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# Threefold Configuration Tellurium(IV). Crystal Structure of Trimethyltelluronium Tetraphenylborate

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Trimethyltelluronium tetraphenylborate has been prepared by metathesis and its crystal structure determined by a single-crystal X-ray diffraction study. The salt crystallizes in the space group *Cmcm* of the orthorhombic system with four $(CH)_3TeB(C_6H_5)_4$ units per unit cell. Lattice parameters (25 °C) are $a = 11.445$ (3) Å, $b = 15.539$ (2) Å and $c = 12.955$ (4) Å. The structure was determined by Patterson and Fourier methods from automatic diffractometer data collected on an Enraf-Nonius CAD4 using Mo K$\alpha$ radiation filtered by a graphite-crystal incident-beam monochromator. Refinement by the least-squares technique led to a conventional $R$ factor (on $F$) of 0.053 for 644 reflections having $F_o^2 > 3\sigma(F_o^2)$. The trimethyltelluronium cation is pyramidal and disordered across a mirror plane such that two half-occupancy tellurium atoms, separated by 1.321 (2) Å, share two methyl carbon atoms of full occupancy and have each a methyl carbon at half occupancy. The mean Te–C distance is 2.14 (2) Å and the mean C–Te–C angle 92 (1)°. Secondary bonding interactions involving tellurium are absent, resulting in the first structural example of threefold configuration tellurium(IV). The possibility of greater stereochemical influence of the lone pair in the absence of secondary bonding is considered. The tetraphenylborate anion has crystallographic symmetry *mm* ($C_{2v}$) with normal interatomic distances and angles. Boron is out of the planes of the phenyl rings which are distorted from a regular hexagonal geometry. The structure is layered such that a trigonal bipyramid of tetraphenylborate ions surrounds each cation site. A corresponding salt, triphenyltelluronium tetraphenylborate, $(C_6H_5)_3TeB(C_6H_5)_4$, was also prepared which crystallizes in the triclinic system. Lattice parameters (25 °C) are $a = 18.610$ (8) Å, $b = 27.050$ (6) Å, $c = 15.157$ (6) Å, $\alpha = 106.20$ (2)°, $\beta = 113.96$ (3)°, and $\gamma = 96.09$ (3)°. The measured density of 1.38 g/cm$^3$ agrees with the calculated density (1.389 g/cm$^3$) for $Z = 8$.

## Introduction

It is well established that tellurium(IV) in its many and varied compounds possesses a four-, five-, or sixfold configuration.[2] The triorganotelluronium salts, $R_3TeX$, are no exception, and recent structural studies of these compounds show tellurium to have a five- or sixfold configuration with weak bonding interactions between the cation and anion.[3-7] This type of weak interaction, known as secondary bonding, has been reviewed by Alcock.[8] The most outstanding manifestation of the secondary bonding in these telluronium salts is the resulting oligomerization occurring in the solid state. Triphenyltelluronium thiocyanate, for example, is bioligomeric and consists of an ionic dimer and tetramer.[4] The corresponding cyanate[5] and chloride[6] salts are ionic tetramers. In all of these salts the anion is a potential donor, and the coordination number of tellurium is greater than that expected from the compounds's stoichiometry.

We now report the first structural study of a triorganotelluronium salt where secondary interaction is absent due to the presence of a noncoordinating counteranion, $B(C_6H_5)_4^-$. Both the trimethyltelluronium and triphenyltelluronium salts have been prepared, and that of $(CH_3)_3TeB(C_6H_5)_4$ has been studied structurally.

## Experimental Section

Trimethyltelluronium tetraphenylborate and triphenyltelluronium tetraphenylborate were prepared as voluminous precipitates by mixing equimolar aqueous solutions of $(CH_3)_3TeCl$ or $(C_6H_5)_3TeCl$ with $NaB(C_6H_5)_4$. The compounds were collected and then washed with water and recrystallized from a mixture of *N*,*N*-dimethylformamide (DMF) and water. The preparation of $(CH_3)_3TeB(C_6H_5)_4$ was greatly facilitated by using DMF as the initial solvent and adding water to crystallize the product. The preparation of $(C_6H_5)_3TeB(C_6H_5)_4$ was first reported by Wittig and Fritz, who reacted tetraphenyltellurium with triphenylborane.[9]

Unit cell parameters for both compounds were obtained by computer-centering 25 reflections followed by least-squares refinement of the setting angles. The results are reported in Table I. In the case of $(C_6H_5)_3TeB(C_6H_5)_4$, which crystallizes in the triclinic system with a relatively large unit cell, reduction of the cell revealed no hidden symmetry. Two crystals of $(CH_3)_3TeB(C_6H_5)_4$ were examined photographically and both showed *mm* symmetry. The observed systematic extinctions were $hkl$, $h + k \neq 2n$, and $h0l$, $l \neq 2n$. Space groups *Cmcm*, *Ama*2, and *Cmc*2$_1$ were considered. *Cmcm* was confirmed by refinement. Agreement of the observed and calculated densities was obtained for $Z = 4$.

**Table I.** Crystal Data (25 °C)

| | | |
|---|---|---|
| compound | $(CH_3)_3TeB(C_6H_5)_4$ | $(C_6H_5)_3TeB(C_6H_5)_4$ |
| space group | *Cmcm* | *P*1 or *P*$\bar{1}$ |
| $a$, Å | 11.445 (3) | 18.610 (8) |
| $b$, Å | 15.539 (2) | 27.050 (6) |
| $c$, Å | 12.955 (4) | 15.157 (6) |
| $\alpha$, deg | 90 | 106.20 (2) |
| $\beta$, deg | 90 | 113.96 (3) |
| $\gamma$, deg | 90 | 96.09 (3) |
| $V$, Å$^3$ | 2304 | 6483 |
| $Z$ | 4 | 8 |
| $\rho_{calcd}$, g/cm$^3$ | 1.418 | 1.389 |
| $\rho_{measd}$,[a] g/cm$^3$ | 1.41 (1) | 1.38 (1) |

[a] By flotation in carbon tetrachloride–cyclohexane mixture.

Intensity data were collected on $(CH_3)_3TeB(C_6H_5)_4$ at Molecular Structure Corp. on an Enraf-Nonius CAD4 automatic diffractometer using graphite-monochromatized Mo K$\alpha$ radiation. The prism used measured 0.15 × 0.15 × 0.13 mm. The width at half-height from $\omega$ scans was 0.15°.

A total of 1172 independent reflections were collected by the $\theta$–$2\theta$ scan technique in the range $0 < 2\theta(\text{Mo K}\alpha) > 50°$. A takeoff angle of 2.8° was used with a counter aperture width of 2.0 mm. The incident-beam collimator diameter was 0.7 mm and the crystal to detector distance was 21 cm. A variable scan rate of 4–40°/min (in $2\theta$) was used with moving-crystal moving-counter background counts where scan time per background time equaled 2.0. The scan range was from $2\theta(\text{Mo K}\alpha_1) - 0.5°$ to $2\theta(\text{Mo K}\alpha_2) + 0.5°$.

Intensities and standard deviations of intensities were calculated as described previously.[4] The factor $p$ introduced to downweight intense reflections was set to 0.05. No significant change was observed in the intensities of three check reflections measured periodically. Lorentz and polarization corrections were applied to the data. Extinction and absorption corrections were unnecessary. $\psi$ scans of the relatively uniform prism showed less than 5% variation in transmission. The calculated linear absorption coefficient (Mo K$\alpha$), $\mu$, is 13.5 cm$^{-1}$.

The structure was solved by the Patterson method which showed the position of the Te atom. Least-squares refinement of the atom position resulted in agreement factors of $R_1$ ($\sum||F_o| - |F_c||/\sum|F_o|$) = 0.30 and of $R_2$ ($[\sum w(|F_o| - |F_c|)^2/\sum wF_o^2]^{1/2}$) = 0.35. The remaining nonhydrogen atoms were located in succeeding difference Fourier syntheses. The $(CH_3)_3Te^+$ ion was found to be disordered across a mirror plane about an *mm* site in the crystal. Refinement in the acentric space group *Ama*2 did not resolve the disorder problem. The final structure, refined in space group *Cmcm*, resulted in two

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**Table II.** Atomic Positional and Thermal Properties for Atoms in $(CH_3)_3TeB(C_6H_5)_4$[a]

| atom | $x$ | $y$ | $z$ | $U_{11}$[b] | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| Te[d] | 0.05771 (9) | 0.22295 (9) | 1/4 | 0.0533 (5) | 0.0871 (8) | 0.0803 (8) | −0.0022 (7) | 0 | 0 |
| C1 | 0 | 0.2282 (4) | 0.6511 (5) | 0.070 (8) | 0.042 (7) | 0.026 (6) | 0 | 0 | 0 |
| C2 | −0.1032 (5) | 0.2631 (3) | 0.6095 (4) | 0.069 (4) | 0.042 (4) | 0.031 (4) | 0 | 0 | −0.012 (4) |
| C3 | −0.1035 (5) | 0.3257 (3) | 0.5331 (4) | 0.065 (3) | 0.054 (3) | 0.046 (3) | 0.007 (3) | 0.005 (3) | 0.001 (3) |
| C4 | 0 | 0.3564 (5) | 0.4954 (6) | 0.085 (4) | 0.055 (3) | 0.050 (3) | 0.017 (3) | −0.001 (3) | 0.005 (3) |
| C5 | 0.1130 (7) | 0.0995 (5) | 3/4 | 0.116 (6) | 0.041 (4) | 0.047 (4) | 0 | 0 | 0.014 (4) |
| C6 | 0.1629 (5) | 0.0673 (3) | 0.6600 (4) | 0.074 (5) | 0.040 (4) | 0.040 (4) | −0.000 (4) | 0 | 0 |
| C7 | 0.2546 (6) | 0.0098 (4) | 0.6586 (5) | 0.097 (4) | 0.061 (3) | 0.043 (3) | 0.018 (3) | −0.003 (3) | 0.003 (3) |
| C8 | 0.3000 (8) | −0.0185 (6) | 3/4 | 0.122 (5) | 0.067 (3) | 0.060 (4) | 0.030 (4) | 0.014 (4) | 0.000 (3) |
| C9[d] | 0 | 0.1371 (7) | 0.3711 (8) | 0.100 (6) | 0.069 (6) | 0.101 (7) | 0.045 (5) | 0 | 0 |
| C10 | 0.1069 (16) | 0.2845 (14) | 1/4 | 0.29 (1) | 0.071 (7) | 0.059 (6) | 0 | 0 | 0.020 (6) |
| B | 0 | 0.1624 (8) | 3/4 | 0.16 (1) | 0.21 (1) | 0.08 (1) | −0.181 (7) | 0 | 0 |

| atom | $x$ | $y$ | $z$ | $B$,[c] Å$^2$ | atom | $x$ | $y$ | $z$ | $B$,[c] Å$^2$ |
|---|---|---|---|---|---|---|---|---|---|
| H1 | −0.181 (3) | 0.240 (3) | 0.627 (3) | 4 (1) | H4 | 0.133 (4) | 0.087 (3) | 0.601 (4) | 5 (1) |
| H2 | −0.178 (3) | 0.340 (3) | 0.510 (3) | 5 (1) | H5 | 0.289 (4) | −0.007 (4) | 0.589 (4) | 8 (1) |
| H3 | 0 | 0.401 (4) | 0.455 (5) | 5 (2) | H6 | 0.352 (5) | −0.048 (4) | 3/4 | 4 (2) |

[a] Values in parentheses above and in other tables are estimated standard deviations in the last significant figures. [b] The form of the anisotropic thermal ellipsoid is $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + kl\beta_{23})]$; the $U_{ij}$ are related to the dimensionless $\beta_{ij}$ employed during refinement as $U_{ij} = \beta_{ij}/2\pi^2 a_i^* a_j^*$ for $i = j$ and as $\beta_{ij}/4\pi^2 a_i^* a_j^*$ for $i \neq j$. [c] Isotropic temperature factor, $B$, in the expression $\exp[-B \sin^2 \theta / 2\lambda^2]$. [d] Weight = 0.5.

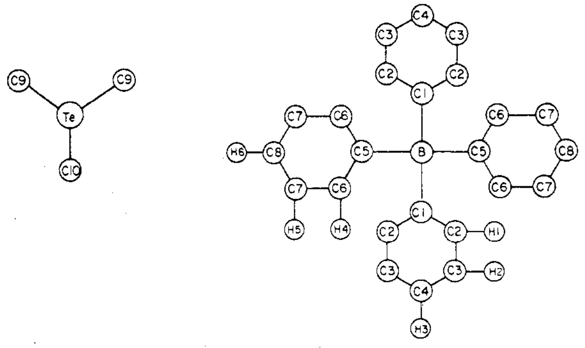

**Figure 1.** Atom labeling scheme.

half-occupancy tellurium atoms (with a site separation of 1.321 (2) Å) sharing two methyl carbons (C9) at full occupancy and having each a methyl carbon (C10) at half occupancy. Hydrogen atoms of the $B(C_6H_5)_4^-$ ion were located and refined isotropically after anisotropic refinement of all nonhydrogen atoms.

Calculations were performed on a PDP 11/45 using the Enraf-Nonius structure determination package[10] and on a Xerox Sigma 9 system. Atomic scattering factors for all atoms were taken from the compilation of Cromer and Waber[11] and corrected for both the real and imaginary parts of anomalous dispersion.[12] In the full-matrix least-squares refinement the function minimized was $\sum w(|F_o| - |F_c|)^2$ where the weight $w$ is defined as $4F_o^2/\sigma^2(F_o^2)$. Only the 664 reflections having $F_o^2 > 3\sigma(F_o^2)$ were used in the refinement. After the final complete cycles of refinement, no parameter was found to shift by more than 0.5 of its estimated standard deviation. No unusual trends appeared in plots of $\sum w(|F_o| - |F_c|)^2$ vs. $|F_o|$, $\lambda^{-1} \sin \theta$, reflection order in data collection, and various classes of indices. The final agreement factors (defined above) are $R_1 = 0.053$ and $R_2 = 0.070$. The final difference Fourier map showed no residual electron density as high as hydrogen atoms on a previous difference Fourier map.

The final postional and thermal parameters for the atoms are given in Table II. The observed and calculated structure factors and the root-mean-square amplitudes of thermal vibration are available as supplementary material.

## Results and Discussion

Trimethyltelluronium tetraphenylborate consists of discrete $(CH_3)_3Te^+$ and $B(C_6H_5)_4^-$ ions with the shortest tellurium–boron distances being 5.37 and 6.58 Å. An atom labeling scheme is presented in Figure 1, and interatomic distances and angles are given in Table III. Disorder of the cation is discussed in the Experimental Section and is illustrated in Figures 2 and 3.

The trimethyltelluronium ion is of the $AX_3E$ type and has the expected trigonal-pyramidal geometry with approximate $C_{3v}$ symmetry. Presumably, the fourth tetrahedral site along the pseudo-$C_3$ axis is occupied by the tellurium lone pair of electrons.[13] Secondary interactions involving the tellurium atom, as have been found in all other structural determinations

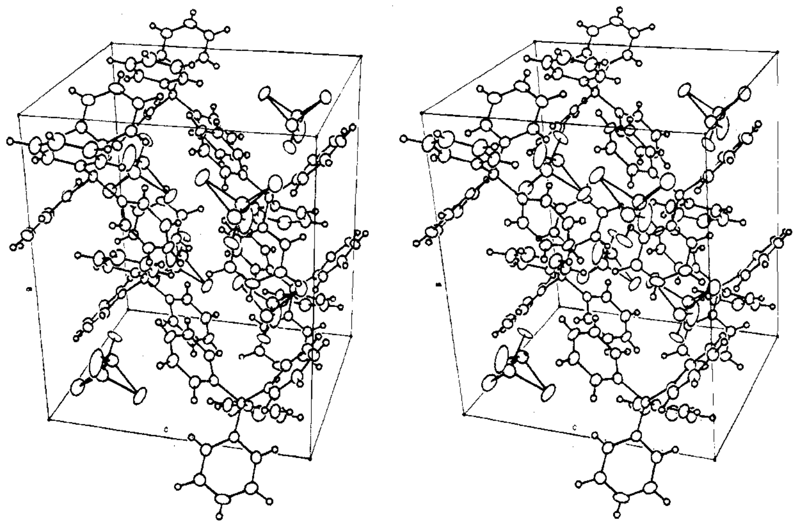

**Figure 2.** Stereoscopic unit cell view of $(CH_3)_3TeB(C_6H_5)_4$.

**Table III.** Interatomic Distances (Å) and Angles (deg) in $(CH_3)_3TeB(C_6H_5)_4$

| | | | |
|---|---|---|---|
| Te–C9 | 2.162 (7) | C5–C6 | 1.391 (5) |
| Te–C10 | 2.113 (14) | C6–C7 | 1.379 (6) |
| Te–B[a] | 5.366[b] | C7–C8 | 1.365 (6) |
| Te–Te[c] | 1.321 (2) | C2–H1 | 0.98 (4) |
| C9–C9′[d] | 3.138[b] | C3–H2 | 0.93 (4) |
| C9–C10 | 3.034[b] | C4–H3 | 0.87 (7) |
| C10–C10[c] | 2.447[b] | C6–H4 | 0.89 (4) |
| B–C1 | 1.639 (8) | C7–H5 | 1.02 (5) |
| B–C5 | 1.621 (8) | C8–H6 | 0.75 (6) |
| C1–C2 | 1.408 (5) | Te–C4 | 3.853 (7) |
| C2–C3 | 1.387 (6) | Te–H1 | 3.44 (4) |
| C3–C4 | 1.367 (6) | Te–H3 | 3.89 (6) |
| C9–Te–C9 | 93.0 (4) | C6–C7–C8 | 119.1 (6) |
| C9–Te–C10 | 90.4 (5) | C7–C8–C7 | 120.3 (8) |
| C1–B–C1 | 102.8 (6) | C1–C2–H1 | 122 (2) |
| C1–B–C5 | 112.1 (7) | C3–C2–H1 | 115 (2) |
| C5–B–C5 | 105.9 (7) | C2–C3–H2 | 113 (3) |
| B–C1–C2 | 122.7 (3) | C4–C3–H2 | 127 (3) |
| B–C5–C6 | 123.0 (3) | C3–C4–H3 | 119.6 (5) |
| C2–C1–C2 | 114.1 (6) | C5–C6–H4 | 116 (3) |
| C1–C2–C3 | 123.1 (5) | C7–C6–H4 | 120 (3) |
| C2–C3–C4 | 119.8 (5) | C6–C7–H5 | 118 (3) |
| C3–C4–C3 | 120.1 (7) | C8–C7–H5 | 123 (3) |
| C6–C5–C6 | 113.9 (6) | C7–C8–H6 | 119.8 (4) |
| C5–C6–C7 | 123.8 (5) | | |

[a] Shortest Te–B distance. [b] Estimated standard deviation was not calculated; comparable values are assumed. [c] Closest disorder site separation distance. [d] Primed atom at $[0, y, {}^1/_2 - z]$.

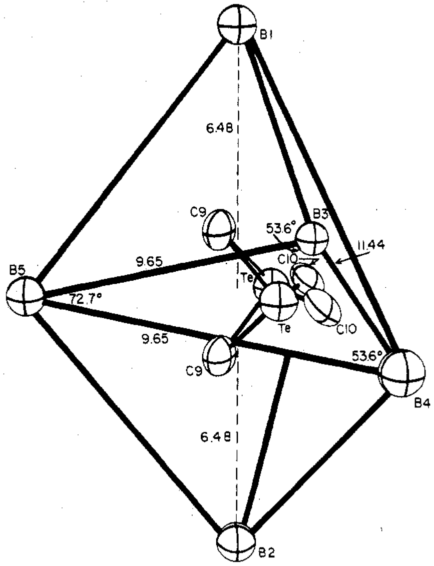

Figure 3. Trigonal-bipyramidal cavity and cation disorder in $(CH_3)_3TeB(C_6H_5)_4$. Distances are in Å.

of telluronium salts,[3–7,14] are absent, giving tellurium in this compound a genuine threefold configuration. We suggest that a similar situation prevails in $(C_6H_5)_3TeB(C_6H_5)_4$. The existence of these salts demonstrates the stability of the triorganotelluronium ion in the absence of secondary bonding and provides, to our best knowledge, the first examples of three-"coordinate" tellurium(IV). These compounds thereby serve to extend the range of coordination numbers known for tellurium(IV) from five to six.

The mean value[15] of the two independent Te–C distances (2.14 (2) Å) is that expected for a covalent Te–C single bond[16] and agrees with the values found in other triorganotelluronium salts.[3–7] The two independent C–Te–C angles are 90.45 (5) and 93.0 (4)°, however, and fall at the low end of the range of C–Te–C angles observed in other organotellurium compounds.[17] The mean value of 92 (1)° appears slightly smaller than the mean values of the C–Te–C angles found in other $(CH_3)_3Te^+$ and $(C_6H_5)_3Te^+$ salts, which range from 95 to 97°.[3–7] In comparison with the C–Te–C angles in $(CH_3)_3TeCl$ (93.8 (2), 94.1 (2), and 95.7 (2)°),[7] for example, those in the tetraphenylborate salt differ by 7–10$\sigma$ and 2–7$\sigma$. The distance of tellurium from the three-carbon methyl plane also differs, being longer (1.21 Å) in tetraphenylborate than in the chloride (1.12 Å). The same distance in $(CH_3)_3Te(CH_3TeI_4)$ is 1.08 Å.[3] Although these differences may be only marginally significant, the fact that the C–Te–C angles are at the low end in the range of C–Te–C angles might suggest that in the absence of secondary bonding the stereochemical activity of the tellurium lone pair is increased. This effect, if operative, should be small, since the secondary bonding, when present, is very weak. Our finding coincides with the observations made by Krebs and co-workers for the coordination and bonding in $[TeCl_3^+][AlCl_4^-]$[14] and $TeCl_4$[18] as compared to those in $TeCl_6^-$, where a decreasing stereochemical activity of the tellurium lone pair appears as the amount of Te–Cl covalent bonding increases. More definitive observations about the stereochemistry of tellurium(IV) in the absence of secondary bonding, however, will require additional accurate structural data, preferably a structure with no disorder, and for $R_3TeX$ compounds, a noncoordinating anion, X. Although $(C_6H_5)_3TeB(C_6H_5)_4$ would appear to meet these criteria, steric crowding of the phenyl rings would probably preclude any observation of the small changes sought in the C–Te–C angles, which in the $R_3TeX$ compounds appear to be slightly larger for R = $C_6H_5$ than for R = $CH_3$. We note that the cations in the oligomeric and polymeric $R_3TeX$ compounds, as well as in the corresponding $R_3SeX$ salts, have so far appeared structurally invariant despite changes in X.[3–5,19,20]

The tetraphenylborate ion has crystallographic symmetry *mm* ($C_{2v}$) with boron atoms in the *Cmcm* positions 4(c), $\pm(0, y, {}^1/_4) + C$. Accordingly, C1, C4, and H3 lie in a mirror at positions 8(f), $\pm(0, y, z; 0, y, {}^1/_2 - z) + C$, and C5, C8, and H6 at positions 8(g), $\pm(x, y, {}^1/_4; \bar{x}, y, {}^1/_4) + C$. Identical symmetry was found in tetraethylammonium tetraphenylborate[21] which is isomorphous with the present compound. The mean value of the six C–B–C angles is 109 (2)° and is identical with that found in other accurate structural determinations involving the tetraphenylborate ion.[21–24] The two B–C distances are not significantly different (<3$\sigma$) and have a mean value of 1.630 (9) Å. The two independent phenyl rings are planar (Table IV) with no carbon atom deviating by more than one estimated standard deviation or hydrogen atom by more than two esd's. Both B–C bonds are out of their respective phenyl ring planes with the boron atoms deviating by 0.17 and 0.10 Å. Deviations of this magnitude were observed in other

**Table IV.** Weighted Least-Squares Planes for the Phenyl Rings in the Tetraphenylborate Anion[a]

Plane 1: $A = -0.0009, B = -0.7112, C = -0.7030, D = -8.4532$

| atom | C1 | C2 | C3 | C4 | H1 | H2 | H3 | B |
|---|---|---|---|---|---|---|---|---|
| dist, Å | 0.001 (7) | −0.004 (5) | −0.000 (6) | 0.002 (8) | 0.09 (4) | 0.06 (4) | −0.12 (7) | −0.172 (9) |

Plane 2: $A = 0.6497, B = 0.7602, C = -0.0049, D = 1.9665$

| atom | C5 | C6 | C7 | C8 | H4 | H5 | H6 | B |
|---|---|---|---|---|---|---|---|---|
| dist, Å | 0.002 (7) | −0.002 (6) | 0.001 (6) | −0.002 (9) | 0.01 (4) | 0.07 (5) | 0.03 (6) | −0.096 (9) |

Dihedral Angle (1–2): −57.5°

[a] Planes are of the form $Ax + By + Cz + D = 0$ where *x*, *y*, and *z* are orthogonalized coordinates.

tetraphenylborate salts[21,23,24] but not in that of [(nas)Ni(NCS)][$B(C_6H_5)_4$], where three of the B–C bonds are coplanar with the respective phenyl ring.[22] A distortion of the phenyl rings from a regular hexagonal structure occurs in a manner first reported by Di Vaira and Orlandini,[22] and later by Segal and Lippard.[23] Starting from the boron-bonded carbon atom and proceeding, carbon by carbon to the para position, we note that the mean value of each of the two independent C–C–C angles follows the order 114.0 (1), 123.4 (4), 119.4 (4), and 120.2 (1)°, respectively, with the total mean value being 119 (1)°. Di Vaira and Orlandini suggested that the distortion of the phenyl rings originated from steric crowding about the boron atom.[23] In the present case, we note that the boron-bonded C–C distances are longer than the remaining C–C distances by $0.5\sigma$ to $5\sigma$. Although the differences may not be significant, their direction is consistent with the C–C–C angle trend and with Di Vaira and Orlandini's interpretation of the distortion. A similar small closure of this angle is observed in tetraphenylmethane.[25]

The six independent C–C distances in the phenyl rings have a mean value of 1.383 (6) Å which may be compared with 1.383 (4) and 1.382 (3) Å reported in ref 22 and 23, respectively. The mean C–H value is 0.91 (5) Å. The dihedral angle between the independent rings is 57.5°. That between the mirror-related rings containing C1 is 89.3° while that between the mirror-related rings containing C5 is 81.0°. The mean inter-ring dihedral angle is 70° and is in the range of values (66–71°) reported for other tetraphenylborate salts. Some intraionic C–C contacts for the $B(C_6H_5)_4^-$ ion in the present structure are C1–C1 = 2.562 Å, C5–C5 = 2.587 Å, C1–C5 = 2.704 Å, C1–C6 = 3.121 Å, and C2–C5 $[\bar{x}, y, {}^3/_4]$ = 3.129 Å.

Packing in the crystal is illustrated in Figure 2 and is dominated by the tetraphenylborate ion. The structure consists of layers of $B(C_6H_5)_4^-$ and $(CH_3)_3Te^+$ ions parallel to the *xy* plane such that three $B(C_6H_5)_4^-$ ions surround each $(CH_3)_3Te^+$ ion. As a consequence, a belt of four phenyl rings perpendicular to the *xy* plane and consisting of two rings from one anion and one each from two other anions surround each cation site. In addition, two phenyl rings, one from each flanking layer, face the cation site giving it a total of six rings whose normals point generally in the direction toward the disordered cation. The resulting immediate environment of the cation is analogous to a geodesic enclosure having eight faces. In this case six faces are occupied by phenyl rings and two are holes or openings leading to cations in the adjoining layers.

A predominant feature of this layered structure is the trigonal bipyramid of boron atoms that surrounds the cation site. The orientation of the disordered cation, which appears to be in a minimum energy configuration in the trigonal-bipyramidal cavity, is illustrated in Figure 3. The C10 site disorder lies in the equatorial plane and is in closest proximity to the longest equatorial B–B distance. The axial B1–B2 distance is slightly longer than the B3–B4 equatorial distance, consistent with the C9–C9 distance (parallel to B1–B2) being longer than the C10–C10 site separation distance. In the crystal, the equatorial plane of the bipyramid coincides with the *xy* mirror plane. The *yz* mirror plane contains B1 and B2 and bisects the B3–5–4 angle.

One possible cause for the disorder in the structure is the large space available to the cation in the bipyramidal cavity. Subtracting the $(C_6H_5)_3Te^+$ volume (obtained from $(C_6H_5)_3TeCl$,[6] where no disorder occurs) from that of $(C_6H_5)_3TeB(C_6H_5)_4$ (Table I), where we assume no excess volume, one obtains 384 Å$^3$ as an estimate of the required volume of the tetraphenylborate ion. Interestingly, the unit volume of $(CH_3)_3TeB(C_6H_5)_4$, $V/Z$, minus this value (384 Å$^3$) leads to 192 Å$^3$, the volume of the trigonal-bipyramidal cavity. This volume is 57 Å$^3$ in excess of that required by the $(CH_3)_3Te^+$ ion in $(CH_3)_3TeCl$,[7] where no disorder occurs.

The closest cation–anion contacts in $(CH_3)_3TeB(C_6H_5)_4$, exclusive of those involving methyl hydrogen atoms, are C10–H1 $[^1/_2 + x, {}^1/_2 - y, {}^1/_2 + z]$ = 2.93 Å, C9–H4, 3.43 Å, C10–H3 $[x, y, {}^1/_2 - z]$ = 3.44 Å, Te–H1 $[^1/_2 + x, {}^1/_2 - y, {}^1/_2 + z]$ = 3.44 Å, and C10–H2 $[\bar{x}, y, z]$ = 3.57 Å. All other cation–anion contacts are greater than 3.8 Å.

Finally, we note that the properties of $(C_6H_5)_3TeB(C_6H_5)_4$ are similar to those of $(CH_3)_3TeB(C_6H_5)_4$. Both salts melt or decompose above 200 °C and are insoluble in water, a property consistent with the tetraphenylborate-dominated packing in $(CH_3)_3TeB(C_6H_5)_4$. In $(C_6H_5)_3TeB(C_6H_5)_4$, the ions are more nearly the same size and packing would not be expected to be dominated by either ion. The abundance and shielding effect of the phenyl groups, however, would likely preclude water solubility. A similar situation prevails in $(C_6H_5)_3Te(NCS)$.[4]

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**Registry No.** $(CH_3)_3TeB(C_6H_5)_4$, 70320-99-9; $(C_6H_5)_3TeB(C_6H_5)_4$, 70321-00-5; $(CH_3)_3TeCl$, 18987-55-8; $(C_6H_5)_3TeCl$, 1224-13-1.

**Supplementary Material Available:** The observed and calculated structure factors and the root-mean-square amplitudes of thermal vibration (4 pages). Ordering information is given on any current masthead page.

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