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P-803-1

STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$C_{26}H_{48}N_2S_8Zn_5$
Color; Habit	colorless transparent prism
Crystal size (mm)	0.161 x 0.230 x 0.368
Crystal System	Monoclinic
Space Group	C2/c
Unit Cell Dimensions	a = 25.797(2) Å
	b = 17.773(2) Å
	c = 21.655(2) Å
	β = 112.00(1)°
Volume	9208.6(15) Å^3
	Cell determination used 39 reflections in 2θ range from 3.42° to 24.69°
Z	8
Formula weight	972.0
Density(calc.)	1.402 Mg/m^3
Absorption Coefficient	2.947 mm^{-1}
F(000)	3968

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Data Collection

Diffractometer Used	Siemens R3m/V
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	293
Monochromator	Highly oriented graphite crystal
2θ Range	3.0 to 37.0°
Scan Type	ω
Scan Speed	Variable; 7.00 to 40.00°/min. in ω
Scan Range (ω)	2.00°
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 25.0% of total scan time
Standard Reflections	3 measured every 97 reflections Observed standard intensity ratios showed no substantial changes during course of data collection; observed variation was $\pm$ 1.86%. Intensities were scaled on standard intensity ratios.
Index Ranges	$0 \leq h \leq 22$, $-15 \leq k \leq 15$ $-19 \leq l \leq 17$
Reflections Collected	6803
Independent Reflections	3418 (R_{int} = 4.58%)
Observed Reflections	1611 ($F > 2.0\sigma(F)$)
Absorption Correction	Semi-empirical
Min./Max. Transmission	0.8289 / 0.9826

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Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\Sigma w(F_o - F_c)^2$
Absolute Structure	N/A
Extinction Correction	N/A
Hydrogen Atoms	Riding model, fixed isotropic U set to 1.25U(equiv) of parent atom.
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0020F^2$
Number of Parameters Refined	182
Final R indices (obs. data)	R = 6.98 %, wR = 9.83 % Rw = 6.72 %
R Indices (all data)	R = 13.89 %, wR = 11.48 %
Goodness-of-Fit	1.32
Largest and Mean Δ/σ	0.084, 0.005
Data-to-Parameter Ratio	8.9:1
Largest Difference Peak	1.00 $e\text{Å}^{-3}$
Largest Difference Hole	-0.81 $e\text{Å}^{-3}$

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$Zn_{10}S_4$

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Table 1. Atomic coordinates ($x10^4$) and equivalent isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U(eq)
Zn(1)	0	1936(4)	2500	32(3)
Zn(2)	331(2)	3365(3)	1536(2)	38(3)
Zn(3)	0	4774(4)	2500	37(3)
Zn(4)	988(2)	3365(3)	3304(2)	32(2)
Zn(5)	1326(2)	4834(3)	2289(2)	40(2)
Zn(6)	657(2)	1894(3)	4281(2)	41(2)
N(1)	2011(12)	5523(18)	2309(15)	47(10)
C(1)	2140(17)	6079(23)	2702(19)	50(13)
C(2)	2592(18)	6529(24)	2871(20)	53(13)
C(3)	2907(18)	6341(23)	2547(21)	69(15)
C(4)	2815(16)	5741(22)	2090(19)	42(12)
C(5)	2342(15)	5309(22)	1954(18)	47(12)
C(6)	2764(18)	7153(24)	3273(20)	85(17)
C(7)	3145(19)	5561(24)	1687(22)	100(18)
N(2)	1051(12)	1217(18)	5162(15)	45(10)
C(8)	1377(15)	667(20)	5064(19)	43(12)
C(9)	1680(16)	225(24)	5625(21)	55(14)
C(10)	1690(15)	351(20)	6217(18)	36(11)
C(11)	1351(16)	917(21)	6291(19)	40(12)
C(12)	1047(14)	1342(19)	5775(17)	31(11)
C(13)	2037(21)	-349(26)	5609(24)	118(21)
C(14)	1342(16)	1122(22)	6916(19)	73(15)
S(1)	750(4)	2542(6)	2410(5)	31(3)
S(2)	410(4)	1064(6)	3396(5)	43(3)
C(15)	-211(18)	514(25)	3446(21)	78(16)
C(16)	-53(19)	3(25)	4065(22)	104(19)
S(3)	1093(4)	4040(6)	1377(5)	39(3)
C(17)	730	4648	596	72(16)
C(18)	888	4428	86	112(38)
C(18')	1059	5089	347	100(39)
S(4)	-378(4)	4183(6)	1491(5)	34(3)
S(5)	-9(5)	2649(6)	488(5)	47(4)
C(19)	645	2032	539	75(16)
C(20)	533	1828	-138	74(37)
C(20')	532	1510	236	89(46)
C(20")	838	2347	168	94(81)

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S(6)	-698(5)	5650(6)	2554(5)	49(4)
C(21)	-370(17)	6068(24)	3403(19)	75(17)
C(22)	-700(19)	6725(26)	3556(22)	110(20)
S(7)	1737(4)	4148(6)	3262(5)	43(3)
C(23)	2161(16)	3540(21)	2969(19)	61(15)
C(24)	2482(17)	2992(23)	3530(19)	76(15)
S(8)	1409(4)	2582(6)	4253(5)	38(3)
C(25)	1535(18)	3251(24)	4948(20)	76(16)
C(26)	2053(20)	3704(27)	5105(24)	125(22)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

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Table 2. Bond lengths (A)

Zn(1)-S(1)	2.287 (12)	Zn(1)-S(2)	2.397 (11)
Zn(1)-S(1A)	2.287 (12)	Zn(1)-S(2A)	2.397 (11)
Zn(2)-S(1)	2.314 (11)	Zn(2)-S(3)	2.433 (13)
Zn(2)-S(4)	2.310 (12)	Zn(2)-S(5)	2.460 (12)
Zn(3)-S(4)	2.287 (10)	Zn(3)-S(6)	2.417 (13)
Zn(3)-S(4A)	2.287 (10)	Zn(3)-S(6A)	2.417 (13)
Zn(4)-S(1)	2.319 (11)	Zn(4)-S(7)	2.410 (13)
Zn(4)-S(8)	2.380 (11)	Zn(4)-S(4A)	2.305 (13)
Zn(5)-N(1)	2.138 (34)	Zn(5)-S(3)	2.316 (11)
Zn(5)-S(7)	2.318 (11)	Zn(5)-S(6A)	2.290 (13)
Zn(6)-N(2)	2.161 (29)	Zn(6)-S(2)	2.311 (12)
Zn(6)-S(8)	2.315 (12)	Zn(6)-S(5A)	2.339 (14)
N(1)-C(1)	1.265 (50)	N(1)-C(5)	1.401 (59)
C(1)-C(2)	1.346 (60)	C(2)-C(3)	1.300 (74)
C(2)-C(6)	1.376 (58)	C(3)-C(4)	1.414 (58)
C(4)-C(5)	1.375 (55)	C(4)-C(7)	1.466 (72)
N(2)-C(8)	1.356 (53)	N(2)-C(12)	1.349 (52)
C(8)-C(9)	1.412 (52)	C(9)-C(10)	1.294 (63)
C(9)-C(13)	1.381 (68)	C(10)-C(11)	1.381 (57)
C(11)-C(12)	1.333 (47)	C(11)-C(14)	1.410 (62)
S(2)-C(15)	1.913 (50)	C(15)-C(16)	1.541 (63)
S(3)-C(17)	1.928 (10)	C(17)-C(18)	1.371 (1)
C(17)-C(18')	1.404 (1)	S(4)-Zn(4A)	2.305 (13)
S(5)-C(19)	1.979 (12)	S(5)-Zn(6A)	2.339 (14)
C(19)-C(20)	1.430 (1)	C(19)-C(20')	1.111 (1)
C(19)-C(20")	1.227 (1)	S(6)-C(21)	1.867 (40)
S(6)-Zn(5A)	2.290 (13)	C(21)-C(22)	1.552 (68)
S(7)-C(23)	1.814 (47)	C(23)-C(24)	1.535 (52)
S(8)-C(25)	1.849 (44)	C(25)-C(26)	1.487 (67)

Generation of symmetry equivalent atoms found in this table:

Atom	Generated from	transformation
Zn(4A),Zn(5A),Zn(6A)	Zn(4),Zn(5),Zn(6)	-x, y, 0.5-z
S(1A),S(2A),S(4A),S(5A),S(6A)	S(1),S(2),S(4),S(5),S(6)	-x, y, 0.5-z

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Table 3. Bond angles (°)

S(1)-Zn(1)-S(2)	104.1(4)	S(1)-Zn(1)-S(1A)	123.8(6)
S(2)-Zn(1)-S(1A)	111.4(4)	S(1)-Zn(1)-S(2A)	111.4(4)
S(2)-Zn(1)-S(2A)	99.5(5)	S(1A)-Zn(1)-S(2A)	104.1(4)
S(1)-Zn(2)-S(3)	105.8(4)	S(1)-Zn(2)-S(4)	123.6(5)
S(3)-Zn(2)-S(4)	110.6(4)	S(1)-Zn(2)-S(5)	108.8(4)
S(3)-Zn(2)-S(5)	98.8(4)	S(4)-Zn(2)-S(5)	106.5(4)
S(4)-Zn(3)-S(6)	106.4(4)	S(4)-Zn(3)-S(4A)	125.3(6)
S(6)-Zn(3)-S(4A)	107.9(4)	S(4)-Zn(3)-S(6A)	107.9(4)
S(6)-Zn(3)-S(6A)	99.9(6)	S(4A)-Zn(3)-S(6A)	106.4(4)
S(1)-Zn(4)-S(7)	107.3(5)	S(1)-Zn(4)-S(8)	104.2(4)
S(7)-Zn(4)-S(8)	104.9(4)	S(1)-Zn(4)-S(4A)	125.0(4)
S(7)-Zn(4)-S(4A)	104.9(4)	S(8)-Zn(4)-S(4A)	109.1(4)
N(1)-Zn(5)-S(3)	108.3(10)	N(1)-Zn(5)-S(7)	100.9(8)
S(3)-Zn(5)-S(7)	109.8(4)	N(1)-Zn(5)-S(6A)	105.0(10)
S(3)-Zn(5)-S(6A)	122.7(4)	S(7)-Zn(5)-S(6A)	107.8(5)
N(2)-Zn(6)-S(2)	105.6(9)	N(2)-Zn(6)-S(8)	101.3(9)
S(2)-Zn(6)-S(8)	106.8(5)	N(2)-Zn(6)-S(5A)	105.6(10)
S(2)-Zn(6)-S(5A)	123.1(4)	S(8)-Zn(6)-S(5A)	112.1(4)
Zn(5)-N(1)-C(1)	117.9(32)	Zn(5)-N(1)-C(5)	120.3(24)
C(1)-N(1)-C(5)	121.3(36)	N(1)-C(1)-C(2)	129.1(47)
C(1)-C(2)-C(3)	110.7(40)	C(1)-C(2)-C(6)	133.8(50)
C(3)-C(2)-C(6)	115.4(43)	C(2)-C(3)-C(4)	126.6(42)
C(3)-C(4)-C(5)	118.3(43)	C(3)-C(4)-C(7)	126.8(39)
C(5)-C(4)-C(7)	114.6(35)	N(1)-C(5)-C(4)	113.9(34)
Zn(6)-N(2)-C(8)	112.2(26)	Zn(6)-N(2)-C(12)	127.0(24)
C(8)-N(2)-C(12)	120.4(30)	N(2)-C(8)-C(9)	116.3(39)
C(8)-C(9)-C(10)	123.7(41)	C(8)-C(9)-C(13)	123.4(45)
C(10)-C(9)-C(13)	112.7(37)	C(9)-C(10)-C(11)	117.6(34)
C(10)-C(11)-C(12)	120.9(40)	C(10)-C(11)-C(14)	122.5(33)
C(12)-C(11)-C(14)	116.3(37)	N(2)-C(12)-C(11)	120.8(36)
Zn(1)-S(1)-Zn(2)	102.5(4)	Zn(1)-S(1)-Zn(4)	101.1(5)
Zn(2)-S(1)-Zn(4)	100.4(4)	Zn(1)-S(2)-Zn(6)	99.2(4)
Zn(1)-S(2)-C(15)	104.5(12)	Zn(6)-S(2)-C(15)	104.8(15)
S(2)-C(15)-C(16)	113.2(28)	Zn(2)-S(3)-Zn(5)	98.3(5)
Zn(2)-S(3)-C(17)	104.3(4)	Zn(5)-S(3)-C(17)	107.0(5)
S(3)-C(17)-C(18)	110.7(4)	S(3)-C(17)-C(18')	119.0(3)
Zn(2)-S(4)-Zn(3)	101.6(4)	Zn(2)-S(4)-Zn(4A)	100.5(4)

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Zn(3)-S(4)-Zn(4A)	100.6(5)	Zn(2)-S(5)-C(19)	103.5(4)
Zn(2)-S(5)-Zn(6A)	97.9(5)	C(19)-S(5)-Zn(6A)	109.5(5)
S(5)-C(19)-C(20)	104.0(3)	S(5)-C(19)-C(20')	113.6(3)
S(5)-C(19)-C(20")	104.9(4)	Zn(3)-S(6)-C(21)	103.4(14)
Zn(3)-S(6)-Zn(5A)	100.4(4)	C(21)-S(6)-Zn(5A)	101.0(16)
S(6)-C(21)-C(22)	115.3(25)	Zn(4)-S(7)-Zn(5)	103.7(4)
Zn(4)-S(7)-C(23)	105.4(14)	Zn(5)-S(7)-C(23)	97.3(13)
S(7)-C(23)-C(24)	108.6(31)	Zn(4)-S(8)-Zn(6)	102.4(4)
Zn(4)-S(8)-C(25)	102.2(13)	Zn(6)-S(8)-C(25)	102.2(16)
S(8)-C(25)-C(26)	113.7(38)		

Generation of symmetry equivalent atoms found in this table:

Atom	Generated from	transformation
Zn(4A),Zn(5A),Zn(6A)	Zn(4),Zn(5),Zn(6)	-x, y, 0.5-z
S(1A),S(2A),S(4A),S(5A),S(6A)	S(1),S(2),S(4),S(5),S(6)	-x, y, 0.5-z

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Table 4. Anisotropic displacement coefficients ($Å^2 x 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zn(1)	30(5)	32(5)	29(5)	0	6(4)	0
Zn(2)	30(4)	49(4)	34(4)	-1(3)	10(3)	0(3)
Zn(3)	33(5)	31(5)	42(5)	0	10(4)	0
Zn(4)	26(3)	39(4)	33(4)	-2(3)	14(3)	2(2)
Zn(5)	27(3)	45(4)	44(4)	-7(3)	9(3)	5(3)
Zn(6)	32(3)	50(4)	32(3)	4(3)	4(3)	10(3)

The anisotropic displacement exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$

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Table 5. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U
H(1)	1882	6188	2914	59
H(3)	3225	6656	2614	84
H(5)	2235	4892	1650	55
H(6A)	2501	7194	3487	105
H(6B)	2746	7597	3013	105
H(6C)	3134	7100	3604	105
H(7A)	3464	5888	1804	131
H(7B)	2917	5627	1225	131
H(7C)	3269	5048	1766	131
H(8)	1391	595	4631	48
H(10)	1929	58	6587	47
H(12)	819	1743	5830	39
H(13A)	2000	-394	5154	165
H(13B)	1946	-821	5759	165
H(13C)	2414	-214	5879	165
H(14A)	1570	793	7262	99
H(14B)	959	1078	6874	99
H(14C)	1466	1631	7025	99
H(15A)	-349	212	3051	99
H(15B)	-499	855	3450	99
H(16A)	-377	-273	4050	131
H(16B)	83	302	4463	131
H(16C)	233	-341	4063	131
H(17A)	826	5167	700	87
H(17B)	332	4600	460	87
H(17C)	475	4979	692	87
H(17D)	508	4319	245	87
H(18A)	703	4736	-298	133
H(18B)	1286	4483	221	133
H(18C)	787	3911	-22	133
H(18D)	822	5358	-39	116
H(18E)	1274	5440	682	116
H(18F)	1307	4769	229	116
H(19A)	677	1594	810	95
H(19B)	984	2320	720	95
H(19C)	850	1899	996	95

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H(19D)	886	2331	391	95
H(19E)	534	1527	393	95
H(19F)	916	2018	987	95
H(20A)	834	1526	-159	93
H(20B)	190	1547	-310	93
H(20C)	498	2275	-399	93
H(20D)	866	1238	281	87
H(20E)	298	1202	387	87
H(20F)	335	1639	-224	87
H(20G)	1160	2076	169	124
H(20H)	561	2359	-276	124
H(20I)	944	2852	321	124
H(21A)	0	6223	3454	103
H(21B)	-348	5676	3717	103
H(22A)	-500	6899	4002	135
H(22B)	-1071	6575	3510	135
H(22C)	-723	7122	3247	135
H(23A)	2429	3828	2859	78
H(23B)	1931	3259	2586	78
H(24A)	2716	2653	3405	91
H(24B)	2210	2710	3638	91
H(24C)	2709	3279	3911	91
H(25A)	1545	3000	5344	103
H(25B)	1239	3616	4815	103
H(26A)	2096	4057	5456	159
H(26B)	2052	3969	4718	159
H(26C)	2359	3353	5248	159

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Refinement Summary

Model	m---	n--	R(F)%	Rw(F)	S---
1.all nonhydrogen atoms isotropic; two terminal CH3 groups are disordered: C18 is disordered into C18 and C18', C20 into C20,C20' and C20". Occupancy of C18 varied so that occupancy of C18+C18'=1.00; also with C20, C20'and C20"; C18 and C18' share common isotropic U and C20,C20',C20" have their occupancies adjusted so that their individual isotropic U's are nearly equal.	1611	168	8.29	7.85	1.76
2.all Zn's anisotropic, other atoms isotropic; disordered atoms treated as before.	1611	197	7.59	7.18	1.71
3.same as 2, include hydrogens in idealized positions (riding model) with fixed isotropic U's set to 1.25 U(equiv) of parent atom for ordered atoms only.	1611	194	7.63	7.10	1.69
3.final: same as 3, but fix disorderd atoms and include their hydrogens in idealized positions also(riding model); isotropic U of hydrogens set to 1.25 U(equiv) of parent atom.	1611	182	6.98	6.72	1.32

SPECIAL NOTES : The crystal quality was not the best: it did not diffract much beyond 2-theta of 35 degrees and the peak profiles were somewhat broader than usual at the base (.7-.9 degrees in omega). The reflections were generally very weak; data collection was terminated at 2-theta=37 degrees, at this point there were about 20% observed reflections. Due to the small number of observed reflections, not all atoms could be refined anisotropically. The standard deviations of bond lengths and angles will be larger because of the crystal quality and lack of data.

Final Difference Map

highest amplitude 1.001 e/Å^3

lowest amplitude -0.807 e/Å^3

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Peak	Density ($e/Å^3$)	Neighbor	Distance (Å)	Neighbor	Distance (Å)	Neighbor	Distance (Å)
1.	1.001	peak 4	1.45	peak 5	1.27		
2.	1.001	peak 3	1.70				
3.	0.985	peak 2	1.70				
4.	0.968	peak 1	1.45				
5.	0.942	peak 1	1.27				
6.	0.933	peak 7	1.56				
7.	0.916	peak 6	1.56				
8.	0.820	peak 8	0.61				
9.	0.811	peak 8	0.421				
==========these peaks are less than lowest amplitude==========							
10.	0.715	C(22)	0.91	H(22a)	1.34	H(22c)	0.76
11.	0.689	S(6)	1.61	C(21)	0.65	H(21a)	0.82
12.	0.671	peak 2	1.23	peak 7	1.31		
13.	0.628	C(6)	0.93	H(6a)	0.88	H(6b)	0.86
14.	0.610	C(13)	0.62	H(13a)	0.52	H(13c)	1.10
15.	0.593	-----					

NOTES ON MOLECULAR STRUCTURE AND CRYSTAL PACKING

The quality of the data suffers from crystal quality: recall that the available diffraction sphere was limited. There is one-half dimer in the asymmetric unit and 4 dimers per unit cell. The dimer is completed by a two-fold rotation along the 0,y,1/4 axis. The dimer consists of a network fused alternating Zn-S 6-membered rings using 10Zn and 16S atoms. All the Zn atoms are tetrahedrallly coordinated: Zn1,Zn2,Zn3,Zn4 are all bonded to 4 S atoms. Zn5 and Zn6 are bonded to 3 S and 1 N atoms. All the S atoms are all bonded to three other atoms: 2 Zn and 1 Et, except for S1 and S4 which are bonded to 3 Zn atoms. There seems to be long Zn-S bonds: Zn1-S2 2.397,Zn2-S3 2.434,Zn2-S5 2.460, Zn3-S6 2.417,Zn4-S7 2.410,Zn4-S8 2.380 and the others which may be considered short (2.287-2.350). The averaged bond lengths and standard deviations (square root of [sum(D*D)/N-1], where N=number of bonds, D=bond length-average bond length) have been calculated for chemically equivalent units.

The averaged bond lengths and standard deviations follow:

R=range=max.value-min.value

R/S=range/std.dev.

Zn-S1= Zn-S long bonds Zn-S2= Zn-S short bonds Zn-S3= Zn-S all bonds

C-C1= bond C-C(ring) C-C2=C-C single bonds,ring

C-C3=C-C single bonds,Et(not including disordered atom bonds)

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Bond	N= No.	Ave.	Std. dev.	R	R/S	Bond	No.	Ave.	dev.	R	R/S
Zn-S1	6	2.4163	0.0282	0.080	2.84	Zn-S2	12	2.3130	0.0191	0.063	3.30
Zn-S3	18	2.3449	0.0555	0.173	3.12	Zn-N	2	2.1495	0.0163	0.023	1.41
S-C	6	1.8917	0.0598	0.130	2.18	N-C	4	1.3428	0.0567	0.136	2.39
C-C1	8	1.3569	0.0464	0.120	2.59	C-C2	4	1.4077	0.0506	0.095	1.88
C-C3	4	1.5288	0.0287	0.065	2.26						