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SYNTHESIS, CHARACTERISATION AND CRYSTAL STRUCTURE OF POLYMERIC CHLORO(DIETHYL TELLURIDE)COPPER(I), $(Et_2Te \cdot CuCl)_\infty$

RAJ K. CHADHA * and JOHN E. DRAKE

Department of Chemistry, University of Windsor, Windsor, Ontario N9B 3P4 (Canada)

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Summary

Diethyl telluride reacts with both copper(I) and copper(II) chloride to give polymeric chloro(diethyl telluride)copper(I) which is characterised by elemental analysis and by its IR and ^{1}H NMR spectra. X-ray analysis shows that its structure contains infinite sheets in which two Et_2Te ligands are bridged between two di-μ-chlorodicopper(I) cores. The Cu–Te distances are 2.535(1) and 2.625(1) Å and the geometry around both copper and tellurium atoms is that of a distorted tetrahedron. The angles around copper range from 95.5(1) to 122.6(1)° and around tellurium from 95.6(1) to 130.5(1)°. The crystals are monoclinic, $C2/c$ with a 19.761(11), b 7.114(4), c 11.760(7) Å, β 111.17(1)°, V 1542(2) Å^3, ρ_{calcd} 2.45, ρ_{obsd} 2.43 g/cm^3, $Z = 8$ for 1220 unique "observed" reflections and the structure refined to an R index of 0.0243.

Introduction

A large number of coordination compounds of diaryl tellurides [2–6] and dialkyl tellurides [7–11] have been prepared and their structures examined by conductivity measurements [3,5] and IR [3,5,9,11], ^{1}H NMR [10,11] and Mössbauer spectroscopy [4,5]. Crystallographic studies of only three compounds, namely $Ph_2Te \cdot HgI_2$ [2], *trans*-$Pd(SCN)_2 \cdot [Te(CH_2CH_2CH_2SiMe_3)_2]_2$ [7] and $[Pt(o\text{-}PhTeC_6H_4PPh_2)_2][Pt(SCN)_4]$ [12] have been reported. The palladium and platinum complexes are square planar but the mercury complex is tetrameric as a result of Hg–I bridges.

Although numerous studies on the structures of copper(I) salts with sulphides, amines, phosphines and arsines have been carried out [13–19], to our knowledge no crystal structure of a copper(I) salt with a selenide or telluride donor has been reported. In this paper, we therefore describe the synthesis and characterisation of a

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diethyl telluride complex of copper(I) chloride, which has an unusual layered structure, in which Cu_2Cl_2 units are held together by bridging Et_2Te ligands. This is the first reported structure of a bridging tellurium ligand, although a bridging structure for the adduct $(n\text{-}Bu_4N)_2[(Cl_3Pt)(\mu\text{-}TeMe_2)]_2$ was proposed on the basis of NMR studies [20].

Results and discussion

Synthesis and spectroscopic studies. The reaction between Et_2Te and CuCl in 1/1 or 2/1 mole ratios in a dilute HCl/EtOH medium yields $Et_2Te \cdot CuCl$ as the only product. The reaction of Et_2Te and $CuCl_2$ in absolute EtOH also gives $Et_2Te \cdot CuCl$ as an insoluble product. Evaporation of the filtrate gave a white precipitate of Et_2TeCl_2. Copper(II) is thus reduced to copper(I) during the synthesis of the adduct, while Et_2Te is oxidised to Et_2TeCl_2. Although the reduction of cupric ion in the presence of ligands containing a sulfur [16,21] or selenium [22] atom is well known, reduction by tellurium containing species is not.

The compound, $Et_2Te \cdot CuCl$, possesses an unpleasant odour resembling that of Et_2Te and is completely insoluble in non-polar solvents and only sparingly soluble in CH_3CN and DMSO. This reflects its polymeric nature. Its molar conductance in CH_3CN and its conductivity over a range of concentrations are similar to those of $[(p\text{-tol})_2Te]_2 \cdot CuCl$ where it was suggested that the behaviour was associated with the solvation process [3]. $Et_2Te \cdot CuCl$ is diamagnetic and its UV and visible spectra show bands corresponding closely to those in free Et_2Te so there is no evidence of bands other than those associated with the ligand.

The IR spectrum shows bands typical of the ethyl groups along with some additional features at 510, 502, 210, 180 and 152 cm^{-1} which have structural implications. The two bands at 510 and 502 cm^{-1} clearly correspond to the asymmetric and symmetric C–Te stretches which are usually observed in various organotellurium compounds at ca. 500 cm^{-1} [23]. A strong band at 185 cm^{-1} has been tentatively assigned to the Cu–Te stretching band in RTeCu compounds [24], which corresponds well to the band observed at 180 cm^{-1}. The CuCl stretching vibrations in a di-μ-chlorodicopper(I) group have been assigned previously to 232 and 162 cm^{-1} [25], which corresponds to the observation of bands at 210 and 152 cm^{-1}.

Description of the structure. From Fig. 1 it is apparent that CuCl is indeed present as a dimerised bridging group, with each Te atom also bridging two Cu atoms. The overall structure is a polymeric layer. The four-coordination about tellurium is greatly distorted from that of a regular tetrahedron. The CTeC bond angle of 96.4(2)° is similar to that found in most compounds containing two or more C–Te bonds regardless of the coordination about tellurium. Thus, whether tellurium is two-coordinated as in $(p\text{-tol})_2Te$ [26], three coordinated as in $R_3Te^+X^-$ [27,28], $R_2Te \cdot Pd(SCN)_2$ [7] or $Ph_2Te \cdot HgI_2$ [2] or four coordinated as in R_2TeX_2 compounds [29–33], the CTeC angle remains close to $98 \pm 3°$. This suggests that it may be the optimum requirements of the bonding orbitals of carbon and tellurium that determine the angle rather than any stereochemical and/or lone-pair effects.

The Te–C distances of 2.145(5) and 2.161(5) Å agree well with the reported range of 2.12–2.18 Å in organotellurium compounds [2,7,26–33]. The telluride ligands bound to copper atoms direct their ethyl groups away from each other and from the CuTe layers (Fig. 2) and this is the outstanding feature of the unit cell packing. This

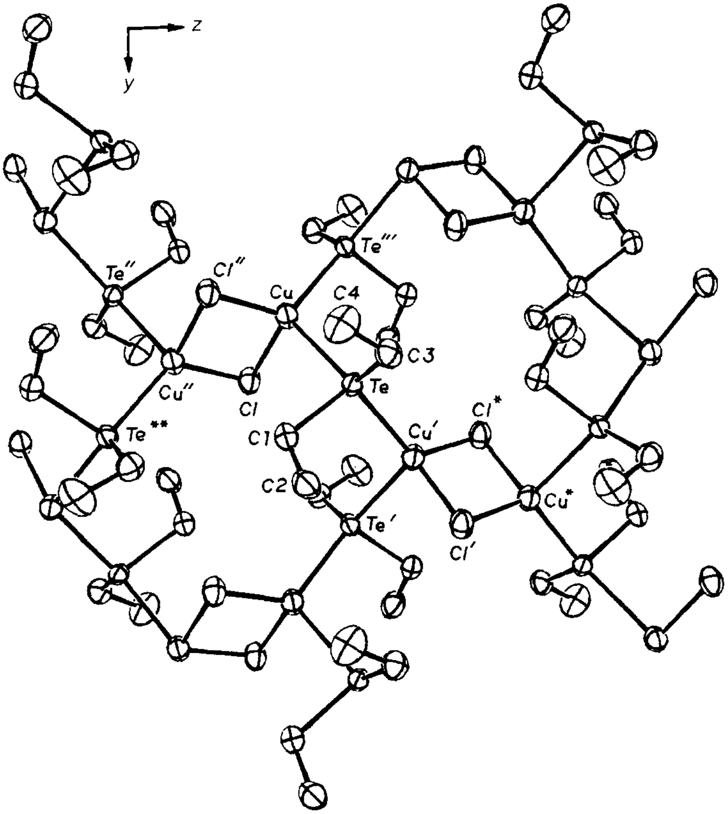

Fig. 1. ORTEP drawing showing a polymeric sheet of $Et_2Te \cdot CuCl$ down the *x* axis. The atoms are drawn with 50% probability ellipsoids. Hydrogen atoms are omitted for clarity. For symmetry related positions, see footnotes to Tables 1 and 2.

leaves no room for the layers of CuTe to interact with one another and results in the formation of sheets perpendicular to the *a* axis. The CuTeCu′, 130.5(1)°, angle is greater than the tetrahedral angle as is to be expected from the relatively small CTeC angle. However, this large value suggests that the opening up could also be related to the steric requirements of the two Cu_2Cl_2 units around each of the Te atoms.

The center of each CuCl dimer is a crystallographic centre of symmetry which requires the Cu_2Cl_2 unit to be planar. However, this unit is "lozenge" shaped with alternate short and long Cu–Cl bridging distances (2.321(1) and 2.394(1) (Å) and with obtuse angles at copper (100.0(1)°) and acute angles at chlorine (80.0(1)°). Similar differences were found in other dimeric copper(I) complexes, such as $[(Me_2PhAs)_2CuCl]_2$ [18], $[(cHx)_3P \cdot CuCl]_2$ [23], Cu(s-thiourea)$_3$BF$_4$ [17] and Cu(s-Me$_2$thiourea)$_3$BF$_4$ [17]. Alternate Cu_2Cl_2 units are parallel to one another, whereas adjacent units are almost perpendicular (dihedral angle, 97.2(1)°). The four tellurium atoms surrounding the Cu_2Cl_2 plane also form a perfect plane with their center coinciding with the center of the Cu_2Cl_2 unit. These two planes are approximately perpendicular to one another; the dihedral angle is 84.6(1)°.

Each copper atom in the dimer is four coordinate with distorted tetrahedral geometry. The dihedral angle between planes defined by atoms TeCuTe‴ and

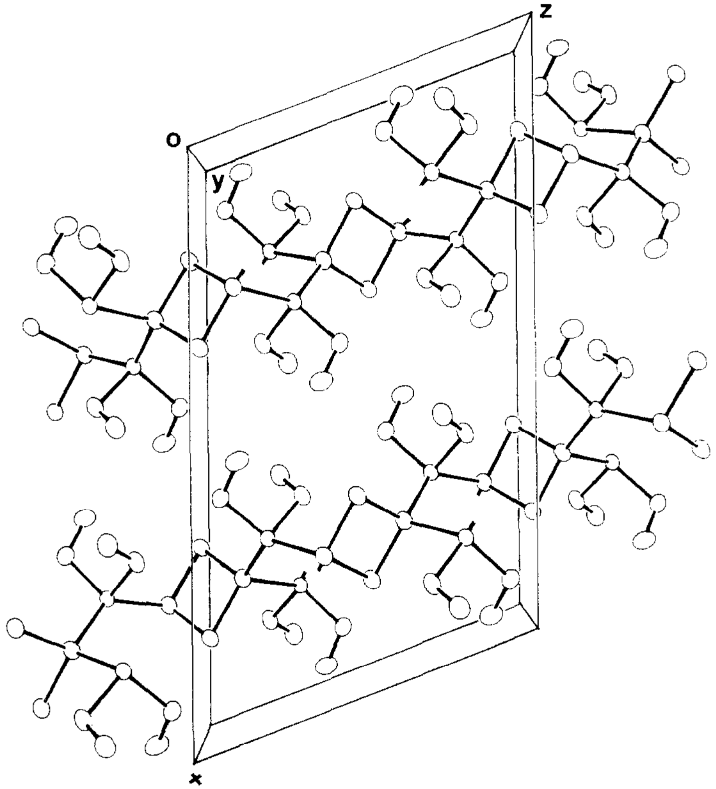

Fig. 2. Unit cell packing of the infinite layers of $Et_2Te \cdot CuCl$.

ClCuCl″ is 83.4(1)° compared to the value of 90° for a regular tetrahedron. The direction of the twist minimises non-bonded intermolecular interactions. Only one of the TeCuCl angles is close to tetrahedral, the other three fall into two categories; greater (122.6(1) and 118.2(1)°) and smaller (95.5(1)°) than 109.5°. The TeCuTe angle of 107.5(1)° is considerably smaller than the PCuP angles of 120–140° in the copper(I)-tertiary phosphine adducts [15], possibly reflecting the lesser space requirements of the two ethyl groups on tellurium.

The Cu–Te distances of 2.535(1) and 2.625(1) Å are only slightly shorter than the sum of the covalent radii (1.35 Å for tetrahedral copper(I) and 1.32 Å for tetrahedral tellurium) [34] so that coordination can involve little or no Cu–Te π-bonding. Similar conclusions were drawn for Cu–P/As [18] and Pd–Se/Te [7,12,35] complexes.

The chlorine atoms of the Cu_2Cl_2 group are close enough to the tellurium atoms (3.718(1) Å compared to the sum of the Van der Waals radii of 4.0 Å) [34] to suggest the possibility of secondary interactions [29,30].

Experimental

Starting materials. Et_2Te was prepared according to the method of Tschugaeff and Chlopin [36], and Et_2TeCl_2 by its chlorination in CCl_4. Anhydrous $CuCl_2$ was prepared by refluxing the hydrated salt with $SOCl_2$. Commercial CuCl was treated with sulfurous acid before use.

Preparation of adduct. (a) The addition product was prepared by mixing a solution of CuCl (2 g, 0.02 mol) in dilute HCl with a solution of Et_2Te (7.5 g, 0.04 mol) in 95% EtOH. A dull white precipitate of the product separated out, it was filtered, washed with EtOH and dried in vacuo. Additional amounts of the compound separated from the filtrate on keeping it overnight. This gave crystals suitable for X-ray analysis. (4.3 g; total yield 75%), m.p. 82–83°C. Anal. Found: C, 16.44; H, 3.88; Cl, 12.69; Te, 44.81; Cu, 21.78; $C_4H_{10}TeCuCl$ calcd.: C, 16.87; H, 3.51; Cl, 12.47; Te, 44.83, Cu, 22.31%. IR (cm^{-1}): 510s, 502s, 288w, 271m, 210s, 180s, 152m, 135w, 86s, 75w, 54m. ^1H NMR (DMSO-d_6): δ 1.40 (t, 6H, CH_3), 2.48 (q, 4H, CH_2), $J(CH_2\text{-}CH_3) \approx 8$ Hz. UV and UV-Vis (nm (ϵ)): 212 (1.4×10^4), 224 (1.1×10^6), 364 (2.9×10^3). Molar conductance values in CH_3CN (Ω^{-1} mol^{-1} cm^2, (mmol l^{-1})): 58.5 (11.0), 57.6 (16.0), 56.7 (20.2), 55.7 (27.0), 55.0 (60), 54.2 (102.0), 53.9 (174.2).

(b) Et_2Te (4.2 g, 0.023 mol) in absolute EtOH was added dropwise to the chilled $CuCl_2$ (2 g, 0.015 mol) in the same solvent. The solution was stirred for 6 h. The solid product on separating was filtered, washed well with EtOH and finally with ether and dried in vacuum. The compound was found to be identical with the compound prepared earlier from CuCl and Et_2Te by comparison of their melting points and spectra.

Evaporation of solvent from the filtrate gave a white precipitate, which was identified as Et_2TeCl_2, m.p. 112–114°C. IR (cm^{-1}): 502s(ν(Te–C)), 444m, 407m, 385w, 358s, 339w, 302w, 280s (ν_s(Te–Cl)), 265w(ν_{as})Te–Cl), 252m, 227w.

Physical measurements. IR spectra were recorded in Nujol mulls with a Perkin–Elmer 180 spectrophotometer using CsI and polythene disks. ^1H NMR spectra were obtained at 60 MHz using a Varian A-60 instrument. The solution spectra of Et_2Te and its adduct with CuCl in absolute ethanol were scanned over a 12,500–50,000 cm^{-1} range in 1 cm matched quartz cell on a Specord UV & VIS spectrophotometer. The Gouy method was used for measuring the magnetic susceptibilities of the adducts at room temperature. Carbon and hydrogen microanalyses were performed by Sh. L.K. Khullar of Panjab University. Te, Cl and Cu were determined using standard gravimetric techniques.

X-Ray crystallographic analysis

A crystal of dimensions 0.15 × 0.23 × 0.27 mm was sealed in a thin walled glass capillary tube and mounted and aligned on a Syntex $P2_1$ automated diffractometer with graphite-monochromatised Mo-K_α radiation (λ 0.71069 Å). The unit cell dimensions were calculated from the setting angles of 15 reflections having $15° < 2\theta < 30°$. The lattice constants are a 19.761(11), b 7.114(4), c 11.760(7) Å, β 111.17(1)°, V 1542(2)Å^3. For $Z = 8$, the calculated density is 2.45 g cm^{-3}; the density measured by flotation in CCl_4/CH_2I_2 is 2.43 g cm^{-3}. The intensity data were measured by using a θ–2θ scan mode with a variable scan speed of 2 to 5°/min. The intensities of 3 check reflections monitored every 60 reflections changed only by 1% during data collection. Of the 1610 reflections measured ($4° < 2\theta < 50°$), 1220 independent reflections had $I > 3\sigma(I)$ and only these were used in subsequent calculations. The intensities were corrected for Lorentz and polarisation effects and for absorption (μ 64.45 cm^{-1}); the absorption correction factors ranged between 2.42 and 4.24. Systematic absences (hkl, $h + k = 2n + 1$; $h0l$, $l = 2n + 1$) indicate the space groups $C2/c$ or Cc. The former was used and later assumed correct because of successful refinement of the structure.

TABLE 1

FINAL FRACTIONAL COORDINATES AND THERMAL PARAMETERS[a] ($Å^2 \times 10^3$) FOR NON-HYDROGEN ATOMS OF $Et_2Te \cdot CuCl$ WITH STANDARD DEVIATIONS IN PARENTHESES

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Te	0.30841(1)	0.42000(4)	0.29367(2)	34.2(2)	33.8(2)	26.9(2)	−2.3(1)	11.8(1)	−4.3(1)
Cu	0.24928(3)	0.17367(9)	0.11989(5)	43.3(3)	44.1(4)	33.7(3)	−2.9(2)	16.6(3)	1.2(2)
Cl	0.3289(1)	0.1060(2)	0.0208(1)	44(1)	53(1)	35(1)	15(0)	18(0)	7(0)
C(1)	0.3560(3)	0.5888(6)	0.1911(4)	56(3)	42(3)	34(2)	−4(2)	21(2)	1(2)
C(2)	0.4044(3)	0.7420(8)	0.2639(4)	63(3)	51(3)	45(3)	−13(3)	21(2)	6(2)
C(3)	0.4094(2)	0.3272(7)	0.4296(4)	41(3)	46(3)	35(2)	1(2)	8(2)	3(2)
C(4)	0.4610(3)	0.2395(10)	0.3760(5)	39(3)	77(4)	63(3)	15(3)	13(2)	−9(3)

[a] Anisotropic temperature factors are of the form $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

TABLE 2

INTERATOMIC DISTANCES (Å) AND ANGLES (°) [a]

Te–Cu	2.625(1)	Cu–Te–Cu′	130.5(1)
Te–Cu′	2.535(1)	Cu–Te–C(1)	95.6(1)
Cu–Cl	2.394(1)	Cu′–Te–C(1)	100.3(1)
Cu–Cl″	2.321(1)	Cu–Te–C(3)	113.9(1)
Te–C(1)	2.145(5)	Cu′–Te–C(3)	110.5(1)
Te–C(3)	2.161(5)	C(1)–Te–C(3)	96.4(2)
C(1)–C(2)	1.499(7)	Te–Cu–Te‴	107.5(1)
C(3)–C(4)	1.514(7)	Te–Cu–Cl	95.5(1)
Cu...Cu″	3.031(1)	Te‴–Cu–Cl	118.2(1)
Cl...Cl″	3.612(1)	Te–Cu–Cl″	109.7(1)
Te...Cl	3.718(1)	Te‴–Cu–Cl″	122.6(1)
Te...Te‴	4.162(1)	Cl–Cu–Cl″	100.0(1)
		Cu–Cl–Cu″	80.0(1)
		Te–C(1)–C(2)	113.9(4)
		Te–C(3)–C(4)	113.5(4)

[a] ′ = $0.5-x, 0.5+y, 0.5-z$; ″ = $0.5-x, 0.5-y, -z$; ‴ = $0.5-x, y-0.5, 0.5-z$.

Positions of the tellurium and copper atoms were determined from a sharpened Patterson synthesis using SHELX. The remaining non-hydrogen atoms were located by successive difference Fourier syntheses. Complete anisotropic refinement minimising the function $w(|F_0|-|F_c|)^2$ converged at $R=\Sigma||F_0|-|F_c||/\Sigma|F_0|=0.0243$ and $R_{wF}=[\Sigma w(|F_0|-|F_c|)^2/\Sigma wF_0^2]^{1/2}=0.0277$ based on 64 variables. Hydrogen atoms were included at the idealised positions (C–H 0.95 Å, angle HCH 109.5°) with isotropic thermal parameters set at 0.1 Å^2 greater than that of the corresponding carbon. A weighting scheme of the form $w=1/[\sigma^2(F)+\rho F^2]$ was employed with a final ρ value of 0.001. In the last cycle no parameter changed by more than 0.05. The largest peak in the final difference map is 0.8 e/Å^3.

Sources of scattering factors and computer programs employed have been given elsewhere [29]. The final atomic coordinates and anisotropic thermal parameters for non-hydrogen atoms are given in Table 1 and important distances and angles in Table 2 and equations of mean planes in Table 3. Tables of hydrogen atom coordinates and isotropic thermal parameters, and observed and calculated structure factors (9 pages) may be obtained from the authors.

TABLE 3

EQUATIONS OF PLANES EXPRESSED WITH REFERENCE TO THE CELL AXES, AND DIHEDRAL ANGLES (°) [a]

Plane (1): Cu, Cl, Cu″, Cl″: $0.5930X+0.7499Y+0.2933Z=3.5421$
Plane (2): Cu′, Cl′ Cu★ Cl★: $0.5930X-0.7499Y+0.2933Z=-0.0679$
Plane (3): Te, Te‴, Te″, Te★★: $0.8537X-0.5113Y-0.0990Z=3.2002$
Plane (4): Te, Cu, Te‴ : $0.8558X-0.5170Y-0.0163Z=3.2984$
Dihedral angles between planes (1) and (2) 97.2(1)
(1) and (3) 84.6(1)
(1) and (4) 83.4(1)

[a] For symmetry related positions ′, ″ and ‴, see footnote of Table 2; ★ is $x, 1-y, 0.5+z$; ★★ is $x, 1-y, -0.5+z$.

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