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Crystal structure of diaqua-dichlorido-bis(μ_3 -6,6'-(hydrazine-1,2-diylidenebis(methanylylidene))bis(2-methoxyphenolato) $\kappa^6 O,N:N',O',O':O''$)trizinc(II) - ethanol (1/2), $C_{36}H_{44}Cl_2N_4O_{12}Zn_3$

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Abstract

$C_{36}H_{44}Cl_2N_4O_{12}Zn_3$, orthorhombic, *Pbca* (no. 61), $a = 8.3378(8)$ Å, $b = 21.188(2)$ Å, $c = 22.802(2)$ Å, $Z = 4$, $V = 4028.3(7)$ Å³, $R_{gt}(F) = 0.0513$, $wR_{ref}(F^2) = 0.1193$, $T = 296(2)$ K.

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Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

A solution of $ZnCl_2$ (13.6 mg, 0.10 mmol) in 4 mL ethanol was slowly added to a solution of 1,2-bis(2-hydroxy-3-methoxybenzylidene)hydrazine (H_2L) (15.0 mg, 0.05 mmol) and triethylamine (20 μ L) in 4 mL dichloromethane with general stirring for 10 mins. The resulting mixture was then filtered and left undisturbed. Well shaped crystals of the title compound were obtained by slow evaporation within 1 week.

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Table 1: Data collection and handling.

Crystal:	Yellow prism
Size:	$0.19 \times 0.15 \times 0.12$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	1.97 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω -scans
$\theta_{\max}$, completeness:	26.1° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	21203, 3979, 0.064
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2941
$N(\text{param})_{\text{refined}}$:	263
Programs:	Bruker programs [1], SHELX [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Zn1	0.27058(6)	0.05640(2)	0.60046(2)	0.03807(17)
Zn2	0.0000	0.0000	0.5000	0.0376(2)
Cl1	0.45353(16)	−0.01952(6)	0.59165(6)	0.0550(3)
N1	0.2095(4)	0.11003(17)	0.52642(15)	0.0377(9)
N2	0.1410(4)	0.07756(16)	0.47835(15)	0.0367(9)
O1	0.4726(4)	0.21293(15)	0.70957(15)	0.0539(9)
O2	0.3775(4)	0.13172(14)	0.63142(14)	0.0464(8)
O3	−0.0610(4)	−0.00776(14)	0.41380(12)	0.0381(7)
O4	−0.1516(4)	−0.04626(15)	0.31222(13)	0.0470(8)
O5	−0.2066(4)	0.06240(15)	0.51371(14)	0.0504(9)
H5A	−0.2887	0.0424	0.5017	0.061*
H5B	−0.1939	0.0936	0.4907	0.061*
O6	0.7174(5)	0.1108(2)	0.6196(2)	0.0768(12)
H6	0.6373	0.1253	0.6036	0.115*
C1	0.5366(7)	0.2558(2)	0.7509(2)	0.0673(17)
H1A	0.6124	0.2831	0.7319	0.101*
H1B	0.5895	0.2328	0.7816	0.101*
H1C	0.4515	0.2806	0.7674	0.101*
C2	0.3937(6)	0.2375(2)	0.6623(2)	0.0404(11)
C3	0.3644(6)	0.3003(2)	0.6531(2)	0.0487(12)
H3	0.4031	0.3298	0.6799	0.058*
C4	0.2779(7)	0.3210(2)	0.6046(2)	0.0590(15)
H4	0.2570	0.3638	0.5993	0.071*
C5	0.2239(7)	0.2777(2)	0.5647(2)	0.0570(14)
H5	0.1660	0.2915	0.5323	0.068*
C6	0.2542(6)	0.2122(2)	0.5717(2)	0.0421(11)
C7	0.3410(6)	0.1908(2)	0.6211(2)	0.0385(10)
C8	0.1961(6)	0.1710(2)	0.5266(2)	0.0413(11)
H8	0.1444	0.1895	0.4949	0.050*
C9	0.1901(5)	0.0929(2)	0.42688(19)	0.0369(10)

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
H9	0.2695	0.1236	0.4243	0.044*
C10	0.1308(5)	0.0660(2)	0.37258(18)	0.0346(10)
C11	0.1973(6)	0.0910(2)	0.3207(2)	0.0437(11)
H11	0.2781	0.1211	0.3233	0.052*
C12	0.1458(6)	0.0720(2)	0.2664(2)	0.0462(12)
H12	0.1894	0.0899	0.2327	0.055*
C13	0.0280(6)	0.0259(2)	0.2620(2)	0.0457(12)
H13	−0.0080	0.0130	0.2253	0.055*
C14	−0.0345(5)	−0.0003(2)	0.31131(18)	0.0363(10)
C15	0.0112(5)	0.0196(2)	0.36839(18)	0.0335(10)
C16	−0.1745(7)	−0.0829(2)	0.2597(2)	0.0548(14)
H16A	−0.2161	−0.0562	0.2292	0.082*
H16B	−0.2491	−0.1164	0.2673	0.082*
H16C	−0.0737	−0.1004	0.2474	0.082*
C17	0.8195(8)	0.1607(3)	0.6369(3)	0.0743(18)
H17A	0.9296	0.1460	0.6353	0.089*
H17B	0.7959	0.1715	0.6773	0.089*
C18	0.8058(9)	0.2182(4)	0.6007(3)	0.097(2)
H18A	0.8332	0.2085	0.5609	0.145*
H18B	0.8776	0.2499	0.6155	0.145*
H18C	0.6977	0.2337	0.6024	0.145*

Experimental details

H atoms were included in the refinement at calculated positions [C–H aromatic = 0.93 Å] and treated as riding models with $U_{iso}(H) = 1.2 U_{eq}(C)$ [2].

Comment

Zinc(II) compounds attracted attention in the field of molecular materials owing to their potential applications [3, 4]. Schiff bases are an important and popular kind of ligands due to their simple synthesis, versatility, and diverse range of applications [5, 6]. Therefore, a large number of Schiff bases complexes have been synthesized and characterized [7].

The molecular structure of the title structure contains a discrete neutral $[Zn_3(L)_2(H_2O)_2Cl_2]$ ($H_2L = 1,2$ -bis(2-hydroxy-3-methoxybenzylidene)hydrazine) and two ethanol molecules. As shown in the figure, the asymmetric unit of the title structure consists of one and a half Zn(II) ions, one dianionic ligand, one chloride anion, one coordinated water molecule and one ethanol. Zn1 exhibits a distorted

square-pyramidal geometry, coordinated by three oxygens, one nitrogen atom, and one chloride anion. While Zn2 is located on an inversion center, and an octahedral sphere surrounded by two nitrogen atoms, two oxygen atoms from two ligands, and two water molecules. Bond lengths of Zn–O are in the range of 1.959(3) Å to 2.235(3) Å, and the Zn–N bond distances are 2.080(4) and 2.098(4) Å, which are normal [8]. Ethanol molecules stay in the lattices of the supramolecular structure.

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