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Synthesis and Characterization of 1-Phenyl-3-(4-Thiocarbamidophenyl)- Prop-2-Ene-1-One and 1-Phenyl-3-(4-Substitutedthio-Carbamidophenyl)Prop-2-Ene-1-Ones

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ABSTRACT

Recently, in our laboratory a novel series of 1-phenyl-3-(4-thiocarbamido phenyl)prop-2-ene-1-one**(IIIa-e)** were synthesized by the interaction of 1-phenyl-3-(4-chlorophenyl)prop-2-en-1-one with different thiourea in isopropanol medium. Justification and determination of structure of synthesized compounds were done on the basis of chemical characteristics, elemental analysis and spectral studies.

INTRODUCTION

Chalcone is an exceedingly imperative compound and used as an intermediate for the synthesis of various oxygen, nitrogen and sulphur containing heterocycles and heteroacycles. It is an important group of natural products and possesses a wide range of biological activity such as anti-microbial[1], anti-cancer[2] and anti-tubercular[3]. These compounds are also known as benzal- acetophenone or benzylidene-acetophenone due to presence of two reactive benzene rings, having replaceable halo group. It was evident that various compounds are reported by Donda[4].

In this laboratory Tayade *et al*[5], Pund[6], Raghuwanshi[7] and Barange[8] synthesized new series of thiocarbamides, isothiobiurets, isodithiobiurets and successfully cyclized them into thiadiazoles, dithiazoles, dithiazines, thiadiazines, triazines, 1,2,4,6-thiatri-azepines by exploring the synthetic applications of thiocarbamido, isothiobiureto, isodithiobiureto molecules along with alkyl/aryl-isothiocynates, alkyl/aryl-isocyanodichlorides and also liquid bromine in CCl_4/$CHCl_3$ as oxidative cyclization agent and also aqueous ethanolic sodium bicarbonate medium as isomerizing medium. Their anti-microbial, anti-fungal along with agricultural applications and physiochemical parameters were also successfully investigated[9-10].

The literature survey proved that 1-phenyl-3-(4-chlorophenyl)prop-2-ene-1-one (I) benzamide derivatives are known for their anti-inflammatory, immunomodulatory[11-12], anti-tumoral[13], anti-psychotic[14] and anti-allergic activities[15].

By considering all these facts this research scheme was designed and synthesis of 1-phenyl-3-(4-thiocarbamidophenyl)prop-2-ene-1-one and 1-phenyl-3-(4-substitutedthio carbamidophenyl)prop-2-ene-1-ones were successfully carried out by the interactions of 1-phenyl-3-(4-halophenyl)prop-2-ene-1-one with various thiourea in isopropanol medium. The newly synthesized compounds may possess more practical utility and also the new thiocarbamido substituents may enhance the potency of the molecules.

MATERIALS AND METHODS

Materials

All the chemicals used in the present research were MERCKS (India Made). Starting compounds (Ia-e) were synthesized by literature method[16].

Method

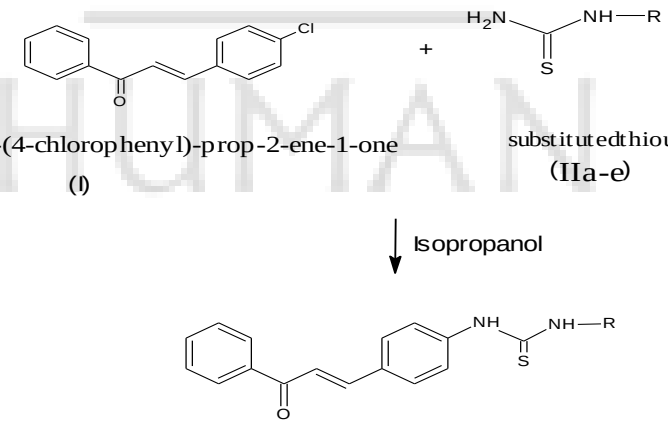
In the present investigation, conventional method was adopted such as refluxing under water bath to attain constant temperature for the synthesis of all the compounds. Using paraffin oil melting points of all the synthesized compounds estimated and uncorrected. The carbon, hydrogen and nitrogen analysis was carried out on Carlo-Ebra-1106 analyzer and Colman-N-analyzer-29 respectively. IR spectra were recorded on SHIMADZU 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

The interaction of 1-phenyl-3-(4-chlorophenyl)prop-2-en-1-one with substituted thiourea in isopropanol medium. Yellow crystals were obtained after distillation of excess solvent, which recrystallized from ethanol to obtain1-phenyl-3-(4-substitutedthio carbamidophenyl)prop-2-ene-1-ones **(IIIa-e).**

The Probable reaction is depicted below **(Scheme-I)**



1-phenyl-3-(4-chlorophenyl)-prop-2-ene-1-one (I)

substitutedthiourea (IIa-e)

1-Phenyl-3-(4-phenylthiocarbamido phenyl)-prop-2-ene-1-one (IIIa-e)

R= H, -ph, -o-CH_3-ph, -m-CH_3-ph, -p-CH_3-ph

Similarly, 1-Phenyl-3-(4-thiocarbamidophenyl)prop-2-ene-1-one **(IIIa),** 1-Phenyl-3-(4-phenylthiocarbamidophenyl)prop-2-ene-1-one **(IIIb),**1-Phenyl-3-(4-o-methylphenylthiocarbamidophenyl)prop-2-ene-1-one **(IIIc)**, 1-Phenyl-3-(4-m-methyl

phenylthiocarbamidophenyl)prop-2-ene-1-one **(IIId),** 1-Phenyl-3-(4-p-methylphenyl thiocarbamidophenyl)prop-2-ene-1-one**(IIIe),** were synthesized by the interactions of 1-phenyl-3-(4-chlorophenyl)prop-2-ene-1-one**(I)** with thiourea**(IIa),** phenylthiourea **(IIb),** o-methyl phenylthiourea**(IIc),** m-methylphenyllthiourea**(IId),** p-methylphenyl thiourea **(IIe)** respectively by the above mentioned method.

RESULTS AND DISCUSSION

All the synthesized compounds has elemental analysis, IR Spectra and PMR spectral analysis given below,

1-Phenyl-3-(4-thiocarbamidophenyl)prop-2-ene-1-one(IIIa)

Pale yellow solid, $C_{16}H_{14}N_2OS$, Yield-94%, M.P.-203^{0}C Composition-found (calculated) C-67.06 (68.06), H-5.00 (6.00), N-10.92(9.92) and S-9.95(11.36); **FTIR (KBr) ν cm^{-1}:**3031.30(ArC-H stretching), 3460.22(N-H stretching), 1667.82(C=O stretching), 1125.23(C=S stretching) and 1232.17 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 1H of –CH=CH-allyl at δ 2.62 ppm, multiplet of 9H of Ph at δ 6.143-7.20 ppm and singlet of 2H of –NH at δ 9.50, 3.58 ppm; Mol. Wt.: 282.

1-phenyl-3-(4-phenylthiocarbamido phenyl)prop-2-ene-1-one (IIIb)

Lemon yellow solid, $C_{22}H_{18}N_2OS$, Yield-86%, M.P.-219^{0}C Composition-found (calculated) C-72.70 (73.71), H-6.07 (5.06), N-7.82 (7.82) and S-9.95 (8.95); **FTIR (KBr) ν cm^{-1}:**3055.03 (ArC-H stretching), 3286.48 (N-H stretching), 1675.08 (C=O stretching), 1091.63 (C=S stretching) and 1494.63 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H of –CH=CH- at δ 2.52-3.56 ppm, multiplet of 14H of Ph at δ 6.75-7.91 ppm and singlet of 2H of –NH at δ 10.08-10.17ppm; Mol. Wt.: 342.

1-phenyl-3-(4-o-methylphenylthiocarbamidophenyl)prop-2-ene-1-one (IIIc)

Dark yellow solid, $C_{23}H_{20}N_2OS$, Yield-90%, M.P.-197^{0}C Composition-found (calculated) C-73.15 (74.16), H-4.42 (5.41), N-6.92 (7.52) and S-9.61 (8.61); **FTIR (KBr) ν cm^{-1}:**3267.19 (ArC-H stretching), 3415.70 (N-H stretching), 1658.67 (C=O stretching), 1153.35 (C=S stretching) and 1235.53 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 2.35-3.50 ppm, multiplet of 13H of Ph at δ 6.74-7.94ppm, singlet of 2H of –NH at δ 9.66-4.54 ppm respectively, singlet of 3H, CH_3 at δ 1.45 ppm; Mol. Wt.: 363.

1-Phenyl-3-(4-m-methylphenylthiocarbamidophenyl)prop-2-ene-1-one(IIId)

Yellow solid, $C_{23}H_{20}N_2OS$, Yield-88%, M.P.-192^{0}C Composition-found (calculated) C-73.16 (74.16), H-6.41 (5.41), N-7.52 (7.52) and S-7.61 (8.61); **FTIR (KBr) ν cm^{-1}:**3254.23 (ArC-H stretching), 3350.12 (N-H stretching), 1665.25 (C=O stretching), 1165.21 (C=S stretching) and 1238.08 (C-N stretching);**^{1}H NMR (400 MHz $CDCl_3$ δ ppm)** doublet of 2H of –CH=CH- at δ 2.61-3.74 ppm respectively, multiplet of 13H of Ph at δ 6.8-7.81ppm, singlet of 2H of –NH at δ 9.62-3.51 ppm respectively and singlet of 3H of CH_3 at δ 1.43ppm ; Mol. Wt.: 363.

1-Phenyl-3-(4-p-methylphenylthiocarbamidophenyl)prop-2-ene-1-one(IIIe)

Yellow solid, $C_{23}H_{20}N_2OS$, Yield-89%, M.P.-199^{0}C Composition-found (calculated) C-73.14 (74.16), H-6.46 (5.41), N-7.02 (7.52) and S-7.61 (8.61); **FTIR (KBr) ν cm^{-1}:**3257.23 (ArC-H stretching), 3352.29 (N-H stretching), 1655.26 (C=O stretching), 1117.05 (C=S stretching) and 1143.86 (C-N stretching); **^{1}H NMR (400 MHz $CDCl_3$ δ ppm**) doublet of 2H of –CH=CH- at δ 2.57-3.74 ppm, multiplet of 13H of Ph at δ 6.8-7.74 ppm, singlet of 2H of –NH at δ 9.58-4.4ppm respectively and singlet of 3H of methyl at δ1.95 ppm ; Mol. Wt.: 363.

CONCLUSION

All the synthesized compounds 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 thiocarbamides.

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