SHORT PAPER

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The first metal complexes of 1,4-oxatellurane: synthesis and crystal structure of its platinum(II) complex[†]

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The palladium(II) and platinum(II) complexes of 1,4-oxatellurane (**L**), having the composition [MCl_2**L_2**], are synthesized and characterized spectroscopically and structurally (Pt complex only; Pt–Cl 2.3169(9) Å; Pt–Te 2.5945(3) Å) for the first time.

There are few known transition metal complexes[1,2] of Te heterocycles. The [$PdCl_2$(tellurocyclpentane)$_2$] is the first complex of tellurocyclopentane to be characterized structurally[4]. McWhinnie and his coworkers[4,5] have studied the reactions of Te heterocycles with $Fe_3(CO)_{12}$ and [{Rh(η^5-C_5Me_5)Cl_2}] very recently. The detelluration of Te-heterocycles is a common feature of these reactions. The complexes containing the heterocycles intact have also been isolated. The 1,4-oxatellurane[6] has been known for more than 50 years but its metal complexes have never been characterized structurally whereas 1,4-dioxane is well established as a ligand. In this paper the first synthesis and crystal structure of *trans*-[$PtCl_2$(1,4-oxatellurane)$_2$] (**1**) are reported along with the synthesis and spectral data of its palladium(II) analogue, which is also synthesized for the first time but does not give crystals of good quality. The molecular structure of **1** is shown in Fig. 1 and is centrosymmetric with Pt at the centre of symmetry. The Pt has square planar geometry. The Pt-Te bond length of **1** (Table 1) is similar to the average bond length of 2.582 Å found in *trans*-diiodobis(methylphenyltelluride)platinum(II)[7a] . In two other Pt(II) complexes with linear Te–Pt–Te sequence, the bis[o-diphenylphosphine)tellurophenyl)phenyl-P,Te]platinum(II) cation[7b] and the chlorobis{[2-(4-ethoxyphenyl)ethyltelluride]amino-Te,Te′,N}platinum(II) cation[7c] the tellurium ligands are bi and tridentate and there the Pt–Te bonds are slightly shorter, 2.575(1) and 2.561 (average) Å respectively. This may be due to stabilizing effect of chelation. Again the *trans* influence is manifested when a tellurium ligand is *trans* to another ligand. *Trans* to Cl the Pt-Te bond length is close to 2.52 Å[8a,b], while *trans* to P it is 2.553 Å in the 5,5-bis(dimethylphenylphosphine-2-thia-4-tellura-1,3-diazolium)platinate(II) anion[8c]. *Trans* to carbon (phenyl) it was even found to be 2.693(2) Å,[8d] reflecting the strong *trans* influence of aryl groups. The Pt-Cl bond length is close to the statistical value[8e] of 2.323(38) Å found for terminally bonded chloride in four coordinate platinum compounds. The Te–C bond lengths are quite close to the standard statistical values of 2.158(30) Å for a Te–C(sp^3) bond and 2.116(20) for a Te–C(aryl) bond[8f] .

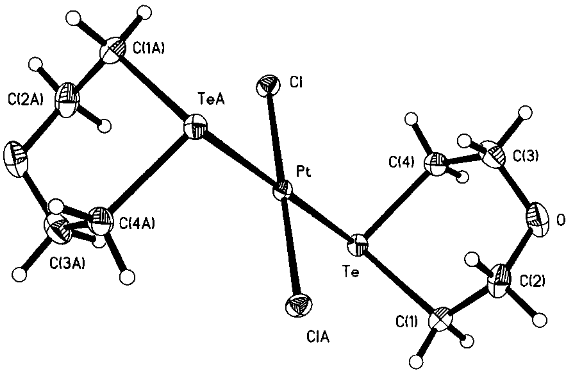


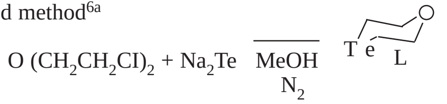
Fig 1 Molecular structure of *trans*-[$PtCl_2$(1,4-oxatellurane)$_2$] (**1**)

Table 1 Selected bond lengths (Å) and angles (°)

	Bond length (Å)		
Pt–Te	2.5945 (3)	Pt–Cl	2.3169 (9)
Te–C(1)	2.152 (4)	Te–C (4)	2.152 (4)
	Bond angles (°)		
Te–Pt–Te(A)	180	Cl–Pt–Cl (A)	180
Te–Pt–Cl	87.11 (3)	Te–Pt–Cl (A)	92.89 (3)
Pt–Te–C(1)	104.27 (12)	Pt–Te–C (4)	100.56 (11)
C(1)–Te–C(4)	91.1 (2)		

Experimental

1,4-Oxatellurane (viscous orange oil) was synthesized by the published method[6a]



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(equation 1). ^1H and $^{13}C\{^1H\}$ NMR spectra were recorded on a Bruker Spectrospin DPX 300 NMR spectrometer at 300.1316507 MHz. The C, H and N analyses were carried out on Perkin Elmer elemental analyser 240 C. Tellurium was estimated titrimetrically[6b]. The synthesis of Pt/Pd(II) complex was carried out by the procedure described below.

Synthesis of [$PtCl_2$**L_2**] (**1**): Potassium tetrachloroplatinate(II) (0.2 g, 0.5 mmol) dissolved in 10 ml of water was mixed with a solution of **L** (0.19 g 1.0 mmol) made in 10 ml of acetone. The mixture was stirred for 2 h and poured into 100 ml of water. The complex **1** was extracted from aqueous layer into 60–70 ml of CH_2Cl_2. The extract was washed with water, dried over anhydrous Na_2SO_4 and concentrated to ~ 10 ml on a rotary evaporator. The concentrated was layered with 5–7 ml of petroleum ether (40–60 °C). The orange yellow needles shaped crystals were grown. Yield 69 % m.p. 162 °C (d). Analyses: Found: C, 14.75; H, 2.61; Te, 37.45 %; $C_8H_{16}Te_2O_2PtCl_2$ requires C, 14.44, H, 2.41; Te, 38.36 %. NMR (^1H, $CDCl_3$, 25 °C) δ, 2.80–2.94 (m, 8H, CH_2Te), 4.13–4.40 (m, 8H, OCH_2); ($^{13}C\{^1H\}$, $CDCl_3$, 25 °C) δ, 5.47 (CH_2Te), 68.81 (OCH_2).

Synthesis of [$PdCl_2$**L_2**] (**2**): The **L** (0.27 g, 1.4 mmol) and Na_2PdCl_4 (0.2 g 0.7 mmol) were reacted as described above for the platinum complex. The yield of red-orange crystals of **2** was 79 % and m.p. 145 °C (d). Analyses: Found: C, 16.91; H, 3.01; Te, 43.93

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[†] This is a Short Crystallographic Paper, there is therefore no corresponding material in *J Chem. Research (M).*

%; $C_8H_{16}Te_2O_2PdCl_2$ requires C, 16.66, H, 2.77; Te, 44.26 %. NMR (^1H, $CDCl_3$, 25 °C) δ, 2.89-2.92 (m, 8H, CH_2Te), 4.29-4.32 (m, 8H, OCH_2); ($^{13}C\{^1H\}$, $CDCl_3$, 25 °C) δ, 6.79 (CH_2Te), 69.26 (OCH_2).

X-ray analysis

The X-ray data were collected on a Bruker-AXS SMART2K CCD diffractometer, using graphite monochromated Mo–Kα radiation ($\lambda = 0.71073$ Å). In excess of a full sphere of reflections were collected. The data were reduced and the structures solved using the programs SMART, SAINT and SHELXTL.[9a,b] All non-hydrogen atoms were refined anisotropically and the hydrogen atoms were given a isotropic displacement factor equal to 1.5 times the equivalent isotropic displacement factor of the parent carbon atom. The crystal data are as follows.

Crystal data: Chemical formula: $C_8H_{16}Cl_2O_2Te_2Pt$; formula weight: 665.40, temperature: 123(2) K; crystal system: monoclinic; space group: C2/c; μ (mm^{-1}) 14.45; unit cell dimension: $a = 16.6455(8)$ Å, $b = 9.9.1933(4)$ Å, $c = 9.8301(4)$ Å; $\alpha = 90°$ $\beta = 11.229(3)°$, $\gamma = 90°$; volume: 1402.19(11) $Å^3$; z = 4; reflections collected 12238; independent reflections 2515 (Rint 0.0328); Final R indices [$I > 2\sigma(I)$]: R1 = 0.0271 wR2 = 0..0578.

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