Synthesis of Substituted *Se*-Phenyl Selenocarboxylates from Terminal Alkynes

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Keywords: *Se*-Phenyl selenocarboxylates / Selenoacetylenes / Alkynes / Acyl transfer

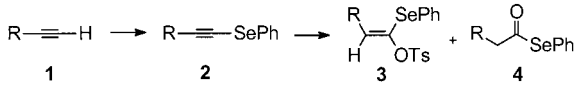
Se-Phenyl selenocarboxylates have been conveniently prepared from (phenylseleno)acetylenes by treatment with *p*-toluenesulfonic acid monohydrate in dichloromethane. This easy conversion is compatible with a broad range of oxygen- and nitrogen-containing functional groups. The *Se*-phenyl selenocarboxylates were easily converted into the corresponding esters and amides.

Introduction

The development of easy and efficient methods for the preparation of selenocarboxylic esters has attracted considerable attention because of their importance as intermediates in organic synthesis.[1a−1c] In fact, among other applications, they are employed as precursors of acyl radicals,[2a−2c] as mild acyl transfer reagents[3a−3c] and as intermediates in the synthesis of ketones.[4a,4b]

Although various syntheses of selenocarboxylic esters have been reported[5a−5i] many of them suffer from difficulties arising from the handling of the reagents used as selenium sources because of their sensitivity to air and moisture. In other cases the preparation of selenocarboxylic esters is difficult and requires strongly basic or acidic reaction conditions.

In previous work[6] we reported that alkynyl phenyl selenides **2** react with *p*-toluenesulfonic acid monohydrate in dichloromethane to give the (*Z*)-α-(phenylseleno)vinyl *p*-toluenesulfonates **3** (Scheme 1) as a result of a regio- and stereospecific *cis* addition. At the same time we also observed that the compounds **3** were contaminated with the selenocarboxylic esters **4**. However, when the reaction was repeated using dry *p*-toluenesulfonic acid, compounds **3** were the only reaction products which were obtained in excellent yields. This stereospecific addition of *p*-toluenesulfonic acid to the triple bond is interesting from a mechanistic point of view. Moreover, we have successfully shown that compounds **3** also have some synthetic importance. For example they can be conveniently employed to effect the synthesis of α-(phenylseleno) acids, esters[7] and lactones.[8]

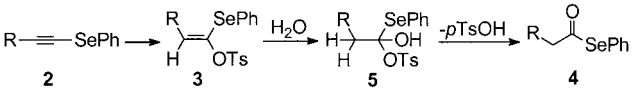


Scheme 1

The observation that in the presence of *p*-toluenesulfonic acid monohydrate compounds **4** were also present in the final reaction mixtures suggested that, when properly modified, our procedure could be easily extended to effect a very simple and mild synthesis of selenocarboxylic esters.[9] In this paper we describe a simple procedure to efficiently convert various substituted (phenylseleno)acetylenes **2** into *Se*-phenyl selenocarboxylates **4**. The starting products **2** are readily available since they can be obtained in good yields from the terminal alkynes **1**.[10a][10b]

Results and Discussion

The reaction of (phenylseleno)acetylenes **2** with an excess of *p*-toluenesulfonic acid monohydrate, in dichloromethane at 40 °C gave the adducts **3** to which 1 equiv. of water was regiospecifically added to afford the intermediates **5**. These compounds were not isolated since they readily eliminate a molecule of *p*-toluenesulfonic acid to give the *Se*-phenyl selenocarboxylates **4** (Scheme 2). The regioselectivity of the addition reaction is a consequence of carbocation stabilization by the selenium atom.



Scheme 2

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Dichloromethane was essential for the efficient formation of the selenocarboxylic esters. In fact, under the same experimental conditions, other solvents, such as DMF, dioxane or acetonitrile, did not promote the formation of compounds **4**.

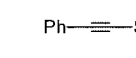
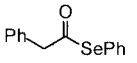
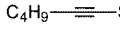
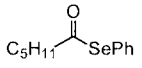
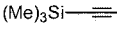
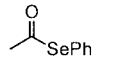
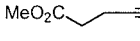
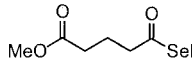
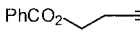
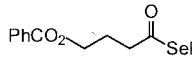
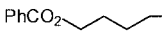
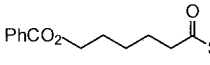
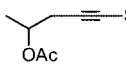
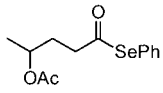
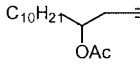
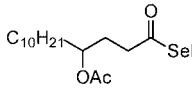
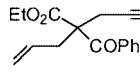
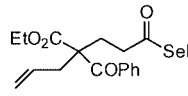
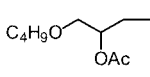
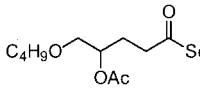
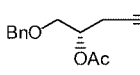
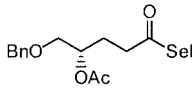
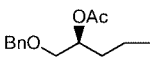
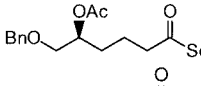
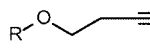
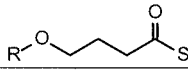
As indicated in Table 1, several types of *Se*-phenyl selenocarboxylates were obtained in fairly good yields. The reaction is highly chemoselective. In fact, different functional groups such as esters, ketones, carbon−carbon double bonds, ethers and acetals were not affected under the reaction conditions employed. Substrates **2j**, **2k**, and **2l** did not give the corresponding selenocarboxylic esters in good yields owing to a competitive intramolecular reaction which led to the formation of the corresponding lactones.[11] Braga et al. recently published[12] the results of their synthesis of *Se*-methyl selenocarboxylates from (methylseleno)acetylenes by treatment with *p*-toluenesulfonic acid in dichloromethane at 40 °C in the presence of silica gel. Under these conditions the reactions of **2b** and **2r** gave only 55% yields of the corresponding selenocarboxylic esters after 23 h. Similar results were obtained when trifluoroacetic acid and silica gel were employed. The course of the reaction can be monitored by TLC and the formation and disappearance of compounds **3** (Scheme 2) could be clearly observed especially in the slower reactions (hydrolysis reaction times range from 1 to 9 h).

In order to explore the range of application of this reaction, experiments were carried out starting from several alkynyl phenyl selenides containing a sulfonylamido, acetamido or phthalimido group. As indicated in Table 2, *Se*-phenyl β-, γ- and δ-(sulfonylamido or -phthalimido)selenocarboxylates were obtained in good yields. In several cases better results were obtained by increasing the reaction temperature to 60 °C. This was particularly important in those cases in which a phthalimido group was present in the molecule.

The reactions of the alkynyl phenyl selenides **7** with an excess of *p*TsOH monohydrate in dichloromethane at 40 °C did not afford the corresponding β-oxy-functionalized selenocarboxylic esters but instead gave the α,β-unsaturated *Se*-phenyl selenocarboxylates (Scheme 3).

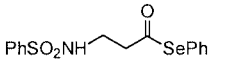
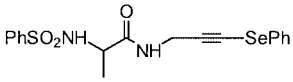
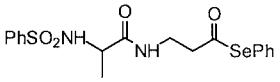
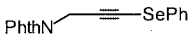
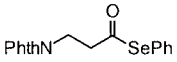
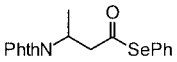
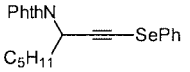
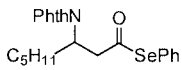
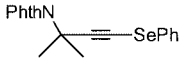
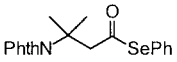
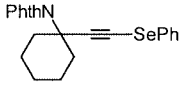
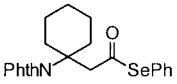
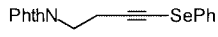
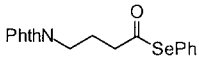
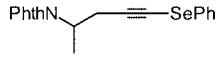
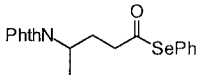
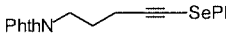
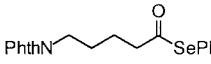
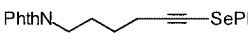
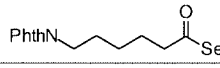
Clearly, with these substrates the *p*-toluenesulfonic acid, instead of adding to the triple bond, preferentially pro-

Table 1. Reaction of (phenylseleno)acetylenes **2** with *p*TsOH in dichloromethane at 40 °C

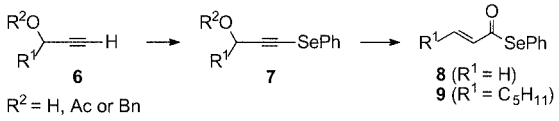
Phenylselenoacetylenes	2	Time [h]	*Se*-Phenyl Selenocarboxylates	4	Yield [%]
	**2a**	6		**4a**	72[a]
	**2b**	9		**4b**	78
	**2c**	2		**4c**	65
	**2d**	1		**4d**	67
	**2e**	6		**4e**	75
	**2f**	6		**4f**	74
	**2g**	8		**4g**	76[a]
	**2h**	7		**4h**	66
	**2i**	3		**4i**	74
	**2j**	2		**4j**	56[a]
	**2k**	3		**4k**	51[a]
	**2l**	7		**4l**	60[a]
	**2m**	8		**4m**	62[b,c]

[a] The reaction was carried out at 60 °C. [b] Traces of the α anomer were also present. [c] R = 2,3,4,6-tetra-*O*-acetyl-β-D-glucopyranosyl.

Table 2. Reaction of *N*-substituted (phenylseleno)acetylenes **2** with *p*TsOH in dichloromethane at 40 °C

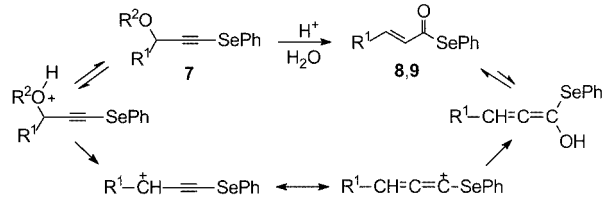
Substrates	2	Time [h]	*Se*-Phenyl Selenocarboxylates	4	Yield [%]
	**2n**	2		**4n**	78[a]
	**2o**	3		**4o**	51[b]
	**2p**	16		**4p**	95
	**2q**	2		**4q**	72
	**2r**	2		**4r**	85
	**2s**	2		**4s**	87[c]
	**2t**	1		**4t**	77
	**2u**	6		**4u**	80[a]
	**2v**	3		**4v**	65[a]
	**2w**	3		**4w**	70[a]
	**2x**	4		**4x**	89[a]

[a] The reaction was effected at 60 °C. [b] The reaction was effected at 30 °C. [c] *p*TsOH (0.5 mmol) was employed.



Scheme 3

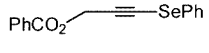
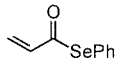
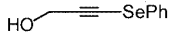
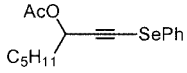
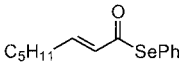
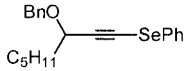
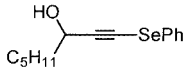
tonates the oxygen atom giving rise to a Meyer−Schuster rearrangement[13] as indicated in Scheme 4. The α,β-unsaturated *Se*-phenyl selenocarboxylates **8** or **9** were obtained in good to excellent yields (Table 3). Better results were obtained with the alkynes **7b** and **7e** in which the oxygen atom was that of a hydroxy group.



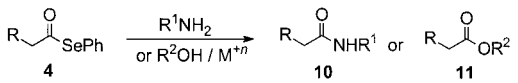
Scheme 4

The results described above and collected in Tables 1 and 2 clearly indicate that the present procedure represents a very convenient method to effect the chemo- and regioselective synthesis of *Se*-phenyl selenocarboxylates. In view of the weakness of the carbon−selenium bond the selenocarboxylic esters are expected to be more reactive than the corresponding thio or oxo esters.[5c,14a−14c] This property makes selenocarboxylic esters valuable acyl transfer agents. They are also very selective in their reactions with different nucleophiles. Thus, as indicated in Scheme 5, the *Se*-phenyl selenocarboxylates **4** react at room temperature in dichloro-

Table 3. Reaction of substituted (phenylseleno)acetylenes **7** with *p*TsOH at 40 °C

Substrates	7	Time [h]	Products		Yield [%]
	**7a**	6		**8**	50
	**7b**	5		**8**	85[a]
	**7c**	7		**9**	50
	**7d**	3		**9**	67
	**7e**	20		**9**	70[a]

[a] Yields of the corresponding esters.



Scheme 5

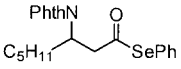
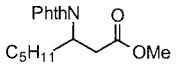
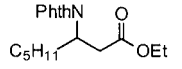
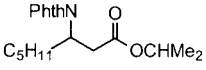
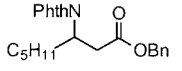
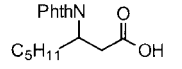
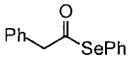
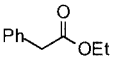
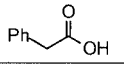
methane with amines or with amino acid derivatives to afford the corresponding amides **10** in good yields. The results of these experiments are collected in Table 4. The phenylseleno moiety was recovered as diphenyl diselenide in every case.

When the acylation of an amino acid with a hydroxy group in the side-chain was performed, the only product obtained was the *N*-acylation product **10d**. Products derived from the acylation of the hydroxy group were not observed. As indicated by the NMR spectrum of the crude reaction mixture compound **10k** was obtained as a single product. This indicates that the amino acid moiety did not suffer any racemization during the acylation reaction.

The acylation of alcohols is more difficult than that of amines and the use of mercuric chloride or copper(II) chloride in acetonitrile is usually necessary.[3a,3b] We have effected some experiments starting with the selenocarboxylic esters **4a** and **4w** (Scheme 5). These reactions were carried out by using an excess (2.2 mmol) of dry $CuCl_2$ in acetonitrile in the presence of 5 mmol of alcohol. As indicated in Table 5 these conversions occurred in good yields. In these reactions diphenyl diselenide was also recovered. The acylation reaction did not take place when diethyl ether or dichloromethane were used as the solvent. Experiments with chiral nonracemic amino-substituted selenocarboxylic esters of type **4** are presently under way.[11]

As a final experiment we treated the *Se*-phenyl selenocarboxylates **4a** and **4w** with a 30% solution of H_2O_2 [15] in tetrahydrofuran at room temperature (Table 5). The corresponding carboxylic acids **11g** and **11e** were obtained in good yields.

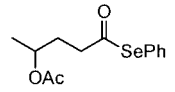
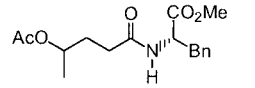
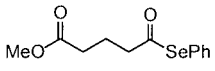
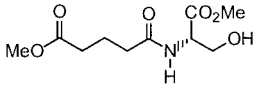
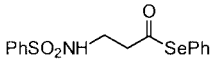
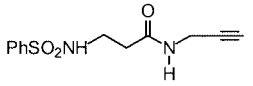
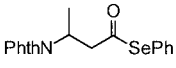
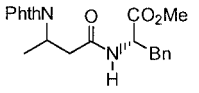
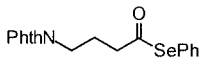
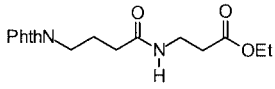
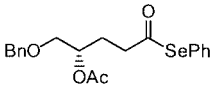
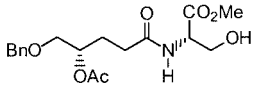
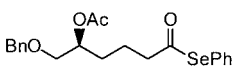
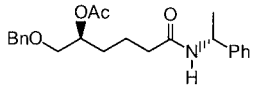
Table 5. Conversion of *Se*-phenyl selenocarboxylates **4** into esters or acids

Substrates	4	Time [h]	Products	11	Yield [%]
	**4w**	2		**11a**	89
	4w	2		**11b**	83
	4w	4		**11c**	72
	4w	2		**11d**	62
	4w	2		**11e**	81
	**4a**	2		**11f**	83
	4a	1		**11g**	71

Conclusions

The procedure reported herein offers several advantages over other methods described in the literature. An important point is that the use of heavy metal selenolates or volatile selenium compounds is avoided. Moreover, the starting materials are readily available, the phenylseleno derivatives can be easily handled, the reactions can be effected under mild conditions and substrates containing several types of functional groups can be used. Finally, when the selenocar-

Table 4. Reaction of *Se*-phenyl selenocarboxylates **4** with amines

Substrates	4	Time [h]	Amides	10	Yield [%]
	**4g**	48		**10g**	68[a,b]
	**4d**	8		**10d**	86[a]
	**4n**	4		**10n**	98
	**4q**	48		**10q**	70[a,b]
	**4u**	2		**10u**	81[a]
	**4k**	16		**10k**	92[a,c]
	**4l**	48		**10l**	82[c]

[a] Et_3N was added. [b] 1:1 mixture of the two diastereoisomers. [c] Single diastereoisomer.

boxylic esters are used as acylating agents, selenium can be recovered at the end of the process as diphenyl diselenide.

Experimental Section

General Remarks: ^{1}H and ^{13}C NMR spectra were recorded with a Bruker Avance DR 200 spectrometer at 200 and 50.3 MHz, respectively; unless otherwise specified, $CDCl_3$ was used as the solvent. Chemical shifts (δ) are reported in ppm relative to TMS; *J* values are given in Hz. FT-IR spectra were recorded with a Jasco model 410 spectrometer. GC-MS analyses were obtained with an HP-6890 gas chromatograph (dimethyl silicone column, 12.5 m) equipped with an HP-5973 mass-selective detector at an ionizing voltage of 70 eV. Melting points are uncorrected. Optical rotations were measured in a 50-mm cell with a Jasco DIP-1000 digital polarimeter. Elemental analyses were carried out with a Carlo Erba 1106 elemental analyzer. Et_2O and CH_2Cl_2 (commercial grade) were used without purification. DMF, MeOH and EtOH were dried by using standard procedures. Column chromatography was performed on Merck silica gel 60 (70−230 mesh).

Preparation of Terminal Alkynes: Compounds **1a**−**c** and **1p** were commercial products. The preparation of alkynes **1h**, **1j**, **1n** and **1x** have already been described in the literature.[8] Alkyne **1d** was prepared by esterification of the corresponding acid with diazomethane and directly employed in the synthesis of **2d**. Substituted alkynes **1e**−**g**, **6a** and **6c** were obtained by conventional acylation of commercially available alkynols. Compound **1i** was prepared by alkylation of the corresponding allyl β-oxo ester[16] with propargyl bromide, and compound **6d** was obtained from the corresponding alkynol by treatment with benzyl bromide. Alkyne **1m** was obtained by a standard *O*-glycosylation procedure[17] from 2,3,4,6-tetra-*O*-acetyl-α-D-glucopyranosyl bromide. Compound **1o** was prepared by the condensation of propargylamine and *rac*-*N*-(phenylsulfonyl)-alanine with 1,2-dicyclohexylcarbodiimide. Alkynes **1p**, **1s** and **1t** were synthesized from the corresponding amines by reaction with *N*-(ethoxycarbonyl)phthalimide.[18] Substrates **1q**, **1r** and **1u**−**x** were obtained by reaction of the commercially available alkynols with phthalimide under Mitsunobu conditions.[19] Yields and physical and spectroscopic data of all new alkynes are reported below.

But-3-ynyl Benzoate (1e): Yield 70% (0.63 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.08 (t, $^4J_{H,H}$ = 2.6 Hz, 1 H, CH), 2.67 (td, $^3J_{H,H}$ = 6.8, $^4J_{H,H}$ = 2.6 Hz, 2 H, CH_2), 4.42 (t, $^3J_{H,H}$ = 6.8 Hz, 2 H, CH_2), 7.30−7.61 (m, 3 H, CH), 8.0−8.1 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 18.9, 62.4, 69.9, 79.8, 128.1 (2 C), 129.4 (2 C), 130.2, 132.8, 166.0 ppm. GC-MS: *m*/*z* (%) = 174 (3) [M^+], 122 (26), 105 (100), 77 (48), 51 (17). $C_{11}H_{10}O_2$ (174.2): calcd. C 75.84, H 5.79; found C 75.70, H 6.01.

Hex-5-ynyl Benzoate (1f): Yield 82% (0.50 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.6−1.8 (m, 2 H, CH_2) 1.82−1.96 (m, 2 H, CH_2), 2.0 (t, $^4J_{H,H}$ = 2.6 Hz, 1 H, CH), 2.28 (td, $^3J_{H,H}$ = 6.9, $^4J_{H,H}$ = 2.6 Hz, 2 H, CH_2), 4.36 (t, $^3J_{H,H}$ = 6.2 Hz, 2 H, CH_2), 7.35−7.65 (m, 3 H, CH), 8.0−8.1 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 18.1, 25.1, 27.8, 64.4, 68.8, 83.7, 128.4 (2 C), 129.6 (2 C), 130.4, 132.9, 166.6 ppm. GC-MS: *m*/*z* (%) = 202 (9) [M^+], 123 (22), 105 (100), 77 (81), 51 (25). $C_{13}H_{14}O_2$ (202.2): calcd. C 77.20, H 6.98; found C 77.34, H 7.21.

1-Methylbut-3-ynyl Acetate (1g): Yield 66% (0.42 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.35 (d, $^3J_{H,H}$ = 6.3 Hz, 3 H, CH_3), 2.01 (t, $^4J_{H,H}$ = 2.7 Hz, 1 H, CH), 2.06 (s, 3 H, CH_3), 2.43 (ddd, $^2J_{H,H}$ = 16.7, $^3J_{H,H}$ = 6.3, $^4J_{H,H}$ = 2.7 Hz, 1 H, CH_2), 2.5 (ddd, $^2J_{H,H}$ = 16.7, $^3J_{H,H}$ = 5.6, $^4J_{H,H}$ = 2.7 Hz, 1 H, CH), 5.01 (dquint, $^3J_{H,H}$ = 6.3, 5.6 Hz, 1 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 19.4, 21.6, 25.9, 68.9, 70.8, 80.8, 170.7 ppm. GC-MS: *m*/*z* (%) = 87 (57) [M − 39]$^+$, 43 (100). $C_7H_{10}O_2$ (126.1): calcd. C 66.65, H 7.99; found C 66.75, H 7.81.

Ethyl 2-Benzoyl-2-(prop-2-ynyl)pent-4-enoate (1i): Yield 62% (0.50 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.12 (t, $^3J_{H,H}$ = 7.1 Hz, 3 H, CH_3), 2.03 (d, $^4J_{H,H}$ = 2.7 Hz, 1 H, CH), 2.94 (d, $^4J_{H,H}$ = 2.7 Hz, 2 H, CH_2), 2.97 (dd, $^2J_{H,H}$ = 14.3, $^3J_{H,H}$ = 7.6 Hz, 1 H, CH_2), 3.03 (dd, $^2J_{H,H}$ = 14.3, $^3J_{H,H}$ = 7.6 Hz, 1 H, CH_2), 4.18 (m, 2 H, CH_2), 5.08- 5.14 (m, 2 H, CH_2), 5.52 (ddt, $^3J_{H,H}$ = 16.5, 10.5, 7.6 Hz, 1 H, CH), 7.40−7.64 (m, 2 H, CH), 7.52−7.58 (m, 1 H, CH), 7.82−7.87 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 14.3, 23.9, 37.4, 60.5, 62.3, 72.4, 79.2, 120.5, 128.8 (2 C), 129.0 (2 C), 131.7, 133.4, 136.0, 172.0, 195.5 ppm. GC-MS: *m*/*z* (%) = 270 (2) [M^+], 197 (6), 165(15), 105 (100), 77 (35). $C_{17}H_{18}O_3$ (270.3): calcd. C 75.53, H 6.71; found C 75.69, H 6.53.

But-3-ynyl 2,3,4,6-Tetra-*O*-acetylhexopyranoside (1m): Yield 30% (0.24 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.94 (s, 3 H, CH_3), 1.96 (s, 3 H, CH_3), 1.98 (s, 3 H, CH_3), 2.02 (s, 3 H, CH_3), 2.02 (d, $^4J_{H,H}$ = 2.6 Hz, 1 H, CH), 2.4 (td, $^3J_{H,H}$ = 6.9, $^4J_{H,H}$ = 2.6 Hz, 2 H, CH_2), 3.55−3.70 (m, 2 H, CH_2), 3.9 (ddd, $^3J_{H,H}$ = 9.5, 4.7, 2.3 Hz, 1 H, CH), 4.1 (dd, $^2J_{H,H}$ = 12.3, $^3J_{H,H}$ = 2.3 Hz, 1 H, CH_2), 4.2 (dd, $^2J_{H,H}$ = 12.3, $^3J_{H,H}$ = 4.7 Hz, 1 H, CH_2), 4.55 (d, $^3J_{H,H}$ = 7.9 Hz, 1 H, CH), 4.95 (dd, $^3J_{H,H}$ = 9.5, 7.9 Hz, 1 H, CH), 5.05 (t, $^3J_{H,H}$ = 9.5 Hz, 1 H, CH), 5.2 (t, $^3J_{H,H}$ = 9.5 Hz, 1 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 19.7, 20.4, 20.5 (3 C), 61.8, 67.7, 68.1, 71.0, 71.7, 72.6, 74.2, 80.4, 100.6, 169.2, 169.4, 170.0, 170.4 ppm. $C_{18}H_{24}O_{10}$ (400.4): calcd. C 54.00, H 6.04; found C 54.22, H 6.17.

2-(1-Methylprop-2-ynyl)-1*H*-isoindole-1,3(2*H*)-dione (1q): Yield 87% (0.88 g); m.p. 104−106 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.76 (d, $^3J_{H,H}$ = 7.2 Hz, 3 H, CH_3), 2.49 (d, $^4J_{H,H}$ = 2.5 Hz, 1 H, CH), 5.25 (qd, $^3J_{H,H}$ = 7.2, $^4J_{H,H}$ = 2.5 Hz, 1 H, CH), 7.75−7.87 (m, 2 H, CH), 7.88−7.97 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 20.0, 36.8, 71.2, 86.2, 123.4 (2 C), 131.8 (2 C), 134.1 (2 C), 166.9 (2 C) ppm. GC-MS: *m*/*z* (%) = 199 (37) [M^+], 184 (100), 130 (20), 105 (8), 76 (25). $C_{12}H_9NO_2$ (199.2): calcd. C 72.35, H 4.55, N 7.03; found C 72.53, H 4.68, N 6.90.

2-(1-Pentylprop-2-ynyl)-1*H*-isoindole-1,3(2*H*)-dione (1r): Yield 86% (2.19 g); m.p. 48−50 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.8 (t, $^3J_{H,H}$ = 6.8 Hz, 3 H, CH_3), 1.1−1.5 (m, 6 H, CH_2), 1.86−2.20 (m, 2 H, CH_2), 2.36 (d, $^4J_{H,H}$ = 2.1 Hz, 1 H, CH), 4.90−5.08 (m, 1 H, CH), 7.63−7.90 (m, 4 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.8, 22.4, 25.7, 30.8, 33.1, 41.2, 71.9, 80.2, 123.2 (2 C), 131.5 (2 C), 134.0 (2 C), 166.6 (2 C) ppm. GC-MS: *m*/*z* (%) = 226 (13) [M − 29]$^+$, 212 (13), 199 (8), 184 (100), 130 (16). FT-IR: $\tilde{\nu}$ = 2924, 2118, 1773, 1708, 1387, 1087 cm^{-1}. $C_{16}H_{17}NO_2$ (255.3): calcd. C 75.27, H 6.71, N 12.53; found C 74.49, H 6.60, N 12.31.

2-(1,1-Dimethylprop-2-ynyl)-1*H*-isoindole-1,3(2*H*)-dione (1s): Yield 70% (1.50 g); m.p. 122−124 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.95 (s, 6 H, CH_3), 2.54 (s, 1 H, CH), 7.65−7.87 (m, 4 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 29.2 (2 C), 51.9, 71.0, 85.7, 122.8 (2 C), 131.7 (2 C), 133.8 (2 C), 167.7 (2 C) ppm. GC-MS: *m*/*z* (%) = 213 (77) [M^+], 198 (100), 130

(94), 76 (30). $C_{13}H_{11}NO_2$ (213.3): calcd. C 73.20, H 5.20, N 6.57; found C 73.55, H 5.00, N 6.42.

2-(1-Ethynylcyclohexyl)-1*H*-isoindole-1,3(2*H*)-dione (1t): Yield 88% (0.67 g); m.p. 120−122 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.22−1.38 (m, 1 H, CH_2), 1.61−1.88 (m, 5 H, CH_2), 2.34−2.42 (m, 2 H, CH_2), 2.46−2.54 (m, 2 H, CH_2), 2.58 (s, 1 H, CH), 7.67−7.72 (m, 2 H, CH), 7.77−7.82 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 22.9 (2 C), 25.0, 35.9 (2 C), 58.1, 73.8, 83.4, 122.8 (2 C), 131.7 (2 C), 133.8 (2 C), 168.1 (2 C) ppm. GC-MS: *m*/*z* (%) = 253 (20) [M$^+$], 199 (23), 148 (93), 130 (100), 105 (56), 77 (37), 51 (20). $C_{16}H_{15}NO_2$ (253.3): calcd. C 75.87, H 5.97, N 5.53; found C 76.00, H 6.13, N 5.37.

2-But-3-ynyl-1*H*-isoindole-1,3(2*H*)-dione (1u): Yield 97% (0.97 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.00 (t, $^4J_{H,H}$ = 2.7 Hz, 1 H, CH), 2.62 (td, $^3J_{H,H}$ = 7.1, $^4J_{H,H}$ = 2.7 Hz, 2 H, CH_2), 3.9 (t, $^3J_{H,H}$ = 7.1 Hz, 2 H, CH_2), 7.68−7.80 (m, 2 H, CH), 7.80−7.91 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 18.2, 36.5, 70.2, 80.2, 123.3 (2 C), 131.9 (2 C), 133.9 (2 C), 167.9 (2 C) ppm. GC-MS: *m*/*z* (%) = 199 (25) [M$^+$], 160 (100), 133 (15), 105 (8), 77 (18). $C_{12}H_9NO_2$ (199.2): calcd. C 72.35, H 4.55, N 7.03; found C 72.49, H 4.40, N 6.89.

2-(1-Methylbut-3-ynyl)-1*H*-isoindole-1,3(2*H*)-dione (1v): Yield 30% (0.32 g); m.p. 55−57 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.45 (d, $^3J_{H,H}$ = 6.9 Hz, 3 H, CH_3), 1.83 (t, $^4J_{H,H}$ = 2.6 Hz, 1 H, CH), 2.69 (ddd, $^2J_{H,H}$ = 16.7, $^3J_{H,H}$ = 6.9, $^4J_{H,H}$ = 2.6 Hz, 1 H, CH_2), 2.85 (ddd, $^2J_{H,H}$ = 16.7, $^3J_{H,H}$ = 8.9, $^4J_{H,H}$ = 2.6 Hz, 1 H, CH_2), 4.48 (dquint, $^3J_{H,H}$ = 8.9, 6.9 Hz, 1 H, CH), 7.58−7.69 (m, 2 H, CH), 7.70−7.82 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 18.0, 23.6, 46.3, 70.1, 80.7, 123.2 (2 C), 131.8 (2 C), 133.9 (2 C), 167.9 (2 C) ppm. GC-MS: *m*/*z* (%) = 213 (1) [M$^+$], 174 (100), 147 (38), 130 (60), 77 (12), 50 (16). $C_{13}H_{11}NO_2$ (213.2): calcd. C 73.22, H 5.20, N 6.57; found C 73.34, H 5.32, N 6.38.

2-(Pent-4-ynyl)-1*H*-isoindole-1,3(2*H*)-dione (1w): Yield 81% (0.86 g); m.p. 87−89 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.78 (quint, $^3J_{H,H}$ = 7.1 Hz, 2 H, CH_2), 1.82 (d, $^4J_{H,H}$ = 2.6 Hz, 1 H, CH), 2.12 (td, $^3J_{H,H}$ = 7.1, $^4J_{H,H}$ = 2.6 Hz, 2 H, CH_2), 3.65 (t, $^3J_{H,H}$ = 7.1 Hz, 2 H, CH_2), 7.55−7.60 (m, 2 H, CH), 7.65−7.70 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 16.2, 27.2, 37.0, 69.0, 83.2, 123.1 (2 C), 132.0 (2 C), 133.8 (2 C), 168.2 (2 C) ppm. GC-MS: *m*/*z* (%) = 213 (6) [M$^+$], 185 (23), 160 (100), 130 (18), 105 (15), 77 (18). $C_{13}H_{11}NO_2$ (213.2): calcd. C 73.22, H 5.20, N 6.57; found C 72.97, H 5.38, N 6.70.

Prop-2-ynyl Benzoate (6a): Yield 81% (1.30 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.54 (t, $^4J_{H,H}$ = 2.4 Hz, 1 H, CH), 4.92 (d, $^4J_{H,H}$ = 2.4 Hz, 2 H, CH_2), 7.38−7.62 (m, 3 H, CH), 8.00−8.12 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 52.3, 74.9, 77.6, 128.3 (2 C), 129.7 (2 C), 132.3, 133.2, 165.6 ppm. GC-MS: *m*/*z* (%) = 160 (12) [M$^+$], 105 (100), 77 (79), 51 (3). $C_{10}H_8O_2$ (160.2): calcd. C 74.99, H 5.03; found C 75.29, H 4.91.

1-Pentylprop-2-ynyl Acetate (6c): Yield 92% (0.77 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.9 (t, $^3J_{H,H}$ = 6.6 Hz, 3 H, CH_3), 1.25−1.56 (m, 6 H, CH_2), 1.70−1.85 (m, 2 H, CH_2), 2.09 (s, 3 H, CH_3), 2.47 (d, $^4J_{H,H}$ = 1.5 Hz, 1 H, CH), 5.34 (td, $^3J_{H,H}$ = 6.6, $^4J_{H,H}$ = 1.5 Hz, 1 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.7, 20.8, 22.3, 24.4, 31.1, 34.4, 63.7, 73.2, 81.2, 169.7 ppm. GC-MS: *m*/*z* (%) = 111 (15) [M − 47]$^+$, 97 (22), 79 (26), 70 (33), 43 (100). $C_{10}H_{16}O_2$ (168.2): calcd. C 71.39, H 9.59; found C 71.58, H 9.28.

{[(1-Pentylprop-2-ynyl)oxy]methyl}benzene (6d): Yield 52% (1.11 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.89 (t, $^3J_{H,H}$ = 6.7 Hz, 3 H, CH_3), 1.2−1.5 (m, 6 H, CH_2), 1.65−1.84 (m, 2 H, CH_2), 2.65 (d, $^4J_{H,H}$ = 2.0 Hz, 1 H, CH), 4.08 (td, $^3J_{H,H}$ = 6.5, $^4J_{H,H}$ = 2.0 Hz, 1 H, CH), 4.5 (d, $^2J_{H,H}$ = 11.8 Hz, 1 H, CH_2), 4.8 (d, $^2J_{H,H}$ = 11.8 Hz, 1 H, CH_2), 7.2−7.4 (m, 5 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 14.0, 22.5, 24.8, 31.5, 35.6, 68.5, 70.5, 73.7, 83.1, 127.6, 127.9 (2 C), 128.3 (2 C), 138.0 ppm. GC-MS: *m*/*z* (%) = 216 (15) [M$^+$], 130 (5), 91 (100), 77 (14), 41 (9). $C_{15}H_{20}O$ (216.3): calcd. C 71.39, H 9.59; found C 71.50, H 9.62.

Preparation of Alkyl Phenyl Selenides: All the alkynyl phenyl selenides were prepared from the corresponding alkynes according to the method described in the literature.[10a,10b] Compounds **2a**−**c**,[10b] **2h**,[8] **2j**,[8] **2n**[7] and **2x**[7] have already been described in the literature. Compounds **7b** and **7e** were obtained by hydrolysis of **7a** and **7c** using a potassium hydroxide solution (0.1 M in methanol) and were directly employed in the subsequent reaction. Yields and physical and spectroscopic data of the new products are reported below.

Methyl 5-(Phenylseleno)pent-4-ynoate (2d): Yield 73% (0.39 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.53−2.66 (m, 2 H, CH_2), 2.71−2.83 (m, 2 H, CH_2), 3.69 (s, 3 H, CH_3), 7.20−7.35 (m, 3 H, CH), 7.45−7.54 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 16.3, 33.2, 51.6, 59.1, 102.0, 126.7, 128.1, 128.6 (2 C), 129.2 (2 C), 171.9 ppm. GC-MS: *m*/*z* (%) = 268 (53) [M$^+$], 195 (38), 163 (47), 157 (23), 115 (91), 128 (100), 77 (43), 51 (47). $C_{12}H_{12}O_2Se$ (267.2): calcd. C 53.96, H 4.53; found C 54.15, H 4.69.

4-(Phenylseleno)but-3-ynyl Benzoate (2e): Yield 66% (0.78 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 3.91 (t, $^3J_{H,H}$ = 6.7 Hz, 2 H, CH_2), 4.47 (t, $^3J_{H,H}$ = 6.7 Hz, 2 H, CH_2), 7.12−7.25 (m, 3 H, CH), 7.35−7.60 (m, 5 H, CH), 8.0−8.1 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 21.0, 62.6, 65.7, 99.7, 126.8, 128.3 (3 C), 128.7 (2 C), 129.3 (2 C), 129.6 (3 C), 132.9, 166.1 ppm. GC-MS: *m*/*z* (%) = 208 (25) [M − 122]$^+$, 128 (100), 105 (60), 70. $C_{17}H_{14}O_2Se$ (329.5): calcd. C 62.03, H 4.29; found C 61.88, H 4.42.

6-(Phenylseleno)hex-5-ynyl Benzoate (2f): Yield 83% (0.74 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$ 25 °C, TMS): δ = 1.66−1.86 (m, 2 H, CH_2), 1.87−2.05 (m, 2 H, CH_2), 2.57 (t, $^3J_{H,H}$ = 7.0 Hz, 2 H, CH_2), 4.38 (t, $^3J_{H,H}$ = 6.4 Hz, 2 H, CH_2), 7.20−7.38 (m, 3 H, CH), 7.40−7.65 (m, 5 H, CH), 8.04−8.15 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 20.3, 25.4, 28.0, 58.5, 64.5, 103.7, 126.8, 128.4 (2 C), 128.8 (2 C), 129.4 (2 C), 129.6 (3 C), 130.4, 132.9, 166.6 ppm. GC-MS: *m*/*z* (%) = 358 (5) [M$^+$], 201 (12), 157 (12), 128 (25), 105 (100), 77 (45). $C_{19}H_{18}O_2Se$ (357.3): calcd. C 63.88, H 5.08; found C 64.02, H 5.23.

1-Methyl-4-(phenylseleno)but-3-ynyl Acetate (2g): Yield 83% (0.76 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.35 (d, $^3J_{H,H}$ = 6.3 Hz, 3 H, CH_3), 2.05 (s, 3 H, CH_3), 2.72 (dd, $^2J_{H,H}$ = 16.9, $^3J_{H,H}$ = 6.3 Hz, 1 H, CH_2), 2.77 (dd, $^2J_{H,H}$ = 16.9, $^3J_{H,H}$ = 5.6 Hz, 1 H, CH), 5.07 (dquint, $^3J_{H,H}$ = 6.3, 5.6 Hz, 1 H, CH), 7.25−7.35 (m, 3 H, CH), 7.50−7.60 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 19.2, 21.2, 27.5, 60.4, 68.7, 99.4, 126.4, 126.8, 128.7 (2 C), 129.3 (2 C), 170.3 ppm. GC-MS: *m*/*z* (%) = 282 (5) [M$^+$], 222 (29), 141 (85), 115 (42), 71 (12), 43 (100). $C_{13}H_{14}O_2Se$ (281.2): calcd. C 55.54, H 5.02; found C 55.37, H 4.83.

Ethyl 2-Benzoyl-2-[3-(phenylseleno)prop-2-ynyl]pent-4-enoate (2i): Yield 50% (0.38 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS):

δ = 1.10 (t, $^3J_{H,H}$ = 7.1 Hz, 3 H, CH_3), 2.95−3.10 (m, 2 H, CH_2), 3.23 (s, 2 H, CH_2), 4.18 (q, $^3J_{H,H}$ = 7.1 Hz, 2 H, CH_2), 5.05−5.10 (m, 2 H, CH_2), 5.50−5.60 (m, 1 H, CH), 7.20−7.30 (m, 3 H, CH), 7.35−7.50 (m, 4 H, CH), 7.52−7.60 (m, 1 H, CH), 7.80−7.90 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 14.3, 26.0, 37.8, 60.7, 62.3, 62.7, 98.9, 120.6, 127.3, 128.8 (2 C), 129.1 (2 C), 129.3 (3 C), 129.8 (2 C), 131.8, 133.4, 136.0, 172.0, 195.4 ppm. GC-MS: *m*/*z* (%) = 426 (1) [M^+], 353 (6), 269 (5), 195 (12), 157 (3), 105 (100), 77 (40). $C_{23}H_{22}O_3Se$ (425.4): calcd. C 64.95, H 5.21; found C 64.78, H 5.37.

4-(Phenylseleno)but-3-ynyl 2,3,4,6-Tetra-*O*-acetyl-β-D-glucopyranoside (2m): Yield 65% (0.36 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.96 (s, 3 H, CH_3), 1.98 (s, 3 H, CH_3), 2.00 (s, 3 H, CH_3), 2.05 (s, 3 H, CH_3), 2.70 (t, $^3J_{H,H}$ = 6.8 Hz, 2 H, CH_2), 3.65−3.75 (m, 2 H, CH_2), 3.95 (ddd, $^3J_{H,H}$ = 9.5, 4.7, 2.3 Hz, 1 H, CH), 4.10 (dd, $^2J_{H,H}$ = 12.3, $^3J_{H,H}$ = 2.3 Hz, 1 H, CH_2), 4.25 (dd, $^2J_{H,H}$ = 12.3, $^3J_{H,H}$ = 4.7 Hz, 1 H, CH_2), 4.57 (d, $^3J_{H,H}$ = 7.9 Hz, 1 H, CH), 4.96 (dd, $^3J_{H,H}$ = 9.5, 7.9 Hz, 1 H, CH), 5.05 (t, $^3J_{H,H}$ = 9.5 Hz, 1 H, CH), 5.18 (t, $^3J_{H,H}$ = 9.5 Hz, 1 H, CH), 7.15−7.30 (m, 3 H, CH), 7.45−7.50 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 21.0 (2 C), 21.1 (2 C), 22.3, 62.2, 68.3, 68.6, 68.7, 71.5, 72.2, 73.1, 80.4, 100.6, 127.4, 127.5, 129.3 (2 C), 129.9 (2 C), 169.7 (2 C), 170.6, 171.0 ppm. $C_{24}H_{28}O_{10}Se$ (555.4): calcd. C 51.90, H 5.08; found C 52.11, H 5.31.

***N*-[3-(Phenylseleno)prop-2-ynyl]-2-[(phenylsulfonyl)amino]-propanamide (2o):** Yield 48% (0.40 g); oil, ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.25 (d, $^3J_{H,H}$ = 7.1 Hz, 3 H, CH_3), 3.93 (dq, $^3J_{H,H}$ = 8.0, 7.1 Hz, 1 H, CH), 3.98−4.27 (m, 2 H, CH_2), 6.69 (d, $^3J_{H,H}$ = 8.0 Hz, 1 H, NH), 7.19−7.35 (m, 3 H, CH), 7.38−7.58 (m, 6 H, CH−NH), 7.84−7.92 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 18.7, 30.5, 52.5, 63.0, 98.7, 126.8 (2 C), 126.9, 128.8 (2 C), 128.9 (2 C), 129.3 (2 C), 132.6 (2 C), 139.6, 171.5 ppm. $C_{18}H_{18}N_2O_3SSe$ (421.4): calcd. C 51.32, H 4.31, N 6.65; found C 51.47, H 4.19; N 6.25.

2-[3-(Phenylseleno)prop-2-ynyl]-1*H*-isoindole-1,3(2*H*)-dione (2p): Yield 88% (0.90 g); m.p. 104−106 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 4.68 (s, 2 H, CH_2), 7.15−7.34 (m, 3 H, CH), 7.40−7.52 (m, 2 H, CH), 7.60−7.75 (m, 2 H, CH), 7.76−7.90 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 28.3, 63.6, 97.2, 123.2 (2 C), 126.9, 127.8, 128.8 (2 C), 129.3 (2 C), 131.8 (2 C), 133.9 (2 C), 166.7 (2 C) ppm. GC-MS: *m*/*z* (%) = 341 (37) [M^+], 264 (71), 194 (70), 184 (100), 160 (25), 130 (38), 105 (19), 77 (36), 51 (21). $C_{17}H_{11}NO_2Se$ (340.2): calcd. C 60.02, H 3.26, N 4.12; found C 60.21, H 3.40, N 4.01.

2-[1-Methyl-3-(phenylseleno)prop-2-ynyl]-1*H*-isoindole-1,3(2*H*)-dione (2q): Yield 68% (1.00 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.85 (d, $^3J_{H,H}$ = 7.2 Hz, 3 H, CH_3), 5.48 (q, $^3J_{H,H}$ = 7.2 Hz, 1 H, CH), 7.20−7.45 (m, 3 H, CH), 7.50−7.68 (m, 2 H, CH), 7.70−7.81 (m, 2 H, CH), 7.82−7.95 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 20.4, 38.4, 74.6, 101.3, 123.3 (2 C), 126.1, 127.0, 128.8 (2 C), 129.6 (2 C), 131.8 (2 C), 134.1 (2 C), 166.8 (2 C) ppm. GC-MS: *m*/*z* (%) = 355 (11) [M^+], 278 (16), 198 (100), 130 (60), 105 (19), 77 (24). $C_{18}H_{13}NO_2Se$ (354.3): calcd. C 61.04, H 3.70, N 3.95; found C 60.99, H 3.62, N 4.02.

2-[1-Pentyl-3-(phenylseleno)prop-2-ynyl]-1*H*-isoindole-1,3(2*H*)-dione (2r): Yield 86% (0.87 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.86 (t, $^3J_{H,H}$ = 6.8 Hz, 3 H, CH_3), 1.11−1.60 (m, 6 H, CH_2), 2.01−2.36 (m, 2 H, CH_2), 5.25 (t, $^3J_{H,H}$ = 7.05 Hz, 1 H, CH), 7.20−7.40 (m, 3 H, CH), 7.50−7.60 (m, 2 H, CH), 7.65−7.80 (m, 2 H, CH), 7.87−7.90 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.9, 22.3, 25.9, 30.9, 33.5, 41.4, 63.5, 100.4, 123.3 (2 C), 126.8, 128.4, 128.6 (2 C), 129.4 (2 C), 131.7 (2 C), 134.0 (2 C), 166.9 (2 C) ppm. FT-IR: $\tilde{\nu}$ = 2928, 2178, 1774, 1715, 1472, 1382 cm^{-1}. GC-MS: *m*/*z* (%) = 411 (5) [M^+], 340 (12), 254 (39), 207 (100). $C_{22}H_{21}NO_2Se$ (410.4): calcd. C 64.39, H 5.16, N 3.41; found C 64.59, H 5.22, N 3.71.

2-[1,1-Dimethyl-3-(phenylseleno)prop-2-ynyl]-1*H*-isoindole-1,3(2*H*)-dione (2s): Yield 86% (0.63 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.08 (s, 6 H, CH_3), 7.17−7.38 (m, 3 H, CH), 7.50−7.70 (m, 4 H, CH), 7.71−7.85 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 29.2 (2 C), 53.2, 62.9, 105.7, 122.7 (2 C), 126.6, 128.4 (2 C), 128.8, 129.3 (2 C), 131.7 (2 C), 133.7 (2 C), 167.6 (2 C) ppm. FT-IR: $\tilde{\nu}$ = 2989, 2172, 1713, 1366, 1067 cm^{-1}. GC-MS: *m*/*z* (%) = 369 (54) [M^+], 274 (100), 188 (44), 130 (82), 104 (60), 76 (41). $C_{19}H_{15}NO_2Se$ (368.3): calcd. C 61.97, H 4.11, N 3.80; found C 61.73, H 4.23, N 3.53.

2-{1-[(Phenylseleno)ethynyl]cyclohexyl}-1*H*-isoindole-1,3(2*H*)-dione (2t): Yield 87% (0.77 g); m.p. 105−108 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.25−1.45 (m, 1 H, CH_2), 1.65−1.95 (m, 5 H, CH_2), 2.35−2.70 (m, 4 H, CH_2), 7.20−7.40 (m, 3 H, CH), 7.60−7.65 (m, 2 H, CH), 7.70−7.77 (m, 2 H, CH), 7.80−7.85 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 23.5 (2 C), 25.6, 36.4 (2 C), 59.9, 65.8, 104.2, 123.2 (2 C), 127.2, 129.0 (2 C), 129.5, 129.9 (2 C), 132.2 (2 C), 134.3 (2 C), 168.6 (2 C) ppm. GC-MS: *m*/*z* (%) = 409 (10) [M^+], 224 (22), 198 (17), 181 (16), 157 (6), 148 (28), 130 (37), 105 (100), 77 (24). $C_{22}H_{19}NO_2Se$ (408.3): calcd. C 64.72, H 4.69, N 3.43; found C 64.58, H 4.816, N 3.29.

2-[4-(Phenylseleno)but-3-ynyl]-1*H*-isoindole-1,3(2*H*)-dione (2u): Yield 70% (0.75 g); m.p. 72−74 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.88 (t, $^3J_{H,H}$ = 7.1 Hz, 2 H, CH_2), 3.92 (t, $^3J_{H,H}$ = 7.1 Hz, 2 H, CH_2), 7.10−7.27 (m, 3 H, CH), 7.35−7.50 (m, 2 H, CH), 7.53−7.75 (m, 2 H, CH), 7.76−7.89 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 20.2, 36.5, 70.2, 99.7, 123.1 (2 C), 126.6, 126.7, 128.8 (2 C), 129.2 (2 C), 131.8 (2 C), 133.8 (2 C), 167.7 (2 C) ppm. GC-MS: *m*/*z* (%) = 355 (18) [M^+], 208 (43), 198 (52), 160 (100), 128 (92), 105 (16), 77 (35). $C_{18}H_{13}NO_2Se$ (354.3): calcd. C 61.04, H 3.70, N 3.95; found C 61.23, H 3.86, N 3.68.

2-[1-Methyl-4-(phenylseleno)but-3-ynyl]-1*H*-isoindole-1,3(2*H*)-dione (2v): Yield 66% (0.49 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.48 (d, $^3J_{H,H}$ = 6.9 Hz, 3 H, CH_3), 2.86 (dd, $^2J_{H,H}$ = 16.9, $^3J_{H,H}$ = 6.9 Hz, 1 H, CH_2), 3.14 (dd, $^2J_{H,H}$ = 16.9, $^3J_{H,H}$ = 8.8 Hz, 1 H, CH_2), 4.55 (dquint, $^3J_{H,H}$ = 8.8, 6.9 Hz, 1 H, CH), 7.00−7.15 (m, 3 H, CH), 7.20−7.30 (m, 2 H, CH), 7.55−7.65 (m, 2 H, CH), 7.66−7.76 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 18.1, 25.7, 46.5, 60.0, 100.4, 123.1 (2 C), 126.7, 126.9, 128.7 (2 C), 129.3 (2 C), 131.8 (2 C), 133.9 (2 C), 168.1 (2 C) ppm. GC-MS: *m*/*z* (%) = 369 (6) [M^+], 212 (14), 174 (100), 130 (30), 115 (12). $C_{19}H_{15}NO_2Se$ (368.3): calcd. C 61.97, H 4.11, N 3.80; found C 62.04, H 4.08, N 3.55.

2-[5-(Phenylseleno)pent-4-ynyl]-1*H*-isoindole-1,3(2*H*)-dione (2w): Yield 84% (0.62 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.92 (tt, $^3J_{H,H}$ = 7.1, 6.8 Hz, 2 H, CH_2), 2.46 (t, $^3J_{H,H}$ = 7.1 Hz, 2 H, CH_2), 3.75 (t, $^3J_{H,H}$ = 6.8 Hz, 2 H, CH_2), 7.10−7.30 (m, 3 H, CH), 7.35−7.45 (m, 2 H, CH), 7.55−7.65 (m, 2 H, CH), 7.66−7.80 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 18.5, 27.4, 37.7, 69.0, 102.9, 123.2 (2 C), 126.7, 128.6 (2 C), 129.0, 129.4 (2 C), 132.1 (2 C), 133.9 (2 C), 168.3 (2 C) ppm. GC-MS: *m*/*z* (%) = 369 (12) [M^+], 212 (82), 160 (100), 141 (20),

115 (24), 77 (24). $C_{19}H_{15}NO_2Se$ (368.3): calcd. C 61.97, H 4.22, N 3.80; found C 62.13, H 4.03, N 3.66.

3-(Phenylseleno)prop-2-ynyl Benzoate (7a): Yield 95% (0.60 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 5.14 (s, 2 H, CH_2), 7.12−7.66 (m, 8 H, CH), 7.95−8.18 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 53.5, 68.1, 97.9, 127.2, 127.8, 128.3 (2 C), 129.2 (2 C), 129.5 (2 C), 129.7 (2 C), 132.3, 133.1, 165.7 ppm. GC-MS: *m*/*z* (%) = 316 (10) [M$^+$], 193 (33), 157 (3), 105 (100), 77 (39), 51 (14). $C_{16}H_{12}O_2Se$ (315.2): calcd. C 60.98, H 3.84; found C 61.16, H 3.99.

1-Pentyl-3-(phenylseleno)prop-2-ynyl Acetate (7c): Yield 98% (0.64 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.9 (t, $^3J_{H,H}$ = 6.7 Hz, 3 H, CH_3), 1.22−1.58 (m, 6 H, CH_2), 1.75−1.90 (m, 2 H, CH_2), 2.09 (s, 3 H, CH_3), 5.50 (t, $^3J_{H,H}$ = 6.6 Hz, 1 H, CH), 7.20−7.38 (m, 3 H, CH), 7.44−7.55 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.9, 20.9, 22.4, 24.7, 31.2, 34.6, 64.9, 66.1, 101.5, 126.9, 127.1, 128.9 (2 C), 129.4 (2 C), 169.9 ppm. GC-MS: *m*/*z* (%) = 324 (11) [M$^+$], 183 (10), 157 (9), 125 (64), 71 (17), 43 (100). $C_{16}H_{20}O_2Se$ (323.3): calcd. C 59.45, H 6.24; found C 59.59, H 6.44.

{[3-(Benzyloxy)oct-1-ynyl]seleno}benzene (7d): Yield 96% (0.72 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.88 (t, $^3J_{H,H}$ = 6.6 Hz, 3 H, CH_3), 1.15−1.60 (m, 6 H, CH_2), 1.75−1.90 (m, 2 H, CH_2), 4.29 (t, $^3J_{H,H}$ = 6.4 Hz, 1 H, CH), 4.52 (d, $^2J_{H,H}$ = 11.8 Hz, 1 H, CH_2), 4.83 (d, $^2J_{H,H}$ = 11.8 Hz, 1 H, CH_2), 7.2−7.4 (m, 8 H, CH), 7.48−7.58 (m, 2 H) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 14.0, 22.5, 25.0, 31.4, 35.7, 65.5, 69.7, 70.5, 103.2, 127.0 (2 C), 127.6 (2 C), 127.9 (2 C), 128.3 (2 C), 128.9, 129.4 (2 C), 137.9 ppm. GC-MS: *m*/*z* (%) = 372 (1) [M$^+$], 221 (11), 129 (16), 105 (19), 91 (100), 77 (21), 67 (18). $C_{21}H_{24}OSe$ (371.4): calcd. C 67.98, H 6.51; found C 67.85, H 6.71.

Preparation of Alkynes 2k and 2l:[20] *n*BuLi (5.82 mL of a solution 1.6 M in hexane) was added to a solution of (trimethylsilyl)acetylene or 1-(triisopropylsilyl)-1-propyne (9.86 mmol) in dry tetrahydrofuran (20 mL) at −20 °C. After stirring for 30 min, hexamethylphosphoramide (4 mL) and benzyl (*S*)-(+)-glycidyl ether (5.48 mmol) were added. The mixture was allowed to slowly reach room temperature and tetrabutylammonium fluoride (2.74 mmol) was added. The progress of the reaction (12−18 h) was monitored by TLC. The reaction mixture was quenched with ammonium chloride solution (20 mL) and extracted with diethyl ether. The combined organic layers were dried with sodium sulfate and the solvents evaporated. The reaction product was purified by column chromatography on silica gel using a mixture of diethyl ether/light petroleum (4:6) as eluent. The two alkynes were isolated in 75% yields and then treated with AcCl in a solution of pyridine/dichloromethane (1:3) at 0 °C. After the usual workup, compounds **1k** and **1l** were obtained in pure form. These were then employed in the synthesis of **2k** and **2l**, respectively, according to the method described above. Yields and physical and spectroscopic data are reported below.

(1*S*)-1-[(Benzyloxy)methyl]-4-(phenylseleno)but-3-ynyl Acetate (2k): Yield 62% (0.62 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.00 (s, 3 H, CH_3), 2.76 (dd, $^2J_{H,H}$ = 17.0, $^3J_{H,H}$ = 5.8 Hz, 1 H, CH_2), 2.84 (dd, $^2J_{H,H}$ = 17.0, $^3J_{H,H}$ = 6.6 Hz, 1 H, CH_2), 3.61 (dd, $^2J_{H,H}$ = 10.5, $^3J_{H,H}$ = 4.6 Hz, 1 H, CH_2), 3.65 (dd, $^2J_{H,H}$ = 10.5, $^3J_{H,H}$ = 5.1 Hz, 1 H, CH_2), 4.48 (d, $^2J_{H,H}$ = 12.1 Hz, 1 H, CH_2), 4.53 (d, $^2J_{H,H}$ = 12.1 Hz, 1 H, CH_2), 5.08 (dddd, $^3J_{H,H}$ = 6.6, 5.8, 5.1, 4.6 Hz, 1 H, CH), 7.15−7.30 (m, 8 H, CH), 7.40−7.48 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 21.0, 22.8, 60.6, 69.6, 70.6, 73.3, 99.0, 126.9 (2 C), 127.6 (2 C), 127.7 (2 C), 128.4 (2 C), 128.8 (2 C), 129.4 (2 C), 137.8, 170.3. $C_{20}H_{20}O_3Se$ (387.3): calcd. C 62.03, H 5.21; found C 62.21, H 5.49.

(1*S*)-1-[(Benzyloxy)methyl]-5-(phenylseleno)pent-4-ynyl Acetate (2l): Yield 72% (0.42 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.94 (dt, $^3J_{H,H}$ = 8.5, 7.3 Hz, 2 H, CH_2), 2.06 (s, 3 H, CH_3), 2.50 (d, $^3J_{H,H}$ = 7.3 Hz, 2 H, CH_2), 3.54 (d, $^3J_{H,H}$ = 4.8 Hz, 2 H, CH_2), 4.48 (d, $^2J_{H,H}$ = 10.9 Hz, 1 H, CH_2), 4.58 (d, $^2J_{H,H}$ = 10.9 Hz, 1 H, CH_2), 5.19 (tt, $^3J_{H,H}$ = 8.5, 4.8 Hz, 1 H, CH), 7.10−7.35 (m, 8 H, CH), 7.45−7.55 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 16.8, 21.0, 29.9, 58.5, 70.6, 71.5, 73.1, 102.8, 126.8, 127.5, 127.6 (2 C), 128.3, 128.7 (2 C), 128.8, 128.9, 129.3 (2 C), 137.8, 170.5. $C_{21}H_{22}O_3Se$ (401.4): calcd. C 62.85, H 5.53; found C 63.02, H 5.71.

Synthesis of *Se*-Phenyl Selenocarboxylates. General Procedure: (Phenylseleno)acetylenes **2** (1 mmol) were dissolved in dichloromethane (15 mL) and *p*-toluenesulfonic acid monohydrate (2 mmol) was added. The resulting suspension was heated at 40 or 60 °C. The progress of the reaction was monitored by TLC. Solid K_2CO_3 was added and then the mixture was filtered. The filtrate was dried, concentrated and purified by column chromatography on silica gel using a mixture of light petroleum and diethyl ether as eluent. Compounds **4a**, **4b** and **4c** have already been described in the literature.[5e] Yields and physical and spectroscopic data of all the other compounds are reported below.

Methyl 5-Oxo-5-(phenylseleno)pentanoate (4d): Yield 67% (0.27 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.98 (quint, $^3J_{H,H}$ = 7.2 Hz, 2 H, CH_2), 2.38 (t, $^3J_{H,H}$ = 7.2 Hz, 2 H, CH_2), 2.76 (t, $^3J_{H,H}$ = 7.2 Hz, 2 H, CH_2), 3.65 (s, 3 H, CH_3), 7.40−7.52 (m, 5 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 20.2, 32.4, 46.1, 51.3, 126.1, 128.6, 129.1 (2 C), 135.5 (2 C), 172.7, 199.1 ppm. GC-MS: *m*/*z* (%) = 255 (4) [M − 31]$^+$, 157 (13), 129 (100), 101 (33), 69 (50). $C_{12}H_{14}O_3Se$ (285.2): calcd. C 50.55, H 4.95; found C 50.76, H 4.77.

4-Oxo-4-(phenylseleno)butyl Benzoate (4e): Yield 75% (0.39 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.21 (tt, $^3J_{H,H}$ = 7.2, 6.2 Hz, 2 H, CH_2), 2.94 (t, $^3J_{H,H}$ = 6.2 Hz, 2 H, CH_2), 4.42 (t, $^3J_{H,H}$ = 6.2 Hz, 2 H, CH_2), 7.35−7.71 (m, 8 H, CH), 8.05−8.20 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 28.5, 44.9, 64.1, 126.7, 128.8 (2 C), 129.4, 129.8 (2 C), 130.0 (2 C), 130.5, 133.4, 136.2 (2 C), 166.8, 199.9 ppm. GC-MS: *m*/*z* (%) = 191 (3) [M − 157]$^+$, 105 (100), 77 (17). $C_{17}H_{16}O_3Se$ (347.3): calcd. C 58.81, H 4.64; found C 58.95, H 4.87.

6-Oxo-6-(phenylseleno)hexyl Benzoate (4f): Yield 74% (0.28 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.35−1.53 (m, 2 H, CH_2), 1.60−1.80 (m, 4 H, CH_2), 2.65 (t, $^3J_{H,H}$ = 7.2 Hz, 2 H, CH_2), 4.23 (t, $^3J_{H,H}$ = 6.4 Hz, 2 H, CH_2), 7.25−7.55 (m, 8 H, CH), 7.90−8.05 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 25.1, 25.4, 28.5, 47.3, 64.7, 126.9, 128.4 (2 C), 128.9, 129.4 (2 C), 129.6 (2 C), 130.4, 132.9, 135.8 (2 C), 166.6, 200.1 ppm. GC-MS: *m*/*z* (%) = 219 (4) [M − 157]$^+$, 105 (100), 77 (14): $C_{19}H_{20}O_3Se$ (375.3): calcd. C 60.81, H 5.37; found C 60.95, H 5.55.

1-Methyl-4-oxo-4-(phenylseleno)butyl Acetate (4g): Yield 76% (0.60 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.25 (d, $^3J_{H,H}$ = 6.3 Hz, 3 H, CH_3), 1.95 (td, $^3J_{H,H}$ = 7.5, 6.3 Hz, 2 H, CH_2), 2.01 (s, 3 H, CH_3), 2.78 (t, $^3J_{H,H}$ = 7.5 Hz, 2 H, CH_2), 4.95 (sext, $^3J_{H,H}$ = 6.3 Hz, 1 H, CH), 7.35−7.45 (m, 3 H, CH), 7.50−7.60 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 19.8, 21.2, 30.9, 43.4, 69.6, 126.2, 128.9, 129.3 (2 C), 135.7 (2 C), 170.5, 199.5 ppm. GC-MS: *m*/*z* (%) = 199 (1) [M − 101]$^+$, 157 (15), 143 (34), 101 (100), 83 (27), 43 (76). $C_{13}H_{16}O_3Se$ (299.2): calcd. C 52.19, H 5.39; found C 52.00, H 5.58.

1-[3-Oxo-3-(phenylseleno)propyl]undecyl Acetate (4h): Yield 66% (0.56 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.89 (t, $^3J_{H,H}$ = 6.7 Hz, 3 H, CH_3), 1.18−1.38 (m, 16 H, CH_2), 1.40−1.64 (m, 2 H, CH_2), 1.75−2.1 (m, 2 H, CH_2), 2.02 (s, 3 H, CH_3), 2.72 (t, $^3J_{H,H}$ = 7.5 Hz, 2 H, CH_2), 4.88 (tt, $^3J_{H,H}$ = 7.4, 5.0 Hz, 1 H, CH), 7.30−7.40 (m, 3 H, CH), 7.46−7.55 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.9, 20.9, 22.5, 25.1, 29.2 (3 C), 29.3 (3 C), 31.7, 33.9, 43.3, 72.8, 126.3, 128.7, 129.2 (2 C), 135.6 (2 C), 170.3, 199.1 ppm. GC-MS: *m*/*z* (%) = 269 (6) [M − 157]$^+$, 227 (100), 191 (11), 157 (12), 97 (16), 43 (50). $C_{22}H_{34}O_3Se$ (425.5): calcd. C 62.12, H 8.06; found C 62.44, H 7.89.

Ethyl 2-Benzoyl-2-[3-oxo-3-(phenylseleno)propyl]pent-4-enoate (4i): Yield 74% (0.20 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.10 (t, $^3J_{H,H}$ = 7.1 Hz, 3 H, CH_3), 2.42 (ddd, $^2J_{H,H}$ = 14.3, $^3J_{H,H}$ = 9.8, 3.8 Hz, 1 H, CH_2), 2.45−2.65 (m, 2 H, CH_2), 2.73 (ddd, $^2J_{H,H}$ = 14.3, $^3J_{H,H}$ = 9.8, 3.3 Hz, 1 H, CH_2), 2.75−2.87 (m, 2 H, CH_2), 4.15 (q, $^3J_{H,H}$ = 7.1 Hz, 2 H, CH_2), 5.05−5.15 (m, 2 H, CH_2), 5.60 (ddt, $^3J_{H,H}$ = 17.0, 9.9, 7.5 Hz, 1 H, CH_2), 7.34−7.40 (m, 3 H, CH), 7.41−7.51 (m, 4 H, CH), 7.53−7.59 (m, 1 H, CH), 7.82−7.87 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.8, 28.0, 37.6, 42.0, 59.6, 61.1, 119.7, 126.1, 128.3 (2 C), 128.6 (3 C), 128.8 (2 C), 129.3, 131.4, 132.9, 135.5, 135.7, 172.4, 195.9, 199.0 ppm. GC-MS: *m*/*z* (%) = 287 (26) [M − 157]$^+$, 241 (6), 157 (4), 105 (100), 77 (22), 55 (5). $C_{23}H_{24}O_4Se$ (443.4): calcd. C 62.31, H 5.46; found C 62.12, H 5.60.

1-(Butoxymethyl)-4-oxo-4-(phenylseleno)butyl Acetate (4j): Yield 56% (0.21 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.91 (t, $^3J_{H,H}$ = 7.2 Hz, 3 H, CH_3), 1.20−1.62 (m, 4 H, CH_2), 2.08 (s, 3 H, CH_3), 1.90−2.20 (m, 2 H, CH_2), 2.77 (t, $^3J_{H,H}$ = 7.5 Hz, 2 H, CH_2), 3.30−3.58 (m, 4 H, CH_2), 5.02 (dq, $^3J_{H,H}$ = 7.4, 5.0 Hz, 1 H, CH), 7.20−7.39 (m, 3 H, CH), 7.40−7.55 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.8, 19.1, 20.9, 26.3, 31.5, 43.2, 70.8, 71.3 (2 C), 127.8, 128.8, 129.2 (2 C), 135.7 (2 C), 170.1, 199.3. $C_{17}H_{24}O_4Se$ (371.3): calcd. C 55.00, H 6.52; found C 55.21, H 6.89.

(1*S*)-1-[(Benzyloxy)methyl]-4-oxo-4-(phenylseleno)butyl Acetate (4k): Yield 51% (0.31 g); oil. $[\alpha]_D^{25}$ = −2.82 (*c* = 3.94 in $CHCl_3$). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.00 (s, 3 H, CH_3), 1.90−2.04 (m, 2 H, CH_2), 2.70 (t, $^3J_{H,H}$ = 7.5 Hz, 2 H, CH_2), 3.44 (dd, $^2J_{H,H}$ = 10.5, $^3J_{H,H}$ = 4.7 Hz, 1 H, CH_2), 3.47 (dd, $^2J_{H,H}$ = 10.5, $^3J_{H,H}$ = 5.1 Hz, 1 H, CH_2), 4.44 (d, $^2J_{H,H}$ = 12.1 Hz, 1 H, CH_2), 4.50 (d, $^2J_{H,H}$ = 12.1 Hz, 1 H, CH_2), 4.94−5.05 (m, 1 H, CH), 7.15−7.35 (m, 8 H, CH), 7.40−7.50 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 21.0, 26.3, 43.2, 70.5, 71.3, 73.1, 126.2, 127.6, 127.7, 128.4, 128.6, 128.8, 128.9, 129.3 (2 C), 129.7, 135.7 (2 C), 137.7, 170.4. $C_{20}H_{22}O_4Se$ (405.3): calcd. C 59.27, H 5.47; found C 59.46, H 5.61.

(1*S*)-1-[(Benzyloxy)methyl]-5-oxo-5-(phenylseleno)pentyl Acetate (4l): Yield 60% (0.25 g); oil. $[\alpha]_D^{20}$ = −3.07 (*c* = 1.50 in $CHCl_3$). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.58−1.80 (m, 4 H, CH_2), 2.08 (s, 3 H, CH_3), 2.64−2.78 (m, 2 H, CH_2), 3.50 (d, $^3J_{H,H}$ = 8.3 Hz, 2 H, CH_2), 4.45 (d, $^2J_{H,H}$ = 12.1 Hz, 1 H, CH_2), 4.55 (d, $^2J_{H,H}$ = 12.1 Hz, 1 H, CH_2), 4.95−5.10 (m, 1 H, CH), 7.25−7.45 (m, 8 H, CH), 7.35−7.55 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 20.1, 21.1, 29.9, 46.9, 70.7, 71.9, 73.1, 126.3, 127.5, 127.6 (2 C), 128.3 (2 C), 128.8, 129.3 (2 C), 135.7 (2 C), 137.9, 170.6, 199.8. $C_{21}H_{24}O_4Se$ (419.4): calcd. C 60.15, H 5.77; found C 60.01, H 5.92.

***Se*-Phenyl 4-[(2,3,4,6-Tetra-*O*-acetyl-β-D-glucopyranosyl)oxy]-butaneselenoate (4m):** Yield 62% (0.23 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.0 (tdd, $^3J_{H,H}$ = 7.1, 6.4, 5.7 Hz, 2 H, CH_2), 2.01 (s, 3 H, CH_3), 2.03 (s, 3 H, CH_3), 2.07 (s, 3 H, CH_3), 2.08 (s, 3 H, CH_3), 2.80 (t, $^3J_{H,H}$ = 7.1 Hz, 2 H, CH_2), 3.58 (dt, $^2J_{H,H}$ = 9.9, $^3J_{H,H}$ = 6.4 Hz, 1 H, CH_2), 3.70 (ddd, $^3J_{H,H}$ = 9.6, 4.8, 2.3 Hz, 1 H, CH), 3.90 (dt, $^2J_{H,H}$ = 9.9, $^3J_{H,H}$ = 5.7 Hz, 1 H, CH_2), 4.13 (dd, $^2J_{H,H}$ = 12.3, $^3J_{H,H}$ = 2.3 Hz, 1 H, CH_2), 4.25 (dd, $^2J_{H,H}$ = 12.3, $^3J_{H,H}$ = 4.8 Hz, 1 H, CH_2), 4.50 (d, $^3J_{H,H}$ = 8.0 Hz, 1 H, CH), 4.98 (dd, $^3J_{H,H}$ = 9.6, 8.0 Hz, 1 H, CH), 5.18 (t, $^3J_{H,H}$ = 9.6 Hz, 1 H, CH), 5.20 (t, $^3J_{H,H}$ = 9.6 Hz, 1 H, CH), 7.35−7.44 (m, 3 H, CH), 7.45−7.55 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 20.5 (2 C), 20.6 (2 C), 25.0, 43.5, 61.9, 67.8, 68.2, 71.2, 71.7, 72.7, 100.6, 128.5, 128.9, 129.3 (2 C), 135.7 (2 C), 169.3 (2 C), 170.2, 170.6, 199.7. $C_{24}H_{30}O_{11}Se$ (573.4): calcd. C 50.27, H 5.27; found C 50.03, H 5.44.

***Se*-Phenyl 3-[(Phenylsulfonyl)amino]propaneselenoate (4n):** Yield 78% (0.33 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.90 (t, $^3J_{H,H}$ = 6.0 Hz, 2 H, CH_2), 3.20 (t, $^3J_{H,H}$ = 6.0 Hz, 2 H, CH_2), 5.22 (br. s, 1 H, NH), 7.15−7.65 (m, 8 H, CH), 7.74−7.93 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 38.7, 46.8, 126.2, 126.9 (2 C), 129.1 (2 C), 129.4 (2 C), 132.7 (2 C), 135.7 (2 C), 139.7, 199.9. FT-IR: $\tilde{\nu}$ = 3290, 1713, 1328, 1162 cm^{-1}. GC-MS: *m*/*z* (%) = 368 (3) [M$^+$], 213 (51), 185 (72), 157 (100), 91 (15), 51 (15). $C_{15}H_{15}NO_3SSe$ (368.3): calcd. C 48.93, H 4.11, N 3.80; found C 49.15, H 4.23, N 3.55.

***Se*-Phenyl 3-({2-[(Phenylsulfonyl)amino]propanoyl}amino)propaneselenoate (4o):** Yield 51% (0.23 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.22 (d, $^3J_{H,H}$ = 7.1 Hz, 3 H, CH_3), 2.83 (t, $^3J_{H,H}$ = 5.6 Hz, 2 H, CH_2), 3.41 (td, $^3J_{H,H}$ = 5.6, 4.5 Hz, 2 H, CH_2), 3.79 (dq, $^3J_{H,H}$ = 7.5, 7.1 Hz, 1 H, CH), 6.08 (d, $^3J_{H,H}$ = 7.5 Hz, 1 H, NH), 6.92 (t, $^3J_{H,H}$ = 4.5 Hz, 1 H, NH), 7.20−7.66 (m, 8 H, CH), 7.80−7.94 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 19.1, 35.2, 46.5, 52.6, 125.9, 127.1 (2 C), 129.1 (3 C), 129.4, 132.8 (2 C), 135.7 (2 C), 139.7, 171.7, 195.1. $C_{18}H_{20}N_2O_4SSe$ (439.4): calcd. C 49.21, H 4.59, N 6.38; found C 49.39, H 4.41, N 4.33.

***Se*-Phenyl 3-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)propaneselenoate (4p):** Yield 95% (0.68 g); m.p. 90−93 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 3.10 (t, $^3J_{H,H}$ = 7.0 Hz, 2 H, CH_2), 3.99 (t, $^3J_{H,H}$ = 7.0 Hz, 2 H, CH_2), 7.25−7.40 (m, 3 H, CH), 7.40−7.54 (m, 2 H, CH), 7.57−7.63 (m, 2 H, CH), 7.44−7.88 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 33.6, 44.9, 123.0 (2 C), 128.4, 128.7, 129.1 (2 C), 131.7 (2 C), 133.8 (2 C), 135.4 (2 C), 167.4 (2 C), 197.0. FT-IR: $\tilde{\nu}$ = 3030, 1775, 1716, 1396 cm^{-1}. GC-MS: *m*/*z* (%) = 202 (65) [M − 157]$^+$, 160 (100), 77 (11). $C_{17}H_{13}NO_3Se$ (358.2): calcd. C 57.00, H 3.66, N 3.91; found C 57.19, H 3.80, N 3.68.

***Se*-Phenyl 3-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)butaneselenoate (4q):** Yield 72% (0.27 g); m.p. 76−78 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.55 (d, $^3J_{H,H}$ = 7.0 Hz, 3 H, CH_3), 3.24 (dd, $^2J_{H,H}$ = 16.0, $^3J_{H,H}$ = 5.7 Hz, 1 H, CH_2), 3.66 (dd, $^2J_{H,H}$ = 16.0, $^3J_{H,H}$ = 8.8 Hz, 1 H, CH_2), 4.91 (dqd, $^3J_{H,H}$ = 8.8, 7.0, 5.7 Hz, 1 H, CH), 7.30−7.55 (m, 5 H, CH), 7.65−7.95 (m, 4 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 18.8, 43.5, 50.2, 123.3 (2 C), 126.0, 129.0, 129.4 (2 C), 131.9 (2 C), 134.0 (2 C), 135.7 (2 C), 167.9 (2 C), 197.6 ppm. GC-MS: *m*/*z* (%) = 216 (48) [M − 157]$^+$, 174 (100), 130 (20). $C_{18}H_{15}NO_3Se$ (372.3): calcd. C 58.08, H 4.06, N 3.76; found C 58.33, H 4.19, N 3.55.

***Se*-Phenyl 3-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)octaneselenoate (4r):** Yield 85% (0.29 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.7−0.9 (m, 3 H, CH_3), 1.1−1.4 (m, 6 H, CH_2), 1.79−1.80 (m, 1 H, CH_2), 1.95−2.25 (m, 1 H, CH_2), 3.18 (dd,

$^2J_{H,H}$ = 15.9, $^3J_{H,H}$ = 5.2 Hz, 1 H, CH_2), 3.6 (dd, $^2J_{H,H}$ = 15.9, $^3J_{H,H}$ = 9.3 Hz, 1 H, CH_2), 4.71 (ddt, $^3J_{H,H}$ = 9.3, 5.2, 4.6 Hz, 1 H, CH), 7.25−7.45 (m, 3 H, CH), 7.50−7.60 (m, 2 H, CH), 7.70−7.80 (m, 2 H, CH), 7.80−7.90 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.8, 22.3, 25.8, 31.1, 32.2, 47.8, 49.1, 123.2 (2 C), 125.9, 128.9, 129.3 (2 C), 131.6 (2 C), 133.9 (2 C), 135.6 (2 C), 168.1 (2 C), 197.6. FT-IR: $\tilde{\nu}$ = 2929, 1772, 1711, 1370, 976 cm^{-1}. $C_{22}H_{23}NO_3Se$ (428.4): calcd. C 61.68, H 5.41, N 3.27; found C 61.44, H 5.69, N 3.02.

***Se*-Phenyl 3-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)-3-methylbutaneselenoate (4s):** Yield 87% (0.19 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.78 (s, 6 H, CH_3), 3.53 (s, 2 H, CH_2), 7.23−7.35 (m, 3 H, CH), 7.37−7.48 (m, 2 H, CH), 7.55−7.67 (m, 2 H, CH), 7.68−7.78 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 27.5 (2 C), 55.6, 57.9, 122.6 (2 C), 126.3, 128.7, 129.1 (2 C), 131.8 (2 C), 133.6 (2 C), 135.5 (2 C), 169.1 (2 C), 196.8. FT-IR: $\tilde{\nu}$ = 2930, 1771, 1708, 1317, 1069 cm^{-1}. GC-MS: *m*/*z* (%) = 230 (60) $[M - 157]^+$, 188 (100), 130 (34), 83 (43). $C_{19}H_{17}NO_3Se$ (386.3): calcd. C 59.08, H 4.44, N 3.63; found C 59.19, H 4.58, N 3.38.

***Se*-Phenyl [1-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)cyclohexyl]-ethaneselenoate (4t):** Yield 77% (0.33 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.25−1.90 (m, 10 H, CH_2), 3.15−3.25 (m, 2 H, CH_2), 7.27−7.34 (m, 3 H, CH), 7.35−7.45 (m, 2 H, CH), 7.68−7.74 (m, 2 H, CH), 7.75−7.85 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 23.0 (2 C), 25.5, 35.2 (2 C), 54.4, 62.8, 123.2, 123.4, 127.0, 129.3, 129.7 (2 C), 132.4 (2 C), 134.2 (2 C), 135.9 (2 C), 170.2 (2 C), 197.3 ppm. GC-MS: *m*/*z* (%) = 253 (10) $[M - 174]^+$, 196 (12), 148 (80), 130 (81), 106 (100), 77 (25), 51 (10). $C_{22}H_{21}NO_3Se$ (426.4): calcd. C 61.97, H 4.96, N 3.29; found C 61.71, H 5.11, N 3.07.

***Se*-Phenyl 4-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)butaneselenoate (4u):** Yield 80% (0.42 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.90−2.25 (m, 2 H, CH_2), 2.62−2.92 (m, 2 H, CH_2), 3.60−3.90 (m, 2 H, CH_2), 7.15−7.58 (m, 5 H, CH), 7.60−7.90 (m, 4 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 24.0, 36.8, 44.6, 123.1 (2 C), 128.7, 129.1 (2 C), 129.8, 131.9 (2 C), 133.9 (2 C), 135.6 (2 C), 168 (2 C), 203.6 ppm. GC-MS: *m*/*z* (%) = 345 (1) $[M - 28]^+$, 216 (100), 160 (62), 130 (20), 78 (14). $C_{18}H_{15}NO_3Se$ (372.3): calcd. C 58.08, H 4.06, N 3.76; found C 57.92, H 4.23, N 3.61.

***Se*-Phenyl 4-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)pentaneselenoate (4v):** Yield 65% (0.36 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.48 (d, $^3J_{H,H}$ = 6.9 Hz, 3 H, CH_3), 2.00−2.25 (m, 1 H, CH_2), 2.35−2.60 (m, 1 H, CH_2), 2.60−2.80 (m, 2 H, CH_2), 4.30−4.50 (m, 1 H, CH), 7.20−7.45 (m, 5 H, CH), 7.65−7.75 (m, 2 H, CH), 7.75−7.90 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 18.6, 29.0, 45.1, 46.5, 123.2 (2 C), 126.2, 128.8, 129.2 (2 C), 131.8 (2 C), 134.0 (2 C), 135.7 (2 C), 168.2 (2 C), 199.1 ppm. GC-MS: *m*/*z* (%) = 230 (100) $[M - 157]^+$, 174 (29), 148 (22), 130 (30), 83 (15). $C_{19}H_{17}NO_3Se$ (386.3): C,59.08, H 4.44, N 3.63; found C 59.20, H 4.26, N 3.80.

***Se*-Phenyl 5-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)pentaneselenoate (4w):** Yield 70% (0.38 g); m.p. 58−60 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.65−1.85 (m, 4 H, CH_2), 2.76 (t, $^3J_{H,H}$ = 6.9 Hz, 2 H, CH_2), 3.68 (t, $^3J_{H,H}$ = 6.6 Hz, 2 H, CH_2), 7.30−7.40 (m, 3 H, CH), 7.40−7.55 (m, 2 H, CH), 7.65−7.75 (m, 2 H, CH), 7.75−7.90 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 22.5, 27.7, 37.3, 46.8, 123.2 (2 C), 126.9, 128.9, 129.3 (2 C), 132.0 (2 C), 134.0 (2 C), 135.8 (2 C), 168.3 (2 C), 199.8 ppm. GC-MS: *m*/*z* (%) = 230 (76) $[M - 157]^+$, 186 (24), 160 (100), 130 (15), 77 (15). $C_{19}H_{17}NO_3Se$ (386.3): calcd. C 59.08, H 4.44, N 3.63; found C 59.23, H 4.67, N 3.48.

***Se*-Phenyl 6-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)hexaneselenoate (4x):** Yield 89% (0.25 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.32−1.50 (m, 2 H, CH_2), 1.60−1.82 (m, 4 H, CH_2), 2.70 (t, $^3J_{H,H}$ = 7.2 Hz, 2 H, CH_2), 3.67 (t, $^3J_{H,H}$ = 7.0 Hz, 2 H, CH_2), 7.30−7.45 (m, 3 H, CH), 7.45−7.55 (m, 2 H, CH), 7.65−7.75 (m, 2 H, CH), 7.75−7.90 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 24.9, 26.1, 28.2, 37.7, 47.2, 123.2 (2 C), 126.7, 128.8, 129.3 (2 C), 132.1 (2 C), 133.9 (2 C), 139.8 (2 C), 168.4 (2 C), 200.0. $C_{20}H_{19}NO_3Se$ (400.3): calcd. C 60.01, H 4.78, N 3.50; found C 60.39, H 4.81, N 3.44.

***Se*-Phenyl Prop-2-eneselenoate (8):** Oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 5.73 (dd, $^2J_{H,H}$ = 1.9, $^3J_{H,H}$ = 9.0 Hz, 1 H, CH_2), 6.50−6.24 (m, 2 H, CH), 7.27−7.44 (m, 3 H, CH), 7.45−7.58 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 127.1, 128.9, 129.3 (2 C), 129.7, 135.8 (2 C), 136.6, 199.0. FT-IR: $\tilde{\nu}$ = 3059, 1692, 1439, 967 cm^{-1}. GC-MS: *m*/*z* (%) = 212 (26) $[M^+]$, 157 (31), 77 (29), 55 (100). C_9H_8OSe (211.1): calcd. C 51.22, H 3.82; found C 51.06, H 3.99.

***Se*-Phenyl (2*E*)-Oct-2-eneselenoate (9):** Oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.88 (t, $^3J_{H,H}$ = 6.5 Hz, 3 H, CH_3), 1.20−1.62 (m, 6 H, CH_2), 2.18 (qd, $^3J_{H,H}$ = 6.9, 1.3 Hz, 2 H, CH_2), 8.14 (dt, $^3J_{H,H}$ = 15.4, 1.3 Hz, 1 H, CH), 8.91 (dt, $^3J_{H,H}$ = 15.4, 6.9 Hz, 1 H, CH), 7.30−7.45 (m, 3 H, CH), 7.48−7.58 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.8, 22.3, 27.5, 31.3, 32.2, 126.8, 128.7, 129.2 (2 C), 130.1, 135.9 (2 C), 146.9, 195.9. FT-IR: $\tilde{\nu}$ = 2928, 1694, 1625, 1021 cm^{-1}. GC-MS: *m*/*z* (%) = 282 (1) $[M^+]$, 157 (15), 125 (95), 77 (11), 41 (18). $C_{14}H_{18}OSe$ (281.2): calcd. C 59.80, H 6.45; found C 59.96, H 6.67.

Synthesis of Amides: The amine (1.5 mmol) was added at room temperature to a stirred solution of **4** (1 mmol) in dichloromethane (10 mL). When the chlorohydrate of the amine was employed it was necessary to add triethylamine (1.5 mmol). After complete consumption of the *Se*-phenyl selenocarboxylate, the solvents were evaporated. The residue was purified by chromatography on a silica gel column using a mixture of diethyl ether and methanol as eluent. The amides **10** were obtained in a pure form and diphenyl diselenide was recovered. Yields and physical and spectroscopic data of the amides thus obtained are reported below.

Methyl 2-{[4-(Acetoxy)pentanoyl]amino}-3-phenylpropanoate (10g): Yield 68% (0.22 g); oil; 1:1 mixture of two diastereoisomers. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.20 (d, $^3J_{H,H}$ = 6.2 Hz, 3 H, CH_3), 1.21 (d, $^3J_{H,H}$ = 6.2 Hz, 3 H, CH_3), 1.75−1.95 (m, 4 H, CH_2), 2.03 (s, 3 H, CH_3), 2.05 (s, 3 H, CH_3), 2.15−2.30 (m, 4 H, CH_2), 3.10 (dd, $^2J_{H,H}$ = 13.7, $^3J_{H,H}$ = 5.5 Hz, 2 H, CH_2), 3.20 (dd, $^2J_{H,H}$ = 13.7, $^3J_{H,H}$ = 5.7 Hz, 2 H, CH_2), 3.74 (s, 3 H, CH_3), 3.75 (s, 3 H, CH_3), 4.85−5.00 (m, 4 H, CH), 6.05 (d, $^3J_{H,H}$ = 7.8 Hz, 1 H, NH), 6.10 (d, $^3J_{H,H}$ = 7.8 Hz, 1 H, NH), 7.05−7.15 (m, 4 H, CH), 7.22−7.28 (m, 6 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 19.9 (2 C), 21.2 (2 C), 31.4, 31.5, 32.3 (2 C), 37.7 (2 C), 52.3 (2 C), 52.9, 53.0, 70.1 (2 C), 127.0 (2 C), 128.4 (4 C), 129.1 (4 C), 135.7 (2 C), 170.7 (2 C), 171.5 (2 C), 172.0 (2 C) ppm. GC-MS: *m*/*z* (%) = 321 (1) $[M^+]$, 162 (100), 131 (20), 120 (33), 101 (84), 91 (16), 43 (20). $C_{17}H_{23}NO_5$ (321.4): calcd. C 63.54, H 7.21, N 4.36; found C 63.79, H 7.47, N 4.13.

Methyl 5-{[(1*S*)-1-(Hydroxymethyl)-2-methoxy-2-oxoethyl]amino}-5-oxopentanoate (10d): Yield 86% (0.20 g); oil. $[\alpha]_D^{20}$ = −13.26 (*c* = 0.40 in CH_3OH). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.96 (quint, $^3J_{H,H}$ = 7.2 Hz, 2 H, CH_2), 2.36 (t, $^3J_{H,H}$ = 7.2 Hz, 2

H, CH_2), 2.40 (t, $^3J_{H,H}$ = 7.2 Hz, 2 H, CH_2), 3.25 (br. s, 1 H, OH), 3.68 (s, 3 H, CH_3), 3.78 (s, 3 H, CH_3), 4.05−3.7 (m, 2 H, CH_2), 4.63 (dt, $^3J_{H,H}$ = 7.8, 3.6 Hz, 1 H, CH), 7.20 (d, $^3J_{H,H}$ = 7.8 Hz, 1 H, NH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 20.5, 32.8, 34.8, 51.4, 52.3, 54.4, 62.3, 171.0, 172.9, 173.8. $C_{10}H_{17}NO_6$ (247.2): calcd. C 48.58, H 6.93, N 5.67; found C 48.65, H 7.12, N 5.44.

***N*-(Prop-2-ynyl)-3-[(phenylsulfonyl)amino]propanamide (10n):** Yield 98% (0.23 g); m.p. 122−124 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 2.35 (t, $^4J_{H,H}$ = 2.6 Hz, 2 H, CH_2), 2.42 (t, $^3J_{H,H}$ = 6.7 Hz, 2 H, CH_2), 3.15 (t, $^3J_{H,H}$ = 6.7 Hz, 2 H, CH_2), 3.94 (d, $^4J_{H,H}$ = 2.6 Hz, 1 H, CH), 4.50 (s, 2 H, NH), 7.45−7.65 (m, 3 H, CH), 7.80−7.90 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 28.3, 35.1, 38.7, 58.7, 70.8, 126.4 (2 C), 128.7 (2 C), 132.2, 139.7, 171.0. $C_{12}H_{14}N_2O_3S$ (266.3): calcd. C 54.12, H 5.30, N 10.52; found C 54.21, H 5.13, N 10.30.

Methyl (2*S*)-2-{[3-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)butanoyl]amino}-3-phenylpropanoate (10q): Yield 70% (0.20 g); m.p. 109−112 °C; 1:1 mixture of two diastereoisomers. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.32 (d, $^3J_{H,H}$ = 6.6 Hz, 3 H, CH_3), 1.34 (d, $^3J_{H,H}$ = 6.9 Hz, 3 H, CH_3), 2.58−2.66 (m, 2 H, CH_2), 2.75−3.20 (m, 6 H, CH_2), 3.45 (s, 3 H, CH_3), 3.55 (s, 3 H, CH_3), 4.65−4.85 (m, 4 H, CH), 6.24 (d, $^3J_{H,H}$ = 7.9 Hz, 2 H, NH), 6.85−6.95 (m, 2 H, CH), 6.95−7.25 (m, 8 H, CH), 7.55−7.80 (m, 8 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 18.6 (2 C), 37.7 (2 C), 39.7, 39.8, 44.0, 44.1, 52.1, 52.2, 53.0 (2 C), 123.1 (4 C), 127.0 (2 C), 128.4 (2 C), 128.5 (2 C), 129.0 (2 C), 129.1 (2 C), 131.9 (4 C), 133.8 (2 C), 133.9 (2 C), 135.8, 135.9, 168.2 (4 C), 169.5 (2 C), 171.9 (2 C) ppm. GC-MS: *m*/*z* (%) = 394 (2) [M^+], 216 (35), 174 (100), 162 (92), 130 (21), 91 (13). $C_{22}H_{22}N_2O_5$ (394.4): calcd. C 66.99, H 5.62, N 7.10; found C 66.74, H 5.88, N 6.83.

Ethyl 3-{[4-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)butanoyl]amino}propanoate (10u): Yield 81% (0.27 g); m.p. 99−102 °C. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.28 (t, $^3J_{H,H}$ = 7.1 Hz, 3 H, CH_3), 2.04 (tt, $^3J_{H,H}$ = 6.6, 6.4 Hz, 2 H, CH_2), 2.22 (t, $^3J_{H,H}$ = 6.6 Hz, 2 H, CH_2), 2.55 (t, $^3J_{H,H}$ = 6.1 Hz, 2 H, CH_2), 3.51 (q, $^3J_{H,H}$ = 6.1 Hz, 2 H, CH_2), 3.74 (t, $^3J_{H,H}$ = 6.4 Hz, 2 H, CH_2), 4.26 (q, $^3J_{H,H}$ = 7.1 Hz, 2 H, CH_2), 6.52 (t, $^3J_{H,H}$ = 6.1 Hz, 1 H, NH), 7.70−7.78 (m, 2 H, CH), 7.79−7.90 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 14.0, 24.6, 33.5, 34.0, 34.8, 37.1, 60.5, 123.1 (2 C), 131.9 (2 C), 133.8 (2 C), 168.3 (2 C), 171.7, 172.3 ppm. GC-MS: *m*/*z* (%) = 334 (4) [M^+], 216 (38), 159 (77), 116 (100), 77 (13). $C_{17}H_{20}N_2O_5$ (332.3): calcd. C 61.44, H 6.07, N 8.43; found C 61.64, H 6.23, N 8.19.

Methyl (2*S*)-2-{[(4*S*)-4-Acetoxy-5-(benzyloxy)pentanoyl]amino}-3-hydroxypropanoate (10k): Yield 92% (0.13 g); oil. $[\alpha]_D^{21}$ = +21.06 (*c* = 2.10 in $CHCl_3$). ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.82−1.94 (m, 1 H, CH_2), 2.10 (s, 3 H, CH_3), 2.03−2.13 (m, 1 H, CH_2), 2.23 (ddd, $^2J_{H,H}$ = 15.0, $^3J_{H,H}$ = 8.9, 6.5 Hz, 1 H, CH_2), 2.35 (ddd, $^2J_{H,H}$ = 15.0, $^3J_{H,H}$ = 6.9, 5.7 Hz, 1 H, CH_2), 3.40 (br. s, 1 H, OH), 3.53 (d, $^3J_{H,H}$ = 4.9 Hz, 2 H, CH_2), 3.78 (s, 3 H, CH_3), 3.94 (dd, $^2J_{H,H}$ = 11.5, $^3J_{H,H}$ = 3.2 Hz, 1 H, CH_2), 3.98 (dd, $^2J_{H,H}$ = 11.5, $^3J_{H,H}$ = 3.2 Hz, 1 H, CH_2), 4.50 (d, $^2J_{H,H}$ = 12.1 Hz, 1 H, CH_2), 4.57 (d, $^2J_{H,H}$ = 12.1 Hz, 1 H, CH_2), 4.67 (dt, $^3J_{H,H}$ = 7.5, 3.2 Hz, 1 H, CH), 5.15−5.07 (m, 1 H, CH), 6.59 (d, $^3J_{H,H}$ = 7.5 Hz, 1 H, NH), 7.25−7.40 (m, 5 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 21.2, 26.6, 31.7, 52.6, 54.8, 62.8, 71.1, 71.7, 73.1, 127.6 (2 C), 127.7, 128.4 (2 C), 137.7, 170.1, 171.6, 171.8. FT-IR: $\tilde{\nu}$ = 3315, 2952, 1738, 1243 cm^{-1}. GC-MS: *m*/*z* (%) = 243 (3) [$M - 124]^+$, 155 (11), 101 (14), 91 (100). $C_{18}H_{25}NO_7$ (367.4): calcd. C 58.85, H 6.86, N 3.81; found C 58.71, H 6.975, N 3.73.

(1*S*)-1-[(Benzyloxy)methyl]-5-oxo-5-{[(1*R*)-1-phenylethyl]amino}-pentyl Acetate (10l): Yield 82% (0.15 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 1.45 (d, $^3J_{H,H}$ = 6.9 Hz, 3 H, CH_3), 1.55−1.76 (m, 4 H, CH_2), 2.05 (s, 3 H, CH_3), 2.10−2.24 (m, 2 H, CH_2), 3.50 (m, 2 H, CH_2), 4.46 (d, $^2J_{H,H}$ = 12.1 Hz, 1 H, CH_2), 4.56 (d, $^2J_{H,H}$ = 12.1 Hz, 1 H, CH_2), 4.98−5.10 (m, 1 H, CH), 5.10 (dq, $^3J_{H,H}$ = 7.8, 6.9 Hz, 1 H, CH), 5.96 (d, $^3J_{H,H}$ = 7.8 Hz, 1 H, NH), 7.20−7.40 (m, 10 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 21.1, 21.2, 21.7, 30.1, 35.9, 48.6, 70.9, 72.1, 73.1, 126.1 (2 C), 127.2, 127.5, 127.6 (2 C), 128.3 (2 C), 128.5 (2 C), 137.9, 143.2, 170.7, 171.5 ppm. GC-MS: *m*/*z* (%) = 323 (4) [$M - 61]^+$, 207 (16), 163 (20), 120 (100), 105 (58), 91 (68). $C_{23}H_{29}NO_4$ (383.5): calcd. C 72.04, H 7.62, N 4.02; found C 71.89, H 7.45, N 3.86.

Conversion of *Se*-Phenyl Selenocarboxylates into *O*-Alkyl Esters: Anhydrous alcohol (5 mmol) was added to a mixture of *Se*-phenyl selenocarboxylates **4** (1 mmol) and anhydrous copper(II) chloride (2.2 mmol) in dry acetonitrile (5 mL). The mixture was stirred at room temperature and monitored by TLC. After 2 h, the selenocarboxylic ester was completely consumed and the reaction mixture was diluted with dichloromethane. A 10% NaOH solution (0.2 mL) was added and stirring was continued for a few minutes. The reaction mixture was then filtered through Celite and the filtrate concentrated. The crude product was purified by column chromatography on silica gel using a 8:2 mixture of light petroleum and diethyl ether as eluent. Compound **11f** is commercially available and compound **11a** has already been described in the literature.[21] Yields and physical and spectroscopic data of all the other compounds are reported below.

Ethyl 3-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)octanoate (11b): Yield 83% (0.13 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.8 (t, $^3J_{H,H}$ = 6.5 Hz, 3 H, CH_3), 1.15 (t, $^3J_{H,H}$ = 6.9 Hz, 3 H, CH_3), 1.1−1.3 (m, 6 H, CH_2), 1.6−1.9 (m, 1 H, CH), 1.95−2.20 (m, 1 H, CH), 2.74 (dd, $^2J_{H,H}$ = 15.8, $^3J_{H,H}$ = 5.2 Hz, 1 H, CH_2), 3.16 (dd, $^2J_{H,H}$ = 15.8, $^3J_{H,H}$ = 9.8 Hz, 1 H, CH_2), 4.03 (q, $^3J_{H,H}$ = 6.9 Hz, 2 H, CH_2), 4.65 (tt, $^3J_{H,H}$ = 9.8, 5.21 Hz, 1 H, CH), 7.7−7.8 (m, 2 H, CH), 7.8−7.9 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.9, 14.0, 22.3, 25.9, 31.2, 32.3, 37.0, 48.1, 60.5, 123.1 (2 C), 131.7 (2 C), 133.8 (2 C), 168.3 (2 C), 170.9 ppm. GC-MS: *m*/*z* (%) = 317 (28) [$M]^+$, 272 (19), 246 (34), 230(50), 174 (45), 160 (66), 148 (19), 130 (29), 91 (71). $C_{18}H_{23}NO_4$ (317.4): calcd. C 68.12, H 7.30, N 4.41; found C 68.33, H 7.52, N 4.29.

Isopropyl 3-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)octanoate (11c): Yield 72% (0.12 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.8 (t, $^3J_{H,H}$ = 6.5 Hz, 3 H, CH_3), 1.05 (d, $^3J_{H,H}$ = 6.2 Hz, 3 H, CH_3), 1.1 (d, $^3J_{H,H}$ = 6.2 Hz, 3 H, CH_3), 1.15−1.40 (m, 6 H, CH_2), 1.60−1.85 (m, 1 H, CH_2), 1.95−2.20 (m, 1 H, CH_2), 2.7 (dd, $^2J_{H,H}$ = 15.6, $^3J_{H,H}$ = 5.2 Hz, 1 H, CH_2), 3.15 (dd, $^2J_{H,H}$ = 15.6, $^3J_{H,H}$ = 9.9 Hz, 1 H, CH_2), 4.63 (tt, $^3J_{H,H}$ = 9.6, 5.2 Hz, 1 H, CH), 4.9 (sept, $^3J_{H,H}$ = 6.2 Hz, 1 H, CH), 7.60−7.75 (m, 2 H, CH), 7.75−7.85 (m, 2 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.9, 21.5, 21.6, 22.4, 25.9, 31.2, 32.3, 37.4, 48.2, 67.9, 123.1 (2 C), 131.8 (2 C), 133.9 (2 C), 168.3 (2 C), 170.4 ppm. GC-MS: *m*/*z* (%) = 331 (29) [M^+], 272 (30), 230(66), 200 (40), 174 (100), 160 (80), 148 (28), 130 (29), 124 (14). $C_{19}H_{25}NO_4$ (331.4): calcd. C 68.86, H 7.60, N 4.23; found C 69.00, H 7.81, N 4.05.

Benzyl 3-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)octanoate (11d): Yield 62% (0.13 g); oil. ^{1}H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.85 (t, $^3J_{H,H}$ = 6.4 Hz, 3 H, CH_3), 1.1−1.4 (m, 6 H, CH_2), 1.6−1.8 (m, 1 H, CH_2), 1.9−2.2 (m, 1 H, CH_2), 2.82 (dd, $^2J_{H,H}$ =

15.8, $^3J_{H,H}$ = 5.2 Hz, 1 H, CH_2), 3.25 (dd, $^2J_{H,H}$ = 15.8, $^3J_{H,H}$ = 9.8 Hz, 1 H, CH_2), 4.6−4.8 (m, 1 H, CH), 5.05 (m, 2 H, CH_2), 7.1−7.3 (m, 5 H, CH), 7.60−7.85 (m, 4 H, CH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.8, 22.3, 25.8, 31.1, 32.2, 36.9, 48.0, 66.3, 123.1 (2 C), 128.0, 128.2 (2 C), 128.3 (2 C), 131.6 (2 C), 133.7 (2 C), 135.4, 168.2 (2 C), 170.7 ppm. GC-MS: *m*/*z* (%) = 288 (1) $[M - 91]^+$, 273 (100), 230 (20), 160 (34), 130 (12), 91 (71). $C_{23}H_{25}NO_4$ (379.4): calcd. C 72.80, H 6.64, N 3.69; found C 72.65, H 6.88, N 3.42.

Conversion of *Se*-Phenyl Selenocarboxylates into Carboxylic Acids: *Se*-Phenyl selenocarboxylate **4** (1 mmol) was dissolved in tetrahydrofuran (10 mL) at room temperature and a 30% solution of hydrogen peroxide (0.4 mL) was added. When TLC analysis indicated that the selenocarboxylic ester had been completely consumed, anhydrous sodium sulfate was added and the resulting suspension filtered off. The filtrate was concentrated and the pure acids **11e** and **11g** were obtained after column chromatography using a 96:4 mixture of dichloromethane and methanol as eluent. Compound **11g** is a commercial product.

3-(1,3-Dioxo-1,3-dihydro-2*H*-isoindol-2-yl)octanoic Acid (11e): Yield 81% (0.10 g); m.p. 75−78 °C. ^1H NMR (200 MHz, $CDCl_3$, 25 °C, TMS): δ = 0.85 (t, $^3J_{H,H}$ = 6.5 Hz, 3 H, CH_3), 1.10−1.31 (m, 6 H, CH_2), 1.60−1.81 (m, 1 H, CH), 1.95−2.11 (m, 1 H, CH), 2.82 (dd, $^2J_{H,H}$ = 16.5, $^3J_{H,H}$ = 5.5 Hz, 1 H, CH_2), 3.21 (dd, $^2J_{H,H}$ = 16.5, $^3J_{H,H}$ = 9.3 Hz, 1 H, CH_2), 4.65 (ddt, $^3J_{H,H}$ = 9.3, 5.5, 4.9 Hz, 1 H, CH), 7.76−7.90 (m, 4 H, CH), 8.3 (br. s, 1 H, OH) ppm. ^{13}C NMR (50 MHz, $CDCl_3$, 25 °C, TMS): δ = 13.9, 22.3, 25.8, 31.1, 32.2, 36.7, 47.7, 129.2 (2 C), 131.6 (2 C), 133.9 (2 C), 168.3 (2 C), 176.3. FT-IR: $\tilde{\nu}$ = 3002, 2925 1773.7, 1712.5, 1376.4 cm^{-1}. $C_{16}H_{19}NO_4$ (289.3): calcd. C 62.42, H 6.62, N 4.84; found C 62.50, H 6.73, N 4.70.

Acknowledgments

Financial support from MIUR, National Project "Stereoselezione in Sintesi Organica. Metodologie e Applicazioni", FIRB Project "Progettazione, Preparazione e Valutazione Biologica e Farmacologica di Nuove Molecole Organiche Quali Potenziali Farmaci Innovativi", Consorzio CINMPIS and the University of Perugia, Progetti di Ateneo, is gratefully acknowledged.

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Received March 18, 2004

