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Research Article

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

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Abstract: In this laboratory a novel series of 1-phenyl-3-[4-(2,4-dithio-3-allyl-5-substituted-1,3,5-triazino) aminophenyl]-prop-2-ene-1-ones (IIa-e) was synthesized by the isomerization of 1-phenyl-3-[4-(2-allylimino-4-substitutedimino-1,3,5-dithiazino)aminophenyl] prop-2-ene-1-ones (Ia-e)in 10% aqueous ethanolic sodium bicarbonate medium. The structure of all the synthesized compounds was justified on the basis of chemical characteristics, elemental analysis and spectral analysis.
Keywords: aminophenyl, chemical characteristics, elemental analysis, spectral analysis.

INTRODUCTION

1,3,5-Triazine nucleus have attracted a great attention among chemists due to its diverse biological activities such as medicinal, pharmaceutical, industrial and agricultural sciences. The literature survey reveals that 1,3,5-triazine nucleus containing compounds have varieties of applications and significances in medicinal, pharmaceutical, agricultural and industrial fields [1-5]. Antidiabetic [6-8], anti-tumor [9, 10], anti-inflammatory [11], anti-depresent [12], hypoglycaemic[13] properties also showed by 1,3,5-Triazine molecule.

Therefore, it is quite interesting to investigate the isomerization of 1-phenyl-3-[4-(2-allylimino-4-substitutedimino-1,3,5-dithiazino)aminophenyl] 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-allyl-5-substituted-1,3,5-triazino) aminophenyl]-prop-2-ene-1-ones **(IIa-e)** hence this work was carried out.

MATERIALS & METHOD

Materials

All the chemical used in the present research were MERCKS (India Made). Starting compounds (Ia-e) were synthesized by literature method [14].

Method

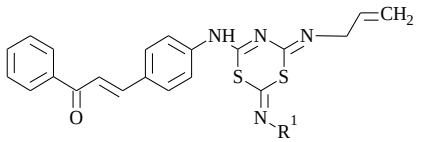
Method adopted for the synthesis of all the compounds in the present investigation was conventional refluxing under water bath to attain constant temperature. Melting points of all the synthesized compounds estimated using paraffin oil and uncorrected. The carbon, hydrogen and nitrogen analysis was carried 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.

EXPERIMENTAL

General Procedure

1-phenyl-3-[4-(2-allylimino-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-allyl-5-substituted-1,3,5-triazino) aminophenyl]-prop-2-ene-1-ones **(IIa-e)** hence this work was carried out.

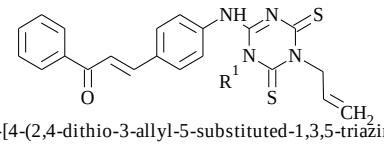
The tentative reaction is given below**,**



1-phenyl-3-[4-(2-allylimino-4-substi- tutedimino-1,3,5-dithiazino)aminophenyl] prop-2-ene-1

(Ia-e)

$NaHCO_3$ / EtOH Isomerisation



1-phenyl-3-[4-(2,4-dithio-3-allyl-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-allylimino-4-allylimino-1,3,5-dithiazino)aminophenyl]prop-2-ene-1-ones**(Ia),** 1-phenyl-3-[4-(2-allylimino-4-ethylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(Ib),** 1-phenyl-3-[4-(2-allylimino-4-t-butylimino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(Ic)**, 1-phenyl-3-[4-(2-allylimino-4-phenyl imino-1,3,5-dithiazino)-aminophenyl]-prop-2-ene-1-ones **(Id)** and 1-phenyl-3-[4-(2-allylimino-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-allyl-5-allyl-1,3,5-triazino) aminophenyl]-prop-2-ene-1-ones **(IIa)**, 1-phenyl-3-[4-(2,4-dithio-3-allyl-5-ethyl-1,3,5-triazino)aminophenyl]-prop-2-ene-1-ones **(IIb),** 1-phenyl-3-[4-(2,4-dithio-3-allyl-5-t-butyl-1,3,5-triazino)aminophenyl]-prop-2-ene-1-ones**(IIc)**, 1-phenyl-3-[4-(2,4-dithio-3-allyl-5-phenyl-1,3,5-triazino)aminophenyl]-prop-2-ene-1-ones**(IId),** 1-phenyl-3-[4-(2,4-dithio-3-allyl-5-p-Cl-phenyl-1,3,5-triazino)aminophenyl]-prop-2-ene-1-ones**(IIe)**

RESULT & DISCUSION

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

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

Yellow solid, $C_{24}H_{22}N_4OS_2$, Yield-78%, M.P.-151^{0}C Composition-found(calculated) C-63.56(64.55), H-5.96(4.97), N-11.06(12.55) and S-13.34(14.36); **FTIR (KBr) ν cm^{-1}:**3011.88(ArC-H stretching), 3382.55(N-H stretching),1677.72(C=O stretching), 1226.67(C-N stretching) and 1133.71 (C=S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H, -CH=CH- at δ 3.28-3.51ppm,multiplet of 9H of Ph at δ 6.62-8.02ppm, singlet of 1H of –NH at δ 8.43ppm, quintet of 1H and double doublet of 2H of allyl at δ2.32, 1.24and 2.13respectively, quintet of 1H and double doublet of 2H of allyl at δ2.29, 1.28 and 2.08respectively; Mol. Wt.: 486.

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

Lemon yellow solid, $C_{23}H_{22}N_4OS_2$, Yield-74%, M.P.-156^{0}C Composition-found(calculated) C-62.54(63.57), H-6.13(5.66), N-11.93(12.11) and S-13.89(13.86); **FTIR (KBr) ν cm^{-1}:**3027.86 (ArC-H stretching), 3397.96 (N-H stretching), 1674.09 (C=O stretching), 1239.23 (C-N stretching) and 1136.96 (C=S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)**doublet of 2H of –CH=CH- at δ 3.22-3.66ppm, multiplet of 9H of Ph at δ 6.58-8.04ppm, singlet of 1H of –NH at δ 8.51ppm, quintet of 1H, doublet 2H and doublet of 2H of allyl at δ2.28, 1.18and 2.03respectively, quartet of 2H and triplet of 3H of ethyl at δ1.30and δ 1.39respectively; Mol. Wt.: 464.

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

Yellow solid, $C_{25}H_{26}N_4OS_2$, Yield-71%, M.P.-142^{0}C Composition-found(calculated) C-63.90(64.90), H-6.75(5.79), N-9.70(10.72) and S-13.31(12.27); **FTIR (KBr) ν cm^{-1}:**3024.93 (ArC-H stretching), 3405.71 (N-H stretching), 1647.42 (C=O stretching), 1217.46(C-N stretching) and 1135.26 (C=S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 3.33-3.43ppm, multiplet of 9H of Ph at δ 6.69-8.06ppm,singlet of 1H of –NH at δ 8.49ppm,Singlet of 9H at δ 1.16ppm, quintet of 1H doublet 2H and doublet of 2H of allyl at δ2.32, 1.37 and 2.16respectively; Mol. Wt.:499.

1-phenyl -3-[4-(2,4-dithio-3-allyl-5-phenyl-1,3,5-triazino)aminophenyl]-prop-2-ene-1-on es (IId)

Yellow solid, $C_{27}H_{22}N_4OS_2$, Yield-82%, M.P.-147^{0}C Composition-found(calculated) C-66.15(67.19),

H-5.63(4.59), N-10.62(11.61) and S-12.24(13.29); **FTIR (KBr) ν cm^{-1}:**3029.66 (ArC-H stretching), 3391.74 (N-H stretching), 1652.36(C=O stretching), 1219.97 (C-N stretching) and 1118.64(C=S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)**doublet of 2H of –CH=CH- at δ 3.23-3.46ppm, multiplet of 14H of Ph at δ 6.73-8.02ppm, quintet of 1H, doublet 2H and doublet of 2H of allyl at δ2.16, 1.35 and 2.08 respectivelyand singlet of 1H of –NH at δ δ 8.32ppm; Mol. Wt.: 525.

1-phenyl-3-[4-(2,4-dithio-3-allyl-5-p-Cl-phenyl -1,3,5-triazino)aminophenyl]-prop-2-ene-1-ones (IIe)

Paleyellow solid, $C_{27}H_{21}N_4OS_2Cl$, Yield-78%, M.P.-139^{0}C Composition-found(calculated) C-61.70(62.72), H-5.07(4.09), N-11.87(10.84), S-11.36(12.40) and Cl-7.85(6.86); **FTIR (KBr) ν cm^{-1}:**3036.72(ArC-H stretching), 3398.63(N-H stretching), 1659.13(C=O stretching), 1226.86(C-N stretching) and 1127.56(C=S stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 3.35-3.67ppm, multiplet of 11H of Ph at δ 6.61-8.07ppm, pentate of 1H, doublet 2H and doublet of 2H of allyl at δ2.11, 1.34 and 2.02 respectivelyand singlet of 1H of –NH at δ 8.61ppm; Mol. Wt.: 566.5.

CONCLUSION

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

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