Inorg. Chem. **2001,** *40,* 5169−5176

Spectroscopic and Structural Studies on Polyfluorophenyl Tellurides and Tellurium(IV) Dihalides

Thomas M. Klapötke,* Burkhard Krumm, Peter Mayer, Kurt Polborn, and Oliver P. Ruscitti

Department of Chemistry, Ludwig-Maximilians University of Munich, Butenandtstrasse 5-13 (D),
D-81377 Munich, Germany

Received February 5, 2001

Bis(fluorophenyl) tellurides R_2Te (R = $C_6F_2H_3$ (**1**), $CF_3C_6F_4$ (**2**), $CF_3C_6F_4OC_6F_4$ (**3**), and C_6F_5 (**4**)) are synthesized by the facile reaction of Na_2Te with bromo-fluorobenzenes, RBr. The corresponding bis(fluorophenyl)tellurium(IV) dihalides, R_2TeHal_2 (Hal = F, Cl, and Br) (**5**−**16**), are obtained by the oxidation of **1**−**4** with mild halogenating agents (XeF_2, SO_2Cl_2, and Br_2). The dihalides show temperature-dependent NMR spectra. On the basis of the ^{19}F NMR spectra of the two series, $(C_6F_2H_3)_2TeHal_2$ (Hal = F (**5**), Cl (**9**), and Br (**13**)) and R_2TeCl_2 (R = $C_6F_2H_3$ (**9**), $CF_3C_6F_4$ (**10**), $CF_3C_6F_4OC_6F_4$ (**11**), and C_6F_5 (**12**)), the coalescence temperatures, T_c, and free enthalpies, $\Delta G^\ddagger$, of rotation of the TeC bonds are determined. The activation enthalpies for the dichlorides/dibromide **9**−**13** are in the range of 14.4−15.2 kcal mol^{-1} and that for the difluoride **5** is considerably lower at 10.7 kcal mol^{-1}. In addition to thorough spectroscopic characterization of **1**−**16**, the crystal structures of the monotellurides **2** and **4** as well as of the tellurium(IV) dihalides **5**, **6**, **9**, **10**, and **13** were determined. The dihalides show interesting intermolecular Te···Hal contacts, significantly shorter than the sum of the van der Waals radii, leading to different networks of association.

Introduction

The chemistry of organo tellurides has been known for more than 150 years,[1] and its progress is demonstrated in the applications for organic syntheses [2−5] and utilization in the semiconductor industry.[6−8] The first tellurium compound with perfluorinated groups, $(CF_3)_2Te_2$, was reported relatively late in the early 1960s.[9] The preparation of $(C_6F_5)_2Te$ followed, but its characterization is still incomplete. The early synthesis of $(C_6F_5)_2Te$ was accomplished with the use of mercury reagents, i.e., treatment of elemental tellurium either with bis(pentafluorophenyl)mercury at 230 °C or, in low yields, using pentafluoroiodobenzene or the combination of C_6F_5Li and $TeCl_4$.[10] A more convenient alternative for the preparation of $(C_6F_5)_2Te$ was found in the reaction of Na_2Te with C_6F_5Br in liquid ammonia or THF under photolytic conditions.[11] In the recent past, several reports of tellurium compounds with perfluorinated groups have appeared,[11−23] but little is known about organo tellurides with partially fluorinated aromatic substituents.[24] Quite recently, we reported the syntheses and structures of the first fluorinated and nonfluorinated diphenyltellurium(IV) diazides.[25]

In this paper the syntheses, spectroscopic studies, structures, and bonding properties of some new fluorophenyltellurium compounds are discussed. The already known pentafluorophenyl derivatives were included to complete their spectroscopic data as well as the crystal structure of $(C_6F_5)_2Te$, which completes the series of the higher bis(pentafluorophenyl) chalcogenides $(C_6F_5)_2X$ (X = S,[26] Se,[27] and Te).

Results and Discussion

Synthesis. For the preparation of the bis(fluorophenyl) tellurides, a modified procedure of the literature method for

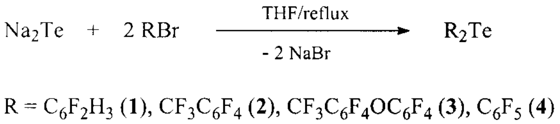
---

* To whom correspondence should be addressed. Fax: +49-89-2180-7492. E-mail: tmk@cup.uni-muenchen.de.

(1) Wöhler, F. *Liebigs Ann. Chem.* **1840**, *35*, 111.
(2) Engman, L. *Acc. Chem. Res.* **1985**, *18*, 274.
(3) Osaka, A.; Suzuki, H. *Tetrahedron Lett.* **1983**, *24*, 5109.
(4) Clive, D. L. J.; Chittattu, G. J.; Farina, V.; Kiel, W. A.; Menchen, S. M.; Russell, C. G.; Singh, A.; Wong, C. K.; Curtis, N. J. *J. Am. Chem. Soc.* **1980**, *102*, 4438.
(5) Butcher, T. S.; Detty, M. R. *J. Org. Chem.* **1999**, *64*, 5677.
(6) Steigerwald, M. L.; Sprinkle, L. R. *J. Am. Chem. Soc.* **1987**, *109*, 245.
(7) Steigerwald, M. L.; Sprinkle, L. R. *Organometallics* **1988**, *7*, 245.
(8) Hoke, W. E.; Lemonias, P. J. *Appl. Phys. Lett.* **1985**, *46*, 398.
(9) Bell, T. N.; Pullman, B. J.; West, B. O. *Aust. J. Chem.* **1963**, *16*, 722.
(10) Cohen, S. C.; Reddy, M. L. N.; Massey, A. G. *J. Organomet. Chem.* **1968**, *11*, 563.
(11) Kasemann, R.; Lichenheim, L.; Nowicki, G.; Naumann, D. *Z. Anorg. Allg. Chem.* **1995**, *621*, 213.
(12) Rainville, D. P.; Zingaro, R. A.; Meyers, R. A. *J. Fluorine Chem.* **1980**, *16*, 245.
(13) Wilkes, B.; Naumann, D. *J. Fluorine Chem.* **1985**, *27*, 115.
(14) Kasemann, R.; Naumann, D. *J. Fluorine Chem.* **1988**, *41*, 321.
(15) Kasemann, R.; Naumann, D. *J. Fluorine Chem.* **1990**, *48*, 207.
(16) Baum, M.; Beck, J.; Haas, A.; Herrendorf, W.; Monsé, C. *J. Chem. Soc., Dalton Trans.* **2000**, 11.
(17) Aramini, J.; Batchelor, R. J.; Jones, C. H. W.; Einstein, F. W. B.; Sharma, R. D. *Can. J. Chem.* **1987**, *47*, 2643.
(18) Naumann, D.; Ehmanns, L.; Tebbe, K.-F.; Crump, W. *Z. Anorg. Allg. Chem.* **1993**, *619*, 1269.
(19) Aust, J.; Haas, A.; Kauch, M.; Pritsch, A.; Stange, B. *J. Fluorine Chem.* **1999**, *96*, 147.
(20) Jones, C. H. W.; Sharma R. D.; Naumann, D. *Can. J. Chem.* **1986**, *64*, 987.
(21) Gockel, S.; Haas, A.; Probst, V.; Boese, R.; Müller, I. *J. Fluorine Chem.* **2000**, *102*, 301.
(22) Boese, R.; Haas, A.; Herkt, S.; Pryka, M. *Chem. Ber.* **1995**, *128*, 423.
(23) Beck, J.; Haas, A.; Herrendorf, W.; Heuduk, H. *J. Chem. Soc., Dalton Trans.* **1996**, 4463.
(24) Lewe, T.; Naumann, D.; Nowicki, G.; Schneider, H.; Tyrra, W. *Z. Anorg. Allg. Chem.* **1997**, *623*, 122.
(25) Klapötke, T. M.; Krumm, B.; Mayer, P.; Ruscitti, O. P. *Inorg. Chem.* **2000**, *39*, 5426.
(26) Minkwitz, R.; Preut, H.; Sawatzki, J. *Z. Anorg. Allg. Chem.* **1989**, *569*, 158.
(27) Klapötke, T. M.; Krumm, B.; Polborn, K. *Eur. J. Inorg. Chem.* **1999**, 1359.

10.1021/ic010149r CCC: $20.00

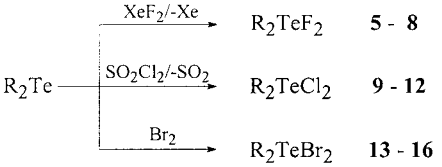
Scheme 1. Preparation of Bis(fluorophenyl) Tellurides



$$Na_2Te + 2\ RBr \xrightarrow[-\ 2\ NaBr]{THF/reflux} R_2Te$$

$$R = C_6F_2H_3\ (\mathbf{1}),\ CF_3C_6F_4\ (\mathbf{2}),\ CF_3C_6F_4OC_6F_4\ (\mathbf{3}),\ C_6F_5\ (\mathbf{4})$$

Scheme 2. Halogenation of Bis(fluorophenyl) Tellurides



$(C_6F_5)_2Te$ was used.[11] Good yields were obtained by simply refluxing disodium telluride [28] and the corresponding bromobenzene for 16 h in THF (Scheme 1). Purification of the products was achieved by vacuum sublimation. Other methods to produce C_6F_5 (**4**) gave only moderate yields.[10,11,23] The tellurides, R_2Te ($R = C_6F_2H_3$ (**1**), $CF_3C_6F_4$ (**2**), $CF_3C_6F_4OC_6F_4$ (**3**), and **4**), are air and moisture stable compounds and soluble in common organic solvents.

Studies on the variation of the aromatic substituents at RBr according to Scheme 1 revealed that the presence of both fluorine atoms in the ortho position to bromine is crucial to produce a reaction with Na_2Te. Since bromobenzene does not react with Na_2Te, diphenyl telluride is, therefore, prepared by other methods.[29] The introduction of one fluorine in the 2-position, bromo-2-fluorobenzene, and one other fluorine in the 4-position, bromo-2,4-difluorobenzene, respectively, was also found to be unsuitable to form the corresponding tellurides upon prolonged treatment with Na_2Te. The fact that bromoalkanes[28] and bromo-2,6-difluorobenzene do react but bromobenzene does not react with Na_2Te leads to the conclusion that not only does the high nucleophilic power of Te^{2-} play a role but, in addition, the bromobenzene must contain two fluorine atoms in the ortho position to produce enough electrophilicity at the CBr carbon to enable a reaction with the strong nucleophilic telluride anion.

The reaction of **1**−**4** with mild halogenating agents, XeF_2, SO_2Cl_2, and Br_2, led to the formation of the corresponding bis(fluorophenyl)tellurium(IV) dihalides (**5**−**16**) (Scheme 2). The formation of a tellurium−iodine bond with fluoroaromatic substituents was not possible. The tellurides **(1**−**4)** do not react with iodine or other iodination agents. For example, the reaction of **4** with equimolar amounts of ICl only resulted in chlorination to give **12**. Treatment of the difluoride **8** with trimethylsilyl iodide led to a redox reaction, and I_2, Me_3SiF, and **4** were identified. Other methods to form a TeI bond could not be verified.[30]

Vibrational Spectra. The diaryltellurium(IV) dihalides **(5**−**16)** show in the Raman spectra the characteristic intense peaks of the Te−Hal stretching vibration. As expected, their frequencies decrease from F to Br: ν_{TeF} is found at ∼480 cm^{-1}, ν_{TeCl} at ∼280 cm^{-1}, and ν_{TeBr} at ∼170 cm^{-1} (see Experimental Section). In contrast to aliphatic tellurides where ν_{TeC} can be assigned,[28] the TeC stretching vibrations in compounds **1**−**16** are of a weaker intensity and are not characteristic due to overlapping with the other vibrations of the aromatic moiety.

NMR Spectra. The ^{19}F NMR spectra of the bis(fluorophenyl)tellurium(IV) dihalides, R_2TeHal_2 (**5**−**16**), show an interesting temperature-dependent dynamic behavior. Therefore, variable temperature ^{19}F NMR spectra were recorded for two selected sets of compounds; the series with variation in the tellurium bound halogens and the aromatic substituent kept constant, i.e., $(C_6F_2H_3)_2TeHal_2$ (Hal = F (**5**), Cl (**9**), and Br (**13**)), and the series of bis(fluorophenyl)tellurium(IV) dichlorides, where $R = C_6F_2H_3$ (**5**), $CF_3C_6F_4$ (**10**), $CF_3C_6F_4OC_6F_4$ (**11**), and C_6F_5 (**12**). The restricted rotation of the TeC bonds can be observed with the *o*-F resonances and, where appropriate, with the *meta*-F resonances (the temperature dependency is also visible for the *m*-H ^1H NMR resonances of **5**, **9**, and **13** as well as in the ^{13}C NMR spectra of R_2TeHal_2 but was not further examined in this study). Due to the commonly known solvent dependency, all spectra were run in the same solvent. The temperature-dependent ^{19}F NMR spectra were recorded for all selected compounds in toluene-d_8 in the range of −90 to +100 °C, with data obtained for 10 fixed temperatures and additional 1 °C steps around the point of coalescence. Reaching a certain coalescence temperature causes the merging of two separate resonances for *o(m)*-F, (2(3)-F), and above that temperature the resonances merge into a sharpening single resonance. As a representative example, the variable temperature ^{19}F NMR spectra series of **5** was selected and is shown in Figure 1. Here, in addition to the C−F resonances, the TeF_2 resonance is visible at a higher field. With regard to the dynamic behavior of the C−F resonance, a significantly higher temperature dependency of the TeF_2 resonance is obvious, shifting to a lower field with increasing temperature.

From the spectra, the coalescence temperature, T_c, for each 2-F resonance and the corresponding chemical shift difference, $\Delta\nu$, are determined. The shift difference, $\Delta\nu$, is determined for all compounds at −60 °C where a negligible slow exchange rate is present,[31] leading to relatively sharp discrete resonances. With these data, the free enthalpy of activation, $\Delta G^{\ddagger}$, is calculated (Table 1) by applying the Eyring equation.[31] For the 3-F resonances of compounds **10**, **11**, and **12**, similar values of $\Delta G^{\ddagger}$ were obtained. In general, the coalescence temperature of 3-F is lower (**10**, $T_c = 297$ K; **11**, $T_c = 314$ K; and **12**, $T_c = 303$ K) than that of the corresponding 2-F resonance. Therefore, identical values should be obtained for the rotation barrier of the same TeC bond at the same substituent, which is the case for **10**, **11**, and **12** within the error limits.

All activation barriers are within a 0.8 kcal mol^{-1} range around 15 kcal mol^{-1}, except the value obtained for the difluoride **5,** which was ∼5 kcal mol^{-1} lower than the dichlorides and dibromides. For these compounds it seems that the variation of the fluoroaromatic substituent does not result in a significant change of the activation barrier, as is observed for the change of the axial halogen substituent from Cl/Br to F. Accordingly, noticeable in Table 1 is the large difference between the T_c and $\Delta\nu$ values of the dichlorides/dibromide (∼300−350 K and ∼500−1300 Hz) and those of the difluoride **5** (233 K and 189 Hz).

Another phenomenon observed in the ^{19}F NMR spectra of R_2TeHal_2 (also visible in Figure 1) is the occurence of one large

---

(28) Karaghiosoff, K.; Klapötke, T. M.; Krumm, B.; Ruscitti, O. P. *J. Organomet. Chem.* **1999**, *577*, 69.

(29) (a) Krafft, F.; Lyons, R. E. *Chem. Ber.* **1894**, *27*, 1768. (b) Günther, W. H. H.; Nepywoda, J.; Chun, J. Y. C. *J. Organomet. Chem.* **1974**, *74*, 19. (c) Rheinboldt, H.; Petragnani, N. *Chem. Ber.* **1956**, *89*, 1270. (d) McWhinnie, W. R.; Patel, M. G. *J. Chem. Soc., Dalton Trans.* **1972**, 194.

(30) (a) Srivasta, T. N.; Srivasta, R. C.; Singh, M. *J. Organomet. Chem.* **1978**, *110*, 449. (b) Srivasta, T. N.; Srivasta, R. C.; Srivasta, V. K. *Indian J. Chem., Sect. A* **1983**, *22*, 503. (c) Srivasta, T. N.; Srivasta, R. C.; Singh, M. *Indian J. Chem., Sect. A* **1980**, *22*, 435.

(31) Oki, M. *Applications of Dynamic NMR Spectroscopy to Organic Chemistry*; VCH Publishers: Weinheim (Germany), 1985.

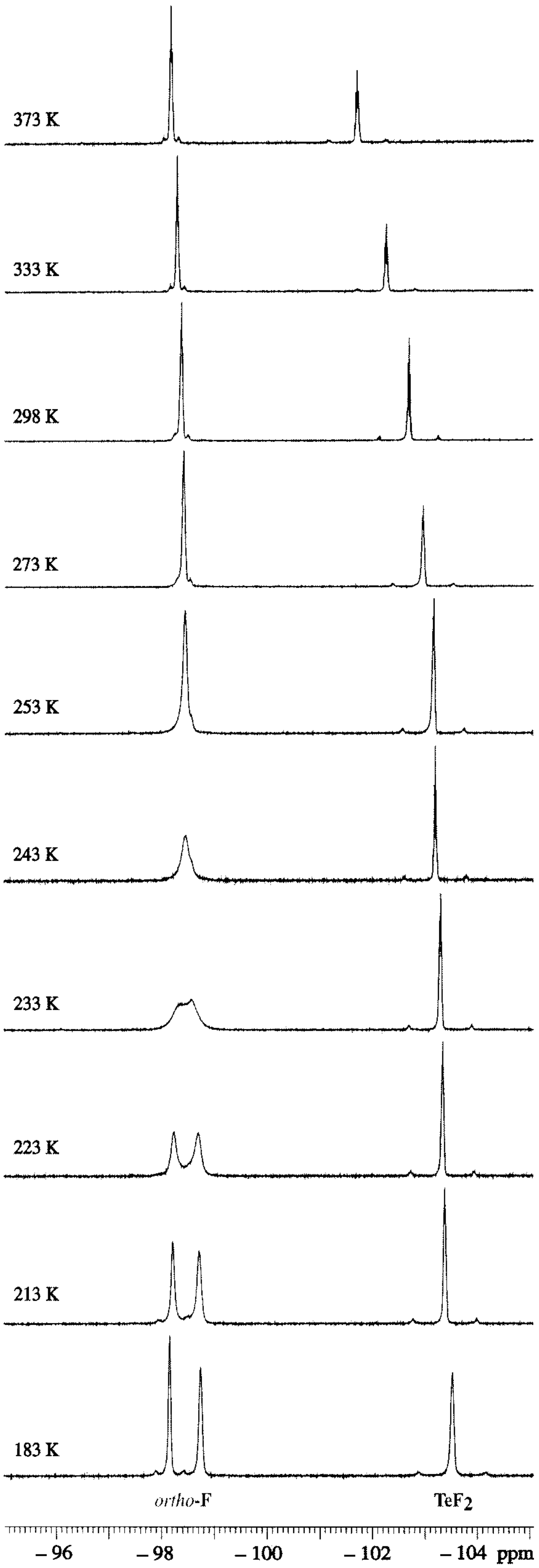


Figure 1. ^{19}F NMR variable temperature spectra of $(C_6F_2H_3)_2TeF_2$ (**5**) in toluene-d_8.

and one small value of the $^3J_{Te-F}$ coupling constant of both the 2-F resonances below the coalescence temperature. For example, in **9**, values of $^3J_{Te-F} = 187.3$ (at lower field) and 50.3 Hz (at higher field) are found at −20 °C in toluene-d_8. However, a definite assignment of both 2-F and 3-F resonances in R_2TeHal_2 is not possible.

Table 1. Variable Temperature ^{19}F NMR Studies of the *o*-F Resonances of R_2TeHal_2 (Toluene-d_8)

R_2TeHal_2	T_c (K)	$\Delta\nu$ (Hz)	$\Delta G^{\ddagger}$ (kcal mol^{-1})
$(C_6F_2H_3)_2TeF_2$ (**5**)	233	189	10.7
$(C_6F_2H_3)_2TeBr_2$ (**13**)	335	751	14.7
$(C_6F_2H_3)_2TeCl_2$ (**9**)	344	703	15.2
$(CF_3C_6F_4)_2TeCl_2$ (**10**)	335	1309	14.4
$(CF_3C_6F_4OC_6F_4)_2TeCl_2$ (**11**)	338	871	14.8
$(C_6F_5)_2TeCl_2$ (**12**)	343	930	14.9

In another experiment, the dynamic behavior of the ^{125}Te resonance was examined at low temperature. Suitable for this study was the resonance of **5**. At 25 °C the signal is split into a triplet of quintets. The coupling pattern arises from coupling to two TeF fluorine atoms, where each of the three lines is split into quintets due to coupling with four equivalent fluorine atoms in the ortho position of the difluorophenyl rings. Lowering the temperature (spectra were recorded in acetone-d_6 due to increased solubility at lower temperatures) below the coalescence temperature ($T_c = -40$ °C) results in an obvious change in the coupling pattern; at −90 °C a triplet of triplets of triplets exists. The coupling to the TeF_2 fluorine atoms remains unchanged, but each line is split into triplets (large coupling to two *o*-fluorines) which are then each split again into triplets (small coupling to the other two *o*-fluorines). This confirms the similar results exemplarily discussed for **9** in the ^{19}F NMR spectra.

Furthermore, the variable temperature ^{125}Te NMR study clearly excludes the possibility of pseudorotation around Te. In addition, a dissociation of R_2TeHal_2 into halogenide and telluronium cations or a fluorine exchange in R_2SeF_2 systems, as suggested earlier,[32,33] can be ruled out. Recent variable temperature ^{1}H NMR studies on 2,6-dimethoxy/dimethyl substituted diphenyltellurium(IV) dihalides were explained with restricted rotations around TeC bonds but were ascribed to the supposed bulkiness of these particular substituents.[34] As shown by our compounds of sterically nondemanding R_2TeHal_2 systems, the restricted rotation around TeC bonds is a generally observed phenomenon for derivatives of this type.

Description of the Crystal Structures. The property of some of the tellurides and their tellurium(IV) dihalides to crystallize as single crystals provides an opportunity to investigate their solid-state structures with the use of single-crystal X-ray diffraction techniques. Crystal data and structure refinements of compounds **2**, **4**, **5**, **6**, **9**, **10**, and **13** are given in Table 2. Their molecular structures are shown in Figures 2−8. Suitable crystals were obtained by slow solvent evaporation (chloroform and benzene), and those of **4** were obtained by vacuum sublimation.

The tellurides **2** and **4** are the first fluoroaromatic-substituted tellurium(II) compounds, except the cyclic perfluorotelluranthrene[12] for which a crystal structure has already been determined. The only crystal structure suitable for a structural comparison is the one determined for the non-fluorinated analogue of **2**, $(4\text{-}CH_3C_6H_4)_2Te$,[35] while $(C_6H_5)_2Te$ is a liquid at ambient temperature. In **2** (Figure 2) and **4** (Figure 3) the Te−C bonds are 2.09−2.12 Å in length and slightly longer than those in $(4\text{-}CH_3C_6H_4)_2Te$ (2.05 Å). The C−Te−C angles in **2**

---

(32) (a) Wynne, K. J. *Inorg. Chem.* **1970**, *9*, 299. (b) Wynne, K. J.; Puckett, J. *Chem. Commun.* **1968**, 1532.

(33) Gombler, W. *Phosphorus, Sulfur Silicon Relat. Elem.* **1988**, *38*, 231.

(34) (a) Asahara, M.; Tanaka, M.; Erabi, T.; Wada, M. *J. Chem. Soc., Dalton Trans.* **2000**, 3493. (b) Takaguchi, Y.; Fujihara, H.; Furukawa, N. *J. Organomet. Chem.* **1995**, *498*, 49.

(35) Blackmore, W. R.; Abrahams, S. C. *Acta Crystallogr.* **1955**, *8*, 317.

Table 2. Crystal Data and Structure Refinements for **2**, **4**, **5**, **6**, **9**, **10**, and **13**

	2	**4**	**6**	**10**	**5**	**9**	**13**
empirical formula	$C_{14}F_{14}Te$	$C_{12}F_{10}Te$	$C_{28}F_{32}Te_2$	$C_{14}Cl_2F_{14}Te$	$C_{12}H_6F_6Te$	$C_{12}H_6Cl_2F_4Te$	$C_{12}H_6Br_2F_4Te$
formula weight	561.73	461.72	1199.45	632.63	391.77	424.68	513.58
color	pale yellow	pale yellow	colorless	colorless	colorless	colorless	yellow
crystal size (mm)	0.20 × 0.10 × 0.04	0.33 × 0.43 × 0.60	0.40 × 0.32 × 0.11	0.33 × 0.16 × 0.04	0.30 × 0.20 × 0.10	0.20 × 0.17 × 0.07	0.17 × 0.15 × 0.03
cryst. system	monoclinic	monoclinic	monoclinic	monoclinic	tetragonal	orthorhombic	orthorhombic
space group	$P2_1$	$P2_1$	$P2_1$	$P2_1/n$	$I4_1/acd$	*Pbca*	*Pbca*
temperature (K)	200(3)	294(2)	200(3)	200(3)	200(3)	200(3)	200(3)
ρ_{calcd} (g cm^{-3})	2.399	2.361	2.366	2.414	2.058	2.202	2.509
μ (cm^{-1})	20.68	24.06	19.35	21.60	24.08	27.68	80.92
a (Å)	15.042(3)	9.324(2)	11.8267(9)	13.7666(7)	18.0889(9)	11.1630(6)	11.3495(5)
b (Å)	5.0180(6)	7.512(3)	13.002(1)	6.5075(3)	18.0889(9)	8.2000(4)	8.4819(4)
c (Å)	10.305(1)	9.687(2)	12.022(1)	20.544(1)	15.4579(9)	27.987(1)	28.251(2)
β (deg)	90.88(2)	106.82(2)	114.373(9)	108.971(6)			
V (Å^3)	777.8(2)	649.5(3)	1683.9(3)	1740.5(2)	5058.0(5)	2561.8(2)	2719.6(3)
Z	2	2	2	4	16	8	8
reflcns collected	2888	2155	13150	8517	12862	6562	10149
reflcns unique	1943	2020	6150	3317	1231	2337	2504
R_{int}	0.0484	0.0076	0.0289	0.0335	0.0510	0.0502	0.0432
$R1$, w$R2^a$ (2σ data)	0.0450, 0.1071	0.0273, 0.0739	0.0284, 0.0730	0.0247, 0.0657	0.0174, 0.0398	0.0401, 0.0983	0.0211, 0.0452
$R1$, w$R2^a$ (all data)	0.0555, 0.1113	0.0287, 0.0784	0.0299, 0.0737	0.0281, 0.0739	0.0212, 0.0410	0.0495, 0.1029	0.0320, 0.0470
Goof s^b on F^2	0.990	1.278	1.016	1.138	1.054	0.989	0.919
Flack parameter	0.02(5)	−0.03(3)	−0.01(1)				

^{a} $R1 = (\sum||F_o| - |F_c||)/\sum|F_o|$. $wR2 = \{\sum[w(F^2_o - F^2_c)^2]/\sum[w(F^2_o)^2]\}^{1/2}$ with $w^{-1} = \sigma^2(F^2_o) + (aP)^2$. ^{b} $s = \{\sum[w(F^2_o - F^2_c)^2]/(N_o - N_p)\}^{1/2}$.

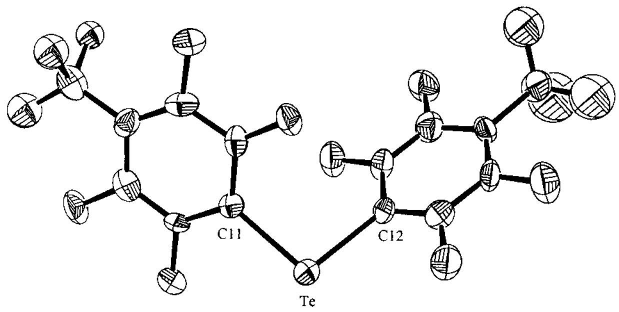


Figure 2. Molecular structure of $(CF_3C_6F_4)_2Te$ (**2**), with thermal ellipsoids at the 50% probability level. Selected bond lengths (Å) and angles (deg): Te−C(11) 2.122(7), Te−C(12) 2.11(1); C(12)−Te−C(11) 98.8(4).

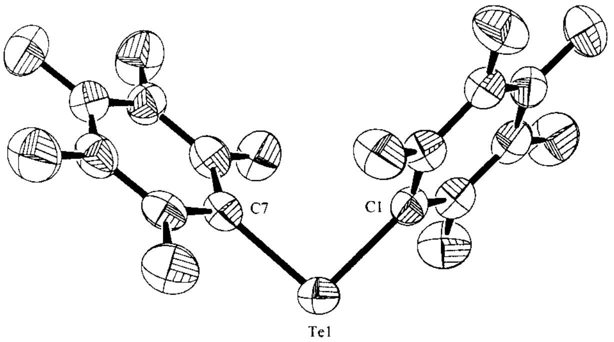


Figure 3. Molecular structure of $(C_6F_5)_2Te$ (**4**), with thermal ellipsoids at the 50% probability level. Selected bond lengths (Å) and angles (deg): Te(1)−C(1) 2.101(6), Te(1)−C(7) 2.094(6); C(7)−Te(1)−C(1) 93.3(2).

(98.8°) and **4** (93.3°) are smaller than that in $(4\text{-}CH_3C_6H_4)_2Te$ (101°). This difference between the fluorinated and the non-fluorinated compounds is due to the influence of the electronegativity of fluorine in **2** and **4**, thereby resulting in the weakening of the tellurium carbon bonds. An unusually large difference (5°) in the C−Te−C angles is observed for the compounds **2** and **4** that are practically identical in chemistry. This may be explained by the different packing effects that exist in both compounds due to the various weaker Te···F interactions, which are shorter than the tellurium−fluorine van der Waals radii[36] (vdWr TeF 3.53 Å). In **2** there is one Te···F interaction of tellurium with a fluorine atom of the CF_3 group of a second molecule (3.31(1) Å), leading to an approximate trigonal-bipyramidal geometry around Te. Since the CF_3 group is disordered, a second interaction with the same tellurium center is observed (3.40(2) Å). In **4** there exist two different Te···F interactions, one to an *o*-fluorine atom of a second molecule (3.31(1) Å) and the other to a *m*-fluorine atom of a third molecule (3.36(1) Å) that causes a distorted octahedral geometry around tellurium.

The crystal structures of a series of fluorophenyltellurium(IV) derivatives, namely, $(CF_3C_6F_4)_2TeF_2$ (**6**), **10**, **5**, **9**, and **13**, show on the first view in all cases, as expected, a Ψ-trigonal-bipyramidal geometry around tellurium.[37] The heptafluorotolyl and difluorophenyl groups and the formally nonbonding electron pair of the tellurium occupy the equatorial plane, whereas the halogen atoms are located in the axial position. In agreement with the known crystal structures of **8**, **12**, and **16**,[17,18] the Te−Hal bond length and the Hal−Te−Hal angle increase with increasing size of the halogen. This is induced by a larger sterical demand of the bromine atom as compared to a chlorine or a fluorine atom. All of the structures investigated here show secondary Te···Hal contacts of varying size, depending on the nature of the halogen, leading to hexa- and/or heptacoordination of tellurium, depending on the nature of the phenyl substituent.

The heptafluorotolyl substituted difluoride **6** forms infinite chains of Te_2F_2 dimers linked by another Te···F interaction (Figure 4), with one alternating hexa- and one heptacoordinated tellurium center. However, the structure of a similar difluoride **8** consists of isolated dimers.[17] In contrast to this, the partially fluorinated difluoride **5** represents polymeric chains (Figure 6) consisting of distorted Ψ-pentagonal-bipyramidal units around Te, resembling the structure of the non-fluorinated diphenyl-

---

(36) Bondi, A. *J. Phys. Chem.* **1964**, *68*, 441.

(37) (a) Gillespie, R. J.; Nyholm, R. S. *Q. Rev. Chem. Soc.* **1957**, *11*, 339. (b) Gillespie, R. J. *Angew. Chem., Int. Ed. Engl.* **1967**, *6*, 819. (c) Gillespie, R. J. *J. Chem. Educ.* **1970**, *47*, 18. (d) Gillespie, R. J. *J. Chem. Soc.* **1963**, 4672. (e) Gillespie, R. J. *Can. J. Chem.* **1960**, *38*, 818. (f) Gillespie, R. J. *Can. J. Chem.* **1961**, *39*, 318.

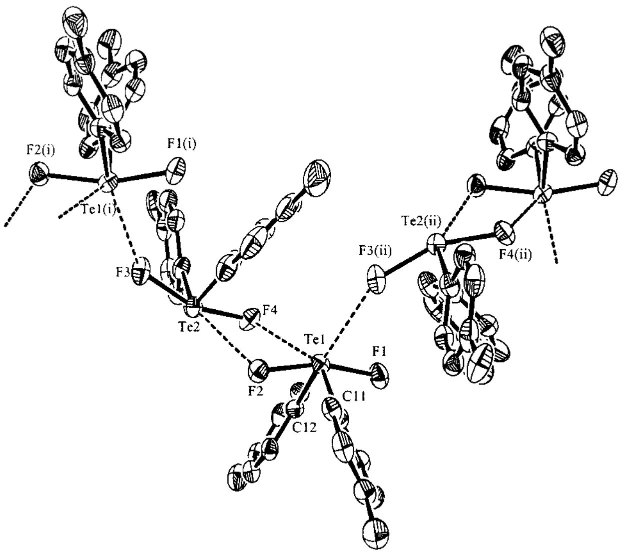


Figure 4. Molecular structure of $(CF_3C_6F_4)_2TeF_2$ (**6**), with thermal ellipsoids at the 50% probability level. Fluorine atoms of the $CF_3C_6F_4$ substituent omitted and only TeF_2C_2 units labeled for clarity. Selected bond lengths (Å) and angles (deg): Te(1)−F(1) 1.957(3), Te(1)−F(2) 1.995(3), Te(2)−F(3) 1.981(3), Te(2)−F(4) 1.983(3), Te(1)···F(4) 2.891(3), Te(1)···F(3(ii)) 3.197(4), Te(2)···F(2) 2.939(3), Te(1)−C(11) 2.102(5), Te(1)−C(12) 2.115(5), Te(2)−C(13) 2.105(5), Te(2)−C(14) 2.096(6); F(1)−Te(1)−F(2) 164.7(1), C(11)−Te(1)−F(1) 85.03(1), C(12)−Te(1)−F(1) 86.5(1), C(11)−Te(1)−F(2) 84.4(1), C(12)−Te(1)−F(2) 84.4(1), C(11)−Te(1)−C(12) 99.2(1), F(3)−Te(2)−F(4) 159.9(1), C(13)−Te(2)−F(3) 84.7(1), C(14)−Te(2)−F(3) 84.8(1), C(13)−Te(2)−F(4) 81.5(1), C(14)−Te(2)−F(4) 84.7(1), C(13)−Te(2)−C(14) 105.4(2), F(1)−Te(1)···F(3(*ii*)) 82.4(1), F(2)−Te(1)···F(3(*ii*)) 109.0(1), C(11)−Te(1)···F(3(*ii*)) 91.8(2), C(12)−Te(1)···F(3(*ii*)) 163.7(1), Te(1)−F(2)···Te(2) 112.6(1), Te(2)−F(3)···Te(1(*i*)) 144.7(2), with $i = 1 - x, y + \frac{1}{2}, 2 - z$ and $ii = 1 - x, y - \frac{1}{2}, 2 - z$.

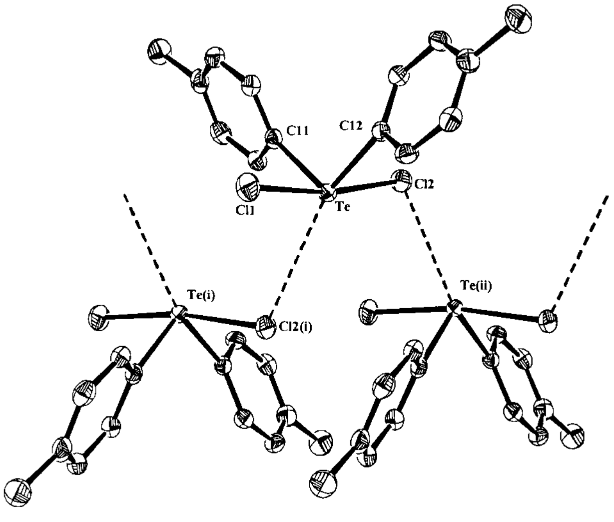


Figure 5. Molecular structure of $(CF_3C_6F_4)_2TeCl_2$ (**10**), with thermal ellipsoids at the 50% probability level. Fluorine atoms of the $CF_3C_6F_4$ substituent omitted and only $TeCl_2C_2$ units labeled for clarity. Selected bond lengths (Å) and angles (deg): Te−Cl(1) 2.4222(7), Te−Cl(2) 2.5040(7), Te−C(11) 2.111(3), Te−C(12) 2.115(2), Te···Cl(2(*i*)) 3.4207(7); Cl(1)−Te−Cl(2) 170.86(3), C(11)−Te−Cl(1) 89.21(7), C(11)−Te−Cl(2) 85.65(7), C(11)−Te−C(12) 103.7(1), C(12)−Te−Cl(1) 87.66(7), C(12)−Te−Cl(2) 86.23(7), Cl(2)−Te···Cl(2(*i*)) 103.60(2), Te−Cl(2) ···Te(*ii*) 111.40(2), C(11)−Te···Cl(2(*i*)) 84.22(2) with $i = 1\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$ and $ii = 1\frac{1}{2} - x, y + \frac{1}{2}, \frac{1}{2} - z$.

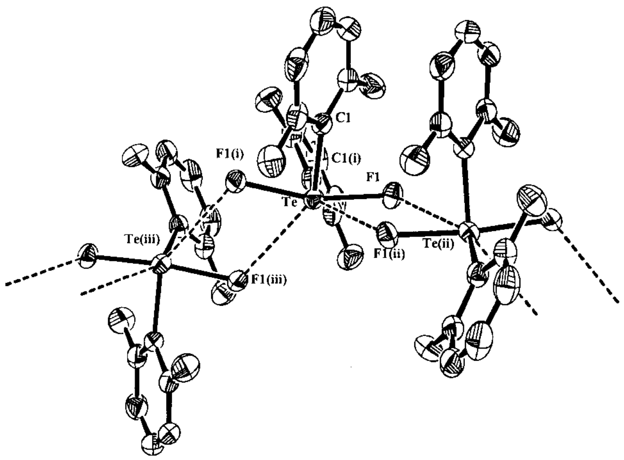


Figure 6. Molecular structure of $(C_6F_2H_3)_2TeF_2$ (**5**), with thermal ellipsoids at the 50% probability level. Only TeF_2C_2 units labeled for clarity. Selected bond lengths (Å) and angles (deg): Te−F(1) 1.999(1), Te···F(1(*iii*)) 2.999(1), Te(*ii*)···F(1(*i*)) 2.999(1), Te−C(1) 2.093(2); F(1)−Te−F(1(*i*)) 163.21(7), C(1)−Te−F(1) 83.86(6), C(1)−Te−F(1) 85.73(6), C(1)−Te−C(1(*i*)) 103.2(1), F(1(*i*))-Te···F(1(*iii*)) 62.06(5), Te−F(1(*i*))···Te(*ii*) 117.93(5), F(1(*iii*))-Te(*ii*)···F(*i*) 62.07(5), Te(*ii*)···F(1(*i*))-Te 117.93(5) with $i = \frac{1}{4} - y, \frac{1}{4} - x, \frac{1}{4} - z$; $ii = \frac{1}{2} - x, y, -z$; and $iii = \frac{1}{4} - y, x - \frac{1}{4}, z + \frac{1}{4}$.

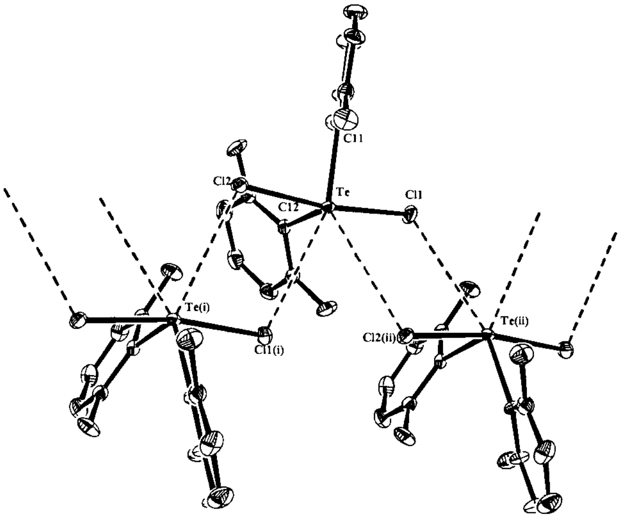


Figure 7. Molecular structure of $(C_6F_2H_3)_2TeCl_2$ (**9**), with thermal ellipsoids at the 50% probability level. Only $TeCl_2C_2$ units labeled for clarity. Selected bond lengths (Å) and angles (deg): Te−Cl(1) 2.501(1), Te−Cl(2) 2.482(1), Te−C(11) 2.111(5), Te−C(12) 2.105(4), Te···Cl(2(*i*)) 3.729(1), Cl(2)···Te(*i*) 3.639(1); Cl(1)−Te−Cl(2) 170.62(4), C(11)−Te−C(12) 103.7(1), Cl(1)−Te−C(11) 85.6(1), Cl(1)−Te−C(12) 88.1(1), Cl(2)−Te−C(11) 86.3(1), Cl(2)−Te−C(12) 89.4(1), Cl(1(*i*))···Te−Cl(2) 70.50(3), Te−Cl(2)···Te(*i*) 109.25(1), Cl(2)···Te(7)−Cl(1(*i*)) 68.67(3), Te(*i*)-Cl(1(*i*))···Te 111.50(4) with $i = -x - \frac{1}{2}, y - \frac{1}{2}, z$ and $ii = -x - \frac{1}{2}, y + \frac{1}{2}, z$.

tellurium(IV) difluoride $(C_6H_5)_2TeF_2$.[38] Two identical Te···F interactions in the Te_2F_2 units are found for **5** (2.999(1) Å), and two different but even shorter interactions are present in the dimeric Te_2F_2 units of **6** (2.891(3)/2.939(3) Å) along with a weaker linking Te···F interaction (3.197(4) Å), which connects the dimers. These distances are ~1 Å longer than the regular TeF bond lengths (1.96−2.00 Å) but significantly shorter than the tellurium−fluorine van der Waals radii (vdWr TeF 3.53 Å).

The structure of the heptafluorotolyl substituted dichloride **10** (Figure 5) consists of chains linked by Te···Cl contacts involving three molecules (3.4207(7) Å; vdWr TeCl 3.81 Å). Again, different motifs are found in the structure of the partially fluorinated dichloride **9** (Figure 7), where Te_2Cl_2 dimers with Te···Cl contacts, similar to **5** but of a weaker nature, exist (3.639(1)/3.729(1) Å). Similar Te···Br contacts of comparable

---

(38) Berry, F. J.; Edwards, A. J. *J. Chem. Soc., Dalton Trans.* **1980**, 2306.

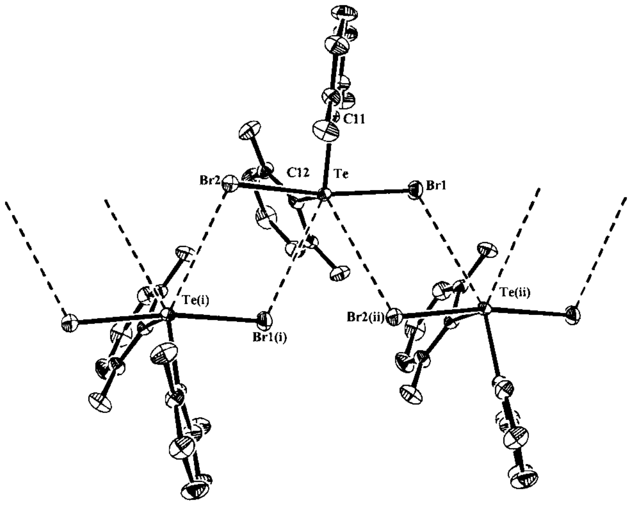


Figure 8. Molecular structure of $(C_6F_2H_3)_2TeBr_2$ (**13**), with thermal ellipsoids at the 50% probability level. Only $TeBr_2C_2$ units labeled for clarity. Selected bond lengths (Å) and angles (deg): Te−Br(1) 2.6612(4), Te−Br(2) 2.6436(4), Te−C(11) 2.113(3), Te−C(12) 2.106(4), Te···Br(1(*i*)) 3.7509(4), Te(*i*)···Br(2) 3.8057(4); Br(1)−Te−Br(2) 171.40(1), C(11)−Te−Br(1) 85.22(8), C(12)−Te−Br(1) 89.41(9), C(11)−Te−Br(2) 86.59(8), C(12)−Te−Br(2) 90.00(9), C(11)−Te−C(12) 103.9(1), Te···Br(1(*i*))-Te(*i*) 109.99(1), Br(1(*i*))−Te(*i*)···Br(2) 70.03(1), Te(*i*)···Br(2)−Te 108.86(1), Br(2)−Te···Br(1(*i*)) 71.14(1) with $i = -x - {}^1/_2, y - {}^1/_2, z$ and $ii = -x - {}^1/_2, y + {}^1/_2, z$.

size (3.7509(4)/3.8057(4) Å; vdWr TeBr 3.91 Å), forming Te_2Br_2 units, are present in the structure of the partially fluorinated dibromide **13** (Figure 8). The tellurium−chlorine/bromine contacts are in a similar range as found for $(C_6F_5)_2TeHal_2$ (Hal_2 = Cl and Br).[18]

Additional distances shorter than the sum of the van der Waals radii exist between all *o*-fluorine atoms of the fluorophenyl groups and tellurium. These exist for the monotellurides **2** and **4** (3.2−3.3 Å), the tellurium(IV) difluorides **5** and **6** (3.0−3.1 Å), and the tellurium(IV) dichlorides **9** and **10** as well as the dibromide **13** (3.3−3.4 Å). Similar values are reported or found for perfluoroaryl substituted iodine compounds, with iodine of comparable size to tellurium.[39,40]

Conclusion

The syntheses, spectroscopic, and structural studies of bis(polyfluorophenyl) tellurides and their corresponding tellurium(IV) dihalides have been described. Dynamic ^{19}F NMR spectroscopic investigations of the latter allowed the determination of the coalescence temperatures, T_c, and calculation of the free enthalpies, $\Delta G^\ddagger$, of the restricted rotation around the TeC bonds. The first crystal structures of fluoroaromatic monotellurides and some new polyfluorophenyltellurium(IV) dihalides were determined. The tellurium(IV) compounds have trigonal-bipyramidal arrangement around the tellurium atom and exhibit additional characteristic intermolecular interactions between tellurium and the halogens, creating polymeric structures in the crystal lattice. Weaker intermolecular Te···F interactions from the fluoroaromatic substituents are also present in the structures of the monotellurides.

Experimental Section

Materials. The solvents THF, chloroform, and diethyl ether are distilled and stored under dry nitrogen prior to use. All other chemicals are used as received (Aldrich, Fluorochem, and ABCR). Na_2Te and $CF_3C_6F_4OC_6F_4Br$ were prepared by literature methods.[28,40]

General Considerations. All reactions are carried out with standard Schlenk techniques and under nitrogen as an inert gas. Yields are not optimized. Raman spectra are recorded on a Perkin−Elmer Spectrum 2000 NIR FT-Raman spectrometer as neat solids; the infrared spectra are recorded on a Nicolet 520 Fourier transform infrared (FT-IR) spectrometer between KBr pellets. ^1H, ^{19}F, $^{13}C\{^1H\}$, and ^{125}Te NMR spectra are obtained on a JEOL EX 400 or JEOL Eclipse 400 instrument using $CDCl_3$ as solvent, except where otherwise indicated. The chemical shifts are with respect to $(CH_3)_4Si$, $CFCl_3$, and $(CH_3)_2Te$. Due to the temperature dependence of the ^{125}Te shifts, all samples were recorded at 25 °C and without a decoupler, if not otherwise indicated. Long time NMR measurements (more than 12 h) of the diaryltellurium(IV) dibromides are recorded in non-chlorine-containing solvents, because of possible bromine−chlorine exchange. Mass spectra are recorded on Finnigan MAT 95Q spectrometer using electron impact (EI) techniques. Multi-isotope-containing fragments refer to the isotope with the highest abundance. Elemental analyses are performed in-house. Melting points are determined on a Büchi B540 instrument.

X-ray Crystallography. For **4** an Enraf Nonius CAD 4 diffractometer was employed for data collection using Mo Kα radiation. The structure was solved by direct methods (SHELXS86), and was refined by means of the full matrix least-squares procedures using SHELXL93 (Table 2). All atoms were refined anisotropically. Crystal data for **2**, **5**, **6**, **9**, **10**, and **13** were recorded on a Stoe IPDS area detector using Mo Kα radiation. The structures were solved by direct methods (SIR97) and were refined using SHELXL93 (Table 2). All non-hydrogen atoms were refined anisotropically.

General Procedure for the Preparation of R_2Te (R = $C_6F_2H_3$, $CF_3C_6F_4$, $CF_3C_6F_4OC_6F_4$, and C_6F_5). Into a suspension of 37 mmol of Na_2Te in 50 mL of THF, 76 mmol of the bromobenzene RBr is added, within a period of 30 min, at −30 °C. After being stirred at this temperature for 1 h, the mixture is heated for 18 h under reflux. The mixture is then cooled to ambient temperature, and a black precipitate is filtered off and washed twice with 15 mL of THF. From the remaining light orange solution, the solvent is removed under vacuum. The crude products are sublimed for purification, between 50 and 140 °C/0.05 mbar, to obtain the pure diaryl tellurides as odorless pale yellow crystalline solids.

Properties and Spectral Data for $(C_6F_2H_3)_2Te$ (1). Sublimed at 70 °C/0.05 mbar, yield 68%, mp 83−84 °C; IR 1664 w, 1600 s, 1591 s, 1576 s, 1566 s, 1540 m, 1455 vs, 1384 w, 1369 w, 1303 w, 1282 m, 1268 m, 1231 s, 1193 w, 1155 m, 1083 m, 1035 w, 982 vs, 882 w, 787 vs, 780 vs, 754 m, 721 s, 699 m, 556 m, 548 m, 535 m, 506 m, 308 m cm^{-1}; Raman 3090 (45), 3030 (10), 1601 (30), 1565 (5), 1455 (5), 1269 (35), 1152 (20), 1084 (15), 1036 (25), 779 (5), 756 (15), 597 (10), 558 (100), 537 (15), 502 (5), 385 (25), 311 (10), 241 (40), 213 (70), 187 (10), 126 (15) cm^{-1}; ^1H NMR δ 7.28 (4-H, m, 1H), 6.88 (3-H, m, 2H); ^{19}F NMR δ −89.5 (2-F, m); $^{13}C\{^1H\}$ NMR δ 164.2 (C2, dm, $^1J_{C-F}$ = 245.2 Hz), 131.8 (C4, t, $^3J_{C-F}$ = 10.0 Hz), 110.9 (C3, dm, $^2J_{C-F}$ = 26.0 Hz), 89.3 (C1, t, $^2J_{C-F}$ = 30.0 Hz); ^{125}Te NMR δ 212 (quin, $^3J_{Te-F}$ = 17.1 Hz); MS (EI) (*m*/*e* (^{130}Te) (species, intensity)) 356 (M^+, 99), 336 (M^+ − HF, 5), 243 ($C_6H_3F_2Te^+$, 50), 226 (M^+ − Te, 100), 130 (Te^+, 5), 113 ($C_6H_3F_2^+$, 30), 94 ($C_6H_3F^+$, 10). Anal. Calcd for $C_{12}H_6F_4Te$: C, 40.7; H, 1.8. Found: C, 40.6; H, 1.8.

Properties and Spectral Data for $(CF_3C_6F_4)_2Te$ (2). Sublimed at 90 °C/0.05 mbar, yield 80%, mp 85−86 °C; IR 1643 s, 1597 m, 1570 w, 1564 w, 1540, 1481 vs, 1470 vs, 1411 m, 1385 w, 1353 m, 1333 vs, 1325 vs, 1288 w, 1261 w, 1199 s, 1180 s, 1157 vs, 972 vs, 922 m, 787 m, 715 s, 642 w, 547 vw, 500 vw, 424 vw, 418 vw cm^{-1}; Raman 1645 (75), 1598 (10), 1384 (45), 1324 (10), 1266 (5), 1179 (10), 1147 (10), 926 (5), 790 (15), 715 (25), 647 (5), 539 (10), 501 (100), 443 (25), 403 (40), 310 (15), 285 (10), 205 (15), 189 (15), 116 (35) cm^{-1}; ^{19}F NMR δ −57.0 (4-CF_3, t, 3F, $^4J_{F-F}$ = 20.8 Hz), −115.3 (2-F, m, 2F), −138.2 (3-F, m, 2F); ^{13}C NMR δ 147.5 (C2, dm, $^1J_{C-F}$ = 245.2 Hz), 143.4 (C3, dm, $^1J_{C-F}$ = 264.4 Hz), 120.6 (CF_3, q, $^1J_{C-F}$ = 275.2 Hz), 111.7 (C4, m), 96.2 (C1, t, $^2J_{C-F}$ = 30.0 Hz); ^{125}Te NMR δ 397 (quin, $^3J_{Te-F}$ = 34.3 Hz); MS (EI) (*m*/*e* (^{130}Te) (species, intensity)) 564 (M^+, 100), 545 (M^+ − F, 10), 347 ($CF_3C_6F_4Te^+$, 50), 217

---

(39) Frohn, H. J.; Görg, S.; Henkel, G.; Läge, M. *Z. Anorg. Allg. Chem.* **1995**, *621*, 1251.

(40) Klapötke, T. M.; Krumm, B.; Polborn, K. *Z. Anorg. Allg. Chem.* **2000**, *626*, 2047 and references therein.

($CF_3C_6F_4^+$, 10), 198 ($CF_3C_6F_3^+$, 20), 179 ($CF_3C_6F_2^+$, 36), 148 ($C_6F_4^+$, 5), 130 (Te^+, 5). Anal. Calcd for $C_{14}F_{14}Te$: C, 29.9. Found: C, 29.9.

Properties and Spectral Data for $(CF_3C_6F_4OC_6F_4)_2Te$ (3). Sublimed at 140 °C/0.05 mbar, yield 41%, mp 93−95 °C; IR 1659 s, 1629 s, 1512 vs, 1485 vs, 1430 m, 1384 m, 1344 s, 1322 w, 1266 w, 1228 vs, 1192 s, 1154 vs, 1117 s, 1026 s, 1000 vs, 974 s, 918 w, 877 s, 836 w, 809 w, 787 m, 746 w, 717 s, 695 w, 639 m, 567 w, 550 w, 537 w, 496 w, 439 w, 409 w, 373 w cm^{-1}; Raman 1832 (20), 1660 (50), 1631 (60), 1429 (15), 1390 (30), 718 (40), 538 (35), 496 (100), 441 (40), 389 (55), 304 (5) cm^{-1}; ^{19}F NMR δ −56.4 (4′-CF_3, t, 3F, $^4J_{F-F}$ = 21.6 Hz), −115.4 (2-F, m, 2F), −139.8 (3′-F, m, 2F), −153.6 (3-F, m, 2F), −154.8 (2′-F, m, 2F); ^{13}C NMR δ 147.7 (dm, $^1J_{C-F}$ = 252.2 Hz)/144.8 (dm, $^1J_{C-F}$ = 262.1 Hz)/140.3 (dm, $^1J_{C-F}$ = 244.5 Hz)/139.5 (dm, $^1J_{C-F}$ = 256.8 Hz) (CF, dm), 137.7/136.0 (CO, m), 120.6 (CF_3, q, $^1J_{C-F}$ = 274.4 Hz), 106.5 (CCF_3, m), 87.1 (C1, t, $^2J_{C-F}$ = 32.3 Hz); ^{125}Te NMR δ 326 (quin, $^3J_{Te-F}$ = 46.4 Hz); MS (EI) (*m*/*e* (^{130}Te) (species, intensity)) 892 (M^+, 48), 873 (M^+ − F, 6), 728 (M^+ − OC_6F_4, 100), 709 (M^+ − F − OC_6F_4 , 15), 511 (M^+ − $CF_3C_6F_4OC_6F_4$, 65), 439 (M^+ − F − 2$CF_3C_6F_4$, 20), 381 ($CF_3C_6F_4OC_6F_4^+$, 32), 362 ($CF_2C_6F_4OC_6F_4^+$, 18), 217 ($CF_3C_6F_4^+$, 30), 198 ($CF_3C_6F_3^+$, 25), 148 ($C_6F_4^+$, 50), 130 (Te^+, 10). Anal. Calcd for $C_{26}F_{22}O_2Te$: C, 35.1. Found: C, 35.1.

Properties and Spectral Data for $(C_6F_5)_2Te$ (4). Sublimed at 50 °C/0.05 mbar, yield 68%, mp 57−58 °C; IR 1635 m, 1549 w, 1512 vs, 1481 vs, 1383 m, 1280 w, 1141 w, 1086 s, 1002 m, 971 s, 795 m, 719 w, 621 m cm^{-1}; Raman 1636 (30), 1515 (5), 1480 (5), 1393 (10), 1271 (10), 1138 (5), 1086 (10), 970 (5), 800 (5), 624 (10), 585 (55), 492 (100), 444 (45), 388 (60), 355 (25), 283 (15), 233 (15), 201 (55), 181 (20), 140 (25), 123 (25), 84 (35) cm^{-1}; ^{19}F NMR δ −115.4 (2-F, m, 2F), −149.2 (4-F, t, 1F, $^3J_{F-F}$ = 19.0 Hz), −159.0 (3-F, m, 2F); ^{13}C NMR δ 147.8 (C2, dm, $^1J_{C-F}$ = 250.7 Hz), 143.0 (C3, dm, $^1J_{C-F}$ = 257.0 Hz), 137.0 (C4, dm, $^1J_{C-F}$ = 263.7 Hz), 85.0 (C1, tm, $^2J_{C-F}$ = 29.1 Hz); ^{125}Te NMR δ 305 (quinquin, $^3J_{Te-F}$ = 49.3 Hz, $^4J_{Te-F}$ = 7.7 Hz); MS (EI) (*m*/*e* (^{130}Te) (species, intensity)) 464 (M^+, 100), 445 (M^+ − F, 2), 334 (M^+ − Te, 4), 297 ($C_6F_5Te^+$, 88), 167 ($C_6F_5^+$, 16), 148 ($C_6F_4^+$, 15), 130 (Te^+, 12), 117 ($C_5F_3^+$, 13). Elemental analysis given in ref 10.

General Procedure for the Preparation of R_2TeF_2 (R = $C_6F_2H_3$, $CF_3C_6F_4$, $CF_3C_6F_4OC_6F_4$, and C_6F_5). Into a solution of 0.7 mmol of diaryl telluride, R_2Te, in 15 mL of $CFCl_3$, 0.7 mmol of xenon difluoride is added at 0 °C. After being stirred for 15 min at 0 °C and further for 30 min at 25 °C, the volatile materials were removed and colorless powders were obtained in quantitative yield. The diaryltellurium(IV) difluorides are analytically pure.

Properties and Spectral Data for $(C_6F_2H_3)_2TeF_2$ (5). Melting point 196−199 °C (dec); IR 3091 m, 1668 w, 1648 vw, 1610 vs, 1596 s, 1585 vs, 1546 m, 1528 w, 1464 vs, 1444 s, 1385 w, 1315 w, 1287 m, 1272 s, 1234 vs, 1152 m, 1142 w, 1125 w, 1088 s, 1042 w, 992 vs, 970 m, 888 m, 883 w, 783 vs, 756 s, 699 m, 592 w, 559 m, 534 m, 504 m, 494 m, 466 m, 382 m, 324 w, 307 m cm^{-1}; Raman 3092 (70), 3048 (10), 1610 (15), 1598 (10), 1587 (10), 1465 (10), 1447 (5), 1291 (10), 1271 (45), 1154 (25), 1090 (10), 1043 (25), 994 (5), 757 (20), 703 (5), 591 (15), 560 (100), 540 (25), 506 (25), 476 (60, ν_{TeF}), 383 (25), 316 (5), 256 (45), 214 (35), 185 (30), 150 (30), 135 (35), 105 (20) cm^{-1}; ^{1}H NMR δ 7.58 (3-H, m, 2H), 7.12 (4-H, m, 1H); ^{19}F NMR δ −98.4 (2-F, m, 2F, $^3J_{Te-F}$ = 96.2 Hz), −111.2 (TeF, quin, 2F, $^4J_{F-F}$ = 6.9 Hz, $^1J_{Te-F}$ = 249.7 Hz); ^{13}C NMR (DMSO-d_6) δ 162.7 (C2, dd, $^1J_{C-F}$ = 250.2 Hz), 135.6 (C4, t, $^3J_{C-F}$ = 10.4 Hz), 116.8 (C3, tm, $^2J_{C-F}$ = 27.6 Hz), 112.9 (C1, dd, $^2J_{C-F}$ = 23.4 Hz, $^4J_{C-F}$ = 3.1 Hz); ^{125}Te NMR (DMSO-d_6) δ 1085 (tquin, $^1J_{Te-F}$ = 565 Hz, $^3J_{Te-F}$ = 64.7 Hz); MS (EI) (*m*/*e* (^{130}Te) (species, intensity)) 394 (M^+, 5), 375 (M^+ − F, 20), 356 (M^+ − 2F, 40), 281 (M^+ − $C_6H_3F_2$, 50), 262 ($C_6H_3F_2TeF^+$, 100), 243 ($C_6H_3F_2Te^+$, 25), 226 (M^+ − TeF_2, 28), 149 (TeF^+, 5), 130 (Te^+, 5), 113 ($C_6H_3F_2^+$, 16). Anal. Calcd for $C_{12}H_6F_6Te$: C, 36.8; H, 1.5. Found: C, 36.7; H, 1.7.

Properties and Spectral Data for $(CF_3C_6F_4)_2TeF_2$ (6). Melting point 154−156 °C; IR 1701 m, 1647 m, 1603 w, 1539 w, 1492 vs, 1460 vs, 1418 m, 1384 w, 1326 vs, 1259 m, 1185 s, 1157 s, 1079 w, 1054 w, 980 vs, 927 s, 793 m, 761 w, 742 w, 715 s, 668 w, 651 w, 531 m, 510 m, 495 m, 476 m (ν_{TeF}), 428 m cm^{-1}; Raman 1649 (20), 1398 (10), 1327 (10), 1166 (5), 927 (15), 793 (10), 715 (30), 651 (10), 531 (20), 502 (100), 476 (20, ν_{TeF}), 439 (30), 402 (30), 309 (15), 211 (20), 166 (30), 124 (35) cm^{-1}; ^{19}F NMR δ −57.2 (4-CF_3, t, 3F, $^4J_{F-F}$ = 22.5 Hz), −101.3 (TeF, quin, 2F, $^4J_{F-F}$ = 8.7 Hz, $^1J_{Te-F}$ = 296.5 Hz), −124.8 (2-F, m, 2F), −135.5 (3-F, m, 2F); ^{13}C NMR δ 146.2 (C2, dm, $^1J_{C-F}$ = 255.2 Hz), 144.4 (C3, dm, $^1J_{C-F}$ = 269.1 Hz), 120.6 (CF_3, q, $^1J_{C-F}$ = 276.0 Hz), 119.8 (C4, m), 96.2 (C1, qt, $^2J_{C-F}$ = 36.1 Hz, $^3J_{C-F}$ = 12.3 Hz); ^{125}Te NMR δ 1051 (tm, $^1J_{Te-F}$ = 303 Hz); MS (EI) (*m*/*e* (^{130}Te) (species, intensity)) 602 (M^+, 10), 583 (M^+ − F, 10), 564 (M^+ − 2F, 100), 545 (M^+ − 3F, 20), 434 (M^+ − TeF_2, 7), 385 (M^+ − $CF_3C_6F_4$, 45), 366 ($CF_3C_6F_4TeF^+$, 12), 347 ($CF_3C_6F_4Te^+$, 65), 217 ($CF_3C_6F_4^+$, 30), 198 ($CF_3C_6F_3^+$, 20), 179 ($CF_3C_6F_2^+$, 35), 149 (TeF^+, 10), 130 (Te^+, 12). Anal. Calcd for $C_{14}F_{16}Te$: C, 28.0. Found: C, 28.3.

Properties and Spectral Data for $(CF_3C_6F_4OC_6F_4)_2TeF_2$ (7). Melting point 199−202 °C; IR 1659 m, 1634 m, 1513 vs, 1494 vs, 1430 m, 1395 m, 1344 vs, 1323 w, 1279 w, 1262 m, 1227 vs, 1192 s, 1155 s, 1120 m, 1083 m, 1029 m, 1001 s, 982 s, 877 s, 836 w, 804 m, 794 m, 717 s, 640 m, 514 w, 474 m (ν_{TeF}), 407 w cm^{-1}; Raman 1660 (30), 1635 (30), 1488 (10), 1431 (15), 1403 (15), 1346 (10), 784 (10), 718 (25), 571 (10), 537 (20), 498 (100), 477 (45, ν_{TeF}), 440 (35), 389 (45), 287 (15), 147 (10), 119 (10) cm^{-1}; ^{19}F NMR δ −56.4 (4′-CF_3, t, 3F, $^4J_{F-F}$ = 21.7 Hz), −101.1 (TeF, quin, 2F, $^4J_{F-F}$ = 8.7 Hz, $^1J_{Te-F}$ = 328.2 Hz), −126.2 (2-F, m, 2F), −139.2 (3′-F, m, 2F), −152.0 (3-F, m, 2F), −154.3 (2′-F, m, 2F); ^{125}Te NMR δ 1070 (m); MS (EI) (*m*/*e* (^{130}Te) (species, intensity)) 930 (M^+, 5), 911 (M^+ − F, 5), 892 (M^+ − 2F, 100), 873 (M^+ − 3F, 10), 762 (M^+ − TeF_2, 55), 743 (M^+ − F − TeF_2, 11), 728 (M^+ − 2F − OC_6F_4, 5), 659 (M^+ − 2F − $CF_3C_6F_4O$, 10), 549 (M^+ − $CF_3C_6F_4OC_6F_4$, 8), 530 ($CF_3C_6F_4OC_6F_4TeF^+$, 15), 511 ($CF_3C_6F_4OC_6F_4Te^+$, 35), 381 ($CF_3C_6F_4OC_6F_4^+$, 30), 363 ($CF_2C_6F_4$-$OC_6F_4^+$, 10). Anal. Calcd for $C_{26}F_{24}O_2Te$: C, 33.7. Found: C, 35.1.

Properties and Spectral Data for $(C_6F_5)_2TeF_2$ (8). Melting point 160−162 °C; IR 1642 s, 1594 m, 1518 vs, 1500 vs, 1396 m, 1377 m, 1292 m, 1152 m, 1094 vs, 1010 m, 979 vs, 806 m, 757 w, 721 w, 623 w, 587 w, 528 m, 489 s, 477 m (ν_{TeF}), 442 w, 381 m cm^{-1}; Raman 1640 (25), 1517 (10), 1404 (10), 1151 (10), 1090 (10), 808 (10), 587 (70), 529 (30), 507 (55), 493 (100), 479 (35, ν_{TeF}), 444 (65), 395 (55), 353 (30), 282 (20), 260 (20), 208 (25), 183 (30), 165 (25), 131 (30) cm^{-1}; ^{19}F NMR δ −101.2 (TeF, quin, 2F, $^4J_{F-F}$ = 8.6 Hz, $^1J_{Te-F}$ = 336.4 Hz), −126.6 (2-F, m, 2F), −144.0 (4-F, m, 1F), −156.0 (3-F, m, 2F); ^{13}C NMR δ 146.6 (C2, dm, $^1J_{C-F}$ = 254.4 Hz), 144.8 (C3, dm, $^1J_{C-F}$ = 262.1 Hz), 137.8 (C4, dm, $^1J_{C-F}$ = 254.4 Hz), 111.0 (C1, m); ^{125}Te NMR δ 1080 (tquin, $^1J_{Te-F}$ = 340 Hz, $^3J_{Te-F}$ = 77.7 Hz); MS (EI) (*m*/*e* (^{130}Te) (species, intensity)) 502 (M^+, 9), 483 (M^+ − F, 23), 464 (M^+ − 2F, 100), 445 (M^+ − 3F, 7), 335 (M^+ − C_6F_5, 53), 316 ($C_6F_5TeF^+$, 64), 297 ($C_6F_5Te^+$, 99), 167 ($C_6F_5^+$, 96), 148 ($C_6F_4^+$, 24), 130 (Te^+, 18), 117 ($C_5F_3^+$, 50). Anal. Calcd for $C_{12}F_{12}Te$: C, 28.8. Found: C, 29.0.

General Procedure for the Preparation of R_2TeCl_2 (R = $C_6F_2H_3$, $CF_3C_6F_4$, $CF_3C_6F_4OC_6F_4$, and C_6F_5). Into 15 mL of sulfuryl chloride is added 0.9 mmol of R_2Te at 25 °C. When this mixture was stirred for 18 h and the volatile materials were removed, analytically pure diaryltellurium(IV) dichlorides were formed as colorless powders in quantitative yield.

Properties and Spectral Data for $(C_6F_2H_3)_2TeCl_2$ (9). Melting point 285 °C (dec); IR 3092 m, 3063 w, 1634 w, 1602 vs, 1591 s, 1579 s, 1539 w, 1464 vs, 1457 vs, 1443 m, 1382 w, 1307 w, 1291 w, 1271 w, 1237 m, 1175 vw, 1150 m, 1078 m, 1034 w, 996 vs, 987 vs, 890 w, 796 s, 790 s, 753 m, 695 m, 554 m, 530 w, 504 m cm^{-1}; Raman 3089 (15), 3075 (10), 1603 (5), 1269 (10), 1147 (5), 1080 (10), 1035 (10), 755 (10), 555 (20), 506 (5), 378 (10), 276 (100, ν_{TeCl}), 215 (10), 151 (15), 120 (20) cm^{-1}; ^{1}H NMR δ 7.60 (3-H, m, 2H), 7.15 (4-H, t, 1H, $^3J_{H-H}$ = 7.9 Hz); ^{19}F NMR δ −93.5 (2-F, m, 1F, $^3J_{Te-F}$ = 96.2 Hz), −98.4 (2-F, m, 1F); ^{13}C NMR (DMSO-d_6) δ 162.7 (C2, dm, $^1J_{C-F}$ = 257.5 Hz), 135.8 (C4, t, $^3J_{C-F}$ = 10.5 Hz), 112.7 (C3, dm, $^2J_{C-F}$ = 21.5 Hz), 111.2 (C1, t, $^2J_{C-F}$ = 25.8 Hz); ^{125}Te NMR (DMSO-d_6) δ 693 (m); MS (EI) (*m*/*e* (^{35}Cl/^{130}Te) (species, intensity)) 391 (M^+ − Cl, 6), 356 (M^+ − 2Cl, 100), 337 (M^+ − 2Cl − F, 1), 278 ($C_6H_3F_2TeCl^+$, 3), 243 ($C_6H_3F_2Te^+$, 49), 226 (M^+ − $TeCl_2$, 85), 130 (Te^+, 4), 113 ($C_6H_3F_2^+$, 25), 94 ($C_6H_3F^+$, 10). Anal. Calcd for $C_{12}H_6Cl_2F_4Te$: C, 33.9; H, 1.4; Cl, 16.7. Found: C, 33.4; H, 1.5; Cl, 16.9.

Properties and Spectral Data for $(CF_3C_6F_4)_2TeCl_2$ (10). Melting point 218−221 °C; IR 1643 m, 1602 m, 1532 w, 1493 w, 1395 w, 1354 m, 1323 vs, 1292 w, 1257 m, 1212 s, 1190 s, 1160 vs, 1062 vw, 1043 vw, 980 vs, 924 s, 785 w, 761 vw, 751 vw, 716 s, 646 m, 547 m, 422 w cm^{-1}; Raman 1645 (5), 1399 (5), 1327 (5), 1258 (5), 1208 (5), 1154 (5), 924 (5), 787 (5), 716 (5), 647 (5), 541 (5), 503 (30), 439 (10), 398 (15), 280 (100, ν_{TeCl}), 211 (10), 192 (10), 155 (10), 119 (35), 107 (30) cm^{-1}; ^{19}F NMR δ −57.1 (4-CF_3, t, 3F, $^4J_{F-F}$ = 22.5 Hz), −120.2/−124.8 (2,6-F, s, 1F), −134.2/−135.2 (3,5-F, s, 1F); ^{13}C NMR δ 148.0−142.6 (C2, C3, br), 120.0 (CF_3, q, $^1J_{C-F}$ = 277.7 Hz), 117.8 (C4, m), 115.0 (C1, m); ^{125}Te NMR δ 656 (m); MS (EI) (*m*/*e* (^{35}Cl/^{130}Te) (species, intensity)) 599 ($M^+ - Cl$, 5), 564 ($M^+ - 2Cl$, 100), 545 ($M^+ - 2Cl - F$, 17), 347 ($CF_3C_6F_4Te^+$, 83), 328 ($CF_3C_6F_3Te^+$, 6), 217 ($CF_3C_6F_4^+$, 13), 198 ($CF_3C_6F_3^+$, 31), 179 ($CF_3C_6F_2^+$, 62), 149 (TeF^+, 10), 130 (Te^+, 10), 117 ($C_5F_3^+$, 7), 93 ($C_3F_3^+$, 5), 69 (CF_3^+, 7). Anal. Calcd for $C_{14}Cl_2F_{14}Te$: C, 26.6; Cl, 11.2. Found: C, 26.1; Cl, 11.6.

Properties and Spectral Data for $(CF_3C_6F_4OC_6F_4)_2TeCl_2$ (11). Melting point 183−185 °C; IR 1659 m, 1630 m, 1512 vs, 149 vs, 1430 m, 1394 m, 1344 s, 1323 w, 1277 w, 1263 w, 1227 s, 1193 m, 1157 m, 1121 m, 1028 m, 1000 s, 981 s, 922 vw, 876 m, 837 w, 808 w, 787 w, 717 s, 670 w, 639 m cm^{-1}; Raman 1659 (20), 1631 (25), 1488 (10), 1428 (10), 1403 (10), 718 (15), 536 (10), 501 (30), 493 (30), 442 (15), 388 (20), 281 (100, ν_{TeCl}), 148 (5), 123 (10) cm^{-1}; ^{19}F NMR δ −56.4 (4′-CF_3, t, 3F, $^4J_{F-F}$ = 21.7 Hz), −121.5/-125.0 (2,6-F, s, 1F), −139.1 (3′-F, m, 2F), −150.7/-151.8 (3,5-F, s, 1F), −154.0 (2′-F, m, 2F); ^{125}Te NMR δ 665 (m); MS (EI) (*m*/*e* (^{35}Cl/^{130}Te) (species, intensity)) 927 ($M^+ - Cl$, 1), 892 ($M^+ - 2Cl$, 100), 873 ($M^+ - 2Cl - F$, 10), 762 ($M^+ - TeCl_2$, 15), 728 ($M^+ - 2Cl - OC_6F_4$, 5), 659 ($M^+ - 2Cl - CF_3C_6F_4O$, 3), 511 ($CF_3C_6F_4OC_6F_4Te^+$, 40), 381 ($CF_3C_6F_4OC_6F_4^+$, 13), 362 ($CF_2C_6F_4OC_6F_4^+$, 6), 130 (Te^+, 8). Anal. Calcd for $C_{26}Cl_2F_{22}O_2Te$: C, 32.5; Cl, 7.4. Found: C, 32.2; Cl, 8.3.

Properties and Spectral Data for $(C_6F_5)_2TeCl_2$ (12). Melting point 206−208 °C; IR 1638 s, 1518 vs, 1497 vs, 1488 vs, 1396 s, 1371 w, 1348 w, 1293 m, 1155 m, 1151 m, 1101 vs, 1076 m, 1027 w, 1004 s, 976 vs, 795 m, 615 w, 317 w cm^{-1}; Raman 1637 (10), 1517 (5), 1149 (5), 1094 (5), 796 (5), 617 (5), 586 (20), 493 (30), 442 (20), 385 (20), 349 (10), 276 (100, ν_{TeCl}), 233 (5), 208 (20), 126 (35) cm^{-1}; ^{19}F NMR δ −121.9 (2-F, m, 1F), −125.3 (2-F, m, 1F), −143.3 (4-F, t, 1F, $^3J_{F-F}$ = 21.7 Hz), −155.6 (3-F, m, 1F), −156.7 (3-F, m, 1F); ^{13}C NMR δ 147.1 ($^1J_{C-F}$ = 252.2 Hz)/145.7 ($^1J_{C-F}$ = 251.4 Hz) (C2, dm), 145.0 (C4, tm, $^1J_{C-F}$ = 263.7 Hz), 137.8 ($^1J_{C-F}$ = 256.8 Hz)/145.7 ($^1J_{C-F}$ = 256.8 Hz) (C3, dm), 108.6 (C1, t, $^2J_{C-F}$ = 25.0 Hz); ^{125}Te NMR δ 665 (tm, $^3J_{Te-F}$ = 57.7 Hz); MS (EI) (*m*/*e* (^{35}Cl/^{130}Te) (species, intensity)) 499 ($M^+ - Cl$, 5), 464 ($M^+ - 2Cl$, 89), 445 ($M^+ - 2Cl - F$, 2), 332 ($C_6F_5TeCl^+$, 8), 297 ($C_6F_5Te^+$, 100), 167 ($C_6F_5^+$, 34), 149 (TeF^+, 22), 130 (Te^+, 18), 117 ($C_5F_3^+$, 30), 93 ($C_3F_3^+$, 10). Anal. Calcd for $C_{12}Cl_2F_{10}Te$: C, 27.1; Cl, 13.3. Found: C, 26.8; Cl, 13.6.

General Procedure for the Preparation of R_2TeBr_2 (R = $C_6F_2H_3$, $CF_3C_6F_4$, $CF_3C_6F_4OC_6F_4$, and C_6F_5). Into a solution of 0.7 mmol of R_2Te in 15 mL of $CFCl_3$, 1 mmol of bromine is added over a period of 10 min at 0 °C. During the addition, a yellow powder precipitated. After the mixture was stirred further for 90 min, the volatile materials were removed and analytically pure diaryltellurium(IV) dibromides resulted in quantitative yield.

Properties and Spectral Data for $(C_6F_2H_3)_2TeBr_2$ (13). Melting point 275 °C (dec); IR 1666 w, 1642 w, 1610 vs, 1596 s, 1585 s, 1546 m, 1517 w, 1464 vs, 1443 s, 1385 w, 1315 w, 1307 w, 1287 m, 1271 s, 1233 vs, 1186 vw, 1179 vw, 1152 s, 1088 s, 1042 w, 987 vs, 969 m, 888 m, 883 m, 783 vs, 756 s, 699 s, 589 w, 559 m, 543 m, 504 m, 494 m, 466 m, 397 m, 322 m, 307 m cm^{-1}; Raman 3084 (5), 3075 (5), 3048 (5), 3031 (5), 1602 (5), 1267 (5), 1147 (5), 1079 (5), 1074 (5), 1032 (5), 754 (5), 553 (5), 379 (10), 302 (5), 249 (5), 214 (5), 171 (100, ν_{TeBr}), 148 (10), 102 (5) cm^{-1}; ^{1}H NMR δ 7.60 (3-H, m, 2H), 7.15 (4-H, t, 1H, $^3J_{H-H}$ = 7.9 Hz); ^{19}F NMR δ −91.9 (2-F, m, 1F, $^3J_{Te-F}$ = 175.1 Hz), −94.9 (2-F, m, 1F); ^{13}C NMR (DMSO-d_6) δ 161.8 (C2, dm, $^1J_{C-F}$ = 255.8 Hz), 136.0 (C4, t, $^3J_{C-F}$ = 10.8 Hz), 112.8 (C3, dm, $^2J_{C-F}$ = 23.1 Hz), 107.7 (C1, t, $^2J_{C-F}$ = 25.4 Hz); ^{125}Te NMR (DMSO-d_6) δ 650 (m); MS (EI) (*m*/*e* (^{79}Br/^{130}Te) (species, intensity)) 435 ($M^+ - Br$, 8), 356 ($M^+ - 2Br$, 100), 337 ($M^+ - 2Br - F$, 2), 243 ($C_6H_3F_2Te^+$, 48), 224 ($M^+ - TeBr_2$, 71), 149 (TeF^+, 2), 130 (Te^+, 4), 113 ($C_6H_3F_2^+$, 22), 94 ($C_6H_3F^+$, 10). Anal. Calcd for $C_{12}H_6Br_2F_4Te$: C, 28.1; H, 1.2; Br, 31.1. Found: C, 27.4; H, 1.2; Br, 31.4.

Properties and Spectral Data for $(CF_3C_6F_4)_2TeBr_2$ (14). Melting point 275−278 °C (dec); IR 1643 m, 1602 m, 1566 vw, 1532 w, 1491 vs, 1411 w, 1394 w, 1354 m, 1323 vs, 1290 w, 1256 m, 1211 s, 1186 s, 1172 s, 1160 vs, 1058 vw, 1040 vw, 980 s, 923 s, 784 w, 758 vw, 715 s, 645 m, 548 w, 502 vw, 421 w cm^{-1}; Raman 1643 (5), 1397 (5), 716 (5), 503 (10), 432 (10), 397 (5), 192 (10), 169 (100, ν_{TeBr}), 117 (5), 96 (5) cm^{-1}; ^{19}F NMR δ −57.1 (4-CF_3, t, 3F, $^4J_{F-F}$ = 23.0 Hz), −119.1/-122.5 (2,6-F, s, 1F), −134.1/−135.1 (3,5-F, s, 1F); ^{13}C NMR (C_6D_6) δ 145.0 (C2, dm, $^1J_{C-F}$ = 255.4 Hz), 141.9 (C3, dm, $^1J_{C-F}$ = 259.0 Hz), 120.1 (CF_3, q, $^1J_{C-F}$ = 275.6 Hz), 113.8 (C4, m), 113.6 (C1, m); ^{125}Te NMR δ 647 (m); MS (EI) (*m*/*e* (^{79}Br/^{130}Te) (species, intensity)) 643 ($M^+ - Br$, 1), 624 ($M^+ - Br - F$, 1), 564 ($M^+ - 2Br$, 100), 545 ($M^+ - 2Br - F$, 19), 434 ($M^+ - TeBr_2$, 2), 347 ($CF_3C_6F_4Te^+$, 81), 328 ($CF_3C_6F_3Te^+$, 5), 217 ($CF_3C_6F_4^+$, 17), 198 ($CF_3C_6F_3^+$, 20), 179 ($CF_3C_6F_2^+$, 38), 149 (TeF^+, 6), 130 (Te^+, 6). Anal. Calcd for $C_{14}Br_2F_{14}Te$: C, 23.3; Br, 22.2. Found: C, 22.8; Br, 22.3.

Properties and Spectral Data for $(CF_3C_6F_4OC_6F_4)_2TeBr_2$ (15). Melting point 195−198 °C; IR 1658 m, 1629 m, 1513 vs, 1494 vs, 1430 m, 1392 m, 1345 vs, 1323 w, 1276 w, 1263 w, 1227 vs, 1193 m, 1155 s, 1120 m, 1027 m, 1000 s, 981 m, 920 w, 877 s, 838 w, 807 w, 787 w, 717 m, 639 m cm^{-1}; Raman 1659 (10), 1628 (15), 1488 (5), 1400 (5), 718 (5), 535 (5), 502 (15), 492 (15), 442 (10), 388 (10), 299 (5), 215 (10), 200 (5), 171 (100, ν_{TeBr}) cm^{-1}; ^{19}F NMR δ −56.4 (4′-CF_3, t, 3F, $^4J_{F-F}$ = 21.7 Hz), −120.3/−124.0 (2,6-F, s, 1F), −139.0 (3′-F, m, 2F), −150.6/-151.7 (3,5-F, s, 1F), −154.0 (2′-F, m, 2F); ^{125}Te NMR δ 628 (m); MS (EI) (*m*/*e* (^{79}Br/^{130}Te) (species, intensity)) 892 ($M^+ - 2Br$, 100), 873 ($M^+ - 2Br - F$, 11), 762 ($M^+ - TeBr_2$, 73), 743 ($M^+ - TeBr_2 - F$, 16), 728 ($M^+ - 2Br - OC_6F_4$, 5), 659 ($M^+ - 2Br - CF_3C_6F_4O$, 4), 511 ($CF_3C_6F_4OC_6F_4Te^+$, 45), 460 ($CF_3C_6F_4OC_6F_4Br^+$, 36), 381 ($CF_3C_6F_4OC_6F_4^+$, 43), 362 ($CF_2C_6F_4OC_6F_4^+$, 9), 130 (Te^+, 11), 117 ($C_5F_3^+$, 17). Anal. Calcd for $C_{26}Br_2F_{22}O_2Te$: C, 29.8; Br, 15.2. Found: C, 29.5; Br, 16.4.

Properties and Spectral Data for $(C_6F_5)_2TeBr_2$ (16). Melting point 208−210 °C; IR 1637 s, 1519 vs, 1494 vs, 1487 vs, 1419 w, 1394 s, 1371 w, 1334 w, 1292 m, 1262 m, 1153 m, 1099 vs, 1075 m, 1024 w, 1001 m, 977 vs, 866 w, 792 m, 756 w, 716 w, 614 w, 586 w, 368 w cm^{-1}; Raman 1635 (10), 792 (5), 615 (5), 586 (15), 554 (5), 520 (5), 492 (20), 442 (10), 384 (10), 282 (5), 232 (5), 204 (20), 167 (100, ν_{TeBr}), 126 (5) cm^{-1}; ^{19}F NMR δ −120.7 (2-F, m, 1F), −122.4 (2-F, m, 1F), −143.2 (4-F, t, 1F, $^3J_{F-F}$ = 20.8 Hz), −155.4 (3-F, m, 1F), −156.6 (3-F, m, 1F); ^{13}C NMR δ 147.5 ($^1J_{C-F}$ = 243.7 Hz)/145.7 ($^1J_{C-F}$ = 241.4 Hz) (C2, dm), 145.1 (C4, tm, $^1J_{C-F}$ = 265.2 Hz), 138.2 ($^1J_{C-F}$ = 268.2 Hz)/137.5 ($^1J_{C-F}$ = 257.5 Hz) (C3, dm), 103.8 (C1, t, $^2J_{C-F}$ = 24.2 Hz); ^{125}Te NMR δ 626 (quinm, $^3J_{Te-F}$ = 69.8 Hz); MS (EI) (*m*/*e* (^{79}Br/^{130}Te) (species, intensity)) 543 ($M^+ - Br$, 12), 464 ($M^+ - 2Br$, 100), 376 ($C_6F_5TeBr^+$, 4), 334 ($M^+ - TeBr_2$, 4), 297 ($C_6F_5Te^+$, 83), 167 ($C_6F_5^+$, 4), 149 (TeF^+, 15), 130 (Te^+, 12), 117 ($C_5F_3^+$, 16), 93 ($C_3F_3^+$, 5). Anal. Calcd for $C_{12}Br_2F_{10}Te$: C, 23.2; Br, 25.7. Found: C, 22.9; Br, 25.9.

Acknowledgment. The University of Munich and the Fonds der Chemischen Industrie are gratefully acknowledged for financial support. We thank Mr. Ingo Schwab for his enthusiasm with the preparation of some compounds.

Supporting Information Available: Crystallographic files in CIF format for compounds **2**, **4**, **5**, **6**, **9**, **10**, and **13**. This material is available free of charge via the Internet at http://pubs.acs.org.

IC010149R