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Synthesis and Structural Characterization of (2E,3E)-N^1,N^2-*Bis*(4-chlorobenzylidene)ethane-1,2-diamine

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Present compound (2E,3E)-N^1,N^2-*bis*(4-chlorobenzylidene)ethane-1,2-diamine ($C_{16}H_{14}C_{l4}N_2$, M_r = 305.19) was synthesized and characterized by elemental analysis, FT-IR, ^{1}H NMR and single crystal X-ray diffraction. The crystal belongs to monoclinic, space group Cc, with a = 10.166(2), b = 10.345(2), c = 26.650(5) Å, β = 91.91(3)º, V = 2801.1(10) Å^3, Z = 8, D_c = 1.447 g/cm^3, λ = 0.71073 Å, μ(MoK$_\alpha$) = 0.454 mm^{-1}, $F_{(000)}$ = 1264. The final refinement gave R = 0.0652, wR(F^2) = 0.1582 for 4,578 observed reflections with I > 2σ(I). X-Ray diffraction analysis reveals that the asymmetric unit of the title compound consists of two independent molecules. Each independent molecule adopts an E configuration about the central C=N functional bond. The dihedral angles between the two benzene rings are 2.3(2) and 0.5(2)º, respectively in two independent molecules. The molecules are linked through C-H···Cl hydrogen bonds interactions.

Key Words: (2E,3E)-N^1,N^2-*Bis*(4-chlorobenzylidene)ethane-1,2-diamine, Synthesis, Structure characterization.

INTRODUCTION

The 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]. In this paper, (2E,3E)-N^1,N^2-*bis*(4-chlorobenzylidene)ethane-1,2-diamine was synthesized and its molecular structure was characterized by elemental analysis, FT-IR, ^{1}H NMR and X-ray crystallographic techniques.

EXPERIMENTAL

All the reagents were of AR grade and used without further purification. IR spectra (4000-400 cm^{-1}), as KBr pellets, were recorded on a Nicolet FT-IR 510P spectrometer. ^{1}H NMR spectra were measured with a Bruker ALP 80 nuclear magnetic resonance spectrometer (CD_3COCD_3 as solvent, TMS as internal standard).

Synthesis of the title compound: To a solution of 1,2-diamino ethane (0.3 g, 5 mmol) and potassium acetate (0.98 g, 10 mmol) in 10 mL distilled water, 4-chlorobenzaldehyde (0.7 g, 5 mmol) in 20 mL ethanol was added drop by drop, the solution was stirred magnetically for 1 h at reflux temperature. After cooling to room temperature, the product was filtered and dried. Yield 89.2 %. 10 mg of the title compound were dissolved in 20 mL ethanol and the solution was kept at room temperature. The single crystal suitable for X-ray determination was obtained by evaporation from ethanol solution after a week. Anal. calcd. (%) for $C_{16}H_{14}N_2Cl_4$: C 62.95, H 4.59, N 9.18. Found (%): C 63.06, H 4.63, N 9.15. Selected IR (KBr, λ_{max}, cm^{-1}): 1635 (s, C=N), 1416 (s, C-N), 1090 (s, Ar-Cl). ^{1}H NMR data (CD_3COCD_3, ppm): δ = 8.14 (s, 2H), 7.60 (d, 4H), 7.35 (d, 4H), 3.90 (m, 4H).

Data collection and structure determination: A selected crystal of the title compound was mounted on a SMART CCD diffractometer. The reflection data were measured at 298 K, using a graphite monochromator MoK$_\alpha$ (λ = 0.71073 Å) radiation with an ω-2θ scan mode. The total reflections were 7,917 with 4,578 independent ones (R_{int} = 0.0679), of which 361 were observed with I > 2σ(I). Intensities were corrected for Lorentz and polarization effects and empirical absorption and all data were corrected using SADABB[16] program.

The structure was solved by direct methods using SHELXS-97[17] program. All the non-hydrogen atoms were refined on F^2 anisotropically by full-matrix least squares method. All hydrogen atoms were placed in the geometrically calculated positions. The contributions of these hydrogen atoms were included in the structurefactor calculations. The

atomic scattering factors and anomalous dispersion corrections were taken from International Table for X-ray crystallography[18]. The final least-square cycle gave R = 0.0652 and ωR = 0.1582 (w = 1/[σ^2(Fo^2) + (0.0882P)^2 + 0.0000P], where P = (Fo^2 + 2Fc^2)/3). S = 1.018, $(\Delta\rho)_{min}$ = -0.488 and $(\Delta\rho)_{max}$ = 0.472 e/Å^3. CIF file containing complete information on the studied structure was deposited with CCDC, deposition number 693623 and is freely available upon request from the following web site: www.ccdc.cam.ac.uk/data_request/cif

RESULTS AND DISCUSSION

The selected bond distances and bond angles are listed in Table-1. A displacement ellipsoid plot with atomic numbering scheme is shown in Fig. 1 and a perspective view of the crystal packing in the unit cell is shown in Fig. 2.

TABLE-1
SELECTED BOND LENGTHS (Å) AND BOND ANGLES (º)

Bond	Length (Å)	Bond	Angle (º)
Cl(1)-C(3)	1.736(6)	C(7)-N(1)-C(8)	117.6(5)
Cl(2)-C(14)	1.746(6)	C(10)-N(2)-C(9)	115.9(5)
Cl(3)-C(19)	1.740(6)	C(23)-N(3)-C(24)	116.7(5)
Cl(4)-C(30)	1.740(6)	C(26)-N(4)-C(25)	116.8(2)
N(1)-C(7)	1.268(8)	N(1)-C(7)-C(6)	122.9(6)
N(1)-C(8)	1.468(7)	N(1)-C(8)-C(9)	111.0(4)
N(2)-C(10)	1.259(8)	N(2)-C(9)-C(8)	110.3(4)
N(2)-C(9)	1.4440(7)	N(2)-C(10)-C(11)	123.7(5)
N(3)-C(23)	1.280(8)	N(3)-C(23)-C(22)	122.2(6)
N(3)-C(24)	1.459(8)	N(3)-C(24)-C(25)	110.8(4)
N(4)-C(26)	1.263(7)	N(4)-C(25)-C(24)	110.2(5)
N(4)-C(25)	1.459(8)	N(4)-C(26)-C(27)	123.5(6)

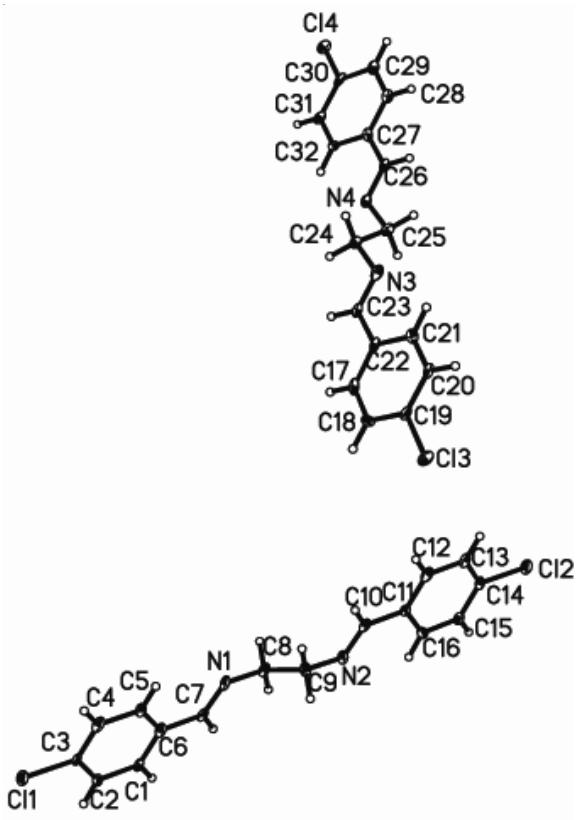

Fig. 1. Molecular structure with atomic numbering scheme

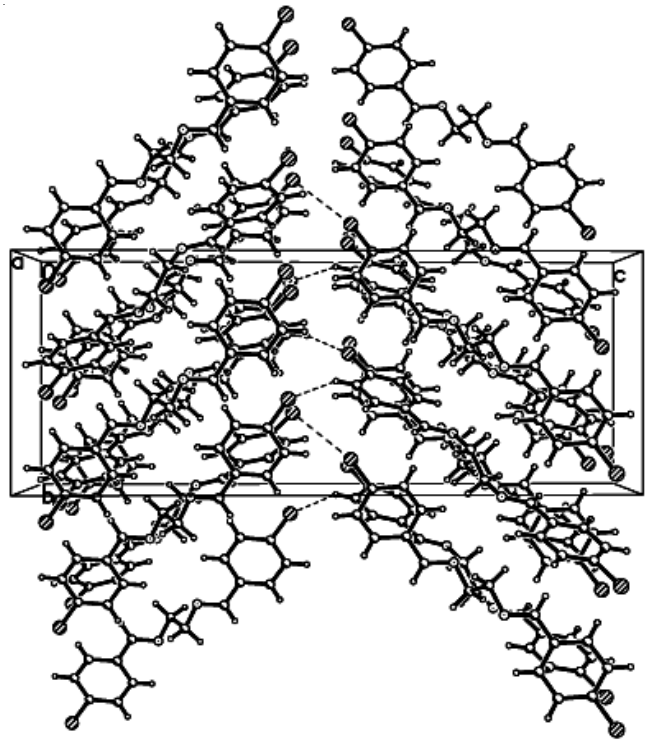

Fig. 2. View of crystal packing down the a-axis

As seen from Fig. 1, the asymmetric unit of (2E,3E)-N^1,N^2-*bis*(4-chlorobenzylidene) ethane-1,2-diamine consists of two independent molecules. In the independent molecule, two 4-chlorobenzaldehyde groups are bridged by ethane-1,2-diamine *via* two C=N double bonds in a roughly linear geometry. Each molecule adopts an E configuration about the central C=N functional bond. The dihedral angles between the two benzene rings are 2.3(2) and 0.5(2)º, respectively in two independent molecules.

All the bond lengths and bond angles in the title compound are within normal ranges and comparable to those in the similar compounds[13,14,19]. The C7-N1 bond length of 1.268(8) Å, the C10-N2 bond length of 1.259(8) Å, the C23-N3 bond length of 1.280(8) Å and the C26-N4 bond length of 1.263(7) Å conform to the value for a double bond, while the C8-N1 bond length of 1.468(7) Å, the C9-N2 bond length of 1.440(7) Å, the C24-N3 bond length of 1.459(8) Å and the C25-N4 bond length of 1.459(8) Å conform to the value for a single bond. The C=N bond lengths are close to the corresponding bond length in N,N'-*bis*(3- nitrobenzylidene)butane-1,4-diamine [1.267(3) Å][19]. The molecules are linked through C-H···Cl hydrogen bonds interactions (Fig. 2 and Table 2).

TABLE-2
HYDROGEN BOND SCHEMES (Å, º)

D-H···A	D-H	H···A	D-A	D-H···A
C2-H2···Cl4[a]	0.93	2.864	3.616	139
C29-H29···Cl1[b]	0.93	2.900	3.613	135

Symmetry codes: (a) -1+x, -y, -1/2 + z; (b) 3/2 + x, 1/2-y, 1/2 + z.

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