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Research Article

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Synthesis of 2-{(*Z*)-[(4-methylphenyl)imino]methyl}phenol Schiff base

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ABSTRACT

2-{(Z)-[(4-methylphenyl)imino]methyl}phenol Schiff base ($C_{14}H_{13}NO$),were synthesized and the structure was elucidated in the bases of ^{1}HNMR and X-ray. The X-ray establish the conformation of the molecule, which indicate the compound is crystalline in the monoclinic P 21/c with a = 19.3092(19) Å, b = 4.7611(4) Å, c = 12.2222(11) Å a = 90°, b = 102.087(10)°, g = 90°and Z = 4. Two benzene rings and azomethine group are practically coplanar, as a result of intramolecular hydrogen bond involving the O atom of hydroxyl group and N atom of azomethine group.

Key words: Salicylaldehyde, p-Toluidine, Schiff base and x-ray

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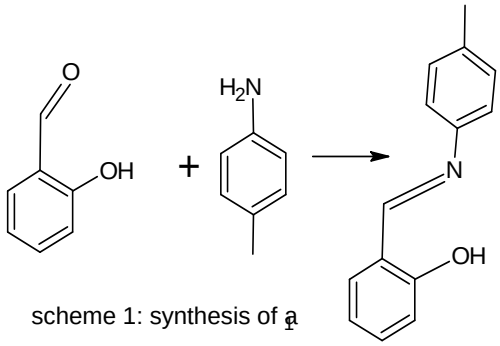
INTRODUCTION

Schiff bases(1,2) are well known for their biological applications as antibacterial, antifungal, anticancer, antiviral agents and as herbicides, furthermore; they have industrial applications (3), anti-tubercular activities(4), chelating abilities as chelating ligand in coordination chemistry(5) and widely applied in enantioselective compounds (6).

EXPERIMENTAL SECTION

The Schiff-bases was prepared as in (Scheme 1) by the usual condensation reaction**(7)**, in which salicylaldehyde(SA) (0.08mole) was drop wisely added to the amine (p-Toluidine) (0.08mole) in DMF with continuous stirring. After complete addition the reaction mixture was heated under reflux for about three hours. And as an application of green chemistry Salicylaldehyde and p-Toluidine mixed together without solvent give the same crystal product after about one week.

Data collection about device : CAD-4 EXPRESS (Enraf–Nonius, 1994); cell refinement: CAD-4 EXPRESS; data reduction: HELENA (Spek, 1996); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: XCIF in SHELXTL (Sheldrick, 2008).



scheme 1: synthesis of a

RESULTS AND DISCUSSION

After the reaction mixture was cooled to ambient temperature, the resulting clear yellow crystal was formed; then the crystal filtered off, washed with cold ether. Crystal collected in a good yield 80% (M.p. 100-101°C).

The structure of Schiff base (I) was elucidated on the bases of:
1) ^{1}H-NMR
The ^{1}H-NMR spectrum of Schiff-base (a1) (200 MHz), using DMSOd6 as solvent, showed the multiple signals of the aromatic protons of two phenyl rings, generally overlay the chemical shift range δ (p.p.m): 2.35 (Me, s, 3H), 6.80–7.40 (m, ArH, 8H), 8.60 (HC═N, s, 1H), 13.60 (OH, s, 1H)[8].

2) X-ray single crystal:
The title molecule, $C_{14}H_{13}NO$, exists in the solid state figure 1, the crystal have intrahydrogen bond between N_1 atom and H in O_1 [9].

Table 1: Crystal data and H- bond geometry

$C_{14}H_{13}N$ O M_r = 211.25 Monoclinic P 21/c Unit cell dimensions a = 19.3092(19) Å b = 4.7611(4) Å c = 12.2222(11) Å α= 90°. β = 102.087(10)° γ = 90°. V = 1098.72(17) Å^3 Z = 4 T = 291(2)K D = 1.277 Mg/m^3 M = 0.081 mm^{-1} Crystal size 0.4 x 0.3 x 0.05 mm^3 Theta range for data collection = 3.24 to 25.03°.	Data collection Refinement method: Full-matrix least-squares on F^2 Absorption correction: Semi-empirical from equivalents T_{min} = 0.8538, T_{max} = 1.00000 Refinement R[$F^2 > 2\sigma(F^2)$] = 0.0783 S= 1.038 4144 reflection 448 parameters Δρ max = 0.186 e.Å^{-3} Δρ min = -0.183 e.Å^{-3}

Hydrogen-bond geometry (A, °)

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(0B)...N(1)	0.92(3)	1.76(3)	2.598(2)	150(3)

Supplementary materials

Table 2: Atomic coordinates (x10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3)

	x	y	Z	U(eq)
N(1)	2544(1)	4526(3)	9175(1)	37(1)
C(15)	2133(1)	5947(4)	8422(2)	37(1)
C(7)	3529(1)	-371(4)	7664(2)	45(1)
C(4)	3500(1)	1331(4)	9785(2)	43(1)
C(5)	3021(1)	2500(4)	8906(1)	35(1)
C(14)	1639(1)	7995(4)	8677(1)	35(1)
C(2)	4019(1)	-1508(4)	8536(2)	40(1)
C(10)	1111(1)	10557(4)	9990(2)	58(1)
C(13)	1192(1)	9422(4)	7819(2)	45(1)
C(6)	3039(1)	1598(4)	7831(2)	42(1)
C(3)	3990(1)	-630(4)	9606(2)	46(1)
C(9)	1598(1)	8597(4)	9780(2)	44(1)
C(11)	674(1)	11907(5)	9127(2)	58(1)
C(12)	713(1)	11365(4)	8033(2)	53(1)
O(1)	2029(1)	7316(4)	10649(1)	65(1)
C(1)	4550(1)	-3644(4)	8340(2)	53(1)

Table 3: Bond lengths [Å] and angles [°]

N(1)-C(15)	1.276(2)
N(1)-C(5)	1.419(2)
C(15)-C(14)	1.444(2)
C(15)-H(15A)	0.93
C(7)-C(6)	1.376(2)
C(7)-C(2)	1.379(2)
C(7)-H(7A)	0.93
C(4)-C(3)	1.379(3)
C(4)-C(5)	1.380(2)
C(4)-H(4A)	0.93
C(5)-C(6)	1.390(2)
C(14)-C(13)	1.388(2)
C(14)-C(9)	1.396(2)
C(2)-C(3)	1.385(3)
C(2)-C(1)	1.499(3)
C(10)-C(11)	1.365(3)
C(10)-C(9)	1.387(3)
C(10)-H(10A)	0.93
C(13)-C(12)	1.372(3)
C(13)-H(13A)	0.93
C(6)-H(6A)	0.93
C(3)-H(3A)	0.93
C(9)-O(1)	1.350(2)
C(11)-C(12)	1.380(3)
C(11)-H(11A)	0.93
C(12)-H(12A)	0.93
O(1)-H(0B)	0.92(3)
C(1)-H(1A)	0.96
C(1)-H(1B)	0.96
C(1)-H(1C)	0.96
C(15)-N(1)-C(5)	121.89(15)
N(1)-C(15)-C(14)	122.75(16)
N(1)-C(15)-H(15A)	118.6
C(14)-C(15)-H(15A)	118.6
C(6)-C(7)-C(2)	122.29(18)
C(6)-C(7)-H(7A)	118.9
C(2)-C(7)-H(7A)	118.9
C(3)-C(4)-C(5)	121.33(17)
C(3)-C(4)-H(4A)	119.3
C(5)-C(4)-H(4A)	119.3
C(4)-C(5)-C(6)	117.75(17)
C(4)-C(5)-N(1)	117.10(16)
C(6)-C(5)-N(1)	125.15(16)
C(13)-C(14)-C(9)	118.51(17)
C(13)-C(14)-C(15)	120.08(16)
C(9)-C(14)-C(15)	121.41(16)
C(7)-C(2)-C(3)	117.05(18)
C(7)-C(2)-C(1)	121.61(17)
C(3)-C(2)-C(1)	121.33(18)
C(11)-C(10)-C(9)	120.4(2)
C(11)-C(10)-H(10A)	119.8
C(9)-C(10)-H(10A)	119.8
C(12)-C(13)-C(14)	121.51(19)
C(12)-C(13)-H(13A)	119.2
C(14)-C(13)-H(13A)	119.2
C(7)-C(6)-C(5)	120.32(17)
C(7)-C(6)-H(6A)	119.8
C(5)-C(6)-H(6A)	119.8
C(4)-C(3)-C(2)	121.23(18)
C(4)-C(3)-H(3A)	119.4
C(2)-C(3)-H(3A)	119.4
O(1)-C(9)-C(10)	119.25(18)
O(1)-C(9)-C(14)	121.08(18)
C(10)-C(9)-C(14)	119.66(18)
C(10)-C(11)-C(12)	120.7(2)
C(10)-C(11)-H(11A)	119.6
C(12)-C(11)-H(11A)	119.6
C(13)-C(12)-C(11)	119.2(2)
C(13)-C(12)-H(12A)	120.4
C(11)-C(12)-H(12A)	120.4
C(9)-O(1)-H(0B)	106.8(17)
C(2)-C(1)-H(1A)	109.5
C(2)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1B)	109.5
C(2)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4: Anisotropic displacement parameters ($Å^2 x\ 10^3$)

The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U11	U22	U33	U23	U13	U12
N(1)	39(1)	36(1)	38(1)	0(1)	10(1)	0(1)
C(15)	42(1)	37(1)	34(1)	-1(1)	12(1)	-3(1)
C(7)	51(1)	46(1)	43(1)	-5(1)	17(1)	-1(1)
C(4)	50(1)	43(1)	35(1)	0(1)	9(1)	3(1)
C(5)	36(1)	32(1)	38(1)	-2(1)	12(1)	-3(1)
C(14)	34(1)	33(1)	40(1)	-3(1)	11(1)	-2(1)
C(2)	40(1)	31(1)	52(1)	1(1)	16(1)	-4(1)
C(10)	65(1)	58(1)	57(1)	-7(1)	30(1)	8(1)
C(13)	44(1)	44(1)	47(1)	-2(1)	8(1)	-1(1)
C(6)	45(1)	44(1)	37(1)	-1(1)	7(1)	7(1)
C(3)	48(1)	43(1)	45(1)	7(1)	5(1)	8(1)
C(9)	47(1)	44(1)	43(1)	0(1)	15(1)	0(1)
C(11)	48(1)	48(1)	85(2)	-4(1)	27(1)	8(1)
C(12)	44(1)	47(1)	65(1)	4(1)	6(1)	4(1)
O(1)	81(1)	78(1)	39(1)	2(1)	17(1)	26(1)
C(1)	51(1)	39(1)	72(1)	-1(1)	22(1)	4(1)

Table 5: Hydrogen coordinates ($x\ 10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$)

	x	y	z	U(eq)
H(15A)	2153	5652	7677	45
H(7A)	3529	-956	6938	55
H(4A)	3492	1876	10513	51
H(10A)	1081	10956	10724	69
H(13A)	1219	9051	7082	54
H(6A)	2720	2328	7221	51
H(3A)	4308	-1375	10215	55
H(11A)	346	13205	9279	70
H(12A)	419	12306	7448	63
H(1A)	4493	-3975	7551	79
H(1B)	4478	-5367	8708	79
H(1C)	5020	-2957	8634	79
H(0B)	2293(15)	6030(60)	10350(20)	114(11)

Table 6: Torsion angles [°]

C(5)-N(1)-C(15)-C(14)	179.32(15)
C(3)-C(4)-C(5)-C(6)	1.2(3)
C(3)-C(4)-C(5)-N(1)	-179.21(16)
C(15)-N(1)-C(5)-C(4)	172.30(16)
C(15)-N(1)-C(5)-C(6)	-8.1(3)
N(1)-C(15)-C(14)-C(13)	-178.25(16)
N(1)-C(15)-C(14)-C(9)	2.0(3)
C(6)-C(7)-C(2)-C(3)	1.4(3)
C(6)-C(7)-C(2)-C(1)	-179.76(17)
C(9)-C(14)-C(13)-C(12)	-0.7(3)
C(15)-C(14)-C(13)-C(12)	179.54(17)
C(2)-C(7)-C(6)-C(5)	-0.5(3)
C(4)-C(5)-C(6)-C(7)	-0.8(3)
N(1)-C(5)-C(6)-C(7)	179.63(16)
C(5)-C(4)-C(3)-C(2)	-0.3(3)
C(7)-C(2)-C(3)-C(4)	-1.1(3)
C(1)-C(2)-C(3)-C(4)	-179.85(17)
C(11)-C(10)-C(9)-O(1)	179.24(18)
C(11)-C(10)-C(9)-C(14)	-0.3(3)
C(13)-C(14)-C(9)-O(1)	-178.63(17)
C(15)-C(14)-C(9)-O(1)	1.2(3)
C(13)-C(14)-C(9)-C(10)	0.9(3)
C(15)-C(14)-C(9)-C(10)	-179.35(18)
C(9)-C(10)-C(11)-C(12)	-0.6(3)
C(14)-C(13)-C(12)-C(11)	-0.1(3)
C(10)-C(11)-C(12)-C(13)	0.8(3)

Symmetry transformations used to generate equivalent atoms:

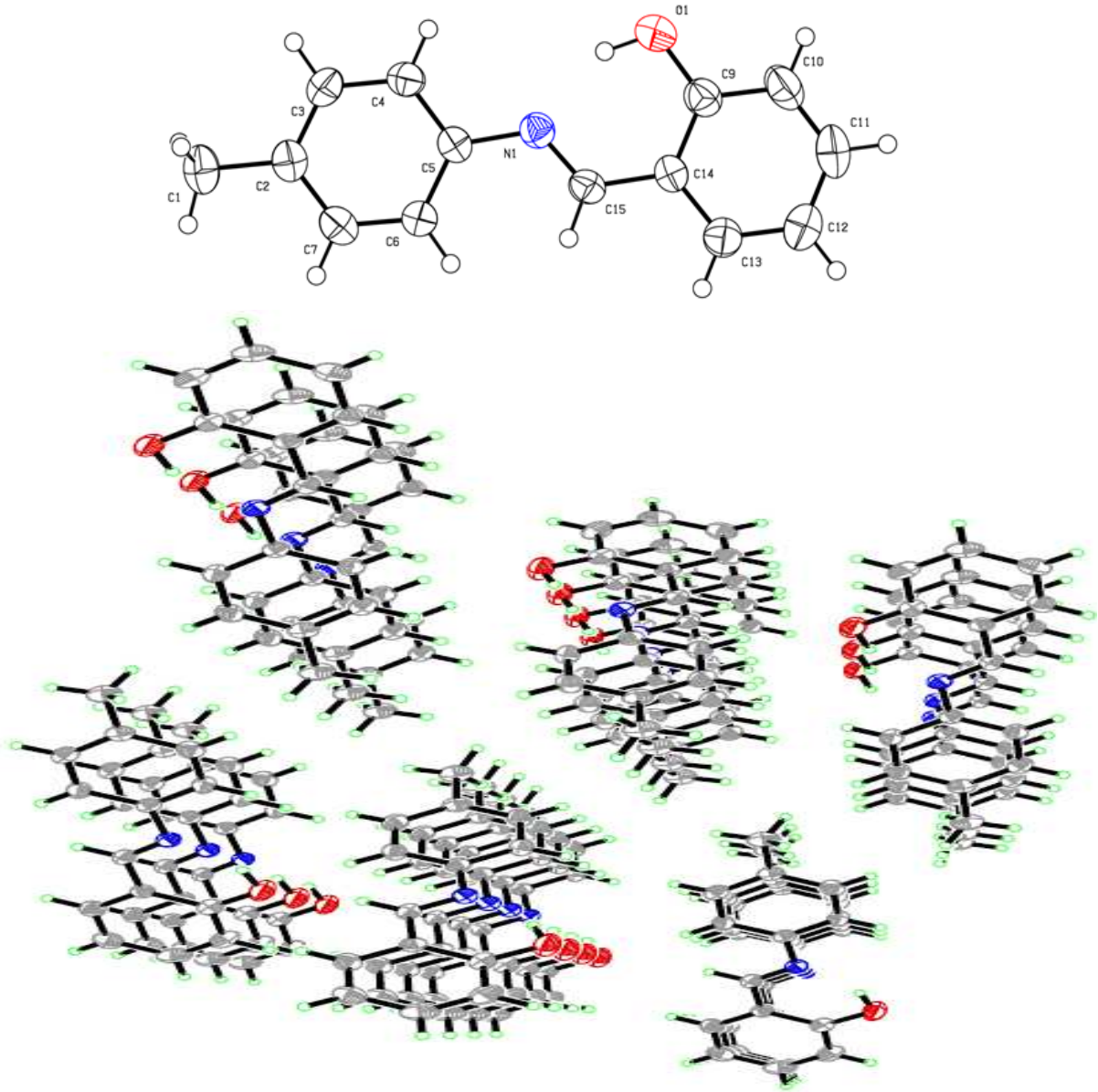


Figure 1: Molecular structure of the title compound with thermal ellipsoids at the 50% probability level

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