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A simple and efficient approach for the synthesis of functionalized naphtho[2,1-*b*]furan *via* an one-pot, three-component reaction between Meldrum's acid, arylglyoxals, and β-naphthol

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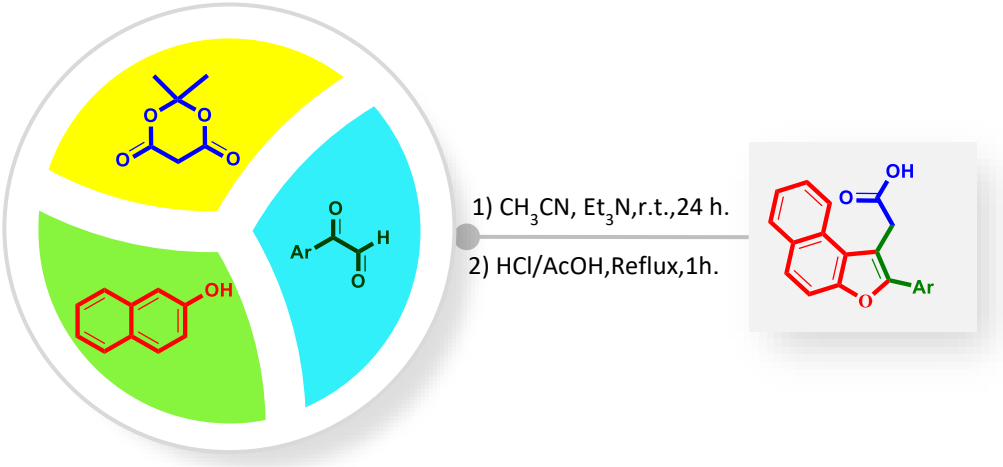
Abstract

A simple and efficient method for the synthesis of 2-(2-(aryl)naphtho[2,1-*b*]furan-1-yl)acetic acid derivatives *via* an one-pot three-component reaction of Meldrum's acid, arylglyoxals, and β-naphthol in the presence of triethylamine (Et_3N) is reported. The protocol avoids the use of expensive catalysts, chromatographic separation and provides a wide range of novel naphtho[2,1-*b*]furans in excellent yields. Spectral data and elemental analysis have characterized the newly synthesized compounds.



Keywords: Naphtho[2,1-*b*]furan, Meldrum's acid, arylglyoxal, β-naphthol, three-component reaction
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Introduction

Naphthofuran is a bicyclic heterocyclic structure that results from the fusion of a twelve-membered naphthalene ring to a five-membered furan ring.[1] The interest to the synthesis and study of the naphtho[2,1-*b*]furan nucleus is due to the wide spectrum of significant biological activity exhibited by this class of heterocycles, including antibacterial,[2] antimicrobial,[3] anthelmintic,[4] analgestic,[5] and oestrogenic properties.[6] Moreover, naphthofurans and its derivatives are important building units found as key structural motifs in a large number of biologically important natural products (Figure 1). In continuation of our studies on the application of natural products, we cite here several examples. (±) Heritol (**I**) has been isolated from the mangrove plant *Heritiera littoralis*, which is a novel ichthyotoxin.[7] A naphthofuran-containing heterocycle, Balsaminone A (**II**) was isolated from the pericarp of the fruit of *Impatiens Balsamina* L. (Balsaminaceae), which exhibited significant antipruritic activity. In addition, this compound is used in traditional Chinese medicine to treat articular rheumatism, bruises, and beri-beri.[8,9] Furomollugin (**III**) has been isolated from several members of the Rubiaceae family and from *Rubia cordifolia*, and is cytotoxic to human colon carcinoma cells (HT-29). Furomollugin also possesses a strong inhibitory activity against DNA topoisomerases I and II.[10] The nitro derivatives of naphtho[2,1-*b*]furans have been extensively surveyed for their mutagenic activities, for example, 7-methoxy-2-nitronaphtho[2,1-*b*]furan (**IV**). The genotoxicity of (R7000) as well as that of other nitrofurans, is because of the presence of the nitro group, actively reduced in bacteria by endogenous nitroreductases, (R7000) is one of the strongest mutagens described for mammalians.[1]

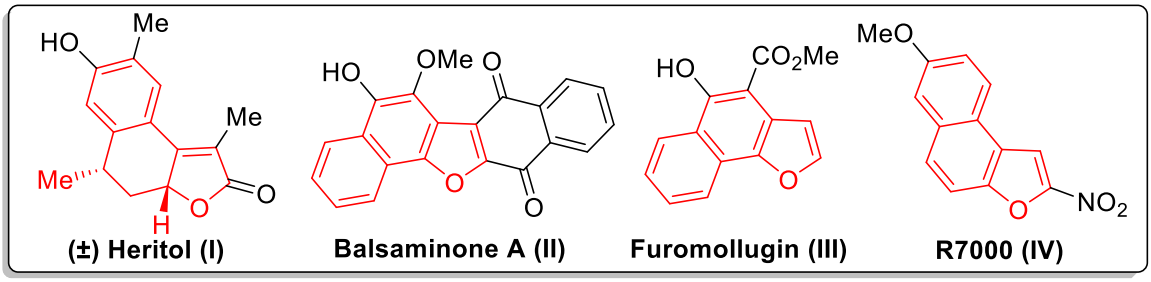
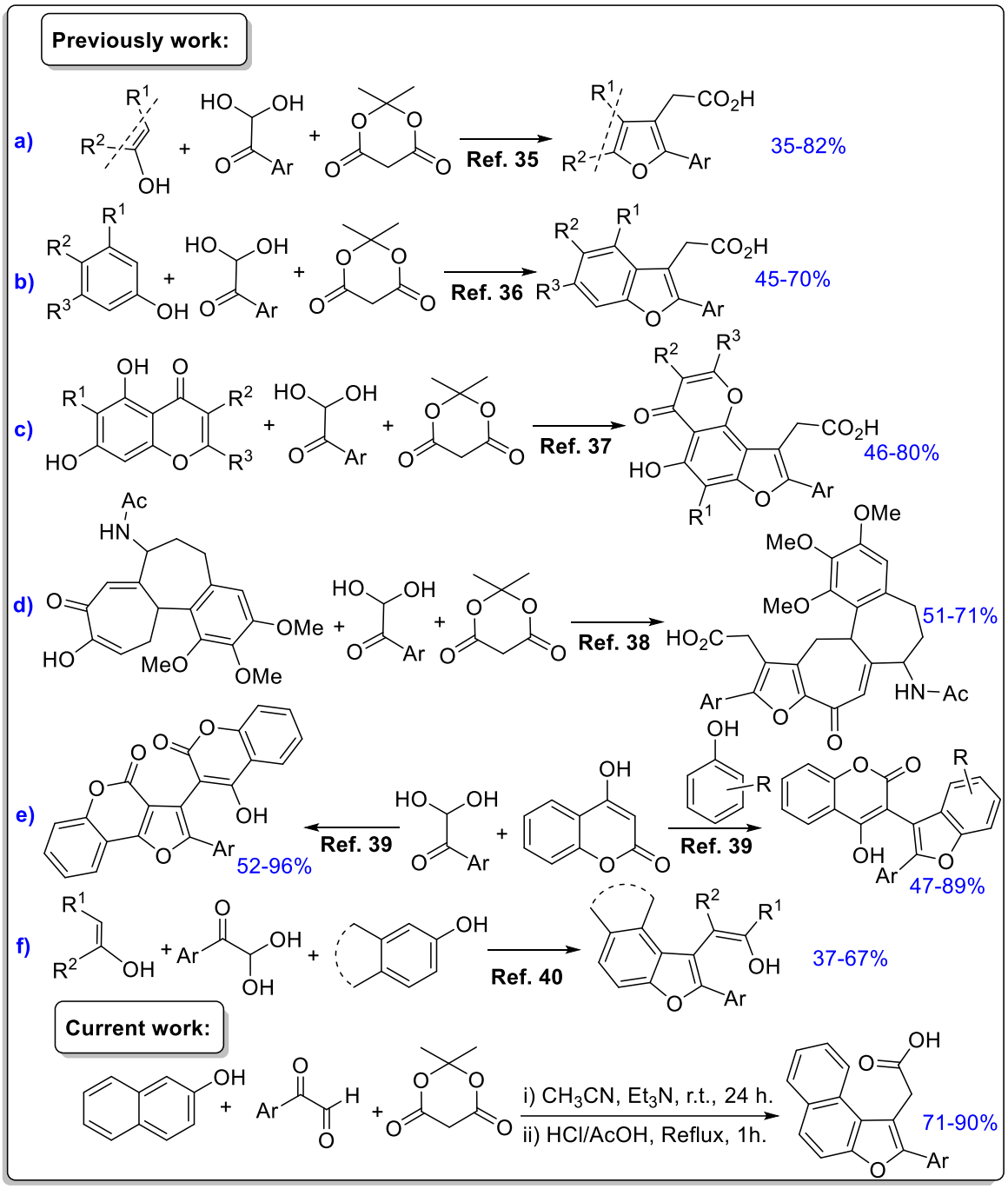


Figure 1. Examples of biologically important natural products containing of naphthofurans.

Since the naphthofuran scaffold displays several biological activities, the synthesis of this group of compounds has been realized *via* several routes, including various metal-catalyzed,[11–13] multicomponent methods,[14–16] carbon–oxygen bond formation reactions,[17] photochemical cyclizations,[18,19] Wittig reactions,[20] various catalysts,[21,22] cyclization reactions,[23,24] Diels–Alder cycloadditions,[25] and various other methods.[26–30] No doubt, all the existing methods are useful and effective, but have significant limitations such as, the use of an expensive catalyst, a special apparatus, low yields, difficult work-up, or a multistep synthetic sequence. In continuation of our attempts to develop novel synthetic routes for the preparation of oxygen-containing organic compounds, using multicomponent approaches,[31–34] we describe herein a simple and efficient approach for the synthesis naphtho[2,1-*b*]furans **4** through an one-pot three-component reaction of Meldrum's acid **1**, arylglyoxal **2**, and a β-naphthol **3** in a two-stage processes.

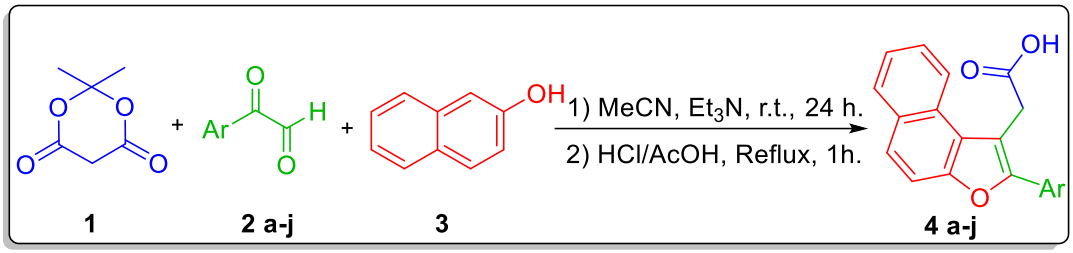
Results and Discussion

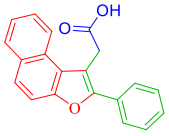
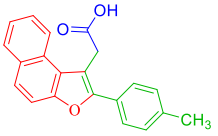
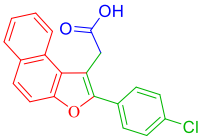
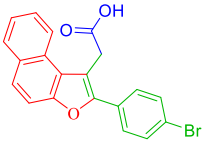
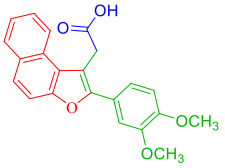
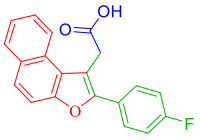
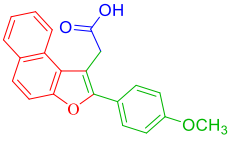
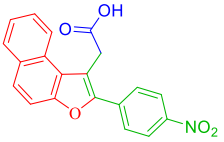
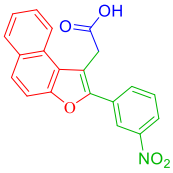
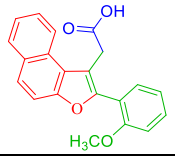
To initiate our study, the reaction of Meldrum's acid **1** (2.25 mmol), arylglyoxals **2** (2.50 mmol), and β-naphthol **3** (1.50 mmol) was investigated as the model reaction in the presence of Et_3N (3.75 mmol) for 24 hours in 5.0 mL of CH_3CN, and then for 1 hour in 5.0 mL of the mixture of hydrochloric acid and acetic acid. Based on previous works, it was illustrated that similar synthesis of furan derivatives (Scheme 1a-f) using cyclic dicarbonyl compounds, arylglyoxals, and enols are two-stage processes.[35-39] It should be noted that, triethylamine was chosen as a base for the studied method. In addition, a mixture of HCl/AcOH is the optimal reaction medium for the second stage of the process.



Scheme 1. Synthesis of furan derivatives.

Table 1. Synthesis of 2-(2-(aryl)naphtho[2,1-*b*]furan-1-yl)acetic acid derivatives

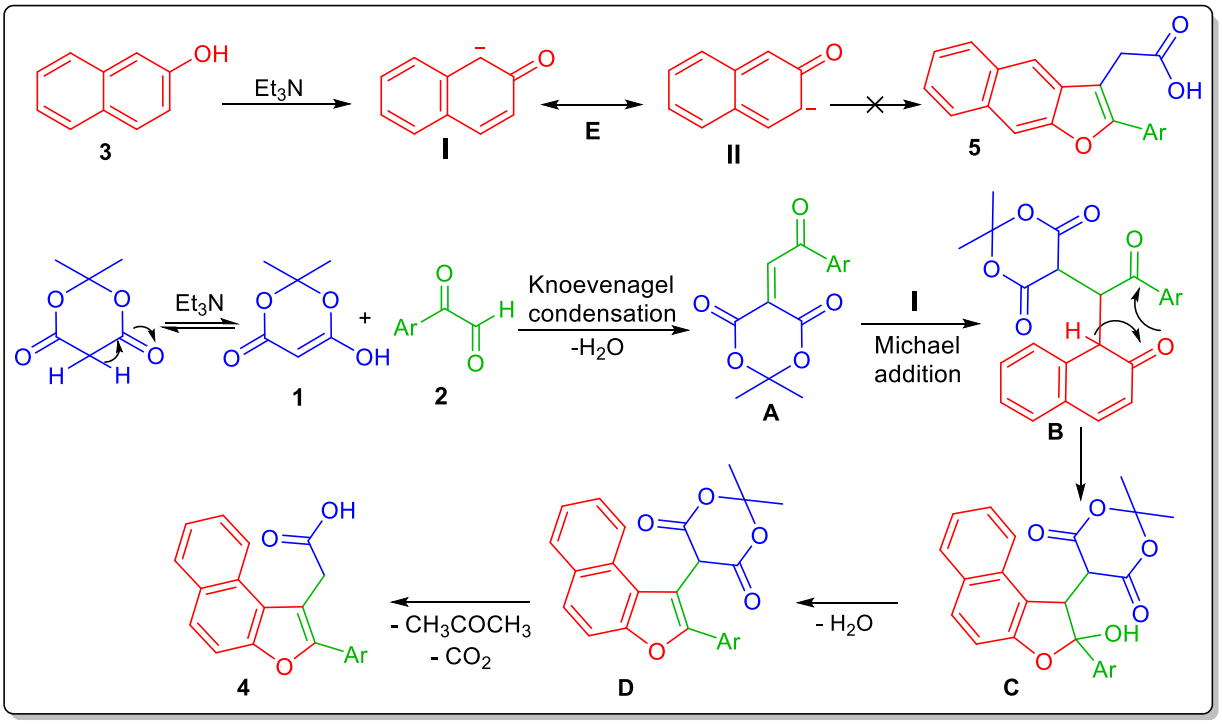


Compound	Ar	Product	Yield (%)[a]
4a	C_6H_5		80
4b	4-$CH_3C_6H_4$		77
4c	4-ClC_6H_4		85
4d	4-BrC_6H_4		84
4e	3,4-$(OCH_3)_2C_6H_3$		71
4f	4-FC_6H_4		87
4g	4- $OCH_3C_6H_4$		73
4h	4-$NO_2C_6H_4$		90
4i	3-$NO_2C_6H_4$		88
4j	2-$OCH_3C_6H_4$		75

[a] Isolated yields

To explore the scope of the transformation, different arylglyoxals were reacted with Meldrum's acid, and β-naphthol under the same conditions, and the results are summarized in Table 1. As can be seen from Table 1, the nature of the arylglyoxal resulted in products with different reaction yields. When the arylglyoxal derivatives containing electron-withdrawing groups (for example, halide and nitro) were employed, a higher yield was obtained.

All the synthesized compounds, previously unknown to the best of our knowledge, were characterized by IR, ^{1}H and ^{13}C NMR, CHN analysis, and melting points. The IR spectrum of 2-(2-phenyl-naphtho[2,1-*b*]furan-1-yl)acetic acid **4a** showed an absorption band at 3419 cm^{-1} for OH, and 1700 cm^{-1} for C=O group. For instance, the ^{1}H NMR spectrum of compound **4a** consisted of one singlet at δ= 4.17 ppm for the two hydrogens of the methylene group. The aromatic protons resonated in the region δ= 7.47–8.35 ppm, and a broad singlet for OH was observed at δ = 12.87 ppm. The ^{13}C NMR spectrum of compound **4a** showed 18 distinct signals in agreement with the proposed structure. Spectral information of other products is given in the experimental section.



Scheme 2. Proposed mechanism for the reaction.

A proposed mechanism for the formation of 2-(2-(aryl)-naphtho[2,1-*b*]furan-1-yl)acetic acid **4** is presented in Scheme 2. Initially, the triethylamine base abstracts the acidic proton of Meldrum's acid and forms the enol form **1**. Then, an intermediate **A** is prepared from the Knoevenagel condensation between the enol form **1** and arylglyoxal **2**. Then, Michael addition of the β-naphthol anion **E** (as Michael donor) to the α,β-unsaturated γ-dicarbonyl compound **A** (as Michael acceptor) provides intermediate **B**, which subsequently undergoes an intramolecular nucleophilic addition to form intermediate **C**. Then, intermediate **D** is formed by elimination of H_2O. Finally, Further cleavage of the Meldrum's acid fragment proceeded in the acidic medium (HCl/AcOH) and was followed by elimination of CO_2 and acetone leading to the formation of the target product **4**. On the other hand, it is important to note that the reaction can lead to the formation of two isomeric 2-(2-(aryl)-naphtho[2,1-*b*]furan-1-yl)acetic acid **4**, and 2-(2-(aryl)-naphtho[2,3-*b*]furan-3-yl)acetic acid **5** as a result of the addition at different positions of the β-naphthol anion **E**. However, in all cases only naphtho[2,1-*b*]furan derivatives **4** were

obtained and we did not detect the presence of isomer **5**. It should be noted that the isomeric product **5** is not formed due to resonance effects. Usually nucleophilic addition occurs more readily at the I position than at the II position because the intermediate for I-addition is more stable than that for II-addition. Presumably, the reason is that the most favorable resonance structures for either intermediate are those that have one fully aromatic ring.

Conclusions

In summary, we have designed an efficient, and simple procedure for the construction of 2-(2-(aryl)naphtho[2,1-*b*]furan-1-yl)acetic acid derivatives *via* a one-pot three-component reaction of Meldrum's acid, arylglyoxals, and β-naphthol in the presence of triethylamine in a two stage process. This method has the advantages of the availability of the starting materials, negligible formation of by-products, simple isolation and purification, and excellent yields benign procedure compared with the reported protocols.

Experimental Section

General. All commercially available reagents and other solvents were purchased from Aldrich or Merck and used without further purification. Melting points were obtained on a Kruss Optronic KSP1N automatic melting point apparatus and are uncorrected. IR spectra were acquired on a Bruker FT-IR Equinox-55 spectrometer. Peaks are reported in wavenumbers (cm^{-1}). All of the NMR spectra were recorded on a Varian model UNITY Inova 500 MHz (^1H: 500, ^{13}C: 125 MHz) NMR spectrometer. Chemical shifts of ^1H and ^{13}C-NMR are reported in parts per million (ppm) from tetramethylsilane (TMS) as an internal standard in DMSO-d_6 as solvents. Elemental analysis were done by Carlo Erba EA 1108 instrument.

General procedure for the synthesis of 2-(2-(aryl)-naphtho[2,1-*b*]furan-1-yl)acetic acid 4a–j. A mixture of Meldrum's acid (**1**) (2.25 mmol), arylglyoxal (**2**) (2.50 mmol), and β-naphthol (**3**) (1.50 mmol) was stirred in CH_3CN (5.0 mL) in the presence of (3.75 mmol) of Et_3N as base. The mixture was stirred at room temperature for 24 hours, then, (2.5 mL:2.5 mL) HCl/AcOH was added, and obtained mixture was stirred for 1 hour at reflux conditions. After completion of the reaction, determined by TLC, the solvent was removed under reduced pressure and the resulting crude product was purified by simple filtration and washing with cold ethanol to give the pure compounds **4a–j** (71–90%).

2-(2-Phenylnaphtho[2,1-*b*]furan-1-yl)acetic acid (4a). White solid; yield 80%; mp: 236-238 °C. IR: 3419, 1700 cm^{-1}. ^1H NMR: δ 4.17 (s, 2H, CH_2), 7.48 (t, 1H, *J* 7.5 Hz, ArH), 7.53 (t, 1H, *J* 7.5 Hz, ArH), 7.58 (t, 2H, *J* 8.0 Hz, ArH), 7.64 (t, 1H, *J* 8.0 Hz, ArH), 7.80 (d, 2H, *J* 8.0 Hz, ArH), 7.83 (d, 1H, *J* 9.0 Hz, ArH), 7.88 (d, 1H, *J* 9.0 Hz, ArH), 8.07 (d, 1H, *J* 8.0 Hz, ArH), 8.34 (d, 1H, *J* 8.0 Hz, ArH), 12.87 (bs, 1H, OH) ppm. ^{13}C NMR: δ 32.4, 112.1, 112.7, 123.0, 123.2, 124.9, 126.5, 126.9, 127.7, 128.3, 129.2, 129.5, 129.6, 130.2, 131.0, 151.7, 152.5, 172.6 ppm.
Anal. Calcd for $C_{20}H_{14}O_3$ (302.33): C, 79.46; H, 4.67; Found: C, 79.14; H, 4.58%.

2-(2-(*P*-tolyl)naphtho[2,1-*b*]furan-1-yl)acetic acid (4b). White solid; yield 77%; mp: 256-257 °C. IR: 3425, 1700 cm^{-1}. ^1H NMR: δ 2.36 (s, 3H, CH_3), 4.12 (s, 2H, CH_2), 7.36 (d, 2H, *J* 8.0 Hz, ArH), 7.50 (t, 1H, *J* 8.5 Hz, ArH), 7.61 (t, 1H, *J* 8.5 Hz, ArH), 7.66 (d, 2H, *J* 8.0 Hz, ArH), 7.80 (d, 1H, *J* 8.5 Hz, ArH), 7.85 (d, 1H, *J* 8.5 Hz, ArH), 8.05 (d, 1H, *J* 8.5 Hz, ArH), 8.30 (d, 1H, *J* 8.5 Hz, ArH), 12.90 (bs, 1H, OH) ppm. ^{13}C NMR: δ 21.3, 32.4, 111.5, 112.7, 123.1,

123.2, 124.8, 126.3, 126.9, 127.4, 127.6, 128.2, 129.6, 130.0, 130.9, 138.9, 151.5, 152.7, 172.6 ppm. Anal. Calcd for $C_{21}H_{16}O_3$ (316.36): C, 79.73; H, 5.10; Found: C, 79.52; H, 5.01%.

2-(2-(4-Chlorophenyl)naphtho[2,1-*b*]furan-1-yl)acetic acid (4c). White solid; yield 85%; mp: 251-253 °C. IR: 3450, 1709 cm^{-1}. ^1H NMR: δ 4.14 (s, 2H, CH_2), 7.60-7.62 (m, 3H, ArH), 7.78-7.81 (m, 4H, ArH), 7.87 (d, 1H, *J* 9.0 Hz, ArH), 8.05 (d, 1H, *J* 8.0 Hz, ArH), 8.30 (d, 1H, *J* 8.0 Hz, ArH), 12.94 (bs, 1H, OH) ppm. ^{13}C NMR: δ 32.4, 112.7, 122.9, 123.2, 124.9, 125.0, 126.9, 127.1, 128.2, 129.0, 129.3, 129.6, 129.6, 130.9, 133.9, 151.2, 151.8, 172.4 ppm. Anal. Calcd for $C_{20}H_{13}ClO_3$ (336.77): C, 71.33; H, 3.89; Found: C, 71.68; H, 3.92%.

2-(2-(4-Bromophenyl)naphtho[2,1-*b*]furan-1-yl)acetic acidacid (4d). White solid; yield 84%; mp: 246-248 °C. IR: 3449, 1696 cm^{-1}. ^1H NMR: δ 4.15 (s, 2H, CH_2), 7.52 (t, 1H, *J* 7.0 Hz, ArH), 7.63 (t, 1H, *J* 7.0 Hz, ArH), 7.73-7.77 (m, 4H, ArH), 7.81 (d, 1H, *J* 9.0 Hz, ArH), 7.88 (d, 1H, *J* 9.0 Hz, ArH), 8.05 (d, 1H, *J* 8.0 Hz, ArH), 8.32 (d, 1H, *J* 8.0 Hz, ArH), 12.94 (bs, 1H, OH) ppm. ^{13}C NMR: δ 32.3, 112.7, 122.6, 122.9, 123.2, 125.0, 126.9, 127.1, 128.2, 129.3, 129.5, 129.7, 131.0, 132.5, 151.3, 151.8, 171.4, 172.6 ppm. Anal. Calcd for $C_{20}H_{13}BrO_3$ (381.23): C, 63.01; H, 3.44; Found: C, 62.64; H, 3.47%.

2-(2-(3,4-Dimethoxyphenyl)naphtho[2,1-*b*]furan-1-yl)acetic acid (4e). White solid; yield 71%; mp: 207-209 °C. IR: 3448, 1702 cm^{-1}. ^1H NMR: δ 3.84 (s, 3H, OCH_3), 3.86 (s, 3H, OCH_3), 4.15 (s, 2H, CH_2), 7.16 (d, 1H, *J* 8.5 Hz, ArH), 7.36 (d, 1H, *J* 8.5 Hz, ArH), 7.38 (s, 1H, ArH), 7.53 (t, 1H, *J* 8.0 Hz, ArH), 7.63 (t, 1H, *J* 8.0 Hz, ArH), 7.83 (d, 1H, *J* 9.0 Hz, ArH), 7.86 (d, 1H, *J* 9.0 Hz, ArH), 8.06 (d, 1H, *J* 8.0 Hz, ArH), 8.37 (d, 1H, *J* 8.0 Hz, ArH), 12.87 (bs, 1H, OH) ppm. ^{13}C NMR: δ 32.5, 56.0, 56.1, 110.9, 111.4, 112.7, 112.7, 120.7, 122.8, 123.2, 123.3, 124.8, 126.1, 126.8, 128.2, 129.6, 131.0, 149.4, 150.0, 151.4, 153.0, 173.0 ppm. Anal. Calcd for $C_{22}H_{18}O_5$ (362.38): C, 72.92; H, 5.01; Found: C, 72.83; H, 4.95%.

2-(2-(4-Fluorophenyl)naphtho[2,1-*b*]furan-1-yl)acetic acid (4f). White solid; yield 87%; mp: 239-241 °C. IR: 3449, 1703 cm^{-1}. ^1H NMR: δ 4.14 (s, 2H, CH_2), 7.41-7.45 (m, 2H, ArH), 7.53 (t, 1H, *J* 7.0 Hz, ArH), 7.64 (t, 1H, *J* 7.0 Hz, ArH), 7.82-7.89 (m, 4H, ArH), 8.07 (d, 1H, *J* 8.0 Hz, ArH), 8.34 (d, 1H, *J* 8.0 Hz, ArH), 12.96 (bs, 1H, OH) ppm. ^{13}C NMR: δ 32.3, 112.0, 112.7, 116.5, 122.9, 123.2, 124.9, 126.6, 126.7, 127.0, 128.2, 129.6, 130.0, 131.0, 151.7, 161.7, 163.7, 172.7 ppm. Anal. Calcd for $C_{20}H_{13}FO_3$ (320.32): C, 74.99; H, 4.09; Found: C, 74.51; H, 4.12%.

2-(2-(4-Methoxyphenyl)naphtho[2,1-*b*]furan-1-yl)acetic acid (4g). White solid; yield 73%; mp: 221-224 °C. IR: 3361, 1678 cm^{-1}. ^1H NMR: δ 3.84 (s, 3H, OCH_3), 4.12 (s, 2H, CH_2), 7.14 (d, 2H, *J* 9.0 Hz, ArH), 7.52 (t, 1H, *J* 7.5 Hz, ArH), 7.62 (t, 1H, *J* 7.5 Hz, ArH), 7.73 (d, 2H, *J* 9.0 Hz, ArH), 7.81 (d, 1H, *J* 9.0 Hz, ArH), 7.85 (d, 1H, *J* 9.0 Hz, ArH), 8.06 (d, 1H, *J* 7.5 Hz, ArH), 8.33 (d, 1H, *J* 7.5 Hz, ArH), 12.86 (bs, 1H, OH) ppm. ^{13}C NMR: δ 32.4, 55.8, 110.8, 112.7, 115.0, 122.7, 123.2, 123.2, 124.8, 126.0, 126.8, 128.2, 129.2, 129.6, 130.9, 151.4, 152.8, 160.2, 172.8 ppm. Anal. Calcd for $C_{21}H_{16}O_4$ (332.36): C, 75.89; H, 4.85; Found: C, 75.47; H, 4.77%.

2-(2-(4-Nitrophenyl)naphtho[2,1-*b*]furan-1-yl)acetic acid (4h). White solid; yield 90%; mp: 251-253 °C. IR: 3408, 1709 cm^{-1}. ^1H NMR: δ 4.27 (s, 2H, CH_2), 7.56 (t, 1H, *J* 8.0 Hz, ArH), 7.67 (t, 1H, *J* 8.0 Hz, ArH), 7.86 (d, 1H, *J* 9.0 Hz, ArH), 7.95 (d, 1H, *J* 9.0 Hz, ArH), 8.06-8.10 (m, 3H, ArH), 8.35-8.40 (m, 3H, ArH), 13.01 (bs, 1H, OH) ppm. ^{13}C NMR: δ 32.4, 112.8, 115.5, 122.9, 123.2, 124.8, 125.3, 127.4, 128.0, 128.2, 128.2, 129.8, 131.0, 136.2, 147.2, 150.0, 152.5, 172.3 ppm. Anal. Calcd for $C_{20}H_{13}NO_5$ (347.33): C, 69.16; H, 3.77; N, 4.03; Found: C, 69.44; H, 3.81; N, 4.08%.

2-(2-(3-Nitrophenyl)naphtho[2,1-*b*]furan-1-yl)acetic acid (4i). White solid; yield 88%; mp: 245-248 °C. IR: 3440, 1704 cm^{-1}. ^1H NMR: δ 4.23 (s, 2H, CH_2), 7.56 (t, 1H, *J* 7.5 Hz, ArH), 7.67 (t, 1H, *J* 7.5 Hz, ArH), 7.85-7.89 (m, 2H, ArH), 7.94 (d, 1H, *J* 9.0 Hz, ArH), 8.09 (d, 1H, *J* 8.0 Hz, ArH), 8.25 (d, 1H, *J* 8.0 Hz, ArH), 7.30 (d, 1H, *J* 8.0 Hz, ArH), 8.38 (d, 1H, *J* 8.0 Hz, ArH), 8.61 (s, 1H, ArH), 13.01 (bs, 1H, OH) ppm. ^{13}C NMR: δ 32.3, 112.8, 114.1, 122.0, 122.8, 123.2, 123.6, 125.2, 127.3, 127.5, 128.2, 129.7, 131.0, 131.2, 131.6, 133.5, 148.7, 150.0, 152.2, 172.4 ppm. Anal. Calcd for $C_{20}H_{13}NO_5$ (347.33): C, 69.16; H, 3.77; N, 4.03; Found: C, 69.47; H, 3.68; N, 3.95%. **2-(2-(2-Methoxyphenyl)naphtho[2,1-*b*]furan-1-yl)acetic acid (4j).** White solid; yield 75%; mp: 217-219 °C. IR: 3419,

1709 cm^{-1}. ^1H NMR: δ 3.79 (s, 3H, OCH_3), 3.90 (s, 2H, CH_2), 7.12 (t, 1H, *J* 8.0 Hz, ArH), 7.20 (d, 1H, *J* 8.0 Hz, ArH), 7.49-7.54 (m, 3H, ArH), 7.61 (t, 1H, *J* 8.5 Hz, ArH), 7.79 (d, 1H, *J* 9.0 Hz, ArH), 7.84 (d, 1H, *J* 9.0 Hz, ArH), 8.05 (d, 1H, *J* 8.0 Hz, ArH), 8.23 (d, 1H, *J* 8.0 Hz, ArH), 12.56 (bs, 1H, OH) ppm. ^{13}C NMR: δ 32.2, 55.9, 112.5, 112.8, 113.6, 118.9, 121.0, 122.9, 123.5, 124.6, 125.9, 126.7, 128.3, 129.4, 130.8, 131.5, 131.6, 150.5, 152.1, 157.4, 172.6 ppm. Anal. Calcd for $C_{21}H_{16}O_4$ (332.36): C, 75.89; H, 4.85; Found: C, 75.29; H, 4.73%.

Supplementary Material

IR, ^1H and ^{13}C NMR spectra of compounds **4a-j** are available in the Supplementary material file associated with this paper.

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