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STUDY ON 4-METHOXYBENZOHYDROXAMATE COMPLEXES OF ARYLTELLURIUM (IV) AND DIARYLTELLURIUM (IV)

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ABSTRACT

Twelve new complexes of the 4- methoxybenzohydroxamate with aryltellurium (IV) and diaryltellurium (IV) of type $RTeCl_2.L$, $RTeCl.L_2$, $R_2TeCl.L$ and $R_2Te.L_2$ (where R = 4-methoxyphenyl, 4-hydroxyphenyl, 3-methyl-4-hydroxyphenyl and L = 4-methoxybenzohydroxamate) have been synthesized by reactions of $RTeCl_3$ and R_2TeCl_2 with potassium 4-methoxybenzohydroxamate. They have been characterized by elemental analyses, molar conductance, FT-IR and ^1H NMR spectroscopy. The spectral studies predict the bonding of ligand through hydroxylamine and carbonyl oxygens (*O, O'*) coordination to give penta- and hexa- coordinated tellurium (IV) complexes. The complexes also have been screened for their antimicrobial activity against various bacteria and fungi organisms and it has been observed that they are more active against fungi as compared to Gram +ve and Gram –ve bacteria.

Key words: 4-Methoxybenzohydroxamate, Aryltellurium (IV), Diaryltellurium (IV), Antimicriobial activity.

INTRODUCTION

Hydroxamic acids, containing -C(O)NHOH functionality, constitute a very unique family of chemicals that possess a wide spectrum of biological activities[1]. These weak acids are one of most important families of organic bioligands, which can form coordination compounds with a variety of metals ions[2-4]. They acts as selective inhibitors of many enzymes and consequently possess hypotensive, anticancer, antimalarial, antituberculosis antifungal properties and hence been used in the design of therapeutic targets for cancer[5-6], alzheimer′s disease[7], malaria[8] and haemochromatosis[9,10]. Anion derived from hydroxamic acids i.e. hydroxamates are known to chelate to a number of metal ions[11-16] to form stable

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complexes. By far the most important application of hydroxamic acid and hydroxamates is as metal ions chelators, which have been reviewed by various workers[13-16].

Arylhydroxamates including benzohydroxamates can chelate to the metal ions *via* a number of coordination modes, not only the *O, O′* mode but also *N, O*- modes, most common being the chelation through *O, O*- mode[17-26]. Also, aryltellurium (IV) trichlorides are known[27-40] to behave as lewis acids and form complexes with various N- , O- and S-donor bases, diaryltellurium (IV) dichlorides also form such complexes but only with strong chelating ligands[41-43].

In view of biological relevance of hydroxamates and acceptor properties of aryltellurium (IV) trichlorides and dichlorides and in continuation of our earlier work[44,45] on hydroxamates of tellurium (IV), we report herein the synthesis, characterization and antimicrobial studies on some new hydroxamate complexes of the type $RTeCl_2.L$, $RTeCl.L_2$, $R_2TeCl.L$ and $R_2Te.L_2$; where R = 4-methoxyphenyl, 4-hydroxyphenyl and 3-methyl-4-hydroxyphenyl and L = 4-methoxybenzohydroxamate.

EXPERIMENTAL

Materials and methods

All the chemicals used were of Anal AR grade. All preparations were carried out under an atmosphere of dry nitrogen and the solvents used were purified and dried by standard method[46,47]. 4-Methoxyphenyltellurium (IV) trichloride[48,49], bis (4-methoxyphenyl) tellurium (IV) dichloride[49,50], 4-hydroxyphenyltellurium (IV) trichloride[51], bis(4-hydroxyphenyl) tellurium (IV) dichloride[51], 3-methyl-4-hydroxyphenyltellurium (IV) trichloride[52] and bis (3-methyl-4-hydroxyphenyl)tellurium (IV) dichloride[52] were prepared by the reactions of $TeCl_4$ with anisole/phenol/*o*-cresol as reported in the literature[48-52].

Preparation of potassium 4-methoxybenzohydroxamate (KL)

The potassium salt of 4-methoxybenzohydroxamic acid has been obtained in two steps.

(a) Preparation of ethyl ester of 4-methoxybenzoic acid[46]

To 0.3 mole of 4-methoxybenzoic acid, was added an excess (upto 3 moles) of ethyl alcohol and 1 mL of conc. H_2SO_4 in a reaction flask. The contents were then refluxed for about 3-6 hours till whole of the acid dissolved in ethanol. The reaction mixture was cooled and transferred to about 75 mL of water in a separating funnel. This was shaken thoroughly

and then allowed to settle. The lower layer of ester was removed and was washed with saturated sodium bicarbonate till no effervescence. Finally ester layer was washed with water and dried over anhydrous Na_2SO_4.

(b) Preparation of potassium salt of 4-methoxybenzohydroxamic acid

The potassium salt was obtained by the method reported by Houser and Renfrow[53]. Cooled solution of KOH (56.1 g in about 140 mL methanol) was added to methanolic solution of hydroxylamine hydrochloride (69.49 g in about 240 mL) with constant shaking and cooling. The mixture was allowed to cool for 24 hrs in an ice bath to ensure complete precipitation of KCl, which was removed by filteration. To this filtrate was added 50 mL of ethyl ester of 4-methoxybenzohydroxamic acid prepared above. The reaction mixture was kept in air tight flask at room temperature for 2-3 days to yield the fine crystals of potassium 4-methoxybenzohydroxamate. This was filtered and dried in air. Yield 80%, m. p. > 300^oC (dec.).

Preparation of 4-methoxybenzohydroxamate complexes of aryltellurium (IV)

Aryltellurium (IV) trichlorides, $RTeCl_3$ (R = 4-methoxyphenyl, 4-hydroxyphenyl, 3-methyl-4-hydroxyphenyl), when reacted with potassium 4-methoxybenzohydroxamate in different molar ratios, yield $RTeCl_2.L$ and $RTeCl.L_2$ type complexes.

$RTeCl_2.L$

A warm saturated methanolic solution of potassium 4-methoxybenzohydroxamate (0.41 g, 2 mmol) was added dropwise to a solution of aryltellurium(IV) trichloride (2 mmol) in about 20 mL chloroform/methanol. An immediate precipitation of KCl resulted, which was removed by filteration. The filterate was refluxed for 3-4 hrs to precipitate out any KCl and clear solution was then concentrated to about one third of the original volume and kept overnight to yield crystalline product. This was filtered, washed with chloroform and dried in a vacuum desiccator over P_4O_{10}.

$RTeCl.L_2$

The saturated solution of aryltellurium (IV) trichloride (2 mmol) in chloroform/ methanol was added dropwise with constant stirring to a saturated methanolic solution of potassium 4-methoxybenzohydroxamate (0.82 g, 4 mmol). An immediate change in colour with precipitation of KCl took place, which was removed by filteration. The contents were then refluxed for about 3-4 hrs. The clear solution thus obtained was concentrated to about one third of original volume and left overnight to obtain coloured crystalline product, which was filtered, washed with chloroform and dried in a vacuum desiccator over P_4O_{10}.

Preparation of 4-methoxybenzohydroxamate complexes of diaryltellurium (IV)

Diaryltellurium (IV) dichlorides, R_2TeCl_2 (R = 4-methoxyphenyl, 4-hydroxyphenyl and 3-methyl-4-hydroxyphenyl), when treated with potassium 4-methoxybenzohydroxamate yield both 1:1 and 1:2 complexes of the type $R_2TeCl.L$ and $R_2Te.L_2$. These have been synthesized by the same procedure as for 4-methoxybenzohydroxamates of aryltellurium (IV) described above.

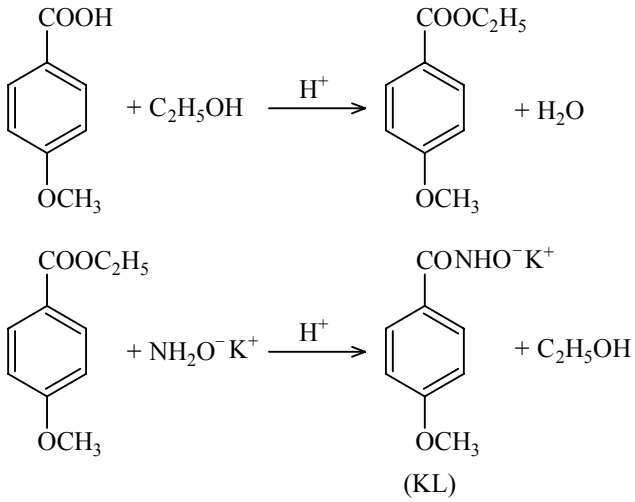
Physical studies

Conductance studies were performed under dry condition at 25 ± 2°C in DMSO with a dip type conductivity cell on microprocessor based conductivity bridge type MICROSIL.

Infrared spectra (4000-400 cm^{-1}) were recorded in KBr pellets on Alpha Bruker FT-IR spectrometer. Proton Magnetic Resonance spectra were recorded in DMSO-d_6 using TMS as an internal reference on BRUKER AVANCE II 400 NMR spectrometer at Sophisticated Analytical Instrumentation Facility, Panjab University, Chandigarh. The antimicrobial screening was carried out by Tube Dilution Method.

RESULTS AND DISCUSSION

Preparation of potassium 4-methoxybenzohydroxamate can be represented as below:



(KL)

Aryltellurium (IV) trichlorides and diaryltellurium (IV) dichlorides when reacted with potassium 4-methoxybenzohydroxamate (KL) in 1:1 and 1:2 molar ratios give the corresponding aryltellurium (IV) hydroxamates.

Table 1: Analytical data, molar conductance and physical properties of aryltellurium (IV) 4-methoxybenzo hydroxamates

Compd. No.	Complex (R)	Empirical formula (Formula wt.)	Colour Yield (%)	Analyses % Found (Calculated)					M. P. (°C)	Λ_M at *ca.* 10^{-3}M ohm^{-1} cm^2mol^{-1} in DMSO
				Te	Cl	C	H	N		
1	$RTeCl_2.L$ (4-methoxyphenyl)	$C_{15}H_{15}Cl_2NO_4Te$ (471.67)	Light brown (65)	26.89 (27.04)	14.81 (15.05)	37.89 (38.19)	3.30 (3.18)	2.53 (2.97)	100-102	62.65
2	$RTeCl.L_2$ (4-methoxyphenyl)	$C_{23}H_{23}ClN_2O_7Te$ (602.31)	Light cream (70)	20.95 (21.18)	5.62 (5.89)	45.18 (45.86)	3.95 (3.82)	4.22 (4.65)	106-108	34.27
3	$RTeCl_2.L$ (4-hydroxyphenyl)	$C_{14}H_{13}Cl_2NO_4Te$ (457.66)	Brown (75)	27.45 (27.87)	15.12 (15.51)	36.23 (36.74)	2.69 (2.84)	2.87 (3.06)	65-68	48.31
4	$RTeCl.L_2$ (4-hydroxyphenyl)	$C_{22}H_{21}ClN_2O_7Te$ (588.30)	Light brown (80)	21.24 (21.68)	5.91 (6.03)	44.35 (44.91)	3.30 (3.57)	4.29 (4.76)	72-74	35.39
5	$RTeCl_2.L$ (3-methyl-4-hydroxyphenyl)	$C_{15}H_{15}Cl_2NO_4Te$ (471.67)	Dark brown (60)	26.83 (27.04)	14.82 (15.05)	37.78 (38.19)	2.99 (3.18)	2.75 (2.97)	70-72	52.48
6	$RTeCl.L_2$ (3-methyl-4-hydroxyphenyl)	$C_{23}H_{23}ClN_2O_7Te$ (602.31)	Brown (70)	20.91 (21.18)	5.42 (5.89)	45.38 (45.86)	4.07 (3.82)	4.28 (4.65)	78-80	38.75
7	$R_2TeCl.L$ (4-methoxyphenyl)	$C_{22}H_{22}ClNO_5Te$ (543.30)	Dull white (75)	23.01 (23.48)	6.32 (6.53)	48.35 (48.63)	4.19 (4.05)	2.11 (2.58)	90-92	65.70
8	$R_2Te.L_2$ (4-methoxyphenyl)	$C_{30}H_{30}N_2O_8Te$ (673.94)	Light cream (80)	18.51 (18.92)	_	53.02 (53.47)	4.29 (4.45)	3.94 (4.16)	84-86	49.93
9	$R_2TeCl.L$ (4-hydroxyphenyl)	$C_{20}H_{18}ClNO_5Te$ (515.28)	Dark pink (85)	24.50 (24.76)	6.52 (6.89)	46.28 (46.61)	3.85 (3.49)	2.47 (2.72)	82-84	45.77
10	$R_2Te.L_2$ (4-hydroxyphenyl)	$C_{28}H_{26}N_2O_8Te$ (645.92)	Light pink (80)	19.22 (19.75)	_	51.69 (52.06)	4.35 (4.02)	4.19 (4.34)	118-120	39.15
11	$R_2TeCl.L$ (3-methyl-4-hydroxyphenyl)	$C_{22}H_{22}ClNO_5Te$ (543.30)	Cream (70)	23.04 (23.48)	6.20 (6.53)	48.07 (48.63)	3.83 (4.05)	2.21 (2.58)	99-101	53.94
12	$R_2Te.L_2$ (3-methyl-4-hydroxyphenyl)	$C_{30}H_{30}N_2O_8Te$ (673.94)	Light cream (65)	18.20 (18.92)	_	53.03 (53.47)	4.81 (4.45)	3.98 (4.16)	120-124	39.76

Values of Λ_M reported[54, 55] for 1:1 electrolytes in DMSO = 50 – 70 ohm^{-1} cm^2 mol^{-1}

$$RTeCl_3 + KL \longrightarrow RTeCl_2.L + KCl$$

$$RTeCl_3 + 2\,KL \longrightarrow RTeCl.L_2 + 2\,KCl$$

$$R_2TeCl_2 + KL \longrightarrow R_2TeCl.L + KCl$$

$$R_2TeCl_2 + 2\,KL \longrightarrow R_2Te.L_2 + 2\,KCl$$

The analytical data, physical properties and yields for these aryltellurium(IV) 4-methoxybenzohydroxamates are compiled in Table 1.

Conductance studies

Molar conductance, Λ_M data at *ca.* 10^{-3} M for aryltellurium(IV) benzohydroxamates in DMSO lie in the range 34.27-65.70 ohm^{-1} cm^2 mol^{-1}, which predict the weak to 1:1 electrolyte[54,55] type behaviour of these hydroxamates in DMSO, probably due to ionization into $RTeCl.L^+$/ $RTe.L_2^+$/ $R_2Te.L^+$ and Cl^- in DMSO. The dissociation for $R_2Te.L_2$, which do not contain any Cl^-, may be due to steric factors and donor behaviour of DMSO to result in probable dissociation into $R_2Te.L.DMSO^+$ and L^- ions.

Infrared spectral studies

The infrared spectra of the aryltellurium(IV) 4-methoxybenzohydroxamates are quite complexes and an attempt has therefore been made to identify the donor sites of 4-methoxybenzohydroxamate ligand by comparing with those of parent aryltellurium (IV) chlorides and potassium 4-methoxybenzohydroxamate, which indicated clear differences.

The principal infrared absorption bands of ligand, KL are due to $\nu_{(C=O)}$, $\nu_{(C-N)}$, $\nu_{(N-O)}$ and $\nu_{(N-H)}$ stretching vibrations of the hydroxamate group, which appeared in the spectrum at 1607 cm^{-1}, 1383 cm^{-1}, 955 cm^{-1} and 3247 cm^{-1}, respectively, Table 2.

Table 2: IR data (cm^{-1}) for potassium 4-methoxybenzohydroxamate and complexes

Compound	$\nu_{(C=O)}$	$\nu_{(C-N)}$	$\nu_{(N-O)}$
KL	1607 s	1383 m	955 m
1	1600 vs	1463 m	1022 m
2	1569 s	1443 m	1023 s
3	1570 sh	1432 m	1023 m

Cont…

Compound	$\nu_{(C=O)}$	$\nu_{(C\square N)}$	$\nu_{(N\square O)}$
4	1571 vs	1440 m	1023 s
5	1602 sh	1437 m	1022 m
6	1570 sh	1435 m	1023 m
7	1569 sh	1463 m	1024 s
8	1580 s	1460 s	1024 s
9	1575 s	1444 s	1022 m
10	1575 s	1444 m	1023 m
11	1602 vs	1437 m	1022 m
12	1580 sh	1440 m	1023 m

s = strong, vs = very strong, m = medium, w = weak, sh = shoulder

The absorption band occurring at 1607 cm^{-1} in parent hydroxamate attributes to $\nu_{(C=O)}$ mode, which is shifted to lower wave numbers and appeared at 1569-1602 cm^{-1} in aryltellurium (IV) 4-methoxybenzohydroxamates. This band in some cases appears as a shoulder, which may be due to mixing of $\nu_{C=C}$ of aryl moiety. The absorption band due to $\nu_{(C-N)}$ mode occurring at 1383 cm^{-1} in free KL has been found to shift towards higher region at 1432-1463 cm^{-1} in the complexes. The band at around 3247 cm^{-1} due to $\nu_{(N-H)}$ mode in KL did not undergo any change, however could not be ascertained due to phenolic OH group in some of the aryltellurium moieties. This rules out the involvement of coordination through nitrogen atom. The sharp band occurring at 955 cm^{-1} in potassium 4-methoxybenzohydroxamate ascribed to $\nu_{(N-O)}$ mode has been observed to move towards higher wave number and appeared at about 1022 cm^{-1} in aryltellurium (IV) hydroxamates.

A shift in $\nu_{(C=O)}$ mode to lower wave number and $\nu_{(N-O)}$ mode to higher wave numbers are suggestive of bonding of 4-methoxybenzohydroxamate ion *via* oxygen atoms of carbonyl and hydroxylamine group[17,56-60]. The formation of Te-O bond however, could not be confirmed due to non availability of far IR data.

Proton magnetic resonance spectral studies

Proton magnetic resonance spectra of aryltellurium (IV) 4-methoxybenzo hydroxamates are very complex and a lot of overlapping of aryl proton singals of the ligand and aryltellurium (IV) moiety takes place, thus making the independent assignment almost impossible. The chemical shift data for the complexes are presented in the Table 3.

Table 3: ^{1}H NMR Spectral data of aryltellurium (IV) 4-methoxybenzohydroxamates

Compound	Chemical shift, δ ppm in DMSO-d_6
1	3.83, 3.85 (s, 6H, OCH_3), 6.92-8.39 (cm, 8H, aryl protons of RTe and L), 11.11 (s, 1H, NH)
2	3.81, 3.85 (s, 9H, OCH_3), 6.92-8.38 (cm, 12H, aryl protons of RTe and L), 11.08 (s, 2H, NH)
3	3.84 (s, 3H, OCH_3), 6.82-8.28 (cm, 9H, aryl protons of RTe & L + phenolic OH of R), 11.11 (s, 1H, NH)
4	3.83 (s, 6H, OCH_3), 6.82-8.28 (cm, 13H, aryl protons of RTe & L + phenolic OH of R), 10.16, 11.11 (s, 2H, NH)
5	2.53 (s, 3H, CH_3 of R), 3.82 (s, 3H, OCH_3), 6.83-7.93 (cm, 7H, aryl protons of R and L), 8.14 (s, 1H, phenolic OH of RTe), 10.14 (s, 1H, NH).
6	2.18 (s, 3H, CH_3 of R), 3.83 (s, 6H, OCH_3), 6.92-7.77 (cm, 11H, aryl protons of R and L), 9.92 (s, 1H, phenolic OH of RTe), 11.07 (s, 2H, NH)
7	3.83 (s, 9H, OCH_3), 6.92-8.17 (cm, 12H, aryl protons of R_2Te and L), 11.09 (s, 1H, NH)
8	3.35, 3.81 (s, 12H, OCH_3), 6.89-8.18 (cm, 16H, aryl protons of R_2Te and L), 11.08 (s, 2H, NH)
9	3.81 (s, 3H, OCH_3), 6.77-7.87 (cm, 12H, aryl protons of R_2Te and L), 8.17 (s, 2H, phenolic OH of R_2Te), 11.08 (s, 1H, NH)
10	3.82 (s, 6H, OCH_3), 6.77-7.91 (cm, 16H, aryl protons of R_2Te and L), 8.88 (s, 2H, phenolic OH of R_2Te), 11.09 (s, 2H, NH)

s = singlet, d = doublet, cm = complex multiplet, compound 11 & 12 poor solubility- spectra not well resolved

Free 4-methoxybenzohydroxamic acid shows[61-64] two downfield singlets at 11.06 and 8.94 δ ppm due to NH and NOH protons, the aryl protons are centred at 7.71 δ ppm.

All the complexes show singlets at around 11.1 δ ppm, which may be assigned to -NH of benzohydroxamate group[61,62,64]. This rules out the linkage of 4-methoxybenzo-hydroxamate through nitrogen atom. Absence of NOH proton signals around 8.9 δ ppm confirms the deprotonation of this proton and subsequently linkage to tellurium atom.

Further, the aryl protons of aryltellurium (IV), diaryltellurium (IV) and 4-benzohydroxamate groups exhibit a lot of overlapping of signals and are observed as

complex multiplets in the region 6.77-8.39 δ ppm, as observed in ^{1}H NMR Spectra of organotin (IV) and aryltellurium(IV) complexes of hydroxamic acids[44,45,56]. Also, a careful examination of ^{1}H NMR spectra of complexes reveal the shielding of aryl protons of RTe/R_2Te compared to $RTeCl_3$/R_2TeCl_2[51,52,65,66] due to flow of electron density from the ligand to the aryltellurium moiety as a result of complexation.

Thus, on the basis of infrared and proton magnetic resonance spectral studies it may be concluded that 4-methoxybenzohydroxamate acts as a bidentate (*O, O*) ligand involving the hydroxamate (-NHO) and carbonyl oxygens, giving rise to penta coordinated tellurium complexes in $RTeCl_2$.L and R_2TeCl.L and hexa coordinated in $RTeCl.L_2$ and $R_2Te.L_2$. The suggested structures are as shown in Fig. 1.

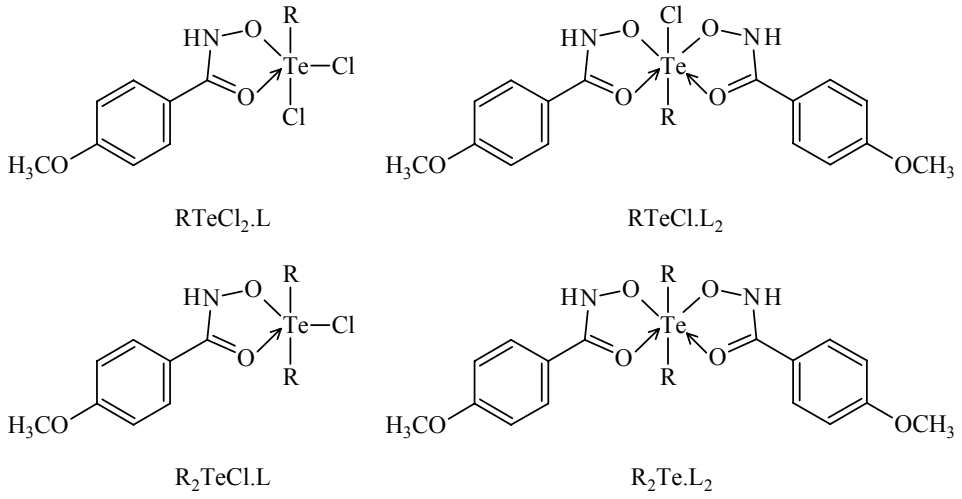


Fig. 1: Proposed structures of complexes

Antimicrobial activities

The 4-methoxybenzohydroxamate and newly synthesized aryltellurium (IV) hydroxamate complexes were screened for their *in vitro* antimicrobial potential against Gram positive bacteria: *S. aureus* ATCC 11632 and *B. cereus* MTCC 7350, Gram negative bacteria *E. coli* ATCC 35218, *P. aeruginosa* ATCC 23564 and *S. typhi* ATCC 15499; fungal strains *A. niger*, *A. fumigates* and *A. flavus* by tube dilution method[67]. Dilution of test and standard compounds were prepared double strength nutrient broth- I.P (Antibacterial) and Sabouraud Dextrose Broth –I. P. (Antifungal)[68]. The samples were incubated at 37 ± 1ºC for 24 hr (bacteria), 25 ± 1ºC for 7 days (*A. niger*), 30 ± 1ºC for 15 days (*A. flavus*), 35 ± 1ºC for 72 hr (*A. fumigates*), respectively and results were recorded in terms of MIC (the lowest concentration of test substances, which inhibited) values are presented in the Table 4.

Table 4: Minimum inhibitory concentration, MIC (µg/mL); (-) resistant

Compound	Bacteria species						Fungal strains		
	S. aureus (ATCC 11632)	*S. typhi* (ATCC 15499)	*P. aeruginosa* (ATCC 23564)	*E. coli* (ATCC 35218)	*B. cereus* (MTCC 7350)	*P. rettgeri* (DRDE strain)	*A. niger*	*A. fumigates*	*A. flavus*
KL	2.5	1.25	2.5	2.5	0.625	2.5	-	10	2.5
$RTeCl_2.L$ (4-methoxyphenyl)	20	-	5	10	-	1.25	-	1.25	5
$RTeCl_2.L$ (4-hydroxyphenyl)	10	10	20	-	5	10	10	10	-
$RTeCl.L_2$ (4-hydroxyphenyl)	20	-	20	-	10	20	-	-	-
$RTeCl_2.L$ (3-methyl-4-hydroxy phenyl)	10	10	-	-	-	0.625	20	5	1.25
$R_2TeCl.L$ (4-methoxyphenyl)	2.5	1.25	-	5	10	5	5	-	2.5
$R_2TeCl.L$ (4-hydroxyphenyl)	1.25	2.5	1.25	5	-	-	5	2.5	-
$R_2Te.L_2$ (4-hydroxyphenyl)	5	-	-	-	1.25	2.5	-	-	2.5

The data show that the hydroxamate complexes of aryltellurium(IV) exhibit more antifungal activity as compared to potassium 4-methoxybenzohydroxamate, whereas the antibacterial activities are not in the order. Also, the antibacterial activities of the complexes derived from diaryltellurium(IV) are greater than those of corresponding aryltellurium(IV) complexes.

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