Journal of Organometallic Chemistry 693 (2008) 3787–3790

Contents lists available at ScienceDirect

Journal of Organometallic Chemistry

journal homepage: www.elsevier.com/locate/jorganchem

A simple and general preparation of vinylic sulfides, selenides and tellurides

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ARTICLE INFO

Article history:
Received 15 August 2008
Received in revised form 22 September 2008
Accepted 23 September 2008
Available online 30 September 2008

Keywords:
Vinylic sulfides
Vinylic selenides
Vinylic tellurides
Nickel catalysis

ABSTRACT

A general method for the synthesis of vinylic chalcogenides by nucleophilic and Ni-catalyzed vinylic substitution on vinylic halides by phenyl chalcogenolates is described. The reactions were regio and stereoselective for the nickel catalyzed substitution, and mixture of isomers was observed for some examples in the thermal process in DMF.

1. Introduction

Organochalcogen compounds play an important role in modern organic synthesis in view of their chemo, regio, and stereoselective reactions [1–12] and the useful biological activities, as recently reviewed [13]. From the different classes of organochalcogen compounds, vinylic chalcogenides constitute a very useful group and have attracted considerable attention in recent years as synthetic precursors [14–16]. Many vinylic sulfide, selenide and telluride preparations involve the use of expensive catalysts or starting materials that are not readily available or the products are obtained as mixtures of regio or stereoisomers [17–26]. There is still a lack of simple, efficient methods for their preparation. This is a serious drawback that overcomes their preparation on large scale and their use in organic synthesis. In the search for a general method that allows the preparation of vinylic sulfides, selenides and tellurides in an easy and inexpensive way, in large-scale reactions, we sought the use of vinylic halides as starting materials, since many methods are available for their diastereoselective preparation [27,28].

2. Results and discussion

The corresponding diphenyl dichalcogenides, that are commercially available or easily prepared in large-scale reaction, by well established methods [1,29,30], would be a very convenient source of phenylchalcogenide anion for the vinylic substitution reaction on vinylic halides. At the first experiments in the search for the best reactional conditions, the dichalcogenides were reduced by $NaBH_4$ in ethanol and β-bromo styrene was added, followed by heating at reflux. Unfortunately, no reaction was observed. In view of the need for a higher temperature to perform the reaction, we decided to remove the ethanol using a Dean–Stark apparatus and adding DMF as the solvent. After addition of the appropriate vinylic halide, the reaction was heated at reflux temperature for 2 h, and we observed total consumption of starting materials, Scheme 1. These conditions were then applied to other examples as described in Table 1. On the synthesis of the vinylic sulfides, the reaction with β-bromostyrene and 4-chloro β-bromostyrene furnish the *E*-styryl sulfides **5a** and **5b** in 67 and 72% yield, respectively (entries 1 and 2, Table 1). The reaction with *Z*-vinylic halides **4c** and **4e**, however, furnishes the *Z*-vinylic sulfides **5c** and **5e** along with ca. 10% of the *E*-isomer (entries 3 and 5, Table 1). In the case of 4-chloro β-bromostyrene, pure *Z*-isomer **5d** was isolated (entry 4). The reaction with the internal halo alkenes **4f–g** followed a different pathway, and the corresponding *Z*-vinylic sulfides **5c** and **8** were isolated. In this case, in view of the lower nucleophilic character of the thiolate anion, the formed product suggests that a prior elimination reaction was occurring, generating the terminal alkyne followed by trans-selective addition of the thiol or thiolate giving the corresponding *Z* product, as expected from previous results [31,32]. We next turned our attention to the selenium derivatives, reacting with the same vinylic halides. In this case, the reactions showed to be very selective and pure *E* and *Z* isomers could be isolated by the reaction with *E* and *Z* β–bromostyrenes. The only exception was in the reaction with internal vinylic halides, where a 1:1 mixture of the expected products **6f** and **6g** along with *Z*–vinylic selenides **6c** and **9** could be isolated. We suppose that in this case, in view of the higher nucleophilicity of the selenolate an-

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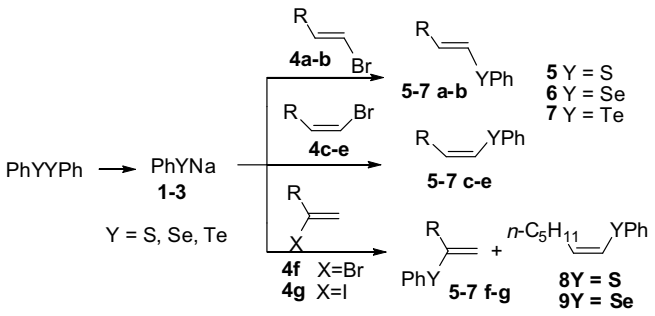
doi:10.1016/j.jorganchem.2008.09.039



Scheme 1. Thermal phenylchalcogenolates substitution on vinyl halides.

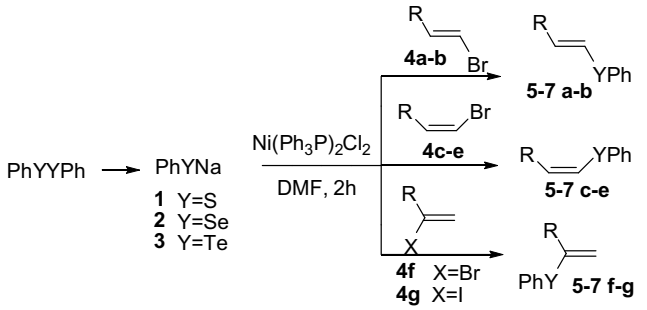
Table 1
Preparation of vinyl chalcogenides from vinyl halides according to Scheme 1

Entry	Y	R	Vinyl halide	Products	Yield (%)
1	S	C_6H_5	**4a**	**5a**	67
2	S	4-Cl–C_6H_4	**4b**	**5b**	72
3	S	C_6H_5	**4c**	**5c + 5a**[a]	84
4	S	4-Cl–C_6H_4	**4d**	**5d**	86
5	S	4-CH_3–C_6H_4	**4e**	**5e + *E***[a]	66
6	S	C_6H_5	**4f**	**5f**	73
7	S	C_5H_{11}	**4g**	**8**	53
8	Se	C_6H_5	**4a**	**6a**	75
9	Se	4-Cl–C_6H_4	**4b**	**6b**	75
10	Se	C_6H_5	**4c**	**6c**	80
11	Se	4-Cl–C_6H_4	**4d**	**6d**	84
12	Se	4-CH_3–C_6H_4	**4e**	**6e**	87
13	Se	C_6H_5	**4f**	**6f + 6c**[a]	58
14	Se	C_5H_{11}	**4g**	**6g + 9**[a]	58
15	Te	C_6H_5	**4a**	**7a**	70
16	Te	4-Cl–C_6H_4	**4b**	**7b**	80
17	Te	C_6H_5	**4c**	**7c**	78
18	Te	4-Cl–C_6H_4	**4d**	**7d**	88
19	Te	4-CH_3–C_6H_4	**4e**	**7e**	81
20	Te	C_6H_5	**4f**	**7f**	65
21	Te	C_5H_{11}	**4g**	**7g**	64

[a] See text for details.

Table 2
Preparation of vinyl chalcogenides by Ni-catalyzed vinyl substitution

Entry	Y	R	Vinyl halide	Products	Yield (%)
1	S	C_6H_5	**4a**	**5a**	72
2	S	4-Cl–C_6H_4	**4b**	**5b**	82
3	S	C_6H_5	**4c**	**5c**	98
4	S	4-Cl–C_6H_4	**4d**	**5d**	86
5	S	4-CH_3–C_6H_4	**4e**	**5e**	70
6	S	C_6H_5	**4f**	**5f**	70
7	S	C_5H_{11}	**4g**	**5g**	71
8	Se	C_6H_5	**4a**	**6a**	90
9	Se	4-Cl–C_6H_4	**4b**	**6b**	78
10	Se	C_6H_5	**4c**	**6c**	85
11	Se	4-Cl–C_6H_4	**4d**	**6d**	82
12	Se	4-CH_3–C_6H_4	**4e**	**6e**	90
13	Se	C_6H_5	**4f**	**6f**	67
14	Se	C_5H_{11}	**4g**	**6g**	62
15	Te	C_6H_5	**4a**	**7a**	72
16	Te	4-Cl–C_6H_4	**4b**	**7b**	80
17	Te	C_6H_5	**4c**	**7c**	85
18	Te	4-Cl–C_6H_4	**4d**	**7d**	88
19	Te	4-CH_3–C_6H_4	**4e**	**7e**	82
20	Te	C_6H_5	**4f**	**7f**	65
21	Te	C_5H_{11}	**4g**	**7g**	66



Scheme 2. Ni-catalyzed phenylchalcogenolates substitution on vinyl halides.

ion, there is a competition between direct substitution (to give **6f** and **6g**) and the elimination/addition mechanism to give **6c** and **9**. Following the study with the tellurium derivatives, all the reactions gave the expected products, i.e. *E*, *Z* and internal vinylic tellurides. In this case, no mixture of products was observed, probably as a consequence of the higher nucleophilicity of tellurolate anion.

In a search for a cleaner reaction pathway to prepare vinylic chalcogenides avoiding the formation of isomers as discussed above, we decided to perform the reactions using a cheap metal catalyst. Nickel was our first choice since we described recently coupling reactions using the Ni catalyst [33] and some years ago it was described the Ni-bipy vinylic selenolate substitution on activated vinyl bromides [34,35]. From the many nickel species, we decided to use $Ni(Ph_3P)_2Cl_2$ since it is very stable, cheap and easy to prepare. In the search for a good experimental condition, which could furnish the desired products in an easy way, we followed the same procedure used in the study described above, which was the cleavage of diphenyl dichalcogenides by $NaBH_4$ in ethanol, followed by addition of DMF (the removal of ethanol was observed not to be necessary), the vinylic halide and the catalyst (5 mol%) and the reaction heated at the appropriate temperature. Under these conditions, with appropriate heating for each different chalcogenium nucleophilic specie (see below), we were pleased to observe that desired products were formed in very clean reactions and in good yields. The results described on Table 2, Scheme 2, summarize the results of this study. For the reaction using the sulfur analogs, all reactions were performed at 60 °C and the desired products were obtained in high yields, with very high regio and stereoselectivity (entries 1–7, Table 2). Similar results were observed on the reactions with the selenium and tellurium analogs, the only difference being on the necessary temperature to reaction to occur, 70 °C for selenium and 110 °C for tellurium analogs, respectively. In all cases, no mixture of products was observed and in all examples studied medium to high yields of the products was observed. In general, the yields using the Ni catalyst were higher than the thermal process.

The reactions were performed on a 5 mmol scale, but it can be scaled up with comparable yields and reaction times. In conclusion, we have developed a simple and efficient method for the preparation of vinylic chalcogenides from vinylic halides and diphenyl dichalcogenides, based on very simple procedures that allow the preparation of vinylic chalcogenides in high yields, and most important in large-scale reactions.

3. Experimental

3.1. General

All ^1H (400 and 200 MHz) and ^{13}C (100 and 50 MHz) NMR spectra were recorded on Bruker DPX 400 and DPX 200 instruments, using $CDCl_3$ as solvent. Chemical shifts (δ) are expressed in ppm downfield from internal tetramethylsilane or $CHCl_3$, and *J* values are given in Hz. Infrared spectra were recorded on a Nicollet–Magna spectrometer. Elemental analyses were carried out on a Perkin–Elmer 2400. All reactions were performed in flame-dried glassware

under a positive pressure of argon. Air- and moisture sensitive reagents and solvents were transferred via syringe, and were introduced into reaction vessels through a rubber septum. All the reactions were monitored by thin layer chromatography (TLC) carried out using Merck 60 F_{254} plates with a 0.25 mm thickness. Column chromatography was carried out using Merck silica gel 60 (230–400 mesh).

3.2. General procedure for the uncatalyzed preparation of vinylic chalcogenides

To a two-necked 50 mL round-bottomed flask equipped with a Dean–Stark and a reflux condenser were added diphenyl dichalcogenides (2.5 mmol), ethanol (10 mL), sodium borohydride (0.270 g, 7.5 mmol) and the mixture was stirred at room temperature. After the mixture was clear (ca. 10 min), DMF (15 mL) and the vinylic halide (5 mmol) were added. Then the mixture was stirred at reflux temperature for 2 h while the ethanol was collected in a Dean–Stark apparatus. The reaction mixture was cooled to room temperature and extracted with ethyl acetate (3 × 50 mL). The organic layer was washed with NH_4Cl saturated aqueous solution (50 mL) and water (100 mL), and dried over $MgSO_4$. The solvent was removed and the residue was purified by silica-gel chromatography eluting with hexanes.

3.3. General procedure for the preparation of vinylic chalcogenides catalyzed by $Ni(PPh_3)_2Cl_2$

To a two-necked 50 mL round-bottomed flask equipped with a Dean–Stark and a reflux condenser were added diphenyl dichalcogenides (2.5 mmol), ethanol (10 mL), sodium borohydride (0.270 g, 7.5 mmol) and the mixture was stirred at room temperature. After the mixture was clear (ca. 10 min), DMF (15 mL) was added. After 10 min the vinylic halide (5 mmol) and $Ni(Ph_3P)_2Cl_2$ (0.170 g; 0.25 mmol) were added. Then the mixture was stirred at 60 °C (for diphenyldisulfide), at 70 °C (for diphenyldiselenide) or 110 °C (for diphenylditelluride) for 2 h. After that, was cooled to room temperature and extracted with ethyl acetate (3 × 50 mL). The organic layer was washed with NH_4Cl saturated aqueous solution (50 mL) and water (100 mL), and dried over $MgSO_4$. The solvent was removed and the residue was purified by silica-gel chromatography eluting with hexanes.

(*E*)-Phenyl(styryl)sulfide (**5a**) [36]: ^{1}H NMR (200 MHz, $CDCl_3$) δ 6.70 (d, J = 15.4 Hz, 1H); 6.86 (d, J = 15.4 Hz, 1H); 7.19–7.34 (m, 8H); 7.37–7.42 (m, 2H). ^{13}C NMR (50 MHz, $CDCl_3$) δ 123.2, 125.9, 126.7, 127.4, 128.5, 129.0, 129.6, 131.6, 135.1, 136.3.

(*E*)-(4-Chlorostyryl)(phenyl)sulfide (**5b**) [37]: ^{1}H NMR (200 MHz, $CDCl_3$) δ 6.51 (d, J = 15.6 Hz, 1H); 6.86 (d, J = 15.6 Hz, 1H); 7.23–7.43 (m, 9H). ^{13}C NMR (50 MHz, $CDCl_3$) δ 124.6, 127.3, 127.2, 128.8, 129.2, 129.7, 130.2, 133.1, 134.7, 135.0.

(*Z*)-Phenyl(styryl)sulfide (**5c**): m.p. 39–41 °C (Lit. [38] 39–42 °C; Lit. [39] 43–44 °C).

(*Z*)-(4-Chlorostyryl)(phenyl)sulfide (**5d**) [40,41]: m.p. 84–86 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.49 (d, J = 10.8 Hz, 1H); 6.46 (d, J = 10.8 Hz, 1H); 7.18–7.32 (m, 5H); 7.40–7.43 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 125.8, 126.9, 127.3, 128.4, 129.1, 129.9, 130.1, 132.6, 134.9, 135.7.

(*Z*)-(4-Methylstyryl)(phenyl)sulfide (**5e**) [41]: m.p. 51–53 °C. ^{1}H NMR (200 MHz, $CDCl_3$) δ 2.36 (s, 3H); 6.43 (d, J = 10.6 Hz, 1H); 6.57 (d, J = 10.6 Hz, 1H); 7.17–7.47 (m, 9H). ^{13}C NMR (50 MHz, $CDCl_3$) δ 21.3, 124.8, 127.0, 127.4, 128.7, 129.0, 129.1, 130.0, 133.7, 136.4, 137.0.

Phenyl(1-phenylvinyl)sulfide (**5f**) [42]: ^{1}H NMR (200 MHz, $CDCl_3$) δ 5.27 (s, 1H); 5.65 (s, 1H); 7.20–7.70 (m, 10H). ^{13}C NMR (50 MHz, $CDCl_3$) δ 115.7, 127.0, 127.3, 128.2, 128.4, 129.0, 131.9, 133.8, 138.7, 144.4.

Hept-1-en-2-yl(phenyl)sulfide (**5g**) [43]: ^{1}H NMR (200 MHz, $CDCl_3$) δ 0.88 (t, J = 7.0 Hz, 3H); 1.15–1.38 (m, 4H); 1.55 (qui, J = 7.2 Hz, 2H); 2.22 (t, J = 7.4 Hz, 2H); 4.87 (s, 1H); 5.14 (s, 1H); 7.29–7.46 (m, 5H). ^{13}C NMR (50 MHz, $CDCl_3$) δ 14.0, 22.4, 28.1, 31.1, 36.5, 112.4, 127.5, 127.6, 129.0, 133.2, 146.1.

(*E*)-Phenyl(styryl)selenide (**6a**) [44]: ^{1}H NMR (200 MHz, $CDCl_3$) δ 6.84 (d, J = 15.6 Hz, 1H); 7.15 (d, J = 15.6 Hz, 1H); 7.20–7.29 (m, 8H); 7.48–7.60 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 119.3, 125.9, 127.2, 127.4, 128.5, 129.1, 130.1, 132.3, 134.9, 136.8.

(*E*)-(4-Chlorostyryl)(phenyl)selenide (**6b**) [37]: ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.05 (d, J = 16.4 Hz, 1H); 7.50 (d, J = 16.4 Hz, 1H); 7.15–7.35 (m, 8H); 7.72–7.75 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$ δ 120.7, 127.1, 127.6, 128.7, 129.4, 129.6, 132.8, 133.0, 133.1, 135.4.

(*Z*)-Phenyl(styryl)selenide (**6c**): m.p. 43–45 °C (Lit. [45] 44–45 °C).

(*Z*)-(4-Chlorostyryl)(phenyl)selenide (**6d**): m.p. 80–82 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.79 (d, J = 10.4 Hz, 1H); 6.89 (d, J = 10.4 Hz, 1H); 7.28–7.32 (m, 7H); 7.53–7.58 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 124.9, 127.8, 128.6, 128.8, 129.4, 129.5, 131.3, 132.8, 132.9, 135.7. MS: m/z = 296.0, 295.1, 294.3, 214.0, 179.1, 156.3, 136.0, 101.0, 89.0, 77.1, 51.3. Anal. Calc. for $C_{14}H_{11}ClSe$: C: 57.26; H: 3.75. Found: C: 56.83; H: 3.67%.

(*Z*)-(4-Methylstyryl)(phenyl)selenide (**6e**) [37]: m.p. 45–47 °C ^{1}H NMR (200 MHz, $CDCl_3$) δ 6.69 (d, J = 10.4 Hz, 1H); 6.91 (d, J = 10.4 Hz, 1H); 7.13–7.31 (m, 8H); 7.52–7.55 (m, 2H). ^{13}C NMR (50 MHz, $CDCl_3$) δ 21.2, 122.7, 127.5, 128.2, 129.0, 129.2, 130.1, 131.7, 132.6, 134.4, 137.1.

Phenyl-(1-phenylvinyl)selenide (**6f**) [46]: ^{1}H NMR (200 MHz, $CDCl_3$) δ 5.37 (s, 1H); 5.90 (s, 1H); 7.21–7.30 (m, 8H); 7.50–7.59 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 117.8, 127.3, 127.7, 128.2, 128.3, 129.2, 131.5, 134.1, 140.1, 141.9.

Hept-1-en-2-yl(phenyl)selenide (**6g**) [47]: ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.86 (t, J = 7.0 Hz, 3H); 1.20–1.32 (m, 4H); 1.49 (qui, J = 7.2 Hz, 2H); 2.31 (t, J = 7.4 Hz, 2H); 5.42 (s, 1H); 5.96 (s, 1H); 7.20–7.30 (m, 3H); 7.77–7.79 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 22.4, 29.1, 30.9, 42.1, 113.1, 124.3, 127.9, 129.3, 130.7, 139.3.

(*E*)-Phenyl(styryl)telluride (**7a**) [48]: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.08 (d, J = 16.4 Hz, 1H); 7.52 (d, J = 16.4 Hz, 1H); 7.18–7.28 (m, 8H); 7.71–7.73 (d, J = 8.3Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 101.5, 113.5, 126.0, 127.7, 127.8, 128.4, 129.4, 137.7, 138.0, 143.1.

(*E*)-(4-Chlorostyryl)(phenyl)telluride (**7b**) [49]: ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.98 (d, J = 16.4 Hz, 1H); 7.51 (d, J = 16.4 Hz, 1H); 7.15–7.32 (m, 7H); 7.67–7.78 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 102.8, 113.1, 127.2, 128.1, 128.7, 129.5, 133.4, 136.5, 138.1, 141.1.

(*Z*)-Phenyl(styryl)telluride (**7c**) [50]: m.p. 40–42 °C; ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.06 (d, J = 10.6 Hz, 1H); 7.43 (d, J = 10.6 Hz, 1H); 7.13–7.37 (m, 8H); 7.70–7.75 (m, 2H). ^{13}C NMR (50 MHz, $CDCl_3$) δ 109.15, 115.3, 127.3, 127.4, 128.0, 128.4, 129.3, 136.8, 137.9, 138.8.

(*Z*)-(4-Chlorostyryl)(phenyl)telluride (**7d**): m.p. 72–74 °C (Lit. [51] 72.5–74 °C).

(*Z*)-(4-Methylstyryl)(phenyl)telluride (**7e**): m.p. 54–56 °C (Lit. [51] 54.5–55.5 °C).

Phenyl-(1-phenylvinyl)telluride (**7f**): ^{1}H NMR (200 MHz, $CDCl_3$) δ 5.6 (s, 1H); 6.28 (s, 1H); 7.12–7.30 (m, 6H); 7.60–7.80 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 114.4, 125.2, 127.5, 128.0, 128.1, 129.4, 137.9, 139.1, 142.8. MS: m/z = 310.0, 308.0, 206.9, 180.0, 130.0, 103.3, 77.2, 51.0. Anal. Calc. for $C_{14}H_{12}Te$: C: 54.62, H: 3.93; Found: C: 54.82, H: 3.85%.

Hept-1-en-2-yl(phenyl)telluride (**7g**): ^{1}H NMR (200 MHz, $CDCl_3$) δ 0.86 (t, J = 7.0 Hz, 3H); 1.20–1.35 (m, 4H); 1.49 (qui, J = 7.2 Hz, 2H); 2.31(t, J = 7.4 Hz, 2H); 5.42 (s, 1H); 5.96 (s, 1H); 7.17–7.27 (m, 3H); 7.76–7.80 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$)

δ 14.0, 22.4, 28.0, 29.5, 34.0, 114.2, 128.0, 128.6, 129.3, 131.0, 136.5. MS: m/z = 304.0 245.9, 207.9, 153.0, 129.9, 118.15, 97.15, 91.10, 77.7, 67.0, 55.1, 51.1.

Acknowledgements

The authors thank FAPERGS, CAPES and MCT/CNPq for financial support.

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