Synthesis and X-ray diffraction data of N^1,N^2-di(2-hydroxy) benzylidenbenzene-1,2-di-imine, $C_{20}H_{16}N_2O_2$

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The Schiff base N^1,N^2-di(2-hydroxy)benzylidenebenzene-1,2-di-imine was prepared from salicylaldehyde and 1,2-diaminobenzene by reflux in ethanol for 6 h obtaining an orange crystalline solid. The Schiff base solid was separated by filtration and washed with ethanol and ethylic ether, and finally, it was dried in a vacuum system for 2 h. The X-ray powder diffraction pattern for this new compound showed that the crystalline compound belongs to the monoclinic system and space group $P2_1/c$ (No. 14) with refined unit-cell parameters $a = 5.9672$ (7) Å, $b = 16.561$ (1) Å, $c = 16.337$ (2) Å, $\beta = 91.41°$ (1). The volume of the unit cell is $V = 1614.1$ (2) Å^3. © *2018 International Centre for Diffraction Data*. [doi:10.1017/S0885715618000027]

Key words: tetradentate Schiff base, X-ray powder diffraction, bioactive compounds

I. INTRODUCTION

Schiff bases are organic compounds where its common structural feature is an imine group, which is generally represented as R-CH=N-R′ where R and R′ correspond to groups such as alkyl, aryl, cyclo alkyl, or heterocyclic groups (Das and Linert, 2016). These organics compounds are used in multiple applications ranging from analytical detection (Ghosh *et al.*, 2016; Shabbir *et al.*, 2016), biological activities (Rosu *et al.*, 2011; Zoubi, 2013), and inorganic chemical processes (Pioquinto-Mendoza *et al.*, 2015).

Schiff bases structures were introduced initially by Hugo Schiff in 1861 (Schiff, 1869), and since then multiple variations in the peripheral structure of the azomethine group (−*C*=*N*−) have been proposed, observing its multifunctionality. The biological activity of Schiff bases has been explained by the ability of the nitrogen atom to form hydrogen bonds with active centres of cells, or malignant malformations, leading to its application in antifungal (Abo-Aly *et al.*, 2015), antibacterial (Mondal *et al.*, 2015), and anticancer activity (Amer *et al.*, 2013).

In the field of catalysis, Schiff bases have been used as organic ligands in the formation of coordination complexes with many transition metals in different oxidation states (Gupta and Sutar, 2008), such as V(IV) (Alsalim *et al.*, 2013), Ti(IV) (De Rosa *et al.*, 2006), Re(V) (Grzegorczyk *et al.*, 2014), Mo(VI) (Wang *et al.*, 2016), Mn(III) (Sarkar *et al.*, 2016), and they have been extensively investigated as an effective catalysts for oxidation processes. In this context, particular attention has been given to manganese complexes with Schiff base Salen ligands (*N*,*N*′-bis(salicylidene)ethylenediamine) (Valyaev *et al.*, 2016), which function as catalysts in asymmetric organic synthesis (Xia *et al.*, 2005) and asymmetric epoxidation of unfunctionalized olefins (Chen *et al.*, 2012).

Manganese–Salen complexes have demonstrated their synthetic utility in oxidation reaction through some examples, such as the asymmetric epoxidation of cis-cinnamates as a key step to obtain the side chain of Taxol (Jacobsen *et al.*, 1994), used as an anticancer drug, or in the synthesis of intermediates to obtain Diltiazem which is a powerful drug used in the treatment of hypertension, angina pectoris, and some treatments for cardiac dysrhythmia (Bell *et al.*, 1996). In this work, we report results on the molecular characterization (Fourier transform infrared and nuclear magnetic resonance [NMR]) and X-ray powder diffraction (XRPD) data for the compound N^1,N^2-di(2-hydroxy)benzilidenebencen-1,2-di-imine, as a special ligand for many interesting applications of Schiff base compounds.

II. EXPERIMENTAL

A. Synthesis

All materials were commercial and were used without further purification unless otherwise noted. All solvents were thoroughly degassed prior to use. 1,2-phenylenediamine (1.4 mmol, 0.16 g) was dissolved with warming in the minimum volume of anhydrous ethanol and was slowly stirred at 25 °C for 1 h. After that 2-hydroxybenzaldehyde (2.8 mmol, 0.342 g) was added into the solution and the mixture was stirred for 8 h at reflux temperature resulting in the precipitation of an orange solid which was filtered and washed with ethyl ether. The title compound ($C_{20}H_{16}N_2O_2$) was purified by crystallization in methanol. Its synthesis is shown in

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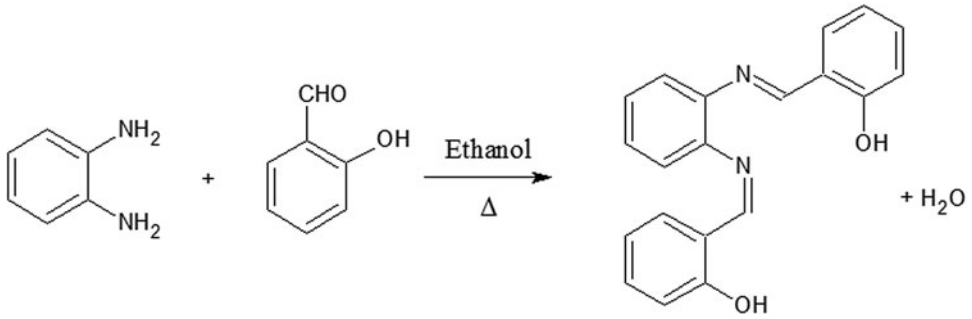

Figure 1. Synthesis of N^1,N^2-di(2-hydroxy)benzylidenebenzene-1,2-di-imine.

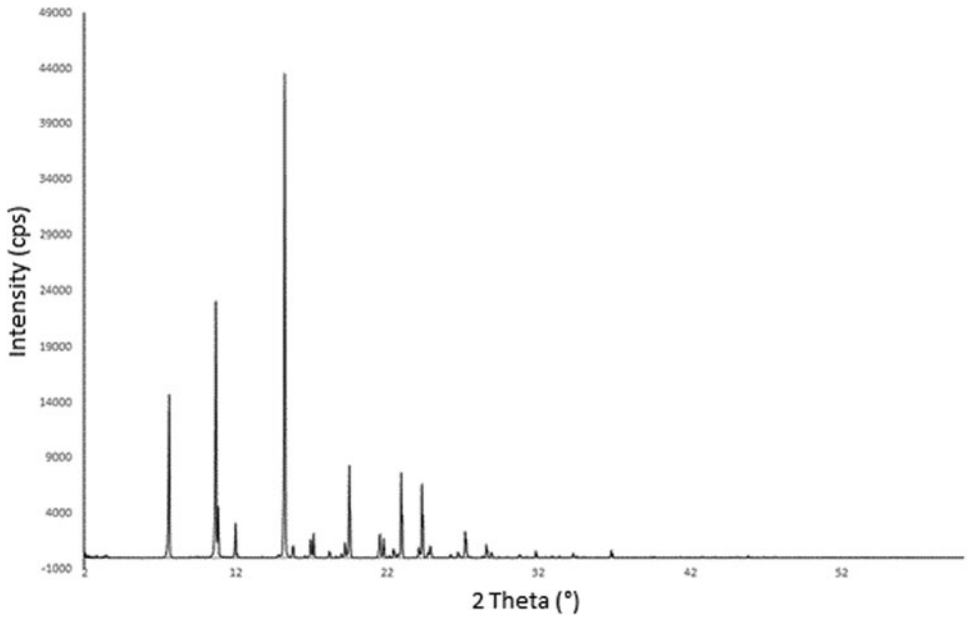

Figure 2. X-ray powder diffraction pattern of the compound N^1,N^2-di(2-hydroxy)benzylidenebenzene-1,2-di-imine. The background has been subtracted.

Figure 1. The yield was 81.6%; the melting point is 167 °C; the measured density is 1.255 g/cm^3, which was taken by the flotation method in an aqueous solution of potassium iodine. ATR-IR; *v*: 3053 (O-H); 1610 (C=N); 1396 (C-O). ^{1}H-NMR (400 MHz, $CDCl_3$, Me_4Si); showed δ (ppm) = 13.02 (s, 2H, OH), 8.64 (s, 2H, HC=N), 7.44–7.32 (m, 6H; aromatic), 7.26–7.21 (m, 2H; aromatic), 7.05 (d, J = 8.2 Hz, 2H; aromatic), 6.92 (td, J = 7.5, 1.1 Hz, 2H; aromatic).

B. Powder data collection

A small amount of the N^1,N^2-di(2-hydroxy) bencylidenbenzene-1,2-di-imine was gently ground in a mortar and sieved to a grain size less than 38 µm. The specimen was mounted on a polymethyl methacrylate (PMMA) specimen holder. The XRPD pattern was recorded with a D8 ADVANCE BRUKER diffractometer operating in Bragg–Brentano geometry equipped with a Cu-target X-ray tube (40 kV and 30 mA), a nickel filter and a 1-dimensional LynxEye detector. A divergence slit of 0.6 mm and primary and secondary soller slits of 2.5° were used. The scan range was 2° to 70° 2θ with a step size of 0.01526° and a count time of 2 s per step. The data were collected at room temperature (298 K).

Powder analytical software was used to subtracting the background (Sonnerveld and Visser, 1975), smoothing (Savitzky and Golay, 1964), and stripping off the $K\alpha_2$ component (Rachinger, 1948) and the second derivative method was used to determine the position and intensities of the diffraction maxima from each reflection.

III. RESULTS AND DISCUSSION

The X-ray powder pattern of the compound N^1,N^2-di (2-hydroxy)bencylidenbenzene-1,2-di-imine is shown in Figure 2. XRPD data for the compound are given in the Table I. All reflections were indexed using the DICVOL14 program (Boultif and Louër, 2004) on a monoclinic unit cell. The space group, $P2_1/c$ (No. 14), estimated by the program CHEKCELL (Laugier and Bochu, 2002) was compatible with the systematic absences and with the crystal density. The unit-cell parameters of the title compound were refined with the program NBS*AIDS83 software (Miguell *et al.*, 1981). Its crystal data, X-ray density and figures of

TABLE I. X-ray powder diffraction data of N^1,N^2-di(2-hydroxy)bencylidenbenzene-1,2-di-imine, $C_{20}H_{16}N_2O_2$.

$2\theta_{obs}$ (°)	d_{obs} (Å)	$(I/I_0)_{obs}$	H	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$ (°)
7.600	11.6230	32	0	1	1	7.596	11.6288	−0.004
10.684	8.2739	54	0	2	0	10.675	8.2807	−0.009
10.841	8.1544	11	0	0	2	10.825	8.1662	−0.016
11.980	7.3815	8	0	2	1	11.973	7.3856	−0.007
12.093	7.3128	1	0	1	2	12.074	7.3242	−0.019
14.833	5.9676	1	1	0	0	14.838	5.9654	0.005
15.229	5.8133	100	0	2	2	15.226	5.8144	−0.003
15.782	5.6108	3	1	1	0	15.777	5.6125	−0.005
16.568	5.3463	1	−1	1	1	16.569	5.3460	0.001
16.806	5.2712	2	1	1	1	16.808	5.2704	0.002
16.943	5.2288	4	0	3	1	16.940	5.2298	−0.003
17.138	5.1698	5	0	1	3	17.131	5.1719	−0.007
18.178	4.8763	2	−1	0	2	18.185	4.8746	0.007
18.630	4.7590	1	1	0	2	18.620	4.7615	−0.01
18.977	4.6727	1	−1	1	2	18.963	4.6762	−0.014
19.219	4.6144	4	1	2	1	19.214	4.6157	−0.005
19.403	4.5711	21	0	3	2	19.393	4.5735	−0.01
19.498	4.5490	4	0	2	3	19.498	4.5490	0
21.435	4.1421	4	0	4	0	21.444	4.1403	0.009
21.507	4.1284	5	1	2	2	21.510	4.1278	0.003
21.751	4.0827	4	0	0	4	21.749	4.0831	−0.002
22.135	4.0127	4	0	4	1	22.131	4.0134	−0.004
22.415	3.9632	2	0	1	4	22.408	3.9644	−0.007
22.470	3.9536	4	−1	1	3	22.469	3.9538	−0.001
22.679	3.9177	1	1	3	1	22.681	3.9173	0.002
22.923	3.8765	19	0	3	3	22.924	3.8763	0.001
24.074	3.6937	3	0	4	2	24.080	3.6928	0.006
24.293	3.6609	17	0	2	4	24.285	3.6621	−0.008
24.679	3.6045	2	1	3	2	24.671	3.6056	−0.008
24.839	3.5817	3	1	2	3	24.837	3.5819	−0.002
26.179	3.4013	1	1	4	0	26.178	3.4014	−0.001
26.676	3.3390	1	−1	4	1	26.674	3.3393	−0.002
26.826	3.3207	2	1	4	1	26.827	3.3206	0.001
27.155	3.2812	7	0	3	4	27.142	3.2827	−0.013
27.461	3.2453	2	0	5	1	27.454	3.2462	−0.007
28.284	3.1528	1	−1	2	4	28.290	3.1521	0.006
28.545	3.1245	4	1	4	2	28.546	3.1244	0.001
28.860	3.0911	2	1	2	4	28.864	3.0907	0.004
29.354	3.0402	1	0	2	5	29.370	3.0386	0.016
29.925	2.9835	1	2	0	0	29.933	2.9827	0.008
30.737	2.9065	1	0	4	4	30.729	2.9072	−0.008
30.796	2.9011	1	−2	1	1	30.792	2.9015	−0.004
31.184	2.8659	2	1	4	3	31.175	2.8666	−0.009
31.583	2.8306	1	0	5	3	31.593	2.8297	0.01
31.803	2.8115	2	0	3	5	31.806	2.8112	0.003
32.178	2.7796	1	2	0	2	32.176	2.7797	−0.002
32.887	2.7212	1	0	6	1	32.882	2.7216	−0.005
33.372	2.6828	1	1	2	5	33.371	2.6829	−0.001
34.264	2.6150	1	0	6	2	34.265	2.6149	0.001
34.527	2.5956	1	1	4	4	34.529	2.5955	0.002
35.046	2.5584	2	2	1	3	35.051	2.5581	0.005
36.780	2.4416	1	0	3	6	36.784	2.4414	0.004
36.861	2.4365	4	−2	0	4	36.848	2.4373	−0.013
37.547	2.3935	2	−1	2	6	37.554	2.3931	0.007
37.861	2.3744	2	−1	5	4	37.842	2.3755	−0.019
39.628	2.2725	2	0	7	2	39.629	2.2725	0.001
41.335	2.1825	2	−1	7	1	41.341	2.1822	0.006
42.807	2.1108	1	1	4	6	42.805	2.1109	−0.002
45.122	2.0077	2	1	3	7	45.129	2.0075	0.007
45.827	1.9785	1	1	6	5	45.840	1.9780	0.013
46.869	1.9369	2	−1	4	7	46.872	1.9368	0.003

merit M_{20} (de Wolff, 1968) and F_{20} (Smith and Snyder, 1979) are compiled in the Table II (see online supplementary material). In the powder diffraction pattern is observed an amorphous halo around 10° 2θ corresponding to the contribution from the sample holder (PMMA) which was subtracted by DIFFRAC.EVALUATION (EVA) software.

TABLE II. Crystallographic parameters for the compound N^1,N^2-di (2-hydroxy)bencylidenbenzene-1,2-di-imine, $C_{20}H_{16}N_2O_2$.

N^1,N^2-di(2-hydroxy)bencylidenbenzene-1,2-di-imine	
a (Å)	5.9672 (7)
b (Å)	16.561 (2)
c (Å)	16.337 (2)
β (°)	91.41 (1)
V (Å^3)	1614.1 (2)
Z	4
M_{20}	44.8
F_{30}	128.0 (0.0060, 39)
D_m (g/cm^3)	1.255
D_x (g/cm^3)	1.302
Formula weight (g/mol)	316.35

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715618000027.

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