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Selenium dioxide as an alternative reagent for the direct α -selenoamidation of aryl methyl ketones

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Abstract: A general strategy for the preparation of *N*, *N*-dialkyl-2-oxo-2arylethaneselenoamides is described. The single step method involves direct coupling of aryl methyl ketones with secondary amines and selenium dioxide in DMSO. The reactions proceeded smoothly at room temperature to provide a number of the α -oxo-selenoamides in good to excellent yields.

During the past decade, organoselenium compounds have attracted much attention in the field of synthetic chemistry because of their interesting biological activities^{1,2} and also as important

reaction intermediates.³ Selenoamides⁴ constitute a class of organoselenium compounds which have been considered to be important precursors for the synthesis of various selenium containing heterocycles⁵ and as pharmaceutical agents.⁶ The α -oxo-selenomides having C=Se bond formation attached directly to the α -carbon of the C=O group are not very common and as per our literature survey, only few methods are available for their synthesis.⁷⁻⁹ The reported methods employed selenylating agents such as ω -selenocyanatoacetophenones (Scheme 1a),⁷ dihaloalkanes-selenium combination (Scheme 1b)⁸ and more recently, Murai *et al* reported the synthesis of α -oxo-selenomides from the reaction of carbonyl compounds with selenocarbamoyllithiums (Scheme 1c).⁹ In all of the above methods the selenylating agents are themselves multi-step synthetic intermediates. Although these methods are quite effective, the use of strong base, harsh reaction conditions and multiple step procedure severely limit their scope of application. Hence, a new methodology for an efficient synthesis of selenoamides starting from easily available starting materials and under mild reaction conditions is highly desirable.

Scheme 1: Synthesis of α -oxo-selenoamides.

Previous Work



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Our Work



Recently, we have demonstrated the versatility of selenium dioxide in organic syntheses where the reagent participated in the reactions as an oxidizing agent in presence of Lewis or Bronsted acids while getting itself reduced to elemental selenium.¹⁰ Thus driven by our continued interest on the synthetic utility of selenium dioxide, we now demonstrate a new reaction where selenium is incorporated in the product, thereby providing an alternative method for the synthesis of α -oxo-selenoamides in a simple one step synthesis. In this paper, we wish to report the coupling of aryl methyl ketones with secondary amines and selenium dioxide in one step leading to an efficient synthetic procedure for α -oxo-selenoamides at room temperature.

Initially when acetophenone (**1a**, 1 equiv) was treated with selenium dioxide (**2**, 1 equiv) and diethylamine (**3a**, 1 equiv) at room temperature for 8 h the reaction product **4a** was formed in 30% yield (Table 1, entry 1). Our efforts to optimize the reaction by varying the stoichiometries of the amine showed no improvement in the product yield (Table 1, entries 2-3). The optimized condition was achieved when the reaction was carried out using dimethyl sulfoxide as the solvent which resulted in the formation of **4a** in 65% yield in 2 h (Table 1, entry 4). Further attempts to improve the efficiency of the reaction by varying the amount of amine and using different solvents provided no significant result (Table 1, entries 5-11).

Table 1: Optimization of the reaction conditions^a



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entry	Substrate 3a (equiv)	Solvent	t (h)	Yield(%) ^b
1	1	-	8	30
2	1.5	-	8	44
3	2	-	8	45
4	1	DMSO	2	65
5	1.25	DMSO	2.5	63
6	1.5	DMSO	2.5	65
7	1	H_2O	12	0
8	1	EtOH	12	0
9	1	DCM	12	20
10	1	CH ₃ CN	12	35
11	1	THF	12	0

^{*a*}Reaction conditions: ketone (**1a**) (1.0 mmol), SeO₂ (1.0 mmol), solvent (0.5 mL), room temperature. ^{*b*}Isolated yields.

Under the optimized reaction conditions, the scope of the reaction of aryl methyl ketones and amines was investigated. First, we carried out the reaction of aromatic ketones with different amines. Secondary amines such as diethylamine (**3a**), pyrrolidine (**3b**), piperidine (**3c**) and morpholine (**3d**) reacted favourably to give their corresponding products (**4a**, 62%; **4b**, 57%; **4c**, 73%; **4d**, **52**%) in moderate to good yields (Scheme 2). It was observed that reactions with diethylamine (**3a**) and piperidine (**3c**) were more effective than with pyrrolidine (**3b**) and morpholine (**3d**), which is probably due to the weaker nucleophilicity of the latter. Secondly, substituted aromatic ketones having electron donating **1b** (*p*-Me), **1e** (*o*-OMe), **1i** (*m*-OH), **1j** (*p*-OH), **1n** (*p*-OMe), and electron withdrawing groups **1c** (*p*-NO₂), **1d** (*m*-NO₂), **1h** (*p*-Cl), **1m** (*p*-Br), were allowed to react with the amines (**3a-3d**). Despite the electronic effects of the substituents of the benzene ring of ketones, the reactions proceeded smoothly to give the desired products (**4e**, 74%; **4f**, 96%; **4g**, 93%; **4h**, 80%; **4k**, 86%; **4l**, 78%; **4m**,61%; **4p**, 70%;

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4q, 58%; **4r**, 55%; **4s**, 81%) in good yields (Scheme 2). The scope of the reaction was also extended to *di*- and *tri*-substituted acetophenones which also gave the corresponding products (**4i**, 53%; **4j**, 55%; **4n**, 54%) in satisfactory yields (Scheme 2). The procedure was also found to work well for ketones with extended aromatic ring such as 2-acetylnaphthalene (**1l**) to give the corresponding product (**4o**, 84%) in good yield (Scheme 2). The solid products formed well-defined crystals and their XRD data (**4f**, **4h**, **4o**, **4r**) (included in the supporting information) further confirmed the structures of the synthesized compounds.

To further strengthen the generality of the method the reaction was extended to the heteroaryl ketones (5). Thus when 2-acetylfuran (5a), 2-acetylthiophene (5b), 2-acetylpyrrole (5c) and 2-acetylpyridine (5d) were allowed to react with diethylamine (3a), pyrrolidine (3b) and piperidine (3c) respectively, their corresponding products (6a, 71%; 6b, 56%; 6c, 51%; 6d, 92%) were obtained in good yields (Scheme 3).

Scheme 2: Substrate scope of aryl methyl ketones^a

$$R^{O} + SeO_{2} + HN^{O} \xrightarrow{DMSO} R^{O} + Se^{O}_{2} + HN^{O}_{2} \xrightarrow{Tt, 2-6 h} R^{O}_{3} \xrightarrow{Se}^{Tt, 2-6 h}$$
1(a-n) 2 3(a-d) 4(a-r)

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temperature. ^bIsolated yields.

Scheme 4: Control Experiment



Evidently the reaction proceeded very well with aliphatic secondary amines (Scheme 4). When acetophenone (1a) was allowed to react with aniline (7a) under the same reaction conditions, formation of the expected product was not observed (Scheme 4a). This may probably be due to the resonance effect of the aromatic ring which render the reaction unreactive. Similarly, when aliphatic primary amine (*n*-propyl amine 9a) was allowed to react with acetophenone (1a) at room temperature, no reaction took place. However, at elevated temperature TLC of the reaction mixture after 1 h displayed formation of intractable multiple products (Scheme 4b). Further to establish whether phenyl glyoxal (11a), the well known oxidation product of acetophenone (1a) with SeO₂ is involved in the reaction, we carried out a reaction using glyoxal (11a) with selenium dioxide under the same reaction conditions. The formation of the expected product was, however not observed (Scheme 4c).

The probable mechanism of the reaction may be depicted as in Scheme 5. The reaction of the enolized ketone (**1**) with selenium dioxide to generate the intermediate **14**,^{10a} a potential umpolung of the aryl ketone¹¹ is believed to be the first step. Subsequent nucleophilic attack by the secondary amine on the α -carbon resulted in the intermediate **15**. The propensity of Se to get reduced to its lower oxidation state resulted in the deprotonation of the α -hydrogenand loss

of another molecule of water leading to the formation of the product **4**. The reaction mechanism proposed above was further strengthened by the fact that the *O*-silyl vinyl ether (**16**) derived from *p*-bromoacetophenone (prepared as per the reported procedure),¹² when reacted with piperidine (**3c**) gave the expected 1-(4-bromophenyl)-2-(piperidin-1-yl)-2-selenoxoethanone (**4t**) in 94% yield (Scheme 6).

Scheme 5: Plausible Mechanism



Scheme 6: Synthesis of 1-(4-bromophenyl)-2-(piperidin-1-yl)-2-selenoxoethanone (4t)



In conclusion, we have demonstrated the application of SeO_2 as a unique selenium source for the synthesis of α -oxoselenoamides from aryl methyl ketones and amines. The method is simple and provides an efficient approach to selenoamide compounds without using any catalyst, acid or base under mild reaction conditions at room temperature. Moreover, the direct use of the easily available starting materials adds to the overall synthetic significance of this procedure.

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EXPERIMENTAL SECTION

General Methods. All chemicals were purchased from commercial companies and were used without further purification unless noted. Melting points were recorded by open capillary tube method and are uncorrected. IR spectra were recorded on a Perkin Elmer Spectrum 400 FTIR instrument, and the frequencies are expressed in cm⁻¹. ¹H-NMR and ¹³C-NMR spectra were recorded on Bruker Avance II-400 spectrometer in CDCl₃ and DMSOd₆ (Chemical shifts are recorded in ppm with TMS as internal standard). ⁷⁷Se NMR spectra were recorded on Mercury Plus 300Hz NMR Spectrometer in ppm using Me₂Se as internal standard. Mass spectral data were obtained with a Waters UPLC-TQD mass spectrometer (ESI-MS). Elemental analyses (C, H, N) were carried out on Perkin Elmer 2400 Series II. All reactions were monitored by thin layer chromatography (TLC) using pre-coated aluminium sheets (silica gel 60 F254 0.2-mm thickness). Flash chromatography was carried out on silica gel (230-400 mesh).

General procedure for α -oxo selenoamides

To a stirring mixture of aryl or heteroaryl methyl ketones (**1**or **5**) (1.0 mmol) and selenium dioxide (1.0 mmol) in DMSO (0.5 mL) amine (**3**) (1.0 mmol) was added. The reaction was allowed to stir for 2-6 h at room temperature. After completion the reaction was diluted with ethyl acetate (10 mL) and washed with brine (2x10 mL). The organic layer was separated, dried over anhydrous NaSO₄ and the solvent removed using rotatory evaporator. The compound was then purified by flash chromatography on silica gel (230-400 mesh) using ethyl acetate and hexane as eluent.

N,N-diethyl-2-oxo-2-phenylselenoacetamide (4a):⁸ The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (9:1); orange oil (227 mg 62%);IR (KBr): 3054, 2978, 2935, 2873, 1658, 1518, 1448, 1286, 1241, 1074, 1053, 697 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.94 (d, *J* = 6.8 Hz, 2H), 7.51 (t, *J* = 7.2 Hz, 1H), 7.40 (t, *J* = 7.2 Hz, 2H), 4.11 (d, *J* = 6.8 Hz, 2H), 3.43 (q, *J* = 7.2 Hz, 2H), 1.40 (t, *J* = 6.8 Hz, 3H), 1.17 (t, *J* = 7.6 Hz, 3H) ppm; ¹³C NMR 10

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(100 MHz, CDCl₃): δ 200.4, 188.4, 133.9, 133.4, 129.7, 128.7, 49.4, 47.8, 13.2, 11.2 ppm. MS (ES⁺) calcd for C₁₂H₁₅NOSe 269.0, found m/z 270.2 [M + H]⁺

1-phenyl-2-(pyrrolidin-1-yl)-2-selenoxoethanone (4b): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); orange solid (151 mg 57%); mp 59-61°C; IR (KBr): 3077, 2980, 2931, 2883, 1693, 1589, 1570, 1488, 1403, 1286, 1200, 1092, 1015 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): *δ* 7.96-7.94 (m, 2H), 7.54-7.50 (m, 1H), 7.42-7.37 (m, 2H), 3.87-3.83 (m, 2H), 3.34-3.31 (m, 2H), 2.08-2.01 (m, 4H) ppm; ¹³C NMR (100 MHz, CDCl₃): *δ* 196.3, 189.9, 134.2, 132.6, 130.0, 128.8, 54.4, 52.6, 26.3, 23.8 ppm.MS (ES⁺) calcd for C₁₂H₁₃NOSe 267.0, found m/z 268.2 [M + H]⁺. Anal. Calcd for C₁₂H₁₃NOSe:C, 54.14; H, 4.92; N, 5.26.Found:C, 54.17; H, 4.85; N, 5.29.

1-phenyl-2-(piperidin-1-yl)-2-selenoxoethanone (4c): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (9:1); yellow solid (203 mg 73%); mp 74-76°C; IR (KBr): 3065, 2935, 2853, 1658, 1523, 1449, 1241, 1226, 1001 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.96 (d, *J*=7.2 Hz, 2H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.41 (t, *J* = 8 Hz, 2H), 4.30 (t, *J* = 5.6 Hz, 2H), 3.46 (t, *J* = 5.2 Hz, 2H), 1.83 (quint, *J* = 5.6 Hz, 2H), 1.72 (quint, *J* = 5.6 Hz, 2H), 1.57(s, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 199.1, 188.8, 134.0, 133.3, 129.6, 128.8, 54.6, 52.0, 26.3, 25.3, 23.8 ppm. MS (ES⁺) calcd for C₁₃H₁₅NOSe 281.0, found m/z 282.0 [M + H]⁺. Anal. Calcd for C₁₃H₁₅NOSe: C, 55.72; H, 5.40; N, 5.00.Found: C, 55.79; H, 5.36; N, 5.03.

1-morpholino-2-phenylethane-1,2-dione (4d): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); orange solid (146 mg 52%); mp 103-105°C; IR (KBr): 3054, 2967, 2851, 1649, 1511, 1448, 1275, 1264, 1105, 1019 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.97-7.95 (m, 2H), 7.56-7.52 (m, 1H), 7.42 (t, *J* = 8 Hz, 2H), 4.38 (t, *J* = 4.8 Hz, 2H), 3.89 (t, *J* = 4.8 Hz, 2H), 3.64 (t, *J* = 4.4 Hz, 2H), 3.51 (t, *J* = 4.8 Hz, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 201.0, 188.9, 134.3, 133.2, 129.7, 128.9, 66.3, 66.2, 53.6, 50.8 ppm. MS (ES⁺) calcd for C₁₂H₁₃NO₂Se 283.0, found m/z 284.2 [M + H]⁺. Anal. Calcd for C₁₂H₁₃NO₂Se: C, 51.07; H, 4.64; N, 4.96. Found: C, 51.04; H, 4.67; N, 4.99.

N,*N*-diethyl-2-oxo-2-(p-tolyl)ethaneselenoamide (4e): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (9:1);orange oil (207 mg 74%); IR (KBr): 3028, 2977, 2935, 2873, 1654, 1605, 1570, 1513, 1447, 1381, 1286, 1250, 1178, 1072 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.83 (d, *J*=8.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 4.09 (s, 2H), 3.42(q, *J* = 7.2 Hz, 2H), 2.34 (s, 3H), 1.39 (t, *J* = 7.2 Hz, 3H), 1.17 (t, *J* = 7.2 Hz, 3H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 200.6, 188.4, 145.0, 130.8, 129.9, 129.4, 49.3, 47.8, 21.8, 13.2, 11.2 ppm. MS (ES⁺) calcd for C₁₃H₁₇NOSe 283.0, found m/z 284.2 [M + H]⁺. Anal. Calcd for C₁₃H₁₇NOSe: C, 55.32; H, 6.07; N, 4.96. Found: C, 55.38; H, 6.03; N, 4.91.

N,N-diethyl-2-(4-nitrophenyl)-2-oxoethaneselenoamide (4f): The product was isolated by flash chromatography using hexane/ethyl acetate (4:1); orange solid (293 mg 94%); mp 123-125°C; IR (KBr): 3103, 2978, 2939, 1670, 1526, 1436, 1346, 1244, 1201, 1074 cm⁻¹;¹H NMR (400 MHz, CDCl₃): δ 8.23 (d, *J* = 8.8 Hz, 2H), 8.11(d, *J* = 9.2 Hz, 2H), 4.11(q, *J* = 7.2 Hz, 2H), 3.47(q, *J*=7.6 Hz, 2H), 1.42(t, *J* = 7.2 Hz, 3H), 1.21(t, *J* = 7.2 Hz, 3H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 197.5, 183.6, 149.3, 137.8, 129.7, 122.7, 48.6, 47.1, 12.4, 10.2 ppm; ⁷⁷Se NMR (57.25 MHz, CDCl₃): δ 681.631. MS (ES⁺) calcd for C₁₂H₁₄N₂O₃Se 314.0, found m/z 337.3 [M + Na]⁺.

N,N-diethyl-2-(3-nitrophenyl)-2-oxoethaneselenoamide (4g): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); orange solid (299 mg 96%); mp 198-200°C; IR (KBr): 3082, 2979, 2937, 2874, 1664, 1644, 1532, 1437, 1350, 1290, 1238, 1097, 703 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.75 (s, 1H), 8.36-8.27 (m, 2H), 7.61 (t, *J*=8.0, 1H), 4.12 (q, *J* = 7.2 Hz, 2H), 3.47(q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H), 1.22 (t, *J*= 7.2 Hz, 3H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 198.3, 184.6, 148.3, 135.4, 135.1, 129.9, 127.8, 124.3, 49.7, 48.2, 13.5, 11.3 ppm. MS (ES⁺) calcd for C₁₂H₁₄N₂O₃Se 314.0, found m/z 315.4 [M + H]⁺. Anal. Calcd for C₁₂H₁₄N₂O₃Se: C, 46.02; H, 4.51; N, 8.94. Found: C, 46.10; H, 4.45; N, 8.89.

N,N-diethyl-2-(2-methoxyphenyl)-2-oxoethaneselenoamide (4h): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); orange solid (237 mg 80%); mp 113-115°C; IR (KBr): 3076, 2982, 2940, 2838, 1637, 1597, 1537, 1437, 1383, 1297, 1281, 1112, 1015, 760 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.95 (dd, *J* = 2, 2 Hz, 1H), 7.48-7.44 (m,

1H), 7.03- 6.99 (m, 1H), 6.88 (d, *J*= 8.4, 1H), 4.02 (q, *J*=6.8 Hz, 2H), 3.75 (s, 3H), 3.47 (q, *J*=6.8 Hz, 2H) , 1.36 (t, *J* = 7.2 Hz, 3H), 1.24 (t, *J* = 6.8 Hz, 3H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 201.4, 186.2, 157.7, 134.2, 130.7, 123.2, 120.2, 111.3, 54.5, 48.3, 46.3, 11.6, 9.9 ppm; ⁷⁷Se NMR (57.25 MHz, CDCl₃): δ 525.721. MS (ES⁺) calcd for C₁₃H₁₇NO₂Se 299.04, found m/z 300.2 [M + H]⁺.

2-(2,4-dimethylphenyl)-*N,N*-diethyl-2-oxoethaneselenoamide (4i): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (9:1); orange oil (156 mg 53%); IR (KBr): 3077, 2980, 2931, 2883, 1693, 1589, 1570, 1488, 1403, 1286, 1200, 1092, 1015 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.52 (d, *J* = 7.6 Hz, 1H), 7.02(s, 1H), 6.97 (d, *J* = 8.4 Hz, 1H), 4.09 (q, *J* = 6.8 Hz, 2H), 3.47(q, *J* = 6.8 Hz, 2H), 2.58(s, 3H), 2.28 (s, 3H), 1.39 (t, *J* = 7.2 Hz, 3H), 1.21 (t, *J*= 7.2 Hz, 3H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 202.0, 189.8, 143.9, 141.9, 133.4, 131.9, 129.4, 126.3, 49.4, 47.9, 22.1, 21.5, 13.1, 11.1 ppm. MS (ES⁺) calcd for C₁₄H₁₉NOSe 297.0, found m/z 298.2 [M + H]⁺. Anal. Calcd for C₁₄H₁₉NOSe: C, 56.76; H, 6.46; N, 4.73. Found: C, 56.73; H, 6.48; N, 4.70.

2-(3,4-dimethoxyphenyl)-*N*,*N*-diethyl-2-oxoethaneselenoamide (4j): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); yellow solid (180 mg 55%); mp 125-127°C; IR (KBr): 3077, 2980, 2931, 2883, 1693, 1589, 1570, 1488, 1403, 1286, 1200, 1092, 1015 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.55-7.49 (m, 2H), 6.81(d, *J* = 8.4 Hz, 1H),4.11 (s, 2H), 3.88 (s, 3H), 3.87(s, 3H), 3.43(q, *J* = 7.6 Hz,2H), 1.40 (t, *J* = 6.8 Hz, 3H), 1.19 (t, *J* = 7.2 Hz, 3H) ppm;¹³C NMR (100 MHz, CDCl₃): δ 199.5, 187.0, 152.9, 148.1, 125.2, 124.3, 110.0, 109.2, 55.1, 55.0, 48.3, 46.8, 12.2, 10.2 ppm; ⁷⁷Se NMR (57.25 MHz, CDCl₃): δ 638.233. MS (ES⁺) calcd for C₁₄H₁₉NO₃Se 329.0, found m/z 330.2 [M + H]⁺. Anal. Calcd for C₁₄H₁₉NO₃Se: C, 51.22; H, 5.83; N, 4.27. Found: C, 51.33; H, 5.78; N, 4.29.

1-(4-chlorophenyl)-2-(pyrrolidin-1-yl)-2-selenoxoethanone (4k):⁷ The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); yellow solid (257 mg 86%); mp 143-145°C; IR (KBr): 3060, 2964, 2874, 1656, 1586, 1514, 1445, 1262, 1158, 1090 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.92 (d, *J* = 8.4 Hz, 2H), 7.37(d, *J* = 8.8 Hz, 2H), 3.85(t, *J* = 6.8 Hz, 2H), 3.33(t, *J* = 5.6 Hz, 2H), 2.09-2.03 (m, 4H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 195.7, 13

188.4, 140.6, 131.4, 131.3, 129.1, 54.5, 52.6, 26.3, 23.8 ppm; ⁷⁷Se NMR (57.25 MHz, CDCl₃): δ 692.568. MS (ES⁺) calcd for C₁₂H₁₂ClNOSe 300.9, found m/z 302.1 [M + H]⁺.

1-(3-hydroxyphenyl)-2-(pyrrolidin-1-yl)-2-selenoxoethanone (4l): Theproduct was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1);yellow oil(268 mg 78%);IR (KBr): 3309, 3060, 2982, 2950, 2873, 1661, 1596, 1527, 1445, 1291, 1207, 1153, 1056 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.46-7.42 (m, 2H), 7.23 (t, *J* = 8.0 Hz, 1H), 7.01-6.98 (m,1H), 6.78 (s, 1H), 3.84 (t, *J* = 7.2 Hz, 2H), 3.32 (t, *J* = 6.8, Hz, 2H), 2.06-2.01 (m, 4H) ppm;¹³C NMR (100 MHz, CDCl₃): δ 195.8, 190.1, 156.6, 133.5, 130.1, 122.2, 122.1,116.2, 54.6, 52.9, 26.2, 23.8 ppm. MS (ES⁺) calcd for C₁₂H₁₃NO₂Se 283.0, found m/z 284.2 [M + H]⁺.Anal. Calcd for C₁₂H₁₃NO₂Se: C, 51.07; H, 4.64; N, 4.96. Found: C, 51.11; H, 4.60; N, 4.92.

1-(4-hydroxyphenyl)-2-(piperidin-1-yl)-2-selenoxoethanone (4m): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); yellow solid (180 mg 61%); mp 148-150°C; IR (KBr): 3433, 3165, 2948, 2937, 2861, 1651, 1609, 1539, 1435, 1313, 1281, 1242, 1115, 1002, 614 cm⁻¹;¹H NMR (400 MHz, CDCl₃): δ 7.80 (d, *J* = 8.4 Hz, 2H), 6.97 (s, 1H), 6.77 (d, *J* = 8.4 Hz, 2H), 4.27 (s, 2H), 3.46 (s, 2H), 1.80-1.18(m, 6H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 198.2, 188.0, 163.5, 132.3, 124.4, 116.0, 54.4, 51.8, 26.4, 25.5, 23.5 ppm. MS (ES⁺) calcd for C₁₃H₁₅NO₂Se 297.0, found m/z 298.2 [M + H]⁺. Anal. Calcd for C₁₃H₁₅NO₂Se: C, 52.71; H, 5.10; N, 4.73. Found: C, 52.77; H, 5.07; N, 4.71.

1-mesityl-2-(piperidin-1-yl)-2-selenoxoethanone (4n): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (9:1); orange solid (173 mg 54%); mp 93-95°C; IR (KBr): 3021, 2943, 2925, 2856, 1644, 1608, 1518, 1442, 1377, 1235, 1221, 1113, 1001 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 6.77 (s, 2H), 4.24 (t, *J* = 5.6 Hz, 2H), 3.77(t, *J* = 6.0 Hz, 2H), 2.66(s, 6H), 2.22(s, 3H), 1.81-1.77(m, 6H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 200.6, 189.9, 140.5, 136.4, 136.2, 129.4, 54.7, 53.7, 26.1, 25.4, 24.0, 21.2, 20.3 ppm. MS (ES⁺) calcd for C₁₆H₂₁NOSe 323.0, found m/z 324.3 [M + H]⁺. Anal. Calcd for C₁₆H₂₁NOSe: C, 59.62; H, 6.57; N, 4.35.Found: C, 59.70; H, 6.51; N, 4.38.

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1-(naphthalen-2-yl)-2-(piperidin-1-yl)-2-selenoxoethanone (40): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (9:1); yellow solid (277 mg 84%); mp 140-142°C; IR (KBr): 3060, 2938, 2917, 2852, 1644, 1628, 1513, 1445, 1352, 1290, 1213, 1188, 1108 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): *δ* 8.49 (s, 1H), 7.99 (dd, *J* = 2.0, 1.6 Hz,1H), 7.89-7.79 (m, 3H), 7.56-7.46 (m, 2H), 4.35 (t, *J* = 5.6 Hz, 2H), 3.49(s, 2H),1.86 (quint, *J* = 5.6 Hz, 2H),1.72 (quint, *J*= 5.6 Hz, 2H), 1.58 (s, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃): *δ* 199.1, 188.9, 136.0, 132.4, 132.0,130.6, 129.6, 129.1, 128.7, 127.9, 127.0, 124.4, 54.7, 52.1, 26.3, 25.4, 23.8 ppm; ⁷⁷Se NMR (57.25 MHz, CDCl₃): *δ* 605.130; MS (ES⁺) calcd for C₁₇H₁₇NOSe 331.0, found m/z 332.2 [M + H]⁺.

1-(4-bromophenyl)-2-morpholino-2-selenoxoethanone (4p): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); yellow solid(252 mg 70%);mp 177-179°C; IR (KBr): 3059, 2966, 2914, 2856, 1654, 1584, 1505, 1435, 1398, 1271, 1231, 1115 cm⁻¹;¹H NMR (400 MHz, DMSOd₆): δ 7.23 (d, *J*=8.8 Hz, 2H), 7.13 (d, *J* = 8.8 Hz, 2H), 3.68 (s, 2H), 3.22(t, *J* = 4.8 Hz, 2H), 2.94 (s, 4H), ppm; ¹³C NMR (100 MHz, DMSOd₆): δ 197.3, 186.8, 132.1, 132.0, 131.1, 128.5, 65.6, 65.5, 53.7, 50.7 ppm; ⁷⁷Se NMR (57.25 MHz, CDCl₃): δ 663.013. MS (ES⁺) calcd for C₁₂H₁₂BrNO₂Se 360.9, found m/z 362.2 [M + H]⁺. Anal. Calcd for C₁₂H₁₂BrNO₂Se: C, 39.91; H, 3.35; N, 3.88.Found: C, 39.94; H, 3.29; N, 3.81.

1-(4-methoxyphenyl)-2-morpholino-2-selenoxoethanone (4q):⁷ The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); yellow solid (180 mg 58%); mp 172-174°C; IR (KBr): 2994, 2975, 2932, 2851, 1650, 1593, 1519, 1441, 1316, 1264, 1234, 1161, 1112, 1023 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.94 (d, *J* = 8.8 Hz, 1H), 6.89 (d, *J* = 8.8 Hz, 1H), 4.37 (t, *J* = 4.8 Hz,2H), 3.88 (t, *J* = 5.2 Hz, 2H), 3.81 (s, 3H), 3.63 (s, 2H), 3.51 (s, 2H) ppm;¹³C NMR (100 MHz, DMSOd₆): δ 198.6, 187.4, 164.0, 131.8, 125.4, 114.4, 65.6, 65.5, 55.7, 53.5, 50.6 ppm. MS (ES⁺) calcd for C₁₃H₁₅NO₃Se 313.0, found m/z 314.2 [M + H]⁺.

2-morpholino-1-(3-nitrophenyl)-2-selenoxoethanone (4r): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (9:1); orange solid (179 mg 55%); mp 196-198°C; IR (KBr): 3071, 2976, 2921, 2862, 1661, 1612, 1529, 1460, 1355, 1259, 1230, 15

1107, 1020, 703 cm⁻¹; ¹H NMR (400 MHz, CDCl₃+DMSOd₆1:4):δ 8.65 (s, 1H), 8.49 (d, *J* = 7.6 Hz, 1H), 8.36 (d, *J* = 8 Hz, 1H), 7.85 (t, *J* = 8.0 Hz, 1H), 4.35 (t, *J* = 4.4 Hz, 2H), 3.88 (t, *J* = 4.8 Hz, 2H), 3.64 (s, 2H), 3.63 (s, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃+DMSOd₆1:4):δ 196.3, 185.0, 148.0, 135.3, 134.4, 130.8, 128.2, 123.2, 65.7, 65.5, 53.8, 50.8 ppm. MS (ES⁺) calcd for C₁₂H₁₂N₂O₄Se 328.0, found m/z 351.2 [M + Na]⁺.

1-(3-nitrophenyl)-2-(piperidin-1-yl)-2-selenoxoethanone (4s): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); yellow solid (263 mg 81%);mp 115-117°C; IR (KBr): 3082, 2956, 2923, 2857, 1664, 1533, 1444, 1351, 1278, 1223, 1091, 704 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 8.75 (s, 1H), 8.36- 8.29 (m, 2H) 7.62 (t, *J* = 8.0 Hz, 1H), 4.31(t, *J* = 5.6 Hz, 2H), 3.50 (t, *J* = 5.6 Hz, 2H), 1.86 (quint, *J* = 5.2 Hz, 2H), 1.76 (quint, *J* = 5.2 Hz, 2H), 1.62 (quint, *J* = 5.2 Hz, 2H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 196.1, 184.0, 147.4, 134.4, 133.9, 129.0, 126.9, 123.2, 53.9, 51.2, 25.5, 24.4, 22.8 ppm. MS (ES⁺) calcd for C₁₃H₁₄N₂O₃Se 326.0, found m/z 327.2 [M + H]⁺. Anal. Calcd forC₁₃H₁₄N₂O₃Se: C, 48.01; H, 4.34; N, 8.61. Found: C, 48.11; H, 4.30; N, 8.65.

1-(4-bromophenyl)-2-(piperidin-1-yl)-2-selenoxoethanone (4t): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (9:1); orange solid (337 mg 94%); mp 138-140°C; IR (KBr): 3076, 2936, 2856, 1662, 1579, 1519, 1438, 1221, 1005 cm⁻¹; ¹H NMR (400 MHz, DMSOd₆): δ 7.85 (d, *J*=8.4 Hz, 2H), 7.79 (d, *J* = 8.8 Hz, 2H), 4.29 (t, *J* = 4.8 Hz, 2H), 3.54 (t, *J* = 4.8 Hz, 2H), 1.78-1.68 (m, 4H), 1.72 (s, 2H) ppm; ¹³C NMR (100 MHz, DMSOd₆): δ 195.2, 186.1, 131.6, 131.5, 130.5, 127.8, 53.8, 50.9, 25.4, 24.5, 22.4 ppm. MS (ES⁺) calcd for C₁₃H₁₄BrNOSe 358.9, found m/z 359.9 [M + H]⁺. Anal. Calcd for C₁₃H₁₄BrNOSe: C, 43.48; H, 3.93; N, 3.90. Found: C, 43.53; H, 3.89; N, 3.94.

N,*N*-diethyl-2-(furan-2-yl)-2-oxoethaneselenoamide (6a): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); orange oil(183 mg 71%); IR (KBr): 3077, 2980, 2931, 2883, 1693, 1589, 1570, 1488, 1403, 1286, 1200, 1092, 1015 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.58 (dd, *J* = 0.8, 0.8 Hz, 1H), 7.24 (dd, *J* = 0.8, 0.8 Hz, 1H), 6.50 (dd, *J* = 1.6, 1.6 Hz, 1H), 4.05 (q, *J* = 7.2 Hz, 2H), 3.47 (q, *J* = 6.8 Hz, 2H), 1.36 (t, *J* = 7.2 Hz, 3H), 1.19 (t, *J* = 7.2 Hz, 2H)

Hz, 3H) ppm; ¹³C NMR (100 MHz, CDCl₃): δ 198.1, 177.2, 149.6, 147.8, 121.2, 112.7, 49.3, 48.2, 13.2, 11.0 ppm. MS (ES⁺) calcd for C₁₀H₁₃NO₂Se259.0, found m/z 260.2 [M + H]⁺. Anal. Calcd for C₁₀H₁₃NO₂Se: C, 46.52; H, 5.08; N, 5.43. Found: C, 46.56; H, 5.01; N, 5.49.

2-(pyrrolidin-1-yl)-2-selenoxo-1-(thiophen-2-yl)ethanone (6b): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); orange solid (152 mg 56%); mp 89-91°C; IR (KBr): 3076, 2978, 2923, 2868, 1639, 1523, 1445, 1405, 1271, 1124 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): *δ* 7.79 (dd, *J* = 1.2, 1.2 Hz, 1H), 7.66 (dd, *J* = 1.2, 1.2 Hz, 1H), 7.07 (dd, *J* = 4.0, 4.0 Hz, 1H), 3.83-3.79 (m, 2H), 3.42-3.37 (m, 2H), 2.07-2.03 (m, 4H) ppm; ¹³C NMR (100 MHz, CDCl₃): *δ* 194.5, 183.4, 139.7, 136.1, 136.0, 128.5, 54.7, 52.7, 26.3, 23.9 ppm; ⁷⁷Se NMR (57.25 MHz, CDCl₃): *δ* 698.543.MS (ES⁺) calcd for C₁₀H₁₁NOSSe 272.9, found m/z 274.1 [M + H]⁺.

2-(piperidin-1-yl)-1-(1H-pyrrol-2-yl)-2-selenoxoethanone (6c): The product was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); yellow solid(136 mg 51%); mp142-144°C; IR (KBr): 3270, 2936, 2867, 1611, 1520, 1400, 1240, 1099 cm⁻¹; ¹H NMR (400 MHz, DMSOd₆): δ 12.22 (s, 1H), 7.26-7.24 (m, 1H), 6.87-6.85 (m, 1H), 6.30-6.28 (m, 1H), 4.24 (s, 2H), 3.58 (t, *J* = 5.6 Hz, 2H), 1.81-1.70 (m, 4H), 1.57 (s, 2H) ppm; ¹³C NMR (100 MHz, DMSOd₆): δ 197.2, 179.9, 128.2, 127.9, 119.7, 111.1, 54.3, 52.1, 26.5, 25.5, 23.5ppm. MS (ES⁺) calcd for C₁₁H₁₄N₂OSe 270.0, found m/z 271.0 [M + H]⁺.

N,N-diethyl-2-oxo-2-(pyridin-2-yl)ethaneselenoamide (6d): Theproduct was isolated by flash chromatography using hexane/ethyl acetate as eluent (4:1); orange solid(247 mg 92%); mp 79-81°C; IR (KBr): 3049, 2983, 2944, 2873, 1667, 1532, 1433, 1270, 1205, 1074 cm⁻¹; ¹H NMR (400 MHz, DMSOd₆): δ 8.70-8.68 (m, 1H), 8.10-8.02 (m, 2H), 7.66-7.62 (m, 1H), 4.09 (q, *J* = 7.2 Hz, 2H), 3.57 (q, *J* = 7.2 Hz, 2H), 1.35 (t, *J* = 7.2 Hz, 3H), 1.20 (t, *J* = 7.2 Hz, 3Hppm; ¹³C NMR (100 MHz, DMSOd₆): δ 200.1, 186.4, 152.1, 149.8, 138.1, 128.2, 124.1, 50.2, 47.7, 13.4, 11.5ppm. MS (ES⁺) calcd for C₁₁H₁₄N₂OSe 270.0, found m/z 271.0 [M + H]⁺.

ASSOCIATED CONTENT

Supporting Information

Copies of ¹H NMR and ¹³C NMR Spectra and X-ray Crystallography data for **4f**, **4h**, **4o**, **4r**, **6b**, **6c** and **6d**.

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