SYNTHESIS OF 2,6-DIBENZYLIDENECYCLOHEXANONE AND E-7-BENZYLIDENE-3-PHENYL-3,3a,4,5,6,7-HEXAHYDRO-2H-INDAZOLE

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Abstract : 2-(phenyl(phenylamino)methyl)cyclohexanone (Mannich base) in acetic acid under reflux condition undergoes elimination reaction to give 2,6-dibenzylidene cyclohexanone. It is further used for the synthesis of (E)-7-benzylidene-3phenyl-3,3a,4,5,6,7-hexahydro-2H-indazole using a mild mild catalyst ammonium chloride.

Keywords : Indazole, Dibenzylidene Cyclohexanone, heterocyclic compounds, biological activities, β-amino carbonyl compounds.

I. INTRODUCTION

Indazole is one important class of heterocyclic compounds. The indazole nucleus is a seldom used in medicinal chemistry but it is an effective Pharmacophor, shows interesting biological activities and powerful pharmacological activities such as antimicrobial,[1] anti-inflammatory,[2] antipsychotic,[3] antitumor,[4] antiproliferative,[5] antibacterial[6], trichomocidal,[7] analgesic,[8] antipyretic,[8] antiprotozoal,[8] antispermatogenic.[9] It also useful in the treatment of hypertension,[10] obesity,[11] rheumatoid arthritis.[12]

Literature survey reveals that limited no of methods reported for the synthesis of Indazoles. Therefore it is necessary to find convenient method for the synthesis. Here we developed a new and environment friendly method for the synthesis of Indazoles with trans stereoselectivity.

The α,β – unsaturated ketones play an important role as an intermediate in the synthesis of various heterocyclic compounds such as pyrrazolines and isoxazolines. Generally these compounds are prepared by Claisen-Schmidt condensation from aromatic aldehyde and ketones.[13,14,15,16] The α,β – unsaturated derivatives of cyclohexanones shows biological activities such as antiangiogenic,[17] cytotoxicity,[18] cholesterol lowering activity.[19] These are also used in the pharmaceuticals, perfumes and agrochemicals.[20]

The synthesis of α,α'-bis-(substituted benzylidene)-cycloalkanone are catalysed by strong acids,[21] base,[22] various reagents used Cp_2ZrH_2,[23] $RuCl_3$,[24] $TiCl_3(CF_3SO_3)$, SmI_3,[25] $Mg(HSO_4)_2$, $InCl_3$,[26] $SOCl_2$, $Yb(OTF)_3$, silica supported phosphorous pentoxide (P_2O_5/SiO_2). However most of the reported reactions suffer from reverse or side reactions such as self condensation of ketones and aldehydes giving the corresponding product in lower yield,[27] long reaction procedure, harsh reaction conditions, expensive reagents. It is therefore very important to find more convenient method for the preparation of bis benzylidene cyclohexanone. Here we reported novel and simple method for the synthesis of bis benzylidene cyclohexanone from β-amino carbonyl compounds and its application in the synthesis of indazoles.

II. MATERIAL AND METHODS

Melting points were determined on electrothermal model 9100 apparatus and are uncorrected. Merck, pre-coated silica gel 60 F254 (Aluminum sheets) plates were used for analytical TLC. IR spectra were recorded (in KBr pellets) on Shimadzu spectrophotometer. ^{1}H NMR spectra were recorded (in $CDCl_3$) on Varian Mercury 400 MHz spectrometer using TMS as an internal standard.

General Procedure for the synthesis of (2E,6E)-2,6-dibenzylidenecyclohexanone-

The compound 2-(phenyl(phenylamino)methyl)cyclohexanone in AcOH refluxed for 5-10 min. Progress of reaction followed by TLC. After cooling the reaction mixture in ice yellow coloured crystals obtained are filtered and washed with aqueous EtOH. Product obtained with good yield and excellent purity.

General Procedure for the synthesis of (E)-7-benzylidene-3-phenyl-3,3a,4,5,6,7-hexahydro-2H-indazole-

To a compound of (2E,6E)-2,6-dibenzylidenecyclohexanone (1 eq) in Ethanol ammonium chloride was added (0.05eq), the mixture was refluxed to that added hydrazine hydrate (80%) (4 eq) dropwise for 15 min. The heating continued for 1- 2 h. TLC followed the progress of reaction. After the reaction was completed, the reaction mixture kept at 0^0C for 2-3 h. White crystals obtained are filtered and washed with aqueous ethanol. The product obtained with good yield and excellent purity.

III. Result and Discussion-

When 2-((3-chlorophenylamino)(phenyl)methyl)cyclohexanone refluxed for 10 min in acetic acid and cooled to 0°C fine yellow crystals obtained. The product was bis benzylidene cyclohexanone. Same reaction studied in various solvents such as MeOH, water, DMF, DMSO, $CHCl_3$, DCM, AcOH, among these AcOH was found to be good solvent as reaction completed immediately

and pure crystals obtained on cooling. For generalisation of reaction we used various substituted β-amino carbonyl compounds. The result shown in below table no. 1.

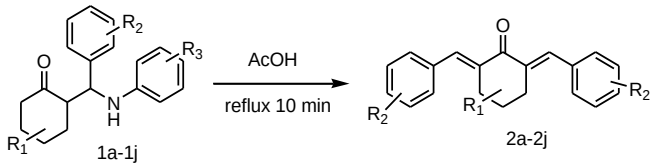
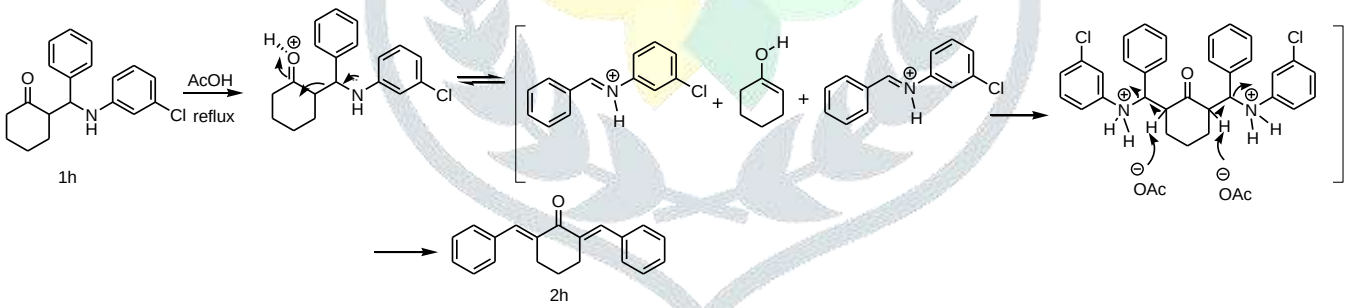


Table no 1.

Sr. No.	R_1	R_2	R_3	M.P.	% Yield
a	H	4-CH_3	3-Cl	138-140	35
b	H	4-Br	3-Cl	156-158	38
c	H	2,3-diCl	3-Cl	90-95	45
d	H	2,4-diCl	3-Cl	160-163	40
e	H	4-Cl	3-Cl	120-124	42
f	H	4-F	2-Cl	128-130	43
g	4- CH_3	2,3-diCl	3-Cl	118-120	41
h	H	H	3-Cl	109-112	40
i	H	2,3-diCl	4-Cl	91-94	40
J	H	4-Cl	2-Cl	121-123	36

Proposed mechanism –The Mannich base in acetic acid under reflux condition undergoes retrosynthesis and formation of synthons cyclohexanone and Schiff base. Further cyclohexanone from both enolisable sides reacts with two molecules of Schiff base afforded corresponding dimannich base which further undergoes eliminaton reaction and gives corresponding dibenzylidene cyclohexanone with good yield.



Further we used these substituted (2E,6E)-2,6-dibenzylidenecyclohexanone for the synthesis of various substituted (E)-7-benzylidene-3-phenyl-3,3a,4,5,6,7-hexahydro-2H-indazole. For the continuation of our work on ammonium chloride we tried the reaction using ammonium chloride catalyst, using this the product obtained in good yield and excellent purity, for the optimization of reaction condition did the reaction in various solvents such as DCM, EtOH, DMF, DMSO, AcOH, the reaction goes well in EtOH in 10 min. White crystals obtained on cooling the reaction mixture was filtered and washed with aqueous ethanol (1:1). The result shown in below table no. 2.

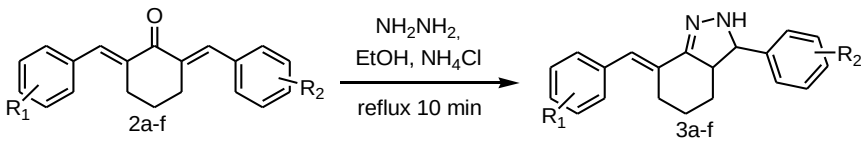


Table No. 2

Sr. No.	R_1	R_2	M.P.	% Yield
a	H	4-Cl	132-138	80
b	H	2,3-diCl	79-82	83
c	H	4-CH_3	115-130	75
d	H	H	75-80	70
e	H	Napthaldehyde	91-92	74
f	H	4-Br	162-172	81

Characterisation:-

(2E,6E)-2,6-bis(4-methylbenzylidene)cyclohexanone (2a)

Yellow solid, m.p. 138-140 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (s, 2H), 7.40 (d, *J* = 8.1 Hz, 4H), 7.31 – 7.19 (m, 4H), 2.99 – 2.91 (m, 4H), 2.41 (s, 6H), 1.87 – 1.75 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.43, 138.80, 136.87, 135.53, 133.25, 130.47, 129.14, 28.53, 23.04, 21.40. IR (KBr):2915, 1659.35, 1596.92, 1267.39, 1153.86, 817.10, 520.97, cm^{-1}

(2E,6E)-2,6-bis(4-bromobenzylidene)cyclohexanone (2b)

Yellow solid, m.p. 156-158 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (s, 2H), 7.58 – 7.52 (m, 4H), 7.34 (d, *J* = 8.4 Hz, 4H), 2.90 (td, *J* = 6.6, 2.0 Hz, 4H), 1.87 – 1.75 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.83, 136.55, 135.83, 134.76, 131.80, 131.66, 122.96, 28.38, 22.81. IR (KBr): 2922.13, 1665.54, 1568.74, 1482.59, 1267.69, 1136.26, 827.22, 518.94, cm^{-1}.

(2E,6E)-2,6-bis(2,3-dichlorobenzylidene)cyclohexanone (2c)

Yellow solid, m.p. 90-95 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (s, 2H), 7.47 (dd, *J* = 6.2, 3.3 Hz, 2H), 7.30 – 7.20 (m, 4H), 2.75 (td, *J* = 6.7, 1.8 Hz, 4H), 1.90 – 1.60 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.41, 138.20, 136.63, 134.11, 133.64, 132.99, 130.21, 128.55, 126.82, 28.29, 23.02. IR (KBr): 2928.10, 1592.89, 1407.55, 1136.20, 1044.48, 788.22, 730.18, 551.12, 455.57.

(2E,6E)-2,6-bis(2,4-dichlorobenzylidene)cyclohexanone (2d)

Yellow solid, m.p. 160-163 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (s, 2H), 7.48 (s, 2H), 7.28 (d, *J* = 0.9 Hz, 4H), 2.80 – 2.73 (m, 4H), 1.83 – 1.74 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.26, 138.05, 135.82, 134.88, 133.10, 132.86, 131.17, 129.71, 126.72, 28.36, 23.03. IR (KBr): 2924.36, 1604.44, 1468.60, 1132.84, 1027.24, 824.00, 743.27, 558.73, 466.46.

(2E,6E)-2,6-bis(4-chlorobenzylidene)cyclohexanone (2e)

Yellow solid, m.p. 120-124 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (s, 2H), 7.42 – 7.35 (m, 8H), 2.94 – 2.84 (m, 4H), 1.85 – 1.76 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.80, 136.43, 135.77, 134.63, 134.32, 131.60, 128.69, 28.38, 22.82. IR (KBr): 2927.85, 1607.60, 1489.74, 1265.95, 1106.01, 1087.18, 521.74, 454.20, 398.59.

(2E,6E)-2,6-bis(4-fluorobenzylidene)cyclohexanone (2f)

Yellow solid, m.p. 128-130 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (s, 1H), 7.54 – 7.42 (m, 2H), 7.17 – 7.07 (m, 2H), 2.98 – 2.83 (m, 2H), 1.88 – 1.73 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.99, 163.92, 161.44, 135.87, 132.31, 115.64, 115.43, 28.35, 22.91. IR (KBr): 2929.97, 1602.74, 1503.47, 1267.62, 1156.96, 835.36, 530.72, 488.99.

(2E,6E)-2,6-bis(2,3-dichlorobenzylidene)-4-methylcyclohexanone (2g)

Yellow solid, m.p. 118-120 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88 (d, *J* = 1.8 Hz, 2H), 7.48 (dd, *J* = 7.6, 1.7 Hz, 2H), 7.27 – 7.20 (m, 4H), 2.83 (dd, *J* = 15.5, 3.3 Hz, 2H), 2.41 – 2.29 (m, 2H), 1.96 – 1.82 (m, 1H), 1.02 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.18, 137.34, 136.66, 134.42, 133.65, 133.00, 130.21, 128.56, 126.83, 36.23, 29.44, 21.32.

(2E,6E)-2,6-dibenzylidenecyclohexanone (2h)

Yellow solid, m.p. 109-112 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (s, 2H), 7.50 (d, *J* = 7.4 Hz, 4H), 7.44 (t, *J* = 7.4 Hz, 4H), 7.39 – 7.34 (m, 2H), 3.02 – 2.89 (m, 4H), 1.82 (dt, *J* = 12.4, 6.3 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.41, 136.96, 136.23, 136.01, 130.39, 128.61, 128.41, 28.48, 23.04.

(2E,6E)-2,6-bis(2,3-dichlorobenzylidene)cyclohexanone (2i)

Yellow solid, m.p. 91-94 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (s, 2H), 7.46 (dd, *J* = 6.2, 3.3 Hz, 2H), 7.31 – 7.21 (m, 4H), 2.74 (td, *J* = 6.7, 1.8 Hz, 4H), 1.91 – 1.61 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.41, 138.20, 136.64, 134.11, 133.65, 132.99, 130.21, 128.55, 126.82, 28.29, 23.02. IR (KBr): 2929.10, 1593.89, 1407.55, 1136.20, 1045.48, 788.22, 731.18, 551.12, 455.57.

(2E,6E)-2,6-bis(4-chlorobenzylidene)cyclohexanone (2j)

Yellow solid, m.p. 121-123 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 (s, 2H), 7.41 – 7.34 (m, 8H), 2.93 – 2.83 (m, 4H), 1.86 – 1.76 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 189.81, 136.43, 135.77, 134.63, 134.32, 131.60, 128.69, 28.38, 22.82. IR (KBr): 2928.85, 1608.60, 1489.74, 1265.95, 1106.02, 1087.17, 521.74, 454.22, 398.60.

3-(4-chlorophenyl)-3,3a,4,5,6,7-hexahydro-2H-indazole(3a)

White solid, m.p. 132-138 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.4 Hz, 2H), 7.39 – 7.31 (m, 4H), 7.28 (d, *J* = 8.5 Hz, 2H), 7.16 (d, *J* = 2.7 Hz, 1H), 5.99 (s, 1H), 4.51 (d, *J* = 13.9 Hz, 1H), 2.98 (dd, *J* = 15.5, 0.9 Hz, 1H), 2.76 (ddd, *J* = 13.8, 11.5, 5.9 Hz, 1H), 2.42 – 2.31 (m, 1H), 2.02 (dddd, *J* = 16.5, 10.3, 5.7, 2.9 Hz, 2H), 1.60 – 1.36 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.36, 138.93, 135.12, 133.49, 132.94, 131.57, 130.86, 128.77, 128.41, 128.38, 125.14, 72.61, 53.89, 28.56, 28.49, 24.35.

(E)-7-(2,4-dichlorobenzylidene)-3-(2,4-dichlorophenyl)-3,3a,4,5,6,7-hexahydro-2H-indazole (3b)

White solid, m.p. 79-82 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.5 Hz, 1H), 7.47 – 7.28 (m, 5H), 7.24 (d, *J* = 1.6 Hz, 2H), 7.17 (d, *J* = 2.7 Hz, 1H), 5.10 (d, *J* = 13.7 Hz, 1H), 2.91 – 2.74 (m, 2H), 2.40 – 2.27 (m, 1H), 2.26 – 2.16 (m, 2H), 1.97 – 1.88 (m, 2H), 1.68 (ddd, *J* = 15.4, 12.8, 3.0 Hz, 1H), 1.49 – 1.35 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 155.56, 136.80, 135.31, 134.23, 133.78, 133.57, 133.36, 133.14, 131.44, 129.61, 129.42, 129.19, 127.57, 126.52, 122.32, 68.17, 54.05, 29.13, 28.77, 24.57. IR (KBr): 2919.63, 1584.25, 1468.51, 1103.60, 862.70, 816.56, 768.75, 617.48, 578.09,458.72,

(E)-7-(4-methylbenzylidene)-3-p-tolyl-3,3a,4,5,6,7-hexahydro-2H-indazole (3c)

White solid, m.p. 115-130 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 7.6 Hz, 2H), 7.32 – 7.14 (m, 6H), 4.51 (d, *J* = 13.9 Hz, 1H), 3.07 (d, *J* = 15.1 Hz, 1H), 2.83 (td, *J* = 13.5, 5.9 Hz, 1H), 2.41 (s, 6H), 2.03 (dd, *J* = 63.2, 10.8 Hz, 3H), 1.51 (ddd, *J* = 38.6, 24.7, 12.1 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.04, 137.46, 137.36, 137.02, 133.99, 130.41, 129.69, 129.31, 128.94, 127.27, 127.09, 126.33, 73.09, 53.77, 28.72, 24.49, 21.18. IR (KBr): 2920.79, 1509.50, 1042.06, 811.46, 775.71, 530.60, cm^{-1}.

(E)-7-benzylidene-3-phenyl-3,3a,4,5,6,7-hexahydro-2H-indazole (3d)

White solid, m.p. 75-80 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.51 (m, 1H), 7.45 – 7.32 (m, 10H), 4.54 (d, *J* = 13.9 Hz, 1H), 3.06 – 2.98 (m, 1H), 2.88 – 2.79 (m, 1H), 2.45 – 2.36 (m, 1H), 2.28 – 2.11 (m, 2H), 1.95 – 1.83 (m, 1H), 1.51 (ddd, *J* = 13.4, 11.0, 2.9 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 157.14, 140.23, 136.75, 131.06, 130.21, 129.80, 129.68, 128.79, 128.61, 128.19, 127.81, 127.15, 126.52, 73.19, 53.80, 28.62, 24.45. IR (KBr): 2930.93, 1447.24, 828.27, 766.04, 696.71, 532.25, 418.62.

(E)-3-(naphthalen-1-yl)-7-(naphthalen-1-ylmethylene)-3,3a,4,5,6,7-hexahydro-2H-indazole (3e)

White solid, m.p. 91-92 ^{0}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 – 8.17 (m, 2H), 8.06 (d, *J* = 6.9 Hz, 1H), 7.95 – 7.83 (m, 6H), 7.57 (m, 6H), 5.42 (d, *J* = 13.9 Hz, 1H), 3.17 (ddd, *J* = 13.7, 12.0, 5.9 Hz, 1H), 2.86 (d, *J* = 15.0 Hz, 1H), 2.41 (ddd, *J* = 15.8, 7.3, 4.3 Hz, 1H), 2.17 – 2.06 (m, 1H), 1.85 – 1.67 (m, 2H), 1.44 – 1.31 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 156.51, 136.25, 134.07, 133.75, 133.64, 132.96, 132.37, 131.83, 129.10, 128.86, 128.50, 128.25, 127.91, 127.16, 126.15, 126.00, 125.96, 125.74, 125.18, 125.14, 124.94, 124.29, 123.39, 69.69, 54.08, 30.04, 29.20, 24.76.

(E)-7-(4-bromobenzylidene)-3-(4-bromophenyl)-3,3a,4,5,6,7-hexahydro-2H-indazole (3f)

White solid, m.p. 162-172 ^{0}C; ^{1}H NMR (400 MHz, DMSO) δ 7.54 (d, *J* = 8.0 Hz, 4H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 7.9 Hz, 2H), 6.98 (s, 1H), 4.38 (d, *J* = 14.2 Hz, 1H), 2.81 – 2.64 (m, 2H), 2.34 (d, *J* = 14.2 Hz, 1H), 1.94 – 1.75 (m, 2H), 1.58 – 1.25 (m, 2H). ^{13}C NMR (101 MHz, DMSO) δ 153.98, 141.17, 136.12, 132.82, 132.03, 131.83, 131.71, 131.46, 129.67, 123.32, 120.73, 120.56, 72.00, 53.24, 28.30, 24.28. IR (KBr): 2920.54, 1490.38, 1003.41, 829.13, 770.18, 515.16.

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