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Synthesis and crystal structure of (1*E*,3*E*)-2-hydroxy-5-methylisophthalaldehyde *O,O*-di(2-(((*E*)-(2-hydroxynaphthalen-1-yl)methylene)amino)oxy)ethyl) dioxime, C₃₅H₃₂N₄O₇

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Abstract

C₃₅H₃₂N₄O₇, triclinic, $P\bar{1}$ (no. 2), $a = 9.3625(6)$ Å, $b = 12.7558(7)$ Å, $c = 14.5965(9)$ Å, $\alpha = 68.833(2)^\circ$, $\beta = 80.891(2)^\circ$, $\gamma = 69.242(2)^\circ$, $V = 1519.15(16)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0447$, $wR_{\text{ref}}(F^2) = 0.1500$, $T = 296(2)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The starting materials 1,2-bis(aminoxyl)ethane and 2-[*O*-(1-ethoxyamide)]oxime-2-naphthol can also be prepared according to similar reports [4]. To an ethanol solution (10 mL) of 2-hydroxy-5-methyl-1,3-benzenedialdehyde

Table 1: Data collection and handling.

Crystal:	Pale-yellow block
Size:	0.27 × 0.25 × 0.22 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max} , completeness:	25.0°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11069, 5310, 0.036
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4002
$N(\text{param})_{\text{refined}}$:	419
Programs:	Bruker [1], Olex2 [2], SHELX [3]

(164 mg, 1 mmol) was added an ethanol solution (20 mL) of 2-[*O*-(1-ethoxyamide)]oxime-2-naphthol (492 mg, 2 mmol). The mixed solution was heated under refluxing for 4 h, then the solution was concentrated under reduced pressure and recrystallized from trichloromethane/ethanol (*v/v*, 1:5). Yield: 54.5%. **Elemental analysis:** Anal. Calcd. for C₃₅H₃₂N₄O₇(%): C, 67.73; H, 5.20; N, 9.03. Found (%): C, 67.85; H, 5.09; N, 8.92. A small amount of the title compound was dissolved in a methylene chloride/methanol (*v/v*, 2:5) solution (7 mL), and the filtrate was left undisturbed for about one week to form pale-yellow block crystals.

Experimental details

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

Discussion

As is generally known, salen compound and its derivatives are a class of very important N₂O₂ chelating ligands in inorganic chemistry [5], their metal complexes not only show a variety of catalytic and optical properties [6], but also have some advantages in the field of antibiosis, treatment of tumors, biological activities and so on [7]. Salamo compounds form metal complexes with interesting properties. Now, the synthesis of structurally-specific salamo metal complexes with superior properties for applications has become an important chemical field [8, 9].

The current work reports the synthesis of the title compound. All bond lengths are in normal ranges [10]. X-ray crystallographic data reveals that the title compound crystallizes in the triclinic, space group $P\bar{1}$, and the overall molecule

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C1	0.2740(3)	0.75453(17)	0.50150(16)	0.0282(5)
C2	0.3722(3)	0.76707(18)	0.55753(17)	0.0318(5)
H2	0.377697	0.841826	0.547449	0.038*
C3	0.4588(3)	0.67054(19)	0.62620(16)	0.0301(5)
H3	0.521561	0.680233	0.663847	0.036*
C4	0.4556(2)	0.55499(18)	0.64170(15)	0.0259(5)
C5	0.5538(3)	0.45324(18)	0.70817(16)	0.0300(5)
H5	0.618438	0.462494	0.744809	0.036*
C6	0.5557(3)	0.34167(19)	0.71972(16)	0.0320(5)
H6	0.622617	0.275533	0.762650	0.038*
C7	0.4558(3)	0.32782(18)	0.66624(16)	0.0318(5)
H7	0.456650	0.251907	0.674180	0.038*
C8	0.3577(2)	0.42352(17)	0.60297(16)	0.0266(5)
H8	0.290901	0.411886	0.569556	0.032*
C9	0.3548(2)	0.54112(17)	0.58673(15)	0.0230(5)
C10	0.2600(2)	0.64453(17)	0.51634(15)	0.0237(5)
C11	0.1477(2)	0.63527(17)	0.46331(15)	0.0252(5)
H11	0.129992	0.563156	0.480343	0.030*
C12	−0.1103(3)	0.79848(18)	0.27607(17)	0.0331(5)
H12A	−0.102315	0.870509	0.279993	0.040*
H12B	−0.218026	0.807923	0.277745	0.040*
C13	−0.0393(3)	0.78361(18)	0.18085(17)	0.0328(5)
H13A	−0.069470	0.859935	0.129012	0.039*
H13B	0.071020	0.756755	0.184517	0.039*
C14	−0.0666(3)	0.50879(18)	0.21698(16)	0.0293(5)
H14	−0.147468	0.531946	0.176631	0.035*
C15	−0.0038(2)	0.38449(17)	0.27799(15)	0.0246(5)
C16	−0.0517(3)	0.29950(18)	0.26418(16)	0.0290(5)
H16	−0.121340	0.323764	0.215781	0.035*
C17	0.0010(3)	0.17973(18)	0.32023(16)	0.0291(5)
C18	0.1039(2)	0.14602(17)	0.39273(15)	0.0253(5)
H18	0.140470	0.066377	0.430914	0.030*
C19	0.1543(2)	0.22774(17)	0.41013(15)	0.0230(5)
C20	0.1003(2)	0.34778(17)	0.35186(15)	0.0232(5)
C21	−0.0526(3)	0.08991(19)	0.30332(19)	0.0420(7)
H21A	−0.143449	0.130291	0.265813	0.063*
H21B	−0.074379	0.036570	0.365478	0.063*
H21C	0.025892	0.045713	0.267907	0.063*
C22	0.2593(3)	0.19266(17)	0.48840(15)	0.0256(5)
H22	0.286573	0.250844	0.498603	0.031*
C23	0.4650(3)	−0.03924(17)	0.67766(16)	0.0274(5)
H23A	0.520226	−0.094466	0.642754	0.033*
H23B	0.379101	−0.062896	0.714106	0.033*
C24	0.5679(2)	−0.03840(17)	0.74589(15)	0.0264(5)
H24A	0.620743	−0.118892	0.786200	0.032*
H24B	0.643494	−0.001899	0.708789	0.032*
C25	0.4894(2)	0.12087(17)	0.90608(14)	0.0242(5)
H25	0.383896	0.153081	0.900457	0.029*
C26	0.5647(2)	0.15566(17)	0.96514(14)	0.0242(5)
C27	0.7219(3)	0.10693(18)	0.97643(16)	0.0275(5)
C28	0.7970(3)	0.1447(2)	1.02896(17)	0.0356(6)
H28	0.901650	0.109304	1.037597	0.043*
C29	0.7173(3)	0.2321(2)	1.06681(17)	0.0374(6)
H29	0.769381	0.258776	1.098213	0.045*
C30	0.5563(3)	0.28404(19)	1.05980(16)	0.0318(5)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C31	0.4738(3)	0.3726(2)	1.10191(19)	0.0448(7)
H31	0.526013	0.398460	1.133971	0.054*
C32	0.3187(3)	0.4211(2)	1.0965(2)	0.0497(7)
H32	0.265631	0.480343	1.123773	0.060*
C33	0.2404(3)	0.3805(2)	1.04933(19)	0.0455(7)
H33	0.134450	0.411961	1.046910	0.055*
C34	0.3174(3)	0.2951(2)	1.00667(16)	0.0338(5)
H34	0.262850	0.270015	0.975558	0.041*
C35	0.4782(3)	0.24487(18)	1.00924(15)	0.0265(5)
N1	0.0733(2)	0.72409(15)	0.39411(13)	0.0277(4)
N2	−0.0103(2)	0.58537(14)	0.21910(13)	0.0280(4)
N3	0.3142(2)	0.08539(14)	0.54279(12)	0.0255(4)
N4	0.5661(2)	0.04671(14)	0.86184(12)	0.0250(4)
O1	0.1957(2)	0.85582(12)	0.43273(12)	0.0370(4)
H1	0.143077	0.840563	0.402604	0.055*
O2	−0.04058(18)	0.69986(13)	0.36053(11)	0.0354(4)
O3	−0.08315(19)	0.70003(12)	0.15647(11)	0.0347(4)
O4	0.14830(19)	0.42582(12)	0.37205(11)	0.0319(4)
H4	0.111597	0.492685	0.333134	0.048*
O5	0.41295(17)	0.07882(12)	0.61031(10)	0.0285(4)
O6	0.47299(16)	0.02884(12)	0.80667(10)	0.0267(4)
O7	0.81160(18)	0.02206(14)	0.93834(12)	0.0381(4)
H7A	0.759148	0.007039	0.907906	0.057*

of the title ligand has a C-type structure (see the figure). In addition, there are three intramolecular O1—H1···N1, O4—H4···N2, and O7—H7A···N4 hydrogen bonds and some weak intermolecular C—H···O hydrogen bonds.

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