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$InCl_3$-catalyzed efficient synthesis of spiro-perimidine derivatives

Zahra Yasaei, Peiman Mirzaei, Ayoob Bazgir *

Department of Chemistry, Shahid Beheshti University, G.C, PO Box 19396, 4716 Tehran, Iran

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ABSTRACT

$InCl_3$ was found to be a mild and effective catalyst for the simple and efficient synthesis of spiro-perimidine derivatives by the reaction of naphthalene-1,8-diamine and active carbonyl compounds in water at room temperature. This protocol includes some important aspects like the use of water as a "green" reaction medium, good yielding of products and mild reaction conditions.

1. Introduction

Spirocyclic systems containing one carbon atom common to two rings are structurally interesting [1,2]. The presence of the sterically constrained spiro structure in various natural products also adds to the interest in the investigations of spiro compounds [3]. Spiro compounds represent an important class of naturally occurring substances characterised by their highly pronounced biological properties [4,5]. Perimidines (peri-naphtho-fused pyrimidine systems) are an important class of heterocyclic compounds, since many of this heterocyclic system exhibit biological and pharmaceutical activity [6]. The interest in pyrido-fused perimidine derivatives stems from the appearance of these heterocyclic systems in many biologically active compounds [6,7]. Considering the important biological properties of perimidines, a number of methods have been reported for the synthesis of these heterocycles [6–10]. However, the preparation of perimidine derivatives using naphthalene-1,8-diamine with ketones has not been much developed. To our knowledge, in several methods, protonic acids have been used for this transformation as catalysts [11]. However, their strong acidity led to the occurrence of side reactions and substrates. Reddy and Rao reported synthesis of 2-methyl-2-aryl-2,3-dihydro-1*H*-perimidines using naphthalene-1,8-diamine with aromatic ketones in acetic acid or methanol with moderate product yields (60 %) [11c]. The methods of acid-catalyzed reactions are not very efficient. Therefore, there is a need for a new efficient catalyst for this organic transformation. Very recently, synthesis of perimidine derivatives has been reported using catalytic amounts of $BiCl_3$ [12], $RuCl_3$ [13] and $Yb(OTf)_3$ [14].

In recent years, indium (III) chloride ($InCl_3$) has received considerable attention as an inexpensive, non-toxic, readily available catalyst for various transformations under mild and convenient conditions, affording the corresponding products in excellent yields with high selectivity [15].

Due to the biological activity of perimidine derivatives, and our interest in the synthesis of heterocyclic compounds [16–31], herein, we report a simple and efficient method for the preparation of spiro-perimidines in water. In fact, as clearly stated by Sheldon [32], it is generally recognized that "the best solvent is no solvent and if a solvent (diluent) is needed it should preferably be water". The use of water as the reaction medium represents a remarkable benefit since this green solvent is highly polar and therefore immiscible with most organic compounds; moreover, the water soluble catalyst resides and operates in the aqueous phase and separation of the organic materials is thus easy.

* Corresponding author.
E-mail address: a_bazgir@sbu.ac.ir (A. Bazgir).

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2. Results and discussion

To achieve suitable catalyst and solvent for the synthesis of spiro-perimidines, various solvent and catalysts have been investigated in the reaction of naphthalene-1,8-diamine **1** and isatin **2a** as a model reaction at room temperature (Fig. 4). As could be seen in Fig. 4, the best result was obtained with a 20 mol % of $InCl_3$ as the catalyst in water at room temperature (Fig. 4). Using a lower amount of catalyst resulted in lower yields, while a higher amount of catalyst did not affect reaction times and yields (Fig. 4). When this reaction was carried out without any catalyst, TLC and ^1H NMR spectra of the reaction mixture showed a combination of starting materials and numerous products, the yield of the expected product was very poor (Fig. 4, entry 5). Fig. 4 demonstrates that water was the best choice of solvent and the use of $InCl_3$ in water improved the yield of the product.

Encouraged by this success, we extended the reaction of naphthalene-1,8-diamine **1** with various isatins **2a-h** under similar conditions ($H_2O/InCl_3$), furnishing the respective spiro[indoline-3,2′-perimidin]-2-ones **3a-h** in good yields. The optimized results are summarized in Fig. 5.

Table 1 compares efficiency of $InCl_3$ (time, yield, reaction conditions) with the efficiency of other catalysts in the synthesis of perimidine derivatives obtained by other catalysts. It is clear from Fig. 5 that $InCl_3$ is comparable to the catalysts used previously for the synthesis of perimidines.

As expected, when the isatin **2** was replaced by acenaphthylene-1,2-dione **4**, 1′,3′-dihydro-2*H*-spiro[acenaphthylene-1,2′-perimidin]-2-one **5** was obtained in 78 % yield under the same reaction conditions (Fig. 1).

To further explore the potential of this protocol for perimidines synthesis, we investigated reaction of naphthalene-1,8-diamine **1** with 2-indanone **6** or pyrimidine-tetraone **7** and obtained tetrahydrospiroindene-perimidine **8** and 1,3-dihydro-1′*H*-spiro[perimidine-2,5′-pyrimidine]-2′,4′,6′(3′*H*)-trione **9** in excellent yields (Fig. 2).

When the piperidin-4-one **10**, cyclododecanone **11** and 11*H*-indeno[2,1-*b*]quinoxalin-11-one **12** was selected as active carbonyl compounds (Fig. 3), the desired spiro-perimidines **13**, **14** and **15** were obtained in good yields.

When this reaction was carried out with ninhydrine **16**, benzoquinone **17** and naphthoquinone **18** in same conditions ($H_2O/InCl_3$), TLC and ^1H NMR spectra of the reaction mixture showed a combination of starting materials and numerous products, the yield of the expected product was very poor.

Table 1
Comparison of efficiency of various catalysts in synthesis of perimidines.

Catalyst	Conditions	Yield (%)	Time (h)	Ref.
$InCl_3$	H_2O/r.t.	77–91	3–4	This work
$BiCl_3$	EtOH/r.t	79–92	0.5–12	[12]
$RuCl_3$	EtOH/r.t	78–91	0.5–24	[13]
$Yb(OTf)_3$	EtOH/r.t	75–93	0.5–32	[14]

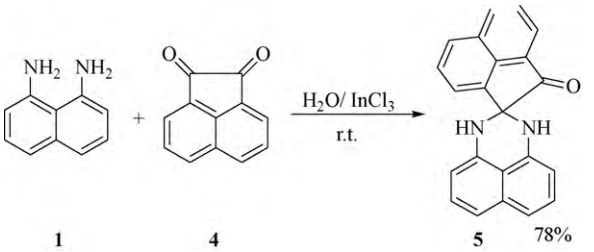

Fig. 1. .

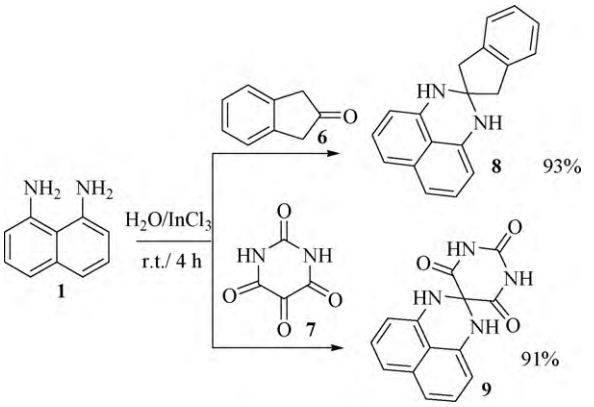

Fig. 2. .

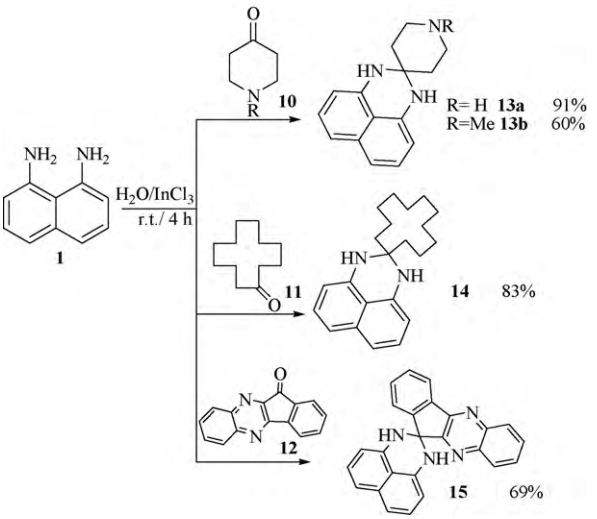

Fig. 3. .

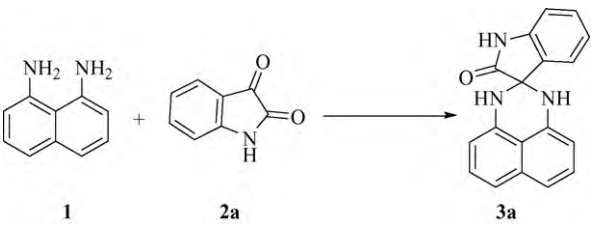

Fig. 4. Effect of reaction conditions.

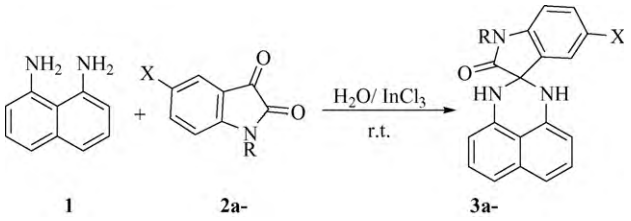

Fig. 5. Synthesis of spiro[indoline-3,2′-perimidin]-2-ones **3**.

All synthesized compounds are stable solids whose structures are fully supported by IR, ^{1}H and ^{13}C NMR spectroscopy, mass spectrometry, and elemental analysis.

3. Conclusions

In conclusion, $InCl_3$ has been found to be an efficient catalyst for the preparation of spiro-perimidine derivatives via a cyclo-condensation reaction in water. This protocol includes some important aspects like the use of water as a "green" reaction medium, high atom economy and mild reaction conditions.

4. Experimental

4.1. Materials and techniques

Melting points were taken on an Electro-thermal 9100 apparatus and left uncorrected. IR spectra were obtained on a Shimadzu IR-470 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on a BRUKER DRX-300 AVANCE spectrometer at 300.13 and 75.47 MHz. NMR spectra were obtained on solutions in DMSO using TMS as internal standard. All of the chemicals were purchased from Fluka, Merck and Aldrich and used without purification.

4.2. General procedure for the preparation of spiro-perimidines

A mixture of naphthalene-1,8-diamine (1 mmol), active carbonyl compounds (1 mmol) and $InCl_3$ (20 mol %) in water (5 ml) was stirred at room temperature for appropriate time (the progress of reaction was monitored by TLC). After completion of the reaction, the reaction mixture was filtered and the precipitate washed with water and then ether to afford the pure product.

*4.2.1. 1′,3′-Dihydrospiro[indoline-3,2′-perimidin]-2-one **3a***

White powder; m.p. 244–246 °C. IR (KBr): 3361, 3211, 1722, 1597/cm. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 6.44–7.35 (12H, m, H-Ar and 2NH), 10.24 (1H, s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): 67.9, 105.5, 110.2, 111.8, 116.0, 122.3, 125.8, 127.2, 130.2, 130.8, 133.9, 141.0, 143.0, 177.9. MS, m/z: 287 (M^+). Analytically calculated for $C_{18}H_{13}N_3O$: C 75.25; H 4.56; N 14.63 %. Found: C 75.16; H 4.51; N 14.55 %.

*4.2.2. 5-Bromo-1′,3′-dihydrospiro[indoline-3,2′-perimidin]-2-one **3b***

Cream powder; m.p. 236 °C (dec). IR (KBr): 3362, 3047, 1723, 1599/cm. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 6.43–7.34 (11H, m, H-Ar and 2NH), 10.24 (1H, s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ = 67.9, 105.4, 110.1, 111.8, 116.0, 122.3, 125.7, 127.2, 130.2, 130.8, 133.9, 141.0, 143.0, 177.9. MS, m/z: 367 (M^+), 365 (M^+). Analytically calculated for $C_{18}H_{12}BrN_3O$: C 59.03; H 3.30; N 11.47 %. Found: C 59.9; H 3.24; N 11.40 %.

*4.2.3. 5-Nitro-1′,3′-dihydrospiro[indoline-3,2′-perimidin]-2-one **3c***

Light brown powder; m.p. 263 °C (dec). IR (KBr): 3345, 3054, 2917, 1745, 1624/cm. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 6.47–8.33 (11H, m, H-Ar and 2NH), 11.02 (1H, s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ = 67.7, 106.0, 110.6, 111.8, 116.7, 120.9, 127.3, 127.9, 131.1, 133.9, 140.1, 142.6, 149.6, 178.0. MS, m/z: 332 (M^+). Analytically calculated for $C_{18}H_{12}N_4O_3$: C 65.06; H 3.64; N 16.86 %. Found: C 65.15; H 3.71; N 16.93 %.

*4.2.4. 1-Methyl-1′,3′-dihydrospiro[indoline-3,2′-perimidin]-2-one **3d***

Light pink powder; m.p. 281 °C (dec). IR (KBr): 3356, 3069, 2917, 1705, 1603/cm. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 3.10 (3H, s, CH_3), 6.43–7.46 (12H, m, H-Ar and 2NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ = 26.1, 67.8, 105.5, 109.0, 111.8, 116.1, 123.0, 125.3, 127.2, 129.5, 130.9, 133.9, 140.8, 144.4, 175.9. MS, m/z: 301 (M^+). Analytically calculated for $C_{19}H_{15}N_3O$: C 75.73; H 5.02; N 13.94 %. Found: C 75.62; H 5.07; N 13.86 %.

*4.2.5. 1-Ethyl-1′,3′-dihydrospiro[indoline-3,2′-perimidin]-2-one **3e***

Brown powder; m.p. 250–252 °C. IR (KBr): 3374, 3048, 1707, 1598/cm. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 1.15 (3H, bs, CH_3), 3.60 (2H, bs, CH_2), 6.43–7.44 (12H, m, H-Ar and 2NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ = 12.9, 34.2, 67.7, 105.5, 109.1, 111.8, 116.1, 122.8, 125.5, 127.2, 129.7, 130.9, 133.9, 140.9, 143.5, 175.5. MS, m/z: 315 (M^+). Analytically calculated for $C_{20}H_{17}N_3O$: C 76.17; H 5.43; N 13.32 %. Found: C 76.10; H 5.38; N 13.41 %.

*4.2.6. 1-Benzyl-1′,3′-dihydrospiro[indoline-3,2′-perimidin]-2-one **3f***

Dark pink powder; m.p. 168 °C. IR (KBr): 3332, 1750, 1682, 1587/cm. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 4.79 (2H, s, CH_2), 6.47–7.35 (17H, m, H-Ar and 2NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ = 42.8, 67.8, 105.6, 109.7, 111.8, 116.2, 123.1, 125.6, 127.2, 127.7, 129.0, 129.5, 130.8, 133.9, 136.5, 140.8, 143.5, 176.0. MS, m/z: 377 (M^+). Analytically calculated for $C_{25}H_{19}N_3O$: C 79.55; H 5.07; N 11.13 %. Found: C 79.66; H 5.14; N 11.08 %.

*4.2.7. 5-Bromo-1-methyl-1′,3′-dihydrospiro[indoline-3,2′-perimidin]-2-one **3g***

Dark brown powder; m.p. 265–258 °C (dec). IR (KBr): 3343, 1693, 1603/cm. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 3.05 (3H, s, CH_3), 6.42–7.64 (11H, m, H-Ar and 2NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ = 26.3, 67.8, 105.7, 111.2, 111.8, 114.6, 116.4, 126.8, 127.2, 131.8, 133.5, 133.9, 140.4, 143.7, 175.3. MS, m/z: 381 (M^+), 379 (M^+). Analytically calculated for $C_{19}H_{14}BrN_3O$: C 60.02; H 3.71; N 11.05 %. Found: C 60.10; H 3.77; N 12.99 %.

4.2.8. 1-Methyl-5-nitro-1′,3′-dihydrospiro[indoline-3,2′-perimidin]-2-one **3h**

Dark yellow powder; m.p. 274 °C (dec). IR (KBr): 3347, 3043, 2927, 1728, 1605/cm. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 3.15 (3H, s, CH_3), 6.46–8.43 (11H, m, H-Ar and 2NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ = 26.7, 67.4, 106.1, 109.5, 116.9, 120.3, 127.3, 128.0, 130.5, 133.6, 133.9, 140.0, 143.1, 150.6, 176.2. MS, m/z: 346 (M^+). Analytically calculated for $C_{19}H_{14}N_4O_3$: C 65.89; H 4.07; N 16.18 %. Found: C 65.81; H 4.15; N 16.11 %.

4.2.9. 1′,3′-Dihydro-2H-spiro[acenaphthylene-1,2′-perimidin]-2-one **5**

Brown powder; m.p. 279 °C (dec). IR (KBr): 3450, 3043, 2933, 1677, 1629/m. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 7.27–8.82 (14H, m, H-Ar and 2NH). MS, m/z: 322 (M^+). Analytically calculated for $C_{22}H_{14}N_2O$: C 81.97; H 4.38; N 8.69 %. Found: C 81.88; H 4.31; N 8.77 %.

Due to very low solubility of the product **5**, we cannot report the ^{13}C NMR data for this product.

4.2.10. 1,1′,3,3′-Tetrahydrospiro[indene-2,2′-perimidine] **8**

Light pink powder; m.p. 282 °C (dec). IR (KBr): 3339, 3038, 2938, 1597/cm. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 3.12 (4H, s, $2CH_2$), 6.42–7.18 (12H, m, H-Ar and 2NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ = 46.5, 74.9, 105.2, 113.0, 115.4, 125.3, 126.9, 127.4, 134.8, 140.7, 142.4. MS, m/z: 272 (M^+). Analytically calculated for $C_{19}H_{16}N_2$: C 83.79; H 5.92; N 10.29 %. Found: C 83.72; H 5.86; N 10.35 %.

4.2.11. 1,3-Dihydro-1′H-spiro[perimidine-2,5′-pyrimidine]-2′,4′,6′(3′H)-trione **9**

Brown powder; m.p. 201 °C (dec). IR (KBr): 3412, 3327, 2927, 1754, 1687 cm^{-1}. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 6.78-7.59 (8H, m, H-Ar and 2NH), 9.46 (2H, bs, 2NH). MS, m/z: 282 (M^+). Analytically calculated for $C_{14}H_{10}N_4O_3$: C 59.57; H 3.57; N 19.85 %. Found: C 59.50; H 3.51; N 19.79 %.

Due to very low solubility of the product **9**, we cannot report the ^{13}C NMR data for this product.

4.2.12. 1,3-Dihydrospiro[perimidine-2,4′-piperidine] **13a**

Light pink powder; m.p. 282 °C (dec). IR (KBr): 3364, 3206, 2938, 1599/cm. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 1.87 (4H, bs, $2CH_2$), 3.26 (4H, bs, $2CH_2$), 6.51–7.15 (8H, m, H-Ar and 2NH), 9.13 (1H, s, NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ = 33.3, 48.7, 62.1, 105.1, 112.4, 115.5, 127.5, 134.5, 140.9. MS, m/z: 239 (M^+). Analytically calculated for $C_{15}H_{17}N_3$: C 75.28; H 7.16; N 17.56 %. Found: C 75.17; H 7.11; N 16.63 %.

4.2.13. 1′-Methyl-1,3-dihydrospiro[perimidine-2,4′-piperidine] **13b**

Dark brown powder; m.p. 115 °C (dec). IR (KBr): 3301, 3027, 1603/cm. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 1.91 (4H, bs, $2CH_2$), 2.28 (3H, s, CH_3), 2.79 (4H, bs, $2CH_2$), 6.50–7.54 (8H, m, H-Ar and 2NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ = 33.5, 42.7, 49.9, 61.5, 105.3, 112.4, 115.7, 127.3, 134.7, 140.8. MS, m/z: 253 (M^+). Analytically calculated for $C_{16}H_{19}N_3$: C 75.85; H 7.56; N, 16.59 %. Found: C 75.93; H 7.64; N 16.65 %.

4.2.14. 1′,3′-dihydrospiro[cyclododecane-1,2′-perimidine] **14**

Pink powder; m.p. 185 °C. IR (KBr): 3365, 3038, 2926, 1605 cm^{-1}. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 1.35–1.60 (22H, m, $11CH_2$), 6.14–7.11(8H, m, H-Ar and 2NH). ^{13}C NMR (75 MHz, DMSO-d_6): δ = 19.0, 22.1, 22.5, 26.0, 26.2, 33.2, 39.1, 39.4, 40.2, 40.5, 40.7, 68.5, 104.6, 112.9, 114.7, 127.4, 134.6, 141.9. MS, m/z: 322 (M^+). Analytically calculated for $C_{22}H_{30}N_2$: C 81.94; H 9.38; N 8.69 %. Found: C 81.86; H 9.43; N 8.76 %.

4.2.15. 1′,3′-dihydrospiro[indeno[2,1-b]quinoxaline-11,2′-perimidine] **15**

Brown powder; m.p. Greater than 290 °C. IR (KBr): 3369, 3033, 2917, 1598/cm. ^{1}H NMR (300 MHz, DMSO-d_6): δ = 6.42–8.11 (16H, m, H-Ar and 2NH). MS, m/z: 372 (M^+). Analytically calculated for $C_{25}H_{16}N_4$: C 80.63; H 4.33; N 15.03 %. Found: C 80.52; H 4.39; N 15.11 %.

Due to very low solubility of the product **15**, we cannot report the ^{13}C NMR data for this product.

Acknowledgements

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