

Selenium in Organic Synthesis: A Novel Route to 1-Phenylselenobutadienes and 1,4-Dicarbonyl Compounds

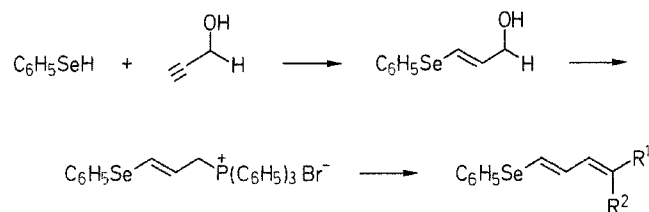
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Two approaches to 1-phenylseleno-1,3-butadienes **5** are reported, via phenylselenoalkenyldene phosphoranes and phenylselenoalkenals, respectively. 1,4-Dicarbonyl compounds are prepared from the 1-phenylselenobutadienes or from the phenylselenoalkenals.

Vinyl selenides are promising synthetic intermediates.¹ Some time ago we developed methods for the synthesis of these intermediates based on the addition of selenophenol to acetylenes² and on the Wittig reaction of selenium containing phosphoranes³ and phosphonates⁴ with carbonyl compounds. We have demonstrated that these intermediates can be efficiently converted to carbonyl compounds through hydrolysis.^{1,3,4} In the present work we have employed these methods to develop an efficient route to 1-phenylselenobutadienes and 1,4-dicarbonyl compounds, which are precursors of the cyclopentenone ring systems present in jasmonoids, prostaglandins, and rethrolones.

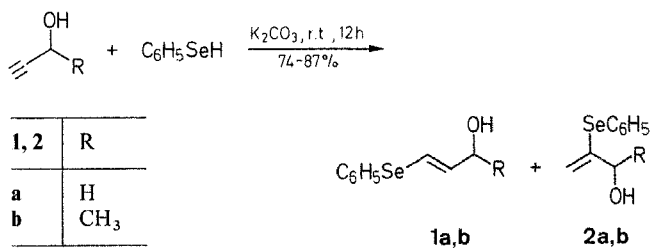
Our first approach to 1-phenylselenobutadienes was planned as a three step sequence involving the addition of selenophenol to propargylic alcohols, transformation of the alcohols to phosphonium salts, and then Wittig reaction of the salts with carbonyl compounds to olefins (Scheme A)



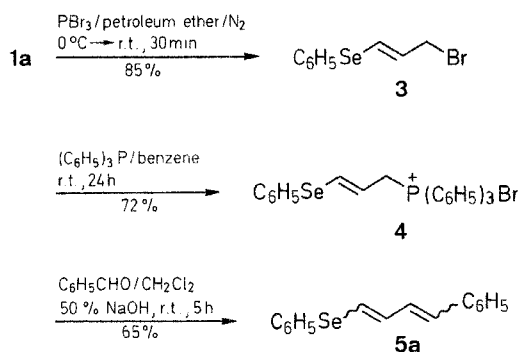
Scheme A

In the first step of the sequence, we found that selenophenol adds to propargyl alcohol (R=H) under base catalysis to afford a mixture of regioisomers **1** and **2** in a 2.5:1.0 ratio, which were easily separated by flash chromatography. The presence of a methyl substituent at C-1 of the propargylic alcohol enhances the regioselectivity. In this case, only a trace of the regioisomer **2** was observed. In both cases, the vinylic selenides **1** exhibit the expected *Z*-stereochemistry.²

Reaction of the alcohol **1a** with phosphorous tribromide in petroleum ether (30–60°C) at 0°C under a stream of nitrogen furnished the corresponding bromide **3** in 85% yield. Partial isomerization of the double bond was observed in the bromination, leading to a mixture of the *Z*- and *E*-bromides **3**. These



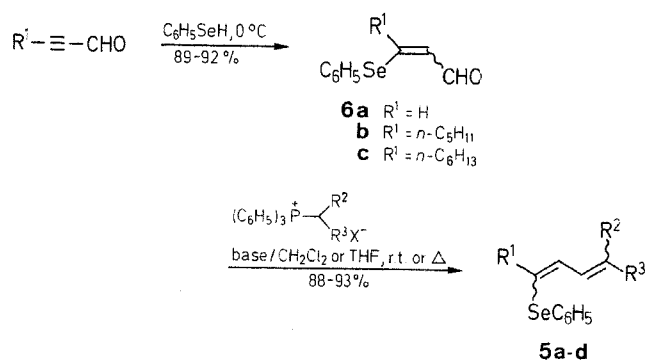
bromides are unstable and were therefore used without further purification to prepare the corresponding phosphonium derivative **4** (72% yield).



The Wittig reaction of the phosphonium salt **4** with benzaldehyde under phase transfer conditions led to the corresponding 1-phenylselenobutadiene **5a** in 65% yield.

Since the first step of this route to 1-phenylselenobutadienes exhibits low regioselectivity, we envisaged an alternative approach to these intermediates starting from propargylic aldehydes.

The addition of selenophenol to propynal occurs without base catalyst, and leads to the corresponding vinylic selenide **6a** in 92% yield. In this case, only a single regioisomer was observed, probably because the addition occurs via a reversible Michael reaction. In contrast, the addition of selenophenol to 2-octynal and 2-nonynal is very slow if the reaction is performed in the absence of a basic catalyst. The system of choice to effect this reaction was found to be potassium carbonate in a mixture of tetrahydrofuran/*t*-butanol. Under these conditions, the reaction occurs rapidly at room temperature to give the vinylic selenides **6b, c** as single products in good yields. Mixtures of *Z*- and *E*-vinylic selenides were formed, however, with the *Z*-isomers being the major components.

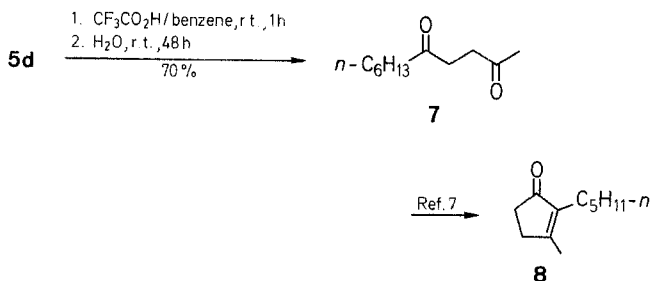


5	R ¹	R ²	R ³	Yield (%)
a	H	H	C ₆ H ₅	91
b	H	H	C ₆ H ₅ Se	93
c	H	H	COOC ₂ H ₅	90
d	<i>n</i> -C ₆ H ₁₃	CH ₃	C ₆ H ₅ Se	88

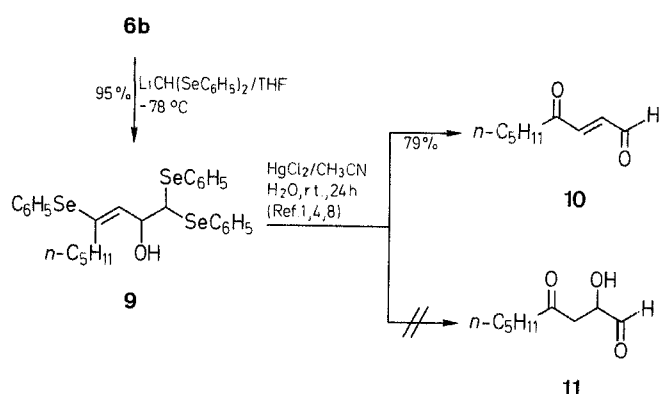
These compounds are valuable three carbon homologating reagents, as will be discussed next. In particular aldehyde **6a** is a versatile compound for oxetane ring formation.^{5,6}

The aldehydes **6** react with Wittig reagents under a variety of experimental conditions to give the 1-phenylselenobutadienes **5a-d** in excellent yields.

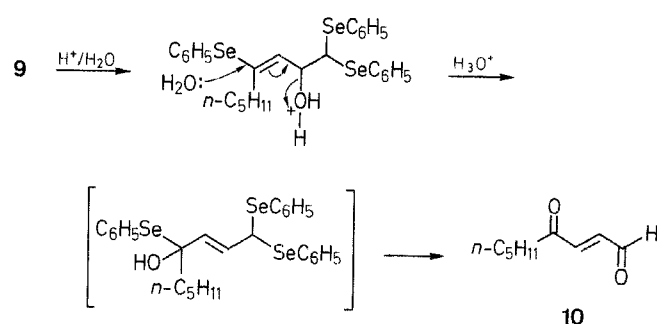
Compound **5d** was hydrolyzed with trifluoroacetic acid^{1,4} in benzene and then treated with water to give in 70% yield the 1,4-diketone **7**, which has been transformed to dihydrojasmonone **8** using described methodologies.⁷



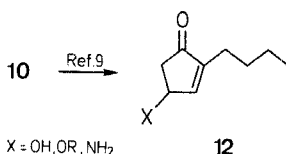
On the other hand, the aldehyde **6b** reacts with the lithium derivative of bis(phenylseleno)methane to give the alcohol **9**, which can be hydrolyzed with mercuric (II) chloride in acetonitrile/water^{1,4,8} to give in 79% yield the 1,4-dicarbonyl compound **10**, and not the 2-hydroxy derivative **11**.



Mechanistically, this reaction probably follows the pathway depicted below:



Trans-1,4-dicarbonyl compounds like **10** can be transformed in a number of ways to substituted cyclopentenones of the type **12** or **8**.⁹



In conclusion, two routes have been developed to 1-phenylselenobutadienes and 1,4-dicarbonyl compounds, substances which should prove useful in the construction of the cyclopentenone ring systems that are present in several important classes of natural products.

Selenophenol,¹⁰ diphenyldiselenide,¹¹ phenylselenenylbromide,¹² bromomethyl(phenyl)selenide,³ bis(phenylseleno)methane,¹³ tris(phenylseleno)methane,¹⁴ propargylaldehyde,¹⁵ 2-octynal,¹⁶ 2-nonynal,¹⁷ (phenylseleno)methyltriphenylphosphonium bromide,³ were prepared by known methods.

Addition of Selenophenol to Propargyl Alcohol:

Selenophenol (1.57 g, 10 mmol) is added to 2-propyn-1-ol (0.62 g, 11 mmol) and potassium carbonate (0.30 g) at room temperature. After stirring for 12 h the mixture is diluted with ether (50 ml), washed with saturated solution of sodium bicarbonate, (1 × 30 ml), dried with magnesium sulfate and evaporated. The residue is purified by flash chromatography, eluting with hexane/ethyl acetate (7:3).

Fraction 1: (Z)-3-Hydroxy-1-phenylselenopropene (1a); yield: 1.32 g (62%).

C₉H₁₀OSe calc. C 50.73 H 4.73
(213.1) found 50.69 4.82

IR (Film): $\nu = 3350, 1610, 1580, 790, 740, 690 \text{ cm}^{-1}$.

¹H-NMR (CCl₄): $\delta = 3.60$ (br s, 1 H); 4.20 (dd, $J = 5 \text{ Hz}$, 1 Hz, 2 H); 6.05 (dt, $J = 5 \text{ Hz}$, 9 Hz, 1 H); 6.40 (dt, $J = 9 \text{ Hz}$, 1 Hz, 1 H); 6.90–7.20 (m, 3 H); 7.20–7.50 ppm (m, 2 H).

Fraction 2: 3-Hydroxy-2-phenylselenopropene (2a); yield: 0.53 g (25%).

C₉H₁₀OSe calc. C 50.73 H 4.73
(213.1) found 50.68 4.85

IR: (Film) = 3350, 1610, 1575, 740, 690 cm^{-1} .

¹H-NMR (CCl₄): $\delta = 2.90$ (br s, 1 H); 4.12 (dd, $J = 1.2 \text{ Hz}$, 1.5 Hz, 2 H); 5.35 (dt, $J = 1.2 \text{ Hz}$, 1.6 Hz, 1 H); 5.86 (dt, $J = 1.5 \text{ Hz}$, 1.6 Hz, 1 H); 7.1–7.4 (m, 3 H); 7.4–7.7 ppm (m, 2 H).

(Z)-3-Hydroxy-1-phenylseleno-1-butene (1b):

The same procedure described above is used to prepare **1b**. The product is purified by flash chromatography eluting with hexane/ethyl acetate (7:3); yield: 1.68 g (74%).

C₁₀H₁₂OSe calc. C 52.89 H 5.33
(227.2) found 52.81 5.40

IR (Film): $\nu = 3350, 1610, 1580, 735, 690 \text{ cm}^{-1}$.

¹H-NMR (CCl₄): $\delta = 1.28$ (d, $J = 7 \text{ Hz}$, 3 H); 2.30 (br s, 1 H); 4.54 (q, $J = 7 \text{ Hz}$, 1 H); 6.00 (dd, $J = 9 \text{ Hz}$, 7 Hz, 1 H); 6.45 (dd, $J = 7 \text{ Hz}$, 1 Hz); 7.0–7.7 ppm (m, 5 H).

3-Bromo-1-(phenylseleno)-1-propene (3):

To a solution of (Z)-3-hydroxy-1-phenylselenopropene (**1a**; 0.85 g, 4.0 mmol) in light petroleum (10 ml) at 0°C, under a stream of nitrogen,

is added phosphorus tribromide (0.55 g, 2.0 mmol) in light petroleum (5 ml). The mixture is stirred for 30 min at room temperature, treated with ice-water and extracted with light petroleum (3 × 20 ml). The organic phase is washed with saturated brine (30 ml), dried with magnesium sulfate and the solvent is removed under reduced pressure; yield: 0.92 g (85%) (mixture of *Z*- and *E*-isomers; 50:50 by $^1\text{H-NMR}$).

$\text{C}_9\text{H}_9\text{BrSe}$ calc. C 39.17 H 3.29
(276.0) found 39.73 3.12

IR (Film): $\nu = 1600, 730, 690\text{ cm}^{-1}$.

$^1\text{H-NMR}$ (CCl_4): $\delta = 3.8\text{--}3.9$ (m, 2H); 3.83 (d, $J = 8\text{ Hz}$); 3.96 (d, $J = 8\text{ Hz}$, 2H); 5.6–6.9 (m, 2H); 6.9–7.7 ppm (m, 5H).

[3-(Phenylseleno)-2-propenyl]triphenylphosphonium Bromide (4):

A mixture of 3-bromo-1-(phenylseleno)-2-propene (3; 0.93 g, 3.34 mmol) in benzene (10 ml) and triphenylphosphine (1.45 g, 4.00 mmol) is stirred for 24 h at room temperature. The solvent is then evaporated under reduced pressure and the residue recrystallized from dichloromethane/ethyl acetate; yield: 1.29 g (72%); m.p. 202–205 °C (mixture of *Z*- and *E*-isomers).

$\text{C}_{27}\text{H}_{24}\text{BrPSe}$ calc. C 60.25 H 4.49
(538.4) found 60.76 4.17

IR (Film): $\nu = 1610, 1590, 1575, 1440, 750, 740, 725, 590\text{ cm}^{-1}$.

$^1\text{H-NMR}$ (CDCl_3): $\delta = 4.53\text{--}5.10$ (m, 2H); 6.67–8.18 ppm (m, 22H).

4-Phenyl-1-(phenylseleno)-1,3-butadiene (5a):

To a solution of 3-(phenylseleno)-2-propenyltriphenylphosphonium bromide (4; 0.70 g, 1.1 mmol) and benzaldehyde (0.10 g, 1.0 mmol) in dichloromethane (4 ml), is added 50% sodium hydroxide (4 ml). The mixture is stirred for 5 h at room temperature. Then the phases are separated and the residue is filtered through a short column of silica gel eluting with hexane; yield: 0.29 g (65%) (mixture of 4 isomers; 18:15:33:34).

$\text{C}_{16}\text{H}_{14}\text{Se}$ calc. C 67.39 H 4.95
(285.3) found 67.27 4.94

IR (Film): $\nu = 1610, 1600, 1575, 790, 775\text{ cm}^{-1}$.

$^1\text{H-NMR}$ (CCl_4): $\delta = 6.0\text{--}6.9$ (m, 4H); 6.9–7.6 ppm (m, 10H).

3-(Phenylseleno)-2-nonenal (6c):

To a solution of 2-nonyl (1.66 g, 12 mmol) and potassium carbonate (0.10 mg, 0.5 mmol) in tetrahydrofuran (10 ml) and *t*-butanol (2 ml) at room temperature is added dropwise selenophenol (1.57 g, 10 mmol) over a period of 30 min. The mixture is then diluted with ether (20 ml) and washed with saturated solution of sodium bicarbonate (20 ml), dried with magnesium sulfate and the solvent evaporated. The residue is purified by flash chromatography eluting with hexane/ether (1:1); yield: 2.65 g (89%) (mixture of *Z*- and *E*-isomers, 80:20 by $^1\text{H-NMR}$).

$^1\text{H-NMR}$ (CCl_4): $\delta = 0.7\text{--}0.9$ (m, 3H); 1.0–1.9 (m, 8H); [2.26 (t, $J = 8\text{ Hz}$, *Z*-isomer); 2.85 (t, $J = 8\text{ Hz}$, *E*-isomer), 2H]; [5.69 (d, $J = 8\text{ Hz}$, *Z*-isomer); 6.47 (dt, $J = 4\text{ Hz}$, 1 Hz, *E*-isomer), 1H]; 7.16–7.83 (m, 5H); [9.67 (d, $J = 8\text{ Hz}$, *E*-isomer); 9.79 ppm (d, $J = 4\text{ Hz}$, *Z*-isomer, 1H)].

$\text{C}_{15}\text{H}_{20}\text{OSe}$ calc. C 61.03 H 6.83
(295.3) found 61.81 6.76

IR (Film, *Z* + *E*-isomers): $\nu = 1665, 1575, 740, 690\text{ cm}^{-1}$.

The same procedure is used to prepare 3-(phenylseleno)-2-octenal (6b). The *Z*- and the *E*-isomers are separated by flash chromatography eluting with hexane/ethyl acetate (8:2); yield: 2.59 g (92%), a mixture of *Z*- and *E*-isomers, 82:18 by $^1\text{H-NMR}$. *Z*-Isomer; yield: 1.92 g (68%).

$\text{C}_{14}\text{H}_{18}\text{OSe}$ calc. C 59.80 H 6.45
(281.3) found 59.79 6.60

IR (Film): $\nu = 1660, 1575, 745, 695\text{ cm}^{-1}$.

$^1\text{H-NMR}$ (CCl_4): $\delta = 0.77$ (t, $J = 6\text{ Hz}$, 3H); 0.9–1.8 (m, 6H); 2.22 (t, $J = 7\text{ Hz}$, 2H); 6.41 (dt, $J = 4\text{ Hz}$, 0.8 Hz, 1H); 7.0–7.4 (m, 3H); 7.4–7.7 (m, 2H); 9.80 ppm (d, $J = 4\text{ Hz}$, 1H).

E-Isomer; yield: 0.44 g (16%).

$\text{C}_{14}\text{H}_{18}\text{OSe}$ calc. C 59.80 H 6.45
(281.3) found 59.79 6.60

IR (Film): $\nu = 1660, 1560, 760, 705\text{ cm}^{-1}$.

$^1\text{H-NMR}$ (CCl_4): $\delta = 0.90$ (t, $J = 6\text{ Hz}$, 3H); 1.1–2.0 (m, 6H); 2.81 (t, $J = 7\text{ Hz}$, 2H); 5.65 (d, $J = 7\text{ Hz}$, 1H); 7.2–7.7 (m, 5H); 9.60 ppm (d, $J = 7\text{ Hz}$, 1H).

3-(Phenylseleno)acrylaldehyde (6a):

Selenophenol (2.35 g, 15 mmol) is added to propynal (0.87 g, 16 mmol) at 0 °C and the mixture is stirred for 15 min. The product is distilled (60 °C/0.05 torr). The *Z*- and *E*-isomers (~1:1) are separated by flash chromatography eluting with hexane/ether (1:1).

Z-isomer; yield: 1.4 g (47%).

IR (Film): $\nu = 1650, 1570, 750, 695\text{ cm}^{-1}$.

$^1\text{H-NMR}$ (CCl_4): $\delta = 6.68$ (dd, $J = 9.0\text{ Hz}$, 2.0 Hz, 1H); 7.79 (dd, $J = 9.0\text{ Hz}$, 2.0 Hz, 1H); 7.2–7.4 (m, 3H); 7.4–7.7 (m, 2H); 9.77 ppm (dd, $J = 2.0\text{ Hz}$, 2 Hz, 1H).

E-isomer; yield: 1.3 g (44%).

IR (Film): $\nu = 1670, 1575, 740, 695\text{ cm}^{-1}$.

$^1\text{H-NMR}$ (CCl_4): $\delta = 6.13$ (dd, $J = 15.0\text{ Hz}$, 8.0 Hz, 1H); 8.06 (d, $J = 15.0\text{ Hz}$, 1H); 7.1–7.4 (m, 3H); 7.4–7.7 (m, 2H); 9.33 ppm (d, $J = 8.0\text{ Hz}$, 1H).

1,4-Bis(phenylseleno)-1,3-butadiene (5b):

(Phenylseleno)triethyltriphenylphosphonium bromide (1.40 g, 2.8 mmol) and (*Z*)-3-(phenylseleno)acrylaldehyde (6a; 0.53 g, 2.5 mmol) are dissolved in dichloromethane (4 ml). To this solution is added 50% sodium hydroxide (4 ml) and the mixture is vigorously stirred at room temperature for 30 min. The phases are separated, the organic phase is dried with magnesium sulfate and the solvent is evaporated. The residue is filtered through a short column of silica gel eluting with light petroleum; yield: 0.85 g (93%, isomer ratio not determined; the compound decomposes in the column).

$\text{C}_{16}\text{H}_{14}\text{Se}_2$ calc. C 52.78 H 3.88
(364.2) found 52.45 3.80

IR (Film): $\nu = 1685, 1590, 730, 680, 660\text{ cm}^{-1}$.

$^1\text{H-NMR}$ (CCl_4): $\delta = 6.2\text{--}6.9$ (m, 4H); 7.0–7.7 ppm (m, 10H).

The same procedure is used to prepare 4-phenyl-1-(phenylseleno)-1,3-butadiene (5a) yield: 0.65 g (9%, mixture of 3 isomers, 4:38:58).

5-(Phenylseleno)-2,4-pentadienoic Acid Ethyl Ester (5c):

To a solution of carboxy-methylene-triphenylphosphorane (0.77 g, 2.2 mmol) in dichloromethane (15 ml) is added a solution of (*Z*)-3-(phenylseleno)acrylaldehyde (6a; 0.42 g, 2.0 mmol) in dichloromethane (5 ml). The mixture is stirred under reflux for 7 h. Then the solvent is evaporated and the residue is purified by flash chromatography eluting with dichloromethane/light petroleum (1:3); yield: 0.51 g (90%, a mixture of 3 isomers, 2:19:79).

$\text{C}_{13}\text{H}_{14}\text{O}_2\text{Se}$ calc. C 55.54 H 5.02
(281.2) found 55.52 5.02

IR (Film): $\nu = 1710, 1615, 1580, 790, 735, 690, 670\text{ cm}^{-1}$.

$^1\text{H-NMR}$ (CCl_4): $\delta = 0.95$ (t, 1.43 (t, $J = 8\text{ Hz}$, 3H, mixture of *Z*- and *E*-isomers); 3.80, 4.40 (2 q, $J = 8\text{ Hz}$, 2H, mixture of *Z*- and *E*-isomers); 5.4–7.7 ppm (m, 9H).

2,5-Bis(phenylseleno)-2,4-undecadiene (5d):

To a solution of ethylenetriphenylphosphorane, prepared from ethyltriphenylphosphonium bromide (5.20 g, 14 mmol) in tetrahydrofuran (20 ml) and *n*-butyllithium (6.1 ml of a 2.3 molar solution in hexane, 14 mmol), is added dropwise a solution of phenyl selenenyl bromide (1.66 g, 7 mmol) in tetrahydrofuran (10 ml). The mixture is stirred for 30 min at room temperature and then 3-(phenylseleno)-2-nonenal (6b) (2.1 g, 6 mmol) is added. The formation of a white crystalline precipitate is observed at once. After stirring for 1 h at room temperature, the precipitate is filtered and washed with light petroleum. The filtrate is washed successively with saturated solution of ammonium chloride (30 ml), brine (30 ml), dried with magnesium sulfate and the solvent evaporated. The residue is purified by flash chromatography eluting with hexane; yield: 3.00 g (88%, the isomer ratio not determined, while the compound decomposes in the column).

$\text{C}_{23}\text{H}_{28}\text{Se}_2$ calc. C 59.76 H 6.10
(462.4) found 59.17 6.05

IR (Film): $\nu = 1690, 1610, 1580, 790, 740, 690\text{ cm}^{-1}$.

$^1\text{H-NMR}$ (CCl_4): $\delta = 0.83$ (t, $J = 5\text{ Hz}$, 3H); 1.01–1.8 (m, 8H); 1.9–2.6 (m, 5H); 6.3–6.9 (m, 2H); 7.0–7.7 ppm (m, 10H).

2,5-Undecadiene (7):

To a solution of 2,5-bis(phenylseleno)-2,4-undecadiene (5d; 0.46 g, 1 mmol) in benzene (5 ml) at room temperature is added trifluoroacetic acid (1 ml). After stirring for 1 h at room temperature water (1 ml) is

added and the mixture is stirred for 48 h. Then the mixture is diluted with water (5 ml) and extracted with ether (3 × 10 ml). The organic layer is washed with water (3 × 10 ml), dried with magnesium sulfate and the solvent is evaporated. The residue is distilled at reduced pressure; yield: 0.13 g (70%); b.p. 55°C/0.05 torr (Lit.¹⁸, b.p. 93°C/2 torr). ¹H-NMR and IR spectra are in accordance with the literature.¹⁸

(Z)-1,1,4-Tris(phenylseleno)-3-nonene-2-ol (9):

To a solution of bis(phenylseleno)methane (0.65 g, 2.0 mmol) in tetrahydrofuran (10 ml) at -78°C is added lithium diisopropylamide [2.2 mmol, prepared from a 2.2 molar hexane solution of *n*-butyllithium (1 ml) and diisopropyl amine (0.22 g, 2.2 mmol)] over a period of 30 min. Then (Z)-3-(phenylseleno)-2-octenal (6b; 0.56 g, 2.0 mmol) in tetrahydrofuran (5 ml) is added at -78°C. A solution of saturated ammonium chloride (4 ml) is added at the same temperature and the mixture is stirred for 5 min, heated to room temperature and diluted with ether (20 ml). The phases are separated, the organic layer is dried with magnesium sulfate and the solvent is evaporated. The product is purified by flash chromatography eluting with light petroleum/ethyl acetate (1:1); yield: 1.15 g (95%).

C₂₇H₃₀OSe calc. C 53.41 H 4.98
(449.5) found 53.48 5.01

IR (Film): ν = 3440, 1620, 785, 740, 690, 670 cm⁻¹.

¹H-NMR (CCl₄): δ = 0.77 (t, *J* = 5 Hz, 3 H); 0.9–1.7 (m, 6 H); 1.9–2.3 (m, 2 H); 3.1 (br s, 1 H); 4.50 (d, *J* = 3 Hz, 1 H); 4.90 (dd, *J* = 3 Hz, 7 Hz, 1 H); 6.07 (d, *J* = 7 Hz, 1 H); 6.9–7.7 ppm (m, 15 H).

(E)-1,4-Dioxo-2-nonene (10):

To a solution of 1,1,4-tris(phenylseleno)-3-nonene-2-ol (9; 0.61 g, 1.0 mmol) in acetonitrile/water (4:1) (20 ml) is added mercuric chloride (0.87 g, 3.2 mmol) in the same mixture of solvents (30 ml). The mixture is stirred for 24 h at room temperature. The crystalline precipitate is filtered through Celite and washed with ether (20 ml). The filtrate is washed successively with saturated solution of sodium hydrogen carbonate and brine. The organic layer is dried with magnesium sulfate and the solvent is evaporated. The residue is purified by flash chromatography eluting with benzene/ether (9:1).
yield: 0.10 g (79%).

¹H-NMR and IR data are in accordance with the literature.¹⁹

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