

Electronic Supplementary Information

Synthesis of Enantiomerically Pure Bis(2,2-dimethyl-1,3-dioxolanylmethyl)chalcogenides and Dichalcogenides

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General Information: The reactions were monitored by TLC carried out on Merck silica gel (60 F₂₅₄) by using UV light as visualizant agent and 5% vanillin in 10% H₂SO₄ and heat as developing agents. Baker silica gel (particle size 0.040-0.063 mm) was used for flash chromatography. Proton nuclear magnetic resonance spectra (¹H NMR) were obtained at 300 MHz on a Varian Gemini NMR and at 400 MHz on Bruker DPX 400 spectrometers. Spectra were recorded in CDCl₃ and DMSO-*d*₆ solutions. Chemical shifts are reported in ppm, referenced to tetramethylsilane (TMS) as the external reference. Coupling constants (*J*) are reported in Hertz. Carbon-13 nuclear magnetic resonance spectra (¹³C NMR) were obtained at 75 MHz on a Varian Gemini NMR and at 100 MHz on Bruker DPX 400 spectrometers. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl₃ and DMSO-*d*₆. Low-resolution mass spectra were obtained with a Shimadzu GC-MS-QP2010 mass spectrometer. Mass spectra were obtained for all compounds on a LTQ Orbitrap Discovery mass spectrometer (Thermo Fisher Scientific). This hybrid system meets the LTQ XL linear ion trap mass spectrometer and an Orbitrap mass analyzer. The experiments were performed via direct infusion of sample (flow: 10 µL/min) in the positive-ion mode using electrospray ionization. Elemental composition calculations for comparison were executed using the specific tool included in the Qual Browser module of Xcalibur (Thermo Fisher Scientific, release 2.0.7) software. Elemental analyses CHN were performed on a model 2400 Series II - Perkin Elmer elemental analysis instrument. Optical rotations were measured with a JASCO P-2000 Polarimeter in CH₂Cl₂ solutions as the solvents with percent concentrations. All enantiomeric excesses were obtained using Shimadzu GC-2010 chromatograph and Agilent 6820 GC System using the following column: HYDRODEX[®]-β-3P (25 m x 0.25 mm ID) - Macherey-Nagel.

General procedure for the directly synthesis of bis-1,3-dioxolanymethyl chalcogenides 3:

To a mixture of respective chalcogen **2** (0.5 mmol) in PEG-400 (4.0 mL) under N₂ atmosphere, was added NaBH₄ (0.042g, 1.1 mmol) and the mixture was heated slowly to 50 °C, stirring for 40 min. The respective (*R*)- or (*S*)-tosyl dioxolane **1** were then added at 50 °C. The reaction progress was monitored by TLC. After the time indicated in Table 2, the solution was cooled to room temperature and water (10.0 mL) and ethyl acetate (15.0 mL) were added. The organic phase was washed with water (2x 10.0 mL), separated, dried over MgSO₄, and the solvent was evaporated under reduced pressure. The product was isolated by column

chromatography using hexane/ethyl acetate (97:3) as eluent.

General procedure for the directly synthesis of bis-1,3-dioxolanymethyl dichalcogenides 4:

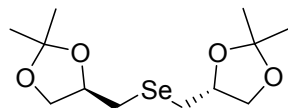
In a two-necked round-bottomed flask containing a suspension of the elemental chalcogenium **2** (2.0 mmol) in THF (2.0 mL) under N₂ atmosphere, lithium triethyl borohydride (2.0 mmol; 2.0 mL of a sol. 1M in THF) was added. The reaction mixture was stirred for 30 min at room temperature. The respective (*R*)- or (*S*)-tosyl dioxolane **1** (0.85 mmol) were then added as a solution in THF (7.5 mL) at reaction mixture. The reaction progress was followed by TLC. After, the complete consumption of starting material saturated aqueous NH₄Cl (25.0 mL) was added to the reaction mixture. Then, the solution was diluted with dichloromethane (30.0 mL) and washed with water (3 x 10.0 mL). The organic phase was separated, dried over MgSO₄, and the solvent was evaporated under reduced pressure. The product was isolated by column chromatography using hexane/ethyl acetate (97:3) as eluent.

General procedure for synthesis of (*R*)-3-(2,3-dihydroxypropylselanyl)propanenitrile 6b:

(i) To a mixture of diselenide (*R*)-**4a** (0.390g, 1.0 mmol) in PEG-400 (4.0 mL) under N₂ atmosphere, NaBH₄ (0.046g, 1.2 mmol) was added at room temperature and the mixture stirred during 30 min. After, it was added acrylonitrile (0.053g, 1.0 mmol) and the mixture was stirred for additional 2 h at 50 °C. Then, the solution was cooled to room temperature, diluted with ethyl acetate (10.0 mL) and washed with water (3x 10.0 mL). The organic phase was separated, dried over MgSO₄, and the solvent was evaporated under reduced pressure. The product was isolated by column chromatography using hexane/ethyl acetate (80:20) as the eluent.

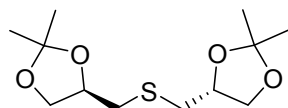
(ii) **Deprotection step:** to a mixture of (*R*)-**6a** (0.125g, 0.5 mmol) in water (2.0 mL), ZnBr₂ (0.111g, 0.5 mmol) was added and the resulting solution was stirred for 24 h under reflux. Then, the solution was cooled to room temperature, received in brine (15.0 mL) and the product was extracted with ethyl acetate (3x 10.0 mL). The organic phase was separated, dried over MgSO₄, and the solvent was evaporated under reduced pressure. The product was isolated by column chromatography using hexane/ethyl acetate (50:50) as eluent.

Spectral data of the compounds:



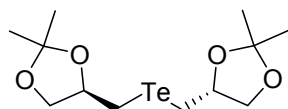
(*R,R*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)selenide (3a): Yield:

0.130 g (84%, 99.9 ee); Yellowish oil; $[\alpha]_{\text{D}}^{20}$: + 47.47 (c 0.92, CH_2Cl_2). ^1H NMR (CDCl_3 , 400 MHz): δ 4.28-4.34 (m, 2H); 4.11 (dd, J = 8.3 and 6.0 Hz, 2H); 3.69 (dd, J = 8.3 and 6.4 Hz, 2H); 2.87 (dd, J = 12.5 and 5.6 Hz, 2H); 2.70 (dd, J = 12.5 and 7.2 Hz, 2H); 1.42 (d, J = 0.6 Hz, 6H); 1.35 (d, J = 0.6 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3); δ (ppm): 109.5, 75.9, 69.3, 27.1, 26.9, 25.5. MS: m/z (rel. int.) 310 (8.8), 101 (73.6), 43 (100.0). HRMS (ESI): m/z calcd for $\text{C}_{12}\text{H}_{22}\text{O}_4\text{Se}$ $[\text{M}+\text{Na}]^+$: 333.0581; found: 333.0593.



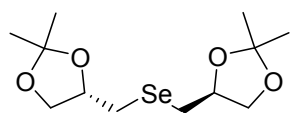
(*R,R*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)sulfide (3b): Yield:

0.098 g (75%, 99.9 ee); Colorless oil; $[\alpha]_{\text{D}}^{20}$: + 35.13 (c 0.44, CH_2Cl_2). ^1H NMR (CDCl_3 , 300 MHz): δ 4.23-4.31 (m, 2H); 4.10 (dd, J = 8.3 and 6.0 Hz, 2H); 3.72 (dd, J = 8.3 and 6.3 Hz, 2H); 2.84 (dd, J = 13.4 and 5.7 Hz, 2H); 2.67 (dd, J = 13.4 and 6.9 Hz, 2H); 1.43 (s, 6H); 1.36 (s, 6H). ^{13}C NMR (100 MHz, CDCl_3); δ (ppm): 109.5, 75.5, 68.7, 35.7, 26.8, 25.4. MS: m/z (rel. int.) 262 (3.7), 101 (100.0), 43 (81.4). HRMS (ESI): m/z calcd for $\text{C}_{12}\text{H}_{22}\text{O}_4\text{S}$ $[\text{M}+\text{H}]^+$: 263.1317; found: 263.1312.

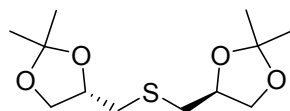


(*R,R*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)telluride (3c): Yield:

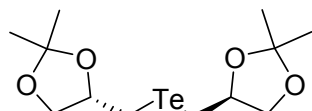
0.108 g (60%, 99.9 ee); Yellowish oil; $[\alpha]_{\text{D}}^{20}$: + 28.95 (c 0.70, CH_2Cl_2). ^1H NMR (CDCl_3 , 400 MHz): δ 4.30-4.36 (m, 2H), 4.14 (dd, J = 8.2 and 5.9 Hz, 2H), 3.62 (dd, J = 8.2 and 6.7 Hz, 2H), 2.95 (dd, J = 12.1 and 5.6 Hz, 2H), 2.78 (dd, J = 12.1 and 7.3 Hz, 2H), 1.43 (d, J = 0.6 Hz, 6H), 1.34 (d, J = 0.7 Hz, 6H). ^{13}C NMR (100 MHz, CDCl_3); δ (ppm): 109.7, 76.9, 70.5, 27.0, 25.7, 6.6. MS: m/z (rel. int.) 360 (18.3), 101 (23.3), 43 (100.0). HRMS (ESI): m/z calcd for $\text{C}_{12}\text{H}_{22}\text{O}_4\text{Te}$ $[\text{M}+\text{H}]^+$: 361.0659; found: 361.0653.



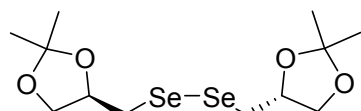
(*S,S*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)selenide (3a): Yield: 0.109 g (70%, 99.9 ee); Yellowish oil; $[\alpha]_{\text{D}}^{20}$: - 48.60 (*c* 1.08, CH₂Cl₂). The characterization data from NMR, MS and HRMS spectra were identical in all aspects with those of (*R,R*)-(+)-**3a** enantiomer.



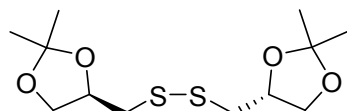
(*S,S*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)sulfide (3b): Yield: 0.085 g (65%, 99.9 ee); Colorless oil; $[\alpha]_{\text{D}}^{20}$: - 48.58 (*c* 0.45, CH₂Cl₂). The characterization data from NMR, MS and HRMS spectra were identical in all aspects with those of (*R,R*)-(+)-**3b** enantiomer.



(*S,S*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)telluride (3c): Yield: 0.099 g (55%, 99.9 ee); Yellowish oil; $[\alpha]_{\text{D}}^{20}$: - 32.25 (*c* 0.56, CH₂Cl₂). The characterization data from NMR, MS and HRMS spectra were identical in all aspects with those of (*R,R*)-(+)-**3c** enantiomer.

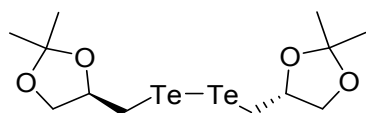


(*R,R*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)diselenide (4a): Yield: 0.133 g (80%, 99.9 ee); Orangish oil; $[\alpha]_{\text{D}}^{20}$: + 34.01 (*c* 0.48, CH₂Cl₂). ¹H NMR (CDCl₃, 400 MHz): δ 4.34-4.41 (m, 2H); 4.14 (dd, *J* = 8.4 and 6.0 Hz, 2H); 3.74 (dd, *J* = 8.4 and 6.1 Hz, 2H); 3.20 (dd, *J* = 12.4 and 5.8 Hz, 2H); 3.07 (dd, *J* = 12.4 and 7.1 Hz, 2H); 1.44 (s, 6H); 1.36 (s, 6H). ¹³C NMR (100 MHz, CDCl₃); δ (ppm): 109.6, 75.9, 69.0, 33.0, 27.0, 25.5. MS: *m/z* (rel. int.) 390 (10.5), 115 (76.5), 101 (21.2), 57 (97.5), 43 (100.0). HRMS (ESI): *m/z* calcd for C₁₂H₂₂O₄Se₂ [M+H]⁺: 390.9927; found: 390.9921.



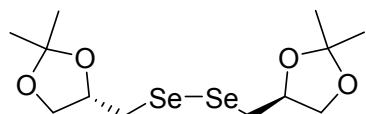
(*R,R*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)disulfide (4b):

Yield: 0.052 g (42%, 99.9 ee). Colorless oil; $[\alpha]_{\text{D}}^{20}$: + 24.66 (c 0.52, CH_2Cl_2). ^1H NMR (CDCl_3 , 300 MHz): δ 4.33-4.41 (m, 2H); 4.14 (dd, J = 8.4 and 6.0 Hz, 2H); 3.76 (dd, J = 8.4 and 6.0 Hz, 2H); 2.98 (dd, J = 13.5 and 5.7 Hz, 2H); 2.82 (dd, J = 13.5 and 7.0 Hz, 2H); 1.43 (s, 6H); 1.36 (s, 6H). ^{13}C NMR (75 MHz, CDCl_3); δ (ppm): 109.6, 74.7, 68.6, 42.1, 26.9, 25.5. MS: m/z (rel. int.) 294 (11.8), 101 (91.7), 43 (100.0). HRMS (ESI): m/z calcd for $\text{C}_{12}\text{H}_{22}\text{O}_4\text{S}_2$ $[\text{M}+\text{H}]^+$: 295.1032; found: 294.9932.



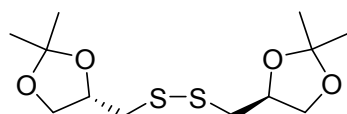
(*R,R*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)ditelluride (4c):

Yield: 0.098 g (47%, 99.9 ee); Reddish oil; $[\alpha]_{\text{D}}^{20}$: + 30.13 (c 0.54, CH_2Cl_2). ^1H NMR (CDCl_3 , 400 MHz): δ 4.25-4.31 (m, 2H); 4.14 (dd, J = 8.2 and 6.0 Hz, 2H); 3.67 (dd, J = 8.2 and 6.5 Hz, 2H); 3.45 (dd, J = 11.7 and 5.8 Hz, 2H); 3.36 (dd, J = 11.7 and 7.1 Hz, 2H); 1.44 (s, 6H); 1.35 (s, 6H). ^{13}C NMR (100 MHz, CDCl_3); δ (ppm): 109.7, 77.6, 69.9, 27.1, 25.7, 8.7. MS: m/z (rel. int.) 488 ($\text{M}^+ - 2$, 1.0), 115 (32.1), 57 (64.8), 43 (100.0). HRMS (ESI): m/z calcd for $\text{C}_{12}\text{H}_{22}\text{O}_4\text{Te}_2$ $[\text{M}+\text{Na}]^+$: 508.9511; found: 508.9533.



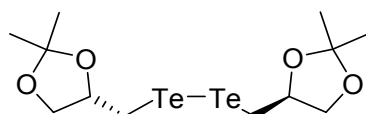
(*S,S*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)diselenide (4a):

Yield: 0.124 g (75%, 99.9 ee); Orangish oil; $[\alpha]_{\text{D}}^{20}$: - 39.11 (c 0.53, CH_2Cl_2). The characterization data from NMR, MS and HRMS spectra were identical in all aspects with those of (*R,R*)-(+)-**4a** enantiomer.



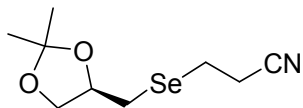
(*S,S*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)disulfide (4b):

Yield: 0.037 g (30%, 99.9 ee); Colorless oil; $[\alpha]_{\text{D}}^{20}$: - 24.69 (c 0.27, CH_2Cl_2). The characterization data from NMR, MS and HRMS spectra were identical in all aspects with those of (*R,R*)-(+)-**4b** enantiomer.



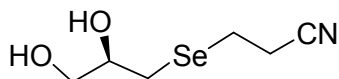
(*S,S*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)ditelluride (4c):

Yield: 0.102 g (49%, 99.9 ee). Reddish oil; $[\alpha]_{\text{D}}^{20}$: - 33.74 (*c* 0.39, CH₂Cl₂). The characterization data from NMR, MS and HRMS spectra were identical in all aspects with those of (*R,R*)-(+)-**4c** enantiomer.



(*R*)-3-(2,2-dimethyl-1,3-dioxolanylmethylselanyl)propanenitrile 6a:

Yield: 0.219 g (88%, 99.9 ee). Colorless oil; $[\alpha]_{\text{D}}^{20}$: + 17.10 (*c* 0.57, CH₂Cl₂). ¹H NMR (CDCl₃, 400 MHz): δ 4.31-4.37 (m, 1H); 4.11 (dd, *J* = 8.3 and 6.1 Hz, 1H); 3.68 (dd, *J* = 8.3 and 6.6 Hz, 1H); 2.93-2.78 (m, 6H); 1.44 (s, 3H); 1.36 (d, *J* = 0.5 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃); δ (ppm): 118.7, 109.6, 76.0, 69.2, 27.2, 26.8, 25.4, 19.6, 18.2. MS: *m/z* (rel. int.) 249 (2.1), 101 (31.5), 43 (100.0). Anal. Calcd for C₉H₁₅NO₂Se: C 43.56, H 6.09, N 5.64. Found: C 43.42, H 5.98, N 5.76.



(*R*)-3-(2,3-dihydroxypropylselanyl)propanenitrile 6b:

Yield: 0.041 g (40%, 99.9 ee). Colorless oil; $[\alpha]_{\text{D}}^{20}$: - 22.64 (*c* 0.66, CH₂Cl₂). ¹H NMR (DMSO-*d*₆, 300 MHz): δ 3.98 (d, *J* = 5.0 Hz, 1H); 3.74 (t, *J* = 5.6 Hz, 1H); 2.67-2.77 (m, 1H); 2.37-2.48 (m, 2H); 1.98-2.02 (m, 2H); 1.84-1.91 (m, 3 H); 1.72 (dd, *J* = 12.5 and 6.8 Hz, 1H). ¹³C NMR (75 MHz, DMSO-*d*₆); δ (ppm): 120.2, 71.6, 64.9, 28.3, 18.9, 18.0. MS: *m/z* (rel. int.) 209 (19.3), 133 (30.8), 54 (100.0). HRMS (ESI): *m/z* calcd for C₆H₁₁NO₂Se [M+Na]⁺: 231.9853; found: 231.9833.

SELECTED SPECTRA

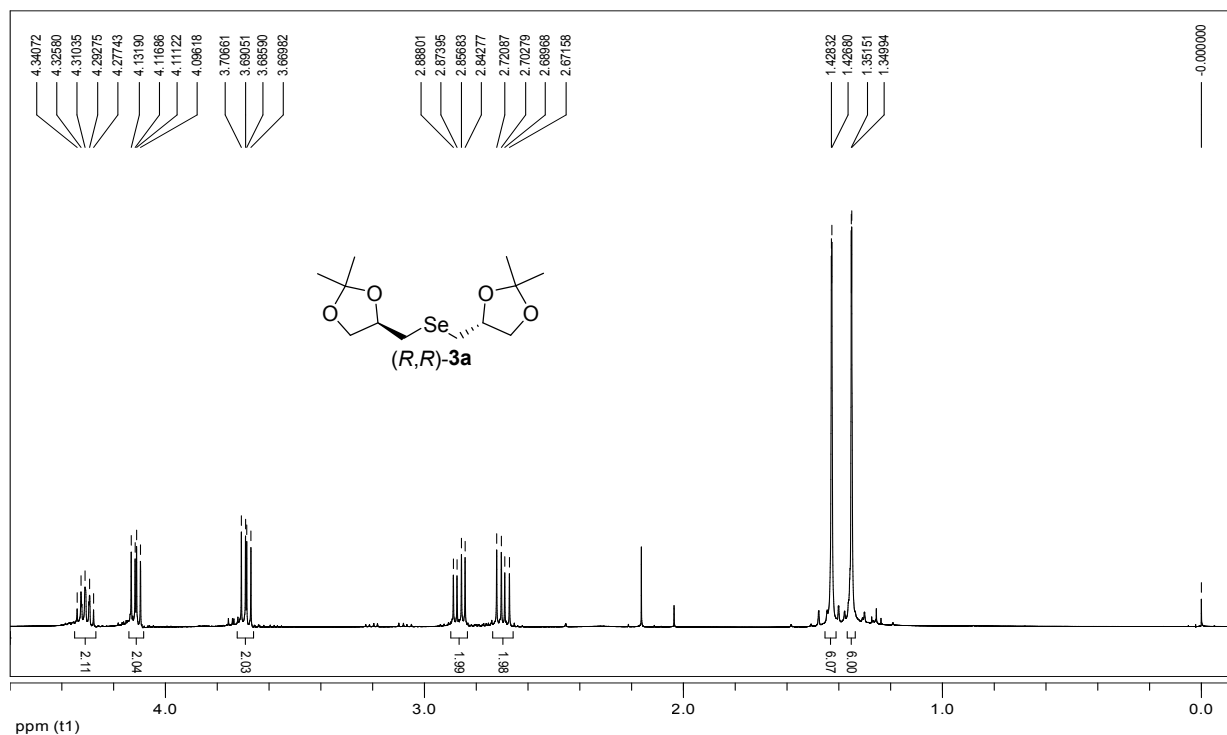


Figure 1. ^1H NMR (400 MHz, CDCl_3) spectrum of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)selenide (**3a**).

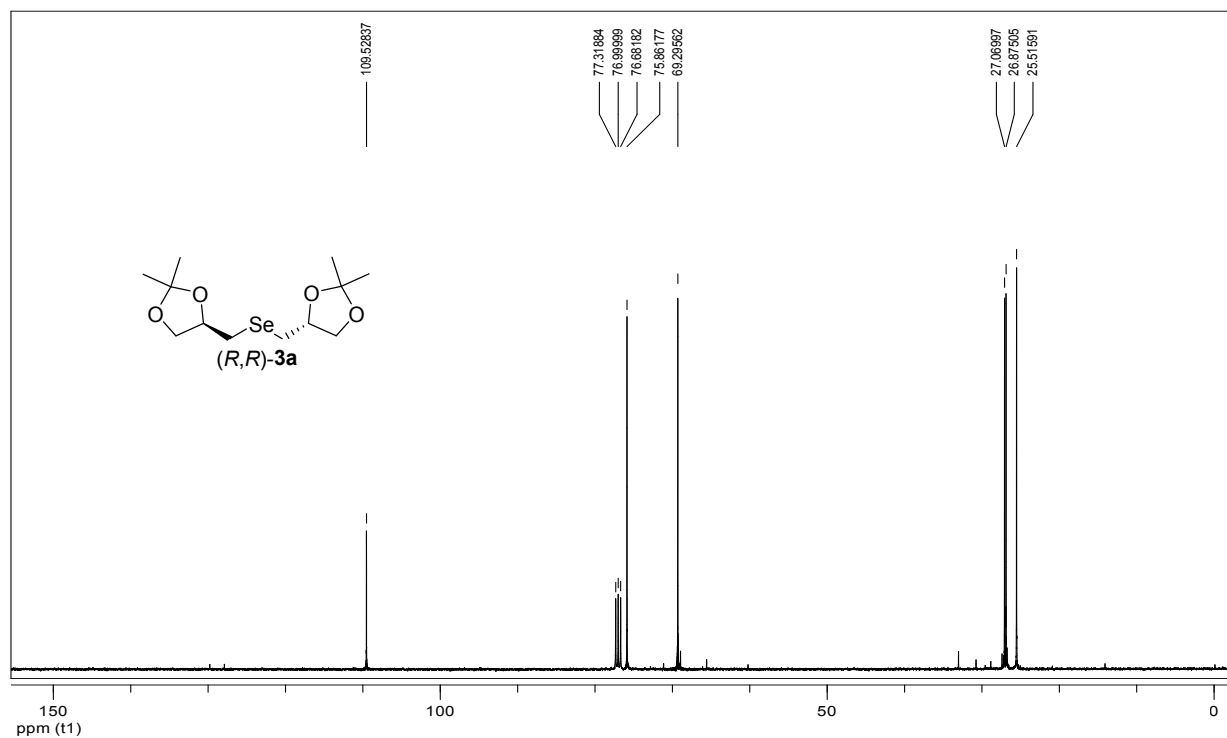


Figure 2. ^{13}C NMR (100 MHz, CDCl_3) spectrum of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)selenide (**3a**).

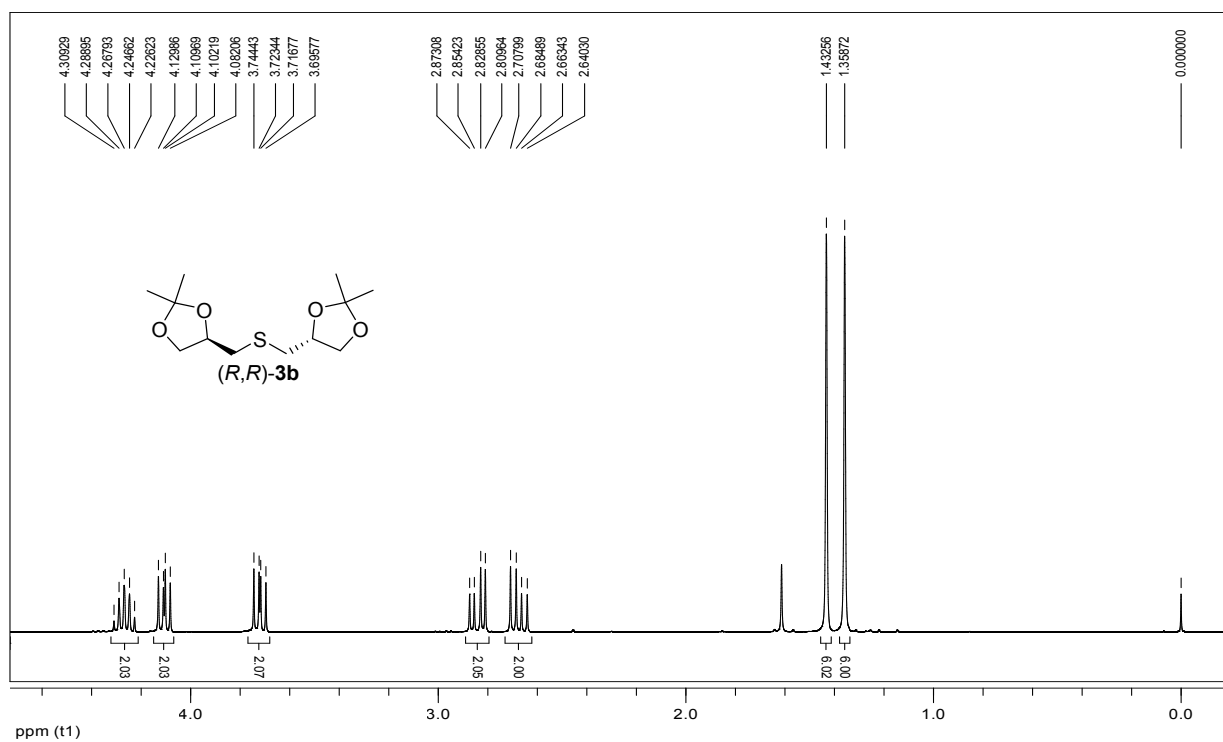


Figure 3. ¹H NMR (300 MHz, CDCl₃) spectrum of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)sulfide (**3b**).

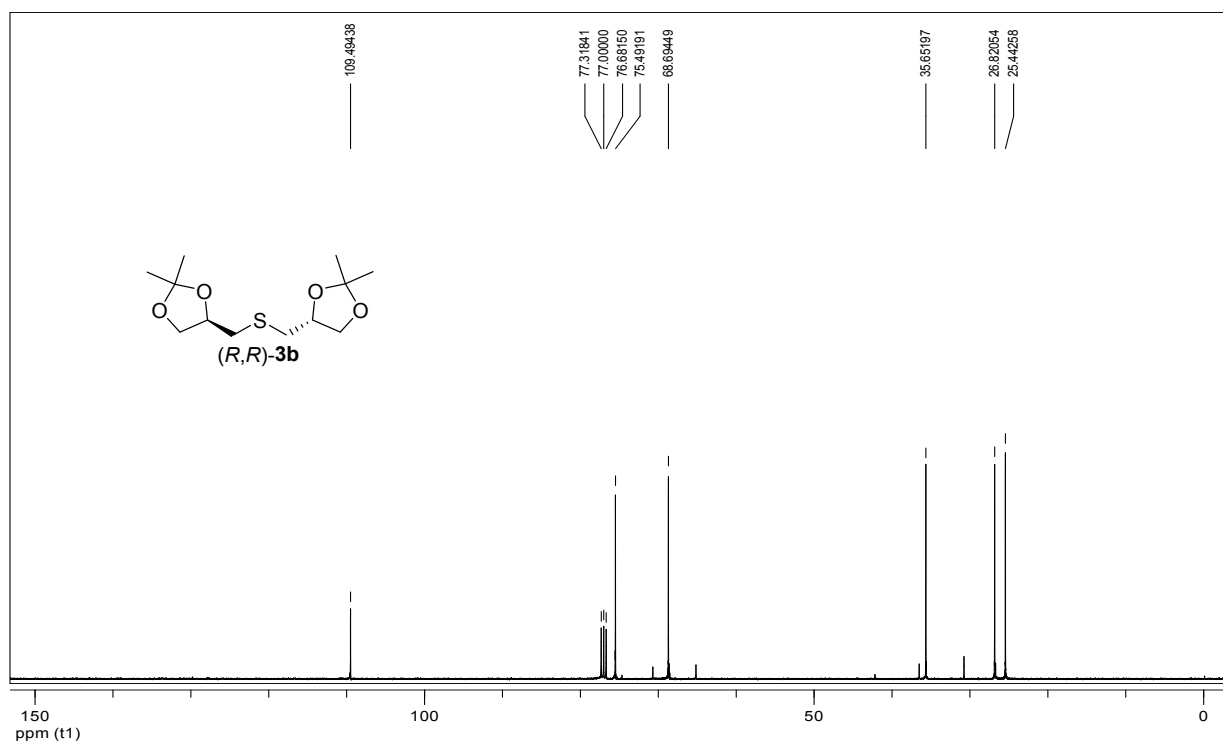


Figure 4. ¹³C NMR (100 MHz, CDCl₃) spectrum of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)sulfide (**3b**).

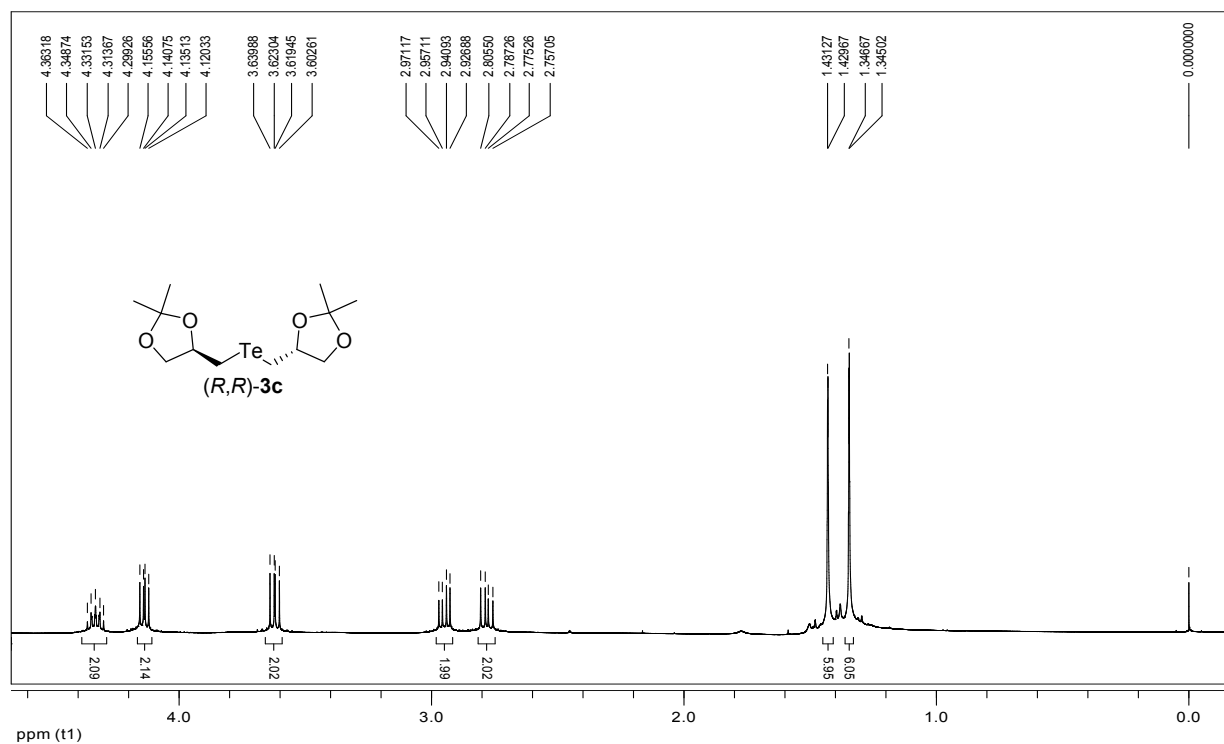


Figure 5. ¹H NMR (400 MHz, CDCl₃) spectrum of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)telluride (**3c**).

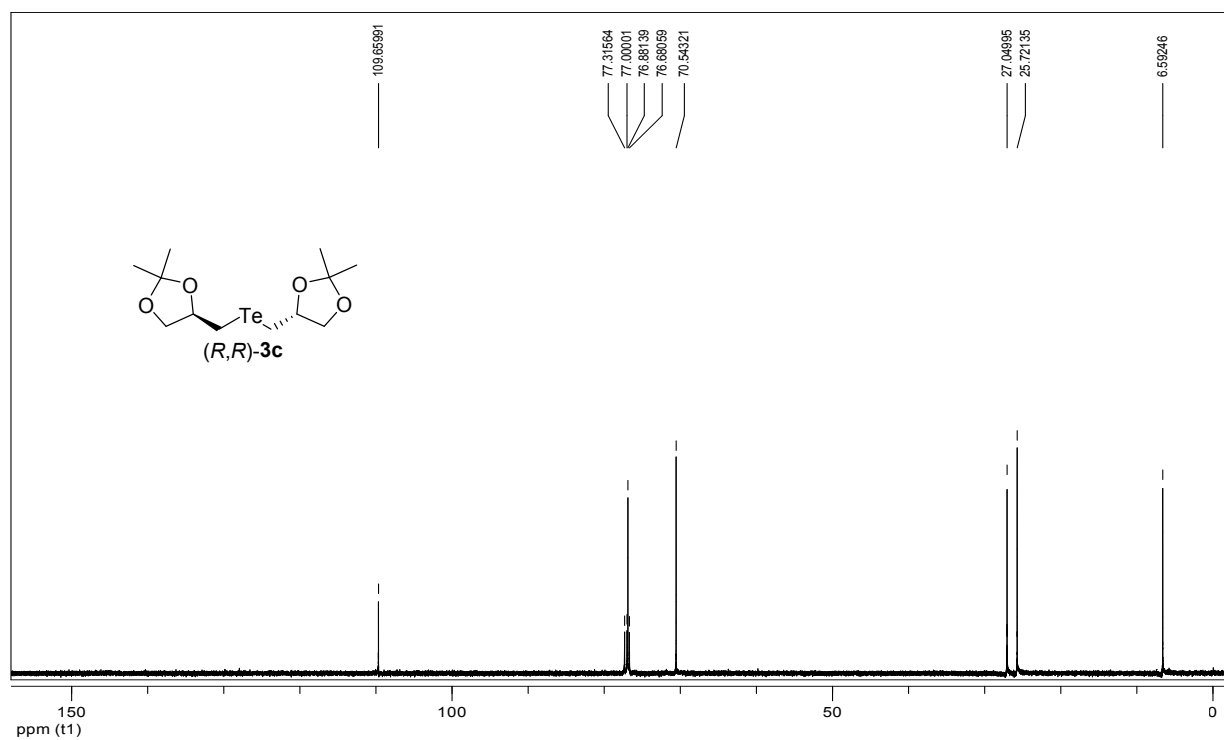


Figure 6. ¹³C NMR (100 MHz, CDCl₃) spectrum of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)telluride (**3c**).

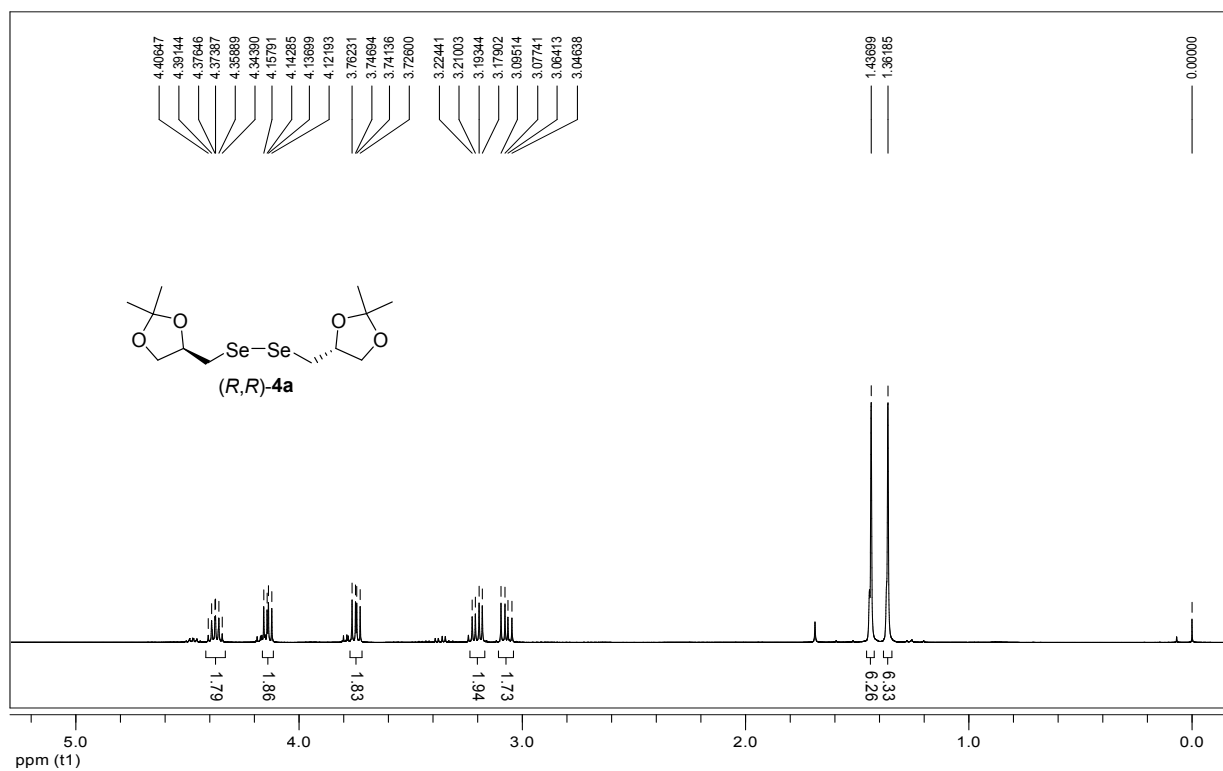


Figure 7. ¹H NMR (400 MHz, CDCl₃) spectrum of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)diselenide (**4a**).

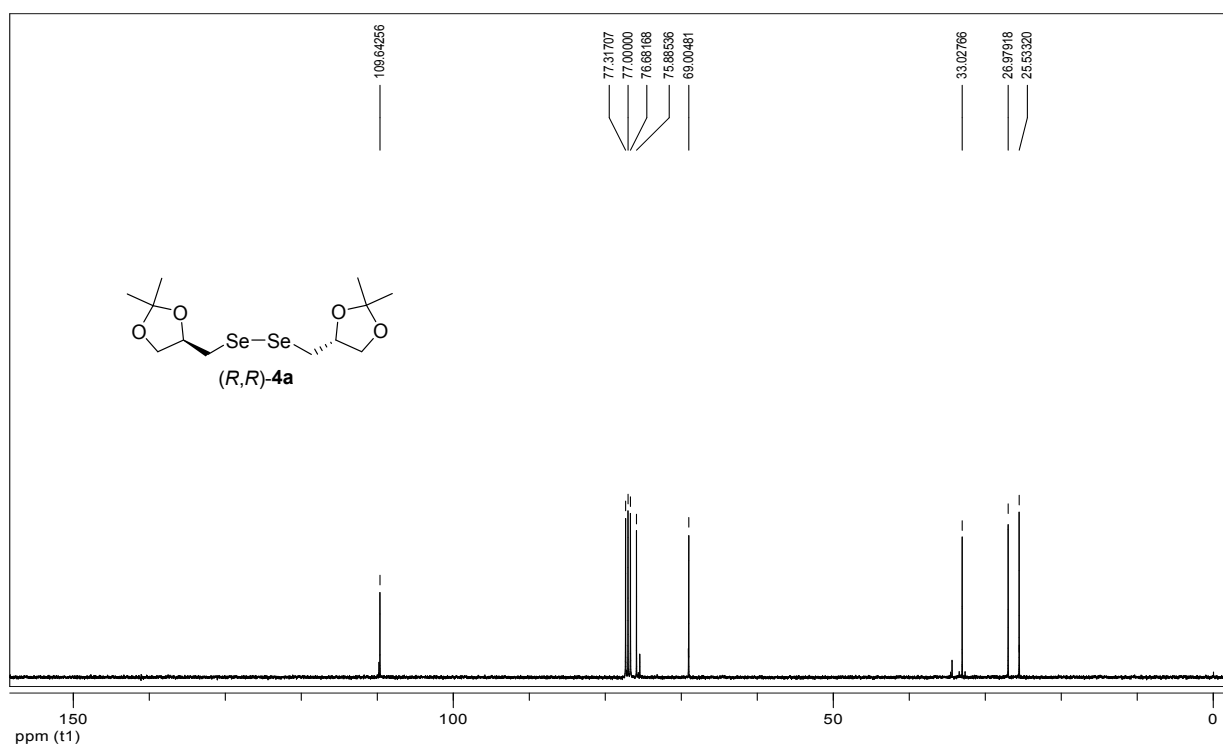


Figure 8. ¹³C NMR (100 MHz, CDCl₃) spectrum of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)diselenide (**4a**).

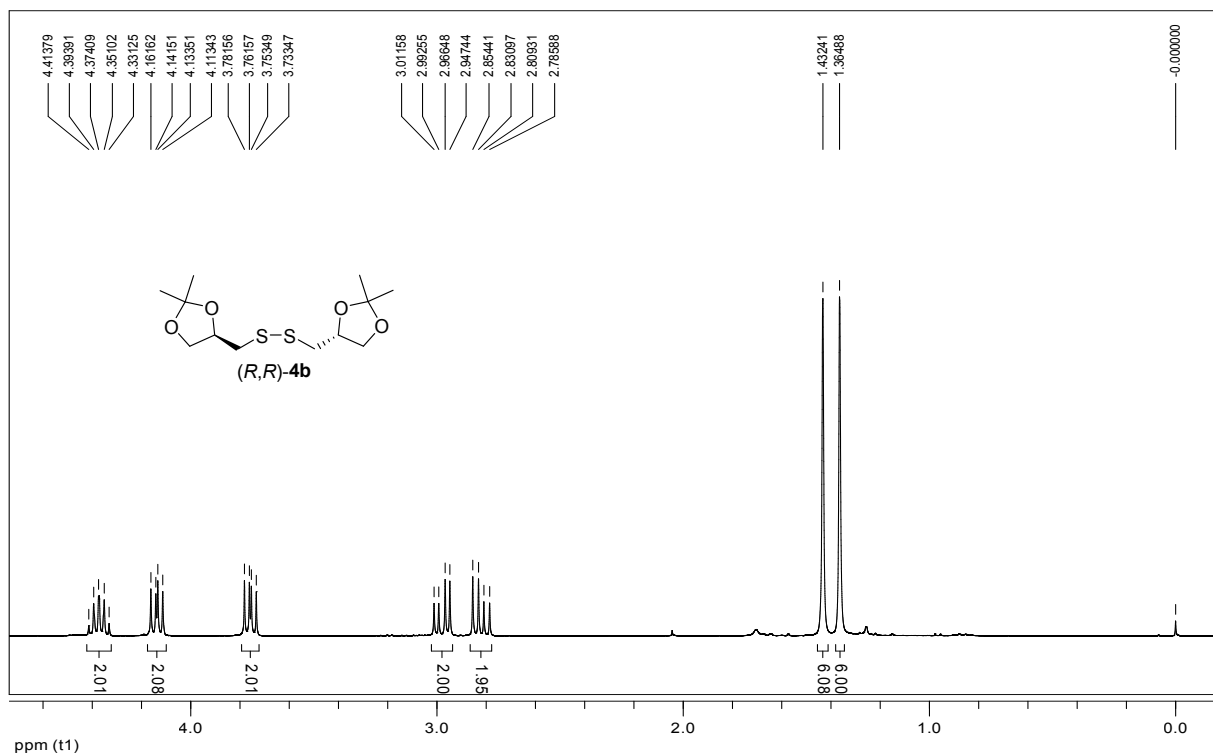


Figure 9. ¹H NMR (300 MHz, CDCl₃) spectrum of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)disulfide (**4b**).

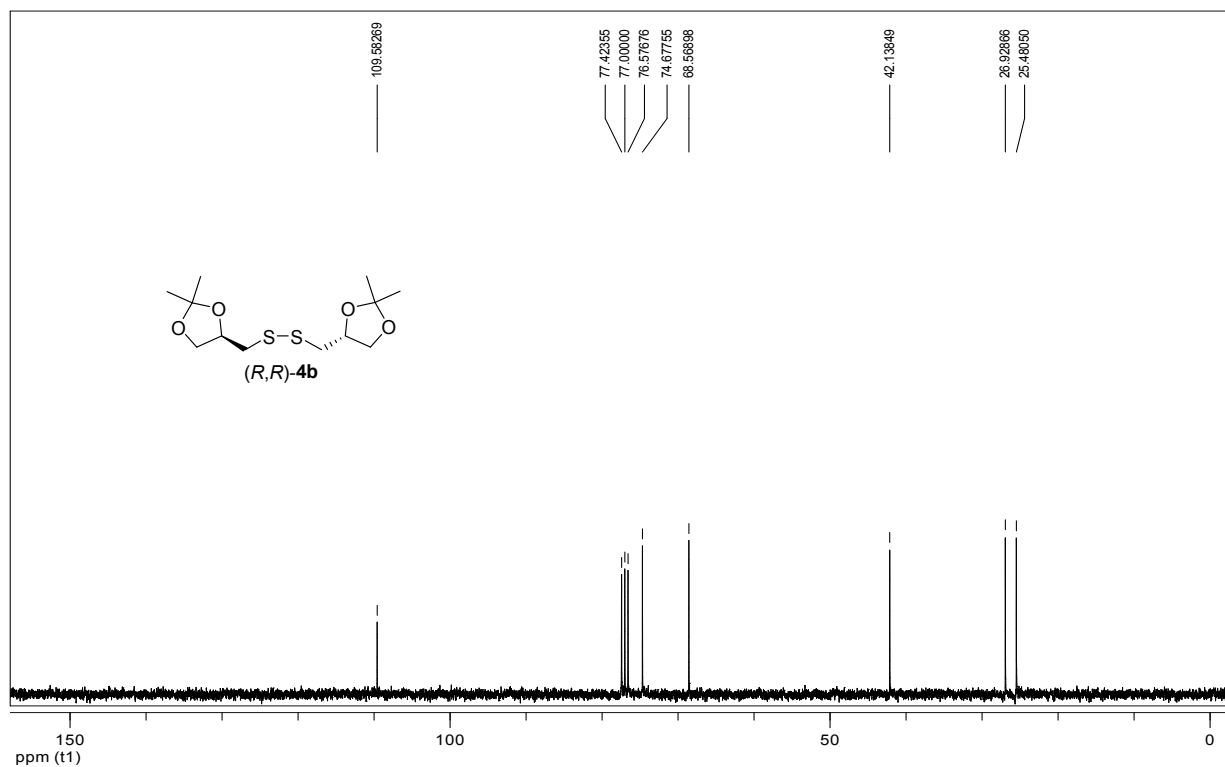


Figure 10. ¹³C NMR (75 MHz, CDCl₃) spectrum of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)disulfide (**4b**).

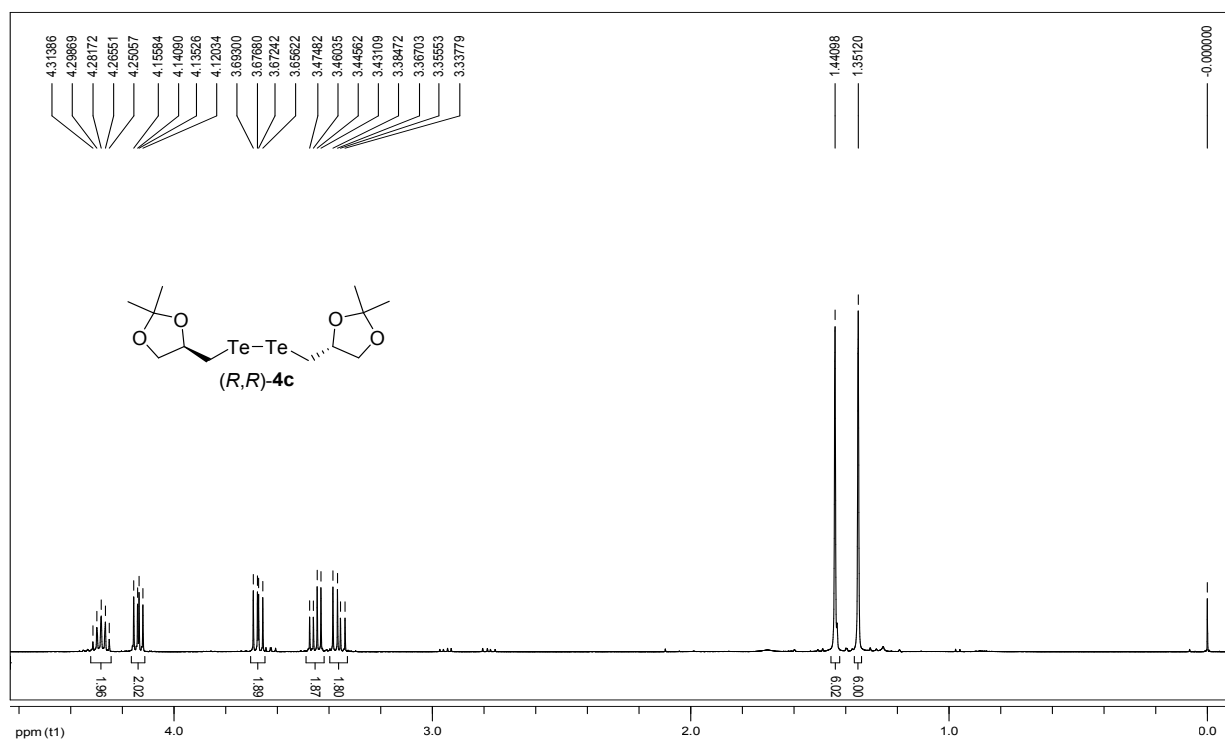


Figure 11. ¹H NMR (400 MHz, CDCl₃) spectrum of (*R,R*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)ditelluride (**4c**).

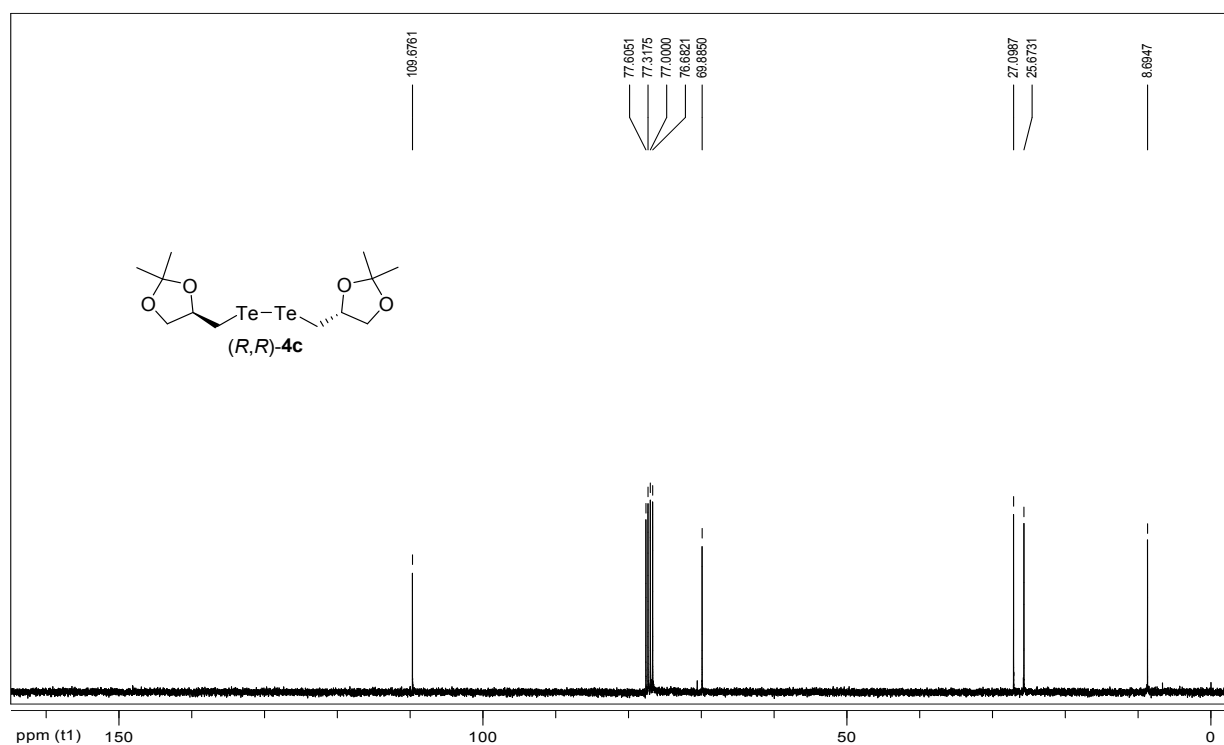


Figure 12. ¹³C NMR (100 MHz, CDCl₃) spectrum of (*R,R*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)ditelluride (**4c**).

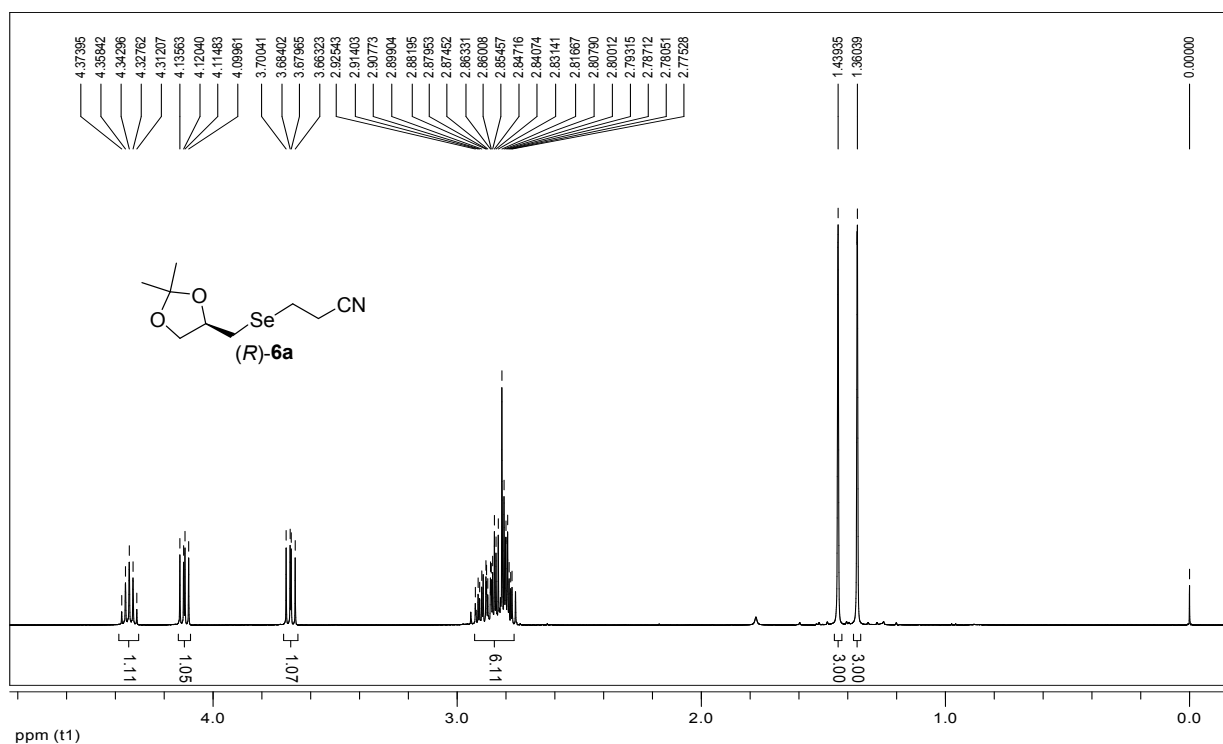


Figure 13. ¹H NMR (400 MHz, CDCl₃) spectrum of (*R*)-3-(2,2-dimethyl-1,3-dioxolanylmethylselanyl)propanenitrile (**6a**).

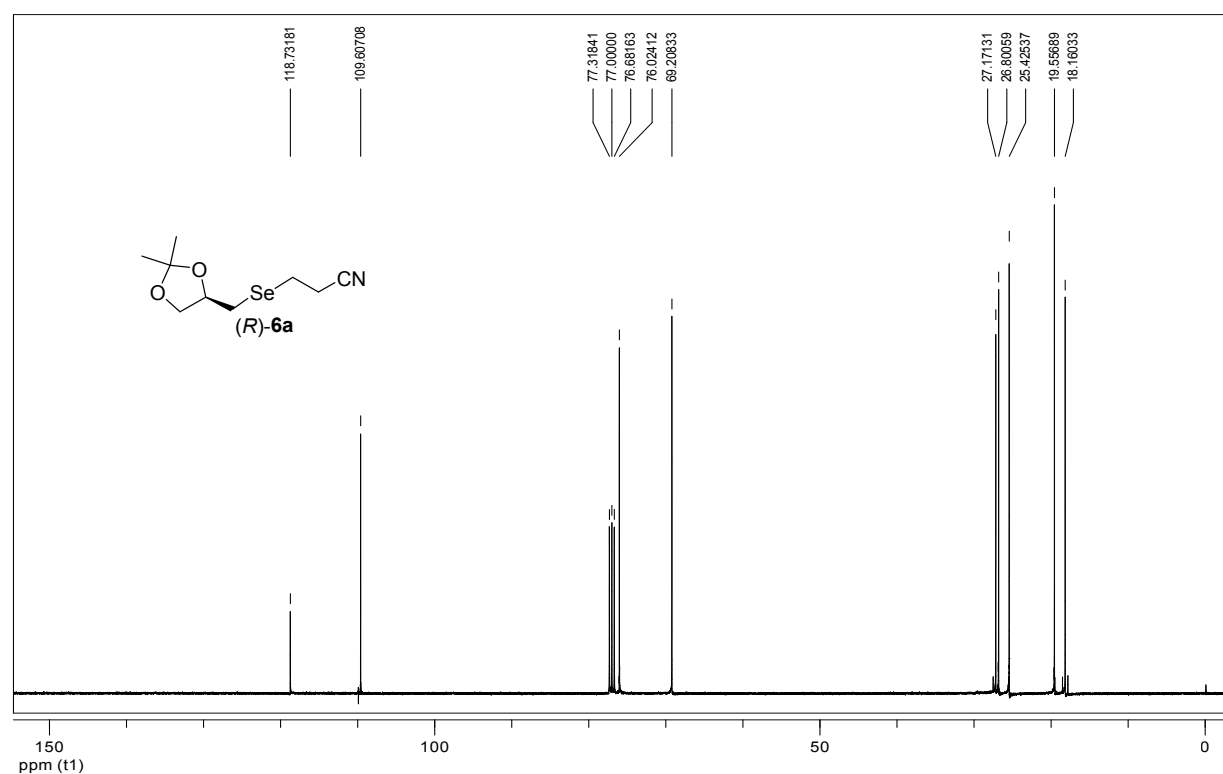


Figure 14. ¹³C NMR (100 MHz, CDCl₃) spectrum of (*R*)-3-(2,2-dimethyl-1,3-dioxolanylmethylselanyl)propanenitrile (**6a**).

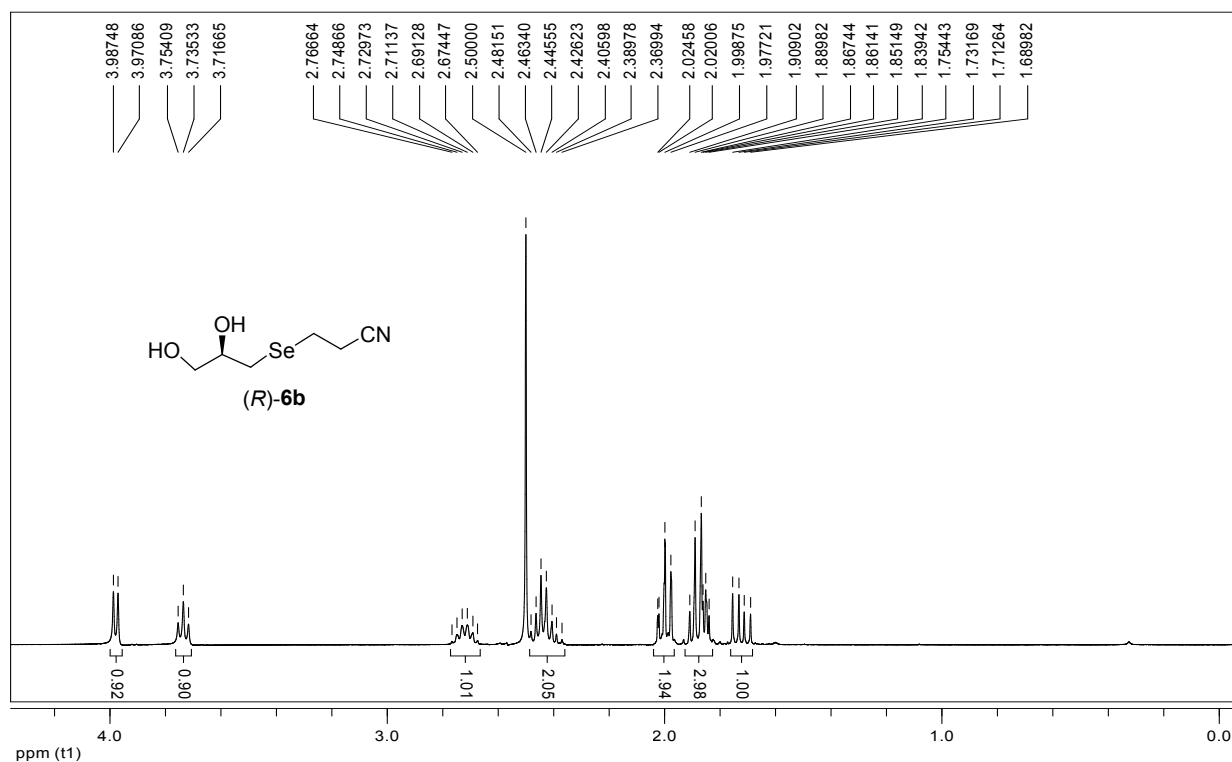


Figure 15. ¹H NMR (300 MHz, DMSO-*d*₆) spectrum of (R)-3-(2,3-dihydroxypropylselanyl)propanenitrile (**6b**).

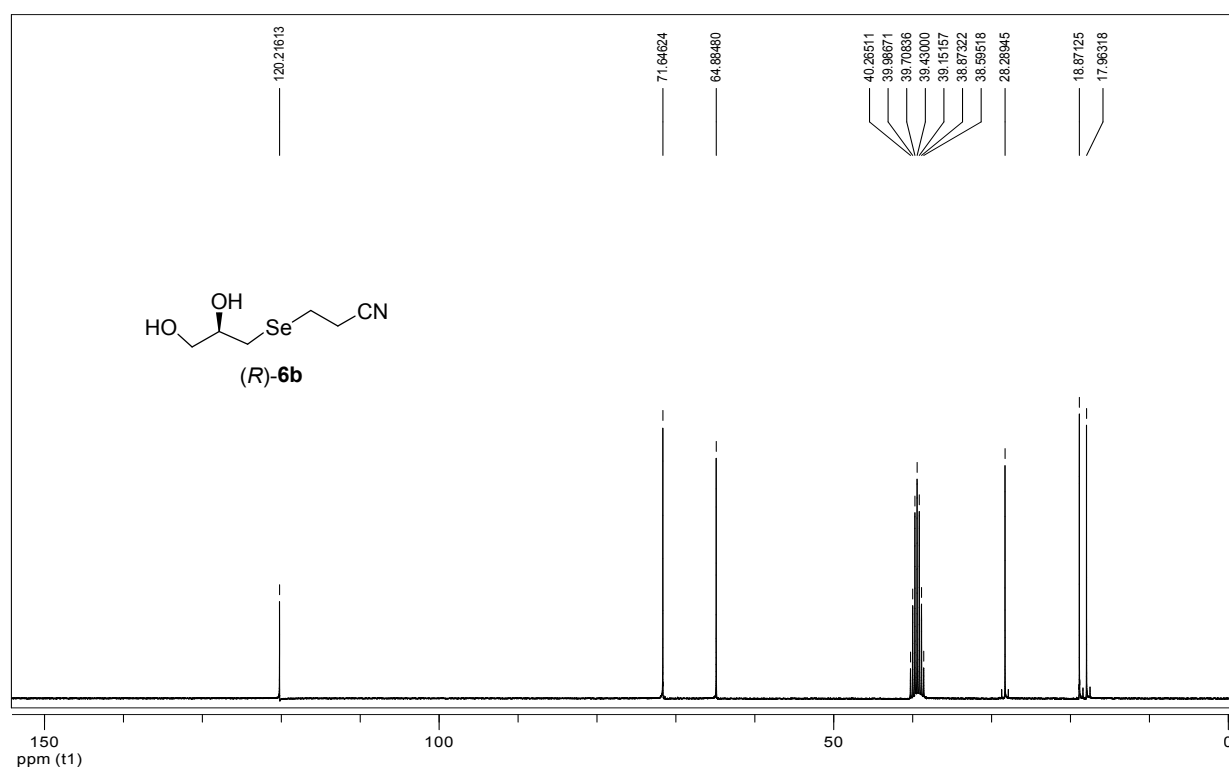


Figure 16. ¹³C NMR (75 MHz, DMSO-*d*₆) spectrum of (R)-3-(2,3-dihydroxypropylselanyl)propanenitrile (**6b**).

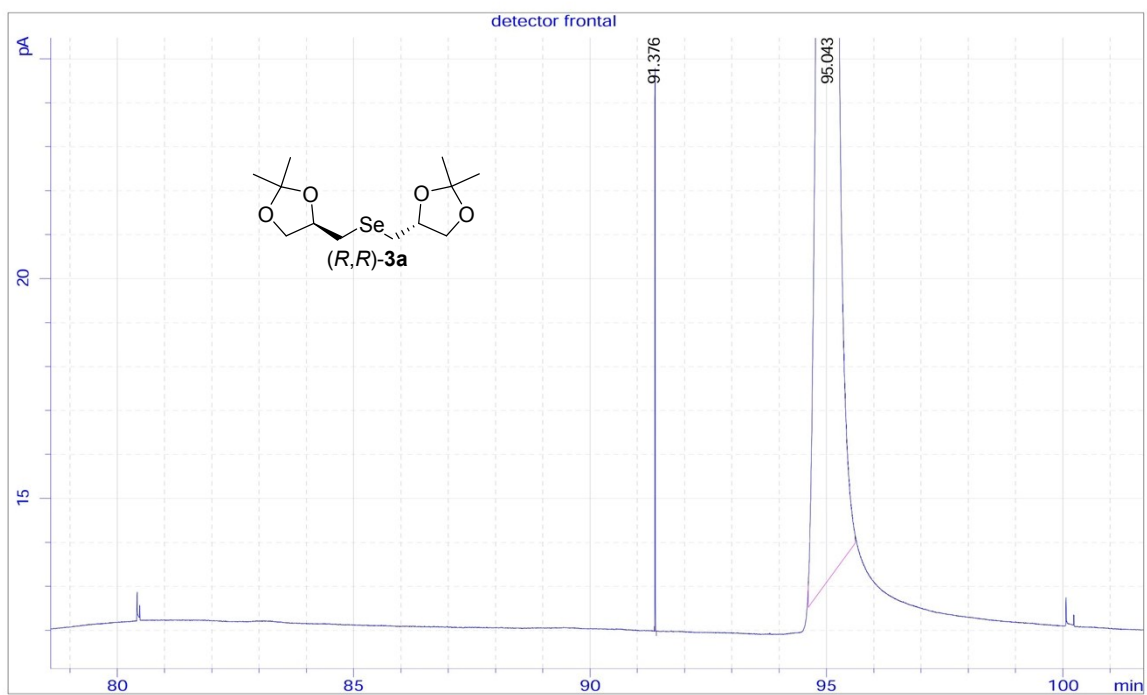


Figure 17. Chiral GC analysis of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)selenide (**3a**).

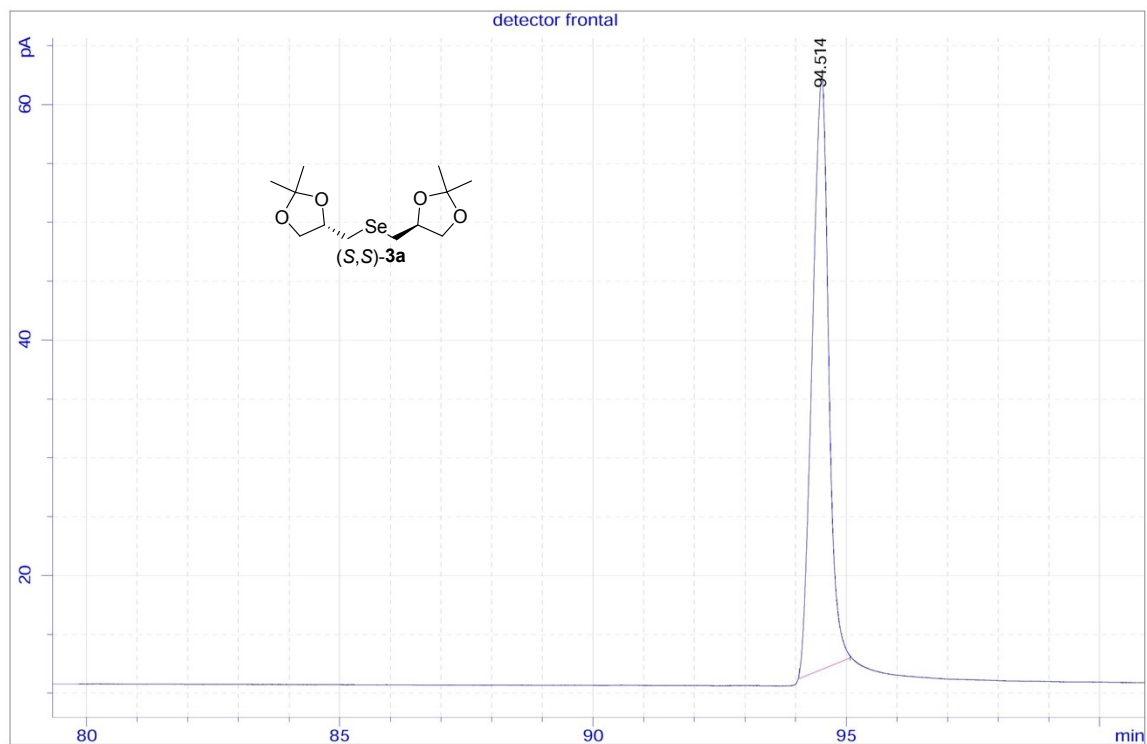
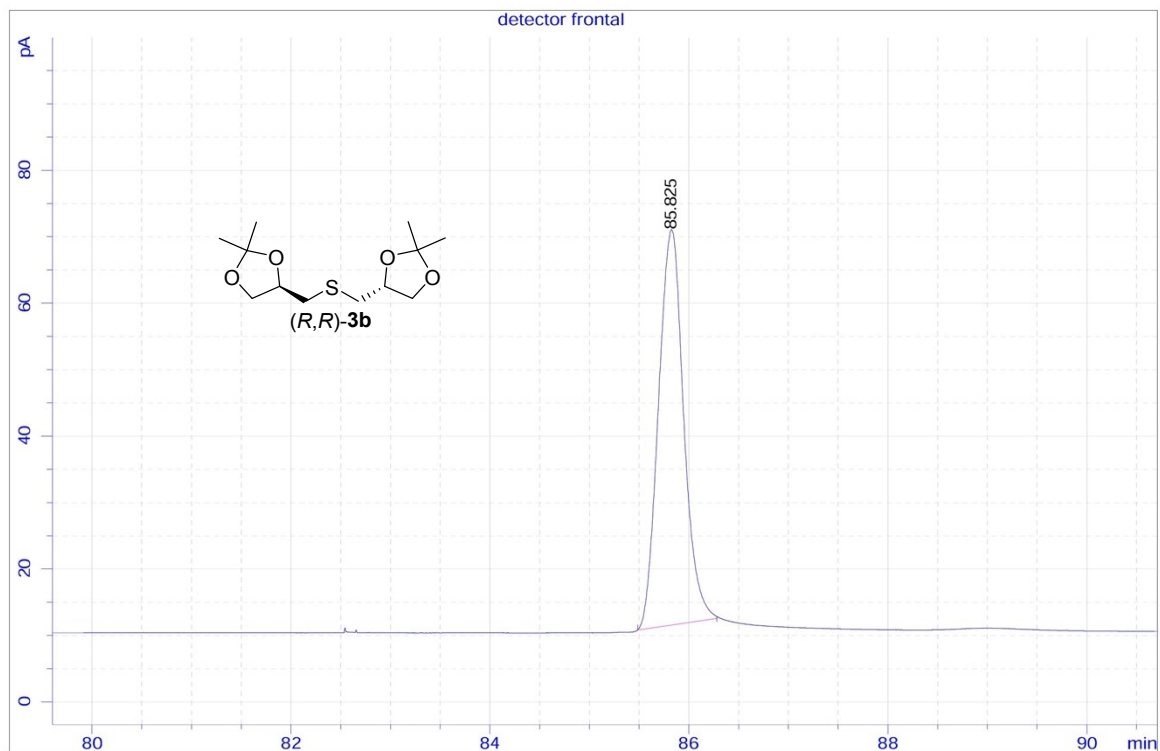
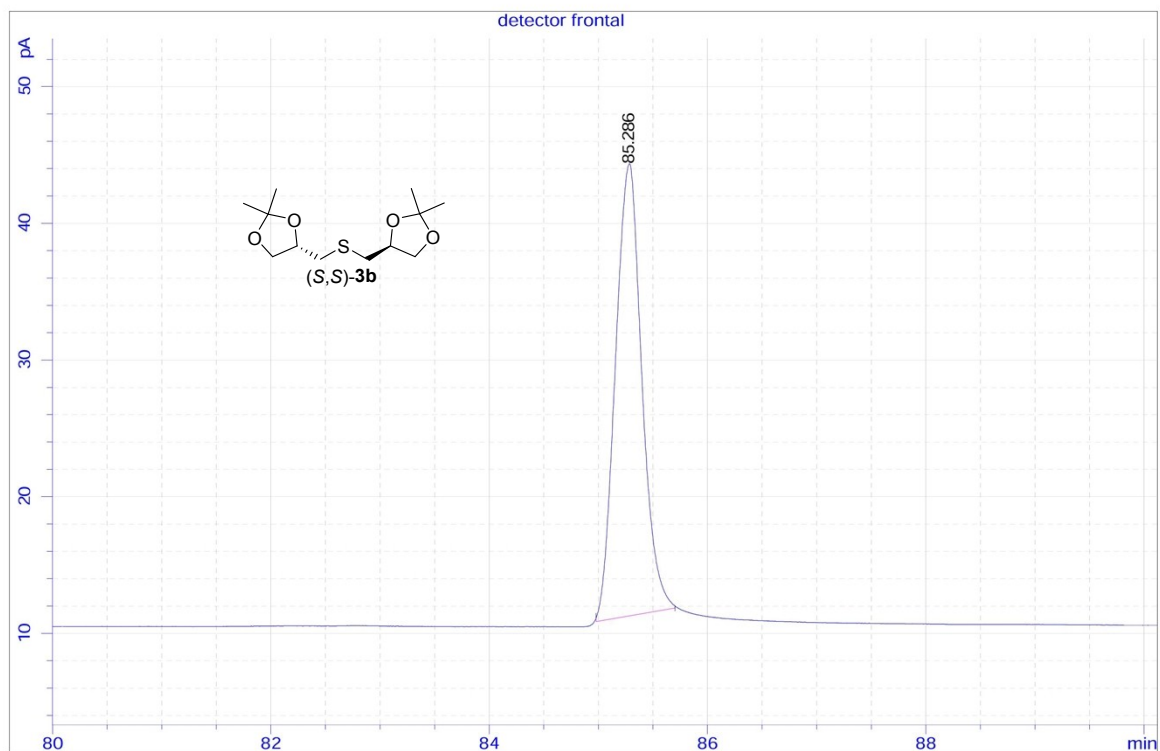


Figure 18. Chiral GC analysis of *(S,S)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)selenide (**3a**).



Peak#	R.Time [Min]	Area	Area %
1	85,825	1044,92111	100,00000

Figure 19. Chiral GC analysis of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)sulfide (**3b**).



Peak#	R.Time [Min]	Area	Area %
1	85,286	555,44867	100,00000

Figure 20. Chiral GC analysis of *(S,S)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)sulfide (**3b**).

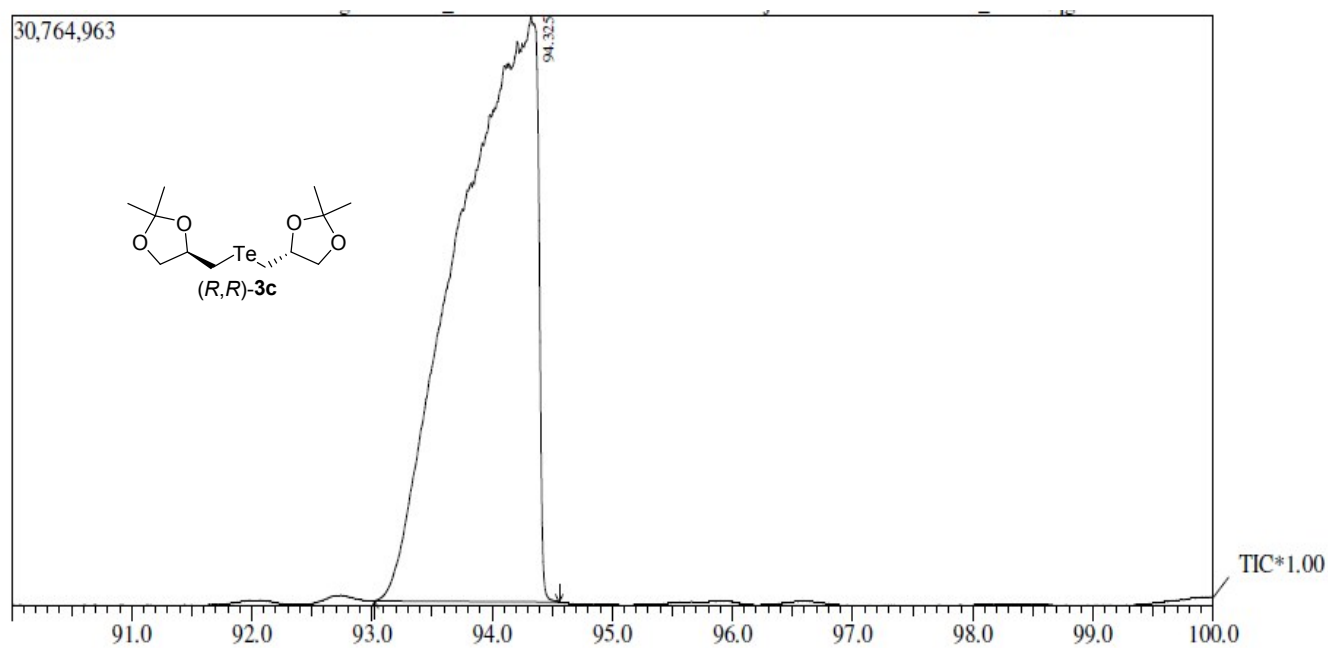


Figure 21. Chiral GC analysis of (*R,R*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)telluride (**3c**).

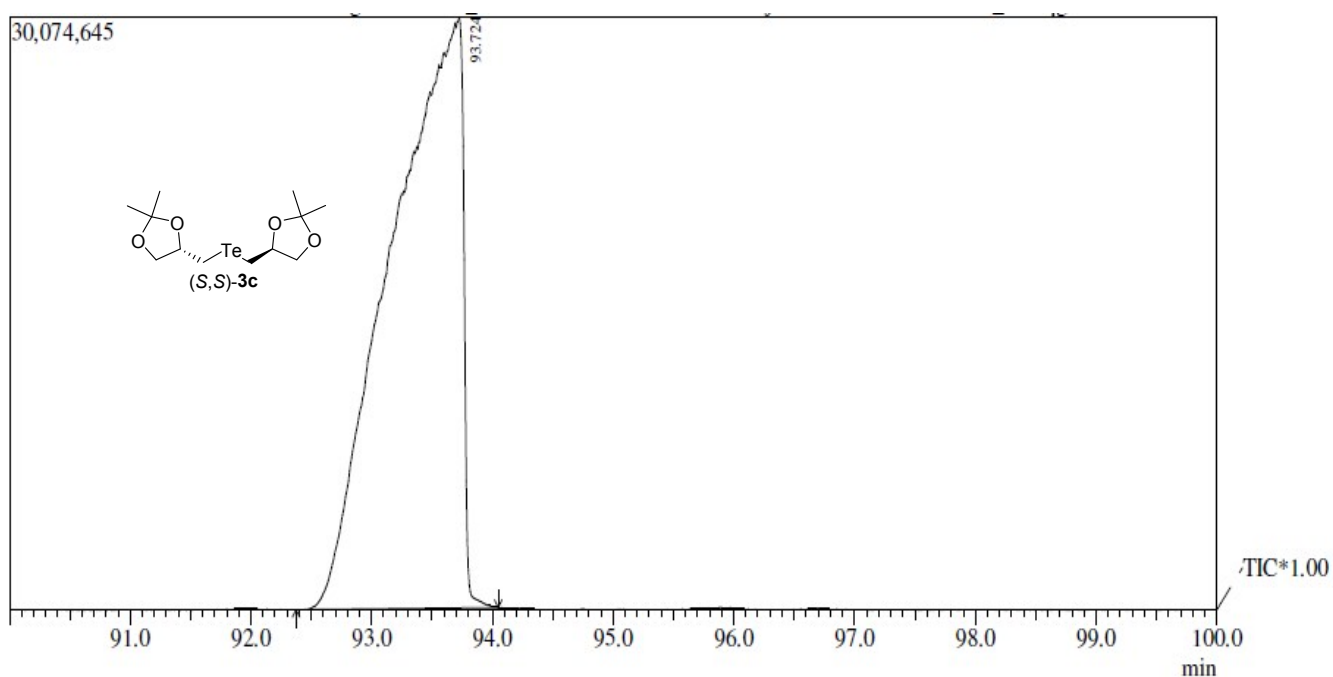


Figure 22. Chiral GC analysis of (*S,S*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)telluride (**3c**).

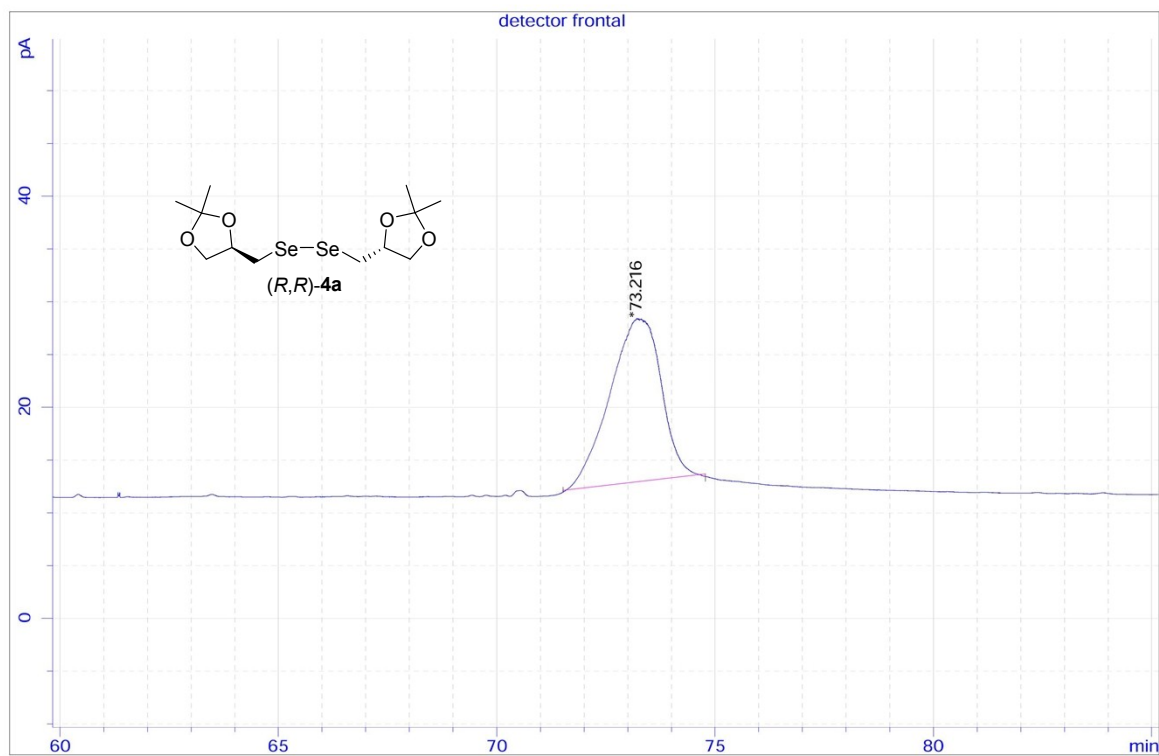


Figure 23. Chiral GC analysis of (*R,R*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)diselenide (**4a**).

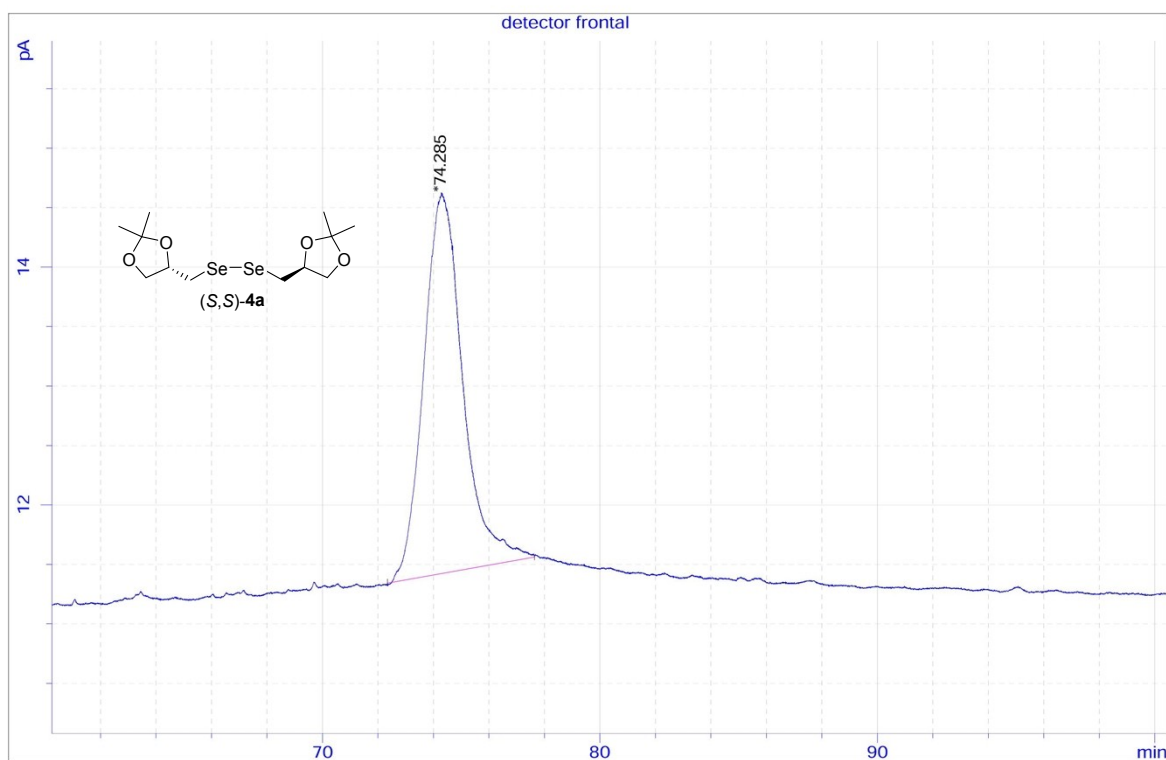


Figure 24. Chiral GC analysis of (*S,S*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)diselenide (**4a**).

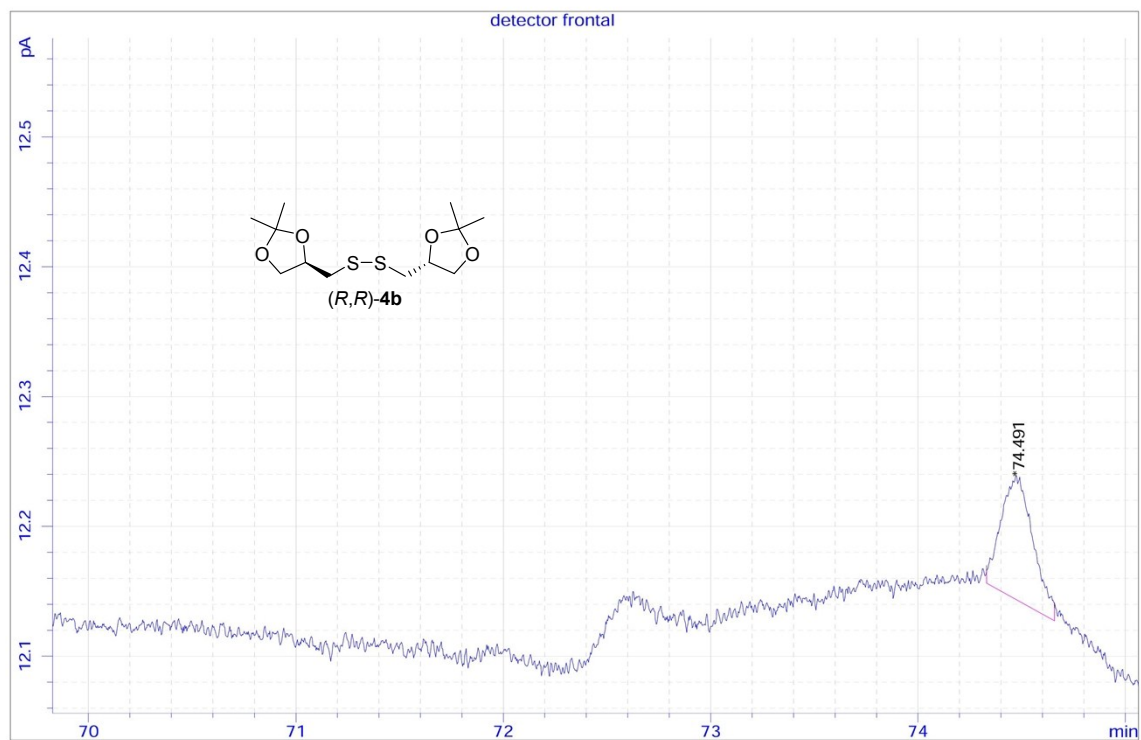


Figure 25. Chiral GC analysis of (*R,R*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)disulfide (**4b**).

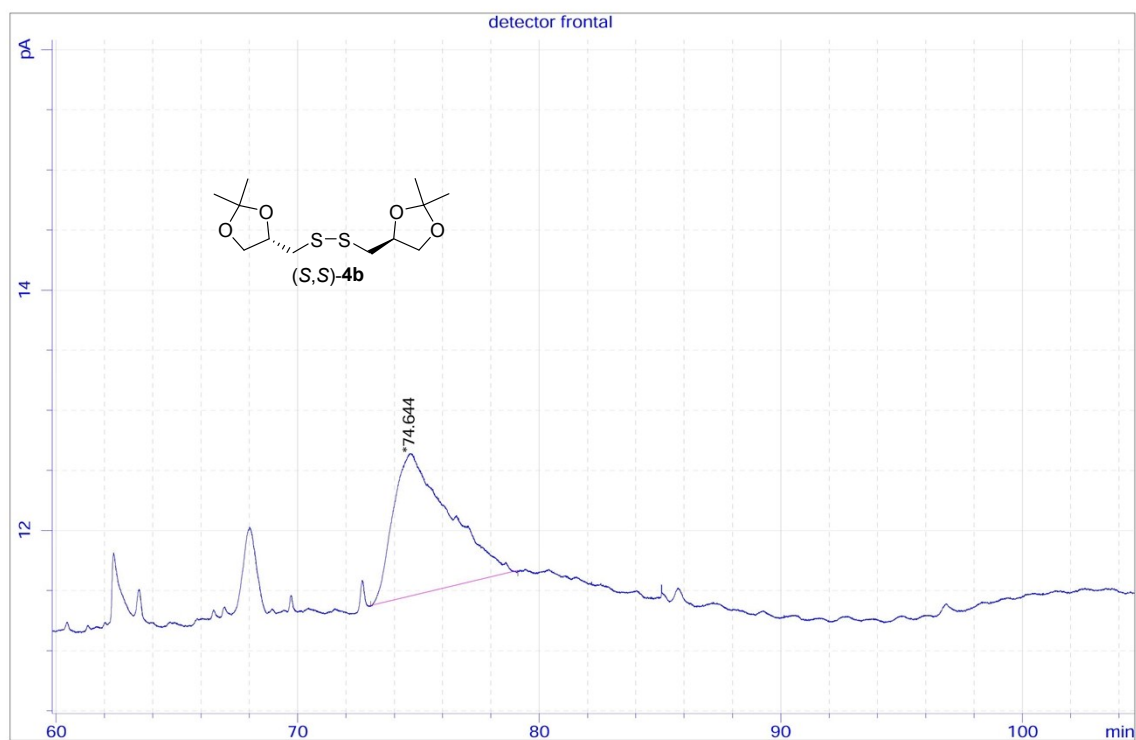
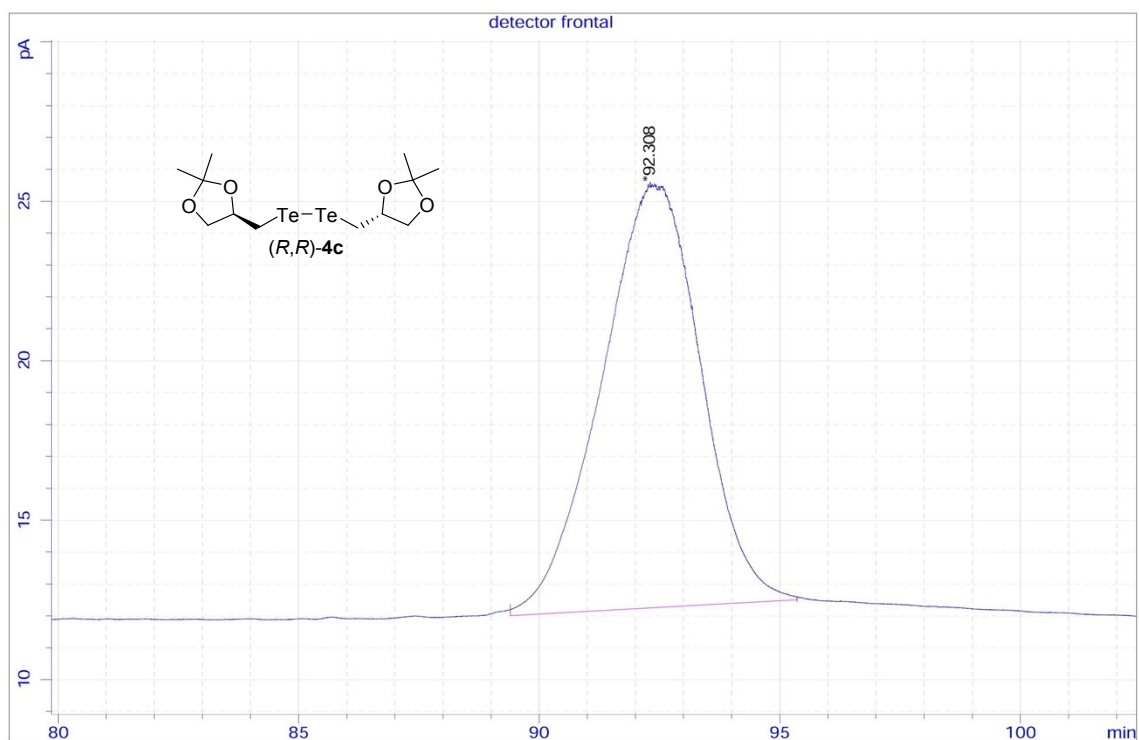
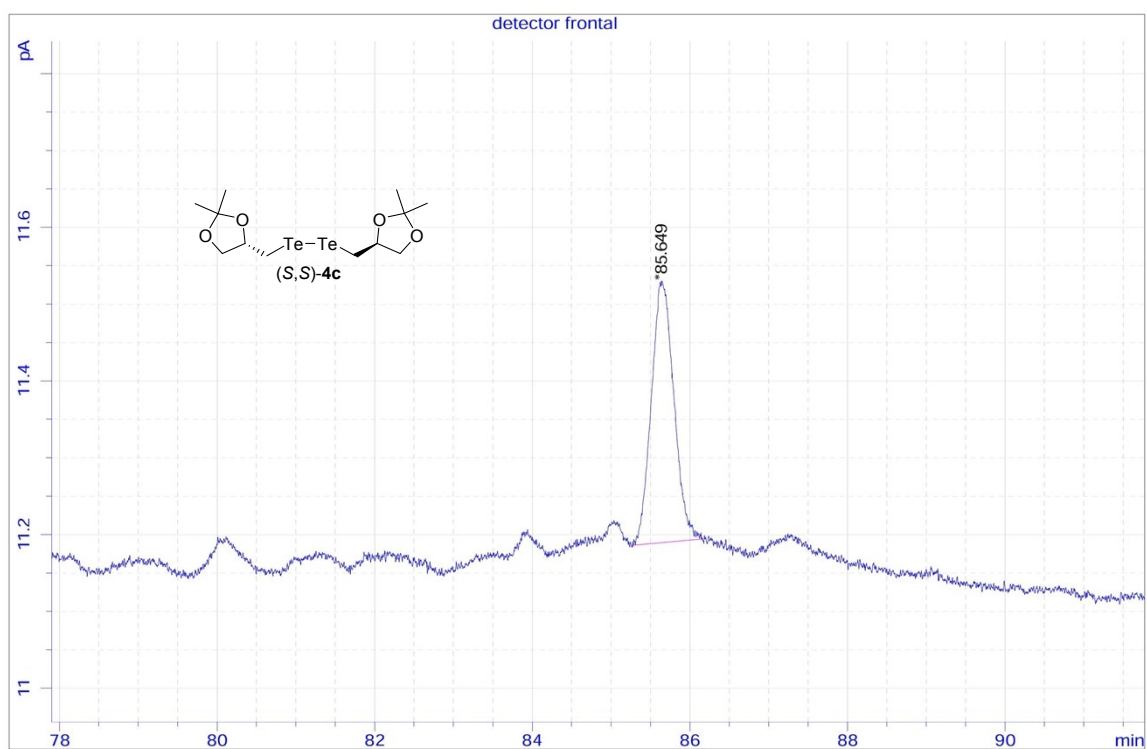


Figure 26. Chiral GC analysis of (*S,S*)-bis(2,2-dimethyl-1,3-dioxolanylmethyl)disulfide (**4b**).



Peak#	R.Time [Min]	Area	Area %
1	92,308	548,90123	100,00000

Figure 27. Chiral GC analysis of *(R,R)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)ditelluride (**4c**).



Peak#	R.Time [Min]	Area	Area %
1	85,649	2,71087	100,00000

Figure 28. Chiral GC analysis of *(S,S)*-bis(2,2-dimethyl-1,3-dioxolanylmethyl)ditelluride (**4c**).

Figure 30. Chiral GC analysis of (*R*)-3-(2,3-dihydroxypropylselanyl)propanenitrile (**6b**).