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NaIO$_4$-driven oxidative dimerization and Cu(I)-catalyzed oxidative decarbonylation: modular synthesis of 1,2-naphthoquinones and aryl naphtho[2,*b*]furans

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Sodium metaperiodate-mediated oxidative C–C homocoupling of diverse β-naphthols to 1,2-naphthoquinones in an aqueous medium, using 18-crown-6 as an additive, is reported. Subsequent Cu(I) chloride-assisted decarbonylative oxidation of these resulting 1,2-diketones under an oxygen atmosphere affords the corresponding naphthofurans in good yields. These findings demonstrate the effectiveness of orthogonal oxidative protocols in facilitating the efficient synthesis of functionalized biaryl scaffolds from readily available β-naphthols.

Introduction

1,2-Naphthoquinones (NQs) and their functionalized derivatives are important structural motifs commonly found in a wide range of bioactive natural products and functional materials.[1] These NQ-containing natural products exhibit diverse biological activities, including antimicrobial, antidiabetic, anticancer, and anti-inflammatory properties.[2] Another intriguing class of chemical compounds, naphtho[2,*b*]furans, constitute essential structural components in numerous natural products and synthetic compounds.[3] Structurally related derivatives from both 1,2-naphthoquinones and naphthofurans are also widely present in polyketide natural products and play a significant role in imparting different activity profiles to these bioactive natural products (Fig. 1).[4] Binaphthyls, an important class of naphthol derivatives, are traditionally synthesized through transition metal-catalyzed C–C coupling reactions of aryl precursors, typically preactivated as halogenated or metallated derivatives.[5] In recent years, several transition metal-free approaches employing various types of inorganic or organic oxidants and oxidant-free electrochemical methods have been reported for the chemo- and regioselective synthesis of biaryls, including binaphthyl derivatives.[6–8]

As far as the typical oxidative dimerization of 2-naphthols is concerned, different methods have surfaced from time to time (Fig. 1). These include, dimerization triggered by titanium or molybdenum based peroxo complexes[9] (Fig. 2a, i); regio- and enantioselective photoinduced oxidative coupling enabled by a homochiral Ru/Pd heterometallic coordination cage[7e] (Fig. 2a, ii); coupling of naphthols in the presence of $K_2S_2O_8$/CF_3COOH[7d] (Fig. 2a, iii); and hypoiodite/Lewis-acid assisted Brønsted acid (LBA)-*co*-catalysed tandem oxidation/cross-coupling of 1,2-dihydroxynaphthalene and 2-naphthol[7f] (Fig. 2a, iv). Similarly, for the synthesis of naphthofurans, different methods have emerged, including: base mediated intramolecular condensation/decarboxylation of benzophenone acids[10] (Fig. 2b, i); acid-promoted [3 + 2] cascade annulation of 2-naphthols with *Z*-enoate propargylic alcohols[11] (Fig. 2b, ii); cascade annulation of propargyl amines with β-naphthols[12] (Fig. 2b, iii); and cascade intramolecular

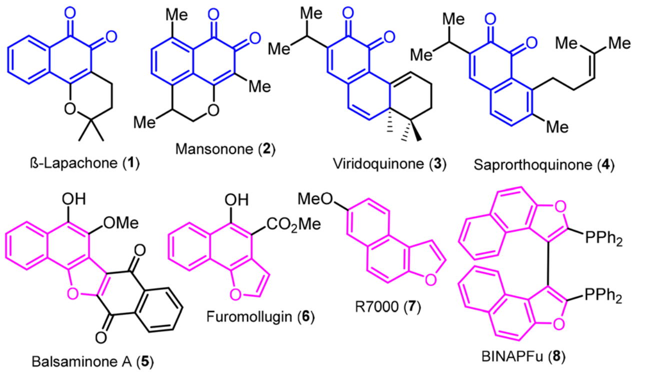

Fig. 1 Representative examples of bioactive natural products harbouring 1,2-naphthoquinone and naphthofuran moieties.

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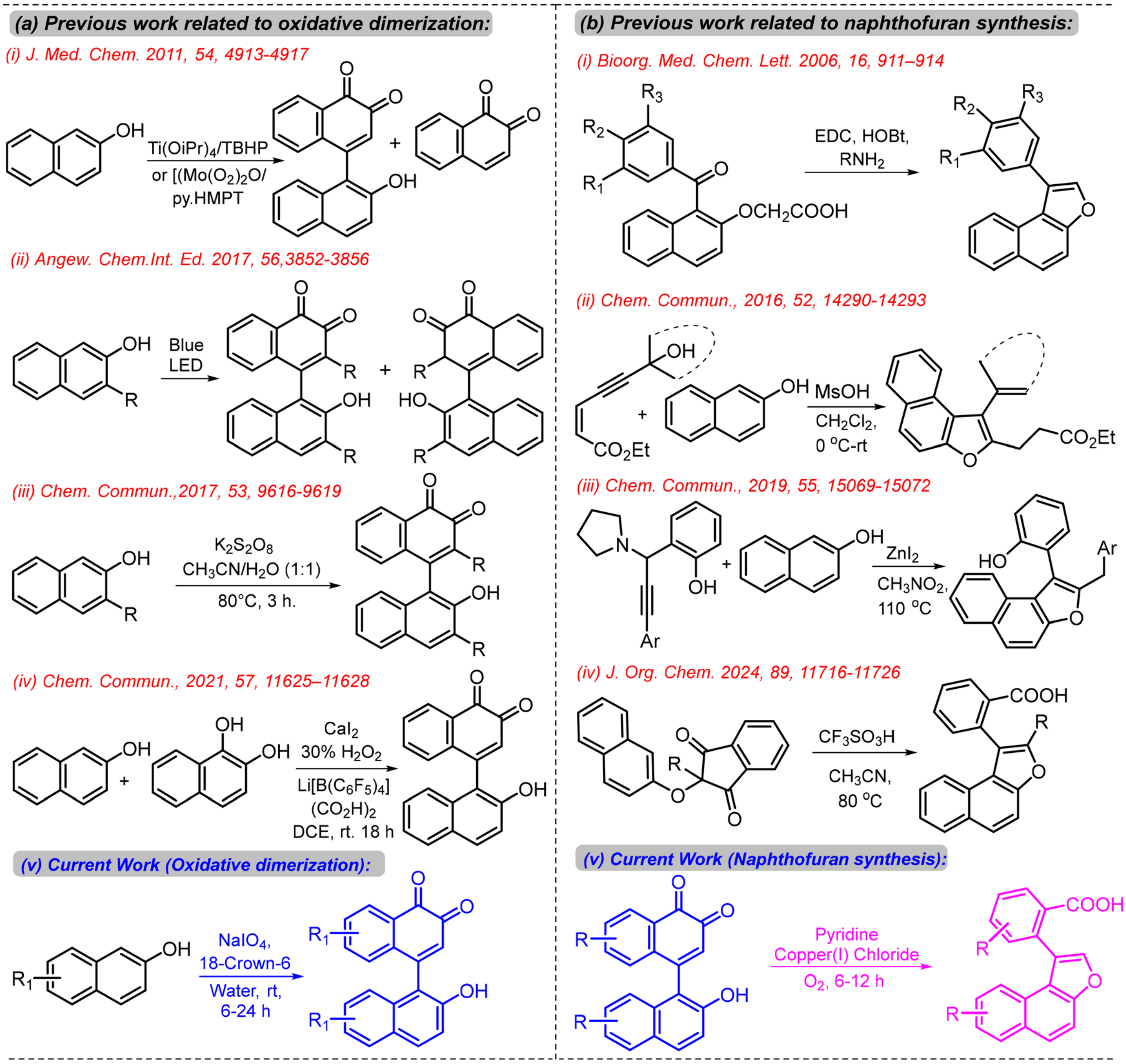


Fig. 2 Recent approaches towards oxidative dimerization of 2-naphthols, naphthofuran synthesis and our work.

Friedel–Crafts reaction, rearrangement, and aromatization sequence catalyzed by Brønsted acid[13] (Fig. 2b, iv).

While the oxidation protocols for 2-naphthols discussed above have proven valuable within their respective domains, their broader applicability across structurally diverse 2-naphthols remains underexplored. This gap presents an opportunity for the development of novel and more versatile methodologies.

In line with our ongoing efforts toward advancing oxidative transformations and functionalization strategies for 2-naphthols, we herein report a $NaIO_4$-mediated, peroxide free, oxidative dimerization approach for substituted 2-naphthols, performed in water with 18-crown-6 as an additive (Fig. 2a, v). Furthermore, we explored a copper(I)-mediated decarbonylative oxidation of the cyclic 1,2-diketones using inexpensive Cu(I)Cl as the catalyst under an oxygen atmosphere, with pyridine as the solvent (Fig. 2b, v). Both these oxidation strategies are operationally simple, compatible with a variety of 2-naphthol substrates, and consistently deliver the desired products in good to excellent yields.

Results and discussion

Given the considerations outlined above, there is a clear need and significant opportunity to develop more efficient and sustainable methods for the oxidative dimerization of 2-naphthols and, more critically, look for the decarbonylative oxidation of cyclic 1,2-diketones – a transformation for which reported strategies remain scarce. In this context, we disclose two efficient oxidative protocols: $NaIO_4$-mediated oxidative dimerization and Cu(I)Cl-mediated decarbonylative oxidation which can be implemented sequentially to afford cyclic 1,2-diketones and naphthofurans, respectively. Our study began with the screening of commonly available oxidants such as $KMnO_4$, Pb$(OAc)_4$, HIO_4, NMO, PCC, PDC *etc.*, using 2-naphthol (**9a**) as the starting material. Initial results showed that some oxidants, notably HIO_4 and *m*-CPBA, led to the decomposition of the substrate, while others were either ineffective or afforded the desired product in suboptimal yields. Specifically, PCC and PDC in a THF : H_2O (1 : 1 v/v) solvent system yielded the target compound in low amounts *via* intermediate **9ai**. Increasing the equivalents of these oxidants led to significant decomposition of both the starting material and the intermediate.

In the presence of $NaIO_4$ under the same reaction conditions, the desired product was obtained in a low yield (27%), but a substantial portion of the starting material was converted into the intermediate. This observation prompted us to focus further on $NaIO_4$ and explore different solvent systems. Reactions in pure aprotic solvents such as THF and CH_2Cl_2 showed no conversion, directing our attention to polar protic solvents. These protic solvents, either alone or in combination with water, gave the desired product, albeit in insufficient yields. Using pure water as the solvent resulted in particularly low yields, likely due to solubility limitations.

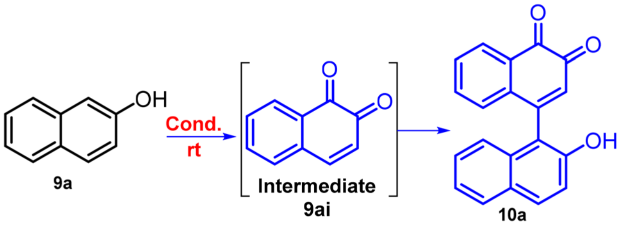
A breakthrough came when 18-crown-6 was employed as an additive in combination with $NaIO_4$ (3 equiv.) in water at room temperature. Under these conditions, the reaction proceeded efficiently without solubility issues, and the desired compound was obtained in a significantly improved yield of 63%. Further attempts to enhance the reaction yields using other additives such as TBAB or micellar solutions to improve solubility and local substrate confinement were unsuccessful. Consequently, we settled with the conditions mentioned in entry 21 (Table 1) as the optimized protocol.

With the standardized reaction conditions in hand, we explored the substrate scope of transformation. A variety of substituted 2-naphthols (**9b–9q**), either commercially available or synthesized *via* Suzuki coupling, were subjected to the reaction. Remarkably, all substrates successfully underwent oxidative dimerization to afford the corresponding biaryl products (**10b–10q**) in moderate to good yields (Table 2). In order to demonstrate the practical applicability of this methodology, two reactions (**9a** and **9m**) were executed at the gram scale (>2 g) and both these experiments proceeded smoothly and furnished the corresponding products (**10a** and **10m**) with negligible changes in yield and reaction profile (Table 2).

Regarding the plausible mechanism of this reaction, the starting material **9a** reacts with $NaIO_4$ to generate intermediate **I**. This intermediate then undergoes an intramolecular oxygen transfer to the most nucleophilic *ortho* position, leading to the formation of intermediate **II**. Subsequent rearrangement of **II** yields the cyclic intermediate **III**. Cleavage of **III**, followed by reaction with another molecule of 2-naphthol furnishes intermediate **IV**. Aromatization of **IV** to **V** followed by final oxidation generates enone, ultimately leading to the formation of the final compound **10a**, as depicted in Scheme 1.

Following our successful results with 2-naphthols, we next investigated the reactivity of 1-naphthol under the optimized reaction conditions. Interestingly, it initially formed the same intermediate (**9ai**) as observed in the 2-naphthol series, which over time converted exclusively to the corresponding naphthoquinone (see the SI).

Table 1 General standardisation of oxidative dimerization reaction



Entry	Oxidant	Solvent/additive	Time (h)	Yield[a] (%)
1	$KMnO_4$ (1 eq.)	THF : H_2O	24	ND
2	$Pb(OAc)_4$ (1 eq.)	—	—	ND
3	HIO_4 (1 eq.)	—	12	IM
4	NMO (1 eq.)	—	24	ND
5	TEMPO (1 eq.)	—	—	ND
6	PCC (1 eq.)	—	6	20%
7	PDC (1 eq.)	—	9	12%
8	SeO_2 (1 eq.)	—	24	ND
9	*m*-CPBA (1 eq.)	—	12	IM
10	$NaIO_4$ (1 eq.)	—	—	27%
11	−(1 eq.)	CH_2Cl_2	24	ND
12	−(1 eq.)	THF	24	ND
13	−(1 eq.)	EtOH : H_2O	10	35%
14	−(1 eq.)	EtOH	20	24%
15	−(1 eq.)	MeOH	15	34%
16	−(1 eq.)	MeOH : H_2O	12	38%
17	−(1 eq.)	CH_2Cl_2 : H_2O	2.5	28%
18	−(1 eq.)	H_2O	24	15%
19	−(1 eq.)	H_2O, 18-crown-6	12	42%
20	−(2 eq.)	H_2O, 18-crown-6	8	55%
21	**−(3 eq.)**	**H_2O, 18-crown-6**	**6**	**63%**
22	−(3 eq.)	H_2O, SDS[b]	9	34%
23	−(3 eq.)	H_2O, TBAB	24	ND
24	−(3 eq.)	H_2O, CTAB[b]	9	41%
25	−(3 eq.)	H_2O, TWEEN-20[b]	20	33%

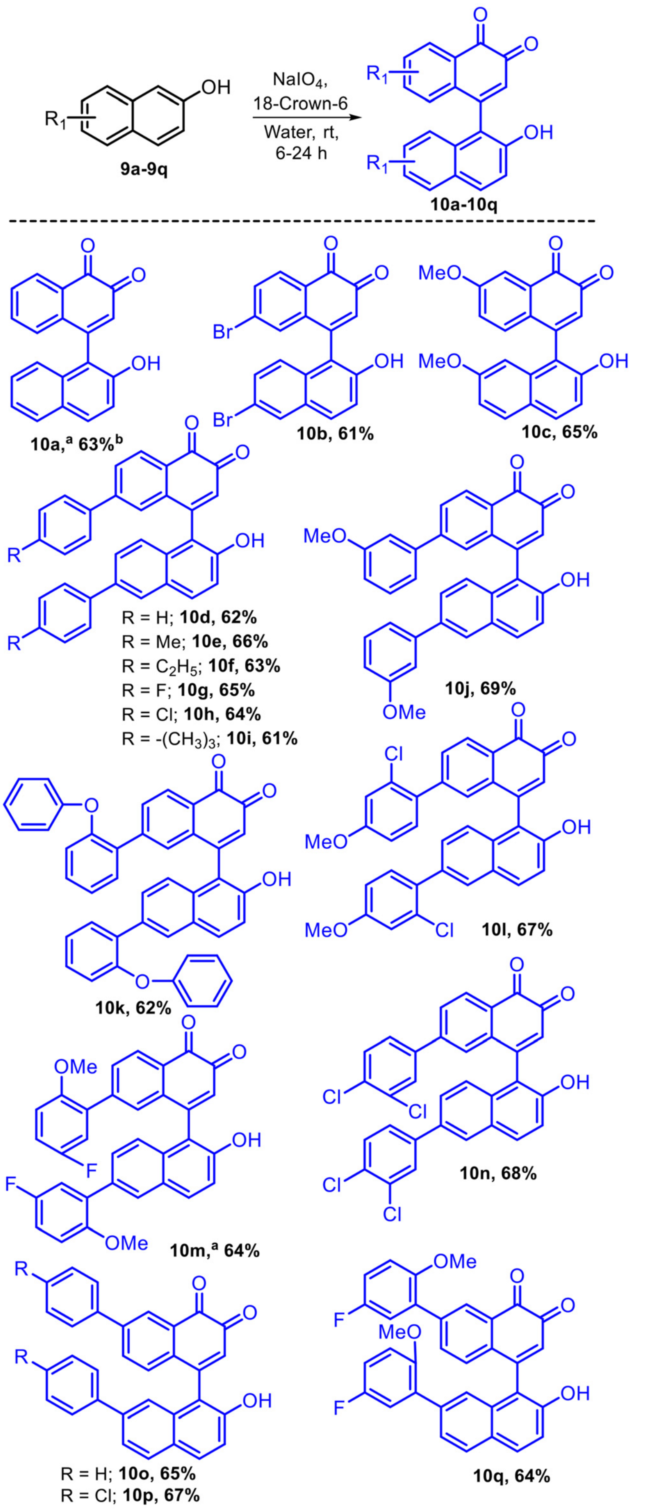
[a] Yields reported are isolated yields. [b] Solvent systems represent micellar solutions above CMG; ND = not detected; IM = intractable mixture.

With access to a diverse set of cyclic diketones, our next objective was to explore the synthetic utility of these interesting scaffolds, focusing primarily on Lewis acid- or metal-catalyzed oxygenation/oxidation strategies. Toward this end, we carried out a detailed optimization of the reaction conditions under molecular oxygen atmosphere with pyridine as the solvent, using the dimerized compound **10a** as the model substrate.

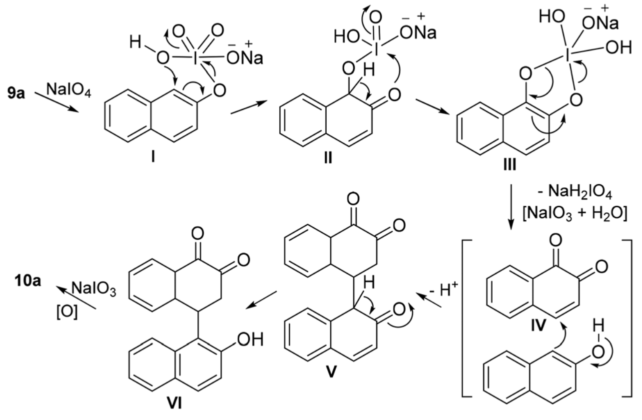
A broad screening of various Lewis acids and metal catalysts was then conducted at room temperature (Table 3). Among the tested catalysts, only Cu(I)Cl proved effective, enabling an oxidative decarbonylation of diketone **10a** to furnish the naphthofuran **11a** in a highly regioselective manner and in good yield of 77%. In contrast, all other catalysts screened failed to produce the desired product. Furthermore, no reaction occurred when solvents other than pyridine were employed, underscoring the critical role of both the metal catalyst and solvent in facilitating this transformation.

To explore the substrate scope of the reaction, a series of dimerized diketones bearing various substituents (**10a–10c**,

Table 2 General standardisation of the oxidative dimerization reaction

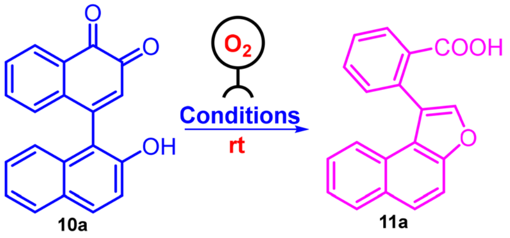


[a] Yields reported are isolated yields. [b] **10a** and **10m** were synthesized at a >1.0 g scale.



Scheme 1 Plausible mechanism of oxidative dimerization.

Table 3 General standardisation of the decarbonylative oxidation reaction

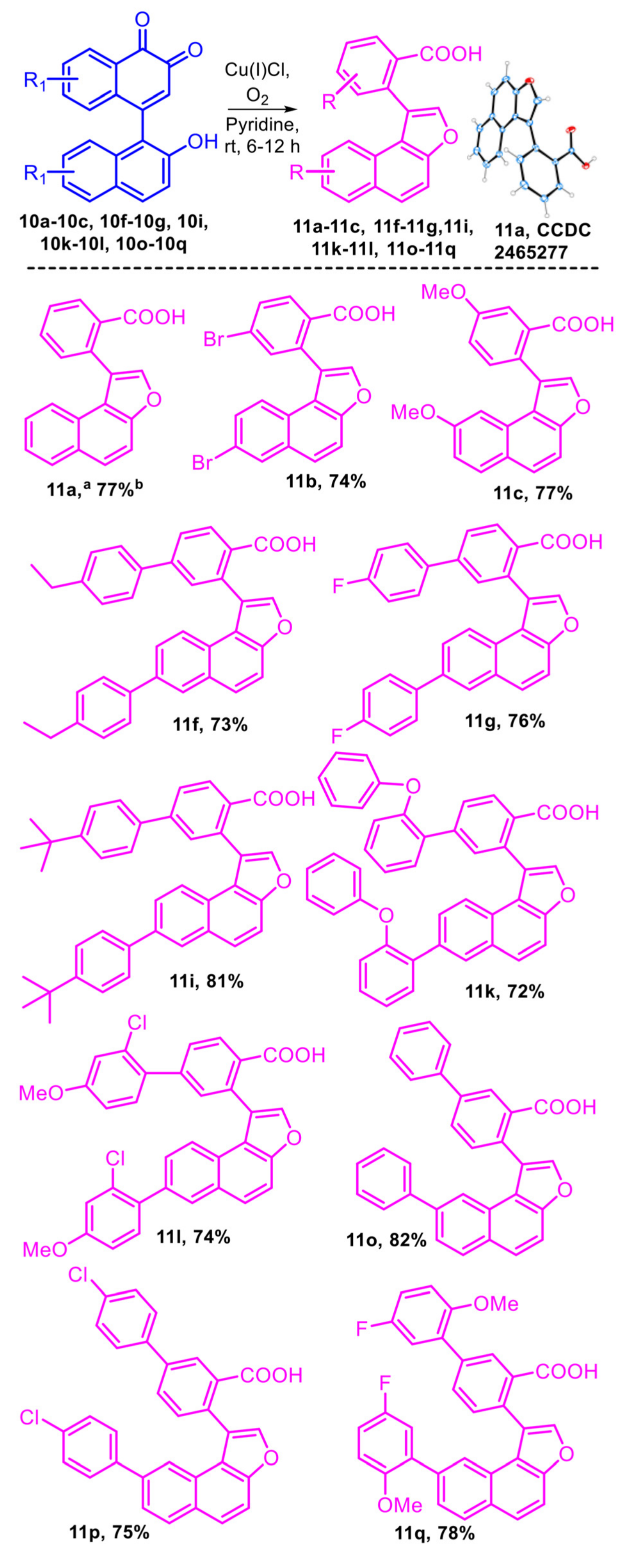


Entry	Catalyst	Solvent	Time (h)	Yield[a] (%)
1	$FeCl_3$	Pyridine	30	ND
2	$NiCl_2$	—	—	ND
3	$CuBr_2$	—	—	ND
4	$Cu(OTf)_2$	—	—	ND
5	Cu(I)Br	—	18	25%
6	$Cu(OAc)_2$	—	—	ND
7	$CuCl_2$	—	24	ND
8	$ZnCl_2$	—	—	ND
9	$Pd(OAc)_2$	—	—	ND
10	$PdCl_2$	—	—	ND
11	$CoCl_2$	—	—	ND
12	$FeCl_2$	—	—	ND
13	$SnCl_2$	—	—	ND
14	$CeCl_3$	—	—	ND
15	$Sc(OTf)_2$	—	—	ND
16	$Yb(OTf)_2$	—	—	ND
17	**Cu(I)Cl**	—	**6**	**77%**
18	—	Xylene	24	ND
19	—	DME	—	ND
20	—	DMF	—	ND
21	—	Toluene	—	ND
22	—	DCM	—	ND
23	—	Chlorobenzene	—	ND

[a] Yields reported are isolated yields; ND = not detected; IM = intractable mixture.

10f–10g, **10i**, **10k–10l**, and **10o–10q**) were subjected to the optimized reaction conditions. Gratifyingly, all substrates underwent the transformation smoothly, affording the corresponding naphthofurans (**11a–11c**, **11f–11g**, **11i**, **11k–11l**, and **11o–11q**) in good yields. The structures of the resulting compounds were unambiguously confirmed, with the structure of compound **11a** being validated through single-crystal X-ray diffraction analysis (Table 4). Pertinent to mention, acyclic

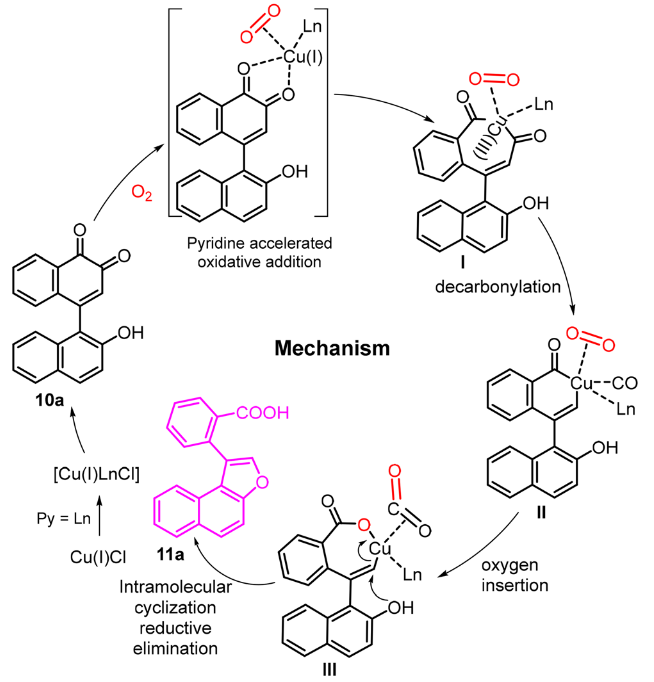
Table 4 Substrate scope for decarbonylative oxidation



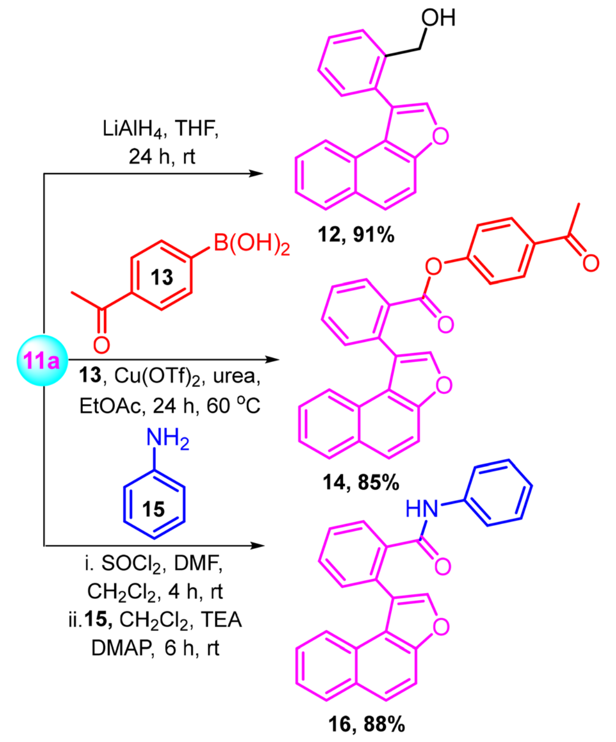
[a] Yields reported are isolated yields. [b] **11a** was synthesized at a >0.5 g scale.

diketones like benzils and phenanthridine dione, under similar reaction conditions, resulted in the corresponding carboxylic acids, see the SI.[14]

Mechanistically, the transformation is proposed to proceed *via* a pyridine-accelerated oxidative addition, leading to the formation of metallacycle **I**. This intermediate then undergoes sequential decarbonylation to generate species **II**, followed by a disproportionative oxygen insertion to afford intermediate



Scheme 2 Plausible reaction pathway for decarbonylative oxidation.



Scheme 3 Synthetic utility of naphthofuran acids.

III. Finally, intramolecular cyclization triggered by free hydroxyl followed by reductive elimination in **III** furnishes the target compound **11a** (Scheme 2).[15]

To further illustrate the synthetic versatility of the biaryl acid derivatives, compound **11a** was subjected to a series of functional group transformations. Reduction of the carboxylic acid moiety in **11a** using $LiAlH_4$ proceeded smoothly to furnish the corresponding primary alcohol **12** in excellent yield[16] (91%). Subsequent esterification of **11a** with boronic acid **13**, under well-established reaction conditions, afforded ester **14** in 85% yield.[17] Additionally, amide coupling of **11a** with aniline **15**, employing slightly modified reaction conditions, delivered the corresponding amide **16** in 88% yield[18] (Scheme 3).

Conclusions

In conclusion, we have developed an efficient two-step oxidative strategy for converting readily available β-naphthols into valuable aryl naphthofuran scaffolds *via* cyclic 1,2-diketones. The first step involves a sodium metaperiodate-mediated oxidative homocoupling of β-naphthols in aqueous medium, promoted by 18-crown-6, to generate 1,2-naphthoquinones. This is followed by a Cu(I) chloride-catalyzed oxidative decarbonylation under an oxygen atmosphere, affording the corresponding aryl naphthofurans in good yields. These orthogonal oxidative protocols offer a practical and modular approach for synthesizing functionalized naphthofurans from simple 2-naphthol precursors.

Experimental section

General procedure for the synthesis

(a) Substituted 1,2-naphthoquinones (10a–10q). A round-bottomed flask equipped with a magnetic stir bar was charged with 2-napthol, **9a** (800 mg, 5.549 mmol, 1.0 equiv.) and 18-crown-6 (293.00 mg, 1.11 mmol, 0.2 equiv.) in water (40 mL). The reaction mixture was stirred at room temperature for 10 minutes, followed by the addition of sodium periodate (3.5 g, 16.65 mmol, 3 equiv.) and was allowed to stir at room temperature for 6 hours. The progress of the reaction was monitored by TLC. After completion, the reaction mixture was quenched with water and extracted with ethyl acetate (30 mL × 3). The combined organic layer was dried over anhydrous Na_2SO_4, concentrated under reduced pressure and subjected to column chromatography using hexanes : ethyl acetate (80 : 20, v/v) as an eluent to afford product **10a** (525 mg, 63% yield). A similar procedure was followed for the synthesis of remaining compounds **10b–10q** (61–69% yield).

(b) Substituted naphtho[2,*b*]furans. To a stirred solution of **10a** (500 mg, 1.665 mmol, 1.0 equiv.) in pyridine (15 mL), copper(I) chloride (165 mg, 1.665 mmol, 1.0 equiv.) was added. The reaction mixture was stirred at room temperature under oxygen for 6 hours. The completion of reaction was monitored by TLC. After completion, the reaction mixture was quenched with a saturated aqueous solution of copper(II)sulfate and extracted with ethyl acetate (15 mL × 3). The combined organic layer was dried over anhydrous sodium sulphate and concentrated under reduced pressure to obtain a crude mixture, which was further purified by column chromatography using hexanes : ethyl acetate (70 : 30, v/v) as an eluent to afford the desired product **11a** (370 mg, 77%).

A similar procedure was followed for the synthesis of compounds **11b**, **11c**, **11f–11g**, **11i**, **11k–11l**, and **11o–11q** (72–82% yield).

Author contributions

SR conceptualized the work and wrote the manuscript. MA, GAR, ART and WIL did the experimental work and compiled the SI.

Conflicts of interest

There are no conflicts to declare.

Data availability

All the analytical data (NMR and XRD) related to this publication is available in the SI.

Supplementary information contains the experimental details and characterization data of all the new compounds, including copies of ^1H and ^{13}C NMR spectra. See DOI: **https://doi.org/10.1039/d5ob01064k**.

CCDC 2465277 contains the supplementary crystallographic data for this paper.[19]

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