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Synthesis of ferrocenyl quinolines

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ABSTRACT

A convenient one-pot synthesis of ferrocenyl-substituted quinolines via a molecular iodine-catalyzed reaction of ferrocenylimines with enolizable aldehydes is reported. First, nucleophilic addition of the in situ generated enol to ferrocenylimine produces β-anilinopropionaldehyde, which then undergoes intramolecular Friedel–Crafts reaction to give dihydroquinoline derivative. Finally, subsequent dehydration and aerobic oxidation affords ferrocenyl quinolines.

1. Introduction

Quinolines are present in a wide range of natural and unnatural compounds with remarkable medicinal activities [1]. In this regard, quinolines have occupied a unique position in the design and synthesis of novel biologically active compounds since they are often used as antiinflammatory, antiasthmatic, antituberculosis, antibacterial, antihypertensive, antitumor and, most notably, antimalarial agents [2,3]. In the light of recent studies [4,5], it might be expected that combination of a ferrocenyl moiety with such structures may increase their biological activities or create new medicinal properties. It is noteworthy to mention that due to its unique structure, different membrane-permeation behavior and anomalous metabolism, ferrocene is frequently integrated into an organic compound in order to have enhanced or unexpected biological activities [4,5]. Although quinolines are among the most extensively studied heterocyclic compounds [3,6], ferrocenyl-substituted quinolines are not often found in the literature [7]. Therefore, the synthesis of quinoline derivatives directly linked to a ferrocene unit, such as 2-ferrocenylquinolines, is of considerable interest since their properly substituted 2-aryl analogues are biologically active and exist in the structures of various antitumor agents [8]. Quinolines are usually prepared by Skraup [9], Doebner–Miller [10], Riehm [11], Combes [12], Conrad–Limbach [13], Knorr [14], Friedländer [15], Povarov [16], Camps [17], Niementowski [18], Gould–Jacobs [19], and Pfitzinger [20] quinoline syntheses. Recently, as shown by the Shimizu [21], Baba [22] and Wang [23] research groups, the reactions of aromatic imines (in situ synthesized or isolated) with enolizable aliphatic aldehydes in the presence of metal, Brønsted or Lewis acid catalysts also led to formation of quinoline derivatives. In this respect, molecular iodine has gained considerable importance as a mild and nontoxic Lewis acid catalyst since it catalyzed various organic reactions with high efficiency and selectivity [24]. Owing to carbonyl activating property [25], molecular iodine was successfully used as a catalyst in the quinoline forming reactions of imines and aldehydes as well [23,26]. However, to the best of our knowledge, such reactions catalyzed by iodine were not used for the synthesis of ferrocenyl-substituted quinolines. As part of a program to synthesize new ferrocenyl-substituted heterocyclic compounds as potential pharmaceuticals [27], we have investigated molecular iodine-catalyzed reactions of ferrocenyl imines with enolizable aliphatic aldehydes to afford 2-ferrocenylquinolines. We herein report the results of this study.

2. Results and discussion

The reactions were initially examined under a variety of conditions, such as refluxing THF, benzene, dioxane and toluene with varying amounts of molecular iodine catalyst. The best results were obtained as follows: ferrocenylimine **1** (1.2 equiv.) was reacted with aldehyde **2** (1.0 equiv.) in the presence of molecular iodine (0.1 equiv.) at 100 °C in dioxane for 2 h, and the products were isolated by flash chromatography. The results are summarized in Table 1.

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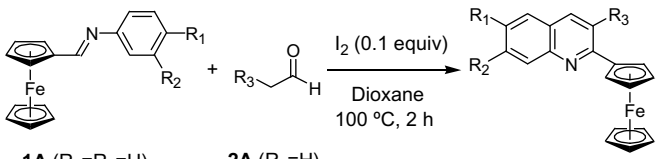
Table 1
Iodine-catalyzed reactions of ferrocenylimines **1** with enolizable aldehydes **2**



1A (R_1=R_2=H) **2A** (R_3=H)
1B (R_1=Cl, R_2=H) **2B** (R_3=CH_3)
1C (R_1=Br, R_2=H)
1D (R_1=H, R_2=CH_3)

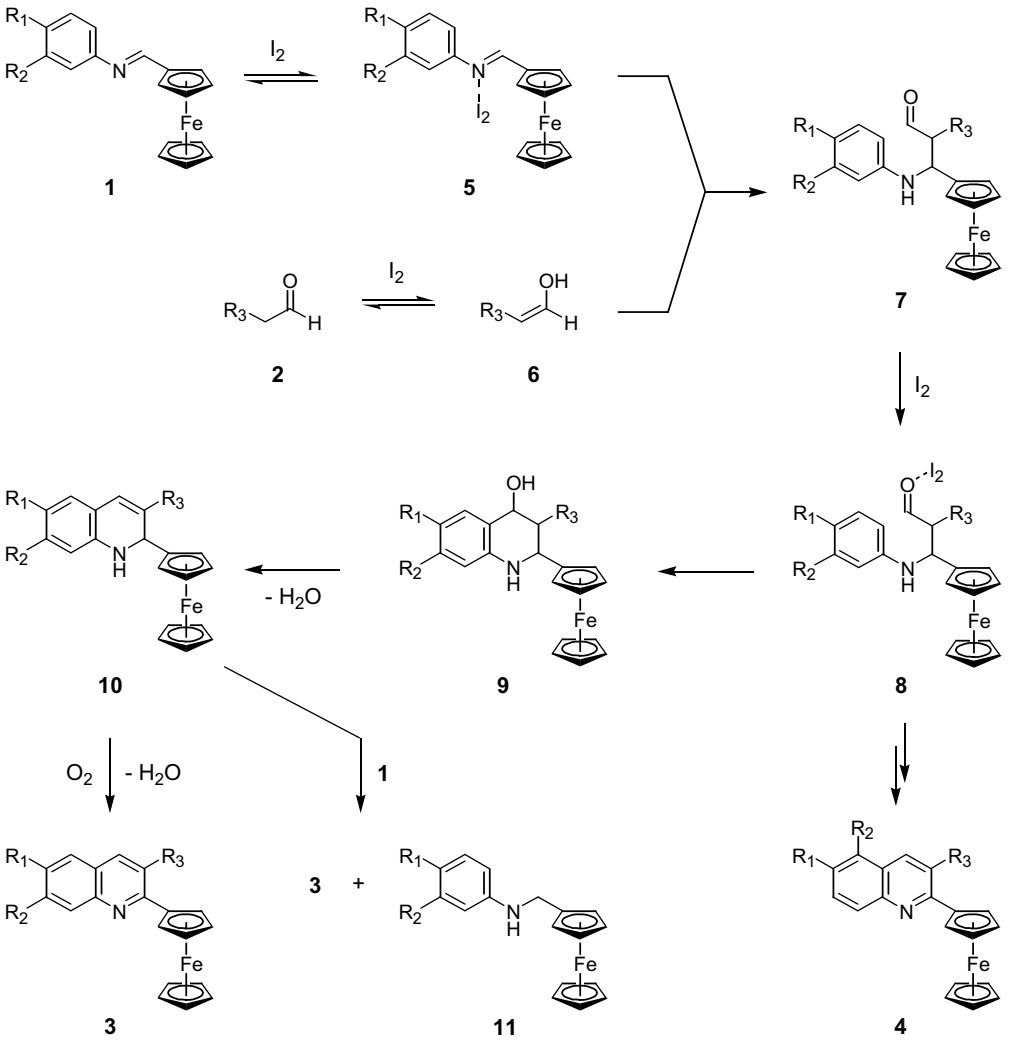
3

Entry[a]	Reacting Partners	R_1	R_2	R_3	Products (isolated yield, %)
A	**1A** + **2A**	H	H	H	**3A** (78)
B	**1B** + **2A**	Cl	H	H	**3B** (63)
C	**1C** + **2A**	Br	H	H	**3C** (65)
D	**1D** + **2A**	H	CH_3	H	**3D** (88)
E	**1D** + **2B**	H	CH_3	CH_3	**3E** (25)

[a] Entry letters define R_1, R_2 and R_3 groups for compound **3**.

As can be seen from Table 1, the reaction between *N*-(ferrocenylidene)aniline (**1A**) and acetaldehyde (**2A**) led to the formation of 2-ferrocenylquinoline (**3A**) in 78% yield (Entry A). Similarly, the reaction of acetaldehyde (**2A**) with 4-chloro-*N*-(ferrocenylidene)aniline (**1B**) and 4-bromo-*N*-(ferrocenylidene)aniline (**1C**) afforded 6-chloro-2-ferrocenylquinoline (**3B**) and 6-bromo-2-ferrocenylquinoline (**3C**) in 63% and 65% yields, respectively (Entries B and C). Interestingly, from the reaction between *N*-(ferrocenylidene)-3-methylaniline (**1D**) and acetaldehyde (**2A**), only one regioisomer, namely 2-ferrocenyl-7-methylquinoline (**3D**), was isolated in 88% yield (Entry D). The reaction of ferrocenylimine **1D** with propionaldehyde (**2B**) resulted in a complex mixture, from which only 2-ferrocenyl-3,7-dimethylquinoline (**3E**) was isolated even though in low yield (25%) (Entry E). Formation of polymeric by-products as well as the partial hydrolysis of starting imine **1D** lowered the yield of **3E**.

The mechanism proposed for the formation of quinolines **3** is depicted in Scheme 1. It is well known that in the presence of Lewis acids such as iodine, imines such as **1** can be activated. Similarly, in the presence of iodine, aldehydes such as **2** can be easily equilibrated with their enols. As anticipated, the reaction between in situ generated enol **6** and iodine-activated imine **5** affords β-anilinopropionaldehyde **7**. The intramolecular Friedel–Crafts reaction of iodine-activated β-anilinopropionaldehyde **8** produces tetrahydroquinolinol derivative **9**. The subsequent dehydration in **9** leads to formation of dihydroquinoline **10**. Finally, the aerobic oxidation of **10** yields the expected quinoline derivative **3**. Alternatively, dihydroquinoline **10** can be oxidized to quinoline **3** by the



Scheme 1.

starting imine **1** to some extent since it was reported that imines can behave as hydrogen acceptor in these types of reactions [21c]. In this case, reaction should also produce a secondary amine, i.e. (ferrocenylmethyl)aniline derivative **11** (Scheme 1). However, the formation of such amines in these reactions was not detected. It should be mentioned that iodine-activated β-propionaldehyde **8** can also produce ferrocenyl quinoline **4**, an isomer of **3**, via a similar mechanism (Scheme 1). However, the formation of such quinolines in these reactions was not observed.

3. Conclusion

We have investigated the iodine-catalyzed reaction between ferrocenylimines **1** and enolizable aldehydes **2** to afford 2-ferrocenylquinolines **3**. In all cases, the expected 2-ferroceylquinolines **3** were obtained from these reactions. Due to the ready availability of ferrocenylimines **1** and aldehydes **2**, this practical one-pot method represents a versatile synthesis of ferrocenyl-substituted quinolines **3**.

4. Experimental

4.1. General consideration

Nuclear Magnetic Resonance (^{1}H and ^{13}C) spectra were recorded on a Bruker Spectrospin Avance DPX400 Ultrashield (400 MHz) spectrometer. Chemical shifts are reported in parts per million (δ) downfield from an internal tetramethylsilane reference. Coupling constants (J values) are reported in hertz (Hz), and spin multiplicities are indicated by the following symbols: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet). DEPT ^{13}C NMR information is given in parenthesis as C, CH, CH_2 and CH_3. Infrared spectra were recorded on a Bruker Vertex 70 Spectrometer using attenuated total reflection (ATR). Band positions are reported in reciprocal centimeters (cm^{-1}). Mass spectra (MS) were obtained on a Bruker Daltonics spectrometer using Electrospray Ionization (ESI) with Micro-Tof; m/z values are reported (For each measurement, the mass scale was recalibrated with sodium formiate clusters, and samples were dissolved and measured in MeOH). High resolution mass spectra (HRMS) were also obtained on a Bruker Daltonics spectrometer. Flash chromatography was performed using thick-walled glass columns and 'flash grade' silica (Merck 230–400 mesh). The relative proportion of solvents in mixed chromatography solvents refers to the volume:volume ratio. All other commercially available reagents and reactants were obtained in reagent grade and used without purification. All reaction solvents were distilled and/or dried for purity according to standard literature procedures. The inert atmosphere was created by slight positive pressure (ca. 0.1 psi) of argon.

4.2. Synthesis of starting materials

Ferrocenylimines, known also as ferrocenylaldimines or Schiff bases, were prepared by condensation of ferrocenecarboxaldehyde with corresponding aniline derivatives according to known literature procedures [28].

4.3. General procedure for the synthesis of ferrocenyl quinolines **3** (Table 1)

To a solution of ferrocenylimine **1** (0.72 mmol) and aldehyde **2** (0.60 mmol) in 3 mL of dioxane was added iodine (15.36 mg, 0.06 mmol). The resulting mixture was then heated at reflux for 2 h. After the reaction was complete, the mixture was cooled to 25 °C, and the solvent was removed on a rotary evaporator. Final purification of the obtained residue was achieved through flash chromatography on silica gel using hexane/EtOAc (24/1) as eluent. The products given in Table 1 were isolated with the indicated yields.

4.4. Spectral data for products

4.4.1. 2-Ferrocenylquinoline [(Quinolin-2-yl)ferrocene] (**3A**)

^{1}H NMR ($CDCl_3$): δ 8.06 (d, 1H, J = 8.5 Hz), 8.02 (d, 1H, J = 8.5 Hz), 7.74 (d, 1H, J = 8.0 Hz), 7.66 (pseudo t, 1H, J = 7.4 Hz), 7.56 (d, 1H, J = 8.5 Hz), 7.45 (pseudo t, 1H, J = 7.4 Hz), 5.08 (pseudo t, 2H, J = 1.6 Hz), 4.47 (pseudo t, 2H, J = 1.6 Hz), 4.05 (s, 5H); ^{13}C NMR ($CDCl_3$): δ 159.5 (C), 148.3 (C), 135.4 (CH), 129.3 (CH), 129.0 (CH), 127.5 (CH), 126.7 (C), 125.4 (CH), 119.5 (CH), 84.0 (C), 70.4 (CH), 69.7 (CH), 68.0 (CH); IR (neat): 3092, 3061, 2922, 2851, 1614, 1599, 1556, 1510, 1424, 1280, 1129, 1104, 1092, 910, 907, 815, 756 cm^{-1}; MS (ESI, m/z): 314.06 $[M+H]^+$; HRMS (ESI): calc. for $C_{19}H_{16}FeN$: 314.0632 $[M+H]^+$. Found: 314.0627. The spectral data are in agreement with those previously reported [7a,7b,7d].

4.4.2. 6-Chloro-2-ferrocenylquinoline [(6-Chloroquinolin-2-yl)-ferrocene] (**3B**)

^{1}H NMR ($CDCl_3$): δ 7.94 (m, 2H), 7.70 (s, 1H), 7.56 (m, 2H), 5.05 (s, 2H), 4.47 (s, 2H), 4.04 (s, 5H); ^{13}C NMR ($CDCl_3$): δ 160.0 (C), 146.6 (C), 134.5 (CH), 130.9 (CH), 130.5 (CH), 130.2 (C), 127.2 (C), 126.2 (CH), 120.3 (CH), 83.3 (C), 70.6 (CH), 69.7 (CH), 68.0 (CH); IR (neat): 3094, 2923, 2853, 1597, 1548, 1503, 1485, 1411, 1379, 1327, 1281, 1189, 1132, 1106, 1094, 1074, 1026, 999, 948, 873, 853, 811 cm^{-1}; MS (ESI, m/z): 370.00 $[M+Na]^+$, 348.02 $[M+H]^+$; HRMS (ESI): calc. for $C_{19}H_{14}ClFeNNa$: 370.0062 $[M+Na]^+$. Found: 370.0057; calc. for $C_{19}H_{15}ClFeN$: 348.0242 $[M+H]^+$. Found: 348.0237.

4.4.3. 6-Bromo-2-ferrocenylquinoline [(6-Bromoquinolin-2-yl)-ferrocene] (**3C**)

^{1}H NMR ($CDCl_3$): δ 7.91 (d, 1H, J = 8.5 Hz), 7.89 (d, 1H, J = 8.5 Hz), 7.88 (s, 1H), 7.70 (dd, 1H, J = 8.5, 2.0 Hz), 7.54 (d, 1H, J = 8.5 Hz), 5.04 (pseudo t, 2H, J = 1.6 Hz), 4.47 (pseudo t, 2H, J = 1.6 Hz), 4.04 (s, 5H); ^{13}C NMR ($CDCl_3$): δ 160.1 (C), 146.9 (C), 134.4 (CH), 132.7 (CH), 130.7 (CH), 129.5 (CH), 127.8 (C), 120.2 (CH), 118.9 (C), 83.4 (C), 70.7 (CH), 69.7 (CH), 68.0 (CH); IR (neat): 3094, 3069, 2923, 1593, 1545, 1500, 1381, 1327, 1281, 1188, 1134, 1105, 1062, 1025, 944, 880, 844 cm^{-1}; MS (ESI, m/z): 413.95 $[M+Na]^+$, 391.97 $[M+H]^+$; HRMS (ESI): calc. for $C_{19}H_{15}BrFeN$: 391.9737 $[M+H]^+$. Found: 391.9733.

4.4.4. 2-Ferrocenyl-7-methylquinoline [(7-Methylquinolin-2-yl)-ferrocene] (**3D**)

^{1}H NMR ($CDCl_3$): δ 7.97 (d, 1H, J = 8.5 Hz), 7.84 (s, 1H), 7.62 (d, 1H, J = 8.2 Hz), 7.49 (d, 1H, J = 8.5 Hz), 7.29 (d, 1H, J = 8.2 Hz), 5.06 (s, 2H), 4.45 (s, 2H), 4.04 (s, 5H), 2.55 (s, 3H); ^{13}C NMR ($CDCl_3$): δ 159.4 (C), 148.5 (C), 139.6 (C), 135.1 (CH), 128.2 (CH), 127.6 (CH), 127.2 (CH), 124.7 (C), 118.7 (CH), 84.1 (C), 70.3 (CH), 69.6 (CH), 67.9 (CH), 21.8 (CH_3); IR (neat): 3094, 3037, 2914, 2854, 1625, 1595, 1558, 1517, 1435, 1277, 1104, 1091, 1027, 998, 874, 844, 811 cm^{-1}; MS (ESI, m/z): 350.06 $[M+Na]^+$, 328.08 $[M+H]^+$; HRMS (ESI): calc. for $C_{20}H_{18}FeN$: 328.0789 $[M+H]^+$. Found: 328.0783.

4.4.5. 2-Ferrocenyl-3,7-dimethylquinoline [(3,7-Dimethylquinolin-2-yl)ferrocene] (**3E**)

^{1}H NMR ($CDCl_3$): δ 7.83 (s, 1H), 7.79 (s, 1H), 7.58 (d, 1H, J = 8.2 Hz), 7.27 (d, 1H, J = 8.2 Hz), 5.09 (s, 2H), 4.44 (s, 2H), 4.10 (s, 5H), 2.74 (s, 3H), 2.54 (s, 3H); ^{13}C NMR ($CDCl_3$): δ 158.3 (C), 146.9 (C), 138.5 (C), 136.4 (CH), 128.3 (C), 127.9 (CH), 127.8 (CH),

126.2 (CH), 124.9 (C), 85.5 (C), 70.0 (CH), 69.8 (CH), 69.5 (CH), 21.8 (CH_3), 21.4 (CH_3); IR (neat): 3092, 3071, 3051, 2969, 2920, 2854, 1625, 1599, 1502, 1453, 1410, 1383, 1323, 1268, 1140, 1105, 1074, 999, 896, 877, 823, 808, 779 cm^{-1}; MS (ESI, *m*/*z*): 342.09 $[M+H]^+$; HRMS (ESI): calc. for $C_{21}H_{20}FeN$: 342.0945 $[M+H]^+$. Found: 342.0940.

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