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Investigation of organoyltellurium(IV) halide (dithiolate) complexes, crystal structure of di(2-iodo-2λ^4-benzotellurol-2-yl diethyldithiocarbamate), $[C_8H_8Te(I)(S_2CNEt_2)]_2$

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Abstract

^{125}Te NMR data indicate the formation of mixed-ligand species $C_8H_8Te(X)(S\text{–}S)$ (X = Cl, Br, I; S–S = S_2CNEt_2, $S_2P(OR)_2$, S_2COR) in mixtures of $C_8H_8TeX_2$ and $C_8H_8Te(S\text{–}S)_2$. These ligand redistribution reactions are solvent dependent for S–S = $S_2P(OR)_2$, S_2COR. No evidence was found for the existence of the mixed-ligand species $C_8H_8Te(F)(S\text{–}S)$. Crystals of $[C_8H_8Te(I)(S_2CNEt_2)]_2$ are orthorhombic, space group *Pccn* with $Z = 8$, a 13.061(2), b 19.130(2), c 14.043(2). The compound is dimeric through asymmetric iodine bridges; there is evidence of a stereochemically active lone pair which gives an overall seven coordinate 1 : 2 : 2 : 2 geometry about the tellurium atom.

Introduction

The investigation of hypervalent organoyltellurium complexes has been largely restricted to investigation of the stereochemistry adopted in the solid state and comparatively few data have been reported for such complexes in solution. It was recently shown that dithiolate ligand exchange in the series of complexes $C_8H_8Te(S\text{–}S)_2$ (where S–S = S_2CNEt_2, $S_2P(OEt)_2$, S_2COEt) is rapid at room temperature, but that at low temperature this exchange slows such that the stereochemistry adopted in solution approximates to that determined for the solid state [1]. The compounds with S–S = $S_2P(OEt)_2$, S_2COEt are associated in the solid state and both show evidence for a stereochemically active lone electron pair at the tellurium atom. Furthermore, the crystal structure of $C_8H_8TeF_2$ comprises a two dimensional polymer in which there is again evidence for the presence of a stereochemically

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active lone electron pair on the tellurium atom [2]. The structures of two crystal modifications of $C_8H_8TeI_2$ have been reported, and both are associated. The structure of the α-modification [3] is similar to that of $C_8H_8TeF_2$ but the β-form of $C_8H_8TeI_2$ [4] shows no evidence for a stereochemically lone electron pair on tellurium. In view of these observations we considered it of interest to investigate a series of complexes lying between the $C_8H_8Te(S{-}S)_2$ and $C_8H_8TeX_2$ series, and we now report NMR evidence for the formation of $C_8H_8Te(X)(S{-}S)$(X = Cl, Br, I) in solution as well as the crystal and molecular structure of $[C_8H_8Te(I)(S_2CNEt_2)]_2$.

Experimental

NMR spectra were recorded with a JEOL FX 100 spectrometer, generally with broad band proton decoupling. A JEOL NM 5471 controller was used for temperature control, and the temperatures in the probe were measured with a platinum resistance thermometer. ^{125}Te spectra were recorded at 31.4 MHz, usually on a 20 kHz spectral window; the pulse width was 22 μs and the pulse delay 50 ms. Spectra were recorded in the presence of $Cr(acac)_3$ to reduce relaxation times. ^{125}Te chemical shifts were determined relative to external 0.7 *M* aqueous K_2TeO_3.

Preparation of the complexes $C_8H_8TeX_2$ (X = I, Br, Cl, F)

The complex $C_8H_8TeI_2$ was prepared by the method described by Ziolo and Günther [5]. The complexes $C_8H_8TeX_2$ (X = F, Cl, Br) were prepared by the reaction depicted in the following equation:

$$C_8H_8TeI_2 + 2AgX \rightarrow C_8H_8TeX_2 + 2AgI$$

In a typical experiment 1.5 g of $C_8H_8TeI_2$ (3 mmol) was stirred together with 1.0 g of AgCl (7 mmol) in 200 cm^3 of tetrahydrofuran for several hours or until the orange colour of $C_8H_8TeI_2$ had been discharged. The solution was filtered and then allowed to evaporate slowly. Large colourless crystals of $C_8H_8TeCl_2$ were obtained in almost quantitative yield.

Preparation of $C_8H_8Te(I)(Etdtc)$

Method A. To a suspension of $C_8H_8TeI_2$ (4 mmol) in dichloromethane (200 cm^3) was added NaEtdtc (4 mmol) (Etdtc = S_2CNEt_2). The solution was stirred for 3 h or until no orange $C_8H_8TeI_2$ crystals remained. The solution was filtered and evaporated to dryness. The crude product, $C_8H_8Te(I)(Etdtc)$ was recrystallized from CS_2/ether to give a quantitative yield of the yellow crystalline $C_8H_8Te(I)(Etdtc)$ (m.p. 141–143°C d).

Method B. To a solution of $C_8H_8Te(Etdtc)_2$ (4 mmol) in dichloromethane (200 cm^3) was added $C_8H_8TeI_2$ (4 mmol). This mixture was stirred for 1 h and then taken to dryness. The yellow solid was recrystallized from CS_2/ether to give pure $C_8H_8Te(I)(Etdtc)$ in quantitative yield (m.p. 141–143°C d).

Preparation of $C_8H_8Te(S{-}S)_2$

These complexes were prepared from reaction between $C_8H_8TeI_2$ [5] (50 mmol) with NaEtdtc · $3H_2O$, KEtdtp or KEtxan (Etdtp = $S_2P(OEt)_2$; Etxan = S_2COEt) (100 mmol) in dichloromethane solution as previously described [1].

Crystallography

The crystal of $[C_8H_8Te(I)(Etdtc)]_2$ was mounted on a CAD-4F single crystal, four-circle, automatic diffractometer. Accurate cell dimensions were obtained from the setting angles of 25 reflections by a least-squares procedure. A pre-scan showed the crystals to be orthorhombic, with systematic absences consistent with the space group *Pccn* [6]. Intensity data were collected by the $\omega:2\theta$ scan method to a maximum Bragg angle of 28.0°, using Mo-K_α radiation. Monitoring of three reflections, every 4000 s X-ray exposure time indicated an 11% increase in intensity during the data collection. The data were corrected for these intensity changes as well as for Lorentz, polarization, and absorption effects. A total of 6126 reflections were measured, of which 4239 were unique (R_{amal} 0.021) and 2597 were considered observed, $I \geq 2\sigma(I)$.

The positions of the tellurium atoms were found from a three-dimensional Patterson map. The other non-hydrogen atoms were located from subsequent difference maps. The structure was refined by a full-matrix least-squares refinement procedure, with anisotropic temperature factors assigned to all atoms. All hydrogen atoms were located from the difference maps, and were constrained at geometrical estimates with a C–H bond length of 1.08 Å. Refinement was continued with weighting schemes of type $w = k(\sigma^2(F) + gF^2)^{-1}$, where k and g were varied during the refinement. It was then noted that there were several intense low-order reflections which showed the effects of extinction, and so the structure was refined using a refinable isotropic extinction parameter, x, such that $F^{\star}_{\text{calc}} = F_{\text{calc}}(1 - (0.0001x\,F^2)/\sin\theta))$. The refinement converged with R 0.033, R_w 0.032, k 1.000, g 0.00055 and x 0.00066(1). The final difference map showed four peaks, of heights ranging from 1.20 to 0.77 e Å^{-3}, close to the tellurium atoms. All other peaks heights were less than 0.49 e Å^{-3}. Final fractional atomic coordinates are given in Table 1. Tables of anisotropic temperature factors for heavy atoms, coordinates of H atoms and their isotropic temperature factors, least-squares planes, and lists of calculated and observed structure factors may be obtained from the authors.

Table 1

Final fractional atomic coordinates for $[C_8H_8Te(I)(S_2CNEt_2)]_2$

Atom	x	y	z
C(1)	0.1647(4)	−0.0779(3)	0.2293(4)
C(2)	0.0994(4)	−0.1411(3)	0.2503(4)
C(3)	0.1377(5)	−0.1938(3)	0.3066(4)
C(4)	0.0775(6)	−0.2507(4)	0.3302(5)
C(5)	−0.0221(6)	−0.2543(3)	0.2965(5)
C(6)	−0.0618(5)	−0.2021(3)	0.2393(4)
C(7)	−0.0002(4)	−0.1445(3)	0.2151(4)
C(8)	−0.0406(4)	−0.0877(3)	0.1514(4)
Te	0.06930(2)	−0.00470(2)	0.15541(2)
I	0.15627(3)	−0.06827(2)	−0.03107(3)
S(1)	−0.01660(11)	0.03225(9)	0.30552(12)
S(2)	0.18908(10)	0.09860(9)	0.28196(12)
C(9)	0.0789(4)	0.0921(3)	0.3436(4)
N	0.0573(4)	0.1286(3)	0.4201(3)
C(10)	0.1340(5)	0.1764(4)	0.4624(5)
C(11)	0.2013(7)	0.1378(5)	0.5352(5)
C(12)	−0.0453(5)	0.1280(4)	0.4678(4)
C(13)	−0.1110(5)	0.1858(4)	0.4293(5)

Calculations were carried out using the programs SHELX-76 [7], ORTEP [8], DISTAN [9] and MEAN PLANE [10] on a VAX 11/780 computer at the University Computer Centre. Scattering curves for atomic H, C, N, and S were those listed by Sheldrick [7], while those of Te and I were taken from ref. 11, the values being corrected for the real and imaginary dispersion terms [12].

Crystal data. $C_{13}H_{18}INS_2Te$, *M* 506.91, orthorhombic, *Pccn* [No. 56], *a* 13.061(2), *b* 19.130(2), *c* 14.043(2) Å, *U* 3508.7 Å^3, *Z* 8, D_c 1.919, D_m 1.94 g cm^3; measured reflections 6126, number of unique reflections 2597, Mo-K_α (graphite-monochromatized) radiation λ 0.71069 Å, crystal dimensions, (010) 0.069, $(0\bar{1}0)$ 0.087, $(\bar{1}10)$ 0.116, $(1\bar{1}0)$ 0.103, (110) 0.103, $(\bar{1}\bar{1}0)$ 0.116, ±(001) 0.190, ±(111) 0.160, $\pm(\bar{1}11)$ 0.160, $\pm(1\bar{1}1)$ 0.160, $\pm(11\bar{1})$ 0.160 mm, absorption coefficient 36.56 cm^{-1}, max. and min. transmission factors, 0.603 and 0.480, *F*(000) 1920.

Results and discussion

Solution NMR

^{125}Te spectroscopy shows that the compound $C_8H_8TeF_2$ does not react with NaEtdtc in dcm solution. Likewise, a ^{125}Te spectrum of equimolar amounts of $C_8H_8TeF_2$ and $C_8H_8Te(Etdtc)_2$ in tetrachloroethane (tce), dcm or dmf solution shows only the two resonances corresponding to the starting reagents. Similarly, a ^{125}Te spectrum of a dichloromethane solution containing an equimolar ratio of $C_8H_8TeF_2$ with $C_8H_8Te(Etdtp)_2$ or $C_8H_8TeF_2$ with $C_8H_8Te(Etxan)_2$ gives only resonances due to the starting reagents. The inability to form $C_8H_8Te(F)(S{-}S)$ species is somewhat surprising, since the fluoride atoms in $C_8H_8TeF_2$ are labile, as is the dithiolate ligand in $C_8H_8Te(S{-}S)_2$. However, the compounds $C_8H_8TeX_2$ (X = Cl, Br, I) do react with alkali metal salts of alkyl xanthates [KS_2COR], dialkyldithiophosphates [$KS_2P(OR)_2$] and dialkyldithiocarbarbamates [NaS_2CNR_2] (R = Et, ^{i}Pr), and one or both halides may be replaced by a dithiolate ligand. The ^{125}Te shifts for $C_8H_8Te(X)(S{-}S)$ within each dithiolate ligand series moves to higher frequency as the electronegativity of the halide increases (Table 2). In general, only the dithiocarbamate derivatives $C_8H_8Te(X)(S_2CNR_2)$ could be isolated. For X = I, the three dithiolate compounds, $C_8H_8Te(I)(Etxan)$, $C_8H_8Te(I)(Etdtp)$ and $C_8H_8Te(I)(Etdtc)$ show large variations in their behaviour in solution. The compound $C_8H_8Te(I)(Etxan)$ may also be obtained by mixing equimolar quantities of $C_8H_8TeI_2$ and $C_8H_8Te(Etxan)_2$ in dichloromethane solution. The ^{125}Te spectrum of this solution at 25 °C shows that $C_8H_8Te(I)(Etxan)$ is the main species in solution, together with a trace of $C_8H_8Te(Etxan)_2$. The complex $C_8H_8Te(I)(Etxan)$ is unstable in dichloromethane solution, from which characteristic red/orange crystals of $C_8H_8TeI_2$ separated within 24 h. A ^{125}Te spectrum recorded one day after the initial preparation showed that the intensity of the resonance due to the species $C_8H_8Te(Etxan)_2$ had increased, with a corresponding decrease in the intensity of the resonance due to $C_8H_8Te(I)(Etxan)$. When the reaction is carried out in dmf solvent, at −40 °C, a statistical equilibrium mixture of the three species in eq. 1 is observed. The proportions within the equilibrium mixture do not appear to vary with time.

$$C_8H_8Te(Etxan)_2 + C_8H_8TeI_2 \leftrightarrow 2C_8H_8Te(I)(Etxan) \qquad (1)$$

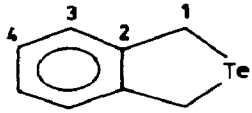
^{125}Te NMR spectra indicate that mixing equimolar quantities of $C_8H_8TeI_2$ and

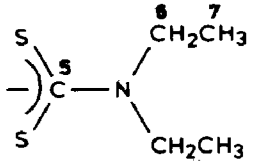
Table 2

NMR data for complexes $C_8H_8Te(X)(S{-}S)$ (X = I, Br, Cl, S–S = xanthate, dithiophosphate, dithiocarbamate)

Compound	$\delta(^{125}Te)$	$\delta(^{13}C)$ [a]						
		1	2	3	4	5	6	7
$C_8H_8Te(Cl)(Etdtc)$ [b]	−869							
$C_8H_8Te(Br)(Etdtc)$ [b]	−982							
$C_8H_8Te(I)(Etdtc)$ [b]	−1006							
$C_8H_8Te(I)(Etdtc)$ [c]	−1070							
		46.7	139.5	127.8	129.9	195.0	49.4	12.3
$C_8H_8Te(Cl)(Etdtp)$ [b,f]	−975							
$C_8H_8Te(Br)(Etdtp)$ [b,f]	−888							
$C_8H_8Te(I)(Etdtp)$ [b,e]	−926							
$C_8H_8Te(Cl)(Etxan)$ [b]	−923							
$C_8H_8Te(Br)(Etxan)$ [b]	−935							
		47.3	140.0	127.7	130.5	218.4	72.3	13.5
$C_8H_8Te(I)(Etxan)$ [b]	−964							
$C_8H_8Te(I)(Etxan)$ [d]	−1037							
		47.2	139.7	128.4	130.5	218.5	72.7	14.4
$C_8H_8Te(I)(^iPrxan)$ [b]	−1044							
		47.2	140.0	128.6	130.6	220.4	81.4	21.8

[a] Assignment as illustrated.





[b] In dmf at −40 °C. [c] In dcm at 30 °C. [d] In dcm at 0 °C. [e] $\delta(^{31}P)$ 94.6. [f] $\delta(^{31}P)$ 94.0.

$C_8H_8Te(Etdtp)_2$ in dichloromethane solution does not give the mixed species $C_8H_8Te(I)(Etdtp)$. Neither is the latter formed when $C_8H_8TeI_2$ is mixed with a molar equivalent amount of KEtdtp in dichloromethane, the solution exhibiting NMR spectra of the initial reagents. Similarly, no mixed-ligand species is observed when $C_8H_8TeI_2$ is mixed with $C_8H_8Te(Etdtp)_2$ at 80 °C in tetrachloroethane. However changing the solvent has a pronounced effect, and in dmf solution an equimolar mixture of $C_8H_8TeI_2$ and KEtdtp gives a statistical distribution of products similar to that in eq. 1. The same products are observed by ^{125}Te NMR, in dmf when equimolar amounts of $C_8H_8TeI_2$ and $C_8H_8Te(Etdtp)_2$ are mixed (Table 2). The compound $C_8H_8TeI_2$ is more soluble in dmf, and presumably this is the reason why statistical distributions of products are observed in the above mixtures in dmf but not in dcm solution.

NMR study of $C_8H_8Te(I)(Etdtc)$

Unlike the species $C_8H_8Te(I)(Etxan)$ and $C_8H_8Te(I)(Etdtp)$, whose behaviour in solution seems dependent on the nature of the solvent, the compound $C_8H_8Te(I)(Etdtc)$ is formed irrespective of the nature of the solvent. ^{125}Te spectra show that when an equimolar amount of $C_8H_8TeI_2$ and NaEtdtc is mixed in dichloromethane, only $C_8H_8Te(I)(Etdtc)$ is formed in solution. The same single product is obtained when $C_8H_8TeI_2$ is treated with an equimolar ratio of $C_8H_8Te(Etdtc)_2$ in dcm or dmf solution.

The ^{125}Te spectrum of $C_8H_8Te(I)(Etdtc)$ in dcm at 30 °C shows a single sharp resonance at −1070 ppm. The same compound in dmf gives a signal at $\delta(^{125}Te)$ −1006 ppm, and it is likely that the appreciable difference in chemical shifts is due to interaction between dmf solvent, acting as a Lewis base, and the tellurium(IV) centre. The ^{13}C spectrum at 30 °C in dichloromethane shows equivalent benzylic methylene carbon atoms (Table 2). Similarly the methyl and methylene carbon atoms of the dithiocarbamate residue are equivalent, and each gives a single resonance. Three aromatic ^{13}C resonances are observed, which suggests that the C_8H_8Te group lies on a symmetry axis. Cooling the soltuion to −40 °C causes the ^{13}C resonances of the methyl and methylene carbons of the dithiocarbamate ligand to broaden, with all other resonances remaining sharp. A further lowering of temperature to −80 °C causes each of the methyl and methylene ^{13}C resonances to split into two (i.e. $\delta(^{13}C)$ 11.2 and 12.2 ppm, and $\delta(^{13}C)$ 49.1 and 49.9 ppm, respectively; Fig. 1). At temperatures above about −80 °C it is likely that there is rapid intramolecular monodentate-bidentate dithiocarbamate ligand exchange; at −80 °C this process is slow on the NMR time-scale so that the dithiocarbamate ligand becomes effectively bidentate [13,1]. It has also been shown that $S_2C–N$ bond rotation is also slow at this temperature [14–16]. Once the dithiocarbamate ligand is effectively bidentate the ethyl groups become non-equivalent (i.e. anisochronous), as can be seen from Fig. 1.

Although dimeric in the solid state, molecular weight measurements in chloroform at 37 °C show the complex $C_8H_8Te(I)(Etdtc)$ to be only partially associated

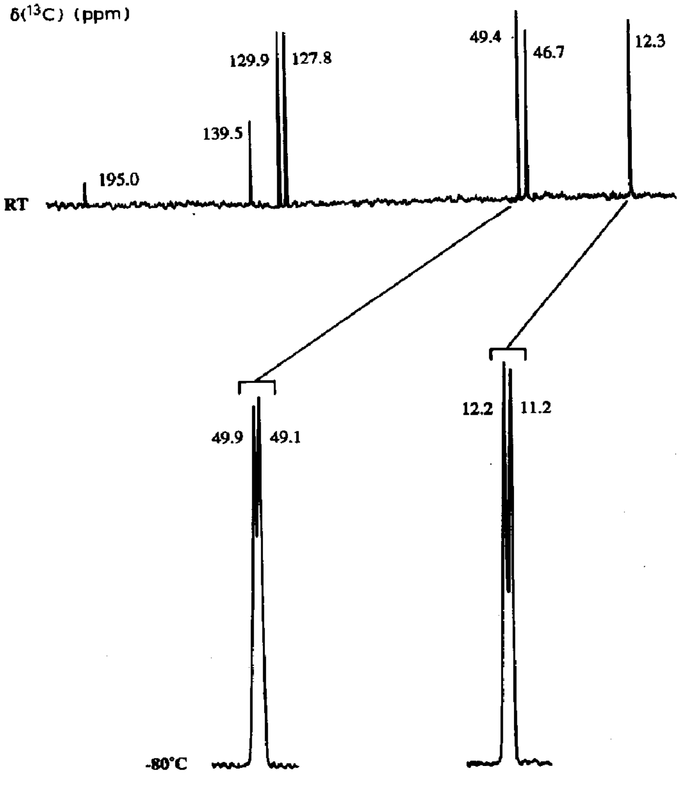


Fig. 1. ^{13}C NMR spectra for $C_8H_8Te(I)(Etdtc)$ in dichloromethane solution.

Table 3

Important bond lengths (Å) and angles (°) for $[C_8H_8Te(I)S_2CNEt_2)]_2$

Te–I	3.1027(5)	Te–I^1	3.6983(5)
Te–C(1)	2.143(6)	Te–C(8)	2.141(6)
Te–S(1)	2.490(2)	Te–S(2)	3.084(2)
S(1)–C(9)	1.776(6)	S(2)–C(9)	1.684(6)
C(9)–N	1.312(7)	N–C(10)	1.481(9)
C(10)–C(11)	1.537(11)	N–C(12)	1.498(8)
C(12)–C(13)	1.500(10)		
C(1)–Te–C(8)	85.3(2)	C(1)–Te–S(1)	92.15(15)
C(1)–Te–S(2)	81.07(15)	C(1)–T–I	86.54(15)
C(1)–Te–I^1	159.83(15)	C(8)–Te–S(1)	86.03(15)
C(8)–Te–S(2)	146.14(15)	C(8)–Te–I	86.13(15)
C(8)–Te–I^1	74.54(15)	S(1)–Te–S(2)	63.83(5)
S(1)–Te–I^1	86.19(4)	S(2)–Te–I	123.49(3)
S(2)–Te–I^1	115.74(3)	I–Te–I^1	92.355(12)
Te–I–Te^1	87.645(12)	Te–S(1)–C(9)	97.0(2)
Te–S(2)–C(9)	79.4(2)	S(1)–C(9)–S(2)	119.5(3)
S(1)–C(9)–N	116.0(4)	S(2)–C(9)–N	124.4(4)
C(9)–N–C(10)	120.8(5)	C(12)–N–C(10)	115.5(5)
C(9)–N–C(12)	123.7(5)	N–C(10)–C(11)	110.9(6)
N–C(12)–C(13)	110.2(5)		

($M_{calc.}$ 506.9, range of M_{obs}. 528–561 within the concentration range 7.73–28.87 g l^{-1}) with the monomer-dimer equilibrium $2C_8H_8Te(I)(Etdtc) \leftrightarrow [C_8H_8Te(I)(Etdtc)]_2$ favouring monomer formation under these conditions. The NMR experiments were carried out with more highly concentrated solutions than those used for the molecular weight determination, and so dimer formation would be favoured. Furthermore since dimer formation is likely to be favoured at lower temperatures it is possible that in solution at −80 °C, the compound $C_8H_8Te(I)(Etdtc)$ is dimeric, with a configuration resembling that found in the solid state, the structure being based on a 1 : 2 : 2 : 2 geometry with a stereochemically active lone electron pair at the tellurium atom (see below).

Low temperature ^{13}C studies for the mixtures containing the species $C_8H_8Te(I)(Etdtp)$ and $C_8H_8Te(I)(Etxan)$ could not be carried out owing to the superposition of resonances arising from the individual components of the mixture.

Description of the structure of $[C_8H_8Te(I)(Etdtc)]_2$

Relevant bond lengths, and angles are listed in Table 3. An ORTEP diagram of the molecule, showing the numbering scheme, is shown in Fig. 2. The compound is dimeric in the solid state, as a result of asymmetric iodine bridges [3.1027(5), 3.6983(5) Å]. The dithiocarbamate ligand in $C_8H_8Te(I)(Etdtc)$ is chelated to the tellurium atom, with one long and one short tellurium–sulphur bond [i.e. Te–S(2) 3.084(2) and Te–S(1) 2.490(2) Å respectively]. The long tellurium–sulphur distance is significantly less than the sum of the Van der Waals radii (3.86 Å) [17], so that the coordination number of the tellurium atom can be assigned unambiguously as seven, with the seventh position, opposite the C_8H_8 group, being occupied by a stereochemically active lone pair. The shorter tellurium–sulphur distances in $[C_8H_8Te(I)(Etdtc)]_2$ indicate that the dithiocarbamate ligand is more strongly bonded

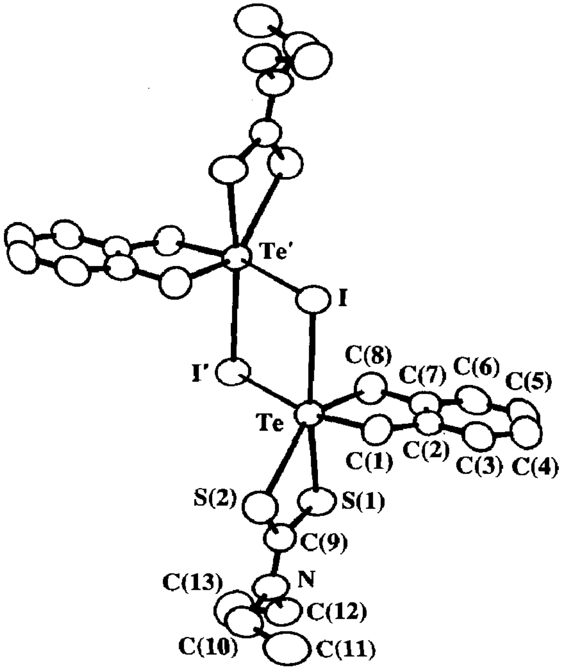


Fig. 2. ORTEP diagram for $[C_8H_8Te(I)(Etdtc)]_2$ showing the numbering scheme employed.

to the tellurium atom, than are those in $C_8H_8Te(Etdtc)_2$ [1]. The tellurium–iodine bonds [i.e. Te–I 13.103(1), Te–I^1 3.698(1) Å; where ^{1} refers to that atom related by the symmetry operation $-x$, $-y$, $-z$], are similar to those observed in the two forms of $C_8H_8TeI_2$ [3,4]. The geometry about the tellurium atom in $[C_8H_8Te(I)(Etdtc)]_2$ is a distortion of that observed in α-$C_8H_8TeI_2$ [3]. The distortion appears to be due to the restricted bite of the dithiocarbamate ligand.

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