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# The interplay of secondary $Te \cdots N$ , $Te \cdots O$ , $Te \cdots I$ and I $\cdots I$ interactions, $Te \cdots \pi$ contacts and $\pi$ -stacking in the supramolecular structures of [{2-(4-nitrobenzylideneamino)-5-methyl}phenyl](4-methoxyphenyl)tellurium dihalides

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#### Abstract

The unsymmetrically substituted diorganotellurium dihalides  $[2-(4,4'-NO_2C_6H_4CHNC_6H_3Me]RTeX_2$  (R = 4-MeOC<sub>6</sub>H<sub>4</sub>, X = Cl, 1a; Br, 1b; I, 1c; R = 4-MeC<sub>6</sub>H<sub>4</sub>; X = Cl, 2; R = C<sub>6</sub>H<sub>5</sub>, X = Cl, 3) were prepared in good yields and characterized by solution and solid-state <sup>125</sup>Te NMR spectroscopy, IR spectroscopy and X-ray crystallography. In the solid-state, molecular structures of 1a and 1c possess scarcely observed 1,4-type intramolecular Te···N secondary interaction. Crystal packing of these compounds show an unusually rich diversity of intermolecular secondary, Te···O, Te···I and I···I interactions, Te··· $\pi$  contacts as well as extensive  $\pi$ -stacking of the organic substituents.

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Keywords: Intramolecular coordination; Secondary interactions; Diorganotellurium dihalides

## 1. Introduction

The crystal structures of most diorganotellurium dihalides are characterized by the presence of considerably longer intermolecular  $\text{Te} \cdots X$  secondary bonds. The number and directionality of these interactions and the influence on the geometry of the tellurium atoms in diorganotellurium dihalides has been thoroughly investigated [1]. By contrast the less commonly observed

<sup>1</sup> Present address: Institut für Chemie, Freie Universität Berlin, Fabeckstrasse 34-36, 14195 Berlin, Germany. secondary  $\text{Te} \cdots \pi$  [2] interactions remain comparatively less understood. On the other hand secondary  $\text{Te} \cdots N$ ,  $\text{Te} \cdots O$  interactions have mostly been identified to exist intramolecularly [3]. The formation of, in general, a fivemembered ring by chelation through secondary  $\text{Te} \cdots N(O)$  interaction is reported to modulate the stability and reactivity of organotellurium derivatives with important consequences upon their applications [4]. We have recently reported [5] the existence of strained four-membered rings formed by 1,4-type intramolecular secondary  $\text{Te} \cdots O$  interaction among diorganotellurium dihalides and their influence on the intermolecular secondary  $\text{Te} \cdots X$  interaction.

We now describe the synthesis and full characterization of five unsymmetrically substituted diorganotellu-

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rium dihalides,  $[2-(4,4'-NO_2C_6H_4CHNC_6H_3Me]RTeX_2$ (R = 4-MeOC<sub>6</sub>H<sub>4</sub>, X = Cl, **1a**; Br, **1b**; I, **1c**; R = 4-MeC<sub>6</sub>H<sub>4</sub>; X = Cl, **2**; R = C<sub>6</sub>H<sub>5</sub>, X = Cl, **3**). Compounds **1–3** may be considered as derivatives of 4-nitro-4'-methylbenzylidene aniline (NMBA), an organic substrate known to possess second-order non-linear optical (NLO) properties [6]. The crystal structures of **1a** and **1c** show an unusually rich diversity of secondary Te···N, Te···O, Te···I and I···I interactions, Te··· $\pi$ contacts as well as extensive  $\pi$ -stacking in the solid-state. A fundamental understanding of these non-covalent bonds and their bearing on the packing order is a prerequisite in the context of crystal engineering [7].

## 2. Results and discussion

### 2.1. Synthetic aspects

The diorganotellurium dichlorides, **1a**, **2** and **3** were obtained in good yield, by transmetallation of aryl-tellurium trichlorides,  $RTeCl_3$  (R = 4-MeOC<sub>6</sub>H<sub>4</sub>, 4-MeC<sub>6</sub>H<sub>4</sub>, C<sub>6</sub>H<sub>5</sub>) with [{2-(4-nitrobenzylideneamino)-5-methyl}phenyl]mercury chloride, R'HgCl (R' = 2-(4,4'-NO<sub>2</sub>C<sub>6</sub>H<sub>4</sub>CHNC<sub>6</sub>H<sub>3</sub>Me)) using a slightly modified method of Wu and coworkers [8] (Scheme 1). The halide exchange reaction of **1a** with KBr and KI provided the related diorganotellurium dibromide and diiodide,  $R'(4-MeOC_6H_4)TeX_2$  (X = Br, **1b**; I, **1c**) in very good yield (Scheme 1).

Compounds 1–3 were obtained as yellow, red/brown crystalline solids that are soluble in chloroform and dichloromethane and were characterized by multinuclear NMR spectroscopy and IR spectroscopy. The IR spectra of all the compounds show absorption bands at ~1620 cm<sup>-1</sup> due to v(C=N) which is comparable to that reported for R'HgCl [9]. The <sup>1</sup>H NMR spectra consist of a high field (~8.8 ppm) signal for the methine proton in addition to the Me/OMe and ring proton signals [8]. The tellurated C atom of the N-ring, as expected, is deshielded and appears at ~132 ppm in the <sup>13</sup>C NMR spectra of 1–3 as compared to the free Schiff base. The signal at ~144 ppm can be assigned to the methine car-

bon atom [10]. Both these signals appear to be unaffected by the electronegativity of the halogen. <sup>125</sup>Te NMR spectra of all the dihalides, 1-3 consist of a single resonance. The <sup>125</sup>Te MAS NMR chemical shift of 1a (882 ppm) closely resembles the <sup>125</sup>Te chemical shift in CDCl<sub>3</sub> (886 ppm) and suggests that the molecular structures are very similar in solution and the solid state. The  $^{125}$ Te chemical shifts (~882 ppm) of the dichlorides, **1a**, 2, 3 are almost unaffected by the inductive effect of the R group but move to upfield in case of the diiodide, 1c (840 ppm) with respect to the dichloride, 1a. The case of the dibromide, 1b (777 ppm) is surprising. A telluronium type structure for **1b**,  $[RR'TeBr]^+Br'$ , which could possibly explain this large upfield shift is not supported by conductivity measurements in acetonitrile. Repeated attempts to grow single crystal of 1b failed due to its tendency to deposit Te metal in solution on standing.

## 2.2. Crystal and molecular structure of 1a and 1c

The molecular and crystal structures of 1a and 1c are shown in Figs. 1-4. Relevant crystal data and selected bond parameters are collected in Tables 1 and 2. Consistent with the VSEPR theory, the geometry of the tellurium atoms in 1a and 1c can be described as distorted trigonal bipyramidal when considering the primary coordination sphere with the  $C_2X_2$  donor set (X = Cl, I) and the stereochemically active lone pair (Figs. 1 and 3). The average (primary) Te-C, Te-Cl and Te-I bond lengths resemble values reported for other diorganotellurium dihalides [1]. In the secondary coordination sphere of 1a and 1c, the tellurium atoms are intramolecularly coordinated (1,4-type) by the azomethine N atom of the Schiff base moieties (Figs. 1and 3). The shorter  $Te \cdots N$  internuclear distances and reduced N1-C2-C1 and Te-C1-C2 bond angles indicate the attractive secondary  $Te \cdots N$  interaction in 1a and 1c to be fairly strong though weaker than that operative  $[(C_6H_5)(2-Me_2NCH_2C_6H_4)TeX]^+X^ (Te \cdot \cdot \cdot N =$ in 2.389(12), X = Br [11] and 2.44(2) Å, X = I [12]) Presence of less strained five membered ring and positive charge on tellurium in case of the latter are probably responsible for the stronger secondary Te...N interac-





Fig. 1. Molecular structure of **1a** showing 30% probability displacement ellipsoids and the crystallographic numbering scheme.

tion. In contrast to many other diorganotellurium dichlorides, compound **1a** shows no secondary  $\text{Te} \cdots \text{Cl}$  interactions [1]. Also there is no evidence for secondary  $\text{Cl} \cdots \text{Cl}$  interactions in the crystal structure of **1a**; the

shortest distance between two Cl atoms in the crystal structure is 4.416(2) Å (between Cl1 and Cl2 of an adjacent molecule). However, the proximity of the tellurium to a methoxy group of an adjacent molecule suggests that a Te $\cdots \pi$  interaction is operative (Fig. 2). The Te··· $\pi$  and  $\pi$  (C = Y)··· $\pi$  (C = Y) contacts associated with quinoid electron delocalization across the paramethoxy phenyl as well as the NMBA fragments appear to be the intermolecular associative forces in absence of Te···Cl and Cl···Cl interactions. In the solid-state, compound 1c contains two crystallographically independent, yet similar conformers featuring Te1A and Te1B (Fig. 4). Besides the intramolecular N-coordination, the secondary coordination spheres of Te1A and Te1B are defined by a secondary Te···O interaction of 3.258(9) Å (O1A of an adjacent nitro group; symmetry operation: 1 - x, 2 - y, 3 - z) and a secondary Te···I interaction of 3.927(8) Å (I2B of an adjacent molecule; symmetry



Fig. 2. Crystal structure of 1a, viewed along the a-axis.



Fig. 3. Molecular structure of one of the two crystallographically independent conformers of 1c showing 30% probability displacement ellipsoids and the crystallographic numbering scheme.



Fig. 4. Crystal structure of 1c, viewed along the *a*-axis.

Table 1 Crystal data and structure refinement for **1a** and **1c** 

	1a	1c
Formula	$C_{21}H_{18}Cl_2N_2O_3Te \cdot 0.5CHCl_3$	$C_{21}H_{18}I_2N_2O_3Te$
Formula weight $(g \text{ mol}^{-1})$	604.56	727.77
Crystal system	Monoclinic	Triclinic
Crystal size, mm	$0.25 \times 0.30 \times 0.55$	$0.08 \times 0.40 \times 0.95$
Space group	C2/c	$P\overline{1}$
a (Å)	24.501(3)	10.0878(17)
b (Å)	7.6443(10)	15.491(3)
<i>c</i> (Å)	25.868(4)	16.247(3)
α (°)	90	77.616(3)
β (°)	101.421(2)	82.594(3)
γ (°)	90	86.944(4)
$V(\text{\AA}^3)$	4748.8(11)	2458.2(7)
Ζ	8	4
$D_{\rm calc} { m Mg}{ m m}^{-3}$	1.691	1.966
$T(\mathbf{K})$	103(2)	148(2)
$\mu (\mathrm{mm}^{-1})$	1.673	3.744
<i>F</i> (000)	2376	1360
Number of reflections collected	17616	12756
Number of independent reflections/ $R_{int}$	5787	8498
Number of reflections observed with $(I > 2\sigma(I))$	4900	7017
$R_1(F)(I > 2\sigma(I))$	0.022	0.046
$wR_2F^2$ (all data)	0.065	0.127
$(\Delta/\sigma)_{\rm max}$	0.00010(4)	0.00078(18)
Largest difference peak/hole (e Å <sup>-3</sup> )	0.551/-0.446	1.367 (near Te)/-1.390 (near Te)

operation: 1 - x, 3 - y, 1 - z), respectively. In the crystal structure of **1c** there is also a secondary I...I contact of 3.711(5) Å between I1A and I2B (symmetry operation: 1 - x, 3 - y, 2 - z), which is comparable to those reported previously for other diorganotellurium diiodides [13]. Compounds **1a** and **1c** crystallize in centrosymmetric space groups and in their crystal lattice symmetry-related *N*-(4-methylphenyl)-4-nitrobenzaledeneimine moieties are associated by extensive  $\pi$ -stacking and adopt head-to-tail conformations similar to the NMBA molecules in the triclinic modification (Figs. 2 and 4). Consequently, all putative second-order NLO effects are expected to be nullified [6].

## 3. Experimental

## 3.1. General

Preparative work was performed under dry nitrogen. The starting materials [{2-(4-nitrobenzylideneamino)-5-methyl}phenyl]mercury chloride and aryltellurium trichlorides were prepared according to literature methods [9,14]. Solution <sup>1</sup>H (300.13 MHz), <sup>13</sup>C (75.47 MHz) and <sup>125</sup>Te (85.29 MHz) NMR spectra were recorded in CDCl<sub>3</sub> on Varian 300 DRX, 300 Unity Plus and JEOL GX 270 NMR spectrometers, using Me<sub>4</sub>Si and Me<sub>2</sub>Te as internal standards. Solid-state <sup>125</sup>Te (126.2 MHz)

Table 2 Selected bond parameters (Å, °) for 1a and 1c

	1a (X = Cl)	1c (X = I)	
		Molecule A	Molecule B
Tel-X1	2.507(1)	2.933(3)	2.993(2)
Te1-X2	2.475(1)	3.022(3)	2.953(2)
Te1–N1	2.879(2)	2.958(7)	2.888(7)
Te1-C1	2.107(3)	2.159(7)	2.152(7)
Te1-C15	2.104(3)	2.172(7)	2.174(7)
X1–Te1–X2	175.04(2)	174.97(2)	170.41(2)
X1–Te1–N1	81.44(5)	79.17(9)	84.78(10)
X1–Te1–C1	87.65(6)	90.94(17)	84.44(17)
X1–Te1–C15	89.89(6)	92.04(17)	93.74(17)
X2–Te1–N1	96.58(5)	95.89(9)	85.67(10)
X2–Te1–C1	87.52(7)	85.33(17)	89.21(17)
X2–Te1–C15	89.67(6)	91.79(17)	94.17(17)
N1-Te1-C1	53.61(8)	53.79(19)	54.13(19)
N1-Te1-C15	150.48(8)	149.37(19)	151.24(19)
C1–Te1–C15	98.12(9)	97.61(23)	97.12(23)

CP MAS NMR spectra were acquired using a JEOL Eclipse Plus 400 NMR spectrometer equipped with a high speed locked 4 mm probe operating at spinning frequencies between 8 and 9 KHz. Experimental condition: pulse width 1 ms, relaxation delay 120 s, 400-900 transients. The isotropic chemical shifts are referenced against Me<sub>2</sub>Te using solid Te(OH)<sub>6</sub> as the secondary reference ( $\delta_{iso}$  692.1, 685.5) [15]. Definitions  $\delta_{iso}$ (ppm) =  $-\sigma_{iso} = -(\sigma_{11} + \sigma_{22} + \sigma_{33})/3; \zeta \text{ (ppm)} = \sigma_{33} - \sigma_{iso} \text{ and}$  $\eta = (\sigma_{22} - \sigma_{11})/(\sigma_{33} - \sigma_{iso})$  where  $\sigma_{11}$ ,  $\sigma_{22}$ and  $\sigma_{33}$  (ppm) are the principal tensor components of the shielding anisotropy (SA), sorted as follows  $|\sigma_{33} - \sigma_{iso}| > |\sigma_{11} - \sigma_{iso}| > |\sigma_{22} - \sigma_{iso}|$ . IR spectra were recorded as KBr pellets using a Perkin-Elmer RX1 Spectrometer. Microanalyses were carried out using a Carlo Erba 1108 make analyzer. Tellurium was estimated volumetrically and the halogen content gravimetrically as silver halide [16].

# 3.2. Synthesis of (4-methoxyphenyl)[{2-(4nitrobenzylideneamino)-5-methyl}phenyl]tellurium dichloride (1a)

solution of [{2-(4-nitrobenzylideneamino)-5-Α methyl}phenyl]mercury chloride (2.37 g, 5.00 mmol) and 4-methoxyphenyltellurium trichloride (1.70 g, 5.00 mmol) in chloroform (250 ml) was heated under reflux for 20 h. Precipitated HgCl<sub>2</sub> was filtered off and the solvents reduced to approximately one fifth in vacuo. Addition of petroleum ether (b.p. 40-60 °C) (50 mL) induced the precipitation of solid, which was recrystallized twice from CHCl<sub>3</sub> to give yellow crystals of **1a**. Yield: (1.88 g, 66%); m.p. 220 °C. Anal. Found: C, 42.68; H, 3.06; N, 4.80; Cl, 20.03; Te, 20.87. Calcd. for C21H18N2O3Cl2-Te · 0.5CHCl<sub>3</sub> (604.61): C, 42.71; H, 3.08; N, 4.63; Cl, 20.52; Te, 21.11. IR (cm<sup>-1</sup>): 1623.5 ( $v_{CH=N}$ ). <sup>1</sup>H NMR:  $\delta$  2.36 (s), 3.94 (s), 7.15–8.41 (m), 8.88 (s). <sup>13</sup>C NMR: δ 21.56, 55.63, 115.75, 116.49, 118.75, 124.29,

130.11, 130.54, 133.86, 137.27, 144.22, 156.65, 162.38. <sup>125</sup>Te NMR:  $\delta$  886.2. <sup>125</sup>Te MAS NMR:  $\delta_{iso}$  882.4 ( $\eta$ : 0.45  $\zeta$ : 313;  $\sigma_{11}$ : -1110,  $\sigma_{22}$ : -969,  $\sigma_{33}$ : -570).

Compounds 2 and 3 were prepared similarly. For (4methylphenyl)[{2-(4-nitrobenzylideneamino)-5-methyl} phenyl]tellurium dichloride, 2, Yield: (63%); m.p. 230 °C. Anal. Found: C, 47.47; H, 3.21; N, 5.46; Cl, 13.07; Te, 25.07. Calcd. for C<sub>21</sub>H<sub>18</sub>N<sub>2</sub>O<sub>2</sub>Cl<sub>2</sub>Te (528.89): C, 47.69; H, 3.43; N, 5.30; Cl, 13.41; Te, 24.12. IR (cm<sup>-1</sup>): 1624.0 ( $\nu_{CH=N}$ ). <sup>1</sup>H NMR:  $\delta$  2.38 (s), 2.56 (s), 7.42–8.40 (m), 8.88 (s). <sup>13</sup>C NMR:  $\delta$  21.47, 76.58, 116.42, 124.21, 130.05, 130.63, 130.83, 133.78, 135.42, 140.44, 141.13, 142.49, 156.56.  $^{125}$ Te NMR:  $\delta$  880.0. For (phenyl)[{2-(4-nitrobenzylideneamino)-5-methyl} phenyl]tellurium dichloride, 3, Yield: (55%); m.p. 252 °C. Anal. Found: C, 45.88; H, 3.32; N, 5.18; Cl, 14.00; Te, 25.12. Calcd. for C<sub>20</sub>H<sub>16</sub>N<sub>2</sub>O<sub>2</sub>Cl<sub>2</sub>Te (514.86): C, 46.65; H, 3.13; N, 5.44; Cl, 13.77; Te, 24.78. IR (cm<sup>-1</sup>): 1621.5 ( $v_{CH=N}$ ). <sup>1</sup>H NMR:  $\delta$  2.356 (s), 7.428– 8.417 (m), 8.897 (s). <sup>13</sup>C NMR:  $\delta$  21.62, 77.09, 116.58, 124.37, 129.97, 130.18, 130.58, 131.91, 134.00, 135.68, 140.51, 141.36, 156.75. <sup>125</sup>Te NMR:  $\delta$  881.1.

# 3.3. Synthesis of (4-methoxyphenyl)[{2-(4nitrobenzylideneamino)-5-methyl}phenyl]tellurium dibromide (**1b**) and diiodide (**1c**)

A solution of **1a** (550 mg, 1.00 mmol) in chloroform (50 mL) was stirred with a twofold excess of KI for 12 h. A colour change from yellow to red occurred. The mixture was filtered and the solvents reduced to approximately one fifth in vacuo. Addition of petroleum ether 40–60 °C (10 mL) induced crystallization, providing red/brown crystals of **1c**. Yield (611 mg, 84%); m.p. 196 °C. Anal. Found: C, 34.88; H, 2.54; N, 4.03; I, 34.52; Te, 17.07. Calcd. for C<sub>21</sub>H<sub>18</sub>N<sub>2</sub>O<sub>3</sub>I<sub>2</sub>Te (727.82): C, 34.66; H, 2.42; N, 3.84; I, 34.87; Te, 17.53. IR (cm<sup>-1</sup>): 1615.5 ( $v_{CH=N}$ ). <sup>1</sup>H NMR:  $\delta$  2.39 (s), 3.93 (s), 7.03–8.43 (m), 8.90 (s). <sup>13</sup>C NMR:  $\delta$  21.56, 55.62, 115.83, 116.57, 124.29, 131.10, 130.13, 133.86, 138.73, 144.18, 156.46, 162.31. <sup>125</sup>Te NMR:  $\delta$  840.7.

**1b** was prepared similarly from **1a** and KBr. Yield: (82%); m.p. 180 °C. Anal. Found: C, 39.73; H, 2.64; N, 4.60; Br, 24.10; Te, 21.07. Calcd. for  $C_{21}H_{18}N_2O_3Br_2Te$  (633.79): C, 39.80; H, 2.86; N, 4.42; Br, 25.21; Te, 20.13. IR (cm<sup>-1</sup>): 1620.5 ( $v_{CH=N}$ ). <sup>1</sup>H:  $\delta$  2.355 (s), 3.935 (s), 7.154–8.407 (m), 8.881 (s). <sup>13</sup>C NMR:  $\delta$  21.54, 55.59, 116.11, 116.77, 124.34, 130.18, 132.19, 133.71, 140.46, 144.12, 156.07, 162.15. <sup>125</sup>Te NMR:  $\delta$  777.1.

## 4. Crystallography

Single crystals of **1a** and **1c** suitable for X-ray crystallography were obtained by slow evaporation of chloroform solutions. Intensity data were collected on a Bruker PS4 diffractometer with graphite-monochromated Mo K $\alpha$  (0.7107 Å) radiation. Data were reduced and corrected for absorption using the programs SAINT and SADABS [17]. The structures were solved by direct methods and difference Fourier synthesis using SHELXS-97 implemented in the program WINGX 2002 [18]. 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. Crystallographic parameters and details of the data collection and refinement are given in Table 1. Figures were created using DIAMOND [19].

## 5. Supplementary material

Crystallographic data for the structure analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC No. for **1a** 246743 and for **1c** is 246745. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, U.K. Fax: +44-1223-336-033; or E-mail: deposite@ccdc.cam.ac.uk or www: http:// www.ccdc.cam.ac.uk.

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