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Research Article

Synthesis and Characterization of (2*E*) -1- [4- (2, 4-Dithio-3-Ethylimino -5-Substitutedimino -1, 3, 5- Triazino-6-Yl) Aminophenyl] -3- (3, 4-Dimethoxyphenyl) Prop -2- En-1-One

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Abstract: Present research work comprises, isomerisation of series of (2E)-1-[4-(2-ethylimino-4-substitutedimino)-1,3,5-dithiazino-6-yl) aminophenyl]-3-(3,4-dimethoxy phenyl)prop-2-en-1-one (Ia-e) were successfully carried using sodium bicarbonate in aqueous ethanol in to (2E)-1-[4-(2,4-dithio-3-ethyl-5-substituted-1,3,5-triazino-6-yl) aminophenyl]-3-(3,4-dimethoxyphenyl) prop-2-en-1-one (IIa-e). All the synthesized compounds were justified on the basis of chemical tests, elemental study and spectral characterization.
Keywords: isomerization, sodium bicarbonate, ethanol, spectral characterization.

INTRODUCTION

Heterocycles are the basic unit of the any natural product or synthetic drugs. Activities of drugs are due to presence of heterocyclic ring. 1, 3, 5-Triazine is one of the extremely biologically credible organic heterocycle among the world of heterocycles. Tremendous activities of the 1, 3, 5-Triazine are to the structural arrangement in it. According the structure appearance, 1, 3, 5-Triazine is a supposed to be nitrogen reach heterocycles, hence alternative nitrogen in the six memer heterocycle results in the supreme biological heteroycle [1-3].

The heterocyclic compounds containing nitrogen and nitrogen and sulphur in the ring had gained immense importance in human life due to their noticeable and varieties of applications in pharmaceutical [4, 5], medicinal [6, 7], industrial [8, 9] and agricultural sciences [10]. The heterocycles containing s-triazines in the nucleus had en successfully tested against various microes and it was found that they possess potential therapeutical value [11] for several diseases. So these compounds possess their own importance in medical faculty, pharmaceutical, industrial and agricultural field. Some triazines contain anticancer [5], antitumor [12], antidiabtic [5, 6] and anti-inflammatory [10-12] properties.

Considering all the references, it was decided to synthesize a novel series of (2E)-1-[4-(2,4-dithio-3-ethyl-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-ethylimino-4-substitutedimino)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 two hours.

MATERIALS & METHOD

Materials

The entire chemicals used in the present research were MERCKS (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 ath 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

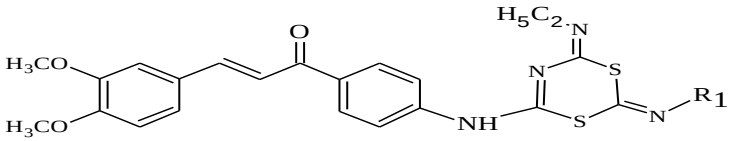
General Procedure

(2*E*)-1-[4-(2-ethylimino-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% aqeous sodium bicarbonate solution. During heating reactant dissolved into the solvent. After distillation of excess solvent yellow crystals were obtained, which recrystalized from glacial acetic acid to obtain (2E)-1-[4-(2,4-dithio-3-ethyl-5-substituted-1,3,5-triazino-6-yl)aminophenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIa-e).**

The tentative reaction is given below-

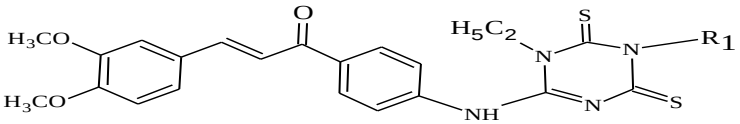
Reaction-



(2*E*)-1-[4-(2-ethylimino-4-substitutedimino-1,3,5-dithiazino-6-yl)aminophenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one

(Ia-e)

$NaHCo_3$/EtOH | **Isomerisation**



(2*E*)-1-[4-(2,4-Dithio-3-ethyl-5-substituted-1,3,5-triazino-6-yl)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

Similarly, (2*E*)-1-{4-[2-ethylimino-4-(prop-2-en-1-yl)imino-1,3,5-dithi- azino-6yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(Ia),** (2*E*)-1-{4-[2-ethylimino-4-ethylimino-1,3,5-dithiazino-6yl]aminophenyl}-3-(3,4-dimethoxy phenyl)prop-2-en-1-one **(Ib),** (2*E*)-1-{4-[2-ethylimino-4-(2-methylprop-2-yl)imino-1,3,5-dithiazino-6yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(Ic),** (2*E*)-1-{4-[2-ethylimino-4-phenylimino-1,3,5-dithiazino-6yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(Id),** (2*E*)-1-{4-[2-ethylimino-4-(4-chlorophenyl) imino-1,3,5-dithiazino-6yl]aminophenyl}-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-ethyl-5-(prop-2-en-1-yl)-1,3,5-triazino]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIa)**, (2*E*)-1-[4-(2,4-dithio-3-ethyl-5-ethyl-1,3,5-triazino)aminophenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIb),** (2*E*)-1-{4-[2,4-dithio-3-ethyl-5-(2-methylprop-2-yl)-1,3,5-triazino]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIc),** (2*E*)-1-[4-(2,4-dithio-3-ethyl-5-phenyl-1,3,5-triazino)aminophenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IId),** (2*E*)-1-{4-[2,4-dithio-3-(prop-2-en-1-yl)-5-(4-chlorophenyl)-1,3,5-triazino]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIe).**

RESULT & DISCUSION

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

(2*E*)-1-{4-[2,4-dithio-3-ethyl-5-(prop-2-en-1-yl)-1,3,5-triazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIa)

Yellow solid, $C_{25}H_{26}N_4O_3S_2$, Yield-80%, M.P.-158^{0}C Composition-found(calculated) C-61.49 (60.71), H-5.60 (5.30), N-11.33 (11.23) and S-12.68 (12.97); **FTIR (KBr) ν cm^{-1}:**3064.77 (ArC-H stretching), 3349.46 (N-H stretching), 1685.19 (C=O stretching), 1145.03 (C=S stretching), 1047.88 (C-O-C stretching) and 1236.17 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** singlet of 6H of $-OCH_3$ at δ 3.41ppm, singlet of 2H of –CH=CH- at δ 3.62-3.77ppm, multiplet of 7H of Ph at δ 6.63-8.21ppm, singlet of 1H of –NH at δ 9.82ppm, quartet of 2H and triplet of 3H of ethyl at δ1.44 and δ 1.38respectively and pentate of 1H, 2H and of 2H of allyl at δ2.18, 1.28 and 2.11respectively; Mol. Wt.: 494.

(2*E*)-1-[4-(2,4-dithio-3-ethyl-5-ethyl-1,3,5-triazino-6-yl)aminophenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIb)

Yellow solid, $C_{24}H_{26}N_4O_3S_2$, Yield-78%, M.P.-166^{0}C Composition-found(calculated) C-60.17 (59.73), H-5.64 (5.43), N-11.61 (11.61) and S-12.64 (12.29); **FTIR (KBr) ν cm^{-1}:**3045.83 (ArC-H stretching), 3371.38 (N-H stretching), 1682.11 (C=O stretching), 1140.22 (C=S stretching), 1061.07 (C-O-C stretching) and 1243.85 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** singlet of 6H of –OCH_3 at δ 3.37ppm, singlet of 2H of –CH=CH- at δ 3.67-3.76ppm, multiplet of 7H of Ph at δ 6.67-8.19ppm, singlet of 1H of –NH at δ 9.83ppm, quartet of 2H and triplet of 3H of ethyl at δ1.40 and δ 1.38respectively and quartet of 2H and triplet of 3H of ethyl at δ1.49 and δ 1.38respectively; Mol. Wt.: 482.

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

Yellow solid, $C_{26}H_{30}N_4O_3S_2$, Yield-76%, M.P.-170^{0}C Composition-found(calculated) C-61.46 (61.15), H-5.45 (5.92), N-10.97 (10.97) and S-12.46 (12.12); **FTIR (KBr) ν cm^{-1}:**30021.76 (ArC-H stretching), 3351.60 (N-H stretching), 1682.52 (C=O stretching), 1138.15 (C=S stretching), 1061.75 (C-O-C stretching) and 1215.53 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) singlet of 6H of –OCH_3 at δ 3.41ppm, singlet of 2H of –CH=CH- at δ 3.68-3.75ppm, multiplet of 7H of Ph at δ 6.59-8.13ppm, singlet of 1H of –NH at δ 9.77ppm, quartet of 2H and triplet of 3H of ethyl at δ1.43 and δ 1.39respectively and singlet of 9H, CH_3 at δ 1.35ppm; Mol. Wt.: 510.

(2*E*)-1-[4-(2,4-dithio-3-ethyl-5-phenyl-1,3,5-triazino-6-yl)aminophenyl]-3-(3,4-dimeth- oxyphenyl)prop-2-en-1-one (IId)

Yellow solid, $C_{28}H_{26}N_4O_3S_2$, Yield-78%, M.P.-155^{0}C Composition-found(calculated) C-63.61 (63.37), H-5.43 (4.94), N-10.56 (10.56) and S-11.70 (12.08); **FTIR (KBr) ν cm^{-1}:**3062.74 (ArC-H stretching), 3357.86 (N-H stretching), 1682.77 (C=O stretching), 1139.25 (C=S stretching), 1046.83 (C-O-C stretching) and 1244.08 (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 3.67-3.79ppm, multiplet of 12H of Ph at δ 6.65-8.12ppm, singlet of 1H of –NH at δ 9.83ppm and quartet of 2H and triplet of 3H of ethyl at δ1.43 and δ 1.34respectively; Mol. Wt.: 530.

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

Yellow solid, $C_{28}H_{25}N_4O_3S_2Cl$, Yield-79%, M.P.-145^{0}C Composition-found(calculated) C-59.61 (59.51), H-3.22 (4.46), N-6.27 (6.27), S-8.70 (8.49) and Cl-12.81 (11.35); **FTIR (KBr) ν cm^{-1}:**3064.43 (ArC-H stretching), 3343.26 (N-H stretching), 1657.82 (C=O stretching), 1157.83 (C=S stretching), 1057.62 (C-O-C stretching) and 1143.86 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) singlet of 6H of –OCH_3 at δ 3.41ppm, singlet of 2H of –CH=CH- at δ 3.63-3.88ppm, multiplet of 11H of Ph at δ 6.63-8.19ppm, singlet of 1H of –NH at δ 9.79ppm and quartet of 2H and triplet of 3H of ethyl at δ1.45 and δ 1.41respectively; Mol. Wt.: 564.5.

CONCLUSION

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

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