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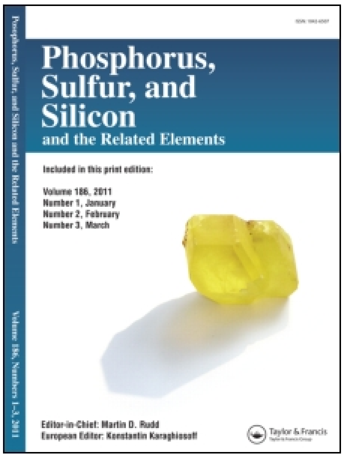

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Michael Baum; Hans Bock; Alois Haas; Zdenek Havlas; Christian Monse; Bahman Solouki

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SYNTHESIS OF NEW PERFLUORINATED TELLURACARBONYLS AND 1,3-DITELLURETANES

MICHAEL BAUM[a], HANS BOCK[b], ALOIS HAAS[a], ZDENEK HAVLAS[c], CHRISTIAN MONSÉ[a], BAHMAN SOLOUKI[b]
[a] Ruhr-University Bochum, Lehrstuhl für Anorg. Chemie II, D-44780 Bochum, Germany; [b] University Frankfurt, Institut für Anorganische Chemie, D-60539 Frankfurt, Germany; [c] Czech Academy of Science, Institute of Org. Chemistry, 16610 Prague 6, Czech Republic

Abstract:
With the preparation of $C_2F_5(F)C{=}Te$, the third, non-resonance stabilised perfluorinated telluracarbonyl has been isolated and characterised by mass spectrometry. It was prepared *via* pyrolysis of the novel compound $Me_3SnTe(n\text{-}C_3F_7)$ at 550 °C, 10^{-3} Torr in 45% yield.The compound $C_2F_5(F)C{=}Te$ is only stable at -196 °C and dimerises quantitatively a few degrees above this temperature to the corresponding mixture of *cis/trans*-2,4-bis(pentafluoroethyl)-2,4-difluoro-1,3-ditelluretane.

Keywords: Telluracarbonyls / 1,3-Ditelluretanes / Bis(perfluoroalkyl)ditellurides / Trimethylstannylperfluoroalkyltellurides

INTRODUCTION:

The required precursors for the synthesis of the perfluorinated telluracarbonyls $Te(R_f)_2$ and $Te_2(R_f)_2$ were prepared by three different methods[1]:

Method:

a $Hg(R_f)_2 + 3\,Te \xrightarrow{i} (R_f)Te(R_f) + (R_f)TeTe(R_f)$

b $Hg(C_2F_5)I + 3\,Te \xrightarrow{i} C_2F_5TeC_2F_5 + C_2F_5TeTeC_2F_5$

c $(R_f)I + 3\,Te + 3\,Cu \longrightarrow (R_f)Te(R_f)\ \underline{1} + (R_f)TeTe(R_f)\ \underline{2}$

Scheme 1 (*i*) Catalytic amounts of Cu;
1a: $R_f = n\text{-}C_3F_7$ (43%); **1b**: $R_f = n\text{-}C_4F_9$ (33%)

The preparation of the novel ditellurides **2a** and **2b** introduces two important precursors for the synthesis of new telluracarbonyls[2].

$$\underset{\mathbf{1a/1b}}{Te(R_f)_2} \xrightarrow[\text{furane}]{h\nu} Te_2(R_f)_2 + \text{furane-derivatives}$$

$R_f = CF_3CF_2CF_2$ (12 h) **2a**, 55%

$n\text{-}C_4F_9$ (12 h) **2b**, 48%

Scheme 2

Transformation of compound **1a/1b** or **2a/2b** into the required trimethylstannyl perfluoroalkyl tellurides **3** and **4** is accomplished by treating trimethylstannane, Me_3SnH.

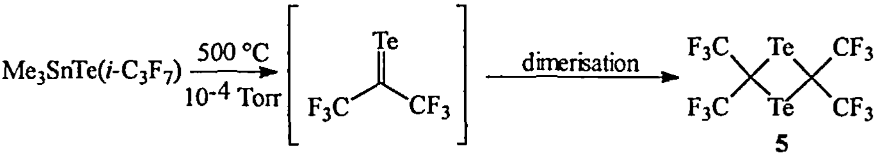
$$(R_f)Te(R_f) + Me_3SnH \xrightarrow{(C_2H_5)_2O} Me_3SnTe(R_f) + (R_f)H$$

$R_f = CF_3CF_2CF_2$ **3**, 45%

$n\text{-}C_4F_9$ **4**, 40%

Scheme 3

Pyrolysis of $Me_3SnTe(i\text{-}C_3F_7)$ leads to the novel 1,3-ditelluretane **5**. The monomeric hexafluortelluraacetone cannot be observed.



Scheme 4

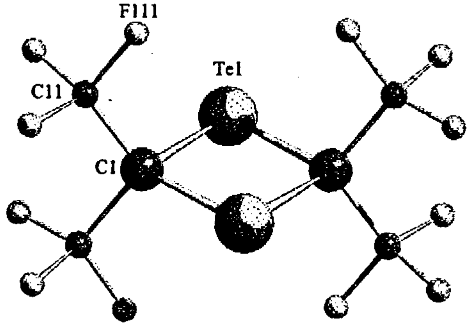
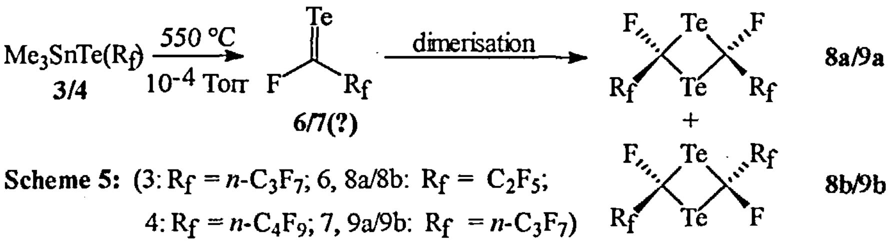


Figure 1: X-ray structure of $[TeC(CF_3)_2]_2$ 5 (red crystals)
(See Colour Plate at back of issue)

The pyrolysis of $Me_3SnTe(R_f)$ (**3/4**) is the suitable reaction for the synthesis of new perfluorinated telluracarbonyls.



Scheme 5: (3: $R_f = n\text{-}C_3F_7$; 6, 8a/8b: $R_f = C_2F_5$;
4: $R_f = n\text{-}C_4F_9$; 7, 9a/9b: $R_f = n\text{-}C_3F_7$)

During the pyrolysis of compound **3**, the pyrolysis apparatus was coupled to the gas inlet of a mass spectrometer, the mass spectra of pure **6** was obtained. **6** was trapped at -196 °C with green colour. A few degrees above this temperature **6** dimerises almost quantitatively to the cis-/trans-mixture of red **8a/8b**. The mixtures of the ditelluretanes cannot be seperated by physical methods, including preparative gas chromatography. All analyses were obtained on mixtures of **8a/8b** and **9a/9b** which are a violet liquids at 22 °C. IR-spectras and a mass spectra of **7**, which is also of green colour at -196 °C, could not be measured yet.

Table 1: mass spectra of 6

fragment	m/z (%)
M^+	280 (5)
$TeCF^+$	161 (3,5)
Te^+	130 (7)
CF_3^+	69 (23)
N_2^+	28 (100)

Structural aspects:

PE-spectra were recorded for the following compounds: $TeCF_2$, $[TeCF_2]_2$, $TeC(F)CF_3$ and $[TeC(F)CF_3]_2$. The analysis of the spectra was carried out by comparison with calculated data and as far as available with spectra of the corresponding thio- and seleno-compounds[3]. Furthermore structures of the compounds have been determined by calculations. These calculations are directly connected with orbital energies. Therefore the calculated structures can be compared with x-ray data. Two different ab-initio-methods are used for the calculations (HF = hypothetical function; GF = green function), which are based on the program "Gaussian 94: NEC-SX-4-Super-Ux-G94", version D3. In consideration of the "Koopman-rule" PE-spectra and calculated orbital energies are comparable. The detailed results will be published soon.

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