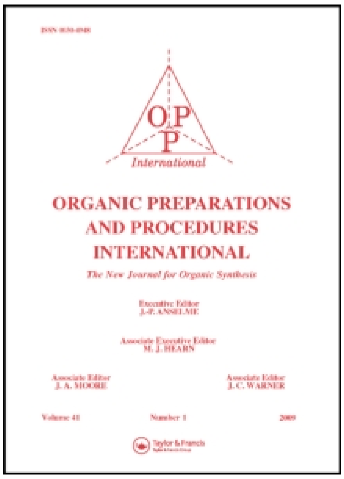

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PREPARATION OF bis(2,2,2-TRIFLUOROETHYL)SELENIDE AND TELLURIDE AND OF BIS(2,2,2-TRIFLUOROETHYL)DISELENIDE AND DITELLURIDE

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PREPARATION OF bis(2,2,2-TRIFLUOROETHYL)SELENIDE AND TELLURIDE AND OF bis(2,2,2-TRIFLUOROETHYL)DISELENIDE AND DITELLURIDE

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Organoselenium and organotellurium compounds are useful synthons in organic synthesis.[1] Dialkyl tellurides find applications as precursors for the growth of the semiconductor cadmium mercury telluride (CMT) by MOCVD, useful in infrared detectors and related devices.[2] Diorganyl sulphides and selenides containing 2,2,2-trifluoroethyl group have successfully been used for the synthesis of thioynamines and selenoynamines respectively.[3] We therefore sought to develop a convenient method for the preparation of new organoselenium and organotellurium compounds containing 2,2,2-trifluoroethyl groups.

bis(2,2,2-Trifluoroethyl)selenide (**1**), telluride (**2**), diselenide (**3**) and ditelluride (**4**) have been prepared for the first time by reaction of 1-iodo-2,2,2-trifluoroethane with sodium selenide or telluride

$$\mathrm{nE} + 2\mathrm{Na} \xrightarrow{\text{liq. NH}_3} \mathrm{Na_2E_n}$$

$$\mathrm{Na_2E_n} + 2\mathrm{CF_3CH_2I} \longrightarrow \mathrm{(CF_3CH_2)_2E_n} + 2\mathrm{NaI}$$

(E = Se or Te; n = 1 or 2)

and diselenide or ditelluride, generated from sodium and elemental selenium or tellurium in 2:1 and 1:1 ratios, respectively, in liquid ammonia.

EXPERIMENTAL SECTION

All reactions were carried out under dry nitrogen to prevent the oxidation of oxygen sensitive selenide and telluride ions. All bps are uncorrected. Infrared spectra were obtained on a Perkin Elmer 1430 spectrometer. ^{1}H NMR spectra were recorded in CCl_4 using TMS as an internal standard on Varian EM 390L spectrometer, while ^{19}F NMR spectra were determined on a Joel FX 90Q FT NMR spectrometer in CCl_4 using $CFCl_3$ as an external standard.

bis(2,2,2-Trifluoroethyl)selenide and Telluride (1 and 2).- Sodium (0.23 g, 10 mmol) was added in small portions to a well stirred suspension of elemental selenium or tellurium (5 mmol) in anhydrous liquid ammonia (50 mL) at -40°. The reaction mixture was stirred vigorously for 2 hrs and 1-iodo-2,2,2-trifluoroethane (2.10 g, 10 mmol) was added dropwise over a period of 30 min with stirring. The ammonia was then allowed to boil off slowly and the residue was hydrolyzed with water (25 mL) at room temperature and extracted with dichloromethane (3 x 25 mL). The combined organic layers were washed with water (3 x 25 mL), dried over sodium sulfate for 6 hrs and filtered. The solvent was stripped off and the residue was distilled under reduced pressure to afford the title compounds.

$(CF_3CH_2)_2Se$ (**1**), bp. 95°/760 torr, yield 1.12 g (91%).

Anal. Calcd. for $C_4H_4F_6Se$: C, 19.59; H, 1.63; Se, 32.24. Found: C, 19.50; H, 1.62; Se, 32.08

IR (KBr): 3020 (w) , 2960 (m) , 2875 (m) , 1460 (m) , 1405 (m) , 1385 (s), 1280 (s), 1265 (s), 1135 (s), 1060 (s), 740 (w), 700 (w), 642 (m) cm^{-1}; ^{1}H NMR: δ 3.24 (q, 4H, CH_2); ^{19}F NMR: δ -66.66 (t, 6F, CF_3).

$(CF_3CH_2)_2Te$ (**2**), bp. 40°/15 torr, yield 1.32 g (90%).

Anal. Calcd. for $C_4H_4F_6Te$: C, 16.35; H, 1.36; Te, 43.46 Found: C, 16.30; H, 1.35; Te, 43.40

IR (KBr): 3000 (s), 1480 (s), 1420 (s), 1380 (s), 1290 (s), 1260 (s), 1205 (s), 1110 (s), 1065 (s), 1040 (s), 820 (s), 800 (w), 670 (s), 640 (m), 540 (w), 515 (w) cm^{-1}; ^{1}H NMR: δ 3.38 (q, 4H, CH_2); ^{19}F NMR: δ -61.50 (t, 6F, CF_3).

bis(2,2,2-Trifluoroethyl)diselenide and Ditelluride (3 and 4).- Sodium (0.23 g, 10 mmol) was added in small portions to a well stirred suspension of elemental selenium or tellurium (10 mmol) in anhydrous liquid ammonia (50 mL) at -40°. The reaction mixture was stirred vigorously for 3 hrs and 1-iodo-2,2,2-trifluoroethane (2.10 g, 10 mmol) was added dropwise over a period of 30 min with stirring. The ammonia was then allowed to evaporate slowly and the residue hydrolyzed with water (25 mL) at room temperature and extracted with dichloromethane (3 x 25 mL), The combined organic layers were washed with water (3 x 25 mL) and dried over anhydrous sodium sulfate for 6 hrs. The solvent was stripped off and the residue was distilled under reduced pressure to afford the title compounds.

$(CF_3CH_2)_2Se_2$ (**3**), bp. 50°/6 torr, yield 1.30g (80%).

Anal. Calcd. for $C_4H_4F_6Se_2$: C, 14.81; H, 1.23 ; Se, 48.76. Found: C, 14.65; H, 1.22; Te, 48.58

IR (KBr): 3000 (m), 2940 (w), 2840 (s), 1460 (m), 1410 (s), 1375 (s), 1290 (s), 1260 (s), 1215 (s), 1120 (s), 1044 (s), 760 (w), 700 (s), 640 (m) cm^{-1}; ^{1}H NMR: δ 3.60 (q, 4H, CH_2) ; ^{19}F NMR: δ -64.50 (t, 6F, CF_3).

$(CF_3CH_2)_2Te_2$ (4), bp. 60°/2 torr, yield 1.90 g (90%).

Anal. Calcd. for $C_4H_4F_6Te_2$: C, 11.40; H, 0.95; Te, 60.59. Found: C, 11.40; H, 0.92; Te, 60.39

IR (KBr): 2980 (s), 2940 (s), 2880 (s), 1470 (s), 1415 (s), 1370 (s), 1275 (s), 1255 (s), 1200 (s), 1100 (s), 1040 (s), 780 (w), 650 (sh), 630 (s), 508 (w) cm^{-1}; ^{1}H NMR: δ 4.10 (q, 4H, CH_2); ^{19}F NMR: δ -66.00 (t, 6F, CF_3).

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SYNTHESIS OF PENTAFLUOROPHENYL-4-(*N*-MALEIMIDOMETHYL) CYCLOHEXANE-1-CARBOXYLATE (FMCC)

Submitted by (03/11/93)
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Current research in our laboratory necessitated the synthesis of succinimidyl-4-(*N*-maleimidomethyl)cyclohexane-1-carboxylate (SMCC), or an equivalent analogue. SMCC is a heterobifunctional cross-linking reagent with a maleimido functionality linked to a succinimidyl active ester and has been used extensively in a variety of recent biotechnological endeavors. For example, SMCC has been used to couple enzymes to antibodies for the development of immunoassays,[1] linkage of toxins to antibodies or cell-specific protein ligands for targeted delivery of therapeutic agents,[2] and coupling of radiolabels to antibodies for tumor imaging.[3] Unfortunately, our attempts to synthesize