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Silver nitrate promoted synthesis of tetraphenyl derivatives and diaryl ketones

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Dedicated to Prof. Tien-Yau Luh on the occasion of his 76th anniversary

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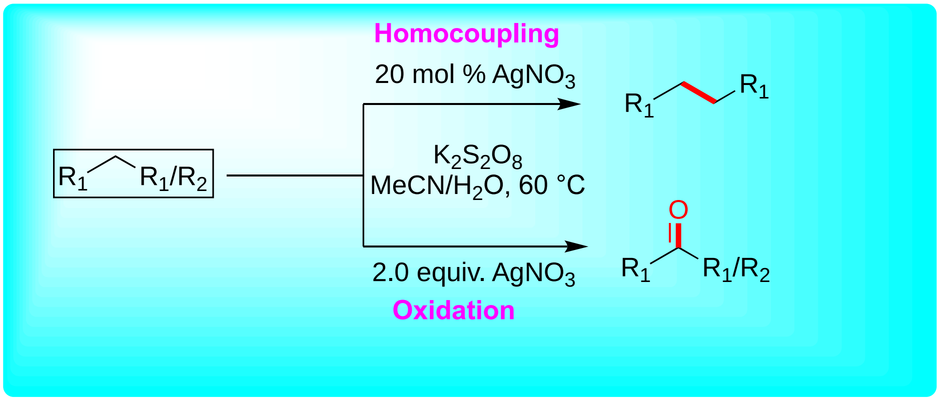
Abstract

Here in we report a novel silver nitrate-promoted oxidative pathway for the efficient synthesis of tetra substituted aryl derivatives (homodimers) and diaryl ketones. The oxidation of sp^3 C-H bond has been extensively studied by tuning the amount of the silver nitrate to afford the tetraphenyl or diaryl ketone, respectively with good selectivity under mild and simple conditions. This developed protocol offers a facile and general route to access a variety of value of tetraphenyl and diaryl ketones derivatives with moderate to good yield.



Keywords: C(sp^3)-H bond, Oxidation, Silver nitrate, Homocoupling, Aryl ketones

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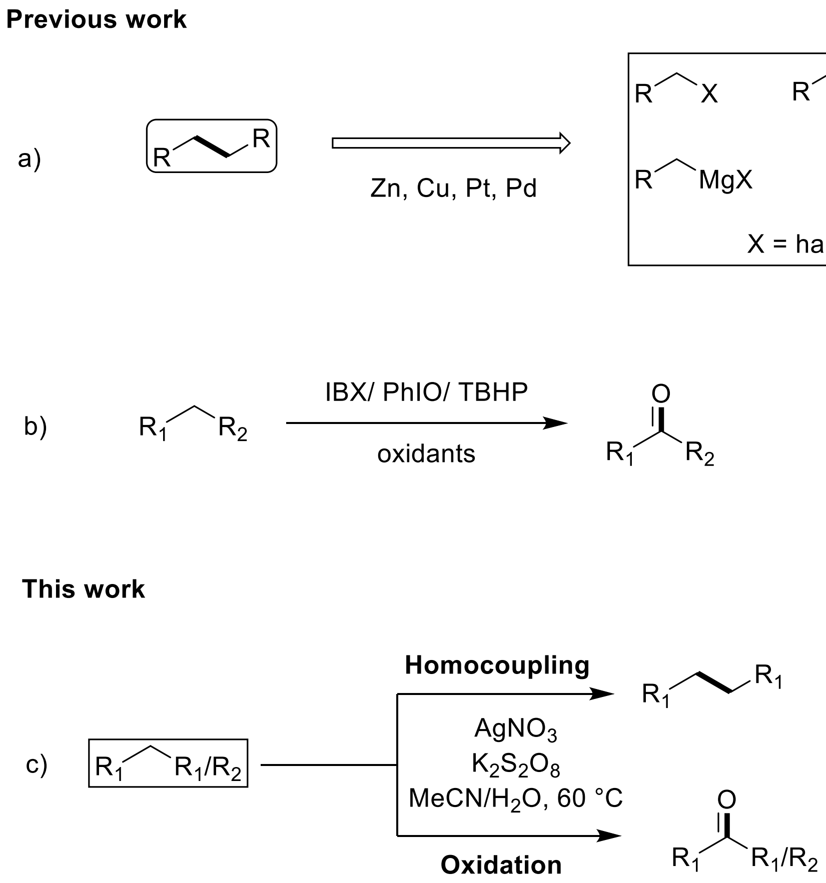
Introduction

The selection of carbon-carbon (C-C) bond formation is indispensable in organic synthetic chemistry, being extensively utilized for the synthesis of numerous derivatives in various applications. Despite the array of methods available for C-C bond formations, C(sp^3)-H bond activation is still a rare occurrence in synthetic transformations. The di-benzyl core structure is a highly versatile scaffold, offering the potential for the synthesis of a range of compounds in natural products and pharmaceuticals. [1-4] The most conventional method for obtaining di-benzyl derivatives is reducing carbon-carbon multiple bonds. [5, 6] The oxidative homo coupling reaction imposes valuable tools, and the use of oxidizing reagents triggered the transformation of toluene derivatives into dibenzylic compounds. [7] Given their significance, various metal-catalyzed methods (Rh, Cu, Pt, Pd) have been developed utilizing benzyl magnesium halide, benzyl boronic acid, phenyl acetic acid, benzyl zinc bromide through a homocoupling approach (Scheme 1a). [8-11] In recent years, the rapid advancement of Ag-catalyzed transformations in organic synthesis has overcome the early idea that silver impeded catalytic activity. Silver catalysts combined with $K_2S_2O_8$ allow for various oxidative C-H bond functionalizations to generate distinct C-C, C-O, and C-N bonds. [12-14] Herein, a novel silver-mediated oxidative homocoupling strategy is presented, facilitating the coupling of two molecules of the same substrate. In contrast, diarylketones are well known compounds that are widely employed in the synthesis of various applications. [15, 16] The oxidation of sp^3 C-H bonds has been extensively studied as an effective way to convert alkylarenes into their respective ketones, yet it remains largely underexplored. However, several methods have been known for the synthesis of aromatic ketones such as Friedel Crafts acylation, [17] transition-metal catalyzed coupling reactions, [18] or by oxidation of alcohols into ketones. [19] Tremendous progress has been made through the use of catalytic amounts of transition metal complexes, and various oxidants (Scheme 1b). [20-23] In addition, the Xu group developed potassium *tert*-butoxide promoted oxidative process by using molecular oxygen. [24] Based on the literature mentioned above, there are certain drawbacks, such as toxic, expansive catalysts, explosive oxidants, and hostile environments. We have successfully overcome and uncovered an effective way to achieve oxidation of benzylic C(sp3)-H via a silver-catalyzed reaction. Based on the above literature precedent, we show that the oxidation of benzylic C(sp^3)-H can be achieved through a silver-catalyzed reaction. By controlling the equivalence of silver nitrate in the oxidative process, two selective products were obtained under mild conditions (Scheme 1c).

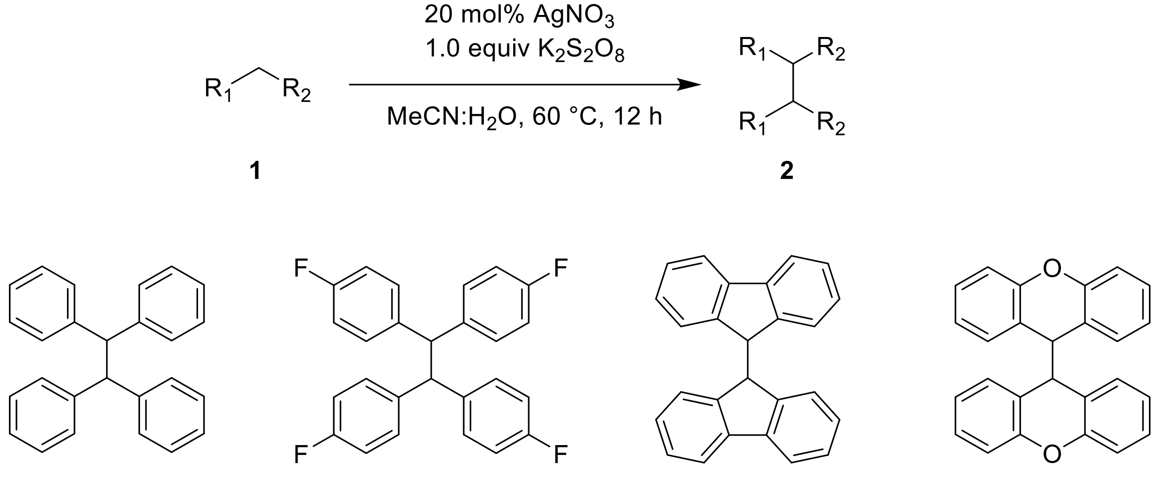


Scheme 1. Previous and this work

Results and Discussion

At the outset, we assessed the viability of the oxidative reaction of diphenylmethane (**1a**, 0.2mmol) and $AgNO_3$ (0.04 mmol), $K_2S_2O_8$ (0.2 mmol) in acetonitrile and water (1:1) ratio at 60˚C for 12 h which afforded a homocoupling dimer of tetaphenyl product **2a** in 38%. With this result in hand, we prepared a number of derivatives of benzylic substrates (Table 1). Bis (4-fluorophenyl) methane was tolerated under this condition to get the dimer product **2b**. Next, cyclic benzylic substrates were examined. The reaction of fluorene was a useful substrate and yielded the bisfluorene **2c** in 40% yield. In addition, 9*H* – xanthene could also be employed to give the desired bixanthene **2d** in 45% yield.

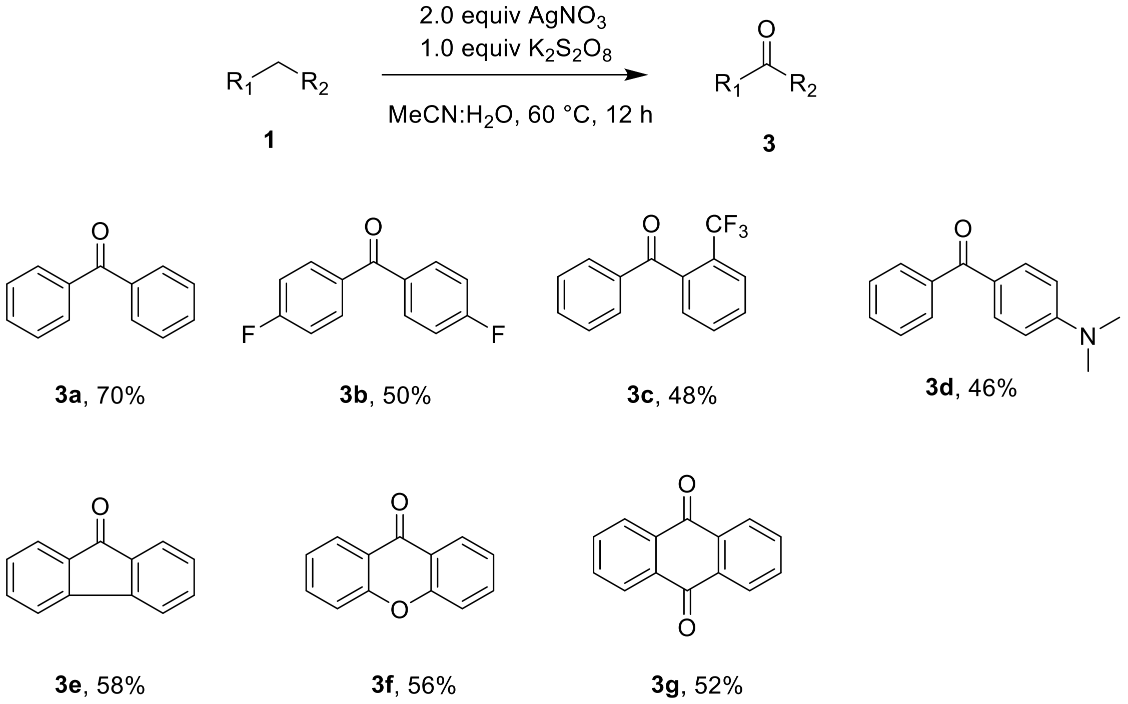
Table 1. Tetraphenyl derivatives **2**

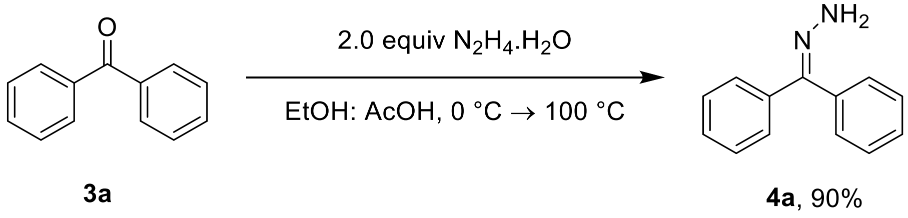


2a, 38% **2b**, 30% **2c**, 40% **2d**, 45%

Next, we embarked on the study of oxidative processes to extend our strategy by selecting silver nitrate equivalence and observing selective oxidation to give diaryl ketones (Table 2). Diphenylmethane **1a** (0.2 mmol), silver nitrate (0.4 mmol), potassium persulfate (0.2 mmol) in acetonitrile and water (1:1) ratio at 60˚C for 12-24 h reaction was performed. The yield of product **3a** was found to be 70% yield. We assessed the broad applicability of the oxidative transformation by utilizing a range of diaryl methane compounds. The introduction of fluoro group **3b** was tolerated. Electron-deficient diaryl methane derivative was employed as starting substrate, resulting of **3c** in a 48% yield. Moreover, the incorporation of a dimethylamine moiety on a diaryl methane substrate was also evaluated, yielding **3d** with moderate results. The reaction progressed smoothly for the cyclic derivatives of fluorene, and 9*H*-xanthene substrates to give the corresponding ketone derivatives (**3e-f**). The oxidation of 9, 10-dihydroanthracene to anthracne-9, 10-dione **3g** was observed upon reaction. The process was further demonstrated through its application to the synthesis of a hydrazine derivative from benzophenone, thus illustrating its synthetic utility (Scheme 2).

Table 2. Diaryl ketones **3**.





Scheme 2. Synthetic transformations

Conclusions

We have developed a selective and efficient single protocol for the synthesis of symmetrical tetraphenyl and diaryl ketone derivatives by controlling the equivalence of silver nitrate. The oxidative silver-catalyzed homocoupling was successfully implemented with both acyclic and cyclic substrates, thus extending the scope of the oxidation reaction for the effective production of diaryl ketones. The versatility of synthetic transformations has been made evident through their practical applications.

Experimental Section

General. All chemicals were purchased from commercial vendors (Sigma Aldrich, Alfa Aesar, TCI, and matrix scientific) and used directly without further purification unless otherwise noted. Well-cleaned and oven-dried glassware was used for the experiments. The reaction was monitored by thin layer chromatography (TLC), purchased as pre-coated with silica gel 60 F254 from Merck. Column chromatography was performed using the MACHEREY-NAGEL silica gel 60 mit with a mixture of ethyl acetate/hexane or hexane as an eluent. ^{1}H NMR spectra were recorded on 400 MHz & 600 MHz, ^{13}C-NMR spectra were recorded on 100 MHz & 151 MHz, and Varian Mercury spectrometer using $CDCl_3$ as solvent. The spectra were recorded and presented as chemical shifts (ppm). The multipliers were provided in s (singlet), d (doublet), t (triplet), q (quartet), br (broad singlet), m (multiplet), and dd (doublet of doublet). The coupling constants (*J*) were reported in Hz.

General procedure for synthesis of tetraphenyl derivatives. A 15 mL vial was charged with **1a-d** (0.2 mmol), silver nitrate (0.04 mmol), potassium persulfate (0.2 mmol) in acetonitrile and water (1:1) ratio were added. The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 12-24 h and the reaction was carried out. After completion of the reaction extracted with ethyl acetate (3 mL x 5), the organic layer was combined and washed with brine solution (5 mL). The organic layer was dried over anhydrous $MgSO_4$, filtered and concentrated under vacuum. The crude product was purified by column chromatography (Hexane, silica gel) and obtained as solid **2a-2d**. Furthermore, the obtained desired products were characterized by NMR, the data are shown given below and the ^{1}H-NMR and ^{13}C-NMR spectra of the products were matched with the literature data. [25, 26]

General procedure for synthesis of aryl ketones derivatives. A 15 mL vial was charged with **1a-g** (0.2 mmol), silver nitrate (0.4 mmol), potassium persulfate (0.2 mmol) in the acetonitrile and water (1:1) ratio were added. The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 12-24 h and the reaction was carried out. After completion of the reaction extracted with ethyl acetate (3 mL x 5), organic layer combined and washed with brine solution (5 mL). The organic layer was dried over anhydrous $MgSO_4$, filtered and concentrated under vacuum. The crude product was purified by column chromatography (Hexane, silica gel) and obtained as solid **3a-3g**. Furthermore, the obtained desired products were characterized by NMR, the data are shown below and the ^{1}H-NMR & ^{13}C-NMR spectra of the products were matched with the previous literatures. [24, 27-29]

1,1,2,2-Tetraphenylethane (2a).[30] After the reaction based on the general procedure: Diphenyl methane (0.2 mmol), silver nitrate (0.04 mmol), potassium persulfate (0.2 mmol) in acetonitrile and water (1:1) ratio were added. The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 12 h. The crude mixture was purified by column chromatography (silica gel) using hexane as solvent and obtained as white solid (26 mg, 38%); mp 225-226 °C ; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.18-7.16 (m, 8H), 7.13-7.09 (m, 8H), 7.03-7.00 (m, 4H), 4.78 (s, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ143.6, 128.7, 128.3, 126.0, 56.5.

1,1,2,2-Tetrakis(4-fluorophenyl)ethane (2b).[30] After the reaction according to the general procedure: bis(4-fluorophenyl)methane (0.2 mmol), silver nitrate (0.04 mmol), potassium persulfate (0.2 mmol) in acetonitrile and water (1:1) ratio were added. The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 12 h. The crude mixture was purified by column chromatography (silica gel) using hexane as solvent and obtained as white solid (25 mg, 30%); **mp** 312-313 °C ;^{1}H-NMR (600 MHz, $CDCl_3$) δ 7.05-7.03 (m, 8H),6.84-6.81 (m, 8H), 4.62 (s, 2H); ^{13}C-NMR (151 MHz, $CDCl_3$) δ 161.3 (d, J_{C-F} = 246.5 Hz), 138.7 (d, J_{C-F} = 3 Hz), 129.8 (d, J_{C-F} = 8.0 Hz), 115.5 (d, J_{C-F} = 21 Hz), 55.3.

9*H*,9'*H*-9,9'-Bifluorene (2c).[7] After the reaction according to the general procedure: 9*H*-fluorene (0.2 mmol), silver nitrate (0.04 mmol), potassium persulfate (0.2 mmol) in the acetonitrile and water (1:1) ratio were added. The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 24 h. The crude mixture was purified by column chromatography (silica gel) using hexane as solvent and obtained as white solid (26.5 mg, 40%); mp 241-242 °C ; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.0 Hz, 4H), 7.29 (d, *J* = 8.0 Hz, 4H),7.10 (t, *J* =

8.0 Hz, 4H), 6.96 (d, *J* = 8.0 Hz, 4H), 4.85(s,2H); ^{13}C-NMR (100 MHz, CDCl$_3$) δ 144.6, 141.5, 127.3, 126.7, 124.0, 119.6, 49.8

9*H*,9'*H*-9,9'-Bixanthene (2d).[31] After the reaction according to the general procedure: 9H-xanthene (0.2 mmol), silver nitrate (0.04 mmol), potassium persulfate (0.2 mmol) in the ratio of acetonitrile and water (1:1). The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 24 h. The crude mixture was purified by column chromatography (silica gel) using hexane as solvent and obtained as white solid (33 mg, 45%); mp 201-202 °C ; ^{1}H-NMR (400 MHz, CDCl$_3$) δ 7.24-7.20 (m, 4H), 6.96-6.88 (m, 8H), 6.68 (dd, *J* = 8.0 Hz, 4.0 Hz, 4H), 4.21(s, 2H); ^{13}C-NMR (100 MHz, CDCl$_3$) δ 153.0, 129.1, 128.1, 122.6, 121.8, 115.8, 49.5

Benzophenone (3a).[32] After the reaction based on the general procedure: Diphenyl methane (0.2 mmol), silver nitrate (0.4 mmol), potassium persulfate (0.2 mmol) was added in acetonitrile and water (1: 1) ratio. The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 24 h. The crude mixture was purified by column chromatography (silica gel) using hexane as solvent and obtained as white solid (25 mg, 70%); mp 48-49 °C ; ^{1}H-NMR (400 MHz, CDCl$_3$) δ 7.82-7.80 (m, 4H), 7.61-7.58 (m, 2H), 7.51-7.47 (m,4H); ^{13}C-NMR (100 MHz, CDCl$_3$) δ 196.9, 137.7, 132.6, 130.2, 128.4.

Bis(4-fluorophenyl)methanone (3b).[33] After the reaction according to the general procedure: bis(4-fluorophenyl)methane (0.2 mmol), silver nitrate (0.4 mmol), potassium persulfate (0.2 mmol) in the acetonitrile and water (1:1) ratio were added. The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 24 h. The crude mixture was purified by column chromatography (silica gel) using hexane as solvent and obtained as white solid (22 mg, 50%); mp 101-102 °C ; ^{1}H-NMR (400 MHz, CDCl$_3$) δ 7.84-7.80 (m, 4H), 7.19-7.15 (m, 4H); ^{13}C-NMR (100 MHz, CDCl$_3$) δ 193.8, 165.3 (d, J_{C-F} 254 Hz), 133.7 (d, J_{C-F} 3 Hz), 132.5 (d, J_{C-F} 8 Hz), 115.5 (d, J_{C-F} 22 Hz)

Phenyl(2-(trifluoromethyl)phenyl)methanone (3c).[34]After the reaction according to the general procedure: 1-benzyl-2-(trifluoromethyl)benzene (0.2 mmol), silver nitrate (0.4 mmol), potassium persulfate (0.2 mmol) in acetonitrile and water (1:1) ratio were added. The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 24 h. The crude mixture was purified by column chromatography (silica gel) using hexane as solvent and obtained as white solid (24 mg, 48%); mp 60-61 °C ;^{1}H-NMR (400 MHz, CDCl$_3$) δ 7.79-7.77 (m, 3H), 7.64-7.58 (m, 3H), 7.47-7.43 (m, 2H), 7.40-7.37 (m, 1H); ^{13}C-NMR (100 MHz, CDCl$_3$) δ 198.9, 138.4, 136.5, 134.0, 131.5, 130.3, 129.9, 128.6, 128.2, 126.8 (q, J_{C-F} =4.6 Hz), 123.7 (d, J_{C-F} 272.2Hz)

(4-(Dimethylamino)phenyl)(phenyl)methanone (3d).[32] After the reaction according to the general procedure: 4-benzyl-*N,N*-dimethylaniline (0.2 mmol), silver nitrate (0.4 mmol), potassium persulfate (0.2 mmol) in acetonitrile and water (1:1) ratio were added. The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 12 h. The crude mixture was purified by column chromatography (silica gel) using hexane as solvent and obtained as white solid (21 mg, 46%); mp 89-90 °C ;^{1}H-NMR (400 MHz, CDCl$_3$) δ 7.80 (d, *J* 8.0 Hz, 2H), 7.73 (d, *J* 4.0 Hz, 2H), 7.53-7.473 (m, 3H), 6.68 (d, *J* 8.0 Hz, 2H); ^{13}C-NMR (100 MHz, CDCl$_3$) δ 195.3, 153.4, 139.4, 132.9, 131.2, 129.6, 128.1, 124.9, 110.7, 40.2.

9H-Fluoren-9-one (3e).[35] After the reaction according to the general procedure: 9H-fluorene (0.2 mmol), silver nitrate (0.4 mmol), potassium persulfate (0.2 mmol) in acetonitrile and water (1:1) ratio were added. The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 12 h. The crude mixture was purified by column chromatography (silica gel) using hexane as solvent and obtained as white solid (21 mg, 58%); mp 82-83 °C ;^{1}H-NMR (400 MHz, CDCl$_3$) δ 7.76 (d, *J* 7.4 Hz, 2H), 7.54-7.47 (m, 4H), 7.30 (td, *J* 7.36 Hz, *J* 1.24Hz, 2H); ^{13}C-NMR (100 MHz, CDCl$_3$) δ 192.4, 144.6, 134.8, 134.3, 129.2, 124.5, 120.4.

9*H*-Xanthen-9-one (3f).[36] After the reaction according to the general procedure: 9H-xanthene (0.2 mmol), silver nitrate (0.4 mmol), potassium persulfate (0.2 mmol) was added in acetonitrile and water (1: 1) ratio. The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 24 h. The crude mixture was purified by column chromatography (silica gel) using hexane as solvent and obtained as white solid (22 mg, 56%); mp 171-172 °C ; ^{1}H-NMR (600 MHz, $CDCl_3$) δ 8.36-8.35 (m, 2H), 7.75-7.72 (m, 2H), 7.52-7.50 (m, 2H), 7.41-7.38 (m, 2H); ^{13}C-NMR (151 MHz, $CDCl_3$) δ177.4, 156.4, 135.0, 126.9, 124.1, 118.1, 116.7.

Anthracene-9,10-dione (3g).[37] After the reaction according to the general procedure: 9,10-dihydroanthracene (0.2 mmol), silver nitrate (0.4 mmol), potassium persulfate (0.2 mmol) in acetonitrile and water (1:1) ratio were added. The resulting mixture was stirred under nitrogen atmosphere at 60 °C for 24 h. The crude mixture was purified by column chromatography (silica gel) using hexane as solvent and obtained as white solid (22 mg, 52%); mp 283-285 °C ; ^{1}H-NMR (600 MHz, $CDCl_3$) δ 8.33 (q, *J* 6.0 Hz, 4H), 7.82 (q, *J* 6.0 Hz, 4H); ^{13}C-NMR (151 MHz, $CDCl_3$) δ 183.3, 134.3, 133.7, 127.4.

(Diphenylmethylene)hydrazine (4a).[38] A mixture of benzophenone (0.2 mmol), hydrazine monohydrate (2.0 equiv.), and acetic acid (0.06 mmol) was stirred at 100 °C for 21 h. After cooled to room temperature, solvent and volatile materials were removed under reduced pressure to afford corresponding product as a white solid (35 mg, 90%) ; mp 94-95 °C ; ^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.52-7.44 (m, 5H), 7.29-7.26 (m, 5H), 5.42 (bs, 2H); ^{13}C-NMR (100 MHz, $CDCl_3$) δ 149.2, 138.5, 133.1, 129.5, 128.2, 126.6.

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Supplementary Material

For NMR spectra see the Supplementary Material.

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