Crystal structure of tris(triphenyltelluronium) fluoride tetrafluoroborate hemi(hexafluorosilicate) chloroform trisolvate, $[Te(C_6H_5)_3]_3[F][BF_4][SiF_6]_{0.5} \cdot 3CHCl_3$

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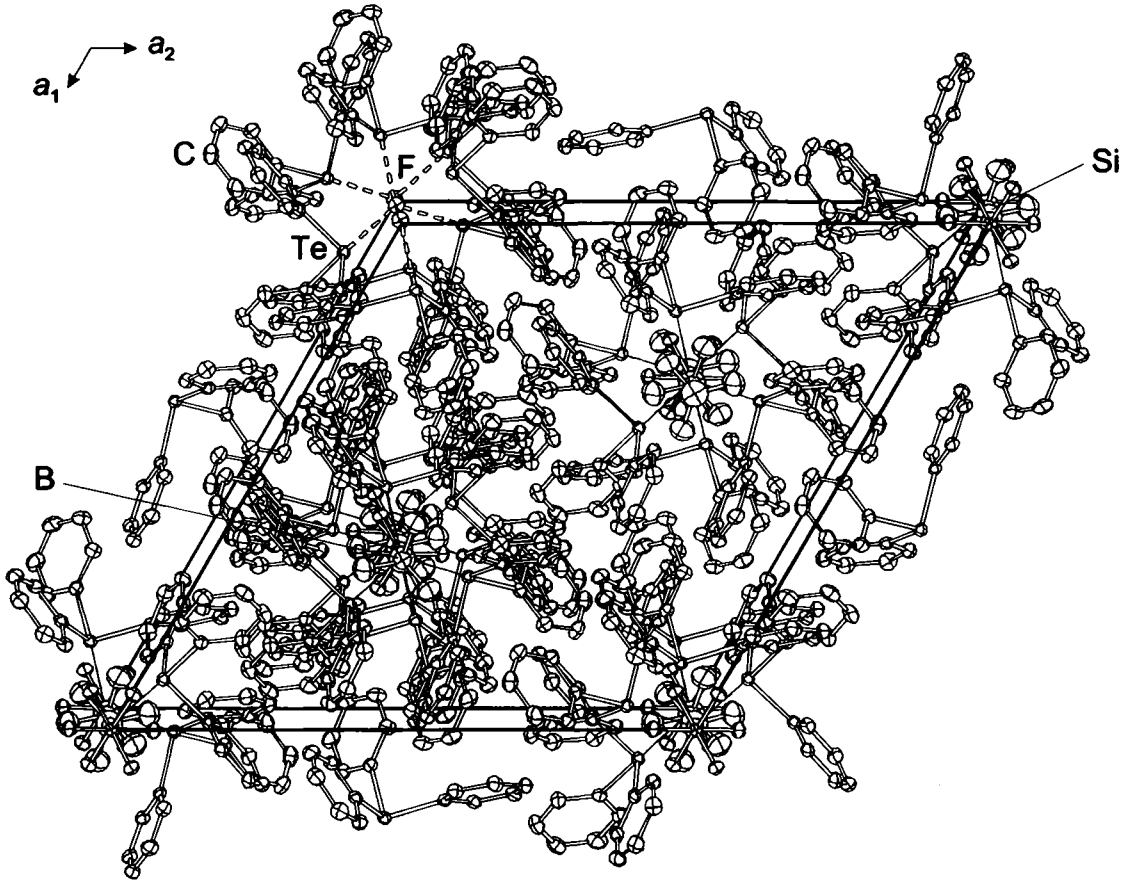

Abstract

$C_{57}H_{48}BCl_9F_8Si_{0.5}Te_3$, trigonal, $R\bar{3}$ (no. 148), $a = 19.1711(1)$ Å, $c = 29.6004(1)$ Å, $V = 9421.7$ Å^3, $Z = 6$, $R_{gt}(F) = 0.048$, $wR_{ref}(F^2) = 0.129$, $T = 200$ K.

Source of material

A solution of 1.0 mmol $[Ph_3Te]Cl$ in 15 mL of water is stirred for 2 h with 1.0 mmol AgF in a plastic apparatus at ambient temperatures. All volatile material is then pumped off in vacuo. Slow recrystallization from $CHCl_3$/*n*-hexane in a glass vessel yielded $[Ph_3Te]_3[F][BF_4][SiF_6]_{0.5} \cdot 3CHCl_3$ as colorless crystals.

Experimental details

Twofold disordered $CHCl_3$ solvate molecules are found in the crystal structure; disordered Cl atoms were therefore refined isotropically.

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Discussion

Telluronium salts $[R_3Te]X$ have been known for more than hundred years [1,2]. Some experimental work has been carried out since then, synthesizing compounds of the latter type with various substituents. In the 1970's Ziolo reported on the first telluronium pseudohalides $[(C_6H_5)_3Te]X$ (X = N_3, CN, OCN, SCN, SeCN) [3,4]. The preparation and characterization of non-ionic tris(perfluorophenyl)tellurium(IV) halogenides $(R_F)_3TeCl$ and $(R_F)_3TeBr$ ($R_F = C_6F_5$, 4-$CF_3C_6F_4$) was achieved recently [5,6], and also trimethyltelluronium and selenonium pseudohalides have been thoroughly investigated [7,8]. Telluronium halides and -pseudohalides bearing non-fluorinated substituents are in most cases widely ionic and show strong electrolyte behavior in solution [9,10]. In contrast, scarce information exists about telluronium fluorides. Emeleus et al. have apparently prepared the compounds $[R_3Te]F$ ® = Me, Ph) in the 1940s, and characterized them by means of elemental analysis [11]. McWhinnie et al. also reported $[Ph_3Te]F$ and found no ^{125}Te—^{19}F NMR coupling,

thereby indicating an ionic character of this compound [12]. We therefore decided to further elucidate these species during our investigations on telluronium pseudohalides and were able to obtain a crystal structure of a crystalline material from the reaction of $[Ph_3Te]Cl$ with silver fluoride. Obviously, handling in glass apparatus caused significant formation of $[BF_4]^-$ and $[SiF_6]^{2-}$ and their incorporation into the crystal individual, thereby increasing the telluronium/fluoride ratio. This leads to an interesting pyramidal-shaped threefold coordination of fluoride anions by telluronium cations. The Te—F (2.673(3) Å) distance lies between the sum of the van der Waals radii (3.51 Å) [13] and the Te—F bond lengths usually found in R_2TeF_2 compounds (1.9 Å – 2.0 Å) [14]. All three $[Ph_3Te]^+$ moieties coordinate within one hemisphere around the fluoride anion, the second half of space above the coordination pyramid is devoid of contacts within 5 Å.

Table 1. Data collection and handling.

Crystal:	colorless cube, size 0.12 × 0.12 × 0.12 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	18.37 cm^{-1}
Diffractometer, scan mode:	Nonius 95mm KappaCCD, φ/ω
$2\theta_{max}$:	46.5°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	63042, 3015
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2719
$N(param)_{refined}$:	235
Programs:	SIR97 [15], SHELXL-97 [16], DIAMOND [17]

Table 2. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	Occ.	x	y	z	U_{iso}
H(21)	18*f*		0.8259	0.6768	0.1006	0.052
H(31)	18*f*		0.8046	0.5726	0.0514	0.063
H(41)	18*f*		0.8987	0.5947	–0.0047	0.067
H(51)	18*f*		1.0103	0.7218	–0.0143	0.069
H(61)	18*f*		1.0300	0.8277	0.0330	0.053
H(22)	18*f*		0.8074	0.8461	0.0667	0.044
H(32)	18*f*		0.6788	0.8185	0.0859	0.050
H(42)	18*f*		0.6327	0.7889	0.1602	0.062
H(52)	18*f*		0.7143	0.7795	0.2154	0.056
H(62)	18*f*		0.8431	0.8058	0.1973	0.049
H(23)	18*f*		0.9989	0.9147	0.2110	0.046
H(33)	18*f*		1.0372	0.8753	0.2773	0.059
H(43)	18*f*		1.0501	0.7609	0.2768	0.062
H(53)	18*f*		1.0248	0.6853	0.2114	0.061
H(63)	18*f*		0.9833	0.7217	0.1453	0.050
H(1A)	18*f*	0.564	0.2158	0.6460	0.0502	0.101
H(1B)	18*f*	0.436	0.2195	0.6634	0.0523	0.101
Cl(1A)	18*f*	0.564(6)	0.2072(5)	0.6832(4)	0.1239(2)	0.121(2)
Cl(2A)	18*f*	0.564	0.1178(5)	0.5345(5)	0.0804(3)	0.132(3)
Cl(3A)	18*f*	0.564	0.1133(5)	0.6761(5)	0.0504(3)	0.150(3)
Cl(1B)	18*f*	0.436	0.0794(6)	0.5641(6)	0.0376(3)	0.123(3)
Cl(2B)	18*f*	0.436	0.1461(6)	0.6921(6)	0.1003(4)	0.135(4)
Cl(3B)	18*f*	0.436	0.1709(8)	0.5559(7)	0.1059(4)	0.155(4)

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F(1)	6*c*	0	0	0.1576(2)	0.049(3)	U_{11}	0.038(4)	½U_{11}	0	0
Te	18*f*	0.95814(2)	0.86357(2)	0.11277(1)	0.0295(3)	0.0278(3)	0.0286(3)	0.0132(2)	0.0032(2)	0.0023(2)
C(11)	18*f*	0.9302(4)	0.7615(4)	0.0716(2)	0.037(4)	0.035(4)	0.031(3)	0.020(3)	–0.001(3)	–0.002(3)
C(21)	18*f*	0.8633(4)	0.6862(4)	0.0771(2)	0.043(4)	0.041(4)	0.040(4)	0.016(3)	0.004(3)	–0.003(3)
C(31)	18*f*	0.8511(5)	0.6243(5)	0.0482(3)	0.059(5)	0.036(4)	0.052(5)	0.015(4)	–0.001(4)	–0.007(3)
C(41)	18*f*	0.9065(5)	0.6376(5)	0.0146(3)	0.079(6)	0.046(5)	0.050(5)	0.036(5)	–0.004(4)	–0.012(4)
C(51)	18*f*	0.9727(5)	0.7128(5)	0.0090(3)	0.071(6)	0.062(5)	0.051(5)	0.042(5)	0.016(4)	–0.004(4)
C(61)	18*f*	0.9847(4)	0.7755(4)	0.0372(2)	0.040(4)	0.043(4)	0.047(4)	0.020(3)	0.005(3)	–0.002(3)
C(12)	18*f*	0.8376(4)	0.8286(4)	0.1301(2)	0.029(3)	0.027(3)	0.033(3)	0.012(3)	0.006(3)	0.002(3)
C(22)	18*f*	0.7887(4)	0.8326(4)	0.0970(2)	0.035(4)	0.032(3)	0.042(4)	0.015(3)	0.007(3)	0.004(3)
C(32)	18*f*	0.7130(4)	0.8168(4)	0.1086(2)	0.035(4)	0.035(4)	0.048(4)	0.012(3)	0.007(3)	0.005(3)
C(42)	18*f*	0.6848(4)	0.7982(4)	0.1526(3)	0.036(4)	0.042(4)	0.078(6)	0.020(4)	0.016(4)	0.000(4)
C(52)	18*f*	0.7334(4)	0.7934(4)	0.1853(3)	0.043(4)	0.045(4)	0.046(4)	0.018(3)	0.019(3)	0.001(3)
C(62)	18*f*	0.8096(4)	0.8087(4)	0.1746(2)	0.044(4)	0.039(4)	0.037(4)	0.018(3)	0.005(3)	0.000(3)
C(13)	18*f*	0.9869(4)	0.8214(4)	0.1726(2)	0.028(3)	0.032(3)	0.035(3)	0.011(3)	0.003(3)	0.006(3)
C(23)	18*f*	1.0034(4)	0.8675(4)	0.2112(2)	0.041(4)	0.038(4)	0.036(4)	0.019(3)	0.003(3)	0.006(3)
C(33)	18*f*	1.0266(5)	0.8445(5)	0.2505(2)	0.056(5)	0.053(5)	0.034(4)	0.024(4)	–0.002(3)	0.005(3)
C(43)	18*f*	1.0341(5)	0.7767(5)	0.2501(3)	0.049(4)	0.053(5)	0.048(5)	0.021(4)	–0.003(4)	0.018(4)
C(53)	18*f*	1.0187(5)	0.7316(5)	0.2113(3)	0.055(5)	0.041(4)	0.060(5)	0.026(4)	–0.006(4)	0.010(4)
C(63)	18*f*	0.9945(4)	0.7531(4)	0.1720(2)	0.042(4)	0.031(4)	0.046(4)	0.013(3)	–0.001(3)	0.005(3)
Si(1)	3*a*	0	0	0	0.029(1)	U_{11}	0.021(2)	½U_{11}	0	0
F(2)	18*f*	0.9266(2)	0.9295(2)	0.0328(1)	0.036(2)	0.037(2)	0.032(2)	0.013(2)	0.005(2)	0.004(2)
B(1)	6*c*	⅔	⅓	0.0123(6)	0.059(7)	U_{11}	0.07(1)	½U_{11}	0	0
F(11)	6*c*	⅔	⅓	0.0585(3)	0.127(6)	U_{11}	0.053(6)	½U_{11}	0	0
F(21)	18*f*	0.5923(4)	0.3170(4)	–0.0016(3)	0.090(5)	0.113(5)	0.134(6)	0.052(4)	–0.029(4)	0.003(4)
C(1A)	18*f*	0.1698(7)	0.6330(7)	0.0710(4)	0.098(8)	0.090(8)	0.072(7)	0.052(7)	0.020(6)	0.010(6)

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