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SYNTHESIS AND SPECTRAL CHARACTERIZATION OF CHALCONE DERIVATIVES OF 1,3,5-DITHAZINES

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ABSTRACT

*Novel series of (2E)-1-{4-[2-(2-methylpropan-2-y)-4-substitutedimino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIIa-e)** had been synthesized by refluxing (2E)-1-[4-(5-(2-methylpropan-2-yl)-2,4-dithiobiureto) phenyl]-3-(3,4-dimethoxy phenyl)prop-2-en-1-one **(I)** with alkyl/aryl isocyanodichlorides **(IIa-e)** in acetone medium for 2 hours. The structures of all the synthesized compounds were justified on the basis of chemical characteristics, elemental analysis and spectral studies.*

Keywords:(2E)-1-[4-(5-(2-methylpropan-2-yl)-2,4-dithiobiureto)phenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one, alkyl/arylisocyanodichlorides, and (2E)-1-{4-[2-(2-methylpropan-2-y)-4-substitutedimino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one etc.

INTRODUCTION

Heterocycles containing organic compounds are more interesting due to their convenience in wide range of applications [1-3]. Size of heterocycles and variety of hetero atoms present in the heterocycles encompasses variety of applications [2-6]. Nitrogen and sulphur containing five member, six member, five member fused heterocycles with aromatic ring and six member fused heterocycles [3, 9-12]. More especially the heterocycles bringing nitrogen and sulphur in a same ring are known for tremendous biological and industrial applications [9, 11]. 1,3,5-dithiazine is a one of the six member heterocycles contains two sulphur atoms and one nitrogen atom and acts as a potent drug the in medicinal, agricultural and industrial fields.

Every 1,3,5-dithiazino moiety has different applications according to the substituent attached to the basic nucleus of the 1,3,5-dithiazine [1, 4-6]. It has been also observed during literature study that, 1,3,5-dithiazino nucleus and its derivatives possesses effective properties [4-6].

Taking the ideas in consideration, it was planned to synthesize some derivatives of 1,3,5-dithiazine in laboratory. It is quite interesting to investigate one step cyclisation of (2*E*)-1-{4-[5-(2-methylpropan-2-yl)-2,4-dithio-biureto]phenyl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one **(I)** with N-substituted isocyanodichlorides **(IIa-e)** in acetone medium to isolate (2*E*)-1-{4-[2-(2-methylpropan-2-y)-4-substitutedimino-1,3,5-dithiazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(IIIa-e).**

MATERIAL AND METHOD

Materials:

All the chemicals used in this method are MERCKS (India Made). Compounds (I) is synthesized using reference method [5, 6].

Method:

Method used in the present research is conventional refluxing on water bath at stable temperature.

Experimental:

General Procedure:

The interaction of (2*E*)-1-{4-[5-(2-methylprop-2- yl)-2,4-dithiobiureto]phenyl-3-(3,4-dimethoxyphenyl)prop-2-en-1-one **(I)** with alkyl/aryl isocyanodichloride **(IIa-e)** in 1:1 molar ratio refluxed on water bath in acetone medium for 2 hours. During heating evolution of hydrochloride gas was clearly noticed. Product obtained was basified with dilute ammonium hydroxide and recrystalised from ethanol.

Similar, procedure was adopted for the synthesis of all the derivatives in the series.

The probable reaction for the formation of products is depicted below,

RESULT AND DISCUSSION

Reaction data obtained and spectral characterization of all the synthesized compounds **(IIIa-e)** are given below,

Spectral Analysis:

(2E)-1-{4-[2-(2-methylprop-2-yl)imino-4-(prop-2-en-1-yl)imino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one (IIIa):

Cream yellow solid, $C_{27}H_{30}N_4O_3S_2$, Yield-79%, M.P.-168^{0}C Composition-found(calculated) C-63.59 (62.04), H-5.8 (5.79), N-10.72 (10.72) and S-12.50 (12.27); **FTIR (KBr) ν cm**-3037.46-3019.82 (ArC-H stretching), 1577.71 (S-C=N stretching), 735.42 (C-S stretching), 1654.48 (C=O stretching), 1032.63 (C-O-C stretching) and 3326.62 (N-H stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm** singlet of 6H, OCH_3 at δ 4.42ppm, doublet of 2H, -CH=CH- at δ 3.32-3.64ppm, multiplet of 7H of Ph at δ 6.63-8.01ppm, Singlet of 1H of NH at δ 8.21ppm, singlet of 9H, CH_3 at δ 1.36ppm and pentate of 1H, doublet 2H and doublet of 2H of allyl at δ2.4, 1.31 and 2.13respectively; Mol. Wt.: 522.

(2E)-1-{4-[2-(2-methylprop-2-yl)imino-4-ethylimino-1,3,5-dithiazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIIb):

Dark yellow solid, $C_{26}H_{30}N_4O_3S_2$, Yield-78%, M.P.-156^{0}C Composition-found(calculated) C-60.78 (61.15), H-6.12 (5.92), N-10.97 (10.97) and S-13.01 (12.56); **FTIR (KBr) ν cm**-3045.42-3027.16 (ArC-H stretching), 1581.14 (S-C=N stretching), 738.16 (C-S stretching), 1659.35 (C=O stretching), 1021.46 (C-O-C stretching) and 3356.19 (N-H stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm** singlet of 6H, OCH_3 at δ 4.41ppm, doublet of 2H, -CH=CH- at δ 2.64-3.58ppm, multiplet of 7H of Ph at δ 6.70-8.05ppm, Singlet of 1H of NH at δ 8.5ppm, singlet of 9H, CH_3 at δ 1.37ppm and quartet of 2H and triplet of 3H of ethyl at δ1.39 and δ 1.27respectively; Mol. Wt.: 510.

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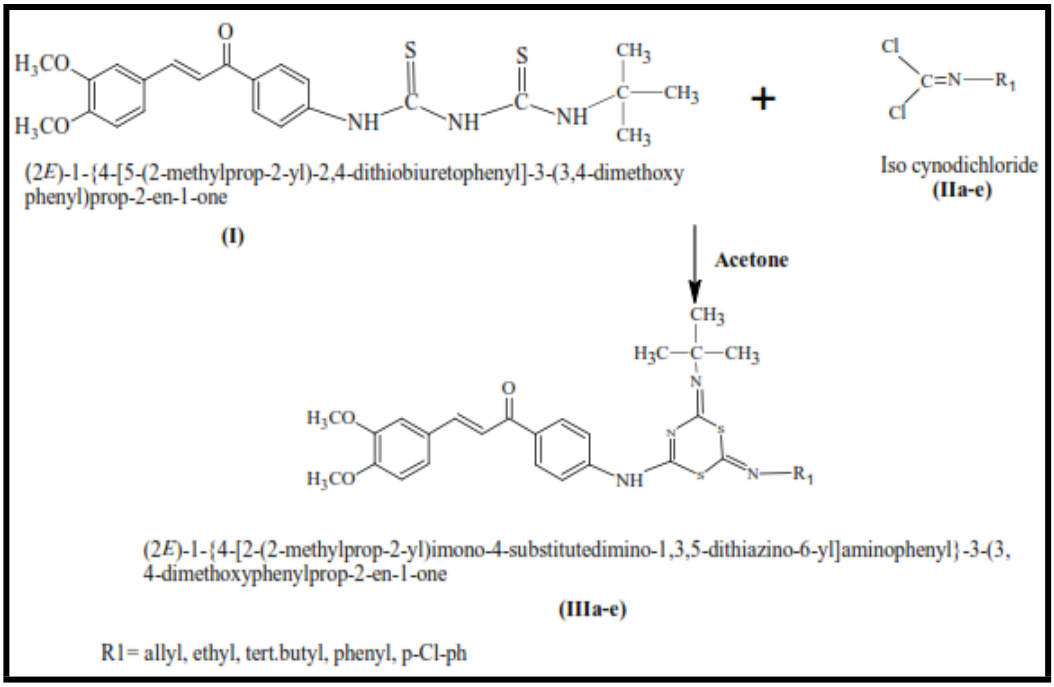

(2E)-1-{4-[2-(2-methylprop-2-yl)imino-4-(2-methylprop-2-yl)imino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIIc):

Pale yellow solid, $C_{23}H_{34}N_4O_3S_2$, Yield-80%, M.P.-154^{0}C Composition-found(calculated) C-61.20(62.43), H-6.86(6.36), N-10.73(10.40) and S-9.14(8.91); **FTIR (KBr) ν cm**$^{-1}$-3060-3030(Ar-H stretching), 3377.12(N-H starching), 1589.23 (S-C=N str), 730.97(C-S (=str), 1658.67(C=O str), 1026.06(C-O-C str); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** 18H of $-(CH_3)_3$ at 2.64ppm, 3.86ppm,singlet of 6H of $-CH_3$ at 4.43ppm, doublet of 2H of –CH=CH- at 3.46ppm-3.62ppm, multiplets of 7H of ph at 6.63ppm-8.12ppm and singlet of 1H of –NH at 8.16ppm respectively; MASS Spectra: m/z= 302.98and fragments at 284.98, 360,96, 379.01and molecular ion peak at 504..

(2E)-1-{4-[2-(2-methylprop-2-yl)imino-4-phenylimino-1,3,5-dithiazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIId):

Yellow solid, $C_{30}H_{30}N_4O_3S_2$, Yield-79%, M.P.-163^{0}C Composition-found(calculated) C-64.12(64.49), H-5.14(5.41), N-10.03(10.03) and S-12.12(11.48); **FTIR (KBr) ν cm**-3033.25-3019.46 (ArC-H stretching), 1577.19 (S-C=N stretching), 748.16 (C-S stretching), 1659.54 (C=O stretching), 1031.22 (C-O-C stretching) and 3319.58 (N-H stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm** singlet of 6H, OCH_3 at δ 4.42ppm, doublet of 2H, -CH=CH- at δ 2.62-3.59ppm, multiplet of 7H of Ph at δ 6.79-7.89ppm, Singlet of 1H of NH at δ 8.24ppm, singlet of 9H, CH_3 at δ 1.32ppm and multiplet of 5H, Ph at δ 6.55-8.21ppm; Mol. Wt.: 558.

(2E)-1-{4-[2-(2-methylprop-2-yl)imino-4-(4-chlorophenyl)imino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (IIIe):

Yellow solid, $C_{32}H_{29}N_4O_3S_2Cl$, Yield-74%, M.P.-174^{0}C Composition-found(calculated) C-59.88 (60.75), H-5.12 (4.93), N-9.75 (9.45), S-10.45 (10.45) and Cl-6.75(5.98); **FTIR (KBr) ν cm**-3029.65-3022.16 (ArC-H stretching), 1582.16 (S-C=N stretching), 737.01 (C-S stretching), 1659.16 (C=O stretching), 1029.16 (C-O-C stretching) and 3322.07 (N-H stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm** singlet of 6H, OCH_3 at δ 4.39ppm, doublet of 2H, -CH=CH- at δ 2.61-3.71ppm, multiplet of 7H of Ph at δ 6.77-8.32ppm, Singlet of 1H of NH at δ 8.37ppm, singlet of 9H, CH_3 at δ 1.31ppm and multiplet of 4H, Ph at δ 6.57-8.12ppm; Mol. Wt.: 616.5.

CONCLUSION

The reaction data obtained supports the synthesis of series **(IIIa-e)**. Spectral data also confirms the synthesized compounds **(IIIa-e)**. A variety of such chalcone analogs of 1,3,5-dithiazines can be synthesized using the same method. This method is cheaper, convenient and less time consumable.

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