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Synthesis and Characterization of 5-Substituted Derivatives of 2, 4-Dithio-3-phenyl-6-chalcone-1,3,5-Triazines

Siddharth A. Waghmare[1*], Dipak T. Tayade[2]

[1]*Department of Chemistry, Ghulam Nabi Azad College, Arshitakli, Dist. Akola-444 404 (MS) India.*
[2]*Department if Chemistry, Govt. Vidarbha Institute of Science & Humanities, Amravati-444 604(MS) India*

A B S T R A C T

Present research work involves, Synthesis of novel series of (2E)-1-[4-(2,4-dithio-3-phenyl-5-substituted-1,3,5-triazino-6-yl)aminophenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIa-e)** by the isomerisation of series of (2*E*)-1-[4-(2-phenylimino-4-substitutedimino)amino-1,3,5-dithiazino-6-yl)aminophenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(Ia-e)** under the presence of sodium bicarbonate in aqueous ethanol. All the synthesized compounds were justified on the asis of chemical tests, elemental study and spectral characterization.

Keywords: Isomerisation, sodium bicarbonate, ethanol and spectral Characterization

A R T I C L E I N F O

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***Corresponding Author**
Siddharth A. Waghmare
Department of Chemistry,
Ghulam Nabi Azad College, Arshitakli,
Dist. Akola-444 404 (MS) India.
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1. Introduction

Heterocyclic plays a crucial role in the in the activities of the drugs or natural products. Nitrogen and sulphur are the important class of the hetero atoms in the formation of heterocycles. Many times nitrogen and sulphur in the same heterocycles reflects variety of applications. 1,3,5-Triazine is one of the enormously biologically potent heterocycle among the encyclopedia of heterocycles. Marvelous activities of the 1,3,5-Triazine are to the basic structure. The heterocycles containing s-triazines in the nucleus had successfully tested against various microorganisms and it

was found that they possess potential therapeutical value for several diseases. So these compounds possess their own importance in medical faculty, pharmaceutical, industrial and agricultural field. Different substituent's at different positions displayed variety of applications. Day by day bacteria becoming resistant to the existing drugs; Hence need of todays era is to design and synthesize new heterocycles based drugs.

Considering all the significances of the s-triazine and its different derivatives, it was planned to synthesize novel series of (2E)-1-[4-(2,4-dithio-3-phenyl-5-substituted-1,3,5-triazino-6-yl)aminophenyl]-3-(3,4-dimethoxy phenyl) prop-2-en-1-one (IIa-e) by the isomerisation of series of (2*E*)-1-[4-(2-phenyl-imino-4-substitute dimino) amino-1,3,5-dithiazino-6-yl) aminophenyl]-3-(3,4-dimethoxy phenyl) prop-2-en-1-one **(Ia-e)** on refluxing with 10% aqueous sodium bicarbonate in ethanol on water bath for half hours. The method used in the present synthesis is simple, cheaper and less time consumable.

2. Experimental

Materials: The entire chemicals used in the present research were MERCKS Chemicals (India Made). Starting compounds **(Ia-e)** were synthesized by literature method[7].

Method

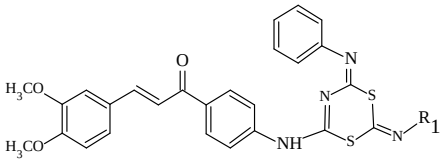
Method adopted for the synthesis of all the compounds in the present investigation was conventional refluxing under water bath to attain constant temperature. Melting points of all the synthesized compounds estimated using paraffin oil and uncorrected. The carbon, hydrogen and nitrogen analysis was carried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

(2*E*)-1-[4-(2-phenylimino-4-substitutedimino) amino-1, 3, 5-dithiazino-6-yl) amino phenyl]-3-(3,4-dimethoxy phenyl) prop-2-en-1-one **(Ia-e)** was isomerized by 10% aqueous sodium bicarbonate solution ethanol medium. During heating reactants dissolved into the solvent. After completion of the reaction, excess solvent distilled out. Yellow crystals were obtained, which on recrystalized from glacial acetic acid to obtain (2E)-1-[4-(2,4-dithio-3-phenyl-5-substituted-1,3,5-triazino6-yl) amino phenyl]-3-(3,4-dimethoxy phenyl) prop-2-en-1-one **(IIa-e)**. \

Similarly,(2*E*)-1-{4-[2-phenylimino-4-(prop-2-en-1-yl) imino-1, 3, 5-dithiazino-6yl]amino phenyl}-3-(3,4-dimethoxy phenyl)prop-2-en-1-one **(Ia),** (2*E*)-1-[4-(2-phenyl imino-4-ethylimino)-1,3,5-dithiazino-6yl] amino phenyl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one **(Ib),** (2*E*)-1-{4-[2-phenylimino-4-(2-methyl prop-2-yl)imino-1,3,5-dithia- zino-6yl] amino phenyl}-3-(3,4-dimethoxy phenyl) prop-2-en-1-one **(Ic),** (2*E*)-1-[4-(2-phenyl imino-4-phenylimino)-1,3,5-dithiazino-6yl]aminophenyl}-3-(3,4-dimethoxy phenyl) prop-2-en-1-one **(Id),** (2*E*)-1-{4-[2-phenylimino-4-(4-chlorophenyl)imino-1,3,5-dithiazino-6yl] amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(Ie)** were reacted with 10% Sodium bicarbonate in ethanol by oboe mentioned method to synthesize (2*E*)-1-{4-[2, 4-dithio-3-phenyl-5-(prop-2-en-1-yl)-1, 3, 5-triazino] amino phenyl}-3-(3,4-dimethoxy phenyl)prop-2-en-1-one **(IIa)**, (2*E*)-1-[4-(2,4-dithio-3-phenyl-5-ethyl-1,3,5-triazino)amino phenyl]-3-(3,4-dimethoxy phenyl)prop-2-en-1-one **(IIb)**, (2*E*)-1-{4-[2,4-dithio-3-phenyl-5-(2-methylprop-2-yl) -1, 3, 5-triazino]amino phenyl}-3-(3,4-dimethoxy phenyl) prop-2-en-1-one **(IIc)**, (2*E*)-1-{4-[2,4-dithio-3-phenyl-5-(4-chloro phenyl)-1,3,5-triazino]aminophenyl}-3-(3,4-dimethoxy phenyl) prop-2-en-1-one **(IIe)** respectively**.**

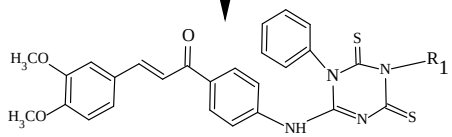
Reaction-



(2*E*)-1-[4-(2-phenylimino-4-substitutedimino-1,3,5-dithiazino)aminopheny l]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one

(Ia-e)

NaHCo3/EtOH **Isomerisation**



(2*E*)-1-[4-(2,4-Dithio-3-phenyl-5-substituted-1,3,5-triazino)aminophenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one

(IIa-e)

Where R_1= -allyl, ethyl, -t-butyl, -phenyl,-p-cl-phenyl

Reaction Scheme

3. Results and Discussion

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

(2*E*)-1-{4-[2,4-dithio-3-phenyl-5-(prop-2-en-1-yl)-1,3,5-triazino-6-yl] amino phenyl}-3-(3,4-dimethoxy phenyl) prop-2-en-1-one (IIa)

Cream yellow solid, $C_{28}H_{25}N_4O_3S_2$, Yield-72%, M.P.-173^{0}C Composition-found(calculated) C-63.74 (63.49), H-4.61 (4.76), N-10.58 (10.58) and S-11.16 (12.11); **FTIR (KBr) ν cm-1:** 3069.64 (ArC-H stretching), 3349.69 (N-H stretching), 1687.26 (C=O stretching), 1139.82 (C=S stretching), 1032.06 (C-O-C stretching) and 1212.66 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) singlet of 6H of $–OCH_3$ at δ 3.40ppm, singlet of 2H of –CH=CH- at δ 3.63-3.77ppm, multiplet of 12H of Ph at δ 6.69-8.11ppm, singlet of 1H of –NH at δ 9.82ppm and pentate of 1H, 2H and of 2H of allyl at δ2.21, 1.31 and 2.10respectively; Mol. Wt.: 529.

(2*E*)-1-[4-(2,4-dithio-3-phenyl-5-ethyl-1,3,5-triazino6-yl) aminophenyl]-3-(3,4-dimethoxy phenyl)prop-2-en-1-one (IIb): Dark yellow solid, $C_{27}H_{25}N_4O_3S_2$, Yield-80%, M.P.-

150^0C, Composition-found(calculated) C-64.45 (62.65), H-4.76 (4.87), N-10.82 (10.82) and S-11.46 (12.39); **FTIR (KBr) ν cm^{-1}:** 3033.62 (ArC-H stretching), 3361.39 (N-H stretching), 1685.49 (C=O stretching), 1139.64 (C=S stretching), 1034.13 (C-O-C stretching) and 1234.68 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) singlet of 6H of $–OCH_3$ at δ 3.40ppm, singlet of 2H of –CH=CH- at δ 3.63-3.81ppm, multiplet of 12H of Ph at δ 6.70-8.15ppm, singlet of 1H of –NH at δ 9.78ppm and quartet of 2H and triplet of 3H of ethyl at δ1.43 and δ 1.38respectively; Mol. Wt.: 517.

(2*E*)-1-{4-[2, 4-dithio-3-phenyl-5-(2-methylprop-2-yl)-1, 3,5-triazino6-yl]amino phenyl}-3-(3,4-dimethoxy phenyl) prop-2-en-1-one (IIc):

Yellow solid, $C_{30}H_{30}N_4O_3S_2$, Yield-79%, M.P.-170^0C, Composition-found(calculated) C-65.43 (64.49), H-5.75 (5.41), N-10.03 (10.03) and S-11.49 (11.48); **FTIR (KBr) ν cm^{-1}:1**3072.16 (ArC-H stretching), 3350.50 (N-H stretching), 1687.60 (C=O stretching), 1138.46 (C=S stretching), 1038.16 (C-O-C stretching) and 1235.44 (C-N stretching); ^{1}H NMR (400 MHz $CDCl_3$ δ ppm) singlet of 6H of $–OCH_3$ at δ 3.38ppm, singlet of 2H of –CH=CH- at δ 3.66-3.87ppm, multiplet of 12H of Ph at δ 6.65-8.11ppm, singlet of 1H of –NH at δ 9.80ppm and singlet of 9H, CH_3 at δ 1.29ppm; Mol. Wt.: 558.

(2*E*)-1-{4-[2,4-dithio-3-phenyl-5-phenyl-1,3,5-triazino6-yl]aminophenyl}-3-(3,4-dimeth- oxy phenyl) prop-2-en-1-one (IId):

Yellow solid, $C_{30}H_{30}N_4O_3S_2$, Yield-78%, M.P.-176^0C, Composition-found(calculated) C-66.64 (64.49), H-4.19 (5.13), N-9.68 (9.68) and S-11.52 (10.88); FTIR (KBr) ν cm^{-1}: 3060.82 (ArC-H stretching), 3309.62 (N-H stretching), 1658.67 (C=O stretching), 1141.78 (C=S stretching), 1026.06 (C-O-C stretching) and 1242.07 (C-N stretching); ^{1}H NMR (400 MHz $CDCl_3$ δ ppm) singlet of 6H of $–OCH_3$ at δ3.37-3.9ppm, singlet of 2H of –CH=CH- at δ3.82-3.94ppm, singlet of 1H of –NH at δ 9.84pm and multiplet of 17H of Ph at δ 6.97-8.10ppm; MASS Fragments: The fragmentations pattern obtained gave base peak at (M+) 303.02 and different fragments at 136.95, 288.09, 305.02, 325.00, 478 and 518.97.

(2*E*)-1-{4-[2,4-dithio-3-phenyl-5-(4-chlorophenyl)-1,3,5-triazino6-yl] amino phenyl}-3-(3,4-dimethoxy phenyl) prop -2-en-1-one (IIe):

Yellow solid, $C_{32}H_{25}N_4O_3S_2Cl$, Yield-80%, M.P.-176^0C, Composition-found(calculated) C-61.42 (62.68), H-4.35 (4.11), N-9.14 (9.14), S-10.91 (10.46) and Cl-6.65 (5.78); **FTIR (KBr) ν cm^{-1}:** 3044.86 (ArC-H stretching), 3354.16 (N-H stretching), 1685.55 (C=O stretching), 1138.16 (C=S stretching), 1031.46 (C-O-C stretching) and 1216.53 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** singlet of 6H of $–OCH_3$ at δ 3.38ppm, singlet of 2H of –CH=CH- at δ 3.63-3.71ppm, multiplet of 16H of Ph at δ 6.65-8.11ppm and singlet of 1H of –NH at δ 9.79ppm; Mol. Wt.: 612.5.

4. Conclusion

All the synthesized compound were analyzed, found and confirmed by their elemental study, IR spectra and PMR spectra. Similar method and procedure can e adopted for the synthesis of variety of derivatives of 1, 3, 5-triazines.

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