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COMMUNICATION

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Rapid, operationally simple, and metal-free NBS mediated one-pot synthesis of 1,2-naphthoquinone from 2-naphthol

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Abstract. A metal-free, one-pot synthesis of 1,2-naphthoquinone was accomplished from 2-naphthol by utilizing economically cheap NBS under open air conditions. Initial formation of 1,1-dibromonaphthalen-2-one and subsequent transformation afforded the 1,2-naphthoquinone. This oxidation was completed within 30 min and had broad substrate scope. Moreover, this system tolerated heterocyclic systems and was also applicable to 1,3-dicarbonyl systems. This practical approach with short reaction times, a simple workup, and insensitivity to moisture could override the usage of expensive and hazardous oxidizing and metal reagents.

Keywords: Naphthoquinone(s), Metal-free, Oxidation, NBS, Vicinal tricarbonyls, Chemoselective

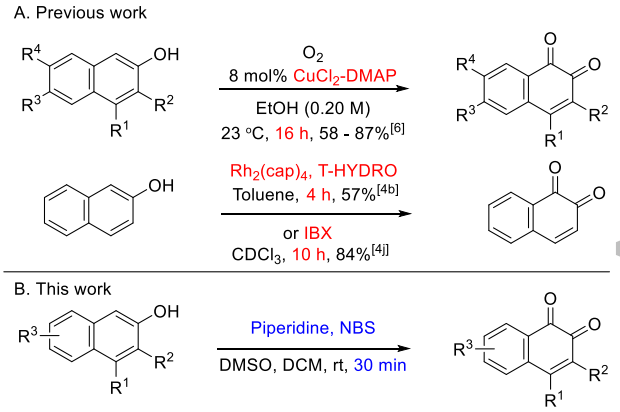
Introduction

Naphthoquinones, one of many valuable scaffolds distributed in plants, fungi, etc., have received wide attention and a large scope of application in organic and medicinal chemistry. They were found to exhibit a wide range of biological activities including antibacterial, antiviral, trypanocidal, and antifungal activities.[1]

Figure 1: 1,2-NQ containing natural products

1,2-Naphthoquinone (1,2-NQ), in particular, has received much attention due to its high affinity to form covalent bonds with protein and DNA.[2] There are several natural products that contain 1,2-NQ as a core structure and display valuable biological activities (Figure 1). In addition to these, the 1,2-NQ core plays key roles as a catalyst in oxidative transformation reactions[3a] and as a highly reactive intermediate for the construction of heterocyclic compounds.[3b]

Thus, it is not surprising that several elegant works have been developed for the synthesis of this privileged scaffold.[4a] 1,2-NQs were usually prepared from 1- and 2-naphthols through the oxidative (dearomatization) reaction.[4b-g] Most of the common approaches included using excess hypervalent iodine reagents (e.g. IBX,[4h-j] DMP,[4k-l] and PIFA[4m]), TBHP,[5a] oxone[5b] or the expensive Fremy's salt.[5c] Recently, Oh et al., reported that copper-catalyzed synthesis of 1,2-NQ under aerobic oxidation required 16 h to complete.[6] However, the aforementioned methodologies suffer from some drawbacks, including high costs of the reagent, elevated temperatures, and/or longer reaction times (Scheme 1). As a result, development of the novel method using mild, readily available, and cheaper reagent is highly desirable.



Scheme 1: Previous work and our approach on synthesis of 1,2-NQ

Oxidative dearomatization reactions are widely utilized in organic synthesis since several natural

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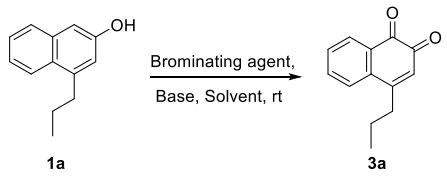
products contain unaromatized rings in their core structure.[7] As such, *N*-bromo succinimide (NBS) represents a more user-friendly oxidative, and non-toxic reagent widely utilized in laboratories and industries.[8] In general, NBS is used for bromination,[9a-d] and oxidative dearomatization reactions,[4h,9e] not only because it is very cheap, but also because its side product can be easily removed. As outlined in the Scheme 1, we envisioned that this privileged structure could be accessed through sequential NBS-mediated bromination followed by oxidation in the presence of piperidine as a base.

Results and Discussion

To pursue our goal, we selected 4-alkyl naphthol **1a**[6], as a model substrate and used 3.0 equiv. of NBS in DMSO at room temperature under open air conditions to discover the suitable condition. 1,2-NQ was obtained in 10% yield (Table 1, entry 1). The influences of the other bases were also investigated (entries 3-7). However, bases such as pyrrolidine, Et_3N, DABCO, and DMAP were ineffective and led to side products. Interestingly, when using 7.0 equiv. of piperidine, an improved yield (68%, entry 9) was observed. Subsequently, the screenings of NBS analogues and other halogen sources were also carried out (entries 10-13). Next, the screening of a range of solvents was investigated (entries 14-16). To our delight, a 90% isolated yield was achieved in a DMSO/DCM (1:2) (entry 16). Varying the amount of NBS and/or piperidine was proved to be ineffective (entries 17-20). Based on all the above entries, we concluded that the optimal condition was: 1.0 equiv. of **1a**, 3.0 equiv. of NBS, and 7.0 equiv. of piperidine in 0.06M DMSO/DCM (1:2).

To examine the scope of this protocol, the optimized condition was applied to various 2-naphthol derivatives. As shown in Table 2, this oxidation reaction exhibited a wide scope of substituted 2-naphthols and produced the corresponding products in good to excellent yields. When the mono-alkyl group was substituted in the 4th position of the 2-naphthol, the reaction produced good to excellent yields (**3a-g**). Substituted phenyl ring was also tolerated, leading to the corresponding products in high yields (**3h-k**). In particular, the halogen-substituted phenyl ring had relatively excellent yields (**3l-n**). Similarly, 3,4-di-substituted naphthol substrates did not lead to any negative impacts on the reaction (**3o-r**). However, the position of the substituent significantly affected the reaction yield: only 33% yield was obtained with the 6-chloro substituted naphthol (**3s**). On the other hand, 2-naphthols with electron donating methyl group (as a mixture of isomers) successfully undergoes this oxidation reaction and delivered 1,2-NQs **3u-v** in very high yields (90% for **3u** and 97% for **3v**). It should be noted that the reaction conditions could accommodate various functional groups such as ester and halide; these groups can be utilized for further synthetic elaborations. In addition, 9-phenanthrol was applied to this reaction condition, which provided the expected product **3t** in 90% yield.

Table 1. Optimization of reaction conditions[a]



1a **3a**

[a]Unless otherwise stated, the reaction condition: Naphthol

Entry	Brominating agent	Base	Solvent	Yield (%)[b]
1	NBS	-	DMSO	10
2	NBS	K_2CO_3[c]	DMSO	16
3	NBS	Pyrrolidine[c]	DMSO	SR[d]
4	NBS	Et_3N[c]	DMSO	SR[d]
5	NBS	Pyridine[c]	DMSO	13
6	NBS	DABCO[c]	DMSO	SR[d]
7	NBS	DMAP[c]	DMSO	SR[d]
8	NBS	Piperidine[c]	DMSO	25
9	NBS	Piperidine	DMSO	68
10	Br_2-dioxane	Piperidine	DMSO	45
11	DBDMH	Piperidine	DMSO	10
12	NCS	Piperidine	DMSO	SR[d]
13	NIS	Piperidine	DMSO	17
14	NBS	Piperidine	DMSO/THF	51
15	NBS	Piperidine	DCM	72
16	NBS	Piperidine	DMSO/DCM[e]	90
17	NBS[f]	Piperidine	DMSO/DCM[e]	28
18	NBS[g]	Piperidine	DMSO/DCM[e]	73
19	NBS	Piperidine[c]	DMSO/DCM[e]	58
20	NBS	Piperidine[h]	DMSO/DCM[e]	83

1a (0.21 mmol), bromination agent (3.0 equiv.) and base (7.0 equiv.) in solvent (0.18 M) at room temperature for 30 min under open air conditions. [b]Isolated yield. [c]5.0 equiv. of base was used. [d]SR: Side Reaction. [e]Solvent ratio: 0.06 M (DMSO:DCM= 1:2). [f]2.5 equiv. of NBS was used. [g]4.0 equiv. of NBS was used. [h]10.0 equiv of base was used.

We next focused on utilizing unsubstituted 2-naphthol and 4-phenyl-1-naphthols as shown in Scheme 2. The corresponding products were obtained in low yield, highly likely due to the Michael addition at the 4th position of unsubstituted 2-naphthol **1w** and non-selective bromination of 1-naphthol, respectively. For example, 4-phenyl-1-naphthol, derived from the 4-bromo-1-naphthol using Sonogashira coupling, provided the desired 1,2-NQ **3h** in 38% yield. Tactically, we hypothesized a masking (at the 4th position) to prevent the Michael addition, allowing us to deliver the 1,2-NQ.[4h,6]

The benzylic position is highly prone to oxidation and delivers the corresponding oxidation compounds

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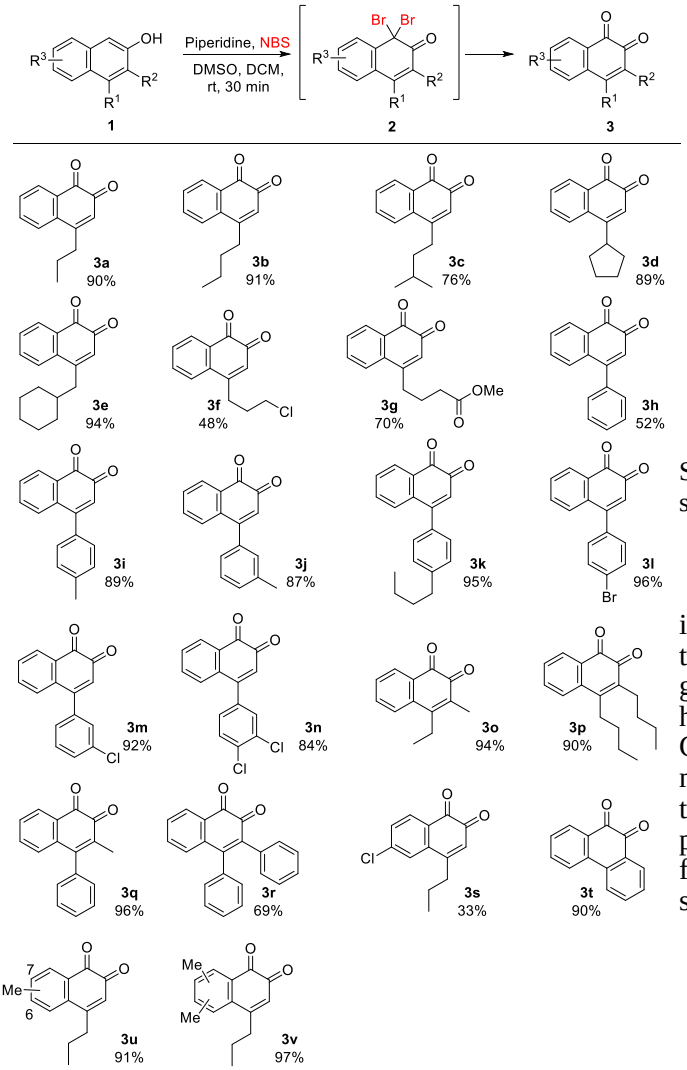
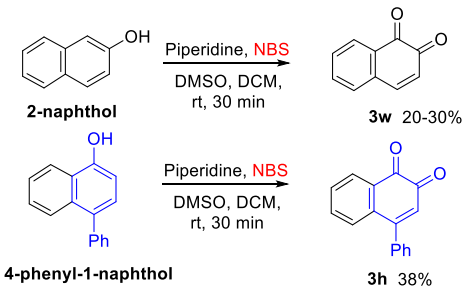
in the presence of NBS.[10a-c] Intriguingly, when we applied our optimized condition to 4-benzyl-2-naphthol **4a**, the desired 1,2-NQ **5a** was obtained in a chemoselective-manner as shown in Scheme 3. The substrate scope was extended to heterocyclic systems to examine the generality of this reaction. A furan-containing heterocycle **4b**[10d] was successfully oxidized and produced **5b** in good yield. With 6-quinolinol,[10e] chemoselective oxidation also occurred on the phenyl ring without *N*-oxide formation to afford 2-methyl quinoline-5,6-dione (**5c**).

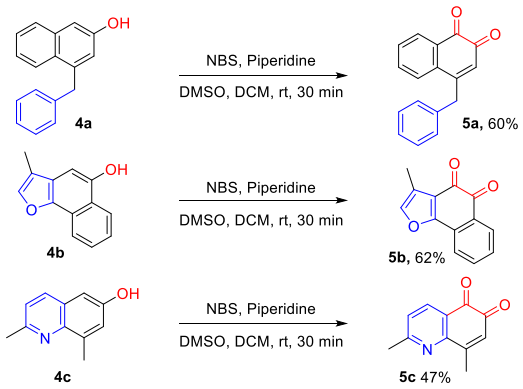
Table 2. Synthesis of 1,2-naphthoquinone derivatives[a]



[a]Reaction condition: Naphthols **1** (0.2 mmol) in DMSO (1.2 mL) and DCM (2.4 mL), piperidine (1.4 mmol, 7.0 equiv.), then NBS (0.6 mmol, 3.0 equiv.) at room temperature for 30 min in open air condition, yields are isolated yields.

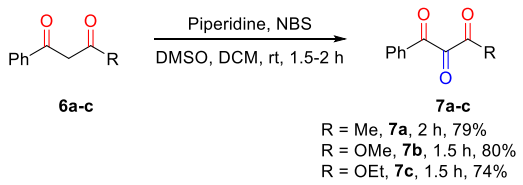


Scheme 2: Reaction with unsubstituted 2-naphthol and 4-phenyl-1-naphthol



Scheme 3: Reaction with benzylic and heterocyclic systems

Vicinal tricarbonyl compounds also serve as important building blocks in synthetic chemistry due to their highly electrophilic nature of the carbonyl groups. Depending on nitrogen nucleophiles, various heterocyclic compounds can be constructed.[11] Consequently, various biologically important molecules can be synthesized from vicinal tricarbonyls.[12] Thus, we attempted to expand our protocol to synthesize vicinal tricarbonyl compounds from 1,3-dicarbonyl derivatives. This result was summarized in Scheme 4.



Scheme 4: Reaction on 1,3-dicarbonyl systems

A gram scale synthesis of **6c** was successfully achieved under the optimized condition, resulting in about 71% yield of the vicinal tricarbonyl derivative **7c**. Similarly, 9-phenanthrol (**1t**) also reacted efficiently and afforded the corresponding product **3t** in 84% yield (Scheme 5).

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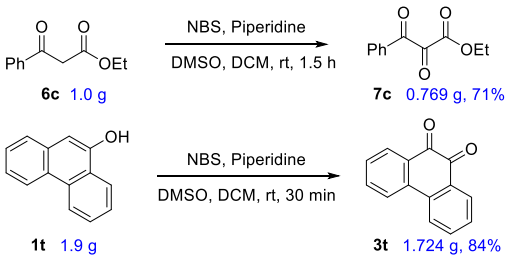
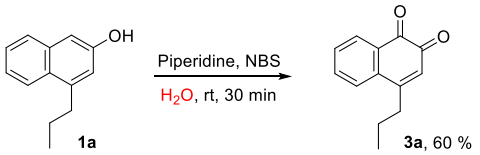


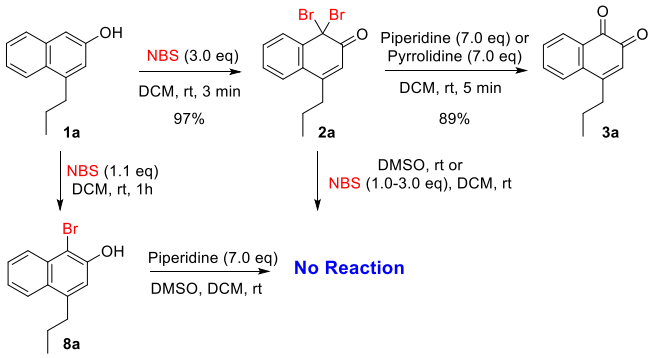
Scheme 5: Gram scale synthesis

Replacement of organic solvents with water is highly appreciated in concern of being environmentally benign. To this end, we also conducted the reaction in water as a solvent and the 1,2-NQ **3a** was obtained in good yield (60%) without any problems. When the reaction was performed under an inert atmosphere (N_2), it produced nearly the same yield (90%) with that of open air conditions. This indicates that this reaction does not depend on molecular oxygen.[6]



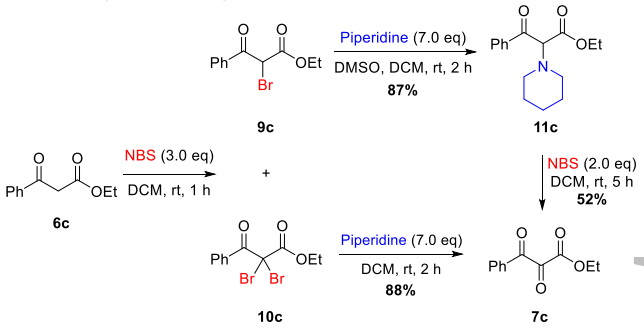
Scheme 6: Reaction in water and inert conditions

To figure out the reaction pathway, several control experiments were performed (Schemes 7 and 8). We only obtained the mono-bromo derivative **8a** when using 1.1 equiv. of NBS. A further reaction with piperidine failed to produce the naphthoquinone. This implies that this reaction cannot proceed via the Kornblum type oxidation. On the other hand, we obtained dibromo derivative **2a** with 97% yield when using 3.0 equiv. of NBS. Treatment of piperidine or pyrrolidine to the dibromo compound **2a** gave the final product **3a** in 89% yield, whereas reactions without a base (e.g. DMSO or NBS/DCM condition) failed to deliver the desired 1,2-NQ.



Scheme 7: Control experiment A

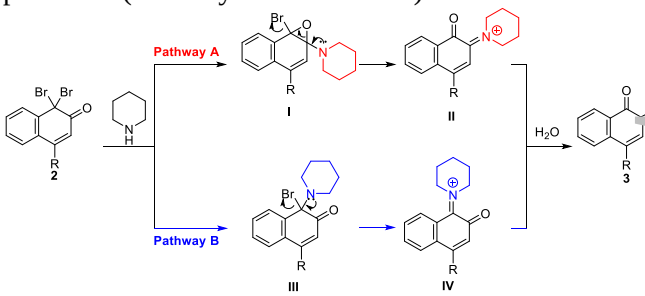
Similarly, in the case of tricarbonyl synthesis, both mono- and dibromo derivatives were obtained by utilizing 3.0 equiv. of NBS with **6c**. The dibromo intermediate **10c** was converted into the final product by utilizing 7.0 equiv. of piperidine. However, the mono-bromo derivative **9c**, only produced the piperidine substituted product **11c**, which was transformed into the final product **7c** with 2.0 equiv. of NBS (Scheme 8).



Scheme 8: Control experiment B

Based on the above results, a plausible mechanism was proposed for the NBS mediated oxidation reaction as depicted in Scheme 9. Piperidine could attack the carbonyl group of the dibromo intermediate **2** to afford a hemiaminal, which was spontaneously transformed into an oxirane intermediate **I**. Then, the rearrangement of **I**, followed by the subsequent hydrolysis of the resulting iminium intermediate **II**, can lead to the final product **3** (Pathway A of Scheme 9).

Alternatively, nucleophilic attack of piperidine on the dibromo carbon could lead to the formation of **III**, which may undergo subsequent elimination of the bromide, resulting in the intermediate **IV**. Subsequent hydrolysis of **IV** could generate the final product **3** (Pathway B of Scheme 9).



Scheme 9: Plausible reaction mechanism

Conclusion

We have developed an efficient synthetic method for 1,2-naphthoquinones from 2-naphthols by a facile one-pot procedure, utilizing NBS in the presence of piperidine under metal-free conditions. This one-pot method would allow the rapid construction of 1,2-NQs without the use of toxic metals or expensive complex reagents. Furthermore, it is also applicable to heterocyclic systems, and can be utilized for the synthesis of vicinal tricarbonyl systems.

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Conflicts of interest

There are no conflicts to declare.

Experimental Section

General procedure: To a solution of 2-naphthol derivatives **1** (0.21 mmol) in DMSO (1.2 mL) and dichlromethane (2.4 mL) was added drop wise piperidine (0.15 mL) and the reaction mixture was stirred at room temperature. Then, NBS (114 mg, 0.64 mmol) was added to the reaction mixture; stirring was continued until reaction was completed as monitored by TLC (5 min – 30 min). Then 10.0 mL of water was added, and extracted with dichloromethane, dried over $MgSO_4$. The solvent was removed under reduced pressure, and the residue was purified by column chromatography (silica gel, 20-50% ethyl acetate in hexanes) to afford the 1,2-naphthoquinone derivatives **3**.

Acknowledgements

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Rapid, operationally simple, and metal-free NBS mediated one-pot synthesis of 1,2-naphthoquinone from 2-naphthol

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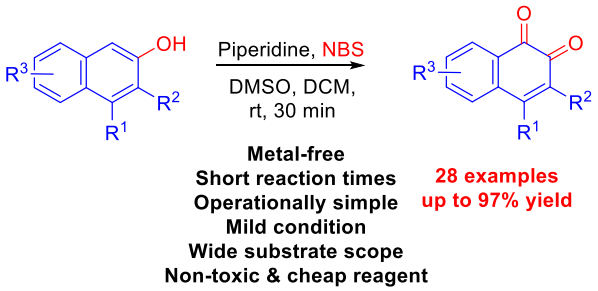

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