

# Reaction of Tellurium Tetrachloride with Propargyl Halides

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**Abstract**—The reaction of tellurium tetrachloride with propargyl bromide in boiling benzene regio- and stereoselectively afforded bis[(*Z*)-3-bromo-2-chloroprop-1-en-1-yl]tellurium dichloride which was readily reduced to bis[(*Z*)-3-bromo-2-chloroprop-1-en-1-yl] telluride. Propargyl chloride reacted with tellurium tetrachloride preliminarily stored for several months (and containing decomposition products) to give a mixture of bis[(*Z*)-2,3-dichloroprop-1-en-1-yl]tellurium dichloride and regioisomeric [(*Z*)-1,3-dichloroprop-1-en-2-yl]-[(*Z*)-2,3-dichloroprop-1-en-1-yl]tellurium dichloride at a ratio of 3:2, whereas the latter was formed as the sole product in the reaction with freshly prepared tellurium tetrachloride. Reduction of the regioisomer mixture produced the corresponding tellurides. The structure of the isolated compounds was confirmed by  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{125}\text{Te}$  NMR spectra.

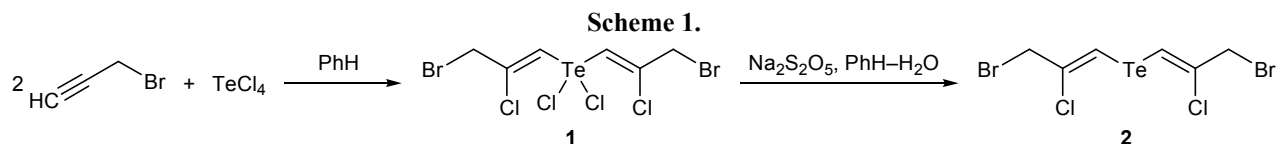
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We previously reported on the synthesis of bis[(*Z*)-2,3-dichloroprop-1-en-1-yl]tellurium dichloride and bis[(*Z*)-2,3-dichloroprop-1-en-1-yl] telluride via regio- and stereoselective addition of tellurium tetrachloride to propargyl chloride and subsequent reduction of the resulting tellurium dichloride with sodium metabisulfite [1]. Unlike reactions of propargyl halides with sulfur dichloride [2] and selenium dichloride and dibromide [2, 3], which followed the anti-Markovnikov addition pattern, tellurium tetrachloride reacted with propargyl chloride under analogous conditions [1] to produce only the Markovnikov *syn* adduct. It is known that tellurium tetrahalides add to terminal alkynes exclusively according to Markovnikov [4, 5]. However, the anti-Markovnikov adduct was obtained in the reaction of  $\text{TeCl}_4$  with trimethylsilylacetylene [4, 5], which suggests that anti-Markovnikov adducts can be selectively obtained by reactions of tellurium tetrahalides with alkynes. In continuation of our studies on reactions of tellurium tetrachloride with propargyl halides, the present work was aimed at synthesizing analogs of bis[(*Z*)-2,3-dichloroprop-1-en-1-yl]tellurium dichloride and bis[(*Z*)-2,3-dichloroprop-

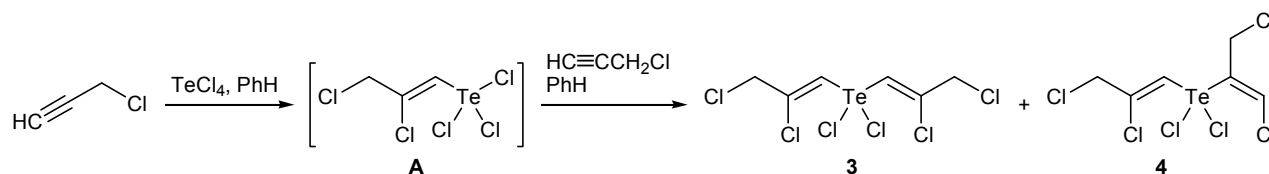
1-en-1-yl] telluride containing bromomethyl groups with a more nucleofugal bromine atom.

The reaction of tellurium tetrachloride with excess propargyl bromide in boiling benzene (reaction time 18 h) was regio- and stereoselective, and the product was the corresponding Markovnikov *syn* adduct, bis[(*Z*)-3-bromo-2-chloroprop-1-en-1-yl]tellurium dichloride (**1**, 83%). The subsequent reduction of **1** with sodium metabisulfite gave 84% of bis[(*Z*)-3-bromo-2-chloroprop-1-en-1-yl] telluride (**2**) (Scheme 1).

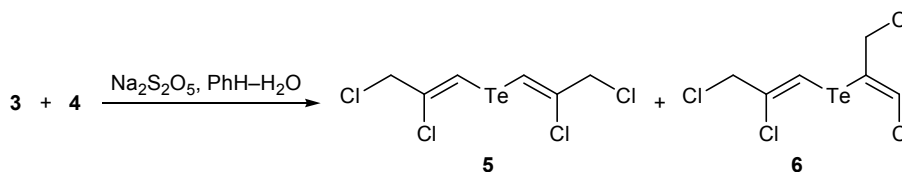
The structure of **1** and **2** was confirmed by the  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{125}\text{Te}$  NMR data, as well as by the mass spectrum of **2** which contained no molecular ion peak but fragment ion peaks with  $m/z$  282 [ $M - \text{CH}=\text{CClCH}_2\text{Br} - \text{H}$ ] $^+$  and 153 [ $\text{CH}=\text{CClCH}_2\text{Br}$ ] $^+$ . Compounds **1** and **2** displayed in the  $^1\text{H}$  NMR spectra signals from vinylic and methylene protons, and the  $=\text{CHCl}$  signal of **1** appeared in a weaker field relative to the corresponding signal of **2**, while the chemical shifts of the methylene protons in **1** and **2** were almost similar. Analogous pattern was observed in the  $^{13}\text{C}$  NMR spectrum where the doublet of triplets belonging to the  $sp^2$ -carbon atom



Scheme 2.



Scheme 3.



of **1** was also displaced downfield relative to the =CH signal of **2**; the positions of the  $\text{CH}_2$  signals (t.d.) in the spectra of both compounds almost coincided with each other. Fine structure analysis of the  $^{13}\text{C}$  NMR spectra clearly indicated the formation of Markovnikov adducts: as followed from the direct  $^{13}\text{C}$ - $^1\text{H}$  coupling constants for =CH carbon nuclei [ $^1J_{\text{CH}} = 185$  (**1**), 170.6 Hz (**2**)], that carbon atom is linked to the chalcogen atom [6]. The same is proved by the direct  $^{13}\text{C}$ - $^{125}\text{Te}$  coupling constant ( $^1J_{\text{CTe}} = 297$  Hz [7–10]) in the  $^{13}\text{C}$  NMR spectrum of **2**. The *Z,Z* configuration of the double bonds in **1** and **2** was assigned on the basis of the  $^{13}\text{C}$ - $^1\text{H}$  coupling constants between the methylene carbon atom and =CH hydrogen atom ( $^3J_{\text{CH}} = 4$  Hz) [11]. In addition, the 2D  $^1\text{H}$  NOESY spectrum of **2** showed cross peaks between the vinylic and methylene protons.

Tellurium dichloride **1** is stable, and no its isomerization occurred on prolonged heating in boiling benzene (for 25 h).

Further study of the reaction of tellurium tetrachloride with propargyl chloride has revealed that the reaction course is influenced by impurities present in tellurium tetrachloride. The reaction is regio- and stereoselective [1] with tellurium tetrachloride prepared by the chlorination of tellurium with sulfur chloride [12]. When we used commercial  $\text{TeCl}_4$  (from Aldrich) preliminarily stored for several months in a refrigerator, we obtained 93% of a mixture of bis-[(*Z*)-2,3-dichloroprop-1-en-1-yl]tellurium dichloride (**3**) and regioisomeric [(*Z*)-1,3-dichloroprop-1-en-2-yl][(*Z*)-2,3-dichloroprop-1-en-1-yl]tellurium dichloride (**4**) at a ratio of 3:2 (Scheme 2).

The reduction of a mixture of **3** and **4** in benzene with an aqueous solution of sodium metabisulfite gave a mixture of bis[(*Z*)-2,3-dichloroprop-1-en-1-yl] tellu-

ride (**5**) and (*Z*)-1,3-dichloroprop-1-en-2-yl (*Z*)-2,3-dichloroprop-1-en-1-yl telluride (**6**) at the same ratio (3:2) in an overall yield of 75%.

The formation of isomeric tellurium dichloride **4** and telluride **6** in the above reactions was confirmed by the  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{125}\text{Te}$  NMR data. In the  $^1\text{H}$  NMR spectra ( $\text{DMSO-}d_6$ ), the =CHTe and =CHCl vinylic protons resonated, respectively, at  $\delta$  8.32 and 7.66 ppm for **4** and  $\delta$  7.30 and 6.84 ppm for **6**. The corresponding carbon signals appeared in the  $^{13}\text{C}$  NMR spectra as doublets of triplets at  $\delta_{\text{C}}$  129.28 and 138.12 ppm (**4**) and  $\delta_{\text{C}}$  110.00 and 131.58 ppm (**6**). Analysis of the direct  $^{13}\text{C}$ - $^1\text{H}$  coupling constants clearly showed that the downfield carbon signal belongs to the carbon atom linked to tellurium [ $^1J_{\text{CH}} = 188.3$  (**4**), 173.6 Hz (**6**)] and that the upfield signal belongs to the carbon atom linked to chlorine [ $^1J_{\text{CH}} = 206$  (**4**), 204.2 Hz (**6**)] [6]. Furthermore, this assignment was confirmed by the  $^{13}\text{C}$ - $^{125}\text{Te}$  coupling constants for the =CHTe and =CHCl carbon atoms in **4** ( $^1J_{\text{CTe}} = 300.8$  Hz and  $^2J_{\text{CTe}} = 35.3$  Hz, respectively) [7–10]. The methylene carbon atoms resonated in the  $^{13}\text{C}$  NMR spectra of **4** and **6** as triplets of doublets. The presence of cross-peaks between the vinylic and methylene protons of both propenyl groups in the 2D  $^1\text{H}$  NOESY spectra indicated their *cis* orientation with respect to the C=C double bond.

The formation of unsymmetrical Markovnikov/anti-Markovnikov adducts together with symmetrical bis-anti-Markovnikov adducts was observed by us previously in the electrophilic addition of sulfur dichloride to propargyl halides [2]. Isomeric tellurium dichloride **4** is likely to be formed via addition of primary 1:1 adduct **A** to the second propargyl chloride molecule both according and contrary to the Markovnikov rule with gradual accumulation of the anomalous anti-Markovnikov adduct (Scheme 2). This mechanism

is supported by gradual increase of the intensity of signals of both isomers **3** and **4** with simultaneous decrease of the intensity of the vinylic proton signal of intermediate adduct **A** in the  $^1\text{H}$  NMR spectra of the reaction mixture. On the other hand, tellurium dichloride **3** remained intact on heating in boiling benzene for several days, which ruled out its isomerization into **4**. The sharp increase of the rate of the anomalous anti-Markovnikov addition in the second step of the reaction with commercial tellurium tetrachloride may be rationalized by the presence of decomposition products (in a sample stored for a long time) such as tellurium oxychloride, tellurium dioxide, HCl, and other micro impurities that can catalyze this process. Freshly prepared tellurium tetrachloride contains no such impurities, and its reaction with propargyl chloride is regio- and stereoselective.

Thus, our results have revealed loss of regioselectivity in the reaction of propargyl chloride with commercial tellurium tetrachloride (Aldrich) preliminarily stored for several months at  $-4^\circ\text{C}$  and hence containing decomposition products. In this case, a 3:2 mixture of the symmetrical bis-Markovnikov adduct and unsymmetrical Markovnikov/anti-Markovnikov adduct is formed.

## EXPERIMENTAL

The  $^1\text{H}$  (400.13 MHz),  $^{13}\text{C}$  (100.61 MHz), and  $^{125}\text{Te}$  (76.30 MHz) NMR spectra were recorded on a Bruker DPX-400 spectrometer using tetramethylsilane ( $^1\text{H}$ ,  $^{13}\text{C}$ ) or  $\text{Me}_2\text{Te}$  ( $^{125}\text{Te}$ ) as internal standard. The mass spectrum of **2** was obtained using an Agilent 5975 mass-selective detector (electron impact, 70 eV). The spectral studies were performed using the equipment of the Baikal Joint Analytical Center, Siberian Branch, Russian Academy of Sciences.

Propargyl chloride was prepared by chlorination of propargyl alcohol with thionyl chloride in benzene [13]. Tellurium tetrachloride was either commercial product (Aldrich) or prepared by chlorination of tellurium with sulfur chloride [12].

**Bis[(Z)-3-bromo-2-chloroprop-1-en-1-yl]tellurium dichloride (1).** A solution of 1.900 g (7.1 mmol) of  $\text{TeCl}_4$  and 5.158 g (43.4 mmol) of propargyl bromide (a 80% solution in toluene) in 55 mL of benzene was heated for 18 h under reflux. The black solid was filtered off, and the solvent and excess propargyl bromide were removed from the filtrate under reduced pressure. Yield 2.987 g (83%), brown powder, mp 103–105°C (decomp.).  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ),

$\delta$ , ppm 4.27 s (4H,  $\text{CH}_2$ ), 7.88 s (2H,  $=\text{CHTe}$ ).  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ),  $\delta_{\text{C}}$ , ppm: 32.43 t.d ( $\text{CH}_2$ ,  $^1J_{\text{CH}} = 156.5$ ,  $^3J_{\text{CH}} = 4$  Hz), 125.50 d.t ( $=\text{CHTe}$ ,  $^1J_{\text{CH}} = 185$ ,  $^3J_{\text{CH}} = 4$  Hz), 144.28 q ( $=\text{CCl}$ ,  $^2J_{\text{CH}} = 4.5$  Hz).  $^{125}\text{Te}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta_{\text{Te}}$  687.2 ppm, t ( $^2J_{\text{TeH}} = 6.5$  Hz). Found, %: C 14.27; H 1.47; Cl 27.93; Br 31.38; Te 24.75.  $\text{C}_6\text{H}_6\text{Cl}_4\text{Br}_2\text{Te}$ . Calculated, %: C 14.20; H 1.19; Cl 27.95; Br 31.50; Te 25.15. *M* 507.33.

**Bis[(Z)-3-bromo-2-chloroprop-1-en-1-yl] telluride (2).** A solution of 2.49 g (11.2 mmol) of  $\text{Na}_2\text{S}_2\text{O}_5$  in 6 mL of water was added to a solution of 0.569 g (1.12 mmol) of tellurium dichloride **1** in 20 mL of benzene, and the mixture was stirred for 12 h at room temperature. The benzene layer was separated, the aqueous layer was extracted with chloroform, the extract was combined with the organic phase and dried over  $\text{MgSO}_4$ , and the solvent was removed under reduced pressure. Yield 0.409 g (84%), white powder, mp 75–77°C.  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ),  $\delta$ , ppm: 4.22 s (4H,  $\text{CH}_2$ ), 7.31 s (2H,  $=\text{CHTe}$ ).  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ),  $\delta_{\text{C}}$ , ppm: 35.66 t.d ( $\text{CH}_2$ ,  $^1J_{\text{CH}} = 155.9$ ,  $^3J_{\text{CH}} = 4.2$  Hz), 109.46 d.t ( $=\text{CHTe}$ ,  $^1J_{\text{TeC}} = 297$ ,  $^1J_{\text{CH}} = 170.6$ ,  $^3J_{\text{CH}} = 4.2$  Hz), 136.20–136.36 m ( $=\text{C}$ ,  $^2J_{\text{TeC}} = 40$  Hz).  $^{125}\text{Te}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta_{\text{Te}}$  543.1 ppm, t ( $^2J_{\text{TeH}} = 6.2$  Hz). Mass spectrum,  $m/z$  ( $I_{\text{rel}}$ , %): 282 (6) [ $M - \text{CH}=\text{CClCH}_2\text{Br} - \text{H}$ ] $^+$ , 234 (4), 190 (12), 153 (50) [ $\text{CH}=\text{CClCH}_2\text{Br}$ ] $^+$ , 127 (19), 117 (24), 109 (27), 83 (100). Found, %: C 16.81; H 1.59; Br 36.31; Cl 15.93; Te 29.46.  $\text{C}_6\text{H}_6\text{Cl}_2\text{Br}_2\text{Te}$ . Calculated, %: C 16.51; H 1.39; Br 36.62; Cl 16.25; Te 29.24. *M* 436.42.

**Reaction of propargyl chloride with tellurium tetrachloride.** A solution of 1.350 g (5 mmol) of  $\text{TeCl}_4$  and 2.250 g (30 mmol) of propargyl chloride in 25 mL of benzene was heated for 14 h under reflux with stirring. The precipitate was filtered off, the solvent and excess propargyl chloride were removed under reduced pressure, and the residue was washed with hexane and dried under reduced pressure. Yield 1.624 g (93%), a mixture of compounds **3** and **4** at a ratio of 3:2 (according to the  $^1\text{H}$  NMR data).

**Bis[(Z)-2,3-dichloroprop-1-en-1-yl]tellurium dichloride (3).**  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm: in  $\text{CDCl}_3$ : 4.38 d (4H,  $\text{CH}_2$ ,  $^3J = 1.2$  Hz), 7.94 t (2H,  $=\text{CHTe}$ ,  $^3J = 1.2$  Hz); in  $\text{DMSO}-d_6$ : 4.76 s (4H,  $\text{CH}_2$ ), 8.52 s (2H,  $=\text{CHTe}$ ); published data [1]: 4.75 s (4H,  $\text{CH}_2$ ), 8.51 s (2H,  $=\text{CH}$ ).  $^{13}\text{C}$  NMR spectrum ( $\text{DMSO}-d_6$ ),  $\delta_{\text{C}}$ , ppm: 47.25 t.d ( $\text{CH}_2$ ,  $^1J_{\text{CH}} = 157.0$ ,  $^3J_{\text{CH}} = 4.0$  Hz), 127.02 d.t ( $=\text{CHTe}$ ,  $^1J_{\text{CH}} = 188.3$ ,  $^3J_{\text{CH}} = 3.6$ ,  $^1J_{\text{TeC}} = 258$  Hz), 143.04 s ( $=\text{C}$ ,  $^2J_{\text{TeC}} = 44.4$  Hz); published data [1]: 47.03 ( $\text{CH}_2$ ), 127.08 ( $=\text{CHTe}$ ,  $^1J_{\text{TeC}} = 260$  Hz), 143.08

(C=).  $^{125}\text{Te}$  NMR spectrum (DMSO- $d_6$ ):  $\delta_{\text{Te}}$  723.5 ppm ( $\delta_{\text{Te}}$  724 ppm [1]).

**[(Z)-1,3-Dichloroprop-1-en-2-yl][(Z)-2,3-dichloroprop-1-en-1-yl]tellurium dichloride (4).**  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm: in  $\text{CDCl}_3$ : 4.38 s [2H, =C(Cl)CH $_2$ ], 5.02 d [2H, =C(Te)CH $_2$ ], 7.33 s (1H, =CHCl), 7.86 t (1H, =CHTe); in DMSO- $d_6$ : 4.73 s [2H, =C(Cl)CH $_2$ ], 4.95 s [2H, =C(Te)CH $_2$ ], 7.66 s (1H, =CHCl), 8.32 s (1H, =CHTe).  $^{13}\text{C}$  NMR spectrum (DMSO- $d_6$ ),  $\delta_{\text{C}}$ , ppm: 46.95 t.d [=C(Te)CH $_2$ ,  $^1J_{\text{CH}} = 156.1$ ,  $^3J_{\text{CH}} = 4.0$  Hz], 47.25 t.d [=C(Cl)CH $_2$ ,  $^1J_{\text{CH}} = 157.0$ ,  $^3J_{\text{CH}} = 4.0$  Hz], 129.28 d.t (=CHTe,  $^1J_{\text{CH}} = 188.3$ ,  $^3J_{\text{CH}} = 4.0$ ,  $^1J_{\text{CTe}} = 300.8$  Hz), 138.12 d.t (=CHCl,  $^1J_{\text{CH}} = 206$ ,  $^3J_{\text{CH}} = 6$ ,  $^2J_{\text{CTe}} = 35.3$  Hz), 143.04 s (=C,  $^2J_{\text{CTe}} = 44.4$  Hz), 143.43 s (=C).  $^{125}\text{Te}$  NMR spectrum (DMSO- $d_6$ ):  $\delta_{\text{Te}}$  847.0 ppm. Found, %: C 17.10; H 1.48; Cl 50.52; Te 30.76.  $\text{C}_6\text{H}_6\text{Cl}_6\text{Te}$ . Calculated, %: C 17.22; H 1.45; Cl 50.84; Te 30.50.

**Reduction of isomeric bis(dichloropropenyl)-tellurium dichlorides 3 and 4.** A solution of 1.467 g (6.6 mmol) of  $\text{Na}_2\text{S}_2\text{O}_5$  in 7 mL of water was added to a solution of 0.274 g (0.65 mmol) of isomer mixture 3/4 in 5 mL of benzene, and the mixture was stirred for 5 h at room temperature. The mixture was treated with 10 mL of water and extracted with chloroform, and the extract was dried over  $\text{CaCl}_2$  and evaporated to isolate 0.170 mg (75%) of a mixture of compounds 5 and 6 at a ratio of 3:2 ( $^1\text{H}$  NMR).

**Bis[(Z)-2,3-dichloroprop-1-en-1-yl] telluride (5).**  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ),  $\delta$ , ppm: 4.26 s (4H, CH $_2$ ), 7.28 s (2H, =CHTe); published data [1]: 4.24 s (4H, CH $_2$ ), 7.25 s (2H, =CH).  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ),  $\delta_{\text{C}}$ , ppm: 48.45 t (CH $_2$ ,  $^1J_{\text{CH}} = 154.2$ ,  $^3J_{\text{CH}} = 4.0$  Hz), 108.12 d (=CHTe,  $^1J_{\text{CH}} = 171$  Hz), 135.08 s (=C); published data [1]: 48.21 (CH $_2$ ), 107.83 (=CHTe,  $^1J_{\text{TeC}} = 294$  Hz), 131.52 (=C).  $^{125}\text{Te}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta_{\text{Te}}$  525.1 ppm ( $\delta_{\text{Te}}$  528 ppm [1]).

**(Z)-1,3-Dichloroprop-1-en-2-yl (Z)-2,3-dichloroprop-1-en-1-yl telluride (6).**  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ),  $\delta$ , ppm: 4.27 s [2H, =C(Te)CH $_2$ ], 4.63 s [2H, =C(Cl)CH $_2$ ], 6.84 s (1H, =CHCl), 7.30 s (1H, =CHTe).  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ),  $\delta_{\text{C}}$ , ppm: 48.45 t.d [=C(Te)CH $_2$ ,  $^1J_{\text{CH}} = 154.2$ ,  $^3J_{\text{CH}} = 4.0$  Hz], 47.33 t.d [=C(Cl)CH $_2$ ,  $^1J_{\text{CH}} = 153.7$ ,  $^3J_{\text{CH}} = 8.4$  Hz], 110.00 d.t

(=CHTe,  $^1J_{\text{CH}} = 173.6$ ,  $^3J_{\text{CH}} = 4.2$  Hz), 131.58 d.t (=CHCl,  $^1J_{\text{CH}} = 204.2$ ,  $^3J_{\text{CH}} = 5.8$  Hz), 135.08 s (=C), 135.08 s (=C).  $^{125}\text{Te}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta_{\text{Te}}$  635.4 ppm. Found, %: C 20.30; H 1.85; Cl 41.12; Te 36.46.  $\text{C}_6\text{H}_6\text{Cl}_4\text{Te}$ . Calculated, %: C 20.74; H 1.74; Cl 40.81; Te 36.72. *M* 347.52.

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