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Crystal structure of (*E*)-2-hydroxybenzaldehyde *O*-(2-(((*E*)-(4-(dimethylamino)benzylidene)amino)oxy)ethyl)oxime, C₁₈H₂₁N₃O₃

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Abstract

C₁₈H₂₁N₃O₃, monoclinic, $P2_1/n$, $a = 5.3025(3)$ Å, $b = 9.7824(7)$ Å, $c = 33.3847(19)$ Å, $\beta = 93.699(6)^\circ$, $V = 1728.10(19)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0677$, $wR_{\text{ref}}(F^2) = 0.1892$, $T = 290.7(3)$ K.

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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

All reagents are analytically pure, 1,2-bis(aminoxy)ethane and salicylaldehyde *O*-(1-ethyloxyamide)oxime were prepared according the methods reported earlier [4–6]. 4-(*N,N'*-dimethylamino)benzaldehyde (229.75 mg, 1.54 mmol) in ethanol (50 mL) was slowly dropped to a solution of the salicylaldehyde *O*-(1-ethyloxyamide)oxime (301.84 mg, 1.54 mmol) in ethanol (50 mL) with the mixture stirred at 328 K for 24 h. After vacuum distillation, dilution with ethanol/hexane (8 mL, 1:4) and cooling in freezer for 12 h, the precipitate was filtered, purified by recrystallization with ethanol/hexane (1:4) and dried in vacuum yielding the white powder. Yield: 75.5%, m.p. 347.15–348.15 K. Elemental

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.27 × 0.14 × 0.05 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ^{−1}
Diffractometer, scan mode:	SuperNova, ω -scans
θ_{max} , completeness:	25.7°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7044, 3277, 0.035
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1775
$N(\text{param})_{\text{refined}}$:	211
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>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	−0.2019(4)	0.2529(3)	0.11029(6)	0.0797(7)
O2	0.0003(4)	0.2511(2)	0.03252(6)	0.0782(7)
O3	0.5505(5)	0.1132(2)	−0.03188(7)	0.0872(7)
H3	0.4373	0.1184	−0.0163	0.131*
N1	0.7311(6)	0.2782(4)	0.29939(9)	0.1065(12)
N2	0.0000(5)	0.2187(3)	0.13851(7)	0.0731(8)
N3	0.1971(4)	0.2346(3)	0.00699(7)	0.0609(7)
C1	0.5677(6)	0.2899(4)	0.26551(9)	0.0794(10)
C2	0.3824(9)	0.3882(4)	0.26234(11)	0.1012(13)
H2	0.3713	0.4516	0.2829	0.121*
C3	0.2127(9)	0.3949(4)	0.22946(12)	0.1050(14)
H3A	0.0903	0.4631	0.2284	0.126*
C4	0.2180(6)	0.3038(4)	0.19788(9)	0.0721(9)
C5	0.4099(6)	0.2106(5)	0.20039(10)	0.0920(12)
H5	0.4259	0.1502	0.1792	0.110*
C6	0.5802(6)	0.2026(5)	0.23307(10)	0.1003(14)
H6	0.7071	0.1369	0.2334	0.120*
C7	0.0265(7)	0.3104(4)	0.16476(10)	0.0772(10)
H7	−0.0808	0.3857	0.1629	0.093*
C8	−0.2353(6)	0.1466(4)	0.08160(9)	0.0741(10)
H8A	−0.3868	0.1652	0.0646	0.089*
H8B	−0.2617	0.0614	0.0956	0.089*
C9	−0.0198(6)	0.1289(3)	0.05569(9)	0.0701(9)
H9A	0.1353	0.1135	0.0721	0.084*
H9B	−0.0491	0.0508	0.0381	0.084*
C10	0.2313(6)	0.3395(3)	−0.01402(10)	0.0769(9)
H10	0.1287	0.4154	−0.0109	0.092*
C11	0.4236(6)	0.3464(3)	−0.04269(8)	0.0588(8)
C12	0.5742(5)	0.2355(3)	−0.05083(8)	0.0611(8)
C13	0.7535(7)	0.2481(4)	−0.07867(11)	0.0913(12)
H13	0.8535	0.1735	−0.0844	0.110*

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C14	0.7853(7)	0.3696(5)	−0.09788(10)	0.0886(12)
H14	0.9097	0.3771	−0.1162	0.106*
C15	0.6384(7)	0.4796(4)	−0.09075(10)	0.0858(11)
H15	0.6604	0.5619	−0.1041	0.103*
C16	0.4565(7)	0.4670(4)	−0.06344(10)	0.0850(10)
H16	0.3530	0.5413	−0.0588	0.102*
C17	0.7135(9)	0.3686(5)	0.33270(11)	0.1241(17)
H17A	0.7691	0.4581	0.3255	0.186*
H17B	0.8182	0.3353	0.3551	0.186*
H17C	0.5412	0.3731	0.3399	0.186*
C18	0.9132(8)	0.1696(5)	0.30321(13)	0.1288(18)
H18A	0.8270	0.0833	0.3029	0.193*
H18B	1.0133	0.1795	0.3280	0.193*
H18C	1.0209	0.1734	0.2812	0.193*

analysis-Anal. Calcd. for C₁₈H₂₁N₃O₃: C, 66.04%; H, 6.47%; N, 12.84%. Found: C, 66.33%; H, 6.55%; N, 12.60%. Prism colorless crystals of the title compound were obtained by slow evaporation of the title compound (3.27 mg, 0.01 mmol) from dichloromethane/hexane/ethanol (4 mL, 1:1:2) at room temperature after about 2 weeks.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions so as to be constrained to ride on their parent atoms.

Discussion

Proved by endless scientific researches to be of multifunction, of novel bonding and of good stability [7–10], and having been studied widely in many fields of chemistry and material science [11–14], the oxime type compounds can be applied to obtain optical materials, biological systems, interesting magnetic properties and building blocks for cyclic supramolecular structures [15–17], and is a hot topic in contemporary coordination chemistry.

The title compound is only built up by the molecule shown in the figure, in which all bond lengths are within the normal ranges. There are intramolecular hydrogen bond (O3–H3···N3), C–H··· π interactions (C3–H3A···Cg1) (C1–C6), C18–H18B···Cg2 (C11–C16) and C10–H10B···Cg2 (C11–C16). Because of the weak interactions, the molecules are interlinked to form an infinite 3D structure.

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