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Tris(trifluoromethyl)tellurate(II), $[Te(CF_3)_3]^-$, the dimeric bis(trifluoromethyl)chlorotellurate(II), $[(CF_3)_2TeCl]_2^{2-}$, and bis(trifluoromethyl)iodate(I), $[I(CF_3)_2]^-$: the first hypervalent alkyl [10-Te-3] and [12-Te-4] species and a novel [10-I-2] compound

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Dedicated to Professor Karl O. Christe on the occasion of his 65th birthday

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Abstract

The reactions of $(CH_3)_3SiCF_3/[(CH_3)_4N]F$ with $Te(CF_3)_2$ and CF_3I, respectively in THF or glyme at −60 °C yield $[(CH_3)_4N][Te(CF_3)_3]$ and $[(CH_3)_4N][I(CF_3)_2]$. Both salts were isolated as colorless and extremely reactive solids. The NMR spectra are discussed; the $^1J_{F,C}$ coupling constants of the CF_3 groups of the linear CF_3–Te–CF_3 and CF_3–I–CF_3 units exhibit extremely high values. Under comparable conditions and also in CH_2Cl_2 at ambient temperature, salts with the anion $[(CF_3)_2TeCl]^-$ are formed from the reactions $Te(CF_3)_2$, $[TAS][(CH_3)_3SiF_2]/(CH_3)_3SiCl$ and [PNP]Cl, respectively. The crystal structure (triclinic; *P*-1) of $[TAS]_2[(CF_3)_2Te(\mu\text{-}Cl)_2Te(CF_3)_2]$ exhibits dimeric chloro-bridged Te–Cl units. © 2001 Published by Elsevier Science B.V.

Keywords: Tellurium; Iodine; Trifluoromethyltellurates(II); Trifluoromethyliodate(I); NMR; Crystal structure

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1. Introduction

Nucleophilic trifluoromethylation reactions with $(CH_3)_3SiCF_3$ are activated by addition of fluoride ions [1]. Recently, we could prove that at very low temperature $[(CH_3)_3Si(CF_3)F]^-$ and $[(CH_3)_3Si(CF_3)_2]^-$ are formed as reactive intermediates, which undergo fast nucleophilic trifluoromethylations even at very mild conditions [2]. Thus, we prepared $[(CH_3)_4N][Bi(CF_3)_4]$ as a novel hypervalent alkyl bismuth compound reacting $Bi(CF_3)_3$, $(CH_3)_3SiCF_3$ and $[(CH_3)_4N]F$ at low temperature [3].

Herein we report on results of the reactions of $(CH_3)_3SiCF_3/[(CH_3)_4N]F$ with $Te(CF_3)_2$ and CF_3I, respectively. While perfluoroalkyl and aryl [10-I-2] species are known for more than a decade [4], [10-Te-3] species are limited to triaryltellurates. $Li[TeAr_3]$ ($Ar = C_6H_5$, $4\text{-}CH_3C_6H_4$) [5,6] and $Li[Te(C_6H_5)(C_6F_5)Ar]$ ($Ar = C_6F_5$, $C_6F_3Cl_2$) [7] were identified in solution by NMR spectroscopy. $Te(CF_3)_2$ [8] and CF_3I [9,10] show weak Lewis acid properties which are demonstrated by the dependence of the ^{19}F NMR chemical shifts on the strength of the coordinating donor. Therefore, bases such as CF_3^- or Cl^- should coordinate to both weak Lewis acids.

2. Results and discussion

2.1. Preparation and properties of $[(CH_3)_4)N][Te(CF_3)_3]$ and $[(CH_3)_4N][I(CF_3)_2]$

The novel salts $[(CH_3)_4N][Te(CF_3)_3]$ and $[(CH_3)_4N][I(CF_3)_2]$ were prepared according to the reaction sequence shown in Scheme 1.The salts were isolated as colorless, highly reactive solids. Whereas the iodate(I) decomposes between 0 °C and room temperature, the tellurate(II) is stable up to 110 °C (visible decomposition).

In solution, both compounds decompose above −30 °C to give CF_3H, $Te(CF_3)_2$ and CF_3I, respectively, as the only fluorine containing products.

The NMR data (Table 1) of both anions are remarkable. $[I(CF_3)_2]^-$ is detected in the ^{19}F NMR spectrum as a sharp

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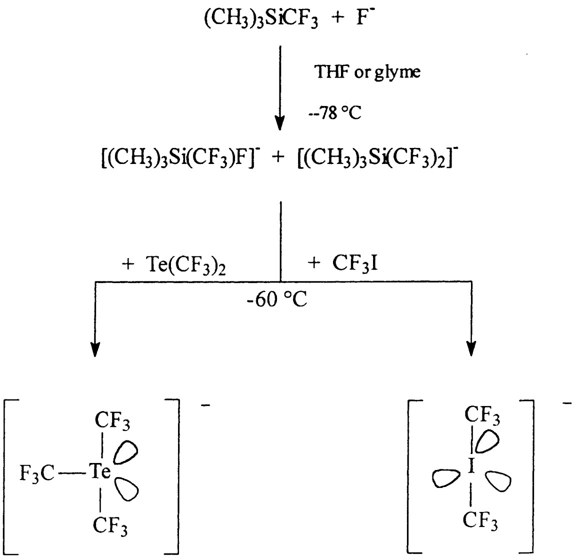

Scheme 1.

singlet at δ -40 ± 2 (-60 °C). It is unsymmetrically surrounded by ^{13}C satellites with absolute values of the couplings $^1J_{CF} = 441 \pm 2$ Hz and $^4J_{FF} = 12 \pm 1$ Hz. In comparison with NMR data of CF_3I in basic solvents [9–11] $[I(CF_3)X]^-$ (X = Cl, Br) [12] and trifluoromethyliodine(III) derivatives [11,13–15] the novel compound shows the greatest upfield shift together with greatest absolute value of the $^1J_{CF}$ coupling. On the basis of structural data for $[I(C_6F_5)_2]^-$ [4] and the VSEPR model, we suggest a linear C–I–C unit in $[I(CF_3)_2]^-$.

Table 1
NMR data of $[N(CH_3)_4][Te(CF_3)_3]$, $[N(CH_3)_4][I(CF_3)_2]$ and $[PNP][Te(CF_3)_2Cl]$

	$[Te(CF_3)_3]^-$	$[I(CF_3)_2]^-$	$[Te(CF_3)_2Cl]^-$
Solvent (temperature)	Glyme (−40 °C)	THF-d_8 (−30 °C)	CH_2Cl_2 (21 °C)
^{19}F (282.35 MHz)			
$\delta(CF_3)_{ax}$	−45.7, q, [a]6F	−41.1, s[b]	
$^1J_{CF}$ (Hz)	415	442	
$\delta(CF_3)_{eq}$	−29.4, sept, [c]3F		−28.2, s[d]
$^1J_{CF}$ (Hz)	354		360
^{13}C (75.45 MHz)[e]			–[f]
$\delta(CF_3)_{ax}$	128.9, qq	117.9, qq	
$^3J_{CF}$ (Hz)	24	67	
$\delta(CF_3)_{eq}$	114.2, q, sept		111.4, qq[g]
$^3J_{CF}$ (Hz)	15		3
^{125}Te (94.79 MHz)			
δ	601, m[h]		1154, sept[i]

[a] $^2J_{TeF} = 230$ Hz, $^4J_{FF} = 6$ Hz.
[b] $^4J_{FF} = 12$ Hz.
[c] $^2J_{TeF} = 111$ Hz, $^4J_{FF} = 6$ Hz.
[d] $^2J_{TeF} = 124$ Hz, $^4J_{FF} = 6$ Hz.
[e] $\delta([N(CH_3)_4]^+)$ 53.2, q, $^1J_{CH} = 143$ Hz.
[f] The signals of the cation are detected in the proton-decoupled spectrum between 135 and 126 ppm as higher order multiplets due to spin–spin interactions with ^{31}P nuclei.
[g] $^1J_{TeC} = 486$ Hz ($^{13}C\{^{19}F\}$).
[h] 14-line multiplet, distance between lines approximately 113 Hz.
[i] $^2J_{TeF} = 124$Hz.

An increase in the absolute value of the $^1J_{CF}$ coupling appears to be general in molecules with linear CF_3–element–CF_3 units (cf. $[(CH_3)_3Si(CF_3)_2]^-$ (378 Hz) and $(CH_3)_3SiCF_3$ (323 Hz) [2]). Also the absolute value of 67 Hz of the $^3J_{CF}$ coupling appears to be noteworthy compared with characteristic values of trifluoromethyl compounds of the elements of the fifth period varying between 2 and 10 Hz [16–18].[1]

Similar effects are found in the NMR spectra of $[Te(CF_3)_3]^-$ (Table 1). On the basis of the ^{19}F NMR spectra (Fig. 1), $[Te(CF_3)_3]^-$ is T-shaped. The axial CF_3 groups are detected at δ -45.7, $^1J_{CF}$ 415 Hz, approximately 20 ppm upfield from $Te(CF_3)_2$ [18], whereas the shift of the equatorial group at δ -29.4 remains nearly unaffected (cf. Fig. 1). The ^{13}C and ^{125}Te NMR spectra support C_{2v} symmetry (ψ-trigonal bipyramidal geometry, AB_3E_2 system) rather than C_{3v} symmetry. The ^{125}Te NMR spectrum exhibits an at least 14-line multiplet -$^2J_{TeF(ax)}$ is approximately twice the absolute value of $^2J_{TeF(eq)}$—about 700 ppm upfield from $Te(CF_3)_2$ ($\delta(^{125}Te)$ 1368 [18]).

The reactivities of the homoleptic tellurate and iodate are demonstrated by nucleophilic substitutions as shown in Scheme 2 [2,19–24]. All reactions proceed spontaneously at −78 to −60 °C. The products were identified on the basis of published ^{19}F NMR data. Besides the resonances of the compounds mentioned in Scheme 2, exclusively the signals of $Te(CF_3)_2$ and CF_3I, respectively and CF_3H were detected.

2.2. Preparation of salts with the anion $[Te(CF_3)_2Cl]^-$ and the crystal structure of $[TAS]_2[(CF_3)_2Te(\mu\text{-}Cl)_2Te(CF_3)_2]$

Attempts to grow single crystals of $[(CH_3)_4N][Te(CF_3)_3]$ or $[TAS][Te(CF_3)_3]$ from solutions remained unsuccessful. But we separated few crystals of $[(CH_3)_4N][Te(CF_3)_2Cl]$ as well as $[TAS][Te(CF_3)_2Cl]$ as crystalline impurities which were suitable for crystal structure determination.

The formation of this salt might be explained as shown in the following equations:

$$[CAT]F + (CH_3)_3SiCl \rightarrow [CAT]Cl + (CH_3)_3SiF;$$
$$CAT = N(CH_3)_4, TAS$$

$$[CAT]Cl + Te(CF_3)_2 \rightarrow [CAT][Te(CF_3)_2Cl];$$
$$CAT = N(CH_3)_4, TAS$$

and is caused by the impurity of up to 0.5% $(CH_3)_3SiCl$ in commercially available $(CH_3)_3SiCF_3$ which was used without further purification.

Therefore, we tried to prepare salts with the $[Te(CF_3)_2Cl]^-$ anion reacting either $Te(CF_3)_2$, $(CH_3)_3SiCl$ and $[TAS][(CH_3)_3SiF_2]$ at low temperature in THF or directly $Te(CF_3)_2$ and $[CAT]Cl$ ($CAT = N(CH_3)_4$,

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[1] $SnCF_3$ compounds: $^3J_{CF} = 5.4$–9 Hz; $Sb(CF_3)_3$: $^3J_{CF} = 3.8$ Hz; $Te(CF_3)_2$: $^3J = 2.6$ Hz.

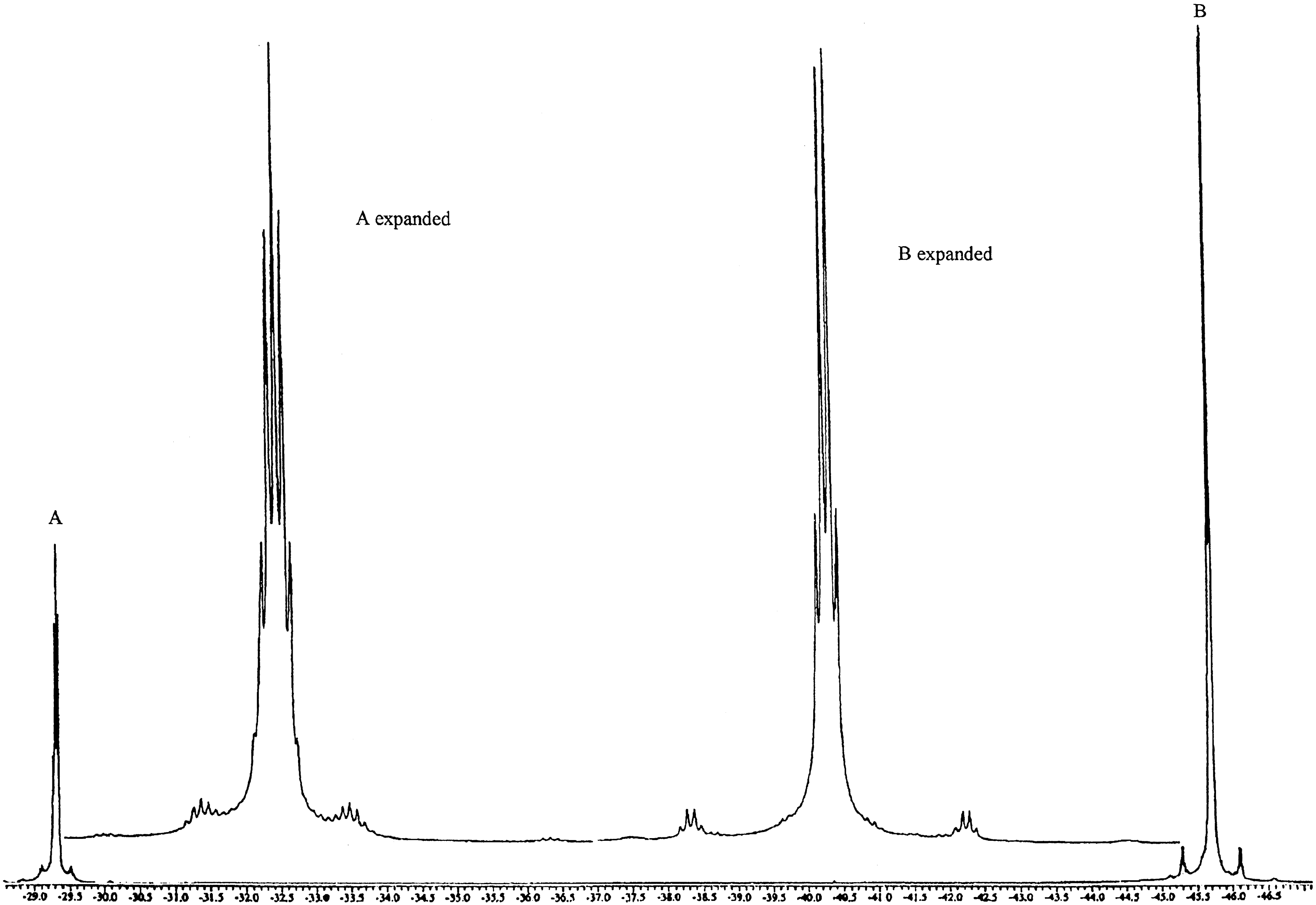
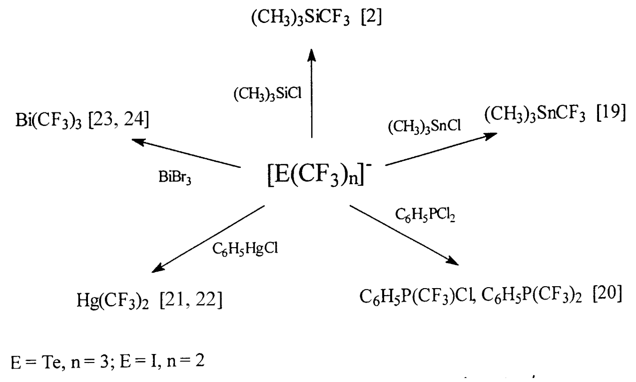


Fig. 1. ^{19}F NMR spectrum of the anion $[Te(CF_3)_3]^-$ (glyme, −40 °C)—A: signal of the equatorial CF_3 group; B: signal of the axial CF_3 groups.



Scheme 2.

$P(C_6H_5)_4$, PNP) in a variety of solvents. Best results were obtained reacting [PNP]Cl and excess of $Te(CF_3)_2$ in CH_2Cl_2 solution. After evaporation of all volatile components and washing of the residue with *n*-pentane, $[PNP][Te(CF_3)_2Cl]$ was obtained as a colorless solid decomposing at 104 °C.

In contrast to the NMR data of $[Te(CF_3)_3]^-$, those of $[Te(CF_3)_2Cl]^-$ exhibit the expected tendencies compared with $Te(CF_3)_2$ [8,18] and $[I(CF_3)Cl]^-$ [12]. The ^{19}F resonance is significantly shifted upfield by ca. 7 ppm and the absolute values of the $^1J_{CF}$ and $^2J_{TeF}$ couplings are increased (353 Hz [8] versus 359 Hz and 48 Hz (CH_2Cl_2) [8] versus 124 Hz). The ^{13}C resonance (111.4 ppm) shows the expected quartet of quartets with a typical $^3J_{FC}$ of 3 Hz. A septet is detected in the ^{125}Te NMR spectrum at δ 1154 approximately 214 ppm upfield from neat $Te(CF_3)_2$ [18].

Table 2
Crystallographic data of $[(TAS)_2][(CF_3)_2Te(\mu\text{-}Cl)_2Te(CF_3)_2]$ and their determination

Formula	$C_{16}H_{36}Cl_2F_{12}N_6S_2Te_2$
Formula weight (g/mol)	930.73
Unit cell parameters	$a = 9.550(4)$ Å
	$b = 10.008(3)$ Å
	$c = 10.476(5)$ Å
	$\alpha = 108.74(5)°$
	$\beta = 110.89(5)°$
	$\gamma = 98.30(4)°$
Cell volume (Å^3)	870.0(6)
Z	1
Crystal system	Triclinic
Space group	*P*-1 (no. 2)
Measuring device	Stoe IPDS
Radiation	Mo Kα (graphite monochromator; $\lambda = 71.07$ pm)
Temperature (K)	170
Range of 2θ	$5° < 2\theta < 50°$
Index range	$-12 \leq h \geq 12$
	$-13 \leq k \geq 12$
	$-13 \leq l \geq 13$
Range of φ; φ-increment	$0° < \varphi < 250°$; 2°
Number of exposures	125
Irradiation/exposure (min)	8
Detector distance (mm)	60
Absorption correction	
Absorption coefficient (mm^{-1})	2.09
Measured reflexions	6088
Unique reflexions	2811
Observed with $I > 2\sigma(I)$	2339
R_{int}	0.1490
Structure determination	SHELXS-86 and SHELXL-93
Scattering factors	Internal tables, volume C
Goodness of fit	1.001
$R1$; $wR2$ ($I_0 > 2\sigma(I)$)	0.0668; 0.1788
$R1$; $wR2$ (all data)	0.0755; 0.1861

Table 3
Selected bond lengths (Å) and angles (°) of $[(TAS)_2][(CF_3)_2Te(\mu\text{-}Cl)_2Te(CF_3)_2]$

Te1–C1	2.209(93)
Te1–C2	2.223(44)
Te1–Cl1	3.047(57)
Te1–Cl1^i	3.233(126)
C2–F23	1.329(28)
C2–F22	1.310(17)
C2–F21	1.312(43)
C1–Te1–C2	87.33(3)
Cl1^i –Te1–Cl1	95.95(2)
Cl1–Te1–Cl1^i –Te1^i	0.00(2)
C2–Cl1–Cl1–C1	0.59(1)

^{i} Symmetry codes: (i) $-x$, $-y$, $-z$.

Collection of fractional data of $[TAS][Te(CF_3)_2Cl]$ was carried out at −70 °C. The new salt crystallizes triclinic in the space group *P*-1. Crystallographic data and details of the crystal structure determination are summarized in Table 2. Selected bond lengths and angles are given in Table 3.

Crystalographic data showed that the anion forms a dimer (Fig. 2). Therefore, the composition of the compound is best expressed by $[TAS]_2[(CF_3)_2Te(\mu\text{-}Cl)_2Te(CF_3)_2]$.

The surroundings of both Te atoms (C_2TeCl_2) are planar as expected. The Te–C bond lengths (220.9 and 222.3 pm) are slightly elongated compared with those in $CF_3TeTeCF_3$ (217.5 and 218.6 pm) [25] and in CF_3TeCF_3 (gas phase, 215.8 pm) [26]. As expected, the Te–Cl bond lengths are significantly longer (304.7 and 323.3 pm) than those in the Te(IV) compound $(CF_3)_2TeCl_2$ (gas phase, 239.2 pm) [26].

3. Experimental

3.1. General experimental procedures

All experiments were carried out in a dry nitrogen atmosphere using Schlenk techniques. Solvents were purified according to literature procedures [27]. $(CH_3)_3SiCF_3$, $[TAS][(CH_3)_3SiF_2]$, [PNP]Cl and $(CH_3)_3SiCl$ were used as received. $Te(CF_3)_2$ [18], CF_3I [28] and $[(CH_3)_4N]F$ [29] were synthesized according to literature procedures.

NMR spectra were recorded on Bruker spectrometers AC 200 and AMX 300; frequencies (external standards): ^{13}C 75.45 MHz (TMS), ^{19}F 188.3 and 282.4 MHz (CCl_3F in $(CD_3)_2CO$), ^{125}Te 94.78 MHz ($(CH_3)_2Te$); positive shifts denote downfield resonances.

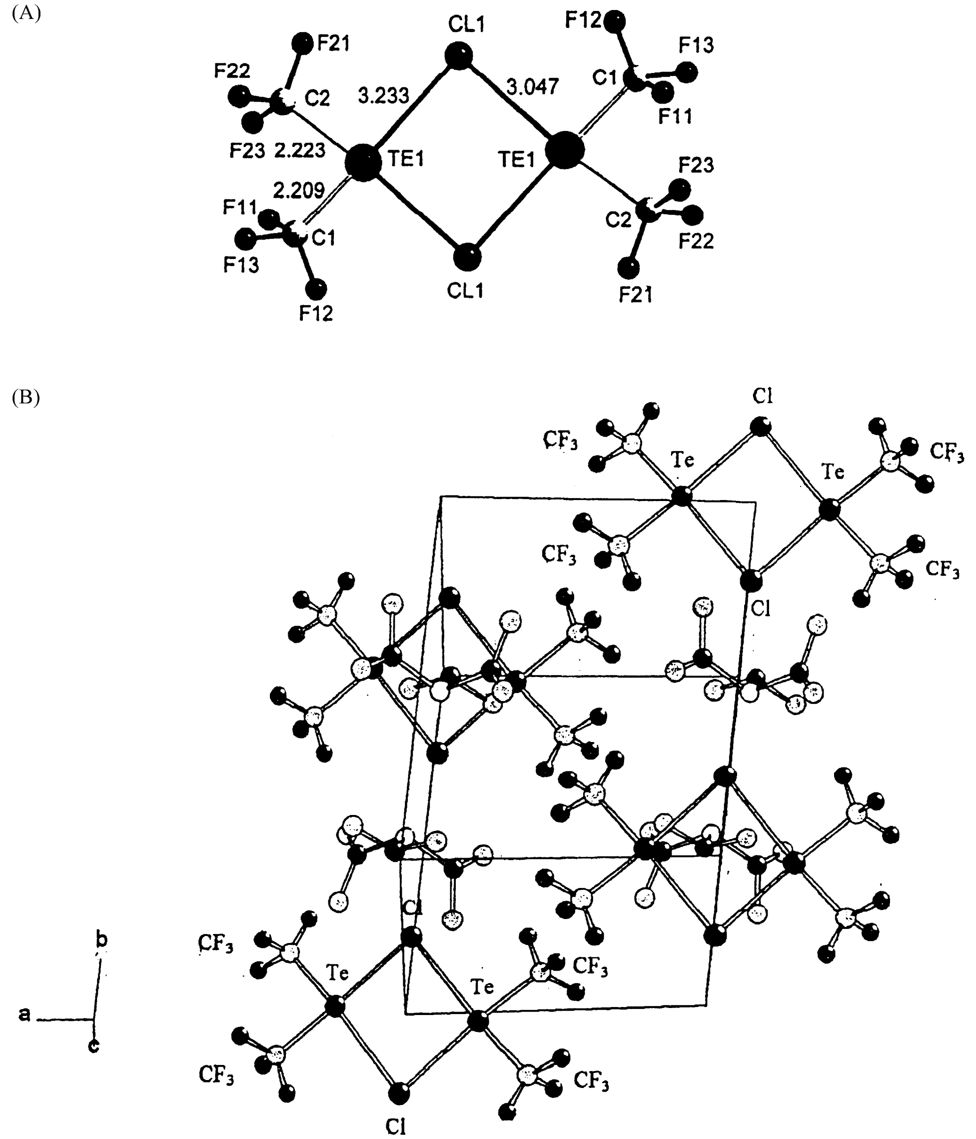


Fig. 2. Crystal structure of $[TAS]_2[(CF_3)_2Te(\mu\text{-}Cl)_2Te(CF_3)_2]$—A: molecular structure of the anion; B: view on the unit cell.

3.2. Preparation of $[(CH_3)_4N][Te(CF_3)_3]$

$Te(CF_3)_2$ (15 mmol) was condensed into a carefully dried Schlenk tube at −196 °C. Glyme (10 ml) or THF were added to the frozen compound. The reactants were warmed to ca. −100 °C. $(CH_3)_3SiCF_3$ (15 mmol) was added dropwise. To the well-stirred mixture $[(CH_3)_4N]F$ (12 mmol) was gradually added. The mixture was allowed to warm to −60 °C and stirred for approximately 2 h. A white solid precipitated from the yellow solution (excess $Te(CF_3)_2$). Cold *n*-pentane was added to complete precipitation. All volatile components ($(CH_3)_3SiF$, excess $Te(CF_3)_2$ and $(CH_3)_3SiCF_3$, *n*-pentane, glyme or THF) were removed by vacuum condensation at −30 °C over a period of approximately 24 h. $[(CH_3)_4N][Te(CF_3)_3]$ was obtained in quantitative yield relative to $[(CH_3)_4N]F$ as a colorless, highly reactive solid which decomposes at 110 °C.

Analysis: found (calcd.)—F 41.39 (41.83), Te 30.85 (31.21)%. For NMR spectra see Table 1.

3.3. Preparation of $[(CH_3)_4N][I(CF_3)_2]$

CF_3I (2 mmol) was condensed in a carefully dried 8 mm NMR tube at −196 °C. Glyme (2.5 ml) or THF were added. After warming to −100 °C $(CH_3)_3SiCF_3$ (2 mmol) and $[(CH_3)_4N]F$ (1.8 mmol) were added. The mixture was slowly warmed to −60 °C and stirred for 2 h (NMR spectroscopic control). Cold *n*-pentane (2 ml) was added. $[(CH_3)_4N][I(CF_3)_2]$ quantitatively precipitated on storing the mixture at −78 °C for several hours (NMR control). All volatile components were condensed in vacuo at −30 °C over a period of 24 h to give the iodate as a colorless, highly reactive solid in quantitative yield which decomposes between 0 °C and room temperature. For NMR spectra see Table 1.

3.4. Preparation of [PNP][$(CF_3)_2TeCl$]

$Te(CF_3)_2$, 0.57 ml (2.1 mmol), was condensed onto a solution of 1.04 g (1.8 mmol) [PNP]Cl in 5 ml CH_2Cl_2 at −196 °C. The reaction mixture was allowed to warm to ambient temperature. Above −40 °C a clear yellow solution developed. After stirring for 10 min at room temperature, the mixture was stored at −30 °C overnight. All volatile compounds were condensed in vacuo at 0 °C giving a pale yellow residue which was washed with *n*-pentane and again dried in vacuo. [PNP][$Te(CF_3)_2Cl$] was obtained as a colorless solid decomposing at 104 °C. A slow decay of the derivative into [PNP]Cl and $Te(CF_3)_2$ was observed at room temperature. Therefore, data of elemental analysis only roughly match with calculated values. It has to be mentioned that CHN analysis was made after 3 days of storage of the sample at ambient temperature.

Analysis: found (calcd.)—C 57.06 (54.36), H 4.39 (3.60), N 1.87 (1.67), Cl 3.93 (4.22), F 14.41 (13.58), Te 14.91 (15.20)%. For NMR spectra see Table 1.

Raman: 3057 (100), 2960 (5), 1588 (59), 1575 (5), 1440 (5), 1163 (6), 1111 (28), 1030 (10), 1000 (82), 727 (3), 709 (5), 685 (4), 664 (19), 617 (7), 534 (4), 362 (6), 268 (3), 227 (42), 194 (5), 176 (1).

IR (*KBr pellet*): 3056 (m), 2992 (w), 1483 (s), 1439 (vs), 1283 (vs, br), 1265 (vs, br), 1194 (s), 1184 (s), 1165 (m), 1115 (vs, br), 1063 (vs, br), 1030 (s), 997 (s), 762 (m), 748 (s), 723 (vs), 694 (vs), 546 (vs), 534 (vs), 502 (s).

3.5. Crystal structure of $[TAS]_2\,[(CF_3)_2Te(\mu\text{-}Cl)_2Te(CF_3)_2]$

The reflection intensities of a small crystal were measured with an image plate diffractometer (STOE IPDS) at 170 K. The structure solution was successful in the triclinic space group *P*-1 using the direct methods provided by the program SHELXS-86. A refinement based on least square procedures was carried out with the program SHELXL-93. For all non-hydrogen atoms anisotropic displacement parameters were used. The positions of the hydrogen atoms were calculated according to the riding model but not refined. The crystallographic data have been deposited with the Cambridge Crystallographic Data Center (CCDC, 12 Union Road, Cambridge CB2 1EZ; deposit@ccdc.cam.ac.uk) and are available on quoting the deposition number CCDC 164063.

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