B3	0.4443 (3)	0.8737 (5)	0.2746 (4)	0.023 (2)
B4	0.4103 (3)	0.7869 (6)	0.3936 (4)	0.025 (2)
B5	0.4443 (4)	0.9014 (6)	0.5005 (5)	0.032 (2)
B6	0.4097 (3)	0.9873 (6)	0.3817 (4)	0.027 (2)
B7	1/2	1.0415 (7)	0.3107 (7)	0.022 (2)
B8	1/2	1.0593 (9)	0.4470 (6)	0.030 (3)
C9	0.4092 (3)	0.4908 (5)	0.2157 (3)	0.022 (1)
C10	0.3533 (3)	0.3806 (5)	0.2525 (4)	0.028 (1)
C11	0.2822 (3)	0.3428 (6)	0.1975 (4)	0.035 (2)
C12	0.2659 (3)	0.4129 (6)	0.1033 (4)	0.034 (2)
C13	0.3206 (3)	0.5208 (6)	0.0640 (4)	0.036 (2)
C14	0.3932 (3)	0.5586 (5)	0.1198 (4)	0.026 (1)

† Fixed to define origin.

Table 2. *Selected geometric parameters* (Å, °)

P—C1	1.871 (6)	C1—B4	1.729 (6)
P—C9	1.828 (4)	C2—B4	1.714 (6)
C1—C2	1.666 (9)	C2—B5	1.705 (8)
C1—B3	1.732 (7)		
C1—P—C9	104.7 (2)	P—C1—B4	113.6 (3)
C9—P—C9^{i}	105.6 (2)	P—C9—C10	113.6 (3)
P—C1—C2	108.5 (4)	P—C9—C14	127.9 (3)
P—C1—B3	129.7 (3)		
C9—P—C1—C2	124.6 (1)	C9—P—C1—B4	59.4 (4)
C9—P—C1—B3	−13.3 (4)		

Symmetry code: (i) $1 - x, y, z$.

Non-H atoms were refined with anisotropic displacement parameters and H atoms of the carborane cage with fixed isotropic displacement parameters. H atoms of the phenyl ring were placed at calculated positions (C—H = 0.95 Å and U_H equal to U_{eq} of carrying atom) and not refined.

Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993*a*). Data reduction: *TEXSAN* (Molecular Structure Corporation, 1993*b*). Structure solution: *SHELXS*86 (Sheldrick, 1985). Structure refinement: *Xtal*3.2 (Hall, Flack & Stewart, 1992). Molecular graphics: *ORTEP*II (Johnson, 1976) in *Xtal*3.2. Preparation of material for publication: *ATABLE* and *BONDLA* in *Xtal*3.2.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1245). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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cis-(Diethyldithiocarbamato)diiodo(phenyl)tellurium(IV), $PhTe(S_2CNEt_2)I_2$, and its Methoxy-Substituted Mixed Br/I Analogue *cis*-Bis(0.4-bromo/0.6-iodo)(diethyldithiocarbamato)(4-methoxyphenyl)tellurium(IV), *p*-$MeOC_6H_4Te(S_2CNEt_2)(Br_{0.4}I_{0.6})_2$

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Abstract

The Te^{IV} complexes $PhTe(S_2CNEt_2)I_2$, (1), and *p*-$MeOC_6H_4Te(S_2CNEt_2)(Br_{0.4}/I_{0.6})_2$, (2), have been synthesized by reacting $PhTeI_3$ with NaS_2CNEt_2, and *p*-$MeOC_6H_4Te(S_2CNEt_2)_2I$ with Br_2, respectively. In (2), both I atoms are partially replaced by Br atoms in a 3:2 ratio. The structures display distorted octahedral Te coordination with two symmetrically coordinated S atoms [Te—S 2.550 (2)–2.569 (2) Å in three independent molecules of (1) and 2.523 (1) and 2.535 (1) Å in (2)] and with two *cis*-disposed halogen atoms [Te—I 2.941 (1)–2.986 (1) Å in (1) and 3.003 (4) and 3.049 (3) Å in (2); Te—Br 2.962 (8) and 2.967 (8) Å in (2)] in equatorial positions. The aryl group is axial in both complexes [Te—C 2.137 (6)–2.146 (6) and 2.123 (5) Å in (1) and (2), respectively] and the second axial posi-

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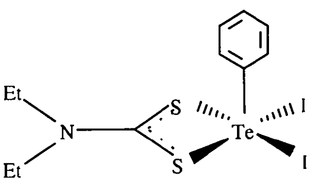
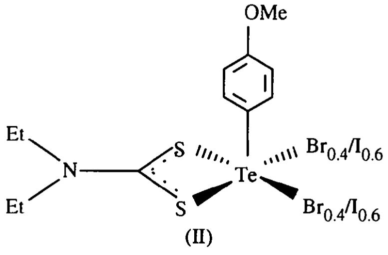
tion is occupied by a halogen atom of a neighbouring molecule [Te···I 3.898 (1)–4.233 (1) Å in (1); Te···I 3.872 (3) Å and Te···Br 3.676 (6) Å in (2); *trans* angles C—Te···I(Br) 153.4 (2)–177.7 (2)°] so that the molecules are joined into (quasi)centrosymmetric pairs by these secondary interactions.

Comment

The structures of three symmetrically independent molecules of (1) and the molecule of (2) are very similar. In all cases, the Te atom has a distorted octahedral (tetragonal bipyramidal) environment with the symmetrically coordinated bidentate *S*,*S*-chelate dithiocarbamate ligand and two *cis*-disposed halogen atoms in its equatorial plane. An aryl group is situated in one axial position (Figs. 1 and 2). The second axial site is weakly coordinated by a halogen atom of a neighbouring complex. It is interesting that the manner of molecular association is the same both in the two independent dimers of (1) (the first one is quasi-centrosymmetrical, and the second one is derived by a crystallographic centre of symmetry) and in the centrosymmetric dimer of (2) (Figs. 3 and 4). The comparative weakness of these additional interactions is manifested by their dependence on crystal packing effects: in the first dimer of (1) the Te···I contacts are 4.192 and 4.233 (1) Å [*trans* angles C—Te···I 153.4 and 158.8 (2)°], but in the second dimer of (1) and in (2) these contacts are substantially shorter, being 3.898 (2) and 3.872 (3) Å, respectively [*trans* angles 170.2 (2) and 175.6 (1)°]. The Te···Br distance in (2) (corresponding to the dibromine-substituted minor component) is 3.676 (6) Å [*trans* angle C—Te···Br 177.7 (2)°]. The above distances may be compared with the typical van der Waals contacts of 3.91 and 4.04 Å for Te···Br and Te···I, respectively (Bondi, 1964).



(I)



(II)

Generally, secondary bonding interactions of this type are rather common for Te^{IV} complexes, especially for the aryl-substituted derivatives [in spite of the widespread conception of a 'stereochemically active lone electron pair' in these compounds (Husebye, 1983)]. There are many examples, when some easily polarizable electronegative groups are situated at this 'empty' place (see, for example, Husebye, Kudis & Lindeman, 1995*a*), which would be impossible if the lone electron pair of the Te atom was really stereochemically active (occupies this 'empty' position). Apparently, in aryl-substituted Te^{IV} complexes the lone electron pair may be considerably inert (*e.g.* localized in the *s* orbital of Te) due to the strong negative σ-inductive effect of the aryl group.

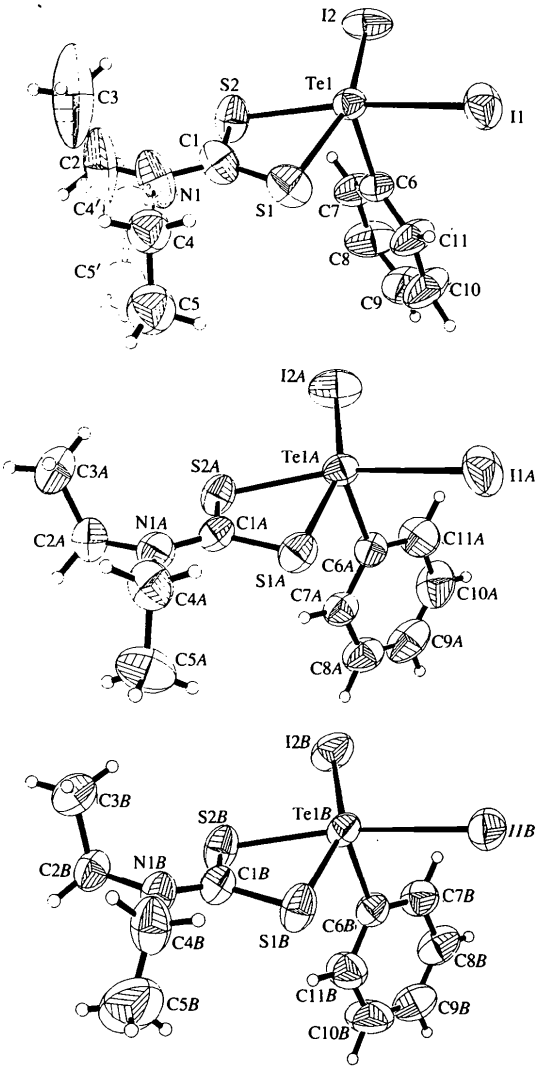


Fig. 1. Perspective views of the three symmetrically independent molecules of (1) in a uniform orientation, showing the labelling of non-H atoms. The alternative position for the disordered ethyl group in the first molecule (top) is shown by dashed lines. The second molecule (middle) is shown as inverted (relative to Table 1) for comparison with the other molecules. Displacement ellipsoids are plotted at the 50% probability level.

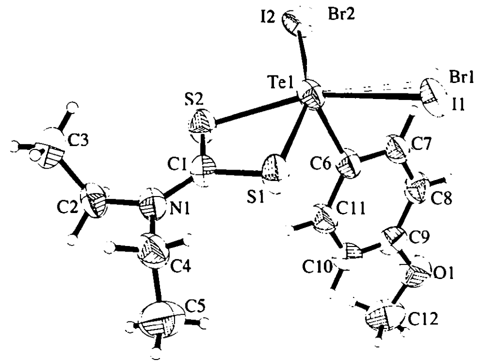


Fig. 2. Perspective view of (2) showing the labelling of the non-H atoms. The less populated Br-atom positions and the corresponding bonds are depicted by dashed lines. Displacement ellipsoids are plotted at the 50% probability level.

The manner of valence coordination found in (1) and (2) is fairly new for $\mathrm{Te^{IV}}$ compounds: there is only one example of a complex with the same type and geometry of coordination, dibromo(*O*,*O*′-dimethyldithiophosphato-*S*,*S*′)-*p*-methoxyphenyltellurium(IV), (3) (Chadha, Drake, McManus, Quinlan & Sarkar, 1987), but the dithiophosphate ligand seems to exert a weaker *trans* influence than the dithiocarbamate one. Thus, the Te—S distances in the complexes (1) [2.550–2.569 (2) Å] and (2) [2.523 and 2.535 (1) Å] are noticeably shorter than in (3) (2.632 and 2.728 Å), and Te—Br distances in (2) [2.962 (8) and 2.967 (8) Å] are much longer than in (3) (2.616 and 2.676 Å).

Moreover, in (1) and (2) the Te—S bonds are shorter than those of analoguous monohalogenobis(dithiocarbamato) complexes that have pentagonal coordination in the equatorial plane, evidently as a result of two 'pure' S—Te—Hal *trans* interactions [in (1) and (2) the *trans* angles S—Te—I/Br are in the range 151–162°]. For example, in the structure of bis(diethyldithiocarbamato)(iodo)(phenyl)tellurium(IV), (4) (Husebye & Maartmann-Moe, 1994), the lengths of Te—S bonds opposite to the halogen atom are 2.629 (1)–2.661 (1) Å (*trans* angles S—Te—I 141.1–143.0°), and in bromobis(dimethyldithiocarbamato)(4-methoxyphenyl)tellurium(IV), (5) (Husebye, Kudis & Lindeman, 1995*b*), they are 2.623 (1) and 2.629 (1) Å (*trans* angle S—Te—Br 142.1°).

On the other hand, the Te—I distances in (1) and (2) (2.941–3.049 Å) are also shortened compared with those of (4) [3.100 (1) and 3.151 (1) Å]. This may be a result both of the smaller sterical overload of the octahedral complexes (1) and (2) and of the joint *trans* influence of the two thio ligands in the pentagonal bipyramidal complex (4).

The Te—I bonds in (1) and (2) also seem to be influenced by intermolecular interactions. While in the first (weakly bonded) dimer of (1) the Te—I bond lengths vary 'statistically' within the range 2.941 (1)–2.952 (1) Å, in the second (more strongly bonded) dimer of (1) the bridging Te—I bond is 0.016 (1) Å longer than the terminal one, and in the dimer of (2) this difference increases to 0.046 (3) Å. However, the Te—Br distances are practically equal in (2) [2.962 (8) and 2.967 (8) Å], though in (3) the bridging Te—Br distance is markedly longer than the terminal one (2.676 and 2.616 Å, respectively), in good agreement with the shorter intermolecular Te· · ·Br distance (3.810 Å, *trans*-angle C—Te· · ·Br 159.4°).

The influence of the secondary bonds on the length of the *trans*-disposed Te—C(Ar) bond is not so marked. In the three independent molecules of (1), the Te—Ph bond lengths are practically equal within experimental accuracy [2.137 (6)–2.146 (6) Å]; in (2), the Te—Ar bond is even slightly shorter [2.123 (5) Å], apparently as a result of the of the electronegative *p*-methoxy-substituent.

The orientation of the benzene group relative to the equatorial plane of the Te atom is staggered in both structures: one of the *o*-C atoms is situated between two halogen ligands, and the other is between two S atoms

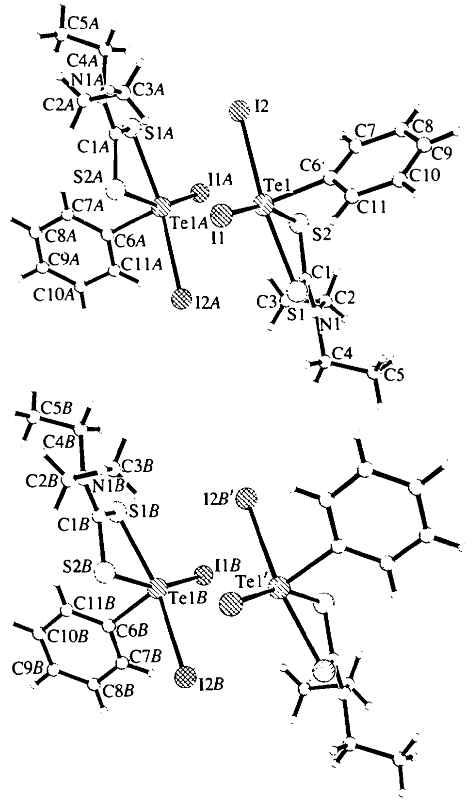


Fig. 3. Perspective view of the two symmetrically independent dimers of (1) in the crystal [situated in a general position with an approximate non-crystallographic centre of symmetry (top), and derived by a crystallographic centre of symmetry (bottom)]. Intermolecular Te· · ·I contacts are shown by dashed (longer) or unbroken (shorter) lines.

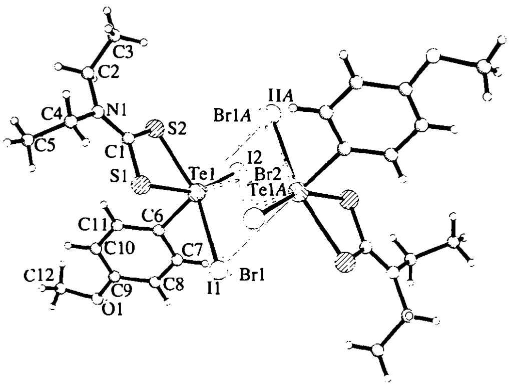


Fig. 4. Perspective view of the dimer of (2) in the crystal. The less populated Br-atom positions and corresponding bonds are depicted by dashed lines.

(see Figs. 3 and 4, Tables 3 and 4). The only exception is the first symmetrically independent molecule of (1), in which the phenyl group is turned facially towards the dithiocarbamate ligand, and each of its *o*-C atoms is placed between S and I atoms. This is a good example of the effect of packing on the molecular conformation. In any case, both conformers seem to have a minimum of steric strain.

The *p*-methoxyphenyl ligand in (2) has the usual flattened π-conjugated structure: the torsion angle C—C—O—C is only 15.0 (9)°. A usual π-conjugated structure is also observed for the dithiocarbamate ligands: the C(sp^2)—N bond distances are 1.29 (1)–1.34 (1) Å; the twist along this bond does not exceed 3.6 (5)°; the configuration of the N atoms is planar within 0.016 (8) Å [the only exception is the disordered ligand in the first symmetrically independent molecule of (1) in which the twist along the C(sp^2)—N bond is 5–10 (1)°]. In all cases, the ethyl groups of the dithiocarbamate ligands have an *anti* orientation.

Experimental

(1) was synthesized from Ph_2Te_2 by addition of elemental I_2 in chloroform solution, followed by treatment with an excess of NaS_2CNEt_2 in CH_2Cl_2. Red crystals formed upon addition of *n*-hexane/diisopropyl ether and were then recrystallized from a ethanol/dichloromethane (1:1) mixture. (2) was synthesized by reacting (p-$MeOC_6H_4$)$Te(S_2CNEt_2)_2I$, dissolved in CH_2Cl_2, with Br_2, dissolved in CCl_4 (molar ratio 2:1). Recrystallization from a combination of ethanol/dichloromethane/toluene (2:1:1 volume) yielded a mixture of yellow crystals of (p-$MeOC_6H_4$)$Te(S_2CNEt_2)_2(Br_{0.4}/I_{0.6})$ (Husebye, Kudis & Lindeman, 1995*a*) and red crystals of compound (2).

Compound (1)

Crystal data

$[TeI_2(C_5H_{10}NS_2)(C_6H_5)]$
$M_r = 606.76$
Triclinic
$P\bar{1}$
$a = 9.126\,(1)$ Å
$b = 12.631\,(1)$ Å
$c = 24.532\,(4)$ Å
$\alpha = 97.32\,(1)°$
$\beta = 99.59\,(1)°$
$\gamma = 105.46\,(1)°$
$V = 2643.0\,(6)$ Å^3
$Z = 6$
$D_x = 2.287$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 24 reflections
$\theta = 10–11°$
$\mu = 5.412$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.52 × 0.22 × 0.13 mm
Red

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/1.3\theta$ scans
Absorption correction: numerical (Gaussian method)
$T_{min} = 0.328$, $T_{max} = 0.560$
15 345 measured reflections
15 345 independent reflections

9160 observed reflections $[I > 2\sigma(I)]$
$\theta_{max} = 29.96°$
$h = 0 \rightarrow 12$
$k = -17 \rightarrow 17$
$l = -34 \rightarrow 33$
3 standard reflections
frequency: 120 min
intensity decay: 2.8%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0465$
$wR(F^2) = 0.1138$
$S = 1.221$
15321 reflections
465 parameters
H atoms: riding and rotating model with fixed U_{iso}
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 8.0474P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.039$
$\Delta\rho_{max} = 2.397$ e Å^{-3} (adjacent to Te and I)
$\Delta\rho_{min} = -1.538$ e Å^{-3} (adjacent to Te and I)
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2) *for* (1)

U_{iso} for C4, C4′, C5, C5′; $U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	U_{eq}/U_{iso}
Te1	0.73354 (5)	0.02302 (3)	0.16876 (2)	0.04526 (10)
I1	0.92681 (7)	0.13829 (4)	0.27996 (2)	0.06904 (15)
I2	0.41210 (6)	0.02446 (5)	0.16935 (3)	0.0782 (2)
S1	0.9671 (2)	−0.0167 (2)	0.13380 (9)	0.0659 (5)
S2	0.6416 (2)	−0.0939 (2)	0.06922 (9)	0.0681 (5)
N1	0.8650 (10)	−0.1736 (7)	0.0405 (3)	0.089 (2)
C1	0.8321 (9)	−0.1015 (7)	0.0779 (3)	0.064 (2)
C2	0.7529 (18)	−0.2398 (12)	−0.0098 (6)	0.138 (6)
C3	0.662 (2)	−0.3455 (15)	0.0023 (7)	0.211 (12)
C4†	1.050 (2)	−0.1625 (17)	0.0515 (8)	0.070 (5)
C4′†	0.999 (2)	−0.2039 (16)	0.0450 (8)	0.064 (4)
C5†	1.145 (3)	−0.0831 (19)	0.0221 (10)	0.089 (6)
C5′†	1.099 (3)	−0.1390 (18)	0.0122 (9)	0.080 (5)
C6	0.7774 (8)	0.1749 (5)	0.1362 (3)	0.0497 (14)
C7	0.6663 (9)	0.1911 (7)	0.0952 (4)	0.068 (2)
C8	0.6989 (11)	0.2880 (9)	0.0729 (4)	0.091 (3)
C9	0.8372 (13)	0.3663 (8)	0.0909 (4)	0.092 (3)
C10	0.9521 (13)	0.3489 (8)	0.1309 (4)	0.102 (4)
C11	0.9207 (10)	0.2528 (7)	0.1531 (3)	0.081 (3)
Te1*A*	0.42807 (5)	−0.30588 (3)	0.17498 (2)	0.04752 (10)
I1*A*	0.21936 (9)	−0.41632 (5)	0.06601 (2)	0.0835 (2)
I2*A*	0.75325 (7)	−0.30277 (6)	0.17620 (3)	0.0887 (2)
S1*A*	0.1961 (2)	−0.26413 (15)	0.20876 (8)	0.0555 (4)
S2*A*	0.5170 (2)	−0.19752 (15)	0.27613 (8)	0.0572 (4)

N1*A*	0.2990 (7)	−0.1071 (5)	0.3013 (3)	0.0551 (13)
C1*A*	0.3334 (7)	−0.1804 (5)	0.2664 (3)	0.0486 (14)
C2*A*	0.4103 (10)	−0.0396 (7)	0.3519 (3)	0.071 (2)
C3*A*	0.4966 (12)	0.0716 (7)	0.3419 (5)	0.095 (3)
C4*A*	0.1450 (10)	−0.0872 (7)	0.2912 (4)	0.070 (2)
C5*A*	0.0377 (11)	−0.1521 (9)	0.3224 (5)	0.090 (3)
C6*A*	0.3861 (8)	−0.4658 (5)	0.2005 (3)	0.0492 (14)
C7*A*	0.3143 (8)	−0.4849 (6)	0.2450 (3)	0.054 (2)
C8*A*	0.2844 (9)	−0.5911 (7)	0.2587 (4)	0.069 (2)
C9*A*	0.3306 (11)	−0.6711 (7)	0.2312 (5)	0.083 (3)
C10*A*	0.4033 (13)	−0.6510 (7)	0.1876 (5)	0.087 (3)
C11*A*	0.4314 (11)	−0.5472 (6)	0.1709 (4)	0.074 (2)
Te1*B*	0.57840 (5)	0.64404 (3)	0.45639 (2)	0.05111 (11)
I1*B*	0.28098 (6)	0.68444 (4)	0.40896 (3)	0.0754 (2)
I2*B*	0.64634 (9)	0.67357 (5)	0.58167 (2)	0.0821 (2)
S1*B*	0.6218 (2)	0.5893 (2)	0.35799 (8)	0.0638 (5)
S2*B*	0.8263 (3)	0.5849 (2)	0.46319 (8)	0.0654 (5)
N1*B*	0.8203 (7)	0.4686 (5)	0.3648 (2)	0.0548 (13)
C1*B*	0.7658 (8)	0.5394 (5)	0.3910 (3)	0.0521 (15)
C2*B*	0.9376 (8)	0.4215 (6)	0.3932 (3)	0.057 (2)
C3*B*	0.8609 (10)	0.3174 (6)	0.4138 (4)	0.072 (2)
C4*B*	0.7658 (11)	0.4249 (8)	0.3044 (3)	0.077 (2)
C5*B*	0.8673 (19)	0.4805 (12)	0.2704 (5)	0.137 (5)
C6*B*	0.6907 (8)	0.8186 (5)	0.4622 (3)	0.0514 (15)
C7*B*	0.6465 (10)	0.8945 (6)	0.4969 (3)	0.064 (2)
C8*B*	0.7100 (12)	1.0074 (6)	0.4998 (4)	0.075 (2)
C9*B*	0.8148 (12)	1.0450 (7)	0.4683 (4)	0.080 (2)
C10*B*	0.8574 (12)	0.9698 (8)	0.4334 (5)	0.092 (3)
C11*B*	0.7949 (10)	0.8555 (7)	0.4301 (4)	0.075 (2)

† Occupancy = 0.5.

Table 2. *Selected geometric parameters* (Å, °) *for* (1)

Te1—C6	2.137 (6)	Te1*A*—I1*A*	2.9422 (15)
Te1—S2	2.566 (2)	Te1*A*—I2*A*	2.9524 (9)
Te1—S1	2.569 (2)	S1*A*—C1*A*	1.735 (7)
Te1—I2	2.9408 (9)	S2*A*—C1*A*	1.728 (7)
Te1—I1	2.9477 (14)	Te1*B*—C6*B*	2.144 (7)
Te1—I2*A*	4.1922 (13)	Te1*B*—S1*B*	2.550 (2)
I2—Te1*A*	4.2326 (12)	Te1*B*—S2*B*	2.552 (2)
S1—C1	1.704 (8)	Te1*B*—I1*B*	2.9697 (11)
S2—C1	1.746 (8)	Te1*B*—I2*B*	2.9864 (10)
Te1*A*—C6*A*	2.146 (6)	Te1*B*—I2*B*^{i}	3.898 (2)
Te1*A*—S1*A*	2.553 (2)	S1*B*—C1*B*	1.731 (7)
Te1*A*—S2*A*	2.561 (2)	S2*B*—C1*B*	1.736 (7)
C6—Te1—S2	91.4 (2)	S1*A*—Te1*A*—I2*A*	159.04 (5)
C6—Te1—S1	89.6 (2)	S2*A*—Te1*A*—I2*A*	88.50 (6)
S2—Te1—S1	70.48 (7)	I1*A*—Te1*A*—I2*A*	112.43 (4)
C6—Te1—I2	90.4 (2)	C6*A*—Te1*A*—I2	158.8 (2)
S2—Te1—I2	89.50 (6)	Te1*A*—I2*A*—Te1	73.31 (3)
S1—Te1—I2	159.97 (5)	C1*A*—S1*A*—Te1*A*	85.0 (2)
C6—Te1—I1	91.8 (2)	C1*A*—S2*A*—Te1*A*	84.9 (2)
S2—Te1—I1	162.31 (5)	C6*B*—Te1*B*—S1*B*	94.4 (2)
S1—Te1—I1	92.16 (6)	C6*B*—Te1*B*—S2*B*	95.7 (2)
I2—Te1—I1	107.86 (4)	S1*B*—Te1*B*—S2*B*	70.33 (7)
C6—Te1—I2*A*	154.3 (2)	C6*B*—Te1*B*—I1*B*	88.1 (2)
Te1—I2—Te1*A*	72.75 (3)	S1*B*—Te1*B*—I1*B*	91.02 (5)
C1—S1—Te1	85.3 (3)	S2*B*—Te1*B*—I1*B*	161.16 (5)
C1—S2—Te1	84.5 (3)	C6*B*—Te1*B*—I2*B*	89.8 (2)
C6*A*—Te1*A*—S1*A*	93.9 (2)	S1*B*—Te1*B*—I2*B*	155.41 (5)
C6*A*—Te1*A*—S2*A*	93.6 (2)	S2*B*—Te1*B*—I2*B*	85.15 (5)
S1*A*—Te1*A*—S2*A*	70.62 (6)	I1*B*—Te1*B*—I2*B*	113.36 (4)
C6*A*—Te1*A*—I1*A*	88.6 (2)	C6*B*—Te1*B*—I2*B*^{i}	170.2 (2)
S1*A*—Te1*A*—I1*A*	88.40 (5)	Te1*B*—I2*B*—Te1*B*^{i}	80.06 (4)
S2*A*—Te1*A*—I1*A*	159.01 (5)	C1*B*—S1*B*—Te1*B*	85.6 (2)
C6*A*—Te1*A*—I2*A*	89.1 (2)	C1*B*—S2*B*—Te1*B*	85.4 (2)

I2*A*—Te1—I2—Te1*A*	−0.64 (2)
S2—Te1—S1—C1	−7.1 (3)
S1—Te1—S2—C1	7.0 (3)
Te1—S1—C1—S2	11.3 (5)
Te1—S2—C1—S1	−11.3 (5)
S2—Te1—C6—C11	−126.3 (7)
S1—Te1—C6—C11	−55.8 (7)
I2—Te1—C6—C11	144.2 (7)
I1—Te1—C6—C11	36.3 (7)
S2—Te1—C6—C7	48.7 (6)
S1—Te1—C6—C7	119.2 (6)
I2—Te1—C6—C7	−40.8 (6)
I1—Te1—C6—C7	−148.7 (6)
Te1—I2—Te1*A*—I2*A*	0.90 (2)
I2—Te1*A*—I2*A*—Te1	−0.63 (2)
I2—Te1—I2*A*—Te1*A*	0.92 (2)
S2*A*—Te1*A*—S1*A*—C1*A*	9.0 (2)
S1*A*—Te1*A*—S2*A*—C1*A*	−9.0 (2)
Te1*A*—S2*A*—C1*A*—S1*A*	14.2 (3)
Te1*A*—S1*A*—C1*A*—S2*A*	−14.2 (3)
S1*A*—Te1*A*—C6*A*—C11*A*	150.9 (6)
S2*A*—Te1*A*—C6*A*—C11*A*	−138.4 (6)
I1*A*—Te1*A*—C6*A*—C11*A*	62.5 (6)
I2*A*—Te1*A*—C6*A*—C11*A*	−49.9 (6)
S1*A*—Te1*A*—C6*A*—C7*A*	−28.5 (5)
S2*A*—Te1*A*—C6*A*—C7*A*	42.3 (5)
I1*A*—Te1*A*—C6*A*—C7*A*	−116.8 (5)
I2*A*—Te1*A*—C6*A*—C7*A*	130.7 (5)
S2*B*—Te1*B*—S1*B*—C1*B*	10.1 (2)
S1*B*—Te1*B*—S2*B*—C1*B*	−10.1 (2)
C4*B*—N1*B*—C1*B*—S1*B*	0.6 (11)
C2*B*—N1*B*—C1*B*—S2*B*	−0.5 (10)
Te1*B*—S1*B*—C1*B*—S2*B*	−15.7 (4)
Te1*B*—S2*B*—C1*B*—S1*B*	15.7 (4)
S1*B*—Te1*B*—C6*B*—C11*B*	−22.1 (7)
S2*B*—Te1*B*—C6*B*—C11*B*	48.6 (7)
I1*B*—Te1*B*—C6*B*—C11*B*	−112.9 (7)
I2*B*—Te1*B*—C6*B*—C11*B*	133.7 (7)
S1*B*—Te1*B*—C6*B*—C7*B*	153.4 (6)
S2*B*—Te1*B*—C6*B*—C7*B*	−136.0 (6)
I1*B*—Te1*B*—C6*B*—C7*B*	62.5 (6)
I2*B*—Te1*B*—C6*B*—C7*B*	−50.9 (6)

Symmetry code: (i) $1 - x, 1 - y, 1 - z$.

Compound (2)

Crystal data

[$TeBr_{0.8}I_{1.2}(C_5H_{10}NS_2)$-($C_7H_7O$)]
$M_r = 599.19$
Triclinic
$P\overline{1}$
$a = 9.9482\ (12)$ Å
$b = 10.428\ (3)$ Å
$c = 11.004\ (2)$ Å
$\alpha = 66.95\ (2)°$
$\beta = 65.934\ (11)°$
$\gamma = 75.94\ (2)°$
$V = 954.4\ (4)$ Å^3
$Z = 2$
$D_x = 2.085$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 24 reflections
$\theta = 12.6$–$14.8°$
$\mu = 5.386$ mm^{-1}
$T = 293\ (2)$ K
Flat prism
$1.04 \times 0.34 \times 0.14$ mm
Red

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: numerical (Gaussian method)
$T_{\min} = 0.151$, $T_{\max} = 0.495$
5544 measured reflections
5544 independent reflections

4485 observed reflections [$I > 2\sigma(I)$]
$\theta_{\max} = 29.96°$
$h = 0 \rightarrow 13$
$k = -13 \rightarrow 14$
$l = -13 \rightarrow 15$
3 standard reflections
frequency: 120 min
intensity decay: 4.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0427$
$wR(F^2) = 0.1245$
$S = 1.108$
5521 reflections

$(\Delta/\sigma)_{\max} = 0.410$
$\Delta\rho_{\max} = 1.722$ e Å^{-3} (adjacent to Te and I/Br)
$\Delta\rho_{\min} = -1.903$ e Å^{-3} (adjacent to Te and I/Br)

199 parameters
H atoms: riding and rotating model with $U_{\rm iso}$ refined for groups
$w = 1/[\sigma^2(F_o^2) + (0.0791P)^2 + 1.5737P]$ where $P = (F_o^2 + 2F_c^2)/3$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Te1	0.04660 (4)	0.03535 (3)	0.76279 (3)	0.04207 (10)
I1	0.2362 (3)	0.1281 (3)	0.8664 (3)	0.0528 (5)
Br1	0.2023 (7)	0.1392 (7)	0.8806 (7)	0.0484 (10)
I2	−0.2112 (4)	0.2143 (3)	0.6845 (4)	0.0488 (3)
Br2	−0.2032 (10)	0.2339 (7)	0.6974 (9)	0.0464 (8)
S1	0.2269 (2)	−0.18068 (12)	0.7989 (2)	0.0497 (3)
S2	−0.02116 (15)	−0.14714 (13)	0.70432 (15)	0.0486 (3)
O1	0.4367 (5)	0.3439 (5)	0.1442 (4)	0.0676 (11)
N1	0.1525 (5)	−0.3857 (4)	0.7581 (4)	0.0426 (8)
C1	0.1226 (5)	−0.2552 (5)	0.7561 (5)	0.0399 (8)
C2	0.0623 (6)	−0.4507 (5)	0.7243 (6)	0.0492 (11)
C3	−0.0651 (7)	−0.5157 (7)	0.8526 (7)	0.0620 (14)
C4	0.2794 (6)	−0.4749 (5)	0.7929 (6)	0.0524 (11)
C5	0.4078 (8)	−0.4848 (10)	0.6640 (9)	0.083 (2)
C6	0.1879 (6)	0.1311 (5)	0.5539 (5)	0.0435 (9)
C7	0.1887 (6)	0.2761 (5)	0.5045 (5)	0.0469 (10)
C8	0.2744 (6)	0.3428 (5)	0.3689 (6)	0.0527 (11)
C9	0.3605 (6)	0.2656 (6)	0.2786 (6)	0.0506 (11)
C10	0.3622 (6)	0.1227 (6)	0.3279 (6)	0.0516 (11)
C11	0.2754 (6)	0.0544 (5)	0.4655 (6)	0.0487 (10)
C12	0.4960 (8)	0.2801 (9)	0.0375 (7)	0.077 (2)

Table 4. *Selected geometric parameters* (Å, °) *for* (2)

Te1—C6	2.123 (5)	Te1—I1	3.049 (3)
Te1—S1	2.5233 (15)	Te1—Br1$^{\rm i}$	3.676 (6)
Te1—S2	2.5349 (14)	Te1—I1$^{\rm i}$	3.872 (3)
Te1—Br1	2.967 (8)	S1—C1	1.726 (4)
Te1—Br2	2.962 (8)	S2—C1	1.737 (5)
Te1—I2	3.003 (4)		
C6—Te1—S1	92.63 (14)	S2—Te1—I2	84.78 (5)
C6—Te1—S2	94.87 (13)	C6—Te1—I1	87.84 (14)
S1—Te1—S2	71.27 (4)	S1—Te1—I1	80.04 (7)
C6—Te1—Br1	91.4 (2)	S2—Te1—I1	151.26 (7)
S1—Te1—Br1	84.62 (13)	I2—Te1—I1	123.92 (7)
S2—Te1—Br1	155.30 (13)	C6—Te1—Br1$^{\rm i}$	177.7 (2)
C6—Te1—Br2	89.0 (2)	C6—Te1—I1$^{\rm i}$	175.64 (14)
S1—Te1—Br2	162.24 (9)	Te1—I1—Te1$^{\rm i}$	84.52 (7)
S2—Te1—Br2	90.98 (9)	Te1—Br1—Te1$^{\rm i}$	89.2 (2)
Br1—Te1—Br2	113.03 (15)	C1—S1—Te1	86.2 (2)
C6—Te1—I2	88.7 (2)	C1—S2—Te1	85.6 (2)
S1—Te1—I2	156.04 (5)	S1—C1—S2	116.6 (3)
S2—Te1—S1—C1	−3.0 (2)	Br2—Te1—C6—C11	−126.5 (4)
S1—Te1—S2—C1	3.0 (2)	I2—Te1—C6—C11	−120.2 (4)
C4—N1—C1—S1	−1.9 (7)	I1—Te1—C6—C11	115.8 (4)
C2—N1—C1—S2	−3.3 (7)	S1—Te1—C6—C7	−145.8 (4)
Te1—S1—C1—S2	4.6 (3)	S2—Te1—C6—C7	142.8 (4)
Te1—S2—C1—S1	−4.6 (3)	Br1—Te1—C6—C7	−61.1 (4)
S1—Te1—C6—C11	35.9 (4)	Br2—Te1—C6—C7	51.9 (4)
S2—Te1—C6—C11	−35.6 (4)	I2—Te1—C6—C7	58.2 (4)
Br1—Te1—C6—C11	120.5 (4)	I1—Te1—C6—C7	−65.8 (4)

Symmetry code: (i) $-x, -y, 2 - z$.

Both ethyl groups in one of the three independent molecules of (1) were disordered. For one of the ethyl groups the disorder was successfully resolved [C(4)/C(4′) and C(5)/C(5′) were refined isotropically with occupancy factors of 0.5 for each position], but for the other, C(2), C(3), it failed (anisotropic refinement gave very large amplitudes of the thermal motion of the atoms). An absorption correction for (2) was made assuming that the crystal contains an equimolar ratio of Br and I atoms. The positions of the non-stoichiometrically replaced Br atoms were located in a difference Fourier synthesis. The I/Br occupancy factors were refined at an early stage (isotropic for all non-H atoms) by a least-squares procedure using the restrictions $U_{\rm iso}({\rm Br}_j) = U_{\rm iso}({\rm I}_j)$, $g({\rm Br}_j) + g({\rm I}_j) = 1$ and $g({\rm Br1}) = g({\rm Br2})$, and then fixed.

For both compounds, program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1060). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-*p*-Tolylvanillaldimine, $C_{15}H_{15}NO_2$

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Abstract

The title compound, 2-methoxy-4-(*p*-tolyliminomethyl)-phenol, contains two phenyl rings bridged by a C═N imino moiety, the planes of which are inclined at an

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