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A Novel Stereoselective Synthesis of (2*Z*)-2-Arylsulfanylallylic Alcohols by Tin–Lithium Exchange of (*E*)-α-Stannylvinyl Sulfides

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ABSTRACT: *Tin–lithium exchange reaction of (E)-α-stannylvinyl sulfides* **1** *with n-butyllithium gave (Z)-α-arylsulfanylvinyllithiums* **2**, *which reacted with aldehydes or ketones* **3** *to afford stereoselectively (2Z)-2-arylsulfanylallylic alcohols* **4** *in good to high yields*. © 2008 Wiley Periodicals, Inc. Heteroatom Chem 19:639–643, 2008; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20487

INTRODUCTION

Stereodefined trisubstituted alkenes exist widely in both natural and nonnatural products [1–5], but their synthesis is still a challenging problem in synthetic organic chemistry [6,7]. Disubstituted alcohols are among the most versatile intermediates in organic synthesis [8,9] and are pervasive in natural products and commercially important pharmaceuticals [10–13]. Thus, the stereocontrolled synthesis of substituted allylic alcohols has attracted much interest in organic chemistry [14–19]. Substituted allylic alcohols are usually prepared by the Reformatsky reaction of the corresponding ketones [20], or by a Horner–Wadsworth–Emmons reaction [21,22], but a mixture of isomers is often obtained. Although substantial progress has been made in the synthesis of (*E*)-di- and (*E*)-trisubstituted allylic alcohols [23–27], the one-pot synthesis of (*E*)-2,3-disubstituted allylic alcohols remains a formidable challenge [28].

The stereocontrolled synthesis of allylic alcohols containing metal or heteroatom functional groups is also of considerable interest in organic synthesis because many useful functional group transformations can be achieved by introduction and removal of metal or heteroatom functions. Lee and Kim [29] reported efficient preparation of enantiomerically pure (3*E*)-4-(tributylstannyl)but-3-en-2-ol by lipase-mediated resolution. The stereoselective synthesis of (2*Z*)-2-(butylseleno)allylic alcohols [30], (2*Z*)-2-silylallylic alcohols [31], (2*E*)-3-silylallylic alcohols [32], (2*E*)-2-sulfonylallylic alcohols [33,34], and (2Z)-2-arylselenoallylic alcohols [35] has also been described in the literature. Recently, Huang and Xie [36] reported an efficient synthesis of (*Z*)-2,3-difunctionalized allylic alcohols by the stereoselective Michael–Aldol tandem reaction of phenylselenomagnesium bromide with acetylenic sulfones and aldehydes. Silveira et al. [37] described

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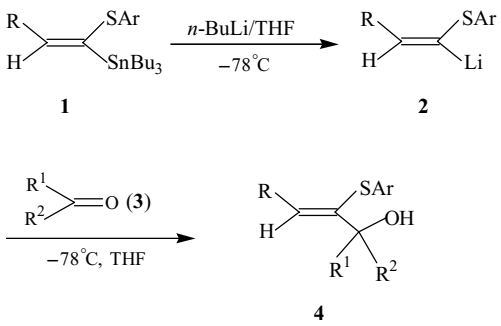
the synthesis of 2-alkylthioallylic alcohols by the Te–Li exchange reaction of α-tellurenylvinyl sulfides with *n*-butyllithium, followed by treatment with aldehydes, but the reaction was rather complicated and a mixture of isomers was obtained. Tanaka and coworkers [38] reported the synthesis of (2*Z*)-2-phenylthio-3-methoxycarbonylallylic alcohols by palladium-catalyzed thioesterification of alkynes with *O*-methyl *S*-phenyl thiocarbonate. Very recently, Zeni and coworkers [39] described a highly stereoselective one-pot procedure to prepare (2*Z*)-2-phenylthio-3-phenylselenoallylic alcohols via addition of chalcogens to alkynes. Despite the availability of synthetic methods of (2*Z*)-2-arylsulfanylallylic alcohols as described above, there still exists a need for new selective and convenient procedures. Herein, we wish to report that (2*Z*)-2-arylsulfanylallylic alcohols could be conveniently synthesized in one pot by the tin–lithium exchange reaction of (*E*)-α-stannylvinyl sulfides with *n*-butyllithium, followed by the treatment with aldehydes or ketones.

RESULTS AND DISCUSSION

(*E*)-α-Stannylvinyl sulfides **1** were prepared regio- and stereoselectively by the palladium-catalyzed hydrostannylation reaction of alkynylsulfides according to a literature procedure [40]. (*E*)-α-Stannylvinyl sulfides **1** are very useful 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. Vinylstannanes can undergo the tin–lithium exchange reaction with *n*-butyllithium to afford the corresponding vinyllithiums with the retention of the configuration [41,42]. With a convenient route to the (*E*)-α-stannylvinyl sulfides **1**, we decided to establish the feasibility of using **1** in tin–lithium exchange reactions with *n*-butyllithium. We observed that the tin–lithium exchange reaction of (*E*)-α-stannylvinyl sulfides **1** with *n*-butyllithium at −78°C in THF for 30 min gave (*Z*)-α-arylsulfanylvinyllithiums **2**, which reacted with aldehydes or ketones **3** to afford stereoselectively the desired (2*Z*)-2-arylsulfanylallylic alcohols **4** (Scheme 1). Typical results are summarized in Table 1.

As shown in Table 1, both a variety of aldehydes and ketones could react rapidly with the intermediates **2** at −78°C to afford the corresponding (2*Z*)-2-arylsulfanylallylic alcohols **4** in good to high yields. This methodology for (2*Z*)-2-arylsulfanylallylic alcohols has a large scope because a variety of (*E*)-α-stannylvinyl sulfides can be conveniently prepared by the hydrostannylation of alkynylsulfides with high regio- and stereoselectivities and high yields [40]. Investigations of the crude products **4** by ^{1}H NMR spectroscope (400 MHz) showed their isomeric purities to be more than 98%. The configuration of intermediates **2** could be confirmed from compounds **5**, which were obtained by hydrolysis with aqueous NH_4Cl (Scheme 2). The stereochemistry of compounds **5** was easily established, since ^{1}H NMR spectra of **5a–b** give rise to a doublet at $\delta = 6.14$ or 6.53 with a coupling constant of 9.2 or 10.8 Hz, which is consistent with a *Z*-configuration. In addition, the *Z*-configuration of the compound **4c** was confirmed by the nuclear Overhauser effect spectroscopy (NOESY) in the ^{1}H NMR spectrum. An enhancement of the allylic protons was observed as the vinylic proton ($\delta = 6.40$) of **4c** was irradiated. The correlation between the allylic protons and aromatic proton was also observed. There was no correlation between the vinylic proton ($\delta = 6.40$) and aromatic proton. The nuclear Overhauser effect (NOE) results indicate that **4c** has the expected *Z*-configuration.

In summary, a convenient one-pot synthetic method for (2*Z*)-2-arylsulfanylallylic alcohols has been developed by tin–lithium exchange reaction of

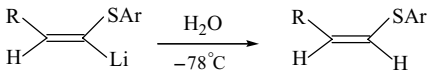


SCHEME 1

TABLE 1 Synthesis of (2*Z*)-2-Arylsulfanylallylic Alcohols **4a–j**

R	*Ar*	R^1	R^2	*Product*	*Yield (%)*[a]
n-C_4H_9	Ph	Ph	H	**4a**	83
n-C_4H_9	4-$CH_3C_6H_4$	Ph	H	**4b**	88
n-C_4H_9	4-$CH_3C_6H_4$	CH_3	CH_3	**4c**	76
n-C_4H_9	Ph	Ph	CH_3	**4d**	85
Ph	Ph	Ph	CH_3	**4e**	82
CH_3OCH_2	Ph	CH_3	CH_3	**4f**	87
Ph	4-$CH_3C_6H_4$	CH_3	CH_3	**4g**	81
n-C_4H_9	4-$CH_3C_6H_4$	n-C_6H_{13}	H	**4h**	76
CH_3OCH_2	Ph	n-C_6H_{13}	H	**4i**	87
CH_3OCH_2	Ph	—$(CH_2)_5$—		**4j**	83

[a]Isolated yield based on the (*E*)-α-stannylvinyl sulfide **1** used.



2

5a: R= *n*-C_4H_9, Ar= 4-$CH_3C_6H_4$, 90%
5b: R = Ph, Ar= 4-$CH_3C_6H_4$, 87%

SCHEME 2

(*E*)-α-stannylvinyl sulfides with *n*-butyllithium, followed by treatment with aldehydes or ketones. Compared with the method reported [37], this methodology has the advantages of readily available starting materials, straightforward and simple procedures, high stereoselectivity, and high yields.

EXPERIMENTAL

THF was distilled from sodium benzophenone ketyl prior to use. IR spectra were obtained on a Perkin–Elmer 683 instrument as neat films. ^{1}H NMR spectra were recorded on a Bruker AC-400 (400 MHz) spectrometer with TMS as an internal standard using $CDCl_3$ as solvent. ^{13}C NMR spectra were recorded on a Bruker AC-400 (100 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 (2Z)-2-Arylsulfanylallylic Alcohols **4a–j**

To a solution of (*E*)-α-stannylvinyl sulfide **1** (0.5 mmol) in THF (5 mL) at −78°C under argon, a solution of *n*-BuLi (0.2 mL, 0.5 mmol, 2.5 M solution in hexane) was added dropwise. After 30 min of stirring at this temperature, aldehyde or ketone (0.6 mmol) was added. The mixture was stirred for 1 h at −78°C, then diluted with ethyl acetate (20 mL) and washed with brine (4 × 10 mL). The organic layer was separated and dried over $MgSO_4$, and the solvent was removed under vacuum. The residue was purified by column chromatography on silica gel (eluent: light petroleum ether/EtOAc, 10:1).

(Z)-1-Phenyl-2-phenylsulfanyl-2-hepten-1-ol **4a**. IR (film) ν (cm^{-1}): 3410, 3061, 3029, 2957, 2928, 1714, 1632, 1582, 1477, 1454, 1024, 909, 738, 699; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.33–7.13 (m, 10H), 6.36 (t, $J = 7.2$ Hz, 1H), 5.20 (s, 1H), 2.37–2.31 (m, 2H), 2.18 (br, 1H), 1.41–1.25 (m, 4H), 0.87 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 141.7, 140.0, 135.6, 135.1, 129.0, 128.4, 128.3, 127.8, 126.7, 125.9, 76.8, 31.1, 29.5, 22.4, 13.9; MS *m/z*: 298 (M^+, 5.2), 280 (22), 189 (39), 149 (66), 105 (75), 91 (82), 77 (100); Anal. Found: C, 76.28; H, 7.14. $C_{19}H_{22}OS$ Calcd: C, 76.46; H, 7.43%.

(Z)-1-Phenyl-2-(4-methylphenyl)sulfanyl-2-hepten-1-ol **4b**. IR (film) ν (cm^{-1}): 3417, 3062, 3029, 2957, 2925, 1715, 1632, 1600, 1491, 1454, 1086, 910, 806, 699; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.32–7.26 (m, 5H), 7.14 (d, $J = 8.0$ Hz, 2H), 7.05 (d, $J = 8.0$ Hz, 2H), 6.27 (t, $J = 7.2$ Hz, 1H), 5.17 (s, 1H), 2.38–2.30 (m, 6H), 1.40–1.25 (m, 4H), 0.87 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 141.8, 139.1, 136.0, 135.7, 131.7, 129.8, 129.0, 128.3, 127.7, 126.7, 76.5, 31.1, 29.5, 22.4, 21.0, 13.9; MS *m/z*: 312 (M^+, 4.6), 189 (100), 163 (31), 129 (53), 105 (64), 91 (84), 77 (47); Anal. Found: C, 76.60; H, 7.49. $C_{20}H_{24}OS$ Calcd: C, 76.88; H, 7.74%.

(Z)-2-Methyl-3-(4-methylphenyl)sulfanyl-3-octen-2-ol **4c**. IR (film) ν (cm^{-1}): 3460, 3076, 3031, 2956, 2925, 1628, 1596, 1491, 1467, 1361, 971, 825, 801; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.10 (d, $J = 8.0$ Hz, 2H), 7.03 (d, $J = 8.0$ Hz, 2H), 6.40 (t, $J = 7.2$ Hz, 1H), 2.35 (br, 1H), 2.28 (s, 3H), 2.26–2.20 (m, 2H), 1.41 (s, 6H), 1.37–1.22 (m, 4H), 0.84 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 139.5, 138.1, 134.9, 133.5, 129.6, 126.7, 74.3, 31.0, 30.2, 29.4, 22.4, 20.9, 13.9; MS *m/z*: 264 (M^+, 7.1), 91 (48), 71 (62), 57 (100), 43 (98); Anal. Found: C, 72.41; H, 8.87. $C_{16}H_{24}OS$ Calcd: C, 72.67; H, 9.15%.

(Z)-2-Phenyl-3-phenylsulfanyl-3-octen-2-ol **4d**. IR (film) ν (cm^{-1}): 3475, 3059, 3025, 2957, 2929, 1687, 1599, 1582, 1478, 1447, 1266, 760, 739, 700; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.48–7.17 (m, 10H), 6.43 (t, $J = 6.8$ Hz, 1H), 3.01 (s, 1H), 2.31–2.26 (m, 1H), 2.18–2.14 (m, 1H), 1.75 (s, 3H), 1.39–1.21 (m, 4H), 0.84 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 146.4, 139.4, 138.8, 136.8, 128.8, 128.3, 128.2, 127.0, 125.5, 125.3, 78.1, 30.8, 30.2, 28.8, 22.5, 13.9; MS *m/z*: 312 (M^+, 3.8), 192 (100), 149 (54), 110 (61), 77 (33), 43 (43); Anal. Found: C, 76.71; H, 7.87. $C_{20}H_{24}OS$ Calcd: C, 76.88; H, 7.74%.

(Z)-2,4-Diphenyl-3-phenylsulfanyl-3-buten-2-ol **4e**. IR (film) ν (cm^{-1}): 3433, 3059, 3025, 2957, 2931, 1719, 1600, 1582, 1493, 1445, 1026, 763, 739, 699; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.59–7.05 (m, 16H), 3.20 (s, 1H), 1.89 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 148.1, 146.1, 135.2, 129.4, 128.7, 128.3, 128.1, 128.0, 127.6, 127.4, 126.5, 125.7, 125.6, 124.8, 78.8, 30.2; MS *m/z*: 332 (M^+, 6.3), 280 (61), 212 (97), 178 (31), 115 (56), 91 (76), 77 (61), 43 (100); Anal. Found: C, 79.27; H, 5.87. $C_{22}H_{20}OS$ Calcd: C, 79.48; H, 6.06%.

(Z)-2-Methyl-3-phenylsulfanyl-5-methoxy-3-penten-2-ol **4f**. IR (film) ν (cm^{-1}): 3431, 3059, 2976, 2927, 1715, 1582, 1478, 1455, 1371, 1185, 1121, 825, 740, 691; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.27–7.21 (m, 4H), 7.15–7.13 (m, 1H), 6.54 (t, $J = 5.2$ Hz, 1H), 4.05 (d, $J = 5.2$ Hz, 2H), 3.28 (s, 3H), 2.30 (br, 1H), 1.45 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 141.5, 136.2, 134.3, 129.1, 126.8, 125.6, 74.2, 71.2, 58.5, 29.0; MS *m/z*: 238 (M^+, 6.4), 220 (12), 110 (57), 91 (49), 77 (42), 45 (76), 43 (100); Anal. Found: C, 65.21; H, 7.47. $C_{13}H_{18}O_2S$ Calcd: C, 65.51; H, 7.61%.

(Z)-2-Methyl-3-(4-methylphenyl)sulfanyl-4-phenyl-3-buten-2-ol **4g**. IR (film) ν (cm^{-1}): 3325, 3078, 3022, 2986, 2921, 1626, 1597, 1490, 1446, 1358, 971, 799, 691; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.62–7.59 (m, 2H), 7.41 (s, 1H), 7.26–7.19 (m, 3H), 7.08 (d, $J = 8.4$ Hz, 2H), 6.96 (d, $J = 8.4$ Hz, 2H), 2.37 (br, 1H), 2.28 (s, 3H), 1.52 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 140.3, 135.9, 135.3, 135.0, 132.7, 129.6, 129.4, 128.0, 127.9, 127.3, 75.5, 29.6, 20.9; MS *m/z*: 284 (M^+, 11.5), 266 (17), 124 (27), 91 (52), 77 (39), 43 (100); Anal. Found: C, 75.78; H, 6.86. $C_{18}H_{20}OS$ Calcd: C, 76.01; H, 7.09%.

(Z)-1-Hexyl-2-(4-methylphenyl)sulfanyl-2-hepten-1-ol **4h**. IR (film) ν (cm^{-1}): 3410, 3019, 2956, 2927, 1713, 1632, 1492, 1457, 806; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.17 (d, $J = 8.0$ Hz, 2H), 7.06 (d, $J = 8.0$ Hz, 2H), 6.23 (t, $J = 7.2$ Hz, 1H), 4.08 (t, $J = 6.4$ Hz, 1H), 2.37-2.29 (m, 5H), 1.82 (br, 1H), 1.66–1.54 (m, 2H), 1.40–1.21 (m, 12H), 0.90–0.85 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 138.6, 135.8, 135.7, 132.3, 129.7, 128.5, 75.6, 36.3, 31.8, 31.2, 29.4, 29.1, 25.7, 22.6, 22.4, 21.0, 14.1, 13.9; MS *m/z*: 320 (M^+, 99), 302 (59), 259 (63), 236 (96), 231 (76), 161 (81), 124 (100), 91 (96), 55 (88); Anal. Found: C, 74.83; H, 9.79. $C_{20}H_{32}OS$ Calcd: C, 74.94; H, 10.06%.

(Z)-1-Hexyl-2-phenylsulfanyl-4-methoxy-2-buten-1-ol **4i**. IR (film): ν (cm^{-1}): 3427, 3059, 2927, 2856, 1716, 1637, 1583, 1478, 1456, 1377, 1194, 1120, 741, 691; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.30–7.24 (m, 4H), 7.20–7.16 (m, 1H), 6.38 (t, $J = 5.6$ Hz, 1H), 4.28–4.23 (m, 1H), 4.14–4.08 (m, 2H), 3.33 (s, 3H), 1.92 (br, 1H), 1.73–1.66 (m, 1H), 1.59–1.52 (m, 1H), 1.39–1.18 (m, 8H), 0.86 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 138.8, 134.9, 133.8, 129.1, 128.8, 126.3, 74.7, 70.2, 58.4, 36.2, 31.7, 29.1, 25.6, 22.6, 14.1; MS *m/z*: 294 (M^+, 34), 205 (64), 167 (82), 135 (85), 109 (84), 71 (100), 55 (80); Anal. Found: C, 69.06; H, 8.87. $C_{17}H_{26}O_2S$ Calcd: C, 69.34; H, 8.90%.

(Z)-1-(1-Phenylsulfanyl-3-methoxy)propenylcyclohexanol **4j**. IR (film) ν (cm^{-1}): 3433, 3058, 2932, 2853, 1716, 1629, 1582, 1478, 1439, 1121, 739, 690; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.27–7.20 (m, 4H), 7.14–7.10 (m, 1H), 6.53 (t, $J = 5.6$ Hz, 1H), 4.05 (d, $J = 5.6$ Hz, 2H), 3.27 (s, 3H), 1.90 (br, 1H), 1.76–1.56 (m, 10H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 141.9, 136.5, 134.7, 129.1, 126.6, 125.4, 74.9, 71.3, 58.5, 36.1, 25.5, 21.9; MS *m/z*: 279 ($M^+ + 1$, 5.2), 261 (38), 152 (95), 110 (53), 91 (100), 71 (57); Anal. Found: C, 68.74; H, 7.87. $C_{16}H_{22}O_2S$ Calcd: C, 69.02; H, 7.97%.

General Procedure for the Synthesis of (Z)-Vinyl Sulfides **5a–b**

To a solution of (*E*)-α-stannylvinyl sulfide **1** (0.5 mmol) in THF (5 mL) at $-78^\circ C$ under argon was added dropwise a solution of *n*-BuLi (0.2 mL, 0.5 mmol, 2.5 M solution in hexane). After 30 min of stirring at this temperature, the mixture was hydrolyzed with saturated aq. NH_4Cl (5 mL) and extracted with Et_2O (2 × 20 mL). The organic extract was washed with water (2 × 10 mL), dried with $MgSO_4$, filtered and concentrated under vacuum. The residue was purified by column chromatography on silica gel eluting with light petroleum ether.

(Z)-1-(4-Methylphenyl)sulfanyl-1-hexene **5a**. IR (film) ν (cm^{-1}): 3074, 2958, 2926, 1716, 1609, 1493, 1459, 1376, 806, 690; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.24 (d, $J = 8.0$ Hz, 2H), 7.10 (d, $J = 8.0$ Hz, 2H), 6.14 (d, $J = 9.2$ Hz, 1H), 5.75 (dt, $J = 9.2$, 7.2 Hz, 1H), 2.32 (s, 3H), 2.27–2.19 (m, 2H), 1.36–1.23 (m, 4H), 0.89 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 136.2, 132.9, 132.6, 129.7, 129.3, 123.6, 31.2, 29.4, 22.4, 21.0, 14.0; Anal. Found: C, 75.39; H, 8.72. $C_{13}H_{18}S$ Calcd: C, 75.67; H, 8.79%.

(Z)-1-Phenyl-2-(4-methylphenyl)sulfanylethene **5b**. IR (film): ν (cm^{-1}) 3073, 2973, 1607, 1590, 1564, 1492, 846, 811, 731; ^1H NMR (400 MHz, $CDCl_3$) δ: 7.54–7.51 (m, 2H), 7.40–7.15 (m, 5H), 7.14 (d, $J = 8.0$ Hz, 2H), 6.53 (d, $J = 10.8$ Hz, 1H), 6.47–6.43 (m, 1H), 2.33 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 137.5, 136.6, 132.7, 130.6, 130.0, 128.8, 128.4, 127.1, 127.0, 126.5, 21.2; Anal. Found: C, 79.42; H, 6.05. $C_{15}H_{14}S$ Calcd: C, 79.60; H, 6.23%.

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