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Synthesis and Characterization of 1-Phenyl-3-[4-(2-Substitutedimino-4-Substitutedimino-1,3,5-Dithiazino)Aminophenyl]-Prop-2-Ene-1-Ones

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ABSTRACT

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

INTRODUCTION

The heterocyclic compounds containing drugs having 1,3,5-dithiazino or 1,3,5-thiadiazino nucleus are widely used in medicinal, biochemical, biotechnological and pharmaceutical sciences[1-8]. In the view of nucleus containing dithiazine ring possess anti-helminthic, antifungal, antiviral, antibacterial and anti-tuberculostatic properties[9-10] had been reported. The literature survey reveals that the heterocyclic compounds having 1,3,5-thiadiazine nucleus enhanced its medicinal, agricultural and industrial activities of the drugs and medicines. 1,3,5-dithiazino nucleus shows fungicidal[11] and insecticidal[12] properties.

We wish to report herein a simple and rapid procedure for the synthesis of 1-phenyl-3-[4-(2-substitued imino-4-substituted imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIba-be)** which had been synthesized by the interaction of 1-phenyl-3-[4-(5-ethyl-2,4-dithiobiureto) phenyl]-Prop-2-ene-1-ones **(Va)** with various isocyanodichlorides**(VIIa-e)** in acetone medium.

MATERIALS AND METHOD

Materials

All the chemicals used in the present research were MERCKS (India Made). Starting compounds (Ia-e) were synthesized by literature method[13].

Method

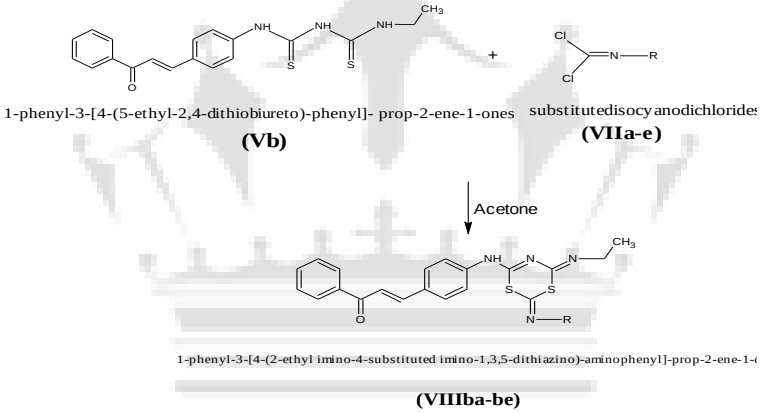
Method adopted for the synthesis of all the compounds in the present investigation was conventional refluxing under water bath to attain constant temperature. Melting points of all the synthesized compounds estimated using paraffin oil and uncorrected. The carbon, hydrogen and nitrogen analysis were carried out on Carlo-Ebra-1106 analyzer and Colman-N-analyzer-29 respectively. IR spectra were recorded on SHIMADZU FTIR spectrometer in the range 4000-400 cm^{-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.

EXPERIMENTAL

General Procedure

1-phenyl-3-[4-(2-substitued imino-4-substituted imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIba-be)** had been synthesized by the interaction of 1-phenyl-3-[4-(5-ethyl-2,4-dithiobiureto) phenyl]-Prop-2-ene-1-ones **(Vb)** with various isocyanodichlorides**(VIIa-e)** in acetone medium. The reaction mixture was refluxed for 4 hours and filtered in hot condition. During heating, reactant dissolved into the solvent. After distillation of excess solvent yellow crystals were obtained, which recrystallized from glacial acetic acid to obtain 1-phenyl-3-[4-(2-substitued imino-4-substituted imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIba-be).**

The tentative reaction is given below**:**



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

Similarly, 1-phenyl-3-[4-(2-ethylimino-4-allylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIba),** 1-phenyl-3-[4-(2-ethylimino-4-ethylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIbb),** 1-phenyl-3-[4-(2-ethylimino-4-t-butylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIbc)**, 1-phenyl-3-[4-(2-ethylimino-4-phenyl imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIbd)** and 1-phenyl-3-[4-(2-ethylimino-4-p-Cl-phenylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIbe)** were synthesized by the interaction of 1-phenyl-3-[4-(5-ethyl-2,4-dithiobiureto)-phenyl]- prop-2-ene-1-one**(Vb)** with allylisocyanodichloride (VIIa), ethylisocyanodichloride (VIIb), t-butylisocyanodichloride (VIIc), phenylisocyanodichloride (VIId) and p-Cl-phenylisocyano dichloride (VIIe) as per the above mentioned method.

RESULT AND DISCUSSION

Elemental, IR and PMR spectral analysis of all the synthesized compounds is given below:

1-phenyl-3-[4-(2-ethylimino-4-allylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones (VIIIba)

lemon yellow solid, $C_{23}H_{22}N_4OS_2$, Yield-88%, M.P.-162^{0}C Composition-found(calculated) C-62.54(63.57), H-6.13(5.10), N-11.93(12.89) and S-13.89(14.76); **FTIR (KBr) ν cm^{-1}:**3052.87(ArC-H stretching), 3226.64(N-H stretching),1669.61 (C=O stretching), 1481.12(S-C=N stretching) and 672.45(C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H, -CH=CH- at δ 2.22-3.66 ppm,multiplet of 9H of Ph at δ 2.22-3.66 ppm, singlet of 1H of –NH at δ 8.15 ppm, quintet of 1H and double doublet of 2H of allyl at δ2.06, 1.23 and 2.24 respectively, quartet of 2H and triplet of 3H of ethyl at δ1.44 and δ 1.33 respectively; Mol. Wt.: 434.

1-3-[4-(2-ethylimino-4-ethylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones (VIIIbb)

Yellow solid, $C_{22}H_{22}N_4OS_2$, Yield-74%, M.P.-159^{0}C Composition-found(calculated) C-61.21(62.53), H-6.25(5.25), N-12.21(13.26) and S-16.28(15.18); **FTIR (KBr) ν cm^{-1}:**3051.89(ArC-H stretching), 3238.62(N-H stretching), 1668.66(C=O stretching), 1478.94(S-C=N stretching) and 678.54(C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H of –CH=CH- at δ 2.11-3.16 ppm, multiplet of 9H of Ph at δ 6.77-7.58 ppm, singlet of 1H of –NH at δ 8.07 ppm, quartet of 4H and triplet of 6H of ethyl at δ1.17 and δ 1.26 respectively; Mol. Wt.: 422.

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

Pale yellow solid, $C_{24}H_{26}N_4OS_2$, Yield-67%, M.P.-163^{0}C Composition-found(calculated) C-62.98(63.97), H-4.8(5.82), N-13.37(12.43) and S-13.15(14.23); **FTIR (KBr) ν cm^{-1}:**3054.65(ArC-H stretching), 3246.73(N-H stretching), 1656.32(C=O stretching), 1481.24(S-C=N stretching) and 668.64(C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 2.49-3.37 ppm, multiplet of 9H of Ph at δ 6.95-7.64 ppm,singlet of 1H

of –NH at δ 8.01 ppm, Singlet of 9H at δ 1.33 ppm pentate of 1H, quartet of 2H and triplet of 3H of ethyl at δ1.21 and δ 1.29 respectively; Mol. Wt.:450.

1-phenyl-3-[4-(2-ethylimino-4-phenyl imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones (VIIIbd)

Yellow solid, $C_{26}H_{22}N_4OS_2$, Yield-69%, M.P.-159^{0}C Composition-found(calculated) C-65.34(66.36), H-5.74(4.71), N-10.96(11.91) and S-12.60(13.63); **FTIR (KBr) ν cm^{-1}:**3058.89(ArC-H stretching), 3282.62(N-H stretching), 1660.66(C=O stretching), 1488.94(S-C=N stretching) and 688.54(C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H of –CH=CH- at δ 2.57-3.53 ppm, multiplet of 14H of Ph at δ 7.01-7.85 ppm, quartet of 2H and triplet of 3H of ethyl at δ1.22 and δ 1.20 respectively; Mol. Wt.: 470.

1-phenyl-3-[4-(2-ethylimino-4-p-Cl-phenylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones (VIIIbe)

Yellow solid, $C_{26}H_{21}N_4OS_2Cl$, Yield-79%, M.P.- 167^{0}C Composition-found(calculated) C-60.81(61.83), H-3.17(4.19), N-12.12(11.09), S-11.77(12.70) and Cl-8.11(7.02); **FTIR (KBr) ν cm^{-1}:** 3061.52(ArC-H stretching), 3295.95(N-H stretching), 1669.50(C=O stretching), 1466.60(S-C=N stretching) and 692.86(C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 2.60-3.58 ppm, multiplet of 9H of Ph at δ 6.99-7.95 ppm, multiplet of 4H, Ph at δ 6.64-7.32 ppm, quartet of 2H and triplet of 3H of ethyl at δ1.19 and δ 1.39 respectively and singlet of 1H of –NH at δ 8.28 ppm; Mol. Wt.: 505.5.

CONCLUSION

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

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