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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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Nanded(M.S.), Department of Chemistry, Yeshwant Mahavidyalaya, Nanded.

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 reactions¹.

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 hydroxide².

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:

Quinolones⁴ 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.

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

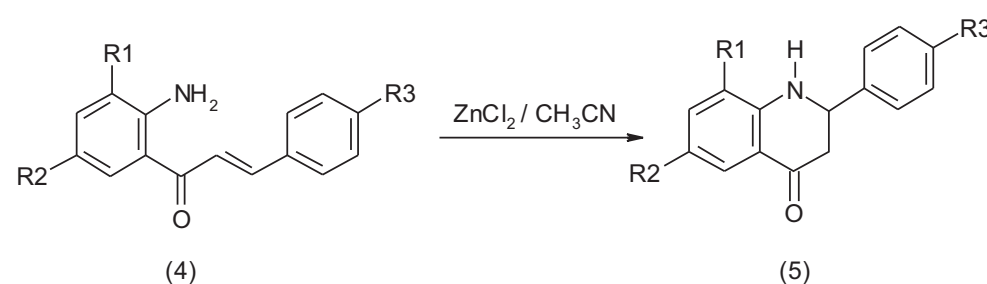
| Sr. No. | Compound | M.P. (°C) | Mol. Weight | % Yield |
|---------|--|-----------|-------------|---------|
| 1. | 2-phenyl-8-chloro-4-quinolone | 242 | 258 | 87 |
| 2. | 2-phenyl-8-bromo-4-quinolone | 245 | 303 | 82 |
| 3. | 2-phenyl-8-iodo-4-quinolone | 248 | 350 | 80 |
| 4. | 2-phenyl-8-fluoro-4-quinolone | 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. ¹H NMR spectra were recorded at room temperature on a 300 MHz. Varian Inova Spectrometer in CDCl₃ using TMS as internal standard level for all the experiments. The reactions were monitored by TLC using pre-coated plates (Merck).

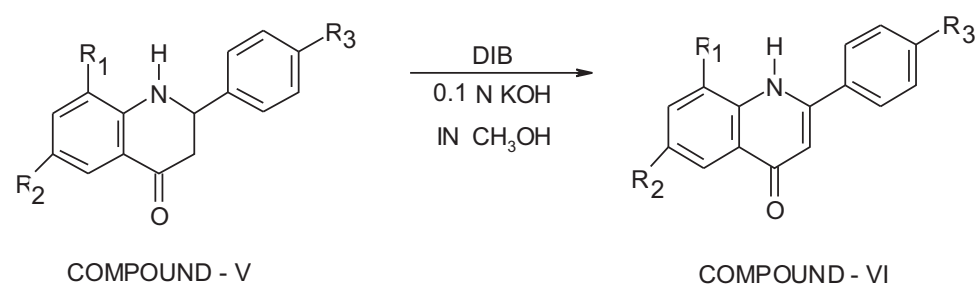
Preparation of 2,3-dihydro-2-phenyl-4-quinolone:



A solution of substituted 1-(2'-aminophenyl)-3-phenyl-2-propene-1-one (3.0 m moles) and Zinc chloride (1 M in Et₂O, 3.3 m mole) in CH₃CN (12 ml) was heated to 80°C for (24 hrs) after evaporation of CH₃CN the mixture was poured into saturated solution of NH₄Cl (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 :



To compound 5 (2 m.mole) was added a solution of 0.1 N KOH in CH₃OH (60 ml 6 m. mole) and Diacetoxy Iodo benzene (7.09 mg 2.2 m mole) at room temperature. The mixture was heated to 60°C for 16 hrs. After evaporation of CH₃OH, 0.05 N HCl (50 ml) was slowly added to the mixture at 0°C. The resulting precipitate was separated by filtration washed with H₂O and re-crystallized by CH₃OH.

SPECTRAL ANALYSIS:

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.

SPECTRAL ANALYSIS:

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, 772 cm.



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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