X-ray Structure Determination of 8

Crystals of approximate dimensions 0.544 x 0.254 x 0.132 mm^3 was mounted on a MiTeGen cryoloop in a random orientation. Preliminary examination and data collection were performed using a Bruker X8 Kappa Apex II Charge Coupled Device (CCD) Detector system single crystal X-Ray diffractometer equipped with an Oxford Cryostream LT device. All data were collected using graphite monochromated Mo Kα radiation (λ= 0.71073 Å) from a fine focus sealed tube X-Ray source. Preliminary unit cell constants were determined with a set of 36 narrow frame scans. Typical data sets consist of combinations of ϖ and ϕ scan frames with typical scan width of 0.5° and counting time of 10 seconds/frame at a crystal to detector distance of 4.0 cm. The collected frames were integrated using an orientation matrix determined from the narrow frame scans. Apex II and SAINT software packages (*Bruker Analytical X-Ray, Madison, WI, 2016*) were used for data collection and data integration. Analysis of the integrated data did not show any decay. Final cell constants were determined by global refinement of 7781 reflections harvested from the complete data set. Collected data were corrected for systematic errors using SADABS (*Reference: Bruker Analytical X-Ray, Madison, WI, 2016*) based on the Laue symmetry using equivalent reflections.

Crystal data and intensity data collection parameters are listed in Table 1S. Structure solution and refinement were carried out using the SHELXTL- PLUS software package (*Sheldrick, G.M. (2008). Acta Cryst. A64,112-122*). The structure was solved and refined successfully in the monoclinic space group $P2_1/c$. Full matrix least-squares refinements were carried out by minimizing $\Sigma w(F_o^2-F_c^2)^2$. The non-hydrogen atoms were refined anisotropically to convergence. The 7-membered ring is disordered and the two orientations were refined with geometrical restraints to 82:18%. All hydrogen atoms were treated using appropriate riding

model (AFIX m3). The final residual values and structure refinement parameters are listed in Table 1S.

Complete listings of positional and isotropic displacement coefficients for hydrogen atoms, anisotropic displacement coefficients for the non-hydrogen atoms and other geometrical parameters are listed as supplementary material (Tables 2S to 7S). Table of calculated and observed structure factors are available in electronic format.

Table 1S. Crystal data and structure refinement for **8.**

Empirical formula	$C_{17}\ H_{18}\ O_4\ S$	
Formula weight	318.37	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 5.6186(2) Å	α= 90°.
	b = 28.3140(12) Å	β= 92.382(3)°.
	c = 9.5145(4) Å	γ = 90°.
Volume	1512.31(11) $Å^3$	
Z	4	
Density (calculated)	1.398 Mg/m^3	
Absorption coefficient	0.230 mm^{-1}	
F(000)	672	
Crystal size	0.544 x 0.254 x 0.132 mm^3	
Theta range for data collection	2.260 to 33.335°.	
Index ranges	$-8\leq h\leq 8$, $-43\leq k\leq 43$, $-14\leq l\leq 14$	
Reflections collected	32743	
Independent reflections	5861 [R(int) = 0.0478]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9145 and 0.8529	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5861 / 175 / 286	
Goodness-of-fit on F^2	1.039	
Final R indices [I>2sigma(I)]	R1 = 0.0412, wR2 = 0.0966	
R indices (all data)	R1 = 0.0584, wR2 = 0.1051	
Largest diff. peak and hole	0.511 and -0.489 $e.Å^{-3}$	

Table 2S. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **8**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	7904(1)	6044(1)	4618(1)	15(1)
O(2)	9236(2)	6404(1)	5685(1)	18(1)
O(3)	9683(2)	5770(1)	3972(1)	22(1)
O(4)	6258(2)	6308(1)	3744(1)	21(1)
O(1)	6170(3)	7020(1)	6560(2)	23(1)
C(1)	7857(4)	7194(1)	5946(2)	16(1)
C(2)	9548(13)	6877(2)	5281(10)	15(1)
C(3)	11371(6)	6971(1)	4442(4)	18(1)
C(4)	12162(3)	7414(1)	3915(2)	19(1)
C(5)	11420(3)	7850(1)	4249(2)	17(1)
C(6)	9673(3)	7989(1)	5230(2)	15(1)
C(7)	8193(3)	7702(1)	5929(2)	16(1)
C(8)	9389(3)	8518(1)	5447(1)	17(1)
C(9)	8177(3)	8735(1)	4130(2)	20(1)
C(10)	11754(6)	8766(1)	5819(4)	22(1)
O(1')	6458(19)	7109(3)	6374(12)	23(1)
C(1')	8150(30)	7213(4)	5669(13)	16(1)
C(2')	9810(60)	6861(8)	5180(50)	17(3)
C(3')	11660(20)	6864(4)	4355(18)	11(2)
C(4')	12737(15)	7267(3)	3767(9)	18(1)
C(5')	12153(13)	7712(2)	3904(8)	16(1)
C(6')	10291(11)	7933(2)	4621(7)	10(1)
C(7')	8635(13)	7721(2)	5370(9)	20(2)
C(8')	10261(13)	8470(2)	4525(7)	20(1)
C(9')	7830(17)	8680(3)	4485(10)	20(1)
C(10')	11840(40)	8669(6)	5730(20)	35(4)
C(11)	6431(2)	5697(1)	5821(1)	14(1)
C(12)	4391(2)	5873(1)	6410(1)	17(1)
C(13)	3194(2)	5589(1)	7332(1)	18(1)
C(14)	4001(2)	5136(1)	7674(1)	16(1)
C(15)	6042(2)	4970(1)	7064(1)	19(1)

C(16)	7271(2)	5248(1)	6139(1)	18(1)
C(17)	2677(2)	4837(1)	8682(1)	23(1)

Table 3S. Bond lengths [Å] and angles [°] for **8**.

S(1)-O(3)	1.4259(9)
S(1)-O(4)	1.4279(9)
S(1)-O(2)	1.6007(9)
S(1)-C(11)	1.7446(11)
O(2)-C(2)	1.408(4)
O(2)-C(2')	1.42(2)
O(1)-C(1)	1.2363(19)
C(1)-C(7)	1.451(2)
C(1)-C(2)	1.468(3)
C(2)-C(3)	1.351(3)
C(3)-C(4)	1.429(3)
C(3)-H(3)	0.9500
C(4)-C(5)	1.346(2)
C(4)-H(4)	0.9500
C(5)-C(6)	1.437(2)
C(5)-H(5)	0.9500
C(6)-C(7)	1.357(2)
C(6)-C(8)	1.5226(19)
C(7)-H(7)	0.9500
C(8)-C(9)	1.529(2)
C(8)-C(10)	1.531(3)
C(8)-H(8)	1.0000
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
C(10)-H(10A)	0.9800
C(10)-H(10B)	0.9800
C(10)-H(10C)	0.9800
O(1')-C(1')	1.223(10)
C(1')-C(2')	1.455(14)
C(1')-C(7')	1.492(11)
C(2')-C(3')	1.327(13)
C(3')-C(4')	1.418(11)
C(3')-H(3')	0.9500

C(4')-C(5')	1.309(9)
C(4')-H(4')	0.9500
C(5')-C(6')	1.418(9)
C(5')-H(5')	0.9500
C(6')-C(7')	1.337(9)
C(6')-C(8')	1.525(7)
C(7')-H(7')	0.9500
C(8')-C(9')	1.488(11)
C(8')-C(10')	1.530(14)
C(8')-H(8')	1.0000
C(9')-H(9'1)	0.9800
C(9')-H(9'2)	0.9800
C(9')-H(9'3)	0.9800
C(10')-H(10D)	0.9800
C(10')-H(10E)	0.9800
C(10')-H(10F)	0.9800
C(11)-C(16)	1.3847(15)
C(11)-C(12)	1.3897(15)
C(12)-C(13)	1.3843(16)
C(12)-H(12)	0.9500
C(13)-C(14)	1.3946(16)
C(13)-H(13)	0.9500
C(14)-C(15)	1.3894(16)
C(14)-C(17)	1.4999(16)
C(15)-C(16)	1.3867(16)
C(15)-H(15)	0.9500
C(16)-H(16)	0.9500
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800

O(3)-S(1)-O(4)	118.90(6)
O(3)-S(1)-O(2)	107.59(5)
O(4)-S(1)-O(2)	108.17(5)
O(3)-S(1)-C(11)	109.55(5)
O(4)-S(1)-C(11)	111.33(5)

O(2)-S(1)-C(11)	99.45(5)
C(2)-O(2)-S(1)	119.5(5)
C(2')-O(2)-S(1)	118(2)
O(1)-C(1)-C(7)	120.20(18)
O(1)-C(1)-C(2)	118.9(2)
C(7)-C(1)-C(2)	120.9(2)
C(3)-C(2)-O(2)	117.0(3)
C(3)-C(2)-C(1)	130.9(3)
O(2)-C(2)-C(1)	111.9(2)
C(2)-C(3)-C(4)	129.4(3)
C(2)-C(3)-H(3)	115.3
C(4)-C(3)-H(3)	115.3
C(5)-C(4)-C(3)	128.25(17)
C(5)-C(4)-H(4)	115.9
C(3)-C(4)-H(4)	115.9
C(4)-C(5)-C(6)	129.05(14)
C(4)-C(5)-H(5)	115.5
C(6)-C(5)-H(5)	115.5
C(7)-C(6)-C(5)	127.19(13)
C(7)-C(6)-C(8)	117.00(14)
C(5)-C(6)-C(8)	115.74(12)
C(6)-C(7)-C(1)	133.02(15)
C(6)-C(7)-H(7)	113.5
C(1)-C(7)-H(7)	113.5
C(6)-C(8)-C(9)	109.23(12)
C(6)-C(8)-C(10)	112.85(18)
C(9)-C(8)-C(10)	110.98(19)
C(6)-C(8)-H(8)	107.9
C(9)-C(8)-H(8)	107.9
C(10)-C(8)-H(8)	107.9
C(8)-C(9)-H(9A)	109.5
C(8)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
C(8)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5

C(8)-C(10)-H(10A)	109.5
C(8)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(8)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5
O(1')-C(1')-C(2')	122.3(12)
O(1')-C(1')-C(7')	119.3(10)
C(2')-C(1')-C(7')	118.3(11)
C(3')-C(2')-O(2)	113.6(12)
C(3')-C(2')-C(1')	135.5(16)
O(2)-C(2')-C(1')	110.9(11)
C(2')-C(3')-C(4')	126.5(12)
C(2')-C(3')-H(3')	116.8
C(4')-C(3')-H(3')	116.8
C(5')-C(4')-C(3')	128.5(8)
C(5')-C(4')-H(4')	115.7
C(3')-C(4')-H(4')	115.7
C(4')-C(5')-C(6')	131.6(6)
C(4')-C(5')-H(5')	114.2
C(6')-C(5')-H(5')	114.2
C(7')-C(6')-C(5')	127.1(6)
C(7')-C(6')-C(8')	118.2(6)
C(5')-C(6')-C(8')	114.7(5)
C(6')-C(7')-C(1')	132.0(7)
C(6')-C(7')-H(7')	114.0
C(1')-C(7')-H(7')	114.0
C(9')-C(8')-C(6')	114.1(6)
C(9')-C(8')-C(10')	112.0(10)
C(6')-C(8')-C(10')	108.5(9)
C(9')-C(8')-H(8')	107.3
C(6')-C(8')-H(8')	107.3
C(10')-C(8')-H(8')	107.3
C(8')-C(9')-H(9'1)	109.5
C(8')-C(9')-H(9'2)	109.5
H(9'1)-C(9')-H(9'2)	109.5

C(8')-C(9')-H(9'3)	109.5
H(9'1)-C(9')-H(9'3)	109.5
H(9'2)-C(9')-H(9'3)	109.5
C(8')-C(10')-H(10D)	109.5
C(8')-C(10')-H(10E)	109.5
H(10D)-C(10')-H(10E)	109.5
C(8')-C(10')-H(10F)	109.5
H(10D)-C(10')-H(10F)	109.5
H(10E)-C(10')-H(10F)	109.5
C(16)-C(11)-C(12)	121.43(10)
C(16)-C(11)-S(1)	119.67(8)
C(12)-C(11)-S(1)	118.88(8)
C(13)-C(12)-C(11)	118.48(10)
C(13)-C(12)-H(12)	120.8
C(11)-C(12)-H(12)	120.8
C(12)-C(13)-C(14)	121.38(10)
C(12)-C(13)-H(13)	119.3
C(14)-C(13)-H(13)	119.3
C(15)-C(14)-C(13)	118.71(10)
C(15)-C(14)-C(17)	120.99(11)
C(13)-C(14)-C(17)	120.30(11)
C(16)-C(15)-C(14)	120.92(10)
C(16)-C(15)-H(15)	119.5
C(14)-C(15)-H(15)	119.5
C(11)-C(16)-C(15)	119.07(10)
C(11)-C(16)-H(16)	120.5
C(15)-C(16)-H(16)	120.5
C(14)-C(17)-H(17A)	109.5
C(14)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(14)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4S. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **8**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	16(1)	15(1)	14(1)	1(1)	3(1)	1(1)
O(2)	23(1)	13(1)	18(1)	2(1)	-3(1)	-1(1)
O(3)	23(1)	20(1)	23(1)	-2(1)	10(1)	2(1)
O(4)	22(1)	24(1)	18(1)	6(1)	-2(1)	1(1)
O(1)	20(1)	21(1)	29(1)	6(1)	10(1)	-1(1)
C(1)	17(1)	16(1)	15(1)	2(1)	3(1)	1(1)
C(2)	15(2)	13(1)	16(1)	3(1)	0(1)	-2(1)
C(3)	17(1)	16(1)	19(1)	-1(1)	2(1)	5(1)
C(4)	18(1)	19(1)	20(1)	-1(1)	7(1)	1(1)
C(5)	19(1)	14(1)	18(1)	1(1)	5(1)	-2(1)
C(6)	17(1)	14(1)	13(1)	1(1)	2(1)	0(1)
C(7)	18(1)	14(1)	16(1)	2(1)	4(1)	1(1)
C(8)	23(1)	13(1)	15(1)	-1(1)	4(1)	-1(1)
C(9)	27(1)	14(1)	19(1)	3(1)	2(1)	1(1)
C(10)	26(1)	20(2)	22(1)	-3(1)	0(1)	-5(1)
O(1')	20(1)	21(1)	29(1)	6(1)	10(1)	-1(1)
C(1')	17(1)	16(1)	15(1)	2(1)	3(1)	1(1)
C(2')	13(6)	10(4)	28(10)	0(4)	-1(4)	9(4)
C(3')	5(3)	11(4)	18(4)	3(4)	4(2)	5(3)
C(4')	16(4)	18(3)	20(3)	1(3)	3(3)	2(2)
C(5')	16(3)	10(3)	21(3)	-4(2)	5(2)	5(2)
C(6')	12(3)	9(2)	10(3)	1(2)	5(2)	2(2)
C(7')	24(3)	18(3)	18(3)	10(2)	13(3)	6(2)
C(8')	33(3)	9(2)	18(3)	3(2)	4(2)	1(2)
C(9')	27(1)	14(1)	19(1)	3(1)	2(1)	1(1)
C(10')	59(7)	9(6)	37(6)	-6(4)	-4(5)	-2(4)
C(11)	14(1)	14(1)	14(1)	0(1)	1(1)	0(1)
C(12)	16(1)	15(1)	20(1)	-1(1)	2(1)	2(1)
C(13)	15(1)	20(1)	19(1)	-2(1)	4(1)	0(1)
C(14)	17(1)	18(1)	14(1)	-1(1)	0(1)	-4(1)
C(15)	20(1)	16(1)	21(1)	3(1)	2(1)	1(1)

C(16)	17(1)	16(1)	21(1)	1(1)	4(1)	3(1)
C(17)	25(1)	26(1)	20(1)	2(1)	3(1)	-7(1)

Table 5S. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **8**.

	x	y	z	U(eq)
H(3)	12255	6704	4162	21
H(4)	13365	7399	3244	22
H(5)	12154	8102	3768	20
H(7)	7170	7866	6530	19
H(8)	8315	8566	6247	20
H(9A)	9172	8683	3322	30
H(9B)	7962	9075	4273	30
H(9C)	6620	8586	3951	30
H(10A)	12772	8756	5008	34
H(10B)	12555	8606	6620	34
H(10C)	11447	9096	6068	34
H(3')	12331	6566	4138	14
H(4')	14053	7207	3198	21
H(5')	13160	7926	3438	19
H(7')	7566	7933	5790	23
H(8')	11013	8559	3628	24
H(9'1)	6820	8516	3776	30
H(9'2)	7927	9016	4243	30
H(9'3)	7143	8646	5410	30
H(10D)	12080	9008	5590	53
H(10E)	13382	8507	5763	53
H(10F)	11063	8618	6626	53
H(12)	3831	6182	6185	20
H(13)	1795	5705	7739	21
H(15)	6603	4661	7283	23
H(16)	8670	5132	5729	21
H(17A)	2690	4992	9603	35
H(17B)	3448	4527	8774	35
H(17C)	1028	4796	8326	35

Table 6S. Torsion angles [°] for **8**.

O(3)-S(1)-O(2)-C(2)	100.84(19)
O(4)-S(1)-O(2)-C(2)	-28.77(19)
C(11)-S(1)-O(2)-C(2)	-145.04(19)
O(3)-S(1)-O(2)-C(2')	92.4(9)
O(4)-S(1)-O(2)-C(2')	-37.2(9)
C(11)-S(1)-O(2)-C(2')	-153.5(9)
S(1)-O(2)-C(2)-C(3)	-80.5(8)
S(1)-O(2)-C(2)-C(1)	103.1(6)
O(1)-C(1)-C(2)-C(3)	172.6(8)
C(7)-C(1)-C(2)-C(3)	-9.5(13)
O(1)-C(1)-C(2)-O(2)	-11.6(9)
C(7)-C(1)-C(2)-O(2)	166.3(4)
O(2)-C(2)-C(3)-C(4)	-177.5(5)
C(1)-C(2)-C(3)-C(4)	-1.9(15)
C(2)-C(3)-C(4)-C(5)	7.4(8)
C(3)-C(4)-C(5)-C(6)	1.0(4)
C(4)-C(5)-C(6)-C(7)	-7.8(3)
C(4)-C(5)-C(6)-C(8)	175.29(17)
C(5)-C(6)-C(7)-C(1)	0.1(3)
C(8)-C(6)-C(7)-C(1)	176.9(2)
O(1)-C(1)-C(7)-C(6)	-171.4(2)
C(2)-C(1)-C(7)-C(6)	10.8(6)
C(7)-C(6)-C(8)-C(9)	-105.97(16)
C(5)-C(6)-C(8)-C(9)	71.26(17)
C(7)-C(6)-C(8)-C(10)	130.1(2)
C(5)-C(6)-C(8)-C(10)	-52.7(2)
S(1)-O(2)-C(2')-C(3')	-76(4)
S(1)-O(2)-C(2')-C(1')	103(3)
O(1')-C(1')-C(2')-C(3')	175(5)
C(7')-C(1')-C(2')-C(3')	-9(7)
O(1')-C(1')-C(2')-O(2)	-3(5)
C(7')-C(1')-C(2')-O(2)	174(2)
O(2)-C(2')-C(3')-C(4')	-176(2)
C(1')-C(2')-C(3')-C(4')	6(8)

C(2')-C(3')-C(4')-C(5')	0(4)
C(3')-C(4')-C(5')-C(6')	-2.0(17)
C(4')-C(5')-C(6')-C(7')	0.6(13)
C(4')-C(5')-C(6')-C(8')	179.5(8)
C(5')-C(6')-C(7')-C(1')	-1.6(15)
C(8')-C(6')-C(7')-C(1')	179.6(10)
O(1')-C(1')-C(7')-C(6')	-178.4(11)
C(2')-C(1')-C(7')-C(6')	5(3)
C(7')-C(6')-C(8')-C(9')	-35.0(9)
C(5')-C(6')-C(8')-C(9')	146.0(7)
C(7')-C(6')-C(8')-C(10')	90.6(13)
C(5')-C(6')-C(8')-C(10')	-88.4(12)
O(3)-S(1)-C(11)-C(16)	7.92(12)
O(4)-S(1)-C(11)-C(16)	141.48(10)
O(2)-S(1)-C(11)-C(16)	-104.67(10)
O(3)-S(1)-C(11)-C(12)	-170.24(9)
O(4)-S(1)-C(11)-C(12)	-36.68(11)
O(2)-S(1)-C(11)-C(12)	77.16(10)
C(16)-C(11)-C(12)-C(13)	-0.04(18)
S(1)-C(11)-C(12)-C(13)	178.09(9)
C(11)-C(12)-C(13)-C(14)	0.19(18)
C(12)-C(13)-C(14)-C(15)	-0.40(18)
C(12)-C(13)-C(14)-C(17)	179.69(11)
C(13)-C(14)-C(15)-C(16)	0.46(18)
C(17)-C(14)-C(15)-C(16)	-179.63(11)
C(12)-C(11)-C(16)-C(15)	0.10(18)
S(1)-C(11)-C(16)-C(15)	-178.02(9)
C(14)-C(15)-C(16)-C(11)	-0.32(19)

Symmetry transformations used to generate equivalent atoms:

Table 7S. Hydrogen bonds for **8** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(3)-H(3)...O(4)#1	0.95	2.56	3.414(3)	150.0
C(3')-H(3')...O(4)#1	0.95	2.37	3.100(10)	133.5

Symmetry transformations used to generate equivalent atoms:

#1 x+1,y,z

Projection view with 50% probability ellipsoids- disorder components omitted for clarity: