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Treatment of 2-*tert*-butyl-1-benzoselenopyrylium salts **1A** and 2-phenyl-1-benzoselenopyrylium salts **1B** with an alkyl(phenyl)magnesium halide resulted in nucleophilic addition at the C-4 position to give the corresponding 2,4-disubstituted 4*H*-selenochromenes **2A** and **2B** in good yields, respectively. The obtained selenochromenes **2** were then easily converted into the 4-substituted 2-*tert*-butyl-1-benzoselenopyrylium salts **6A** by treatment with triphenylcarbenium tetrafluoroborate in high yields. The 4-substituted 2-phenyl derivatives **6B** were also obtained in a similar manner. The reaction of the unsubstituted 1-benzoselenopyrylium salt **1C** with an alkylmagnesium halide is also described.

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Research on thiopyrylium salts [2], six-membered heteroaromatic cations containing a sulfur, has attracted considerable attention in recent years due to their difference from pyrylium salts [3] in characteristic properties and variegated reactivity. In spite of the impressive development of both chemistries of the pyrylium salts and thiopyrylium salts, only a small effort on the synthesis of their selenium analogues, the selenopyrylium salts, [4] have been made. With regard to the 1-benzoselenopyrylium salts, a few perchlorate derivatives [5,6,7] were known; only one 2,4-diphenyl substituted selenopyrylium salt [6] was prepared as the 1-benzoselenopyrylium salts having two carbon functional groups. In addition, the reaction [7] of the unsubstituted 1-benzoselenopyrylium salt with phenylmagnesium bromide as the Grignard reagent to produce the 2-phenyl- or 4-phenyl-selenochromene has scarcely been examined. To the best of our knowledge, no 2- and/or 4-(di)alkyl substituted derivatives have been known until our study.

We have been deeply interested in the chemistry [8-13] (syntheses, structure and physical properties and reactions) of the telluropyrylium salts and selenopyrylium salts and related compounds in recent years. Previously, we described the facile preparation [12] of the 1-benzoselenopyrylium salts including the 2-alkyl-1-benzoselenopyrylium salts and their reactions [13] with several nucleophiles to give the 4-functionalized 4*H*-selenochromenes

in good to excellent yields. Therefore, this report describes the practical synthetic route for the 4*H*-selenochromenes having carbon functional groups at the C-2 and C-4 positions by the reaction of the selenopyrylium salts with the Grignard reagents, and the conversion of the obtained selenochromenes into the corresponding 1-benzoselenopyrylium salts.

The reaction of the 2-*tert*-butyl-1-benzoselenopyrylium tetrafluoroborate **1A** [12] and 2-phenyl-1-benzoselenopyrylium tetrafluoroborate **1B** [12] with 1.2 equivalents of methylmagnesium iodide in diethyl ether at 0 °C resulted in nucleophilic addition at the C-4 position to produce the 4-methyl-4*H*-selenochromenes **2Aa** and **2Ba** in 63 and 53% yields, respectively. 4-Ethyl-4*H*-selenochromenes **2Ab**, **2Bb**, 4-benzyl-4*H*-selenochromenes **2Ac**, **2Bc** and 4-phenyl-4*H*-selenochromene **2Ad** were similarly obtained by treatment with the corresponding Grignard reagents in good to high yields. Their regioisomers, the 4-substituted 2*H*-selenochromenes have never been produced. 2,4-Diphenyl-4*H*-selenochromene **2Bd** has already been synthesized by the reaction of 2-phenylselenopyrylium perchlorate [5] with phenylmagnesium bromide, however, there have been no reports on the successful synthesis of selenochromenes carrying two general carbon functional groups on the selenochromene ring until this study. The results and the spectral data of the products are summarized in Table 1.

On the other hand, the reaction of the parent selenopyrylium salt **1C** with the alkylmagnesium bromide resulted in a nucleophilic reaction at both the C-2 and C-4 positions as shown in Scheme 2. The salt **1C** was treated with methylmagnesium iodide to afford 4-methyl-4*H*-selenochromene **3a**, 2-methyl-2*H*-selenochromene **4a** and 2-methyl-4*H*-selenochromene **5** in 12, 6 and 5% yields, respectively. When ethylmagnesium bromide was used as the Grignard reagent, the salt **1C** gave the 4-ethyl-4*H*-chromene **3b** (11% yield) and 2-ethyl-2*H*-chromene **3b** (24% yield); the isomeric product of the latter was not

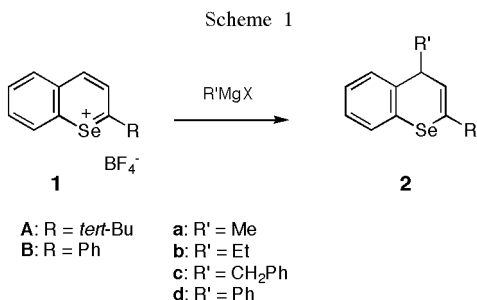
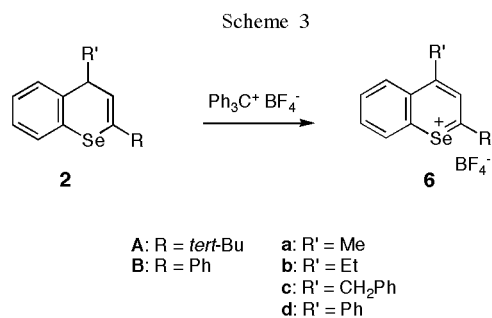
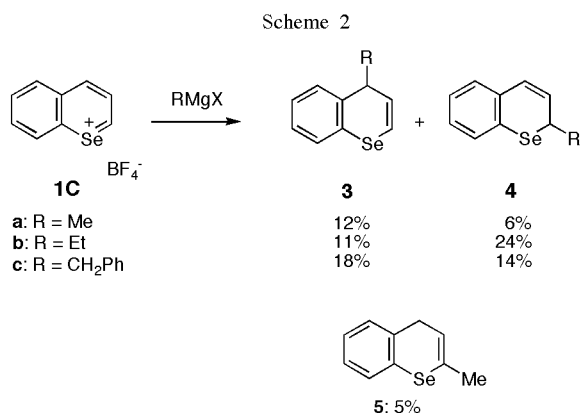


Table 1
4-Substituted 2-*tert*-Butyl-4*H*-selenochromenes **2A** and 2-Phenyl-4*H*-selenochromenes **2B**

Compd. No.	Appearance Yield (%)	Formula HRMS Calcd (Found)	¹ H NMR (90 MHz, CDCl ₃ , J = Hz)				
			3-H	4-H	R-H	Ph-H (5-, 6-, 7-, 8-H)	R'-H
2Aa	yellow oil 63	C ₁₄ H ₁₈ Se 266.0574 (266.0571)	5.83 (d, J = 5)	3.05 (dq, J = 7, 5)	1.20 (9H, s) <i>tert</i> -Bu	6.9-7.3, 7.4-7.6 (3H, m, 1H,m)	1.48 (3H, d, J =7) Me
2Ba	yellow oil 53	C ₁₆ H ₁₄ Se 286.0261 (286.0256)	6.24 (d, J = 5)	3.26 (dq, J = 7, 5)		7.0-7.5 (9H, m) R = Ph	1.46 (3H, d, J =7) Me
2Ab	colorless oil 65	C ₁₅ H ₂₀ Se 280.0731 (280.0732)	5.90 (d, J = 6)	3.06 (dt, J = 7, 6)	1.19 (9H, s) <i>tert</i> -Bu	7.1-7.4, 7.5-7.7 (3H, m, 1H, m)	1.03, 1.88 (3H, dt, J = 7, 7, 2H, q, J = 7) Et
2Bb	yellow oil 52	C ₁₇ H ₁₆ Se 300.0418 (300.0425)	6.43 (d, J = 6)	3.43 (dt, J = 7, 6)		7.1-7.7 (9H, m) R = Ph	1.00, 1.85 (3H, dt, J = 7, 7, 2H, q, J = 7) Et
2Ac	yellow oil 81	C ₂₀ H ₂₂ Se 342.0888 (342.0816)	5.88 (d, J = 7)	3.70 (ddd, J = 7, 7, 8)	1.15 (9H, s) <i>tert</i> -Bu	7.0-7.6 (9H, m) R' = CH ₂ Ph	2.69, 3.12 (1H, dd,J = 8, 12, 1H, dd, J = 7, 12) R' = CH ₂ Ph
2Bc	yellow oil 73	C ₂₂ H ₁₈ Se 362.0575 (362.0512)	6.31 (d, J = 7)	3.95 (ddd, J = 7, 7, 9)		7.1-7.6 (14H, m) R = Ph, R' = CH ₂ Ph	2.95, 3.12 (1H, dd, J = 9, 13, 1H, dd, J = 7, 13) R' = CH ₂ Ph
2Ad	colorless prisms mp 108-109 °C 86	C ₁₉ H ₂₀ Se 328.0731 (328.0715)	6.13 (d, J = 5)	4.18 (d, J = 5)	1.23 (9H, s) <i>tert</i> -Bu		6.6-7.6 (9H, m)

obtained in this case. A similar reaction of the salt **1C** with benzylmagnesium bromide proceeded to give the 4-benzyl **3c** and 2-benzyl **4c** derivatives in 18 and 14% yields, respectively. The preparation of the 2-substituted selenochromenes has been achieved by the reduction of the corresponding selenochromon-4-ones [14] with diisobutylaluminum hydride or lithium aluminum hydride reduction of 2-alkyl-1-benzoselenopyrylium salts, [13] however, the 4-substituted derivatives could not be prepared using the method. All the selenochromenes **2A**, **2B**, **3** and **4** except for **5** are hitherto unknown compounds except **2Bd**.



Next, in order to obtain the 1-benzoselenopyrylium salts having carbon functional groups at both the C-2 and C-4 positions, the reaction of the selenochromenes **2A** with triphenylcarbenium tetrafluoroborate (Ph₃C⁺ BF₄⁻) was carried out. Treatment of 2-*tert*-butyl-4-methyl-selenochromene **2Aa** with 1.1 equivalents of Ph₃C⁺ BF₄⁻ in nitromethane at room temperature afforded the desired 2-*tert*-butyl-4-methyl-1-benzoselenopyrylium tetrafluoroborate **6Aa** in 80% yield as stable green prisms. Application of the synthesis for the other 2,4-disubstituted 1-benzoselenopyrylium salt **6** was also successful. All salts **6a**, **6b**, **6c** and **6d** were isolated, quite stable and not sensitive to air and light; but readily decomposed when in contact with a protic solvent such as water and methanol. The

results and the spectral data of the products **6** were listed in Table 2. 2,4-Diphenyl-1-benzoselenopyrylium salt have been obtained, but it is as the perchlorate, and no ^{13}C nmr spectral data for the known compound was given in the literature [5], so ^1H nmr and ^{13}C nmr spectral data of **6Bd** are included in Table 2.

have also observed that the 1-benzyl-2-benzotelluropyrylium salts **9** [8] could be isolated and exist in a solvent as an equilibrium mixture of the salts **9** and the 1-benzylidene compounds **10**, which are the β -hydrogen eliminated compounds of the salts **9**. Thus, we suspect that the δ -hydrogen elimination of the 4-benzyl-1-benzoselenopyrylium salts

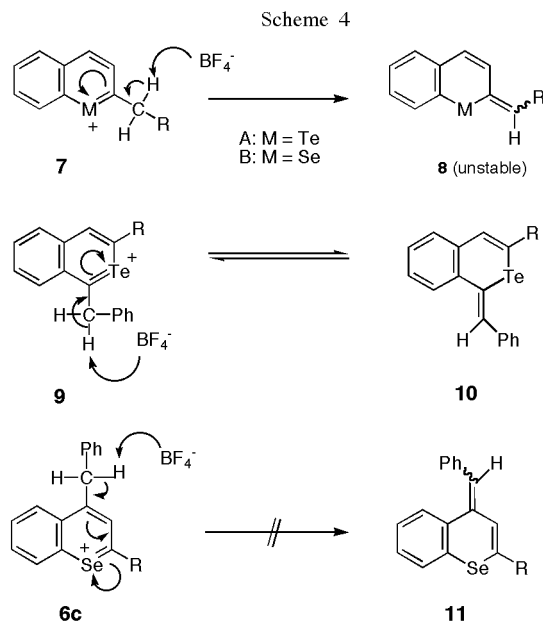
Table 2
2,4-Disubstituted 1-Benzoselenopyrylium Salts **6**

Compd. No	Appearance Yield (%)	Formula Analysis		IR ν BF ₄ ⁻ (cm ⁻¹)	¹ H NMR (400 MHz, CD ₃ CN, J = Hz)				¹³ C NMR (100 MHz, CD ₃ CN)
		Calcd C	(Found) H		3-H	R-H	Ph-H (5-, 6-, 7-, 8-H)	R ¹ -H	
6Aa	green prisms mp 176-178 °C 80	C ₁₄ H ₁₇ BF ₄ Se		1084	8.72 (s)	1.70 (9H, s)	8.12-8.16, 8.68-8.71, 8.94-8.97 (2H, m, 1H, <i>tert</i> -Bu m, 1H, m)	3.12 (3H, s) Me	26.06 (q), 31.05 (q), 45.80 (s), 131.38 (d), 132.24 (s), 132.64 (d), 133.71 (d), 133.82 (d), 134.61 (d), 148.79 (s), 168.04 (s), 209.79 (s)
		47.90 (47.60)	4.88 (4.74)						
6Ba	yellow prisms mp 161-164 °C 75	C ₁₆ H ₁₃ BF ₄ Se		1087	8.84 (s)	7.71-7.75, 7.83-7.87, 8.07-8.15, 8.68-8.70, 8.95-8.97 (2H, m, 1H, m, 4H, m, 1H, m, 1H, m)	3.16 (3H,s) Me	26.29 (q), 129.86 (d), 131.30 (d), 131.69 (d), 132.49 (s), 133.09 (d), 133.41 (d) 133.74 (d), 135.01 (d), 136.10 (d), 137.97 (s), 149.24 (s), 168.71 (s), 188.58 (s)	
		51.79 (51.75)	3.53 (3.67)						
6Ab	yellow prisms mp 154-155 °C 95	C ₁₅ H ₁₉ BF ₄ Se		1084	8.70 (s)	1.70 (9H, s)	8.13-8.18, 8.71-8.73, 8.99-9.01 (2H, m, 1H, <i>tert</i> -Bu m, 1H, m)	1.51, 3.55 (3H, t, J = 7.6, 2H, q, J = 7.6) Et	15.80 (q), 31.13 (q), 32.38 (t), 45.84 (s), 131.38 (s), 131.70 (d), 132.74 (d), 133.79 (d), 134.59 (d), 149.66 (s), 173.35 (s), 210.02 (s)
		49.35 (49.03)	5.25 (5.10)						
6Bb	yellow prisms mp 131-132 °C 90	C ₁₇ H ₁₅ BF ₄ Se		1084	8.81 (s)	7.65-7.79, 7.81-7.90, 8.08-8.15, 8.70, 9.00 (3H, m, 1H, m, 3H, m, 1H, d, J = 8.0, 1H, d, J = 8.0)	1.53, 3.57 (3H, t, J = 7.6, 2H, q, J = 7.6) Et	15.63 (t), 32.52 (q), 129.80 (d), 130.51 (s), 131.54 (d), 131.62 (d), 132.32 (d), 132.72 (d), 133.77 (d), 134.93 (d), 136.01 (d), 137.95 (s), 149.90 (s), 173.87 (s), 188.75 (s)	
		53.02 (53.28)	3.93 (4.06)						
6Ac	yellow prisms mp 113-115 °C 79	C ₂₀ H ₂₁ BF ₄ Se		1033	8.67 (s)	1.66 (9H, s)	7.17-7.38, 8.06-8.14, 8.69-8.73, 8.97-9.03 <i>tert</i> -Bu (5H, m, 2H, m, 1H, m, 1H, m) R' = CH ₂ Ph	4.87 (2H, s) CH ₂ Ph	31.01 (q), 43.83 (t), 46.02 (s), 128.39 (d), 130.02 (d), 130.12 (d), 131.65 (s), 131.72 (d), 132.74 (d), 133.85 (d), 133.88 (d), 134.53 (d), 138.28 (s), 149.89 (s), 168.13 (s), 210.76 (s)
		56.24 (56.04)	4.50 (4.75)						
6Bc	yellow prisms mp 125-127 °C 84	C ₂₂ H ₁₇ BF ₄ Se		1080	8.78 (s)	7.26-7.43, 7.68-7.72, 7.82-7.86, 8.01-8.10, 8.66-8.70, 8.97-8.99 (5H, m, 2H, m, 1H, m, 3H, m, 1H, m, 1H, m) R' = CH ₂ Ph	4.89 (2H, s) CH ₂ Ph	44.02 (t), 128.42 (d), 129.51 (d), 129.88 (d), 130.09 (d), 130.14 (d), 131.59 (d), 131.74 (d), 133.17 (d), 133.43 (d), 133.85 (d), 134.88 (d), 136.27 (d), 138.05 (s), 138.32 (s), 150.22 (s), 168.67 (s), 189.46 (s)	
		59.10 (59.15)	3.83 (3.86)						
6Ad	yellow prisms mp 150-151 °C 78	C ₁₉ H ₁₉ BF ₄ Se		1084	8.60 (s)	1.72 (9H, s)	7.70-7.77, 8.00-8.07, 8.15-8.19, 8.56, 8.80 <i>tert</i> -Bu (5H, m, 1H, m, 1H, m, 1H, d, J = 8.3, 1H, d, J = 8.3) Ph	31.23 (q), 46.09 (s), 130.10 (d), 130.93 (d), 131.32 (d), 133.04 (d), 133.69 (d), 134.61 (d), 135.55 (d), 139.57 (s), 150.60 (s), 154.54 (s), 167.05 (s), 209.63 (s)	
		55.24 (55.09)	4.64 (4.36)						
6Bd	orange prisms mp 191-197 °C 87	C ₂₁ H ₁₅ BF ₄ Se		1058	8.74 (s)	7.69-7.79, 7.84-7.88, 8.02-8.06, 8.10-8.18, 8.81, 8.57 (7H, m, 1H, m, 1H, m, 3H, m, 1H, d, J = 8.5, 1H, d, J = 8.5) Ph	130.06 (d), 130.18 (d), 131.07 (d), 131.25 (d), 131.69 (d), 131.74 (d), 132.30 (d), 133.01 (d), 133.71 (d), 134.98 (d), 136.10 (d), 136.18 (d), 138.18 (s), 139.68 (s), 151.00 (s), 167.65 (s), 188.41 (s)		
		58.23 (57.91)	3.49 (3.64)						

It has been found that BF_4^- , the counter anion of the 1-benzotelluropyrylium salts **7A** [11] and 1-benzoselenopyrylium salts **7B** [12] having a primary alkyl group at the C-2 position, abstracts the β -hydrogen of the methylene carbon of the alkyl group forming the unstable *exo*-methylidene compounds **8A** and **8B**. Furthermore, we

6c proceeds to form the 4-benzylideneselenochromenes **11**. However, the 4-benzyl derivatives **6c** could be isolated as the stable crystalline products; the δ -hydrogen elimination did not occur.

In conclusion, the present results provide a facile synthetic route for the preparation of the selenochromenes having gen-



eral carbon functional groups at the C-2 and C-4 positions and their conversion into the corresponding 1-benzoselenopyrylium salts. Further studies on not only the details and utility of these selenopyrylium salts, but also the synthesis of the other functionalized salts are now in progress.

EXPERIMENTAL

General Methods.

Melting points were measured on a Yanagimoto micro melting point hot stage apparatus and are uncorrected. The IR spectra were recorded on a Hitachi 270-30 spectrometer. Mass and high resolution mass spectra were recorded on a JEOL JMS-DX300 instrument. ^1H NMR spectra were determined with a PMX-60SI (60 MHz), JEOL EX-90A (90 MHz) or JEOL JNM-GSX 400 (400 MHz) spectrometer in deuteriochloroform or deuterioacetonitrile using tetramethylsilane as an internal standard and J values are given in Hz. Microanalyses were performed in the Microanalytical Laboratory of this Faculty.

Reaction of 1-Benzoselenopyrylium Salts **1** with Methylmagnesium Bromide.

Methylmagnesium bromide (1.2 mmol) in ether solution (2 mL) was slowly added to a suspended mixture of the 1-benzoselenopyrylium salt **1** (1 mmol) in ether (5 mL) at 0 °C under an argon atmosphere. The resulting mixture was stirred at room temperature for 1 hour until the disappearance of the starting selenopyrylium salt, and quenched by the addition of saturated aqueous ammonium chloride solution (10 mL). The resulting mixture was extracted with diethyl ether (30 mL x 3). The organic extracts were washed with brine, dried over magnesium sulfate and evaporated *in vacuo*. The residue was chromatographed on silica gel, with *n*-hexane as an eluent to give **2a**.

Reaction of 1-Benzoselenopyrylium salts **1** with Ethylmagnesium Bromide Salts.

The selenopyrylium salt **1** was treated with ethylmagnesium bromide instead of methylmagnesium bromide and worked up as described for the preparation of **2a** to give **2b**.

Reaction of 1-Benzoselenopyrylium Salts **1** with Benzylmagnesium Chloride.

The selenopyrylium salt **1** was treated with benzylmagnesium chloride instead of methylmagnesium bromide and worked up as described for the preparation of **2a** to give **2c**.

Reaction of 1-Benzoselenopyrylium Salts **1** with Phenylmagnesium Bromide.

The selenopyrylium salt **1** was treated with phenylmagnesium bromide instead of methylmagnesium bromide and worked up as described for the preparation of **2a** to give **2d**. Crystalline products **2Ad** and **2Bd** were recrystallized from acetone - *n*-hexane.

Reaction of 1-Benzoselenopyrylium Salts **1C** with Methylmagnesium Bromide.

The selenopyrylium salt **1C** was treated with methylmagnesium bromide instead of methylmagnesium bromide and worked up as described for the preparation of **2a** to give **3a**, **4a** and **5**.

4-Methyl-4H-selenochromene (**3a**).

Compound **3a** (25 mg) was obtained in 12% yield as a colorless oil; ^1H NMR (90 MHz, deuteriochloroform): 1.42 (3H, d, $J = 7$ Hz, 4-Me), 3.30 (1H, dq, $J = 7, 6$ Hz, 4-H), 6.27 (1H, dd, $J = 6, 8$ Hz, 3-H), 6.87 (1H, d, $J = 8$ Hz, 2-H), 7.1-7.6 (4H, m, phenyl protons); high resolution mass m/z : M^+ calcd for $\text{C}_{10}\text{H}_{10}\text{Se}$, 209.9948; found, 209.9947.

2-Methyl-2H-selenochromene (**4a**).

Compound **4a** (13 mg) was obtained in 6% yield as a yellow oil; ^1H NMR (90 MHz, deuteriochloroform): 1.52 (3H, d, $J = 7$ Hz, 2-Me), 3.81 (1H, dq, $J = 7, 5$ Hz, 2-H), 5.72 (1H, dd, $J = 5, 11$ Hz, 3-H), 6.41 (1H, d, $J = 11$ Hz, 4-H), 6.9-7.5 (4H, m, phenyl protons); high resolution mass m/z : M^+ calcd for $\text{C}_{10}\text{H}_{10}\text{Se}$, 209.9948; found, 209.9951.

2-Methyl-4H-selenochromene (**5**).

Compound **5** (11 mg) was obtained in 5% yield as a yellow oil. This compound was identical with authentic sample [12].

Reaction of Selenopyrylium Salts **1C** with Ethylmagnesium Bromide.

The selenopyrylium salt **1C** was treated with ethylmagnesium bromide instead of methylmagnesium bromide and worked up as described for the preparation of **2a** to give **3b** and **4b**.

4-Ethyl-4H-selenochromene (**3b**).

Compound **3b** (25 mg) was obtained in 11% yield as a yellow oil; ^1H NMR (90 MHz, deuteriochloroform): 0.90 and 1.69 (3H, t, $J = 7$ Hz, 2H, dq, $J = 7, 7$ Hz, 4-Et), 3.27 (1H, dt, $J = 7, 7$ Hz, 4-H), 6.30 (1H, dd, $J = 6, 8$ Hz, 3-H), 6.80 (1H, d, $J = 8$ Hz, 2-H), 6.9-7.5 (4H, m, phenyl protons); high resolution mass m/z : M^+ calcd for $\text{C}_{11}\text{H}_{12}\text{Se}$, 224.0104; found, 224.0097.

2-Ethyl-2H-selenochromene (**4b**).

Compound **4b** (54 mg) was obtained in 24% yield as a yellow oil; ^1H NMR (90 MHz, deuteriochloroform) 0.97 and 1.77 (3H, t, $J = 7$ Hz, 2H, dq, $J = 7, 7$ Hz, 2-Et), 3.57 (1H, dt, $J = 7, 6$ Hz, 2-H), 5.75 (1H, dd, $J = 6, 10$ Hz, 3-H), 6.45 (1H, d, $J = 10$

Hz, 2-H), 6.9-7.5 (4H, m, phenyl protons); high resolution mass m/z : M^+ calcd for $C_{11}H_{12}Se$, 224.0104; found, 224.0099.

Reaction of Selenopyrylium Salts **1C** with Benzylmagnesium Bromide.

The selenopyrylium salt **1C** was treated with benzylmagnesium bromide instead of methylmagnesium bromide and worked up as described for the preparation of **2a** to give **3c** and **4c**.

4-Benzyl-4H-selenochromene (**3c**).

Compound **3c** (52 mg) was obtained in 18% yield as a yellow oil; 1H NMR (90 MHz, deuteriochloroform): 2.93 (2H, d, $J = 8$ Hz, CH_2Ph), 3.73 (1H, dt, $J = 6, 8$ Hz, 4-H), 6.24 (1H, dd, $J = 6, 10$ Hz, 3-H), 6.89 (1H, d, $J = 10$ Hz, 2-H), 6.9-7.5 (9H, m, phenyl protons); high resolution mass m/z : M^+ calcd for $C_{16}H_{14}Se$, 286.0261; found, 286.0240.

2-Benzyl-2H-selenochromene (**4c**).

Compound **4c** (40 mg) was obtained in 14% yield as a yellow oil; 1H NMR (90 MHz, deuteriochloroform): 2.69 and 3.33 (each 1H, d, $J = 8$ Hz, CH_2Ph), 3.90 (1H, ddd, $J = 6, 8, 8$ Hz, 2-H), 5.67 (1H, dd, $J = 6, 11$ Hz, 3-H), 6.40 (1H, d, $J = 11$ Hz, 4-H), 7.0-7.5 (9H, m, phenyl protons); high resolution mass m/z : M^+ calcd for $C_{16}H_{14}Se$, 286.0261; found, 286.0236.

General Procedure for the Synthesis of the 2,4-Disubstituted 1-Benzoselenopyrylium Salts **6**.

Triphenylcarbenium tetrafluoroborate (3.63 g, 11 mmol) was added to a stirred solution of the selenochromene **2A**, **B** (10 mmol) in dry nitromethane (20 mL) and the mixture was stirred at room temperature for 30 minutes. To the reaction mixture was added dry diethyl ether (ca. 200 mL) to precipitate the selenopyrylium salt **6**. All salts **6** were recrystallized from chloroform.

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