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A Novel 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 **(VIIIda-de)** had been synthesized by the interaction of 1-phenyl-3-[4-(5-phenyl-2,4-dithiobiureto) phenyl]-Prop-2-ene-1-ones **(Vd)** with various iso cyano dichlorides**(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 separated out, this on basification with ammonium hydroxide gave the product. The structures of all synthesized compounds were justified on the basis of chemical characteristics, elemental analysis, and spectral studies.

INTRODUCTION

The literature survey reveals that the heterocyclic compounds having 1,3,5-thiadiazine nucleus enhanced its medicinal, agricultural, pharmaceutical and industrial activities of the drugs and medicines. Fungicidal[1-3] and insecticidal[4] properties showed by 1,3,5-thiadiazine nucleus. Hence nowadays the drugs containing thiadiazine nucleus are extensively used in medical, biochemical and biotechnological faculties. 1,3,5-thiadiazine nucleus also found anti-HIV[229] property. Some researchers[5-7] had been briefly investigated important reactions of substituted iso cyano dichlorides found 1,3,5-dithiazino or 1,3,5-thiadiazino molecule as a parent nucleus enhance pharmaceutical, agricultural and industrial activities of that drug[8-11].

It is quite intriguing to investigate 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 **(VIIIda-de)** had been synthesized by the interaction of 1-phenyl-3-[4-(5-phenyl-2,4-dithiobiureto) phenyl]-Prop-2-ene-1-ones **(Vd)** with various iso cyano dichlorides**(VIIa-e)** in acetone medium.

MATERIALS & METHODS

Materials

All the chemicals used in the present research were MERCKS (India Made). Starting compounds (Ia-e) were synthesized by literature method[12].

Method

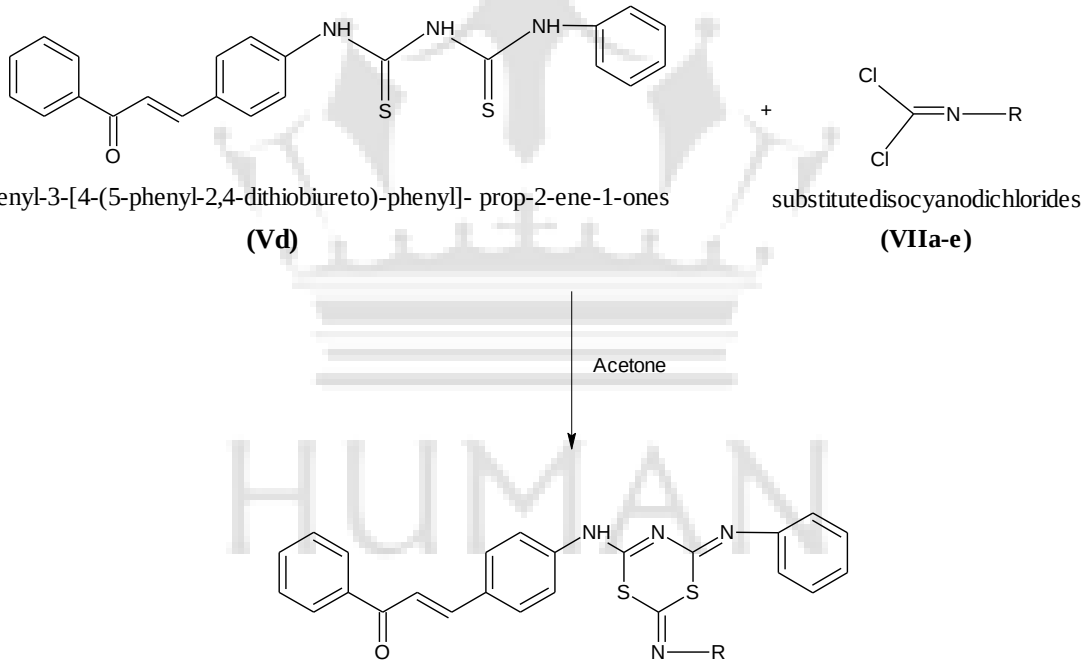
Method adopted for the synthesis of all the compounds in the present investigation was conventional refluxing under water bath to attain constant temperature. Melting points of all the synthesized compounds estimated using paraffin oil and uncorrected. The carbon, hydrogen, and nitrogen analysis was carried out on the 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 **(VIIIda-de)** had been synthesized by the interaction of 1-phenyl-3-[4-(5-phenyl-2,4-dithiobiureto) phenyl]-Prop-2-ene-1-ones **(Vd)** with various iso cyano dichlorides **(VIIa-e)** in acetone medium. The reaction mixture was refluxed 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 obtain1-phenyl-3-[4-(2-substitued imino-4-substituted imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIda-de)**

The tentative reaction is given below**,**



1-phenyl-3-[4-(5-phenyl-2,4-dithiobiureto)-phenyl]- prop-2-ene-1-ones **(Vd)**

substitutedisocyanodichlorides **(VIIa-e)**

1-phenyl-3-[4-(2-phenyl imino-4-substituted imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIda-de)**

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

Similarly, 1-phenyl-3-[4-(2-phenylimino-4-allylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIda),** 1-phenyl-3-[4-(2-phenylimino-4-ethylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIdb),** 1-phenyl-3-[4-(2-phenylimino-4-t-butylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIdc)**, 1-phenyl-3-[4-(2-phenylimino -

4-phenyl imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIdd)** and 1-phenyl-3-[4-(2-phenylimino-4-p-Cl-phenylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(VIIIde)** were synthesized by the interaction of 1-phenyl-3-[4-(5-phenyl-2,4-dithiobiureto)-phenyl]-prop-2-ene-1-one**(Ve)** with allylisocyanodichloride (VIIa), ethylisocyanodichloride (VIIb), t-butylisocyanodichloride (VIIc), phenylisocyanodichloride (VIId) and p-Cl-phenylisocyano dichloride (VIIe) as per above mentioned method.

RESULTS & DISCUSION

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

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

Lemon yellow solid, $C_{27}H_{22}N_4OS_2$, Yield-77%, M.P.-171^{0}C Composition-found(calculated) C-66.16 (67.19), H-5.58 (4.59), N-10.68 (11.61) and S-12.25 (13.29); **FTIR (KBr) ν cm^{-1}:**3046.89 (Ar-CH stretching), 3351.26 (N-H stretching),1651.66 (C=O stretching), 1567.26 (S-C=N stretching) and 691.38 (C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H, -CH=CH- at δ 2.72-3.86 ppm, multiplet of 9H of Ph at δ 6.87-8.04 ppm, multiplet of 5H, Ph at δ 6.69-7.82 ppm, singlet of 1H of –NH at δ 8.21 ppm, quintet of 1H, doublet 2H and doublet of 2H of allyl at δ2.28, 1.36 and 2.33 respectively; Mol. Wt.: 482.

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

Lemon yellow solid, $C_{26}H_{22}N_4OS_2$, Yield-81%, M.P.-167^{0}C Composition-found (calculated) C-65.34 (66.36), H-5.72 (4.71), N-10.90 (11.91) and S-12.62 (13.63); **FTIR (KBr) ν cm^{-1}:**3059.21 (Ar-CH stretching), 3347.46 (N-H stretching), 1656.27 (C=O stretching), 1573.46 (S-C=N stretching) and 692.78 (C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H of –CH=CH- at δ 2.61-3.36 ppm, multiplet of 9H of Ph at δ 6.87-7.98 ppm, singlet of 1H of –NH at δ 8.38 ppm, multiplet of 5H, Ph at δ 6.57-7.62 ppm, quartet of 2H and triplet of 3H of ethyl at δ1.51 and δ 1.31 respectively; Mol. Wt.: 470.

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

Pale solid, $C_{28}H_{26}N_4OS_2$, Yield-78%, M.P.-168^{0}C Composition-found(calculated) C-66.21 (67.44), H-5.90 (6.96), N-10.03 (11.24) and S-11.32 (12.86); **FTIR (KBr) ν cm^{-1}:**3064.24 (Ar-CH stretching), 3357.34 (N-H stretching), 1657.56 (C=O stretching), 1584.24 (S-C=N stretching) and 687.16 (C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 2.48-3.66 ppm, multiplet of 9H of Ph at δ 6.55-8.11ppm, singlet of 1H of –NH at δ 8.44 ppm, multiplet of 5H, Ph at δ 6.87-8.05 ppm, singlet of 9H, CH_3 at δ 1.16 ppm; Mol. Wt.:498.

1-phenyl-3-[4-(2-phenylimino-4-phenylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones (VIIIdd)

Lemon yellow solid, $C_{30}H_{22}N_4OS_2$, Yield-85%, M.P.-170^{0}C Composition-found(calculated) C-68.43(69.47), H-5.30 (4.28), N-9.79 (10.80) and S-11.40 (12.36); **FTIR (KBr) ν cm^{-1}:**3035.45 (Ar-CH stretching), 3363.62 (NH stretching), 1664.45 (C=O stretching), 1591.16 (S-C=N stretching) and 692.40 (C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H of –CH=CH- at δ 3.42-3.96 ppm, multiplets of 14H of Ph at δ 7.00-7.96 ppm, multiplet of 5H, Ph at δ 6.79-7.00 ppm, singlet of 1H of NH at δ 8.12 ppm; Mol. Wt.: 518.

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

Yellow solid, $C_{30}H_{21}N_4OS_2Cl$, Yield-74%, M.P.- 179^{0}C Composition-found(calculated) C-64.12(65.15), H-4.80(3.83), N-9.14(10.13), S-10.54(11.59) and Cl-7.45(6.41); **FTIR (KBr) ν cm^{-1}:**3069.46 (Ar-CH stretching), 3344.25 (NH stretching), 1642.04(C=O stretching), 1588.21 (S-C=N stretching) and 716.65 (C-S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 2.52-3.86 ppm, multiplet of 9H of Ph at δ 6.57-7.95 ppm, multiplet of 5H, Ph at δ 6.36-7.42 ppm multiplet of 4H, Ph at δ 6.47-7.72 ppm and singlet of 1H of –NH at δ 8.27 ppm; Mol. Wt.: 553.5.

CONCLUSION

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

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