Hydroselenation of Alkynes by Lithium Butylselenolate: An Approach in the Synthesis of Vinylic Selenides

Gilson Zeni,^{a,*} Marcelo P. Stracke,^a Cristina W. Nogueira,^a
Antonio L. Braga,^a Paulo H. Menezes,^b Helio A. Stefani^c

^aLaboratório de Síntese, Reatividade, Avaliação, Toxicológica e Farmacológica de
Organocalcogenios-CCNE-UFSM – 97105-900-Santa Maria - RS – Brazil
gzeni@quimica.ufsm.br

^bDepartamento de Química Fundamental – UFPE – Recife – Brazil

^cFaculdade de Ciências Farmacêuticas – USP - Sao Paulo - Brazil

SUPPORTING INFORMATION

Materials and Methods

Proton nuclear magnetic resonance spectra (¹H NMR) were obtained at 200 MHz on a Bruker DPX-200 NMR spectrometer or at 400 MHz on a Bruker DPX-400 NMR spectrometer. Spectra were recorded in CDCl₃ solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl₃ or tetramethylsilane (TMS) as the external reference. Data are reported as follows: chemical shift (δ), multiplicity, coupling constant (*J*) in Hertz and integrated intensity. Carbon-13 nuclear magnetic resonance spectra (¹³C NMR) were obtained either at 50 MHz on a Bruker DPX-200 NMR spectrometer or at 100 MHz on a Bruker DPX-400 NMR spectrometer. Spectra were recorded in CDCl₃ solutions. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl₃. Abbreviations to denote the multiplicity of a particular signal are s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), sex (sextet) and m (multiplet). High resolution mass spectra were recorded on a Kratos MS50TC double focusing magnetic sector mass spectrometer using EI at 70 eV. Column chromatography was performed using Merck Silica Gel (230-400 mesh) following the methods described by Still¹. Thin layer chromatography (TLC) was performed using Merck Silica Gel GF₂₅₄, 0.25 mm thickness. For visualization, TLC plates were either placed under ultraviolet light, or stained with iodine vapor, or acidic vanillin. Most reactions were monitored by TLC for disappearance of starting material. The following solvents were dried and purified by distillation from the reagents indicated: tetrahydrofuran from sodium with a benzophenone ketyl indicator. All other solvents were ACS or HPLC grade unless otherwise noted. Air- and moisture-

¹ Still, W.C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1975**, *43*, 2923-2925

sensitive reactions were conducted in flame-dried or oven dried glassware equipped with tightly fitted rubber septa and under a positive atmosphere of dry nitrogen or argon. Reagents and solvents were handled using standard syringe techniques. Temperatures above room temperature were maintained by use of a mineral oil bath with an electrically heated coil connected to a Variac controller.

Typical procedure for hydroselenation of alkynes by [BuSeLi]: To a suspension of the elemental selenium (0.079 g; 1 mmol) in dry THF (5 mL) under argon and magnetic stirring was added *n*-butyllithium (0.4 mL of a 2.5 M solution in hexane, 1 mmol). A yellow solution was formed. To this solution was added the appropriate alkynes (1 mmol) in ethanol (10 mL). The mixture was then heated at reflux for 24 h. After this time, the mixture was cooled to room temperature and diluted with ethyl acetate (20 mL) and washed with brine (2x20 mL). The organic phase was separated, dried over MgSO₄ and concentrated under vacuum. The residue was purified by flash chromatography and eluted with hexane/ethyl acetate (80:20). **Selected spectral and analytical data for 2a - (1-Butylselanyl-2-phenyl-vinyl)-benzene:** Yield: 0.189 g (60%). ¹H RMN: CDCl₃, 400 MHz, δ(ppm): 7.50-7.17 (m, 11H), 2.81 (t, *J* = 7.3 Hz, 2H), 1.53 (quint, *J* = 7.3 Hz, 2H), 1.34 (sex, *J* = 7.3 Hz, 2H), 0.87 (t, *J* = 7.2 Hz, 3H). ¹³C RMN: CDCl₃, 100 MHz, δ(ppm): 137.85, 134.44, 131.28, 128.01, 123.02, 32.74, 29.76, 22.30, 13.29. IR (KBr, film) ν 3028, 2892, 1702, 1597, 1492, 1210, 954. MS (EI, 70 eV) *m/z* (relative intensity): 258 (12), 180 (100), 102 (77), 77 (45), 57 (21). HRMS Calcd. For C₁₈H₂₀Se: 316.07221. Found: 316.07148.

4-Butylselanyl-3,8-dimethyl-dec-4-en-6-yne-3,8-diol – 2b: Yield: 0.182 g (55%). ¹H RMN: CDCl₃, 400 MHz, δ(ppm): 6.33 (s, 1H), 3.04 (t, *J* = 7.3 Hz, 2H), 2.23 (s, 2H), 1.73-1.69 (m, 4H), 1.52-1.38 (m, 8H), 1.09-1.01 (m, 5H), 0.93 (t, *J* = 7.3 Hz, 3H), 0.86 (t, *J* = 7.3 Hz, 3H). ¹³C RMN: CDCl₃, 100 MHz, δ(ppm): 153.34, 113.44, 99.60, 82.86, 81.40, 69.22, 67.42, 36.46, 34.00, 32.43, 29.09, 28.33, 26.52, 22.98, 13.57, 9.01, 8.24. IR (KBr, film) ν 3356, 3029, 2875, 1661, 1492, 1210, 957. MS (EI, 70 eV) *m/z* (relative intensity): 314 (44), 296 (17), 275 (23), 196 (100), 178 (54), 160 (33). HRMS Calcd. for C₁₆H₂₈O₂S: 332.13251. Found: 332.13322.

2-Butylselanyl-hex-2-en-4-yne-1,6-diol – 2c: Yield: 0.170 g (69%). ¹H RMN: CDCl₃, 200 MHz, δ(ppm): 6.16 (t, *J* = 1.7 Hz, 1H), 4.43 (s, 2H), 4.28 (s, 2H), 2.97-2.87 (m, 4H), 1.74 (quint, *J* = 7.2 Hz, 2H), 1.51 (sex, *J* = 7.2 Hz, 2H), 0.96 (t, *J* = 7.2 Hz, 3H). ¹³C RMN: CDCl₃, 100 MHz, δ(ppm): 146.80, 109.04, 94.55, 82.80, 66.01, 51.45, 32.67, 25.58, 22.85, 13.50. IR (KBr, film) ν 3380, 3058, 2873, 1595, 1492, 1218, 954. MS (EI, 70 eV) *m/z* (relative intensity): 230 (40), 212 (15), 191 (34), 112 (100), 94 (22), 76 (31). HRMS Calcd. for C₁₀H₁₆O₂Se: 248.03653. Found: 248.03781.

(2-Butylselanyl-hex-1-enyl)-benzene – 2e: Yield: 0.141 g (48%). ¹H RMN: CDCl₃, 400 MHz, δ(ppm): 7.38-7.17 (m, 5H), 6.89 (d, *J* = 2.3 Hz, 1H), 2.90 (t, *J* = 7.3 Hz, 2H), 2.39 (t, *J* = 7.2 Hz, 2H), 1.58-1.40 (m, 8H), 0.88 (t, *J* = 7.3 Hz, 3H). ¹³C RMN: CDCl₃, 100 MHz, δ(ppm): 134.01, 128.74, 128.39, 128.18, 128.13, 128.01, 127.27, 124.09, 32.98, 30.78, 29.74, 22.90, 22.53, 18.99, 13.67, 13.41. IR (KBr, film) ν 3054, 2933, 1642, 1447, 1265,

935. MS (EI, 70 eV) m/z (relative intensity): 239 (21), 160 (100), 83 (38), 77 (45). HRMS Calcd. for $C_{16}H_{24}Se$: 296.10582. Found: 296.10741.

2-Butylselanyl-3-phenyl-prop-2-en-1-ol – 2f: Yield: 0.175 g (65%). 1H RMN: $CDCl_3$, 200 MHz, δ (ppm): 7.53-7.23 (m, 5H), 7.03 (s, 1H), 4.47 (s, 2H), 2.77 (t, $J = 7.2$ Hz, 2H), 2.23 (s, 1H), 1.65 (quint, $J = 7.3$ Hz, 2H), 1.42 – 1.27 (m, 2H), 0.81 (t, $J = 7.3$ Hz, 3H). ^{13}C RMN: $CDCl_3$, 50 MHz, δ (ppm): 136.73, 132.97, 131.14, 131.60, 129.03, 128.23, 127.95, 127.30, 67.96, 32.35, 25.20, 23.81, 13.46. IR (KBr, film) ν 3389, 2925, 2853, 1738, 1487, 1442, 952. MS (EI, 70 eV) m/z (relative intensity): 252 (40), 213 (23), 134 (100), 116 (10). HRMS Calcd. for $C_{13}H_{18}OSe$: 270.05139. Found: 270.05211.

(1-Butylselanyl-oct-1-en-3-ynyl)-benzene – 2g: Yield: 0.210 g (66%). 1H RMN: $CDCl_3$, 400 MHz, δ (ppm): 7.5-7.2 (m, 5H), 6.91 (s, 1H), 2.89 (t, $J = 7.3$, 2H), 2.38 (t, $J = 7.3$ Hz, 2H), 1.68 (quint, $J = 7.3$ Hz, 2H), 1.56-1.42 (m, 6H), 0.96 (t, $J = 7.3$ Hz, 6H). ^{13}C RMN: $CDCl_3$, 100 MHz, δ (ppm): 136.77, 132.48, 128.92, 128.31, 127.30, 126.21, 124.71, 122.16, 84.82, 65.05, 34.60, 32.58, 31.45, 22.64, 21.95, 19.27, 13.82, 13.51. IR (KBr, film) ν 3055, 2957, 2863, 1631, 1454, 959. MS (EI, 70 eV) m/z (relative intensity): 263 (35), 184 (100), 107 (75), 77 (41). HRMS Calcd. for $C_{18}H_{24}Se$: 320.10557. Found: 320.10682.

(1-Butylselanyl-4-phenyl-but-1-en-3-ynyl)-benzene – 2h: Yield: 0.227 g (67%). 1H RMN: $CDCl_3$, 200 MHz, δ (ppm): 7.54-7.29 (m, 10H), 6.26 (s, 1H), 2.64 (t, $J = 6.7$ Hz, 2H), 1.61 (quint, $J = 7.2$ Hz, 2H), 1.37 (sex, $J = 7.2$ Hz, 2H), 0.79 (t, $J = 7.2$ Hz, 3H). ^{13}C RMN: $CDCl_3$, 100 MHz, δ (ppm): 147.77, 139.89, 136.10, 131.24, 128.05, 127.41, 126.09, 125.83, 123.44, 107.93, 96.70, 88.40, 32.36, 26.41, 22.77, 13.35. IR (KBr, film) ν 3020, 2870, 2212, 1631, 1459, 1199, 960. MS (EI, 70 eV) m/z (relative intensity): 283 (28), 204 (100), 127 (29), 77 (40). HRMS Calcd. for $C_{20}H_{20}Se$: 340.07119. Found: 340.07233.

3-Butylselanyl-2-methyl-6-phenyl-hex-3-en-5-yn-2-ol – 2i: Yield: 0.202 g (63%). 1H RMN: $CDCl_3$, 400 MHz, δ (ppm): 7.46-7.28 (m, 5H), 6.56 (s, 1H), 3.13 (t, $J = 7.3$ Hz, 2H), 2.5 (s, 1H), 1.69 (quint, $J = 7.3$ Hz, 2H), 1.46-1.44 (m, 8H), 0.87 (t, $J = 7.3$ Hz, 3H). ^{13}C RMN: $CDCl_3$, 100 MHz, δ (ppm): 155.44, 131.12, 128.98, 128.64, 128.18, 128.14, 126.87, 123.27, 112.26, 96.04, 87.63, 74.58, 32.26, 29.04, 28.33, 22.80, 13.40. IR (KBr, film) ν 3330, 2964, 2871, 1721, 1460, 1266, 958. MS (EI, 70 eV) m/z (relative intensity): 304 (21), 265 (44), 186 (100), 168 (75), 77 (11). HRMS Calcd. for $C_{17}H_{22}OSe$: 322.085818. Found: 322.087731.

1-Butylselanyl-1-methylsulfanyl-hex-1-ene – 2j: Yield: 0.168 g (59%). 1H RMN: $CDCl_3$, 400 MHz, δ (ppm): 7.53-7.24 (m, 5H), 6.20 (s, 1H), 3.1 (t, $J = 7.2$ Hz, 2H), 2.39-2.31 (m, 3H), 1.71-1.39 (m, 4H), 0.94 (t, $J = 7.3$ Hz, 3H). ^{13}C RMN: $CDCl_3$, 100 MHz, δ (ppm): 133.15, 129.03, 128.51, 128.39, 128.23, 127.95, 127.30, 123.19, 33.69, 29.69, 23.02, 17.41, 13.57. IR (KBr, film) ν 2959, 2864, 1607, 1455, 1237, 954. MS (EI, 70 eV) m/z (relative intensity): 229 (17), 150 (100), 103 (75). HRMS Calcd. for $C_{13}H_{18}SSe$: 286.03547. Found: 286.03692.

(2-Butylselanyl-hex-1-enyl)-phosphonic acid diethyl ester – 2l: Yield: 0.227 g (64%). ¹H RMN: CDCl₃, 200 MHz, δ(ppm): 5.93 (d, *J* = 13.6 Hz, 1H), 5.46 (d, *J* = 13.6 Hz, 1H), 4.14 (dq, ³*J*_{PH} = 7.4 Hz, *J* = 7.2 Hz, 4H), 3.02 (t, *J* = 7.3 Hz, 4H), 1.73-1.25 (m, 14H), 0.96-0.92 (m, 6H). ¹³C RMN: CDCl₃, 100 MHz, δ(ppm): 164.45 (d, ²*J*_{P-C} = 6.3 Hz), 139.35, 114.25 (d, ¹*J*_{P-C} = 188 Hz), 62.04 (d, ²*J*_{P-C} = 5 Hz), 36.37, 32.46, 30.65, 25.92, 23.12, 22.93, 22.29, 21.92, 16.38 (d, ³*J*_{P-C} = 6 Hz), 13.81, 13.486. IR (KBr, film) ν 3024, 2861, 1596, 1488, 1254, 1133, 1010, 955. MS (EI, 70 eV) *m/z* (relative intensity): 299 (23), 220 (100), 130 (31), 84 (17). HRMS Calcd. for C₁₄H₂₉O₃PSe: 356.10677. Found: 356.10812.

(2-Butylselanyl-vinyl)-benzene – 5a: Yield 0.191 g (80%); ¹H RMN: CDCl₃, 200 MHz, δ(ppm): 7.42-7.17 (m, 5H); 6.89 (d, *J* = 10.6 Hz; 1H); 6.62 (d, *J* = 10.6 Hz; 1H); 2.81 (t, *J* = 7.3 Hz, 2H); 1.81 (quint, *J* = 7.3 Hz, 2H), 1.52 (sex, *J* = 7.3 Hz, 2H), 0.95 (t, *J* = 7.2 Hz, 3H). ¹³C RMN: CDCl₃, 100 MHz, δ(ppm): 137.60, 129.21, 128.19, 126.76, 123.12, 32.72, 28.94, 22.71, 13.50. IR (KBr, film) ν 3064, 2850, 1589, 1493, 955. MS (EI, 70 eV) *m/z* (relative intensity): 183 (25), 104 (100), 77 (35). HRMS Calcd. for C₁₂H₁₆Se: 240.04178. Found: 240.04252.

4-Butylselanyl-2-methyl-but-3-en-2-ol – 5c: Yield: 0.139 g (63%). ¹H RMN: CDCl₃, 200 MHz, δ(ppm): 6.24 (d, *J* = 10.3 Hz, 1H), 6.01 (d, *J* = 10.3 Hz, 1H), 2.72 (t, *J* = 7.2 Hz, 2H), 2.18 (s, 1H), 1.76 (quint, *J* = 7.2 Hz, 2H), 1.45-1.26 (m, 8H), 0.96 (t, *J* = 7.2 Hz, 3H). ¹³C RMN: CDCl₃, 50 MHz, δ(ppm): 137.34, 119.06, 72.46, 32.64, 29.65, 27.71, 23.08, 13.48. IR (KBr, film) ν 3289, 3025, 2891, 1709, 1601, 1499, 1213, 949. MS (EI, 70 eV) *m/z* (relative intensity): 204 (32), 165 (21), 86 (100), 68 (57). HRMS Calcd. for C₉H₁₈OSe: 222.05237. Found: 222.05381.

1-Butylselanyl-3-methyl-pent-1-en-3-ol – 5d: Yield: 0.145 g (62%). ¹H RMN: CDCl₃, 400 MHz, δ(ppm): 6.26 (d, *J* = 10.2 Hz, 1H), 5.91 (d, *J* = 10.2 Hz, 1H), 2.62 (t, *J* = 7.2 Hz, 2H), 2.04 (s, 1H), 1.68-1.60 (m, 4H), 1.48-1.25 (m, 2H), 1.31 (s, 3H), 0.94 (t, *J* = 7.2 Hz, 6H). ¹³C RMN: CDCl₃, 100 MHz, δ(ppm): 137.39, 119.62, 71.15, 35.11, 32.68, 29.65, 29.59, 27.23, 13.52, 8.12. IR (KBr, film) ν 3361, 3033, 2881, 1658, 1488, 1207, 937. MS (EI, 70 eV) *m/z* (relative intensity): 218 (34), 179 (45), 100 (100), 82 (41). HRMS Calcd. for C₁₀H₂₀OSe: 236.06798. Found: 236.06843.

2-Butylselanyl-pent-1-en-3-ol – 5e: Yield: 0.146 g (78%). ¹H RMN: CDCl₃, 200 MHz, δ(ppm): 5.96 (s, 1H), 5.12 (s, 1H), 4.08-4.05 (m, 1H), 2.88-2.51 (m, 2H), 2.3 (s, 1H), 1.79-1.6 (m, 4), 1.43-1.60 (m, 2H), 0.96 (t, *J* = 7.2 Hz, 6H). ¹³C RMN: CDCl₃, 50 MHz, δ(ppm): 134.50, 122.33, 72.22, 32.69, 29.21, 26.79, 22.95, 13.37, 9.35. IR (KBr, film) ν 3382, 3049, 2865, 1589, 1487, 1219, 929. MS (EI, 70 eV) *m/z* (relative intensity): 204 (27), 165 (55), 86 (100). HRMS Calcd. for C₉H₁₈OSe: 222.05231. Found: 222.05312.

1-(2-Butylselanyl-vinyl)-cyclohexanol – 5f: Yield: 0.156 g (60%). ¹H RMN: CDCl₃, 400 MHz, δ(ppm): 6.24 (d, *J* = 10.2 Hz, 1H), 6.01 (d, *J* = 10.2 Hz, 1H), 3.08 (s, 1H), 2.62 (t, *J* = 7.2 Hz, 2H), 1.93-1.89 (m, 2H), 1.69-1.4 (m, 10H), 1.25-1.22 (m, 2H), 0.91 (t, *J* = 7.2 Hz, 3H). ¹³C RMN: CDCl₃, 100 MHz, δ(ppm): 136.58, 119.49, 74.83, 37.22, 32.45, 27.56,

25.15, 21.84, 21.74, 13.31. IR (KBr, film) ν 3391, 3047, 2922, 1637, 1440, 1260, 930. MS (EI, 70 eV) m/z (relative intensity): 244 (17), 205 (41), 126 (100), 108 (25). HRMS Calcd. for $C_{12}H_{22}OSe$: 262.08367. Found: 262.08454.

1-(2-Butylselanyl-vinyl)-piperidine – 5g: Yield: 0.126 g (51%). 1H RMN: $CDCl_3$, 200 MHz, δ (ppm): 6.39 (dt, $J = 9.5$ Hz, $J = 1.3$ Hz, 1H), 6.31 (dt, $J = 9.4$ Hz, $J = 4.9$ Hz, 1H), 2.89 (dd, $J = 4.9$ Hz, $J = 1.2$ Hz, 2H), 2.35-2.29 (m, 6H), 1.66-1.18 (m, 8H), 0.89 (t, $J = 7.2$ Hz, 3H). ^{13}C RMN: $CDCl_3$, 100 MHz, δ (ppm): 131.24, 109.98, 63.56, 61.40, 32.56, 23.03, 22.02, 13.68, 7.02. IR (KBr, film) ν 3050, 2961, 2850, 1627, 1449, 951. MS (EI, 70 eV) m/z (relative intensity): 206 (42), 127 (100). HRMS Calcd. for $C_{11}H_{21}NOSe$: 263.07889. Found: 263.07911.

2-Butylselanyl-prop-2-en-1-ol – 6h: Yield: 0.152 g (79%). 1H RMN: $CDCl_3$, 200 MHz, δ (ppm) 5.71 (s, 1H), 5.26 (s, 1H), 4.35 (s, 2H), 2.79 (t, $J = 7.2$ Hz, 3H), 1.76 (quint, $J = 7.2$ Hz, 2H), 1.51 (sex, $J = 7.2$ Hz, 2H), 0.96 (t, $J = 7.2$ Hz, 3H). ^{13}C RMN: $CDCl_3$, 50 MHz, δ (ppm): 140.98, 114.18, 66.74, 32.71, 24.99, 22.86, 13.38. IR (KBr, film) ν 3377, 3017, 2955, 2863, 1627, 1448, 1179, 968. MS (EI, 70 eV) m/z (relative intensity): 176 (55), 137 (24), 58 (100). HRMS Calcd. for $C_7H_{14}OSe$: 194.02109. Found: 194.02191.

(2-Butylselanyl-vinyl-1-d1)-benzene - 7: Yield 0.192 g (80%); 1H RMN: $CDCl_3$, 200 MHz, δ (ppm): 7.43-7.21 (m, 5H); 6.60 (s, 1H); 2.82 (t, $J = 7.3$ Hz, 2H); 1.81-1.38 (m, 4H), 0.86 (t, $J = 7.2$ Hz, 3H). ^{13}C RMN: $CDCl_3$, 50 MHz, δ (ppm): 128.62, 128.23, 126.80, 123.12, 32.77, 28.65, 22.75, 13.55. IR (KBr, film) ν 3050, 3017, 2950, 2921, 2863, 1592, 1582, 1561, 1480, 1452, 1030, 1029, 765, 788. MS (EI, 70 eV) m/z (relative intensity): 184 (33), 105 (100), 77 (44). HRMS Calcd. for $C_{12}H_{15}DSe$: 241.04795. Found: 241.04852.

2-Butylselanyl-3-phenyl-prop-2-en-1-ol-d - 9: Yield: 0.176 g (65%). 1H RMN: $CDCl_3$, 200 MHz, δ (ppm): 7.50-7.22 (m, 5H), 4.45 (s, 2H), 3.06 (s, 1H), 2.78 (t, $J = 7.2$ Hz, 2H), 1.59-1.37 (m, 4H), 0.88 (t, $J = 7.3$ Hz, 3H). ^{13}C RMN: $CDCl_3$, 50 MHz, δ (ppm): 136.57, 132.43, 131.4, 128.85, 128.10, 127.73, 127.00, 67.40, 32.08, 24.30, 22.58, 13.27. IR (KBr, film) ν 3385, 3063, 3021, 2957, 2930, 2875, 1587, 1593, 1572, 1479, 1461, 1033, 1021, 760, 781. MS (EI, 70 eV) m/z (relative intensity): 253 (41), 214 (37), 135 (100), 117 (18), 77 (14). HRMS Calcd. for $C_{13}H_{17}DOSe$: 271.05858. Found: 271.05923.

Procedure for [2-Butylselanyl-1-(hydroxy-phenyl-methyl)-hex-1-enyl]-phosphonic acid diethyl ester - 11: To a suspension of the elemental selenium (0.079 g, 1 mmol) in dry THF (5 mL) under argon and magnetic stirring was added *n*-butyllithium (0.4 mL of a 2.5 M solution in hexane; 1 mmol). A yellow solution was formed. Then alkynylphosphonate (0.286 g, 1.2 mmol) is added and after 1 h the benzaldehyde (0.107g, 1mmol) is added and the solution is stirring at room temperature for 4 h. After this time the mixture is diluted with ethyl acetate (20 mL) and washed with brine (2x20 mL). The organic layer is separated, dried ($MgSO_4$) and the solvent is evaporated. The residue is purified by silica gel column chromatography eluting with hexane. Yield: 0.369 g (78%). 1H RMN: $CDCl_3$, 400 MHz, δ (ppm): RMN 1H : $CDCl_3$, 200 MHz, δ (ppm): 7.44-7.20 (m, 5H), 4.68 (s, 1H), 4.12 (dq, $^3J_{PH} = 7.4$ Hz, $J = 7.2$ Hz, 4H), 2.78 (t, $J = 7.3$ Hz, 4H), 1.71-1.30 (m, 14H), 0.95 (t, J

= 7.3 Hz, 6H). ^{13}C RMN: CDCl_3 , 100 MHz, $\delta(\text{ppm})$: 157.71 (d, $^2J_{\text{P-C}} = 6.3$ Hz), 142.78, 128.38, 128.03, 127.37, 126.84, 126.81, 125.63, 110.01 (d, $^1J_{\text{P-C}} = 188$ Hz), 75.8, 61.92 (d, $^2J_{\text{P-C}} = 5$ Hz), 36.45, 32.48, 32.23, 30.69, 25.64, 23.12, 22.73, 16.33 (d, $^3J_{\text{P-C}} = 6$ Hz), 13.90, 13.48. MS (EI, 70 eV) m/z (relative intensity): 444 (45), 405 (21), 326 (100), 238 (18), 191 (75), 173 (62), 82 (35), 77 (39). HRMS Calcd. for $\text{C}_{21}\text{H}_{35}\text{O}_4\text{PSe}$: 462.14382. Found: 462.14412.

SELECTED SPECTRA

Current Data Parameters
 NAME z3sxomcd
 EXPNO 1
 PROCNO 2003

F2 - Acquisition Parameters
 Date_ 20031000
 Time 12.38
 INSTRUM spect
 PROBHD 5 mm Dual 13
 PULPROG zgpg30
 TO 65536
 SOLVENT CDC13
 NS 1286
 DS 0
 SWH 11037.527 Hz
 FIDRES 0.168419 Hz
 AQ 2.968308 sec
 RG 32768
 DM 45.300 usec
 DE 5.00 usec
 TE 300.0 K
 D12 0.0002000 sec
 PL13 18.00 dB
 O1 1.00000000 sec
 CPDPRG2 waltz16
 PCPD2 100.00 usec
 SF02 200.1308005 MHz
 NUC2 1H
 PL2 -6.00 dB
 PL12 18.00 dB
 P1 6.50 usec
 DE 5.00 usec
 SF01 50.3279929 MHz
 NUC1 13C
 PL1 -3.00 dB
 D11 0.03000000 sec

F2 - Processing parameters
 SI 32768
 SF 50.3227353 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 22.00 cm
 F1P 214.145 ppm
 F1 10776.34 Hz
 F2P -5.190 ppm
 F2 -261.19 Hz
 PPMCM 9.96976 ppm/cm
 HZCM 501.70578 Hz/cm

Current Data Parameters
 NAME z3gm49n1
 EXPNO 3
 PROCNO 2003

F2 - Acquisition Parameters
 Date_ 20030812
 Time 20.21
 INSTRUM spect
 PROBHD 5 mm Dual 13
 PULPROG zg
 TO 65536
 SOLVENT CDC13
 NS 8
 DS 0
 SWH 4789.272 Hz
 FIDRES 0.073078 Hz
 AQ 6.8420086 sec
 RG 114
 DM 104.400 usec
 DE 4.50 usec
 TE 298.0 K
 D1 1.00000000 sec
 P1 12.00 usec
 DE 4.50 usec
 SF01 400.1322807 MHz
 NUC1 1H
 PL1 -3.00 dB

F2 - Processing parameters
 SI 32768
 SF 400.1300133 MHz
 WDW no
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.50 cm
 F1P 11.652 ppm
 F1 4662.12 Hz
 F2P -0.318 ppm
 F2 -127.16 Hz
 PPMCM 0.58387 ppm/cm
 HZCM 233.62300 Hz/cm

Current Data Parameters
NAME z3uopicd
EXPNO 1
PROCNO 2003

F2 - Acquisition Parameters
Date_ 20030708
Time 19.22
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 455
DS 0
SWH 23148.148 Hz
FIDRES 0.353213 Hz
AQ 1.4156276 sec
RG 32768
DM 21.600 usec
DE 4.50 usec
TE 300.0 K
D12 0.0000000 sec
PL13 16.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 106.00 usec
SF02 400.1316005 MHz
NUC2 1H
PL2 -3.00 dB
PL12 16.00 dB
P1 7.60 usec
DE 4.50 usec
SF01 100.6237954 MHz
NUC1 13C
PL1 -6.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 100.6127891 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 21.00 cm
F1P 179.524 ppm
F1 18062.42 Hz
F2P 1.359 ppm
F2 136.72 Hz
PPMCM 8.48406 ppm/cm
HZCM 853.60443 Hz/cm

Current Data Parameters
NAME z3sensh1
EXPNO 11
PROCNO 2003

F2 - Acquisition Parameters
Date_ 20030930
Time 22.11
INSTRUM spect
PROBHD 5 mm Dual 13
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 4789.272 Hz
FIDRES 0.073078 Hz
AQ 6.8420086 sec
RG 228.1
DM 104.400 usec
DE 4.50 usec
TE 298.0 K
D1 1.00000000 sec
P1 12.00 usec
DE 4.50 usec
SF01 400.1322807 MHz
NUC1 1H
PL1 -3.00 dB

F2 - Processing parameters
SI 32768
SF 400.1300080 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.50 cm
F1P 9.574 ppm
F1 3990.95 Hz
F2P -0.206 ppm
F2 -62.40 Hz
PPMCM 0.49659 ppm/cm
HZCM 198.69998 Hz/cm

Current Data Parameters
NAME z3j047cd
EXPNO 2
PROCNO 2003

F2 - Acquisition Parameters
Date_ 20030128
Time 19:37
INSTRUM spect
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 11427
DS 0
SWH 11037.527 Hz
FIDRES 0.168419 Hz
AQ 2.9688308 sec
RG 32768
DW 45.300 usec
DE 5.00 usec
TE 300.0 K
D12 0.00002000 sec
PL13 20.00 dB
D1 1.00000000 sec
CPDPRG2 waltz16
PCPD2 110.00 usec
SF02 200.1308005 MHz
NUC2 1H
PL2 -3.00 dB
PL12 20.00 dB
P1 11.00 usec
DE 5.00 usec
SF01 50.3279929 MHz
NUC1 13C
PL1 -3.00 dB
D11 0.03000000 sec

F2 - Processing parameters
SI 32768
SF 50.3227333 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 22.00 cm
F1P 215.000 ppm
F1 10819.39 Hz
F2P -5.000 ppm
F2 -251.61 Hz
PPMCM 10.00000 ppm/cm
HZCM 503.22736 Hz/cm