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Synthesis of 1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-substituted-1,3,5-triazino) aminophenyl]-prop-2-ene-1-ones

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A B S T R A C T

Recently in this laboratory a potent series of 1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-substituted-1,3,5-triazino) amino phenyl]-prop-2-ene-1-ones (IIa-e) was synthesized by the isomerization of 1-phenyl-3-[4-(2-phenylimino-4-substitutedimino-1,3,5-dithiazino) amino phenyl] prop-2-ene-1-ones (Ia-e) in 10% aqueous ethanolic sodium bicarbonate medium. Recrystallized synthesized compounds and their structure was justified on the basis of chemical characteristics, elemental analysis and spectral analysis.

Keywords: amino phenyl, piperidine, Heterocyclic, pipyrazine

A R T I C L E I N F O

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1. Introduction

Heterocyclic compounds are well known for diverse biological activities observed for different molecules containing the 1, 3, 5-triazine nucleus. Such as various pipyrazine and piperidine and known as derivatives of s-triazines, which showed good antibacterial activities[1]. Using combinatorial synthesis a large number of 1,3,5-triazines containing different substituent, for development of solid phase methodlogies[2].1,3,5-Triazine nucleus have

wide range of applications in molecular probes[3] fluorescent markers[4] organic light-emitting diodes (OLED)[5] photovoltaic cells[6] and in traditional textile and polymer fields[7]which have organic fluorescent heterocyclic chromophore. Such materials are also employed as optical brighteners and UV light-absorbing material for optical lenses and in photography[8]. Some triazino compounds also showed remarkable antimicrobial activities[10-11]. Therefore, it is quite interesting to investigate the isomerization of 1-phenyl-3-[4-(2-phenylimino-4-substitutedimino-1, 3, 5-dithiazino) amino phenyl] prop-2-ene-1-ones **(Ia-e)** on isomerizing by refluxing with 10% aqueous sodium bicarbonate solution in ethanol to isolate 1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-substituted-1,3,5-triazino) amino phenyl]-prop-2-ene-1-ones **(IIa-e)** hence this work was carried out.

2. Experimental

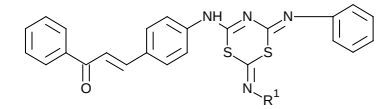
Materials: The entire chemical used in the present research was MERCKS (India Made). Starting compounds (Ia-e) were synthesized by literature method[9].

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

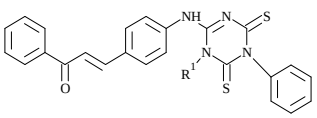
1-phenyl-3-[4-(2-phenylimino-4-substitutedimino-1, 3, 5-dithiazino) amino phenyl]prop -2-ene-1-ones **(Ia-e)** was isomerized by 10% aqeous sodium bicarbonate solution. Reactant dissolved into the solvent during heating. After distillation of excess solvent yellow crystals were obtained, which recrystallized from glacial acetic acid to isolate 1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-substituted-1, 3, 5-triazino)aminophenyl]-prop-2-ene-1-ones **(IIa-e)** hence this work was carried out. The tentative reaction is given below**,**



1-phenyl-3-[4-(2-phenylimino-4-substitutedimino-1,3,5-dithiazino)amino phenyl] prop-2-ene-1-

(Ia-e)

$NaHCO_3$ / EtOH | Isomerisation



1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-substituted-1,3,5-triazino) aminophenyl]-prop-2-ene-1-ones

(IIa-e)

Where R^1= allyl, ethyl, t-butyl, phenyl, p-Cl-phenyl

Similarly, 1-phenyl-3-[4-(2-phenylimino-4-allylimino-1 , 3,5-dithiazino) amino phenyl] prop-2-ene-1-ones**(Ia),** 1-phenyl-3-[4-(2-phenylimino-4-ethylimino-1,3,5-dithiazino)-amino phenyl]-prop-2-ene-1-ones **(Ib),** 1-phenyl-3-[4-(2-phenylimino-4-t-butylimino-1,3,5-dithiaz ino)-amino phenyl] -prop-2-ene-1-ones **(Ic)**, 1-phenyl-3-[4-(2-phenyl imino-4-phenyl imino-1,3,5-dithiazino)-amino phenyl]-prop-2-ene-1-ones **(Id)** and 1-phenyl-3-[4-(2-phenylimino-4-p-Cl-phenylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(Ie)** were interacted with 10% Sodium bicarbonate in ethanol by above mentioned method to obtain 1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-allyl-1, 3, 5-triazino) amino phenyl]-prop-2-ene-1-ones **(IIa)**, 1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-ethyl-1,3,5-triazino) amino phenyl]-prop-2-ene-1-ones **(IIb),** 1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-t-butyl-1,3,5-triazino)aminophenyl]-prop-2-ene-1-ones **(IIc)**, 1-pheny l-3-[4-(2,4-dithio-3-phenyl-5-phenyl-1,3,5-triazino)aminophenyl]-prop-2-ene-1-ones **(IId),** 1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-p-Cl-phenyl -1,3,5-triazino) amino phenyl]-prop-2-ene-1-one s**(IIe)**

3. Results and Discussion

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

1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-allyl-1,3,5-triazino) amino phenol]-prop-2-ene-1-ones (IIa)

Yellow solid, $C_{27}H_{22}N_4OS_2$, Yield-75%, M.P.-174^{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}:** 3040.64(ArC-H stretching), 3341.69(N-H stretching),1639.26(C=O stretching), 1223.66(C-N stretching) and 1132.82 (C=S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H, -CH=CH- at δ 3.23-3.37ppm,multiplet of 14H of Ph at δ 6.64-8.00ppm, singlet of 1H of –NH at δ 8.36ppm, quintet of 1H, doublet 2H and doublet of 2H of allyl at at δ2.31, 1.34 and 2.06respectively; Mol. Wt.: 482.

1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-ethyl-1,3,5 triazino) amino phenyl]-prop-2-ene-1-ones (IIb),

Yellow solid, $C_{26}H_{22}N_4OS_2$, Yield-87%, M.P.-177^{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}:** 3032.62(ArC-H stretching), 3338.39(N-H stretching), 1643.49(C=O stretching), 1229.68 (C-N stretching) and 1137.64 (C=S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)**doublet of 2H of –CH=CH- at δ 3.36-3.51ppm, multiplet of 12H of Ph at δ 6.60-7.95ppm, singlet of 1H of –NH at δ 8.18ppm,quartet of 2H and triplet of 3H of ethyl at δ1.33 and δ 1.28respectively; Mol. Wt.: 470.

1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-t-butyl-1,3,5-triazino) amino phenyl]-prop-2-ene-1-ones (IIc),

Yellow solid, $C_{28}H_{26}N_4OS_2$, Yield-76%, M.P.-179^{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}:** 3047.16(ArC-H stretching), 3346.50(N-H stretching), 1645.60(C=O stretching), 1226.44 (C-N stretching) and 1139.46 (C=S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 3.46-3.67ppm, multiplet of 12H of Ph at δ 6.75-8.10ppm,singlet of 1H of –

NH at δ 8.30ppm, singlet of 9H, CH_3 at δ 1.23ppm; Mol. Wt.:498.

1-pheny l-3-[4-(2, 4-dithio-3-phenyl-5-phenyl-1, 3, 5-triazino) amino phenyl]-prop-2-ene-1-ones (IId),

Yellow solid, $C_{30}H_{22}N_4OS_2$, Yield-89%, M.P.-160^{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}:** 3058.89 (ArC-H stretching), 3382.91 (N-H stretching), 1664.45 (C=O stretching), 1218.93 (C-N stretching) and 1180.35 (C=S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 3.62-3.74ppmmultiplets of 19H of Ph at δ 7.02-7.98ppm, singlet of 1H of NH at δ 8.54pm; Mol. Wt.: 518.

1-phenyl-3-[4-(2,4-dithio-3-phenyl-5-p-Cl-phenyl -1,3,5-triazino)amino phenyl]-prop-2-ene-1-one s (IIe)

Yellow solid, $C_{30}H_{21}N_4OS_2Cl$, Yield-71%, M.P.- 169^{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)vcm^{-1}:** 3037.86 (ArC-H stretching), 3370.16(N-H stretching), 1669.55(C=O stretching), 1223.53(C-N stretching) and 1187.16(C=S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 3.73-3.81ppm, multiplet of 18H of Ph at δ δ 6.59-8.07ppm,multiplet of 5H, Ph at δ 6.86-7.42 ppm multiplet of 4H, Ph at δ 6.97-7.72ppmand singlet of 1H of –NH at δ 8.54pm; Mol. Wt.: 553.5.

4. Conclusion

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

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