

SYNTHESIS AND CHARACTERIZATION OF SOME NEW 3H-N-(SUBSTITUTED PHENYL)-1,2-BENZISOXAZOLES

V. Sareen, V. Khatri and V. Kumar

Department of Chemistry, University of Rajasthan, Jaipur-302004, India
E-mail: sareenparmod@yahoo.com

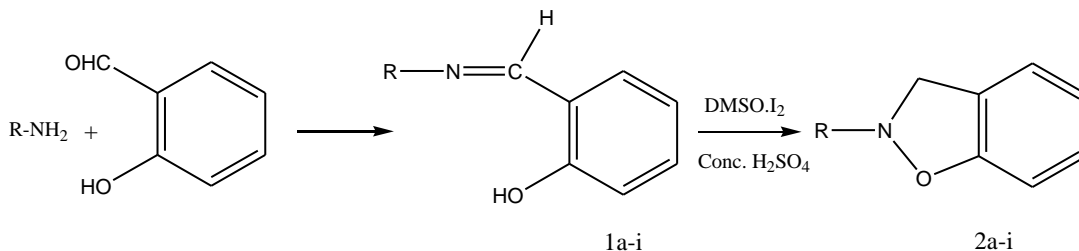
Abstract: Some new 3H-N-(2-substituted phenyl)-1,2-benzisoxazoles have been prepared by refluxing Schiff bases with DMSO.I₂-H₂SO₄. The structures of all these newly synthesized compounds have been confirmed by IR, ¹H NMR spectra and elemental analysis.

Introduction:

Isioxazole derivatives are found to possess biological and pharmaceutical activities such as antifungal,¹ antitumor,² antioxidant,³ antimicrobial,⁴ nematicidal,⁵ anti-inflammatory,⁶ anticancer,⁷ antiviral,⁸ treatment of leishmaniasis,⁹ and treatment of patients with active arthritis.¹⁰ In continuation of our work on heterocyclic compounds^{11,12} we have now synthesized some new 1, 2-benzisoxazoles derivatives. Benzisoxazole derivatives have been used in various biological activities such as antimicrobial activities.¹³

Experimental

Melting points were determined in open capillaries and are uncorrected. The IR spectra (cm⁻¹) were recorded on a SHIMADZU 8400S FT-IR spectrometer in KBr pellets. ¹HNMR spectra were recorded on BRUKER AVANCE II 400 spectrometer (300 MHz) using CDCl₃ as an internal standard. Chemical shifts are expressed in δ ppm. Purity of all the compounds were checked by TLC on silica gel plate.



R, a = 4-NO₂.C₆H₄,
b = 3-NO₂.C₆H₄,
c = 2, 5-diCl.C₆H₃,

d = 4-CH₃.C₆H₄,
e = 4-COOH.C₆H₄,
f = 4-SO₃H.C₆H₄,

g = 2-COOH.C₆H₄,
h = -CSNH₂,
i = -CSNH.C₆H₄

N-[(2-Hydroxyphenyl)-methylidiny]-4-Nitroaniline (1a)

It was prepared by refluxing the mixture of 4-nitroaniline (0.01 mol) and salicylaldehyde in ethanol (0.01mol) for 5-6 hrs on a water bath. The reaction mixture was cooled and crude product was crystallized from ethanol to give 1a.

3H-N-(4-Nitrophenyl)-1,2-benzisoxazole (2a)

3H-N-(4-nitrophenyl)-1,2-benzisoxazole was prepared by cyclization of N-[(2-Hydroxyphenyl)-methylidiny]-4-nitroaniline (0.01 mol) in DMSO (40 ml) and I₂ in presence of concentrated H₂SO₄ by heating the reaction mixture on water bath for 1 hr. After completion the mixture was poured into cold water, filtered and crystallized from ethanol to give 2a.

Result and discussion

I.R. spectra shows peaks at 1530 cm⁻¹ (C-N), 3050 cm⁻¹ (aromatic C-H), 1070 cm⁻¹ (C-O) and ¹HNMR shows peaks at δ 6.9-7.4 (aromatic protons) and at δ 2.38 (2H,d,>CH₂) ppm, which confirms the formation of 3H-N-(4-substituted phenyl)-1,2-benzisoxazoles.

Table 1: Physical and analytical data of the compounds

Compounds	M. P (°C)	Yield %	Mol. Formula	Elemental Analysis			
				N %		S%	
				Found	Calc.	Found	Calc.
2a	98	70	C ₁₃ H ₁₀ N ₂ O ₃	11.55	11.57	-	-
2b	138	67	C ₁₃ H ₁₀ N ₂ O ₃	11.58	11.57	-	-
2c	118	72	C ₁₃ H ₉ NOCl ₂	5.24	5.26	-	-
2d	140	62	C ₁₄ H ₁₃ NO	6.62	6.63	-	-
2e	106	60	C ₁₄ H ₁₁ NO ₃	5.77	5.80	-	-
2f	92	52	C ₁₃ H ₁₁ NO ₄ S	5.06	5.05	11.56	11.55
2g	122	55	C ₁₄ H ₁₁ NO ₃	5.79	5.80	-	-
2h	240	57	C ₈ H ₈ N ₂ OS	15.54	15.55	17.75	17.77
2i	126	59	C ₁₄ H ₁₂ N ₂ OS	10.92	10.93	12.49	12.50

References

1. M. M. M. Santos, N. Faria, J. Iley, S. J. Coles, M. B. Hursthouse, M. L. Martins, R. Moreira, *Bioorg. Med. Chem. Lett.* **20**, 193 (2010).
2. D. Patrizia, A. Carbone, P. Barraja, G. Kelter, H.H. Fiebig, G. Cirrincione, *Bioorg. Med. Chem.* **18**, 4524 (2010).
3. A. Padmaja, C. Rajasekhar, A. Muralikrishna, V. Padmavathi, *Eur. J. Med. Chem.* **46**, 5034 (2011).
4. D. Bajaj, O. A. Mahodaya, P. V. Tekade, *Heterocycl. Lett.*, **3**, 219, (2013).
5. A. Srinivas, A. Nagaraj, C. S. Reddy, *Eur. J. Med. Chem.* **45**, 2353 (2010).
6. T. Karabasanagouda, A. V. Adhikari, M. Girisha, *Indian J. Chem.* **48B**, 430 (2009).
7. A. Kamal, E. V. Bharathi, J. S. Reddy, M. Janaki, et al. *Eur. J. Med. Chem.* **46**, 691

- (2011).
8. Y. S. Lee, S. M. Park, B. H. Kim, *Bioorg. Med. Chem. Lett.* **19**, 1126 (2009).
 9. S. N. Suryawanshi, A. Tiwari, N. Chandra, Ramesh. S. Gupta, *Bioorg. Med. Chem. Lett.* **22**, 6559 (2012).
 10. X. H. Zhang, L. Y. Wang, Y. H. Zhan, Y. L. Fu, et al. *J. Mol. Struct.* **994**, 371 (2011).
 11. V. Sareen, V. Khatri, P. Jain and K. Sharma, *Phosphorus, Sulphur and Silicon*, **185**, 140 (2010).
 12. V. Sareen, U. Gupta, V. Khatri and S. Chugh, *Indian J. Heterocycl. Chem.*, **18**, 239, (2009).
 13. V. Sareen, U. Gupta, V. Khatri, S. Chugh, D. Shinde and S. Sareen, *Heterocycl. Lett.*, **1**, 25, (2011).

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