

Three component one-pot synthesis of 4*H*-benzo-[*b*]-pyran derivatives using [(diacetoxyiodo)benzene] (DIB) as a hypervalent iodine catalyst

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Abstract

The three components one pot synthesis of 2-amino-4*H*-benzo-[*b*]-pyran derivatives were obtained in good to excellent yields within short reaction time *via* the condensation reaction of dimedone, aldehydes and malanonitrile or ethylcyanoacetate using the catalytic amount of [(diacetoxyiodo)benzene] (DIB) as hypervalent iodine in aqueous ethanol under reflux conditions have been discussed. This aqua mediated knoevenagel-cyclocondensation of various aromatic and hetero-aromatic aldehydes along with the aldehydes like aryl-sulphonyloxybenzaldehyde, aryl-carbonyloxybenzaldehyde also leads to the product under the same reaction conditions. High yields, short reaction times, one pot condensation, operational simplicity, easy work-up, and purification of products by non-chromatographic methods are some additional features of the present protocol.

Keywords: 4*H*-benzo-[*b*]-pyran; hypervalent iodine; aldehydes; dimedone; multi-component reaction; aqueous alcohol.

Introduction

The one-pot multi-component condensation represents a possible instrument to perform an ideal synthesis because Wender defined

the 'ideal synthesis' as one in which the target components are produced in one step, in quantitative yields from readily available and inexpensive starting materials in resource

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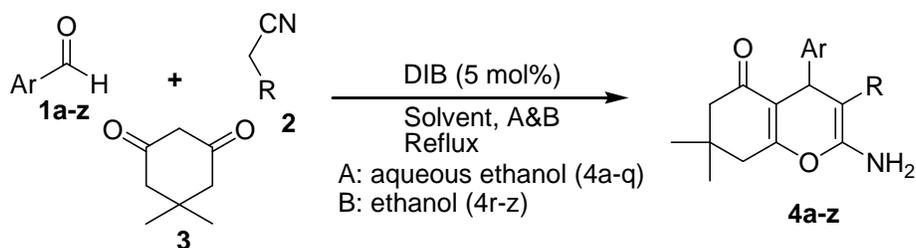
effective and environmentally acceptable process [1]. Nowadays, in the development of new processes, ecological impact must also be taken into account and solvents are prominent features, as they are generally used in large quantities. Such a consideration has prompted synthetic organic chemists to rediscover the potential of water as solvent for organic reactions [2, 3].

Pyrans belongs to an important class of heterocyclic compounds which shows a wide range of biological activities [4], such as spasmolytic, diuretic, anti-coagulant, anti-cancer, anti-anaphylactic activity [5-9]. In addition, they can be used as cognitive enhancers, for the treatment of neurodegenerative disease, including Alzheimer's disease, amyotrophic lateral sclerosis, Huntington's disease, Parkinson's disease, AIDS associated dementia and Down's syndrome as well as for the treatment of Schizophrenia and Myoclonus [10]. They become synthons [11, 12] of some natural products when hydrogen atom of pyran ring is substituted by amino or cyano group. In addition to their biological importance, some 4*H*-benzo-[*b*]-pyrans have been widely used as photoactive materials [13].

The conventional methods reported for the synthesis of 4*H*-benzo-[*b*]-pyrans are

carried out by DMF [14], ionic liquid [15], microwave irradiation [16-19], ultrasound irradiations [20], KF-alumina [21], DABCO [22], TBAF [23], TBAB [24], HDMBAB [25], quaternary ammonium salt [26], IRA-400 (OH) [27], *L*-proline [28], rare earth perfluorooctanoate [29], molecular iodine [30], TFE [31], potassium phosphate [32], urea [33], and nano particles [34, 35]. These methods have their own merits and demerits such as low yields, long reaction time, harsh reaction conditions and tedious work-up procedures. Consequently, there is a need to develop alternative methods for the synthesis of 4*H*-benzo-[*b*]-pyran derivatives under mild conditions.

(Diacetoxyiodo)benzene (DIB) has been known for a long time [36]. It is potent, often oxidizing agent [37]. It also shows applications in several other useful transformations including α -functionalization of carbonyl compounds, carbon-carbon bond forming reactions, and cyclizations [38]. In the present work, we report highly efficient one-pot three component synthesis of 4*H*-benzo-[*b*]-pyrans in the presence of (diacetoxyiodo)benzene (DIB) as hypervalent iodine catalyst in 1:1 aqueous-alcohol medium (Scheme1).



Scheme 1. Synthesis of 2-amino-4*H*-tetrahydrobenzo-[*b*]-pyrans

Experimental

General

All chemicals were purchased from SD fine & Qualigens and used without further purification. All yields were referred to isolate products after purification. Melting points were determined by open capillary method and are uncorrected. IR spectra were recorded on KBr discs on a FT IR Jasco - 4100 type A and the values are expressed as max cm^{-1} , $^1\text{H NMR}$ and $^{13}\text{C NMR}$ spectra were recorded on Bruker advance II 400 NMR spectrophotometer using tetramethylsilane (TMS) as an internal standard. The chemical shift values are recorded on scale and the coupling constants (J) are in hertz. Known compounds were characterized by comparison of their spectral and physical data with literature values. The progress of the reaction was monitored by TLC using aluminium plates with silica gel 60 F₂₅₄ (Merck).

General procedure for the synthesis of 2-amino-4*H*-tetrahydrobenzo-[*b*]-pyrans

In the preparation of 2-amino-4*H*-tetrahydro-

Benzo-[*b*]-pyrans, an equimolar mixture of an aromatic aldehyde, malanonitrile or ethylcyanoacetate, dimedone (2.0 mmol), and (diacetoxyiodo)benzene (5 mol%) was dissolved in 10 mL aqueous ethanol (1:1) for 4a-q and ethanol for 4r-z and refluxed for the specific time indicated in Table 1. The progress of the reaction was monitored by TLC. After completion of the reaction, ice cold water (15.0 mL) was added. The solid product separate out was filtered to yield the corresponding crude products. The crude products were further purified by recrystallization with ethanol to afford pure product 4 in good to excellent yields.

Selected spectral data of the synthesized 2-amino-4*H*-tetrahydrobenzo[*b*]pyrans

2-Amino-4-(4-hydroxy-3-ethoxyphenyl)-3-cyano-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile (4p)

IR (KBr): 3467, 3331, 2186, 1681, 1663, 1212, 1039 cm^{-1} ; $^1\text{H NMR}$ (400 MHz, DMSO): 0.99 (3H, s, CH₃), 1.07 (3H, s, CH₃), 1.35 (3H, t, $J=6.9$ Hz, Et), 2.11 (1H, d,

$J=16.1$ Hz, H-6), 2.23 (1H, d, $J=16.1$ Hz, H-6'), 2.50 (2H, s, H-8), 3.99 (2H, q, $J=7.1$ Hz, Et), 4.08 (1H, s, H-4), 6.53 (1H, d, $J=8.1$ Hz, H-Ar), 6.66 (2H, d, $J=6.8$ Hz, H-Ar), 6.72 (2H, br s, NH₂), 8.55 (1H, s, OH) ppm; ¹³C NMR (CDCl₃, 100 MHz) 14.72, 26.58, 28.47, 31.71, 34.91, 50.01, 58.71, 63.85, 112.91, 113.01, 115.38, 119.40, 119.85, 135.73, 145.53, 146.23, 158.34, 162.10, 195.69 ppm; C₂₈H₂₂N₂O₄ MS (ESI⁺): m/z 355 (M+H)⁺.

2-Amino-4-(5-bromo-4-hydroxy-3-methoxyphenyl)-3-cyano-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (4q)

IR (KBr): 3403, 3323, 2188, 1681, 1663, 1213, 1044, 531 cm⁻¹; ¹H NMR (400 MHz, DMSO): 1.01 (3H, s, CH₃), 1.07 (3H, s, CH₃), 2.14 (1H, d, $J=16.1$ Hz, H-6), 2.24 (1H, d, $J=16.1$ Hz, H-6'), 2.52 (2H, s, H-8), 3.80 (3H, s, OCH₃), 4.12 (1H, s, H-4), 6.70 (1H, s, H-Ar), 6.79 (1H, s, H-Ar), 6.84 (2H, br s, NH₂), 9.12 (1H, s, OH) ppm; ¹³C NMR (100 MHz, DMSO): 26.71, 28.48, 31.71, 34.87, 50.04, 56.01, 58.13, 109.05, 110.21, 112.54, 119.55, 122.61, 136.58, 142.44, 148.05, 158.38, 162.21, 195.48 ppm; C₁₉H₁₉BrN₂O₄ MS (ESI⁺): m/z 419 (M⁺).

2-Amino-4-(4-benzoylphenyl)-3-cyano-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (4r):

IR (KBr): 3364, 3196, 2190, 1735, 1681, 1264, 1060 cm⁻¹; ¹H NMR (400 MHz, DMSO): 1.01 (3H, s, CH₃), 1.07 (3H, s, CH₃), 2.16 (1H, d, $J=16.1$ Hz, H-6), 2.25 (1H, d, $J=16.1$ Hz, H-6'), 2.50 (2H, s, H-8), 4.27 (1H, s, H-4), 6.86 (2H, br s, NH₂), 7.16 (2H, d, $J=6.8$ Hz, H-Ar), 7.26 (2H, d, $J=6.7$ Hz, H-Ar), 7.56 (2H, t, $J=7.8$ Hz, H-Ar), 7.70 (1H, t, $J=7.4$ Hz, H-Ar), 8.13 (2H, t, $J=7.1$ Hz, H-Ar) ppm; ¹³C NMR (100 MHz, DMSO): 26.89, 28.29, 31.81, 35.06, 49.96, 58.07, 112.59, 119.66, 121.69, 128.23, 128.89, 128.96, 129.72, 134.02, 142.43, 149.12, 158.51, 162.56, 164.54, 195.73 ppm; C₂₅H₂₂N₂O₄ MS (ESI⁺): m/z 415 (M+H)⁺.

2-Amino-4-(4-toluenesulphonylphenyl)-3-cyano-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile (4s)

IR (KBr): 3438, 3333, 2187, 1676, 1254, 1094, 1041 cm⁻¹; ¹H NMR (400 MHz, DMSO): 0.96 (3H, s, CH₃), 1.07 (3H, s, CH₃), 2.12 (1H, d, $J=16.1$ Hz, H-6), 2.22 (1H, d, $J=16.1$ Hz, H-6'), 2.47 (3H, s, CH₃), 2.52 (2H, s, H-8), 4.21 (1H, s, H-4), 6.85 (2H, br s, NH₂), 6.89 (2H, d, $J=6.8$ Hz, H-Ar), 7.15 (2H, d, $J=6.7$ Hz, H-Ar), 7.40 (2H, d, $J=8.2$ Hz, H-Ar), 7.67 (2H, d, $J=8.2$ Hz, H-Ar) ppm; ¹³C NMR (100 MHz, DMSO): 21.14, 26.76, 28.26, 31.75, 34.97, 49.88, 57.67, 112.32, 119.52, 121.85, 128.04,

128.67, 130.16, 131.60, 143.83, 145.68, 147.49, 158.53, 162.64, 195.63 ppm;

C₂₅H₂₄N₂O₅S MS (ESI⁺): m/z 465 (M+H)⁺.

2-Amino-4-(2-chlorobenzoquinoline)-3-cyano-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile (4u)

IR (KBr): 3473, 3315, 2194, 1688, 1248 cm⁻¹; ¹H NMR (400 MHz, DMSO): 1.02 (3H, s, CH₃), 1.05 (3H, s, CH₃), 2.10 (1H, d, *J*=16.1 Hz, H-6), 2.26 (1H, d, *J*=16.1 Hz, H-6'), 2.51 (2H, s, H-8), 4.88 (1H, s, H-4), 7.20 (2H, br s, NH₂), 7.78 (2H, m, H-Ar), 7.91 (1H, d, *J*=8.9 Hz, H-Ar), 7.97 (2H, d, *J*=8.9 Hz, H-Ar), 8.01 (1H, m, H-Ar), 8.37 (1H, s, H-Ar), 8.99 (1H, s, H-Ar) ppm; ¹³C NMR (100 MHz, DMSO): 27.05, 28.23, 31.79, 49.88, 119.25, 123.58, 124.69, 125.65, 127.54, 128.12, 128.17, 128.82, 129.30, 143.94, 184.21, 158.77, 163.54, 195.88 ppm; C₂₅H₂₀ClN₃O₂ MS (ESI⁺): m/z 430 (M+H)⁺.

Results and discussion

Initially, 4-chlorobenzaldehyde was selected as a probe aldehyde to optimize the amount of catalyst. The formation of the (4b) did not proceed in the absence of the (dicaetoxyiodo)benzene, even after refluxing the reaction mixture for 6 hrs. The amount of catalyst was optimized during the reactions and it is observed that the 5 mol% of the catalyst was sufficient to progress the reaction (Table 1). Larger amounts of the catalyst did not improve the results to a greater extent. The results are summarized in Table 1. The various solvents were studied as a model experiment for 5 mole% of (dicaetoxyiodo)benzene. The results are summarized in Table 2. It has been observed that the alcohol-water as a solvent gives better results than other solvents.

Table 1. Optimization of the catalyst amount for the synthesis of (4b)^a

Entry	Catalyst (mol%)	Time (min.)	Yield (%) ^b
1	0	360	--
2	2.5	60	80
3	5.0	30	89
4	10.0	30	89

^aAll reactions were carried out under reflux conditions.

^bIsolated yields

Table 2. Optimization of solvent for the synthesis of (**4b**)^a under reflux conditions

Entry	Solvent	Time (min.)	Yield (%) ^b
1	Ethyl acetate	210	85
2	THF	300	67
3	Acetonitrile	90	56
4	Ethanol	60	89
5	Water	240	51
6	Ethanol-water (1:1)	30	89

^aAll reactions were carried out using 5 mol% of DIB.^bIsolated yields

From these results, other aromatic aldehydes have been reacted with dimedone, and malanonitrile in ethanol-water under reflux conditions. The results are summarized in Table 3. It was evident that several aromatic aldehydes converted to the corresponding products in high yields over the DIB catalyst. Benzaldehyde and other aromatic aldehydes containing electron-withdrawing groups (such as Nitro, halide, etc.), electron donating groups (such as hydroxyl, alkoxy) and heterocyclic aldehyde such as thiophene 2-carboxaldehyde were employed and reacted well to give the corresponding 4*H*-benzo-[*b*]-pyran derivatives in good to excellent yields. The solid products obtained were crystallized in ethanol and identified by melting point, IR, ¹H NMR, ¹³C NMR, and mass spectroscopy. In all the cases, the spectroscopic data completely matched the reported values in

the literature. ¹H NMR spectra of tetrahydrobenzo-[*b*]-pyrans showed characteristic peaks at δ = 4.08-4.88 ppm for H-4, two doublets for the two diastereotopic protons H-6 and H-6' at δ = 2.10-2.26 ppm, and two singlet peaks at δ = 0.96-1.07 ppm for two distereotopic methyl groups on C-7. Compound 4r shows peaks for nine protons in aromatic region, 4s showed one extra singlet for three protons at δ = 2.47 ppm and for 4u seven protons observed in aromatic region.

When malanonitrile, aromatic aldehydes and dimedone were treated with DIB in 1:1 aqueous ethanol under reflux condition (Scheme 1, Entries 1-17), the desired 2-amino-3-cyano-4-aryl-7,7-dimethyl-5-oxo-4*H*-5,6,7,8-tetrahydrobenzo-[*b*]-pyran derivatives 4a-4q were obtained in good to excellent yields (77-95%, Table 3) within a very short period of time (30-90 min). An

exception was about the compounds 4r-4u for which the reaction did not go to completion under those conditions. In 1:1 aqueous ethanol desired products 4r and 4s does not obtain. Due to the low solubility of pyrazole aldehyde (4t) and 2-chlorobenzoquinoline (4u) in aqueous ethanol, synthesis of 4t and 4u was carried out in ethanol. Therefore the reaction was conducted in refluxing alcohol for three hours to obtain the desired products. Due to low solubility and low reactivity of cyanoacetate (4v-4z) as compared with malanonitrile, the reactions were carried out in ethanol at reflux condition for 2 hours (Scheme 1, Entries 18-26).

A plausible mechanism for the formation of benzopyran is outlined in scheme 2. The reaction proceeds *via* the initial formation of olefin (4) from the Knoevenagel

condensation of aldehyde (1) and active methylene compound (2). Dimedone in solution is in enolic form ($K_{\text{enol}}/K_{\text{keto}}=20$) [27] that in the presence of (diacetoxyiodo)benzene could be converted to 5 with elimination of acetic acid [39]. This (5) could react with olefin (4) resulting in formation of intermediate (6) which subsequently cyclises to afford the desired product (7) after proton transfer and tautomerism.

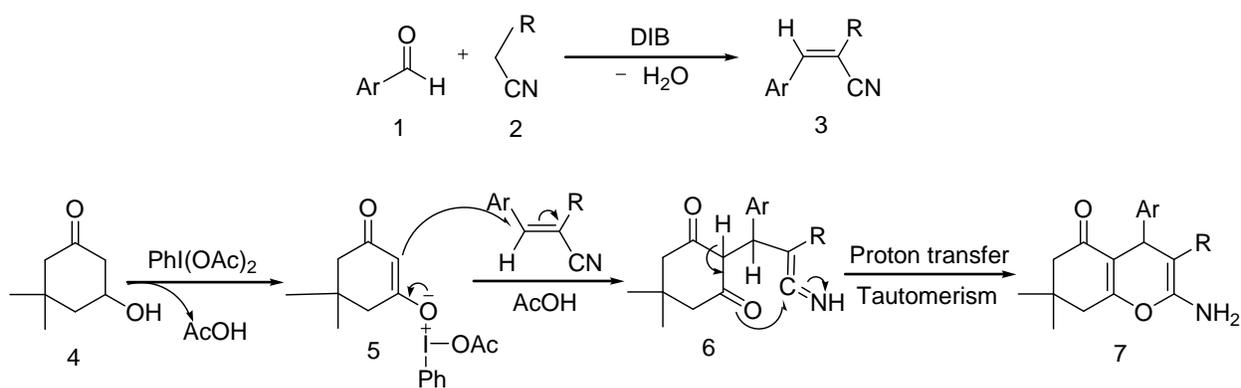
To show the advantage of the present method, a comparison of the efficiency of catalytic activity of the DIB with several methods is presented in table 4. It is observed that the present method is better than some of the earlier methods reported in terms of catalyst amount and reaction time.

Table 3. Synthesis of 5-Oxo-5,6,7,8-tetrahydro-4*H*- benzo-[*b*]-pyrans in the presence of 5 mol% DIB under reflux conditions

Entry	Ar- (1)	Product (4)	R	Time (min.)	Yield (%)	M.P. °C (Lit.)
1 ^a	C ₆ H ₅ -	4a	CN	60	83	228-230 (231-235)[22]
2 ^a	4-Cl C ₆ H ₄ -	4b	CN	30	89	198-200 (207-209)[17]
3 ^a	3-OH C ₆ H ₄ -	4c	CN	60	83	200-202 (227-228)[21]
4 ^a	4-OH C ₆ H ₄ -	4d	CN	60	77	200-202 (204-205)[22]
5 ^a	4-(MeO) C ₆ H ₄ -	4e	CN	60	85	194-196 (198-200)[17]
6 ^a	3,4,-(OCH ₃) ₂ C ₆ H ₃ -	4f	CN	60	80	174-176 (181-183)[17]
7 ^a	3,4,5-(OCH ₃) ₃ C ₆ H ₂ -	4g	CN	60	95	202-204 (208-210)[33]
8 ^a	3-NO ₂ C ₆ H ₄ -	4h	CN	30	95	200-202 (204-206)[33]

9^a	4-NO ₂ C ₆ H ₄ -	4i	CN	30	94	170-172 (177-178)[18]
10^a	2-Cl C ₆ H ₄ -	4j	CN	60	92	194-196 (191-192)[31]
11^a	3-Cl C ₆ H ₄ -	4k	CN	60	89	238-240 (232-234)[32]
12^a	4-Br C ₆ H ₄ -	4l	CN	30	86	208-210 (205-207)[33]
13^a	1-naphthyl	4m	CN	60	85	218-220 (214-215)[23]
14^a	2-thiophenyl	4n	CN	60	87	210-212 (210-112)[15]
15^a	4-OH,3-OMe C ₆ H ₃ -	4o	CN	90	88	240-242 (239-240)[17]
16^a	4-OH,3-OEt C ₆ H ₃ -	4p	CN	90	80	240-242 c
17^a	5-Br,4-OH,3-OMe C ₆ H ₂ -	4q	CN	60	86	258-260 c
18^b	4-(OCOPh) C ₆ H ₄ -	4r	CN	180	85	266-268 c
19^b	4-(OSO ₂ Ph) C ₆ H ₄ -	4s	CN	180	80	210-212 c
20^b	1,3-diphenyl-1 <i>H</i> -pyrazol-4-yl	4t	CN	180	90	202-204 (209-211)[9]
21^b	2-Cl benzoquinoline, 3-carbaldehyde	4u	CN	180	90	260-262 c
22^b	3-NO ₂ C ₆ H ₄ -	4v	CO ₂ Et	120	88	180-182 (182-184)[20]
23^b	4-NO ₂ C ₆ H ₄ -	4w	CO ₂ Et	120	94	186-188 (181-183)[20]
24^b	3,4- (OCH ₃) ₂ C ₆ H ₃ -	4x	CO ₂ Et	120	87	156-158 (155-157)[24]
25^b	4-Cl C ₆ H ₄ -	4y	CO ₂ Et	120	84	148-150 (150-152)[20]
26^b	4-Br C ₆ H ₄ -	4z	CO ₂ Et	120	86	162-164 (159-163)[22]

^aA: The reaction was conducted in H₂O/EtOH (1:1). ^bB: The reaction was conducted in EtOH. ^cNewly synthesized compound characterized by IR, ¹H NMR, ¹³C NMR and Mass spectra.



Scheme 2. Plausible mechanism for the synthesis of 2-amino-4*H*-tetrahydrobenzo-[b]-pyrans

Table 4. Catalytic activity and reaction conditions comparison of DIB with other reported catalysts

Entry	Product	Catalyst /Reaction condition	Time	Yield (%)	Ref.
1	4a	L-proline (10 mol%)/Ethanol/reflux	4.0 hr.	90	[28]
2	4a	KF-Al ₂ O ₃ /Ethanol/reflux	2.0 hr.	90	[21]
3	4a	RE(PFO) ₃ (5 mol%)/Ethanol/60°C	5.0 hr.	90	[29]
4	4a	I ₂ (10 mol%)/DMSO/reflux	3.2 hr.	92	[22]
5	4a	Trifluoroethanol/reflux	5.0 hr.	90	[31]
6	4a	DIB (5 mol%)/Ethanol-Water/reflux	1.0 hr.	83	This work
7	4h	KF-Al ₂ O ₃ /Ethanol/reflux	1.5 hr.	90	[21]
8	4h	RE(PFO) ₃ (5 mol%)/Ethanol/60°C	4:0 hr.	83	[29]
9	4h	I ₂ (10 mol%)/DMSO/reflux	4:0 hr.	86	[22]
10	4h	Trifluoroethanol/reflux	5.0 hr.	92	[31]
11	4h	DIB (5 mol%)/Ethanol-Water/reflux	0.5 hr.	95	This work

Conclusion

We have developed a three component one-pot protocol for the synthesis of benzopyran derivatives using a catalytic amount (diacetoxyiodo)benzene in aqueous ethanolic and ethanolic medium. This Knoevenagel-cyclocondensation of various aromatic and hetero-aromatic aldehydes along with the aldehydes like aryl-sulphonyloxybenzaldehyde, aryl-carbonyloxybenzaldehyde also leads to the product under these reaction conditions. The developed methodology is simple, novel and highly efficient, which afforded various tetrahydrobenzo-[b]-pyrans in good to excellent yields.

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