INTERNATIONAL JOURNAL OF PURE AND APPLIED RESEARCH IN ENGINEERING AND TECHNOLOGY

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SPECIAL ISSUE FOR INTERNATIONAL LEVEL CONFERENCE "ADVANCES IN SCIENCE, TECHNOLOGY & MANAGEMENT" (IC-ASTM)

CHARACTERIZATION AND ANTIMICROBIAL STUDY OF NEWLY SYNTHESIZED ARYL THIOCARBAMIDES AND RESPECTIVE DITHIAZOLIDENES

SAMIDHA S. KADU[1], GAJANAN V. KORPE[2]

1. J. D. Patil Sangludkar Mahavidyalaya, Daryapur Dist. Amravati
2. P.G. Department of Chemistry, Shri. Shivaji College, Akola (MS) India.

Accepted Date: 05/09/2017; Published Date: 10/10/2017

Abstract: Several and bis-thiocarbamides and respective 1,2,4-dithiazolidines were synthesized by the interaction of glycosylisothiocyanates and *o*-phenylenediamine and further with N-phenyl-S-chloroisothiocarbamoyl chloride. The identities of these newly synthesized compounds were established on the basis of usual chemical transformations, IR, ^{1}H NMR, ^{13}C NMR and Mass spectral studies. All the synthesized compounds have been evaluated for their antibacterial and antifungal activity against different bacteria and fungi by agar diffusion method.

Keywords: Machine Translation, Natural Language Understanding, MT evaluation, Interlingua.

Corresponding Author: SAMIDHA S. KADU

Co Author: - GAJANAN V. KORPE

Access Online On:

www.ijpret.com

How to Cite This Article:

Samidha S. Kadu, IJPRET, 2017; Volume 6 (2): 284-287

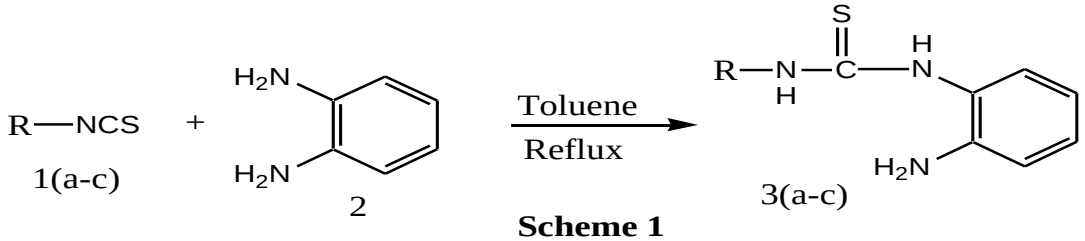
PAPER-QR CODE

INTRODUCTION

Thiourea and its derivatives represent well-known important group of organic compounds due to the diverse application in fields such as medicine, agriculture, coordination, and analytical chemistry[1]. They also can be used as selective analytical reagents, especially for the determination of metals in complex interfering materials[2-4].
Isothiocyanates are precursors of a wide range of N-thiocarbamoyl derivatives; their tendency to undergo nucleophilic additions and cycloadditions make them highly important intermediates in organic synthesis[5] for the preparation of heterocyclic compounds[6,7]. Thus heterocyclic compounds have been used as anti-tumoral[8,9] or antiviral agents, including AIDS[10,11] and hepatitis B[12,13] treatments.
In view of the advantage conferred by thiourea and 1,2,4-dithiazolidenes, it was interesting to carry out synthesis of various thiocarbamides and 1,2,4-dithiazolidenes derivatives by the interaction of and aryl isothiocyanate with *O*-phenylenediamine and further with N-phenyl-S-chloroisothiocarbamoyl chloride. (**Scheme 1 and Scheme 2**).

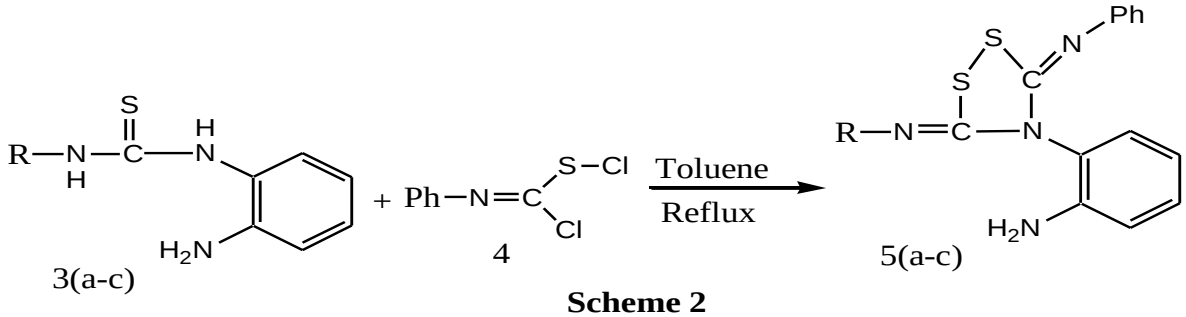
EXPERIMENTAL

All melting points are uncorrected and were obtained in capillary using paraffin bath. Specific rotations of the newly synthesized compounds were measured on an Equip-Tronic digital polarimeter model no. EQ 801 at 30^{0}C in $CHCl_3$. IR spectra were recorded on a Shimatzu FTIR spectrophotometer, ^{1}H NMR on a Bruker DRX-300 (300 MHz FT) NMR spectrometer in $CDCl_3$ solution with TMS as an internal reference. The Mass spectra were recorded on a Jeol SX -102 FAB mass spectrometer. Purity of the compound was checked by thin layer chromatography using merck silica gel-coated aluminium plates and petroleum ether: ethyl acetate as eluent and iodine vapours as a visualizing agent.



Scheme 1

Where R= 1. Phenyl 2. *o*-tolyl 3. *o*-chloro phenyl



Scheme 2

Characterization data of compounds 3(a-c) and 5(a-c) Table-1

Compound	% yield	M.P. (^{O}C)
3a	76	244
3b	67	188
3c	80	176
5a	79	196
5b	65	183
5c	82	210

Preparation of aryl thiocarbamides3(a-c)

These have been prepared by the interaction of various aryl isocyanates which were prepared by earlier known method and *o*-phenylenediamine in boiling toluene medium.(**Scheme 1**)

Preparation of N-phenyl-S-chloro isothiocarbamoyl chloride (4):

The required N-phenyl-S-chloro isothiocarbamoyl chloride was prepared by earlier known method by controlled chlorination of phenyl isothiocynate.

Preparation of 3-phenylimino-4-phenyl-(*o*-amino)-5-phenyl imino-1,2,4 dithiazolidines 5(a-c)

To an ice cold chloroformic solution of 1-phenyl-o-amino-3-phenylthiocarbamide **(3a)** ice cold chloroformic solution of N-phenyl-S-chloro isothiocarbamoyl chloride **(4)** was added and after 1h at room temperature the reaction mixture was gently refluxed for 3h. Reaction was monitored by TLC. After the completion of reaction solvent chloroform was distilled off and the resultant syrupy mass was triturated several times with light petroleum (60-80^{O}C) to afford a pale yellow solid.

This solid was basified with cold dilute ammonium hydroxide solution to 3-phenylimino-4-phenyl-(*o*-3'-phenyl thiocabamide)-5-phenyl imino-1,2,4 dithiazolidines **5(a)** as granular solid. This was crystallized from ethanol. The product was found to be non-desulphurizable when boiled with alkaline plumbite solution. m.p. 144°C. (**Scheme 2**)

All other aryl limino-1,2,4 dithiazolidine **(3b-f)** were prepared in a similar manner.

1-phenyl(o-amino)-3-phenyl thiocarbamide (3a):

IR (KBr) ν cm^{-1} : 3340, 3389 (N-H str.), 3012 (Ar. C-H str.), 2960 (Ali. C-H str.), 1371 (Ar. C-N str.),

1-phenyl(o-amino)-3-o-tolyl thiocarbamide (3b):

IR (KBr) ν cm^{-1} : 3440, 3370 (N-H str.), 2945 (Ali. C-H str.), 1369 (Ar. C-N str.), 1230 (C=S str.) ; **^{1}H NMR** in $CDCl_3$ at δ ppm: 2.88 (3H, s, CH_3), 5.45 (2H, s, NH), 3.55 (1H, s, NH), 2.75 (1H, s, NH), 6.98-7.84 (8H, m, Ar-H);

1-phenyl(o-amino)-3-o-chlro phenyl thiocarbamide (3c):

IR (KBr) ν cm^{-1} : 3380, 3290 (N-H str.), 2935 (Ali. C-H str.), 1369 (Ar. C-N str.), 1228 (C=S str.); **^{1}H NMR** in $CDCl_3$ at δ ppm: 3.81 (3H, s, CH_3), 5.5 (2H, s, NH), 4.05 (1H, s, NH), 3.75 (1H, s, NH), 7.21-7.56 (8H, m, Ar-H);

3-phenylimino-4-phenyl-(*o*-amino)-5-phenyl imino-1,2,4 dithiazolidines 5(a)

IR (KBr) ν cm^{-1} : *3340, 3389 (N-H str.),* 3012 (Ar. C-H str.), 2960 (Ali. C-H str.), 1371 (Ar. C-N str.), ; **^{1}H NMR** in $CDCl_3$ at δ ppm: 5.45 (2H, s, NH), 7.84-8.34 (14H, m, Ar-H);

3-phenylimino-4-phenyl-(*o*-amino)-5-tolyl imino-1,2,4 dithiazolidines 5(b)

IR (KBr) ν cm^{-1} : 3440, 3370 (N-H str.), 2945 (Ali. C-H str.), 1369 (Ar. C-N str.), 1230 (C=S str.) ; **^{1}H NMR** in $CDCl_3$ at δ ppm: 2.88 (3H, s, CH_3), 5.45 (2H, s, NH), 7.28-7.64 (13H, m, Ar-H);

3-phenylimino-4-phenyl-(*o*-amino)-5-tolyl imino-1,2,4 dithiazolidines (5c):

IR (KBr) ν cm^{-1} : 3380, 3290 (N-H str.), 3035 (Ar. C-H str.), 1369 (Ar. C-N str.), **^{1}H NMR** in $CDCl_3$ at δ ppm: 4.58 (2H, s, NH), 7.53-8.01 (13H, m, Ar-H);

ANTIMICROBIAL ACTIVITY:

These newly synthesized thiocarbamides and dithiazolidenes were screened for their microbial activity against different pathogenic microbes for their antibacterial and antifungal activities using well method. The compounds were screened for antibacterial activity against *E. coli*, *S. aureus*, *P. vulgaris* and for antifungal activity against *C. albicancs* and *A. niger* in potato dextrose agar medium.

Procedure for antimicrobial screening:

Media used (Nutrient broth) : Peptone – 10 g, NaCl – 10 g and yeast extract 5 g, Agar 20 g in 1000 ml of distilled water. Initially, the stock culture of bacteria were revived by inoculating in broth media and grown at 37 ^{0}C for 18 h. The agar plates of the above media were prepared and wells were made in the plate. Each plate was inoculated with 18 h old culture (100 µL, 10^4cfu) and spread evenly on the plate. After 20 min. the wells were filled with different concentrations of samples. The control wells were filled with Gentamycin. All the plates were incubated

at 37 ^{O}C for 24 h and the diameter of inhibition zones were noted in mm. The activity was quantitatively assessed on the basis of inhibition zone.

Most of the compound showed strong to moderate activity while some showed poor activity against the tested microorganisms.

ACKNOWLEDGEMENT

Authors are thankful to CDRI, Lucknow and to SAIF, Chandigarh for providing spectral data. Authors also thank Dr. S. P. Deshmukh, Head Department of Chemistry, Head, Department of microbiology and Dr. S. G. Bhadange, Principal, Shri. Shivaji College, Akola and Dr. R. M. Bhise Principal J. D. Patil Sangludkar Mv., Daryapur for encouragement and providing necessary facilities.

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