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Crystal structure of 1,2-bis[2-(pent-3-en-4-yl-2-one)-aminophenoxy]ethane, $C_{24}H_{28}N_2O_4$

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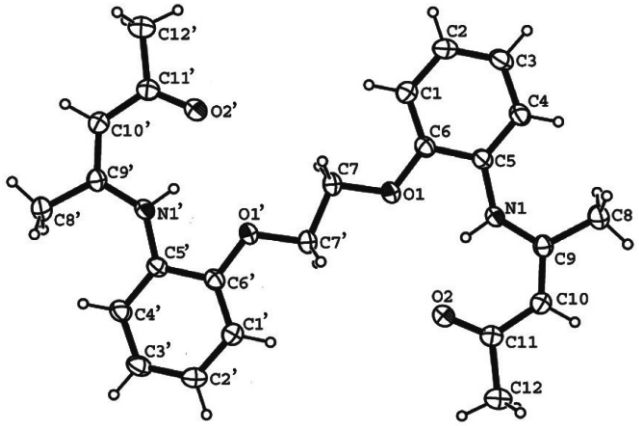

Abstract

$C_{12}H_{14}NO_2$, monoclinic, $P2_1/n$ (no. 14), a = 14.239(5) Å, b = 5.148(2) Å, c = 15.068(6) Å, β = 99.17(1)°, V = 1090.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.037, $wR_{ref}(F^2)$ = 0.111, T = 273 K.

Source of material

Solution of acetylacetone (0.02 mol) in ethanol (20 ml) was added to a stirred ethanol solution (20 ml) of 1,2-bis(2-aminophenoxy)-ethane (0.01 mol) in a three-necked flask. The reaction mixture was refluxed for 5 h, then the solution was cooled to room temperature. The yellow precipitated residue was removed from the solution by filtration and then washed with ethanol 3 times. Single crystals suitable for X-ray diffraction were obtained from an ethanol/chloroform mixture (1:1, v:v) by slow evaporation at room temperature.

Discussion

Schiff base-metal complexes have been widely investigated for their properties and applications in different fields of catalysis and materials chemistry [1]. The Schiff base ligands derived from benzoylacetone have found immense applications, mostly due to the benzoylacetoneiminato moiety being a very reactive nucleophilic center for easily incorporating a variety of metal ions for different purposes [2].

In the crystal structure of the title compound, the two benzene rings are linked by an diether chain, forming a non-coplanar structure, but with the two rings parallel to each other. The bond lengths and bond angles are in the normal ranges. The intramolecular N1–H1A···O2 hydrogen bond links the amine N1 atoms to the enolic O2 atoms, the intramolecular N1–H1A···O1 hydrogen bond links the amine N1 atoms to the ether chain O1 atoms. The molecules are also connected by C–H···π interactions, with ∠C2–H2···Cg1^{i} = 129.75°, d(H2···Cg1) = 3.151(2) Å, ∠C8–H8A···Cg1^{ii} = 141.72°, d(H8A···Cg1) = 2.706(2) Å, where Cg1 are the centroids of the phenyl rings formed by atoms C1,C2,C3,C4,C5,C6 (symmetry codes: i: ½–x,–½+y,–½–z; ii: x,1+y,z).

Table 1. Data collection and handling.

Crystal:	size 0.21 × 0.29 × 0.31 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.85 cm^{-1}
Diffractometer, scan mode:	Bruker CCD, φ/ω
$2\theta_{max}$:	50.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8982, 1921
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1531
$N(param)_{refined}$:	140
Program:	SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.8167	0.6776	0.3493	0.065
H(2)	4*e*	0.6680	0.6561	0.2604	0.069
H(3)	4*e*	0.5608	0.3456	0.2884	0.071
H(4)	4*e*	0.5984	0.0612	0.4067	0.069
H(7A)	4*e*	0.9414	0.7193	0.4733	0.068
H(7B)	4*e*	0.9679	0.5079	0.4056	0.068
H(8A)	4*e*	0.6172	–0.3068	0.4788	0.073
H(8B)	4*e*	0.5805	–0.0880	0.5370	0.087
H(8C)	4*e*	0.6046	–0.3640	0.5784	0.098
H(10)	4*e*	0.7452	–0.3991	0.6704	0.064
H(12A)	4*e*	0.9811	–0.3081	0.7772	0.125
H(12B)	4*e*	0.9177	–0.5470	0.7417	0.125
H(12C)	4*e*	0.8818	–0.3481	0.8076	0.125
H(1A)	4*e*	0.8197	0.0764	0.5515	0.061

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	4*e*	0.7729(1)	0.5534(3)	0.3612(1)	0.0525(9)	0.058(1)	0.0532(9)	–0.0018(8)	0.0104(7)	0.0054(7)
C(2)	4*e*	0.6839(1)	0.5400(3)	0.3077(1)	0.064(1)	0.058(1)	0.0477(8)	0.0078(8)	–0.0007(7)	0.0034(7)
C(3)	4*e*	0.6199(1)	0.3562(3)	0.3248(1)	0.057(1)	0.056(1)	0.0577(9)	0.0013(8)	–0.0132(7)	–0.0041(8)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(4)	4*e*	0.6426(1)	0.1857(3)	0.3960(1)	0.0516(9)	0.0495(9)	0.065(1)	–0.0076(7)	–0.0076(7)	0.0012(7)
C(5)	4*e*	0.7300(1)	0.1970(3)	0.45169(9)	0.0421(8)	0.0416(8)	0.0478(8)	0.0017(6)	0.0007(6)	–0.0033(6)
C(6)	4*e*	0.7961(1)	0.3828(3)	0.43151(9)	0.0394(8)	0.0527(9)	0.0458(8)	0.0016(6)	0.0064(6)	–0.0027(6)
C(7)	4*e*	0.9569(1)	0.5375(3)	0.4667(1)	0.0396(8)	0.068(1)	0.063(1)	–0.0072(7)	0.0117(7)	0.0096(8)
C(8)	4*e*	0.6222(1)	–0.2348(4)	0.5381(1)	0.049(1)	0.077(1)	0.066(1)	–0.0128(9)	0.0070(8)	0.0056(9)
C(9)	4*e*	0.7225(1)	–0.1503(3)	0.5696(1)	0.0421(8)	0.0453(8)	0.0521(8)	–0.0014(6)	0.0107(6)	–0.0043(6)
C(10)	4*e*	0.7749(1)	–0.2683(3)	0.6426(1)	0.0511(9)	0.0529(9)	0.0568(9)	–0.0025(7)	0.0100(7)	0.0071(7)
C(11)	4*e*	0.8707(1)	–0.2089(3)	0.6799(1)	0.0522(9)	0.056(1)	0.0602(9)	0.0036(8)	0.0038(7)	0.0081(8)
C(12)	4*e*	0.9170(1)	–0.3673(4)	0.7587(1)	0.072(1)	0.090(1)	0.082(1)	0.005(1)	–0.007(1)	0.030(1)
N(1)	4*e*	0.76226(8)	0.0416(2)	0.52719(8)	0.0404(7)	0.0503(7)	0.0589(8)	–0.0068(6)	–0.0046(5)	0.0055(6)
O(1)	4*e*	0.88229(7)	0.3759(2)	0.48712(7)	0.0365(6)	0.0759(8)	0.0623(7)	–0.0090(5)	0.0026(5)	0.0164(5)
O(2)	4*e*	0.91758(8)	–0.0348(3)	0.65026(9)	0.0564(7)	0.0806(9)	0.0850(9)	–0.0136(6)	–0.0125(6)	0.0260(7)

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