Heteroatom Chemistry
Volume 22, Number 3/4, 2011

Free Radical Addition of α-Telluroesters to Alkenes

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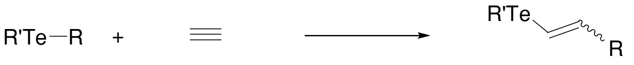
Received 19 October 2010; revised 11 January 2011

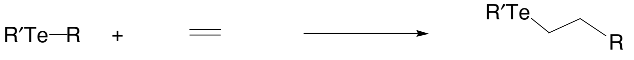
ABSTRACT: *α-(Phenyltelluro)esters* **2** *reacted with electron-rich alkenes* **3** *in the presence of a catalytic amount of azobisisobutyronitrile (AIBN) as an initiator to give the corresponding addition products* **4**. *This carbotelluration of alkenes proceeded via a radical chain mechanism involving the bimolecular homolytic substitution (S_H2) reaction on tellurium as the key step, wherein tellurium plays an important role in a kinetic fashion to realize the rapid group transfer process resulting in suppressing the competing olefin polymerization.* © 2011 Wiley Periodicals, Inc. Heteroatom Chem 22:518–522, 2011; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.20716

INTRODUCTION

Ever since we disclosed free radical addition of diorganyl tellurides to alkynes to form alkenyl tellurides in 1992 [1], as exemplified in Eq. (1), several examples of carbotelluration of alkynes have been reported so far by us [2,3] and other groups [4,5]. Carbotelluration of alkynes would provide useful synthetic tools in organic chemistry because new carbon–carbon and carbon–tellurium bonds are formed in one step giving rise to vinyl tellurides, which can allow further synthetic manipulation [6]. If alkene inserts between Te—R bond similarly as in the case of alkynes, alkyl tellurides can be formed as shown in Eq. (2). However, this reaction, carbotelluration of alkenes, does not seem to be an easy process to proceed efficiently (vide infra).



(1)



(2)

Plausible mechanisms of carbotelluration of alkenes (Eq. (2)) are outlined in Scheme 1 assuming that the reaction proceeds by a similar mechanism as the reaction of alkynes. Initiation steps are composed of the addition of 1-cyano-1-methylethyl radical (In•), generated by thermal decomposition of azobisisobutyronitrile (AIBN), to alkene to form an alkyl radical **1′**, and the abstraction of the PhTe group from telluride PhTeR **2** by **1′** to provide intermediate radical R• (steps 1 and 2). Steps 3 and 4 constitute the propagation sequence. Addition of R• to alkene **3** and the following abstraction of the PhTe group from the starting telluride **2** affords the addition product **4** with regeneration of the key

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Dedicated to Professor Kin-ya Akiba on the occasion of his 75th birthday.

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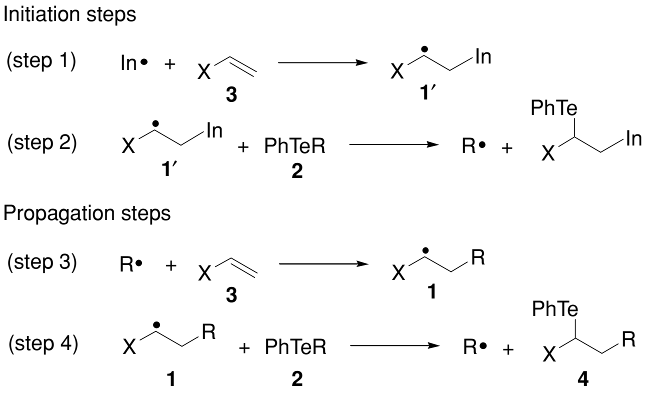
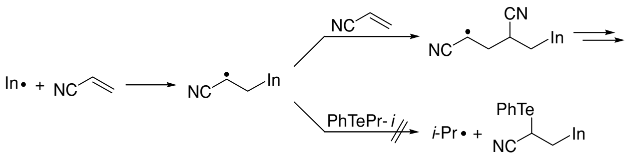


SCHEME 1 Carbotelluration of alkenes.

intermediate R•. Thus, both steps 3 and 4 should proceed rapidly to achieve efficient radical chain sequence giving carbotelluration product **4**. Since alkyl radicals are known to add to alkenes more rapidly than to alkyne [7], the key process of this transformation is a bimolecular homolytic substitution (S_H2) reaction on Te (step 4); i.e., if this process is slow, radical addition to olefins predominates to afford polymerization or telomerization products [8]. As a successful example of carbotelluration of alkenes, Crich and coworkers [9] reported an intramolecular version employing tellurobenzoates carrying olefinic moiety at the ortho position. However, there are no examples of intermolecular carbotelluration of alkenes.

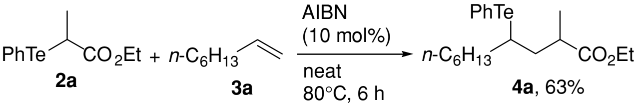
RESULTS AND DISCUSSION

First, we carried out a reaction of PhTePr-*i* with acrylonitrile at 80°C using AIBN (10 mol%) as an initiator; however, acrylonitrile polymerized completely and PhTePr-*i* was recovered unreacted (Eq. (3)). This result suggests that 1-cyanoalkyl radical attacked acrylonitrile predominantly rather than telluride probably because alkyl radical conjugated with a CN group formed by radical addition to acrylonitrile is more stable than *i*-Pr radical, which can be generated by S_H2 on Te (Eq. (3)). The S_H2 reaction on Te is an equilibrium process in which the most stable radical eliminates from hypervalent intermediates [10]. Yamago and Yoshida applied similar systems to radical polymerizations of styrene derivatives [11].



(3)

Then we examined the reaction of α-(phenyltelluro)esters **2** with electron-rich alkenes due to the following reasons: (i) $PhTeCH_2CO_2Et$ **2b** is known to undergo rapid bimolecular homolytic substitution on Te with *n*-octyl radical in a rate of $2.3 \times 10^7\ M^{-1}\ s^{-1}$ at 50°C [12]. (ii) Radicals **1** ($X = n\text{-}C_6H_{13}$, $R^\bullet = CH_2CO_2R'$) are not stabilized by conjugation and are considered to have similar reactivity as *s*-octyl radical toward α-telluroesters **2**. (iii) We already developed a convenient synthetic method of α-telluroesters [13]. As expected, treatment of α-(phenyltelluro)esters **2a** with the same equivalent of 1-octene **3a** in the presence of 10 mol% of AIBN at 80°C for 6 h resulted in the formation of carbotelluration product **4a** in 63% yield (Eq. (4)).

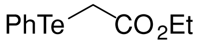
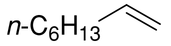
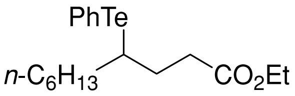
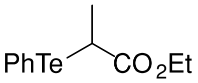
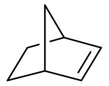
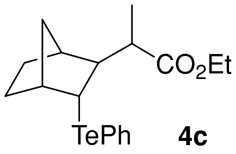
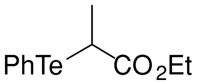
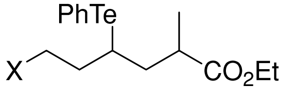
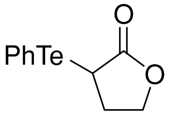
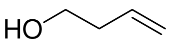
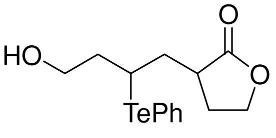


(4)

Several examples of carbotelluration of alkenes are summarized in Table 1. α-Telluroesters having an H atom or a methyl substituent on the α-carbon added to the terminal double bonds regioselectively to give the corresponding adducts **4** in good yields. However, as generally seen in radical reactions, the reaction was sluggish toward internal alkenes such as 4-octene. The yields of adducts were significantly improved when an excess amount of alkene was used. For example, when 5 equiv of the alkene was used, the corresponding adduct was obtained almost quantitatively based on the telluride (run 3). Alkenes having a functional group such as Br, OH, or CO_2H also gave the products in good yields. It is worth to note that bromoalkane moiety of **3c** is tolerant to the present reaction because bromoalkanes can also undergo atom transfer reaction under radical conditions (run 4). Similar addition reactions of α-bromoesters to alkenes in the presence of peroxide, known as the Kharasch reaction, are well recognized [14]. However, under the conditions of run 4, only 27% of the expected adduct was obtained by the reaction of ethyl 2-bromopropioate with 1-octene. The higher reactivity of α-telluroester over α-bromoester would be ascribable to more facile S_H2 process on Te than on Br via stable hypervalent radical intermediates of tellurium (it was suggested that the S_H2 reaction on Te is about 10 times faster than that on I; see ref. 1a.) and also to smaller bond dissociation energy of C—Te bonds than that of C—Br bonds.

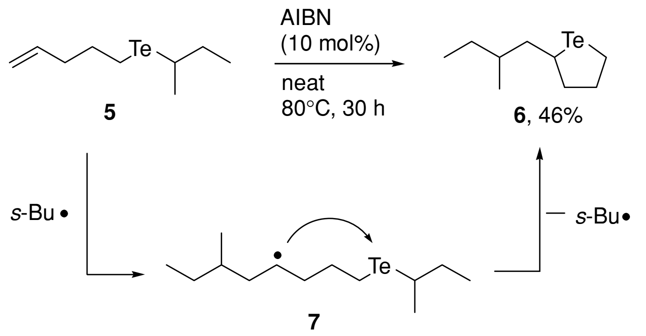
Tellurides having a C—C double bond at an appropriate position undergo a cyclization reaction. For example, when *s*-butyl 4-pentenyl telluride

TABLE 1 Radical Addition of α-(Phenyltelluro)esters to Alkenes[a]

Run	*Telluride*	*Alkene*	*Product*	*Yield (%)*[b]
1	 **2b**	 **3a**	 **4b**	$49(72)^{c}$
2	 **2a**	 **3b**		54^{d}
3				63^{e} $(97)^{c,e}$
4	**2a**	**3c**: X = Br	**4d**	61^{e}
5	**2a**	**3d**: X = OH	**4e**	67^{e}
6	**2a**	**3e:** X = CO_2H	**4f**	66^{e}
	 **2c**	 **3d**	 **4g**	65^{e}

[a] Conditions: telluride (1.0 mmol), alkene (1.0 mmol), AIBN (0.1 mmol), 80°C, 6 h.
[b] Isolated yield.
[c] 5.0 mmol of alkene was used.
[d] A 52/48 mixture of (exo, endo)/(exo, exo) isomers.
[e] A ca. 1/1 mixture of two isomers.

5 was heated at 80°C in the presence of AIBN, cyclization took place to afford the corresponding telluracyclopentane **6** in 46% yield (Eq. (5)). In this reaction, *s*-butyl radical adds to a C—C double bond to form **7** and a subsequent intramolecular S_H2 reaction on Te affords telluracyclopentane **6**. This result demonstrates that the intramolecular S_H2 reaction on Te can take place efficiently even when substantial energy gain is not expected as in the present case of secondary alkyl–secondary alkyl exchange.



(5)

In summary, free radical addition of tellurides to alkenes was attained by the use of α-telluroesters and electron-rich alkenes. The structures of tellurides and alkenes exert crucial effects on the thermodynamics of each reaction process to achieve this carbotelluration of alkenes efficiently.

EXPERIMENTAL

General

Melting points were determined on a Yanagimoto micro melting point apparatus. ^{1}H and ^{13}C NMR spectra were recorded on a JEOL (JEOL Ltd., Akishimashi, Tokyo, Japan) JNM-GSX-270 (270 and 68 MHz, respectively) spectrometer using Me_4Si (in $CDCl_3$) as an internal standard. Infrared (IR) spectra were determined on a Perkin-Elmer (PerkinElmer, Inc., Waltham, MA) model 1600 spectrometer. Mass spectra were obtained on a JEOL (JEOL Ltd.) JMS-DX303. Elemental analyses were performed on PerkinElmer 240C apparatus.

General Procedure

Into a glass tube (ϕ = 6 mm), α-telluroester **2** (1 mmol), alkene **3** (1 mmol), and AIBN (0.1 mmol) were placed under argon. The tube was sealed and heated at 80°C for 6 h. The reaction mixture was washed out using chloroform, and the product was purified by column chromatography on silica gel using *n*-hexane/Et$_2$O (20/1) as an eluent or by recycling preparative high-performance liquid chromatography (HPLC).

*Ethyl 2-methyl-4-(phenyltelluro)decanoate (***4a***).* Obtained as a mixture of ca. 1/1 diastereomers; ^{1}H NMR (270 MHz, CDCl$_3$) δ 0.86–2.17 (m, 21H), 2.75–2.86 (m, 1H), 3.18–3.23 (m, 1H), 4.01–4.15 (m, 2H), 7.17–7.34 (m, 3H); 7.77–7.81 (m, 2H); ^{13}C NMR (68 MHz, CDCl$_3$) δ14.0, 14.1, 16.6, 17.6, 22.5, 28.7, 28.8, 29.1, 29.3, 30.2, 31.1, 31.6, 37.2, 37.9, 39.4, 39.6, 41.2, 41.6, 60.09, 60.13, 110.8, 110.9, 127.85, 127.88, 128.88, 128.91, 140.35, 140.40, 176.1; IR(NaCl) 2957, 2927, 2854, 1732, 1574, 1460, 1434, 1377, 1257, 1179, 1018, 910, 733, 693 cm^{-1}; MS (EI), *m*/*e* (relative intensity, %) 420 (M$^+$, 8), 213 (100), 207 (19), 167 (39), 149 (17) 139 (12), 111 (14), 97 (39), 83 (72). HRMS calcd. for C$_{19}$H$_{30}$O$_2$Te: 420.1308, found: 420.1326.

*Ethyl 4-(phenyltelluro)decanoate (***4b***).* ^{1}H NMR (270 MHz, CDCl$_3$) δ 0.84–0.89 (m, 3H), 1.19–1.74 (m, 13H), 1.89–2.08 (m, 2H), 2.42–2.57 (m, 2H), 3.17–3.27 (m, 1H), 4.07 (q, $J = 7.3$ Hz, 2H), 7.15–7.32 (m, 3H); 7.76–7.78 (m, 2H); ^{13}C NMR (68 MHz, CDCl$_3$) δ14.1, 14.2, 22.6, 28.9, 29.3, 31.7, 32.3, 32.5, 34.3, 37.3, 60.3, 111.1, 128.0, 129.0, 140.3, 173.2; IR (NaCl) 3066, 2955, 2926, 2854, 1736, 1574, 1474, 1374, 1178, 734, 693 cm^{-1}; MS (EI), *m*/*e* (relative intensity, %) 406 (M$^+$, 14), 207 (20), 199 (100), 153 (72), 135 (51) 111 (30) 97 (55). HRMS calcd. for C$_{18}$H$_{28}$O$_2$Te: 406.1151, found: 406.1148.

*2-(1-Ethoxycarbonylethyl)-1-(penyltelluro)norbornane (***4c***).* The ratio of the isomers, (exo, endo) to (exo, exo), was 52/:48. Assignment was based on the chemical shift of exo/endo protons of norbornanes.

(exo, exo) isomer: ^{1}H NMR (270 MHz, CDCl$_3$) δ 1.12–1.64 (m, 12H), 1.84–1.90 (m, 1H), 2.16–2.27 (m, 3H), 3.30 (bs, 1H), 4.12 (q, $J = 7.3$ Hz, 2H), 7.17–7.19 (m, 2H), 7.26–7.32 (m, 1H), 7.78–7.81 (m, 2H); ^{13}C NMR (68 MHz, CDCl$_3$) δ14.2, 15.8, 28.4, 29.5, 35.1, 36.1, 40.7, 43.1, 45.0, 53.3, 60.2, 112.3, 127.8, 129.1, 139.6, 176.0; IR(NaCl) 2954, 2871, 1732, 1574, 1474, 1454, 1434, 1376, 1258, 1177, 1158, 1148, 1053, 1018, 734, 693 cm^{-1}; MS (EI), *m*/*e* (relative intensity, %) 402 (M$^+$, 34), 207 (11), 195 (100), 167 (25), 149 (22), 121 (82), 93 (50). HRMS calcd for C$_{18}$H$_{24}$O$_2$Te: 402.0838, found: 402.0841.

(exo, endo) isomer: ^{1}H NMR (270 MHz, CDCl$_3$) δ 1.09–1.59 (m, 12H), 1.91–1.97 (m, 1H), 2.16–2.30 (m, 3H), 3.51 (bs, 1H), 4.12 (q, $J = 7.3$ Hz, 2H), 7.14–7.20 (m, 2H), 7.24–7.29 (m, 1H), 7.74–7.80 (m, 2H); ^{13}C NMR (68 MHz, CDCl$_3$) δ14.1, 16.2, 28.0, 29.8, 34.9, 38.5, 38.9, 42.8, 43.9, 53.2, 60.6, 112.3, 127.68, 129.0, 139.3, 175.5; IR (NaCl) 2957, 2871, 1728, 1574, 1474, 1455, 1434, 1259, 1177, 1061, 1018, 734, 693 cm^{-1}; MS (EI), *m*/*e* (relative intensity, %) 402 (M$^+$, 39), 207 (18), 195 (100), 167 (27), 149 (22), 121 (90), 93 (55). HRMS calcd. for C$_{18}$H$_{24}$O$_2$Te: 402.0838, found: 402.0839.

*Ethyl 6-Bromo-2-methyl-4-(phenyltelluro)hexanoate (***4d***).* Obtained as a mixture of ca. 1/1 diastereomers; ^{1}H NMR (270 MHz, CDCl$_3$) δ 1.11–1.35 (m, 12H), 1.57–1.68 (m, 2H), 1.95–2.95 (m, 6H), 3.59 (t, $J = 6.8$ Hz, 2H), 3.60 (t, $J = 6.8$ Hz, 2H), 4.05–4.18 (m, 4H), 7.19–7.25 (m, 4H), 7.28–7.37 (m, 2H), 7.71–7.84 (m, 4H); ^{13}C NMR (68 MHz, CDCl$_3$) δ14.23, 16.4, 17.6, 27.6, 28.3, 33.6, 33.8, 39.4, 39.5, 39.7, 40.4, 40.6, 41.3, 60.37, 60.40, 109.6, 109.9, 128.4, 128.5, 129.3, 140.8, 140.9, 175.9, 176.0; IR (NaCl) 3066, 2976, 2034, 1732, 1574, 1474, 1434, 1378, 1258, 1182, 1018, 735, 694 cm^{-1}; MS (EI), *m*/*z* (relative intensity, %) 442 (M$^+$, 42), 397 (11), 363 (20), 235 (98), 207 (69), 155 (93), 127 (100); HRMS calcd. for C$_{15}$H$_{21}$BrO$_2$Te: 441.9788, found: 441.9781.

*Ethyl 6-hydroxy-2-methyl-4-(phenyltelluro)hexanoate (***4e***).* Obtained as a mixture of ca. 1/1 diastereomers; ^{1}H NMR (270 MHz, CDCl$_3$) δ 1.09 (d, $J = 6.8$ Hz, 2H), 1.13 (d, $J = 6.8$ Hz, 3H), 1.17–1.29 (m, 6H), 1.57–1.74 (m, 1H), 1.88–2.17 (m, 1H), 2.48 (s, 1H), 2.60 (s, 1H), 3.99–4.13 (m, 2H), 7.16–7.33 (m, 3H), 7.76–7.80 (m, 2H); ^{13}C NMR (68 MHz, CDCl$_3$) δ14.2, 16.5, 17.7, 25.6, 26.0, 39.5, 39.6, 40.4, 41.2, 41.3, 60.36, 60.40, 62.0, 62.1, 110.8, 111.0, 128.1, 129.1, 140.4, 176.3, 176.5; IR (NaCl) 3435, 3065, 2976, 2935, 1728, 1574, 1473, 1434, 1378, 1300, 1258, 1181, 1083, 1044, 1018, 998, 735, 694 cm^{-1}; MS (EI), *m*/*z* (relative intensity, %) 380 (M$^+$, 20), 207 (27), 173 (100), 155 (38), 143 (24), 127 (50), 99 (57). HRMS calcd. for C$_{15}$H$_{22}$O$_3$Te: 380.0631, found: 380.0628.

*6-(1-Ethoxycarbonylethyl)-4-(phenyltelluro)heptanoate (***4f***).* Obtained as a mixture of ca. 1/1 diastereomers; ^{1}H NMR (270 MHz, CDCl$_3$) δ 1.10–1.27 (m, 6H), 1.56–1.68 (m, 1H), 1.85–2.18 (m, 3H), 2.48–2.72 (m, 2H), 2.78–2.92 (m, 1H), 3.06–3.16 (m, 1H), 4.02–4.16 (m, 2H), 7.18–7.24 (m, 2H), 7.27–7.36 (m, 1H), 7.75–7.81 (m, 2H); ^{13}C NMR (68

MHz, $CDCl_3$) δ14.2, 16.6, 17.6, 29.1, 29.8, 32.0, 32.6, 34.0, 34.1, 39.4, 39.6, 41.0, 41.4, 60.40, 60.43, 110.0, 110.2, 128.35, 128.40, 129.21, 140.7, 176.16, 176.19, 179.16; IR (NaCl) 3066, 2977, 2934, 1732, 1710, 1446, 1434, 1378, 1257, 1181, 1018, 735, 694 cm^{-1}; MS (EI), *m/z* (relative intensity, %) 408 (M^+, 25), 363 (8), 207 (20), 155 (100), 137 (38), 127 (55), 109 (30). HRMS calcd for $C_{16}H_{22}O_4Te$: 408.0580, found: 408.0577.

*2-(4-Hydroxy-2-phenyltelluro)-γ-butyrolactone (***4g***)*. Obtained as a mixture of ca. 1/1 diastereomers; ^1H NMR (270 MHz, $CDCl_3$) δ 1.25 (s, 1H), 1.31 (s, 1H), 1.57–2.28 (m, 12H), 2.35–2.44 (m, 1H), 2.81–2.93 (m, 1H), 3.02–3.15 (m, 1H), 3.20–3.31 (m, 1H), 3.75–3.95 (m, 4H), 4.06–4.21 (m, 2H), 4.27–4.33 (m, 2H), 7.20–7.37 (m, 6H), 7.77–7.81 (m, 4H); ^{13}C NMR (68 MHz, $CDCl_3$) δ24.9, 27.2, 28.7, 29.2, 38.4, 38.5, 39.1, 39.4, 40.26, 40.34, 61.93, 61.96, 66.5, 110.4, 110.7, 128.4, 128.5, 129.3, 129.4, 140.4, 140.5, 179.3, 179.4; IR (NaCl) 3460, 2909, 1766, 1574, 1474, 1434, 1375, 1160, 1023, 736, 695 cm^{-1}; MS (EI), *m/z* (relative intensity, %) 364 (M^+, 46), 287 (10), 207 (28), 157 (48), 139 (100), 127 (78), 95 (70). HRMS calcd for $C_{14}H_{18}O_3Te$: 364.0318, found: 364.0313.

*s-Butyl 4-pentenyl Telluride (***5***)*. ^1H NMR (270 MHz, $CDCl_3$) δ 0.98 (t, $J = 6.8$ Hz, 3H), 1.57–1.91 (m, 7H), 2.14 (q, $J = 7.3$ Hz, 2H), 2.65 (t, $J = 7.3$ Hz, 2H), 3.17 (sextet, $J = 7.3$ Hz, 1H), 4.96–5.08 (m, 2H), 5.72–5.87 (m, 1H); ^{13}C NMR (68 MHz, $CDCl_3$) δ0.6, 14.0, 20.2, 24.5, 31.7, 33.1, 36.1, 115.2, 137.7; IR (NaCl) 3070, 2962, 2926, 2870, 1640, 1452, 1376, 1178, 1142, 1129, 995, 912 cm^{-1}. Anal. calcd. for $C_9H_{18}Te$: C, 42.59; H, 7.15. Found: C, 42.37; H, 7.19.

*2-(2-Methylbutyl)telluracyclopentane (***6***)*. Obtained as a mixture of ca. 1/1 diastereomers; ^1H NMR (270 MHz, $CDCl_3$) δ 0.83–0.90 (m, 6H), 1.02–1.46 (m, 3H), 1.56–1.75 (m, 2H), 1.80–1.97 (m, 2H), 2.21–2.38 (m, 2H), 3.10–3.21 (m, 2H), 4.07–4.16 (m, 1H); ^{13}C NMR (68 MHz, $CDCl_3$) δ6.17, 6.48, 11.0, 11.4, 18.6, 19.4, 28.3, 30.3, 30.7, 31.9, 34.5, 37.1, 38.2, 43.0, 43.5, 46.7, 46.9; IR (NaCl) 2958, 2871, 1460, 1437, 1377, 1245, 1136, 784 cm^{-1}. Anal calcd. for $C_9H_{18}Te$: C, 42.59; H, 7.15. Found: C, 42.55; H, 7.14.

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