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# THE CRYSTAL AND MOLECULAR STRUCTURE OF ACETATO(2-PHENYLAZOPHENYL-*C*,*N*′)TELLURIUM(II) AND (2-PHENYLAZOPHENYL-*C*,*N*′)THIOCYANATOTELLURIUM(II)

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## Summary

The crystal and molecular structures of acetato(2-phenylazophenyl-*C*,*N*′)tellurium(II) (I) and (2-phenylazophenyl-*C*,*N*′)thiocyanatotellurium(II) (II) have been determined. The structure of the former consists of discrete molecules in which weak intramolecular Te · · · O [2.953(4) Å] interactions occur. However, the structure of the latter compound shows a very weak intermolecular Te · · · N [3.535(3) Å] interaction, which links the molecules into dimers. The coordination about tellurium can be considered as approximately trigonal bipyramidal with the carbon atom and the two lone pairs in the equatorial positions and either N and O (I) or N and S (II) in the axial positions.

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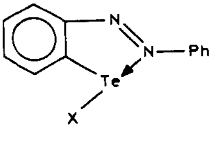
## Introduction

The structures of two (phenylazophenyl-*C*,*N*′)tellurium(II) compounds (III) have been determined. For X = Cl [1] there are weak intermolecular Te · · · Cl contacts *trans* to the Te–C bond, but when X = dimethyldithiocarbamate [2] the material is monomeric with a long intramolecular Te · · · S (3.225 Å) contact giving a distorted square planar arrangement about the central tellurium atom. In this paper we extend



(III)

the series to X = thiocyanate and acetate. Thiocyanate is ambidentate and whilst a thiocyanate-linkage to soft tellurium(II) is expected, intermolecular Te · · · N contacts are possible. It was also of interest to determine the extent to which the harder acetato-ligand would mimic the unsymmetrically bidentate dimethyldithiocarbamate.

## Experimental

### *Synthesis*

*Acetato(2-phenylazophenyl-C,N')tellurium(II)*

A solution of sodium acetate (0.082 g, 0.001 mol) in dry methanol (20 cm$^3$) was added, with stirring and under dinitrogen, to a solution of (2-phenylazophenyl-*C,N'*)tellurium(II) chloride [1] (0.344 g, 0.001 mol) in dichloromethane (25 cm$^3$) at room temperature. On completing the addition, the mixture was stirred for a further 20 min and the solvents were removed on a rotary evaporator. Dichloromethane (100 cm$^3$) was added to the residue with vigorous stirring. The solution was filtered to remove insoluble sodium chloride and the solvent was removed on a rotary evaporator. Methanol (5 cm$^3$) was added to the residue, which was stirred well and filtered off. The crude product was recrystallized from petroleum ether (60–80), under conditions of slow evaporation, golden-orange crystals being obtained which melted with decomposition at 121–122°C. (Yield 75%). Found: C, 45.8; H, 3.15; N, 7.65. $C_{14}H_{12}N_2O_2Te$ calcd.: C, 45.7; H, 3.26; N, 7.62%.

*(2-Phenylazophenyl-C,N')thiocyanatotellurium(II)*

A solution of ammonium thiocyanate (0.152 g, 0.002 mol) in dry methanol (40 cm$^3$) was added slowly, with stirring and under dinitrogen, to a refluxing mixture of (2-phenylazophenyl-*C,N'*)tellurium(II) chloride [1] (0.688 g, 0.002 mol) and dichloromethane (50 cm$^3$). On completing the addition, the mixture was heated for a further 5 min and cooled to room temperature, with continuous stirring. The solvents were removed on a rotary evaporator and dichloromethane (200 cm$^3$) was added to the residue with vigorous stirring. The solution was filtered to remove insoluble sodium chloride and the solvent was removed on a rotary evaporator. Methanol (8 cm$^3$) was added to the product, which was stirred well and filtered off. The crude product recrystallized from methanol to give orange-red crystals, which melted with decomposition at 122°C. (Yield 60%). Found: C, 42.2; H, 2.25; N, 11.1; S, 7.80. $C_{13}H_9N_3STe$ calcd.: C, 42.6; H, 2.46; N, 11.5; S, 8.73%.

Conductivity ($\Omega$ cm$^2$ mol$^{-1}$) = 5.25, in nitromethane at room temperature (concentration $10^{-3}$ mol l$^{-1}$).

### *Physical measurements*

Infrared spectra were determined for KBr discs with a Perkin–Elmer 599 instrument.

Elemental analysis was by the Analytical Services Section, Chemistry Department, University of Aston.

### *X-ray structure analysis of I and II*

After preliminary examination by photographic methods, the final cell dimen-

TABLE 1

CRYSTAL AND EXPERIMENTAL PARAMETERS

| | I | II |
|---|---|---|
| Molecular formula | $C_{14}H_{12}N_2O_2Te$ | $C_{13}H_9N_3STe$ |
| Molecular weight | 367.9 | 366.9 |
| Crystal size (mm) | 0.55 × 0.45 × 0.05 | 0.45 × 0.125 × 0.025 |
| Cell constants (Å) $a$ | 5.420(4) | 7.357(5) |
| $b$ | 11.361(2) | 9.300(1) |
| $c$ | 12.321(3) | 10.188(1) |
| (°) $\alpha$ | 65.24(3) | 70.50(1) |
| $\beta$ | 79.57(3) | 85.65(2) |
| $\gamma$ | 86.94(3) | 83.09(2) |
| Cell volume (Å$^3$) | 677.3 | 651.8 |
| Space group | $P\bar{1}$ | $P\bar{1}$ |
| $Z$ | 2 | 2 |
| Density (g cm$^{-3}$) calcd. | 1.804 | 1.869 |
| Density (g cm$^{-3}$) measd. | 1.791 | 1.872 |
| Absorption coefficient (mm$^{-1}$) | 2.06 | 2.27 |
| Data collection range ($\lambda$ 0.71069 Å) $2\theta$(°) | 4–50 | 4–55 |
| Scan range ($\omega$°) | $1.1 + 0.35 \tan\theta$ | $1.2 + 0.35 \tan\theta$ |
| Scan speed range (° min$^{-1}$) | 3.3–0.9 | 3.3–1.2 |
| Total data measured | 2712 | 3134 |
| Significant data [$I > 2.5\sigma(I)$] | 2152 | 2457 |
| Least-squares weights $w = [\sigma^2(F) + KF^2]^{-1}$ | $K = 0.0015$ | $K = 0.0010$ |
| Final $R$ (%) | 4.05 | 3.73 |
| Final $R_w$ (%) | 5.06 | 4.72 |
| Residual electron density in final difference map ($\epsilon$ Å$^{-3}$) | −1.14 to +1.52 | −0.85 to +1.58 |

TABLE 2

FRACTIONAL ATOMIC COORDINATES FOR $(C_{12}H_9N_2)Te(O_2C.Me)$ (I)

| Atom | $x$ | $y$ | $z$ |
|---|---|---|---|
| Te(1) | 0.03646(5) | 0.24362(2) | 0.26102(2) |
| O(1) | −0.1815(7) | 0.2181(3) | 0.1410(4) |
| O(2) | 0.0519(8) | 0.0492(4) | 0.1592(4) |
| N(1) | 0.1287(8) | 0.4547(4) | 0.3430(4) |
| N(2) | 0.1837(7) | 0.3374(3) | 0.3670(3) |
| C(1) | −0.0242(9) | 0.5051(4) | 0.2560(4) |
| C(2) | −0.1055(9) | 0.4289(4) | 0.2036(4) |
| C(3) | −0.2682(11) | 0.4817(5) | 0.1203(5) |
| C(4) | −0.3491(12) | 0.6062(5) | 0.0920(5) |
| C(5) | −0.2700(13) | 0.6807(5) | 0.1458(5) |
| C(6) | −0.1088(11) | 0.6309(4) | 0.2264(5) |
| C(7) | 0.3382(8) | 0.2785(4) | 0.4563(4) |
| C(8) | 0.3278(10) | 0.1443(4) | 0.5196(4) |
| C(9) | 0.4780(12) | 0.0855(5) | 0.6065(5) |
| C(10) | 0.6390(11) | 0.1604(6) | 0.6303(5) |
| C(11) | 0.6490(10) | 0.2937(5) | 0.5659(5) |
| C(12) | 0.4986(9) | 0.3526(4) | 0.4798(4) |
| C(13) | −0.1209(9) | 0.1165(4) | 0.1225(4) |
| C(14) | −0.2850(11) | 0.0880(5) | 0.0493(6) |

TABLE 3

FRACTIONAL ATOMIC COORDINATES FOR $(C_{12}H_9N_2)Te(SCN)$ (II)

| Atom | $x$ | $y$ | $z$ |
|---|---|---|---|
| Te | −0.21681(4) | −0.23761(3) | −0.03151(3) |
| S | −0.1508(3) | 0.0560(2) | −0.1409(2) |
| N(1) | −0.2430(5) | −0.5329(4) | −0.0932(4) |
| N(2) | −0.2555(5) | −0.4848(4) | 0.0105(4) |
| N(3) | −0.4975(10) | 0.1805(6) | −0.2455(6) |
| C(1) | −0.2020(6) | −0.4213(5) | −0.2170(5) |
| C(2) | −0.1800(6) | −0.2721(5) | −0.2230(4) |
| C(3) | −0.1406(7) | −0.1648(5) | −0.3503(5) |
| C(4) | −0.1270(7) | −0.2063(6) | −0.4689(5) |
| C(5) | −0.1505(8) | −0.3544(6) | −0.4648(5) |
| C(6) | −0.1873(8) | −0.4621(5) | −0.3384(5) |
| C(7) | −0.2948(6) | −0.5959(5) | 0.1433(4) |
| C(8) | −0.3090(6) | −0.7479(5) | 0.1582(5) |
| C(9) | −0.3482(8) | −0.8481(6) | 0.2879(6) |
| C(10) | −0.3733(8) | −0.7990(6) | 0.4023(5) |
| C(11) | −0.3575(8) | −0.6490(6) | 0.3873(5) |
| C(12) | −0.3184(7) | −0.5439(6) | 0.2586(5) |
| C(13) | −0.3547(10) | 0.1288(6) | −0.2015(6) |

sions and reflection intensities were measured with graphite-monochromated Mo-$K_\alpha$ radiation on an Enraf–Nonius CAD-4 diffractometer operating in the $\omega$-$2\theta$ scan mode. Two standard reflections were measured every hour to check the stability of the system. Absorption corrections were not applied.

The structures were solved by Patterson and Fourier methods. The refinements were by least-squares, using anisotropic temperature factors for the non-hydrogen atoms. Hydrogen atoms were located from difference Fourier maps and included in the calculations but their parameters were not refined. Refinement was terminated when all calculated shift/error ratios were $< 0.1$. Details of crystal and experimental parameters are in Table 1. Final atomic coordinates for I and II are in Tables 2 and 3. Lists of structure factors, thermal parameters and hydrogen atom coordinates are available from the authors. Computation were carried out on the Honeywell computer at the University of Birmingham with SHELX [3]. Pictures were drawn with PLUTO [4].

## Discussion

The reaction of (2-phenylazophenyl-*C*,*N*′)tellurium(II) chloride with sodium acetate or sodium thiocyanate gave acetato(2-phenylazophenyl-*C*,*N*′)-tellurium(II) or (2-phenylazophenyl-*C*,*N*′)thiocyanatotellurium(II) respectively. Both compounds were found to be stable in air for several months and have satisfactory elemental analysis and IR data (Table 4).

### *Structural data*

Pertinent distances, angles and torsion angles are given in Tables 5 (compound I) and 6 (compound II). The tellurium coordination geometries in both compounds are illustrated in Figs. 1 and 2. The packing arrangements are shown in Figs. 3 and 4. In

TABLE 4

IR DATA (cm$^{-1}$) FOR ($C_{12}H_9N_2$)Te($O_2$C.Me) (I) AND ($C_{12}H_9N_2$)Te(SCN) (II)

| Compound | $\nu_{as}$(COO) | $\nu_s$(COO) | $\nu$(TeO) | $\nu$(CN) |
|---|---|---|---|---|
| I | 1640 | 1298 | 250 | – |
| II | – | – | – | 2120 |

both compounds, the coordination about tellurium is approximately trigonal bipyramidal with the carbon atom and the two lone pairs in the equatorial positions and either N(2) and O(1) (compound I) or N(2) and S(1) (compound II) in the axial positions. Thus in both structures the position *trans* to the bonded carbon atom is unoccupied. Both compounds are almost T-shaped. In compound I the tellurium atom is approached by a fourth atom, O(2) of the acetate group at a distance of 2.953(4) Å which illustrates the tendency of Te$^{II}$ to achieve square planar geometry as has been reported for many Te$^{II}$ complexes [5]. However, in the present structure, distortions from square planar geometry are very considerable (Table 5). In compound II tellurium is approached by N(3) of a neighbouring molecule at a distance

TABLE 5

BOND DISTANCES (Å), ANGLES (°) AND SELECTED TORSION ANGLES (°) IN I (E.s.d.'s for torsion angles are ca. 0.6°)

| | | | |
|---|---|---|---|
| Te–O(1) | 2.167(4) | N(1)–N(2) | 1.270(5) |
| Te···O(2) | 2.953(4) | N(2)–C(7) | 1.420(6) |
| Te–N(2) | 2.260(4) | C(7)–C(8) | 1.391(6) |
| Te–C(2) | 2.074(4) | C(8)–C(9) | 1.383(8) |
| C(1)–C(2) | 1.404(6) | C(9)–C(10) | 1.397(9) |
| C(2)–C(3) | 1.396(8) | C(10)–C(11) | 1.383(8) |
| C(3)–C(4) | 1.375(7) | C(11)–C(12) | 1.378(8) |
| C(4)–C(5) | 1.397(8) | C(12)–C(7) | 1.386(6) |
| C(5)–C(6) | 1.363(9) | O(1)–C(13) | 1.283(6) |
| C(6)–C(1) | 1.393(7) | O(2)–C(13) | 1.205(6) |
| C(1)–N(1) | 1.387(7) | C(13)–C(14) | 1.509(7) |
| N(2)–Te–O(1) | 159.0(1) | C(7)–N(2)–Te | 126.2(3) |
| C(2)–Te–O(1) | 85.3(2) | N(2)–C(7)–C(8) | 118.8(4) |
| C(2)–Te–N(2) | 73.9(2) | N(2)–C(7)–C(12) | 121.0(4) |
| C(2)–C(1)–C(6) | 120.2(5) | C(8)–C(7)–C(12) | 120.2(4) |
| C(1)–C(2)–C(3) | 119.2(4) | C(7)–C(8)–C(9) | 119.4(5) |
| C(2)–C(3)–C(4) | 119.8(5) | C(8)–C(9)–C(10) | 120.3(5) |
| C(3)–C(4)–C(5) | 120.7(5) | C(9)–C(10)–C(11) | 119.7(5) |
| C(4)–C(5)–C(6) | 120.2(5) | C(10)–C(11)–C(12) | 120.2(5) |
| C(1)–N(1)–N(2) | 113.1(4) | Te–O(1)–C(13) | 112.3(3) |
| N(1)–C(1)–C(6) | 118.6(4) | Te–C(2)–C(3) | 126.4(3) |
| N(1)–C(1)–C(2) | 121.0(4) | Te–C(2)–C(1) | 114.5(3) |
| N(1)–N(2)–Te | 117.4(3) | O(1)–C(13)–O(2) | 123.7(5) |
| N(1)–N(2)–C(7) | 116.4(4) | O(1)–C(13)–C(14) | 114.0(4) |
| N(2)–Te···O(2) | 152.9(1) | O(2)–C(13)–C(14) | 122.3(4) |
| O(2)···Te–O(1) | 47.8(1) | | |
| C(7)–N(2)–N(1)–C(1) | −179.5 | N(2)–N(1)–C(1)–C(2) | 0.5 |
| N(1)–N(2)–C(7)–C(8) | 155.7 | N(2)–N(1)–C(1)–C(6) | 176.6 |
| N(1)–N(2)–C(7)–C(12) | −24.9 | | |

TABLE 6

BOND DISTANCES (Å), ANGLES (°) AND SELECTED TORSION ANGLES (°) IN II (E.s.d.'s for torsion angles are ca. 0.5°)

| | | | |
|---|---|---|---|
| Te–S | 2.672(1) | N(2)–N(1) | 1.270(5) |
| Te–N(2) | 2.243(3) | N(2)–C(7) | 1.436(6) |
| Te–C(2) | 2.073(4) | C(7)–C(8) | 1.386(6) |
| C(1)–C(2) | 1.397(6) | C(8)–C(9) | 1.372(7) |
| C(2)–C(3) | 1.382(6) | C(9)–C(10) | 1.380(8) |
| C(3)–C(4) | 1.379(7) | C(10)–C(11) | 1.370(8) |
| C(4)–C(5) | 1.395(7) | C(11)–C(12) | 1.383(7) |
| C(5)–C(6) | 1.374(7) | C(12)–C(7) | 1.405(7) |
| C(6)–C(1) | 1.403(6) | S–C(13) | 1.646(7) |
| C(1)–N(1) | 1.379(6) | C(13)–N(3) | 1.158(9) |
| S–Te–N(2) | 166.8(1) | C(2)–C(1)–N(1) | 121.7(4) |
| S–Te–C(2) | 92.6(1) | C(1)–N(1)–N(2) | 112.9(4) |
| N(2)–Te–C(2) | 74.2(2) | N(1)–N(2)–C(7) | 115.9(3) |
| Te–S–C(13) | 98.4(2) | N(1)–N(2)–Te | 117.4(3) |
| S–C(13)–N(3) | 179.3(6) | Te–N(2)–C(7) | 126.7(3) |
| Te–C(2)–C(3) | 127.1(3) | N(2)–C(7)–C(12) | 117.0(4) |
| Te–C(2)–C(1) | 113.7(3) | N(2)–C(7)–C(8) | 121.9(4) |
| C(1)–C(2)–C(3) | 119.1(4) | C(8)–C(7)–C(12) | 121.1(4) |
| C(2)–C(3)–C(4) | 119.5(4) | C(7)–C(8)–C(9) | 119.1(5) |
| C(3)–C(4)–C(5) | 122.1(4) | C(8)–C(9)–C(10) | 120.7(5) |
| C(4)–C(5)–C(6) | 118.7(5) | C(9)–C(10)–C(11) | 120.0(5) |
| C(5)–C(6)–C(1) | 119.8(4) | C(10)–C(11)–C(12) | 121.3(5) |
| C(6)–C(1)–C(2) | 120.8(4) | C(11)–C(12)–C(7) | 117.8(5) |
| C(6)–C(1)–N(1) | 117.4(4) | | |
| C(1)–N(1)–N(2)–C(7) | −179.0 | N(2)–N(1)–C(1)–C(6) | −179.2 |
| N(1)–N(2)–C(7)–C(12) | 3.1 | N(2)–N(1)–C(1)–C(2) | −0.7 |
| N(1)–N(2)–C(7)–C(8) | −176.9 | | |

of 3.535(3) Å, indicating a possible very weak interaction. This nitrogen atom is situated 1.660(5) Å from the tellurium coordination plane (Fig. 4).

In I the organic ligand is not planar. There is a twist of ca. 25° of the free phenyl ring relative to the other phenyl ring and the nitrogen atoms, which are coplanar to within ±0.025 Å. The tellurium atom lies in this plane and O(1) and O(2) deviate by 0.039 and −0.55 Å respectively. C(13) and C(14) of the acetate group are displaced

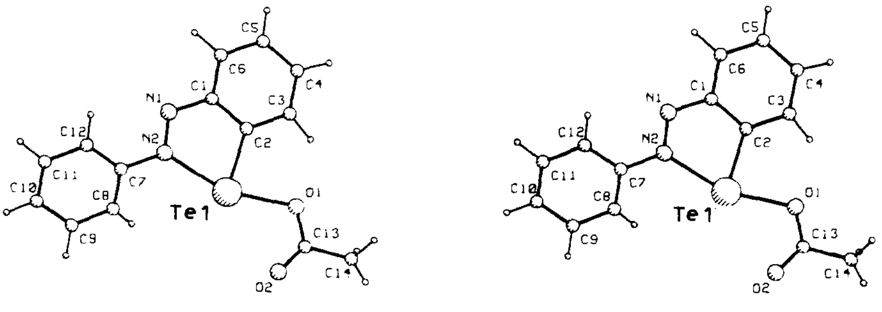


Fig. 1. Stereoscopic view of molecule I showing the atom numbering.

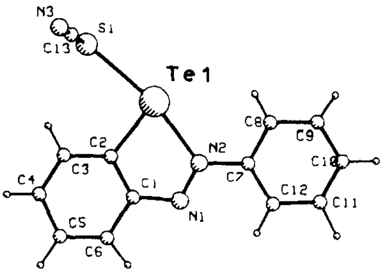


Fig. 2. View of molecule II showing the atom numbering.

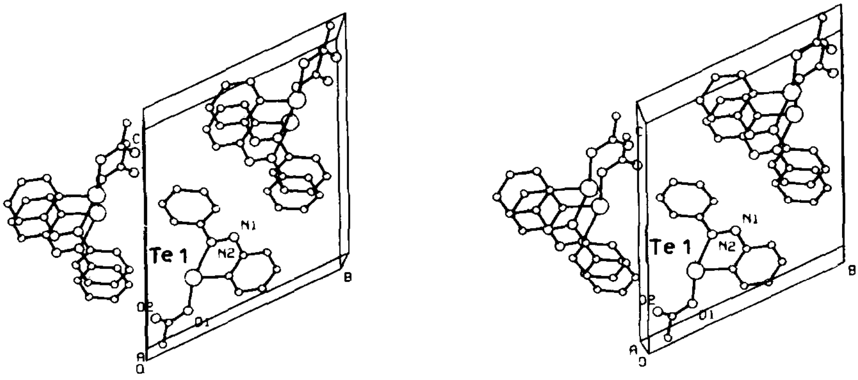


Fig. 3. Stereoscopic view of the packing arrangement in the crystal structure of I.

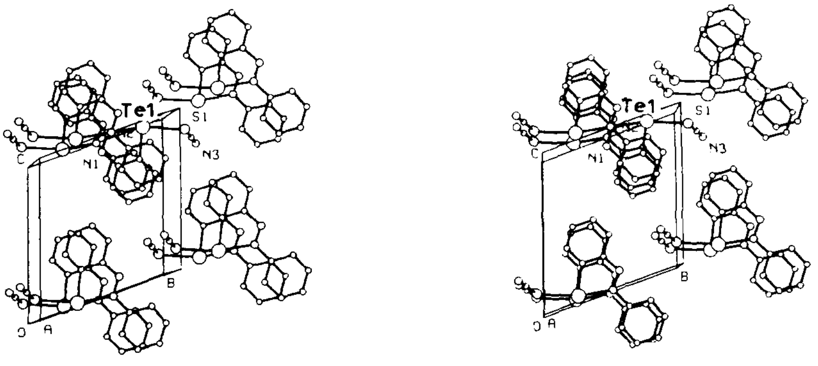


Fig. 4. Stereoscopic view of the packing arrangement in the crystal structure of II.

by −0.22 and −0.07 Å from the plane. The acetate group itself is accurately coplanar. A similar twist of the free phenyl ring occurs in the crystal structure of dimethyldithiocarbamato(2-phenylazophenyl-*C*, *N*′)tellurium(II) [2].

In compound II the situation is different. The organic ligand is planar to within ±0.04 Å with the tellurium atom close to the plane (deviation from mean plane 0.02 Å). The sulphur atom is displaced by 0.10 Å. The thiocyanato group is nearly linear (N–C–S angle 179.3(6)°) and is oriented approximately perpendicular to the Te-organic ligand plane, angle Te–S–C, 98.4(2)° (Fig. 2). Similar geometries have been reported for other thiocyanate complexes of $Te^{II}$ and $Te^{IV}$ e.g. $Te(etu)_2(SCN)_2$ [6], (where etu = ethylenethiourea), $[(CH_3)_4N][C_6H_5Te(SCN)_2]$ [7] and $[Te(HOCH_2$-$CH_2(CH_3)NCS_2)_3]$ [8].

The bonding between tellurium and the (2-phenylazophenyl) group is quite similar in the two compounds. The Te–N bonds are 2.260(4) Å in I and 2.243(3) Å in II and the Te–C bonds 2.074(4) and 2.073(4) Å. These values may be compared with the results obtained in the crystal structures of (2-phenylazophenyl-*C*,*N*′)tellurium(II) chloride [1] and dimethyldithiocarbamate [2] [Te–C 2.04(2), 2.10(1) and Te–N 2.23(2), 2.340(7) Å]. They also agree with the sum of the covalent radii for Te (axial direction) [9] and nitrogen (2.24 Å) and Te (equatorial direction) [9] and $sp^2$-hybridised carbon (2.08 Å), considering the Te coordination to be essentially trigonal bipyramidal as previously postulated. The N–Te–C angles are also similar in the four compounds.

Te–O distances (compound I) 2.167(4) and 2.953(4) Å are in good agreement with those found in bis(*p*-methoxyphenyl)tellurium(IV) diacetate [mean 2.161 and 2.988 Å] [10]. The Te–O(1) bond length, 2.167(4) Å, is in good agreement with the sum of covalent radii if the axial radius of tellurium is taken to be 1.54 Å [9] giving an estimated length of 2.20 Å for Te–O. However, the length is approximately 0.1–0.2 Å longer than many other previously reported values [11–13], but differences in geometry make comparisons difficult.

The Te · · · O(2) length (2.953 Å) is longer than the sum of covalent radii [9,14], but shorter than the sum of the Van der Waals radii 3.6 Å according to Pauling [14] or 3.46 Å according to Bondi [15], indicating a weak intramolecular interaction. Similar interactions have also been reported in e.g. 2,6-diacetylpyridine (C,N,O)tellurium(IV)trichloride (2.878 Å) [16] and in two modifications of $TeO_2$ (2.89 and 2.73 Å) [17].

Weak interactions clustered around the tellurium lone pairs of electrons in the equatorial plane, roughly *trans* to the carbon atom of an organic ligand, appear to be common in organotellurium(II) derivatives [5,9].

The Te–S(1) distance (compound II) of 2.672(1) Å is slightly larger than the sum of the covalent radii (2.58 Å) [9,14] but agrees well with values found for other $Te^{II}$-thiocyanate complexes [6,7], range 2.665–2.702 Å. The next closest interaction involves a nitrogen atom from a neighbouring molecule ($-x$, $-y$, $-z$), Te...N(3) 3.535(3) Å, which is close to the Van der Waals distance (3.7 Å) [14] or (3.56 Å) [15]. If this weak interaction is significant, it would link the molecules into dimers. Bond length in the phenylazophenyl groups are generally in good agreement with those found for free azobenzene [18]. However, in the moiety involved in bonding to tellurium, significant differences occur. Thus the N=N double bond, 1.270(5) Å in both I and II is slightly, but significantly, longer than the value (1.243(3) Å) in free azobenzene, or than the sum of the covalent radii (1.24 Å) [14]. The C–N bond to the complexed phenyl ring is 1.387(7) in I and 1.379(6) Å in II compared with 1.420(6) (I) and 1.436(6) Å (II) to the free phenyl ring and 1.433 Å in free azobenzene. This significant shortening of the C–N bond and lengthening of the N=N bond in the complexed moieties may indicate electron delocalisation in the five-membered heterocyclic ring. Such an effect has been predicted on the basis of Mössbauer data for (2-phenylazophenyl-*C*,*N*′)tellurium(II) chloride [9].

C–S [1.646(7) Å] and C≡N [1.158(9) Å] in the thiocyanate group (compound II) are in good agreement with those found for $Te^{II}$ thiocyanate complexes [6,7], range

1.594–1.643 for C–S and 1.128–1.179 Å for C≡N. The C–S distance is considerably shorter than the value (1.74 Å) [14] calculated from the single bond radii of sulphur and *sp* hybridized carbon.

The dimensions of the acetate group (compound I) are in good agreement with those found in bis(*p*-methoxyphenyl)tellurium(IV) diacetate [10], mean values, C–C 1.503, C=O 1.216 and C–O 1.307 Å.

*Infrared data*

The separation of $\nu_{as}$ and $\nu_s$ (COO) for the acetato ligand is 342 $cm^{-1}$. This separation, and the high frequency of $\nu_{as}$(COO) at 1640 $cm^{-1}$ suggest significant "ester" character for the acetato group, a situation which has also been observed for $R_2Te(OOC \cdot CH_3)_2$ (R = Ph, *p*-$EtOC_6H_4$) [19] where unidentate or very unsymmetrically bidentate acetate groups were suggested. The X-ray data are consistent with a similar interpretation of the data for I since the Te–O bonds are very different (2.167, 2.953 Å), thus the IR data indicate that the longer contact of 2.953 Å must be extremely weak. A $\nu$(TeO) band was assigned for $Ph_2Te(OOC \cdot CH_3)_2$ at 280 $cm^{-1}$ [19], a lower frequency might be expected for the $Te^{II}$ compound and comparison of the lower frequency region of I with that of $(C_{12}H_9N_2)TeCl$ [1] suggested that $\nu$(TeO) could be assigned at 250 $cm^{-1}$.

For II, the strong sharp band for $\nu$(CN) at 2120 $cm^{-1}$ is entirely in accord with expectation for S-bonded thiocyanate [20].

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## References

1 R.E. Cobbledick, F.W.B. Einstein, W.R. McWhinnie and F.H. Musa, J. Chem. Res., (S) (1979) 145; J. Chem. Res., (M) (1979) 1901.
2 M.A.K. Ahmed, A.E. McCarthy, W.R. McWhinnie and F.J. Berry, J. Chem. Soc., Dalton Trans., in press.
3 G.M. Sheldrick, SHELX, Program for Crystal Structure Determination, University of Cambridge, (1978).
4 W.D.S. Motherwell, PLUTO 78, "Program for plotting molecular and crystals structures", Report 'Cambridge Crystallographic Files', University of Manchester Regional Computer Centre (Manchester, 1981).
5 S. Huseby, Proceedings of the 4th International Conference on the Organic Chemistry of Se and Te, F.J. Berry and W.R. McWhinnie (Eds.), University of Aston in Birmingham, 1983, p. 298.
6 K. Åse, K. Bøjum, O. Foss and K. Marøy, Acta Chem. Scand., 25 (1971) 2457.
7 S. Hauge and O. Vikane, Acta Chem. Scand., A29 (1975) 755.
8 S. Huseby and A. Thowsen, Acta Chem. Scand., A35 (1981) 386.
9 W.R. McWhinnie and Z. Monsef-Mirzai, "Proceedings of the 4th International Conference on the Organic Chemistry of Se and Te", Eds. F.J. Berry and W.R. McWhinnie, University of Aston in Birmingham, (1983) 3.
10 N.W. Alcock, W.D. Harrison and C. Howes, J. Chem. Soc., Dalton Trans., (1984) 1709.
11 C.S. Mancinelli, D.D. Titus and R.F. Ziolo, J. Organomet. Chem., 140 (1977) 113.
12 R.O. Day and R.R. Holmes, Inorg. Chem., 20 (1981) 3071.
13 N.W. Alcock and W.D. Harrison, J. Chem. Soc., Dalton Trans., (1982) 1421.

14 L. Pauling, The Nature of the Chemical Bond, 3rd Ed., Cornell University Press, Ithaca, New York, (1960).
15 A. Bondi, J. Phys. Chem., 68 (1964) 441.
16 H.J. Gysling, H.R. Luss and S.A. Gardner, J. Organomet. Chem., 184 (1980) 417.
17 N.W. Alcock, Advan. Inorg. Chem. Radiochem., 15 (1972) 1.
18 C.J. Brown, Acta Cryst., 21 (1966) 146.
19 B.C. Pant, W.R. McWhinnie and N.S. Dance, J. Organomet. Chem., 63 (1973) 305.
20 A. Sabatini and I. Bertini, Inorg. Chem., 4 (1965) 1665.