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ORIGINAL PAPER

3,4-Dihalo-2(5*H*)-furanones: a novel oxidant for the Glaser coupling reaction

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Abstract 5-Alkoxy-3,4-dihalo-2(5*H*)-furanones could be used as a kind of novel oxidant in the Glaser coupling reaction. The screening of reaction conditions showed that both $PdCl_2(PPh_3)_2$ and 3,4-dichloro-5-methoxy-2(5*H*)-furanone played crucial roles in the reaction. A possible reaction mechanism was proposed according to the reactivity of 3,4-dihalo-2(5*H*)-furanones. The new method easily allows the syntheses of alkyl and aryl substituted 1,3-diyne compounds. However, carbyne polymer was unexpectedly obtained when using trimethylsilyl acetylene as the substrate under the Glaser reaction condition.

Keywords 2(5*H*)-Furanone chemistry · Green chemistry · Oxidative coupling · Alkynes · Reaction mechanisms

Introduction

As a kind of structural unit, 2(5*H*)-furanone is frequently found in many natural products [1, 2], and many compounds containing 2(5*H*)-furanone structure have been considered as potential insecticides, bactericides, fungicides, antibiotics, anticancer agents, antiinflammatories, allergy inhibitors, antisoriasis agents, and cyclooxygenase inhibitors [2–6]. Therefore, in order to synthesize 2(5*H*)-furanone derivatives, there has been a continuous interest in developing efficient and convenient methods [6–9], including many synthetic methods using some simpler 2(5*H*)-furanones as organic intermediates [10]. For example, in order to use available 3,4-dihalo-2(5*H*)-furanones as intermediates, many kinds of reactions, such as the Sonogashira reaction [11], Knoevenagel reaction [12], Suzuki reaction [13], Friedel-Crafts reaction [14], and Michael addition-elimination reaction [15–17], have been reported recently.

On the basis of our previous studies on the reaction of 5-alkoxy-3,4-dihalo-2(5*H*)-furanones [10, 15–17], we hope to investigate their Sonogashira reaction with terminal alkynes under the protection of nitrogen atmosphere. Surprisingly, the formation of many 1,3-diyne compounds along with little expected Sonogashira compounds was observed. Although the Sonogashira reaction of other 3,4-dihalo-2(5*H*)-furanones with terminal alkynes has been reported before [11], the phenomenon of producing many Glaser coupling compounds was never mentioned to the best of our knowledge. Moreover, no reports have been made on the role of 3,4-dihalo-2(5*H*)-furanones used as the oxidant in the Glaser coupling reaction before.

As an important carbon-carbon bond formation method, the Glaser coupling reaction has been extensively used to synthesize various highly valuable functional molecules containing a buta-1,3-diyne fragment in natural product chemistry, material science, and supramolecular chemistry [18–23]. Considerable efforts have been directed to the development of the Glaser coupling reaction [24–27]. The oxidant plays a crucial role in the Glaser coupling reaction, and its choice has always been a focus of attention. Usually, air (or oxygen) has been used extensively as the oxidant because of its cheapness and availability [28–33].

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However, in order to develop novel catalytic systems or avoid other side reactions caused by oxygen [34, 35], more and more other oxidants, including chloroacetone [36], iodine [37], benzoquinone [34], trimethylamine oxide [38], sodium percarbonate [39], ethyl bromoacetate [35, 40], and nitrobenzene [41], have been explored and used in inert argon (or nitrogen) atmosphere to generate 1,3-diyne compounds.

Even so, these oxidants also have many disadvantages. For example, iodine may take part in the side reaction and lead to homopolymerization of the substrate diyne benzene [34]. More importantly, many of the above oxidants, such as chloroacetone, ethyl bromoacetate, trimethylamine oxide, and nitrobenzene, are irritating, corrosive, or highly toxic. To the best of our knowledge, now only sodium percarbonate is an environment-friendly oxidant with easy handling and storage [39]. Thus, it is still urgent to develop safe, effective, and convenient oxidants for the Glaser coupling reaction in inert atmosphere. Herein, we hope to report that 3,4-dihalo-2(5*H*)-furanones **2** can be novel effective and green oxidants for the Glaser coupling reaction (Scheme 1), especially 3,4-dichloro-5-methoxy-2(5*H*)-furanone (**2d**; the preparation method is shown in Scheme 2).

Results and discussion

Optimization of reaction conditions

The unexpected result of the Sonogashira reaction showed that the reaction conditions, including the reaction temperature and time, the base, the kinds and dosage of the main catalyst, affected the Glaser coupling reaction. Therefore, choosing phenylacetylene as the model substrate, we first investigated the optimization of reaction conditions (Table 1).

The influences of reaction temperature on the conversion of **1a** showed that the lower the temperature, the lower the conversion (Table 1, entries 1–4). When the reaction was carried out at 50 °C, diyne **3a** could be obtained in a conversion of 100% (entry 4). The influences of some common bases, such as K_2CO_3, Et_3N, and KF, were also examined (entries 4–6), and the results indicated that K_2CO_3 was the most effective base and the isolated yield of **3a** was 80% (entry 4).

The reaction time also affected the reaction significantly. Shortening the reaction time lowered the yield (Table 1, entries 4, 7–10), and the suitable time should be 72 h (entry 4). However, without the oxidative reagent **2c**, only a little product could be isolated (entry 11). This control experiment showed that **2c** was indispensable for the reaction system after the degassing step, and it was the oxidant indeed.

When the reaction was carried out for the same time (60 h), the influences of different palladium catalysts were evaluated for the new homocoupling reaction system (Table 1, entries 10, 12, 13). When $PdCl_2(PPh_3)_2$ was used as the catalyst, a satisfactory yield could also be obtained (entry 10). If the catalyst was replaced by $Pd(OAc)_2$ or $PdCl_2$, the coupling reaction afforded the product in moderate yields (entries 12 and 13). This observation suggested that the presence of PPh_3 as phosphine ligand could promote the reaction. The investigation of the influences of $PdCl_2(PPh_3)_2$ dosage (entries 9, 14–16) showed that the suitable palladium catalyst dosage was 1.0 mol% of the substrate phenylacetylene (entry 16).

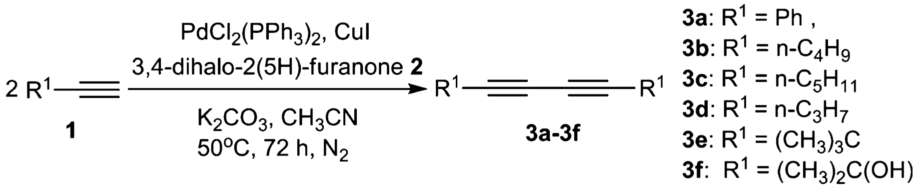
Thus, choosing phenylacetylene as the model substrate, 1.0 equiv. 3,4-dibromo-5-methoxy-2(5*H*)-furanone (**2c**) as the oxidant, 2.50 mol% CuI as cocatalyst, and acetonitrile as solvent, the optimized reaction conditions could be summarized in the following: 50 °C, 72 h, 2.0 equiv. K_2CO_3 as the base, and 1.0 mol% $PdCl_2(PPh_3)_2$ as the main catalyst.

Influences of different oxidants

Under the above-optimized conditions, the influences of different 3,4-dihalo-2(5*H*)-furanones as oxidant on the formation of 1,4-diphenylbuta-1,3-diyne (**3a**) in N_2 atmosphere were then investigated (Table 2). The results indicated that the oxidative reagents have an important influence on the reaction.

It could be seen that 3,4-dichloro-2(5*H*)-furanones (Table 2, entries 2, 4, 6) usually had better oxidation results than 3,4-dibromo-2(5*H*)-furanones (entries 1, 3, 5). This may be related to the less steric hindrance of chlorine as compared to bromine [15–17]. Meanwhile, for different 5-substituents of 3,4-dihalo-2(5*H*)-furanones, the best was 5-methoxy, and the worst was 5-hydroxy. Because of the isomerism, 3,4-dihalo-5-hydroxy-2(5*H*)-furanones **2a**, **2b** had a certain acidity (Scheme 3) [12, 42, 43], which

Scheme 1



Scheme 2

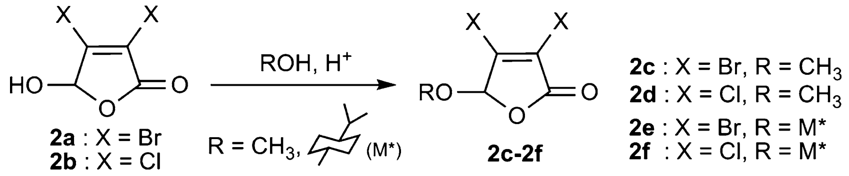
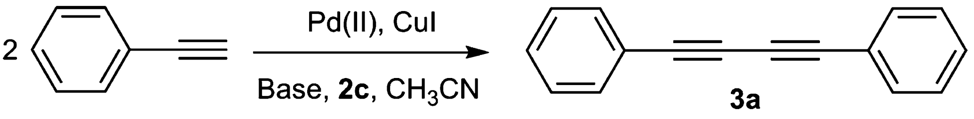


Table 1 Optimization of reaction conditions for the Glaser coupling reaction of **3a**



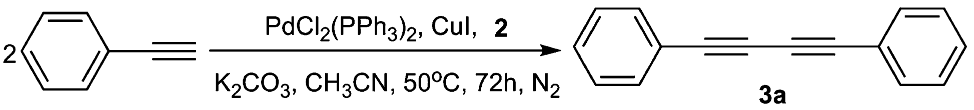
Entry[a]	Pd catalyst (mol%)	Base	*t* (h)	*T* (°C)	Yield (%)
1	$PdCl_2(PPh_3)_2$ (1.25)	K_2CO_3	72	r.t.	51[b]
2	$PdCl_2(PPh_3)_2$ (1.25)	K_2CO_3	72	30	73[b]
3	$PdCl_2(PPh_3)_2$ (1.25)	K_2CO_3	72	40	84[b]
4	$PdCl_2(PPh_3)_2$ (1.25)	K_2CO_3	72	50	80 (100[b])
5	$PdCl_2(PPh_3)_2$ (1.25)	Et_3N	72	50	62
6	$PdCl_2(PPh_3)_2$ (1.25)	KF	72	50	78
7	$PdCl_2(PPh_3)_2$ (1.25)	K_2CO_3	24	50	41
8	$PdCl_2(PPh_3)_2$ (1.25)	K_2CO_3	36	50	48
9	$PdCl_2(PPh_3)_2$ (1.25)	K_2CO_3	48	50	63
10	$PdCl_2(PPh_3)_2$ (1.25)	K_2CO_3	60	50	76
11	$PdCl_2(PPh_3)_2$ (1.25)	K_2CO_3	72	50	2[c]
12	$Pd(OAc)_2$ (1.25)	K_2CO_3	60	50	58
13	$PdCl_2$ (1.25)	K_2CO_3	60	50	63
14	$PdCl_2(PPh_3)_2$ (0.50)	K_2CO_3	48	50	46
15	$PdCl_2(PPh_3)_2$ (0.75)	K_2CO_3	48	50	62
16	$PdCl_2(PPh_3)_2$ (1.00)	K_2CO_3	48	50	63

[a] Reaction conditions: phenylacetylene (1 mmol), CuI (2.50 mol%), 5-methoxy-3,4-dibromo-2(5*H*)-furanone (**2c**, 1 equiv.), CH_3CN (5 cm^3), base (2 equiv.), and nitrogen atmosphere

[b] Conversion of **3a** was determined by GC analysis, and in other cases the yield was the isolated yield of **3a**

[c] As a control experiment, there is no **2c** in the reaction system after the degassing step

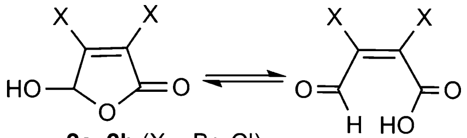
Table 2 Influences of different oxidants on the reaction



Entry[a]	Oxidant	X	5-Substituent	Yield (%)[b]
1	**2a**	Br	HO	59
2	**2b**	Cl	HO	70
3	**2c**	Br	CH_3O	80
4	**2d**	Cl	CH_3O	90
5	**2e**	Br	Menthoxy	66
6	**2f**	Cl	Menthoxy	77

[a] *Reaction conditions*. Phenylacetylene (1 mmol), $PdCl_2(PPh_3)_2$ (1.0 mol%), CuI (2.50 mol%), 3,4-dihalo-2(5*H*)-furanone **2** (1 equiv.), K_2CO_3 (2 equiv.), and 5 cm^3 CH_3CN

[b] Isolated yield



2a, **2b** (X = Br, Cl)

Scheme 3

lowered the alkalinity of the reaction systems. Therefore, for 3,4-dibromo-5-hydroxy-2(5*H*)-furanone (**2a**), the yield was the lowest of all because of the above two reasons (entry 2).

For 3,4-dihalo-5-menthoxy-2(5*H*)-furanones **2e**, **2f**, perhaps the larger steric hindrance of the 5-substituent was disadvantageous for the reaction. Therefore, because of the less steric hindrance of both chlorine and methoxy, 3,4-dichloro-5-methoxy-2(5*H*)-furanone (**2d**) was the most efficient oxidant for the homocoupling reaction, and the isolated yield of **3a** was 90% (Table 2, entry 4). Thus, using **2d** as the oxidant, the reaction of more terminal alkynes was investigated.

Application scope of the novel oxidant

Under the above-optimized conditions, the results of different substrates showed that the combination of $PdCl_2(PPh_3)_2$, CuI, K_2CO_3, and 3,4-dichloro-5-methoxy-2(5*H*)-furanone (**2d**) was an effective catalytic system for the Glaser coupling reaction. Not only the homocoupling of aromatic alkyne gave diyne **3a** in good yield (90%), but also the aliphatic alkynes gave the products in yields of 76–88% (Table 3).

Usually, terminal aliphatic alkynes are sluggish in undergoing Glaser dimerization because of the weaker acidity of the acetylenic proton [44, 45]. However, in our reaction system, the homocoupling was smoothly carried out to afford the corresponding diynes **3b**–**3e** in good to excellent yields (mostly over 80%). At the same time, some functional groups on the alkynes, e.g., hydroxy, also had no effects on the reaction under the performed conditions, and the yield of **3f** was 86% (Table 3, Entry 6).

To our surprise, when we treated trimethylsilylacetylene (**1g**) under the same optimized conditions, no Glaser coupling diyne **3g** was obtained (Scheme 4), but a dark powder was isolated. Moreover, the dark powder was amorphous and insoluble in any known solvent. According to the literature, these properties and its FT-IR characterization (Fig. 1, the IR test is one of the main characterization methods for the carbyne polymer because of its insolubility in most solvents) [46–53] indicated that the product should be carbyne (Scheme 4), especially *β*-carbyne, as the absorptions at 1,611 and 1,395 cm^{-1} were attributed to cumulative double bonds [46, 49–53].

There are two possible reasons for the unexpected formation of carbyne under the Glaser coupling reaction condition. Firstly, the trimethylsilyl group is often employed as a protective group for terminal alkynes and could easily be removed to give $-C\equiv CH$ under the basic condition [54–56], so trimethylsilylacetylene (**1g**) or 1,4-bis(trimethylsilyl)-buta-1,3-diyne (**3g**) was unstable. Of course, the coupling reaction of alkynylsilanes (**1g** or **3g**) also may be mediated by Cu(I) under oxidative conditions [57]. Secondly, the *β*-carbyne could be formed via the Cu(I)-catalyzed coupling of the $-C\equiv CH$ group [48]. The two reasons affected each other, which led to the formation of carbyne under the Glaser coupling reaction condition.

Carbyne can be utilized in many fields, such as biomedicine (used as surgical sutures and organ replacement) [58], and carbon material science [59–61] (for example, used as the material of the conversion into diamond [62] or fullerene [63]). Therefore, it is notable that the formation of *β*-carbyne under the Glaser coupling reaction condition may be a novel mild and convenient method for the synthesis of carbyne compared with the previous reported routes [46–53] once the reaction conditions are further optimized.

Plausible mechanism of 3,4-dihalo-2(5H)-furanones as oxidant

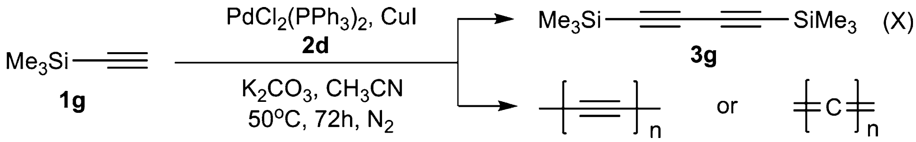
Compared with other oxidants used in the Glaser coupling reaction, such as trimethylamine oxide (Me_3NO) [38],

Table 3 The yields and physical constants of the compounds **3a**–**3f**

Entry[a]	Compound	Appearance	Yield[b]/%	M.p./°C (lit. data)
1	**3a**	White solid	90	85.0–86.4 (86–88 [65])
2	**3b**	Colorless oil	80	— ([65])
3	**3c**	Colorless oil	76	— ([65])
4	**3d**	Colorless oil	85	— ([65])
5	**3e**	White solid	88	127.4–129.3 (128–130 [38])
6	**3f**	White needle	86	128.0–130.0 (131–133 [66])

[a] *Reaction conditions*. Alkynes **3** (1 mmol), $PdCl_2(PPh_3)_2$ (1.00 mol%), CuI (2.50 mol%), 5-methoxy-3,4-dichloro-2(5*H*)-furanone **2d** (1 equiv.), K_2CO_3 (2 equiv.), and CH_3CN (5 cm^3); [b] Isolated yield

Scheme 4



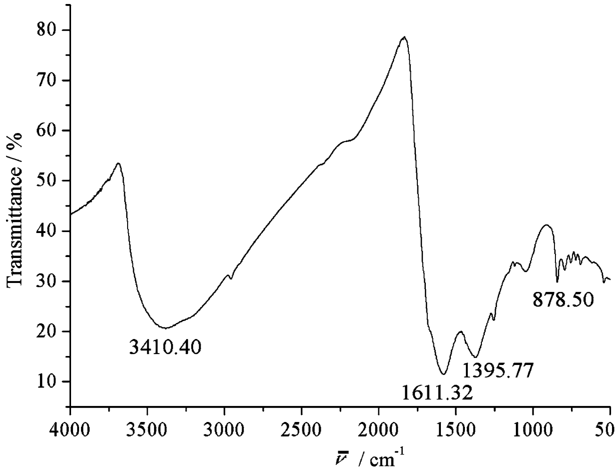


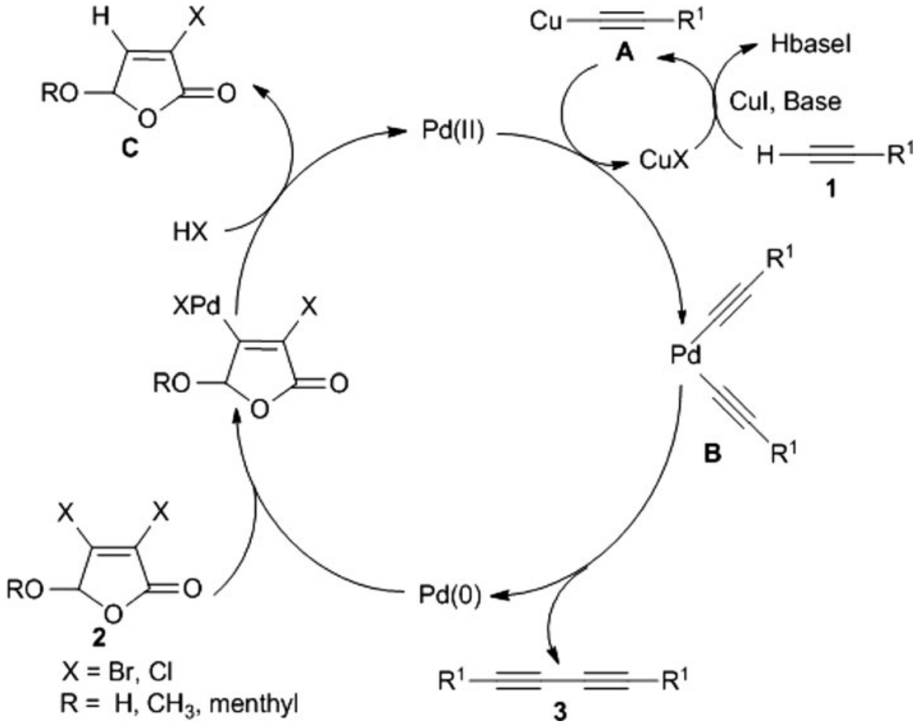
Fig. 1 FT-IR spectrum of the product of trimethylsilylacetylene (**1g**)

ethyl bromoacetate [35, 40], and chloroacetone [36], 3,4-dihalo-2(5*H*)-furanones are a kind of easily available oxidant (Scheme 2) [15–17] with higher thermal stability and better oxidation resistance, especially better environmental friendliness. In order to further expand their applications in organic synthesis, we proposed a possible reaction mechanism of 3,4-dihalo-2(5*H*)-furanones as an oxidant based on the previous reports on the mechanism of the Glaser coupling reaction [29, 35, 38, 64] and the reactivity of 3,4-dihalo-2(5*H*)-furanones [3, 4, 11].

As outlined in Scheme 5, firstly, with the aid of a base the reaction of terminal alkynes **1** with CuI afforded the intermediate **A** readily. Then, the replacement of Pd(II) with intermediate **A** would occur to form a dialkynylpalladium(II) intermediate **B** and regenerate the active Cu(I) species. Reductive elimination of the dialkynylpalladium(II) intermediate **B** could form the desired diyne **3** and Pd(0). Finally, Pd(0) was oxidized by 5-alkoxy-3,4-dihalo-2(5*H*)-furanones **2** to regenerate the active Pd(II) species leading to a new catalytic cycle.

In summary, using 5-alkoxy-3,4-dihalo-2(5*H*)-furanones as the oxidant, we have successfully developed a novel pathway for the palladium-catalyzed homocoupling reaction. This oxidant with higher thermal stability is more environmentally friendly and convenient than most previously reported oxidants. These unexpected results and systematic investigations are beneficial to further enrich the chemistry of 2(5*H*)-furanones.

Scheme 5



Experimental

All reagents and solvents were obtained commercially and used without further purification. All melting points were determined on an X-5 digital melting points apparatus. ^{1}H and ^{13}C NMR spectra were obtained in $CDCl_3$ or DMSO-d_6 on a Varian DRX-400 MHz spectrometer using tetramethylsilane (TMS) as an internal standard. The mass spectra were recorded on a Finnigan Trace DSQ at an ionization voltage of 70 eV. Using **2a**, **2b**, natural menthol, and methanol as starting materials, **2c**–**2f** could be easily prepared according to the literature [15–17] (but the resolution step for **2e** and **2f** was omitted in this case, Scheme 2).

General procedure for the Glaser coupling reaction

After degassing, a mixture of alkyne (1 mmol), $PdCl_2(PPh_3)_2$ (1.0 mol%), CuI (2.50 mol%), K_2CO_3 (2 equiv.), 3,4-dichloro-5-methoxy-2(5*H*)-furanone (**2d**, 1 equiv.), and 5 cm^3 MeCN was stirred under N_2 at 50 °C for 72 h. Once the reaction was complete, the reaction mixture was diluted with 5 cm^3 water and then extracted with ethyl acetate (10 cm^3 × 2). The combined organic layers were dried with magnesium sulfate and concentrated under a vacuum to give a crude product, which was purified by column chromatography on silica gel with gradient eluent of mixtures of *n*-hexane and dichloromethane to afford compounds **3a**–**3f** (Scheme 1) for analysis. IR and ^{1}H NMR spectra were found to be identical with the data described in the literature [38, 65, 66], and some products selected at random were further confirmed by MS.

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