

Three-component synthesis of fused indenopyridines from aromatic aldehydes, 2-bromo-2*H*-indene-1,3-dione and aminouracil or aminopyrazole

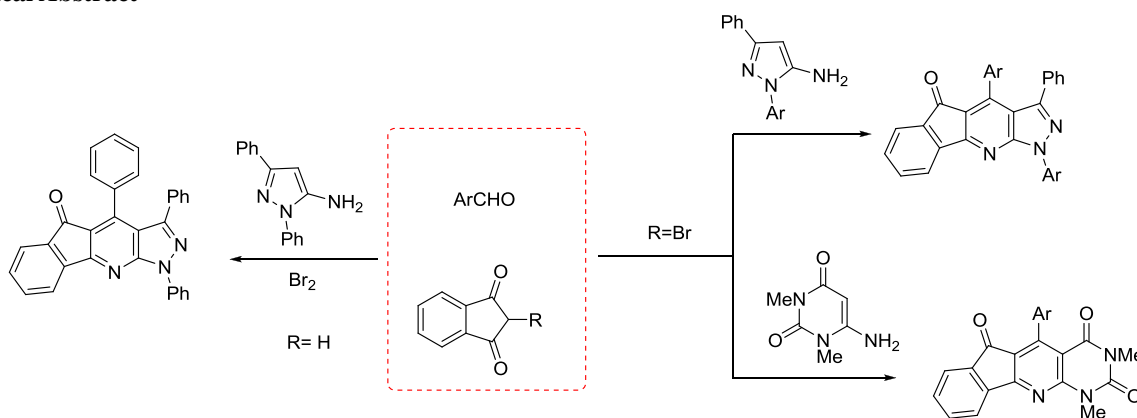
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Abstract Three-component synthesis of fused indenopyridines containing pyrazole or uracil moiety by the reaction of benzaldehydes, 2-bromo-1*H*-indene-1,3(2*H*)-dione, and pyrazoles or uracil, catalyzed by ammonium acetate is reported.

applications. These compounds have been reported to possess a wide spectrum of biologic activities such as anti-hyperglycemic, analgesic, anti-inflammatory, antipyretic, antibacterial, hypoglycemic, sedative-hypnotic properties [6–11]. In particular among heterocycle-fused pyrazoles,

Graphical Abstract



Keywords Indenopyridine · Pyrazole · Uracil · 2-Bromo-1*H*-indene-1,3(2*H*)-dione

Introduction

Pyrazoles have gained more attention in recent years for biological [1, 2], medicinal [3, 4], and agricultural [5]

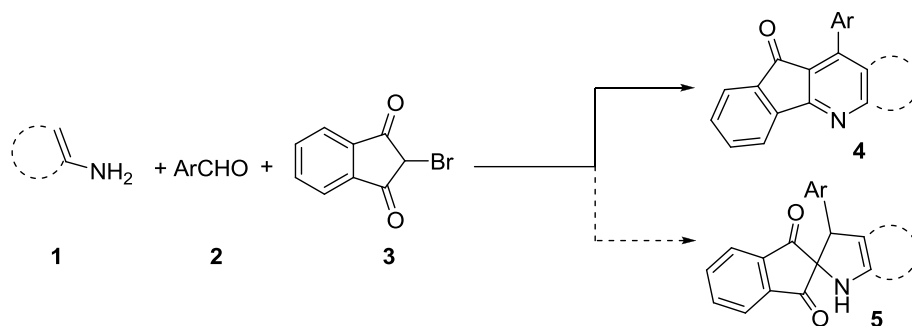
pyrazolopyridines are an important class of heterocycles with broad pharmacological activities such as possible anti-viral agent [12, 13], potent p38 kinase inhibitors [14], HIV reverse transcriptase inhibitors [15].

As a privileged scaffold, pyridine is a ubiquitous subunit in various products with remarkable biological activities such as antimicrobial [16–18], anticancer [19] anticonvulsant [20], antiviral [21], anti-HIV [22], antifungal, and antimycobacterial [23]. Particularly, among of this category, indenopyridines are attractive due to showing a wide range of biological activities such as calcium antagonistic [24], antioxidant [25], antihistamine, and antidepressant [26–28], and also act as phosphodiesterase (PDE) inhibitors [29], NK-1, and dopamine receptor ligands [30, 31].

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Scheme 1 Synthesis of indenopyridines containing pyrazole or uracil 4



Very recently, we described a modified reaction for the synthesis of spiro indenofuran-triones by the reaction of 1,3-dicarbonyl compounds, aldehydes, and cyclic α -halo ketones [32–35]. As a continuation of our previous work for heterocycles synthesis using cyclic α -halo ketones [32–35], we herein investigate replacement of enamine compounds instead of 1,3-dicarbonyl compounds in the reaction. Notably, the expected product **5** was not obtained, while indenopyridines containing pyrazole or uracil moiety **4** was isolated in good yields (Scheme 1).

Experimental

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. NMR spectra were recorded on a BRUKER DRX-300 AVANCE spectrometer. NMR spectra were obtained on solutions in DMSO- d_6 . IR spectra were recorded using a BOMEM MB-Series. Elemental analyses were performed using a Herecus CHN-O-Rapid analyzer. The chemicals used in this work were obtained from Fluka and Merck and were used without purification.

Due to very low solubility of the products **4**, we cannot report the ^{13}C NMR data for these products.

General procedure for preparation of 1,3-diphenylindeno[1,2-*b*]pyrazolo[4,3-*e*]pyridin-5(1*H*)-ones (4) A mixture of pyrazoles (1 mmol), aldehydes (1 mmol), 2-bromo-1*H*-indene-1,3(2*H*)-dione (1 mmol) and NH_4OAc (30 mol%) in refluxing acetic acid (2 mL) was stirred for 24 h. After completion of the reaction (confirmed by TLC), the reaction mixture was cooled to room temperature. Then, the precipitate was filtered and washed with methanol (5 mL) to afford the pure product **4**.

1,3,4-Triphenylindeno[1,2-*b*]pyrazolo[4,3-*e*]pyridin-5(1*H*)-one (4a) Yellow powder (71 %); mp > 270 °C. IR (KBr) ($\nu_{\text{max}}/\text{cm}^{-1}$): 1703, 1615, 1562. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 7.03 (4H, m, H-Ar), 7.12–7.28

(6H, m, H-Ar), 7.45–7.48 (1H, m, H-Ar), 7.59–7.74 (5H, m, H-Ar), 7.98–8.01 (1H, m, H-Ar), 8.33 (2H, d, $J = 7.6$ Hz, H-Ar). MS (EI, 70 eV) m/z : 449 (M^+). Anal. Calcd. for $\text{C}_{31}\text{H}_{19}\text{N}_3\text{O}$: C, 82.83; H, 4.26; N, 9.35. Found: C, 82.76; H, 4.22; N, 9.26.

4-(4-Nitrophenyl)-1,3-diphenylindeno[1,2-*b*]pyrazolo[4,3-*e*]pyridin-5(1*H*)-one (4b) Cream powder (77 %); mp > 270 °C. IR (KBr) ($\nu_{\text{max}}/\text{cm}^{-1}$): 1709, 1600, 1559. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 7.06–7.11 (4H, m, H-Ar), 7.21–7.23 (1H, m, H-Ar), 7.46–7.70 (7H, m, H-Ar), 7.77–7.82 (1H, m, H-Ar), 7.97–8.08 (3H, m, H-Ar), 8.34 (2H, d, $J = 8.1$ Hz, H-Ar). MS (EI, 70 eV) m/z : 494 (M^+). Anal. Calcd. for $\text{C}_{31}\text{H}_{18}\text{N}_4\text{O}_3$: C, 75.29; H, 3.67; N, 11.33. Found: C, 75.34; H, 3.59; N, 11.24.

4-(4-Bromophenyl)-1,3-diphenylindeno[1,2-*b*]pyrazolo[4,3-*e*]pyridin-5(1*H*)-one (4c) Yellow powder (65 %); mp 245–247 °C. IR (KBr) ($\nu_{\text{max}}/\text{cm}^{-1}$): 1709, 1603, 1554. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 7.01–7.07 (6H, m, H-Ar), 7.18–7.27 (3H, m, H-Ar), 7.31–7.44 (2H, m, H-Ar), 7.50–7.61 (5H, m, H-Ar), 7.96–7.98 (1H, m, H-Ar), 8.31 (2H, d, $J = 6.9$ Hz, H-Ar). MS (EI, 70 eV) m/z : 529 ($\text{M}^+ + 2$), 527 (M^+). Anal. Calcd. for $\text{C}_{31}\text{H}_{18}\text{BrN}_3\text{O}$: C, 70.46; H, 3.43; N, 7.95. Found: C, 70.38; H, 3.37; N, 7.90.

4-(4-Methoxyphenyl)-1,3-diphenylindeno[1,2-*b*]pyrazolo[4,3-*e*]pyridin-5(1*H*)-one (4d) Yellow powder (73 %); mp 205–207 °C. IR (KBr) ($\nu_{\text{max}}/\text{cm}^{-1}$): 1710, 1607, 1558. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 3.71 (3H, s, CH_3), 6.62 (2H, d, $J = 8.1$ Hz, H-Ar), 6.98–7.22 (7H, m, H-Ar), 7.44–7.70 (7H, m, H-Ar), 7.95 (1H, d, $J = 6.9$ Hz, H-Ar), 7. (1H, m, H-Ar), 8.31 (2H, d, $J = 7.8$ Hz, H-Ar). MS (EI, 70 eV) m/z : 479 (M^+). Anal. Calcd. for $\text{C}_{32}\text{H}_{21}\text{N}_3\text{O}_2$: C, 80.15; H, 4.41; N, 8.76. Found: C, 80.24; H, 4.47; N, 8.38.

1-(4-Methoxyphenyl)-3,4-diphenylindeno[1,2-b]pyrazolo[4,3-e]pyridin-5(1H)-one (4e) Yellow powder (68 %); mp > 270 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1704. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 3.86 (3H, s, CH₃), 7.03–7.32 (12H, m, H-Ar), 7.56–7.61 (2H, m, H-Ar), 7.66–7.77 (1H, m, H-Ar), 7.98 (1H, d, $J = 7.5$ Hz, H-Ar), 8.17 (2H, d, $J = 8.8$ Hz, H-Ar). MS (EI, 70 eV) m/z : 449 (M^+). Anal. Calcd. for C₃₂H₂₁N₃O₂: C, 80.15; H, 4.41; N, 8.76. Found: C, 80.08; H, 4.36; N, 8.70.

1-(4-Methoxyphenyl)-4-(4-nitrophenyl)-3-phenylindeno[1,2-b]pyrazolo[4,3-e]pyridin-5(1H)-one (4f) Yellow powder (69 %); mp 262–264 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1708, 1561, 1510. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 3.85 (3H, s, CH₃), 6.97–7.08 (4H, m, H-Ar), 7.17–7.22 (3H, m, H-Ar), 7.47–7.61 (4H, m, H-Ar), 7.71–7.76 (1H, m, H-Ar), 7.93–7.98 (3H, m, H-Ar), 8.15 (2H, d, $J = 8.8$ Hz, H-Ar). MS (EI, 70 eV) m/z : 524 (M^+). Anal. Calcd. for C₃₂H₂₀N₄O₄: C, 73.27; H, 3.84; N, 10.68. Found: C, 73.18; H, 3.93; N, 10.72.

4-(4-Chlorophenyl)-1-(4-methoxyphenyl)-3-phenylindeno[1,2-b]pyrazolo[4,3-e]pyridin-5(1H)-one (4g) Yellow powder (66 %); mp 243–245 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1705, 1563, 1505. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 3.86 (3H, s, CH₃), 6.92 (1H, bs, H-Ar), 7.01–7.26 (10H, m, H-Ar), 7.56–7.61 (2H, m, H-Ar), 7.71–7.75 (1H, m, H-Ar), 7.95 (1H, d, $J = 7.4$ Hz, H-Ar), 8.15 (1H, d, $J = 8.9$ Hz, H-Ar). MS (EI, 70 eV) m/z : 513 (M^+). Anal. Calcd. for C₃₂H₂₀ClN₃O₂: C, 74.78; H, 3.92; N, 8.18. Found: C, 74.73; H, 3.85; N, 8.27.

1-(4-Bromophenyl)-4-(4-nitrophenyl)-3-phenylindeno[1,2-b]pyrazolo[4,3-e]pyridin-5(1H)-one (4h) Yellow powder (80 %); mp > 270 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1711, 1561, 1498. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 7.04–7.09 (4H, m, H-Ar), 7.20–7.22 (1H, m, H-Ar), 7.48–7.61 (4H, m, H-Ar), 7.74–7.83 (3H, m, H-Ar), 7.93–8.04 (3H, m, H-Ar), 8.34 (2H, d, $J = 8.3$ Hz, H-Ar). MS (EI, 70 eV) m/z : 574 ($\text{M}^+ + 2$), 572 (M^+). Anal. Calcd. for C₃₂H₂₀ClN₃O₂: C, 64.93; H, 2.99; N, 9.77. Found: C, 64.83; H, 3.07; N, 9.72.

1-(4-Bromophenyl)-4-(4-chlorophenyl)-3-phenylindeno[1,2-b]pyrazolo[4,3-e]pyridin-5(1H)-one (4i) Yellow powder (50 %); mp 252–253 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1706, 1561, 1491. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 6.95–7.17 (9H, m, H-Ar), 7.44–7.64 (6H, m, H-Ar), 7.95 (1H, d, $J = 7.5$ Hz, H-Ar), 8.27 (2H, d, $J = 8.1$ Hz, H-Ar). MS (EI, 70 eV) m/z : 563 ($\text{M}^+ + 2$), 561 (M^+). Anal. Calcd.

for C₃₁H₁₇BrClN₃O: C, 66.15; H, 3.04; N, 7.47. Found: C, 66.09; H, 3.10; N, 7.40.

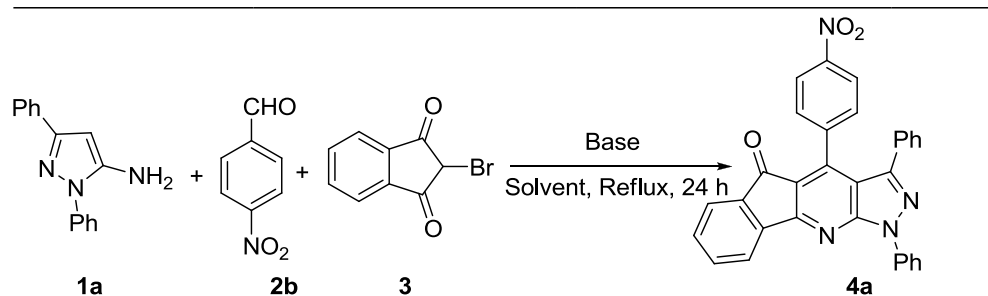
1-(4-Bromophenyl)-3-phenyl-4-(p-tolyl)indeno[1,2-b]pyrazolo[4,3-e]pyridin-5(1H)-one (4j) Yellow powder (70 %); mp 245–247 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1711, 1577, 1492. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 2.26 (3H, s, CH₃), 6.91–7.21 (8H, m, H-Ar), 7.58 (2H, bs, H-Ar), 7.80 (3H, m, H-Ar), 7.98 (1H, bs, H-Ar), 8.32 (2H, bs, H-Ar). MS (EI, 70 eV) m/z : 543 ($\text{M}^+ + 2$), 541 (M^+). Anal. Calcd. for C₃₂H₂₀BrN₃O: C, 70.86; H, 3.72; N, 7.75. Found: C, 70.78; H, 3.78; N, 7.84.

1-(4-Bromophenyl)-3-phenyl-4-(thiophen-2-yl)indeno[1,2-b]pyrazolo[4,3-e]pyridin-5(1H)-one (4k) Yellow powder (73 %); mp 267–269 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1706, 1558, 1492. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 6.84–6.97 (2H, m, H-Ar), 7.12–7.28 (5H, m, H-Ar), 7.54–7.77 (7H, m, H-Ar), 7.87–7.91 (1H, m, H-Ar). MS (EI, 70 eV) m/z : 535 ($\text{M}^+ + 2$), 533 (M^+). Anal. Calcd. for C₂₉H₁₆BrN₃OS: C, 65.17; H, 3.02; N, 7.86. Found: C, 65.08; H, 3.10; N, 7.81.

1,3-Dimethyl-5-phenyl-1H-indeno[2',1':5,6]pyrido[2,3-d]pyrimidine-2,4,6(3H)-trione (4m) Cream powder (95 %); mp > 270 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1701, 1685, 1604. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 3.14 (3H, s, CH₃), 3.75 (3H, s, CH₃), 7.22–7.39 (4H, m, H-Ar), 7.49–7.66 (3H, m, H-Ar), 7.75–7.80 (1H, m, H-Ar), 7.98 (1H, d, $J = 7.1$ Hz, H-Ar). MS (EI, 70 eV) m/z : 369 (M^+). Anal. Calcd. for C₂₂H₁₅N₃O₃: C, 71.54; H, 4.09; N, 11.38. Found: C, 71.44; H, 4.01; N, 11.30.

1,3-Dimethyl-5-(4-nitrophenyl)-1H-indeno[2',1':5,6]pyrido[2,3-d]pyrimidine-2,4,6(3H)-trione (4n) Yellow powder (92 %); mp > 270 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1705, 1689, 1611. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 3.15 (3H, s, CH₃), 3.73 (3H, s, CH₃), 7.53–7.66 (4H, m, H-Ar), 7.75–7.80 (1H, m, H-Ar), 7.98 (1H, d, $J = 7.3$ Hz, H-Ar), 8.29 (2H, d, $J = 8.5$ Hz, H-Ar). MS (EI, 70 eV) m/z : 414 (M^+). Anal. Calcd. for C₂₂H₁₄N₃O₅: C, 63.77; H, 3.41; N, 13.52. Found: C, 63.68; H, 3.33; N, 13.63.

5-(4-Methoxyphenyl)-1,3-dimethyl-1H-indeno[2',1':5,6]pyrido[2,3-d]pyrimidine-2,4,6(3H)-trione (4o) Cream powder (87 %); mp > 270 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1706. ^1H NMR (300 MHz, DMSO- d_6): δ_{H} 3.14 (3H, s, CH₃), 3.74 (3H, s, CH₃), 3.83 (3H, s, CH₃), 6.93 (2H, d, $J = 8.5$ Hz,

Table 1 Optimization of the reaction


Entry	Solvent, reflux	Base (mol%)	Yield (%) ^a
1	CH ₃ CN	NH ₄ OAc (30)	45
2	EtOH	NH ₄ OAc (30)	40
3	H ₂ O	NH ₄ OAc (30)	43
4	HOAc	DBU (30)	70
5	HOAc	K ₂ CO ₃ (30)	65
6	HOAc	CsCO ₃ (30)	71
7	HOAc	NaOAc (30)	70
8	HOAc	NH ₄ OAc (30)	77
9	HOAc	NH ₄ OAc (20)	69
10	HOAc	NH ₄ OAc (40)	76
11	HOAc	–	<30

Pyrazol-5-amine **1a** (1 mmol), 4-nitrobenzaldehyde **2b** (1 mmol) and indene-1,3(2H)-dione **3** (1 mmol)

^a Isolated yield

H-Ar), 7.17 (2H, d, $J = 8.5$ Hz, H-Ar), 7.61–7.79 (3H, m, H-Ar), 7.97 (1H, d, $J = 7.7$ Hz, H-Ar). MS (EI, 70 eV) m/z : 399 (M^+). Anal. Calcd. for C₂₃H₁₇N₃O₄: C, 69.17; H, 4.29; N, 10.52. Found: C, 69.10; H, 4.23; N, 10.61.

1,3,4-Triphenyl-4,10-dihydroindeno[1,2-b]pyrazolo[4,3-e]pyridin-5(1H)-one (7) White powder (66 %); mp > 270 °C. IR (KBr) ($\nu_{\max}/\text{cm}^{-1}$): 1704, 1601, 1543. ¹H NMR (300 MHz, DMSO-*d*₆): δ_{H} 5.67(1H, s, CH), 7.09 (1H, m, H-Ar), 7.24–7.43 (5H, m, H-Ar), 7.52–7.65 (7H, m, H-Ar), 7.77 (3H, m, H-Ar), 7.99–8.01 (3H, m, H-Ar), 11.05 (1H, s, NH). MS (EI, 70 eV) m/z : 451 (M^+). Anal. Calcd. for C₃₁H₂₁N₃O: C, 82.46; H, 4.69; N, 9.31. Found: C, 82.40; H, 4.62; N, 9.25.

Results and discussion

To achieve suitable conditions for the synthesis of indenopyridines **4**, various reaction conditions and bases have been investigated in the reaction of pyrazol-5-amine **1a**, 4-nitrobenzaldehyde **2b**, and indene-1,3(2H)-dione **3** as a model reaction (Table 1). Screening of the solvent revealed that acetic acid is the most suitable reaction media,

providing indeno[1,2-b]pyrazolo[4,3-e]pyridin-5(1H)-one **4b** in 77 % yield in the presence of 30 mol % of NH₄OAc (entry 8). It was found that when increasing the amount of NH₄OAc from 20 to 30 mol%, the isolated yield increased from 69 to 77 %, respectively (entries 8 and 9), and more amounts of the NH₄OAc did not improve the yield (entry 10). It should be mentioned that when the reaction was carried out in the absence of NH₄OAc, the yield of the product was low (entry 11). When this reaction was carried out with other bases such as DBU, NaOAc, CsCO₃, or K₂CO₃, the yield of the expected product was reduced.

According to the optimized conditions, the three-component condensation reaction of 1,3-diphenyl-1H-pyrazol-5-amines **1a–c** or uracil **1d**, aldehydes **2**, and 2-bromo-1H-indene-1,3(2H)-dione **3** in the presence of NH₄OAc (30 mol%) proceeded in HOAc under reflux conditions to afford indenopyridines containing pyrazole or uracil moiety diones **4**, in good yields after 24 h (Table 2). This protocol followed the GAP chemistry [36–39], which can avoid traditional chromatography and recrystallization purification methods. According GAP chemistry, pure products were obtained simply by filtration and washing off the solid with methanol.

To demonstrate the key role of 2-bromo-1H-indene-1,3(2H)-dione **3** in the reaction, we investigated the reaction of 1,3-indandione **6** with 1H-pyrazol-5-amine **1a** and

Table 2 Indenopyridines consist of pyrazole or uracil **4**

Product	X	y	Yield (%)
4a	H	H	71
4b	H	NO ₂	77
4c	H	Br	65
4d	H	OMe	73
4e	OMe	H	68
4f	OMe	NO ₂	69
4g	OMe	Cl	66
4h	Br	NO ₂	80
4i	Br	Cl	50
4g	Br	Me	70
4k	Br	Thiophene	73
4m	—	H	95
4n	—	NO ₂	92
4o	—	OMe	87

4-nitrobenzaldehyde **2b** in the same reaction conditions (Scheme 2). The indenopyrazolopyridin-5(1*H*)-one **4a** was not detected at all, while 4-(4-nitrophenyl)-1,3-diphenyl-4,10-dihydroindeno[1,2-*b*]pyrazolo[4,3-*e*]pyridin-5(1*H*)-one **7** was obtained in 66 % yield. In the HNMR spectrum of **7**, the benzylic proton is observed as a singlet at 5.67 ppm which confirms the proposed structure of **7**.

Although the exact mechanism of the reaction is not very clear, a plausible mechanism for the formation of product **4** is proposed in Scheme 3. Initially, nucleophilic reaction of aminopyrazole **1** to iminium salt **8** (formed in situ by the reaction of amine and aldehyde), followed by acid–base reaction produced intermediate **9**. Then, nucleophilic reaction of 2-bromo-1*H*-indene-1,3(2*H*)-dione **3** to intermediate **9** followed by intracyclization afforded intermediate **10**.

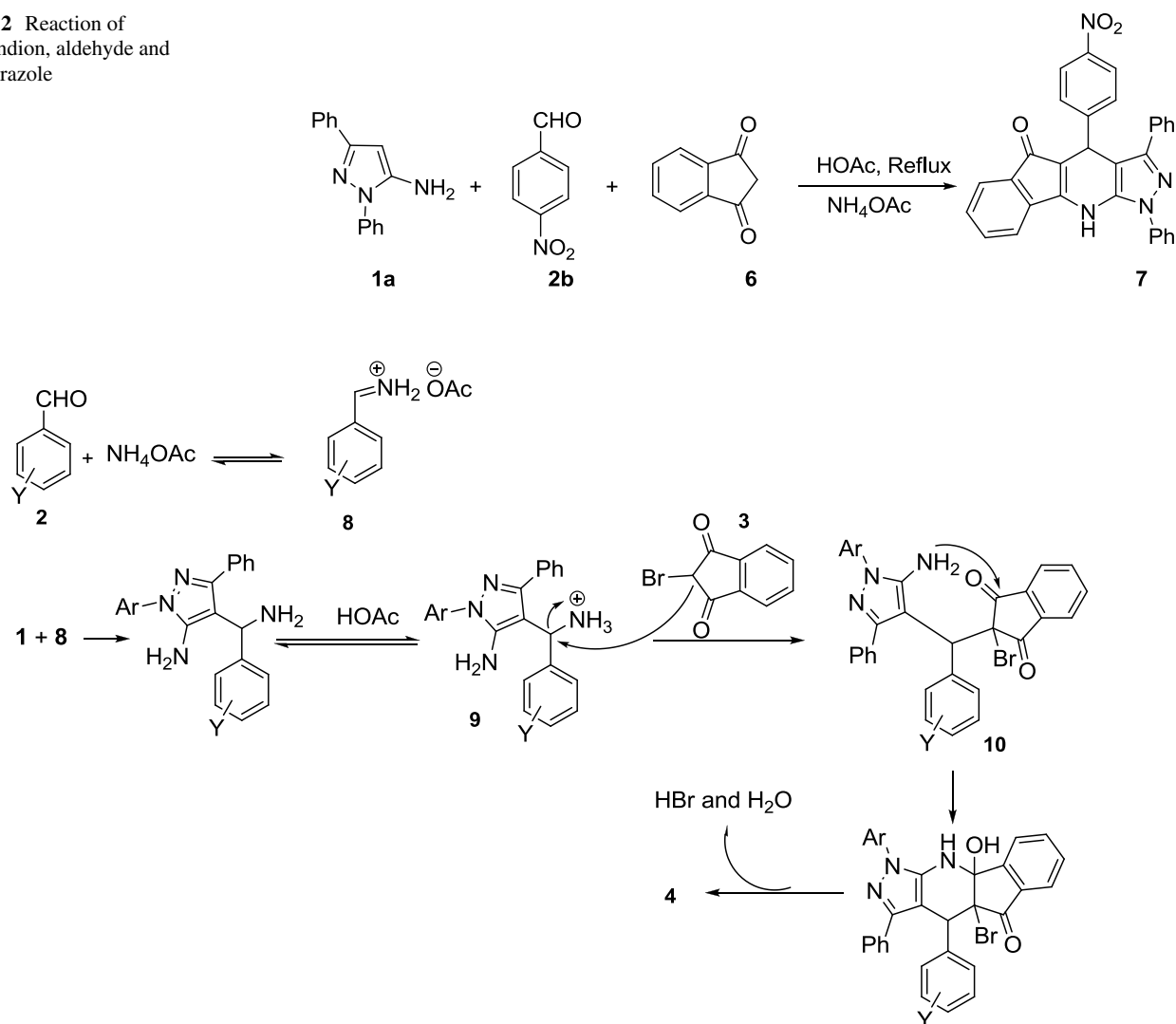
Eventually, elimination of HBr and H₂O from **10** produced the corresponding product **4**.

Finally, in situ generation of 2-bromo-1*H*-indene-1,3(2*H*)-dione **3** by the reaction of 1,3-indandione **6** and bromine was investigated in the reaction. Therefore, the reaction of 1,3-diphenyl-1*H*-pyrazol-5-amine **1a**, 4-nitro aldehyde **2b**, 1,3-indandione **6** and bromine under same reaction conditions afforded the same product **4** in 65 % yield after 24 h (Scheme 4).

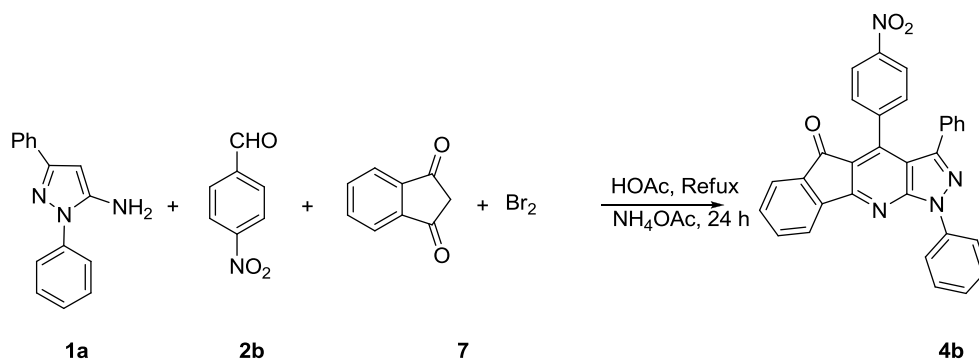
Conclusion

In this paper, 2-bromo-1*H*-indene-1,3(2*H*)-dione as a cyclic α -halo compound was used for the synthesis of

Scheme 2 Reaction of 1,3-indandione, aldehyde and aminopyrazole



Scheme 3 Proposed mechanism



Scheme 4 In situ generation of 2-bromoindene-dione **3** in the reaction

indenopyridines containing densely pyrazole or uracil in good yields via multi-component coupling reactions. Prominent among the advantages of this new method are novelty, operational simplicity, good yields, and easy work-up procedures employed.

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