Supporting Information

Synthesis of Difluoromethylselenoesters from Aldehydes via a Radical Process

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1. General information

All reactions were carried out underone atmosphere. All reagents were used as received unless otherwise noted. Flash chromatography was performed with silica gel (200-300 mesh). NMR spectra were recorded on a Bruker Ascend 400 spectrometer at 400 MHz (¹H NMR), 101 MHz (13C NMR), 376 MHz (19F NMR), on a BrukerAscend 600 spectrometer at 600 MHz (1H NMR), 151 MHz (¹³C NMR) or on a JEOL ECZ400R spectrometer at 376 MHz (¹⁹F NMR). ¹H NMR chemical shifts are reported in delta (δ) units, in parts per million (ppm) downfield from tetramethylsilane. Splitting patterns are designated as s, singlet; d, doublet; t, triplet; m, multiplet, br, broad. Coupling constants J are quoted in Hz. Spectra are referenced internally to the residual proton resonance in CDCl₃ (δ 7.26 ppm), or with tetramethylsilane (TMS, δ 0.00 ppm) as the internal standard. ¹³C NMR chemical shifts are reported in ppm relative to the center line of a triplet at 77.16 ppm for chloroform-d. ¹⁹F NMR chemical shifts are reported relative to inter standard CFCl₃ at 0.0 ppm. Infrared (IR) data were recorded as films on potassium bromide plates on a Bruker Tensor 27 FT-IR spectrometer. Absorbance frequencies are reported in reciprocal centimeters (cm⁻¹). Mass spectra were acquired on a BrukerDaltonics S2 MicroTof-Q II mass spectrometer. X-ray crystal structure analyses were measured on Bruker Smart APEXIICCD instrument using Mo-K α radiation. The structures were solved and refined using the SHELXTL software package. 2a,¹ 2b,¹ 2c, 2d,² 1ad,³ 1ae,⁴ 1ca,⁵ 1cb,⁶ 1cc⁷were prepared according to literature methods.

2. General procedure

Synthesis of **2b**: To a dry two-necked 100 mL flask bottomed flask equipped with a magnetic stirrer were added(benzylselanyl)carbonitrile (15.76 g, 80 mmol, 1.0 equiv). The flask was

evacuated and refilled with argonthree times. Then, anhydrous DMF (40 mL), TMSCF₂H (20 mL, 160 mmol, 2.0 equiv) were sequentially added to thesystem via syringe. The reaction mixture was cooled to 0 °C, and CsF (36.45 g,240 mmol, 3.0 equiv) was carefully added. The reaction was stirred at 0 °C under an argon atmosphere for 72 h. The reaction mixture was then partitioned between water and pentane. The layers wereseparated and the organic layer was washed with aqueoussaturated brine solution, and then dried over MgSO₄. After removing the pentane by rotary evaporation, theresidue was purified by flash chromatography on silica gel to give **2b** as a colorless oil.

Synthesis of **2c**: To a flask equipped with a magnetic stir bar were added the benzyl(difluoromethyl)selane (2.21g, 10 mmol, 1.0 equiv.), sulfuryl chloride (0.8 mL, 10 mmol, 1.0 equiv.) and anhydrous THF (5 mL). The reaction mixture was stirred at 0 °C for 1h and then cooled down to -78 °C. Anhydrous DCM (25 mL) was added followed by sodium benzenesulfinate (1.81g, 11 mmol, 1.1 equiv.). The reaction was stirred until complete conversion of the active reagent ClSeCF₂H at -78 °C. The reaction mixture was then filtered over a pad of silica (rinsed with DCM) and the filtrate was concentrated to dryness. The crude residue was purified by chromatography to afford the desired product **2c** as a yellow oil.

For **3a-3cc**: A 10 mL round bottomed flask equipped with a stirring bar was charged with benzyl(difluoromethyl)selane (121 mg, 0.5 mmol, 1.0 equiv), aldehydes (0.75 mmol, 1.5 equiv), and DCE (2.5 mL) followed by sequential addition of AIBN (164 mg, 1.0 mmol, 2.0 equiv). The reaction was allowed to stir at 50 °C under an argon atmosphere for 24 h. The solvent was filtered and the filtrate was evaporated in vacuo. The residue was purified by flash chromatography on silica gel.

3. Characterization datas for compounds 2b – 3cc



Benzyl(difluoromethyl)selane (2b). Colorless oil (36%).Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.40$). ¹H NMR (400 MHz, CDCl₃) δ 7.34 – 7.23 (m, 5H), 7.05 (t, J = 55.2 Hz, 1H), 4.09 (s, 2H); ¹⁹F NMR (376 MHz, CDCl₃) δ -92.88 (d, J = 55.2 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 137.4, 129.1 (2C), 128.9 (2C), 127.5, 115.8 (t, J = 287.0 Hz), 26.4 (t, J = 2.9 Hz). IR (KBr): v = 3030, 1495, 1454, 1295, 1191, 1059, 911, 759, 697, 607 cm⁻¹. HRMS (ESI) for $C_8H_8F_2SeNa+$ (M+Na⁺): Calcd: 244.9651, Found: 244.9652.



Se-(difluoromethyl)-benzenesulfonoselenoate (2c). Yellow oil (65%).Eluant: ethyl acetate/petroleum ether (1:50, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.91 (d, J = 7.7 Hz, 2H), 7.68 (t, J = 7.4 Hz, 1H), 7.64 (t, J = 54.2 Hz, 1H), 7.58 (t, J = 7.8 Hz, 2H); ¹⁹F NMR (376 MHz, CDCl₃) δ -90.44 (d, J = 54.1 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 147.9, 134.6, 129.7 (2C), 126.6 (2C), 120.6 (t, J = 293.7 Hz). IR (KBr): v = 2926, 1448, 1329, 1138, 1064, 906, 681, 650, 577, 528 cm⁻¹. HRMS (ESI) for C₇H₆F₂O₂SSeNa+ (M+Na⁺): Calcd: 294.9114, Found: 294.9113.



Se-(difluoromethyl)-4-methylbenzoselenoate (3a). Yellow solid (113 mg, 90%). Mp: 46-47 °C. Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.71 (d, *J* = 8.2 Hz, 2H), 7.61 (t, *J* = 53.6 Hz, 1H), 7.30 (d, *J* = 8.1 Hz, 2H), 2.43 (s, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.08 (d, *J* = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 190.5 (t, *J* = 2.8 Hz), 146.3, 135.4 (t, *J* = 2.5 Hz), 130.0 (2C), 127.9 (2C), 120.4 (t, *J* = 283.3 Hz), 22.0. IR (KBr): v = 2922, 1690, 1603, 1456, 1269, 1177, 1051, 816, 621 cm⁻¹. HRMS (ESI) for C₉H₈F₂OSeNa (M+Na⁺): Calcd: 272.9601, Found: 272.9598.



Se-(difluoromethyl)-4-ethylbenzoselenoate (3b). Yellow oil (109 mg, 82%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (600 MHz, CDCl₃) δ 7.73 (d, J = 8.2 Hz, 2H), 7.61 (t, J = 53.6 Hz, 1H), 7.32 (d, J = 8.2 Hz, 2H), 2.72 (q, J = 7.6 Hz, 2H), 1.27 (t, J = 7.6 Hz, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.09 (d, J = 53.6 Hz, 2F); ¹³C NMR (151 MHz, CDCl₃) δ 190.5(t, J = 4.2 Hz), 152.4, 135.6(t, J = 2.5 Hz), 128.9 (2C), 128.0 (2C), 120.4 (t, J = 283.2 Hz), 29.2, 15.1. IR (KBr): v = 2926, 1696, 1604, 1270, 1210, 1176, 1073, 880, 842, 767, 691, 611 cm⁻¹. HRMS (ESI) for C₁₀H₁₀F₂OSeNa (M+Na⁺): Calcd: 286.9757, Found: 286.9760.



Se-(difluoromethyl)-4-isopropylbenzoselenoate (3c). Yellow oil (102 mg, 73%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.74 (d, J = 8.3 Hz, 2H), 7.61 (t, J = 53.6 Hz, 1H), 7.35 (d, J = 8.3 Hz, 2H), 3.20 – 2.76 (m, 1H), 1.27 (d, J = 6.9 Hz, 6H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -96.08 (d, J = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 190.5 (t, J = 2.9 Hz), 157.0, 135.7 (t, J = 2.6 Hz), 128.0 (2C), 127.5 (2C), 120.4(t, J = 283.3 Hz), 34.6, 23.7 (2C). IR (KBr): v = 2962, 2925, 1690, 1603, 1463, 1414, 1269, 1210, 1179, 1055, 875, 841, 747, 689, 626, 610 cm⁻¹. HRMS (ESI) for C₁₁H₁₂F₂OSeNa (M+Na⁺): Calcd: 300.9914, Found: 300.9907.



Se-(difluoromethyl)-4-isobutylbenzoselenoate (3d). Yellow oil (135 mg, 92%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.72 (d, *J* = 8.3 Hz, 2H), 7.61 (d, *J* = 53.6 Hz, 1H), 7.27 (d, *J* = 8.1 Hz, 2H), 2.54 (d, *J* = 7.2 Hz, 2H), 1.96 – 1.85 (m, 1H), 0.91 (d, *J* = 6.6 Hz, 6H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.08 (d, *J* = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 190.5 (t, *J* = 2.9 Hz), 150.0, 135.6 (t, *J* = 2.6 Hz) 130.0 (2C), 127.8 (2C), 120.4(t, *J* = 283.3 Hz), 45.6, 30.2, 22.4 (2C). IR (KBr): v = 2957, 2925, 1687, 1604, 1466, 1413, 1270, 1207, 1176, 1071, 879, 818, 792, 691, 652, 623 cm⁻¹. HRMS (ESI) for C₁₂H₁₄F₂OSeNa (M+Na⁺): Calcd: 315.0070, Found: 315.0070.



Se-(difluoromethyl)-4-(tert-butyl)benzoselenoate (3e). Yellow solid (130 mg, 89%). Mp: 58-59 °C. Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, J = 8.3 Hz, 2H), 7.62 (t, J = 53.6 Hz, 1H), 7.51 (d, J = 8.4 Hz, 2H), 1.35 (s, 9H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.08 (d, J = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 190.5 (t, J = 2.9 Hz), 159.2, 135.2 (t, J = 2.6 Hz), 127.7 (2C), 126.3 (2C), 120.4 (t, J = 283.2 Hz), 35.5, 31.1 (3C). IR (KBr): $\nu = 2965$, 1683, 1597, 1267, 1215, 1180, 1106, 1072, 1050, 881, 721, 690, 590 cm⁻¹. HRMS (ESI) for C₁₂H₁₄F₂OSeNa (M+Na⁺): Calcd: 315.0070, Found: 315.0071.



Se-(difluoromethyl)-4-chlorobenzoselenoate (3f). Yellow solid (103 mg, 76%). Mp: 35-36 °C. Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, *J* = 8.7 Hz, 2H), 7.61 (t, *J* = 53.6 Hz, 1H), 7.49 (d, *J* = 8.7 Hz, 2H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -95.78 (d, *J* = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 190.0 (t, *J* = 2.9 Hz), 141.6, 136.2 (t, *J*= 2.6 Hz), 129.7 (2C), 129.0 (2C), 120.1 (t, *J* = 284.3 Hz). IR (KBr): v = 2924, 1689, 1587, 1573, 1486, 1399, 1269, 1202, 1070, 875, 834, 688 cm⁻¹. HRMS (ESI) for C₈H₅ClF₂OSeNa (M+Na⁺): Calcd: 292.9054, Found: 292.9066.



Se-(difluoromethyl)-4-bromobenzoselenoate (3g). Yellow solid (85 mg, 54%). Mp: 57-58 °C. Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.69 – 7.64 (m, 4H), 7.60 (d, J = 53.5 Hz, 1H); ¹⁹F NMR (376 MHz, CDCl₃) δ -95.78 (d, J = 53.4 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 190.3 (t, J = 2.9 Hz), 136.7(t, J = 2.4 Hz), 132.7 (2C), 130.3, 129.0 (2C), 120.1 (t, J = 284.4 Hz). IR (KBr): v = 2923, 1682, 1582, 1482, 1395, 1268, 1199, 1174, 1065, 1009, 874, 816, 705, 686, 615 cm⁻¹. HRMS (ESI) for C₈H₆BrF₂OSe (M+H⁺): Calcd: 314.8730, Found: 314.8729.



Se-(difluoromethyl)-4-iodobenzoselenoate (3h). Yellow solid (85 mg, 47%). Mp: 50-51 °C. Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (600 MHz, CDCl₃) δ 7.88 (d, *J* = 8.6 Hz, 2H), 7.60 (t, *J* = 53.5 Hz, 1H), 7.51 (d, *J* = 8.6 Hz, 2H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -95.79 (d, *J* = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 190.6 (t, *J* = 2.9 Hz), 138.7 (2C), 137.2 (t, *J* = 2.3 Hz), 128.8 (2C), 120.0 (t, *J* = 284.4 Hz), 103.2. IR (KBr): v = 2921, 2851, 1672, 1578, 1561, 1477, 1389, 1263, 1204, 1175, 1114, 1044, 1005, 872, 826, 686, 617 cm⁻¹. HRMS (ESI) for C₈H₃IF₂OSeNa (M+Na⁺): Calcd: 384.8411, Found: 384.8414.



Se-(difluoromethyl)-[1,1'-biphenyl]-4-carboselenoate (3i). Yellow solid (131 mg, 84%). Mp: 77-78 °C. Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, *J* = 8.5 Hz, 2H), 7.68 (d, *J* = 8.5 Hz, 2H), 7.63 (t, *J* = 53.6 Hz, 1H), 7.62 – 7.57 (m, 2H), 7.49 – 7.44 (m, 2H), 7.44 – 7.38 (m, 1H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -95.89 (d, *J* = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 190.5 (t, *J* = 2.8 Hz), 147.8, 139.3, 136.4 (t, *J* = 2.4 Hz), 129.2 (2C), 128.9, 128.3 (2C), 127.9 (2C), 127.4 (2C), 120.3 (t, *J* = 283.7 Hz). IR (KBr): v = 2922, 1680, 1600, 1200, 1182, 1085, 1047, 891, 768, 688 cm⁻¹. HRMS (ESI) for C₁₄H₁₀F₂OSeNa (M+Na⁺): Calcd: 334.9757, Found: 334.9753.



Se-(difluoromethyl)-4-methoxybenzoselenoate (3j). Yellow solid (100 mg, 75%). Mp: 38-39 °C. Eluant: ethyl acetate/petroleum ether (1:30, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.77 (d, J = 8.9 Hz, 2H), 7.60 (t, J = 53.6 Hz, 1H), 6.95 (d, J = 8.9 Hz, 2H), 3.88 (s, 3H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -95.95 (d, J = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 188.8 (t, J = 2.8 Hz), 165.1, 130.6 (t, J = 2.5 Hz), 130.2 (2C), 120.5 (t, J = 283.0 Hz), 114.5 (2C), 55.8. IR (KBr): v = 2936, 1686, 1596, 1575, 1507, 1265, 1211, 1165, 1059, 1026, 878, 835, 784, 691, 648, 611 cm⁻¹. HRMS (ESI) for C₉H₈F₂O₂SeNa (M+Na⁺): Calcd: 288.9550, Found: 288.9548.



Se-(difluoromethyl)-4-(methylthio)benzoselenoate (3k). Yellow solid (115 mg, 81%). Mp: 65-66 °C. Eluant: ethyl acetate/petroleum ether (1:50, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.69 (d, J = 8.7 Hz, 2H), 7.61 (t, J = 53.6 Hz, 1H), 7.27 (d, J = 8.7 Hz, 2H), 2.52 (s, 3H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -95.90 (d, J = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 189.6 (t,

J= 2.6 Hz), 149.0, 133.8 (t, J = 2.4 Hz), 128.0 (2C), 125.3 (2C), 120.4 (t, J = 283.5 Hz), 14.8. IR (KBr): v = 2923, 1679, 1586, 1488, 1434, 1401, 1322, 1268, 1214, 1185, 1072, 1036, 883, 821, 733, 712, 689, 619 cm⁻¹. HRMS (ESI) for C₉H₈F₂OSSeNa (M+Na⁺): Calcd: 304.9321, Found: 304.9316.



Se-(difluoromethyl)-4-((tert-butoxycarbonyl)oxy)benzoselenoate (3l). Yellow solid (127 mg, 72%). Mp: 33-34 °C. Eluant: ethyl acetate/petroleum ether (1:30, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.84 (d, J = 8.8 Hz, 2H), 7.61 (t, J = 53.5 Hz, 1H), 7.33 (d, J = 8.8 Hz, 2H), 1.57 (s, 9H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -95.88 (d, J = 53.4 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 189.8 (t, J = 2.8 Hz), 156.1, 150.8, 135.1 (t, J = 2.4 Hz), 129.3 (2C), 122.1 (2C), 120.2 (t, J = 284.0 Hz), 84.8, 27.8 (3C). IR (KBr): v = 2983, 1759, 1686, 1599, 1504, 1371, 1272, 1222, 1202, 1140, 1069, 875, 843, 779, 690, 646, 621 cm⁻¹. HRMS (ESI) for C₁₃H₁₄F₂O₄SeNa (M+Na⁺): Calcd: 374.9918, Found: 374.9918.



Methyl-4-(((difluoromethyl)selanyl)carbonyl)benzoate (**3m**). Yellow gum (103 mg, 70%).Eluant: ethyl acetate/petroleum ether (1:50, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 8.16 (d, J = 8.1 Hz, 2H), 7.87 (d, J = 8.1 Hz, 2H), 7.62 (t, J = 53.4 Hz, 1H), 3.96 (s, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -95.85 (d, J = 53.4 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 191.1 (t, J = 2.9 Hz), 165.8, 140.9 (t, J = 2.4 Hz), 135.6, 130.5 (2C), 127.6 (2C), 120.0 (t, J = 284.6 Hz), 52.8. IR (KBr): v = 2925, 2854, 1728, 1688, 1280, 1198, 1109, 1072, 885, 821, 773, 731, 690 cm⁻¹. HRMS (ESI) for C₁₀H₉F₂O₃Se (M+H⁺): Calcd: 294.9679, Found: 294.9685.



Se-(difluoromethyl)-4-(dimethylamino)benzoselenoate (3n). Yellow solid (97 mg, 69%). Mp: 101-102 °C. Eluant: ethyl acetate/petroleum ether (1:50, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.65 (d, J = 8.8 Hz, 2H), 7.60 (t, J = 53.9 Hz, 1H), 6.61 (d, J = 8.8 Hz, 2H), 3.06 (s, 6H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -95.93 (d, J = 53.9 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 186.9 (t, J = 2.6 Hz), 154.7, 130.3 (2C), 124.7 (t, J = 2.4 Hz), 121.0 (t, J = 281.7 Hz), 111.0 (2C), 40.1 (2C). IR (KBr): v = 2925, 1675, 1593, 1553, 1531, 1376, 1167, 1066, 875, 821 cm⁻¹. HRMS (ESI) for $C_{10}H_{11}F_2NOSeNa$ (M+Na⁺): Calcd: 301.9866, Found: 301.9868.



Se-(difluoromethyl)-benzoselenoate (3o). Yellow gum (102 mg, 86%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.82 (d, J = 7.5 Hz, 2H), 7.67 (t, J = 7.4 Hz, 1H), 7.62 (d, J = 53.5 Hz, 1H), 7.53 – 7.48 (m, 2H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -96.06 (d, J = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 191.2 (t, J = 2.8 Hz), 137.8 (t, J = 2.3 Hz), 135.0, 129.4 (2C), 127.7 (2C), 120.3 (t, J = 283.6 Hz). IR (KBr): v = 2923, 1689, 1449, 1270, 1203, 1073, 877, 767, 670, 624 cm⁻¹. HRMS (ESI) for C₈H₆F₂OSeNa (M+Na⁺): Calcd: 258.9444, Found: 258.9444.



Se-(difluoromethyl)-3-methylbenzoselenoate (3p). Yellow gum (80 mg, 64%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.61 (t, *J* = 53.6 Hz, 1H),

7.61 – 7.60 (m, 2H), 7.48 – 7.45 (m, 1H), 7.38 (t, J = 7.9 Hz, 1H), 2.43 (s, 3H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -96.14 (d, J = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 191.2 (t, J = 2.7 Hz), 139.5, 137.9 (t, J = 2.2 Hz), 135.8, 129.2, 128.1, 125.1, 120.4 (t, J = 283.5 Hz), 21.4. IR (KBr): v = 2924, 1687, 1600, 1457, 1270, 1242, 1148, 1058, 947, 930, 808, 790, 769, 686, 665 cm⁻¹. HRMS (ESI) for C₉H₈F₂OSeNa (M+Na⁺): Calcd: 272.9601, Found: 272.9590.



Methyl-3-(((difluoromethyl)selanyl)carbonyl)benzoate (3q). Yellow gum (109 mg, 74%). Eluant: ethyl acetate/petroleum ether (1:50, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 8.44 (t, J = 1.5 Hz, 1H), 8.33 – 8.30 (m, 1H), 8.00 – 7.97 (m, 1H), 7.63 (t, J = 53.4 Hz, 1H), 7.61 (t, J = 7.8 Hz, 1H), 3.97 (s, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -95.83 (d, J = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 190.7 (t, J = 3.0 Hz), 165.7, 138.2 (t, J = 2.5 Hz), 135.6, 131.6, 131.5, 129.6, 128.8, 120.1 (t, J = 284.5 Hz), 52.8. IR (KBr): $\nu = 2924$, 1725, 1692, 1600, 1440, 1280, 1181, 1059, 983, 914, 823, 757, 722, 682, 650, 632 cm⁻¹. HRMS (ESI) for C₁₀H₈F₂O₃SeNa (M+Na⁺): Calcd: 316.9499, Found: 316.9499.



Se-(difluoromethyl)-2-methylbenzoselenoate (3r). Yellow oil (108 mg, 86%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.71 – 7.69 (m, 1H), 7.55 (t, *J* = 53.5 Hz, 1H), 7.48 (t, *J* = 7.5 Hz, 1H), 7.35 – 7.28 (m, 2H), 2.52 (s, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.71 (d, *J* = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 192.3 (t, *J* = 2.8 Hz), 137.6, 137.4 (t, *J* = 2.3 Hz), 133.4, 132.3, 129.5, 126.6, 120.8 (t, *J* = 283.3 Hz), 21.1. IR

(KBr): v = 2924, 1699, 1601, 1569, 1457, 1382, 1269, 1205, 1189, 1057, 873, 761, 715, 670, 664,
630 cm⁻¹. HRMS (ESI) for C₉H₈F₂OSeNa (M+Na⁺): Calcd: 272.9601, Found: 272.9605.



Se-(difluoromethyl)-2-chlorobenzoselenoate (3s). Yellow oil (98 mg, 72%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.69 – 7.67 (m, 1H), 7.58 (t, *J* = 53.3 Hz, 1H), 7.51 – 7.49 (m, 2H), 7.42 – 7.38 (m, 1H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -96.77 (d, *J* = 53.3 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 190.2 (t, *J* = 3.1 Hz), 137.2 (t, *J* = 2.3 Hz), 133.9, 131.7, 131.1, 129.7, 127.4, 120.3 (t, *J* = 284.7 Hz). IR (KBr): v = 2925, 1701, 1586, 1467, 1434, 1263, 1191, 1055, 878, 760, 727, 687, 651, 625 cm⁻¹. HRMS (ESI) for C₈H₅ClF₂OSeNa (M+Na⁺): Calcd: 292.9054, Found: 292.9061.



Se-(difluoromethyl)-2-bromobenzoselenoate (3t). Yellow oil (110 mg, 70%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.69 (dd, J = 7.7, 1.3 Hz, 1H), 7.63 (dd, J = 7.4, 1.9 Hz, 1H), 7.58 (t, J = 54.9 Hz, 1H), 7.47 – 7.38 (m, 2H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.45 (d, J = 53.3 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 191.3 (t, J = 3.1 Hz), 139.4 (t, J = 2.4 Hz), 135.0, 133.8, 129.7, 127.9, 120.3 (t, J = 285.0 Hz), 118.6. IR (KBr): v = 2924, 1698, 1584, 1461, 1430, 1267, 1195, 1049, 875, 759, 721, 680, 642, 624 cm⁻¹. HRMS (ESI) for C₈H₅BrF₂OSeNa (M+Na⁺): Calcd: 336.8549, Found: 336.8553.





Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.56 (t, J = 53.4 Hz, 1H), 7.55 (d, J = 8.0 Hz, 1H), 7.52 (s, 1H), 7.23 (d, J = 8.0 Hz, 1H), 2.39 (s, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.59 (d, J = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 190.5 (t, J = 2.9 Hz), 145.4, 136.3 (t, J = 2.5 Hz), 135.7, 130.1, 128.6, 120.5 (t, J = 284.5 Hz), 118.9, 21.4. IR (KBr): v = 2922, 1718, 1691, 1595, 1475, 1379, 1267, 1190, 1045, 894, 802, 686, 610 cm⁻¹. HRMS (ESI) for C₉H₇BrF₂OSeNa (M+Na⁺): Calcd: 350.8706, Found: 350.8711.



Se-(difluoromethyl)-3-bromo-4-methoxybenzoselenoate (3v). Yellow solid (131 mg, 76%). Mp: 78-79 °C. Eluant: ethyl acetate/petroleum ether (1:30, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 8.00 (s, 1H), 7.75 (d, J = 8.7 Hz, 1H), 7.59 (t, J = 53.5 Hz, 1H), 6.95 (d, J = 8.7 Hz, 1H), 3.98 (s, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -95.70 (d, J = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 188.1 (t, J = 2.8 Hz), 161.1, 132.9, 131.5 (t, J = 2.4 Hz), 129.2, 120.3 (t, J = 283.9 Hz), 112.8, 111.6, 56.9. IR (KBr): v = 2921, 1714, 1681, 1591, 1557, 1493, 1399, 1281, 1259, 1188, 1068, 1015, 926, 909, 787, 688, 670, 644, 622 cm⁻¹. HRMS (ESI) for C₉H₇BrF₂O₂SeNa (M+Na⁺): Calcd: 366.8655, Found: 366.8657.



Se-(difluoromethyl)-3,4-dimethoxybenzoselenoate (3w). Yellow solid (123 mg, 83%). Mp: 51-52 °C. Eluant: ethyl acetate/petroleum ether (1:30, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.61 (t, *J* = 53.6 Hz, 1H), 7.47 – 7.45 (m, 1H), 7.31 (d, *J* = 1.9 Hz, 1H), 6.92 (d, *J* = 8.5 Hz, 1H), 3.95 (s, 3H), 3.92 (s, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -95.93 (d, *J* = 53.6 Hz, 2F); ¹³C NMR

(101 MHz, CDCl₃) δ 189.0 (t, J = 2.7 Hz), 154.9, 149.5, 130.7 (t, J = 2.5 Hz), 123.2, 120.5 (t, J = 283.2 Hz), 110.7, 109.2, 56.4, 56.2. IR (KBr): v = 2936, 1683, 1586, 1511, 1463, 1415, 1264, 1245, 1142, 1064, 1020, 975, 950, 790, 757, 691, 648 cm⁻¹. HRMS (ESI) for C₁₀H₁₁F₂O₃Se (M+H⁺): Calcd: 296.9836, Found: 296.9834.



Se-(difluoromethyl)-3,5-dimethoxybenzoselenoate (3x). Yellow solid (120 mg, 81%). Mp: 41-42 °C. Eluant: ethyl acetate/petroleum ether (1:30, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.59 (t, J = 53.5 Hz, 1H), 6.93 – 6.92 (m, 2H), 6.71 (s, 1H), 3.83 (s, 6H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.18 (d, J = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 191.1 (t, J = 3.0 Hz), 161.4 (2C), 139.8 (t, J = 2.3 Hz), 120.3 (t, J = 283.8 Hz), 107.0, 105.4 (2C), 55.8 (2C). IR (KBr): $\nu = 2941$, 1688, 1590, 1457, 1426, 1354, 1299, 1269, 1206, 1159, 1058, 980, 926, 846, 752, 688 cm⁻¹. HRMS (ESI) for C₁₀H₁₀F₂O₃SeNa (M+Na⁺): Calcd: 318.9655, Found: 318.9652.



Se-(difluoromethyl)-naphthalene-1-carboselenoate (3aa). Yellow gum (125 mg, 87%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 8.61 (d, J = 8.6 Hz, 1H), 8.08 (d, J = 8.2 Hz, 1H), 8.00 (d, J = 7.2 Hz, 1H), 7.90 (d, J = 8.1 Hz, 1H), 7.66 (t, J = 53.6Hz, 1H), 7.65 (t, J = 7.1 Hz, 1H), 7.58 (t, J = 7.5 Hz, 1H), 7.53 (t, J = 7.8 Hz, 1H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.69 (d, J = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 192.5 (t, J = 2.9 Hz), 135.2 (t, J = 2.4 Hz), 135.0, 134.0, 129.6, 129.2, 128.6, 128.5, 127.4, 125.0, 124.7, 120.9 (t, J =283.6 Hz). IR (KBr): v = 2923, 1695, 1507, 1267, 1221, 1171, 1047, 888, 803, 771, 745, 689, 657 cm⁻¹. HRMS (ESI) for C₁₂H₈F₂OSeNa (M+Na⁺): Calcd: 308.9601, Found: 308.9605.



Se-(difluoromethyl)-naphthalene-2-carboselenoate (3ab). Yellow solid (129 mg, 90%). Mp: 41-42 °C. Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 8.36 (s, 1H), 8.00 (d, J = 8.1 Hz, 1H), 7.92 (t, J = 9.4 Hz, 2H), 7.84 – 7.82 (m, 1H), 7.68 (t, J = 54.5 Hz, 1H), 7.69 – 7.59 (m, 2H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -95.91 (d, J = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 191.0 (t, J = 2.8 Hz), 136.5, 135.1 (t, J = 2.4 Hz), 132.5, 130.1, 129.9, 129.6, 129.4, 128.1, 127.6, 122.7, 120.4 (t, J = 283.7 Hz). IR (KBr): v = 2923, 1686, 1260, 1159, 1046, 970, 913, 814, 775, 682 cm⁻¹. HRMS (ESI) for C₁₂H₈F₂OSeNa (M+Na⁺): Calcd: 308.9601, Found: 308.9604.



Se-(difluoromethyl)-1-bromonaphthalene-2-carboselenoate (3ac). Yellow solid (146 mg, 80%). Mp: 66-67 °C. Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 8.45 (d, J = 8.3 Hz, 1H), 7.91 (d, J = 8.5 Hz, 1H), 7.88 (d, J = 7.8 Hz, 1H), 7.66 (t, J = 53.3 Hz, 1H), 7.72 – 7.64 (m, 2H), 7.56 (d, J = 8.5 Hz, 1H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -96.32 (d, J = 53.4 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 192.7 (t, J = 2.9 Hz), 137.8 (t, J = 2.5 Hz), 135.6, 132.3, 129.1, 129.0 (2C), 128.8, 128.5, 123.6, 120.3 (t, J = 285.3 Hz), 119.8. IR (KBr): v = 2922, 1717, 1456, 1258, 1193, 1071, 996, 928, 766, 689 cm⁻¹. HRMS (ESI) for C₁₂H₇BrF₂OSeNa (M+Na⁺): Calcd: 386.8706, Found: 386.8711.



Se-(difluoromethyl)-1-tosyl-1H-indole-3-carboselenoate (3ad). Yellow solid (176 mg, 82%). Mp: 126-127 °C. Eluant: ethyl acetate/petroleum ether (1:60, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 8.26 (s, 1H), 8.08 (d, *J* = 7.5 Hz, 1H), 7.94 (d, *J* = 8.3 Hz, 1H), 7.85 (d, *J* = 8.3 Hz, 2H), 7.67 (t, *J* = 53.5 Hz, 1H), 7.38 (dt, *J* = 20.4, 7.4 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 2.37 (s, 3H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -95.26 (d, *J* = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 182.0 (t, *J* = 2.9 Hz), 146.6, 134.9, 134.2, 132.9, 130.5 (2C), 127.5 (2C), 126.6, 126.0, 125.5, 122.4 (t, *J* = 2.8 Hz), 122.2, 120.1 (t, *J* = 284.2 Hz), 113.5, 21.8. IR (KBr): v = 2925, 1683, 1531, 1446, 1380, 1293, 1176, 1066, 964, 806, 749, 717, 689, 661 cm⁻¹. HRMS (ESI) for C₁₇H₁₃F₂NO₃SSeNa (M+Na⁺): Calcd: 451.9642, Found: 451.9637.



Se-(difluoromethyl)-benzofuran-3-carboselenoate (3ae). Yellow solid(105 mg, 76%).Mp: 51-52 °C. Eluant: ethyl acetate/petroleum ether (1:60, $R_f = 0.30$).¹H NMR (400 MHz, CDCl₃) δ 8.29 (s, 1H), 8.04 – 8.02 (m, 1H), 7.71 (t, *J* = 53.5 Hz, 1H), 7.56 – 7.54 (m, 1H), 7.44 – 7.38 (m, 2H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -95.28 (d, *J* = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 181.4 (t, *J* = 2.8 Hz), 155.9, 151.9, 126.7, 125.3, 124.2 (t, *J* = 2.8 Hz), 122.7, 122.1, 119.7 (t, *J* = 284.5 Hz), 112.1. IR (KBr): ν = 2925, 1687, 1543, 1478, 1450, 1352, 1265, 1151, 1124, 1055, 853, 806, 746, 688, 643 cm⁻¹. HRMS (ESI) for C₁₀H₆F₂O₂SeNa (M+Na⁺): Calcd: 298.9393, Found: 298.9392.



Se-(difluoromethyl)-benzo[b]thiophene-3-carboselenoate (3af). Yellow solid (110 mg, 75%). Mp: 40-41 °C. Eluant: ethyl acetate/petroleum ether (1:80, R_f = 0.30). ¹H NMR (400 MHz, CDCl₃) δ 8.45 (d, *J* = 8.2 Hz, 1H), 8.29 (s, 1H), 7.84 (d, *J* = 7.8 Hz, 1H), 7.66 (t, *J* = 53.5 Hz, 1H), 7.52 – 7.42 (m, 2H); ¹⁹F NMR (376 MHz, CDCl₃) δ -95.59 (d, *J* = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 182.9 (t, *J* = 3.0 Hz), 140.1, 139.0, 135.8 (t, *J* = 2.7 Hz), 134.7, 126.6, 126.4, 124.6, 122.7, 120.4 (t, *J* = 283.9 Hz). IR (KBr): v = 2922, 1683, 1490, 1458, 1424, 1377, 1268, 1062, 1044, 873, 789, 756, 730, 685 cm⁻¹. HRMS (ESI) for C₁₀H₆F₂OSSeNa (M+Na⁺): Calcd: 314.9165, Found: 314.9168.



Se-(difluoromethyl)-1-tosyl-1H-pyrrole-2-carboselenoate (3ag). Yellow solid (150 mg, 79%). Mp: 107-108 °C. Eluant: ethyl acetate/petroleum ether (1:60, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.89 (d, *J* = 8.4 Hz, 2H), 7.86 (dd, *J* = 3.0, 1.7 Hz, 1H), 7.47 (t, *J* = 53.4 Hz, 1H), 7.34 (d, *J* = 7.6 Hz, 2H), 7.16 (dd, *J* = 3.9, 1.6 Hz, 1H), 6.38 (t, *J* = 3.5 Hz, 1H), 2.44 (s, 3H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -95.86 (d, *J* = 53.4 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 176.3 (t, *J* = 3.4 Hz), 145.9, 134.9, 131.5, 131.0 (t, *J* = 3.8 Hz), 129.8 (2C), 128.6 (2C), 126.1, 120.3 (t, *J* = 283.6 Hz), 111.3, 21.9. IR (KBr): v = 2922, 1702, 1422, 1402, 1375, 1247, 1193, 1174, 1141, 1062, 1016, 813, 751, 691, 667 cm⁻¹. HRMS (ESI) for C₁₃H₁₁F₂NO₃SSeNa (M+Na⁺): Calcd: 401.9485, Found: 401.9484.



Se-(difluoromethyl)-furan-2-carboselenoate (3ah). Yellow gum (81 mg, 71%). Eluant: ethyl acetate/petroleum ether (1:60, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.66 (s, 1H), 7.62 (t, J = 53.4 Hz, 1H), 7.25 (d, J = 3.3 Hz, 1H), 6.64 – 6.62 (m, 1H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -96.48 (d, J = 53.4 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 178.2 (t, J = 3.3 Hz), 151.0, 147.8, 119.5 (t, J = 283.3 Hz), 116.7, 113.5. IR (KBr): v = 2920, 2851, 1688, 1563, 1461, 1253, 1073, 1014, 814, 763, 691 cm⁻¹. HRMS (ESI) for C₆H₄F₂O₂SeNa (M+Na⁺): Calcd: 248.9237, Found: 248.9238.



Se-(difluoromethyl)-thiophene-2-carboselenoate (3ai). Yellow gum (84 mg, 69%). Eluant: ethyl acetate/petroleum ether (1:80, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.79 (d, J = 4.4 Hz, 1H), 7.75 (d, J = 3.8 Hz, 1H), 7.63 (t, J = 53.4 Hz, 1H), 7.18 (t, J = 4.4 Hz, 1H); ¹⁹F NMR (376 MHz, CDCl₃) δ -95.20 (d, J = 53.4 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 180.8 (t, J = 3.1 Hz), 142.3 (t, J = 3.3 Hz), 135.4, 133.3, 128.4, 120.1 (t, J = 284.4 Hz). IR (KBr): v = 2924, 1669, 1511, 1407, 1351, 1270, 1233, 1198, 1045, 867, 844, 772, 723, 691, 670, 633 cm⁻¹. HRMS (ESI) for C₆H₄F₂OSSeNa (M+Na⁺): Calcd: 264.9008, Found: 264.9007.



Se-(difluoromethyl)-dodecaneselenoate (3ba). Yellow oil (115 mg, 73%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.44 (t, J = 53.5 Hz, 1H), 2.68 (t, J = 7.5 Hz, 2H), 1.71 – 1.64 (m, 2H), 1.36 – 1.26 (m, 16H), 0.88 (t, J = 6.9 Hz, 3H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -96.27 (d, J = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 198.2 (t, J = 1.8 Hz), 120.1 (t, J = 283.4 Hz), 49.2 (t, J = 2.5 Hz), 32.0, 29.70, 29.67, 29.5 (2C), 29.3, 28.8, 25.0, 22.8, 14.2. IR (KBr): v = 2923, 2854, 1729, 1465, 1271, 1062, 722, 688 cm⁻¹. HRMS (ESI)

for C₁₃H₂₄F₂OSeNa (M+Na⁺): Calcd: 337.0853, Found: 337.0856.



Se-(difluoromethyl)-tetradecaneselenoate (3bb). Yellow oil (130 mg, 76%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.44 (t, J = 53.5 Hz, 1H), 2.68 (t, J = 7.5 Hz, 2H), 1.71 – 1.64 (m, 2H), 1.36 – 1.26 (m, 20H), 0.88 (t, J = 6.8 Hz, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.27 (d, J = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 198.2 (t, J = 2.1 Hz), 120.1 (t, J = 283.4 Hz), 49.2 (t, J = 2.5 Hz), 32.1, 29.80, 29.78, 29.75, 29.7, 29.49, 29.46, 29.3, 28.8, 25.0, 22.8, 14.3. IR (KBr): v = 2922, 2853, 1729, 1465, 1271, 1063, 722, 688 cm⁻¹. HRMS (ESI) for C₁₅H₂₈F₂OSeNa (M+Na⁺): Calcd: 365.1166, Found: 365.1161.



Se-(difluoromethyl)-octadecaneselenoate (3bc). Yellow gum (162 mg, 81%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.44 (t, J = 53.5 Hz, 1H), 2.68 (t, J = 7.5 Hz, 2H), 1.71 – 1.64 (m, 2H), 1.36 – 1.26 (m, 28H), 0.88 (t, J = 6.8 Hz, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.26 (d, J = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 198.2 (t, J = 1.5 Hz), 120.1 (t, J = 283.4 Hz), 49.2 (t, J = 2.5 Hz), 32.1, 29.84 (3C), 29.82, 29.81, 29.80, 29.75, 29.7, 29.51, 29.47, 29.3, 28.8, 25.0, 22.8, 14.3.IR (KBr): v = 2921, 2852, 1730, 1465, 1271, 1064, 721, 688 cm⁻¹. HRMS (ESI) for C₁₉H₃₆F₂OSeNa (M+Na⁺): Calcd: 421.1792, Found: 421.1781.



Se-(difluoromethyl)-3-(benzo[d][1,3]dioxol-5-yl)-2-methylpropaneselenoate (3bd). Yellow gum (118 mg, 73%). Eluant: ethyl acetate/petroleum ether (1:30, $R_f = 0.30$). ¹H NMR (400 MHz,

CDCl₃) δ 7.41 (t, J = 53.6 Hz, 1H), 6.74 (d, J = 7.8 Hz, 1H), 6.63 (s, 1H), 6.60 (d, J = 7.9 Hz, 1H), 5.94 (s, 2H), 3.01 (dd, J = 13.7, 6.4 Hz, 1H), 2.94 – 2.85 (m, 1H), 2.61 (dd, J = 13.6, 7.8 Hz, 1H), 1.19 (d, J = 6.9 Hz, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.19 (dd, J = 53.5, 30.7 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 202.2 (t, J = 1.6 Hz), 148.0, 146.6, 131.6, 122.3, 120.0 (t, J = 283.7Hz), 109.4, 108.5, 101.1, 55.2 (t, J = 2.3 Hz), 39.0, 16.4.IR (KBr): v = 2925, 1719, 1505, 1490, 1444, 1248, 1191, 1070, 1040, 937, 874, 809, 690 cm⁻¹. HRMS (ESI) for C₁₂H₁₂F₂O₃SeNa (M+Na⁺): Calcd: 344.9812, Found: 344.9810.



Se-(difluoromethyl)-3,5,5-trimethylhexaneselenoate (3be). Yellow oil (106 mg, 78%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.44 (t, J = 53.5 Hz, 1H), 2.69 (dd, J = 15.1, 5.7 Hz, 1H), 2.52 (dd, J = 15.1, 8.2 Hz, 1H), 2.17 – 2.06 (m, 1H), 1.26 (dd, J = 14.1, 4.2 Hz, 1H), 1.15 (dd, J = 14.1, 6.3 Hz, 1H), 1.02 (d, J = 6.7 Hz, 3H), 0.91 (s, 9H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -96.28 (d, J = 53.5 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 197.6 (t, J = 1.8 Hz), 120.2 (t, J = 283.4 Hz), 58.4 (t, J = 2.4 Hz), 50.4, 31.2, 30.0 (3C), 27.6, 22.6.IR (KBr): $\nu = 2956, 1726, 1468, 1366, 1271, 1061, 988, 959, 687$ cm⁻¹. HRMS (ESI) for C₁₀H₁₈F₂OSeNa (M+Na⁺): Calcd: 295.0383, Found: 295.0385.



Se-(difluoromethyl)-(1R,5S)-6,6-dimethylbicyclo[3.1.1]hept-2-ene-2-carboselenoate(3bf). Yellow oil (101 mg, 72%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$).¹H NMR (400 MHz, CDCl₃) δ 7.47 (t, *J* = 53.7 Hz, 1H), 6.80 – 6.78 (m, 1H), 2.79 (td, *J* = 5.6, 1.3 Hz, 1H), 2.55 – 2.41 (m, 3H), 2.20 – 2.15 (m, 1H), 1.34 (s, 3H), 1.11 (d, *J* = 9.4 Hz, 1H), 0.78 (s, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.19 (d, J = 53.7 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 188.7 (t, J = 2.6 Hz), 149.9 (t, J = 2.2 Hz), 140.5, 120.5 (t, J = 282.7 Hz), 41.8, 40.5, 38.1, 32.8, 31.3, 25.8, 21.0. IR (KBr): v = 2928, 1682, 1615, 1470, 1418, 1369, 1268, 1205, 1168, 1127, 1041, 916, 806, 723, 689, 621 cm⁻¹. HRMS (ESI) for C₁₁H₁₄F₂OSeNa (M+Na⁺): Calcd: 303.0070, Found: 303.0074.



Se-(difluoromethyl)-(E)-dec-2-eneselenoate (3bg). Yellow oil (98 mg, 69%). Eluant: ethyl acetate/petroleum ether (1:100, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) 7.49 (t, J = 53.6 Hz, 1H), 6.93 – 6.86 (m, 1H), 6.08 (d, J = 15.4 Hz, 1H), 2.23 (q, J = 7.2 Hz, 2H), 1.50 – 1.46 (m, 2H), 1.30 – 1.26 (m, 8H), 0.88 (t, J = 6.3 Hz, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -96.07 (d, J = 53.6 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 188.5 (t, J = 2.5 Hz), 150.7, 130.6 (t, J = 2.7 Hz), 120.2 (t, J = 283.1 Hz), 32.6, 31.8, 29.2, 29.1, 27.8, 22.7, 14.2. IR (KBr): v = 2926, 2856, 1694, 1625, 1465, 1269, 1061, 778, 690 cm⁻¹. HRMS (ESI) for C₁₁H₁₈F₂OSeNa (M+Na⁺): Calcd: 307.0383, Found: 307.0381.



(**1R**,**2S**,**5R**)-**2**-**isopropyl-5**-**methylcyclohexyl-3**-(((**difluoromethyl**)**selanyl**)**carbonyl**)**benzoate** (**3ca**). Yellow oil (94 mg, 45%). Eluant: ethyl acetate/petroleum ether (1:60, R_f = 0.30). ¹H NMR (400 MHz, CDCl₃) δ 8.44 (s, 1H), 8.32 (d, *J* = 7.7 Hz, 1H), 7.98 (d, *J* = 7.7 Hz, 1H), 7.63 (t, *J* = 53.5 Hz, 1H), 7.60 (t, *J* = 7.8 Hz, 1H), 4.97 (td, *J* = 10.7, 4.0 Hz, 1H), 2.12 (d, *J* = 11.8 Hz, 1H), 1.95 – 1.89 (m, 1H), 1.75 (d, *J* = 11.9 Hz, 2H), 1.57 (s, 2H), 1.18 – 1.09 (m, 2H), 0.95 – 0.92 (m, 7H), 0.80 (d, *J* = 6.9 Hz, 3H); ¹⁹F NMR (376 MHz, CDCl₃) δ -95.78 (d, *J* = 53.7 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ190.8 (t, *J* = 2.9 Hz), 164.7, 138.2 (t, *J* = 2.5 Hz), 135.6, 132.3, 131.3, 129.6, 128.8, 120.1 (t, J = 284.4 Hz), 76.0, 47.3, 41.0, 34.4, 31.6, 26.8, 23.8, 22.2, 20.9, 16.7. IR (KBr): v = 2922, 1721, 1702, 1646, 1600, 1458, 1377, 1298, 1278, 1188, 1074, 951, 932, 884, 845, 757, 687 cm⁻¹. HRMS (ESI) for C₁₉H₂₄F₂O₃SeNa (M+Na⁺): Calcd: 441.0751, Found: 441.0751.



4-(((difluoromethyl)selanyl)carbonyl)phenyl-2-(4-isobutylphenyl)propanoate (3cb). Yellow oil (130 mg, 59%). Eluant: ethyl acetate/petroleum ether (1:60, $R_f = 0.30$). ¹H NMR (400 MHz, CDCl₃) δ 7.82 – 7.78 (m, 2H), 7.60 (t, J = 53.5 Hz, 1H), 7.29 (d, J = 8.1 Hz, 2H), 7.17 (s, 2H), 7.15 (d, J = 1.7 Hz, 2H), 3.96 (q, J = 7.1 Hz, 1H), 2.48 (d, J = 7.2 Hz, 2H), 1.92 – 1.82 (m, 1H), 1.62 (d, J = 7.1 Hz, 3H), 0.91 (d, J = 6.6 Hz, 6H); ¹⁹F NMR (376 MHz, CDCl₃) δ -95.80 (d, J = 53.8 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 189.8 (t, J = 2.9 Hz), 172.5, 156.0, 141.3, 136.8, 135.2 (t, J = 2.5 Hz), 129.8 (2C), 129.2 (2C), 127.3 (2C), 122.5 (2C), 120.2 (t, J = 284.0 Hz), 45.4, 45.2, 30.3, 22.5 (2C), 18.5. IR (KBr): v = 2955, 1762, 1696, 1598, 1500, 1463, 1205, 1163, 1132, 1068, 883, 847, 690, 620 cm⁻¹. HRMS (ESI) for C₂₁H₂₂F₂O₃SeNa (M+Na⁺): Calcd: 463.0594, Found: 463.0615.



Se-(difluoromethyl)-4-(((3S,8R,9S,10S,13R,14S,17R)-10,13-dimethyl-17-((R)-6-meth-ylhepta n-2-yl)hexadecahydro-1H-cyclopenta[a]phenanthren-3-yl)oxy)benzoseleno-ate (3cc). Yellow oil (128 mg, 41%). Eluant: ethyl acetate/petroleum ether (1:80, R_f = 0.30). ¹H NMR (400 MHz,

CDCl₃) δ 7.75 (d, J = 8.8 Hz, 2H), 7.60 (d, J = 53.6 Hz, 1H), 6.94 (d, J = 8.7 Hz, 2H), 4.65 (s, 1H), 1.99 – 1.48 (m, 12H), 1.35 – 0.83 (m, 31H),0.65 (s, 3H); ¹⁹ F NMR (376 MHz, CDCl₃) δ -95.87 (d, J = 53.9 Hz, 2F); ¹³C NMR (101 MHz, CDCl₃) δ 188.7 (t, J = 2.7 Hz), 163.7, 130.3 (2C), 129.9 (t, J = 2.8 Hz), 120.6 (t, J = 282.3 Hz), 115.9 (2C), 73.1, 56.7, 56.4, 54.3, 42.7, 40.1, 39.74, 39.66, 36.3, 36.0, 35.9, 35.6, 32.71, 32.66, 32.1, 28.6, 28.4, 28.2, 25.8, 24.3, 24.0, 23.0, 22.7, 21.0, 18.8, 12.2, 11.6. IR (KBr): v = 2924, 1683, 1597, 1572, 1505, 1458, 1377, 1306, 1263, 1212, 1161, 1069, 879, 837, 690, 622 cm⁻¹. HRMS (ESI) for C₃₅H₅₂F₂O₂SeNa (M+Na⁺): Calcd: 645.2993, Found: 645.2994.

4. Procedures for radical mechanism experiment



A 5 mL round bottomed flask equipped with a stirring bar was charged with **1a** (18 mg, 0.15 mmol, 1.5 equiv), **2b** (22 mg, 0.1 mmol, 1.0 equiv), AIBN (32.8 mg, 0.2 mmol, 2.0 equiv) and DCE (1.0 mL) followed by sequential addition of three radical-trappingreagents, TEMPO (62.5 mg, 0.4 mmol, 4.0 equiv), BHT (88 mg, 0.4 mmol, 4.0 equiv) or 1,1-diphenylethylene (72 mg, 0.4 mmol, 4.0 equiv), respectively. The reaction was allowed to stir at 50 °C under an argon atmosphere. After 24 h, no product **3a**was detected (eq. 1, 2 and 3).

$$1a + 2b \xrightarrow{\text{standard}} 3a + N \equiv H + N \equiv H \equiv H = 100 \text{ (4)}$$

$$88\% \quad 4 \qquad 5$$

A 5 mL round bottomed flask equipped with a stirring bar was charged with **1a** (18 mg, 0.15 mmol, 1.5 equiv), **2b** (22 mg, 0.1 mmol, 1.0 equiv), AIBN (32.8 mg, 0.2 mmol, 2.0 equiv), and DCE (1.0 mL). The reaction was allowed to stir at 50 °C under an argon atmosphere. After 24 h, not only **3a** is generated, but isobutyronitrile**4** and 2,2,3,3-tetramethylsuccinonitrile **5**were detected in the reaction mixture by ESI-HRMS analysis (eq. 4). **4**: HRMS (ESI) for C₄H₇NNa (M+Na⁺): Calcd: 92.0471, Found: 92.0470;**5**: HRMS (ESI) for C₈H₁₃N₂ (M+H⁺): Calcd: 137.1073, Found: 137.1073.



Mass Spectrum SmartFormula Report											
Analysis	Info	E-\WYO_20200618-GR	1-200 d	Acquisition Date	2020/6/18 15:18:05						
Method Sample N Comment	ame	tune_low 50-500.m	L-2277.U	Operator Instrument / Ser#	service micrOTOF-Q II 10)280					
Acquisiti Source Typ Focus Scan Begin Scan End	on Parameter ^{De}	ESI Active 50 m/z 1500 m/z	Ion Polarity Set Capillary Set End Plate Offset Set Collision Cell RF	Positive 4500 V -500 V 110.0 Vpp	Set Nebulizer Set Dry Heater Set Dry Gas Set Divert Valve	0.3 Bar 180 °C 4.0 I/min Source					
100- 0-200-	<u>~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~</u>	136.0629	- <u></u>		137.1073	+MS, 0.1	0.2min #(8-9)				
	Meas. m/z	# Formula	m/z err Mean [ppm] err [ppm]	rdb N-Ru ej¥Conf m le	Sigma Std I Mi	Std Std I Std m/ ean VarNorm Di m/z	z Std ff Comb Dev				
	107.1070	1 C8H13N2	137.1073 0.0 0.0	3.5 ok even	54.22 0.0884 0.0	000 0.0475 0.000	0 0.8427				



A 5 mL round bottomed flask equipped with a stirring bar was charged with **1f** (21 mg, 0.15 mmol, 1.5 equiv), **2b** (22 mg, 0.1 mmol, 1.0 equiv), AIBN (32.8 mg, 0.2 mmol, 2.0 equiv), TEMPO (15.6 mg, 0.1 mmol, 1.0 equiv), and DCE (1.0 mL). The reaction was allowed to stir at 50 °C under an argon atmosphere. After 24 h, no product **3f**was detected, while the isobutyronitrile**4**, 2,2,3,3-tetramethylsuccinonitrile **5**and 2,2,6,6-tetramethylpiperidin-1-yl 4-chlorobenzoate **6**were detected in the reaction mixture by ESI-HRMS analysis (eq. 5). **4**: HRMS (ESI) for C₄H₇NNa (M+Na⁺): Calcd: 92.0471, Found: 92.0469;**5**: HRMS (ESI) for C₈H₁₃N₂ (M+H⁺): Calcd: 137.1073, Found: 137.1070; **6**: HRMS (ESI) for C₁₆H₂₂ClNO₂Na (M+Na⁺): Calcd: 318.1231, Found: 318.1232.

				Ma	ss Sp	ectru	im Sr	mart	Formu	la Rep	ort				
Analysis Info									Acquisition Date		20	2020/6/18 15:25:26			
Analysis Name F:\WYQ-20200618-GRL-3a.d				L-3a.d											
Method		tune	e_low 50-500.m							Oper	rator	Se	ervice		~~
Sample Name Comment									Insu	ument / Ser#	m		1 102	80	
Acquisiti	on Parameter														
Source Type			ESI		Ion Polarity	'		Posit	ive		Set Nebuliz	ter	0.	3 Bar	
Focus Soon Pagin		Active			Set End Plate Offset			4500 V			Set Dry Heater		180 °C		
Scan End			1500 m/z		Set Collision Cel		110.0 Vpp			Set Divert Valve		Source			
Intens.														+MS, 0.5-0.5	min #(28-29)
400-															
-			90.9746												
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	Maga m/z	#	Formula	m/7	0.55	Mean	rdb	N Du	o.VConf	m Ciama	Ctd I	Ctd	Std I	Std m/z	Ctd
	Weds. III/Z	#	Formula	111/2	[ppm]	err	IUD	le	el+00III	moigina	3101	Mean	VarNorm	Diff	Comb
						[ppm]						m/z			Dev
	92.0469	1	C4H7NNa	92 0471	21	21	15	ok	even	27 11	0.0418	0.0002	0.0253	0.0002	0 8427





A 5 mL round bottomed flask was charged with **1a** (18 mg, 0.15 mmol, 1.5 equiv), **2b** (22 mg, 0.1 mmol, 1.0 equiv), and DCE (0.5 mL). The reaction was stirred under an argon atmosphere at room temperature. After 12 h, it was found that no product **3a**was detected (eq. 6).

A 5 mL round bottomed flask was charged with **1a** (18 mg, 0.15 mmol, 1.5 equiv), **2b** (22 mg, 0.1 mmol, 1.0 equiv), and DCE (0.5 mL). The reaction was stirred with 23 W white LED bulb irradiation under an argon atmosphere at room temperature. After 24 h, the solvent was evaporated in vacuo. The residue was purified by flash chromatography on silica gel to afford **3a** in 13% yield.

A 5 mL round bottomed flask was charged with **1a** (18 mg, 0.15 mmol, 1.5 equiv), **2b** (22 mg, 0.1 mmol, 1.0 equiv), and DCE (0.5 mL). The reaction was stirred with 10 W blue LED bulb irradiation under an argon atmosphere at room temperature. After 24 h, the solvent was evaporated in vacuo. The residue was purified by flash chromatography on silica gel to afford **3a** in 25% yield.

5. References

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6. ¹H, ¹⁹F, ¹³C NMR spectra of compounds 2b – 3cc



¹H NMR spectra of compound **2b**





¹³C NMR spectra of compound **2b**



¹H NMR spectra of compound **2c**



¹⁹F NMR spectra of compound **2c**



¹³C NMR spectra of compound **2c**



¹H NMR spectra of compound **3a**



¹⁹F NMR spectra of compound **3a**



¹³C NMR spectra of compound **3a**



¹H NMR spectra of compound **3b**



¹⁹F NMR spectra of compound **3b**



¹³C NMR spectra of compound **3b**



¹H NMR spectra of compound **3c**



¹⁹F NMR spectra of compound **3c**



¹³C NMR spectra of compound **3c**



¹H NMR spectra of compound **3d**



¹⁹F NMR spectra of compound **3d**



¹³C NMR spectra of compound **3d**


¹H NMR spectra of compound **3e**



¹⁹F NMR spectra of compound **3e**



¹³C NMR spectra of compound **3e**



¹H NMR spectra of compound **3f**



¹⁹F NMR spectra of compound **3f**



¹³C NMR spectra of compound **3f**



¹H NMR spectra of compound **3g**



¹⁹F NMR spectra of compound **3g**



¹³C NMR spectra of compound **3g**



¹H NMR spectra of compound **3h**



¹⁹F NMR spectra of compound **3h**



¹³C NMR spectra of compound **3h**







¹⁹F NMR spectra of compound **3i**



¹³C NMR spectra of compound **3i**



¹H NMR spectra of compound **3**j







¹³C NMR spectra of compound **3**j



¹H NMR spectra of compound **3**k



¹⁹F NMR spectra of compound **3**k



¹³C NMR spectra of compound **3**k



¹H NMR spectra of compound **3**l







¹³C NMR spectra of compound **3**I



¹H NMR spectra of compound **3m**



¹⁹F NMR spectra of compound **3m**



¹³C NMR spectra of compound **3m**



¹H NMR spectra of compound **3n**



¹⁹F NMR spectra of compound **3n**



¹³C NMR spectra of compound **3n**



¹H NMR spectra of compound **30**



¹⁹F NMR spectra of compound **30**



¹³C NMR spectra of compound **30**



¹H NMR spectra of compound **3p**



¹⁹F NMR spectra of compound **3p**



¹³C NMR spectra of compound **3p**



¹H NMR spectra of compound **3q**



¹⁹F NMR spectra of compound **3q**



¹³C NMR spectra of compound **3q**



¹H NMR spectra of compound **3r**



 $^{19}\mathrm{F}$ NMR spectra of compound 3r



¹³C NMR spectra of compound 3r



¹H NMR spectra of compound **3s**



¹⁹F NMR spectra of compound **3s**



¹³C NMR spectra of compound **3s**



¹H NMR spectra of compound **3t**



¹⁹F NMR spectra of compound **3t**



¹³C NMR spectra of compound **3t**



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¹H NMR spectra of compound **3u**



 $^{19}\mathrm{F}$ NMR spectra of compound $\mathbf{3u}$



S60

¹³C NMR spectra of compound **3u**



¹H NMR spectra of compound 3v



 $^{19}\mathrm{F}$ NMR spectra of compound 3v



¹³C NMR spectra of compound **3v**



¹H NMR spectra of compound **3**w



¹⁹F NMR spectra of compound **3w**



¹³C NMR spectra of compound **3**w



¹H NMR spectra of compound 3x







¹³C NMR spectra of compound **3**x



¹H NMR spectra of compound **3aa**



¹⁹F NMR spectra of compound **3aa**



¹³C NMR spectra of compound **3aa**



¹H NMR spectra of compound **3ab**



¹⁹F NMR spectra of compound **3ab**



¹³C NMR spectra of compound **3ab**



¹H NMR spectra of compound **3ac**



¹⁹F NMR spectra of compound **3ac**



¹³C NMR spectra of compound **3ac**



¹H NMR spectra of compound **3ad**



¹⁹F NMR spectra of compound **3ad**



¹³C NMR spectra of compound **3ad**


$^1\mathrm{H}$ NMR spectra of compound 3ae



¹⁹F NMR spectra of compound **3ae**



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¹³C NMR spectra of compound **3ae**



¹H NMR spectra of compound **3af**



¹⁹F NMR spectra of compound **3af**



¹³C NMR spectra of compound **3af**







¹⁹F NMR spectra of compound **3ag**



¹³C NMR spectra of compound **3ag**



¹H NMR spectra of compound **3ah**



¹⁹F NMR spectra of compound **3ah**



¹³C NMR spectra of compound **3ah**



¹H NMR spectra of compound **3ai**



¹⁹F NMR spectra of compound **3ai**



¹³C NMR spectra of compound **3ai**



¹H NMR spectra of compound **3ba**







¹³C NMR spectra of compound **3ba**







¹⁹F NMR spectra of compound **3bb**



¹³C NMR spectra of compound **3bb**



¹H NMR spectra of compound **3bc**







¹³C NMR spectra of compound **3bc**



¹H NMR spectra of compound **3bd**



¹⁹F NMR spectra of compound **3bd**



¹³C NMR spectra of compound **3bd**



¹H NMR spectra of compound **3be**



¹⁹F NMR spectra of compound **3be**



¹³C NMR spectra of compound **3be**



¹H NMR spectra of compound **3bf**



¹⁹F NMR spectra of compound **3bf**



¹³C NMR spectra of compound **3bf**



¹H NMR spectra of compound **3bg**



¹⁹F NMR spectra of compound **3bg**



¹³C NMR spectra of compound **3bg**



¹H NMR spectra of compound **3ca**



¹⁹F NMR spectra of compound **3ca**



¹³C NMR spectra of compound **3ca**



¹H NMR spectra of compound **3cb**



¹⁹F NMR spectra of compound **3cb**



¹³C NMR spectra of compound **3cb**



¹H NMR spectra of compound **3cc**



¹⁹F NMR spectra of compound **3cc**



¹³C NMR spectra of compound **3cc**



7. X-ray crystalstructure of compounds 3g and 3ad

X-ray Crystal Structure of *Se*-(difluoromethyl)-4-bromobenzoselenoate (3g)(CCDC:1985689)



X-ray Crystal Structure of *Se*-(difluoromethyl)-1-tosyl-1H-indole-3-carboselenoate (3ad)(CCDC:1985690)

