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Microwave-assisted solvent-free synthesis of N,N-bis(salicylidene)-2,2-dimethylpropane-1,3-diamine

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Abstract: A fast and efficient method has been developed for the synthesis of N,N bis(salicylidene)-2,2-dimethylpropane-1,3-diamine, H_2[salpnMe], under microwave irradiation. The tetradenatate Schiff-base H_2[salpnMe] has been prepared and characterized by IR, ^{13}CNMR, ^{1}HNMR.

Keywords: Schiff-base; H_2[salpnMe]; microwave; salicylaldehyde; Solvent free.

Introduction

Hugo Schiff described the condensation between an aldehyde and an amine leading to a Schiff base in 1864 [1]. Schiff base compounds as ligands have gained importance because of the physiological and pharmacological activities associated with them. They constitute an interesting class of chelating agents capable of coordination with metal ions given complex which serve as models for biological system [2-4].

Solvent-free microwave irradiation is well known as environmentally benign method, which offers several advantages including shorter reaction times, cleaner reaction profiles and simple experimental/product isolation procedures[5,6]. Microwave irradiation presents a powerful tool toward organic reactions. We now to report the synthesis of N,N-bis(salicylidene)-2,2-dimethylpropane-1,3-diamine through the reaction of salicylaldehyde and dimethylpropane-1,3-diamine in the presence of silica gel under solvent-free conditions using microwave irradiation.

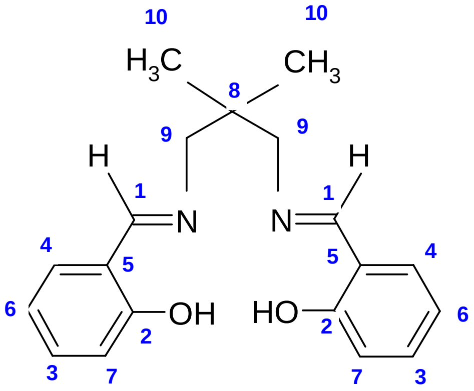
Results and Discussion

The structure of title compound has been assigned by spectroscopic data. In the IR spectrum, the characteristic Schiff base C=N stretching frequency is formed in the region between υ=1580-1610 cm^{-1} as a signal strong band. The OH stretching frequency is found at υ= 3450 cm^{-1} with a particular width.

Also, ν(CH) stretching frequency in 2963 cm^{-1} indicates the existence of methyl and propylene groups of the amine residue of this compound. The absorption bands at 1367 cm^{-1} are attributed to ν(CH) vibrations of CH_3 group.

In the ^{1}H NMR spectrum, the broad signals at δ = 13-14 *ppm* are assigned to the protons of the hydroxyl groups. Two protons of CH=N have the same chemical shifts at δ = 8-9 *ppm* and signals around δ = 6.8-7.5 *ppm* are attributed to the protons of aromatic rings (CH=CH). Two observed singlet peaks at δ = 3.29-3.47 *and* δ = 0.19 *ppm* are assigned to methyl and CH_2, repectively. There are ten signals in ^{13}CNMR spectrum. The peak at δ = 23.61 *ppm* could be assigned to the methyl group of propylene. The signals of C^2, C^3, C^4, C^5, C^6 and C^7 appear at δ = 160.62, 132.22, 131.56, 118.51, 118.41 and 116.36 *ppm*, respectively. The peak at δ = 35.66 *ppm* is related to CH_2 in propylene part.

Based on the presented ^{1}HNMR, ^{13}CNMR and IR spectroscopy for this compound, structure can be proposed as shown in scheme 1.



Scheme 1. The structure of *H_2[salpnMe]*

Experimental Section

A mixture of dimethylpropane-1,3-diamine (0.01 mol, 1.02 g) and salicylaldehyde (0.02 mol, 2.44 g) and 3g silica gel was mixed in mortar to provided a yellow soft powder, and then irradiated at 300W for 3 min. The progress of the reaction was monitored by TLC. After the completion of the reaction, a solid product was washed with MeOH and after the concentration of the solution, the yellow precipitate was obtained and washed with MeOH, dried and purified by recrystallizaton from MeOH.

IR (KBr, *cm^{-1}*): 3450(w), 3062(w), 2963(m), 1610(s), 1581(s), 1461(s), 1435(s), 1370(m), 1276(s), 1199(s), 1148(m), 872(m), 852(m), 763(s), 657(m).

^{1}HNMR (DMSO-d_6, *ppm*) δH: 13.58 (s, 2H, OH), 8.52 (s, 2H,-CH=N-), 6.86-7.43 (m, 8H, aromatic), 3.46 (s, 4H, CH_2) and 0.97 (s, 6H, CH_3).

^{13}CNMR (DMSO-d_6, *ppm*) δC: 166.45 (C^1), 160.62 (C^2), 132.2 (C^3), 131.56 (C^4), 118.51 (C^5), 118.41 (C^6), 116.36 (C^7), 67.05 (C^8), 35.66 (C^9), 23.61(C^{10}).

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