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The crystal structures of 2-(2'-pyridyl)phenyltellurium(II) bromide and of the inclusion compound bis[2-(2'-pyridyl)phenyltellurium(II) chloride] · *p*-ethoxyphenyl-mercury(II) chloride

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Abstract

In 2-(2'-pyridyl)phenyltellurium(II) bromide (1) the coordination about tellurium may be described as *pseudo*-trigonal bipyramidal with bromine (Te–Br = 2.707(11) Å) and nitrogen (Te–N = 2.236(11) Å) atoms occupying axial positions. The equatorial plane comprises a carbon atom (Te–C = 2.111(6) Å) and two lone pairs of electrons. There are no significant intermolecular interactions between the six independent molecules in the unit cell. Bis[2-(2'-pyridyl)phenyltellurium(II) chloride] · *p*-ethoxyphenylmercury(II) chloride (2) may be regarded as an "inclusion compound" obtained by replacement of two RTeX (X = Cl or Br) molecules by two *p*-ethoxyphenylmercury(II) chloride entities. There is approximately linear coordination about mercury (C–Hg–Cl = 179.2°(4), Hg–C = 2.044(14) and Hg–Cl = 2.328(4) Å) and 2-(2'-pyridyl)phenyltellurium(II) chloride, with a structure similar to that of (1) above (Te–N = 2.236(6), Te–Cl = 2.558(1), Te–C = 2.080(25) Å). There are no significant intermolecular contacts.

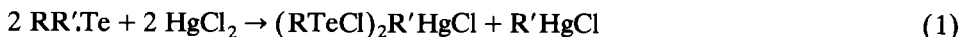
Introduction

The synthesis of 2-(2'-pyridyl)phenyltellurium(II) bromide, RTeBr (1), was previously reported [1]. A routine examination of the mass spectrum of this material revealed the presence of ions containing up to 3 tellurium atoms. An X-ray crystal structure determination was undertaken to see whether there were intermolecular interactions in the solid state, since such features in the mass spectra of organotellurium compounds can indicate association in the crystals.

The reaction of 2-(2'-pyridyl)phenyl(*p*-ethoxyphenyl)tellurium(II), RR'Te, with HgCl₂ in refluxing ethanol or acetonitrile was recently reported [2] to give a yellow

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material, $(\text{RTeCl})_2\text{R}'\text{HgCl}$ (**2**). The mass balance is in accord with the following stoichiometry:



The constitution of **2** was determined by analysis and by multinuclear NMR spectroscopy but no information was available about possible intermolecular interactions in the solid state. Suitable crystals of **2** have now been obtained and X-ray structural data are presented in this paper. It transpired that, conceptually, there are points of similarity between the structures of **1** and **2**, and so both structures are considered together.

Experimental and results

Crystallographic analyses

Orange crystals of **1** were obtained by slow evaporation of a DMF solution at room temperature and a suitable crystal was mounted on an Enraf–Nonius CAD-4 diffractometer (Aston University). Data were collected at room temperature by a $\omega-2\theta$ scan. As a check on crystal stability, three representative reflexions were measured every two hours. Lattice parameters were obtained by least squares analysis from the setting angles of 24 reflexions. Following unsuccessful attempts to solve the structure in space group $P\bar{1}$ it was eventually solved [3] by Patterson

Table 1
Crystallographic and experimental data

	1	2
Formula	$\text{C}_{11}\text{H}_8\text{BrNTe}$	$\text{C}_{30}\text{H}_{25}\text{Cl}_3\text{HgN}_2\text{OTe}_2$
Mol.wt.	361.7	991.7
Crystal size, mm	$0.35 \times 0.13 \times 0.23$	$0.2 \times 0.2 \times 0.1$
Space group	$P\bar{1}$	$P\bar{1}$
System	triclinic	triclinic
a , Å	11.217(2)	9.7060(10)
b , Å	12.026(2)	11.8079(9)
c , Å	13.346(4)	14.1891(7)
α , deg	77.87(2)	73.94(3)
β , deg	77.17(2)	84.82(3)
γ , deg	70.75(2)	74.89(3)
V , Å ³	1639	1508
Z	6	2
D_{calc} , g cm ⁻³	2.20	2.18
μ , mm ⁻¹	6.65	8.15
λ (Mo- K_α), Å	0.71069	0.71073
Temp, °C	25	20
No. of unique data	6520	4204
Observed data [$ F > 5\sigma F $]	4961	3206
Scan range (θ)	2–25	2–25
Weighting scheme	$w = 1/[\sigma^2(F) + 0.0003F^2]$	$w = 1/[\sigma^2(F) + 0.0016F^2 + 0.4]$
Absorption correction	empirical (DIFABS)	empirical (DIFABS)
R	0.050	0.049
R_w	0.058	0.069

Table 2

Fractional atomic coordinates ($\times 10^4$) with e.s.d.'s in parentheses and isotropic temperature factors ($\text{\AA}^2 \times 10^3$) for 1

	x	y	z	$U_{\text{eq}}^a / U_{\text{iso}}$
Te(e')	1083(23)	7287(21)	3573(20)	64
Te(f')	14414(21)	4603(21)	8699(21)	39
Te(a)	8554	9095	7794	46
Te(b)	6159(2)	1392(2)	14(2)	49
Te(c)	9719(2)	142(2)	4362(1)	47
Te(d)	7116(1)	2568(1)	6506(1)	52
Te(e)	4540(2)	4428(2)	842(2)	45
Te(f)	11388(2)	7031(2)	5661(2)	45
Br(a)	6174(3)	8965(3)	7945(3)	96
Br(b)	3630(2)	1542(3)	460(3)	81
Br(c)	12243(2)	53(3)	4098(3)	75
Br(d)	9465(3)	2764(4)	6262(3)	109
Br(e)	6541(3)	4720(3)	-636(3)	80
Br(f)	9034(3)	7032(3)	5433(3)	84
N(a)	10570(15)	8925(14)	7917(13)	44(4)
N(b)	8225(14)	1101(13)	-69(13)	42(4)
N(c)	7645(15)	526(15)	4371(14)	50(4)
N(d)	5090(17)	2704(16)	6480(15)	62(5)
N(e)	2856(14)	4495(14)	2172(13)	52(4)
N(f)	13303(15)	6702(14)	6097(14)	49(4)
C(1a)	8920(20)	7880(18)	9159(18)	54(6)
C(2a)	8009(19)	7352(17)	9817(17)	49(5)
C(3a)	8319(22)	6536(20)	10689(19)	68(6)
C(4a)	9470(24)	6249(23)	10944(23)	82(8)
C(5a)	10436(23)	6688(20)	10282(20)	69(7)
C(6a)	10134(19)	7568(18)	9378(17)	52(5)
C(7a)	11051(18)	8145(17)	8675(17)	47(5)
C(8a)	12295(21)	7919(20)	8834(20)	64(6)
C(9a)	13107(26)	8521(22)	8085(22)	81(8)
C(10a)	12619(23)	9301(21)	7308(20)	69(7)
C(11a)	11314(21)	9532(20)	7181(19)	63(6)
C(1b)	6665(18)	70(17)	1288(16)	48(5)
C(2b)	5770(20)	-466(18)	2011(17)	57(5)
C(3b)	6211(22)	-1307(20)	2820(20)	70(6)
C(4b)	7456(27)	-1660(26)	2937(26)	102(9)
C(5b)	8355(23)	-1176(20)	2224(19)	71(7)
C(6b)	7942(19)	-292(18)	1415(17)	54(5)
C(7b)	8767(17)	271(16)	634(15)	41(5)
C(8b)	10066(22)	35(21)	685(21)	72(7)
C(9b)	10774(21)	674(19)	-116(18)	62(6)
C(10b)	10208(21)	1464(20)	-860(19)	66(6)
C(11b)	8875(19)	1708(18)	-839(17)	52(5)
C(1c)	9410(15)	1476(14)	3058(14)	34(4)
C(2c)	10342(18)	1852(16)	2426(15)	47(5)
C(3c)	10056(19)	2749(17)	1590(17)	56(5)
C(4c)	8760(18)	3199(17)	1371(16)	51(5)
C(5c)	7872(19)	2712(17)	1992(17)	52(5)
C(6c)	8147(16)	1875(15)	2862(15)	41(5)
C(7c)	7165(17)	1368(16)	3565(15)	44(5)
C(8c)	5876(19)	1676(19)	3500(18)	60(6)
C(9c)	5120(20)	1158(18)	4204(18)	59(6)

Table 2 (continued).

	x	y	z	U_{eq}^a / U_{iso}
C(10c)	5598(20)	321(19)	5009(18)	61(6)
C(11c)	6848(20)	27(20)	5087(19)	62(6)
C(1d)	6699(19)	3787(17)	5170(17)	51(5)
C(2d)	7541(21)	4317(18)	4520(18)	57(6)
C(3d)	7225(26)	5057(23)	3614(23)	84(8)
C(4d)	5969(24)	5356(23)	3435(22)	81(8)
C(5d)	5127(23)	4777(20)	4077(20)	71(7)
C(6d)	5451(19)	4047(17)	4949(17)	51(5)
C(7d)	4581(19)	3443(18)	5678(17)	51(5)
C(8d)	3332(24)	3615(23)	5566(22)	76(7)
C(9d)	2691(26)	2949(23)	6283(22)	82(8)
C(10d)	3179(27)	2262(24)	7088(24)	87(8)
C(11d)	4444(23)	2074(22)	7215(21)	73(7)
C(1e)	3937(17)	6212(16)	1177(15)	47(5)
C(2e)	4566(18)	7053(17)	602(16)	51(5)
C(3e)	4104(21)	8172(20)	891(19)	66(6)
C(4e)	3098(21)	8479(21)	1692(19)	67(6)
C(5e)	2562(19)	7613(18)	2189(18)	56(5)
C(6e)	2961(16)	6514(15)	1936(14)	40(4)
C(7e)	2354(17)	5563(16)	2517(15)	47(5)
C(8e)	1340(16)	5720(16)	3325(14)	43(4)
C(9e)	896(20)	4745(18)	3801(18)	59(6)
C(10e)	1428(20)	3668(20)	3515(18)	62(6)
C(11e)	2455(21)	3471(21)	2686(19)	70(6)
C(1f)	11393(18)	5832(17)	7066(16)	47(5)
C(2f)	10340(19)	5450(18)	7576(17)	54(5)
C(3f)	10439(23)	4638(21)	8487(20)	72(7)
C(4f)	11571(25)	4282(24)	8916(24)	87(8)
C(5f)	12530(23)	4641(20)	8424(20)	70(7)
C(6f)	12495(19)	5505(18)	7473(17)	54(5)
C(7f)	13605(18)	5914(17)	6926(16)	48(5)
C(8f)	14737(21)	5633(20)	7239(20)	68(6)
C(9f)	15642(25)	6199(22)	6682(21)	83(8)
C(10f)	15386(21)	6995(20)	5800(18)	62(6)
C(11f)	14183(20)	7283(19)	5523(18)	55(6)

^a $U_{eq} = \frac{1}{3}$ (trace of the orthogonalized U_{ij} tensor).

and Fourier methods in space group $P1$ and refined [4] by least squares. Details of the data collection are given in Table 1. The tellurium and bromine atoms were refined anisotropically and all other non-hydrogen atoms isotropically. Hydrogen atoms were placed in calculated positions riding on their respective bonded atoms. The inverse structure gave virtually identical values for R and R_w .

Two of the molecules (labelled e and f in Table 2) are affected by disorder, corresponding to a 180° rotation of ca. 6% of the $C_{11}H_8NTe$ residues about the central bond so that the disordered tellurium atoms, Te(e') and Te(f') make contacts of 3.02(3) and 2.78(2) Å with neighbouring bromine atoms.

Computations were carried out on the University of Birmingham Honeywell Computer. Figure 1 was drawn with PLUTO-88 at the Manchester Computer Centre.

Crystallographic data for **2** were measured on an Enraf-Nonius CAD-4 four circle diffractometer (University of Surrey) by a ω - 2θ scan. Details of the data

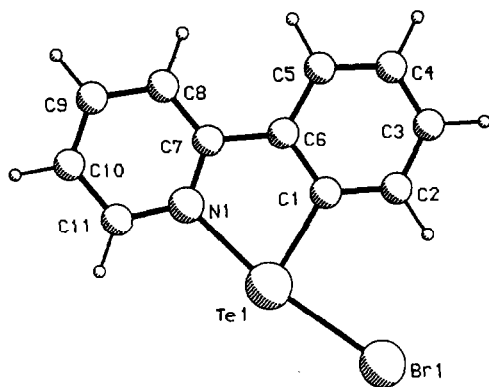


Fig. 1. One molecule of compound 1 showing the atom labelling.

collection are given in Table 1. Initial coordinates for the mercury, two tellurium atoms, and one six membered ring were derived from a Patterson search using the program SHELXS-86 [3]. Subsequent atoms were found from Fourier synthesis using SHELXS-76 [4] and SDP PLUS [5]. Hydrogen atoms were placed in calculated positions. Refinement was by full-matrix least-squares calculations using positional parameters and anisotropic temperature factors for all non-hydrogen atoms except

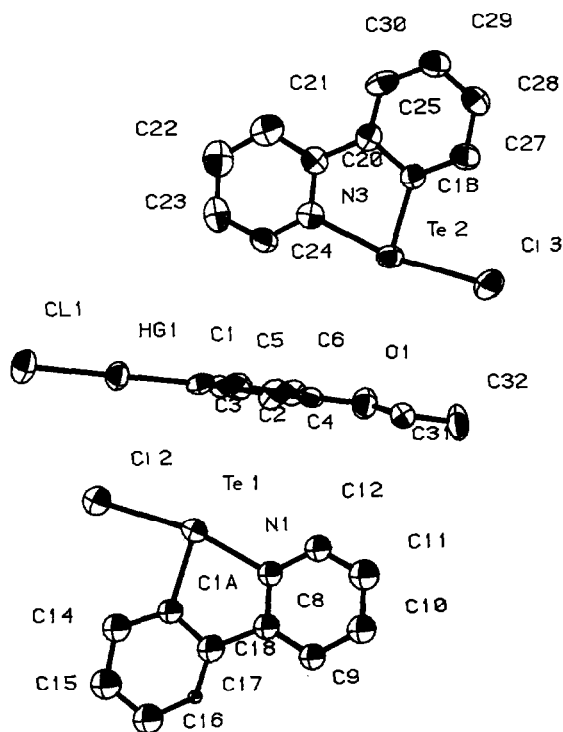


Fig. 2. The structure of $2C_{11}H_8ClNTe-C_8H_9ClHgO$ (compound 2) showing the atom labelling. Hydrogen atoms have been omitted.

Table 3

Fractional atomic coordinates ($\times 10^5$ for Hg; $\times 10^4$ for Te and Cl; $\times 10^3$ for C, N and O) and isotropic temperature factors ($\text{\AA}^2 \times 10^3$) for 2

Atom	x	y	z	U_{eq}^a / U_{iso}
Hg(1)	5729(7)	1458(6)	35215(5)	45
Te(1)	3330(1)	-13293(1)	3455(1)	39
Te(2)	13456(1)	7157(1)	8412(1)	39
Cl(1)	-1682(5)	310(4)	4265(3)	58
Cl(2)	601(5)	-12588(4)	3521(5)	71
Cl(3)	10733(5)	7821(4)	8570(4)	66
C(1)	254(2)	1(1)	285(1)	38
C(2)	396(2)	10(1)	137(1)	44
C(3)	267(2)	5(1)	185(1)	38
C(4)	518(2)	-25(1)	186(1)	35
C(5)	377(2)	11(1)	329(1)	48
C(6)	509(2)	-25(1)	284(1)	44
O(1)	643(1)	-34(1)	130(1)	51
C(31)	771(2)	67(1)	179(1)	48
C(32)	890(2)	77(2)	103(1)	67
N(1)	562(1)	-1429(1)	348(1)	36(3)
C(8)	589(2)	-1550(1)	377(1)	40(4)
C(9)	729(2)	-1621(1)	379(1)	40(4)
C(10)	837(2)	-1560(1)	352(1)	51(4)
C(11)	804(2)	-1435(2)	323(1)	55(4)
C(12)	665(2)	-1372(1)	320(1)	43(4)
C(1A)	329(2)	-1514(2)	398(1)	36(4)
C(14)	205(2)	-1552(1)	425(1)	49(4)
C(15)	218(2)	-1679(2)	462(1)	58(5)
C(16)	350(2)	-1762(1)	464(1)	54(4)
C(17)	453(2)	-1710(2)	434(1)	11(3)
C(18)	461(2)	-1598(1)	407(1)	45(4)
N(3)	1577(1)	621(1)	837(1)	40
C(20)	1603(2)	497(1)	864(1)	38
C(21)	1746(2)	429(2)	863(1)	54
C(22)	1852(2)	492(2)	834(1)	63
C(23)	1817(2)	619(2)	809(1)	58
C(24)	1679(2)	676(1)	812(1)	48
C(25)	1479(2)	448(1)	889(1)	39
C(1B)	1342(2)	535(1)	888(1)	37
C(27)	1222(2)	490(1)	917(1)	46
C(28)	1233(2)	366(1)	946(1)	51
C(29)	1361(*2)	284(1)	946(1)	49
C(30)	1480(2)	326(1)	918(1)	48

^a $U_{eq} = \frac{1}{3}$ (trace of the orthogonalized U_{ij} tensor).

ring atoms of the molecule labelled C1A, N1, C8–C18 (see Fig. 2), which were refined isotropically. The weighting scheme used is that of Killean and Lawrence [6]. Computations were carried out at the University of Surrey on a MicroVAX II computer. Figure 2 was drawn with ORTEP. Final atomic coordinates are listed in Table 3. Complete lists of bond lengths and angles, and lists of thermal parameters, hydrogen atom coordinates, and structure factors are available from the authors.

Discussion

The molecular structures of RTeBr (**1**) and (RTeCl)₂·R'HgCl (**2**) [R = 2-(2'-pyridyl)phenyl and R' = *p*-ethoxyphenyl] are shown in Figs. 1 and 2, respectively. Selected bond lengths and bond angles for **1** and **2** are listed in Table 4.

In both structures organotellurenyl halide RTeX (X = Cl, Br) molecules are present. In each case the coordination about tellurium can be considered as essentially *pseudo*-trigonal bipyramidal with the tellurated carbon and two lone pairs making up the equatorial coordination, and the halogen atom X and pyridyl-nitrogen atom the axial coordination. Since the position *trans* to the tellurated carbon is unoccupied, the molecule is essentially 'T'-shaped with the Te–C bond representing the stem.

In both **1** and **2**, the average Te–N distance is 2.24 Å. This value is in agreement with the postulated value for an axial Te–N [7] bond length and is comparable to those reported for papTe(SCN) (2.243 Å) [8] and papTeCl (2.23 Å) [9] (pap = 2-phenylazophenyl). The Te–N interaction holds the organic ligand in a planar geometry. In **1** the maximum angle between the phenyl and 2-pyridyl planes is only 3.08° (range 0.85–3.08° over the six independent molecules); in **2** this angle is similar, 2.19 and 2.40° in the two independent molecules. With the help of the two Te^{II} structures determined here and those reported elsewhere [1] a useful bank of information has been collected for molecules of the type RTeL [R = 2-(2'-

Table 4

Selected bond lengths (Å) and bond angles (°)

	1	2
Te–Br/Cl	2.707(11) ^a	2.558(1) ^b
Te–C	2.111(6) ^a	2.080(25) ^b
Te–N	2.236(11) ^a	2.236(6) ^b
Hg–Cl		2.328(4)
Hg–C		2.044(14)
N–Te–Br/Cl	169.2(2) ^a	168.4(4) ^b
N–Te–C	75.9(5) ^a	76.8(8) ^b
C–Te–Br/Cl	93.6(5) ^a	91.7(4) ^b
C–Hg–Cl		179.2(4) ^b

^a Mean of six values. ^b Mean of two values. E.s.d.'s in mean values calculated from $[\sum(\bar{x} - x_i)^2 / n(n-1)]^{1/2}$.

Table 5

Trans influence on Te···N interactions

Compound	Atom <i>trans</i> to N	Te···N distance (Å)	Ref.
RTeBr	Br	2.24 (mean)	present work
(RTeCl) ₂ R'HgCl	Cl	2.24 (mean)	present work
RTe(dmdtc)	S	2.354(4)	10
RTeR'	C	2.695(4)	10

R = 2-(2'-pyridyl)phenyl; R' = *p*-ethoxyphenyl; dmdtc = dimethyldithiocarbamate.

pyridyl)phenyl-] illustrating the dependence of the strength of the Te–N interaction on the group *trans* to the nitrogen atom. Relevant data are shown in Table 5. Strong Te–N interactions are seen when L = Cl, Br. However, the presence of a Te–C covalent bond *trans* to the Te ··· N vector significantly weakens the interaction. For these Te^{II} complexes a *trans* influence series can be suggested as follows: Br ~ Cl < S < C, in agreement with results obtained previously from consideration of other tellurium complexes [10]. For **1** all six independent molecules have the RTe group planar to within ± 0.08 Å with the bromine atom displacements from this plane in the range 0.10 to 0.23 Å. In **2**, the RTe groups are planar to within ± 0.06 Å with the chlorine atom displacements 0.08 and 0.13 Å. The small value for the C–Te–N bond angles (for **1** range 75.1–77.9° and for **2** 76.0–77.5°) is due to the constraint of the five-membered chelate ring. It is this constraint that distorts the Te coordination from ideal *pseudo*-trigonal bipyramidal values.

The Te–C distance (in **1** mean = 2.11 Å; in **2** mean = 2.08 Å) is in good agreement with the sum of the Pauling single bond covalent radii [11] and with the mean value given by Allen et al. [12] for Te–C(aromatic) of 2.116 Å.

In **1** the bromine atom lies *trans* to nitrogen, with N–Te–Br angles ranging from 168.1 to 169.9°. The mean Te–Br bond length of 2.71 Å may be compared with Te^{IV}–Br distances of 2.59–2.67 Å in RTeBr₃ and 2.63–2.76 Å in [2-(dimethylaminomethyl)phenyl]tellurium(IV) tribromide [13]. However, it is shorter than the distances in the Te^{II} complexes PhTetuBr (3.11 Å) [14], PhTe(etu)Br (2.83 Å) [15] and PhTe(esu)Br (3.05 Å) [16], in which the bromine atom is *trans* to carbon. In **2** the Te–Cl bond distance of 2.56 Å is similar to that in 2-phenylazophenyl-*C,N*-tellurium(II) chloride, in which Te–Cl is 2.55 Å [9].

There are no significant intermolecular contacts in **1**. Similarly, in the crystal of **2** there are no close contacts between the RTeCl and R'HgCl molecules; this crystal structure can be considered as an inclusion compound.

In the R'HgCl moiety each mercury atom is linearly coordinated to a chloride and a carbon atom. The HgCl and Hg–C distances of 2.33 Å and 2.04 Å, respectively, are in close agreement to those reported by Constable [17] for 2-(2'-pyridyl)phenylmercury(II) chloride, in which the two crystallographically independent molecules have Hg–Cl 2.31 and 2.34 Å and Hg–C both 2.07 Å. The phenyl group is planar to within ± 0.02 Å, with the mercury and oxygen atoms displaced 0.1 Å and 0.03 Å from this plane.

Conceptually the structures **1** and **2** described in this paper are related. Compound **1** has six RTeBr molecules in the unit cell and there is no significant intermolecular interaction. Compound **2** also has six molecules in the unit cell with no obvious intermolecular interactions between them; compound **2** is in a sense an "inclusion compound" derived from a structure such as **1** by replacement of two RTeX molecules by two R'HgCl molecules.

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