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(1*S*,2*R*)-Methyl 2-Ethoxy-(*E*)-1-(1(*E*)-propenyl)cyclopropane-1-carboxylate

(3b).^{11a} **2b** (200 mg, 1.43 mmol), Rh₂(S-DOSP)₄ (27 mg, 0.014 mmol), ethyl vinyl ether (0.68 mL, 7.2 mmol), pentane (40 mL), (5:95--10:90); yield 198 mg (75%); 93% ee, determined by ¹H NMR (300 MHz) using (-)-Pr(hfc)₃ (0.16 equiv). [α]²⁵_D = 47.9° (c 1.5, CHCl₃).

(1*R*,2*R*,3*R*)-Methyl 2-Ethoxy-3-methyl-1β-((*E*)-2-phenylethenyl)cyclo-propane-1-carboxylate (3c).^{11a} **2a** (357 mg, 1.77 mmol), Rh₂(S-DOSP)₄ (33 mg, 0.018 mmol), ethoxy propenyl ether (0.98 mL, 8.82 mmol, 1 : 1 *E/Z* mixture), pentane (60 mL), (5:95--10:90); yield 235 mg (51%); 65% ee, determined by HPLC (OD column); flow rate 1 mL/min, 0.5% 2-propanol in hexane; UV 254nm; T_R = 14 min (major), 15 min (minor); [α]²⁵_D = 55.5 (c 0.85, CHCl₃).

(1*R*,5*R*,6*R*)-Methyl 6-((*E*)-2-Phenylethenyl)bicyclo[3.1.0]hexane-6-carboxylate (3d).¹² **2a** (200 mg, 0.99 mmol), Rh₂(S-DOSP)₄ (19 mg, 0.010 mmol), 2,3-dihydrofuran (0.38 mL, 4.95 mmol), pentane (65 mL), (10:90); yield 203 mg (84%); 86% ee, determined by HPLC (OJ column); flow rate 1 mL/min, 10% 2-propanol in hexane; UV 254nm; T_R = 20 min (major), 24 min (minor).

(1*R*,5*R*,6*R*)-Methyl 6-((*E*)-1-Propenyl)bicyclo[3.1.0]hexane-6-carboxylate (3e).
2b (250 mg, 1.78 mmol), Rh₂(S-DOSP)₄ (34 mg, 0.018 mmol), 2,3-dihydrofuran (0.67 mL, 8.9 mmol), (pentane 50 mL), (5:95); yield 220 mg (68%); 86% ee, determined by ¹H NMR (400 MHz) using (-)-Pr(hfc)₃ (0.06 equiv). [α]²⁵_D = -51.3 (c 2.0, CHCl₃). FTIR (neat) 2951, 2898, 1713, 1242 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 5.83 (dq, 1 H, J = 15.8, 6.5 Hz), 5.46 (dq, 1 H, J = 15.8, 1.6 Hz), 4.24 (d, 1 H, J = 5.6 Hz), 4.09-4.01 (m, 1 H), 3.62 (s, 3 H), 3.62-3.51 (m, 1 H), 2.38 (t, 1 H, J = 6.0 Hz), 2.24-2.16 (m, 1 H), 1.94-1.86 (m, 1 H), 1.93 (dd, 3 H, J = 6.5, 1.6 Hz). ¹³C NMR (75.45 MHz, CDCl₃) δ 171.9, 133.9, 119.9, 71.4, 69.7, 51.7, 34.5, 31.7, 25.1, 18.3. Anal. Calcd for C₁₀H₁₄O₃: C, 65.92; H, 7.74. Found: C, 65.80; H, 7.69.

~~-2- $\alpha\alpha$~~

(1*R*,6*R*,7*R*)-Methyl 7-((E)-2-Phenylethenyl)bicyclo[4.1.0]heptane-7-carboxylate (3f).

^{11a} **2a** (420 mg, 2.07 mmol), Rh₂(S-DOSP)₄ (39 mg, 0.02 mmol), dihydropyran (0.95 mL, 10.4 mmol), pentane (40 mL), (5:95--10:90); yield 352 mg (66%); 88% ee, determined by HPLC (OD column); flow rate 1 mL/min, 2% 2-propanol in hexane; UV 254nm; T_R = 10 min (minor), 11.6 min (major); [α]²⁵_D = 14.2 (c 2.0, CHCl₃).

~~-2- $\alpha\alpha$~~ ~~-2- $\alpha\alpha$~~

(1*R*,6*R*,7*R*)-Methyl 7-((E)-1-Propenyl)bicyclo[4.1.0]heptane-7-carboxylate (3g)

^{11a} **2b** (250 mg, 1.78 mmol), Rh₂(S-DOSP)₄ (34 mg, 0.018 mmol), dihydropyran (0.81 mL, 8.9 mmol), pentane (50 mL), (5:95); yield 139 mg (57%); 87% ee; determined by ¹H NMR (400 MHz) using (-)-Pr(hfc)₃ (0.033 eq). [α]²⁵_D = 14.9 (c 1.0, CHCl₃).

~~-2- $\alpha\alpha$~~ -

(1*R*,5*R*,6*S*)-Methyl 6-(1-cyclopentenyl)bicyclo[3.1.0]hexane-6-carboxylate (3h).

2c (262 mg, 1.58 mmol), Rh₂(S-DOSP)₄ (34 mg, 0.018 mmol), dihydrofuran (0.68 mL, 9.05 mmol), pentane (60 mL), (5:95); yield 158 mg (49 %); 76% ee, determined by ¹H NMR (300 MHz) using (-)-Pr(hfc)₃ (0.034 equiv), [α]²⁵_D = -29.1°(c 2.2, CHCl₃). FTIR (neat) 2950, 2896, 1713, 1253, 1243, 1080 cm⁻¹; ¹H NMR (300 MHz) δ 5.71 (t, 1 H, J = 1.8 Hz), 4.24 (d, 1 H, J = 5.9 Hz), 4.0 (dd, 1 H, J = 11.5, 7.1, 8.4, 4.4 Hz), 3.60 (s, 3 H), 3.46 (q, 1 H, J = 8.4 Hz), 2.47-2.18 (m, 6 H), 1.98-1.81 (m, 3 H). ¹³C NMR (75.5 MHz, CDCl₃) δ 171.5, 153.6, 131.7, 71.5, 70.8, 51.9, 34.4, 34.0, 32.8, 31.8, 26.1, 23.4. Anal. Calcd for C₁₂H₁₆O₃: C, 69.21; H, 7.74. Found: C, 69.38; H, 7.70.

~~-2- $\alpha\alpha$~~

(1*R*,6*R*,7*S*)-Methyl 6-(1-cyclopentenyl)bicyclo[4.1.0]heptane-7-carboxylate (3i).

2c (200 mg, 1.2 mmol), Rh₂(S-DOSP)₄ (23 mg, 0.012 mmol), dihydropyran (0.55 mL, 6.0 mmol), (pentane, 50 mL), (5:95); yield 99 mg (53%); 88% ee, determined by ¹H NMR (300 MHz) using (-)-Pr(hfc)₃ (0.038 equiv), [α]²⁵_D = -11.9 (c 1.9, CHCl₃). FTIR (neat) 2950, 2853, 1716, 1254, 1243, 1078 cm⁻¹; ¹H NMR (500 MHz) δ 5.76 (s, 1 H), 3.94 (d, 1 H, J = 7.3 Hz),

3.61 (s, 3 H), 3.57 (dt, 1 H, $J = 10.7, 7.0$ Hz), 3.33 (td, 1 H, $J = 11.3, 2.4$ Hz), 2.58-2.49 (m, 1 H), 2.41-2.31 (m, 3 H), 2.24-1.82 (m, 5 H), 1.44-1.26 (m, 2 H); ^{13}C NMR (125.7 Hz) δ 173.3, 136.7, 132.0, 64.4, 62.6, 52.1, 34.5, 32.8, 31.0, 24.7, 23.4, 21.7, 17.4. Anal. Calcd for $\text{C}_{13}\text{H}_{18}\text{O}_3$: C, 70.24; H, 8.16. Found: C, 70.10; H, 8.06.

(1*R*,1*aR*,6*bR*)-Methyl 1*a*,6*b*-Dihydro-1-((*Z*)-(2-phenylethenyl)-1*H*-cyclopropa[b]benzofuran-1-carboxylate (3j). **2a** (300 mg, 1.48 mmol), $\text{Rh}_2(\text{S-DOSP})_4$ (28 mg, 0.015 mmol), benzofuran (0.82 mL, 7.4 mmol), pentane (60 mL), (5:95--10:90); yield 248 mg (57%); 96% ee, determined by HPLC (OD column); flow rate 1 mL/min, 2.5% 2-propanol in hexane; UV 254nm; $T_R = 10$ min (major), 13 min (minor). FTIR (neat) 3025, 2951, 1715, 1241 cm⁻¹; ^1H NMR (300 MHz, CDCl_3) δ 7.39 (dd, 1 H, $J = 7.4, 1.0$ Hz), 7.21-7.04 (m, 6 H), 6.91 (dt, 1 H, $J = 6.5, 1.0$ Hz), 6.76 (d, 1 H, $J = 8.1$ Hz), 6.47 (d, 1 H, $J = 16.2$ Hz), 5.56 (dd, 1 H, $J = 16.2, 0.8$ Hz), 5.25 (dd, 1 H, $J = 5.5, 0.8$ Hz), 3.74 (s, 3 H), 3.58 (d, 1 H, $J = 5.5$ Hz). ^{13}C NMR (75.5 MHz, CDCl_3) δ 173.2, 160.3, 137.9, 137.0, 128.5, 128.3, 127.6, 126.2, 125.5, 125.2, 121.3, 116.5, 109.9, 71.0, 52.5, 37.6, 26.6. HRMS Calcd for $\text{C}_{19}\text{H}_{16}\text{O}_3$, 292.1100, Found: 292.1089.

Methyl ($4\alpha,5\alpha$)-4-Ethoxy-5-methylcyclopent-1-ene-1-carboxylate (4b).^{11a} **3b** (140 mg, 0.76 mmol), (5:95); yield 93 mg (66%); 11% ee, determined by HPLC (OD); flow rate 1.0 mL/min, 1% 2-propanol in hexane; UV 254nm; $T_R = 6.4$ min (minor), 7.5 min (major).

(3*S*,4*R*,5*R*)-Methyl 4-Ethoxy-5-methyl-3-phenylcyclopent-1-ene-1-carboxylate (5c).^{11a} **3c** (236 mg, 0.91 mmol), (5:95); yield 170 mg (72%); 43% ee, determined by HPLC (OD); flow rate 1 mL/min, 2% 2-propanol in hexane; UV 254nm; $T_R = 5.6$ min (major), 7.6 min (minor); $[\alpha]^{25}_D = -44.3^\circ$ (*c* 0.84, CHCl_3).

(1*R*,5*R*,8*S*)-Methyl 8-Phenyl-4-oxabicyclo[3.3.0]oct-6-ene-6-carboxylate (6d). **3d** (158 mg, 0.65 mmol), (10:90); yield 104 mg (66%); 86% ee, determined by HPLC (OD); flow rate 1 mL/min, 2% 2-propanol in hexane; UV 254nm; $T_R = 9.8$ min (major), 12 min (minor);

$[\alpha]^{25}_D = -133.7$ (*c* 1.2, CHCl_3). FTIR (neat) 2947, 2854, 1718 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 7.33-7.20 (m, 3 H), 7.12-7.09 (m, 2 H), 6.79 (m, 1 H), 4.50 (d, 1 H, *J* = 6.2 Hz), 3.99 (br s, 1 H), 3.89-3.68 (m, 3 H), 3.78 (s, 3 H), 2.20-1.97 (m, 2 H). ^{13}C NMR (75.5 MHz, CDCl_3) δ 165.2, 144.9, 141.1, 137.0, 128.9, 127.5, 127.1, 89.9, 67.1, 59.0, 51.6, 49.0, 30.9. HRMS Calcd for $\text{C}_{15}\text{H}_{16}\text{O}_3$, 244.1099, Found: 244.1107.

(1*R*,5*R*,8*R*)-Methyl 8-Methyl-4-oxabicyclo[3.3.0]oct-6-ene-6-carboxylate (6e). *3e* (110 mg, 0.6 mmol), (5:95); yield 79 mg (72%); 85% ee, determined by HPLC (OJ); flow rate 1 mL/min, 1% 2-propanol in hexane; UV 254nm; T_R = 8.1 min (major), 8.8 min (minor); $[\alpha]^{25}_D$ = -9.1 (*c* 1.1, CHCl_3). FTIR (neat) 2953, 2871, 1715, 1223 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 6.66 (br s, 1 H), 4.18 (d, 1 H, *J* = 6.3 Hz), 3.76-3.53 (m, 3 H), 3.72 (s, 3 H), 2.85-2.75 (m, 1 H), 2.14-2.02 (m, 1 H), 1.93-1.84 (m, 1 H), 1.05 (d, 3 H, *J* = 7.4 Hz). ^{13}C NMR (75.5 MHz, CDCl_3) δ 165.4, 148.1, 135.2, 88.9, 67.0, 51.4, 48.3, 47.2, 30.9, 18.1. Anal. Calcd for $\text{C}_{10}\text{H}_{14}\text{O}_3$: C, 65.92; H, 7.74. Found: C, 65.76; H, 7.64.

(1*R*,6*R*,9*S*)-Methyl 9-Phenyl-5-oxabicyclo[4.3.0]non-7-ene-7-carboxylate (7f). *3f* (112 mg, 0.43 mmol), (5:95); yield 98 mg (87%); 82% ee, determined by HPLC (OD); flow rate 1.4 mL/min, 2.5% 2-propanol in hexane; UV 254nm; T_R = 6.8 min (major), 9 min (minor); $[\alpha]^{25}_D$ = -125.5 (*c* 0.95, CHCl_3).

(1*S*,6*R*,9*R*)-Methyl 9-Methyl-5-oxabicyclo[4.3.0]non-7-ene-7-carboxylate (7g). *11a* *3g* (134 mg, 0.68 mmol), (5:95); yield 107 mg (80%); 84% ee, determined by HPLC (OD); flow rate 1 mL/min, 1% 2-propanol in hexane; UV 254nm; T_R = 7.7 min (minor), 8.6 min (major); $[\alpha]^{25}_D$ = -72.7 (*c* 2.1, CHCl_3).

(*R,S,R*)-Methyl 2,3,3a,5,6,7,7a,7b-Octahydro-pentaleno[1,2-b]furan-4-carboxylate (8h). *3h* (110 mg, 0.54 mmol), (10:90); yield 87 mg (79%); 74% ee, determined

by HPLC (OD); flow rate 1 mL/min, 0.8% 2-propanol in hexane; UV 254nm; $T_R = 9.7$ min (minor), 10.5 min (major); $[\alpha]^{25}_D = -58.3$ (c 1.3 CHCl₃). FTIR (neat) 2952, 2869, 1711, 1227, 1128 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 4.57 (dd, 1 H, $J = 8.1, 3.7$ Hz), 3.92-3.70 (m, 3 H), 3.72 (s, 3 H), 3.04-2.95 (m, 1 H), 2.64-2.53 (m, 1 H), 2.43-2.32 (m, 1 H), 2.12-1.78 (m, 4 H), 1.22-1.13 (m, 2 H); ¹³C NMR (125.7 MHz, CDCl₃) δ 169.1, 165.6, 123.9, 85.7, 68.5, 60.0, 54.5, 51.1, 32.4, 28.7, 26.8, 25.5. Anal. Calcd for C₁₂H₁₆O₃: C, 69.21; H, 7.74. Found: C, 69.37; H, 7.71.

(R,S,R)-Methyl 2,3,3a,3b,5,6,7,7a-Octahydro-1H-4-oxa-cyclopenta[a]indene-8-carboxylate (8i). 3i (119 mg, 0.54 mmol). (10:90); yield 83 mg (69%); 86% ee, determined by HPLC (OD); flow rate 1 mL/min, 1% 2-propanol in hexane; UV 254 nm; $T_R = 8.5$ min (minor), 9.8 min (major); $[\alpha]^{25}_D = -81.4$ (c 1.24, CHCl₃). FTIR (neat) 2947, 2860, 1708, 1434 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 3.93 (dd, 1 H, $J = 8.5, 7.7$ Hz), 3.78-3.55 (m, 2 H), 3.69 (s, 3 H), 3.37 (m, 1 H), 2.95 (ddd, 1 H, $J = 12.5, 6.5, 6.5$ Hz), 2.64-2.34 (m, 2 H), 2.16-1.86 (m, 4 H), 1.56-1.13 (m, 4 H); ¹³C NMR (75.5 MHz, CDCl₃) δ 168.0, 165.7, 126.3, 81.8, 62.3, 51.0, 50.7, 45.7, 29.5, 28.6, 26.8, 26.0, 22.9; Anal. Calcd for C₁₃H₁₈O₃: C, 70.24; H, 8.16. Found: C, 70.14; H, 8.22.

(3S,3aS,8bR)-Methyl 3-phenyl-8bH-cyclopenta[b]benzofuran-1-carboxylate (9j). 3j (146 mg, 0.50 mmol), (5:95); yield 89 mg (61%); 11% ee, determined by HPLC (OJ); flow rate 1 mL/min, 15% 2-propanol in hexane; UV 254nm; $T_R = 10.5$ min (major), 16 min (minor). FTIR (neat) 3027, 2949, 1721, 1477, 1254 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 7.64-7.38 (m, 7 H), 7.23 (d, 1 H, $J = 2.4$ Hz), 7.15 (t, 1 H, $J = 7.3$ Hz), 7.10 (d, 1 H, $J = 7.3$ Hz), 6.43 (dd, 1 H, $J = 1.8, 7.9$ Hz), 4.41 (m, 1 H), 4.34 (br d, 1 H, $J = 8.5$ Hz), 4.10 (s, 3 H). ¹³C NMR (75.454 MHz, CDCl₃) δ 164.4, 158.4, 149.8, 141.8, 135.3, 129.6, 129.2, 129.1, 127.4,

127.3, 124.6, 121.0, 110.7, 90.1, 60.2, 54.2, 51.9. Anal. Calcd for C₁₉H₁₆O₃: C, 78.06; H, 5.52. Found: C, 77.95; H, 5.54.

KONG V-85

Solvent: CDCl₃

Ambient temperature

GEMINI-300 "rossy.chem.buffalo.edu"

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Acq. time 3.747 sec

Width 4500.5 Hz

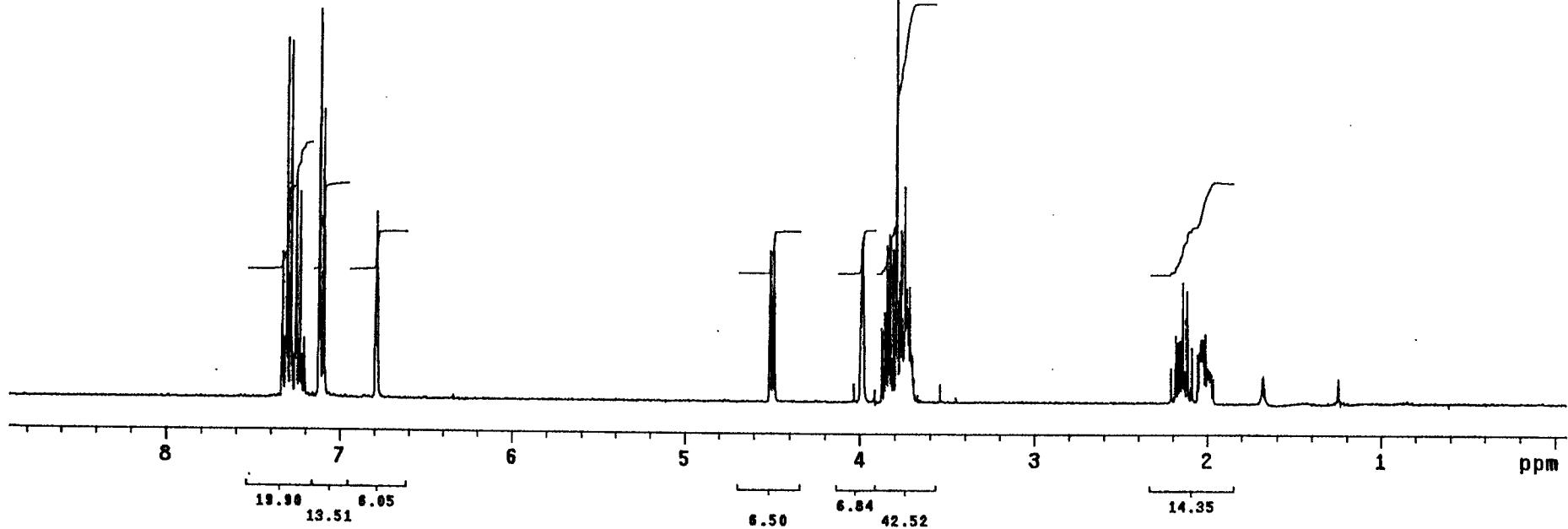
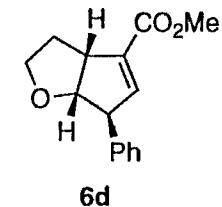
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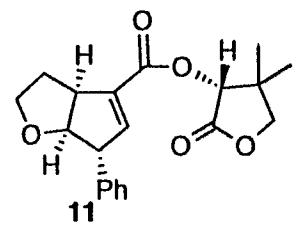
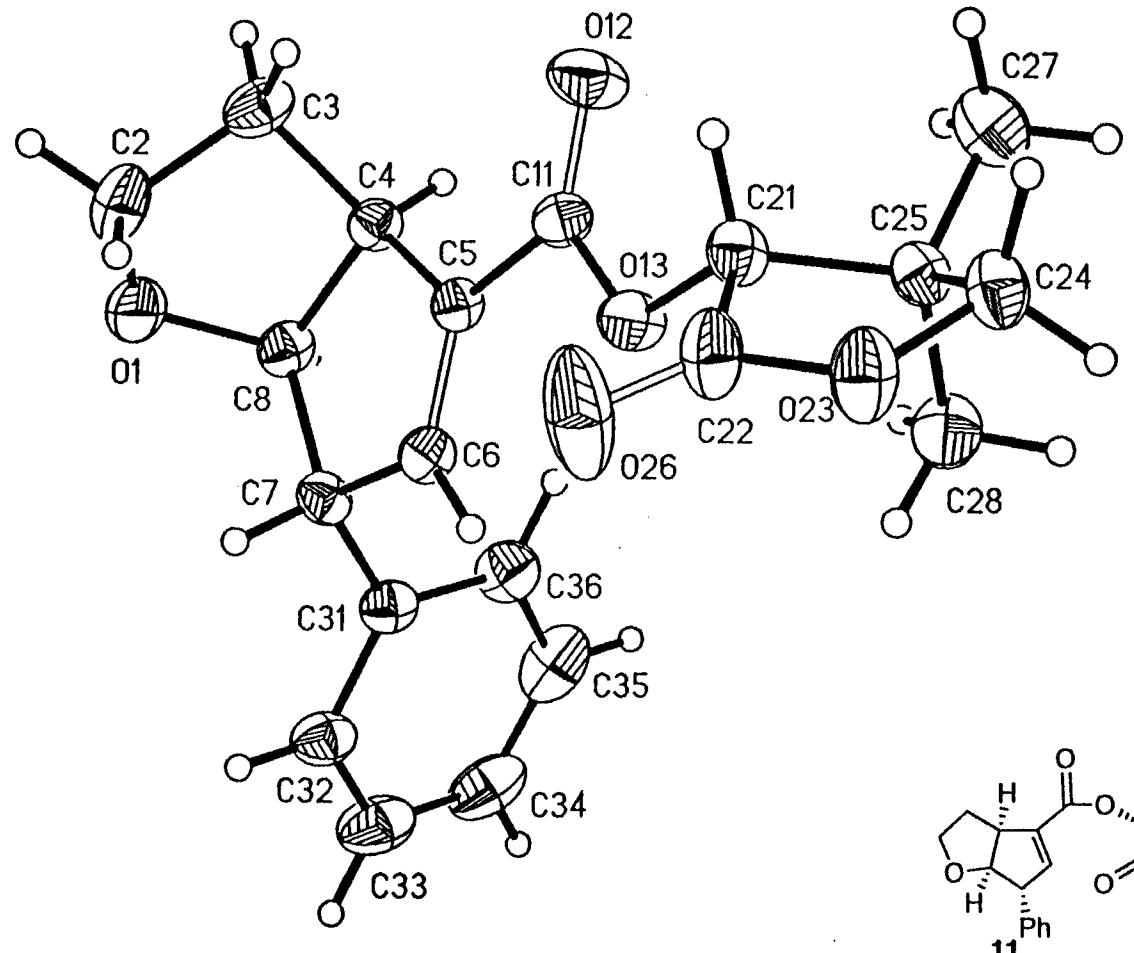
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DATA PROCESSING

FT size 65536

Total time 1 minute





STRUCTURE DETERMINATION SUMMARYCrystal Data

Empirical Formula	C ₂₀ H ₂₂ O ₅
Color; Habit	colorless rectangular parallelepiped
Crystal Size (mm)	0.7 x 0.4 x 0.4
Crystal System	Orthorhombic
Space Group	P2 ₁ 2 ₁ 2 ₁ (No. 19)
Unit Cell Dimensions	<u>a</u> = 6.506(7) Å <u>b</u> = 12.470(13) Å <u>c</u> = 21.652(19) Å
Volume	1757(3) Å ³
Z	4
Formula weight	342.4
Density(calc.)	1.295 Mg/m ³
Absorption Coefficient	0.086 mm ⁻¹
F(000)	728

Data Collection

Diffractometer Used	Siemens R3m/V
Radiation	Mo K α ($\lambda = 0.71073 \text{ \AA}$)
Temperature (K)	296
Monochromator	Highly oriented graphite crystal
2 θ Range	6.0 to 45.0°
Scan Type	2 θ - θ
Scan Speed	Constant; 2.00°/min. in ω
Scan Range (ω)	0.60° plus K α -separation
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 25.0% of total scan time
Standard Reflections	3 measured every 197 reflections
Index Ranges	$0 \leq h \leq 7$, $0 \leq k \leq 13$ $-23 \leq l \leq 23$
Reflections Collected	2683
Independent Reflections	2302 ($R_{\text{int}} = 1.62\%$)
Observed Reflections	1903 ($F > 4.0\sigma(F)$)
Absorption Correction	N/A

Solution and Refinement

System Used	Siemens SHELXTL PLUS RELEASE 4.11(VMS)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(F_o - F_c)^2$
Absolute Structure	$\eta = +0.17(3.89)$
Extinction Correction	$x = 0.0026(4)$, where $F^* = F [1 + 0.002x F^2 / \sin(2\theta)]^{-1/4}$
Hydrogen Atoms	Riding model, Isotropic U's refined
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0012F^2$
Number of Parameters refined	228
Final R indices (obs. data)	$R = 3.55\%$, $wR = 4.48\%$
R Indices (all data)	$R = 5.12\%$, $wR = 5.15\%$
Goodness-of-Fit	0.97
Largest and Mean Δ/σ	0.001, 0.000
Data-to-Parameter Ratio	8.3:1
Largest Difference Peak	$0.26 \text{ e}\text{\AA}^{-3}$
Largest Difference Hole	$-0.21 \text{ e}\text{\AA}^{-3}$

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	x	y	z	U(eq)
O(1)	2584(4)	6177(2)	5126(1)	68(1)
C(2)	2142(5)	6660(3)	5707(2)	62(1)
C(3)	4031(6)	7274(2)	5865(1)	54(1)
C(4)	5738(4)	6536(2)	5638(1)	39(1)
C(5)	6440(4)	5707(2)	6097(1)	35(1)
C(6)	6011(5)	4716(2)	5919(1)	40(1)
C(7)	4984(5)	4670(2)	5304(1)	41(1)
C(8)	4688(5)	5866(2)	5134(1)	44(1)
C(11)	7454(5)	6003(2)	6681(1)	40(1)
O(12)	7796(4)	6895(2)	6851(1)	69(1)
O(13)	7947(3)	5130(2)	7016(1)	50(1)
C(21)	8794(5)	5274(2)	7617(1)	44(1)
C(22)	7882(6)	4420(3)	8029(2)	58(1)
O(23)	9282(3)	4088(2)	8431(1)	60(1)
C(24)	11188(5)	4658(3)	8332(1)	51(1)
C(25)	11101(5)	5097(2)	7674(1)	42(1)
O(26)	6186(4)	4073(3)	8015(1)	104(1)
C(27)	12349(6)	6124(3)	7613(2)	66(1)
C(28)	11838(5)	4252(3)	7219(2)	59(1)
C(31)	6162(5)	4033(2)	4829(1)	43(1)
C(32)	5279(5)	3152(3)	4540(2)	53(1)
C(33)	6293(8)	2606(3)	4091(2)	73(2)
C(34)	8180(8)	2909(3)	3906(2)	76(2)
C(35)	9135(6)	3769(4)	4187(2)	77(2)
C(36)	8109(6)	4335(3)	4656(2)	60(1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2. Bond lengths (Å)

O(1)-C(2)	1.422 (5)	O(1)-C(8)	1.422 (4)
C(2)-C(3)	1.488 (5)	C(3)-C(4)	1.524 (5)
C(4)-C(5)	1.504 (4)	C(4)-C(8)	1.535 (4)
C(5)-C(6)	1.324 (4)	C(5)-C(11)	1.474 (4)
C(6)-C(7)	1.490 (4)	C(7)-C(8)	1.548 (4)
C(7)-C(31)	1.509 (4)	C(11)-O(12)	1.192 (4)
C(11)-O(13)	1.346 (4)	O(13)-C(21)	1.424 (4)
C(21)-C(22)	1.510 (5)	C(21)-C(25)	1.522 (5)
C(22)-O(23)	1.327 (4)	C(22)-O(26)	1.186 (5)
O(23)-C(24)	1.445 (4)	C(24)-C(25)	1.526 (4)
C(25)-C(27)	1.522 (5)	C(25)-C(28)	1.521 (5)
C(31)-C(32)	1.388 (5)	C(31)-C(36)	1.373 (5)
C(32)-C(33)	1.358 (5)	C(33)-C(34)	1.345 (7)
C(34)-C(35)	1.381 (6)	C(35)-C(36)	1.406 (6)

Table 3. Bond angles ($^{\circ}$)

C(2)-O(1)-C(8)	107.5(2)	O(1)-C(2)-C(3)	104.7(3)
C(2)-C(3)-C(4)	102.5(3)	C(3)-C(4)-C(5)	115.1(2)
C(3)-C(4)-C(8)	103.5(2)	C(5)-C(4)-C(8)	103.4(2)
C(4)-C(5)-C(6)	112.6(2)	C(4)-C(5)-C(11)	122.1(2)
C(6)-C(5)-C(11)	125.3(3)	C(5)-C(6)-C(7)	113.0(3)
C(6)-C(7)-C(8)	103.4(2)	C(6)-C(7)-C(31)	113.7(3)
C(8)-C(7)-C(31)	114.0(2)	O(1)-C(8)-C(4)	106.7(2)
O(1)-C(8)-C(7)	112.6(2)	C(4)-C(8)-C(7)	107.4(2)
C(5)-C(11)-O(12)	125.5(3)	C(5)-C(11)-O(13)	111.5(2)
O(12)-C(11)-O(13)	123.0(3)	C(11)-O(13)-C(21)	118.8(2)
O(13)-C(21)-C(22)	107.4(2)	O(13)-C(21)-C(25)	116.0(2)
C(22)-C(21)-C(25)	103.7(2)	C(21)-C(22)-O(23)	109.8(3)
C(21)-C(22)-O(26)	127.4(3)	O(23)-C(22)-O(26)	122.8(3)
C(22)-O(23)-C(24)	109.7(3)	O(23)-C(24)-C(25)	106.5(2)
C(21)-C(25)-C(24)	99.5(2)	C(21)-C(25)-C(27)	113.4(3)
C(24)-C(25)-C(27)	111.3(3)	C(21)-C(25)-C(28)	111.0(2)
C(24)-C(25)-C(28)	110.1(3)	C(27)-C(25)-C(28)	111.0(3)
C(7)-C(31)-C(32)	120.9(3)	C(7)-C(31)-C(36)	120.6(3)
C(32)-C(31)-C(36)	118.4(3)	C(31)-C(32)-C(33)	121.2(3)
C(32)-C(33)-C(34)	121.1(4)	C(33)-C(34)-C(35)	119.8(4)
C(34)-C(35)-C(36)	119.7(4)	C(31)-C(36)-C(35)	119.8(3)

Table 4. Anisotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	63(2)	61(1)	80(2)	16(1)	-31(1)	-6(1)
C(2)	48(2)	72(2)	66(2)	15(2)	2(2)	20(2)
C(3)	75(2)	47(2)	42(2)	17(2)	-3(2)	2(1)
C(4)	43(2)	34(2)	39(2)	0(1)	-1(1)	6(1)
C(5)	39(2)	36(2)	30(1)	2(1)	-3(1)	3(1)
C(6)	46(2)	39(2)	36(2)	1(2)	-3(1)	8(1)
C(7)	46(2)	41(2)	35(2)	-4(1)	-6(1)	0(1)
C(8)	52(2)	44(2)	37(2)	7(2)	-4(1)	3(1)
C(11)	46(2)	34(2)	40(2)	1(1)	2(1)	3(1)
O(12)	103(2)	40(1)	63(2)	-4(1)	-31(1)	1(1)
O(13)	69(1)	41(1)	40(1)	1(1)	-18(1)	4(1)
C(21)	50(2)	49(2)	32(2)	0(2)	-7(1)	1(1)
C(22)	43(2)	76(2)	55(2)	-3(2)	-4(2)	20(2)
O(23)	50(1)	81(2)	48(1)	-2(1)	-5(1)	24(1)
C(24)	44(2)	67(2)	43(2)	-1(2)	-5(2)	-2(1)
C(25)	41(2)	49(2)	35(2)	-4(2)	0(1)	-2(1)
O(26)	53(2)	148(3)	113(2)	-33(2)	-19(2)	73(2)
C(27)	66(2)	75(2)	57(2)	-16(2)	3(2)	1(2)
C(28)	59(2)	72(2)	47(2)	8(2)	3(2)	-11(2)
C(31)	55(2)	39(2)	35(2)	3(2)	-2(1)	6(1)
C(32)	72(2)	44(2)	42(2)	6(2)	-8(2)	-5(2)
C(33)	108(3)	56(2)	55(2)	18(3)	-10(2)	-13(2)
C(34)	107(4)	67(3)	54(2)	34(3)	10(2)	0(2)
C(35)	75(3)	87(3)	70(3)	21(2)	23(2)	26(2)
C(36)	62(2)	55(2)	61(2)	-1(2)	10(2)	7(2)

The anisotropic displacement exponent takes the form:

$$-2\pi^2(h^2 a^2 U_{11} + \dots + 2hka^2 b^2 U_{12})$$

Table 5. H-Atom coordinates ($\times 10^4$) and isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	x	y	z	U
H(2A)	966	7122	5675	73
H(2B)	1885	6126	6017	71
H(3A)	4036	7949	5650	61
H(3B)	4160	7406	6300	69
H(4A)	6879	6939	5479	32
H(6A)	6340	4093	6159	46
H(7A)	3648	4356	5350	27
H(8A)	5280	6000	4735	42
H(21A)	8436	5973	7770	57
H(24A)	11310	5234	8624	50
H(24B)	12337	4184	8379	74
H(27A)	12270	6391	7198	67
H(27B)	13758	5982	7717	83
H(27C)	11804	6651	7893	73
H(28A)	11770	4519	6803	92
H(28B)	10978	3630	7257	77
H(28C)	13232	4063	7315	74
H(32A)	3927	2931	4664	57
H(33A)	5665	1998	3895	100
H(34A)	8879	2529	3583	105
H(35A)	10494	3980	4066	111
H(36A)	8748	4937	4855	64