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COMMUNICATION

SYNTHESIS OF *cis*-$(Me_3Sn)_2Fe(CO)_4$ BY REACTION OF $(Me_3Sn)_3Sb$ WITH $Fe_2(CO)_9$. FORMATION OF $(Me_3E)_3SbFe(CO)_4$ (E = Si, Ge)

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Abstract—$(Me_3Sn)_3Sb$ and $Fe_2(CO)_9$ reacted to form $(Me_3Sn)_2Fe(CO)_4$ in 79% yield. From $(Me_3E)_3Sb$(E = Si or Ge) and $Fe_2(CO)_9$ the complexes $(Me_3E)_3SbFe(CO)_4$ were obtained

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Reactions of tris(trimethylsilyl-, -germyl-, and -stannyl)- stibines with oxygen,[1] alkylhalides,[2] and halogens,[3] result in the transfer of the organoelement (IVB) substituents to the non-metal atoms. In contrast the organoelement(IVB)stibine molecule remains intact, when it reacts with carbonyl complexes of the metals Cr, Mo, W, Ni, and coordination compounds of the type $(Me_3E)_3Sb$-$M(CO)_n$ (E = Si, Ge, Sn; M = Cr, Mo, W; $n = 5$; M = Ni, $n = 3$)[4] are obtained.

We report now the first example of a organoelement(IVB) group migration from antimony to a transition metal.

Equimolar amounts of $(Me_3Sn)_3Sb$ and $Fe_2(CO)_9$ react in *n*-pentane at room temperature to form *cis*-$(Me_3Sn)_2Fe(CO)_4$[5] in 80% yield, together with $Fe(CO)_5$ and a dark precipitate, which contains antimony.

The first step of this reaction, where $(Me_3Sn)_3Sb$ serves as stannylating agent, is presumably the formation of $(Me_3Sn)_3SbFe(CO)_4$. This assumption is supported by the reactions of $(Me_3Si)_3Sb$ and $(Me_3Ge)_3Sb$ with $Fe_2(CO)_9$, which result in the formation of the derivatives $(Me_3E)_3SbFe(CO)_4$ (E = Si or Ge) as colourless crystals, soluble in common organic solvents. These iron complexes are thermally much less stable, than analogous compounds of VI A metals or Ni.[4] They decompose even at −25°C with formation of dark solids and they ignite spontaneously in the air. Structural evidence is based mainly on the ir spectra The frequencies in the ν(CO) region are typical for the axial isomers of $LFe(CO)_4$ complexes.[6] The observation of the characteristic absorptions of the $(Me_3E)_3Sb$ skeleton including the E_3Sb valence vibrations proves, that the ligands are coordinated without fragmentation. The ^{1}H-NMR spectra show the expected singlet signal for each complex at positions which, as in other organoelementstibine carbonyl complexes,[4] are close to the resonance frequencies of the free ligands.

EXPERIMENTAL

Reactions are carried out in Ar atmosphere. *cis*-Tetracarbonylbis(trimethylstannyl)iron 6.13 g (10 mmol) $(Me_3Sn)_3Sb$ and 3.64 g (10 mmol) $Fe_2(CO)_9$ were stirred in 50 cm^3 *n*-pentane for 12 hr. After filtration the solvent was removed *in vacuo* leaving 3.6 g of $(Me_3Sn)_2Fe(CO)_4$. ^{1}H-NMR (C_6H_6): $\delta = 0.45$ ppm, $^1J_{HC}$ $117/119_{Sn} = 50$ Hz. IR (Nujol): 2056, 1996, 1985, 1974 cm^{-1} (Lit.[7]: identical data). MS (EI, 30 eV, 25°C) m/z (r.i.%): 481 (10) $M^+ - Me$, 453 (2) $M^+ - Me - CO$, 452 (2), 397 (2), 369 (2), 318 (100)$M^+ - Me_4Sn$, 290 (30), 262 (8), 234 (3), 206 (7), 165 (3) Me_3Sn; only the most intense signal of each ion is given, isotopic abundances agreed with those expected. $C_{10}H_{18}FeO_4Sn_2$ (495.5). Found: C, 24.24 (24.22); H, 3.59 (3.66)%.

Tetracarbonyl(tristrimethylsilylstibine)iron 3.4 g (10 mmol) $(Me_3Si)_3Sb$ and 3.6 g (10 mmol) $Fe_2(CO)_9$ were stirred in 50 cm^3 *n*-pentane for 12 hr. Work up as described above and recrystallization at −25°C from pentane gives 1.5 g (30%) of $(Me_3Si)_3SbFe(CO)_4$. ^{1}H-NMR (C_6H_6) $\delta = 0.26$ ppm (S). IR (Nujol): 2040 st, 1945 w, 1910 m ν(CO), 318 m νSi_3Sb, cm^{-1}.

Tetracarbonyl(tristrimethylgermylstibine) iron 0.7 g (1.5 mmol) $(Me_3Ge)_3Sb$ and 0.5 g (1.5 mmol) $Fe_2(CO)_9$ were stirred in 20 cm^3 *n*-pentane for 12 hr. Filtration and crystallization gives 0.15 g (15%) $(Me_3Ge)_3SbFe(CO)_4$. ^{1}H-NMR (C_6H_6)

$\delta = 0.35$ ppm (S). IR (Nujol): 2040 st, 1950 w, 1915 m ν(CO), 230 w νGe_3Sb; cm^{-1}.

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