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Synthesis, & Characterization of "2,6-diamino-1-(anilinooxy)hexan-1-ol" &their imine derivatives

Aarohi Ahuja[1], Astitva Agarwal[2], Anil Kumar Koshal[3] and Abhishek Shandilya[4]

[1,2,4] *Department of Chemistry, Delhi Public School, New Delhi, 110022, India,*
[3]*Department of Chemistry, Government P.G. College, Lateri, Vidisha (MP) 464114, India,*

Abstract:
The aim of this study was to synthesize a novel 2,6-diamino-1-(anilinooxyhexan-1-ol having imine moiety and their identification by spectral techniques. 2,6-diamino-1-(anilinooxyhexan-1-olwas synthesized from the "2,6-diaminohexanoic acid (Lysine amino acid) with aniline" in a 1:1 molar ratio. Imine derivatives of 2,6-diamino-1-(anilinooxyhexan-1-olwere synthesized by using various derivatives of aromatic aldehydes. Furthermore, 2,6-diamino-1-(anilinooxyhexan-1-oland their imine derivatise were characterized by using IR and NMR spectral techniques.

Key words: *Lysine amino acid, Aromatic aldehyde, Novel imine moiety.*

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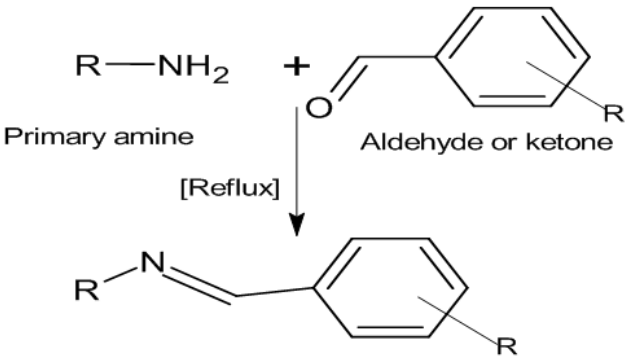
I. Introduction:

Imine and their aromatic derivatives have attained much emphasis in the area of pharmaceutical chemistry owing to their versatile applications in various fields including industrial processes, biological, and analytical systems.[01] These are formed by the condensation between aldehydes/ketones with primary amines in presence of some catalysis such as acetic acid and these are also called Schiff bases or azomethine.[02]

Many of them are reported as effective agents homogenous and heterogeneous catalysis many syntheticreactions. The importance of imines lies in the fact that they act as chelating ligands and complex with transition metal due to a loan pair is present on the nitrogen atom of imine moiety (C=N) as reaction Scheme: [01].[03]In the most of cases the complexes of imine are more pharmacological potential as compared to their parent ligand imines.[04]They showed many biologically applications as such antifungal, antimicrobial, antiviral and anti-tumour.[05]Many of these Schiff base can act as model compounds in various biological reactions.[06]



Reaction Scheme: [01]

II. Experimental:

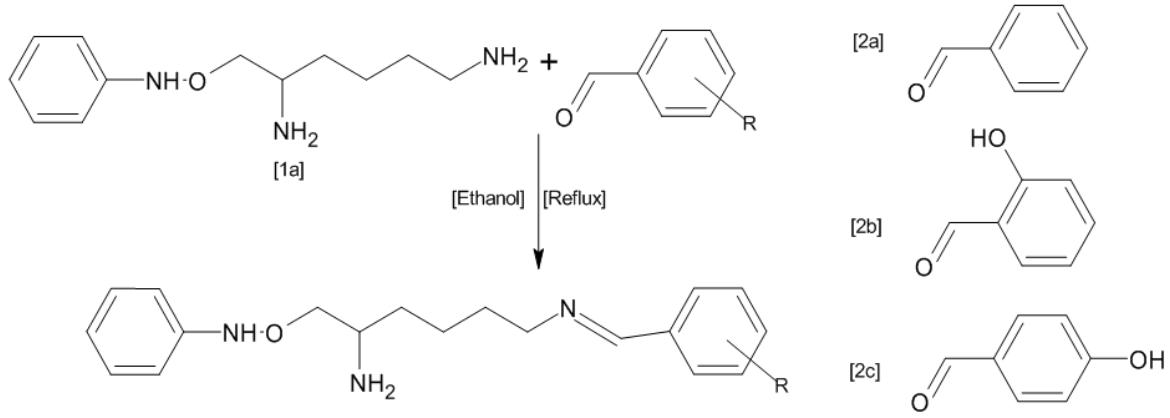
All required reagentsand solvent were purchased from Merck. The 2,6-diamino-1-(anilinooxy)hexan-1-ol is synthesized by employing known procedure. Further imines derivatives were synthesized by condensation of 2,6-diamino-1-(anilinooxy)hexan-1-ol and benzaldehyde.[07]

***N*-({2-amino-6-[(*E*)-benzylideneamino]hexyl}oxy)aniline**(2a) was prepared by the condensation of respective benzaldehyde aldehyde &2,6-diamino-1-(anilinooxy)hexan-1-ol followed by adding few drops of

glacial acetic acid. The 2,6-diamino-1-(anilinooxy)hexan-1-ol and benzaldehyde were taken in 1:1 molar ratio in ethanol in round bottom flask and refluxed the reaction mixture for two hour on water bath.

The untaintedness synthesized compound and the progress and reaction compilation were confirmed by thine layer chromatography (TLC). Synthesized compounds were purified by recrystallized inethanol. The melting point were checked and found incorrect. Another two derivatise were also synthesized and purified by above mention method.

Other derivatives of imine ***4-[(E)-{[5-amino-6-(anilinooxy)hexyl]imino}methyl]phenol (2b), and 2-[(E)-{[5-amino-6-(anilinooxy)hexyl]imino}methyl]phenol (2c),*** were prepared by using the method which was used for (2a).



Reaction Scheme: [02]

III. CharacterizationbyIR and ^{1}HNMR studies:

The melting point of the***N*-({2-amino-6-[(*E*)-benzylideneamino]hexyl}oxy)aniline** (2a),***4-[(E)-{[5-amino-6-(anilinooxy)hexyl]imino}methyl]phenol*** (2b***),*** and ***2-[(E)-{[5-amino-6-(anilinooxy)hexyl]imino}methyl]phenol*** (2c) were determined by open capillary method & found uncorrected.

The IR studies of the synthesized compound were recorded with perkin Elmer spectrophotometer in KBr phase from 400 cm^{-1} to 4000 cm^{-1}.Theimine derivatives were subjected to characterized by the IR spectra was recorded KBr phase.

^{1}HNMRspectra were carried from the Bruker spectrophotometer at 400 MHz, compounds were dissolved in deuterated solvents (DMSO-*d6* or CDCl3) solution with the TMS an internal standard.

[2a]:*N-({2-amino-6-[(E)-benzylideneamino]hexyl}oxy)aniline:*

Anewly synthesized compounds ***N*-({2-amino-6-[(*E*)-benzylideneamino]hexyl}oxy)aniline** (2a), was prepared by above described method. Yellow powder, yield 82 %, m.p131-135°C.

IR (cm^{-1}): 3160, 3172 (NH_2, NH), 2925 (aromatic C–H), 1630 (C=N), 1213 (C-O), 1588 (C=C). **^{1}H-NMR:** (300 MHz, CDCl3) δ (ppm): 8.4 (s, 1H, HC=N), 4.65 (s, 1H, NH), 4.96 (s, 2H, NH_2), 3.11–1.02 (m, 11H, CH_2-CH-$(CH_2)_3$-CH_2), 6.70–7.82 (m, 10 H, Ar-H).

[2b]: ***2-[(E)-{[5-amino-6-(anilinooxy)hexyl]imino}methyl]phenol:***

2-[(E)-{[5-amino-6-(anilinooxy)hexyl]imino}methyl]phenol (2b) were synthesized by above mentioned processor. Pale yellow powder, yield 65%, m.p 122–126 °C.

IR (cm^{-1}): 3156, 3168 (NH_2, NH), 2921 (aromatic C–H), 1625 (C=N), 1218(C-O), 1581 (C=C), 3344 (O-H).**^{1}H-NMR:** (300 MHz, CDCl3) δ (ppm): 9.6 (s, 1H, HC=N), 4.02 (s, 1H, NH), 3.98 (s, 2H, NH_2), 3.09–1(m, 11H, CH_2-CH-$(CH_2)_3$-CH_2), 6.78 –7.72 (m, 10H, Ar-H), 9.17 (s, 1H, OH).

[2c]:*4-[(E)-{[5-amino-6-(anilinooxy)hexyl]imino}methyl]phenol:*

According toabove mentionedsynthesis procedure the newly***4-[(E)-{[5-amino-6-(anilinooxy)hexyl]imino}methyl]phenol***(2c) were synthesized. Pale yellow pow, yield 71%, m.p 130–133 °C.

IR (cm^{-1}): 3148, 31 (NH_2, NH), 2924 (aromatic C–H), 1618 (C=N), 1214(C-O), 1578 (C=C), 3404 (O-H).**^{1}H-NMR:** (300 MHz, CDCl3) δ (ppm): 3.36–3.49 (s, 1H, HC=N), 3.65-3.80 (s, 1H, NH), 3.96 (s, 2H, NH_2), 3.11–1.02 (m, 11H, CH_2-CH-$(CH_2)_3$-CH_2), 6.70–7.82 (m, 10 H, Ar-H), 8.60 (s, 1H, OH).

IV. Conclusion:

The color, yield of novel synthesized compound were noted and the melting point of the compound were determined by open capillary. The comparison of IR spectra of the synthesized compoundssuggests the

presence ofimine moiety (C=N)and other groups of such as NH_2, CO-NH, OH and aromatic ring in the synthesized compounds.[08-10] On the comparison of the IR spectra of (2a), (2b), and (2c)indicates that the OH group is present in (2b), and (2c)in bothwhile it is absence in (2a).[11]The ^{1}HNMR data also suggesting the presence of OH in (2b), and (2c). The ^{1}HNMR data also indicate the presence of imine moiety (C=N), OH, aromatic hydrogen, NH_2 and NH groups in the synthesized compounds.[12]

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