International Journal of Chemistry and Pharmaceutical Sciences

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Synthesis and Characterization of 1-Phenyl-3-[4-(2-p-Cl-phenylimino-4-substitutedimino-1, 3, 5-dithiazino) amino phenyl]-prop-2-ene-1-ones

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A B S T R A C T

Recently in this laboratory series of 1-phenyl-3-[4-(2-p-Cl-phenylimino-4-substituted imino-1,3,5-dithiazino)-amino phenyl]-prop-2-ene-1-ones (VIIIea-ee) had been synthesized by the interaction of 1-phenyl-3-[4-(5-p-Cl-phenyl-2,4-dithiobiureto) phenyl]-Prop-2-ene-1-ones (Ve) 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 structures of all synthesized compounds were justified on the basis of chemical characteristics, elemental analysis and spectral studies.

Keywords: anti-HIV drugs, Heterocyclic, 1, 3, 5-dithiazines

A R T I C L E I N F O

CONTENTS

Article History: Received 26 July 2016, **Accepted** 29 August 2016, **Available Online** 27 September 2016

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Manuscript ID: IJCPS3109

PAPER-QR CODE

Citation: Dipak T. Tayade. Synthesis and Characterization of 1-Phenyl-3-[4-(2-p-Cl-phenylimino-4-substitutedimino-1,3,5-dithiazino) amino phenyl]-prop-2-ene-1-ones. *Int. J. Chem, Pharm, Sci.*, 2016, 4(9): 483-485.

1. Introduction

The study of heterocyclic compounds in organic chemistry have enormous important to synthesize novel series of compounds. Due to utility in agriculture[1] as fungicidal[2], insecticide[3] while 1, 3, 5-dithiazines are also effective against copper corrosion[4] and used in lubricating oil[5].Somethiadiazines are used in the treatment of cancer[6]

and some are anti-HIV[7-8] drugs. 1, 3, 5-dithiazino nucleus and its derivatives possesses antiviral, antifungal, antibacterial, anti-tuberculostatic and anti-helminthic activities[9-10] was reported.

We wish to report herein a simple and rapid procedure for the synthesis of 1-phenyl-3-[4-(2-p-Cl-phenylimino-4-substitutedimino-1,3,5-dithiazino)-amino phenyl]-prop-2-ene-1-ones (VIIIea-ee) had been synthesized by the interaction of 1-phenyl-3-[4-(5-p-Cl-phenyl-2,4-dithiobiureto) phenyl]-Prop-2-ene-1-ones **(Ve)** with various isocyanodichlorides**(VIIa-e)** in acetone medium.

2. Experimental

Materials

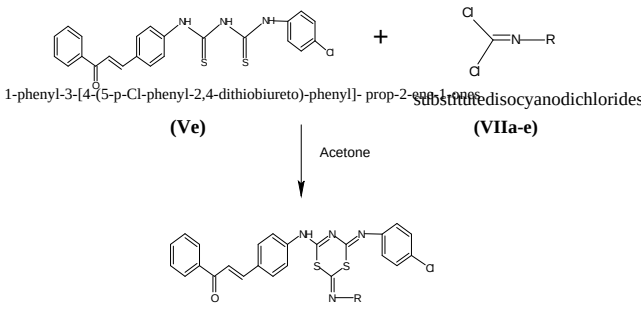
All the chemical used in the present research were MERCKS (India Made). Starting compounds (Ia-e) were synthesized by literature method[11].

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-p-Cl-phenylimino-4-substituted imino-1, 3,5-dithiazino)-amino phenyl]-prop-2-ene-1-ones (VIIIea-ee) had been synthesized by the interaction of 1-phenyl-3-[4-(5-p-Cl-phenyl-2,4-dithiobiureto) phenyl]-Prop-2-ene-1-ones (Ve) with various isocyanodichlorides(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 obtain1-phenyl-3-[4-(2-substitued imino-4-substituted imino-1,3,5-dithiazino)-amino phenyl]-prop-2-ene-1-ones (VIIIea-ee) The tentative reaction is given below,



Similarly, 1-phenyl-3-[4-(2-p-Cl-phenylimino-4-allylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones (VIIIea) 1-phenyl-3-[4-(2-p-Cl-phenylimino-4-ethylimino-1, 3, 5-dithiazino)-amino phenyl]-prop-2-ene-1-ones (VIIIeb), 1-phenyl-3-[4-(2-p-Cl-phenylimino-4-t-butylimino-1,3,5-dithiazino)-amino phenyl]-prop-2-ene-1-ones (VIIIec), 1-phenyl-3-[4-(2-p-Cl-phenylimino-4-phenylimino-1, 3, 5-dithiazino)-amino phenyl]-prop-2-ene-1-ones (VIIIed) and 1-phenyl-3-[4-(2-p-Cl-phenylimino-4-p-Cl-phenylimino-1, 3, 5-dithiazino)-aminophenyl]-prop-2-ene-1-ones (VIIIee) were synthesized by the interaction of 1-phenyl-3-[4-(5-p-Cl-phenylimino-2,4-dithiobiureto)-phenyl]- prop-2-ene-1-one(Ve) with allylisocyano dichloride (VIIa), ethyl isocyano dichloride (VIIb), t-butylisocyanodichloride (VIIc), phenyl isocyano dichloride (VIId) and p-Cl-phenyl isocyano dichloride (VIIe). As per above mentioned method.

3. Results and Discussion

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

1-phenyl-3-[4-(2-p-Cl-phenylimino-4-allylimino-1, 3, 5-dithiazino)-amino phenyl]-prop-2-ene-1-ones (VIIIea)

Lemon yellow solid, $C_{27}H_{21}N_4OS_2Cl$, Yield-71%, M.P.-179^{0}C Composition-found(calculated) C-61.71(62.72), H-5.09(4.09), N-9.79 (10.84) , Cl-5.78 (6.86) and S-11.46(12.40);FTIR (KBr)νcm^{-1}:3058.46(ArC-H stretching), 3356.85(N-Hstretching),1668.87(C=O stretching), 1579.16 (S-C=N stretching) and.710.35(C-S stretching); ^{1}H NMR (400 MHz $CDCl_3$ δ ppm) doublet of 2H, -CH=CH- at δ 2.72-3.36ppm,multiplet of 9H of Ph at δ 6.55-8.05ppm, multiplet of 4H, Ph at δ 6.34-7.78ppm, singlet of 1H of –NH at δ 8.45ppm, quintet of 1H, doublet 2H and doublet of 2H of allyl at δ2.34, 1.31 and 2.24respectively; Mol. Wt.: 517.5.

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

Dark yellow solid, $C_{26}H_{21}N_4OS_2Cl$, Yield-78%, M.P.-169^{0}C Composition-found (calculated) C-60.80(61.83), H-5.20(4.19), N-10.08 (11.09), Cl-8.09(7.02) and S-11.76(12.70); FTIR (KBr)νcm^{-1}: 3076.52 (ArC-H stretching), 3337.28(N-H stretching), 1664.65(C=O stretching), 1577.51 (S-C=N stretching) and 725.62(C-S stretching); ^{1}H NMR (400 MHz $CDCl_3$ δ ppm)doublet of 2H of –CH=CH- at δ 2.48-2.76ppm, multiplet of 9H of Ph at δ6.72-8.06ppm, singlet of 1H of –NH at δ 8.33ppm,multiplet of 4H, Ph at δ 6.57-7.88ppm,quartet of 2H and triplet of 3H of ethyl at δ1.26 and δ 1.39 respectively; Mol. Wt.: 505.5.

1-phenyl-3-[4-(2-p-Cl-phenylimino-4-t-butylimino-1, 3, 5-dithiazino)-amino phenyl]-prop-2-ene-1-ones (VIIIec)

Pale yellow solid, $C_{28}H_{25}N_4OS_2Cl$, Yield-83%, M.P.-185^{0}C Composition-found(calculated) C-62.09(63.08), H-5.72(4.73), N-9.45(10.51), Cl-7.67(6.65) and S-11.07(12.03); FTIR (KBr) ν cm^{-1}:3069.34(ArC-H stretching), 3341.28(N-H stretching), 1649.16(C=O stretching), 1560.54(S-C=N stretching) and 722.19(C-S stretching); ^{1}H NMR (400 MHz $CDCl_3$ δ ppm) doublet of 2H of –CH=CH- at δ 2.81-3.76ppm, multiplet of 9H of Ph at δ 6.48-7.83ppm,singlet of 1H of –NH at δ

8.06ppm,multiplet of 4H, Ph at δ 6.39-7.52ppm, singlet of 9H, CH_3 at δ 1.37ppm; Mol. Wt.:533.5.

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

Yellow solid, $C_{30}H_{21}N_4OS_2Cl$, Yield-77%, M.P.-177^{0}C Composition-found (calculated) C-64.20(65.15), H-4.83 (3.83), N-9.11(10.13), Cl-7.39(6.41) and S-10.57(11.59); FTIR (KBr) ν cm^{-1}:3062.16(ArC-H stretching), 3359.68(N-H stretching), 1668.25(C=O stretching), 1575.62(S-C=N stretching) and 739.06(C-S stretching); ^{1}H NMR (400 MHz $CDCl_3$ δ ppm)doublet of 2H of –CH=CH- at δ 2.66-3.56ppm, multiplet of 9H of Ph at δ 6.69-7.94ppm, multiplet of 4H, Ph at δ 6.68-8.09ppm, multiplet of 5H, Ph at δ 6.49-7.78ppm, singlet of 1H of NH at δ 8.39ppm; Mol. Wt.: 553.5.

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

Yellow solid, $C_{30}H_{20}N_4OS_2Cl_2$, Yield-80%, M.P.- 174^{0}C Composition-found (calculated) C-60.39 (61.33), H-4.40(3.43), N-8.56(9.54), S-11.98(10.91) and Cl-11.10(12.07);FTIR (KBr)νcm^{-1}:3061.26(ArC-H stretching), 3377.41(N-H stretching), 1674.27(C=O stretching), 1573.68(S-C=N stretching) and 743.65(C-S stretching); ^{1}H NMR (400 MHz $CDCl_3$ δ ppm) doublet of 2H of –CH=CH- at δ 2.52-3.66ppm, multiplet of 9H of Ph at δ 6.55-8.01ppm,multiplet of 8H, Ph at δ 6.44-7.78ppmand singlet of 1H of –NH at δ 8.21ppm; Mol. Wt.: 634.

4. Conclusion

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

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