

КРАТКИЕ СООБЩЕНИЯ

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CRYSTAL STRUCTURE OF *N,N'*-BIS-(2,5-DIMETHOXYBENZYLIDENE)-1,3-DIAMINOPROPANE© 2011 A. D. Khalaji¹*, K. Fejfarova², M. Dusek²¹Department of Chemistry, Faculty of Science, Golestan University, Gorgan, Iran²Institute of Physics of the ASCR, Na Slovance 2, 182 21 Prague 8, Czech Republic

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The Schiff-base compound *N,N'*-Bis-(2,5-dimethoxybenzylidene)-1,3-diaminopropan, (2,5-MeO-ba)₂pn, is synthesized and characterized by elemental analysis, FT—IR and ¹H NMR spectroscopy. The crystal structure of (2,5-MeO-ba)₂pn is determined by X-ray single crystal diffraction analysis. The X-ray diffraction analysis at 120 K reveals that (2,5-MeO-ba)₂pn crystallizes in the orthorhombic system, *Fdd2* space group with $a = 29.339(9)$ Å, $b = 29.4073(11)$ Å, $c = 4.4524(2)$ Å, $V = 3840.8(3)$ Å³, $\mu = 0.72$ mm⁻¹, and $Z = 8$.

Key words: X-ray diffraction, orthorhombic symmetry, Schiff base.

Schiff bases derived from substituted benzaldehyde [1—3] are widely used as chelating ligands forming transition metal complexes [4—6]. They have attracted considerable attention in the last years because the complexes are very interesting in many fields, such as catalysis [7] and magnetism [8]. Recently, several Schiff-base compounds with antibacterial activity have been investigated [9, 10]. In this paper, the title compound (2,5-MeO-ba)₂pn was synthesized and its molecular structure was characterized by elemental analysis, FT—IR and ¹H NMR spectroscopy, and X-ray structure analysis (Fig. 1).

Experimental. All reagents and solvents for the synthesis and spectroscopic studies were commercially available and used as received without further purification. ¹H NMR spectra were measured with a BRUKER DRX-500 AVANCE spectrometer at 500 MHz and all chemical shifts are reported in δ units downfield from TMS. The infrared spectrum was recorded on a JASCO 680 plus FT—IR spectrophotometer as a KBr pellet.

2,5-Dimethoxybenzaldehyde (0.332 g, 0.2 mmol) and 1,3-propanediamine (0.074 g, 0.1 mmol) were dissolved in ethanol (50 ml). The mixture was stirred at room temperature for 1 h to give a clear solution. Suitable crystals of the title compound (2,5-MeO-ba)₂pn for the X-ray study were formed by slow evaporation of the solvent over 8 days at room temperature (yield 76 %). The crystals are colorless. IR (KBr pellet, cm⁻¹): 2829—3008 (m, C—H aromatic and aliphatic), 1638 (s, C=N), 1495—1610 (m, C=C aromatic). ¹H NMR (CDCl₃, δ (ppm)): 1.78 (2H, q), 3.66 (4H, s), 3.77 (6H, s), 3.81 (6H, s), 6.83 (2H, d), 6.92 (2H, dd), 7.49 (2H, d), 8.67 (2H, s).

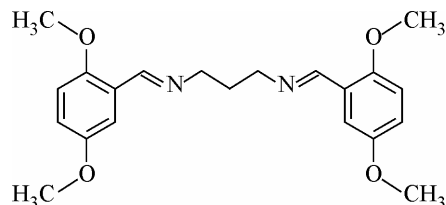


Fig. 1. Chemical diagram of the title compound (2,5-MeO-ba)₂pn

Crystallographic measurements were made at 120 K with a four-circle CCD diffractometer Gemini (Oxford diffraction, Ltd.) with mirror collimated CuK α radiation. The crystal structures were solved by direct methods with the SIR2002 program [11] and refined with the Jana2006 program package [12] by full-matrix least-squares technique on F^2 . The molecular structure plots were prepared using ORTEP III [13]. All hydrogen atoms were discernible in difference Fourier maps and could

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Table 1

Crystallographic and structure refinement of (2,5-MeO-ba)₂pn

Empirical formula	C ₂₁ H ₂₆ N ₂ O ₄
Formula weight	370.4
Crystal system	Orthorhombic
Space group	<i>Fdd2</i>
<i>T</i> , K	120
<i>a</i> , <i>b</i> , <i>c</i> , Å	29.3339(9), 29.4073(11), 4.4524(2)
<i>V</i> , Å ³	3840.8(3)
<i>Z</i>	8
μ , mm ⁻¹	0.72
Crystal size, mm ³	0.30 × 0.08 × 0.03
<i>T</i> _{min} / <i>T</i> _{max}	0.753 / 0.978
Measured / Independent reflections	8654 / 856
Reflection with <i>I</i> > 3 σ (<i>I</i>)	810
Parameters	122
<i>R</i> _{int}	0.028
<i>S</i>	1.69
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)]	0.027
<i>wR</i> (<i>F</i> ²)	0.076
$\Delta\rho_{\max}$ / $\Delta\rho_{\min}$, eÅ ⁻³	0.11 / -0.10

Table 2

Selected geometric parameters (Å, deg.)

O1—C1	1.373(2)	N1—C7	1.268(2)	C3—C4	1.385(3)
O1—C10	1.419(2)	N1—C8	1.4587(19)	C4—C5	1.408(2)
O2—C4	1.372(2)	C1—C2	1.390(2)	C5—C6	1.385(2)
O2—C11	1.427(2)	C5—C7	1.474(3)	C1—C6	1.384(3)
		C8—C9	1.5195(13)	C2—C3	1.388(3)
C1—O1—C10	117.01(13)	C1—C2—C3	120.26(18)	C4—C5—C7	120.55(16)
C4—O2—C11	117.30(15)	O2—C4—C3	124.49(14)	C6—C5—C7	120.47(15)
C7—N1—C8	116.94(15)	C8—C9—C8 ⁱ	114.15(10)	C1—C6—C5	121.37(15)
O1—C1—C2	124.80(16)	N1—C8—C9	109.37(10)	N1—C7—C5	122.30(17)
O1—C1—C6	115.91(14)	C3—C4—C5	119.78(16)	O2—C4—C5	115.73(16)
C2—C1—C6	119.29(17)	C4—C5—C6	118.96(17)		

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

be refined to reasonable geometry. According to common practice they were nevertheless kept in ideal positions during the refinement. The isotropic atomic displacement parameters of hydrogen atoms were evaluated as 1.2**U*_{eq} of the parent atom. Crystallographic data and details of the data collection and structure refinements are listed in Table 1. Bond distances and angles are listed in Table 2.

Crystal structure description. The X-ray structural determination of the title compound (2,5-MeO-ba)₂pn confirmed the assignment of its structure from spectroscopic data. The asymmetric unit

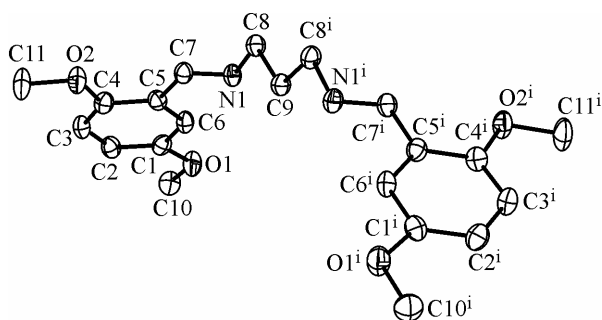


Fig. 2. Molecular structure of the title compound (2,5-MeO-ba)₂pn with the atom numbering scheme. Displacement ellipsoids are drawn at the 50 % probability level

of the title compound, along with the atom numbering scheme, is depicted in Fig. 2. Bond lengths and angles have normal values similar to those in previously published Schiff-base compounds [1–3]. The C7=N1 bond length of 1.268(2) Å conforms to the value for a double bond, while the C8—N1 bond length of 1.458(19) Å conforms to the value for a single bond. The C7—N1—C8 bond angle is 116.94(15)°, which is consistent with the *sp*² hybrid character of the N1 atom. Two 2,5-dimethoxybenzylidene groups are bridged by the —CH₂—CH₂— fragment *via* two C=N double bonds, with the distance between aromatic rings being *ca* 8.299 Å. Possible C—H...O and C—H...π interactions were found to be too weak to be considered seriously.

Supplementary Material. Crystallographic data (excluding structure factors) for the structure reported in this paper has been deposited with the Cambridge Crystallographic Data Center, CCDC Nos. 736105. Copies of the data can be obtained free of charge on application to The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, fax: +44 1223 336 033, e-mail: deposit@ccdc.cam.ac.uk or <http://www.ccdc.cam.ac.uk>.

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