International Journal of Chemistry and Pharmaceutical Sciences

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Synthesis and Spectral Characterization of Some 1,3,5-Dithazines with Chalcone Backbone

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A B S T R A C T

Recently, a series of (2E)-1-{4-[2-(prop-2-en-1-yl)-4-substitutedimino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxy phenyl) 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 in 1:1 molar proportion for 2 hours. All the synthesized compounds structures were justified on the basis of chemical tests, elemental study and spectral characterization.

Keywords: 3,4-dimethoxy phenyl, aryl isocyanodichlorides, amino phenyl

A R T I C L E I N F O

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Article History: Received 15 March 2016, **Accepted** 19 April 2016, **Available Online** 27 June 2016

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Manuscript ID: IJCPS3096

PAPER-QR CODE

Citation: Dipak T. Tayade. Synthesis and Spectral Characterization of Some 1,3,5-Dithazines with Chalcone Backbone. *Int. J. Chem, Pharm, Sci.*, 2016, 4(6): 333-335.

1. Introduction

Heterocycles are the basic unit of any natural product and synthetic drug. Activity of any drug in the universe is due to the presence of heterocycles as a basic component. Among all variety of heterocycles has been reported for the medicinal, pharmaceutical, industrial and agricultural purposes. Again the activity of the heterocycles is depend up on the size and presence of hetero atom in it. Wide range of heterocycles in a todays era contain nitrogen and sulphur as hetero atoms. 1,3,5-dithiazines are unique class of heterocycles due to their biological application. 1,3,5-

dithiazine is six member heterocycle contain three hetero atoms. Activity of 1,3,5-dithiazine is due to the presence of nitrogen and suphur heteroatom in it. Change of substituent on the 1, 3, 5-dithiazine gives variety of derivatives also various biological application.

In the present research, it has been planned to design the 1,3,5-dithiazines containing chalcones as one of the substituent on 1,3,5-dithiazine. It is quite interesting to investigate one step cyclisation of (2E)-1-[4-(5-(prop-2-en-1-yl)-2,4-dithiobiureto) phenyl]-3-(3,4-dimethoxy phenyl) prop-2-en-1-one (I) with N-substituted isocyanodi chlorides (IIa-e) in acetone medium to isolate (2E)-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).

2. Experimental

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.

General Procedure

The interaction of (2E)-1-{4-[5-(2-methylprop-2-yl)-2,4-dithiobiureto] phenyl-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (I) with alkyl/aryl isocyano-di-chloride (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 recrystalized from ethanol. Similar, procedure was adopted for the synthesis of all the derivatives in the series.

Similarly, (2E)-1-{4-[5-(prop-2-en-1-yl)-2,4-dithiobiureto] phenyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (I), were interacted with N-(prop-2-en-1-yl) isocyano-d-ichloride (VIIa), N-ethyl isocyanodichloride (VIIb) N-t-butyl isocyano-di-chloride (VIIc), N-phenyl isocyano-di-chloride (VIId), N-(4-chlorophenyl) isocyanodichloride (VIIe) by above mentioned method to isolate (2E)-1-{4-[2-(prop-2-en-1-yl)imino-4-(prop-2-en-1-yl) imino-1,3,5-dithiazino-6-yl]aminop henyl}-3-(3,4-dimethoxyphenyl)prop-2-en-1-one (VIIIaa), (2E)-1-{4-[2-(prop-2-en-1-yl)imino-4-ethylimino-1,3,5-dithiazino-6-yl]amino phen yl}-3-(3,4-dimethoxy phenyl)prop-2-en-1-one (VIIIab), (2E)-1-{4-[2-(prop-2-en-1-yl)imino-4-(2-methylprop-2-yl)imino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenyl) prop-2-en-1-one (VIIIac), (2E)-1-{4-[2-(prop-2-en-1-yl) imino-4-phenyl imino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4dimethoxy phenyl)prop-2-en-1-one (VIIIad) and (2E)-1-{4-[2-(prop-2-en-1-yl)imino-4-(4-chlorophenyl)imino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxy phenyl)prop-2-en-1-one (VIIIae) respectively.

3. Results and Discussion

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

(2*E*)-1-{4-[2-(prop-2-en-1-yl) imino-4-(prop-2-en-1-yl) imino-1, 3, 5-dithiazino-6-yl] amino phenyl}-3-(3, 4 dimethoxy phenyl) prop-2-en-1-one (IIIa)

Pale yellow solid, $C_{26}H_{26}N_4O_3S_2$, Yield-76%, M.P.-146^{0}C Composition-found(calculated) C-60.59 (61.34), H-5.22 (5.17), N-11.06 (11.06) and S-12.75 (12.66); **FTIR (KBr) ν cm^{-1}**-3071.22-3013.02 (ArC-H stretching), 1592.25 (S-C=N stretching), 745.19 (C-S stretching), 1659.55(C=O stretching), 1028.56(C-O-C stretching) and 3322.15 (N-H stretching); **^{1}H NMR (400 MHz CDCl$_3$ δ ppm)** singlet of 6H, OCH_3 at δ 4.20ppm,, doublet of 2H, -CH=CH- at δ 3.02-3.4ppm,, multiplet of 7H of Ph at δ 6.62-7.90ppm, Singlet of 1H of NH at δ 8.20ppm and pentate of 2H, doublet 4H and doublet of 4H of (prop-2-en-1-yl) at δ2.73, 2.01and 2.32 respectively; Mol. Wt.: 506.

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

Yellow solid, $C_{25}H_{26}N_4O_3S_2$, Yield-71%, M.P.-152^{0}C Composition-found(calculated) C-61.03 (60.71), H-6.57 (5.30), N-11.33 (11.33) and S-13.52 (12.97); **FTIR (KBr) ν cm^{-1}**-3004.55-3000.46 (ArC-H stretching), 1597.46 (S-C=N stretching), 745.67 (C-S stretching), 1652.16 (C=O stretching), 1032.19 (C-O-C stretching) and 3347.79 (N-H stretching); **^{1}H NMR (400 MHz CDCl$_3$ δ ppm)** singlet of 6H, OCH_3 at δ 4.25ppm, doublet of 2H, -CH=CH- at δ 2.89-3.18ppm, multiplet of 7H of Ph at δ 6.71-8.21ppm, Singlet of 1H of NH at δ 8.23ppm, pentate of 1H, doublet 2H and doublet of 2H of (prop-2-en-1-yl) at δ2.61, 1.97 and 2.31respectively and quartet of 2H and triplet of 3H of ethyl at δ1.32 and δ 1.30respectively; Mol. Wt.: 494.

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

Yellow solid, $C_{27}H_{30}N_4O_3S_2$, Yield-68%, M.P.-160^{0}C Composition-found(calculated) C-62.34 (62.04), H-5.84 (5.79), N-10.72 (10.72) and S-12.91 (12.27); **FTIR (KBr) ν cm^{-1}**-3010.46-3005.41 (ArC-H stretching), 1587.15 (S-C=N stretching), 738.98 (C-S stretching), 1661.25 (C=O stretching), 1031.44 (C-O-C stretching) and 3322.16 (N-H stretching); **^{1}H NMR (400 MHz CDCl$_3$ δ ppm)** singlet of 6H, OCH_3 at δ 4.31ppm, doublet of 2H, -CH=CH- at δ 3.01-3.8ppm, multiplet of 7H of Ph at δ 6.66-8.11ppm, Singlet of 1H of NH at δ 8.22ppm, pentate of 1H, doublet 2H and doublet of 2H of (prop-2-en-1-yl) at δ2.51, 1.52 and 2.32respectively and singlet of 9H, CH_3 at δ 1.34ppm ; Mol. Wt.: 522.

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

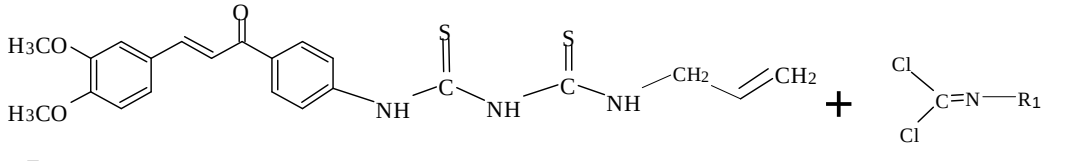
Yellow solid, $C_{29}H_{26}N_4O_3S_2$, Yield-74%, M.P.-157^{0}C Composition-found(calculated) C-64.3 (64.18), H-5.11 (4.83), N-10.32 (10.32) and S-11.45 (11.82); **FTIR (KBr) ν cm^{-1}**-3022.19-3019.54 (ArC-H stretching), 1587.13 (S-C=N stretching), 740.61 (C-S stretching), 1665.22 (C=O stretching), 1032.91 (C-O-C stretching) and 3309.12 (N-H stretching); **^{1}H NMR (400 MHz CDCl$_3$ δ ppm)** singlet of 6H, OCH_3 at δ 4.37ppm, doublet of 2H, -CH=CH- at δ 3.21-3.61ppm, multiplet of 7H of Ph at δ 6.65-7.89ppm, Singlet of 1H of NH at δ 8.23ppm, pentate of 1H, doublet

2H and doublet of 2H of (prop-2-en-1-yl) at δ2.41, 2.31 and 2.21respectively and multiplet of 5H, Ph at δ 6.65-7.92ppm; Mol. Wt.: 542.

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

Yellow solid, $C_{29}H_{25}N_4O_3S_2Cl$, Yield-81%, M.P.-160^{0}C Composition-found(calculated) C-59.23 (60.35), H-4.97 (4.37), N-9.71 (9.71), S-12.06 (11.11) and Cl-6.15(6.14); **FTIR (KBr) ν cm^{-1}**-3045.19-3015.23 (ArC-H stretching), 1573.82 (S-C=N stretching), 741.09 (C-S stretching), 1658.59 (C=O stretching), 1029.54 (C-O-C stretching) and 3320.17 (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 2.62-3.64ppm, multiplet of 7H of Ph at δ 6.68-8.32ppm, Singlet of 1H of NH at δ 8.24ppm, pentate of 1H, doublet 2H and doublet of 2H of (prop-2-en-1-yl) at δ2.43, 2.32 and 2.14respectively and multiplet of 4H, Ph at δ 6.26-7.02ppm; Mol. Wt.: 576.5.

Reaction



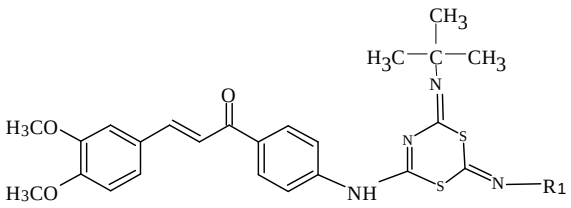
(2*E*)-1-{4-[5-(prop-2en-1-yl)-2,4-dithiobiuretophenyl]-3-(3,4-dimethoxyphenyl)prop-2-en-1-one

(I)

Iso cynodichloride

(IIa-e)

Acetone



(2*E*)-1-{4-[2-(prop-2en-1-yl)imono-4-substitutedimino-1,3,5-dithiazino-6-yl]aminophenyl}-3-(3,4-dimethoxyphenylprop-2-en-1-one

(IIIa-e)

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

4. Conclusion

The data obtained from the present reaction scheme supports the synthesis of series **(IIIa-e)**. Spectral analysis 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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