C—H Activation, a New Strategy for Synthesis of 3-Substituted Phthalides

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Abstract Phthalides are an important class of compounds owing to their role as the key structural motifs in bioactive natural products and pharmaceutical synthesis. C—H activation has become a promising strategy for preparation of phthalides due to its advantages of high efficiency and atomic economy compared with the traditional methods. In this paper, we summarized recent advances on synthesis of 3-substituted phthalides via C—H activation according to different transition metal catalytic systems.

Keywords synthesis, 3-substituted phthalides, C—H activation, catalysis

Introduction

Phthalides, known as α-hydroxymethyl benzozolactone, is composed of a γ-lactone ring and a benzene ring. 3-Substituted phthalides have been widely present in natural products and clinic drugs,[1-4] which are important pharmacoactive groups and intermediates in pharmaceutical synthesis. Additionally, they have important and significant biological activity (Figure 1). The traditional method (Scheme 1) for synthesizing 3-substituted phthalides[5] usually have some drawbacks of many steps and low atom utilization. As a potent tool developed in recent years, C—H activation is presently widely used in the preparation of many drugs possessing several advantages of simplicity, high efficiency and environmentally friendly. Herein, we reviewed nearly 30 methods for synthesis of 3-substituted phthalides via C—H activation in recent years to provide an overview in this research field.

Scheme 1 The traditional methods for synthesizing 3-substituted phthalides

![Scheme 1](https://example.com/scheme1.png)

Synthesis of 3-Substituted Phthalides Using Palladium Catalysts

As early as 1998, Miura et al.[6] firstly reported that Pd(OAc)2 catalyzed the reaction of benzoic acid with olefins (Scheme 2). The reaction with 10 mol% of Pd(OAc)2 as a catalyst, 1 equivalent of Cu(OAc)2·H2O as an oxidant, when benzoic acid has a substituent at the ortho position, a five-membered heterocycle is formed through the migration and insertion of olefin to obtain 3-substituted phthalides due to the steric effects. Otherwise, it is easy to form a six-member heterocyclic. Notably, they disclosed a case of α-n-butyrate acylate as the reactant, and the alkylation product 3-butyphthalate acetate is obtained by Michael addition reaction.

The C—C and C—O coupling reactions of benzoic acid were reported with a Pd(OAc)2 catalyst by Yu et al.[7] (Scheme 3). Dibromomethane with benzoic acid generated five-membered ring lactone. The addition of inorganic base plays a key role in promoting reaction such as K2HPO4, KHCO3 and Na2CO3. It has good adaptability to both electron-rich and electron-deficient substrates.

In the following research, Martin et al.[8] reported an improved reaction to synthesize 3-substituted phthalides. Pd(OAc)2 was used as a catalyst, Ag2CO3 (3 equiv.) was added as a base, and N-acetyltyrosine also played an important role in promoting the reaction as ligands (Scheme 4). Compared with previous works, the system achieved higher yields and substrate compatibility.

Figure 1 Representative examples of 3-substituted phthalides and biologically.
Review

Scheme 2  Pd-catalyzed alkenylation and alkylation of benzoic acid with olefin and proposed mechanism

Using the Pd(OAc)₂ and Ag₂O, Lee et al.⁹ established the coupling C—C and C—O reaction of ortho-benzoic acid to generate heterocyclic ring (Scheme 5). When the ortho-position of benzoic acid is hydrogen, the reaction yielded a six-member heterocyclic isoumar. When the ortho-position of benzoic acid is CH₃ or OCH₃, the product is a five-member heterocyclic 3-substituted phthalide. According to the proposed mechanism, the coupling of styrene and benzoic acid undergoes a similar Heck coupling. When the ortho position of benzoic acid has steric hindrance, the migration and insertion of Pd tend to form a five-membered ring. Otherwise, it tends to form a six-membered ring. However, the system is only effective for electron-rich benzoic acid and styrene.

Scheme 5  Pd catalyzed coupling of benzoic acid with styrene and proposed mechanism

Lately, Zeng et al.¹⁰ developed a method for synthesis of phthalides using benzoic acid and bromoalkynes as substrates through carboxylate-directed ortho-C—H activation. The reaction process was promoted by ligands with Pd(OAc)₂ as catalyst (Scheme 6). The reaction has a wide range of substrates and a variety of phthalides have been synthesized by alkynyl-annulation.

Scheme 6  Cyclization of benzoic acid and bromoalkynes by ortho-C—H activation

Almost in the same time, Gogoi et al.¹¹ reported a novel decarboxylative alkyne insertion reaction to synthesize six-membered compounds with Pd(II) as the catalyst (Scheme 7). According to the proposed mechanism, the cyclization reaction of hydroxynaphthoquinones and disubstituted alkynes was carried out through C—H/C—C activation, acetylene insertion,
internal molecular cyclization and decarbonylation to obtain high yield 3-alkyl-phthalides.

Synthesis of 3-Substituted Phthalides Using Rhodium Catalysts

Rhodium catalysts are another type of transition metal catalysts in C—H activation reactions, which is widely used in synthesis of 3-substituted phthalides.

In the presence of two silver additives (AgOTf and Ag$_2$CO$_3$), [Cp*RhCl$_2$]$_2$ can catalyze the reaction of benzoic acid ortho-C—H activation with aldehydes (Scheme 8).[12] Both additives are necessary and neither of them is dispensable. The use of [Cp*RhCl$_2$]$_2$ alone as a catalyst doesn’t generate any desired product, which suggests that AgOTf is functioning as a chloride trapping agent to remove the chloride ligands from the [Cp*RhCl$_2$]$_2$ complex and to generate the active species Cp*Rh(III)(OTf)$_2$. With the help of Ag$_2$CO$_3$, benzoic acid is deprotonated, which coordinates to Rh(III) through one of the carboxylic oxygens. Since the Grignard type reaction occurs between a stable carbon anion and an electrophile, the highest yield can be obtained by coupling electron-rich acids with electron-deficient aldehydes.

The next reaction is similar to the above report and is carried out under similar conditions with [Cp*RhCl$_2$] catalyst and stoichiometric Ag$_2$CO$_3$ (Scheme 9).[13] The difference in this reaction is that it’s a dehydrogenation process, so Ag$_2$CO$_3$ acts as an oxidant here.

A strategy similar to the above approach involves replacing benzoic acid with N-substituted benzimidates (Scheme 10).[14] The imidate group acts in the same way as the carboxyl group to promote ortho-C—H activation, firstly by the insertion of aldehydes and then by the nucleophilic acyl substitution of the resulting alkoxide to the imidate. The final hydrolysis of the imidate affords phthalides.
Another method for synthesis of 3-substituted phthalides is chemoselective coupling of benzoic acid and alkenes, which uses \([\text{Cp}^*\text{RhCl}_2]\) as the catalyst and NaClO as the oxidant in \(\text{H}_2\text{O/ACOH}\) (Scheme 11). Because the yield is halved when acetic acid is substituted for pivalic acid or trifluoroacetic acid, the nature of the additive acid is crucial to the outcome of the reaction.

**Scheme 11** Rh-catalyzed annulative coupling of benzoic acids and electrophilic alkenes for synthesis of 3-substituted phthalides in the presence of \(\text{Cl(III)}\) and proposed mechanism

An additional method for synthesis of 3-substituted phthalides uses benzoic acids and alkenes as substrates, \([\text{COD}]{\text{RhCl}_2}\) as a catalyst, \(\text{Cu(OAc)}_2\cdot\text{H}_2\text{O}\) as an oxidant, and dicyclopentadiene (DCPD) as an additive (Scheme 12).

**Scheme 12** Rh-catalyzed annulative coupling of benzoic acids and electrophilic alkenes for synthesis of 3-substituted phthalides in the presence of \(\text{Cu(II)}\)

Similar to the above reactions, Jiang et al. reported that Rh(III) can catalyze the coupling reaction of benzoic acid and olefins in the oxygen environment (Scheme 13). The difference is that this reaction produced a mixture of mono- and di-substituted products. The primary product depends on the nature and location of substituents on the benzene ring: \(m\)-substituted benzoic acids mainly afford 3-substituted phthalides (9a); unsubstituted and \(p\)-substituted benzoic acids preferentially give 3,7-disubstituted phthalides (6b and 7b).

When \([\text{Cp}^*\text{RhCl}_2]\) is used as a catalyst in the presence of \(\text{Cu(OAc)}_2\cdot\text{H}_2\text{O}\) and \(\alpha\)-xylene as solvent, the reaction of alkyl acrylates and benzoic acid does not provide monosubstituted products. Instead, two disubstituted products were obtained: 3-alkyl-7-vinylphthalide (10a or 11a) and 3-alkylidene-7-vinylphthalide (10b or 11b) (Scheme 14). The result is different from the reaction seen before.

-Benzamide may be used instead of benzoic acid as the substrate for synthesis of phthalides but only a tertiary benzamide can be used. The best results are obtained from benzimidazole benzoamide derivatives as reported (Scheme 15).

**Scheme 13** Rh-catalyzed annulative coupling of benzoic acids and electrophilic alkenes for synthesis of 3-substituted phthalides in the presence of \(\text{O}_2\)

**Scheme 14** Rh-catalyzed annulative coupling of benzoic acid and electrophilic alkenes for synthesis of 3-alkyl- and 3-ylidene phthalides

Acid additive (AcOH) and low-polarity solvent (DCE) must be used to achieve maximum yield and better chemical selectivity. The annulative coupling likely consists of four tandem reactions: (1) an olefination, in which benzimidazole acts as a directing group; (2) an acetylation, in which benzimidazole acts as a leaving group and is replaced by acetate; (3) acetate hydrolysis with liberation of the COOH group; and (4) cyclization.

The method of enantioselective synthesis of 3-substituted phthalides is ketone hydroacylation with the ee values up to 97%, and the key to the reaction is the cationic diphosphine Rh(I) complex. When toluene was heated in the presence of
Scheme 15  Rh-catalyzed annulative coupling of N-benzoyl benzimidazole and electrophilic alkenes for synthesis of 3-substituted phthalides and proposed mechanism

Scheme 16  Rh-catalyzed hydroacylation of α-ketobenzaldehydes for the synthesis of 3-substituted phthalides

Scheme 17  Ru-catalyzed coupling of benzoic acids and electrophilic alkenes in water for synthesis of 3-substituted phthalides

Scheme 18  Ru-catalyzed annulative coupling of benzoic acids and electrophilic alkenes in PEG-400/water for synthesis of 3-substituted phthalides and proposed mechanism

Scheme 19  Ru-catalyzed annulative coupling of benzoic acids and electrophilic alkenes for the synthesis of 3-substituted phthalides

Scheme 20  Ru-catalyzed annulative coupling of benzoic acids and electrophilic alkenes for synthesis of 3-substituted phthalides in GVL in the presence of O\textsubscript{2}

In a similar manner, Baidya and co-workers developed\textsuperscript{[23]} Ru(II)-catalyzed annulative coupling from benzoic acids and aryl vinyl sulfones with acetonitrile as the solvent (Scheme 19).

Scheme 21  RuHCl(CO)(PPh\textsubscript{3})\textsubscript{3} as a catalyst, phthalaldehyde could successfully convert into phthalide after heating in toluene (Scheme 17).\textsuperscript{[21]} The reaction involves an intramolecular Tishchenko-type lactonization, which consists of three steps: (1) hydroruthenation of the keto carbonyl group with formation of an alkoxyruthenium complex, (2) intramolecular alkoxy-ruthenation of aldehyde carbonyl to provide acetel-type complex, and (3) β-hydride elimination to liberate phthalide.

In 2015, Cai \textit{et al.}\textsuperscript{[22]} reported the similar reaction conditions as above. If water was replaced with a mixture of polyethylene glycol 400 (PEG-400) and water (3:2, v/v), the catalyst in the coupling of benzoic acid and electrophilic alkenes could be recycled for 6 times without losing the catalytic activity (Scheme 18). This may be due to the coordination between polymer oxygen and metal, and PEG-400/H\textsubscript{2}O mixture has strong adsorption capacity for both metal complexes.
Synthesis of 3-Substituted Phthalides from Other Transition Metal Catalysts

Yang et al.\textsuperscript{[30]} developed a method to prepare 3-substituted phthalides with high enantiomeric selectivity. In the presence of indium metal, CoBr\textsubscript{2} was reduced in situ to produce chiral Co(0) diphosphine complex (Scheme 22), and acetonitrile was used as solvent and the reaction temperature needs 80 °C.

In 2019, Wang et al.\textsuperscript{[31]} reported [4+1] cyclization catalyzed by metal rhenium (Re) with benzamides and aldehydes as substrates, which provides a convenient method for synthesis of phthalide derivatives (Scheme 23). It shows a unique reaction pattern compared to the above catalysis using Rh and Pd in terms of the simplicity of the initial substrates, various electrical substrate types, and elimination of by-products. Notably, after this reaction has undergone coordination, C—H bond activation, and insertion processes, quenching of A with acid gives alcohol B, which undergoes intramolecular N- or O-attacked cyclization, affording lactam C or iminoether D, respectively. The O-attacked cyclization is preferred in the reactions of electron-deficient and electron-neutral aldehydes to afford iminoether D. Then, the acid-promoted hydrolysis of D gives rise to the formation of the final product E. When electron-rich aldehydes are used, the N-attacked cyclization might occur to afford lactam C from alcohol B.

Lately, Kuninobu et al.\textsuperscript{[32]} reported synthesis of phthalides from benzoate and propylene oxide via C—H bond activation mediated by manganese and boron (Scheme 24). In their research, triphenylboron is essential to facilitate the reaction. This is an example of an oxygen guiding group assisting manganese to catalyze the C—H activation.

Electrochemical C—H activation with iridium catalysis was also studied by Ackermann and co-workers,\textsuperscript{[33]} which was synergistic by redox catalysts (Scheme 25). Iridium catalytic electrocatalysis has a wide range of substrate applications and good tolerance to various functional groups, especially to the chemical sensitive bromine and iodide aromatics.

Conclusions and Perspectives

So far, nearly 30 protocols through C—H activation have been developed for direct synthesis of 3-substituted phthalides. Nowadays, it becomes more and more potent tool for synthesis
of these phthalide derivatives. However, there are still two challenges in the following: (1) There is no direct or semi-direct method to construct the 3-substituted phthalide with a saturated alky chain at the 3-position; (2) The large number of factors (substrate, catalyst, oxidant, additives, solvent, as well as reagents ratio and concentration) that affect chemico- and regio-selectivity and are difficult to control. Thus, these aspects mentioned above still should be further improved to establish more effective strategies to synthesis of 3-substituted phthalides via C–H activation, and the steric configuration of the 3-position substitution of phthalides is also effectively controlled through the selective use of chiral catalysts. In the meantime, besides transition metals used to catalyze the reaction, photochemical and electrochemical catalyzed reactions via the activation of C–H bond to construct the 3-substituted phthalide are also vigorously developed now.

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