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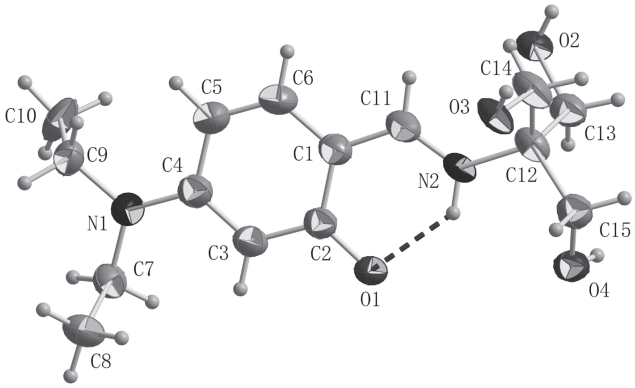
Crystal structure of (*E*)-5-(diethylamino)-2-(((1,1,2-trihydroxyethyl)iminio)methyl)phenolate, $C_{15}H_{24}N_2O_4$



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Abstract

$C_{15}H_{24}N_2O_4$, orthorhombic, $Pca2_1$, $a = 18.528(6)$ Å, $b = 8.123(2)$ Å, $c = 20.797(6)$ Å, $V = 3129.9(16)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0396$, $wR_{ref}(F^2) = 0.1082$, $T = 296(2)$ K.

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The organic cation of the title 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

A mixture of Tris(hydroxymethyl)methyl aminomethane (1.21 g, 10 mmol) and 4-diethylaminosalicylaldehyde (1.93 g, 10 mmol), in anhydrous ethanol (30 mL) was heated at 373 K for about 3 h. Then the solvent was removed under

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Table 1: Data collection and handling.

Crystal:	Block
Size:	0.34 × 0.32 × 0.30 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, *φ* & *ω*
θ_{max}:	25.0°
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	28099, 5520, 0.030
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 5009
$N(param)_{refined}$:	389
Programs:	SAINT [6], SHELX [7] OLEX2 [8]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.11258(11)	−1.5999(3)	−0.18701(11)	0.0364(5)
N1	−0.27117(10)	−1.5313(2)	−0.04469(9)	0.0425(4)
O1	−0.17877(8)	−1.4237(2)	−0.25550(8)	0.0425(4)
C2	−0.17327(11)	−1.4951(2)	−0.19980(10)	0.0320(4)
H2	−0.0920	−1.5012	−0.3010	0.041*
H2A	0.1309	−1.5852	−0.2654	0.062*
N2	−0.05562(8)	−1.5602(2)	−0.28971(9)	0.0344(4)
O2	0.09973(8)	−1.51574(19)	−0.25843(8)	0.0415(4)
C3	−0.22536(11)	−1.4750(3)	−0.15125(11)	0.0350(5)
H3	−0.0126	−1.9519	−0.3672	0.073*
H3A	−0.2646	−1.4068	−0.1593	0.042*
N3	−0.51140(10)	−1.0872(2)	−0.60567(9)	0.0428(4)
O3	−0.02868(8)	−1.86377(18)	−0.35428(9)	0.0487(4)
C4	−0.22084(11)	−1.5527(3)	−0.09173(10)	0.0363(5)
H4	−0.3556	−0.9047	−0.3462	0.039*
H4A	−0.0240	−1.2911	−0.3960	0.079*
N4	−0.31754(8)	−0.9635(2)	−0.35184(9)	0.0328(4)
O4	−0.05638(9)	−1.3596(2)	−0.39843(10)	0.0530(4)
C5	−0.15992(13)	−1.6587(3)	−0.08054(12)	0.0513(6)
H5	−0.1554	−1.7137	−0.0415	0.062*
O5	−0.44725(8)	−0.87590(17)	−0.39983(8)	0.0375(3)
C6	−0.10954(12)	−1.6786(3)	−0.12625(12)	0.0487(6)
H6	−0.2571	−1.3263	−0.2791	0.059*
H6A	−0.0707	−1.7475	−0.1176	0.058*
O6	−0.29299(7)	−1.27128(17)	−0.28751(8)	0.0393(3)
C7	−0.32987(13)	−1.4129(3)	−0.05086(12)	0.0452(5)
H7	−0.1301	−0.8676	−0.3724	0.102*
H7A	−0.3138	−1.3220	−0.0775	0.054*
H7B	−0.3412	−1.3692	−0.0086	0.054*
O7	−0.17393(9)	−0.8796(3)	−0.36954(11)	0.0679(6)
C8	−0.39777(14)	−1.4852(4)	−0.07984(16)	0.0619(7)
H8	−0.3523	−0.6946	−0.2371	0.077*
H8A	−0.3881	−1.5205	−0.1230	0.093*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H8B	−0.4351	−1.4032	−0.0803	0.093*
H8C	−0.4132	−1.5777	−0.0546	0.093*
O8	−0.34396(9)	−0.7935(2)	−0.23962(9)	0.0511(4)
C9	−0.26939(13)	−1.6224(3)	0.01574(12)	0.0499(6)
H9A	−0.2472	−1.7288	0.0083	0.060*
H9B	−0.3185	−1.6415	0.0300	0.060*
C10	−0.2288(3)	−1.5354(6)	0.06820(16)	0.0999(15)
H10A	−0.1816	−1.5057	0.0529	0.150*
H10B	−0.2242	−1.6070	0.1047	0.150*
H10C	−0.2545	−1.4378	0.0805	0.150*
C11	−0.05792(11)	−1.6256(3)	−0.23215(11)	0.0377(5)
H11	−0.0201	−1.6946	−0.2204	0.045*
C12	0.00268(10)	−1.5775(3)	−0.33657(10)	0.0355(5)
C13	0.06697(11)	−1.4690(3)	−0.31726(12)	0.0382(5)
H13A	0.1030	−1.4734	−0.3511	0.046*
H13B	0.0507	−1.3559	−0.3138	0.046*
C14	0.02878(11)	−1.7555(3)	−0.34138(13)	0.0414(5)
H14A	0.0644	−1.7641	−0.3754	0.050*
H14B	0.0517	−1.7869	−0.3013	0.050*
C15	−0.02615(13)	−1.5184(3)	−0.40131(13)	0.0462(5)
H15A	0.0129	−1.5184	−0.4323	0.055*
H15B	−0.0626	−1.5951	−0.4163	0.055*
C16	−0.36061(11)	−1.0352(3)	−0.45735(10)	0.0358(5)
C17	−0.42964(11)	−0.9559(2)	−0.45191(10)	0.0315(4)
C18	−0.47793(11)	−0.9722(3)	−0.50326(10)	0.0352(5)
H18	−0.5218	−0.9167	−0.5013	0.042*
C19	−0.46293(11)	−1.0683(3)	−0.55738(10)	0.0370(5)
C20	−0.39450(12)	−1.1496(3)	−0.56101(11)	0.0461(6)
H20	−0.3832	−1.2148	−0.5964	0.055*
C21	−0.34645(12)	−1.1308(3)	−0.51272(11)	0.0459(6)
H21	−0.3020	−1.1831	−0.5161	0.055*
C22	−0.57628(13)	−0.9872(3)	−0.60904(13)	0.0488(6)
H22A	−0.5881	−0.9691	−0.6539	0.059*
H22B	−0.5660	−0.8806	−0.5901	0.059*
C23	−0.64085(15)	−1.0583(4)	−0.57631(18)	0.0688(8)
H23A	−0.6542	−1.1594	−0.5970	0.103*
H23B	−0.6802	−0.9818	−0.5788	0.103*
H23C	−0.6296	−1.0796	−0.5320	0.103*
C24	−0.49889(13)	−1.1982(3)	−0.66007(11)	0.0469(5)
H24A	−0.5451	−1.2346	−0.6768	0.056*
H24B	−0.4731	−1.2946	−0.6449	0.056*
C25	−0.4566(2)	−1.1203(4)	−0.71354(15)	0.0698(9)
H25A	−0.4463	−1.2014	−0.7458	0.105*
H25B	−0.4122	−1.0771	−0.6968	0.105*
H25C	−0.4843	−1.0326	−0.7322	0.105*
C26	−0.30988(11)	−1.0348(2)	−0.40741(11)	0.0362(5)
H26	−0.2668	−1.0906	−0.4147	0.043*
C27	−0.26658(10)	−0.9752(2)	−0.29812(10)	0.0354(5)
C28	−0.28930(12)	−1.1203(3)	−0.25488(11)	0.0379(5)
H28A	−0.2550	−1.1302	−0.2198	0.045*
H28B	−0.3362	−1.0964	−0.2364	0.045*
C29	−0.18939(11)	−0.9996(3)	−0.32215(13)	0.0460(6)
H29A	−0.1558	−0.9891	−0.2866	0.055*
H29B	−0.1842	−1.1090	−0.3403	0.055*
C30	−0.27238(12)	−0.8177(3)	−0.25927(12)	0.0450(5)
H30A	−0.2413	−0.8245	−0.2219	0.054*
H30B	−0.2567	−0.7251	−0.2852	0.054*

reduced pressure. The resulting residue was purified by recrystallization from ethanol to obtain a crystal.

Experimental details

All hydrogen atoms were introduced by a riding model. The methyl groups were idealized and refined using rigid groups allowed to rotate about the N—C bond (AFIX 137 option of the SHELXL-2013 program) [7]. The U_{iso} values of the hydrogen atoms of the methyl groups were set to $1.5U_{eq}$(C) and the U_{iso} values of all other hydrogen atons were set to $1.2U_{eq}$(C).

Discussion

o-Hydroxy Schiff bases belong to an important class of organic compounds due to their crystallography and biological activity [1–5]. From the crystallography point of view, o-hydroxy Schiff-bases are of great interest to the investigators mostly due to the intramolecular H-bond.

The asymmetric unit of the title compound contains two molecules. One of them is shown in the figure. Their geometric parameters are very similar. They both are in the zwitterionic form in the solid state, with the H atom of the phenol group being transferred to the imine N atom. Consequently, they both adopt an *E*-configuration about the central C = N double bond.

In both molecules an intramolecular NH···O hydrogen bond is present. Adjacent molecules are connected *via classical* NH···O hydrogen bonds to form a 2D network parallel to the *ab* plane.

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