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ORIGINAL PAPER

Synthesis and Crystal Structure of 2-(2,3,4-Trimethoxy-6-Methylbenzylideneamino)phenol

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Abstract The title compound 2-(2,3,4-trimethoxy-6-methylbenzylideneamino)phenol ($C_{17}H_{19}NO_4$, $M_r = 301.33$) was synthesised and characterized by elemental analysis, IR spectra and single crystal X-ray diffraction. The crystal belongs to monoclinic, space group $P21/c$, with $a = 10.4458(14)$, $b = 8.3870(10)$, $c = 17.780(2)$ Å, $\beta = 91.102(2)^{\circ}$, $V = 1557.4(3)$ Å^3, $Z = 4$, $D_c = 1.285$ g/cm^3, $\lambda = 0.71073$ Å, μ(Mo $K\alpha$) = 0.092 mm^{-1}, $F(000) = 640$. The final refinement gave $R = 0.0452$, $wR(F^2) = 0.1065$ for 2,743 observed reflections with $I > 2\sigma(I)$. X-ray diffraction analysis reveals that the molecule adopts an E configuration at the C=N functional bond. The dihedral angle between the two phenyl rings is 38.3(3)°. The crystal structure is stabilized by C–H···O, O–H···O and O–H···N hydrogen bonds and π–π stacking interactions.

Keywords 2-(2,3,4-Trimethoxy-6-methylbenzylideneamino)phenol · Synthesis · Crystal structure

Introduction

Schiff bases are used as substrates in the preparation of a number of industrial and biologically active compounds via ring closure, cycloaddition and replacement reactions [1]. Schiff bases are known to have biological activities such as antimicrobial [2–4], antitumor [5], and herbicidal properties [6]. They have also been widely used as versatile ligands involved in various metal chelation reactions to form metal complexes [7–9], which are very intreresting in many fields, such as catalysis and enzymatic reactions [10, 11] and magnetism [12]. Recently, a few Schiff base compounds with antibacterial activity have been investigated [4, 13–15]. However, to the best of our knowledge, the Schiff base 2-(2,3,4-trimethoxy-6-methylbenzylideneamino)phenol and the complexes derived from it have never been reported so far. Investigation on the structure of the Schiff base compound may be helpful to design and synthesise new metal complexes. In this paper, the title compound was synthesised and its molecular structure was investigated by elemental analysis, FT-IR. and X-ray crystallographic techniques.

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Experimental Procedures

Reagents and Techniques

Infrared absorption spectra were obtained from a Nicolet NEXUS 670 FT-IR spectrometer in KBr discs and were reported in cm^{-1} units. Carbon, nitrogen and hydrogen analyzes were performed on an Elemental Analysensteme GmbH Vario EL analyzer. 2,3,4-Trimethoxy-6-methylbenzaldehyde, 2-aminophenol and ethylalcohol were purchased from Weifang Runze (China).

The Synthesis of 2-(2,3,4-Trimethoxy-6-Methylbenzylideneamino)phenol

A mixture of 2-aminophenol (0.695 g, 5 mmol) and 2,3,4-trimethoxy-6-methylbenzaldehyde (1.04 g, 5 mmol) in ethanol (30 mL) was refluxed for 2 h. After cooling the precipitate was filtered and dried. The product was recrystallized in ethanol. Yield: 91%. A 20 mg of the

product was dissolved in ethanol of 20 mL. The solution was filtered to remove impurities, and then left for crystallization at room temperature. Anal. Calcd. (%) for $C_{17}H_{19}NO_4$: C 67.77, H 6.31, N 4.65. Found (%): C 67.86, H 6.54, N 4.56. Selected IR data (cm^{-1}): 1,636 cm^{-1} (s, –C=N), 1,200 cm^{-1} (s, Ar–O).

X-ray Structure Determination

The single crystal X-ray data of title compound was collected on a Bruker SMART diffractometer with a graphite monochromatized Mo–Kα radiation ($\lambda = 0.71073$ Å). The structure was solved by SHELXS-97 and refined with SHELXL-97 [16]. The positions of the H atoms bonded to C atoms were calculated (C–H distance 0.96 Å), and refined using a riding model. All the H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with O–H and C–H distances of 0.82 and 0.96 Å, respectively, and Uiso(H) = 1.2–1.5Ueq(C) and Uiso(H) = 1.5Ueq(O). The details of the X-ray data collection, experimental conditions, and structure solution and structure refinements are given in Table 1. Some selected bond distances and angles are listed in Table 2. The molecular structures with the atom-numbering scheme are shown in Fig. 2. Crystallographic data (excluding structure factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. 698899.

Table 1 Crystal and experimental data for the title compound

Compound	$C_{17}H_{19}NO_4$
Color/shape	Light yellow/block
Formula weight	301.33
CCDC deposit no.	698899
Crystal system	Monoclinic
Space group	$P2_1/c$
Unit cell dimensions	$a = 10.4458(14)$ Å
	$b = 8.387(1)$ Å
	$c = 17.780(2)$ Å
	$\beta = 91.102(2)°$
Volume	1557.4(3) $Å^3$
Z	4
Density (calculated)	1.285 g cm^{-3}
Absorption coefficient	0.092 mm^{-1}
$F(000)$	640
Crystal size	0.50 × 0.46 × 0.35 mm^3
θ range for data collection	1.95–25.01°
Index ranges	$-10 \le h \le 12$; $-9 \le k \le 9$; $-20 \le l \le 21$
Reflections collected	7,524
Independent reflections	2,743
Reflections observed ($I > 2\sigma(I)$)	1,531
Refinement method	Full-matrix least-squares on F^2
Data/parameters	2,743/204
Goodness-of-fit on F^2	1.040
R indices [$I > 2\sigma(I)$]	$R_1 = 0.0452$; w$R_2 = 0.1065$
R indices (all data)	$R_1 = 0.0998$; w$R_2 = 0.1444$
Largest diff. peak and hole	0.157 and −0.186 e. $Å^{-3}$

Table 2 Selected bond lengths (Å) and bond angles (°)

N(1)–C(1)	1.261(3)	N(1)–C(12)	1.411(3)
O(1)–C(3)	1.370(3)	O(1)–C(8)	1.385(4)
O(2)–C(4)	1.385(3)	O(2)–C(9)	1.437(3)
O(3)–C(5)	1.361(3)	O(3)–C(10)	1.417(3)
O(4)–C(13)	1.360(3)		
C(1)–N(1)–C(12)	120.6(2)	C(3)–O(1)–C(8)	118.0(2)
C(4)–O(2)–C(9)	116.1(2)	C(5)–O(3)–C(10)	118.4(2)
N(1)–C(1)–C(2)	126.7(2)	O(1)–C(3)–C(4)	121.1(2)
O(1)–C(3)–C(2)	117.0(2)	C(3)–C(4)–O(2)	120.1(2)
O(2)–C(4)–C(5)	120.2(2)	O(3)–C(5)–C(6)	124.7(2)
O(3)–C(5)–C(4)	115.5(2)	C(17)–C(12)–N(1)	125.6(2)
C(13)–C(12)–N(1)	116.0(3)	O(4)–C(13)–C(14)	118.8(3)
O(4)–C(13)–C(12)	120.7(3)		

Results and Discussion

The Schiff reaction of 2,3,4-trimethoxy-6-methylbenzaldehyde with 2-aminophenol in a 1:1 ratio which was confirmed by the elemental analysis and IR spectra.

The title compound consists of benzylidenimin and *o*-hydroxyl phenyl groups, and they do not share a common plane (Fig. 1). The benzene ring (C2–C7) is nearly planar to within 0.003(2) Å, the displacements of C8, C9 and C10 are −0.918(3), −0.984(2) and 0.169(3) Å, respectively, from this plane. The another one (C12–C17) is nearly planar to within 0.005(3) Å. The dihedral angle between the two benzene ring planes is 38.3(3)°. The bond lengths and bond angles are as expected. C1–N1 is a double bond

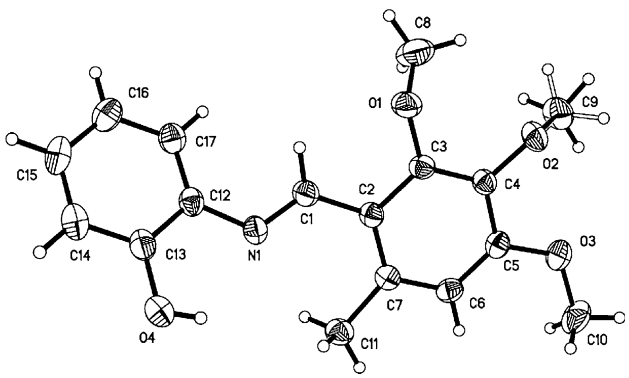

Fig. 1 The molecular structure of the title compound

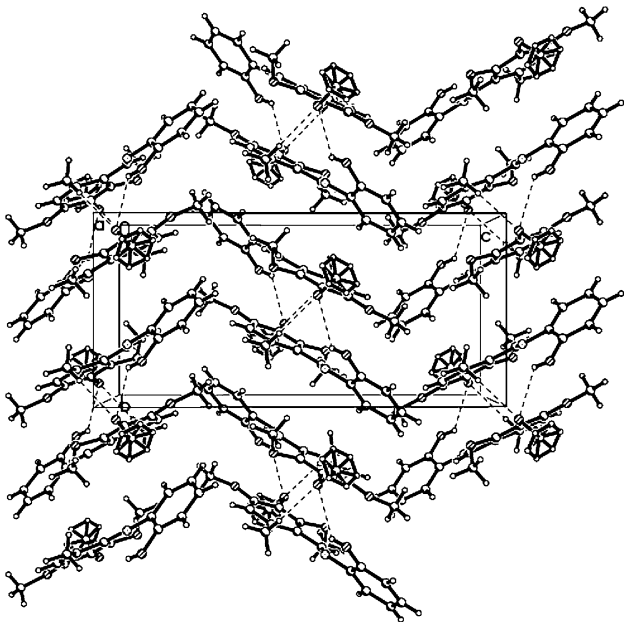

Fig. 2 Crystal packing of the title compound

Table 3 Hydrogen bond schemes (Å, °)

D–H···A	D–H	H···A	D–A	D–H···A
O4–H4···O2^{i}	0.82	2.482	3.068	129
C9–H9C···O3	0.96	2.438	3.069	136

Symmetry codes: (i) $1-x$, $2-y$, $-z$

[1.261(3) Å] [15]. While the N1–C12 bond [1.411(3) Å] is found to have normal single-bond length. Bond lengths C3–O1 [1.370(3) Å], C4–O2 [1.385(3) Å] and C5–O3 [1.361(3) Å] are in good agreement with the values reported in the literature [17].

The crystal structure is stabilized by intermolecular hydrogen bonds C–H···O and O–H···O, and the title molecule forms hydrogen bonded dimers (Fig. 2; Table 3). The existence of two benzene rings is very helpful to form a strong π–π stacking interactions [18].

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