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Synthesis, crystal structures and antioxidant studies of Pd(II) and Ru(II) complexes of 2-(4-methoxyphenyltelluro) ethanol

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ABSTRACT

Pd(II) (**1**) and Ru(II) (**2**) complexes of 2-(4-methoxyphenyltelluro)ethanol (**L**), were synthesized using $Na_2[PdCl_4]$ and $[Ru(\eta^6\text{-}p\text{-cymene})Cl_2]_2$ as metal ion sources. The new complexes (**1** and **2**) were characterized by ^1H, $^{13}C\{^1H\}$ NMR, FT-IR, UV–Visible spectroscopy and elemental analyses. Their structures were also confirmed by single crystal X-Ray diffraction. The characterizationdata revealed that the ligand **L** coordinated to M(II) ion as a monodentate telluroether ligand in both **1** and **2**. The structure of *cis*-$[PdCl_2(\mathbf{L})_2]$ (**1**) was square planar about Pd in which two molecules of **L** coordinated to palladium (II) in *cis*-fashion. Interestingly, two molecules of **1** were held by strong Te···Cl and aromatic π···π secondary interactions and oriented in staggered conformation with short Pd-Pd [3.1712 (5) Å] distance forming a dinuclear palladium cluster $(cis\text{-}[PdCl_2(\mathbf{L})_2])_2$ $(\mathbf{1})_2$. The complex $[Ru(\eta^6\text{-}p\text{-cymene})Cl_2(\mathbf{L})]$ (**2**) has half-sandwich, pueudo-octahedral structure. There exists several intermolecular hydrogen bonds of type: OH···Cl and OH···O in **1** and CH···Cl, CH···O and OH···Cl in **2**. These interactions results in supramolecular assemblies in the new complexes **1** and **2**. *In-vitro* antioxidant activity of **L**, **1** and **2** was investigated using DPPH assay. Vitamin-C was used as a standard antioxidant. All three compounds showed to exhibit antioxidant activity. The free radical scavenging activity was quantitatively expressed in terms of their IC_{50} values which found in the order: Vitamin-C ~**2** > **L** > **1**.

1. Introduction

Synthesisedtelluroether and hybrid organotellurium ligands and their coordination chemistry have been extensively studied with transition metals [1]. Though tellurium (Te) was long been considered a non-essential trace element but the biological role of organotellurium compounds has still been not completely established [2] in comparison to those of its congeners S and Se [3] and it can't be left unconcern. Organotellurium compounds are promising because some of the current studies have demonstrated that they are potential enzyme mimetics [4], antioxidants [5] and have shown to overshine as free radical scavengers [6] under biologically relevant conditions when compared to those of the corresponding S and Se compounds and presence of other functional groups also expected to be more pronounced on their activity [7].

Since the discovery of *cis*-platin, an anticancer drug, the interest in the synthesis and characterization of palladium (Pd) and ruthenium (Ru) complexes of ligands containing different donor atoms has been increased because of their potential biological [8] and efficient catalytic activities [9], mimic platinum (Pt) and iron (Fe) respectively under similar physiological conditions [10], Pd is cost effective and Ru exhibit easily accessible oxidation states (II/III) [11], improved solubility, high reactivity, less toxicity to normal tissues and high activity of their complexes [10–12]. Recently, the quasi-octahedral half-sandwich complexes containing [(η^6-arene) Ru(II)] species have been demonstrated to exhibit excellent biological activity [8c,12,13,14]. The Ru and Pd complexes have been investigated in the treatment of cancer [8a,c,d] but are yet to be explored in chemotherapy to a large extent [15].

[1] Organotellurium ligands have shown potential biological activity [5] and have been proved to be excellent antioxidants [16]. They behaves as hemilabile ligands with soft metals especially Ru and Pd, their complexes containing metal-tellurium (M – Te) bond have been explored as efficient catalysts for various organic transformations [1,9]. They show high *trans influence* and stabilise the unstable organotellurium compounds even in aqueous solution [1g,17]. However, the metal complexes containing M – Te bond

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have never been explored in biological activities [8]. Therefore, the designing and synthesis of potential Ru and Pd complexes of organotellurium ligands as antioxidants would be very much interesting.

The synthesis and structural characterization of metal complexes of hybrid organotellurium ligands of (Te, O) type containing ethereal oxygen (R-O-R) have been well studied [18,1c] in which they behaved as monodentate telluroethers, but those containing alcoholic (R—OH) group are not fully explored [19]. Hence, herein we have explored the coordination chemistry of 2-(4-methoxyphenyltelluro)ethanol (**L**) with Pd(II) and Ru(II) species using Na_2PdCl_4 and [Ru (η^6-*p*-cymene)Cl_2]$_2$ respectively as metal ion sourses. The resulted complexes, *cis*-[Pd(**L**)$_2$Cl$_2$] (**1**) and [Ru (η^6-*p*-cymene)Cl_2(**L**)] (**2**) were structurally characterized by ^{1}H, ^{13}C{^{1}H} NMR, FT-IR, UV Visible spectroscopy, elemental analysis and single crystal X-ray diffraction techniques. These compounds (**L**, **1** and **2**) were also explored *in-vitro* free radical scavenging activity studies using DPPH radicals. The results of these investigations are presented in this article.

2. Experimental section

2.1. Reagents and analytical methods

Tellurium (Te), sodium tetrachloropalladate (Na_2PdCl_4), dichloro (*p*-cymene)ruthenium (II) dimer ([$RuCl_2$(*p*-cymene)]$_2$), 1,1-diphenyl-2-picrylhydrazyl (DPPH), 2-chloroethanol and anisole were purchased from Sigma Aldrich Ind. Ltd. All other chemicals and reagents used were of analytical reagent (AR) grade and the common reagents and solvents were purchased from Spectrochem Ind. Pvt. Ltd. and used without further purification. All the chemical reactions were carried out under oxygen and moisture free nitrogen (N_2) atmosphere. Analytical thin layer chromatography (TLC) was conducted on Merck-60 F254 silica gel pre-coated on aluminum sheets. TLC plates were viewed under UV-light, using potassium permanganate solution, ninhydrin stain and/or iodine spray. Flash column chromatography was performed using silica gel 230—400 mesh obtained from Merck Ind. Pvt. Ltd. The tellurium precursor compounds, bis-(4-methoxyphenyl)ditelluride (Ar_2Te_2) was prepared as per the reported procedure [20]. Melting points were determined in open capillary tubes closed at one-end and were reported uncorrected. Infrared spectra were recorded on a Jasco FT-IR-4100 instrument in the wavenumber range of 4000-400 cm^{-1}. ^{1}H and ^{13}C{^{1}H} NMR spectra were recorded at 400 and 100 MHz respectively with TMS as an internal standard on AVANCE-II Bruker 400 MHz spectrometer and the chemical shifts are given in δ ppm. UV—Visible absorption spectra were recorded using Thermo scientific Evolution-220 spectrophotometer in the spectral window of 200—800 nm. Bruker APEX-II CCD has been used in the single crystal X-ray data collection of complex **1**, and Rigaku CrysAlisPro for that of complex **2**. The structures were solved using Olex2 [21] and SHELXTL [22] structure solution programs by direct methods. SADABS has been used in the absorption correction of complex **1** and the software incorporated in CrysalisPro for that of complex **2**.

Synthesis of complex *cis*-[$PdCl_2$(L)$_2$] (1)

Na_2PdCl_4 (0.294 g, 1.0 mmol) was dissolved in 5 mL of distilled water and stirred magnetically for 2—3 min. A solution of ligand **L** (0.560 g, 2.0 mmol) made in 10 mL of acetone was added to the above solution under vigorous stirring. After which the resulting solution was stirred further 2 h for completion of complexation reaction. The orange-red solution so obtained was poured into 50 mL of distilled water from this, complex **1** was extracted into methylenedichloride (2 × 25 mL). The organic layer was dried under anhydrous sodium sulfate. When the solvent was distilled off under reduced pressure using rotary evaporator resulted an orange-red solid. Orange-red single crystals of complex **1** were grown after 3—4 days when the solution of above solid in a 1:1 mixture of chloroform and n-hexane was stored at 4—5 °C in refrigerator. The crystal were filtered and air dried.

Yield: 75%; M.P: 122—125 °C; Elemental analysis: Found (Calculated) for $C_{18}H_{24}Cl_2O_4PdTe_2$: C, 29.34 (29.31); H, 3.28 (3.25); FT-IR (KBr, υ cm^{-1}): 3446 (OH), 1586 (C=C), 590 (C-Te, aryl), 507 (C-Te, alkyl); ^{1}H NMR (DMSO-d_6, δ ppm): 3.273 (bs, 2H, $TeCH_2$), 3.643 (bs 2H, OCH_2), 3.785 (s, 3H, OCH_3), 5.21 (s, 1H, OH), 6.921—6.941 (d, J = 8.0 Hz, 2H, ArH *ortho* to Te), 7.60 (bs, ArH *meta* to Te); ^{13}C{^{1}H} NMR (DMSO-d_6, δ ppm): 25.1 (Te-CH_2), 55.33 (OCH_3), 58.25 (O-CH_2), 106.13 (ArC-Te), 115.64 (ArC *meta* to Te), 138.04 (ArC *ortho* to Te), 160.72 (ArC-OCH_3).

Synthesis of complex [Ru(*p*-cymene)Cl_2(L)] (2)

A solution of [$RuCl_2$(*p*-cymene)]$_2$ (0.612 g, 1.0 mmol) in 10 mL of dry MDC was stirred for 2—3 min and then was added a solution of **L** (0.560 g, 2.0 mmol) in 20 mL of dry MDC at room temperature. The resulting orange solution was stirred further for 1 h. The consumption of the ligand during this time was steadily monitored by TLC. Then, the solution was concentrated to 2—3 mL on rotary evaporator. An orange-red precipitate of **2** was obtained by slow addition of n-hexane (3 mL) with vigorous magnetic stirring. The precipitate was filtered and then recrystallized from a 1:1 mixture of dry MDC and n-hexane at 4—5 °C in a refrigerator. The orange-red crystals of **2** obtained after 3 days were filtered and air dried.

Yield: 65%; M.P: 117—120 °C; Elemental analyses: Found (Calculated) for $C_{19}H_{26}Cl_2O_2RuTe$: C, 38.90 (38.94); H, 4.47 (4.47); FT-IR (KBr, υ cm^{-1}): 3429 (OH), 1581 (C=C), 590 (C-Te aryl), 521 (C-Te alkyl); ^{1}H NMR ($CDCl_3$, δ ppm), 1.225—1.250 (d, J = 10.0 Hz, 3H, CH_3 of *i*-Pr), 1.266—1.292 (d, J = 10.4 Hz, 3H, CH_3 of *i*-Pr), 2.104 (s, 3H, CH_3 *para* to *i*-Pr),2.715 (bs, 1H, $TeCH_2$), 2.802—2.856 (m, 1H, CH of *i*-Pr), 3.41 (bs, 1H, $TeCH_2$), 3.481 (bs, 1H, OCH_2), 3.726 (bs, 1H, OCH_2), 3.848 (s, 3H, OCH_3), 4.90 (bs, 1H, ArH of *p*-cymene), 5.252, 5.257, and 5.422 (3bs, 3H, ArH of *p*-cymene), 6.901—6.919 (d, J = 8.0 Hz, 2H, ArH *ortho* to Te), 7.882—7.901 (d, J = 8.0 Hz, 2H, ArH *meta* to Te); ^{13}C{^{1}H} NMR: ($CDCl_3$, δ ppm), 18.45 (Te-CH_2), 18.48 (*p*-cymene CH_3), 22.41 and 23.64 (CH_3 of *i*-Pr of *p*-cymene), 30.86, (CH of *i*-Pr of *p*-cymene), 55.34 (OCH_3), 59.68 (O-CH_2), 81.59 (ArC of *p*-cymene *meta* to *i*-Pr), 84.95 (ArC of *p*-cymene *ortho* to *i*-Pr), 98.20 (ArC-CH_3 of *p*-cymene), 104.52 (ArC-Te), 106.38 (ArC-*i*-Pr of *p*-cymene), 115.45 (ArC *meta* to Te), 137.48 (ArC *ortho* to Te), 161.36 (ArC-OCH_3).

2.2. Free radical scavenging activity (DPPH assay)

A stock solution of DPPH (0.1 mM) was made in 50% methanol. The solutions of different concentrations (10, 20, 30, 40, 50 and 60 μM) of compounds (**L**, **1** and **2**) were prepared in 50% methanol. Then, to each of these solutions 140 μL of above DPPH solution was added and then incubated at 37 °C for 30 min in dark. The absorbance was measured at λ_{max}, 517 nm against 50% methanol as blank. The actual absorbance was recorded as the difference in the absorbance of the control (absorbance without test sample) and the test sample.

3. Results and discussion

3.1. Synthesis of ligand (L) and complexes (1-2)

The ligand, 4-$MeOC_6H_4TeCH_2CH_2OH$ (**L**) was synthesized

according to the procedure reported in the literature by Singh*et al.* [19] who explored the coordination chemistry of ligand (**L**) with Pd(II), Hg(II) and Pt (II) species wherein these complexes were synthesized by reaction of ligand in the form of sodium alkoxide (4-$MeOC_6H_4TeCH_2CH_2ONa$) with the corresponding metal chlorides ($PdCl_2$ and $HgCl_2$) and $K_2PtCl_4$by reacting them (L:M) in 1:1 ratio. They have reported that the **L** behaved as monobasic bidentate (Te,O^-) ligand with M-Cl-M bridging in complexes. In this paper, we have synthesized the Pd(II) (**1**) and Ru(II) (**2**) complexes by direct reaction of **L** with Na_2PdCl_4 and [Ru (η^6-*p*-cymene)Cl_2]$_2$ respectively in 2:1 (L:M) ratio at room temperature (27–28 °C). The chemical equations for the reactions involved in the synthesis of ligand **L**and its new complexes **1** and **2** are shown in Scheme 1.

3.2. Characterization of complexes (1-2)

The composition of complexes **1** and **2** determined by elemental analysis was in agreement with that calculated according to their empirical formulae. The complexes were found soluble in chloroform, dichloromethane (MDC), ethanol, methanol, tetrahydrofuran (THF), acetonitrile, dimethylformamide (DMF) and dimethyl sulfoxide (DMSO) but were found insoluble in water, diethyl ether, n-hexane, n-heptane and toluene. The structural characterization of complexes **1** and **2** was carried out by FT-IR, ^{1}H, ^{13}C{^{1}H} NMR, UV–Visible spectroscopy and single crystal X-ray diffraction.

3.3. FT-IR spectra

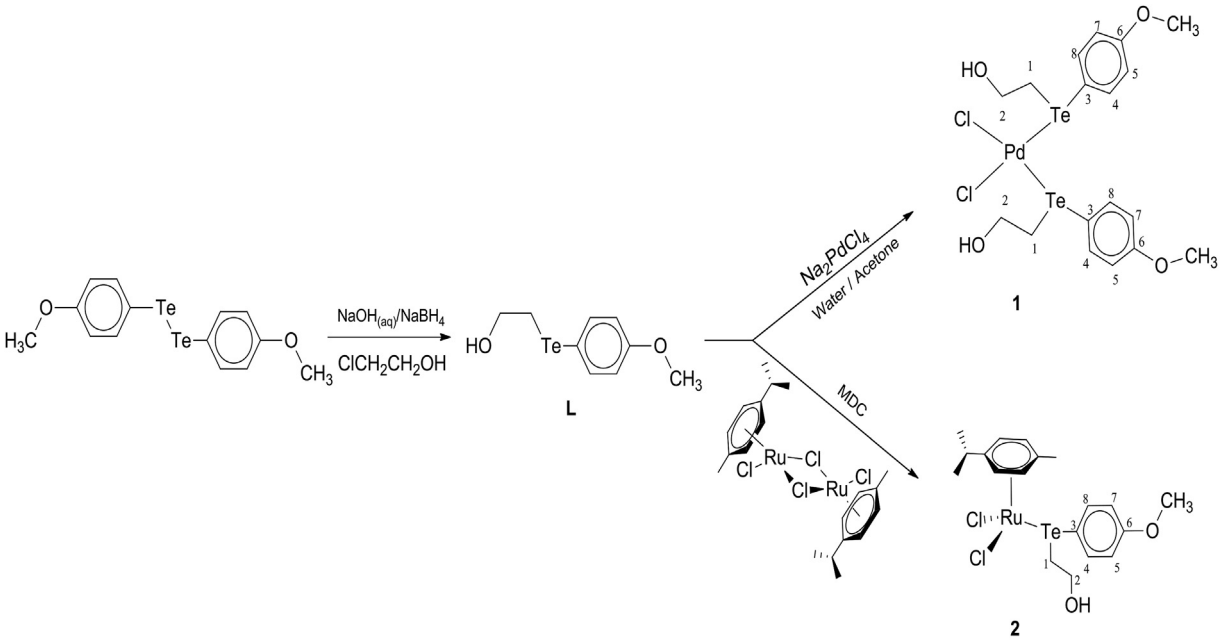
In the IR spectra of complexes **1** and **2**, a strong absorption band was observed at ν, 3446 and 3429 cm^{-1} respectively which was attributed to the O-H stretching and this band showed blue shift of above 200 cm^{-1} when compared to the IR spectrum of free ligand (ν, 3200 cm^{-1}). This observation indicated that the presence of free OH group and not invoved in the coordination with metal unlike reported before [19] but the shift was due to the strong secondary interactions. The ArC-Te stretching vibrational band was appeared at ν, ~590 cm^{-1} while the RC-Te band was appeared at ν, 507 and 521 cm^{-1} respectively in **1** and **2** as expected for a coordinated organotelluroether ligand [23,1h]. The other IR bands were appeared at characteristic wavenumbers. The representative FT-IR spectra of **1** and **2** are given in Fig. S1 and Fig. S2 respectively as an supplementary information (SI).

3.4. ^{1}H and ^{13}C{^{1}H} NMR spectra

The ^{1}H and ^{13}C{^{1}H} NMR spectra of **1** and **2** are represented in Figs. S3, S4, S5.**1**and**S6** respectively asSI. In the ^{1}H NMR spectrum of **1**, the peaks appeared broad. The CH_2Te and aromatic (*ortho* and *meta* to Te) protons showed downfield shift of about δ, 0.21–0.26 ppm respectively in comparison with those signals in the ^{1}H NMR spectrum of ligand [19]. In the ^{1}H NMR spectrum of complex **2**, the CH_2Te protons and aromatic (*ortho* and *meta* to Te) protons showed downfield shift of δ, 0.15–0.20 ppm [19]. The signals for O-H proton was observed at 5.21 in **1** and was significantly deshielded due to its involvement in strong secondary interactions and no signal was observed for O-H proton in **2**. The signals for other protons in **1** and **2** appeared at characteristic chemical shifts even though the peaks were broad.

In the ^{13}C{^{1}H} NMR spectrum of **2**, the peaks for C-1 and C-3 carbons appeared at 22.41 and 104.52 ppm whereas in **1**, they appeared at 25.1 and 106.13 ppm respectively. These signals showed downfield shift of about δ, 5 and 8 ppm in **1** and **2** respectively when compared to the corresponding signals in free ligand [19]. But these signals in complex **1** were found in very low intensity as indicated in Fig. S5.2 which may be due to the involvement of Te in Te···Cl secondary interactions. These shifts were in agreement with the Pd(II) and Ru(II) complexes of similar ligand, 2-(4-methoxyphenyltelluro)ethyl amine [1h, 23].

The results observed from the in the FT-IR, ^{1}H and ^{13}C{^{1}H} NMR spectra revealed that the ligand (**L**) coordinated to the palladium (II) and ruthenium (II) centers through Te hence, acting as a monodentate telluroether in both **1** and **2** and the OH group remained uncoordinated.



Scheme 1. Synthesis of ligand, **L** and its complexes, *cis*-[$PdCl_2$(**L**)$_2$] (**1**), and [Ru (η^6-*p*-cymene)Cl_2(**L**)] (**2**).

3.5. Electronic spectra

The UV–Visible spectra of the 2-(4-methoxyphenyltelluro) ethanol (**L**) and its palladium (II) (**1**) and ruthenium (II) (**2**) complexes were recorded in DMSO and are shown in SI (Fig. S7). In the electronic absorption spectra of **L**, **1** and **2**, there exists an intense absorption band at λ_{max}, ~253 nm. This prominent band arises from the ligand centered (LC) π - π* intra-ligand charge transfer (ILCT) transition. A weak absorption band observed at λ_{max}, ~320 nm in the ligand **L** and in its complexes **1** and **2** was also attributed to the n - π* ILCT transition.

3.6. Single crystal X-ray diffraction

Single crystals of palladium (**1**) and ruthenium (**2**) complexes of 4-$MeOC_6H_4TeCH_2CH_2OH$ (**L**), were obtained from a solution of chloroform:n-hexane (1:1) for complex **1** and from MDC:diethyl ether (1:1) for complex **2**. The orange-red crystals were formed when these solutions stored at 4–5 °C for 3–4 days in the refrigerator. The crystal data and structure refinement parameters collected for the complexes **1** and **2** are given in Table 1. The crystal data revealed that both the complexes crystallized in monoclinic crystal system. The molecular structures of **1** and **2** are shown in Fig. 1 and Fig. 2 respectively. The selected bond lengths and bond angles of **1** and **2** are given in Table 2.

3.7. Crystal structure of 1

The molecular structure of **1** demonstrated that the two molecules of ligand **L** coordinated to palladium (II) center through the Te only in *cis*-fashion resulting the complex as *cis*-[$PdCl_2$(**L**)$_2$] (**1**) and acquired square planar geometry at Pd(II) ion. The Pd (1)–Te (1), Pd (1)–Te (2), Pd (1)–Cl (1) and Pd (1)–Cl (2) bond lengths found were 2.5373(7), 2.5405(6), 2.3823(12) and 2.3615 (13) Å respectively. The Pd-Te [2.530 (1) to 2.546 (1) Å] and [Pd-Cl 2.351 (1) to 2.359 (1) Å]

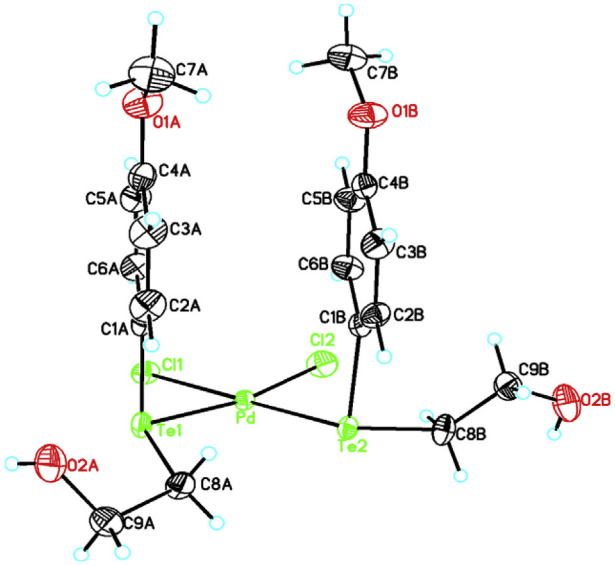


Fig. 1. (a) Molecular structure of complex*cis*-[$PdCl_2$(**L**)$_2$] (**1**).

bond lengths are in agreement with those reported for *cis*-Pd(II) complexes of monodentate telluroether ligands, (C_4H_3E)TeMe (where E = O and S) [24]. However, the Pd-Te [2.5817 (3) to 2.6052 (2) Å] bonds shorter and Pd-Cl [2.290 (2) to 2.329 (2) Å] are longer than those observed in some of the *trans*-Pd(II) complexes of monodentate telluroether ligands [25]. The short Pd-Te and long Pd-Cl bonds observed in complex **1** were due to the strong *trans influence of* Te of **L** [1g] which is higher than that reported for *cis*-complexes of other telluroether ligands [24].

In crystalline solid of complex **1**, there were three significant weak Te···Cl [3.332and 3.452 Å] and two aromatic π···π [3.744(3)-3.745(3) Å] secondary interactions as per the classification by

Table 1
Crystal data and structure refinement parameters for **1** and **2**.

Compound	1	2
Empirical formula	$C_{18}H_{24}Cl_2O_4Te_2Pd$	$C_{19}H_{26}Cl_2O_2RuTe$
Formula weight	735.86	585.97
Temperature, K	293 (2)	295 (2)
Wavelength, Å	1.54178	0.71073
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/c$
a, Å	10.384 (2)	9.9617 (9)
b, Å	8. 6405 (17)	8.3670 (7)
c, Å	24.827 (5)	25.303 (2)
α,γ°	90	90
β°	94.18 (3)	90.736 (8)
Volume, Å^3	2221.6 (8)	2108.8 (3)
Z	4	4
Density (cal.), Mg/m^3	2.203	1.846
μ/mm^{-1}	29.444	2.363
F (000)	1392	1144
Size, mm^3	0.22 × 0.25 × 0.27	0.29 × 0.22 × 0.17
Theta (θ) range (°)	6.672 to 64.57	2.045 to 33.362
Index ranges	−11 ‹ = h ‹ = 12 −10‹ = k ‹ = 9 −28‹ = l ‹ = 15	−14 ≤ h<=14, 0 ≤ k<=12, −16 ≤ l<=38
Reflections	13072	7470
Independent reflections	3639 [R_{int} = 0.0374]	7439 [R_{int} = 0.09494]
Data/Restraints/Parameters	3639/3/253	7439/21/243
Goodness-of-fit on F^2	1.054	1.299
Final R indices [I > 2sigma(I)]	R1 = 0.0317, w*R*2 = 0.0811	R1 = 0.0987, w*R*2 = 0.1509
R indices (all data)	R1 = 0.0332, w*R*2 = 0.0823	R1 = 0.1107, w*R*2 = 0.1551
Largest diff. peak and hole, eA^{-3}	1.212 and −1.015	1.088 and −1.806
CCDC No.	1040585	1852878

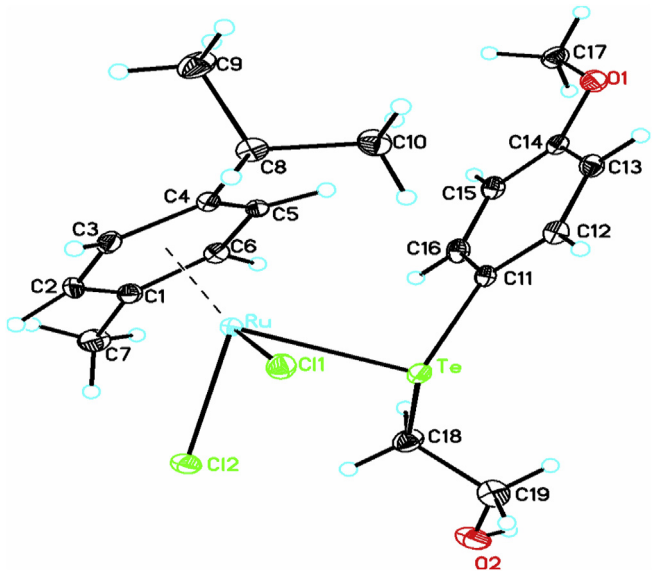


Fig. 2. Molecular structure of complex [$RuCl_2$(*p*-cymene) (**L**)] (**2**).

Table 2
Selected bond lengths (Å) and bond angles (°) of **1** and **2**.

1			
Pd-Cl (1)	2.3816 (12)	Cl (2)-Pd-Te (2)	94.29 (4)
Pd-Cl (2)	2.3613 (13)	Cl (1)-Pd-Te (2)	172.15 (3)
Pd-Te (2)	2.5405 (6)	Te (1)-Pd-Te (2)	91.358 (12)
Pd-Te (1)	2.5374 (7)	Cl (2)-Pd-Pd#1	99.29 (5)
Pd-Pd#1	3.1712 (10)	Cl (1)-Pd-Pd#1	88.31 (3)
Cl (2)-Pd-Cl (1)	92.30 (4)	Te (1)-Pd-Pd#1	87.37 (3)
Cl (2)-Pd-Te (1)	170.86 (4)	C (1A)-Te (1)-Pd	95.30 (12)
Cl (1)-Pd-Te (1)	81.58 (3)	C (8A)-Te (1)-Pd	111.86 (12)
Symmetry transformations used to generate equivalent atoms: #1 -x+1,-y+1,-z+1			
2			
Ru-Te	2.6438 (7)	Cl (2)-Ru-Te	84.97 (6)
Ru-Cl (1)	2.421 (2)	C (18)-Te-Ru	108.4 (8)
Ru-Cl (2)	2.424 (2)	C (18A)-Te-Ru	99.1 (7)
C (11)-Te-Ru	104.64 (18)	Cl (1)-Ru-Te	82.27 (5)

Jeffrey [26] forming a dinuclear palladium cluster (*cis*-[$PdCl_2$(**L**)$_2$])$_2$ (**1**)$_2$ and the *cis*-[$PdCl_2$(**L**)$_2$] molecules were oriented in staggered conformation with short Pd-Pd [3.1712 (5) Å] bonds as shown in Fig. S8.

One of the $-CH_2$-CH_2-OH group was placed over two positions due to dynamic disorderness rising from thermal vibrations. The two positions of the group were treated by using PART command to obtain site occupancy ratio 0.60(3):0.40(3). During final stages of the refinement all the disordered atoms C8BA, C8BB, C9BA, C9BB, O2BA and O2BB were refined anisotropically and their anisotropic displacement factors were treated as equivalent to the corresponding carbon/oxygen atoms in the other $-CH_2$-CH_2-OH group.

The *cis*-complexes of palladium (II) with monodentate telluroether ligands are rare [24]. Few *cis*-complexes of Pd(II) were reported for ditelluroether [27,25b] and hybrid telluroether [28] ligands. The Pd-Pd distance [3.171 (7) Å] in **1** was comparatively shorter and stronger than that of *trans*-complexes of palladium (II) with monodentate telluroether [3.214 (1) Å] [25d], and hybrid tridentate organotellurium [3.203 (1) Å] [29] ligands as well as that of *cis*-complexes of palladium (II) with bidentate ditelluroether [3.1993 (3) Å] [30] and other hybrid organotellurim [3.4156 (6) Å] [31] ligands.

Further, complex **1** contain moderate to strong intermolecular secondary interactions of OH$^{\cdots}$O [2.35 Å], CH$^{\cdots}$O [2.64 Å] and CH$^{\cdots}$Cl [2.51 to 2.95 Å] type as shown in Fig. S9 and its packing diagram is shown in Fig. S10 (SI). These bonding parameters present in complex **1** are given in Table 3.

3.8. Crystal structure of 2

The crystal structure of complex **2** also revealed that the ligand **L** found to act as monodentate telluroether resulting [Ru (η^6-*p*-cymene)Cl_2(**L**)] (**2**). In **2**, the Ru-Te (1), Ru-Cl (1) and Ru-Cl (2) bond lengths found were 2.644(7), 2.421(2) and 2.424 (2) Å respectively. These bond lengths are in agreement with the reported values of Ru(II) complexes of other monodentate organotellurium ligands [18f,32,25d].The complex **2** has pseudo-octahedral geometry at Ru(II) ion and acquired half-sandwich also known as piano-stool structure with η^6-*p*-cymene, two chloro (Cl^-) and **L**. In **2**, there exists significant intermolecular secondary interactions as given in Table 3 such as OH$^{\cdots}$Cl [2.36 to 2.88 Å], CH$^{\cdots}$Cl [2.74–2.93 Å] and C-H$^{\cdots}$O [2.27 Å] which results in supramolecular assembly. These interactions are shorter than sum of their corresponding van der Waals radii 2.95 Å (H$^{\cdots}$Cl) and 2.72 Å (H$^{\cdots}$O) respectively. Intermolecularsecondary interactions and crystal packing present in [Ru (η^6-*p*-cymene)Cl_2(**L**)] (**2**)are shown in Figs. S11 and S12 respectively (SI).

Secondary interactions between chalcogens [E$^{\cdots}$E] or chalcogen and heteroatoms [E$^{\cdots}$X] (where, X = N, O, S, F, Cl, Br and I) in organochalcogen compounds or their complexes results in supramolecular structures [33]. These non-bonded interactions reported to play important roles in biological applications particularly in the DNA binding [34], catalytic antioxidant activity [5] and/or otherwise enhance the stability.

3.9. DPPH free radical scavenging activity

2, 2-diphenyl-1-picrylhydrazyl (DPPH) [35] is a stable free radical that has long been used widely as an antioxidant assay for the evaluation of *in-vitro* free radical scavenging capacity of newly synthesized compounds. In the presence of an antioxidant, the DPPH radical quenched to reduced form with the loss of its violet color [36] and then the change in absorbance at 517 nm was

Table 3
Hydrogen-bonds (Å, °) in **1** and **2**.

D—H···A	D—H	H···A	D···A	D—H···A
1				
O (2A)-H (2A) ... O (1B)#3	0.82	2.35	2.951 (6)	130.3
C (8A)-H (8AA) ... O (2BB'b)#2	0.97	2.64	3.440 (13)	140.4
C (7B)-H (7BA) ... Cl (2)#4	0.96	2.94	3.795 (6)	148.7
C (8BA'a)-H (8BD'a) ... Cl (2)	0.97	2.95	3.569 (14)	123.1
C (9BA'a)-H (9BB'a) ... Cl (1)#5	0.97	2.95	3.654 (13)	129.9
C (8BB'b)-H (8BC'b) ... Cl (2)	0.97	2.51	3.263 (15)	134.7
Symmetry codes: #1 -x+1,-y+1,-z+1; #2 -x+1,-y+2,-z+1; #3 -x+1/2,y-1/2,-z+1/2; #4 -x+3/2,y+1/2,-z+1/2; #5 x,y+1,z				
2				
O (2)—H (2) ... Cl (2)#1	0.82	2.55	3.30 (2)	152.4
O (2A)-H (2A1) ... Cl (1)#1	0.82	2.88	3.455 (18)	128.9
O (2A)-H (2A1) ... Cl (2)#1	0.82	2.36	3.11 (2)	153.0
C (19)-H (19A) ... O (1)#2	0.97	2.27	3.11 (3)	145.3
C (18)-H (18A) ... Cl (2)	0.97	2.93	3.59 (4)	126.4
C (18A)-H (18C)···Cl (2)	0.97	2.74	3.43 (3)	128.0
C (19A)-H (19C)···Cl (2)#1	0.97	2.79	3.40 (2)	121.8
Symmetry transformations used to generate equivalent atoms: #1 -x+1,y-1/2,-z+3/2 #2 -x,y+1/2,-z+3/2				

Table 4
$\pi^{\cdots}\pi$ secondary interactions between benzene ring of ligand in **1**.

Cg(I) Res(I) Cg(J)	Cg$^{\cdots}$Cg	Slippage
Cg1 [1] - > Cg2	3.745 (3)	0.564
Cg2 [1] - > Cg1	3.744 (3)	0.602

measured by UV–Visible spectrophotometer. *In-vitro* antioxidant activity of 2-(4-methoxyphenylrelluro)ethanol (**L**), an organotellurium ligand and its newly synthesized Pd(II) and Ru(II) complexes (**1** and **2**) was assessed in accordance with the method of Choi et al. [36] using DPPH assay. The percentage inhibition of free radicals generated from DPPH by varying concentrations (10–60 μM) with an increment of 10 μM of compounds (**L**, **1** and **2**) was measured. The measurements were made similarly for a standard compound Vitamin-C (Vit-C). A graph was plotted for % inhibition against concentration of each compound and Vit-C. The graph obtained is shown in Fig. 3 from which the concentration of a compound required to inhibit 50% of the radicals (IC_{50}) was determined. The IC_{50} values of **L**, **1** and **2** were found in the range of 9–33 μM also represented by bar diagram in Fig. 4. These results suggested that the above organotellurium ligand and its palladium and ruthenium complexes containing M – Te bond(s) showed antioxidant activity. Among the compounds (**L**, **1** and **2**) tested, the ruthenium complex, **2** (IC_{50}, 9.8) found an effective antioxidant in compared with standard, Vit-C (IC_{50}, 11.9).

4. Conclusions

The Pd(II) (**1**) and Ru(II) (**2**) complexes of 2-(4-methoxyphenyltelluro)ethanol, a telluroether ligand (**L**) containing OH functionality were synthesized and structurally characterized by ^{1}H, $^{13}C\{^{1}H\}$ NMR, UV–Visible, FT-IR spectroscopy, elemental analysis. The molecular structures of *cis*-[$PdCl_2(L)_2$] (**1**), [Ru (η^6-*p*-cymene)Cl_2(**L**)] (**2**) were further confirmed by single crystal X-ray diffraction. The data revealed that in both **1** and **2**, the **L** coordinated through Te only and acting as a monodentate telluroether ligand. In crystalline solid of **1**, the two monodentate telluroether (**L**) were found coordinated to Pd(II) ion in *cis*-fashion and between which there were strong intermolecular *Te*$^{\cdots}$Cl and aromatic $\pi^{\cdots}\pi$ secondary interactions. The two molecules of **1** configured in staggered conformation held through a short Pd-Pd bond resulted in di-nuclear palladium clusters, (*cis*-[$PdCl_2$(**L**)$_2$])$_2$ (**1**)$_2$. The complex **2** exhibit pseudo-octahedral geometry and half-sandwich structure about the central Ru(II) ion. In the crystals of **1** and **2** the complex molecules were held by strong intermolecular secondary interactions of type *OH*$^{\cdots}$Cl, *CH*$^{\cdots}$Cl and *C* – *H*$^{\cdots}$O resulted in supramolecular structures. The ligand and complexes were explored for the antioxidant activities for first time. All the compounds (**L**, **1** and **2**) showed excellent free radical scavenging activity and the IC_{50} values (9–33 μM) demonstrated that these compounds can act as moderate to strong antioxidants among which the ruthenium complex (**2**) could be an effective antioxidant and is highly in agreement with standard antioxidant Vit-C.

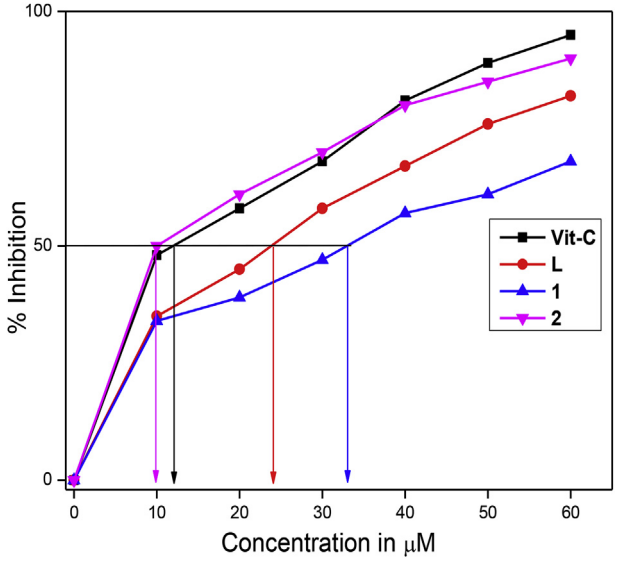


Fig. 3. Antioxidant activity as percentage inhibition of DPPH radicals against concentration of **L**, **1**, **2** and Vit-C.

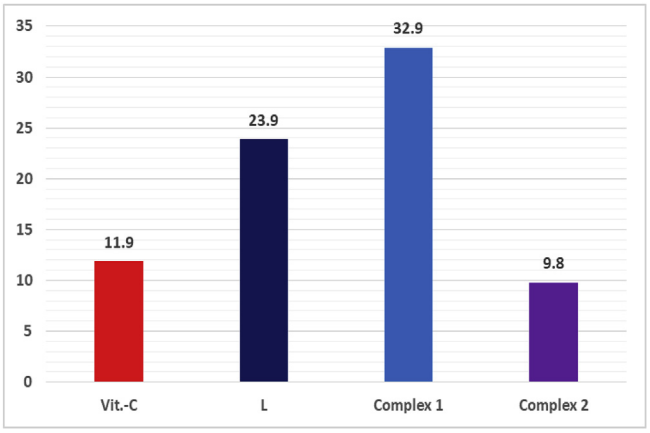


Fig. 4. IC_{50} values of **L**, **1**, **2** and Vit-C.

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Appendix A. Supplementary data

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