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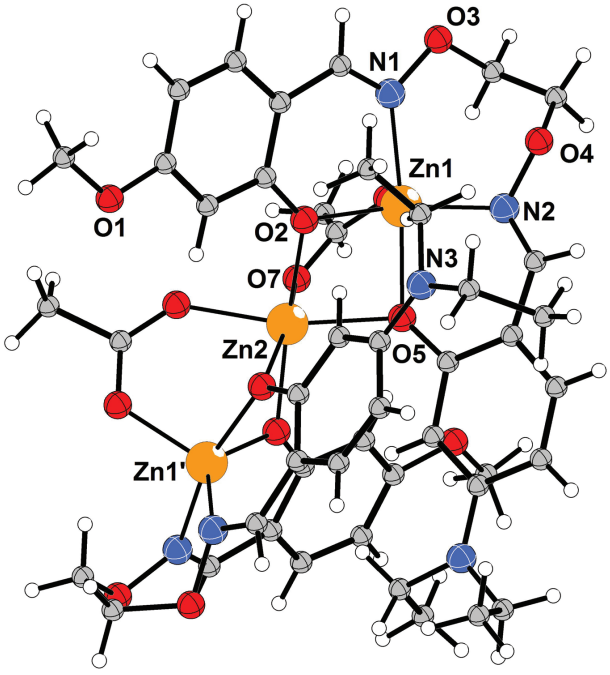
Crystal structure of bis{5-(*N*,*N*′-diethylamine)-5′-methoxy -2,2′-[ethylenediyldioxybis(nitrilomethylidyne)]diphenolato}-bis(μ_2-acetato-κ^2O:O')trizinc(II), $C_{46}H_{56}Zn_3N_6O_{14}$



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Abstract

$C_{46}H_{56}Zn_3N_6O_{14}$, monoclinic, $C2/c$ (no. 15), $a = 10.6703(7)$ Å, $b = 27.091(4)$ Å, $c = 17.9132(16)$ Å, $\beta = 96.246(8)°$, $V = 5147.4(10)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0647$, $wR_{ref}(F^2) = 0.1865$, $T = 291(1)$ K.

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The crystal structure of the title complex is shown in the figure ($' = 1 - x$, y, $0.5 - z$). Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	clear light colourless block
Size:	0.28 × 0.17 × 0.16 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.45 mm^{-1}
Diffractometer, scan mode:	SuperNova Dual Eos, ω
θ_{max}, completeness:	26.0°, 99.7%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10608, 5060, 0.062
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2787
$N(param)_{refined}$:	316
Programs:	CrysAlis^{PRO} [18], SHELXS, SHELXL [19]

Source of material

A methanol solution (3.0 mL) of zinc(II) acetate (3.29 mg 0.015 mmol) was added to a chloroform solution (2.0 mL) of H_2L (5-(N,N′-diethylamine)-5′-methoxy-2,2′-[ethylenediyldioxybis(nitrilomethylidyne)] diphenol) (3.91 mg, 0.010 mmol) at room temperature. After the mixture was stirred for 1 h the mixture was filtered off. The resulting filtrate was left undisturbed for about one week to form prismatic crystals suitable for X-ray crystallographic analysis. Yield: 37.8%. Elemental analysis – Anal. Calcd. for $C_{46}H_{56}Zn_3N_6O_{14}$: C, 49.63%; H, 5.07%; N, 7.55%. Found: C, 49.77%; H, 5.01%; N, 7.35%.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

Discussion

Salen-like compounds are important N_2O_2 chelating ligands in inorganic chemistry [1–5]. They are able to form different types of metal complexes [6–9]. These compounds can be applied to the biological system [10], used as luminescence materials [11–13] and supramolecular building units [14–17].

The title compound consists of three Zn(II), two completely deprotonated $(L)^{2-}$ units and two acetate ligands, to form the trinuclear complex (see the figure). The coordination sphere of terminal Zn(II) atom is completed by N_2O_2 donors of $(L)^{2-}$ unit and one oxygen atom of the acetate ligand. The sphere of central Zn(II) atom was completed by four phenolic

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.26347(6)	0.59405(3)	0.32454(4)	0.0471(3)
Zn2	0.5000	0.57518(4)	0.2500	0.0430(3)
O1	0.8214(4)	0.5690(2)	0.5166(3)	0.0753(16)
O2	0.4419(3)	0.57849(16)	0.3607(2)	0.0470(11)
O3	0.0921(4)	0.5647(2)	0.4497(3)	0.0708(15)
O4	0.0765(4)	0.66252(19)	0.3861(3)	0.0718(15)
O5	0.3457(3)	0.62540(15)	0.2373(2)	0.0459(11)
O6	0.1932(4)	0.53602(17)	0.2682(3)	0.0578(12)
O7	0.3725(4)	0.51796(16)	0.2204(2)	0.0545(12)
N1	0.2172(4)	0.5713(2)	0.4320(3)	0.0544(15)
N2	0.1549(5)	0.6555(2)	0.3260(3)	0.0584(16)
N3	0.4969(7)	0.7486(2)	0.0790(4)	0.097(3)
C1	0.4994(5)	0.5716(2)	0.4295(3)	0.0432(15)
C2	0.6290(5)	0.5732(2)	0.4423(3)	0.0478(16)
H2	0.6752	0.5792	0.4021	0.057*
C3	0.6922(6)	0.5661(3)	0.5124(4)	0.0537(18)
C4	0.6266(6)	0.5558(3)	0.5729(4)	0.062(2)
H4	0.6686	0.5505	0.6205	0.074*
C5	0.4983(6)	0.5536(3)	0.5606(4)	0.0582(19)
H5	0.4536	0.5463	0.6010	0.070*
C6	0.4315(5)	0.5617(2)	0.4913(4)	0.0486(16)
C7	0.8915(6)	0.5613(4)	0.5877(4)	0.090(3)
H7A	0.9799	0.5618	0.5821	0.135*
H7B	0.8694	0.5298	0.6073	0.135*
H7C	0.8725	0.5869	0.6216	0.135*
C8	0.2954(6)	0.5610(3)	0.4876(4)	0.0566(19)
H8	0.2618	0.5518	0.5313	0.068*
C9	0.0069(6)	0.5758(3)	0.3870(4)	0.0572(19)
H9A	−0.0691	0.5566	0.3898	0.069*
H9B	0.0435	0.5655	0.3422	0.069*
C10	−0.0281(6)	0.6280(3)	0.3790(4)	0.073(2)
H10A	−0.0757	0.6327	0.3302	0.087*
H10B	−0.0835	0.6360	0.4168	0.087*
C11	0.1762(6)	0.6971(3)	0.2967(4)	0.066(2)
H11	0.1315	0.7239	0.3123	0.079*
C12	0.2613(7)	0.7069(3)	0.2422(4)	0.065(2)
C13	0.3409(5)	0.6715(2)	0.2130(4)	0.0481(16)
C14	0.4167(6)	0.6865(2)	0.1581(4)	0.0534(17)
H14	0.4684	0.6632	0.1387	0.064*
C15	0.4185(7)	0.7343(3)	0.1313(5)	0.076(2)
C16	0.3410(9)	0.7699(3)	0.1609(6)	0.111(4)
H16	0.3421	0.8026	0.1454	0.133*
C17	0.2648(8)	0.7551(3)	0.2127(6)	0.101(3)
H17	0.2111	0.7784	0.2300	0.121*
C18	0.5097(12)	0.7985(4)	0.0545(8)	0.144(6)
H18A	0.4293	0.8152	0.0545	0.173*
H18B	0.5315	0.7983	0.0033	0.173*
C19	0.6090(13)	0.8272(4)	0.1034(10)	0.195(9)
H19A	0.6157	0.8599	0.0835	0.292*
H19B	0.6888	0.8107	0.1042	0.292*
H19C	0.5855	0.8292	0.1536	0.292*
C20	0.5733(10)	0.7132(4)	0.0432(7)	0.115(4)
H20A	0.6521	0.7285	0.0334	0.138*
H20B	0.5926	0.6852	0.0763	0.138*
C21	0.5059(14)	0.6971(5)	−0.0255(8)	0.181(6)
H21A	0.5583	0.6753	−0.0510	0.272*
H21B	0.4830	0.7251	−0.0568	0.272*
H21C	0.4311	0.6798	−0.0151	0.272*
C22	0.2622(6)	0.5089(3)	0.2325(4)	0.0540(17)
C23	0.2031(7)	0.4612(3)	0.2010(5)	0.082(3)
H23A	0.1771	0.4652	0.1483	0.122*
H23B	0.1311	0.4533	0.2265	0.122*
H23C	0.2637	0.4350	0.2081	0.122*

oxygen atoms which have been deprotonated in two [Zn(L)] chelates and two oxygen atoms of the acetate ligands, thus, constitute three octahedral geometries around three hexa-coordinated Zn(II) atoms, respectively. The two acetate ions coordinate to the three Zn(II) atoms via a μ_2-$\kappa^2O{:}O'$ mode.

References

1. Xu, L.; Li, N.; Zhao, L.: Crystal structure of 4-chloro-4′, 6′-dichloro-2, 2′-[propane-1,3-diyldioxybis(nitrilomethylidyne)]diphenol, $C_{17}H_{14}C_{l3}N_2O_4$. Z. Kristallogr. NCS **231** (2016) 967–968.
2. Zhao, L.; Wang, F.; An, Q. Q.; Zhao, J. X.: Crystal structure of (E)-1-(4-(((E)-3,5-dichloro-2-hydroxybenzylidene)amino)phenyl)ethan-1-one O-ethyl oxime, $C_{17}H_{16}C_{l2}N_2O_2$. Z. Kristallogr. NCS **231** (2016) 1045–1046.
3. Chai, L. Q.; Zhang, Y. L.; Ding, X. Q.: Crystal structure of 2, 2′-dinitro-2, 2′-[(1,4-butylene)dioxybis(nitrilomethylidyne)]dibenzene, $C_{18}H_{18}N_4O_6$. Z. Kristallogr. NCS **225** (2010) 685–686.
4. Sun, Y. X.; Liu, G. H.; Wang, J.; Liu, S. H.; Wang, H.: Crystal structure of bis(3-ethoxy-4-hydroxybenzaldehyde) O,O′-(butane-1,4-diyl)dioxime, $C_{22}H_{28}N_2O_6$. Z. Kristallogr. NCS **227** (2012) 123–124.
5. Wang, P.; Zhao, L.: Synthesis, structure and spectroscopic properties of the trinuclear cobalt(II) and nickel(II) complexes based on 2-hydroxynaphthaldehyde and bis(aminooxy)alkane. Spectrochim. Acta A **135** (2015) 342–350.
6. Sun, Y. X.; Gao, X. H.: Synthesis, characterization, and crystal structure of a new CuII complex with Salen-type ligand. Synth. React. Inorg. Met. Org. Nano Met. Chem. **41** (2011) 973–978.
7. Wang, P.; Zhao, L.: An infinite 2D supramolecular cobalt(II) complex based on an asymmetric Salamo-type ligand: synthesis, crystal structure, and spectral properties. Synth. React. Inorg. Met. Org. Nano Met. Chem. **46** (2016) 1095–1101.
8. Akine, S.; Taniguchi, T.; Nabeshima, T.: Cooperative formation of trinuclear zinc(II) complexes via complexation of a tetradentate oxime chelate ligand, Salamo, and zinc(II) acetate. Inorg. Chem. **43** (2004) 6142–6144.
9. Song, X. Q.; Liu, P. P.; Liu, Y. A.; Zhou, J. J.; Wang, X. L.: Two dodecanuclear heterometallic [Zn_6Ln_6] clusters constructed by a multidentate salicylamide salen-like ligand: synthesis, structure, luminescence and magnetic properties. Dalton Trans. **45** (2016) 8154–8163.

10. Wu, H. L.; Bai, Y. C.; Zhang, Y. H.; Li, Z.; Wu, M. C.; Chen, C. Y.; Zhang, J. W.: Synthesis, crystal structure, antioxidation and DNA-binding properties of a dinuclear copper(II) complex with bis(N-salicylidene)-3-oxapentane-1,5-diamine. J. Coord. Chem. **67** (2014) 3054–3066.
11. Dong, W. K.; Sun, Y. X.; Zhao, C. Y.; Dong, X. Y.; Xu, L.: Synthesis, structure and properties of supramolecular Mn(II), Co(II), Ni(II) and Zn(II) complexes containing Salen-type bisoxime ligands. Polyhedron **29** (2010) 2087–2097.
12. Dong, W. K.; Zhang, Y. P.; Zhao, C. Y.; Tang, X. L.; Lv, Z. W.; Zou, Z.: Synthesis, crystal structure and fluorescence property of a five- and six-coordinated trinuclear zinc(II) complex: {[ZnL(OAc)]$_2$Zn}.CH_3COCH_3. Chinese J. Chem. **26** (2008) 1821–1825.
13. Dong, W. K.; Ma, J. C.; Zhu, L. C.; Sun, Y. X.; Akogun, S. F.; Zhang, Y.: A series of heteromultinuclear zinc(II)-lanthanide(III) complexes based on 3-MeOsalamo: syntheses, structural characterizations, and luminescent properties. Cryst. Growth Des. **16** (2016) 6903–6914.
14. Yu, T. Z.; Zhang, K.; Zhao, Y. L.; Yang, C. H.; Zhang, H.; Fan, D. W.; Dong, W. K.: A new trinuclear zinc(II) complex possessing five- and six-coordinated central ions and its photoluminescent property. Inorg. Chem. Commun. **10** (2007) 401–403.
15. Yu, T. Z.; Zhang, K.; Zhao, Y. L.; Yang, C. H.; Zhang, H.; Qian, L.; Fan, D. W.; Dong, W. K.; Chen, L. L.; Qiu, Y. Q.: Synthesis, crystal structure and photoluminescent properties of an aromatic bridged Schiff base ligand and its zinc complex. Inorg. Chim. Acta **361** (2008) 233–240.
16. Song, X. Q.; Peng, Y. J.; Chen, G. Q.; Wang, X. R.; Liu, P. P.; Xu, W. Y.: Substituted group-directed assembly of Zn(II) coordination complexes based on two new structural related pyrazolone based Salen ligands: syntheses, structures and fluorescence properties, Inorg. Chim. Acta **427** (2015) 13–21.
17. Akine, S.; Nabeshima, T.: Cyclic and acyclic oligo(N_2O_2) ligands for cooperative multi-metal complexation. Dalton Trans. **47** (2009) 10395–10408.
18. Agilent. CrysAlis^{PRO}. Agilent Technologies Ltd, Yarnton, Oxfordshire, England, 2014.
19. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.