Available online www.jocpr.com

Journal of Chemical and Pharmaceutical Research, 2016, 8(8):1027-1029

Research Article

ISSN : 0975-7384
CODEN(USA) : JCPRC5

Design and characterization of 1-phenyl-3-[4-(2-t-butylimino-4-substituted imino-1, 3, 5-dithiazino) aminophenyl]-prop-2-ene-1-ones

Sanghapal S. Padhen[1*] and Dipak T. Tayade[2]

[1]*Department of Chemistry, Rajarshee Shahu Science College, Chandur Rly Dist Amravati -444 604 (MS) India*
[2]*Department if Chemistry, Govt. Vidarbha Institute of Science & Humanities, Amravati-444 604(MS) India*

ABSTRACT

*Recently in this laboratory series of 1-phenyl-3-[4-(2-t-butylimino-4-substituted imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIca-ce)** had been synthesized by the interaction of 1-phenyl-3-[4-(5-t-butyl-2,4-dithiobiureto) phenyl]-Prop-2-ene-1-ones **(Vc)** with various isocyanodichlorides **(VIIa-e)** in acetone medium. The reaction mixture was reflux 4 hours and filtered in hot condition. After distillation of excess of solvents crystals were separated out, this on basification with ammonium hydroxide gave product. The structure of all synthesized compounds were justified on the basis of chemical characteristics, elemental analysis and spectral studies.*

INTRODUCTION

Recently heterocyclic compounds are synthesize from previous one used as an intermediate. Heterocyclic compounds are more intriguing due to their utility in various fields. Nowadays the drug containing 1,3,5-dithiazino or 1,3,5-thiadiazino nucleus are widely used[1-5]. Also the literature survey reveals that when the compounds containing 1,3,5-dithiazino or 1,3,5-thiadiazino molecule as a parent nucleus then that molecule will enhance potency of that drug in medicinal, agricultural and industrial fields [7-11]. Dithiazines are also found to be effective on treatment of cancer [12].

We wish to report herein a simple and rapid procedure for the synthesis of 1-phenyl-3-[4-(2-t-butylimino-4-substitutedimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIca-ce)** had been synthesized by the interaction of 1-phenyl-3-[4-(5-t-butyl-2,4-dithiobiureto) phenyl]-Prop-2-ene-1-ones **(Vc)** with various isocyano dichlorides **(VIIa-e)** in acetone medium.

EXPERIMENTA SECTION

Materials

All the chemical used in the present research were MERCKS (India Made). Starting compounds (Ia-e) were synthesized by literature method [6].

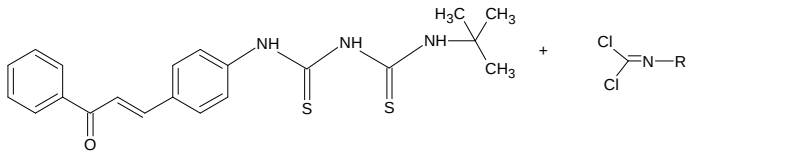
Method

Method adopted for the synthesis of all the compounds in the present investigation was conventional refluxing under water bath to attain constant temperature. Melting points of all the synthesized compounds estimated using paraffin oil and uncorrected. The carbon, hydrogen and nitrogen analysis was carried out on Carlo-Ebra-1106 analyzer and Colman-N-analyzer-29 respectively. IR spectra were recorded on SCIMADZU FTIR spectrometer in the range 4000-400 cm-1:[1] in KBr pellets. PMR spectra were recorded on BRUKER AVANCE II 400 NMR spectrometer with TMS as an internal standard using $CDCl_3$ and DMSO-d_6 as a solvent.

General Procedure

1-phenyl-3-[4-(2-t-butylimino-4-substituted imino-1,3,5-dithiazino)-aminophen yl]-prop-2-ene-1-ones **(VIIIca-ce)** had been synthesized by the interaction of 1-phenyl-3-[4-(5-t-butyl-2,4-dithiobiureto) phenyl]-Prop-2-ene-1-ones **(Vc)** with various isocyano dichlorides **(VIIa-e)** in acetone medium. The reaction mixture was reflux 4 hours and filtered in hot condition. During heating reactant dissolved into the solvent. After distillation of excess solvent yellow crystals were obtained, which recrystalized from glacial acetic acid to obtain 1-phenyl-3-[4-(2-substitued imino-4-substituted imino-1,3,5-dithiazino)-aminophen yl]-prop-2-ene-1-ones **(VIIIca-ce)**

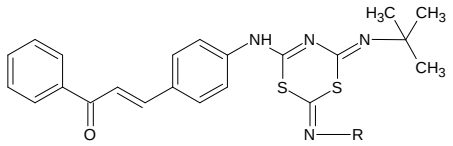
The tentative reaction is given below**,**



1-phenyl-3-[4-(5-t-butyl-2,4-dithiobiureto)-phenyl]- prop-2-ene-1-ones substitutedisocyanodichlorides

(Vc) **(VIIa-e)**

Acetone



1-phenyl-3-[4-(2-t-butyl imino-4-substituted imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-(

(VIIIca-ce)

(R= allyl, ethyl, t-butyl, phenyl, p-Cl-phenyl.)

Similarly,1-phenyl-3-[4-(2-t-butylimino-4-allylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIca),** 1-phenyl-3-[4-(2- t-butylimino -4-ethylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIcb),** 1-phenyl-3-[4-(2- t-butylimino -4-t-butylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIcc)**, 1-phenyl-3-[4-(2- t-butylimino -4-phenyl imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIcd)** and 1-phenyl-3-[4-(2- t-butylimino-4-p-Cl-phenylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIce)** were synthesized by the interaction of 1-phenyl-3-[4-(5- t-butyl-2,4-dithiobiureto)-phenyl]- prop-2-ene-1-one**(Vc)** with allylisocyanodichloride (VIIa), ethylisocyanodichloride (VIIb), t-butylisocyanodichloride (VIIc), phenylisocyanodichloride (VIId) and p-Cl-phenylisocyano dichloride (VIIe). As per above mentioned method.

RESULTS AND DISCUSION

Elemental and IR Spectra and PMR spectral analysis of all the synthesized compound is given below,

1-phenyl-3-[4-(2-t-butylimino-4-allylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones (VIIIca)

lemon yellow solid, $C_{25}H_{26}N_4OS_2$, Yield-82%, M.P.-192^{0}C Composition-found(calculated) C-63.89 (64.90), H-6.65 (5.66), N-11.11 (12.11) and S-12.85 (13.86); **FTIR (KBr) ν cm^{-1}:** 3049.82 (ArC-H stretching), 3286.62 (N-H stretching), 1653.48 (C=O stretching), 1477.71 (S-C=N stretching) and 685.42 (C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H, -CH=CH- at δ 3.22-3.74ppm, multiplet of 9H of Ph at δ 6.83-8.00ppm, singlet of 1H of –NH at δ 8.31ppm, quintet of 1H and double doublet of 2H of allyl at δ2.26, 1.13 and 2.34respectively, singlet of 9H, CH_3 at δ 1.39ppm; Mol. Wt.: 522.

1-phenyl-3-[4-(2- t-butylimino -4-ethylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones (VIIIcb)

Dark yellow solid, $C_{24}H_{26}N_4OS_2$, Yield-72%, M.P.-185^{0}C Composition-found(calculated) C-62.96 (63.97), H-6.82 (5.82), N-11.41 (12.43) and S-13.21 (14.23); **FTIR (KBr) ν cm^{-1}:** 3047.16 (ArC-H stretching), 3276.19 (N-H stretching), 1676.35 (C=O stretching), 1481.14 (S-C=N stretching) and 667.16 (C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H of –CH=CH- at δ 2.74-3.78ppm, multiplet of 9H of Ph at δ 6.97-7.93ppm, singlet of 1H of –NH at δ 8.66ppm, singlet of 9H, CH_3 at δ 1.54ppm, quartet of 2H and triplet of 3H of ethyl at δ1.59 and δ 1.37ppm respectively; Mol. Wt.: 450.

1-phenyl-3-[4-(2- t-butylimino -4-t-butylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones (VIIIcc)
Yellow solid, $C_{26}H_{30}N_4OS_2$, Yield-75%, M.P.-188^{0}C Composition-found(calculated) C-64.23 (65.24), H-7.35 (6.32), N-10.69 (11.70) and S-12.28 (13.40); **FTIR (KBr) ν cm^{-1}:** 3040 (ArC-H stretching), 3267.12 (N-H stretching), 1673.67 (C=O stretching), 1489.23 (S-C=N stretching) and 730.97 (C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 3.22-3.76ppm, multiplet of 9H of Ph at δ 6.83-7.92ppm, singlet of 1H of –NH at δ 8.36ppm, singlet of 18H of CH_3 at δ 2.54-3.43 ppm; Mol. Wt.:478.

1-phenyl-3-[4-(2- t-butylimino -4-phenyl imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones (VIIIcd)
Yellow solid, $C_{28}H_{26}N_4O_3S_2$, Yield-69%, M.P.-179^{0}C Composition-found(calculated) C-66.40 (67.44), H-6.23 (5.26), N-10.23 (11.24) and S-11.89 (12.86); **FTIR (KBr) ν cm^{-1}:** 3049.46 (ArC-H stretching), 3279.58 (N-H stretching), 1653.54 (C=O stretching), 1484.19 (S-C=N stretching) and 688.16 (C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H of –CH=CH- at δ 2.32-3.29ppm, multiplet of 9H of Ph at δ 6.99-7.95ppm, multiplet of 5H, Ph at δ 6.45-7.21ppm, singlet of 9H, CH_3 at δ 1.38ppm; Mol. Wt.: 498.

1-phenyl-3-[4-(2-t-butylimino-4-p-Cl-phenylimino-1,3,5-dithiazino)-aminophenyl]-prop -2-ene-1-ones (VIIIce)
Yellow solid, $C_{28}H_{25}N_4OS_2Cl$, Yield-76%, M.P.- 191^{0}C Composition-found(calculated) C-62.07 (63.08), H-5.7 2 (4.73), N-9.55 (10.51), S-11.01 (12.03) and Cl-7.60 (6.65); **FTIR (KBr) ν cm^{-1}:** 3042.16 (ArC-H stretching), 3262.07 (N-H stretching), 1664.16 (C=O stretching), 1482.16 (S-C=N stretching) and 687.01 (C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 2.21-3.41ppm, multiplet of 9H of Ph at δ 6.87-8.02ppm, multiplet of 4H, Ph at δ 6.63-7.32ppm, singlet of 9H, CH_3 at δ 1.33ppm and singlet of 1H of –NH at δ 8.37ppm; Mol. Wt.: 505.5.

CONCLUSION

All the synthesized compound were analyzed, found and confirmed by their elemental study, IR spectra and PMR spectra.

REFERENCES

[1] Tayade D.T., Kshirsagar A.M., *Der. Chemica. Sinica*, 4(4), **2013,** 25-35. 145.
[2] Kumar S., Upmanya N., Afzal O., bawa S., *Chemical Biology and Drug Design*, DOI:10.1111/cbdd.12339, **2014**.
[3] Bhagwatkar A.K., Tayade D.T., *Orbital Brazi. Elec. J. Chem*., 3(1), **2011,** X-2-4.
[4] Tayade D.T., Raghuwanshi M.R., Bhagwatkar A.K., Aswale S.R., *Canadian Int. J. chemistry*, 3(2), **2011**, 74-78.
[5] Tayade D.T., Pund D.A., Bhagwatkar R.A., Patil S.U., *Ind. J. Chem. Sci*., 8(3), **2011**, 1695-1698.
[6] Tayade D.T., "A Contribution to the chemistry of nitrogen, nitrogen and sulphur containing heteroacyclic and heterocyclic compounds", *Ph.D. Thesis, Amravati University, Amravati*, **1996**.
[7] Pund D.A, Bhagwatkar R. A, Tayade D.T, Rathod D.B., *Rasayan J. Chem.*, 3(2), **2010**, 246-249.
[8] Deshmukh A.Y, Rathod D.B, Tayade D.T and Patil S.U, Bhagwatkar R.A, *Asian Journal of Chemistry*, 22(10), **2010,** 8252-8254.
[9] Tayade D.T, Bhagwatkar R. A, and Panpalia R. C, *Canadian Int. J. of Chem*, 2(2), **2010,** 40-43.
[10] Tayade D.T., Pund D. A., Bhagwatkar R. A., Rathod D. B., Bhagwatkar N.A, *Canadian Int. J. of Chem.*, 3(1), **2010,** 36-41.
[11] Patil S.U., Raghuvanshi P.B., Tayade D.T., *J. Ind. Chem Soc*., 84, **2007**.
[12] Zhang Y.,Qiao R.Z. and Zhang Z.Y., *J.Chin.Chem.Soc.*, 49(3), **2002,**369.