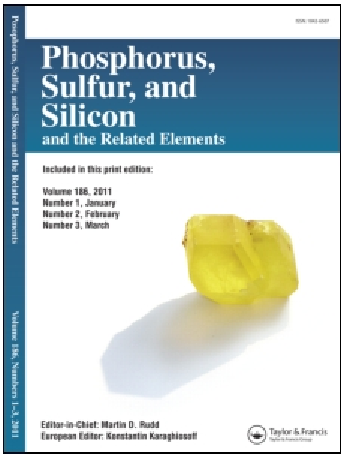

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DIARYLTELLURIUM(IV) CARBOXYLATES: SYNTHESIS *VIA* TELLUROXIDES AND THEIR CHARACTERIZATION

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DIARYLTELLURIUM(IV) CARBOXYLATES: SYNTHESIS *VIA* TELLUROXIDES AND THEIR CHARACTERIZATION

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The synthesis of some new diaryltellurium(IV) dicarboxylates (salicylate, benzoate, cinnamate) and carboxylates (oxalate, *o*-phthalate, succinate) by the reactions of bis(*p*-methoxyphenyl)telluroxide and bis(*p*-hydroxyphenyl)telluroxide with the corresponding carboxylic acid is reported. The resulting carboxylates have been subjected to elemental analyses, conductance and cryoscopic measurements, infra-red and proton magnetic resonance spectral studies. Solution studies reflect the non-ionic nature of these compounds. Infra-red spectral studies predict the unidentate nature of salicylate, benzoate and cinnamate and bidentate nature of oxalate, *o*-phthalate and succinate groups. A PMR spectral study confirms their proposed stoichiometry. These compounds possess a trigonal bipyramidal structure with a tetra-coordinate central tellurium atom.

Keywords: Bis(*p*-methoxyphenyl)-; bis(*p*-hydroxyphenyl)telluroxides; diaryltellurium(IV) carboxylates

INTRODUCTION

Petragnani et al[1] reported the preparation of diaryltellurium dicarboxylates by treatment of diaryltellurium dichlorides with silver carboxylates. Pant et al[2–4] also reported the synthesis of diacetates, dibenzoates by reacting dichlorides with silver carboxylates. Srivastava et al[5,6] synthesized some symmetrical as well as unsymmetrical diaryltellurium carboxylates by the metathetical reaction between freshly prepared silver carboxylates and organotellurium dichlorides. This method was very time-consuming and later on modified[7] and the diaryltellurium carboxylates

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were prepared by direct treatment of the dichloride with the carboxylic acid in the presence of silver oxide which involves the "in situ" formation of silver carboxylates.

A number of diaryltellurium dicarboxylates were obtained by the treatment of the corresponding dichlorides with a basic anionic resin such as Amberlite IR 45}OH⁻ in which OH⁻ was previously exchanged with carboxylate anion[7].

Tamagaki et al[8] synthesized diphenyltellurium dicarboxylates in high yield employing diphenyltelluroxide and carboxylic acid anhydride, a method used previously by Sedekov and coworkers[9]. Reactions of diphenyltelluroxide with carboxylic acid also yield the carboxylates[8] and this appears to be the only report on synthesis of diaryltellurium carboxylates involving reactions between telluroxides and carboxylic acids. Diphenyltellurium dicarboxylates are also reported[8,10] by the "carboxylate exchange" process from diphenyltellurium diacetates.

McWhinnie et al[11] reported the synthesis of diaryltellurium carboxylates by the reactions of diaryltellurium dichlorides with sodium salts of *o*-phthalic acid and tetrabromo *o*-phthalic acid.

A few diaryltellurium carboxylates are known[5,6] to possess considerable biocidal activities. In view of this, it was thought desirable to synthesize and characterize some new bis(*p*-methoxyphenyl)tellurium(IV) carboxylates and bis(*p*-hydroxyphenyl)tellurium(IV) carboxylates by reactions of the corresponding diaryltelluroxides with carboxylic acids such as salicylic acid, benzoic acid, cinnamic acid, oxalic acid, *o*-phthalic acid and succinic acid.

RESULTS AND DISCUSSION

The formation of bis(*p*-anisyl)tellurium(IV) dichloride and bis(*p*-hydroxyphenyl)tellurium(IV) dichloride by reactions of tellurium tetrachloride with anisole[12–16] and phenol[17–19] involves the electrophilic substitution of the aromatic ring by a chlorotellurium group at a position *para* to the methoxy or hydroxyl group.

$$2\text{R-H} + \text{TeCl}_4 \dashrightarrow \text{R}_2\text{TeCl}_2 + 2\text{HCl}$$

The alkaline hydrolysis of these dichlorides yield the diaryltelluroxides[15,17–21].

$$R_2TeCl_2 + 2NaOH \dashrightarrow R_2Te(OH)_2 + 2NaCl$$

$$R_2Te(OH)_2 \xrightarrow{\Delta} R_2TeO + H_2O$$

These diaryltelluroxides which are weakly basic in nature[16,22] react with mono- and dicarboxylic acids to yield the diaryltellurium(IV)dicarboxylates and diaryltellurium(IV) carboxylates.

$$R_2TeO + 2HL \dashrightarrow R_2Te[L]_2 + H_2O$$

(HL = monocarboxylic acids – salicylic acid, benzoic acid and cinnamic acid)

$$R_2TeO + H_2[L_2] \dashrightarrow R_2Te[L_2] + H_2O$$

($H_2[L_2]$ = Dicarboxylic acids – oxalic acid, *o*-phthalic acid and succinic acid)

The reaction of bis(*p*-hydroxyphenyl)telluroxide with benzoic acid, cinnamic acid and succinic acid did not give any carboxylate, probably because of the less basic nature of this telluroxide as compared to bis(*p*-anisyl) telluroxide.

The bis(*p*-methoxyphenyl)tellurium(IV) carboxylates are white crystalline solids whereas bis(*p*-hydroxyphenyl)tellurium(IV) carboxylates are light orange in color. All these compounds are fairly stable in dry air. These are soluble in polar organic solvents except the oxalates and succinates which are insoluble. The bis(*p*-hydroxyphenyl)tellurium(IV) carboxylates are comparatively less soluble than the corresponding bis(*p*-anisyl)tellurium(IV) carboxylates.

Conductance and Cryoscopic Studies

The molar conductance data (Table I) in nitrobenzene, acetonitrile, acetone and dimethylsulphoxide reflect non-electrolyte type behaviour of these diaryltellurium(IV) carboxylates. The Λ_M values at *ca* 10^{-3} M for these compounds are much lower than those reported[23] for 1:1 electrolytes, indicating their non-electrolytic nature in solutions. The cryoscopic data for carboxylates having sufficient solubility in nitrobenzene (Table I) well support the results of conductance measurements and reflect their monomeric nature. The solution behaviour of these carboxylates is thus similar to those of other carboxylates reported in the literature[4,5]. It may be mentioned here that the molecular weight determined by McWhinnie et al[11] for *o*-phthalates in benzene reflects their dimeric nature.

TABLE I Molar conductance and molecular weight data of diaryltellurium(IV) carboxylates

R	*Compound*	*Molar conductance* Λ_M *at ca.* 10^{-3} *M* ohm^{-1} cm^2 mol^{-1}			*Conc. range in mmoles/L*	*Formula Weight*	*Average Mol.wt. found*
		Nitrobezene	*Acetonitrile*	*Acetone*			
p-$CH_3OC_6H_4$	$R_2Te\ [C_6H_4(OH)COO]_2$	0.15	6.90	3.90	3 57–34.90	616.1	572.1
	$R_2Te\ [C_6H_5COO]_2$	0.15	3.90	0.96	2.03–33.09	584.1	560.1
	$R_2Te\ [C_6H_5CHCHCOO]_2$	0.18	3.00	0.72	3.33–26.56	636.2	604.5
	$R_2Te\ [(COO)_2]$	a	a	a	a	429.9	--
	$R_2Te\ [C_6H_4(COO)_2]$	0.27	11.40	5.05	2.91–9.14	505.9	508.3
	$R_2Te\ [CH_2COO)_2]$	a	a	a	a	457.9	--
p-HOC_6H_4	$R_2Te\ [C_6H_4(OH)COO]_2$	$^{a}(22.08)^{b}$	a	a	a	588.0	--
	$R_2Te\ [(COO)_2]$	$^{a}(22.26)^{b}$	a	a	a	401.8	--
	$R_2Te\ [C_6H_4(COO)_2]$	$^{a}(24.62)^{b}$	a	a	a	477.9	--

Values of Λ_M reported[23] for 1:1 electrolyte, nitrobenzene=20–30, acetonitrile = 120–160, acetone = 100–140 and DMSO = 35–70.

a. Insufficient solubility
b. Values in DMSO

Infrared Spectra

The IR spectra of diaryltellurium(IV) carboxylates are quite complex and therefore, an attempt has been made to assign the group frequencies associated with the carboxylate group and thus to predict the linkage between tellurium and the carboxylate group.

The ν_{as}COO in dicarboxylates (salicylate, benzoate and cinnamate) appear at around 1630 ± 15 cm^{-1} whereas ν_sCOO appear at about 1295 ± 10 cm^{-1}. This difference ($\Delta\nu$COO) indicates the unidentate nature of these carboxylates[3–5,24,25]. Also a splitting of the C=O stretching frequency is observed in these dicarboxylates which may be attributed to a solid state effect, different environments of two carboxylate groups or coupling of vibrations of different carboxylate groups[4,7].

ν_{as}(C=O) in monocarboxylates (oxalate, *o*-phthalate, succinate) appear between 1695–1615 cm^{-1} and ν_s(CO) appear between 1300–1240 cm^{-1}. The bands between 1400–1350 cm^{-1} may be assigned to ν_s(CO) + ν(CC)[24]. The separation of ν_{as}(CO) and ν_s(CO), which can be taken as a measure of ester like character of the carboxylate group, is very similar to that noted for derivatives of monocarboxylic acids[4,11]. This also reflects the bidentate chelate structure for these carboxylates[24].

$\nu_{Te=0}$ at about 720 cm^{-1} in the parent telluroxide disappears in these carboxylates and new weak to medium intensity bands around 285 ± 5 cm^{-1} appear which may be assigned to the tellurium-oxygen stretching frequency[4,6].

Proton Magnetic Resonance Spectra

^{1}H NMR spectral data for bis(*p*-methoxyphenyl)tellurium(IV) disalicylate, dibenzoate, *o*-phthalate and bis(*p*-hydroxyphenyl)tellurium(IV) *o*-phthalate are presented in Table II. The absence of COOH protons in the spectra of these carboxylates indicates the linkage of tellurium to the carboxylate group after deprotonation. The ratio of aromatic to methoxy/hydroxyl protons supports the proposed stoichiometry of these compounds[5,7,11]. Also, the aryl protons of diaryltelluroxide[19] appear to be deshielded in these carboxylates probably because of the greater electron withdrawing nature of the carboxylatotellurium group as compared to a telluroxide group.

TABLE II IR and PMR spectral data of diaryltellurium(IV) carboxylates

R	*Compound*	ν_{as}*(COO)* *(cm^{-1})*	ν_{s}*(COO)* *(cm^{-1})*	$\nu_{Te\text{-}O}$	*PMR (δ) (ppm)*
p-$CH_3OC_6H_4$	$R_2Te\ [C_6H_4(OH)COO]_2$	1631 (s)	1301 (s)	285 (m)	3.86 (s, 6H); 6.85 (m, 4H); 7.05 (d, *J 9Hz*, 4H); 7.37 (m,2H); 7.72 (d, *J 9Hz*, 2H); 7.89 (d, *J 9Hz*, 4H); 11 26 (s, 2H)[a]
	$R_2Te\ [C_6H_5COO]_2$	1637 (m) 1621 (m)	1294 (s)	285 (m)	3.79 (s,6H); 7.01 (d, *J 9Hz*, 4H); 7.35–8.0 (m, 14H)[a]
	$R_2Te\ [C_6H_4CHCHCOO]_2$	1638 (s) 1614 (s)	1295 (s) 1286 (s)	281 (w)	
p-HOC_6H_4	$R_2Te\ [C_6H_4(OH)COO]_2$	1629 (m)	1302 (m) 1247 (s)	286 (m)	
p-$CH_3OC_6H_4$	$R_2Te\ [(COO)_2]$	1689 (m) 1660 (s)	1300 (s) 1242 (s)	286 (m)	
	$R_2Te\ [C_6H_4(COO)_2]$	1645 (s)	1295 (s) 1254 (s)	284 (w)	3.77 (s,6H); 7.02 (d, *J 9Hz*, 4H); 7 35 (m, 2H); 7.51 (m, 2H); 7.86 (d, *J 9Hz*, 4H)[a]
	$R_2Te\ [(CH_2COO)_2]$	1642 (s) 1615 (m)	1294 (s) 1255 (s)	286 (m)	
p-HOC_6H_4	$R_2Te\ [(COO)_2]$	1634 (s)	1241 (s,b)	285 (m)	
	$R_2Te\ [C_6H_4(COO)_2]$	1695 (s) 1641 (m)	1281 (s)	282 (m)	6.94 (d, *J 9Hz*, 4H); 7.53–7.56 (m, 4H); 7.75 (d, *J 9Hz*, 4H); 10.10 (s,b,2H)[b]

a. In $CDCl_3$
b. In DMSO

On the basis of results presented in the paper, these compounds may have an ester type structure with probably ψ- trigonal bipyramidal arrangement of groups about the central four-coordinate tellurium atom with one site being occupied by a lone pair of electrons[5,6]. These carboxylates may even contain intermolecular Te-O secondary bonds in the solid state[26,27] which could not be ascertained due to non-availability of x-ray data.

EXPERIMENTAL

Reagents

Tellurium tetrachloride used was from E. Merck and was purified by a standard method[28] using a sublimation process. Phenol used was of BDH, Anal R quality and anisole was from SISCO Research Laboratories (India) and these were purified by standard methods[29] before use. Salicylic acid, benzoic acid, cinnamic acid, oxalic acid, *o*-phthalic acid and succinic acid were from LOBA CHEMIE, Extra Pure grade.

The solvents used were dried and purified by standard methods[29,30] before use.

Preparation of Diaryltelluroxides, R_2TeO

(R = p-methoxyphenyl and p-hydroxyphenyl)

Bis(*p*-methoxyphenyl)telluroxide was prepared by alkaline hydrolysis of bis(*p*-methoxyphenyl)tellurium dichloride[20,21] which in turn was obtained by reaction of tellurium tetrachloride with anisole[12–15].

Bis(*p*-hydroxyphenyl)telluroxide was prepared by alkaline hydrolysis[17,19] of bis(*p*-hydroxyphenyl)tellurium dichloride. The dichloride was prepared by reactions of tellurium tetrachloride with phenol as reported in the literautre[17,19].

Synthesis of Bis(p-methoxyphenyl)tellurium(IV) carboxylates

$R_2Te\ [L]_2$ (R = p-methoxyphenyl, L = salicylate, $C_6H_4(OH)COO^-$; benzoate, $C_6H_5COO^-$; cinnamate, $C_6H_5CHCHCOO^-$)

A saturated solution of bis(*p*-methoxyphenyl)telluroxide (5 mmol) in dry methanol was added to a stirred solution of 10 mmol of carboxylic acid (salicylic acid/benzoic acid/cinnamic acid) in the minimum amount of the same solvent. The contents upon stirring for about 2 hours yielded a white solid which was filtered, washed with methanol and dried in a vacuum desiccator over P_4O_{10}.

$R_2Te[L_2]$ (*R = p-methoxyphenyl,* $[L_2]$ *= oxalate,* $(COO)_2^{2-}$*; o-phthalate,* $C_6H_4(COO)_2^{2-}$*; succinate,* $(CH_2COO)_2^{2-}$)

A saturated solution of 5 mmol of dicarboxylic acid in dry methanol was added dropwise to a stirred methanolic solution of bis(*p*-methoxyphenyl)telluroxide (5 mmol). The contents upon stirring for about 2–3 hours yielded a white colored solid which was filtered, washed with methanol/chloroform and dried in vacuo over P_4O_{10}.

Synthesis of bis(p-hydroxyphenyl)tellurium(IV) carboxylates

$R_2Te[L]_2$ (*R = p-hydroxyphenyl and L = salicylate,* $C_6H_4(OH)COO^-$)

A saturated solution of 2 mmol of salicylic acid in dry methanol was added to a suspension of bis(*p*-hydroxyphenyl)telluroxide (1 mmol). The clear red solution thus obtained was stirred for 3–4 hours. Turbidity if any, was filtered off and the clear solution was concentrated. This resulted in the separation of an orange colored solid, which was filtered, washed with chloroform and dried in a vacuum desiccator over P_4O_{10}.

$R_2Te[L_2]$ (*R = p-hydroxyphenyl),* $[L_2]$ *= oxalate,* $(COO)_2^{2-}$*; o-phthalate,* $C_6H_4(COO)_2^{2-}$)

To a stirred suspension of bis(*p*-hydroxyphenyl)telluroxide (2 mmol) in dry methanol a saturated solution of carboxylic acid (oxalic acid / *o*-phthalic acid) in methanol was added dropwise. This yielded a clear orange solution. The contents were stirred for about 2–3 hours, concentrated to about one third of original volume and kept in a vacuum desiccator overnight. An orange colored solid separated out. This was filtered off, washed with chloroform and dried in vacuo.

The purity of these compounds was checked by T.L.C. using silica gel-G. The analytical data and physical properties of the complexes are presented in Table III.

TABLE III Physical characteristics and elemental analyses of diaryltellurium(IV) carboxylates

R	*[L] / [L$_2$]*	*Compound (Empirical Formula)*	*Color*	*M.P. °(C)*	*Yield (%)*	*Elemental Analysis (%)*		
						C Found (Calcd)	*H Found (Calcd)*	*Te Found (Calcd)*
p-$CH_3OC_6H_4$	$C_6H_4(OH)COO$	$R_2Te\ [L]_2$ ($C_{28}H_{24}O_8Te$)	White	195–196	75	54.18 (54.59)	3.48 (3.93)	21 05 (20.71)
	C_6H_5COO	$R_2Te\ [L]_2$ ($C_{28}H_{24}O_6Te$)	White	207–208	>80	56.98 (57.58)	3.82 (4.14)	21.13 (21.84)
	$C_6H_5CHCHCOO$	$R_2Te\ [L]_2$ ($C_{22}H_{18}O_6Te$)	White	155–157	80	60.10 (60.41)	4.02 (4.44)	20.74 (20.06)
	C_2O_4	$R_2Te\ [L_2]$ ($C_{16}H_{14}O_6Te$)	White	165–166	80	44.34 (44.70)	3.00 (3.28)	29.30 (29.68)
	$C_6H_4(COO)_2$	$R_2Te\ [L_2]$ ($C_{22}H_{18}O_6Te$)	White	167–169	>60	51.89 (52.22)	3.14 (3.59)	25.52 (25.22)
	$(CH_2COO)_2$	$R_2Te\ [L_2]$ ($C_{18}H_{18}O_6Te$)	White	> 250	90	46.92 (47.22)	3.45 (3.96)	28.11 (27.87)
p-HOC_6H_4	$C_6H_4(OH)COO$	$R_2Te\ [L]_2$ ($C_{26}H_{20}O_8Te$)	Light orange	175–178[a]	65	52.56 (53.11)	3.06 (3.43)	21.69 (21.70)
	C_2O_4	$R_2Te\ [L_2]$ ($C_{14}H_{10}O_6Te$)	Light orange	136–138[a]	60	42.02 (41.85)	2.22 (2.51)	31.26 (31.76)
	$C_6H_4(COO)_2$	$R_2Te\ [L_2]$ ($C_{20}H_{14}O_6Te$)	Light orange	145–146	65	49.74 (50.27)	2.49 (2.95)	26.39 (26.70)

[a] Decompose.

Analytical Methods and Physical Measurements

Carbon and hydrogen analyses were obtained microanalytically from the Regional Sophisticated Instrumentation Centre, Panjab University, Chandigarh. The tellurium content was estimated volumetrically[31]. Conductivity was measured at 35 ± 1°C with a conductivity bridge type 305 Systronics model and the molecular weights were determined by the cryoscopic method in nitrobenzene. IR and far IR spectra were recorded at Regional Sophisticated Instrumentation Centre; Indian Institute of Technology, Chennai on a BRUKER IFS 66v FT-IR spectrometer using KBr pellets/Polyethypellets techniques. ^{1}H NMR spectra were recorded at Regional Sophisticated Instrumentations Centre, Indian Institute of Technology, Bombay on a BRUKER DPX-300 spectrometer operating at 300 MHz in $CDCl_3$/DMSO-d_6 using tetramethylsilane as an internal reference.

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