Enantioselective Synthesis of (-)-Sclerophytin A by a Stereoconverging Epoxide Hydrolysis

Bin Wang, Armando P. Ramirez, Justin Slade and James P. Morken *

Department of Chemistry, Merkert Chemistry Center, Boston College, Chestnut Hill, MA 02467

Supporting Information

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- **X-ray crystal structure of racemic 6:**

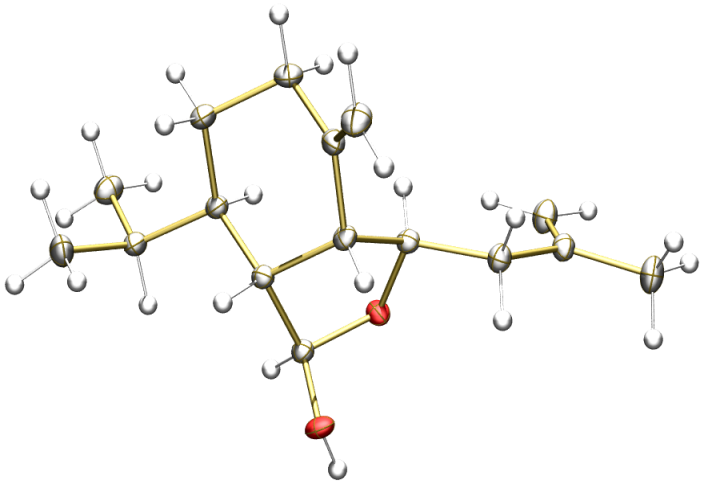

Table 1. Crystal data and structure refinement for C16H26O2.

Identification code	C16H26O2	
Empirical formula	C16 H26 O2	
Formula weight	250.37	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/n 1	
Unit cell dimensions	a = 20.398(7) Å	α= 90°.
	b = 5.6912(19) Å	β= 112.062(4)°.
	c = 26.770(9) Å	γ = 90°.
Volume	2880.2(17) $Å^3$	
Z	8	
Density (calculated)	1.155 Mg/m^3	
Absorption coefficient	0.074 mm^{-1}	
F(000)	1104	
Crystal size	0.27 x 0.05 x 0.03 mm^3	
Theta range for data collection	1.64 to 28.33°.	
Index ranges	-26<=h<=27, -7<=k<=7, -35<=l<=34	
Reflections collected	33322	
Independent reflections	7103 [R(int) = 0.0347]	
Completeness to theta = 28.33°	98.8 %	
Absorption correction	Semi-empirical from equivalents	

Max. and min. transmission	0.9978 and 0.9804
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7103 / 52 / 483
Goodness-of-fit on F^2	1.035
Final R indices [I>2sigma(I)]	R1 = 0.0463, wR2 = 0.1126
R indices (all data)	R1 = 0.0579, wR2 = 0.1209
Extinction coefficient	na
Largest diff. peak and hole	0.496 and -0.206 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for C16H26O2. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	3491(1)	-2359(1)	723(1)	16(1)
O(2)	3093(1)	1528(1)	665(1)	18(1)
C(1)	2892(1)	-842(2)	616(1)	14(1)
C(2)	2423(1)	-1321(2)	30(1)	14(1)
C(3)	1979(1)	-3570(2)	-39(1)	15(1)
C(4)	1717(1)	-4442(2)	-624(1)	18(1)
C(5)	2333(1)	-4892(2)	-807(1)	20(1)
C(6)	2741(1)	-2653(2)	-761(1)	17(1)
C(7)	2996(1)	-1523(2)	-215(1)	14(1)
C(8)	3583(1)	-2900(2)	224(1)	14(1)
C(9)	1367(1)	-3254(2)	162(1)	17(1)
C(10)	757(1)	-1772(2)	-216(1)	24(1)
C(11)	1082(1)	-5622(2)	257(1)	23(1)
C(12)	2826(1)	-1643(2)	-1180(1)	23(1)
C(13)	4317(1)	-2247(2)	260(1)	16(1)
C(14)	4917(1)	-3769(2)	613(1)	17(1)
C(15)	4830(1)	-5748(2)	840(1)	22(1)
C(16)	5638(1)	-2876(2)	680(1)	23(1)
O(3)	3976(1)	1702(1)	1759(1)	16(1)
O(4)	4368(1)	-2195(1)	1841(1)	19(1)
C(17)	4571(1)	171(2)	1868(1)	15(1)
C(18)	5058(1)	758(2)	2444(1)	14(1)

C(19)	5487(1)	3011(2)	2474(1)	15(1)
C(20)	5771(1)	3997(2)	3049(1)	19(1)
C(21)	5163(1)	4574(2)	3232(1)	20(1)
C(22)	4749(1)	2395(2)	3226(1)	18(1)
C(23)	4505(1)	1029(2)	2706(1)	15(1)
C(24)	3877(1)	2201(2)	2256(1)	14(1)
C(25)	6077(1)	2674(2)	2250(1)	18(1)
C(26)	6715(1)	1304(2)	2625(1)	24(1)
C(27)	6316(1)	5039(2)	2110(1)	24(1)
C(28)	4617(1)	1678(3)	3649(1)	27(1)
C(29)	3171(1)	1278(2)	2232(1)	17(1)
C(30)	2522(1)	2469(2)	1840(1)	19(1)
C(31)	2539(1)	4520(2)	1601(1)	24(1)
C(32)	1847(1)	1245(3)	1761(1)	34(1)

Table 3. Bond lengths [Å] and angles [°] for C16H26O2.

O(1)-C(1)	1.4347(13)
O(1)-C(8)	1.4509(13)
O(2)-C(1)	1.4012(14)
O(2)-H(2O)	0.866(13)
C(1)-C(2)	1.5242(15)
C(1)-H(1)	0.985(11)
C(2)-C(3)	1.5384(15)
C(2)-C(7)	1.5444(15)
C(2)-H(2)	0.987(11)
C(3)-C(4)	1.5350(16)
C(3)-C(9)	1.5424(15)
C(3)-H(3)	0.984(11)
C(4)-C(5)	1.5315(16)
C(4)-H(4A)	0.992(12)
C(4)-H(4B)	0.982(12)
C(5)-C(6)	1.5015(17)
C(5)-H(5A)	0.997(12)
C(5)-H(5B)	0.991(12)
C(6)-C(12)	1.3278(17)

C(6)-C(7)	1.5012(15)
C(7)-C(8)	1.5391(15)
C(7)-H(7)	0.988(11)
C(8)-C(13)	1.5108(16)
C(8)-H(8)	0.984(11)
C(9)-C(11)	1.5260(17)
C(9)-C(10)	1.5287(16)
C(9)-H(9)	1.001(12)
C(10)-H(10A)	0.983(13)
C(10)-H(10B)	0.985(13)
C(10)-H(10C)	0.999(13)
C(11)-H(11A)	0.980(12)
C(11)-H(11B)	0.986(13)
C(11)-H(11C)	0.985(13)
C(12)-H(12A)	0.986(12)
C(12)-H(12B)	0.967(12)
C(13)-C(14)	1.5074(15)
C(13)-H(13A)	0.980(12)
C(13)-H(13B)	0.981(11)
C(14)-C(15)	1.3230(17)
C(14)-C(16)	1.5005(16)
C(15)-H(15A)	0.979(12)
C(15)-H(15B)	0.986(12)
C(16)-H(16A)	0.981(13)
C(16)-H(16B)	0.978(13)
C(16)-H(16C)	0.988(13)
O(3)-C(17)	1.4311(13)
O(3)-C(24)	1.4485(13)
O(4)-C(17)	1.4025(14)
O(4)-H(4O)	0.876(13)
C(17)-C(18)	1.5238(15)
C(17)-H(17)	0.994(11)
C(18)-C(19)	1.5376(16)
C(18)-C(23)	1.5452(15)
C(18)-H(18)	0.992(11)
C(19)-C(20)	1.5332(16)

C(19)-C(25)	1.5436(16)
C(19)-H(19)	0.981(12)
C(20)-C(21)	1.5300(16)
C(20)-H(20A)	0.994(12)
C(20)-H(20B)	0.995(12)
C(21)-C(22)	1.4971(17)
C(21)-H(21A)	0.989(12)
C(21)-H(21B)	0.999(12)
C(22)-C(28)	1.3248(17)
C(22)-C(23)	1.5047(16)
C(23)-C(24)	1.5416(15)
C(23)-H(23)	0.989(12)
C(24)-C(29)	1.5122(16)
C(24)-H(24)	0.989(12)
C(25)-C(27)	1.5252(17)
C(25)-C(26)	1.5257(16)
C(25)-H(25)	1.002(12)
C(26)-H(26A)	0.976(13)
C(26)-H(26B)	0.994(13)
C(26)-H(26C)	0.979(13)
C(27)-H(27A)	0.979(13)
C(27)-H(27B)	0.998(13)
C(27)-H(27C)	0.981(13)
C(28)-H(28A)	0.974(12)
C(28)-H(28B)	0.978(13)
C(29)-C(30)	1.5064(16)
C(29)-H(29A)	0.973(12)
C(29)-H(29B)	0.984(12)
C(30)-C(31)	1.3378(18)
C(30)-C(32)	1.4846(18)
C(31)-H(31A)	0.965(12)
C(31)-H(31B)	0.982(12)
C(32)-H(32A)	0.985(14)
C(32)-H(32B)	1.045(14)
C(32)-H(32C)	0.973(14)

C(1)-O(1)-C(8)	109.74(8)
C(1)-O(2)-H(2O)	107.0(10)
O(2)-C(1)-O(1)	111.30(9)
O(2)-C(1)-C(2)	108.46(9)
O(1)-C(1)-C(2)	104.91(8)
O(2)-C(1)-H(1)	110.2(8)
O(1)-C(1)-H(1)	105.6(8)
C(2)-C(1)-H(1)	116.2(8)
C(1)-C(2)-C(3)	112.68(9)
C(1)-C(2)-C(7)	99.60(8)
C(3)-C(2)-C(7)	113.14(9)
C(1)-C(2)-H(2)	111.3(8)
C(3)-C(2)-H(2)	109.7(8)
C(7)-C(2)-H(2)	110.1(7)
C(4)-C(3)-C(2)	110.74(9)
C(4)-C(3)-C(9)	112.11(9)
C(2)-C(3)-C(9)	112.23(9)
C(4)-C(3)-H(3)	107.2(8)
C(2)-C(3)-H(3)	107.4(8)
C(9)-C(3)-H(3)	106.8(8)
C(5)-C(4)-C(3)	111.62(9)
C(5)-C(4)-H(4A)	108.4(8)
C(3)-C(4)-H(4A)	110.1(8)
C(5)-C(4)-H(4B)	108.9(8)
C(3)-C(4)-H(4B)	110.8(8)
H(4A)-C(4)-H(4B)	106.8(11)
C(6)-C(5)-C(4)	109.18(10)
C(6)-C(5)-H(5A)	111.1(8)
C(4)-C(5)-H(5A)	108.7(8)
C(6)-C(5)-H(5B)	110.3(8)
C(4)-C(5)-H(5B)	110.4(8)
H(5A)-C(5)-H(5B)	107.1(11)
C(12)-C(6)-C(7)	122.01(11)
C(12)-C(6)-C(5)	122.90(11)
C(7)-C(6)-C(5)	114.91(9)
C(6)-C(7)-C(8)	114.26(9)

C(6)-C(7)-C(2)	113.89(9)
C(8)-C(7)-C(2)	102.15(9)
C(6)-C(7)-H(7)	109.2(8)
C(8)-C(7)-H(7)	109.1(7)
C(2)-C(7)-H(7)	107.9(7)
O(1)-C(8)-C(13)	110.86(9)
O(1)-C(8)-C(7)	105.22(8)
C(13)-C(8)-C(7)	113.10(9)
O(1)-C(8)-H(8)	105.5(8)
C(13)-C(8)-H(8)	111.4(8)
C(7)-C(8)-H(8)	110.3(8)
C(11)-C(9)-C(10)	109.30(10)
C(11)-C(9)-C(3)	111.30(9)
C(10)-C(9)-C(3)	113.45(10)
C(11)-C(9)-H(9)	108.3(8)
C(10)-C(9)-H(9)	107.7(8)
C(3)-C(9)-H(9)	106.6(8)
C(9)-C(10)-H(10A)	111.0(9)
C(9)-C(10)-H(10B)	110.5(10)
H(10A)-C(10)-H(10B)	107.6(13)
C(9)-C(10)-H(10C)	113.0(9)
H(10A)-C(10)-H(10C)	107.8(13)
H(10B)-C(10)-H(10C)	106.6(13)
C(9)-C(11)-H(11A)	110.4(9)
C(9)-C(11)-H(11B)	111.6(9)
H(11A)-C(11)-H(11B)	108.1(13)
C(9)-C(11)-H(11C)	110.6(9)
H(11A)-C(11)-H(11C)	108.3(13)
H(11B)-C(11)-H(11C)	107.6(13)
C(6)-C(12)-H(12A)	121.8(9)
C(6)-C(12)-H(12B)	122.0(9)
H(12A)-C(12)-H(12B)	116.2(13)
C(14)-C(13)-C(8)	116.84(10)
C(14)-C(13)-H(13A)	110.5(8)
C(8)-C(13)-H(13A)	107.0(8)
C(14)-C(13)-H(13B)	108.9(8)

C(8)-C(13)-H(13B)	109.0(8)
H(13A)-C(13)-H(13B)	103.8(11)
C(15)-C(14)-C(16)	121.82(11)
C(15)-C(14)-C(13)	123.99(10)
C(16)-C(14)-C(13)	114.17(10)
C(14)-C(15)-H(15A)	123.4(9)
C(14)-C(15)-H(15B)	119.3(8)
H(15A)-C(15)-H(15B)	117.4(12)
C(14)-C(16)-H(16A)	111.0(9)
C(14)-C(16)-H(16B)	109.3(9)
H(16A)-C(16)-H(16B)	109.7(12)
C(14)-C(16)-H(16C)	111.0(9)
H(16A)-C(16)-H(16C)	109.6(13)
H(16B)-C(16)-H(16C)	106.2(13)
C(17)-O(3)-C(24)	109.33(8)
C(17)-O(4)-H(4O)	106.5(11)
O(4)-C(17)-O(3)	111.32(9)
O(4)-C(17)-C(18)	109.31(9)
O(3)-C(17)-C(18)	104.73(8)
O(4)-C(17)-H(17)	110.7(8)
O(3)-C(17)-H(17)	104.7(8)
C(18)-C(17)-H(17)	115.9(8)
C(17)-C(18)-C(19)	112.22(9)
C(17)-C(18)-C(23)	99.90(9)
C(19)-C(18)-C(23)	113.07(9)
C(17)-C(18)-H(18)	111.3(8)
C(19)-C(18)-H(18)	109.5(8)
C(23)-C(18)-H(18)	110.6(8)
C(20)-C(19)-C(18)	110.53(9)
C(20)-C(19)-C(25)	112.32(9)
C(18)-C(19)-C(25)	112.80(9)
C(20)-C(19)-H(19)	107.8(8)
C(18)-C(19)-H(19)	107.3(8)
C(25)-C(19)-H(19)	105.7(8)
C(21)-C(20)-C(19)	110.74(9)
C(21)-C(20)-H(20A)	110.1(8)

C(19)-C(20)-H(20A)	110.3(8)
C(21)-C(20)-H(20B)	110.3(8)
C(19)-C(20)-H(20B)	109.2(8)
H(20A)-C(20)-H(20B)	106.1(11)
C(22)-C(21)-C(20)	109.89(10)
C(22)-C(21)-H(21A)	111.1(8)
C(20)-C(21)-H(21A)	111.0(8)
C(22)-C(21)-H(21B)	108.2(8)
C(20)-C(21)-H(21B)	108.6(8)
H(21A)-C(21)-H(21B)	107.9(12)
C(28)-C(22)-C(21)	122.76(11)
C(28)-C(22)-C(23)	121.89(11)
C(21)-C(22)-C(23)	115.33(10)
C(22)-C(23)-C(24)	112.88(9)
C(22)-C(23)-C(18)	115.26(9)
C(24)-C(23)-C(18)	102.76(9)
C(22)-C(23)-H(23)	110.3(8)
C(24)-C(23)-H(23)	107.0(8)
C(18)-C(23)-H(23)	108.1(8)
O(3)-C(24)-C(29)	110.67(9)
O(3)-C(24)-C(23)	105.82(8)
C(29)-C(24)-C(23)	112.40(9)
O(3)-C(24)-H(24)	106.7(8)
C(29)-C(24)-H(24)	110.1(7)
C(23)-C(24)-H(24)	110.9(8)
C(27)-C(25)-C(26)	109.86(10)
C(27)-C(25)-C(19)	110.69(10)
C(26)-C(25)-C(19)	113.86(10)
C(27)-C(25)-H(25)	107.9(8)
C(26)-C(25)-H(25)	106.9(8)
C(19)-C(25)-H(25)	107.3(8)
C(25)-C(26)-H(26A)	110.3(9)
C(25)-C(26)-H(26B)	112.4(9)
H(26A)-C(26)-H(26B)	108.7(13)
C(25)-C(26)-H(26C)	110.2(10)
H(26A)-C(26)-H(26C)	108.6(13)

H(26B)-C(26)-H(26C)	106.6(13)
C(25)-C(27)-H(27A)	111.6(10)
C(25)-C(27)-H(27B)	110.5(9)
H(27A)-C(27)-H(27B)	106.9(13)
C(25)-C(27)-H(27C)	110.8(9)
H(27A)-C(27)-H(27C)	108.5(13)
H(27B)-C(27)-H(27C)	108.4(13)
C(22)-C(28)-H(28A)	120.6(9)
C(22)-C(28)-H(28B)	122.4(10)
H(28A)-C(28)-H(28B)	117.1(13)
C(30)-C(29)-C(24)	116.60(10)
C(30)-C(29)-H(29A)	109.0(8)
C(24)-C(29)-H(29A)	108.3(8)
C(30)-C(29)-H(29B)	109.4(8)
C(24)-C(29)-H(29B)	107.5(8)
H(29A)-C(29)-H(29B)	105.5(11)
C(31)-C(30)-C(32)	122.04(11)
C(31)-C(30)-C(29)	123.47(11)
C(32)-C(30)-C(29)	114.41(11)
C(30)-C(31)-H(31A)	123.2(9)
C(30)-C(31)-H(31B)	118.4(9)
H(31A)-C(31)-H(31B)	118.2(12)
C(30)-C(32)-H(32A)	113.0(11)
C(30)-C(32)-H(32B)	108.5(10)
H(32A)-C(32)-H(32B)	113.6(14)
C(30)-C(32)-H(32C)	105.2(11)
H(32A)-C(32)-H(32C)	105.1(16)
H(32B)-C(32)-H(32C)	111.1(15)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for C16H26O2. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	16(1)	20(1)	12(1)	0(1)	4(1)	3(1)

O(2)	20(1)	14(1)	14(1)	-2(1)	1(1)	-2(1)
C(1)	14(1)	14(1)	14(1)	-1(1)	4(1)	0(1)
C(2)	14(1)	14(1)	12(1)	0(1)	4(1)	1(1)
C(3)	14(1)	13(1)	15(1)	-1(1)	3(1)	0(1)
C(4)	16(1)	19(1)	17(1)	-4(1)	4(1)	-3(1)
C(5)	21(1)	21(1)	16(1)	-6(1)	6(1)	-1(1)
C(6)	14(1)	21(1)	14(1)	-2(1)	4(1)	2(1)
C(7)	13(1)	15(1)	13(1)	0(1)	3(1)	0(1)
C(8)	14(1)	15(1)	12(1)	0(1)	4(1)	0(1)
C(9)	15(1)	18(1)	17(1)	-4(1)	6(1)	-2(1)
C(10)	16(1)	22(1)	32(1)	-1(1)	7(1)	2(1)
C(11)	23(1)	23(1)	23(1)	-1(1)	10(1)	-6(1)
C(12)	21(1)	32(1)	16(1)	-1(1)	6(1)	-2(1)
C(13)	15(1)	16(1)	17(1)	2(1)	5(1)	0(1)
C(14)	13(1)	22(1)	15(1)	0(1)	4(1)	1(1)
C(15)	17(1)	24(1)	22(1)	5(1)	4(1)	1(1)
C(16)	14(1)	28(1)	25(1)	5(1)	6(1)	-1(1)
O(3)	16(1)	20(1)	11(1)	1(1)	4(1)	5(1)
O(4)	22(1)	16(1)	15(1)	-2(1)	2(1)	-1(1)
C(17)	15(1)	15(1)	14(1)	0(1)	4(1)	1(1)
C(18)	15(1)	14(1)	12(1)	0(1)	3(1)	2(1)
C(19)	15(1)	16(1)	14(1)	1(1)	4(1)	1(1)
C(20)	18(1)	20(1)	17(1)	-2(1)	4(1)	-2(1)
C(21)	23(1)	20(1)	17(1)	-4(1)	7(1)	0(1)
C(22)	16(1)	20(1)	14(1)	0(1)	3(1)	4(1)
C(23)	16(1)	15(1)	13(1)	2(1)	4(1)	1(1)
C(24)	15(1)	15(1)	11(1)	0(1)	4(1)	2(1)
C(25)	15(1)	22(1)	15(1)	-1(1)	4(1)	0(1)
C(26)	16(1)	31(1)	24(1)	1(1)	4(1)	6(1)
C(27)	22(1)	28(1)	23(1)	1(1)	9(1)	-5(1)
C(28)	28(1)	34(1)	16(1)	0(1)	8(1)	-3(1)
C(29)	17(1)	18(1)	17(1)	2(1)	6(1)	0(1)
C(30)	14(1)	24(1)	18(1)	0(1)	6(1)	3(1)
C(31)	19(1)	26(1)	23(1)	4(1)	4(1)	3(1)
C(32)	18(1)	32(1)	46(1)	7(1)	3(1)	0(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for C16H26O2.

	x	y	z	U(eq)
H(2O)	3326(7)	1800(30)	1004(5)	27
H(1)	2692(7)	-1250(20)	887(5)	17
H(2)	2109(6)	30(20)	-130(5)	16
H(3)	2291(7)	-4790(20)	188(5)	18
H(4A)	1398(7)	-3260(20)	-868(5)	22
H(4B)	1442(7)	-5900(20)	-666(5)	22
H(5A)	2639(7)	-6150(20)	-577(5)	23
H(5B)	2154(7)	-5470(20)	-1184(5)	23
H(7)	3168(6)	80(20)	-240(5)	17
H(8)	3499(7)	-4600(20)	170(5)	17
H(9)	1569(7)	-2410(20)	515(5)	20
H(10A)	421(8)	-1370(30)	-45(6)	35
H(10B)	496(8)	-2650(30)	-549(6)	35
H(10C)	918(8)	-280(20)	-327(6)	35
H(11A)	746(7)	-5400(30)	435(6)	34
H(11B)	1465(7)	-6660(30)	484(6)	34
H(11C)	837(8)	-6440(30)	-88(5)	34
H(12A)	2651(7)	-2380(30)	-1540(5)	28
H(12B)	3063(7)	-150(20)	-1150(6)	28
H(13A)	4395(7)	-600(20)	378(5)	20
H(13B)	4324(7)	-2210(20)	-104(5)	20
H(15A)	4366(7)	-6350(30)	809(6)	26
H(15B)	5249(7)	-6670(20)	1058(5)	26
H(16A)	6008(7)	-3970(30)	897(6)	34
H(16B)	5713(8)	-1340(20)	856(6)	34
H(16C)	5676(8)	-2630(30)	327(5)	34
H(4O)	4133(8)	-2510(30)	1500(5)	28
H(17)	4758(7)	540(20)	1584(5)	18
H(18)	5385(6)	-560(20)	2611(5)	17

H(19)	5163(7)	4180(20)	2239(5)	19
H(20A)	6061(7)	5420(20)	3070(5)	22
H(20B)	6095(7)	2830(20)	3296(5)	22
H(21A)	5342(7)	5310(20)	3593(5)	24
H(21B)	4841(7)	5720(20)	2973(5)	24
H(23)	4348(7)	-560(20)	2762(5)	18
H(24)	3895(7)	3930(20)	2298(5)	17
H(25)	5870(7)	1740(20)	1909(5)	21
H(26A)	7025(8)	880(30)	2437(6)	37
H(26B)	6576(8)	-150(30)	2766(6)	37
H(26C)	6984(8)	2260(30)	2939(6)	37
H(27A)	6641(8)	4850(30)	1921(6)	36
H(27B)	6575(8)	5950(30)	2445(6)	36
H(27C)	5909(7)	5970(30)	1882(6)	36
H(28A)	4368(7)	210(20)	3637(6)	25(4)
H(28B)	4759(8)	2580(30)	3984(6)	35(4)
H(29A)	3161(7)	1370(20)	2592(5)	21
H(29B)	3153(7)	-410(20)	2148(5)	21
H(31A)	2972(7)	5280(30)	1623(6)	28
H(31B)	2089(7)	5230(30)	1367(6)	28
H(32A)	1435(8)	1970(30)	1480(6)	52
H(32B)	1797(9)	1110(30)	2135(6)	52
H(32C)	1892(10)	-300(30)	1621(7)	52

Table 6. Torsion angles [°] for C16H26O2.

C(8)-O(1)-C(1)-O(2)	-92.17(10)
C(8)-O(1)-C(1)-C(2)	24.93(11)
O(2)-C(1)-C(2)-C(3)	-161.09(9)
O(1)-C(1)-C(2)-C(3)	79.88(10)
O(2)-C(1)-C(2)-C(7)	78.73(10)
O(1)-C(1)-C(2)-C(7)	-40.30(10)
C(1)-C(2)-C(3)-C(4)	-160.71(9)
C(7)-C(2)-C(3)-C(4)	-48.68(12)
C(1)-C(2)-C(3)-C(9)	73.15(11)

C(7)-C(2)-C(3)-C(9)	-174.81(9)
C(2)-C(3)-C(4)-C(5)	57.27(12)
C(9)-C(3)-C(4)-C(5)	-176.53(9)
C(3)-C(4)-C(5)-C(6)	-59.33(12)
C(4)-C(5)-C(6)-C(12)	-120.53(12)
C(4)-C(5)-C(6)-C(7)	54.58(12)
C(12)-C(6)-C(7)-C(8)	-115.69(13)
C(5)-C(6)-C(7)-C(8)	69.15(12)
C(12)-C(6)-C(7)-C(2)	127.44(12)
C(5)-C(6)-C(7)-C(2)	-47.72(13)
C(1)-C(2)-C(7)-C(6)	163.85(9)
C(3)-C(2)-C(7)-C(6)	44.00(12)
C(1)-C(2)-C(7)-C(8)	40.14(10)
C(3)-C(2)-C(7)-C(8)	-79.70(10)
C(1)-O(1)-C(8)-C(13)	124.20(9)
C(1)-O(1)-C(8)-C(7)	1.60(11)
C(6)-C(7)-C(8)-O(1)	-150.28(9)
C(2)-C(7)-C(8)-O(1)	-26.82(10)
C(6)-C(7)-C(8)-C(13)	88.58(12)
C(2)-C(7)-C(8)-C(13)	-147.97(9)
C(4)-C(3)-C(9)-C(11)	72.39(12)
C(2)-C(3)-C(9)-C(11)	-162.23(9)
C(4)-C(3)-C(9)-C(10)	-51.37(13)
C(2)-C(3)-C(9)-C(10)	74.01(12)
O(1)-C(8)-C(13)-C(14)	71.77(12)
C(7)-C(8)-C(13)-C(14)	-170.33(9)
C(8)-C(13)-C(14)-C(15)	7.57(17)
C(8)-C(13)-C(14)-C(16)	-173.55(10)
C(24)-O(3)-C(17)-O(4)	88.42(10)
C(24)-O(3)-C(17)-C(18)	-29.59(11)
O(4)-C(17)-C(18)-C(19)	161.63(9)
O(3)-C(17)-C(18)-C(19)	-79.01(11)
O(4)-C(17)-C(18)-C(23)	-78.31(10)
O(3)-C(17)-C(18)-C(23)	41.06(10)
C(17)-C(18)-C(19)-C(20)	161.00(9)
C(23)-C(18)-C(19)-C(20)	48.92(12)

C(17)-C(18)-C(19)-C(25)	-72.32(11)
C(23)-C(18)-C(19)-C(25)	175.60(8)
C(18)-C(19)-C(20)-C(21)	-59.66(12)
C(25)-C(19)-C(20)-C(21)	173.40(9)
C(19)-C(20)-C(21)-C(22)	60.48(12)
C(20)-C(21)-C(22)-C(28)	126.77(12)
C(20)-C(21)-C(22)-C(23)	-51.86(13)
C(28)-C(22)-C(23)-C(24)	106.43(13)
C(21)-C(22)-C(23)-C(24)	-74.92(12)
C(28)-C(22)-C(23)-C(18)	-135.91(12)
C(21)-C(22)-C(23)-C(18)	42.74(13)
C(17)-C(18)-C(23)-C(22)	-160.12(9)
C(19)-C(18)-C(23)-C(22)	-40.68(12)
C(17)-C(18)-C(23)-C(24)	-36.92(10)
C(19)-C(18)-C(23)-C(24)	82.53(10)
C(17)-O(3)-C(24)-C(29)	-117.03(10)
C(17)-O(3)-C(24)-C(23)	5.00(11)
C(22)-C(23)-C(24)-O(3)	145.72(9)
C(18)-C(23)-C(24)-O(3)	20.94(10)
C(22)-C(23)-C(24)-C(29)	-93.38(12)
C(18)-C(23)-C(24)-C(29)	141.84(9)
C(20)-C(19)-C(25)-C(27)	-73.50(12)
C(18)-C(19)-C(25)-C(27)	160.78(9)
C(20)-C(19)-C(25)-C(26)	50.84(13)
C(18)-C(19)-C(25)-C(26)	-74.88(12)
O(3)-C(24)-C(29)-C(30)	-67.49(13)
C(23)-C(24)-C(29)-C(30)	174.44(9)
C(24)-C(29)-C(30)-C(31)	-13.56(17)
C(24)-C(29)-C(30)-C(32)	169.73(11)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for C16H26O2 [Å and °].

D-H…A	d(D-H)	d(H…A)	d(D…A)	<(DHA)

O(2)-H(2O)...O(3)	0.866(13)	1.958(13)	2.8053(13)	165.9(14)
O(4)-H(4O)...O(1)	0.876(13)	1.999(13)	2.8534(14)	164.6(15)

Symmetry transformations used to generate equivalent atoms:

- **X-ray crystal structure of racemic α-9:**

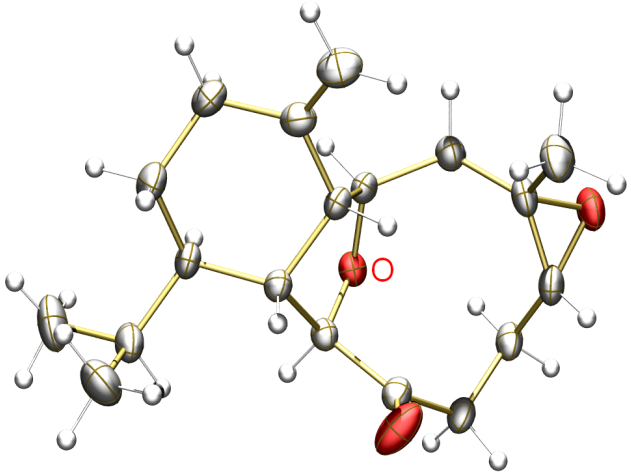

Table 8. Crystal data and structure refinement for sad.

Identification code	C19H28O3	
Empirical formula	C19 H28 O3	
Formula weight	304.41	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 11.642(6) Å	α= 90°.
	b = 7.337(4) Å	β= 104.046(7)°.
	c = 20.650(10) Å	γ = 90°.
Volume	1711.1(14) $Å^3$	
Z	4	
Density (calculated)	1.182 Mg/m^3	
Absorption coefficient	0.078 mm^{-1}	
F(000)	664	
Crystal size	0.18 x 0.03 x 0.02 mm^3	
Theta range for data collection	2.37 to 27.00°.	
Index ranges	-14<=h<=14, -9<=k<=9, -26<=l<=26	
Reflections collected	18626	
Independent reflections	3743 [R(int) = 0.0794]	
Completeness to theta = 27.00°	99.9 %	

Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9984 and 0.9861
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3743 / 28 / 283
Goodness-of-fit on F^2	1.042
Final R indices [I>2sigma(I)]	R1 = 0.0634, wR2 = 0.1085
R indices (all data)	R1 = 0.1139, wR2 = 0.1245
Extinction coefficient	na
Largest diff. peak and hole	0.259 and -0.200 e.Å^{-3}

Table 9. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for sad. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	8519(1)	-770(2)	9629(1)	38(1)
O(2)	7455(1)	1925(2)	7883(1)	26(1)
O(3)	10133(1)	429(3)	7605(1)	52(1)
C(1)	9216(2)	-214(3)	9173(1)	31(1)
C(2)	8151(2)	-1333(3)	8934(1)	30(1)
C(3)	6974(2)	-525(3)	8572(1)	32(1)
C(4)	6828(2)	218(3)	7867(1)	26(1)
C(5)	7244(2)	-998(3)	7367(1)	25(1)
C(6)	6306(2)	-2278(3)	7000(1)	31(1)
C(7)	5353(2)	-1410(4)	6467(1)	40(1)
C(8)	5899(2)	-305(4)	5996(1)	38(1)
C(9)	6705(2)	1205(3)	6362(1)	28(1)
C(10)	7690(2)	400(3)	6927(1)	24(1)
C(11)	8237(2)	1797(3)	7451(1)	24(1)
C(12)	9493(2)	1353(3)	7848(1)	27(1)
C(13)	9955(2)	2221(3)	8527(1)	31(1)
C(14)	9318(2)	1791(3)	9074(1)	31(1)
C(15)	8317(3)	-3350(4)	8861(1)	44(1)
C(16)	6340(2)	-4038(4)	7124(1)	41(1)
C(17)	7208(2)	2423(3)	5891(1)	38(1)
C(18)	6256(3)	3625(4)	5457(1)	56(1)

C(19)	7852(3)	1381(5)	5447(1)	54(1)

Table 10. Bond lengths [Å] and angles [°] for sad.

O(1)-C(1)	1.443(3)
O(1)-C(2)	1.455(3)
O(2)-C(11)	1.424(2)
O(2)-C(4)	1.446(3)
O(3)-C(12)	1.203(3)
C(1)-C(2)	1.469(3)
C(1)-C(14)	1.494(3)
C(1)-H(1)	0.969(15)
C(2)-C(15)	1.505(3)
C(2)-C(3)	1.513(3)
C(3)-C(4)	1.524(3)
C(3)-H(3A)	0.993(15)
C(3)-H(3B)	0.997(15)
C(4)-C(5)	1.529(3)
C(4)-H(4)	0.995(15)
C(5)-C(6)	1.499(3)
C(5)-C(10)	1.542(3)
C(5)-H(5)	0.987(15)
C(6)-C(16)	1.315(3)
C(6)-C(7)	1.503(3)
C(7)-C(8)	1.519(3)
C(7)-H(7A)	0.982(16)
C(7)-H(7B)	0.988(16)
C(8)-C(9)	1.528(3)
C(8)-H(8A)	0.993(16)
C(8)-H(8B)	1.000(15)
C(9)-C(17)	1.538(3)
C(9)-C(10)	1.540(3)
C(9)-H(9)	0.988(15)
C(10)-C(11)	1.514(3)
C(10)-H(10)	0.967(15)

C(11)-C(12)	1.529(3)
C(11)-H(11)	1.014(15)
C(12)-C(13)	1.514(3)
C(13)-C(14)	1.529(3)
C(13)-H(13A)	0.992(15)
C(13)-H(13B)	1.003(15)
C(14)-H(14A)	0.978(15)
C(14)-H(14B)	0.968(15)
C(15)-H(15A)	0.973(17)
C(15)-H(15B)	0.996(17)
C(15)-H(15C)	0.978(16)
C(16)-H(16A)	0.951(16)
C(16)-H(16B)	0.969(16)
C(17)-C(19)	1.523(4)
C(17)-C(18)	1.525(4)
C(17)-H(17)	0.973(16)
C(18)-H(18A)	0.981(17)
C(18)-H(18B)	0.978(17)
C(18)-H(18C)	0.984(17)
C(19)-H(19A)	0.981(17)
C(19)-H(19B)	0.993(17)
C(19)-H(19C)	0.994(17)

C(1)-O(1)-C(2)	60.94(14)
C(11)-O(2)-C(4)	108.90(15)
O(1)-C(1)-C(2)	59.95(14)
O(1)-C(1)-C(14)	116.36(19)
C(2)-C(1)-C(14)	126.4(2)
O(1)-C(1)-H(1)	112.9(13)
C(2)-C(1)-H(1)	116.0(13)
C(14)-C(1)-H(1)	113.4(13)
O(1)-C(2)-C(1)	59.12(13)
O(1)-C(2)-C(15)	111.35(18)
C(1)-C(2)-C(15)	117.7(2)
O(1)-C(2)-C(3)	113.01(18)
C(1)-C(2)-C(3)	122.4(2)

C(15)-C(2)-C(3)	117.3(2)
C(2)-C(3)-C(4)	119.28(19)
C(2)-C(3)-H(3A)	109.4(13)
C(4)-C(3)-H(3A)	107.6(13)
C(2)-C(3)-H(3B)	108.4(13)
C(4)-C(3)-H(3B)	106.6(12)
H(3A)-C(3)-H(3B)	104.6(18)
O(2)-C(4)-C(3)	110.44(17)
O(2)-C(4)-C(5)	106.30(16)
C(3)-C(4)-C(5)	117.10(19)
O(2)-C(4)-H(4)	106.0(12)
C(3)-C(4)-H(4)	107.6(12)
C(5)-C(4)-H(4)	108.8(12)
C(6)-C(5)-C(4)	113.33(18)
C(6)-C(5)-C(10)	115.24(18)
C(4)-C(5)-C(10)	102.53(17)
C(6)-C(5)-H(5)	110.8(12)
C(4)-C(5)-H(5)	109.6(12)
C(10)-C(5)-H(5)	104.7(12)
C(16)-C(6)-C(5)	122.2(2)
C(16)-C(6)-C(7)	122.7(2)
C(5)-C(6)-C(7)	115.0(2)
C(6)-C(7)-C(8)	110.2(2)
C(6)-C(7)-H(7A)	110.4(14)
C(8)-C(7)-H(7A)	108.8(14)
C(6)-C(7)-H(7B)	109.2(14)
C(8)-C(7)-H(7B)	110.6(13)
H(7A)-C(7)-H(7B)	108(2)
C(7)-C(8)-C(9)	111.84(19)
C(7)-C(8)-H(8A)	109.7(13)
C(9)-C(8)-H(8A)	107.7(14)
C(7)-C(8)-H(8B)	108.3(14)
C(9)-C(8)-H(8B)	109.3(14)
H(8A)-C(8)-H(8B)	110.0(18)
C(8)-C(9)-C(17)	112.95(18)
C(8)-C(9)-C(10)	110.56(19)

C(17)-C(9)-C(10)	111.95(18)
C(8)-C(9)-H(9)	106.9(13)
C(17)-C(9)-H(9)	107.9(13)
C(10)-C(9)-H(9)	106.1(12)
C(11)-C(10)-C(9)	112.82(18)
C(11)-C(10)-C(5)	99.91(16)
C(9)-C(10)-C(5)	113.97(17)
C(11)-C(10)-H(10)	108.5(12)
C(9)-C(10)-H(10)	110.9(12)
C(5)-C(10)-H(10)	110.2(12)
O(2)-C(11)-C(10)	105.71(17)
O(2)-C(11)-C(12)	110.54(16)
C(10)-C(11)-C(12)	114.89(18)
O(2)-C(11)-H(11)	106.1(11)
C(10)-C(11)-H(11)	113.1(11)
C(12)-C(11)-H(11)	106.2(11)
O(3)-C(12)-C(13)	120.4(2)
O(3)-C(12)-C(11)	120.30(19)
C(13)-C(12)-C(11)	119.08(19)
C(12)-C(13)-C(14)	118.04(19)
C(12)-C(13)-H(13A)	105.3(13)
C(14)-C(13)-H(13A)	107.9(12)
C(12)-C(13)-H(13B)	105.0(12)
C(14)-C(13)-H(13B)	111.9(12)
H(13A)-C(13)-H(13B)	108.2(18)
C(1)-C(14)-C(13)	111.94(19)
C(1)-C(14)-H(14A)	107.2(13)
C(13)-C(14)-H(14A)	106.5(13)
C(1)-C(14)-H(14B)	109.2(14)
C(13)-C(14)-H(14B)	112.2(13)
H(14A)-C(14)-H(14B)	109.6(19)
C(2)-C(15)-H(15A)	108.7(16)
C(2)-C(15)-H(15B)	108.6(16)
H(15A)-C(15)-H(15B)	108(2)
C(2)-C(15)-H(15C)	113.4(16)
H(15A)-C(15)-H(15C)	108(2)

H(15B)-C(15)-H(15C)	110(2)
C(6)-C(16)-H(16A)	123.5(15)
C(6)-C(16)-H(16B)	119.9(14)
H(16A)-C(16)-H(16B)	116(2)
C(19)-C(17)-C(18)	109.3(2)
C(19)-C(17)-C(9)	114.1(2)
C(18)-C(17)-C(9)	111.9(2)
C(19)-C(17)-H(17)	107.1(14)
C(18)-C(17)-H(17)	108.2(14)
C(9)-C(17)-H(17)	105.8(13)
C(17)-C(18)-H(18A)	110.2(18)
C(17)-C(18)-H(18B)	107.3(18)
H(18A)-C(18)-H(18B)	109(2)
C(17)-C(18)-H(18C)	113.1(17)
H(18A)-C(18)-H(18C)	108(3)
H(18B)-C(18)-H(18C)	109(3)
C(17)-C(19)-H(19A)	111.6(18)
C(17)-C(19)-H(19B)	106.0(17)
H(19A)-C(19)-H(19B)	109(2)
C(17)-C(19)-H(19C)	112.0(17)
H(19A)-C(19)-H(19C)	111(2)
H(19B)-C(19)-H(19C)	107(3)

Symmetry transformations used to generate equivalent atoms:

Table 11. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for sad. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + \ldots + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	49(1)	43(1)	21(1)	5(1)	4(1)	1(1)
O(2)	30(1)	26(1)	23(1)	1(1)	6(1)	3(1)
O(3)	30(1)	67(1)	52(1)	-26(1)	-1(1)	11(1)
C(1)	32(1)	35(1)	22(1)	5(1)	2(1)	8(1)
C(2)	40(1)	29(1)	20(1)	6(1)	5(1)	4(1)
C(3)	30(1)	39(2)	26(1)	5(1)	6(1)	0(1)

C(4)	20(1)	31(1)	25(1)	4(1)	2(1)	2(1)
C(5)	20(1)	28(1)	24(1)	2(1)	1(1)	2(1)
C(6)	29(1)	34(1)	31(1)	-4(1)	9(1)	-5(1)
C(7)	32(1)	48(2)	34(1)	1(1)	-2(1)	-11(1)
C(8)	37(2)	46(2)	25(1)	1(1)	-4(1)	-8(1)
C(9)	27(1)	34(1)	20(1)	2(1)	-1(1)	0(1)
C(10)	23(1)	28(1)	20(1)	-1(1)	4(1)	-1(1)
C(11)	27(1)	24(1)	20(1)	0(1)	5(1)	0(1)
C(12)	26(1)	27(1)	26(1)	-1(1)	2(1)	-2(1)
C(13)	30(1)	32(1)	25(1)	0(1)	-3(1)	-3(1)
C(14)	37(1)	32(1)	21(1)	0(1)	-1(1)	-1(1)
C(15)	53(2)	33(2)	40(2)	8(1)	2(1)	3(1)
C(16)	40(2)	36(2)	47(2)	-9(1)	10(1)	-6(1)
C(17)	44(2)	42(2)	23(1)	3(1)	0(1)	-9(1)
C(18)	78(2)	55(2)	31(2)	15(1)	4(1)	8(2)
C(19)	56(2)	69(2)	43(2)	16(2)	21(1)	0(2)

Table 12. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for sad.

	x	y	z	U(eq)
H(1)	9966(15)	-850(30)	9247(10)	37
H(3A)	6751(19)	460(30)	8847(10)	38
H(3B)	6350(17)	-1470(30)	8544(10)	38
H(4)	5975(14)	510(30)	7687(9)	31
H(5)	7960(15)	-1670(30)	7598(9)	30
H(7A)	4864(19)	-600(30)	6668(11)	48
H(7B)	4830(18)	-2370(30)	6220(10)	48
H(8A)	5264(17)	280(30)	5650(10)	46
H(8B)	6371(19)	-1150(30)	5781(11)	46
H(9)	6216(17)	1980(30)	6582(10)	34
H(10)	8311(15)	-130(30)	6750(9)	29
H(11)	8274(17)	3060(20)	7260(9)	29

H(13A)	9902(19)	3550(20)	8445(10)	37
H(13B)	10814(14)	1880(30)	8662(10)	37
H(14A)	9811(17)	2290(30)	9489(9)	37
H(14B)	8537(15)	2330(30)	8986(10)	37
H(15A)	7638(19)	-3980(30)	8951(13)	65
H(15B)	9036(18)	-3740(30)	9202(11)	65
H(15C)	8390(20)	-3700(40)	8415(9)	65
H(16A)	6974(18)	-4620(30)	7432(10)	49
H(16B)	5737(18)	-4830(30)	6862(10)	49
H(17)	7788(18)	3210(30)	6178(10)	45
H(18A)	5700(20)	2880(40)	5129(12)	85
H(18B)	6660(20)	4480(40)	5221(13)	85
H(18C)	5800(20)	4330(40)	5716(13)	85
H(19A)	8210(20)	2210(30)	5181(13)	82
H(19B)	7240(20)	630(40)	5142(12)	82
H(19C)	8450(20)	530(30)	5708(13)	82

Table 13. Torsion angles [°] for sad.

C(2)-O(1)-C(1)-C(14)	118.6(2)
C(1)-O(1)-C(2)-C(15)	110.4(2)
C(1)-O(1)-C(2)-C(3)	-115.1(2)
C(14)-C(1)-C(2)-O(1)	-102.2(2)
O(1)-C(1)-C(2)-C(15)	-99.5(2)
C(14)-C(1)-C(2)-C(15)	158.3(2)
O(1)-C(1)-C(2)-C(3)	99.2(2)
C(14)-C(1)-C(2)-C(3)	-3.0(3)
O(1)-C(2)-C(3)-C(4)	137.0(2)
C(1)-C(2)-C(3)-C(4)	70.0(3)
C(15)-C(2)-C(3)-C(4)	-91.4(3)
C(11)-O(2)-C(4)-C(3)	126.08(18)
C(11)-O(2)-C(4)-C(5)	-1.9(2)
C(2)-C(3)-C(4)-O(2)	-73.3(3)
C(2)-C(3)-C(4)-C(5)	48.5(3)
O(2)-C(4)-C(5)-C(6)	-147.69(17)

C(3)-C(4)-C(5)-C(6)	88.4(2)
O(2)-C(4)-C(5)-C(10)	-22.83(19)
C(3)-C(4)-C(5)-C(10)	-146.78(18)
C(4)-C(5)-C(6)-C(16)	-107.2(3)
C(10)-C(5)-C(6)-C(16)	135.1(2)
C(4)-C(5)-C(6)-C(7)	74.8(2)
C(10)-C(5)-C(6)-C(7)	-42.9(3)
C(16)-C(6)-C(7)-C(8)	-126.0(3)
C(5)-C(6)-C(7)-C(8)	52.0(3)
C(6)-C(7)-C(8)-C(9)	-59.7(3)
C(7)-C(8)-C(9)-C(17)	-176.3(2)
C(7)-C(8)-C(9)-C(10)	57.3(3)
C(8)-C(9)-C(10)-C(11)	-159.74(19)
C(17)-C(9)-C(10)-C(11)	73.4(2)
C(8)-C(9)-C(10)-C(5)	-46.7(2)
C(17)-C(9)-C(10)-C(5)	-173.59(18)
C(6)-C(5)-C(10)-C(11)	160.63(18)
C(4)-C(5)-C(10)-C(11)	37.04(19)
C(6)-C(5)-C(10)-C(9)	40.1(3)
C(4)-C(5)-C(10)-C(9)	-83.5(2)
C(4)-O(2)-C(11)-C(10)	26.8(2)
C(4)-O(2)-C(11)-C(12)	-98.14(18)
C(9)-C(10)-C(11)-O(2)	81.8(2)
C(5)-C(10)-C(11)-O(2)	-39.64(19)
C(9)-C(10)-C(11)-C(12)	-156.09(18)
C(5)-C(10)-C(11)-C(12)	82.5(2)
O(2)-C(11)-C(12)-O(3)	146.7(2)
C(10)-C(11)-C(12)-O(3)	27.2(3)
O(2)-C(11)-C(12)-C(13)	-38.5(3)
C(10)-C(11)-C(12)-C(13)	-158.00(19)
O(3)-C(12)-C(13)-C(14)	-123.1(3)
C(11)-C(12)-C(13)-C(14)	62.2(3)
O(1)-C(1)-C(14)-C(13)	-173.64(17)
C(2)-C(1)-C(14)-C(13)	-102.9(3)
C(12)-C(13)-C(14)-C(1)	53.6(3)
C(8)-C(9)-C(17)-C(19)	-54.5(3)

C(10)-C(9)-C(17)-C(19)	71.1(3)
C(8)-C(9)-C(17)-C(18)	70.3(3)
C(10)-C(9)-C(17)-C(18)	-164.1(2)

Symmetry transformations used to generate equivalent atoms:

- **X-ray crystal structure of racemic 11:**

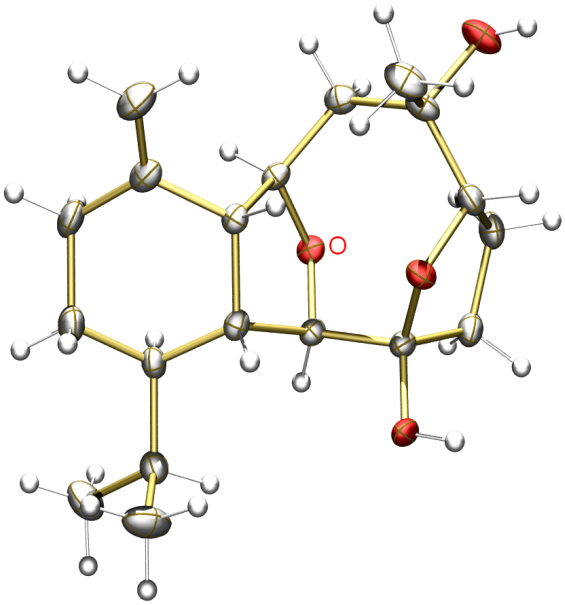

Table 1. Crystal data and structure refinement for sad.

Identification code	C19H30O4	
Empirical formula	C19 H30 O4	
Formula weight	322.43	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P b c a	
Unit cell dimensions	a = 9.7599(15) Å	α= 90°.
	b = 19.355(3) Å	β= 90°.
	c = 19.543(3) Å	γ = 90°.
Volume	3691.8(10) $Å^3$	
Z	8	
Density (calculated)	1.160 Mg/m^3	
Absorption coefficient	0.080 mm^{-1}	
F(000)	1408	
Crystal size	0.08 x 0.06 x 0.03 mm^3	
Theta range for data collection	2.35 to 27.00°.	
Index ranges	0<=h<=12, 0<=k<=24, 0<=l<=24	
Reflections collected	4030	

Independent reflections	4030 [R(int) = 0.0690]
Completeness to theta = 27.00°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9976 and 0.9937
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4030 / 2 / 214
Goodness-of-fit on F^2	1.060
Final R indices [I>2sigma(I)]	R1 = 0.0600, wR2 = 0.1432
R indices (all data)	R1 = 0.0809, wR2 = 0.1525
Extinction coefficient	na
Largest diff. peak and hole	0.525 and -0.215 e.$Å^{-3}$

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

	x	y	z	U(eq)
C(1)	4134(2)	7467(1)	4794(1)	19(1)
C(2)	4585(2)	7759(1)	5482(1)	25(1)
C(3)	4547(2)	8539(1)	5391(1)	28(1)
C(4)	3496(2)	8649(1)	4820(1)	23(1)
C(5)	3862(2)	9175(1)	4263(1)	23(1)
C(6)	5286(2)	9078(1)	3925(1)	22(1)
C(7)	5821(2)	8369(1)	3705(1)	18(1)
C(8)	4878(2)	7923(1)	3251(1)	18(1)
C(9)	5217(2)	7998(1)	2500(1)	23(1)
C(10)	6310(2)	7528(1)	2233(1)	28(1)
C(11)	6010(2)	6780(1)	2441(1)	27(1)
C(12)	6053(2)	6710(1)	3220(1)	20(1)
C(13)	4978(2)	7185(1)	3558(1)	18(1)
C(14)	5336(2)	7333(1)	4309(1)	18(1)
C(15)	2700(2)	9227(1)	3747(1)	31(1)
C(16)	4580(2)	8459(1)	2114(1)	33(1)
C(17)	5931(2)	5957(1)	3465(1)	24(1)
C(18)	7155(2)	5516(1)	3252(1)	36(1)
C(19)	4625(2)	5596(1)	3236(2)	40(1)

O(1)	3400(2)	6844(1)	4834(1)	25(1)
O(2)	3245(1)	7981(1)	4514(1)	21(1)
O(3)	3911(2)	9840(1)	4596(1)	32(1)
O(4)	6176(1)	7944(1)	4291(1)	19(1)

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

C(1)-O(1)	1.404(2)
C(1)-O(2)	1.429(2)
C(1)-C(2)	1.524(3)
C(1)-C(14)	1.530(3)
C(2)-C(3)	1.521(3)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(4)	1.531(3)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-O(2)	1.446(2)
C(4)-C(5)	1.532(3)
C(4)-H(4A)	1.0000
C(5)-O(3)	1.444(2)
C(5)-C(15)	1.521(3)
C(5)-C(6)	1.550(3)
C(6)-C(7)	1.529(3)
C(6)-H(6A)	0.9900
C(6)-H(6B)	0.9900
C(7)-O(4)	1.451(2)
C(7)-C(8)	1.543(3)
C(7)-H(7A)	1.0000
C(8)-C(9)	1.512(3)
C(8)-C(13)	1.553(3)
C(8)-H(8A)	1.0000
C(9)-C(16)	1.323(3)
C(9)-C(10)	1.496(3)
C(10)-C(11)	1.531(3)

C(10)-H(10A)	0.9900
C(10)-H(10B)	0.9900
C(11)-C(12)	1.530(3)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-C(17)	1.539(3)
C(12)-C(13)	1.544(3)
C(12)-H(12A)	1.0000
C(13)-C(14)	1.535(3)
C(13)-H(13A)	1.0000
C(14)-O(4)	1.440(2)
C(14)-H(14A)	1.0000
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-H(16A)	0.9500
C(16)-H(16B)	0.9500
C(17)-C(19)	1.521(3)
C(17)-C(18)	1.526(3)
C(17)-H(17A)	1.0000
C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800
C(19)-H(19A)	0.9800
C(19)-H(19B)	0.9800
C(19)-H(19C)	0.9800
O(1)-H(1O)	0.8400
O(3)-H(3O)	0.838(10)
O(3)-H(3OB)	0.843(10)

O(1)-C(1)-O(2)	108.03(15)
O(1)-C(1)-C(2)	114.59(16)
O(2)-C(1)-C(2)	104.73(16)
O(1)-C(1)-C(14)	106.31(16)
O(2)-C(1)-C(14)	110.27(15)
C(2)-C(1)-C(14)	112.84(16)

C(3)-C(2)-C(1)	104.96(17)
C(3)-C(2)-H(2A)	110.8
C(1)-C(2)-H(2A)	110.8
C(3)-C(2)-H(2B)	110.8
C(1)-C(2)-H(2B)	110.8
H(2A)-C(2)-H(2B)	108.8
C(2)-C(3)-C(4)	103.89(17)
C(2)-C(3)-H(3A)	111.0
C(4)-C(3)-H(3A)	111.0
C(2)-C(3)-H(3B)	111.0
C(4)-C(3)-H(3B)	111.0
H(3A)-C(3)-H(3B)	109.0
O(2)-C(4)-C(3)	106.86(16)
O(2)-C(4)-C(5)	109.84(16)
C(3)-C(4)-C(5)	116.99(17)
O(2)-C(4)-H(4A)	107.6
C(3)-C(4)-H(4A)	107.6
C(5)-C(4)-H(4A)	107.6
O(3)-C(5)-C(15)	105.28(16)
O(3)-C(5)-C(4)	106.30(17)
C(15)-C(5)-C(4)	109.92(17)
O(3)-C(5)-C(6)	105.69(16)
C(15)-C(5)-C(6)	113.20(19)
C(4)-C(5)-C(6)	115.59(17)
C(7)-C(6)-C(5)	122.29(17)
C(7)-C(6)-H(6A)	106.8
C(5)-C(6)-H(6A)	106.8
C(7)-C(6)-H(6B)	106.8
C(5)-C(6)-H(6B)	106.8
H(6A)-C(6)-H(6B)	106.6
O(4)-C(7)-C(6)	111.69(16)
O(4)-C(7)-C(8)	106.21(15)
C(6)-C(7)-C(8)	117.37(16)
O(4)-C(7)-H(7A)	107.0
C(6)-C(7)-H(7A)	107.0
C(8)-C(7)-H(7A)	107.0

C(9)-C(8)-C(7)	112.05(16)
C(9)-C(8)-C(13)	116.72(16)
C(7)-C(8)-C(13)	104.72(15)
C(9)-C(8)-H(8A)	107.7
C(7)-C(8)-H(8A)	107.7
C(13)-C(8)-H(8A)	107.7
C(16)-C(9)-C(10)	123.1(2)
C(16)-C(9)-C(8)	121.0(2)
C(10)-C(9)-C(8)	115.88(18)
C(9)-C(10)-C(11)	110.19(17)
C(9)-C(10)-H(10A)	109.6
C(11)-C(10)-H(10A)	109.6
C(9)-C(10)-H(10B)	109.6
C(11)-C(10)-H(10B)	109.6
H(10A)-C(10)-H(10B)	108.1
C(12)-C(11)-C(10)	110.10(17)
C(12)-C(11)-H(11A)	109.6
C(10)-C(11)-H(11A)	109.6
C(12)-C(11)-H(11B)	109.6
C(10)-C(11)-H(11B)	109.6
H(11A)-C(11)-H(11B)	108.2
C(11)-C(12)-C(17)	113.04(17)
C(11)-C(12)-C(13)	110.72(17)
C(17)-C(12)-C(13)	112.30(16)
C(11)-C(12)-H(12A)	106.8
C(17)-C(12)-H(12A)	106.8
C(13)-C(12)-H(12A)	106.8
C(14)-C(13)-C(12)	111.38(16)
C(14)-C(13)-C(8)	102.31(15)
C(12)-C(13)-C(8)	115.18(16)
C(14)-C(13)-H(13A)	109.2
C(12)-C(13)-H(13A)	109.2
C(8)-C(13)-H(13A)	109.2
O(4)-C(14)-C(1)	108.24(15)
O(4)-C(14)-C(13)	105.00(14)
C(1)-C(14)-C(13)	116.66(16)

O(4)-C(14)-H(14A)	108.9
C(1)-C(14)-H(14A)	108.9
C(13)-C(14)-H(14A)	108.9
C(5)-C(15)-H(15A)	109.5
C(5)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(5)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(9)-C(16)-H(16A)	120.0
C(9)-C(16)-H(16B)	120.0
H(16A)-C(16)-H(16B)	120.0
C(19)-C(17)-C(18)	108.63(19)
C(19)-C(17)-C(12)	114.16(18)
C(18)-C(17)-C(12)	112.63(18)
C(19)-C(17)-H(17A)	107.0
C(18)-C(17)-H(17A)	107.0
C(12)-C(17)-H(17A)	107.0
C(17)-C(18)-H(18A)	109.5
C(17)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
C(17)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
C(17)-C(19)-H(19A)	109.5
C(17)-C(19)-H(19B)	109.5
H(19A)-C(19)-H(19B)	109.5
C(17)-C(19)-H(19C)	109.5
H(19A)-C(19)-H(19C)	109.5
H(19B)-C(19)-H(19C)	109.5
C(1)-O(1)-H(1O)	109.5
C(1)-O(2)-C(4)	111.23(15)
C(5)-O(3)-H(3O)	113(4)
C(5)-O(3)-H(3OB)	115(4)
H(3O)-O(3)-H(3OB)	119(6)
C(14)-O(4)-C(7)	110.44(14)

Symmetry transformations used to generate equivalent atoms:

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

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	20(1)	18(1)	20(1)	3(1)	3(1)	1(1)
C(2)	29(1)	29(1)	18(1)	0(1)	4(1)	3(1)
C(3)	34(1)	29(1)	21(1)	-7(1)	4(1)	2(1)
C(4)	22(1)	19(1)	26(1)	-5(1)	3(1)	2(1)
C(5)	22(1)	15(1)	30(1)	-5(1)	-6(1)	-1(1)
C(6)	22(1)	21(1)	24(1)	0(1)	-5(1)	-6(1)
C(7)	16(1)	22(1)	18(1)	3(1)	-2(1)	-4(1)
C(8)	17(1)	20(1)	16(1)	0(1)	-2(1)	-1(1)
C(9)	25(1)	26(1)	18(1)	0(1)	-4(1)	-11(1)
C(10)	31(1)	36(1)	16(1)	0(1)	4(1)	-8(1)
C(11)	30(1)	31(1)	19(1)	-6(1)	3(1)	-4(1)
C(12)	17(1)	23(1)	20(1)	-5(1)	2(1)	-1(1)
C(13)	13(1)	23(1)	17(1)	0(1)	0(1)	-2(1)
C(14)	17(1)	19(1)	17(1)	2(1)	0(1)	-1(1)
C(15)	27(1)	24(1)	42(1)	-3(1)	-11(1)	6(1)
C(16)	36(1)	38(1)	24(1)	8(1)	-3(1)	-7(1)
C(17)	26(1)	24(1)	23(1)	-2(1)	1(1)	2(1)
C(18)	35(1)	30(1)	44(1)	-9(1)	-2(1)	6(1)
C(19)	32(1)	24(1)	65(2)	4(1)	3(1)	-7(1)
O(1)	26(1)	19(1)	29(1)	2(1)	13(1)	-2(1)
O(2)	19(1)	17(1)	26(1)	-1(1)	0(1)	1(1)
O(3)	35(1)	19(1)	43(1)	-8(1)	-6(1)	1(1)
O(4)	18(1)	21(1)	18(1)	1(1)	-4(1)	-2(1)

Table 5. Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^{\ 3}$) for sad.

	x	y	z	U(eq)
H(2A)	3950	7612	5850	30
H(2B)	5521	7601	5598	30
H(3A)	5458	8718	5255	33
H(3B)	4255	8771	5818	33
H(4A)	2622	8805	5039	27
H(6A)	5299	9375	3513	27
H(6B)	5970	9272	4246	27
H(7A)	6684	8449	3442	22
H(8A)	3917	8089	3318	21
H(10A)	7211	7670	2419	33
H(10B)	6352	7562	1728	33
H(11A)	5094	6644	2270	32
H(11B)	6698	6469	2233	32
H(12A)	6973	6879	3371	24
H(13A)	4058	6959	3535	21
H(14A)	5893	6940	4492	21
H(15A)	1830	9290	3990	46
H(15B)	2660	8802	3475	46
H(15C)	2860	9622	3444	46
H(16A)	4812	8503	1644	39
H(16B)	3892	8746	2307	39
H(17A)	5919	5967	3976	29
H(18A)	7028	5042	3418	55
H(18B)	7995	5710	3450	55
H(18C)	7230	5513	2752	55
H(19A)	4621	5120	3410	60
H(19B)	4584	5589	2735	60
H(19C)	3829	5844	3417	60
H(1O)	2690	6904	5070	37
H(3O)	4600(40)	9890(30)	4840(30)	49
H(3OB)	3150(30)	9980(30)	4740(30)	49

Table 6. Torsion angles [°] for sad.

O(1)-C(1)-C(2)-C(3)	-147.27(17)
O(2)-C(1)-C(2)-C(3)	-29.1(2)
C(14)-C(1)-C(2)-C(3)	90.9(2)
C(1)-C(2)-C(3)-C(4)	25.5(2)
C(2)-C(3)-C(4)-O(2)	-13.0(2)
C(2)-C(3)-C(4)-C(5)	-136.54(18)
O(2)-C(4)-C(5)-O(3)	172.35(15)
C(3)-C(4)-C(5)-O(3)	-65.7(2)
O(2)-C(4)-C(5)-C(15)	58.9(2)
C(3)-C(4)-C(5)-C(15)	-179.12(17)
O(2)-C(4)-C(5)-C(6)	-70.8(2)
C(3)-C(4)-C(5)-C(6)	51.2(2)
O(3)-C(5)-C(6)-C(7)	161.37(18)
C(15)-C(5)-C(6)-C(7)	-83.9(2)
C(4)-C(5)-C(6)-C(7)	44.1(3)
C(5)-C(6)-C(7)-O(4)	-71.4(2)
C(5)-C(6)-C(7)-C(8)	51.6(3)
O(4)-C(7)-C(8)-C(9)	-138.07(16)
C(6)-C(7)-C(8)-C(9)	96.2(2)
O(4)-C(7)-C(8)-C(13)	-10.62(19)
C(6)-C(7)-C(8)-C(13)	-136.34(17)
C(7)-C(8)-C(9)-C(16)	-92.7(2)
C(13)-C(8)-C(9)-C(16)	146.6(2)
C(7)-C(8)-C(9)-C(10)	86.6(2)
C(13)-C(8)-C(9)-C(10)	-34.1(2)
C(16)-C(9)-C(10)-C(11)	-130.5(2)
C(8)-C(9)-C(10)-C(11)	50.2(2)
C(9)-C(10)-C(11)-C(12)	-63.6(2)
C(10)-C(11)-C(12)-C(17)	-173.13(17)
C(10)-C(11)-C(12)-C(13)	59.9(2)
C(11)-C(12)-C(13)-C(14)	-158.85(16)
C(17)-C(12)-C(13)-C(14)	73.7(2)
C(11)-C(12)-C(13)-C(8)	-42.9(2)
C(17)-C(12)-C(13)-C(8)	-170.34(16)

C(9)-C(8)-C(13)-C(14)	151.20(16)
C(7)-C(8)-C(13)-C(14)	26.67(18)
C(9)-C(8)-C(13)-C(12)	30.2(2)
C(7)-C(8)-C(13)-C(12)	-94.32(18)
O(1)-C(1)-C(14)-O(4)	-175.88(14)
O(2)-C(1)-C(14)-O(4)	67.26(19)
C(2)-C(1)-C(14)-O(4)	-49.5(2)
O(1)-C(1)-C(14)-C(13)	66.1(2)
O(2)-C(1)-C(14)-C(13)	-50.8(2)
C(2)-C(1)-C(14)-C(13)	-167.53(17)
C(12)-C(13)-C(14)-O(4)	89.83(18)
C(8)-C(13)-C(14)-O(4)	-33.75(18)
C(12)-C(13)-C(14)-C(1)	-150.36(17)
C(8)-C(13)-C(14)-C(1)	86.06(19)
C(11)-C(12)-C(17)-C(19)	-58.8(2)
C(13)-C(12)-C(17)-C(19)	67.3(2)
C(11)-C(12)-C(17)-C(18)	65.6(2)
C(13)-C(12)-C(17)-C(18)	-168.20(18)
O(1)-C(1)-O(2)-C(4)	144.17(16)
C(2)-C(1)-O(2)-C(4)	21.6(2)
C(14)-C(1)-O(2)-C(4)	-100.04(18)
C(3)-C(4)-O(2)-C(1)	-5.5(2)
C(5)-C(4)-O(2)-C(1)	122.38(17)
C(1)-C(14)-O(4)-C(7)	-96.40(17)
C(13)-C(14)-O(4)-C(7)	28.87(19)
C(6)-C(7)-O(4)-C(14)	117.83(16)
C(8)-C(7)-O(4)-C(14)	-11.28(19)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for sad [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1O)...O(4)#1	0.84	1.96	2.7937(19)	173.4
O(3)-H(3O)...O(3)#2	0.838(10)	1.890(15)	2.721(3)	171(6)

Symmetry transformations used to generate equivalent atoms:

#1 x-1/2,-y+3/2,-z+1 #2 -x+1,-y+2,-z+1