Crystal structure of bis(4-dimethylaminophenyl)telluride, $(4\text{-}Me_2NC_6H_4)_2Te$

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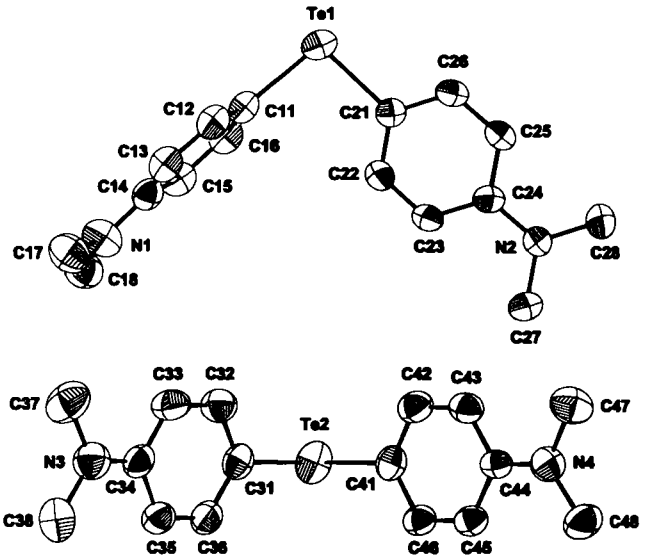

Abstract

$C_{16}H_{20}N_2Te$, triclinic, $P\bar{1}$ (No. 2), $a = 9.9090(6)$ Å, $b = 10.405(3)$ Å, $c = 15.803(2)$ Å, $\alpha = 102.29(1)°$, $\beta = 94.142(7)°$, $\gamma = 94.05(1)°$, $V = 1581.7$ Å^3, $Z = 4$, $R_{gt}(F) = 0.031$, $wR_{ref}(F^2) = 0.087$, $T = 298$ K.

Source of material

The precursor $(4\text{-}Me_2NC_6H_4)_2TeCl_2$ was syntesized by refluxing in dioxane (50 ml), for 3 h, a mixture of $TeCl_4$ [1] (1.46 g, 5.42 mmol) with $4\text{-}Me_2NC_6H_4HgCl$ [2] (3.92 g, 11.0 mmol). Cooling at 273 K with stirring precipitated a white Hg_2Cl:dioxane complex, which was filtered off. The solution was dried and the green-yellowish solid was extracted with chloroform to obtain $(4\text{-}Me_2NC_6H_4)_2TeCl_2$ (1.62 g, 3.69 mmol). Recrystallization from acetone produced yellow needles, mp 460 K – 461 K with decomposition (461 K – 462 K [3]). The $(4\text{-}Me_2NC_6H_4)_2TeCl_2$ (1.30 g, 2.96 mmol) was mixed with melted $Na_2S{\cdot}9H_2O$ (9.00 g, 37.5 mmol), which produced an exothermic reaction. The mixture was kept at 383 K for 10 min and then cooled with water (20 ml) at 273 K. The orange-yellow crystalline solid was filtered off and washed with cold water (0.163 g, 0.444 mmol, 15%). Crystals suitable for X-ray diffraction were obtained from slow evaporation of a solution in chloroform.

Discussion

Within our program of synthetic and structural studies of organotellurium compounds [4], we prepared the title compound and determined its crystal structure. The asymmetric unit contains two independent molecules of $(4\text{-}Me_2NC_6H_4)_2Te$. The molecules are V-shaped, as predicted by the valence-shell electron-pair repulsion (VSEPR) model for an AX_2E_2 molecule [5]. Within experimental error (i.e. 3 e.s.d.'s) the four Te—C distances are equal (average: 2.110 Å), and their values are comparable to the standard value of 2.116(20) Å for Te-C(aryl) [6], and to 2.110(3) and 2.112(3) Å, observed in $(4\text{-}MeOC_6H_4)_2Te$ [7], and to 2.105(5) Å and 2.125(5) Å, in $(2\text{-}PhC_6H_4)_2Te$ [8]. As expected, the C–Te–C angles (average: 97.1°) are considerably smaller than the tetrahedral value (109.5°), because of the repulsion of the lone pairs of electrons on the bonded pairs. These angles are similar to 96.2(2)°, found in $(2\text{-}PhC_6H_4)_2Te$ [8] and to 96.3(2)° in [2-(4-$MeOC_6H_4N$:CH)$C_6H_4]_2Te$ [9]. The bond lengths in the organic moieties are in good agreement with the tabulated values [6]. Both independent molecules are essentially similar. The main difference lies in the orientation of the ring systems [ring/plane/ring dihedral angles: C1n/C–Te1–C/C2n, 79.1(2)° and 1.4(2)°; C3n/C–Te2–C/C4n, 81.2(2)° and 85.4(2)°], probably because of the packing forces, which are entirely of van der Waals type.

Table 1. Data collection and handling.

Crystal:	pale brown prism, size 0.15 × 0.18 × 0.39 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	18.70 cm^{-1}
Diffractometer, scan mode:	Enraf Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	49.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5570, 5570
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3701
$N(param)_{refined}$:	343
Programs:	SHELXS-97 [10], SHELXL-97 [11], ORTEP-3 [12], WinGX [13]

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

Atom	Site	x	y	z	U_{iso}
H(12)	2*i*	0.0373	–0.4625	0.5978	0.077
H(13)	2*i*	–0.1693	–0.4532	0.6526	0.079
H(15)	2*i*	–0.1364	–0.0609	0.6829	0.080
H(16)	2*i*	0.0748	–0.0701	0.6320	0.080
H(171)	2*i*	–0.4706	–0.3484	0.7278	0.142
H(172)	2*i*	–0.3362	–0.4119	0.7475	0.142
H(173)	2*i*	–0.3988	–0.4298	0.6513	0.142
H(181)	2*i*	–0.4543	–0.1453	0.7434	0.135
H(182)	2*i*	–0.3711	–0.0860	0.6778	0.135
H(183)	2*i*	–0.3090	–0.0732	0.7737	0.135
H(22)	2*i*	0.0029	–0.2429	0.4457	0.068
H(23)	2*i*	–0.0628	–0.2249	0.3075	0.068

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(25)	2*i*	0.3180	–0.2523	0.2461	0.077
H(26)	2*i*	0.3848	–0.2636	0.3857	0.075
H(271)	2*i*	–0.0694	–0.2003	0.0935	0.117
H(272)	2*i*	–0.0731	–0.1150	0.1878	0.117
H(273)	2*i*	–0.1219	–0.2659	0.1669	0.117
H(281)	2*i*	0.1284	–0.2215	0.0586	0.112
H(282)	2*i*	0.2244	–0.3023	0.1062	0.112
H(283)	2*i*	0.2376	–0.1477	0.1330	0.112
H(32)	2*i*	–0.2937	–0.4557	–0.0540	0.089
H(33)	2*i*	–0.0857	–0.4739	–0.1042	0.091
H(35)	2*i*	–0.0489	–0.0838	–0.0939	0.086
H(36)	2*i*	–0.2606	–0.0690	–0.0455	0.080
H(371)	2*i*	0.2297	–0.4037	–0.1751	0.153
H(372)	2*i*	0.0822	–0.4705	–0.2049	0.153

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(373)	2*i*	0.1436	–0.4585	–0.1090	0.153
H(381)	2*i*	0.2521	–0.2013	–0.1662	0.135
H(382)	2*i*	0.1815	–0.1103	–0.0934	0.135
H(383)	2*i*	0.1181	–0.1403	–0.1901	0.135
H(42)	2*i*	–0.4448	–0.4351	0.1272	0.081
H(43)	2*i*	–0.3795	–0.4277	0.2703	0.074
H(45)	2*i*	–0.3545	–0.0345	0.3200	0.068
H(46)	2*i*	–0.4129	–0.0417	0.1763	0.069
H(471)	2*i*	–0.2910	–0.3261	0.4904	0.129
H(472)	2*i*	–0.2447	–0.3943	0.4002	0.129
H(473)	2*i*	–0.3992	–0.4009	0.4157	0.129
H(481)	2*i*	–0.2901	–0.1238	0.5196	0.134
H(482)	2*i*	–0.3980	–0.0661	0.4640	0.134
H(483)	2*i*	–0.2434	–0.0468	0.4501	0.134

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}
Te(1)	2*i*	0.26932(3)	–0.27655(3)	0.55711(2)	0.0674(2)	0.0721(2)	0.0647(2)	0.0099(2)	–0.0076(2)	0.0185(2)
N(1)	2*i*	–0.3062(4)	–0.2521(4)	0.7001(3)	0.076(3)	0.065(3)	0.100(3)	0.006(2)	0.011(2)	0.004(2)
N(2)	2*i*	0.0781(4)	–0.2303(5)	0.1765(3)	0.056(2)	0.147(4)	0.062(2)	0.030(2)	0.015(2)	0.037(3)
C(11)	2*i*	0.0794(5)	–0.2669(5)	0.6088(3)	0.073(3)	0.064(3)	0.049(2)	0.007(2)	0.000(2)	0.017(2)
C(12)	2*i*	0.0031(5)	–0.3810(4)	0.6154(3)	0.084(3)	0.049(3)	0.061(3)	0.012(2)	0.003(2)	0.011(2)
C(13)	2*i*	–0.1217(5)	–0.3754(4)	0.6473(3)	0.085(3)	0.048(3)	0.063(3)	0.000(2)	0.001(2)	0.013(2)
C(14)	2*i*	–0.1787(5)	–0.2571(5)	0.6719(3)	0.067(3)	0.062(3)	0.050(2)	–0.001(2)	–0.004(2)	0.002(2)
C(15)	2*i*	–0.1018(5)	–0.1423(5)	0.6659(3)	0.081(3)	0.051(3)	0.064(3)	0.010(2)	–0.002(2)	0.006(2)
C(16)	2*i*	0.0251(5)	–0.1480(4)	0.6351(3)	0.082(3)	0.046(3)	0.068(3)	–0.001(2)	0.000(2)	0.011(2)
C(17)	2*i*	–0.3844(5)	–0.3703(6)	0.7073(4)	0.079(4)	0.085(4)	0.119(5)	–0.008(3)	0.014(3)	0.023(3)
C(18)	2*i*	–0.3650(5)	–0.1289(6)	0.7259(4)	0.084(4)	0.091(4)	0.094(4)	0.019(3)	0.014(3)	0.010(3)
C(21)	2*i*	0.2009(4)	–0.2562(4)	0.4312(3)	0.054(2)	0.040(2)	0.051(2)	0.002(2)	–0.001(2)	0.002(2)
C(22)	2*i*	0.0676(4)	–0.2441(4)	0.4058(3)	0.051(2)	0.063(3)	0.056(2)	0.008(2)	0.010(2)	0.010(2)
C(23)	2*i*	0.0281(4)	–0.2337(4)	0.3224(3)	0.041(2)	0.067(3)	0.062(3)	0.009(2)	0.004(2)	0.013(2)
C(24)	2*i*	0.1185(4)	–0.2358(4)	0.2611(3)	0.047(2)	0.055(3)	0.057(2)	0.004(2)	–0.002(2)	0.010(2)
C(25)	2*i*	0.2538(4)	–0.2490(5)	0.2864(3)	0.046(2)	0.084(3)	0.067(3)	0.012(2)	0.012(2)	0.021(2)
C(26)	2*i*	0.2936(4)	–0.2572(4)	0.3701(3)	0.044(2)	0.076(3)	0.069(3)	0.010(2)	0.001(2)	0.021(2)
C(27)	2*i*	–0.0579(4)	–0.2004(6)	0.1543(3)	0.060(3)	0.111(4)	0.068(3)	0.012(3)	–0.004(2)	0.035(3)
C(28)	2*i*	0.1753(5)	–0.2250(5)	0.1133(3)	0.068(3)	0.094(4)	0.065(3)	0.002(3)	0.014(2)	0.022(3)
Te(2)	2*i*	–0.49420(3)	–0.24530(4)	0.00349(2)	0.0640(2)	0.1032(3)	0.0643(2)	0.0166(2)	–0.0050(2)	0.0103(2)
N(3)	2*i*	0.0866(4)	–0.2908(4)	–0.1323(3)	0.075(3)	0.080(3)	0.088(3)	0.009(2)	0.016(2)	0.014(2)
N(4)	2*i*	–0.3271(4)	–0.2277(4)	0.3986(2)	0.073(2)	0.070(3)	0.066(2)	0.012(2)	0.000(2)	0.021(2)
C(31)	2*i*	–0.3010(5)	–0.2606(5)	–0.0428(3)	0.066(3)	0.074(3)	0.047(2)	0.010(2)	–0.002(2)	0.007(2)
C(32)	2*i*	–0.2446(5)	–0.3803(5)	–0.0619(3)	0.074(3)	0.063(3)	0.083(3)	0.002(3)	0.008(3)	0.012(3)
C(33)	2*i*	–0.1191(5)	–0.3912(5)	–0.0918(3)	0.081(3)	0.052(3)	0.092(4)	0.010(3)	0.009(3)	0.010(3)
C(34)	2*i*	–0.0394(5)	–0.2814(5)	–0.1044(3)	0.081(3)	0.062(3)	0.048(2)	0.014(3)	0.002(2)	0.006(2)
C(35)	2*i*	–0.0975(5)	–0.1595(5)	–0.0860(3)	0.092(4)	0.059(3)	0.059(3)	0.004(3)	0.003(3)	0.006(2)
C(36)	2*i*	–0.2248(5)	–0.1505(5)	–0.0565(3)	0.082(3)	0.063(3)	0.055(3)	0.017(3)	0.005(2)	0.010(2)
C(37)	2*i*	0.1399(6)	–0.4162(6)	–0.1574(4)	0.086(4)	0.096(4)	0.119(5)	0.027(3)	0.016(3)	0.005(4)
C(38)	2*i*	0.1663(5)	–0.1759(6)	–0.1467(3)	0.083(4)	0.114(5)	0.078(4)	0.011(3)	0.012(3)	0.029(3)
C(41)	2*i*	–0.4339(4)	–0.2398(5)	0.1356(3)	0.056(3)	0.071(3)	0.061(3)	0.012(2)	0.011(2)	0.010(2)
C(42)	2*i*	–0.4251(4)	–0.3538(5)	0.1656(3)	0.062(3)	0.057(3)	0.080(3)	0.007(2)	0.014(2)	0.005(2)
C(43)	2*i*	–0.3871(4)	–0.3489(4)	0.2522(3)	0.060(3)	0.053(3)	0.075(3)	0.008(2)	0.008(2)	0.017(2)
C(44)	2*i*	–0.3602(4)	–0.2309(4)	0.3128(3)	0.041(2)	0.046(2)	0.060(2)	0.002(2)	0.002(2)	0.009(2)
C(45)	2*i*	–0.3715(4)	–0.1161(4)	0.2814(3)	0.051(2)	0.052(3)	0.064(3)	0.003(2)	0.004(2)	0.006(2)
C(46)	2*i*	–0.4070(4)	–0.1201(4)	0.1951(3)	0.057(2)	0.052(3)	0.063(3)	0.010(2)	0.007(2)	0.010(2)
C(47)	2*i*	–0.3144(5)	–0.3471(5)	0.4287(4)	0.088(4)	0.079(4)	0.091(4)	0.005(3)	–0.014(3)	0.031(3)
C(48)	2*i*	–0.3135(6)	–0.1059(5)	0.4635(3)	0.132(5)	0.070(3)	0.059(3)	0.010(3)	–0.004(3)	0.003(3)

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