CODEN [USA]: IAJPBB ISSN: 2349-7750

INDO AMERICAN JOURNAL OF
PHARMACEUTICAL SCIENCES

http://doi.org/10.5281/zenodo.1217987

Available online at: http://www.iajps.com Research Article

GREEN SYNTHESIS OF N'-SUBSTITUTED-N"-[4-HYDROXY-3-(1H-INDOL-2-YL) PHENYLDICARBONODITHIOIMIDEDIAMIDE

D.T.Tayade* and R.D.Isankar

Department of Chemistry, Government Vidarbha Institute of Science and Humanities, Amravati 444 604, Maharashtra State, India.

Abstract:

In this work we synthesized a novel series of N'-substituted-N"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimidediamide by green synthesis parameters from N'-[4-hydroxy-3-(1H-indol-2-yl) phenyl] thiourea and substituted thioformamide. Previously such types of reactions were carried out in acetone medium but we developed new reaction condition in which percentage of acetone was reduced. During synthesis we carried out these reactions in various compositions of ethanol-acetone medium. This method has broad applications due to its efficiency for accelerating a course of organic reactions and increases yields, higher selectivity and lower quantities of byproducts. This method has consequently easier work-up and products obtained are in comparatively pure form. In 70% ethanol-acetone medium we got maximum yields. Justification and confirmations of structure of synthesized compounds were carried by usual elemental analysis, chemical characteristics and spectral studies.

Keywords: *N'-Substituted-N"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimide diamide, thioformamide.*

Corresponding Author:

D.T.Tayade,

Department of Chemistry,

Government Vidarbha Institute of Science and Humanities,

Amravati 444 604, Maharashtra State, India.

E-mail: ramisankarrd@gmail.com, skdtayade@gmail.com

QR code

Please cite this article in press D.T.Tayade and R.D.Isankar., ***Green Synthesis of N'-Substituted-N"-[4-Hydroxy-3-(1H-Indol-2-YL) Phenyldicarbonodithioimidediamide,*** *Indo Am. J. P. Sci, 2018; 05(04).*

INTRODUCTION:

Indole and 2,4-dithiobiureto nucleus containing heteroacycles and heterocycles are known for their important potent biological activities[1-11] as well as these can be easily used as a good intermediate[12-15] for synthesis of abundant significantly active numerous five, six and seven member heterocycles. It was observed n'-substituted-n"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimidediamide that sulphur and nitrogen of 2,4-dithiobiureto are responsible for changing biological applications. Literature survey showed that interactions of N'-[4-hydroxy-3-(1H-indol-2-yl)phenyl]thiourea and substituted thioformamide are still lacking, hence we carried out these interactions in ethanol-acetone medium obtained by somewhat suitable and eco-friendly green synthetic method.

RESULTS AND DISCUSSION:

Synthesis of N'-Ethyl-N"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimidediamide:

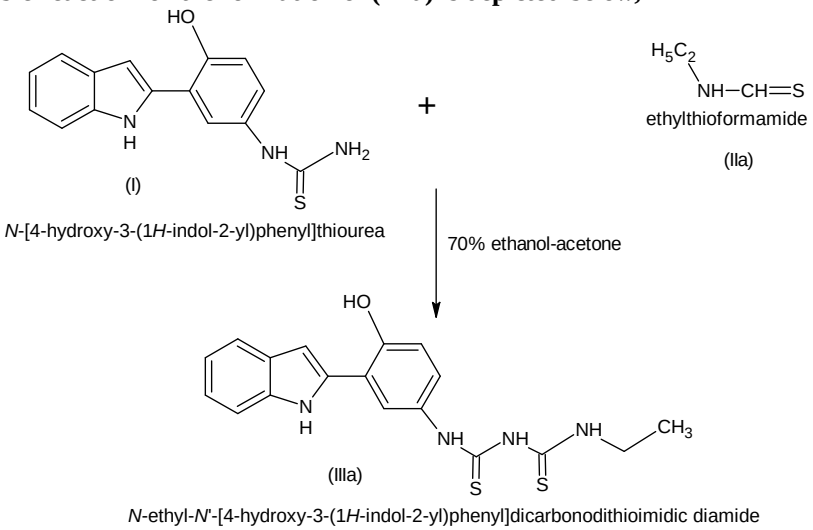
Interaction of N-[4-hydroxy-3-(1H-indol-2-yl)phenyl]thiourea (I) and ethylthio formamide was carried out in 70% ethanol-acetone medium for 2 hours on water bath. After distillation of excess solvent; pale yellow crystals of product were separated out. Recrystallised from aqueous ethanol. Yield- 92%, m.p. 202^{0}C . During developing reaction conditions it was observed that 70% ethanol-acetone mixture is the best solvent which increase yield of product as well as curtail time span of reaction by maintaining the purity of product. Results obtained during work is depicted in **Table No. 1**

Table No. 1

Sr. No.	Solvents	Time span (Hrs)	Yield (%)
1.	Acetone*	16	47
2.	Ethanol*	06	58
3.	Benzene	08	28
4.	Carban tetrachloride	12	32
5.	Ethanol-Acetone(40%)	03	60
6.	Ethanol-Acetone(50%)	03	68
7.	Ethanol-Acetone(60%)	03	74
8.	**Ethanol-Acetone(70%)**	**02**	**92**
9.	Ethanol-Acetone(80%)	03	66
10	Ethanol-Acetone(70%)	03	61

* **:** Literature solvents

The probable reaction for the formation of (IIIa) is depicted below,



Properties of (IIIa): Molecular formula: $C_{18}H_{18}N_4O_1S_2$, yellow crystalline solid, m.p. 202^0C. Rf value was found to be 0.35, by using dioxane solvent on silica gel-G having layer thickness 0.3 mm. Gave Lassigne's positive test for nitrogen and sulpur. It was desulphurised by alkaline plumbite solution indicating that sulphur is present in open chain. It gave green colour when neutral ferric chloride solution indicating that phenolic group is present. It gave picrate having m.p. 178^0. **Elemental analysis: Found (Calculated)** C: 57.75(57.30), H:4.50(4.49), N:15.78(15.73), S:18.00(17.97) **IR Spectrum** [(KBr pellets) cm^{-1}]: 3342.00 (O-H stretching); 3644.00 (N-H stretching); 2518.31 (C-H stretching); 1664.00(N=C-N stretching); 1684.00 (C=C stretching); 1596.00 (N-C=S stretching); 1149 (C-N stretching)., **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** indole -NH proton at δ12.8583 ppm, flanked thioamido -NH protons at δ 9.4863 ppm, Ar-H protons at δ 7.4358-6.8346 ppm, Ar-OH proton at δ 5.6753 ppm, terminal -NH proton at δ 3.4988 ppm,-CH_2 protons at δ 2.5367-2.3605 ppm, -CH_3 protons at δ 0.9651-0.8489 ppm.

Similarly,

N'-Methyl-N"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimidediamide, **N'**-t-butyl-N"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimidediamide, **N'**-phenyl-N"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimidediamide and **N'**-Cl-phenyl-N"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimidediamide were synthesized by interactions of N'-[4-hydroxy-3-(1H-indol-2-yl)phenyl]thiourea and substituted thioformamide (IIb), t-butyliso thioformamide (IIc), phenyl thioformamide (IId) and p-Cl-phenyl thioformamide (IIe) above mentioned method and given in **TableNo.2**

TableNo.2

Sr. No.	**N'-Substituted**-N"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimidediamide	Yield %	M.P. ^{0}C
1	**N'-Methyl**-N"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimidediamide	94	186
2	**N'**-t-**Butyl**-N"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimidediamide	92	208
3	**N'-Phenyl**-N"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimidediamide	90	192
4	**N'-p-Cl-Phenyl**-N"-[4-hydroxy-3-(1H-indol-2-yl) phenyldicarbonodithioimidediamide	93	190

REFERENCES:

1. Kerzarea D. R. and Khedekar P. B., *Indian J Pharm Sci Bioscientific Res.*, 6(1) **2016,** 144-156**.**
2. Dhani R, Avinash A., Salenaagina S. K., Saicharan M. V., Masthanaiah T., Rathnam P. R. and Chandana S. V., *J. Chem. Pharm. Res*., 3(5), **2011,** 519-523**.**
3. Gillman P. K., Bartlett J. R., Bridges P. K., Hunt A., Patel A. J., Kantamaneni B. D. and Curzon G., *J. Neurochem.*, **1981**, 37, 410–417.
4. Naoki M., Akihito T., Makiko N. Chie F. S, Chikawa T.I., Isomura T. and Nomura K. *Biotechnol. Biochem*., 74 (9), **2010,**1794–1801.
5. Abdulsalam A., Bouhfid R. and El Mokhtar E., *ARKIVOC* , (xii) , **2009,** 337-346 .
6. Synthesis of Indole and Oxindole Derivatives Incorporating Pyrrolidino, Pyrrolo or Imidazolo moieties, Stanley Rehn Stockholm, **2004**.
7. Tayade D.T., Waghmare S.A. , *American journal of pharmtech research*, 6(3), **2016,** 605-612**.**
8. D.T. Tayade , F.Z. Mohammad, *Ind. J of Pharmaceutical sci and research*, 2248-9126, 4(4), **2014**1-3,**.**
9. Tayade D.T., Thombare R.D. ,*European journal of pharmaceutical and medical research* , 3(3), **2016,** 443-446.
10. Lunge M.S., Shaikh R.S., Tayade D.T., *International Journal of Advanced Research*, 4(1), **2016,** 908-912**.**
11. Tayade D.T.,Padhen S.S., *European journal of pharmaceutical and medical research*, 3(8), **2016,** 538-540.
12. Tayade D.T.,Kale P.R., *International Journal of AppliedResearch*, 3(3), **2016,** 370-374**.**
13. Tayade D.T.,Ingole S.P., *European journal of pharmaceutical and medical research*, 3(7),**2016.**
14. Tayade D.T., Raghuwanshi M.R., Bhagwatkar A.K., Aswale S.R., *Canadian Int. J. chemistry*, 3(2), **2011**, 74-78.
15. Sing A.K., Mishra G. and Joyti K., *J. App. Pharm. Sci.*, 01(5), **2011**, 44-49. Ekhallas N., *Journal of American Sciences*, 6(8), **2010**, 54-57.