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ARTICLE TYPE

Synthesis of Benzil Derivatives via Oxidation of Alkynes Catalyzed by Pd–Fe₃O₄ Heterodimer Nanocrystals

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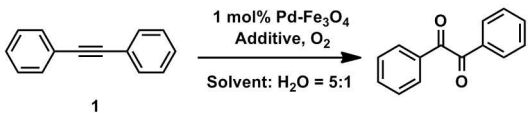
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An efficient, iterative, catalytic, Wacker-type oxidation of alkynes to 1,2-diketones using a Pd–Fe₃O₄ heterodimer nanocrystalline catalyst has been developed. This process has a wide substrate scope and affords 1,2-diketo compounds in excellent yields under atmospheric conditions. The operational procedure using the Pd–Fe₃O₄ nanocatalyst is extremely easy, and the catalyst can be recovered by employing simple magnetic separation, enabling the recycling of the catalyst for five times without loss of catalytic activity.

1,2-Dicarbonyl derivatives are valuable structural motifs often embedded in many natural products¹ and biologically active compounds.² Among these derivatives, benzil derivatives are employed for a number of interesting applications, such as corrosion inhibitors of mild steel,³ photosensitive agents in photocurable coatings,⁴ and carboxylesterase (CE) inhibitors.⁵ Moreover, 1,2-dicarbonyl derivatives can be used as precursors to many biologically active compounds or as building blocks for the synthesis of quinoxalines, triazines, and imidazoles.⁶

In light of their usefulness, the development of convenient synthetic pathways for 1,2-diketones has attracted much attention, and several synthetic methods have been reported, including substitutions of keto acid chloride or oxalyl chloride⁷ and oxidations of hydroxyketone derivatives.⁸ Among several other methods,⁹ oxidation of 1,2-diarylalkynes¹⁰ can be a very straightforward approach to the synthesis of diketones. The 1,2-diarylalkynes are easily prepared by standard Sonogashira coupling reactions of aryl alkynes and aryl halides.¹¹ A variety of reagents have been employed to oxidize 1,2-diarylalkynes for the synthesis of benzil compounds, such as manganese¹² and chromium reagents,¹³ sulfur trioxide-dioxane complexes,¹⁴ ozone,¹⁵ dioxiranes,¹⁶ iodo- or bromo-succinimide,¹⁷ and orthoperiodic acid.¹⁸ However, these reagents are highly toxic, and the procedures associated with their use are expensive, low yielding, limited in terms of functional group tolerance, poorly chemoselective, and produce environmentally hazardous wastes.

Recently, in response to these drawbacks, new alkyne oxidation reactions have been developed that utilize homogenous catalysts such as iron(III) bromide,¹⁹ palladium(II) sources with copper co-catalyst,²⁰ and gold-catalyzed transformations.²¹ The use of a heterogeneous palladium source such as palladium on carbon (Pd/C) has been reported for the synthesis of benzil derivatives

Table 1 Optimization of Wacker-type oxidation^a


Entry	Solvent	Additive	Temp. (°C)	Time (h)	Yield (%) ^b
1	DMSO	CuBr ₂	95	28	62
2	DMF	CuBr ₂	95	28	3
3	H ₂ O	CuBr ₂	95	28	6
4	Toluene	CuBr ₂	95	28	-
5	1,4-Dioxane	CuBr ₂	95	28	98
6	1,4-Dioxane	CuBr ₂	75	28	85
7	1,4-Dioxane	-	95	28	-
8	1,4-Dioxane	CuCl ₂	95	28	87
9	1,4-Dioxane	Cu(OAc) ₂	95	28	8
10	1,4-Dioxane	CuI	95	28	-

^a Reaction conditions: Compound 1 (0.5 mmol), Pd–Fe₃O₄ (1.0 mol%), additive (0.05 mmol), solvent (5.0 mL), H₂O (1.0 mL), O₂ balloon. ^b

⁵⁰ Yield of isolated product.

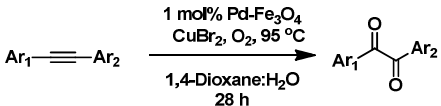
with DMSO and molecular oxygen as dual oxidants.²² There has been increased interest in the use of magnetically recyclable palladium nanoparticles and nanomaterials²³ for the development of sustainable, efficient, heterogeneous catalysts for practical organic synthesis. Iron oxide-based catalysts have many advantages including their facile recovery by an external neodymium magnet, thus obviating complicated separation/filtration processes. Thus, magnetically recoverable iron oxide catalysts offer great potential for industrial applications.

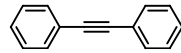
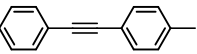
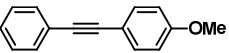
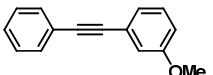
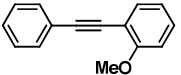
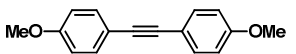
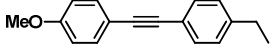
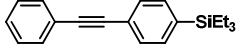
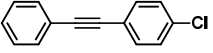
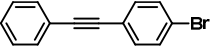
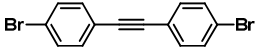
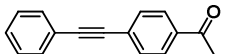
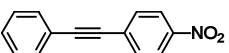
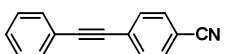
In our laboratory, we have been interested in utilizing efficient, heterobimetallic catalysts based on the magnetically recyclable iron oxide nanoparticles for many synthetic organic applications. We have thus reported the efficient synthesis of Pd- and Rh-Fe₃O₄ bimetallic nanocrystals and their applications to Suzuki couplings,²⁴ nitroarene reductions,²⁵ Heck and Sonogashira cross-

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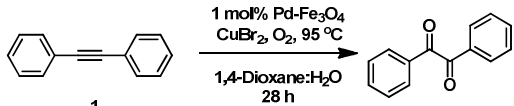
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Table 2 Substrate scope of the Wacker-type oxidation using Pd-Fe₃O₄^a


Entry	Substrate	Yield (%) ^b
1		98
2		92
3		90
4		88
5		86
6		86
7		92
8		85
9		82
10		94
11		86
12		91
13		83
14		77

^a Reaction conditions: Substrate (0.5 mmol), Pd-Fe₃O₄ (1.0 mol%), CuBr₂ (0.05 mmol), 1,4-dioxane (5.0 mL), H₂O (1.0 mL), O₂ balloon, 95°C. ^b Yield of isolated product.

Table 3 Recyclability of the Pd-Fe₃O₄ catalyst for the Wacker-type oxidation^a


Entry	Yield (%) ^b
1	98
2	96
3	96
4	92
5	83

^a Reaction conditions: **1** (0.5 mmol), Pd-Fe₃O₄ (1.0 mol%), CuBr₂ (0.05 mmol), 1,4-dioxane (5.0 mL), H₂O (1.0 mL), O₂ balloon, 95 °C. ^b Yield of isolated product.

¹⁵ couplings,²⁶ direct catalytic C–H arylations,²⁷ Wacker oxidations of terminal olefins,²⁸ and Suzuki polycondensations for the synthesis of conjugated polymers.²⁹ We have also reported the synthesis and applications of nanoflake-shaped iron oxide/Pt

toward highly efficient hydrogenation reactions.³⁰ Herein, we report the efficient synthesis of benzil derivatives from 1,2-diarylacetynes under oxygen atmosphere using Pd-Fe₃O₄ heterodimer nanocrystals as a reusable and durable catalyst system.

²⁰ Our first attempt at the Pd-Fe₃O₄-catalyzed oxidation of an acetylene derivative was carried out using diphenylacetylene (**1a**) and 1 mol% catalyst in DMSO under 1 atm oxygen in the presence of 10 mol% CuBr₂. This reaction afforded the corresponding benzil (**2a**) in 62% yield after 28 h at 95 °C (Table ²⁵ 1, entry 1). We then carried out an extensive screening of solvents, such as DMSO, DMF, H₂O, toluene and 1,4-dioxane with 10 mol% CuBr₂ under otherwise identical reaction conditions (Table 1, entries 1-5). Among the solvents examined, 1,4-dioxane was identified as most effective for the Wacker-type ³⁰ oxidation. The addition of a Cu(II) salt was absolutely necessary since no product formation was observed in its absence (Table 1, entry 7). Replacing CuBr₂ by CuCl₂, Cu(OAc)₂, or CuI resulted in decreased reactivity in each case (Table 1, entries 8-10). In fact, no reaction was observed with CuI. Finally, optimal conditions ³⁵ were identified by employing 1 mol% of Pd-Fe₃O₄ and 10 mol% of CuBr₂ in dioxane/H₂O under 1 atm of O₂ in a balloon, which yielded 98% of the desired product (Table 1, entry 5). When the reaction temperature decrease to 75 °C, the yield was decreased slightly under otherwise the same conditions (Table 1, entry 6).

⁴⁰ With the optimized reaction conditions, the substrate scope was then examined. As shown in Table 2, good to excellent product yields were obtained for a variety of diaryl-substituted alkyne derivatives. Reactions of substrates equipped with electron-donating substituents, such as *p*-methyl, *o*-, *m*- and *p*-methoxy groups, provided excellent yields of the desired products (Table 2, entries 2-5). Sterically hindered, *o*-substituted substrate

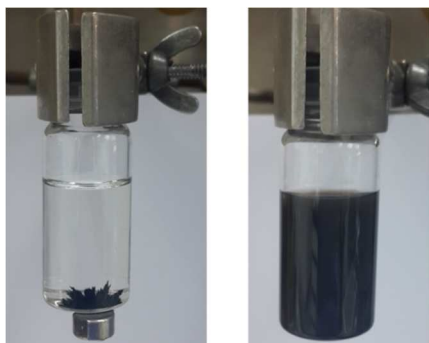


Fig. 1 Magnetic separation of Pd-Fe₃O₄ after the reaction.

also gave good yield of the desired product (entry 5). Reactions of diphenylacetylenes with two *p*-substitutions, such as 1,2-bis(4-methoxyphenyl)ethyne and 1-ethyl-4-((4-methoxyphenyl)ethynyl)benzene gave good yield to the corresponding diketo products (Table 2, entries 6-7). A trimethylsilyl-substitution was also well tolerated under the reaction conditions (Table 2, entry 8). In addition, reactions of substrates having one or two halide substitutions at the *para* position(s) also gave rise to good to excellent yields (Table 2, entries 9-11). Reactions of substrates having other electron-withdrawing groups, such as *p*-acetyl and *p*-nitro groups proceeded with good yields (Table 2, entries 12-13). The only reaction that provided a moderate yield (77%) was that employing *p*-cyano-substituted diphenylacetylene (Table 2, entry 14).

The recyclability of the Pd-Fe₃O₄ nanocatalyst was confirmed by its ability to repeatedly catalyze the oxidation of diphenylacetylene. After the reaction, the catalyst from the reaction mixture was simply collected using an external neodymium magnet. Then, without filtration, the catalyst was washed five times with EtOAc, twice with water, and was dried *in vacuo* for 2 h. The recovered catalyst could then be reused immediately in the next reaction. In this experiment, more than 99% of the nanocrystals could be recovered through the use of an external magnet and through catalyst purification by dispersion and collection cycles. The results are presented in Table 3. The reactivity of the catalyst consistently remained unchanged up to the fourth recycling experiment. After the fifth run, however, the yield of the product was slightly diminished to 83%, indicating a slight decrease in the activity of the Pd-Fe₃O₄ catalyst.

As shown in Fig. 1, the Pd-Fe₃O₄ nanocrystals exhibited very good dispersion during and after the oxidation reaction. After the reaction was complete, the nanocrystals could be gathered easily using an external magnet. The transmission electron microscope (TEM) image of the Pd-Fe₃O₄ nanocrystals after five oxidation cycles showed that the size and morphology of the nanocrystals had not changed (Fig. S2 and S3). Moreover, the powder X-ray diffraction (XRD) pattern of the catalyst did not show any noticeable change from those of the catalyst before use (Fig. S4 and S5). In addition, when the catalyst system was analyzed after the first and fifth reaction by inductively coupled plasma atomic emission spectroscopy (ICP-AES), the nanoparticles showed 1.9 and 1.2 wt% of Pd, respectively. About 37% of palladium was lost after 5 cycles. This observation indicates that a small amount

of Pd might have leached out from the original catalyst system upon each recycling experiment. However, there was no detectable change in Fe content.

Conclusions

In summary, a convenient, heterogeneous, Wacker-type oxidation of alkynes to benzils under aerobic conditions using 1 mol% of Pd-Fe₃O₄ nanocatalyst was developed. This transformation showed high efficiency with a variety of substrates and excellent functional group tolerance. Moreover, owing to its magnetic property, the catalyst could be conveniently recovered using an external permanent magnet, obviating the need for filtration before reuse. The nanocatalyst was recycled five times without loss of its catalytic activity. Further studies to understand the detailed reaction mechanism and exact reacting species of the Pd-Fe₃O₄ catalyst system are in progress. Under the optimized reaction conditions, high yields, good functional group tolerance, and efficient recyclability of the Pd-Fe₃O₄ nanocrystals indicate that this system exhibits great applicability for large scale applications. Further research into various Pd-Fe₃O₄-catalyzed reactions is currently under progress in our laboratory.

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Notes and references

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 † Electronic Supplementary Information (ESI) available: [details of any supplementary information available should be included here]. See DOI: 10.1039/b000000x/
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