International Journal of Applied Research 2023; 9(1): 453-454



International Journal of Applied Research

ISSN Print: 2394-7500 ISSN Online: 2394-5869 Impact Factor: 8.4 IJAR 2023; 9(1): 453-454 www.allresearchjournal.com Received: 23-11-2022 Accepted: 28-12-2022

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Study of some new isatin derivatives

Dr. Lalan Kumar Jha

Abstract

The main objective in this paper deals with the synthesis and characterization of substituted 3 [(5-Benzylidene-2phenyl)-3.5 dihydro- 4H-imdazole-4-one-3-(4-benzoylhydrazono)] indole-2ones (vi). In that different isatin hydrazones (ii) are reacted with 2-phenyl 5-benzylidene 3N (4-acetyle phenyl) 1.5 dihydro-imidazole-4-one (v) to afford final compound (vi). The structures of newly synthesized compounds were characterized by IR, ¹HNMR and Mass Spectra.

Keywords: Isatin derivatives, benzylidene, benzoylhydrazono

Introduction

It is evident from literature than isatin derivatives are known to be associated with broad spectrum of biological activity like anti-bacterial anti-cancer anti-inflammatory analgesic anti-viral anti-fungal anti-tubercular and anti-depressent ^[1-3] Isatin hydrazones have been reported to posses anticonvulsant property also. This prompted us to synthesize some new isatin hydrazones containing imidazolone moiety.

Hence, the present paper comprises synthesis and characterization of 3-[(5-Benzylidene-2-phenyl)-35-dihydro-4H imidazole-4-one-3-(4-benzoylhydraz-ones)]-indole-2-ones (vi).

Methods

All the melting point were determined by open capillary method and are uncorrected. Purity of compound was checked by TLC on Silica Gel-GF coated plates. IR spectra was recorded (in KBR) on FTIR 8401 using Shimadzu instrument. ¹HNMR were recorded on 300 MHz Bruker DPX using CDCL₃ and Mass Spectra was determined using EI-MS technique on VG AUTOSPEC instrument.

Synthesis of isatin hydrazone

An appropriate istain or indole-2, 3 dione (I, 0.01mol) was dissolved in alcohol and added hydrazine hydrate (99%, 0.015 mol) while shaking. The for 3 hours. The resultant yellow crystalline solid was filtered Washed rapidly with small portion of cold water and finally with small quantity of cold alcohol. The product was dried and purified by recrystallization from chloroform. The compound thus obtained were characterized by comparison. With their physical constant reported in the lit [4-7].

M.W.: 161, M.F.: C₈H₇N₃O, M.P.: 220 °C. Yeild 74.5%

Similarly other derivatives of istinhydrazones are prepared by above method.

Synthesis of 2-phenyl 5-benzylidene -3N-(4-acetyl phenyl) 1,5-dihydro-imidazole-4one (v)

Equimolar quantity of p- amino methyl benzoate (iv, 0.001 mol) and 4-Bendylidence-2-phenyloxazole-5-one (iii, 0.01 mol) intimately mixed and heated over oil bath at 140-145 °C for 45-50 minute until jelly like mass was not obtained. The product was then filtered air dried and purified by recrystallization from methanol.

M.W.: 382, M.F.: C₂₄H₁₈N₂O₃, M.P.: 181 °C, yield 53%

Spectral Interpretation of compound

IR (KBr): 1280.65 cm^{-1} (O-C = O bending), 1712 cm^{-1} (CO streching), 2993 cm^{-1} (ArCH streching)

Corresponding Author: Dr. Lalan Kumar Jha Assistant Professor, Department of Chemistry, B.M. College, Rahika, Madhubani, Bihar, India MR (δ): 3.90 (s, 3H, CH3), 2.1 (s, 1H, CH), 7-8 (m, 14H, AR-H)

MASS: Molecular ion peak at m/z 546 and Base peak at m/z 105

Synthesis of 3 –[5-Benzylidene-2-pheny) 3,5-dihydro-4H-imidazole-4-one-1-(4- benzoyl- hydarzono)-indole-2-ones (vi): A mixture of equimolar quantity of isatin hydrazones (ii, 0.01 mol) and 2-phenyl 5-benzylidene 3N (4-acetyl phenyl) 1,5 dihydro-imidazole-4-one (v,0.01 mol) was dissolved in methanol containing a catalytic amount of potassium hydroxide and heated under refluxed for 5-6 hrs. The reaction mixture was cooled, and neutralized with

concentrated hydrochloric acid. Then filtered the resultant product, dried and purified by recrystallization from methanol.

M.W.: 546, M.F.: C₃₁H₂₁N₅O₃Cl, M.P.: 208 ⁰C. yield 80%

Spectral Interpretation of compound

IR (KBr): 1643 cm⁻¹ (CO stretching), 1689 cm⁻¹(CONH stretching), 2923 (ArCH stretching), 3253 cm⁻¹ (NH stretching)

NMR (δ): 10.1 (s, 1H, Aromatic NH), 10,2 (s,1H NHCO), 2.5(s,1H CH), 7-8 (m, 18H, AR-H)

Mass: Molecular ion peak at m/z 546 and Base peak at m/z 105

Table 1: Physical characterization data of synthesized compound

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S. N.	Compoud code	R	R	M.P. (° C)	Molecular Formula	Mocular Weight	Yeild (%)
1	Via	Н	Н	208	$C_{31}H_{21}O_3N_5$	509	83
2	Vib	5-CL	Н	252	C31H20O3N5Cl	544	73
3	Vic	5-F	Н	237	C ₃₁ H ₂₀ O ₃ N ₅ F	527	76
4	Vid	5-Br	Н	265	$C_{31}H_{20}O_3N_5Br$	590	80
5	Vie	4-L5-F	Н	232	$C_{31}H_{20}O_3N_5FCI$	563	72
6	Vif	5-CH ₃	Н	198	$C_{32}H_{23}O_3N_5$	524	67
7	Vig	5-NO ₂	Н	270	$C_{31}H_{20}O_5N_6$	555	63
8	Vih	5-COOH	Н	>300	$C_{32}H_{21}O_5N_6$	556	76
9	Vii	7-COOH	Н	215	$C_{32}H_{21}O_5N_5$	556	77
10	Vij	7-COOH ₃	Н	200	C33H23O5N5	570	51
11	Vik	Н	ACETYL	152	C33H23O4N5	552	71
12	Vil	Н	METHYL	143	C32H23O3N5	524	75
13	Vim	5-Br	ACETYL	183	C33H22O4N5Br	632	71
14	Vin	5-Br	METHYL	187	C32H22O3N5Br	604	70

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