

Studies On Some Benzoyl Hydrazone Derivatives

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ABSTRACT

The different hydrazones are obtained by condensation of 2-hydroxy benzohydrazide with various aldehydes viz. *p*-methoxybenzaldehyde, benzaldehyde, *p*-methylbenzaldehyde, *p*-bromobenzaldehyde and *α*-naphthaldehyde. The hydrazone derivatives have been characterized by elemental analysis and various spectral techniques such as UV, IR, ¹H-NMR, mass spectroscopy.

KEYWORDS : 2-hydroxy benzohydrazide; Aldehydes.

INTRODUCTION

There has been growing interest in the study of hydrazones because of their physiological activity, coordinative capability and applications^{1,2} in analytical chemistry. The ligands containing donor atoms like N, O are important to prepare metal complexes which shows biological activity. The presence of functional group, usually hydroxyl, azomethine, sufficiently near the site of coordination in the hydrazone ligands, gives a five or six membered chelate ring on reaction with metal ion, which shows structural variations³ depending upon the type of substituent present on aromatic ring.

EXPERIMENTAL

Materials and Methods

All the chemicals used were of the analytical grade (AR). The 2-hydroxy benzoyl hydrazide was prepared by the method reported in the Vogel⁴. The ester like methyl salicylate and hydrazine hydrate 80% were obtained from HIMEDIA while aldehydes such as *p*-methoxybenzaldehyde, benzaldehyde, *p*-methylbenzaldehyde, *p*-bromobenzaldehyde, *α*-naphthaldehyde were obtained from THOMAS BAKER. Solvents like ethanol whenever used were distilled and purified according to standard procedures⁴.

The UV spectra were recorded on Chemito UV-2600 Double Beam spectrophotometer. IR spectra of all the ligands were recorded in KBr on a Perkin-Elmer Precisely Spectrum 100 FT-IR Spectrometer in the region 4000-400 cm⁻¹. ¹H-NMR spectra were recorded in DMSO-d₆ on a Bruker 300MHz Spectrometer, Mass spectra were recorded on a GCMS.

Preparation of 2-hydroxy benzohydrazide from corresponding ester

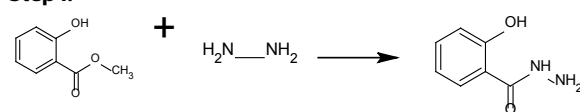
A mixture of methyl salicylate (1.3 ml, 10 mmol) and hydrazine hydrate 80% (1.2 ml, 20 mmol) were refluxed in ethanol for 3hrs on water bath. The resulting reaction mixture was allowed to cool to room temperature. The separated product was filtered washed with ethanol and purified by recrystallisation from ethanol (yield 85%).

Synthesis of hydrazones with various aldehydes

The 2-hydroxy benzohydrazide (0.01 M) in ethanol was taken in a two neck round bottom flask fitted with water condenser stirred for 10 min and heated gently on heating mantle till to get clear solution. A solution of substituted aromatic aldehyde (0.01 M) in ethanol was added slowly to this stirred solution. This reaction mixture was further reflux on water bath for 3hrs. When this solution was cooled to room temperature, precipitate formed was separated by filtration and purified by recrystallization from ethanol (yield 80%).

REACTION SCHEME OF HYDRAZONE DERIVATIVES

Step I:

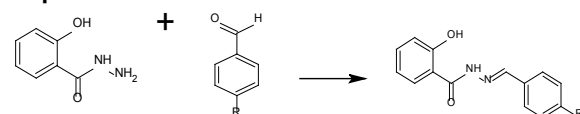


Methyl salicylate

Hydrazine

2-hydroxy benzohydrazide

Step II:

R = -OCH₃, -H, -CH₃, -Br, -Ph

RESULTS AND DISCUSSION

All the compounds are micro crystalline and thermally stable at room temperature. The elemental analysis data presented in Table-1 which is in good agreement with the molecular formula of the compounds and also supported by molecular ion peak observed in mass spectra.

Electronic spectra

UV spectra of compounds in ethanol was recorded. The bands observed in the range 280-290 nm can attributed to the n→π* transition of the electron of the unshared electron pair on heteroatoms present in the compounds. The bands observed in the range 184-220 nm assigned to π* transition of aromatic chromophore of the compounds.

Infrared Spectra

The FTIR spectra of the ligands were recorded in KBr over the range 4000-400 cm⁻¹. A broad band observed around 3350 cm⁻¹ due to O-H stretching mode. The band at 3050 cm⁻¹ assigned to Csp²-H mode. The peak around 2800 cm⁻¹ can be assigned to N=C-H mode. The intense absorption at 1625 cm⁻¹ is due to C=O amide moiety and peak at around 1600 cm⁻¹ is for C=N. The weak shoulder near 1450 cm⁻¹ assigned to Ar-H.

¹H-NMR Spectra

The ¹H-NMR spectra of all the compounds recorded in DMSO-d₆ solution and the data presented in Table-2.

Table 1: Physical and analytical data of hydrazone derivatives

Compound	Substituent	Molecular Formula	Molecular ion peak (M ⁺)	Yield %	M.P. (°C)	Color	% Elemental analysis Found (Calculated)		
							C	H	N
L1	-OCH ₃	C ₁₅ H ₁₄ O ₃ N ₂	270	78	221	White Shiny	66.64 (66.66)	5.25 (5.22)	10.35 (10.36)
L2	-H	C ₁₄ H ₁₂ O ₂ N ₂	240	76	242	White Shiny	70.01 (69.99)	5.04 (5.03)	11.63 (11.66)
L3	-CH ₃	C ₁₅ H ₁₄ O ₂ N ₂	254	80	239	White Shiny	70.88 (70.85)	5.50 (5.55)	11.04 (11.02)
L4	-Br	C ₁₄ H ₁₁ N ₂ O ₂ Br	319	79	266	White Shiny	52.67 (52.69)	3.51 (3.47)	8.76 (8.78)
L5	-Ph	C ₁₈ H ₁₄ O ₂ N ₂	290	78	219	Pale yellow	74.50 (74.47)	4.82 (4.86)	9.66 (9.65)

Table 2: ¹H-NMR spectral data of the compounds (δ in ppm)

Inference	L1	L2	L3	L4	L5
-NH	12.057	11.980	12.026	11.884	11.758
-OH	11.527	11.722	11.596	11.834	11.418
-CH=N-	8.338	8.427	8.364	8.428	9.123
Aromatic	6.828-7.859	6.833-7.909	6.811-7.883	6.869-7.914	6.848-8.794
-OCH ₃	3.784	-	-	-	-
-CH ₃	-	-	2.324	-	-

REFERENCES

1. R. M. Patil and N. V. Thakkar, Synth. React. Inorg. Met.-Org.Chem., 30(6),1159 (2000). | 2. Rakesh Tada, Naimish Chavda and Manish K. Shah, J. Chem. Pharm. Res., 3(2), 290 (2011). | 3. Sunny S. Tarve, Sagar V. Sanap and Raju M. Patil, Indian Journal of Applied Research, 5(4), 823 (2015). | 4. Furnis BS, Hannaford AJ, Smith PWG and Tatchell AR. Vogel Textbook of Practical Organic Chemistry 5th Ed. ELBS Longman London, 1989. |