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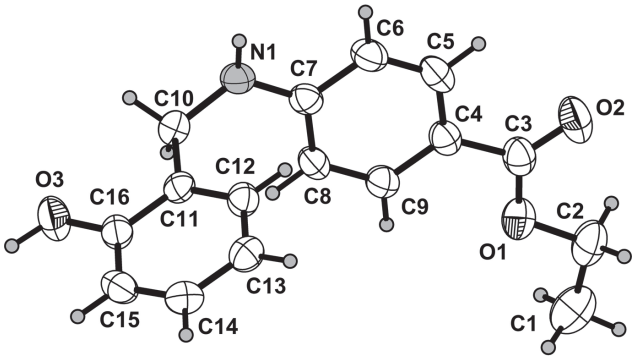
The crystal structure of ethyl 4-((2-hydroxybenzyl) amino)benzoate, a Schiff base, $C_{16}H_{17}NO_3$



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Abstract

$C_{16}H_{17}NO_3$, triclinic, $P\bar{1}$ (no. 2), $a = 7.5445(7)$ Å, $b = 8.3326(9)$ Å, $c = 12.4291(10)$ Å, $\alpha = 94.468(8)°$, $\beta = 91.063(7)°$, $\gamma = 116.644(10)°$, $V = 695.01(13)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0461$, $wR_{ref}(F^2) = 0.1240$, T = 291 K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

In a typical reaction, (*E*)-ethyl 4-((2-hydroxybenzylidene) amino)benzoate [4] (10 mmol, 2.43 g) was dissolved in 50 mL of ethanol and reduced by addition of excess sodium borohydride (10 mmol, 0.38 g) 1 L: 1.4 $NaBH_4$. The solution was stirred until the yellow color disappeared. Then the solution was diluted with 8–10 times of water and the pH was

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.92 × 0.39 × 0.20 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.9 cm^{-1}
Diffractometer, scan mode:	Xcalibur, ω scans
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4651, 2434, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1732
$N(param)_{refined}$:	190
Programs:	SHELX [1], CrysAlis^{PRO} [2], OLEX2 [3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O3	0.2431(2)	0.64218(19)	1.14652(11)	0.0598(4)
O1	0.2129(2)	0.33784(19)	0.43007(10)	0.0582(4)
N1	0.2399(2)	0.8356(2)	0.84692(12)	0.0444(4)
O2	0.3272(2)	0.5998(2)	0.35672(11)	0.0696(5)
C12	0.5806(3)	0.8033(2)	0.93366(14)	0.0422(5)
H12	0.5819	0.8437	0.8659	0.051*
C7	0.2496(2)	0.7549(2)	0.74763(13)	0.0395(5)
C16	0.4130(3)	0.6923(2)	1.09167(14)	0.0419(5)
C11	0.4093(2)	0.7500(2)	0.98980(13)	0.0375(5)
C10	0.2226(3)	0.7537(2)	0.94677(14)	0.0429(5)
H10A	0.1178	0.6307	0.9359	0.051*
H10B	0.1822	0.8184	1.0013	0.051*
C4	0.2609(3)	0.5933(2)	0.54248(14)	0.0427(5)
C8	0.1917(3)	0.5702(2)	0.73036(14)	0.0445(5)
H8	0.1479	0.4990	0.7876	0.053*
C13	0.7485(3)	0.7986(3)	0.97458(15)	0.0468(5)
H13	0.8610	0.8344	0.9348	0.056*
C9	0.1984(3)	0.4919(3)	0.62980(14)	0.0445(5)
H9	0.1603	0.3688	0.6203	0.053*
C15	0.5816(3)	0.6874(2)	1.13391(15)	0.0488(5)
H15	0.5822	0.6485	1.2020	0.059*
C6	0.3127(3)	0.8568(3)	0.65947(15)	0.0485(5)
H6	0.3517	0.9801	0.6686	0.058*
C3	0.2714(3)	0.5149(3)	0.43446(15)	0.0489(5)
C5	0.3177(3)	0.7772(3)	0.55969(15)	0.0512(5)
H5	0.3601	0.8477	0.5021	0.061*
C14	0.7482(3)	0.7401(2)	1.07515(16)	0.0499(5)
H14	0.8610	0.7362	1.1035	0.060*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C2	0.2216(3)	0.2473(3)	0.32734(16)	0.0631(7)
H2A	0.3581	0.2917	0.3074	0.076*
H2B	0.1462	0.2687	0.2710	0.076*
C1	0.1348(4)	0.0514(3)	0.3412(2)	0.0821(8)
H1A	0.1451	−0.0128	0.2760	0.123*
H1B	−0.0025	0.0076	0.3566	0.123*
H1C	0.2057	0.0329	0.4000	0.123*
H1	0.298(3)	0.956(3)	0.8532(15)	0.060(6)*
H3	0.265(4)	0.623(3)	1.220(2)	0.102(9)*

adjusted to 6 by addition of 12% HCl. The white precipitate was collected and dried in air.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The methyl, methylene and aromatic CH groups were idealized and refined using rigid groups (AFIX 137, 43 and 23 options of SHELXL program [1]), with $U_{iso}(H) = 1.5U_{eq}(C)$ for the methyl groups and $U_{iso}(H) = 1.2U_{eq}(C)$ for the others. The NH and OH hydrogen atoms were refined freely.

Comment

Schiff bases have found widespread use in many disciplines. These compounds are mainly used in medicinal chemistry, in which they are reported as antibacterial, antimalarial and antifungal [5]. Many Schiff bases have been used as ligands to obtain controllable coordinative geometries with transition metals and lanthanides. The characterization of several Schiff base ligands resulting from the condensation of salicylaldehyde and β-diketone has already been reported [6].

There is one title molecule in the asymmetric unit. The bond lengths for C—N (1.378(2) Å and 1.443(2) Å) are different showing the conjugation to the adjacent aryl and alkyl moiety, respectively. The average bond lengths in the rings C11—C16 and C4—C9 are 1.382 Å (σ = 0.007) and 1.386 Å (σ = 0.010), respectively. All geometric parameters are in the range of other Schiff bases [5–7]. The C–O bond length of the hydroxyl group O3—C16 (1.371(2) Å) and that of the ester moiety O1—C3 (1.333(2) Å), O1—C2 (1.449(2) Å) and O2 = C3 (1.211(2) Å), are in the expected ranges. The nitrogen atom is sp^3 with a C7—N1—C10 angle of 123.77(17)°. Whithin the crystal structure, the molecules are connected by strong hydrogen bonds with O3···O2 = 2.763(5) Å and π-π interactions between two rings C11—C16 of two molecules (3.723(1) Å).

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