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Synthesis, Physical Characterization, Antibacterial and Antifungal Activities of 2-((E)-1-(2-((E)-1-(2-Hydroxyphenyl)ethylideneamino) phenylamino) ethyl) phenol

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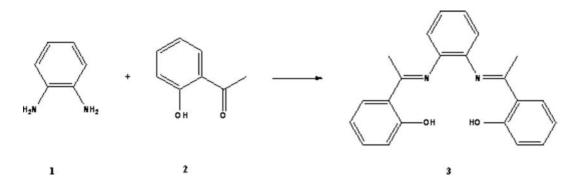
Keywords: 2-Hydroxyacetophenone, 1,2-phenylenediamine, Schiff base, AM1, B3LYP.

Abstract:

In this paper we report the synthesis of 2-((E)-1-(2-((E)-1-(2-hydroxyphenyethylideneamino)))phenylamino) ethyl) phenol.In addition to its synthesis we present AM1 and B3LYP/6-31G* calculations to characterize the physical properties of this molecule. Finally, the antifungal and antibacterial activities of this derivative have been evaluated

Introduction

Schiff bases are an important class of ligands, such ligands and their metal complexes have a variety of applications including biological, clinical, analytical and industrial in addition to their important roles in catalysis and organic synthesis [1]. Those that having multidentate coordination sites are known to form complexes with transition metal ions readily [2]. Such complexes play an important role in bioinorganic chemistry and redox enzyme systems [3] and may provide the basis of models for active sites of biological systems [4] or act as catalysts [5]. Schiff base compounds are of increasing interest because such dinuclear systems are known to act as a paramagnetic building block for multidimensional expanded structures as well as for their important roles in biological systems, e.g., in many metalloenzymes, redox and nonredox proteins and also as a catalyst in olefin epoxidation [6]. Ardakani and his coworkers have reported a selective nitrate PVC membrane electrode from 2-hydroxyacetophenone [7]. Synthesis and metal ion uptake studies of chelating resins by use of 2-hydroxyacetophenone have been reported by Samal [8].



Results and Discussion:

1, 2- pheneylenediamine **1** (1.08g, 10mmol) and 2-Hydroxyacetophenone **2** (2.71 g, 20 mmol) were dissolved in 25 ml of warm ethanol. The reaction mixture was refluxed for 7h at 85 °C, and allowed to stand. The solid crystals were filtered off and washed with ethanol. The pure Schiff base **3** was isolated as

a light brown crystalline solid (yield 89%). We next performed theoretical calculations to present a viable structure for the product. All calculations in this work where carried out with the AM1 level of theory using the GAUSSIAN 03 suite of programs. More information about these methods is available elsewhere. Figure 1 presents the optimized structure of the molecule with bond lengths and bond angles shown. We obtained a melting point (mp) value 108-110 °C, and IR (KBr, cm⁻¹): 3368(OH) (B3LYP/6-31G*: 3217); 1620(C=N) (B3LYP/6-31G*: 1629), as well as NMR.

¹H-NMR: 14.27 (1H, OH, s), 7.34 (4H, Ar, d), 7.15 (2H, Ar, d), 6.89 (2H, Ar, d), 6.74 (2H, Ar, d), 2.6 (6H, CH₃, s).

¹³C-NMR: 173.12 (C = N), 162.13 (COH), 115.75-138.21 (aromatic carbons), 17.19 (CH₃).

MS (m/z): 345 (M+1), 329, 227, 133, 65.

All calculations in this work where carried out with the AM1 level of theory using the GAUSSIAN 03 [9] suite of programs. In addition we have carried very intense B3LYP/6-31G* optimizations and frequency calculations. More information about these methods is available elsewhere [10]. Figure 1 presents the optimized structure of the molecule with bond lengths and bond angles shown as well as the theoretical IR vibrational spectrum.

Table 1 shows the thermodynamic properties for the complex in figure 1 where T (temperature in K), S (entropy in J mol-1 K-1), Cp (heat capacity at constant pressure in kJ mol-1 K-1), and Δ H=H° - H°298.15 (enthalpy content, in kJ mol-1), T1=100 K, T2=298.15 K, and T3=1000 K calculated AM1 and B3LYP/6-31G* frequencies. The fits were performed according to the equations implemented by the National Institute of Standards and Technology (NIST) [11]. These equations have been very good at predicting physical properties of various molecules, as we have tested in the past [12-14].

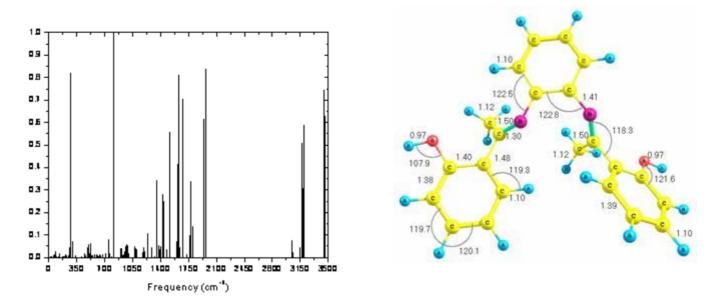


Figure 1. AM1 optimized structure and its theoretical IR vibrational spectrum for molecule 3.

		Fitted Thermodynamic Equation (T/1000=t)	100 K	298.15 K	1000 K
3	Cp	$-43.40526+1651.94082*t -933.47093*t^{2} + 174.57639*t^{3} + 0.49907*t^{-2}$	162.2	373.91	851.29

S	$42.38515*\ln(t) + 1150.64098*t + 29.96975*t^{2}/2$ -367.20075*t ³ /3 + 723.17337/(2*t ²) + 162.84889	432.48	703.25	1444.07
ΔΗ	$133.5428*t + 1417.88899*t^{2}/2 - 523.88367*t^{3}/3 - 52.55339*t^{4}/4 + 0.2264/t - 563.55851$	10.32	63.16	526.8

Table 1. Thermodynamic properties of the molecules in Figure 1-2 (A-B), calculated at the AM1 level and B3LYP/6-31G* level of theory, where C_p is the heat capacity in J mol⁻¹ K⁻¹, S is the entropy in J mol⁻¹ K⁻¹, and Δ H is the standard enthalpy kJ mol⁻¹. These where fitted to the Shomate equations which are implemented by the JANAF tables of the NIST databases. These equations converged to an R² value of 0.999 on average.

Antibacterial and antifungal activity tests

Derivative **3** was evaluated for its *in vitro* biological properties against human pathogens [15]. This compound was found to possess no antifungal activities against *S. cerevisiae* (ATCC 28383) and no antibacterial activities against Gram-positive and Gram-negative bacteria have been noticed (Table 2).

Sampla	Antimicrobial acti	Antimicrobial activity (MIC), µg/mL				
Sample CIP	S. cerevisiae	S. aureus	C. albicans	E. Coli		
CIF	(ATCC 28383)	(4.83)	(1180-79)	(54127)		
3	>50	>50	>50	>50		

Table 2. Antimicrobial activity of Schiff base 3

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