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N-[2-(4-methoxyphenyltelluro)ethyl]phthalimide (L^1): synthesis, oxidation by ruthenium(III) chloride and ligation with ruthenium(II). Crystal structures of L^1, its oxidized product and of [$RuCl_2$(*p*-cymene)·L^1]

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Abstract

N-[2-(4-methoxyphenyltelluro)ethyl]phthalimide (L^1) synthesized by reacting $ArTe^-$ (generated in situ) with *N*-(2-bromoethyl)phthalimide, has been characterized structurally. The Te–C(alkyl) (2.147(5) Å) is somewhat longer than Te–C(aryl) (2.111(4) Å). The L^1 on reaction with $RuCl_3 \cdot xH_2O$ results in a novel heterocycle, *Te*–chloro,*Te*–anisyl-1a-aza-4-oxa-3-tellura-1*H*,2*H*,4*aH*-9-fluorenone (**1**), which is characterized structurally and is a unique example of a tellura heterocycle containing oxygen as well as nitrogen in the same ring. The oxidation state of Te changes to + IV in the formation of **1**. The Te–Cl and Te–O bond lengths in **1** are 2.604(2) and 2.038(4) Å, respectively. The reaction of L^1 with [$RuCl_2$(*p*-cymene)]$_2$ gives [$RuCl_2$(*p*-cymene)·L^1] (**2**) which is characterized structurally. The Ru–C (av), Ru–Cl and Ru–Te bond lengths are 2.192(1), 2.417(1)–2.434(1) and 2.651(5) Å, respectively. The difference in Te–C(alkyl) and Te–C(aryl) bond lengths is not affected on the ligation of L^1 with ruthenium(II). © 2000 Elsevier Science S.A. All rights reserved.

Keywords: *N*-[2-(4-methoxyphenyltelluro)ethyl]phthalimide; *Te*–chloro,*Te*–anisyl-1a-aza-4-oxa-3-tellura-1*H*,2*H*,4*aH*-9-fluorenone; Ruthenium(III); Ruthenium(II); Complex; Crystal structure

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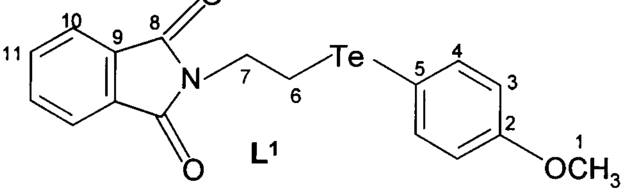
1. Introduction

The chemistry [1–7] of organotellurium ligands including hybrid ones is of some current interest. The main reason for interest in the hybrid ones is the fact that they can make us understand better the ligation of noblest metalloid tellurium vis a vis other very well known donor sites. *N*-[2-(4-methoxyphenyltelluro)ethyl]phthalimide (L^1) is one such ligand, which may behave as a (Te, N) type of donor [8] easily in mononuclear complexes. Its palladium(II) complexes are reported [8] but characterized only spectroscopically. Crystal structure of L^1 has also not been reported so far. In continuation of our earlier studies [8] on L^1 and its palladium complexes, we have now solved the reported single crystal structure of L^1 so that its ligation characteristics are understood better, and synthesized Ru(II) complex, [Ru(*p*-cymene)Cl_2(L^1)], which is also structurally characterized. The reaction of L^1 with $RuCl_3 \cdot xH_2O$ results in its oxidation, giving a Te, O and N containing novel heterocycle, *Te*–chloro,*Te*–anisyl-1a-aza-4-oxa-3-tellura-1*H*,2*H*,4*aH*-9-fluorenone, characterized by crystal structure determination. These results are the subject of this paper.



L^1

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2. Experimental

The C and H analyses were carried out with a Perkin–Elmer elemental analyzer 240 C. The ^{1}H- and ^{13}C{^{1}H}-NMR spectra were recorded on a Bruker Spectrospin DPX-300 NMR spectrometer at 300.13 and 75.47 MHz, respectively. IR spectra in the range 4000–250 cm^{-1} were recorded on a Nicolet Protége 460 FTIR spectrometer as KBr or CsI pellets. Molecular weights were determined in chloroform using a Knauer vapour pressure osmometer model A028 at a concentration ∼1 mM. The conductance measurements were made using an ORION conductivity meter model 162. The melting points determined in open capillary are reported as such. Bis(4-methoxyphenyl)ditelluride was prepared by the published method [9]. *N*-(2-bromoethyl)phthalimide was used as received from Aldrich (USA). Tellurium estimation was made by a standard method [10].

2.1. Synthesis of *N*-[2-(4-methoxyphenyltelluro)ethyl]phthalimide (**L^1**)

The published method [8] was used after some modifications. It is detailed below.

Bis(4-methoxyphenyl)ditelluride (1 g, 2.13 mmol) was refluxed in methanol (50 ml) under nitrogen atmosphere. Sodium borohydride (0.2 g in 2 ml of 1 M NaOH) was added drop wise until the solution became colourless. A solution of *N*-(2-bromoethyl)phthalimide (1.08 g, 4.26 mmol) made in 5 ml of THF was added and the mixture refluxed further for 30 min. It was cooled to room temperature and poured into 100 ml of water. **L^1** from the aqueous phase was extracted into 100 ml of chloroform. The extract was dried over anhydrous sodium sulphate and concentrated to ca. 20 ml on a rotary evaporator under reduced pressure. The concentrate was mixed with hexane (25–30 ml). The resulting precipitate was recrystallized with chloroform–petroleum ether mixture (1:2) to grow single crystals (yellow needle type) of **L^1**. Yield ca. 71%; m.p. 120°C.

Anal. Calc. for $C_{17}H_{15}NO_3Te$: C, 49.93; H, 3.70; N, 3.43; Te, 31.20. Found: C, 50.25; H, 3.65; N, 3.40; Te, 30.65%. ^{1}H-NMR ($CDCl_3$): δ, 3.06–3.11 (t, 2H, $TeCH_2$), 3.75 (s, 3H OCH_3), 4.05–4.09 (t, 2H, NCH_2), 6.67–6.70 (d, 2H, ArH *m* to Te), 7.60–7.80 (m, 6H, ArH *o* to Te + phthalimide ring protons). ^{13}C{^{1}H}-NMR ($CDCl_3$) δ, 5.2 (C_6), 39.9 (C_7), 55.0 (C_1), 99.4 (C_5), 115.2 (C_3), 123.2 (C_{10}), 132.1 (C_9), 133.8 (C_4), 140.8 (C_4), 159.8 (C_2), 167.9 (C_8).

2.2. Reaction of *L^1* with $RuCl_3 \cdot xH_2O$ (**1**)

Ruthenium(III) chloride hydrate (0.21 g, 1 mmol) was dissolved in methanol (25 ml). The solution of **L^1** (0.82 g, 2 mmol) made in 10 ml of chloroform was added to it dropwise. The mixture was stirred for 1 h and solvent was removed on a rotary evaporator under reduced pressure. The residue was redissolved in 10 ml of acetonitrile, filtered and layered with 20 ml of diethyl ether. The pale brown crystals appeared after three days were separated and air-dried. Yield ca. 35%; m.p. 192–194°C.

Anal. Calc. for $C_{17}H_{16}ClNO_3Te$: C, 45.85; H, 3.59; N, 3.14; Te, 28.65. Found: C, 47.16; H, 3.04; N, 2.92; Te, 29.05%. ^{1}H-NMR ($CDCl_3$): δ, 3.51–3.68 (t, 2H, $TeCH_2$), 3.91 (s, 3H OCH_3), 4.48–4.81 (t, 2H, NCH_2), 5.54 (s, 1H, CH), 7.19–7.22 (d, 2H, ArH *m* to Te), 7.46–7.60 (m, 4H, phthalimide ring protons), 8.09–8.12 (d, 2H, ArH *o* to Te). ^{13}C{^{1}H}-NMR ($CDCl_3$) δ, 37.6 (C_7), 53.8 (C_6), 55.6 (C_1), 83.8 (N–C–H), 115.7 (C_3), 123.5–135.4 (Ar–C of phthalimide ring), 143.9 (C_4), 162.2 (C_2), 168.7 (C_8).

2.3. Synthesis of [$RuCl_2$(*p*-cymene) **L^1**] (**2**)

The [$RuCl_2$(*p*-cymene)]$_2$ (0.62, 1 mmol) was dissolved in 10 ml of dichloromethane. The solution of **L^1** (0.966 g, 2 mmol) made in 20 ml of dichloromethane was added to it with vigorous stirring. The mixture was stirred further for 3 h. The solvent was removed on a rotary evaporator under reduced pressure. The residue was dissolved in acetone (5 ml) and layered with 25 ml of petroleum ether (40–60°C). The dark red microcrystalline solid was separated and dried in vacuo. The half of this solid was dissolved in 5 ml of dichloromethane and layered with 25 ml of petroleum ether (40–60°C). The dark red coloured single crystals were separated. Yield ca. 75%; m.p. 180°C. Molecular weight: Found 736.3 (Calc. 714.8)

Anal. Calc. for $C_{27}H_{29}NO_3TeRuCl_2$ C, 45.35; H, 4.06; N, 1.95; Te, 17.84. Found: C, 45.28; H, 4.59; N, 1.76; Te, 18.46%. ^{1}H-NMR ($CDCl_3$): δ, 1.25–1.27 (d, 6H, CH_3 of *i*-Pr), 2.11 (s, 3H, CH_3 *p* to *i*-Pr), 2.78–2.90 (sp, 1H, CH of *i*-Pr), 3.72 (s, 3H, OCH_3), 3.81–4.01 (m, 4H, $TeCH_2 + NCH_2$), 5.25–5.39 (m, 4H, ArH of *p*-cymene), 6.83–6.86 (d, 2H, ArH *m* to Te).7.53–7.79 (m, 4H, ArH of phthalimide ring), 7.84–7.87 (d, 2H, ArH, *o* to Te). ^{13}C{^{1}H}-NMR ($CDCl_3$) δ 16.4, 16.7 (C_6), 18.5 (CH_3 of *i*-Pr), 22.1, 22.4 (CH_3), 30.8 (CH), 36.7, 38.6 (C_7), 55.3 (C_1), 80.5, 81.0, 81.3, 81.7, 85.0, 85.5 (ring C of *p*-cymene *o* and *m* to *i*-Pr), 97.9–98.3 (ArC linked to *i*-Pr), 104.4 (C_5), 115.2, 115.4 (C_3), 122.2–123.5 (C_{10}), 131.8–132.0 (C_9), 133.8 (C_{11}), 136.8–137.2 (C_4), 143.8 (ArC linked to CH_3), 161.3 (C_2), 167.4 (C_8).

2.4. X-ray diffraction

The X-ray data were collected on an Enraf–Nonius CAD-4 automated four circle diffractometer for **L^1** and

Table 1
Crystallographic data measurements and refinements of **L**^{1}, **1** and **2**

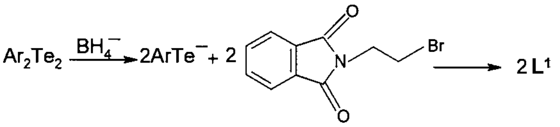
	Compound		
	L^1	**1**	**2**
Empirical formula	$C_{17}H_{15}NO_3Te$	$C_{17}H_{16}ClNO_3Te$	$C_{17}H_{15}C_{12}NO_3RuTe$
Formula weight	408.90	445.36	580.87
T (K)	293(2)	293(2)	295(2)
Crystal system	Monoclinic	Triclinic	Monoclinic
Space group	$P2_1/c$	$P\bar{1}$	$P2_1/c$
Unit cell dimensions			
a (Å)	7.116(2)	7.5497(10)	12.3942(8)
b (Å)	16.368(4)	8.0185(10)	21.7609(14)
c (Å)	13.756(3)	14.6195(10)	10.3092(7)
α (°)	90	93.369(10)	90
β (°)	99.30	92.928(10)	95.889(1)
γ (°)	90	112.961(10)	90
V (Å^3)	1581.1(6)	810.9(2)	2765.8(3)
Z	4	2	4
D_{calc} (g cm^{-3})	1.718	1.824	1.395
μ (cm^{-1})	18.93	20.13	18.05
F(000)	800.0	436	1112
Reflections collected	3017	3085	30 483
Independent reflections	2780 ($R_{int} = 0.0230$)	2848 ($R_{int} = 0.0060$)	6718 ($R_{int} = 0.0936$)
Absorption	None	None	None
Corrections			
Data/restraint/parameters	2780/0/259	2840/0/273	6718/0/351
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0324$, $wR_2 = 0.059$	$R_1 = 0.0277$, $wR_2 = 0.0932$	$R_1 = 0.0355$, $wR_2 = 0.0866$
R indices (all data)	$R_1 = 0.0613$, $wR_2 = 0.0748$	$R_1 = 0.0306$, $wR_2 = 0.1404$	$R_1 = 0.0572$, $wR_2 = 0.0950$
Goodness-of-fit on F^2	1.157	1.087	1.040
Largest difference peak and hole (e Å^{-3})	0.601 and −0.481	0.696 and −1.108	0.778 and −0.766

1 using graphite monochromated Mo–K$_\alpha$ radiation ($\lambda = 0.71073$ Å) whereas for **2** Bruker-AXS SMART2K CCD diffractometer was used. The unit cell was determined from 25 randomly selected selected reflections using the automatic search index and least-squares routine. The structure solutions were made by the programme SHELXS-86 [11] and for structure refinement SHELXL-93 programme [12] was used. Some details of data collection and refinement are given in Table 1. The full details about coordinates, bond lengths and angles, structure factors and thermal parameters may be retrieved from Cambridge crystallographic data base.

3. Results and discussion

3.1. Crystal structures

The **L**^{1} is formed by the reactions given in Eq. (1) and characterized structurally. The molecular structure of **L**^{1} as determined by single crystal X-ray diffraction is shown in Fig. 1. It may act as a (Te, N) donor easily and perhaps (Te, N, O) donor also in bimetallic complexes. However none of the bimetallic complexes of **L**^{1} synthesized by us so far, has given crystals suitable for X-ray diffraction.



(1)

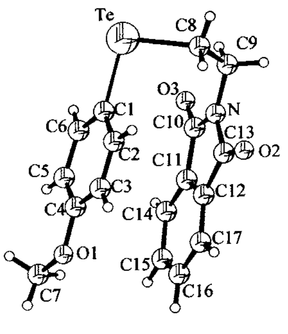
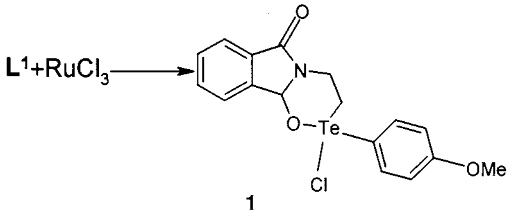


Fig. 1. Molecular structure of N-[2-(4-methoxyphenyltelluro)phthalimide (**L**^{1})

Table 2
Selected bond lengths (Å) and angles (°)

L^1		**1**		**2**	
Te–C(1)	2.111(4)	Te(1)–C(1)	2.106(5)	Ru–Cl(1)	2.417(1)
Te–C(8)	2.147(5)	Te(1)–C(8)	2.132(6)	Ru–Cl(2)	2.434(1)
C(9)–N	1.454(5)	Te(1)–O(2)	2.038(4)	Ru–C(1C)	2.211(3)
C(10)–N	1.393(5)	Te(1)–Cl(1)	2.604(2)	Ru–C(2C)	2,215(3)
C(13)–N	1.394(5)	C(13)–O(2)	1.426(7)	Ru–C(3C)	2.194(4)
C(10)–O(3)	1.208(5)	C(13)–N	1.450(8)	Ru–C(4C)	2.196(4)
C(13)–O(2)	1.213(5)	C(10)–N	1.369(8)	Ru–C(5C)	2.157(3)
C(10)–C(11)	1.485(6)	C(9)–N	1.433(8)	Ru–C(6C)	2.1849(3)
C(12)–C(13)	1.488(6)	C(10)–O(1)	1.218(8)	Ru–Te	2.651(8)
C(1)–Te–C(8)	97.4(2)	C(9)–C(8)	1.530(9)	Te–C(10)	2.156(4)
N–C(10)–O(3)	124.0(4)	C(1)–Te(1)–Cl(1)	87.9(2)	Te–C(11)	2.111(3)
N–C(13)–O(2)	124.0(4)	C(1)–Te(1)–O(2)	92.0(2)	C(9)–C(10)	1.523(5)
N–C(10)–C(11)	106.4(3)	C(1)–Te(1)–C(8)	100.0(2)	N–C(9)	1.438(5)
N–C(10)–C(13)	111.1(3)	C(8)–Te(1)–Cl(1)	82.2(2)	N–C(6)	1.353(5)
		C(8)–Te(1)–O(2)	92.4(2)	N–C(5)	1.464(5)
		O(2)–Te(1)–Cl(1)	174.4(1)	C(6)–O(2)	1.221(4)
		O(2)–C(13)–N	111.9(5)	Cl(1)–Ru–Cl(2)	88.50(4)
		C(13)–N–C(9)	120.2(5)	Cl(1)–Ru–Te	90.40(2)
		N–C(9)–C(8)	111.5(5)	Te–Ru–Cl(2)	81.31(3)
		C(9)–C(8)–Te(1)	112.1(4)	Cl(1)–Ru–C(1C)	149.75(9)
				Cl(2)–Ru–C(1C)	90.36(10)
				Te–Ru–C(1C)	119.28(10)
				Cl(1)–Ru–C(2C)	112.80(10)
				Cl(2)–Ru–C(2C)	97.04(10)
				Te–Ru–C(2C)	157.02(10)
				Ru–Te–C(10)	104.57(10)
				Ru–Te–C(11)	106.58(9)
				C(10)–Te–C(11)	94.29(15)

The selected bond lengths and angles of $\mathbf{L^1}$ are given in Table 2. The Te–C(1) is shorter than Te–C(8), as is generally reported [13] for Te–C(aryl) in comparison to Te–C(alkyl). The C(1)–Te–C(8) angle is consistent with the literature reports made earlier for a similar angle, in the case of alkyl aryl tellurides [13].



1 (2)

The $\mathbf{L^1}$ on reaction with $RuCl_3{\cdot}xH_2O$ results in **1**, in which Te is oxidized to the + IV oxidation state and a novel heterocycle ring containing N, Te, and O is generated (Eq. (2)). The structure of *Te*-chloro,*Te*-anisyl-1a-aza-4-oxa-3-tellura-*1H*,*2H*,*4aH*-9-fluorenone (**1**) established by determining its single crystal structure with X-ray diffraction is shown in Fig. 2. The geometry of Te in **1** is typically that of a Te(IV) species, which may be derived from a trigonal bipyramidal arrangement by placing at one corner of the trigonal plane a lone pair. The oxygen and chlorine atoms are *trans* to each other. The selected bond lengths and angles are given in Table 2. The C(1)–Te–C(8) angle in **1** is a little more than that of $\mathbf{L^1}$. The Te–C bond lengths are also somewhat shortened on the formation of **1**. So far no tellurium heterocycle containing three heteroatoms is known and therefore **1** is the first such example. The Te–O(2) and Te–Cl(1) bond lengths are concurrent with

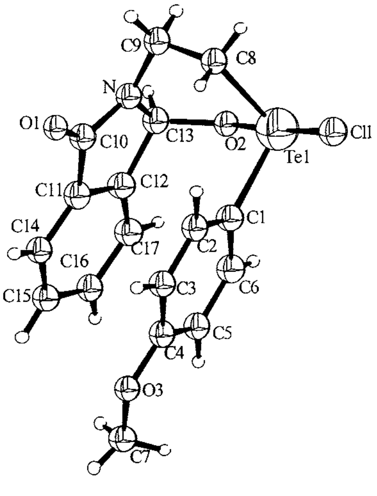


Fig. 2. Molecular structure of *Te*-chloro,*Te*-anisyl-1a-aza-4-oxa-3-tellura-1*H*,2*H*,4*aH*-9-fluorenone (**1**)

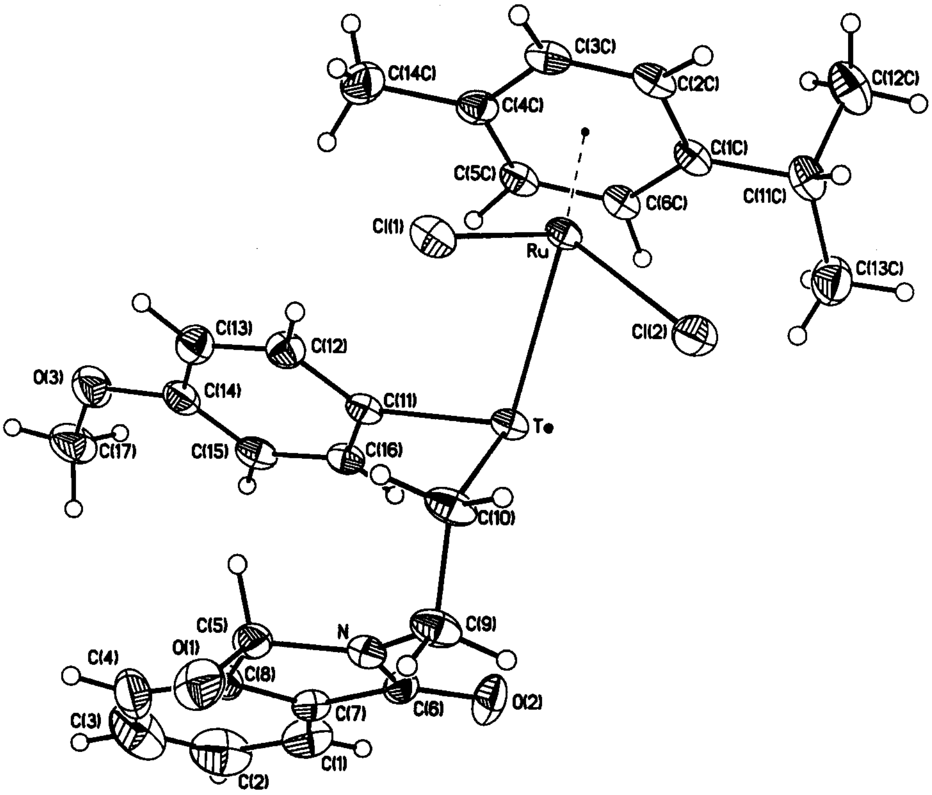


Fig. 3. Molecular structure of [RuCl$_2$(p-cymene)·**L**^{1}] (**2**)

the values 1.890(8)–2.116(8) and 2.315(5)–2.564(1) Å, respectively, reported in the literature [14,15] for these bonds. The reaction of [(p-cymene)RuCl$_2$]$_2$ with **L**^{1} results in **2**, which is also characterized structurally (Fig. 3). The coordination of **L**^{1} in **2** is through Te only. The Ru–Te and Ru–Cl bond lengths in **2** are normal, and in agreement with the literature values, 2.636(1)–2.655(1) and 2.467(4) Å [5c, 16], respectively. The half sandwich compound **2** has p-cymene in a η^6 bonding mode (Ru–C(av.) 2.192(1) Å). The Te–C(alkyl) in **2** has also been found to be longer than Te–C(aryl) as it is in **L**^{1} and **1**.

3.2. ^{1}H- and ^{13}C{^{1}H}-NMR spectra

The **L**^{1} exhibits characteristic ^{1}H- and ^{13}C-NMR spectra. The two NMR spectra of **1**, formed through oxidation of tellurium of **L**^{1}, are also characteristic. The ^{1}H-NMR spectrum of **1** has a CH signal at 5.54 ppm, and also exhibits ca. 0.4–0.5 ppm deshielded TeCH$_2$, NCH$_2$ and ArTe signals (with respect to that of free **L**^{1}), as expected due to oxidation of tellurium from +II to +IV oxidation state and cyclization. In ^{13}C-NMR spectrum of **1** also, signal due to TeCH$_2$ shows ca. 50 ppm down field shift with respect to that of free **L**^{1}, concurring well with the formation of Te(IV). The ^{1}H-NMR spectrum of **2** exhibits a deshielding in CH$_2$Te ($\sim$0.74 ppm) and ArH signals ($\sim$0.17 to 0.20 ppm) with respect to those of free **L**^{1}. Similarly ^{13}C-NMR spectrum of **2** has deshielded CH$_2$Te ($\sim$11 ppm) and C$_5$ ($\sim$6 ppm) signals with respect to that of free **L**^{1}. Both these observation suggest that the ligation of **L**^{1} with Ru in solution is also through Te only as shown in Fig. 3 for the crystal.

Acknowledgements

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