DOI: 10.1002/adsc.201601353

Advanced Synthesis & Catalysis

Green and Practical Oxidative Deoximation of Oximes to Ketones or Aldehydes with Hydrogen Peroxide/Air by Organoselenium Catalysis

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Received: December 8, 2016; Revised: February 11, 2017; Published online: ■■ ■■, 0000

Supporting information for this article is available under http://dx.doi.org/10.1002/adsc.201601353.

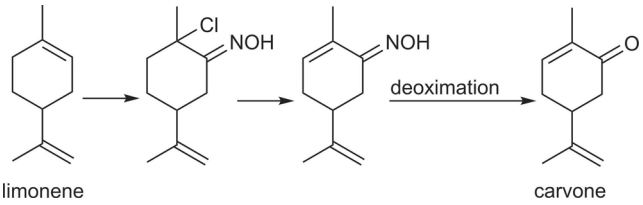
Abstract: Organoselenium-catalyzed oxidative deoximations afforded ketones and aldehydes under mild conditions. The reactions employ hydrogen peroxide and air as clean oxidants and lead to a waste-free and metal-free deprotection protocol for carbonyl protection strategies as well as the green synthesis of ketones and aldehydes. The mechanisms of this interesting organoselenium-catalyzed reaction have been investigated by control experiments as well as the selenium 77 nuclear magnetic resonance (^{77}Se NMR) tests. This novel reaction largely expands the application scope of organoselenium catalysis.

Keywords: aldehydes; deoximation; ketones; organoselenium catalysis; oximes

Introduction

Organoselenium catalysis is a distinctive research topic just unfolding during the last decade.[1] In comparison with many transition metals, selenium is a metabolizable element that will not accumulate in the body and is thus safer for both organisms and the environment.[2] Selenium is especially rich in China and therefore less expensive than many noble metals. Moreover, organoselenium compounds are usually less toxic than inorganic selenium compounds. Some of them such as diphenyl diselenide and the anti-inflammatory analgesic drug *ebselen* [2-phenylbenzisoselenazol-3(2*H*)-one][3] show only moderate toxicity. Therefore, the emerging of organoselenium catalysis brings unprecedented opportunities for organic chemists to discover new synthetic reactions,[4] especially in green preparations of the industrial-oriented chemicals owing to the green features of organoselenium catalysis.[5–8] In addition, many organoselenium catalysts are robust and can be recycled and reused many times without obvious deactivation.[5a,7,8] Our group aims to develop heterogeneous[8,9] or homogeneous[6,7] catalytic technologies with application potential in industry. During the past few years, we have reported a series of recoverable and scalable organoselenium-catalyzed green transformations.[6–8]

On the other hand, since oximes are stable and easily prepared, oximation–deoximation strategies has been frequently employed in protection, characterization, and purification of carbonyl compounds.[10] This protocol has also been widely employed in synthesis including the total synthesis of *erythronolide A*.[11] Moreover, because oximes can also be obtained from non-carbonyl compounds,[12] the deoximation strategy can provide efficient approaches to transform other functionalities into the carbonyl moiety. The production of the spice *carvone* is a typical example (Scheme 1) in the field.[13] However, although deoximation has become a more and more important transformation in organic chemistry,[10,14] the current deoximation methods usually require the use of metal or non-metal chemical reagents that can generate large amounts of waste,[15] halogen- or nitro group-contain-



Scheme 1. Synthesis of *carvone* from non-carbonyl starting materials *via* deoximation.

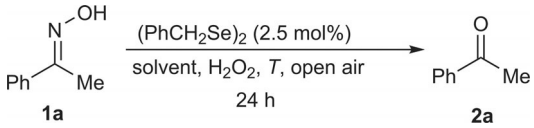
ing solvents or additives,[16] or expensive metal catalysts.[17] To solve the above mentioned issues and with the rapid developments in materials chemistry, some nano catalysts were also developed and used in deoximation reactions under greener and milder conditions.[18] In our own cases, during the continuing studies on organoselenium catalysis,[6–8] we observed an unexpected deoximation method. The reaction is metal-free, additive-free and employs H_2O_2/air as the clean oxidant. The organoselenium catalyst was easily available[19] and sufficiently robust to be recycled and reused. Herein, we wish to report our findings.

Results and Discussion

The deoximation of acetophenone oxime **1a** was initially chosen as the model reaction to optimize the conditions. On heating 1 mmol of **1a** with 0.3 mmol of H_2O_2 in the presence of 0.025 mmol of $(PhCH_2Se)_2$ in 2 mL of ethanol at 80 °C in open air for 24 h, the desired product **2a** was generated in only 37% GC yield (Table 1, entry 1). The reaction performed in water was also tested, but led to poor product yield as well (Table 1, entry 2). Using MeCN as solvent, the yield of **2a** was enhanced to 68% (Table 1, entry 3). The reaction in 1,4-dioxane led to **2a** in reduced 65% yield (Table 1, entry 4) while the non-polar solvents such as cyclohexane, toluene and petroleum ether were all unfavorable for the reaction (Table 1, entries 5–7). Reduced H_2O_2 dosages depressed the reaction (Table 1, entries 8–10), but using more than 30% of H_2O_2/**1a** did not improve the product yield as expected (Table 1, entries 11 *vs.* 3). It was then found that 60 °C was the preferable reaction temperature, giving **2a** in 85% yield (Table 1, entries 14 *vs.* 3, 12 and 13).

Under the optimized conditions, as series of ketoximes **1** was employed to synthesize the corresponding ketones **2**. Catalyzed by $(PhCH_2Se)_2$, the deoximation of acetophenone oxime **1a** gave **2a** in 82% isolated yield (Table 2, entry 1). The reaction of 1-phenylbutan-1-one oxime **1b** led to butyrophenone **2b** in 67% yield (Table 2, entry 2). After introducing electron-donation groups, the reactions of substrates **1c–e** were restrained and resulted in the reduced product yields (Table 2, entries 3–5). The deoximation reactions of electron-deficient substrates **1f–i** occurred smoothly to give **2f–i** in 70–76% yields (Table 2, entries 6–9). The protocol was also favorable for the deoximations of diarylmethanone oximes **1j–n** (Table 2, entries 10–14) and electron-deficient substrates afforded higher product yields than did the electron-enriched ones (Table 2, entries 13 and 14 *vs.* 11 and 12). The bulky phenyl-fused substrate 3,4-dihydronaphthalen-1(2*H*)-oxime **1o** could also led to a moderate product yield

Table 1. Optimization of the reaction conditions for the deoximation of **1a**.[a]

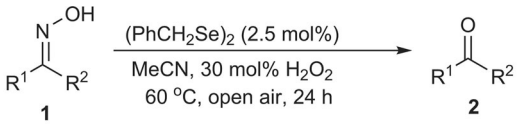


1a **2a**

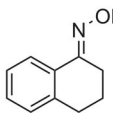
Entry	Solvent	H_2O_2/**1a**[b]	T [°C]	**2a** [%][c]
1	EtOH	30%	80	37
2	H_2O	30%	80	35
3	MeCN	30%	80	68
4	1,4-dioxane	30%	80	65
5	cyclohexane	30%	80	20
6	toluene	30%	80	16
7	PE[d]	30%	80	18
8	MeCN	15%	80	26
9	MeCN	19%	80	37
10	MeCN	26%	80	59
11	MeCN	40%	80	67
12	MeCN	30%	20	32
13	MeCN	30%	40	71
14	MeCN	30%	60	85

[a] *Reaction conditions:* 1 mmol of **1a**, 0.025 mmol of $(PhCH_2Se)_2$, 30% H_2O_2 (weight concentration) and 2 mL of solvent were employed.
[b] Molar ratio of H_2O_2 *vs.* **1a**.
[c] GC yields based on **1a**.
[d] Petroleum ether (bp 90–120 °C).

Table 2. Deoximation of ketoximes.[a]



1 **2**

Entry	Substrate **1**: R^1, R^2	**2**: Yield [%][b]
1	**1a**: Ph, Me	**2a**: 82
2	**1b**: Ph, C_3H_7	**2b**: 67
3	**1c**: 4-MeC_6H_4, Me	**2c**: 57
4	**1d**: 3-MeC_6H_4, Me	**2d**: 58
5	**1e**: 4-$MeOC_6H_4$, Me	**2e**: 50
6	**1f**: 4-ClC_6H_4, Me	**2f**: 76
7	**1g**: 3-ClC_6H_4, Me	**2g**: 75
8	**1h**: 2-ClC_6H_4, Me	**2h**: 70
9	**1i**: 4-$NO_2C_6H_4$, Me	**2i**: 75
10	**1j**: Ph, Ph	**2j**: 81
11	**1k**: 4-MeC_6H_4, 4-MeC_6H_4	**2k**: 63
12	**1l**: 4-$MeOC_6H_4$, 4-$MeOC_6H_4$	**2l**: 68
13	**1m**: 4-ClC_6H_4, 4-ClC_6H_4	**2m**: 80
14	**1n**: 4-FC_6H_4, 4-FC_6H_4	**2n**: 72
15	**1o**: 	**2o**: 55

[a] Reactions were performed under the optimized conditions described in Table 1, entry 13.
[b] Isolated yields based on substrate **1**.

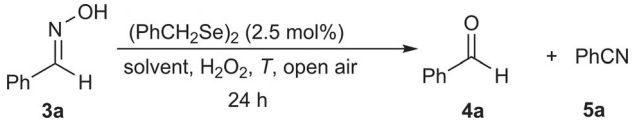
in the organoselenium-catalyzed deoximation (Table 2, entry 15).

We then tried to employ the organoselenium-catalyzed deoximation reaction on aldoximes and benzaldehyde oxime **3a** was selected as the model substrate for optimization oft he conditions. Considering that the product benzaldehyde **4a** might be oxidized into benzoic acid under oxidative conditions, the reaction was initially performed with less H_2O_2 and at a lower temperature. Heating **3a** with 22.5 mol% of H_2O_2 in the presence of $(PhCH_2Se)_2$ catalyst in MeCN at 40 °C afforded the product **4a** and by-product benzonitrile **5a** in 68% total yield, with a **4a**/**5a** ratio of 38:62 (Table 3, entry 1). To reduce the dehydration by-product, we performed the reaction in water, but this led to the generation of **5a** as the overwhelming major product (Table 3, entry 2). Reaction in 1,4-dioxane resulted in poor product yield (Table 3, entry 3). Interestingly, petroleum ether was found to be favorable solvent for the reaction, giving an excellent yield of product, in which the **4a**/**5a** ratio was 87:13 (Table 3, entry 4). Reaction performed at 60 °C led to increased product yield and **4a** selectivity (Table 3, entry 5). But further elevated reaction temperature obviously promoted the dehydration as well as other side reactions (Table 3, entries 6 and 7 *vs.* 5). Both product yield and **4a** selectivity decreased with reduced H_2O_2 dosages (Table 3, entries 8–10 *vs.* 5, 11). Using 45 mol% of H_2O_2 led to the excellent product yield of 99% with **4a** selectivity at 96% (Table 3, entry 12). The reaction was hardly improved with increased H_2O_2 dosages (Table 3, entry 13).

A series of aldoximes **3** was then treated with H_2O_2 in the presence of $(PhCH_2Se)_2$ catalyst. Benzaldehyde oxime **3a** led to the deoximation product benzaldehyde **4a** in excellent yield (Table 4, entry 1). Substrates bearing electron-donation groups, **3b**, **3c** resulted in decreased product yields (Table 4, entries 2 and 3). The electron-enriched substrates **3d**, **3e** even afforded nitriles as the major products (Table 4, entries 4 and 5). The reactions of electron-deficient substrates **3f–i** gave aldehydes **4f–i** in moderate yields, while the dehydration by-products were not observed (Table 4, entries 6–9). Bulky substrates **3j**, **3k** were also tested and the 2-substituted substrate **3k** was obviously preferable for the deoximation reaction (Table 4, entries 11 *vs.* 10). The protocol could be applied to heterocycle-containing substrates and the deoximation of thiophene-2-carbaldehyde oxime **3l** occurred to produce **4l** (Table 4, entry 12).

It is notable that this organoselenium-catalyzed oxidative deoximation is very practical and the catalyst is sufficiently robust to be recycled and reused. A scaled-up reaction with 50 mmol of **1a** was tested, giving 4.5 g of **2a** (in 75% yield) after distillation under reduced pressure. The residue, which contained

Table 3. Optimization of the reaction conditions for the deoximation of **3a**.[a]

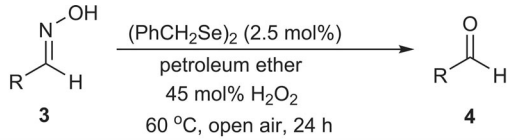


3a 4a 5a

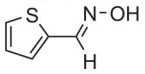
Entry	Solvent	H_2O_2/**3a**[b]	T [°C]	Yield [%]	(**4a**/**5a**)[c]
1	MeCN	22.5%	40	68	(38:62)
2	H_2O	22.5%	40	75	(5:95)
3	1,4-dioxane	22.5%	40	41	(39:61)
4	PE[d]	22.5%	40	91	(87:13)
5	PE[d]	22.5%	60	95	(94:6)
6	PE[d]	22.5%	80	93	(85:15)
7	PE[d]	22.5%	100	65	(37:63)
8	PE[d]	7.5%	60	72	(48:52)
9	PE[d]	15%	60	75	(70:30)
10	PE[d]	20%	60	89	(87:13)
11	PE[d]	30%	60	96	(95:5)
12	PE[d]	45%	60	99	(96:4)
13	PE[d]	60%	60	99	(97:3)

[a] *Reaction conditions:* 1 mmol of **3a**, 0.025 mmol of $(PhCH_2Se)_2$, 30% H_2O_2 (weight concentration) and 2 mL of solvent were employed.
[b] Molar ratio of H_2O_2 *vs.* **3a**.
[c] Total GC yields of **4a** and **5a** based on **3a**; values in parentheses are the molar ratios of **4a**/**5a** in the product.
[d] Petroleum ether (bp 90–120 °C).

Table 4. Deoximation of aldoximes.[a]



3 4

Entry	Substrate **3**: R	**4**: Yield [%][b]
1	**3a**: Ph	**4a**: 88
2[c]	**3b**: 3-MeC_6H_4	**4b**: 60
3[c]	**3c**: 4-*t*-BuC_6H_4	**4c**: 62 (85:15)
4[d]	**3d**, 2,4,6-$Me_3C_6H_2$	**4d**: 81 (7:93)
5	**3e**: 4-$MeOC_6H_4$	**4e**: 61 (39:61)
6[c]	**3f**: 4-ClC_6H_4	**4f**: 67
7[c]	**3g**: 3-ClC_6H_4	**4g**: 56
8[c]	**3h**: 2-ClC_6H_4	**4h**: 52
9[c]	**3i**: 4-$NO_2C_6H_4$	**4i**: 50
10[c]	**3j**: 1-$C_{10}H_7$	**4j**: 51 (60:40)
11[d]	**3k**: 2-$C_{10}H_7$	**4k**: 58 (86:14)
12[d]	**3l**: 	**4l**: 56 (67:33)

[a] Unless specially annotated, the reactions were performed under the optimized conditions described in Table 3, entry 12.
[b] Isolated yields based on substrate **3**; values in the parentheses are the molar ratios of aldehyde/nitrile in the product.
[c] 60% of H_2O_2/**3** was employed.
[d] Reaction performed at 80 °C.

the organoselenium catalytic species, could be reused directly in the next reaction run, revealing the possibility for catalyst recycle and reuse in large-scale production with industrial purposes in near future (Figure 1).

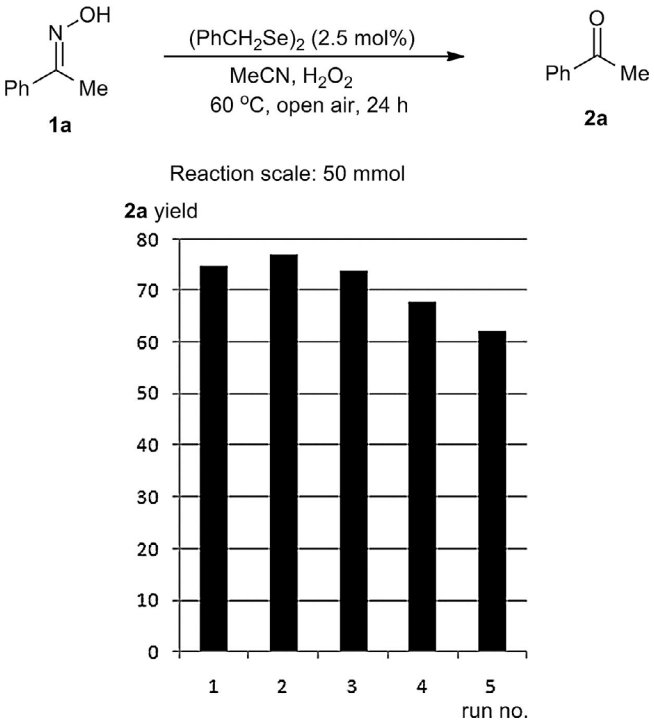
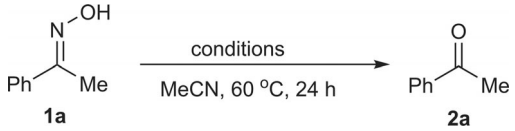


Figure 1. Catalyst recycle and reuse for scaled-up reaction.

Control experiments were performed for a mechanism study. Reactions with a series of different diselenides as catalyst were initially tested to examine their substituent effects (Table 5, entries 1–5): The reaction using $(PhSe)_2$ as the catalyst with a similar electronic effect to $(PhCH_2Se)_2$ gave rise to a decreased product yield (Table 5, entry 1 *vs.* Table 2, entry 1), probably due to its larger steric hindrance that impeded the catalytic Se sites approaching to the substrates. The same reaction could be improved with catalytic $Yb(OTf)_3$, a Lewis acid that could activate oxime substrates (Table 5, entries 2 *vs.* 1). Reactions using an electron-deficient diselenide $(4\text{-}FC_6H_4Se)_2$ or $[3,5\text{-}(CF_3)_2C_6H_4Se]_2$ as catalyst gave further depressed product yields (Table 5, entries 3 and 4), although their steric hindrances were similar to that of $(PhSe)_2$. The typical bulky catalyst $(1\text{-}C_{10}H_7Se)_2$ was unfavorable for the reaction and led to an even lower product yield than $(PhSe)_2$ (Table 5, entries 5 *vs.* 1). The above experimental results indicated that a nucleophilic attack of organoselenium catalytic species to the positive carbon center of the substrate might occur first in the mechanism course, like that in Baeyer–Villiger reactions.[7a–c] The catalytic selenium species of the reaction were also investigated through control experiments (Table 5, entries 6–9): Reactions under N_2 protection led to **2a** in very poor yield (Table 5, entry 6), but with sufficient chemical oxidant such as 100 mol% of PhSe(O)OH, the reaction without air or H_2O_2 also produced **2a** in moderate yield (Table 5, entry 7), showing that air was an essential oxidant for the reaction. Although performed in open air, the reaction could not happen without H_2O_2 when using the diselenide catalyst (Table 5, entry 8). In contrast, use of similar reaction conditions led to **2a** in 18% yield with the commercially available organoseleninic acid PhSe(O)OH as catalyst (Table 5, entry 9). These phenomena suggested that diselenide was inactive for the reaction, but in the presence of H_2O_2, it could be oxidized to the organoseleninic acid, which was the real catalytic species. Because organoseleninic acid was easily reduced to diselenides and lost activity,[6b,d] a certain amount of H_2O_2 was necessary for the reaction to maintain sufficiently oxidative conditions, although the reaction could employ air as oxidant.

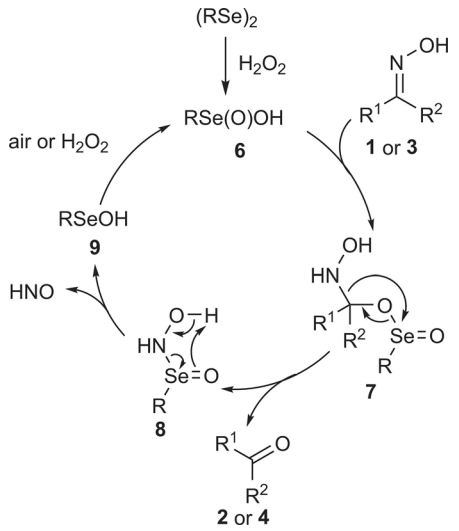
Table 5. Control experiments.[a]



Entry	Conditions[b]	**2a** [%][c]
1	$(PhSe)_2$ (2.5%), H_2O_2 (30%), open air	66
2	$(PhSe)_2$ (2.5%), $Yb(OTf)_3$ (0.5%), H_2O_2 (30%), open air	73
3	$(4\text{-}FC_6H_4Se)_2$ (2.5%), H_2O_2 (30%), open air	60
4	$[3,5\text{-}(CF_3)_2C_6H_4Se]_2$ (2.5%), H_2O_2 (30%), open air	52
5	$(1\text{-}C_{10}H_7Se)$ (2.5%), H_2O_2 (30%), open air	36
6	$(PhCH_2Se)_2$ (2.5%), H_2O_2 (30%), N_2	15
7	PhSe(O)OH (100%), without H_2O_2, N_2	64
8	$(PhCH_2Se)_2$ (2.5%), without H_2O_2, open air	no reaction
9	PhSe(O)OH (5%), without H_2O_2, open air	18

[a] *Reaction conditions:* 1 mmol of **1a** and 2 mL of MeCN were employed.
[b] Molar ratio *vs.* **1a** in the parentheses.
[c] Isolated yields based on **1a**.

On the basis of the experimental results as well as references, a plausible mechanism was supposed (Scheme 2). The *pre*-catalyst diselenides was initially oxidized to the organoseleninic acid **6** by H_2O_2, which then reacted with the substrates **1** or **3** through a nucleophilic addition and gave the intermediate **7**.[7a–c] Because of the nucleophilic addition step, the electron-enriched catalysts with low steric hindrance were preferable for the reaction (Table 2, entry 1 *vs.* Table 5, entries 5 and 7–9). At this step, electron-defi-



Scheme 2. Possible mechanisms.

cient substrates possess strongly positive carbon centers, and are favorable for the reaction, in accordance with the experimental results in Table 2, entries 6–9 *vs.* 3–5, entries 13, 14 *vs.* 11, 12 and Table 4, entries 6–9 *vs.* 4, 5. Since the nitrogen of the oxime could coordinated with a Lewis acid metal which enhanced the electron positivity of its adjacent carbon, the addition of $Yb(OTf)_3$ improved the reaction (Table 5, entries 6 *vs.* 5). The nitrogen transfer in **7** released the deoximation products **2** or **4** and led to the intermediate **8**, which decomposed to HNO and RSeOH **9** through a selenoxide *syn*-elimination process.[20] Since RSeOH was an active species, it could be oxidized by air or H_2O_2 to regenerate the organoseleninic acid **6** and restart the catalytic cycle.

Compared with the substrate amount, the employed amount of H_2O_2 was insufficient to complete the whole reaction. Therefore, oxidation of RSeOH **9** by air to regenerate RSe(O)OH **6** should be very important step in the mechanism circle. ^{77}Se NMR analysis was used to support the hypothesis [Eq. (1)]. We

$$(PhSe)_2 + H_2O_2 \xrightarrow{N_2} PhSeOH \xrightarrow{air} PhSe(O)OH \quad (1)$$

^{77}Se NMR chemical shift: 1056 ppm 1181 ppm

employed $(PhSe)_2$ for a test because the data of related organoselenium species had been reported and could be employed for comparison with our results. On treating $(PhSe)_2$ with an equivalent of H_2O_2 under N_2 protection, the signal of PhSeOH at 1056 ppm could be observed (Figure 2a); In comparison, the same reaction exposed to air only led to PhSe(O)OH, as indicated by the signal at 1181 ppm (Figure 2b).[6c,21] The results clearly demonstrated the process of oxidation of PhSeOH by air to regenerate PhSe(O)OH in the catalytic circle. Thus, although this mechanism remains to be fully clarified and alternative processes may also exist, Scheme 2 should be the most likely mechanism based on the above tests and the related literature reports.

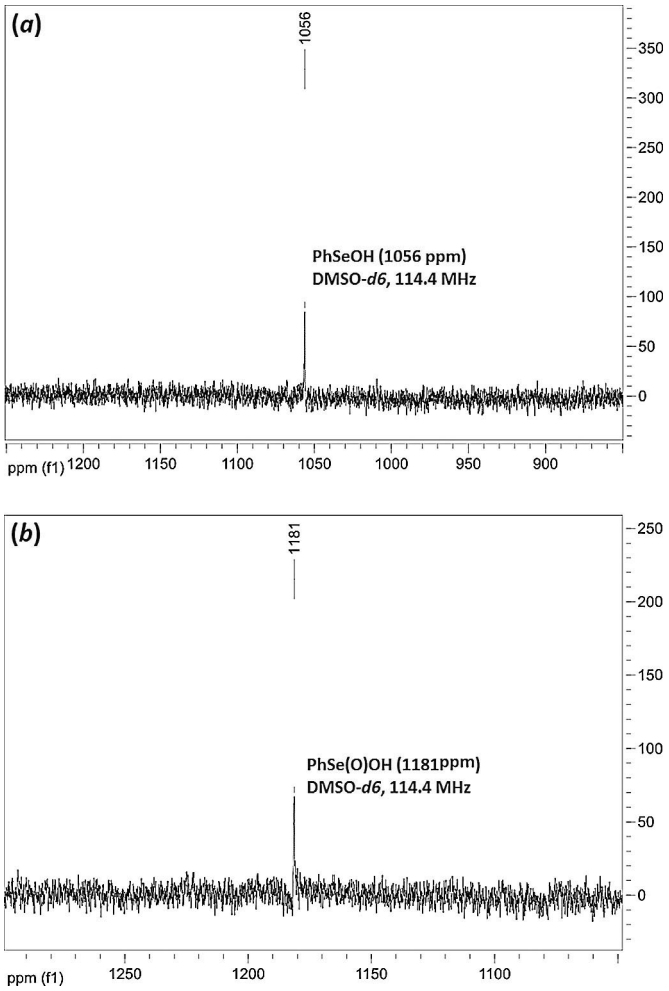


Figure 2. ^{77}Se NMR spectra: (a) reaction of $(PhSe)_2$ with an equivalent of H_2O_2 under N_2 protection; (b) exposure of PhSeOH under air.

Conclusions

In conclusion, we have reported an organoselenium-catalyzed deoximation. The reaction employs H_2O_2/air as green oxidant and generates no wastes. The findings provide new tools for organic synthesis and largely expand the application scope of organoselenium catalysis, which is a unique subject with industrial application potential. More investigations in the field are ongoing in our laboratory.

Experimental Section

General Methods

Reagents were purchased from commercial sources with purities of more than 98% and were directly used as received. Solvents were of analyticallx pure grade (AR) and directly used without any special treatment. Melting points were measured by a WRS-2A digital instrument. IR spectra were measured on a Bruker Tensor 27 infrared spectrometer. ^{1}H NMR spectra were recorded on Bruker Avance 600/400 instruments (600 or 400 MHz) using $CDCl_3$ as the solvent and Me_4Si as the internal standard. Chemical shifts for ^{1}H NMR are referred to internal Me_4Si (0 ppm) and J values are shown in Hz.[77] Se NMR spectra were recorded on a Bruker Avance 600 instrument (114.4 MHz).

Typical Procedure for the Deoximation of Ketoxime

In a reaction tube, 0.025 mmol of $(PhCH_2Se)_2$ (2.5 mol%), 0.3 mmol of H_2O_2 (30 w/w%) and 2 mL of MeCN were stirred at 60 °C for 1 h. 1 mmol of ketoxime **1** was then added. The mixture was stirred at 60 °C in the open air for 24 h and cooled to room temperature. The solvent was evaporated under vacuum and the residue was separated by preparative TLC (eluent: petroleum ether:EtOAc = 15:1) to afford the corresponding products **2**.

Typical Procedure for the Deoximation of Aldoxime 3a

In a reaction tube, 0.025 mmol of $(PhCH_2Se)_2$ (2.5 mol%), 0.45 mmol of H_2O_2 (30 w/w%) and 2 mL of petroleum ether (bp 90–120 °C) were stirred at 60 °C for 1 h. 1 mmol of aldoxime **3** was then added. The mixture was stirred at 60 °C in open air for 24 h and cooled to room temperature. The solvent was evaporated under vacuum and the residue was separated by preparative TLC (eluent: petroleum ether: EtOAc = 15:1) to afford the corresponding product **4**.

Detailed Procedure for Catalyst Recycle and Reuse in a Scaled-Up Reaction

To a 250-mL round-bottom flask, 0.43 g of $(PhCH_2Se)_2$ (1.25 mmol), 8.5 g of 30% H_2O_2 and 100 mL of MeCN were added. After stirring the mixture at 60 °C for 1 h, 6.75 g of acetophenone oxime **1a** (50 mmol) were added. The mixture was stirred at 60 °C in the open air for 24 h. The solvent was evaporated on a rotary evaporator and the residue was transferred to a 25-mL round-bottom flask and distilled under reduced pressure to give product **2a**; yield: 4.5 g; bp 75–82 °C/10 mm Hg. The residue was collected and reused as the catalyst in the next reaction run.

Detailed Procedure for ^{77}Se NMR Studies

1 mmol of $(PhSe)_2$ and 1 mmol of H_2O_2 were stirred in 1 mL of MeCN under N_2 protection for 0.5 h. The mixture was then dissolved in DMSO-d_6 and sent to ^{77}Se NMR analysis immediately to provide the spectrum in Figure 2a. In comparison, the same reaction performed in air led to the spectrum in Figure 2b.

Characterization of the Products

Acetophenone (2a): Oil; IR (film): ν = 3063, 1685, 1596, 1449, 1360, 1266, 965, 760, 691, 569, 530 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, TMS): δ = 7.96 (d, J = 6.6 Hz, 2H), 7.55(s, 1H), 7.45 (s, 2H), 2.59 (s, 3H); *known compound*.[22]

Butyrophenone (2b): Oil; IR (film): ν = 2962, 2930, 1686, 1597, 1495, 1215, 693 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, TMS): δ = 7.97 (d, J = 7.8 Hz, 2H), 7.56 (t, J = 7.2 Hz, 1H), 7.47 (t, J = 7.8 Hz, 2H), 2.96 (d, J = 7.2 Hz, 2H), 1.80 (m, 2H), 1.02 (t, J = 7.8 Hz, 3H); *known compound*.[22]

1-(*p*-Tolyl)ethan-1-one (2c): Oil; IR (film): ν = 3004, 2825, 1930, 1683, 1608, 1424, 1359, 1267, 1182, 1118, 1021, 955, 816, 672, 585, 461 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, TMS): δ = 7.85 (d, J = 8.4 Hz, 2H), 7.25 (d, J = 7.8 Hz, 2H), 2.56 (s, 3H), 2.39 (s, 3H); *known compound*.[22]

1-(*m*-Tolyl)ethan-1-one (2d): Oil; IR (film): ν = 2924, 1685, 1594, 1431, 1359, 1277, 1192, 1087, 960, 789, 693, 594, 466 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, TMS): δ = 7.77–7.75 (m, 2H), 7.37–7.35 (m, 2H), 2.59 (s, 3H), 2.41 (s, 3H); *known compound*.[22]

1-(4-Methoxyphenyl)ethan-1-one (2e): Solid, mp 36.0–37.6 °C (lit. 36–38 °C); IR (KBr): ν = 3065, 3004, 2959, 2842, 2571, 2054, 1941, 1676, 1598, 1510, 1424, 1359, 1298, 1174, 1116, 1076, 1026, 958, 835, 677, 579, 504, 456 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, TMS): δ = 7.95 (d, J = 8.4 Hz, 2H), 6.94 (d, J = 8.4 Hz, 2H), 3.87 (s, 3H), 2.56 (s, 3H); *known compound*.[22]

1-(4-Chlorophenyl)ethan-1-one (2f): Oil; IR (film): ν = 3090, 3006, 1687, 1569, 1486, 1427, 1396, 1358, 1260, 1094, 1012, 958, 829, 761, 524 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.89 (d, J = 7.8 Hz, 2H), 7.43 (d, J = 7.8 Hz, 2H), 2.58 (s, 3H); *known compound*.[22]

1-(3-Chlorophenyl)ethan-1-one (2g): Oil; IR (film): ν = 3071, 3007, 1690, 1572, 1473, 1423, 1358, 1251, 1166, 1078, 961, 897, 792, 680, 591, 471 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, TMS): δ = 7.93 (t, J = 1.8 Hz, 1H), 7.84 (d, J = 7.8 Hz, 1H), 7.54–7.52 (m, 1H), 7.42 (t, J = 7.8 Hz, 1H), 2.60 (s, 3H); *known compound*.[22]

1-(2-Chlorophenyl)ethan-1-one (2h): Oil; IR (film): ν = 3067, 3006, 2926, 2315, 1698, 1589, 1430, 1358, 1280, 1242, 1096, 1041, 961, 759, 670, 591, 534, 464 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, TMS): δ = 7.56–7.54 (m, 1H), 7.43–7.38 (m, 2H), 7.34–7.31 (m, 1H), 2.65 (s, 3H); *known compound*.[22]

1-(4-Nitrophenyl)ethan-1-one (2i): Solid, mp 74.3–78.9 °C (lit. 75–78 °C); IR (KBr): ν = 3105, 2923, 2858, 2313, 1822, 1689, 1606, 1521, 1437, 1344, 1251, 960, 852, 741, 684, 592, 503 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, TMS): δ = 8.33 (d, J = 9.0 Hz, 2H), 8.14 (d, J = 8.4 Hz, 2H), 2.70 (s, 3H); *known compound*.[22]

Benzophenone (2j): solid, mp 47.3–49.6 °C (lit. 47–49 °C); IR (KBr): ν = 3061, 1659, 1597,1447, 1316, 1277, 1000, 929, 809, 763, 700, 639 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, TMS): δ = 7.79 (d, J = 7.8 Hz, 4H), 7.56 (t, J = 7.2 Hz, 2H), 7.45 (t, J = 7.8 Hz, 4H); *known compound*.[22]

Di-*p*-tolylmethanone (2k): Solid, mp 89.3–94.2 °C (lit. 90–93 °C); IR (KBr): ν = 3042, 2922, 2311, 1646, 1603, 1451, 1351, 1281, 1171, 928, 826, 748, 677, 583, 467 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, TMS): δ = 7.70 (d, J = 8.4 Hz, 4H), 7.27 (d, J = 8.4 Hz, 4H), 2.42 (s, 6H); *known compound*.[22]

Bis(4-methoxyphenyl)methanone (2l): Solid, mp 140.6–147.1 °C (lit. 141–146 °C); IR (KBr): ν = 2919, 2851, 2311,

1638, 1601, 1503, 1460, 1317, 1255, 1161, 1019, 975, 844, 765, 676, 587, 512 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, TMS): δ = 7.80 (d, J = 7.8 Hz, 4H), 6.97 (d, J = 7.2 Hz, 4H), 3.89 (s, 6H); *known compound*.[22]

Bis(4-chlorophenyl)methanone (2m): Solid, mp 143.2–147.8 °C (lit. 144–147 °C); IR (KBr): ν = 3081, 2920, 2311, 1924, 1650, 1583, 1481, 1285, 1153, 1087, 968, 840, 752, 665, 504, 458 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, TMS): δ = 7.74 (d, J = 6.6 Hz, 4H), 7.48 (d, J = 7.2 Hz, 4H); *known compound*.[22]

Bis(4-fluorophenyl)methanone (2n): Solid, mp 100.9–104.6 °C (lit. 102–105 °C); IR (KBr): ν = 3064, 2922, 2313, 1922, 1646, 1591, 1502, 1297, 1230, 1152, 1101, 967, 850, 762, 671, 583, 542, 495 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, TMS): δ = 7.82 (s, 4H), 7.19 (t, J = 7.2 Hz, 4H); *known compound*.[22]

3,4-Dihydronaphthalen-1(2*H*)-one (2o): Oil; IR (film): ν = 3064, 2942, 2876, 1685, 1601, 1451, 1328, 1286, 1226, 1185, 1119, 1025, 963, 900, 764, 645, 555, 487 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, TMS): δ = 8.04 (d, J = 7.8 Hz, 1H), 7.48 (t, J = 7.2 Hz, 1H), 7.32 (t, J = 7.8 Hz, 1H), 7.26 (d, J = 8.4 Hz, 1H), 2.98 (t, J = 6.6 Hz, 2H), 2.67 (t, J = 6.6 Hz, 2H), 2.17–2.12 (m, 2H); *known compound*.[22]

Benzaldehyde (4a): Oil; IR (film): ν = 3066, 2819, 2737, 2697, 1703, 1598, 1455, 1392, 1204, 826, 747, 688, 650, 451 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, TMS): δ = 10.03 (s, 1H), 7.90 (t, J = 7.2 Hz, 2H),7.65 (t, J = 7.8 Hz, 1H), 7.55 (t, J = 7.8 Hz, 2H); *known compound*.[22]

3-Methylbenzaldehyde (4b): Oil; IR (film): ν = 3044, 3026, 2726, 1703, 1603, 1477, 1330, 1215, 996, 781, 688, 589 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, TMS): δ = 9.98 (s, 1H), 7.68 (d, J = 8.4 Hz, 2H), 7.44–7.40 (m, 2H), 2.42 (s, 3H); *known compound*.[22]

4-(*tert*-Butyl)benzaldehyde (4c) and 4-(*tert*-butyl)benzonitrile (5c): Oil; IR (**4c** and **5c**, film): ν = 2966, 1697, 1608, 1413, 1269, 1157, 1075, 854, 785, 674, 544 cm^{-1}; ^1H NMR (**4c**, 600 MHz, $CDCl_3$, TMS): δ = 9.98 (s, 1H), 7.83 (d, J = 8.4 Hz, 2H), 7.56 (d, J = 8.4 Hz, 2H), 1.35 (s, 9H); ^1H NMR (**5c**, 600 MHz, $CDCl_3$, TMS): δ = 7.59 (d, J = 8.4 Hz, 2H), 7.50–7.47 (m, 2H), 1.33 (s, 9H); *known compounds*.[6b,22]

2,4,6-Trimethylbenzaldehyde (4d) and 2,4,6-trimethylbenzonitrile (5d): Oil; IR (**4d** and **5d**, film): ν = 2925, 2863, 2218, 1685, 1610, 1459, 1370, 1215, 1029, 854, 779, 710, 660, 581 cm^{-1}; ^1H NMR (**4d**, 600 MHz, $CDCl_3$, TMS): δ = 10.53(s, 1H), 6.87 (s, 2H), 2.56 (s, 6H), 2.31(s, 3H); ^1H NMR (**5d**, 600 MHz, $CDCl_3$, TMS): δ = 6.91 (s, 2H), 2.45 (s, 6H), 2.31(s, 3H); *known compounds*.[22]

4-Methoxybenzaldehyde (4e) and 4-methoxybenzonitrile (5e): Oil; IR (**4e** and **5e**, film): ν = 3022, 2977, 2938, 2842, 1689, 1603, 1509, 1459,1306, 1260, 1170, 1025,835, 683, 600, 550 cm^{-1}; ^1H NMR (**4e**, 600 MHz, $CDCl_3$, TMS): δ = 9.88 (s, 1H), 7.84 (d, J = 8.4 Hz, 2H), 7.01 (d, J = 8.4 Hz, 2H), 3.89(s, 3H); ^1H NMR (**5e**, 600 MHz, $CDCl_3$, TMS): δ = 7.59 (d, J = 9.0 Hz, 2H), 6.96(d, J = 8.4 Hz, 2H), 3.86 (s, 3H); *known compounds*.[6b,22]

4-Chlorobenzaldehyde (4f): Solid, mp 45.8–47.3 °C (lit. 46 °C); IR (KBr): ν = 3060, 2926, 2843, 2735, 1704, 1589, 1485, 1374, 1291, 1207, 1165, 1090, 1011, 826, 694, 541, 482 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, TMS): δ = 9.99 (s, 1H), 7.84 (d, J = 8.4 Hz, 2H), 7.52 (d, J = 8.4 Hz, 2H); *known compound*.[22]

3-Chlorobenzaldehyde (4g): Oil; IR (film): ν = 3069, 2836, 2729, 2234, 1702, 1576, 1473, 1434, 1375, 1278, 1197, 1027, 889, 789, 683, 566 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, TMS): δ = 9.98 (s, 1H), 7.85 (d, J = 1.2 Hz, 1H), 7.77 (d, J = 7.8 Hz, 1H), 7.61 (t, J = 7.2 Hz, 1H), 7.50–7.48 (m, 1H); *known compound*.[22]

2-Chlorobenzaldehyde (4h): Oil; IR (film): ν = 3069, 2866, 2753, 1656, 1647, 1550, 1446, 1288, 1203, 1127, 1046, 824, 758, 709, 634, 564 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, TMS): δ = 10.47 (s, 1H), 7.92–7.90 (m, 1H), 7.54–7.51 (m, 1H), 7.45 (d, J = 7.8 Hz, 1H), 7.39 (t, J = 7.2 Hz,1H); *known compound*.[22]

4-Nitrobenzaldehyde (4i): Solid; mp 102.0–107.3 °C (lit. 103–106 °C); IR (KBr): ν = 3061, 2646, 1706, 1607, 1533, 1348, 1206, 1008, 814, 735, 673, 568 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$, TMS): δ = 10.17 (s, 1H), 8.41 (d, J = 8.4 Hz, 2H), 8.10–8.08 (m, 2H); *known compound*.[22]

1-Naphthaldehyde (4j) and 1-naphthonitrile (5j): Oil; IR (**4j** and **5j**, film): ν = 3097, 2923, 2727, 2222, 1688,1635, 1578, 1509, 1344, 1216, 882, 772, 708, 690, 563, 452 cm^{-1}; ^1H NMR (**4j**, 600 MHz, $CDCl_3$, TMS): δ = 10.37 (s, 1H), 9.25 (d, J = 8.4 Hz, 1H), 8.07 (d, J = 8.4 Hz, 1H), 7.96–7.95 (m, 1H), 7.90–7.87 (m, 1H), 7.68–7.65 (m, 1H), 7.61–7.56 (m, 2H); ^1H NMR (**5j**, 600 MHz, $CDCl_3$, TMS): δ = 8.22 (d, J = 8.4 Hz, 1H), 8.04 (d, J = 8.4 Hz, 1H), 7.90–7.87 (m, 2H), 7.68–7.65 (m, 1H), 7.61–7.56 (m, 1H), 7.49–7.47 (m, 1H); *known compounds*.[22]

2-Naphthaldehyde (4k) and 2-naphthonitrile (5k): Oil; IR (**4k** and **5k**, film): ν = 3059, 2823, 2719, 2226, 1695, 1628, 1463, 1350, 1264, 1214, 1165, 1119, 963, 904, 864, 819, 750, 628, 475 cm^{-1}; ^1H NMR (**4k**, 400 MHz, $CDCl_3$, TMS): δ = 10.14(s, 1H), 8.31 (s, 1H), 7.99–7.85 (m, 4H), 7.65–7.55 (m, 2H); ^1H NMR (**5k**, 400 MHz, $CDCl_3$, TMS): δ = 8.20 (s, 1H), 7.99–7.85(m, 3H), 7.65–7.55(m, 3H); *known compound*.[22]

Thiophene-2-carbaldehyde (4l) and thiophene-2-carbonitrile (5l): Oil; IR (**4l** and **5l**, film): ν = 2924, 2863, 2209, 1641, 1510, 1342, 1219, 985, 726, 668, 592 cm^{-1}; ^1H NMR (**4l**, 400 MHz, $CDCl_3$, TMS): δ = 9.96 (d, J = 1.2 Hz, 1H), 7.80–7.77 (m, 2H), 7.24–7.22 (m, 1H); ^1H NMR (**5l**, 400 MHz, $CDCl_3$, TMS): δ7.65–7.61 (m, 2H), 7.15–7.13 (m,1H); *known compound*.[22]

Acknowledgements

This work was financially supported by NNSFC (21202141), Priority Academic Program Development (PAPD) of Jiangsu Higher Education Institutions, the Open Project Program of Jiangsu Key Laboratory of Zoonosis (R1509), the Innovation Foundation of Yangzhou University (2015CXJ009) and the High Level Talent Support Project of Yangzhou University. We thank the testing centre of Yangzhou University for assistance.

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