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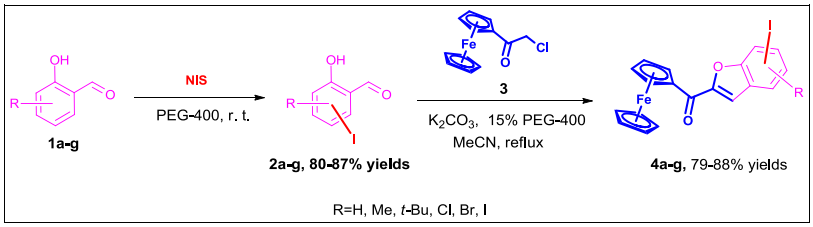

In the present investigation, the first construction of a series of structurally new 2-ferrocenoyl-substituted iodobenzofurans hybrids has been achieved through a simple and mild two-step procedure, involving the iodination of salicylaldehydes using *N*-iodosuccinimide reagent in eco-friendly PEG-400 medium at room temperature followed by one-pot Rap–Stoermer reaction with 1-chloroacetylferrocene in refluxing MeCN with the presence of K_2CO_3 as base and PEG-400 as the activated additive. These newly synthesized compounds belong to a new class of ferrocene-benzofuran hybrids and could be good candidates for the development of compounds for use in medicinal chemistry.

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INTRODUCTION

2-Aroybenzofurans have received considerable interest from the medicinal community because of their well-documented biological properties such as antimicrobial [1], anticonvulsant, anti-inflammatory [2], and antifungal activities [3] and selective cytotoxicity against a tumorigenic cell line [4]. Especially, some iodinated derivatives of such molecules have been recently reported, and their biological and pharmacological properties look promising. For example, *Amiodarone* is widely used in the treatment of ventricular tachyarrhythmia and atrial fibrillation [5]. In this regard, an impressive report from Cui *et al.* described that iodinated 2-aroybenzofuran displayed high affinity for Aβ aggregates and could be used as potential probes for detecting β-amyloid plaques in the AD brain [6]. In addition, the presence of the derivatizable iodo group, representing potentially useful synthetic building blocks in synthetic organic chemistry, is another reason for the interest in the synthesis of such compounds. As a consequence, interest in the synthesis of new families of iodo-substituted 2-aroybenzofuran derivatives continues unabated.

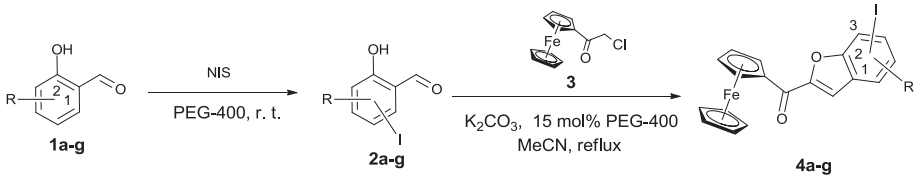
On the other hand, it has been well established that interchange of an aromatic or heterocyclic ring with ferrocene core in some organic compounds possessing a certain property (e.g., biological activity) might lead to new hybrids with enhanced or unexpected chemical and pharmacological properties compared with that of the parent compound [7,8]. As an example, when a ferrocenyl group was used to modify chloroquine, the obtained ferroquine exhibits higher antimalarial activity [9]. This fact could be rationalized as being due to the unique properties of the ferrocene nucleus such as membrane permeation, aqueous stability, anomalous metabolism, and redox behavior. As a result, the ferrocene-heterocycle hybrid has been an attractive synthetic target, and much synthetic effort has been devoted to design and synthesize more novel and interesting such molecules to expand the structure diversity for current medicinal chemistry needs [10–12].

Considering the aforementioned valid points as well as the combination principles for drug design [13], we felt that it would be of synthetic importance to the construction of 2-ferrocenoyl-containing iodobenzofurans. Such compounds would likely possess significant biological activities or could be potentially applied as useful synthetic building blocks for the development of new medicinal products with interesting properties. To the best of our knowledge, the construction of this type of hybrid molecules still remained unknown. Accordingly, with this context and in continuation of our studies on the synthesis of highly interesting types of hybrid heterocycles [14–19], we would like to report, herein, the first synthesis of a series of meaningful 2-ferrocenoyl-based iodobenzofurans.

RESULT AND DISCUSSION

Our strategy to reach this goal is outlined in Scheme 1. The application of the Rap–Stoermer reaction appeared to suit us, because it might provide the opportunity for direct construction of the targeted compounds from 1-chloroacetyl ferrocene and iodinated salicylaldehydes.

Scheme 1. Route for 2-ferrocenoyl-substituted iodobenzofurans (**4a–g**).



1a, 2a, 4a: R=H; **1b, 2b, 4b**: R=5-Me; **1c, 2c, 4c**: R=3-Me
1d, 2d, 4d: R=3-*t*-Bu; **1e, 2e, 3e**: R=5-*t*-Bu; **1f, 2f, 4f**: R=5-Cl
1g, 2g, 4g: R=5-Br

Because the number of commercially available iodo-substituted salicylaldehydes is exceedingly small, our synthesis commenced with the iodination of some salicylaldehydes. Prior to the current investigation, there are some reported methods involving the iodination of salicylaldehyde such as the use of ICl in AcOH at 120°C [20], KI/KIO_3 in HAC and H_3PO_4 at 50–70°C [21], and I_2 in pyridine and dioxane [22]. Although they provided useful access to iodo-substituted salicylaldehydes, these iodination methodologies suffered from one or more drawbacks arising out of its toxic, corrosive, and environmentally hazardous nature. Thus, in our search for a green and mild protocol for the iodination of salicylaldehydes, we are delightful to find that treatment of some commercially available salicylaldehydes **1a–g** with *N*-iodosuccinimide (NIS) in eco-friendly PEG-400 medium at room temperature delivered good isolated yields of the iodinated salicylaldehyde **2a–g**. The results obtained thus far are summarized in Table 1.

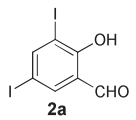
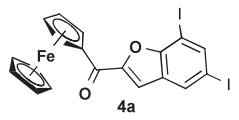
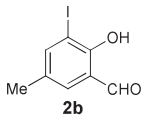
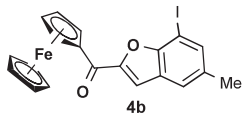
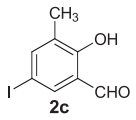
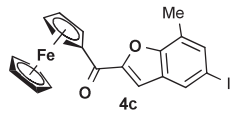
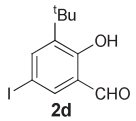
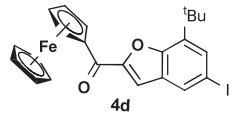
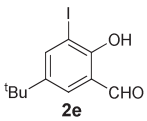
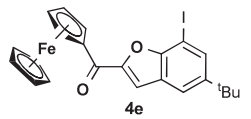
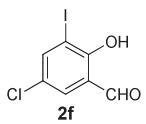
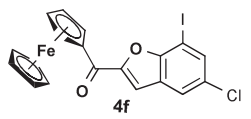
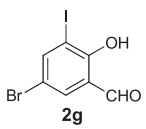
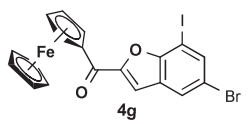
Having a series of the iodo-substituted salicylaldehydes **2a–g** in hand, our attention was transferred to their Rap–Stoermer reaction with 1-chloroacetylferrocene for building the 2-ferrocenoylbenzofuran system. At this stage, we first investigated the reaction of 3-iodo-5-methylsalicylaldehyde (**2b**) with 1-chloroacetylferrocene (**3**) under solvent-free conditions [23,24] or using 4-dimethylaminopyridine [25] as catalyst in water in accordance with literature methods. Although these procedures are very easy, eco-friendly, and impressive, the purported approaches were ineffective in our hands, and the reaction did not proceed satisfactorily, giving poor yields of highly impure products. Our attempts to follow the route as described by Cui *et al.* [6] by using K_2CO_3 as base in acetone at room temperature were also frustrated by low yield. After many trials, we found that when the reaction was run in the presence of PEG-400 (15 mol%) as the activated additive and K_2CO_3 as the base in refluxing MeCN for 5 h [monitored by thin-layer chromatography (TLC)], the desired 2-ferrocenoyl-7-iodo-5-methylbenzo[*b*]furan (**4b**) was obtained in a good yield of 85%. Presumably, the reaction may be the fact that PEG-400 could stabilize the transition state [26], thereby resulting in an increase in the reaction rate. The amount of 15 mol% PEG-400 was sufficient to push this reaction forward, and more than that amount resulted in a lower yield, which might be because the transition state could be solvated in the larger amount of PEG-400. Subsequently, we further extended the reaction to other iodo-salicylaldehydes in a similar fashion. As expected, this reaction invariably led to the formation of the corresponding 2-ferrocenoyl-substituted iodobenzofurans in satisfactory yields, and the results of this series of experiments are compiled in the Table 1.

To the best of our knowledge, none of the newly synthesized compounds have yet been reported, and their structures were easily established based on spectral data elemental analyses, which were in good agreement with the compounds expected. For instance, the IR spectrum of reaction product **4a** clearly showed a typical stretching vibration band at 1632 cm^{-1} due to the carbonyl group, supporting the signal of its ^{13}C NMR spectrum at 184.72 ppm. Its ^1H NMR spectrum showed the usual pattern of monosubstituted ferrocene (Fc) system—two magnetically inequivalent *ortho* protons (H-2′ and H-5′) at 5.25 downfield from the signals of the two *meta* protons (H-3′ and H-4′) at 4.68, and a strong signal at 4.20 due to the five equivalent hydrogens of the unsubstituted cyclopentadienylidene ring. A particular characteristic was the presence of three benzofuran protons in the aromatic range between 7.45 and 7.71 ppm, which is consistent with the molecular structure suggested. Further, the structure assigned to the reaction product **4a** was fully supported by its high-resolution mass spectrometry (HRMS), which established its molecular formula to be $C_{19}H_{12}FeI_2O_2$ in accordance with the suggested molecular structure. The other synthesized compounds exhibited similar spectral characteristics.

In summary, the synthesis of a series of hitherto unreported 2-ferrocenoyl-substituted iodobenzofurans derivatives has been achieved with good yields under mild reaction conditions. These molecules we have synthesized should allow us, in the future, to investigate structure–activity relationships over various biotests. Moreover, all these molecules could be potentially applied as useful synthetic building blocks for the development of more

Table 1

Yields of the newly synthesized compounds **2a–g** and **4a–g**.

Entry	Iodosalicylaldehyde	Yield (%)[a]	Product	Yield (%)[a]
1	 **2a**	83	 **4a**	81
2	 **2b**	87	 **4b**	85
3	 **2c**	84	 **4c**	79
4	 **2d**	81	 **4d**	88
5	 **2e**	83	 **4e**	85
6	 **2f**	80	 **4f**	82
7	 **2g**	87	 **4g**	84

[a]Isolated yields.

complex ferrocene-based compounds because iodine group on the benzofuran ring can be further elaborated to a variety of other functional groups, for example, via metal-catalyzed coupling reactions. Currently, the studies concerning their application are underway.

EXPERIMENTAL

General. All chemicals (analytical reagent (AR) graded) were commercially available and used without further purification. The melting points were determined by using WRS-1B melting points apparatus and were uncorrected. The IR spectra were obtained as KBr pellets in the range of 400–4000 cm^{-1} on a Shimadzu FTIR-8400S spectrophotometer (Shimadzu, Japan). ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance NMR spectrometer (Bruker Company, Switzerland) using $CDCl_3$ as the solvent. The reported chemical shifts (δ values) are given in parts per million downfield from tetramethylsilane as the internal standard. The mass spectra were determined using an MSD VL ESI1 spectrometer. HRMS (ESI) data were acquired on a Bruker Customer micrOTOF-Q 125 high-resolution mass spectrometer with ESI. The progress of reactions was monitored by TLC on silica gel GF254 using hexane/ethyl acetate mixture as eluent (4:1, v/v).

General procedure for the synthesis of iodo-salicylaldehydes 2a–g. Respective substituted salicylaldehyde (**1a–g**) (1.0 mmol) and NIS (0.27 g, 1.2 mmol) (unsubstituted salicylaldehyde required 2.2 mmol of NIS) was dissolved in PEG-400 (4 mL) and then stirred at room temperature for 4 h. After completion of the reaction as monitored by TLC, the reaction mixture was poured into water. The crude product obtained after filtration and washing with water was recrystallized from ethanol to give pure **2a–g**.

3,5-Diiodosalicylaldehyde (2a). Yield 83%, mp 104–105°C (Lit. [21]: 101–103°C); ^{1}H NMR ($CDCl_3$, 400 MHz): 11.47 (s, 1H, OH), 9.90 (s, 1H, CHO), 8.31 (s, 1H, Ben-H), 8.08 (s, 1H, Ben-H); MS (ESI, *m/z*): 375.1 $[M+H]^+$.

5-Methyl-3-iodosalicylaldehyde (2b). Yield 87%, mp 90–91°C; ^{1}H NMR ($CDCl_3$, 400 MHz): 11.88 (s, 1H, OH), 9.65 (s, 1H, CHO), 7.95 (s, 1H, ArH), 2. 83 (s, 3H, CH_3); ^{13}C NMR ($CDCl_3$, 100 MHz): 189.33, 155.69, 148.06, 138.35, 115.20, 87.44, 81.53, 31.35; MS (ESI, *m/z*): 263.0 $[M+H]^+$.

3-Methyl-5-iodosalicylaldehyde (2c). Yield 84%, mp 87–88°C (Lit. [27]: 85–86°C); ^{1}H NMR ($CDCl_3$, 400 MHz): 10.90 (s, 1H, OH), 10.00 (s, 1H, CHO), 7.91 (d, 1H, Ben-H), 7.79 (s, 1H, Ben-H), 2.17 (s, 3H, CH_3); MS (ESI, *m/z*): 263.1 $[M+H]^+$.

3-tert-Butyl-5-iodosalicylaldehyde (2d). Yield 81%, mp 53–54°C (Lit. [20]: 55°C); ^{1}H NMR (DMSO, 600 MHz): 11.75 (s, 1H, OH), 9.93 (s, 1H, CHO), 8.01 (s, 1H, ArH), 7.70 (s, 1H, ArH), 1.36 (s, 9H, *t*-Bu); MS (ESI, *m/z*): 305.1 $[M+H]^+$.

5-tert-Butyl-3-iodosalicylaldehyde (2e). Yield 73%, mp 78–79°C (Lit. [28]: 75–77°C); ^{1}H NMR (DMSO, 600 MHz) 11.34 (s, 1H, OH), 9.95 (s, 1H, CHO), 8. 05 (s, 1H, ArH), 7.83 (s, 1H, ArH), 1.28 (s, 9H, *t*-Bu); MS (ESI, *m/z*): 304.9 $[M+H]^+$.

5-Chloro-3-iodosalicylaldehyde (2f). Yield 80%, mp 78–78°C (Lit. [29]: 77°C); ^{1}H NMR ($CDCl_3$, 400 MHz): 11.65 (s, 1H, OH), 9.66 (s, 1H, CHO), 7.91 (s, 1H, ArH), 7.50 (s, 1H, ArH); MS (ESI, *m/z*): 283.0, 284.8 $[M+H]^+$.

5-Bromo-3-iodosalicylaldehyde (2g). Yield 87%, mp 81–81°C (Lit. [29]: 80°C); ^{1}H NMR ($CDCl_3$, 400 MHz): 11.71 (s, 1H, OH), 9.70 (s, 1H, CHO), 8.07 (s, 1H, ArH), 7.67 (s, 1H, ArH); MS (ESI, *m/z*): 326.9, 329.1 $[M+H]^+$.

General procedure for the synthesis of 2-ferrocenoyl-substituted iodobenzofurans (4a–g). To a stirred solution of 1-chloroacetyl ferrocene (**3**) (0.262 g, 1.0 mmol) and respective iodo-salicylaldehydes (**2a–g**) (1.1 mmol) in acetonitrile (12 mL) was added K_2CO_3 (0.276 g, 2.0 mmol) and PEG-400 (15 mmol%). The resulting reaction mixture was heated at refluxing temperature for 5 h. After the reaction was complete (TLC), the mixture was cooled at room temperature and poured into water, and the resulting solid, which precipitated on standing, was collected. Recrystallization from EtOH gave the corresponding pure product **4a–g**.

2-Ferrocenoyl-5,7-diiodobenzo[b]furan (4a). Dark red solid, mp 170–171°C. IR (KBr) ν/cm^{-1}: 3106, 1632 (C=O), 1548, 1442, 1374, 1309, 1284, 1234, 1168, 824, 808; ^{1}H NMR (500 MHz, CDCl3) δ (ppm): 7.71 (d, J = 1.4 Hz, 1H, furan-H), 7.55 (d, J = 6.8 Hz, 1H, Ben-H), 7.45 (dd, J = 6.8, 1.4 Hz, 1H, Ben-H), 5.25 (s, 2H, Fc-H), 4.68 (s, 2H, Fc-H), 4.20 (s, 5H, Fc-H); ^{13}C NMR (125 MHz, $CDCl_3$) δ (ppm): 184.72, 155.33, 143.08, 134.16, 131.68, 130.91, 129.26, 111.15, 87.70, 79.87, 73.63, 71.25, 70.43; HRMS (ESI, *m/z*). Calcd. for $C_{19}H_{13}FeI_2O_2$ $[M+H]^+$ 582.8354. Found 582.8370.

2-Ferrocenoyl-7-iodo-5-methylbenzo[b]furan (4b). Dark red solid, mp 140–141°C. IR (KBr) ν/cm^{-1}: 3084, 2916, 1628 (C=O), 1552, 1440, 1377, 1311, 1291, 1233, 1140, 825, 807; ^{1}H NMR (400 MHz, CDCl3) δ (ppm): 8.65 (d, J = 2.0 Hz, 1H, Ben-H), 8.59 (d, J = 2.4 Hz, 1H, Ben-H), 7.79 (s, 1H, Furan-H), 5.34 (t, J = 2.0 Hz, 2H, Fc-H), 4.78 (t, J = 2.0 Hz, 2H, Fc-H), 4.15 (s, 5H, Fc-H), 2.05 (s, 3H, Me); ^{13}C NMR (100 MHz, CDCl3) δ (ppm): 185.27, 154.06, 136.55, 132.62, 129.17, 129.01, 124.61, 111.04, 87.39, 80.07, 73.48, 71.05, 70.43, 15.08; HRMS (ESI, *m/z*). Calcd. for $C_{20}H_{16}FeIO_2$ $[M+H]^+$ 470.9544. Found 470.9523.

2-Ferrocenoyl-5-iodo-7-methylbenzo[b]furan (4c). Dark red solid, mp 105–106°C. IR (KBr) ν/cm^{-1}: 3078, 2964, 1629 (C=O), 1541, 1445, 1375, 1328, 1286, 1232, 1134, 842, 809; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.34 (d, J = 2.0 Hz, 1H, Ben-H), 8.06 (d, J = 2.0 Hz, 1H, Ben-H), 7.67 (s, 1H, furan-H), 5.47 (t, J = 2.0 Hz, 2H, Fc-H), 4.80 (t, J = 2.0 Hz, 2H, Fc-H), 4.24 (s, 5H, Fc-H), 2.05 (s, 3H, Me); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 184.88, 156.73, 141.57, 132.68, 130.71, 129.83, 126.41, 111.03, 93.13, 80.93, 73.88, 71.34, 70.52, 34.18; HRMS (ESI, *m/z*). Calcd. for $C_{20}H_{16}FeIO_2$ $[M+H]^+$ 470.9544. Found 470.9532.

2-Ferrocenoyl-5-iodo-7-t-butylbenzo[b]furan (4d). Dark red solid, mp 106–107°C. IR (KBr) ν/cm^{-1}: 3072, 2968, 2866, 1630, 1552, 1439, 1372, 1335, 1284, 1233, 1125, 824, 802; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.60 (d, J = 2.0 Hz, 2H, Ben-H), 7.55 (d, J = 1.7 Hz, 1H, furan-H), 5.33 (t, J = 1.9 Hz, 2H, Fc-H), 4.69 (t, J = 1.9 Hz, 2H, Fc-H), 4.22 (s, 5H, Fc-H), 1.39 (s, 9H, ^{t}Bu-H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 184.78, 153.11, 137.86, 133.80, 131.65, 130.17, 129.66, 111.09, 88.00, 79.96, 73.31, 70.91, 70.30, 34.46, 29.75; HRMS (ESI, *m/z*). Calcd. for $C_{23}H_{22}FeIO_2$ $[M+H]^+$ 513.0014. Found 512.9992.

2-Ferrocenoyl-5-t-butyl-7-iodobenzo[b]furan (4e). Dark red solid, mp 102–103°C. IR (KBr) ν/cm^{-1}: 3080, 2950, 2903, 1635, 1557, 1443, 1374, 1327, 1287, 1219, 1140, 845, 819; ^{1}H NMR (400 MHz,$CDCl_3$) δ (ppm): 8.54 (d, J = 2.2 Hz, 1H, Ben-H), 8.30 (d, J = 2.3 Hz, 1H, Ben-H),

7.74 (s, 1H, furan-H), 5.32 (t, $J = 1.9$ Hz, 2H, Fc-H), 4.75 (t, $J = 1.9$ Hz, 2H, Fc-H), 4.22 (s, 5H, Fc-H), 1.68 (s, 9H, ^{t}Bu-H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 185.32, 153.49, 137.43, 132.80, 131.35, 131.01, 129.83, 110.97, 91.06, 80.18, 75.17, 73.86, 71.35, 34.89, 31.69; HRMS (ESI, *m/z*). Calcd. for $C_{23}H_{22}FeIO_2$ $[M+H]^+$ 513.0014. Found 513.0021.

5-Chloro-2-ferrocenoyl-7-iodobenzo[b]furan (4f). Dark red solid, mp 125–126°C. IR (KBr) ν/cm^{-1}: 3091, 1628, 1545, 1454, 1376, 1351, 1287, 1216, 1147, 820, 805; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.61 (d, $J = 2.8$ Hz, 1H, Furan-H), 7.47 (d, $J = 1.7$ Hz, 1H, Ben-H), 7.27–7.30 (m, 1H, Ben-H), 5.30 (t, $J = 1.9$ Hz, 2H, Fc-H), 4.68 (t, $J = 1.9$ Hz, 2H, Fc-H), 4.21 (s, 5H, Fc-H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 185.75, 154.35, 137.96, 133.36, 131.38, 129.57, 125.14, 111.50, 89.13, 81.26, 73.82, 71.14, 70.47; HRMS (ESI, *m/z*). Calcd. for $C_{19}H_{13}^{35}ClFeIO_2$ $[M+H]^+$ 490.8998. Found 490.9012.

5-Bromo-2-ferrocenoyl-7-iodobenzo[b]furan (4g). Dark red solid, mp 137–138°C. IR (KBr) ν/cm^{-1}: 3060, 1633, 1555, 1452, 1376, 1342, 1288, 1212, 1140, 828, 807; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.72 (s, 1H, Furan-H), 7.61 (s, 1H, Ben-H), 7.54–7.58 (m, 1H, Ben-H), 5.27 (t, $J = 1.7$ Hz, 2H, Fc-H), 4.65 (t, $J = 1.9$ Hz, 2H, Fc-H), 4.20 (s, 5H, Fc-H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm): 186.26, 153.47, 138.28, 132.59, 132.13, 129.77, 126.24, 110.76, 88.94, 81.35, 73.60, 71.37, 70.41; HRMS (ESI, *m/z*). Calcd. for $C_{19}H_{13}^{79}BrFeIO_2$ $[M+H]^+$ 534.8493. Found 534.8502.

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