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THE MOLECULAR CHARACTERIZATION OF AN ORGANOTELLURIUM "ANHYDRIDE," BIS(ISOTHIOCYANATODIPHENYLTELLURIUM(IV)) OXIDE, $((C_6H_5)_2TeNCS)_2O$

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SUMMARY

The first structural characterization of a member of the class of compounds $R_2Te(X)OTe(X)R_2$ is reported. Three-dimensional X-ray crystal structure analysis shows that $(Ph_2TeNCS)_2O$ is molecular in the solid state. The molecule has two-fold symmetry, with the rotation axis passing through the oxygen atom and bisecting the Te-O-Te angle. The ether-like Te-O-Te moiety is bent, 121.7(4)°, with a Te-O distance of 1.985(4)Å. An intermolecular Te-S contact at 3.416(3)Å completes a square-pyramidal geometry around the tellurium atom, and links the molecules into chains running parallel to the c axis. The Te-N bond distance, 2.40(1)Å, is considerably longer than the sum of covalent radii, indicating a tendency toward ionic character in the bond. Refinement of the structure, based on 1662 reflections collected by automatic diffractometry, converged to a conventional R factor of 6.5% and a weighted R factor of 6.2%. Crystal data for $(Ph_2TeNCS)_2O$ are as follows: space group C2/c, $a = 18.540(5)$Å, $b = 13.487(6)$Å, $c = 10.934(4)$Å, $\beta = 94.72(2)°$, $V = 2725$Å^3 (23 ± 1°C) and $Z = 4$.

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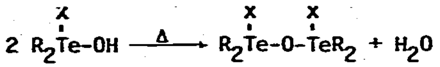
INTRODUCTION

Organotellurium hydroxy halides, which are formed from compounds of the type R_2TeX_2 (X = halide), $R_2Te(OH)_2$ or R_2TeO, lose water relatively easily to form species known as organotellurium anhydrides, as in reaction (1):[1,2]



$$2\ R_2\overset{X}{Te}\text{-}OH \xrightarrow{\Delta} R_2\overset{X}{Te}\text{-}O\text{-}\overset{X}{Te}R_2 + H_2O \qquad (1)$$

In earlier literature these andydrides have also been called dihalotetraorganylditelluronium oxides.[3] Although several synthetic routes to these compounds have been explored, their characterization has been ignored. In fact, the structure of these compounds has never been established, but only inferred, mainly from chemical analyses data and from a history of the compound's synthesis.

To provide a structural characterization of a member of this class of compounds, and to continue our characterization and stereochemical studies of Te(IV) and Se(IV) species,[4-7] we have determined the structure of bis(isothiocyanatodiphenyltellurium(IV)) oxide, $(Ph_2TeNCS)_2O$.

EXPERIMENTAL

Previously unreported $(Ph_2TeNCS)_2O$ was synthesized by mixing aqueous solutions of $Ph_2TeC\ell_2$ (near boiling) and excess NaNCS.[8] The resulting white precipitate was filtered, dried and recrystallized from $CHC\ell_3$ to yield suitable specimens for diffraction.

Crystals of $(Ph_2TeNCS)_2O$ are monoclinic with unit cell dimensions $a = 18.540(5)$Å, $b = 13.487(6)$Å, $c = 10.934(4)$Å, $\beta = 94.72(2)°$ and $V = 2725$Å^3 (23 ± 1°C). The density by floatation ($CC\ell_4$, $Br_2CHCHBr_2$) is 1.68(2) g/cm^3, and agrees with the calculated value of 1.70 g/cm^3 for four molecules per unit cell. Systematic extinctions, $hk\ell$: $h+k\neq 2n$ and $h0\ell$: $\ell\neq 2n$, indicate C-centering, space groups C2/c or Cc.

Intensity data were collected by Molecular Structure Corporation* by the θ-2θ scan technique on a Syntex P$\bar{1}$ automatic diffractometer using graphite monochromatized Mo-Kα radiation and a crystal prism measuring 0.20 x 0.20 x 0.24mm. Intensities and standard deviations of intensities were calculated using the formulas $I = S(c - RB)$ and $\sigma(I) = [S^2(c + R^2B) + (pI)^2]^{\frac{1}{2}}$ where S is the scan rate, here ranging from 4° to 24°/min., c is the total integrated peak count, R is the ratio of scan time to background counting time, equal to 2.0, B is the total background count, and p, here set equal to 0.07, a factor introduced to downweight intense reflections. The range of data was $0° < 2\theta(\text{MoK}\alpha) < 45°$ with a scan range from $2\theta(\text{MoK}\alpha_1) - 0.9°$ to $2\theta(\text{MoK}\alpha_2) + 0.9°$. The intensities of 3984 independent reflections (including systematic extinctions) were measured: solution and refinement of the structure were based on the 1662 reflections having $F > \sigma(F)$. Periodic checks on the crystal and electronic stability indicated no significant changes throughout the data collection. The intensities were corrected for Lorentz and polarization effects, but not for absorption. The calculated linear absorption coefficient (MoKα), μ, is 24 cm^{-1}.

The structure was solved by Patterson and Fourier techniques and was refined by least-squares methods. The scattering factors were taken from the compilation of Cromer and Waber[9]; those for Te and S were corrected for the real and imaginary components of anomalous dispersion using the values of Cromer and Liberman.[10] The function minimized in the least-squares refinement was $\Sigma w(|F_{obs}| - |F_{calc}|)^2$ where $w = 1/\sigma^2(F)$. Anisotropic thermal parameters were used for all of the non-hydrogen atoms. The refinement converged to a final R factor $(\Sigma||F_{obs}| - |F_{calc}||/\Sigma|F_{obs}|)$ of 0.0646 and a weighted R factor$(\Sigma w(|F_{obs}| - |F_{calc}|)^2/\Sigma w|F_{obs}|^2)$ of 0.0624. The goodness of fit, $(\Sigma w(F_{obs} - F_{calc})^2/(\text{no. of reflections - no. of variables}))^{\frac{1}{2}}$, equals 1.04. All hydrogen atoms were located and their positions refined in the final four cycles of refinement. Each was assigned an isotropic temperature factor one unit greater than that of the attached carbon atom. No atom moved more than 0.02σ in the final cycle of refinement. The centrosymmetric space group C2/c, which gave successful refine-

Table I

Positional and Thermal Parameters of the Non Hydrogen Atoms[a,b,c]

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Te	260(.3)	8368(.5)	4060(.7)	18(.2)	41(.4)	79(.6)	0(.3)	-1(.3)	-4(.6)
O	0	7651(6)	1/4	15(3)	39(6)	82(10)	0	2(4)	0
N	619(6)	9017(7)	6060(10)	40(4)	58(7)	125(12)	-8(4)	5(6)	-4(8)
C	974(6)	9605(8)	6611(10)	30(4)	37(6)	85(11)	8(4)	9(6)	3(8)
S	1459(2)	10431(3)	7369(4)	27(1)	76(2)	146(4)	-6(1)	2(2)	-46(3)
C(11)	1140(5)	7381(7)	4372(10)	14(3)	51(7)	96(12)	6(4)	0(5)	-6(8)
C(12)	1347(6)	7033(9)	5556(11)	26(4)	60(8)	80(12)	4(4)	-1(5)	-8(8)
C(13)	1927(7)	6387(9)	5702(15)	35(5)	64(10)	137(17)	11(5)	-19(8)	10(10)
C(14)	2271(7)	6071(10)	4725(15)	26(4)	56(9)	160(20)	11(5)	-4(8)	16(11)
C(15)	2053(6)	6382(9)	3577(13)	28(4)	62(9)	109(15)	1(5)	18(16)	8(9)
C(16)	1472(6)	7030(8)	3399(12)	25(4)	46(7)	112(14)	9(4)	8(6)	22(8)
C(21)	-497(5)	7529(8)	4928(10)	25(4)	67(8)	73(11)	0(4)	4(5)	2(8)
C(22)	-1010(7)	8017(12)	5546(13)	35(5)	100(11)	110(15)	4(6)	7(7)	-8(11)
C(23)	-1578(8)	7452(18)	-6010(17)	33(6)	164(23)	136(21)	1(9)	29(8)	0(17)
C(24)	-1613(10)	6484(16)	5856(18)	57(8)	119(17)	171(23)	-18(9)	32(10)	-28(12)
C(25)	-1089(9)	5983(13)	5260(18)	53(7)	88(12)	223(26)	-24(8)	36(11)	-8(15)
C(26)	-533(7)	6512(10)	4797(15)	39(5)	63(9)	179(19)	-21(6)	26(8)	-5(12)

a) All values x 10^4 except x and z for oxygen which are special positions.

b) Values in parentheses above and in Table II are estimated standard deviations in the last digits as derived from the inverse matrix of the final least-squares refinement cycle.

c) The temperature factors are of the form $\exp[-(h^2\beta_{11} + k^2\beta_{22} + \ell^2\beta_{33} + 2hk\beta_{12} + 2h\ell\beta_{13} + 2k\ell\beta_{23})]$.

ment, was assumed throughout the analysis. The thermal motions of atoms, as reflected by their rms displacements along the principal axes of the temperature factor ellipsoids, showed no anomalies and were reasonable in magnitude and direction.

The final positional and thermal parameters for the atoms are given in Tables I and II.*

Table II

Positional and Thermal Parameters of the Hydrogen Atoms[a]

Atom	x	y	z	B,Å^2
H(12)	112(6)	727(9)	615(12)	4.9
H(13)	208(7)	611(10)	654(13)	6.4
H(14)	258(7)	558(9)	479(12)	6.1
H(15)	223(7)	618(9)	288(12)	5.5
H(16)	136(6)	729(9)	257(11)	5.0
H(22)	-104(7)	875(10)	559(13)	6.7
H(23)	-180(9)	776(14)	632(17)	8.5
H(24)	-196(9)	601(12)	619(15)	9.1
H(25)	-110(7)	514(12)	523(14)	8.9
H(26)	-18(7)	624(9)	415(13)	7.0

a) The positional parameters have been multiplied by 10^3. Hydrogen atoms were assigned a temperature factor one unit greater than that of the last refined isotropic temperature factor of the attached carbon atom.

RESULTS AND DISCUSSION

The crystal structure of the organotellurium "anhydride," bis(isothiocyanatodiphenyltellurium(IV)) oxide, consists of $(Ph_2TeNCS)_2O$ mole-

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* The table of structure factors has been deposited as NAPS Document No.03094 (12 pages). Order from ASIS/NAPS, c/o Microfiche Publications, P.O. Box 3513, Grand Central Station, New York, N.Y. 10017. A copy can be secured by citing the document number, remitting $5.00 for photocopies or $3.00 for microfiche. Advance payment is required. Make checks payable to Microfiche Publications. Outside the United States and Canada, postage is $3.00 for a photocopy or $1.00 for a fiche.

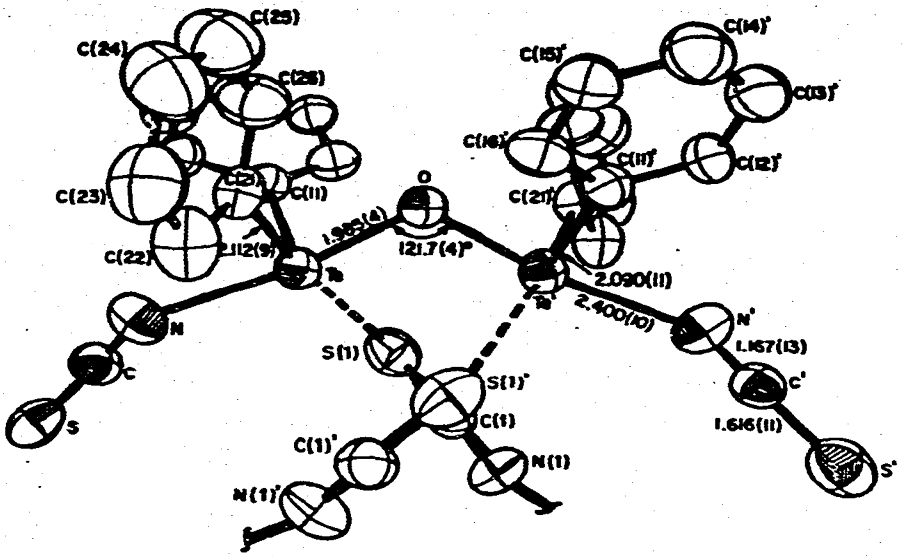

Figure 1. Atom labelling scheme and selected interatomic distances in $(Ph_2TeNCS)_2O$.

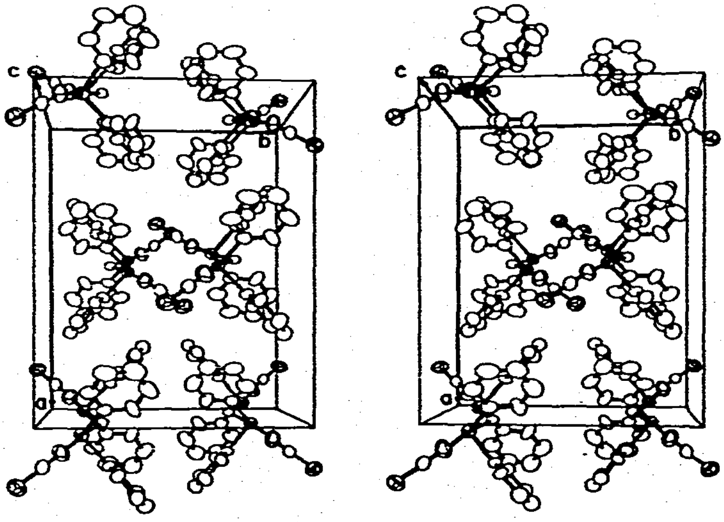

Figure 2. Stereoscopic view of the unit cell of $(Ph_2TeNCS)_2O$. Intermolecular interactions are omitted for clarity.

cules linked by intermolecular Te-S contacts. The molecular structure and unit cell contents are shown in Figures 1 and 2, respectively, and interatomic angles are given in Table III.

The $(Ph_2TeNCS)_2O$ molecule possesses two-fold symmetry, with the oxygen atom positioned on the rotation axis. The reported asymmetric unit is half the molecule about the axis 0, y, ½. There are four molecules in the unit cell.

Each tellurium atom is covalently bonded to four atoms, C(11), C(21), N, and O. A short intermolecular contact (3.416(3)Å) with a sulfur atom, S(1) [x, 2-y, -½ + z], completes a distorted square-pyramidal configuration about the tellurium. The Te-S(1) contact is considerably shorter than the sum of van der Waals radii, which is 3.86Å[11] or 4.05Å[12], depending on the source. Such contacts, or secondary interactions, serve to link the $(Ph_2TeNCS)_2O$ molecules into molecular chains running parallel to the c axis as illustrated in Figure 3. An additional sulfur atom, S(1)' [x̄, 2-y, 1-z], approaches the Te atom (3.796(3)Å) from the open side of

Table III

Interatomic Angles in Degrees

Atoms	Angle	Atoms	Angle
C(11)-Te-C(21)	97.2(4)	C(11)-Te-N	86.4(4)
N-Te-O	172.2(3)	C(21)-Te-N	86.0(4)
Te-N-C	145.3(9)	C(11)-Te-O	87.8(3)
N-C-S	179.3(1.0)	C(21)-Te-O	90.1(3)
S(1)-Te-S(1)'[a]	97.8(8)	Te-O-Te'	121.7(4)
O-Te-S(1)'	73.6(2)	C(11)-Te-S(1)'	161.3(3)
N-Te-S(1)'	112.4(2)	C(21)-Te-S(1)	173.7(3)
O-Te-S(1)	83.6(1)	C(11)-Te-S(1)	81.8(3)
N-Te-S(1)	100.1(3)	C(21)-Te-S(1)'	81.1(3)

[a] S(1): x, 2-y, -½+z; S(1)': x̄, 2-y, 1-z.

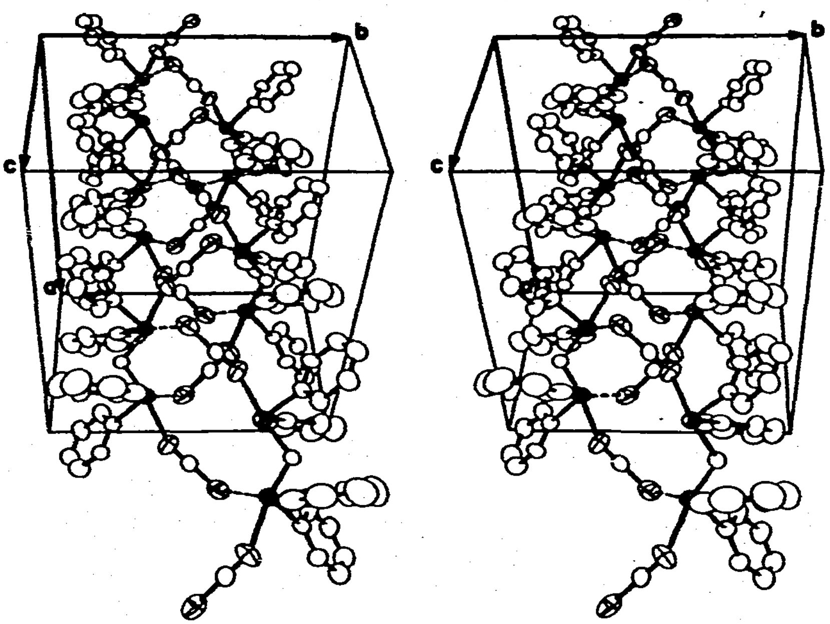

Figure 3. Molecular chaining of $(Ph_2TeNCS)_2O$ along the c axis.

the square pyramid. The much longer Te-S(1)' distance, along with the stronger interaction of this sulfur with the other tellurium in the molecule, Te', leads us to conclude that the square-pyramidal description is best in this case. Although reports of secondary interactions involving Te(IV) and halogen or oxygen are common,[31] only a few reports of such interactions between Te(IV) and S have appeared. The shorter Te-S contact is similar to the average Te-S distance in $Ph_3Te(NCS)$, 3.437(7)Å, where the Te···S and Te···N secondary interactions are intramolecular, and lead to the formation of dimeric and tetrameric species in the same unit cell.[5]

In spite of the wide variation in bond type and bond length the angles about the tellurium atom deviate from the ideal square-pyramidal values by an average of only 5.6°. The base of the square pyramid (N, O, C(21), S(1)) is essentially planar (see Table IV*). The tellur-

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* Table IV, Least-Square Planes and Dihedral Angles in $(Ph_2TeNCS)_2O$, has been deposited along with the Structure Factors Table. Please see preceding footnote.

ium atom deviates from this plane by less than 0.1Å, which is within the range of deviations observed for the other atoms in the plane. The tellurium atom is displaced 0.14Å from the axial plane (C(11), C(21), S(1)) toward the oxygen atom, and 0.09Å from the other axial plane (C(11), N, O). The dihedral angles between the basal or equatorial plane and the respective axial planes are 96° and 80°. The dihedral angle between the two axial planes is 97°. The fact that the C(11, 21)-Te-O angles are greater than the C(11, 21)-Te-N angles may indicate some repulsion between the phenyl groups and the localized electrons of the oxygen atom.

The C(11)-Te-C(21) angle is 97.2(4)°, and is within the range of C-Te-C angles reported in Table V for known structures of R_2TeX_2 and R_3TeX compounds. The angles range from 91 to 103°; that in α-$(CH_3)_2TeC\ell_2$[13] is 98.2(1.1)°, and the average in $Ph_3Te(NCS)$[5] is 97.3(6)°. The geometry of the R_2TeX_2 compounds has invariably been described as distorted trigonal bipyramidal with one equatorial site vacant. In at least one case, however, that of α-$(CH_3)_2TeC\ell_2$, two secondary interactions are reported which give tellurium an octahedral geometry.[13] In the R_3TeX compounds, the secondary interactions lead to both five (square pyramidal) and six coordinate (octahedral) tellurium. In the present case, although the spatial distribution of atoms around Te in the isolated asymmetric unit, $(Ph_2TeNCS)O$, is similar to that in the R_2TeX_2 compounds, the geometry of tellurium is best described as square pyramidal, similar to the five coordinate tellurium atoms in $Ph_3Te(NCS)$[5] and $Ph_3Te(NCO)$[6]. Inclusion of the longer Te-S contact (3.796(3)Å) would expand the coordination around tellurium to distorted octahedral.

The two independent Te-C distances do not differ significantly and have a mean value of 2.101(14)Å. The two distances (2.090(11) and 2.112(9)Å) are nearly identical to the sum of the single bond radii, 2.14Å,[12] and fall at the low end of the range of Te-C distances in Table V.

Table V

Structural Comparison of R_2TeX_2, $(R_2TeX)_2O$ and R_3TeX Compounds[a]

Compound	Te-C(Å)	C-Te-C(Deg)[b]	X-Te-X(Deg)	Ref.
<u>R_2TeX_2</u>				
α-$(CH_3)_2TeCl_2$	2.09(2)	98.2(1.1)*	172.3(3)*	13
α-$(CH_3)_2TeI_2$	2.14(7)	94(3)	177.9(7)	15
$(CH_3CH(Cl)CH_2)_2TeCl_2$	2.148(54)	98.2*	169.1*	14
Ph_2TeBr_2	2.14(3)	96.3(1.2)*	178.0(2)*	16
$(p\text{-}Cl\text{-}Ph)_2TeI_2$	2.12(3)	101.1(1.0)*	173.5(1)*	29
<u>$(R_2TeX)_2O$</u>				
$(Ph_2TeNCS)_2O$	2.101(14)	97.2(4)*	172.2(3)*	-
<u>R_3TeX</u>				
$(CH_3)_3Te^+$	2.07(8)	95(2)	-	30
$(CH_3)TeI_4^-$	2.15(8)*	-	-	30
$Ph_3Te(NCS)$	2.130(18)	97.3(6)	-	5
$Ph_3Te(NCO)$	2.130(22)	95.7(8)	-	6

[a] Starred values are single observed values; non-starred values are mean values.

[b] The actual range of all angles is from 91° to 103°.

The N-Te-O axis is non linear at 172.2(3)° and is bent 7.8° toward the phenyl groups. The plane of the N-Te-O bend is essentially perpendicular to the C(11)-Te-C(21) plane at 91.2° and is nearly coplanar with the Te-O-Te' plane at 8.5°. Interestingly, the bend is in the direction of that found in the dialkyltellurium dihalides,[13,14,15] but in the opposite direction of that found in Ph_2TeBr_2.[16] The less bulky ligands, N and O versus Br, may account for this difference.[17]

The oxygen atom connects two asymmetric units, and as such is two coordinate. Although axial elongation is the rule in the similar R_2TeX_2

molecules, we observe a Te-O distance (1.985(4)Å) only slightly shorter than the sum of the single bond radii, 2.03Å.[12] Additionally, we observe the Te-O-Te' angle to be 121.7(4)°, substantially greater than that expected for two coordinate oxygen involving only σ bonding. Similarly valued chalcogen-oxygen-chalcogen angles are found in $S_2O_7^{2-}$ (124.2(5)°)[18], SeO_2 (125(5)°)[19] and in solid $Te_2O_4 \cdot HNO_3$ (98.2(1.1)°-126.2(8)°).[20] Although this valence angle widening might conceivably be a steric effect between the non bonded tellurium atoms (vide infra), we cannot rule out the possible effect of π bonding between Te and O,[21,22] noting that a combination of axial elongation and π-bond shortening could conceivably lead to the observed equilibrium Te-O distance. In either case, the relatively "short" Te-O distance is consistent with a considerable trans influence as evidenced by the relatively long Te-N distance of 2.400(10)Å. For reference, the sum of the single bond radii is 2.07Å.[12] Although no strict comparison can be made, we note that the present Te-N distance has a bond order (∿.3) considerably less than those typically observed in axial elongation in the R_2TeX_2 dihalides (∿.5)[13,16]

An unambiguous standard reference for the pure single-bond Te-O distance is unavailable as are other pure single-bond X-O distances.[23] We note that the present Te-O distance is longer than the equatorial Te-O distances in trigonal bipyramidal α[24] and β[25] TeO_2 (α: 1.903(20); β: 1.88(2), 1.93(2)Å), but agrees with the slightly longer axial distances (α: 2.082(23); β: 2.07(2), 2.19(2)Å).

Because of the lack of structural data for other organochalcogen oxides having (VI)-O-(VI) moieties, it is difficult to assess steric effects on the Te-O-Te angle. The Te-Te' distance is 3.468(2)Å, 0.3Å longer than the Te-Te distance in β-TeO_2 (3.17(3)Å)[25], where tellurium-tellurium interaction is suspected, but 0.3Å shorter than the Te-Te distance in α-TeO_2 (3.740(1)Å) where no interaction is suspected.[24] The geometry of $(Ph_2TeNCS)_2O$ is such that some repulsion between the tellurium atoms might be expected. The molecule, however, does not appear to be sterically strained, for we note that the shortest carbon-carbon contacts between opposing phenyl groups of the asymmetric units are 3.84(2)Å (C(16)-C(26)') and 3.98(2)Å (C(16)-C(21)'). All intermolecular contacts aside from the two Te-S contacts, are greater than or equal to van der Waals distances. The nearest Te intermolecular contact, [$\bar{x}$, 2-y, 1-z], is 4.986(2)Å.

The phenyl rings are planar with no carbon atom deviating more than 0.02Å from the least-squares plane of the ring (Table IV). The dihedral angle between the two independent phenyl rings is 71.7°, and is within the range of dihedral angles between phenyl rings in the Ph_3Te^+ cation.[5] The mean C-C distance and C-C-C angle are normal with respective values of 1.38(7)Å and 120(5)°. The mean C-H distance of the ten values is 0.9(4)Å.

The isothiocyanate ion has a Te-N-C angle of 145.3(9)° and an N-C-S angle of 179.3(1.0)°. The N-C and C-S distances are 1.17(1) and 1.62(1)Å, respectively, and are similar to those in $Ph_3Te(NCS)$[5] and $Ph_3Se(NCS)$[27]. The C-S distance, however, is significantly shorter than that in KNCS,[28] 1.689(13)Å. Intermolecular interaction in $(Ph_2TeNCS)_2O$ causes Davydov splitting of the N-C asymmetric stretching frequency (2050, 2038 cm^{-1} (± 3 cm^{-1})) in both the infrared (KBr matrix and mineral oil mull) and Raman spectra of the compound.[8]

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