

## NITROGEN-TELLURIUM-CONTAINING HETEROCYCLES.

### 4.\* X-RAY STRUCTURE OF 2-PHENYLBENZOTELLURAZOLE

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The crystal and molecular structure of 2-phenylbenzotellurazole is determined by x-ray methods. The heterocyclic fragment of the molecule is planar. The dihedral angle between planes of the heterocycle and the 2-phenyl substituent is 31.2°. Somewhat shortened Te-N intermolecular contacts (3.43 Å) are seen in the crystal. These do not substantially affect the physical properties of the compound.

Data on five-membered nitrogen-tellurium-containing heterocycles are limited [2]. Only benzisotellurazole [3] and 1,2,5-telluradiazole [4] have been studied by x-ray methods. In the present report, we describe the crystal and molecular structure of a representative of the new benzotellurazole heterocyclic system [5-7], 2-phenylbenzotellurazole (I).

The molecular structure and bond lengths and angles in it are shown in Fig. 1. The atomic coordinates appear in Table 1. The heterocyclic fragment of 2-phenylbenzotellurazole is planar. The atoms of this fragment deviate from the plane by 0.001-0.017 Å: Te, 0.016; N, 0.006; C<sub>(1)</sub>, 0.002; C<sub>(2)</sub>, 0.011; C<sub>(3)</sub>, 0.006; C<sub>(4)</sub>, 0.010; C<sub>(5)</sub>, 0.001; and C<sub>(6)</sub>, 0.017 Å. The dihedral angle between the planes of the heterocyclic and phenyl rings is 31.2°. Such twisting of the phenyl substituent around C<sub>(7)</sub> relative to the plane of the remaining part of the molecule minimizes the steric hindrance. The intramolecular Te-H<sub>(13)</sub> distance is 2.93 Å. However, this intramolecular contact should be much closer (Te-H<sub>(13)</sub> 2.72 Å) according to the planar model of I.

The Te-C bond lengths (2.080 and 2.123 Å) are comparable to analogous distances in other five- [2] and six-membered [8] tellurium-containing heterocycles (2.07-2.12 Å).

The angles around the heteroatoms in I differ considerably from corresponding angles in benzoxazole (5-nitro-2-cyclohexylaminobenzoxazole [9]) and benzothiazole (2-(2'-hydroxy-phenyl)benzothiazole [10]) derivatives. The C<sub>(1)</sub>-N-C<sub>(7)</sub> angle increases in the series benzoxazole (103.8°), benzothiazole (110.8°), and benzotellurazole (116.9°). Conversely, the C<sub>(2)</sub>-X-C<sub>(7)</sub> angle for the group VIA X heteroatoms decreases much more significantly in the same order: benzoxazole (103.8°), benzothiazole (88.6°), and benzotellurazole (78.6°). The decrease of the C-C-C angle and the increase of the C-N-C angle is caused by a lengthening of the X-C bond on increasing the size of the X heteroatom in the order X = O, S, Te. Moreover, the angle at the tellurium atom in 2-phenylbenzotellurazole (I) differs insignificantly from analogous angles in a number of other five-membered tellurium-containing heterocycles: α-tellurophenecarboxylic acid (81.5°) [11], dibenzotellurophene (81.7°) [12], benzisotellurazole (80.0°) [3], and 1,2,5-telluradiazole (82.5°) [4]. The bond lengths and angles in the annelated benzene ring and the 2-phenyl substituent have standard values.

The short intermolecular contacts between the nitrogen and tellurium atoms are especially interesting. Similar Te-N contacts observed in benzisotellurazole (2.40 Å) [3] and 1,2,5-telluradiazole (2.76 Å) [4] (the sum of van der Waals radii for tellurium and nitrogen is 3.70 Å [13]) are probably responsible for the anomalously high melting point and low solubility in ordinary organic solvents of tellurium-containing heterocycles in comparison to other

\*For Communication 3, see [1].

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TABLE 1. Atomic Coordinates in I ( $\times 10^5$  for Te,  $\times 10^4$  for N and C,  $\times 10^3$  for H)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	Atom	<i>x</i>	<i>y</i>	<i>z</i>
Te	12014(2)	11163(4)	24333(2)	C <sub>(11)</sub>	3733(4)	-3954(7)	539(3)
N	3662(3)	1980(5)	2830(2)	C <sub>(12)</sub>	2840(4)	-4238(7)	1005(3)
C <sub>(1)</sub>	3202(3)	3478(6)	3312(2)	C <sub>(13)</sub>	2596(4)	-2742(7)	1610(3)
C <sub>(2)</sub>	2016(3)	3510(6)	3271(3)	H <sub>(3)</sub>	60	504	368
C <sub>(3)</sub>	1498(4)	5025(7)	3728(3)	H <sub>(4)</sub>	184	765	460
C <sub>(4)</sub>	2204(4)	6497(7)	4248(3)	H <sub>(5)</sub>	392	759	471
C <sub>(5)</sub>	3397(4)	6464(7)	4311(3)	H <sub>(6)</sub>	480	493	392
C <sub>(6)</sub>	3901(4)	4949(7)	3852(3)	H <sub>(9)</sub>	467	61	145
C <sub>(7)</sub>	2951(4)	653(6)	2368(3)	H <sub>(10)</sub>	508	-199	36
C <sub>(8)</sub>	3246(3)	-965(6)	1772(3)	H <sub>(11)</sub>	390	-507	6
C <sub>(9)</sub>	4161(4)	-711(7)	1322(3)	H <sub>(12)</sub>	235	-559	90
C <sub>(10)</sub>	4398(4)	-2197(8)	705(3)	H <sub>(13)</sub>	191	-296	195

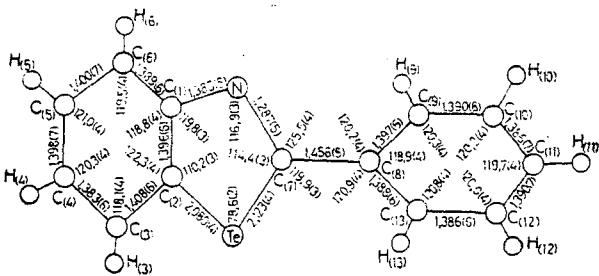


Fig. 1. Geometry of 2-phenylbenzotellurazole.

benzisochalcogenoazoles and 1,2,5-chalcogenodiazoles. Intermolecular Te-N contacts (3.43 Å) that are longer than those in the aforementioned tellurium-containing heterocycles are also seen in 2-phenylbenzotellurazole.

These relatively weak contacts, shortened in comparison to the sum of van der Waals radii for tellurium and nitrogen by about 0.3 Å, do not have a substantial effect on the physical properties of 2-phenylbenzotellurazole. The melting point of this compound (118–120°C [5]) is almost the same as for its selenium analog (116°C [14]). It is very soluble in ordinary organic solvents.

#### EXPERIMENTAL

2-Phenylbenzotellurazole was synthesized by the method given in [6].

The x-ray structural study of I was carried out on a Syntex P2<sub>1</sub> diffractometer ( $\theta/2\theta$  scanning, Mo K $\alpha$  radiation, graphite monochromator,  $2\theta_{\max} = 60^\circ$ ).

Yellow crystals of I, C<sub>13</sub>H<sub>9</sub>NTe, are monoclinic:  $a = 11.870(1)$ ,  $b = 6.557(6)$ ,  $c = 14.403(2)$  Å,  $\beta = 103.107(9)^\circ$ ,  $V = 1091.9(2)$  Å<sup>3</sup>,  $\rho_{\text{exp}} = 1.83(3)$  g/cm<sup>3</sup>,  $\rho_{\text{calc}} = 1.87$  g/cm<sup>3</sup>,  $\mu_{\text{Mo}} = 27.9$  cm<sup>-1</sup>,  $Z = 4$ , space group P2<sub>1</sub>/ $a$ .

Intensities of 3009 reflections were measured. Of these, 2451 with  $I \geq 1.96$  were used in the calculations. All calculations were performed on a Nova-1200 minicomputer using XTL programs.

The structure of I was solved by the heavy atom method and refined by anisotropic full-matrix least squares for all nonhydrogen atoms. The hydrogen atom positions were calculated for sp<sup>2</sup>-hybridized carbon atoms and C-H distances of 1.05 Å. The final R-factor was 0.030.

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