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Supplementary Information

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"Borane-Mediated Aldol Cycloreduction of Mono-Enone Mone-Ketones: Diastereoselective Formation of Quaternary Centers"

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1a

¹H NMR (400 MHz, CDCl₃): δ 1.82 (qt, *J* = 7.2 Hz, 2H), 2.15 (s, 3H), 2.33 (m, 2H), 2.50 (t, *J* = 7.2 Hz, 2H), 6.89 (m, 1H), 7.01 (m, 1H), 7.46 (m, 2H), 7.55 (m, 1H), 7.92 (m, 2H). ¹³C NMR (100 MHz, CDCl₃): δ 22.4, 30.5, 32.3, 43.0, 126.6, 128.7, 128.7, 132.9, 138.0, 148.7, 190.7, 208.2. FTIR (NaCl): 2935, 2253, 1713, 1670, 1620, 1598, 1578, 1448, 1357, 1288, 1227, 1159, 907, 740, 650 cm⁻¹. HRMS: calculated [M+1] for C₁₄H₁₆O₂ 217.1229, found 217.1229. M.P.: 61 – 62 °C.

2a

¹H NMR (400 MHz, CDCl₃): δ 1.85 (qt, *J* = 7.2 Hz, 2H), 2.16 (s, 3H), 2.38 (m, 2H), 2.52 (t, *J* = 7.2 Hz, 2H), 7.07 (m, 2H), 7.56 (m, 2H), 7.95 (m, 4H), 8.43 (m, 1H). ¹³C NMR (100 MHz, CDCl₃): 22.5, 30.5, 32.4, 43.1, 124.7, 126.5, 127.0, 128.0, 128.5, 128.7, 129.7, 130.2, 132.7, 135.3, 135.6, 148.6, 190.4, 208.2. FTIR (NaCl): 2942, 2253, 1713, 1666, 1630, 1617, 1468, 1356, 1294, 1220, 1188, 1126, 1095, 912, 741, 651 cm⁻¹. HRMS: calculated [M+1] for C₁₈H₁₈O₂, 267.1385, found 267.1392. M.P.: 69 - 70 °C.

3a

¹H NMR (400 MHz, CDCl₃): δ 1.79 (qt, *J* = 9.6 Hz, 2H), 2.11 (s, 3H), 2.31 (m, 2H), 2.45 (t, *J* = 9.6 Hz, 2H), 6.77 (m, 1H), 7.06 (m, 2H), 7.67 (m, 2H). ¹³C NMR (100 MHz, CDCl₃): 22.2, 30.3, 32.0, 42.8, 126.1, 128.5, 132.2, 134.1, 145.3, 148.0, 182.3, 208.4. FTIR (NaCl): 3054, 2987, 2358, 1714, 1657, 1610, 1515, 1415, 1354, 1265, 1083, 913, 744 cm⁻¹. HRMS: calculated for [M+1] for C₁₂H₁₄O₂S 223.0793, found 223.0795. M.P.: 48 - 50 °C.

4a

¹H NMR (400 MHz, CDCl₃): δ 1.82 (qt, *J* = 7.2 Hz, 2H), 2.15 (s, 3H), 2.32 (m, 2H), 2.50 (t, *J* = 7.2 Hz, 2H), 6.55 (m, 1H), 6.81 (m, 1H), 7.10 (m, 1H), 7.24 (m, 1H), 7.61 (m, 1H). **¹³C NMR** (100 MHz, CDCl₃): 22.3, 30.5, 32.2, 43.0, 112.6, 117.9, 125.6, 146.7, 147.9, 153.4, 178.0, 208.2. **FTIR** (NaCl): 2900, 253, 1794, 1713, 1666, 1620, 1568, 1467, 1395, 1298, 1165, 1090, 1014, 912, 742, 650 cm⁻¹. **HRMS**: calculated for [M+1]⁺ for C₁₂H₁₄O₃, 207.1021, found 207.1015. **M.P.**: 58 - 60 °C.

5a

¹H NMR (400 MHz, CDCl₃): δ 1.79 (qt, *J* = 9.6 Hz, 2H), 2.13 (s, 3H), 2.29 (m, 2H), 2.48 (t, *J* = 9.6 Hz, 2H), 3.98 (s, 3H), 6.15 (m, 1H), 6.74–6.99 (m, 4H). ¹³C NMR (100 MHz, CDCl₃): 22.7, 30.4, 32.0, 38.2, 43.1, 108.4, 119.5, 127.6, 131.6, 131.7, 144.7, 180.0, 208.4. FTIR (NaCl): 2949, 2253, 1712, 1656, 1605, 1527, 1460, 1407, 1308, 1237, 1095, 1068, 984, 912, 742, 650 cm⁻¹. HRMS: calculated for [M+1]⁺ for C₁₃H₁₇N₂O₂, found 220.1338.

S11

6a

¹H NMR (400 MHz, CDCl₃): δ 2.15 (s, 3H), 2.63 (m, 4H), 6.93 (m, 2H), 7.48 (m, 3H), 7.88 (m, 2H). ¹³C NMR (100 MHz, CDCl₃): 26.5, 30.0, 41.5, 126.5, 128.4, 132.6, 137.6, 147.4, 190.5, 206.7. FTIR (NaCl): 3055, 2987, 2685, 2411, 2306, 1716, 1671, 1651, 1622, 1599, 1579, 1447, 1422, 1366, 1265, 1180, 979, 896, 737, 704 cm⁻¹. HRMS: calculated for [M+]⁺ for C₁₃H₁₄O₂ 203.1072, found 203.1064.

S13

1b

^1H NMR (400 MHz, CDCl_3): δ 1.20 (s, 3H), 1.33 (m, 2H), 1.56 (m, 1H), 1.79 (m, 5H), 3.31 (dd, $J = 3.6, 12.0$ Hz, 1H), 4.45 (br, 1H), 7.49 (m, 2H), 7.60 (m, 1H), 7.95 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3): 21.5, 25.8, 27.3, 29.9, 39.2, 51.7, 70.4, 128.5, 129.1, 133.9, 138.2, 207.4. FTIR (NaCl): 3481, 3081, 2991, 2954, 2912, 2815, 1671, 1598, 1575, 1485, 1425, 1416, 1363, 1251, 1203, 987 cm^{-1} . HRMS: calculated for $[\text{M}+1]$ for $\text{C}_{14}\text{H}_{18}\text{O}_2$ 219.1385, found 219.1390. M.P.: 59-60 $^\circ\text{C}$.

2b

¹H NMR (300 MHz, CDCl₃): δ 1.25 (s, 3H), 1.38 (m, 2H), 1.60 (m, 1H), 1.90 (m, 5H), 3.31 (dd, *J* = 3.0, 12.2 Hz, 1H), 4.60 (br, 1H), 7.61 (m, 2H), 7.94 (m, 4H), 8.48 (s, 1H). ¹³C NMR (75 MHz, CDCl₃): 21.6, 25.8, 27.5, 30.1, 39.3, 51.7, 70.5, 124.0, 127.2, 128.0, 129.0, 129.1, 130.0, 130.4, 132.7, 134.2, 136.1, 207.3. FTIR(NaCl): 3961, 3699, 3613, 3421, 3023, 2996, 2925, 2888, 2802, 2654, 2301, 1651, 1642, 1461, 1256, 906 cm⁻¹. HRMS: calculated for [M+1] for C₁₈H₂₀O₂ 269.1541, found 269.1539. M.P.: 66 - 68 °C.

3b

¹H NMR (400 MHz, CDCl₃): δ 1.21 (s, 3H), 1.31 (m, 2H), 1.54 (m, 1H), 1.82 (m, 5H), 3.11 (dd, *J* = 3.6, 12.4 Hz, 1H), 4.27 (br, 1H), 7.16 (m, 1H), 7.69 (m, 1H), 7.76 (m, 1H). ¹³C NMR (100 MHz, CDCl₃): 21.6, 25.9, 27.7, 30.2, 39.2, 53.9, 70.4, 128.6, 132.8, 135.2, 144.2, 199.5. FTIR (NaCl): 2936, 2862, 2253, 1793, 1636, 1563, 1518, 1460, 1443, 1414, 1386, 1288, 1263, 1235, 1212, 908, 726, 650 cm⁻¹. HRMS: calculated for [M+1] for C₁₂H₁₆O₂S 225.0949, found 225.0955.

4b

¹H NMR (400 MHz, CDCl₃): δ 1.20 (s, 3H), 1.31 (m, 2H), 1.53 (m, 1H), 1.82 (m, 5H), 3.12 (dd, *J* = 3.6, 12.2 Hz, 1H), 4.16 (br, 1H), 6.58 (m, 1H), 7.27 (m, 1H), 7.64 (m, 1H). **¹³C NMR** (100 MHz, CDCl₃): 21.5, 25.8, 27.0, 30.0, 39.2, 52.5, 70.4, 112.8, 118.7, 147.4, 152.4, 195.1. **FTIR**(NaCl): 3054, 2986, 2938, 2859, 2685, 2305, 1652, 1567, 1464, 1443, 1422, 1396, 1265, 1166, 956, 909, 741, 705 cm⁻¹. **HRMS**: calculated for [M+1] for C₁₂H₁₆O₃ 209.1178, found 209.1175.

5b

¹H NMR (400 MHz, CDCl₃): δ 1.18 (s, 3H), 1.28 (m, 2H), 1.51 (m, 1H), 1.78 (m, 5H), 2.98 (dd, *J* = 3.6, 12.2 Hz, 1H), 3.93 (s, 3H), 4.73 (br, 1H), 6.14 (m, 1H), 6.86 (m, 1H), 6.99 (m, 1H). ¹³C NMR (100 MHz, CDCl₃): 21.7, 26.0, 28.0, 30.3, 38.4, 39.3, 52.7, 70.6, 108.6, 120.6, 130.7, 132.6, 197.1. FTIR (NaCl): 3442, 3054, 2986, 2937, 2305, 1623, 1422, 1408, 1392, 1265, 1096, 1068, 945, 901, 896, 738, 705 cm⁻¹. HRMS: calculated for [M+1] for C₁₃H₁₉NO₂, 222.1494, found 222.1486.

6b

¹H NMR (400 MHz, CDCl₃): δ 1.38 (s, 3H), 1.93 (m, 6H), 3.47 (dd, *J* = 9.0, 10.6 Hz, 1H), 4.51 (br, 1H), 7.48 (m, 2H), 7.58 (m, 1H), 7.94 (m, 2H). ¹³C NMR (100 MHz, CDCl₃): 22.5, 27.0, 30.7, 41.1, 54.3, 81.2, 128.5, 128.9, 133.8, 137.4, 205.6. FTIR(NaCl): 3470, 3156, 2972, 2876, 2253, 1662, 1598, 1461, 1449, 1377, 1229, 912, 734, 650 cm⁻¹. HRMS: calculated for [M+1]⁺ for C₁₃H₁₆O₂, 205.1229, found 205.1225.

Crystallographic Material for **1b**.

X-ray Experimental.

Table 1. Crystallographic Data for **1b**.

Table 2. Fractional coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2$) for the non-hydrogen atoms of **1b**.

Table 3. Bond Lengths ($\text{\AA}$) and Angles ($^\circ$) for the non-hydrogen atoms of **1b**.

Table 4. Anisotropic thermal parameters for the non-hydrogen atoms of **1b**.

Table 5. Fractional coordinates and isotropic thermal parameters ($\text{\AA}^2$) for the hydrogen atoms of **1b**.

Table 6. Torsion Angles ($^\circ$) for the non-hydrogen atoms of **1b**.

Figure 1. View of **1b** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. Hydrogen atoms are drawn to an arbitrary size. The dashed line is indicative of a H-bonding interaction with geometry: O7-H7 $\cdots$ O10, O $\cdots$ O 2.752(1) $\text{\AA}$, H $\cdots$ O 2.02(2) $\text{\AA}$, O-H $\cdots$ O 137(2) $^\circ$.

Figure 2. Unit cell packing diagram for **1b**. The view is approximately down the **a** axis.

X-ray Experimental for $C_{14}H_{18}O_2$: Crystals grew as large colorless lathes and prisms by slow cooling from 90% hexane and 10% ethyl acetate. The data crystal was cut into an irregular shape from a larger crystal and had approximate dimensions; 0.22x0.17x0.15 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with $MoK\alpha$ radiation ($\lambda = 0.71073\text{\AA}$). A total of 286 frames of data were collected using ω -scans with a scan range of 1° and a counting time of 148 seconds per frame. The data were collected at -120°C using a Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 1. Data reduction were performed using DENZO-SMN.¹ The structure was solved by direct methods using SIR92² and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.³ The hydrogen atom positions were observed in a ΔF map and refined with isotropic displacement parameters. The function, $\sum w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.0377*P)^2 + (0.3829*P)]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.0964, with $R(F)$ equal to 0.0395 and a goodness of fit, S , = 0.993. Definitions used for calculating $R(F)$, $R_w(F^2)$ and the goodness of fit, S , are given below.⁴ The data were corrected for secondary extinction effects. The correction takes the form: $F_{corr} = kF_c/[1 + (2.0(6)\times 10^{-5}) * F_c^2 \lambda^3/(\sin 2\theta)]^{0.25}$ where k is the overall scale factor. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).⁵ All figures were generated using SHELXTL/PC.⁶ Tables of positional and thermal parameters, bond lengths and angles, torsion angles, figures and lists of observed and calculated structure factors are located in tables 1 through 7.

References

- 1) DENZO-SMN. (1997). Z. Otwinowski and W. Minor, *Methods in Enzymology*, **276**: Macromolecular Crystallography, part A, 307 – 326, C. W. Carter, Jr. and R. M. Sweets, Editors, Academic Press.
- 2) SIR92. (1993). A program for crystal structure solution. Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. *J. Appl. Cryst.* **26**, 343-350.
- 3) Sheldrick, G. M. (1994). SHELXL97. Program for the Refinement of Crystal Structures. University of Gottingen, Germany.
- 4) $R_w(F^2) = \{\Sigma w(|F_o|^2 - |F_c|^2)^2 / \Sigma w(|F_o|^4)\}^{1/2}$ where w is the weight given each reflection.
 $R(F) = \Sigma(|F_o| - |F_c|) / \Sigma |F_o|$ for reflections with $F_o > 4(\sigma(F_o))$.
 $S = [\Sigma w(|F_o|^2 - |F_c|^2)^2 / (n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.
- 5) International Tables for X-ray Crystallography (1992). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.
- 6) Sheldrick, G. M. (1994). SHELXTL/PC (Version 5.03). Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.

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

Empirical formula	C ₁₄ H ₁₈ O ₂	
Formula weight	218.28	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2 ₁ /c	
Unit cell dimensions	a = 10.4562(2) Å	α = 90°.
	b = 10.8950(2) Å	β = 102.338(2)°.
	c = 10.7241(2) Å	γ = 90°.
Volume	1193.48(4) Å ³	
Z	4	
Density (calculated)	1.215 Mg/m ³	
Absorption coefficient	0.080 mm ⁻¹	
F(000)	472	
Crystal size	0.22 x 0.17 x 0.15 mm	
Theta range for data collection	3.10 to 27.49°.	
Index ranges	-13 ≤ h ≤ 13, -14 ≤ k ≤ 14, -13 ≤ l ≤ 13	
Reflections collected	4755	
Independent reflections	2717 [R(int) = 0.0162]	
Completeness to theta = 27.49°	99.1 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	2717 / 0 / 218	
Goodness-of-fit on F ²	0.993	
Final R indices [I > 2σ(I)]	R ₁ = 0.0395, wR ₂ = 0.0918	
R indices (all data)	R ₁ = 0.0481, wR ₂ = 0.0964	
Extinction coefficient	2.0(6) × 10 ⁻⁵	
Largest diff. peak and hole	0.303 and -0.176 e.Å ⁻³	

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **1b**. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
C1	2524(1)	6794(1)	2126(1)	25(1)
C2	3953(1)	6387(1)	2188(1)	22(1)
C3	4040(1)	5018(1)	1839(1)	28(1)
C4	3188(1)	4743(1)	529(1)	33(1)
C5	1771(1)	5109(1)	472(1)	36(1)
C6	1675(1)	6455(1)	824(1)	30(1)
O7	1988(1)	6137(1)	3061(1)	33(1)
C8	2441(1)	8172(1)	2335(1)	34(1)
C9	4858(1)	6574(1)	3481(1)	25(1)
O10	4446(1)	6687(1)	4464(1)	37(1)
C11	6306(1)	6520(1)	3566(1)	24(1)
C12	6873(1)	6812(1)	2539(1)	28(1)
C13	8225(1)	6769(1)	2681(1)	34(1)
C14	9015(1)	6416(1)	3834(1)	36(1)
C15	8456(1)	6109(1)	4853(1)	34(1)
C16	7111(1)	6167(1)	4725(1)	28(1)

Table 3. Bond lengths [Å] and angles [°] for **1b**.

C1-O7	1.4395(13)	C6-H6B	1.006(15)
C1-C8	1.5236(16)	O7-H7	0.901(19)
C1-C6	1.5307(16)	C8-H8A	0.994(15)
C1-C2	1.5464(15)	C8-H8B	0.997(17)
C2-C9	1.5163(15)	C8-H8C	1.006(16)
C2-C3	1.5456(15)	C9-O10	1.2274(13)
C2-H2	0.996(12)	C9-C11	1.4986(15)
C3-C4	1.5246(16)	C11-C12	1.3948(15)
C3-H3A	1.018(14)	C11-C16	1.3976(15)
C3-H3B	0.994(14)	C12-C13	1.3895(17)
C4-C5	1.5225(19)	C12-H12	0.983(13)
C4-H4A	0.991(15)	C13-C14	1.3865(18)
C4-H4B	1.028(14)	C13-H13	0.982(15)
C5-C6	1.5230(17)	C14-C15	1.3863(19)
C5-H5A	0.982(15)	C14-H14	0.969(16)
C5-H5B	1.005(15)	C15-C16	1.3847(17)
C6-H6A	0.994(14)	C15-H15	0.979(15)
		C16-H16	0.971(14)
O7-C1-C8	110.13(9)	C4-C3-H3B	109.3(8)
O7-C1-C6	106.20(9)	C2-C3-H3B	109.0(8)
C8-C1-C6	109.42(10)	H3A-C3-H3B	107.4(11)
O7-C1-C2	110.10(8)	C5-C4-C3	110.94(10)
C8-C1-C2	111.16(9)	C5-C4-H4A	109.7(8)
C6-C1-C2	109.72(9)	C3-C4-H4A	109.6(8)
C9-C2-C1	114.21(9)	C5-C4-H4B	110.0(8)
C9-C2-C3	107.06(9)	C3-C4-H4B	109.0(8)
C1-C2-C3	111.88(9)	H4A-C4-H4B	107.5(11)
C9-C2-H2	108.6(7)	C4-C5-C6	110.95(10)
C1-C2-H2	106.8(7)	C4-C5-H5A	109.6(9)
C3-C2-H2	108.1(7)	C6-C5-H5A	109.3(9)
C4-C3-C2	111.08(9)	C4-C5-H5B	109.4(8)
C4-C3-H3A	110.5(8)	C6-C5-H5B	109.4(8)
C2-C3-H3A	109.4(8)	H5A-C5-H5B	108.2(12)

C5-C6-C1	113.72(10)	C12-C11-C9	122.54(10)
C5-C6-H6A	109.1(8)	C16-C11-C9	118.25(10)
C1-C6-H6A	107.2(8)	C13-C12-C11	120.00(11)
C5-C6-H6B	111.2(8)	C13-C12-H12	119.0(8)
C1-C6-H6B	106.4(8)	C11-C12-H12	121.0(8)
H6A-C6-H6B	109.0(11)	C12-C13-C14	120.35(11)
C1-O7-H7	107.1(12)	C12-C13-H13	119.2(8)
C1-C8-H8A	111.4(8)	C14-C13-H13	120.5(8)
C1-C8-H8B	109.4(10)	C15-C14-C13	119.93(11)
H8A-C8-H8B	108.4(12)	C15-C14-H14	120.4(9)
C1-C8-H8C	110.5(8)	C13-C14-H14	119.6(9)
H8A-C8-H8C	108.3(12)	C16-C15-C14	120.05(11)
H8B-C8-H8C	108.7(13)	C16-C15-H15	119.7(9)
O10-C9-C11	119.20(10)	C14-C15-H15	120.3(9)
O10-C9-C2	122.24(10)	C15-C16-C11	120.46(11)
C11-C9-C2	118.38(9)	C15-C16-H16	120.7(8)
C12-C11-C16	119.20(10)	C11-C16-H16	118.8(8)

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **1b**. 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
C1	23(1)	25(1)	28(1)	2(1)	9(1)	1(1)
C2	23(1)	24(1)	22(1)	1(1)	8(1)	0(1)
C3	33(1)	25(1)	25(1)	0(1)	6(1)	5(1)
C4	42(1)	26(1)	28(1)	-4(1)	2(1)	4(1)
C5	36(1)	33(1)	36(1)	-3(1)	1(1)	-7(1)
C6	25(1)	32(1)	33(1)	2(1)	3(1)	1(1)
O7	29(1)	39(1)	34(1)	4(1)	14(1)	-3(1)
C8	35(1)	28(1)	41(1)	-3(1)	9(1)	5(1)
C9	26(1)	25(1)	24(1)	-1(1)	8(1)	1(1)
O10	32(1)	56(1)	25(1)	-5(1)	11(1)	3(1)
C11	25(1)	22(1)	25(1)	-3(1)	6(1)	0(1)
C12	28(1)	29(1)	27(1)	-2(1)	8(1)	-2(1)
C13	31(1)	34(1)	40(1)	-3(1)	15(1)	-4(1)
C14	24(1)	33(1)	50(1)	-6(1)	6(1)	0(1)
C15	30(1)	31(1)	37(1)	-3(1)	-1(1)	3(1)
C16	31(1)	26(1)	27(1)	-1(1)	6(1)	1(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for 1b.

	x	y	z	U(eq)
H2	4284(12)	6885(11)	1544(11)	24(3)
H3A	4990(14)	4795(13)	1861(13)	36(3)
H3B	3744(13)	4507(13)	2492(13)	37(4)
H4A	3235(14)	3856(14)	338(13)	41(4)
H4B	3548(14)	5216(13)	-151(13)	40(4)
H5A	1406(14)	4598(14)	1065(14)	43(4)
H5B	1240(14)	4965(13)	-414(14)	45(4)
H6A	1967(13)	6973(12)	175(12)	31(3)
H6B	750(14)	6683(13)	857(13)	37(4)
H7	2537(19)	6254(16)	3822(17)	65(5)
H8A	2962(14)	8420(13)	3186(14)	41(4)
H8B	1510(17)	8409(15)	2286(15)	56(5)
H8C	2779(14)	8636(14)	1661(14)	43(4)
H12	6333(13)	7073(12)	1716(13)	34(3)
H13	8613(14)	6994(13)	1956(14)	42(4)
H14	9954(16)	6361(14)	3913(14)	49(4)
H15	9008(15)	5839(14)	5663(14)	46(4)
H16	6709(13)	5951(12)	5432(13)	35(3)

Table 6. Torsion angles [°] for **1b**.

O7-C1-C2-C9	-58.18(12)
C8-C1-C2-C9	64.13(12)
C6-C1-C2-C9	-174.71(9)
O7-C1-C2-C3	63.63(11)
C8-C1-C2-C3	-174.06(9)
C6-C1-C2-C3	-52.90(11)
C9-C2-C3-C4	-178.40(10)
C1-C2-C3-C4	55.77(12)
C2-C3-C4-C5	-56.69(13)
C3-C4-C5-C6	55.94(14)
C4-C5-C6-C1	-55.31(14)
O7-C1-C6-C5	-65.70(12)
C8-C1-C6-C5	175.46(10)
C2-C1-C6-C5	53.26(13)
C1-C2-C9-O10	20.25(15)
C3-C2-C9-O10	-104.18(12)
C1-C2-C9-C11	-164.56(9)
C3-C2-C9-C11	71.02(12)
O10-C9-C11-C12	-157.44(11)
C2-C9-C11-C12	27.22(15)
O10-C9-C11-C16	22.33(16)
C2-C9-C11-C16	-153.02(10)
C16-C11-C12-C13	-1.02(16)
C9-C11-C12-C13	178.75(10)
C11-C12-C13-C14	1.09(18)
C12-C13-C14-C15	-0.23(18)
C13-C14-C15-C16	-0.69(18)
C14-C15-C16-C11	0.75(18)
C12-C11-C16-C15	0.10(16)
C9-C11-C16-C15	-179.67(10)

Figure 1. View of **1b** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. Hydrogen atoms are drawn to an arbitrary size. The dashed line is indicative of a H-bonding interaction with geometry: O7-H7 $\cdots$ O10, O $\cdots$ O 2.752(1)Å, H $\cdots$ O 2.02(2)Å, O-H $\cdots$ O 137(2)°.

Figure 2. Unit cell packing diagram for 1. The view is approximately down the *a* axis.