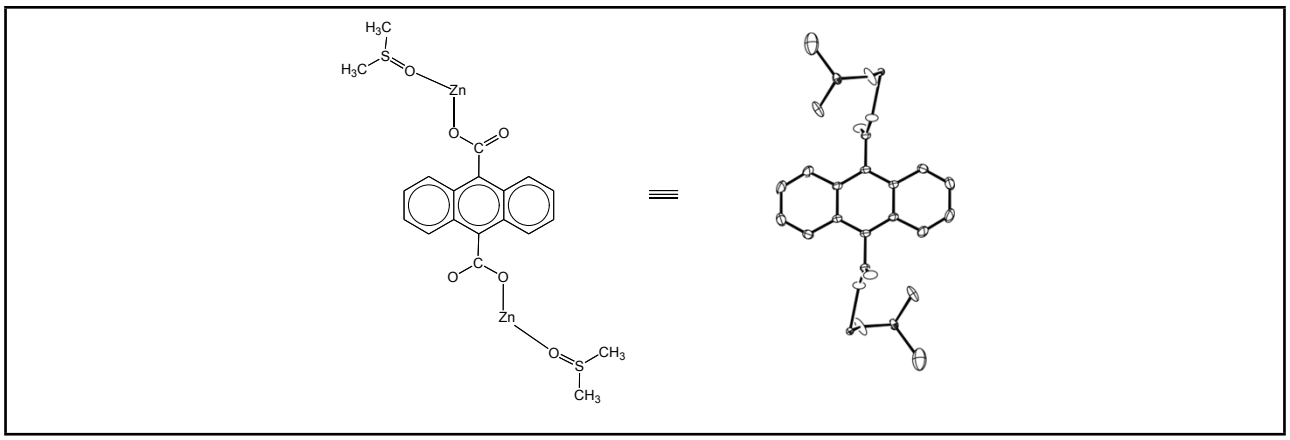
X-ray Structure Determination of Compound ZnADC



Compound ZnADC, $C_{19}H_{17}SO_5Zn$, crystallizes in the orthorhombic space group Pnma (systematic absences hk0: =odd and 0kl: k+l=odd) with a=7.3053(7)Å, b=17.5056(14)Å, c=12.6731(12)Å, V=1620.7(3)Å^3, Z=4, and d_{calc}=1.733 g/cm^3 . X-ray intensity data were collected on a Bruker APEXII CCD area detector employing graphite-monochromated Mo-Kα radiation (λ=0.71073 Å) at a temperature of 143(1)K. Preliminary indexing was performed from a series of thirty-six 0.5° rotation frames with exposures of 10 seconds. A total of 1492 frames were collected with a crystal to detector distance of 37.6 mm, rotation widths of 0.5° and exposures of 30 seconds:

scan type	2θ	ω	ϕ	χ	frames
ϕ	-23.00	325.83	12.48	28.88	739
ϕ	-15.50	258.48	47.74	19.46	488
ω	-8.00	320.62	277.32	84.71	68
ω	-10.50	306.95	272.07	99.72	80
ω	17.00	321.08	318.36	83.36	117

Rotation frames were integrated using SAINT[i], producing a listing of unaveraged F^2 and $\sigma(F^2)$ values which were then passed to the SHELXTL[ii] program package for further processing and structure solution. A total of 22496 reflections were measured over the ranges $1.98 \leq \theta \leq 27.53°$, $-9 \leq h \leq 9$, $-22 \leq k \leq 22$, $-15 \leq l \leq 16$ yielding 1929 unique reflections (Rint = 0.0557). The intensity data were corrected for Lorentz and polarization effects and for absorption using SADABS[iii] (minimum and maximum transmission 0.6511, 0.7456).

The structure was solved by direct methods (SHELXS-97[iv]). Refinement was by full-matrix least

squares based on F^2 using SHELXL-97.[v] All reflections were used during refinement. The weighting scheme used was $w=1/[\sigma^2(F_o^2)+ (0.0485P)^2 + 1.6449P]$ where $P = (F_o^2 + 2F_c^2)/3$. Non-hydrogen atoms were refined anisotropically and hydrogen atoms were placed in idealized positions and were not refined. Refinement converged to R1=0.0366 and wR2=0.0894 for 1413 observed reflections for which $F > 4\sigma(F)$ and R1=0.0578 and wR2=0.0996 and GOF =1.090 for all 1929 unique, non-zero reflections and 128 variables.[vi] The maximum Δ/σ in the final cycle of least squares was 0.000 and the two most prominent peaks in the final difference Fourier were +0.970 and -0.660 e/Å^3.

Table 1. lists cell information, data collection parameters, and refinement data. Final positional and equivalent isotropic thermal parameters are given in Tables 2. and 3. Anisotropic thermal parameters are in Table 4. Tables 5. and 6. list bond distances and bond angles. Figure 1. is an ORTEP[vii] representation of the molecule with 30% probability thermal ellipsoids displayed.

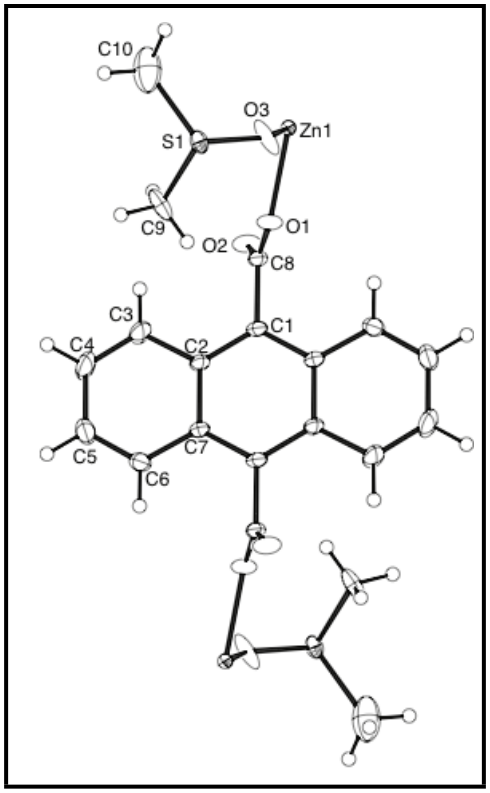


Figure 1. ORTEP drawing of the title compound with 30% probability thermal ellipsoids.

Table 1. Summary of Structure Determination of Compound 99210

Empirical formula	$C_{19}H_{17}SO_5Zn$
Formula weight	422.76
Temperature	143(1) K
Wavelength	0.71073 Å
Crystal system	orthorhombic
Space group	Pnma
Cell constants:	
a	7.3053(7) Å
b	17.5056(14) Å
c	12.6731(12) Å
Volume	1620.7(3) $Å^3$
Z	4
Density (calculated)	1.733 Mg/m^3
Absorption coefficient	1.674 mm^{-1}
F(000)	868
Crystal size	0.25 x 0.02 x 0.01 mm^3
Theta range for data collection	1.98 to 27.53°
Index ranges	$-9 \leq h \leq 9$, $-22 \leq k \leq 22$, $-15 \leq l \leq 16$
Reflections collected	22496
Independent reflections	1929 [R(int) = 0.0557]
Completeness to theta = 27.53°	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7456 and 0.6511
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1929 / 108 / 128
Goodness-of-fit on F^2	1.090
Final R indices [I>2sigma(I)]	R1 = 0.0366, wR2 = 0.0894
R indices (all data)	R1 = 0.0578, wR2 = 0.0996
Largest diff. peak and hole	0.970 and -0.660 $e.Å^{-3}$

Table 2. Refined Positional Parameters for Compound 99210

Atom	x	y	z	U_{eq}, Å^2
Zn1	0.24380(6)	0.2500	0.74642(4)	0.01113(15)
S1	-0.0501(2)	0.28725(9)	0.54161(12)	0.0182(3)
O1	0.3440(3)	0.16631(12)	0.64993(17)	0.0258(5)
O2	0.6508(3)	0.16382(12)	0.65647(18)	0.0276(5)
O3	-0.0086(4)	0.2500	0.6548(2)	0.0329(8)
C1	0.4975(4)	0.06727(17)	0.5613(2)	0.0165(6)
C2	0.5591(5)	0.06963(16)	0.4567(2)	0.0177(6)
C3	0.6182(7)	0.13911(19)	0.4079(3)	0.0390(10)
C4	0.6778(7)	0.1404(2)	0.3073(3)	0.0434(11)
C5	0.6851(6)	0.0730(2)	0.2467(3)	0.0290(7)
C6	0.6303(5)	0.00628(18)	0.2885(2)	0.0204(6)
C7	0.5647(4)	0.00120(16)	0.3944(2)	0.0142(6)
C8	0.4978(5)	0.13890(17)	0.6287(2)	0.0197(6)
C9	0.1315(7)	0.2500	0.4613(4)	0.0345(12)
C10	-0.0013(6)	0.3948(3)	0.5415(4)	0.0579(12)

$U_{eq}=1/3[U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}aa^*bb^*\cos\gamma+2U_{13}aa^*cc^*\cos\beta+2U_{23}bb^*cc^*\cos\alpha]$

Table 3. Positional Parameters for Hydrogens in Compound 99210

Atom	x	y	z	U_{iso}, Å^2
H3	0.6170	0.1841	0.4478	0.038
H4	0.7154	0.1869	0.2771	0.038
H5	0.7280	0.0749	0.1766	0.038
H6	0.6348	-0.0383	0.2473	0.038
H9a	0.2475	0.2704	0.4855	0.038
H9b	0.1137	0.2644	0.3891	0.038
H9c	0.1353	0.1953	0.4668	0.038
H10a	-0.0863	0.4234	0.5884	0.038
H10b	-0.0102	0.4184	0.4730	0.038
H10c	0.1220	0.4071	0.5692	0.038

Table 4. Refined Thermal Parameters (U's) for Compound 99210

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Zn1	0.0120(2)	0.0101(2)	0.0113(2)	0.000	0.00125(19)	0.000
S1	0.0178(7)	0.0248(7)	0.0119(6)	0.0018(6)	-0.0025(6)	-0.0002(6)
O1	0.0371(13)	0.0169(11)	0.0235(11)	-0.0083(9)	0.0008(10)	0.0056(10)
O2	0.0370(14)	0.0187(11)	0.0271(12)	-0.0112(10)	-0.0080(11)	-0.0005(10)
O3	0.0148(16)	0.075(3)	0.0091(14)	0.000	0.0000(12)	0.000
C1	0.0197(14)	0.0115(12)	0.0182(13)	-0.0042(11)	-0.0015(12)	0.0014(11)
C2	0.0245(15)	0.0118(13)	0.0170(13)	-0.0029(11)	-0.0019(12)	-0.0028(12)
C3	0.078(3)	0.0165(16)	0.0228(16)	-0.0032(13)	0.0020(18)	-0.0182(18)
C4	0.079(3)	0.0226(18)	0.0282(18)	0.0046(14)	0.0044(19)	-0.0235(19)
C5	0.0368(18)	0.0316(17)	0.0187(15)	0.0020(13)	0.0056(15)	-0.0063(16)
C6	0.0232(16)	0.0201(15)	0.0178(14)	-0.0037(12)	0.0005(13)	0.0023(13)
C7	0.0142(13)	0.0116(13)	0.0167(13)	-0.0023(11)	-0.0023(11)	0.0005(11)

C8	0.0331(16)	0.0121(13)	0.0140(13)	-0.0018(11)	-0.0011(13)	0.0022(13)
C9	0.034(3)	0.058(3)	0.011(2)	0.000	0.004(2)	0.000
C10	0.038(2)	0.081(3)	0.055(3)	0.032(2)	0.001(2)	0.009(2)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2b^*c^*U_{23}kl+2a^*c^*U_{13}hl+2a^*b^*U_{12}hk)]$

Table 5. Bond Distances in Compound 99210, Å

Zn1-O1	2.044(2)	Zn1-O1#1	2.044(2)	Zn1-O2#2	2.062(2)
Zn1-O2#3	2.062(2)	Zn1-O3	2.179(3)	Zn1-O3#4	2.200(3)
S1-S1#1	1.304(3)	S1-O3	1.605(3)	S1-C9	1.795(5)
S1-C10	1.916(5)	O1-C8	1.251(4)	O2-C8	1.250(4)
O2-Zn1#4	2.062(2)	O3-S1#1	1.605(3)	O3-Zn1#2	2.200(3)
C1-C7#5	1.399(4)	C1-C2	1.401(4)	C1-C8	1.517(4)
C2-C3	1.431(4)	C2-C7	1.435(4)	C3-C4	1.347(5)
C4-C5	1.408(5)	C5-C6	1.344(5)	C6-C7	1.428(4)
C7-C1#5	1.399(4)	C9-S1#1	1.795(5)		

Table 6. Bond Angles in Compound 99210, °

O1-Zn1-O1#1	91.58(13)	O1-Zn1-O2#2	178.15(10)	O1#1-Zn1-O2#2	87.17(9)
O1-Zn1-O2#3	87.17(9)	O1#1-Zn1-O2#3	178.15(10)	O2#2-Zn1-O2#3	94.04(13)
O1-Zn1-O3	89.10(9)	O1#1-Zn1-O3	89.10(9)	O2#2-Zn1-O3	92.24(9)
O2#3-Zn1-O3	92.24(9)	O1-Zn1-O3#4	92.62(9)	O1#1-Zn1-O3#4	92.62(9)
O2#2-Zn1-O3#4	86.07(9)	O2#3-Zn1-O3#4	86.07(9)	O3-Zn1-O3#4	177.52(9)
S1#1-S1-O3	66.03(7)	S1#1-S1-C9	68.69(7)	O3-S1-C9	102.66(18)
S1#1-S1-C10	169.26(14)	O3-S1-C10	111.39(18)	C9-S1-C10	102.64(17)
C8-O1-Zn1	136.5(2)	C8-O2-Zn1#4	135.9(2)	S1#1-O3-S1	47.95(14)
S1#1-O3-Zn1	129.53(17)	S1-O3-Zn1	129.53(17)	S1#1-O3-Zn1#2	110.67(16)
S1-O3-Zn1#2	110.67(16)	Zn1-O3-Zn1#2	113.13(13)	C7#5-C1-C2	120.6(3)
C7#5-C1-C8	118.9(3)	C2-C1-C8	120.5(3)	C1-C2-C3	122.1(3)
C1-C2-C7	120.3(3)	C3-C2-C7	117.6(3)	C4-C3-C2	121.3(3)
C3-C4-C5	121.0(3)	C6-C5-C4	120.1(3)	C5-C6-C7	121.6(3)
C1#5-C7-C6	122.6(3)	C1#5-C7-C2	119.0(3)	C6-C7-C2	118.3(3)
O2-C8-O1	127.5(3)	O2-C8-C1	116.6(3)	O1-C8-C1	115.9(3)
S1#1-C9-S1	42.61(15)				

Symmetry transformations used to generate equivalent atoms:

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

#4 x+1/2,-y+1/2,-z+3/2 #5 -x+1,-y,-z+1

[i]Bruker (2009) SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

[ii]Bruker (2009) SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.

[iii]Sheldrick, G.M. (2007) SADABS. University of Gottingen, Germany.

[iv]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[v]Sheldrick, G.M. (2008) Acta Cryst. A64,112-122.

[vi]$R1 = \Sigma||F_o| - |F_c|| / \Sigma |F_o|$
$wR2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^2)^2]^{½}$
$GOF = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{½}$
where n = the number of reflections and p = the number of parameters refined.

[vii]"ORTEP-II: A Fortran Thermal Ellipsoid Plot Program for Crystal Structure Illustrations". C.K. Johnson (1976) ORNL-5138.