P: ISSN NO.: 2321-290X RNI : UPBIL/2013/55327 VOL-6* ISSUE-4*(Part-2) December- 2018
E: ISSN NO.: 2349-980X **Shrinkhla Ek Shodhparak Vaicharik Patrika**

Synthesis and Characterization of Some Thiazole Derivatives

Bharat Chaubey
Assistant Professor,
Dept. of Chemistry,
SVNPG College,
Kalan Sultanpur, U.P., India

Abstract

Substituted thiourea condensed with Chloro acetic acid in acidic medium to form thiazolidinone and then further this derivative reacts with aromatic aldehydes to give 2-imino-5-benzylidene -4- thiazolidinone. Further thiazolidinones reacts with hydrazine to formed thiazole-pyrazolines.

Keywords: Thiazolidinone, Thiazole-pyrazolines.

Introduction

Heterocyclic compounds are very widely distributed in nature and are essential to life in various ways. Thiazole is a well known heterocyclic compound with two hetero atoms. Sulphur and nitrogen at position 1 and 3. The tetra hydro derivatives of thiazole are known as thiazolidine and oxo-derivative of thiazolidine is called thiazolidinone.

Thiazole derivatives have important role in heterocyclic chemistry and are found wide applicability in the field of medicinal chemistry. These derivatives possess various types of biological activities such as pesticidal, insecticidal, anticonvulsant, antimicrobial, antibacterial, antifungal and antiviral.

Experimental

Melting points were taken in an open capillary tube and are uncorrected. The IR spectra were recorded in KBr on Perkin Elmer-720 spectrophotometer. The ^{1}H-NMR spectra were recorded in $CDCl_3$ on varian A-60 D spectrophotometer. The chemical shifts are recorded in ppm downfield from TMS, which are used as an internal standard.

Preparation of 2-substituted imino-4-thiazolidinone

To stirring solution of thiourea (10.133 g, 0.133 mol) in 100 ml water was added chloroacetic acid (12.613 g, 0.133 mol) in presence of concentrated HCl and was refluxed for 7 hours. The precipitate was collected and crystallized from water, ethanol mixture (50:50) to give 13g (83%) of 2-imino-4-thiazolidinone, m.p. 220^{0} C.

Preparation of 2-imino-5-substituted benzylidene-4-thiazolidinone

A mixture of 2-imino-4-thiazolidinone (11.5 g, 0.10 mol) and benzaldehyde (10.6 g, 0.10 mol) and 25 ml glacial acetic acid were taken. The entire contents of the flask were refluxed for four hours and cooled to room temperature. The product was filtered and crystallized in ethanol water mixture (50:50) to give 15.1 g (75%) of 2- imino-5-substituted benzylidene-4-thiazolidinone.

Preparation of 2- phenylimino-5-benzylidene-4-thiazolidinone

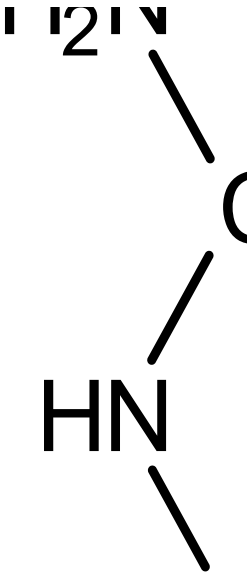
11g (74%) was obtained by the reaction of 2-imino-phenyl-4-thiazolidinone 10g (0.05 mol) and 5g (0.05 mol) of benzaldehyde in 25 ml of glacial acetic acid. The colourless solid was obtained, m.p.224^{0} C.

Preparation of 3-substituted phenyl-2,6-dihydro-5H-pyrazole [3,4-d] [1,3]thiazol-5-imine-4,5-dihydro-pyrazole [4,3-e] [1,3]thiazin-6 [H]-imine

10.2 g (0.05 mol) of 2-imino-5-benzylidene-4-thiazolidinone 1.6 g (0.05 mol) of hydrazine and 20 ml of glacial acetic acid were taken in a 100 ml round bottom flask. The mixture was refluxed for three hours and left overnight. The crystals that separated were filtered at pump, washed with ethanol and crystallized from ethanol, water mixture (50:50)to give 14.7 g (68%) of 3-substituted phenyl-2,6-dihydro-5H-pyrazole [3,4-d] [1,3]thiazol-5-imine-4,5-dihydro-pyrazole [4,3-e] [1,3]thiazin-6 [H]-imine.

Result and discussion

Substituted thiourea (I) condensed with chloroacetic acid (II) in acidic medium to form thiazolidinone (III) and then further this derivative reacts with aromatic aldehydes to give 2-imino-5-benzylidene-4-thiazolidinone (IV). Further thiazolidinone (IV) react with hydrazine to formed thiazole-pyrazolines (V) (scheme-1).



2-imino-5-substituted-benzylidene-4-thiazolidinone

The reaction of 2- imino-4-thiazolidinone with benzaldehyde in equimolar proportion in presence of glacial acetic acid at room temperature yielded crystals of 2-imino-5 -benzylidene-4-thiazolidinone filtered, dried and recrystallised from ethanol: water. The elemental analysis of this compound corresponds to molecular formula $C_{10}H_8N_2SO$. The IR spectrum of this compound shows characteristic absorption bands at 1620 cm^{-1} and 1730 cm^{-1} for C=O and C=N group respectively. UV spectrum shows λ_{max} 240 and 250 nm for C=O and C=N grouping. PMR spectrum exhibits singlet (1H) at δ 2.6 for CH proton, multiplet (5H) at δ 7.30 – 7.80 for aromatic protons and singlet (2H) at δ 8.10 for NH proton.

2-phenylimino-5-substituted-benzylidene-4-thiazolidinone

The reaction of 2-phenylimino-4-thiazolidinone with a benzaldehyde in equimolar quantity in presence of glacial acetic acid at room temperature yielded crystals of 2-phenylimino-5 -benzylidene-4-thiazolidinone filtered, dried and recrystallised from ethanol: water. The elemental analysis of this compound corresponds to molecular formula $C_{16}H_{12}N_2SO$. The IR spectrum of this compound shows characteristic absorption bands at 1653 cm^{-1} and 1660 cm^{-1} for C=O and C=N group respectively. UV spectrum shows λ_{max} 245 nm for C=N. Grouping PMR spectrum of compound exhibits singlet (1H) at δ 2.7 for CH proton, multiplet (9H) at δ 7.25 – 7.65 for aromatic protons and singlet (2H) at δ 8.08 for NH proton.

3-substituted phenyl-2,6-dihydro-5H-pyrazolo [3,4-d] [1,3]thiazol-5-imine-4,5-dihydro-pyrazolo [4,3-e] [1,3]thiazin-6 [H]-imine

The reaction of 2-imino-5-sustituted-benzylidene-4-thiazolidinone with hydrazine hydrate in equimolar quantity in presence of glacial acetic acid at room temperature yielded crystals of 3-substituted phenyl-2,6-dihydro-5H-pyrazolo [3,4-d] [1,3]thiazol-5-imine-4,5-dihydro-pyrazolo [4,3-e] [1,3]thiazin-6 [H]-imine, filtered, dried and recrystallised from ethanol: water. The elemental analysis of this compound corresponds to molecular formula $C_{10}H_8N_4S$. The IR spectrum of this compound shows characteristic absorption bands at 1612 cm^{-1} and 756 cm^{-1} for C=N and C—S group respectively. UV spectrum shows λ_{max} 240 – 260 nm for C=N. Grouping PMR spectrum exhibits multiplet (5H) at δ 7.22 – 8.27 for aromatic protons and singlet (3H) at δ 8.11 for NH proton.

Aim of the Study

Most of the alkaloids which are nitrogenous bases occuring in plants and many antibiotics contain heterocyclic ring systems. A large number of heterocyclic compounds obtainable only by laboratory synthesis, have valuable properties as chemotherapeutic agents, drugs, dyestuffs, copolymers ect. This dissertation deals with the studies on thiazole derivatives.

Conclusion

All the compounds shows antifungal activities to some extent and these may be treated as antifungal agents. Thus nitrogen and sulphur containing heterocyclic conpounds have fungicidal activity.

References

A.D. Kennedy and A.L. Summers, J Heterocyclic chem, 18,409 (1981).

B.S. Holla, C.S. Prasanna, B. Poojary, K.S. Rao, K. Shridhara and U.G. Bhat, Indian J. chem Sect. B, 43, 864 (2004).

N.J. Gaikwad, M. Yunus, H.A. Husain and D.B. Meshram, Indian.J. Heterocyclic chem 12, 165 (2002).

A. Kshirsagar, M.P. Turaskar, V.M. Kulkarni, S. Dhanashire and V Kadam International J. of chem.Tech. research 1, 696 (2009).

C.G. Bande and N.J. Gaikwad, Bioorg. Med. Chem 12, 215 (2004).

N. Arikan, D. Sumeregen and B. Dulger Turk J. chem. 32,147 (2008).

R. Sharma, D.P. Nagda and G.L. Talesara Arkivoc, 1,1 (2006).

Salman Ahmad Khan, Mohammad Yusuf European Journal of medicinal chemistry 44, 2597-2600 (2009).

B. Shivarama, Holla, KV Malini, B. Sooryanarayana Rao, B.K. Sarojini N. Sucheta Kumari, European Journal of medicinal chemistry 38, 313-318 (2003).

N.P. Das and As Mittra, J. Ind. Chem. Soc 55, 907 (1978).

N. Kaushik Basu, A Bopda-Waffo, A. Basu, Frontiers in Bio Science 13, 3857 (2008).