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Assembly of diverse structural types of organotellurium compounds in the reactions of (4-MeO-C_6H_4)$_2$TeO with pyridine carboxylic acids

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ABSTRACT

The reactions of Ar_2TeO (Ar = *4*-MeO-C_6H_4) with *2*-, *3*- and *4*-pyridine carboxylic acids (LH) afforded different organotelluroxane structural types depending on the stoichiometry of the reactants and the conditions of the reaction. $Ar_2Te(L)OH$ (**1a**–**1c**) are formed in a 1:1 reaction of Ar_2TeO with LH in the presence of water. On the other hand a 1:2 reaction under anhydrous conditions leads to the formation of Ar_2TeL_2 (**2a–2c**). A 2:2 reaction under anhydrous conditions affords the ditelluroxanes $Ar_2Te(L)OTe(L)Ar_2$ (**3a**–**3c**) while tritelluroxanes $Ar_2Te(L)OTeAr_2OTe(L)Ar_2$ (**4a–4c**) are formed in 3:2 reactions. Interestingly, **3a**–**3c** are formed in the reaction of **2a**–**2c** with Ar_2TeO. The former can be hydrolyzed to **1a**–**1c** while the latter upon reaction with Ar_2TeO lead to the formation of the tritelluroxanes **4a**–**4c**. Attempts to metalate **2a** with $PdCl_2(MeCN)_2$ leads to a transfer of the carboxylate ligand to palladium affording Ar_2TeCl_2 and PdL_2. X-ray crystal structures of representative examples of the family of **1**, **2** and **3** reveal interesting supramolecular structures and the formation of a novel $[TeO]_2$ structural unit. The latter results from intermolecular secondary Te···O interactions.

1. Introduction

Organotelluroxanes, in general, and organotellurium oxides, in particular, although known for a long time, are only recently being investigated in a systematic manner [1–32]. We have been interested in organostannoxanes for some time now and have been able to discover synthetic routes for a large variety of organostannoxane structural types [33–41]. Most of these are assembled by the reactions of organotin oxides, -hydroxides or -oxide-hydroxides with a variety of protic acids such as carboxylic, phosphinic, phosphoric or sulfonic acids [33–41]. We were interested in investigating the corresponding reactions with organotellurium oxides to find out if divergent structural types can be formed by modulating the nature of protic acid. As part of this research activity we have recently reported the reactions of Ar_2TeCl_2 (Ar = *4*-MeO-C_6H_4) with 1,1′-ferrocenedicarboxylic acid ($L'H_2$) which afforded the heterometallic macrocycle $[Ar_2TeL']_2$. [42]. Similar macrocycles $[R_2SnL']_2$ (R = *n*Bu, Bn) were also isolated in the reactions of R_2SnCl_2 with $L'H_2$ [42]. Encouraged by these results we have investigated the reactions of Ar_2TeO with *2*-, *3*- and *4*-pyridine carboxylic acids (LH). We have been able to isolate $Ar_2Te(L)(OH)$ (**1**), $Ar_2Te(L)_2$ (**2**), $Ar_2Te(L)OTe(L)Ar_2$ (**3**) and $Ar_2Te(L)OTe(Ar)_2OTe(L)Ar_2$ (**4**). The synthesis, reactivity and structural characterization of these divergent product types is discussed herein. During the course of these investigations, Beckmann and coworkers have also reported formation of similar structural types in the reactions of R_2TeO (R = Ph, *4*-MeO-C_6H_4, *4*-Me_2N-C_6H_4) with phenol and *o*-nitrophenol [29]. Such correspondence of products formed in the reactions with diverse reagents such as carboxylic acids and phenols is quite rare among organotin compounds.

2. Results and discussion

All the three pyridine carboxylic acids (LH) (*2*-, *3*- and *4*-) react with Ar_2TeO giving the same product type (Scheme 1). The nature of the product formed does not depend on which pyridine carboxylic acid was used but on the stoichiometry of the reactants and the reaction conditions.

A 1:1 reaction of Ar_2TeO with LH in presence of a slight excess of water affords the hydroxycarboxylates $[Ar_2Te(OH)L]$ (**1a**, **1b**, **1c**) (Scheme 1). Infrared spectra of these compounds show peaks at 3424, 3431 and 3424 cm^{-1}, respectively, due to the OH stretching frequency. ^{125}Te NMR shows singlets at 977.1 (**1b**) and 976.8 (**1c**). The ESI-MS spectrum of **1c** shows a peak at 736.9 which corresponds to $[M+C_6H_6+CH_3C_6H_5+2CH_3CN+H]^+$. A 1:2 reaction of Ar_2TeO and LH affords the dicarboxylates Ar_2TeL_2 (**2a**–**2c**) (Scheme 1). ^{125}Te NMR of these compounds show the presence of singlets which are slightly downfield shifted in comparison to compounds belonging to the family **1**: 1056.7 (**2a**), 1001.1 (**2b**) and 1046.4 (**2c**) ppm. A 2:2 reaction of Ar_2TeO and LH under anhydrous conditions afforded the condensed ditelluroxanes **3a**–**3c** [^{125}Te NMR of

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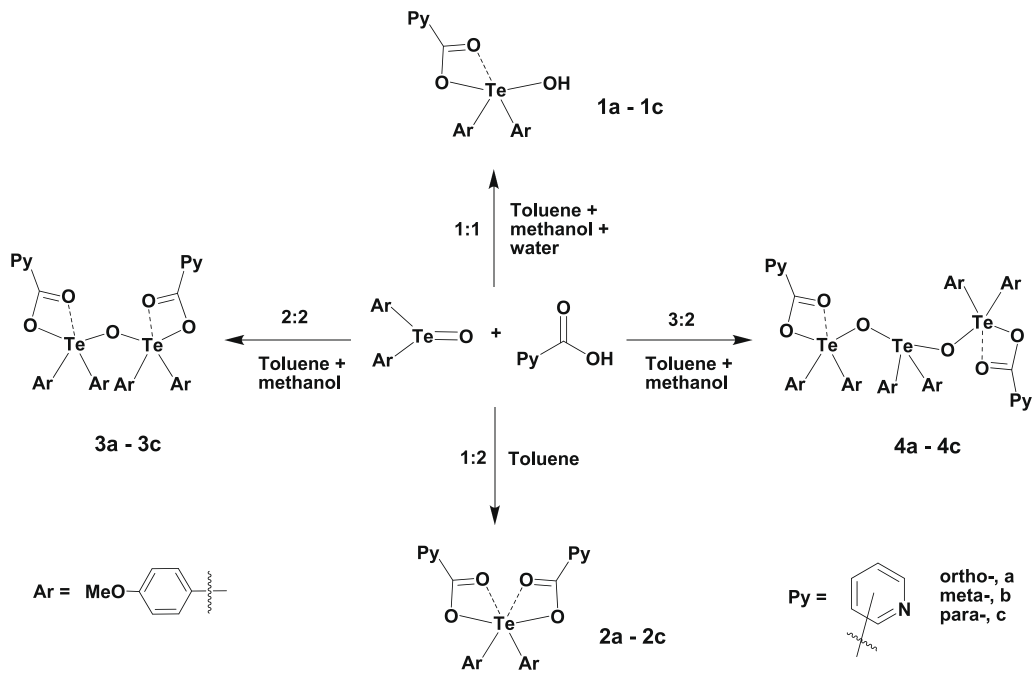
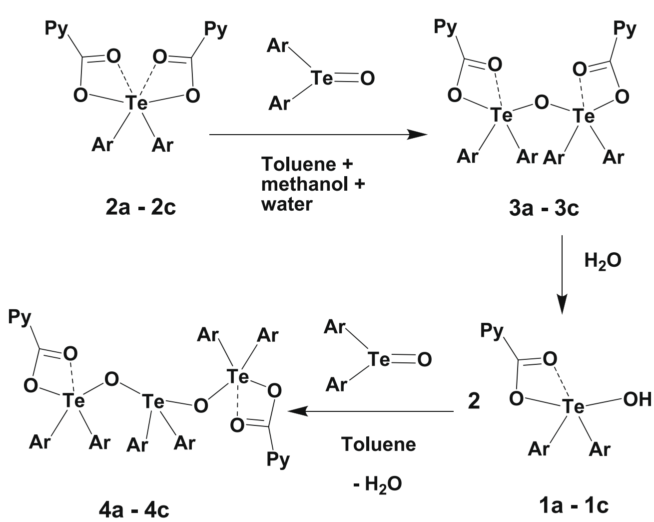


Scheme 1.

3a: 1033.0 ppm]. Representative examples of the family of compounds **1**, **2** and **3** have been characterized by single crystal X-ray methods (*vide infra*). Tritelluroxanes **4a**–**4c** were isolated in a 3:2 reaction of Ar_2TeO with LH. These could not be characterized by single crystal X-ray diffraction methods. However, their ^{125}Te NMR shows the presence of two signals [*cf.* ^{125}Te of **4a**: 967.6 and 975.2 ppm] suggesting their formation. Also the tritelluroxanes **4a**–**4c** are formed in reaction of **1a**–**1c** with Ar_2TeO (Scheme 2). From the work of Kobayashi it is already known that Ar_2TeO reacts with cationic ditelluroxanes to afford oligotelluroxanes [20,22]. In this case Ar_2TeO functions as a nucleophile facilitating the formation of new Te–O–Te bonds. Such a mechanism also appears to be operating in the conversion of **2a**–**2c** to **3a**–**3c** upon reaction with Ar_2TeO (Scheme 2). Interestingly, compounds **3a**-**3c** upon reaction with water afford the hydroxycarboxylates **1a**–**1c** (Scheme 2).

It was of interest to check the metalation behavior of these compounds in view of the fact that they contained pyridyl nitrogen atoms for coordination. However, the reaction of **2a** with $PdCl_2(MeCN)_2$ resulted in the complete transfer of the carboxylate ligand from tellurium to palladium resulting in the formation of PdL_2 and Ar_2TeCl_2 (Scheme 3). Such a transfer of carboxylate ligand from a main-group metal to a transition metal ion has been reported by us earlier and involves organotinpyrazolyl carboxylates [43].

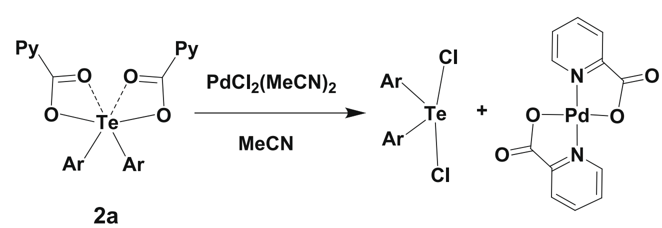


Scheme 2.

2.1. X-ray crystal structures of **1a**, **1c**, **2a**, **2b**, **3b** and **3c**

Crystal and cell parameter data for **1a**, **1c**, **2a**, **2b**, **3b** and **3c** are given in Table 1. In view of the similarity of structures within a given family only representative structures are discussed herein. The remaining information is summarized in Supplementary material.

The molecular structure of **1a** is shown in Fig. 1. Two molecules are present in the asymmetric unit of which only one is shown in Fig. 1a. The tellurium atom is present in a *see-saw* arrangement because of the influence of a stereochemically active lone pair. The two oxygen atoms are *trans* with respect to each other with a O5–Te–O3 bond angle of 168.57(11)°. The Te–O distance involving the Te–OH group is considerably shorter (Te1–O5, 1.998(3) Å) in comparison to the one which involves the carboxylate oxygen atom (Te1–O3, 2.401(3) Å). A similar situation has been found by Beckmann and co-workers in (p-MeO-C_6H_4)$_2$Te(OPh)OH where the Te–OH distance was 1.980(4) Å [29]. A long Te–O contact involving the carbonyl oxygen atom (Te1···O4: 2.8728(40) Å) is also found in the present instance. This distance is smaller than the sum of van der Waals radii (3.60 Å) and greater than the sum of covalent radii (2.03 Å) of tellurium and oxygen atoms [44].



Scheme 3.

Table 1
X-ray crystallographic data for compounds **1a**, **1c**, **2a**, **2b**, **3b** and **3c**.

	1a	**1c**	**2a**	**2b**	**3b**	**3c**
Empirical formula	$C_{20}H_{19}NO_5Te$	$C_{20}H_{19}NO_5Te$	$C_{52}H_{44}N_4O_{13}$ Te_2	$C_{26}H_{22}N_2O_6Te$	$C_{197.50}H_{180}N_8O_{38}Te_8$	$C_{191}H_{176}N_8O_{36}Te_8$
Formula weight	480.96	480.96	1188.11	586.06	4294.29	4180.2
Temperature [K]	100(2)	100(2)	273(2)	100(2)	153(2)	100(2)
Wavelength [Å]	0.71073	0.71073	0.71073	0.71073	0.71073	0.71073
Crystal system	Triclinic	Triclinic	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	$P\bar{1}$	$P\bar{1}$	*P21/c*	*C2/c*	*P21/n*	*P21/n*
a [Å]	10.7129(12)	9.9767(15)	20.878(4)	23.5218(17)	15.2510(13)	14.8863(13)
b [Å]	11.5508(13)	13.604(2)	15.079(3)	15.1997(17)	23.540(2)	23.393(2)
c [Å]	17.185(2)	15.596(2)	15.972(3)	15.3941(13)	25.744(2)	26.083(2)
α [°]	92.308(2)	102.016(3)	90	90	90	90
β [°]	103.340(2)	104.604(3)	91.518(3)	120.141(2)	101.988(2)	98.992(2)
γ [°]	112.953(2)	107.970(3)	90	90	90	90
V [Å^3]	1885.1(4)	1852.0(5)	5026.7(15)	4759.6(18)	9040.5(14)	8971.5(14)
Z, $D_{calc.}$ [g cm^{-3}]	4, 1.695	4, 1.725	4, 1.570	8, 1.636	2, 1.578	2, 1.547
μ [mm^{-1}]	1.609	1.638	1.229	1.295	1.351	1.358
$F(0\,0\,0)$	952	952	2368	2336	4282	4164
Crystal size [mm]	0.1 × 0.08 × 0.04	0.07 × 0.03 × 0.02	0.08 × 0.05 × 0.04	0.11 × 0.08 × 0.06	0.08 × 0.06 × 0.03	0.1 × 0.07 × 0.04
θ range [°]	2.14–26.00	2.25–27.00	2.08–28.35	2.58–25.00	2.08–26.50	2.12–26.50
Limiting indices	$-13 \leqslant h \leqslant 12$, $-14 \leqslant k \leqslant 13$, $-16 \leqslant l \leqslant 21$	$-12 \leqslant h \leqslant 10$, $-17 \leqslant k \leqslant 16$, $-18 \leqslant l \leqslant 19$	$-27 \leqslant h \leqslant 18$, $-19 \leqslant k \leqslant 18$, $-21 \leqslant l \leqslant 21$	$-27 \leqslant h \leqslant 27$, $-18 \leqslant k \leqslant 11$, $-17 \leqslant l \leqslant 18$	$-18 \leqslant h \leqslant 19$, $-29 \leqslant k \leqslant 29$, $-20 \leqslant l \leqslant 32$	$-18 \leqslant h \leqslant 15$, $-17 \leqslant k \leqslant 29$, $-30 \leqslant l \leqslant 32$
Reflections collected	10 480	11 234	32 376	12 118	51 975	51 760
Independent reflections (R_{int})	7216 (0.0152)	7838 (0.0399)	12 307 (0.0904)	4200 (0.0415	18 675 (0.0747)	18 580 (0.0737)
Data/restraints/ parameters	7216/0/493	7838/4/466	12 307/38/662	4200/12/318	18 675/23/1156	18 580/0/1103
Goodness-of-fit on F^2	1.095	1.050	0.926	1.054	0.993	1.018
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0354$, $wR_2 = 0.0960$	$R_1 = 0.0873$, $wR_2 = 0.2149$	$R_1 = 0.0758$, $wR_2 = 0.1677$	$R_1 = 0.0380$, $wR_2 = 0.0883$	$R_1 = 0.0495$, $wR_2 = 0.1062$	$R_1 = 0.0559$, $wR_2 = 0.1336$
R indices (all data)	$R_1 = 0.0390$, $wR_2 = 0.1085$	$R_1 = 0.1477$, $wR_2 = 0.2735$	$R_1 = 0.1864$, $wR_2 = 0.2231$	$R_1 = 0.0470$, $wR_2 = 0.0949$	$R_1 = 0.0826$, $wR_2 = 0.1274$	$R_1 = 0.0911$, $wR_2 = 0.1662$

The molecular structure of **2b** is shown in Fig. 2a. The two carboxylate ligands bind to the central tellurium atom in a chelating anisobidentate manner (*cf.* Te–O5, 2.136(3); Te–O6, 2.9524(26) Å), resulting in an overall *skewed-trapezoidal* geometry. In spite of the chelating coordination of the carboxylate ligands the O5–Te–O3 bond angle (165.19(10)°) is not distorted appreciably and is in fact comparable to that found in $Ph_2Te(OPh)_2$ (166.0(2)°) [29].

The molecular structure of **3c** is shown in Fig. 3a. Although two molecules are present in its asymmetric unit, only one of them is shown. The ditelluroxane is characterized by an acute Te1–O7–Te2 angle of 116.3(2)° which is comparable to that found in $(R'O)R_2TeOTeR_2(OR')$ (R = *p*-MeOC$_6$H$_4$; R′ = *o*-O$_2$NC$_6$H$_4$) [29]. The Te–O bond distances involving the central oxygen atom (av. 2.008(4) Å) are shorter than the Te–O bond distances involving the carboxyl oxygen atom (av. 2.291(4) Å). As in the case of the other family of compounds discussed already **3c** is also characterized by the presence of long Te···O contacts (av. 3.063(5) Å).

2.2. Supramolecular structures

The Te–OH units present in **1a** do not interact with each other. Two different molecules present in different asymmetric units interact with each other to afford a dimeric structure. In this interaction the O**H** group (H5BO) participates in a bifurcated hydrogen bonding with a pyridyl nitrogen atom (N1A) and a carboxylate oxygen (O4A). This leads to the formation of an interesting Te_2O_2 [Te1A-O4B-Te1B-O4A] structural motif (Te1A···O4B, 2.8059(36); Te1A···O4A, 2.8728 (40); Te1B···O4B, 3.0522(38); Te1B···O4A, 2.7282(33) Å). Further C–H···O interactions (between O5B and a pyridyl C–H(H20B); O5A and H20A) leads to the formation of a one-dimensional chain (Supplementary material). Although **1c** also forms a hydrogen-bonded dimer similar to that of **1b**, further supramolecular interactions are different due to the involvement of the *p*-OMe group (Supplementary material). In contrast to the situation found here, (*p*-MeO-C$_6$H$_4$)$_2$Te(OPh)OH does not appear to generate intermolecular secondary Te···O interactions [29].

The crystal structures of **2a** and **2b** show the formation of a centrosymmetric dimeric unit which occurs as a result of reciprocatory intermolecular Te···O interactions (*cf.* in **2b**: Te···O4, 3.4194(40) Å). Further supramolecular interactions (C–H···N and C–H···O) generate a 3D-supramolecular structure in **2b** (Supporting Information). In **2a** the two independent molecules present in the asymmetric unit interact with only molecules of *their* type leading to chain-structures (Supplementary material). This is facilitated by intermolecular C–H···N interactions. Again, the corresponding (*p*-Me$_2$N-C$_6$H$_4$)$_2$Te(OPh)$_2$ and $Ph_2Te(OPh)_2$ do not exhibit secondary Te···O interactions [29].

The supramolecular structures of **3b** and **3c** are similar and involve two orthogonal O=C–O–Te–O–Te–O–C=O motifs which interact with each other through secondary Te···O interactions affording cyclic structures. Such supramolecular ring formation appears to be quite unique among organotelluroxanes (Fig. 3b and 4).

2.3. Conclusions

In conclusion, we report the formation of four different structural types in the reactions of Ar_2TeO with pyridine carboxylic acids. The nature of the carboxylic acid (*2*-, *3*- or -*4*) does not affect the course of reaction. On the other hand, the product formed depends on the stoichiometry of the reactants. Among the four prod-

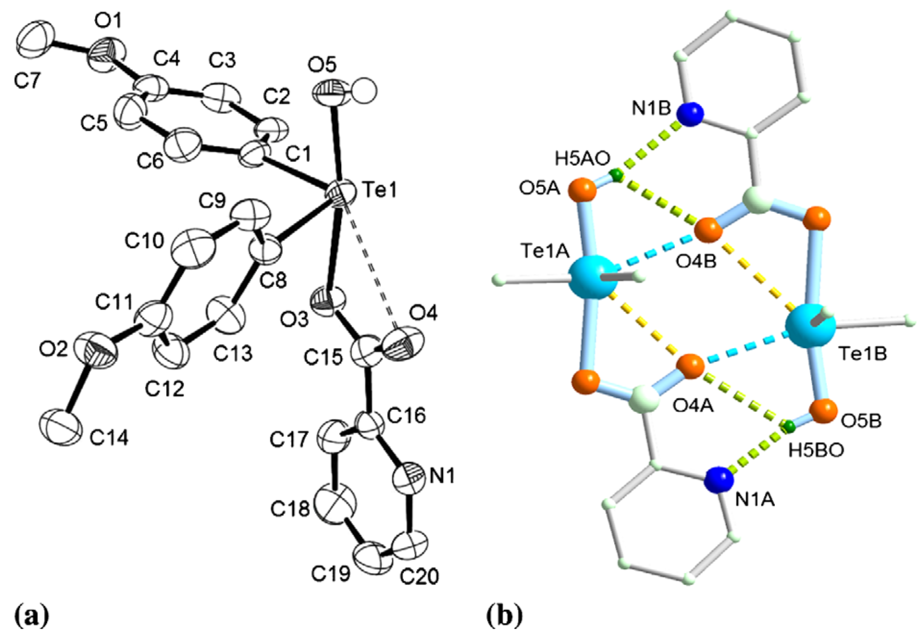


Fig. 1. (a) ORTEP drawing of **1a** with 50% probability thermal ellipsoids (only one molecule (molecule 'a') present in the asymmetric unit is shown). Hydrogen atoms, molecule 'b' and solvent molecules have been omitted for clarity. Selected bond distances (Å) and angles (°) are as follows: Te1–C1, 2.113(4); Te1–C8, 2.112(4); Te1–O3, 2.401(3); Te1–O5, 1.998(3); Te1···O4, 2.8728(40); O5–Te1–C8, 92.17(15); O5–Te1–C1, 85.81(14); C8–Te1–C1, 94.13(16); O5–Te1–O3, 168.57(11); C8–Te1–O3, 86.83(14); C1–Te1–O3, 82.90(13). See Figure S2 in the Supporting information for a complete asymmetric unit. (b) A $[TeO]_2$ dimeric unit is formed through intermolecular Te···O, O–H···O and O–H···N interactions between the two molecules present in the asymmetric unit. Atoms involved in interactions are lying in a plane. Anisyl groups have been omitted for clarity. The bond distances (Å) and angles (°) involved are: Te1A···O4A, 2.8728(40); Te1A···O4B, 2.8059(36); Te1B···O4B, 3.0522(38); Te1B···O4A, 2.7282(33); H5AO···N1B, 2.1276(35); H5AO···O4B, 2.4005(42); H5BO···N1A, 1.9548(35); H5BO···O4A, 2.4750(39); O5A–H5AO···N1B, 162.764(239); O5A–H5AO···O4B,123.245(243); O5B–H5BO···N1A, 165.577(241); O5B–H5BO···O4A, 119.755(237).

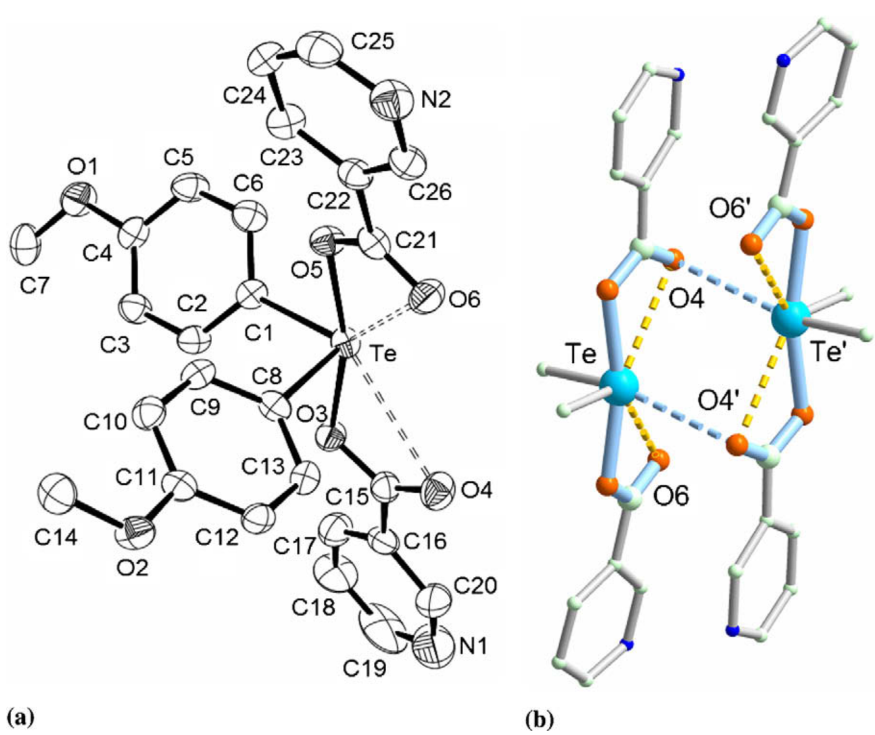


Fig. 2. (a) ORTEP drawing of **2b** with 50% probability thermal ellipsoids. Hydrogen atoms have been omitted for clarity. Selected bond distances (Å) and angles (°) are as follows: Te–C1, 2.102(4); Te–C8, 2.095(4); Te–O3, 2.164(3); Te–O5, 2.136(3); Te···O4, 3.0280(26); Te···O6, 2.9524(26); C8–Te–C1, 98.17(15); C8–Te–O5, 86.26(13); C1–Te–O5, 83.12(13); C1–Te–O3, 83.79(13); C8–Te–O3, 88.77(13); O5–Te–O3, 165.19(10). (b) A centrosymmetric $[TeO]_2$ dimeric unit formed through reciprocatory intermolecular Te···O interactions. Anisyl groups have been omitted for clarity. Bond distance (Å): Te···O4□, 3.4196(40) Å.

uct types isolated in the present instance, two are monotellurium derivatives, while the others are a ditelluroxane and tritelluroxane. The structures of these products are similar to those isolated from the reaction of diorganotellurium oxides with phenols. However, in the present instance we have been able to observe interesting supramolecular structures involving a novel $[TeO]_2$ dimeric struc-

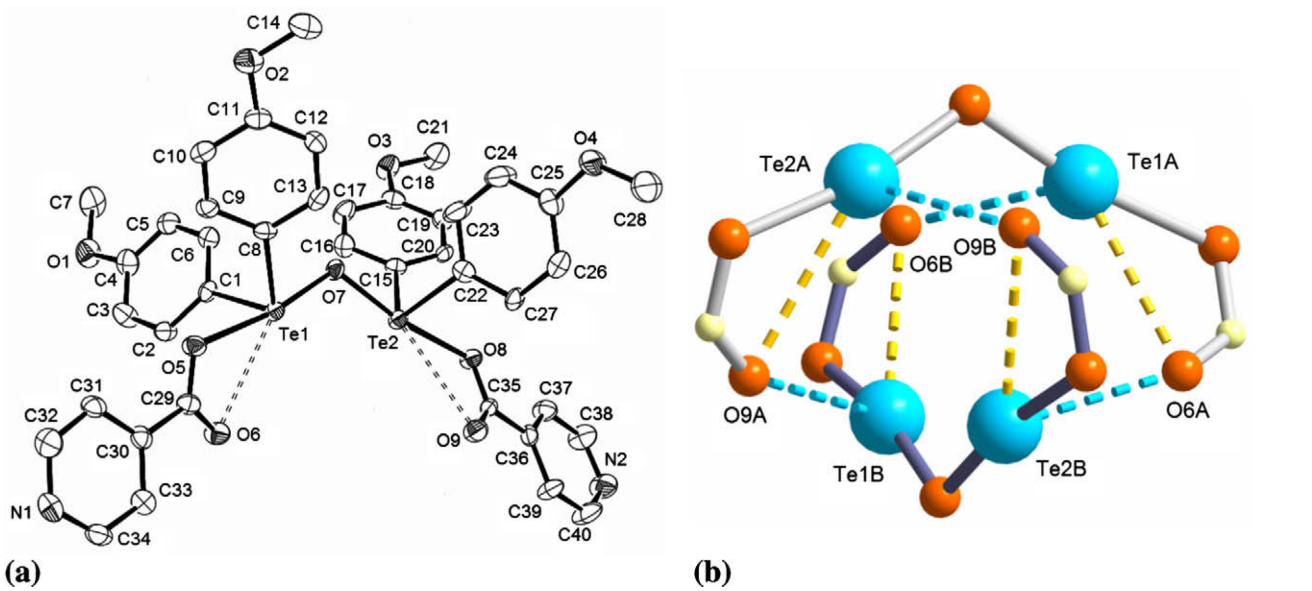


Fig. 3. (a) ORTEP drawing of **3c** with 50% probability thermal ellipsoids (only molecule 'a' is shown). Hydrogen atoms, molecule 'b' and solvent molecules have been omitted for clarity. Selected bond distances (Å) and angles (°) are as follows: Te1–C1, 2.105(6); Te1–C8, 2.102(6); Te2–C15, 2.111(6); Te2–C22, 2.103(6); Te1–O5, 2.308(4); Te1–O7, 2.001(4); Te2–O7, 2.015(4); Te2–O8, 2.273(4); Te1···O6, 3.0044(52); Te2···O9, 3.1218(49); Te1–O7–Te2, 116.3(2); O7–Te1–C8, 87.8(2); O7–Te1–C1, 91.8(2); C8–Te1–C1, 95.8(2); O7–Te1–O5, 170.64(17); C8–Te1–O5, 84.1(2); C1–Te1–O5, 84.4(2); O7–Te2–C22, 90.7(2); O7–Te2–C15, 88.9(2); C22–Te2–C15, 95.2(2); O7–Te2–O8, 169.11(17); C22–Te2–O8, 85.6(2); C15–Te2–O8, 81.3(2). See Fig. S17 in the Supporting information for a complete asymmetric unit. (b) A dimeric unit formed as a result of interactions between molecule 'a' and molecule 'b' through intermolecular Te···O interactions. Bond distances (Å) and angles (°) involved are: Te1A···O6A, 3.0044(52); Te2A···O9A, 3.1218(49); Te1B···O6B, 3.0461(49); Te2B···O9B, 3.0657(62); Te1A···O6B, 3.4567(61); Te2A···O9B, 3.0182(74); Te2B···O6A, 3.1232(50); Te1B···O9A, 3.0426(50).

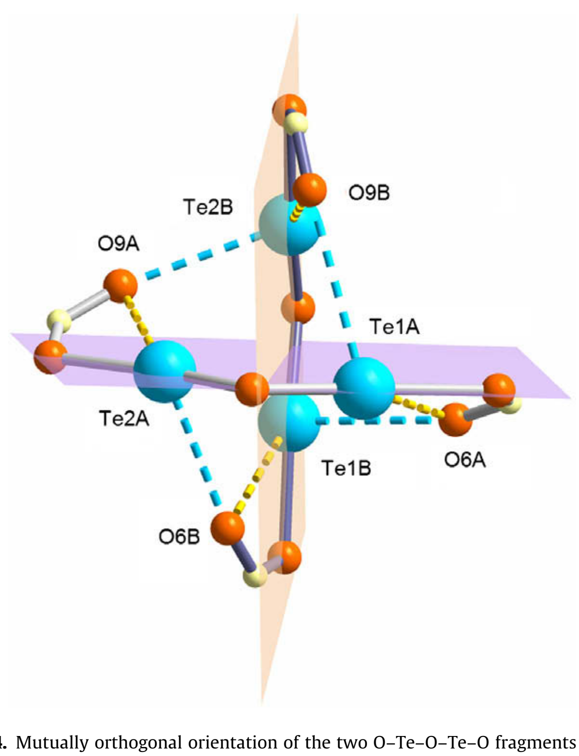


Fig. 4. Mutually orthogonal orientation of the two O–Te–O–Te–O fragments in **3c**.

tural motif which is formed as a result of intermolecular secondary Te···O interactions.

3. Experimental

3.1. Reagents and general procedures

All reactions were carried out using anhydrous solvents unless otherwise stated. The solvents were purified by standard procedures and stored under nitrogen atmosphere and over activated molecular sieves [45]. Tellurium tetrachloride, *ortho*-, *meta*- and *para*-pyridinecarboxylic acids and palladium(II) chloride were purchased from Aldrich Chemical Co. (USA) and were used as received. Bis(*p*-methoxyphenyl)telluroxide was prepared from the reaction of anisole with tellurium tetrachloride followed by its oxidation with NaOH as reported in the literature [46]. Sodium hydroxide (RANKEM) was purchased from RFCL Limited, New Delhi, India and was used as such. Anisole was purchased from s.d. Fine. Chem. Ltd., Mumbai, India and distilled under N_2 atmosphere before use. Melting points were measured by using a JSGW melting point apparatus and are uncorrected. Elemental Analyses of the compounds were obtained using a EAI elemental analyzer CE-440 model. The analyses of compounds were carried out on fully dried samples. IR spectra were recorded as KBr pellets with a Bruker Vector 22 FTIR spectrophotometer operating from 4000 to 400 cm^{-1}. Electrospray ionization mass spectra were recorded with a WATERS–HAB213 spectrometer by using capillary 2.7 kV. NMR (^{1}H, ^{13}C and ^{125}Te NMR) spectra were recorded with a JEOL JNM LAMBDA 400 model spectrometer or a JEOL JNM DELTA 500 model spectrometer.

3.2. Syntheses

*3.2.1. Synthesis of di-p-anisyltellurium hydroxy pyridylcarboxylates, (4-MeO-C_6H_4)$_2$Te($O_2CC_5H_4N$)(OH) (**1a–1c**)*

Pyridinecarboxylic acid (0.25 g, 2.0 mmol) and bis(*p*-anisyl)telluroxide (0.71 g, 2.0 mmol) were dissolved in toluene-methanol (15 mL each) containing ~1 mL of water. Prolonged (~24 h) heating at reflux under constant stirring gave a clear solution. The reaction mixture was allowed to cool to room temperature and the volatiles were removed completely under vacuum. The resulting solid was recrystallized from its water–methanol (1:10 mL) solution.

1a: Yield: 0.80 g (83%). m.p. 142–143 °C. IR (KBr)/cm^{-1}: ν_{asym} (COO), 1603.24, ν_{asym} (OH), 3423.95. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.75 (s, 6H, OMe), 6.84–7.76 (m, 12H, arom) ppm. ^{13}C NMR

(500 MHz, $CDCl_3$): δ 170.72 (OCO), 161.64 (*p*-anisyl), 151.89 (*2*-pyridyl), 149.24 (*6*-pyridyl), 136.71 (*o*-anisyl), 134.34 (*3 and 5*-pyridyl), 125.42 (*4*-pyridyl), 124.73 (*i*-anisyl), 115.11 (*m*-anisyl), 55.46 (OMe) ppm. Anal. Calc. for $C_{20}H_{19}NO_5Te$ (480.97): C, 49.94; H, 3.98; N, 2.91. Found: C, 49.81; H, 3.83; N, 2.83%. ESI-MS: *m*/*z* (%) = 361.0052 (100) [(4-OMe-Ph)$_2$Te(OH)]$^+$, 78.9738 (16) [(4-OMe-Ph)$_2$TeO + H_2O + H)]$^+$, 717.0108 (4) [2(4-OMe-Ph)$_2$TeO+H)]$^+$, 736.9713 (28) [M+C_6H_6+$CH_3C_6H_5$+2CH_3CN+H]$^+$.

1b: Yield: 0.75 g (78%). m.p. 161–166 °C. IR (KBr)/cm^{-1}: ν_{asym} (COO), 1602.13, ν_{asym} (OH), 3431.17. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.73 (s, 6H, OMe), 6.94–8.54 (m, 12H, arom) ppm. ^{13}C NMR (500 MHz, $CDCl_3$): δ 165.96 (OCO), 156.31 (*p*-anisyl), 142.47 (*2*-pyridyl), 141.69 (*6*-pyridyl), 139.13 (*3*-pyridyl), 134.03 (*4*-pyridyl), 133.33 (*o*-anisyl), 130.44 (*5*-pyridyl), 128.24 (*i*-anisyl), 119.35 (*m*-anisyl), 60.40 (OMe) ppm. ^{125}Te NMR (157.8 MHz, DCM + $CDCl_3$): δ = 977.1 (s) ppm. Anal. Calc. for $C_{20}H_{19}NO_5Te$ (480.97): C, 49.94; H, 3.98; N, 2.91. Found: C, 50.21; H, 3.75; N, 2.96%.

1c: Yield: 0.79 g (81%). m.p. 175–182 °C. IR (KBr)/cm^{-1}: ν_{asym} (COO), 1625.42, ν_{asym} (OH), 3423.74. ^{13}C NMR (500 MHz, $CDCl_3$): δ 170.21 (OCO), 161.22 (*p*-anisyl), 153.27 (*2*- and 6-pyridyl), 149.44 (*4*-pyridyl), 137.23 (*3*- and 5-pyridyl), 134.60 (*o*-anisyl), 124.52 (*i*-anisyl), 115.08 (*m*-anisyl), 55.79 (OMe) ppm. ^{125}Te NMR (157.8 MHz, DCM + $CDCl_3$): δ = 976.8 (s) ppm. Anal. Calc. for $C_{20}H_{19}NO_5Te$ (480.97): C, 49.94; H, 3.98; N, 2.91. Found: C, 49.78; H, 3.78; N, 2.87%.

Alternatively, compounds **1a–1c** have been prepared by the hydrolysis of **3a–3c**, respectively. In a general procedure, product type **3** (0.095 g, 0.1 mmol) was heated at reflux in 10 mL aqueous-methanol (~50%) for about 24 h. The solution was cooled to room temperature and concentrated up to 1–2 mL under vacuum and ~15 mL methanol was added. Again it was reduced to about 5 mL. Slow evaporation of solution afforded colorless crystals of respective product type **1** in 45–59% yields.

3.2.2. Synthesis of di-p-anisyltellurium bis(pyridylcarboxylates), (4-MeO-C_6H_4)$_2$Te($O_2CC_5H_4N$)$_2$ (**2a–2c**)

A mixture of pyridinecarboxylic acid (0.25 g, 2.0 mmol) and bis(*p*-anisyl)telluroxide (0.36 g, 1.0 mmol) were taken together in dry toluene (~50 mL) and stirred under heating at reflux with the use of a Dean–Stark apparatus for 8 h. The reaction mixture was allowed to come to room temperature and the volume reduced to about 10 mL and about 2 mL of dry methanol was added. Slow evaporation of the solution afforded colorless crystals which were collected by decantation and washed with cold diethyl ether.

2a: Yield: 0.48 g (82%). m.p. 168–171 °C. IR (KBr)/cm^{-1}: ν_{asym} (COO), 1659.16. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.75 (s, 6H, OMe), 6.88–8.73 (m, 16H, arom) ppm. ^{13}C NMR (500 MHz, $CDCl_3$): δ 169.69 (OCO), 161.78 (*p*-anisyl), 149.50 (*2*-pyridyl), 149.27 (*6*-pyridyl), 136.96 (*o*-anisyl), 135.05 (*3*-pyridyl), 127.77 (*5*-pyridyl), 126.26 (*4*-pyridyl), 125.26 (*i*-anisyl), 115.33 (*m*-anisyl), 55.37 (OMe) ppm. ^{125}Te NMR (157.8 MHz, DCM + $CDCl_3$): δ = 1056.7 (s) ppm. Anal. Calc. for $C_{26}H_{22}N_2O_6Te$ (586.06): C, 53.28; H, 3.78; N, 4.78. Found: C, 53.02; H, 3.68; N, 4.89%. ESI-MS: *m*/*z* (%) = 124.0388 (26) [C_5H_4NCOOH+H]$^+$, 360.9916 (100) [(4-OMe-Ph)$_2$Te(OH)]$^+$, 528.9963 (6) [(4-OMe-Ph)$_2$TeO+C_5H_4NCOOH+HCOOH], 717.0009 (15) [2(4-OMe-Ph)$_2$TeO+H)]$^+$, 744.9789 (41) [M+$CH_3C_6H_5$+CH_3OH + H]$^+$, 822.0154 (18) [M + C_5H_4NCOOH + HCOOH + 2CH_3OH + H]$^+$.

2b: Yield: 0.43 g (73%). m.p. 182–183 °C. IR (KBr)/cm^{-1}: ν_{asym} (COO), 1642.74. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.78 (s, 6H, OMe), 6.96–9.07 (m, 16H, arom) ppm. ^{13}C NMR (500 MHz, $CDCl_3$): δ 170.22 (OCO), 162.05 (*p*-anisyl), 152.71 (*2*-pyridyl), 151.23 (*6*-pyridyl), 137.39 (*3*-pyridyl), 134.34 (*4*-pyridyl), 127.59 (*o*-anisyl), 125.30 (*5*-pyridyl), 123.15 (*i*-anisyl), 115.54 (*m*-anisyl), 55.42 (OMe) ppm. ^{125}Te NMR (157.8 MHz, DCM+$CDCl_3$): δ = 1001.1 (s) ppm. Anal. Calc. for $C_{26}H_{22}N_2O_6Te$ (586.06): C, 53.28; H, 3.78; N, 4.78. Found: C, 53.35; H, 3.69; N, 4.82%. ESI-MS: *m*/*z* (%) = 124.0334 (93) [C_5H_4NCOOH+H]$^+$, 360.9918 (100) [(4-OMe-Ph)$_2$Te(OH)]$^+$, 528.9966 (4) [(4-OMe-Ph)$_2$TeO+C_5H_4NCOOH+HCOOH], 717.0073 (18) [2(4-OMe-Ph)$_2$TeO+H)]$^+$, 744.9816 (39) [M+$CH_3C_6H_5$+CH_3OH + H]$^+$, 822.0072 (36) [M + C_5H_4NCOOH + HCOOH + 2CH_3OH + H]$^+$.

2c: Yield: 0.46 g (78%). m.p. 155–157 °C. IR (KBr)/cm^{-1}: ν_{asym} (COO), 1641.46. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.89 (s, 6H, OMe), 7.04–8.28 (m, 16H, arom) ppm. ^{13}C NMR (500 MHz, $CDCl_3$): δ 173.75 (OCO), 161.86 (*p*-anisyl), 150.45 (*2*- and 6-pyridyl), 134.98 (*4*-pyridyl), 133.66 (*3*- and 5-pyridyl), 132.66 (*o*-anisyl), 126.21 (*i*-anisyl), 116.58 (*m*-anisyl), 55.49 (OMe) ppm. ^{125}Te NMR (157.8 MHz, DCM+$CDCl_3$): δ = 1046.4 (s) ppm. Anal. Calc. for $C_{26}H_{22}N_2O_6Te$ (586.06): C, 53.28; H, 3.78; N, 4.78. Found: C, 53.21; H, 3.84; N, 4.69%. ESI-MS: *m*/*z* (%) = 124.0360 (53) [C_5H_4NCOOH+H]$^+$, 360.9973 (100) [(4-OMe-Ph)$_2$Te(OH)]$^+$, 528.9946 (3) [(4-OMe-Ph)$_2$TeO+C_5H_4NCOOH+HCOOH], 717.0005 (6) [2(4-OMe-Ph)$_2$TeO+H)]$^+$, 744.9876 (33) [M+$CH_3C_6H_5$+CH_3OH+H]$^+$, 822.0219 (5) [M+C_5H_4NCOOH+HCOOH+2CH_3OH+H]$^+$.

3.2.3. Synthesis of tetra-p-anisylditelluroxane bis(pyridylcarboxylates), (4-MeO-C_6H_4)$_2$Te($O_2CC_5H_4N$)OTe($O_2CC_5H_4N$)(4-MeO-C_6H_4)$_2$ (**3a–3c**)

Pyridinecarboxylic acid (0.25 g, 2.0 mmol) and bis(*p*-anisyl)telluroxide (0.71 g, 2.0 mmol) were dissolved in toluene-methanol (20 mL each). The reaction mixture was heated at reflux under stirring for ~16 h. After allowing the reaction mixture to come to room temperature volatiles were removed completely under vacuum. The solid, thus obtained was dissolved in a mixture of chloroform-methanol (10 mL) which on slow evaporation gave colorless crystals.

3a: Yield: 0.72 g (76%). m.p. 131–132 °C. IR (KBr)/cm^{-1}: ν_{asym} (COO), 1603.48. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.74 (s, 12H, OMe), 6.92–8.93 (m, 24H, arom) ppm. ^{13}C NMR (500 MHz, $CDCl_3$): δ 170.68 (OCO), 161.91 (*p*-anisyl), 150.12 (*2*-pyridyl), 141.59 (*6*-pyridyl), 134.19 (*o*-anisyl), 129.12 (*3*-pyridyl), 128.32 (*5*-pyridyl), 125.38 (*4*-pyridyl), 123.34 (*i*-anisyl), 115.32 (*m*-anisyl), 55.53 (OMe) ppm. ^{125}Te NMR (157.8 MHz, DCM+$CDCl_3$): δ = 1033.0 (s) ppm. Anal. Calc. for $C_{40}H_{36}N_2O_9Te_2$ (943.92): C, 50.90; H, 3.84; N, 2.97. Found: C, 51.17; H, 3.60; N, 2.93%.

3b: Yield: 0.63 g (65%). m.p. 132–136 °C. IR (KBr)/cm^{-1}: ν_{asym} (COO), 1602.68. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.89 (s, 12H, OMe), 6.90–8.64 (m, 24H, arom) ppm. ^{13}C NMR (500 MHz, $CDCl_3$): δ 170.69 (OCO), 161.66 (*p*-anisyl), 151.86 (*2*-pyridyl), 149.22 (*6*-pyridyl), 136.75 (*3*-pyridyl), 135.32 (*4*-pyridyl), 134.34 (*o*-anisyl), 125.46 (*5*-pyridyl), 124.74 (*i*-anisyl), 115.12 (*m*-anisyl), 55.46 (OMe) ppm. Anal. Calc. for $C_{40}H_{36}N_2O_9Te_2$ (943.92): C, 50.90; H, 3.84; N, 2.97. Found: C, 50.93; H, 3.77; N, 3.16%.

3c: Yield: 0.68 g (72%). m.p. 160 °C. IR (KBr)/cm^{-1}: ν_{asym} (COO), 1603.99. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.84 (s, 12H, OMe), 6.94–8.69 (m, 24H, arom) ppm. ^{13}C NMR (500 MHz, $CDCl_3$): δ 170.31 (OCO), 160.46 (*p*-anisyl), 149.37 (*2*- and 6-pyridyl), 137.52 (*4*-pyridyl), 135.42 (*3*- and 5-pyridyl), 134.37 (*o*-anisyl), 124.59 (*i*-anisyl), 115.15 (*m*-anisyl), 55.79 (OMe) ppm. Anal. Calc. for $C_{40}H_{36}N_2O_9Te_2$ (943.92): C, 50.90; H, 3.84; N, 2.97. Found: C, 51.09; H, 3.96; N, 2.79%.

In an alternate procedure, product type **2** (0.06 g, 0.1 mmol) and bis(*p*-anisyl)telluroxide (0.036 g, 0.1 mmol) were heated at reflux together in ~10 mL toluene-methanol containing small amount of water aqueous-methanol for 6 h under stirring condition. The resulting solution was cooled to room temperature and concentrated to dryness. Recrystallization from chloroform–methanol solution afforded colorless crystals product type **3** in 41–56% yields.

*3.2.4. Synthesis of hexa-p-anisyltritelluroxanes bis(pyridylcarboxylate), $(4\text{-MeO-}C_6H_4)_2Te(O_2CC_5H_4N)OTe(4\text{-MeO-}C_6H_4)_2OTe(O_2CC_5H_4N)(4\text{-MeO-}C_6H_4)_2$ (**4a–4c**)*

A 2:3 mixture of pyridinecarboxylic acid (0.123 g, 1.0 mmol) and bis(*p*-anisyl)telluroxide (0.54 g, 1.5 mmol) were taken in toluene–methanol (15 mL each) and stirred under heating at reflux conditions for ~12 h. After cooling to room temperature the reaction mixture was concentrated to ~5 mL and 10 mL of toluene added. Slow evaporation of this mixture gave colorless crystals.

4a: Yield: 0.48 g (74%). m.p. 180–188 °C. IR (KBr)/cm^{-1}: ν_{asym} (COO), 1595.70. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.75 (18H, OMe), 6.89–7.78 (m, 32H, arom) ppm. ^{13}C NMR (500 MHz, $CDCl_3$): δ 170.21 (OCO), 161.43 (*p*-anisyl), 152.56 (*2*-pyridyl), 149.25 (*6*-pyridyl), 137.25 (*o*-anisyl), 134.44 (*3- and 5*-pyridyl), 125.69 (*4*-pyridyl), 124.45 (*i*-anisyl), 115.01 (*m*-anisyl), 55.82 (OMe) ppm. ^{125}Te NMR (157.8 MHz, DCM+$CDCl_3$): δ = 967.6 and 975.2 (s) ppm in 2:1 ratio for terminal and inner tellurium atoms respectively. Anal. Calc. for $C_{54}H_{50}N_2O_{12}Te_3$ (1301.78): C, 49.82; H, 3.87; N, 2.15. Found: C, 50.0; H, 3.79; N, 2.31%.

4b: Yield: 0.37 g (57%). m.p. 151–152 °C. IR (KBr)/cm^{-1}: ν_{asym} (COO), 1604.36. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.79 (18H, OMe), 6.88–9.06 (m, 32H, arom) ppm. ^{13}C NMR (500 MHz, $CDCl_3$): δ 170.95 (OCO), 161.67 (*p*-anisyl), 151.54 (*2*-pyridyl), 151.22 (*6*-pyridyl), 137.10 (*3*-pyridyl), 133.97 (*4*-pyridyl), 128.61 (*o*-anisyl), 128.32 (*5*-pyridyl), 122.96 (*i*-anisyl), 115.06 (*m*-anisyl), 55.49 (OMe) ppm. Anal. Calc. for $C_{54}H_{50}N_2O_{12}Te_3$ (1301.78): C, 49.82; H, 3.87; N, 2.15. Found: C, 49.59; H, 4.01; N, 2.09%.

4c: Yield: 0.41 g (62%). m.p. 192–195 °C. IR (KBr)/cm^{-1}: ν_{asym} (COO), 1595.67. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.73 (18H, OMe,), 6.98–8.54 (m, 32H, arom) ppm. ^{13}C NMR (500 MHz, $CDCl_3$): δ 174.97 (OCO), 166.36 (*p*-anisyl), 158.04 (*2*- and 6-pyridyl), 141.97 (*4*-pyridyl), 139.64 (*3*- and 5-pyridyl), 130.34 (*o*-anisyl), 128.78 (*i*-anisyl), 119.53 (*m*-anisyl), 60.57 (OMe) ppm. Anal. Calc. for $C_{54}H_{50}N_2O_{12}Te_3$ (1301.78): C, 49.82; H, 3.87; N, 2.15. Found: C, 49.63; H, 3.97; N, 2.23%.

In an alternate procedure, product type **1** (0.096 g, 0.2 mmol) and bis(*p*-anisyl)telluroxide (0.036 g, 0.1 mmol) were heated at reflux together in dry toluene using Dean–Stark apparatus for 10 h. The reaction mixture was allowed to come at room temperature and concentrated to 5 mL. Slow evaporation of the solution afforded colorless crystals of product type **4** in 37–45% yields.

3.3. X-ray crystallography

All measurements for **1a**, **1c**, **2a**, **2b, 3b** and **3c** were made on CCD Bruker SMART APEX diffractometer. Crystallographic data and refinement parameters are summarized in Table 1. Single crystals suitable for X-ray crystallographic analyses for **1a** and **1c** were obtained by slow evaporation their aqueous methanol solutions. Compounds **2a** and **2b** were obtained by slow evaporation of toluene solutions while chloroform-methanol solutions were used for crystallization of **3b** and **3c**. Data were collected [at 273(2) K, **2a**; 153(2) K, **3b**, 100(2) K, **1a**, **1c**, **2b**, and **3c**] using graphite-monochromated Mo Kα radiation (λ_α = 0.71073 Å). The program SMART [47] was used for collecting frames of data, indexing reflection, and determining lattice parameters, SAINT [47] for integration of the intensity of reflections and scaling, SADABS [48] for absorption correction and SHELXTL [49–50] for space group and structure determination. Full-matrix least-squares refinements on F^2, using all data, were carried out with anisotropic displacement parameters applied to all non-hydrogen atoms. Hydrogen atoms were included in geometrically calculated positions using a riding model and were refined isotropically. The figures were created using DIAMOND 3.1d software [51].

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Appendix A. Supplementary material

CCDC 718395, 718396, 718397, 718398, 718399 and 718400 contain the supplementary crystallographic data for **1a**, **1c**, **2a**, **2b**, **3b** and **3c**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/data_request/cif. Supplementary material (Tables of selected bond length and angles for compound **1a**, **2a, 2b, 3b, 1c** and **3c**, and additional figures) associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2009.03.042.

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