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Palladium-Catalyzed Oxidative Direct *ortho*-C–H Acylation of Arenes with Aldehydes under Aqueous Conditions

Fuhong Xiao,*[a] Shuqing Chen,[a] Huawen Huang,[a] and Guo-Jun Deng*[a]

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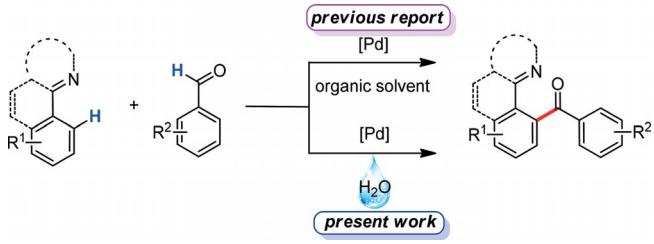
Palladium-catalyzed *ortho*-acylation of arenes with aldehydes in the presence of *tert*-butyl hydroperoxide (TBHP) as the oxidant under aqueous conditions has been demonstrated. The acylation reaction exhibits excellent regioselectivity and wide functional group tolerance.

Introduction

Diaryl ketones are fundamental building blocks in the synthesis of pharmaceuticals, natural products, functional materials, and agrochemicals.[1] The traditional methods for the synthesis of aryl ketones mainly rely on Friedel–Crafts acylation of arenes in the presence of Lewis acids or on oxidation of the corresponding secondary alcohols.[2] In recent years, great efforts have been paid to the development of direct acylation without the use of acids.[3]

Transition-metal-catalyzed oxidative acylations of aromatic sp^2 C–H bonds in proximity to various directing groups – such as pyridines,[4] oximes,[5] anilides,[6] indole,[7] 2-arylbenzoxazoles,[8] *N*-benzyltriflamides,[9] azoxybenzenes,[10] and others[11] – with aldehydes have been successfully exploited (Scheme 1). For example, in 2009, Cheng reported the first example of palladium-catalyzed acylation of 2-arylpyridines through *ortho*-C–H bond activation with aldehydes to give diaryl ketones in dry xylene.[4a] Shortly afterwards, Li found that both aromatic and aliphatic aldehydes could be successfully applied in this reaction under neat conditions.[4b] In 2010, Yu and co-workers utilized oximes as a directing group for direct C–H bond acylation, cross-coupling aromatic oximes and aldehydes in toluene.[5a] In 2011, the groups of Yu, Kwong, Wang, and Li independently published their results on the acylation of anilides with aldehydes.[6] Besides aldehydes, we found that alcohols could also be used as efficient acylating reagents through in situ oxidation.[12]



Scheme 1. C–H bond acylation with aldehydes.

However, most of the procedures described above were carried out in organic solvents. From the perspective of green chemistry, water is a desirable solvent for chemical transformation because of its natural, inexpensive, nontoxic, and environmentally friendly character.[13] During the last few years, occasional examples of acylation with aldehydes in water have been reported. In 2013, Novák and coworkers further improved the acylation of anilides with aromatic aldehydes. Good to excellent yields of the desired products were obtained under aqueous conditions.[14a] In spite of this, direct acylation of arenes with aldehydes is still very little exploited. Here we wish to describe a new palladium-catalyzed sp^2 C–H activation reaction for the synthesis of aryl ketones from aldehydes with use of water as the sole solvent. The utilization of aqueous media and anionic surfactants enables high efficiency of this transformation through "on water" effect catalysis[15] (Scheme 1).

Results and Discussion

In our initial study, acetophenone *O*-methyl oxime (**1a**) and benzaldehyde (**2a**) were chosen as model substrates for optimization of the reaction conditions, and selected results are summarized in Table 1. To our delight, a 22% yield of the desired product was obtained in the presence of 5 mol-% $PdCl_2$ and 2 equiv. of *tert*-butyl hydrogen peroxide (TBHP) in H_2O at 50 °C after 12 h (Entry 1). To improve the solubility of the reactants in water, 5 mol-% sodium dodecylsulfate (SDS) was added to the system. Various palladium catalysts including $Pd(OAc)_2$, $Pd(CH_3CN)Cl_2$, and $Pd(acac)_2$ were examined (Entries 3–5), and of them

[a] Key Laboratory for Environmentally Friendly Chemistry and Application, Ministry of Education, College of Chemistry, Xiangtan University,
Xiangtan 411105, P. R. China
E-mail: fhxiao@xtu.edu.cn
gjdeng@xtu.edu.cn

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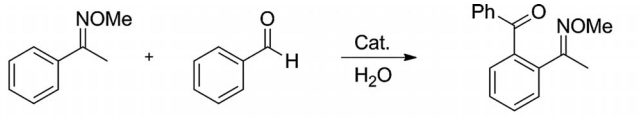
$Pd(OAc)_2$ was found to be most effective in this coupling reaction. Variation of the phase-transfer catalysts showed that SDS is superior to other phase-transfer catalysts such as TBAB, TBAI, SDBS (sodium dodecylbenzenesulfonate), 18-C-6 (18-crown-6), or dibenzo-18-crown-6 (Entries 6–11). A screening of oxidants showed that TBHP gave the best result, with TBP (*tert*-butyl peroxide), BQ, CHP (cumene hydroperoxide), and O_2 being found to be inferior (Entries 12–15).

Table 1. Optimization of the reaction conditions.[a]



1a **2a** **3a**

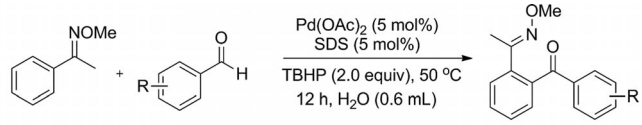
Entry	Cat.	Oxidant	PTC	Yield[b] [%]
1	$PdCl_2$	TBHP		22
2	$PdCl_2$	TBHP	SDS	42
3	$Pd(OAc)_2$	TBHP	SDS	82
4	$Pd(CH_3CN)Cl_2$	TBHP	SDS	45
5	$Pd(acac)_2$	TBHP	SDS	68
6	$Pd(OAc)_2$	TBHP	TBAB	10
7	$Pd(OAc)_2$	TBHP	TBAI	12
8	$Pd(acac)_2$	TBHP	SDBS	55
9	$Pd(OAc)_2$	TBHP	15-C-5	52
10	$Pd(OAc)_2$	TBHP	Dibenzo-18-C-6	66
11	$Pd(OAc)_2$	TBHP	18-C-6	71
12	$Pd(OAc)_2$	TBP	SDS	2
13	Pd(OAc)2	BQ	SDS	0
14	$Pd(OAc)_2$	CHP	SDS	70
15	$Pd(OAc)_2$	O_2	SDS	0

[a] Conditions: **1a** (0.4 mmol), **2a** (1 mmol), $Pd(OAc)_2$ (5 mol-%), SDS (5 mol-%), TBHP (2.0 equiv.), 50 °C, 12 h, H_2O (0.6 mL), air. [b] GC yield.

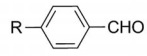
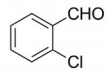
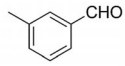
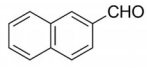
A variety of substituted aromatic aldehydes were treated with acetophenone *O*-methyl oxime (**1a**) under the optimized conditions (Table 2). In general, aromatic aldehydes containing either electron-withdrawing or -donating groups on the aromatic ring all coupled efficiently with acetophenone *O*-methyl oxime to give the desired acylated products in moderate to good yields. Aldehydes **2b**, **2c**, and **2g**, with *para*-substituted electron-donating groups [Me, OMe, and $C(CH_3)_3$], gave the corresponding products **3b**, **3c**, and **3g** in high yields (Entries 2, 3, and 7). A slightly lower yield was obtained when 4-fluorobenzaldehyde (**2d**) was used, the desired product being produced in 53% yield (Entry 4). Moderate to good yields were achieved when 2-chlorobenzaldehyde, 3-methylbenzaldehyde, and 2-naphthaldehyde were used (Entries 8–10).

Furthermore, we examined the reactions between 2-phenylpyridine and various substituted aldehydes (Table 3). We reoptimized the reaction conditions and were delighted

Table 2. Reactions between acetophenone *O*-methyl oxime and aldehydes.[a]

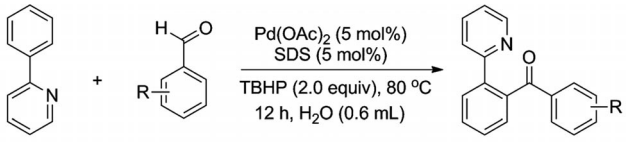


1a **2** **3**

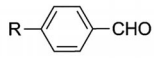
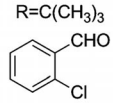
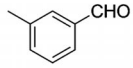
Entry	Aldehydes		Product	Yield[b] [%]
				
1	R=H	**2a**	**3a**	70
2	$R=CH_3$	**2b**	**3b**	76
3	$R=OCH_3$	**2c**	**3c**	75
4	R=F	**2d**	**3d**	53
5	R=Cl	**2e**	**3e**	78
6	R=Br	**2f**	**3f**	72
7	$R=C(CH_3)_3$	**2g**	**3g**	75
8		**2h**	**3h**	57
9		**2i**	**3i**	80
10		**2j**	**3j**	77

[a] Conditions: **1a** (0.4 mmol), **2** (1 mmol), $Pd(OAc)_2$ (5 mol-%), SDS (5 mol-%), TBHP (2.0 equiv.), 50 °C, 12 h, H_2O (0.6 mL), air. [b] Isolated yield.

Table 3. Reactions between 2-phenylpyridine and aldehydes.[a]



4a **2** **5**

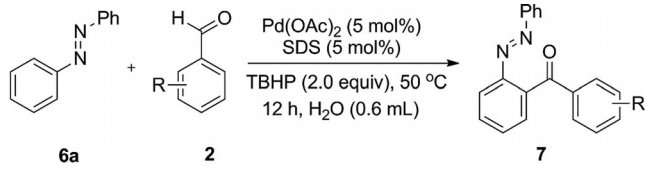
Entry	Aldehydes		Product	Yield[b] [%]
				
1	R=H	**2a**	**5a**	78
2	$R=CH_3$	**2b**	**5b**	75
3	$R=OCH_3$	**2c**	**5c**	72
4	R=F	**2d**	**5d**	80
5	R=Cl	**2e**	**5e**	73
6	R=Br	**2f**	**5f**	70
7	$R=C(CH_3)_3$	**2g**	**5g**	75
8		**2h**	**5h**	58
9		**2i**	**5i**	67

[a] Conditions: **6a** (0.4 mmol), **2** (1 mmol), $Pd(OAc)_2$ (5 mol-%), SDS (5 mol-%), TBHP (2.0 equiv.), 50 °C, 12 h, H_2O (0.6 mL), air. [b] Isolated yield.

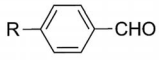
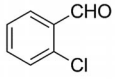
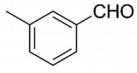
to find that the reactions could be smoothly performed at 80 °C, with the desired product **5a**, for example, being afforded in 78% yield (Entry 1). The directed acylation of 2-phenylpyridine with various aldehyde derivatives was conducted under the optimized reaction conditions. In general, reactions between 2-phenylpyridine and the aldehyde derivatives either with electron-donating groups [such as CH_3, OCH_3, or $C(CH_3)_3$] or with electron-withdrawing groups (such as F, Cl, or Br) on the aromatic ring gave moderate to good yields. The positions of the substituents on the benzaldehyde phenyl ring significantly affected the reaction yields, and the use of 2-chlorobenzaldehyde resulted in product **5h** in only 58% yield (Entry 8).

Subsequently, diphenyldiazene was employed for transformations of this kind with various aldehydes. These results are shown in Table 4. In general, aldehydes possessing electron-withdrawing groups (such as F, Cl, or Br) on the phenyl ring gave higher yields than those with electron-donating groups (such as CH_3, OCH_3). The positions of the substituents on the benzaldehyde phenyl ring also significantly affected the reaction yields, and the use of 2-chlorobenzaldehyde resulted in product **7g** in only 53% yield (Entry 7).

Table 4. Reactions between diphenyldiazene and aldehydes.[a]



6a **2** **7**

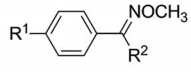
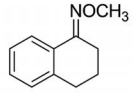
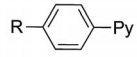
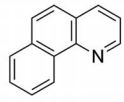
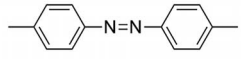
Entry	Aldehydes		Product	Yield[b] [%]
				
1	R=H	**2a**	**7a**	73
2	$R=CH_3$	**2b**	**7b**	61
3	$R=OCH_3$	**2c**	**7c**	62
4	R=F	**2d**	**7d**	71
5	R=Cl	**2e**	**7e**	73
6	R=Br	**2f**	**7f**	75
7		**2h**	**7g**	53
8		**2i**	**7h**	68

[a] Conditions: **6a** (0.4 mmol), **2** (1 mmol), $Pd(OAc)_2$ (5 mol-%), SDS (5 mol-%), TBHP (2.0 equiv.), 50 °C, 12 h, H_2O (0.6 mL), air. [b] Isolated yield.

To expand the scope of this direct acylation reaction further, we next investigated direct C–H bond acylation by cross-coupling of benzaldehyde (**2a**) with aryl ketone oximes, 2-arylpyridines, and aromatic azo compounds, with the results shown in Table 5. The initial investigations were focused on reactions between ketone oximes and benzaldehyde. All of the aryl ketone oximes bearing electron-donating (compounds **1b**, **1c**) or electron-withdrawing (compound **1d**) groups on the phenyl ring afforded the desired products in moderate to good yields (Entries 1–3). Propiophenone *O*-methyl oxime (**1e**) and benzaldehyde *O*-methyl oxime (**1f**) were also reactive toward benzaldehyde and gave the desired products in 67% and 53% yields (Entries 4 and 5). Likewise, effective transformation of the tetralone analogue **1g** was also accomplished with benzaldehyde as the coupling partner (Entry 6). Moreover, 2-arylpyridines and aromatic azo compounds were employed to react with **2a** under the optimized conditions (Entries 7–11). When benzo[*h*]quinoline (**4e**) was applied in the system, an 85% yield of the acylation product was isolated (Entry 10).

Table 5. Different directing groups for the synthesis of ketones.[a]

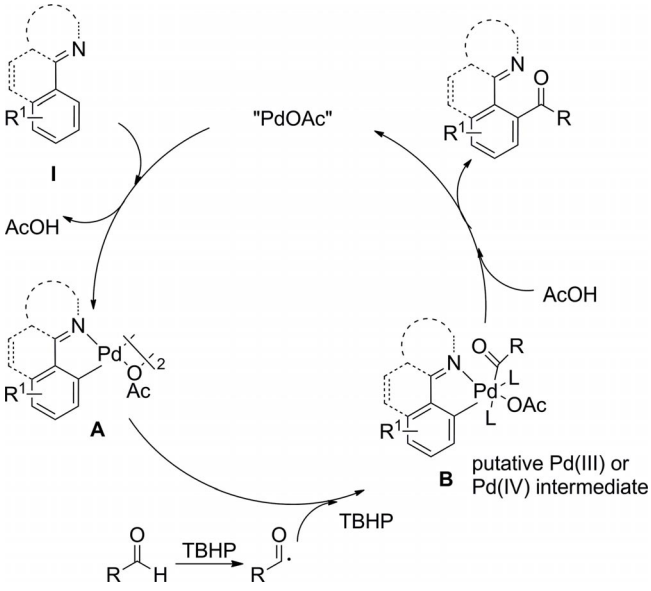
1, 4 or 6 **2a** **8**

Entry	Substrate of directing groups		Product	Yield[b] [%]
				
1	$R^1=CH_3$, $R^2=CH_3$	**1b**	**8a**	65[c]
2	$R^1=OCH_3$, $R^2=CH_3$	**1c**	**8b**	68[c]
3	$R^1=Cl$, $R^2=CH_3$	**1d**	**8c**	78[c]
4	$R^1=H$, $R^2=CH_2CH_3$	**1e**	**8d**	67[c]
5	$R^1=H$, $R^2=H$	**1f**	**8e**	53[c]
6		**1g**	**8f**	82[c]
				
7	$R=CH_3$	**4b**	**8g**	67[d]
8	$R=OCH_3$	**4c**	**8h**	52[e]
9	R=Cl	**4d**	**8i**	71[e]
10		**4e**	**8j**	85[e]
11		**6b**	**8k**	51[d]

[a] Conditions: **1** (0.4 mmol), **2a** (1 mmol), $Pd(OAc)_2$ (5 mol-%), SDS (5 mol-%), TBHP (2.0 equiv.), 50 °C, 12 h, H_2O (0.6 mL), air. [b] Isolated yield. [c] 50 °C. [d] 80 °C. [e] 100 °C.

On the basis of previous studies and our observations, a possible mechanism for the reaction is illustrated (Scheme 2). The palladium catalyst could react with **I** to form a cyclopalladated intermediate **A** by chelation-directed C–H activation, as has been confirmed in many related reports.[4b,5a,16] The Pd^{II} intermediate **A** could then react with the acyl radical, generated in situ by hydrogen atom abstraction from the aldehyde, to afford the Pd^{IV} or dimeric Pd^{III} intermediates **B** through oxidative addition. Finally, intermediates **B** could undergo reductive elimination to

generate the acylation product, and Pd^{II} would be regenerated for the next catalytic cycle.



Scheme 2. Proposed mechanism.

Conclusions

In summary, a highly efficient, environmentally benign, and regioselective synthetic method for the synthesis of diaryl ketones has been developed. The reactions took place at mild temperature, in water as the solvent. Halogen and other functional groups were well tolerated under the optimized reaction conditions. This method affords an alternative approach for the synthesis of biologically important diaryl ketones.

Experimental Section

General Methods: All experiments were carried out under air. Flash column chromatography was performed over silica gel (48–75 μm). ^{1}H NMR and ^{13}C NMR spectra were recorded with a Bruker-AV (400 and 100 MHz, respectively) instrument internally referenced to $SiMe_4$ or chloroform signals. MS analyses were performed with an Agilent 5975 GC–MS instrument (EI). High-resolution mass spectra were recorded at the Center for Mass Spectrometry, Peking University. The structures of known compounds were further corroborated by comparing their ^{1}H NMR spectroscopic data and MS data with those in the literature. All reagents were used as received from commercial sources without further purification.

General Procedure – Compound 3a: A 10 mL oven-dried reaction vessel was charged with acetophenone *O*-methyl oxime (**1a**, 70 μL, 0.4 mmol), benzaldehyde (**2a**, 100 μL, 1.0 mmol), *tert*-butyl hydroperoxide (115 μL, 0.8 mmol), palladium acetate (4.5 mg, 5 mol-%), and sodium dodecylsulfate (5.8 mg, 5 mol-%). H_2O (0.6 mL) was added to the sealed reaction vessel by syringe. The resulting solution was stirred at 50 °C for 12 h. After the mixture had cooled to room temperature the volatiles were removed under vacuum and the residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate 9:1) to give **3a** as a pale yellow solid; yield: 71 mg (70%).

{2-[1-(Methoxyimino)ethyl]phenyl}(phenyl)methanone (3a):[17] CAS number 1401733-54-7. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.71 (d, J = 7.4 Hz, 2 H), 7.52–7.48 (m, 5 H), 7.42–7.38 (m, 2 H), 3.68 (s, 3 H), 2.03 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.4, 153.9, 138.9, 138.2, 136.4, 132.4, 130.1, 129.3, 128.9, 128.5, 128.1, 127.6, 61.6, 14.3 ppm. MS (EI): *m*/*z* (%) = 253, 222, 208, 152, 77.

{2-[1-(Methoxyimino)ethyl]phenyl}(*p*-tolyl)methanone (3b):[17] CAS number 1401733-58-1. White solid, yield 76%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.62 (d, J = 8.0 Hz, 2 H), 7.52–7.48 (m, 2 H), 7.45–7.44 (m, 2 H), 7.20 (d, J = 7.9 Hz, 2 H), 3.69 (s, 3 H), 2.40 (s, 3 H), 2.03 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.3, 154.1, 143.3, 139.2, 136.4, 135.6, 130.0, 129.6, 129.0, 128.9, 128.5, 127.7, 61.7, 21.7, 14.5 ppm. MS (EI): *m*/*z* (%) = 267, 236, 222, 119, 91.

{2-[1-(Methoxyimino)ethyl]phenyl}(4-methoxyphenyl)methanone (3c, CAS 1401733–61–6):[17] White solid, yield 75%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.71 (d, J = 8.7 Hz, 2 H), 7.50–7.44 (m, 4 H), 6.89 (d, J = 8.7 Hz, 2 H), 3.86 (s, 3 H), 3.70 (s, 3 H), 2.04 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.3, 163.2, 154.2, 139.2, 136.4, 131.8, 130.1, 129.9, 128.7, 128.4, 127.8, 113.5, 61.7, 55.4, 14.7 ppm. MS (EI): *m*/*z* (%) = 283, 252, 238, 135, 77.

(4-Fluorophenyl){2-[1-(methoxyimino)ethyl]phenyl}methanone (3d): Pale yellow solid, yield 53%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.75–7.72 (m, 2 H), 7.56–7.45 (m, 4 H), 7.10–7.05 (m, 2 H), 3.66 (s, 3 H), 2.04 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.0, 165.4 (d, J = 253 Hz), 153.8, 138.7, 136.2, 134.7 (d, J = 3.2 Hz), 131.8 (d, J = 9.2 Hz), 130.2, 128.7, 128.6, 127.7, 115.4 (d, J = 21.8 Hz), 61.7, 14.3 ppm. MS (EI): *m*/*z* (%) = 271, 240, 226, 123, 95. HRMS calcd. for $C_{16}H_{15}FNO_2$ $[M + H]^+$ 272.1081; found 272.1081.

(4-Chlorophenyl){2-[1-(methoxyimino)ethyl]phenyl}methanone (3e):[5a] CAS number 1240389-12-1. White solid, yield 78%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.65 (d, J = 8.44 Hz, 2 H), 7.56–7.43 (m, 4 H), 7.37 (d, J = 8.5 Hz, 2 H), 3.67 (s, 3 H), 2.05 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.1, 153.5, 138.7, 136.6, 131.8, 130.5, 130.2, 129.3, 128.7, 128.6, 128.5, 127.6, 61.6, 14.1 ppm. MS (EI): *m*/*z* (%) = 287, 256, 152, 111, 75.

(4-Bromophenyl){2-[1-(methoxyimino)ethyl]phenyl}methanone (3f):[5b] Pale yellow solid, yield 72%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.58–7.43 (m, 8 H), 3.70 (s, 3 H), 2.05 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.3, 153.5, 138.3, 137.0, 136.0, 131.5, 130.6, 130.2, 128.7, 128.6, 127.5, 127.4, 61.6, 14.0 ppm. MS (EI): *m*/*z* (%) = 331, 300, 286, 154, 76.

[4-(*tert*-Butyl)phenyl]{2-[1-(methoxyimino)ethyl]phenyl}methanone (3g): Pale yellow solid, yield 75%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.67 (d, J = 8.06 Hz, 2 H), 7.50 (s, 2 H), 7.44–7.41 (m, 4 H), 3.70 (s, 3 H), 2.04 (s, 3 H), 1.34 (s, 9 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.1, 156.2, 154.2, 139.0, 136.5, 135.3, 129.9, 129.4, 128.8, 128.3, 127.8, 125.1, 61.5, 35.0, 31.1, 14.6 ppm. MS (EI): *m*/*z* (%) = 309, 278, 176, 117, 57. HRMS calcd. for: $C_{20}H_{24}NO_2$ $[M + H]^+$ 310.1801; found 310.1801.

(2-Chlorophenyl){2-[1-(methoxyimino)ethyl]phenyl}methanone (3h): Pale yellow solid, yield 57%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.56 (t, J = 8.12 Hz, 2 H), 7.47–7.39 (m, 5 H), 7.30–7.26 (m, 1 H), 3.88 (s, 3 H), 1.99 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 195.5, 155.8, 138.2, 138.1, 137.8, 132.4, 131.8, 131.5, 131.0, 130.6, 130.4, 128.6, 128.5, 126.3, 61.7, 15.3 ppm. MS (EI): *m*/*z* (%) = 287, 256, 152, 111, 75. HRMS calcd. for: $C_{16}H_{15}ClNO_2$ $[M + H]^+$ 288.0785; found 288.0785.

{2-[1-(Methoxyimino)ethyl]phenyl}(*m*-tolyl)methanone (3i):[5b] Pale yellow solid, yield 80%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.55–7.47 (m, 6 H), 7.34–7.26 (m, 2 H), 3.70 (s, 3 H), 2.37 (s, 3 H), 2.02 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = δ = 197.6, 154.1, 138.9, 138.1, 137.9, 136.5, 133.2, 130.1, 129.7, 128.9, 128.4, 128.0, 127.6, 126.6, 61.5, 21.2, 14.4 ppm. MS (EI): *m*/*z* (%) = 267, 236, 222, 176, 91.

{2-[1-(Methoxyimino)ethyl]phenyl}(naphthalen-2-yl)methanone (3j): Yellow solid, yield 77%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.08 (s, 1 H), 7.96–7.82 (m, 4 H), 7.59–7.50 (m, 6 H), 3.63 (s, 3 H), 2.02 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.4, 154.0, 138.9, 136.5, 135.5, 135.3, 132.3, 131.0, 130.1, 129.3, 129.0, 128.5, 128.14, 128.12, 127.7, 127.68, 126.5, 124.9, 61.5, 14.3 ppm. MS (EI): *m*/*z* (%) = 303, 272, 202, 127, 77. HRMS calcd. for: $C_{20}H_{18}NO_2$ [M + H]$^+$ 304.1332; found 304.1332.

Phenyl[2-(pyridin-2-yl)phenyl]methanone (5a):[12] CAS number 198478-48-7. White solid, yield 78%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.50 (d, *J* = 4.36 Hz, 1 H), 7.71–7.54 (m, 5 H), 7.45–7.43 (m, 1 H), 7.28–7.15 (m, 4 H), 7.06–7.01 (m, 2 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 198.1, 156.7, 148.9, 139.5, 137.9, 136.2, 132.2, 130.1, 129.4, 129.0, 128.7, 128.4, 127.9, 122.6, 121.8 ppm. MS (EI): *m*/*z* (%) = 259, 230, 182, 127, 77.

[2-(Pyridin-2-yl)phenyl](*p*-tolyl)methanone (5b):[12] CAS number 1173294-90-0. White solid, yield 75%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.39 (d, *J* = 4.30 Hz, 1 H), 7.77 (d, *J* = 7.64 Hz, 1 H), 7.62–7.47 (m, 7 H), 7.09–7.01 (m, 3 H), 2.32 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.9, 156.9, 149.0, 143.1, 139.6, 139.5, 136.2, 135.2, 130.0, 129.7, 128.9, 128.8, 128.7, 128.3, 122.8, 121.8, 21.5 ppm. MS (EI): *m*/*z* (%) = 273, 244, 182, 127, 91.

(4-Methoxyphenyl)[2-(pyridin-2-yl)phenyl]methanone (5c):[12] CAS number 1236046-45-9. White solid, yield 72%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.42 (d, *J* = 4.28 Hz, 1 H), 7.77 (d, *J* = 7.68 Hz, 1 H), 7.68 (d, *J* = 8.64 Hz, 2 H), 7.61–7.45 (m, 5 H), 7.06–7.03 (m, 1 H), 6.76 (d, *J* = 8.72 Hz, 2 H), 3.79 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.9, 162.9, 156.9, 149.1, 139.6, 139.4, 136.1, 131.8, 130.7, 130.0, 128.9, 128.7, 128.3, 122.9, 121.8, 113.3, 55.3 ppm. MS (EI): *m*/*z* (%) = 289, 260, 182, 127, 77.

(4-Fluorophenyl)[2-(pyridin-2-yl)phenyl]methanone (5d):[12] CAS number 1236046-46-0. White solid, yield 80%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.36 (d, *J* = 4.12 Hz, 1 H), 7.78–7.51 (m, 8 H), 7.07–7.00 (m, 1 H), 6.93 (t, *J* = 8.52 Hz, 2 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.6, 165.2 (d, *J* = 252.5 Hz), 156.5, 148.9, 139.3, 139.1, 136.4, 134.2 (d, *J* = 2.62 Hz), 131.9 (d, *J* = 9.23 Hz), 130.2, 128.9, 128.7, 128.5, 122.6, 122.0, 115.1 (d, *J* = 21.76 Hz) ppm. MS (EI): *m*/*z* (%) = 277, 248, 182, 127, 95.

(4-Chlorophenyl)[2-(pyridin-2-yl)phenyl]methanone (5e):[12] CAS number 1173294-91-1. White solid, yield 73%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.34 (d, *J* = 4.2 Hz, 1 H), 7.77 (d, *J* = 7.68 Hz, 1 H), 7.63–7.52 (m, 7 H), 7.23 (d, *J* = 8.4 Hz, 2 H), 7.04 (t, *J* = 5.92 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.7, 156.4, 148.8, 139.3, 139.0, 138.4, 136.4, 130.6, 130.2, 128.9, 128.58, 128.55, 128.54, 128.2, 122.4, 122.0 ppm. MS (EI): *m*/*z* (%) = 293, 264, 182, 127, 111.

(4-Bromophenyl)[2-(pyridin-2-yl)phenyl]methanone (5f):[12] CAS number 1173294-92-2. Pale yellow solid, yield 70%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.34 (d, *J* = 4.24 Hz, 1 H), 7.77 (d, *J* = 7.72 Hz, 1 H), 7.62–7.52 (m, 7 H), 7.40 (d, *J* = 8.4 Hz, 2 H), 7.05 (t, *J* = 5.88 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.9, 156.3, 148.8, 139.3, 138.9, 136.8, 136.4, 131.2, 130.7, 130.3, 128.9, 128.58, 128.56, 127.2, 122.4, 122.1 ppm. MS (EI): *m*/*z* (%) = 337, 308, 182, 127, 76.

[4-(*tert*-Butyl)phenyl][2-(pyridin-2-yl)phenyl]methanone (5g): White solid, yield 75%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.40 (d, *J* = 4.36 Hz, 1 H), 7.76 (d, *J* = 7.68 Hz, 1 H), 7.65–7.46 (m, 7 H), 7.30 (d, *J* = 8.4 Hz, 2 H), 7.02 (t, *J* = 5.98 Hz, 1 H), 1.27 (s, 9 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.8, 157.0, 156.0, 149.1, 139.7, 139.6, 136.1, 135.1, 130.0, 129.6, 128.9, 128.89, 128.2, 124.9, 122.8, 121.7, 34.9, 31.0 ppm. MS (EI): *m*/*z* (%) = 315, 286, 182, 127, 77. HRMS calcd. for: $C_{22}H_{22}NO$ [M + H]$^+$ 316.1695; found 316.1694.

(2-Chlorophenyl)[2-(pyridin-2-yl)phenyl]methanone (5h):[12] CAS number 1271322-37-2. White solid, yield 78%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.36 (d, *J* = 4.12 Hz, 1 H), 7.77 (d, *J* = 7.68 Hz, 1 H), 7.70–7.48 (m, 8 H), 7.40–7.36 (m, 1 H), 7.01 (t, *J* = 6.0 Hz, 1 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.1, 157.2, 149.0, 140.5, 139.3, 138.1, 136.1, 132.9, 131.5, 131.1, 130.5, 130.2, 129.3, 128.5, 125.9, 122.8, 121.8 ppm. MS (EI): *m*/*z* (%) = 293, 258, 230, 182, 127.

[2-(Pyridin-2-yl)phenyl](*m*-tolyl)methanone (5i):[12] CAS number 1271322-40-7. White solid, yield 67%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.38 (d, *J* = 4.36 Hz, 1 H), 7.76 (d, *J* = 7.64 Hz, 1 H), 7.62–7.46 (m, 7 H), 7.22–7.13 (m, 2 H), 7.04–7.01 (m, 1 H), 2.28 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 198.3, 156.9, 149.0, 139.7, 139.6, 137.73, 137.70, 136.2, 133.1, 130.1,129.9, 129.1, 128.8, 128.3, 127.9, 126.9, 122.7, 121.9, 21.1 ppm. MS (EI): *m*/*z* (%) = 273, 244, 182, 127, 91.

(*E*)-Phenyl[2-(phenyldiazenyl)phenyl]methanone (7a):[10c] CAS number 54184-68-8. Red solid, yield 73%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.95 (d, *J* = 7.56 Hz, 1 H), 7.77 (d, *J* = 7.12 Hz, 2 H), 7.66–7.60 (m, 3 H), 7.48–7.33 (m, 8 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.1, 152.1, 150.4, 138.5, 136.9, 132.7, 131.3, 130.8, 130.7, 129.4, 128.9, 128.8, 128.3, 122.9, 120.0 ppm. MS (EI): *m*/*z* (%) = 286, 270, 152, 105, 77.

(*E*)-[2-(Phenyldiazenyl)phenyl](*p*-tolyl)methanone (7b):[10c] Red solid, yield 61%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.94 (d, *J* = 7.08 Hz, 1 H), 7.69–7.56 (m, 5 H), 7.47 (s, 2 H), 7.36 (s, 3 H), 7.18 (d, *J* = 6.60 Hz, 2 H), 2.36 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.7, 152.1, 150.3, 143.6, 137.4, 135.9, 131.2, 130.8, 130.5, 129.6, 129.1, 128.9, 128.6, 122.9, 119.7, 21.61 ppm. MS (EI): *m*/*z* (%) = 300, 284, 165, 152, 77.

(*E*)-(4-Methoxyphenyl)[2-(phenyldiazenyl)phenyl]methanone (7c):[10c] Red solid, yield 62%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.93 (d, *J* = 7.04 Hz, 1 H), 7.77 (d, *J* = 7.52 Hz, 2 H), 7.63–7.51 (m, 5 H), 7.37 (s, 3 H), 6.86 (d, *J* = 7.60 Hz, 2 H), 3.82 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 195.7, 163.4, 152.2, 150.1, 137.8, 131.9, 131.4, 131.3, 130.8, 130.4, 128.9, 128.5, 123.0, 119.3, 113.6, 55.4 ppm. MS (EI): *m*/*z* (%) = 316, 300, 168, 135, 77.

(*E*)-(4-Fluorophenyl)[2-(phenyldiazenyl)phenyl]methanone (7d):[10c] CAS number 1421276-20-1. Red solid, yield 71%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.96 (d, *J* = 7.20 Hz, 1 H), 7.80 (s, 2 H), 7.67–7.57 (m, 3 H), 7.47 (s, 2 H) 7.37 (s, 3 H), 7.04 (d, *J* = 7.4 Hz, 2 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 195.5, 166.7, 164.2, 151.9, 150.3, 136.5, 134.8, 131.9 (d, *J* = 9.25 Hz), 131.5, 130.9 (d, *J* = 3.13 Hz), 129.4, 128.9, 128.6, 122.8, 120.2, 115.4 (d, *J* = 21.87 Hz) ppm. MS (EI): *m*/*z* (%) = 304, 199, 170, 95, 77.

(*E*)-(4-Chlorophenyl)[2-(phenyldiazenyl)phenyl]methanone (7e):[10c] CAS number 1421276-19-8. Red solid, yield 73%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.97 (d, *J* = 7.20 Hz, 1 H), 7.72–7.57 (m, 5 H), 7.46–7.34 (m, 7 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 195.8, 151.9, 150.3, 139.1, 136.9, 136.2, 131.5, 131.0, 130.9, 130.7, 129.0, 128.7, 128.6, 122.9, 120.5 ppm. MS (EI): *m*/*z* (%) = 320, 152, 139, 105, 77.

(*E*)-(4-Bromophenyl)[2-(phenyldiazenyl)phenyl]methanone (7f):[10c] CAS number 1421276-18-7. Red solid, yield 75%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.97 (d, J = 7.18 Hz, 1 H), 7.67–7.38 (m, 12 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 195.9, 151.9, 150.3, 137.3, 136.1, 131.6, 131.5, 131.0, 130.9, 130.8, 129.90, 128.7, 127.8, 122.8, 120.5 ppm. MS (EI): *m*/*z* (%) = 364, 180, 152, 105, 77.

(*E*)-(2-Chlorophenyl)[2-(phenyldiazenyl)phenyl]methanone (7g):[10c] CAS number 1421276-28-9. Red solid, yield 53%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.82 (d, J = 7.40 Hz, 1 H), 7.73 (d, J = 7.80 Hz, 1 H), 7.67–7.53 (m, 3 H), 7.41–7.26 (m, 8 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 195.7, 152.3, 151.1, 139.9, 137.1, 132.3, 132.2, 131.7, 131.4, 130.8, 130.7, 130.6, 130.0, 128.8, 126.6, 123.2, 117.7 ppm. MS (EI): *m*/*z* (%) = 320, 285, 152, 105, 77.

(*E*)-[2-(Phenyldiazenyl)phenyl](*m*-tolyl)methanone (7h):[10c] CAS number 1421276-26-7. Red solid, yield 68%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.94 (d, J = 7.48 Hz, 1 H), 7.66–7.45 (m, 7 H), 7.36–7.29 (m, 4 H), 2.33 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.2, 152.1, 150.4, 138.5, 138.1, 137.2, 133.5, 131.2, 130.7, 130.6, 129.7, 128.8, 128.7, 128.2, 126.9, 122.9, 119.8, 21.2 ppm. MS (EI): *m*/*z* (%) = 300, 284, 165, 152, 77.

1-(2-Benzoyl-4-methylphenyl)ethanone *O*-Methyl Oxime (8a):[5b] White solid, yield 65%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.70 (d, J = 7.32 Hz, 1 H), 7.53–7.49 (m, 1 H), 7.41–7.28 (m, 5 H), 3.65 (s, 3 H), 2.41 (s, 3 H), 2.01 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.8, 153.7, 138.8, 138.7, 138.3, 133.5, 132.4, 130.7, 129.5, 129.2, 128.1, 127.5, 61.5, 21.1, 14.2 ppm. MS (EI): *m*/*z* (%) = 267, 236, 222, 105, 77.

{5-Methoxy-2-[1-(methoxyimino)ethyl]phenyl}(phenyl)methanone (8b):[18] CAS number 1401733-63-8. Pale yellow solid, yield 68%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.71 (d, J = 7.48 Hz, 2 H), 7.53–7.50 (m, 1 H), 7.44–7.38 (m, 3 H), 7.06–6.98 (m, 2 H), 3.85 (s, 3 H), 3.64 (s, 3 H), 1.99 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.2, 159.8, 153.3, 140.2, 138.1, 132.5, 129.2, 128.9, 128.2, 127.5, 115.9, 114.0, 61.5, 55.5, 14.1 ppm. MS (EI): *m*/*z* (%) = 283, 252, 238, 105, 77.

{5-Chloro-2-[1-(methoxyimino)ethyl]phenyl}(phenyl)methanone (8c):[18] CAS number 1417412-29-3. Pale yellow solid, yield 78%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.70 (d, J = 7.36 Hz, 2 H), 7.56–7.49 (m, 2 H), 7.45–7.40 (m, 4 H), 3.65 (s, 3 H), 2.01 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 195.9, 152.7, 140.3, 137.5, 134.8, 134.6, 132.8, 130.1, 129.2, 128.9, 128.8, 128.3, 61.7, 14.0 ppm. MS (EI): *m*/*z* (%) = 287, 256, 242, 152, 77.

{2-[1-(Methoxyimino)propyl]phenyl}(phenyl)methanone (8d): White solid, yield 67%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.72 (d, J = 7.16 Hz, 2 H), 7.55–7.46 (m, 6 H), 7.41–7.37 (m, 2 H), 3.66 (s, 3 H), 2.57 (q, J = 7.61 Hz, 2 H), 0.99 (t, J = 7.60 Hz, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.6, 158.5, 139.5, 138.2, 135.3, 132.4, 130.0, 129.5, 129.0, 128.4, 128.1, 127.6, 61.5, 21.3, 10.4 ppm. MS (EI): *m*/*z* (%) = 267, 236, 207, 105, 77.

2-Benzoylbenzaldehyde *O*-Methyl Oxime (8e):[18] CAS number 1417412-48-6. Pale yellow solid, yield 53%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.15 (s, 1 H), 7.90 (d, J = 7.72 Hz, 1 H), 7.78 (d, J = 7.44 Hz, 2 H), 7.61–7.41 (m, 6 H), 3.85 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.0, 146.3, 138.3, 137.5, 133.2, 130.5, 130.1, 129.0, 128.9, 128.4, 127.2, 62.0, 26.2 ppm. MS (EI): *m*/*z* (%) = 239, 208, 130, 105, 77.

[8-(Methoxyimino)-5,6,7,8-tetrahydronaphthalen-1-yl](phenyl)-methanone (8f):[18] CAS number 1401733-65-0. Yellow solid, yield 82%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.71 (d, J = 7.28 Hz, 2 H), 7.50–7.46 (m, 1 H), 7.40–7.28 (m, 4 H), 7.20 (d, J = 7.3 Hz, 1 H), 3.54 (s, 3 H), 2.80 (t, J = 5.92 Hz, 2 H), 2.57 (t, J = 6.68 Hz, 2 H), 1.89–1.82 (m, 2 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.7, 151.7, 140.5, 138.7, 138.3, 132.0, 129.5, 128.9, 128.4, 128.1, 126.3, 61.5, 30.3, 21.1 ppm. MS (EI): *m*/*z* (%) = 279, 248, 202, 115, 77.

[5-Methyl-2-(pyridin-2-yl)phenyl](phenyl)methanone (8g):[12] CAS number 198478-62-5. White solid, yield 67%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.34 (d, J = 4.36 Hz, 1 H), 7.71–7.65 (m, 3 H), 7.54–7.23 (m, 7 H), 7.01–6.95 (m, 1 H), 2.45 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 198.4, 156.6, 148.9, 139.4, 138.6, 137.9, 136.8, 136.1, 132.2, 130.8, 129.6, 129.3, 128.5, 127.9, 122.4, 121.6, 21.1 ppm. MS (EI): *m*/*z* (%) = 273, 244, 196, 167, 77.

[5-Methoxy-2-(pyridin-2-yl)phenyl](phenyl)methanone (8h):[12] CAS number 1173294-82-0. White solid, yield 52%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.31 (d, J = 4.28 Hz, 1 H), 7.73–7.68 (m, 3 H), 7.52–7.36 (m, 3 H), 7.26 (d, J = 5.0 Hz, 2 H), 7.14–7.11 (m, 1 H), 7.05 (d, J = 2.20 Hz, 1 H), 6.97–6.92 (m, 1 H), 3.88 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.9, 159.8, 156.3, 148.8, 140.8, 137.7, 136.2, 132.3, 132.0, 129.9, 129.3, 128.0, 122.0, 121.3, 116.1, 114.0, 55.53 ppm. MS (EI): *m*/*z* (%) = 289, 260, 212, 169, 77.

[5-Chloro-2-(pyridin-2-yl)phenyl](phenyl)methanone (8i):[12] CAS number 1236046-60-8. White solid, yield 71%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.34 (d, J = 4.40 Hz, 1 H), 7.73–7.66 (m, 3 H), 7.59–7.54 (m, 2 H), 7.51–7.39 (m, 3 H), 7.29–7.26 (m, 2 H), 7.04–7.01 (m, 1 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 196.6, 155.5, 149.1, 140.9, 137.9, 137.3, 136.4, 134.8, 132.6, 130.1, 129.9, 129.3, 128.9, 128.1, 122.4, 122.1 ppm. MS (EI): *m*/*z* (%) = 293, 264, 216, 153, 77.

(Benzo[*h*]quinolin-10-yl)(phenyl)methanone (8j):[12] CAS number 198478-50-1. White solid, yield 85%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 8.51 (d, J = 3.0 Hz, 1 H), 8.12–8.05 (m, 2 H), 7.91 (d, J = 8.8 Hz, 1 H), 7.81–7.73 (m, 4 H), 7.63 (d, J = 7.0 Hz, 1 H), 7.43–7.28 (m, 4 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 198.5, 147.0, 144.5, 139.2, 138.8, 135.2, 133.7, 131.6, 129.1, 128.9, 128.6, 128.0, 127.7, 127.6, 126.9, 126.3, 126.1, 121.6 ppm. MS (EI): *m*/*z* (%) = 283, 254, 206, 178, 127, 77.

(*E*)-[5-Methyl-2-(*p*-tolyldiazenyl)phenyl](phenyl)methanone (8k):[10c] CAS number 1421276-37-0. Red solid, yield 51%. ^{1}H NMR ($CDCl_3$, 400 MHz): δ = 7.85 (d, J = 8.16 Hz, 1 H), 7.76 (d, J = 7.32 Hz, 2 H), 7.48–7.42 (m, 2 H), 7.38–7.28 (m, 5 H), 7.10 (d, J = 8.04 Hz, 2 H), 2.48 (s, 3 H), 2.33 (s, 3 H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ = 197.5, 150.2, 148.5, 141.7, 141.4, 138.6, 136.7, 132.6, 131.5, 129.5, 129.3, 129.1, 128.2, 122.7, 120.2, 21.4 ppm. MS (EI): *m*/*z* (%) = 314, 298, 165, 152, 91.

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