

A General Method for the Enantioselective Synthesis of Pantolactone Derivatives

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Supporting Information

General Information. All reactions were carried out under an atmosphere of argon or nitrogen in oven-dried glassware with magnetic stirring. THF and CH_2Cl_2 were purified by passage through a bed of activated alumina.¹ Solvents and reagents were purified prior to use following the guidelines of Perrin and Armarego.² $\text{ScCl}_3(\text{thf})_3$ was prepared according to literature procedure.³ AgSbF_6 was purchased from the Cerac Chemical Co., stored in an inert atmosphere dry box and used without further purification. $\text{Me}_4\text{NHB}(\text{OAc})_3$ was prepared according to literature procedure.⁴ Et_2BOMe was purchased from Aldrich Chemical Co. and used as is after distillation. Purification of reaction products was carried out by flash chromatography using EM Reagent silica gel 60 (230-400 mesh). Analytical thin layer chromatography was performed on EM Reagent 0.25 mm silica gel 60-F plates. Visualization was accomplished with UV light and ceric ammonium nitrate stain followed by heating. Optical rotations were measured on a Jasco DIP-0181 digital polarimeter with a sodium lamp and are reported as follows: $[\alpha]_D^{25}$ (c g/100 mL, solvent). Infrared spectra were recorded on a Perkin Elmer 1600 series FT-IR spectrometer. ^1H NMR spectra were recorded on a Varian Inova-500 (500 MHz) or Varian Mercury-400 (400 MHz) spectrometer and are reported in ppm using solvent as an internal standard (CDCl_3 at 7.26 ppm). Data reported as (s=singlet, d=doublet, t=triplet, q=quartet, m=multiplet, b=broad; integration; coupling constant(s) in Hz). Proton-decoupled ^{13}C NMR spectra were recorded on Varian Inova-500 (125 MHz) or Varian Mercury-400 (100 MHz) spectrometer and are reported in ppm using solvent as an internal standard (CDCl_3 at 77.0 ppm). High resolution mass spectra were obtained on Jeol AX-505 or SX-102 spectrometers at the Harvard University Mass Spectrometry Laboratory. Analytical high performance liquid chromatography (HPLC) was performed on a Hewlett-Packard 1100 Series HPLC with a diode array detector using the indicated chiral column.

General Procedure for the Catalyzed Reaction of Thiosilylketene Acetals with Ethyl Glyoxylate.

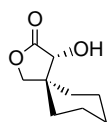
A dry flask was charged with $\text{ScCl}_3(\text{thf})_3$ (0.10 equiv) and phenyl-pyridyl-bis(oxazoliny) ligand (0.11 equiv) in an inert atmosphere (N_2) glove box. In a separate flask, also in an inert atmosphere (N_2) glove box, was charged AgSbF_6 (0.9 equiv). Both flasks were brought out of the glove box and the flask containing the metal/ligand was charged with CH_2Cl_2 (2 mL) and stirred at rt for 30 minutes. The resulting solution was transferred to the flask containing AgSbF_6 via syringe and the mixture stirred for 30 minutes followed by cooling to -78°C . Ethyl glyoxylate (1.5 equiv) and thiosilylketene acetal (1.0 equiv) were added sequentially and stirred for 3 h at -78°C . After the reaction was complete, the mixture was filtered through silica with Et_2O and the silyl ether was hydrolyzed with 1N HCl in EtOAc (30 min, 25°C) to give the hydroxy ester which was purified by flash chromatography.⁵

General Procedure for the Catalyzed Reaction of Enolsilanes with Ethyl Glyoxylate. A dry flask was charged with $\text{ScCl}_3(\text{thf})_3$ (0.15 equiv) and *t*-Bu-pyridyl-bis(oxazoliny) ligand (0.17 equiv) in an

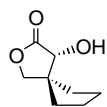
inert atmosphere (N₂) glove box. In a separate flask, also in an inert atmosphere (N₂) glove box, was charged AgSbF₆ (0.14 equiv). Both flasks were brought out of the glove box and the flask containing the metal/ligand was charged with CH₂Cl₂ (1 mL) and stirred at rt for 1 h. The resulting solution was transferred to the flask containing AgSbF₆ via syringe and the mixture stirred for 30 minutes followed by cooling to -78 °C. Ethyl glyoxylate (1.5 equiv), enolsilane (1.0 equiv), and TMS-Cl (2 equiv) were added sequentially and the reaction stirred for 16 h at -35 °C. After the reaction was complete, the mixture was filtered through silica with Et₂O and the silyl ether was hydrolyzed with 1N HCl in EtOAc (30 min, 25 °C) to give the hydroxy ester which was purified by flash chromatography.⁵

General Procedure (A): Raney Nickel Reduction/Cyclization of Thioesters to Pantolactones. To a round bottom flask containing a magnetic stirring bar, was added isopropanol (1.0 mL) and Raney Ni (1.0 mL of a slurry in H₂O, 0.5 g catalyst/mL of settled material). This mixture was purged with hydrogen from a balloon under vigorous stirring. To this suspension of Raney Ni, was added a solution of the aldol adduct (1.0 equiv) in Et₂O (0.52 mL, 0.25 M). The reaction mixture was stirred vigorously until the aldol adduct was completely consumed (15-30 min), as determined by TLC (30% EtOAc/hexanes). The reaction mixture was then filtered through a pad of Celite and washed repeatedly with EtOH (3 x 25 mL). The filtrate was concentrated *in vacuo* to provide the unpurified lactone. Purification by flash chromatography (30–40% EtOAc/hexanes) provided the title compounds.

General Procedure (B): Syn Reduction/Cyclization of Hydroxy-Esters to Pantolactones. To an oven dried flask was added Me₄NHB(OAc)₃ (8.4 equiv) and MeCN/AcOH (1:1 v/v) to afford a 0.9 M solution which was stirred for 30 min at rt before cooling to -20 °C. To this mixture, was added a solution of hydroxy-ester (1.0 equiv) in MeCN (0.65 M) and aged for 1 hr at -20 °C before warming to 0 °C and aging for an additional 16 hr. To the reaction was added sat. Rochelle's salt and sat. NaHCO₃ and then extracted with CH₂Cl₂ (4 x 10 mL). The organic layers were combined and washed with NaHCO₃ (10 mL). The aqueous layer was back-extracted with CH₂Cl₂ (4 x 10 mL). The organic layers were combined and concentrated *in vacuo* to afford a colorless oil which was transferred to a dry flask and charged with benzene (0.23 mL), 3 Å MS (25 mg), and *p*-TSA (0.5 equiv). The reaction was stirred at rt for 30 min. and diluted with Et₂O (2 mL). Triethylamine (6 drops) was added to the mixture which was then filtered through a plug of silica gel and flushed with Et₂O (50 mL). Concentration of the solvent *in vacuo* provided the desired product analytically pure.

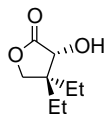


(R)-4-Hydroxy-2-oxa-spiro[4.5]decan-3-one (Table 2, entry 1). The title compound was prepared according to General Procedure A and isolated as a colorless oil (89 % yield): [α]_D²⁵ +16.3 ° (*c* 0.5, CHCl₃); IR (film) 3442, 2938, 2860, 1762, 1559, 1473, 1164, 1103, 990 cm⁻¹; ¹H NMR (400 MHz, CHCl₃) 4.33 (d, 1H, *J*=9.2 Hz), 4.10 (d, 1H, *J*=4.3 Hz), 3.87 (dd, 1H, *J*=1.4, 9.2 Hz), 3.49 (d, 1H, *J*=4.4 Hz), 1.77-1.54 (m, 6H), 1.44-1.35 (m, 2H), 1.26-1.16 (m, 2H); ¹³C NMR (100 MHz, CHCl₃) 178.0, 75.6, 73.7, 44.0, 33.6, 25.8, 25.3, 22.9, 21.8; HRMS (CI, NH₃): Exact mass calcd for C₈H₁₂O₃ [M + NH₄]⁺, 188.1287. Found 188.1283.



(R)-4-Hydroxy-2-oxa-spiro[4.4]nonan-3-one (Table 2, entry 2). The title compound was prepared according to General Procedure A and isolated as a colorless oil (94 % yield): [α]_D²⁵ +19.3 ° (*c* 1.5, CHCl₃); IR (film) 3390, 2952, 2869, 1768, 1318, 1289, 1201, 1146, 1100, 992 cm⁻¹; ¹H NMR (400 MHz, CHCl₃) 4.33 (d, 1H, *J*=3.9 Hz), 4.12 (d, 1H, *J*=8.8 Hz), 4.00 (dd,

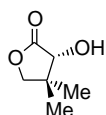
1H, $J=0.9$, 8.8 Hz), 3.38 (br, 1H), 2.03-1.96 (m, 1H), 1.93-1.86 (m, 1H), 1.80-1.58 (m, 5H), 1.44-1.38 (m, 1H); ^{13}C NMR (100 MHz, CHCl_3) 177.8, 76.0, 73.6, 51.6, 33.5, 29.1, 25.0, 24.9; HRMS (CI, NH_3): Exact mass calcd for $\text{C}_8\text{H}_{12}\text{O}_3$ $[\text{M} + \text{NH}_4]^+$, 174.1130. Found 174.1125.



(R)-4,4-Diethyl-3-hydroxy-dihydro-furan-2-one (Table 2, entry 3). The title compound was prepared according to the General Procedure A to provide the pure product as a light yellow oil (95% yield) after flash chromatography 40% EtOAc/hexanes: $[\alpha]_{\text{D}}^{25} -12.0^\circ$ (c 0.33, MeOH); IR (neat) 3423, 2976, 1754, 1637, 1461 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3)

4.21 (br d, 1H, $J = 2.8$ Hz), 4.16 (d, 1H, $J = 8.8$ Hz), 3.87 (d, 1H, $J = 9.2$ Hz), 2.55 (br d, 1H, $J = 2.4$ Hz), 1.65-1.42 (m, 4H), 0.98 (t, 3H, $J = 8.0$ Hz), 0.90 (t, 3H, $J = 7.6$ Hz); ^{13}C NMR (125 MHz, CDCl_3)

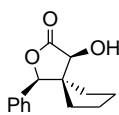
178.2, 74.8, 73.1, 46.7, 28.1, 21.4, 8.49, 8.05; HRMS (CI/ NH_3) exact mass calcd for $\text{C}_8\text{H}_{15}\text{O}_3$ $[\text{M}+\text{H}]^+$, 159.1021. Found 159.1019. Assay of enantiomeric excess: GC analysis (-Cyclodextrin, 60 $^\circ\text{C}$, 3 $^\circ\text{C}/\text{min}$ at 25 psi; t_{r} (major)= 24.38, t_{r} (minor) = 24.0), 95 % ee.



(R)-3-Hydroxy-4,4-dimethyl-dihydro-furan-2-one (Table 2, entry 4). The title compound was prepared according General Procedure A to provide the pure product as a light yellow oil (95% yield) after flash chromatography with 30% EtOAc/hexanes: $[\alpha]_{\text{D}}^{25} -54.0^\circ$ (c 0.25, H_2O); IR (neat) 3442, 2092, 1758, 1638, 1124 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) 4.11 (br s, 1H), 4.03

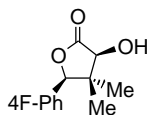
(d, 1H, $J = 9.0$ Hz), 3.94 (d, 1H, $J = 8.5$ Hz), 1.24 (s, 3H), 1.08 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) 177.4, 76.3, 75.7, 40.9, 22.9, 18.8; HRMS (CI/ NH_3) exact mass calcd for $\text{C}_6\text{H}_{11}\text{O}_3$ $[\text{M}+\text{H}]^+$, 131.0708.

Found 131.0710. Assay of enantiomeric excess: GC analysis (-Cyclodextrin, 60 $^\circ\text{C}$, 3 $^\circ\text{C}/\text{min}$ at 25 psi, t_{r} (major)= 16.77, t_{r} (minor) = 17.29), 94% ee.



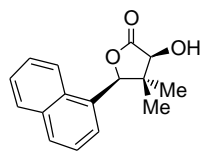
(S,S)-4-Hydroxy-1-phenyl-2-oxa-spiro[4.4]nonan-3-one (Table 2, entry 5). The title compound was prepared according to General Procedure B and isolated as an amorphous white solid (80 % yield). X-ray quality crystals were obtained by recrystallization in hot EtOAc/hexanes: $[\alpha]_{\text{D}}^{25} -14.5^\circ$ (c 0.195, CHCl_3); IR (CHCl_3) 3566, 3108, 2960, 2875, 2359,

1784, 1456 cm^{-1} ; ^1H NMR (400 MHz, CHCl_3) 7.42-7.26 (m, 5H), 5.31 (s, 1H), 4.51 (d, 1H, $J=3.0$ Hz), 2.81 (d, 1H, $J=3.2$ Hz), 2.04-1.89 (m, 2H), 1.71-1.49 (m, 3H), 1.43-1.34 (m, 1H), 1.27-1.20 (m, 1H), 1.01-0.93 (m, 1H); ^{13}C NMR (100 MHz, CHCl_3) 176.9, 134.5, 128.8, 128.6, 126.7, 84.9, 57.2, 34.1, 25.7, 25.0, 24.6; HRMS (EI): Exact mass calcd for $\text{C}_{14}\text{H}_{16}\text{O}_3$ $[\text{M}]^+$, 232.1100. Found 232.1097. Assay of enantiomeric excess: HPLC analysis (Chiracel AD, 10 % $i\text{PrOH}$ /hexanes, 0.7 mL/min, 215 nm; t_{r} (major) = 16.3, t_{r} (minor) = 18.3), 99 % ee.

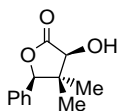


(S,S)-3-Hydroxy-4,4-dimethyl-5-(4-fluorophenyl)-dihydro-furan-2-one (Table 2, entry 6). The title compound was prepared according to General Procedure B and isolated as a colorless oil (71 % yield): $[\alpha]_{\text{D}}^{25} -53.0^\circ$ (c 0.54, CHCl_3); IR (CHCl_3) 3564,

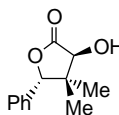
3449, 2970, 2917, 1775, 1611, 1514 cm^{-1} ; ^1H NMR (400 MHz, CHCl_3) 7.26 (t, 2H, $J=8.8$ Hz), 7.09 (t, 2H, $J=8.8$ Hz), 5.09 (s, 1H), 4.34 (s, 1H), 1.27 (s, 3H), 0.62 (s, 3H); ^{13}C NMR (100 MHz, CHCl_3) 176.8, 163.0 (d, $J=248$ Hz), 129.7, 128.0 (d, $J=7.6$ Hz), 115.6 (d, $J=22.1$ Hz), 88.5, 46.5, 22.6, 14.8; HRMS (EI): Exact mass calcd for $\text{C}_{12}\text{H}_{13}\text{O}_3\text{F}$ $[\text{M}]^+$, 224.0849. Found 224.0844.



(S,S)-3-Hydroxy-4,4-dimethyl-5-naphthalen-1-yl-dihydro-furan-2-one (Table 2, entry 7). The title compound was prepared according to General Procedure B and isolated as an amorphous pale yellow solid (75 % yield): $[\alpha]_D^{25} -131.0^\circ$ (c 0.61, CHCl_3); IR (CHCl_3) 3562, 3013, 2971, 1780, 1466, 1121 cm^{-1} ; ^1H NMR (400 MHz, CHCl_3) 7.97-7.87 (m, 3H), 7.61 (d, 1H, $J=6.6$ Hz), 7.56-7.51 (m, 3H), 6.10 (s, 1H), 4.53 (s, 1H), 1.37 (s, 3H), 0.71 (s, 3H); ^{13}C NMR (100 MHz, CHCl_3) 177.1, 133.9, 131.0, 129.7, 129.4, 129.3, 126.6, 125.9, 125.7, 125.3, 123.1, 82.2, 77.9, 47.6, 24.2, 15.7; HRMS (EI): Exact mass calcd for $\text{C}_{16}\text{H}_{16}\text{O}_3$ $[\text{M}]^+$, 256.1099. Found 256.1100.



(S,S)-3-Hydroxy-4,4-dimethyl-5-phenyl-dihydro-furan-2-one (Table 2, entry 8). The title compound was prepared according to General Procedure B and isolated as an amorphous white solid (70 % yield): $[\alpha]_D^{25} -45.1^\circ$ (c 0.41, CHCl_3); IR (CHCl_3) 3567, 3450, 3013, 2970, 2934, 2361, 1785, 1774 cm^{-1} ; ^1H NMR (400 MHz, CHCl_3) 7.42-7.36 (m, 3H), 7.29-7.26 (m, 2H), 5.11 (s, 1H), 4.35 (s, 1H), 2.90 (br, 1H), 1.29 (s, 3H), 0.63 (s, 3H); ^{13}C NMR (100 MHz, CHCl_3) 177.0, 133.9, 128.8, 128.6, 126.2, 86.0, 77.5, 46.5, 22.7, 14.9; HRMS (EI): Exact mass calcd for $\text{C}_{12}\text{H}_{14}\text{O}_3$ $[\text{M}]^+$, 206.0943. Found 206.0939.



(S,R)-3-Hydroxy-4,4-dimethyl-5-phenyl-dihydro-furan-2-one (Table 2, entry 9). To a dry flask was charged 2-Hydroxy-3,3-dimethyl-4-oxo-4-phenyl-butyric acid ethyl ester (24.2 mg, 0.097 mmol), Et_2BOMe (12.6 μL , 0.13 mmol), and THF/MeOH (4:1 v/v, 0.7 mL) and aged at RT for 1 h. After cooling to -78°C , NaBH_4 (9.0 mg, 0.24 mmol) was added and the reaction was aged at -78°C for 21 h. To the reaction was added AcOH (1 mL). After aging at rt for 1 h, the mixture was azeotroped *in vacuo* with MeOH (3 x 50 mL, 50°C) and diluted with Et_2O . The mixture was washed with sat. NaHCO_3 and then back extracted with Et_2O . The organic layers were combined, dried with MgSO_4 and concentrated *in vacuo* to afford a colorless oil which was transferred to a dry flask and charged with benzene (1 mL), 3 Å MS (25 mg), and *p*-TSA (0.5 equiv). The reaction was stirred at rt for 30 minutes and diluted with Et_2O (4 mL). Triethylamine (6 drops) was added to the mixture, which was then filtered through a plug of silica gel and flushed with Et_2O (50 mL). The solvent was removed *in vacuo* and the residue chromatographed (5-15 % EtOAc/hexanes) to afford the title compound as a colorless oil (73 % yield): $[\alpha]_D^{25} -58.5^\circ$ (c 0.59, CHCl_3); IR (CHCl_3) 3577, 3450, 3018, 2967, 1770, 1605, 1455 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) 7.40-7.32 (m, 3H), 7.18-7.15 (m, 2H), 5.28 (s, 1H), 4.21 (d, 1H, $J=3.7$), 2.91 (d, 1H, $J=3.7$), 1.26 (s, 3H), 0.81 (s, 3H); ^{13}C NMR (100 MHz, CHCl_3) 177.3, 135.8, 128.7, 126.0, 88.7, 75.0, 44.0, 21.9, 20.9; HRMS (EI): Exact mass calcd for $\text{C}_{12}\text{H}_{14}\text{O}_3$ $[\text{M}]^+$, 206.0943. Found 206.0942.

¹ Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518-1520.

² Perrin, D. D. and Armarego, W. L. F. *Purification of Laboratory Chemicals*; 3rd ed., Pergamon Press, Oxford, 1988.

³ Manzer, L. E. *Inorg. Synth.* **1982**, *21*, 139-140.

⁴ Evans, D. A.; Chapman, K. T.; Carreira, E. M. *J. Am. Chem. Soc.* **1988**, *110*, 3560-3578.

⁵ See preceeding article in this issue for full characterization of compounds.

X-ray crystallographic data for entry 5, table 2.

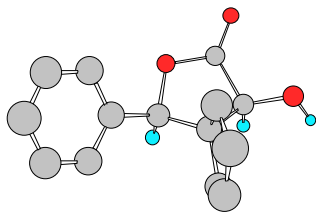


Table 1. Crystal data and structure refinement for JWU2t.

Identification code	jwu2t
Empirical formula	C ₁₄ H ₁₆ O ₃
Formula weight	232.27
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	P2(1)2(1)2(1)
Unit cell dimensions	a = 6.1337(8) Å = 90°.
	b = 7.5487(10) Å = 90°.
	c = 25.784(3) Å = 90°.
Volume	1193.9(3) Å ³
Z	4
Density (calculated)	1.292 Mg/m ³
Absorption coefficient	0.090 mm ⁻¹
F(000)	496
Crystal size	.8 x .8 x 6.5 mm ³
Theta range for data collection	1.58 to 28.29°.
Index ranges	-7<= <i>h</i> <=8, -10<= <i>k</i> <=9, -27<= <i>l</i> <=33
Reflections collected	8021
Independent reflections	2880 [R(int) = 0.0967]
Completeness to theta = 28.29°	99.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	2880 / 0 / 155
Goodness-of-fit on F ²	1.129
Final R indices [I>2sigma(I)]	R1 = 0.0802, wR2 = 0.1823

R indices (all data)	R1 = 0.0889, wR2 = 0.1871
Absolute structure parameter	-2(2)
Largest diff. peak and hole	0.310 and -0.309 e. Å ⁻³

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters Å² $\times 10^3$) for JWU2t. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	-2542(4)	3549(3)	9259(1)	36(1)
C(2)	2588(5)	1400(4)	8140(1)	26(1)
O(3)	125(4)	3015(3)	8699(1)	29(1)
C(4)	2166(4)	1987(4)	8685(1)	24(1)
O(2)	-453(4)	936(3)	9912(1)	37(1)
C(6)	3984(6)	-229(4)	9328(1)	36(1)
C(7)	417(5)	-1008(4)	8948(1)	32(1)
C(8)	4675(5)	830(4)	8003(1)	35(1)
C(9)	3508(7)	309(5)	7133(1)	43(1)
C(10)	-816(5)	2871(4)	9168(1)	27(1)
C(11)	5114(6)	252(5)	7504(2)	42(1)
C(12)	976(5)	1441(4)	7762(1)	30(1)
C(13)	677(5)	1784(4)	9512(1)	26(1)
C(14)	1435(6)	898(5)	7258(1)	38(1)
C(15)	1847(5)	585(4)	9119(1)	23(1)
C(16)	1733(11)	-2618(6)	9070(4)	127(4)
C(17)	3849(10)	-2165(6)	9219(3)	90(2)

Table 2. Bond lengths [Å] and angles [°] for JWU2t.

O(1)-C(10)	1.200(4)
C(2)-C(12)	1.388(4)
C(2)-C(8)	1.396(5)
C(2)-C(4)	1.496(4)
O(3)-C(10)	1.344(4)
O(3)-C(4)	1.473(4)

C(4)-C(15)	1.551(4)
O(2)-C(13)	1.396(4)
C(6)-C(17)	1.490(6)
C(6)-C(15)	1.545(4)
C(7)-C(16)	1.493(6)
C(7)-C(15)	1.552(4)
C(8)-C(11)	1.385(5)
C(9)-C(11)	1.374(6)
C(9)-C(14)	1.385(5)
C(10)-C(13)	1.517(4)
C(12)-C(14)	1.392(5)
C(13)-C(15)	1.538(4)
C(16)-C(17)	1.396(8)

C(12)-C(2)-C(8)	118.8(3)
C(12)-C(2)-C(4)	122.0(3)
C(8)-C(2)-C(4)	119.2(3)
C(10)-O(3)-C(4)	110.1(2)
O(3)-C(4)-C(2)	109.0(2)
O(3)-C(4)-C(15)	103.6(2)
C(2)-C(4)-C(15)	119.7(2)
C(17)-C(6)-C(15)	106.1(3)
C(16)-C(7)-C(15)	105.4(3)
C(11)-C(8)-C(2)	120.7(3)
C(11)-C(9)-C(14)	120.4(3)
O(1)-C(10)-O(3)	121.5(3)
O(1)-C(10)-C(13)	130.4(3)
O(3)-C(10)-C(13)	108.2(3)
C(9)-C(11)-C(8)	119.8(3)
C(2)-C(12)-C(14)	120.3(3)
O(2)-C(13)-C(10)	112.4(3)
O(2)-C(13)-C(15)	116.6(2)
C(10)-C(13)-C(15)	102.3(2)
C(9)-C(14)-C(12)	119.8(3)
C(13)-C(15)-C(6)	113.6(2)

C(13)-C(15)-C(4)	97.6(2)
C(6)-C(15)-C(4)	114.6(2)
C(13)-C(15)-C(7)	112.3(2)
C(6)-C(15)-C(7)	105.7(2)
C(4)-C(15)-C(7)	113.3(2)
C(17)-C(16)-C(7)	111.2(4)
C(16)-C(17)-C(6)	110.1(4)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for JWU2t. The anisotropic displacement factor exponent takes the form: $-2^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	39(1)	36(1)	33(1)	3(1)	4(1)	8(1)
C(2)	31(2)	18(1)	29(2)	1(1)	4(1)	-4(1)
O(3)	39(1)	24(1)	23(1)	0(1)	0(1)	5(1)
C(4)	25(1)	20(1)	26(1)	-1(1)	-1(1)	-5(1)
O(2)	45(1)	44(1)	21(1)	6(1)	1(1)	7(1)
C(6)	34(2)	36(2)	38(2)	4(1)	-8(1)	6(1)
C(7)	39(2)	27(1)	30(2)	-2(1)	-1(1)	-9(1)
C(8)	35(2)	35(2)	36(2)	-3(1)	-1(1)	2(1)
C(9)	63(2)	37(2)	28(2)	-11(1)	17(2)	-5(2)
C(10)	36(2)	21(1)	24(1)	-2(1)	1(1)	-7(1)
C(11)	37(2)	43(2)	46(2)	-13(2)	12(2)	2(2)
C(12)	32(2)	30(1)	27(2)	-2(1)	1(1)	-1(1)
C(13)	31(2)	27(1)	21(1)	-4(1)	-4(1)	1(1)
C(14)	51(2)	36(2)	27(2)	-4(1)	-4(2)	-8(2)
C(15)	30(1)	21(1)	19(1)	-1(1)	-3(1)	-4(1)
C(16)	95(4)	25(2)	262(11)	-23(4)	-83(6)	1(2)
C(17)	88(4)	38(2)	145(6)	-2(3)	-51(4)	21(3)

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

for JWU2t.

	x	y	z	U(eq)
<hr/>				
H(4)	3359	2773	8790	28
H(2)	349	829	10164	55
H(6A)	5235	286	9154	43
H(6B)	4119	-19	9698	43
H(7A)	-949	-1020	9137	38
H(7B)	104	-947	8580	38
H(8)	5782	838	8250	42
H(9)	3815	-50	6796	51
H(11)	6493	-173	7420	50
H(12)	-417	1834	7846	36
H(13)	1756	2581	9668	31
H(14)	353	930	7006	46
H(16A)	1035	-3272	9349	153
H(16B)	1790	-3380	8768	153
H(17A)	4861	-2472	8944	108
H(17B)	4253	-2826	9527	108

Table 6. Torsion angles [°] for JWU2t.

C(10)-O(3)-C(4)-C(2)	151.9(2)
C(10)-O(3)-C(4)-C(15)	23.3(3)
C(12)-C(2)-C(4)-O(3)	-15.7(4)
C(8)-C(2)-C(4)-O(3)	162.9(3)
C(12)-C(2)-C(4)-C(15)	103.2(3)
C(8)-C(2)-C(4)-C(15)	-78.2(4)
C(12)-C(2)-C(8)-C(11)	-2.4(5)
C(4)-C(2)-C(8)-C(11)	179.0(3)
C(4)-O(3)-C(10)-O(1)	-177.1(3)
C(4)-O(3)-C(10)-C(13)	3.4(3)
C(14)-C(9)-C(11)-C(8)	-1.7(6)

C(2)-C(8)-C(11)-C(9)	2.8(5)
C(8)-C(2)-C(12)-C(14)	0.9(4)
C(4)-C(2)-C(12)-C(14)	179.4(3)
O(1)-C(10)-C(13)-O(2)	25.8(4)
O(3)-C(10)-C(13)-O(2)	-154.7(2)
O(1)-C(10)-C(13)-C(15)	151.7(3)
O(3)-C(10)-C(13)-C(15)	-28.9(3)
C(11)-C(9)-C(14)-C(12)	0.2(6)
C(2)-C(12)-C(14)-C(9)	0.1(5)
O(2)-C(13)-C(15)-C(6)	-76.0(3)
C(10)-C(13)-C(15)-C(6)	161.0(2)
O(2)-C(13)-C(15)-C(4)	162.9(2)
C(10)-C(13)-C(15)-C(4)	39.9(3)
O(2)-C(13)-C(15)-C(7)	43.8(3)
C(10)-C(13)-C(15)-C(7)	-79.2(3)
C(17)-C(6)-C(15)-C(13)	129.9(4)
C(17)-C(6)-C(15)-C(4)	-119.1(4)
C(17)-C(6)-C(15)-C(7)	6.4(5)
O(3)-C(4)-C(15)-C(13)	-38.5(2)
C(2)-C(4)-C(15)-C(13)	-160.1(3)
O(3)-C(4)-C(15)-C(6)	-158.9(2)
C(2)-C(4)-C(15)-C(6)	79.6(3)
O(3)-C(4)-C(15)-C(7)	79.8(3)
C(2)-C(4)-C(15)-C(7)	-41.8(4)
C(16)-C(7)-C(15)-C(13)	-123.4(5)
C(16)-C(7)-C(15)-C(6)	0.9(5)
C(16)-C(7)-C(15)-C(4)	127.2(5)
C(15)-C(7)-C(16)-C(17)	-8.6(9)
C(7)-C(16)-C(17)-C(6)	13.2(10)
C(15)-C(6)-C(17)-C(16)	-12.1(8)

Symmetry transformations used to generate equivalent atoms: