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SYNTHESIS AND CHARACTERIZATION OF (2*E*)-1-{4-[2,4-DITHIO-3-(2-METHYLPROPAN-2-YL)IMINO-5-SUBSTITUTEDIMINO-1,3,5-TRIAZINO-6-YL] AMINOPHENYL}-3-(3,4-DIMETHOXYPHENYL)PROP-2-EN-1-ONE

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ABSTRACT

Recently, in the laboratory isomerisation of series of (2*E*)-1-{4-[2-(2-methylpropan-2-yl) imino-4-substitutedimino) amino-1,3,5-dithiazino-6-yl] aminophenyl}-3-(3,4-dimethoxy phenyl)prop-2-en-1-one **(Ia-e)** were carried using sodium bicarbonate in aqueous ethanol in to (2E)-1-{4-[2,4-dithio-3-(2-methylpropan-2-yl)-5-substituted-1,3,5-triazino-**6**-yl] amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIa-e).** The structure of all the synthesized compounds was justified on the basis of chemical tests, elemental data and spectral characterization.

KEYWORDS: (2*E*)-1-{4-[2-(2-methylpropan-2-yl) imino-4-substitutedimino) amino-1,3,5-dithiazino-6-yl] aminophenyl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one **(Ia-e),** sodium bicarbonate, ethanol and spectral Characterization etc.

INTRODUCTION

Heterocycles are the fundamental constituent of the many existing drugs. Activities of the heterocycles are due to the size of the heterocycles and type of hetero atoms present their in. Nitrogen and sulphur hetero atom containing heterocycles are identified for their potential biological applications. 1,3,5-Triazine is also a potent heterocycles for its medicinal, pharmaceutical an Industrial applications. Incredible activities of the 1,3,5-Triazine are to the structural arrangement in it. According the structural study, 1,3,5-Triazine is a invented to be nitrogen rich heterocycles; hence alternative nitrogen in the six member heterocycle outcome in the ultimate biological heteroycle.

1,3,5-Triazine has huge literature of research not only in the medicinal, pharmaceutical but also in the physical chemistry for their industrial due to its physical properties. A extensive variety of newly 1,3,5-Triazines had been added the list of present heterocycles for the a range of functions.

Keeping the referenced sense of knowledge in mind, it was thought to design and synthesize the series of (2E)-1-{4-[2,4-dithio-3-(2-methylpropan-2-yl) -5-substituted-1,3,5-triazino-6-yl] aminophenyl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one **(IIa-e)** by the self isomerisation of series of (2*E*)-1-{4-[3-(2-methylpropan-2-yl) imino-4-substitutedimino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one **(Ia-e)** by conventional refluxing with 10% aqueous sodium bicarbonate solution in ethanol for two hours**.**

MATERIALS AND METHOD

Materials

All the chemical 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 synthesis was conventional refluxing under water bath to maintain constant temperature. Melting points of all the synthesized compounds estimated using paraffin oil and uncorrected. The carbon and hydrogen analysis was carried out on Carlo-Ebra-1106 analyzer. Nitrogen estimation was carried out on Colman-N-analyzer-29. IR spectra were recorded on SCIMADZU FTIR spectrometer in the range 4000-400 cm^{-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

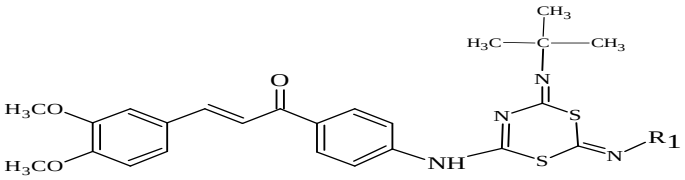
General Procedure

(2*E*)-1-{4-[2-(2-methylprop-2-yl) imino-4-substitutedimino-1,3,5-dithiazino-6yl] aminophenyl}-3-

(3,4-dimethoxyphenyl) prop-2-en-1-one **(Ia-e)** were isomerised by using 10% aqueous sodium bicarbonate solution in ethanol medium. Reaction proceeds, by dissolving the reactants in ethanol and refluxed for half hours. After completion of reaction, excess solvent was distilled out and yellow crystals obtained which on recrystalized from glacial acetic acid to obtain (2*E*)-1-{4-[2,4-dithio-3-(2-methylprop-2-yl)-5-substituted-1,3,5-triazino]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIa-e)**.

The tentative reaction is given below**,**

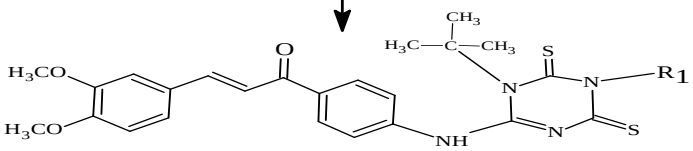
Reaction-



(2*E*)-1-{4-[2-(2-methylpropan-2-yl)imino-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-(2-methylpropan-2-yl)-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-(2-methylpropan-2-yl) imino-4-(prop-2-en-1-yl) imino-1,3,5-dithiazino-6-yl-6yl] aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(Ia),** (2*E*)-1-{4-[2-(2-methylpropan-2-yl)imino-4-ethylimino-1,3,5-dithiazino-6-yl-6yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(Ib),** (2*E*)-1-{4-[2-(2-methylpropan-2-yl)imino-4-(2-methylprop-2-yl)imino-1,3,5-dithiazino-6-yl-6yl] aminophenyl}-3-(3,4-dimeth oxyphenyl) prop-2-en-1-one **(Ic),** (2*E*)-1-{4-[2-(2-methylpropan-2-yl) imino-4-phenylimino-1,3,5-dithiazino-6-yl-6yl] aminophenyl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one **(Id),** (2*E*)-1-{4-[2-(2-methylpropan-2-yl)imino-4-(4-chlorophenyl)imino-1,3,5-dithiazino-6-yl-6yl] aminophenyl}-3-(3,4-dimethoxy phenyl)prop-2-en-1-one **(Ie)** were interacted with 10% Sodium bicarbonate in ethanol by the given method to isolate (2*E*)-1-{4-[2,4-dithio-3-(2-methylpropan-2-yl)-5-(prop-2-en-1-yl)-1,3,5-triazino] aminophenyl}-3-(3,4-dimethoxyphen yl) prop-2-en-1-one **(IIa)**, (2*E*)-1-{4-[2,4-dithio-3-(2-methylpropan-2-yl)-5-ethyl-1,3,5-triazino-6-yl] aminophenyl}-3-(3,4-dimethoxy phenyl) prop-2-en-1-one **(IIb)**, (2*E*)-1-{4-[2,4-dithio-3-(2-methylpropan-2-yl)-5-(2-methylprop-2-yl)-1,3,5-triazino-6-yl] aminophenyl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one **(IIc)**, (2*E*)-1-{4-[2,4-dithio-3-(2-methylpropan-2-yl)-5-phenyl-1,3,5-triazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one**(IId),** (2*E*)-1-{4-[2,4-dithio-3-(2-methylpropan-2-yl)-5-(4-chlorophenyl)-1,3,5-triazino-6-yl] amino phenyl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one **(IIe)** respectively.

RESULT AND DISCUSION

Spectral analysis of the all the synthesized compound using elemental analysis, IR Spectra and PMR spectra is given below,

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

Yellow solid, $C_{27}H_{30}N_4O_3S_2$, Yield-82%, M.P.-164^{0}C Composition-found(calculated) C-63.74 (62.04), H-5.61 (5.79), N-10.72 (10.72) and S-12.5 (12.27); **FTIR (KBr) ν cm^{-1}**-3066.15 (ArC-H stretching), 3355.16 (N-H stretching), 1679.49 (C=O stretching), 1139.64 (C=S stretching), 1065.42 (C-O-C stretching) and 1217.89 (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 3.66-3.88ppm, multiplet of 7H of Ph at δ 6.67-8.14ppm, singlet of 1H of –NH at δ 9.80ppm, singlet of 9H, CH_3 at δ 1.37ppm and pentate of 1H, 2H and of 2H of allyl at δ2.22, 1.31 and 2.10respectively; Mol. Wt.: 522.

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

Yellow solid, $C_{26}H_{30}N_4O_3S_2$, Yield-88%, M.P.-156^{0}C Composition-found(calculated) C-60.54 (61.15), H-6.34 (5.92), N-10.97 (10.97) and S-12.78 (12.56); **FTIR (KBr) ν cm^{-1}**-3067.95 (ArC-H stretching), 3323.74 (N-H stretching), 1683.86 (C=O stretching), 1142.35 (C=S stretching), 1040.62 (C-O-C stretching) and 1265.13 (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 3.65-3.77ppm, multiplet of 7H of Ph at δ 6.65-8.11ppm, singlet of 1H of –NH at δ 9.79ppm, singlet of 9H, CH_3 at δ 1.29ppm and quartet of 2H and triplet of 3H of ethyl at δ1.43 and δ 1.38respectively; Mol. Wt.: 510.

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

Yellow solid, $C_{25}H_{25}N_5O_3S_2$, Yield-88%, M.P.-171^{0}C Composition-found(calculated) C-62.14 (62.43), H-6.55 (6.36), N-10.4 (10.40) and S-10.87 (11.90); **FTIR (KBr) ν cm^{-1}**-3074.32 (ArC-H stretching), 3369.41 (N-H stretching), 1687.60 (C=O stretching), 1141.78 (C=S stretching), 1067.78 (C-O-C stretching) and 1211.21 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** singlet of 6H of –OCH_3 at δ 3.39ppm, singlet of 2H of –CH=CH- at δ 3.61-87ppm, multiplet of 7H of Ph at δ 6.68-8.10ppm, singlet of 1H of –NH at δ 9.81ppm and singlet of 18H of –$(CH_3)_3$ at δ 2.64ppm respectively; **MASS Spectra**.: shows base peak at (M+) 288.14 and different fragments at 73.99, 94.02, 135.99, 274.26, 303.07, 3025.05, 367.00, 437.18 and 479.07.

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

Yellow solid, $C_{30}H_{30}N_4O_3S_2$, Yield-76%, M.P.-168^{0}C Composition-found(calculated) C-64.61 (64.49), H-5.18 (5.41), N-10.03 (10.03) and S-12.49 (11.48); **FTIR (KBr) ν cm^{-1}**-3069.58 (ArC-H stretching), 3357.67 (N-H stretching), 1688.87 (C=O stretching), 1139.81 (C=S stretching), 1069.99 (C-O-C stretching) and 1227.88 (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 3.65-3.88ppm, multiplet of 12H of Ph at δ 6.69-8.14ppm, singlet of 1H of –NH at δ 9.83ppm and singlet of 9H, CH_3 at δ 1.41ppm; Mol. Wt.: 558.

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

Yellow solid, $C_{32}H_{29}N_4O_3S_2Cl$, Yield-78%, M.P.-158^{0}C Composition-found(calculated) C-59.95 (60.75), H-5.75 (4.93), N-9.75 (9.45), S-10.23 (10.45) and Cl-6.66 (5.98); **FTIR (KBr) ν cm^{-1}**-3076.43 (ArC-H stretching), 3359.11 (N-H stretching), 1689.28 (C=O stretching), 1156.82 (C=S stretching), 1029.55 (C-O-C stretching) and 1228.39 (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 3.65-3.87ppm, multiplet of 11H of Ph at δ 6.68-8.14ppm, singlet of 1H of –NH at δ 9.82ppm and singlet of 9H, CH_3 at δ 1.37ppm; Mol. Wt.: 616.5.

CONCLUSION

All the 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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