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# 2,2,2-Trifluoroethyl aryl selenides: $ArSeCH_2CF_3$ ($Ar = C_6H_5$, $p$-$CH_3C_6H_4$) and their derivatives $ArSe(O)CH_2CF_3$ and $ArSe(X)_2CH_2CF_3$ ($X = Cl$, Br, $OOCCF_3$)

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## Abstract

The title selenides were prepared in good yield by reacting the appropriate aryl selenolate anions with 2,2,2-trifluoroethyl tosylate in refluxing tetrahydrofuran. These unsymmetrical bivalent selenides can be oxidised to the corresponding compounds of tetravalent selenium, $ArSe(X)_2CH_2CF_3$ ($X = Cl$ and Br) with elemental chlorine and bromine. The latter, upon basic hydrolysis, gave the selenoxides, $ArSe(O)CH_2CF_3$. Selenoxides have also been formed by direct reaction of the selenides with hydrogen peroxide. Upon reaction with trifluoroacetic anhydride, these gave trifluoroacetate derivatives, $ArSe(OOCCF_3)_2CH_2CF_3$, in quantitative yield. The compounds have been characterized via elemental analysis, infrared spectra, $^1H$ and $^{19}F$ NMR spectra, and mass spectra.

## Introduction

Several perfluoroalkyl/aryl derivatives of selenium [1–4] and tellurium [4–7] have been reported and have been studied to a greater extent than the partially fluorinated alkyl/aryl selenides and tellurides [8, 9]. The growing importance of organoselenium compounds, especially selenides [10] and selenanes [11], in organic synthesis makes their synthesis and chemistry particularly interesting. Other potential applications of such compounds are in the tailoring of new ligands based on fluorinated alkyl/aryl systems [4]. In continuation of our reports on the synthesis of organoselenium compounds [12, 13], we wish to report herein some chemistry of the 2,2,2-trifluoroethyl phenyl/*p*-tolyl selenides and their derivatives.

## Results and discussion

Organyl selenolate anions, $RSe^-$ ($R = $ alkyl/aryl), obtained by the reductive cleavage of diorganyl diselenides, $R_2Se_2$, have been used in the synthesis of unsymmetrical selenides in different media [14]. Diaryl diselenides react with sodium metal in refluxing tetrahydrofuran to produce aryl selenolate anions, $ArSe^-$ ($Ar = C_6H_5$, $p$-$CH_3C_6H_4$), which upon subsequent treatment with 2,2,2-trifluoroethyl tosylate in the presence of hexamethyl phosphoric acid triamide, HMPA, produce the desired selenides, $ArSeCH_2CF_3$, in good yield.

$$Ar_2Se_2 + 2Na \xrightarrow[\text{reflux}]{\text{THF}} 2ArSeNa \xrightarrow{2p\text{-}CH_3C_6H_4SO_3CH_2CF_3}$$

$$2ArSeCH_2CF_3 + 2p\text{-}CH_3C_6H_4SO_3Na$$

$(Ar = C_6H_5/p\text{-}CH_3C_6H_4)$

The selenides thus prepared are stable, colourless, offensive smelling liquids which are miscible with common solvents.

2,2,2-Trifluoroethyl aryl selenides react with chlorine or bromine in dichloromethane at room temperature to produce the corresponding derivatives $ArSe(X)_2CH_2CF_3$ ($X = Cl$ and Br). However, attempts to prepare iodide derivatives under similar conditions were unsuccessful. The chlorides are off-white while the bromides are reddish brown coloured compounds, soluble in slightly polar organic solvents such as dichloromethane and chloroform.

$$ArSeCH_2CF_3 \xrightarrow[CH_2Cl_2]{X_2} ArSe(X)_2CH_2CF_3$$
$$(X = Cl, Br)$$

They are relatively stable compounds at room temperature and have been characterized from IR and NMR studies. They dissociate into the corresponding selenides with elimination of halogen at temperatures higher than 120 °C.

$$ArSe(X)_2CH_2CF_3 \underset{\text{at r.t.}}{\overset{>120\ ^{\circ}C}{\rightleftharpoons}} ArSeCH_2CF_3 + X_2$$

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Upon treatment with dilute aqueous sodium hydroxide solution, these halides produce the corresponding selenoxides, $ArSe(O)CH_2CF_3$, in moderate yield. The yields decreased on employing stronger solutions of NaOH. The selenoxides were also obtained in quantitative yield on oxidising the parent selenides with hydrogen peroxide at low temperature.

$$\begin{tikzcd}
\mathrm{ArSe(X)_2CH_2CF_3} \arrow[r, "\mathrm{NaOH}"] & \mathrm{ArSe(O)CH_2CF_3} & \\
& & \mathrm{ArSeCH_2CF_3} \arrow[ul, "\mathrm{H_2O_2}", "-5\ ^\circ\mathrm{C}"']
\end{tikzcd}$$

The selenoxides are off-white solids which are soluble in dichloromethane, chloroform and benzene, but are less stable than the halides. They decompose slowly on long standing to their parent selenides in agreement with findings of Reich *et al.* [15] on phenyl ethyl selenoxide. However, if allowed to remain in contact with atmospheric oxygen, the selenoxides are converted into selenones. The identity of the selenones was confirmed by various physico-chemical techniques. Thus, mass-spectral studies confirmed the formation of selenones by the presence of molecular ion peaks in their fragmentation pattern.

$$\begin{tikzcd}
\mathrm{ArSeCH_2CF_3} & \mathrm{ArSe(O)CH_2CF_3} \arrow[l, "\mathrm{r.t.}"', "\mathrm{N_2}"] \arrow[dr, "\mathrm{air}"] & \\
& & \mathrm{ArSe(O)_2CH_2CF_3}
\end{tikzcd}$$

The addition of 1 equiv. of trifluoroacetic anhydride directly to $ArSe(O)CH_2CF_3$ at low temperature produced 2,2,2-trifluoroethyl aryl selenium(IV) bistrifluoroacetates in quantitative yield.

$$\mathrm{ArSe(O)CH_2CF_3} \xrightarrow{\mathrm{(CF_3CO)_2O}} \mathrm{ArSe(OOCCF_3)_2CH_2CF_3}$$

The presence of a single sharp peak in the $^{19}F$ NMR spectrum in the region expected for the trifluoroacetate group suggests essentially identical electronic environments for the fluorine atoms of the two trifluoroacetate groups. This is only possible when there is rapid ligand exchange across the selenium atom.

Trifluoroacetate derivatives are the least stable of all the derivatives prepared and decompose readily at room temperature into several unidentified products (on the basis of $^{19}F$ NMR spectra).

The analytical, infrared and mass-spectral data of the newly prepared compounds are listed in the Experimental section and NMR data are given in Tables 1 and 2. The mass spectra showed prominent peaks corresponding to the selenides $[ArSeCH_2CF_3]^+$ in general, although molecular ion peaks of low intensity were also observed in some cases. Mass-spectral peaks have been assigned and listed for some of the compounds.

## Experimental

2,2,2-Trifluoroethyl tosylate [16] and diaryl diselenides [17] were prepared by standard methods. Trifluoroacetic anhydride was prepared by reacting trifluoroacetic acid with phosphorus(V) oxide. Solvents were distilled and purified before use [18].

Infrared (IR) spectra were recorded as neat liquids or Nujol mulls between AgCl plates on a Perkin-Elmer 1430 spectrophotometer. $^1H$ and $^{19}F$ NMR spectra were recorded on a Varian EM-390 (90 MHz) instrument. $Me_4Si$ ($^1H$, 0 ppm) and $CFCl_3$ ($^{19}F$, 0 ppm) were used as internal standards, respectively. Positive chemical shifts are downfield from the reference. Mass spectra were recorded using the electron impact ionisation (EI) mode at 70 eV on a VG Micromass 7070F instrument. Selenium [19] and halogens [18] were determined by standard methods. Carbon and hydrogen were determined microanalytically on a Perkin-Elmer 2400 CHN analyzer.

### *Preparation of $C_6H_5SeCH_2CF_3$ (1a)*

Diphenyl diselenide (15.60 g, 0.05 mol) and sodium (2.3 g, 0.1 mol) were refluxed in dry tetrahydrofuran (~100 ml) for 4 h. Hexamethyl phosphoric acid triamide (HMPA) was then added, followed by 2,2,2-trifluoroethyl tosylate (24.5 g, 0.1 mol) dissolved in 25 ml of dry tetrahydrofuran. Refluxing was continued for a further 4 h. The resulting reaction mixture was cooled and extracted with ether (3×50 ml). The combined extracts were washed with 5% aqueous sodium hydroxide (100 ml) and dried over anhydrous sodium sulphate. Removal of ether, followed by distillation of the product under vacuum, afforded 2,2,2-trifluoroethyl phenyl selenide in 76% yield (18.16 g) (b.p. 66–72 °C/1 Torr; lit. value [8], 66–67 °C/1 Torr). Analysis: Calc. for $C_8H_7F_3Se$ (mol. wt. 239.09): C, 40.19; H, 2.95; F, 23.84; Se, 33.02%. Found: C, 40.21; H, 2.93; F, 23.51; Se, 32.98%. IR ($cm^{-1}$): 3080 (m); 3060 (m); 3020 (w); 2960 (m); 1580 (m); 1480 (m); 1440 (m); 1410 (m); 1290 (s); 1260 (s); 1225 (s); 1110 (s b); 1055 (s); 1020 (m); 1000 (m); 830 (w); 730 (w); 690 (s); 630 (m); 470 (m). MS *m/z*: 240 (100) $[C_6H_5SeCH_2CF_3]^+$; 157 (91.10) $[C_6H_5Se]^+$; 94 (44.44) $[SeCH_2]^+$; 91 (35.55) $[C_6H_5CH_2]^+$; 83 (6.66) $[CH_2CF_3]^+$; 69 (15.55) $[CF_3]^+$.

### *Preparation of p-$CH_3C_6H_4SeCH_2CF_3$ (1b)*

Compound **1b** was prepared in the same manner as **1a** using 17.0 g (0.05 mol) of $(p\text{-}CH_3C_6H_4)_2Se_2$. Yield 20.24 g (80%), b.p. 41 °C/0.2 Torr. Analysis: Calc. for $C_9H_9F_3Se$ (mol. wt. 253.11): C, 42.71; H, 3.58; F, 22.52; Se, 31.19%. Found: C, 42.69; H, 3.59; F, 22.40; Se, 31.17%. IR ($cm^{-1}$): 3010 (m); 2950 (m); 2920 (m); 2860 (w); 1490 (s); 1400 (s); 1280 (s b); 1260 (s b); 1215 (m); 1115 (s); 1010 (w); 830 (m); 800 (s); 69

TABLE 1. $^1H$ and $^{19}F$ NMR data[a] for phenyl-2,2,2-trifluoroethyl selenide and its derivatives[b]

| Compound | $H_\alpha$ | $(H_\beta+H_\gamma)$ | $CH_2CF_3$ | $CH_2CF_3$ | $OOCCF_3$ |
|---|---|---|---|---|---|
| $C_6H_5SeCH_2CF_3$ (**1a**) | 7.51 | 7.21 | 3.09 | −64.80 | – |
| | (m, 2H) | (m, 3H) | (q, 2H) | (t) | |
| $C_6H_5Se(Cl)_2CH_2CF_3$ (**2a**) | 8.16 | 7.75 | 4.85 | −57.59 | – |
| | (m, 2H) | (m, 3H) | (q, 2H) | (t) | |
| $C_6H_5Se(Br)_2CH_2CF_3$ (**3a**) | 8.03 | 7.69 | 4.46 | −58.25 | – |
| | (m, 2H) | (m, 3H) | (q, 2H) | (t) | |
| $C_6H_5Se(O)CH_2CF_3$ (**4a**) | 7.95 | 7.75 | 3.67 | −59.1 | – |
| | (m, 2H) | (m, 3H) | (q, 2H) | (t) | |
| $C_6H_5Se(O_2CCF_3)_2CH_2CF_3$ (**5a**) | 7.75 | 7.59 | 4.46 | −60.66 | −75.94 |
| | (m, 2H) | (m, 3H) | (q, 2H) | (t) | (s) |

[a]All chemical shifts in ppm.

[b]

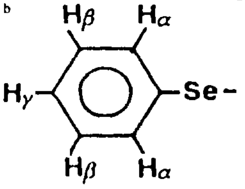

TABLE 2. $^1H$ and $^{19}F$ NMR data[a] for $p$-tolyl-2,2,2-trifluoroethyl selenide and its derivatives[b]

| Compound | $H_\alpha$ | $H_\beta$ | $CH_2CF_3$ | $CH_3$ | $CH_2CF_3$ | $OOCCF_3$ |
|---|---|---|---|---|---|---|
| $p$-$CH_3C_6H_4SeCH_2CF_3$ (**1b**) | 7.56 | 7.10 | 3.19 | 2.33 | −65.61 | – |
| | (d, 2H) | (d, 2H) | (q, 2H) | (s, 3H) | (t) | |
| $p$-$CH_3C_6H_4Se(Cl)_2CH_2CF_3$ (**2b**) | 8.09 | 7.53 | 4.75 | 2.40 | −57.60 | – |
| | (d, 2H) | (d, 2H) | (q, 2H) | (s, 3H) | (t) | |
| $p$-$CH_3C_6H_4Se(Br)_2CH_2CF_3$ (**3b**) | 7.79 | 7.33 | 4.08 | 2.44 | −60.66 | – |
| | (d, 2H) | (d, 2H) | (q, 2H) | (s, 3H) | (t) | |
| $p$-$CH_3C_6H_4Se(O)CH_2CF_3$ (**4b**) | 7.79 | 7.49 | 3.56 | 2.52 | −58.90 | – |
| | (d, 2H) | (d, 2H) | (q, 2H) | (s, 3H) | (t) | |
| $p$-$CH_3C_6H_4Se(O_2CCF_3)_2CH_2CF_3$ (**5b**) | 7.79 | 7.49 | 4.08 | 2.41 | −60.33 | −76.93 |
| | (d, 2H) | (d, 2H) | (q, 2H) | (s, 3H) | (t) | (s) |

[a]All chemical shifts in ppm.
[b]As for Table 1.

(m); 630 (s); 480 (s). MS *m/z*: 254 (57.56) $[CH_3C_6H_4SeCH_2CF_3]^+$; 171 (54.54) $[CH_3C_6H_4Se]^+$; 105 (9.09) $[CH_3C_6H_4CH_2]^+$; 91 (55.30) $[CH_3C_6H_4]^+$; 64 (100) $[CH_2CF_2]^+$.

### *Preparation of $C_6H_5Se(Cl)_2CH_2CF_3$ (2a)*

Chlorine gas was passed through a solution of 0.5 g of $C_6H_5SeCH_2CF_3$ in dry carbon tetrachloride. The solution turned dark brown initially and finally acquired the pale yellow colour of chlorine gas, with the separation of the title compound. The compound was separated and recrystallised from dichloromethane. Yield 0.584 g (90%), m.p. 89 °C (decomp.). Analysis: Calc. for $C_8H_7F_3Cl_2Se$ (mol. wt. 309.99): C, 31.00; H, 2.28; F, 18.39; Cl, 22.87; Se, 25.47%. Found: C, 31.15; H, 2.24; F, 18.43; Cl, 22.85; Se, 25.50%. IR ($cm^{-1}$): 1300 (m); 1270 (w); 1125 (m); 1079 (w); 750 (m b). MS *m/z*: 310 (1.54) $[C_6H_5Se(Cl)_2CH_2CF_3]^+$; 275 (35.90) $[C_6H_5Se(Cl)CH_2CF_3]^+$; 240 (100) $[C_6H_5SeCH_2CF_3]^+$; 192 (14.55) $[C_6H_5SeCl]^+$; 171 (12.72) $[C_6H_5SeCH_2]^+$; 77 (30.00) $[C_6H_5]^+$.

### *Preparation of p-$CH_3C_6H_4Se(Cl)_2CH_2CF_3$ (2b)*

Compound **2b** was prepared in essentially the same way as **2a** using $p$-$CH_3C_6H_4SeCH_2CF_3$ (2.53 g, 0.01 mol). Yield 2.98 g (92%), m.p. 114 °C (decomp.). Analysis: Calc. for $C_9H_9F_3Cl_2Se$ (mol. wt. 324.02): C, 33.36; H, 2.80; F, 17.59; Cl, 21.88; Se, 24.37%. Found: C, 33.21; H, 2.85; F, 17.60; Cl, 21.87; Se, 24.39%. IR ($cm^{-1}$): 1470 (s); 1382 (m); 1295 (s); 1270 (m); 1230 (m); 1120 (s); 1070 (m); 810 (w); 650 (w); 630 (w); 490 (w). MS *m/z*: 289 (3.29) $[CH_3C_6H_4Se(Cl)CH_2CF_3]^+$;

254 (81.54) $[CH_3C_6H_4SeCH_2CF_3]^+$; 171 (100) $[CH_3C_6H_4Se]^+$; 105 (8.49) $[CH_3C_6H_4CH_2]^+$; 91 (88.90) $[CH_3C_6H_4]^+$; 69 (3.81) $[CF_3]^+$; 64 (49.36) $[CH_2CF_2]^+$.

*Preparation of* $C_6H_5Se(Br)_2CH_2CF_3$ *(3a)*

$C_6H_5SeCH_2CF_3$ (1.2 g, 0.005 mol) was reacted with liquid bromine (0.8 g, 0.005 mol) at 0 °C in dichloromethane and the contents brought to room temperature. The product was recrystallised from dry petroleum ether. Yield 1.80 g (90%), m.p. 86 °C (decomp.). Analysis: Calc. for $C_8H_7F_3Br_2Se$ (mol. wt. 398.90): C, 24.09; H, 1.77; F, 14.29; Se, 19.79; Br, 40.06%. Found: C, 23.99; H, 1.71; F, 14.17; Se, 19.68; Br, 40.12%. IR ($cm^{-1}$): 3040 (m); 2970 (m); 2950 (m); 1490 (m); 1455 (m); 1295 (m); 1275 (m); 1150 (s); 1060 (m); 740 (m); 680 (w); 650 (w); 470 (w).

*Preparation of* p-$CH_3C_6H_4Se(Br)_2CH_2CF_3$ *(3b)*

Compound **3b** was prepared from *p*-$CH_3C_6H_4SeCH_2CF_3$ (1.26 g, 0.005 mol) by the same procedure as for **3a**. Yield 1.79 g (87%), m.p. 85 °C (dec.). Analysis: Calc. for $C_9H_9F_3Br_2Se$ (mol. wt. 412.93): C, 26.18; H, 2.20; F, 13.80; Se, 19.12; Br, 38.70%. Found: C, 26.09; H, 2.16; F, 13.85; Se, 19.11; Br, 38.69%. IR ($cm^{-1}$): 1465 (vs); 1380 (s); 1292 (s); 1267(s); 1230 (m); 1118 (m); 1065 (m); 805 (m); 730 (w); 635 (w); 490 (m). MS *m*/*z*: 254 (88.20) $[CH_3C_6H_4SeCH_2CF_3]^+$; 171 (100) $[CH_3C_6H_4Se]^+$; 160 (21.37) $[Br_2]^+$; 105 (9.71) $[CH_3C_6H_4CH_2]^+$; 91 (88.27) $[CH_3C_6H_4]^+$; 64 (26.31) $[CH_2CF_2]^+$.

*Preparation of* $C_6H_5Se(O)CH_2CF_3$ *(4a)*
*Method A*

$C_6H_5Se(Cl)_2CH_2CF_3$ (1.55 g, 0.005 mol) was dissolved in 40 ml of dichloromethane and stirred at 0 °C with 5% sodium hydroxide solution for 1 h. The organic layer was separated, washed with water, and dried over anhydrous sodium sulphate. Evaporation of the solvent *in vacuo* produced a white fluffy powder which was recrystallized from benzene. Yield 0.83 g (65%), m.p. 91 °C.

*Method B*

In a typical preparation, $C_6H_5SeCH_2CF_3$ (1.19 g, 0.005 mol) was placed in a round-bottom flask maintained at −5 °C to which 30% $H_2O_2$ (1.25 ml) was added dropwise with vigorous stirring over 1.5 h. After the removal of volatile components *in vacuo*, compound **4a** was obtained as a white fluffy powder which was recrystallized from benzene. Yield 0.914 g (72%), m.p 91 °C. Analysis: Calc. for $C_8H_7OF_3Se$ (mol. wt. 255.09): C, 37.67; H, 2.77; F, 22.34; Se, 30.95%. Found: C, 37.71; H, 2.69; F, 22.43; Se, 30.86%. IR ($cm^{-1}$): 1455 (s); 1440 (s); 1370 (m); 1290 (m); 1250 (m); 1210 (m); 1120 (m); 1110 (m); 815 (m); 740 (m); 685 (m); 635 (m); 455 (m). MS *m*/*z*: 256 (11.00) $[C_6H_5Se(O)CH_2CF_3]^+$; 240 (65.14) $[C_6H_5SeCH_2CF_3]^+$; 157 (100) $[C_6H_5Se]^+$.

*Preparation of* p-$CH_3C_6H_4Se(O)CH_2CF_3$ *(4b)*

Compound **4b** was prepared in ~70% yield in a manner similar to that used for **4a** employing 1.62 g (0.005 mol) of *p*-$CH_3C_6H_4Se(Cl)_2CH_2CF_3$ (Method A) or 1.265 g (0.005 mol) of *p*-$CH_3C_6H_4SeCH_2CF_3$ (Method B). Analysis: Calc. for $C_9H_9OF_3Se$ (mol. wt. 269.12): C, 40.17; H, 3.37; F, 21.18; Se, 29.34%. Found: C, 40.21; H, 3.14; F, 21.23; Se, 29.40%. IR ($cm^{-1}$): 1460 (vs); 1382 (vs); 1294 (s); 1265 (s); 1208 (m); 1125 (s); 1045 (m); 980 (w); 810 (s); 730 (w); 640 (w); 485 (m). MS *m*/*z*: 270 (3.38) $[CH_3C_6H_4Se(O)CH_2CF_3]^+$; 254 (27.19) $[CH_3C_6H_4SeCH_2CF_3]^+$; 187 (25.36) $[CH_3C_6H_4SeO]^+$; 171 (33.79) $[CH_3C_6H_4Se]^+$; 107 (100) $[CH_3C_6H_4O]^+$; 91 (44.19) $[CH_3C_6H_4]^+$.

*Preparation of* $C_6H_5Se(OOCCF_3)_2CH_2CF_3$ *(5a)*

$C_6H_5Se(O)CH_2CF_3$ (1.28 g, 0.005 mol) was placed in a 50 ml reaction vessel and $(CF_3CO)_2O$ (0.84 ml, 0.006 mol) was condensed on to it at −196 °C. The contents were then allowed to come to room temperature. Excess of trifluoroacetic anhydride was removed *in vacuo* when **5a** was obtained as a white fluffy powder in quantitative yield, m.p. 35 °C (dec.). Analysis: Calc. for $C_{12}H_7O_4F_9Se$ (mol. wt. 465.11): C, 30.97; H, 1.51; F, 36.77; Se, 16.98%. Found: C, 30.88; H, 1.48; F, 36.72; Se, 16.92%. IR ($cm^{-1}$): 3055 (s); 2980 (s); 1745 (s b); 1480 (s); 1450 (s); 1443 (m); 1410 (s); 1380 (s); 1335 (s); 1290 (s b); 1250 (s b); 1000 (m); 912 (m); 850 (m); 790 (s); 640 (m); 610 (m); 520 (w); 470 (m).

*Preparation of* p-$CH_3C_6H_4Se(OOCCF_3)_2CH_2CF_3$ *(5b)*

Compound **5b** was prepared from **4b** (1.35 g, 0.005 mol) in the same way as **5a** in quantitative yield. m.p. 40 °C (dec.). Analysis: Calc. for $C_{13}H_9O_4F_9Se$ (mol. wt. 479.14): C, 32.59; H, 1.89; F, 35.69; Se, 16.48%. Found: C, 32.67; H, 1.85; F, 35.70; Se, 16.43%. IR ($cm^{-1}$): 3055 (s); 2990 (s); 1750 (s b); 1490 (s); 1440 (s); 1390 (s b); 1300 (s b); 1260 (s b); 1200 (s b); 1160 (s b); 1010 (m); 850 (s); 800 (s); 790 (s); 730 (vs); 645 (s); 610 (s); 490 (s b).

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