Su Wenhui, Tian Dayong, Yu Zhichao, Yang Liguang* and Niu Yongsheng
Crystal structure of 2-ethoxy-6-((*E*)-((3-(((*E*)-3-ethoxy-2-hydroxybenzylidene)amino)-2-hydroxypropyl)iminio)methyl)phenolate, $C_{21}H_{26}N_2O_5$

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Abstract

$C_{21}H_{26}N_2O_5$, monoclinic, $P2_1/c$ (no. 14), $a = 19.779(9)$ Å, $b = 5.332(2)$ Å, $c = 18.945(8)$ Å, $\beta = 94.572(6)^\circ$, $V = 1991.4(15)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0425$, $wR_{\text{ref}}(F^2) = 0.1179$, $T = 298(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title molecule was synthesized by refluxing 1,3-diamino-2-propanol (0.09 mL, 1 mmol) and 3-ethoxy salicylaldehyde (0.332 g, 2 mmol). The yield is ca. 32%. **IR(KBr):** 3412(s), 1638(s), 1518(s), 1497(s), 1448(s), 1356(s), 1245(s), 1174(s), 1113(s), 1073(s), 1005(s), 924(s), 894(s), 835(m), 773(m), 716(m), 619(m), 493(m) cm⁻¹. **Elemental analysis:** Found: C, 65.25; H, 6.70; N, 7.21%. Calcd for $C_{21}H_{26}N_2O_5$: C, 65.28; H, 6.73; N, 7.25%.

*Corresponding author: Yang Liguang, College of Chemistry and Environmental Engineering, Anyang Institute of Technology, Anyang 455000, Henan, P.R. China, e-mail: lgyang@ayit.edu.cn

Su Wenhui, Tian Dayong, Yu Zhichao and Niu Yongsheng: College of Chemistry and Environmental Engineering, Anyang Institute of Technology, Anyang 455000, Henan, P.R. China

Table 1: Data collection and handling.

Crystal:	White block
Size:	0.10 × 0.08 × 0.06 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 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} :	16810, 3526, 0.038
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2346
$N(\text{param})_{\text{refined}}$:	257
Programs:	Bruker [1], SHELX [2, 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	0.31435(7)	0.2870(3)	0.60410(7)	0.0610(4)
N1	0.52833(8)	0.8150(3)	0.58091(10)	0.0586(5)
H1	0.5059	0.7241	0.5497	0.070*
C1	0.23404(12)	−0.0447(4)	0.58256(11)	0.0741(7)
H1A	0.2101	0.0358	0.5427	0.111*
H1B	0.2037	−0.1539	0.6051	0.111*
H1C	0.2710	−0.1408	0.5667	0.111*
O2	0.42466(7)	0.5094(3)	0.55743(7)	0.0631(4)
N2	0.76797(8)	0.6324(3)	0.63258(9)	0.0621(5)
C2	0.26117(10)	0.1503(4)	0.63441(10)	0.0621(6)
H2A	0.2251	0.2641	0.6451	0.075*
H2B	0.2787	0.0706	0.6781	0.075*
O3	0.63832(8)	0.4672(2)	0.57454(7)	0.0660(4)
H3	0.6300	0.4733	0.5315	0.099*
C3	0.34677(10)	0.4640(4)	0.64696(10)	0.0535(5)
O4	0.82058(7)	0.4227(3)	0.74802(7)	0.0702(4)
H4	0.7975	0.5225	0.7236	0.105*
C4	0.32721(11)	0.5374(4)	0.71165(10)	0.0618(5)
H4A	0.2898	0.4613	0.7293	0.074*
O5	0.89746(7)	0.0757(3)	0.81068(7)	0.0701(4)
C5	0.36232(12)	0.7251(4)	0.75209(12)	0.0687(6)
H5	0.3481	0.7717	0.7958	0.082*
C6	0.41678(12)	0.8368(4)	0.72701(12)	0.0674(6)
H6	0.4395	0.9623	0.7535	0.081*
C7	0.43987(10)	0.7650(3)	0.66044(11)	0.0568(5)
C8	0.40500(10)	0.5759(3)	0.61793(10)	0.0534(5)
C9	0.49964(11)	0.8726(4)	0.63798(12)	0.0602(5)
H9	0.5204	0.9963	0.6667	0.072*
C10	0.59624(10)	0.8948(3)	0.56642(11)	0.0577(5)
H10A	0.6048	1.0626	0.5848	0.069*
H10B	0.6001	0.8983	0.5157	0.069*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C11	0.64832(9)	0.7127(3)	0.60141(10)	0.0514(5)
H11	0.6419	0.7082	0.6522	0.062*
C12	0.71992(10)	0.8002(4)	0.59315(12)	0.0658(6)
H12A	0.7285	0.8002	0.5434	0.079*
H12B	0.7257	0.9701	0.6110	0.079*
C13	0.80356(10)	0.4834(4)	0.59795(11)	0.0598(5)
H13	0.8002	0.4939	0.5488	0.072*
C14	0.84919(9)	0.2976(4)	0.63202(10)	0.0533(5)
C15	0.88667(10)	0.1361(4)	0.59163(11)	0.0624(6)
H15	0.8839	0.1526	0.5426	0.075*
C16	0.92722(11)	−0.0451(4)	0.62348(11)	0.0677(6)
H16	0.9517	−0.1511	0.5960	0.081*
C17	0.93230(10)	−0.0724(4)	0.69700(11)	0.0634(6)
H17	0.9600	−0.1968	0.7181	0.076*
C18	0.89646(9)	0.0840(4)	0.73848(10)	0.0553(5)
C19	0.85480(9)	0.2728(4)	0.70592(10)	0.0539(5)
C20	0.93531(11)	−0.1217(4)	0.84632(11)	0.0700(6)
H20A	0.9828	−0.1095	0.8374	0.084*
H20B	0.9183	−0.2836	0.8296	0.084*
C21	0.92702(13)	−0.0933(5)	0.92422(11)	0.0879(8)
H21A	0.9456	0.0648	0.9404	0.132*
H21B	0.9505	−0.2270	0.9497	0.132*
H21C	0.8797	−0.0996	0.9321	0.132*

Experimental details

The C-bound H atoms were geometrically placed (C–H = 0.95–0.98 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5 U_{\text{eq}}(\text{C})$. The O- and N-bound H atoms were located in a difference Fourier map but were refined with a distance restraint of N–H = 0.88 ± 0.01 Å, and with $U_{\text{iso}}(\text{H})$ set to 1.2 $U_{\text{eq}}(\text{N})$ [3].

Comment

There are several complexes containing the deprotonated title molecule as an anionic ligand, which have been reported, however, the structure about this ligand has not been reported [4–6].

The compound crystallizes in the monoclinic space group $P2_1/c$ with four formula units in the unit cell. As shown in the figure, each molecule formally consists of two 3-ethoxysalicylidenyl and one 2-hydroxyl-1,3-

propanediamine-diyl moieties. It is observed that the phenolic proton is transferred to the imine nitrogen in the structure of this molecule to yield a ylidic structure. The NH group and phenolate O atom form a strong intramolecular N–H···O hydrogen bond. The N–H···O hydrogen-bonded rings are almost planar and coupled with the cyclohexadiene rings [7, 8].

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