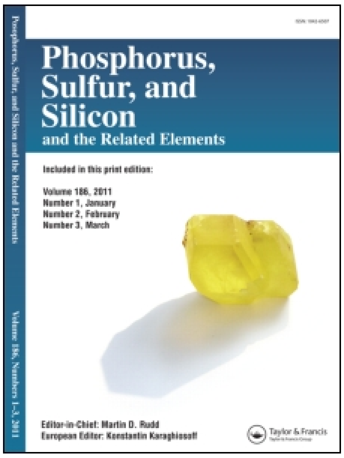

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Synthesis and Characterization of Three Coordinate "T-Shaped" Complexes of Tellurium (II) Incorporating a Naphthyl Tellurium Fragment

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Synthesis and Characterization of Three Coordinate "T-Shaped" Complexes of Tellurium (II) Incorporating a Naphthyl Tellurium Fragment

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*Several new three coordinate tellurium(II) complexes have been prepared by the addition of dinaphthyl ditelluride ($(C_{10}H_7)_2Te_2$) and a halogen (Br_2 or I_2) to various monodentate ligands known to coordinate through their terminal sulfur or selenium atoms to investigate possible electronic and steric effects associated with a large aryl group. Complexes of the type RTeX(L) are described here [R = naphthyl, X = Br, I, L = thiourea (**I** (X = Br), **II** (X = I)), tetramethylthiourea (**III**), selenourea (**IV**), tris(dimethylamino)phosphane selenide (**V**) and N-methylbenzothiazol-2(3H)-thione (**VI**)]. Evidence of the formation of new complexes is presented through microanalytical data and multinuclear NMR spectroscopy in addition to single crystal X-ray diffraction studies of (**I**) and (**III**). Structures are described in detail along with a comparison with related Te (II) complexes.*

Keywords Naphthyl tellurium; T-shaped; tellurium (II); thiourea; X-ray structure

INTRODUCTION

In a recent review, we described structural characteristics of hypervalent, three coordinate tellurium (II) complexes with a general formula RTeX(L), where X = halide, L = doubly bonded chalcogen atom from

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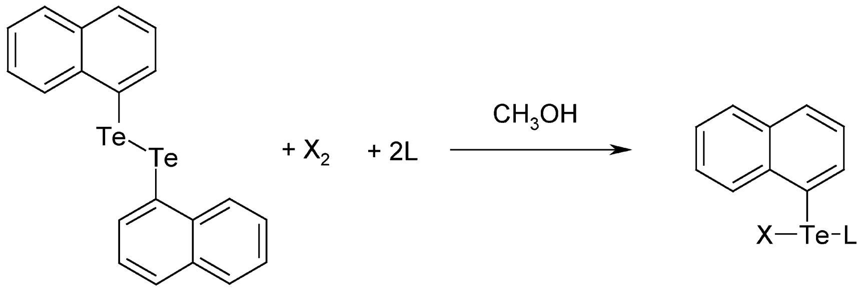
a neutral organic or phosphorus-containing ligand, and R is usually a phenyl group.[1] There are several known complexes of this type that show interesting variations in their structures, which are notably deviations away from the expected T-shape and the *trans* influence of halogens/ligands. Indeed, considerable structural variations between independent molecules of the unit cell have been observed. Although most of the reported complexes have the central tellurium atom coordinated to a (*substituted*) thiourea[2] or (*substituted*) selenourea,[3] to date all of the structurally characterized complexes have used a phenyl group as the aryl fragment attached to the tellurium.[4,5] The reason for this is probably the ready availability of diphenyl ditelluride (Ph_2Te_2) as a convenient starting material. In this article, we report the synthesis and characterization of three coordinate complexes of tellurium (II) that incorporate a naphthyl fragment as the aryl substituent. As part of our continuing studies to investigate the structural subtleties in these molecules, including crystal structure packing features and intermolecular, secondary interactions, we have prepared a series of naphthyl tellurium halide complexes with sulfur and selenium donor ligands.

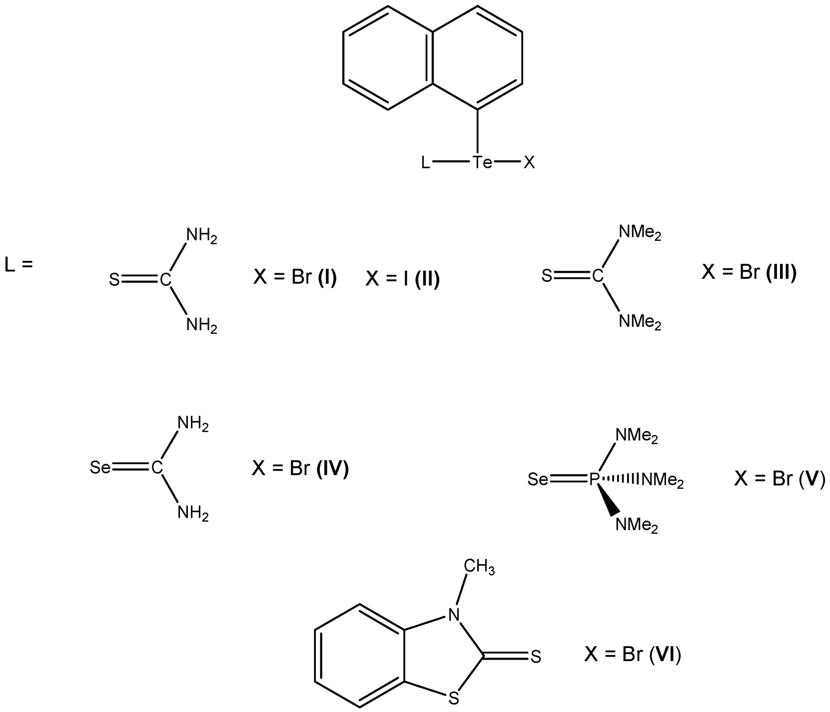
RESULTS AND DISCUSSION

Preparation

Products (**I–VI**) were obtained from the reaction according to Scheme 1 by reacting stoichiometric amounts of the diaryl ditelluride, $[(C_{10}H_7)_2Te_2]$ and the corresponding halogen (Br_2 or I_2) with the appropriate monodentate sulfur or selenium donor ligand in methanol at r.t. In general, there is an instantaneous reaction upon the addition of the halogen solution as the redox reaction takes place; a color change or formation of a precipitate is usually observed.



SCHEME 1 General synthetic scheme for compounds **I–VI**.



SCHEME 2 Structures of the ligands and numbering scheme in this work.

Scheme 2 shows the structures of the ligands used in this article. The resulting compounds were all readily isolated in moderate to high yields as yellow/orange/brown crystalline solids. They are insoluble in hydrocarbon solvents but can be recrystallized from hot methanol or tetrahydrofuran. All compounds were characterized by multinuclear (^{1}H, ^{13}C, ^{31}P) spectroscopy, and molecular structures for **I** and **III** were determined by single crystal X-ray diffraction.

NMR Spectra and Structure

NMR spectra for the title compounds are relatively straightforward. ^{1}H and ^{13}C NMR spectra feature signals from the naphthyl group and the ligands themselves. Upon complexation, there are only slight changes in the spectra compared to the free ligands due to the relatively weak bonds that the ligands form with tellurium. As in previously noted examples,[1,6] the shift is only significant at the C=S/C=Se linkage in the ^{13}C NMR spectrum where a downfield shift of about 3–10 ppm is observed. Compared to free naphthalene, the some of the ^{1}H and ^{13}C NMR shifts for the products exhibit a downfield shift resulting from coordination to the tellurium. Telluration of naphthalene results in a shift of about 10 ppm downfield for the carbons adjacent to the tellurium atom.[7] In all of the complexes reported here, these signals appear in the range δ 142–144 ppm. As expected, the quaternary carbon signals are somewhat weak. In fact, in the NMR spectra discussed in this article, the signal arising from the *ipso* carbon (attached to Te) is not observed at a predicted value of *ca.* 115 ppm; and hence, only 9 signals are observed for the naphthyl carbon resonances from around 126–144 ppm. This phenomenon has been observed and reported previously in the literature.[8] In the related compound $[(C_{10}H_7)Te(C_6H_5)]$, there are 2 signals corresponding to the Te—C carbons at 114.8 and 117.8 ppm.[9] In line with the coordination of the Se=C/S=C moiety to the tellurium atom, there are shifts in the ^{13}C (and to a lesser degree the ^{1}H) NMR signals from the ligand. For instance, in (**I**) and (**III**), there are upfield shifts of around 5–10 ppm between free thiourea and the complex, which is typical of shifts seen in thio and selenourea derivatives of Se/Te.[10] Unfortunately, many of the previously reported articles on three-coordinate complexes of Te(II) do not contain NMR data for which a comparison of spectra can be made.

Crystal and Molecular Structures of $[(C_{10}H_7)TeBr\{S{=}C(NH_2)_2\}]$ (I) and $[(C_{10}H_7)TeBr\{S{=}C(NMe_2)_2\}]$

The molecular structure of (**I**) with the atom numbering scheme is shown in Figure 1, and selected interatomic distances and bond angles are listed in Table I.

As expected for Te(II) compounds with three coordinating ligands and two lone pairs of electrons,[11] the geometry of these molecules is close to T-shaped, according to the Valence Shell Electron Pair Repulsion Theory. It is well known that the phenyl group in tellurium(II) chemistry leads to structures in which four coordinate, square planar complexes are observed in which one of the positions, which is opposite the phenyl group, is vacant. Indeed, Foss explains this by discussing the phenyl group as a “phenate” ion, one that is highly nucleophilic to the point where the tellurium 5p orbital is involved in bonding this groups so well that no “bonding power” is left to coordinate a halide in the fourth site, *trans* to the Te—C bond.[12] Haiduc et al. summarize the pertinent structural feature of the known RTe(L)X derivatives in their review article, pointing out the long distances at which the halide trans to the aryl (phenyl) group is observed.[11]

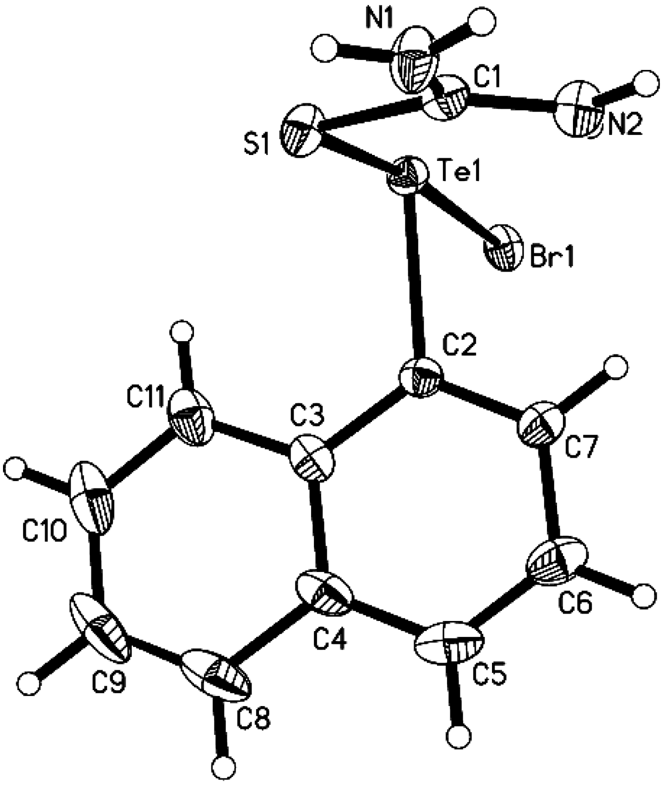


FIGURE 1 ORTEP plot of the molecule $[(C_{10}H_7)TeBr\{S{=}C(NH_2)_2\}]$(**I**). Atoms are drawn with 50% probability ellipsoids.

The naphthyl group is, as expected, planar and does not show any remarkable bond lengths or angles. The Te—C bond lengths fall well within those observed in typical aryl-tellurium bonds; for example, in the related compound [PhTe(tu)Br], a distance of 2.12 Å is

TABLE I Important Interatomic Distances (Å) and Bond Angles (°) for $[(C_{10}H_7)TeBr\{S{=}C(NH_2)_2\}]$ (I)

Te(1)-C(2)	2.129(5)	Te(2)-C(13)	2.132(5)
Te(1)-S(1)	2.5016(14)	Te(2)-S(2)	2.4776(14)
Te(1)-Br(1)	2.9700(6)	Te(2)-Br(2)	3.0740(6)
S(1)-C(1)	1.753(5)	S(2)-C(13)	1.754(6)
C(2)-Te(1)-S(1)	88.95(14)	C(13)-Te(2)-S(2)	91.55(15)
C(2)-Te(1)-Br(1)	85.59(13)	C(13)-Te(2)-Br(2)	87.49(15)
S(1)-Te(1)-Br(1)	173.94(4)	S(2)-Te(2)-Br(2)	178.07(4)
C(1)-S(1)-Te(1)	105.53(17)	C(13)-S(2)-Te(2)	105.78(19)

TABLE II A Comparison of the C=S Bond Length in (I) and Related Compounds

Compound	tu	PhTe(tu)Br	NaphTe(tu)Br (III)	tu_2I_2	$(tu)_3(I_2)_5$
C—S Bond Length (Å)	1.712(8)	1.78(6)	1.753(5); 1.754(6)	1.724	1.735(5)

seen. The two polymorphs of 2-naphthyl ditelluride have Te—C_{av} distances of 2.133 Å (monoclinic P2(1)/c) and 2.131 Å(monoclinic C2/c),[13] and the isomeric 1-napthyl ditelluride has a Te—C length of 2.135(3) Å.[14] Tellurium atoms connected to the naphthalene fragment in 1,8-bis(phenyltelluro)naphthalene show Te—C distances of 2.14(2) Å. Divalent complexes of tellurium have a tendency toward four coordination[15] in a square planar geometry; as has been previously seen, when a phenyl group is one of the ligands, there is a inclination toward the T-shaped three coordinate shape. We can add to this statement by saying that the presence of the naphthyl ligand also imposes a strong *trans* influence on the coordination geometry, so much so that there are no short contacts *trans* to this group. Reference [3] describes some three coordinate tellurium(II) compounds in which a "short" (ca. 3.8 Å) contact *trans* to the phenyl group is observed. Iodine adducts with thiourea (tu), such as (tu_2I_2), have been synthesized and structurally characterized[16] in addition to those containing interhalogens.[17] It is possible to compare the S=C bond length seen in (**I**) with those reported in related compounds. There is a small lengthening of the C=S distance upon coordination with the electron accepting $RTe^{(II)}X$ or isoelectronic I_2 moiety. (Table II). The structural chemistry of iodine adducts as charge transfer complexes has been well documented in reference [17] and articles cited within.

The Te—S distance in (**I**) falls within the range typically found in this type of three-coordinate complex. In (**I**), there are two independent molecules in the unit cell, and it is interesting to note that the bond lengths of Te—S and Te—Br show considerable variation, just as they do in the related molecule previously reported, [PhTeBr{*N*-methylbenzothiazole-2(3*H*)-selone}].[1] Table III summarizes some of the important bond lengths found in related published structures. The sum of the single bond radii (2.41 Å)[18] is shorter than the Te—S bond lengths for (**I**), and we can see that compared to this accepted value, the Te—S bonds are longer by +0.09/+0.07 Å. When looking at the Te—Br distances, the sum of the single bond radii (2.51 Å) is considerably shorter than the Te—Br distances in (**I**; +0.46/+0.56 Å) but not as great as those in [BrTePh(tu)].

TABLE III A Comparison of Important Bond Lengths Between This Work and Previously Published RTe(L)X Compounds

Compound	Te—S (Å)	S=C (Å)	Te—Br (Å)	Σ(Te—S) + (Te—Br) (Å)	Te—S=C (°)
BrTePh(tu) [2]	2.50(2)	1.78(6)	3.11(1)	5.61	108(2)
BrTeNapt(tu) (**I**)*	2.5016(14)	1.753(5)	2.9700(6)	5.4716	105.53(17)
	2.4776(14)	1.754(6)	3.0740(6)	5.5516	105.78(19)
ClTePh(tu) [2]	2.50(2)	1.78(6)	—	—	110(2)

*Two independent molecules in the unit cell.

Crystal and Molecular Structure of $[(C_{10}H_7)TeBr\{S{=}C(NMe_2)_2\}]$(III)

The molecular structure of (**III**) with the atom numbering scheme is shown in Figure 2, and selected interatomic distances and bond angles are listed in Table IV.

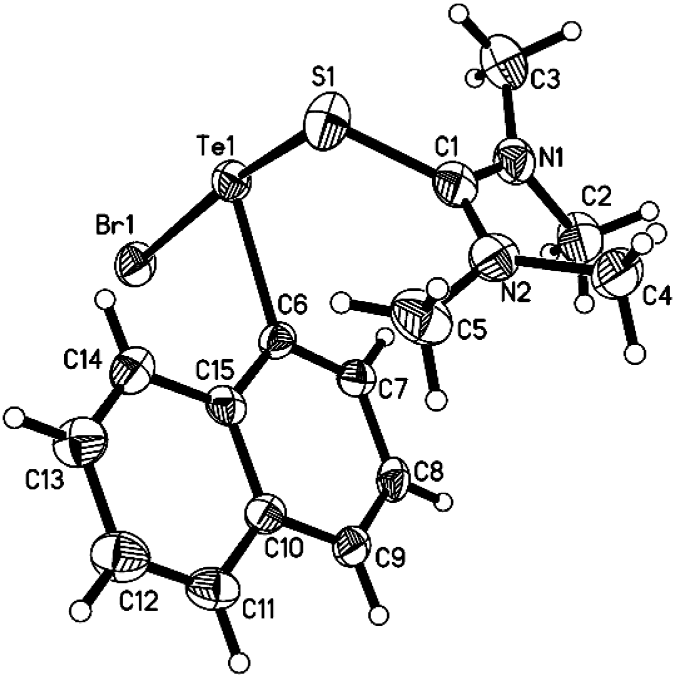


FIGURE 2 ORTEP plot of the molecule $[(C_{10}H_7)TeBr\{S{=}C(NMe_2)_2\}]$(**III**). Atoms are drawn with 50% probability ellipsoids.

TABLE IV Important Interatomic Distances (Å) and Bond Angles (°) for [($C_{10}H_7$)TeBr{S=C(NMe_2)$_2$}] (III)

Te(1)-C(6)	2.169(5)	N(1)-C(1)	1.333(5)
Te(1)-S(1)	2.5930(11)	N(2)-C(1)	1.335(5)
Te(1)-Br(1)	2.8093(5)	N(1)-C(2)	1.475(5)
S(1)-C(1)	1.740(4)	N(1)-C(3)	1.462(6)
		N(2)-C(4)	1.458(5)
		N(2)-C(5)	1.460(6)
C(6)-Te(1)-S(1)	87.71(13)	S(1)-Te(1)-Br(1)	171.14
C(6)-Te(1)-Br(1)	84.71(12)	C(1)-S(1)-Te(1)	103.09

As in (**I**), there are no significant deviations from the expected Te—C bond length, and the strong trans influence of the naphthyl group leaves no short (<4.0 Å) contacts, thereby confirming a T-shaped three coordinate complex. A 2c–2e covalent bond is the best description for this contact. In this complex, the Te—S bond is +0.18 Å longer than the single bond covalent radii, and the Te—Br length is +0.30 Å longer. These deviations are much shorter than the van der Waals contacts. We can clearly say that just as in the phenyl substituted complexes, the thiourea ligand exerts a much stronger *trans* influence than tetramethylthiourea. The sum of the Te—S and Te—Br bonds remains similar through both series of ligands despite the rather extensive lengthening of the Te—Br bond *trans* to the thiourea ligand in (**I**).

EXPERIMENTAL

Materials and Procedures

1-Napthyl ditelluride, thiourea, selenourea, 1,1,3,3-tetramethyl-2-thiourea, bromine, iodine, tris(dimethylamino) phosphine, and *N*-methylbenzothiazol-2-(3*H*)-thione were obtained from Aldrich Chemical Co. and used as supplied. All solvents were of ACS grade and used as supplied by Fischer Scientific. Tris(dimethylamino) phosphane selenide was prepared according to the method of Rauchle et al.[19] Melting points were uncorrected. NMR spectra were recorded on a Bruker AMX200 spectrometer: ^{1}H 250 MHz, ^{13}C 62.8 MHz, and ^{31}P 80.1 MHz. All manipulations were performed under an atmosphere of dry nitrogen using standard Schlenk line techniques.

General Procedure for the Preparation of Halonaphthyl(L)tellurium(II)

Into a Schlenk flask was added 1-naphthyl ditelluride and two stoichiometric equivalents of the monodentate ligand. Methanol (20 mL) was

TABLE V A Comparison of Important Bond Lengths Between This Work and Previously Published Group 16/L Compounds (L = tetramethylthiourea)

Compound	Te—S (Å)	S=C (Å)	Te—Br (Å)	Σ(Te—S) + (Te—Br) (Å)	Te—S=C (°)
BrTePh(tmtu) [4]	2.589(2)	1.765(8)	2.833(1)	5.422	96.7(3)
BrTeNapt(tmtu) (**III**)	2.5930(11)	1.740(4)	2.8093(5)	5.4023	103.19(13)
ClTePh(tmtu) [4]	2.5954(6)	1.745(6)	—	—	96.6(2)
$SeCl_2$(tmtu) [10]	—	1.767(10)	—	—	—
$SeBr_2$(tmtu) [10]	—	1.751(15)	—	—	—

added, and the solution was stirred for 1 h under nitrogen to dissolve all materials. One equivalent of the appropriate halogen dissolved in methanol (10 mL) was added via a syringe resulting in an immediate color change from red-orange to yellow/yellow-orange. The resulting solution was left to stir overnight, and the solvent was removed in vacuo. An addition of hexane (10 mL) resulted in the formation of microcrystalline precipitates, which were isolated by filtration. The recrystallization from methanol (5 mL) or tetrahydrofuran (10 mL) followed by a slow cooling at −20°C gave crystals of the 6 title compounds. (X-ray quality crystals were grown by slow cooling a tetrahydrofuran solution of (**I**) overnight. Details of the preparations, melting points, yields, and microanalyses are given in Table VII).

$[(C_{10}H_7)TeBr\{S{=}C(NH_2)_2\}]$ (**I**). ^1H NMR (CD_3OD, ppm) 3.43 (w, br, 4H, $S{=}C(N\mathbf{H}_2)_2$, 7.38–8.47 (m, 7H, naphthyl); ^{13}C NMR: 127.5–143.1 ($-\mathbf{C}_{10}H_7-$), 174.3 ($S{=}\mathbf{C}(NH_2)_2$). X-ray quality crystals were grown by slow cooling a tetrahydrofuran solution of (**I**) overnight.

$[(C_{10}H_7)TeI\{S{=}C(NH_2)_2\}]$ (**II**). ^1H NMR (CD_3OD, ppm) 3.48 (w, br, 4H, $SC(N\mathbf{H}_2)_2$), 7.32–8.49 (m, 7H, naphthyl); ^{13}C NMR: 127.4–143.6 ($-\mathbf{C}_{10}H_7-$), 174.6 ($S{=}\mathbf{C}(NH_2)_2$).

$[(C_{10}H_7)TeBr\{S{=}C(NMe_2)_2\}]$ (**III**). ^1H NMR (CD_2Cl_2, ppm): 2.78 (s, 12H, $S{=}C\{N(C\mathbf{H}_3)_2\}$), 7.26–8.56 (m, 7H, Napthyl); ^{13}C NMR: 43.9 (s, $S{=}C\{N(\mathbf{C}H_3)_2\}$, 126.3–143.0 ($-\mathbf{C}_{10}H_7-$), 184.7 (s, $S{=}\mathbf{C}\{N(CH_3)_2\}$). X-ray quality crystals were grown by a slow diffusion of *n*-hexane into a dichloromethane solution of (**III**).

$[(C_{10}H_7)TeBr\{Se{=}C(NH_2)_2\}]$ (**IV**). ^1H NMR (CD_3OD, ppm) 3.22 (w, br, 4H, $Se{=}C(N\mathbf{H}_2)_2$), 7.36–8.44 (m, 7H, naphthyl); ^{13}C NMR: 126.8–142.6 ($-\mathbf{C}_{10}H_7-$), 184.0 ($Se{=}\mathbf{C}(NH_2)_2$).

$[(C_{10}H_7)TeBr\{Se{=}P(NMe_2)_3\}]$ (**V**). ^1H NMR (CD_2Cl_2, ppm) 2.65, 2.69 (18H, d, $((N\mathbf{Me}_2)_3)$, 7.29–8.49 (m, 7H, naphthyl); ^{13}C NMR: 37.75, 37.69 $(N\mathbf{Me}_2)_3)$, 127.0–143.7 ($-\mathbf{C}_{10}H_7-$); ^{31}P NMR: 80.9 (s, $J(^{31}P\text{-}^{77}Se)$ 770 Hz).

TABLE VI Preparation and Elemental Analyses for RTe(L)X Derivatives

Starting materials						Elemental analysis found (calc.) %	
$(C_{10}H_7)_2Te_2$ (g, mmol)	X_2 (g, mmol)	L (g, mmol)	Product RTeX(L) (g, %)	M. p. (°C)	Color	*C*	*H*
0.200g/0.39	0.063g/0.39 (X = Br)	0.060/0.79	0.256, 78	156–157	Orange-yellow	37.44 (37.31)	3.85 (3.97)
0.100/0.20	0.051/0.20 (X = I)	0.030/0.40	0.165, 90	136–138	Brown-red	28.73 (28.86)	2.38 (2.42)
0.100/0.20	0.032/0.20	0.053/0.20	0.164, 88	140–142	Orange-yellow	38.65 (38.59)	4.00 (4.10)
0.100/0.20	0.032/0.20	0.049/0.40	0.150, 82	80–82	Brown	28.70 (28.87)	2.35 (2.42)
0.100/0.20	0.032/0.20	0.097/0.40	0.182, 79	108–109	Yellow	33.40 (33.31)	4.36 (4.37)
0.102/0.20	0.032/0.20	0.072/0.40	0.175, 85	139–140	Yellow	41.85 (41.90)	2.67 (2.73)

$[(C_{10}H_7)TeBr\{C_8H_7NS_2\}]$ (**VI**) ^{1}H NMR (CD_2Cl_2, ppm) 3.99 (s, N-C**H**$_3$), 7.22–8.42 (m, 11H [7H, naphthyl; 4H benzothiazole]); ^{13}C NMR: 34.1 (s, N—**C**H$_3$), 126.3–144.0 (—**C**$_{10}$H$_7$— and benzothiazole), 186.1 (s, **C**=S).

X-Ray Structure Determination

Crystallographic data were collected on crystals with dimensions 0.107 × 0.065 × 0.054 mm for **(I)** and 0.116 × 0.056 × 0.053 mm for

TABLE VII X-Ray Crystal Data and Structure Refinement for (I) and (III)

	(**I**)	(**III**)
Empirical formula	$C_{30}H_{38}Br_2N_4O_2S_2Te_2$	$C_{15}H_{19}BrN_2STe$
Formula weight	965.78	466.89
Temperature (K)	110(2)	273(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	P2(1)/c	P2(1)/c
Unit cell dimensions		
a (Å)	9.4867(2)	8.6259(2)
b (Å)	8.9333(2)	14.4617(2)
c (Å)	41.4603(10)	14.0078(2)
α(°)	90	90
β(°)	93.0540(10)	102.7960(10)
γ(°)	90	90
Volume (Å^3)	3508.67(14)	1704.01(5)
Z	4	4
D_{calc} (g cm^{-3})	1.828	1.820
Abs. coefficient (mm^{-1})	4.092	4.206
F(000)	1872	904
Crystal size (mm)	0.107 × 0.065 × 0.054	0.053 × 0.056 × 0.116
θ range for data collection	1.97–28.28	2.80–25.68
Reflections collected	36974	23885
Independent reflections	8648	3232
Refinement method	Full matrix least squares on F^2	Full matrix least squares on F^2
Data/restraints/parameters	8648/12/402	3232/0/222
Goodness-of-fit on F^2	1.152	1.135
Final R indices [$F^2 > 2\sigma(F^2)$]	$R_1 = 0.0444$; $wR_2 = 0.0904$	$R_1 = 0.0295$; $wR_2 = 0.0637$
R indices (all data)	$R_1 = 0.0550$; $wR_2 = 0.0932$	$R_1 = 0.0395$; $wR_2 = 0.0674$
Largest difference peak and hole (e/Å^3)	1.914; −1.485	1.052; −0.849

(III). Data were collected at 110 K on a Bruker X8 Apex using MoKα radiation ($\lambda = 0.71073$ Å). Crystallographic data for (**I**) and (**III**) are summarized in Table VI. All structures were solved by direct methods after the correction of the data using SADABS. All data was processed using the Bruker AXS SHELXTL software, version 6.10.[20] Unless otherwise noted, all non-hydrogen atoms were refined anisotropically, and hydrogen atoms were placed in calculated positions. The crystal structure of **(I)** contains two independent units and two solvent molecules of tetrahydrofuran. One of the solvent molecules is disordered over two positions and is refined as isotropic spheres. Compound **(III)** contains a naphthyl group disordered over two positions in a 78:22 ratio. The smaller portion of the disorder was refined as isotropic spheres.

SUPPLEMENTARY MATERIAL

Crystallographic data for the structural analysis of compounds (**I**) and (**III**) have been deposited at the Cambridge Crystallographic Data Center (CCDC numbers 279431 (**I**) and 279432 (**III**)). Copies of the information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: + 44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk or www.ccdc.cam.ac.uk).

CONCLUSIONS

We have successfully prepared several new complexes containing the naphthyl tellurium(II) fragment. As part of this study, we have been able to compare structures of the originally prepared phenyl tellurium(thiourea) derivatives with the two novel compounds described here. In addition, we are currently investigating the general use of other diaryl ditellurides as starting materials in the coordination chemistry of tellurium(II).

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