Zong-Li Ren, Zhi-Li Wei, Lan Wang, Jian-Chun Ma and Wen-Kui Dong*

Synthesis and crystal structure of 2,2'-ethylenedioxybis(benzimide)-2,2'-bis[O-(1-propyloxyamide)]oxime-4,4',6,6'-tetrachlorodiphenol, $C_{36}H_{34}Cl_4N_4O_8$

Table 1: Data collection and handling.

Crystal:	Clear light green needle
Size:	0.26 × 0.22 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.37 mm ⁻¹
Diffractometer, scan mode:	SuperNova, dual, ω scans
$\theta_{\max}$, completeness:	26.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8189, 3701, 0.012
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2195
$N(\text{param})_{\text{refined}}$:	235
Programs:	CrysAlis ^{PRO} [1], SHELX [2], Olex2 [3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.15219(11)	1.4807(4)	0.59708(13)	0.0671(7)
C2	0.16715(11)	1.6609(4)	0.58401(13)	0.0706(8)
C3	0.12762(11)	1.7811(4)	0.52123(13)	0.0700(8)
H3	0.138040	1.902154	0.513084	0.084*
C4	0.07230(11)	1.7207(4)	0.47035(13)	0.0686(7)
C5	0.05695(11)	1.5413(4)	0.48281(13)	0.0661(7)
H5	0.019819	1.501223	0.448730	0.079*
C6	0.09656(11)	1.4208(4)	0.54577(13)	0.0654(7)
C7	0.07855(11)	1.2414(4)	0.55704(13)	0.0686(7)
H7	0.041398	1.201844	0.520972	0.082*
C8	0.12879(11)	0.8751(4)	0.68994(14)	0.0734(8)
H8A	0.142845	0.972804	0.729936	0.088*
H8B	0.159698	0.822844	0.697226	0.088*
C9	0.09822(12)	0.7004(4)	0.68934(14)	0.0721(8)
H9A	0.068162	0.755368	0.683525	0.086*
H9B	0.082349	0.609006	0.647063	0.086*
C10	0.13741(12)	0.5838(4)	0.76117(14)	0.0795(9)
H10A	0.167218	0.524009	0.767312	0.095*
H10B	0.153318	0.672191	0.804155	0.095*
C11	0.11024(11)	0.1593(4)	0.81554(14)	0.0667(7)
H11	0.073114	0.138763	0.771719	0.080*
C12	0.13708(11)	0.0218(4)	0.87881(13)	0.0628(7)
C13	0.19006(11)	0.0630(4)	0.94719(13)	0.0701(8)
H13	0.208270	0.182056	0.952557	0.084*
C14	0.21584(12)	−0.0674(4)	1.00646(15)	0.0823(9)
H14	0.251862	−0.039185	1.051751	0.099*
C15	0.18842(12)	−0.2452(4)	0.99986(15)	0.0772(8)
H15	0.205713	−0.333207	1.040948	0.093*
C16	0.13548(11)	−0.2879(4)	0.93168(14)	0.0703(7)

<https://doi.org/10.1515/ncrs-2017-0419>

Received February 5, 2018; accepted May 29, 2018; available online June 13, 2018

Abstract

$C_{36}H_{34}Cl_4N_4O_8$, monoclinic, $C2/c$ (no. 15), $a = 33.048(10)$ Å, $b = 6.4688(7)$ Å, $c = 24.301(7)$ Å, $\beta = 133.30(5)^\circ$, $Z = 4$, $V = 3781(3)$ Å³, $R_{\text{gt}}(F) = 0.0477$, $wR_{\text{ref}}(F^2) = 0.1016$, $T = 293(2)$ K.

CCDC no.: 1811864

The crystal structure is shown in the figure. 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 material

2,2'-(Ethylenedioxy)bisbenzaldehyde was prepared according to a literature procedure [4]; 1,3-bis(aminoxy)propane

*Corresponding author: Wen-Kui Dong, School of Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou 730070, P.R. China, e-mail: dongwk@126.com

Zong-Li Ren, Zhi-Li Wei, Lan Wang and Jian-Chun Ma: School of Chemical and Biological Engineering, Lanzhou Jiaotong University, Lanzhou 730070, P.R. China

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H16	0.117310	−0.406612	0.926704	0.084*
C17	0.10915(11)	−0.1557(4)	0.87061(13)	0.0638(7)
C18	0.02580(10)	−0.3597(3)	0.78907(13)	0.0684(7)
H18A	0.046015	−0.486772	0.801013	0.082*
H18B	0.018632	−0.349508	0.821579	0.082*
Cl1	0.02396(3)	1.86723(10)	0.39303(4)	0.0821(3)
Cl2	0.23595(3)	1.73453(10)	0.64494(3)	0.0831(3)
N1	0.11391(10)	1.1325(3)	0.61766(11)	0.0703(6)
N2	0.13647(9)	0.3057(3)	0.81918(11)	0.0692(6)
O1	0.19343(8)	1.3770(3)	0.66015(9)	0.0780(6)
H1B	0.184842	1.256410	0.671146	0.094*
O2	0.08888(8)	0.9685(3)	0.61861(9)	0.0751(5)
O3	0.10232(8)	0.4216(2)	0.75305(9)	0.0739(5)
O4	0.05793(7)	−0.1844(2)	0.80077(9)	0.0692(5)

and 2-[*O*-(1-propyloxyamide)]oxime-4,6-dichlorophenol were synthesized according to a reported method [5, 6]. 2-[*O*-(1-propyloxyamide)]oxime-4,6-dichlorophenol (114.0 mg, 0.60 mmol) in ethanol (10 mL) was added slowly to a solution of 1,3-bis(aminoxy)propane (46.0 mg, 0.43 mmol) in ethanol (10 mL), and was stirred for 6 h at 60 °C. After cooling to room temperature, the product was distilled under reduced pressure. The solution of the product (116.0 mg, 0.43 mmol) in ethanol (10 mL) was added slowly to a solution of 2,2'-(ethylenedioxy)bis(benzaldehyde) (240.0 mg, 0.86 mmol) in ethanol (10 mL), and stirred for 6 h at the same temperature. Precipitates were collected on a suction filter to afford the title compound (286.0 mg, 80.3%). The solution of the title compound in acetone and ethanol was allowed to stand at room temperature for several days, resulting in light green crystals; m.p. 130–131 °C. Elemental analysis: Anal. Calcd. for C₃₆H₃₄Cl₄N₄O₈: C, 53.00%; H, 4.71%; N, 7.27%. Found: C, 53.33%; H, 4.85%; N, 7.05%.

Experimental details

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

Discussion

Salamo-type compounds have drawn much attention in the recent years because these compounds can accommodate one, two or more metal centers and form homo- and heteronuclear metal complexes with interesting properties [7–9]. In addition, they can be used to obtain non-linear optical materials, models of biological systems, magnetic properties, and are building blocks for supramolecular structures [10–14]. The crystal structure of the title compound was determined

by X-ray crystallography (*cf.* the figure). The crystal structure of the title compound is only built up by C₂ symmetrical molecule, in which all bond lengths are in normal ranges. There is one intramolecular hydrogen bond (O1–H1B···N1) [15, 16].

References

1. CrysAlis^{PRO} software system, version 1.171.37.33, Agilent Technologies UK Ltd, Oxford, UK (2011).
2. Sheldrick, G. M.: A short history of SHELX. *Acta Crystallogr. A* **64** (2008) 112–122.
3. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
4. Ma, J. C.; Dong, X. Y.; Dong, W. K.; Zhang, Y.; Zhu, L. C.; Zhang, J. T.: An unexpected dinuclear Cu(II) complex with a bis(Salamo) chelating ligand: synthesis, crystal structure, and photophysical properties. *J. Coord. Chem.* **69** (2016) 149–159.
5. Xu, L.; Li, N.; Zhao, L.: Crystal structure of 4-chloro-4',6'-dichloro-2,2'-[propane-1,3-diylidioxybis(nitrilomethylidyne)]-diphenol, C₁₇H₁₄Cl₃N₂O₄. *Z. Kristallogr. NCS* **231** (2016) 967–968.
6. Chen, L.; Dong, W. K.; Zhang, H.; Zhang, Y.; Sun, Y. X.: Structural variation and luminescence properties of tri- and dinuclear Cu^{II} and Zn^{II} complexes constructed from a naphthalenediol-based bis(Salamo)-type ligand. *Cryst. Growth Des.* **17** (2017) 3636–3648.
7. Song, X. Q.; Liu, P. P.; Liu, Y. A.; Zhou, J. J.; Wang, X. L.: Two dodecanuclear heterometallic [Zn₆Ln₆] clusters constructed by a multidentate salicylamide salen-like ligand: synthesis, structure, luminescence and magnetic properties. *Dalton Trans.* **45** (2016) 8154–8163.
8. 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.
9. Wang, P.; Zhao, L.: Synthesis, structure and spectroscopic properties of the trinuclear cobalt(II) and nickel(II) complexes based on 2-hydroxynaphthaldehyde and bis(aminoxy)alkane. *Spectrochim. Acta Part A* **135** (2015) 342–350.
10. 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.
11. Sun, Y. X.; Gao, X. H.: Synthesis, characterization, and crystal structure of a new Cu^{II} complex with Salen-type ligand. *Synth. React. Inorg. Met. Org. Nano-Met. Chem.* **41** (2011) 973–978.
12. 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.
13. Song, X. Q.; Liu, P. P.; Xiao, Z. R.; Li, X.; Liu, Y. A.: Four polynuclear complexes based on a versatile salicylamide

- salen-like ligand: synthesis, structural variations and magnetic properties. *Inorg. Chim. Acta* **438** (2015) 232–244.
14. Liu, Y. A.; Wang, C. Y.; Zhang, M.; Song, X. Q.: Structures and magnetic properties of cyclic heterometallic tetranuclear clusters. *Polyhedron* **127** (2017) 278–286.
 15. Sun, Y. X.; Liu, G. H.; Wang, J.; Liu, S. H.; Wang, G.: 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.
 16. Liu, P. P.; Wang, C. Y.; Zhang, M.; Song, X. Q.: Pentanuclear sandwich-type Zn^{II} - Ln^{III} clusters based on a new Salen-like salicylamide ligand: structure, near-infrared emission and magnetic properties. *Polyhedron* **129** (2017) 133–140.