

Novel stereoselective synthesis of 1-substituted 1,3-dien-2-yl sulfides via Stille coupling reactions of (*E*)- α -stannylvinyl sulfides with alkenyl iodides

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Received 1 June 2005; received in revised form 6 October 2005; accepted 6 October 2005

Available online 10 November 2005

Abstract

(*E*)- α -stannylvinyl sulfides are new difunctional reagents which undergo Stille coupling reactions with alkenyl iodides to afford stereoselectively 1-substituted 1,3-dien-2-yl sulfides in good yields.

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Keywords: (*E*)- α -stannylvinyl sulfide; 1,3-Dienyl sulfides; Difunctional reagent; Stille coupling; Stereoselective synthesis

1. Introduction

The stereocontrolled synthesis of conjugated dienes is of considerable interest in organic chemistry since such dienes are valuable synthetic intermediates and often encountered in natural compounds, such as insect sex pheromones and Achillea amide [1]. The synthesis of dienes for use in the Diels–Alder reaction is still an important challenge in organic synthesis [2] although other elegant uses of these compounds have been developed [3]. Conjugated dienes are usually prepared by utilizing either a Wittig type approach [4] or coupling reactions of stereodefined vinyl halides with vinyl organometallic compounds catalyzed by transition metals [5]. Recently, Whitby et al. [6] reported the insertion of 1-lithio-1-halobutadiene into organozirconocenes providing a stereocontrolled synthesis of (*E,Z*)-1,3-dienes.

The synthesis of 1,3-dienes containing functional groups is also of considerable interest in recent years. The stereoselective synthesis of 1,3-dienylsilanes [7], 1,3-dienyl selenides [8] and 1,3-dienylstannanes [9] has already been described in the literatures. 1,3-Dienyl sulfides serve as valuable versatile

intermediates since vinyl sulfides are synthetically equivalent to carbonyls and can be stereospecifically converted to alkenes by nickel-catalyzed coupling reactions with Grignard reagents [10]. In addition, 1,3-dienyl sulfides play a very important role in Diels–Alder reactions, where they impart an added level of reactivity and regioselectivity to such cycloadditions [11]. Due to their synthetic utility, a variety of methods have been developed for their preparation [12]. However, the stereoselective synthesis of polysubstituted 1,3-dienyl sulfides has rarely been reported [13]. Liebeskind et al. [14] reported that α -(tributylstannyl)thiophene and iodoolefins were successfully Stille-coupled to afford different products containing sulfur-substituted 1,3-diene structural skeleton. Recently, Jin et al. [15] have developed a convergent approach for the stereoselective synthesis of (*Z,Z*)-2-alkoxy-3-alkyl(aryl)thiobuta-1,3-dienes via Negishi coupling between α -alkyl(aryl)thio vinyl zinc chloride and α -bromo vinyl ether. Vinylstannanes are pivotal intermediates in a wide range of carbon–carbon bond forming reactions [16]. The Stille coupling reactions of vinylstannanes with alkenyl iodides provides a convenient route to stereoselective synthesis of 1,3-dienes [17]. Herein, we wish to report that 1-substituted 1,3-dien-2-yl sulfides could be synthesized by Stille coupling reactions of (*E*)- α -stannylvinyl sulfides with alkenyl iodides.

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2. Results and discussion

Palladium-catalyzed hydrostannylation of alkynylsulfides provides a simple general route for the synthesis of (*E*)- α -stannylvinyl sulfides [18]. (*E*)- α -stannylvinyl sulfides are new difunctional group reagents in which two synthetically versatile groups are linked to the same olefinic carbon atom and can be considered both as vinylstannanes and as vinyl sulfides. With a convenient route to the (*E*)- α -stannylvinyl sulfides **1** we decided to establish the feasibility of using **1** in Stille coupling reactions with alkenyl iodides **2**. Gratifyingly, when the coupling reactions of **1** with a variety of alkenyl iodides **2** were conducted in DMF at room temperature using Pd(PPh₃)₄ and CuI as co-catalyst (Scheme 1), fairly rapid reactions occurred affording stereoselectively the desired coupling products **3** in good yields (Table 1).

It is well documented that the cross-coupling reaction (Stille coupling) of vinylstannanes with organic halides in the presence of a palladium catalyst occurs with retention of configuration [16a,16d]. The *E*-configuration of the compounds **3a**, **3e**, **3f**, **3i** has been proved by their ¹H NMR spectra which show a doublet at $\delta = 6.08$ – 6.89 with a coupling constant of 14.8–15.6 Hz, and this is also the evidence of the retention of the *E*-configuration of the starting compounds **2**. In addition, the *Z*-configuration of the compound **3f** was confirmed by the NOESY in the ¹H NMR spectrum. An enhancement of the allylic protons was observed as the vinylic proton ($\delta = 6.25$) of **3f** was irradiated. There was no correlation between the vinylic proton ($\delta = 6.25$) and aromatic proton. The correlation between the vinylic proton ($\delta = 6.25$) and another vinylic proton ($\delta = 6.30$) was also observed. The NOE results indicate

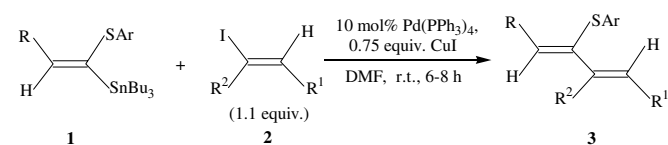


Table 1
Synthesis of 1-substituted 1,3-dien-2-yl sulfides **3a–i**

| Entry | R | Ar | R ¹ | R ² | Product ^a | Yield (%) ^b |
|-------|-----------------------------------------|-------------------------------------------------|-----------------------------------------|----------------|----------------------|------------------------|
| 1 | <i>n</i> -C ₄ H ₉ | Ph | <i>n</i> -C ₄ H ₉ | H | 3a | 79 |
| 2 | CH ₃ OCH ₂ | Ph | <i>n</i> -C ₄ H ₉ | H | 3b | 73 |
| 3 | <i>n</i> -C ₄ H ₉ | 4-ClC ₆ H ₄ | CH ₃ OCH ₂ | H | 3c | 76 |
| 4 | <i>n</i> -C ₄ H ₉ | 4-ClC ₆ H ₄ | -(CH ₂) ₄ - | H | 3d | 82 |
| 5 | Ph | Ph | CH ₃ OCH ₂ | H | 3e | 69 |
| 6 | <i>n</i> -C ₄ H ₉ | 4-CH ₃ C ₆ H ₄ | CH ₃ OCH ₂ | H | 3f | 77 |
| 7 | Ph | Ph | -(CH ₂) ₄ - | H | 3g | 71 |
| 8 | <i>n</i> -C ₄ H ₉ | 4-CH ₃ C ₆ H ₄ | -(CH ₂) ₄ - | H | 3h | 83 |
| 9 | <i>n</i> -C ₄ H ₉ | 4-ClC ₆ H ₄ | Ph | H | 3i | 72 |

^a All the products were characterized by IR, ¹H NMR, ¹³C NMR, MS and elemental analyses.

^b Isolated yield based on the **1** used.

that **3f** has the expected *Z*-configuration and the cross-coupling reaction of (*E*)- α -stannylvinyl sulfides with alkenyl iodides occurs with the configuration retention of both the starting compounds **1** and the compounds **2**.

In conclusion, we have developed a novel route to the stereoselective synthesis of 1-substituted 1,3-dien-2-yl sulfides **3**. The method has the advantages of readily available starting materials, simple procedures, mild reaction conditions and good yields. Investigations into the synthetic applications of 1-substituted 1,3-dien-2-yl sulfides **3** are currently in progress.

3. Experimental

¹H NMR spectra were recorded on a Bruker AC-P400 (400 MHz) spectrometer with TMS as an internal standard using CDCl₃ as the solvent. ¹³C NMR (100 MHz) spectra were recorded on a Bruker AC-P400 (400 MHz) spectrometer using CDCl₃ as the solvent. IR spectra were determined on an FTS-185 instrument as neat films. Mass spectra were obtained on a Finigan 8239 mass spectrometer. Microanalyses were measured using a Yanaco MT-3 CHN microelemental analyser. All reactions were carried out in pre-dried glassware (150 °C, 4 h) and cooled under a stream of dry nitrogen. THF was freshly distilled from sodium-benzophenone prior to its use. DMF was dried by distillation over calcium hydride. (*E*)- α -stannylvinyl sulfides **1** were prepared by Pd(PPh₃)₄-catalyzed hydrostannylation of alkynylsulfides with Bu₃SnH in THF according to the literature procedure [18].

3.1. General procedure for the cross-coupling reactions

(*E*)- α -stannylvinyl sulfide **1** (1.0 mmol) and alkenyl iodide **2** (1.1 mmol) were dissolved in DMF (10 ml) under nitrogen at room temperature. Pd(PPh₃)₄ (0.10 mmol) and CuI (0.75 mmol) were then added. The mixture was stirred for 6–8 h at room temperature and monitored by TLC (SiO₂) for the disappearance of the starting (*E*)- α -stannylvinyl sulfide **1**. The reaction mixture was diluted with diethyl ether (30 ml), filtered and then treated with 20% aqueous KF (10 ml) for 30 min before being dried and concentrated. The residue was purified by column chromatography on silica gel, eluting either with a mixture of diethyl ether and petroleum or just petroleum.

3.1.1. (5*Z*,7*E*)-6-phenylthio-5,7-dodecadiene (**3a**)

IR (film): ν (cm⁻¹) 3059, 2958, 1584, 1478, 1465, 1439, 1378, 961, 737, 689; ¹H NMR (CDCl₃): δ 7.26–7.08 (m, 5H), 6.16 (t, *J* = 7.2 Hz, 1H), 6.08 (d, *J* = 14.8 Hz, 1H), 6.01–5.92 (m, 1H), 2.46–2.40 (m, 2H), 2.04–1.97 (m, 2H), 1.41–1.15 (m, 8H), 0.89–0.79 (m, 6H); ¹³C NMR (CDCl₃): δ 141.5, 137.0, 133.4, 130.9, 130.2, 128.7, 127.1, 124.8, 32.0, 31.4, 30.0, 22.4, 22.3, 22.0, 14.0, 13.9; MS: *m/z* 275 (M⁺ + 1, 100), 219 (11.3), 149 (12.5), 109 (3.8); Anal. Calc. for C₁₈H₂₆S: C, 78.83; H, 9.49. Found: C, 78.59; H, 9.28%.

3.1.2. (2Z,4E)-1-methoxy-3-phenylthio-2,4-nonadiene (3b)

IR (film): ν (cm⁻¹) 3058, 2957, 2926, 1643, 1583, 1478, 1440, 1376, 1119, 961, 739, 690; ¹H NMR (CDCl₃): δ 7.26–7.11 (m, 5H), 6.21 (t, J = 6.0 Hz, 1H), 6.11–6.05 (m, 2H), 4.29 (d, J = 6.4 Hz, 2H), 3.34 (s, 3H), 2.06–2.00 (m, 2H), 1.27–1.14 (m, 4H), 0.81 (t, J = 7.2 Hz, 3H); ¹³C NMR (CDCl₃): δ 135.9, 135.7, 133.5, 130.6, 129.3, 128.8, 127.7, 125.4, 70.5, 58.3, 32.0, 31.2, 22.0, 13.9; MS: m/z 262 (M⁺, 18.9), 249 (39), 233 (21.5), 221 (37), 169 (68), 147 (83), 135 (94), 109 (100); Anal. Calc. for C₁₆H₂₂OS: C, 73.28; H, 8.40. Found: C, 73.01; H, 8.33%.

3.1.3. (2E,4Z)-1-methoxy-4-(4-chlorophenylthio)-2,4-nonadiene (3c)

IR (film): ν (cm⁻¹) 2957, 2925, 1645, 1598, 1476, 1380, 1092, 1011, 962, 814; ¹H NMR (CDCl₃): δ 7.17 (d, J = 8.8 Hz, 2H), 7.08 (d, J = 8.8 Hz, 2H), 6.35–6.28 (m, 2H), 6.05–6.01 (m, 1H), 3.92 (d, J = 5.6 Hz, 2H), 3.25 (s, 3H), 2.46–2.40 (m, 2H), 1.41–1.19 (m, 4H), 0.87 (t, J = 7.2 Hz, 3H); ¹³C NMR (CDCl₃): δ 144.9, 135.2, 132.2, 130.8, 130.0, 128.9, 128.3, 128.2, 72.3, 57.9, 31.2, 30.1, 22.4, 13.9; MS: m/z 297 (M⁺, 11.4), 267 (36), 265 (100), 209 (12.5), 121 (6); Anal. Calc. for C₁₆H₂₁OSCl: C, 64.76; H, 7.08. Found: C, 64.58; H, 6.89%.

3.1.4. (Z)-1-(1-cyclohexenyl)-1-(4-chlorophenylthio)-1-hexene (3d)

IR (film): ν (cm⁻¹) 3019, 2926, 1631, 1574, 1474, 1389, 1092, 1011, 812, 742; ¹H NMR (CDCl₃): δ 7.16 (d, J = 8.8 Hz, 2H), 7.04 (d, J = 8.8 Hz, 2H), 6.23–6.17 (m, 2H), 2.43–2.39 (m, 2H), 2.21–2.17 (m, 2H), 2.07–2.01 (m, 2H), 1.64–1.58 (m, 2H), 1.52–1.48 (m, 2H), 1.42–1.21 (m, 4H), 0.88 (t, J = 7.2 Hz, 3H); ¹³C NMR (CDCl₃): δ 137.0, 136.3, 135.0, 134.1, 130.5, 128.7, 128.3, 128.1, 31.5, 30.4, 28.9, 27.1, 25.9, 22.9, 22.4, 14.0; MS: m/z 307 (M⁺, 100), 279 (7.4), 237 (17.4), 163 (8.5); Anal. Calc. for C₁₈H₂₃S: C, 70.47; H, 7.50. Found: C, 70.18; H, 7.23%.

3.1.5. (1Z,3E)-1-phenyl-2-phenylthio-5-methoxy-1,3-pentadiene (3e)

IR (film): ν (cm⁻¹) 3057, 2926, 1580, 1478, 1439, 1383, 1116, 1039, 977, 746, 687; ¹H NMR (CDCl₃): δ 7.67 (d, J = 7.6 Hz, 2H), 7.31–7.06 (m, 9H), 6.46 (d, J = 15.2 Hz, 1H), 6.24–6.20 (m, 1H), 3.95 (d, J = 5.6 Hz, 2H), 3.22 (s, 3H); ¹³C NMR (CDCl₃): δ 139.6, 136.0, 133.3, 130.2, 129.9, 129.0, 128.3, 128.2, 127.5, 125.5, 72.3, 57.9; MS: m/z 282 (M⁺, 7.8), 251 (100), 142 (17.4); Anal. Calc. for C₁₈H₁₈OS: C, 76.60; H, 6.38. Found: C, 76.43; H, 6.14%.

3.1.6. (2E,4Z)-1-methoxy-4-(4-methylphenylthio)-2,4-nonadiene (3f)

IR (film): ν (cm⁻¹) 3020, 2924, 1646, 1599, 1492, 1454, 1379, 1123, 1087, 962, 804; ¹H NMR (CDCl₃): δ 7.07 (d, J = 8.0 Hz, 2H), 7.01 (d, J = 8.0 Hz, 2H), 6.30 (d, J = 15.6 Hz, 1H), 6.25 (t, J = 7.2 Hz, 1H), 6.08–6.04 (m, 1H), 3.91 (d, J = 5.6 Hz, 2H), 3.22 (s, 3H), 2.47–2.41 (m, 2H), 2.27 (s, 3H), 1.41–1.26 (m, 4H), 0.88 (t, J = 7.2 Hz,

3H); ¹³C NMR (CDCl₃): δ 144.0, 134.9, 132.9, 132.8, 130.7, 129.5, 128.0, 127.3, 72.4, 57.7, 31.3, 30.1, 22.4, 20.9, 13.9; MS: m/z 277 (M⁺ + 1, 44), 276 (M⁺, 7.3), 275 (29), 245 (100), 189 (24), 161 (37), 149 (23), 123 (25), 79 (29); Anal. Calc. for C₁₇H₂₄OS: C, 73.91; H, 8.70. Found: C, 73.63; H, 8.61%.

3.1.7. (Z)-1-(1-cyclohexenyl)-1-phenylthio-2-phenylethene (3g)

IR (film): ν (cm⁻¹) 3057, 3020, 2928, 2856, 1626, 1583, 1491, 1477, 1440, 739, 690; ¹H NMR (CDCl₃): δ 7.59 (d, J = 7.6 Hz, 2H), 7.34–7.15 (m, 8H), 7.04 (s, 1H), 6.35–6.31 (m, 1H), 2.31–2.25 (m, 2H), 2.07–2.02 (m, 2H), 1.61–1.56 (m, 2H), 1.49–1.44 (m, 2H); ¹³C NMR (CDCl₃): δ 137.1, 136.3, 132.2, 129.8, 129.6, 128.6, 128.4, 127.9, 127.4, 125.4, 27.6, 26.0, 22.9, 22.1; MS: m/z 292 (M⁺, 86), 183 (79), 141 (100), 115 (47), 109 (26); Anal. Calc. for C₂₀H₂₀S: C, 82.19; H, 6.85. Found: C, 82.24; H, 6.63%.

3.1.8. (Z)-1-(1-cyclohexenyl)-1-(4-methylphenylthio)-1-hexene (3h)

IR (film): ν (cm⁻¹) 3022, 2926, 1632, 1598, 1492, 1448, 1087, 802; ¹H NMR (CDCl₃): δ 7.04–6.98 (m, 4H), 6.26–6.22 (m, 1H), 6.15 (t, J = 7.6 Hz, 1H), 2.44–2.40 (m, 2H), 2.27 (s, 3H), 2.21–2.17 (m, 2H), 2.05–2.01 (m, 2H), 1.61–1.57 (m, 2H), 1.51–1.46 (m, 2H), 1.42–1.20 (m, 4H), 0.87 (t, J = 7.2 Hz, 3H); ¹³C NMR (CDCl₃): δ 136.2, 135.4, 134.8, 134.5, 134.0, 129.4, 127.8, 127.4, 31.7, 30.3, 27.2, 25.9, 23.0, 22.5, 22.3, 21.0, 14.0; MS: m/z 287 (M⁺ + 1, 100), 217 (14.1), 161 (8.7); Anal. Calc. for C₁₉H₂₆S: C, 79.72; H, 9.09. Found: C, 79.44; H, 9.01%.

3.1.9. (1E,3Z)-1-phenyl-3-(4-chlorophenylthio)-1,3-octadiene (3i)

IR (film): ν (cm⁻¹) 3059, 3025, 2956, 2927, 1624, 1594, 1574, 1493, 1475, 1389, 1092, 958, 814, 751, 692; ¹H NMR (CDCl₃): δ 7.42–7.09 (m, 9H), 6.89 (d, J = 15.6 Hz, 1H), 6.83 (d, J = 15.6 Hz, 1H), 6.42 (t, J = 7.6 Hz, 1H), 2.49–2.44 (m, 2H), 1.45–1.25 (m, 4H), 0.88 (t, J = 7.2 Hz, 3H); ¹³C NMR (CDCl₃): δ 145.5, 137.0, 135.4, 130.8, 129.4, 129.0, 128.6, 128.1, 127.6, 126.6, 31.3, 30.4, 22.5, 14.0; MS: m/z 329 (M⁺, 100), 273 (20.4), 185 (9.6), 129 (13.3); Anal. Calc. for C₂₀H₂₁S: C, 73.06; H, 6.39. Found: C, 72.82; H, 6.16%.

Acknowledgments

We thank the National Natural Science Foundation of China (Project No. 20462002) and the Natural Science Foundation of Jiangxi Province (Project No. 0420015) for financial support.

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