

Reactions of *trans*-2-alkoxy-1-cycloalkyltellurium(IV) trihalides with N-substituted anilines: the X-ray crystal structure of *p*-*N*-ethylanilino(*trans*-2-ethoxy-1-cyclohexyl)tellurium(IV) dibromide

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Abstract

trans-2-Alkoxy-1-cycloalkyltellurium(IV) trihalides react with N-substituted anilines to give *p*-N-substituted anilino(*trans*-2-alkoxy-1-cycloalkyl)tellurium(IV) dihalides. The crystal structure of *p*-*N*-ethylanilino(*trans*-2-ethoxy-1-cyclohexyl)tellurium(IV) dibromide has been determined by X-ray crystallography. Tellurium is four coordinated with pseudo trigonal bipyramidal geometry and an equatorial lone pair of electrons. Single crystals of *p*-EtHNC₆H₄(EtOC₆H₁₀)TeBr₂ are monoclinic with space group *P*2₁/*c* (No. 14), *a* = 9.304(5), *b* = 8.179(2), *c* = 24.932(5) Å, β = 92.34(4)°, *V* = 1896(1) Å³, *D*_{calc} = 1.8738 g cm⁻³ for *Z* = 4. The structure was refined to *R* = 0.0538 (*R*_w = 0.0633).

Introduction

Tellurium tetrachloride reacts with cyclohexene to give *trans*-2-chloro-1-cyclohexyltellurium(IV) trichloride [1] and di(*trans*-2-chloro-1-cyclohexyl)tellurium(IV) dichloride [2] whereas reactions of tellurium tetrachloride and tellurium tetrabromide with cycloalkenes in the presence of alcohols give *trans*-2-alkoxy-1-cycloalkyltellurium(IV) trihalides [3–5]. It is shown here that *trans*-2-alkoxy-1-cycloalkyltellurium(IV) trihalides react with aniline and its simple N-substituted derivatives to give *p*-substituted anilino(*trans*-2-ethoxy-1-cyclohexyl)tellurium(IV) dihalides. The X-ray crystal structure of *p*-*N*-ethylanilino(*trans*-2-ethoxy-1-cyclohexyl)tellurium(IV) dibromide has been determined. Aminoaryltellurium(IV) derivatives are relatively uncommon [6] and these are the first examples of aminoaryl(cycloalkyl)tellurium compounds as far as we are aware.

Experimental

In a typical preparation, *trans*-2-ethoxy-1-cyclohexyltellurium(IV) tribromide (2.00 g, 4.05 mmol) [5] reacted

with an excess of *N*-ethylaniline (4 cm³) at room temperature (24 h). The solid yellow mass was washed with water to remove *N*-ethylaniline hydrochloride. Recrystallisation of the product from chloroform gave orange *p*-*N*-ethylanilino(*trans*-2-ethoxy-1-cyclohexyl)tellurium(IV) dibromide (1.74 g, 3.26 mmol, 80%), m.p. 143–144 °C. Analytical data and m.p.s. for eight anilino(*trans*-2-alkoxy-1-cycloalkyl)tellurium(IV) dihalides are recorded in Table 1. The products form yellow or orange crystals which are reasonably stable in air and recrystallise readily from common organic solvents.

Data collection and structure refinement

A suitable crystal (0.2 × 0.25 × 0.33 mm) of *p*-*N*-ethylanilino(*trans*-2-ethoxy-1-cyclohexyl)tellurium(IV) dibromide was selected for data collection on an Enraf-Nonius CAD4 diffractometer operating in the ω/2θ scan mode with graphite-monochromated Mo Kα radiation (λ = 0.70926 Å). The lattice parameters and orientation matrices were obtained by least-squares refinement using setting angles for 25 reflections (10 ≤ θ ≤ 15). Crystallographic data and information concerning the data collection and refinement procedure are given in Table 2. The intensities were reduced to a standard scale using routine procedures [7]. The data were corrected for Lorentz and polarisation effects.

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TABLE 1. Analysis of *p*-*N*-ethylanilino(*trans*-2-alkoxy-1-cycloalkyl)tellurium(IV) trihalides

Compound	Melting point (°C)	Found (%)				Formula	Calculated (%)			
		C	H	Hal	N		C	H	Hal	N
<i>p</i> -H ₂ NC ₆ H ₄ (EtOC ₆ H ₁₀)TeBr ₂	158–160	33.14	4.11	31.55	2.66	C ₁₄ H ₂₁ Br ₂ NOTe	33.18	4.18	31.54	2.76
<i>p</i> -MeHNC ₆ H ₄ (EtOC ₆ H ₁₀)TeBr ₂	142–143	34.76	4.75	30.71	2.79	C ₁₅ H ₂₃ Br ₂ NOTe	34.60	4.45	30.69	2.69
<i>p</i> -Me ₂ NC ₆ H ₄ (EtOC ₆ H ₁₀)TeBr ₂	178	35.83	4.83	29.81	2.73	C ₁₆ H ₂₅ Br ₂ NOTe	35.93	4.71	29.88	2.62
<i>p</i> -EtHNC ₆ H ₄ (MeOC ₆ H ₁₀)TeBr ₂	174–175	34.54	4.45	30.87	2.58	C ₁₅ H ₂₃ Br ₂ NOTe	34.60	4.45	30.69	2.69
<i>p</i> -EtHNC ₆ H ₄ (EtOC ₆ H ₁₀)TeBr ₂	143–144	35.76	4.76	30.94	2.45	C ₁₆ H ₂₅ Br ₂ NOTe	35.93	4.71	29.88	2.62
<i>p</i> -EtHNC ₆ H ₄ (MeOC ₇ H ₁₂)TeCl ₂	oil	43.52	5.73	15.81	3.25	C ₁₆ H ₂₅ Cl ₂ NOTe	43.10	5.65	15.90	3.14
<i>p</i> -Et ₂ NC ₆ H ₄ (EtOC ₆ H ₁₀)TeBr ₂	152–154	38.43	5.37	28.10	2.31	C ₁₈ H ₂₉ Br ₂ NOTe	38.41	5.19	28.39	2.49
<i>p</i> -PhHNC ₆ H ₄ (EtOC ₆ H ₁₀)TeBr ₂	145–147	41.43	4.49	26.62	2.78	C ₂₀ H ₂₅ Br ₂ NOTe	41.22	4.32	27.42	2.75

TABLE 2. Physical properties and parameters for data collection and refinement of C₁₆H₂₅Br₂NOTe

Formula	C ₁₆ H ₂₅ Br ₂ NOTe
Molecular weight	534.79
Crystal class	monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i> (No. 14)
<i>a</i> (Å)	9.304(5)
<i>b</i> (Å)	8.179(2)
<i>c</i> (Å)	24.932(5)
β (°)	92.34(4)
<i>V</i> (Å ³)	1896(1)
<i>Z</i>	4
<i>D</i> _{calc} (g cm ⁻³)	1.8738
<i>F</i> (000)	1032.00
Linear absorption coefficient, μ (cm ⁻¹)	57.48
Temperature (°C)	18
Scan speed (° min ⁻¹)	variable: 1.6–3.3
Scan width (°)	1.2 + 0.35 tan θ
<i>hkl</i> Range	<i>h</i> : -10 10, <i>k</i> : 0 9, <i>l</i> : 0 27
θ Range (°)	3–23
Standards (period.)	
orientation (200 refls.)	-1 2 13 / 1 4 2 / 5 2 3
intensity (2 h)	no loss
No. measured reflections	2905
No. unique reflections	2614 (<i>R</i> _m = 0.1108)
No. observed reflections (<i>NO</i>) [<i>I</i> > 3 σ (<i>I</i>)]	1299
No. variables (<i>NV</i>)	232
Absorption coefficients	max. 1.367, min. 0.668
Weighting scheme $w = k/[\sigma^2(F) + gF^2]$	<i>k</i> 1.0259, <i>g</i> 0.0277
Agreement factors	
$R = (\sum F_{\text{obs}} - F_{\text{calc}}) / (\sum F_{\text{obs}})$	0.0538
$R_w = [\sum w (F_{\text{obs}} - F_{\text{calc}})^2 / (\sum w F_{\text{obs}})^2]^{1/2}$	0.0633
$GOF = [\sum w (F_{\text{obs}} - F_{\text{calc}})^2 / (NO - NV)]^{1/2}$	0.4777

The absorption corrections were applied by DIFABS [8]. Scattering factors for neutral atoms [9] were corrected for the real part of anomalous dispersion. The structure was solved by SHELXS-86 [10]. Calculations and refinements were performed by SHELX-76 [11].

The systematically absent reflections confirmed the space group as *P*2₁/*c*. The positions of the remaining non-hydrogen atoms were located from successive difference Fourier syntheses. The last isotropic refinement

of the non-hydrogen atoms gave an *R* value equal to 0.1139. Absorption corrections were then applied and subsequent isotropic and anisotropic refinement reduced the *R* factor to 0.0754 and 0.0631, respectively. Hydrogen atoms attached to carbon were placed in their geometrically calculated positions, *d*(C–H) = 1.08 Å, and hydrogen attached to nitrogen was located from the difference map. After omitting several reflections with suspected extinction, the final refinements with

anisotropic temperature factors on the non-hydrogen atoms and isotropic temperature factors on the hydrogen atoms gave $R=0.0538$ and $R_w=0.0633$. The function minimised was $\sum w(|F_o| - |F_c|)^2$ where w is the weight (Table 2). No correction for extinction was applied. In the final difference map, the deepest hole was -0.85 and the highest peak $1.31 \text{ e } \text{Å}^{-3}$ at 0.95 Å from bromine (Br(2)). The figures were drawn with CHEMGRAF [12]. The rigid body vibration analysis of this molecule was carried out by TLS calculation [13].

Results and discussion

Cationic *trans*-2-ethoxy-1-cyclohexyldibromotellurium(IV) undergoes a quasi Friedel-Crafts reaction with aniline to give *p*-anilino(*trans*-2-ethoxy-1-cyclohexyl)-tellurium(IV) dibromide. *trans*-2-Ethoxy-1-cyclohexyl-tellurium(IV) tribromide reacts similarly with an excess of *N*-methyl-, *N,N*-dimethyl-, *N*-ethyl-, *N,N*-diethyl or *N*-phenyl-anilines at room temperature to give *p*-substituted anilino(*trans*-2-ethoxy-1-cyclohexyl)tellurium(IV) dibromides. *trans*-2-Methoxy-1-cyclohexyltellurium(IV) tribromide and *trans*-2-methoxy-1-cycloheptyl-tellurium(IV) trichloride react similarly with *N*-ethyl-aniline to give *p-N*-ethylanilino(*trans*-2-alkoxy-1-cycloalkyl)tellurium(IV) dihalides.

Proton NMR spectra

Proton NMR data for the three *p-N*-ethylanilino(*trans*-2-alkoxy-1-cycloalkyl)tellurium(IV) dihalides in CDCl_3 are summarised in Table 3. Methyl protons of ethoxy and *N*-ethyl groups appear as triplets which overlap the unresolved multiplets of cycloalkyl methylene groups at high field. Amino protons appear as

a broad band and *N*-methylene protons appear at slightly lower field as quartets through coupling with neighbouring methyl protons. Aryltellurium protons appear at low field as four-line AA'BB' signals characteristic of 1,4-disubstituted benzenes [14]. No traces of 1,2- or 1,3-disubstituted products were detected.

A portion of the resolution-enhanced proton spectrum of *p-N*-ethylanilino(*trans*-2-ethoxy-1-cyclohexyl)-tellurium(IV) dibromide is reproduced in Fig. 1. Non-equivalent methylene protons of the ethoxy group appears as two sets of eight lines (quartets of AB signals) through coupling with each other and with neighbouring methyl protons. The cyclohexyl H-CTe (A) and H-COEt (B) protons appear as six-line signals (doublets of triplets) through coupling with each other and with neighbouring cyclohexyl methylene protons. Coupling constants between *trans* diaxial protons (A-B, A-C and B-E) are approximately equal and greater than those between *cis* axial-equatorial protons (A-D and B-F). The cyclohexyl H-COMe (B) proton in *p-N*-ethyl-anilino(*trans*-2-methoxy-1-cyclohexyl)tellurium(IV) dibromide appears as an eight-line signal, shown in Fig. 2, because the *trans* diaxial protons (A-B and B-E) have different coupling constants as in *p*-phenoxyphenyl(*trans*-2-ethoxy-1-cycloalkyl)tellurium(IV) dichlorides [15]. These patterns confirm that the products have 1,2-*trans* configurations and that the substituents occupy equatorial positions in solution.

Carbon-13 NMR spectra

Chemical shifts for the three *p-N*-ethylanilino(*trans*-2-alkoxy-1-cycloalkyl)tellurium(IV) dihalides are summarised in Table 4. The four aryl signals appear at low field with C-Te below the two C-H signals of double intensity and C-N. Ethylamino carbons appear

TABLE 3. Proton NMR data at 400 MHz for *p-N*-ethylanilino(*trans*-2-alkoxy-1-cycloalkyl)tellurium(IV) trihalides in CDCl_3

Compound	Aryl			Amino	Ethylamino				
	$\delta(\text{AA}')$ (ppm)	$\delta(\text{BB}')$ (ppm)	$^3J(\text{A-B})$ (Hz)	$\delta(\text{HN})$ (ppm)	$\delta(\text{H}_2\text{CN})$ (ppm)	$\delta(\text{H}_3\text{C})$ (ppm)	$^3J(\text{H}_2\text{C-CH}_3)$ (Hz)		
<i>p</i> -EtHNC ₆ H ₄ (MeOC ₆ H ₁₀)TeBr ₂	7.97	6.61	9.0	4.02	3.19	1.27	7.1		
<i>p</i> -EtHNC ₆ H ₄ (EtOC ₆ H ₁₀)TeBr ₂	7.97	6.60	9.0	4.05	3.19	1.27	7.1		
<i>p</i> -EtHNC ₆ H ₄ (MeOC ₇ H ₁₂)TeCl ₂	7.93	6.67	9.0	4.00	3.14	1.26	7.1		
	Cycloalkyl						Alkoxy		
	HCTe $\delta(\text{A})$ (ppm)	HCOAlk $\delta(\text{B})$ (ppm)	$^3J(\text{A-D})$ (Hz)	$^3J(\text{A-B})$ $^3J(\text{A-C})$ (Hz)	$^3J(\text{B-E})$ (Hz)	$^3J(\text{B-F})$ (Hz)	$\delta(\alpha)$ (ppm)	$\delta(\beta)$ (ppm)	
<i>p</i> -EtHNC ₆ H ₄ (MeOC ₆ H ₁₀)TeBr ₂	3.96	4.14	4.1	10.6	12.5	4.1	3.49		
<i>p</i> -EtHNC ₆ H ₄ (EtOC ₆ H ₁₀)TeBr ₂	4.05	4.15	4.2	10.9	10.9	4.3	3.55 ^a	1.27	
							3.84		
<i>p</i> -EtHNC ₆ H ₄ (MeOC ₇ H ₁₂)TeCl ₂	3.80	4.15	3.5	10.3	10.3	4.4	3.50		

^a $^2J(\alpha_1-\alpha_2)9.2 \text{ Hz}$, $^3J(\alpha-\beta)6.9 \text{ Hz}$.

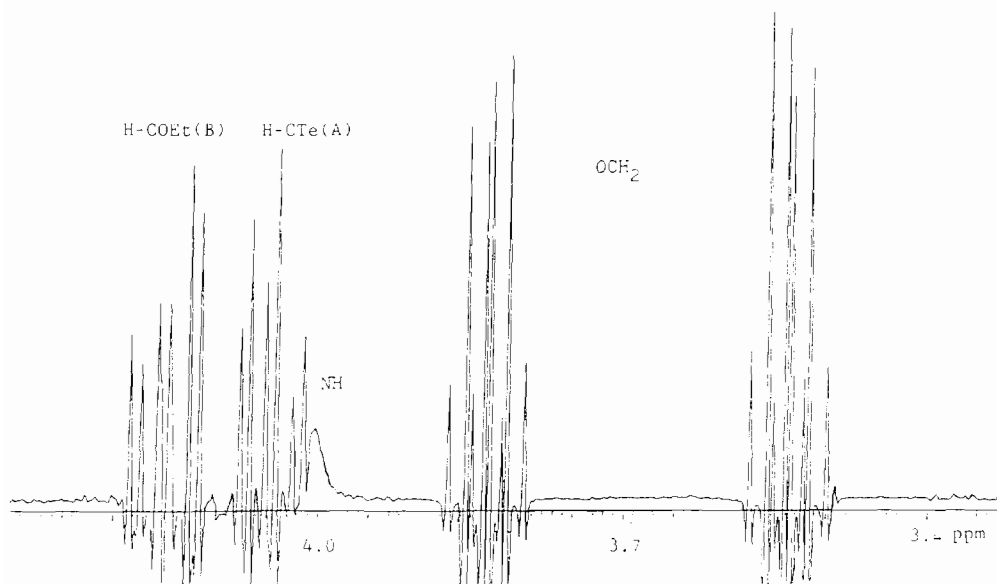


Fig. 1. Resolution enhanced ^1H NMR signals at 400 MHz for H-N, OCH_2 , and cyclohexyl H-CTe (A) and H-COEt (B) protons of *p*-*N*-ethylanilino(*trans*-2-ethoxy-1-cycloalkyl)tellurium(IV) dibromide in CDCl_3 .

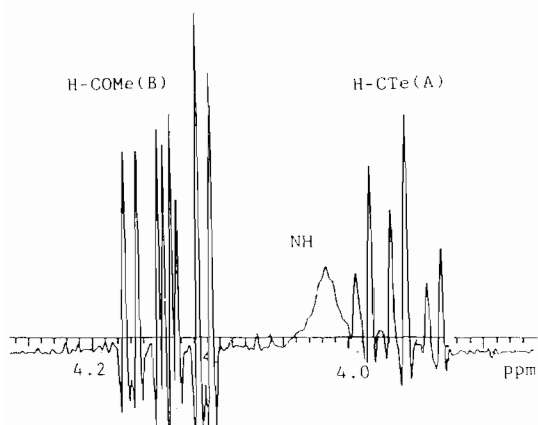


Fig. 2. Resolution enhanced ^1H NMR signals at 400 MHz for H-N and cyclohexyl H-CTe (A) and H-COMe (B) protons of *p*-*N*-ethylanilino(*trans*-2-methoxy-1-cyclohexyl)tellurium(IV) dibromide in CDCl_3 .

at higher field than ethoxy carbons which have chemical shifts similar to those in *trans*-2-alkoxy-1-cycloalkyltellurium(IV) trihalides [5]. Cycloalkyl C-Te appears at higher field than C-Oalk as in *p*-anisyl- and *p*-phenoxyphenyl-(*trans*-2-ethoxy-1-cycloalkyl)tellurium(IV) dichlorides [15].

Crystal structure

The structure of *p*-*N*-ethylanilino(*trans*-2-ethoxy-1-cyclohexyl)tellurium(IV) dibromide is shown in Fig. 3. Positional parameters are given in Table 5 and interatomic distances and angles are listed in Table 6. Tellurium has the pseudo trigonal bipyramidal config-

uration found in other four-coordinated organotellurium(IV) compounds [2, 16] with two bromines in axial positions, two carbon rings in equatorial positions, and the lone pair of electrons of tellurium in the third equatorial position.

Internuclear distances within the coordination sphere are: Te(1)-Br(1) 2.683(2), Te(1)-Br(2) 2.662(2), Te(1)-C(1) 2.18(2), Te(1)-C(9) 2.12(1) Å. As expected, Te-C(alkyl) is longer than Te-C(aryl) and the bond lengths agree well with values reported earlier [2, 3, 16]. The Br(1)-Te(1)-Br(2) bond angle is $176.97(8)^\circ$, smaller than 180° , and the C(1)-Te(1)-C(9) bond angle is $100.0(6)^\circ$, smaller than 120° , presumably because of the repulsive influence of the lone pair of electrons. The non-bonding separation, Te(1)---O(1) 3.058 Å, is significantly longer than the dative bond from oxygen to tellurium in *p*-tolyl(*trans*-2-ethoxy-1-cyclohexyl)tellurium(IV) dichloride, Te(1)-O(1) 2.49(3) Å, where tellurium has a pseudo octahedral configuration with the lone pair of electrons *trans* to cyclohexyl C(1) [3]. The amino N(1) of one molecule of *p*-*N*-ethylanilino(*trans*-2-ethoxy-1-cyclohexyl)tellurium(IV) dibromide is hydrogen bonded weakly to Br(2) of a second molecule through H(110) as shown in Fig. 4. The N(1)-H(110) and H(110)-Br(2) distances are 1.30(10) and 2.378 Å, respectively.

The cyclohexyl ring adopts the usual chair conformation with C(1) 0.60 Å above and C(4) 0.59 Å below the plane through C(2), C(3), C(5) and C(6). The average C-C bond length is 1.52(1) Å. The average C-C-C bond angle of $112.6(1)^\circ$, slightly greater than 111.3° in cyclohexane, indicates some flattening of the ring which is consistent with the average value of

TABLE 4. Carbon-13 NMR chemical shifts for *p*-*N*-ethylanilino(*trans*-2-alkoxy-1-cycloalkyl)tellurium(IV) trihalides in CDCl₃

Compound	Aryl				Alkoxy	Ethylamino	
	$\delta(\text{CTe})$ (ppm)	$\delta(\text{C}_{2,5})$ (ppm)	$\delta(\text{C}_{3,4})$ (ppm)	$\delta(\text{CN})$ (ppm)		$\delta(\text{CN})$ (ppm)	$\delta(\text{CH}_3)$ (ppm)
<i>p</i> -EtHNC ₆ H ₄ (MeOC ₆ H ₁₀)TeBr ₂	150.47	137.77	113.27	107.31	56.50	37.90	14.60
<i>p</i> -EtHNC ₆ H ₄ (EtOC ₆ H ₁₀)TeBr ₂	150.50	137.80	113.30	107.66	64.47 ^a	37.93	14.64
<i>p</i> -EtHNC ₆ H ₄ (MeOC ₇ H ₁₂)TeCl ₂	150.47	136.36	113.30	112.32	57.16	37.93	14.64
Cycloalkyl							
	$\delta(\text{CTe})$ (ppm)	$\delta(\text{COAlk})$ (ppm)	$\delta(\text{CH}_2)$ (ppm)	$\delta(\text{CH}_2)$ (ppm)	$\delta(\text{CH}_2)$ (ppm)	$\delta(\text{CH}_2)$ (ppm)	$\delta(\text{CH}_2)$ (ppm)
<i>p</i> -EtHNC ₆ H ₄ (MeOC ₆ H ₁₀)TeBr ₂	70.66	79.45	31.86	27.59	27.42	23.56	
<i>p</i> -EtHNC ₆ H ₄ (EtOC ₆ H ₁₀)TeBr ₂	70.78	78.10	32.79	27.56	27.43	23.65	
<i>p</i> -EtHNC ₆ H ₄ (MeOC ₇ H ₁₂)TeCl ₂	75.77	81.32	31.73	26.94	26.06	25.92	22.79

^a $\delta(\text{CH}_3)$ 15.48 ppm.

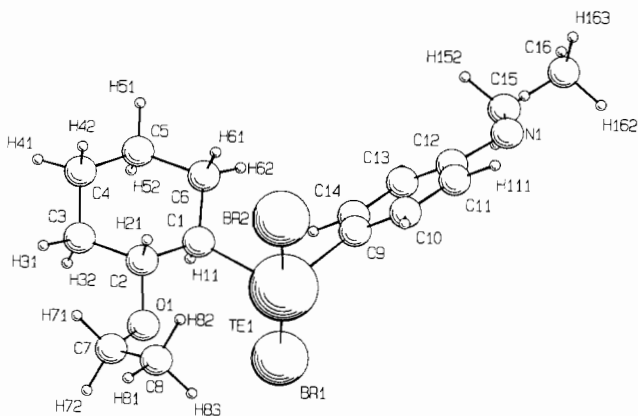


Fig. 3. The *p*-*N*-ethylanilino(*trans*-2-ethoxy-1-cycloalkyl)-tellurium(IV) dibromide molecule.

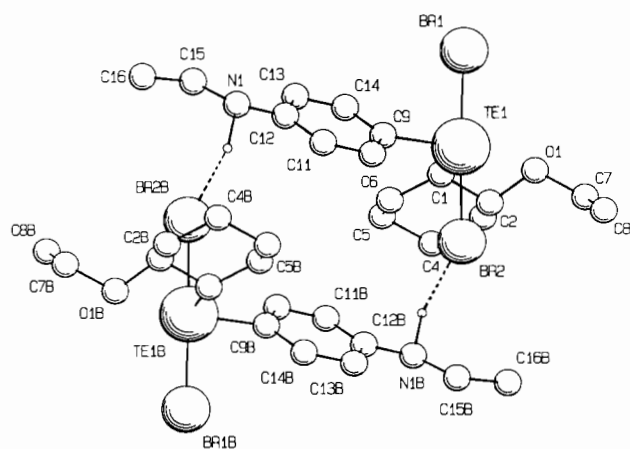


Fig. 4. Hydrogen bonding between *p*-*N*-ethylanilino(*trans*-2-ethoxy-1-cycloalkyl)tellurium(IV) dibromide molecules.

51.2(7)° for the torsional angles. The average C–C bond lengths and C–C–C bond angles in the phenyl ring are 1.39(1) Å and 120.0(5)°, respectively.

TABLE 5. Fractional atomic positional parameters and equivalent isotropic temperature factors (\AA^2) for C₁₆H₂₅Br₂NO₂Te with e.s.d.s. in parentheses

Atom	<i>x/a</i>	<i>y/b</i>	<i>z/c</i>	U_{eq}^a
Tel	0.21047(11)	−0.13725(14)	0.88845(4)	0.0366
Br1	0.10501(19)	0.16002(23)	0.90971(9)	0.0559
Br2	0.30462(23)	−0.43358(25)	0.86315(9)	0.0620
Cl	−0.0025(22)	−0.2371(23)	0.9008(7)	0.0555
C2	−0.0677(21)	−0.2900(19)	0.8461(7)	0.0498
C3	−0.2227(20)	−0.3404(24)	0.8492(9)	0.0624
C4	−0.2447(17)	−0.4622(28)	0.8918(8)	0.0647
C5	−0.1766(16)	−0.4117(20)	0.9477(7)	0.0433
C6	−0.0153(19)	−0.3559(23)	0.9442(7)	0.0557
O1	−0.0513(15)	−0.1556(15)	0.8104(5)	0.0701
C7	−0.0543(19)	−0.2012(25)	0.7545(6)	0.0717
C8	0.0960(22)	−0.2290(42)	0.7327(11)	0.1242
C9	0.3030(12)	−0.1598(18)	0.9673(5)	0.0366
C10	0.4409(10)	−0.2246(20)	0.9733(5)	0.0390
C11	0.5073(16)	−0.2257(22)	1.0240(4)	0.0456
C12	0.4326(12)	−0.1709(15)	1.0677(4)	0.0238
C13	0.2937(13)	−0.1057(18)	1.0628(4)	0.0331
C14	0.2313(13)	−0.1024(16)	1.0114(3)	0.0375
N1	0.5123(13)	−0.1728(15)	1.1186(4)	0.0379
C15	0.4356(20)	−0.1361(29)	1.1672(5)	0.0730
C16	0.5326(22)	−0.1587(29)	1.2177(7)	0.0852

^a $U_{\text{eq}} = 1/3[U_{11} + U_{22} + U_{33}]$.

Some unusual anisotropic temperature parameters among carbon atoms are illustrated in Fig. 5. Those of C(7), C(8), C(15) and C(16) can be attributed to the librations of terminal ethyl groups. Those in the cyclohexane ring, C(1)–C(6), suggest some slight disorder in the ring or libration about the Te–C(1) bond. A TLS calculation for the ethoxycyclohexyl fragment (Fig. 5(a)) suggests a libration ($\pm 4.8^\circ$) about the axis with the libration extending from C(8) to C(6). The temperature parameters in the phenyl ring are less easy to understand. A TLS calculation shows a libration

TABLE 6. Interatomic distances (Å) and angles (°) with e.s.d.s in parentheses. Libration corrected distances are in square brackets

Te(1)–Br(1)	2.683(2)	Te(1)–Br(2)	2.662(2)
Te(1)–C(1)	2.18(2)	Te(1)–C(9)	2.12(1)
C(1)–C(2)	1.53(3) [1.548]	C(2)–C(3)	1.50(3) [1.525]
C(3)–C(4)	1.48(3) [1.484]	C(4)–C(5)	1.56(3) [1.577]
C(5)–C(6)	1.57(3) [1.595]	C(1)–C(6)	1.46(3) [1.469]
C(2)–O(1)	1.43(2) [1.435]	O(1)–C(7)	1.44(2) [1.453]
C(7)–C(8)	1.54(3) [1.559]	C(9)–C(10)	1.39(2) [1.401]
C(10)–C(11)	1.38(2) [1.387]	C(11)–C(12)	1.39(2) [1.407]
C(12)–C(13)	1.40(2) [1.409]	C(13)–C(14)	1.39(2) [1.388]
C(9)–C(14)	1.39(2) [1.407]	C(12)–N(1)	1.44(2) [1.445]
N(1)–C(15)	1.46(2) [1.477]	C(15)–C(16)	1.53(3) [1.533]
Br(1)–Te(1)–Br(2)	177.0(1)	Br(1)–Te(1)–C(1)	88.3(5)
Br(1)–Te(1)–C(9)	91.9(4)	Br(2)–Te(1)–C(1)	90.1(5)
Br(2)–Te(1)–C(9)	90.9(4)	C(1)–Te(1)–C(9)	100.0(6)
Te(1)–C(1)–C(2)	108(1)	Te(1)–C(1)–C(6)	117(1)
C(2)–C(1)–C(6)	115(2)	C(1)–C(2)–C(3)	112(2)
C(2)–C(3)–C(4)	112(2)	C(3)–C(4)–C(5)	114(2)
C(4)–C(5)–C(6)	112(2)	C(1)–C(6)–C(5)	110(2)
C(1)–C(2)–O(1)	107(1)	C(3)–C(2)–O(1)	112(2)
C(2)–O(1)–C(7)	114(1)	O(1)–C(7)–C(8)	113(2)
Te(1)–C(9)–C(10)	118(1)	Te(1)–C(9)–C(14)	121(1)
C(10)–C(9)–C(14)	121(1)	C(9)–C(10)–C(11)	118(1)
C(10)–C(11)–C(12)	120(1)	C(11)–C(12)–C(13)	123(1)
C(12)–C(13)–C(14)	116(1)	C(11)–C(12)–N(1)	116(1)
C(13)–C(12)–N(1)	121(1)	C(9)–C(14)–C(13)	122(1)
C(12)–N(1)–C(15)	118(1)	N(1)–C(15)–C(16)	111(2)

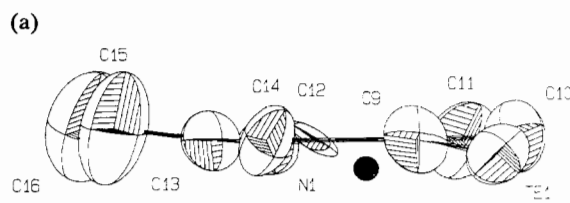
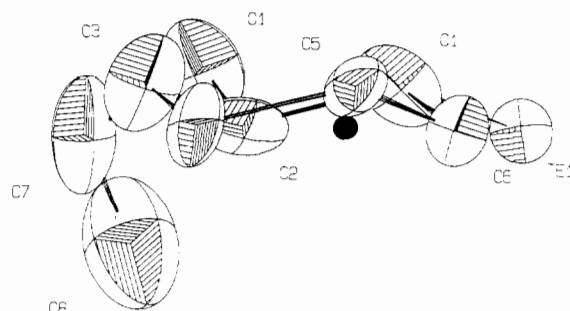


Fig. 5. Projections calculated along the major axes of TLS libration shown as black circles for (a) ethoxycyclohexyl ($\pm 4.8^\circ$) and (b) ethylanilino ($\pm 4.4^\circ$).

($\pm 4.4^\circ$) extending from N-ethyl to phenyl and through the plane of the ring to give a ripple effect (Fig. 5(b)). The librational corrections based on this model increase the average C–C distances by 0.014 Å for the cyclohexyl ring and by 0.009 Å for the phenyl ring. The r.m.s. discrepancies are 0.0143 and 0.0102 for ethoxycyclohexyl and ethylanilino fragments, respectively.

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References

- 1 M. M. Campos and N. Petragnani, *Tetrahedron Lett.*, 6 (1959) 11; *Tetrahedron*, 18 (1962) 521.
- 2 T. S. Cameron, R. B. Amero and R. E. Cordes, *Cryst. Struct. Commun.*, 9 (1980) 533.
- 3 T. S. Cameron, R. B. Amero, C. Chan and R. E. Cordes, *Cryst. Struct. Commun.*, 9 (1980) 543.
- 4 S. Uemura, S.-I. Fukuzawa and A. Toshimitsu, *J. Organomet. Chem.*, 250 (1983) 203.
- 5 M. E. S. Ali, M. A. Malik and B. C. Smith, *Inorg. Chim. Acta*, 162 (1989) 157.
- 6 A. K. Gupta, B. L. Khandelwal and K. Raina, *J. Inorg. Nuclear Chem.*, 39 (1977) 162.
- 7 T. S. Cameron and R. E. Cordes, *Acta Crystallogr., Sect. B*, 35 (1979) 749.
- 8 N. Walker and D. Stuart, *Acta Crystallogr.*, 39, Sect. A, (1983) 158.
- 9 *International Tables for X-ray Crystallography*, Vol. IV, Kynoch, Birmingham, UK, 1974.
- 10 G. M. Sheldrick, SHELXS-86, in G. M. Sheldrick, C. Kruger and R. Goddard (eds.), *Crystallographic Computing*, Oxford University Press, Oxford, 1985, pp. 175–189.

- 11 G. M. Sheldrick, SHELX-76, program for crystal structure determination, University of Cambridge, Cambridge, UK, 1976.
- 12 E. K. Davies, *CHEMGRAF Suite*, Chemical Crystallography Laboratory, Oxford, UK, 1983.
- 13 J. R. Carruthers and D. L. Watkins, *Crystals*, Issue 8, Chemical Crystallography Laboratory, Oxford, UK, 1984, revised 1989.
- 14 F. J. Berry, E. H. Kustan, M. Rowshani and B. C. Smith, *J. Organomet. Chem.*, *99* (1975) 115.
- 15 M. A. Malik, M. E. S. Ali, F. J. Berry, J. Kaur, M. Rowshani and B. C. Smith, *Inorg. Chim. Acta*, *180* (1991) 251.
- 16 T. S. Cameron, R. B. Amero and R. E. Cordes, *Cryst. Struct. Commun.*, *9* (1980) 539; N. W. Alcock and W. D. Harrison, *J. Chem. Soc., Dalton Trans.*, (1982) 251; S. Husebye, E. A. Meyers, R. A. Zingaro, J. V. Comasseto and N. Petragani, *Acta Crystallogr., Sect. C*, *43* (1987) 1147.