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STRUCTURE DETERMINATION SUMMARYCrystal Data

Identification code	Zn(BDC)(H ₂ O). (DMF)
Empirical formula	C ₁₁ H ₁₃ NO ₆ Zn
Color	Colorless
Habit	Prismatic
Crystal size	0.31 x 0.27 x 0.22 mm
Crystal system	Monoclinic
Space group	P2 ₁ /n
Unit cell dimensions	$a = 6.718(3) \text{ \AA}$ $\alpha = 90^\circ$ $b = 15.488(7) \text{ \AA}$ $\beta = 102.83(4)^\circ$ $c = 12.430(8) \text{ \AA}$ $\gamma = 90^\circ$
Volume	1261.0(11) Å ³
Cell Refinement	Siemens P3/VAX rel. 4.2
Z	4
Formula weight	320.59
Density (calculated)	1.689 Mg/m ³
Absorption coefficient	1.970 mm ⁻¹
F(000)	656

Data Collection

Diffractometer	Siemens R3m/V Autodiffractometer
Collection Software	Siemens P3/VAX rel. 4.2
Wavelength	0.71073 Å
Temperature	293(2) K
Monochromator	Graphite
θ range for data collection	2.13 to 25.04°
Measurement Method	ω scan
Measurement Speed	Variable, 1.50 to 14.65°/min. in ω
Measurement Range	1.60° in ω
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 25.0% of total scan time
Standard Reflections	2
Standards Count Interval	48
Standards Decay	0%
Data Reduction	Siemens XDISK rel. 4.2
Index ranges	$-8 \leq h \leq 7$ $-1 \leq k \leq 18$ $-14 \leq l \leq 7$
Reflections collected	2508
Independent reflections	2219 ($R_{\text{int}} = 0.0360$)
Observed Reflections	1524
Observed Criterion	$>2\sigma(I)$
Absorption correction	Psi-scan
Max. and min. transmission	0.648 and 0.543

Structure Solution and Refinement

Structure Solution

Direct Methods

SHELXS-86 (Sheldrick, 1990)

Structure Refinement

Fourier Difference Map

SHELXL-93 (Sheldrick, 1993)

Refinement method

Full-matrix least-squares on F^2

Hydrogen Atoms

Riding model

Weighting Scheme

$w=1/[\sigma^2(F_o^2)+(0.0462P)^2+1.1551P]$
where $P=(F_o^2+2F_c^2)/3$

Data / restraints / parameters

2218 / 0 / 172

Final R indices [$I>2\sigma(I)$]

R1 = 0.0458, wR2 = 0.0923

R indices (all data)

R1 = 0.0842, wR2 = 0.1092

Goodness-of-fit on F^2

1.079

Largest and Mean Δ/σ

-0.001, 0.000

Largest diff. peak and hole

0.631 and -0.613 $e\text{\AA}^{-3}$

Molecular Graphics

Siemens SHELXTL PC rel. 5.03

Publication Material

Siemens SHELXTL PC rel. 5.03

Table 1. Atomic coordinates [$\times 10^4$] and equivalent isotropic displacement parameters [$\text{\AA}^2 \times 10^3$] for $\text{C}_{11}\text{H}_{13}\text{NO}_6\text{Zn}$.
 $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Zn(1)	2003(1)	5050(1)	5771(1)	17(1)
O(1)	-600(6)	3952(3)	3669(3)	41(1)
O(2)	2442(6)	4030(3)	4791(3)	41(1)
O(3)	2841(6)	796(3)	587(3)	43(1)
O(4)	5876(6)	805(3)	1715(3)	44(1)
O(5)	4830(5)	5213(2)	6569(3)	29(1)
C(1)	1948(8)	2990(3)	3372(4)	22(1)
C(2)	599(8)	2584(3)	2513(4)	25(1)
C(3)	1280(8)	1960(3)	1895(4)	25(1)
C(4)	3324(8)	1713(3)	2125(4)	22(1)
C(5)	4671(8)	2112(3)	3003(4)	26(1)
C(6)	3991(8)	2743(3)	3617(4)	28(1)
C(7)	1213(8)	3706(3)	3998(4)	22(1)
C(8)	4067(8)	1054(3)	1419(4)	22(1)
O(1S)	6439(7)	4869(3)	8628(3)	53(1)
N(1S)	6382(9)	3967(4)	10052(4)	49(1)
C(2S)	5644(11)	4286(4)	9060(5)	47(2)
C(3S)	5375(13)	3279(5)	10506(7)	74(2)
C(4S)	8278(12)	4285(6)	10706(6)	73(3)

Table 2. Bond lengths [Å] for $C_{11}H_{13}NO_6Zn$.

Zn(1)-O(5)	1.954(4)	Zn(1)-O(1)#1	2.014(4)
Zn(1)-O(4)#2	2.025(4)	Zn(1)-O(2)	2.055(4)
Zn(1)-O(3)#3	2.068(4)	Zn(1)-Zn(1)#1	2.940(2)
O(1)-C(7)	1.255(6)	O(1)-Zn(1)#1	2.014(4)
O(2)-C(7)	1.243(6)	O(3)-C(8)	1.236(6)
O(3)-Zn(1)#4	2.068(4)	O(4)-C(8)	1.250(6)
O(4)-Zn(1)#5	2.025(4)	C(1)-C(2)	1.388(7)
C(1)-C(6)	1.392(7)	C(1)-C(7)	1.500(7)
C(2)-C(3)	1.375(7)	C(3)-C(4)	1.392(7)
C(4)-C(5)	1.397(7)	C(4)-C(8)	1.502(7)
C(5)-C(6)	1.379(7)	O(1S)-C(2S)	1.231(8)
N(1S)-C(2S)	1.319(8)	N(1S)-C(4S)	1.437(9)
N(1S)-C(3S)	1.442(9)		

Symmetry transformations used to generate equivalent atoms:

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

Table 3. Bond angles [$^{\circ}$] for $C_{11}H_{13}NO_6Zn$.

O(5)-Zn(1)-O(1)#1	101.7(2)	O(5)-Zn(1)-O(4)#2	103.7(2)
O(1)#1-Zn(1)-O(4)#2	92.1(2)	O(5)-Zn(1)-O(2)	98.5(2)
O(1)#1-Zn(1)-O(2)	159.2(2)	O(4)#2-Zn(1)-O(2)	87.8(2)
O(5)-Zn(1)-O(3)#3	97.0(2)	O(1)#1-Zn(1)-O(3)#3	88.0(2)
O(4)#2-Zn(1)-O(3)#3	158.8(2)	O(2)-Zn(1)-O(3)#3	84.6(2)
O(5)-Zn(1)-Zn(1)#1	169.50(10)	O(1)#1-Zn(1)-Zn(1)#1	80.50(12)
O(4)#2-Zn(1)-Zn(1)#1	86.42(13)	O(2)-Zn(1)-Zn(1)#1	78.73(12)
O(3)#3-Zn(1)-Zn(1)#1	72.69(12)	C(7)-O(1)-Zn(1)#1	128.0(3)
C(7)-O(2)-Zn(1)	128.6(4)	C(8)-O(3)-Zn(1)#4	135.9(4)
C(8)-O(4)-Zn(1)#5	119.7(4)	C(2)-C(1)-C(6)	119.2(5)
C(2)-C(1)-C(7)	120.0(5)	C(6)-C(1)-C(7)	120.8(5)
C(3)-C(2)-C(1)	120.5(5)	C(2)-C(3)-C(4)	120.9(5)
C(3)-C(4)-C(5)	118.5(5)	C(3)-C(4)-C(8)	120.4(5)
C(5)-C(4)-C(8)	121.0(5)	C(6)-C(5)-C(4)	120.6(5)
C(5)-C(6)-C(1)	120.4(5)	O(2)-C(7)-O(1)	124.1(5)
O(2)-C(7)-C(1)	118.2(5)	O(1)-C(7)-C(1)	117.6(5)
O(3)-C(8)-O(4)	124.8(5)	O(3)-C(8)-C(4)	117.5(5)
O(4)-C(8)-C(4)	117.7(5)	C(2S)-N(1S)-C(4S)	120.2(6)
C(2S)-N(1S)-C(3S)	122.1(7)	C(4S)-N(1S)-C(3S)	117.7(6)
O(1S)-C(2S)-N(1S)	125.0(7)		

Symmetry transformations used to generate equivalent atoms:

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

Table 4. Anisotropic displacement parameters [$\text{\AA}^2 \times 10^3$] for $\text{C}_{11}\text{H}_{13}\text{NO}_6\text{Zn}$. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [(ha^*)^2 U_{11} + \dots + 2hka^*b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
Zn(1)	17(1)	19(1)	16(1)	0(1)	6(1)	1(1)
O(1)	31(3)	44(3)	41(2)	-22(2)	-3(2)	15(2)
O(2)	32(2)	45(3)	44(3)	-29(2)	3(2)	6(2)
O(3)	35(3)	57(3)	36(2)	-27(2)	5(2)	4(2)
O(4)	37(3)	52(3)	44(3)	-23(2)	8(2)	15(2)
O(5)	21(2)	43(2)	20(2)	0(2)	2(2)	-1(2)
C(1)	26(3)	22(3)	19(3)	-1(2)	10(2)	1(2)
C(2)	21(3)	28(3)	27(3)	0(2)	7(2)	9(2)
C(3)	24(3)	24(3)	25(3)	-5(2)	4(2)	0(2)
C(4)	24(3)	23(3)	20(3)	0(2)	9(2)	0(2)
C(5)	19(3)	29(3)	30(3)	-3(3)	4(2)	4(2)
C(6)	29(3)	29(3)	24(3)	-9(2)	3(2)	4(2)
C(7)	31(3)	15(3)	26(3)	6(2)	17(3)	3(2)
C(8)	27(3)	19(3)	24(3)	1(2)	14(2)	-4(2)
O(1S)	63(3)	64(3)	28(2)	5(2)	2(2)	0(3)
N(1S)	63(4)	47(3)	39(3)	8(3)	13(3)	8(3)
C(2S)	62(5)	45(4)	32(4)	-6(3)	7(3)	9(4)
C(3S)	103(7)	55(5)	69(5)	4(4)	31(5)	-3(5)
C(4S)	80(6)	92(6)	42(4)	22(4)	-2(4)	-10(5)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $\text{C}_{11}\text{H}_{13}\text{NO}_6\text{Zn}$.

	x	y	z	U(eq)
H(51)	5545(5)	4831(2)	6395(3)	43
H(52)	5456(5)	5068(2)	7205(3)	43
H(21)	-775(8)	2736(3)	2354(4)	30
H(31)	363(8)	1699(3)	1315(4)	30
H(53)	6038(8)	1950(3)	3175(4)	31
H(61)	4903(8)	3005(3)	4198(4)	33
H(2S1)	4432(11)	4053(4)	8655(5)	56
H(3S1)	6145(13)	3138(5)	11231(7)	111
H(3S2)	5282(13)	2779(5)	10040(7)	111
H(3S3)	4029(13)	3461(5)	10548(7)	111
H(3S4)	4159(13)	3114(5)	9981(7)	111
H(3S5)	5022(13)	3473(5)	11173(7)	111
H(3S6)	6275(13)	2791(5)	10665(7)	111
H(4S1)	8592(12)	3986(6)	11400(6)	110
H(4S2)	8159(12)	4892(6)	10834(6)	110
H(4S3)	9350(12)	4189(6)	10322(6)	110
H(4S4)	8809(12)	4725(6)	10304(6)	110
H(4S5)	9241(12)	3819(6)	10870(6)	110
H(4S6)	8051(12)	4522(6)	11382(6)	110

