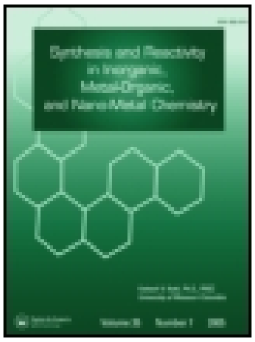

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SYNTHESIS AND CHARACTERIZATION OF PYRIDINE AND BIPYRIDYL COMPLEXES OF 4-HYDROXYPHENYLTELLURIUM TRIHALIDES

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ABSTRACT

New complexes of 4-hydroxyphenyltellurium trihalides with pyridine and 2,2-bipyridyl have been synthesized and characterized by elemental analyses, conductance measurements, molecular weight determinations, IR and ^{1}H NMR spectral studies. Pyridine forms $RTeX_3.L$ and $RTeX_3.L_2$ complexes, whereas bipyridyl, which acts as a bidentate ligand, forms only 1:1 complexes. The complexes are weak to 1:1 electrolytes in solution. Coordination occurs through nitrogen atoms of the ligands.

INTRODUCTION

Aryltellurium trichlorides are known to behave as Lewis acids and form molecular complexes with several nitrogen, oxygen and sulphur donor bases[1-9]. Tellurium in these complexes is penta- or hexa-coordinated,

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but the reports on hexa-coordinated complexes are very few[4,9]. Also, a few such complexes are reported[6,7] to have bactericidal and fungicidal activities. The hydroxyphenyltellurium trihalides, in addition to the acceptor tellurium atom, have an acidic hydroxyl group to interact with bases. In view of this, it was thought desirable to study the reactions of 4-hydroxyphenyltellurium trihalides with some nitrogen donor ligands such as pyridine and 2,2'-bipyridyl. In this paper, we report the synthesis and characterization of some new complexes of the type $RTeX_3.L$ (X = Cl, Br, I; L = pyridine, bipyridyl) and $RTeX_3.L_2$ (L = pyridine).

RESULTS AND DISCUSSION

The reaction of tellurium tetrachloride with phenol proceeds with electrophilic substitution of the $TeCl_3^+$ unit at the *para* position of the aromatic ring, which is contrary to the expectation when the acidic nature of the hydroxyl proton is considered[10,11].

$$\underset{(R\text{-}H)}{HOC_6H_5} + TeCl_4 \rightarrow \underset{(RTeCl_3)}{HOC_6H_4TeCl_3} + HCl \qquad (1)$$

4-Hydroxyphenyltellurium trichloride undergoes halogen-exchange [10,11] when treated with KBr or KI.

$$RTeCl_3 + 3KX \rightarrow \underset{(X = Br, I)}{RTeX_3} + 3KCl \qquad (2)$$

These 4-hydroxyphenyltellurium trihalides when reacted with pyridine form $RTeX_3.Py$ as well as $RTeX_3.Py_2$; whereas with 2,2'-bipyridyl, which acts as a bidentate ligand, only $RTeX_3.Bipy$ type complexes are obtained.

All the complexes are coloured solids, which are fairly stable in dry air except $RTeCl_3.Py_2$ which is hygroscopic. The complexes are soluble in polar organic solvents.The analytical data of the complexes are presented in Table I.

Conductance and Cryoscopic Studies

The molar conductance data of ca. 10^{-3} M solutions in nitrobenzene, acetonitrile and acetone (Table II) reflect the weak to 1:1 electrolytic behaviour of these complexes in solution, presumably due to ionization into $[RTeX_2.L]^+$ or $[RTeX_2.L_2]^+$ and X^- ions. The complexes appear to be more dissociated in acetonitrile than in nitrobenzene and acetone. Also, the adducts of 4-hydroxyphenyltellurium triiodide have larger values of molar conductance in acetonitrile and acetone than those of the tribromide and trichloride adducts. $RTeI_3.Py_2$ has a molar conductance value in acetonitrile and acetone larger than expected for 1:1 electrolytes[12], which may be due to dissociation of this compound beyond a 1:1 type electrolyte or partial decomposition in this solvent. The lower values of molar conductance in some cases may be due to incomplete dissociation or ion-pair formation.

It has been noted[12] that the concentration ranges selected for such measurements are often arbitrarily chosen and that the method of determination requires the assumption of a molecular weight which may be erroneous. The complexes which are predicted as 1:1 electrolytes by molar conductance data and have sufficient solubility were, therefore, investigated in acetonitrile over a wider range (1.0-5.0 mM) and the results subjected to the Onsager Law. Values of equivalent conductance at infinite dilution, Λ_0 and $(A + \omega B \Lambda_0)$ are recorded in Table II. A comparision of calculated and experimental values of $(A + \omega B \Lambda_0)$ shows[13,14] the compounds

TABLE I.
Physical Properties and Analytical Data for 4-Hydroxyphenyltellurium Trihalide Complexes.

Compound (Empirical Formula)	Experimental Average Mol. Wt. (Formula Weight)	Colour	M.P. °C (dec.)	Yield (%)	Analyses Found (Calcd.) %				
					Te	X	C	H	N
$RTeCl_3.Py$ ($C_{11}H_{10}NOTeCl_3$)	219.0 (406.1)	Light green	232-234	65	31.35 (31.41)	26.14 (26.21)	33.01 (32.49)	2.48 (2.46)	3.08 (3.44)
$RTeCl_3.Py_2$ ($C_{16}H_{15}N_2OTeCl_3$)	238.2 (485.1)	Brown	Hygroscopic	60	26.42 (26.85)	21.92 (22.41)	39.60 (39.50)	3.42 (3.09)	5.85 (5.77)
$RTeCl_3.Bipy$ ($C_{16}H_{13}N_2OTeCl_3$)	253.4 (483.3)	Dark brown	66-67	75	25.68 (26.40)	21.87 (22.03)	40.09 (39.72)	3.01 (2.71)	5.82 (5.79)
$RTeBr_3.Py$ ($C_{11}H_{10}NOTeBr_3$)	265.3 (539.6)	Light yellow	208-210	70	23.28 (23.64)	44.12 (44.40)	24.00 (24.46)	2.04 (1.86)	2.80 (2.59)
$RTeBr_3.Py_2$ ($C_{16}H_{15}N_2OTeBr_3$)	302.2 (618.6)	Dark yellow	174-175	60	20.91 (20.63)	38.82 (38.74)	30.77 (31.04)	2.10 (2.44)	4.22 (4.53)
$RTeBr_3.Bipy$ ($C_{16}H_{13}N_2OTeBr_3$)	307.3 (616.8)	Green	162-163	80	20.52 (20.69)	38.71 (38.86)	30.80 (31.13)	2.51 (2.12)	4.28 (4.54)
$RTeI_3.Py$ ($C_{11}H_{10}NOTeI_3$)	336.9 (680.4)	Reddish brown	>260	60	18.65 (18.70)	55.85 (55.90)	19.10 (19.40)	1.60 (1.48)	2.33 (2.06)
$RTeI_3.Py_2$ ($C_{16}H_{15}N_2OTeI_3$)	383.4 (759.4)	Dark red brown	142-145	70	16.30 (16.80)	49.99 (50.13)	25.02 (25.28)	2.10 (1.99)	3.28 (3.69)
$RTeI_3.Bipy$ ($C_{16}H_{13}N_2OTeI_3$)	389.5 (757.5)	Brown	115-118	70	16.42 (16.84)	49.83 (50.25)	25.10 (25.35)	1.84 (1.73)	3.52 (3.70)

TABLE II.

Molar Conductance, Cryoscopic and $(A + \omega B \Lambda_o)$ Data for 4-Hydroxyphenyltellurium Trihalide Complexes.

Compound	Λ_M at ca. 10^{-3} M, ohm^{-1} cm^2 mol^{-1}			Formula Weight	Conc. range, mmol/L	Average Mol. wt. found in Nitrobenzene	Λ_o, ohm^{-1}	$(A + \omega B \Lambda_o)$ in acetonitrile 1.0 - 5.0 mM		Ratio (found/ calcd.)
	Nitro-benzene	Aceto-nitrile	Acetone					found	calcd.	
$RTeCl_3.Py$	13.5	95.2	125.2	406.1	3.11-4.10	219.0	153.00	562.50	385.70	1.45
$RTeCl_3.Py_2$	23.5	100.4	85.2	485.1	2.74-3.43	238.2				
$RTeCl_3$. Bipy	20.0	104.5	89.6	483.3	3.44-8.96	253.4				
$RTeBr_3.Py$	12.5	115.2	34.5	539.6	2.89-3.61	265.3				
$RTeBr_3.Py_2$	15.8	189.0	85.0	618.6	2.47-3.09	302.2				
$RTeBr_3$. Bipy	15.5	135.2	95.2	616.8	2.16-2.70	307.3	142.00	421.00	375.70	1.12
$RTeI_3.Py$	20.1	169.7	139.8	680.4	1.94-2.42	336.9				
$RTeI_3.Py_2$	18.9	249.3	191.9	759.4	2.17-3.91	383.4				
$RTeI_3$.Bipy	16.3	145.3	115.2	757.5	2.19-5.71	389.5	135.00	571.70	368.70	1.54

Values of Λ_M reported[12] for 1:1 electrolytes: nitrobenzene = 20 - 30, acetonitrile = 120-160, acetone = 100-140.

to behave as 1:1 electrolytes. A higher ratio than expected reflects incomplete dissociation or ion-pair formation.

Molecular weights in nitrobenzene (Table II) are much lower than the formula weights, supporting the results of the conductance studies.

IR Spectral Studies

The infrared data for the complexes of 4-hydroxyphenyltellurium trihalides are given in Table III. These spectra are quite complex and, therefore, an attempt has been made to identify the donor sites of the ligands by comparing the spectra of the complexes with those of the parent hydroxyphenyltellurium trihalides, pyridine or bipyridyl.

The complexes show a strong band corresponding to the O-H strectching frequency in the region 3500-3350 cm^{-1}. The OH deformation bands of medium or strong intensities are observed around 1350 cm^{-1}. This certainly suggests that the OH group of 4-hydroxyphenyltellurium trihalides remains intact and does not interact with the base.

There is a distinct shift to higher frequency (~50±10 cm^{-1}) in the pyridine bands appearing at 1587,1572,1484 and 1436 cm^{-1}. This change in the IR spectrum of pyridine upon complexation indicates [3,6-9,15-23] coordination of the nitrogen atom to the tellurium atom of $RTeX_3$. This shift to higher frequency in the pyridine stretching is due to back bonding from tellurium to the pyridine ring through formation of extensive π-bonding[24]. The ring breathing modes of pyridine at 1030 cm^{-1} and 990 cm^{-1} also appear at slightly higher (40± 10 cm^{-1}) frequencies[14]. Also, the two lowest ring vibrations of pyridine located at 602 cm^{-1} (in-plane ring deformation) and 405 cm^{-1} (out-of-plane ring deformation) undergo significant shifts towards higher frequencies (15-60 cm^{-1}) on coordination and this supports the linkage through the nitrogen atoms[19,25].

Like the pyridine complexes, the 2,2'-bipyridyl complexes also show shift to higher frequency in the C=N stretching frequency, which appears at 1564 cm^{-1} in free bipyridyl, thus indicating coordination through nitrogen[1,8,15,26-29]. The pyridine breathing mode at 990 cm^{-1} also shifts to higher frequency (35 ± 5 cm^{-1}). The C-H out-of-plane bending vibration of the ligand at 754 cm^{-1} is split and is at around 758-762 cm^{-1} and 738-718 cm^{-1} in the complexes. This indicates the bidentate nature of bipyridyl[9]. The metal-sensitive in-plane and out-of-plane ring deformation vibrations which are observed at 625 cm^{-1} and 416 cm^{-1} in bipyridyl, shift to higher (10-45 cm^{-1}) frequencies in the complexes, thus indicating coordination through both nitrogens.

Thus, tellurium acquires a coordination number of six in $RTeX_3.Py_2$ and $RTeX_3$.Bipy, whereas in $RTeX_3$.Py it is only penta-coordinated. The $RTeX_3$.Py complexes most likely display a square pyramidal structure with the hydroxyphenyl group occupying the apical position as reported[1-3,9] for other complexes of this type. The $RTeX_3.Py_2$ and $RTeX_3$.Bipy complexes, however, are more difficult to predict with the present available data and due to the presence of a lone pair of electrons in the valence shell and also the possibilities of geometrical isomerism. The suggested structures for these complexes are shown in Figure 1.

^1H NMR Spectral Studies

The proton chemical shift data δ(ppm) of the ligands, parent $RTeCl_3$ and some complexes are given in Table IV.

A comparison of the spectra of the ligand (pyridine and bipyridyl) and 4-hydroxyphenyltellurium trichloride with the complexes shows

TABLE III.

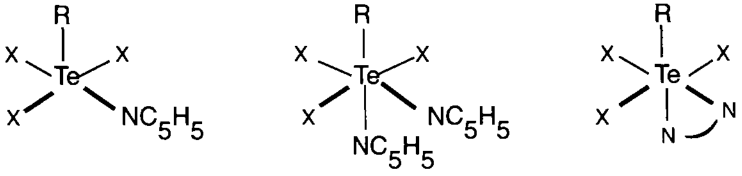
(a) Infrared Data (cm^{-1}) for the Pyridine Complexes of 4-Hydroxyphenyltellurium Trihalides

Compound	υ(C=C) (Sym. ring stretch)	υ(C=N) (In plane anti symm ring vib.)	Ring stretch.	Ring breath.	Ring def. (In-plane)	Ring def. (Out-of-plane)
Pyridine	1587 (s)	1572 (s)	1484 (s) 1436 (vs)	1030 (s) 990 (w)	602 (m)	405 (m)
RTeCl$_3$.Py	1628 (m)	1604 (m)	1548 (m) 1532 (sh) 1492 (vs)	1084 (w) 1030 (vw)	654 (s)	424 (w)
RTeCl$_3$.Py$_2$	1636 (m) 1628 (m)	1604 (m) 1596 (m)	1548 (s) 1492 (vs)	1060 (s) 1040 (vw)	660 (s)	444 (vw)
RTeBr$_3$.Py	1628 (s)	1596 (m)	1540 (m) 1484 (s) 1476 (s)	1050 (s) 1030 (vw)	634 (s)	424 (w)
RTeBr$_3$.Py$_2$	1628 (m)	1612 (m)	1548 (m) 1532 (m)	1075 (sh) 1040 (vw)	662 (s)	444 (wb)
RTeI$_3$.Py	1628 (m)	1596 (m)	1564 (m) 1484 (m) 1468 (s)	1060 (m) 1040 (vw)	658 (s)	424 (vw) 408 (vw)
RTeI$_3$.Py$_2$	1644 (m)	1580 (mb)	1548 (sh) 1524 (w) 1494 (m)	1080 (s) 1040 (s)	662 (w)	418 (m)

(b) Infrared Data (cm^{-1}) for the Bipyridyl Complexes of 4-Hydroxyphenyltellurium Trihalides

Compound	Ring stretch including C=N	Pyridine breathing	C-H Out-of-plane bending	Ring def. (In-plane)	Ring def. (Out-of-plane)
Bipyridyl	1564 (s) 1548 (s) 1452 (s) 1420 (s)	990 (w)	754 (vs) 734 (sh)	625 (s)	416 (w)
$RTeCl_3$.Bipy	1588 (vs) 1564 (m) 1532 (m) 1452 (s) 1404 (m)	1025 (w)	758 (m) 718 (m)	666 (w) 638 (w)	446 (m)
$RTeBr_3$.Bipy	1604 (s) 1580 (s) 1524 (vs) 1484 (s) 1468 (s) 1452 (s) 1420 (s)	1030 (m)	758 (s) 718 (m)	642 (w) 626 (w)	450 (w) 426 (w)
$RTeI_3$.Bipy	1612 (s) 1564 (m) 1548 (m) 1532 (m) 1476 (m) 1408 (m)	1025 (w)	762 (w) 738 (m)	674 (w)	442 (m) 426 (m)

s = strong, w = weak, m = medium, sh = shoulder, v = very, b = broad



[R = 4-hydroxyphenyl; X = Cl, Br, I; $\binom{N}{N}$ = 2,2'-bipyridyl]

Fig. 1. Suggested Structures of the Complexes.

that in the complexes, the ligand protons appear at lower field indicating deshielding whereas the phenyl protons of $RTeCl_3$ appear at higher field indicating their shielding upon complexation. The downfield shifting of protons of the nitrogen-containing donor ligands gives additional evidence[30] that the nitrogens of the ligands are coordinated to the metal atom. Also, the stoichiometry of the complexes is confirmed by ^1H NMR.

These shifts of the chemical shift values in the complexes of the phenyl and ligand protons suggest the non-dissociative nature of tellurium-nitrogen bonds.

EXPERIMENTAL

Reagents

Tellurium tetrachloride was obtained from E. Merck and was purified by a standard method[31]. 4-hydroxyphenyltellurium trichloride was prepared by the method reported in the literature[11] by the reaction of tellurium tetrachloride with phenol. The corresponding tribromide and triiodide were prepared by a halogen-exchange process[10,11] using

TABLE IV.

^{1}H NMR Data of 4-Hydroxyphenyltellurium Trichloride, its Complexes and the Ligands.

Compound	Chemical shift δ (ppm)	Assignment
Pyridine	8.62 (d, 6Hz, 2H)	α
	7.69 (m, 1H)	γ
	7.25 (m, 2H)	β
$RTeCl_3$	7.99 (d, 9Hz, 2H)	ortho to $TeCl_3$ group
	6.99 (d, 9Hz, 2H)	ortho to OH group
	8.85 (s, 1H)	hydroxyl
$RTeCl_3 . Py_2$	8.83 (d, 6H, 4H)	α of pyridine
	8.20-8.43 (m, 2H	γ of pyridine
	& 1H)	hydroxyl of $RTeCl_3$
	7.70-7.94 (m,4H	β of pyridine
	& 2H)	ortho to $TeCl_3$ of phenyl
	6.73-6.90 (m, 2H)	ortho to OH of phenyl
2,2'-Bipyridyl	8.70 (d, 6Hz, 2H)	α to nitrogen
	8.44 (d, 9Hz, 2H)	ortho to pyridyl
	7.84 (m, 2H)	γ to nitrogen
	7.32 (m, 2H)	β to nitrogen
$RTeCl_3$.Bipy	8.80 (d, 6Hz, 2H)	α to nitrogen
	8.53 (d, 9Hz, 2H)	ortho to pyridyl
	8.18- 8.25 (m, 2H	γ to nitrogen
	& 1H)	OH
	7.66- 7.78 (m, 2H	β to nitrogen
	& 2H)	ortho to $TeCl_3$ of phenyl
	6.83- 6.92 (m, 2H)	ortho to OH group

s = singlet, d = doublet and m = multiplet potassium bromide or potassium iodide which were of BDH or Anal R quality. Pyridine and 2,2'-bipyridyl were of Loba Chemie and BDH, Anal R, respectively. All solvents used were dried and purified by standard methods[32,33]. The preparations were carried out under an atmosphere of dry nitrogen.

Synthesis of Complexes: General Methods

$RTeX_3.L$ (X = Cl, Br, I; L = Pyridine, 2,2'-Bipyridyl). A solution of pyridine (0.39 g, 5.0 mmol) in 5 mL of benzene or of bipyridyl (0.78 g, 5.0 mmol) in 10 mL of chloroform was added to a solution of $RTeX_3$ [1.64 g, 2.3 g and 3.0 g (5.0 mmol) for $RTeCl_3$, $RTeBr_3$ and $RTeI_3$, respectively] in about 20 mL of dry methanol. The contents were stirred in a round bottom flask for about 3-4 hours. The solution was then concentrated under vacuum to about one third of its original volume and kept overnight in a vacuum desiccator. This resulted in crystallization of the product. In some cases, however, a little petroleum ether was added for separation of the product. The complex was filtered, washed with benzene and then chloroform and dried over P_4O_{10} in a vacuum desiccator.

$RTeX_3.L_2$ (L = pyridine). A solution of 5.0 mmol of $RTeX_3$ (1.64 g, 2.3 g and 3.0 g for $RTeCl_3$, $RTeBr_3$ and $RTeI_3$, respectively) in about 20 mL dry methanol was added slowly and with constant stirring to a solution of pyridine (0.79 g, 10.0 mmol) in 10 mL of benzene in a round bottom flask. The contents were stirred for about 3-4 hours under an atmosphere of dry N_2. This resulted in the separation of the product. A second crop was obtained after concentration of the filtrate to about one third of its original volume. The complex was filtered, washed with benzene and chloroform and dried over P_4O_{10} in a vacuum desiccator.

All complexes were recrystallized from dry acetonitrile or methanol. The purity of these compounds was checked by thin layer chromatography on silica-gel plates using acetone-benzene (1:4) as irrigant. An iodine chamber was used as developer. All of the compounds were analysed for tellurium, halogen, carbon, hydrogen and nitrogen contents. Tellurium and halogens were determined volumetrically[34] whereas the carbon, hydrogen and nitrogen contents were determined microanalytically by the Regional Sophisticated Instrumentation Centre, Panjab University, Chandigarh and the Indian Institute of Technology, Delhi. The analytical data are compiled in Table I.

Physical Studies

Conductance measurements were carried out under dry conditions at 35 ± 1° C in nitrobenzene (specific conductance = 2.5×10^{-7} ohm^{-1} cm^{-1}), acetonitrile (specific conductance = 1.05×10^{-6} ohm^{-1} cm^{-1}) and acetone (specific conductance = 1.25×10^{-6} ohm^{-1} cm^{-1}) using a highly sensitive type 305 Systronics conductivity bridge and a dip-type conductivity cell with smooth platinum electrodes. Molecular weights were determined cryoscopically in nitrobenzene using a Beckmann thermometer up to the saturation point. ^{1}H NMR spectra were recorded at the Regional Sophisticated Instrumentation Centre, Indian Institute of Technology, Bombay and the Regional Sophisticated Instrumentation Centre, Indian Institute of Technology, Madras, in DMSO-d_6 using TMS as an internal reference. The IR spectra were obtained from the Indian Institute of Technology, Delhi, in KBr pellets using a Nicolet-DX infrared spectrophotometer.

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