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Research Paper

Synthesis Of 4,6 – Diaryl -4,5 – Dihydro – 3- Hydroxy – [2H] Indazoles by Microwave Irradiation in Dry Media

Chemistry

KEYWORDS : 4,6 – diaryl -4,5 – dihydro – 3- hydroxy – [2H] indazoles, microwave irradiation, 1H and 13C NMR.

Dr. S. TAMILSELVI — Associate Professor, Department of Chemistry, Government Arts College (Auto), Kumbakonam.

Dr. K.G. PALANI — Associate Professor, Department of Chemistry, Dr. Ambedkar Govt. Arts College. Viyasar Padi, Chennai.

ABSTRACT

4,6 – diaryl -4,5 – dihydro – 3- hydroxy – [2H] indazoles are synthesized by the reaction of 6- carbethoxy-3,5- diaryl cyclo hex-2-enone with hydrazine hydrate in presence of calcium oxide under microwave irradiation in dry media. The structure of the indazoles are confirmed by elemental analysis, 1H and 13C NMR spectral studies.

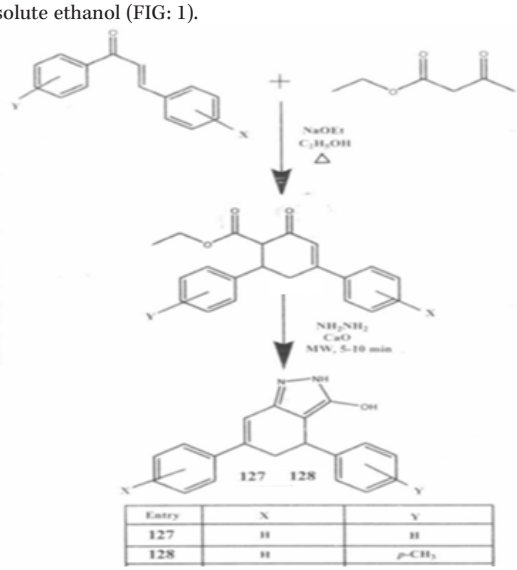
INTRODUCTION

In this method of synthesis of indazoles by microwave irradiation in dry media, the reactions can be run safely under atmospheric pressure with increased amount of products.

The attractive features of this procedure are the mild reaction condition, high yield conversions, solvent – free reaction conditions, operational simplicity, inexpensive and readily available catalyst, all of which make it a useful and attractive strategy for the synthesis of indazoles[1-4].

EXPERIMENTAL

6 – Carbethoxy – 3,5 – diaryl cyclohex -2-enone (0.1mol) was mixed with hydrazine hydrate (0.1mol) in the presence of calcium oxide (200 mg) properly with the help of a glass rod and then irradiated in a microwave oven for 5-10 min at 320w, the reaction mixture was removed from the oven, cooled and shaken with methanol, the catalyst was removed by filtration. The filtrate was dried and washed with excess of water and then extracted with chloroform then the crude was recrystallised from absolute ethanol (FIG: 1).



Entry	X	Y
127	H	H
128	H	*p*-CH_3

FIG: 1 – SYNTHESIS OF SOME 4,6 – DIARYL – 4,5, - DIHYDRO-3-HYDROXY-2[H]-INDAZOLE

4.6 – dipheny1-4, 5-dihydro-3-hrydroxy-2[H]-indazole (127)

The elemental analysis [C_{cal} =79.16, C_{obs} = 79.23; H_{cal}=7.01, H_{obs}=6.99; N_{cal}=9.72, N_{obs}=9.76] is consistent with the proposed molecular formula ($C_{19}H_{16}N_2O$) of **127**

Analysis of ^{1}H NMR Spectrum of 127

In the ^{1}H NMR spectrum of **127 (Plate 34),** a doublet of doublet appeared at 4.19 ppm is due to the H_{4a}. Two multiplets around 2.8 and 3.20 ppm are assigned to methylene protons at C-5. The doublet for one proton at 6.73 ppm has been conveniently assigned to H-7. The aromatic protons are appeared in the range of 7.10 – 7.48 ppm.

PLATE 34: ^{1}H NMR spectrum of 4.6 – dipheny1-4, 5-dihydro-3-hrydroxy-2[H]-indazole

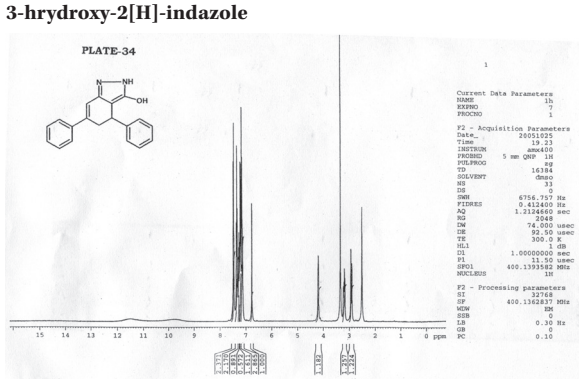
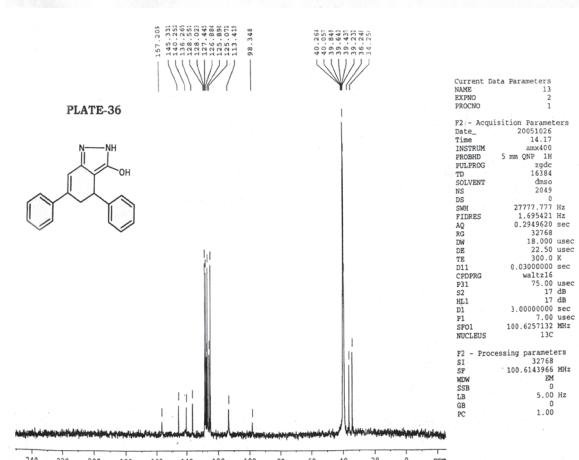


PLATE 36: ^{13}C NMR spectrum of 4.6 – dipheny1-4, 5-dihydro-3-hrydroxy-2[H]-indazole



Analysis of ^{13}C NMR spectrum of 127

In ^{13}C spectrum **(Plate 36)** two reasonances in the aliphatic range 34.2 and 36.2 ppm have been observed.

The remaining ^{13}C resonances in quaternary carbon signals at

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157.2, 113.4, 136.2,98.3 and 140.2, 145.3 ppm are due to C-3, C-7, C-8, C-9 and ipso carbons. The aromatic carbons are observed in range of 125.0-128.5 ppm.

4-(p-methylphenyl)-6-phenyl-4,5-dihydro-3-hydroxy-2-[H]-indazole (128)

Compound 128 is synthesized by the reaction of 6-carbethoxy -3- pheny1-5-(p-methylphenyl) cylcohex-2-enone with hydrazine hydrate in the presence of calcium oxide under microwave irradiation in dry media.

CONCLUSION

The yields of 4,6-diaryl-4,6-dihydro-3-hydroxy-2[H]-indazoles (127-128) are more than 90%. More over, the yields are practically unaffected upon recycling of calcium oxide catalyst upon 4 times in an environmentally friendly manner.

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