
Clinical and Medical Research and Studies

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Synthesis and Evaluation of Hydrazones

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Abstract

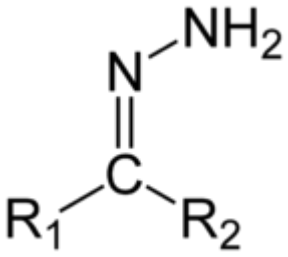
Hydrazones are a class of organic compounds with the structure R1R2C=NNH2. They are related to ketones and aldehydes by the replacement of the oxygen with the NNH2 functional group. They are formed usually by the action of hydrazine on ketones or aldehydes.

Keywords: Omission; environment; multinationals

Introduction

HYDRAZONES:

Hydrazones are a class of organic compounds with the structure R1R2C=NNH2. They are related to ketones and aldehydes by the replacement of the oxygen with the NNH2 functional group. They are formed usually by the action of hydrazine on ketones or aldehydes.



Structure of the hydrazone functional group

Hydrazones has shown that they possess a wide variety of biological activities viz. antimicrobial, anticonvulsant, antidepressant, anti inflammatory, analgesic, antiplatelet, antimalarial, anticancer, antifungal, antitubercular, antiviral, cardio protective.

CHAPTER-2
AIM
AND
OBJECTIVES

AIM AND OBJECTIVES

To select a topic for the research work from the current problems prevailing in the society.
To collect the multidimensional information in depth about each subtopic involved in the area of research work from different sources.
To plan, acquire execute, evaluate and record the different subtopics of the topic research work in scientific manner.
To work collaboratively in harmonial way with other disciplines of sciences for ensuring the successful completion of research work.
To synthesize crude and recrystallized samples of the compounds by conventional, nonconventional methods.
To purify the compounds by different purification techniques.
To characterize the pure compounds by physical methods and spectral methods.

CHAPTER-3
LITERATURE
SURVEY

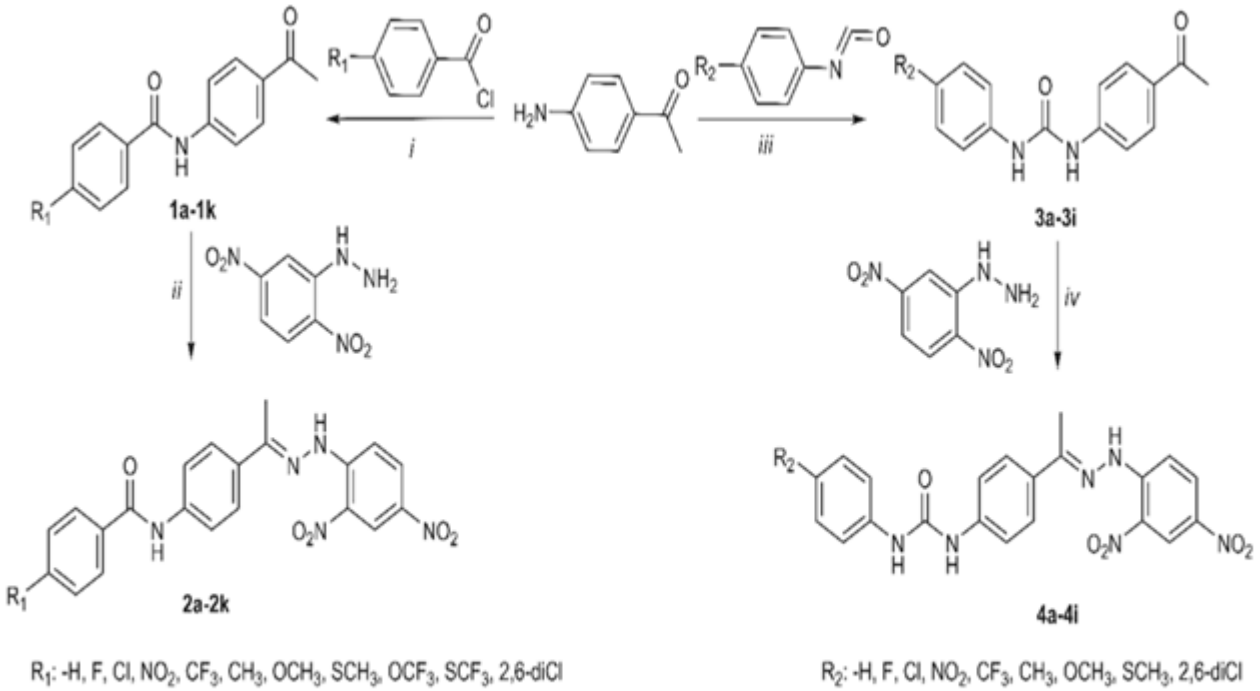
LITERATURE SURVEY

The literature survey was done using search engines like Google, Yahoo, Chemfinder and science Finder along with textbooks, reference books compendia, and journals. Science Finder search includes search of the chemical abstract(CA), chemical abstract plus(CA PLUS)and MEDLINE. The search was done using the keywords like esters, hydrazides,hydrazones etc. The outcome of the survey is presented below.

SYNTHETIC REVIEW:

Reaction Schemes:

Synthesis of new hydrazone derivatives and evaluation of their monoamine oxidase inhibitory activity.

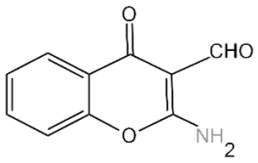


R_1: -H, F, Cl, NO_2, CF_3, CH_3, OCH_3, SCH_3, OCF_3, SCF_3, 2,6-diCl

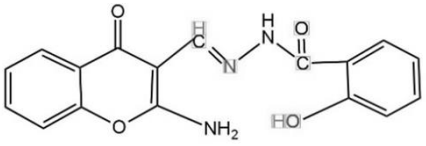
R_2: -H, F, Cl, NO_2, CF_3, CH_3, OCH_3, SCH_3, 2,6-diCl

The synthesis route of all compounds. Reagents: (i) chloroform, reflux; (ii) methanol, reflux; (iii) dichloromethane, 25 °C; (iv) methanol, reflux.

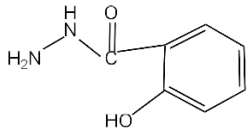
Facile and straightforward synthesis of Hydrazone derivatives

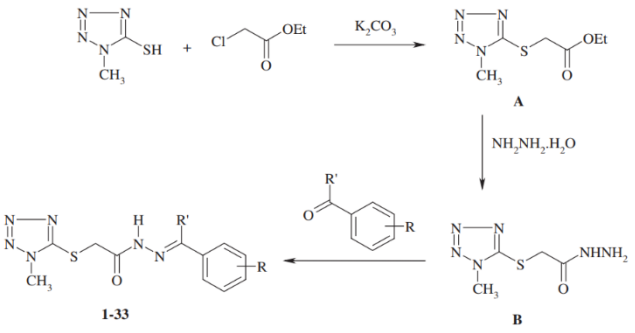


Synthesis and biological evaluation of some hydrazone derivatives as new anticandidal and anticancer agents.



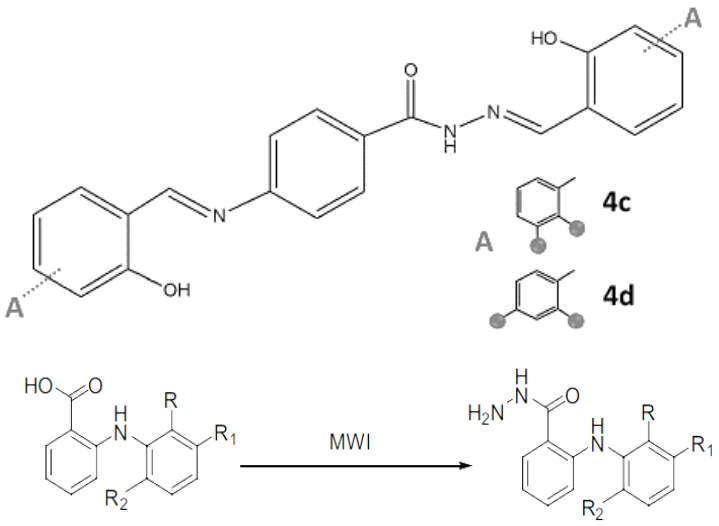
Effective methods for the synthesis of hydrazones, quinazolines, and Schiff bases: reaction monitoring using a chemometric approach.



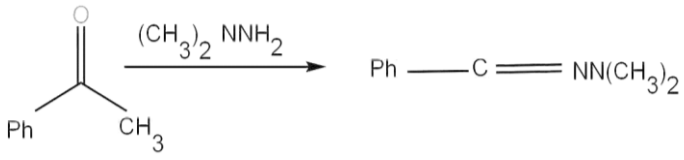


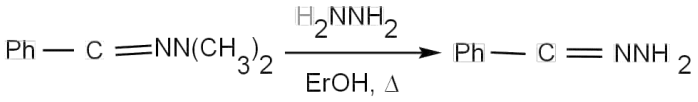
A New Procedure for Preparation ofCarboxylic Acid Hydrazides.

Microwave-Assisted One-Step Synthesis of Fenamic acid Hydrazides from the Corresponding Acids.



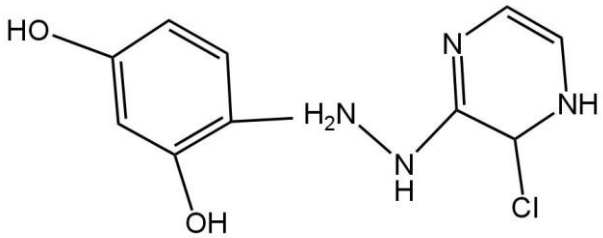
Synthesis and Antitumor Evaluation ofMenthone-Derived Pyrimidine-Urea Compounds as Potential PI3K/Akt/mTOR Signaling Pathway Inhibitor.





Preparation Of Hydrazones: Acetophenone Hydrazone

Biological Activity Review:
Hydrazones as Antibacterial agent
Lee et al. synthesized various hydrazones (1,2) as selective inhibitors of staphylococcus aureus β-ketoacyl carrier protein III.



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Hydrazones as Antifungal agent
Hydrazone derivativessynthesized by Ozdemir et al. (2010) after being screened against different Candida spp have been reported to have promising antifungal potential.

Chapter – 4
Materials and Methods

Apparatus:
Reflux condenser
Bunsen burner
Tripod stand
Water bath
Magnetic stirrer
Hotplate
Ice bath
Beakers
Round bottom flask
Separating funnel
Glass rods
Spatulas
Sample bottles
Thermometers
Capillary tube
Condenser pipes etc

CHEMICALS:
Sodium/potassium dichromate
Concentrated sulphuric acid
Menthol
Ester
Sodium hydroxide
PABA
Ether
Ethanol
5% sodium bicarbonate
Hydrazine hydrate
Methanol
Glacial acetic acid
Distilled water
Chloroform
Methyl salicylate
Recrystallizing solvents

Principle:
The scheme involves 2 steps
Step 1 involvescondensation of l-menthone with benzoylhydrazides in the presence of glacial acetic acid.
Step 2 involves reaction between 4-aminobenzohydrazide and l-menthone.

REACTION PROCEDURE:
STEP 1: Synthesis of l-menthone from menthol

SYNTHESIS:

In a 1-litre round-bottomed flask provided with a mechanical stirrer is placed 120 g (0.4 mole) of crystallized sodium dichromate (or an equivalent amount of potassium dichromate), and to this is added a solution of 100 g(54.3 cc., 0.97 mole) of concentrated sulfuric acid (sp. gr. 1.84) in 600 ml of water. To this mixture 90 g. (0.58 mole) of menthol (crystals, m.p. 41–42°) is added in three or four portions and the mixture stirred. Heat is evolved, and the temperature of the mixture rises to about 55°. As soon as the reaction is complete the temperature falls. The oil is mixed with an equal volume of ether, separated in a separatory funnel, and washed with three 200-ml. portions of 5 % sodium hydroxide solution. The ether is then removed by distillation and the residue distilled under reduced pressure, the menthone being collected at 98–100°/18 mm. If distilled under atmospheric pressure it boils at 204–207°c. The yield is 74–76 g. (83–85 percent of the theoretical amount).

Physical state	Solid
Physical constant	Boiling point: 204-207^{o} c
Recrystallized solvent	Ethanol
Theoretical yield	8.8g
Practical yield	7.3g
Percentage yield	82.95%
Rf value	0.64

STEP 2: Synthesis of benzoyl hydrazides

Place 10 ml of hydrazine hydrate (caution:- corrosive chemical) in a test tube fitted with a short reflux condenser. Add 15ml of methyl salicylate dropwise and heat the mixture gently under reflux for 15mins. Then add just enough methanol through the condenser to produce a clear solution, reflux for further 6 hours, and cool. Filter off the solid of the benzoyl hydrazides.

SYNTHESIS:

Physical state	
Physical constant	
Recrystallized solvent	
Theoretical yield	
Practical yield	
Percentage yield	
Rf value	

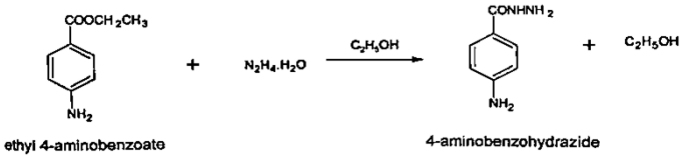
STEP 3: Synthesis of Hydrazones from l-menthone and Benzoyl hydrazide

To solution of l-menthone (1.0g, 6.5mmol) in methanol (25ml), the equimolar amount (6.5mmol) of appropriate benzoyl hydrazide and 2 drops of glacial acetic acid were added. The reaction mixture was refluxed for 4hrs, cooled and then was poured into the ice. The solid mass was filtered, dried &purified by recrystallisation from methanol: water (1:1) system.

SYNTHESIS:

Physical state	
Physical constant	
Recrystallized solvent	
Theoretical yield	
Practical yield	
Percentage yield	
Rf value	

STEP 4: Synthesis of hydrazide from acid ester



SYNTHESIS:

A mixture of acid ester and an excess of hydrazine hydrate were refluxed for about 16 hrs and cooled. The solid was separated by filtration, dried and recrystallized from ethanol to afford hydrazides were characterized on the basis of physical and spectral data. The purity was checked by single spot TLC.

Physical state	Solid
Physical constant	MP- 225-227^0 C
Recrystallized solvent	Ethanol
Theoretical yield	1.5g
Practical yield	1g
Percentage yield	66.6%

Rf value	0.7

STEP5:SynthesisofHydrazones from l-menthone and4-aminobenzohydrazide

To solution of l-menthone (1.0g,6.5mmol) in methanol (25ml), the equimolar amount (6.5mmol) of appropriate 4-aminobenzohydrazide and 2 drops of glacial acetic acid were added. The reaction mixture was refluxed for 4hrs, cooled and then was poured into the ice. The solid mass filtered, dried and purified by recrystallisation from methanol: water (1:1) system.

SYNTHESIS:

Physical state	
Physical constant	
Recrystallized solvent	
Theoretical yield	
Practical yield	
Percentage yield	
Rf value	

CHAPTER 5
RESULTS AND DISCUSSION

Compound profile
Structure:

IUPAC name:
Molecular formula:
Molecular weight:
Theoretical yield:
Practical yield:
Percentage yield:

Compound profile
Structure:

IUPAC name:
Molecular formula:
Molecular weight:
Theoretical yield:
Practical yield:
Percentage yield:

1. TABLE OF CHARACTERIZATION:

COMPOUND NAME

MOLECULAR WEIGHT

PHYSICAL STATE

COLOR

PHYSICAL CONSTANT

RECRYSTALLIZING
SOLVENT

ELUENT

Rf

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[8] Synthesis and biological evaluation of some hydrazone derivatives as new
anticandidal and anticancer agents Article history: Received 30 July 2012, Received in revised form 5 October 2012, Accepted 8 October 2012,Available online 16 October 2012