)-C(1)-C(10)-C(11)	85.23(16)
O(1)-C(1)-C(10)-C(9)	142.78(16)
C(2)-C(1)-C(10)-C(9)	-38.41(18)
O(1)-C(1)-C(10)-C(14)	16.0(2)
C(2)-C(1)-C(10)-C(14)	-165.16(13)
C(9)-C(10)-C(11)-C(12)	-139.63(16)
C(1)-C(10)-C(11)-C(12)	101.20(16)
C(14)-C(10)-C(11)-C(12)	-14.97(18)
C(10)-C(11)-C(12)-C(18)	-176.03(16)
C(10)-C(11)-C(12)-C(13)	3.1(2)
C(11)-C(12)-C(13)-C(17)	125.21(18)
C(18)-C(12)-C(13)-C(17)	-55.6(2)
C(11)-C(12)-C(13)-C(14)	10.2(2)
C(18)-C(12)-C(13)-C(14)	-170.57(16)
C(11)-C(10)-C(14)-C(15)	-93.94(16)
C(9)-C(10)-C(14)-C(15)	30.6(2)
C(1)-C(10)-C(14)-C(15)	153.41(15)
C(11)-C(10)-C(14)-C(13)	20.49(16)

C(9)-C(10)-C(14)-C(13)	145.08(14)
C(1)-C(10)-C(14)-C(13)	-92.16(16)
C(12)-C(13)-C(14)-C(15)	104.04(16)
C(17)-C(13)-C(14)-C(15)	-17.23(18)
C(12)-C(13)-C(14)-C(10)	-19.06(17)
C(17)-C(13)-C(14)-C(10)	-140.33(14)
C(10)-C(14)-C(15)-C(16)	153.70(16)
C(13)-C(14)-C(15)-C(16)	37.18(19)
C(14)-C(15)-C(16)-C(17)	-43.2(2)
C(15)-C(16)-C(17)-C(13)	32.0(2)
C(12)-C(13)-C(17)-C(16)	-121.24(18)
C(14)-C(13)-C(17)-C(16)	-8.7(2)
C(19)-O(3)-C(18)-O(2)	-0.4(3)
C(19)-O(3)-C(18)-C(12)	179.39(17)
C(11)-C(12)-C(18)-O(2)	163.4(2)
C(13)-C(12)-C(18)-O(2)	-15.7(3)
C(11)-C(12)-C(18)-O(3)	-16.5(3)
C(13)-C(12)-C(18)-O(3)	164.48(16)
C(18)-O(3)-C(19)-C(20)	174.47(17)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for cd26337 [Å and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
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