SUPPORTING INFORMATION

Contents

General Experimental

Benzeneseleninic acid (**1**) and benzeneselenenyl chloride (PhSeCl) were obtained from commercial sources. Hydrogen peroxide was titrated[1] prior to use and had a concentration of 30% ± 1%. ^{1}H, ^{13}C and ^{77}Se NMR spectra were recorded at 400 MHz, 101 MHz and 76 MHz, respectively, except for the ^{77}Se NMR spectrum of 4 + 0.5 equiv H_2O_2, which was recorded at 114 MHz. An external reference of diphenyl diselenide (453.0 ppm relative to dimethyl selenide)[2] was employed for ^{77}Se NMR spectra.

Caution: Although no explosions were observed during the course of this work, the peroxyseleninic and peroxyselenonic acids comprise potential explosion hazards and should be treated with appropriate precautions.

Synthetic Procedures

Preparation of benzeneperoxyseleninic acid (3). The general procedure of Syper and Młochowski[3] was followed. Benzeneseleninic acid (**1**) (501.9 mg, 2.64 mmol) was added in portions to 3.0 mL of 30% hydrogen peroxide at -5 ºC with stirring. A fine white precipitate formed and after 1.5 h at -5 ºC it was filtered, washed with a small amount of cold hydrogen peroxide solution and dried by suction, while shielded from light by aluminum foil. This afforded 445.6 mg (82%) of **3** as a white solid with mp 53 ºC (dec); lit.[3] mp 52 ºC (dec). The product could be stored in the solid state for several days at -25 ºC but decomposed rapidly in solution. HRMS (ESI): *m/z* calcd for $C_6H_5O_3{}^{80}Se$: 204.9409 $[M-H]^-$; found: 204.9405.

Preparation of benzeneselenonic acid (4).

A variation of a literature method was employed.[4] Benzeneselenenyl chloride (1.0028 g, 5.23 mmol) was dissolved in 100 mL of chloroform and cooled to 0 ºC. Hydrogen peroxide (2.7 mL, 30%, 26 mmol) was added and the reaction was stirred at room temperature for 2 h, during which the red colour was discharged. Volatile material was concentrated under vacuum to give 0.9713 g (91%) of **4** as a light yellow viscous oil that eventually solidified to a hygroscopic solid; m.p. 58-60 ºC (lit.[5] m.p. 64 ºC); ^{1}H NMR ($CDCl_3$) δ = 10.86 (br s, 1H), 8.09 (m, 2H), 7.79-7.70 ppm (m, 3H); ^{13}C NMR (D_2O) δ = 142.3, 133.5, 129.8, 125.2 ppm; ^{77}Se NMR ($CDCl_3$) δ = 1027.4 ppm; ^{77}Se NMR (D_2O) δ = 1024.9 ppm; HRMS (ESI) *m/z* calcd. for $C_6H_5O_3{}^{80}Se$ $[M-H]^-$: 204.9409; found: 204.9404. This product was also prepared from the oxidation of salt **7** (vide infra).

Preparation of the mixed benzeneselenonium-benzeneselenonate salt 7.

Method 1. Benzeneseleninic acid (**1**) (379 mg, 2.00 mmol) was suspended in 6 mL of acetonitrile and stirred with hydroxen peroxide (107 μL, 30%, 1.0 mmol) at room temperature for 0.5 h, followed by an additional 0.5 h at 75 ºC. The solution was cooled to afford 291 mg (74%) of **7** as colourless needles; m.p. 142-144 ºC. A crystal grown from chloroform was subjected to x-ray diffraction (vide infra).

Method 2. The solid peroxyseleninic acid **3** (64.9 mg, 0.316 mmol) in 1 mL of acetonitrile was slowly heated to 80 ºC and maintained at that temperature for an additional 30 min. Upon cooling, the product **7** crystallized (39.4 mg, 63%) and produced the same x-ray crystal structure as the previous sample. The products from Methods 1 and 2 had the same NMR spectra; ^{1}H NMR ($CDCl_3$-CD_3OD, 95:5) δ = 8.02-7.93 (m, 2H), 7.91-7.83 (m, 2H), 7.82-7.71 (m, 1H), 7.73-7.62

ppm (m, 5H); ^{13}C NMR (CD_3CN) δ = 145.8, 142.5, 134.0, 132.3, 129.9, 129.0, 125.9, 125.7 ppm; ^{77}Se NMR (UDEFT; $CDCl_3$-CD_3OD, 95:5) δ = 1217.5, 1026.3 ppm.

Preparation of selenonium salt 8.

Trifluoroacetic acid (TFA, 19 µL, 0.26 mmol) was added to a solution of 19.8 mg (0.104 mmol) of benzeneseleninic acid (**1**) in 1.0 mL of $CDCl_3$ and the ^{1}H and ^{77}Se NMR spectra were recorded: ^{1}H NMR ($CDCl_3$) δ = 7.77-7.94 (m, 2H), 7.63-7.55 ppm (m, 3H) ; ^{77}Se (UDEFT, $CDCl_3$) NMR δ = 1214.5 ppm.

Decomposition of benzeneperoxyseleninic acid (3).

A sample of **3** was dissolved in CD_3CN at room temperature and the formation of bubbles was observed immediately. After a signal acquisition of 40 min, the ^{77}Se NMR spectrum revealed a peak at 1025.1 ppm from the salt **7** and a much smaller peak at 1162.3 ppm, tentatively attributed to unreacted peroxyseleninic acid **3** that briefly existed during the early stages of acquisition. The weaker peak at 1217.5 ppm from **7** could not be discerned from the baseline under these conditions. Further characterization of **3** was not possible due to its instability in solution.

Further oxidation of compound 7.

Compound **7** (39.6 mg, 0.100 mmol) and hydrogen peroxide (10.2 µL, 30%, 0.10 mmol) were dissolved in $CDCl_3$-CD_3OD (9:1) in an NMR tube and the UDEFT ^{77}Se NMR spectrum was acquired over 2 h. The spectrum showed a strong signal at 1024.6 ppm, in good agreement with the ^{77}NMR spectrum of an authentic sample of selenonic acid **4** (1025.0 ppm) prepared from benzeneselenenyl chloride as described above.

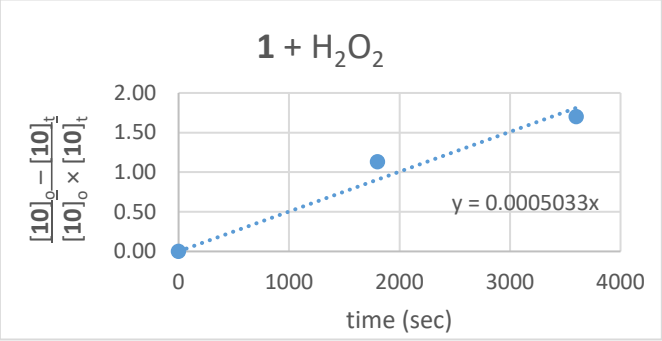
Kinetic Measurements of Cyclooctene Epoxidations

Cyclooctene (55 mg, 0.50 mmol), naphthalene (64 mg, 0.50 mmol) and the appropriate selenium compound (0.50 mmol) were dissolved in 1 mL of dichloromethane. Hydrogen peroxide (51 µL, 30%, 0.50 mmol) was added and the reaction mixture was stirred at room temperature. Progress was monitored by GC analysis at regular intervals by removing 20 µL aliquots and diluting them with 100 µL of diethyl ether. The amount of epoxide formed was determined by peak integration relative to the naphthalene internal standard.

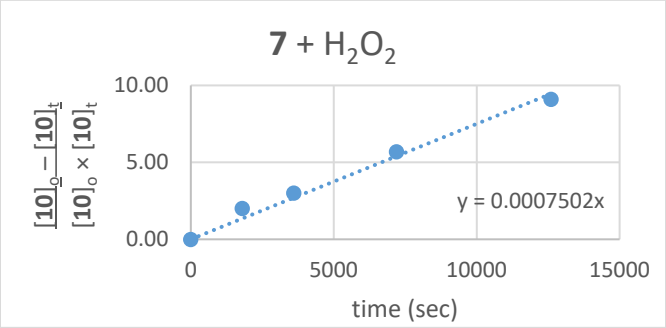
Retention times: cyclooctene R_t = 6.66 min; cyclooctene oxide R_t = 10.01 min; naphthalene R_t = 11.23 min. Column: DB-5 (0.25 micron), 30 m x 0.25 mm; GC parameters: injector temperature 250 °C; 100:1 split ratio; column oven temperature at 50 °C for 2 min, ramped by 10 °C/min to 250 °C; flow rate: 1.0 mL/min; injection volume: 1 µL.

Approximate second order rate constants for the reactions shown in Figure 4 at 22 ºC were determined from the slopes of the following plots.

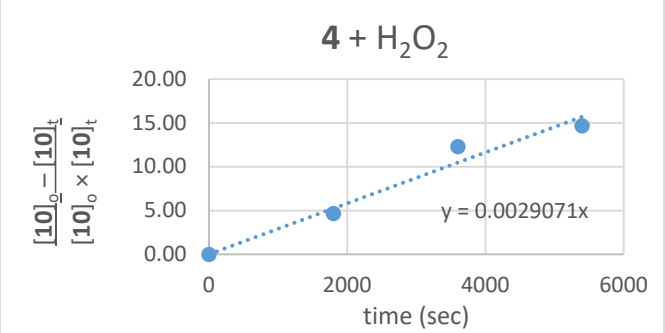
Seleninic acid **1** + H_2O_2: k = 5.0 ×10^{-4} L $mol^{-1}sec^{-1}$



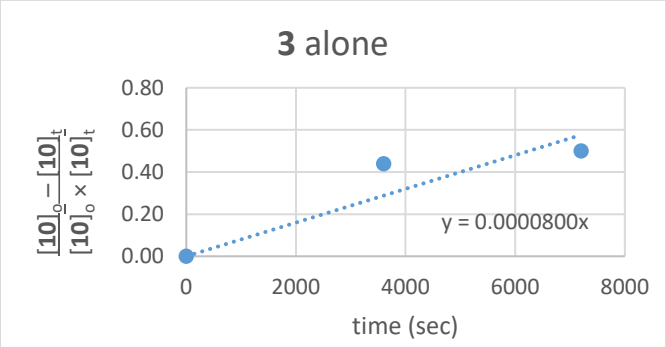
Selenonium selenonate salt **7** + H_2O_2: k = 7.5×10^{-4} L $mol^{-1}sec^{-1}$



Selenonic acid **4** + H_2O_2: k = 29×10^{-4} L $mol^{-1}sec^{-1}$



Peroxyseleninic acid **3** alone: k = 0.80×10^{-4} L $mol^{-1}sec^{-1}$



Polarization effects in peroxy acids 3 and 13, and pK_a Measurements

The greater rate of epoxidation with peroxy acid **13** (generated in situ from **4**)**,** compared to that of **3**, is evident from Figure 4 and can be rationalized as follows. In epoxidations with peroxycarboxylic acids, the alkene π-bond functions as a Lewis base via electron donation to the σ* orbital of the O-O bond.[6] Furthermore, the Bartlett mechanism[7] invokes intramolecular hydrogen-bonding between the carbonyl oxygen and the hydroxyl hydrogen atoms in the transition state. Such epoxidations are facilitated by more strongly electrophilic peracids. In the present case, the more highly oxygenated and electrophilic peroxyselenonic acid **13** displays both a more polarized O-O bond and Se=O bond compared to its peroxyseleninic acid counterpart **3**. This facilitates both O-O bond cleavage and proton transfer from the hydroxyl group to a more strongly basic selenoxide-like oxygen atom of **13** during the epoxidation. The increased electron deficiency in **13** is reflected in the pK_a values of the corresponding acids **4** and **1**, which were found to be 1.82 and 4.56, respectively.[8] The greater acidity of **4** indicates that the selenonate moiety is more strongly electron-withdrawing (and more stable) than its seleninate counterpart, resulting in a more polarized O-O bond in **13.** Charge density calculations further confirmed the relative O-O and Se=O polarization in compounds **3** and **13** (see Computations below).

A solution of 0.5 mmol of either **1** or **4** in deionized water was partly neutralized with a similar solution containing 0.25 mmol of NaOH and each solution was diluted to 10 mL. The pH of the two solutions was measured to be 4.56 and 1.82, respectively. Since pK_a = pH under conditions of 50% neutralization, these values also represent the pK_a's of the two acids under these conditions.

Computations.

DFT computations were performed by means of the Gaussian 09 platform[9] at the B3LYP level of theory, with the 6-311G (2df, 2p) basis set.[10] Geometry and frequency optimizations were carried out on benzeneperoxyseleninic acid (**3**) and benzeneselenonic acid (**4**). In each case optimizations were performed from several different input conformations to ensure discovery of the global minimum energy conformation. The relative energies of the lowest energy conformations indicated that the selenonic acid **4** was more stable than the peroxyseleninic acid isomer **3** by 28.8 kcal mol^{-1}. Z-Matrices and energies are provided for each species. No imaginary frequencies were observed.

Benzeneperoxyseleninic acid (**3**)

```
 Symbolic Z-matrix:
 Charge =  0 Multiplicity = 1
 C                    -7.06411  -0.9484   -0.60879
 C                    -5.73381  -0.69185  -0.96713
 C                    -4.99933   0.28499  -0.28138
 C                    -5.59514   1.00526   0.76271
 C                    -6.92544   0.7487    1.12105
 C                    -7.65993  -0.22813   0.4353
 H                    -7.6249   -1.69424  -1.13238
 H                    -5.27889  -1.24179  -1.76432
 H                    -3.98362   0.48088  -0.55498
 H                    -5.03435   1.75109   1.2863
 H                    -7.38036   1.29864   1.91824
 Se                   -9.5015   -0.58329   0.93136
 O                    -9.6478   -2.12108   1.38654
 O                    -10.59191  -0.26547  -0.50353
 O                    -10.24533  -1.05504  -1.50295
 H                    -10.33254  -1.97176  -1.23161

E = -2859.47770084 a.u.
```

Benzeneselenonic acid acid (**4**)

```
 Symbolic Z-matrix:
 Charge =  0 Multiplicity = 1
 C                    -7.01368  -0.8275   -0.71973
```

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 C                    -5.7027   -0.47462  -1.06719
 C                    -4.99147   0.44097  -0.27994
 C                    -5.59121   1.00368   0.85477
 C                    -6.90219   0.6508    1.20224
 C                    -7.61342  -0.26479   0.41499
 H                    -7.55672  -1.52657  -1.32081
 H                    -5.24478  -0.90427  -1.93358
 H                    -3.9905    0.7104   -0.54524
 H                    -5.04817   1.70276   1.45585
 H                    -7.36011   1.08045   2.06862
 Se                   -9.42825  -0.75328   0.896
 O                    -10.56206  -0.30848  -0.46985
 O                    -9.51498  -2.55608   1.19818
 O                    -10.0382  -0.09447   2.49062
 H                    -10.98057  -0.26104   2.56674

E = -2859.52362888 a.u.
```

Benzeneperoxyselenonic acid (**13**)

```
 Symbolic Z-matrix:
 Charge =  0 Multiplicity = 1
 C                    -6.15621  -0.77014   5.24547
 C                    -4.77165  -0.97703   5.18122
 C                    -4.26587  -2.17949   4.66918
 C                    -5.14463  -3.17507   4.2214
 C                    -6.52919  -2.96818   4.28564
 C                    -7.03498  -1.76572   4.79768
 H                    -6.54239   0.14797   5.63642
 H                    -4.1007   -0.21688   5.52312
 H                    -3.20873  -2.33745   4.62013
 H                    -4.75845  -4.09318   3.83045
 H                    -7.20015  -3.72833   3.94374
 Se                   -8.95166  -1.47932   4.88662
 O                    -9.78227  -3.04625   5.33796
 O                    -9.32044  -0.20758   6.14978
 O                    -9.7198   -0.73877   3.39986
 O                    -11.03411  -0.79161   3.51024
 H                    -11.42995  -0.7778    2.63575

E = -2934.67025931 a.u.
```

While the Mulliken charge densities are essentially identical on the selenium-bonded peroxide oxygens of **3** and **13** (-0.333 vs. -0.332, respectively), they are less negative on the hydroxyl

oxygen of **13** (-0.275) compared to that in **3** (-0.298). The charge densities on Se are +0.983 and +1.369 in **3** and **13**, respectively, while their values on the H-bonded selenoxide oxygens are -0.598 and -0.546, respectively. Thus, both the O-O and Se=O bonds are more polarized in **13**.

XAS Analysis of Salt 7.

The sample was prepared as follows: benzeneseleninic acid (**1**) (9.6 mg, 0.05 mmol) and 29% hydrogen peroxide (6.14 µL, 0.058 mmol) were dissolved in 0.5 mL of $CDCl_3$-CD_3OD (95:5) and allowed to stand for 18 h. The UDEFT ^{77}Se NMR spectrum showed peaks at δ 1217.9 (small) and 1026.7 ppm after this time. The solution was transferred to a vial and volatile material was removed under high vacuum at room temperature. The resulting solid was packed in dry ice and sent for analysis.

Selenium K-edge X-ray absorption (XAS(spectra were collected at the Stanford Synchrotron Radiation Lightsource (SSRL) using beamline 7-3 employing a Si(220) double-crystal monochromator. The SPEAR storage ring operated at 3.0 GeV with a beam current of 500 mA and the incoming and transmitted beam were measured with N2-filled gas ionization chambers. Fluorescence spectra were collected by monitoring the Se Kα12 emission using a 30-element germanium array detector, employing arsenic X-ray filters with a Soller slit assembly to reduce the background signal from scattered radiation. During data collection, in order to reduce thermal vibrations and minimize the damage from the beam, samples were maintained at a temperature of approximately 10 K using an Oxford instruments CF1204 liquid helium flow cryostat. Four to six scans, each of 25 min duration, were collected for each data set and were examined for changes related to beam exposure indicative of photodamage. A hexagonal selenium foil was collected

simultaneously with the data for calibration; the lowest-energy foil was assumed to be 12658.0 eV. Samples were inclined at an angle of 45° to the incident X-ray beam to facilitate measurement of X-ray fluorescence. Data reduction and analysis was carried out as previously described using the EXAFSPAK suite of computer programs.[11] See Figure 1 below.

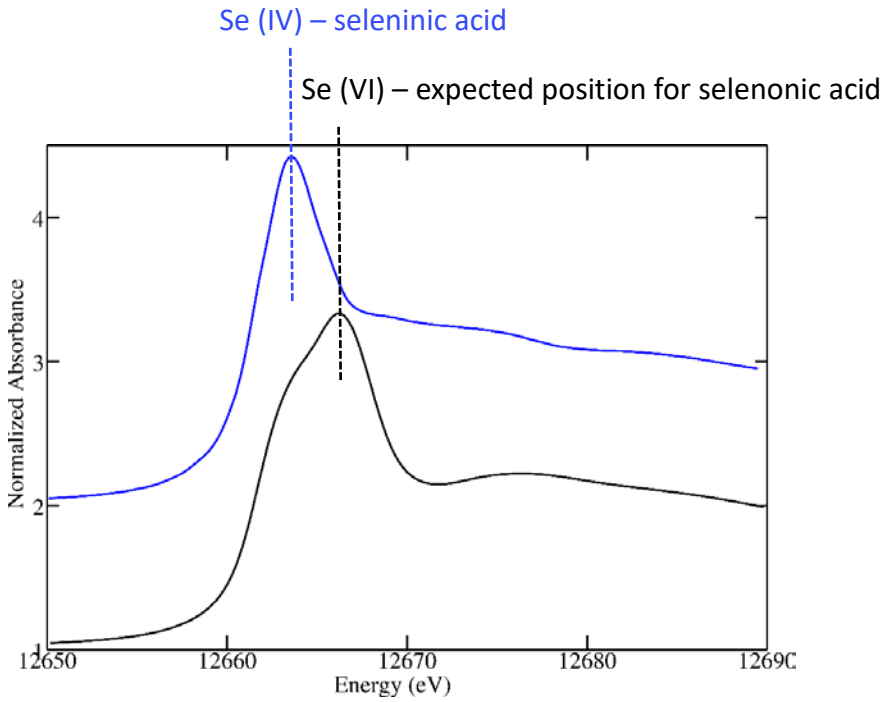


Figure 1. Selenium K-edge X-ray absorption near-edge spectra corresponding to phenylseleninic acid in absence (blue line) or presence (black line) of hydrogen peroxide. The sample with hydrogen peroxide exhibits features indicative of a mixture of Se (IV), and Se (VI).

X-ray Crystal Structure of Salt 7.

Single crystals of **7** were grown by slow evaporation from chloroform solution. A suitable crystal was selected and mounted on a glass loop using Paratone. Diffraction experiments were performed on a Nonius Kappa diffractometer equipped with a Siemens Fine Focus Ceramic Tube

(graphite monochromated Mo Kα, λ = 0.71069 Å) and an APEX II CCD detector. The crystal was kept at 173 K during data collection. Diffractions spots were integrated and scaled with SAINT[12] and the space group was determined with XPREP.[13] Using Olex2,[14] the structure was solved with the ShelXT[15] structure solution program using Intrinsic Phasing and refined with the ShelXL[16] refinementK package using Least Squares minimisation. Acidic H atoms could not be located from the difference map and were placed on the selenonium cation because of the expected greater acidity of **4** and the amphoteric nature of **3**. A response to the two "level A" alerts are provided in the Validation Form of the check CIF file.

Table 1. Crystal data and structure refinement for Compound 7.

Identification code	KS-05-142
Empirical formula	$C_{12}H_{12}O_5Se_2$
Formula weight	394.14
Temperature/K	173.0
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	6.0938(6)
b/Å	21.015(2)
c/Å	10.8842(9)
α/°	90
β/°	101.608(3)
γ/°	90
Volume/Å^3	1365.4(2)
Z	4
ρ_{calc}g/cm^3	1.917
μ/mm^{-1}	5.428
F(000)	768.0
Crystal size/mm^3	0.559 × 0.18 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.96 to 54.198
Index ranges	$-7 \leq h \leq 7$, $-26 \leq k \leq 26$, $-13 \leq l \leq 13$
Reflections collected	18865
Independent reflections	2999 [R_{int} = 0.0667, R_{sigma} = 0.0494]
Data/restraints/parameters	2999/54/210

Goodness-of-fit on F^2	1.140
Final R indexes [I>=2σ (I)]	R_1 = 0.0577, wR_2 = 0.1363
Final R indexes [all data]	R_1 = 0.0730, wR_2 = 0.1432
Largest diff. peak/hole / e $Å^{-3}$	1.45/-1.12

Table 2. Bond Lengths for Compound 11.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Se1	O1	1.620(5)	C4	C5B	1.401(17)
Se1	O2	1.637(5)	C5A	C6A	1.386(14)
Se1	O3	1.608(5)	C5B	C6B	1.402(18)
Se1	C1	1.901(7)	Se2	O4	1.730(5)
C1	C2A	1.412(14)	Se2	O5	1.734(5)
C1	C2B	1.351(16)	Se2	C7	1.919(7)
C1	C6A	1.361(14)	C7	C8	1.383(10)
C1	C6B	1.397(18)	C7	C12	1.374(10)
C2A	C3A	1.417(14)	C8	C9	1.383(11)
C2B	C3B	1.398(17)	C9	C10	1.370(12)
C3A	C4	1.427(15)	C10	C11	1.373(12)
C3B	C4	1.361(16)	C11	C12	1.381(11)
C4	C5A	1.353(15)			

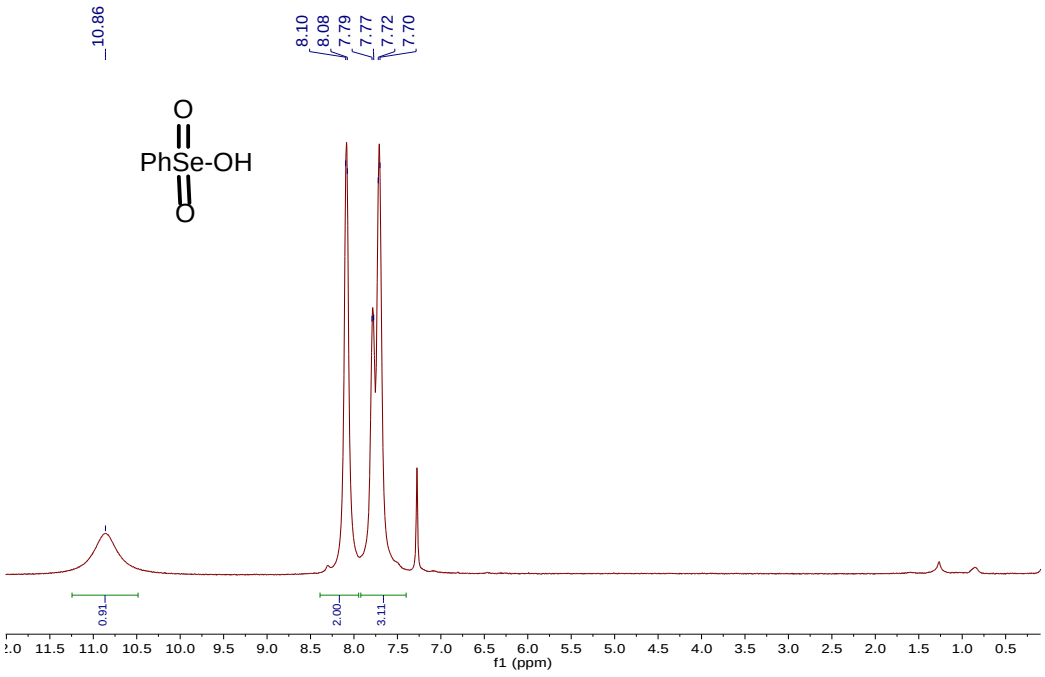
Table 3. Bond Angles for Compound 11.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	Se1	O2	108.9(3)	C5A	C4	C3A	120.8(10)
O1	Se1	C1	106.7(3)	C4	C5A	C6A	119.7(13)
O2	Se1	C1	106.3(3)	C4	C5B	C6B	118.5(19)
O3	Se1	O1	116.1(3)	C1	C6A	C5A	119.7(13)
O3	Se1	O2	110.0(3)	C1	C6B	C5B	118.1(19)
O3	Se1	C1	108.3(3)	O4	Se2	O5	96.9(2)
C2A	C1	Se1	117.2(7)	O4	Se2	C7	93.4(3)
C2B	C1	Se1	120.8(9)	O5	Se2	C7	94.6(3)
C2B	C1	C6B	122.4(13)	C8	C7	Se2	119.5(5)
C6A	C1	Se1	118.4(7)	C12	C7	Se2	118.8(6)
C6A	C1	C2A	124.2(9)	C12	C7	C8	121.7(7)
C6B	C1	Se1	116.4(10)	C7	C8	C9	117.9(7)
C1	C2A	C3A	114.8(12)	C10	C9	C8	121.2(8)

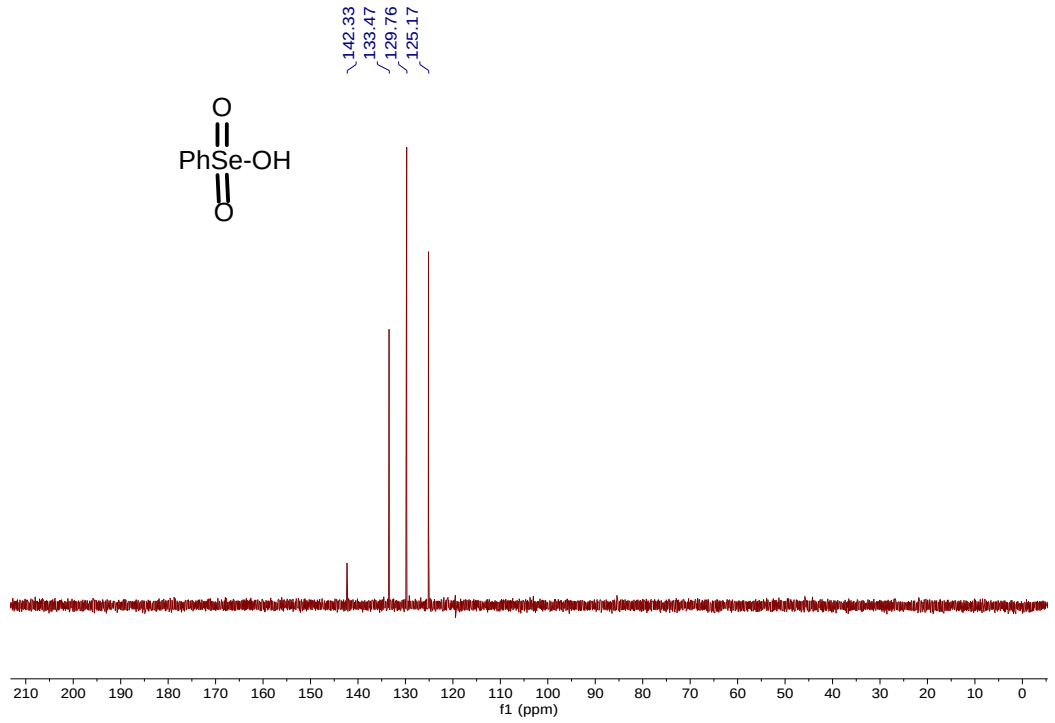
C1	C2B	C3B	118.5(16)	C9	C10	C11	119.9(8)
C2A	C3A	C4	120.4(12)	C10	C11	C12	120.2(8)
C4	C3B	C2B	120.2(15)	C7	C12	C11	119.1(7)
C3B	C4	C5B	120.1(13)				

NMR Spectra

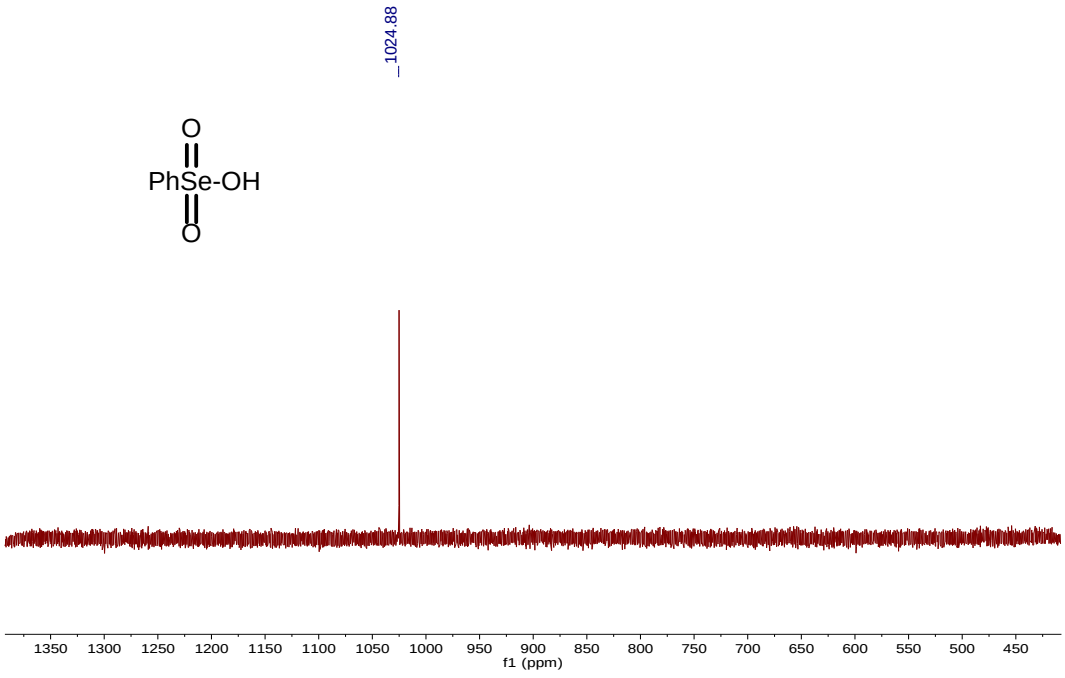
^{1}H NMR Spectrum of 4 ($CDCl_3$)



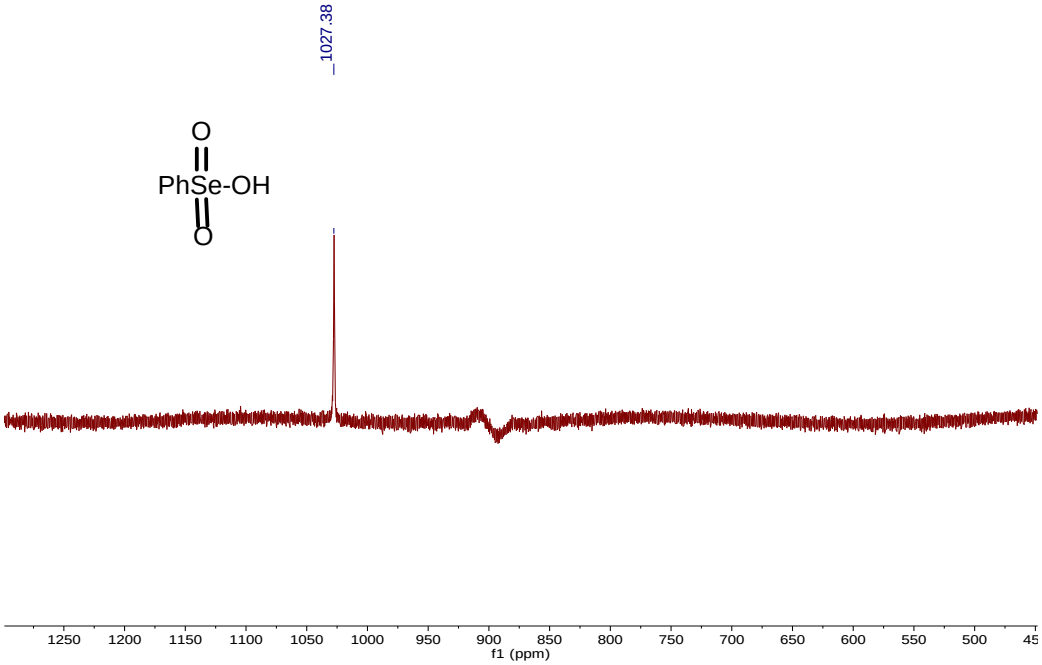
^{13}C NMR Spectrum of 4 (D_2O)



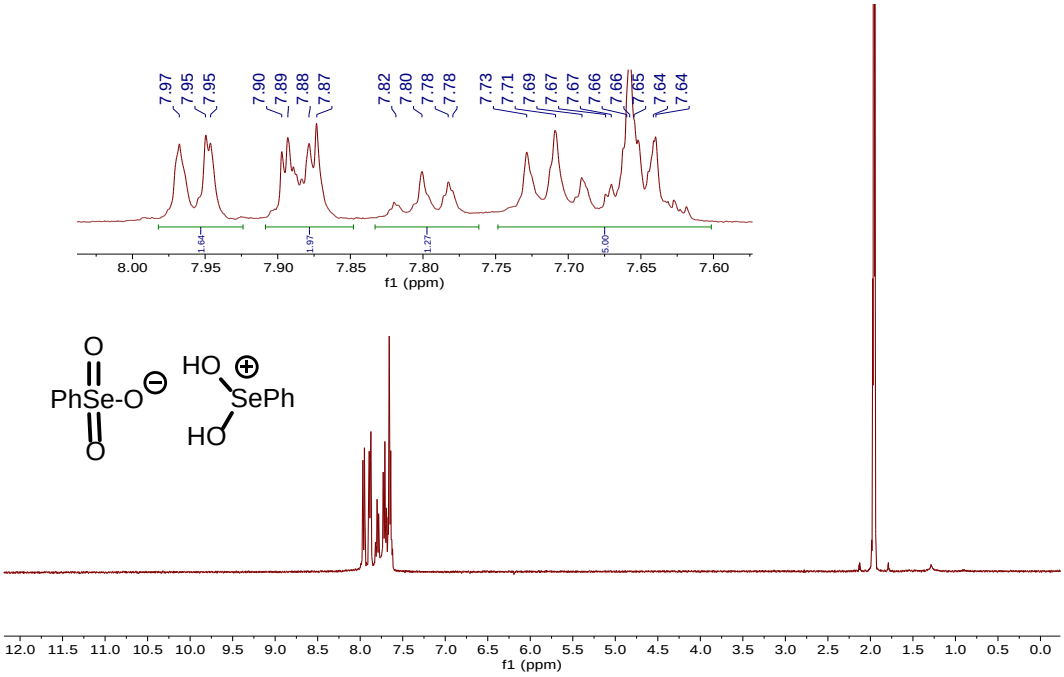
^{77}Se NMR Spectrum of 4 (D_2O)



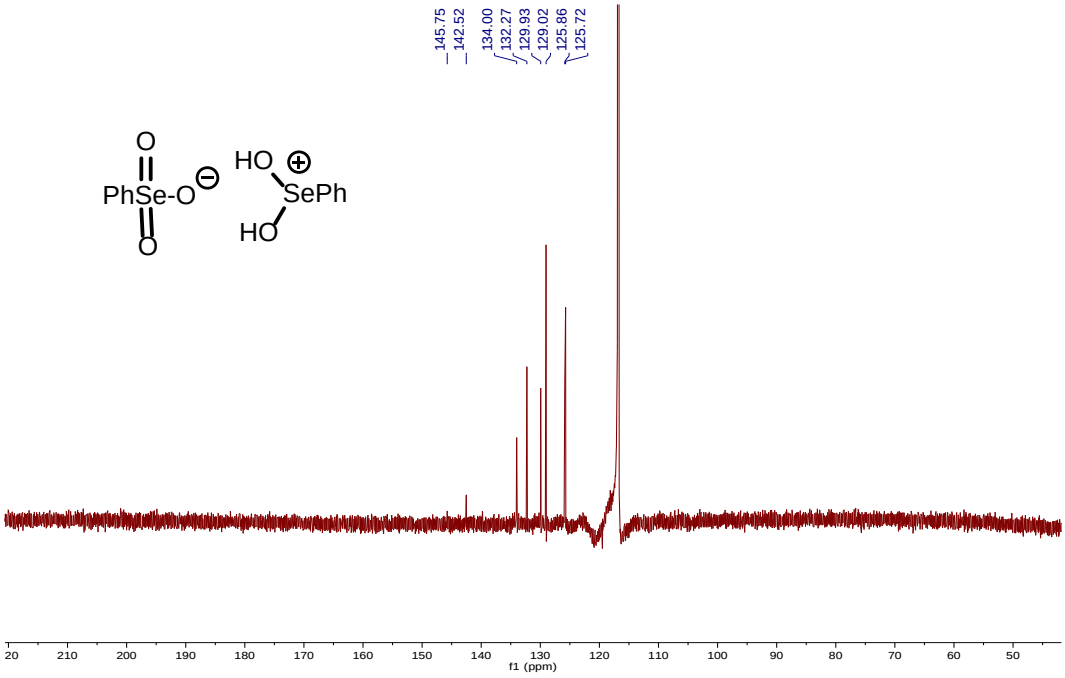
^{77}Se NMR Spectrum of 4 ($CDCl_3$)



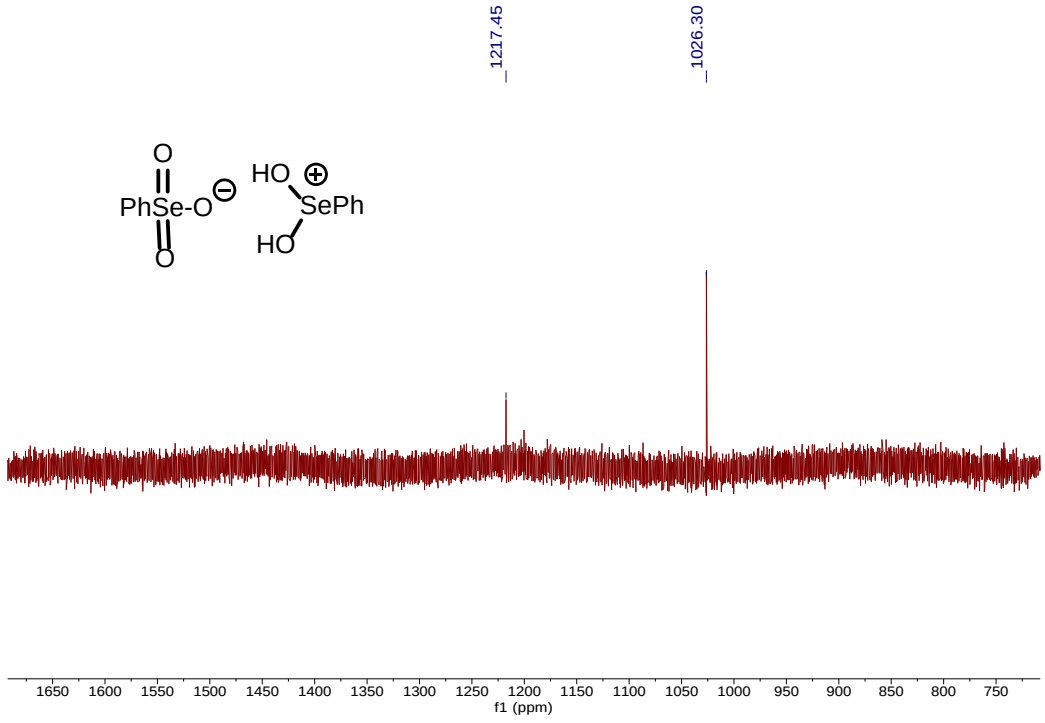
^{1}H NMR Spectrum of 7 (CD_3CN)



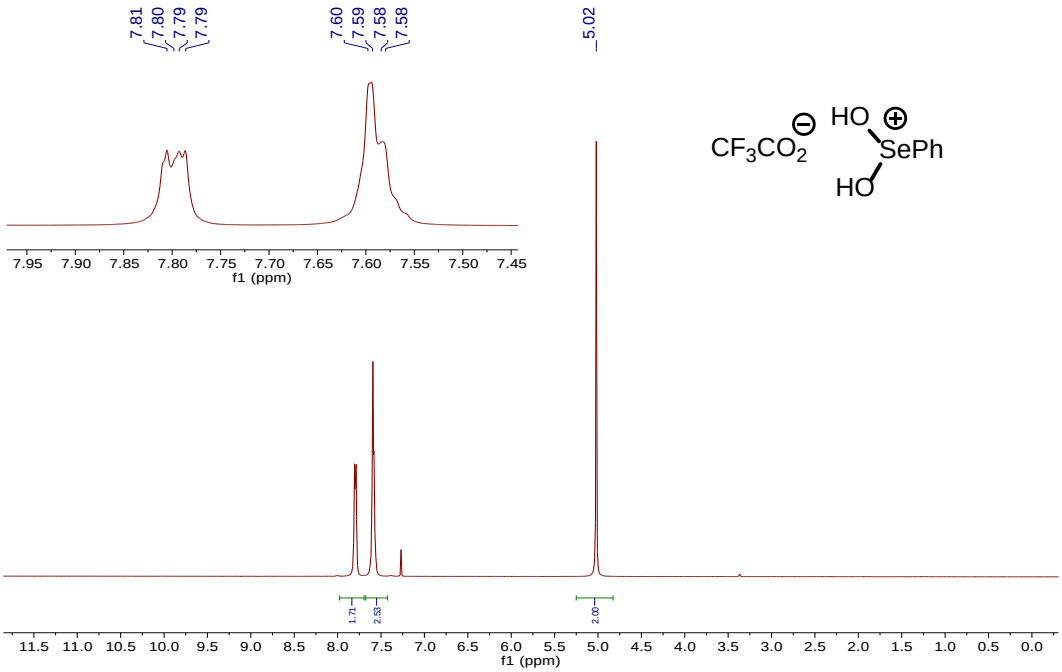
^{13}C NMR Spectrum of 7 (CD_3CN)



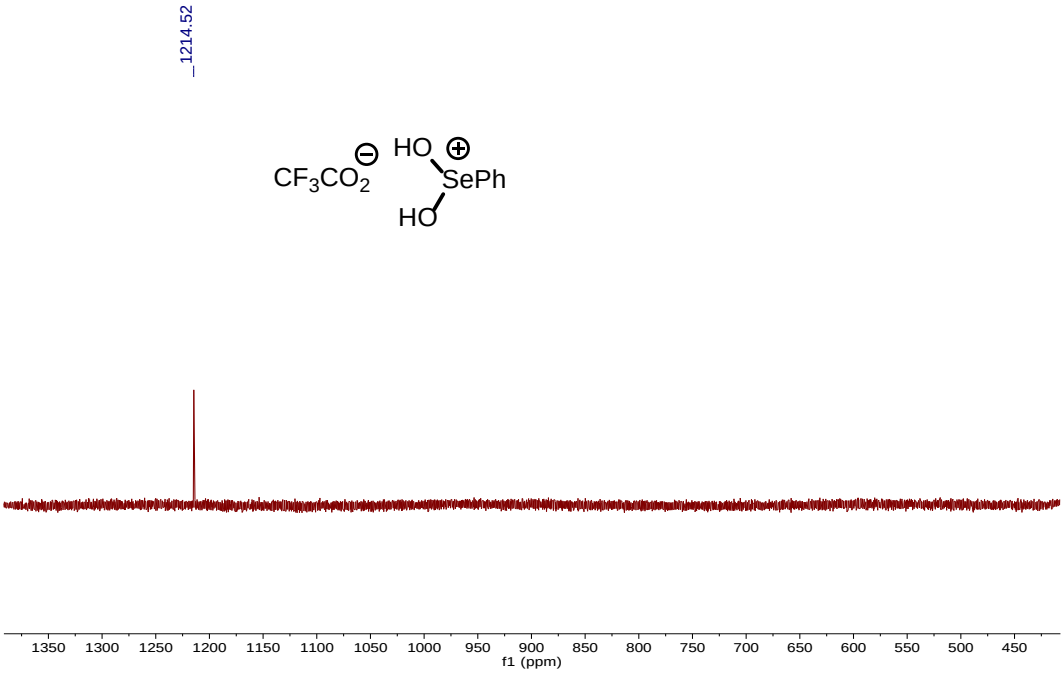
^{77}Se NMR Spectrum of 7 ($CDCl_3$-CD_3OD, 95:5)



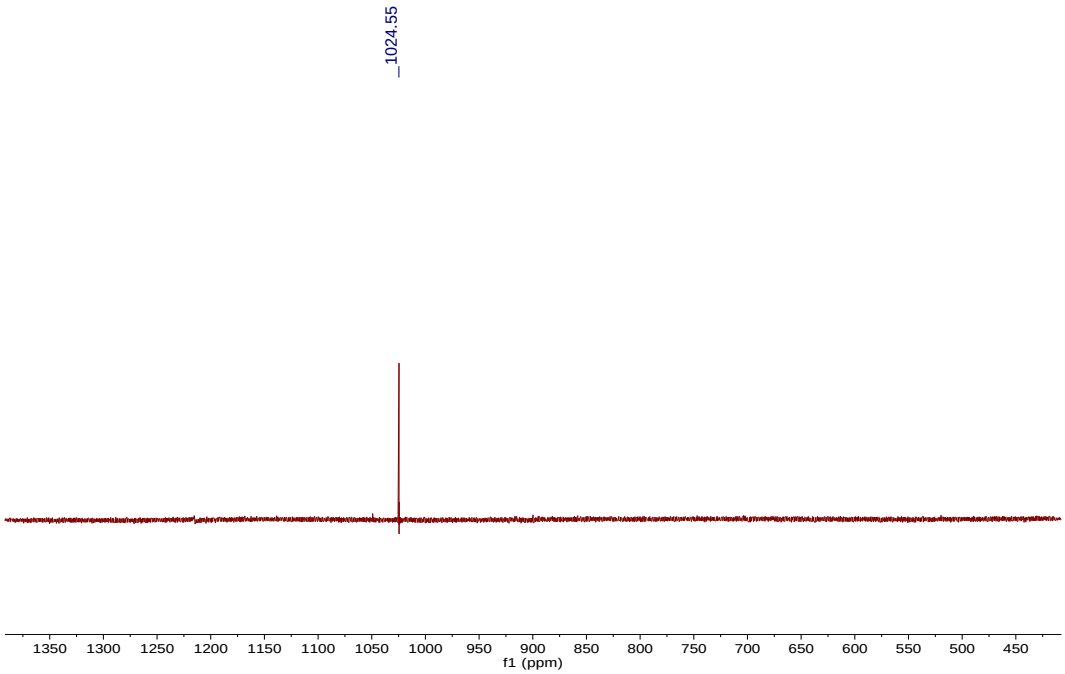
^{1}H NMR Spectrum of Selenonium TFA Salt 8 ($CDCl_3$).



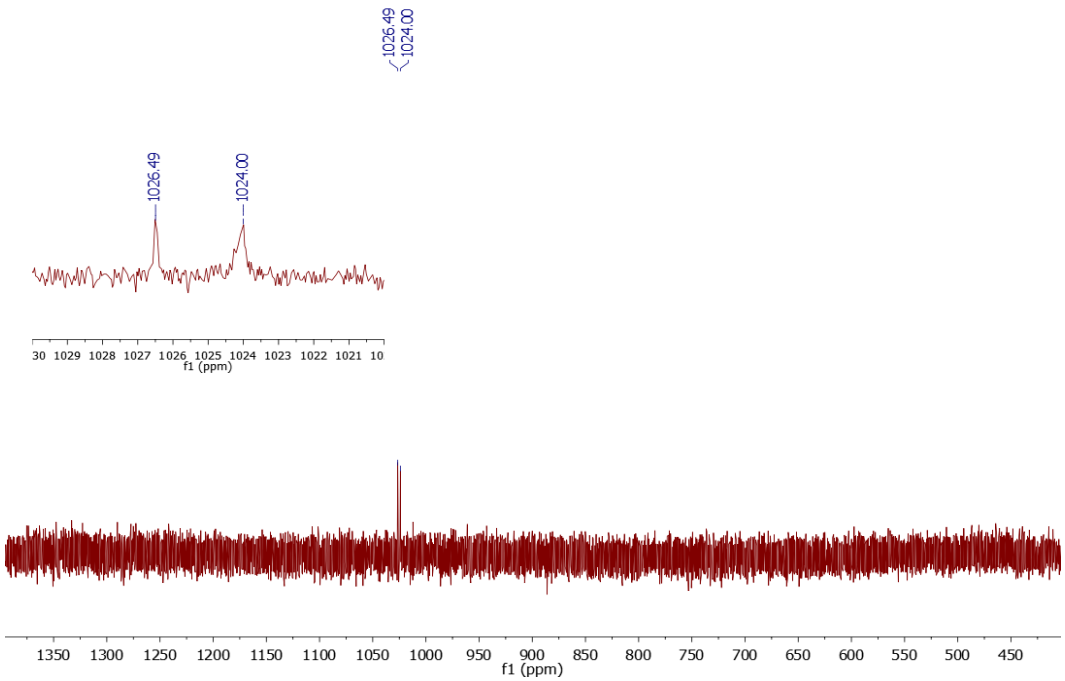
^{77}Se NMR Spectrum of Selenonium TFA Salt 8 ($CDCl_3$).



^{77}Se NMR Spectrum of Salt 7 + 1 equiv H_2O_2 ($CDCl_3$-CD_3OD, 9:1) after 2 h



^{77}Se NMR Spectrum of 4 + 0.5 equiv H_2O_2 ($CDCl_3$)



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