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Applied Chemistry

Elixir Appl. Chem. 62 (2013) 17737-17739

ISSN: 2229-712X

Synthesis, characterization and antimicrobial activity of some hydrazones of 2-amino-5-chloro benzophenone

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ARTICLE INFO

Article history:

Received: 31 July 2013;

Received in revised form:

25 August 2013;

Accepted: 6 September 2013;

Keywords

2–Amino –5–chlorobenzophenone, Hydrazone and Biological activity.

ABSTRACT

Hydrazone compounds derived from 2–Amino –5–chlorobenzophenone. The structures of the compounds are purified and characterized by means of TLC, melting point, IR, ^{1}H NMR spectral data. The antimicrobial activity against bacteria and fungi was studied. The result of preliminary biological tests showed that of these compounds possess biological activaties.

Introduction

Hydrazones continues an important class of biologically active drug molecules[1] which are attracted attention of medicinal chemists due to their wide range of pharmacological properties. These compounds are being synthesized as drugs by many researchers in order to combat diseases with minimal toxicity and maximal effects. These predictions has provided therapeutic pathway to develop new effective biologically active hydrazones.

Hydrazones possessing an azomethine –NH–N=C–proton constitute an important class of compounds for new drug development[2-4]. Therefore, many researchers have synthesized these compounds as target structures and evaluated their biological activities. Hydrazones and substituted hydrazones are synthesized by aldehydes and ketones treated with hydrazine, phenyl hydrazine and 2,4–dinitrophenyl hydrazine. These are imine compounds with a carbon-nitrogen double bond.

Hydrazones are most widely used organic compounds. They are used as pigments and dyes, catalysts, intermediates in organic synthesis and polymer stabilizers. Hydrazones have been shown to exhibit a broad range of biological activities[5] including antimicrobial, antihypertensive, anticonvulsant, antiinflammatory, analgesies, antitubercular, antitumor, antimaterial, antitoxoplasma, antioxidant, antiviral, and hydrazones are also used as whole transporting agents in organic layer photo conductors, as quantitative analytical reagents, especially in colorimetric and fluorimetric determination of metal ions[6-8].

Some hydrazones have also been used as herbicides, insecticides, nematocides, rodenticides and plant growth regulator[7] as well as plasticizer and stabilizers for polymers[9,10]. The metal complexes of hydrzones have potential applications as catalysts[11], luminescent probers[12] and molecular sensor[13].

In continuation of our work on these different hydrazones are report in the paper synthesis from 2–Amino –5–chlorobenzophenone by the reaction between hydrazine and substituted hydrazines.

The melting points were taken in open capillaries and are uncorrected. IR spectra were recorded in KBr on Shimadzu Spectrometer, ^{1}H NMR in DMSO D_6 on Bruker Spectrometer, Using TMS as internal standard.

Material and Methods:

All solvents, reagents and catalysts were of analytical grade and used without further purification. The melting points were determined were uncorrected. The purity of compounds was confirmed by thin layer chromatography using silica Gel glass plates as the stationary phase and with suitable mobile phase.

Preparation of substituted hydrazones

Substituted hydazones are prepared according to the known method from the condensation of 2–Amino –5–chlorobenzophenone with hydrazine[14], phenyl hydrazine and 2,4-Ditrophenyl hydrazine[15] respective by in a molar ratio 1:1.

Synthesis of 2–Amino–5–chlorobenzophenonehydrazone (Compound 1)

4.633g of 2–Amino–5–chlorobenzophenone (0.02mol) and 2.099g of hydrazine hydrochloride (0.02 mol) dissolved in 20mL of ethanol and add few drops of acetic acid were reacted under refluxing conditions. After half an hour of refluxing the reaction mixture was transferred into the 100 mL beaker. The reaction mixture was cooled and kept for overnight. A yellow product was collected by filtration to give yellow crystals.

Synthesis 2–Amino –5–chlorobenzophenonephenylhydrazone (Compound 2)

4.633g of 2–Amino –5–chlorobenzophenone (0.02mol) and 2.162g of phenylhydrazine (0.02mol) dissolved in 20mL of ethanol and added few drops of sulphuric acid were reacted under refluxing conditions. After one hour of refluxing the reaction mixture was transferred into the 100 mL beaker. The reaction mixture was cooled and yellow product was collected by filtration to give yellow crystals.

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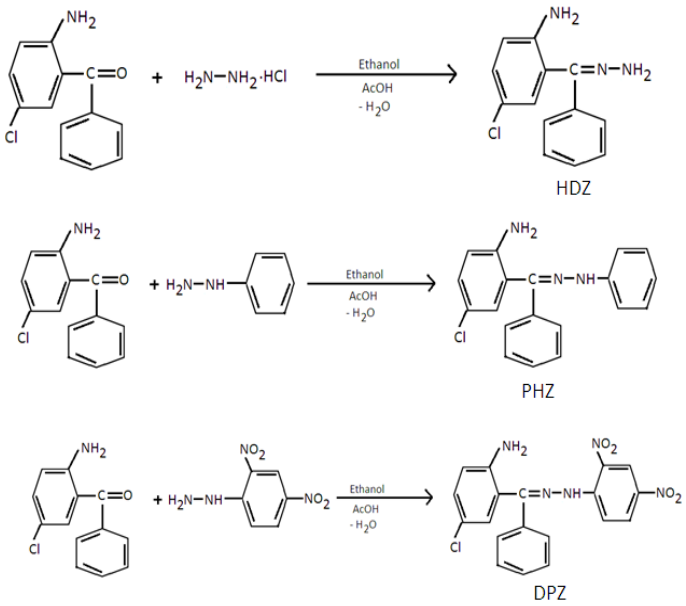
Synthesis 2–Amino–5–chlorobenzophenone–2,4–dinitrophenylhydrazone (Compound 3)

4.633g of 2–Amino–5–chlorobenzophenone (0.02mol) and 3.962g of 2,4–dinitrophenyhydrazine (0.02mol) dissolved in 20mL of ethanol and add few drops of sulphuric acid were reacted under refluxing conditions. After half an hour of refluxing the reaction mixture was transferred into the 100 mL beaker. The solid separated after cooling the reaction mixture. Orange red product was collected by filtration to give orange red crystals.

Scheme of preparation



Result & Discussion

The Synthesized compounds were characterized by physical parameter Melting point, TLC, % yield, Appearance to Confirmed the product and present in the table

Table 1.

S. No	Compound	Molecular Formula	M. Wt	M.P ^{o} (C)	% Yield	Rf value
01	Hydrazone (1)	$C_{13}H_{12}N_3Cl$	282.55	185	82	0.81
02	Phenyl hydrazone (2)	$C_{19}H_{16}N_3Cl$	368.65	205	72	0.73
03	2,4–dinitrophenyl hydazone (3)	$C_{19}H_{14}N_5O_4Cl$	311.82	225	85	0.85

Spectral Data

The structure of synthesized compound has been characterized based on IR and ^{1}H NMR spectral data.

(1) 2–Amino–5–chlorobenzophenonehydrazone

IR (KBr) cm^{-1}: 3421 (NH), 3317 (NH_2) 3059 (aromatic C-H), 1620(C=N), 1531 (aromatic C-C), 1319 (C-N), 1149 (N-N), 825 (p-substitution) and 763 (o-substitution). ^{1}H NMR (DMSO d_6) δ: 6.9 (both NH_2), 4 H, (S) 7 – 7.6 (aromatic H), 8 H (m).

(2) 2–Amino–5–chlorobenzophenonephyenyhydrazone

IR (KBr) cm^{-1}: 3421 (NH), 3317 (NH_2) 3059 (aromatic C-H), 1620(C=N), 1531(aromatic C-C), 1319 (C-N), 1149 (N-N), 825 (p-substitution) and 763 (o-substitution). ^{1}H NMR (DMSO d_6) δ: 3.3 and 4.9 (N H & NH_2), 3 H, (S) 6.6 – 7.7 (aromatic H), 13 H (m).

(3)2–Amino–5–chlorobenzophenonephyeny-2,4-dinitophenylhydrazone

IR (KBr) cm^{-1}: 3394 (NH), 3369 (NH_2) 3049 (aromatic C-H), 1616 (C=N), 1508 (aromatic C-C), 1311 (C-N), 1134 (N-N), 821 (p-substitution) and 763 (o-substitution). ^{1}H NMR (DMSO d_6) δ: 3.4 and 7.0 (N H & NH_2), 3 H, (S) 6.8 – 8.9 (aromatic H), 11 H (m).

Antimicrobial activity:

The antimicrobial activity for he given samples was carried out by Disc Diffusion method. The test microorganism of Gram positive *Staphylococcus aureus* and gram negative *Escherichila coli* and fungus *candida albicans*, *Aspergillus niger* were obtained from Easma Institute of Technology, Aravakurichy, Tamilnadu, India and maintained by periodicalsub culturing on Nutrient agar and sabourad dextrose mediam both bacteria and fungus respectively. The effect produced by the sample was compared with the effect produced by the positive control (Reference standard ciprofloxacin for bacteria and Fluconazole for fungi). The result indicated that compounds were more active against all four organisms with reference to standard. The results are shown in the table 2

Table 2: Zone of inhibition of the synthesized compounds

S. No	Organism tested	Zones of Inhibition (mm) produced by different sample concentrations (µg/ml)			Std. Antibiotics
		Compound 1	Compound 2	Compound 3	
01	*E.Coli*	14	15	15	25
02	*S.aureus*	14	14	14	25
03	*Aspergillus*	09	09	09	20
04	*Candida*	16	14	19	20

Conclusion:

The hydrazone and substituted hydrazones were synthesized and characterized and antimicrobial activity was done using various organisms. The synthesized compounds (compound 1-3) were characterized by spectral data and evaluated for their antimicrobial activity. All compounds were screened for antimicrobial (antibacterial and antifungal) activity and compound 2 and 3 which exhibiting good antibacterial activity against gram negative and mild antibacterial activity against gram positive bacteria. The compound 3 which good antifungal activity.

Acknowledgments:

The authors are thankful to Head, SAIF, IIT/M, Chennai for providing NMR spectra and Also thankful to Head, PG and Research Department of Chemistry, Jamal Mohamed college for providing IR spectra facilities for this work.

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