Vol 2 Issue 10 April 2013

Impact Factor : 0.1870

ISSN No :2231-5063

Monthly Multidisciplinary Research Journal

GoldenResearch Thoughts

IMPACT FACTOR : 0.2105

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RNI MAHMUL/2011/38595 **ISSN No.2230-7850**

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Golden Research Thoughts
Volume 2, Issue. 10, April. 2013
ISSN:-2231-5063
Available online at www.aygrt.isrj.net

ORIGINAL ARTICLE

ECO FRIENDLY ROUTE OF DEHYDROGENATION OF 2,3-DIHYDRO-2-PHENYL-4-QUINOLONE TO 2-PHENYL-4-QUINOLONE USING DIACETOXY IODO BENZENE

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Abstract:

As an alternative reagent to various traditional dehydrogenating reagents Diacetoxy iodo benzene is stable, non-hazardous, acidic and has been successfully used for dehydrogenation reactions[1].
Herein we report a new and ecofriendly route for dehydrogenation of 2,3-dihydro-2-phenyl-4-quinolone to 2-phenyl-4-quinolone using diacetoxy iodo benzene and potassium hydroxide[2].

A series of 2,3-dihydro-2-phenyl-4-quinolones3 has been synthesized using acid-catalyzed one-pot reaction quinolones were prepared through cyclization of the condensation product that were formed by heating of arylamines and ethyl benzoylacetate in toluene. Similarly, the 6 (7 or 8)-substituted 2,3-dihydro-2-phenylquinolones were prepared from the para (ortho or meta)-substituted aniline.

KEYWORDS:

2,3-dihydro-2-phenyl-4-quinolone, 2-phenyl-4-quinolone, DIB.

INTRODUCTION:

Quinolones[4] are analogues of flavanones and thiaflavanones which are characterized by a fused benzoring and phenyl substituent.

Quinolones are broad-spectrum antibiotics that play an important role in treatment of serious bacterial infections, especially hospital-acquired infections and others in which resistance to older antibacterial classes is suspected.

Quinolones are bactericidal agents that target the bacterial DNA gyrase enzyme. Many quinolones have pharmacody-namic properties that result in high intracellular concentra-tions in host inflammatory cells.

RESULT AND DISCUSSION:

A series of novel substituted 2,3-dihydro-2-phenyl-4-quinolones were prepared by cyclisation of substituted 1-(2'aminophenyl)-3-phenyl-2-propene-1-one by using $ZnCl_2$. Substituted 2,3-dihydro-2-phenyl-4-quinolones were dehydrogenated using diacetoxy iodo benzene in 0.1 N KOH.

Title :ECO FRIENDLY ROUTE OF DEHYDROGENATION OF 2,3-DIHYDRO-2-PHENYL-4-QUINOLONE TO 2-PHENYL-4 QUINOLONE USING DIACETOXY IODO BENZENE. Source:Golden Research Thoughts [2231-5063] PANDE G.B. , KENDRE K.L. AND SHIRODKAR S.G. yr:2013 vol:2 iss:10

Table No. 1
PHYSICAL PARAMETERS AND ELEMENTAL ANALYSIS OF 2,3DIHYDRO 2-PHENYL 4-QUINOLONES

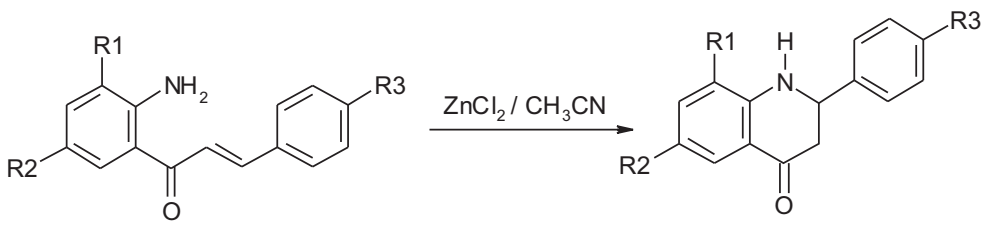
Sr. No.	Compound	M.P. (^{0}C)	Mol. Weight	% Yield
1.	2-phenyl-8-chloro-4-quinolone	242	258	87
2.	2-phenyl-8-bromo-4-quinolone	245	303	82
3.	2-phenyl-8-iodo-4-quinolone	248	350	80
4.	2-phenyl-8-fluoro-4-quinolone	251	242	70
5.	2-phenyl-6,8-chloro-4-quinolone	232	293	86
6.	2-phenyl-6,8-bromo-4-quinolone	258	381	82
7.	2-phenyl-6,8-iodo-4-quinolone	230	475	72
8.	2-(4'chlorophenyl)-6,8-dichloro-4-quinolone	240	292	84
9.	2-(4'methoxyphenyl)-6,8-dichloro-4-quinolone	255	288	72
10.	2-(4'methoxyphenyl)-6,8-dichloro-4-quinolone	245	292	70

EXPETIMENTAL:

All the chemicals used were of S.D. Fine chemicals. All the solvent used were distilled previously. Clay was purchased from Aldrich chemicals.

Melting points were measured in open glass capillaries on a Perfit Electro-thermal melting-point apparatus and are uncorrected. ^{1}H NMR spectra were recorded at room temperature on a 300 MHz. Varian Inova Spectrometer in $CDCl_3$ using TMS as internal standard level for all the experiments. The reactions were monitored by TLC using pre-coated plates (Merck).

Preparation of 2,3-dihydro-2-phenyl-4-quinolone:

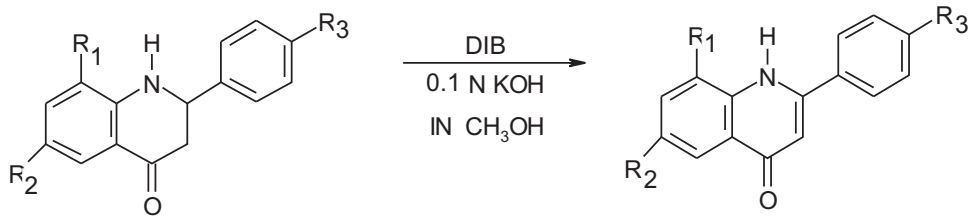


(4) (5)

A solution of substituted 1-(2'aminophenyl)-3-phenyl-2-propene-1-one (3.0 m moles) and Zinc chloride (1 M in Et_2O, 3.3 m mole) in CH_3CN (12 ml) was heated to 800C for (24 hrs) after evaporation of CH_3CN the mixture was poured into saturated solution of NH_4Cl (30 ml) and extracted with methylene chloride (3 x 20 ml).

GENERAL PROCEDURE:

Dehydrogenation of 2,3-dihydro-2-phenyl-4-quinolone to 2-phenyl-4-quinolone :



COMPOUND - V COMPOUND - VI

To compound 5 (2 m.mole) was added a solution of 0.1 N KOH in CH_3OH (60 ml 6 m. mole) and Diacetoxy Iodo benzene (7.09 mg 2.2 m mole) at room temperature. The mixture was heated to 600C for 16 hrs. After evaporation of CH_3OH, 0.05 N HCl (50 ml) was slowly added to the mixture at O^0C. The resulting precipitate was separated by filtration washed with H_2O and re-crystallized by CH3OH.

SPECTRALANALYSIS:

The structures of the products were confirmed from NMR, IR and LCMS. The representative spectral analysis for few of the products is given below. The observed values are in accordance with the literature values.

SPECTRALANALYSIS:

Compound - I

PMR : 4.0 (1H,s), 7.21(5H,m), 4.44(1H,t), 3.07 (2H,d), 7.55 (1H,d), 6.60 (1H,dd), 7.23 (1H,d).
IR : 3432 (NH), 3064, 2964, 1632 (C=O, C=C), 1580, 1546, 1504, 1472, 1450, 1432, 1256, 1140, 770 cm.

Compound - II

PMR :4.0 (1H,s), 7.18 (5H,m), 4.40 (1H,t), 3.02 (2H,d) 7.61 (1H,d) 6.55 (1H,dd) 7.39 (1H,d).
IR : 3430 (NH), 3068, 2960, 1630 (C=O, C=C), 1580, 1546, 1500, 1470, 1452, 1432, 1142, 768 cm.

Compound - III

PMR :4.02 (1H,s), 7.20 (5H,m), 4.42 (1H,t), 3.04 (2H,d), 7.66 (1H,d), 6.43 (1H,dd), 7.60 (1H,d).
IR : 3436 (NH), 3065, 2963, 1628 (C=O, C=C), 1578, 1540, 1502, 1474, 1455, 1432, 1250, 1140, 766 cm.

Compound - IV

PMR :4.04 (1H,s), 7.16 (5H,m), 4.40 (1H,t), 3.05 (2H,d), 7.39 (1H,d), 6.66 (1H,dd), 6.98 (1H,d).
IR : 3436 (NH), 3068, 2966, 1636 (C=O, C=C), 1588, 1548, 1508, 1478, 1458, 1436 1255, 1140, 772cm.

Compound - V

PMR :4.02 (1H,s), 7.12 (5H,m), 4.40 (1H,t), 3.06 (2H,d), 7.52 (1H,s), 7.20(1H,s).
IR : 3436 (NH), 3068 2960, 1630 (C = O, C = C), 1588, 1548, 1506, 1470, 1448, 1436, 1258, 772 cm.

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