

Synthesis and Characterization of Aminophenyl (p-hydroxyphenyl)Tellurium(IV) Dichlorides

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Abstract

The synthesis and characterization of a number of aminophenyl(p-hydroxyphenyl)tellurium(IV) dichlorides with the general formula $RR'TeCl_2$ —where R stands for the p-hydroxyphenyl group and R' denotes substituted amino aromatic moieties like aniline, N-methylaniline, and N,N-dimethylaniline—are reported in this study. Because of their distinctive structural characteristics, adaptable bonding behavior, and prospective uses in organic synthesis, catalysis, and medicinal chemistry, organotellurium compounds have garnered a lot of interest. The coordination behavior and electrical characteristics of these compounds are further improved by the addition of hydroxy and amino functional groups to the tellurium framework. Proton magnetic resonance (PMR) spectroscopy, elemental analysis, conductance measurements, cryoscopic studies, and far-infrared spectroscopy were used to characterize the synthesized tellurium (IV) dichlorides that were obtained by appropriate synthetic procedures. Low molar conductance values from conductance studies in suitable solvents suggested that the synthesized compounds in solution were molecular and non-electrolytic. The suggested stoichiometry of the compounds was validated, and the monomeric behavior was further corroborated by cryoscopic molecular weight measurements. Significant structural information on the coordinating environment surrounding the tellurium core was obtained from far-infrared spectral studies. The para-substitution of the amino aromatic moiety to $RTeCl_2$ was established based on measured vibrational bands. Understanding the bonding properties between the tellurium atom and the organic moiety was greatly aided by PMR spectrum studies. The suggested molecular structures were corroborated by the chemical shift values and splitting patterns, which verified the existence of the substituted phenyl groups. For these aminophenyl (p-hydroxyphenyl)tellurium (IV) dichlorides, a pseudo-trigonal-bipyramidal geometry has been proposed based on the combined analytical and spectral evidence. The research provides important new information about the bonding properties and structural chemistry of organotellurium compounds with amino-substituted aromatic ligands.

Keywords: Organotellurium compounds; Tellurium(IV) dichlorides; Aminophenyl derivatives; PMR spectroscopy; far-infrared spectroscopy; trigonal-bipyramidal geometry

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INTRODUCTION

Organotellurium (IV) halides are an important class of organometallic compounds owing to their varied structural characteristics, distinct reactivity, and wide range of applications in coordination and synthetic chemistry. Grignard reagents, elemental Te, Te (IV) halides, and other organometallic derivatives can be used as starting materials to create these compounds. Because of their high reactivity and ease of handling, tellurium tetrahalides, especially tellurium tetrachloride ($TeCl_4$), are considered the most used and

synthetically significant reagents among these precursors [1]. Although elemental tellurium is helpful in some synthetic processes, it is not as significant as organotellurium compounds as a starting material. Petraghani thoroughly studied the condensation reactions of several aryltellurium trihalides, such as p-anisyl-, p-ethoxyphenyl-, and p-phenoxyphenyltellurium trihalides, with substances including acetone, acetophenone, N,N-dimethylaniline, and resorcinol. Through the interaction of aryltellurium trichlorides and tribromides, these processes successfully generated unsymmetrical diorganyltellurium dihalides. Nevertheless, under comparable circumstances, aryltellurium triiodides were found to be relatively unreactive, suggesting that the halogen atom affects the reactivity and condensation behavior of organotellurium intermediates [2]. The present study investigates the interactions between p-hydroxyphenyltellurium(IV) trichloride and aminophenyl compounds, including aniline, N-methylaniline, and N,N-dimethylaniline. The goal of these reactions is to create a novel class of aminophenyl (p-hydroxyphenyl)tellurium (IV) dichlorides with the general formula $RR'TeCl_2$. The synthesized compounds are expected to provide important insights into the structural traits, bonding behavior, and reactivity patterns of amino-substituted organotellurium derivatives.

EXPERIMENTAL

Reagents

Tellurium tetrachloride was obtained from E. Merck and purified using standard methods. Aniline, N-methylaniline, and N,N-dimethylaniline were procured from Fluka AG (Switzerland). Phenol was obtained from BDH.

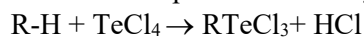
Preparations

Aminophenyls, such as aniline, N-methylaniline, and N, N-dimethylaniline, were stirred with p-hydroxyphenyltellurium(IV) trichloride for approximately 48 h in a dry nitrogen environment to yield the corresponding aminophenyl(p-hydroxyphenyl)tellurium(IV) dichlorides, $RR'TeCl_2$ (where R = p-hydroxyphenyl; R' = aniline, N-methylaniline, and N, N-dimethylaniline). The products were extracted with methanol and recrystallized with acetonitrile.

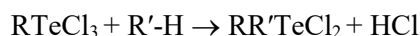
RESULT AND DISCUSSIONS

Synthesis

Phenol appears to undergo Friedel-Crafts-type condensation reactions with tellurium tetrachloride, whereby the trichlorotellurium unit, $TeCl_3^+$, attacks a position para to the electron-releasing hydroxy group, yielding p-hydroxyphenyltellurium(IV) trichloride [3]. The reaction of hydroxyaryltellurium(IV) trichloride with anilines requires a prolonged reaction time and proceeds with the elimination of HCl, resulting in the formation of unsymmetrical diorganyltellurium(IV) dichlorides. These reactions proceed similarly to the reaction of tellurium tetrachloride with aromatic compounds.



(R = Phenol)



(R = Phenol; R' = aniline, N-methylaniline, N, N-dimethylaniline)

Conductance and Cryoscopic Studies

Molar conductance studies in nitrobenzene, acetonitrile, and acetone reflect the molecular nature of the compounds in solution, except for p-dimethylaminophenyl(p-hydroxyphenyl)tellurium(IV) dichloride, which shows partial dissociation in acetonitrile and acetone. This observation is supported by cryoscopic studies in nitrobenzene [4].

IR and Far-IR Studies

The asymmetric and symmetric N-H stretching vibrations, expected at approximately 3450 and 3330 cm^{-1} , respectively, do not appear as separate bands but are mixed with $\nu O-H$ as a broad band. The in-plane N-H bending appears at 1620 cm^{-1} (mixed with skeletal vibrations) in the aniline derivative. The

ν C-N stretching vibrations, which appear at 1280 cm^{-1} and 1350 and 1240 cm^{-1} in aniline and N,N-dimethylaniline, respectively, are shifted to higher frequencies, indicating linkage at the position para to $-\text{NH}_2$ or $\text{N}(\text{CH}_3)_2$ of the aromatic ring. The appearance of a strong intensity band at $\sim 820\text{ cm}^{-1}$ (C-H out-of-plane bending) and $\sim 1580\text{ cm}^{-1}$ ($\nu\text{C}=\text{C}$) confirms the para-substitution.

Fritz and Keller were the first to investigate the spectra of organotellurium compounds in this region and assigned bands between 487 and 455 cm^{-1} to tellurium-phenyl vibrations [5]. Later, McWhinnie reassigned bands near $260\text{--}200\text{ cm}^{-1}$ to the tellurium-phenyl vibrations [6]. Based on IR studies, it is suggested that these dichlorides have a ψ -trigonal-bipyramidal geometry with two halogen atoms trans to each other, as reported for tellurium derivatives [7, 8].

PMR Studies

The PMR data predict the linkage of Te to the carbon atom para to the OH and para to the aminophenyl moiety, as reported for aniline trichlorides [9, 10]. It also helps establish the stoichiometry of these compounds, as shown in Tables 1–5.

Table 1. Analytical data and physical properties.

R	R'	Empirical formula	Analysis found (calculated) %					M.P. (°C)	Color	Yield **
			Te	Cl	C	H	N			
p-hydroxyphenyl	p-aminophenyl	$\text{C}_{12}\text{H}_{11}\text{NO}$	32.54	17.88	36.80	2.44	3.24	110-	Brown	50
		TeCl_2	(33.26)	(18.51)	(37.54)	(32.87)	(3.65)	115		
p-hydroxyphenyl	p-methylaminophenyl	$\text{C}_{13}\text{H}_{13}\text{NO}$	31.63	17.02	38.42	2.75	2.97	*	Purple	52
		TeCl_2	(32.09)	(17.86)	(39.24)	(3.27)	(3.52)			
p-hydroxyphenyl	p-dimethylaminophenyl	$\text{C}_{12}\text{H}_{15}\text{NO}$	30.71	16.67	39.60	3.08	2.96	125-	Greyish	68
		TeCl_2	(31.00)	(17.25)	(40.82)	(3.64)	(3.40)	127	Green	

Table 2. Conductance data.

R	R'	Compound	Nitrobenzene		Acetonitrile		Acetone	
			Molar	Molar	Molar	Molar	Molar	Molar
			Conc.C $\times 10^3$ mol L^{-1}	Conductance $\Lambda_{\text{M}}\text{ohm}^{-1}\text{ cm}^2$ mol^{-1}	Conc.C $\times 10^3$ mol L^{-1}	Conductance $\Lambda_{\text{M}}\text{ohm}^{-1}\text{ cm}^2$ mol^{-1}	Conc.C $\times 10^3$ mol L^{-1}	Conductance $\Lambda_{\text{M}}\text{ohm}^{-1}\text{ cm}^2$ mol^{-1}
p-hydroxyphenyl	p-aminophenyl	RR/TeCl ₂	*	-	**	16.80	1.00	5.70
						13.29	1.67	5.03
						12.62	2.14	4.77
						12.00	2.50	4.56
						11.72	2.71	4.42
p-hydroxyphenyl	p-methylaminophenyl	RR'/TeCl ₂	1.00	2.22	**	13.80	**	4.98
			1.67	1.37		9.34		4.31
			2.14	1.23		8.41		3.93
			2.50	1.13		7.92		3.60
			2.71	1.08		7.74		3.54
p-hydroxyphenyl	p-dimethylaminophenyl	RR'/TeCl ₂	1.00	14.40	1.00	108.00	1.00	90.00
			1.67	14.01	1.67	93.41	1.67	68.26
			2.14	13.74	2.14	92.52	2.14	67.29
			2.50	13.44	2.50	91.20	2.50	66.00
			2.71	12.84	2.71	90.77	2.71	65.22

*Insoluble in the solvent, **Sparingly soluble in the solvent

Value reported for 1:1 electrolyte, Nitrobenzene = 20-30, Acetone = 100-140, Acetonitrile 120-160

Table 3. Cryoscopic data.

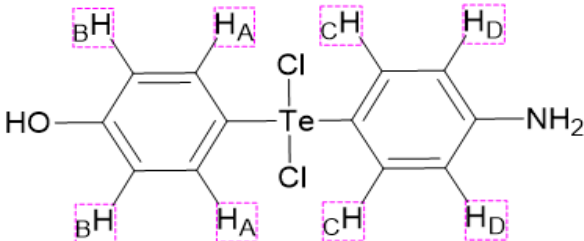
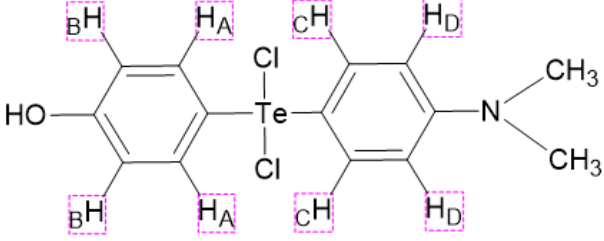
R	R'	Compound	Formula weight	Conc. in mmoles/liter of solvent	Molecular weight
p-hydroxyphenyl	p-aminophenyl	RR'TeCl ₂	383.6	Insoluble	-
p-hydroxyphenyl	p-methylaminophenyl	RR'TeCl ₂	397.6	2.515	336.9
				5.198	348.2
				8.048	359.4
				11.905	398.7
p-hydroxyphenyl	p-dimethylaminophenyl	RR'TeCl ₂	411.6	2.592	359
				5.507	381.9
				8.589	396.8
				11.986	415.6

Table 4. IR and Far-IR data (cm⁻¹).

RR'TeCl ₂	RR'TeCl ₂	RR'TeCl ₂	Assignments	
<i>R</i> = <i>p</i> -hydroxyphenyl <i>R'</i> = <i>p</i> -aminophenyl	<i>R</i> = <i>p</i> -hydroxyphenyl <i>R'</i> = <i>p</i> -methylaminophenyl	<i>R</i> = <i>p</i> -hydroxyphenyl <i>R'</i> = <i>p</i> -dimethylaminophenyl		
3420 mb	-	3418 wb	ν (O-H) + ν (N-H)	
2928 m	-	3067 w 3020 w	Aromatic C-H Stretch	
-	-	2924 m	Aliphatic CH ₃ stretch	
1871 w 1747 w 1701 w 1688 w	-	1959 vw 1882 vw 1798 vw 1747 vw	Overtones and Combination bands	
1620 w	-	-	N-H in-plane bending	Ring Stretch Vibrations
1578 s	-	1574 s	ν C=C	
1542 m 1493 s	-	1489 s		
		1462 m	CH ₃ bending Vibrations	
1423 w	-	1420 m		
1362 w	-	1362 w	δ(OH)	
1339 w	-	1330 w	ν(C-N)	
1269 m	-	1269 w	ν(C-O)	
1242 m 1177 m 1130 w 1057 w	-	1246 w 1177 m 1126 m 1053 w 1029 sh 991 m	Aromatic in-plane vibrations	
903 w 822 s 737 m	-	899 m 833 m 768 s	C-H out-of-plane bending vibrations	
606 sb	-	694 s 648 w 575 w 540 m	Out-of-plane ring-bending vibrations	

471 w 460 m	471 w 448 m	475 m 450 m	Phenyl modes
290 w 268 w	290 m 270 s	287 m	ν_s Te-X
252 w	260 w	253 s	ν_{as} Te-X
235 w	240 m 230 w	235 m 215 s	ν Te-Ph
145 w 118 w	162 w 117 m	163 w 152 w 129 wb 93 w	δ Te-Cl ₂

Table 5. PMR data.

Compounds	Solvent	δ ppm	Assignments
	DMSO-d ₆	3.33 (s, 2H) 6.91 (d, J = 9Hz, 2H) 7.74 (d, J = 9Hz, 2H) 6.49-6.67 (m) 4H 7.01-7.76 (m) 4H (10.08 b, s, 1H)	-NH ₂ mixed with solvent peak -HbB-Ha-Hc-Hd-OH
	DMSO-d ₆	3.05 (s, 6H) 6.84 (d, J = 9Hz, 2H) 8.22 (d, J = 9Hz, 2H) 7.13-7.43 (m, 5H)	-N (CH ₃) ₂ Hb-Ha -Hc, -Hd, -OH

CONCLUSION

The present study successfully demonstrates the synthesis of aminophenyl(p-hydroxyphenyl)tellurium(IV) dichlorides through the condensation of p-hydroxyphenyltellurium(IV) trichloride with various substituted anilines. The structural characterization, supported by conductance, cryoscopic, IR, far-IR, and PMR studies, confirmed the formation of unsymmetrical diorganotellurium(IV) dichlorides with a suggested pseudo-trigonal-bipyramidal geometry. The spectral data validate the para-substitution of the Te center on both aromatic moieties, and the PMR results reinforce the proposed stoichiometry. The partial ionic character observed in some derivatives highlights the influence of substituents on their dissociation behaviors. These findings contribute to a deeper understanding of the reactivity and structural features of organotellurium(IV) compounds, laying the groundwork for further exploration of their potential applications in coordination chemistry and materials science.

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