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Article

Stereoselective Synthesis of (*E*)-*α*-Aryltellurenylvinylstannanes and Their Application in the Synthesis of Stereodefined Trisubstituted Alkenes

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(*E*)-*α*-Aryltellurenylvinylstannanes have been synthesized stereoselectively via the hydrozirconation of alkynylstannanes, followed by the reactions with aryltellurenyl iodides. (*E*)-*α*-Aryltellurenylvinylstannanes can undergo sequential cross coupling reactions with both electrophiles and nucleophiles in the presence of transition metal complexes to form two carbon-carbon bonds in the same olefinic carbon leading to trisubstituted alkenes stereoselectively.

Keywords hydrozirconation, alkynylstannane, (*E*)-*α*-aryltellurenylvinylstannane, trisubstituted alkene, stereoselective synthesis

Introduction

The methodologies for the stereocontrolled synthesis of trisubstituted alkenes are of great interest in organic chemistry since many biologically active compounds occurring in nature possess the structural skeleton of trisubstituted alkenes.[1-4] Difunctional group reagents, which have two different functional groups linked to the olefinic carbon atoms, for example, Sn-Si,[5] Sn-Se,[6] Sn-Zr,[7] Te-Zr,[8] and Te-Br[9] combinations, play an important role in organic synthesis, especially in developing many convenient methods for the stereoselective synthesis of substituted alkenes.

Vinyl tellurides are important intermediates because the tellurium moiety can be replaced by a variety of organic groups always with total retention of the configuration.[10] Vinylstannanes are also important synthetic intermediates and have numerous synthetic utilities in organic synthesis owing to the versatile reactivity of the stannyl group and the carbon-carbon double bond.[11] However, the difunctional group reagent containing tin and tellurium has rarely roused extensive attention. Hydrozirconation is one of the most promising organometallic technique used in organic synthesis and has emerged as a unique hydrometallation with some attractive features,[12] such as the high regioselectivity and stereoselectivity observed with alkynylstannanes.[7] We now report the synthesis of (*E*)-*α*-aryltellurenylvinylstannanes by the hydrozirconation of alkynylstannanes, followed by treatment with aryltellurenyl iodides.

Results and discussion

Alkynylstannanes (**1**) were prepared according to the literature procedures.[7b] The hydrozirconation of **1** with $Cp_2Zr(H)Cl$ in THF at room temperature for 40 min gave (*Z*)-*α*-stannylvinylzirconium (**2**) , which reacted with aryltellurenyl iodides (**3**) to afford (*E*)-*α*-aryltellurenylvinylstannanes (**4**) in good yields (Scheme 1), and the typical results are summarized in Table 1.

Scheme 1

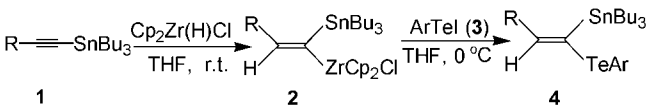

Table 1 (*E*)-*α*-Aryltellurenylvinylstannanes (**4**) prepared according to Scheme 1

Entry	R	Ar	Product	Yield[a]/%
1	*n*-C_4H_9	Ph	**4a**	73
2	*n*-C_4H_9	4-$CH_3C_6H_4$	**4b**	65
3	*n*-C_4H_9	4-ClC_6H_4	**4c**	81
4	CH_3OCH_2	Ph	**4d**	69
5	CH_3OCH_2	4-$CH_3C_6H_4$	**4e**	62
6	CH_3OCH_2	4-ClC_6H_4	**4f**	77
7	Ph	Ph	**4g**	85
8	Ph	4-$CH_3C_6H_4$	**4h**	67
9	Ph	4-ClC_6H_4	**4i**	76

[a] Isolated yield based on **1** used.

The hydrozirconation of alkynylstannanes **1** is 100% regio- and stereoselective as previously described.[7] We observed that the Zr/Te exchange reaction on intermediates **2** occurs with total retention of the configuration. Investigations of the crude products **4** by ^{1}H NMR spectroscopy (300 MHz) showed their isomeric purities to

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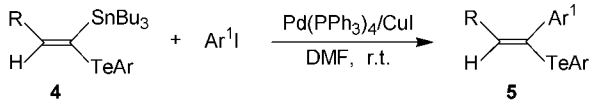
be more than 99%, and one olefinic proton signal of **4a**—**4f** splits characteristically into one triplet with a coupling constant J=7.2 or 5.8 Hz, indicating that the hydrozirconation of the alkynylstannanes had taken place with strong preference for the addition of the zirconium atom at the carbon adjacent to the stannyl group. The stereochemistry for the obtained compounds **4** was confirmed by the NOESY spectra. An enhancement of the aromatic protons next to tellurium was observed as the vinylic proton of **4a** was irradiated. There was no correlation between the vinylic proton and CH_2 bonded to the tin atom. The correlation between the allylic hydrogens and CH_2 bonded to the tin atom was also observed. The NOE results indicate that **4a** has the expected *E* configuration.

Compound **4** are new difunctional group reagents in which two synthetically versatile groups are linked to the same olefinic carbon atom and can be considered as both vinylstannanes and vinyltellurides. The palladium catalyzed coupling reaction of vinylstannanes with organic halides known as Stille coupling has been established as an efficient stereospecific method for the formation of carbon-carbon bonds under mild conditions.[13] Vinyl tellurides have been employed to effect Ni(0)-catalyzed cross coupling reactions with Grignard reagents.[14] Based on the different reactivity of stannyl and tellurenyl groups, (*E*)-*α*-aryltellurenylvinylstannanes (**4**) could undergo sequential cross coupling reactions with electrophiles and nucleophiles in the presence of transition metal complexes to form two carbon-carbon bonds in the same olefinic carbon. Therefore, we carried out the palladium(0)/CuI-cocatalyzed cross coupling reaction of **4** with aryl iodides. When **4** were allowed to react with aryl iodides in the presence of catalytic amounts of $Pd(PPh_3)_4$ and CuI in DMF at room temperature, the corresponding (*E*)-disubstituted vinyltellurides (**5**) were obtained in good yields shown in Scheme 2 and Table 2.

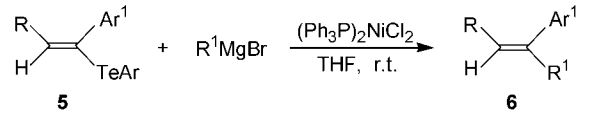
Vinyltellurides are important synthetic intermediates owing to the versatile reactivity of the tellurenyl group and the carbon-carbon double bond.[10] (*E*)-Disubstituted

Scheme 2

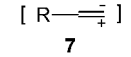


vinyltellurides (**5**) are also effective precursors for preparing stereodefined trisubstituted alkenes. In the presence of bis(triphenylphosphine)nickel(II) chloride catalyst they could easily undergo cross coupling reaction with Grignard reagents to provide an effective method for preparation of trisubstituted alkenes. Indeed, the cross coupling reaction of **5** with Grignard reagents in the presence of $(Ph_3P)_2NiCl_2$ occurred to afford the tellurium free trisubstituted alkenes (**6**) in moderate yields (Scheme 3). The experimental results are summarized in Table 3.

Scheme 3



As compound **4** undergo a two-step cross coupling reaction to form two carbon-carbon bonds on the same olefinic carbon and allow the synthesis of trisubstituted alkenes stereoselectively, **4** can be regarded as the equivalent of the cation-anion synthon (**7**) (Figure 1).



7

Figure 1 Cation-anion synthon **7**.

In conclusion, we have developed a direct route to the synthesis of (*E*)-*α*-aryltellurenylvinylstannanes (**4**) by the hydrozirconation of the alkynylstannanes. Compared to (*E*)-*α*-selanylvinylstannanes[6] whose preparation requires the use of carcinogenic Bu_3SnH as the starting material and $Pd(PPh_3)_4$ as the catalyst, **4** are the

Table 2 (*E*)-Disubstituted vinyltellurides (**5**) prepared according to Scheme 2

Entry	R	Ar	Ar^1	Product	Yield[a]/%
1	*n*-C_4H_9	Ph	Ph	**5a**	64
2	*n*-C_4H_9	Ph	4-ClC_6H_4	**5b**	73
3	*n*-C_4H_9	4-ClC_6H_4	Ph	**5c**	66
4	Ph	Ph	4-ClC_6H_4	**5d**	69
5	CH_3OCH_2	4-ClC_6H_4	Ph	**5e**	61

[a] Isolated yield based on **4** used.

. **Table 3** Stereodefined trisubstituted alkenes (**6**) prepared according to Scheme 3

R	Ar	Ar^1	R^1	Product	Yield[a]/%
n-C_4H_9	Ph	4-ClC_6H_4	*n*-C_4H_9	**6a**	65
CH_3OCH_2	4-ClC_6H_4	Ph	*n*-C_4H_9	**6b**	59
n-C_4H_9	4-ClC_6H_4	Ph	Ph	**6c**	62

[a] Isolated yield based on **5** used.

new difunctional group reagents and can be also used as the effective precursors for the synthesis of the stereodefined trisubstituted alkenes. The procedure for preparing compounds **4** is straightforward and simple.

Experimental

Tetrahydrofuran (THF) was distilled from sodium-benzophenone immediately prior to use. $Cp_2Zr(H)Cl$,[15] $(Ph_3P)_4Pd$[16] and $(Ph_3P)_2NiCl_2$[17] were prepared according to the reported methods. IR spectra were obtained on a Perkin-Elmer 683 instrument as neat films. ^1H NMR spectra were recorded on a Bruker AC-300 (300 MHz) spectrometer using $CDCl_3$ as solvent. Mass spectra were determined on a Finnigan 8230 mass spectrometer. Microanalyses were measured using a Yanaco MT-3 CHN microelemental analyzer.

General procedure for the synthesis of (*E*)-*α*-aryltellurenylvinylstannanes (4a—4i)

To a solution of $Cp_2Zr(H)Cl$ (1.3 mmol) in THF (4 mL) under nitrogen was added a solution of alkynylstannane (**1**) (1.0 mmol) in THF (2 mL). The reaction mixture was stirred at room temperature for 30 min. The resulting solution was cooled to 0 ℃, and a solution of aryltellurenyl iodide (2.0 mmol) in THF (8 mL) was added. The mixture was stirred at 0 ℃ for 30 min, then at room temperature for 30 min. The reaction mixture was diluted with diethyl ether (40 mL) and treated with a 4% $Na_2S_2O_3$ aqueous solution (20 mL). The organic layer was washed with water (3×20 mL) and dried ($MgSO_4$). After removal of the solvent under reduced pressure, the residue was purified by column chromatography on silica gel using light petroleum as eluent.

(*E*)-1-Tributylstannyl-1-phenyltelluro-1-hexene (4a) ^1H NMR ($CDCl_3$, 300 MHz) δ: 7.65—7.59 (m, 2H), 7.24—7.15 (m, 3H), 7.09 (t, J=7.2 Hz, 1H), 2.15—2.08 (m, 2H), 1.54—0.83 (m, 34H); IR (film) ν: 3065, 2956, 2870, 1574, 1463, 1434, 729, 691 cm^{-1}; MS (70 eV) *m/z* (%): 519 (M^+—57, 1.2), 290 (2.4), 205 (6), 117 (17), 77 (46), 57 (57), 41 (100). Anal. calcd for $C_{24}H_{42}SnTe$: C 49.97, H 7.29; found C 49.69, H 7.04.

(*E*)-1-Tributylstannyl-1-(4-methylphenyltelluro)-1-hexene (4b) ^1H NMR ($CDCl_3$, 300 MHz) δ: 7.60—7.51 (m, 2H), 7.12—6.92 (m, 3H), 2.33 (s, 3H), 2.14—2.05 (m, 2H), 1.49—0.72 (m, 34H); IR (film) ν: 3064, 3013, 2955, 2870, 1571, 1486, 1463, 797 cm^{-1}; MS (70 eV) *m/z* (%): 533 (M^+—57, 5.3), 291 (3.8), 219 (6.4), 131 (41), 91 (66), 57 (65), 41 (100). Anal. calcd for $C_{25}H_{44}SnTe$: C 50.82, H 7.45; found C 50.61, H 7.22.

(*E*)-1-Tributylstannyl-1-(4-chlorophenyltelluro)-1-hexene (4c) ^1H NMR ($CDCl_3$, 300 MHz) δ: 7.53 (d, J=8.2 Hz, 2H), 7.15 (d, J=8.2 Hz, 2H), 7.10 (t, J=7.2 Hz, 1H), 2.18—2.10 (m, 2H), 1.49—0.71 (m, 34H); IR (film) ν: 3068, 2926, 2853, 1569, 1470, 1377, 1089, 808 cm^{-1}; MS (70 eV) *m/z* (%): 555 (M^+—57, 2), 291 (1.8), 239 (5), 111 (8), 57 (54), 41 (100). Anal. calcd for $C_{24}H_{41}ClSnTe$: C 47.15, H 6.71; found C 47.28, H 6.60.

(*E*)-1-Tributylstannyl-1-phenyltelluro-3-methoxy-1-propene (4d) ^1H NMR ($CDCl_3$, 300 MHz) δ: 7.79—7.69 (m, 2H), 7.44—7.19 (m, 3H), 6.89 (t, J=5.8 Hz, 1H), 3.86 (d, J=5.8 Hz, 2H), 3.30 (s, 3H), 1.59—0.75 (m, 27H); IR (film) ν: 3066, 2922, 2870, 1574, 1463, 1434, 1113, 731, 691 cm^{-1}; MS (70 eV) *m/z* (%): 507 (M^+—57, 4.3), 289 (2.5), 205 (3.2), 117 (30), 77 (41), 71 (83), 45 (67), 41 (100). Anal. calcd for $C_{22}H_{38}OSnTe$: C 46.78, H 6.73; found C 46.52, H 6.51.

(*E*)-1-Tributylstannyl-1-(4-methylphenyltelluro)-3-methoxy-1-propene (4e) ^1H NMR ($CDCl_3$, 300 MHz) δ: 7.61 (d, J=7.8 Hz, 2H), 7.05 (d, J=7.8 Hz, 2H), 6.79 (t, J=5.8 Hz, 1H), 3.84 (d, J=5.8 Hz, 2H), 3.29 (s, 3H), 2.34 (s, 3H), 1.55—0.86 (m, 27H); IR (film) ν: 3064, 3013, 2955, 2870, 1680, 1588, 1485, 1462, 1113, 798 cm^{-1}; MS (70 eV) *m/z* (%): 521 (M^+—57, 16.2), 289 (7), 219 (2.3), 161 (34), 127 (39), 91 (42), 71 (45), 57 (49), 45 (56), 41 (100). Anal. calcd for $C_{23}H_{40}OSnTe$: C 47.73, H 6.92; found C 47.56, H 6.70.

(*E*)-1-Tributylstannyl-1-(4-chlorophenyltelluro)-3-methoxy-1-propene (4f) ^1H NMR ($CDCl_3$, 300 MHz) δ: 7.61 (d, J=8.1 Hz, 2H), 7.19 (d, J=8.1 Hz, 2H), 6.88 (t, J=5.8 Hz, 1H), 3.87 (d, J=5.8 Hz, 2H), 3.31 (s, 3H), 1.52—0.85 (m, 27H); IR (film) ν: 3068, 2955, 1634, 1568, 1470, 1375, 1113, 1009, 809, 721, 690 cm^{-1}; MS (70 eV) *m/z* (%): 542 (M^+—57, 6), 289 (2.5), 239 (2.9), 127 (54), 111 (7.4), 71 (96), 57 (55), 45 (96), 41 (100). Anal. calcd for $C_{22}H_{37}ClOSnTe$: C 44.09, H 6.18; found C 44.23, H 6.09.

(*E*)-1-Tributylstannyl-1-phenyltelluro-2-phenylethene (4g) ^1H NMR ($CDCl_3$, 300 MHz) δ: 7.79—7.12 (m, 11H), 1.46—0.74 (m, 27H); IR (film) ν: 3054, 3021, 2955, 2870, 1598, 1574, 1487, 1463, 730, 692 cm^{-1}; MS (70 eV) *m/z* (%): 539 (M^+—57, 9.2), 233 (3.4), 205 (5), 179 (28), 77 (65), 57 (80), 41 (100). Anal. calcd for $C_{26}H_{38}SnTe$: C 52.32, H 6.37; found C 52.45, H 6.32.

(*E*)-1-Tributylstannyl-1-(4-methylphenyltelluro)-2-phenylethene (4h) ^1H NMR ($CDCl_3$, 300 MHz) δ: 7.69—7.03 (m, 10H), 2.34 (s, 3H), 1.47—0.73 (m, 27H); IR (film) ν: 3058, 3019, 2954, 2869, 1597, 1486, 1462, 1013, 798, 694 cm^{-1}; MS (70 eV) *m/z* (%): 553 (M^+—57, 4.5), 233 (3), 221 (34), 219 (32), 182 (28), 102 (20), 91 (100). Anal. calcd for $C_{27}H_{40}SnTe$: C 53.09, H 6.55; found C 52.81, H 6.31.

(*E*)-1-Tributylstannyl-1-(4-chlorophenyltelluro)-2-phenylethene (4i) ^1H NMR ($CDCl_3$, 300 MHz) δ: 7.70—7.12 (m, 10H), 1.43—0.73 (m, 27H); IR (film) ν: 3056, 3021, 2955, 2870, 1598, 1567, 1487, 1471, 1378, 1089, 1008, 809, 694 cm^{-1}; MS (70 eV) *m/z* (%): 574 (M^+—57, 4.5), 241 (58), 239 (50), 111 (62), 102 (66), 75 (100). Anal. calcd for $C_{26}H_{37}ClSnTe$: C 49.46, H 5.87; found C 49.22, H 5.65.

General procedure for the synthesis of (*E*)-disubstituted vinyltellurides (5a—5e) To a solution of (*E*)-*α*-aryltellurenylvinylstannane (**4**) (1.0 mmol), aryl iodide (1.1 mmol) and $Pd(PPh_3)_4$ (0.05 mmol) in DMF (5 mL) was added CuI (0.1 mmol) under nitrogen. The reaction mixture was stirred at room temperature for 48

h. The resulting mixture was treated with sat. aq. NH_4Cl (10 mL) and extracted with CH_2Cl_2 (2×15 mL). The organic layer was washed with sat. aq. NH_4Cl (2×10 mL), water (3×20 mL) and dried ($MgSO_4$). After removal of the solvent, the residue was purified by column chromatography on silica gel eluting with light petroleum.

(*E*)-1-Phenyl-1-phenyltelluro-1-hexene (5a) ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.66—7.11 (m, 10H), 6.30 (t, J=7.1 Hz, 1H), 2.11—2.00 (m, 2H), 1.40—1.21 (m, 4H), 0.82 (t, J=7.0 Hz, 3H); IR (film) v: 3065, 2955, 2871, 1594, 1574, 1464, 729, 690 cm^{-1}. Anal. calcd for $C_{18}H_{20}Te$: C 59.41, H 5.50; found C 59.18, H 5.39.

(*E*)-1-(4-Chlorophenyl)-1-phenyltelluro-1-hexene (5b) ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.70—7.08 (m, 9H), 6.29 (t, J=7.2 Hz, 1H), 2.12—2.02 (m, 2H), 1.42—1.20 (m, 4H), 0.84 (t, J=7.0 Hz, 3H); IR (film) v: 3066, 2961, 2870, 1595, 1571, 1463, 730, 692 cm^{-1}. Anal. calcd for $C_{18}H_{19}ClTe$: C 54.27, H 4.77; found C 54.03, H 4.56.

(*E*)-1-Phenyl-1-(4-chlorophenyltelluro)-1-hexene (5c) ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.71—7.10 (m, 9H), 6.31 (t, J=7.0 Hz, 1H), 2.15—2.08 (m, 2H), 1.39—1.18 (m, 4H), 0.86 (t, J=7.0 Hz, 3H); IR (film) v: 3065, 3019, 2957, 2871, 1593, 1575, 1463, 729, 690 cm^{-1}. Anal. calcd for $C_{18}H_{19}ClTe$: C 54.27, H 4.77; found C 54.34, H 4.62.

(*E*)-1-Phenyltelluro-1-(4-chlorophenyl)-2-phenylethene (5d) ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.73—7.07 (m, 15H); IR (film) v: 3068, 1598, 1574, 730, 691 cm^{-1}. Anal. calcd for $C_{20}H_{15}ClTe$: C 57.42, H 3.59; found C 57.18, H 3.41.

(*E*)-1-Phenyl-1-(4-chlorophenyltelluro)-3-methoxy-1-propene (5e) ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.71—7.09 (m, 9H), 6.79 (t, J=5.9 Hz, 1H), 3.88 (d, J=5.7 Hz, 2H), 3.30 (s, 3H); IR (film) v: 3066, 1594, 1574, 1113, 1018, 731, 692 cm^{-1}. Anal. calcd for $C_{16}H_{15}ClOTe$: C 49.74, H 3.89; found C 49.51, H 3.73.

General procedure for the synthesis of trisubstituted alkenes (6a—6c)

To a stirred suspension of $Ni(PPh_3)_2Cl_2$ (0.05 mmol) and (*E*)-disubstituted vinyltelluride (**5**) (1 mmol) in THF (5 mL) was added a solution of R^1MgBr (4 mmol) in THF (6 mL) under nitrogen at room temperature and the mixture was stirred for 48 h. The mixture was treated with sat. aq. NH_4Cl (10 mL) and extracted with ether (2×30 mL). The ethereal solution was washed with water (3×20 mL) and dried ($MgSO_4$). Removal of the solvent under reduced pressure gave an oil which was purified by column chromatography on silica gel eluting with light petroleum.

(*Z*)-5-(4-Chlorophenyl)-5-decene (6a) ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.22 (d, J=8.2 Hz, 2H), 6.91 (d, J=8.2 Hz, 2H), 5.83 (t, J=7.0 Hz, 1H), 2.32—1.96 (m, 4H), 1.63—0.69 (m, 14H); IR (film) v: 3080, 3010, 2956, 2871, 1638, 1594, 1492, 1470, 841, 800 cm^{-1}. Anal. calcd for $C_{16}H_{23}Cl$: C 76.65, H 9.18; found C 76.48, H 9.07.

(*Z*)-1-Methoxy-3-phenyl-2-heptene (6b) ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.34—7.08 (m, 5H), 6.18 (t, J=7.0 Hz, 1H), 3.86 (d, J=5.8 Hz, 2H), 3.25 (s, 3H), 2.36—2.06 (m, 2H), 1.67—1.12 (m, 4H), 0.89 (t, J=6.8 Hz, 3H); IR (film) v: 3058, 3025, 2922, 1645, 1598, 1495, 1464, 1096, 700 cm^{-1}. Anal. calcd for $C_{14}H_{20}O$: C 82.35, H 9.80; found C 82.13, H 9.70.

1,1-Diphenyl-1-hexene (6c) ^{1}H NMR ($CDCl_3$, 300 MHz) δ: 7.46—6.83 (m, 10H), 5.90 (t, J=7.0 Hz, 1H), 2.36—1.89 (m, 2H), 1.68—1.20 (m, 4H), 0.88 (t, J=5.4 Hz, 3H); IR (film) v: 3079, 3056, 3022, 2956, 2871, 1598, 1494, 1443, 699 cm^{-1}. Anal. calcd for $C_{18}H_{20}$: C 91.53, H 8.47; found C 91.31, H 8.31.

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