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SYNTHESIS AND CHARACTERIZATION OF 1-PHENYL-3-[4-(5-SUBSTITUTED-2,4-DITHIOBIURETO)-PHENYL]PROP-2-ENE-1-ONES

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ABSTRACT

A novel series of 1-phenyl-3-[4-(5-substituted-2,4-dithiobiureto)phenyl]- prop-2-ene-1-ones**(IIIa-e)** was synthesized by the interactions of 1-phenyl-3-(4-thiocarbamidophenyl)prop-2-ene-1-one **(I)** with different isothiocyanates**(IIa-e)** in acetone medium. The structure justification of synthesized compounds was done on the basis of elemental analysis, chemical characteristics and spectral studies.

KEYWORD: 1-phenyl-3-[4-(5-substituted-2,4-dithiobiureto)phenyl]- prop-2-ene-1-ones**(IIIa-e)**

INTRODUCTION

In pharmaceutical, agricultural[1-3] fieldhave crucial role of heteroacycles and heterocycles compounds containingthiocarbamido, thiabiurato,dithiazine,dithiobiureto and thioamido nucleus and also have their own identity. The therapeutic value of that drug enhance the potency due to presence of thiocarbamido, thiabiurato,dithiazine,dithiobiureto and thioamidonucleus containing compounds. The various 5,6 and 7 membered nitrogen, nitrogen and sulphur containing heterocycles compounds reported by Pandey[4], Berad[5], Deohate[6].1,2,4-dithiazoles, 1,3,5-dithiazines, 1,3,5-thiadiazines acts as drugs and its shows tremendous value of various medicinal, industrial, agricultural, biochemical applications fields. These types of compound possess most fabulous properties like anti-tumor and anti-cancer.[7-9] Also antitubercular, antibacterial, antifungal, antiviral and anti-inflammatory activities[10-12]showedheterocyclic compounds containing thiocarbamidonucleus.These compounds have wide range of application such as medicinal, biological, agricultural, and industrial and biochemical sciences.[13-14] As per literature survey we design potent series of 1-phenyl-3-[4-(5-substituted-2,4-dithiobiureto)-phenyl]-prop-2-ene-1-ones **(IIIa-e).**

MATERIALS & METHOD

Materials

Allthe chemical used in the present research were MERCKS (India Made). Starting compounds **(I)** were synthesized by literature method.[15]

Method

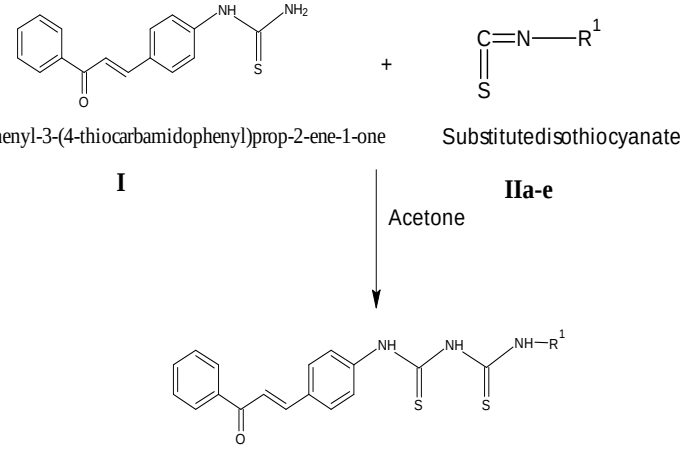
Method adopted for the synthesis of all the compounds in the present investigation was conventional refluxing under water bath to attain constant temperature. Melting points of all the synthesized compounds estimated using paraffin oil and uncorrected. The carbon, hydrogen and nitrogen analysis was carried 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

1-phenyl-3-[4-(5-substituted-2,4-dithiobiureto)-phenyl]-prop-2-ene-1-ones**(IIIa-e)** was synthesized by the interactions of 1-phenyl-3-(4-thiocarbamidophenyl)prop-2-ene-1-one **(I)**with different isothiocyanates**(IIa-e)** in acetone medium reflux for four hours. During heating reactant dissolved into the solvent. After distillation of excess solvent yellow crystals were obtained, which recrystalized from glacial acetic acid to obtain1-phenyl-3-[4-(5-substituted-2,4-dithiobiureto)-phenyl]- prop-2-ene-1-ones **(IIIa-e).**

The tentative reaction is given below**,**



1-phenyl-3-(4-thiocarbamidophenyl)prop-2-ene-1-one Substitutedisothiocyanate

I **IIa-e**

1-phenyl-3-[4-(5-substituted-2,4-dithiobiureto)-phenyl]- prop-2-ene-1

IIIa-e

R^1 = allyl, ethyl, t-But, -phenyl, p-cl-ph

Similarly, synthesis of 1-phenyl-3-[4-(5-allyl-2,4-dithiobiureto)phenyl]-prop-2-ene-1-one**(Va),**1-phenyl-3-[4-(5-ethyl-2,4-dithiobiureto)phenyl]-prop-2-ene-1-one**(Vb),**1-phenyl-3-[4-(5-t-butyl-2,4-dithiobiureto) phenyl]-prop-2-ene-1-one**(Vc)**,1-phenyl-3-[4-(5-phenyl-2,4-dithiobiureto)phenyl]-prop-2-ene-1-one**(Vd),** 1-phenyl-3-[4-(5-p-Cl-phenyl-2,4-dithiobiureto)phenyl]-prop-2-ene-1-one**(Ve)**were synthesized by the interactions of 1-phenyl-3-(4-thiocarbamidophenyl)-prop-2-ene-1-one **(I)** was carried out with allylisothiocynate**(IIa),**ethylisothiocynate**(IIb),** t-butylisothiocynate **(IIc),** phenylisothiocynate**(IId),**p-Cl-phenylisothiocynate**(IIe)** respectively by the above mentioned method.

RESULT & DISCUSION

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

1-phenyl-3-[4-(5-allyl-2,4-dithiobiureto)phenyl]prop-2-ene-1-one(IIa)

Pale yellow solid, $C_{20}H_{19}N_3OS_2$, Yield-86%, M.P.-191^{0}C Composition-found(calculated) C-61.94 (62.96), H-6.04 respectively, triplet of 3H of -CH_3 at δ 1.19ppmand quartet of 2H of –CH_2 - at δ 1.29ppm; Mol. Wt.: 359.

1-phenyl-3-[4-(5-t-butyl-2,4-dithiobiureto)phenyl]prop-2-ene-1-one(IIc)

Darkyellow solid, $C_{21}H_{23}N_3OS_2$, Yield-78%, M.P.-189^{0}C Composition-found(calculated) C-62.42 (63.44), H-6.85 (5.83), N-9.55 (10.57) and S-17.98 (16.13); **FTIR (KBr) ν cm^{-1}:**3058.89 (ArC-H stretching), 3380.98 (N-H

Darkyellow solid, $C_{23}H_{19}N_3OS_2$, Yield-83%, M.P.-198^{0}C Composition-found(calculated) C-62.84 (63.87), H-4.93 (5.85), N-8.60 (8.80) and S-13.38 (12.38); **FTIR (KBr) ν cm^{-1}:**3097.47 (ArC-H stretching), 3377.12 (N-H stretching), 1679.88 (C=O stretching), 1180.35 (C=S stretching), 1658.67 (C=O stretching), 1145.64 (C=S stretching) and 1205.53 (C-N stretching); **^{1}H NMR (400 MHz CDCl$_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 2.50-3.64ppm, multiplet of 9H of Ph at δ 6.62-7.70ppm, singlet of 3H of –NH at δ 3.47, 3.57, 8.13ppm,Singlet of 9H at δ 1.38ppm; Mol. Wt.: 386.

1-phenyl-3-[4-(5-phenyl-2,4-dithiobiureto)phenyl]prop-2-ene-1-one(IId)

stretching) and 1235.08 (C-N stretching);**^{1}H NMR (400 MHz CDCl$_3$ δ ppm)**doublet of 2H of –CH=CH- at δ 2.51, 3.74ppm, multiplet of 14H of Ph at δ 6.74-7.92ppm and singlet of 3H of –NH at δ 4.45, 5.17, 10.04ppm respectively; Mol. Wt.: 398.

(5.02), N-12.03 (11.01) and S-15.80 (16.81); **FTIR (KBr) ν cm^{-1}:**3012.21 (ArC-H stretching), 3352.29 (N-H stretching), 1667.19 (C=O stretching), 1154.26 (C=S stretching) and 1216.17 (C-N stretching); **^{1}H NMR (400 MHz CDCl$_3$ δ ppm)** doublet of 2H of –CH=CH- at δ 2.48-3.74ppm, multiplet of 9H of Ph at δ 6.64-7.81ppm, singlet of 3H of –NH at δ 3.51, 4.01, 8.40ppm respectively, quintet of 1H and double doublet of 2H of allyl at δ 2.54, 1.82 and δ 2.43 respectively; Mol. Wt.: 367.

1-phenyl-3-[4-(5-ethyl-2,4-dithiobiureto)phenyl]prop-2-ene-1-one(IIb)

Yellow solid, $C_{19}H_{19}N_3OS_2$, Yield-83%, M.P.-191^{0}C Composition-found(calculated) C-61.94 (62.96), H-6.04 (5.02), N-12.03 (11.01) and S-15.80 (16.81); **FTIR (KBr) ν cm^{-1}:**3058.89 (ArC-H stretching), 3176.54 (N-H stretching), 1660.60 (C=O stretching), 1091.63 (C=S stretching) and 1243.85 (C-N stretching); **^{1}H NMR (400 MHz CDCl$_3$ δ ppm)**doublet of 2H of –CH=CH- at δ 2.28-3.56ppm, multiplet of 9H of Ph at δ 7.07-7.91ppm, singlet of 3H of –NH at δ 3.49, 3.52, 8.13ppm

1-phenyl-3-[4-(5-p-Cl-phenyl-2,4-dithiobiureto)phenyl]prop-2-ene-1-one(IIe)
Darkyellow solid, $C_{23}H_{18}N_3OS_2Cl$, Yield-75%, M.P.-187^{0}C Composition-found(calculated) C-62.52 (63.54), H-5.31 (4.29), N-8.60 (8.80), S-13.76 (14.75) and Cl-5.07 (4.08); **FTIR (KBr) ν cm^{-1}:**3082.16 (ArC-H stretching), 3370.15 (N-H stretching), 1685.71 (C=O stretching), 1148.54 (C=S stretching) and 1163.86 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 2.42-3.75ppm, multiplet of 11H of Ph at δ 6.64-7.70ppm, 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.: 505.5.

CONCLUSION

All the synthesized compound were analyzed, found and confirmed by their elemental study, IR spectra and PMR spectra.

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