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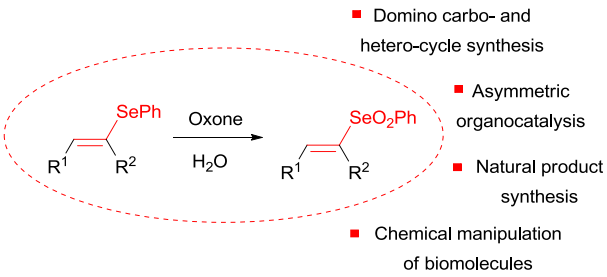
Oxone mediated oxidation of vinyl selenides in water

Martina Palomba,[a] Francesco Trappetti,[a] Luana Bagnoli,[a] Claudio Santi[a] and Francesca Marini*[a]

Abstract: A simple and practical procedure for the oxidation of vinyl selenides into the corresponding selenones using Oxone in water at 60 °C has been reported. Structurally diverse phenyl vinyl selenones were oxidized under heterogeneous conditions without organic co-solvents or additional catalysts.

Introduction

Selenones are organic compounds containing a hexavalent, tetra-coordinated selenium species.[1] These compounds are accessible by direct oxidation of selenides. The initially formed tetravalent selenoxides are converted into the selenones by using an excess of oxidant. Despite the formal similarities, redox properties of selenides are quite different in respect to the analogous sulfides.[1,2] The Se=O bonds have more dipolar character than S=O bonds and, even though the first oxidation of selenides to selenoxides is fast, the decreased electron density on the selenium atom makes selenoxides less susceptible to further oxygen transfer. As a consequence, only few oxidizing agents are strong enough to convert selenides into the corresponding selenones. Organic peroxyacids,[3] such as *m*-chloroperbenzoic acid (*m*-CPBA) or trifluoroperacetic acid in MeOH or dichloromethane, and magnesium bis(monoperoxy phtalate) (MMPP)[4] in alcohols or THF have been successfully employed. Hydrogen peroxide with catalytic amounts of benzenseleninic acid[5] and inorganic oxidants such as potassium permanganate [3] or Oxone[6] are also suitable for the oxidation. Two procedures employing the $HOF{\cdot}CH_3CN$ complex prepared by bubbling dilute F_2 into aqueous acetonitrile[7] and the HMPA molibdenum complex $[MoO(O)_2(H_2O)(HMPA)]$[8] have been developed recently. All these methods show drawbacks in terms of safety, cost, waste, or use of heavy metals or halogenated solvents. Furthermore, none of these protocols is general, being the efficiency of the methods strictly dependent from the structural features of the starting selenide. For example, some selenones are not accessible due to the high instability of the selenoxide intermediates that spontaneously decompose by elimination.[9] Moreover, selenones are sensitive to nucleophiles or bases, due to the excellent leaving ability of the selenonyl group in nucleophilic substitutions. For this reason in many cases they undergo further transformations as soon as they are formed.[6,10] Recently, under-investigated phenyl vinyl selenones (Scheme 1) have been rediscovered as versatile building blocks for the domino synthesis of carbo- and hetero-cycles,[11] and for the synthetically challenging, enantioselective construction of quaternary stereocenters via organocatalysis.[12] They found also useful applications in natural product synthesis[12g] and in nucleoside[13] and carbohydrate[14] chemistry. Many of the developed transformations are Michael-initiated domino reactions in which the selenonyl moiety consecutively plays different roles as strong electron-withdrawing group in the Michael step, as efficient leaving group in the next nucleophilic substitution and, in one case [11f] also as an oxidant after the displacement. Despite the increasing attention, few methods are available for their preparation. In fact, to date, the only effective access to vinyl selenones relies on the oxidation of the corresponding selenides[15] with organic peroxyacids, being the *m*-CPBA the oxidant of choice.[1d-e,11-14] In light of these facts and as part of our interest in the chemistry of vinyl selenones, we decided to explore alternative and greener methods for their preparation. We were inspired by the wide number of oxidative protocols recently employed in the synthesis of sulfones, that involve hydrogen peroxide in the presence of catalysts[16] or Oxone,[17] (triple salt: $2KHSO_5{\cdot}KHSO_4{\cdot}K_2SO_4$). These oxidants, due to the non-toxic nature of the by-products, the easy handling and the low cost are greatly employed for eco-friendly transformations.[18] Preliminary experiments showed that Oxone in water without any organic co-solvent, additional catalyst or promoter generate vinyl selenones in good yields. Structurally diverse phenyl vinyl selenones have been prepared and characterized by nuclear magnetic resonance of ^{77}Se. Although this spectroscopy is considered a very powerful tool for the characterization of selenium–containing compounds,[19] to date, only sporadic data are available for selenones.[3,10d-f,12c]



Scheme 1. Synthesis of phenyl vinyl selenones and synthetic applications.

Results and Discussion

Table 1 shows the results of the experiments carried out with different oxidizing systems on the phenyl (*E*)-2-phenylvinyl selenide **1a** as the model substrate. The results of the typical reaction with *m*-CPBA is reported for comparison.

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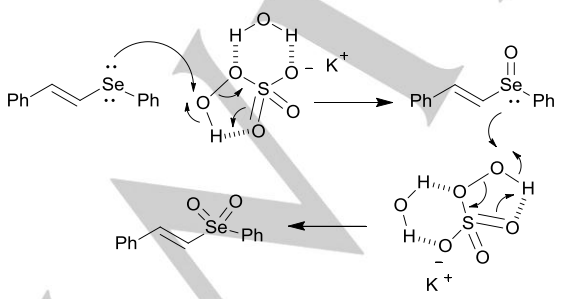
All the experiments carried out with H_2O_2 in combination with different inorganic catalysts failed to generate the selenones even after keeping the reactions for 24 hours. In fact, only the formation of the selenoxide **2a** was observed with variable conversions. The experiment with Oxone in MeOH under reaction conditions previously reported in the literature[6] for the oxidation of aryl alkyl selenides (Table 1, entry 5) was also unsuccessful. In fact, only the 2-methoxy-2-phenylethyl phenyl selenone **4a** was isolated from the crude reaction mixture in 50% yield. The formation of this product can be easily explained: Oxone converted the vinyl selenide **1a** into the corresponding selenone **3a**, but this compound was sensitive to the basic conditions (pH 11) employed and underwent an intermolecular oxa-Michael reaction in the presence of methanol. The experiment carried out in the absence of the basic buffer afforded the vinyl selenoxide **2a** as the major product (Table 1, entry 6). A greater amount of selenone was obtained when the reactions were carried out in water without any co-solvent. The almost exclusive formation of **3a** (83% isolated yield) was observed with 2.2 molar equivalents of Oxone under "on-water" conditions after 14 h heating at 60 °C (Table 1, entry 12) and vigorous stirring. Interestingly, the reaction proceeded chemo-selectively without any epoxidation of the C=C double bonds[18b,c,f,20] Moreover the method is mild enough to avoid the addition of water on the electrophilic carbon-carbon double bond. A control over the degree of oxidation was also possible varying the oxidant loading and lowering the temperature. In fact, the selective access to the vinyl selenoxide **2a** was performed working with 0.6 equivalents of Oxone in water at room temperature (Table 1, entry 11).

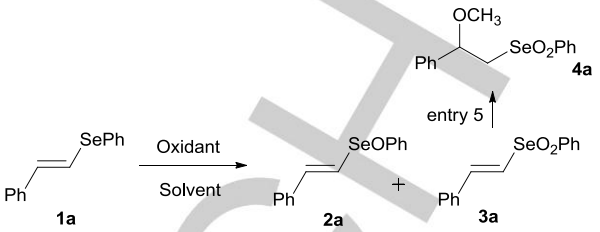
The formation of the selenone **3a** was also observed in several protic and aprotic polar organic solvents, but ^{1}H NMR of the crude reaction mixtures showed consistent amounts of the selenoxide. Only the oxidation in acetonitrile gave a selenoxide: selenone ratio comparable with those obtained in water, however the rate of the oxidation was slower. In fact, an incomplete conversion of the vinyl selenide **1a** was observed, after identical reaction times (Table 1, entry 14).

In order to explain the role of water, a reaction mechanism has been proposed. Water not only readily dissolves the Oxone, but it also modulates the activity of this oxidant.[17a] In fact, the formation of inter- and intra-molecular hydrogen bonds can facilitate the oxygen transfer (Scheme 2).



Scheme 2. Plausible mechanism for Oxone mediated oxidations of vinyl selenides in aqueous medium

Table 1. Comparative study with various oxidative systems and optimization of reaction conditions

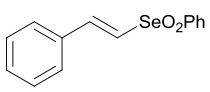
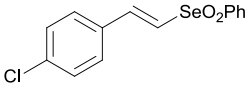
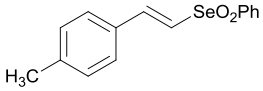
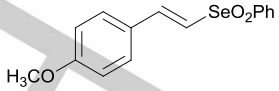
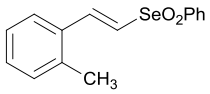
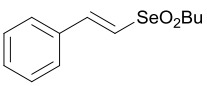
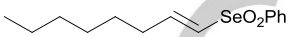
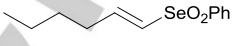
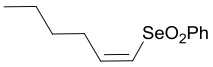
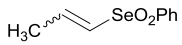
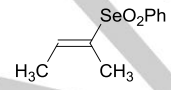
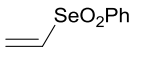
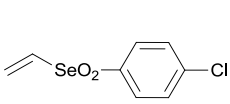
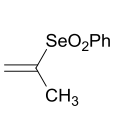
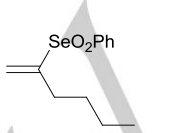
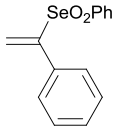
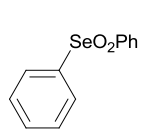


entry	oxidant (equiv.)	conditions	t (h)	conv. (%)[a,b]	2a:3a [a]
1	H_2O_2 (3)	neat, 75 °C	5	88	90:10
2[16a]	H_2O_2 (8) $MnSO_4$ (0.02)	MeCN/0.2 M $NaHCO_3$, rt	24	100	100:0
3[16b]	H_2O_2 (3) $Na_2B_4O_7$ (0.1)	MeOH/0.1 M NaOH, rt	7	low	100:0
4[16d]	H_2O_2 (8) H_3BO_3 (0.5)	neat, rt	2	low	100:0
5[6]	Oxone (2.2)	MeOH/NaOH, rt	1	100	-
6	Oxone (2.2)	MeOH/H_2O, rt	8	100	72:28
7	Oxone (2.2)	H_2O, rt	8	100	60:40
8	Oxone (2.2)	H_2O, 60 °C	8	100 (80)	10:90
9	Oxone (2.2)	H_2O, 60 °C	4	100 (77)	30:70
10	Oxone (1.5)	H_2O, 60 °C	24	100 (70)	13:87
11	Oxone (0.6)	H_2O, rt	4	100 (72)	100:0
12	Oxone (2.2)	H_2O, 60 °C	14	100 (83)	5:95
13	Oxone (2.2)	EtOH, 60 °C	8	100	29:71
14	Oxone (2.2)	MeCN, 60 °C	8	92	8:92
15	Oxone (2.2)	DMF, 60 °C	8	100	81:18
16[1f]	*m*-CPBA(2.6) K_2HPO_4(4.4)	MeOH, rt	12	100	7:93

[a] Conversions and **2a**:**3a** ratios are calculated by ^{1}H NMR on crude mixtures.
[b] Isolated yields are reported in parentheses.

With the best conditions in hand, oxidation reactions of variously substituted vinyl selenides to selenones were examined. Compounds **1a-q** were treated with 2.2 equivalents of Oxone ($2KHSO_5{\cdot}KHSO_4{\cdot}K_2SO_4$) in H_2O at 60° C without additional catalysts, co-solvents or buffers. Reaction products, times and isolated yields are shown in table 2. In parentheses the yields of oxidation reactions carried out with an excess of *m*-CPBA (2.6 to 4 equivalents) are reported for comparison. Several β- aryl or alkyl substituted phenyl vinyl selenides were oxidized into the corresponding selenones in comparable or even better yields than those obtained with *m*-CPBA. Work-up and purification procedures were facilitated in respect to reactions with *m*-CPBA.

Table 2. Synthesis of the selenones **3**[a]

			
3a,14h, 83%,(90%)[ref11q]	**3b**, 15h, 68%, (60%)[ref11a]	**3c**, 3h, 64%, 77%,[b] (67%)[11a]	**3d**, 8h, 53%, (63%)[ref11a]
			
3e, 8h, 86%, (40%)	**3f**, -(50)[b]	**3g**, 14h, 89%, (78%)	**3h**, 15h, 67%, (76%)[ref11o]
			
3i, 14h, 45%	**3j**,[c] 5h, 64%, (45%)[ref11f]	**3k**,-	**3l**,[d] 4h, 70%, 84%,[b] (60%)[ref11b]
				
3m, 4h, 83%,	**3n**,[d] 8h, 50%, (60%)	**3o**, 20h, 60%, (44%)	**3p**, -	**3q**, 3h, 80%

[a] Yields of the oxidations carried out with *m*-CPBA are reported in parenthesis for a comparison. [b] Yield calculated on the crude mixture. [c] The reaction was carried out on a 1:1 mixture of *E*:*Z* selenides (by ^{1}H NMR). [d] Reactions with 0.6 equiv.of Oxone gave selenoxides in poor yields.

The reported yields refer to compounds purified by filtration through a short pad of silica gel, however crude products obtained by simple extraction of the reaction mixtures with EtOAc or by filtration of the suspended solid, as for **3c,** were almost pure by ^{1}H NMR analysis. The oxidations of sterically hindered *Z* vinyl selenides, such as **3i** and **3j** were also successful, albeit in moderate yields. Unfortunately, the butyl (*E*)-2-phenylvinyl selenone **3f** and the trisubstituted vinyl selenone **3k** could not be formed. Probably, the oxidation of **1f** failed in consideration of the high instability of the alkyl selenones. In fact, also the crude **3f** initially obtained when the reaction was performed with *m*-CPBA in dichloromethane, gave rapid decomposition during the chromatography and storage. With the exception of **3p**, the formation of α-substituted vinyl selenones occurred in good yields. Interestingly, the oxidation of diphenyl selenide gave **3q** in 80% yield.

The scale up of the reaction was attempted at a 1g scale on **1l**. The desired selenone **3l** was isolated in 68% yield.

Finally, considering the few information about ^{77}Se NMR resonance of selenones, a set of data have been collected for their characterization. NMR spectra gave chemical shift values around 960 ppm, shifted at lower magnetic fields respect to those reported for the corresponding selenoxides (resonance range: 812-941 ppm).[19] The deshielding is related with the decreased electron density around the selenium atom. Values slightly lower than those observed for alkyl aryl selenones (980-1040 ppm).[3] are compatible with shielding effects due to conjugation. The presence in the IR spectrum of two strong absorptions around 930 and 880 cm-^{1}, diagnostic for the asymmetric and symmetric Se=O stretching frequencies, respectively, completed the characterization.[10d,21]

Conclusions

In conclusion, the development of simple methods for the preparation of synthetically[10-14] and biologically[22] useful selenones is desirable. Our method expands the use of Oxone to the synthesis of vinyl selenones providing a convenient alternative in respect to the conventional procedure with peroxyacids. Other advantages consist in easy handling, clean reactions, no organic wastes derived from oxidant, easy work up, and good yields. The development of a practical synthesis of vinyl selenones should trigger further advances in the chemistry of this interesting class of organoselenium compounds.

Experimental Section

Materials and methods

Commercial reagents and solvents were purchased from Sigma Aldrich, Alfa Aesar or VWR International and used without further purifications. According to literature procedures[23] the starting vinyl selenides **1a-f** were prepared by nucleophilic vinylic substitution of commercially or easily available vinyl bromides or by selenodecarboxylation of cinnamic acids. The vinyl selenides **1g-h** were synthesized from the corresponding alkenes by a sequence of selenobromination and dehydrobromination with LDA.[24] The selenides **1i,n-o** were prepared by reduction with DIBAL-H of the corresponding alkynyl selenides.[25] Finally the vinyl selenides **1j-m** were obtained by treatment of diphenyl diselenide with an excess of commercial vinyl magnesium bromides.[26] ^{1}H NMR, ^{13}C NMR (acquired using a JMODXH pulsed sequence) and ^{77}Se NMR spectra were recorded respectively at 400, 100 and 76.7 MHz, with a Bruker Avance-DRX 400 instrument. Chemical shifts (δ) are reported in ppm respecting residual solvent signals ($CHCl_3$ 7.26 ppm for ^{1}H NMR, $CDCl_3$ 77.0 ppm for ^{13}C NMR). For ^{77}Se NMR the chemical shifts are relative to external diphenyl diselenide in $CDCl_3$ (δ = 463.0 ppm relative to Me_2Se δ = 0.0 ppm). Coupling constants *J* are expressed in Hz. The following abbreviations are used to indicate the multiplicity: s, singlet; d, doublet; t, triplet; q, quartet; (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sex = sextet, m = multiplet;. FT-IR spectra were recorded with a Jasco 410 spectrometer equipped with a diffuse reflectance accessory. High resolution mass spectra (HRMS) were recorded on Agilent 6540-UHD Accurate Mass QTOF LC/MS instrument or APEX IV 7T FTICR. The peaks arising from the selenium–80 isotope are given. The melting points are uncorrected. Thin layer chromatography (TLC) was performed in 60 F254 (Merck) silica gel supported on aluminium sheets. Reaction products were purified by column chromatography on Merck 60 (70-230 mesh) silica gel.

General procedure for the synthesis of selenones 3

To the Oxone triple salt ($2KHSO_5$ $KHSO_4$ K_2SO_4, 2.2 equiv.) dissolved in 5 mL of H_2O the vinyl selenides **1a-p** or the Ph_2Se **1q** (1 equiv.) were added. The reaction mixtures were vigorously stirred at 60 °C for 3-24 hours. Then, the reactions were cooled to room temperature and extracted with ethyl acetate (3 x 5 mL). After drying with Na_2SO_4, the organic extracts were filtered and evaporated under reduced pressure. The crude mixtures were purified through a short plug of silica gel giving access to the corresponding selenones **3**. The vinyl selenoxide **2a** was recovered as a single product when 0.6 equivalents of Oxone were used in the oxidation of **1a**.

Phenyl (*E*)-phenyl vinyl selenoxide 2a [11q]

The crude product was purified by chromatography (gradient elution: from dichloromethane to dichloromethane/methanol 98:2) to afford **2a** as a white solid (m.p. 100-101°C) in 72% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 7.78-7.71 (m, 2H), 7.54-7.42 (m, 6H), 7.38-7.32 (m, 3H), 7.09 (d, *J* = 15.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 141.3, 138.7, 133.6, 131.4, 130.0, 129 9, 129.8 (2C), 128.8 (2C), 127.7 (2C), 126.3 (2C). ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 853. FT-IR (KBr): ν_{max} 972.0 cm^{-1} (Se=O stretching). HRMS (ESI-TOF) m/z: $[M+H]^+$calcd for $C_{14}H_{13}OSe$ 277.0132; found: 277.0121.

Phenyl (*E*)-phenyl vinyl selenone 3a [11q]

The crude product was purified by chromatography (gradient elution: from dichloromethane to dichloromethane/methanol 99:1) to afford **3a** as a white solid (m.p. 87-89°C) in 83% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 8.04 (d, *J* = 7.5 Hz, 2H), 7.92 (d, *J* = 15.6 Hz, 1H), 7.75-7.65 (m, 2H), 7.56-7.43 (m, 6H), 7.19 (d, *J* = 15.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 145.3, 142.0, 134.1, 131.8, 131.5, 130.2 (2C), 129.2 (2C), 128.7 (2C), 127.3, 126.8 (2C). ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 964. FT-IR (KBr): ν_{max} 938.0, 878.4 cm^{-1} (Se=O stretching). HRMS (ESI-TOF) m/z: $[M+H]^+$calcd for $C_{14}H_{13}O_2Se$ 293.0080; found: 293.0086.

(*E*)-2-(4-Chlorophenyl)vinyl phenyl selenone 3b [11a,12b]

The crude product was purified by chromatography (gradient elution: from dichloromethane to dichloromethane/methanol 98:2) to afford **3b** as a white solid (m.p. 159-161°C) in 68% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 8.03 (d, *J* = 7.5 Hz, 2H), 7.87 (d, *J* = 15.3 Hz, 1H), 7.75-7.64 (m, 3H), 7.48 (d, *J* = 8.3 Hz, 2H), 7.43 (d, *J* = 8.3 Hz, 2H), 7.16 (d, *J* = 15.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.8, 141.9, 138.0, 134.2, 130.3 (2C), 130.0, 129.9 (2C), 129.6 (2C), 128.0, 126.8 (2C). ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 963. FT-IR (KBr): ν_{max} 932.9, 880.8 cm^{-1} (Se=O stretching). HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_{14}H_{12}ClO_2Se$ 326.9691; found: 326.9696.

(*E*)-2-(4-Methylphenyl)vinyl phenyl selenone 3c[11a,12b]

The crude product was purified by chromatography (eluent: dichloromethane) to afford **3c** as a white solid (m.p. 145-146 °C) in 64% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 8.05-8.00 (m, 2H), 7.88 (d, *J* = 15.4 Hz, 1H), 7.75-7.63 (m, 3H), 7.44 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 7.13 (d, *J* = 15.4 Hz, 1H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 145.3, 142.7, 142.2, 134.1, 130.2 (2C), 130.0 (2C), 128.8, 128.7 (2C), 126.8 (2C), 126.0, 21.6. ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 964. FT-IR (KBr): ν_{max} 932.4, 877.4 cm^{-1} (Se=O stretching). HRMS (ESI-TOF) m/z: $[M+H]^+$calcd for $C_{15}H_{15}O_2Se$ 307.0237; found: 307.0243.

(*E*)-2-(4-Methoxyphenyl)vinyl phenyl selenone 3d[11a,12b]

The crude product was purified by chromatography (eluent: dichloromethane/methanol 99:1) to afford **3d** as a brownish solid (m.p. 138-140 °C) in 53% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 8.04 (d, *J* = 7.6 Hz, 2H), 7.86 (d, *J* = 15.4 Hz, 1H), 7.74-7.64 (m, 3H), 7.50 (d, *J* = 7.5 Hz, 2H), 7.01 (d, *J* = 15.4 Hz, 1H), 6.96 (d, *J* = 7.8 Hz, 2H), 3.87 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 163.0, 145.4, 142.8, 134.4, 131.1 (2C), 130.6 (2C), 127.2 (2C), 124.7, 124.6, 115.1 (2C), 55.9. ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 965. FT-IR (KBr): ν_{max} 934.8, 880.4 cm^{-1} (Se=O stretching). HRMS (ESI-TOF) m/z: $[M+Na]^+$calcd for $C_{15}H_{14}NaO_3Se$ 345.0005; found: 345.0016.

(*E*)-2-(2-Methylphenyl)vinylphenylselenone 3e[12b]

The crude product was purified by chromatography (eluent: dichloromethane/methanol 99:1) to afford **3e** as a white solid (m.p. 110-112 °C) in 86% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 8.19 (d, *J* = 15.3 Hz, 1H), 8.05 (d, *J* = 7.5 Hz, 2H), 7.75-7.66 (m, 3H), 7.51 (d, *J* = 7.5 Hz, 1H), 7.37 (t, *J* = 7.2 Hz, 1H), 7.29-7.24 (m, 2H), 7.11 (d, *J* = 15.3 Hz, 1H), 2.50 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.1, 142.2, 138.5, 134.1, 131.6, 131.2, 130.6, 130.2 (2C), 128.3, 1271, 126.9 (2C), 126.6, 19.8. ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 964. FT-IR (KBr): ν_{max} 936.3, 879.4 cm^{-1}. HRMS (ESI-TOF) m/z: $[M+Na]^+$calcd for $C_{15}H_{14}NaO_2Se$ 329.0056; found: 329.0058.

(1*E*)-Oct-1-en-1-yl phenyl selenone 3g[12b]

The crude product was purified by chromatography (eluent: dichloromethane/methanol 99:1) to afford **3g** as a yellow oil in 89% yield and 95:5 E/Z ratio. E isomer: ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 8.01-7.85 (m, 2H), 7.75-7.52 (m, 3H), 7.16 (td, *J* = 6.6, 15.1 Hz, 1H,), 6.61 (d, *J* = 15.1 Hz, 1H), 2.37-2.26 (m, 2H), 1.54-1.05 (m, 8H), 0.84 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.5, 141.9, 134.0, 130.1 (2C), 129.7, 126.7 (2C), 32.1, 31.3, 28.6, 27.2, 22.3, 13.9. ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 961. FT-IR (KBr): ν_{max} 938.7, 882.8 cm^{-1}. HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd for $C_{14}H_{20}NaO_2Se$ 323.0526; found: 323.0538.

(*E*)-Hex-1-en-1yl phenyl selenone 3h[11o]

The crude product was purified by chromatography (gradient elution: from light petroleum/ethyl acetate 70:30 to light petroleum/ethyl acetate 50:50) to afford **3h** as a yellow oil in 67% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 8.0-7.93 (m, 2H), 7.74-7.62 (m, 3H), 7.21 (td, *J* = 7.0, 15.1 Hz, 1H), 6.63 (d, *J* = 15.1 Hz, 1H), 2.37 (q, *J* = 7.0 Hz, 2H), 1.51 (quin, *J* = 7.0 Hz, 2H), 1.37 (sex, *J* = 7.3 Hz, 2H), 0.92 (t, *J* = 7.3, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.5, 141.9, 134.0, 130.2 (2C), 129.6, 126.8 (2C), 31.8, 29.4, 22.1, 13.7. ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25 °C): δ = 961. FT-IR (KBr): ν_{max} 937.7, 882.3 cm^{-1}. HRMS (ESI-TOF) m/z: $[M+Na]^+$calcd for $C_{12}H_{16}NaO_2Se$ 295.0213; found: 295.0224.

(*Z*)-Hex-1-en-1yl phenyl selenone 3i

The crude product was purified by chromatography (gradient elution: from light petroleum ether/ethyl acetate 70:30 to light petroleum ether/ethyl acetate 50:50) to afford **3i** as a pale oil in 45% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 8.0-7.93 (m, 2H), 7.71-7.60 (m, 3H), 6.66 (td, *J* = 7.6, 9.3 Hz, 1H), 6.56 (d, *J* = 9.2 Hz, 1H), 2.67 (q, *J* = 7.6 Hz, 2H), 1.40-1.23 (m, 4H), 0.84 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.7, 143.6, 134.0, 131.6, 130.1 (2C), 126.5 (2C), 30.4, 29.2, 22.1, 13.6. ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 962. FT-IR (KBr): ν_{max} 937.7, 883.2 cm^{-1} (Se=O stretching). HRMS (ESI-TOF) m/z: $[M+Na]^+$calcd for $C_{12}H_{16}NaO_2Se$ 295.0213; found: 295.0222.

Phenyl (1*E*/*Z*)-prop-1-en-1-yl selenone 3j[11f]

The crude product was purified by chromatography (gradient elution: from dichloromethane to dichloromethane/methanol 99:1) to afford **3j** (yellow oil) as a 50:50 mixture of *E*:*Z* isomers in 64% yield.

E isomer:^{1}H NMR (400 MHz, CDCl3, 25 °C, TMS): δ = 7.97-7.91 (m, 2H), 7.74-7.57 (m, 3H), 7.17 (qd, *J* = 7.0, 15.0 Hz, 1H), 6.65 (qd, J = 1.4, 15.0 Hz, 1H), 2.03 (dd, *J* = 1.4, 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 145.9, 141.7, 134.1 (2C), 131.0, 130.2 (2C), 126.7, 17.9. ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 960. *Z* isomer: ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ 8.03–7.96 (m, 2H), 7.74-7.57 (m, 3H), 6.77 (qd, *J* = 7.3, 9.3 Hz, 1H), 6.57 (qd, *J* = 1.4, 9.3 Hz, 1H), 2.25 (dd, *J* = 1.4, 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, 25°C): δ = 145.8, 143.1, 134.1 (2C), 132.3, 130.2 (2C), 126.5, 15.5. ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 962. FT-IR (KBr): ν_{max} 937.7, 883.2 cm^{-1} (Se=O stretching).

Phenyl vinyl selenone 3l[11b]

The crude product was purified by chromatography (gradient elution: from dichloromethane to dichloromethane/methanol 98:2) to afford **3l** as a white solid (m.p. 97-102 °C) in 70% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 8.0–7.91 (m, 2H), 7.77–7.59 (m, 3H), 7.0 (dd, *J* = 9.0, 16.5 Hz, 1H), 6.72 (d, *J* = 16.5 Hz, 1H), 6.46 (d, *J* = 9.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ=141.0, 138.8, 134.4, 131.3, 130.3 (2C), 126.9 (2C). ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25 °C): δ = 961. FT-IR (KBr): ν_{max} 981, 928, 882 cm^{-1} (Se=O stretching). HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd for $C_8H_8NaO_2Se$ 238.9587; found: 238.9586.

4-Chlorophenyl vinyl selenone 3m

The crude product was purified by chromatography (gradient elution: from dichloromethane to dichloromethane/methanol 98:2) to afford **3m** as a pale oil in 83% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 7.87 (d, *J* = 8.4 Hz, 2H), 7.59 (d, *J* = 8.4 Hz, 2H), 7.03 (dd, *J* = 9.0, 16.4 Hz, 1H), 6.71 (dd, *J* = 2.1, 16.4 Hz, 1H), 6.47 (dd, *J* = 2.1, 9.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ=141.1, 139.2, 138.5, 131.7, 130.7 (2C), 128.4 (2C). ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25 °C): δ = 960. FT-IR (KBr): ν_{max} 941.6, 883.7 cm^{-1} (Se=O stretching). HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_8H_8ClO_2Se$ 250.9378; found: 250.9370.

Isopropenyl phenyl selenone 3n

The crude product was purified by chromatography (eluent: dichloromethane/methanol 98:2) to afford **3m** as a pale oil in 50% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 7.95-7.88 (m, 2H), 7.72-7.61 (m, 3H), 6.40-6.37 (m, 1H), 6.02-5.97 (m, 1H), 2.20-2.15 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$, 25°C): δ = 149.6, 139.8, 134.2, 130.3 (2C), 127.2 (2C), 124.9, 16.4. ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 976. FT-IR (KBr): ν_{max} 938.2, 881.3 cm^{-1} (Se=O stretching). HRMS (ESI-TOF) m/z: $[M+H]^+$calcd for $C_9H_{10}NaO_2Se$ 252.9743; found: 252.9739.

1-Butylvinyl phenyl selenone 3o

The crude product was purified by chromatography (eluent: dichloromethane/methanol 99:1) to afford **3n** as a yellow oil in 60% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 7.99-7.94 (m, 2H), 7.74-7.60 (m, 3H), 6.50-6.35 (m, 1H), 6.05-5.95 (m, 1H), 2.54-2.40 (m, 2H), 1.53 (quin, *J* = 7.4 Hz, 2H), 1.29 (sex, *J* = 7.4 Hz, 2H), 0.85 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 154.6, 140.7, 134.1, 130.2 (2C), 127.2 (2C), 123.4, 29.6, 29.2, 21.8, 13.5. ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 977. FT-IR (KBr): ν_{max} 939.6, 881.8 cm^{-1} (Se=O stretching). HRMS (ESI-TOF) m/z: $[M+Na]^+$calcd for $C_{12}H_{16}NaO_2Se$ 295.0213; found: 295.0207.

Diphenyl selenone 3q[7,27]

The crude product was purified by chromatography (eluent: dichloromethane/methanol 99:1) to afford **3q** as a colourless solid (m.p. 141-142 °C) in 80% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 8.02-7.96 (m, 4H), 7.69-7.58 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 142.4 (2C), 134.2(2C), 130.3 (4C), 126.8 (4C). ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 966. FT-IR (KBr): ν_{max} 936.7, 880.4 cm^{-1} (Se=O stretching).

2-methoxy-2-phenylethyl phenyl selenone 4a[11r]

The crude product was purified by chromatography (eluent: dichloromethane/methanol 98:2) to afford **4a** as a white solid (m.p. 162-165 °C) in 50% yield. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C, TMS): δ = 8.06 (d, *J* = 7.5 Hz, 2H), 7.75-7.60 (m, 3H), 7.50-7.10 (m, 5H), 4.97 (dd, *J* = 2.2, 11.2 Hz, 1H), 3.92 (dd, *J* = 11.2, 12.8 Hz, 1H), 3.65 (dd, *J* = 2.2, 12.8 Hz, 2H), 3.18 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ = 143.8, 137.0, 133.9, 129.8 (2C), 129.1 (3C), 127.1 (2C), 126.5 (2C), 77.1, 66.8, 56.5. ^{77}Se NMR (76.27 MHz, $CDCl_3$, 25°C): δ = 988. FT-IR (KBr): ν_{max} 926.2, 884.2 cm^{-1} (Se=O stretching). HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd for $C_{15}H_{16}NaO_3Se$ 347.0162; found: 347.0170.

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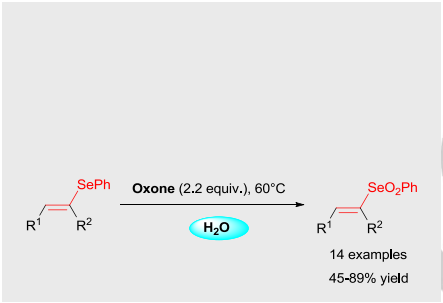

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