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# SYNTHESIS AND SPECTRAL CHARACTERIZATION OF (2E)-1-{4-[2-(4-CHLOROPHENYL)-4-SUBSTITUTEDIMINO-1,3,5-DITHIAZINO-6-yl]AMINOPHENYL}-3-(3,4-DIMET-HOXYPHENYL)PROP-2-EN-1-ONE



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## ABSTRACT

Recently, in a laboratory novel series of (2E)-1{4-[2-(4-chlorophenyl)-4-substitutedimino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**IIIa-e**) had been synthesized in this laboratory by refluxing (2E)-1-{4-[5-(4-chlorophenyl)-2,4-dithiobiureto]phenyl}-3-(3,4-dimethoxy phenyl)prop-2-en-1-one (**I**) with substituted isocyanodichlorides (**IIa-e**) in acetone medium in 1:1 molar proportion for 2 hours. The structures of all the synthesized compounds were justified on the basis of chemical characteristics, elemental analysis and spectral studies.



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## INTRODUCTION

Heterocycles containing compounds are more interesting due to their usefulness in the synthetic. Activity of each heterocycle depends on the structural arrangement. Every heterocycle have its unique application. Supplementary and particularly the heterocycles bringing nitrogen and sulphur in the same ring are known for tremendous biological and industrial applications. 1,3,5-dithiazine is one of the six member heterocycles contains two sulphur atoms and one nitrogen atom and acts as a potent drug in medicinal, agricultural and industrial fields.

Variety of 1,3,5-dithiazines has been reported in the encyclopedia as the synthetic drug for different diseases. Every 1,3,5-dithiazino moiety has different applications according to the substituent attached to the basic nucleus of the 1,3,5-dithiazine. It has been also observed during literature study that 1,3,5-dithiazino nucleus and its derivatives possess effective properties. Some 1,3,5-dithiazines reported different derivatives of 1,3,5-dithiazino moieties as in lubricating oil.

Considering the references, it was thought to synthesize some derivatives of 1,3,5-dithiazine in laboratory by one step cyclisation of (2*E*)-1-{4-[5-(4-chlorophenyl)-2,4-dithiobiureto]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-yl)-4-substitutedimino-1,3,5-dithiazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**IIIa-e**).

## MATERIALS AND METHODS

### Materials

All the chemicals used in this method are MERCKS (Manufactured in India). Compounds (I) are synthesized using reference method<sup>5-6</sup>.

### Method

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

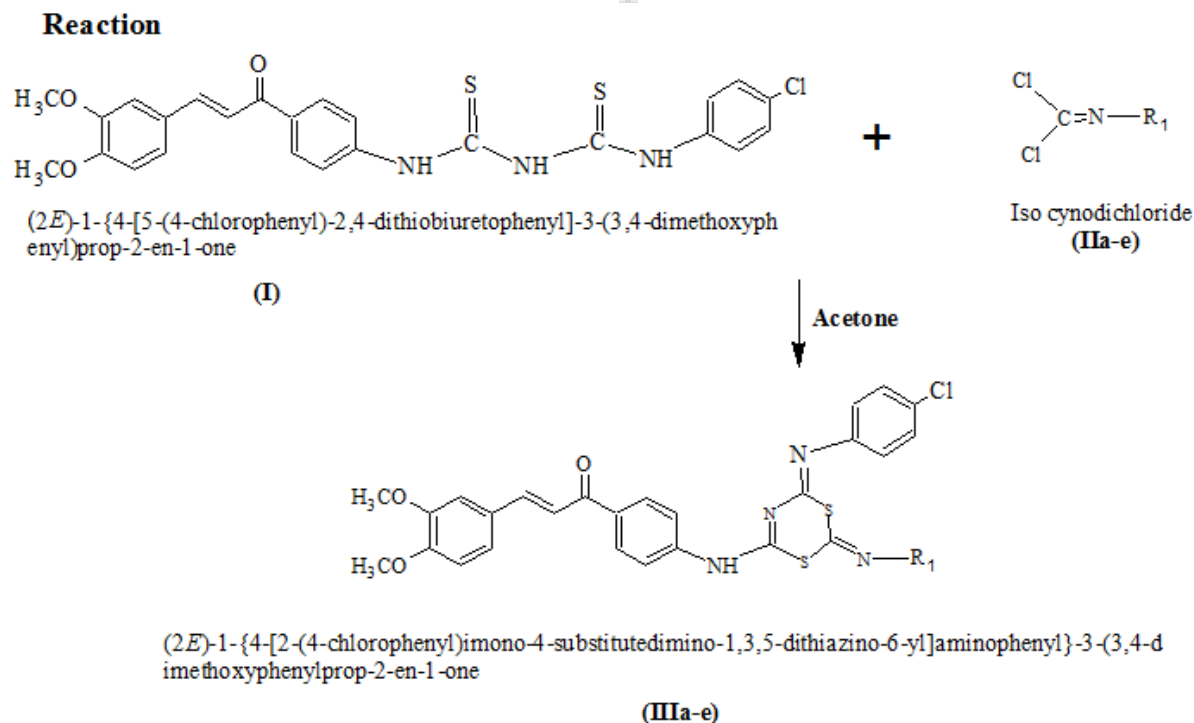
## EXPERIMENTAL

### 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 recrystallised 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,



R1 = allyl, ethyl, tert. butyl, phenyl, p-Cl-ph

Similarly, (2*E*)-1-{4-[5-(4-chlorophenyl)-2,4-dithiobiureto]phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**Ve**) were interacted with N-allyl isocyanodichloride (**VIIa**), N-ethyl isocyanodichloride (**VIIb**), N-t-butyl isocyanodichloride (**VIIc**), N-phenyl isocyanodichloride (**VIIId**), N-(4-chlorophenyl)isocyanodichloride (**VIIe**) by above mentioned method to (2*E*)-1-{4-[2-(4-chlorophenyl)imino-4-(prop-2-en-1-yl)imino-1,3,5-dithiazino-6-yl]amino phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**VIIIea**), (2*E*)-1-{4-[2-(4-

chlorophenyl)imino-4-ethylimino-1,3,5-dithiazino-6-yl]aminophenyl} -3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**VIIIeb**), (2*E*)-1-{4-[2-(4-chloro phenyl)imino-4-(2-methylprop-2-yl)imino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxy phenyl)prop-2-en-1-one (**VIIIec**), (2*E*)-1-{4-[2-(4-chloro phenyl)imino-4-phenylimino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**VIIIed**) and (2*E*)-1-{4-[2-(4-chloro phenyl)imino-4-(4-chlorophenyl)imino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (**VIIIee**) respectively.

## RESULTS AND DISCUSSION

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

### **(2*E*)-1-{4-[2-(4-chlorophenyl)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<sub>29</sub>H<sub>25</sub>N<sub>4</sub>O<sub>3</sub>S<sub>2</sub>Cl, Yield-68%, M.P.-178<sup>0</sup>C, Composition-found(calculated): C-60.12 (60.35), H-4.56 (4.37), N-9.71 (9.71), S-11.06 (11.11) and Cl-5.78 (6.14); **FTIR (KBr) v cm<sup>-1</sup>**-3024.59-3018.46 (ArC-H stretching), 1584.16 (S-C=N stretching), 730.35 (C-S stretching), 1661.87 (C=O stretching), 1028.19 (C-O-C stretching) and 3326.85 (N-H stretching); **<sup>1</sup>H NMR (400 MHz CDCl<sub>3</sub> δ** singlet of 6H, OCH<sub>3</sub> at δ 4.40ppm, doublet of 2H, -CH=CH- at δ 2.82-3.56ppm, multiplet of 7H of Ph at δ 6.65-8.12ppm, Singlet of 1H of NH at δ 8.25ppm, multiplet of 4H, Ph at δ 6.54-8.02ppm, pentate of 1H, doublet 2H and doublet of 2H of allyl at δ 2.24, 1.32 and 2.12 respectively; Mol. Wt.: 576.5.

### **(2*E*)-1-{4-[2-(4-chlorophenyl)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<sub>28</sub>H<sub>25</sub>N<sub>4</sub>O<sub>3</sub>S<sub>2</sub>Cl, Yield-71%, M.P.-168<sup>0</sup>C, Composition-found(calculated) C-59.12 (59.51), H-4.56 (4.46), N-9.91 (9.91), S-11.06 (11.35) and Cl-6.55 (6.27); **FTIR (KBr) v cm<sup>-1</sup>**-3024.68-3019.52 (ArC-H stretching), 1584.51 (S-C=N stretching), 735.62 (C-S stretching), 1674.65 (C=O stretching), 1028.29 (C-O-C stretching) and 3326.28 (N-H stretching); **<sup>1</sup>H NMR (400 MHz CDCl<sub>3</sub> δ** singlet of 6H, OCH<sub>3</sub> at δ 4.42ppm, doublet of 2H, -CH=CH- at δ 2.65-2.56ppm, multiplet of 7H of Ph at δ 6.70-8.01ppm, Singlet of 1H of NH at δ 8.23ppm, multiplet

of 4H, Ph at  $\delta$  6.67-7.98ppm and quartet of 2H and triplet of 3H of ethyl at  $\delta$  1.45 and  $\delta$  1.37 respectively Mol. Wt.: 564.5.

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

Yellow solid,  $C_{30}H_{29}N_4O_3S_2Cl$ , Yield-72%, M.P.-181<sup>0</sup>C, Composition-found(calculated) C-60.21 (60.75), H-4.72 (4.93), N-9.45 (9.45), S-10.78 (10.81) and Cl-5.98 (5.98); **FTIR (KBr)  $\nu$   $cm^{-1}$** -3054.26-301934 (ArC-H stretching), 1580.54 (S-C=N stretching), 730.19 (C-S stretching), 1646.16 (C=O stretching), 1030.45 (C-O-C stretching) and 3354.28 (N-H stretching); **<sup>1</sup>H NMR (400 MHz  $CDCl_3$   $\delta$**  singlet of 6H,  $OCH_3$  at  $\delta$  4.41ppm, doublet of 2H, -CH=CH- at  $\delta$  2.91-3.56ppm, multiplet of 7H of Ph at  $\delta$  6.68-7.93ppm, Singlet of 1H of NH at  $\delta$  8.16ppm, multiplet of 4H, Ph at  $\delta$  6.35-7.62ppm and singlet of 9H,  $CH_3$  at  $\delta$  1.34ppm; Mol. Wt.: 592.5.

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

Yellow solid,  $C_{32}H_{25}N_4O_3S_2Cl$ , Yield-70%, M.P.-167<sup>0</sup>C, Composition-found(calculated) C-62.14 (62.68), H-4.22 (4.11), N-9.14 (9.14), S-10.46 (10.46) and Cl-6.22 (5.78); **FTIR (KBr)  $\nu$   $cm^{-1}$** -3068.25-3022.16 (ArC-H stretching), 1585.62 (S-C=N stretching), 733.06 (C-S stretching), 1660.25 (C=O stretching), 1032.32 (C-O-C stretching) and 3329.68 (N-H stretching); **<sup>1</sup>H NMR (400 MHz  $CDCl_3$   $\delta$**  singlet of 6H,  $OCH_3$  at  $\delta$  4.41ppm, doublet of 2H, -CH=CH- at  $\delta$  2.86-3.76ppm, multiplet of 7H of Ph at  $\delta$  6.65-7.84ppm, Singlet of 1H of NH at  $\delta$  8.29ppm, multiplet of 4H, Ph at  $\delta$  6.78-8.02ppm and multiplet of 5H, Ph at  $\delta$  6.59-7.88ppm; Mol. Wt.: 612.5.

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

Yellow solid,  $C_{31}H_{23}N_4O_3S_2Cl_2$ , Yield-65%, M.P.-1184<sup>0</sup>C, Composition-found(calculated) C-58.9 (58.67), H-3.52 (3.65), N-8.83 (8.83), S-10.12 (10.11) and Cl-11.54 (11.17); **FTIR (KBr)  $\nu$   $cm^{-1}$** -3036-3021.26 (ArC-H stretching), 1583.68 (S-C=N stretching), 732.65 (C-S stretching), 1658.27 (C=O stretching), 1028.29 (C-O-C stretching) and 3327.41 (N-H stretching); **<sup>1</sup>H NMR (400 MHz  $CDCl_3$   $\delta$**  singlet of 6H,  $OCH_3$  at  $\delta$  4.41ppm, doublet of 2H, -CH=CH- at  $\delta$  2.62-

3.76ppm, multiplet of 7H of Ph at  $\delta$  6.65-8.11ppm, Singlet of 1H of NH at  $\delta$  8.18ppm and multiplet of 8H, Ph at  $\delta$  6.54-7.82ppm; Mol. Wt.: 634.

## 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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