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Aryltellurium(IV) trichlorides, aryl(*trans*-2-chloro-1-cycloalkyl)-tellurium(IV) dichlorides, and aryl(*trans*-2-ethoxy-1-cycloalkyl)-tellurium(IV) dichlorides

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Abstract

Aryltellurium(IV) trichlorides react with parent arenes to give diaryltellurium(IV) dichlorides and with cycloalkenes to give aryl(*trans*-2-chloro-1-cycloalkyl)tellurium(IV) dichlorides or aryl(*trans*-2-ethoxy-1-cycloalkyl)tellurium(IV) dichlorides in the presence of ethanol.

Introduction

Tellurium tetrachloride behaves as trichlorotellurium(IV) chloride in substitution reactions with benzene and its derivatives which give aryltellurium(IV) trichlorides [1, 2] and addition reactions with cycloalkenes which give *trans*-2-chloro-1-cyclohexyltellurium(IV) trichlorides [3] or *trans*-2-alkoxy-1-cycloalkyltellurium(IV) trichlorides in the presence of alcohols [4]. Aryltellurium(IV) trichlorides and their addition reactions with cycloalkenes [3] are discussed here. Some new organotellurium compounds have been prepared.

Results and discussion

Solutions of tellurium tetrachloride in chloroform react readily with derivatives of benzene containing an electron-supplying substituent [1]. The ^{1}H NMR spectra of the resulting aryltellurium(IV) trichlorides contain aryl AA'BB' signals characteristic of 1,4-disubstituted benzenes [2, 5]. There is no evidence of 1,2- or 1,3-disubstition, presumably for steric and polar reasons, respectively. Similar reactions with benzene, chlorobenzene and bromobenzene benefit from the addition of anhydrous aluminium chloride but reaction with iodobenzene gives *p*-di-iodobenzene. Tellurium tetrachloride does not appear to react with nitrobenzene even on prolonged boiling with catalyst in the absence of solvent.

Tellurium tetrachloride reacts with *meta*- and *ortho*-dimethoxybenzenes to give 2,4- and 3,4-dimethoxyphenyltellurium(IV) trichlorides, respectively. The ^{1}H NMR spectra of the products contain clear ABX signals. The 2,4-dimethoxyphenyl derivative [6] contains an aryl A proton (*ortho* to tellurium) which is coupled with the aryl B proton (*meta* to tellurium) to give a doublet ($^3J = c.$ 9 Hz) at low field; B is coupled also with the aryl X proton (*meta* to tellurium) to give a doublet of doublets ($^4J = c.$ 2 Hz) at higher field; and X appears as a doublet at highest field. The 3,4-dimethoxyphenyl derivative contains an aryl A proton (*ortho* to tellurium) which is coupled with aryl protons B (*meta* to tellurium) and X (*ortho* to tellurium) to give a doublet of doublets at low field; B appears as a doublet at higher field; and X appears as a doublet at slightly higher field than A.

Tellurium tetrachloride reacts with 2,6-dimethylphenol to give 3,5-dimethyl-4-hydroxyphenyltellurium(IV) trichloride. The ^{1}H and ^{13}C NMR spectra show non-equivalent aryl protons and non-equivalent methyl groups, consistent with restricted rotation about carbon–tellurium.

Aryltellurium(IV) trihalides react with an excess of the parent benzene derivative under more vigorous conditions to give diaryltellurium(IV) dihalides with both aryl groups having the same substitution pattern. The NMR spectra show aryl A protons [2] and carbons *ortho* to tellurium at higher fields than for corresponding aryltellurium(IV) trichlorides. The trichlorides and dichlorides are distinguished also by mass spectra [7] where molecular-ion peaks correspond to fragments which have lost one chlorine,

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TABLE 1. ^{1}H NMR signals for H–CTe (A) and H–CCl or H–COEt (B) cycloalkyl protons of aryl(2-chloro-1-cycloalkyl)tellurium(IV) dichlorides and aryl(2-ethoxy-1-cycloalkyl)tellurium(IV) dichlorides in CDCl$_3$

Compound	H–CTe δ(A) (ppm)	H–CCl δ(B) (ppm)	^3J(A–D) (Hz)	^3J(A–B) ^3J(A–C) (Hz)	^3J(B–E) (Hz)	^3J(B–F) (Hz)
p-MeOC$_6$H$_4$(ClC$_6$H$_{10}$)TeCl$_2$	4.19	4.65	3.9	11.5	11.5	4.5
p-PhOC$_6$H$_4$(ClC$_6$H$_{10}$)TeCl$_2$	4.21	4.66	4.6	11.4	11.4	4.4
p-HOC$_6$H$_4$(ClC$_6$H$_{10}$)TeCl$_2$	4.19	4.65	4.6	11.4	11.4	4.5
p-ClC$_6$H$_4$(ClC$_6$H$_{10}$)TeCl$_2$	4.24	4.66	5.3	11.2	11.2	4.5
2,4-(MeO)$_2$C$_6$H$_3$(ClC$_6$H$_{10}$)TeCl$_2$	4.35	4.67	3.7	11.4	11.4	4.4
3,4-(MeO)$_2$C$_6$H$_3$(ClC$_6$H$_{10}$)TeCl$_2$	4.17	4.65	3.9	11.5	11.5	4.4
3,5-Me$_2$-4-HOC$_6$H$_2$(ClC$_6$H$_{10}$)TeCl$_2$	4.16	4.66	4.5	11.5	11.5	4.5
p-MeOC$_6$H$_4$(ClC$_7$H$_{12}$)TeCl$_2$	4.28	4.93	2.8	10.2	10.2	4.2
p-PhOC$_6$H$_4$(ClC$_7$H$_{12}$)TeCl$_2$	4.32	4.97	3.2	10.0	10.0	4.0
2,4-(MeO)$_2$C$_6$H$_3$(ClC$_7$H$_{12}$)TeCl$_2$	4.42	4.98	2.2	9.8	7.4	4.5
3,4-(MeO)$_2$C$_6$H$_3$(ClC$_7$H$_{12}$)TeCl$_2$	4.26	4.91	3.0	10.6	8.7	4.1
3,5-Me$_2$-4-HOC$_6$H$_2$(ClC$_7$H$_{12}$)TeCl$_2$	4.24	4.90	3.0	10.5	8.7	4.0
		H–COEt				
p-MeOC$_6$H$_4$(EtOC$_6$H$_{10}$)TeCl$_2$	3.86	4.05	[a]	[a]	[a]	[a]
p-PhOC$_6$H$_4$(EtOC$_6$H$_{10}$)TeCl$_2$	3.92	4.01	4.0	10.6	12.3	4.1
p-MeOC$_6$H$_4$(EtOC$_7$H$_{12}$)TeCl$_2$	3.88	4.21	4.7	10.2	10.2	5.5
p-PhOC$_6$H$_4$(EtOC$_7$H$_{12}$)TeCl$_2$	3.93	4.24	4.9	9.1	10.4	6.5

[a] Coupling constants could not be measured accurately.

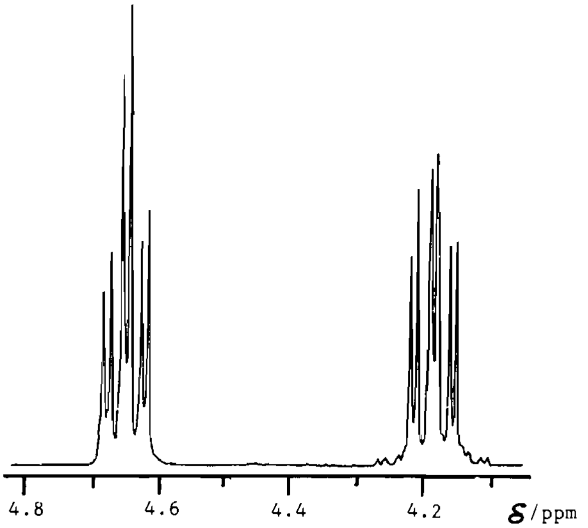

Fig. 1. ^{1}H NMR signals for A and B protons of *p*-anisyl(*trans*-2-chloro-1-cyclohexyl)tellurium(IV) dichloride in CDCl$_3$.

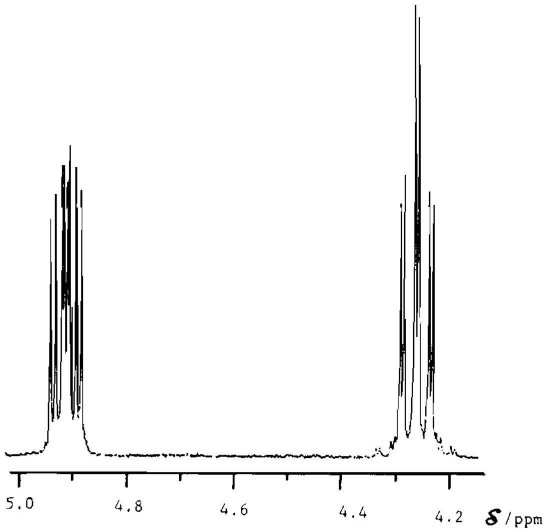

Fig. 2. ^{1}H NMR signals for A and B protons of 3,4-dimethoxyphenyl(*trans*-2-chloro-1-cycloheptyl)tellurium(IV) dichloride in CDCl$_3$.

viz. ArTeCl$_2$ and Ar$_2$TeCl. Attempts to prepare triaryltellurium(IV) chlorides by this type of reaction were unsuccessful.

Aryltellurium(IV) trichlorides and diaryltellurium(IV) dichlorides [1] are reduced to diaryl ditellurides and diaryl tellurides, respectively. Further reactions with sulfuryl chloride, dibromine or diiodine give the corresponding trihalides and dihalides. Diaryl ditellurides react with methyl iodide to give mixtures of aryl(dimethyl)tellurium iodides and aryl(methyl)tellurium di-iodides which are separated by crystallisation. Aryltellurium trichlorides are converted into tribromides or tri-iodides by potassium bromide or iodide in acetone–water.

Cycloalkenes

Campos and Petragnani [3] reported reactions of some aryltellurium(IV) trichlorides with cyclohexene.

TABLE 2. ^{13}C NMR signals for cycloalkyl ring carbons of aryl(2-chloro-1-cycloalkyl)tellurium(IV) dichlorides and aryl(2-ethoxy-1-cycloalkyl)tellurium(IV) dichlorides in $CDCl_3$

Compound	δ (ppm)						
	C–Te	C–Cl	Other cycloalkyl carbons				
p-$MeOC_6H_4(ClC_6H_{10})TeCl_2$	75.29	60.73	37.83	28.76	26.41	25.44	
p-$PhOC_6H_4(ClC_6H_{10})TeCl_2$	75.43	60.49	37.90	28.91	26.51	25.33	
p-$HOC_6H_4(ClC_6H_{10})TeCl_2$	75.38	60.66	37.83	28.75	26.41	25.40	
p-$ClC_6H_4(ClC_6H_{10})TeCl_2$	76.20	60.53	37.67	28.85	26.34	25.28	
$2,4$-$(MeO)_2C_6H_3(ClC_6H_{10})TeCl_2$	73.64	60.59	38.16	29.08	26.37	25.58	
$3,4$-$(MeO)_2C_6H_3(ClC_6H_{10})TeCl_2$	75.54	60.71	37.84	28.85	26.43	25.43	
$3,5$-Me_2-4-$HOC_6H_2(ClC_6H_{10})TeCl_2$	74.95	60.70	37.82	28.71	26.38	25.41	
p-$MeOC_6H_4(ClC_7H_{12})TeCl_2$	77.32	62.27	38.27	29.19	28.95	27.32	22.41
p-$PhOC_6H_4(ClC_7H_{12})TeCl_2$	77.79	62.34	39.23	29.93	28.72	27.53	22.89
$2,4$-$(MeO)_2C_6H_3(ClC_7H_{12})TeCl_2$	73.82	63.20	38.62	29.58	28.19	27.40	22.72
$3,4$-$(MeO)_2C_6H_3(ClC_7H_{12})TeCl_2$	78.36	63.69	37.82	28.47	27.30	26.37	22.71
$3,5$-Me_2-4-$HOC_6H_2(ClC_7H_{12})TeCl_2$	77.19	62.20	38.29	29.27	28.93	27.34	22.53
		C–OEt					
p-$MeOC_6H_4(EtOC_6H_{10})TeCl_2$	73.07	77.47	32.51	27.40	26.89	23.61	
p-$PhOC_6H_4(EtOC_6H_{10})TeCl_2$	73.39	77.45	32.51	27.43	26.97	23.58	
p-$MeOC_6H_4(EtOC_7H_{12})TeCl_2$	76.52	79.76	32.50	26.94	26.48	25.94	22.97
p-$PhOC_6H_4(EtOC_7H_{12})TeCl_2$	76.62	79.81	32.50	27.00	26.50	26.00	23.00

We have investigated addition reactions of various trihalides with cyclohexene and cycloheptene (and one reaction with cyclopentene). The X-ray crystal structures of p-anisyl(*trans*-2-chloro-1-cyclohexyl)-tellurium(IV) dichloride [8] and p-tolyl(*trans*-2-chloro-1-cyclohexyl)tellurium(IV) dichloride [9] confirm that 1,2-*trans* addition has occurred. The cyclohexyl rings have chair conformations with adjacent equatorial positions occupied by tellurium and chlorine.

Tellurium tetrahalides react with cycloalkenes and alcohols to give 2-*trans*-alkoxy-1-cycloalkyltellurium(IV) trihalides [4, 10] but attempted reactions of aryltellurium trichlorides with cyclohexene or cycloheptene in the presence of methanol, n-propanol or i-propanol gave aryl(*trans*-2-chloro-1-cycloalkyl)-tellurium(IV) dichlorides. Two aryltellurium(IV) trichlorides reacted with cycloheptene and ethanol to give aryl(*trans*-2-ethoxy-1-cycloheptyl)tellurium-(IV) dichlorides, presumably by nucleophilic attack of ethoxide on cyclic aryltelluronium intermediates. Similar reactions with cyclohexene and ethanol gave approximately equimolar mixtures of aryl(*trans*-2-chloro-1-cyclohexyl)tellurium(IV) dichloride and aryl(*trans*-2-ethoxy-1-cyclohexyl)tellurium(IV) dichloride for which NMR data are obtained by difference.

Proton NMR spectra

The ^1H NMR spectra of these dichlorides show aryl protons at low field and cycloalkyl methylene protons at high field. Methoxy, methyl and hydroxy substituents of aryl groups appear as singlets. There is no indication of restricted rotation in 3,5-dimethyl-4-hydroxyphenyl(*trans*-2-chloro-1-cycloalkyl)tellurium(IV) dichlorides. Methyl protons of ethoxycycloalkyl derivatives appear as triplets and the non-equivalent methylene protons as quartets of AB signals. Data for protons which were alkenic originally are summarised in Table 1.

Cycloalkyl A protons (H–CTe) in aryl(*trans*-2-chloro-1-cycloalkyl)tellurium(IV) dichlorides appear at higher fields and show greater variations in chemical shifts than cycloalkyl B protons (H–CCl). These A and B protons are coupled with each other and with neighbouring cycloalkyl CH_2 protons so that the A protons appear as six-line multiplets (doublets of triplets) with similar *trans*-diaxial A–B and A–C coupling constants which are greater than *cis* A–D coupling constants. The cycloalkyl B protons appear also as six-line multiplets or as eight-line multiplets (doublets of doublets of doublets) when the three coupling constants are different (*trans*-diaxial A–B > *trans*-diaxial B–E > *cis* B–F). The splitting patterns for A and B protons of p-anisyl(*trans*-2-chloro-1-cyclohexyl)tellurium(IV) dichloride and 3,4-dimethoxyphenyl(*trans*-2-chloro-1-cycloheptyl)tellurium(IV) dichloride are compared in Figs. 1 and 2.

The cycloalkyl A protons in aryl(*trans*-2-ethoxy-1-cycloalkyl)tellurium(IV) dichlorides appear as six-line multiplets at higher field than the cycloalkyl B

TABLE 3. Analysis of organotellurium compounds

Compound	Melting point (°C)	Found (%)			Formula	Calculated (%)		
		C	H	Hal		C	H	Hal
$p\text{-}\mathrm{MeOC_6H_4TeCl_3}$	191[a]	24.48	2.04	30.85	$\mathrm{C_7H_7Cl_3OTe}$	24.65	2.07	31.18
$p\text{-}\mathrm{PhOC_6H_4TeCl_3}$	157[b]	35.65	2.22	26.48	$\mathrm{C_{12}H_9Cl_3OTe}$	35.75	2.25	26.38
$p\text{-}\mathrm{HOC_6H_4TeCl_3}$	224[c]	21.85	1.64	32.07	$\mathrm{C_6H_5Cl_3OTe}$	22.03	1.54	32.52
$p\text{-}\mathrm{ClC_6H_4TeCl_3}$	235	20.69	1.14	41.23	$\mathrm{C_6H_4Cl_4Te}$	20.86	1.17	41.04
$\mathrm{2,4\text{-}(MeO)_2C_6H_3TeCl_3}$	150–151[d]	25.63	2.43	29.00	$\mathrm{C_8H_9Cl_3O_2Te}$	25.89	2.44	28.66
$\mathrm{3,4\text{-}(MeO)_2C_6H_3TeCl_3}$	175	25.60	2.43	28.49	$\mathrm{C_8H_9Cl_3O_2Te}$	25.89	2.44	28.66
$\mathrm{3,4\text{-}(HO)_2C_6H_3TeCl_3}$	194	20.95	1.50	29.74	$\mathrm{C_6H_5Cl_3O_2Te}$	21.01	1.47	31.00
$\mathrm{3,4\text{-}Me_2C_6H_3TeCl_3}$	193–195	28.90	2.85	30.71	$\mathrm{C_8H_9Cl_3Te}$	28.33	2.68	31.36
$\mathrm{3,5\text{-}Me_2\text{-}4\text{-}HOC_6H_2TeCl_3}$	185(dec.)	26.85	2.58	29.69	$\mathrm{C_8H_9Cl_3OTe}$	27.06	2.54	29.95
$p\text{-}\mathrm{ClC_6H_4TeBr_3}$	250–252	15.07	0.80	50.15 7.00	$\mathrm{C_6H_4Br_3ClTe}$	15.05	0.84	50.06 7.40
$p\text{-}\mathrm{BrC_6H_4TeBr_3}$	234–236	14.02	0.81	60.00	$\mathrm{C_6H_4Br_4Te}$	13.77	0.77	61.08
$\mathrm{3,4\text{-}(MeO)_2C_6H_3TeBr_3}$	179	18.36	1.74	48.77	$\mathrm{C_8H_9Br_3O_2Te}$	19.05	1.80	47.52
$\mathrm{3,5\text{-}Me_2\text{-}4\text{-}HOC_6H_2TeBr_3}$	190	19.72	1.81	49.49	$\mathrm{C_8H_9Br_3OTe}$	19.67	1.86	49.07
$p\text{-}\mathrm{ClC_6H_4TeI_3}$	190–194	11.84	0.62	6.06 61.70	$\mathrm{C_6H_4ClI_3Te}$	11.63	0.65	5.72 61.42
$p\text{-}\mathrm{BrC_6H_4TeI_3}$	190–193	10.88	0.64	13.17 53.67	$\mathrm{C_6H_4BrI_3Te}$	10.85	0.61	12.03 57.31
$p\text{-}\mathrm{HOC_6H_4)_2TeBr_2}$	218–220	30.87	2.25	34.34	$\mathrm{C_{12}H_{10}Br_2O_2Te}$	30.43	2.13	33.74
$\mathrm{[3,4\text{-}(MeO)_2C_6H_3]_2TeBr_2}$	103	33.40	3.26	28.69	$\mathrm{C_{16}H_{18}Br_2O_4Te}$	34.21	3.23	28.45
$\mathrm{(3,4\text{-}Me_2C_6H_3)_2TeBr_2}$	138–140	37.80	3.40	32.10	$\mathrm{C_{16}H_{18}Br_2Te}$	38.61	3.65	32.11
$(p\text{-}\mathrm{ClC_6H_4)_2TeI_2}$	188–190	23.95	1.38	14.37 39.35	$\mathrm{C_{12}H_8Cl_2I_2Te}$	23.84	1.33	11.73 41.99
$p\text{-}\mathrm{ClC_6H_4(Me)TeI_2}$	138–140	17.02	1.45	8.37 52.11	$\mathrm{C_7H_7ClI_2Te}$	16.55	1.39	6.98 49.96
$\mathrm{3,4\text{-}(MeO)_2C_6H_3(Me)TeI_2}$	148	20.60	2.41	47.84	$\mathrm{C_9H_{12}I_2O_2Te}$	20.26	2.27	47.57
$p\text{-}\mathrm{ClC_6H_4(Me)_2TeI}$	128–130	24.21	2.64	9.09 32.22	$\mathrm{C_8H_{10}ClITe}$	24.26	2.54	8.95 32.04
$p\text{-}\mathrm{BrC_6H_4(Me)_2TeI}$	128–130	22.28	2.46	21.48 24.56	$\mathrm{C_8H_{10}BrITe}$	21.81	2.29	18.14 28.80
$\mathrm{3,4\text{-}Me_2C_6H_3(Me)_2TeI}$	176–178	30.50	3.85	34.00	$\mathrm{C_{10}H_{15}ITe}$	30.82	3.88	32.56
$(p\text{-}\mathrm{ClC_6H_4Te)_2}$	102–104	30.18	1.74	15.08	$\mathrm{C_{12}H_8Cl_2Te_2}$	30.13	1.69	14.82
$\mathrm{[3,4\text{-}(MeO)_2C_6H_3Te]_2}$	126	36.85	3.81		$\mathrm{C_{16}H_{18}O_4Te_2}$	36.29	3.43	0.00
$\mathrm{(3,4\text{-}Me_2C_6H_3Te)_2}$	46–48	41.37	3.98		$\mathrm{C_{16}H_{18}Te_2}$	41.28	3.90	0.00
$\mathrm{[3,5\text{-}Me_2\text{-}4\text{-}HOC_6H_2Te]_2}$	89	38.79	3.59		$\mathrm{C_{16}H_{18}O_2Te_2}$	38.63	3.65	0.00
$\mathrm{[3,4\text{-}(MeO)_2C_6H_3]_2Te}$	104	47.74	4.43		$\mathrm{C_{16}H_{18}O_4Te}$	47.81	4.51	0.00

[a] Lit. [11] m.p. 196–197 °C, lit. [12] m.p. 190 °C. [b] Lit. [11] m.p. 156–157 °C. [c] Lit. [12] m.p. 213 °C. [d] Lit. [6] m.p. 155–166 °C.

TABLE 4. Aryl(*trans*-2-chloro-1-cycloalkyl)tellurium(IV) dichlorides and aryl(*trans*-2-ethoxy-1-cycloalkyl)tellurium(IV) dichlorides

Compound	Yield (%)	Melting point (°C)	Found (%)			Formula	Calculated (%)		
			C	H	Hal		C	H	Hal
p-$\mathrm{MeC_6H_4(ClC_5H_8)TeCl_2}$	73	85–87	36.87	3.91	27.32	$\mathrm{C_{12}H_{15}Cl_3Te}$	36.66	3.85	27.05
p-$\mathrm{MeOC_6H_4(ClC_6H_{10})TeCl_2}$	88	109–110	36.42	4.02	25.75	$\mathrm{C_{13}H_{17}Cl_3OTe}$	36.89	4.05	25.13
p-$\mathrm{PhOC_6H_4(ClC_6H_{10})TeCl_2}$	80	120[a]	44.10	3.88	22.10	$\mathrm{C_{18}H_{19}Cl_3OTe}$	44.55	3.95	21.92
p-$\mathrm{MeC_6H_4(BrC_6H_{10})TeBr_2}$	58	82–84	28.50	2.89	43.82	$\mathrm{C_{13}H_{17}Br_3Te}$	28.88	3.17	44.34
p-$\mathrm{ClC_6H_4(ClC_6H_{10})TeCl_2}$	29	120	33.19	3.24	32.91	$\mathrm{C_{12}H_{14}Cl_4Te}$	33.70	3.30	33.16
p-$\mathrm{BrC_6H_4(ClC_6H_{10})TeCl_2}$	62	110	30.48	2.95	16.23 22.56	$\mathrm{C_{12}H_{14}BrCl_3Te}$	30.53	3.00	16.93 22.53
2,4-$\mathrm{(MeO)_2C_6H_3(ClC_6H_{10})TeCl_2}$	69	139	36.92	4.17	23.78	$\mathrm{C_{14}H_{19}Cl_3O_2Te}$	37.10	4.23	23.47
3,4-$\mathrm{(MeO)_2C_6H_3(ClC_6H_{10})TeCl_2}$	67	124–125	36.96	4.15	24.01	$\mathrm{C_{14}H_{19}Cl_3O_2Te}$	37.10	4.23	23.47
3,5-$\mathrm{Me_2}$-4-$\mathrm{HOC_6H_2(ClC_6H_{10})TeCl_2}$	53	135–136	37.37	4.28	24.24	$\mathrm{C_{14}H_{19}Cl_3OTe}$	38.46	4.38	24.32
p-$\mathrm{MeOC_6H_4(ClC_7H_{12})TeCl_2}$	43	84–85	38.30	4.43	24.15	$\mathrm{C_{14}H_{19}Cl_3OTe}$	38.46	4.38	24.32
p-$\mathrm{PhOC_6H_4(ClC_7H_{12})TeCl_2}$	94	94	45.68	4.19	21.17	$\mathrm{C_{19}H_{21}Cl_3OTe}$	45.70	4.24	21.30
2,4-$\mathrm{(MeO)_2C_6H_3(ClC_7H_{12})TeCl_2}$	59	121	38.54	4.26	22.68	$\mathrm{C_{15}H_{21}Cl_3O_2Te}$	38.56	4.53	22.76
3,4-$\mathrm{(MeO)_2C_6H_3(ClC_7H_{12})TeCl_2}$	47	104	39.27	4.47	22.80	$\mathrm{C_{15}H_{21}Cl_3O_2Te}$	38.56	4.53	22.76
3,5-$\mathrm{Me_2}$-4-$\mathrm{HOC_6H_2(ClC_7H_{12})TeCl_2}$	52	130–131	40.46	4.65	22.37	$\mathrm{C_{15}H_{21}Cl_3OTe}$	39.92	4.69	23.57
p-$\mathrm{MeOC_6H_4(EtOC_6H_{10})TeCl_2}$, *p*-$\mathrm{MeOC_6H_4(ClC_6H_{10})TeCl_2}$	59	75–80	38.56	4.82	20.52	$\mathrm{C_{28}H_{39}Cl_5O_3Te_2}$	39.28	4.59	20.71
p-$\mathrm{PhOC_6H_4(EtOC_6H_{10})TeCl_2}$, *p*-$\mathrm{PhOC_6H_4(ClC_6H_{10})TeCl_2}$	71	115–120	46.37	4.36	18.82	$\mathrm{C_{38}H_{43}Cl_5O_3Te_2}$	46.56	4.42	18.08
p-$\mathrm{MeOC_6H_4(EtOC_7H_{12})TeCl_2}$	71	110	43.24	5.51	16.22	$\mathrm{C_{16}H_{24}Cl_2O_2Te}$	43.00	5.41	15.87
p-$\mathrm{PhOC_6H_4(EtOC_7H_{12})TeCl_2}$	54	95	49.87	5.16	14.13	$\mathrm{C_{21}H_{26}Cl_2O_2Te}$	49.56	5.15	13.93

[a] Lit. [3] m.p. 121–125 °C.

protons which form either six-line multiplets or eight-line multiplets when the three coupling constants are different (*trans*-diaxial B–E > *trans*-diaxial A–B > *cis* B–F). Both sets of signals appear at higher fields than corresponding signals in *trans*-2-chloro-1-cycloalkyltellurium(IV) trichlorides [4].

^{13}C *NMR spectra*

The ^{13}C NMR aryl signals in aryl(*trans*-2-chloro-1-cycloalkyl)tellurium(IV) and aryl(*trans*-2-ethoxy-1-cycloalkyl)tellurium(IV) dichlorides appear below δ 99 ppm with aryl C–Te at lowest and carbons *ortho* to tellurium at next lowest field. Chemically equivalent carbons produce single lines of greater intensity. Cycloalkyl methylene carbons appear above δ 30 ppm and carbons which were alkenic originally appear in mid-field. Cycloalkyl C–Te carbons in aryl(*trans*-2-chloro-1-cycloalkyl)tellurium(IV) dichlorides appear at lower fields and show greater variations of chemical shifts than C–Cl carbons. The signals are at higher fields in cyclohexyl than cycloheptyl derivatives. Cycloalkyl C–Te signals in aryl(*trans*-2-ethoxy-1-cycloalkyl)tellurium(IV) dichlorides appear at higher fields and show greater variations of chemical shifts than C–OEt signals. As expected, both sets of signals are at higher fields in aryl(*trans*-2-chloro-1-cycloalkyl)tellurium(IV) dichlorides than *trans*-2-ethoxy-1-cycloalkyltellurium(IV) trichlorides [4]. The ^{13}C NMR chemical shifts for cycloalkyl carbons in the dichlorides are recorded in Table 2.

Experimental

In a typical reaction, tellurium tetrachloride (11.70 g, 43.4 mmol), 1,2-dimethoxybenzene (6.30 g, 45.6 mmol), and chloroform (70 cm^3) were heated under reflux (3 h). Hydrogen chloride was evolved and yellow crystalline 3,4-dimethoxyphenyltellurium(IV) trichloride (13.85 g, 37.3 mmol, 86%), melting point (m.p.) 175 °C, separated on cooling. The trichloride (1.75 g, 4.7 mmol) and an excess of 1,2-dimethoxybenzene (5 cm^3) were boiled under reflux (24 h). Hydrogen chloride was evolved and the colour changed to dark brown. Chloroform was added to the mixture at room temperature. The resulting solution was filtered to remove traces of tellurium, shaken with charcoal, and refiltered. The filtrate was evaporated under reduced pressure and crystallisation from light petroleum (b.p. 40–60 °C) gave colourless bis(3,4-dimethoxyphenyl)tellurium(IV) dichloride (1.27 g, 2.7 mmol, 57%), m.p. 150 °C, lit. [5] m.p. 155–156 °C. Other organotellurium compounds were prepared by standard methods [1, 2]. Melting points and analytical data for aryltellurium(IV) trihalides and new organotellurium compounds are recorded in Table 3.

Cycloalkenes

A mixture of *p*-anisyltellurium(IV) trichloride (1.35 g, 4.0 mmol), cycloheptene (5 cm^3) and carbon tetrachloride (5 cm^3) was boiled under reflux (3 h). Traces of insoluble impurities were removed by filtration. The orange filtrate was shaken with charcoal, refiltered, and evaporated under reduced pressure. Crystallisation from light petroleum (b.p. 40–60 °C) gave colourless needles of *p*-anisyl(*trans*-2-chloro-1-cycloheptyl)tellurium(IV) dichloride (0.45 g, 1.0 mmol, 26%), m.p. 84–85 °C. A mixture of *p*-anisyltellurium(IV) trichloride (2.09 g, 6.1 mmol), cycloheptene (2.00 g, 20.8 mmol), ethanol (5 cm^3) and carbon tetrachloride (50 cm^3) was boiled under reflux (2 h), cooled, shaken with charcoal, and filtered. Partial evaporation under reduced pressure gave a transparent viscous residue which crystallised from light petroleum (b.p. 40–60 °C) to give colourless *p*-anisyl(*trans*-2-ethoxy-1-cycloheptyl)tellurium(IV) dichloride (1.95 g, 4.4 mmol, 72%), m.p. 110 °C. Other chlorocycloalkyl and ethoxycycloalkyl derivatives were prepared similarly. Analytical data are recorded in Table 4.

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