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Alkyl hex-1-ynyl tellurides: Syntheses and multinuclear NMR (^{125}Te-, ^{13}C{^{1}H}-, ^{1}H-) studies

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Abstract

Alkyl hex-1-ynyl tellurides, n-$C_4H_9C{\equiv}CTeR$, where R = CH_3, $CH(CH_3)_2$, cyclo-C_6H_{11}, $CH_2CH(CH_3)_2$, $CH_2(CH_2)_6CH_3$, $CH_2(CH_2)_{10}CH_3$, $CH_2CH_2CH(Br)CH_3$, $CH_2C_6H_5$, 4-$CH_2C_6H_4NO_2$, $CH_2CH_2C_6H_5$ and $CH_2CH_2OC_6H_5$, have been prepared in good yields by the anaerobic reaction of lithium hex-1-ynyl tellurolate with the appropriate alkyl halides in the absence of light. While most of these reactions have been performed at − 5 to − 10°C the reaction with cyclo-hexyl halide has been conducted at lower temperature (− 30°C). Alkyl chloro, bromo and iodo derivatives have provided the same products in the same yields. The reaction has produced n-$C_4H_9C{\equiv}CTe(CH_2)_5TeC{\equiv}CC_4H_9$-$n$ when $Br(CH_2)_5Br$ has been used as alkylating agent, while $BrCH_2CH_2CH(Br)CH_3$ has produced only the monotelluride compound n-$C_4H_9C{\equiv}CTeCH_2CH_2CH(Br)CH_3$ even when employing excess lithium hex-1-ynyl tellurolate. In contrast (n-$C_4H_9C{\equiv}C)_2Te$ has been the major tellurium-containing product when $(C_6H_5)_3CCl$, 4-$BrC_6H_4C({=}O)CH_2Br$, $CH_2{=}CHCH_2Br$, $ClCH_2C({=}O)CH_2Cl$ and $ClCH_2C{\equiv}CCH_2Cl$ have been employed. The ^{1}H-NMR spectra of alkyl hex-1-ynyl tellurides display deshielded resonances for the CH_x ($x = 1, 2, 3$) group directly bound to tellurium. In many cases, specific couplings between tellurium and hydrogen are observed around these resonances. Characteristic features in the ^{13}C-NMR spectra include a shielding effect of the sp^3 and sp carbons directly bound to tellurium, and a deshielding effect to the other sp carbon. Closer analysis of the ^{13}C{^{1}H}-NMR spectra reveals satellites due to coupling with tellurium. Nuclear magnetic resonances measurements of the ^{125}Te nucleus show a correlation of ^{125}Te chemical shift to the alkyl group of n-$C_4H_9C{\equiv}CTeR$. In addition, the ^{125}Te-NMR spectra show a splitting of the ^{125}Te nuclear magnetic resonances due to coupling through up to three bonds with ^{1}H nuclei. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

Since the first diorganyl telluride, Et_2Te, was made in 1840, this class of compounds has made important contributions as synthons in organic synthesis [1], in the preparation of new conducting materials [2], as MOCVD precursors to metal telluride films [3], and in medical applications [4]. These applications provide an impetus and incentive for the syntheses and study of the properties of many diaryl tellurides, dialkyl tellurides, alkyl aryl tellurides and tellurovinylorganyl compounds [1–9].

Only limited examples have been reported concerning synthetic efforts to obtain acetylenic tellurides [10–15]. These compounds have been used as a starting material for the synthesis of dialkyl ditellurides, symmetrical and unsymmetrical dialkyl tellurides [16], 1,1,2-trihaloalkenes and dimeric acetylenes [1a]. Also, hydrozirconation of acetylenic tellurides lead to the synthesis of zirconated vinyl tellurides, which produced several types of trisubstituted olefins [17]. Two general approaches are commonly employed for the preparation of acetylenic tellurides, using either electrophilic or nucleophilic tellurium reagents. Alkylation of alkynyl tellurolate anions has been employed for the preparation of unsymmetrical dialkyl tellurides. However, this straightforward synthetic strategy has not been fully explored for the preparation of a wider range of acetylenic tellurides. Although limited data on this class of compounds exist it appears that the stability of alkyl alk-1-ynyl tellurides depends on the nature of the or-

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ganic group linked to tellurium and prolonged exposure to light and air should be avoided to prevent decomposition [16].

Nuclear magnetic resonance chemical shift data of alkyl alk-1-ynyl tellurides are sparse and a systematic study is lacking [5,6,18]. The NMR spectroscopic characteristics of the acetylenic telluride compounds are strongly modulated by the presence of tellurium, the influence of which being negligible beyond three bonds. Values of ^{125}Te resonances have been reported [10] for symmetrical bis(alk-1-ynyl) tellurides in the 321–385 ppm range.

With these perspectives in mind, we report our results on the synthesis of thirteen alkyl hex-1-ynyl tellurides, and discuss their ^{1}H-, ^{13}C- and ^{125}Te-NMR characteristics including a description of coupling constants between tellurium and carbon or proton. These data also provide a valuable tool for assigning substitution patterns to acetylenic tellurides of unknown structure. Discussion on the selective reactivity of the lithium acetylenic tellurolate starting material with various alkyl halides is also provided. A fuller description of the range of accessible compounds should facilitate, and hopefully encourage, further systematic studies of the organic and inorganic chemistry of this class of compounds.

2. Experimental

2.1. General comments

Manipulations were performed in the absence of light and under an inert atmosphere of dry nitrogen or argon using Schlenk line and glove box techniques. Dry, oxygen-free solvents were freshly distilled before utilization. Reactants were purchased from Aldrich. The hex-1-yne was distilled and kept under nitrogen at -15°C. All reagents not packaged under nitrogen were deoxygenated. The products were analyzed by high resolution mass spectra (HP 5971A Mass Selective Detector with HP 5890A Gas Chromatograph, HP-1 column, cross-linked Methyl Siloxane, $25\ \text{m} \times 0.2\ \text{mm} \times 0.33\ \mu\text{m}$, samples were heated from 60–250°C with a rate of 20°C min^{-1}), which featured the same characteristic pattern in all compounds (groups of 9 close peaks with an envelope typical for the 8 stable isotopes of tellurium, a group at 181 m/z due to a $(CH_2)_2C{\equiv}CTe$ fragment, a group at 168 m/z corresponding to a $CH_2C{\equiv}CTe$ fragment, and a group at 81 m/z due to a $CH_3(CH_2)_3C{\equiv}C$ fragment).

The NMR spectra were recorded at room temperature on a Varian VXRS 400 FT-NMR spectrometer. The ^{1}H-, ^{13}C{^{1}H}-, ^{13}C-and ^{125}Te-NMR spectra were measured at a resonance frequency of 400.0, 100.6, 100.6 and 126.24 MHz, respectively. In order to obtain coupling constant values between tellurium and carbon, it was necessary to run ^{13}C-NMR measurements decoupled from ^{1}H nucleus (^{13}C{^{1}H}-NMR) to increase the signal/noise ratio. During ^{125}Te-NMR data acquisition, the flip pulse (PW) and flip angle were 6.0 μs and 60°, respectively, the acquisition time (AT) was 0.16 s and the delay time (D1) between two pulses was 1 s, the gaussian apodization had a time constant of 0.02 s. The ^{125}Te-NMR spectra were all referenced using Te_2Ph_2 (0.5 M in toluene, $\delta(^{125}\text{Te}) = 418$ ppm) as an external reference. Solvents used were d_8-toluene, d_3-acetonitrile or d-chloroform depending on the solubility of the products. Neat samples were hold between two NaCl discs for IR spectroscopic measurements in a Nicolet 5DX interferometer. UV-Vis spectroscopic measurements were run in acetonitrile on a HP 8452A Diode Array Spectrophotometer. The purity of the products was confirmed by gas chromatography with mass spectroscopic detection and ^{1}H-, ^{13}C- and ^{125}Te-NMR spectroscopies.

2.2. Synthesis of lithium hex-1-ynyl tellurolate, *Li[n-$C_4H_9C{\equiv}CTe$], (**Li1**)*

Hex-1-yne (1.15 ml, 10 mmol) in freshly distilled THF (10 ml) was stirred with *n*-butyl lithium (6.25 ml, 1.6 M in hexane, 10 mmol) at -5°C under nitrogen for 20 min. The resulting lithium hex-1-ynyl was canulated to elemental tellurium powder (1.28 g, 10 mmol). The reaction mixture was kept at -5°C and without light, stirred for 2 h, then brought back slowly to room temperature. The solution was then refluxed for 40 min. The reaction mixture containing **Li1** was a yellowish solution with a small amount of black solid. A clear yellow solution was obtained by filtration, which left circa 0.13 g of tellurium on the frit when the reaction conditions were optimal. The solution was then used in subsequent alkylation reactions.

2.3. Preparation of *n*-$C_4H_9C{\equiv}CTeR$ from the reaction of ***Li1*** with organic halides

Reaction of **Li1** with a variety of organic halides employed the same amount of solvent, reaction temperature (-5°C except for reactions with methyl halide (-10°C) and cyclo-hexyl halide (-30°C)) and times. Analyses of **2**–**14** by IR and UV-Vis spectroscopy show the same typical bands: FTIR (cm^{-1}): $\nu(CH_2, CH_3)$ 2983–2821 (s), $\nu(C{\equiv}C)$ ca. 2160 (s), ν(Te–C) 1170–1021(s). UV-vis (nm): 200–300 and 320–420. A typical procedure is provided below for the preparation of *n*-$C_4H_9C{\equiv}CTeCH_3$.

After a solution of **Li1** (10 mmol) was cooled to -10°C, methyl bromide (5 ml, 2 M in diethyl ether, 10 mmol) was added and the reaction mixture was stirred for 15 min in the absence of light. Volatile compounds were evaporated and the residue was diluted in hexane (10 ml). The resulting yellow slurry was filtered through Celite affording a yellow solution. Evaporation of volatiles produced a yellow oil, which should be carefully handled due to its volatility. Further manipulation resulted in the decomposition of the product with formation of black precipitate and the concomitant appearance of a red solution.

2.4. Data for methyl hex-1-ynyl telluride, n-$C_4H_9C{\equiv}CTeCH_3$, (**2**)

GC/MS: *m*/*z* 226; r.t. 7.2 min; yield 76%. ^{1}H-NMR (δ ppm, $CDCl_3$): 0.89 (t, 3H), 1.38 (sextuplet, 2H), 1.47 (quintuplet, 2H), 2.10 (s, 3H), 2.47 (t, 2H). $^{13}C\{^1H\}$-NMR (δ ppm, CD_3CN): -15.75, 13.86, 20.91, 22.47, 31.86, 34.99, 111.46. ^{125}Te-NMR (δ ppm, $CDCl_3$): 162 (quadruplet).

2.5. Data for 2-propyl hex-1-ynyl telluride, n-$C_4H_9C{\equiv}CTeCH(CH_3)_2$, (**3**)

Scale: 2 mmol. Yellow oil (0.43 g, 1.75 mmol, 87% based on Te). GC/MS: *m*/*z* 254; r.t. 8.3 min; yield 100%. ^{1}H-NMR (δ ppm, $CDCl_3$): 0.89 (t, 3H), 1.40 (m, 2H), 1.50 (m, 2H), 1.68 (d, 6H), 2.50 (t, 2H), 3.53 (septet, 1H). $^{13}C\{^1H\}$-NMR (δ ppm, $CDCl_3$): 13.33, 16.45, 20.49, 21.66, 25.28, 30.98, 32.57, 114.29. ^{125}Te-NMR (δ ppm, $CDCl_3$): 483 (septet of doublet).

2.6. Data for cyclo-hexyl hex-1-ynyl telluride, n-$C_4H_9C{\equiv}CTe$-cyclo-C_6H_{11}, (**4**)

Scale: 2 mmol. Orange oil (0.54 g, 1.84 mmol, 92% based on Te). GC/MS: *m*/*z* 292; r.t. 11.5 min; yield 100%. ^{1}H-NMR (δ ppm, $CDCl_3$): 0.83 (t, 3H), 1.28 (m, 3H axial on the cyclohexyl ring), 1.33 (m, 2H), 1.43 (quintuplet, 2H), 1.57 (m, 3H equatorial on the cyclohexyl ring), 1.71 (quartet, 2H axial on the cyclohexyl ring), 2.09 (m, 2H equatorial on the cyclohexyl ring and 1H on the cyclohexyl ring in α position to tellurium), 2.42 (t, 2H). $^{13}C\{^1H\}$-NMR (δ ppm, $CDCl_3$): 13.38, 20.53, 21.66, 25.49 (2C from the cyclohexyl ring), 27.80 (1C from the cyclohexyl ring), 28.00 (1C from the cyclohexyl ring), 31.01, 32.27, 35.88 (2C from the cyclohexyl ring), 113.61. ^{125}Te-NMR (δ ppm, $CDCl_3$): 443 (broad resonance at circa 19°C, sharp resonance at 50°C).

2.7. Data for 2-methylpropyl hex-1-ynyl telluride, n-$C_4H_9C{\equiv}CTeCH_2CH(CH_3)_2$, (**5**)

Scale: 1 mmol. Pale yellow waxy solid (1.99 g, 0.75 mmol, 75% based on Te). GC/MS: *m*/*z* 268; r.t. 9.7 min; yield 100%. ^{1}H-NMR (δ ppm, $CDCl_3$): 0.89 (t, 3H), 1.02 (d, 6H), 1.40 (sextuplet, 2H), 1.48 (quintuplet, 2H), 1.96 (septet, 1H), 2.46 (t, 2H), 2.76 (d, 2H). $^{13}C\{^1H\}$-NMR (δ ppm, $CDCl_3$): 13.43, 20.43, 20.85, 21.64, 23.40, 29.69, 31.18, 33.53, 110.20. ^{125}Te-NMR (δ ppm, $CDCl_3$): 207 (doublet of triplet).

2.8. Data for 1-octyl hex-1-ynyl telluride, n-$C_4H_9C{\equiv}CTe(CH_2)_7CH_3$, (**6**)

Scale: 2 mmol. Yellow oil. GC/MS: *m*/*z* 324; r.t. 11.8 min; yield 79%. ^{1}H-NMR (δ ppm, $CDCl_3$): 0.72 (t, 3H), 0.74 (t, 3H), 1.12 (broad singlet, 10H), 1.24 (m, 2H), 1.33 (m, 2H), 1.70 (m, 2H), 2.32 (t, 2H), 2.59 (t, 2H). $^{13}C\{^1H\}$-NMR (δ ppm, $CDCl_3$): 8.61, 13.10, 13.59, 20.21, 21.42, 22.21, 28.44, 28.73, 30.74, 31.13, 31.39, 32.25, 33.26, 111.74. ^{125}Te-NMR (δ ppm, $CDCl_3$): 273 (triplet of triplet).

2.9. Data for 1-dodecyl hex-1-ynyl telluride, n-$C_4H_9C{\equiv}CTe(CH_2)_{11}CH_3$, (**7**)

Scale: 2 mmol. Yellow oil. GC/MS: *m*/*z* 380; r.t. 13.8 min; yield 87%. ^{1}H-NMR (δ ppm, $CDCl_3$): 0.85 (t, 3H), 0.88 (t, 3H), 1.23 (broad singlet, 18H), 1.38 (m, 2H), 1.48 (m, 2H), 1.85 (quintuplet, 2H), 2.47 (t, 2H), 2.74 (t, 2H). $^{13}C\{^1H\}$-NMR (δ ppm, $CDCl_3$): 9.13, 13.53, 14.06, 20.64, 21.86, 22.65, 28.89, 29.28, 29.48, 29.62, 31.11, 31.50, 31.57, 31.88, 33.95, 112.56. ^{125}Te-NMR (δ ppm, $CDCl_3$): 268 (triplet of triplet).

2.10. Data for 1-(3-bromo)butyl hex-1-ynyl telluride, n-$C_4H_9C{\equiv}CTe(CH_2)_2CH(Br)CH_3$, (**8**)

Scale: 2 mmol. Waxy orange solid. GC/MS: *m*/*z* 346; r.t. 11.1 min; yield 100%. ^{1}H-NMR (δ ppm, $CDCl_3$): 0.87 (t, 3H), 1.39 (m, 2H), 1.47 (m, 2H), 1.71 (d, 3H), 2.29 (m, 2H), 2.46 (t, 2H), 2.72 (m, 1H), 2.94 (m, 1H), 4.21 (m, 1H). The ^{1}H-NMR spectrum revealed non-equivalent protons of the CH_2 group in α-position (2 multiplets at 2.74 and 2.95 ppm). $^{13}C\{^1H\}$-NMR (δ ppm, $C_6D_5CD_3$): 6.27, 13.85, 20.99, 22.31, 26.12, 31.71, 33.74, 43.48, 53.08, 112.60. ^{125}Te-NMR (δ ppm, $CDCl_3$): 283 (triplet of doublet).

2.11. Data for α-(hex-1-ynyl telluride) toluene, n-$C_4H_9C{\equiv}CTeCH_2C_6H_5$, (**9**)

Scale: 2 mmol. Dark yellow oil. GC/MS: *m*/*z* 302; r.t. 11.5 min; yield 100%. ^{1}H-NMR (δ ppm, $CDCl_3$): 0.88 (t, 3H), 1.37 (m, 2H), 1.46 (m, 2H), 2.48 (t, 2H), 4.15 (s, 2H), 7.12 to 7.25 (m, 5H). $^{13}C\{^1H\}$-NMR (δ ppm, $CDCl_3$): 12.13, 13.55, 20.63, 21.84, 31.00, 33.89, 114.96, 126.54, 128.40, 139.76. ^{125}Te-NMR (δ ppm, $CDCl_3$): 456 (triplet).

2.12. Data for α-(hex-1-ynyl-telluride)-4-nitrotoluene, $n\text{-}C_4H_9C{\equiv}CTeCH_2C_6H_4NO_2\text{-}4$, (**10**)

Scale: 1 mmol. Orange waxy solid. GC/MS: *m/z* 341, r.t. 8.7 min; yield 5%. The GC/MS yields were very low due to the fact that the product did not properly transverse the column. However, ^{125}Te-NMR measurement revealed that **10** was the tellurium-containing product. ^{1}H-NMR (δ ppm, $CDCl_3$): 0.90 (t, 3H), 1.38 (m, 2H), 1.47 (m, 2H), 2.51 (t, 2H), 4.18 (s, 2H), 7.34, 7.45, 8.11. $^{13}C\{^1H\}$-NMR (δ ppm, $CDCl_3$): 10.44, 13.15, 20.29, 21.51, 30.70, 33.67, 115.81, 123.19, 128.80, 146.12, 148.58. ^{13}C-NMR (δ ppm, $CDCl_3$): 10.55 (triplet of triplet), 13.18 (quartet of triplet of triplet), 20.26 (triplet of triplet), 21.42 (triplet of sextuplet), 30.63 (triplet of quintuplet), 33.87 (triplet of triplet), 115.67 (triplet of triplet), 123.19, 128.80, 146.12, 148.58. ^{125}Te-NMR (δ ppm, $CDCl_3$): 532 (triplet).

2.13. Data for phenethyl hex-1-ynyl telluride, $n\text{-}C_4H_9C{\equiv}CTe(CH_2)_2C_6H_5$, (**11**)

Scale: 1 mmol. Yellow oil (0.23 g, 0.74 mmol, 74% based on Te). GC/MS: *m/z* 316; r.t. 12.3 min; yield 100%. ^{1}H-NMR (δ ppm, $CDCl_3$): 0.91 (t, 3H), 1.43 (sextuplet, 2H), 1.51 (septet, 2H), 2.50 (t, 2H), 2.96 (t, 2H), 3.17 (t, 2H), 7.20–7.28 (m, aromatic ring, 5H). $^{13}C\{^1H\}$-NMR (δ ppm, $CDCl_3$): 9.40, 13.48, 20.59, 21.80, 31.06, 31.50, 37.78, 113.07, 126.70, 128.42, 142.21. ^{13}C-NMR (δ ppm, $CDCl_3$): 9.51 (triplet), 13.53 (quartet of triplet), 20.59 (triplet of multiplet), 21.83 (triplet of sextuplet), 31.01 (triplet of sextuplet), 31.54 (triplet of triplet), 38.40 (quartet), 113.07 (triplet of triplet), 126.70, 128.42, 142.21. ^{125}Te-NMR (δ ppm, $CDCl_3$): 292 (triplet of triplet).

2.14. Data for 2-(hex-1-ynyl telluride) ethyl phenyl ether, $n\text{-}C_4H_9C{\equiv}CTe(CH_2)_2OC_6H_5$, (**12**)

Scale: 1 mmol. Orange oil (0.26 g, 0.80 mmol, 80% based on Te). GC/MS: *m/z* 332; r.t. 12.3 min; yield 94%. ^{1}H-NMR (δ ppm, $CDCl_3$): 0.89 (t, 3H), 1.41 (m, 2H), 1.48 (m, 2H), 2.48 (t, 2H), 3.09 (t, 2H), 4.37 (t, 2H), 6.88 (m, 3H), 7.27 (m, 2H). $^{13}C\{^1H\}$-NMR (δ ppm, $CDCl_3$): 7.15, 13.46, 20.56, 21.82, 30.75, 30.98, 68.91, 113.16, 114.67, 120.98, 129.38, 158.16. ^{13}C-NMR (δ ppm, $CDCl_3$): 10.55 (triplet of triplet), 13.18 (quartet of triplet), 20.26 (triplet of triplet), 21.55 (triplet of multiplet), 30.63 (triplet of quintuplet), 33.87 (triplet of triplet), 68.91 (triplet of triplet), 114.67, 115.67 (triplet of triplet), 120.98, 129.38, 148.69 (triplet of doublet). ^{125}Te-NMR (δ ppm, $CDCl_3$): 246 (triplet).

2.15. Data for 1,5-di(hex-1-ynyl telluride) pentane, $n\text{-}C_4H_9C{\equiv}CTe(CH_2)_5TeC{\equiv}CC_4H_9$, (**13**)

Scale: 2 mmol. Yellow solid. GC/MS: the product did not transverse properly through the GC/MS column due to its high molecular weight and the mass spectra provided only the first fragment of the compound, *m/z* 360, which corresponds to the product after the loss of one tellurium atom; r.t. 11.7 min; yield 45%. The reaction was redone with a **Li1**:1,5-dibromopentane ratio of 2:1 at −78°C but a mixture of tellurium-containing products was obtained. ^{1}H-NMR (δ ppm, $C_6D_5CD_3$): 0.79 (t, 6H), 1.11 (m, 4H), 1.36 (m, 8H), 1.57 (quintuplet, 2H), 2.32 (m, 8H). $^{13}C\{^1H\}$-NMR (δ ppm, $C_6D_5CD_3$): 8.13, 13.90, 21.11, 22.35, 30.45, 31.28, 31.73, 33.28, 112.63. ^{13}C-NMR (δ ppm, $C_6D_5CD_3$): 8.17 (triplet of triplet), 13.94 (quartet of triplet), 21.11, 22.35, 30.45, 31.28, 31.73, 33.28 (triplet of triplet), 112.63 (triplet of triplet). ^{125}Te-NMR (δ ppm, $C_6D_5CD_3$): 288 (triplet of triplet).

2.16. Preparation of $(n\text{-}C_4H_9C{\equiv}C)_2Te$ (**14**) from the reaction of **Li1** with unsaturated alkyl halides

The unsaturated alkyl halides include: trityl chloride; 4′-bromophenacyl bromide; 3-bromopropene; 1,3-dichloroacetone; and 1,4-dichlorobut-2-yne. These reactions provided dark red solutions containing a large amount of black precipitate and after work-up yellow oils of **14** were obtained. The reaction of **Li1** and 1,3-dichloroacetone is provided as an example.

Compound **Li1** (2 mmol) in THF (10 ml) was added at ambient temperature to 1,3-dichloroacetone (0.254 g, 2 mmol) in THF (10 ml). The reaction mixture was stirred overnight then filtered through Celite. The filtrate was a dark red solution with a large quantity of black precipitate. After evaporation of the volatile components, the product was diluted in hexane (10 ml) and filtered until black solid did not precipitate further. The black solids were collected and combined to give a total of 0.14 g of tellurium (1.1 mmol, circa 55% of the original amount). After evaporation of the solvent, the product was a yellow oil. Isolated yield: 0.17 g, 0.6 mmol, 30% based on Te.

2.17. Data for bis(hex-1-ynyl) telluride, $(n\text{-}C_4H_9C{\equiv}C)_2Te$, (**14**)

GC/MS: *m/z* 292; r.t. 10.7 min; yield 98%. ^{1}H-NMR (δ ppm, $C_6D_5CD_3$): 0.74 (t, 6H), 1.25 (m, 8H), 2.20 (t, 4H). $^{13}C\{^1H\}$-NMR (δ ppm, $C_6D_5CD_3$): 13.72, 20.89, 22.26, 31.24, 32.26, 113.87. ^{125}Te-NMR (δ ppm, $C_6D_5CD_3$): 338.

3. Results and discussion

*3.1. Reaction of lithium hex-1-ynyl tellurolate (**Li1**) with alkyl monohalides.*

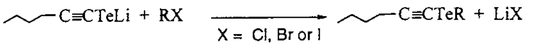
Lithium hex-1-ynyl, produced via hydrogen-lithium exchange between hex-1-yne and *n*-butyl lithium, undergoes [19] insertion of elemental tellurium at temperatures (−5°C) compatible with the stability of the organolithium compound, to produce **Li1** (Eq. (1)). Pale yellow reaction mixtures are routinely obtained that contain a small amount of unreacted tellurium metal. Decomposition to bis(hex-1-ynyl) telluride occurs during attempts to isolate **Li1** and, therefore, the reaction mixture is used in situ for further procedures. Regardless, high conversion to **Li1** is demonstrated by the good yields of products in subsequent alkylation reactions.

Li1

(1)

Li1 reacts with a variety of primary or secondary open chain saturated alkyl halides such as saturated alkyl halides, as well as with α-halotoluene derivatives, and with phenethyl halides. The reaction of $\mathbf{1}^-$ with alkyl halides is independent of the halide, since chloro, bromo and iodo reagents give the same products in the same yields. Alkyl hex-1-ynyl tellurides are yellow or orange products and can be very volatile oils (e.g. **2**) or crystalline materials (e.g. **4**). To our knowledge [22] most of the alkyl hex-1-ynyl tellurides presented here have not been synthesized before; **2** and **14** were reported [15,23].

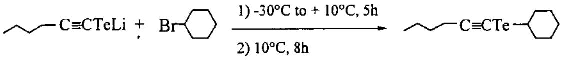
The synthesis of alkyl hex-1-ynyl tellurides is carried out by alkylation [20] of **Li1** at low temperature with increase of the reaction mixture temperature to 20°C after 20 min (Eq. (2)).



2: R = CH_3
3: R = $CH(CH_3)_2$
4: R = *cyclo*-C_6H_{11}
5: R = $CH_2CH(CH_3)_2$
6: R = $(CH_2)_7CH_3$
7: R = $(CH_2)_{11}CH_3$
8: R = $(CH_2)_2CH(Br)CH_3$
9: R = $CH_2C_6H_5$
10: R = $CH_2C_6H_5$-*p*-NO_2
11: R = $(CH_2)_2C_6H_5$
12: R = $(CH_2)_2OC_6H_5$
13: R = $(CH_2)_5TeC{\equiv}C(CH_2)_3CH_3$

(2)

However this procedure affords black slurries on treating **Li1** with cyclo-hexyl halides. Compound **4** is prepared when the reaction mixture is cooled to −30°C and very slowly warmed (Eq. (3)). Analysis of the reaction mixture by ^{125}Te-NMR spectroscopy reveals a broad resonance at 443 ppm at 20°C, while the ^{125}Te resonance is sharp at 50°C, which is consistent with rapid inversion of the cyclo-hexyl ring conformation. Furthermore ^{1}H- and ^{13}C-NMR as well as two-dimensional COSY and HETCOR NMR spectroscopic analyses confirm the existence of **4**.



4

(3)

In addition, two dihaloalkanes also were examined. Reactions of **Li1** with 1,3-dibromobutane in a 2:1 ratio at various temperatures generate only 1-(3-bromo)butyl hex-1-ynyl telluride (**8**), demonstrating that $\mathbf{1}^-$ has substituted only the less hindered bromide. A similar selectivity has also been reported in the formation of 3-bromobutyl phenyl telluride [20] from 1,3-dibromobutane. Reaction of **Li1** with 1,5-dibromopentane in 1:1 stoichiometry generates 1,5-di(hex-1-ynyl telluride) pentane (**13**). Interestingly, when the reaction is performed with a 2:1 ratio of **Li1**:1,5-dibromopentane a mixture of tellurium-containing products is obtained.

During efforts to optimize the synthetic procedure we have found that the exclusion of light and increasing the reaction time produce clean reaction mixtures as evidenced by GC/MS and NMR spectroscopies with isolated yields superior to 70%. As such this represents a simple and efficient improvement over methods described in the literature [20]. In most cases, after evaporation of the volatiles, hexane is added to the reaction mixture and filtration through Celite separates unreacted tellurium metal and lithium halide salt from the product. By avoiding chromatographic work-up this procedure reduces the amount of organic solvent used. Further purification steps have resulted in the decomposition of the telluride with the concomitant formation of a black precipitate of finely divided elemental tellurium even under anaerobic conditions. Alkyl hex-1-ynyl tellurides compounds tend to slowly decompose upon exposure to light and air with the loss of elemental tellurium. This agrees with previously reported extrusion of tellurium from symmetrical acetylenic tellurides [13]. Care with respect to the stoichiometry of this reaction is crucial in order to avoid the formation of **14** and dialkyl telluride. The formation of **14** as a by-product correlates well with the reported [21] formation of symmetrical tellurides as by-products during diaryl telluride synthesis. Prolonged exposure of **14** to light results in the formation of a black precipitate, which indicates a decomposition process.

*3.2. Reaction of **Li1** with unsaturated alkyl halides*

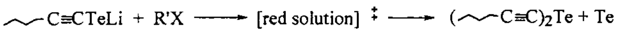
Several attempts have been made to alkylate $\mathbf{1}^-$ with trityl chloride, 4′-bromophenacyl bromide, 3-bromopropene, 1,3-dichloroacetone and 1,4-dichlorobut-2-

Table 1
^{1}H-NMR data for α-sp^3 and β-sp^3 CH$_x$ of products from n-$C_4H_9C{\equiv}CTeLi+RX$ [a,b]

Products	**2** [c,e]	**3** [c,e]	**4** [c,e]	**5** [c,e]	**6** [c,e]	**7** [c,e]
α-CH$_x$	2.10	3.53	2.09	2.76	2.59	2.74
β-CH$_x$		1.68	2.09 [g], 1.71 [h]	1.96	1.70	1.85
$J^2_{Te\text{-}H}$	25			34	34	34
$J^3_{Te\text{–}H}$		29				
Products	**8** [c,f]	**9** [c,e]	**10** [d,e]	**11** [c,e]	**12** [c,e]	**13** [d,f]
α-CH$_x$	2.94, 2.72	4.15	4.18	2.96	3.09	2.32
β-CH$_x$	2.29			3.17	4.37	1.11
$J^2_{Te\text{–}H}$		31	37			

[a] Chemical shifts in ppm, coupling constants in Hz.
[b] $R = n$-C_4H_9, X = Cl, Br, I, $x = 1$, 2 or 3.
[c] Isolated product.
[d] Non-isolated product.
[e] ^{1}H-NMR spectra referenced on $CDCl_3$.
[f] ^{1}H-NMR spectra referenced on $C_6D_5CD_3$.
[g] Equatorial protons.
[h] Axial protons.

yne. These reactions typically provide dark red solutions containing a large quantity of black precipitate (Eq. (4)). After several days under anaerobic conditions and filtration through Celite, the red solutions cleanly provide a yellow oil where **14** is the only tellurium-containing product except in the case of 3-bromopropene. In this latter case, ^{125}Te-NMR spectroscopy reveals the presence of several tellurium-containing products. Lower reaction temperatures do not improve the outcome of this reaction. It remains unclear why these particular alkyl halides do not provide the expected alkyl hex-1-ynyl tellurides.



R' = $CH_2C(=O)CH_2Cl$, $C(C_6H_5)_3$, $CH_2C(=O)C_6H_5Br$-p, $CH_2C{\equiv}CCH_2Cl$, $CH_2CH{=}CH_2$. (4)

The formation of red solutions, which then decompose into **14** by loss of elemental tellurium, may be consistent with the initial formation of a di(hex-1-nyl) polytelluride compound by a radical mechanism [24]. These red compounds exhibit ^{1}H- and ^{13}C-NMR resonances characteristic of a symmetric hex-1-ynyl moiety bound to tellurium. Unfortunately, their instability prevents their isolation and characterization.

3.3. Characteristic features of ^{1}H-NMR spectral alkyl hex-1-ynyl tellurides

Resonances for the hex-1-ynyl moiety are observed at circa 0.85, 1.37, 1.45 and 2.43 ppm. In the alkyl chain hydrogens of α-sp^3 CH$_x$ ($x = 1$, 2, 3) are deshielded by the tellurium atom, which has an electronegativity comparable to that of iodine, but slightly shifted upfield relative to the parent iodoalkane. They resonate between 2.32 and 2.96 ppm for a α-CH$_2$ group if the β-carbon is not aromatic and carries no heteroatom such as oxygen or bromine. If the β-carbon is aromatic then they resonate around 4.16 ppm. The resonance occurs at 2.10 ppm for a α-CH$_3$ group, and at 3.53 ppm for a α-CH group.

In addition, the proximity of tellurium, which has an $I = 1/2$ and a natural abundance of circa 7%, gives rise to ^{125}Te-^{1}H coupling through two bonds that is observed in the ^{1}H-NMR spectra of compounds **2**, **5**, **6**, **7**, **9** and **10** (Table 1). Satellites due to this coupling are observed only around the resonances of hydrogens of α-sp^3 CH$_x$ ($x = 2$, 3), except in the case of compound **3**, where the coupling constant through three bonds is clearly observed around β-sp^3 CH$_3$ (Fig. 1). For the other compounds in this report ^{1}H-^{1}H coupling obstructs ^{125}Te-^{1}H coupling. Coupling features in the spectra between ^{125}Te and ^{1}H facilitate assignment of individual signals.

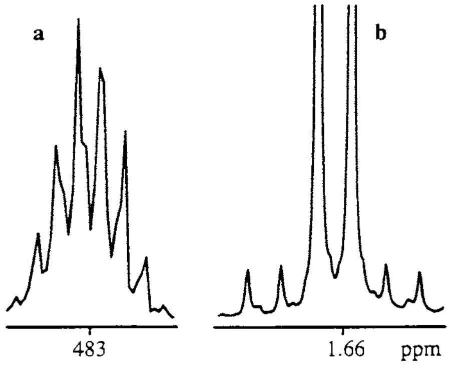


Fig. 1. Te–H couplings in (a) ^{125}Te-NMR spectrum of n-$C_4H_9C{\equiv}CTeCH(CH_3)_2$ and (b) the methyl region of ^{1}H spectrum of n-$C_4H_9C{\equiv}CTeCH(CH_3)_2$.

Table 2
$^{13}C\{^{1}H\}$-NMR and ^{13}C-NMR data for α-sp^3 and β-sp^3 carbon and the 2 sp carbons of products from n-$C_4H_9C{\equiv}CTeLi+RX$ [a,b]

Products	**2** [c,e]	**3** [c,e]	**4** [c,e]	**5** [c,e]	**6** [c,e]	**7** [c,e]	**8** [c,f]
α-sp^3 C	−15.75	16.45	27.80	20.58	8.61	9.13	6.27
α-sp C	34.99	32.57	32.27	33.53	32.25	33.95	33.74
β-sp C	111.46	114.29	113.61	110.20	111.74	112.56	112.60
$J^{1}_{Te-C(\alpha-sp^3)}$	156	128	136		140		164
$J^{1}_{Te-C(\alpha-sp)}$		532	531	528			528
$J^{1}_{Te-C(\beta-sp)}$ [g]	116	116	115	116	114		114

Products	**9** [c,f]	**10** [d,e]	**11** [c,e]	**12** [c,e]	**13** [d,f]	**14** [c,e]
α-sp^3 C	12.13	10.44	9.40	7.15	8.13	
α-sp C	33.89	33.67	31.50	30.75	33.28	32.44
β-sp C	114.91	115.81	113.07	113.16	112.63	113.88
$J^{1}_{Te-C(\alpha-sp^3)}$	152	142	146	154		
$J^{1}_{Te-C(\alpha-sp)}$						536
$J^{1}_{Te-C(\beta-sp)}$			114			111
$J^{3}_{C(\alpha-sp)-H}$		5	5	4	5	
$J^{2}_{C(\beta-sp)-H}$		10	9	8	10	
$J^{4}_{C-H(\beta-sp)}$		6	5	4	5	
J^{1}_{C-H}, J^{2}_{C-H} [g]		145, 4	138	144, 5	141, 4	
J^{1}_{C-H}, J^{2}_{C-H} [h]			128	146, 4		

[a] Chemical shifts in ppm, coupling constants in Hz.

[b] $R = n$-C_4H_9, X = Cl, Br, I.

[c] Isolated product.

[d] Non-isolated product.

[e] $^{13}C\{^{1}H\}$-NMR spectra referenced on $CDCl_3$.

[f] $^{13}C\{^{1}H\}$-NMR spectra referenced on $C_6D_5CD_3$.

[g] Coupling constant J^{1}_{C-H} and J^{2}_{C-H} for α-CH_x groups ($x = 1$, 2 or 3).

[h] Coupling constants J^{1}_{C-H} and J^{2}_{C-H} for β-CH_x groups ($x = 1$, 2 or 3).

3.4. *Characteristic features of ^{13}C-NMR spectra of alkyl hex-1-ynyl tellurides (Table 2)*

The ^{13}C-NMR resonances are significantly influenced by tellurium for both sp C and the α-sp^3 C. Resonances for the hex-1-ynyl group occur at approximately 13, 22, 31, 20, 113, and 33 ppm, respectively, from the CH_3 group to the α-sp C. The very strong shielding α-effect exhibited by the tellurium atom is due to the electronegativity of tellurium and to non-bonded interactions of the substituent with unshared electron pairs of the heteroatom [25]. The alk-1-ynyl moieties of compounds **2** to **14** exhibit ^{13}C-NMR resonances characteristic of sp carbon bonded to tellurium, the β-C shift falls in the 110–119 ppm range, significantly deshielded compared to the α-C chemical shift (30 to 34 ppm). The alkyl chain bound to tellurium has very little influence on the chemical shifts of both sp carbons as evidenced by their constant values throughout the series of compounds. The electron donating effects of the alkyl radicals are transmitted little through the heteroatoms and the triple bond. The alkyl radicals display typical high field chemical shifts (between −16 and 28 ppm) for the α-sp^3 C (Fig. 2).

The assignments of α-sp carbon, β-sp carbon and α-sp^3 carbon are made by means of ^{125}Te-^{13}C spin-spin coupling constants, which depend on carbon hybridization [18]. For example, Fig. 3 contains the satellites in the case of $^{13}C\{^{1}H\}$-NMR spectra of **8**. There are remarkably little data on these parameters in the literature [18]. The J^{1}_{Te-C} coupling constants of the α-sp^3 C, in the 128–156 Hz range, are similar to those reported [26] for $MeTe(CH_2)_nTeMe$ (150–180 Hz) and $RTeCH_2TeR$ (205–210 Hz). The β-sp C resonances

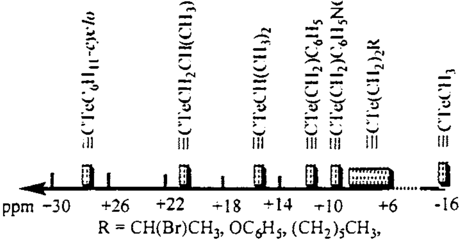


Fig. 2. Correlation of α-sp^3 ^{13}C chemical shift with alkyl substituent of n-$C_4H_9C{\equiv}CTeR$.

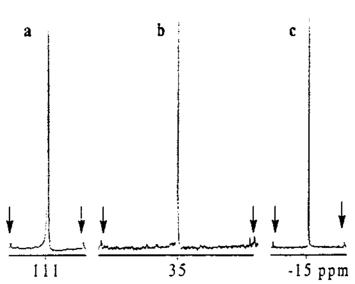


Fig. 3. Te-C couplings in $^{13}C\{^1H\}$-NMR spectrum of *n*-$C_4H_9C{\equiv}CTe(CH_2)_2CH(Br)CH_3$: (a) J^2_{Te-C} on β-sp carbon; (b) J^1_{Te-C} on α-sp carbon; and (c) J^1_{Te-C} on α-sp^3 carbon.

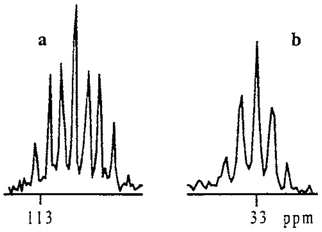


Fig. 4. Long range C-H couplings in ^{13}C-NMR spectrum of *n*-$C_4H_9C{\equiv}CTeCH_2C_6H_4$-p-$NO_2$: (a) for β-sp carbon; (b) for α-sp carbon.

Table 3
^{125}Te-NMR data for alkyl hex-1-ynyl tellurides [a]

Products	**2** [c,e]	**3** [c,e]	**4** [c,e]	**5** [c,e]	**6** [c,e]	**7** [c,e]	**8** [c,f]
$^{125}Te(\delta)$ [b]	162	483	443	207	273	268	283
J^2_{Te-H}	25	37		34	34	34	31
J^3_{Te-H}		29		18	18	18	12
Products	**9** [c,e]	**10** [d,e]	**11** [c,e]	**12** [c,e]	**13** [d,f]	**14** [c,e]	
$^{125}Te(\delta)$ [b]	456	532	292	246	288	332	
J^2_{Te-H}	31	37	34	34	34		
J^3_{Te-H}			18		18		

[a] Chemical shifts in ppm, coupling constants in Hz.
[b] Referenced on Te_2Ph_2, 0.5 M in d_8-toluene at 418 ppm.
[c] Isolated product.
[d] Non-isolated product.
[e] ^{125}Te-NMR measurements run into $CDCl_3$.
[f] ^{125}Te-NMR measurements run into $C_6D_5CD_3$.

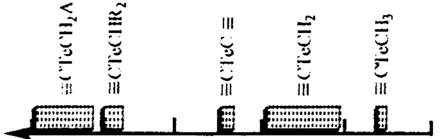


Fig. 5. Correlation of ^{125}Te chemical shift with alkyl substituent of *n*-$C_4H_9C{\equiv}CTeR$.

feature distinct ^{125}Te satellites ($J^2_{Te-C} = 111-116$ Hz). Closer analysis reveals that the α-sp C resonances at 33 ppm exhibit ^{125}Te satellites as well with significantly larger J^1_{Te-C} coupling constants ($J^1_{Te-C} = 536$ Hz), as expected for sp carbons with a large s character. For comparison [10], symmetrical bis(alk-1-ynyl) tellurides display J^1_{Te-C} coupling constants of 538–556 Hz and J^2_{Te-C} of 111–116 Hz.

Long range (four-bond) spin-spin C–H coupling through the tellurium center is evidenced by splitting in ^1H-coupled ^{13}C-NMR spectra (Fig. 4). The α-sp C typically exhibits a triplet of triplet appearing as a five line pattern ($J^3_{C-H} = 5$ and 6 Hz) and the β-sp C a triplet of triplet appearing as a seven line pattern ($J^2_{C-H} = 10$ Hz, $J^4_{C-H} = 4$ Hz). The J^1_{C-H} (ca. 140 Hz) and J^2_{C-H} (ca. 4 Hz) coupling constants for α-sp^3 CH_x ($x = 1, 2, 3$) are consistent with general [27] ranges of 120–160 Hz reported for J^1_{C-H} and 5 Hz for J^2_{C-H}.

3.5. Characteristic features of ^{125}Te-NMR spectra of alkyl hex-1-ynyl tellurides (Table 3)

The chemical shift of tellurium depends not only on the deuterated solvent used but as well on the concentration of the product. However, these concentration effects cause few difficulties, and in this study we have found the ^{125}Te shifts to be the most valuable probe for identifying constituents of mixtures. Fig. 5 illustrates the correlation of ^{125}Te-NMR data to the structure of the tellurides. Methyl hex-1-ynyl telluride (**2**), with tellurium bound to a sp^3 CH_3 group, exhibits a ^{125}Te-NMR shift at 162 ppm. Alkyl hex-1-ynyl tellurides with tellurium bound to a sp^3 CH_2 group without an aromatic ring immediately bound (**5**, **6**, **7**, **8**, **11**, **12** and **13**) feature ^{125}Te-NMR shifts between 200 and 300 ppm. In contrast, if tellurium is bonded to a sp^3 CH_2 with a bound aromatic ring (**9**, **10**), the ^{125}Te-NMR shift is in the 456–532 ppm range depending on the substituents on the aromatic ring. Compounds with tellurium bound to a sp^3 CH group (**3**, **4**) display ^{125}Te-NMR shifts around 480 ppm. If tellurium is bonded to two sp carbon atoms (**14**) its chemical shift is a singlet at 331 ppm, which on several ^{125}Te-NMR spectra evidences the formation of bis(hex-1-ynyl) telluride **14** as a by-product. Several bis(alk-1-ynyl) tellurides [10] have been reported with a ^{125}Te chemical shift between 320–385 ppm. As expected, substitutions more remote than the γ- carbon have negligible effects on the ^{125}Te chemical shift.

There is a marked difference in the J^2_{Te-H} coupling constant between **2**, **3**, **9** and **10** where its value is 25, 37, 31 and 37 Hz, respectively, and the other alkyl hex-1-ynyl tellurides, which display circa 34 ± 1 Hz value. For comparison [10], a 26 Hz value has been reported for $CH_3TeC{\equiv}CTeCH_3$. This difference correlates with the electronic effect of the hydrogens carried by the α-group. The J^2_{Te-H} coupling constant is smaller than 34 Hz if the inductive donor influence on the α-group is lower than the inductive effect of a linear alkyl chain, and vice-versa. For example a phenyl ring (compound **9**) has a lower inductive donor effect to a bound CH_2 group than an alkyl chain, except if it is carrying a strong electron donor para-substituent, such as NO_2 (compound **10**). In contrast, the opposite is observed for the $J^3_{\mathrm{Te-H}}$ coupling constant with respect to the β-carbon, which has an average value of 18 Hz. Symmetrical bis(alk-1-ynyl) tellurides present [10] long-range spin–spin coupling ($J^4_{\mathrm{Te-H}} = 5$ Hz).

Supporting information available. Contains ^{1}H-, ^{13}C- and ^{125}Te-NMR spectra for compounds **2** to **14** [28].

Acknowledgements

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